Risk Management Phase 2

Risk Issue Investigation Development of Recommendations

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RIM 57
Monitoring in the Metal Parts Furnace Discharge Airlock
FINAL REPORT
March 12, 2003

Prepared for:

PROGRAM MANAGER FOR CHEMICAL DEMILITARIZATION
DEPARTMENT OF THE ARMY OPERATIONS SUPPORT COMMAND

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CHEMICAL STOCKPILE DISPOSAL PROGRAM

RISK MANAGEMENT PHASE 2:

RISK ISSUE INVESTIGATION and DEVELOPMENT OF RECOMMENDATIONS

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Prepared for:

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ACRONYMS AND ABBREVIATIONS

ACAMS	Automatic Continuous Air Monitoring System
ANCDF	Anniston Chemical Agent Disposal Facility
BSA	Buffer Storage Area
CBW	Chemical and Biological Weapons
CFD	Computational Fluid Dynamics
CI	Chemical Ionization
CONUS	Continental United States
CPA	Conversion Pad Assembly
CR&E	Continental Research and Engineering
DAAMS	Depot Area Air Monitoring System
DAL	Discharge Airlock
DFS	Deactivation Furnace System
DHHS	Department of Health and Human Services
DMMP	Dimethyl methylphosphonate
ECP	Engineering Change Proposal
EMO	Environmental and Monitoring Office
EPA	U.S. Environmental Protection Agency
°F	Degrees Farenheit
ft	Feet
GB	Nerve Agent (Sarin)
GC	Gas Chromatograph

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ACRONYMS AND ABBREVIATIONS

GC/FPD/MS	Gas Chromatograph/Flame Photometric Detector/Mass Spectrophotometer
GC/EI/MS	Gas Chromatography/Electron Impact/Mass Spectrophotometer
GC/IT/MS	Gas Chromatography/Ion Trap/Mass Spectrophotometer
GCMS	Gas Chromatograph Mass Spectrophotometer
gm	Gram
HD/HT	Mustard Blister Agents
HDC	Heated Discharge Conveyor
hr	Hour
HWC	Hazardous Waste Combustor
JACADS	Johnston Atoll Chemical Agent Disposal System
IITRI	Illinois Institute of Technology Research Institute
INEEL	Idaho National Engineering and Environmental Laboratory
°K	Degrees Kelvin
kg	Kilogram
lbs	Pounds
LC/MS	Liquid Chromatography/Mass Spectrophotometer
LIC	Liquid Incinerator
LOC	Limit of Quantification
Lpm	Liters Per Minute
MACT	Maximum Achievable Control Technology
mg	Milligram
MPF	Metal Parts Furnace

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ACRONYMS AND ABBREVIATIONS

MS	Matrix Spike
MSD	Matrix Spike Duplicate
MVEP	Mandatory Value Engineering Program
NMR	Nuclear Magnetic Resonance
PAS	Pollution Abatement System
PBCDF	Pine Bluff Chemical Agent Disposal Facility
PDAR	Plant Data Acquisition Recording
PMCD	Program Manager for Chemical Demilitarization
PMCSD	Project Manager for Chemical Stockpile Disposal
POHCs	Principal Organic Hazardous Constituents
ppb	Parts Per Billion
ppm	Parts Per Million
QA	Quality Assurance
RCRA	Resource Conservation and Recovery Act
RDTE	Research Development Test and Evaluation
REI	Reaction Engineering International, Inc.
RH	Relative Humidity
RIM	Risk Issues Matrix
RMT	Risk Management Team
ROM	Rough Order of Magnitude
SBIR	Small Business Innovative Research
sec	Second

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ACRONYMS AND ABBREVIATIONS

SETH	Simulated Equipment Test Hardware
SOPS	Standing Operating Procedures
SwRI	Southwest Research Institute
TOCDF	Tooele Chemical Agent Disposal Facility
TSDF	Treatment, Storage, and Disposal Facility
TWA	Time Weighted Average
UDRI	University of Dayton Research Institute
UMCDF	Umatilla Chemical Agent Disposal Facility
VECP	Value Engineering Change Proposal
VX	Nerve Agent
WCL	Waste Control Limit (20 ppb VX, 20 ppb GB, 200 ppb HD)
WDC	Washington Demilitarization Company
WIC	Waste Incineration Container

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EXECUTIVE SUMMARY

On 29 August 2002, the final draft of the Investigation Report into the 12 August 2002 Johnston Atoll Chemical Agent Disposal System (JACADS) unusual occurrence was signed and approved for release from the JACADS Project to the Office of the Program Manager for Chemical Demilitarization (PMCD). During its review of the report, the Edgewood Office of the PMCD identified three areas that merited further discussion and investigation:

- The possibility that the detection of nerve agent (VX) was due to interfering compounds.
- The 259°F temperature for monitoring in the discharge airlock (DAL), derived from a 1962 Cornell Aeronautical Laboratory study was too conservative.
- The possibility that the 18-inch stainless steel probe on the Automatic Continuous Air Monitoring System (ACAMS) sampling line in the DAL contributed to the monitoring problem(s) identified in this report.

The Risk Team evaluated each of these issues.

Analytical Methods

Three different laboratories were used to evaluate the potential for false positives while using the G-analog technique. The purpose of the effort was to confirm or refute the presence of VX in the residue from the sludge that had been improperly loaded in tray 153. All methods used to date have confirmed the presence of VX in the residue collected from tray 153. Based on these multi-laboratory/multi-method results it is reasonable to conclude that there was VX in the residue. This result is consistent with the ACAMS alarms and the confirmed Depot Area Air Monitoring System (DAAMS) tubes that were collected after the tray had been returned to the DAL. In the absence of other results, these results do not support the hypothesis that the positive responses were caused by an interferant.

Thermal Degradation of VX in the Discharge Airlock

Using recently developed detailed chemical kinetic mechanisms for GB and HD, and a calculated rate for the first steps of the dominant VX decomposition pathways, Reaction Engineering International (REI) calculated destruction of the three agents for conditions anticipated to be found in the DAL. The conclusions of the REI expert team was that the data used by the JACADS investigation team is unreliable due to poorly characterized experimental conditions, calculation errors and huge discrepancies between the reported rates and rates of reaction for similar molecules. REI has calculated that effective DAL monitoring can be achieved up to about 760°F for VX, 690°F for GB and 870°F for HD.

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The 18-inch Probe

The 18-inch probe study was completed and a recommendation is made to provide JACADS and Tooele Chemical Agent Disposal Facility (TOCDF) with a viable and effective monitoring program for the discharge airlock and the heated discharge conveyor bin enclosure. JACADS should continue using the existing air-cooled probe with the Conversion Pad Assembly (CPA) at 5-inches from the distal end for ACAMS monitoring; and the CPA at approximately 18-inches from the distal end for DAAMS monitoring at 3 liter per minute (Lpm). JACADS can raise the monitoring temperature to 600°F. This temperature is a conservative temperature that will extend the life of the CPA, and is below the maximum temperature of 760°F for VX as calculated by REI. This will provide JACADS with a viable monitoring approach as well as needed schedule relief to complete closure in a timely and safe manner.

TOCDF should immediately configure the DAL with the air-cooled probe designed at JACADS. TOCDF should place the CPA at the same locations in the probe that JACADS has established. TOCDF should reprogram the logic in the monitoring system for the DAL, to initiate monitoring when the DAL reaches 600°F. Because TOCDF is still processing significant quantities of liquid chemical agent, while JACADS is processing only secondary waste and decommissioning materials, it is recommended that TOCDF monitor the temperature of the DAL itself and not the exhaust duct. The DAL should be cooled to a maximum temperature of 600°F prior to initiating sampling. Measuring the temperature at this location will ensure that the temperature at the monitoring point is at or below the 600°F limitation. This system will provide TOCDF with a safe and effective start-up condition for the VX campaign.

Long Term Mitigating Measures

TOCDF has completed a test of cooling in the DAL using Simulated Equipment Test Hardware (SETH) 155mm munitions. This test was designed to provide the rate of temperature decay for a tray of projectiles. The total cool-down time for a tray of munitions to 600°F was on the order of 3 hours. Under the current DAL configuration, the impact to the program of reduced MPF throughput is estimated at $1.8 billion. This test provided sufficient engineering detail to evaluate mitigating efforts needed to allow a more rapid cooling of the tray in the DAL. Several options being considered to reduce DAL cooling time and increase MPF throughput are:

- Additional 8-inch air inlet valve to the TOCDF DAL to increase cooling air flow
- Removal of insulation from the DAL to facilitate cooling
- Addition of a second airlock to allow rapid cooling and monitoring
- Addition of engineered cooling techniques to the existing DAL, i.e., water-cooled walls, high-velocity air lance, etc.
- Total enclosure or partial enclosure of the munitions-cool-down conveyor to allow additional cooling time under engineering control

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The evaluation of these options and subsequent detailed engineering of the selected option is currently in progress under Phase 3 of RIM 57.

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1 INTRODUCTION

1.1 Risk Management Program Overview

The Project Manager for Chemical Stockpile Disposal (PMCSD) requires the implementation of a Mandatory Value Engineering Program (MVEP) to identify, assess, and propose innovative and creative approaches to improve schedule and cost performance across the Chemical Agent Disposal Program. For the MVEP risk management activities, the PMCSD initially contracted with Washington Demilitarization Company (WDC) to perform an evaluation of plant operations and facility design to evaluate both cost/schedule risks as well as opportunities for improving overall project schedule and cost. EG&G is WDC's team subcontractor under a contractual agreement for the MVEP.

1.2 Background of Risk Issues Matrix (RIM) 57

On 12 August 2002, a residue subsequently characterized as having VX contamination was found on a tray that had been processed through the Johnston Atoll Chemical Agent Disposal System (JACADS) Metals Parts Furnace (MPF). There was no VX agent alarm from the Automatic Continuous Air Monitoring System (ACAMS) unit monitoring the Discharge Airlock (DAL) of the MPF during the normal 22-minute hold time prior to release. An alarm for HD, received within minutes of discharging the tray, alerted operators that something was unusual, and it was restored to the DAL which is under engineering controls. Monitoring was initiated and the ACAMS units in the DAL went into alarm for nerve agent Sarin (GB), VX, and mustard blister agent (HD) after return of the tray. The readings for GB and HD were refuted by analysis of the Depot Area Air Monitoring (DAAMS) tubes. VX was confirmed through analysis of the DAAMS tubes using a Gas Chromatograph – Mass Spectrophotometer (GCMS) Detector. Samples were later recovered from the residue remaining on the tray and analyzed within the JACADS laboratory. This analysis confirmed varying concentrations of VX within the residue.

On 29 August 2002, the final draft of the Investigation Report into the 12 August 2002 JACADS unusual occurrence was signed and approved for release from the JACADS Project to the Office of the Program Manager for Chemical Demilitarization (PMCD). During its review of the report, the Edgewood Office of the PMCD identified three areas that merited further discussion and investigation. Those three areas were:

- The possibility that the detection of nerve agent (VX) was due to interfering compounds.
- The 259°F temperature for monitoring in the DAL, derived from a 1962 Cornell Aeronautical Laboratory study was too conservative.

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- The possibility that the 18-inch stainless steel probe on the ACAMS sampling line in the DAL contributed to the monitoring problem(s) identified in this report.

The risk team was provided full access to supplemental information and resources to allow them to explore the areas identified by PMCD staff.

1.3 System Contractor Responsibilities and Deliverables

It is the responsibility of WDC/EG&G to investigate and make recommendations associated with Risk Issues Matrix (RIM) 57 to reduce cost and schedule. This report addresses these issues. Immediate and long term program needs with specific contract deliverables have been identified to support this effort. Future deliverables will include an update to this report based on findings from ongoing studies, and if appropriate, Value Engineering Change Proposals (VECP) for engineering changes to the Continental United States (CONUS) facilities.

Immediate program needs/deliverables are:

1. **Program Need:** Identify an appropriate temperature for the monitoring of GB and VX; and HD in the DAL.

   Deliverable: An analysis of the reaction kinetics for thermal decomposition of agents VX, GB and HD at the temperatures anticipated in the DAL.

   Deliverable Status: This analysis was completed by Reaction Engineering, International (REI). This document is Appendix A to this report.

2. **Program Need:** Identify an appropriate monitoring configuration that can effectively monitor the DAL at the temperature(s) identified above.

   Deliverable: Develop a monitoring configuration for agent VX that is consistent with the requirements of the PMCD monitoring concept plan.

   Deliverable Status: The risk team has identified a final approach to this issue.

   A stainless steel probe that is 18-inch long, with the Conversion Pad Assembly (CPA) at 5-inches from the distal end for ACAMS monitoring and the CPA at the proximal end of the 18-inch probe for DAAMS monitoring. The test plan and final report for the use of this probe is provided in Appendix C of this report.

3. **Program Need:** Determine if other agent monitoring locations are affected by this temperature dependency, and make a recommendation to provide effective monitoring at these locations. In particular, the Deactivation Furnace System (DFS) Heated Discharge Conveyor (HDC) bin enclosure may require some corrective actions.

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Deliverable: Develop a conceptual model of the air flow and temperature regime in the DAL.

Deliverable Status: This model was developed by Continental Research and Engineering (CR&E) in Denver, Colorado, and some initial output is provided in Appendix D of this report. This model will be used to evaluate the effectiveness of specific mitigation efforts during Phase 3 of this study.

4. **Program Need:** Determine if the currently accepted G-analog technique for the analysis of residue for agent VX is acceptable.

   Deliverable: Prepare an evaluation of the analytical results from deployment of various methods to analyze ash samples for agent VX.

   Deliverable Status: This evaluation has been completed. It compares the different analytical techniques used and how they compare quantitatively and qualitatively to the techniques used by the PMCD program. This report is provided in Appendix E to this report.

5. Identify specific (minor) changes to the existing TOCDF DAL to allow rapid cooling of the DAL to eliminate impact to schedule. These changes could include the installation of a larger inlet valve, or the removal of the DAL insulation material

   Deliverable: Prepare an evaluation of the test results from the SETH munitions test at TOCDF.

   Deliverable Status: This test is complete. A summary of this data is presented in Appendix F.

Long-term needs for the program are:

1. **Program Need:** Identify specific (major) changes to the existing TOCDF DAL to allow rapid cooling of the DAL to eliminate impact to schedule, if necessary.

   Deliverable: Engineering Change Proposal (ECP) for changes to the DAL and/or munitions cool-down conveyor.

   Deliverable Status: Conceptual design has been initiated and will be provided in a separate deliverable to the government.

2. **Program Need:** Identify specific engineering changes to the existing CONUS facilities to ensure appropriate and effective monitoring.

   Deliverable: Evaluation of CONUS facility configurations & ECPs if applicable.

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Deliverable Status: Evaluation of CONUS facility configurations underway and will be provided in a separate deliverable to the government.

3. **Program Need:** An evaluation of the availability of various techniques for adding a thermal dosimetry device for monitoring secondary waste to the testing program at CONUS facilities.

   Deliverable: Engineering analysis of the applicability of using thermal dosimeters for use in evaluating the treatment of secondary wastes.

   Deliverable Status: CR&E is providing this report.

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2 METHODOLOGY FOR EXECUTION OF PHASE 2 INVESTIGATION

Several efforts were initiated in parallel to address the immediate Program Needs. The Risk Team either procured subcontracted services to perform analysis/modeling work or internally performed research/analyses to address these areas. The methodologies employed for a specific deliverable are discussed further in Section 3 and are addressed in detail as necessary in the subject report that satisfies that deliverable. Refer to the Appendix identified in Section 1.3 associated with each deliverable.

Work to address the areas identified as long term program needs remains in progress. As with the immediate deliverables, specific methodologies employed to address each of these areas will be included in the associated deliverable documents.

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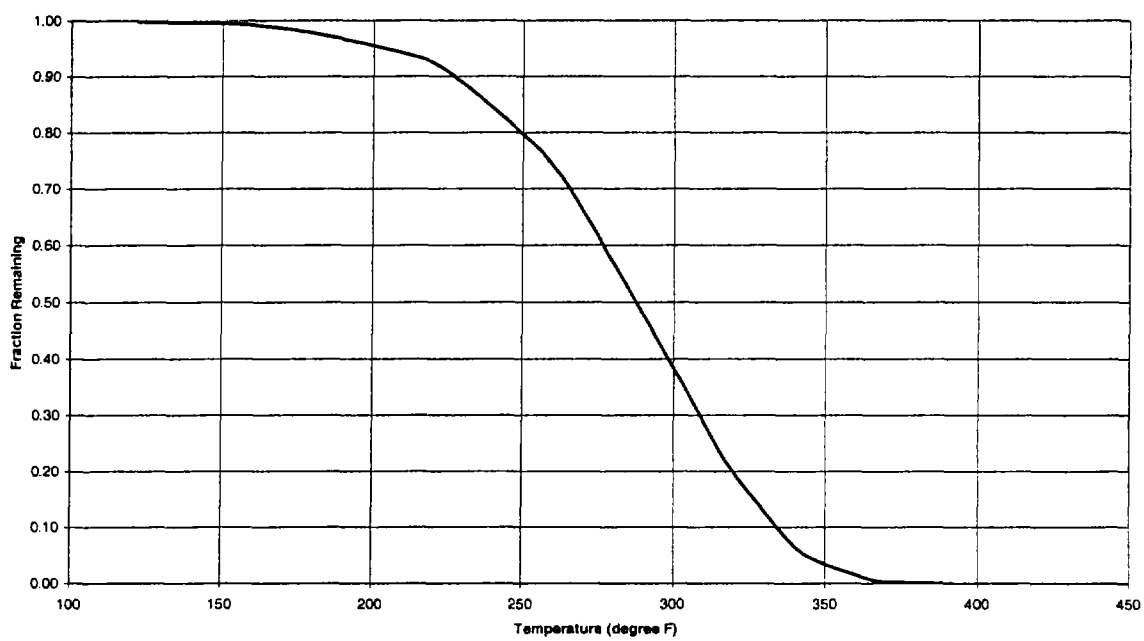
3 PHASE 2 RESULTS

3.1 Decomposition Kinetics of Agents VX, GB and HD at DAL Conditions

Although the root cause of the 12 August, 2002 metal parts furnace DAL unusual occurrence was noncompliance with tray loading procedures, the failure of the ACAMS to detect VX above the Limit of Quantification (LOQ) until the tray was returned to the DAL raised concerns about the effectiveness of the monitoring system in the DAL. During the investigation of the MPF occurrence, a failure mode analysis identified thermal degradation of VX as one explanation for the failure of the monitoring system to detect VX during the initial DAL cycles. In essence the hypothesis was that, even if VX vapors were being generated from the waste, the high temperatures in the DAL caused the VX vapor to be degraded in the 30 seconds (approximate) it took to reach the conversion pad in the ACAMS sampling system. This hypothesis was based on calculations using data in a 1962 Cornell Aeronautical Laboratory report on the thermal decomposition of VX (Cornell 1962). At the time of the investigation this was the only reference available that quantified the gas-phase thermal degradation of VX. A result of the analysis using the Cornell (Cornell 1962) data was a determination that below 259°F (see Figure 3-1) the decomposition rate of VX was sufficiently low to ensure compliance with the PMCD monitoring plan.

Figure 3-1. Fraction of VX Remaining at the CPA vs. Temperature in the DAL



Fraction of VX remaining at the ACAMS as a function of temperature in the DAL Ea = 17.2 kcal/mole, $A = 2.83 \times 10^7$ and a residence time of 30 sec. Based on data from the Cornell report. (Cornell 1962).

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3.1.1 Basis and Assumptions for the Determination of an Effective Monitoring Temperature in the JACADS DAL

1. The purpose of the DAL monitor is to ensure that workers on the cool-down pad are protected in the unlikely event the process fails to completely destroy all agent.

2. To detect potential contamination in the treated waste, the monitoring system must be capable of detecting 75% of the agent vapor concentration generated in the DAL.

3. The gas-phase degradation rate is first-order with an activation energy of 17.2 kcal/mole and a frequency factor of 2.83×10^7 sec^{-1} as calculated from the Cornell data.

3.1.2 Initial Calculations for the JACADS DAL

DAL Physical Parameters

The DAL is located at the discharge end of the metal parts furnace and serves to hold the tray and any residual waste until the material cools. As an added safety measure, an ACAMS verifies that workers will not be exposed to agent vapor when the tray is released. The DAL is a rectangular chamber with interlocked doors on each end. After treatment for the specified holding time in the MPF, the Waste Incineration Container (WIC) is moved into the DAL until the exhaust duct can be monitored for at least one full ACAMS cycle.

The dimensions of the DAL and the gas flow rate are shown below.

Calculation of gas residence time in the DAL:

a. DAL Dimensions: 13.5 feet long by 4 feet wide by 4.5 feet high.
b. DAL Volume = 243 cubic feet
c. Air flow through DAL: 517 cubic feet per minute (design rate)
d. Residence time = 243 cubic feet /517 cubic feet per minute = 0.47 minutes or 28.2 seconds

Calculation of gas residence time in exhaust duct from the DAL to the monitoring probe:

a. Duct is 6 inches in diameter
b. Cross-sectional area is 0.196 square feet
c. Gas velocity = 517 cubic feet per minute / 0.196 square feet = 2638 feet per minute or 44 feet per second
d. Monitoring location is 30 feet from top of DAL.
e. Additional transport time to monitoring probe = 30 feet / 44 feet per second = 0.7 seconds

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The ACAMS pulls an exhaust duct sample from a stainless steel probe inserted approximately 3 inches into the duct. For VX monitoring, the gas is then passed through a CPA impregnated with silver fluoride to convert the VX to a more volatile compound (ethyl methylphosphonofluoridate or G-analog). Conversion of the VX is necessary for transport through 100 feet of heated (120°F) Teflon sampling line to the ACAMS instrument located in the MPF vestibule.

It is desired to locate the CPA as close to the probe tip as possible, but the minimum practical distance is limited by the temperature of the exhaust gas. At higher temperatures the CPA degrades quickly, affecting the conversion efficiency and lifetime of the CPA. The CPA was located 18 inches from the probe tip during the DAL operations prior to the 12 August occurrence.

Calculation of the sample residence time in an ACAMS sampling probe, 18 inches long:

a. The inside diameter of the probe is 1 cm diameter ID with a cross sectional area of 0.79 cm^2
b. ACAMS flowrate is 800 mL/min
c. Length of the probe is 18 inches (45.72 cm)
d. Gas velocity in the probe is 800 cm^3/min/0.79 cm^2 = 1019 cm/min
e. Probe Residence time = 45.72 cm/1019 cm/min = 0.049 min = 2.7 sec.

Therefore, total residence time from the DAL to the CPA = 28.2 seconds + 0.7 seconds + 2.7 seconds = 31.6 seconds (~0.50 min). This assumes no mixing in the DAL i.e. the system acts as a plug flow reactor. This is a conservative worst case assumption.

3.1.3 Thermal Degradation in the DAL

The thermal degradation of chemical agent is assumed to be a first-order reaction that is a function of the temperature and residence time.

The fraction of agent remaining after time t is given by:

$$C/C_o = e^{(-kt)}$$

Where:

C is the concentration after time t
C_o is the initial concentration
k is the rate constant (sec^{-1})
t is the time (sec)

The rate constant is calculated from the Arrhenius equation

$$k = Ae^{(-Ea/RT)}$$

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Where:

A is the frequency factor (sec^{-1})
Ea is the activation energy (kcal/mole)
R is the universal gas constant (1.987 cal deg^{-1} $mole^{-1}$)
T is the temperature in degrees Kelvin.

Activation energies and frequency factors are determined experimentally by plotting the ln(k) versus 1/T. The slope of the linear plot is Ea/R.

$$\ln k = A + Ea/RT$$

3.1.4 Critical Review of the Cornell Report

There is limited literature on the gas-phase decomposition of VX. The initial on-island investigation team had access only to the report on VX decomposition prepared by Cornell Aeronautical Laboratory in 1962. The experimental approach taken by the Cornell researchers may have been appropriate at that time, but the study has weaknesses in both the experimental approach and the interpretation of the data. The state-of-the-art in high temperature thermal stability studies has undergone extensive change over the last 40 years. This has been driven in part by the United States Environmental Protection Agency's (EPA) concern over the thermal stability of principal organic hazardous constituents (POHCs) fed to hazardous waste incinerators. For comparison, some POHCs, with their associated activation energies and pre-exponential factors, are shown in Table 3-1. In addition, modern computational chemistry methods have proven their ability to accurately calculate chemical reaction rates. The investigation team requested that REI review the Cornell study to assess the acceptability of the Cornell data for calculating the decomposition kinetic rate of VX.

REI is under contract to the Army for a Phase II Small Business Innovative Research (SBIR) project to develop component models for the incineration equipment at TOCDF. The goal of that project is to develop computational models using Computational Fluid Dynamics (CFD) and the detailed chemistry and physics of the incineration process.

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Table 3-1. Typical Activation Energies and Pre-Exponential Factors for POHCs

Principle Hazardous Organic Constituent	Dellinger Rank	Absolute Rank	Class	Activation Energy (kcal/mole)	Pre-exponential Rate Expression
Chlorobenzene	19	19	1	84.9	3.2E13
Chloroethane	126	126	4	57.7	6.92E13
Hydrazine (Diamine)	127-130	130	4	55.0	7.94E13
Tetrachloromethane	136-140	138	4	55.0	2.16E12
Trichloroethane(1,1,2)	158-161	161	4	47	2.6E11
Dinitrotoluene (2,4)	168-173	170	4	67.3	2.0E15
Hexachloroethane	202-203	203	5	54	5.01E13
Dimethylhydrazine(1,2)	218-220	220	5	49.6	1.66E13
Dichloropropane(2,2)	224	224	5	54.2	7.94E12
Ethyl Methanesulfonate	261-265	262	5	41.1	1.51E12
Tetranitromethane	284	284	6	38.3	2.0E16

The REI team was contracted to perform an in-depth literature review and analysis of chemical agent thermal stability under the conditions anticipated in the DAL. The conclusions of the REI expert team was that the Cornell data is unreliable due to poorly characterized experimental conditions, calculation errors and huge discrepancies between the reported rates and rates of reaction for similar molecules. The REI report is provided in Appendix A. The REI report noted the following concerns:

1. Poor mixing in the VX inlet tube
2. Failure to obtain steady-state conditions
3. Existence of boundary effects in the reactor
4. Uncertainty in the temperature profile of the reactor
5. Poorly defined residence times due to lack of vapor quenching
6. Errors in data analysis and interpretation.

Additional concerns raised by others with using the Cornell data for this application are:

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- The need to extrapolate from the experimental high temperatures in the study (800-1200°F) and short (1 millisecond) residence times to the lower temperatures in the DAL (300-600°F) and longer residence times (30 sec) anticipated in the DAL.

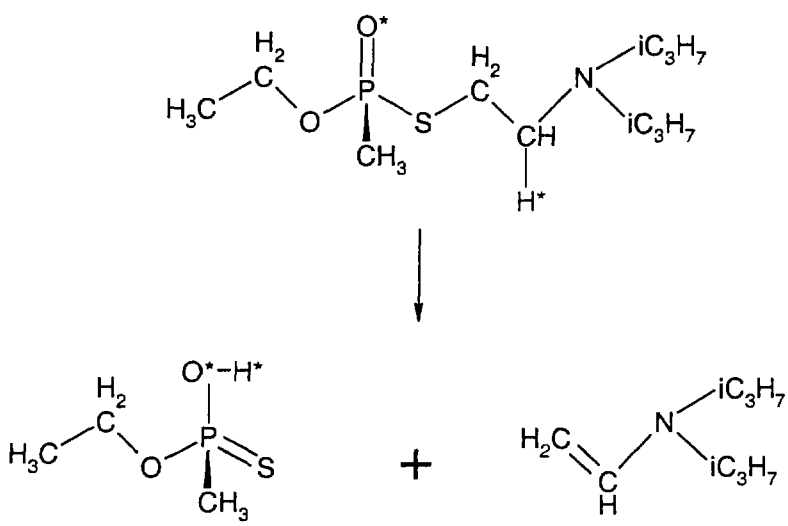
- Errors in calculating the relationship between the kinetic rate constant and temperature will be magnified greatly at the much lower temperatures found in the DAL. Another weakness was the failure to complete an adequate material balance on the VX injected.

3.1.5 Revised Calculations

The REI team reviewed the chemical kinetic mechanisms for organophosphorous compounds and predicted a retro-ene type reaction for VX decomposition. The dominant pathway for VX decomposition is a six-center molecular elimination reaction similar to the findings of Glaude for GB (Glaude 2002). This pathway is illustrated in Figure 3-2.

This type of retro-ene reaction is typical of unimolecular decomposition in molecules this size and is the lowest energy and highest rate unimolecular decomposition path for VX in the gas phase. The result of the REI calculations at the density function level using computational chemistry predicted a minimum Ea of 41.64 kcal/mole for cleaving the carbon – sulfur bond with a predicted frequency factor (*A*) of 5.36 x 10^6 over the temperature range 300-2000°K. Complete details of this study can be found in Appendix A of this document.

Figure 3-2. Dominant Destruction Pathway for VX.



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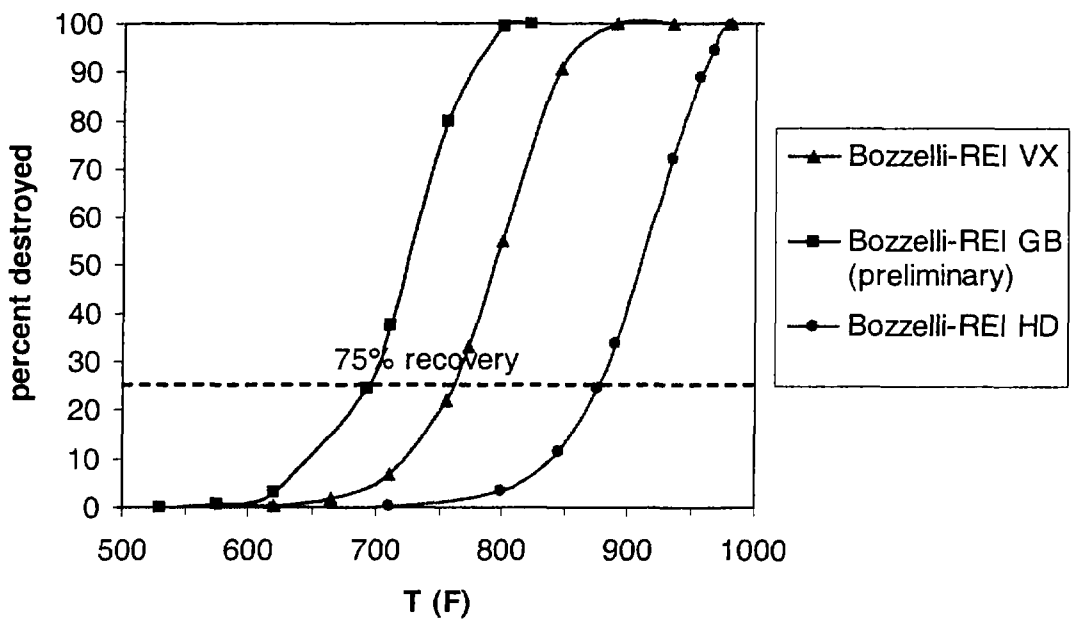
Using recently developed detailed chemical kinetic mechanisms for GB and HD, and a calculated rate for the first steps of the dominant VX decomposition pathways, REI calculated destruction of the three agents for conditions anticipated to be found in the DAL.

REI recalculated agent destruction (using the same calculations as JACADS) as a function of temperature in a fixed-temperature plug flow reactor with a 30-second residence time. Because agent destruction is dominated by unimolecular decomposition, the results are independent of the initial amount of agent assumed for the calculations.

The results are shown in Figure 3-3. Results are also shown using the parameters given by Lapp & Schneider (Cornell 1962) for comparison. As shown on Figure 3-3, effective DAL monitoring can be achieved up to about 760°F for VX, 690°F for GB and 870°F for HD. Effective monitoring is defined by the PMCD Quality Assurance (QA) Plan at 75% recovery of the initial concentration. The dotted line in the figure crosses the curves at the temperature where 75% recovery is predicted to take place.

Figure 3-3. Temperature vs. Percent Agent Destroyed for the DAL at JACADS.



3.1.6 University of Dayton Research Institute

During the investigation into the DAL event, another research study was initiated by the Anniston Project. At the request of the Alabama Department of Environmental Management, the ANCDF team contracted the University of Dayton Research Institute

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(UDRI) to study the thermal stability of the agents GB and VX. Specifically, UDRI was asked to place these compounds on the "Dellinger List" for comparison to the surrogate compounds used during the ANCDF surrogate trial burns. This study was contracted independent of the REI investigation. During review of the draft report by UDRI this study was made available to the authors of this report. An effort was made to jointly evaluate the REI work and the UDRI work with the target being consensus among all investigative teams. UDRI did not posses the computational models that REI had access to, and therefore, identical results were not obtainable. However, the results from UDRI very closely compare to the results obtained by REI. A copy of the UDRI is included here as Appendix G.

3.2 The Heated Discharge Conveyor Bin

The HDC bin is a process discharge point that must be evaluated for effective monitoring. The HDC bin is monitored for the campaign agent prior to bin change-out. At this time, the temperature of the bin enclosure is not known. There are no plants actively processing rockets to allow a determination of the temperature profile in the HDC bin. However, the HDC operates at 1150°F with airflow in the opposite direction of the bin. Although temperatures in the HDC bin are unknown, experience at JACADS would indicate that the temperatures are not expected to be in excess of 600°F. A final recommendation can be made after TOCDF begins processing VX rockets.

3.3 Monitoring Configuration for the Discharge Airlock

At the time of the incident, the DAL was configured with an 18-inch stainless steel sampling probe. For VX, the CPA was positioned approximately 20 inches from the distal (open) end of the probe. It had been placed there to allow the hot gases from the DAL to cool so that the CPA and the Teflon inline filter would remain viable. The 18-inch length of stainless steel tubing served as both a heat exchanger and a sampling probe. Prior to installation of this probe, the conversion pads were observed to darken, become brittle and shed small black particles; the Teflon filters would lose their integrity and allow particulate matter into the ACAMS and DAAMS. With the probe/heat exchanger in place, temperatures at the filter measured 110°F while temperatures at the CPA were estimated at 120°F.

During the initial investigation into the failure to detect VX during the first two cycles in the discharge airlock, JACADS performed a test on the sampling probe to determine if the probe was a contributory factor in the occurrence. This test consisted of sampling a test atmosphere generated by injecting a dilute VX standard in isopropyl alcohol, placed in a polyethylene bag at room temperature. An approximate 1 Time Weighted Average (TWA) VX vapor was developed. This vapor was then monitored using both an ACAMS with a conversion pad placed at the distal end of a used 18-inch stainless steel probe and with a conversion pad placed at the proximal end of the same 18-inch stainless steel probe. The VX concentrations obtained with both configurations were identical.

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This lead the on-island investigative team to conclude that the 18-inch probe was not a contributory factor in the occurrence.

Subsequent to this determination, the PMCD Environmental and Monitoring Office (EMO) and the Department of Health and Human Services (DHHS) questioned the results of this experiment. They cited two concerns:

1. The VX vapor was not pure. The use of a diluted Research Development Test and Evaluation (RDTE) VX standard in isopropyl alcohol may allow the alcohol to act as a carrier solvent in the steel probe.

2. The test was performed at room temperature and not at the elevated temperatures seen in the DAL.

Based on these comments, JACADS was required to reconfigure the probe in the DAL to match the configuration used in the common stack. That is, the probe had to be a stainless steel probe with a Teflon insert that placed the conversion pad at 5 inches from the distal end of the steel probe. Furthermore, JACADS was required to challenge the entire sampling system through the conversion pad.

The original investigation team had recommended that the sampling procedures of the MPF DAL be changed to prohibit monitoring at exhaust gas temperatures exceeding 259°F. Following this requirement, coupled with the recommendation made by EMO that the CPA be placed 5 inches from the distal end of the probe, JACADS reconfigured the DAL ACAMS and began collecting performance data. Immediately following these changes, system performance began to decline. The effort now became one of developing a VX sampling system that was reliable and robust with the CPA near the distal end of the probe.

3.3.1 Modifications to the System

A test sampling system was set up on the DAL exhaust duct and fitted with thermocouples to measure the temperatures of the duct, the CPA, the filter and the gas as it entered the ACAMS/DAAMS. Baseline temperatures under normal operating conditions were measured over two two-day periods, 16-17 October 2002 and 18-19 October 2002, and are shown in Table 3-2. During this time the solenoids controlling the timing and duration of sample flow were actuated when the duct gas cooled to 259°F and simulated sampling was conducted for 28 minutes.

The probe was then repositioned, moving the distal end of the probe from the center of the 6-inch DAL exhaust duct to 0.5 inches from the duct's inner wall. This had the effect of reducing the portion of the probe that is heated in the high temperature gas stream, and moving the CPA slightly farther from the hot duct. This would place the CPA in a cooler environment while preserving the mandated 5 inch distance to the CPA. Engineering calculations indicated that wall effects were negligible at this position.

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Temperature effects of this change were measured over the two-day period 26-27 October 2002. Table 3-2 shows the temperatures resulting from this modification.

Table 3-2. Temperatures Measured During the Probe Study

Dates Measured	T_{duct} °F	T_{CPA}[1] °F	T_{Filt}[2] °F	T_{ACAMS}[3] °F
16-17 Oct. Probe at center of Duct	259	244	112	83
18-19 Oct. Probe at center of duct	259	248	120	87
26-27 Oct. Probe at 0.5 inches into the duct	259	204	112	88

1. Temperature of gas at the CPA
2. Temperature of gas at the Millipore filter
3. Temperature of gas entering the ACAMS

Although continuous monitoring of the CPA temperature showed it did not exceed 210°F at this latter (October 26-27) configuration *during sampling*, the temperature exceeded 300°F during the periods between sampling when no gas was being pulled through the probe. This "heat soak" occurred when a hot WIC was introduced into the DAL and before it was cool enough to sample. During this time, duct gas temperatures approached 600°F. To minimize the heat soak and cool the CPA during the nonsampling periods a stream of cool air was introduced at the proximal end of the probe. This flow was directed toward the duct and over the CPA and sample line during periods when the DAL was not being sampled.

After these changes were made to the monitoring system (Station 67, ACAM-155) on 2 November, the PMCD mandated performance statistics on the DAL ACAMS significantly improved, and performance criteria are now being comfortably met.

3.3.2 Future Operating Conditions of the MPF DAL

Based on preliminary results from REI, it was determined that a monitoring configuration must be developed that is viable at higher temperatures to maximize MPF throughput. Preliminary tests in the JACADS laboratory demonstrated that the CPA is compromised by a continuous 2-hour exposure to temperatures exceeding 200°F. Therefore, a

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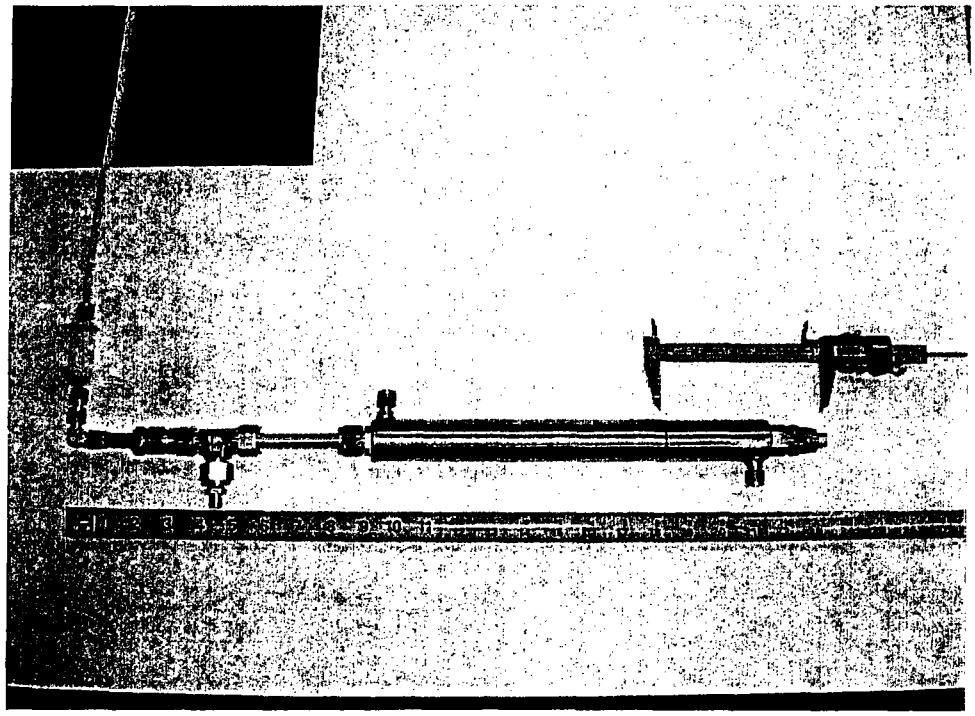
probe configuration that maintains the CPA at 5 inches from the distal end, with the ability to cool the CPA to less than 200°F while sampling substantially hotter air, must be developed.

The JACADS laboratory team fabricated several types of water jackets to enclose the probe and tested them on the test sampling system during periods of MPF operation. The greatest cooling effect was provided by a full-length jacket with both the water inlet and outlet close to the ends of the jacket. Swagelok® unions and fittings were modified to allow the jacket to fit closely to the duct to provide maximum cooling to the probe. (See Figure 3-4 below)

Figure 3-4. Water Jacketed Monitoring Probe



Temperatures were measured by thermocouples installed in the center of the duct, 1-2 feet upstream from the probe penetration; and immediately behind the CPA (<0.125 inches from the pad). Flow of the sample gas was controlled and measured (see Table 3-3). A complete data set can be found in Appendix B of this document.

As demonstrated in Table 3-3 there is a significant statistical difference between the challenge recoveries for the ACAMS station and the DAAMS station. This is believed to be caused by the much higher air-flow of the DAAMS.

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Table 3-3. Typical CPA Challenge Recoveries for the Water-Cooled Test Probes

ACAMS Test Station		DAAMS Test Station	
Flow Rate L/min	Challenge Recovery through the CPA %	Flow Rate L/min	Challenge Recovery through the CPA %
0.8	1.07	4.0	1.04
0.8	1.01	4.0	0.62
0.8	1.02	4.5	0.74
0.8	0.96	4.5	0.84
0.8	0.90	5.0	0.77
0.8	0.97	4.5	0.79
0.8	0.79	3.0	0.95
0.8	0.93	3.0	1.18
0.8	0.82	3.0	1.07
0.8	0.99	3.0	0.84
0.8	0.76	3.0	1.01

To eliminate this problem, JACADS attempted a six-hour CPA change-out and challenge instead of the typical 12-hour CPA change-out and challenge. This change was ineffective, as the CPA was still not passing the post-exposure challenge. Finally, a change in flow rate was tested to bring the DAAMS station into statistical compliance. This change was successful, but will require the site to "split" the DAAMS station into multiple sampling arrays. Based on the data collected to date, the DAAMS station deployed can have a maximum flow rate of 3.0 liters per minute (Lpm).

One concern with the use of this new probe is condensation. Water condensation in the probe would be detrimental to the application of this probe in the DAL for monitoring VX. The DAL exhaust gas that the probe is monitoring originates in the MPF room. This supply air is ultimately derived from the air handling units that supplies the MDB. This air has a dew point of approximately 40-45°F. The final dew point of the air in the DAL is unknown, but it is extremely unlikely that air in-leakage or process input would raise

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the dew point is above 80°F. Therefore, it is essential to keep the temperature inside the probe above this 80°F point.

VX condensation must also be considered. This issue was resolved prior to the start of the VX mine campaign when JACADS was required to configure the VX stack probe with the CPA as close to the distal end as possible. To maintain the blending of dilution air with stack gas, JACADS decided to test a probe with the CPA at 5 inches from the distal end. The probe was then tested in the Buffer Storage Area (BSA) which had a known concentration of VX vapor. The 5-inch probe was tested against a probe that had the CPA at the distal end with no statistical difference between the two probes. This experiment was done at room temperature (~70°F) without VX condensation. Therefore, the water-jacketed probe, if maintained above 80°F, will not have a VX condensation problem.

3.3.3 SwRI Air-Cooled Probe Study

Gas Temperature at 600°F with CPA at 5 Inches Exposed to VX Vapor Puffs

The ability of the DAL probe to transmit the VX vapor challenge atmosphere with gas temperatures of 600°F at the probe tip was first evaluated with the CPA at a distance of 5 inches from the probe tip. At this distance, the temperature of the CPA was within the range of 130 to 150°F (the variability was due to subtle changes in the heating configuration used on the inlet section). Hence, the VX vapor was subjected to a temperature span comparable to the values that the CPA would experience at a distance of 5-inches with the sample flow rate at 0.8 Lpm (ACAMS set up) and at a distance of 18 inches from the DAL probe tip with the ACAMS operating at a flow rate of 6.0 Lpm (DAAMS set up).

The data presented in Table 3-4 was collected with the probe being exposed to "puffs" of VX vapor – the flow of a VX Stock Atmosphere vapor from a Tedlar bag into the probe only occurred during the sampling period. During the analytical phase, only heated breathing air plus room air (ambient temperature) aspirated to accommodate the increased flow rate of the MINICAMS entered the probe. Using this vapor challenge system, the VX concentrations obtained with the CPA at 5 inches and the gas temperature at the probe tip starting at 600°F were compared to the readings obtained with the CPA at the probe tip (0) and the VX challenge atmosphere at a slightly elevated temperature (to minimize condensation).

The data in Table 3-4 indicate that placement of the CPA within the probe at a distance far enough from the probe tip to be compatible with the CPA material (i.e., about 150°F) yielded VX vapor measurements comparable to the reference values obtained at the probe tip at ambient temperatures. Indeed, the MINICAMS VX measurements remained stable even as the gas temperature at the probe tip approached 700°F.

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Table 3-4. Probe Performance with CPA at 5 Inches for VX Vapor Puffs

Time	CPA Site	Gas Temperature at Probe Tip, °F	MINICAMS TWA
Liquid Challenge of CPA = 0.99 TWA			
1113	0	108	0.06*
1121	0	108	0.10*
1129	0	118	3.42
1137	0	121	4.23
1145	0	122	3.94
1153	0	123	3.99
1329	5	590	0.02*
1337	5	585	0.08*
1345	5	609	0.00*
1353	5	608	4.86
1401	5	610	0.19*
1409	5	613	5.17
1417	5	613	0.21*
1425	5	616	5.35
1433	5	617	0.22*
1441	5	618	5.53
1449	5	620	0.23*
1457	5	631	0.08*
1505	5	635	5.52
1513	5	646	0.20*
1521	5	651	5.63
1529	5	667	0.21*
1537	5	680	5.69
1545	5	690	0.23*
1553	5	695	5.57
1601	5	698	0.22*
1609	5	703	5.35
Liquid Challenge of CPA = 1.00 TWA			

* Zero baseline (flow of VX stopped)

The VX concentrations at the 5-inch probe location were somewhat elevated compared to the reference values obtained at the probe tip at ambient temperatures. Two phenomena were believed responsible for this result. First, although the gas temperature within the inlet section was raised slightly to minimize condensation during

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the measurements collected at the probe tip, it appears that some of the VX vapor did in fact plate onto the surfaces of the inlet section causing the ambient reference values to be depressed to some extent. Secondly, as can be seen from the gradual gradient of the zero baseline during the course of the "puff" tests, residual VX appears to be accumulating either within the probe or the inlet section. Interpretations of the chromatographs produced by the MINICAMS during the tests indicate the possibility of potential decomposition products also accumulating within the probe or the inlet section and the presence of those organics being related to the appearance of the VX residue. Simple swabbing of the probe interior with dichloromethane and/or isopropanol readily returned the zero baseline value to a nominal level (i.e., less than 0.1 TWA). As stated in the report, our experience throughout the testing program indicates that periodic cleaning of the DAL probe enhances its performance.

To obtain a truer representation of the transmission efficiency achievable by the DAL probe, the use of a gas-tight syringe to challenge the probe with a puff of VX vapor was adopted. With the CPA at 5 inches from the probe tip, 2 or 3 mL of concentrated VX vapor was injected into the heated air stream at the probe tip. The injection of the VX vapor spanned a duration of only a few seconds shortly after the commencement of the sampling phase (4 minutes). As the reference for calculating the transmission efficiency under these conditions, VX vapor spikes were introduced directly onto the CPA through a 1/16th inch hole drilled through the probe at the 5-inch location.

The results from two separate test dates are presented in Table 3-5 and the first series depicted in Figure 3-5. The results of the gas-tight syringe injections supported the earlier findings using the longer-duration puffs of VX Stock Atmosphere 2 from the Tedlar bag. The gas-tight syringe challenge also represents the worst-case detection scenario for the probe – the accurate measurement of a short-lived puff of VX vapor within the DAL. At a flow rate of 0.8 Lpm and within the analytical limits of the tests, essentially 100 percent of the VX vapor at the probe tip in an air stream heated to 600°F can be detected by the CPA located at 5 inches from the probe tip.

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Table 3-5. Probe Performance for Gas-Tight Syringe Injections with CPA at 5 Inches

Time	MINICAMS TWA	Gas Temperature @ Probe Tip, °F	Gas Temperature @ 5 Inches, °F	Injection Location
1339	0.06	576	135	N/A, zero baseline
1345	2.57	580	132	Probe tip
1351	2.96	589	132	Probe tip
1357	2.45	592	135	Pad at 5-inches
1403	2.47	590	135	Pad at 5-inches
1409	0.07	590	135	N/A, zero baseline
1415	0.03	592	137	N/A, zero baseline
1421	2.42	592	137	Probe tip
1427	2.39	595	125	Pad at 5-inches
1433	2.33	598	126	Pad at 5-inches
1439	2.61	596	125	Probe tip
Average at Pad = 2.41 TWA; Average at Probe Tip = 2.64 TWA Transmission Efficiency = 110 percent				
Liquid Challenge of CPA = 0.92 TWA				
1531	0.14	597	168	N/A, zero baseline
1538	3.06	600	161	Pad at 5-inches
1545	3.21	606	160	Probe tip
Transmission Efficiency = 3.21/3.06 = 105 percent				
1552	0.00	604	-	N/A, zero baseline
1559	2.67	604	155	Pad at 5-inches
1606	2.66	603	160	Probe tip
1613	0.06	602	-	N/A, zero baseline
Transmission efficiency = 2.66/2.67 = 100 percent				
Liquid challenge of CPA = 0.98 TWA				

Gas Temperature at 600°F with CPA at 18 Inches Exposed to VX Vapor Puffs

The gas-tight syringe injection method was selected to document the transmission efficiency of VX vapor at 600°F to a distance of 18 inches from the probe tip since this procedure proved to be the most reliable. As was stated for the 5-inch location tests, this procedure represents the worst-case scenario for challenging the capability of the CPA to accurately detect VX vapors when located 18 inches from the probe tip. The gas temperatures at the 5- and 18-inch locations in the probe were carefully controlled using heating tape and insulation to mimic the temperatures that have been documented in the DAL probe with the ACAMS (i.e., 380 and 160°F, respectively) of the CPA (Figure 3-6). Table 3-6 presents the data from three separate VX puff tests performed on two different days. Figure 3-6 graphically shows the results from the third series of tests.

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The results indicate that at a flow rate of 0.8 Lpm, the transmission efficiency for VX vapor between the probe tip and the CPA at the 18-inch location is approximately 60 percent.

Figure 3-5. Probe Performance for Gas-Tight Syringe Injections with CPA at 5 Inches

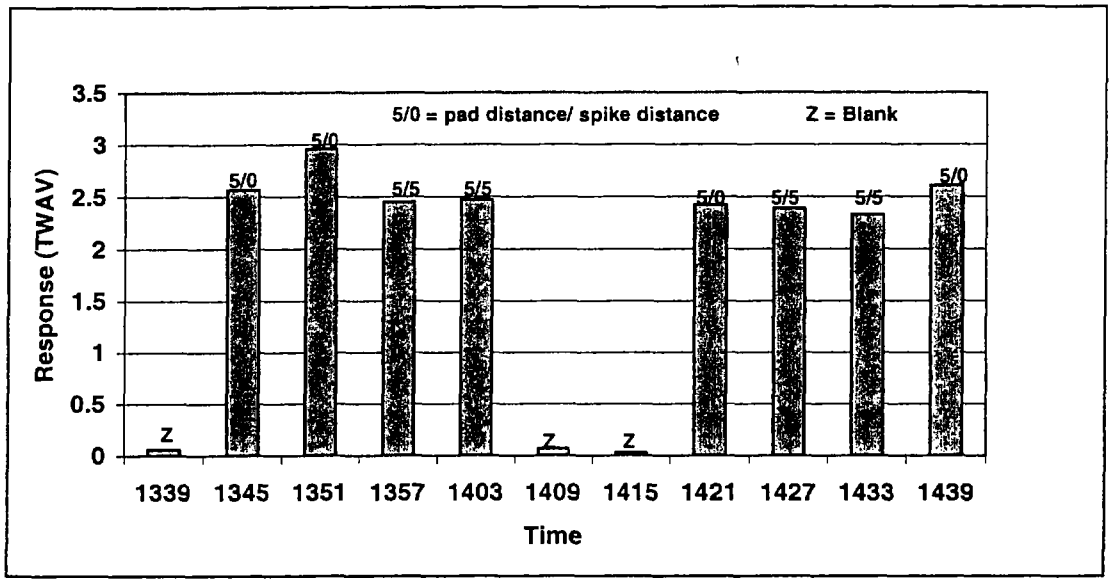

Table 3-6. Probe Performance for Gas-Tight Syringe Injections with CPA at 18 Inches

Time	MINICAMS TWA	Gas Temperatures, °F			Injection Location
		Probe Tip	@ 5 Inches	@ 18 Inches	
1049	0.07	592	390	148	N/A, zero baseline
1056	2.34	595	388	148	Pad at 18-inches
1103	2.35	595	394	150	Pad at 18-inches
1110	0.07	597	396	155	N/A, zero baseline
1117	1.23	594	401	156	Probe tip
1124	1.11	597	405	154	Probe tip
1131	2.00	593	393	154	Pad at 18-inches
1138	0.06	592	-	-	N/A, zero baseline
1145	1.31	593	388	148	Probe tip
1152	1.91	594	394	149	Pad at 18-inches
1159	1.39	592	398	153	Probe tip
1206	2.00	592	403	155	Pad at 18-inches
1213	1.12	595	390	151	Probe tip
Average at Pad = 2.12 TWA; Average at Probe Tip = 1.23 TWA; Efficiency = 58 percent					
1412	2.00	597	362	140	Pad at 18-inches
1419	2.03	598	371	140	Pad at 18-inches
1426	1.31	595	373	144	Probe tip
1433	1.22	594	373	147	Probe tip

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1440	0.05	594	370	148	N/A, zero baseline
1447	1.85	594	365	144	Pad at 18-inches
1454	1.30	597	365	145	Probe tip
1501	1.89	603	368	150	Pad at 18-inches
1508	1.25	602	374	149	Probe tip
1515	2.00	602	369	150	Pad at 18-inches
1522	0.05	602	371	149	N/A, zero baseline
1529	1.11	600	370	145	Probe tip
1536	1.90	601	376	151	Pad at 18-inches
1543	1.20	603	380	154	Probe tip
1550	1.27	603	380	151	Probe tip
1557	1.88	605	382	150	Pad at 18-inches
1604	0.06	606	-	-	N/A, zero baseline
Average at Pad = 1.94 TWA; Average at Probe Tip = 1.24 TWA; Efficiency = 64 percent					
Liquid challenge of CPA = 0.93 TWA					
1311	0.00	618	-	-	N/A, zero baseline
1318	2.29	617	389	147	Pad at 18-inches
1325	2.53	616	-	-	Pad at 18-inches
1332	1.73	617	-	-	Probe tip
1339	1.49	618	394	155	Probe tip
1346	0.03	618	-	-	N/A, zero baseline
1353	2.26	619	385	152	Pad at 18-inches
1400	0.03	618	-	-	N/A, zero baseline
1407	1.33	616	395	157	Probe tip
1414	0.03	618	-	-	N/A, zero baseline
1421	1.27	618	397	160	Probe tip
1428	0.03	616	-	-	N/A, zero baseline
1435	2.27	617	403	159	Pad at 18-inches
Average at Pad = 2.34 TWA; Average at Probe Tip = 1.46 TWA; Efficiency = 62 percent					
Liquid challenge of CPA = 0.98 TWA					

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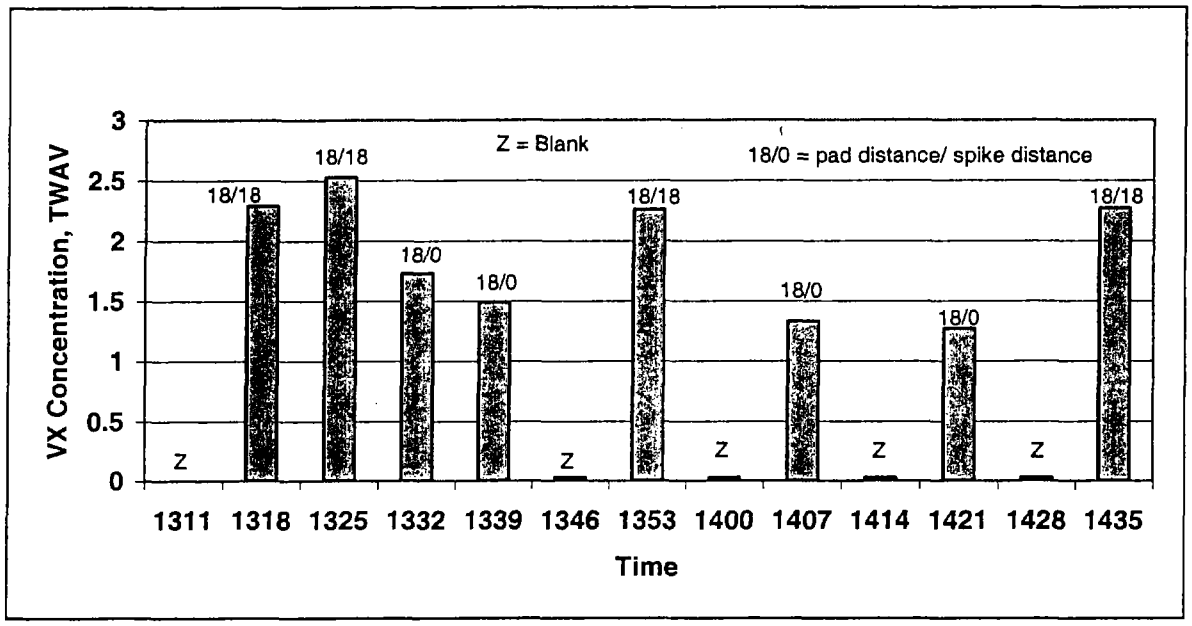
Figure 3-6. Probe Performance for Gas-Tight Syringe Injections with CPA at 18 Inches



Conclusion

The following summarizes the results presented above.

1. Under continuous exposure to a VX vapor atmosphere at ambient temperature the VX is transported through the 18-inch stainless steel probe with high efficiency. A slight increase in the response was observed at both the inlet and with time indicating the there was some acclimation of the surface of the feed system.

2. Buildup of organics in the probe over time inhibited transport, therefore the probes should be "swabbed" with isopropanol when the CPAs are challenged to remove any particulate or residual organics or decomposition products

3. With "puff" exposures in 600°F air at the inlet, with a 0.8 Lpm flow rate, placing the CPA at 5 inches (~150°F) gave vapor measurements comparable to the reference values obtained at ambient values with the CPA at the probe tip. This trend was duplicated up to 703°F.

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4. With "puff" exposures at 600°F at the inlet, with a 0.8 Lpm flow rate, placing the CPA at 5 inches gave vapor measurements comparable to the reference values obtained by injecting the puff onto the CPA at 5 inches at 150°F.

5. With "puff" exposures at 600°F at the inlet, with a 0.8 Lpm flow rate, placing the CPA at 18 inches gave vapor measurements approximately 60% of the reference values obtained by injecting the puff onto the CPA at 18 inches at 150°F.

Based on the REI analysis and the results summarized above it can be concluded that the ACAMS monitoring of the DAL at 600°F would be effective in detecting VX in the discharge duct. The recovery at 18 inches was less than the desired 75% recovery, however, this is conservative because the flow rate in these tests was only 0.8 Lpm compared to the 6 Lpm used at JACADS. This would give a residence time in the probe of 1/7.5th of the 0.8 Lpm used in these tests and would even be less than the residence time at 5 inches (0.36 sec versus 0.75 sec). Based on this it is predicted that recovery for the DAAMS confirmation would be greater than 75% at 6000 Lpm at a point in the probe where the temperature is approximately 150–160°F.

3.4 Evaluation of Analytical Techniques for VX Analysis

3.4.1 Potential for False Positives

At JACADS the method of monitoring for VX actually quantifies VX as the G-analog (ethyl methylphosphonofluoridate). This is necessary since VX is known to be difficult to transport through long sampling lines. The same indirect method is used to analyze extracts of solids and liquids to verify that waste samples are below the 20 parts per billion (ppb) waste control limit (WCL). Whereas the analytical results for VX during the incident at JACADS were unambiguous using the G-analog analytical method, the tendency of VX to hydrolyze in the presence of water, caustic and bleach, raised questions why it would still exist in the sludge after 10 years. The hydrolysis of VX is well documented (MacNaughton1994).

The JACADS laboratory sample extraction procedure for VX uses chloroform to concentrate the VX in a liquid sample so that it can be injected onto a DAAMS tube. Although this indirect method is very sensitive and conservative, previous investigations have shown that under the right conditions it is possible to form VX from decomposition products at high temperature such as at the DAAMS desorber or the inlet of the Gas Chromatograph (GC), if chloroform is used as an extraction solvent (IITRI 1997; Beaudry 1996; Jakubowski). It has also been reported in the PMCD Alternative Technology and Approaches program that when analyzing VX in the presence of high concentrations of VX hydrolysis products there is a high probability of false positives unless very selective GC/MS/MS techniques are used to directly analyze VX, not the G-analog.

Although the methods used at JACADS gave unambiguous confirmation of VX in the residue according to the G-analog method required by PMCD and the RCRA permit,

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there is still a possibility that these results were caused by interferants. Of particular concern would be compounds that form the G-analog upon reaction with the CPA.

3.4.2 Multilaboratory Analysis of Tray 153 Residue

The concerns raised above initiated further testing to determine if an interferant was the cause of the positive VX analyses by the G-analog method. The residue from tray 153 was shipped to the Illinois Institute of Technology Research Institute (IITRI) in Chicago, Illinois and Southwest Research Institute (SwRI), in San Antonio, Texas. The analyses were performed to definitively determine if there was chemical agent VX in the residue using more specific analytical methods than available in the JACADS laboratory (Brewer 1).

A sample of the residue collected on 13 August 2002, one day after the MPF unusual occurrence, was well mixed in the JACADS laboratory. This sample was split into three subsamples for analysis by multiple laboratories. Analysis of the sample by the JACADS laboratory using the G-analog method gave VX concentrations of 210 WCL (4.2 μg/g). The samples were shipped from JACADS to SwRI and IITRI. Additionally, an aliquot was shipped to the Dugway Proving Grounds for analysis at the Idaho National Engineering and Environmental Laboratory (INEEL) under contract to the PM Non-Stockpile. The samples were sent by air on 3 October, 2002.

Multiple laboratories and different analytical methods were used to obtain a higher level of confidence in the results. The analyses were conducted by SwRI using liquid chromatography/mass spectroscopy (LC/MS) and gas chromatography/electron impact/mass spectroscopy (GC/EI/MS). IITRI used gas chromatography/ion trap/mass spectroscopy (GC/IT/MS) with chemical ionization (CI) and INEEL used direct ion trap secondary ion mass spectroscopy.

3.4.3 Analytical Results

Table 3-7 summarizes the sample results from the confirmation testing. A full report of these analyses can be found in Appendix E. All methods confirmed VX in the residue sample consistent with the GC/FPD/MS results from the JACADS laboratory. The VX concentrations varied from 2.3 μg/g to 4.2 μg/g. Formal results have not yet been received from INEEL.

Nuclear magnetic resonance (NMR) spectra were taken to further investigate the presence of VX in the tray 153 residue sample. Four grams of residue were extracted with 20 mL of 90% dichloromethane/10% isopropanol and reduced to dryness. This extract was reconstituted in one milliliter of deuterated chloroform. The sample was transferred to an NMR sample tube for analysis. The NMR spectrum confirmed the presence of VX.

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Table 3-7. Analytical Results from Analysis of Tray 153 Residue

Laboratory and Method	Average Sample Concentration ppm	Relative Standard Deviation (%)	Matrix Spike Recovery (%)	Matrix Spike Duplicate Recovery (%)
JACADS (Derivative)	4.2	14	88	83
IITRI GC/IT/MS (Undiluted)	4.1	4.8*	110	120
IITRI GC/IT/MS (1:10)	3.5	11	NA	NA
SwRI GC/MS (1:10)	2.3	16	88	63
SwRI-LC/MS (Undiluted)	2.4	9.7	86	94

* – Duplicate analyses expressed as relative percent difference
NA – Not Analyzed

3.4.4 Non-chlorinated Extraction Results

As noted above, the presence of chlorinated solvents has been implicated in the formation of VX in some matrices. To avoid any possible issues with chlorinated solvents an additional set of extractions was done with non-chlorinated solvents (Brewer 2). A number of non-chlorinated solvents were evaluated including water (at pH = 4), hexane, hexane/diethyl ether, toluene, acetonitrile, ethyl acetate, and acetone. The best results were obtained with acetone so this solvent was selected to obtain samples for GC/MSD and LC/MS.

Acetone Extractions

Approximately two grams of residue were extracted using two aliquots of acetone, 4 mL each. The samples and acetone were rotary extracted for 1 hour, centrifuged, and filtered. Both acetone fractions were combined. These extracts were analyzed using GC/FPD by the G-analog technique and directly as VX by GC/MS. For LC/MS, an aliquot was diluted into the appropriate mobile phase for direct injection.

To reach the ~20 ppm detection limit of the NMR, samples were generated by combining the extracts of approximately 15 grams of residue. The fractions were combined and concentrated to a one-milliliter final volume. During the concentration step (at approximately 4 milliliter total volume) a white precipitate formed. The sample was filtered through glass wool, rinsed with more acetone and then reduced to one

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milliliter final volume. Two phases were evident, an aqueous lower phase and a dark organic upper phase. The entire final volume was placed into an NMR tube for analysis. After an initial unspiked sample run, the sample was spiked with dimethyl methylphosphonate (DMMP) as an internal standard and re-analyzed to determine concentration. The sample was also analyzed with a VX spike to verify the peak was VX.

Results with Non-chlorinated Solvent

Results from the GC/MSD and LC/MS analysis of the acetone extractions are provided in Table 3-8 below.

Table 3-8. Acetone Extraction Results of Tray 153 Residue

Results from Re-analysis of Tray 153 Sample Samples Assayed Without Using Chlorinated Solvents								
Sample	FPD-analog acetone		GCMSD - direct VX acetone (ion 139)		(ion 167)		LCMS - direct VX water	
	(mg/Kg)		(mg/Kg)				(mg/Kg)	
1	1.33		1.67		4.43		1.59	
2	0.87		1.59		3.49		1.52	
3	1.38		2.1		4.68		1.59	
average	1.2		1.8				1.6	
%RSD	24		15				2.6	
		% recovery		% recovery		% recovery		% recovery
MS	10.13	82	17.1	140	22.1	163	9.5	72
MSD	10.43	83	15.9	127	19.02	133	12.1	96

The samples were analyzed by ^{31}P NMR using a Varian VXR-300 spectrometer. Data were acquired in a gated decoupling mode with long (overnight) acquisitions to give maximum sensitivity. An external phosphoric acid reference was used to assign the chemical shifts of the signals. An external standard was prepared in d_6-acetone at ~30 ppm that allowed a rough estimate of the VX level in the extract. This equates to approximately 1 ppm of VX in the residue.

3.4.5 Conclusions

The purpose of this analytical effort was to confirm or refute the presence of VX in the residue from the sludge that had been improperly loaded in tray 153. Based on these multi-laboratory/multi-method results it is reasonable to conclude that there is VX in the residue. This result is consistent with the ACAMS alarms and the confirmed DAAMS tubes that were collected after the tray had been returned to the DAL. In the absence of other results, these results do not support the hypothesis that the positive responses were caused by an interferant.

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3.5 TOCDF Study of 155 SETH Munitions Processing

3.5.1 Introduction

Following the MPF Discharge Airlock Agent Sampling Incident at Johnston Island, concerns have been expressed that high air temperatures in the MPF DAL do not allow for proper sampling of Chemical Agents. Temperatures in the Discharge Airlock and at sample point locations in the air duct exiting the Discharge Airlock were unknown prior to this test. To properly determine these temperatures, it was necessary to install temporary thermocouples capable of measuring the temperatures in these areas of concern.

Three munition trays containing 48 SETH projectiles (155mm) were used for this test. Trays with 48 projectiles (155mm) presented the worst case scenario for cool down rates in the Discharge Airlock at TOCDF because this load has the highest metal mass (6894 lbs) and is processed at the highest temperatures (1600°F) in the MPF Primary Chamber. The 155mm projectile tray has 12 rows of projectiles with 4 projectiles on each row. The Test Trays were processed through Primary Chamber of the Metal Parts Furnace at projectile processing temperatures (1600°F) prior to entering the Discharge Airlock. Temperature data was then gathered while hot trays were in the Discharge Airlock.

3.5.2 Materials and Methods

A hand held optical pyrometer was used to measure DAL skin temperatures. One infrared sensor and a total of 13 type K thermocouples were used to measure temperatures within the DAL and DAL Exhaust Duct. The thermocouples and infrared sensor were wired into a Data Logger and the data was downloaded to Lap Top Computer. The locations of the thermocouples and infrared sensor are shown on attached Sketch MPF-TC-124-1 Rev 1.

3.5.3 Results and Discussion

The Test was started on 1-8-03 and completed on 1-10-03. Thermocouples #4 and #5 were of the greatest interest because they indicated the highest temperatures and required the longest time to cool to 259°F. Thermocouple #4 was located near the DAL exit door approximately 2 inches above the first row of projectiles. Thermocouple #5 hung down between the projectiles, approximately 5 inches from the top of the projectiles near the 8th row of projectiles. Thermocouple #1 was inserted inside the DAL exhaust duct at the VX sampling probe location, approximately 7 ½ feet up the DAL exhaust duct.

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Tray # 639 was the first tray to enter the DAL and was in the primary chamber for 5 hrs and 45min due to conveyor malfunctions. It took thermocouple #1, 1hr 14min to cool to 600°F and 5 hrs 21 min to cool to 259°F. It took thermocouple #4, 3 hrs 56 min to cool to 600°F and 8 hrs 29 min to cool to 259°F. It took thermocouple #5, 3 hrs 52 min to cool to 600°F and 8 hrs 12 min to cool to 259°F. Thermocouple #12 failed 2 hrs and 10 min after tray #639 entered the DAL and was repaired after the tray exited the DAL.

Tray # 651 was the second tray to enter the DAL and was in the primary chamber for 1 hr 15 min. Thermocouple # 1 reached a maximum temperature of 584.3°F and took 4 hrs 37 min to cool to 259°F. It took thermocouple #4, 3 hrs 6 min to cool to 600°F and 7 hrs 48 min to cool to 259°F. It took thermocouple #5, 3 hrs 3 min to cool to 600°F and 7 hrs 38 min to cool to 259°F. Thermocouple #13 failed, 47 min after Tray #651 entered the DAL.

Tray # 635 was the third tray to enter the DAL and was in the primary chamber for 9 hr 37min prior to entering the DAL. Tray #635 entered zone one just after tray #651 entered zone two and remained in the primary chamber until tray # 651 was able to exit the DAL. It took thermocouple #1, 1 hr 26 min to cool to 600°F and 5 hrs 28 min to cool to 259°F. It took thermocouple #4, 4 hrs 2 min to cool to 600°F and 8 hrs 32 min to cool to 259°F. It took thermocouple #5, 4 hrs 0 min to cool to 600°F and 8 hrs 11 min to cool to 259°F.

An infrared camera was used to capture thermal images of all three test trays when they exited the Discharge Airlock. Temperatures of projectiles in the center of the trays were approximately 50°F higher than those projectiles on the outer perimeter of the tray. See attached Infrared photograph of test tray.

Airflow measurements were taken on the inlet air duct to the DAL using a hot wire anemometer for trays #639 and #651. The average inlet airflow for tray #639 was 214.7 SCFM. Two sets of measurements were taken for tray # 651, their averages were 221.2 SCFM and 208.8 SCFM.

The Skin temperatures of the DAL were taken while tray #651 was cooling within the DAL. The highest temperature recorded on the roof of the DAL was 126°F, for the South Wall 131°F was the highest, and for the North Wall 130°F was the highest. The highest ambient temperature recorded was 60°F.

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4 COST/SCHEDULE BENEFIT ANALYSIS

Table 4-1 provides a gross estimate of $1.8 billion to the program impact for taking no action. At TOCDF, the impact to the schedule from monitoring at 600°F has been to effectively add three or more hours to each processed tray. This three-hour delay per tray will be similar at each of the other sites. Assuming that the MPF is the limiting factor for production during the projectile campaigns, the impact in hours can be calculated. Based on an estimated $300,000 per day facility operating costs, the cumulative impact to cost and schedule can be derived.

The benefit of any changes to the DAL process have not yet been fully determined. Once all of the testing is complete and data are available, a more realistic schedule analysis will be performed to calculate the cost and schedule benefits to the individual site schedules.

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Table 4-1. Cost/Schedule Benefit Analysis

Discharge Airlock - Calculation of Cost Impact to Stockpile Program							
Based on 3 hour impact to Cool Down Cycle as demonstrated by TOCDF							
TOCDF	Agent Campaign	Munitions Campaign	Number of Munitions	Munitions per Tray	Total Number of Trays	Total Cost Impact
	VX	One Ton Containers	638	1	638	
	VX	155mm Energetic Projectiles	53,216	48	1109	
	VX	Spray Tanks	862	1	862	
	HD	One Ton Containers	6,398	1	6398	
	HD	155mm Energetic Projectiles	41,100	48	856	
	HD	155mm Non-Energetic Projectiles	13,563	48	283	
	HD	4.2 Inch Mortars	63,568	96	662	
						$675,477,865
ANCDF						
	GB	105mm Cartridges	74,014	96	771	
	GB	105mm Projectiles	26	96	0.27	
	GB	155mm Projectiles	9,600	48	200	
	GB	8-inch Projectiles	16,026	27	594	
	VX	155mm Projectiles	139,581	48	2908	
	HT	4.2 Inch Cartridges	183,552	96	1912	
	HD	4.2 Inch Cartridges	75,360	96	785	
	HD	105mm Cartridges	23,064	96	240	
	HD	155mm Projectiles	17,643	48	368	
	HD	One Ton Containers	108	1	108	
						$492,847,222

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UMCDF									
	GB		155 mm Projectile			47,406	48	988	
	GB		8-Inch Projectile			14,246	27	528	
	GB		MK-94 (500lb Bomb)			27	2	14	
	GB		MC-1 (750 lb Bomb)			2,418	2	1209	
	VX		155mm Projectiles			32,313	48	673	
	VX		8-Inch projectiles			3,752	27	139	
	VX		Spray Tanks			156	1	156	
	HD		One Ton Container			2,635	1	2635	
									$396,306,568
PBCDF									
	HT		One Ton Containers			3591	1	3591	
	HD		One Ton Containers			107	1	107	
									$231,125,000
			Total Cost Impact					**$1,800,000,000**	

Assumptions:
1) Additional cooling time required per tray = 3.0 hours
2) MPF Furnace Availability is 60% = 14.4 hr/day
3) MPF Furnace is a limiting factor

Based on Table 4-1, the total number of stockpile munitions to process is 28,733. Multiply 28,733 by the average 3 hours for cooling time per tray yields 86,197 added hours. Those 86,197 added hours is divided by the 14.4 hours per day when the MPF furnace is available, which would be an additional 5,986 days to process the munitions at all sites. Based on the $300,000 per day cost of operations at each of these facilities, this would yield a cost savings of just under $ 1.8 billion.

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5 CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The predicted temperature for effective monitoring of VX using the REI analysis for JACADS is 760°F. Data from the post-12 August investigation report indicate that the temperature in the discharge airlock never reached this temperature and consequently the thermal degradation of VX at elevated temperatures could not be a contributory factor in the failure of the monitoring system to detect VX vapor during the first two ACAMS cycles.

Encapsulation of the chemical agent in the cool center of the residue, the other hypothesis identified during the initial investigation, is the most probable explanation for the failure of the monitoring system to detect VX during the first two monitoring cycles. There is abundant physical evidence to conclude that the material did not reach 1000 °F (moisture, uncombusted plastic and burlap). The agent in the center of this mass was therefore not subjected to sufficient heat to destroy it, and was physically insulated from the heat in the metal parts furnace. When the tray containing this residue was placed in the discharge airlock, the evolution of VX from the residue may have been impaired by the charred surface layer until a breach was formed. It is possible that the thermal gradient around the tray provided sufficient heat to thermally decompose the VX prior to entrainment in the cooling air stream and therefore prevented the VX from being detected by the ACAMS. However, the rate of gas flow in this region would only expose the agent to this temperature for a very limited period of time.

The ability to transmit VX through a hot 18-inch stainless steel sampling probe at 600ºF has been established. The study was completed in late February by SwRI and indicated that VX can be successfully transmitted through a probe with minimal loss. This finding provides further confirmation that the monitoring configuration was not a root cause for the monitoring problems associated with the August 12 event. Furthermore, this study has provided a configuration for monitoring at the JACADS and TOCDF sites for the DAL.

A comparison of the water-cooled probe configuration to the air-cooled probe configuration by the SwRI study team has concluded that the water-cooled probe is too cool under the DAL monitoring conditions at JACADS and TOCDF. The study has shown that the transmission of VX is impeded at low temperatures and that an ideal temperature range is about 115°F to 140°F. Under these conditions, VX can be quantitatively transferred through a stainless steel probe, and the CPA can be expected to remain viable for 12 or more hours.

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5.2 Recommendations

5.2.1 JACADS

JACADS should continue using the existing air-cooled probe with the CPA at 5-inches from the distal end for ACAMS monitoring; and the CPA at approximately 18-inches from the distal end for DAAMS monitoring at 3 Lpm. JACADS can raise the monitoring temperature to 600°F. This temperature is a conservative temperature that will extend the life of the CPA, and is below the maximum temperature of 760 °F for VX as calculated by REI. This will provide JACADS with a viable monitoring approach as well as needed schedule relief to complete closure in a timely and safe manner.

5.2.2 TOCDF

TOCDF should immediately configure the DAL with the air-jacketed probe designed at JACADS. TOCDF should place the CPA at the same locations in the probe that JACADS has established. TOCDF should reprogram the logic in the monitoring system for the DAL, to initiate monitoring when the DAL reaches 600°F. To take the most conservative approach for TOCDF, since TOCDF is still processing significant quantities of liquid chemical agent, while JACADS is processing only secondary waste and decommissioning materials, it is recommended that TOCDF monitor the temperature of the DAL itself and not the exhaust duct. The DAL should achieve a maximum temperature of 600°F prior to initiating sampling. Measuring the temperature at this location will ensure that the temperature at the monitoring point is at or below the 600°F limitation. This system will provide TOCDF with a safe and effective start-up condition for the VX campaign.

5.2.3 Long Term Solution

The Risk Team is currently evaluating engineering options to modify or add on to the existing DAL to achieve cooling in a shorter time that what is possible with the current configuration. This is being handled as a Phase 3 effort under RIM 57, and the results will be presented in separate documentation.

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6 PROPOSED PATH FORWARD FOR COMPLETION OF PHASE 2

6.1 Thermal Dosimeters

CR&E is exploring the use of some type of thermal docimetry to measure temperature in the waste while processing sludges. Although the treatment conditions for sludge were determined experimentally and verified through sufficient testing of residues at JACADS, the use of thermal dosimeters will provide another layer of confidence that the sludge material has been heated to 1000°F for 15 minutes.

6.2 ROM Estimate of Resources/Costs Required to Implement Long Term Risk Mitigating Recommendations

Preliminary engineering analysis has been initiated. The engineering estimate to complete conceptual design and detailed design of an anticipated change to facilitate more rapid cooling for TOCDF and the other CONUS sites is approximately $1 million. Procurement and Construction costs will depend on the option selected and are unknown at this time.

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7 CONTRACT ASSESSMENT

Cannot Be Accommodated In Technical Baseline

The issues identified in the RIM 57 investigation are not included in the technical baseline. The monitoring program and configuration is not included in the scope of either WDC or EG&G. The concepts, locations and strategies for monitoring are developed by PMCD for implementation by the systems contractors. Therefore, these issues, associated changes and costs are not covered by the technical baseline at any site.

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The 18-inch Probe

The 18-inch probe study was completed and a recommendation is made to provide JACADS and Tooele Chemical Agent Disposal Facility (TOCDF) with a viable and effective monitoring program for the discharge airlock and the heated discharge conveyor bin enclosure. JACADS should continue using the existing air-cooled probe with the Conversion Pad Assembly (CPA) at 5-inches from the distal end for ACAMS monitoring; and the CPA at approximately 18-inches from the distal end for DAAMS monitoring at 3 liter per minute (Lpm). JACADS can raise the monitoring temperature to 600°F. This temperature is a conservative temperature that will extend the life of the CPA, and is below the maximum temperature of 760°F for VX as calculated by REI. This will provide JACADS with a viable monitoring approach as well as needed schedule relief to complete closure in a timely and safe manner.

TOCDF should immediately configure the DAL with the air-cooled probe designed at JACADS. TOCDF should place the CPA at the same locations in the probe that JACADS has established. TOCDF should reprogram the logic in the monitoring system for the DAL, to initiate monitoring when the DAL reaches 600°F. Because TOCDF is still processing significant quantities of liquid chemical agent, while JACADS is processing only secondary waste and decommissioning materials, it is recommended that TOCDF monitor the temperature of the DAL itself and not the exhaust duct. The DAL should be cooled to a maximum temperature of 600°F prior to initiating sampling. Measuring the temperature at this location will ensure that the temperature at the monitoring point is at or below the 600°F limitation. This system will provide TOCDF with a safe and effective start-up condition for the VX campaign.

Long Term Mitigating Measures

TOCDF has completed a test of cooling in the DAL using Simulated Equipment Test Hardware (SETH) 155mm munitions. This test was designed to provide the rate of temperature decay for a tray of projectiles. The total cool-down time for a tray of munitions to 600°F was on the order of 3 hours. Under the current DAL configuration, the impact to the program of reduced MPF throughput is estimated at $1.8 billion. This test provided sufficient engineering detail to evaluate mitigating efforts needed to allow a more rapid cooling of the tray in the DAL. Several options being considered to reduce DAL cooling time and increase MPF throughput are:

- Additional 8-inch air inlet valve to the TOCDF DAL to increase cooling air flow
- Removal of insulation from the DAL to facilitate cooling
- Addition of a second airlock to allow rapid cooling and monitoring
- Addition of engineered cooling techniques to the existing DAL, i.e., water-cooled walls, high-velocity air lance, etc.
- Total enclosure or partial enclosure of the munitions-cool-down conveyor to allow additional cooling time under engineering control

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(UDRI) to study the thermal stability of the agents GB and VX. Specifically, UDRI was asked to place these compounds on the "Dellinger List" for comparison to the surrogate compounds used during the ANCDF surrogate trial burns. This study was contracted independent of the REI investigation. During review of the draft report by UDRI this study was made available to the authors of this report. An effort was made to jointly evaluate the REI work and the UDRI work with the target being consensus among all investigative teams. UDRI did not posses the computational models that REI had access to, and therefore, identical results were not obtainable. However, the results from UDRI very closely compare to the results obtained by REI. A copy of the UDRI is included here as Appendix G.

3.2 The Heated Discharge Conveyor Bin

The HDC bin is a process discharge point that must be evaluated for effective monitoring. The HDC bin is monitored for the campaign agent prior to bin change-out. At this time, the temperature of the bin enclosure is not known. There are no plants actively processing rockets to allow a determination of the temperature profile in the HDC bin. However, the HDC operates at 1150°F with airflow in the opposite direction of the bin. Although temperatures in the HDC bin are unknown, experience at JACADS would indicate that the temperatures are not expected to be in excess of 600°F. A final recommendation can be made after TOCDF begins processing VX rockets.

3.3 Monitoring Configuration for the Discharge Airlock

At the time of the incident, the DAL was configured with an 18-inch stainless steel sampling probe. For VX, the CPA was positioned approximately 20 inches from the distal (open) end of the probe. It had been placed there to allow the hot gases from the DAL to cool so that the CPA and the Teflon inline filter would remain viable. The 18-inch length of stainless steel tubing served as both a heat exchanger and a sampling probe. Prior to installation of this probe, the conversion pads were observed to darken, become brittle and shed small black particles; the Teflon filters would lose their integrity and allow particulate matter into the ACAMS and DAAMS. With the probe/heat exchanger in place, temperatures at the filter measured 110°F while temperatures at the CPA were estimated at 120°F.

During the initial investigation into the failure to detect VX during the first two cycles in the discharge airlock, JACADS performed a test on the sampling probe to determine if the probe was a contributory factor in the occurrence. This test consisted of sampling a test atmosphere generated by injecting a dilute VX standard in isopropyl alcohol, placed in a polyethylene bag at room temperature. An approximate 1 Time Weighted Average (TWA) VX vapor was developed. This vapor was then monitored using both an ACAMS with a conversion pad placed at the distal end of a used 18-inch stainless steel probe and with a conversion pad placed at the proximal end of the same 18-inch stainless steel probe. The VX concentrations obtained with both configurations were identical.

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4

APPENDIX A

Chemical Agent Decomposition Rates at Temperatures Anticipated in the Discharge Airlock

Chemical Agent Decomposition Rates at Temperatures Anticipated in the Discharge Airlock

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REI Project No. 4440

Feb 20, 2003

REACTION ENGINEERING INTERNATIONAL

EXECUTIVE SUMMARY

Recently an incident occurred in the metal parts furnace discharge airlock (DAL) at Johnston Island in which VX readings were initially low and then were observed to rise. An early analysis attributes these apparently anomalous VX readings to thermal destruction of the VX agent in the DAL before reaching the ACAMS detection system. VX thermal decomposition rates from a forty year old report (Lapp & Schneider, 1962) are cited in support of this hypothesis.

In this paper we contend that the VX thermal decomposition rates used to infer that significant decomposition occurs before the detection equipment is reached are based on a study that does not meet modern standards for accurately determining the rates of chemical reactions. The flaws identified in the Lapp & Schneider (1962) experiments are such that the reported VX thermal decomposition rate will be much too high. Further, based on an examination of all available reported studies, we conclude that reliable measurements of the rate of gas-phase VX decomposition do not exist.

Modern computational chemistry methods have proven their ability to accurately calculate chemical reaction rates. These methods have been used to develop detailed chemical kinetic mechanisms for the pyrolysis and oxidation of GB (Glaude et al., 2002), (Bozzelli et al, unpublished, see below) and HD (Bozzelli et al, unpublished, see below). Estimates for the rate of VX thermal decomposition can be obtained from the theoretically calculated and experimentally verified rates of similar reactions of similar molecules. Using newly calculated rate parameters, we recommend that the program determine a temperature for effective monitoring of agent in the DAL.

Our calculations indicate that less than five percent VX decomposition will take place in the DAL and between the DAL and the ACAMS detection equipment until the temperature in the DAL exceeds 710 F. Consequently, the VX JACADS DAL chemical event that occurred in August, 2002 cannot be attributed to VX thermal decomposition in the pathway from the tray to the ACAMS probe, since the maximum temperature of the exhaust gas in the DAL prior to release of the tray was approximately 450F.

1-INTRODUCTION

The U.S. stockpile of chemical weapons consists of munitions, including mines, rockets, artillery shells, and bombs containing chemical warfare agents (CWAs) stored at eight sites in the continental United States and on Johnston Island in the Pacific Ocean. The CWAs consist of the blister agent mustard gas (H and HD), and organophosphorus nerve agents GB (sarin) and VX. Incineration has been used to destroy the stockpile at Johnston Island and is currently being used to destroy the stockpile at Tooele, Utah. Incinerators are under construction/systemization at three sites: Anniston Army Depot, Alabama, Pine Bluff Arsenal, Arkansas, and Umatilla Depot, Oregon.

The metal parts furnace (MPF) was designed to decontaminate hardware such as drained munitions. Metal parts are conveyed intermittently through the three furnace zones on trays. Jet fuel (JP-5) fired burners provide high temperature combustion products. Condensed agent decontamination is achieved through a combination of evaporation, decomposition and oxidation. The last stage of the MPF is a thermal cool-down in the discharge-air-lock (DAL). During cool-down, gas composition in the DAL

is checked for agent. After cool-down, trays and their contents for which no remaining agent has been detected are placed outside for further cooling.

The MPF at Johnston Island is now being used to treat secondary waste (e.g. sludge) since incineration of munitions there has been completed. The sludge is removed from 55 gallon drums where it was previously collected, spread on trays, and passed through the MPF.

Recently an incident occurred during treatment of one tray processing "sludge", in which VX readings were initially low when the tray was in the DAL and then observed to rise once the tray had been discharged. An early analysis attributes these apparently anomalous, initial, low VX readings for this tray to thermal destruction of the VX agent in the DAL before reaching the ACAMS detection system. VX thermal decomposition rates from a forty year old report (Lapp & Schneider, 1962) are cited in support of this hypothesis.

In this paper we contend that the VX thermal decomposition rates used to infer that significant decomposition occurs before the detection equipment is reached are based on a study that does not meet modern standards for accurately determining the rates of chemical reactions. The flaws identified in these experiments are such that the VX decomposition rate reported by Lapp & Schneider (1962) is much too high, i.e. the extent of VX loss (decomposition) at a given temperature is overpredicted. No experiments using CWAs have been performed since 1988 and it is unlikely that any new experiments will be performed in the future. However, modern theoretical methods have proven their ability to accurately calculate chemical reaction rates. These methods have been used to develop detailed chemical kinetic mechanisms for the pyrolysis and oxidation of GB (Glaude et al., 2002), (Bozzelli et al., unpublished, see below) and HD (Bozzelli et al., unpublished, see below). We have recently calculated the rates of the first steps of the expected dominant VX decomposition pathways. Work on a complete detailed mechanism for VX decomposition and oxidation is ongoing. Using the newly calculated rate parameters, we recommend that the program determine a temperature for effective monitoring of agent in the DAL.

The remainder of the report is organized as follows: in Section 2 we describe the project team for the SBIR program of which this report is an outgrowth. Section 3 gives reasons for concluding that the VX thermal decomposition rates of Lapp & Schneider (1962) are unreliable. Section 4 briefly reviews other work on VX decomposition. Section 5 describes modern models of CWA pyrolysis and oxidation including a calculation of the rates of the primary VX destruction paths. Section 6 shows calculations of agent destruction for the expected DAL conditions, while Section 7 gives conclusions of this report.

2-PROJECT TEAM

Reaction Engineering International (REI) is presently the contractor on a Phase II SBIR (contract no. DAAD19-01-C-0050, program officer, Dr. Robert Shaw) from the Department of Defense to develop component models for incineration equipment at the Tooele Chemical Agent Disposal Facility (TOCDF). We chose to use the incinerators at TOCDF because the incinerators to be used at other Army incineration sites will use

systems similar (in some cases identical) to those at TOCDF. These models are to help with: (1) assessing the health and environmental impact of the incinerators, (2) evaluating potential opportunities to enhance online availability and to increase munitions throughput, and (3) assessing problems with decommissioning and retroactive assessments of questions that may arise after closure.

The goal of the SBIR project is to develop a computational workbench that can be used to analyze the performance of military incinerator systems. The workbench will contain a combination of detailed Computational Fluid Dynamic (CFD) models and fast running process models. The CFD models will include the detailed chemistry and physics required to analyze the incinerator units (i.e., primary furnace and afterburners). Agent chemistry is modeled using reduced chemical kinetic mechanisms based on detailed mechanisms from the literature or developed during the project.

The project team for the SBIR consists of experienced combustion simulation engineers at REI advised by a team of leading experts in combustion chemistry including Dr. Adel Sarofim of the University of Utah and REI, Prof. Fred Gouldin of Cornell University, Prof. Joseph Bozzelli of New Jersey Institute of Technology, Dr. Wing Tsang of NIST, and Dr. Charles Westbrook of Lawrence Livermore National Laboratory. Dr. Richard Magee of Carmagen Engineering, Inc., and Dr. Richard Ward of PMCD act as advisors on the chemical demilitarization program, while Mr. Buddy Webster of Continental Research and Engineering provides incinerator operation experience.

3-EXPERIMENTAL APPROACHES

In this section we make a detailed consideration of the experimental techniques used by Lapp & Schneider (1962).

Thermal decomposition of VX and BIS (2, Ethyl Hexyl) Hydrogen Phosphite (a VX simulant) were studied in a laboratory flow reactor to test the potential for VX decomposition during evaporation and rapid condensation to form mists. The flow reactor carrier-gas was helium. Heating to temperature was by electrical resistance heating. The agent or simulant was injected into the reactor via a side tube – the injection tube (see Figure 5 in Lapp & Schneider which is reproduced in Figure 1 below).

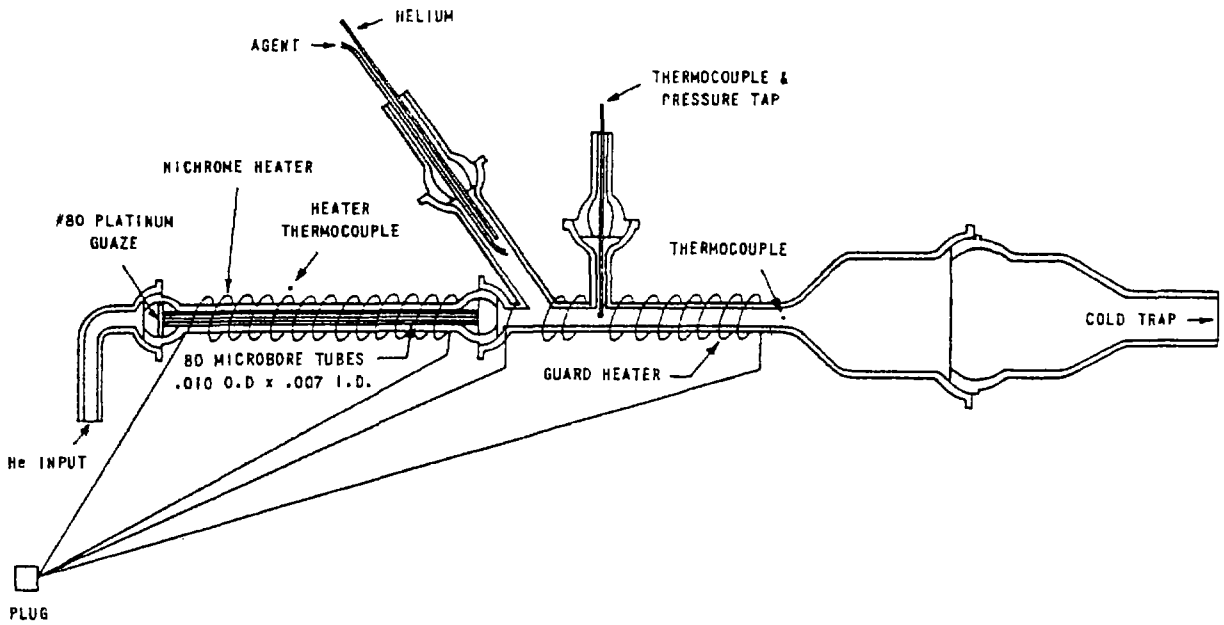

Figure 1. Schematic of the apparatus of Lapp & Schneider (1962). This figure is reproduced from Lapp & Schneider Figure 5, p. 10.

Measurements were made for different reactor temperatures and residence times; the different residence times were achieved by altering the reactor temperature and hence the volume flow rate. Samples were collected from condensate in a cold trap (and apparently adjoining surfaces) through which the discharge of the reactor passed. These samples were analyzed to determine destruction levels for VX or BIS. Sample analysis techniques are not discussed in detail; they were said to be derived from Deiner, et al., (1958).

Data for VX destruction versus residence-time were used to obtain VX half-life versus temperature data. In turn, the half-life data were used to estimate the activation energy assuming first order decomposition kinetics. The reported activation energy for VX is 26 kcal/mole (our review of the data indicates a value of about 17 kcal/mol). This value is exceptionally low and not consistent with expectation based on both current theory and experience in the study of the decomposition of comparable molecules. Sides et al. (1984) pointed out this data reduction error (26 vs. 17 kcal/mole) and commented on other errors in the work of Lapp & Schneider.

For extracting kinetic data, several concerns exist regarding the usefulness of the experimental data reported. The data analysis procedure is based on several implicit assumptions. Agent and He flows are assumed to mix instantaneously thereby dispersing the liquid to fine droplets, vaporizing the droplets, and raising the agent temperature to the reactor temperature instantaneously. Local gas velocities in the reactor are assumed to be uniform. Heterogeneous wall reactions of agent are neglected, and reactions are assumed to be quenched quickly as the flow leaves the reactor. The condensed samples collected in the cold trap are assumed to be representative of the homogeneous, gas phase conditions at the reactor exit. Finally, chemical samples are collected over the duration of the experiment. The data analysis assumes operation is steady and that start-up

transients are of short duration. There are good reasons to believe that most, if not all, of the above conditions are not met.

Poor Mixing Liquid VX is injected at very low flow rates via a side tube with hot walls; conditions in this side tube are ill defined. The inlet flow of liquid VX is quite slow, on the order of 0.1 ml/min or slower. Start-up transients associated with the filling of this tube with agent are probably significant, and some VX decomposition will occur in the injection tube. Gas velocities in the reactor are estimated to be approximately 100 m/s, and there is a large momentum flux mismatch between the reactor flow and the VX injection flow. This mismatch means that the two flows do not rapidly mix. Instead the agent flow is likely to remain in the slower-flowing wall boundary layer of the reactor. Experience over the last twenty plus years with flow reactor studies of chemical reaction rates makes it clear that reactant injection is a critical aspect of flow reactor operation. Injection should occur directly into the reactor in order to achieve rapid, uniform mixing.

Unsteady Reactor Operation VX is injected along with a secondary – small flow – stream of helium into a side arm region of the reactor. The inlet flow of liquid VX is quite low, on the order of 0.1 ml/min or slower.

The overall reactor operation procedure is to initially warm up and achieve steady state of the carrier gas–helium flow at the target temperature and flow velocity. From Table IX of the report it appears that He fluxes for all experiments are 0.085 scfm. This method of operation is fine to condition and achieve steady state temperature and flow of the carrier gas. It is however not a real steady state for the reaction system. Once the carrier flow and temperature of the reactor are at steady state the agent flow is then initiated. At this point steady state does not exist. Liquid flows into the side tube where an initial mixing and perhaps some vaporization occurs. Residence time in the side injection tube is unknown. Eventually a partially-mixed or non-mixed flow of liquid, aerosol particles and gas will flow into the main reactor flow stream. This is also non-steady-state. The time needed to achieve steady state is not described or measured. It is also very likely that some and perhaps much of the VX may decompose in the side injection tube.

Boundary Layers Wall boundary layers of low velocity flow will grow in the reactor. VX is likely to be trapped in these boundary layers meaning that the actual residence time characteristic of a typical VX fluid parcel is significantly longer than that used for data analysis and that local concentrations of VX will be higher than those used for analysis. Wall reactions are known to be a problem for flow reactors especially if the wall material has good catalytic properties, e.g., steel. Quartz is not highly catalytic, but wall reactions cannot be ruled out in these studies. Poor mixing, wall boundary layers and the potential for surface reactions all contribute to making the conditions in the reactor ill-defined.

Temperature Uncertainty Temperature profiles as a function of distance in the reactor test section are not provided; only two temperature measurement points are noted in the experiment description for this test section. It is always desirable to monitor the internal reactor conditions with a movable thermocouple (internal to the reactor) to determine the temperature profile. This can readily be done in a mode where there is

carrier, but not reagent (agent) flow. It is possible that a very non-uniform temperature profile exists.

Poorly Defined Residence Times The residence time/exposure time of VX is determined from the residence time in the reactor, i.e., the reactor length divided by the volume-averaged flow velocity. This relationship assumes that reactions are quenched at the exit of the reactor, which in turn requires the rapid cooling of the gases leaving the reactor. There is no indication in the report that quenching will be sufficiently rapid. Residence times used in the data analysis are approximately 1 millisecond. Quenching times must be small compared to this time, and the task of achieving times on the order of 100 microseconds is far from trivial. For several reasons the actual VX exposure time to high temperature is longer than the time used to estimate decomposition rates. These reasons can be summarized as 1) finite, but unknown residence time in the injection tube, 2) agent trapped in the low velocity wall boundary layer flow and 3) finite, but unknown, quenching times of the flow downstream of the reactor.

Data Analysis and Evaluation of Kinetic Rates Evaluation of data in Table IX, page 27 of Lapp & Schneider (1962) gives an activation energy (E_a) value for VX decomposition of about 17 kcal/mole. This is significantly different from the value given in the report text, which is 26 kcal/mole. This leads to questions on interpretation or reporting of the data by Lapp & Schneider. This error was previously reported by Sides et al. (1984).

An Arrhenius rate expression activation energy of only 17 kcal/mole for decomposition of VX or any molecule this size is exceptionally low. Using this value with a reasonable pre-exponential factor, A, of 10^{12} sec^{-1} (Benson, 1968, 1976) in an elementary reaction analysis would suggest that VX decomposition at room temperature and one atmosphere pressure would have a half-life of less than a second. That is, VX is unstable at normal working atmospheric conditions. Lapp & Schneider's data implies a frequency factor of about 2.7×10^7, which, like their activation energy value, is outside the range of such parameters found for thermal decomposition of molecules the size of VX.

Summary Sides et al. (1984) point out that "Lapp & Schneider made several erroneous statements in their report, and these errors reduce our confidence in their work." We agree. The conditions of the experiment are ill-defined. It is likely that several if not most of the assumptions implicit in the data analysis procedure are not satisfied. VX is not rapidly and uniformly mixed with the He flow. VX exposure times are longer than assumed and wall reactions may be significant. The latter two effects will lead to an over-estimation of VX decomposition rates. Data in this report should not be considered a reliable indication of VX decomposition rates.

4-OTHER WORK ON VX DECOMPOSITION

A number of investigations have calculated Arrhenius-form decomposition rates for VX. We briefly review some of these here.

Dee & Berger (1975) examined the stability of liquid VX during storage and the efficacy of certain stabilizing compounds. Their work is entirely devoted to liquid-phase decomposition, and not applicable to gas-phase decomposition of VX in the DAL. Sides

et al. (1984) give rate parameters from Dee & Berger, but these cannot be found in the version of the Dee & Berger report available to us. Sides et al. report that "Most of the details concerning the work of Dee and Berger are classified."

The Rohrbach et al. (1988) study, like the work of Dee & Berger, was also focused on liquid-phase stability of VX during storage and has little applicability to DAL sampling, which is governed by gas-phase kinetics.

Sides et al. (1986) summarize studies in which liquid agent is placed inside a stainless steel Swagelok union and heated. The Swagelok union is then cooled in liquid nitrogen, opened, and the contents analyzed by gas chromatography. This approach is unlikely to give reliable results due to thermal gradients in the stainless steel container, evaporation, and decomposition occurring in both the gas and liquid phases.

Our conclusion from examining the available reports is that reliable measurements of the rate of gas-phase decomposition of VX and its simulants do not exist.

5-MODELING APPROACHES

This section briefly discusses the advances in kinetic modeling that are used today to develop chemical reaction rates and their application to modeling destruction of CWAs. Models describing agent pyrolysis and oxidation were taken from recent literature or developed by generating chemical kinetic rate constants and thermochemical data for species and reactions not available in the literature. To our knowledge, no experiments using CWAs are being performed or are planned. These detailed chemical kinetic mechanisms are being developed from *ab initio* and density functional computational chemistry calculations and verified by comparison to high quality experimental data for similar substances where available. The types of calculations used to determine these rates are used in hundreds of journal publications yearly in the field of chemical kinetics.

Detailed Mechanism for HD Destruction

A detailed chemical kinetic mechanism for HD pyrolysis and oxidation has recently been created. Thermodynamic data and destruction pathways for HD via retro-ene reaction and H-abstraction were generated by Prof. Bozzelli, along with the necessary thermodynamic data and reaction steps needed to break down the initial products of HD destruction to the point where existing mechanisms can model the completion of the oxidation process. REI has combined these reaction steps for HD and its immediate products with existing mechanisms for combustion chemistry of chlorine (Ho, et al., 1995; Procaccini et al., 2000), sulfur (Nimmo, et al., 1998), and some nitrogen chemistry steps from Dean & Bozzelli (1999). The chemical kinetic rates were estimated from heats of reaction, structural considerations, and estimates of energy barrier heights. Thermochemical information was obtained from bond energy methods using the THERM software. Where necessary, more refined estimates were obtained using BAC-MP4 or group balance isodesmic reactions with *ab initio* and density functional calculations. As has been found for agents GB and VX, HD destruction is dominated by a unimolecular retro-ene reaction with a rate constant given by

$$k = 2.3\times10^{13}e^{(-52.25/RT)}\ \text{sec}^{-1}$$

where R is in kcal/mol·K and T is in Kelvin.

Detailed Mechanism for GB Destruction

The detailed chemical kinetic mechanism for oxidation of nerve agent GB (Glaude et al., 2002) was developed at Lawrence Livermore National Laboratory under the supervision of Dr. Charles Westbrook, by building on previous work (Glaude et al., 2000) on kinetics of organophosphorus GB simulants TMP, DMMP, and DIMP. The most important reaction for the destruction of GB was found to be a six-center unimolecular retro-ene reaction eliminating propene. This reaction is illustrated in Figure 1. Rates of destruction of organophosphorus compounds were verified by comparison to the experiments of Zegers & Fisher (1996, 1998a,b).

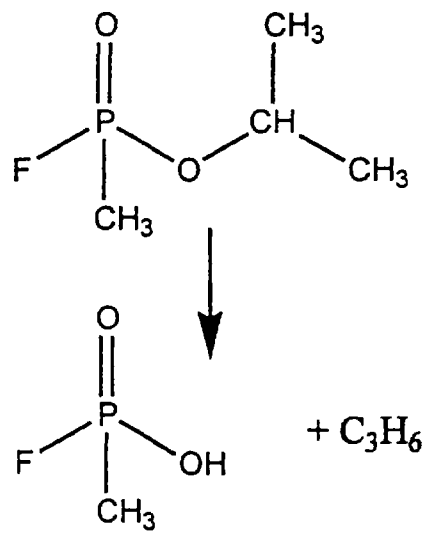

Figure 1. Dominant destruction pathway for GB (from Glaude et al. 2002).

The rate of the dominant GB destruction pathway was estimated by Glaude et al. "based on commonly used principles of bond additivity and hierarchical reaction mechanisms."

To improve the precision of the dominant rate expression for GB destruction in the Glaude et al. mechanism, Bozzelli and Chen have performed calculations on the retro-ene and bond cleavage reactions using density functional computational chemistry and canonical transition state theory and thermochemical kinetics techniques. The calculated rate expression for the lowest energy reaction is:

$$k = 4.03\times10^{7}T^{1.62574}e^{(-40.69/RT)}\ \text{sec}^{-1}$$

where R is in kcal/mol·K and T is in Kelvin. Other retro-ene reaction paths had energies of activation of 62.6 and 62.8 kcal/mole. Bond cleavage reactions had barriers higher than this. The higher retro-ene and bond cleavage reactions are not competitive with the one degenerate low energy (40.69 kcal/mol) reaction in the target temperature range.

Reaction Rates for VX Destruction

The dominant pathway for VX decomposition is a six-center molecular elimination reaction similar to the findings of Glaude et al. (2002) for GB. This pathway is illustrated in Figure 2.

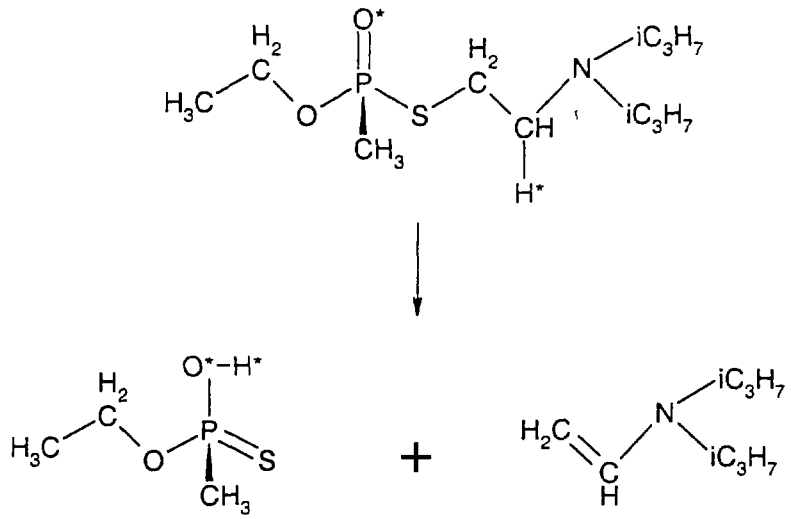

Figure 2. Dominant destruction pathway for VX.

This type of retro-ene reaction is typical of unimolecular decomposition in molecules this size and is the lowest energy and highest rate unimolecular decomposition path for VX in the gas phase.

The rates of this reaction have been calculated using a variety of computational chemistry methods. Details of the methodologies used to determine the rate constants are given in the Appendix. The calculated values are for the reaction illustrated in Figure 2, which is the same reaction previously estimated to be the lowest energy path (Montgomery et al., 2002).

Initial calculations by Prof. Bozzelli at the density functional level B3LYP/6-31G(d,p) gave estimates of the activation energy and rate constants for decomposition of VX. The lowest E_a value found was 36.96 kcal/mole. For details of this type of calculation see Lee et al. (2002). Considerable experience with the B3LYP method shows that it underestimates the activation energy of reactions in about 30% of the molecules studied. Several newer calculations using more reliable methods are in close agreement and show that the B3LYP result is incorrect. Having now completed seven higher-level calculations, we recommend that the B3LYP calculation be rejected completely. Statistically, it is an outlier among the data points generated using the KM and CBS methods.

A series of four high level, calculations using the KMLYP method (Kang & Musgrave, 2001) results in an activation energy of 44.34 to 45.32 kcal/mole. Because of the importance of this decomposition model the following higher level computational methods were successfully completed: G3(MP2), CBSQ/B3LYP and CBS-QB3. These computational methods are considered very high in level and in accuracy, but require

large amounts of accessible disk scratch space, memory and computational time. Overall it took more than a month to get the computations to work fully. The resulting energy barriers from these techniques are:

G3(MP2)	42.17 kcal/mol
CBSQ/B3LYP	41.99 kcal/mol
CBS-QB3	42.57 kcal/mol

The final recommended rate constant is based on the G3(MP2), CBSQ/B3LYP and CBS-QB3 calculations. Averaging the calculated enthalpy differences between the VX molecule and the transition state from these three highest-level calculations and curve fitting to match the calculated rate constants, we arrive at the final recommended rate expression:

$$k = 5.36\times10^{6}T^{1.65068}e^{(-41.64/RT)}\ \mathrm{sec}^{-1}$$

where R is in kcal/mol·K and T is in Kelvin.

The second lowest activation energy found for VX decomposition is 62.68 kcal/mole. This path is also a retro-ene reaction, but it involves cleavage of a carbon–oxygen (ether bond link) instead of a carbon-sulfur bond. KMLYP and B3LYP calculations for this higher Ea reaction produced essentially the same energy barrier and reaction rate constant. Bond cleavage reactions have higher barriers than the retro-ene reactions. The higher activation energy retro-ene and bond cleavage paths are not competitive with the 41.64 kcal/mole reaction path cleaving the carbon-sulfur bond.

A number of references support the proposed rate parameters for the six-center molecular elimination reactions of VX. The oldest and most respected source is Benson (1968, 1976). This book contains a long and detailed discussion in Chapter 3, "Arrhenius Parameters for Gas Phase Reactions", on pages 75-82 in the first edition, about six-center molecular elimination reactions. This chapter includes a table of rates for many reactions of this class. The majority of these reactions are for intramolecular reaction paths similar to the important (low energy) paths in VX and GB, where a large olefin (as in GB) or a large substituted olefin species (in VX) are eliminated. All the reactions listed by Benson have pre-exponential factors ranging from 10^{11} to 10^{13} sec^{-1}. The evaluation and discussion in the book supports these values. The pre-exponential factor given above for the dominant decomposition pathway varies from 1.768×10^{11} to 2.659×10^{12} over the temperature range 300-2000 K. The pre-exponential factor given above, temperature exponent, and activation energy are determined through curve-fitting to account for this variation. More details can be found in the Appendix.

Várnai, & Keserü (1996) provide a reasonably recent summary of kinetic parameters for retro-ene reactions that are typical of unimolecular decomposition in molecules this size. Their values support the suggested parameters.

Validation and Uncertainty

Data on validation of computational chemistry techniques can be found in articles by Montgomery et al. (2000), Curtiss et al. (1999), and Kang & Musgrave (2001). In the article by Montgomery et al. (2000), statistics on the uncertainty between theory and

experiment in the activation energy for approximately 150 compounds is listed. Additional information on verification of theoretically calculated reaction rates can be found in Chen & Bozzelli (1999, 2000) and Sheng et al. (2002). Based on this literature, the uncertainty in the activation energy for the calculations presented in the REI report is +/- 1.5 kcal/mol.

Surface Reactions

The steel walls of the DAL could catalyze destruction of VX. The rate of such catalyzed destruction is likely to be higher than for gas-phase decomposition, but is difficult to determine accurately. At issue is whether wall reactions are significant compared to gas-phase decomposition, for which the rate can be accurately determined using modern computational chemistry techniques. For the reasons discussed below, it is the opinion of the authors that wall reactions will not be important in this system under the conditions of interest.

Mass transfer rates of agent to the surface reduce the importance of surface reactions. Measurements of chemical reaction rates for a variety of compounds in laboratory reactors (Ritter et al. 1990, Ritter & Bozzelli 1990) show consistently that for tube diameters larger than ¾ in. wall effects are negligible compared to the gas-phase reactions, demonstrating mass transfer limitations. The surface-to-volume ratio for a ¾ in. tube reactor is 64 ft^{-1}. The surface-to-volume ratio in the DAL is on the order of 1 ft^{-1}, providing a considerable margin of safety for neglecting wall reactions. (Note: Any agent released into the area above the munitions will have little opportunity to react with the munition surfaces, thus munition surface area is considered unimportant.)

Reaction with Water Vapor

Water vapor will be present in the DAL from atmospheric moisture and combustion products. However, the bimolecular reaction of H_2O with VX will be considerably slower than the unimolecular decomposition pathway for which the rate has been calculated. Reaction of VX with water involves formation of a complex in the gas phase followed by reaction of the complex to new products. This reaction will have only slightly lower activation energy than unimolecular dissociation. The reaction rate will be decreased significantly by the requirement for bimolecular collision and complex formation and the very rapid dissociation of the complex back to reactants.

6-AGENT DESTRUCTION CALCULATIONS

Using recently developed detailed chemical kinetic mechanisms for GB and HD, and a calculated rate for the first steps of the dominant VX decomposition pathways, it is possible to calculate destruction of the three agents for conditions expected to be found in the DAL.

Following the August, 2002 event, JACADS personnel carried out experiments with empty trays to determine the temperature in the DAL when the event occurred. The estimated temperature in the DAL during the recent chemical event is 450 F (505 K). The vapor residence time in the DAL to reach the ACAMS sampling system has been estimated by Dr. Michael MacNaughton of SwRI to be 30 seconds. This temperature and residence time seem to be well-accepted by people in the chem demil community familiar with the event.

Agent destruction has been calculated as a function of temperature in a fixed-temperature plug flow reactor with a 30 second residence time. The starting composition consisted of 1000 ppm agent in air. Because agent destruction is dominated by unimolecular decomposition under these conditions, the results are independent of the initial amount of agent assumed for the calculations. The results are shown in Figure 3.

Figure 3 shows that no thermal decomposition of VX can be expected to take place in 30 seconds at 450 F in the DAL exhaust gas. As shown on the figure, effective monitoring can be achieved up to about 760 F for VX. Effective monitoring is defined at 75% recovery of the initial concentration. This definition is derived from the PMCD QA plan. The dotted line in the figure crosses the curves at the temperature where 75% recovery is predicted to take place.

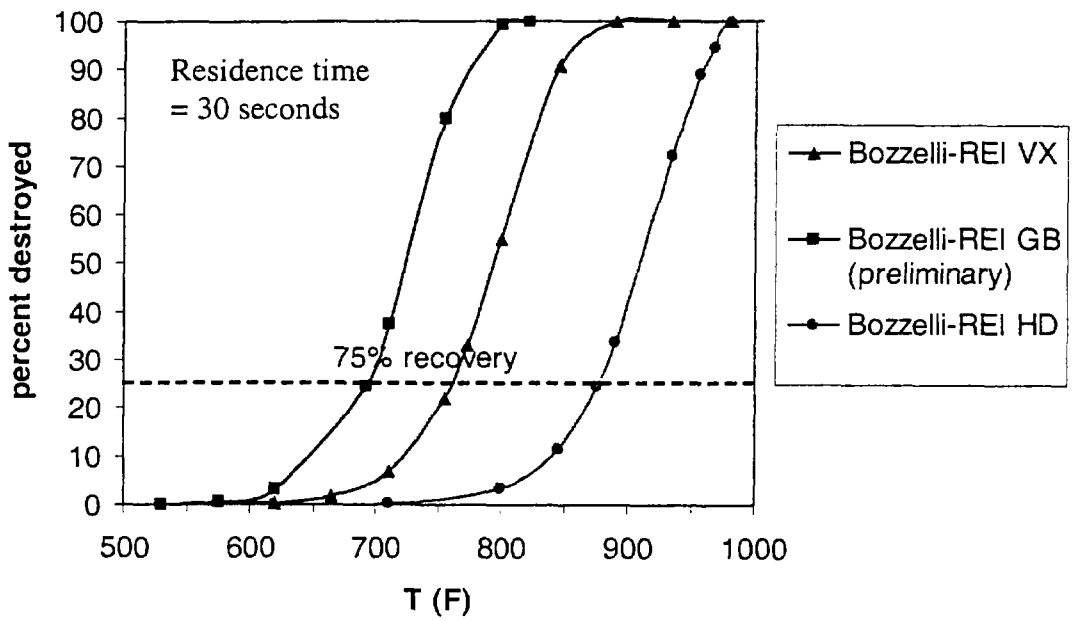

Figure 3. Calculated gas-phase agent destruction in air in a fixed-temperature plug flow reactor with a residence time of 30 seconds.

For a 30 second residence time, the following are the maximum temperatures to assure at least 75% agent recovery:

VX	761 F	(678 K)
GB	692 F	(640 K)
HD	876 F	(742 K)

7-CONCLUSIONS

VX thermal decomposition rates reported by Lapp & Schneider (1962) that have been used to estimate VX thermal decomposition in the DAL are unreliable and significantly over-estimated due to poorly characterized experimental conditions, calculated rates that differ from their own reported data, and large discrepancies between their reported rates and known rates of similar reactions in similar molecules.

Modern computational chemistry methods have proven their ability to accurately calculate chemical reaction rates. These techniques have been used to develop detailed chemical kinetic mechanisms for the pyrolysis and oxidation of GB and HD. Using these methods, the rates of the first steps of the dominant VX decomposition pathways have been calculated. Further, based on an examination of all available VX decomposition studies, reliable measurements of the rate of gas-phase decomposition of VX do not exist. Agent decomposition rates derived from modern computational chemistry predict that no agent will decompose given the expected conditions in the DAL and the piping leading to the ACAMS sampling system for the reported conditions.

Our calculations indicate that less than five percent VX decomposition can be expected to take place in the DAL and between the DAL and the ACAMS detection equipment until the temperature in the DAL exceeds 710 F. Consequently, the VX JACADS DAL chemical event that occurred in August, 2002 cannot be attributed to VX thermal decomposition. Effective monitoring of VX, consistent with the PMCD QA plan, can be achieved at 760 F (See Figure 3). Using these newly calculated VX decomposition rate parameters, it is recommended that the program specify a temperature for agent monitoring in the DAL.

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Appendix: Thermochemical Properties, Reaction Paths and Kinetic Parameters for Unimolecular Dissociation of VX

Executive Summary

A retro-ene reaction is found to be the lowest energy unimolecular decomposition (initiation) reaction for thermal VX decomposition. The enthalpy barrier for this reaction is found to be 42.24 kcal/mol (average of the three highest level calculations). This reaction involves cleaving the sulfur-carbon bond, forming a phosphorous-sulfur double bond and transferring a hydrogen atom from the carbon atom adjacent to the amine group to the phosphonyl oxygen (R3P=O oxygen). In this reaction the phosphorous is the central atom with the atoms in red, blue and green also involved in the process.

$$(C_2H_5O\text{-})P(CH_3)(=O)(S\text{-}C_2H_4\text{-}N(iprop)_2 \rightarrow C_2H_5OP(=S)(OH)CH_3 + C_2H_3\text{-}N(iprop)_2. \quad \text{(I)}$$

A second retro-ene reaction, cleaving an oxygen-carbon bond, forming a new phosphonyl double bond, with transfer of a hydrogen atom from a primary carbon atom to the phosphonyl oxygen atom, is found to have a significantly higher energy barrier (62.68 kcal/mol) and is not an important initiation process.

$$(C_2H_5O\text{-})P(CH_3)(=O)(S\text{-}C_2H_4\text{-}N(iprop)_2 \rightarrow C_2H_4 + O{=}P(OH)SC_2H_4\text{-}N(iprop)_2. \quad \text{(II)}$$

Phosphorous–sulfur and sulfur-carbon bond cleavage reactions are found to be higher than the 62.68 kcal/mole above and consequently are unimportant reactions in the discharge airlock.

Background

Density functional methods are selected as the best choice methods capable of performing the calculations on the large molecular systems in this study, where the basis sets needed for sufficient accuracy can be used and there are a sufficient number of studies in the literature to validate these calculations. Standard enthalpy of formation, ΔH^0_{f298}, entropy, S^0_{298}, and heat capacities $C_p(T)$, are determined for stable molecules, intermediates, and transition states using density functional calculations with the B3LYP (Becke- 3 parameter Lee Young Parr) and the KMLYP (Kang-Musgrave-Lee Young Parr) functionals (Kang & Musgrave, 2001). The calculations are performed using several moderately large basis sets augmented with diffusion and polarization functions. Reactant, product and transition state structures are optimized at the B3LYP/6-31G(d,p) level and with the KMLYP functional with four different basis set configurations up to 6-311G(2d,d,p).

The Gaussian 3 Moeller Plesset 2nd order (G3(MP2)) and the Complete Basis Set (CBS) composite methods of Curtiss and of Peterson, respectively, were also used with representative molecules in order to provide further support and validate the density functional results. These codes are used as implemented in Gaussian 98, a commercially available and highly referenced computational chemistry software package.

The B3LYP density functional computation method was chosen as the initial computational method because it is the most commonly used computational chemistry method in the organic and inorganic chemistry journal literature at this time-- it is nearly a default, initial, calculation level in physical chemistry research.

The KMLYP method with the basis sets noted below was selected because we have previously compared both KMLYP and B3LYP methods on complex reaction systems with up to 9 heavy atoms in the second row of the periodic table and found overall good agreement with higher level CBSQ, G3 and G3(MP2) calculations on transition state parameters. The KMLYP method was implemented because B3LYP, while widely accepted, is known to sometimes give low values for transition state enthalpies and our experience with KMLYP has demonstrated a significant improvement in this problem area over B3LYP.

Kinetic parameters for unimolecular (dissociation and isomerization) reactions are calculated using canonical transition state theory.

Enthalpies of formation ($\Delta H_f°_{298}$) are determined using the KMLYP and CBSQ composite method and from density functional calculated structures by the heat of atomization and by working reaction analysis procedures where possible. Entropy (S) and heat capacity ($C_p(T)$), values from vibrational, translational, and external rotational contributions are calculated using the rigid-rotor-harmonic-oscillator approximation, based on the vibration frequencies and structures obtained from the density functional studies. The contribution to S and $C_p(T)$ from analysis on the internal rotors is incorporated into these themochemical properties, instead of the more conventional use of torsion frequencies. This is an important consideration to the thermochemical and kinetic analysis of this study, because there is a significant loss of internal rotors in transition states, and the entropy change is important to calculation of an accurate pre-exponential factor in kinetics. Entropies ($S°_{298}$) and heat capacities $C_p°(T)$, are determined using geometric parameters and vibrational frequencies obtained at the KMLYP/6-311G(2d,d,p) calculation level of theory.

VX is a large molecule with formula $C_{11}H_{26}O_2NPS$. It has two atoms in the third row of the periodic table, sulfur and phosphorous, and 14 atoms (carbon, nitrogen and oxygen) in the second row. The calculations of transition state properties at sufficient accuracy needed for the kinetic analysis could not be performed at the computation chemistry levels above, with inclusion of orbital basis sets, up to 6-311G(2d,d,p), on a molecular system with this number of second and third row atoms. The thermochemical properties needed for the kinetic parameters were, therefore, determined for each of the two decomposition pathways using representative smaller molecules. These smaller molecules included all the important elements (atoms and bonding) needed for accuracy in determination of the respective transition state; one or two groups that are not active in the reaction and not important to the transition state parameters were removed from the molecule to make the high-level calculations possible.

The VX molecule is $(C_2H_5O-)P(CH_3)(=O)(S-C_2H_4-N(iprop)_2$. The products for the important low barrier decomposition reaction are $C_2H_5OP(=S)(OH)CH_3$ + $C_2H_3-N(iprop)_2$. The isopropyl (iprop, $(CH_3)_2CH--$) groups are not involved in the reaction or in the transition states for reactions I and II.

The B3LYP and KM calculations for Transition state I are calculated using the molecule $(CH_3O-)P(CH_3)(=O)(S-C_2H_4-N(H)_2$, where the isopropyl groups are replaced by H atoms and the ethoxy group (C_2H_5O-) is replaced with a methoxy group (CH3O-). There are two sp3 carbons and one sp3 nitrogen between the isopropyl groups and the

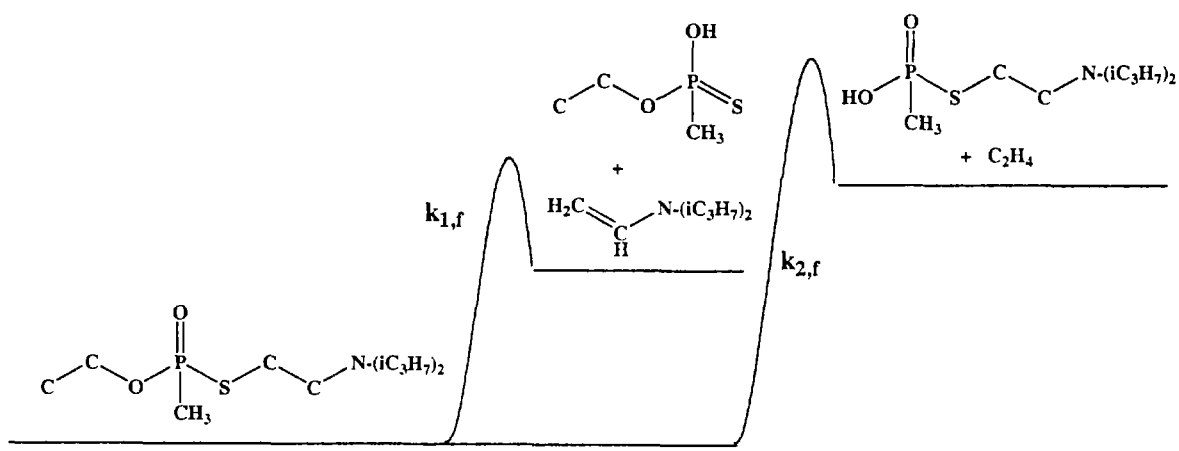
reacting moiety; these saturated (sp3) carbons effectively shield the transition state from any different electronic effects the replaced groups would have or exert. Additionally four KM calculations and one CBSQ/B3LYP, one CBS-QB3, and one G3(MP2) calculation are performed on the molecule $(HO-)P(CH_3)(=O)(S-C_2H_4-N(H)_2$.

Transition state II is calculated for $(OC_2H_5)P(CH_3)(=O)(S-CH_3)$ based on similar reasoning.

This use of smaller molcules allows us to reduce the number of heavy atoms and run the calculations at higher levels. This is a procedure routinely done in *ab initio* thermochemical and kinetic calculations on biological systems, where it is impossible to perform high level calculations on the entire molecule. Hydrogen substitution for these non-active groups (in the kinetic process) on the molecule permits use of the higher level calculations. Overall, two kinetic reaction paths exist, with a different model reactant used for each; four KMLYP and one B3LYP calculations have been run on each reaction path. In addition, for the dominant path the CBSQ/B3LYP, CBS-QB3, and G3(MP2) methods have been used for the dominant path.

Results

Figure A1 illustrates the potential energy diagram of these two VX unimolecular reaction channels and the VX moiety. The values listed in this figure are the rate constant parameters that fit the specific statistical mechanics calculated rate constant from canonical transition state theory over the temperature range from 300 to 2000 Kelvin. The value of the E_a in this fit equation is just slightly different from those in the tables and the agreement of the fit equation with the exact data is illustrated in Table A1, which contains the thermochemical and kinetic parameters versus temperature.



$k_{1f} = 5.36\times10^{6}T^{1.65068}e^{(-41.64RT)}$ sec^{-1}, R in kcal/mol·K, T in K

$k_{2f} = 1.02\times10^{9}T^{0.49842}e^{(-62.68/RT)}$ sec^{-1}, R in kcal/mol·K, T in K

Figure A1. Energy barriers for the two most important unimolecular VX decomposition reactions. (Note: The "*f*" subscript indicates reaction in the forward direction.)

Kinetic Parameters

Transition State I (TSI) The specific reactants, transition states and products are illustrated at the top of Figure A2 on transition state TSI. This reaction involves a hydrogen atom transfer from the carbon atom adjacent to the amine group to the phosphonyl oxygen atoms, cleavage of a sulfur-carbon bond and formation of a sulfur-phosphorous double bond. In this model reaction for the calculation of the transition properties, the two isopropyl groups were removed and hydrogen atoms were substituted onto the nitrogen (amine group) and CH_3 replaces C_2H_5 on the non-reacting oxygen (ether) link. These are the only deviations from the full VX molecule. The vinyl-amine linkage that is formed needs to be kept in the reaction system, thus the nitrogen (amine group) was not removed. The groups on the phosphorous atom were not altered. Note that seven different calculations have been performed that give the same barrier energies from four different basis sets and structure optimizations, using KMLYP functional, as well as three higher level calculations (CBSQ/B3LYP, CBS-QB3, and G3(MP2)). The values from all the calculations are shown in the Figure A2 below the diagram for TSI.

VX => TSI => $C_2H_5OP(=S)(OH)CH_3$ + C_2H_3-N(iprop)$_2$
k = 5.36E+06 T^1.65068 exp(-41.64/RT) E_a in kcal/mol

Transition State II (TSII) The reactant, transition state and products for the calculation (B3LYP and four KMLYP density functional calculation) methods on transition state TSII are illustrated in the lower part of Figure A2. This reaction involves cleavage of the phosphorous-oxygen (ether) link and formation of a sulfur double bond to the P atom, with a hydrogen atom transfer from the ethyl group attached to the ether. In the model reaction for the calculation of this transition state, the sulfur--ethyl di-isopropyl amine group (S--$C_2H_4N(iC_3H_7)_2$ is replaced by a methyl (S--CH_3) group. The groups on the phosphorous atom were not altered.

VX => TSII C_2H_4 + $(CH_3)P(OH)(=O)$ S-C_2H_4-N(iprop)$_2$
kII = 1.02E+09 T^0.49842 exp(-62.68/RT) E_a in kcal/mol

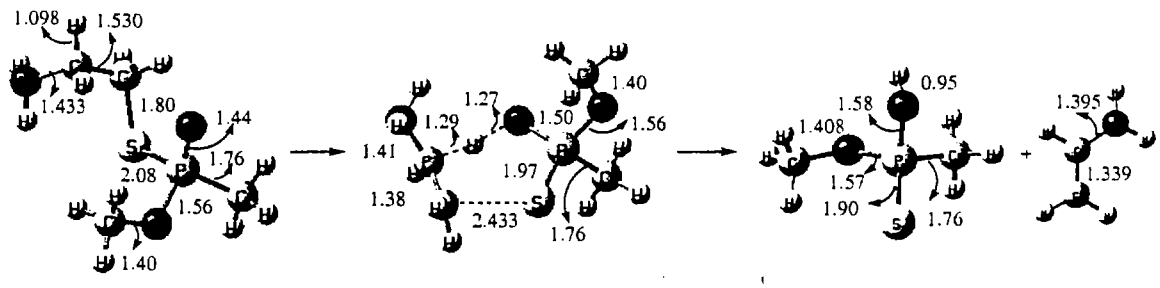
Thermochemical data for the two unimolecular reaction processes are presented in table A1. The data include the heat (enthalpy of formation), entropy at 298 K, and the heat capacities from 300 to 2000 K for VX, transition states and reaction products.

Conclusions

A retro-ene reaction is found to be the lowest energy unimolecular decomposition (initiation) reaction for thermal VX decomposition with a 42.24 kcal/mol enthalpy barrier (average of the three highest level calculations). This reaction involves cleaving the sulfur-carbon bond, forming a phosphorous-sulfur double bond and transferring a hydrogen atom from the carbon atom adjacent to the amine group to the phosphonyl oxygen (R3P=O oxygen). A second retro-ene reaction, cleaving an oxygen-carbon bond, forming a new phosphonyl double bond, with transfer of a hydrogen atom from a primary carbon atom to the phosphonyl oxygen, is found have a significantly higher energy barrier, (62.68 kcal/mol), and is not an important initiation process. The rate constant for the dominant VX decomposition pathway is

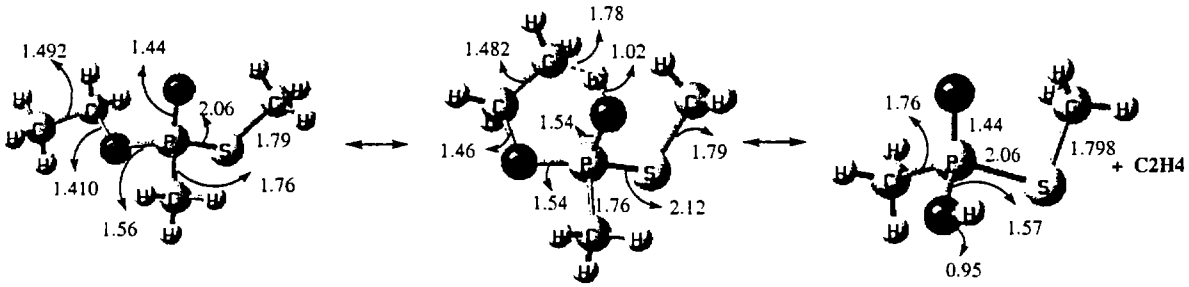
$$k = 5.36\times10^{6}T^{1.65068}e^{(-41.64/RT)}\ \mathrm{sec}^{-1}$$

where R is in kcal/mol·K and T is in Kelvin.



	VX'			TS1				
Barrier (kcal/mol)	B3LYP /6-31G(d,p)	KM1 (BLYP/6-31G(d,p))	KM2 (BLYP/6-311G(d,p))	KM3 (BLYP/6-311+G(d,p))	KM4 (BLYP /6-311G (2d,d,p))	G3(MP2)// B3LYP/6-31G(d,p)	CBS-Q //B3LYP /6-31 G(d,p)*	CBS-QB3*
Forw.	36.96	45.72	44.74	44.34	45.39	42.17	41.99	42.57
Rev.	25.53	26.25	26.99	28.00	26.88	29.33	27.37	

*The CBS-Q and CBS-QB3 calculations were performed using slightly different substitutions for the unimportant part of the VX molecule.



	VX"		TS2		
Barrier (kcal/mol)	B3LYP /6-31G(d,p)	KM1 (BLYP/6-31G(d,p))	KM2 (BLYP/6-311G(d,p))	KM3 (BLYP/6-311+G(d,p))	KM4 (BLYP/6-311G(2d,d,p))
Forw.	62.00	61.64	60.79	61.11	62.56
Rev.	44.74	34.74	37.19	37.86	39.23

Figure A2. Density functional calculation results.

Table A1. Thermodynamics and Kinetics of VX Unimolecular Decomposition Reactions

THERMODYNAMIC and KINETIC ANALYSIS for REACTION
VX => TSI => C2H5OP(=S)(OH)CH3 + C2H3-N(iprop)2
kI = 5.36E+06 T^1.65068 exp(-41.64/RT) Ea in kcal/mol

Hf {Kcal/mol}	.000	42.240
S {cal/mol K}	112.030	104.960

dHr {kcal/mol}	(298K)	=	42.24	dHr avg (298., 1500. K)	=	42.88
dU (dE) {kcal/mol}	(")	=	42.24	dUr avg (298., 1500. K)	=	42.88
dSr {cal/mol K}	(")	=	-7.07	dSr avg (298., 1500. K)	=	-6.39
dGr {kcal/mol}	(")	=	44.35	dGr avg (298., 1500. K)	=	48.62

T (K)	dH(Kcal/mol)	dU(Kcal/mol)	dS(cal/mol K)	(Af/Ar)	dG(Kcal/mol)
300.00	4.224E+01	4.224E+01	-7.085E+00	2.828E-02	4.436E+01
400.00	4.212E+01	4.212E+01	-7.432E+00	2.375E-02	4.509E+01
500.00	4.219E+01	4.219E+01	-7.292E+00	2.548E-02	4.583E+01
600.00	4.235E+01	4.235E+01	-6.999E+00	2.953E-02	4.655E+01
800.00	4.274E+01	4.274E+01	-6.431E+00	3.930E-02	4.789E+01
1000.00	4.306E+01	4.306E+01	-6.078E+00	4.695E-02	4.913E+01
1200.00	4.326E+01	4.326E+01	-5.891E+00	5.158E-02	5.033E+01
1500.00	4.351E+01	4.351E+01	-5.703E+00	5.670E-02	5.207E+01
2000.00	4.392E+01	4.392E+01	-5.468E+00	6.380E-02	5.486E+01

The kinetic parameter fit is for uni-molecular reaction.

The 3 parameters for the model equation of A(T) = Aprime * T^n * exp(-Ea/RT)

Aprime = 5.3804E+06 n = 1.65068 Ea = 4.1641E+04
kI = 5.36E+06 T^1.65068 exp(-41.64/RT) Ea in kcal/mol

Temp(K)	AF(T)	T_K^n	k_calc(T)	k_fit
300.00	1.768E+11	1.224E+04	2.994E-20	3.025E-20
400.00	1.980E+11	1.967E+04	1.910E-12	1.868E-12
500.00	2.655E+11	2.843E+04	9.620E-08	9.591E-08
600.00	3.692E+11	3.840E+04	1.383E-04	1.401E-04
800.00	6.550E+11	6.174E+04	1.376E+00	1.395E+00
1000.00	9.783E+11	8.922E+04	3.799E+02	3.801E+02
1200.00	1.290E+12	1.205E+05	1.702E+04	1.688E+04
1500.00	1.772E+12	1.742E+05	8.092E+05	8.022E+05
2000.00	2.659E+12	2.800E+05	4.216E+07	4.240E+07

k(calc) is the absolute k calculated from transition state theory
k(fit) is result of a mathematical fit to this data using the modified (3 - parameter) Arrhenuis expression above:
k = A T^n exp(-Ea/RT) if Ea in cal/mol R = 1.98cal/mol-K

Table A1. (continued) Thermodynamics and Kinetics of VX Unimolecular Decomposition Reactions

```
THERMODYNAMIC and KINETIC ANALYSIS for REACTION
VX => TSII    C2H4 + (CH3)P(OH)(=O)(S-C2H4-N(iprop)2
kII = 1.02E+09 T^0.49842 exp(-62.68/RT)   Ea in kcal/mol

Hf {Kcal/mol}      .000      62.560
S {cal/mol K}   105.150      92.990

dHr {kcal/mol}    (298K) =       62.56     dHr avg (298., 1500. K) =      62.17
dU (dE) {kcal/mol} (")   =       62.56     dUr avg (298., 1500. K) =      62.17
dSr {cal/mol K}   ( " )  =      -12.16     dSr avg (298., 1500. K) =     -13.04
dGr  {kcal/mol}   ( " )  =       66.19     dGr avg (298., 1500. K) =      73.89
```

T (K)	dH(Kcal/mol)	dU(Kcal/mol)	dS(cal/mol K)	Kc	dG(Kcal/mol)
300.00	6.256E+01	6.256E+01	-1.217E+01	5.801E-49	6.621E+01
400.00	6.240E+01	6.240E+01	-1.260E+01	1.403E-37	6.744E+01
500.00	6.218E+01	6.218E+01	-1.311E+01	9.029E-31	6.873E+01
600.00	6.194E+01	6.194E+01	-1.354E+01	3.001E-26	7.006E+01
800.00	6.160E+01	6.160E+01	-1.403E+01	1.266E-20	7.283E+01
1000.00	6.153E+01	6.153E+01	-1.412E+01	2.920E-17	7.565E+01
1200.00	6.163E+01	6.163E+01	-1.403E+01	5.106E-15	7.847E+01
1500.00	6.179E+01	6.179E+01	-1.391E+01	9.042E-13	8.265E+01
2000.00	6.186E+01	6.186E+01	-1.387E+01	1.617E-10	8.959E+01

The kinetic model fit is for uni-molecular reaction.

The 3 parameters for the model equation of kII = Aprime * T^n * exp(-Ea/RT)

Aprime = 1.02E+09 n = .49842 Ea = 6.2681E+04
kII = 1.02E+09 T^0.49842 exp(-62.68/RT) Ea in kcal/mol

Temp(K)	AF(T)	T_K^n	k_calc(T)	k_fit
300.00	1.371E+10	1.716E+01	3.626E-36	3.771E-36
400.00	1.470E+10	1.981E+01	1.169E-24	1.136E-24
500.00	1.420E+10	2.214E+01	9.407E-18	8.991E-18
600.00	1.372E+10	2.425E+01	3.752E-13	3.631E-13
800.00	1.428E+10	2.799E+01	2.110E-07	2.141E-07
1000.00	1.705E+10	3.128E+01	6.084E-04	6.367E-04
1200.00	2.143E+10	3.425E+01	1.277E-01	1.339E-01
1500.00	2.848E+10	3.828E+01	2.826E+01	2.873E+01
2000.00	3.884E+10	4.419E+01	6.739E+03	6.368E+03

k(calc) is the absolute k calculated from transition state theory
k(fit) is result of a mathematical fit to this data using the modified (3 - parameter) Arrhenuis expression above:
k = A T^n exp(-Ea/RT) if Ea in cal/mol R = 1.98cal/mol-K

Acknowledgement: The assistance of NJIT Research Professor Dr. Chiung-Ju Chen in running the density functional calculations and the data workup is appreciated.

B

APPENDIX B

SPECIAL REPORT DEVELOPMENT AND TESTING OF A VX PROBE FOR THE MPF DISCHARGE AIRLOCK

SwRI Memorandum

SW-I0301701 07 January 2003
MEM-03-30259

TO: Stephen C. DePew, WDC Project General Manager

FROM: Nollie F. Swynnerton, Laboratory Manager

SUBJECT: DEVELOPMENT AND TESTING OF A VX PROBE FOR THE MPF DISCHARGE AIRLOCK

Attached is a special report titled "Development and Testing of a VX Probe for the MPF Discharge Airlock," prepared by SwRI for WDC. The JACADS Laboratory requests that if the document is found acceptable it be transmitted by WDC to Mr. Gary McCloskey of PMCSD, with copies to Mr. Michael Hopcus of the PMCSD Field Office and Mr. Rob Malone of the local SAIC office. Point of contact is Dr. Nollie Swynnerton, ext. 3369.

SW-I0301701/NFS/mif
MEM-03-30259

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cc: *Leo O'Shea, Closure Mgr, JACADS
*Michael G. MacNaughton, SwRI-SAT
*Dennis Forsberg, WDC-JI
*Angelo Angelopolis, WDC-JI
*Plant Manager, WDC-JI
*AMD Manager, SwRI-JI
*Paul Weader, WDC-JI
*DCC-JI (2 copies)

*with Attachments

DEVELOPMENT AND TESTING OF A VX PROBE FOR THE MPF DISCHARGE AIRLOCK

SPECIAL REPORT

Subcontract 9900030
Prime Contract DAAA-09-96-C-0081
SwRI Project 01-5935.01
SW-I0301701
MEM-03-30259

Prepared By

SwRI JACADS Laboratory

Prepared For

Washington Demilitarization Company

January 2003

SOUTHWEST RESEARCH INSTITUTE®
JACADS

BACKGROUND AND INTRODUCTION

Following the incident at JACADS on 12 August 2002 in which agent-containing sludge was improperly loaded onto a tray resulting in incomplete treatment of the chemical agent, attention was focused on the monitoring configuration at the Metal Parts Furnace Discharge Airlock (MPF DAL). This change is focus was necessary because the investigation team could not readily identify the root cause of why the tray was monitored by two complete ACAMS cycles without detecting chemical agent. Although chemical agent VX was detected by both the ACAMS and the DAAMS, after the tray was returned to the DAL, direction was received from the PMCSD Field Office to move the V-G conversion pad assembly (CPA) nearer to the distal end of the probe (consistent with the DHHS-approved common stack method) and to challenge the systems through the entire sampling system. At the time of the incident, the CPA was positioned approximately 20 inches from the distal end of the probe. It had been placed there to allow the hot gases from the DAL to cool so that the V-G conversion pad and the Teflon inline filter would remain viable. The 18-inch length of stainless steel tubing served as both a heat exchanger and a probe. Prior to installation of this probe, V-G pads were observed to darken, become brittle and shed small black particles; the Teflon filters would lose their integrity and allow particulate matter into the ACAMS and DAAMS. With the probe/heat exchanger in place, temperatures at the filter measured 110 ºF, down from approximately 180 ºF. Temperature of the CPA was estimated at 120 ºF.

After the 12 August incident, sampling procedures of the MPF DAL were changed to positively preclude monitoring at exhaust gas temperatures exceeding 259 ºF. In addition, following guidance from PMCSD, the CPA was moved to five inches from the distal end of the probe. Performance statistics of the ACAMS monitoring the MPF DAL immediately began to decline. The challenge now became one of developing a VX sampling system that was reliable and robust with the V-G pad near the distal end of the probe.

MODIFICATIONS TO THE SYSTEM

A test sampling system mimicking the actual monitoring system was set up on the DAL exhaust duct and fitted with thermocouples to measure temperatures of the duct, the V-G pad, the filter and the gas as it entered the ACAMS/DAAMS. Baseline temperatures under normal operating conditions were measured over two two-day periods, 16-17 October 2002 and 18-19 October 2002, and are shown in Table 1. During this time the solenoids controlling the timing and duration of sample flow were actuated when the duct gas cooled to 259 ºF and simulated sampling (pump flow) was conducted for 28 minutes.

The test probe was then repositioned, moving its distal end from the center of the 6-inch DAL exhaust duct to 0.5 inches from the duct's inner wall. This moved the CPA an additional 2.5 inches away from the hot duct and allowed it to remain cooler, at the same time preserving the mandated position of the CPA. Engineering calculations by Dr. Roger Lucheta, JACADS engineer, indicated that wall effects were negligible at this position (Appendix C). Temperature effects of this change were measured over the two-day period 26-27 October 2002. The table below shows the average temperature results of this modification. Important to note is that the actual sample flow rate for these three periods is not known with certainty. The flow indicator read 6 L/min but it was later determined that the actual flow was less because of a leak downstream of the CPA. Later testing showed the important combined effects of sample flow rate and temperature on the lifetime of the V-G pad.

Dates Measured	T_{CPA} [1,2] ºF	T_{Filt} [3] ºF	T_{ACAMS} [4] ºF
16-17 October 2002[5]	244	112	83
18-19 October 2002[5]	248	120	87
26-27 October 2002[6]	204	112	88

1. Temperature of duct gas at start of sampling was 259 ºF
2. Temperature of gas at the CPA
3. Temperature of gas at the Millipore filter at proximal end of probe
4. Temperature of gas entering the ACAMS
5. Distal end of probe at midpoint of 6-inch duct
6. Distal end of probe repositioned to 0.5 inches from duct wall.

Although continuous monitoring of the CPA temperature showed it did not exceed 210 ºF at this configuration *during sampling*, the temperature of the CPA exceeded 300 ºF during the periods *between sampling*. This temperature increase, or "heat soak" occurred when a hot WIC was introduced into the DAL and before the WIC was cool enough to sample. During this event, duct gas temperatures could approach 600 ºF, depending on the load in the WIC. To minimize the heat soak and cool the CPA during the non-sampling periods a stream of cool air was introduced at the proximal end of the probe. This flow was directed toward the duct and over the CPA and sample line during periods when the DAL was not being sampled.

After these changes were made to the monitoring system (Station 67D, ACAM-155) on 2 November, performance statistics on the DAL ACAMS significantly improved (1 Nov.–79%, 15 Nov.–90%, 29 Nov.–98%) and performance criteria are now being comfortably met.

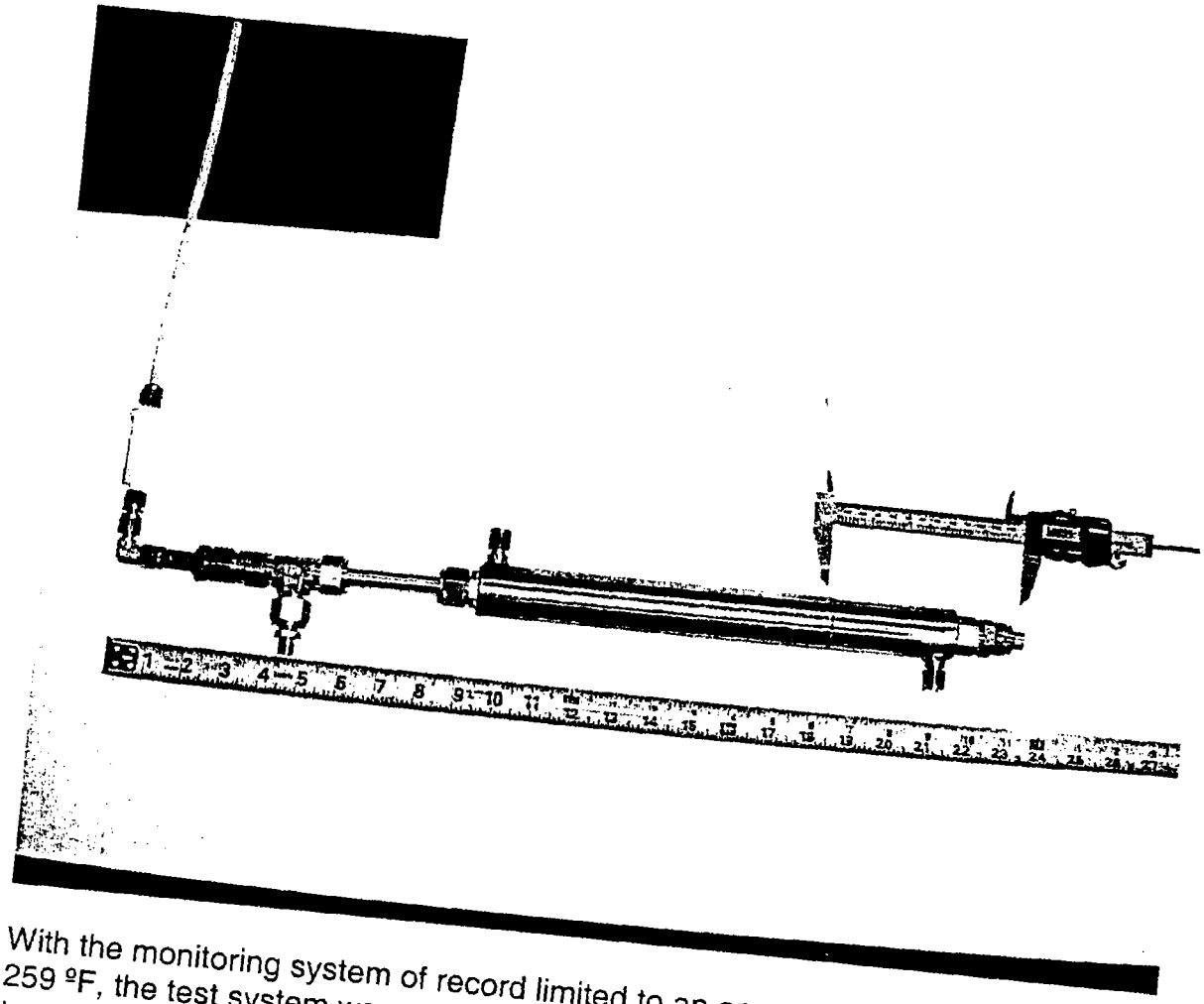
FUTURE OPERATING CONDITIONS OF THE MPF DAL

The Laboratory anticipates that higher temperatures will ultimately be allowed when sampling the DAL. Preliminary tests in our laboratory demonstrated that the ability of the V-G pad to function well is compromised by a continuous 2-hour exposure to temperatures exceeding 200 ºF. Under current operating conditions in the MPF, the V-G pad remains viable for at least 12 hours, but this stability cannot reasonably be expected to continue if duct gas temperatures approach or exceed 500 ºF.

DEVELOPMENT OF A WATER-COOLED PROBE FOR VX SAMPLING OF THE MPF DAL

As noted above, introducing the stainless steel probe and placing the CPA 20 inches from its distal end achieved a dramatic decrease in temperature at the CPA. Cooling was provided by the probe's exposure to ambient air over about 14 inches of its length. See the above table. However, the 20 inches raised concerns that that VX transmission had not been demonstrated at this distance. After the CPA was moved to five inches from the probe's distal end, simply by moving the probe's terminus from the center of the duct to 0.5 inches from the inner wall provided a substantial reduction in CPA temperature. This configuration is satisfactory for 259 ºF. At higher temperatures, to satisfy both the need for a reduced temperature at the CPA and to maintain a 5-inch distance from the probe tip, a more effective cooling technique was required.

The Laboratory and WDC welders fabricated several types of water jackets to enclose the probe and evaluated them in the test sampling system previously located on the duct of the MPF DAL during periods of routine MPF operation. The greatest cooling effect was provided by a full-length jacket with both the water inlet and outlet near to the ends of the jacket. Swagelok® unions and fittings were modified to allow the jacket to fit closely to the duct to provide maximum cooling to the probe. Cooling water was introduced at the end of the jacket nearer the duct. The cool-air blowback system described earlier was retained (see photograph of probe system below).



With the monitoring system of record limited to an operational temperature of 259 ºF, the test system was reconfigured to operate under the higher temperatures experienced during the full DAL cycle. A second test system was fabricated and installed to allow simultaneous testing of ACAMS and DAAMS flow rates. A separate set of solenoids was installed and set to begin sampling one minute after the door opened between the DAL and the third zone of the MPF. The timer was set to close the solenoid 30 minutes later. Both settings were arbitrary but approximated the expected worst case sampling conditions. Temperatures were measured by thermocouples installed 1) in the duct, 2-3 feet upstream from the probe penetrations, and 2) immediately behind the V-G pad (<0.125 inches from the pad). Flow of the sample gas was controlled and measured.

While testing, cooling water temperature and sample gas-flow rates were varied. The CPA was evaluated by periodically removing the pads from the test probes and challenging an ACAMS through the exposed pads. This was done for both ACAMS and DAAMS test stations.

In this configuration, gas temperatures near 600 ºF were reduced to 125 ºF at the CPA with a flow of 0.8 L/min. Temperature of the process cooling water averaged 80 ºF, warm enough to prevent condensation of either water or VX. Under these conditions the CPA remained viable for at least 24 hours. At higher flow rates the cooling was less effective and DAAMS operation at this station must be carefully considered. Challenge data for both systems were statistically treated in the same manner as the operational ACAMS challenge data; therefore, they could be analyzed using the standard INACCMO program specified in the LQAP. A statistical treatment of the data is included in this report as Appendix A.

Data on the water-cooled probe design that was the most successful in reducing temperature at the CPA are limited by the short investigation period; however, the following conclusions can be drawn with some measure of confidence. Data were collected through 03 January 2003, and are attached as Appendix B.

- Performance of the ACAMS easily meets JACADS performance criteria
- Performance of the experimental DAAMS at sample flows of 4.2- and 3.0 L/min is superior to that of the present DAAMS station-of-record, even though it is ingesting a much hotter sample gas at the probe.
- With the water-cooled system and the CPA 5 inches from the distal end of the probe, a flow of 4.5 L/min appears to be the maximum flow rate allowable to prevent CPA degradation. This configuration requires a minimum of four VX probes to maintain the current DAAMS sampling system of 12 total tubes.

Testing of the water-jacketed probe system will continue and baseline data will be analyzed to further prove the utility of the systems.

Recommendations

Based on the investigation of the DAL VX probe configuration it is recommended that the water-cooled probe be implemented if the monitoring temperature limits are raised from the present 259 ºF. The water-cooled probe was found to reduce the temperature of the gas at the CPA of the ACAMS, at a 5-inch distance, down to 125 ºF where it successfully met the performance criteria required of operational monitoring systems.

Ongoing studies at SwRI in San Antonio are investigating the ability of the 18-inch air-cooled probe to transmit VX at 600 ºF. If these tests demonstrate that the air-cooled probe is successful, this would be the preferred option, since it would be less complex and maintenance-intensive than the water-cooled probe. However, until this option is validated, the water-cooled probe is a near-term solution to sampling VX from the DAL should the temperatures limitations be lifted.

APPENDIX A

Statistical Analysis of MPF ACAMS and DAAMS Quality Samples

Statistical Analysis of MPF ACAMS and DAAMS Quality Samples

For the ACAMS, based on 27 challenges (one was excluded because of no cooling water flow), the statistical response rate was 100% with mean = 98% and fairly low variability.

Following is the robust statistical summary of the recoveries at the MPF duct DAAMS.

Station	Flow	N	ROB_MEAN	ROB_SD	SRESP
67D	A	21	61.75	37.06	41
67D	B	13	86.92	22.24	78
67D	C	10	87.41	35.58	69
67X	A	20	69.42	25.95	49
67X	B	13	94.08	7.41	100
67X	C	10	100.30	17.05	96

Flow A is the first, uncontrolled-flow period, B is when the flow was stabilized at 4.2 L/min and C is the time when the flow was reduced to 3.0 L/min.

It is clear that 67X did better during each time period than 67D, particularly the latter two periods. There is not enough data for statistical significance as the variabilities are so large, but the trend is very strong. Note that the flow rate was not adjusted at the DAAMS of record, Station 67D. The results are merely given in order to compare the performance of 67X at various flow rates with a base station that took readings during the same period.

Even though the mean recoveries got better after reducing the flow rate, the statistical response rate went down a bit because the variability went up along with the mean. Also, 10 data points isn't a heck of a lot, so there is some sampling variation as well. Regardless, we cannot say that anything improved by reducing the flow rate. The only thing we can say from these data is that period A was definitely worse than afterwards.

As far as comparing the two stations goes, the following table shows the mean of the differences (i.e., the mean of 67X minus 67D at each time period):

Flow	# Obs	N	Mean	Std Dev	Min	Max
A	21	20	6	30	-30	74
B	13	13	10	29	-44	83
C	10	10	15	34	-20	71

The way to read this is, when the flow was during the A period, there 21 CPA changes with one value missing from the 67X station (so 20 total), the mean of the 20 67X - 67D values was 6.45, the standard deviation was 30.44, the smallest difference was -30 (i.e., 67D was 30 points larger than 67X) and the largest difference was 74 points. Please note that even though it looks like changing the flow from 4.2 to 3.0 increased the difference between the 67X and 67D readings, this is not significant relevant to the variability. In other words, we can't say that the 67X minus 67D difference was larger on average during the 3.0 period than during the 4.2 period.

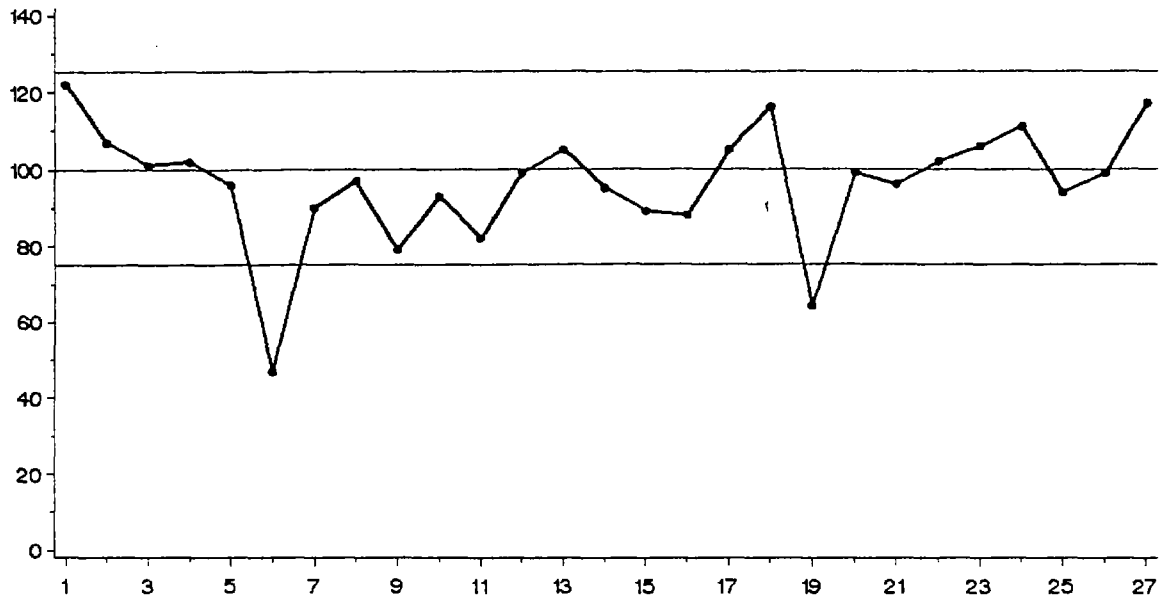
Looking at it non-parametrically, the following summarizes the number of times 67X had a recovery larger than 67D:

Flow = A	11/20 (55%)
Flow = B	10/13 (77%)
Flow = C	5/10 (50%)

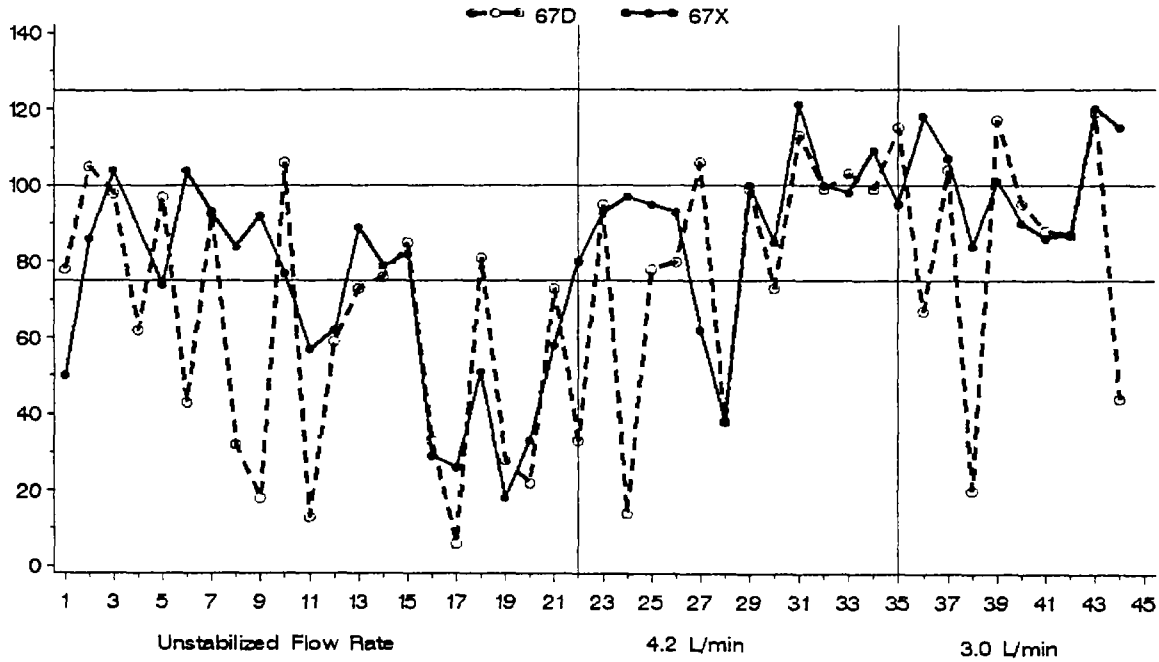
These results conflict with the mean differences because the counts imply B is better than C in terms of the 67X being larger than 67D. The means were thrown off a bit by an outlier during the B period that drug that difference down, while the counts are not significantly different between the B and C periods. In other words, I can't prove the difference between the improvement of 67X during the C period was any larger than the difference during the B time frame.

However, the conclusions still stand - 67X has been performing better than 67D. Overall, 26/43 (60%) of the readings had 67X larger than 67D.

MPF ACAMS Station 67Y Recoveries versus Time Index



MPF DAAMS Recoveries versus Time Index



APPENDIX B

Raw Data for the Statistical Analysis of MPF ACAMS and DAAMS Quality Samples

Station 67 Test Probe CPA Recoveries; Data through 03 Jan 2003

Station 67Y - MPF DAL ACAMS test probe

Data Point Number	Time CPA installed (approx.)	Number of WICS processed	Sample Flow rate through CPA (L/min)	max MPF DAL temp (deg F)	max Temp at CPA (deg F)	Comments	67Y CPA Challenge (VX TWA)
1	1400	8	0.8 2 L/min in purge	600	160	shut down after this test to focus on 67X	1.22
2	1300	3	0.8	450	115	no chiller on ACAMS probe	1.07
3	1915	5	0.8	one tray= 600 four= 450	100	Attempted to challenge thru system but only had a 0.13 recovery	1.01
4	0800	5	0.8	one tray= 600 four= 450	120		1.02
5	1920	6	0.8	590	120		0.96
6	0930	4	0.8	585	110		0.47/0.32
7	1930	5	0.8	585	110		0.90
8	0800	4	0.8	610	105		0.97
9	1912	5	0.8	580	110		0.79
10	0740	6	0.8	600	115		0.93
11	2130	3	0.8	600	100		0.82
12	0730	2	0.8	535	110		0.99
13	0815	3	0.8	600	115		1.05
14	1850	7	0.8	615	100		0.95
15	0900	5	0.8	585	1= 190 4= 100	1300= Chiller installed onto probe, CPAs hit high temp.	0.89
16	1950	5	0.8	585	100		0.88
17	0700	6	0.8	580	100		1.05
18	1930	5	0.95	600	135		1.16
19	0950	5	0.95	590	125	1105= chiller pump stopped working, plant process water installed	0.64
20	1915	5	0.95	615	120		0.99
21	0715	5	0.8	595	80		0.96
22	1925	1	0.8	440	110		1.02
23	0715	3	0.8	570	115		1.06
24	1925	5	0.8	635	120		1.11
25	0715	6	0.8	580	115		0.94
26	1925	5	0.8	570	115		0.99
27	0710	6	0.8	600	120		1.17

Station 67 Test Probe CPA Recoveries; Data through 03 Jan 2003

Station 67X - MPF DAL DAAMS test probe

Data Point Number	Duration CPA sampled	Number of WICS processed	Sample Flow rate through CPA (L/min)	max MPF DAL temp (deg F)	max Temp at CPA (deg F)	Comments	DAAMS of record CPA challenge (VX TWA)	67X CPA Challenge (VX TWA)
1	21 hrs 30 min	6	3.5	600	240		0.78	0.50
2	20 hrs 30 min	11	3.0	1 WIC= 580 10 = 450	1 WIC=190 10 =160		1.05	0.86
3	13 hrs 30 min	6	4.0	450	180	Chiller started at 1400 (chiller set at 70 degrees)	0.98	1.04
4	15 hrs 15 min	8	4.0	1 WIC= 625 7 = 450	1 WIC= 220 7= 180		0.62	NA
5	7 hrs 30 min	4	4.5	1 WIC= 585 3= 450	1 WIC= 240 3= 160	1330= set chiller to 60 degrees. 1915= Noticed condensation in probe	0.97	0.74
6	12 hrs 45 min	5	2.5	1 WIC= 600 4= 450	115* *low flows	Flow problem due to condensation; Chiller readjusted to 70 degrees	0.43	1.04
7	11 hrs 20 min	5	3.5	1 WIC= 600 4= 450	180		0.93	0.93
8	14 hrs 10 min	6	4.5	590	190		0.32	0.84
9	10 hrs	4	6.0	585	230		0.20/0.18	0.94/0.92
10	12 hrs 30 min	5	5.0	585	190		1.06	0.77
11	11 hrs 12 min	4	4.5	1 WIC= 610 3 = 445	1= 230 3= 180		0.13	0.57
12	12 hrs 28 min	5	5.5	580	220	When checking the flow on the chiller, the pump stopped working. hooked up the unchilled water to the probe.	0.59	0.62
13	13 hrs 50 min	6	5.0	600	200		0.73	0.89
14	10 hrs	3	4.5	600	220		0.76	0.79
15	12 hrs 20 min	2	5.5	535	220		0.85	0.82
16	12 hrs 25 min	6	4.5	590	220		0.33	0.29
17	10 hrs 35 min	3	6.5	600	255		0.06	0.26
18	14 hrs 10 min	7	4.5	615	180		0.81	0.51
19	10 hrs 50 min	5	4.5	585	1 WIC= 300 4= 180	1300= Chiller installed onto probe, CPAs hit high temp.	0.28	0.18
20	6 hrs 5 min	2	6.5	585	220		0.22	0.33
21	5 hrs 5 min	3	7.5	585	230		0.73	0.58
22	6 hrs	3	1 WIC= 6.5 2= 4.2	580	1 WIC= 230 2= 160	1010= installed flow controller on the pump to regulate flow rate to 4.2 L/min	0..33	0.80

Station 67 Test Probe CPA Recoveries; Data through 03 Jan 2003

Station 67X - MPF DAL DAAMS test probe (continued)

23	6 hrs 30 min	3	4.2	570	175		0.95	0.93
24	7 hrs	2	4.2	600	180		0.14	0.97
25	7 hrs 20 min	3	4.2	600	195		0.78	0.95
Data Point Number	**Total duration CPA sampled**	**Number of WICS processed**	**Sample Flow rate through CPA (L/min)**	**max MPF DAL temp (deg F)**	**max Temp at CPA (deg F)**	**Comments**	**DAAMS of record CPA challenge (VX TWA)**	**67X CPA Challenge (VX TWA)**
26	5 hrs 28 min	3	4.2	445	160	1105= chiller pump stopped working, plant water installed	0.80	0.93
27	3 hrs 57 min	2	4.2	590	190		1.06	0.62
28	6 hrs	2	4.2	585	190		0.38	0.38
29	6 hrs	3	4.2	615	220		0.99	1.00
30	6 hrs	2	4.2	595	205		0.73	0.85
31	6 hrs 10 min	3	4.2	585	210		1.13	1.21
32	6 hrs 5 min	1	4.2	440	170		0.99	1.00
33	5 hrs 45 min	0	0	110	80		1.03	0.98
34	6 hrs	0	0	95	80		0.99	1.09
35	6 hrs 10 min	3	3.0	570	175	Changed flow rate to 3.0 L/min	1.15	0.95
36	6 hrs	2	3.0	610	170		0.67	1.18
37	5 hrs 50 mn	3	3.0	635	180		1.04	1.07
38	10 hrs 10 min	4	3.0	580	180		0.20	0.84
39	2 hrs	2	3.0	565	150		1.17	1.01
40	6 hrs	2	3.0	570	150		0.95	0.90
41	5 hrs 45 min	3	3.0	470	140		0.88	0.86
42	5 hrs 50 min	3	3.0	600	160		0.87	0.87
43	7 hrs	3	3.0	600	160		1.19	1.20
44	6 hrs	2	3.0	585	*	* Chart recorder malfunctioned	0.44	1.15

APPENDIX C

Calculation of Wall Effects

(AVAILABLE UPON REQUEST)

C

APPENDIX C

TEST PLAN VX PROBE TRANSMISSION AND CPA LIFE

TEST PLAN
VX PROBE TRANSMISSION AND CPA LIFE

1. Introduction

Southwest Research Institute (SwRI) has been tasked to investigate the performance of stainless steel probes and AgF conversion pad assemblies (CPAs) when used to sample VX in high temperature atmospheres such as the Metal Parts Furnace (MPF) discharge airlock (DAL). This task is being conducted in support of the Johnston Atoll Chemical Agent Disposal System (JACADS) operations.

This test plan is prepared to present the approach to conducting this investigation and to serve as work instruction for laboratory operations during the period of performance. Investigation will be conducted at SwRI laboratories located in San Antonio, Texas.

2. Background

The JACADS MPF DAL monitoring system utilizes the ACAMS to collect and analyze samples of the DAL atmosphere to provide additional assurance that workers on the cool down pad will not be exposed to agent if there is a failure in the MPF process. This information is used to verify that conditions are safe to transfer material into or out of the DAL. The DAL environment spans extreme temperature conditions that range from ambient to over 300°C.

The ACAMS method for the agent VX incorporates a derivatization technique in which VX is converted to a stable and volatile compound that is easily and reliably measured using gas chromatography (GC). The MINICAMS incorporates concentration techniques using solid sorbent collection media. Concentration of the target analyte is followed with thermal desorption of the collected material into a GC. The derivatization is accomplished by passing the VX-contaminated air stream through a polyester pad that has been impregnated with silver fluoride (AgF) and located at the inlet to the sampling line.

The ACAMS pulls gas samples from the DAL exhaust duct through a stainless steel probe prior to conversion of the VX to a more volatile G-analog. There has been concern that VX will degrade or condense on the probe prior to conversion. The AgF pad itself and chemical reactions required for derivatizing VX have also been shown to degrade under conditions of high temperature. Prior studies on the common stack validated a 5-inch probe length and that distance is now in use at the DAL under restricted temperature conditions. Under normal operating conditions it has been shown that at a 5-inch setback the AgF pad rapidly loses conversion efficiency. The purpose of this study is determine the transfer efficiency for the stainless steel probe under normal operating conditions, the temperature limits for the existing CPA, and to evaluate alternate support materials for the AgF pad to increase the temperature limits.

3. Objectives

The objectives of the task include:

- Determine the transmission efficiency of the ½-inch stainless steel probe while sampling a 600°F atmosphere.
- Determine the life of the existing CPA for various times and temperatures.
- Evaluate other CPA materials to improve the CPA life at temperatures up to 600 °F.

4. Approach

Testing will be conducted using heated breathing air into which an amount of VX vapor will be injected. Previous probe studies have used ambient temperature gas atmospheres containing small concentrations of carrier solvents. This has raised concern that the data is not representative of the DAL gas stream. Therefore to eliminate the change that solvents enhance the transmission of VX through the probe neat VX will be used to generate the challenge atmosphere. The test configuration is shown in Figure 1.

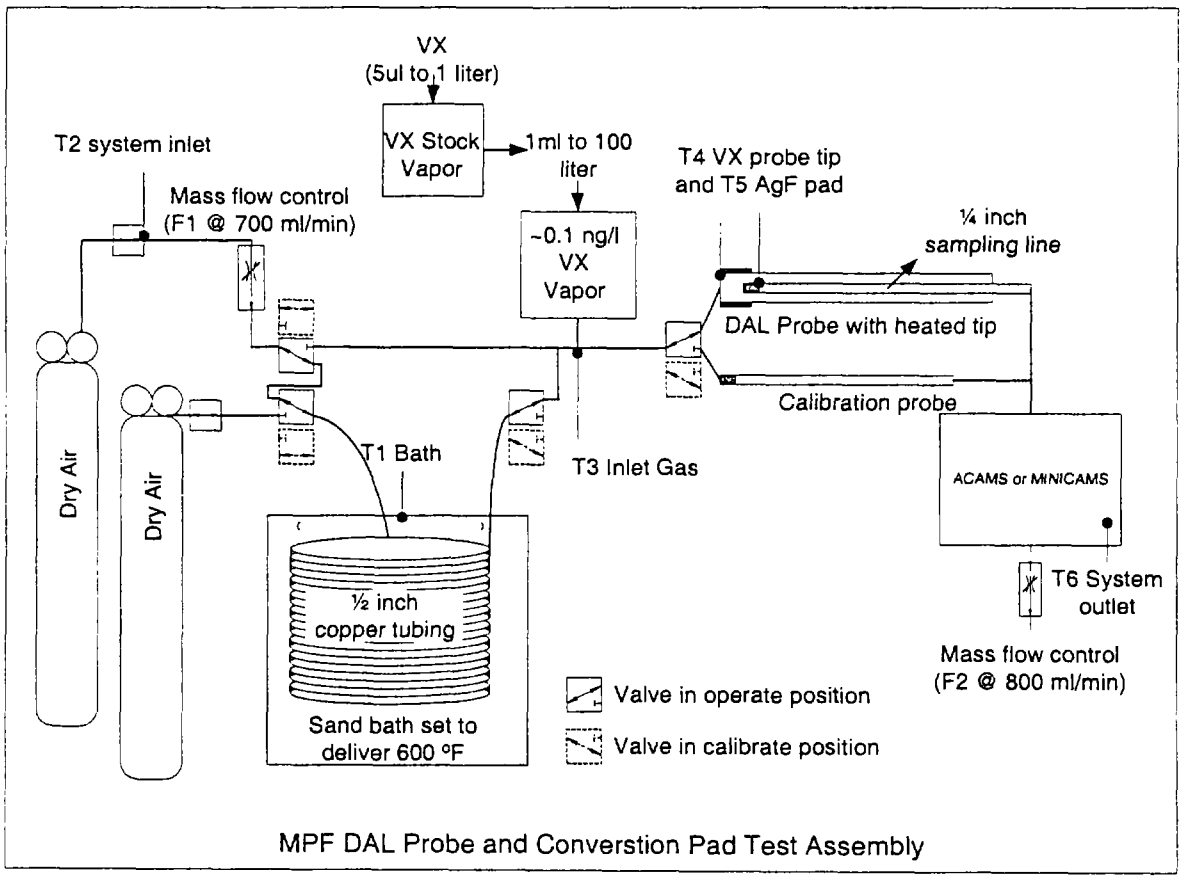


Figure 1. Test Assembly

4.1 VX Vapor Generation

To avoid the presence of carrier solvents neat VX will be used to generate the challenge atmosphere by serial dilution. Stock Atmosphere 1 will be generated using a 1-liter Tedlar bag in to which 5µl of neat VX has been injected. Assuming saturated conditions a headspace concentration of approximately 11 mg/m^3 (11 ug/l) should be established in this bag. After spiking with 5µl VX the bag will be filled using breathing air. The resulting concentration of the bag headspace should be in the range of 10 ug/l. To minimize the presence of the VX-like volatile contaminants of the neat VX in the atmosphere, the stock CASRM solution will be degassed prior to withdrawal of the 5 µL.

From this Stock Atmosphere 1, further dilution will be made to achieve the desired concentration in the challenge gas to be mixed with the heated breathing air. A Stock Atmosphere 2 will be made by adding 1ml (10 ng VX) of the Stock Atmosphere 1 into 100 L of breathing air. The resulting concentration of this VX vapor source will be 10ng/100L or 0.10 ng/L. (10 TWA).

The Stock Atmosphere 2 will be blended approximately 1/8 with heated (600 °F) breathing air to achieve the final challenge gas introduced into the probe.

4.2 Final Challenge Atmosphere Generation

A sand bath that contains a ½-inch coiled copper tube will be used to heat bottled breathng air to 600°F. A mass flow controller will maintain flow rate through the copper tube at 0.7 Lpm. This 0.7 Lpm stream will be mixed with 0.1 Lpm of the VX vapor stream (Standard Atmosphere 2).

The VX vapor stock will be added to the 0.7 Lpm stream at a rate controlled by the MINICAMS flow rate. The MINICAMS will sample at a rate of 0.8 Lpm. While supplying 0.7 Lpm of heated gas to the MINICAMS, the remaining 0.1 Lpm will be drawn from the VX vapor source bag. The key to controlling this atmosphere concentration is the ability to maintain the MINICAMS and heated gas flow rates with mass flow controllers.

4.3 MINICAMS Configuration

The test apparatus will use a MINICAMS rather than an ACAMS as is used at JACADS. The MINICAMS is a commercial equivalent of the ACAMS that uses the same operating principles. The MINICAMS will be modified to incorporate a 20 mm PCT to minimize the breakthrough of the VX that has been reported by other researchers with the shorter PCT. The purpose of the test is to determine the relative concentrations of the VX before and after the probe so any possible differences between the ACAMS and MINICAMS response will not bias the results.

The SS probe inlet will be heated to 600°F for the first few inches but left uncovered to duplicate the configuration at the JACADS DAL. The MINICAMS sampling probe is a

½-inch stainless tube through which a ¼-inch sampling line is run. At the end of this ¼-inch sampling line, the AgF pad is located and positioned by moving the line toward or away from the ½-inch inlet. During testing, the ¼-inch line will be positioned at varying distances within the ½-inch probe.

4.4 Concentration Determination

Although it is intended to maintain a stable challenge atmosphere slight changes in the Stock Atmosphere headspace or dilution ratio may cause changes in the challenge gas concentration. To eliminate this as a variable, the challenge concentration will be monitored directly using the same MINICAMS (control). Since the effect of temperature on the CPA is another variable the control will be monitored in the ambient temperature dry gas bypassing the heated sand bath.

4.5 Probe Testing

The first testing will address the ability of the ½-inch probe to transmit the VX challenge atmosphere. Used probes from JACADS that have been used to sample the DAL exhaust duct for one week will be used for the test. This will duplicate to the greatest extent possible the operating conditions at JACADS.

Testing will begin at a probe tip to CPA distance of 18 inches. The testing sequence will be 1 control cycle and 4 probe cycles. If the probe recovery is the same as the control then the probe testing is assumed to be complete. If the recovery is degraded then the probe will be moved to 12 –inches and the test repeated. If recovery is still degraded the probe will be move to 5 inches. If the 18-inch probe transmission tests do not show significant loss of recovery then consideration will be given to increasing the distance or decreasing the temperature to assess the ability of the probe to transmit VX longer distances at lower temperatures. At the completion of the test sequence the CPA will be removed and tested using a standard liquid challenge.

It should be noted that previous testing at SwRI and Southern Research Institute has documented that the CPAs degrade quickly when they are first used. It is also noted that the injection of an IPA solution appears to "regenerate" the pad. This phenomenon is recognized and will be accounted for in the test design and data interpretation.

4.6 CPA Testing

Testing will begin at a probe tip to CPA distance of 18 inches: Testing will duplicate the normal JACADS DAL cycle which is anticipated to be 28 minutes or a maximum of 4 instrument cycles. The stability of the gas stream will be verified prior to initiation of the test run. Three MINICAMS cycles must be within ± 10 RPD prior to start of the first sequence. The sequence will be 1 control cycle and 4 probe cycles for 10 sequences (40 probe and 11 control). This is equivalent to the normal 12-hour life of the CPA. At completion of the 18-inch distance the CPA will be moved to 12 inches and 5 inches, and

the test repeated. At the completion of each test sequence the CPA will be removed and standard liquid challenged.

5 Data Collection

Data collected during testing will include flow rate, temperature, and MINICAMS results. The following data points are required:

Parameter	Source	Method	Frequency
Flow Rate			
F1	Atmosphere	Internal meter	Mid-cycle
F2	MINICAMS	External meter	Mid-cycle
F3	VX source	Calculated	Mid-cycle
Temperature			
T1	Sand Bath	Thermocouple	Pre, mid, and post
T2	Gas ambient	Thermocouple	Pre and post cycle
T3	Inlet Gas	Thermocouple	Pre and post cycle
T4	Probe Tip	Thermocouple	Pre, mid, and post
T5	AgF Pad	Thermocouple	Pre, mid, and post
T6	Gas ambient	Thermocouple	Pre, mid, and post

The probe and CPA recovery data will be analyzed graphically and statistically. The probe tests will be kept short to minimize the effect of CPA degradation. The recoveries will be analyzed by determining the mean, standard deviation, and relative standard deviation for the set of replicates for each configuration.

6 AgF Support Material Optimization

A literature search will be conducted to determine the four best options for materials to be tested as substrates for the AgF conversion pad to increase the temperature capabilities.

The addition of AgF to the selected materials will likely not be difficult, but determining the efficiency of each one will be more intensive. The usual method of impregnating AgF pads is to heat the substrate and then immerse in the AgF solution.

The testing must show that the material can withstand the conditions of the DAL, which are 600°F and low relative humidity. Given that low RH in the DAL effect of RH on the AgF substrates will not need to be investigated. The gas temperature will start at 600 °F to duplicate the worse case condition and then reduced to 500 °F, and 300 F°.

A stainless steel "tee" fitting will be used for the injection point. The heated gas will be introduced into the "tee" and the CPA challenged through a septum. The CPA will be challenged at 1, 2, 8, 12 hours.

The data from the existing CPA and the alternate materials will be analyzed statistically for mean, standard deviation, and relative standard deviation. The substrate that provides the best recovery at this point will be selected for more detailed testing to determine temperature/time/RH effects on VX recovery to determine the harshest conditions under which the substrate can provide reliable conversion efficiency.

7 Deliverables

A report of results will be submitted in draft form at the completion of the probe and APA pad stability testing. This report will include all data and observations in addition to recommendations for the monitoring configuration applicable to the MPF DAL.

DAL Probe Study

Draft Final Report
Subcontract 99000300
SwRI Project 01-05935

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1. INTRODUCTION

Southwest Research Institute® (SwRI®) was tasked to investigate the performance of stainless steel probes and silver fluoride (AgF) conversion pad assemblies (CPAs) when used to sample VX in high temperature atmospheres such as the metal parts furnace (MPF) discharge airlock (DAL). This task was conducted in support of the Johnston Atoll Chemical Agent Disposal System (JACADS) operations.

2. BACKGROUND

2.1 August MPF Event

The MPF was designed to process agent-contaminated residuals and up to 5X noncombustible material. This treatment has been demonstrated to destroy agent to below the waste control limit. As a final check, the DAL is monitored for agent vapors to provide a final safeguard against possible exposure of personnel from an agent release should a combination of factors result in incomplete destruction of chemical agent. The purpose of monitoring the DAL exhaust dust is not to assure that stack emissions are below a permit limit (the exhaust gases go through an afterburner) nor is it a direct measurement of the potential exposure that would occur to a worker exposure level on the cool down pad. The sole purpose of the monitoring is to provide a go/no go indication as to whether there has been a process failure to achieve 5X and, if not, to allow the tray to be discharged from the airlock (without creating a hazard for personnel on the cool down pad).

At JACADS on 12 August 2002, a tray (Tray 153) carrying improperly loaded spent decontamination solution (SDS) sludge exited the MPF DAL onto the cool-down conveyors (outside of the engineering controls of the Munitions Demilitarization Building, or MDB) after completing its twenty-two minute hold time with no agent alarms. Approximately 2 minutes after the tray was discharged, the Automatic Continuous Air Monitoring System (ACAMS) monitoring the DAL went into alarm indicating the possible presence of agent HD (mustard) and GB. After approximately 15 minutes, the control logic on the door to the DAL ~~was overridden~~cleared and the DAL door was opened and the tray fed back into the DAL, restoring it to engineering controls.

After the tray was returned to the DAL, the ACAMS monitoring the DAL was restarted and then went back into alarm for all three agents. Readings indicating the possible presence of all three agents continued, with potential agent readings for agent VX rising to a peak of 61.18 TWA. Plant operations personnel, fearing that the ACAMS would become saturated, and therefore useless for future analysis, switched the unit from monitoring the DAL to monitoring the MPF vestibule. Depot Area Air Monitoring (DAAMS) tubes were pulled and sent to the laboratory for analysis. The first set of tubes which sampled the DAL for the entire 22-minute hold time prior to discharge and for 11 minutes after monitoring was re-established in the DAL after the return of the tray refuted the ACAMS alarms for agents GB and HD but was indeterminate for VX. A second set of tubes which also sampled the DAL for the entire 22-minute hold time prior to discharge, as well as for 17 minutes after monitoring, was re-established in the DAL after return of Tray 153, also refuted the GB and HD readings but confirmed the presence of VX.

2.2 Root Cause Analysis

While the root cause of the failure to achieve 5X was the improper loading of the tray with SDS sludge, there was concern that the ACAMS had not alarmed during the monitoring cycle in the DAL. Three hypothesizes were proposed for the failure of the ACAMS to detect VX from the uncombusted agent: 1) the temperature in the DAL was sufficient to degrade the VX vapor before it could reach the monitoring system, 2) the sampling system prevented VX from reaching the ACAMS, and 3) the VX was "encapsulated "-in the sludge and was not released until the integrity of the sludge mass was disturbed.[1] Until an extensive investigation showed that thermal degradation of VX was not the cause of the alarm

failure, the operation of the DAL monitoring system was restricted to temperatures below 259 °F. Reaction Engineering International (REI) disproved the issue of the VX thermal degradation in a comprehensive analysis using state-of-the-art thermodynamic modeling[2] and EPA authorized return to the normal operation.

2.3 JACADS Monitoring System

At JACADS, the DAL monitoring system consists of three ACAMS that sample from the DAL exhaust duct. The parameters of the ACAMS have been optimized for the MPF DAL to minimize the false alarms due to interfering combustion-derived hydrocarbons in the MPF. This optimized ACAMS configuration is called ROBOCAMS. The ACAMS is a near-real-time instrument that collects, analyzes, and samples concurrently. The 6-minute sampling, 1-minute purge (when the collected sample is transferred from the pre-concentrator tube [PCT] to the analytical column) and approximately 4-minute analytical cycles guarantee that at least one full and one partial sampling cycle will be completed during the 22-minute residence time of a tray in the DAL. Overall, a full cycle takes 11 minutes. However, analytical results are reported every 7 minutes due to the overlap of the sampling of a second cycle with analysis and reporting from the first cycle. The sample is extracted from the exhaust duct of the DAL by a ½-inch stainless steel tube, passed through a Teflon filter and then transported through a 104.5-foot quarter-inch Teflon sample line to the ACAMS, where it is separated, detected, and quantified using a flame photometric detector. The analytical readout appears on a gas plasma display at the instrument and on a display in the CON. It is also recorded on PDARS. To prevent the monitoring system from being fouled by excessive particulates, the system has a solenoid valve at the end of the 104.5-foot sampling line that diverts the sampling location to the MPF room/vestibule when a tray is not in the DAL. For VX, the sampled gas is passed through an AgF CPA ~~just~~ prior to the filter.

2.4 Monitoring System Investigation

The conclusion of the monitoring system failure analysis was that the sampling system was not a significant contributor. A review of the quality data for the MPF DAL monitoring system found no performance deficiencies. The system had operated properly upon return of the tray to the DAL and the ACAMS had passed all performance criteria in the Laboratory Quality Control Plan (LQCP). The sampling lines were line-tested after the event and the test showed recovery of 106% for the VX line, 110% for the GB line, and 92% for the HD. The AgF CPA was removed from the VX sampling line and checked for contamination and conversion efficiency. No VX was detected on the pad, and the CPA had a conversion efficiency of 98% and 102% for two separate challenges.

2.5 Sampling Probe Concerns

Despite the results of the investigation monitoring system failure analysis, there was concern expressed that the ½-inch stainless steel probe inserted in the DAL duct may have contributed to the failure of the ACAMS to alarm during the first DAL cycle. Approximately 5 months prior to the event, the filter in the sampling line was added to prevent ultrafine particles from clogging the PCT on the ACAMS, especially when scabbled concrete is processed through the DAL. When the filter was added, the CPA and filter were moved from 6 inches to 18 inches from the distal end on 29 March 2002 to prevent the filter from melting. Prior to moving the CPA, it was approximately 6 inches from the duct. Afterwards, a ½-inch stainless steel probe was placed into the centerline of the duct and the CPA was attached to the end of the 18-inch tube. The ½-inch tube allowed the heat to dissipate and lower the temperature to which the CPA and filter were exposed. The maximum temperature allowed by the vendor for the filter is 60 °C (140 °F). The temperature at 6 inches was 80 °C (176 °F), the filter was drooping, and the lab found cement dust in the PCT and ACAMS.

This 18-inch distance is greater than the 5 inches approved by the DHHS for the common stack for starting the VX mine campaign. It must be noted that at this time, there was no guidance or direction from DHHS or EMO to place the AgF pad within this prescribed distance since the DAL probe and the

common stack probe sample from very different environments. The gas in the common stack is at 189 °F and is water saturated. To overcome the water content, the probe adds dry instrument air at 15 times the sample flow. The DAL probe samples from a dry, high-temperature (515 °F) gas stream and uses no dilution air since the ACAMS is already pushed to its maximum sensitivity to reach the 0.2 TWA LOQ monitoring limit. The common stack monitors at the ASC level, 30 times higher than the MPF duct.

2.6 Sampling Probe Testing

Although the DAL probe is longer than the 5 inches at the common stack, subsequent to the event the used probe was tested for its ability to transmit VX vapors. VX vapors were generated in a plastic bag by injection of dilute liquid agent solution. The probe was evaluated by measuring the VX concentration with an ACAMS before and after the probe. The VX concentration after the probe was measured by placing an ACAMS AgF conversion pad at the exit of the probe that drew gas from the bag. The VX concentration in the bag was measured directly with the conversion pad in the bag. Comparing the two values provides an indication of the decomposition or deposition of the agent on the surface of the probe. The tests showed that the probe was transmitting 100% of the VX within the statistical accuracy of the measurements.[3]

2.7 Probe Test Concerns

The results of the testing at JACADS was questioned by the reviewers in the investigation report due to the use of isopropanol in the dilute VX standards used to establish the VX vapor concentrations in the bag and the ambient temperature of the probe during the tests. These concerns generated the requirement to test the ability of the stainless steel probe to transmit VX using neat VX vapor at the temperatures experienced during the normal monitoring operation of the DAL (~ 600 °F). Washington Demilitarization Company subcontracted the investigation to SwRI as part of the Risk Management program for the Program Manager for Chemical Demilitarization (PMCD). Investigation was conducted at SwRI laboratories located in San Antonio, Texas and by the SwRI laboratory at JACADS.

3. OBJECTIVES

The objectives of the task include:

- Determining the transmission efficiency of the ½-inch stainless steel probe while sampling a 600 °F atmosphere.
- Determining the life of the existing CPA for various times and temperatures.
- Evaluating other CPA materials to improve the CPA life at temperatures up to 600 °F.

4. APPROACH

Testing was planned to be conducted using heated breathing air into which an amount of VX vapor was injected. As noted above, the probe studies at JACADS used ambient temperature gas atmospheres containing small concentrations of carrier solvents. This raised concern that the data is not representative of the DAL gas stream. Therefore, to eliminate the chance that solvents enhance the transmission of VX through the probe, neat VX was to be used to generate the challenge atmosphere. The initial test configuration is shown in Figure 1.

A heat tape wrapped stainless steel tube was used to heat bottled breathing air to 600 °F. The key to controlling the challenge atmosphere was the ability to maintain the MINICAMS sample rate and the heated gas flow rate with mass flow controllers. The flows from the mass flow controllers were checked regularly throughout the various tests using a calibrated Buck Meter. The MINICAMS sampling pump was controlled to maintain a flow rate of 0.8 Lpm. A mass flow controller maintained a steady flow of breathing air, typically about 0.72 Lpm, through the heated feed line. The deficit flow created between the

MINICAMS pump and the heated breathing air flow rates, nominally 0.08 Lpm, was satisfied by aspirating VX vapor from a 100-L Tedlar® bag into the feed line immediately prior to the DAL probe tip. The mass flow controller for the heated breathing air was raised or lowered to decrease or increase, respectively, the aspiration flow rate of VX vapor into the probe to yield VX concentrations within the MINICAMS calibration range.

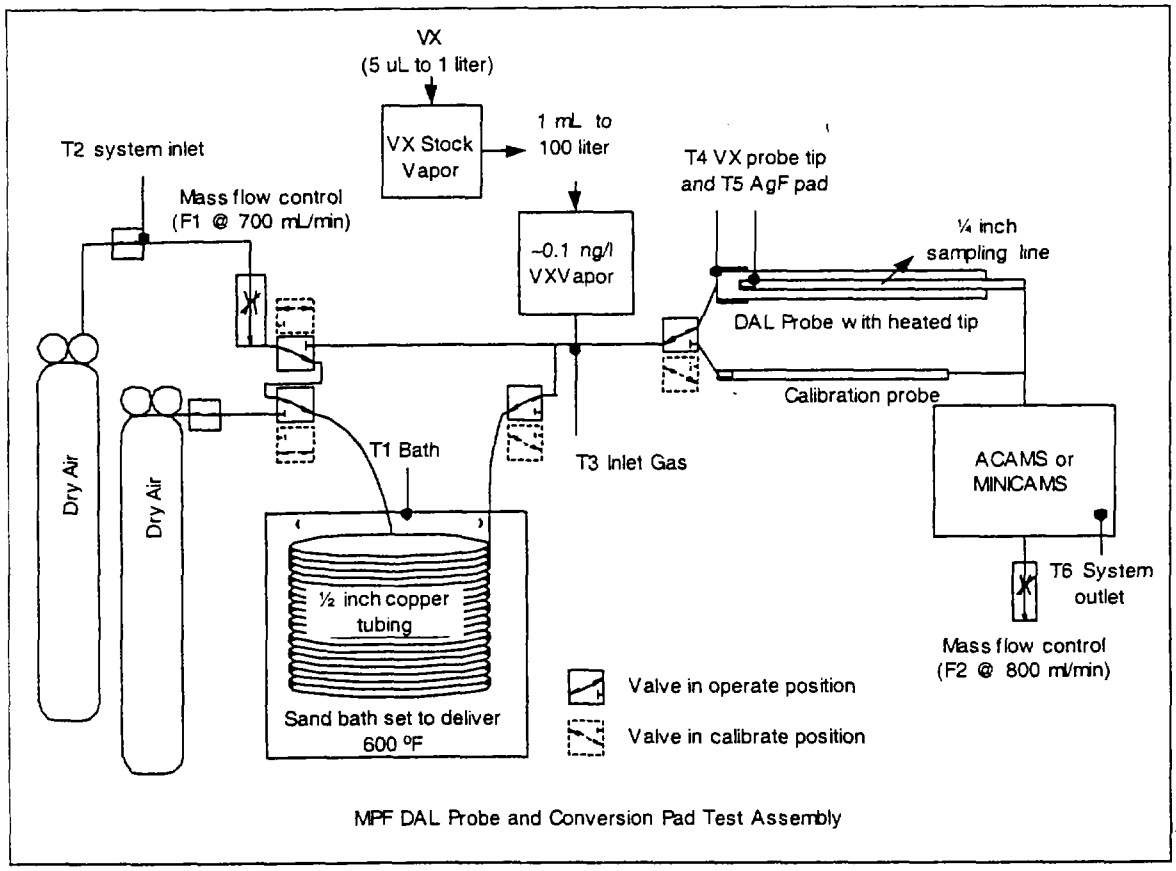


Figure 1. Test Assembly

VX is a high boiling semivolatile compound and is difficult to transport through transfer lines reproducibly. It tends to stick to tubing and valves in the feed system. Originally, multi-port valves were included in the system to allow switching of the VX challenge atmosphere from the test probe (heated) to a control or calibration probe (non-heated) without having to reduce the temperature of the test probe. After experiencing many frustrating tests with erratic results, the valve was removed and a single probe was used to document the VX concentrations at both room and elevated temperatures.

The reliance upon a single probe yielded more reliable results, and data was collected demonstrating that the VX could be transmitted from the probe tip to measurement locations at 5 and 18 inches down the length of the probe. However, there were still reoccurring anomalous readings as the probe was switched between heated and room temperatures. These circumstances hindered the establishment of accurate baseline VX concentrations that made it difficult to document the transmission efficiencies that were actually being achieved by the probe.

These problems were primarily believed to be the result of the inlet section where the VX vapor and the heated breathing air mixed prior to entering the probe. Because VX has a boiling point of 298 °C, any

cool spot on the transfer line from the Tedlar bag into the inlet section would cause deposition and variations in the VX concentrations reaching the probe. Additionally, since the inlet section had to be heated to the temperature of the gas stream (600 °F), there was concern that decomposition of the VX could occur as the vapor traversed the narrow diameter steel transfer line into the inlet section. Several different inlet section configurations to minimize the potential occurrence of these variables were tested in a series of experiments, but documenting the transfer efficiency with any certainty was still elusive.

Eventually, to overcome these shortcomings, a much simpler test system was developed. The final probe test configuration is shown in Figure 2. It consisted of a simple inlet attached to the probe through which a gas tight syringe could introduce a spike (2 to 3 mL) of VX vapor. In addition, a small hole was drilled in the probe at 5 inches and at 18 inches so that gas spike could be introduced directly on the pad to verify the CPA efficiency. This experimental setup had several advantages. One, it eliminated the VX gas feed system with its problematic cool (room) temperature transfer lines so that the MINICAMS was only pulling heated air into the probe. Secondly, the VX vapor spike was introduced quickly without any heating thereby eliminating decomposition concerns. Finally, the CPA could be spiked directly on the CPA at the operating temperature to eliminate concerns about potential temperature effects on the efficiency of the AgF pads.

It is important to note that the implementation of the final probe test configuration only became feasible as the test strategy switched from "continuous" to "puff" VX vapor challenge tests. As discussed later, the early test results revealed that the most daunting, worst-case scenario confronting the DAL probe's ability to effectively transmit VX vapor down the full-length of the probe (18 inches) would be during exposure to a short-lived VX vapor atmosphere occurring within a single sampling cycle rather than a VX vapor release spanning multiple sample cycles.

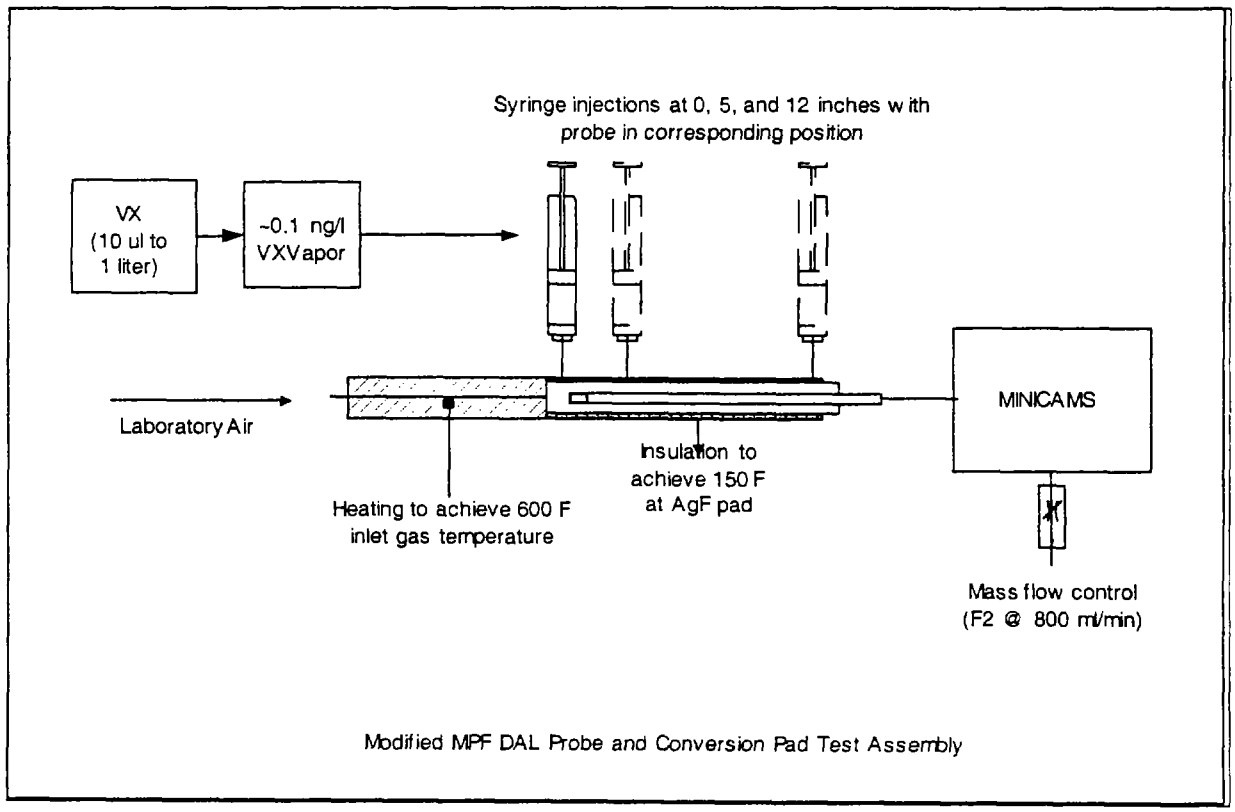


Figure 2. Final Probe Configuration

4.1 VX Vapor Generation

To avoid the presence of carrier solvents, neat VX was used to generate the challenge atmosphere by serial dilution. VX Stock Atmosphere 1 was generated using a 1-L Tedlar bag filled with nitrogen into which 10 µL of neat VX had been injected. Time was permitted for saturated VX vapor concentration to become established in this bag. To minimize the presence of the VX-like volatile contaminants of the neat VX, the stock CASRM solution was degassed prior to withdrawal of the 10 µL.

From this Stock Atmosphere 1, further dilution was made to achieve the desired concentration in the challenge gas to be mixed with the heated breathing air. For the initial studies, Stock Atmosphere 2 was made by adding 15 to 25 mL of the Stock Atmosphere 1 into a 100-L Tedlar bag filled with breathing air. During the subsequent studies utilizing a gas-tight syringe to inject the VX vapor, approximately 65 mL of the Stock Atmosphere 1 was added into a 1-L Tedlar bag filled with breathing air.

4.2 MINICAMS Configuration

The test apparatus used a MINICAMS (Serial Number 3186) rather than an ACAMS as is used at JACADS. The MINICAMS is a commercial equivalent of the ACAMS that uses the same operating principles. The MINICAMS was modified to incorporate a 20 mm PCT to minimize the breakthrough of the VX that has been reported by other researchers with the shorter PCT. The purpose of the test was to compare the relative concentrations of the VX at the probe tip and at the end of the probe, so any possible differences between the ACAMS and MINICAMS response should not have biased the transmission efficiency results.

The operational cycle of the MINICAMS consisted of two discrete periods: a sampling period (set at 180 to 480 seconds depending upon the turnaround time desired for the experiments), followed by an analytical period (nominally 120 to 180 seconds depending upon the selected software program). As stated earlier, the MINICAMS sampling rate was set at 0.8 Lpm. During the analytical period, the sample flow is diverted from the PCT, and the subsequent lowered pressure drop within the instrument causes the flow rate to increase to approximately 1.3 Lpm. Thus, if the Tedlar bag remained connected to the probe during the analytical period, as the mass flow controller maintained the heated breathing air flow at 0.72 Lpm, the aspiration of VX vapor into the probe would increase to around 0.58 Lpm compared to the 0.08 Lpm rate during the sampling period. If exposure of the probe to VX vapor did not affect the measurement of VX vapor, then this increased mass flow of VX during the analytical phase would be irrelevant. As will be discussed later, this may not be the case, and the procedure was instituted to shut-off the flow of VX from the Tedlar bag during the analytical period and allow room air to enter the probe to accommodate the increased flow rate.

4.3 Concentration Determination

Although it was intended to maintain a stable challenge atmosphere, slight changes in the Stock Atmosphere 1 headspace gas concentration, fluctuations in the dilution ratio from Stock Atmosphere 2, or decomposition of the VX in the Tedlar bag could affect the VX challenge gas concentration. To eliminate this as a variable, the challenge concentration was monitored directly at the probe tip at approximately 150 ° F to minimize degradation or deposition. As discussed earlier, this approach did not adequately address the challenge concentration reaching the probe tip when the gas temperature was 600 °F; thus, the implementation of the gas-tight–syringe injection method. Prior to and at the completion of a test sequence, the CPA was tested using a standard liquid challenge.

4.4 Probe Testing

In an attempt to duplicate the operating conditions at JACADS, probes from JACADS used to sample the DAL exhaust duct for one week were obtained for the experiments in this study (DAL probe numbers 115214-20-051 and 115214-20-058). The ½-inch stainless steel probe was connected to the inlet section heated to yield a gas temperature of 600 °F. The configuration of the heating tape and insulation was

carefully arranged at the inlet so that this temperature was confined to within the first inch of the probe tip, duplicating the conditions at the JACADS DAL. A thermocouple was inserted through a port in the inlet section and positioned in the center of the probe tip to measure the challenge gas temperature.

During the latter stages of the program, it was desired to mimic the JACADS DAL temperature profile at the 5 and 18-inch distances which are created by the 6.0 Lpm sampling rate of the ACAMS. As shown in the following section, to replicate those temperatures at the MINICAMS flow rate of 0.8 Lpm, varying layers of insulation had to be applied to the test probe to yield temperatures of approximately 380 and 160 °F at the 5 and 18-inch distances, respectively. This still comprises a conservative testing scenario since the residence time at 6 Lpm in the JACADS DAL probe would only be 1/7.5th of the residence time at our sample flow rate of 0.8 Lpm. Therefore, assuming that a shorter residence time in the probe reduces potential interactions between the VX and the heated surfaces, the percentage of VX transmitted to 18 inches in the JACADS DAL probe should be greater than the efficiencies measured in our tests at 0.8 Lpm.

The MINICAMS sampling line was a ¼-inch Teflon® line extending into the DAL probe. The CPA was positioned at the end of this ¼-inch sampling line. The CPA consisted of two AgF pads inside a short stub of ⅜-Teflon® tubing attached to the end of the ¼-inch Teflon® tubing. This configuration is identical to the JACADS sampling line. During testing, the ¼-inch line was positioned at varying distances within the probe.

5. RESULTS

5.1 Probe Temperature Profile

The ½-inch stainless steel probe inserted into the DAL duct exists only to reduce the temperatures experienced by the CPA and the filter. As shown in Figure 3, at a flow rate of 0.8 Lpm, the temperature drops dramatically from the 600 °F at the probe tip to approximately 140 °-F at 5 inches from the tip. The temperature profile is very flat between 5 and 18 inches, falling to only about 80 °F. The squares (■) are temperatures measured with the test apparatus used in these probe tests, and the diamonds (♦) are those measured with the probe in the DAL duct at JACADS for the ACAMS.

At JACADS, the DAAMS configuration for the DAL requires four DAAMS tubes, each drawing 1.5 Lpm for a total of 6.0 Lpm. The higher flow rate extends the temperature profile in the probe such that at 5 inches the temperature is in excess of 350 °F and falls to 160 to 200 ° F at approximately 18 inches, as illustrated in Figure 4.

5.2 Ambient Temperature, Continuous VX Vapor Flow

The first testing addressed the ability of the DAL probe to transmit the VX challenge atmosphere at ambient conditions (70 °F). Table 1 summarizes the data from three series of tests performed on different dates with the DAL probe with the VX Stock Atmosphere 2 continuously flowing into the probe throughout the MINICAMS operating cycle (a 4-minute sampling period followed by a 2-minute analytical phase). The table presents the VX TWAV concentrations recorded by the MINICAMS with the CPA at 0 inches (probe tip) and at 18 inches from the probe tip. A graphical depiction of one of the test series presented in Table 1 is given in Figure 5. Due to the drastically increased flow rate of VX vapor during the analytical phase as described earlier, allowing the VX to flow into the probe during that period would be equivalent to exposing the CPA to the challenge atmosphere at a constant 0.8 Lpm for a total of 18.5 minutes.

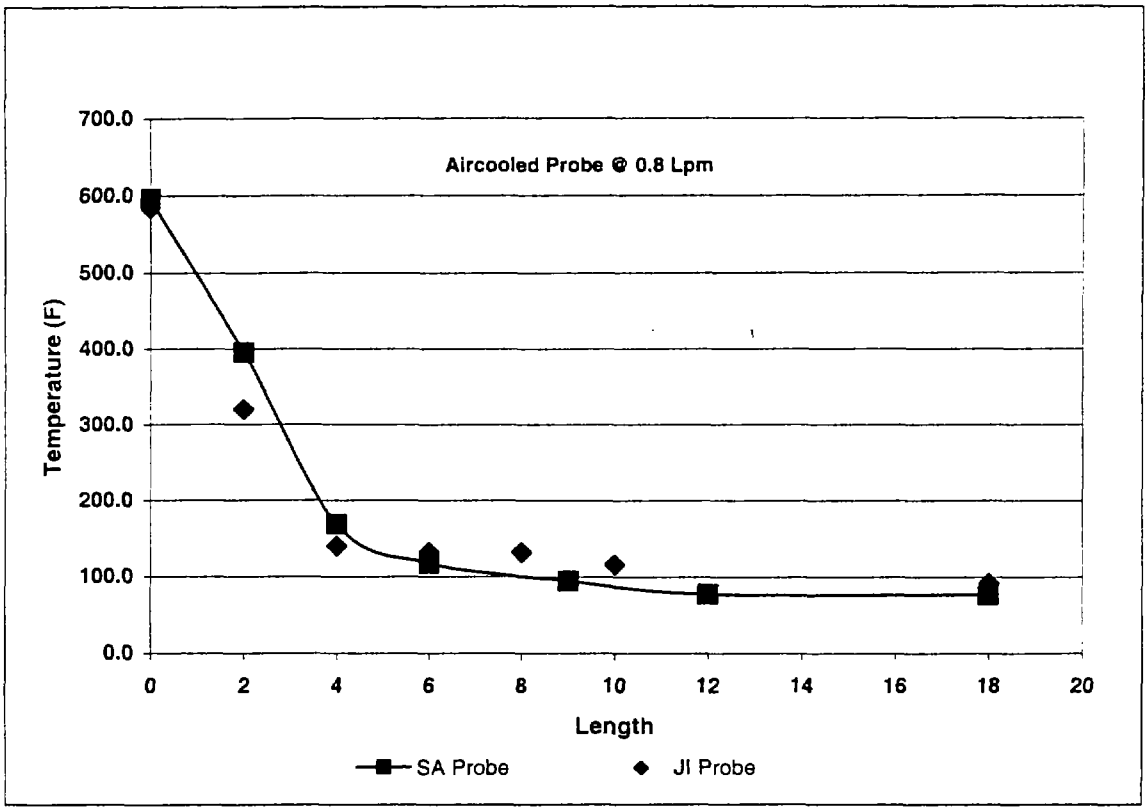


Figure 3. Temperature Profile of the Air-Cooled Probe at 0.8 Lpm

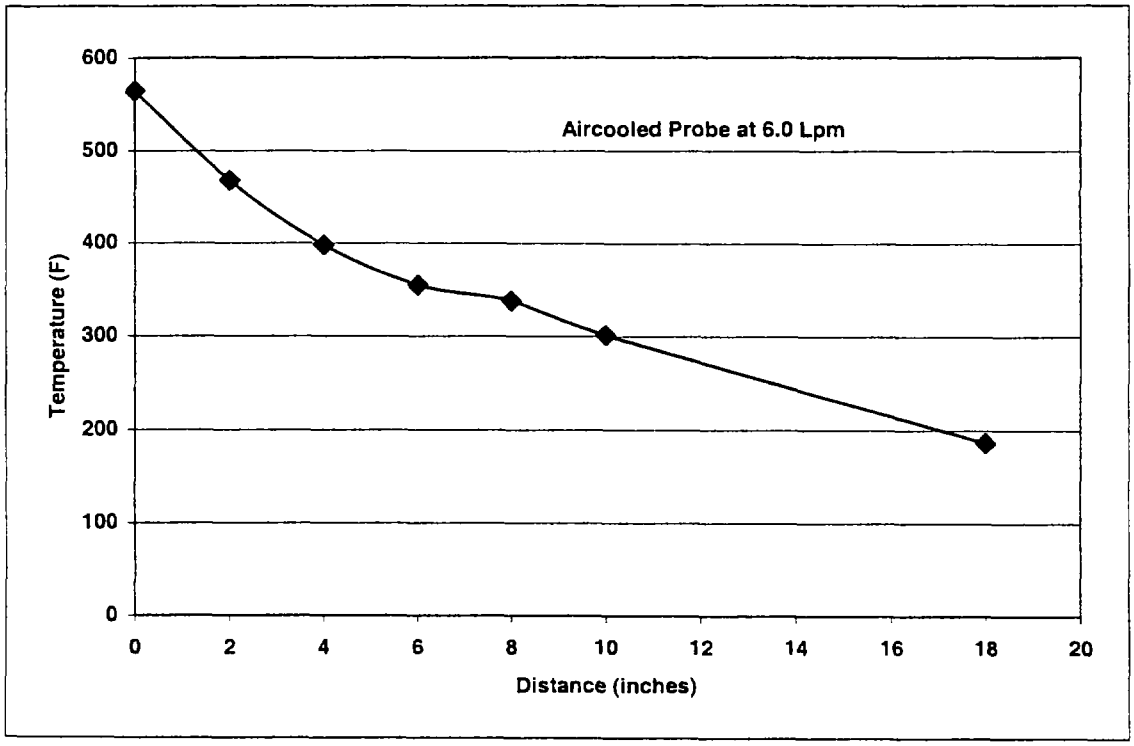


Figure 4. Temperature Profile of the JACADS Air Cooled Inch Probe at a Flow Rate of 6.0 Lpm

Table 1. Performance at Ambient Temperature and Continuous VX Vapor Flow

Time	CPA Site	MINICAMS TWAV	Time	CPA Site	MINICAMS TWAV	Time	CPA Site	MINICAMS TWAV
1006	18	1.24	0942	0	1.11	1119	0	1.75
1012	18	1.30	0948	0	1.16	1125	0	2.01
1018	18	1.32	0954	0	1.20	1131	18	2.11
1030	0	0.08*	1000	18	1.11	1137	18	2.13
1036	0	1.26	1006	18	1.16	1143	18	2.25
1042	0	1.36	1012	18	1.20	1149	0	2.46
1048	0	1.43	1018	18	1.22	1155	0	2.48
1054	0	1.52	1024	0.	1.20			
1106	18	0.09*	1030	0	1.23			
1118	18	1.31	1036	0	1.29			
1124	18	1.34	1042	18	1.40			
1130	18	1.36	1048	18	1.41			
1136	18	1.37						

* Zero baseline (flow of VX stopped)

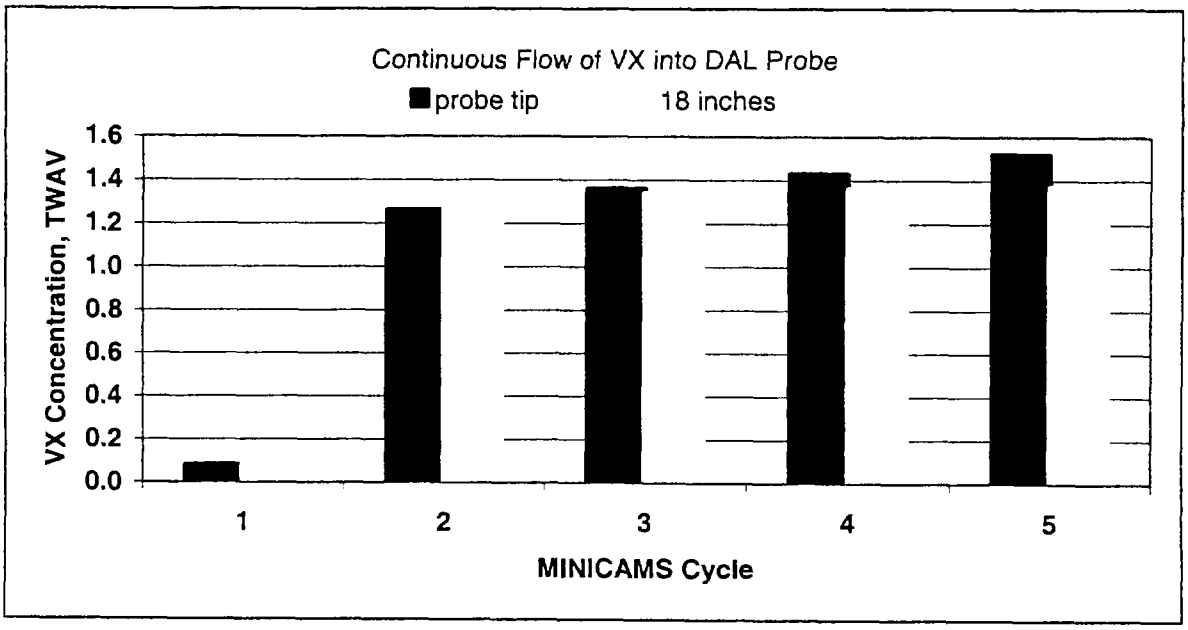


Figure 5. Continuous Flow of VX into DAL Probe at Ambient Temperature

The data show that under the test conditions, as the CPA was moved between the probe tip and 18 inches from the tip, the VX concentrations measured by the MINICAMS remained comparatively stable. This indicates that VX vapor can be effectively transmitted by the DAL probe even at ambient conditions.

There is a general tendency for the VX concentrations to incrementally increase during the duration of the VX flow through the DAL probe. The increasing concentrations at 18 inches parallels similar rises in concentrations at the probe tip, indicating that some passivation of the surfaces in the inlet section preceding the probe ~~was~~ may be occurring. This is further demonstrated by the data in Table 2 in which the zero baseline at the probe tip (0 inches) showed residual VX being present after a series of exposures to VX vapor. Cleaning the probe with dichloromethane (DCM) and/or isopropyl alcohol (IPA) eliminated the residue and restored probe performance. Figure 6 graphically shows the effect of cleaning the probe on the probe's performance. One "lesson learned" during the course of the experiments was that the probe performance could be adversely affected by the presence of organics plating on the surfaces. At elevated gas temperatures, it appeared that high-boiling decomposition products plating on the surfaces acted as a sink for the VX vapors causing artificially low VX concentration measurements. Cleaning the probe with a swab soaked with DCM or IPA always yielded an immediate improvement in the probe performance. Thus, it is highly recommended that when the CPA on the DAL probe is changed that the probe interior also be swabbed with solvent to remove residues that could hinder the transmission of VX vapors down the length of the probe.

Table 2. VX Residue Acumulation Inside Probe

Time	CPA Site	MINICAMS TWAV
1124	18	0.04
1129	18	0.91
1134	18	1.27
1139	0	0.55*
1144	0	0.66*
1149	0	0.08*
1224	0	0.02*
1229	0	1.26
1234	0	1.43
1239	0	0.92*
1244	0	0.27*
Cleaned Probe with DCM & IPA		
1409	0	0.00*
1414	0	0.01*
1419	0	1.15
1424	0	1.46

* Zero baseline (flow of VX stopped)

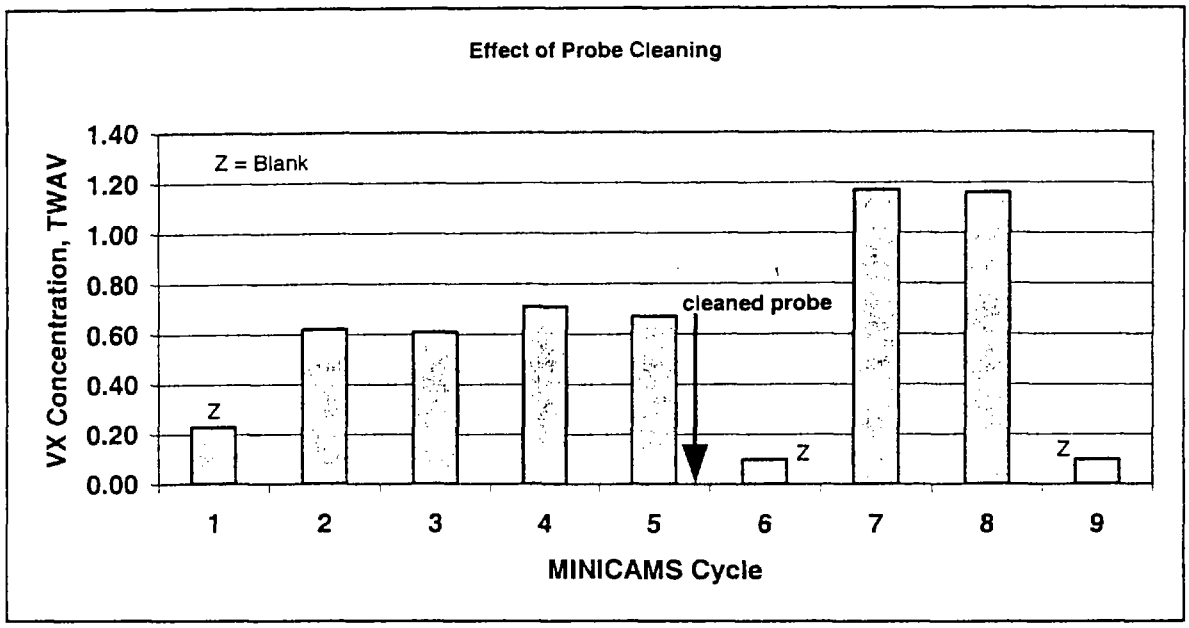


Figure 6. Effect of Probe Cleaning on Probe Performance

5.3 Gas Temperature at 600 °F with CPA at 5 Inches Exposed to VX Vapor Puffs

The ability of the DAL probe to transmit the VX vapor challenge atmosphere with gas temperatures of 600 °F at the probe tip was first evaluated with the CPA at a distance of 5 inches from the probe tip. At this distance, the temperature of the CPA was within the range of 130 to 150 °F (the variability was due to subtle changes in the heating configuration used on the inlet section). Hence, the VX vapor was subjected to a temperature span comparable to the values that the CPA would experience at a distance of 18 inches from the DAL probe tip with the ACAMS operating at a flow rate of 6.0 Lpm.

The data presented in Table 3 was collected with the probe being exposed to "puffs" of VX vapor – the flow of VX Stock Atmosphere 2 vapor from the Tedlar bag into the probe only occurred during the sampling period. During the analytical phase, only the heated breathing air plus room air (ambient temperature) aspirated to accommodate the increased flow rate of the MINICAMS entered the probe. Using this vapor challenge system, the VX concentrations obtained with the CPA at 5 inches and the gas temperature at the probe tip starting at 600 °F were compared to the readings obtained with the CPA at the probe tip (0) and the VX challenge atmosphere at a slightly elevated temperature (to minimize condensation).

Table 3. Probe Performance with CPA at 5 Inches for VX Vapor Puffs

Time	CPA Site	Gas Temperature at Probe Tip, °F	MINICAMS TWAV
Liquid Challenge of CPA = 0.99 TWAV			
1113	0	108	0.06*
1121	0	108	0.10*
1129	0	118	3.42
1137	0	121	4.23
1145	0	122	3.94
1153	0	123	3.99
1329	5	590	0.02*
1337	5	585	0.08*
1345	5	609	0.00*
1353	5	608	4.86
1401	5	610	0.19*
1409	5	613	5.17
1417	5	613	0.21*
1425	5	616	5.35
1433	5	617	0.22*
1441	5	618	5.53
1449	5	620	0.23*
1457	5	631	0.08*
1505	5	635	5.52
1513	5	646	0.20*
1521	5	651	5.63
1529	5	667	0.21*
1537	5	680	5.69
1545	5	690	0.23*
1553	5	695	5.57
1601	5	698	0.22*
1609	5	703	5.35
Liquid Challenge of CPA = 1.00 TWAV			

* Zero baseline (flow of VX stopped)

The data in Table 3 indicates that placement of the CPA within the probe at a distance far enough from the probe tip to be compatible with the CPA material (i.e., about 150 °F) yielded VX vapor measurements comparable to the reference values obtained at the probe tip at ambient temperatures. Indeed, the MINICAMS VX measurements remained stable even as the gas temperature at the probe tip approached 700 °F.

The VX concentrations at the 5-inch probe location were somewhat elevated compared to the reference values obtained at the probe tip at ambient temperatures. Two phenomena were believed responsible for this result. First, although the gas temperature within the inlet section was raised slightly to minimize condensation during the measurements collected at the probe tip, it appears that some of the VX vapor did in fact plate onto the surfaces of the inlet section causing the ambient reference values to be depressed to some extent. Secondly, as can be seen from the gradual gradient of the zero baseline during the course of the "puff" tests, residual VX appears to be accumulating either within the probe or the inlet section. Interpretations of the chromatographs produced by the MINICAMS during the tests indicate the possibility of potential decomposition products also accumulating within the probe or the inlet section and the presence of those organics being related to the appearance of the VX residue. Again, simple swabbing of the probe interior with DCM and/or IPA readily returned the zero baseline value to a nominal level (i.e., less than 0.1 TWAV). As stated earlier, our experience throughout the testing program indicates that periodic cleaning of the DAL probe enhances its performance.

To obtain a truer representation of the transmission efficiency achievable by the DAL probe, the use of a gas-tight syringe to challenge the probe with a puff of VX vapor was adopted. With the CPA at 5 inches from the probe tip, 2 or 3 mL of concentrated VX vapor was injected into the heated air stream at the probe tip. The injection of the VX vapor spanned a duration of only a few seconds shortly after the commencement of the sampling phase (4 minutes). As the reference for calculating the transmission efficiency under these conditions, VX vapor spikes were introduced directly onto the CPA through a 1/16th inch hole drilled through the probe at the 5-inch location.

The results from two separate test dates are presented in Table 4 and the first series depicted in Figure 7. The results of the gas-tight syringe injections supported the earlier findings using the longer-duration puffs of VX Stock Atmosphere 2 from the Tedlar bag. The gas-tight syringe challenge also represents the worst-case detection scenario for the probe – the accurate measurement of a short-lived ~~whiff~~ puff of VX vapor within the DAL. At a flow rate of 0.8 Lpm and within the analytical limits of the tests, essentially 100 percent of the VX vapor at the probe tip in an air stream heated to 600 °F can be detected by the CPA located at 5 inches from the probe tip.

5.4 Gas Temperature at 600 °F with CPA at 18 Inches Exposed to VX Vapor Puffs

The gas-tight syringe injection method was selected to document the transmission efficiency of VX vapor at 600 °F to a distance of 18 inches from the probe tip since this procedure proved to be the most reliable. As was stated for the 5-inch location tests, this procedure represents the worst-case scenario for challenging the capability of the CPA to accurately detect VX vapors when located 18 inches from the probe tip. The gas temperatures at the 5- and 18-inch locations in the probe were carefully controlled using heating tape and insulation to mimic the temperatures that have been documented in the DAL probe with the ACAMS (i.e., 380 and 160 °F, respectively) of the CPA (Figure 3). Table 5 presents the data from three separate VX puff tests performed on two different days. Figure 8 graphically shows the results from the third series of tests. The results indicate that at a flow rate of 0.8 Lpm, the transmission efficiency for VX vapor between the probe tip and the CPA at the 18-inch location is approximately 60 percent.

Table 4. Probe Performance for Gas-Tight Syringe Injections with CPA at 5 Inches

Time	MINICAMS TWAV	Gas Temperature @ Probe Tip, °F	Gas Temperature @ 5 Inches, °F	Injection Location
1339	0.06	576	135	N/A, zero baseline
1345	2.57	580	132	Probe tip
1351	2.96	589	132	Probe tip
1357	2.45	592	135	Pad at 5-inches
1403	2.47	590	135	Pad at 5-inches
1409	0.07	590	135	N/A, zero baseline
1415	0.03	592	137	N/A, zero baseline
1421	2.42	592	137	Probe tip
1427	2.39	595	125	Pad at 5-inches
1433	2.33	598	126	Pad at 5-inches
1439	2.61	596	125	Probe tip
Average at Pad = 2.41 TWAV; Average at Probe Tip = 2.64 TWAV Transmission Efficiency = 110 percent				
Liquid Challenge of CPA = 0.92 TWAV				
1531	0.14	597	168	N/A, zero baseline
1538	3.06	600	161	Pad at 5-inches
1545	3.21	606	160	Probe tip
Transmission Efficiency = 3.21/3.06 = 105 percent				
1552	0.00	604	-	N/A, zero baseline
1559	2.67	604	155	Pad at 5-inches
1606	2.66	603	160	Probe tip
1613	0.06	602	-	N/A, zero baseline
Transmission efficiency = 2.66/2.67 = 100 percent				
Liquid challenge of CPA = 0.98 TWAV				

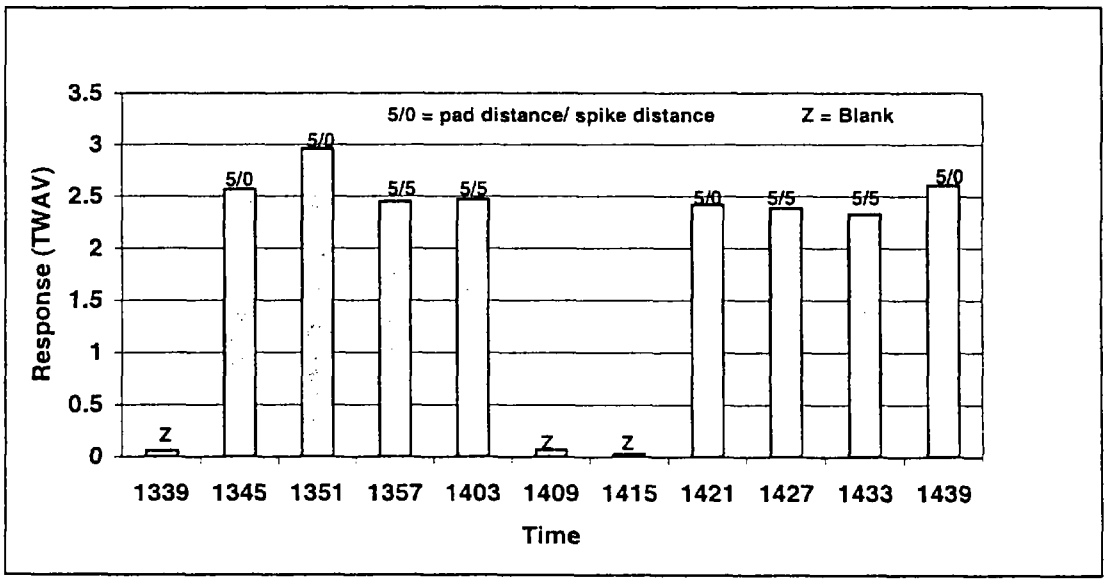
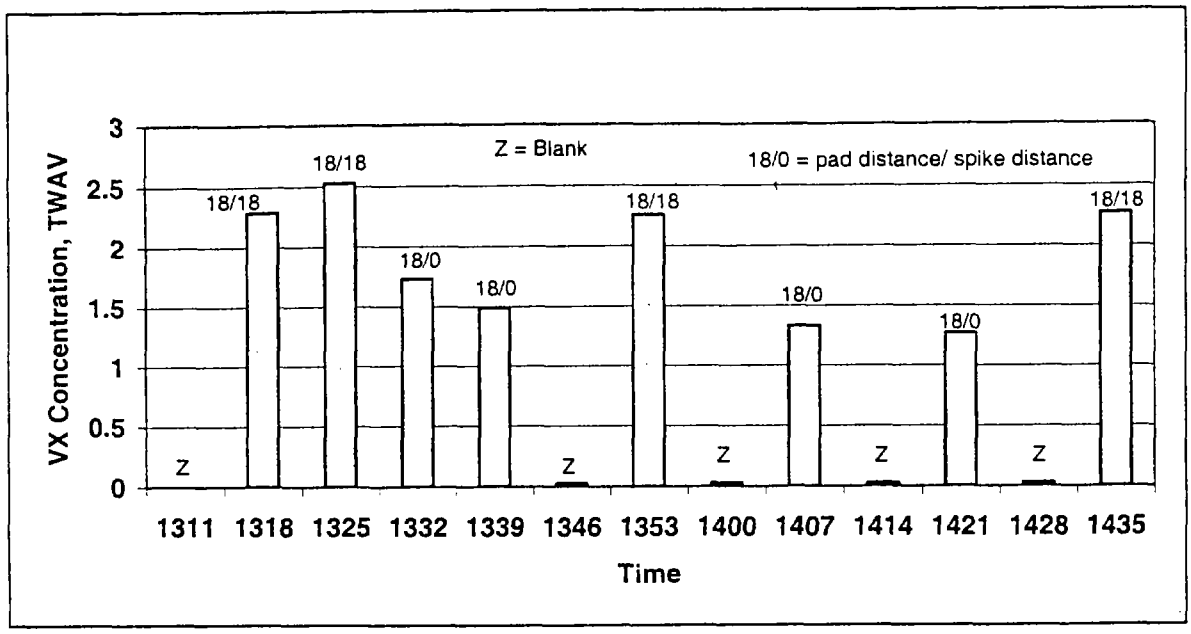


Figure 7. Probe Performance for Gas-Tight Syringe Injections with CPA at 5 Inches

Table 5. Probe Performance for Gas-Tight Syringe Injections with CPA at 18 Inches

Time	MINICAMS TWAV	Gas Temperatures, °F			Injection Location
		Probe Tip	@ 5 Inches	@ 18 Inches	
1049	0.07	592	390	148	N/A, zero baseline
1056	2.34	595	388	148	Pad at 18-inches
1103	2.35	595	394	150	Pad at 18-inches
1110	0.07	597	396	155	N/A, zero baseline
1117	1.23	594	401	156	Probe tip
1124	1.11	597	405	154	Probe tip
1131	2.00	593	393	154	Pad at 18-inches
1138	0.06	592	-	-	N/A, zero baseline
1145	1.31	593	388	148	Probe tip
1152	1.91	594	394	149	Pad at 18-inches
1159	1.39	592	398	153	Probe tip
1206	2.00	592	403	155	Pad at 18-inches
1213	1.12	595	390	151	Probe tip
Average at Pad = 2.12 TWAV; Average at Probe Tip = 1.23 TWAV; Efficiency = 58 percent					
1412	2.00	597	362	140	Pad at 18-inches
1419	2.03	598	371	140	Pad at 18-inches
1426	1.31	595	373	144	Probe tip
1433	1.22	594	373	147	Probe tip
1440	0.05	594	370	148	N/A, zero baseline
1447	1.85	594	365	144	Pad at 18-inches
1454	1.30	597	365	145	Probe tip
1501	1.89	603	368	150	Pad at 18-inches
1508	1.25	602	374	149	Probe tip
1515	2.00	602	369	150	Pad at 18-inches
1522	0.05	602	371	149	N/A, zero baseline
1529	1.11	600	370	145	Probe tip
1536	1.90	601	376	151	Pad at 18-inches
1543	1.20	603	380	154	Probe tip
1550	1.27	603	380	151	Probe tip
1557	1.88	605	382	150	Pad at 18-inches
1604	0.06	606	-	-	N/A, zero baseline
Average at Pad = 1.94 TWAV; Average at Probe Tip = 1.24 TWAV; Efficiency = 64 percent					
Liquid challenge of CPA = 0.93 TWAV					
1311	0.00	618	-	-	N/A, zero baseline
1318	2.29	617	389	147	Pad at 18-inches
1325	2.53	616	-	-	Pad at 18-inches
1332	1.73	617	-	-	Probe tip
1339	1.49	618	394	155	Probe tip
1346	0.03	618	-	-	N/A, zero baseline
1353	2.26	619	385	152	Pad at 18-inches
1400	0.03	618	-	-	N/A, zero baseline
1407	1.33	616	395	157	Probe tip
1414	0.03	618	-	-	N/A, zero baseline
1421	1.27	618	397	160	Probe tip
1428	0.03	616	-	-	N/A, zero baseline
1435	2.27	617	403	159	Pad at 18-inches
Average at Pad = 2.34 TWAV; Average at Probe Tip = 1.46 TWAV; Efficiency = 62 percent					
Liquid challenge of CPA = 0.98 TWAV					

Figure 8. Probe Performance for Gas-Tight Syringe Injections with CPA at 18 Inches



6. CONCLUSIONS

The following summarizes the results presented above.

1. Under continuous exposure to a VX vapor atmosphere at ambient temperature the VX is transported through the 18-inch stainless steel probe with high efficiency. A slight increase in the response was observed at both the inlet and with time indicating the there was some acclimation of the surface of the feed system.
2. Buildup of organics in the probe over time inhibited transport, therefore the probes should be "swabbed" with isopropanol when the CPAs are challenged to remove any particulate or residual organics or decomposition products
3. With "puff" exposures in 600 °F air at the inlet, with a 0.8 LPM flow rate, placing the CPA at 5 inches (~150 °F) gave vapor measurements comparable to the reference values obtained at ambient values with the CPA at the probe tip. This trend was duplicated up to 703 °F.
4. With "puff" exposures at 600 °F at the inlet, with a 0.8 LPM flow rate, placing the CPA at 5 inches gave vapor measurements comparable to the reference values obtained by injecting the puff onto the CPA at 5 inches at 150 °F.
5. With "puff" exposures at 600 °F at the inlet, with a 0.8 LPM flow rate, placing the CPA at 18 inches gave vapor measurements approximately 60% of the reference values obtained by injecting the puff onto the CPA at 18 inches at 150 °F.

Based on the REI analysis and the results summarized above it can be concluded that the ACAMS monitoring of the DAL at 600 °F would be effective in detecting VX in the discharge duct. The recovery at 18 inches was less than the desired 75% recovery, however, this is conservative because the flow rate in these tests was only 0.8 LPM compared to the 6 LPM used at JACADS. This would give a residence

time in the probe of 1/7.5th of the 0.8 LPM used in these tests and would even be less than the residence time at 5 inches (0.36 sec versus 0.75 sec). Based on this it is predicted that recovery for the DAAMS confirmation would be greater than 75% at 6000 LPM at a point in the probe where the temperature is approximately 150 – 160 °F.

---

[1] McCloskey, Gary W., Evans, Mark, and MacNaughton, Michael G., Report of Investigation: VX Contamination on Tray Released from JACADS Metal Parts Furnace Discharge Airlock on 12 August 2002.
[2] Montgomery, Christopher J., Bocklie, Michael J., Sarofim, Adel F., Magee, Richard S., Gouldin, Fredrick C., Bozzelli, Joseph W., and Westbrook, Charles K., Chemical Agent Decomposition Rates at Temperature Anticipated in the Discharge Airlock, REI Project No. 4440, December 23, 2002.
[3] Swynnerton, Nollie F., Testing Transmission of VX Vapor Through MPF Probe, Special Report to WDC SW-10209G02, MEM-0221014, September 17, 2002.

CONTINENTAL
RESEARCH AND
ENGINEERING, LLC

Preliminary CFD Model Results for the JACADS Discharge Airlock

27 September 2002

Model derivation, boundary conditions

First generation model
- Assumptions made for ease of computational efficiency
- Enables evaluation of derivation, verification

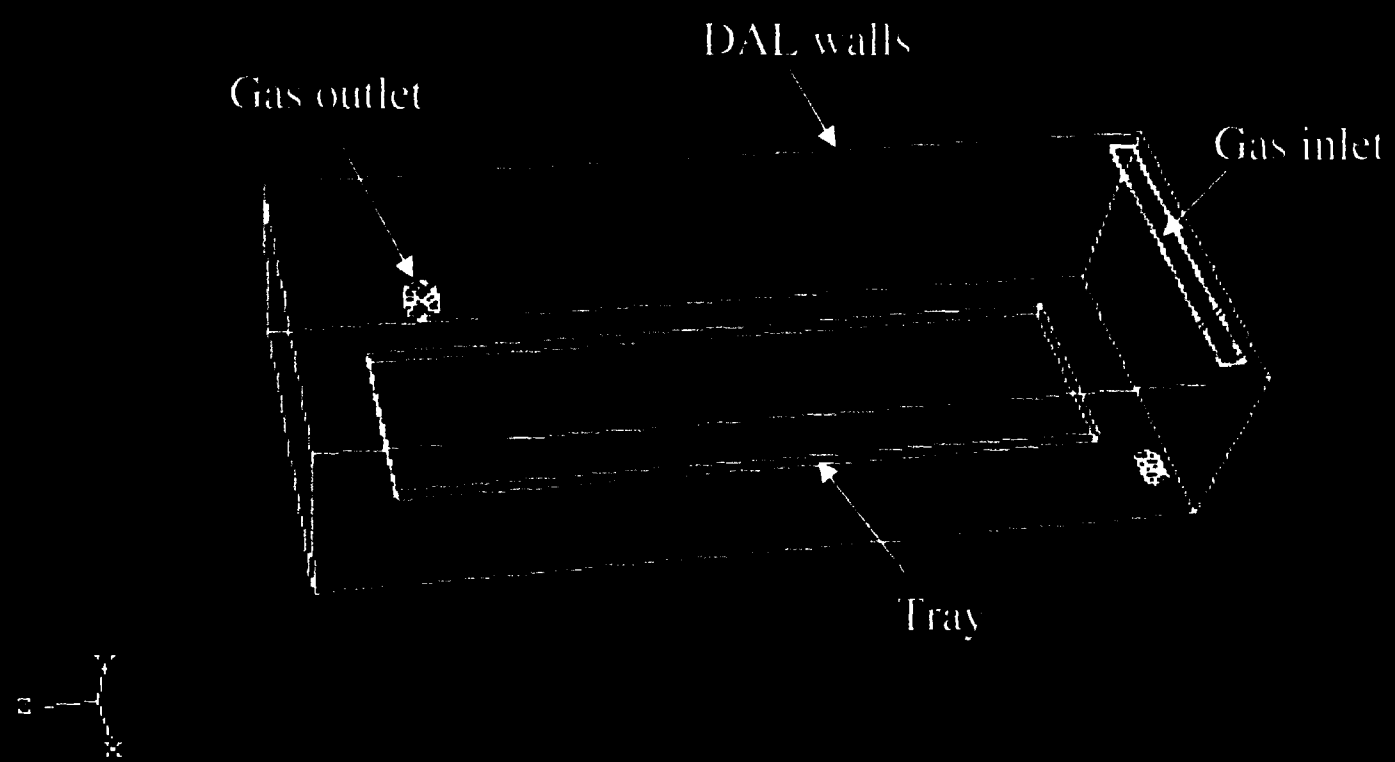
Discharge Airlock meshed model
- Inlet and exit gas ports per available drawings
- Tray: no rollers
- No WIC included in initial drawing

Three dimensional unsteady state problem
- Radiative heat transfer: discrete ordinate model

DAL walls maintained at constant temperature: 250 °F
- No heat loss, no conduction: for initial simplicity

Model construction

Top view of edges: velocity inlet, pressure outlet, DAL wall, tray wall



Materials

Tray

- Steel, initial temperature = 1600 °F
- Height: 0.292 ft, Width: 3 ft, Length: 10 ft

Discharge Airlock

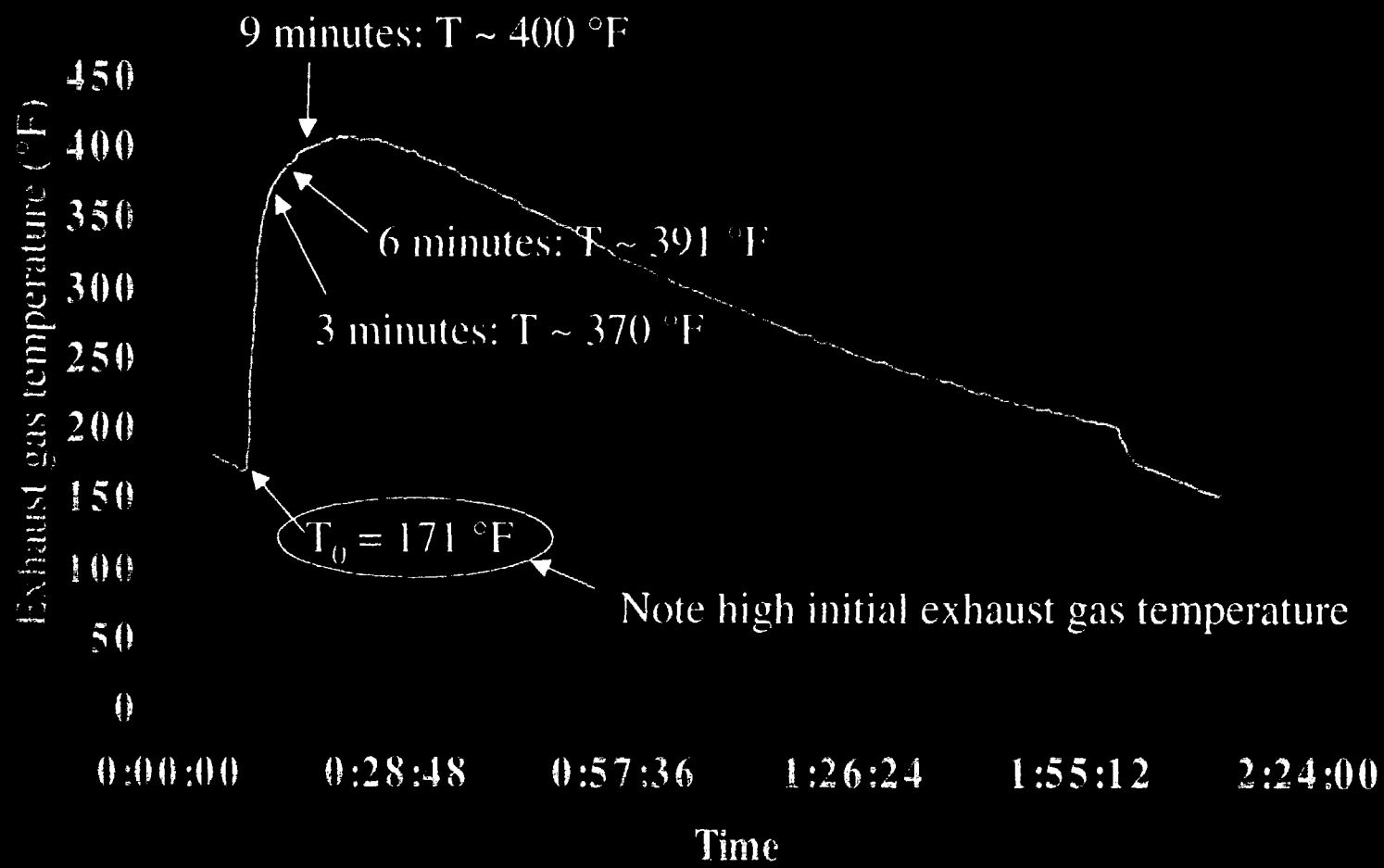
- Walls: Dolomite ($CaO \cdot MgO \cdot 2CO_2$)
- Temperature = 250 °F (constant)
- Height: 3.96 ft, Width: 5.67 ft, Length: 12.5 ft

Gas

- Air, ideal gas assumption to calculate density
- Initial temperature = 80 °F
- Inlet gas velocity: two cases
  - (1) 3.1 ft/s and (2) 6.1 ft/s

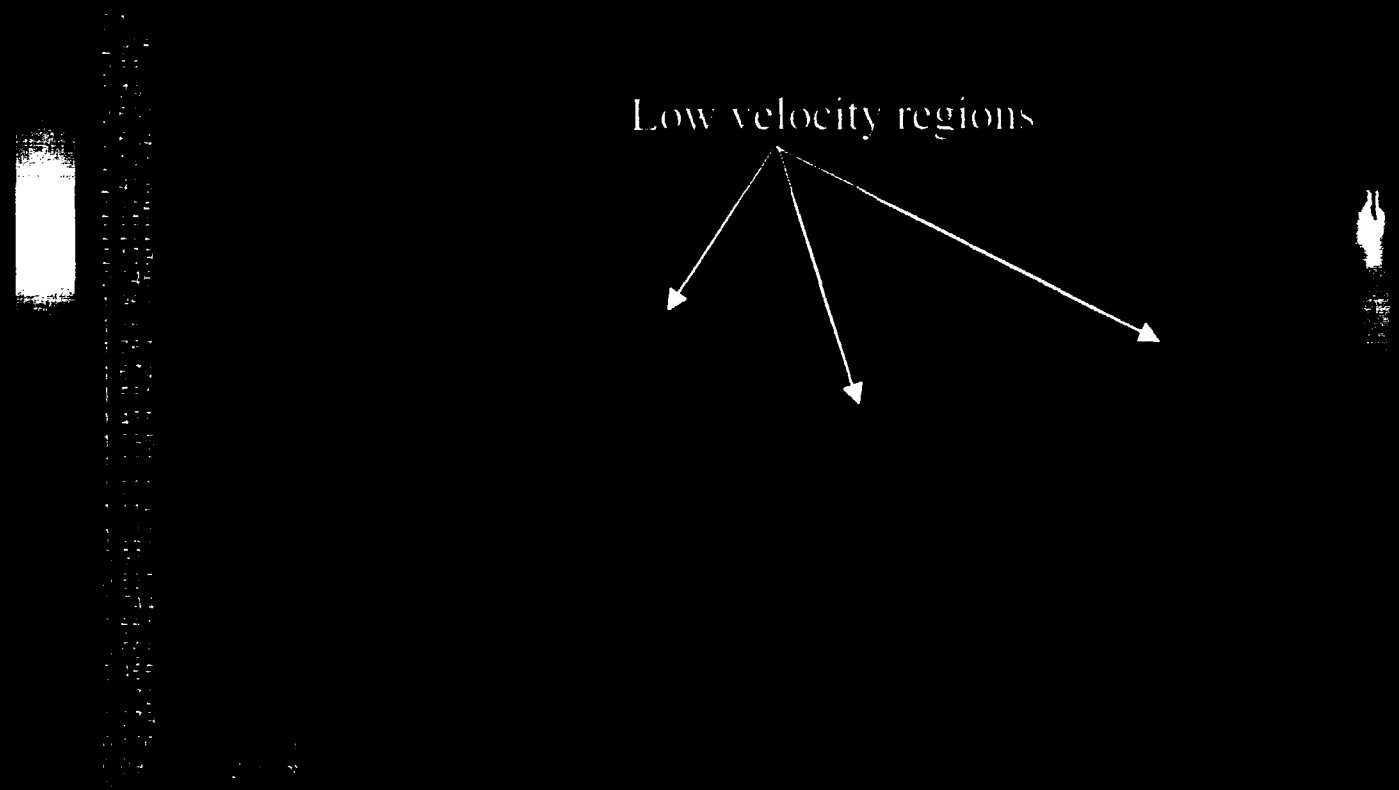
JACADS exhaust gas - metal tray

Initially, compare simulation results to test data for first nine minutes in DAL

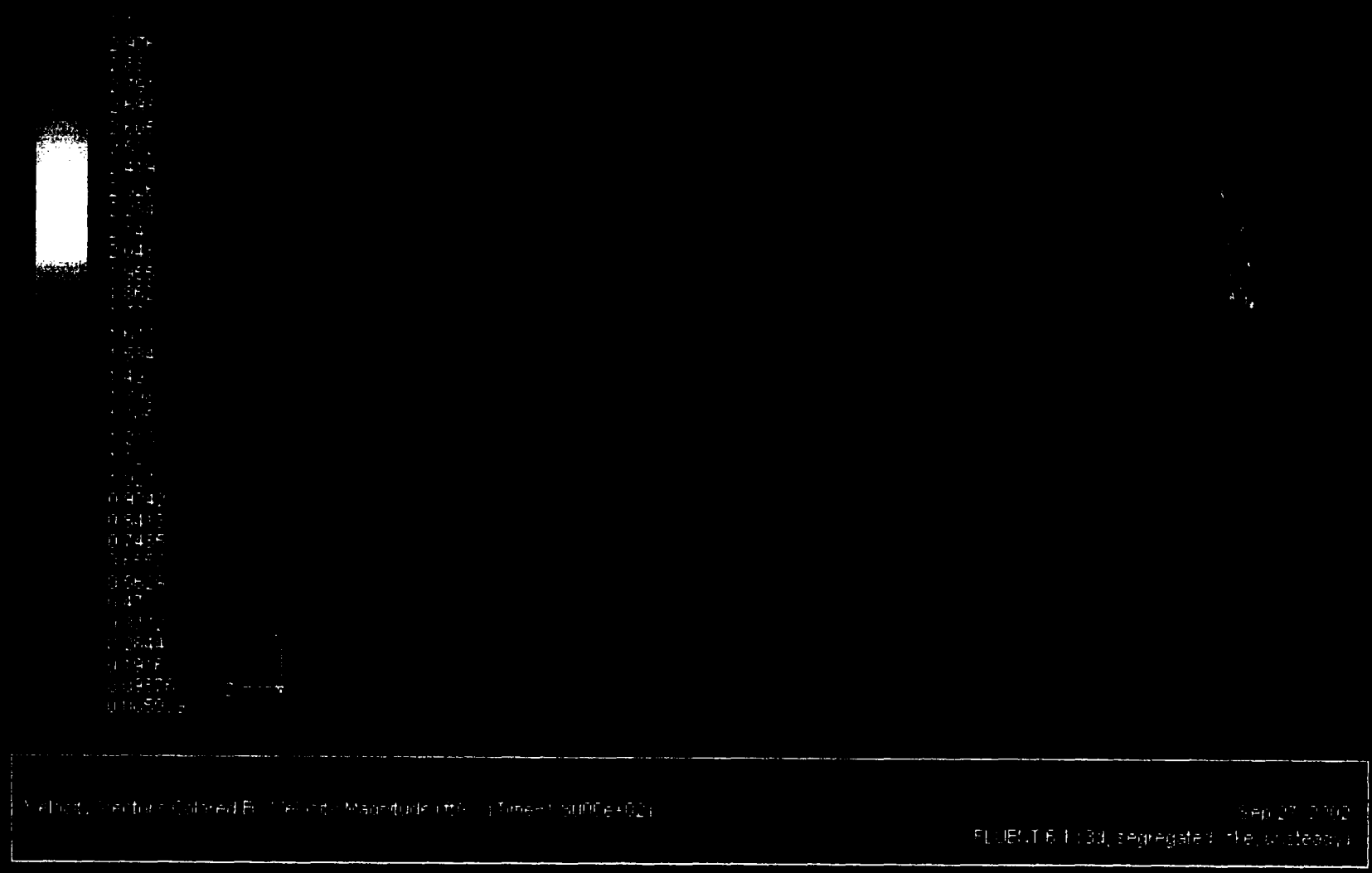


Test data from JACADS experiments: September 5, 2002

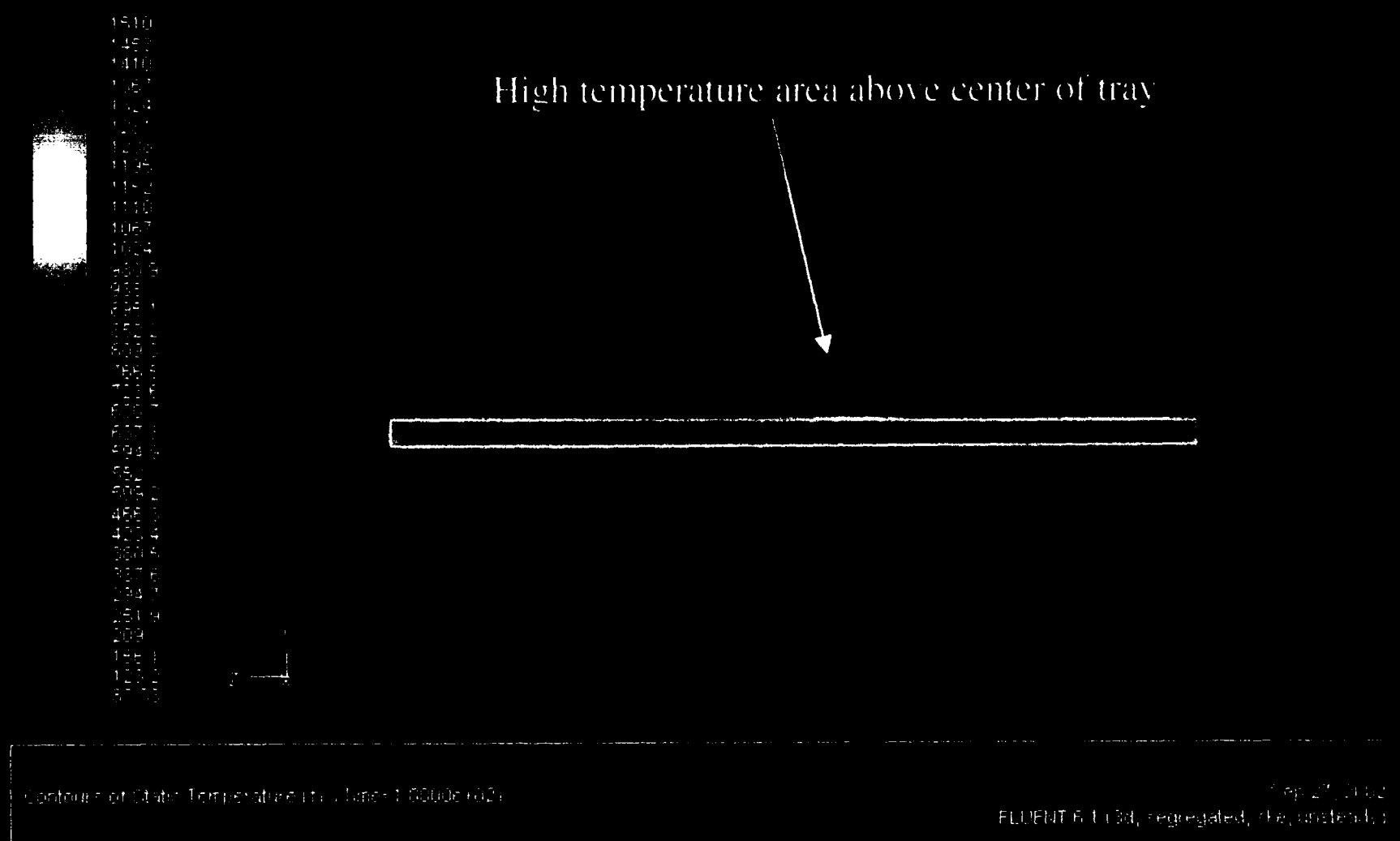
Centerline plane velocity profile: 3 minutes, 3.1 ft/s



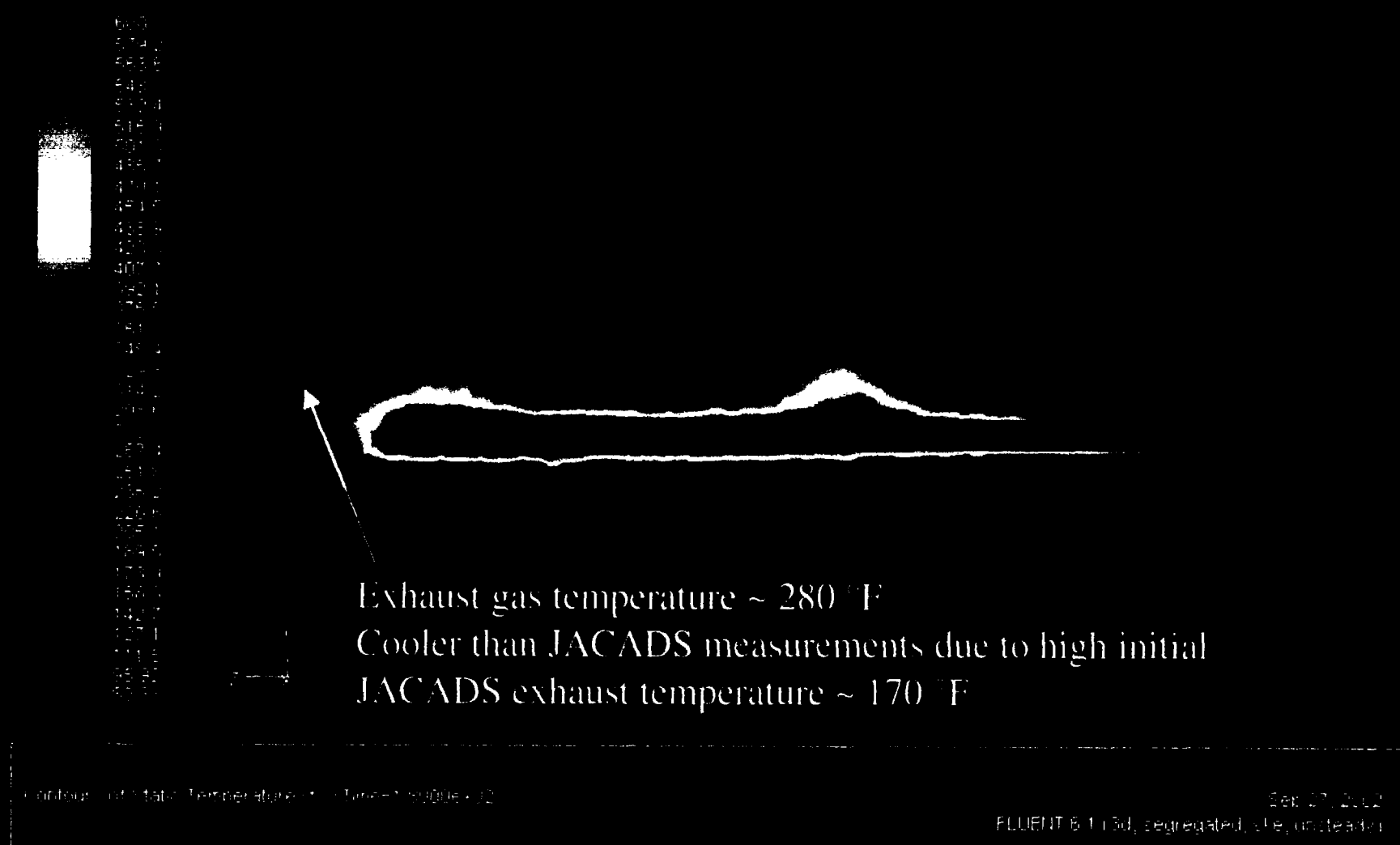
Centerline plane velocity profile: 3 minutes, 3.1 ft/s



Centerline plane temperature profile: 3 minutes, 3.1 ft/s

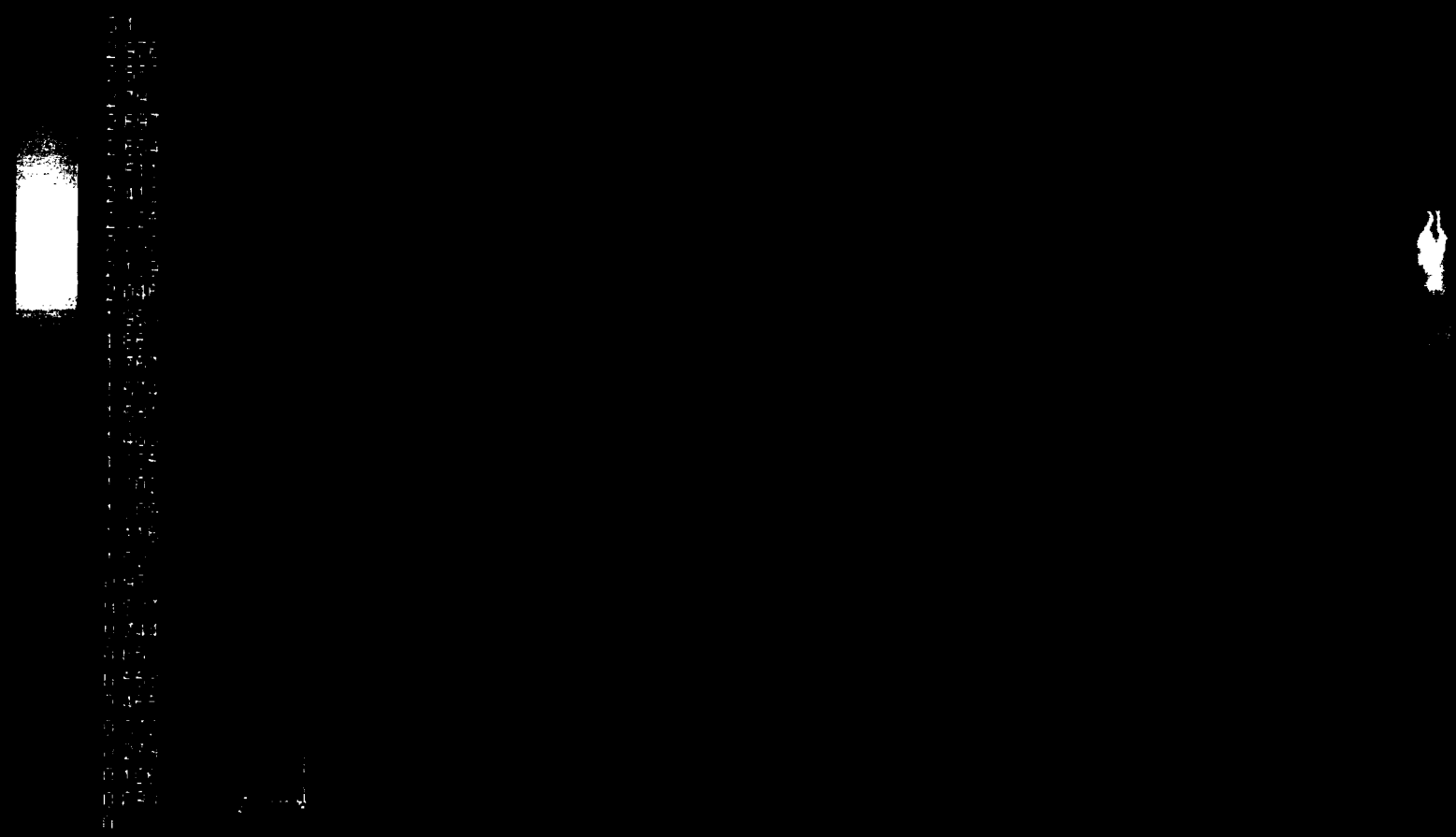


Centerline plane temperature profile: 3 minutes, 3.1 ft/s



Centerline plane velocity profile: 6 minutes, 6.1 ft/s

Centerline plane velocity profile: 6 minutes, 3.1 ft/s

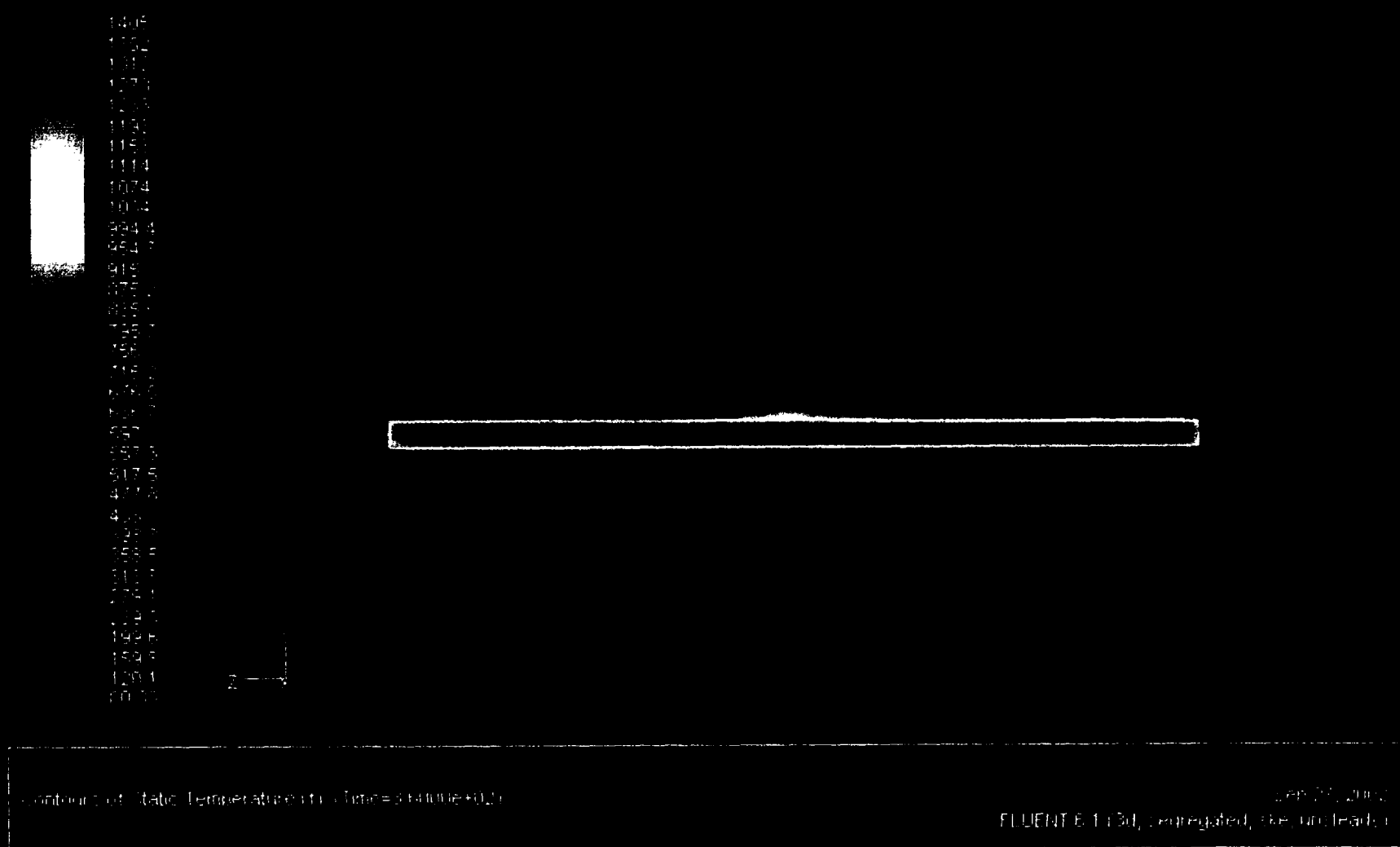


Contours of Velocity Magnitude (ft/s) (Time=3.6000e+02)

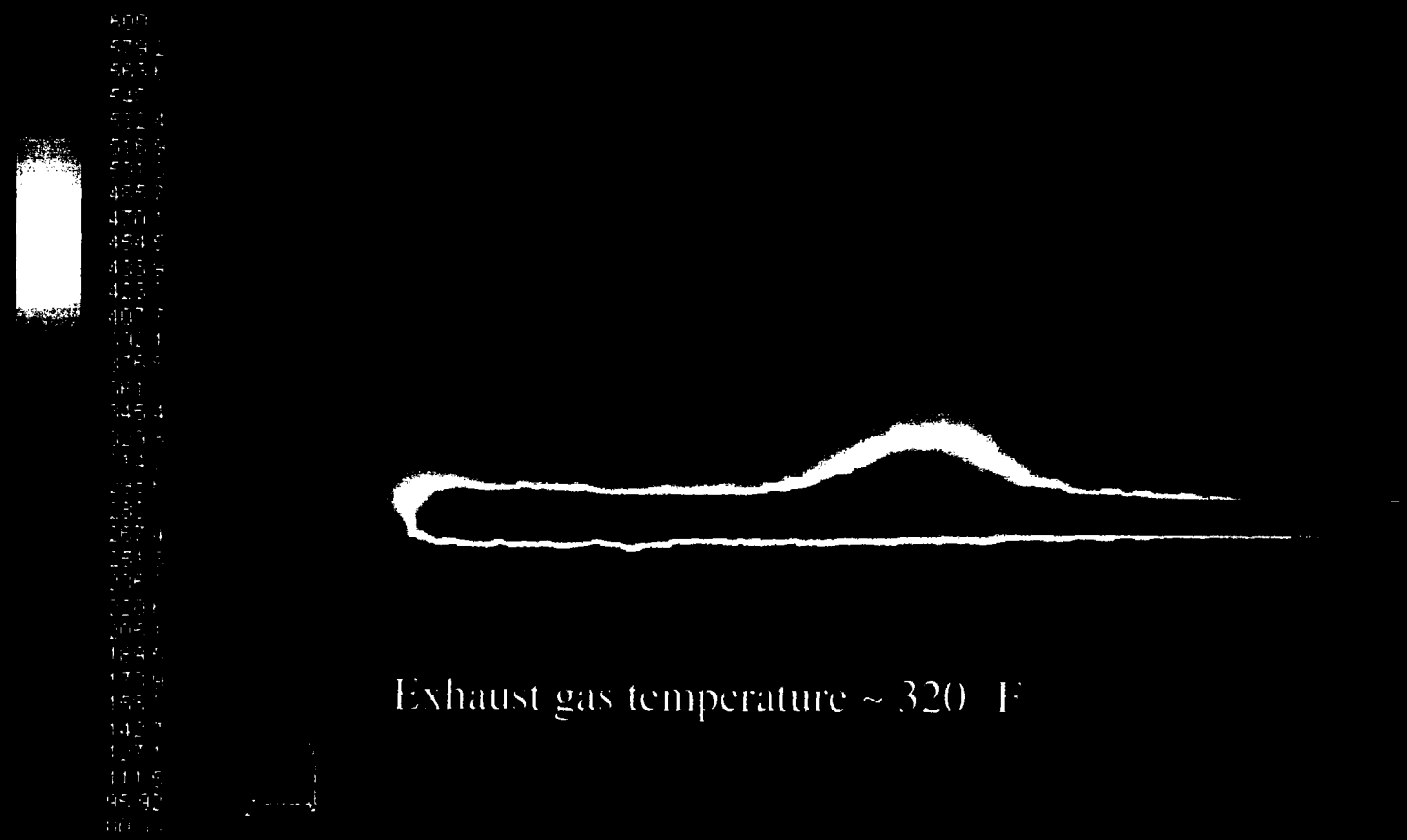
Sep 27, 2002

FLUENT 6.1 (3d, segregated, ske, unsteady)

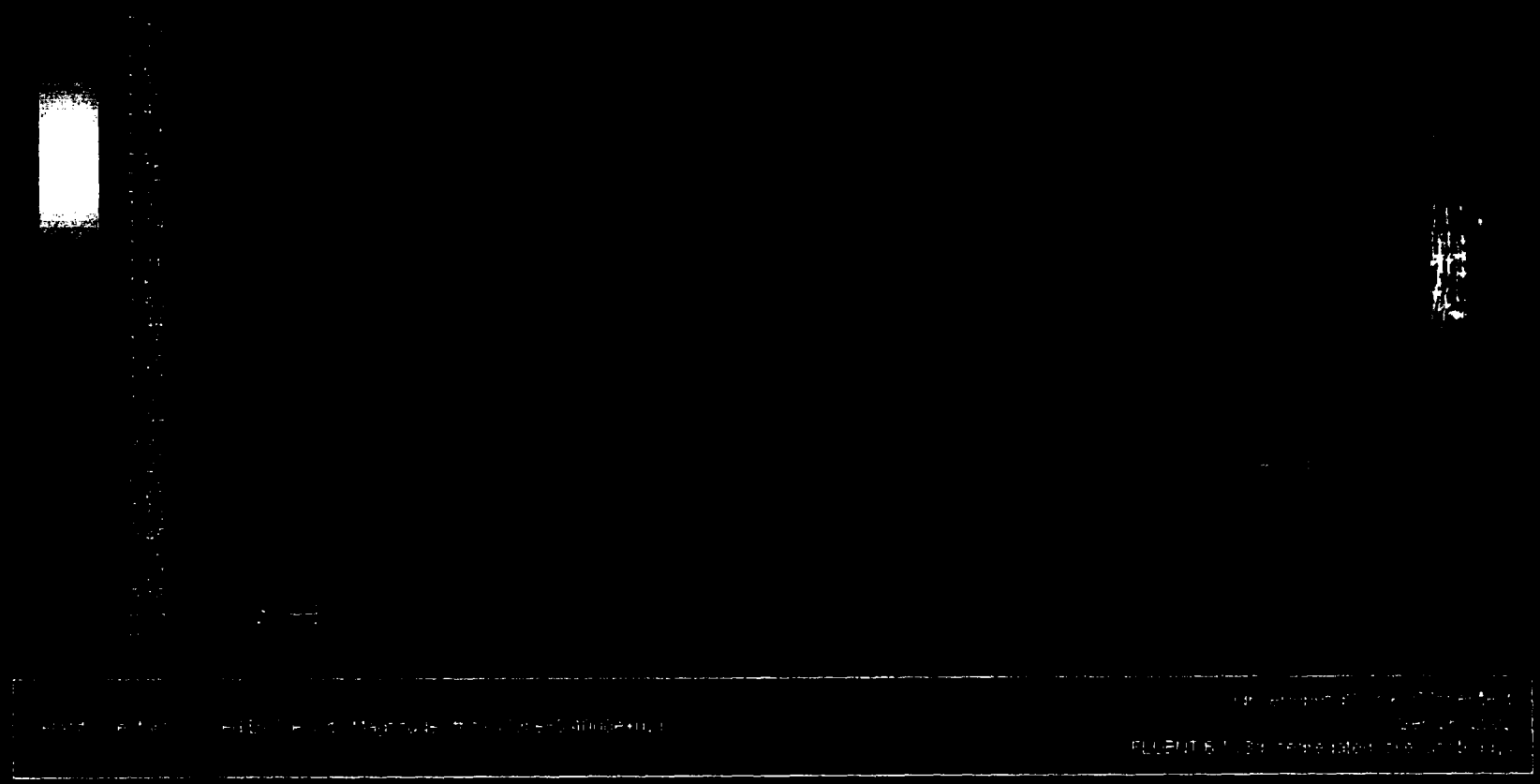
Centerline plane temperature profile: 6 minutes, 3.1 ft/s



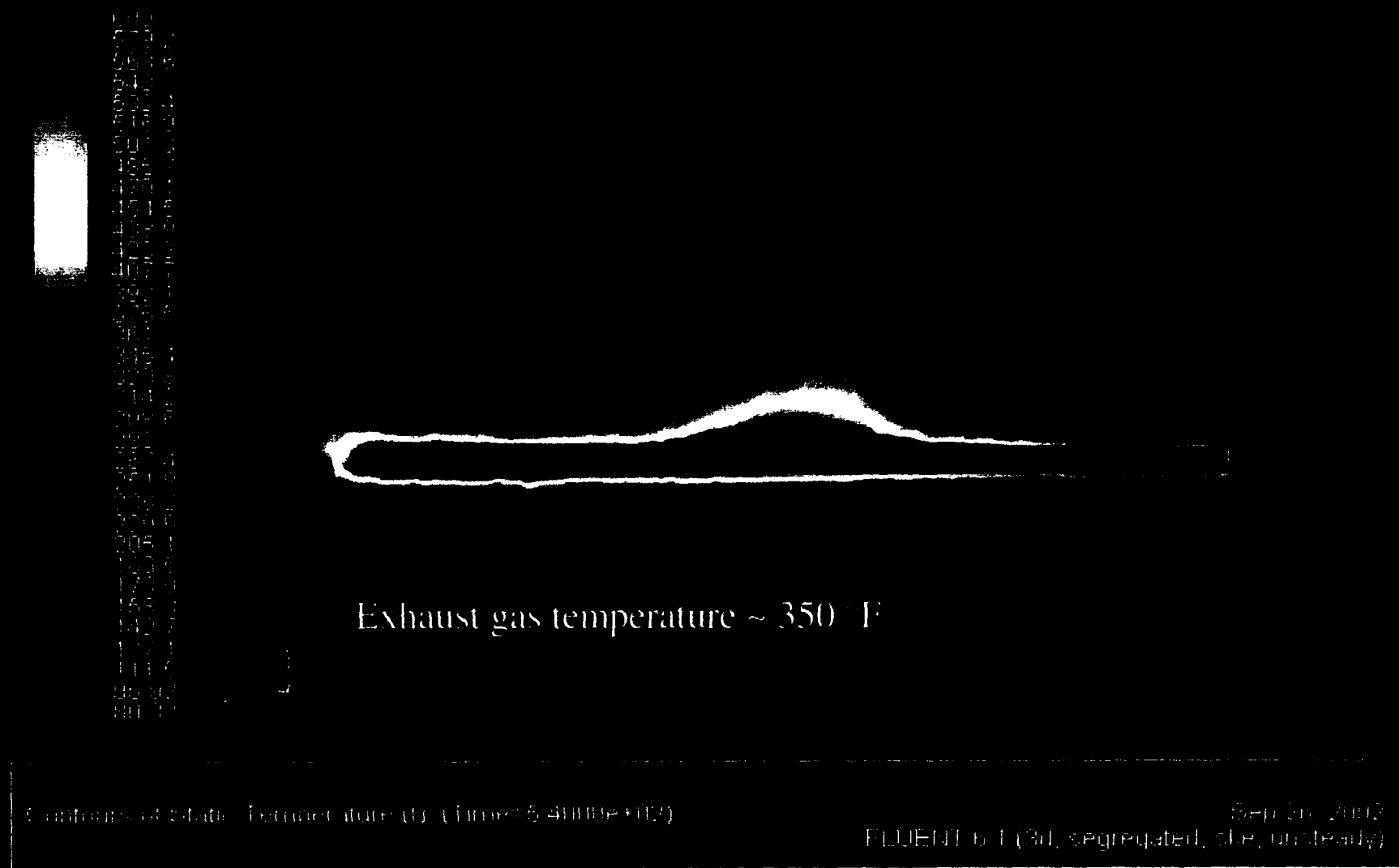
Centerline plane temperature profile: 6 minutes, 3.1 ft/s



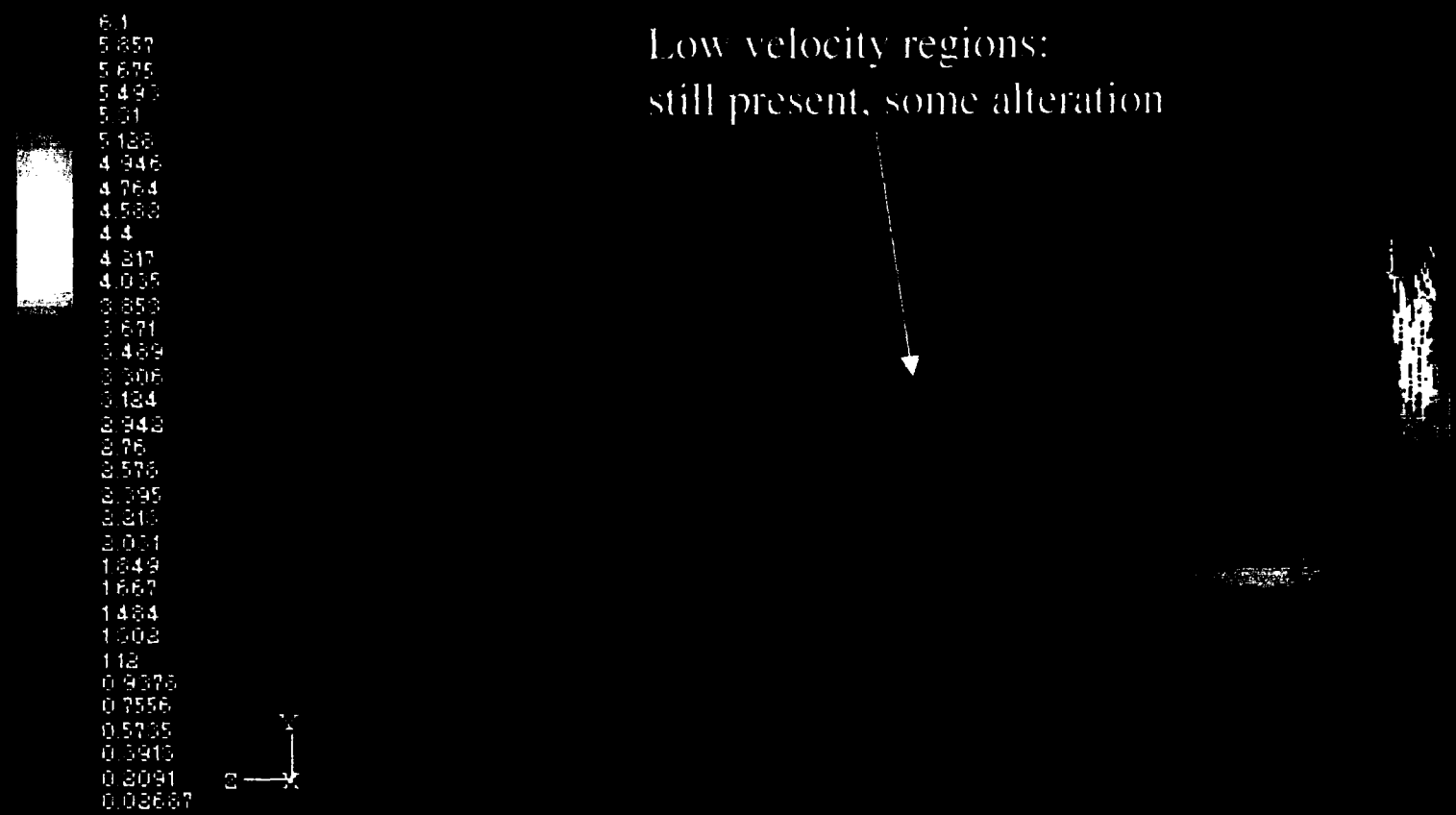
Centerline plane velocity profile: 9 minutes. 3.1 ft/s



Centerline plane temperature profile: 9 minutes, 3.1 ft/s



Centerline plane velocity profile: 3 minutes, 6.1 ft/s



Velocity Vectors Colored By Velocity Magnitude (ft/s) (Time=1.8000e+03)

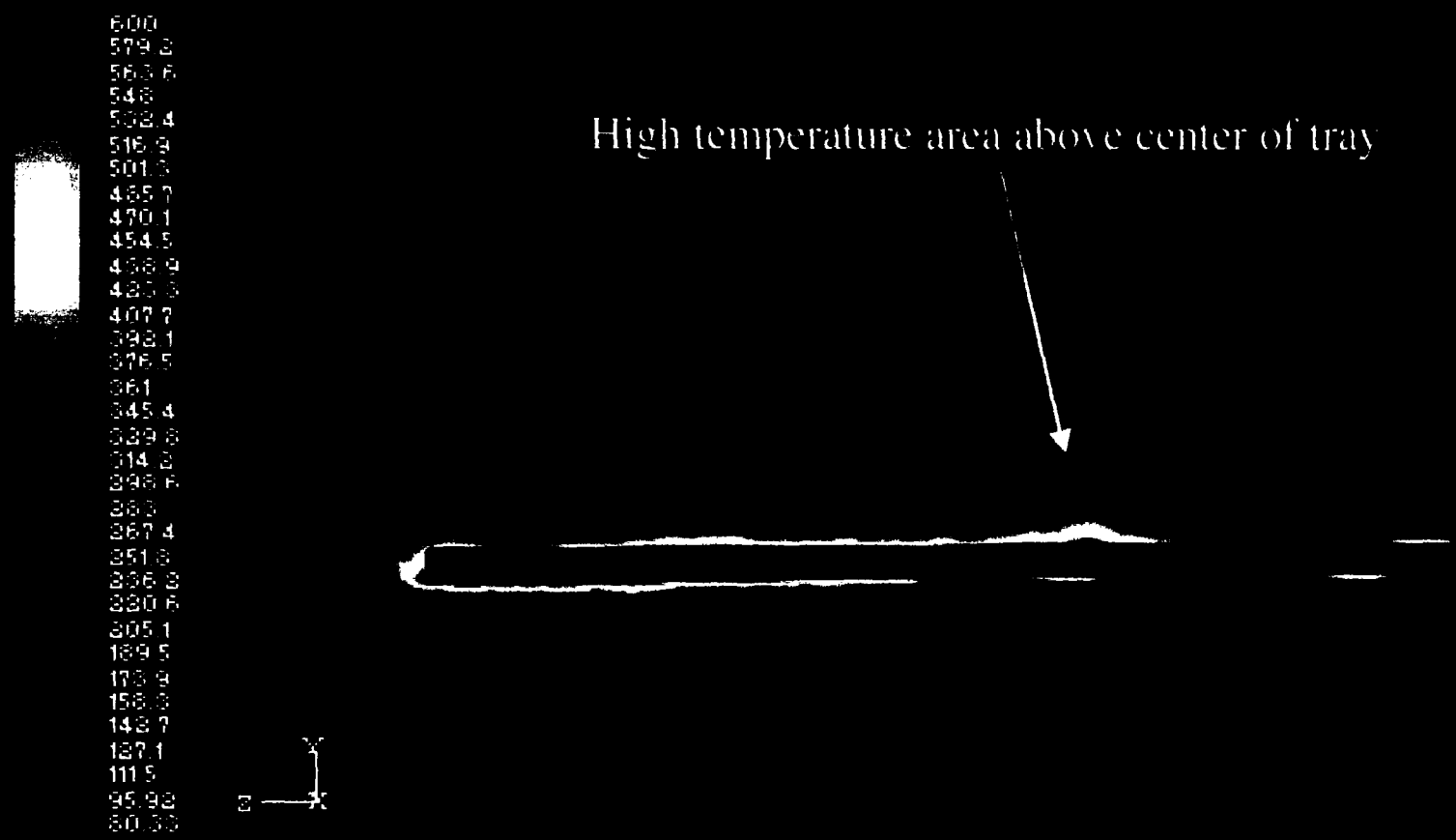
Sep 27, 2003
FLUENT 5.1 (3d, segregated, ske, unsteady)

Centerline plane temperature profile: 3 minutes, 6.1 ft/s

Contours of Static Temperature (t) (Time=1.6000e+02)

Sep 27, 2002
FLUENT 6.1 (3d, segregated, ske, unsteady)

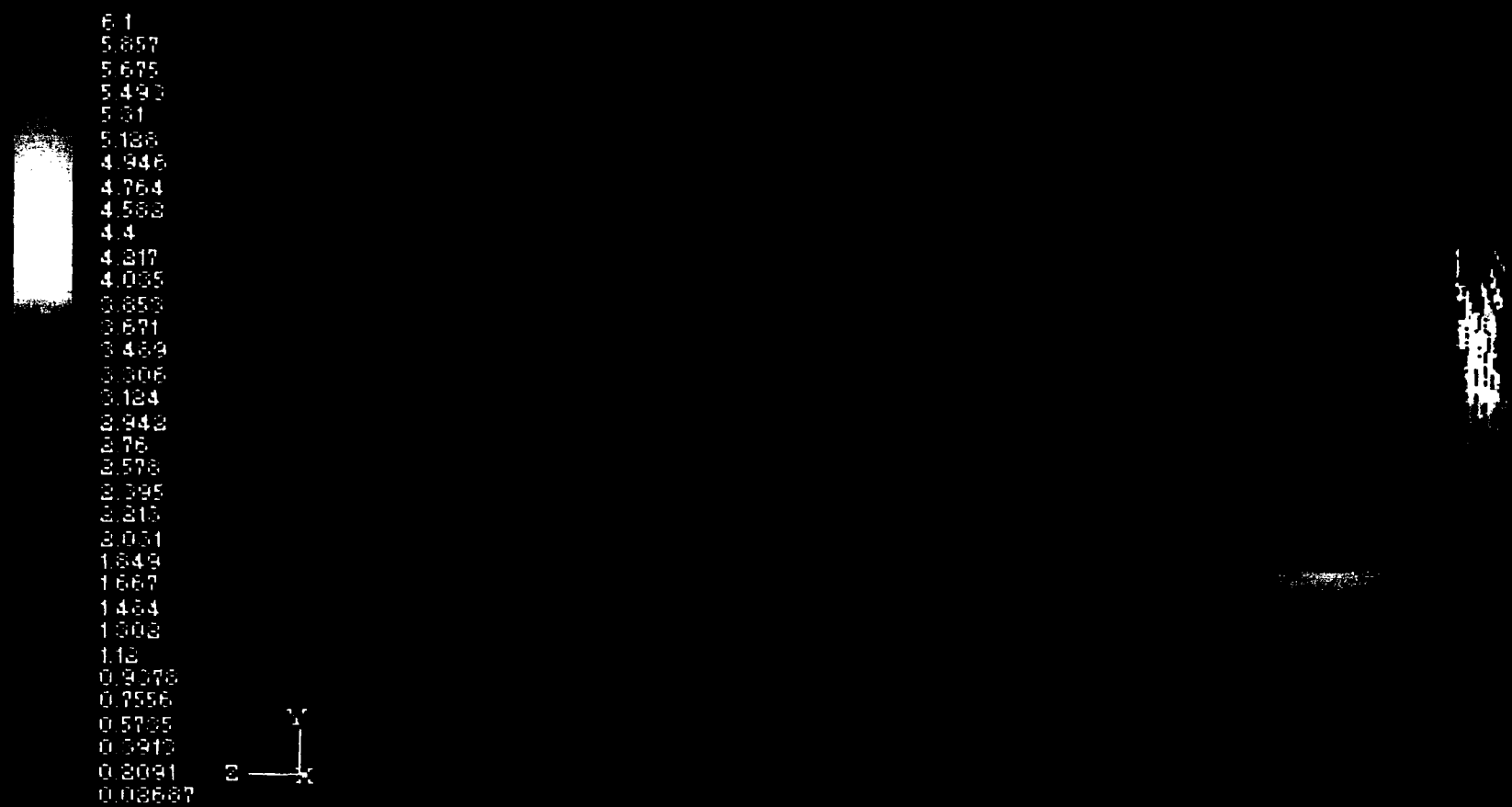
Centerline plane temperature profile: 3 minutes, 6.1 ft/s



Contours of Static Temperature (t) (Time=1.8000e+02)

Sep 27, 2002
FLUENT 6.1 (3d, segregated, ske, unsteady)

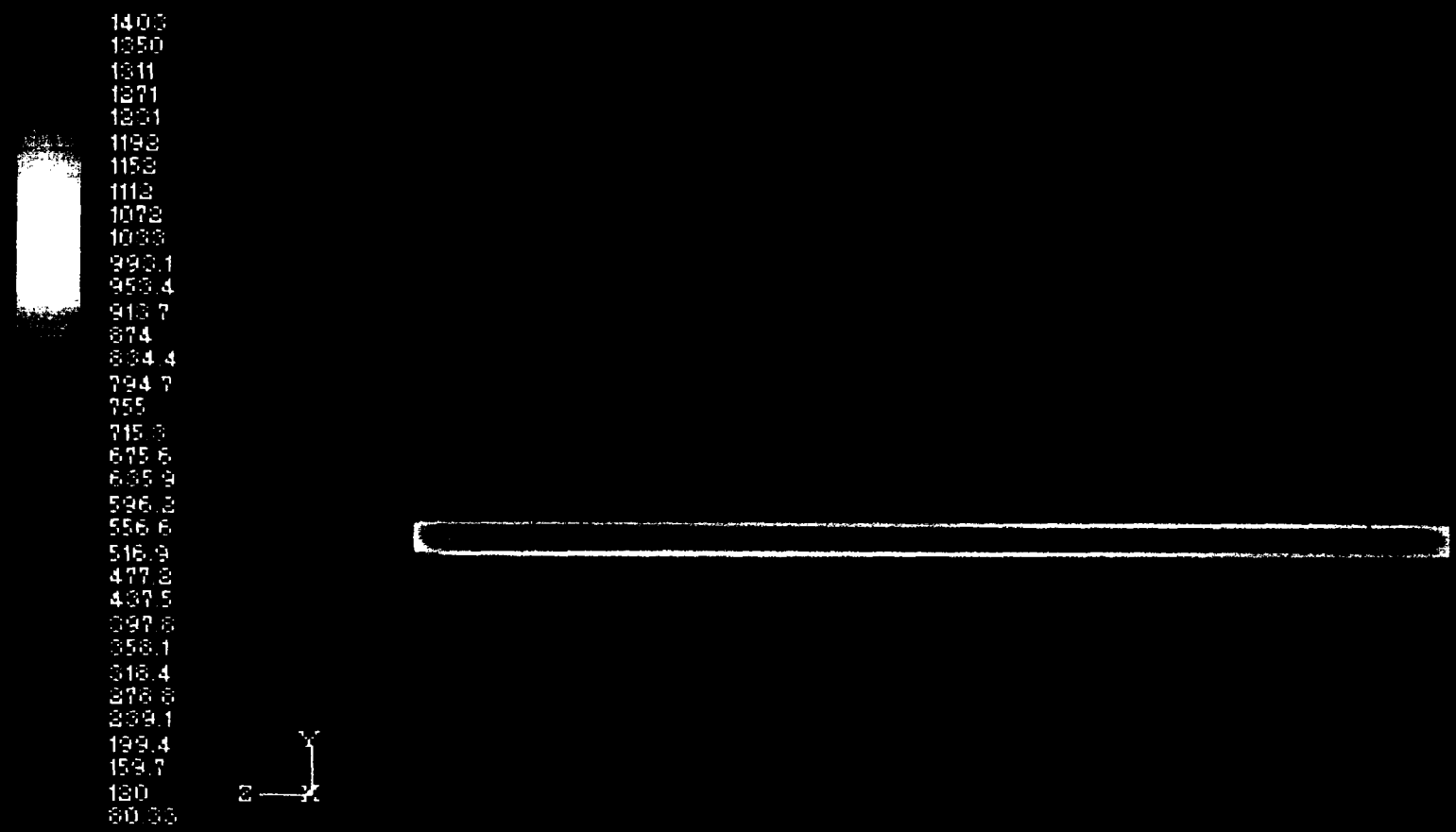
Centerline plane velocity profile: 6 minutes, 6.1 ft/s



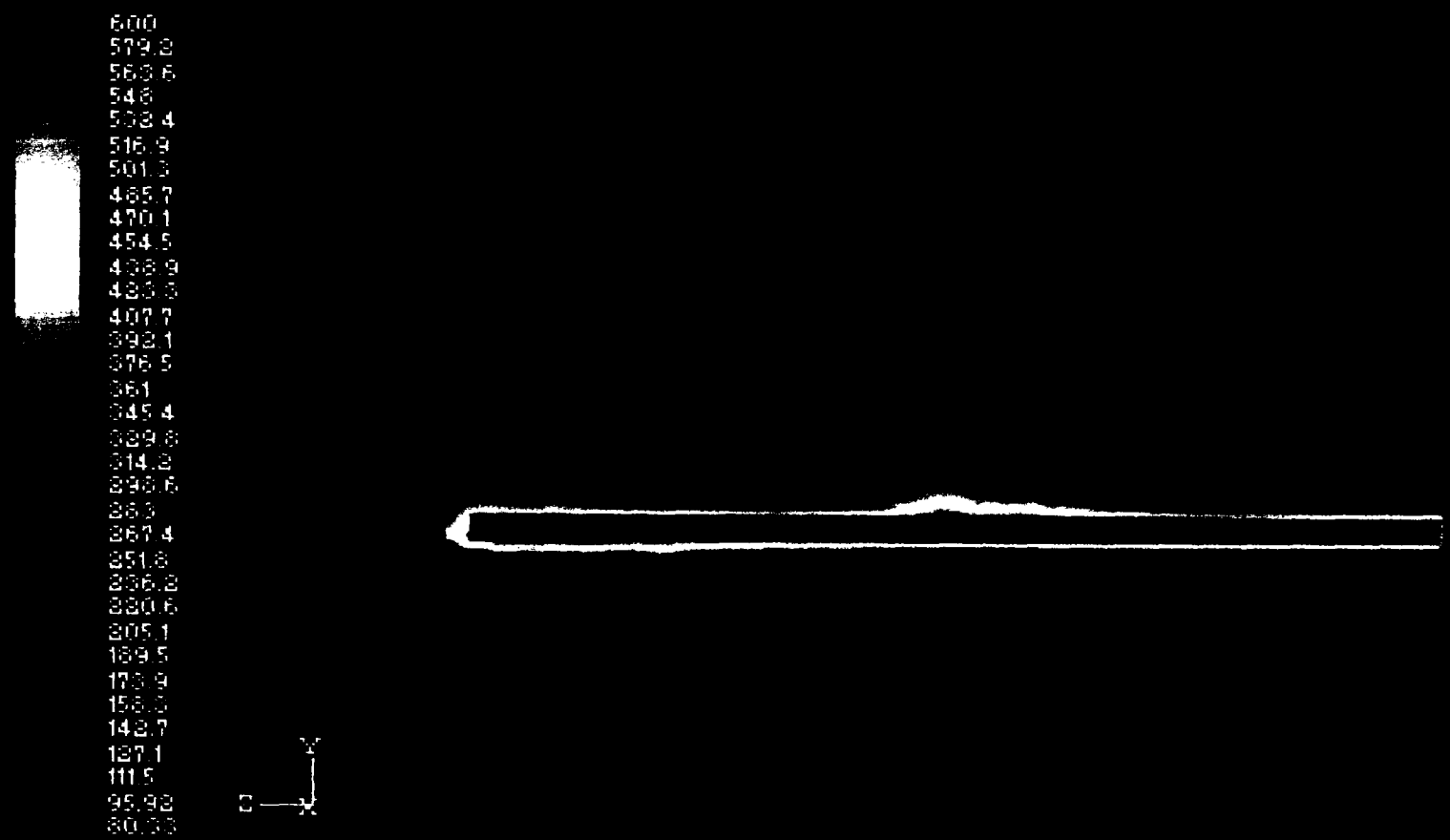
Velocity Vectors Colored By Velocity Magnitude (ft/s) (Time=3.6000e+03)

Sep 27, 2002
FLUENT 6.1 (3d, segregated, ske, unsteady)

Centerline plane temperature profile: 6 minutes, 6.1 ft/s



Centerline plane temperature profile: 6 minutes, 6.1 ft/s

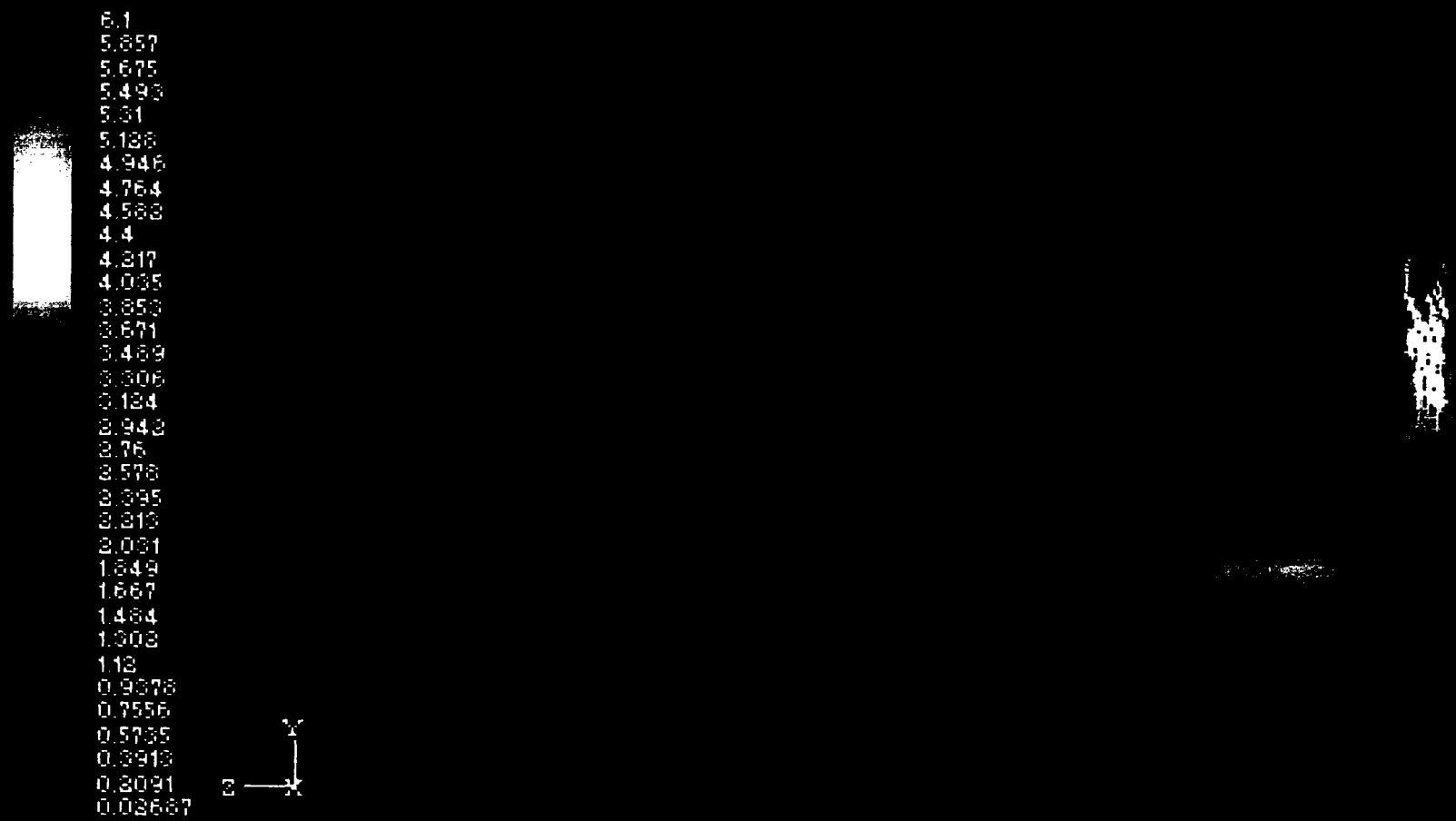


Contours of Static Temperature (t) (Time=3.6000e+02)

Sep 27, 2008

FLUENT 6.1 (3d, segregated, ske, unsteady)

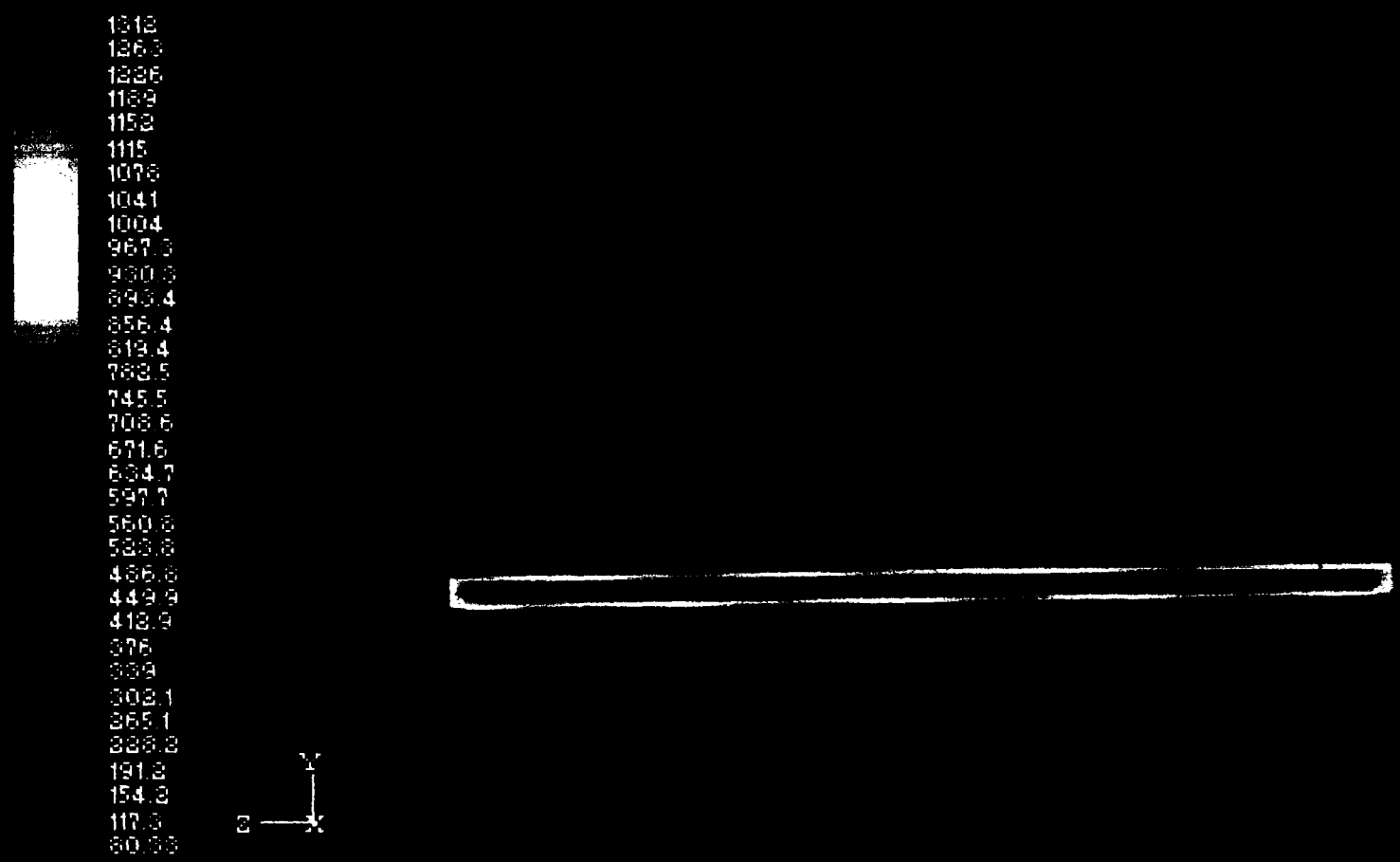
Centerline plane velocity profile: 9 minutes, 6.1 ft/s



Velocity Vectors Colored By Velocity Magnitude (ft/s) (Time=5.4000e+02)

Sep 27, 2003
FLUENT 6.1 (3d, segregated, ske, unsteady)

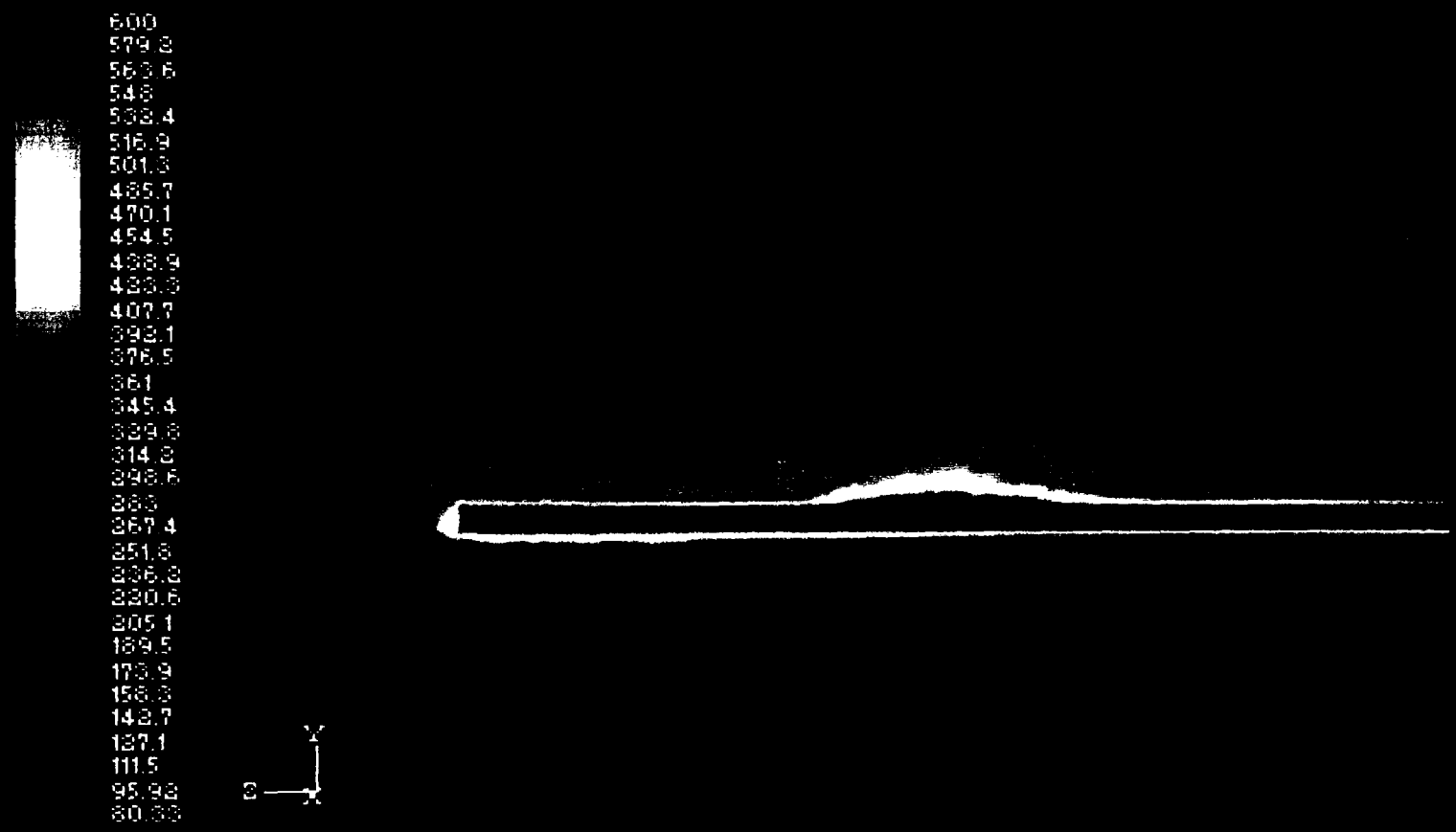
Centerline plane temperature profile: 9 minutes, 6.1 ft/s



Contours of Static Temperature (t) (Time=5.4000e+03)

Sep 27, 2002
FLUENT 6.1 (3d, segregated, ske, unsteady)

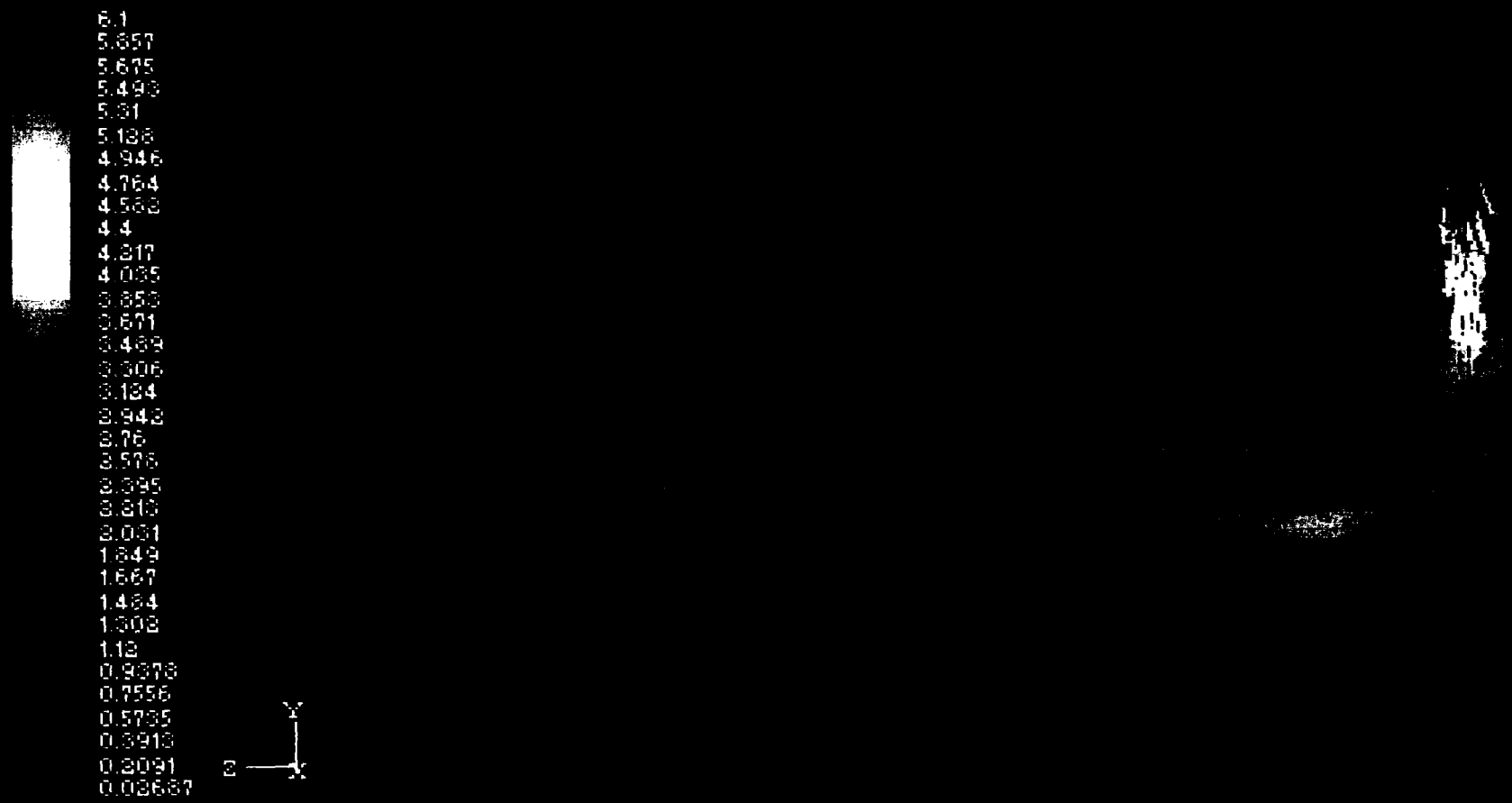
Centerline plane temperature profile: 9 minutes, 6.1 ft/s



Contours of Static Temperature (f) (Time=5.4000e+02)

Sep 27, 2002
FLUENT 6.1 (3d, segregated, ske, unsteady)

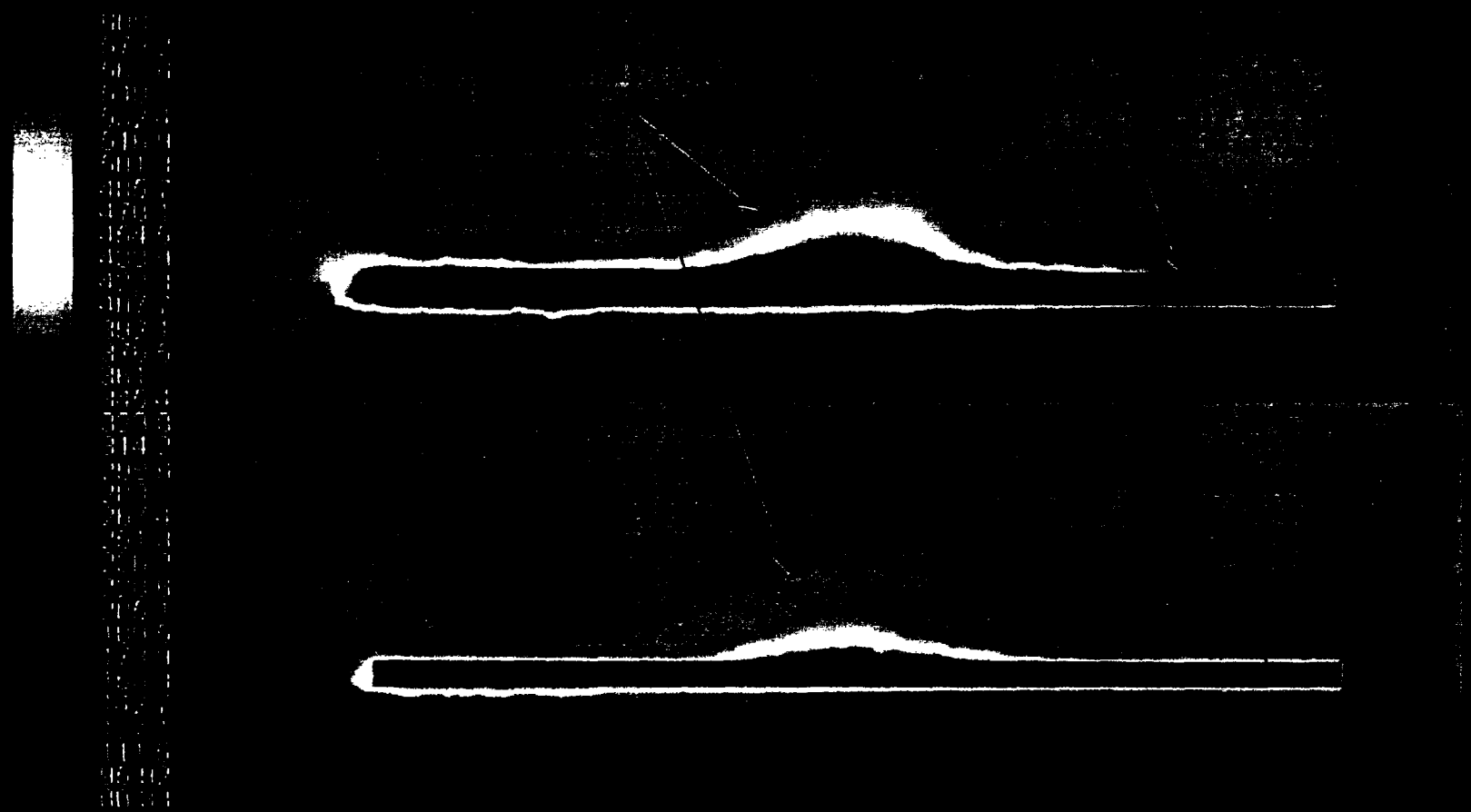
Centerline plane velocity profile: 150 minutes, 6.1 ft/s



Velocity Vectors Colored By Velocity Magnitude (ft/s) (Time=9.0000e+03)

Sep 27, 2002
FLUENT 6.1 (3d, segregated, ske, unsteady)

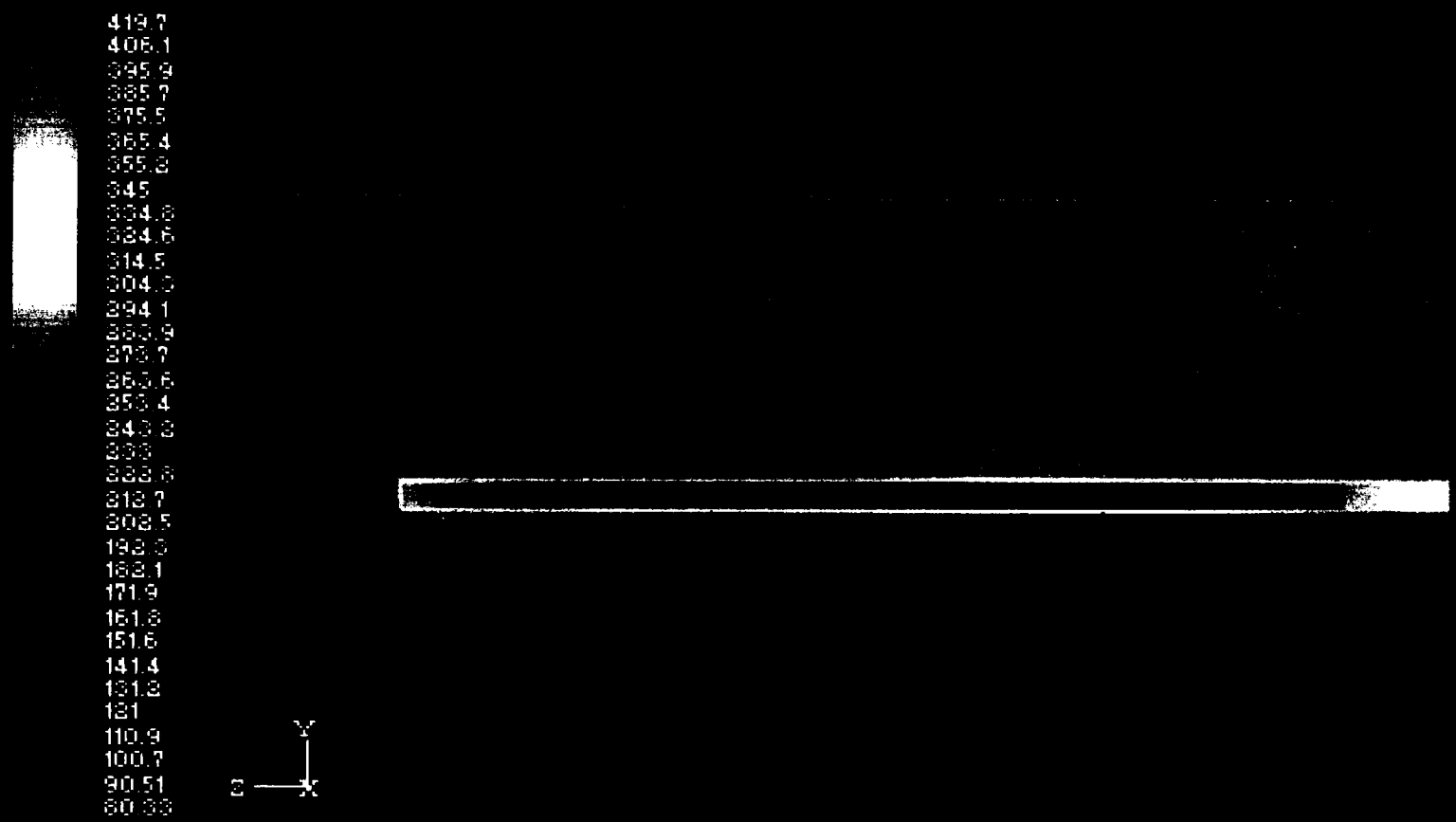
Centerline plane temperature comparisons: 9 minutes



3.1 ft/s

6.1 ft/s

Centerline plane temperature profile: 150 minutes, 6.1 ft/s



Contours of Static Temperature (t) (Time=9.0000e+03)

Sep 27, 2003
FLUENT 6.1 (3d, segregated, ske, unsteady)

Initial conclusions

- Exhaust gas temperature predictions are in reasonable agreement with JACADS DAL temperature measurements
  - Model initial parameters must be modified to match initially higher exhaust gas temperature

- There are poorly mixed regions in the DAL resulting in locally high temperatures

- Increased gas velocity (6.1 ft/s)
  - Alters cooling profile
  - Poorly mixed regions remain

- Subsequent work
  - Adjust initial constraints (e.g. DAL wall boundary conditions)
  - Evaluate effects on increasing flow rate
  - Evaluate effect of alternative inlet gas port design

APPENDIX E

Analysis of VX in Residue Sample from MPF DAL Tray Number 153

Notes on SIMS Analysis of Johnston Island Incinerator Ash

G. S. Groenewold, A. D. Appelhans, G. L. Gresham, January, 2003

Background

Samples of incinerator ash from Johnston Island were received at the West Desert Test Center (WDTC), Dugway Proving Ground, for analysis for VX and related compounds. Prior analyses indicated that VX was present in the ash sample, at low concentrations. These analyses were performed using thermal desorption gas chromatography (TDGC) at WDTC, and gas and liquid chromatography/ mass spectrometry (GC/MS and LC/MS) at Southwest Research Institute (SwRI).

TDGC with flame photometric (FPD), and mass spectrometric detection was used for the analysis at WDTC. The results of this analysis were that low concentrations of VX were present, but could not be quantified because of high background of other organics that were present.

GC/MS and GC/FPD quantitation using both direct, and G-analog approaches were conducted by SwRI, and showed the presence of VX at between 1 and 5 ppm. Quantitation using a direct LC/MS also resulted in detection of a VX concentration within this range. High chemical background was encountered in these analyses. Detection of VX was confirmed using an MS/MS analysis.

The sample was then analyzed using an ion trap secondary ion mass spectrometer (IT-SIMS) that was fabricated at the Idaho National Engineering and Environmental Laboratory (INEEL), and operated at the WDTC. The instrument functions on a substantially different principal than either the TDGC or the extraction GC/MS or LC/MS approaches. Adsorbed organics, like VX and its degradation products, were sputtered into the gas phase, and detected using single stage or multiple stage mass spectrometry (MS or MS/MS). Instrument performance was ascertained by positive analysis of VX on soil particles that exposed to low levels.

Results

MS analyses conducted using the IT-SIMS resulted in a complex spectrum (Figure 1). Signature ions for VX were observed at *m/z* 268 and 128, but the abundance of 268 was very low compared with other ions in the spectrum. The presence of traces of VX on this sample was supported by the MS/MS analysis of *m/z* 268, which was induced to undergo fragmentation to *m/z* 128 (Figure 2). Quantitation studies were not performed, however, for low surface area samples, minimum detection limits have been in the vicinity of 1 ppm. If the incinerator ash has a surface area in the 2 – 5 m^2g^{-1} range, then a VX concentration in the 1 to 5 ppm range would be estimated for this sample.

The limiting factor in the various analyses of the incinerator ash was the presence of high concentrations of other degradation products. The IT-SIMS spectra of the sample offered some insight into the nature of the other compounds present. The abundant ions at *m/z* 162, 160, 114, and a portion of

128 in the MS-1 spectrum arise in part from the VX hydrolysis produce 2-diisopropylaminoethane thiol (DESH). These ions, together with *m/z* 321 constitute the ion signature for the DESH dimer, *bis*(diisopropylaminoethyl) disulfide. The prominent ion at *m/z* 225 is consistent with the mass anticipated from protonated dicyclohexyl urea, an assignment that is substantiated by the formation of a fragment ion at *m/z* 98, also observed in abundance in the spectrum. Dicyclohexyl urea is a VX stabilizer. The abundant ions at *m/z* 306 and 336 have not yet been identified to our satisfaction, but most likely arise from degradation/condensation processes involving the stabilizer and or VX.

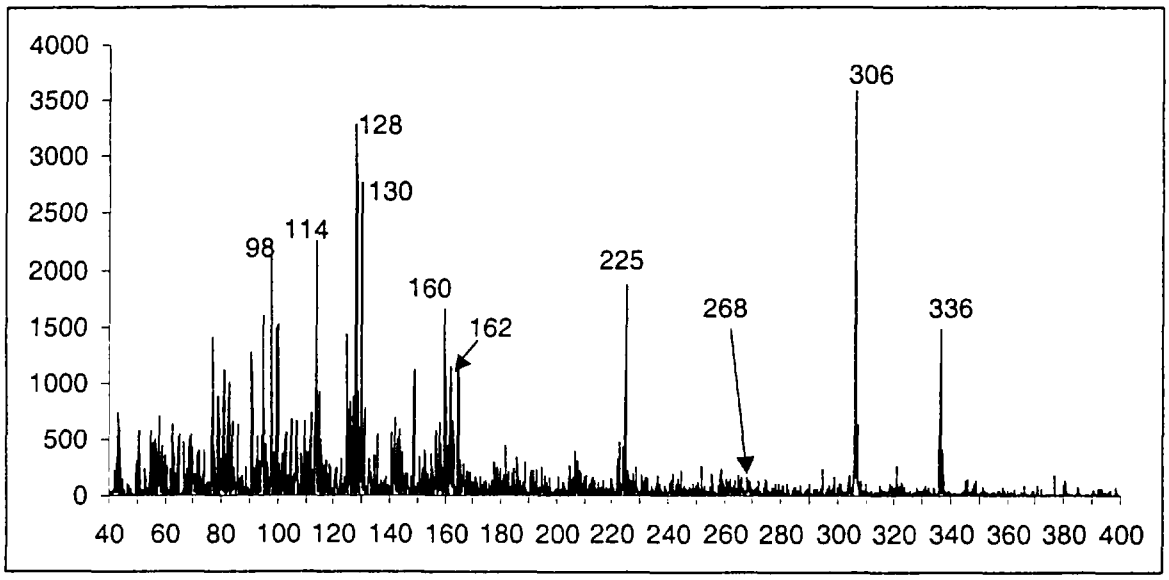


Figure 1. MS-1 analysis of incinerator ash using the IT-SIMS at WDTC. Positive ion spectrum.

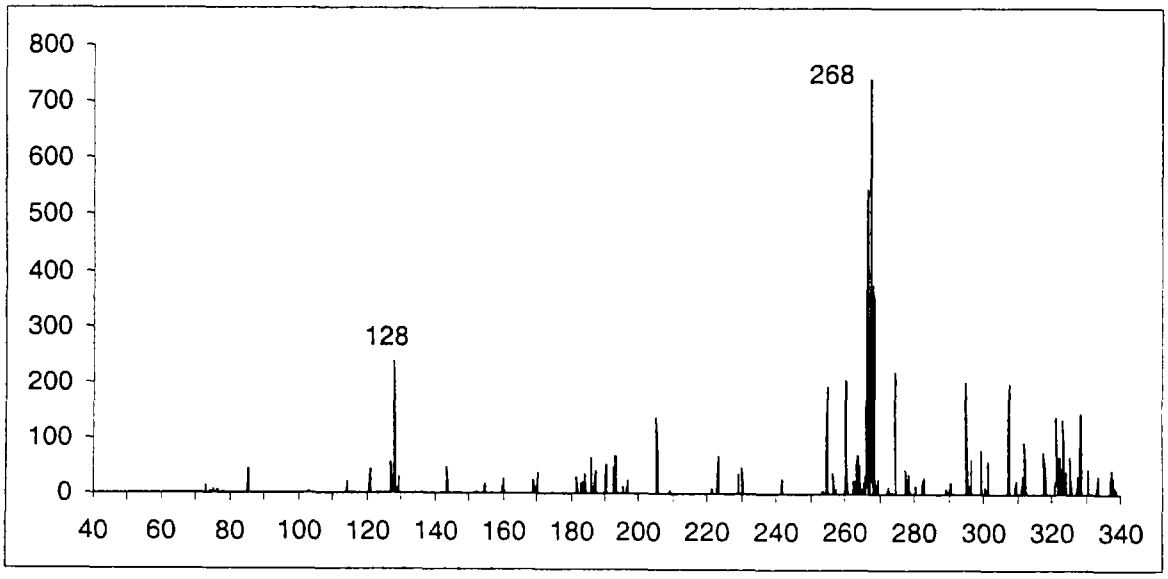


Figure 2. MS/MS analysis of incinerator ash. M/z 268 was isolated ($[VX + H]^+$), and then induced to undergo collision induced fragmentation.

Extract of VX from Tray 153 Ash with Nonchlorinated Solvents

Supplement to

Analysis of VX in Residue Sample from MPF DAL Tray Number 153

FINAL REPORT

Subcontract 99000200
Prime Contract DAAA-09-96-C-0081
SwRI Project 01-05935

Prepared by

Joseph H. Brewer
Michael G. MacNaughton, Ph.D.

Prepared for

Washington Demilitarization Company
2850 Pa'a – Suite 100
Honolulu, Hawaii 96819

January 3, 2003

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January 3, 2003

APPROVED:

Michael G. MacNaughton, Ph.D., P.E.
Vice President
Chemistry and Chemical Engineering Division

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1. **BACKGROUND** 1

2. **APPROACH** 1
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   2.2 Sample Preparation for NMR Analysis 2

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   3.2 NMR Analysis 3

4. **CONCLUSIONS** 3

APPENDICES
A. GC/FPD DATA
B. GC/MSD DATA
C. LC/MS DATA
D. NMR DATA

1. BACKGROUND

The JACADS laboratory sample extraction procedure for VX uses chloroform to concentrate the VX in a liquid sample that can be injected onto a DAAMS tube. While this indirect method is very sensitive and conservative, previous investigations have shown that under the right conditions it is possible to form VX from decomposition products at a high temperature such as with the DAAMS desorber or the inlet of the gas chromatograph, if chloroform is used as an extract solvent.[1,2,3] It has also been reported in the PMCD Alternative Technology and Approaches program that analyzing VX in the presence of high concentrations of VX hydrolysis products, there is a high probability of false positives unless very selective GC/MS/MS techniques are used to directly analyze VX, not the G-analog. While the methods used at JACADS gave unambiguous confirmation of VX in the ash according to the G-analog methods required by PMCD and in the environmental permit, there is still a probability that these positives were caused by interferents. Of particular concern would be the compounds that form the G-analog upon reaction with the AgF pad. This would have to be an organophosphate with a phosphorous-sulfur bond. Froment identified one suspect compound among the 80 plus byproducts of the pyrolytic decomposition of VX at 245°C (473°F).[4] Of a number of organophosphates with organosulfur end groups identified in the residue, one compound in particular, $CH_3P(O)(OCH_2CH_3)(SCH_2CH_3)$, would easily form the ethyl methylphosphonofluoridate upon reaction with the AgF pad. This should be verified by more selective direct methods not as prone to false positives.

The extraction procedures used by SwRI and IITRI to confirm the presence of VX in the ash sample used 10% isopropyl alcohol in dichloromethane. While this extraction solvent has never been implicated in forming VX, some reviewers raised concerns because it is a chlorinated solvent. To verify that the positive VX results reported by both SwRI and IITRI were not the result of VX formation, the Tray 153 ash was extracted and analyzed again using a nonclorinated solvent.

2. APPROACH

A number of nonchlorinated solvents were evaluated including pH 4 water, hexane, hexane/diethyl ether, toluene, acetonitrile, ethyl acetate, and acetone. The best results were obtained with acetone. This solvent was selected to obtain samples for gas chromatography/mass spectrometric detector (GC/MSD) and liquid chromatography/mass spectrometry (LC/MS).

2.1 Extractions for GC/LC/MSD Analysis

Approximately 2 g of ash were extracted using two aliquots of acetone, 4 mL each. The samples and acetone were rotary extracted for 1 hour, centrifuged, and filtered through a 0.45 μ filter. This process was repeated on the residue using another 4 mL of acetone. Both acetone fractions were combined and the resulting final volume measured as 7 mL. Two hundred microliters (200 mL) of VX at 0.1 μg/μL was added to spiked samples. Three replicate 7-mL fractions were assayed using gas chromatography/flame photometric detector (GC/FPD) by the analog technique and directly as VX by GC/MS. For LC/MS, a

---

[1]Nerve Agent VX Irreversibility Test Report, PMCD Report , January 29, 1997.

[2] Beaudry, W.T. and Szafranic, L.L., NMR Characterization of pH Adjusted VX/Caustic and $MeCl_2$ Extracts, July 1996.

[3]Jakubowski, E.M., False Positive Results in VX and G-Agent Analysis Methods: Agent Formation During Hydrolysis Sample Handling and Preparation, Programmatic Lesson Learned Technical Bulletin.

[4]Froment, D., *In Situ* Pryrolysis, Supplement to the Proceedings from the 6th CBW Protection Symposium, May 1998.

50 µL aliquot was diluted into 200 µL mobile phase. This dilution was injected directly into the instrument.

2.2 Sample Preparation for NMR Analysis

To reach the ~20 ppm detection limit of the nuclear magnetic resonance (NMR), samples were generated by combining the extracts of approximately 15 g of charcoal. The residual extracts from the three extracts used for GC/MSD and LC/MS were combined with an additional extract of 10 g of ash. Extraction was performed in the same manner as was used for GC/MS but the extraction time on the first fraction was 16 hours instead of 1 hour. The 10 grams were extracted with 30-mL and 20-mL fractions of acetone. These two fractions, along with approximately 18 mL of the remaining GC/MS extract volume (triplicate at 7 mL each) were combined and concentrated to 1 mL final volume. During the concentration step at approximately 4 mL total volume, a white precipitate was formed. The sample was filtered through glass wool, rinsed with more acetone, then blown down to 1 mL. Two phases were evident, an aqueous lower phase and a dark organic phase upper phase. The entire final volume was placed into an NMR tube for analysis. After an initial unspiked sample run, the sample was spiked with DMMP as an internal standard and re-analyzed to determine concentration. The sample was also run with a VX spike to verify the peak was VX.

3. RESULTS

3.1 GC/LC/MS Analysis

A summary of the results from analysis of the acetone extracts using GC/FPD, GC/MS, and LC/MS is provided in Table 1 and in Appendices A, B, and C.

Table 1. Analysis of the Tray 153 Ash Using Acetone Extraction

Replicate	FPD Analog (mg/kg)	GC/MSD Direct VX (mg/kg)		LC/MS Direct VX (mg/kg)
		Ion 139	Ion 167	
1	1.3	1.7	4.4	1.6
2	0.9	1.6	3.5	1.5
3	1.4	2.1	4.7	1.6
Average	1.2	1.8	4.2	1.6
% RSD	24	15	21.5	2.6
MS Recovery (%)	82	140	163	72
MSD Recovery (%)	83	127	133	96

These results are close to the concentrations reported for the 10% IPA/DCM extract. The GC/FPD analog analysis of the nonchlorinated extracts was approximately 30% of the 4.2 mg/kg reported for the DCM extract. The GC/MSD (ion 167) results were 183% and the LC/MS was 108% of the DCM extracts. The analytical results of both IPA/DCM and acetone extracts indicate that the ash contains VX at approximately 2 ± 2 mg/kg.

3.2 Semiquantitative NMR Analysis

The samples were semiquantitated by ^{31}P NMR using a Varian VXR-300 spectrometer. Data were acquired in a gated decoupling mode with long (overnight) acquisitions to give maximum sensitivity. An external phosphoric acid reference was used to determine the chemical shifts of the signals.

The final extract was a 1-mL sample in two separate phases of 500 µL each. During the final phases of extract preparation, d_6-acetone was included in the extract as a lock solvent for the NMR. The upper phase was filtered through $MgSO_4$ to give a suitable NMR sample and analyzed. No VX was detected. The lower phase was shown by proton NMR to be mostly water. This lower phase was analyzed and shown to contain VX. In an attempt to quantify the VX level, the NMR tube with this phase was spiked with 5 µL of DMMP as an internal standard. Unfortunately, the resulting DMMP signal swamped the VX signal. Since there was no more sample to extract an external standard was prepared in d_6-acetone at ~30 ppm and allowed a semiquantitative estimate of the VX level in the extract of 18 ppm. Based on the 0.5 mL of resulting sample, 15.1 g of ash extracted equates to 0.6 mg/kg. The NMR spectra are shown in Appendix D.

4. CONCLUSIONS

Although previous researchers have identified the potential for forming VX in chlorinated solvents, it does not appear to have occurred in the JACADS analysis of the Tray 153 ash. Four independent analytical methods using 10% IPA in DCM gave results between 2.3 and 4.2 mg/kg. Three independent methods gave results between 1.2 and 4.2 mg/kg using acetone as an extraction solvent. There is no evidence that the VX results in the Tray 153 ash using the G-analog FPD analytical method at JACADS were caused by an interferant or by VX formation during the analytical process.

APPENDIX A

GC/FPD DATA

Acetone Extracts 11/12/02

	Found Mass (ng)	Dilution Factor	DAAMS Spike (uL)	FV (mL)	Sample wt (g)	Sample Conc (ug/g)	avg	
Blank	0	1	8	7				
ASH R1	0.311	10	8	7	2.04	1.33	1.19	
SH R2	0.2	10	8	7	2.02	0.87		%Rec
ASH R3	0.318	10	8	7	2.01	1.38		82%
ASH MS	0.291	80	8	7	2.01	10.13		83%
ASH MSD	0.295	80	8	7	1.98	10.43		

Southwest Research Institute

Agent Analysis

Sample ID: CCS 0.8 ng VX

Sample Type: Unknown

Acq Method Set: FISON_VX_MSET

Date Acquired: 11/13/02 1:37:53 PM

Report Method Name: cmp_report

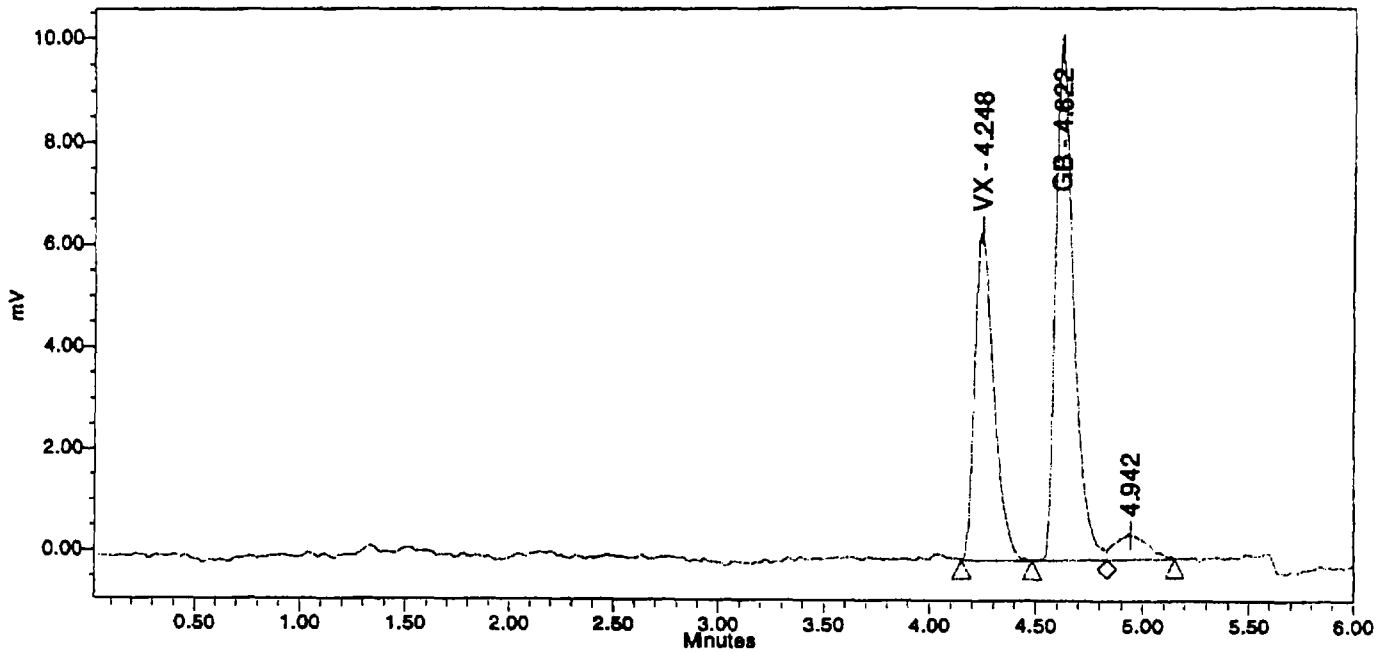
Project Name: MiscAgentwork

User Name: bwilliamson

Processing Method: Fisions_FPD_VX_GB

Date Processed: 11/13/02 1:44:08 PM

Auto-Scaled Chromatogram



Reviewed by: _____

Date: _____

Peak Results

	Name	RT	Area	Height	Amount	Units
1	VX	4.248	40169	6412	0.690	ng
2	GB	4.622	58972	10200	0.691	ng
3		4.942	5198	472		

Southwest Research Institute

Agent Analysis

Sample ID: BLK

Sample Type: Unknown

Acq Method Set: FISON_VX_MSET

Date Acquired: 11/13/02 2:05:14 PM

Report Method Name: cmp_report

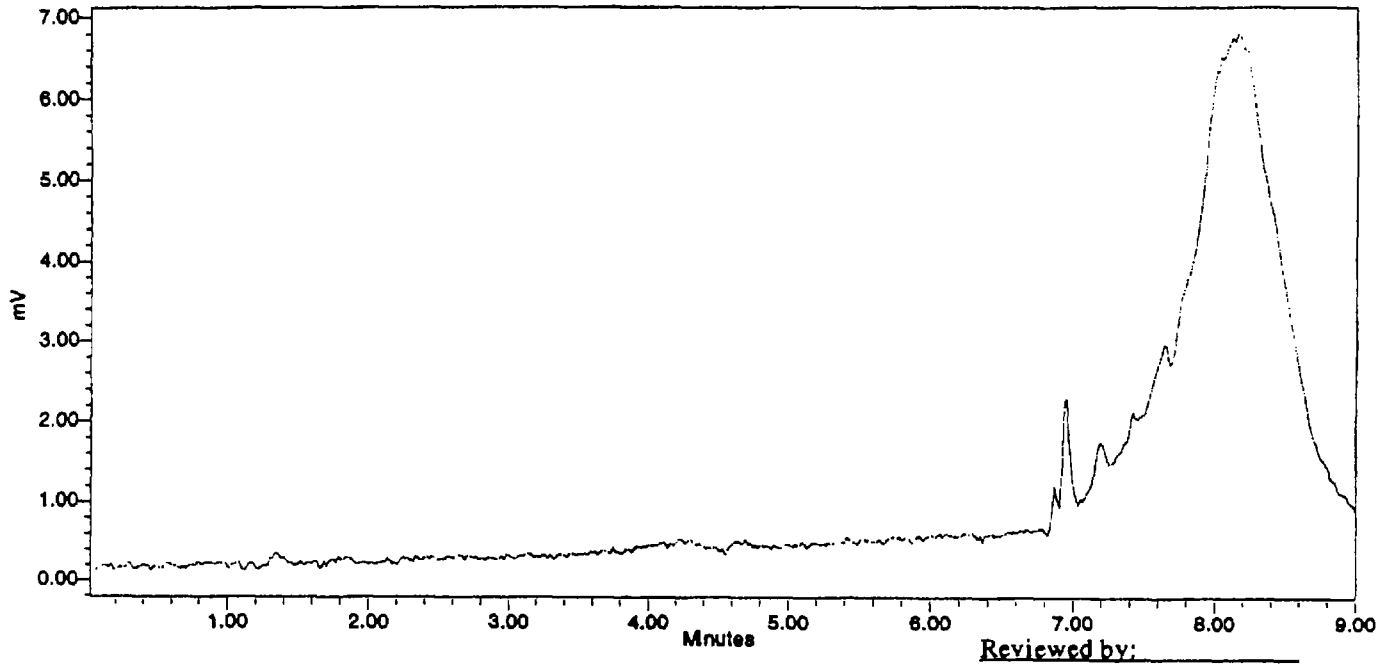
Project Name: MiscAgentwork

User Name: bwilliamson

Processing Method: Fisions_FPD_VX_GB

Date Processed: 11/13/02 2:14:30 PM

Auto-Scaled Chromatogram



Reviewed by: _____

Date: _____

Peak Results

	Name	RT	Area	Height	Amount	Units
1	VX	4.250				
2	GB	4.620				

Southwest Research Institute

Agent Analysis

Sample ID: Tray 153 R1

Sample Type: Unknown

Acq Method Set: FISON_VX_MSET

Date Acquired: 11/13/02 2:16:07 PM

Report Method Name: cmp_report

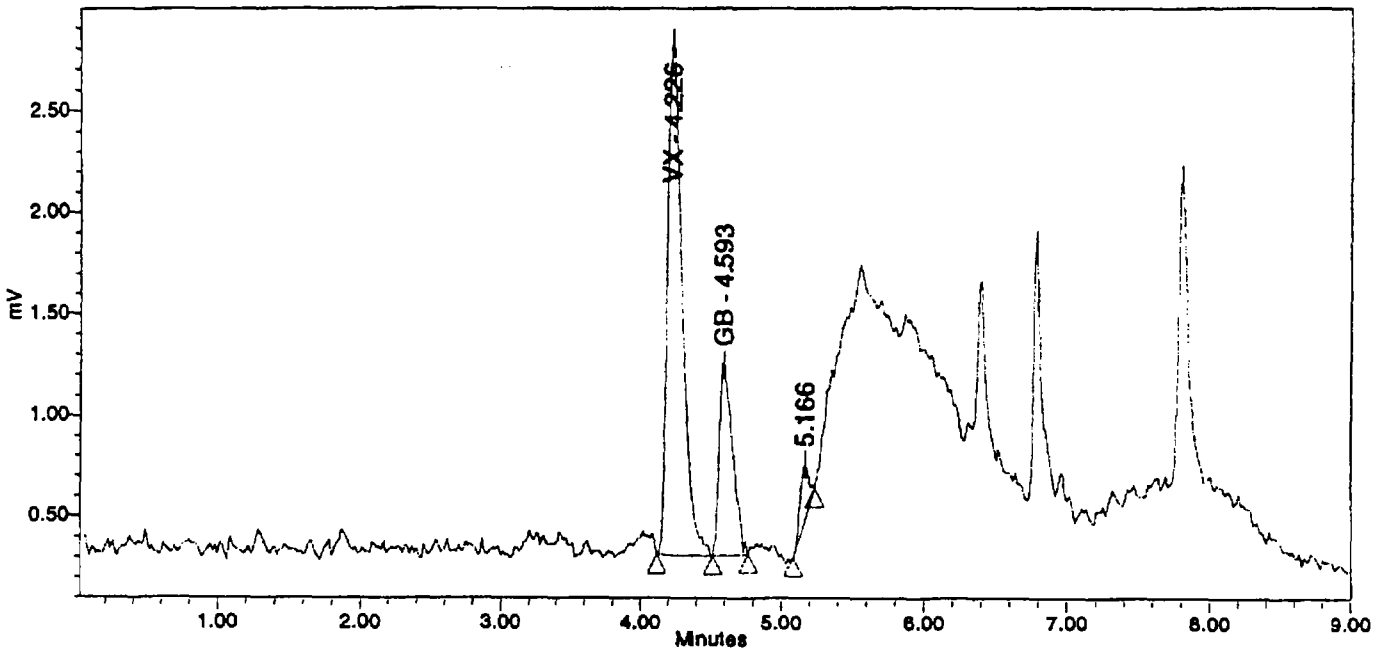
Project Name: MiscAgentwork

User Name: bwilliamson

Processing Method: Fisions_FPD_VX_GB

Date Processed: 11/13/02 2:25:23 PM

Auto-Scaled Chromatogram



Reviewed by: _____

Date: _____

Peak Results

	Name	RT	Area	Height	Amount	Units
1	VX	4.226	18256	2591	0.311	ng
2	GB	4.593	6264	961	0.055	ng
3		5.166	1176	272		

Southwest Research Institute

Agent Analysis

Sample ID: Tray 153 R2

Sample Type: Unknown

Acq Method Set: FISON_VX_MSET

Date Acquired: 11/13/02 2:26:42 PM

Report Method Name: cmp_report

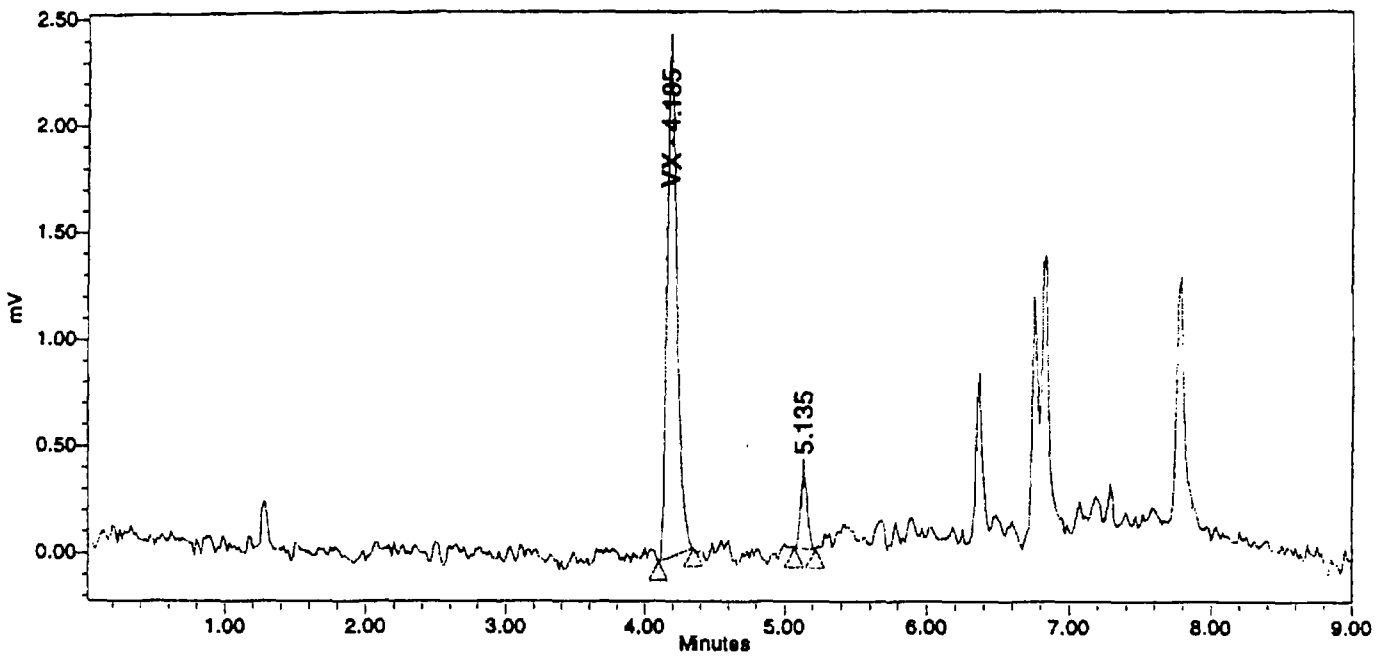
Project Name: MiscAgentwork

User Name: bwilliamson

Processing Method: Fisions_FPD_VX_GB

Date Processed: 11/13/02 2:35:58 PM

Auto-Scaled Chromatogram



Reviewed by: _____

Date: _____

Peak Results

	Name	RT	Area	Height	Amount	Units
1	VX	4.185	11825	2389	0.200	ng
2	GB	4.620				
3		5.135	1239	347		

Southwest Research Institute

Agent Analysis

Sample ID: Tray 153 R3

Sample Type: Unknown

Acq Method Set: FISON_VX_MSET

Date Acquired: 11/13/02 2:37:41 PM

Report Method Name: cmp_report

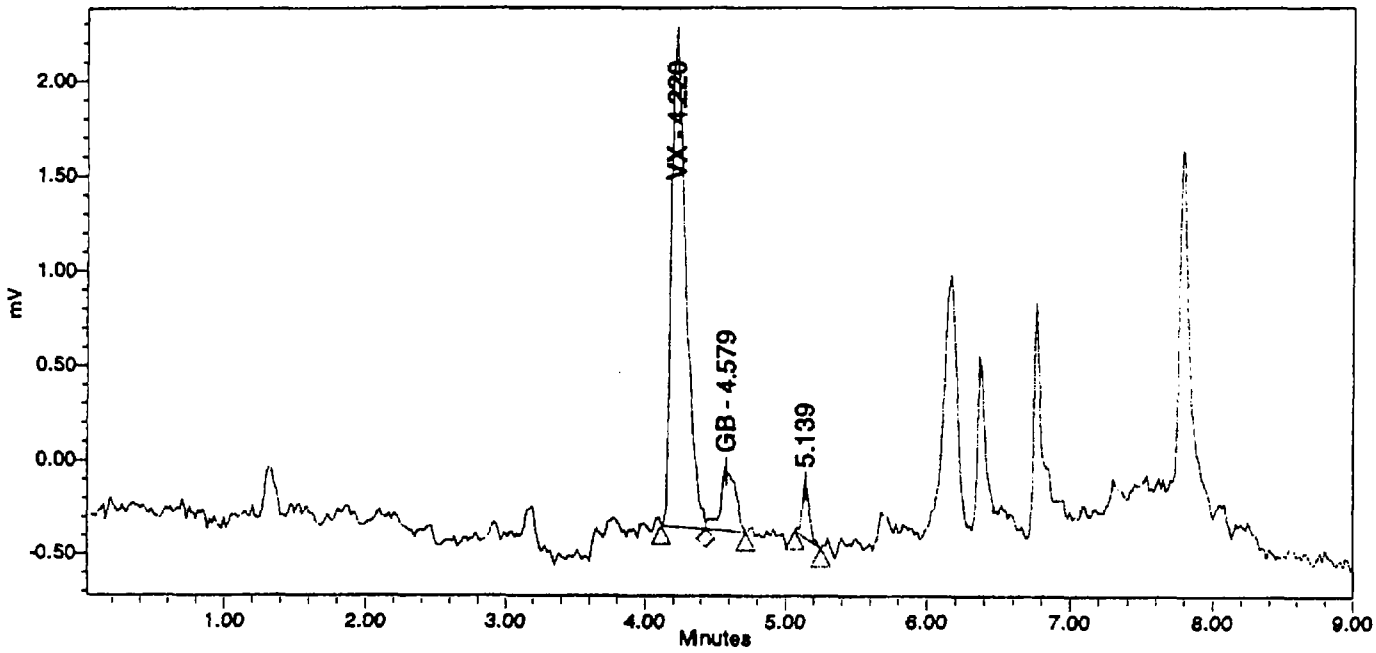
Project Name: MiscAgentwork

User Name: bwilliamson

Processing Method: Fisions_FPD_VX_GB

Date Processed: 11/13/02 2:46:56 PM

Auto-Scaled Chromatogram



Reviewed by: _____

Date: _____

Peak Results

	Name	RT	Area	Height	Amount	Units
1	VX	4.220	18641	2583	0.318	ng
2	GB	4.579	2515	336	0.010	ng
3		5.139	1074	274		

Southwest Research Institute

Agent Analysis

Sample ID: Tray 153 MS 80X

Sample Type: Unknown

Acq Method Set: FISON_VX_MSET

Date Acquired: 11/13/02 2:48:23 PM

Report Method Name: cmp_report

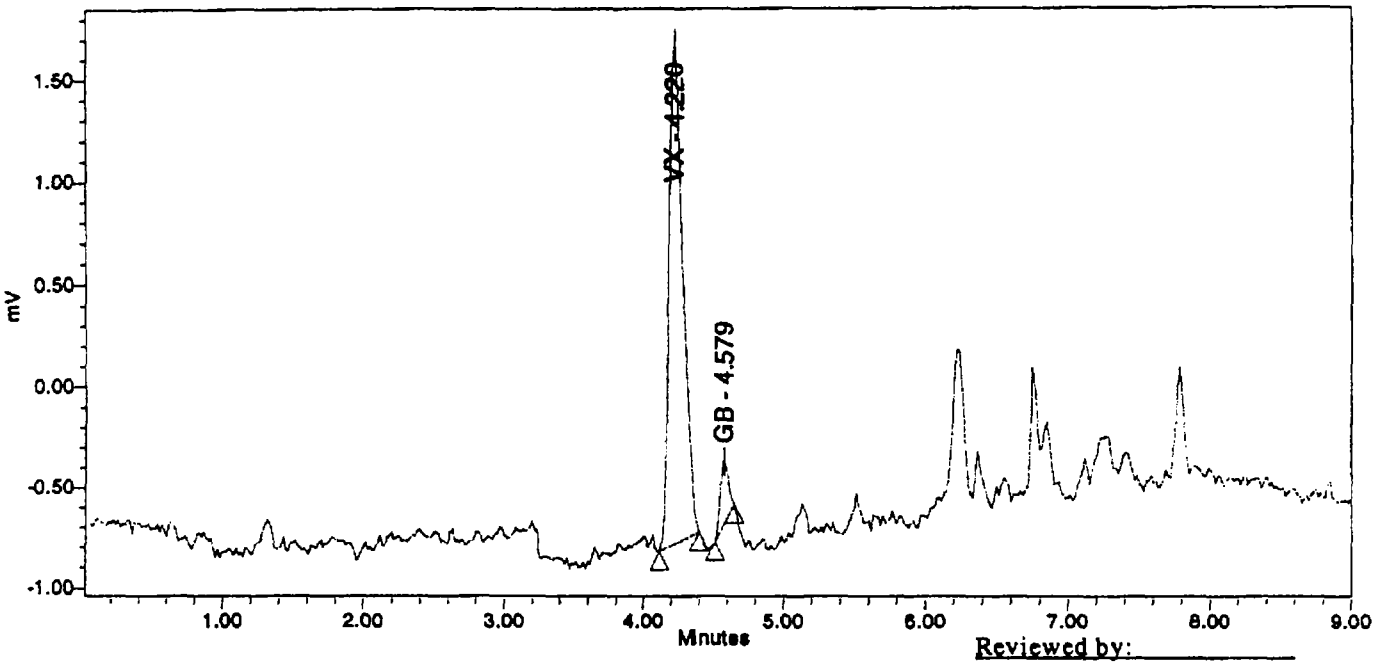
Project Name: MiscAgentwork

User Name: bwilliamson

Processing Method: Fisions_FPD_VX_GB

Date Processed: 11/13/02 2:57:37 PM

Auto-Scaled Chromatogram



Reviewed by: _____

Date: _____

Peak Results

	Name	RT	Area	Height	Amount	Units
1	VX	4.220	17093	2491	0.291	ng
2	GB	4.579	1306	315		ng

Southwest Research Institute

Agent Analysis

Sample ID: Tray 153 MSD 80X

Sample Type: Unknown

Acq Method Set: FISON_VX_MSET

Date Acquired: 11/13/02 2:58:58 PM

Report Method Name: cmp_report

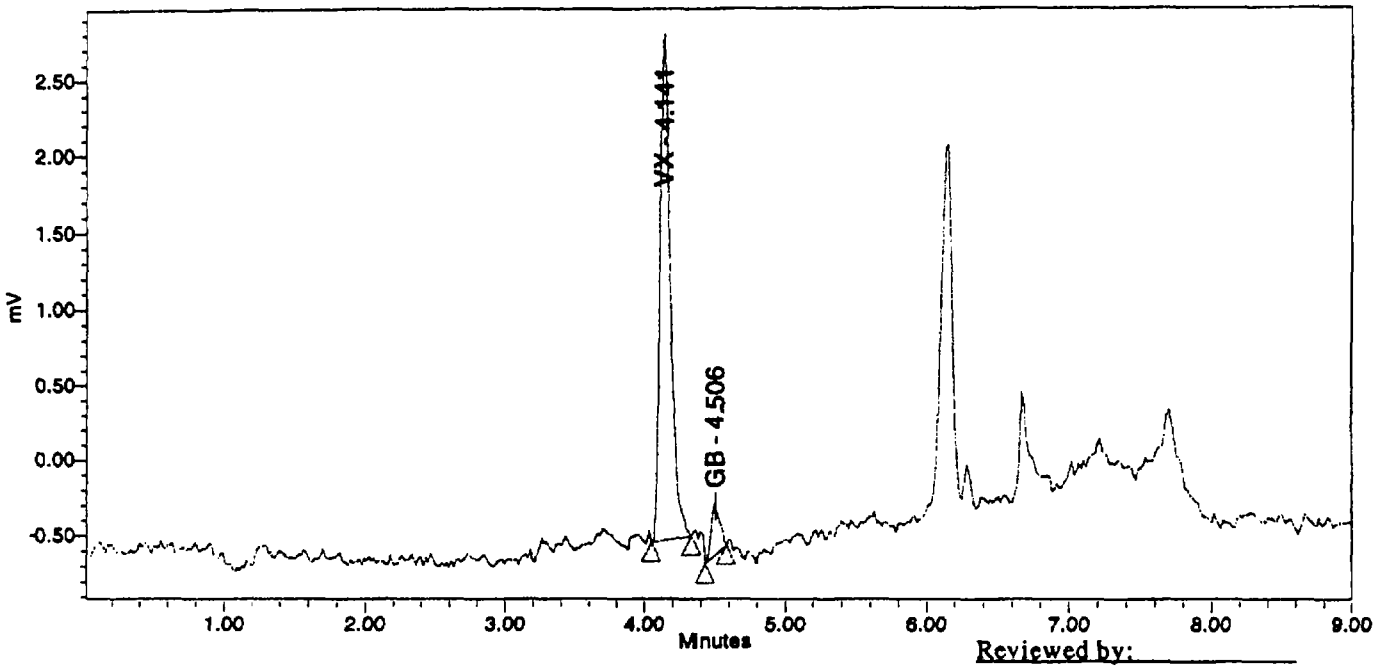
Project Name: MiscAgentwork

User Name: bwilliamson

Processing Method: Fisions_FPD_VX_GB

Date Processed: 11/13/02 3:08:13 PM

Auto-Scaled Chromatogram



Reviewed by: _____

Date: _____

Peak Results

	Name	RT	Area	Height	Amount	Units
1	VX	4.141	17339	3318	0.295	ng
2	GB	4.506	1424	322		ng

APPENDIX B

GC/MSD DATA

Acetone

	RT	on column ng/uL	FV mL	Sample wt g	Found Mass ug/g	
ASH R1						
	139	0.4858	7	2.04	1.67	
	167	1.2916	7	2.04	4.43	
	224	61.0472	7	2.04	209.48	
	252	0.4662	7	2.04	1.60	
						avg
ASH R2						
	139	0.4601	7	2.02	1.59	1.79
	167	1.0061	7	2.02	3.49	4.20
	224	52.9553	7	2.02	183.51	206.75
	252	0.5114	7	2.02	1.77	1.70
ASH R3						
	139	0.6034	7	2.01	2.10	
	167	1.3435	7	2.01	4.68	
	224	65.2543	7	2.01	227.25	
	252	0.4946	7	2.01	1.72	
						%rec
ASH MS						
	139	4.8977	7	2.01	17.06	140%
	167	6.3443	7	2.01	22.09	163%
	224	77.0947	7	2.01	268.49	564%
	252	5.4102	7	2.01	18.84	157%
						%rec
ASH MSD						
	139	4.4856	7	1.98	15.86	127%
	167	5.3803	7	1.98	19.02	133%
	224	62.3583	7	1.98	220.46	123%
	252	4.7278	7	1.98	16.71	135%

Southwest Research Institute
Semivolatile GC/MS Injection Log

OPERATOR: WSW SEQUENCE DATE: 12-Nov-2002 INSTRUMENT: Y-MD800
COLUMN: DB-1701 60M 0.32mm ID 1.0um FILM
CARRIER GAS: HELIUM HD PRESS: 70 kPA SOLVENT: ACETONE
OVEN PROGRAM: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN
GC FILE: VX MS_FILE (SCN / SIR): VX DMV: 300

PROJECT NAME: JACADS PROJECT NUMBER: 05935.03.001
SDG: WO:23238 METHOD: Direct VX MATRIX: ASH

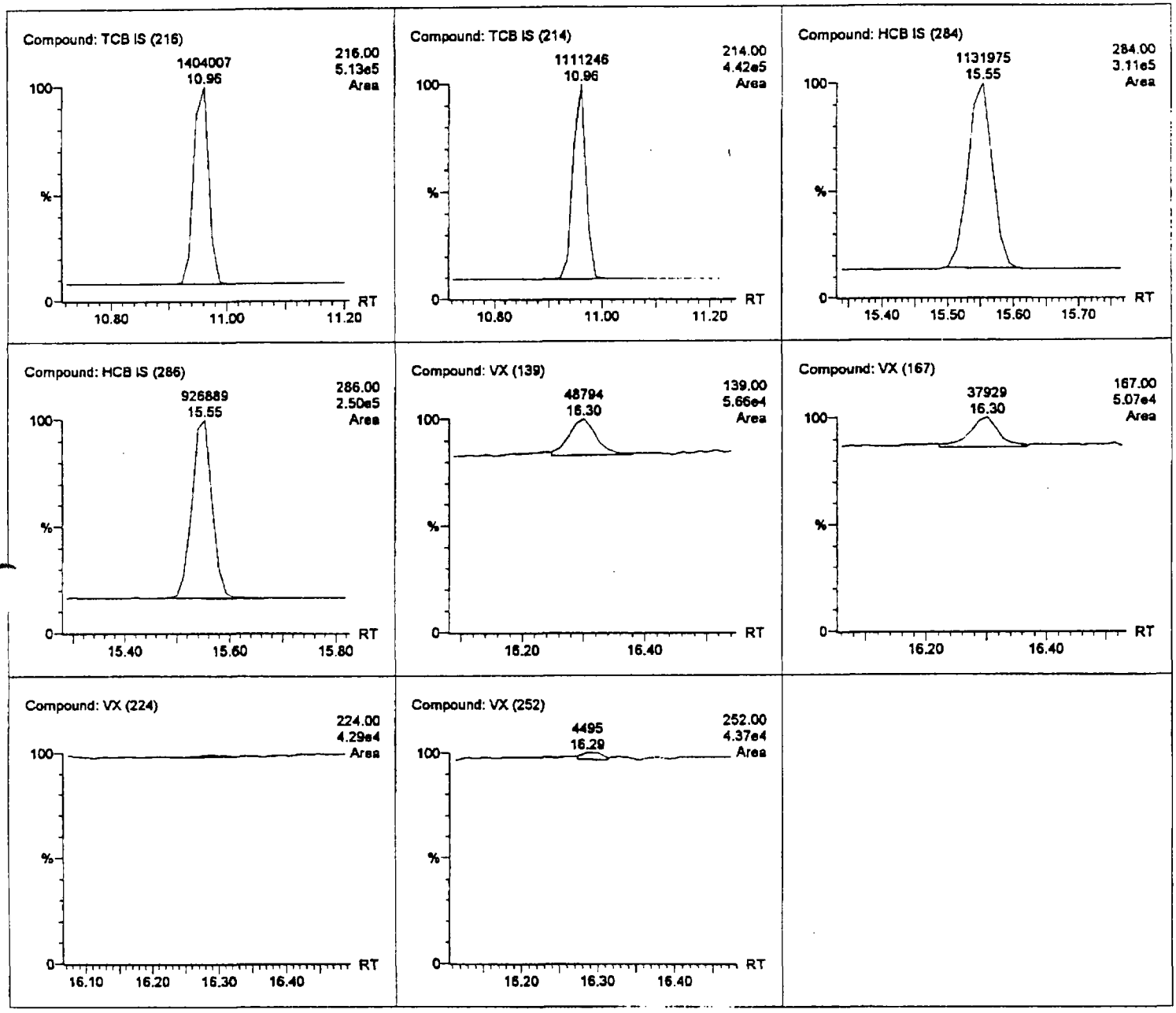
FILENAME	TIME	SAMPLE DESCRIPTION
Y1112201	17:11:27	2uL Slug
Y1112202	17:49:39	2uL STD1 VX 0.1ng/uL, IS 0.1ng/uL
Y1112203	18:24:53	2uL STD2 VX 0.2ng/uL, IS 0.1ng/uL
Y1112204	18:59:57	2uL STD3 VX 0.5ng/uL, IS 0.1ng/uL
Y1112205	19:35:05	2uL STD4 VX 1.0ng/uL, IS 0.1ng/uL
Y1112206	20:10:16	2uL STD5 VX 1.5ng/uL, IS 0.1ng/uL
Y1112207	20:45:11	2uL CCS VX 1.0ng/uL, IS 0.1ng/uL
Y1112208	21:20:10	2uL Ext BLK
Y1112209	21:55:17	2uL 213854 Tray153 ASH R1 ACE
Y1112210	22:30:00	2uL 213854 Tray153 ASH MS ACE
Y1112211	23:04:47	2uL 213854 Tray153 ASH R2 ACE
112212	23:39:34	2uL 213854 Tray153 ASH MSD ACE
Y1112213	00:14:11	2uL 213854 Tray153 ASH R3 ACE
Y1112214	00:48:47	2uL CCS VX 1.0ng/uL, IS 0.1ng/uL ACE
Y1112215	01:23:08	2uL Hexane
Y1112216	01:57:32	2uL CCS1 VX 1.0ng/uL, IS 0.1ng/uL HEX
Y1112217	02:32:11	2uL 213854 Tray153 ASH R1 HEX
Y1112218	03:06:26	2uL 213854 Tray153 ASH R2 HEX
Y1112219	03:40:53	2uL 213854 Tray153 ASH R3 HEX
Y1112220	04:15:02	2uL 213854 Tray153 ASH MS HEX
Y1112221	04:49:03	2uL 213854 Tray153 ASH MSD HEX
Y1112222	05:23:09	2uL CCS2 VX 1.0ng/uL, IS 0.1ng/uL HEX

Southwest Research Institute
Agent (VX) Quantitation Report

3

Iename: C:\MASSLAB\DATA\Y1112202.RAW
Sample ID: 2uL STD1 VX 0.1ng/uL, IS 0.1ng/uL
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 12-Nov-2002 at 17:49:39
Instrument: Y-MD800



Peak List Information - Y1112202

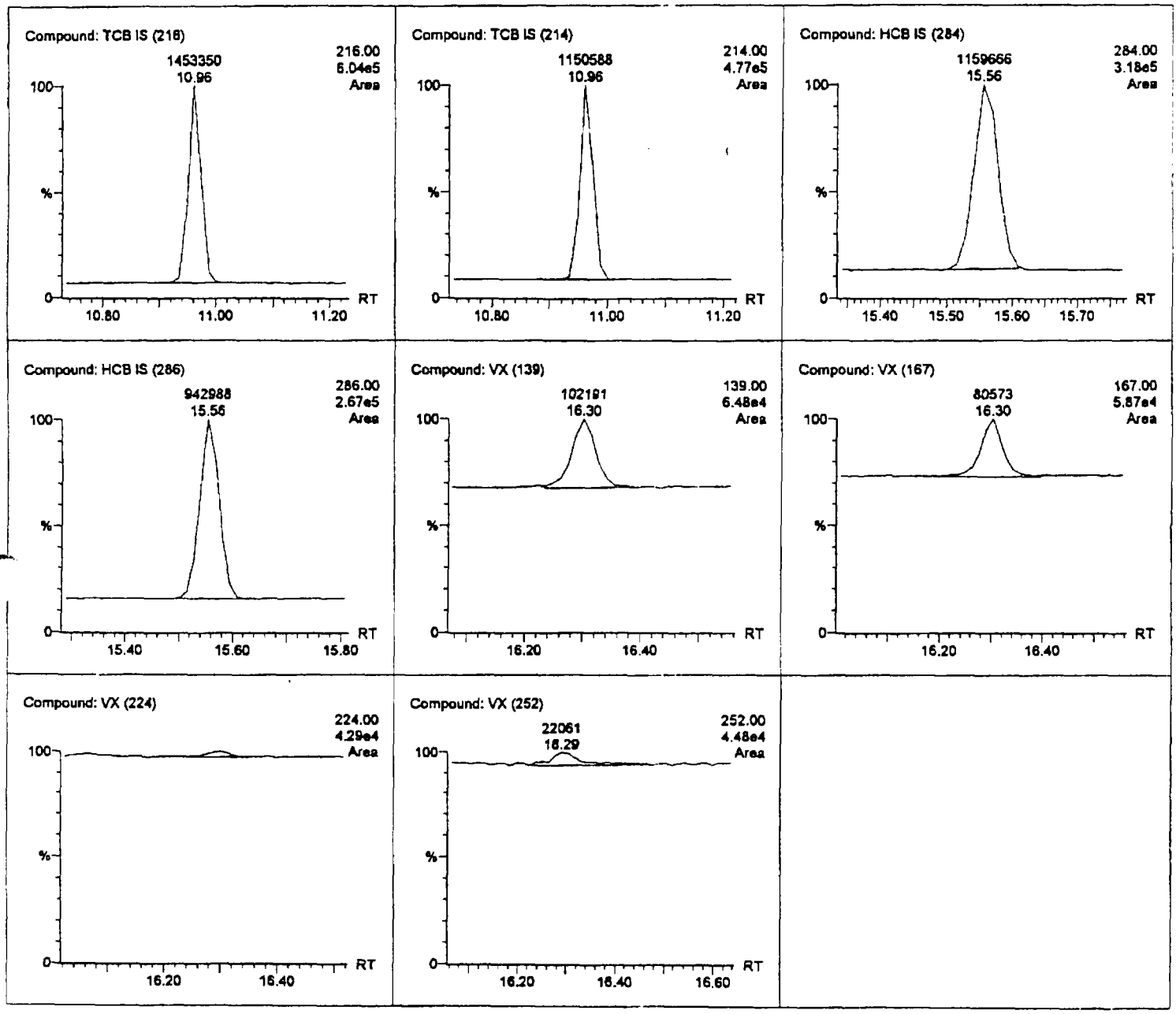
Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.962	IS	1404007	470992	0.1000
2	TCB IS (214)	10.962	IS	1111246	401277	0.1000
3	HCB IS (284)	15.553	IS	1131975	267873	0.1000
4	HCB IS (286)	15.553	IS	926889	209264	0.1000
5	VX (139)	16.301		48794	9456	0.0796
6	VX (167)	16.301		37929	6923	0.0761
	VX (224)	16.288		1795	462	0.0534
	VX (252)	16.288		4495	1449	0.0487

Southwest Research Institute
Agent (VX) Quantitation Report

4

Filename: C:\MASSLAB\DATA\Y1112203.RAW
Sample ID: 2uL STD2 VX 0.2ng/uL, IS 0.1ng/uL
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 12-Nov-2002 at 18:24:53
Instrument: Y-MD800



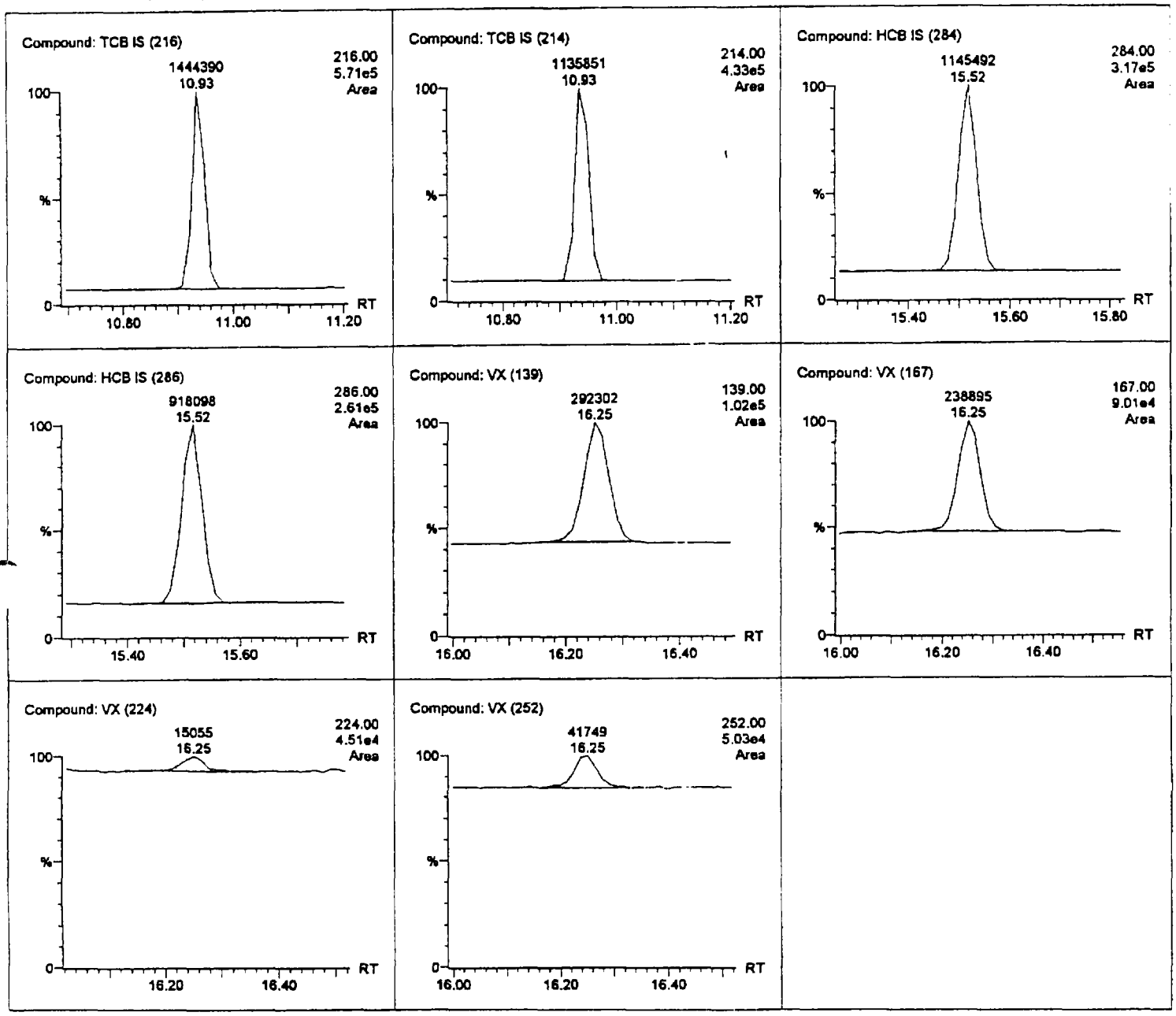
Peak List Information - Y1112203

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.961	IS	1453350	561489	0.1000
2	TCB IS (214)	10.961	IS	1150588	435803	0.1000
3	HCB IS (284)	15.556	IS	1159666	275175	0.1000
4	HCB IS (286)	15.556	IS	942988	225935	0.1000
5	VX (139)	16.303		102191	20938	0.1628
6	VX (167)	16.303		80573	15890	0.1578
	VX (224)	16.290		6193	1088	0.1798
	VX (252)	16.290		22061	2789	0.2331

Southwest Research Institute
Agent (VX) Quantitation Report

.ename: C:\MASSLAB\DATA\Y1112204.RAW
Sample ID: 2uL STD3 VX 0.5ng/uL, IS 0.1ng/uL
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 12-Nov-2002 at 18:59:57
Instrument: Y-MD800



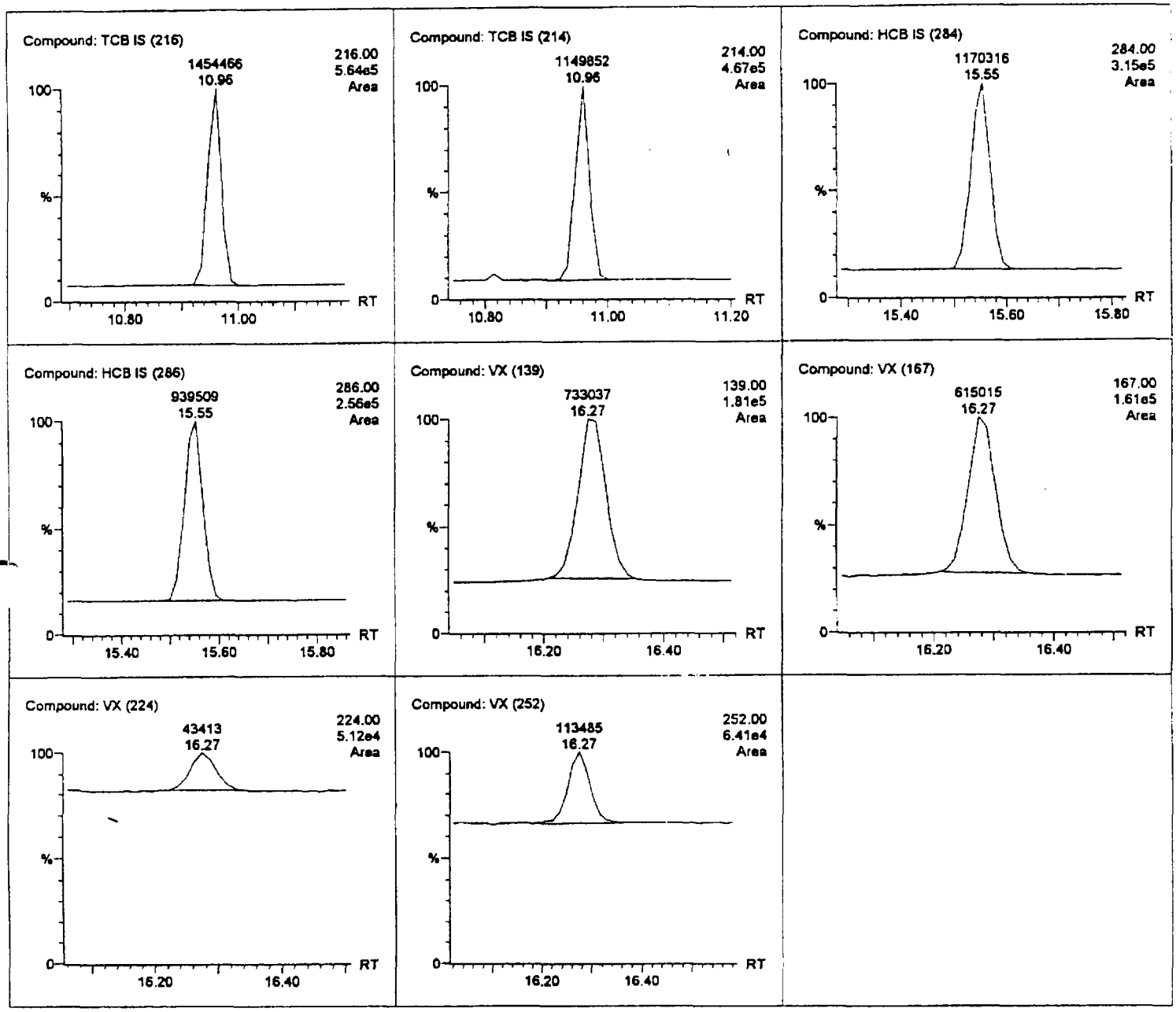
Peak List Information - Y1112204

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.935	IS	1444390	529275	0.1000
2	TCB IS (214)	10.935	IS	1135851	391386	0.1000
3	HCB IS (284)	15.517	IS	1145492	275177	0.1000
4	HCB IS (286)	15.517	IS	918098	219442	0.1000
5	VX (139)	16.251		292302	57673	0.4714
6	VX (167)	16.251		238895	47008	0.4735
	VX (224)	16.251		15055	3234	0.4424
	VX (252)	16.251		41749	7872	0.4467

lename: C:\MASSLAB\DATA\Y1112205.RAW
Sample ID: 2uL STD4 VX 1.0ng/uL, IS 0.1ng/uL
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 12-Nov-2002 at 19:35:05

Instrument: Y-MD800



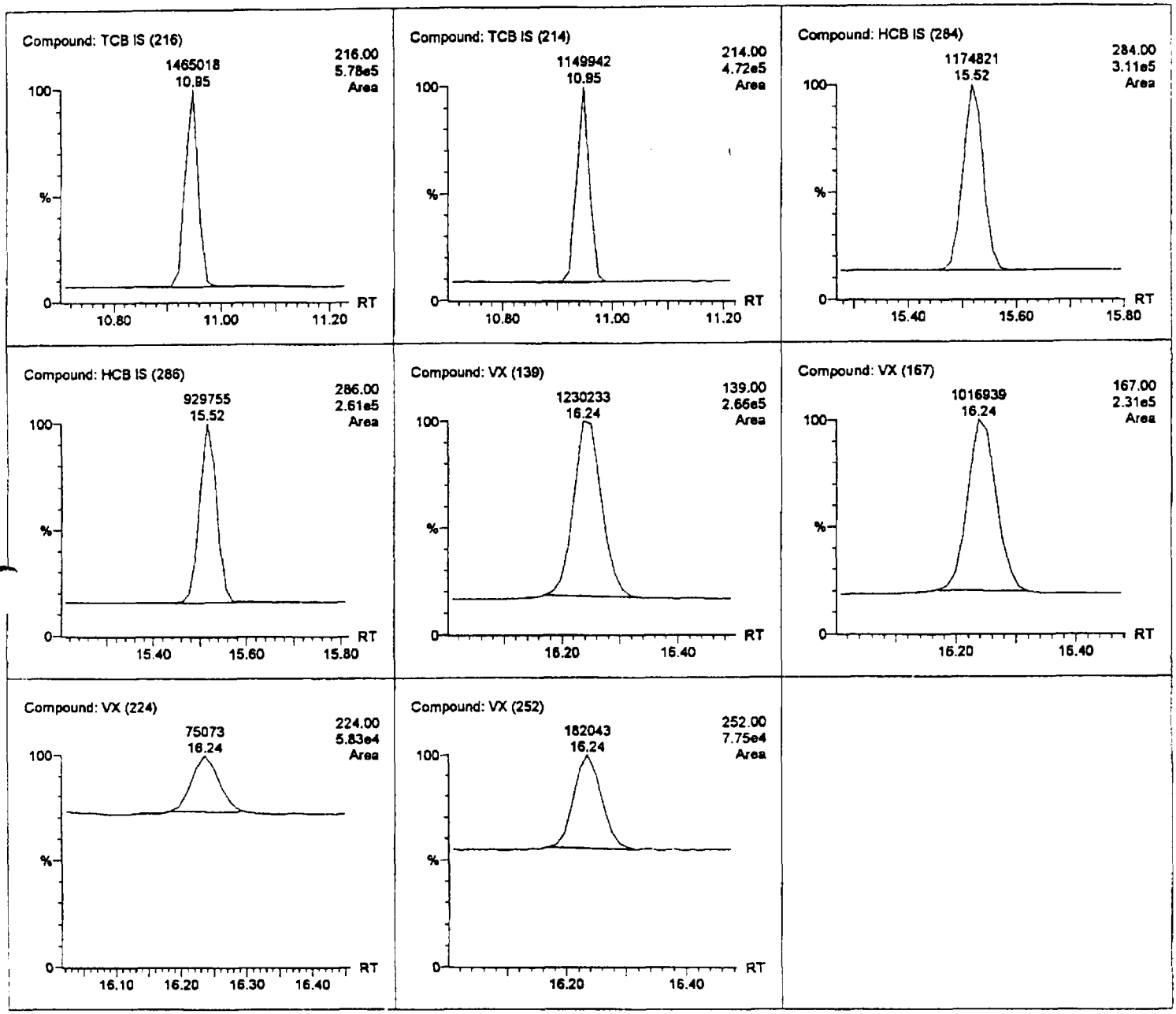
Peak List Information - Y1112205

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.961	IS	1454466	522210	0.1000
2	TCB IS (214)	10.961	IS	1149852	425590	0.1000
3	HCB IS (284)	15.554	IS	1170316	273480	0.1000
4	HCB IS (286)	15.554	IS	939509	214423	0.1000
5	VX (139)	16.275		733037	134367	1.1571
6	VX (167)	16.275		615015	116431	1.1932
	VX (224)	16.275		43413	9118	1.2487
	VX (252)	16.275		113485	21809	1.1884

Southwest Research Institute
Agent (VX) Quantitation Report

Filename: C:\MASSLAB\DATA\Y1112206.RAW
Sample ID: 2uL STD5 VX 1.5ng/uL, IS 0.1ng/uL
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 12-Nov-2002 at 20:10:16
Instrument: Y-MD800



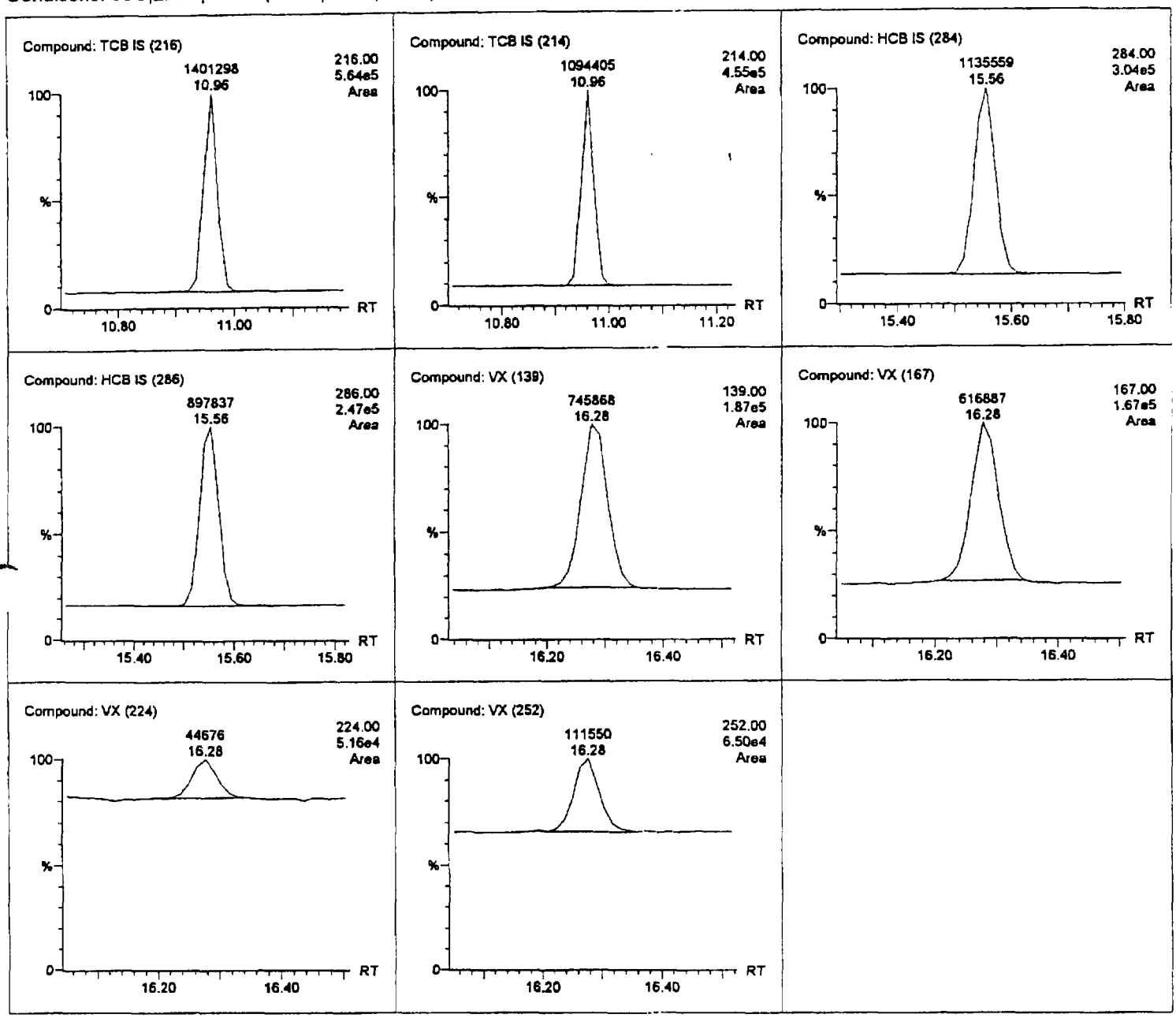
Peak List Information - Y1112206

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.948	IS	1465018	536086	0.1000
2	TCB IS (214)	10.948	IS	1149942	430110	0.1000
3	HCB IS (284)	15.516	IS	1174821	270222	0.1000
4	HCB IS (286)	15.516	IS	929755	220140	0.1000
5	VX (139)	16.237		1230233	218179	1.9346
6	VX (167)	16.237		1016939	185412	1.9654
	VX (224)	16.237		75073	15858	2.1510
	VX (252)	16.237		182043	34528	1.8990

Southwest Research Institute
Agent (VX) Quantitation Report

.ename: C:\MASSLAB\DATA\Y1112207.RAW
Sample ID: 2uL CCS VX 1.0ng/uL, IS 0.1ng/uL
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 12-Nov-2002 at 20:45:11
Instrument: Y-MD800



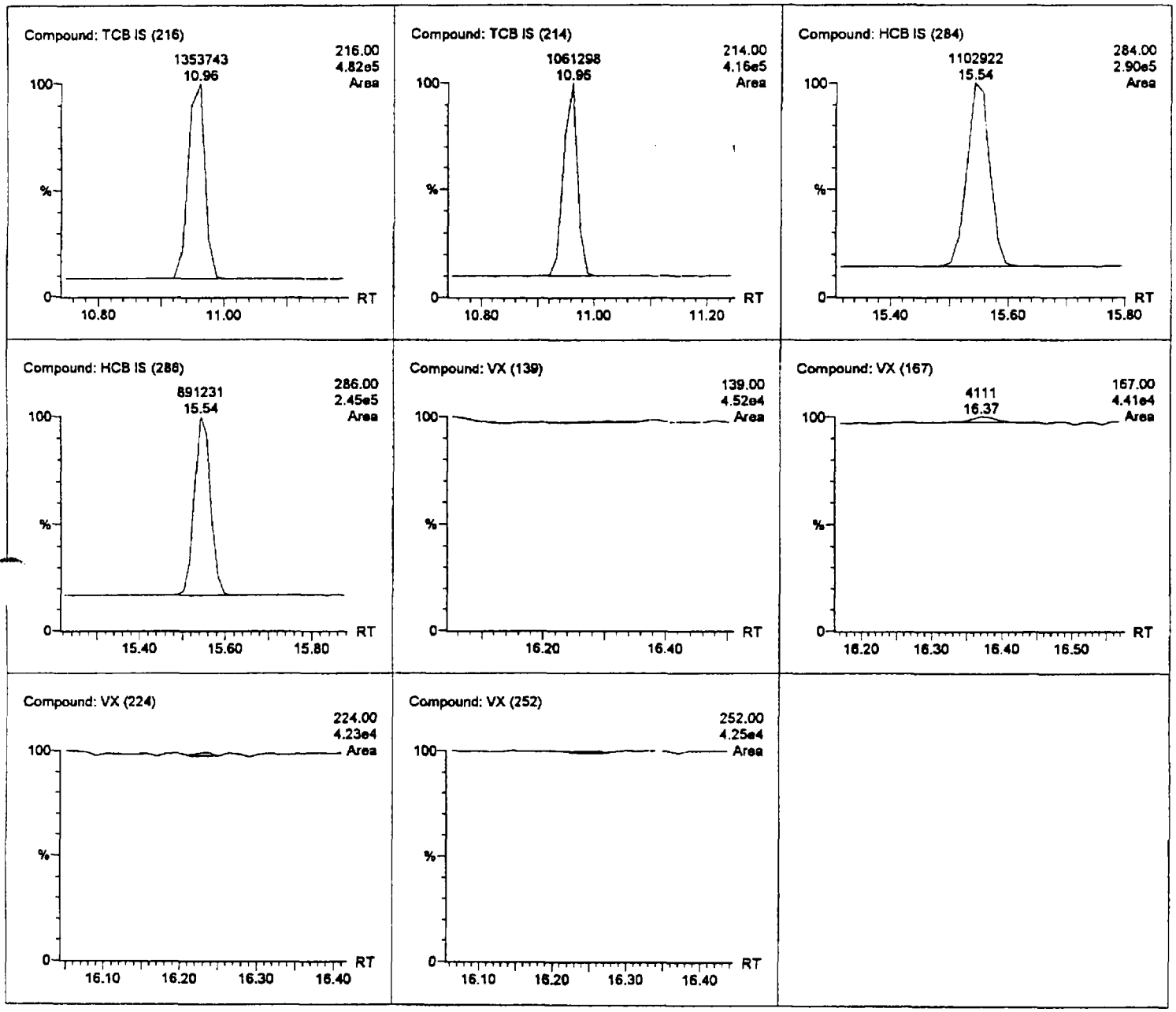
Peak List Information - Y1112207

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.961	IS	1401298	522020	0.1000
2	TCB IS (214)	10.961	IS	1094405	413165	0.1000
3	HCB IS (284)	15.555	IS	1135559	263130	0.1000
4	HCB IS (286)	15.555	IS	897837	205614	0.1000
5	VX (139)	16.277		745868	141369	1.2134
6	VX (167)	16.277		616887	122040	1.2334
	VX (224)	16.277		44676	9552	1.3243
	VX (252)	16.277		111550	22413	1.2039

Southwest Research Institute
Agent (VX) Quantitation Report

ename: C:\MASSLAB\DATA\Y1112208.RAW
Sample ID: 2uL Ext BLK
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 12-Nov-2002 at 21:20:10
Instrument: Y-MD800



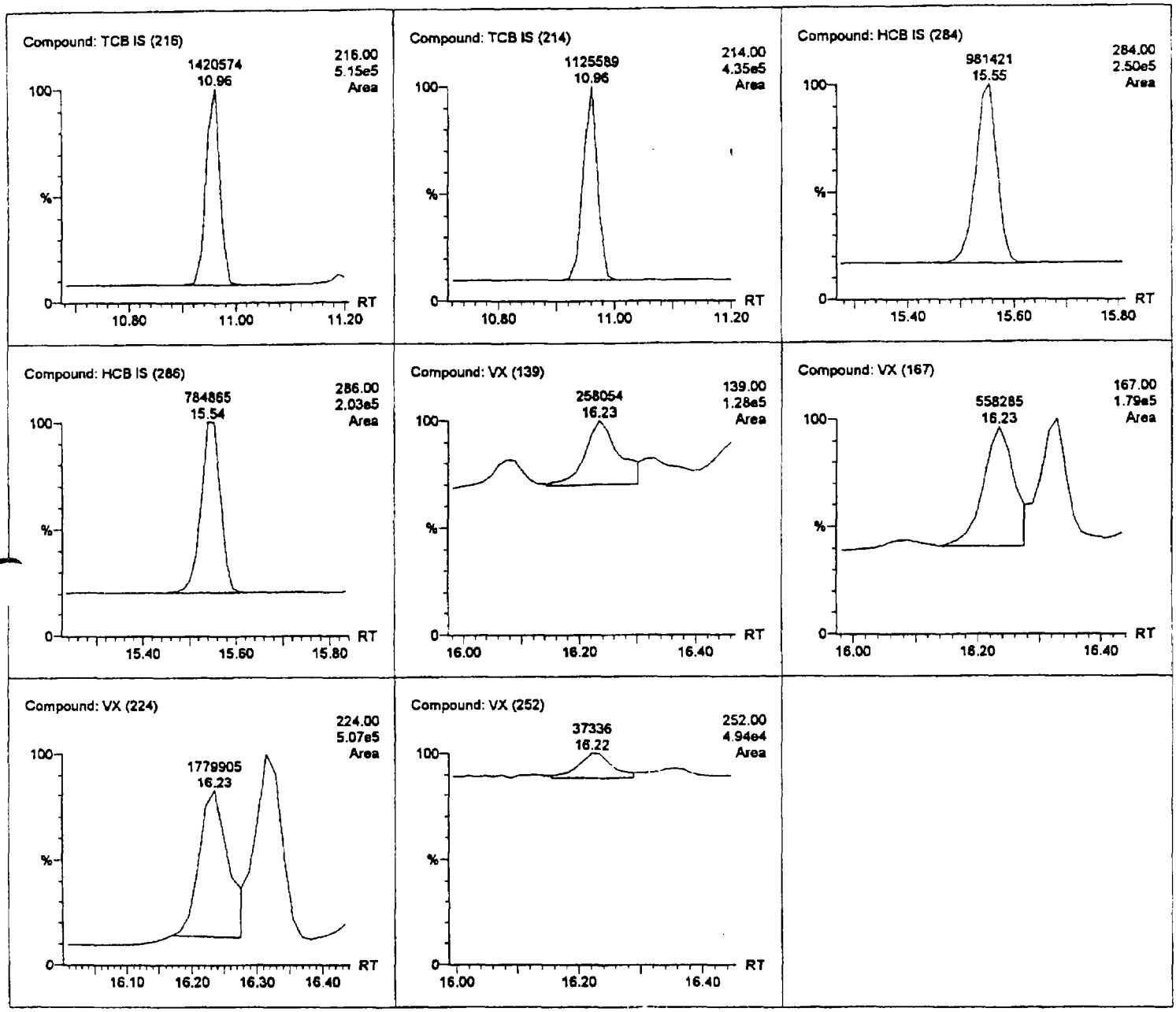
Peak List Information - Y1112208

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.962	IS	1353743	440337	0.1000
2	TCB IS (214)	10.962	IS	1061298	374030	0.1000
3	HCB IS (284)	15.543	IS	1102922	248121	0.1000
4	HCB IS (286)	15.543	IS	891231	203278	0.1000
5	VX (139)	16.304		2246	412	0.0038
6	VX (167)	16.370		4111	1121	0.0085
	VX (224)	16.237		1693	650	0.0517
	VX (252)	16.250		2242	468	0.0249

Southwest Research Institute
Agent (VX) Quantitation Report

ename: C:\MASSLAB\DATA\Y1112209.RAW
Sample ID: 2uL 213854 Tray153 ASH R1 ACE
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 12-Nov-2002 at 21:55:17
Instrument: Y-MD800



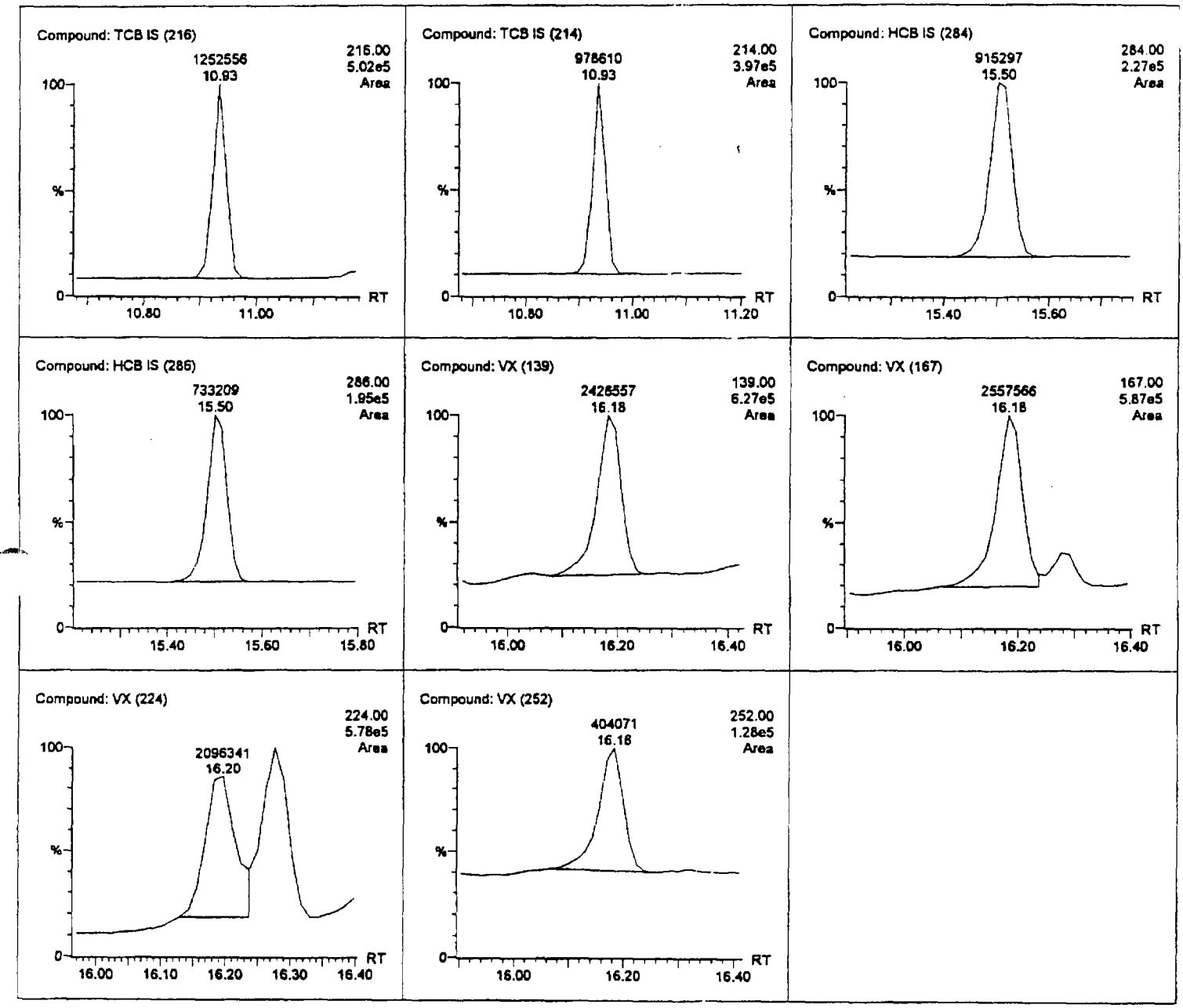
Peak List Information - Y1112209

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.961	IS	1420574	472326	0.1000
2	TCB IS (214)	10.961	IS	1125589	393872	0.1000
3	HCB IS (284)	15.554	IS	981421	207691	0.1000
4	HCB IS (286)	15.541	IS	784865	161246	0.1000
5	VX (139)	16.235		258054	38447	0.4858
6	VX (167)	16.235		558285	99838	1.2916
	VX (224)	16.235		1779905	352234	61.0472
	VX (252)	16.222		37336	5778	0.4662

Southwest Research Institute
Agent (VX) Quantitation Report

.ename: C:\MASSLAB\DATA\Y1112210.RAW
Sample ID: 2uL 213854 Tray153 ASH MS ACE
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 12-Nov-2002 at 22:30:00
Instrument: Y-MD800



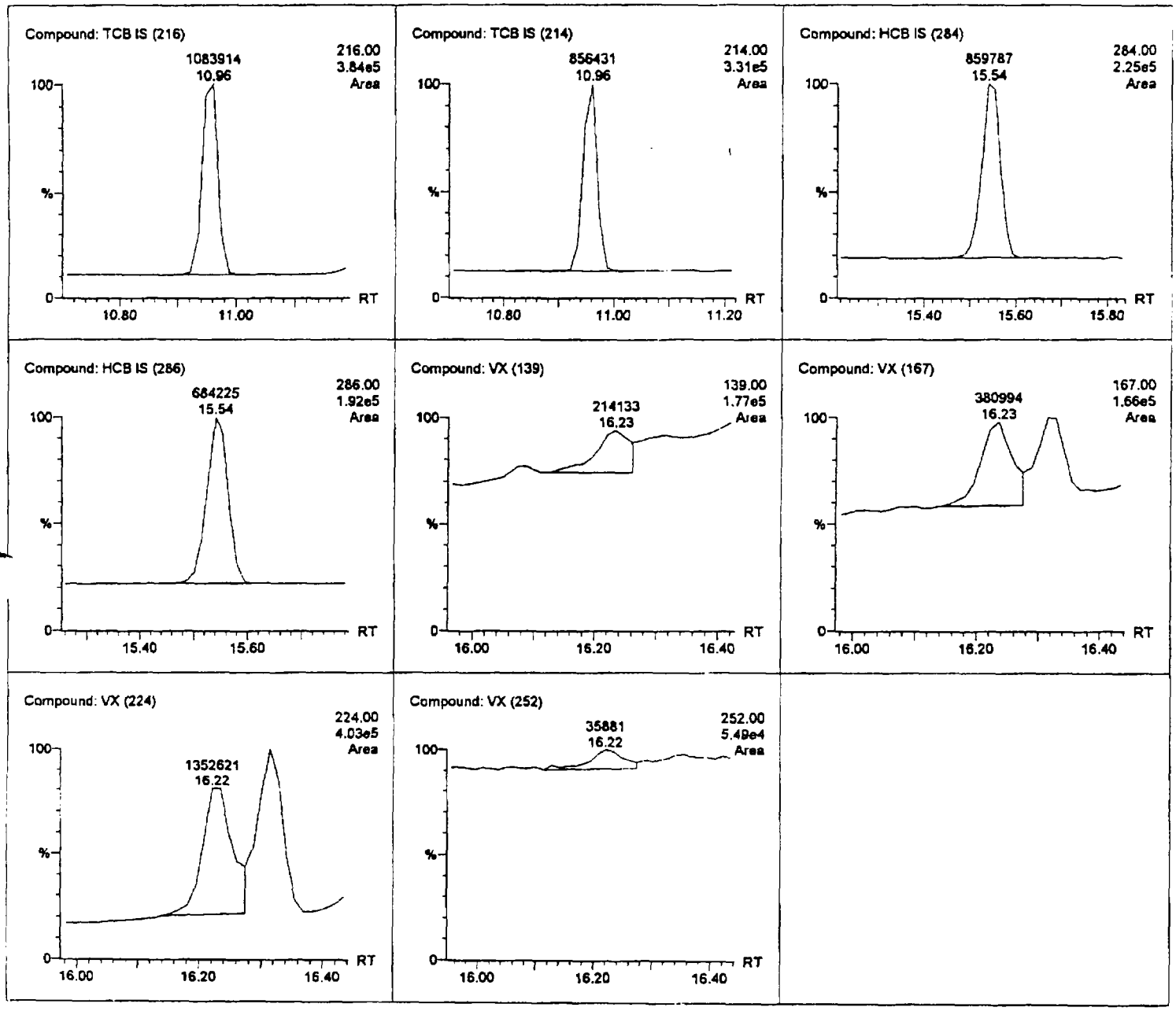
Peak List Information - Y1112210

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.935	IS	1252556	459683	0.1000
2	TCB IS (214)	10.935	IS	978610	354949	0.1000
3	HCB IS (284)	15.502	IS	915297	184352	0.1000
4	HCB IS (286)	15.502	IS	733209	152976	0.1000
5	VX (139)	16.183		2426557	469577	4.8977
6	VX (167)	16.183		2557566	471660	6.3443
	VX (224)	16.197		2096341	391659	77.0947
	VX (252)	16.183		404071	75747	5.4102

Southwest Research Institute
Agent (VX) Quantitation Report

Filename: C:\MASSLAB\DATA\Y1112211.RAW
Sample ID: 2uL 213854 Tray153 ASH R2 ACE
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 12-Nov-2002 at 23:04:47
Instrument: Y-MD800



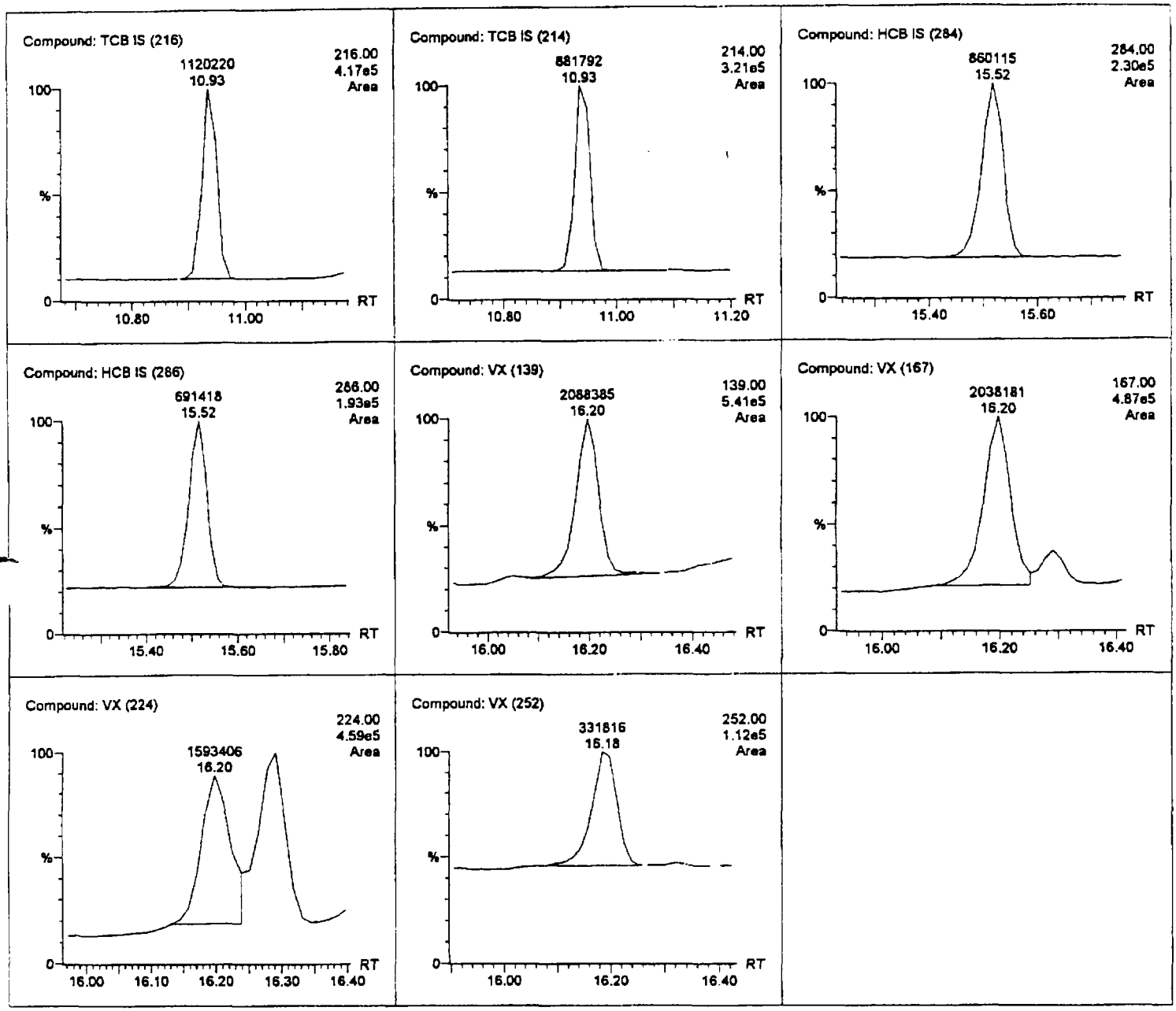
Peak List Information - Y1112211

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.961	IS	1083914	341015	0.1000
2	TCB IS (214)	10.961	IS	856431	288936	0.1000
3	HCB IS (284)	15.541	IS	859787	182722	0.1000
4	HCB IS (286)	15.541	IS	684225	150154	0.1000
5	VX (139)	16.235		214133	35162	0.4601
6	VX (167)	16.235		380994	65107	1.0061
	VX (224)	16.221		1352621	240683	52.9553
	VX (252)	16.221		35881	5055	0.5114

Southwest Research Institute
Agent (VX) Quantitation Report

.ename: C:\MASSLAB\DATA\Y1112212.RAW
Sample ID: 2uL 213854 Tray153 ASH MSD ACE
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 12-Nov-2002 at 23:39:34
Instrument: Y-MD800



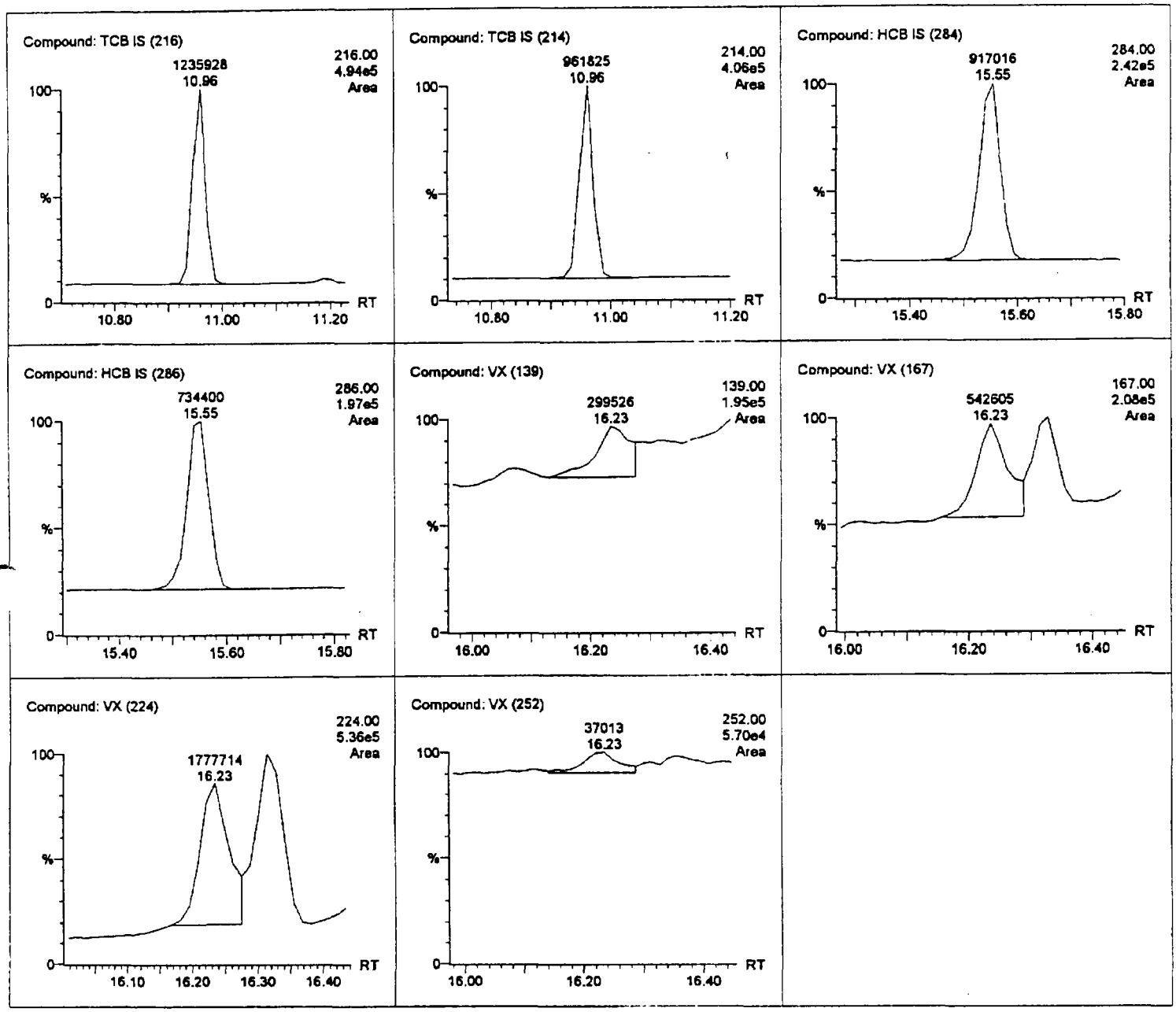
Peak List Information - Y1112212

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.935	IS	1120220	373652	0.1000
2	TCB IS (214)	10.935	IS	881792	278977	0.1000
3	HCB IS (284)	15.517	IS	860115	187203	0.1000
4	HCB IS (286)	15.517	IS	691418	150940	0.1000
5	VX (139)	16.198		2088385	399469	4.4856
6	VX (167)	16.198		2038181	384518	5.3803
	VX (224)	16.198		1593406	322266	62.3583
	VX (252)	16.185		331816	60726	4.7278

Southwest Research Institute
Agent (VX) Quantitation Report

Filename: C:\MASSLAB\DATA\Y1112213.RAW
Sample ID: 2uL 213854 Tray153 ASH R3 ACE
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 13-Nov-2002 at 00:14:11
Instrument: Y-MD800



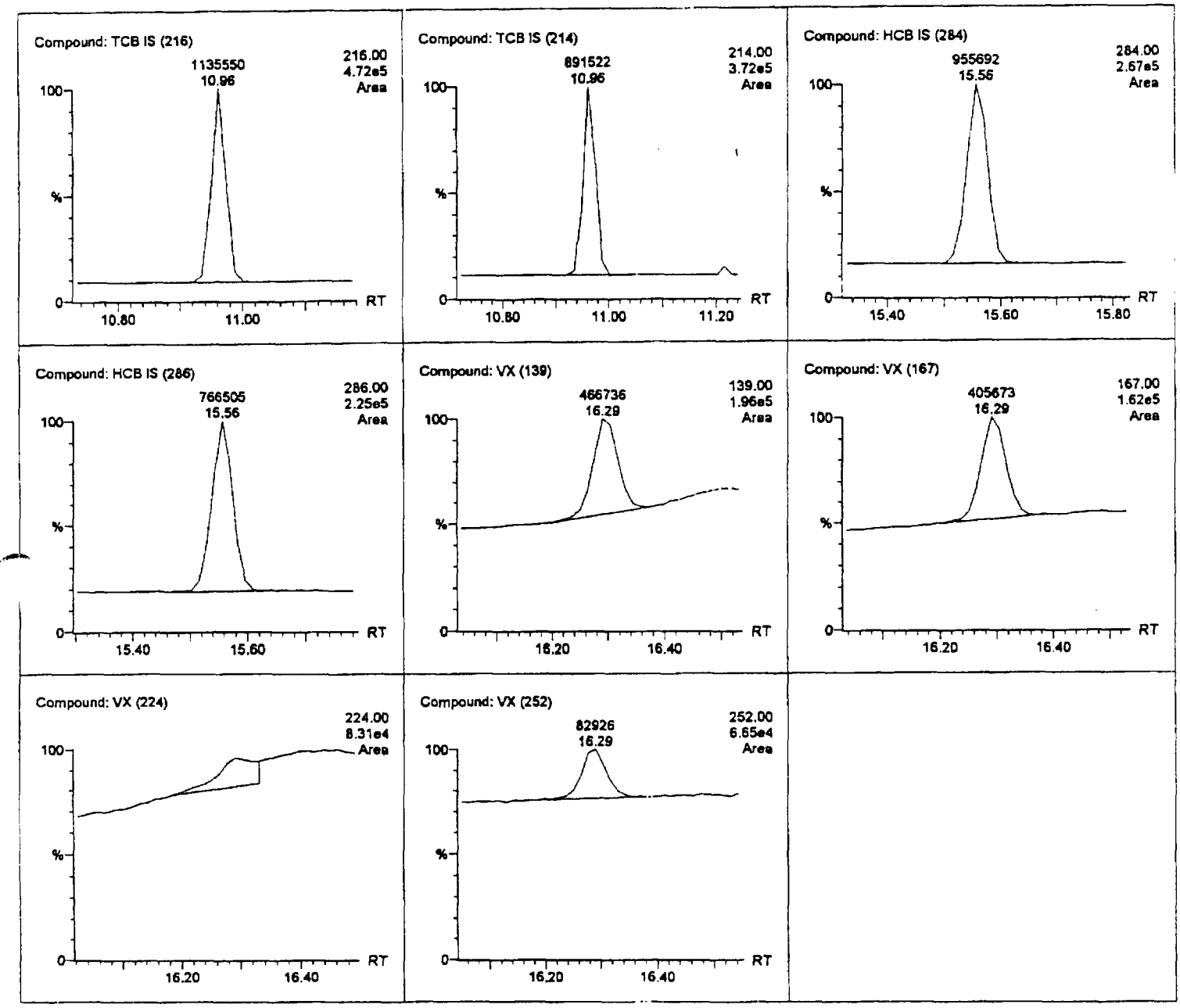
Peak List Information - Y1112213

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.961	IS	1235928	451164	0.1000
2	TCB IS (214)	10.961	IS	961825	363713	0.1000
3	HCB IS (284)	15.554	IS	917016	199277	0.1000
4	HCB IS (286)	15.554	IS	734400	154838	0.1000
5	VX (139)	16.235		299526	46401	0.6034
6	VX (167)	16.235		542605	91321	1.3435
	VX (224)	16.235		1777714	357225	65.2543
	VX (252)	16.235		37013	5441	0.4946

Southwest Research Institute
Agent (VX) Quantitation Report

ename: C:\MASSLAB\DATA\Y1112214.RAW
Sample ID: 2uL CCS VX 1.0ng/uL, IS 0.1ng/uL ACE
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 13-Nov-2002 at 00:48:47
Instrument: Y-MD800



Peak List Information - Y1112214

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.961	IS	1135550	429152	0.1000
2	TCB IS (214)	10.961	IS	891522	330122	0.1000
3	HCB IS (284)	15.556	IS	955692	224516	0.1000
4	HCB IS (286)	15.556	IS	766505	182572	0.1000
5	VX (139)	16.290		466736	89101	0.9022
6	VX (167)	16.290		405673	78731	0.9638
	VX (224)	16.290		83680	11565	2.9473
	VX (252)	16.290		82926	15818	1.0634

APPENDIX C

LC/MS DATA

Run on 11/12/02

Direct VX on Tray 153 ash (LCMS)

Sample ID	inst ng/uL	gm	Dilution Factor	FV uL	Found ng/g	Spike amt (ng/g)	% Rec
CCS1 0.0312	0.035	-	-	-	-	-	112%
Acetone Blk	-	-	4	-	-	-	-
R1	0.114	2.04	4	7000	1560.79	-	-
MS	0.109	2.01	25	7000	9504.85	11000	72%
R2	0.110	2.02	4	7000	1524.48	-	-
MSD	0.138	1.98	25	7000	12159.58	11000	96%
R3	0.114	2.01	4	7000	1585.57	-	-
CCS2 0.0312	0.035	-	-	-	-	-	113%

176 TITLE Direct VX using Acetone to extract

PROJECT NO. 05935.03.001

BOOK NO. 01-0500-002

Work continued from Page

Sequence: C:\HPCHEM\1\SEQUENCE\111202.S

Sequence Table:

Method and Injection Info Part:

Line	Location	SampleName	Method	Inj	SampleType	InjVolume
1	Vial 1	VX 0.250 ug/mL	VX_SIM	2	Sample	20
2	Vial 2	VX 0.125 ug/mL	VX_SIM	1	Sample	20
3	Vial 3	VX 0.0625 ug/mL	VX_SIM	1	Sample	20
4	Vial 4	VX 0.0312 ug/mL	VX_SIM	1	Sample	20
5	Vial 5	VX 0.0156 ug/mL	VX_SIM	1	Sample	20
6	Vial 6	VX 0.007 ug/mL	VX_SIM	1	Sample	20
7	Vial 7	WATER BLK	VX_SIM	1	Sample	20
8	Vial 8	CCS 1 0.0312	VX_SIM	1	Sample	20
9	Vial 9	ACETONE BLK 4X	VX_SIM	1	Sample	20
10	Vial 10	R1 4X	VX_SIM	1	Sample	20
11	Vial 11	R1 MS 25X	VX_SIM	1	Sample	20
12	Vial 12	R2 4X	VX_SIM	1	Sample	20
13	Vial 13	R1 MSD 25X	VX_SIM	1	Sample	20
14	Vial 14	R3 4X	VX_SIM	1	Sample	20
15	Vial 15	WATER BLK2	VX_SIM	1	Sample	20
16	Vial 16	CCS 2 0.0312	VX_SIM	1	Sample	20

SCIENTIFIC BINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA

Work continued to Page

SIGNATURE G. A. Allen

DATE 11/12/02

DISCLOSED TO AND UNDERSTOOD BY DATE WITNESS DATE

3

Calibration Table

COMBO STD

Calib. Data Modified : Thursday, January 02, 2003 2:59:54 PM

Calculate : External Standard
Based on : Peak Area

Rel. Reference Window : 15.000 %
Abs. Reference Window : 0.500 min
Rel. Non-ref. Window : 15.000 %
Abs. Non-ref. Window : 0.500 min
Use Multiplier & Dilution Factor with ISTDs
Uncalibrated Peaks : not reported
Partial Calibration : Yes, identified peaks are recalibrated
Correct All Ret. Times: No, only for identified peaks

Curve Type : Linear
Origin : Included
Weight : Equal

Recalibration Settings:
Average Response : Average all calibrations
Average Retention Time: Floating Average New 75%

Calibration Report Options :
Printout of recalibrations within a sequence:
Calibration Table after Recalibration
Normal Report after Recalibration
If the sequence is done with bracketing:
Results of first cycle (ending previous bracket)

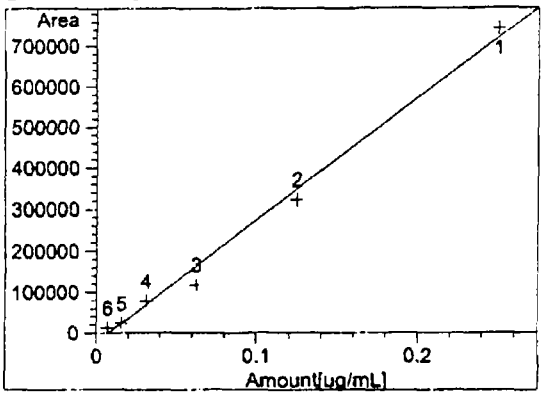
Signal 1: MSD1 268, EIC=267.7:268.7

RetTime [min]	Lvl Sig		Amount [ug/mL]	Area	Amt/Area	Ref	Grp	Name
7.052	1	6	7.00000e-3	1.24100e4	5.64060e-7			VX
		5	1.56000e-2	2.40980e4	6.47356e-7			
		4	3.12000e-2	7.90344e4	3.94765e-7			
		3	6.25000e-2	1.17879e5	5.30207e-7			
		2	1.25000e-1	3.22598e5	3.87479e-7			
		1	2.50000e-1	7.43791e5	3.36116e-7			

Peak Sum Table

No Entries in table

Calibration Curves



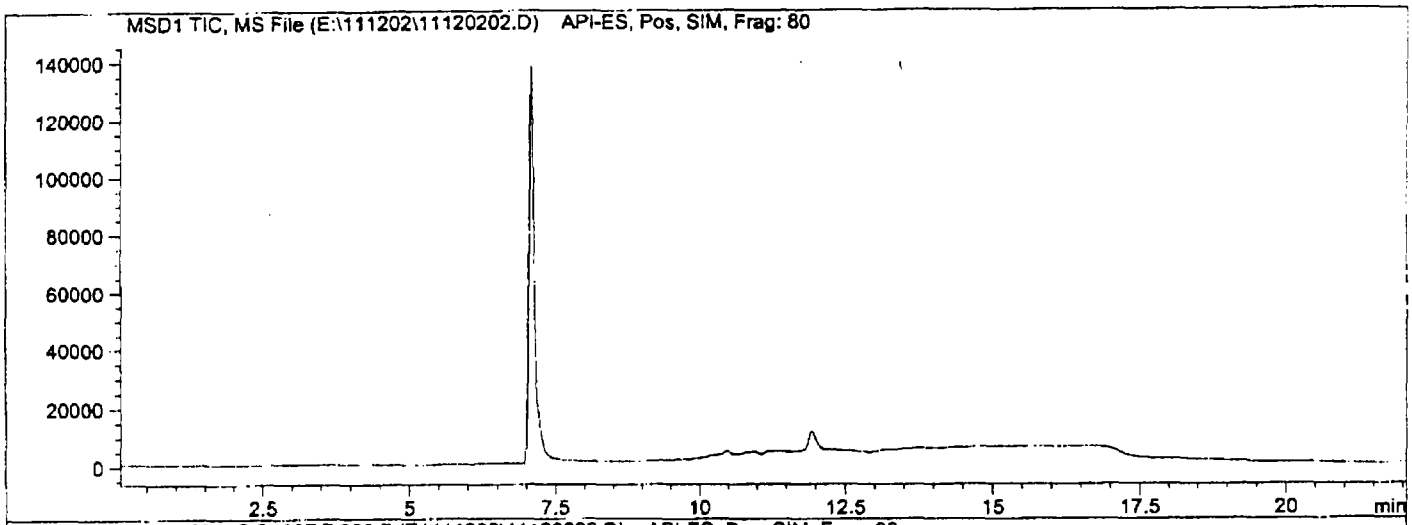
VX at exp. RT: 7.052
MSD1 268, EIC=267.7:268.7
Correlation: 0.99532
Residual Std. Dev.: 28560.19910
Formula: y = mx + b
m: 2.97739e6
b: -23282.81449
x: Amount [ug/mL]
y: Area

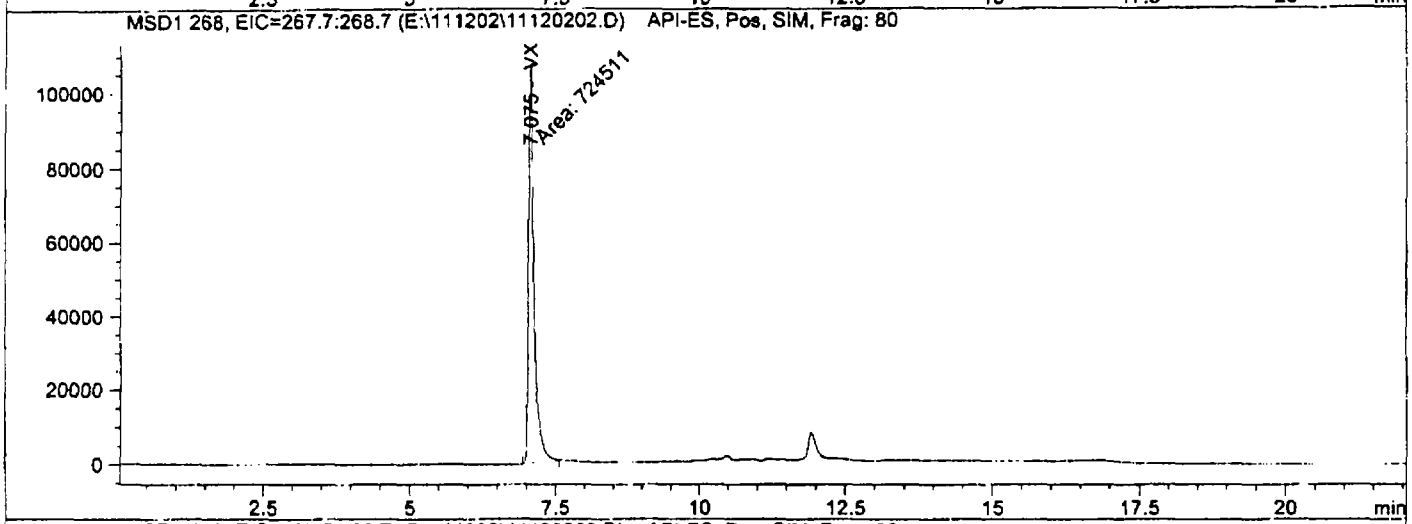
Data File E:\111202\11120202.D Sample Name: VX 0.250 ug/mL

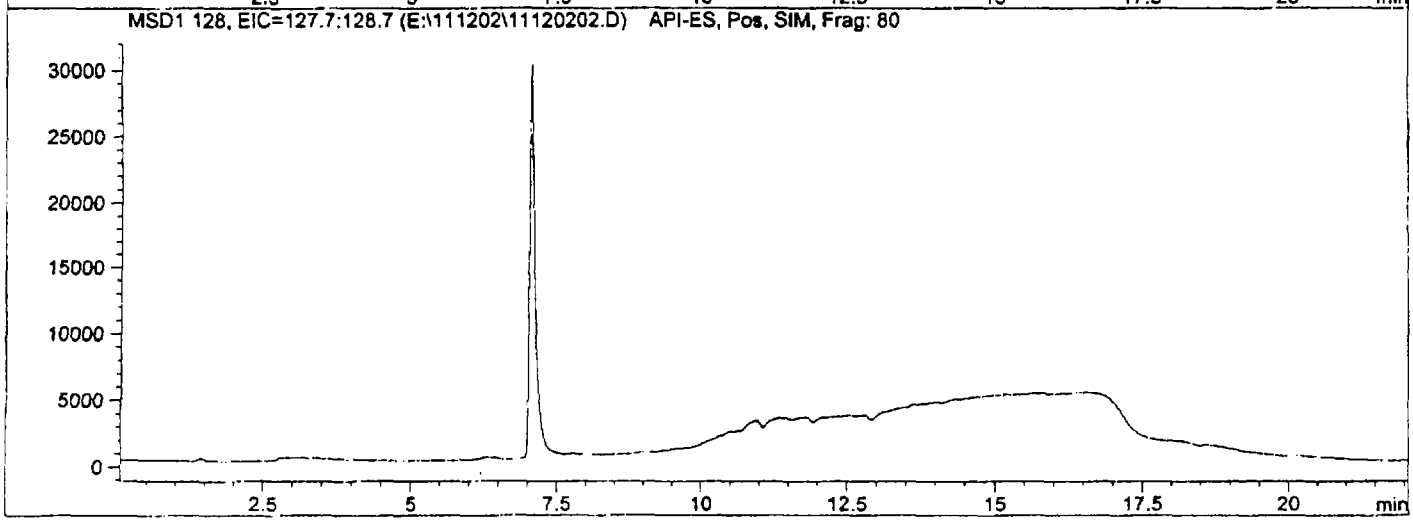
5

Injection Date	: 11/12/2002 4:51:39 PM	Seq. Line :	1
Sample Name	: VX 0.250 ug/mL	Location :	Vial 1
Acq. Operator	: GAA	Inj :	2
Acq. Instrument	: Instrument 1	Inj Volume :	20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 2:51:17 PM by AYY (modified after loading)		

ADP'S







6

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==================================================================
Injection Date  : 11/12/2002 4:51:39 PM          Seq. Line :   1
Sample Name     : VX 0.250 ug/mL                  Location : Vial 1
Acq. Operator   : GAA                                  Inj :   2
Acq. Instrument : Instrument 1                   Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 2:51:17 PM by AYY
                  (modified after loading)
ADP'S
==================================================================

==================================================================
                      External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 2:51:17 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime  Type     Area      Amt/Area    Amount   Grp   Name
 [min]                                  [ug/mL]
-------|------|----------|----------|----------|--|-----------------
  7.075 MM     7.24511e5  3.36116e-7 2.43520e-1     VX

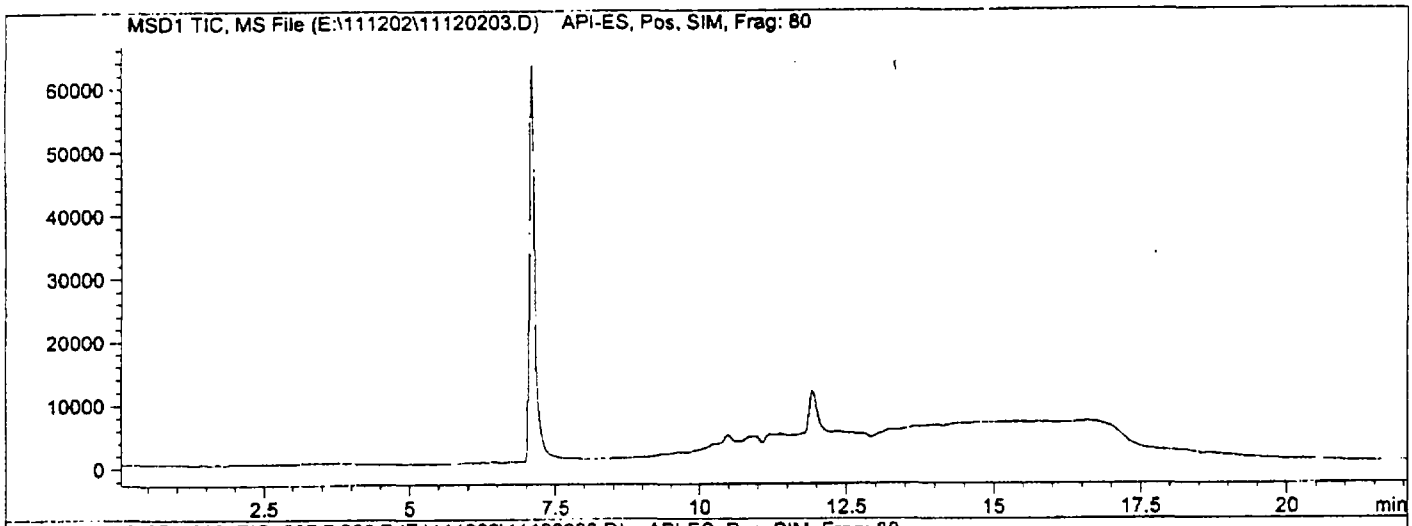
Totals :                               2.43520e-1

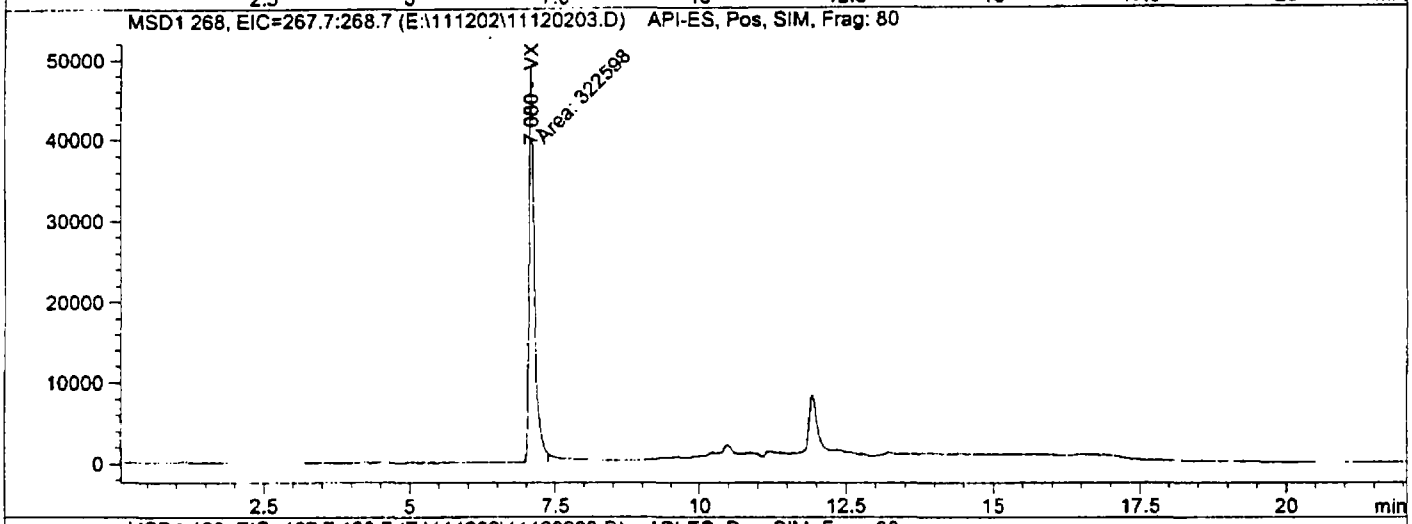

==================================================================
                        *** End of Report ***
```

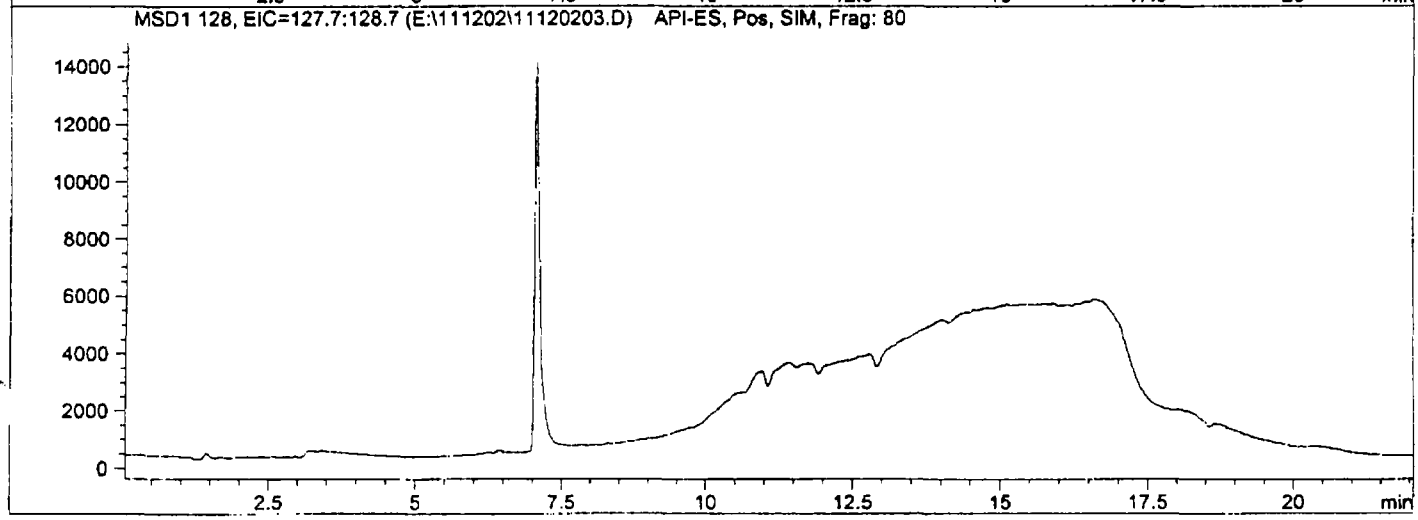
7

Injection Date	: 11/12/2002 5:15:22 PM	Seq. Line	: 2
Sample Name	: VX 0.125 ug/mL	Location	: Vial 2
Acq. Operator	: GAA	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 2:54:01 PM by AYY (modified after loading)		

ADP'S







8

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==================================================================
Injection Date  : 11/12/2002 5:15:22 PM          Seq. Line :   2
Sample Name     : VX 0.125 ug/mL                  Location : Vial 2
Acq. Operator   : GAA                                 Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 2:54:01 PM by AYY
                  (modified after loading)
ADP'S
==================================================================

==================================================================
                      External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      Thursday, January 02, 2003 2:54:01 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime  Type     Area      Amt/Area    Amount   Grp   Name
 [min]                                  [ug/mL]
-------|------|----------|----------|----------|--|-----------------
  7.080 MM     3.22598e5  3.53237e-7 1.13953e-1    VX

Totals :                               1.13953e-1

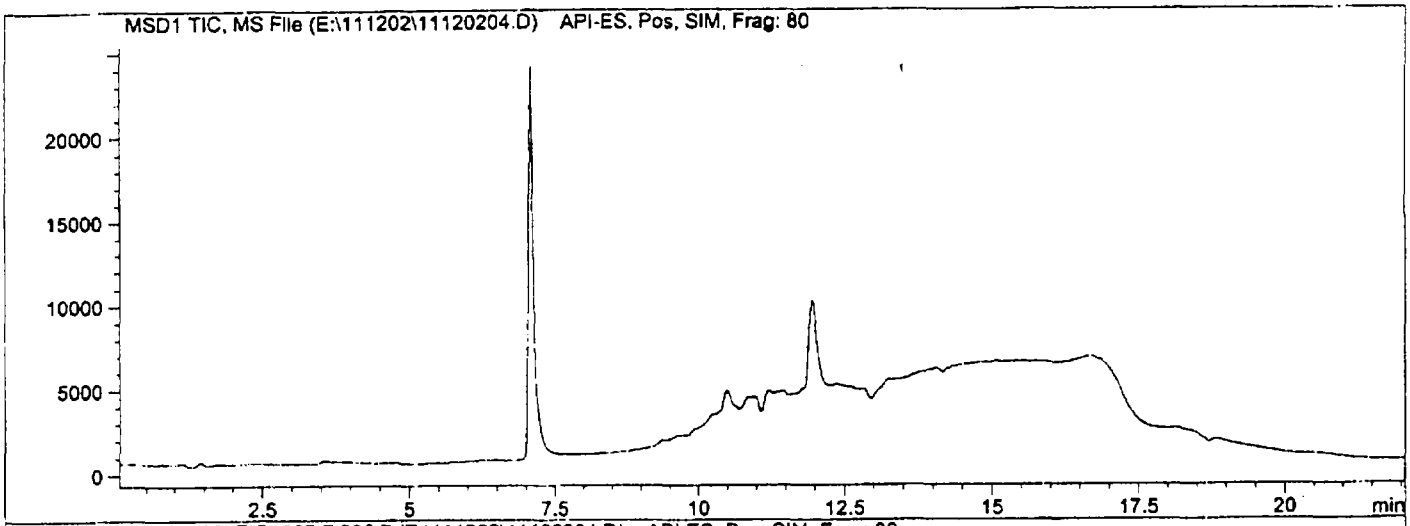

==================================================================
                        *** End of Report ***
```

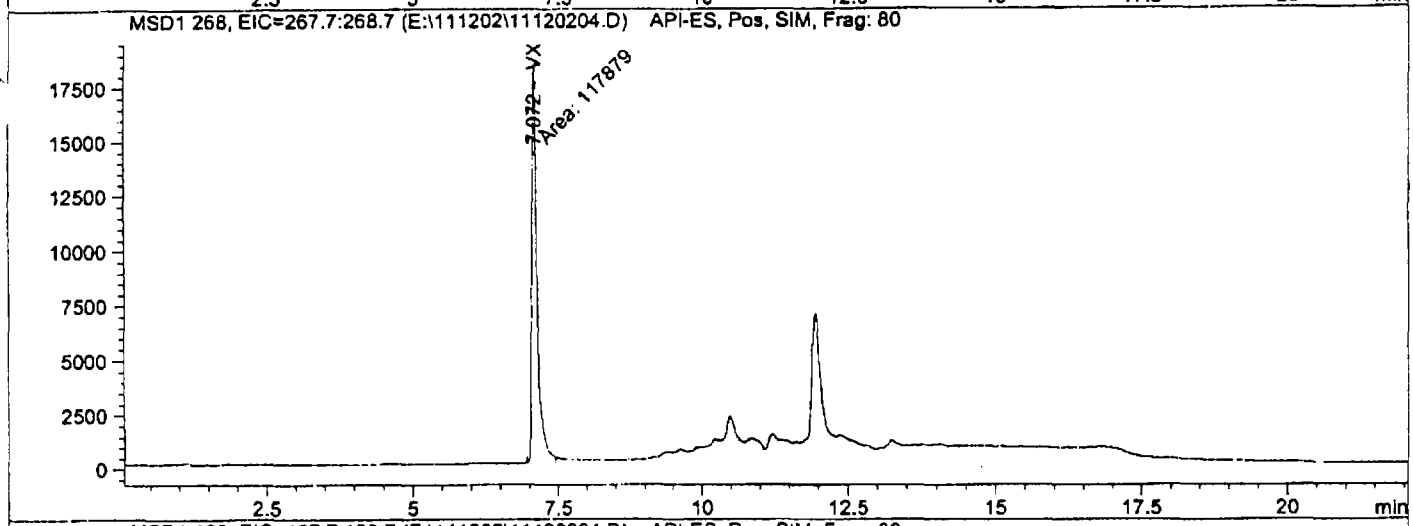
Data File E:\111202\11120204.D Sample Name: VX 0.0625 ug/mL

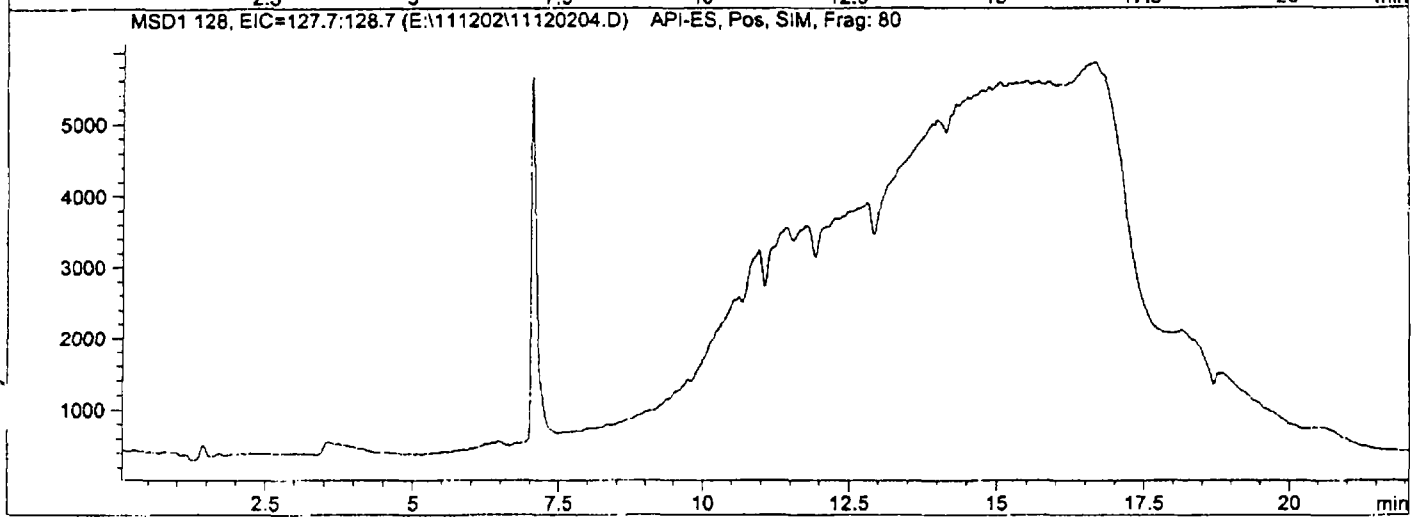
Injection Date	: 11/12/2002 5:39:06 PM	Seq. Line :	3
Sample Name	: VX 0.0625 ug/mL	Location :	Vial 3
Acq. Operator	: GAA	Inj :	1
Acq. Instrument	: Instrument 1	Inj Volume :	20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 2:54:11 PM by AYY (modified after loading)		

9

ADP'S







10

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==================================================================
Injection Date  : 11/12/2002 5:39:06 PM          Seq. Line :   3
Sample Name     : VX 0.0625 ug/mL                 Location : Vial 3
Acq. Operator   : GAA                                  Inj :   1
Acq. Instrument : Instrument 1                   Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 2:54:11 PM by AYY
                  (modified after loading)
ADP'S
==================================================================

==================================================================
                      External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 2:54:11 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime  Type     Area      Amt/Area    Amount   Grp   Name
 [min]                                  [ug/mL]
-------|------|----------|----------|----------|--|------------------
  7.072 MM     1.17879e5  3.82971e-7 4.51441e-2    VX

Totals :                               4.51441e-2

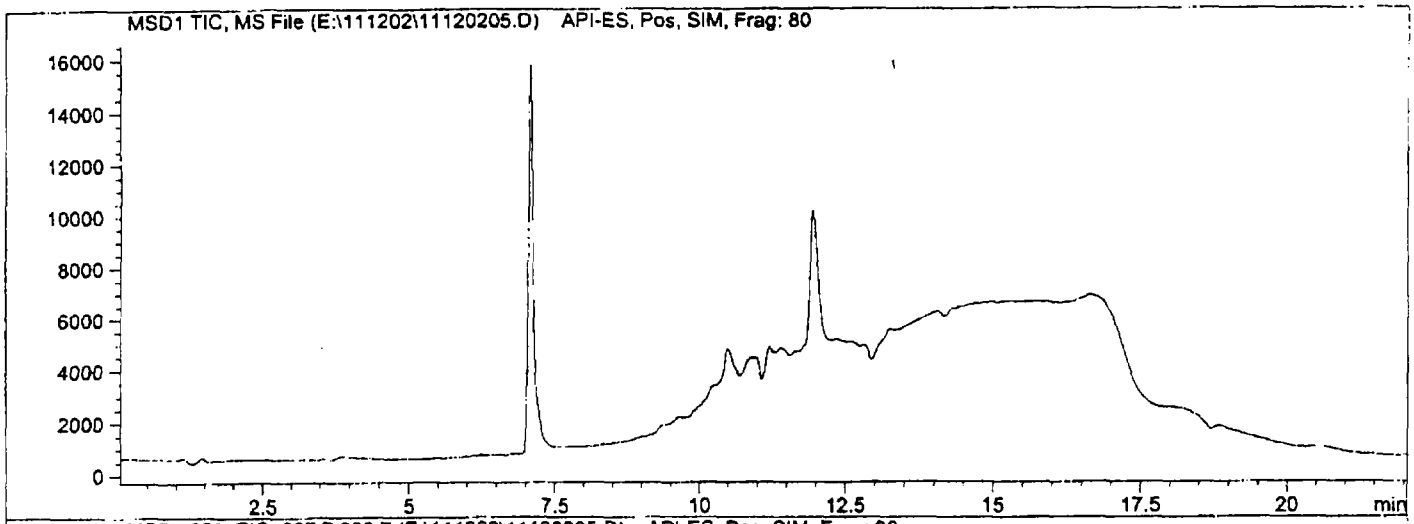

==================================================================
                         *** End of Report ***
```

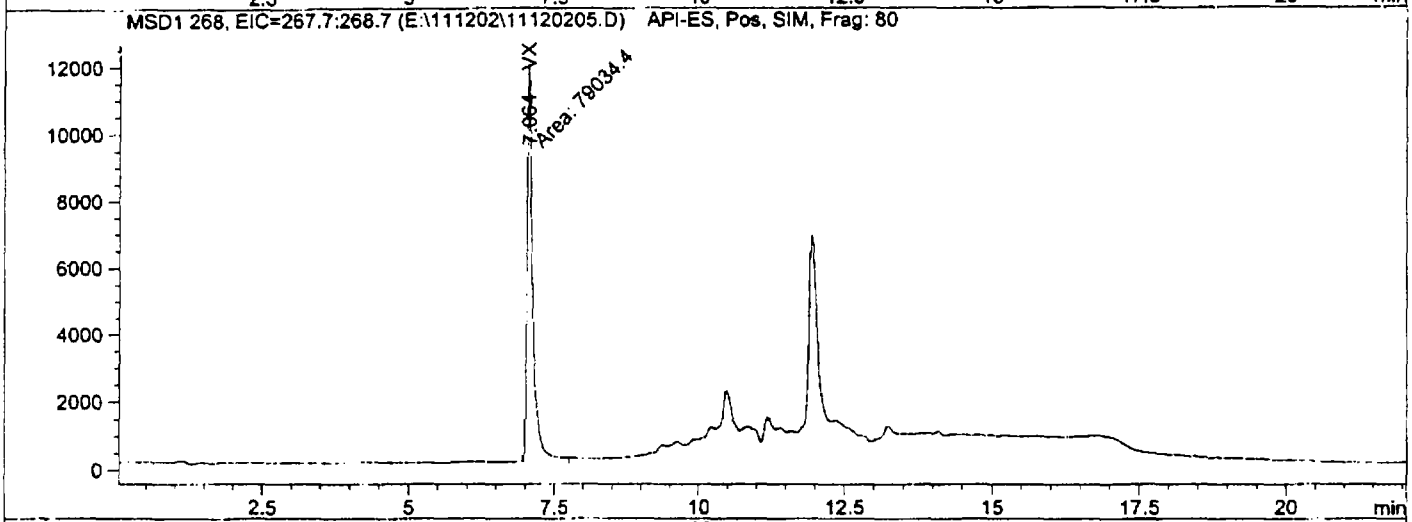
Data File E:\111202\11120205.D Sample Name: VX 0.0312 ug/mL

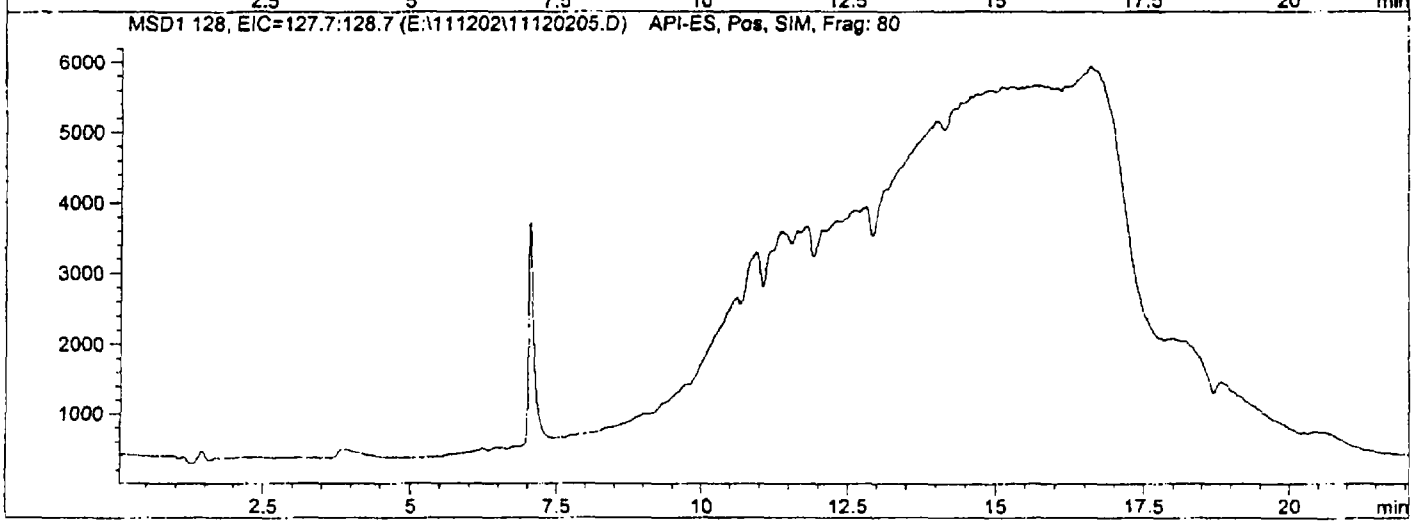
11

Injection Date	: 11/12/2002 6:02:49 PM	Seq. Line :	4
Sample Name	: VX 0.0312 ug/mL	Location :	Vial 4
Acq. Operator	: GAA	Inj :	1
Acq. Instrument	: Instrument 1	Inj Volume :	20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 2:57:14 PM by AYY (modified after loading)		

ADP'S







12

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==================================================================
Injection Date  : 11/12/2002 6:02:49 PM          Seq. Line :   4
Sample Name     : VX 0.0312 ug/mL                 Location : Vial 4
Acq. Operator   : GAA                                  Inj :   1
Acq. Instrument : Instrument 1                   Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 2:57:14 PM by AYY
                  (modified after loading)
ADP'S
==================================================================

==================================================================
                      External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      Thursday, January 02, 2003 2:55:14 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime  Type     Area      Amt/Area    Amount   Grp   Name
 [min]                                  [ug/mL]
-------|------|----------|----------|----------|--|------------------
  7.064 MM     7.90344e4  4.82358e-7 3.81229e-2    VX

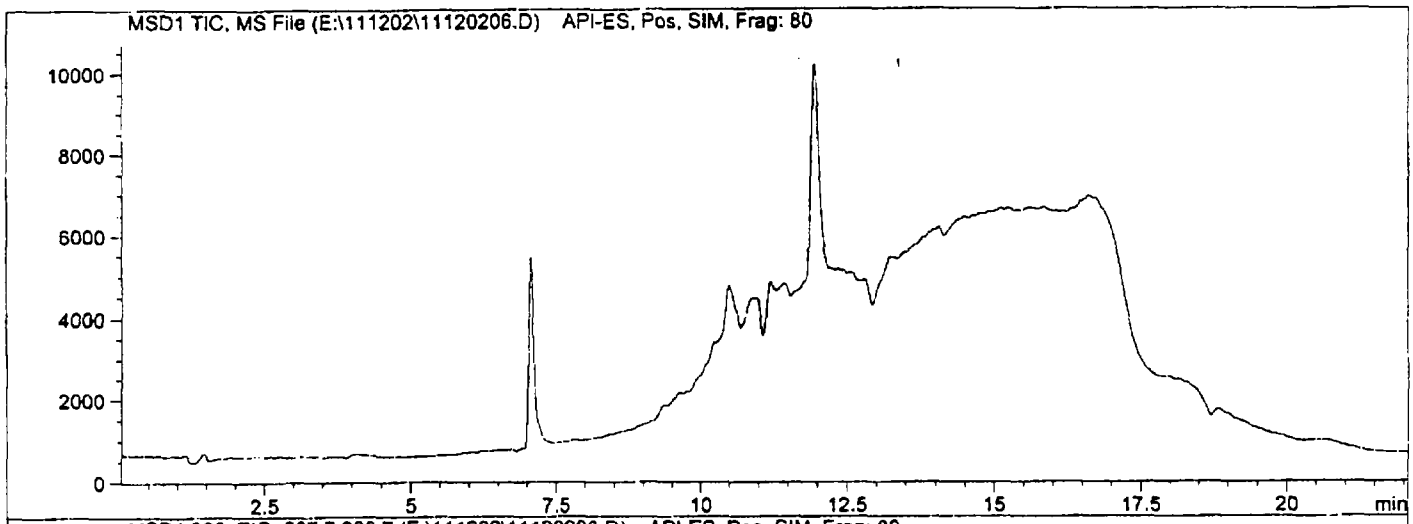
Totals :                               3.81229e-2

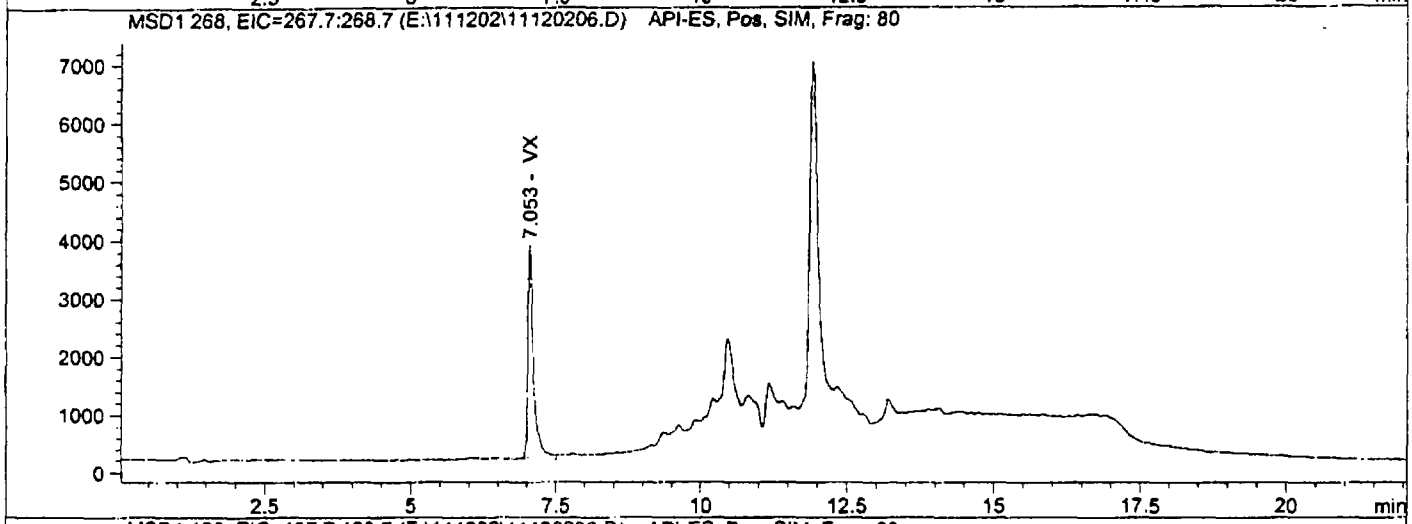

==================================================================
                        *** End of Report ***
```

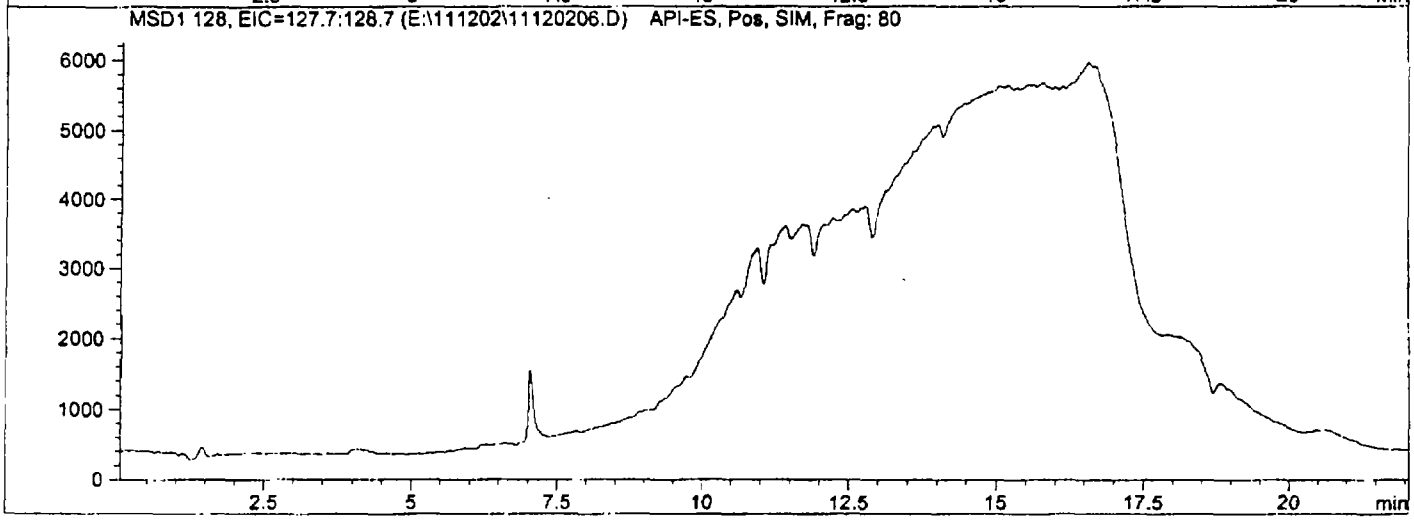
13

Injection Date	: 11/12/2002 6:26:33 PM	Seq. Line	: 5
Sample Name	: VX 0.0156 ug/mL	Location	: Vial 5
Acq. Operator	: GAA	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 2:57:59 PM by AYY (modified after loading)		

ADP'S







14

```
==================================================================
Injection Date  : 11/12/2002 6:26:33 PM          Seq. Line :   5
Sample Name     : VX 0.0156 ug/mL                 Location : Vial 5
Acq. Operator   : GAA                                 Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 2:57:59 PM by AYY
                  (modified after loading)
ADP'S
==================================================================

==================================================================
                      External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      Thursday, January 02, 2003 2:57:58 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime  Type     Area      Amt/Area    Amount   Grp   Name
 [min]                                  [ug/mL]
-------|------|----------|----------|----------|--|-----------------
  7.053 PB     2.40980e4  7.41213e-7 1.78618e-2    VX

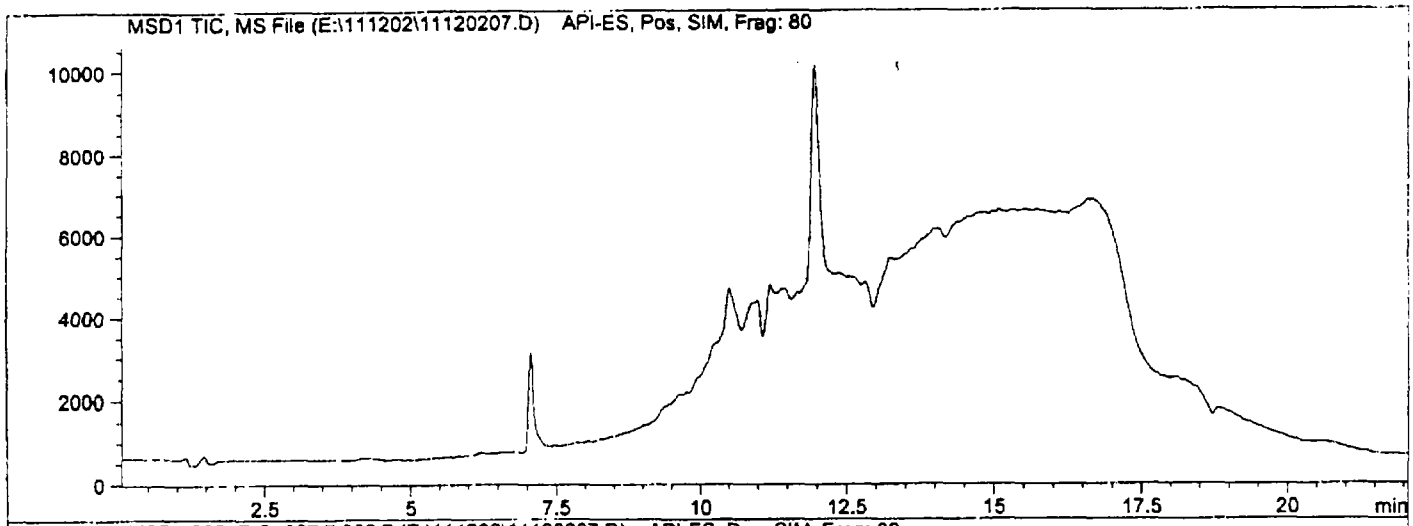
Totals :                               1.78618e-2

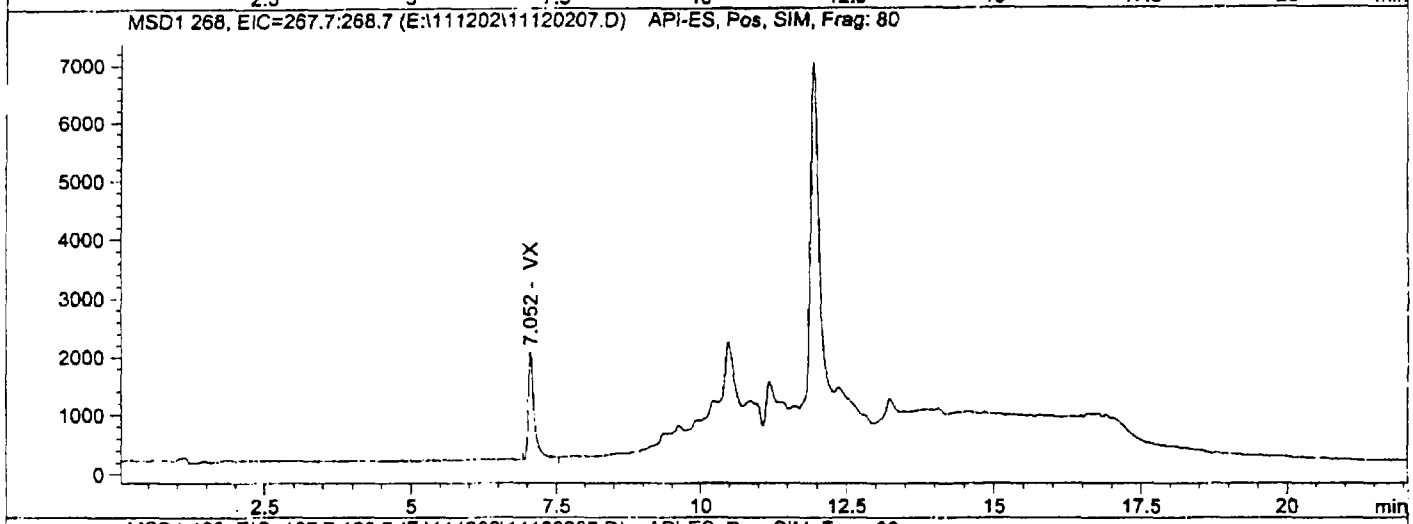

==================================================================
                         *** End of Report ***
```

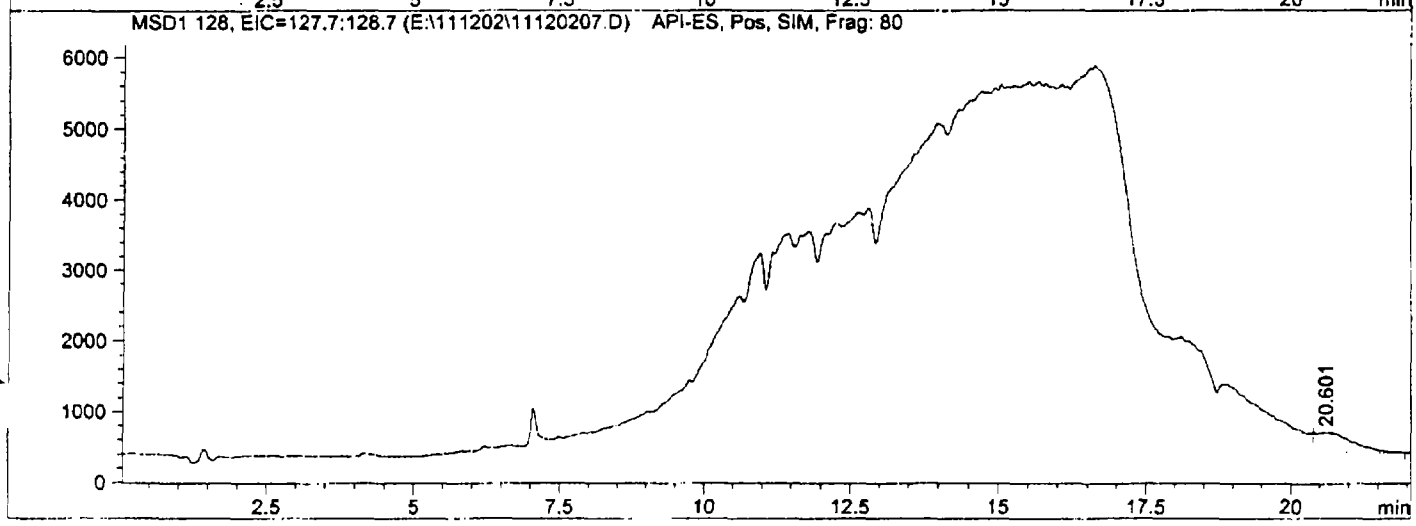
15

Injection Date	: 11/12/2002 6:50:17 PM	Seq. Line :	6
Sample Name	: VX 0.007 ug/mL	Location :	Vial 6
Acq. Operator	: GAA	Inj :	1
Acq. Instrument	: Instrument 1	Inj Volume :	20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 2:59:55 PM by AYY (modified after loading)		

ADP'S







16

```
==================================================================
Injection Date  : 11/12/2002 6:50:17 PM          Seq. Line :   6
Sample Name     : VX 0.007 ug/mL                  Location : Vial 6
Acq. Operator   : GAA                                  Inj :   1
Acq. Instrument : Instrument 1                   Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 2:59:55 PM by AYY
                  (modified after loading)
ADP'S
==================================================================

==================================================================
                      External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      Thursday, January 02, 2003 2:59:54 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

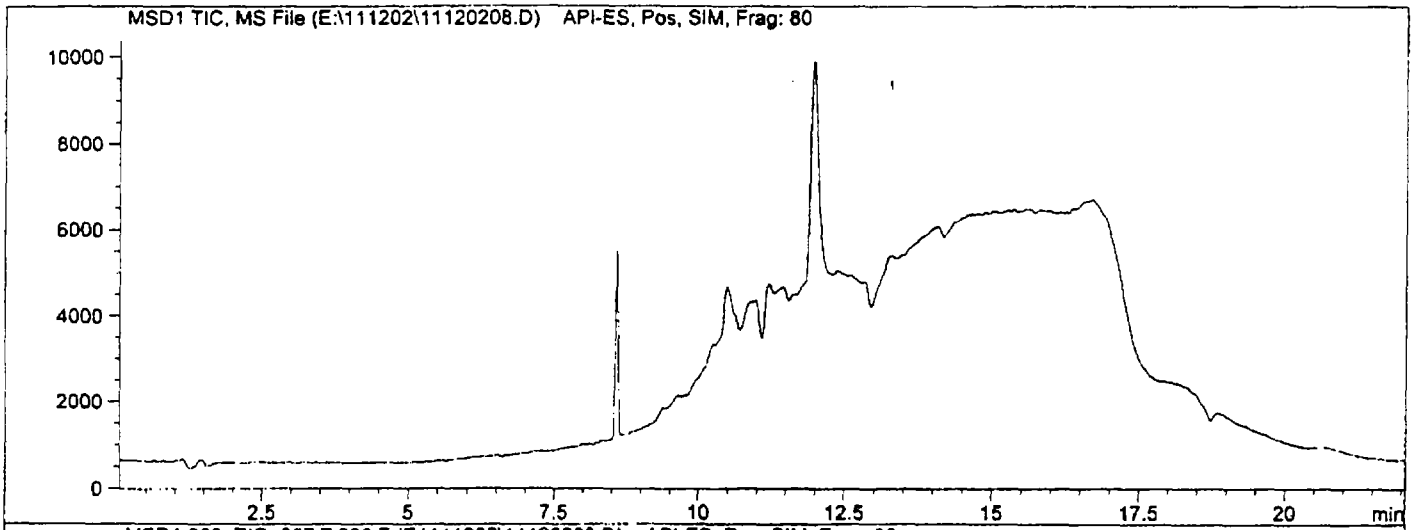
RetTime  Type     Area      Amt/Area    Amount   Grp   Name
 [min]                                  [ug/mL]
-------|------|----------|----------|----------|--|------------------
  7.052 PB     1.24100e4  9.65991e-7 1.19880e-2    VX

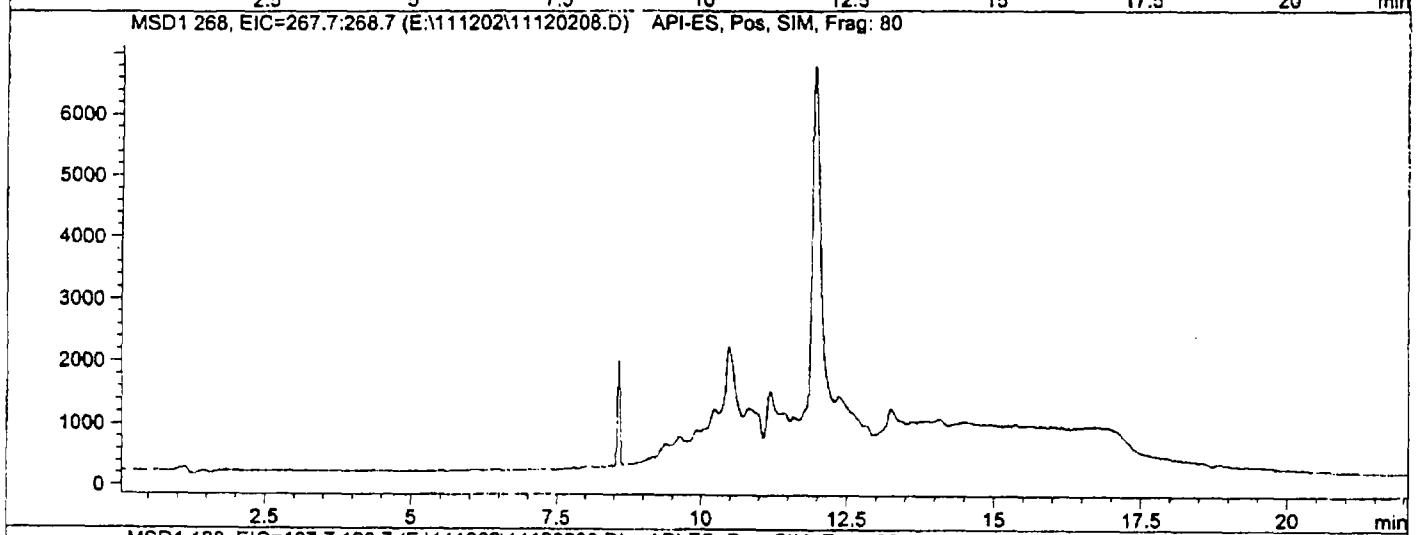
Totals :                               1.19880e-2

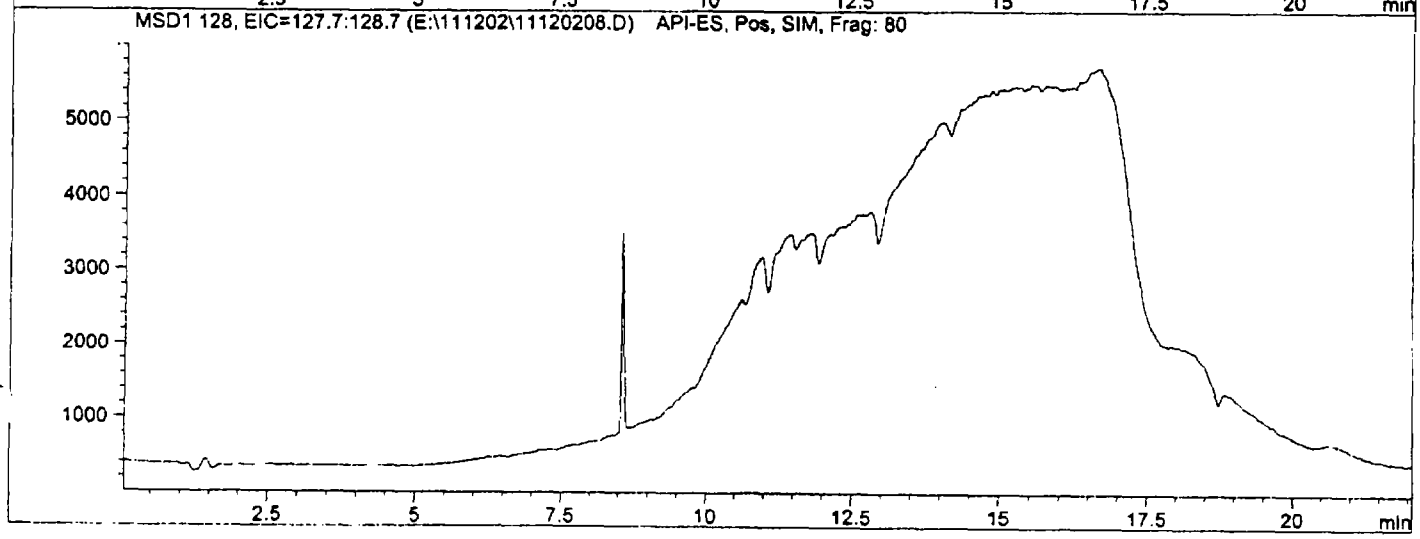

==================================================================
                        *** End of Report ***
```

17

Injection Date	: 11/12/2002 7:14:00 PM	Seq. Line :	7
Sample Name	: WATER BLK	Location :	Vial 7
Acq. Operator	: GAA	Inj :	1
Acq. Instrument	: Instrument 1	Inj Volume :	20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 3:00:21 PM by AYY		
ADP'S			







18

```
==================================================================
Injection Date  : 11/12/2002 7:14:00 PM          Seq. Line :   7
Sample Name     : WATER BLK                       Location : Vial 7
Acq. Operator   : GAA                                  Inj :   1
Acq. Instrument : Instrument 1                   Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 3:00:21 PM by AYY
ADP'S
==================================================================

==================================================================
                    External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 3:00:17 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime  Type     Area     Amt/Area    Amount   Grp   Name
 [min]                                [ug/mL]
-------|------|----------|----------|----------|--|------------------
  7.052            -          -          -         VX

Totals :                                0.00000


1 Warnings or Errors :

Warning : Calibrated compound(s) not found

==================================================================
==================================================================
                       Area Percent Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 3:00:17 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: MSD1 TIC, MS File


Signal 2: MSD1 268, EIC=267.7:268.7

Peak RetTime  Type   Width     Area      Area    Name
  #   [min]          [min]                 %
----|-------|------|-------|----------|--------|---------------------
   1   7.052         0.0000    0.00000   0.0000 VX

Totals :                       0.00000   0.0000


Signal 3: MSD1 128, EIC=127.7:128.7

1 Warnings or Errors :

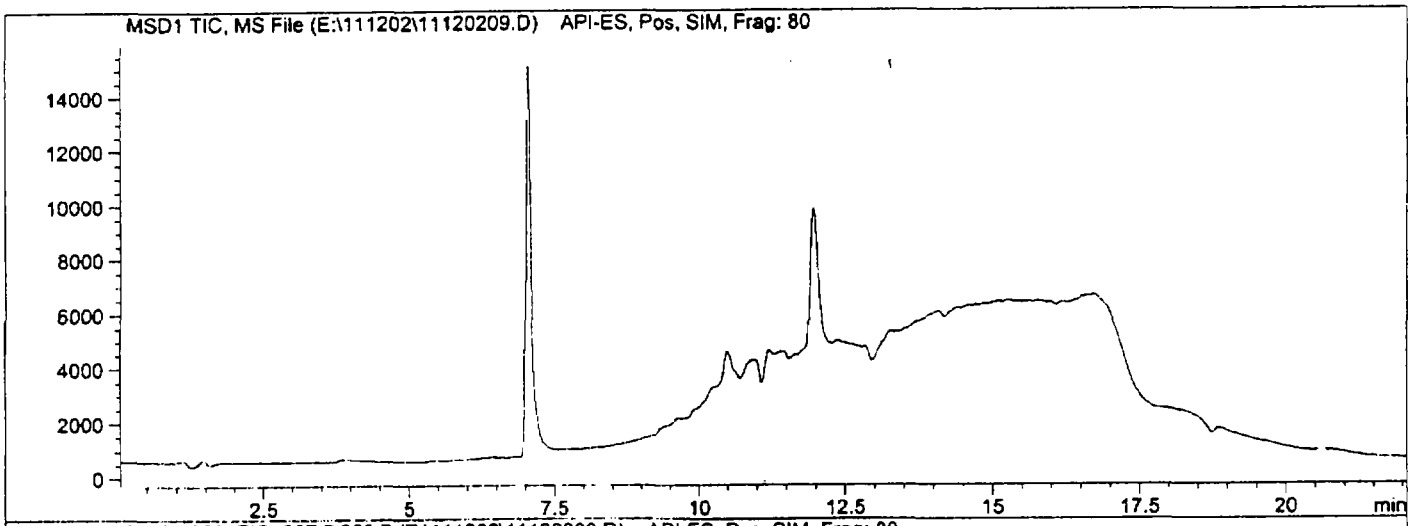
Warning : Calibrated compound(s) not found

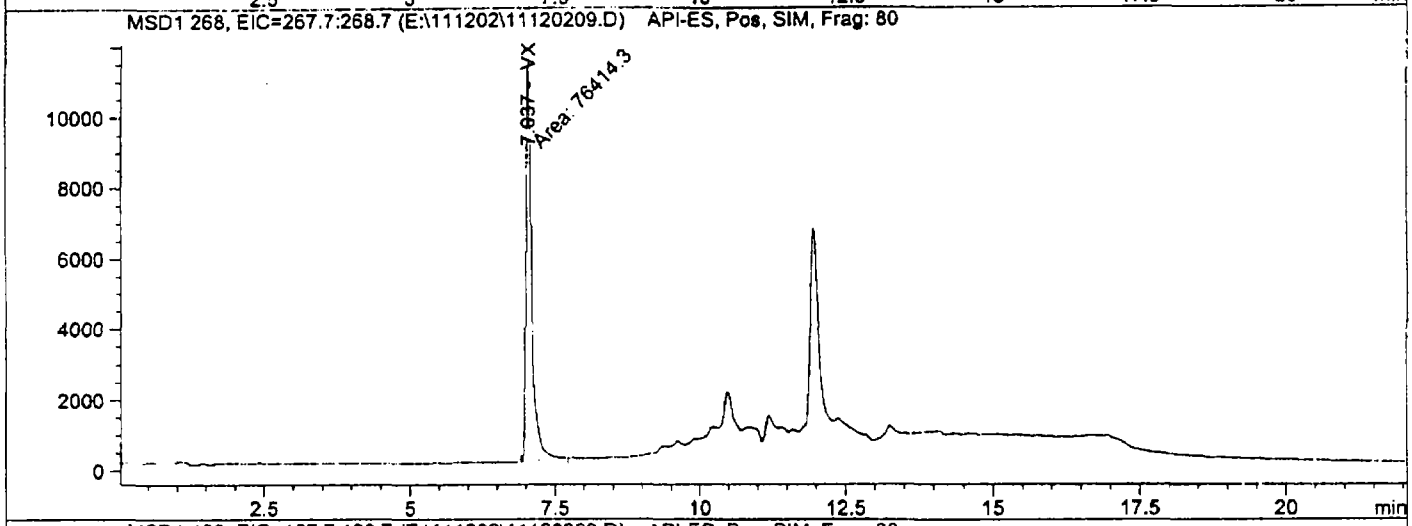
==================================================================
```

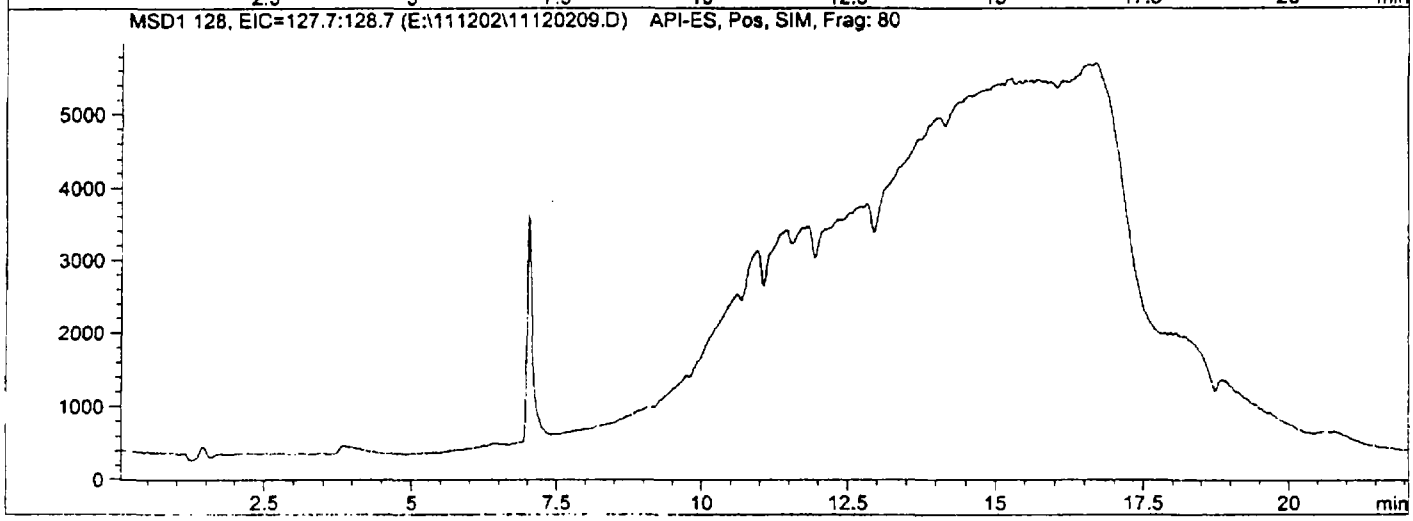
19

Injection Date	: 11/12/2002 7:37:46 PM	Seq. Line :	8
Sample Name	: CCS 1 0.0312	Location :	Vial 8
Acq. Operator	: GAA	Inj :	1
Acq. Instrument	: Instrument 1	Inj Volume :	20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 3:01:47 PM by AYY (modified after loading)		

ADP'S







20

```
==================================================================
Injection Date  : 11/12/2002 7:37:46 PM          Seq. Line :   8
Sample Name     : CCS 1 0.0312                    Location : Vial 8
Acq. Operator   : GAA                                 Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 3:01:47 PM by AYY
                  (modified after loading)
ADP'S
==================================================================

==================================================================
                      External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 3:00:17 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime  Type     Area      Amt/Area    Amount   Grp   Name
 [min]                                  [ug/mL]
-------|------|----------|----------|----------|--|------------------
  7.037 MM     7.64143e4  4.38200e-7 3.34848e-2    VX

Totals :                               3.34848e-2

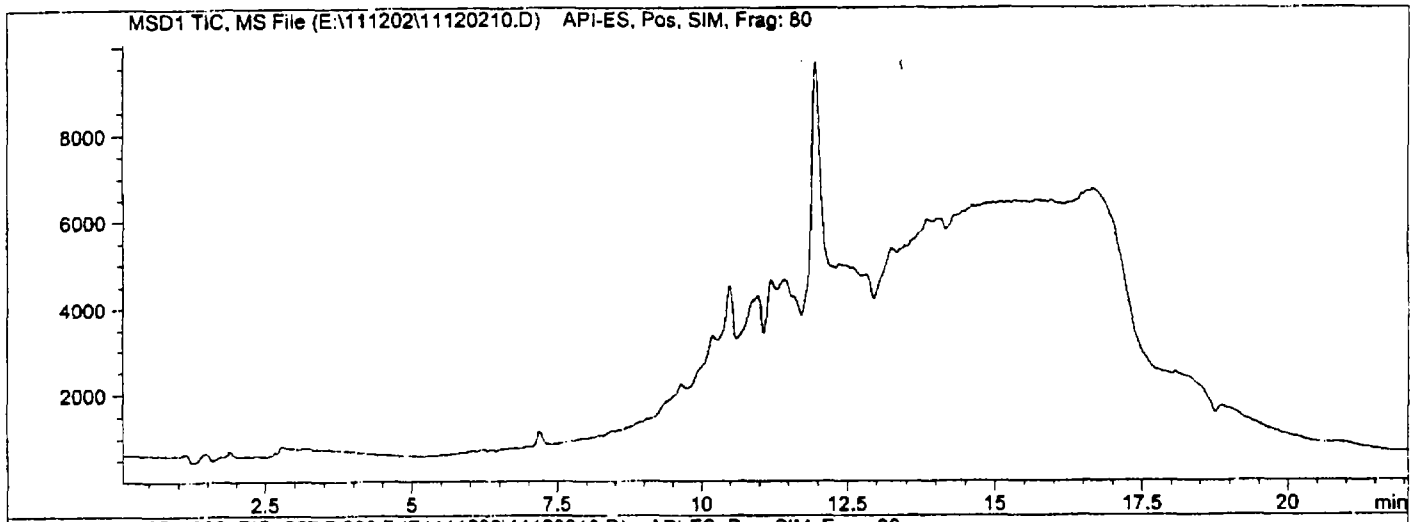

==================================================================
                         *** End of Report ***
```

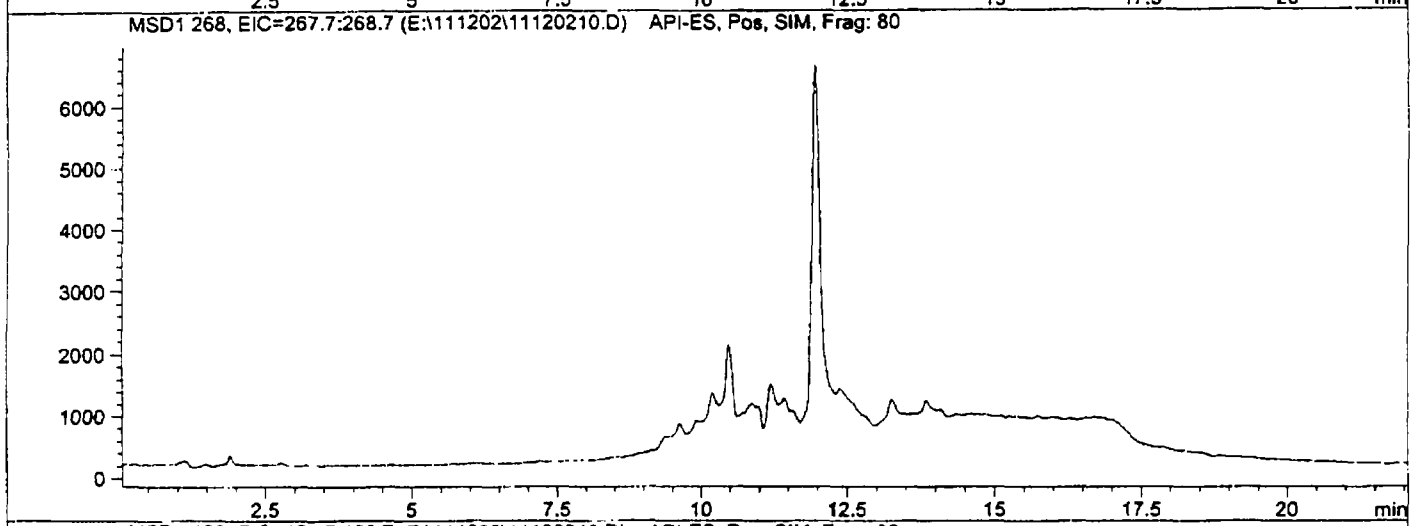
Data File E:\111202\11120210.D Sample Name: ACETONE BLK 4X

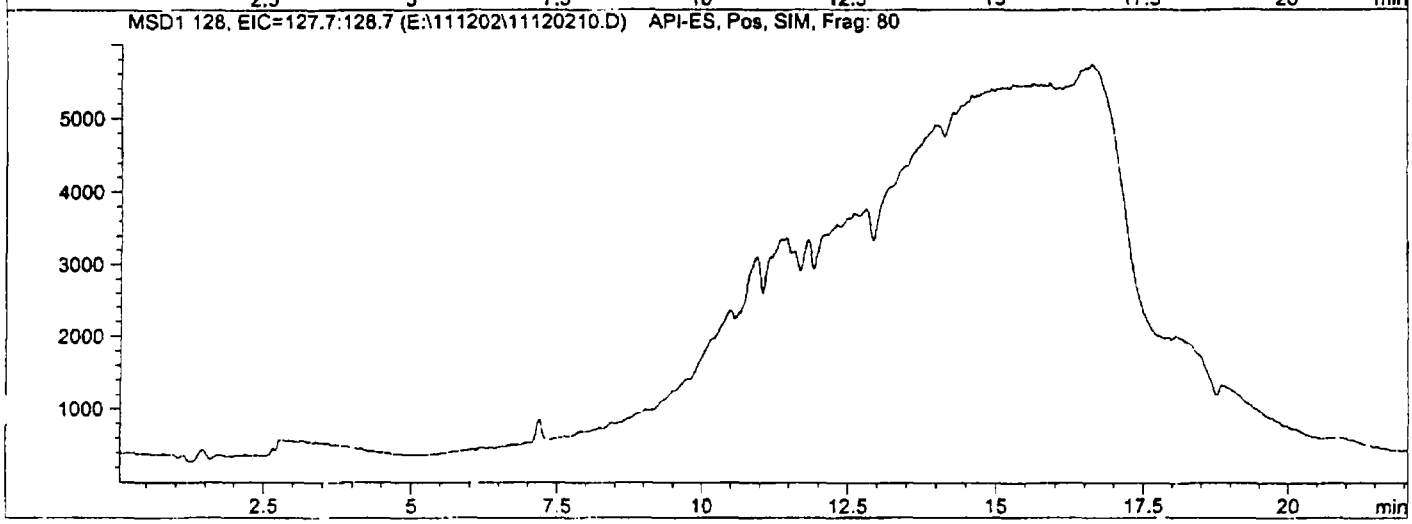
21

Injection Date	: 11/12/2002 8:01:32 PM	Seq. Line :	9
Sample Name	: ACETONE BLK 4X	Location :	Vial 9
Acq. Operator	: GAA	Inj :	1
Acq. Instrument	: Instrument 1	Inj Volume :	20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 3:02:07 PM by AYY (modified after loading)		

ADP'S







22

```
==================================================================
Injection Date  : 11/12/2002 8:01:32 PM          Seq. Line :   9
Sample Name     : ACETONE BLK 4X                  Location : Vial 9
Acq. Operator   : GAA                                 Inj :   1
Acq. Instrument : Instrument 1                   Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 3:02:07 PM by AYY
                  (modified after loading)
ADP'S
==================================================================

==================================================================
                      External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 3:00:17 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime  Type     Area     Amt/Area    Amount   Grp   Name
 [min]                                [ug/mL]
-------|------|----------|----------|----------|--|------------------
  7.052            -          -          -        VX

Totals :                                0.00000


1 Warnings or Errors :

Warning : Calibrated compound(s) not found

==================================================================
==================================================================
                        Area Percent Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 3:00:17 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: MSD1 TIC, MS File


Signal 2: MSD1 268, EIC=267.7:268.7

Peak RetTime  Type   Width     Area       Area    Name
  #   [min]          [min]                  %
----|-------|------|-------|----------|--------|--------------------
   1   7.052         0.0000    0.00000   0.0000 VX

Totals :                      0.00000   0.0000


Signal 3: MSD1 128, EIC=127.7:128.7
```

23

```
==================================================================
Injection Date  : 11/12/2002 8:01:32 PM          Seq. Line :   9
Sample Name     : ACETONE BLK 4X                  Location : Vial 9
Acq. Operator   : GAA                                  Inj :   1
Acq. Instrument : Instrument 1                   Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 3:02:07 PM by AYY
                  (modified after loading)
ADP'S
==================================================================


1 Warnings or Errors :

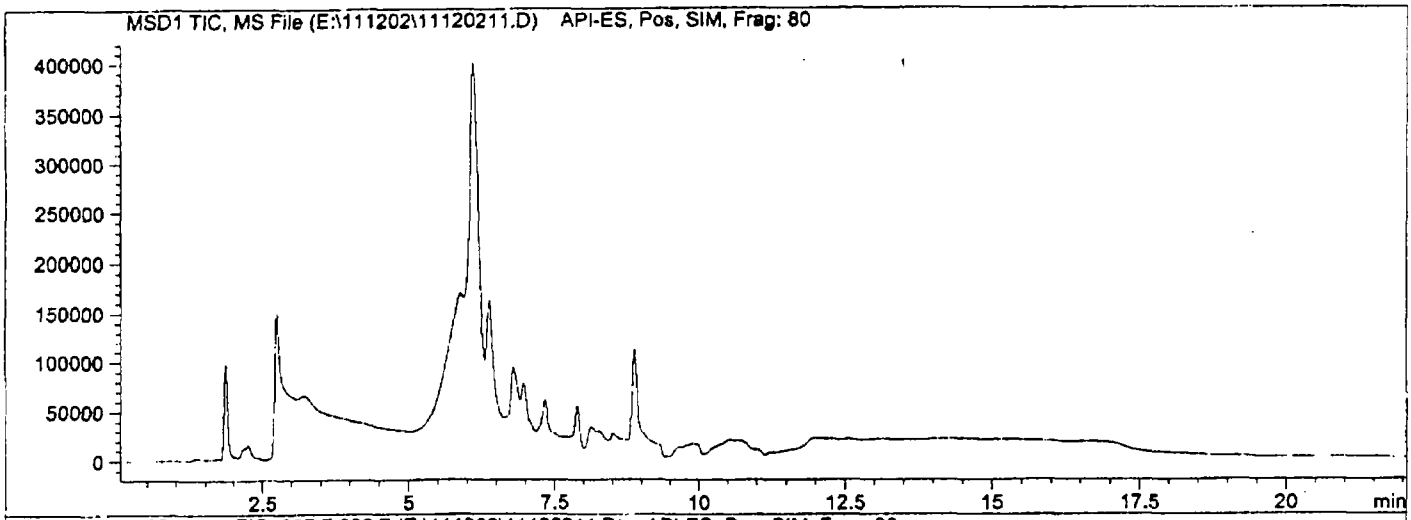
Warning : Calibrated compound(s) not found

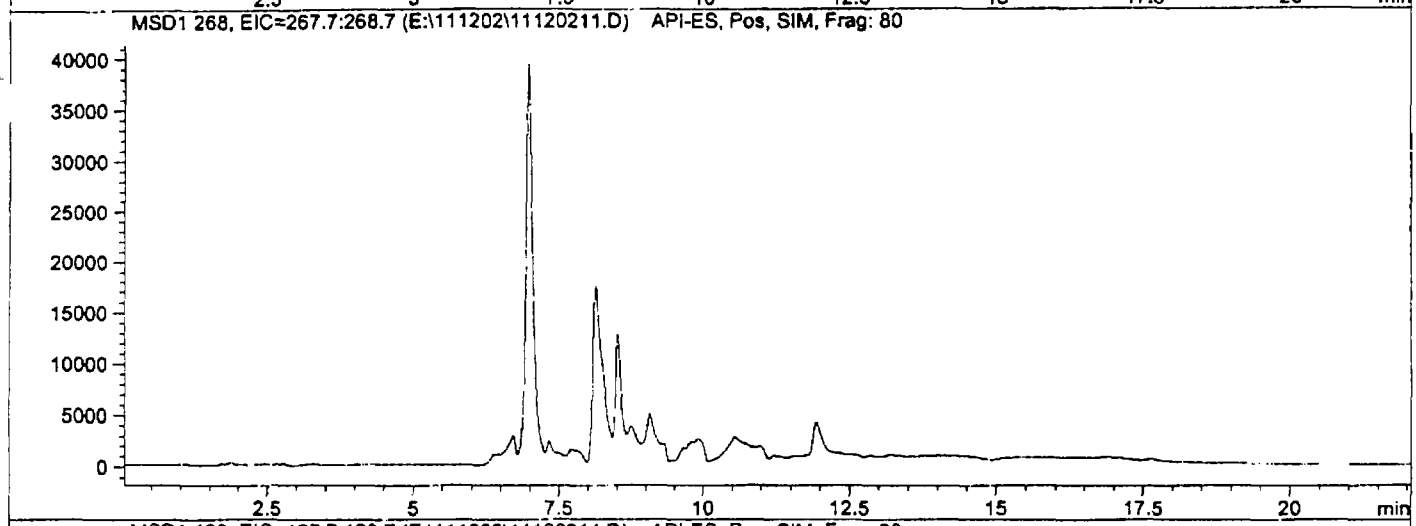
==================================================================
                         *** End of Report ***
```

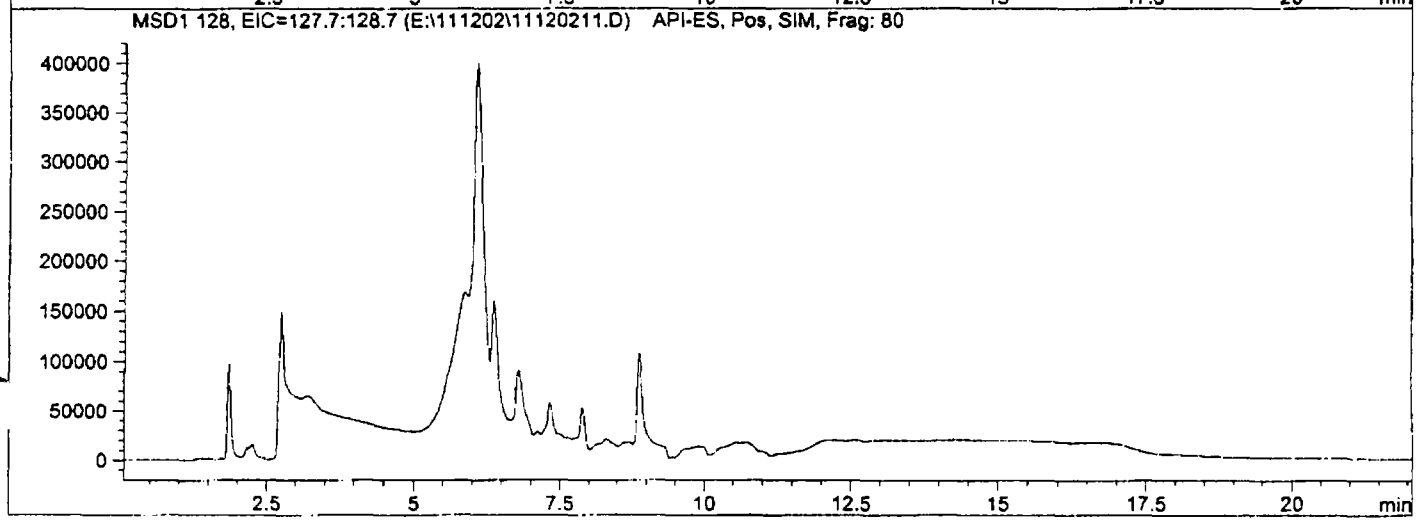
24

Injection Date	: 11/12/2002 8:25:19 PM	Seq. Line	: 10
Sample Name	: R1 4X	Location	: Vial 10
Acq. Operator	: GAA	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 3:02:35 PM by AYY (modified after loading)		

ADP'S







25

```
==================================================================
Injection Date  : 11/12/2002 8:25:19 PM          Seq. Line :  10
Sample Name     : R1 4X                           Location : Vial 10
Acq. Operator   : GAA                                  Inj :   1
Acq. Instrument : Instrument 1                   Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 3:02:35 PM by AYY
                  (modified after loading)
ADP'S
==================================================================

==================================================================
                      External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 3:00:17 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime  Type     Area     Amt/Area    Amount   Grp   Name
 [min]                                [ug/mL]
-------|------|----------|----------|----------|--|------------------
  7.052            -          -          -         VX

Totals :                                0.00000


1 Warnings or Errors :

Warning : Calibrated compound(s) not found

==================================================================
==================================================================
                         Area Percent Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 3:00:17 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: MSD1 TIC, MS File


Signal 2: MSD1 268, EIC=267.7:268.7

Peak RetTime  Type   Width     Area      Area    Name
  #   [min]          [min]                 %
----|-------|------|-------|----------|--------|--------------------
   1   7.052         0.0000    0.00000   0.0000 VX

Totals :                       0.00000   0.0000


Signal 3: MSD1 128, EIC=127.7:128.7
```

26

```
==================================================================
Injection Date  : 11/12/2002 8:25:19 PM          Seq. Line :  10
Sample Name     : R1 4X                           Location : Vial 10
Acq. Operator   : GAA                                 Inj :   1
Acq. Instrument : Instrument 1                   Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 3:02:35 PM by AYY
                  (modified after loading)
ADP'S
==================================================================


1 Warnings or Errors :

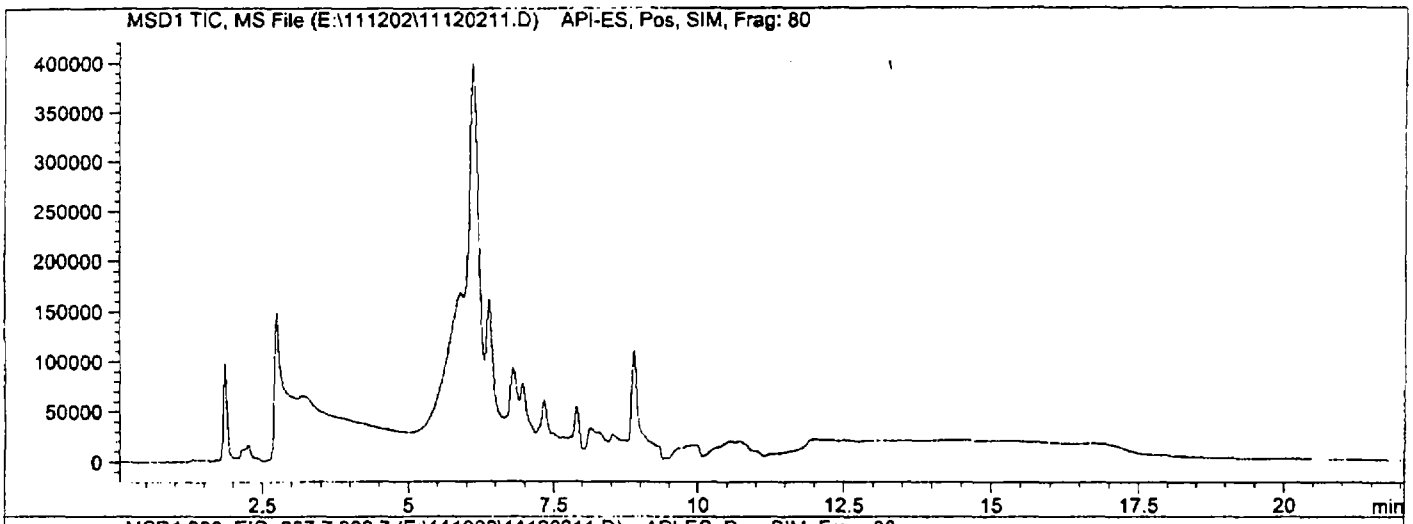
Warning : Calibrated compound(s) not found

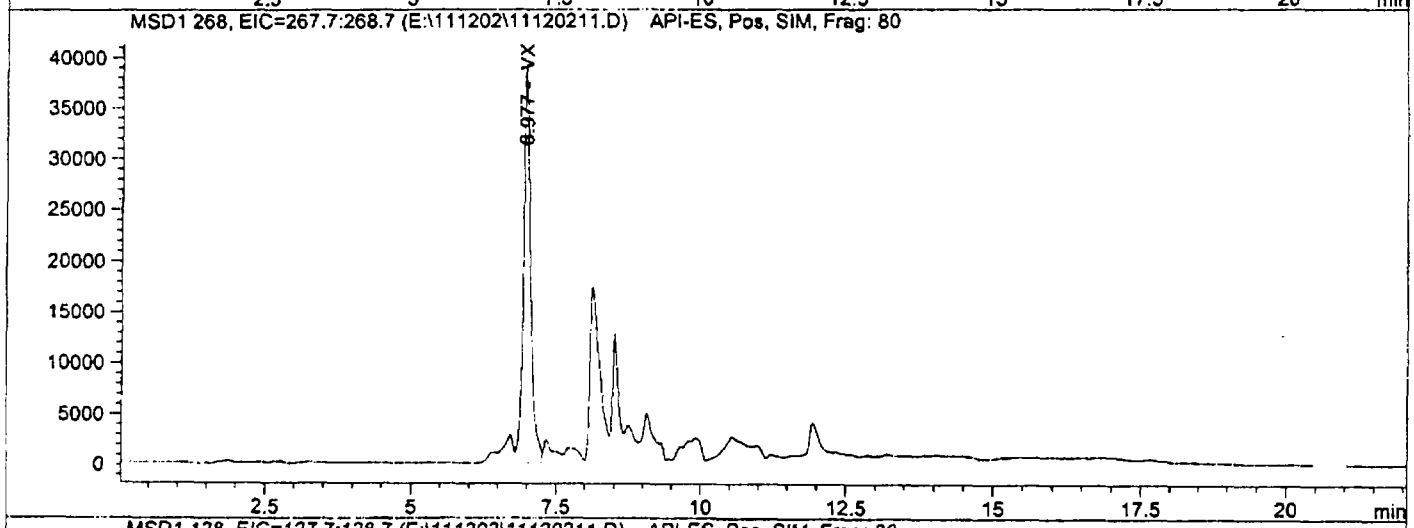
==================================================================
                         *** End of Report ***
```

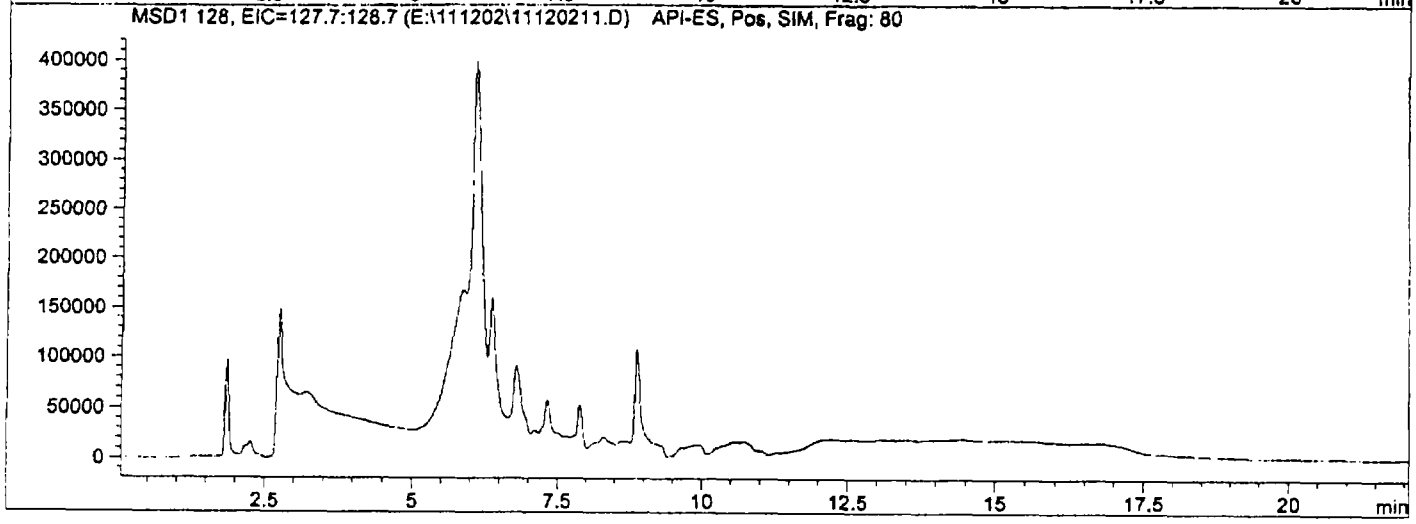
27

Injection Date	: 11/12/2002 8:25:19 PM	Seq. Line	: 10
Sample Name	: R1 4X	Location	: Vial 10
Acq. Operator	: GAA	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 3:06:35 PM by AYY (modified after loading)		

ADP'S







28

```
==================================================================
Injection Date  : 11/12/2002 8:25:19 PM          Seq. Line :  10
Sample Name     : R1 4X                           Location : Vial 10
Acq. Operator   : GAA                                 Inj :   1
Acq. Instrument : Instrument 1                   Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 3:06:35 PM by AYY
                  (modified after loading)
ADP'S
==================================================================

==================================================================
                      External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 3:00:17 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime  Type     Area      Amt/Area    Amount   Grp   Name
 [min]                                  [ug/mL]
-------|------|----------|----------|----------|--|-----------------
  6.977 VV     3.36858e5  3.59079e-7 1.20959e-1    VX

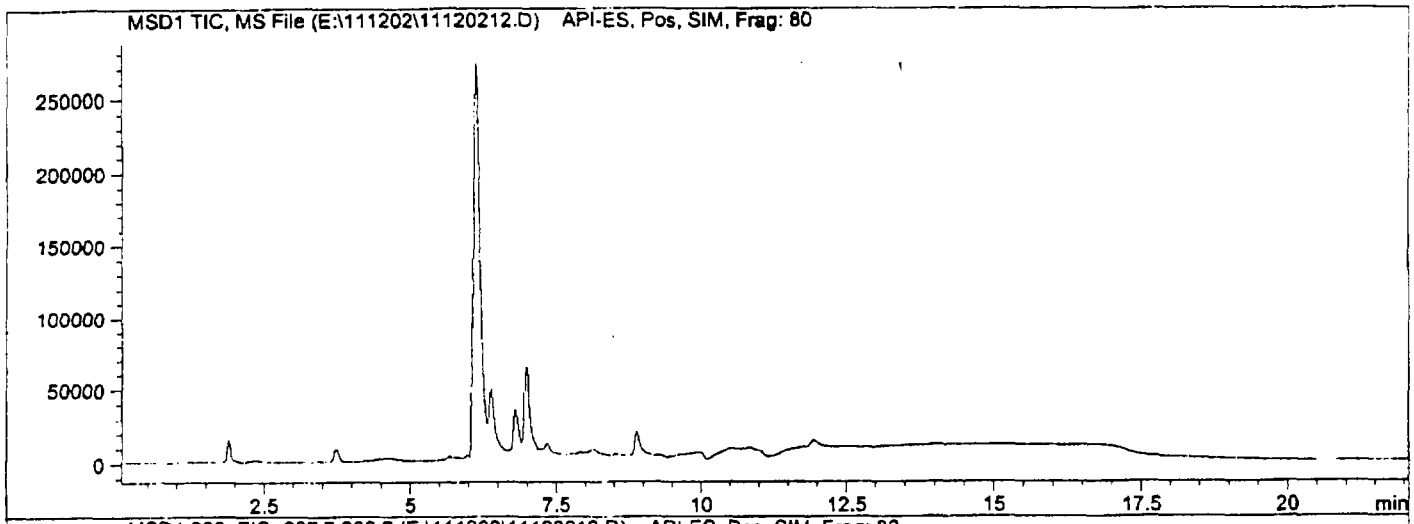
Totals :                               1.20959e-1

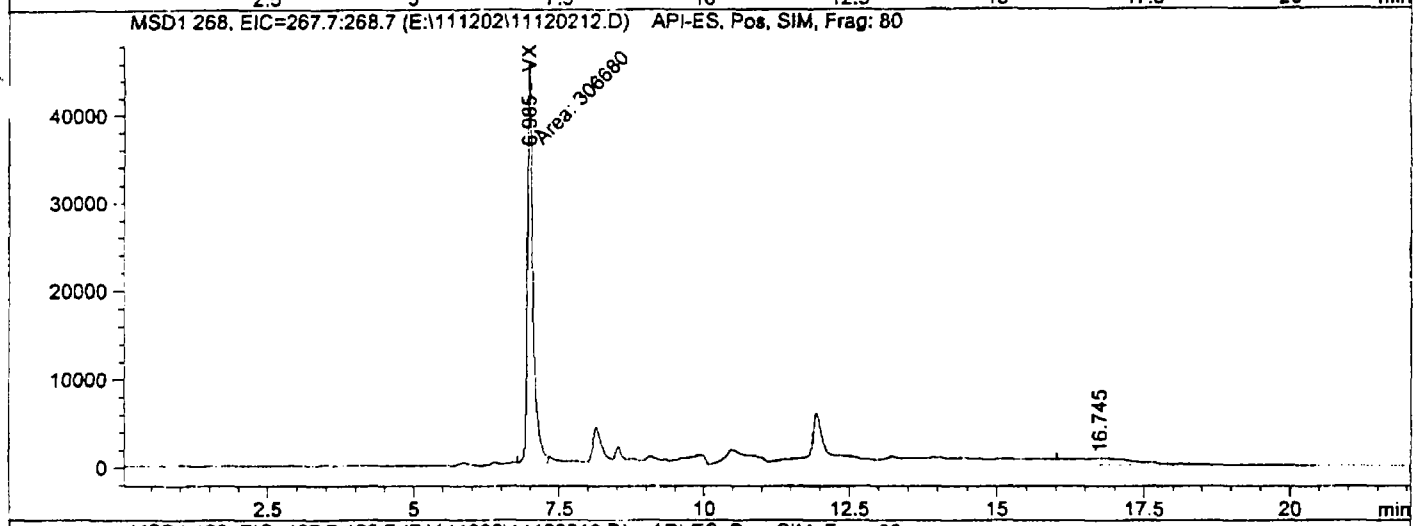

==================================================================
                        *** End of Report ***
```

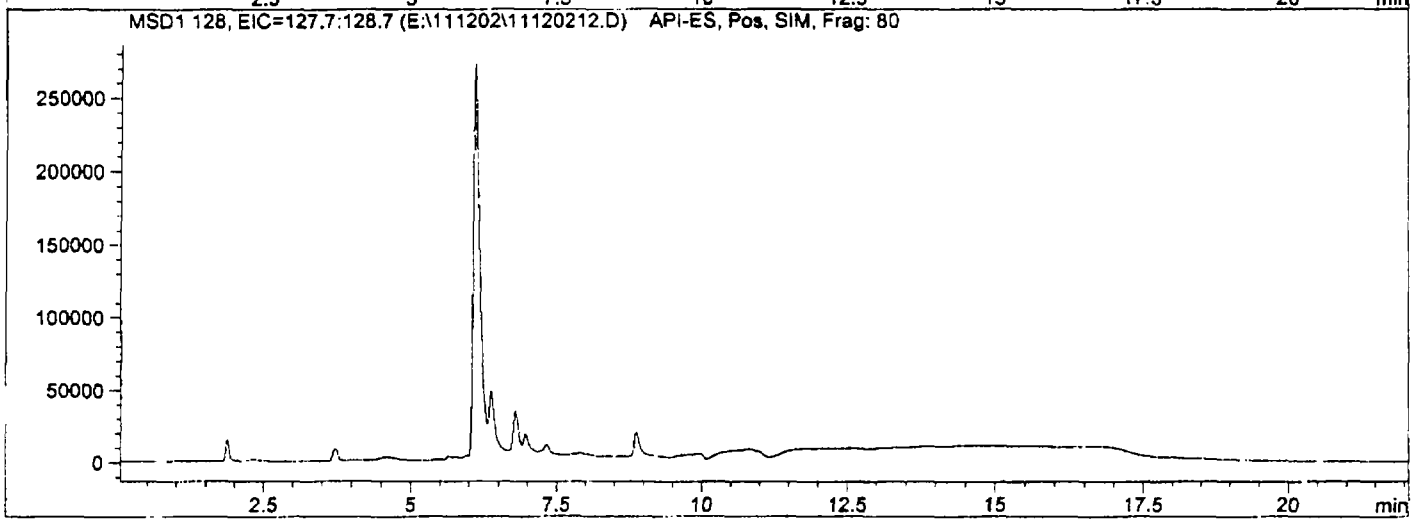
29

Injection Date	: 11/12/2002 8:49:10 PM	Seq. Line	: 11
Sample Name	: R1 MS 25X	Location	: Vial 11
Acq. Operator	: GAA	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 3:11:16 PM by AYY (modified after loading)		

ADP'S







30

```
==================================================================
Injection Date  : 11/12/2002 8:49:10 PM          Seq. Line :  11
Sample Name     : R1 MS 25X                       Location : Vial 11
Acq. Operator   : GAA                                  Inj :   1
Acq. Instrument : Instrument 1                   Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 3:11:16 PM by AYY
                  (modified after loading)
ADP'S
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==================================================================
                      External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 3:00:17 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime  Type     Area      Amt/Area    Amount   Grp   Name
 [min]                                  [ug/mL]
-------|------|----------|----------|----------|--|------------------
  6.985 MM     3.06680e5  3.61364e-7 1.10823e-1    VX

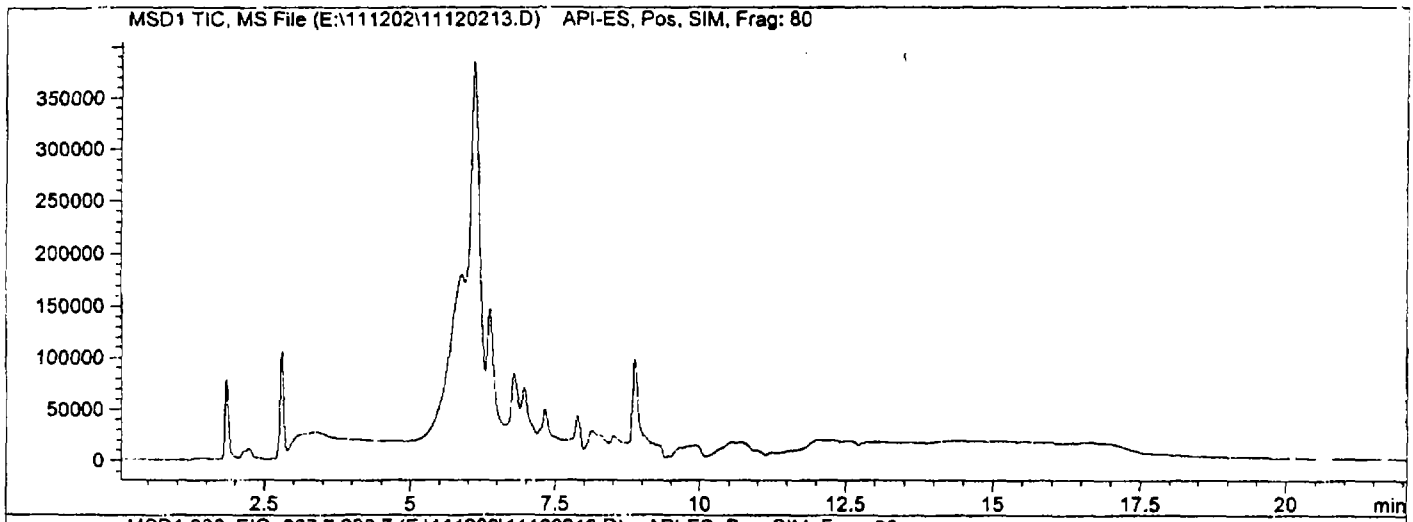
Totals :                               1.10823e-1

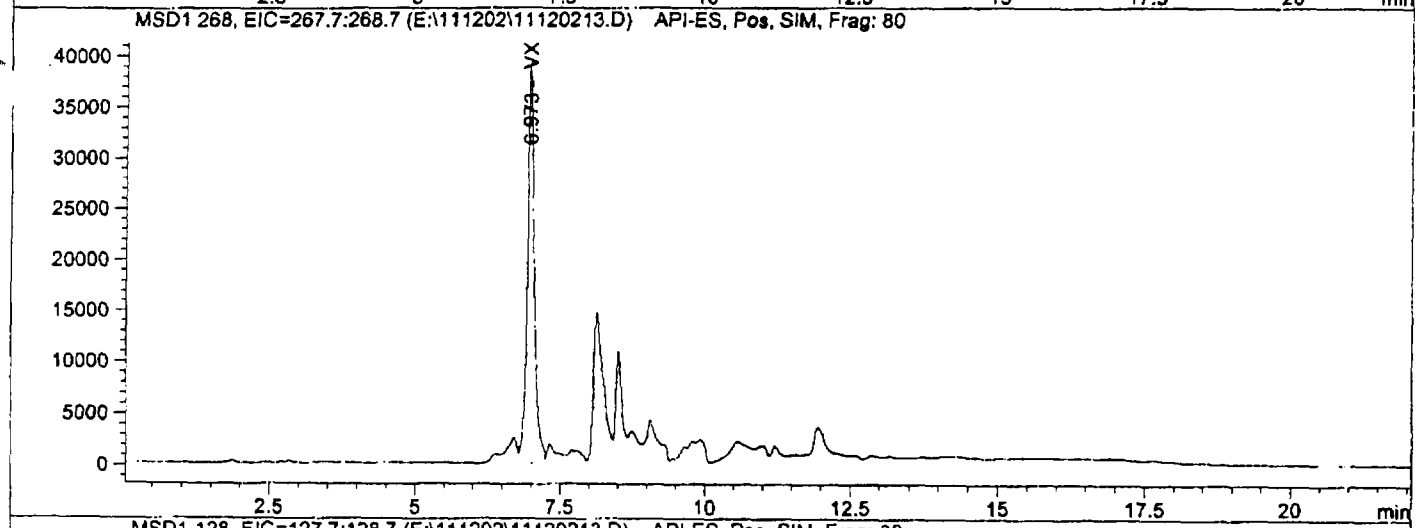

==================================================================
                        *** End of Report ***
```

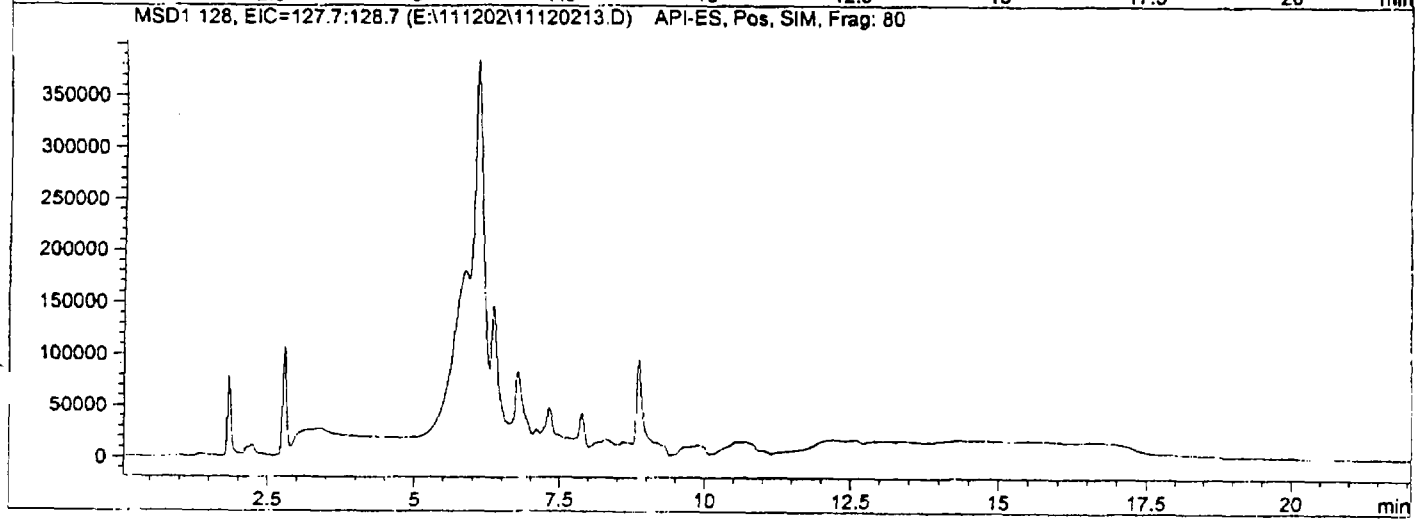
31

Injection Date	: 11/12/2002 9:12:56 PM	Seq. Line	: 12
Sample Name	: R2 4X	Location	: Vial 12
Acq. Operator	: GAA	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 3:18:04 PM by AYY (modified after loading)		

ADP'S







32

```
==================================================================
Injection Date  : 11/12/2002 9:12:56 PM          Seq. Line :  12
Sample Name     : R2 4X                           Location : Vial 12
Acq. Operator   : GAA                                 Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 3:18:04 PM by AYY
                  (modified after loading)
ADP'S
==================================================================

==================================================================
                      External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 3:00:17 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime  Type     Area      Amt/Area    Amount   Grp   Name
 [min]                                  [ug/mL]
-------|------|----------|----------|----------|--|-----------------
  6.973 VV     3.26105e5  3.59845e-7 1.17347e-1    VX

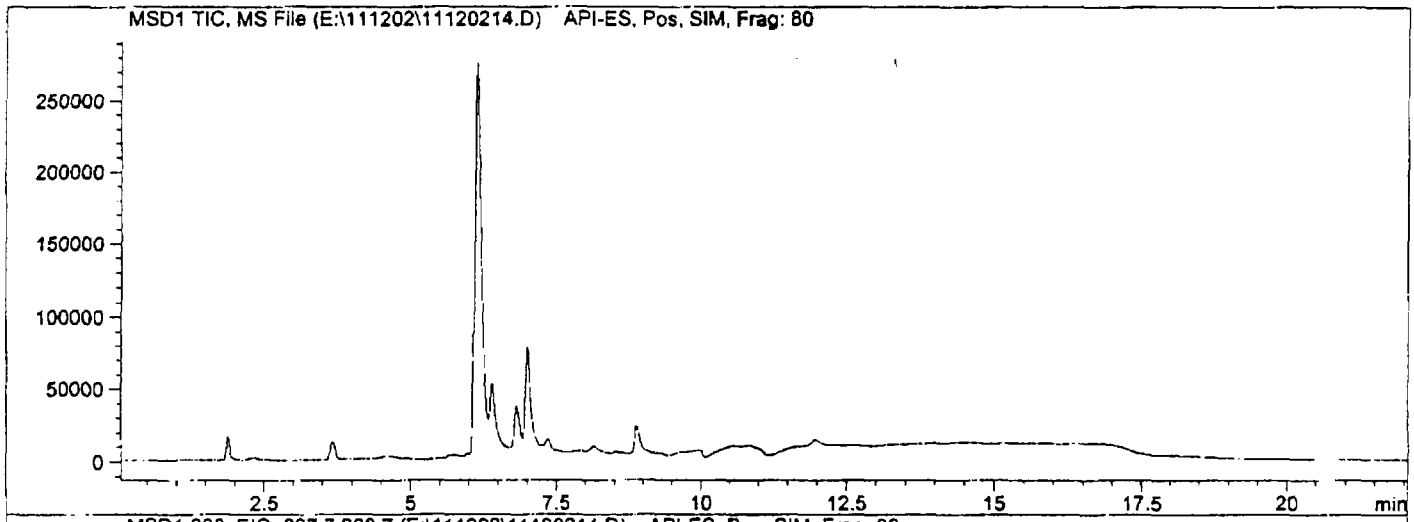
Totals :                               1.17347e-1

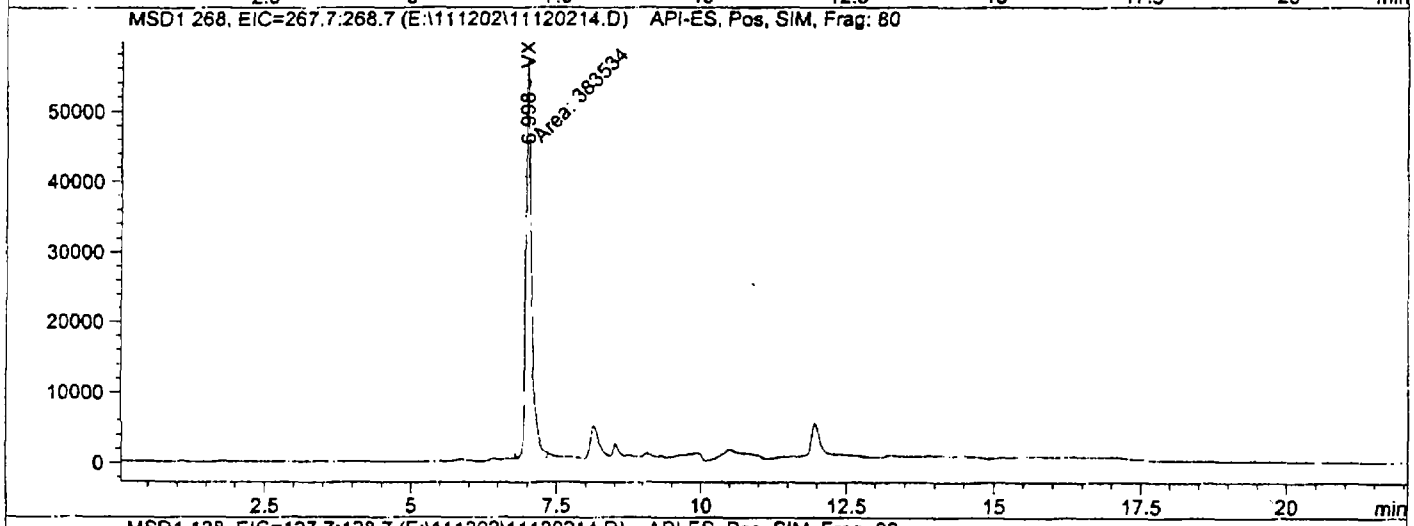

==================================================================
                        *** End of Report ***
```

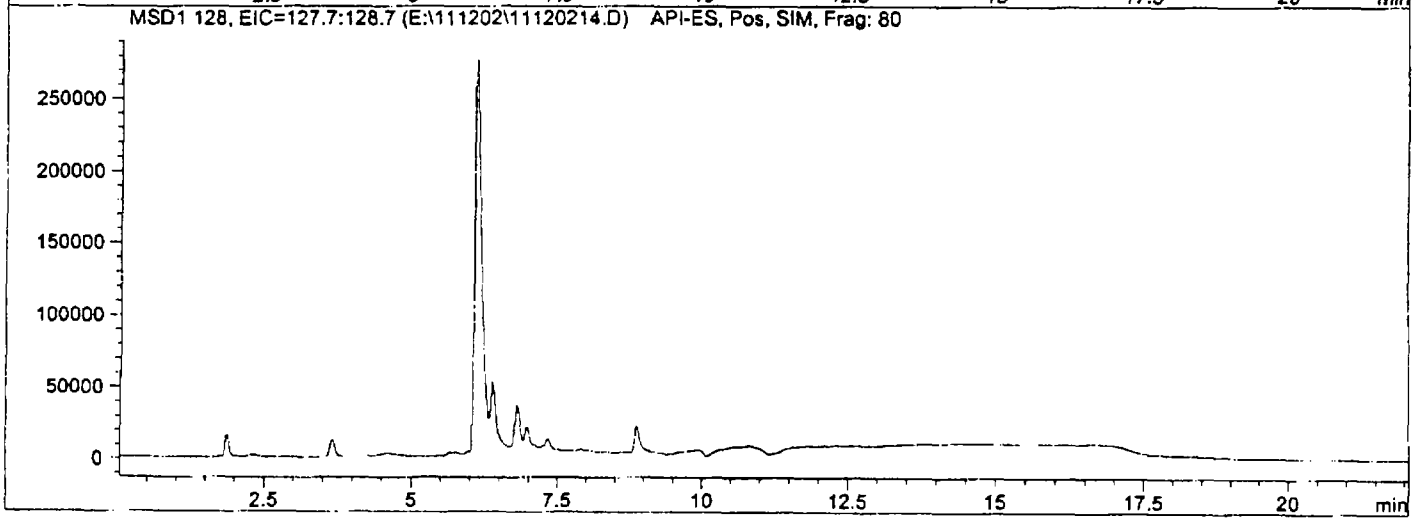
Injection Date	: 11/12/2002 9:36:40 PM	Seq. Line	: 13
Sample Name	: R1 MSD 25X	Location	: Vial 13
Acq. Operator	: GAA	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 3:18:37 PM by AYY (modified after loading)		

ADP'S

33







34

```
==================================================================
Injection Date  : 11/12/2002 9:36:40 PM          Seq. Line :  13
Sample Name     : R1 MSD 25X                      Location : Vial 13
Acq. Operator   : GAA                                  Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 3:18:37 PM by AYY
                  (modified after loading)
ADP'S
==================================================================

==================================================================
                      External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 3:00:17 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime  Type     Area      Amt/Area    Amount   Grp   Name
 [min]                                  [ug/mL]
-------|------|----------|----------|----------|--|------------------
  6.998 MM     3.83534e5  3.56254e-7 1.36635e-1    VX

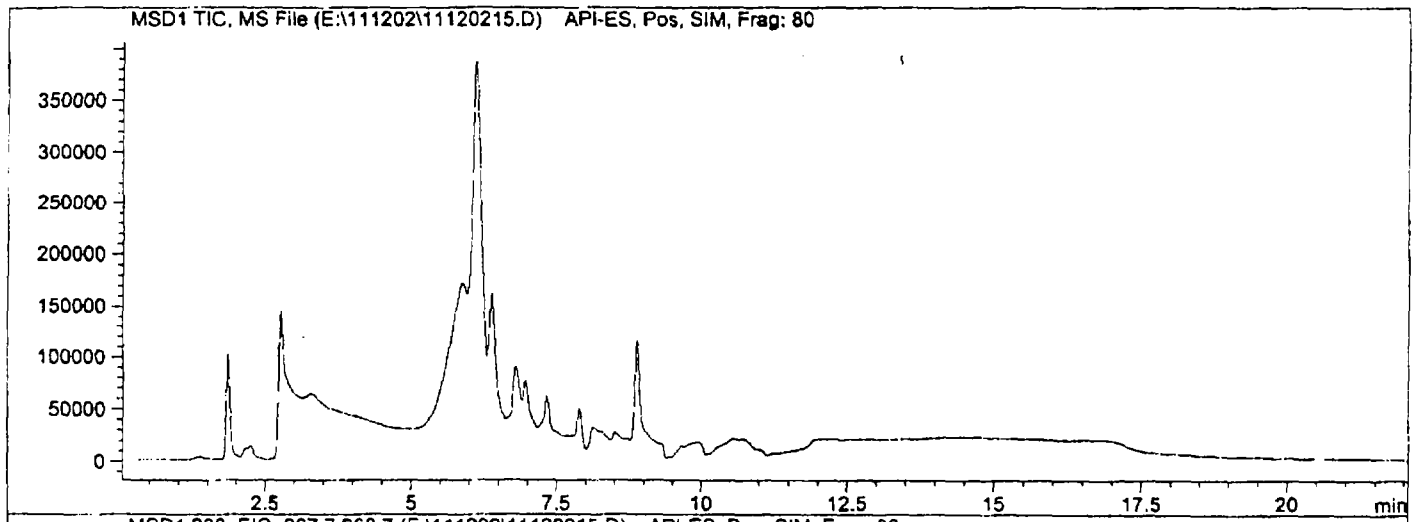
Totals :                               1.36635e-1

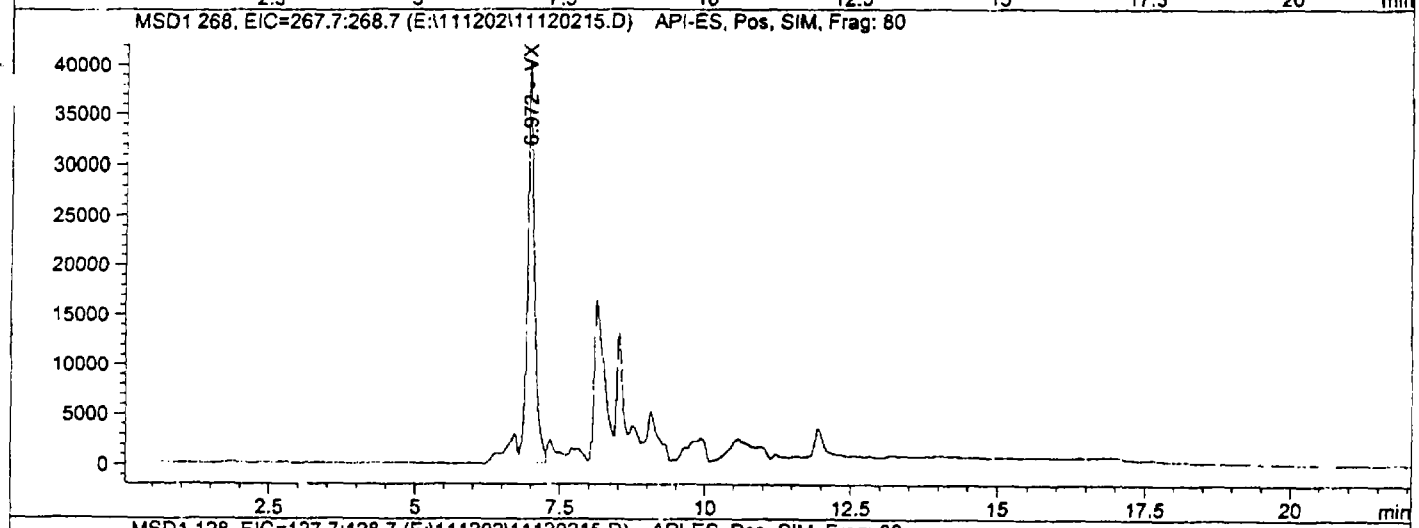

==================================================================
                        *** End of Report ***
```

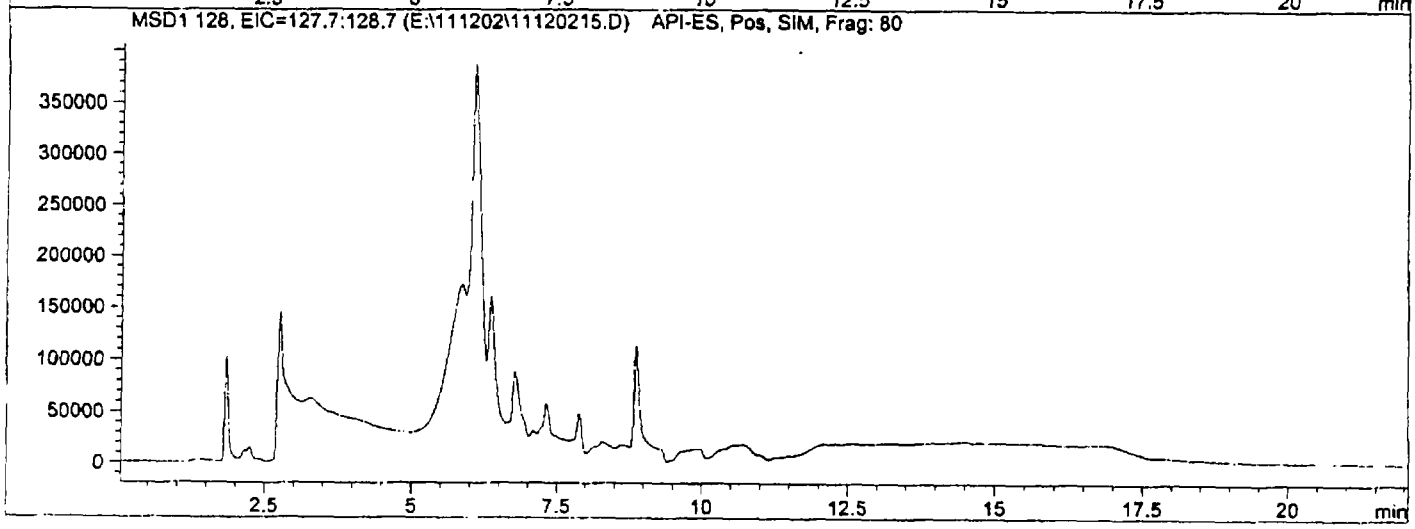
35

Injection Date	: 11/12/2002 10:00:24 PM	Seq. Line	: 14
Sample Name	: R3 4X	Location	: Vial 14
Acq. Operator	: GAA	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 3:19:17 PM by AYY (modified after loading)		

ADP'S







36

```
==================================================================
Injection Date  : 11/12/2002 10:00:24 PM        Seq. Line :  14
Sample Name     : R3 4X                          Location : Vial 14
Acq. Operator   : GAA                                 Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 3:19:17 PM by AYY
                  (modified after loading)
ADP'S
==================================================================

==================================================================
                      External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 3:00:17 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime  Type     Area      Amt/Area    Amount   Grp   Name
 [min]                                  [ug/mL]
-------|------|----------|----------|----------|--|------------------
  6.972 VV     3.35102e5  3.59201e-7 1.20369e-1    VX

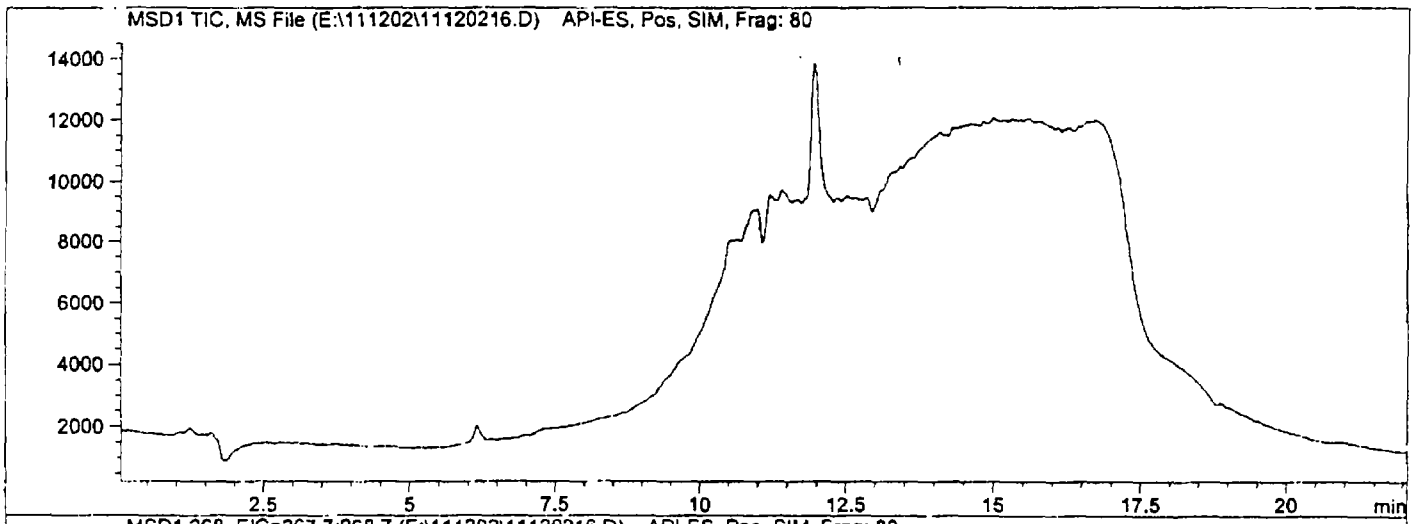
Totals :                               1.20369e-1

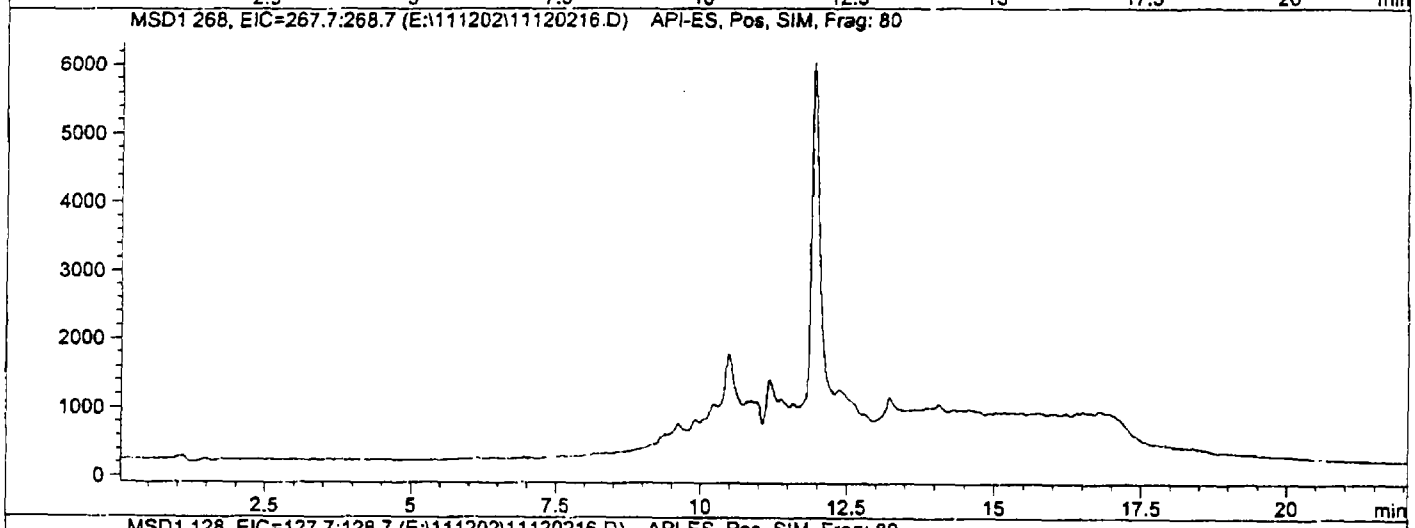

==================================================================
                        *** End of Report ***
```

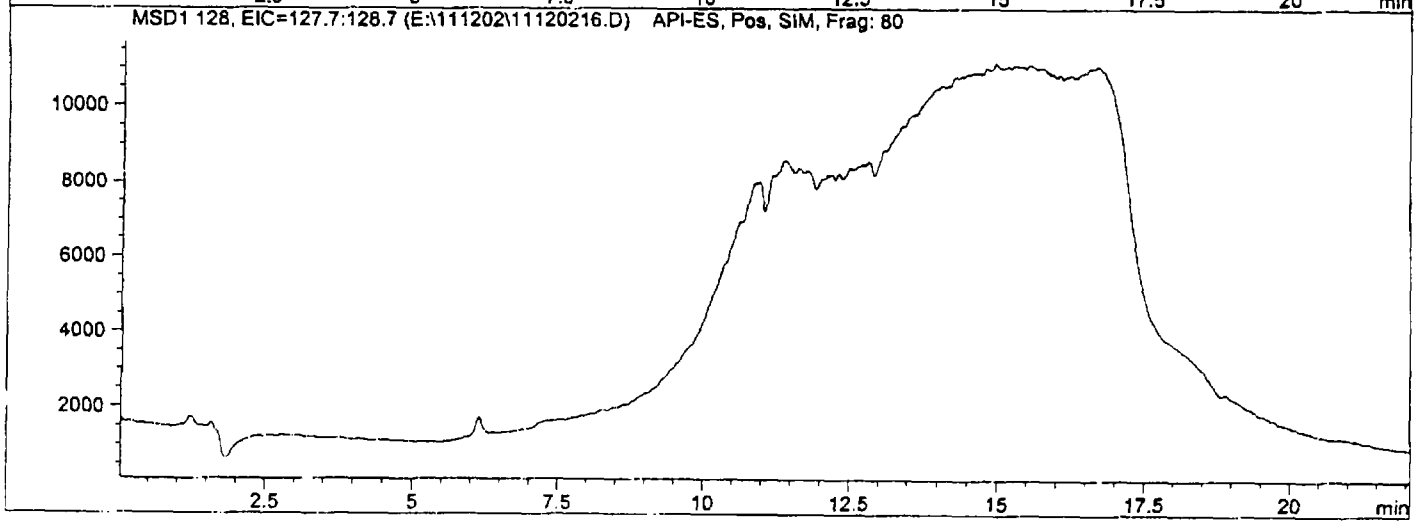
37

Injection Date	: 11/12/2002 10:24:08 PM	Seq. Line	: 15
Sample Name	: WATER BLK2	Location	: Vial 15
Acq. Operator	: GAA	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 3:19:42 PM by AYY (modified after loading)		

ADP'S







38

```
==================================================================
Injection Date  : 11/12/2002 10:24:08 PM        Seq. Line :  15
Sample Name     : WATER BLK2                     Location : Vial 15
Acq. Operator   : GAA                                Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 3:19:42 PM by AYY
                  (modified after loading)
ADP'S
==================================================================

==================================================================
                      External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 3:00:17 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime  Type     Area     Amt/Area    Amount   Grp   Name
 [min]                                [ug/mL]
-------|------|----------|----------|----------|--|------------------
  7.052            -          -          -         VX

Totals :                                0.00000


1 Warnings or Errors :

Warning : Calibrated compound(s) not found

==================================================================
==================================================================
                        Area Percent Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 3:00:17 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: MSD1 TIC, MS File


Signal 2: MSD1 268, EIC=267.7:268.7

Peak RetTime  Type   Width     Area      Area    Name
  #   [min]          [min]                 %
----|-------|------|-------|----------|--------|---------------------
   1   7.052         0.0000    0.00000   0.0000 VX

Totals :                       0.00000   0.0000


Signal 3: MSD1 128, EIC=127.7:128.7
```

39

```
==================================================================
Injection Date  : 11/12/2002 10:24:08 PM        Seq. Line :  15
Sample Name     : WATER BLK2                     Location : Vial 15
Acq. Operator   : GAA                                 Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 3:19:42 PM by AYY
                  (modified after loading)
ADP'S
==================================================================


1 Warnings or Errors :

Warning : Calibrated compound(s) not found

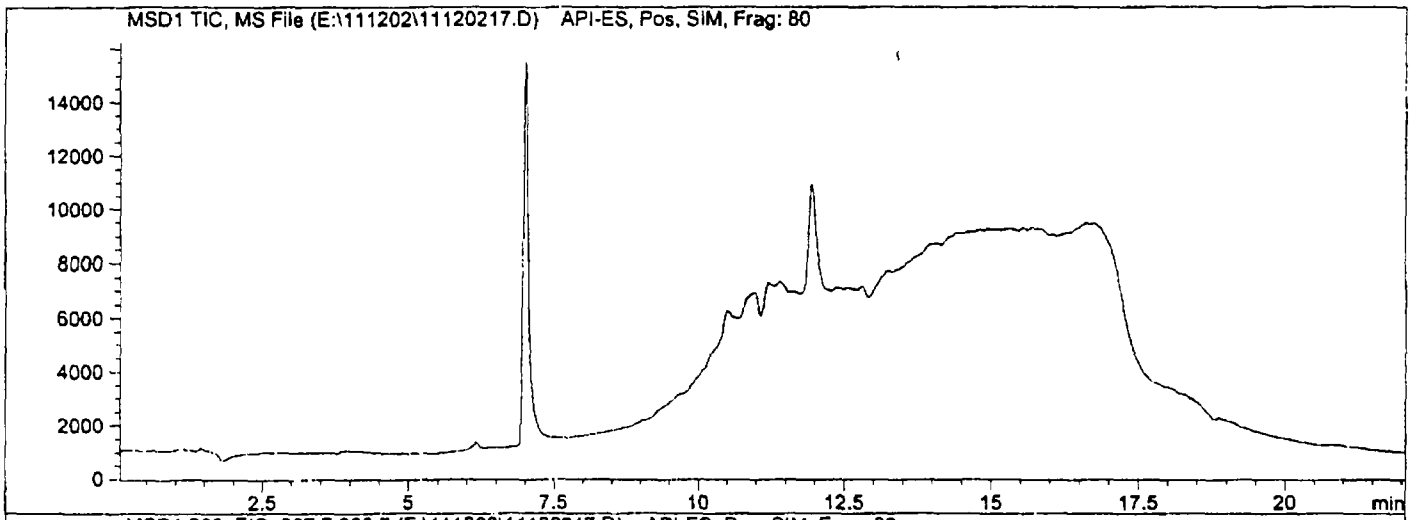
==================================================================
                          *** End of Report ***
```

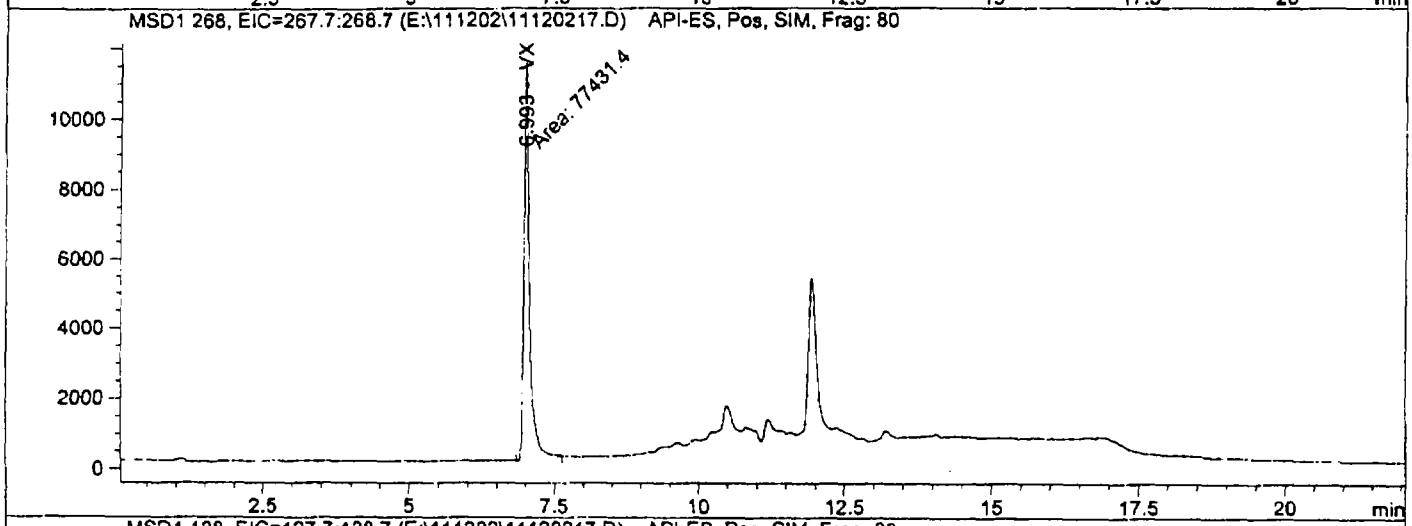
Data File E:\111202\11120217.D Sample Name: CCS 2 0.0312

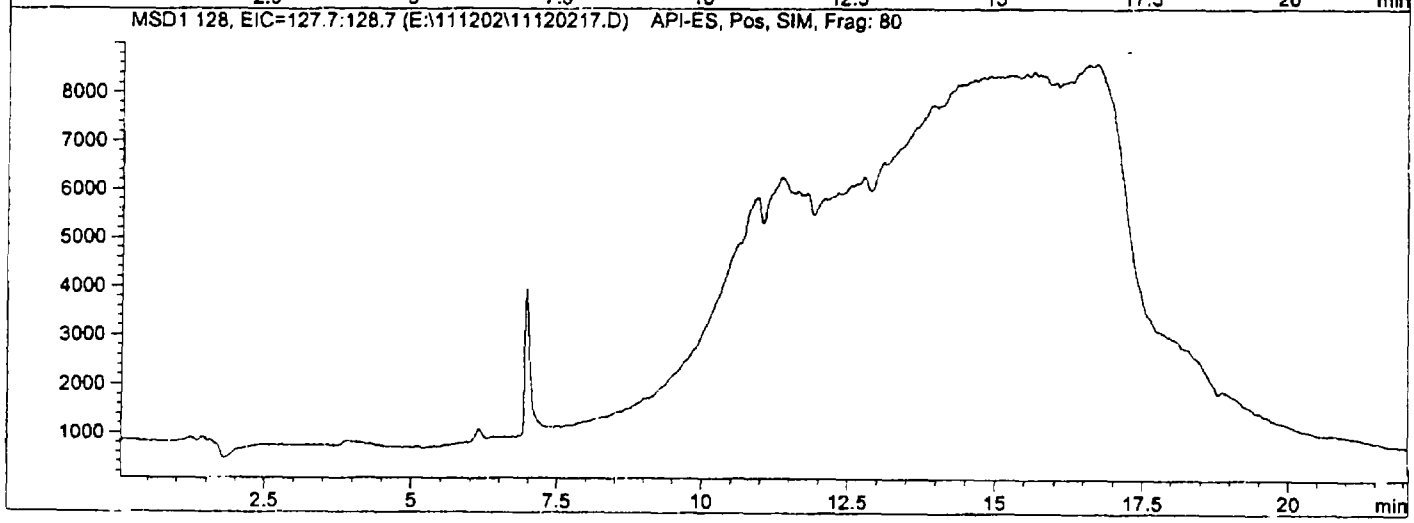
40

Injection Date	: 11/12/2002 10:47:56 PM	Seq. Line	: 16
Sample Name	: CCS 2 0.0312	Location	: Vial 16
Acq. Operator	: GAA	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 20 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_SIM.M		
Last changed	: 11/12/2002 4:20:08 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX_1112.M		
Last changed	: 1/2/2003 3:20:12 PM by AYY (modified after loading)		

ADP'S







41

```
==================================================================
Injection Date  : 11/12/2002 10:47:56 PM        Seq. Line :  16
Sample Name     : CCS 2 0.0312                   Location : Vial 16
Acq. Operator   : GAA                                 Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_SIM.M
Last changed    : 11/12/2002 4:20:08 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX_1112.M
Last changed    : 1/2/2003 3:20:12 PM by AYY
                  (modified after loading)
ADP'S
==================================================================

==================================================================
                      External Standard Report
==================================================================

Sorted By              :      Signal
Calib. Data Modified   :      1/2/2003 3:00:17 PM
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime  Type     Area      Amt/Area    Amount   Grp   Name
 [min]                                  [ug/mL]
-------|------|----------|----------|----------|--|-----------------
  6.993 MM     7.74314e4  4.36856e-7 3.38264e-2    VX

Totals :                               3.38264e-2


==================================================================
                        *** End of Report ***
```

APPENDIX D

NMR DATA

NMR Analysis of Acetone Extract of Tray 153 Ash

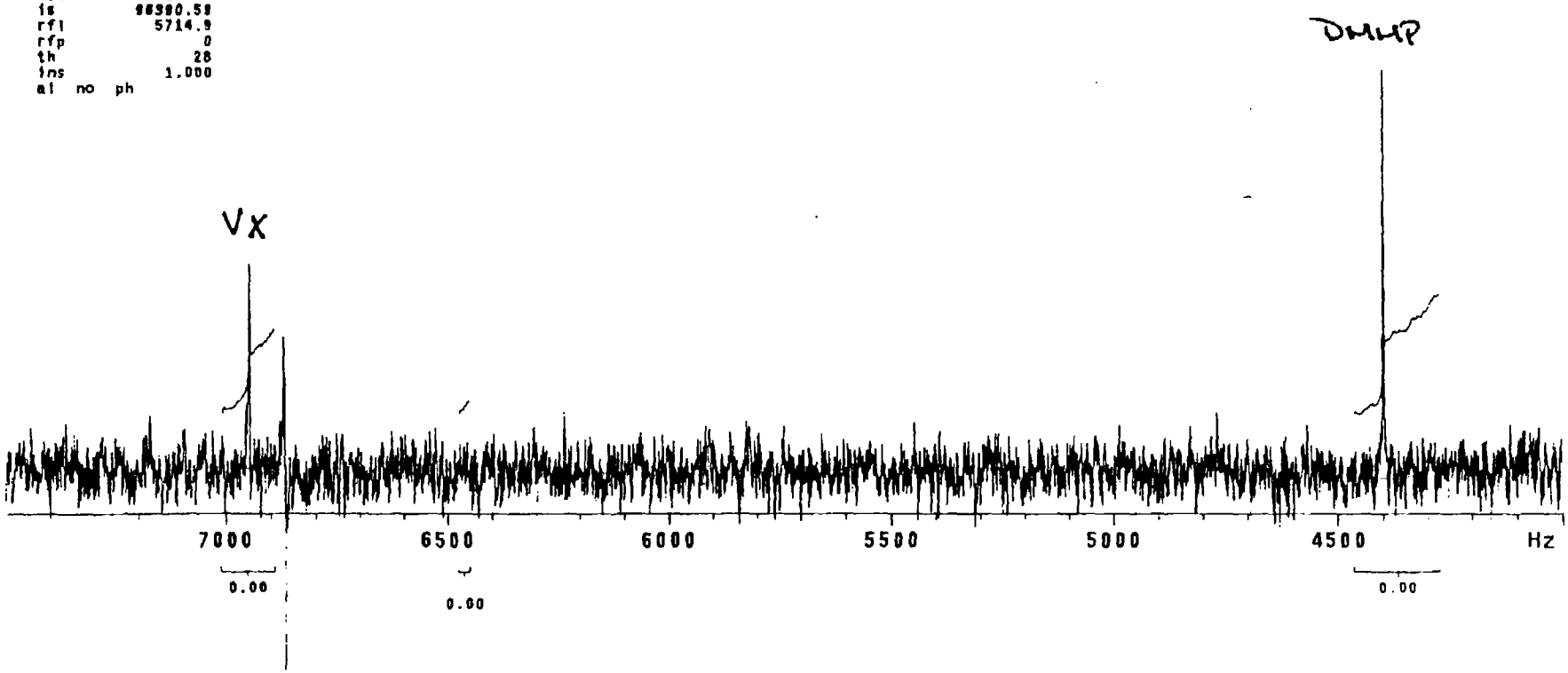
Sample	Peak Ht (mm)	Spike Conc (ug/mL)	Spike Conc. mmole	Spike Response mm/mmole	Sample Conc. mmole	Sample Conc. (ug/mL)	Final Vol. (mL)	Wt. Ash (g)	Sample Conc. mg/kg
R1	19	0			0.07	17.96	0.5	15.1	0.59
DMMP std.	61.5	28	0.23	272.58					
VX std	31	29.3	0.11	282.91					

VX and DMMP std.

exp5 s2pul

SAMPLE		DEC. & VT	
date	Nov 19 2002	dfrq	299.948
solvent	CDCl3	dn	H1
file /export/home/~		dpwr	not used
Marty/VX Ash Nov. ~		dof	0
02/VX_and_DMMP_std		dm	nny
ACQUISITION		dmm	w
sfrq	121.430	dmf	6094
tn	P31	dseq	
at	1.600	dres	1.0
np	96000	homo	n
sw	30007.5	temp	25.0
fb	16500	PROCESSING	
bs	16	lb	1.00
pw	11.7	wtfile	
d1	3.000	proc	ft
tof	9500.0	fn	not used
nt	14000	math	f
ct	14000		
alock	n	werr	
gain	not used	wexp	
FLAGS		wbs	
i1	n	wnt	
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	9899.6		
wp	9498.6		
vs	5000		
sc	0		
wc	240		
hzmm	14.58		
is	96390.59		
rfl	5714.9		
rfp	0		
th	28		
ins	1.000		

al no ph

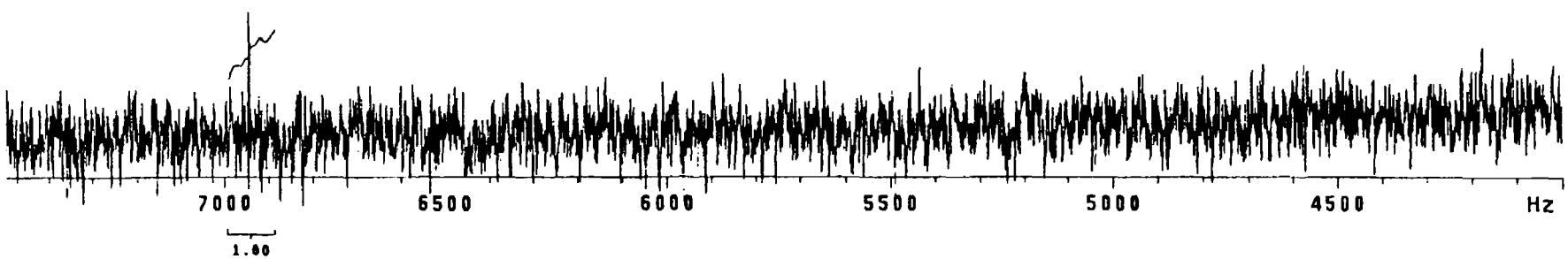


2

Ash Acetone Extract
11-14-2

exp4 s2pul

SAMPLE		DEC. & VT	
date	Nov 14 2002	dfrq	299.948
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	not used
Marty/VX Ash Nov. ~		dof	0
02/P_VX_Ash_Aceton~		dm	nny
e_Ext_11-14-2		dmm	w
ACQUISITION		dmf	6084
sfrq	121.430	dseq	
tn	P31	dres	1.0
at	1.500	homo	n
np	86000	temp	25.0
sw	30007.5	PROCESSING	
fb	16500	lb	1.00
bs	16	wtfile	
pw	11.7	proc	ft
d1	3.000	fn	not used
tof	9500.0	math	f
nt	14000		
ct	14000	werr	
alock	n	wexp	
gain	not used	wbs	
FLAGS		wnt	
fl	n		
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	3399.9		
wp	3499.6		
vs	5000		
sc	0		
wc	240		
hzmm	14.58		
is	100000.00		
rfl	12585.9		
rfp	6946.9		
th	42		
ins	1.000		
al no ph			



Analysis of VX in Residue Sample from MPF DAL Tray Number 153

Subcontract 99000200
Prime Contract DAAA-09-96-C-0081
SwRI Project 05935.01.516

Prepared by

Joseph H. Brewer
Michael G. MacNaughton, Ph.D.

Prepared for

Washington Demilitarization Company
2850 Pa'a – Suite 100
Honolulu, Hawaii 96819

October 28, 2002

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SAN ANTONIO, TEXAS 78228-0510

Analysis of VX in Residue Sample from MPF DAL Tray Number 153

Subcontract 99000200
Prime Contract DAAA-09-96-C-0081
SwRI Project 05935.01.516

Prepared by

Joseph H. Brewer
Michael G. MacNaughton, Ph.D.

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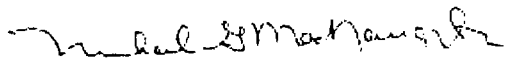
October 28, 2002

APPROVED:



Michael G. MacNaughton, Ph.D., P.E.
Vice President
Chemistry and Chemical Engineering Division

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F. NMR Data

1. PURPOSE

This report describes the approach and results of multilaboratory analysis of ash residue from a tray processed through the Johnston Atoll Chemical Agent Demilitarization System (JACADS) metal parts furnace (MPF). The analyses were performed to definitively determine if there was chemical agent VX in the ash using more specific analytical methods than available in the JACADS laboratory. An additional objective, if the sample was confirmed to be contaminated with VX, was to confirm that the concentration was above the waste control limit (WCL) of 20 µg/kg as determined at JACADS by the G-analog method.

2. BACKGROUND

2.1 Source of Ash Samples

Tray 153 was loaded with approximately 52 pounds of SDS sludge for destruction in the JACADS MPF. The sludge was not loaded in accordance with the Program Director's Operational Memorandum (PDOM) that required that the sludge be spread to a depth no greater than 1.5 inches. After 2 hours in the MPF, the tray was moved to the discharge airlock (DAL) where it was allowed to cool for 22 minutes. During the 22 minutes, the ACAMS monitored the exhaust air for GB, HD, and VX no indication of contamination was found. Subsequently upon exiting the MFP DAL, the ACAMS alarmed for HD and GB. After 19 minutes, the tray was returned to the DAL where the ACAMS indicated all three agents; HD, GB, and VX were above the time-weighted average (TWA). DAAMS tubes did not confirm the presence of GB or HD; however, VX was confirmed by DAAMS. After 5.3 hours in the DAL, the ACAMS readings were reduced to below the limit of quantification (LOQ) for all three agents.

The tray was returned through the MPF into the lower munitions corridor where a sample was taken for analysis. This sample indicated VX concentrations in the sludge ash of 45 to 80 times the 20 µg VX/kg ash WCL. The sludge was visually observed and sampled again 5 days later. Twelve samples were collected from different locations and depths in the ash pile. The pile itself was approximately 9 inches wide and 7 inches high. After samples had been taken, the remaining residue was removed from the tray and placed in a drum liner and then into a drum. The remaining residue weighed approximately 16 pounds as compared to the 52.5 pounds that was reported for the sludge. All samples were positive for VX with concentrations varying from 0.12 WCL to 186 WCL. A wand on an ACAMS sampling line was held close to the surface of the ash for a whole sampling cycle. Even though the surface was stirred during the sampling, only a 0.2 TWA reading was recorded. The samples were analyzed on a GC/FPD/MS using the DAAMS technique and derivitatization by AgF to ethyl methylphosphonofluoridate (G-analog). The peaks identified as VX had the correct retention time (RT). The MS 81 and 99 ions, representative of the G-analog, had the same RTs as the FPD, and they were in the correct ion ratios. This met all criteria for declaring the samples as contaminated with VX above the WCL.

Although the ash displayed evidence of incomplete combustion due the improper loading (unburned burlap and plastic), it was still surprising that VX in the sludge survived the 2 hours in the MPF. Previous studies by IITRI have identified the potential for false positives with high concentration of ethyl methylphosphonic acid (EMPA), diisopropylaminoethanthiol, water and the stabilizer DCCDI (Nerve Agent VX Irreversibility Test Report, PMCD, January 1997). In addition NMR studies at ECBC have also documented the reaction between VX breakdown products to form VX after chloroform extractions (Beaudy, W.T. and Szafraniec, L.L., NMR Characterization of pH Adjusted VX/Caustic and MeCl2 Extracts, July 1996)

Additional analysis was performed in the JACADS laboratory to determine if the peaks observed on FPD and MS were due to interference from another compound. To see if the peak was due to a volatile interference that co-eluted with VX, the samples were directly injected onto the DAAMS without the AgF. There was no FPD peak in the VX window using this technique. Since the technique used at JACADS to monitor and confirm VX uses AgF to convert the VX to the more volatile G-analog, it was suggested that a direct VX method be used. The JACADS laboratory uses direct liquid injection to verify VX standards. A chloroform extract of the sample was injected into a GC/FPD and a FPD peak was observed in the VX window. It must be noted that with a 4minute run time, the chromatographic separation with this method is poor and interference could still co-elute at the VX RT. The liquid solvent extracts were then injected onto a GC/MS that had been converted for direct liquid injection. These analyses were inconclusive due to interferences in the retention time window. Since the GC/FPD/MS that had been converted for direct liquid injection was required to meet the limiting conditions of operation (LCO) for plant operations, it was decided to ship the samples off-site for more definitive analysis.

Southwest Research Institute™ (SwRI®) located in San Antonio, Texas and ITT Research Institute (IITRI®), located in Chicago, Illinois. Idaho National Engineering and Environmental Laboratory (INEEL) were tasked to perform these analyses. This report includes results from the SwRI and IITRI laboratories. Conflicts in the workload at Dugway Proving grounds prevented the expedient analysis of the ash sample by INEEL preventing the inclusion of those results in this report. They will be added as a supplement when they are available.

2.2 Sample Handling

A sample of the ash collected on 13 August 2002, one day after the MPF event was well mixed in the JACADS laboratory. This sample was spilt into three subsamples for analysis by multiple laboratories. Analysis of the sample by the JACADS laboratory using the G-analog method gave VX concentrations of 210 WCL (4.2 μg/g). The samples were shipped from JACADS to the SwRI, IITRI, and Dugway Proving Grounds for the Idaho National Engineering and Environmental Laboratory (INEEL) under contract the PM Non-Stockpile. The samples were sent to the surety laboratories by signature secure commercial air on 3 October 2002.

Chain-of-custody was maintained from the original sampling in the lower munitions corridor through shipment to the participating laboratories. Shipment to the laboratories was under chain-of-custody and compliant with DOT and USA procedures. The samples are solids that have no headspace VX concentrations (3X). The highest extractable concentration was found to be 186 WCL or 3.6 mg/kg, well below the 1000 mg/kg surety threshold. The samples were shipped directly to the laboratories from JACADS. Fifty grams of ash was sent to SwRI®, 50 g to IITRI® and 10 g to INEEL at the Dugway Proving Grounds.

3. APPROACH

Multiple laboratories and four different analytical methods were used to obtain a higher level of confidence in the results. The analyses were conducted by SwRI using liquid chromatography/mass spectroscopy (LC/MS) and gas chromatography/electron impact/mass spectroscopy (GC/IE/MS). IITRI used gas chromatography/ion trap/mass spectroscopy (GC/IT/MS) chemical ionization (CI) and INEEL used direct ion trap secondary ion mass spectroscopy.

3.1 Analytical Procedures

Each analytical laboratory used in-house procedures to analyze the Tray 153 ash sample. The focus was on the confirmation of VX in the sample, however, it was attempted to provide a semiquantification of any VX found, and, at the request of the Environmental Protection Agency, to provide a list of tentatively identified compounds (TICs).

The SwRI analytical procedures for the G-analog GC/FPD method and direct VX methods using GC/MS, and LC/MS are provided in Appendix A. The reports of results from IITRI laboratories with procedures for GC/IT/MS assay are provided in Appendix B.

3.1.1 SwRI Laboratory Method Summary

For GC/MS analysis, 2 grams of ash was extracted with 12 mL of 90% dichloromethane (DCM) and 10% isopropyl alcohol (IPA). For the G-analog method, the DCM/IPA extract was injected directly onto an AgF CPA attached to a DAAMS tube. The DAAMS tube was then thermally desorbed in a specially configured GC/FPD. For GC/MS analysis, 0.1 mL of diisopropylaminoethanol was added to the extract to improve recovery and "tune" the peak shape prior to direct injection into the instrument. The instrument parameters and calibration requirements are included in the method provided in Appendix A.

Initially, the GC/MS extracts run full scan displayed significant background response and the only associated ion identified was 114 m/z. Understanding the presence of the 114 ion can be attributed to a number of compounds including VX decomposition products, another sequence was run using a 2-fold dilution to reduce background response. This dilution did not reduce background interference again limiting results to only the 114 m/z ion.

To eliminate background interference, the DCM/IPA extract was exchanged into water, filtered, pH adjusted, and back-extracted with methylene chloride. This cleanup step reduced the background interference for GC/MS and provided data for all VX ions. The results presented in Table 1 are from SIM analysis using ions 139, 167, 224, and 252. The 167 and 139 m/z ions were used for quantification. The 114 and 127 m/z ions were not monitored in the cleaned extract. There was a slight positive bias noted in the response for ion 224 m/z. Quality control samples included a laboratory control spike and blank that indicated 80% recovery for each of the four ions and no evidence of blank contribution was present.

For LC/MS, 12 mL of the 90% DCM/10% IPA extract was blown down to dryness and brought up in 4 mL of the water. Analytical results from LC/MS analyses were obtained using the 268 m/z, primary ion. Both the primary ion (268) and secondary ion (128) were present in the sample. Quality control samples included laboratory control spikes and blanks. The laboratory control spike indicated 99% recovery and no background contribution was present.

3.1.2 IITRI Laboratory Method Summary

At IITRI, 1- and 5-gram samples of ash were extracted using 20 mL of 90% DCM and 10% IPA. The pH was adjusted to approximately nine using 0.5 mL of triethylamine. The sample was sonicated for 3 minutes and the extract supernatant transferred to a centrifuge tube. The extraction was repeated with a second 20 ml of DCM/IPA. The extracts were centrifuged and then blown down to 5 mL using nitrogen. To enhance recovery 800 µg of diethylamminoethanthiol (DEAET) was added to 1 mL of the extract prior to direct injection into an ion trap GC/MS operated in the chemical ionization-MS/MS. The IITRI report in Appendix B provides a full description of the analytical approach.

3.2 Quality Control

Quality control samples included matrix spikes, blanks, and laboratory control spikes. These were assayed at varied times within the analytical sequence to ensure spike recovery was available both pre- and post-sample analyses. Criteria for MS and MSD recovery is 40% to 160% with duplicate agreement of at least 50%. This broad criterion is needed to address the wide range of matrix effects often encountered when running VX. It is important to note that laboratory quality control requirements are typical of EPA methods with laboratory control spike recovery limits of ±25% and blank values below lowest calibration standards. Tuning, calibration, and continuing calibration requirements are included in each of the methods.

4. RESULTS

4.1 VX Results

The analytical data packages for the work performed at SwRI and IITRI are included in Appendices C and D, respectively. The GC/MS, LC/MS, and GC/IT/MS data packages are provided in the appendices. Data from the GC/FPD analyses conducted at JACADS is not included in this report. Table 1 summarizes the sample results from the three confirmation methods.

Table 1. Analytical Results from Analysis of Tray 153 Samples

Laboratory and Method	Average Sample Concentration (mg/kg)	Relative Standard Deviation (%)	Matrix Spike Recovery (%)	Matrix Spike Duplicate Recovery (%)
JACADS (Derivative)	4.2	14	88	83
IITRI GC/IT/MS (Undiluted)	4.1	4.8*	110	120
IITRI GC/IT/MS (1:10)	3.5	11	NA	NA
SwRI GC/MS (1:10)	2.3	16	88	63
SwRI-LC/MS (Undiluted)	2.4	9.7	86	94

* – Duplicate analyses expressed as relative percent difference
NA – Not Analyzed

All three methods confirmed VX in the ash sample in agreement with the GC/FPD/MS results in the JACADS laboratory. The concentration varied from 2.3μg/g to 4.1 μg/g, again consistent witht the results from the JACADS laboratory using the G-analog method.

4.2 Tentatively Identified Compounds

To investigate decomposition products in the sample, GC/MS full scan analytical runs were searched to identify any unknown compounds. These TICs include the major components (up to 20) within the

sample. Primary focus was on the chromatographic region surrounding the retention time of VX but the entire run period was investigated. These investigations were conducted prior to using the DEAET or other enhancing agent to prevent its contribution to the unknown compound list. Identified compounds and spectral information are included in Appendix E.

All of the TICs presented in the appendix are estimated to be above 20 ppm. Area counts are provided in the appendix for each of the compounds investigated. Results indicate the presence of various amines and phosphonothioic acids that would be expected in waste from a VX waste.

4.3 Nuclear Magnetic Resonance

Nuclear magnetic resonance (NMR) spectra were taken to further investigate the presence of VX in the Tray 153 ash sample. Four grams of ash was extracted with 20 mL 90% DCM and 10% IPA and concentrated to dryness. This concentrated extract was diluted to 1000 µL using deuterated chloroform solvent. The sample was transferred to an NMR sample tube for analysis. The NMR response represents a 18-fold concentration of the GC/MS extract.

Results are presented in Appendix F. Three NMR spectrum are provided: a 200 µg/mL VX reference standard, unspiked sample, and the same sample spiked with 50 µg/mL VX. The appendix includes instrument operating conditions on each of the plots.

5. CONCLUSIONS

The purpose of this analytical effort was to confirm or refute the presence of VX in the ash from the sludge that had been improperly loaded in Tray 153, as determined by the G-analog methods at JACADS. Based on the results from GC/MS, LC/MS at SwRI and GC/IT/MS at IITRI, there is VX in the ash at approximately the same concentration as found by G-analog GC/FPD analysis. This result is consistent with the ACAMS alarms and the confirmed DAAMS tubes that were collected after the tray had been returned to the DAL. The results do not support the hypothesis that the positive responses were caused by an interferent.

APPENDIX A

SwRI ANALYTICAL PROCEDURES

ANALYSIS SOLID SAMPLES FOR VX

DRAFT

1.0 SCOPE

1.1 This procedure describes the preparation and analysis of solid samples for VX.

1.2 This procedure describes several methods which include DAAMS GC/FPD, liquid injection GC/MS and liquid injection LC/MS.

1.3 From guidelines described in the **Surety Chemical Hygiene Plan,** this procedure has been assigned a **risk index of 14** from a possible range of 5(not authorized) to 20 (authorized without review). The category, operational hazard was determined to be **marginal** because of the solvent waste generation and use of dilute agent stock. The category, operational frequency was determined to be **remote**. See Safety procedures Section 7.0 and the **Surety Chemical Hygiene Plan** for additional information.

2.0 PURPOSE

This document provides the description for extraction and analysis of solid samples for VX.

3.0 RESPONSIBILITIES

3.1 It is the responsibility of the Chemical Agent Program Manager (CAPM) to assure that this procedure has been evaluated and is in compliance with operational plans.

3.2 It is the responsibility of all personnel working in the Chemical Surety Lab to comply with this procedure.

4.0 REFERENCES

4.1 Agent handling, PPE, and safety procedures should be followed as identified in the **Surety Chemical Hygiene Plan** (CHP-004) for agent operations.

4.2 See TAP-01-0500-034 for CSM Protective Glove Policy.

4.3 See TAP 01-0500-035 for Personnel Decontamination Stations.

5.0 PROCEDURE

5.1 Materials
5.1.01 Protective Equipment.

5.1.02 Dichloromethane, Isopropyl alcohol, Sodium hydroxide, Sodium chloride, and Silver fluoride conversion pads.

5.1.03 Pipettes, micro-spatulas, test tubes or 3 dram vials with Teflon lined caps, auto sampler vials, appropriate syringes, glass wool, pH paper etc. (see procedure)

5.1.04 Vortex Mixer and/or Centrifuge.

5.1.05 Vacuum Pump.

5.1.06 Gas Chromatograph equipped with a flame photometric detector (GC/FPD), a mass spectrometer (GC/MS), and/or auto sampler.

5.1.07 Liquid Chromatograph equipped with a mass spectrometer and an auto sampler.

5.1.08 3mm DAAMS and 8mm DAAMS Chromosorb-106 Tubes.

5.2 EXTRACTION OF SOLID SAMPLES FOR VX.

5.2.01 Pulverize the solid sample into a uniform powder with a mortar and pestle, if necessary.

5.2.02 Weigh 2.00 $\pm$ 0.05 g of solid into a 3dram centrifuge vial with Teflon-lined screw cap. Record weight into a controlled notebook.

5.2.03 Spike the MS, MSD or blank spike sample with agent. Record spike amounts into controlled notebook. The VX spike should be about ten times the MDL or three to five times the sample concentration of VX.

5.2.04 Add 4.0 mL of 10% 2-propanol in dichloromethane to the solid sample.

5.2.05 Put the lid on the vial.

5.2.06 Shake the sample vigorously for about 60 seconds.

5.2.07 Centrifuge the sample for 1minute at approx. 2000 rpm.

5.2.08 Decant the solvent into a disposable 10cc syringe connected to 0.45um Gelman Nylon acrodisk. Collect the filtered extract into a 3 dram vial.

5.2.09 Repeat steps 5.2.04 through 5.2.08, two more times combining the filtered

extracts.

5.2.10 Add solvent to the combined extracts for each sample to bring the samples to an equivalent volume and record the final volume for each sample.

5.2.11 For GC/FPD analysis, proceed to Step 5.5.02. For GC/MS analysis, proceed to Step 5.5.03. For LC/MS solvent exchange and GC/MS cleanup, proceed to Step 5.3.

5.3 SOLVENT EXCHANGE EXTRACTS FROM IPA/DCM TO WATER

5.3.01 Place an aliquot of the extract, step 5.2.11, into a 1dram vial. Suggest 2.0mL.

5.3.02 Concentrate the extract to about 100uL by gently blowing a stream of Nitrogen into the vial.

5.3.03 Rinse the sides of the vial and the Nitrogen tip with about 200uL of IPA and continue to concentrate the sample to about 100uL.

5.3.04 Add an additional 200uL of IPA.

5.3.05 Bring the sample to a final volume of 4.0mL with HPLC grade water.

5.3.06 For LC/MS analysis, proceed to step 5.5.04. For GC/MS cleanup, proceed to Step 5.4.

5.4 GC/MS CLEANUP

5.4.01 Place an aliquot of the extract, step 5.3.05, into a 1dram vial. Suggest 2.0mL.

5.4.02 Filter the aqueous aliquot using a disposable 10cc syringe connected to 0.45um Gelman Nylon acrodisk. Collect the filtered extract into a 3 dram vial.

5.4.03 Adjust the pH of the aqueous extract to approximately pH 10.

5.4.04 Extract the aqueous aliquot with 1.5mL of 10% IPA/DCM and a micro spatula full of sodium chloride.

5.4.05 Remove the bottom layer (organic layer) for analysis.

5.4.06 Proceed to GC/MS sample analysis, step 5.5.03.

5.5 ANALYTICAL PROCEDURES

5.5.01 The analytical analysis can be performed by any one of the following methods: DAAMS GC/FPD, liquid injection GC/MS and liquid injection LC/MS.

5.5.02 DAAMS (GC/FPD) Method

5.5.02.1 Fill several or 8mm DAAMS tube with densely packed silanized glass wool at the spiking end (short end).

5.5.02.2 An Initial calibration curve will be run and evaluated before samples can be injected. All standards will be spiked through an AgF pad located on the short end (end filled with glass wool) of 8-mm DAAMS tubes on a vacuum manifold with a vacuum of 200-250mL/Min. Steps 5.5.02.4 to 5.4.02.9 will be followed for all standards.

5.5.02.3 For sample injections, connect long side of an 8-mm chromosorb-106 DAAMS tube to a vacuum manifold with a suction of about 200-250 mL/min. Spike 50 uL of the organic layer from the extract, onto the AgF conversion pad connected to the short end side of the DAAMS tube. Continue airflow for 5 minutes after the DAAMS tube has been spiked.

5.5.02.4 The 8mm DAAMS tube is removed from the assembly and connected by swadgelok union to a 3-mm DAAMS tube.

5.5.02.5 The contents of the 8mm DAAMS tube is transferred to the 3-mm tube by putting the 8mm DAAMS tube through an Aluminum heating block set at 220° C and connecting the 8mm DAAMS tube to a Helium purge line set at ~200-250 mL/min continuous flow for 3 minutes.

5.5.02.6 The 3-mm tube is removed from the transfer assembly.

5.5.02.7 For DAAMS injection, the 3-mm tube will be connected to the carrier gas line on the GC. The 3-mm tube is then inserted into a modified injection port and the start button is pressed on the GC.

5.5.02.8 Suggested configurations. Use a DB-624 30 meter column and set the injector to 220 C.

Oven Program:
- VX: 90 C | 2 min | 10C/min| 100 C | 40 C/min | 220 C.

Calibration Standards:
- VX: 0.1, 0.2, 0.5, 1.0, and 1.5ng.

FPD Detector:
- VX uses a Phosphorus filter.

5.5.03 Liquid Injection (GC/MS)

5.5.03.1 GC/MS samples are diluted at this point, should the need arise.

5.5.03.2 Withdraw 1000 uL of extract for each sample or standard and place in an appropriately labeled auto sampler vial.

5.5.03.3 Spike internal standard into each vial at the same concentration. Suggest using Hexachlorobenzene at a concentration of 0.1ng/uL in all standards and samples.

5.5.03.4 Add 1uL of Diisopropylamino ethanol

5.5.03.5 Cap the auto sampler vial and place, in sequence, on the auto sampler tray.

5.5.03.6 Setup the sequence in the software according to the samples and standards on the tray.

5.5.03.7 Press start on the auto sampler.

5.5.03.8 Suggested configurations. Use a DB-1701 30 meter column and set the injector to 220 C.

Oven Program:
- 60 C | 2 min | 15 C/min | 200 C | 5 C/min | 260 C | 5min.

Calibration Standards:
- 0.10, 0.20, 0.50, 1.0, and 1.5ng/uL.

MS (SIM) Detector:
- VX ions: 114, 127, 139, 167, 224, and 252 m/z.
- Hexachlorobenzene: Primary ion 284 m/z, Secondary ion 286 m/z.

- Ions may be added or dropped per sequence in order to reduce interferences based on the sample matrix. If ions are dropped, the instrument needs to be recalibrated.

5.5.04 Liquid Injection (LC/MS)

5.5.04.1 LC/MS samples are diluted at this point, should the need arise.

5.5.04.2 Withdraw 200uL of extract for each sample or standard and place in an appropriately labeled auto sampler vial.

5.5.04.3 Spike 18uL of 30% formic acid into each vial.

5.5.04.4 Cap the auto sampler vial and place in sequence on the auto sampler tray.

5.5.04.5 Setup the sequence in the software according to the samples and standards on the tray. Injection volume must be the same for samples and calibration standards.

5.5.04.6 Press start on the computer.

5.5.04.7 Suggested configurations. Use a C-18 column 7.5cm X 4.6mm.

LC Program:
- Solvent A: HPLC grade water with 0.1% formic acid.
- Solvent B: HPLC grade methanol with 0.1% formic acid.
- Gradient Program:
  - 0.0 min | 0.70 mL/min | 96% Solv. A | 4% Solv. B
  - 3.0 min | 0.70 mL/min | 96% Solv. A | 4% Solv. B
  - 6.0 min | 0.70 mL/min | 0% Solv. A | 100% Solv. B
  - 16.0 min | 0.70 mL/min | 0% Solv. A | 100% Solv. B
  - 18.0 min | 0.70 mL/min | 96% Solv. A | 4% Solv. B
  - 23.0 min | 0.70 mL/min | 96% Solv. A | 4% Solv. B

Calibration Standards:
- 0.063, 0.125, 0.250, 0.500, and 1.00 ng/uL.

Spray Chamber:
- Set gas temp to 350C.
- Drying Gas to 13.0 L/min.
- Nebulizer pressure set to 40psig.

- Capillary voltage to 4000 V (positive mode).

MS (SIM) Detector:

- VX ions: Primary ion 268 m/z, Secondary ion 128 m/z.

5.6 ACCEPTANCE CRITERIA

5.6.01 One of several calibration types may be used; however, it must be employed throughout the whole sample set for that particular agent.

5.6.01.1 Linear Regression – A minimum of five points must be run for this type of calibration. The five point calibration curve is deemed valid if the calculated concentration from the regression curve for all target compounds are $\leq$25% across the working range of the method, with the exception of $\leq$35% at the lowest point on the calibration and the R^2 for the regression is $\geq$0.9900.

5.6.01.2 Quadratic Regression - A minimum of six points must be run for this type of calibration. The five point calibration curve is deemed valid if the calculated concentration from the regression curve for all target compounds are $\leq$25% across the working range of the method, with the exception of $\leq$35% at the lowest point on the calibration and the R^2 for the regression is $\geq$0.9900. This calibration is used only for HD on the FPD.

5.6.01.3 Response Factors – A minimum of five points must be run for this type of calibration. Sample concentrations are calculated from the mean response factor. The five point calibration is deemed valid if the calculated % Relative Standard Deviation (%RSD) is less than or equal to 20%.

5.6.02 Continuing calibration standards must be run at the beginning and end of each sample sequence to verify initial calibration values. Only 20 samples can be run between continuing calibration standards. The continuing spike level should be approximately the same as that of the second highest spike level for the initial calibration.

5.6.03 A continuing calibration standard is deemed valid if the calculated concentration value for each agent is within ± 25 % difference of the spiked amount and the peak retention agrees within ± 5% the retention time window established by the initial calibration. Note, if a response factor calibration is employed, the continuing calibration standard is deemed valid if the continuing response factor is within ± 25 % difference

of the mean response factor from the calibration curve and the peak retention agrees within ± 5% the retention time window established by the initial calibration.

5.6.04 All blanks must be free of agent.

5.6.05 All blank spike, matrix spike, and matrix spike duplicates recoveries must be within QC limits of 60 – 140%.

5.6.06 The RPD between matrix spike and matrix spike duplicate must be within the QC limit of 25%.

5.6.07 Agent peak criteria.

5.6.07.1 Potential Agent peak

5.6.07.1.1 All peaks within agent retention time window will be considered as a potential agent hit. The agent retention time window is ±5% of the average agent retention time found from the two continuing calibration standards (CCS) that bracket the sample in question.

5.6.07.1.2 All potential agent peaks must be above the MDL or the lowest point of the calibration for that method.

5.6.07.2 Confirmation of Agent peaks

5.6.07.2.1 Analysis by GC/MS can be confirmed by the ion traces from the primary and secondary ions. Re-analysis using an alternate column is not required.

- The ion traces must line up within ± 2 scan units, and
- The ratio of the two ions in the sample must match the ratio found in the continuing calibration standards (CCS) ± 30%.

Note that interferences in at least one of the ion traces can distort the identification process.

5.6.07.2.2 Positive results by GC/FPD must be confirmed using either, a different column type or by GC/MS.

5.6.08 For Samples run by liquid injection GC/MS, it is recommended that DFTPP be run every twelve hours before the analytical sequence to insure

that the mass spectrometer is properly tuned. DFTPP injection time represents the start of a twelve-hour tune window in which samples and standards can be injected. DFTPP tune passes if it meets the following criteria:

51 m/z	30.0 – 80.0 percent of mass 198 m/z
68 m/z	Less than 2.0 percent of mass 69 m/z
69 m/z	Present
70 m/z	Less than 2.0 percent of mass 69 m/z
127 m/z	25.0 – 75.0 percent of mass 198 m/z
197 m/z	Less than 1.0 percent of mass 198 m/z
198 m/z	Base peak, 100 percent relative abundance
199 m/z	5.0 – 9.0 percent of mass 198 m/z
275 m/z	10.0 – 30.0 percent of mass 198 m/z
365 m/z	Greater than 0.75 percent of mass 198m/z
441 m/z	Present but less than mass 443 m/z
442 m/z	40.0 – 110.0 percent of mass 198 m/z
443 m/z	15.0 – 24.0 percent of mass 442 m/z

5.7 CALCULATIONS

5.7.01 Initial Calibration Calculations (Concentration is in terms of on column amounts)

5.7.01.1 Linear Equation → Response = B (Conc) + A, where Conc is the calculated concentration and B and A are coefficients calculated by the processing software. For regression output, B is the X coefficient, and A is the constant, where concentration is the Independent variable and Response is the dependent variable. Note: Response is equal to area if no internal standard is used, otherwise it is Target Area * IS Conc. / IS Area. When solved in terms of Concentration, the equation is

Conc. = (Response – A) / B

5.7.01.2 Quadratic Equation→ Area = C $(Conc)^2$ + B (Conc) + A, where Conc is the calculated concentration and C, B, and A are coefficients calculated by the Millennium software. When the equation is solved in terms of Concentration, two possible solutions are found. Only one solution can be valid.

Conc = -B / 2C – sqrt[$(B / 2C)^2$ + ((Area – A) / C)] or
Conc = -B / 2C + sqrt[$(B / 2C)^2$ + ((Area – A) / C)]

5.7.01.3 Calibration by Response Factors (RF)

RF = (Target Area * IS Conc) / (IS Area * Target Conc)

Mean RF is the average of all Initial Calibration Standard Response Factors. To calculate the % Relative Standard Deviation

(%RSD) find the sample standard deviation for the Initial Calibration Standard Response Factors and divide by the Mean Response Factor.

5.7.02 Sample Concentration Calculations (ug/L or ug/Kg)

5.7.02.1 Sample Concentration in Water

ug/L = (A * B) / (C * D)

Where: A = ng or ng/uL on column amount of target compound
B = Sample extract volume in uL
C = DAAMS inj. Volume in uL: for liquid inj. enter (1)
D = Amount of extract in mL.

5.7.02.2 Sample Concentration in Solids

ug/Kg = (A * B) / (C * D)

Where: A = ng or ng/uL on column amount of target compound
B = Sample extract volume in uL
C = DAAMS inj. Volume in uL: for liquid inj. enter (1)
D = Amount of extract in grams.

5.8 REPORTING

5.8.01 Extraction Notebook → will be submitted with every Package. Each extraction page will be titled with Client, work order numbers, SDG, project number, book number, signed and dated by the individual entering the information. Any information relating to the extraction or analysis of the samples, should be recorded. At a minimum, the page should include sample ID, sample amount measured, solvent (include lot numbers), solvent amount used for extraction or dilution, spiked agent (include lot numbers), amount of spiked agent, balance number, TAP reference, and information regarding standard preparations. Upon completion, the page or pages should be reviewed.

5.8.02 Instrument Injection Notebook → will be submitted with every Package. Each extraction page will be titled with Client, work order numbers, SDG, project number, book number, signed and dated by the individual entering the information. Any information relating to the Instrument conditions, standards and sample injections should be recorded. At a minimum, the page should include the oven program, column type, all sample IDs, standard IDs, associated dates, and times of injection. Upon completion, the page or pages should be reviewed.

5.8.03 Raw Data → will be submitted for all standards and samples. It will contain at a minimum, File Name, Injection Date, Time, Sample ID, chromatogram trace or Ion trace for each analyte, analyte retention time, analyte area, and on column analyte concentration.

5.8.04 Form 1 (Sample Concentration Information Form)→ will be submitted for every sample. The form will include sample concentration in terms of ug/L or ug/Kg, if it is detected above the PQL. At a minimum, the form will contain client sample ID, project number, SDG, matrix, sample weight or volume, concentrated extract volume, date received, date extracted, date analyzed, and necessary data reporting qualifiers.

5.8.05 Form 3 (Matrix Spike/Matrix Spike Duplicate Recovery Form)→ will be submitted for each set of MS/MSD extracted and run. The form will include sample concentration, matrix spike concentration, matrix spike dupl. (if applicable), percent MS recovery, percent MSD recovery (if applicable), QC limits and %RPD (if applicable). At a minimum, the form will contain client sample ID, project number, and SDG.

5.8.06 Form 4 (Blank Summary Form) → will be submitted for every extraction blank. The forms purpose is to list all Sample ID's, SwRI ID's, Lab File ID's and sample acquisition dates associated with that particular blank. At a minimum, the form will contain Blank sample ID and Blank date analyzed, project number, and SDG.

5.8.07 Form 5 (DFTPP Tune Check for GC/MS Form) → is recommended for all liquid GC/MS sequences. The form will display a chart verifying passing criteria for the DFTPP GC/MS Instrument Performance Check and it will list all standards and samples run within twelve-hour tune window. At a minimum, the form will contain DFTPP File ID, DFTPP injection date, time, project number, and Instrument.

5.8.08 Form 6 (Initial Calibration Verification Form) → will be submitted for all Initial calibrations. The form will list all initial calibration standards and any additional information required to demonstrate that all ICV criteria have been met. At a minimum, the form will contain Calibration date, time, project number, instrument and SDG.

5.8.09 Form 7 (Continuing Calibration Verification Form) → will be submitted for all Continuing Calibration Verifications. The form will list Continuing Calibration Filename, date, time, and any additional information required to demonstrate that all CCV criteria have been met. At a minimum, the form will contain the Initial Calibration date, time, project number,

instrument and SDG.

5.8.10 Form 8 (Internal Standard Area and RT Summary Form) → will be submitted for each beginning CCV in which internal standards are employed. The form will list the beginning CCV's Internal Standard area, retention time, upper and lower advisory limits for IS area and RT. Below it will list all samples (ID's, area and RT) associated with that CCV and flag any samples in which the IS area or RT is outside the advisory limits. At a minimum, the form will contain the CCV filename, date, time, project number, instrument and SDG.

5.8.11 Forms 1 through 8 will be collected, printed from, and electronically saved in a spreadsheet or Quickforms.

6.0 MAINTENANCE

Not Applicable.

7.0 SAFETY

7.1 Requirements indicated in the **Surety Chemical Hygiene Plan** for agent operations must be met.

7.2 PPE requirement is safety glasses, lab coat, protective gloves, and a mask within arms reach. The availability of response equipment and support personnel should be as indicated in the laboratory Chemical Hygiene Plan (CHP) for agent operations.

7.3 Secondary containment is required for all transfer of agent out of engineering control.

7.4 Response team must be available during agent operation. Monitoring system must be operational for specific agent in use or most volatile agent in use.

7.5 All self-aid and first aid materials must be available.

7.6 Exposure to chemical agent material is possible from contact. Respiratory exposure can result from spills or improper use of ventilation control.

7.6.01 Risk is primarily associated with compromise of protective gloves. Compromise is most likely to occur as agent vials are opened.

7.6.02 Dichloromethane has been reported to cause cancer in laboratory animals, avoid contact with eyes, skin, and clothing. Avoid breathing vapors.

7.6.03 Sodium Hydroxide is corrosive. Causes burns to any area of contact. Reacts with water, acids, and other materials.

7.7 Emergency Response Procedures should be followed as identified in the **Surety Chemical Hygiene Plan** for agent operations.

8.0 RECORDS

8.1 All records will be maintained and reported in accordance with client/contract requirements.

8.2 Extraction notebooks and instrument injection logbooks will be filled out and controlled according SwRI ISO 9001 SOP-01-5.6.

8.3 Raw Data, Quickforms file and/or spreadsheet containing the Forms (1-8) data will be archived.

APPENDIX B

IITRI REPORT OF RESULTS

DIRECT VX AGENT ANALYSIS

IITRI Project No. 246-0-2008
Prime Contract No. DAAA09-96-C-0081

DRAFT FINAL REPORT

Prepared for:

Washington Demilitarization Company
Honolulu, Hawaii 96819

Attention:

Thomas W. Freethy

Prepared by:

IIT Research Institute
10 West 35th Street
Chicago, Illinois 60616-3799

October 25, 2002

PREFACE

This document presents the results of a study entitled "Direct VX Agent Analysis" performed at IIT Research Institute (IITRI), Project No. 246-0-2008, for Washington Demilitarization Company under the Prime Contract No. DAAA09-96-C-0081. The program was initiated on October 7, 2002 and was completed in October 18, 2002. IITRI personnel Yirgaalem Abrha and Michael Miller worked on this project. Experimental data generated in the program are recorded in IITRI logbook C31763. This logbook, as well as associated chromatograms and integration data, will be stored in IITRI archives.

This report has been audited by IITRI's Quality Assurance Department. The entire document has been verified to comply with our quality system and contractual requirements. IITRI is pleased to submit this report to Washington Demilitarization Company for Johnston Atoll Chemical Agent Disposal System (JACADS).

Respectfully submitted,

IIT RESEARCH INSTITUTE

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EXECUTIVE SUMMARY

A previously analyzed ash sample collected from the JACADS demil activities was submitted to IITRI for VX analysis. The extractions employing an ultrasonic extraction method were performed using the analytical approach that was developed for the analysis of VX in a cement matrix. The analyses were performed using a Varian Saturn 2000 gas chromatograph/ion trap mass spectrometer (GC/ITMS) instrument using the operating parameters developed for the analysis of residual VX in VX/caustic neutralization solutions. This analytical method employs chemical ionization (CI) mass spectrometry/mass spectrometry (MS/MS).

In addition to the ash samples, method blanks consisting of anhydrous sodium sulfate (Na_2SO_4) and all glassware and reagents used in this method and method spikes (Na_2SO_4 plus VX spike) were extracted in duplicate. The extraction solvent used was a mixture containing 90% methylene chloride ($MeCl_2$) and 10% 2-propanol (IPA). Five grams (g) of anhydrous sodium sulfate (Na_2SO_4) were mixed with the ash before the addition of the extraction solvent. After addition of the extraction solvent, the samples were pH-adjusted to approximately 9 using triethylamine prior to ultrasonic extraction. Following extraction, the sample extracts were concentrated using a stream on high-purity nitrogen gas. The concentrated sample extracts were spiked with a fixed amount of diethyaminoethanethiol (DEAET), a chemical that is added to the VX standards to assist in the GC/ITMS analysis.

No detectable VX was found in the unspiked method blanks. Method spikes were performed at two levels; the initial concentration was 0.457 microgram (µg)/g and the second level was 0.091 µg/g. The average percent (%) recovery determined for the initial level was 88%; however, the values from the duplicate samples showed variability. It was determined that this spike level was too large; therefore, a second pair of method spikes was prepared at a VX concentration of 0.091 µg/g. The percent recoveries for these spiked duplicates were 88% and 96% with an average of 92%.

The VX levels found in the initial 5 g ash samples exceeded the calibration range. Thus, one milliliter (mL) of each sample extract was diluted to 10 mL using methylene chloride and

reanalyzed. The average VX concentration determined in these diluted extracts was 2.94 μg/g. In order to eliminate the dilution step, the ash sample size was reduced to one gram, and two unspiked and two VX-spiked (2.74 μg/g) extracts were prepared. The average VX concentration determined in the 1-gram ash samples was 4.1 μg/g, and the average recovery (corrected for residual VX in unspiked 1-gram ash sample) from the two spiked samples was 114%.

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SECTION 1
INTRODUCTION

This test report provides the technical approach used to achieve the program objectives for the tests and experiments described in the proposal entitled "Direct VX Agent Analysis." A single sample (ID No. LAWIC 153 B) of ash reportedly derived from the metals part furnace (MPF) in the JACADS system was received on October 7, 2002. The objective of this task is to analyze the ash sample for VX using an ion trap mass spectrometer and a standard operating procedure (SOP) that IITRI had previously developed and validated for the analysis of VX in VX/NaOH (caustic hydrolysate). There is interest in analyzing this sample because prior analyses have indicated the presence of VX even though the MPF is designed to decontaminate to a 5X level. This analysis was performed to provide confirmation of the results previously obtained from the analysis of this ash matrix.

Instruments, equipment, supplies and chemicals used for this task are specified within IITRI SOPs C-Safety-204, "Ultrasonic Extraction Procedure for Residual VX, GB, HD, Nonvolatile and Semivolatile Organic Compounds in Solid Matrices," and C-Analytical-100, "Analysis of Low Level VX in VX/NaOH/H_2O and VX/H_2O Hydrolysate Solutions Using Liquid-Liquid Extraction." The quality assurance (QA) and quality control (QC) measures that were used are presented within these SOPs. The applicable sections of these SOPs were adapted for the ash matrix. The hexane acid extraction steps found in SOP C-Analytical-100 were omitted because the specifics of this procedure were developed for use with the VX/caustic hydrolysate matrix.

SECTION 2
EXPERIMENTAL PROCEDURE

2.1 Sample Extraction Procedure

The samples listed in Table 2-1 were extracted using a solvent mixture of 90% methylene chloride/10% 2-propanol and anUltrasonic Processor Model CPX-500 (Cole-Parmer Instruments) following the procedure presented in IITRI SOP C-Safety-204. The samples were prepared using the extraction procedure presented below:

- To the 5 g or 1 g of ash matrix, 5 grams of sodium sulfate (dried at 400°C for approximately 24 hours) were added and mixed.
- The matrix was spiked (if required) with VX using 25 to 50 microliters (µL) of either a 91.3 microgram per milliliter (µg/mL) or 9.13 µg/mL standard solution prepared in 2-propanol and allowed to dry on the matrix for three to five minutes.
- 20 mL of extraction solvent mixture were added to the matrix.
- The pH was adjusted to approximately 9 using triethylamine.
- The sample was extracted at room temperature (approximately 22°C) using an ultrasonic processor for three minutes at a 70% amplitude setting. The ultrasonic processor was set to pulse 0.5 second on and 0.5 second off.
- The extraction solvent layer was decanted into a 50-mL centrifuge tube.
- The extraction was repeated with another 20 mL of extraction solvent. The second extraction solvent layer was transferred into the same centrifuge tube containing the original extract.
- The remaining matrix sample and sample vessel were rinsed with several milliliters of extraction solvent, and the solvent transferred to the centrifuge tube.
- The total extract was centrifuged for five minutes, and the liquid was transferred into another graduated centrifuge tube.
- The extract was concentrated to the final volume under stream of high-purity nitrogen gas.

- An aliquot of each sample was transferred to an autosampler vial and the appropriate amount of DEAET was added; the sample was submitted for GC/ITMS analysis.
- Samples were stored in glass vials with Teflon-lined screw caps at 0°C.

In addition to the extractions performed using different weights (5 g and 1 g) of the ash matrix, method blanks consisting of anhydrous Na_2SO_4 and all glassware and reagents used in this method and method spikes (Na_2SO_4 plus VX spike) were extracted. Each sample type was prepared in duplicate.

Table 2-1. Samples Prepared Using Ultrasonication Extraction

Extraction Date	Sample Identification	Sample Description	Sample Weight (g)	VX Spike (µg/g)	Final Extract Volume (mL)
10-14-02	XVX-101402-US-1	Method Blank: 5 g Na_2SO_4	---	0	10
	XVX-101402-US-2	Method Blank: 5 g Na_2SO_4	---	0	10
	XVX-101402-US-3	Method Spike: 5 g Na_2SO_4	---	0.457	10
	XVX-101402-US-4	Method Spike: 5 g Na_2SO_4	---	0.457	10
10-15-02	XVX-101502-US-1	Method Spike: 5 g Na_2SO_4	---	0.091	2
	XVX-101502-US-2	Method Spike: 5 g Na_2SO_4	---	0.091	2
	XVX-101502-US-3	5 g Ash + 5 g Na_2SO_4	5.07	0	5
	XVX-101502-US-4	5 g Ash + 5 g Na_2SO_4	5.08	0	5
10-17-02	XVX-101702-US-1	1 g Ash + 5 g Na_2SO_4	1.10	0	5
	XVX-101702-US-2	1 g Ash + 5 g Na_2SO_4	1.00	0	5
	XVX-101702-US-3	1 g Ash + 5 g Na_2SO_4	1.06	2.74	5
	XVX-101702-US-4	1 g Ash + 5 g Na_2SO_4	1.09	2.74	5

2.2 Saturn 2000 GC/ITMS Instrument Operating Parameters

The VX analyses were performed using a Varian Saturn 2000 GC/ITMS employing methanol CI-MS/MS as the technique to isolate the VX from other components in the extracted samples. The Saturn 2000 was mass-tuned each day using perfluorotributylamine (PFTBA) prior to

sample analysis. Additionally, ion trap function and air and water checks were performed daily. The GC/ITMS data were acquired using the conditions presented below.

2.2.1 Varian 3800 GC Conditions.

Column:	Rtx-5MS, 30-meter (m) × 0.25-millimeter (mm) internal diameter (ID), 1.0-micrometer (µm) phase
Carrier Gas:	Helium, ultra high purity at 1 milliliter per minute (mL/min)
Injection Type:	Splitless with purge on at 0.7 min
Injection Volume:	1 µL
Injector Temp:	250°C
Column Temp Program:	100°C (5 min) to 240°C at 5°C/min to 280°C at 25°C/min hold at 280°C for 5 min

2.2.2 Varian Saturn 2000 CI-ITMS/MS Conditions.

General Instrument Parameters:

Ion Trap Temp:	170°C Silcosteel-Treated
Manifold Temp:	50°C
Transfer Line Temp:	280°C
Axial Modulation:	3.5 volts
Electron Multiplier:	AutoTune value + 200 volts
CI Reagent:	Methanol

CI/MS/MS Parameters

Emission Current:	50 microamps
Count Threshold:	1 counts
Multiplier Offset:	+300 volts
Scan Time:	0.65 seconds
Segment Start Time:	24.00 min
Segment End Time:	33.00 min
Segment Low Mass:	122 m/z
Segment High Mass:	270 m/z
Ionization Mode:	CI Auto
Parent Ion Mass:	268.2 m/z
Isolation Window:	1.0 m/z
Isolation Time:	2 milliseconds (msec)
Ion Preparation Technique:	MS/MS

2.3 Preparation of VX Standards

The primary VX stock solution (approximately 1 milligram per milliliter, mg/mL) was prepared from neat VX. The process followed is the standard protocol performed by weighing the VX and diluting with 2-propanol in Class A glassware. The standards for calibrating the Saturn 2000 GC/ITMS were prepared according to SOP C-Analytical-100. The calibrating standards diluted in hexane range from 1.83 µg/mL to 0.046 µg/mL. To each calibration standard, DEAET has been added at a concentration of 0.898 mg/mL. This compound has been added to assist in the VX analysis.

2.4 Sample Analysis

The MS/MS analysis of VX results in the primary formation of the m/z 128 ion that is used for quantitation. Additional confirmational ions (m/z 139 and m/z 167) also are monitored; however, their presence is not required if the correct retention time is determined for the m/z 128. The relative ion ratios determined for the 139/128 and 167/128 may vary. Therefore, no specific criteria for these ratios has been established for positive VX identification. Their presence is used as a confirmatory indicator for VX identification. The Saturn 2000 processing method(s) used to quantify the VX have an acceptance range for these ion ratios that may not be achieved. The pass/fail notation on the data printouts should not be used as an indicator for identification of VX in these samples.

During the analyses of these samples it was observed that the instrument response (m/z 128 area) increased after the analysis of sample extracts. This change in response resulted in the three calibrations being performed during this task. The data from these calibrations are presented in Tables 2-2, 2-3 and 2-4 and a representative chromatogram for a calibration standard is shown in Figure 2-1.

Table 2-2. VX Calibration Performed 10-14-02 Using the Saturn 2000 GC/ITMS

File Name	Standard Identification	VX Concentration (µg/mL)	m/z 128 Ion Area	RT (min)
XVX-10-03-02-1, 1.8 µg 10-14-02001	XVX-10-03-02-1	1.83	712854	28.758
XVX-10-03-02-2, 0.91 µg 10-14-02	XVX-10-03-02-2	0.913	268620	28.778
XVX-10-03-02-3, 0.46 µg 10-14-02	XVX-10-03-02-3	0.457	118646	28.784
XVX-10-03-02-4, 0.18 µg 10-14-02	XVX-10-03-02-4	0.183	54898	28.786
XVX-10-03-02-5, 0.091 µg 10-15-02	XVX-10-03-02-5	0.091	10577	28.802
XVX-10-03-02-5, 0.046 µg 10-15-02	XVX-10-03-02-5	0.046	3477	28.799
XVX-10-03-02-3, 0.46 µg 10-15-02	XVX-10-03-02-3 QC-Check	0.457	83614	28.798

Notes:

Calibration equation for six-point, quadratic fit: $y= 96226.5x^2 + 213859.3x -1749.64$
Correlation coefficient, $R^2= 0.9991$
RT = retention time
µg/mL = microgram per milliliter
QC-Check % difference = -24.5%

Table 2-3. VX Calibration Performed 10-16-02 Using the Saturn 2000 GC/ITMS

File Name	Standard Identification	VX Concentration (µg/mL)	m/z 128 Ion Area	RT (min)
XVX-10-03-02-1, 1.8 µg 10-16-02001	XVX-10-03-02-1	1.83	3119859	28.750
XVX-10-03-02-2, 0.91 µg 10-16-02	XVX-10-03-02-2	0.913	1213277	28.755
XVX-10-03-02-3, 0.46 µg 10-16-02003	XVX-10-03-02-3	0.457	647717	28.758
XVX-10-03-02-4, 0.18 µg 10-16-02	XVX-10-03-02-4	0.183	294563	28.758
XVX-10-03-02-5, 0.091 µg 10-16-02	XVX-10-03-02-5	0.091	73654	28.764
XVX-10-03-02-5, 0.046 µg 10-16-02	XVX-10-03-02-5	0.046	40986	28.765
XVX-10-03-02-3, 0.46 µg 10-16-02004	XVX-10-03-02-3 QC-Check	0.457	688952	28.760

Notes:

Calibration equation for six-point, quadratic fit: $y= 343619.5x^2 + 1056865.5x +24310.9$
Correlation coefficient, $R^2= 0.9976$
RT = retention time
µg/mL = microgram per milliliter
QC-Check % difference = 16.3%

Table 2-4. VX Calibration Performed 10-17-02 Using the Saturn 2000 GC/ITMS

File Name	Standard Identification	VX Concentration (µg/mL)	m/z 128 Ion Area	RT (min)
XVX-10-03-02-1, 1.8 µg 10-17-02001	XVX-10-03-02-1	1.83	3054257	28.748
XVX-10-03-02-2, 0.91 µg 10-18-02	XVX-10-03-02-2	0.913	1281206	28.748
XVX-10-03-02-3, 0.46 µg 10-18-02	XVX-10-03-02-3	0.457	735341	28.754
XVX-10-03-02-4, 0.18 µg 10-18-02	XVX-10-03-02-4	0.183	251252	28.753
XVX-10-03-02-5, 0.091 µg 10-18-02	XVX-10-03-02-5	0.091	99121	28.752
XVX-10-03-02-5, 0.046 µg 10-18-02	XVX-10-03-02-5	0.046	45644	28.756
XVX-10-03-02-3, 0.46 µg 10-18-02001	XVX-10-03-02-3 QC-Check	0.457	620127	28.754

Notes:

Calibration equation for six-point, quadratic fit: $y = 204171.5x^2 + 1286284.6x + 5592.58$
Correlation coefficient, $R^2 = 0.9976$
RT = retention time
µg/mL = microgram per milliliter
QC-Check % difference = -3.0%

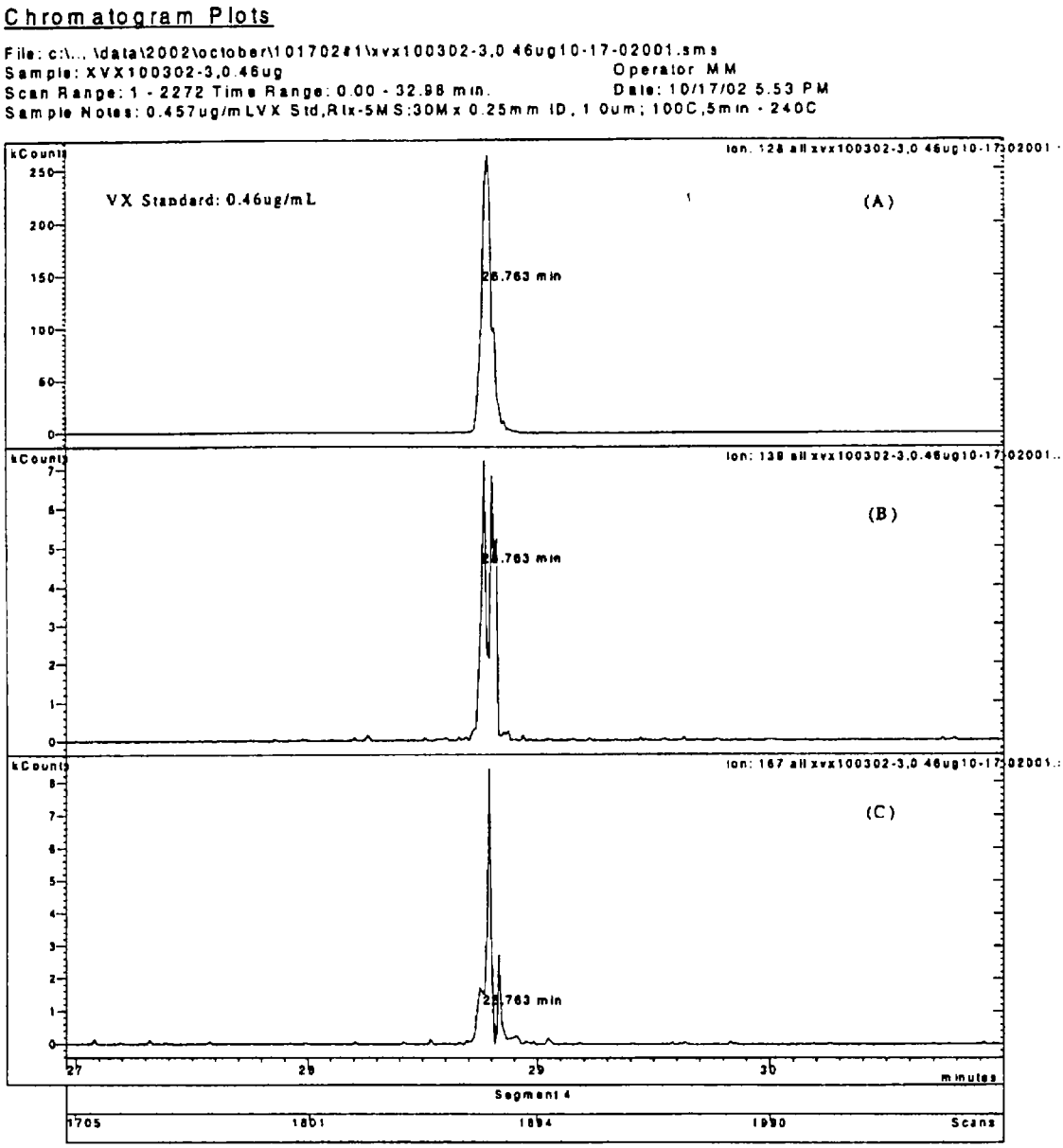


Figure 2-1. Selected-Ion-Chromatograms from the Analysis of a 0.457-µg/mL VX Calibration Standard: (A) m/z 128, (B) m/z 139, (C) m/z 167

The results from the various analyses performed during this task are summarized in Tables 2-5 and 2-6. No detectable VX was found in the unspiked method blanks. Method spikes were performed at two levels, the initial VX spike concentration was 0.457 μg/g, and the second level was 0.091 μg/g. The average percent recovery determined for the initial level was 88%; however, the values from the duplicate samples showed variability. A second pair of method spikes was prepared at a lower VX concentration (0.091 μg/g) and analyzed on 10-15-02 and 10-17-02. The average percent recovery determined on these two days was 129% and 92%, respectively. These samples were reanlyzed after instrument response had stabilized and following recalibration.. A retention time shift was observed in the method spikes when compared with calibration standards. This shift was not observed in the ash sample extracts and was attributed to the reduced quantity of extractable components present in the Na_2SO_4.

The VX levels found in the initial 5 g ash samples exceeded the calibration range; therefore, 1 mL of each sample was diluted to 10 mL using methylene chloride and reanalyzed. These samples were analyzed on 10-16-02 and 10-17-02, and the average VX concentrations that were determined in these diluted extracts were 3.70 μg/g and 2.94 μg/g, respectively. In order to eliminate the dilution step, the ash sample size was reduced to 1 g and two unspiked and two VX-spiked (2.74 μg/g) extracts were prepared. The average VX concentration determined in the 1-gram ash samples was 4.1 μg/g, and the average recovery (corrected for residual VX in unspiked 1-gram ash sample) from the two spiked samples was 114%. Figure 2-2 shows the response for VX detected in the unspiked and VX-spiked 1-gram ash samples.

To assist in the confirmation of the VX response in the diluted (1:10) 5-gram ash samples, two levels of VX were spiked into 1-mL aliquots of these samples. As illustrated in Figure 2-3 the VX spikes support the VX identification in the ash samples.

Table 2-5. GC/ITMS Results from the Samples Analyzed During This Task

Sample Identification		Analyzed concentration (µg/mL)	Final Volume (mL)	VX Spike Level
Sample ID No.	ITMS File ID No.			
Samples prepared on 10-14-02				
XVX-101402-US-1	VX-101402-US-1-10-15-02	0	10	
XVX-101402-US-2	VX-101402-US-2-10-15-02	0	10	
XVX-101402-US-3	VX-101402-US-3-10-15-02	0.295	10	0.457 µg/g
XVX-101402-US-4	VX-101402-US-4-10-15-02	0.109	10	0.457 µg/g
Samples prepared on 10-15-02				
XVX-101502-US-1	VX-101502-US-1-10-15-02	0.260	2	0.091 µg/g
	-1-10-17-02	0.200		
XVX-101502-US-2	VX-101502-US-2-10-15-02	0.327	2	0.091 µg/g
	-2-10-17-02	0.219		
XVX-101502-US-3	VX-101502-US-3-10-15-02	NC	5	
XVX-101502-US-4	VX-101502-US-4-10-16-02	NC	5	
Samples XVX-101502-3 and -4 were diluted 1:10 with $MeCl_2$ on 10-16-02				
XVX-101602-US-3D	VX-101502-US-3D-10-16-02	0.368	1:10	
	VX-101502-US-3D-10-17-02	0.375		
XVX-101602-US-4D	VX-101502-US-4D-10-16-02	0.295	1:10	
	VX-101502-US-4D-10-17-02	0.365		
XVX-101602-US-3DS1[a]	VX-101502-US-3DS-10-17-02	NC	1:10	2.7 µg/mL
XVX-101602-US-4DS1[a]	VX-101502-US-4DS-10-17-02	NC	1:10	2.7 µg/mL
XVX-101602-US-3D	VX-101602-US-3D1-10-17-02	0.302	1:10	
XVX-101602-US-3DS2[a]	VX-101702-US-3DS2-10-17-02	1.02	1:10	0.73 µg/mL
XVX-101602-US-4D	VX-101602-US-4D1-10-17-02	0.285	1:10	
XVX-101602-US-4DS2[a]	VX-101702-US-4DS2-10-17-02	1.01	1:10	0.73 µg/mL
Samples prepared on 10-17-02				
XVX-101702-US-1	VX-101702-US-1-10-18-02	0.84	5	
XVX-101702-US-2	VX-101702-US-2-10-18-02	0.78	5	
XVX-101702-US-3	VX-101702-US-3-10-18-02	1.41	5	2.7 µg/g
XVX-101702-US-4	VX-101702-US-4-10-18-02	1.46	5	2.7 µg/g

Notes:

[a] The 1 mL of the 5-gram diluted ash extracts were spiked with VX to assist in confirming the presence of VX
NC = not calculated

Table 2-6. GC/ITMS Recovery Results from the Samples Analyzed During This Task

Sample Identification		VX Concentration (µg/mL)		Recovery %
Sample ID No.	ITMS File ID No.	Analyzed (µg/mL)	Sample (µg/g)	
Samples prepared on 10-14-02				
XVX-101402-US-1	VX-101402-US-1-10-15-02	0		
XVX-101402-US-2	VX-101402-US-2-10-15-02	0		
XVX-101402-US-3	VX-101402-US-3-10-15-02	0.295	2.95	129.1
XVX-101402-US-4	VX-101402-US-4-10-15-02	0.109	1.09	47.7
Samples prepared on 10-15-02				
XVX-101502-US-1	VX-101502-US-1-10-15-02	0.260	0.52	114.3
	-1-10-17-02	0.200	0.40	87.9
XVX-101502-US-2	VX-101502-US-2-10-15-02	0.327	0.654	143.7
	-2-10-17-02	0.219	0.438	96.3
XVX-101502-US-3	VX-101502-US-3-10-15-02	NC		
XVX-101502-US-4	VX-101502-US-4-10-16-02	NC		
Samples XVX-101502-3 and -4 were diluted 1:10 with $MeCl_2$ on 10-16-02				
XVX-101602-US-3D	VX-101502-US-3D-10-16-02	0.368	3.68	
	VX-101502-US-3D-10-17-02	0.375	3.75	
XVX-101602-US-4D	VX-101502-US-4D-10-16-02	0.295	2.95	
	VX-101502-US-4D-10-17-02	0.365	3.65	
XVX-101602-US-3DS1[a]	VX-101502-US-3DS-10-17-02	NC		
XVX-101602-US-4DS1[a]	VX-101502-US-4DS-10-17-02	NC		
XVX-101602-US-3D	VX-101602-US-3D1-10-17-02	0.302	3.02	
XVX-101602-US-3DS2[a]	VX-101702-US-3DS2-10-17-02	1.02	10.2	98.3
XVX-101602-US-4D	VX-101602-US-4D1-10-17-02	0.285	2.85	
XVX-101602-US-4DS2[a]	VX-101702-US-4DS2-10-17-02	1.01	10.1	99.3
Samples prepared on 10-17-02				
XVX-101702-US-1	VX-101702-US-1-10-18-02	0.84	4.20	
XVX-101702-US-2	VX-101702-US-2-10-18-02	0.78	3.90	
XVX-101702-US-3	VX-101702-US-3-10-18-02	1.41	7.05	111.1
XVX-101702-US-4	VX-101702-US-4-10-18-02	1.46	7.30	119.0

Notes:

[a] The 1 mL of the 5-gram diluted ash extracts were spiked with VX to assist in confirming the presence of VX
NC = not calculated

Chromatogram Plots

Plot 1: c:\... \101702#1\vx-101702-us-1-10-18-02.sms Ion: 128 all
Plot 2: c:\... \101702#1\vx-101702-us-2-10-18-02.sms Ion: 128 all
Plot 3: c:\... \101702#1\vx-101702-us-3-10-18-02.sms Ion: 128 all
Plot 4: c:\... \101702#1\vx-101702-us-4-10-18-02.sms Ion: 128 all

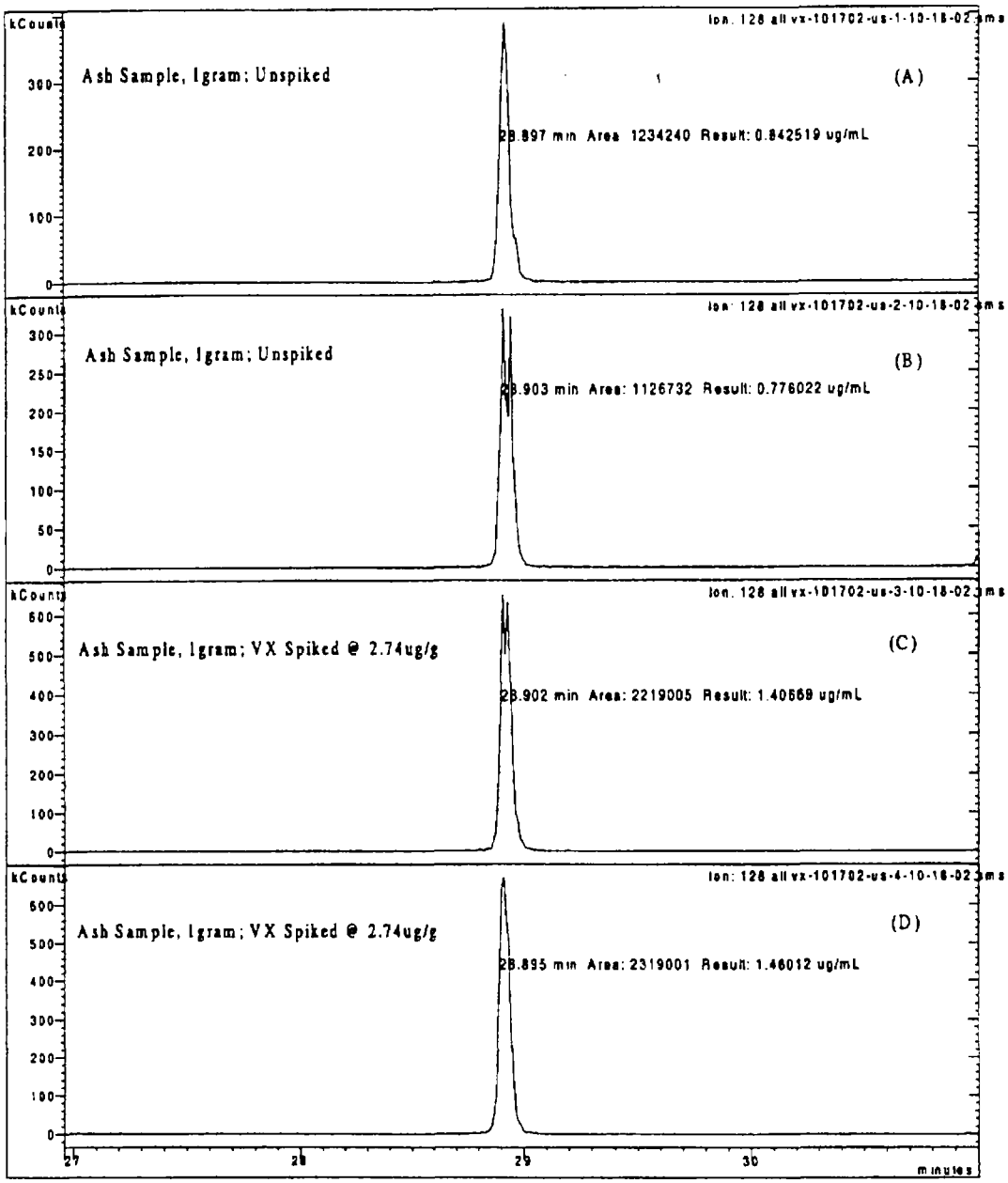


Figure 2-2. GC/ITMS Chromatograms (m/z 128) from the Analyses of Duplicate Unspiked (A & B) and VX-Spiked (C & D) 1-Gram Ash Samples

Chromatogram Plots

Plot 1: c:\... \101702#1\vx-101602-us-3d1-10-17-02.sms Ion: 128 all
Plot 2: c:\... \101702#1\vx-101702-us-3ds2-10-17-02 sms Ion: 128 all
Plot 3: c:\... \101702#1\vx-101602-us-4d1-10-17-02.sms Ion. 128 all
Plot 4: c:\... \101702#1\vx-101702-us-4ds2-10-17-02.sms Ion: 128 all

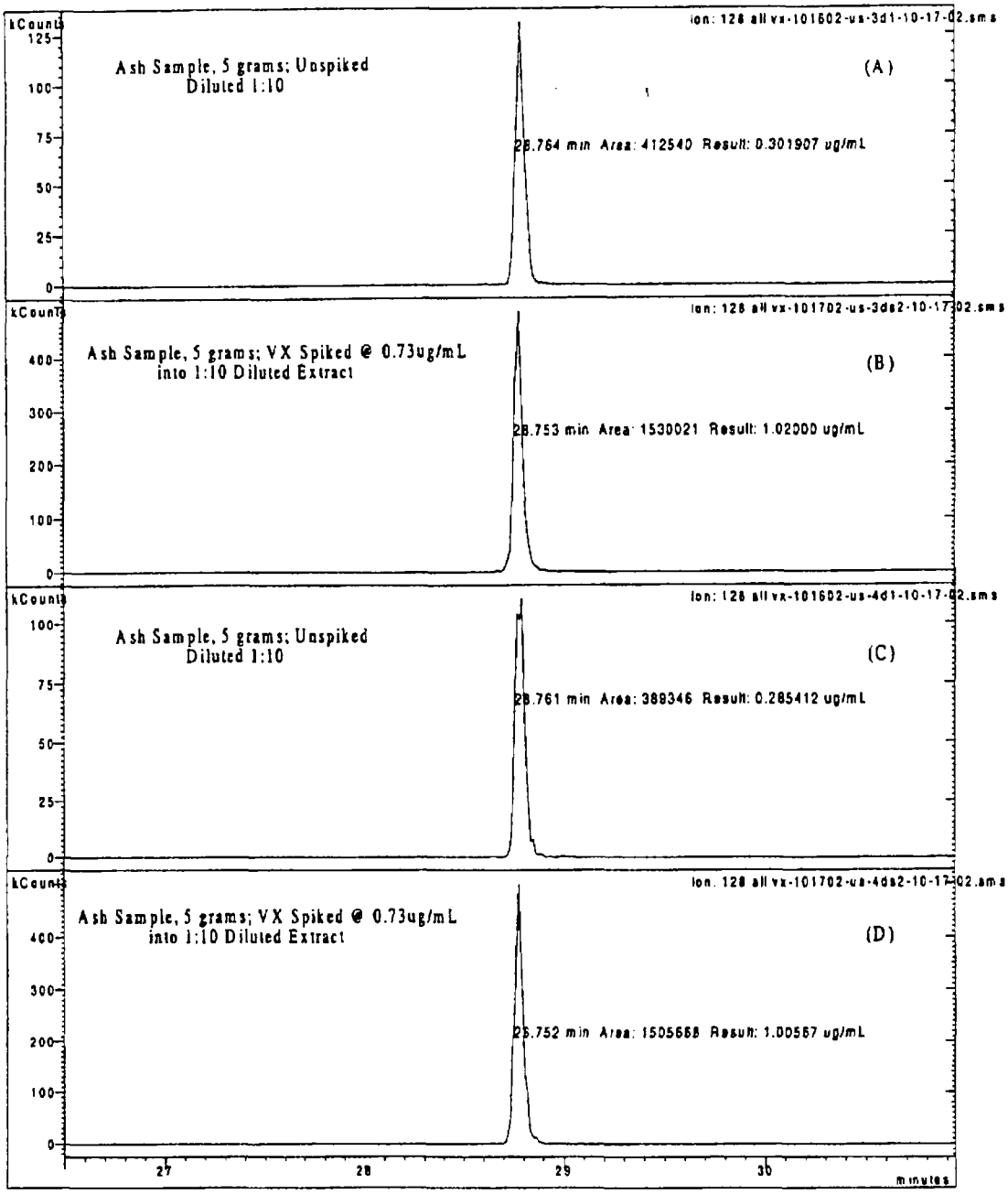


Figure 2-3. GC/ITMS Chromatograms from the Diluted (1:10) 5-Gram Ash Samples: (A) and (C) Are Unspiked, (B) and (D) Are Spiked (0.73 µg/mL VX)

SECTION 3
RESULTS AND CONCLUSIONS

As seen by the recoveries in Table 2-6, the extractions employing an ultrasonic extraction method and analyzing the ash sample for VX using GC/ITMS works well. The average VX concentration determined in these diluted extracts was 2.94 μg/g. In order to eliminate the dilution step, the ash sample size was reduced to one gram, and two unspiked and two VX-spiked (2.74 μg/g) extracts were prepared. The average VX concentration determined in the 1-gram ash samples was 4.1 μg/g, and the average recovery (corrected for residual VX in unspiked 1-gram ash sample) from the two spiked samples was 114%. The spike extracts and the spike on the ash sample confirm the presence of VX in the sample.

SECTION 4
SOP DEVIATIONS

IITRI SOP C-Safety-204 was used with the following deviations:

- Due to the limited sample amount received, the amount of sample used was 5 g and 1 g instead of 25 g.
- Sonication was performed twice using 20 mL of extraction solvent instead of three times using 100 mL of extraction solvent.
- Triethylamine was added to adjust the pH to 9.
- The combined extract was concentrated by blowing done with nitrogen.
- The extracts were analyzed using a GC/ITMS instead of a gas chromatograph-flame photometric detector (GC/FPD).

APPENDIX A
ACRONYMS/ABBREVIATIONS

CI	chemical ionization
DEAET	diethylaminoethanethiol
g	gram
GC/FPD	gas chromatograph/flame photometric detector
GC/ITMS	gas chromatograph/ion trap mass spectrometer
ID	internal diameter
IITRI	IIT Research Institute
IPA	2-proponal
JACADS	Johnston Atoll Chemical Agent Disposal System
m	meter
$MeCl_2$	methylene chloride
mg/mL	microgram per milliliter
mL	milliliter
mL/min	milliliter per minute
mm	millimeter
MPF	metals part furnace
msec	millisecond
MS/MS	mass spectrometry/mass spectrometry
m/z	mass to charge ratio
Na_2Cl_2	sodium sulfate
NC	not calculated
PFTBA	perfluorotributylamine
QA	quality assurance
QC	quality control
RT	retention time
SOP	standard operating procedure
µg/g	microgram per gram
µg/mL	microgram per milliliter

APPENDIX A

ACRONYMS/ABBREVIATIONS (CONT.)

μL	microliter
μm	micrometer

APPENDIX C

SwRI DATA PACKAGE FOR GC/MS

SOUTHWEST RESEARCH INSTITUTE
CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order No.: 23238
VTSR: October 7, 2002

NARRATIVE

CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order Number: 23238
Page#: 1

SwRI CASE NARRATIVE

1. One (1) solid samples was received for analysis by LC/MS and VX Direct:

SwRI ID	Customer ID	Matrix
213854	Tray 153(021002-001)	Solid

2. Samples were received at SwRI on October 7, 2002 for a thirty (30) day turnaround from Validated Time of Sample Receipt (VTSR).

3. Quality Control Sample Identification:
   Tray 153 (021002-001) R2 MS/MSD
   LCS

ANALYSIS

VX by GC/MS

One ash sample from JACADS was extracted and analyzed in triplicate as per request.

A five-point initial calibration curve was performed for VX with a range of 0.1 to 1.5 ng/uL and a mean response factor was calculated. The relative standard deviation was 15.5% for the quantitation ion 167m/z.

A continuing calibration standard CCS was injected before and after the sequence. The percent difference between the CCS and the mean response factor was within the criteria of less than 25% difference for each CCS.

A solvent blank and an LCS were carried through the extraction and cleanup process. The blank was below the lowest point of the calibration curve and the LCS had a recovery of 81%. For the blank and LCS, 2g arbitrarily was placed in the sample wt. column so that a direct comparison could be made between the blank, LCS and the samples.

Initially, the extracts were run directly. Significant background response was noted in the samples for observed ions 114m/z and 127m/z. The samples were reanalyzed omitting ions 114m/z and 127m/z; however, this did not improve the matrix problem. Another sequence was run using samples diluted 2 fold and instrument blanks injected between samples. This did not reduce background interference. A cleanup was performed on the original extracts. Aqueous extracts generated for LC/MS were filtered and extracted with methylene chloride. This cleanup step reduced the background interference and provided data for all SIM ions. The MS recovery was 88% and MSD recovery was 63%. Although the MSD response was outside of ± 35% and the RPD was outside ± 25%, results were deemed acceptable for VX identification.

VX was monitored using four ions 139, 167, 224,and 252m/z in the final cleanup extraction. The 167m/z ion was used for quantification. The 114 and 127m/z were not monitored due to their being associated with background response in initial extract analyses.

Sample Calculation: (213854 MS)

$$\text{Ug/Kg} = \frac{(\text{On Column Conc. ng/uL}) * (\text{Final Vol uL}) * \text{DF}}{(\text{sample weight g})}$$

$$\text{Ug/Kg} = \frac{(1.0749\ \text{ng/uL}) * (8919\text{uL}) * 2.5}{(2.0178\text{g})} = 11878\ \text{ng/g} \quad \text{or } 12000\ \text{ug/Kg}$$

"I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Director or his designee, as verified by the following signature. This report shall not be reproduced except in full, without the written approval of SwRI."

Jo Ann Boyd, Manager
Quality Assurance Unit, Division 01

10/25/02
Date

SOUTHWEST RESEARCH INSTITUTE
CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order No.: 23238
VTSR: October 7, 2002

CHAIN OF CUSTODY

C-O-C #680

3 26

United Engineers & Constructors
A Raytheon Company
Western Operations

CHAIN OF CUSTODY RECORD

PROJ. NO.

PROJ. NAME: JACADS - PMCSD/WDC

SAMPLERS: (SIGNATURE) Consolidated by ECD

SAMPLE STATION NO.	2002 DATE	TIME	DESIGNATE COMP	DESIGNATE GRAB	STATION DESCRIPTION	NO. OF SAMPLE CONTAINERS	SAMPLE TAG NUMBERS	VX	Direct LC/MS					REMARKS
TRAY 153	02OCT	AM		X	SDS Sludge (Solid)	1	021002-001	1	1					Sent per SwRI's request
														PM. Joe Brewer
														AiRbill # 006 277 83965
														Temp. 19.8°C, Jac002208
														Project # 05935.01.515
														Sample Rec'd time 11:50am
														Location, Ref. 91, Surety Lab.
														TAT 30 day as per R. Martinez

ORIGINAL

1. RELINQUISHED BY: (SIGNATURE)	DATE/TIME 2002 02OCT 0730	RECEIVED BY: (SIGNATURE & COMPANY) RTSC	2. RELINQUISHED BY: (SIGNATURE) MYLES OGA	DATE/TIME Oct 3 12:00	RECEIVED BY: (SIGNATURE & COMPANY) 12/7/02 11:50am
3. RELINQUISHED BY: (SIGNATURE)	DATE/TIME 12/7/02 1352	RECEIVED BY: (SIGNATURE & COMPANY)	4. RELINQUISHED BY: (SIGNATURE)	DATE/TIME	RECEIVED BY: (SIGNATURE & COMPANY)
5. RELINQUISHED BY: (SIGNATURE)	DATE/TIME	6. RECEIVED FOR LABORATORY BY: (SIGNATURE & COMPANY)	REMARKS: Sample was Received intact as per Russell Barajas.		DATE/TIME 12/7/02 1352

KEM 12/12/02

DISTRIBUTION: 1 - WHITE 2 - BLUE 3 - GREEN 4 - YELLOW 5 - PINK 6 - GOLDENROD

A1089195I

000003

SAMPLE LOG-IN SHEET

000004

Lab Name
Southwest Research Institute

Received By (Print Name) Khaled Edrisi

Received By (Signature) Khaled E

Page 1 of 1

Log-in Date
10/07/2002

Case Number
JACADS

Remarks 05935.03.001

Sample Delivery Group No.
213854

SAS Number
N/A

			Corresponding			Remarks Condition of Sample Shipment, etc
			EPA Sample #	Sample Tag #	Assigned Lab #	
1.	Custody Seal(s)	N/A Present/Absent* Intact/Broken	Tray 153 (021002-001)	021002-001	213854	Intact
2	Custody Seal Nos.	N/A				
3	Chain-of Custody Records	Present/Absent*				
4	Traffic Reports or Packing Lists	Present/Absent*				
5	Airbill	Airbill/Sticker Present/Absent*				
6.	Airbill No.	00627783965				
7.	Sample Tags	Present/Absent*				
	Sample Tag Numbers	Listed/Not listed on Chain of Custody				
8	Sample Condition	Intact/Broken*/ Leaking				
9	Cooler Temperature	19.8C				
10.	Does Information on custody records, traffic reports, and sample tags agree?	Yes/No*				
11.	Date Received at Lab	10/07/2002				
12	Time Received	11:50:00				

Sample Transfer

Fraction	Fraction
See attached	
Area # RF91 / Surety Lab	Area # 10/9/02
By RM/KE	By KE
On 10/7/02	On

* Contact SMO and attach record of resolution

Reviewed By MgPalomo

Date 10/14/02

Logbook No. Work Order (23238)

Logbook Page No. 4487 1 of 1

FORM DC-1

OLMO4.2

SOUTHWEST RESEARCH INSTITUTE
CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order No.: 23238
VTSR: October 7, 2002

STANDARD PREPARATION LOGS

TITLE JACADS
LOC: 23238

PROJECT NO. 5935.03.001
BOOK NO. 01.0500.007

45

Work continued from Page

VX Cleanup
EK 01.0500.009 pg 184

10/18/02

000005

Southwest Research Institute Semivolatile GC/MS Injection Log

OPERATOR: WSW SEQUENCE DATE: 18-Oct-2002 INSTRUMENT: Y-MD800
COLUMN: DB-1701 30M 0.32mm ID 1.0um FILM
CARRIER GAS: HELIUM HD PRESS: 70 kPA SOLVENT: 10%IPA/DCM
OVEN PROGRAM: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN
GC FILE: VX MS_FILE (SCN / SIR): VX DMV: 300

PROJECT NAME: JACADS PROJECT NUMBER: 05935.03.001
SDG: METHOD: Direct VX MATRIX: ASH

FILENAME	TIME	SAMPLE DESCRIPTION
Y1017223	23:10:33	2uL DFTPP Tune Check
Y1017224	00:56:20	2uL STD1 VX 0.1ng/uL, IS 0.1ng/uL
Y1017225	01:31:27	2uL STD2 VX 0.2ng/uL, IS 0.1ng/uL
Y1017226	02:06:46	2uL STD3 VX 0.5ng/uL, IS 0.1ng/uL
Y1017227	02:41:56	2uL STD4 VX 1.0ng/uL, IS 0.1ng/uL
Y1017228	03:17:11	2uL STD5 VX 1.5ng/uL, IS 0.1ng/uL
Y1017229	03:52:28	2uL CCS1 VX 1.0ng/uL, IS 0.1ng/uL
Y1017230	04:27:36	2uL Ext BLK
Y1017231	05:02:53	2uL Ext BLK MS W
Y1017232	05:37:58	2uL 213854 Tray153 ASH R1 W
Y1017233	06:13:05	2uL 213854 Tray153 ASH MS W
Y1017234	06:48:14	2uL 213854 Tray153 ASH R2 W
Y1017235	07:23:13	2uL 213854 Tray153 ASH MSD W
Y1017236	07:58:19	2uL 213854 Tray153 ASH R3 2X
Y1017237	08:33:08	2uL CCS2 VX 1.0ng/uL, IS 0.1ng/uL
Y1017238	09:07:52	2uL CCS2 VX 1.0ng/uL, IS 0.1ng/uL

Page 1

SCIENTIFIC BINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA

Work continued to Page

SIGNATURE

DISCLOSED TO AND UNDERSTOOD BY DATE

WITNESS DATE 10/18/02

DATE 10/18/02

180 TITLE Extraction and Analysis (VX Direct)
JACADS
WO: 23238

PROJECT NO. 05935.03.515
BOOK NO. 01.0500.009

Work continued from Page

Balance #30

ID		~~Final Vol~~ wt WSW 10/16/02	Final Vol (mL)	VX Spk Amt
BLANK		—	11 mL	—
BLANK MS (LCS)		—	11 mL	22 ug
213854	Tray 153 Rep1	2.0176	11 mL	—
213854	Tray 153 Rep2	2.0192	11 mL	—
213854	Tray 153 Rep3	2.0003	11 mL	—
213854	Tray 153 MS	2.0178	11 mL	22 ug
213854	Tray 153 MSD	2.0125	11 mL	22 ug

VX Spk 200 uL VX @ 110 ng/uL Lot # VX/IPA 9063 CTF-DIL

- Weighed ~2.0 g of ASH into 10 mL centrifuge vial
- Spiked VX in required samples
- Added approx 5.0 mL 10% IPA/DCM and Mixed thoroughly for 1 minute
- Centrifuged samples
- Filtered Extract(1) into 3 dram vial w/ Gelman Nylon Acrodisc 0.45 um
- Added approx 4.0 mL 10% IPA/DCM to the ASH and mixed for 1 minute
- Centrifuged Samples
- Filtered Extract(2) into the same 3 dram vial that Extract 1 is in.
- Added an additional 3.0 mL 10% IPA/DCM to the ASH and mixed for 1 min.
- Centrifuged Samples
- Filtered Extracts(3) into the same 3 dram vial that Extract 1+2 are in.
- Added 10% IPA/DCM to the Combined Filterate to get a Final Vol of 11 mL
-

10% IPA/DCM
25 mL Isopropyl Alcohol Fisher Lot # 010738 was brought to Final Vol of 250 mL w/ DCM Fisher Lot # 023244

SCIENTIFIC BINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA

Work continued to Page

SIGNATURE [signature] DATE 10/16/02

DISCLOSED TO AND UNDERSTOOD BY DATE

WITNESS [signature] DATE 10/16/02

000007

	AMT Added VX@10ng/uL	AMT Added IS@10ng/uL	Final Vol. (uL)	VX Conc. (ng/uL)	IS Conc. (ng/uL)
STD1	10	10	1000	0.10	0.10
STD2	20	10	1000	0.20	0.10
STD3	50	10	1000	0.50	0.10
STD4	100	10	1000	1.00	0.10
STD5	150	10	1000	1.50	0.10

IS Soln @10ng/uL BK:01-0500-009 pg116
VX@10ng/uL Activity Log 8 pg181
For each standard 1uL of Diisopropylaminoetanol, Aldrich Lot# 14222LI

Sample Prep for Extracts on pg 180

- Took 1mL of each sample and placed it in an autosampler Vial
- Spiked 10uL of IS soln @ 10ng/uL pg116
- Spiked 1uL of Di Isopropyl aminoethanol Aldrich LOT #14222LI

WSW

182 TITLE Extraction Prep (Direct VA) by LC/MS JACADS WO: 23238

PROJECT NO. 05935.03.001

BOOK NO. 01-0500-002

Work continued from Page

000008

Sample Prep for LC/MS from pg 180

- Took 2mL of each Sample and placed it in an 1 dram vial
- Using Nitrogen, The solvent was evaporated to about 100uL
- About 300uL of IPA was added to the sample.
- Again the solvent was evaporated to about 100uL, and
- About 200uL of IPA was added to the samples
- The samples were brought to a F.V. of 4.0mL with HPLC water.

- The MS, MSD and Blk Spike were diluted by a Factor of 2
- 200ul of sample was placed in vial and 18ul 30% formic acid was added.
- 50ul of sample was injected directly into LCMS.

LCMS mobile phase solvents..

Add 1ml of formic into 1L HPLC water.

Add 1ml of formic into 1L HPLC MeOH.

formic acid, 99% Acros #14793-0250, lot UN1779
Water, #W5-4 Fisher, lot 017763
MeOH, #A452-4, Fisher, lot 022932

SCIENTIFIC BINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA

Work continued to Page

SIGNATURE [signature] DATE 10/16/02

DISCLOSED TO AND UNDERSTOOD BY DATE

WITNESS [signature] DATE 10/16/02

TITLE Extract Dilution (GC/MS Sample Prep)
JACADS
W0123238

PROJECT NO. 05935.03.001

BOOK NO. 01-0500-009 SWRI

Work continued from Page

000008-A

Samples run on 10/16/02 adversely affected the GC/MS

I reprepared all samples. With the exception of the blank, they were all diluted by a factor of 2.

Blank
1mL of the blank was placed in an autosampler vial

Other Samples
500uL of 10% IPA/DCM was placed in an autosampler vial
500uL of sample was placed in an autosampler vial.

All samples
spiked 10uL of IS soln @ 10ng/uL pg 116
Spiked 1uL of DIAE Aldrich Lot 14222LI

In between each sample in the sequence an instrument Blank was placed to help keep the instrument stable.

WSW

SCIENTIFIC BINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA

Work continued to Page

SIGNATURE

DISCLOSED TO AND UNDERSTOOD BY DATE

WITNESS

DATE 10/17/02

DATE 10/17/02

184 TITLE Extraction Cleanup (GC/MS) Incads WO: 23238

PROJECT NO. 5935.03.001

BOOK NO. 01.0500.009

Work continued from Page

Because the original Extracts were so dirty, The GC/MS was adversely affected. The following procedure was used to clean the samples up for adequate analysis.

The Water Extracts from pg 182 were used for this clean up. (Original Volume was 4.0 mL, but some of the extract was used for LCMS analysis.

- Filtered the aqueous extract through a Gelman 0.45um Nylon acrodisk.
- Added 1 drop of 1N NaOH to ~~the~~ 3.7 mL of the filtered Sample.
- Extracted the Aqueous Soln w/ 1.5mL of DCM and 2 microspa of NaCl
- The DCM layer was collected.
- Samples were prepared for injection w/ Necessary dilutions

Sample ID	Samp Vol	Solv Vol	DIAE spk	IS Soln @10ng/uL	DF
BIK	1000uL	-0-	1uL	10uL	1
BIK MS	500uL	500uL	↓	↓	2
R1	1000uL	-0-			1
R2	1000uL	-0-			1
R3	1000uL	-0-			1
MS	400uL	600uL			2.5
MSD	400uL	600uL			2.5

2g Sample → 1mL DCM → 2mL aliquot DCM → Solvent Exchanged 4mL H_2O → LCMS Analysis; 3.7mL aliquot → Filtered Extracted 1.5mL DCM → GC/MS Samp

Effective Vol 8.919mL

Work continued to Page

SIGNATURE [signature] DATE 10/17/02

DISCLOSED TO AND UNDERSTOOD BY DATE

WITNESS Robert A. Martinez DATE 10/17/02

SOUTHWEST RESEARCH INSTITUTE
CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order No.: 23238
VTSR: October 7, 2002

INJECTION LOGS

Southwest Research Institute
Semivolatile GC/MS Injection Log

000010

OPERATOR: WSW SEQUENCE DATE: 18-Oct-2002 INSTRUMENT: Y-MD800
COLUMN: DB-1701 30M 0.32mm ID 1.0um FILM
CARRIER GAS: HELIUM HD PRESS: 70 kPA SOLVENT: 10%IPA/DCM
OVEN PROGRAM: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN
GC FILE: VX MS_FILE (SCN / SIR): VX DMV: 300

PROJECT NAME: JACADS PROJECT NUMBER: 05935.03.001
SDG: METHOD: Direct VX MATRIX: ASH

FILENAME	TIME	SAMPLE DESCRIPTION
Y1017223	23:10:33	2uL DFTPP Tune Check
Y1017224	00:56:20	2uL STD1 VX 0.1ng/uL, IS 0.1ng/uL
Y1017225	01:31:27	2uL STD2 VX 0.2ng/uL, IS 0.1ng/uL
Y1017226	02:06:46	2uL STD3 VX 0.5ng/uL, IS 0.1ng/uL
Y1017227	02:41:56	2uL STD4 VX 1.0ng/uL, IS 0.1ng/uL
Y1017228	03:17:11	2uL STD5 VX 1.5ng/uL, IS 0.1ng/uL
Y1017229	03:52:28	2uL CCS1 VX 1.0ng/uL, IS 0.1ng/uL
Y1017230	04:27:36	2uL Ext BLK
Y1017231	05:02:53	2uL Ext BLK MS W
Y1017232	05:37:58	2uL 213854 Tray153 ASH R1 W
Y1017233	06:13:05	2uL 213854 Tray153 ASH MS W
17234	06:48:14	2uL 213854 Tray153 ASH R2 W
Y1017235	07:23:13	2uL 213854 Tray153 ASH MSD W
Y1017236	07:58:19	2uL 213854 Tray153 ASH R3 2X
Y1017237	08:33:08	2uL CCS2 VX 1.0ng/uL, IS 0.1ng/uL
Y1017238	09:07:52	2uL CCS2 VX 1.0ng/uL, IS 0.1ng/uL

SOUTHWEST RESEARCH INSTITUTE
CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order No.: 23238
VTSR: October 7 2002

GC/MS ANALYSIS

SOUTHWEST RESEARCH INSTITUTE
CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order No.: 23238
VTSR: October 7, 2002

QC SUMMARY

SOUTHWEST RESEARCH INSTITUTE

Semi-volatile Organics Spike/Spike Duplicate Recovery

Project: 05935.03.001
Sample ID: Tray 153 (021002-001) R2
Instrument: Y-MD800

Case: JACADS
MS File: Y1017233

SDG: 213854
MSD File: Y1017235

Compound	Spike Added (ug/Kg)	Sample Concentration (ug/Kg)	MS Concentration (ug/Kg)	MS %Rec	QC Limit Rec
VX	11000	2300	12000	88	60-140

Compound	Spike Added (ug/Kg)	MSD Concentration (ug/Kg)	MSD %Rec	%RPD	QC Limits	
					RPD	REC
VX	11000	9200	63	*34* *	25	60-140

* Values outside of QC Limits

RPD: 1 of 1 outside limits
Spike Recovery: 0 of 2 outside limits

SOUTHWEST RESEARCH INSTITUTE

Semi-volatile Organics Laboratory Control Spike/Duplicate Recovery

Project: 05935.03.001 Case: JACADS SDG: 213854
Sample ID: BLANK MS LCS File: Y1017231
Instrument: Y-MD800

Compound	LCS Conc (ug/Kg)	LCS Recovery (ug/Kg)	LCS %Rec	QC Limit Rec
VX	11000	8900	81	60-140

* Values outside of QC Limits

Recovery: 0 of 1 outside limits

SOUTHWEST RESEARCH INSTITUTE

Blank Summary

Blank ID: BLANK (10/16/02)

Project: 05935.03.001 | Case: JACADS | SDG: 213854

Instrument: Y-MD800 | Filename: Y1017230 | Analyzed: Oct 18 2002 4:27AM

This Blank Applies to the Following Samples

	Sample ID	SwRI ID	Lab File ID	Analyzed
1	BLANK MS LCS		Y1017231	Oct 18 2002 5:02AM
2	Tray 153 (021002-001) R1	213854	Y1017232	Oct 18 2002 5:37AM
3	Tray 153 (021002-001) R2	213854	Y1017234	Oct 18 2002 6:48AM
4	Tray 153 (021002-001) R2 MS	213854	Y1017233	Oct 18 2002 6:13AM
5	Tray 153 (021002-001) R2 MSD	213854	Y1017235	Oct 18 2002 7:23AM
6	Tray 153 (021002-001) R3	213854	Y1017236	Oct 18 2002 7:58AM

SOUTHWEST RESEARCH INSTITUTE
Internal Standard Area and RT Summary

010004

Project: 05935.03.001 | Case: JACADS | SDG: 213854
Instrument: Y-MD800 | Filename: Y1017229 | Analyzed: Oct 18 2002 3:52AM

	IS1 (HCB) Area	RT				
12 Hour STD	905369	15.55				
Upper Limit	1810738	16.05				
Lower Limit	452684	15.05				
CLIENT SAMPLE NO.						
BLANK (10/16/02)	897582	15.60				
BLANK MSLCS	864023	15.57				
Tray 153 (021002-001) R1	873520	15.60				
Tray 153 (021002-001) R2	855571	15.60				
Tray 153 (021002-001) R2MS	854364	15.60				
Tray 153 (021002-001) R2MSD	837573	15.56				
Tray 153 (021002-001) R3	850480	15.60				

IS1 (HCB) = Hexachlorobenzene

AREA UPPER LIMIT = +100% of internal standard area
AREA LOWER LIMIT = -50% of internal standard area
RT UPPER LIMIT = +0.50 minutes of internal standard RT
RT LOWER LIMIT = -0.50 minutes of internal standard RT

* Values outside of QC limits

SOUTHWEST RESEARCH INSTITUTE
CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order No.: 23238
VTSR: October 7, 2002

SAMPLE DATA

SOUTHWEST RESEARCH INSTITUTE

Semi-volatile Organics Data Reporting Form

Client: JACADS
Case: JACADS
SDG: 213854
Matrix: Solid
Sample wt. (g): 2.0176
Injection Volume (uL): 2.0

Client Sample ID: Tray 153 (021002-001) R1
System ID: 213854
Filename: Y1017232
pH: NA
Conc. Ext. Vol. (uL): 8919

Project Number: 05935.03.001
Instrument: Y-MD800
Date Received: 10/07/2002
Date Extracted: Oct 16, 2002
Date Analyzed: Oct 18 2002 5:37AM
Dilution Factor: 1

CAS No.	Compound	ug/Kg
50782-69-9	VX	2700

DATA REPORTING QUALIFIERS

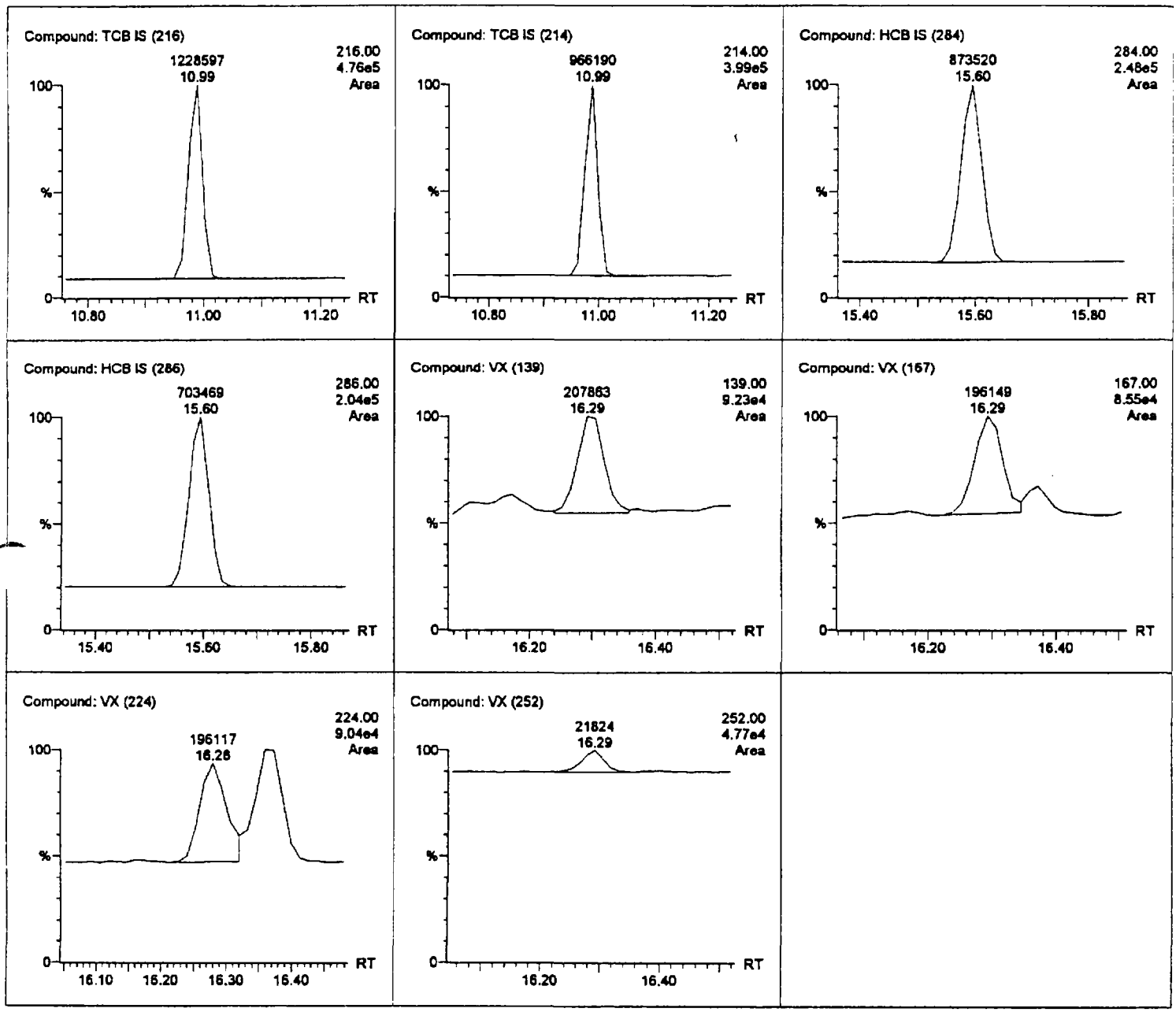
- B This flag is used when the analyte is found in the blank as well as the sample.
- D Concentration value is from dilution analysis.
- E This flag indicates compounds whose concentrations exceed the calibration range.
- J Indicates an estimated value.
- U Indicates compound was analyzed for, but not detected. Report the minimum detection limit for the sample with U (e.g. 10U) based on necessary concentration dilution action (This is not necessarily the instrument detection limit).

Southwest Research Institute
Agent (VX) Quantitation Report

010006

..ename: C:\MASSLAB\DATA\Y1017232.RAW
Sample ID: 2uL 213854 Tray153 ASH R1 W
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 18-Oct-2002 at 05:37:58
Instrument: Y-MD800



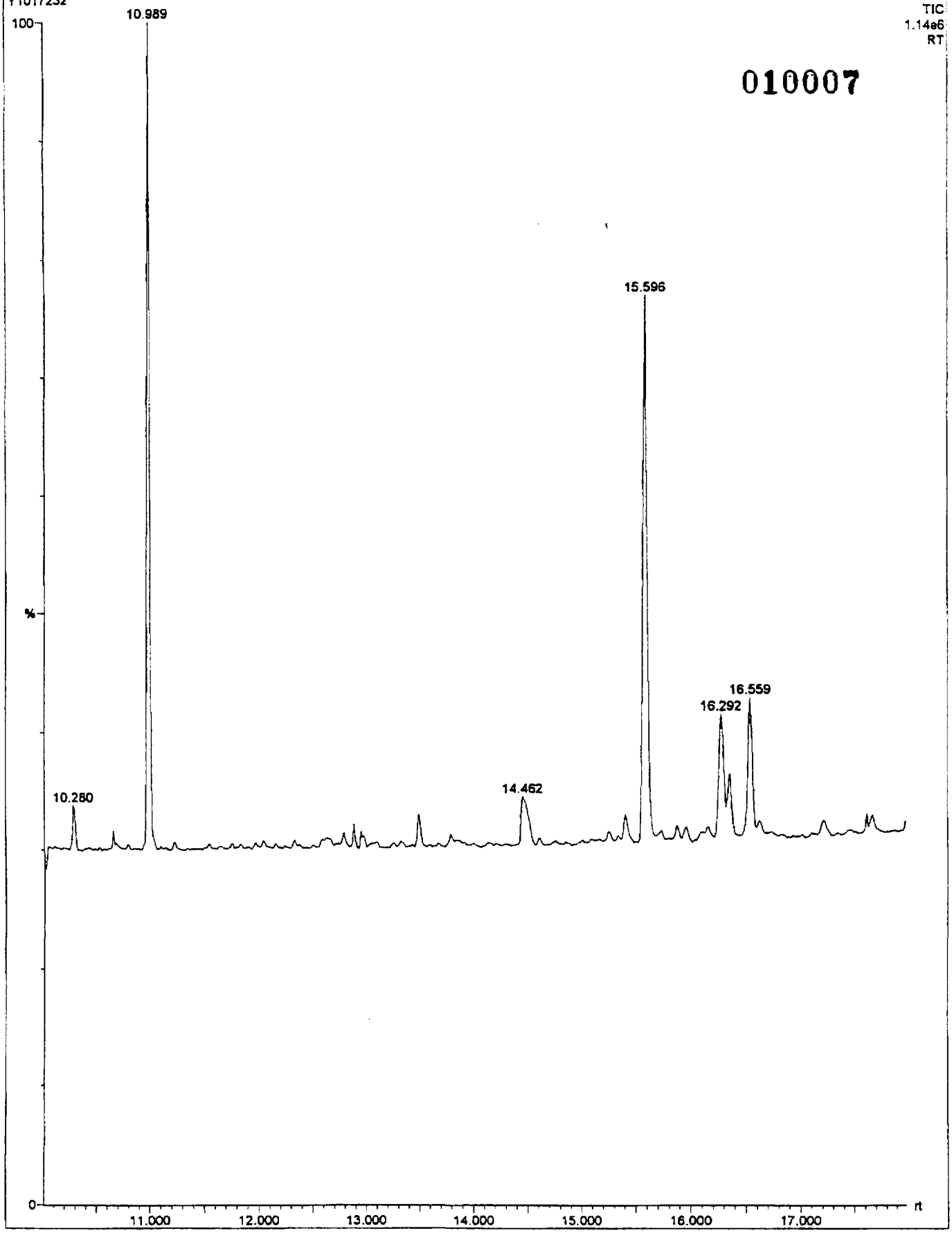
Peak List Information - Y1017232

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.989	IS	1228597	433416	0.1000
2	TCB IS (214)	10.989	IS	966190	357400	0.1000
3	HCB IS (284)	15.596	IS	873520	206263	0.1000
4	HCB IS (286)	15.596	IS	703469	162621	0.1000
5	VX (139)	16.292		207863	41651	0.5093
6	VX (167)	16.292		196149	38861	0.6149
	VX (224)	16.279		196117	41816	10.3182
8	VX (252)	16.292		21824	4925	0.4422

Sample ID: 2uL 213854 Tray153 ASH R1 W
Y1017232

Acquired on 18-Oct-2002 at 05:37:58
SIR of 8 Channels EI+
TIC
1.14e6
RT

010007



SOUTHWEST RESEARCH INSTITUTE

Semi-volatile Organics Data Reporting Form

Client: JACADS
Case: JACADS
SDG: 213854
Matrix: Solid
Sample wt. (g): 2.0192
Injection Volume (uL): 2.0

Client Sample ID: Tray 153 (021002-001) R2
System ID: 213854
Filename: Y1017234
pH: NA
Conc. Ext. Vol. (uL): 8919

Project Number: 05935.03.001
Instrument: Y-MD800
Date Received: 10/07/2002
Date Extracted: Oct 16, 2002
Date Analyzed: Oct 18 2002 6:48AM
Dilution Factor: 1

CAS No.	Compound	ug/Kg
50782-69-9	VX	2300

DATA REPORTING QUALIFIERS

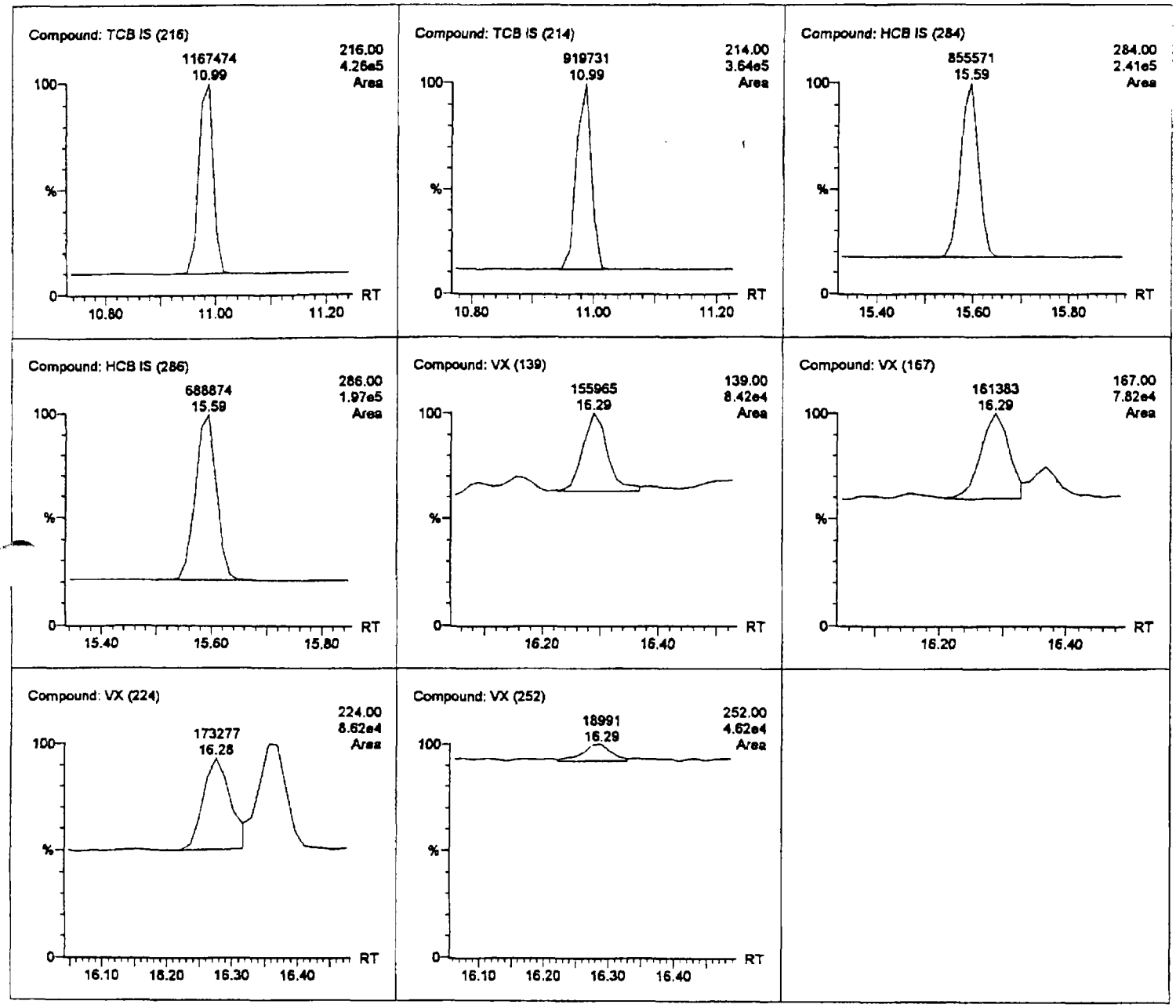
- B This flag is used when the analyte is found in the blank as well as the sample.
- D Concentration value is from dilution analysis.
- E This flag indicates compounds whose concentrations exceed the calibration range.
- J Indicates an estimated value.
- U Indicates compound was analyzed for, but not detected. Report the minimum detection limit for the sample with U (e.g. 10U) based on necessary concentration dilution action (This is not necessarily the instrument detection limit).

Southwest Research Institute
Agent (VX) Quantitation Report

010009

ɔname: C:\MASSLAB\DATA\Y1017234.RAW
Sample ID: 2uL 213854 Tray153 ASH R2 W
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 18-Oct-2002 at 06:48:14
Instrument: Y-MD800



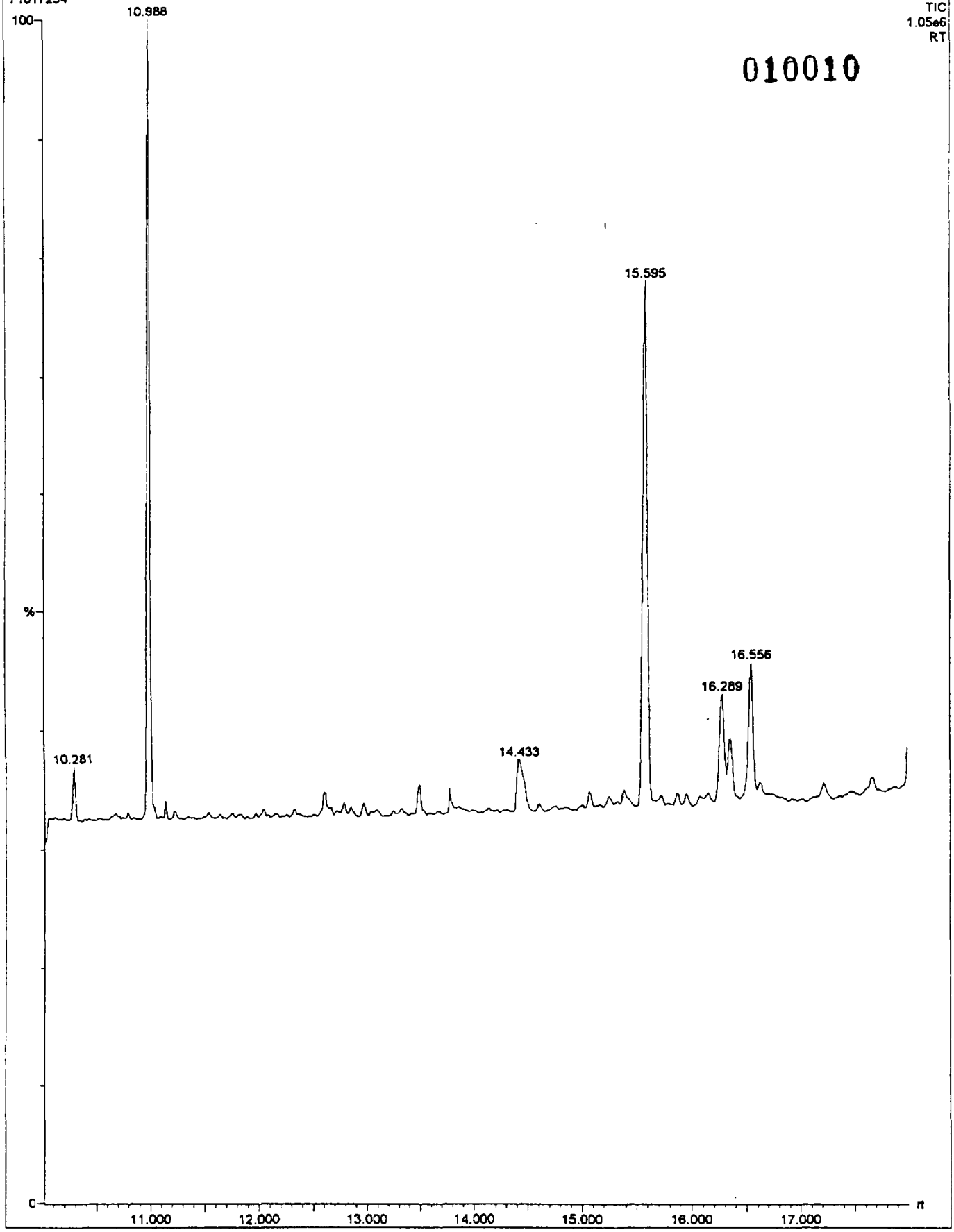
Peak List Information - Y1017234

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.988	IS	1167474	383072	0.1000
2	TCB IS (214)	10.988	IS	919731	322237	0.1000
3	HCB IS (284)	15.595	IS	855571	198595	0.1000
4	HCB IS (286)	15.595	IS	688874	155616	0.1000
5	VX (139)	16.289		155965	31365	0.3902
6	VX (167)	16.289		161383	31735	0.5165
	VX (224)	16.275		173277	36995	9.3078
	VX (252)	16.289		18991	3620	0.3929

Sample ID: 2uL 213854 Tray153 ASH R2 W
Y1017234

Acquired on 18-Oct-2002 at 06:48:14
SIR of 8 Channels EI+
TIC
1.05e6
RT

010010



SOUTHWEST RESEARCH INSTITUTE

Semi-volatile Organics Data Reporting Form

Client: JACADS
Case: JACADS
SDG: 213854
Matrix: Solid
Sample wt. (g): 2.0003
Injection Volume (uL): 2.0

Client Sample ID: Tray 153 (021002-001) R3
System ID: 213854
Filename: Y1017236
pH: NA
Conc. Ext. Vol. (uL): 8919

Project Number: 05935.03.001
Instrument: Y-MD800
Date Received: 10/07/2002
Date Extracted: Oct 16, 2002
Date Analyzed: Oct 18 2002 7:58AM
Dilution Factor: 1

CAS No.	Compound	ug/Kg
50782-69-9	VX	1900

DATA REPORTING QUALIFIERS

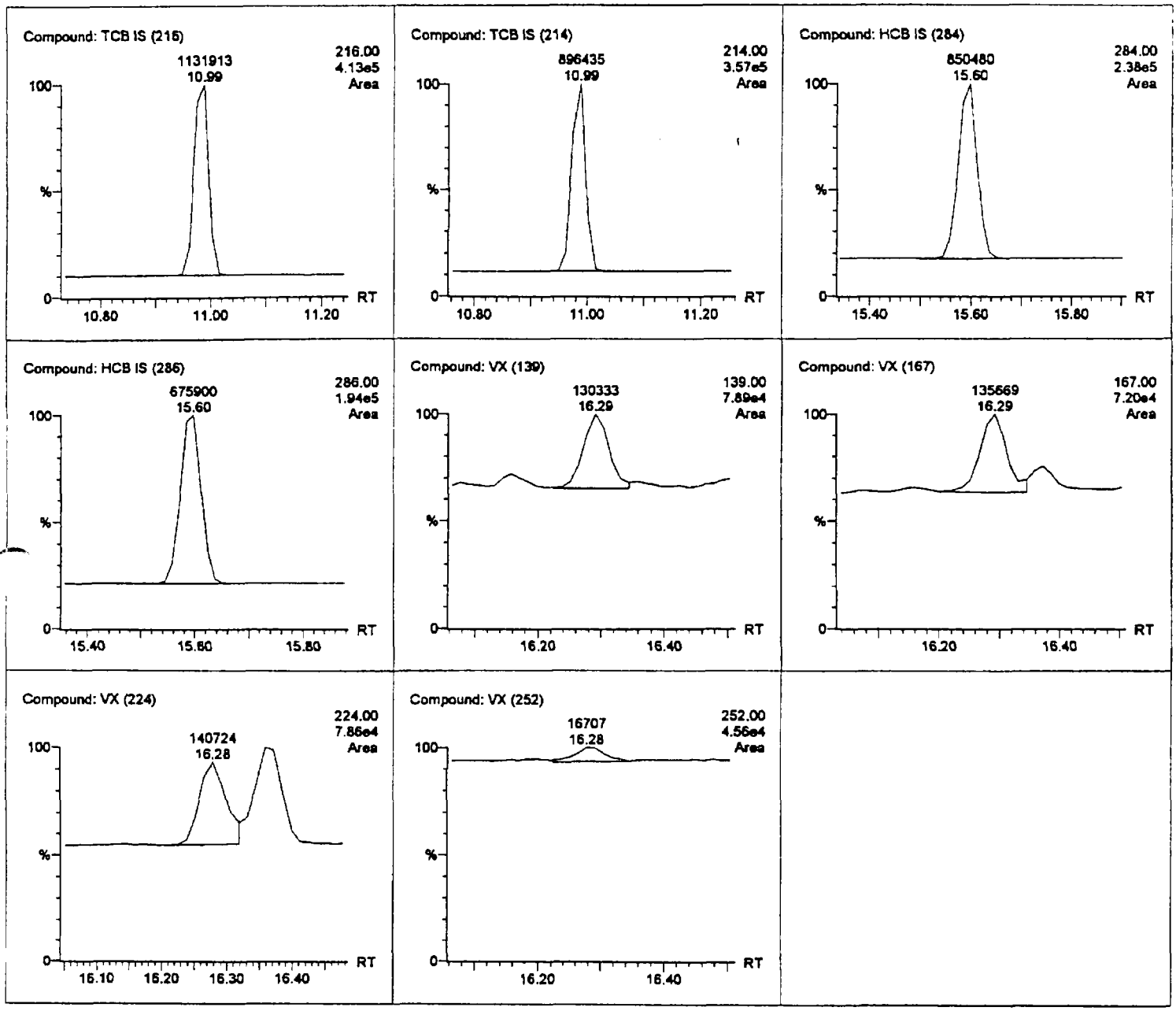
- B This flag is used when the analyte is found in the blank as well as the sample.
- D Concentration value is from dilution analysis.
- E This flag indicates compounds whose concentrations exceed the calibration range.
- J Indicates an estimated value.
- U Indicates compound was analyzed for, but not detected. Report the minimum detection limit for the sample with U (e.g. 10U) based on necessary concentration dilution action (This is not necessarily the instrument detection limit).

Southwest Research Institute
Agent (VX) Quantitation Report

010012

ɜname: C:\MASSLAB\DATA\Y1017236.RAW
Sample ID: 2uL 213854 Tray153 ASH R3 2X
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 18-Oct-2002 at 07:58:19
Instrument: Y-MD800



Peak List Information - Y1017236

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.988	IS	1131913	370280	0.1000
2	TCB IS (214)	10.988	IS	896435	315594	0.1000
3	HCB IS (284)	15.597	IS	850480	196084	0.1000
4	HCB IS (286)	15.597	IS	675900	151901	0.1000
5	VX (139)	16.291		130333	27594	0.3280
6	VX (167)	16.291		135669	26445	0.4368
	VX (224)	16.278		140724	30029	7.6044
-	VX (252)	16.278		16707	3034	0.3477

Sample ID: 2uL 213854 Tray153 ASH R3 2X

Acquired on 18-Oct-2002 at 07:58:19

Y1017236

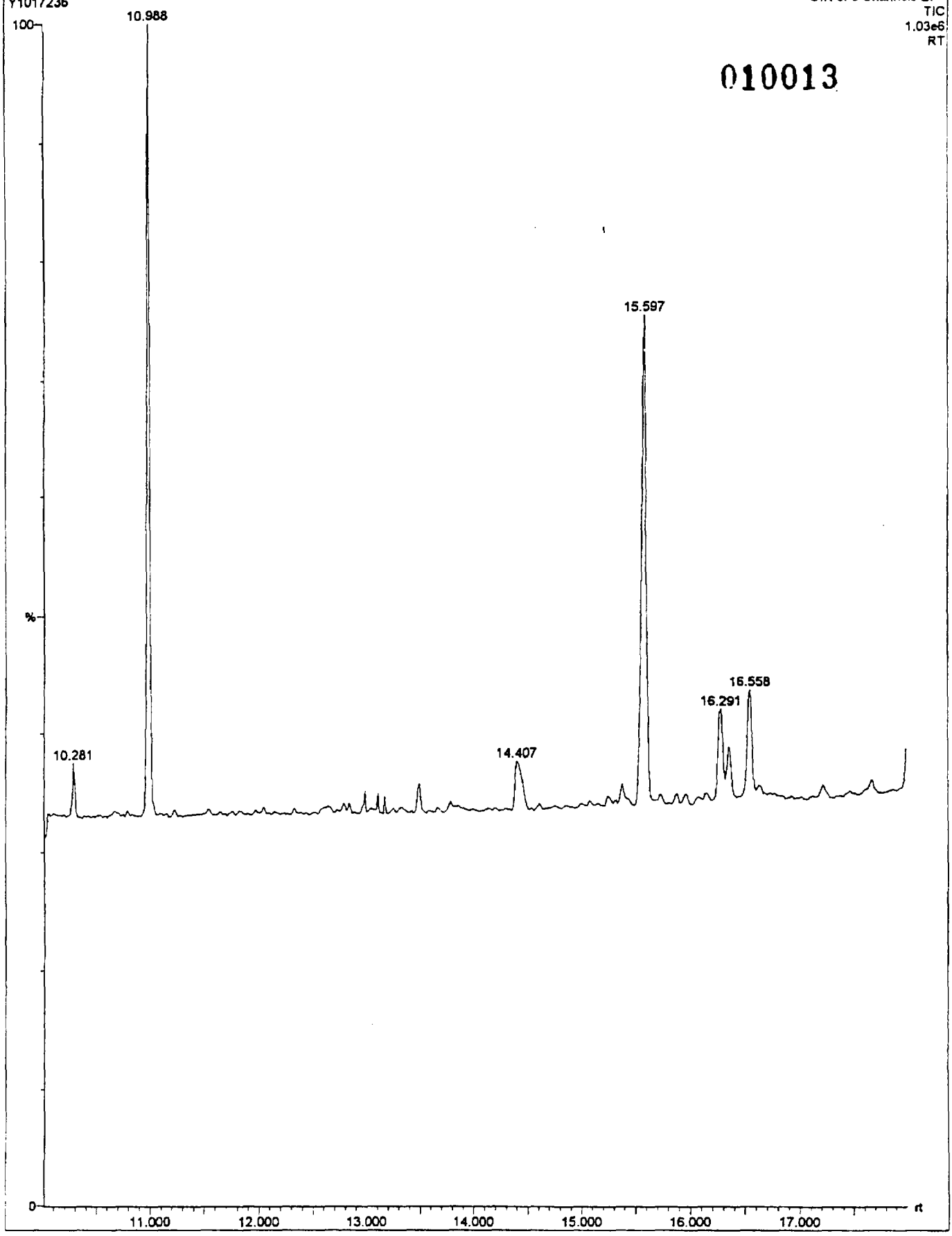
SIR of 8 Channels EI+

TIC

1.03e6

RT

010013



SOUTHWEST RESEARCH INSTITUTE
CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order No.: 23238
VTSR: October 7, 2002

STANDARDS DATA

SOUTHWEST RESEARCH INSTITUTE

Instrument Performance Check

Decafluorotriphenylphosphine (DFTPP)

010014

Project: 05935.03.001 | Case: JACADS | SDG: 213854

File ID: Y1017223 | DFTPP Injection Date: 10/17/02 | DFTPP Injection Time: 23:10:00

Instrument: Y-MD800 | GC Column: DB-1701 30m X 0.32mm ID

m/e	Ion Abundance Criteria	% Relative Abundance	
51	30.0 - 80.0% of mass 198	42.8	
68	Less than 2.0% of mass 69	0.0	0.0(1)
69	Mass 69 relative abundance	40.1	
70	Less than 2.0% of Mass 69	0.3	0.8(1)
127	25.0 - 75.0% of mass 198	44.7	
197	Less than 1.0% of mass 198	0.0	
198	Base Peak, 100% relative abundance	100.0	
199	5.0 - 9.0% of mass 198	6.6	
275	10.0 - 30.0% of mass 198	19.2	
365	Greater than 0.75% of mass 198	1.4	
441	Present, but less than mass 443	11.2	
442	40.0 - 110.0% of mass 198	76.1	
443	15.0 - 24.0% of mass 442	14.6	19.1(2)

1. Value is % of Mass 69 2. Value is % of Mass 442

	This check applies to the following samples				
	Sample No.	SwRI ID	File ID	Date Analyzed	Time Analyzed
1	STD1		Y1017224	10/18/02	00:56:00
2	STD2		Y1017225	10/18/02	01:31:00
3	STD3		Y1017226	10/18/02	02:06:00
4	STD4		Y1017227	10/18/02	02:41:00
5	STD5		Y1017228	10/18/02	03:17:00
6	CCS1		Y1017229	10/18/02	03:52:00
7	BLANK (10/16/02)		Y1017230	10/18/02	04:27:00
8	BLANK MSLCS		Y1017231	10/18/02	05:02:00
9	Tray 153 (021002-001) R1	213854	Y1017232	10/18/02	05:37:00
10	Tray 153 (021002-001) R2MS	213854 MS	Y1017233	10/18/02	06:13:00
11	Tray 153 (021002-001) R2	213854	Y1017234	10/18/02	06:48:00
12	Tray 153 (021002-001) R2MSD	213854 MSD	Y1017235	10/18/02	07:23:00
13	Tray 153 (021002-001) R3	213854	Y1017236	10/18/02	07:58:00
14	CCS2		Y1017237	10/18/02	08:33:00

Finnigan MD Family GC/MS Data System

MassLab v1.400

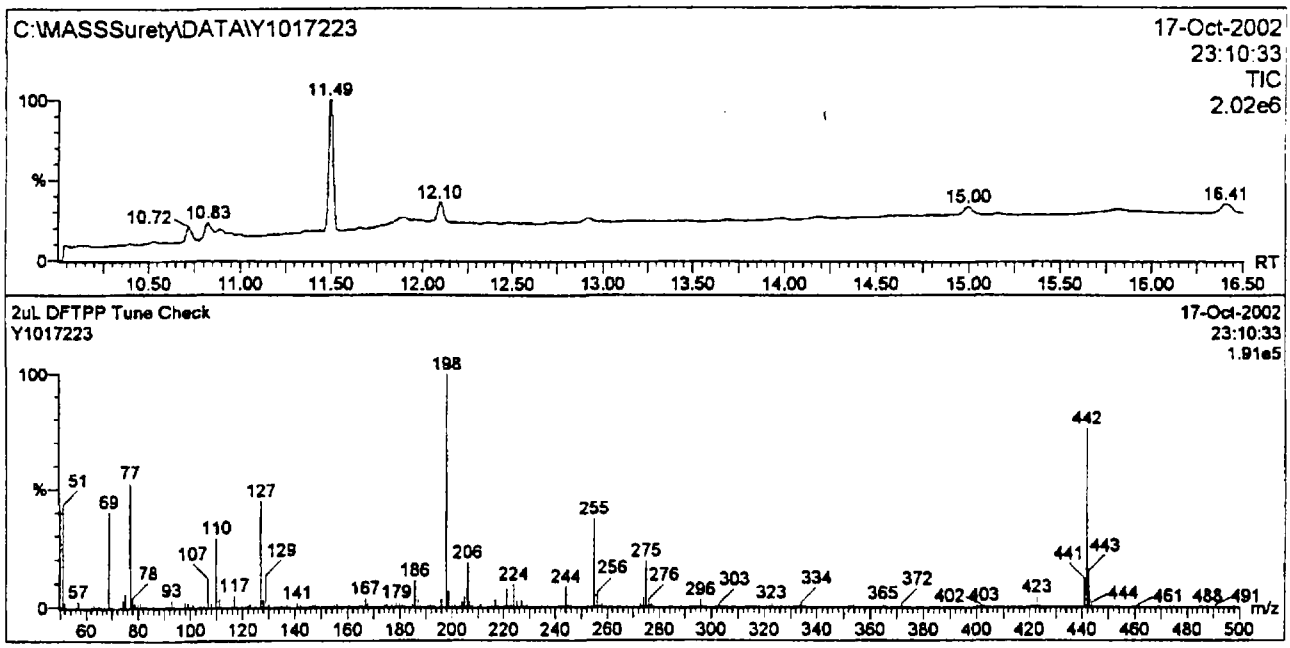
010015

DFTPP Tune Report

Datafile Name : C:\MASSLAB\DATA\Y1017223 Acquisition Date : 17-Oct-2002 23:10:33

Sample : 2uL DFTPP Tune Check Report Date : 17-Oct-2002 23:34:06

Scan Number : 179 To 181 - 170

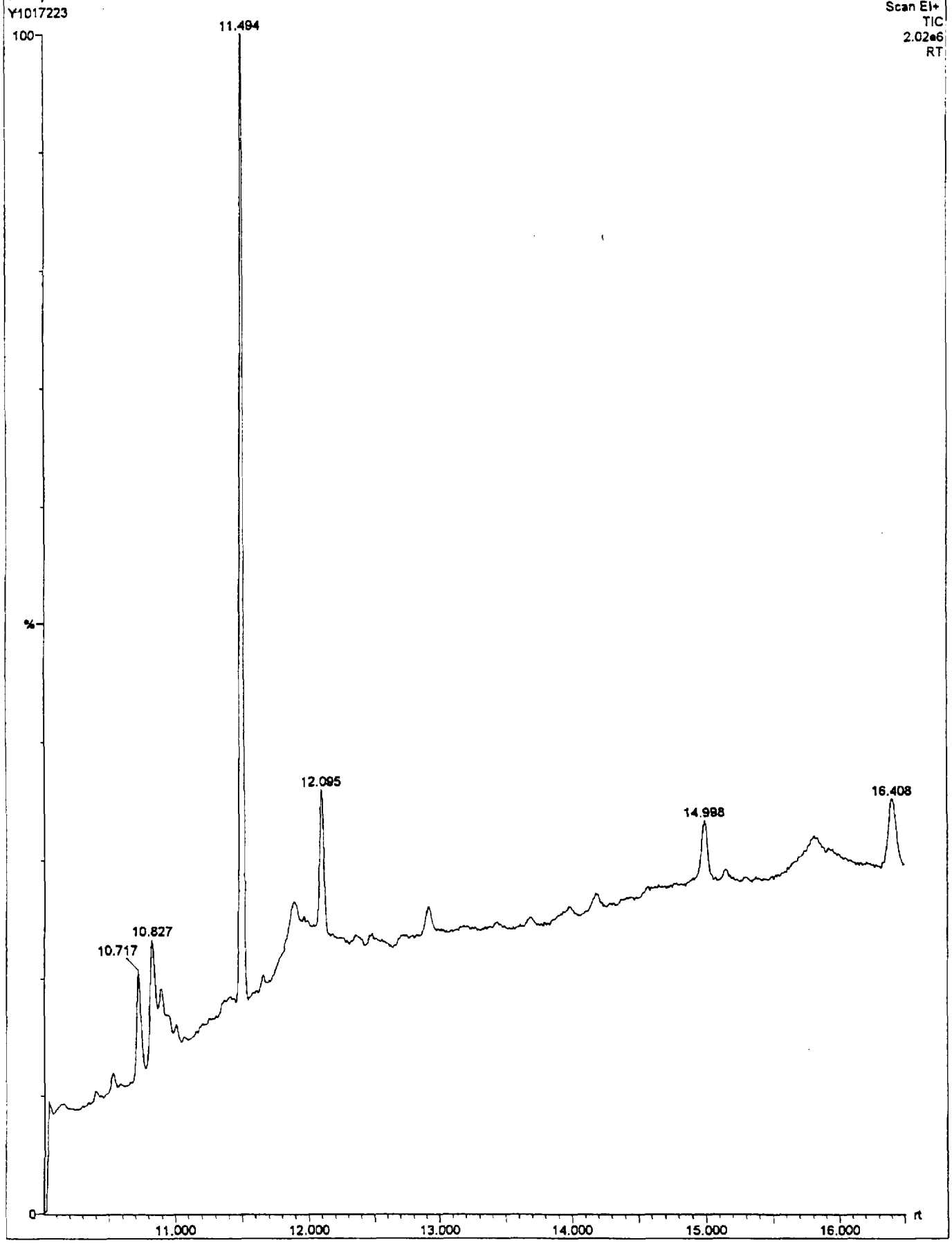


Mass	Criteria	Intensity	
51	30.0 - 80.0 percent of mass 198	42.79	
68	Less than 2.0 percent of mass 69	0.00	(0.00)
69	Present	40.06	
70	Less than 2.0 percent of mass 69	0.33	(0.82)
127	25.0 - 75.0 percent of mass 198	44.66	
197	Less than 1.0 percent of mass 198	0.00	
198	Base peak, 100 percent relative abundance	100.00	
199	5.0 - 9.0 percent of mass 198	6.55	
275	10.0 - 30.0 percent of mass 198	19.16	
365	Greater than 0.75 percent of mass 198	1.38	
441	Present but less than mass 443	11.20	
442	40.0 - 110.0 percent of mass 198	76.06	
443	15.0 - 24.0 percent of mass 442	14.56	(19.14)

Tune Check Passed

Sample ID: 2uL DFTPP Tune Check
Y1017223

Acquired on 17-Oct-2002 at 23:10:33
Scan EI+
TIC
2.02e6
RT



Southwest Research Institute

Semi-volatile Organics Initial Calibration Data

010017

Project: 05935.03.001 Case: JACADS SDG: 213854

Instrument: Y-MD800 GC Column: DB-1701 30m X 0.32mm ID

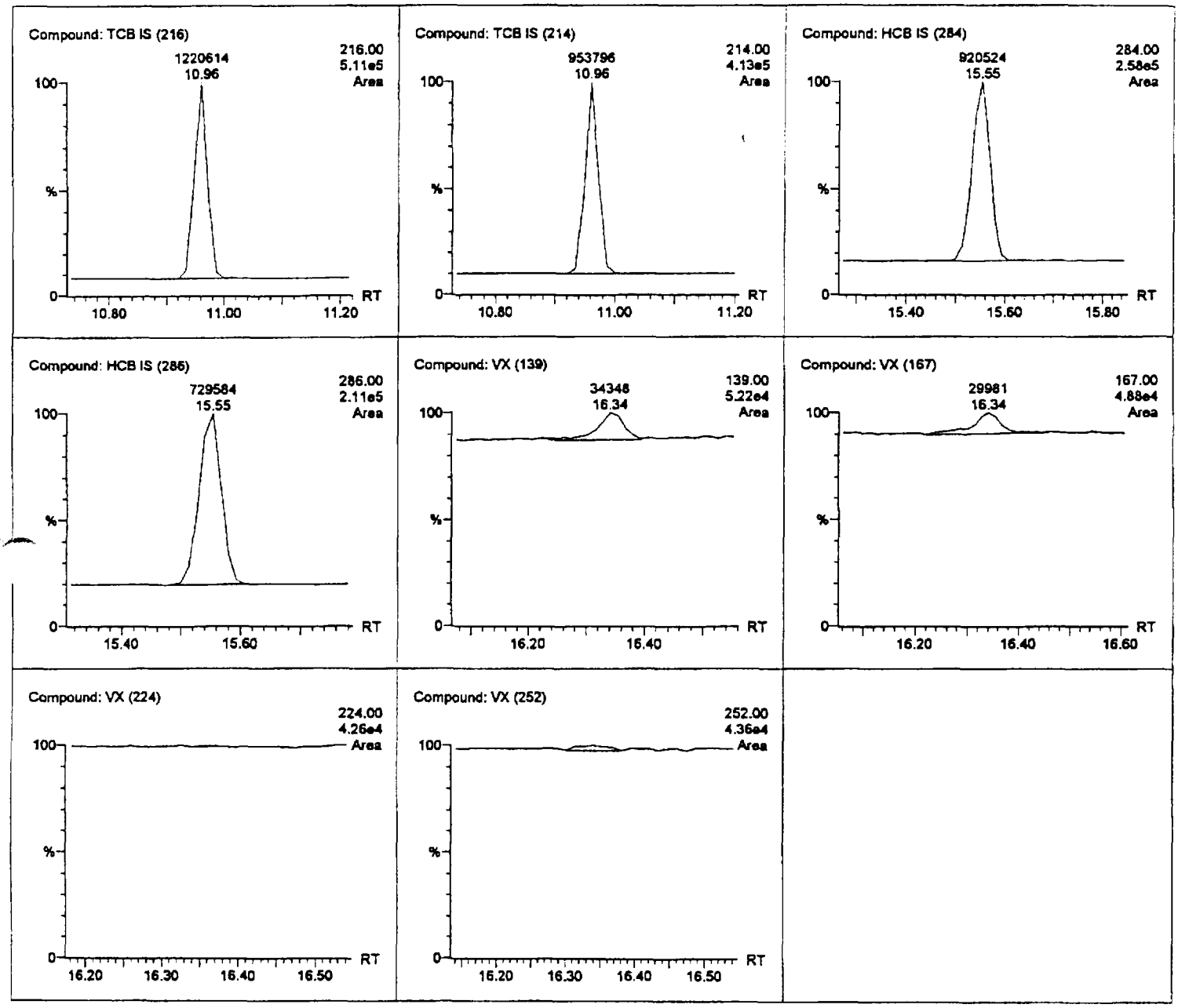
	RRF0.100	RRF0.200	RRF0.500	RRF1	RRF1.500		
Analysis Date	10/18/02	10/18/02	10/18/02	10/18/02	10/18/02		
Analysis Time	00:56:00	01:31:00	02:06:00	02:41:00	03:17:00		
Lab File ID	Y1017224	Y1017225	Y1017226	Y1017227	Y1017228		
Compound	**RRF0.100**	**RRF0.200**	**RRF0.500**	**RRF1**	**RRF1.500**	**Mean**	**% RSD**
VX	0.033	0.031	0.035	0.038	0.045	0.037	15.5

Southwest Research Institute
Agent (VX) Quantitation Report

010018

..ename: C:\MASSLAB\DATA\Y1017224.RAW
Sample ID: 2uL STD1 VX 0.1ng/uL, IS 0.1ng/uL
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 18-Oct-2002 at 00:56:20
Instrument: Y-MD800



Peak List Information - Y1017224

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.961	IS	1220614	468752	0.1000
2	TCB IS (214)	10.961	IS	953796	371603	0.1000
3	HCB IS (284)	15.554	IS	920524	216124	0.1000
4	HCB IS (286)	15.554	IS	729584	169930	0.1000
5	VX (139)	16.341		34348	6530	0.0799
	VX (167)	16.341		29981	4803	0.0892
	VX (224)	16.355		545	252	0.0272
6	VX (252)	16.341		6156	1135	0.1184

Sample ID: 2uL STD1 VX 0.1ng/uL, IS 0.1ng/uL

Acquired on 18-Oct-2002 at 00:56:20

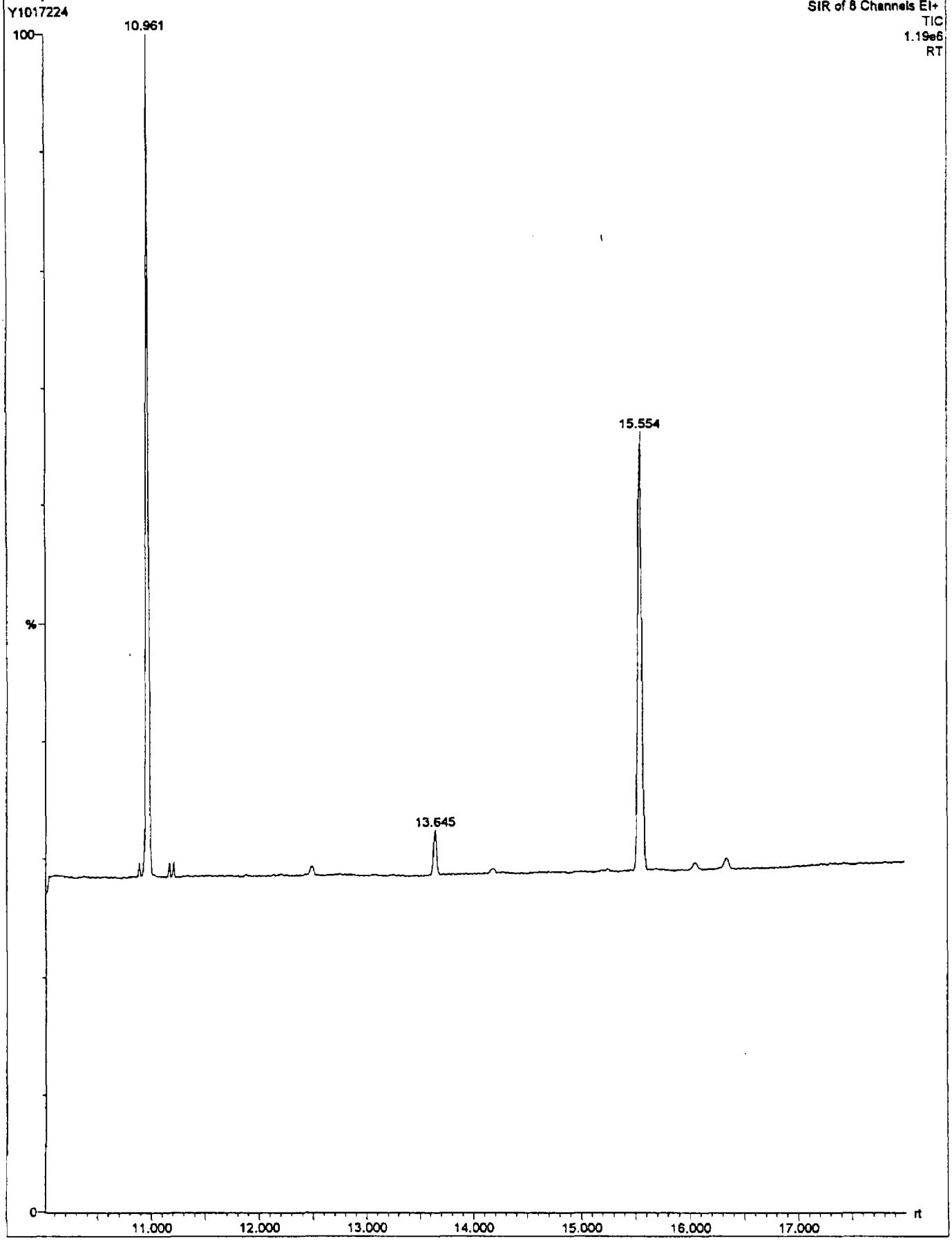
Y1017224

SIR of 8 Channels EI+

TIC

1.19e6

RT

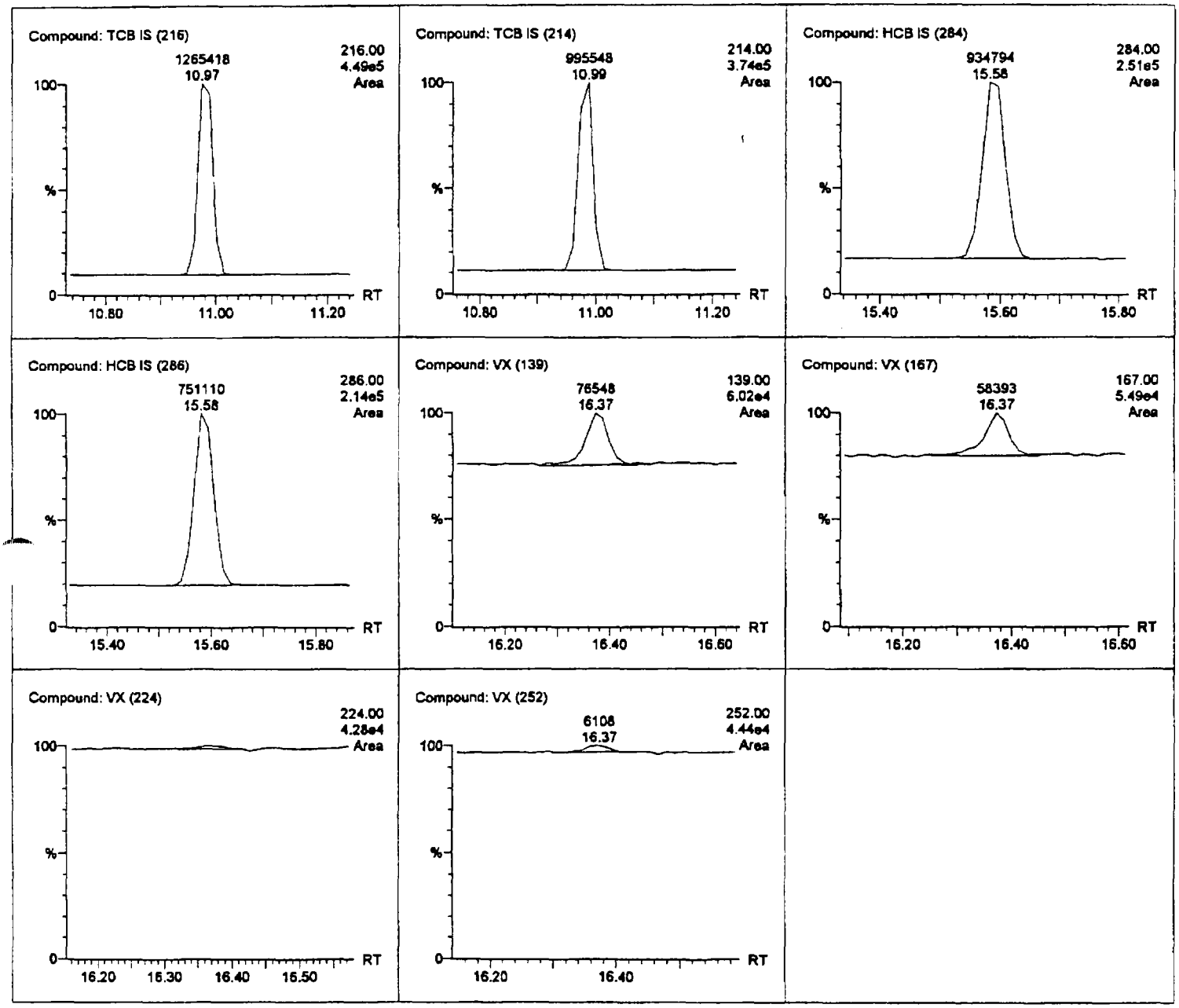


Southwest Research Institute
Agent (VX) Quantitation Report

010020

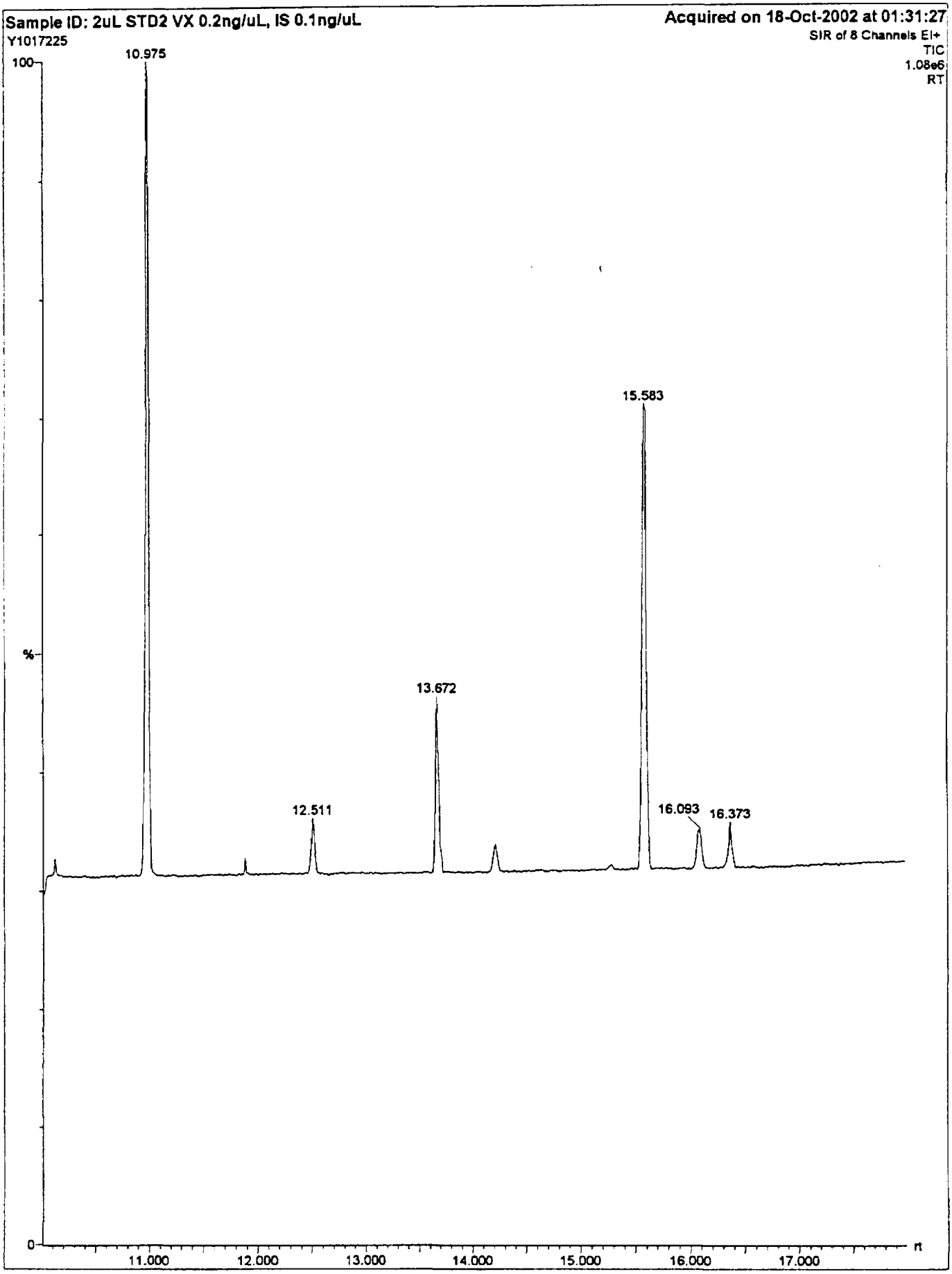
. ..ename: C:\MASSLAB\DATA\Y1017225.RAW
Sample ID: 2uL STD2 VX 0.2ng/uL, IS 0.1ng/uL
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 18-Oct-2002 at 01:31:27
Instrument: Y-MD800



Peak List Information - Y1017225

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.975	IS	1265418	406741	0.1000
2	TCB IS (214)	10.988	IS	995548	332200	0.1000
3	HCB IS (284)	15.583	IS	934794	209163	0.1000
4	HCB IS (286)	15.583	IS	751110	172258	0.1000
5	VX (139)	16.373		76548	14753	0.1753
	VX (167)	16.373		58393	11062	0.1711
	VX (224)	16.360		3165	597	0.1556
6	VX (252)	16.373		6108	1396	0.1156

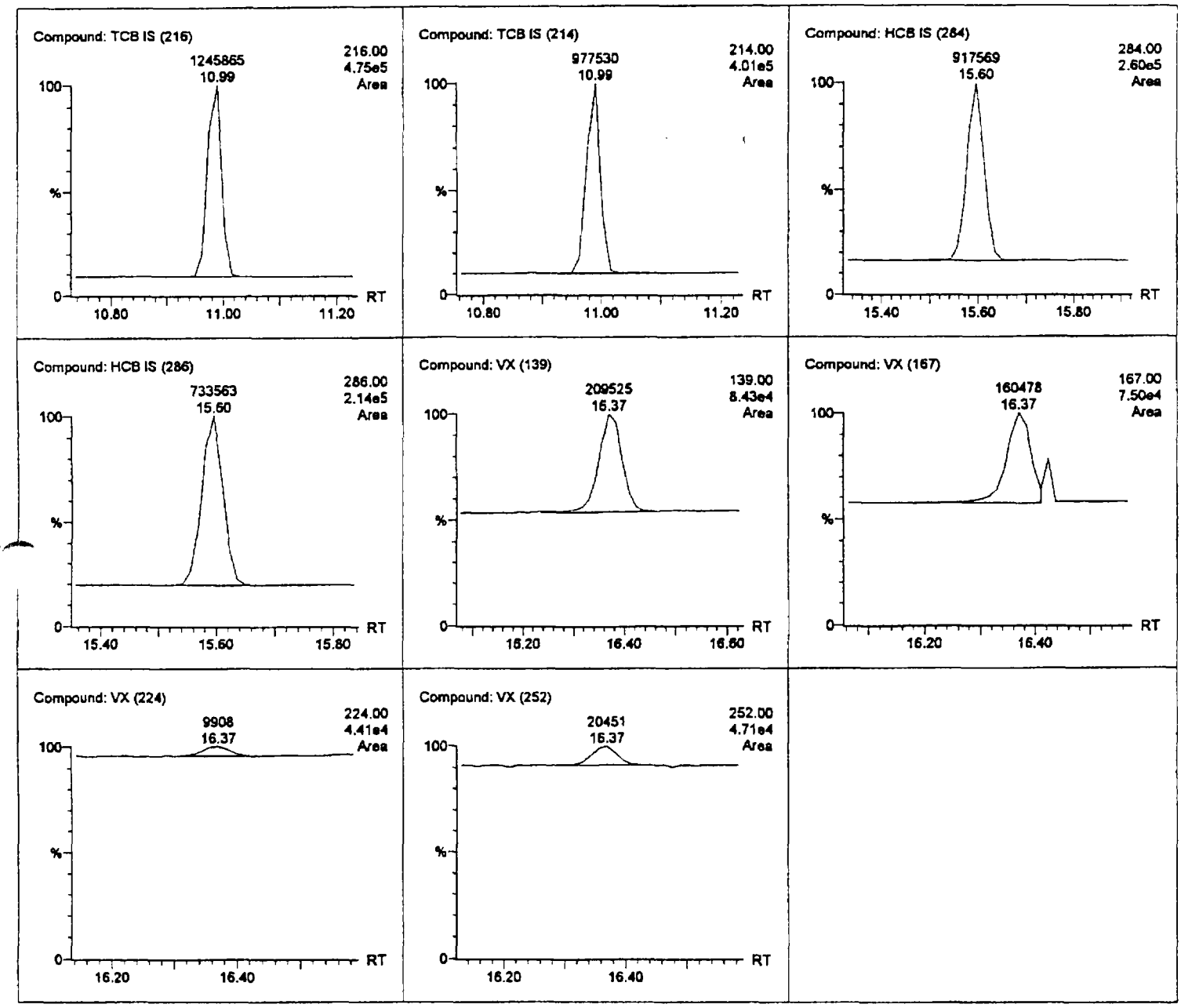

Sample ID: 2uL STD2 VX 0.2ng/uL, IS 0.1ng/uL
Y1017225
Acquired on 18-Oct-2002 at 01:31:27
SIR of 8 Channels EI+
TIC
1.08e6
RT
100
%
0
10.975
12.511
13.672
15.583
16.093
16.373
11.000
12.000
13.000
14.000
15.000
16.000
17.000
m

Southwest Research Institute
Agent (VX) Quantitation Report

010022

Filename: C:\MASSLAB\DATA\Y1017226.RAW
Sample ID: 2uL STD3 VX 0.5ng/uL, IS 0.1ng/uL
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 18-Oct-2002 at 02:06:46
Instrument: Y-MD800

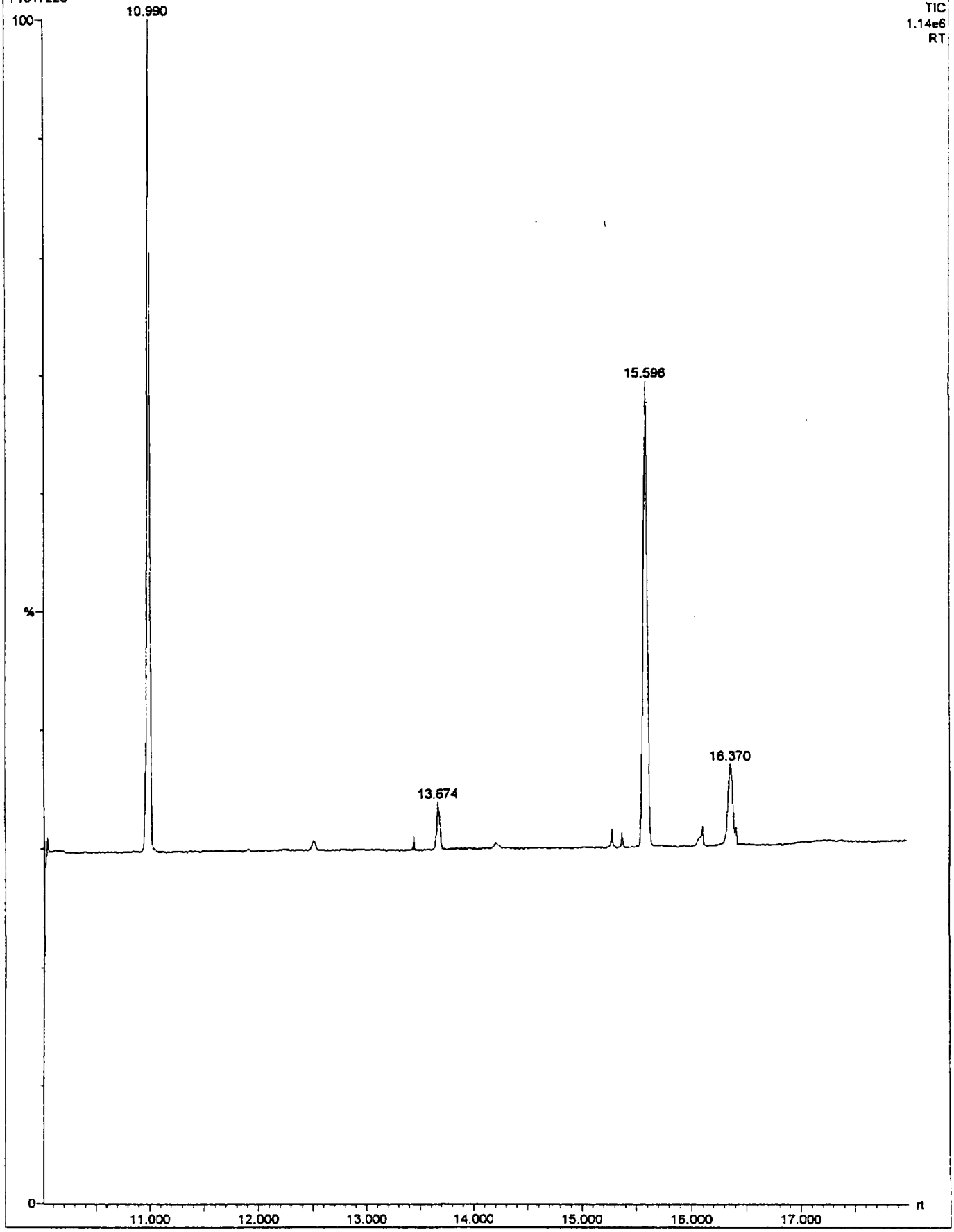


Peak List Information - Y1017226

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.990	IS	1245865	432757	0.1000
2	TCB IS (214)	10.990	IS	977530	359057	0.1000
3	HCB IS (284)	15.596	IS	917569	218547	0.1000
4	HCB IS (286)	15.596	IS	733563	172536	0.1000
5	VX (139)	16.370		209525	39110	0.4888
6	VX (167)	16.370		160478	31861	0.4789
	VX (224)	16.370		9908	1943	0.4963
8	VX (252)	16.370		20451	4196	0.3945

Sample ID: 2uL STD3 VX 0.5ng/uL, IS 0.1ng/uL
Y1017226

Acquired on 18-Oct-2002 at 02:06:46
SIR of 8 Channels EI+
TIC
1.14e6
RT

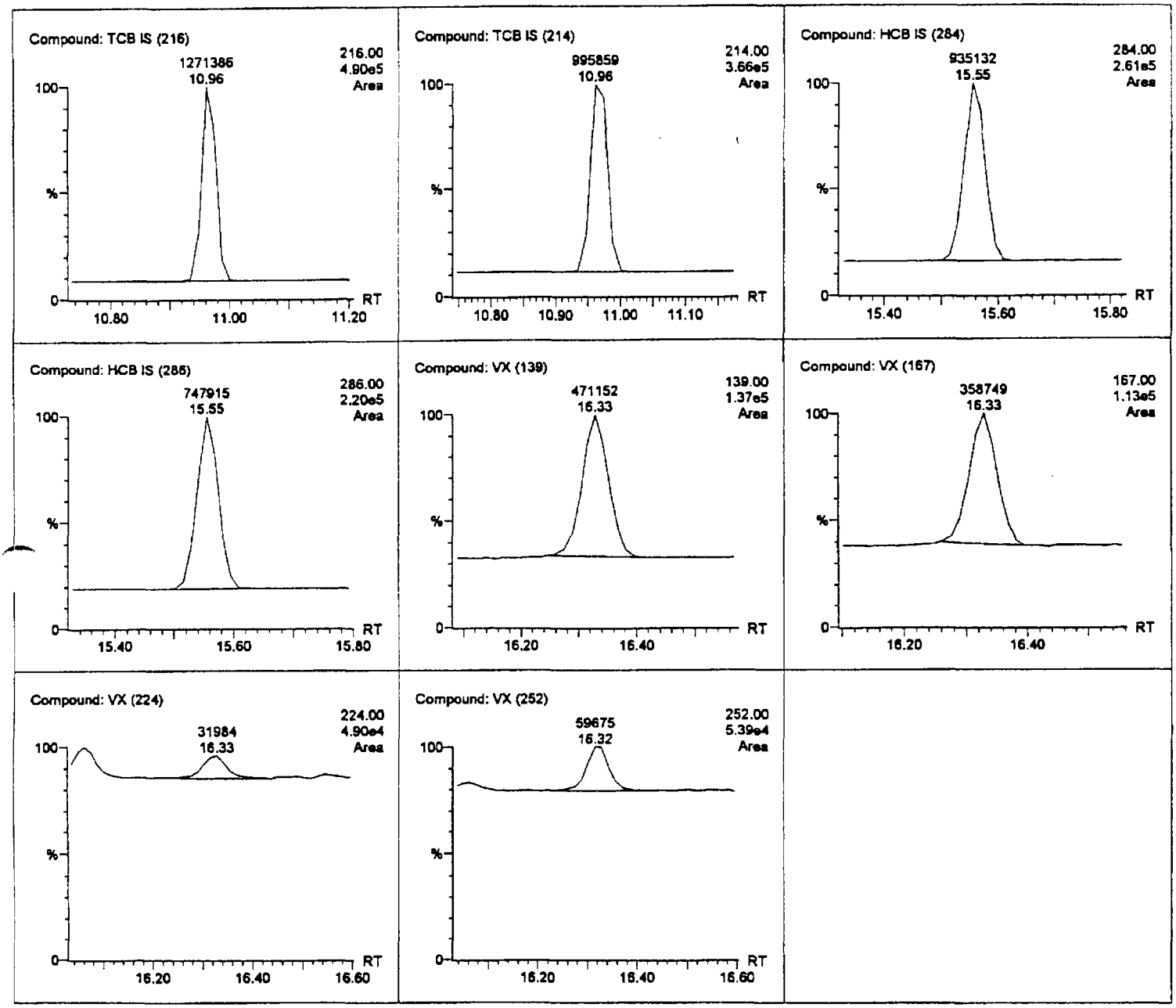


Southwest Research Institute
Agent (VX) Quantitation Report

010024

.ename: C:\MASSLAB\DATA\Y1017227.RAW
Sample ID: 2uL STD4 VX 1.0ng/uL, IS 0.1ng/uL
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 18-Oct-2002 at 02:41:56
Instrument: Y-MD800



Peak List Information - Y1017227

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.961	IS	1271386	447473	0.1000
2	TCB IS (214)	10.961	IS	995859	323968	0.1000
3	HCB IS (284)	15.555	IS	935132	218677	0.1000
4	HCB IS (286)	15.555	IS	747915	177875	0.1000
5	VX (139)	16.329		471152	90640	1.0784
6	VX (167)	16.329		358749	69118	1.0505
	VX (224)	16.329		31984	5140	1.5719
	VX (252)	16.316		59675	11024	1.1294

010025

Sample ID: 2uL STD4 VX 1.0ng/uL, IS 0.1ng/uL

Acquired on 18-Oct-2002 at 02:41:56

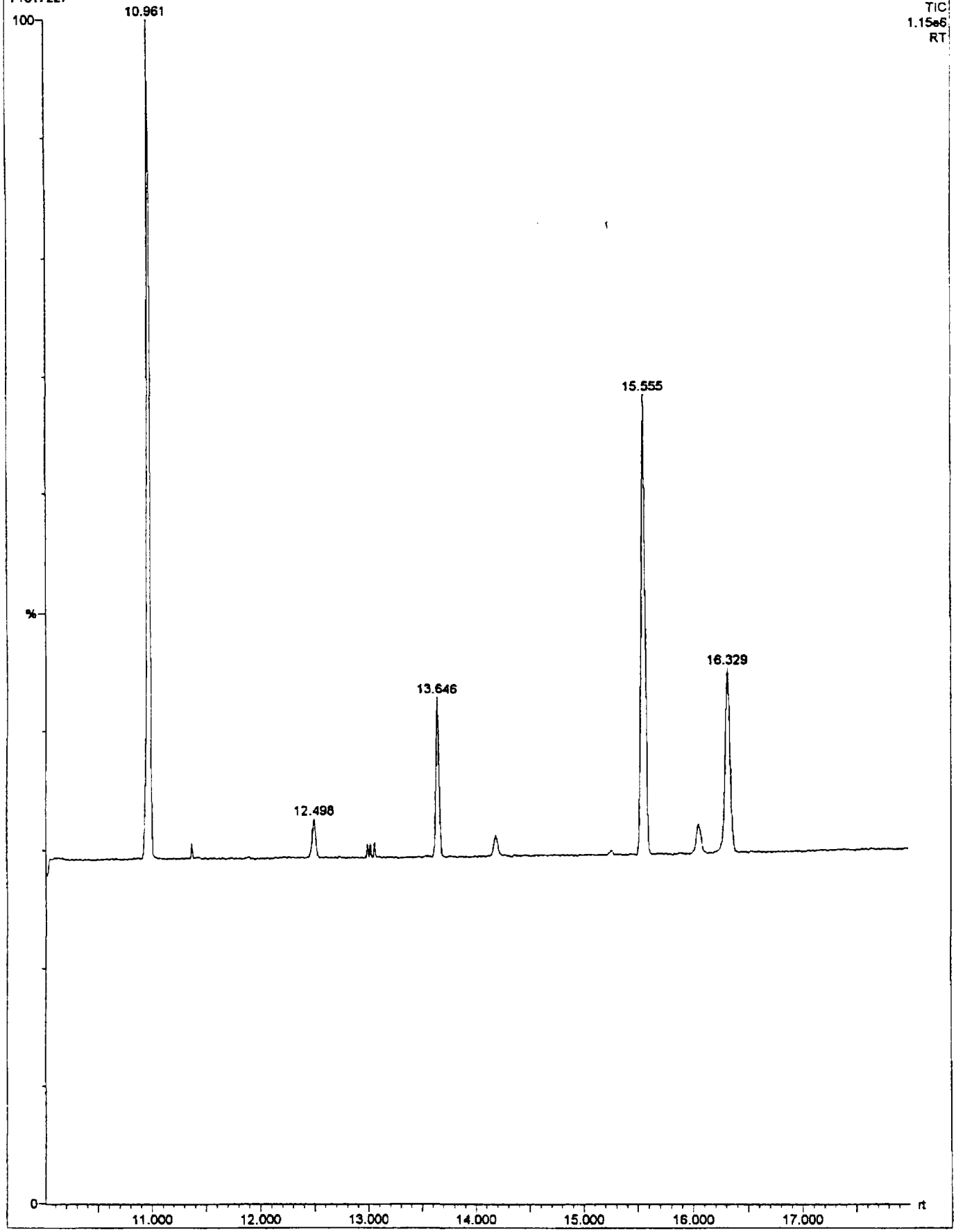
Y1017227

SIR of 8 Channels EI+

TIC

1.15e6

RT

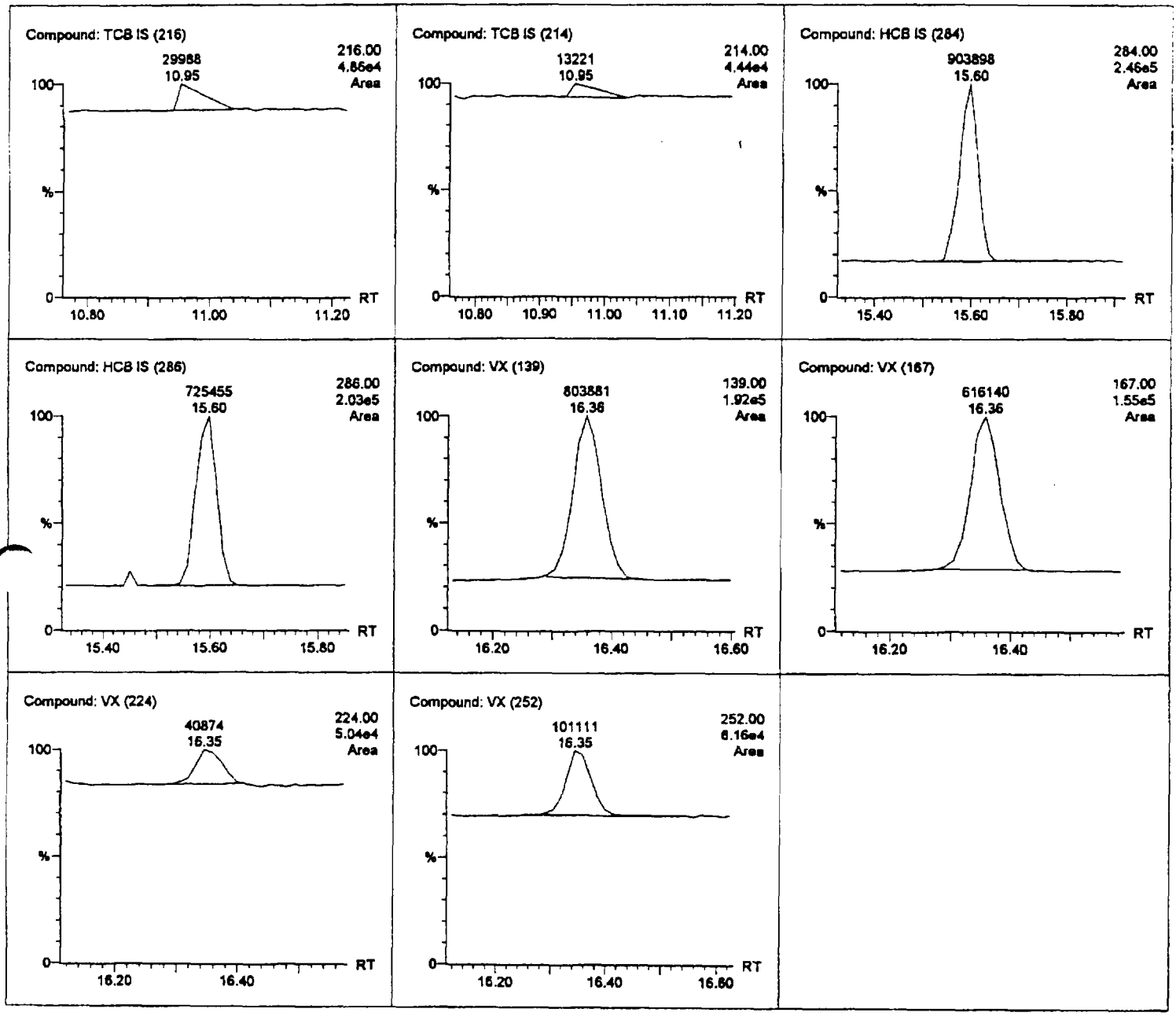


Southwest Research Institute
Agent (VX) Quantitation Report

010026

ɜname: C:\MASSLAB\DATA\Y1017228.RAW
Sample ID: 2uL STD5 VX 1.5ng/uL, IS 0.1ng/uL
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 18-Oct-2002 at 03:17:11
Instrument: Y-MD800



Peak List Information - Y1017228

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.955	IS	29988	6109	0.1000
2	TCB IS (214)	10.955	IS	13221	2751	0.1000
3	HCB IS (284)	15.598	IS	903898	204496	0.1000
4	HCB IS (286)	15.598	IS	725455	160965	0.1000
5	VX (139)	16.358		803881	145583	1.9036
6	VX (167)	16.358		616140	110808	1.8666
	VX (224)	16.345		40874	8169	2.0782
	VX (252)	16.345		101111	18704	1.9798

Southwest Research Institute

Semi-volatile Organics Continuing Calibration Check

010027

Project: 05935.03.001
Instrument: Y-MD800

Case: JACADS
GC Column: DB-1701 30m X 0.32mm ID

SDG: 213854

Lab File ID: Y1017229

Analysis Date: 10/18/02
Initial Date: 10/18/02 10/18/02

Analysis Time: 03:52:00
Initial Time: 00:56:00 03:17:00

Compounds	Mean RRF	RRF1	MIN RRF	%D	MAX %D
VX	0.037	0.040		10.0	25.0

FORM VII

ver (07/17/2002)

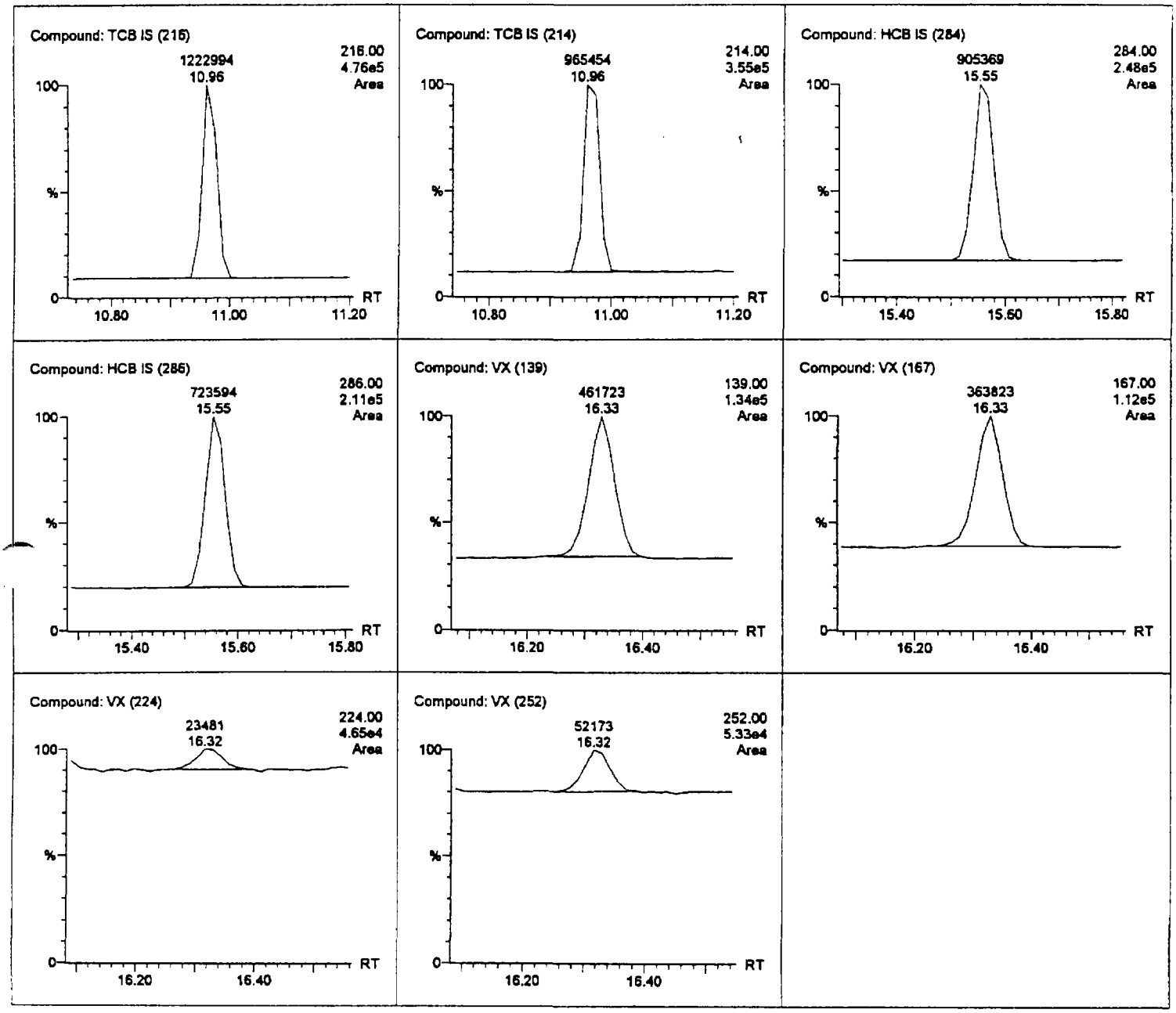
Southwest Research Institute
Agent (VX) Quantitation Report

010028

..ename: C:\MASSLAB\DATA\Y1017229.RAW
Sample ID: 2uL CCS1 VX 1.0ng/uL, IS 0.1ng/uL
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

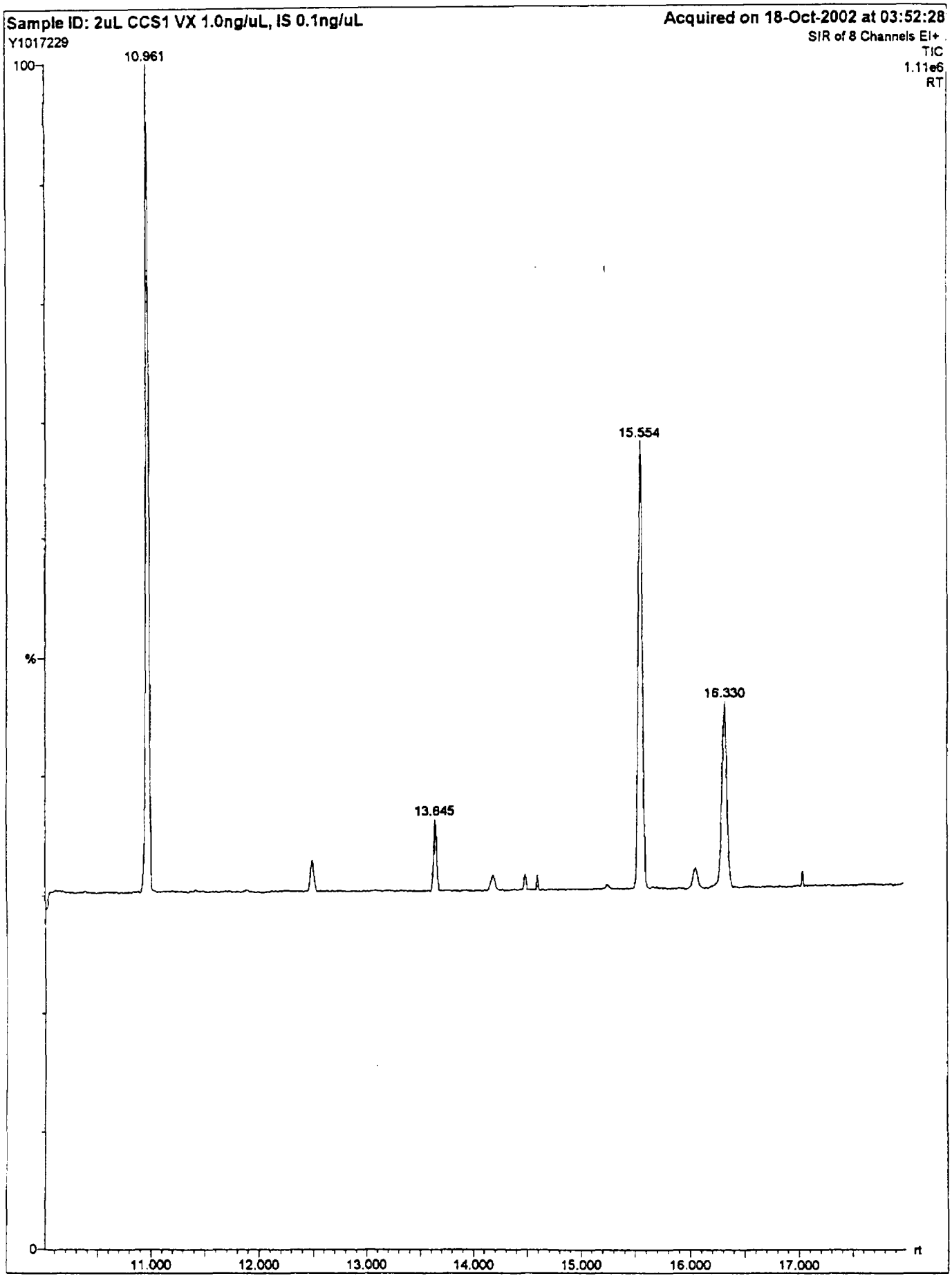
Date Aquired: 18-Oct-2002 at 03:52:28

Instrument: Y-MD800



Peak List Information - Y1017229

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.961	IS	1222994	433681	0.1000
2	TCB IS (214)	10.961	IS	965454	313552	0.1000
3	HCB IS (284)	15.554	IS	905369	206149	0.1000
4	HCB IS (286)	15.554	IS	723594	169571	0.1000
5	VX (139)	16.330		461723	88952	1.0916
[illegible]	VX (167)	16.330		363823	68721	1.1004
	VX (224)	16.317		23481	4460	1.1919
6	VX (252)	16.317		52173	10425	1.0199


Sample ID: 2uL CCS1 VX 1.0ng/uL, IS 0.1ng/uL
Y1017229
Acquired on 18-Oct-2002 at 03:52:28
SIR of 8 Channels EI+
TIC
1.11e6
RT
100
%
0
10.961
13.645
15.554
16.330
11.000
12.000
13.000
14.000
15.000
16.000
17.000
rt

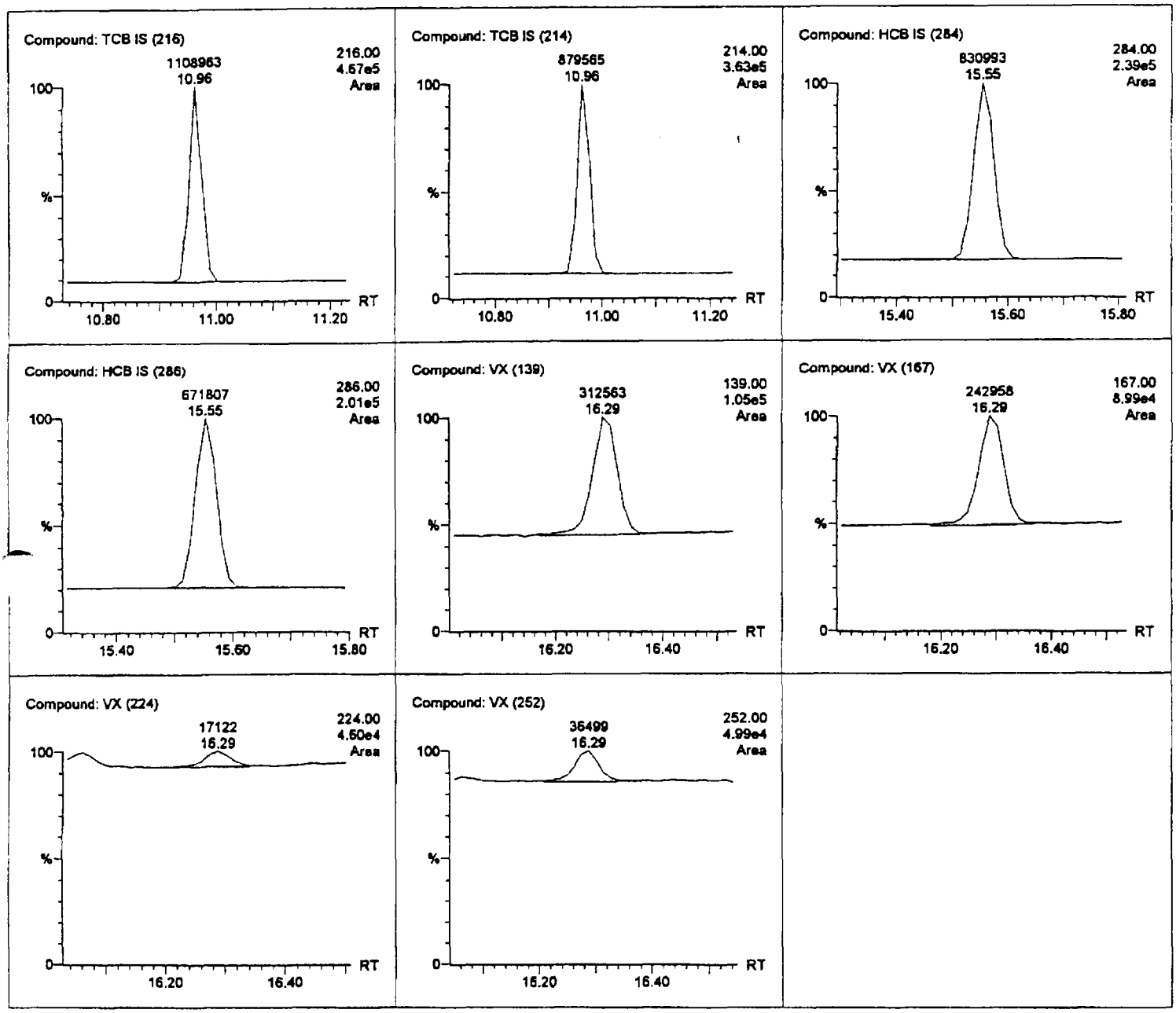
Southwest Research Institute
Agent (VX) Quantitation Report

010031

aname: C:\MASSLAB\DATA\Y1017237.RAW
Sample ID: 2uL CCS2 VX 1.0ng/uL, IS 0.1ng/uL
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 18-Oct-2002 at 08:33:08

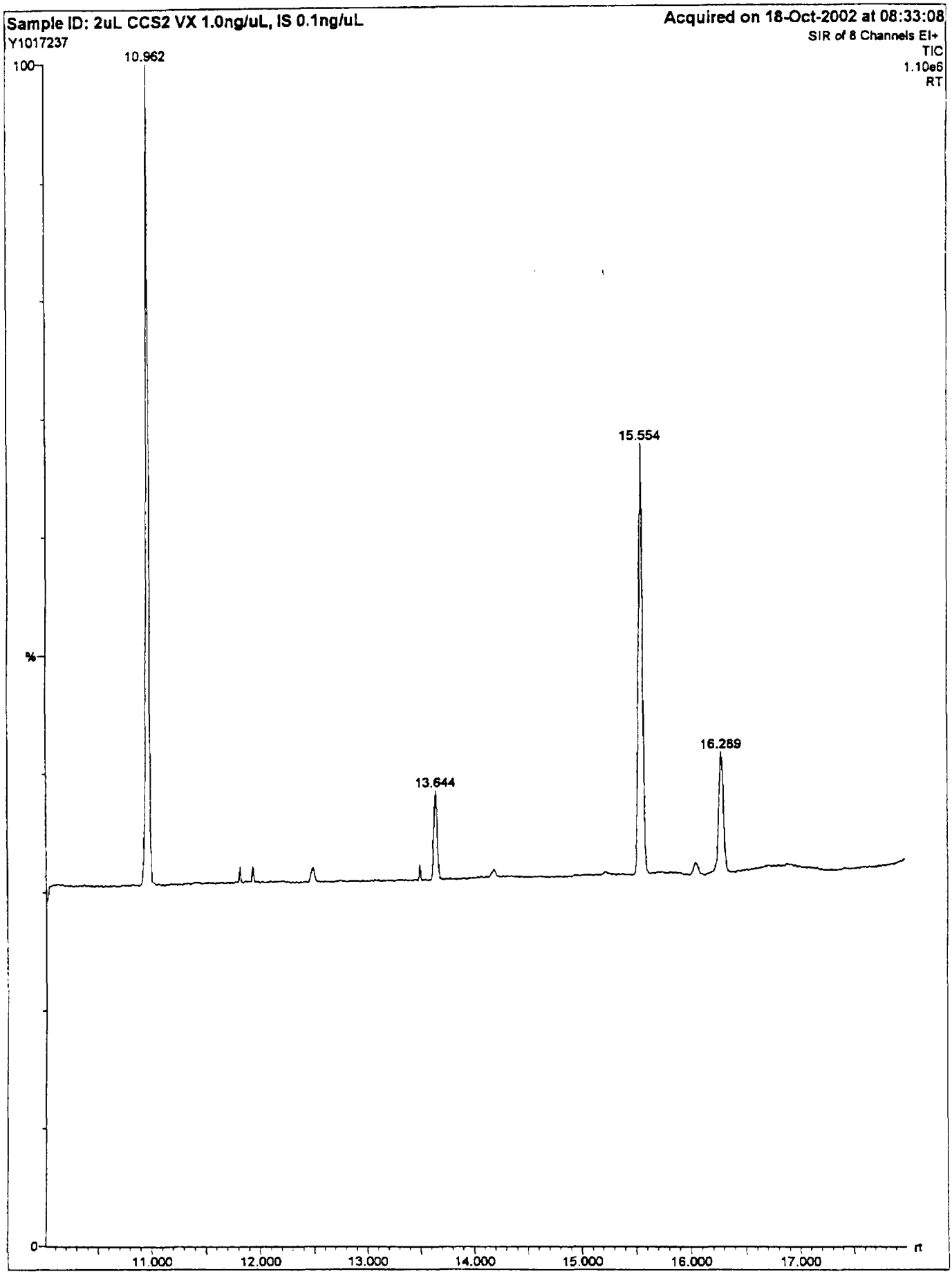
Instrument: Y-MD800



Peak List Information - Y1017237

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.962	IS	1108963	424383	0.1000
2	TCB IS (214)	10.962	IS	879565	321760	0.1000
3	HCB IS (284)	15.554	IS	830993	196496	0.1000
4	HCB IS (286)	15.554	IS	671807	158992	0.1000
5	VX (139)	16.289		312563	57560	0.8051
6	VX (167)	16.289		242958	45628	0.8006
	VX (224)	16.289		17122	3339	0.9469
	VX (252)	16.289		36499	7051	0.7774

010032


Sample ID: 2uL CCS2 VX 1.0ng/uL, IS 0.1ng/uL
Y1017237
Acquired on 18-Oct-2002 at 08:33:08
SIR of 8 Channels EI+
TIC
1.10e6
RT
100
%
0
10.962
13.644
15.554
16.289
11.000
12.000
13.000
14.000
15.000
16.000
17.000
rt

SOUTHWEST RESEARCH INSTITUTE
CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order No.: 23238
VTSR: October 7, 2002

RAW QC DATA

010033

SOUTHWEST RESEARCH INSTITUTE

Semi-volatile Organics Data Reporting Form

Client: JACADS
Case: JACADS
SDG: 213854
Matrix: Solvent
Sample wt. (g): 2
Injection Volume (uL): 2.0

Client Sample ID: BLANK (10/16/02)
System ID:
Filename: Y1017230
pH: NA
Conc. Ext. Vol. (uL): 8919

Project Number: 05935.03.001
Instrument: Y-MD800
Date Received: NA
Date Extracted: Oct 16, 2002
Date Analyzed: Oct 18 2002 4:27AM
Dilution Factor: 1

CAS No.	Compound	ug/Kg
50782-69-9	VX	220 U

DATA REPORTING QUALIFIERS

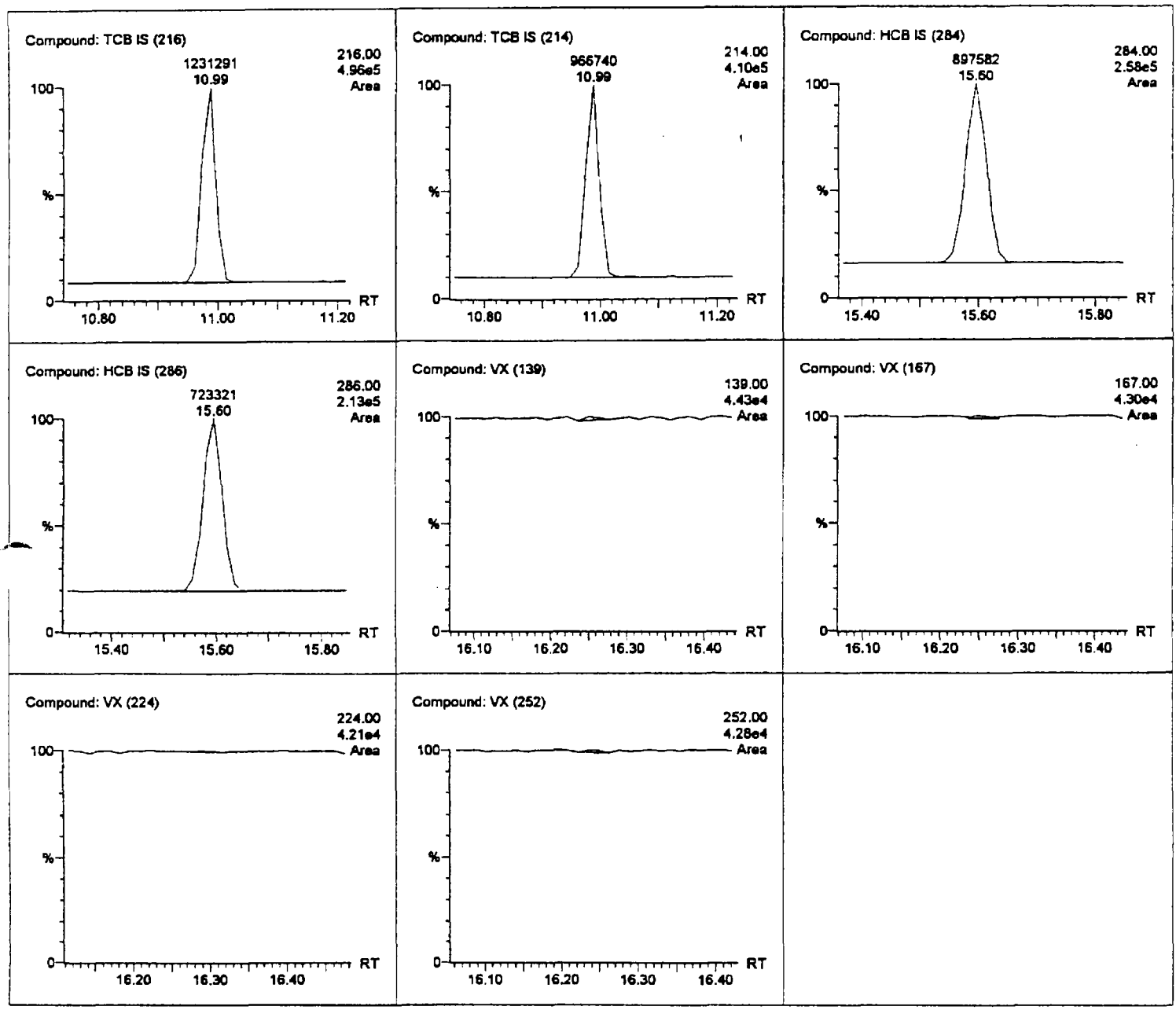
- B This flag is used when the analyte is found in the blank as well as the sample.
- D Concentration value is from dilution analysis.
- E This flag indicates compounds whose concentrations exceed the calibration range.
- J Indicates an estimated value.
- U Indicates compound was analyzed for, but not detected. Report the minimum detection limit for the sample with U (e.g. 10U) based on necessary concentration dilution action (This is not necessarily the instrument detection limit).

Southwest Research Institute
Agent (VX) Quantitation Report

010034

. ..ename: C:\MASSLAB\DATA\Y1017230.RAW
Sample ID: 2uL Ext BLK
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 18-Oct-2002 at 04:27:36
Instrument: Y-MD800



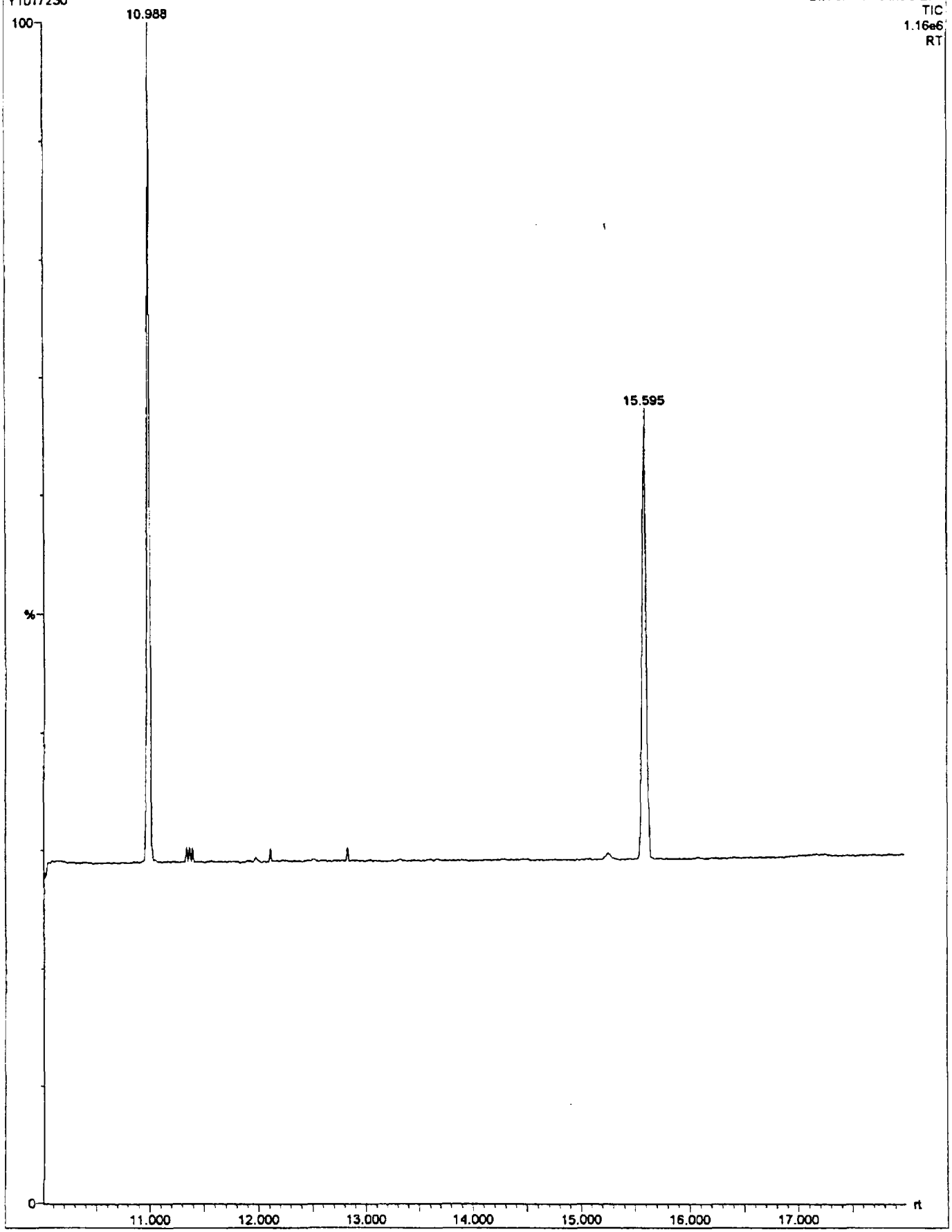
Peak List Information - Y1017230

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.988	IS	1231291	453417	0.1000
2	TCB IS (214)	10.988	IS	966740	368280	0.1000
3	HCB IS (284)	15.595	IS	897582	216405	0.1000
4	HCB IS (286)	15.595	IS	723321	171305	0.1000
5	VX (139)	16.249		1537	667	0.0037
[illegible]	VX (167)	16.249		1704	561	0.0052
	VX (224)	16.289		425	214	0.0218
[illegible]	VX (252)	16.236		1116	412	0.0220

010035

Sample ID: 2uL Ext BLK
Y1017230

Acquired on 18-Oct-2002 at 04:27:36
SIR of 8 Channels EI+
TIC
1.16e6
RT



SOUTHWEST RESEARCH INSTITUTE

Semi-volatile Organics Data Reporting Form

Client: JACADS
Case: JACADS
SDG: 213854
Matrix: Solvent
Sample wt. (g): 2
Injection Volume (uL): 2.0

Client Sample ID: BLANK MS LCS
System ID:
Filename: Y1017231
pH: NA
Conc. Ext. Vol. (uL): 8919

Project Number: 05935.03.001
Instrument: Y-MD800
Date Received: NA
Date Extracted: Oct 16, 2002
Date Analyzed: Oct 18 2002 5:02AM
Dilution Factor: 2

CAS No.	Compound	ug/Kg
50782-69-9	VX	8900 D

DATA REPORTING QUALIFIERS

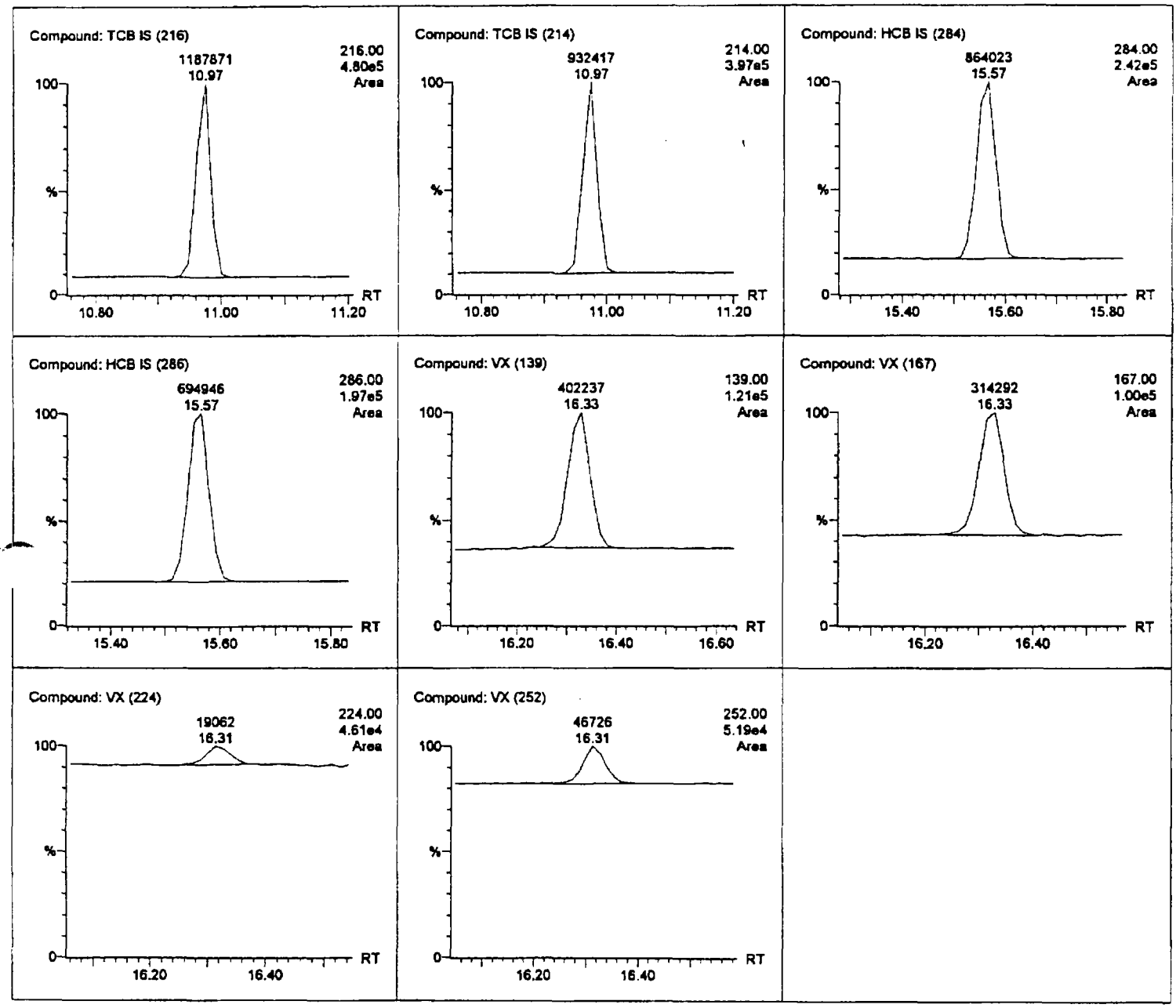
- B This flag is used when the analyte is found in the blank as well as the sample.
- D Concentration value is from dilution analysis.
- E This flag indicates compounds whose concentrations exceed the calibration range.
- J Indicates an estimated value.
- U Indicates compound was analyzed for, but not detected. Report the minimum detection limit for the sample with U (e.g. 10U) based on necessary concentration dilution action (This is not necessarily the instrument detection limit).

Southwest Research Institute
Agent (VX) Quantitation Report

010037

ɔname: C:\MASSLAB\DATA\Y1017231.RAW
Sample ID: 2uL Ext BLK MS W
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

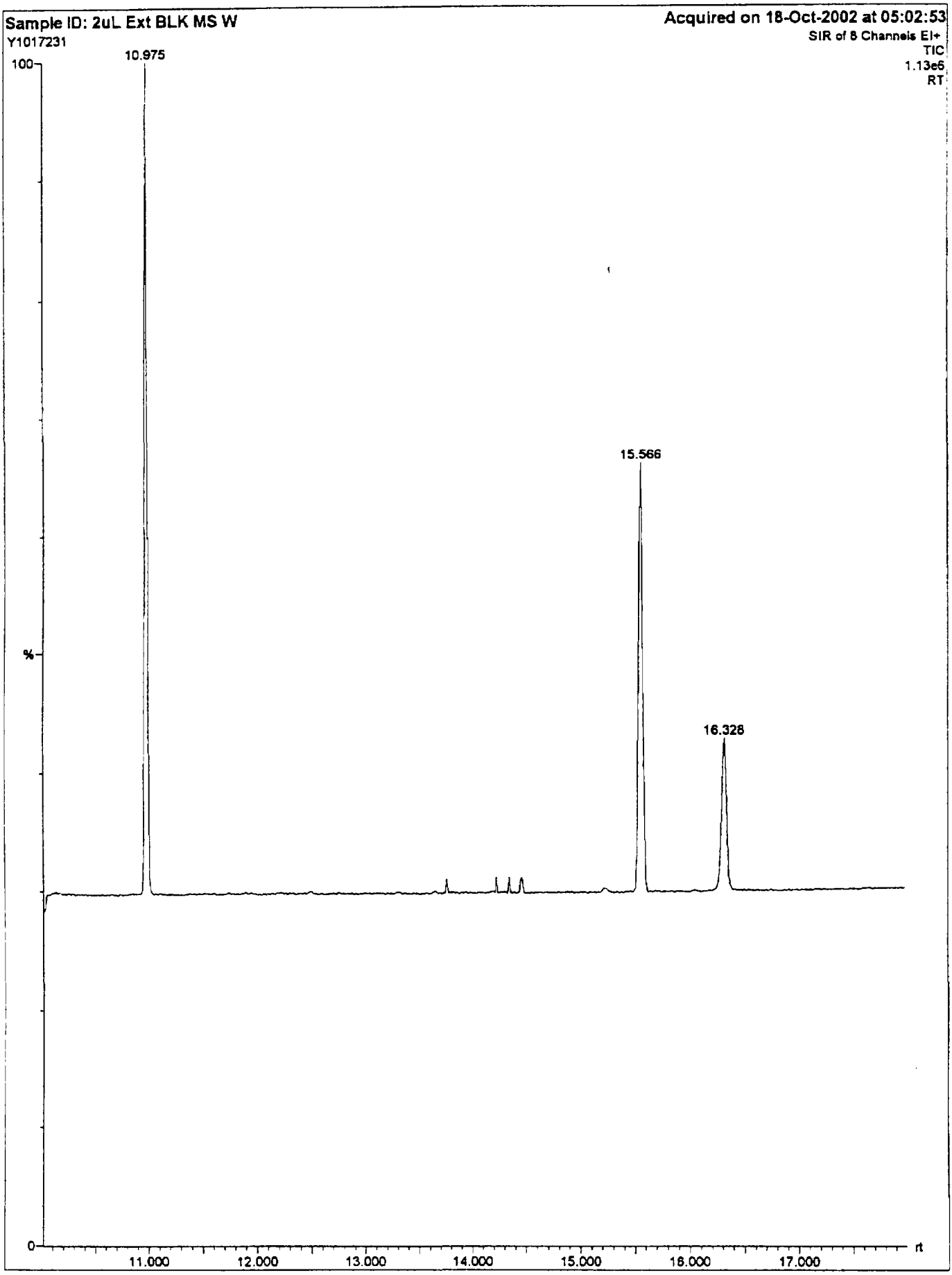
Date Aquired: 18-Oct-2002 at 05:02:53
Instrument: Y-MD800



Peak List Information - Y1017231

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.975	IS	1187871	437877	0.1000
2	TCB IS (214)	10.975	IS	932417	355529	0.1000
3	HCB IS (284)	15.566	IS	864023	200324	0.1000
4	HCB IS (286)	15.566	IS	694946	155726	0.1000
5	VX (139)	16.328		402237	76746	0.9964
6	VX (167)	16.328		314292	57282	0.9961
	VX (224)	16.314		19062	4007	1.0139
	VX (252)	16.314		46726	9224	0.9571

010038


Sample ID: 2uL Ext BLK MS W
Y1017231
Acquired on 18-Oct-2002 at 05:02:53
SIR of 8 Channels EI+
TIC
1.13e6
RT
100
%
0
10.975
15.566
16.328
11.000
12.000
13.000
14.000
15.000
16.000
17.000
rt

SOUTHWEST RESEARCH INSTITUTE

Semi-volatile Organics Data Reporting Form

Client: JACADS
Case: JACADS
SDG: 213854
Matrix: Solid
Sample wt. (g): 2.0178
Injection Volume (uL): 2.0

Client Sample ID: Tray 153 (021002-001) R2 MS
System ID: 213854
Filename: Y1017233
pH: NA
Conc. Ext. Vol. (uL): 8919

Project Number: 05935.03.001
Instrument: Y-MD800
Date Received: 10/07/2002
Date Extracted: Oct 16, 2002
Date Analyzed: Oct 18 2002 6:13AM
Dilution Factor: 2.5

CAS No.	Compound	ug/Kg
50782-69-9	VX	12000 D

DATA REPORTING QUALIFIERS

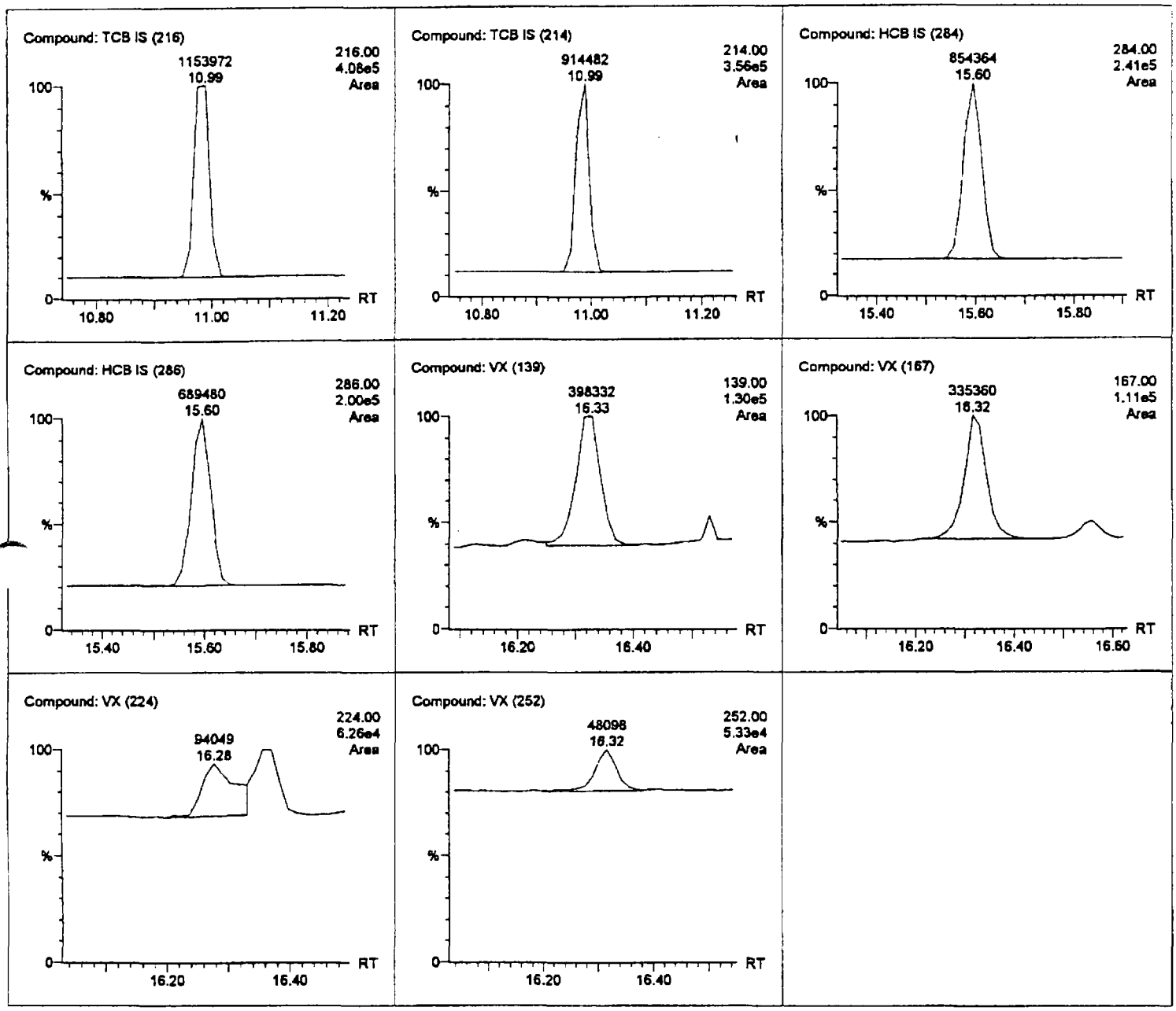
- B This flag is used when the analyte is found in the blank as well as the sample.
- D Concentration value is from dilution analysis.
- E This flag indicates compounds whose concentrations exceed the calibration range.
- J Indicates an estimated value.
- U Indicates compound was analyzed for, but not detected. Report the minimum detection limit for the sample with U (e.g. 10U) based on necessary concentration dilution action (This is not necessarily the instrument detection limit).

Southwest Research Institute
Agent (VX) Quantitation Report

010040

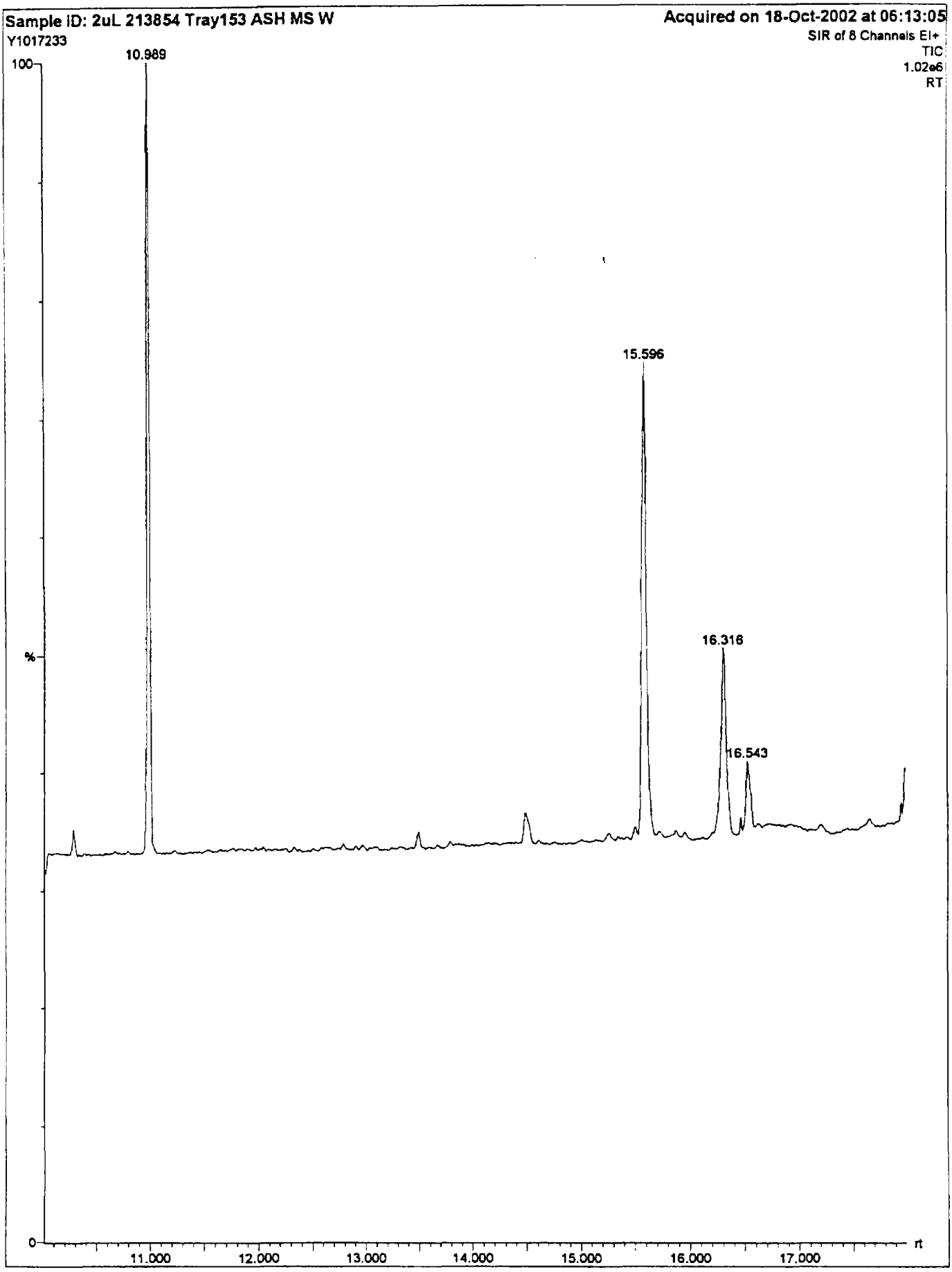
. ..iename: C:\MASSLAB\DATA\Y1017233.RAW
Sample ID: 2uL 213854 Tray153 ASH MS W
Conditions: 60C|2MIN|15C/M|200C|5C/M|260C|5MIN

Date Aquired: 18-Oct-2002 at 06:13:05
Instrument: Y-MD800



Peak List Information - Y1017233

Entry	Name	RT	Flags	Area	Height	Found Conc (ng/uL)
1	TCB IS (216)	10.989	IS	1153972	365184	0.1000
2	TCB IS (214)	10.989	IS	914482	315022	0.1000
3	HCB IS (284)	15.596	IS	854364	198810	0.1000
4	HCB IS (286)	15.596	IS	689480	158479	0.1000
5	VX (139)	16.330		398332	79134	0.9979
[illegible]	VX (167)	16.316		335360	64374	1.0749
	VX (224)	16.276		94049	15626	5.0591
6	VX (252)	16.316		48098	10404	0.9964


Sample ID: 2uL 213854 Tray153 ASH MS W
Y1017233
Acquired on 18-Oct-2002 at 06:13:05
SIR of 8 Channels EI+
TIC
1.02e6
RT
100
%
0
10.989
15.596
16.316
16.543
11.000
12.000
13.000
14.000
15.000
16.000
17.000
rt

SOUTHWEST RESEARCH INSTITUTE
CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order No.: 23238
VTSR: October 7 2002

NARRATIVE

JACADS
SwRI Project Number : 01.05935.03.001
SDG: 213854
SwRI Work Order : 23238
October 18,2002

000001

VX

1. A five point initial calibration curve was performed and results met QC criteria.
   Continuing calibration met the criteria of 60-135% of known concentrations.

2. A mean calibration factor was used to generate the sample concentration.

3 A 2 mL fraction was taken from the original 11 mL sample and was solvent exchanged into water.
   This fraction was taken up to 4 mL final Volume.

4 Blk Spk, MS & MSD required a 2x dilution to get in curve range.
   Recovery for Blank Spike, MS & MSD met criteria of 60-135 %.
   The avg for the 3 reps was used for subtracting out from the MS,MSD

5 Samples directly injected into the LCMS.

6 A Supelco C18 column was used for VX analysis.
   The following conditions were used..
   Mobile Phase: Water / 0.1% formic acid, MeOH / 0.1% formic acid
   Flow Rate 0.700 mL/min. gradient
   LCMS scaning for the following ions...
   VX: 268, 128

Sample Calculation

A x B x C x (1/D) x (E/F)

Where:
A = Instrument value (ug/mL)
B = Dilution factor (DF)
C = Final Volume (mL)
D = Split Volume (mL)
E = Extraction Volume (mL)
F = Sample Wt (g)

Sample R1

wt (g)	Ext Vol mL	Split Volume	Final Volume (mL)	DF	Found ug/mL	Conc. ug/g
2.012	11	2	4	1	0.202	2.205

REVIEWED BY:

_____

DATE: _____

G. Alm 10-18-02

SOUTHWEST RESEARCH INSTITUTE
CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order No.: 23238
VTSR: October 7 2002

STANDARD PREPARATION LOGS

158 TITLE JACADS

WO# 23238

PROJECT NO. 05935.03.001

BOOK NO. 01-0500-002

Work continued from Page

Sequence: C:\HPCHEM\1\SEQUENCE\101602.S

000002

Sequence Table:

Method and Injection Info Part:

Line	Location	SampleName	Method	Inj	SampleType	InjVolume
1	Vial 1	VX STD 1	VX_FS	2	Sample	50
2	Vial 2	VX STD 2	VX_FS	1	Sample	50
3	Vial 3	VX STD 3	VX_FS	1	Sample	50
4	Vial 4	VX STD 4	VX_FS	1	Sample	50
5	Vial 5	VX STD 5	VX_FS	1	Sample	50
6	Vial 6	WATER BLK	VX_FS	1	Sample	50
7	Vial 7	VX CCS1 STD 3	VX_FS	1	Sample	50
8	Vial 8	SOLV BLK	VX_FS	1	Sample	50
9	Vial 9	SOLV BLK MS 2X	VX_FS	1	Sample	50
10	Vial 10	213854 R1	VX_FS	1	Sample	50
11	Vial 11	213854 MS 2X	VX_FS	1	Sample	50
12	Vial 12	213854 R2	VX_FS	1	Sample	50
13	Vial 13	213854 MSD 2X	VX_FS	1	Sample	50
14	Vial 14	213854 R3	VX_FS	1	Sample	50
15	Vial 15	WATER BLK2	VX_FS	1	Sample	50
16	Vial 16	VX CCS2 STD 3	VX_FS	1	Sample	50
17	Vial 17	STD 1 ADP'S	ADPS	2	Sample	50
18	Vial 18	STD 2 ADP'S	ADPS	1	Sample	50
19	Vial 19	STD 3 ADP'S	ADPS	1	Sample	50
20	Vial 20	STD 4 ADP'S	ADPS	1	Sample	50
21	Vial 21	STD 5 ADP'S	ADPS	1	Sample	50
22	Vial 22	CCS1 STD 3 ADPS	ADPS	1	Sample	50
23	Vial 23	BLK SPK	ADPS	1	Sample	50
24	Vial 24	WIPE BLK	ADPS_SA	1	Sample	50
25	Vial 25	WIPE MDL	ADPS_SA	1	Sample	50
26	Vial 26	WIPE MDL	ADPS_SA	1	Sample	50
27	Vial 27	WIPE MDL	ADPS_SA	1	Sample	50
28	Vial 28	WIPE MDL	ADPS_SA	1	Sample	50
29	Vial 29	WIPE MDL	ADPS_SA	1	Sample	50
30	Vial 30	WIPE MDL	ADPS_SA	1	Sample	50
31	Vial 31	WIPE MDL	ADPS_SA	1	Sample	50
32	Vial 32	WATER BLK 2	ADPS_SA	1	Sample	50
33	Vial 33	CCS2 STD 3 ADPS	ADPS_SA	1	Sample	50
34	Vial 34	CONCRETE BLK	ADPS_SA	1	Sample	50
35	Vial 35	CONCRETE A 1	ADPS_SA	1	Sample	50
36	Vial 36	CONCRETE B 1	ADPS_SA	1	Sample	50
37	Vial 37	CONCRETE C 1	ADPS_SA	1	Sample	50
38	Vial 38	CONCRETE A 2	ADPS_SA	1	Sample	50
39	Vial 39	CONCRETE B 2	ADPS_SA	1	Sample	50
40	Vial 40	CONCRETE C 2	ADPS_SA	1	Sample	50
41	Vial 41	CONCRETE A 3	ADPS_SA	1	Sample	50
42	Vial 42	CONCRETE B 3	ADPS_SA	1	Sample	50
43	Vial 43	CONCRETE C 3	ADPS_SA	1	Sample	50
44	Vial 44	WATER BLK 3	ADPS_SA	1	Sample	50
45	Vial 45	CCS3 STD 3 ADPS	ADPS_SA	1	Sample	50

10-16-02

SCIENTIFIC BINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA

Work continued to Page

SIGNATURE

DATE 10/16/02

DISCLOSED TO AND UNDERSTOOD BY

DATE 10/15/02

WITNESS

DATE

TITLE Direct VX LCMS

WO# 23238

PROJECT NO. 05935.03.001

BOOK NO. 01-0500-009

SwRI

Work continued from Page

000003

177-01-01-0500-009 VX 1 ug/ml
took 200ul of VX 10 ug/ml (Act. book #8, Pg 178)
into 2ml FV water

177-02-01-0500-009 VX 0.5 ug/ml
took 1ml VX 1 ug/ml (177-01-01-0500-009)
into 2ml FV water

177-03-01-0500-009 VX 0.25 ug/ml
took 1ml VX 0.5 (177-02-01-0500-009)
into 2ml FV water

177-04-01-0500-009 VX 0.125 ug/ml
took 1ml VX 0.25 ug/ml (177-03-01-0500-009)
into 2ml FV water

177-05-01-0500-009 VX 0.063 ug/ml
took 1ml VX 0.125 (177-04-01-0500-009)
into 2ml FV water

30% formic 300ul of formic acid was placed into 1ml FV water.
Fisher Water # WS-4, lot 017763

Formic 99% #14793-0250 Acros, lot UN1779

10/15/02

D. Alm

SCIENTIFIC BINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA

Work continued to Page

SIGNATURE D. A. Alm DATE 10/15/02

DISCLOSED TO AND UNDERSTOOD BY DATE WITNESS Ruth Mast DATE 10/15/02

SWRI 178 TITLE Working Standard Preparation GB/VX/HD/HN1

PROJECT NO.

BOOK NO. 01 0500 011

Work continued from Page

GB Standards. Prep'd from GB/IPA 091802 SWRI-DIL VIAL #1

Solvent IPA Lot # D10738

1.96 mg/mL (x) = 20 ng/µl (10 mL)
1960 ng/µl (x) = 20 ng/µl (10,000 µl)
1960 ng/µl (x) = 200,000 ng
x = 102 µl

000004

20 ng/µl (x) = 0.14 ng/µl (10 mL)
20 ng/µl (x) = 0.14 ng/µl (10,000 µl)
20 ng/µl (x) = 1400 ng
x = 70 µl

VX STANDARDS prep'd from VX/IPA 9063-CTF-DIL VIAL # 160

0.11 mg/mL (x) = ~~20~~ 10 ng/µl (~~5~~ 10 mL) RJS 30 Oct, 2002
110 ng/µl (x) = ~~20~~ 10 ng/µl (~~5000~~ 10,000 µl) RJS 30 Oct, 2002
110 ng/µl (x) = 100,000 ng
x = 909 µl

~~20~~ 10 ng/µl (x) = 0.014 ng/µl (10 mL)
~~20~~ 10 ng/µl (x) = 0.014 ng/µl (10,000 µl)
~~20~~ 10 ng/µl (x) = 140 ng/
x = ~~7~~ 14 µl RJS 30 Oct. 2002

HD STANDARDS prep'd from HD/HEX 062702-SWRI-DIL Vial #1

Solvent Hexane Lot # 003107

9.90 mg/mL (x) = 40 ng/µl (10 mL)
9900 ng/µl (x) = 40 ng/µl (10,000 µl)
9900 ng/µl (x) = 400,000 ng
x = 40.4 µl

HN1 STANDARD prep'd from HN1/HEX 090402 SWRI-DIL VIAL #1

1.96 mg/mL (x) = 40 ng/µl (10 mL)
1960 ng/µl (x) = 40 ng/µl (10,000 µl)
1960 ng/µl (x) = 400,000 ng
x = 204 µl

SCIENTIFIC BINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA

Work continued to Page

SIGNATURE Robert A Martinez DATE Oct. 3, 2002

DISCLOSED TO AND UNDERSTOOD BY DATE

WITNESS Russell A Barsor DATE Oct 3, 2002

180 TITLE Extraction and Analysis
JACADS
WO: 23238

PROJECT NO. 05935.03.515
BOOK NO. 01.0500.009

Work continued from Page

Balance #30

ID		Final Vol (mL)	Final Vol (mL)	VX Spk Amt
BLANK		—	11 mL	—
BLANK MS (LCS)		—	11 mL	22 ug
213854	Tray 153 Rep1	2.0176	11 mL	—
213854	Tray 153 Rep2	2.0192	11 mL	—
213854	Tray 153 Rep3	2.0003	11 mL	—
213854	Tray 153 MS	2.0178	11 mL	22 ug
213854	Tray 153 MSD	2.0125	11 mL	22 ug

VX Spk 200 uL VX @ 110 ug/uL Lot # VX/IPA 9063 CTF-DIL

- Weighed ~2.0 g of ASH into 10 mL centrifuge vial
- Spiked VX in required samples
- Added approx 5.0 mL 10% IPA/DCM and Mixed thoroughly for 1 minute
- Centrifuged Samples
- Filtered Extract(1) into 3 dram vial w/ Gelman Nylon Acrodisc 0.45 um
- Added approx 4.0 mL 10% IPA/DCM to the ASH and mixed for 1 minute
- Centrifuged Samples
- Filtered Extract(2) into the same 3 dram vial that Extract 1 is in.
- Added an additional 3.0 mL 10% IPA/DCM to the ASH and mixed for 1 min.
- Centrifuged Samples
- Filtered Extracts(3) into the same 3 dram vial that Extract 1 + 2 are in.
- Added 10% IPA/DCM to the Combined Filtrate to get a Final Vol of 11 mL
-

10% IPA/DCM.
25 mL Isopropyl Alcohol Fisher Lot# 018738 was brought to Final Vol of 250 mL w/ DCM Fisher Lot # 023244

SCIENTIFIC BINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA

Work continued to Page

SIGNATURE [signature] DATE 10/16/02

DISCLOSED TO AND UNDERSTOOD BY DATE

WITNESS [signature] DATE 10/16/02

182 TITLE Extraction Prep (Direct VR) by LC/MS JACADS WO: 23238

PROJECT NO. 05935.03.001

BOOK NO. 01-0500-009

Work continued from Page

Sample Prep for LC/MS from pg 180

000006

- Took 2mL of each Sample and placed it in an 1 dram vial
- Using Nitrogen, The solvent was evaporated to about 100uL
- About 300uL of IPA was added to the sample.
- Again the solvent was evaporated to about 100uL, and
- About 200uL of IPA was added to the samples
- The samples were brought to a F.V. of 4.0mL with HPLC water.

- The MS, MSD and Blk Spike were diluted by a Factor of 2
- 200ul of sample was placed in vial and 18ul 30% formic acid was added.
- 50ul of sample was injected directly into LCMS.

LCMS mobile phase solvents..

Add 1ml of formic into 1L HPLC water.

Add 1ml of formic into 1L HPLC MeOH.

formic acid, 99% Acros #14793-0250, lot UN1779
Water, #W5-4 Fisher, lot 017763
MeOH, #A452-4, Fisher, lot 022932

SCIENTIFIC BINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA

Work continued to Page

SIGNATURE [signature] DATE 10/16/02

DISCLOSED TO AND UNDERSTOOD BY DATE

WITNESS [signature] DATE 10/16/02

SOUTHWEST RESEARCH INSTITUTE
CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order No.: 23238
VTSR: October 7 2002

LC/MS ANALYSIS

Run on 10/16/02

	Wt (g)	Ext Vol	Split Vol	Final Vol	DF	Found Conc ug/mL	Conc. ug/g	% REC
Blk	na	na	na	4		0		na
Blk Spk	na	na	na	4	2	0.526		105%
R1	2.012	11	2	4	1	0.202	2.205	na
R2	2.019	11	2	4	1	0.236	2.575	na
R3	2.000	11	2	4	1	0.240	2.638	na
avg							**2.472**	
RMS	2.017	11	2	4	2	0.555	12.101	88%
RMSD	2.012	11	2	4	2	0.585	12.804	94%

H. Alw
10-18-02

SOUTHWEST RESEARCH INSTITUTE
CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order No.: 23238
VTSR: October 7 2002

QC SUMMARY

SOUTHWEST RESEARCH INSTITUTE

010002

VX

Results Data Reporting Form

Client: JACADS	Sample ID	Instrument: LCMS1
Case: JACADS	213854 MS	MS Filename: 10160212
SDG: 213854	213854 MSD	MSD Filename: 10160214
Project Number: 01.05935.03.001	213854	Filename: 10160211
	Solv. Blk MS (LCS)	Filename: 10160210

Compound	Spike Added ug/g	Sample Conc. ug/g	MS Conc. ug/g	MS % Rec	QC Limit Rec
VX	10.907	2.472	12.101	88%	60-135

Compound	Spike Added ug/g	MSD Conc. ug/g	MSD %Rec	%RPD
VX	10.934	12.804	94%	6.8

Blk MS Compound	Spike Added ug/mL	Blk MS Conc. ug/mL	Blk MS % Rec	QC Limit Rec
VX	0.500	0.526	105%	60-135

SOUTHWEST RESEARCH INSTITUTE 010003

Blank Summary

Blank ID:

Project: 05935.03.001 Case: JACADS SDG: 213854

Instrument: LCMS Filename: 10160209 Analyzed: Oct 16 2002 7:26PM

This Blank Applies to the Following Samples

	Sample ID	SwRI ID	Lab File ID	Analyzed
1	LCS		10160210	Oct 16 2002 7:48PM
2	Tray 153 (021002-001) R1	213854	10160211	Oct 16 2002 8:10PM
3	Tray 153 (021002-001) MS	213854	10160212	Oct 16 2002 8:32PM
4	Tray 153 (021002-001) R2	213854	10160213	Oct 16 2002 8:54PM
5	Tray 153 (021002-001) MSD	213854	10160214	Oct 16 2002 9:16PM
6	Tray 153 (021002-001) R3	213854	10160215	Oct 16 2002 9:38PM

SOUTHWEST RESEARCH INSTITUTE
CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order No.: 23238
VTSR: October 7 2002

SAMPLE DATA

SOUTHWEST RESEARCH INSTITUTE 010004

VX

Results Data Reporting Form

Client: JACADS
Case: JACADS
SDG: 213854
Matrix: Solid
Filename: 10160211
Injection Vol (uL): 50
Dilution Factor: 1
Sample Wt (g): 2.018

System ID: 213854 R1
Client Sample ID: Tray 153 (021002-001)
Project Number: 01.05935.03.001
Instrument: LCMS1
Date Received: 10/07/02
Date Extracted: 10/16/02
Date Analyzed: 10/16/02
Final Volume (mL) : 4

CAS No.	Compound	ug/g
50782-69-9	VX	2.205

Data Reporting Qualifiers

B This flag is used when the analyte is found in the blank as well as in the sample.
E This flag indicates compounds whose concentrations exceed the calibration range.
J Indicates an estimated value.
U Indicates compound was analyzed for, but not detected. Report the minimum detection limit for the sample with U (e.g. 10U) based on necessary concentration dilution action.

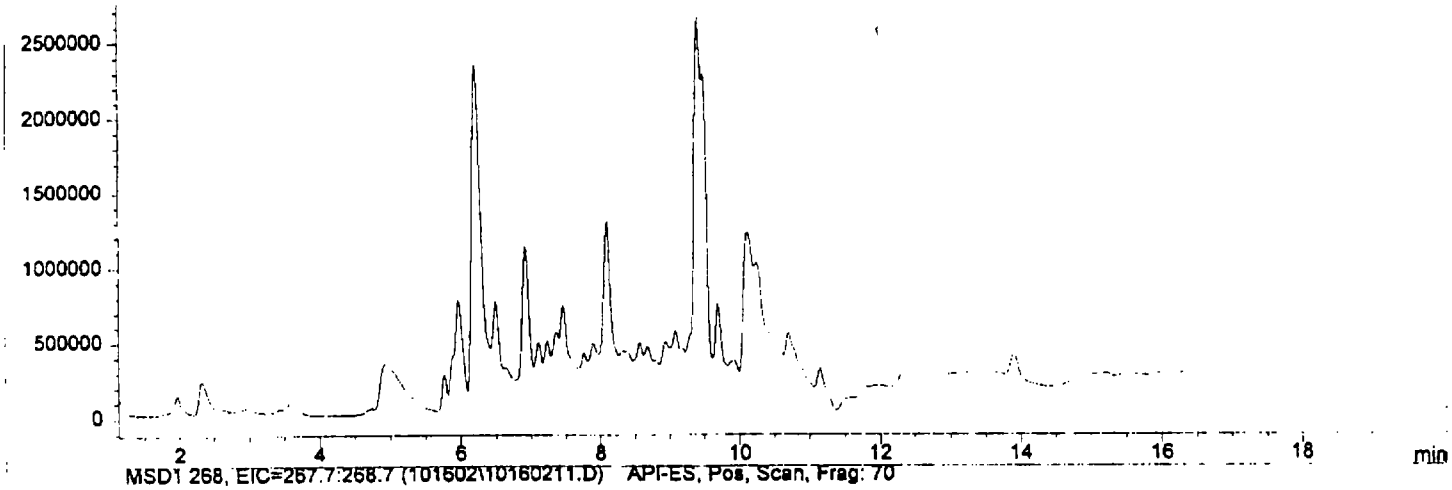
Injection Date : 10/16/2002 8:10:26 PM
Sample Name : 213854 R1
Acq. Operator : GAA

Seq. Line : 10
Location : Vial 10
Inj : 1
Inj Volume : 50 µl

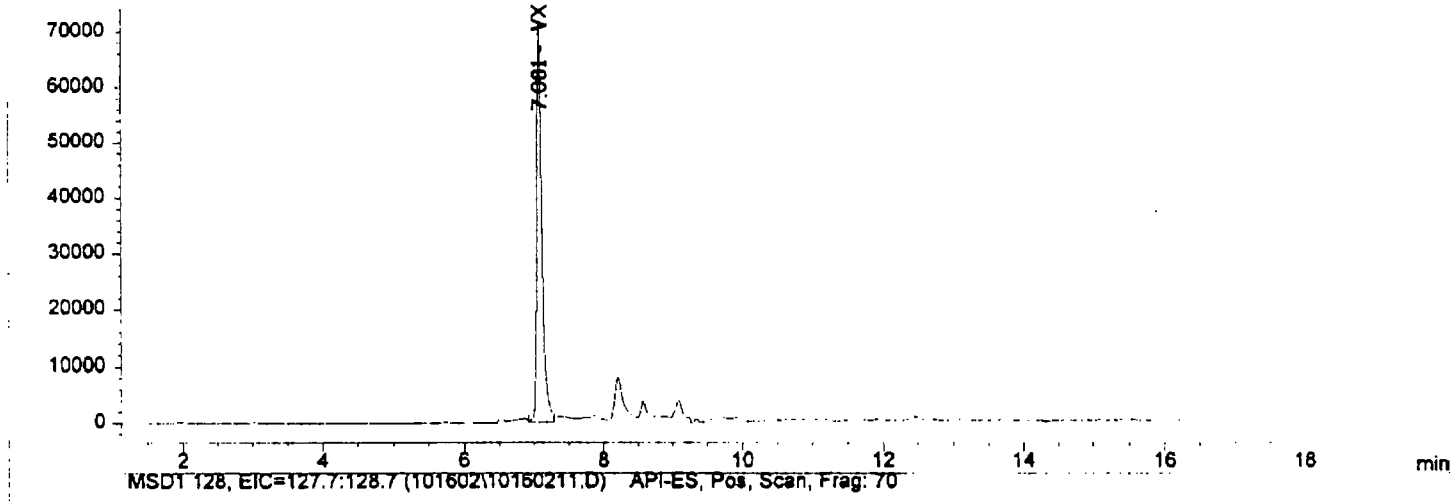
010005

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:54:32 AM by GAA

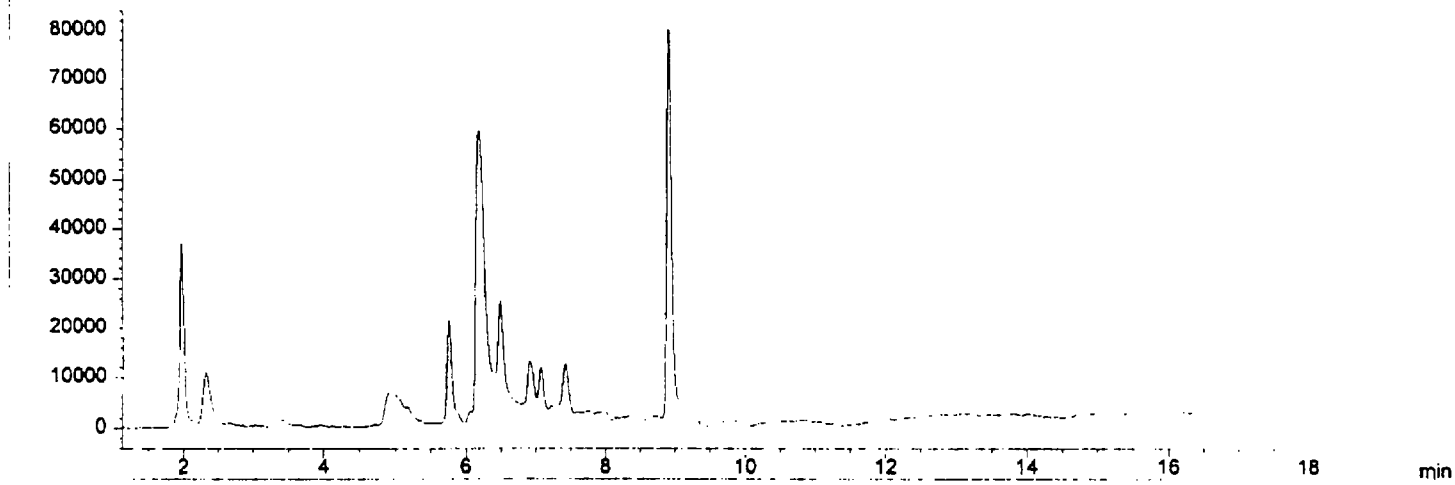
MSD1 TIC, MS File (101602\10160211.D) API-ES, Pos, Scan, Frag: 70



MSD1 268, EIC=267.7:268.7 (101602\10160211.D) API-ES, Pos, Scan, Frag: 70



MSD1 128, EIC=127.7:128.7 (101602\10160211.D) API-ES, Pos, Scan, Frag: 70



Injection Date : 10/16/2002 8:10:26 PM
Sample Name : 213854 R1
Acq. Operator : GAA

Seq. Line : 10
Location : Vial 10
Inj : 1
Inj Volume : 50 µl

010006

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:54:32 AM by GAA

External Standard Report

Sorted By : Signal
Calib. Data Modified : Thursday, October 17, 2002 10:52:50 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime [min]	Type	Area	Amt/Area	Amount [ug/mL]	Grp	Name
7.081	VV	3.62316e5	5.56509e-7	2.01632e-1		VX

Totals : 2.01632e-1

*** End of Report ***

SOUTHWEST RESEARCH INSTITUTE

VX

Results Data Reporting Form

Client: JACADS
Case: JACADS
SDG: 213854
Matrix: Solid
Filename: 10160213
Injection Vol (uL): 50
Dilution Factor: 1
Sample Wt (g): 2.019

System ID: 213854 R2
Client Sample ID: Tray 153 (021002-001)
Project Number: 01.05935.03.001
Instrument: LCMS1
Date Received: 10/07/02
Date Extracted: 10/16/02
Date Analyzed: 10/16/02
Final Volume (mL) : 4

CAS No.	Compound	ug/g
50782-69-9	VX	2.575

Data Reporting Qualifiers

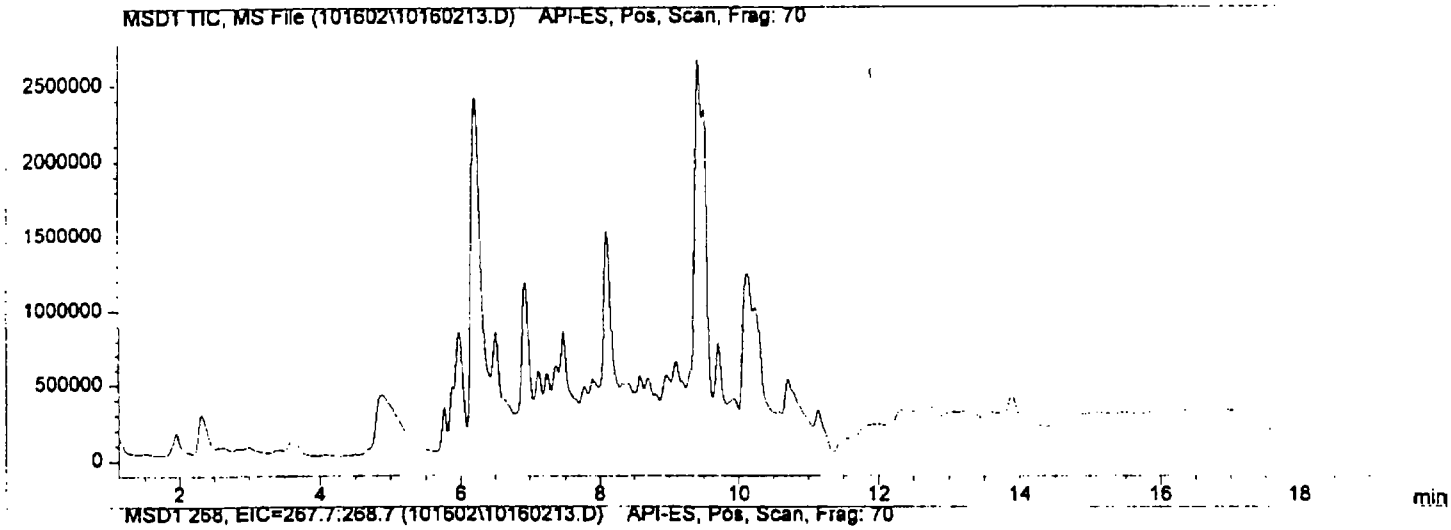
B This flag is used when the analyte is found in the blank as well as in the sample.
E This flag indicates compounds whose concentrations exceed the calibration range.
J Indicates an estimated value.
U Indicates compound was analyzed for, but not detected. Report the minimum detection limit for the sample with U (e.g. 10U) based on necessary concentration dilution action.

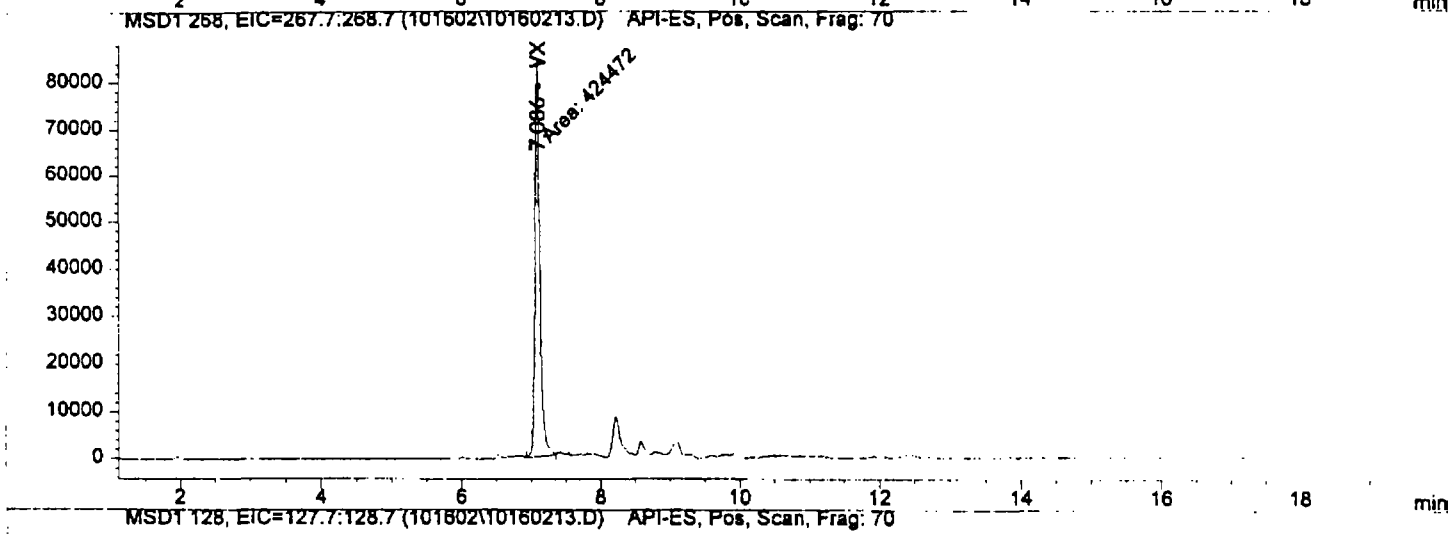
Injection Date : 10/16/2002 8:54:13 PM
Sample Name : 213854 R2
Acq. Operator : GAA

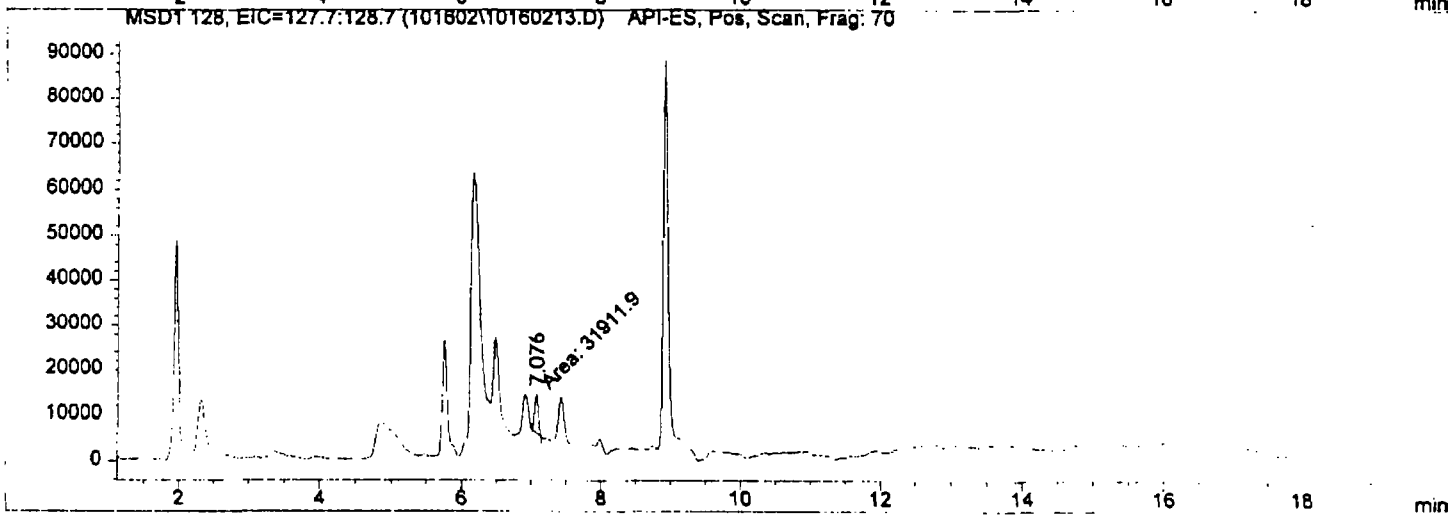
Seq. Line : 12
Location : Vial 12
Inj : 1
Inj Volume : 50 µl

010008

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:54:32 AM by GAA







010009

Injection Date : 10/16/2002 8:54:13 PM
Sample Name : 213854 R2
Acq. Operator : GAA
Seq. Line : 12
Location : Vial 12
Inj : 1
Inj Volume : 50 µl

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:54:32 AM by GAA

External Standard Report

Sorted By : Signal
Calib. Data Modified : Thursday, October 17, 2002 10:52:50 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime [min]	Type	Area	Amt/Area	Amount [ug/mL]	Grp	Name
7.086	MM	4.24472e5	5.56627e-7	2.36273e-1		VX

Totals : 2.36273e-1

*** End of Report ***

SOUTHWEST RESEARCH INSTITUTE

VX

Results Data Reporting Form

Client: JACADS
Case: JACADS
SDG: 213854
Matrix: Solid
Filename: 10160215
Injection Vol (uL): 50
Dilution Factor: 1
Sample Wt (g): 2.000

System ID: 213854 R3
Client Sample ID: Tray 153 (021002-001)
Project Number: 01.05935.03.001
Instrument: LCMS1
Date Received: 10/07/02
Date Extracted: 10/16/02
Date Analyzed: 10/16/02
Final Volume (mL) : 4

CAS No.	Compound	ug/g
50782-69-9	VX	2.638

Data Reporting Qualifiers

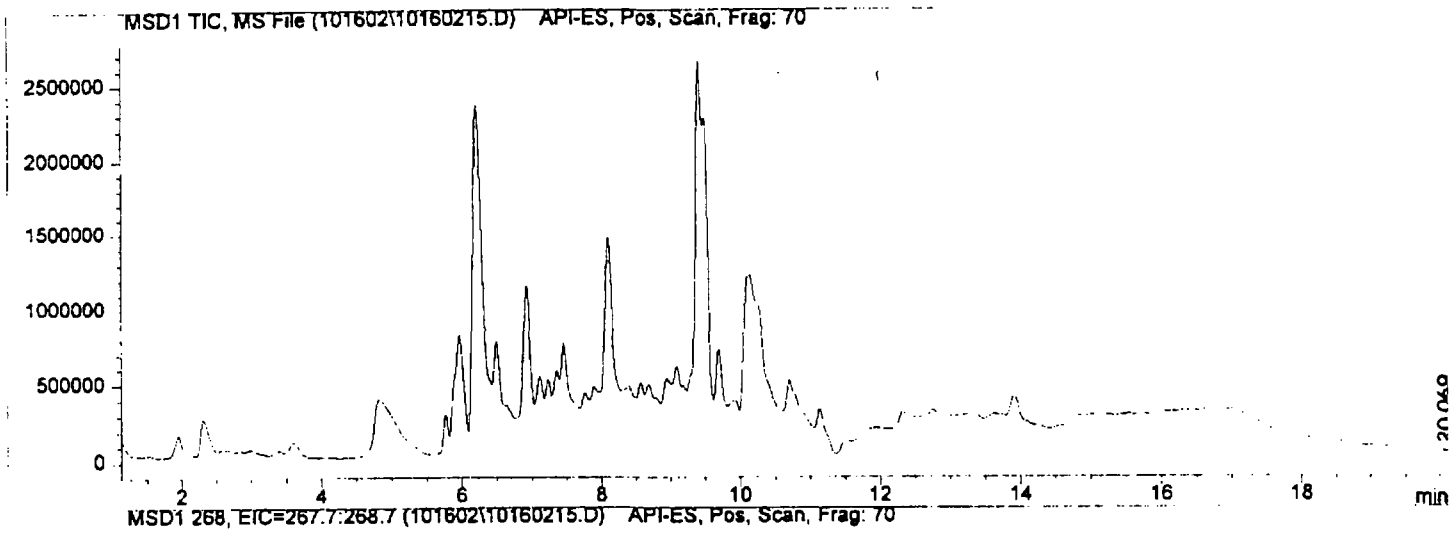
B This flag is used when the analyte is found in the blank as well as in the sample.
E This flag indicates compounds whose concentrations exceed the calibration range.
J Indicates an estimated value.
U Indicates compound was analyzed for, but not detected. Report the minimum detection limit for the sample with U (e.g. 10U) based on necessary concentration dilution action.

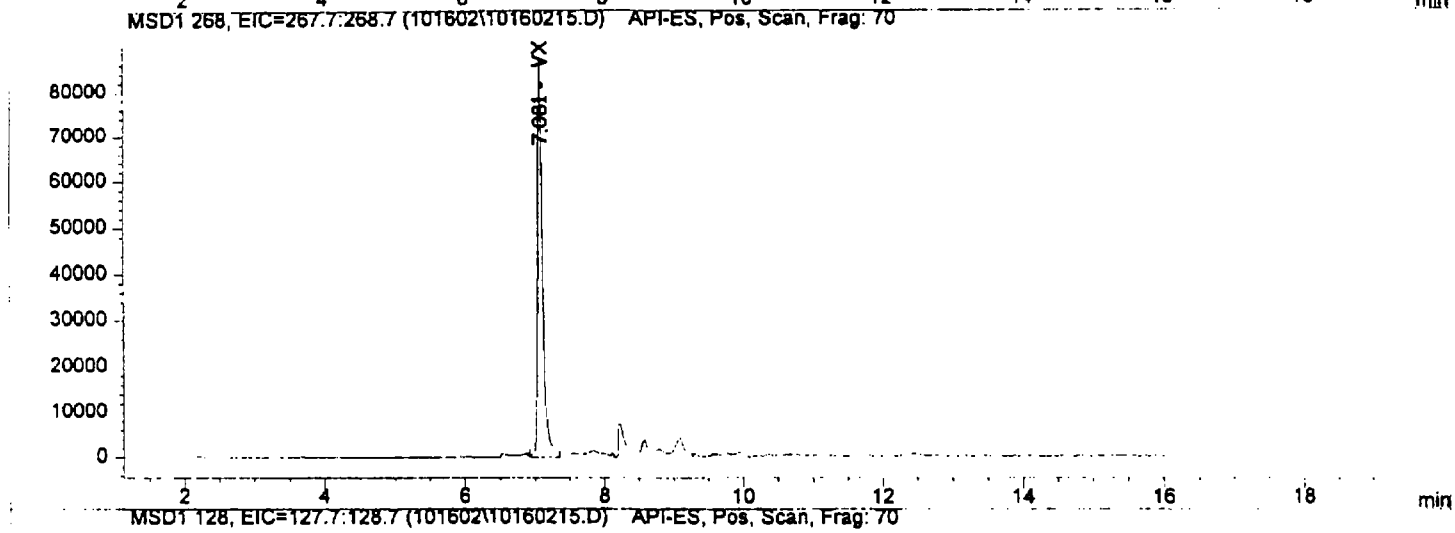
Injection Date : 10/16/2002 9:38:00 PM
Sample Name : 213854 R3
Acq. Operator : GAA

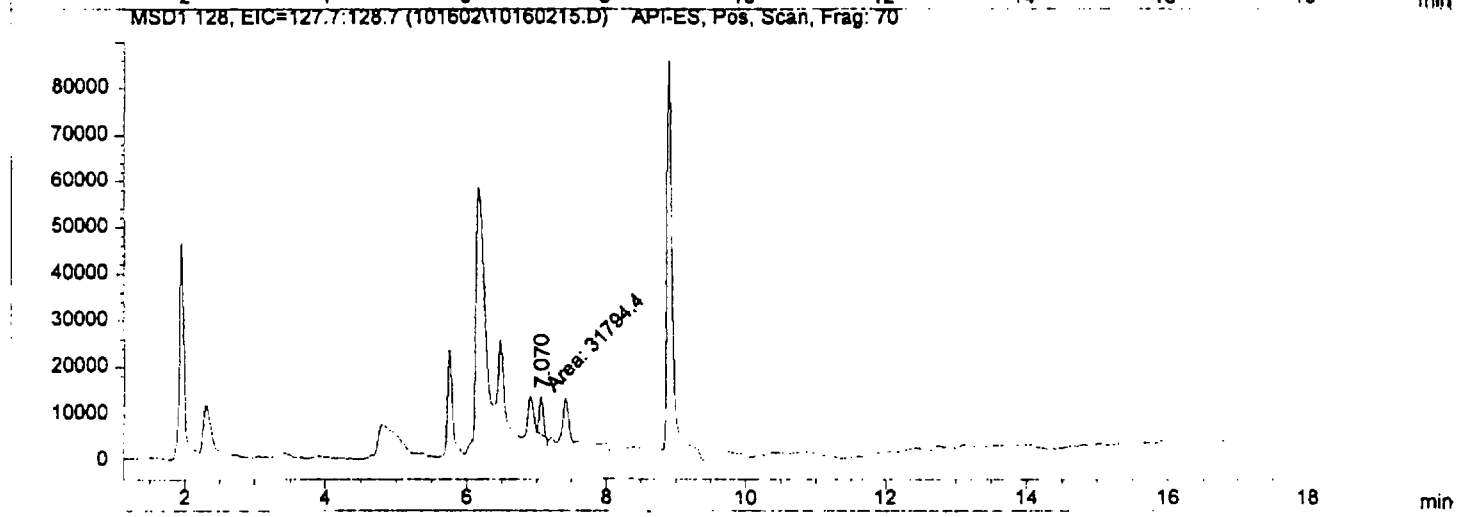
Seq. Line : 14
Location : Vial 14
Inj : 1
Inj Volume : 50 µl

010011

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:54:32 AM by GAA







010012

Injection Date : 10/16/2002 9:38:00 PM
Sample Name : 213854 R3
Acq. Operator : GAA

Seq. Line : 14
Location : Vial 14
Inj : 1
Inj Volume : 50 µl

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:54:32 AM by GAA

External Standard Report

Sorted By : Signal
Calib. Data Modified : Thursday, October 17, 2002 10:52:50 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime [min]	Type	Area	Amt/Area	Amount [ug/mL]	Grp	Name
7.081	VV	4.30812e5	5.56638e-7	2.39806e-1		VX

Totals : 2.39806e-1

*** End of Report ***

SOUTHWEST RESEARCH INSTITUTE
CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order No.: 23238
VTSR: October 7 2002

STANDARDS DATA

Southwest Research Institute
Initial Calibration Data

010013

Project: 01.05935.03.001
Instrument ID: LCMS1

Case: JACADS
LC Column: Supelco C18

SDG: 213854

VX

FILE	DATE	RT	TARGET AREA	THEORETICAL CONC	CALCULATED CONC	PERCENT DEVIATION
10160202	10/16/02 4:53	7.06	1796200	1.000 ug/mL	1.001 ug/mL	0.1%
10160203	10/16/02 5:15	7.07	889296	0.500 ug/mL	0.495 ug/mL	-0.9%
10160204	10/16/02 5:37	7.08	463341	0.250 ug/mL	0.258 ug/mL	3.2%
10160205	10/16/02 5:59	7.09	220406	0.125 ug/mL	0.123 ug/mL	-2.0%
10160206	10/16/02 5:21	7.09	109864	0.063 ug/mL	0.061 ug/mL	-2.5%

r square = 0.999

10-18-02

010014

Calibration Table

CURVE

Calib. Data Modified : Thursday, October 17, 2002 10:52:50 AM

Calculate : External Standard
Based on : Peak Area

Rel. Reference Window : 20.000 %
Abs. Reference Window : 1.000 min
Rel. Non-ref. Window : 5.000 %
Abs. Non-ref. Window : 1.000 min
Uncalibrated Peaks : not reported
Partial Calibration : Yes, identified peaks are recalibrated
Correct All Ret. Times: No, only for identified peaks

Curve Type : Linear
Origin : Ignored
Weight : Equal

Recalibration Settings:
Average Response : Average all calibrations
Average Retention Time: Floating Average New 75%

Calibration Report Options :
Printout of recalibrations within a sequence:
Calibration Table after Recalibration
Normal Report after Recalibration
If the sequence is done with bracketing:
Results of first cycle (ending previous bracket)

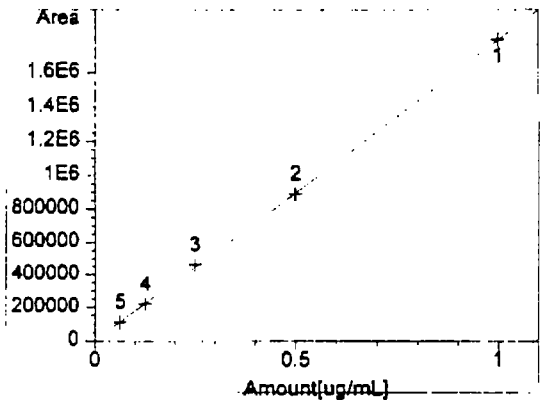
Signal 1: MSD1 268, EIC=267.7:268.7

RetTime [min]	Sig	Lvl	Amount [ug/mL]	Area	Amt/Area	Ref	Grp	Name
7.093	1	5	6.25000e-2	1.09864e5	5.68883e-7			VX
		4	1.25000e-1	2.20406e5	5.67134e-7			
		3	2.50000e-1	4.63341e5	5.39560e-7			
		2	5.00000e-1	8.89296e5	5.62242e-7			
		1	1.00000	1.79620e6	5.56731e-7			

Peak Sum Table

No Entries in table

Calibration Curves



VX at exp. RT: 7.093
MSD1 268, EIC=267.7:268.7
Correlation: 0.99992
Residual Std. Dev.: 10036.01729
Formula: y = mx + b
m: 1.79431e6
b: 524.22461
x: Amount[ug/mL]
y: Area

G. Alw 10-18-02

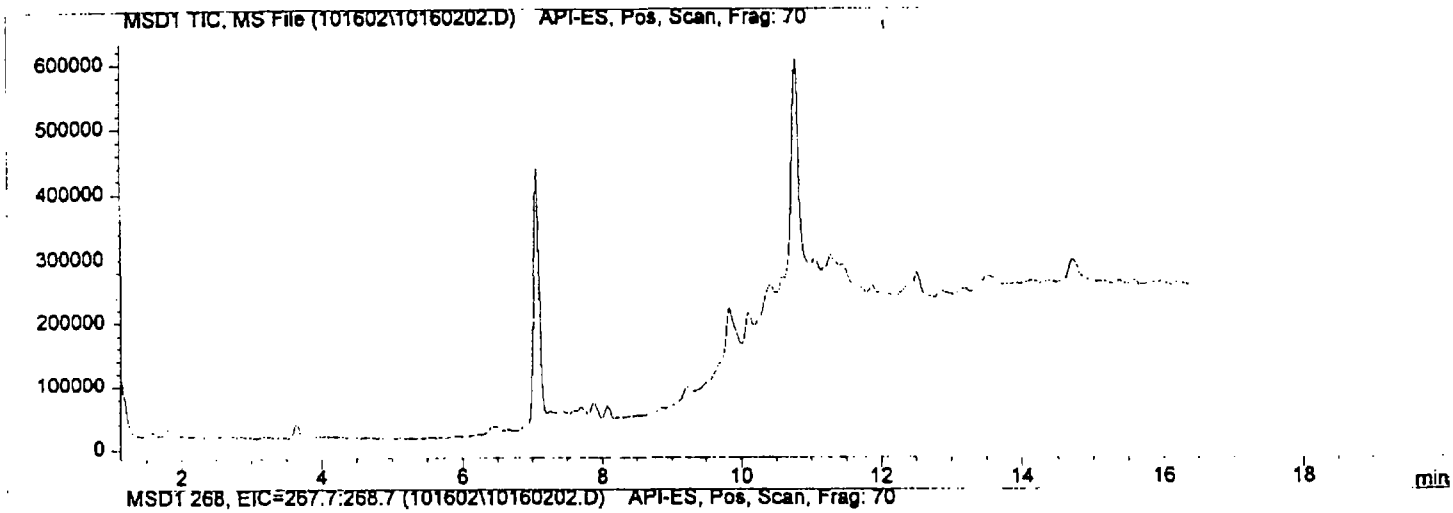
Data File C:\HPCHEM\1\DATA\101602\10160202.D
1 ug/mL.

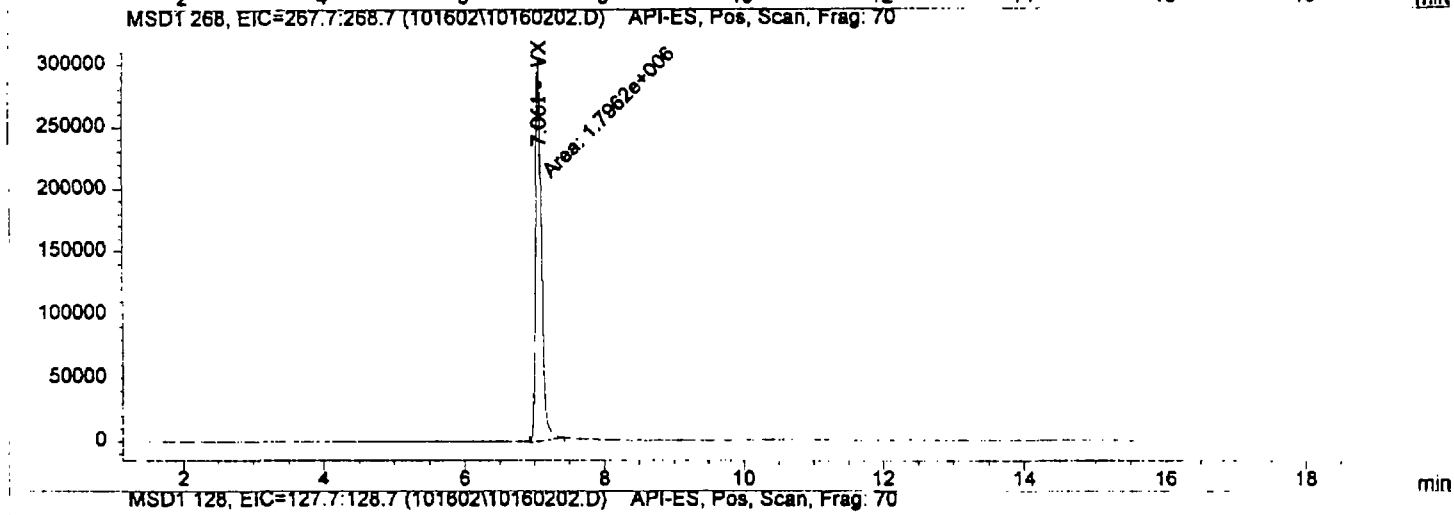
Sample Name: VX STD 1

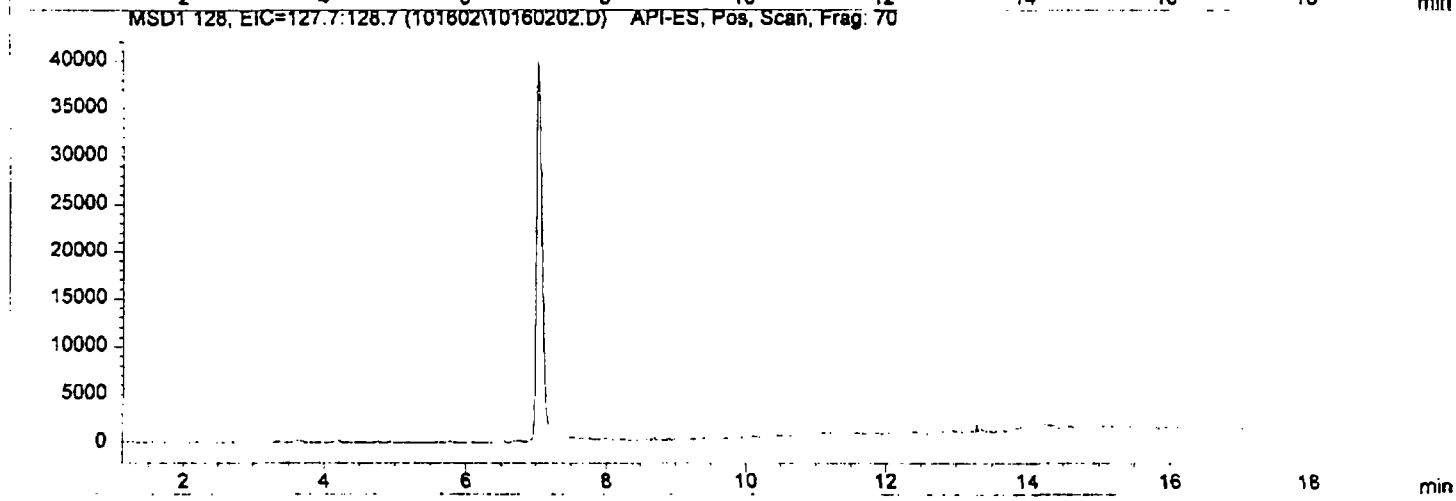
010015

Injection Date	: 10/16/2002 4:53:37 PM	Seq. Line	: 1
Sample Name	: VX STD 1	Location	: Vial 1
Acq. Operator	: GAA	Inj	: 2
		Inj Volume	: 50 µl

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:48:21 AM by GAA







D. Alw 10-18-02

1 ug/mL.

010016

Injection Date : 10/16/2002 4:53:37 PM
Sample Name : VX STD 1
Acq. Operator : GAA
Seq. Line : 1
Location : Vial 1
Inj : 2
Inj Volume : 50 µl

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:48:21 AM by GAA

External Standard Report

Sorted By : Signal
Calib. Data Modified : 10/17/2002 10:48:21 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime [min]	Type	Area	Amt/Area	Amount [ug/mL]	Grp	Name
7.061	MM	1.79620e6	5.56731e-7	1.00000		VX

Totals : 1.00000

1 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

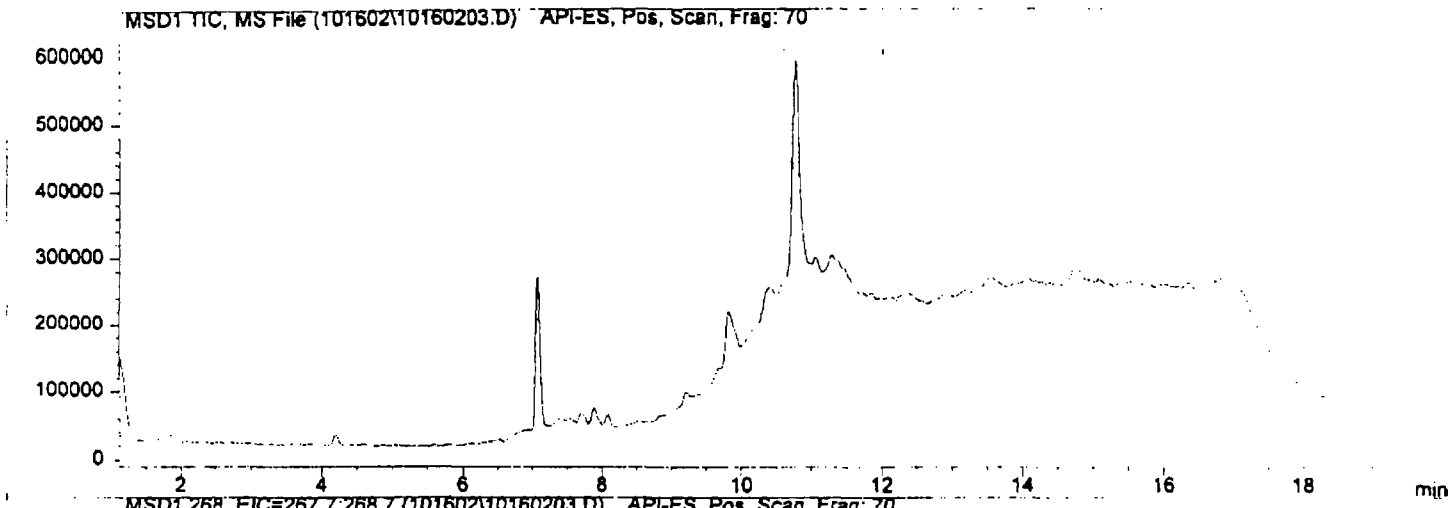
*** End of Report ***

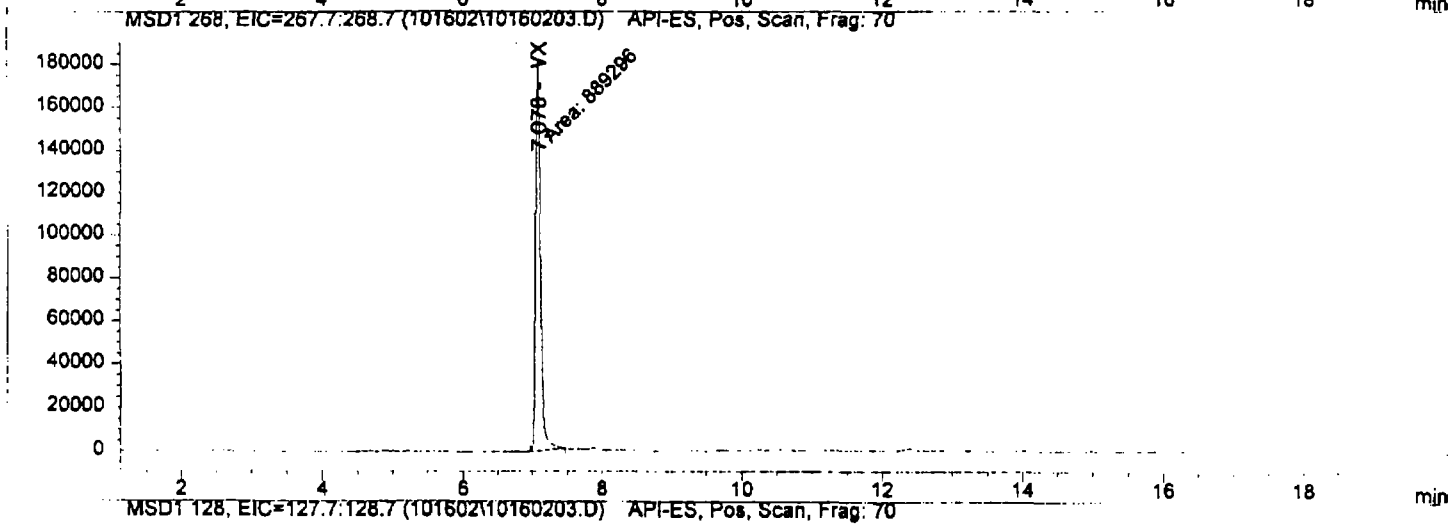
Injection Date : 10/16/2002 5:15:30 PM
Sample Name : VX STD 2
Acq. Operator : GAA

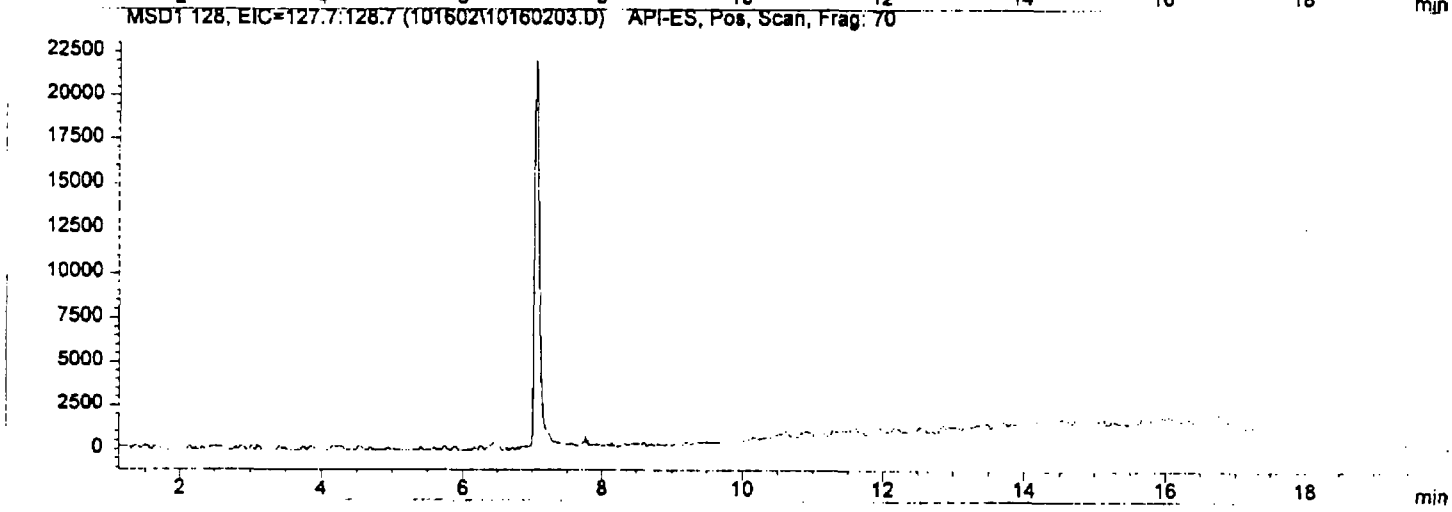
Seq. Line : 2
Location : Vial 2
Inj : 1
Inj Volume : 50 µl

010017

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:49:47 AM by GAA
(modified after loading)







010018

Injection Date : 10/16/2002 5:15:30 PM
Sample Name : VX STD 2
Acq. Operator : GAA

Seq. Line : 2
Location : Vial 2
Inj : 1
Inj Volume : 50 µl

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:49:47 AM by GAA
(modified after loading)

External Standard Report

Sorted By : Signal
Calib. Data Modified : Thursday, October 17, 2002 10:49:46 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime [min]	Type	Area	Amt/Area	Amount [ug/mL]	Grp	Name
7.078	MM	8.89296e5	5.62242e-7	5.00000e-1		VX

Totals : 5.00000e-1

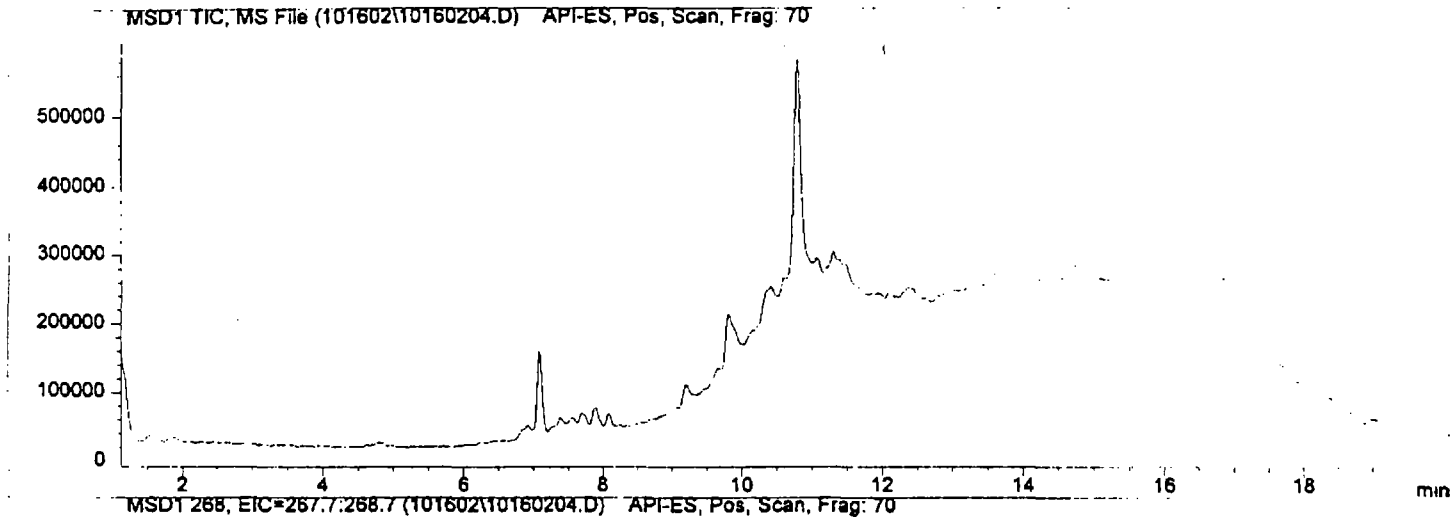
*** End of Report ***

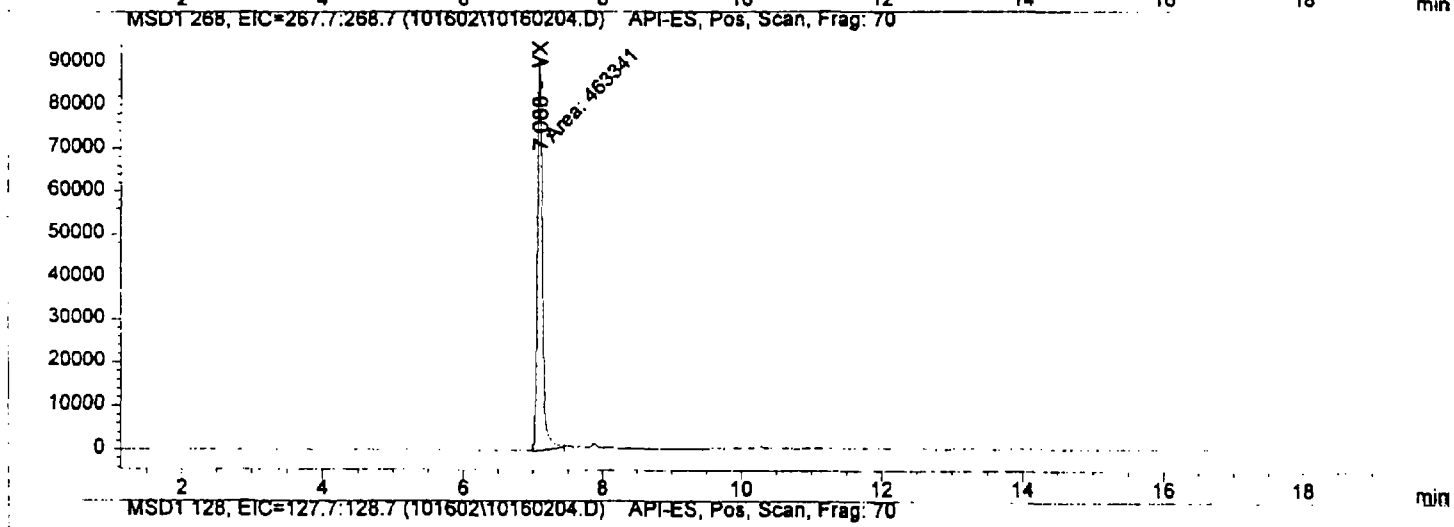
Injection Date : 10/16/2002 5:37:23 PM
Sample Name : VX STD 3
Acq. Operator : GAA

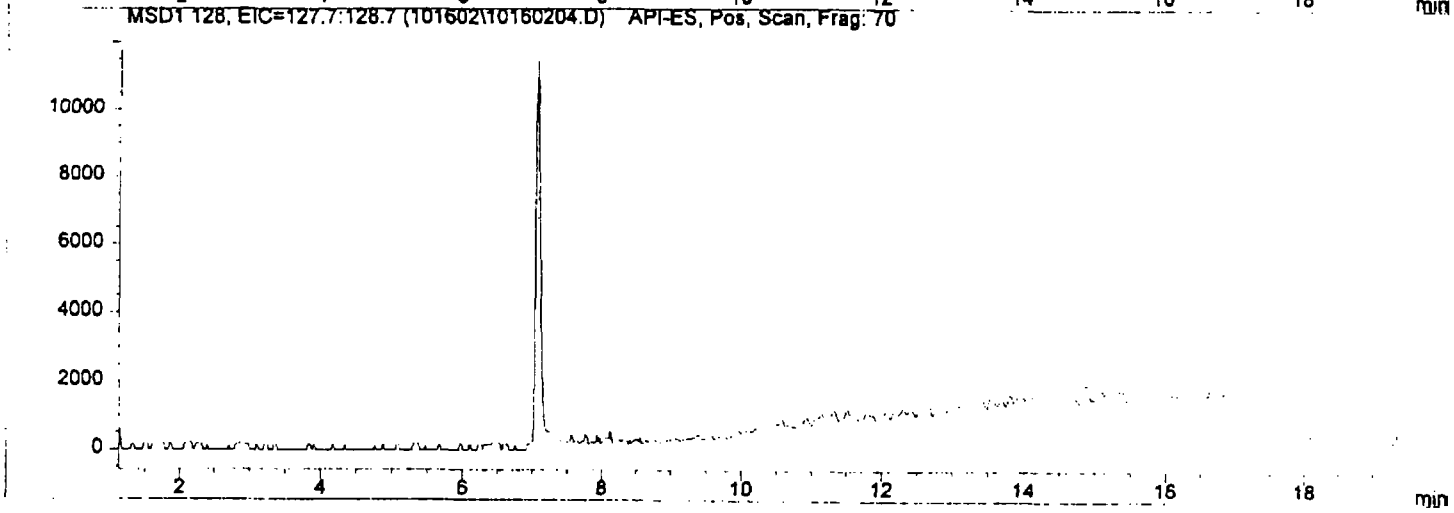
Seq. Line : 3
Location : Vial 3
Inj : 1
Inj Volume : 50 µl

010019

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:50:32 AM by GAA
(modified after loading)







Injection Date : 10/16/2002 5:37:23 PM
Sample Name : VX STD 3
Acq. Operator : GAA

Seq. Line : 3
Location : Vial 3
Inj : 1
Inj Volume : 50 µl

010020

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:50:32 AM by GAA
(modified after loading)

External Standard Report

Sorted By : Signal
Calib. Data Modified : Thursday, October 17, 2002 10:50:31 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime [min]	Type	Area	Amt/Area	Amount [ug/mL]	Grp	Name
7.088	MM	4.63341e5	5.49072e-7	2.54408e-1		VX

Totals : 2.54408e-1

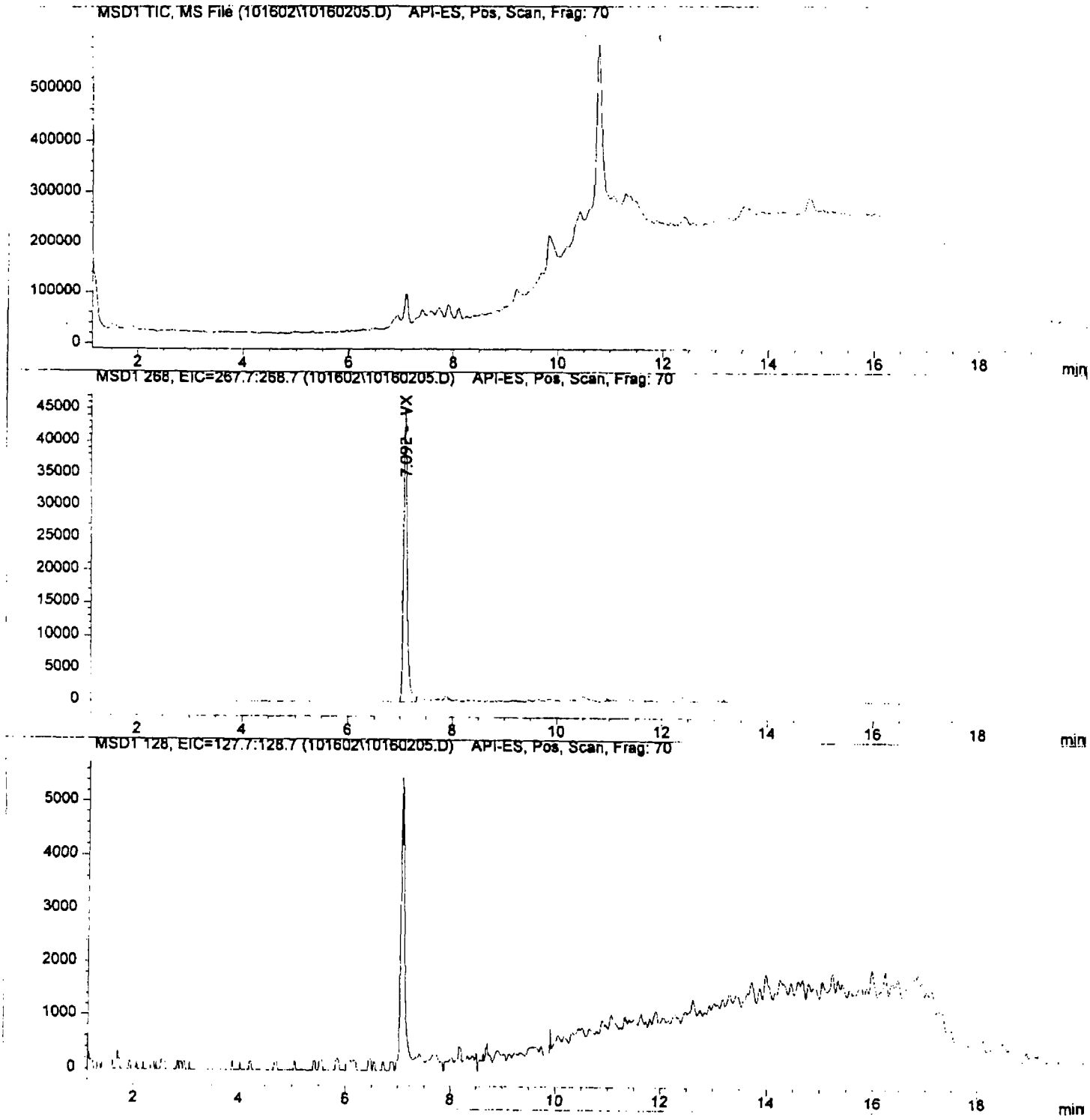
*** End of Report ***

Injection Date : 10/16/2002 5:59:13 PM
Sample Name : VX STD 4
Acq. Operator : GAA

Seq. Line : 4
Location : Vial 4
Inj : 1
Inj Volume : 50 µl

010021

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:51:58 AM by GAA
(modified after loading)



010022

Injection Date : 10/16/2002 5:59:13 PM
Sample Name : VX STD 4
Acq. Operator : GAA

Seq. Line : 4
Location : Vial 4
Inj : 1
Inj Volume : 50 µl

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:51:58 AM by GAA
(modified after loading)

External Standard Report

Sorted By : Signal
Calib. Data Modified : Thursday, October 17, 2002 10:51:57 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime [min]	Type	Area	Amt/Area	Amount [ug/mL]	Grp	Name
7.092	BV	2.20406e5	5.51992e-7	1.21662e-1		VX

Totals : 1.21662e-1

*** End of Report ***

Data File C:\HPCHEM\1\DATA\101602\10160206.D

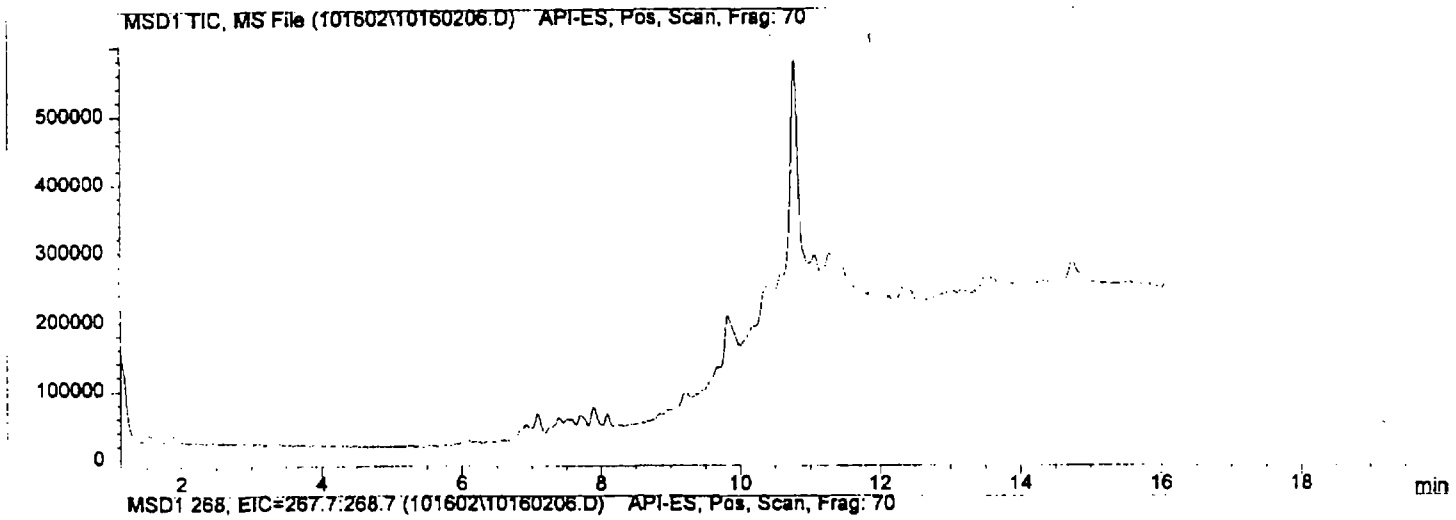
Sample Name: VX STD 5

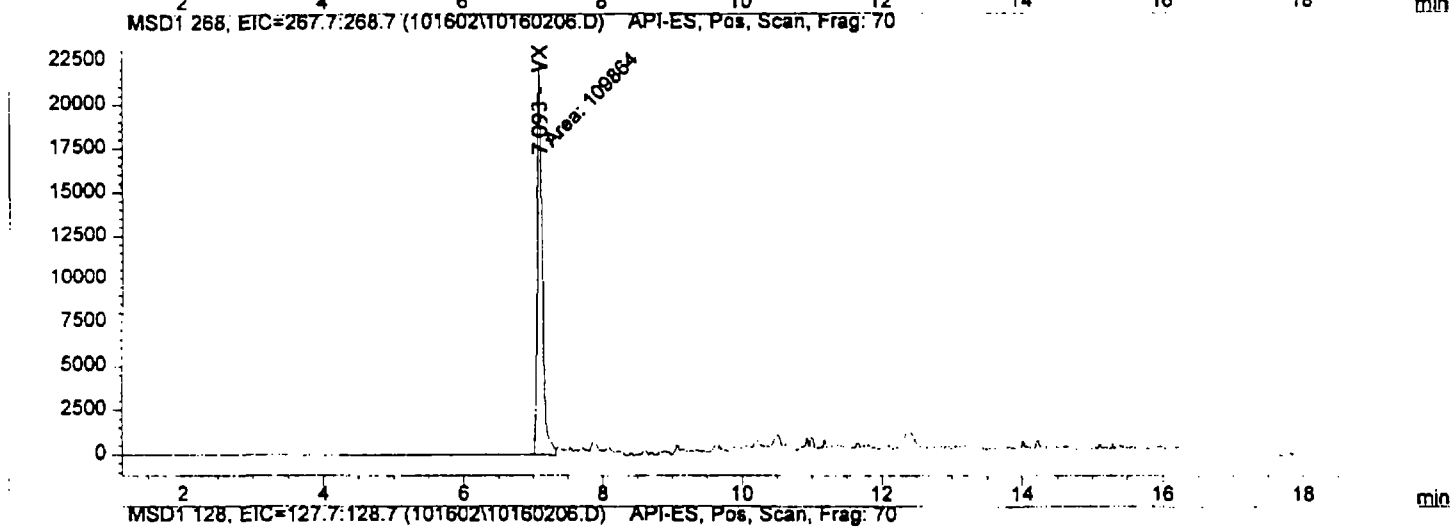
Injection Date : 10/16/2002 6:21:02 PM
Sample Name : VX STD 5
Acq. Operator : GAA

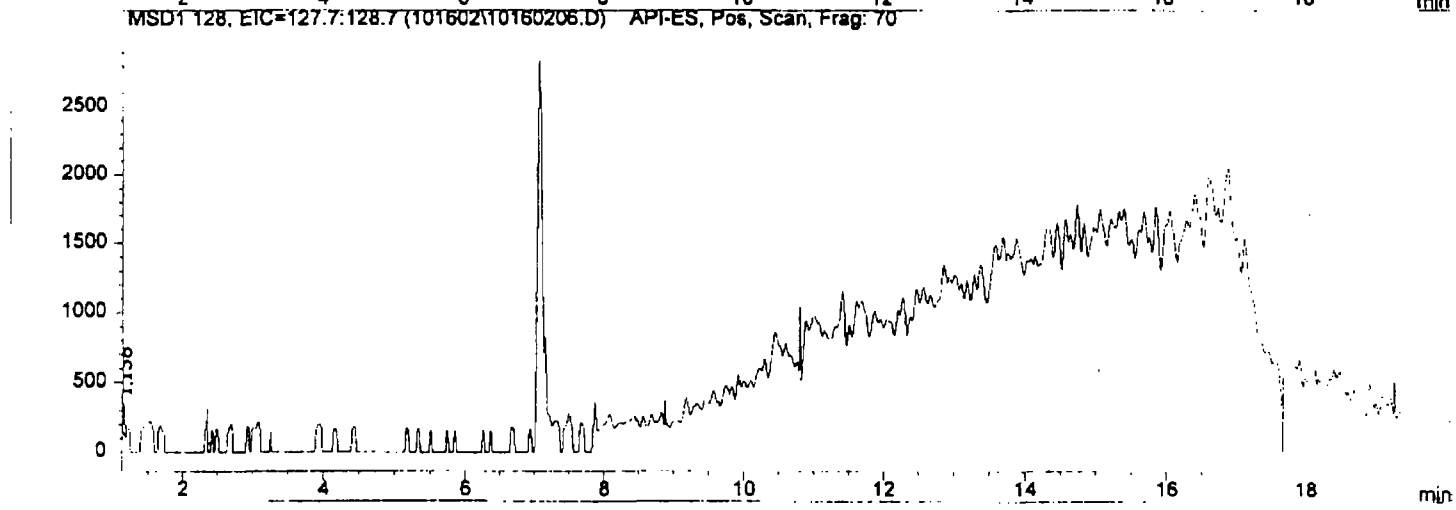
Seq. Line : 5
Location : Vial 5
Inj : 1
Inj Volume : 50 µl

01002?

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:52:51 AM by GAA
(modified after loading)







010024

Injection Date : 10/16/2002 6:21:02 PM
Sample Name : VX STD 5
Acq. Operator : GAA

Seq. Line : 5
Location : Vial 5
Inj : 1
Inj Volume : 50 µl

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:52:51 AM by GAA
(modified after loading)

External Standard Report

Sorted By : Signal
Calib. Data Modified : Thursday, October 17, 2002 10:52:50 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime [min]	Type	Area	Amt/Area	Amount [ug/mL]	Grp	Name
7.093	MM	1.09864e5	5.54657e-7	6.09370e-2		VX

Totals : 6.09370e-2

*** End of Report ***

Southwest Research Institute
Continuing Calibration Data

Project: 01.05935.03.001 Case: JACADS SDG: 213854
Instrument ID: LCMS1 LC Column: Supelco C18

VX

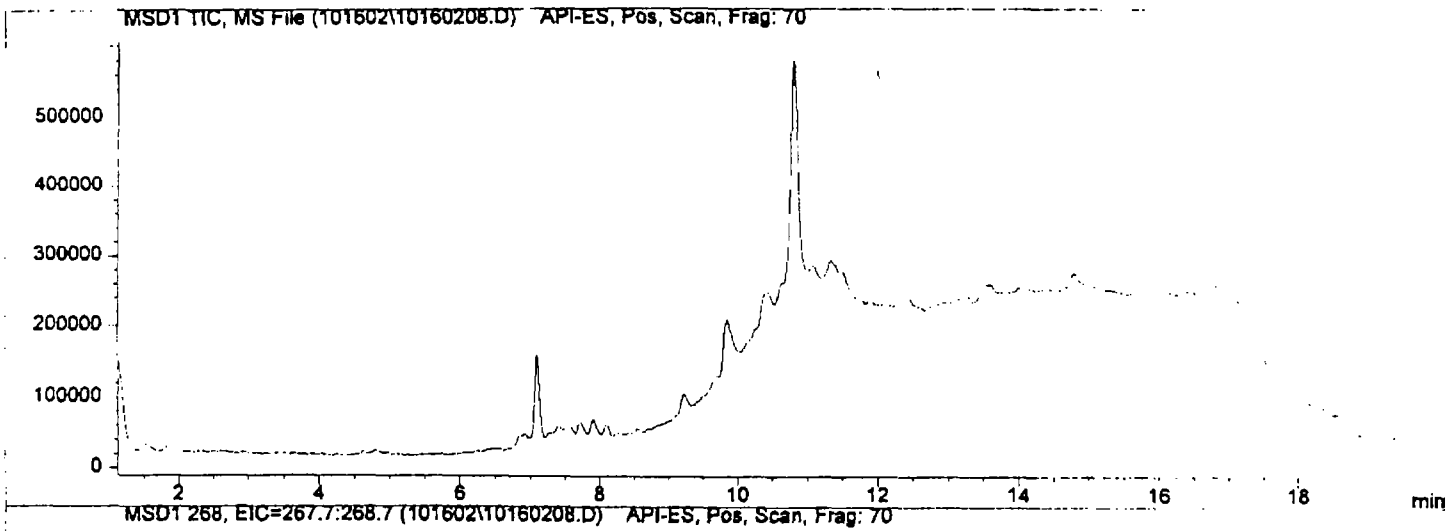
CCS	FILE	DATE	TIME	CALCULATED CONC	PERCENT DEVIATION
Beginning	10160208	10/16/02	07:04 PM	0.275	-9.9%
Ending	10160217	10/16/02	10:21 PM	0.276	-10.4%

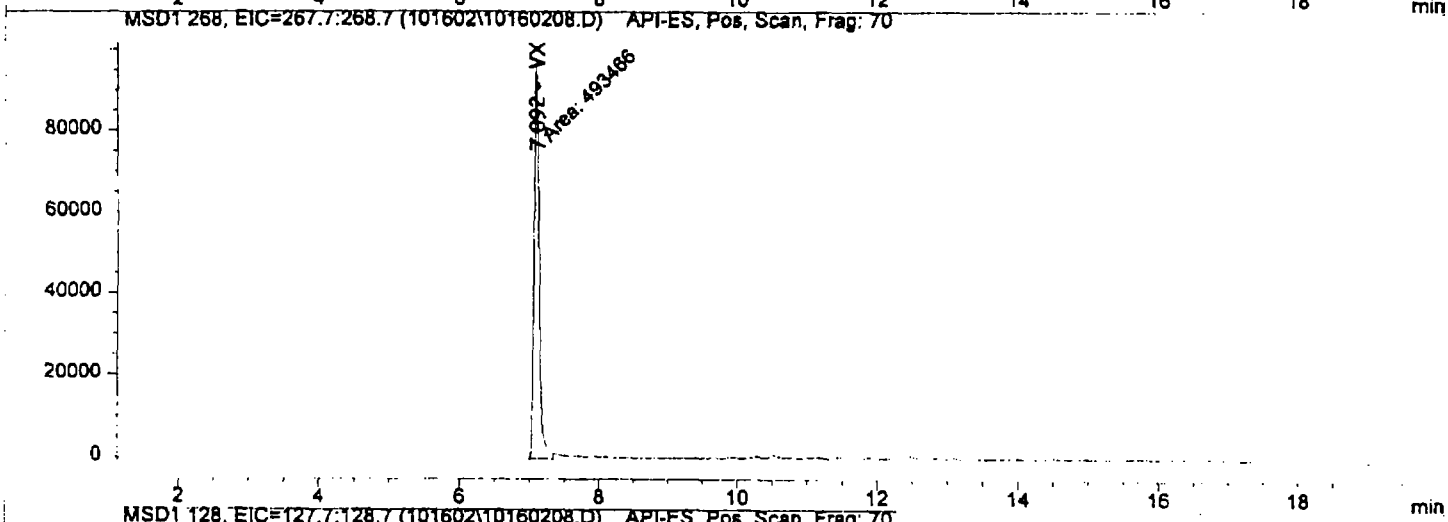
H. Alw 8
10-18-02

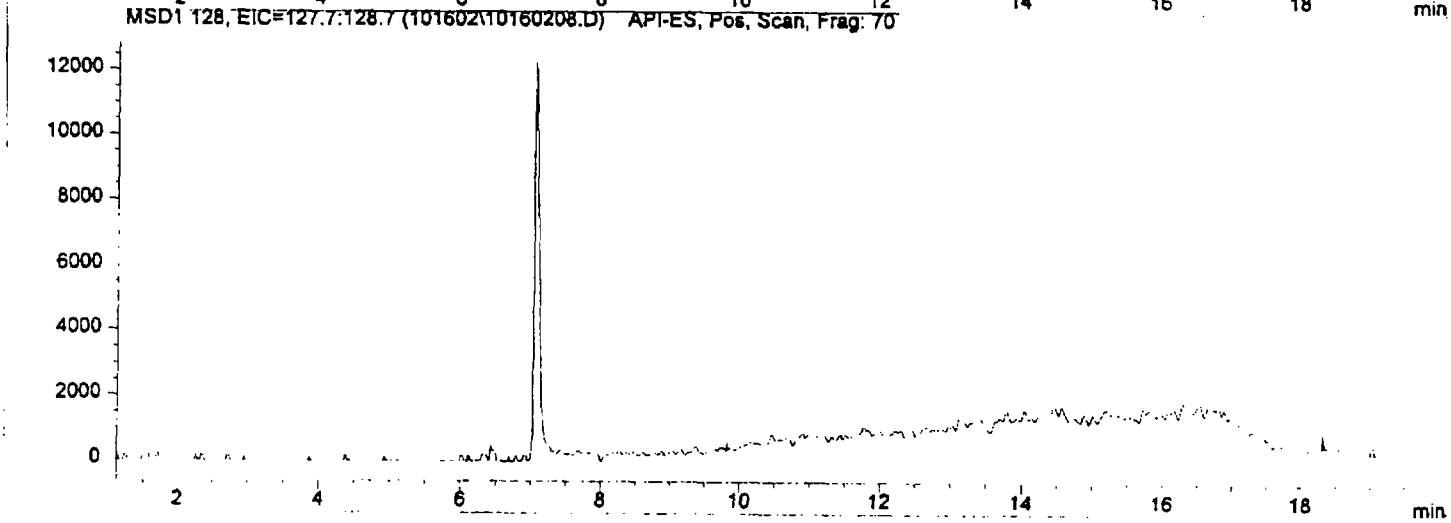
010026

Injection Date	: 10/16/2002 7:04:47 PM	Seq. Line :	7
Sample Name	: VX CCS1 STD 3	Location :	Vial 7
Acq. Operator	: GAA	Inj :	1
		Inj Volume :	50 µl

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:54:32 AM by GAA







010027

Injection Date : 10/16/2002 7:04:47 PM
Sample Name : VX CCS1 STD 3
Acq. Operator : GAA

Seq. Line : 7
Location : Vial 7
Inj : 1
Inj Volume : 50 µl

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:54:32 AM by GAA

External Standard Report

Sorted By : Signal
Calib. Data Modified : Thursday, October 17, 2002 10:52:50 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime [min]	Type	Area	Amt/Area	Amount [ug/mL]	Grp	Name
7.092	MM	4.93466e5	5.56724e-7	2.74724e-1		VX

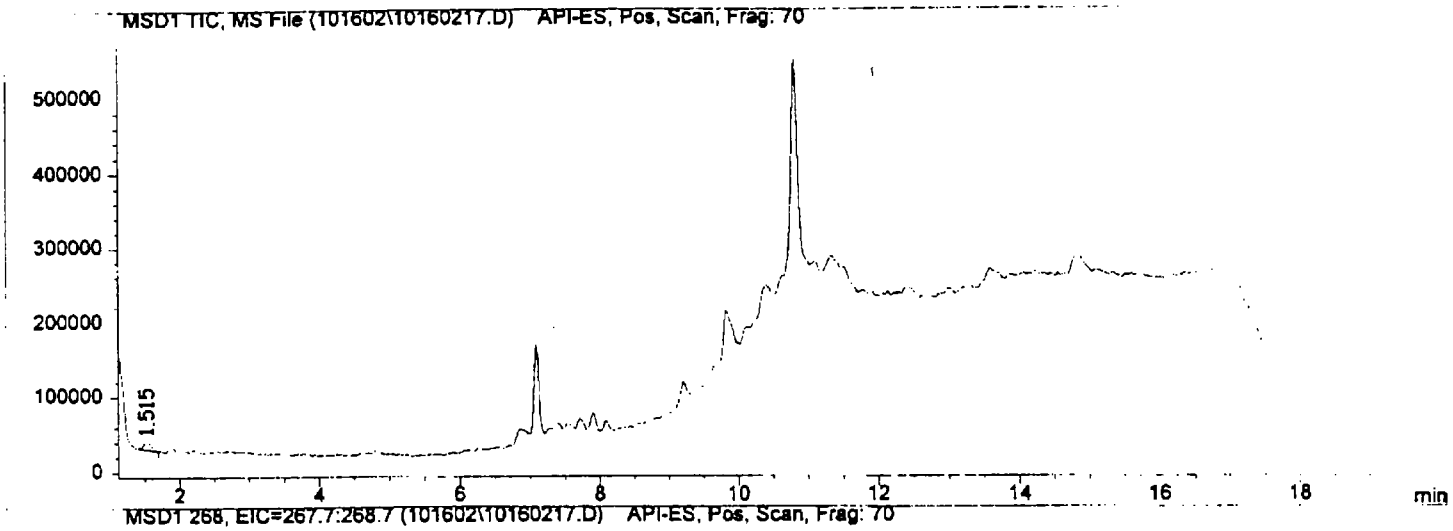
Totals : 2.74724e-1

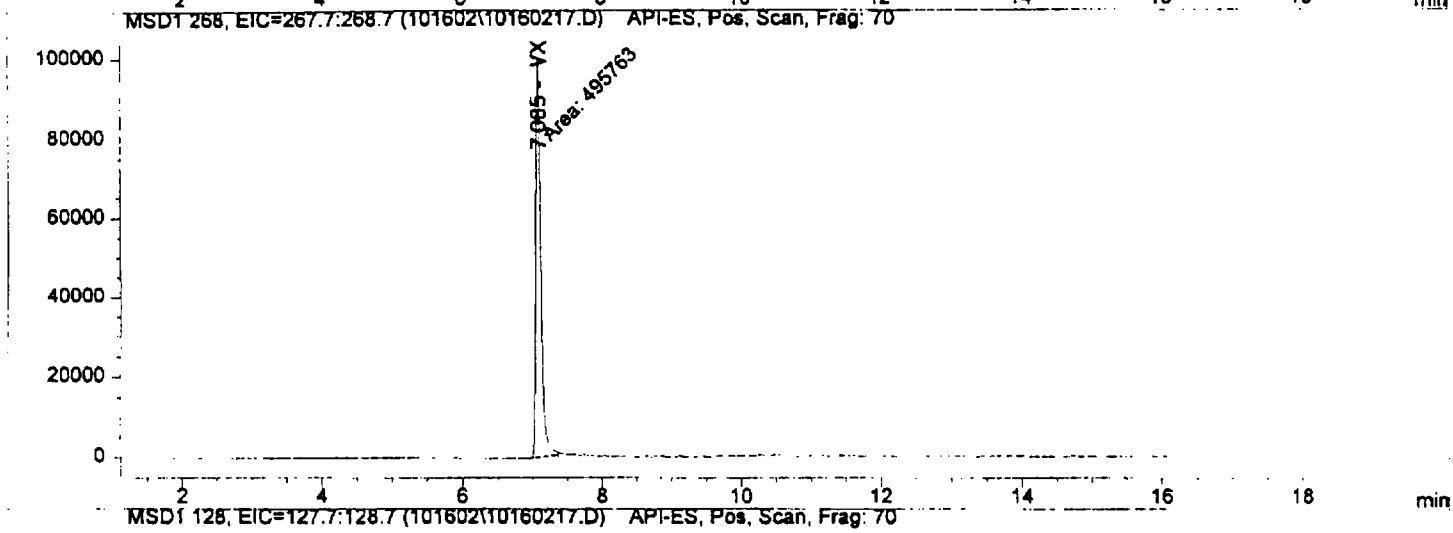
*** End of Report ***

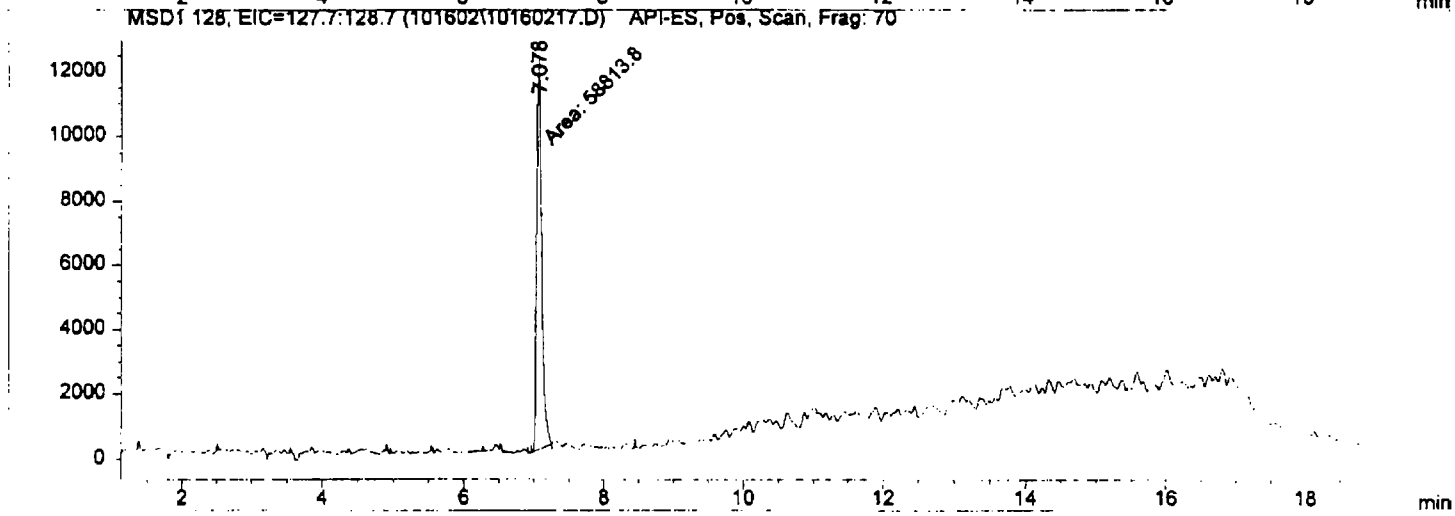
Data File C:\HPCHEM\1\DATA\101602\10160217.D Sample Name: VX CCS2 STD 3

010028

Injection Date	: 10/16/2002 10:21:40 PM	Seq. Line	: 16
Sample Name	: VX CCS2 STD 3	Location	: Vial 16
Acq. Operator	: GAA	Inj	: 1
		Inj Volume	: 50 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_FS.M		
Last changed	: 10/11/2002 5:12:48 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX1016.M		
Last changed	: 10/17/2002 10:54:32 AM by GAA		







010029

Injection Date : 10/16/2002 10:21:40 PM
Sample Name : VX CCS2 STD 3
Acq. Operator : GAA

Seq. Line : 16
Location : Vial 16
Inj : 1
Inj Volume : 50 µl

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:54:32 AM by GAA

External Standard Report

Sorted By : Signal
Calib. Data Modified : Thursday, October 17, 2002 10:52:50 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime [min]	Type	Area	Amt/Area	Amount [ug/mL]	Grp	Name
7.085	MM	4.95763e5	5.56726e-7	2.76004e-1		VX

Totals : 2.76004e-1

*** End of Report ***

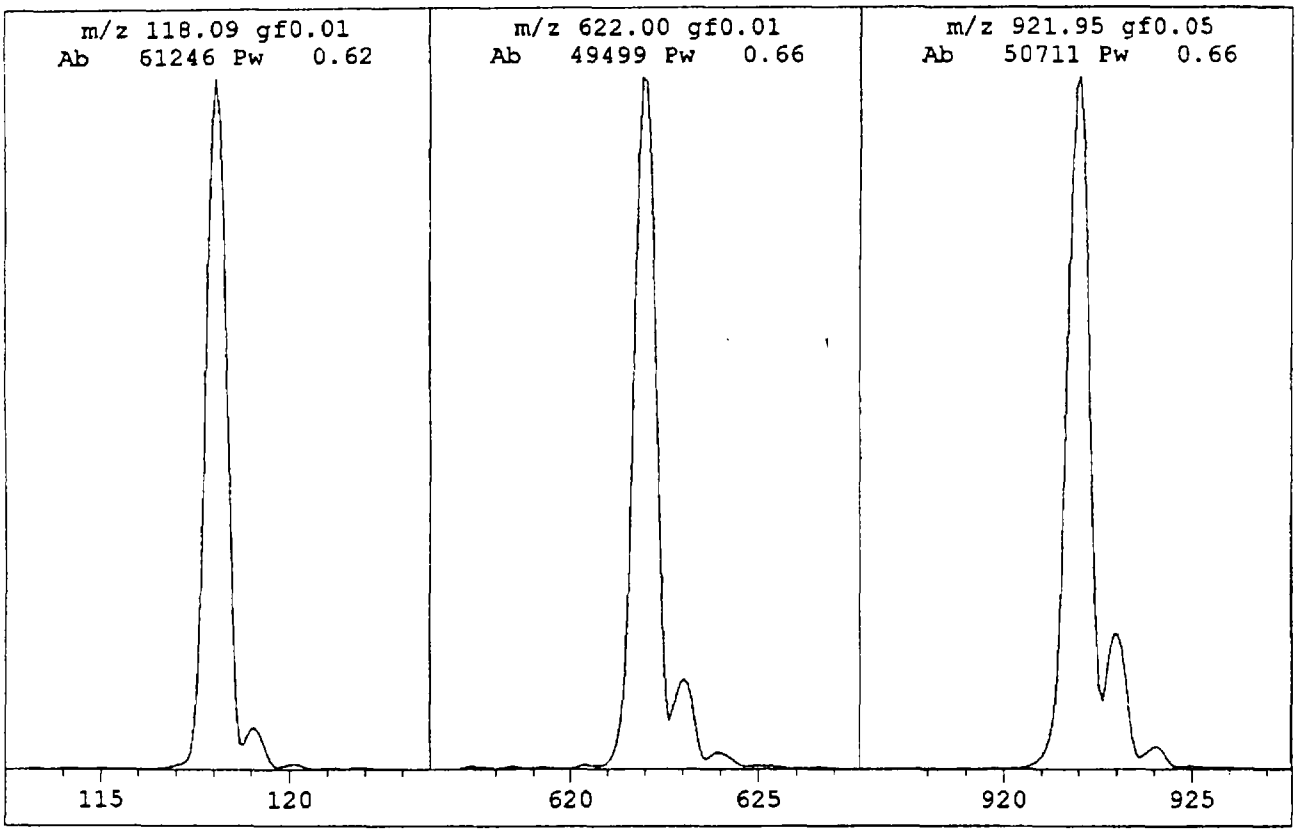
SOUTHWEST RESEARCH INSTITUTE
CLIENT: JACADS
SDG: 213854
SwRI Project Number: 05935.03.001
SwRI Work Order No.: 23238
VTSR: October 7 2002

RAW QC DATA

MSD Check Tune API_ES Positive mode 010030

Instrument : Instrument 1

Wed Oct 16 14:05:21 2002 C:\HPCHEM\1\1946CTUN\ATUNES.TUN



Mass Axis Calibration			Resolution Setting			Signal Intensity
Target Mass	Tune Mass	P/F	Target PW	Peak Width	P/F	Signal
118.08	118.09	pass	0.65	0.62	pass	61248
622.03	622.00	pass	0.65	0.66	pass	49528
922.01	921.95	pass	0.65	0.66	pass	50720

Results Summary

	Mass Axis	Peak Width
Allowed Diff.	<=0.13 amu	<=0.1 amu
Actual Diff.		
Mass 1	pass	pass
Mass 2	pass	pass
Mass 3	pass	pass

S. Ahmed
10-18-02

SOUTHWEST RESEARCH INSTITUTE

VX

Results Data Reporting Form

Client: JACADS
Case: JACADS
SDG: 213854
Matrix: Liquid
Filename: 10160209
Injection Vol (uL): 50
Dilution Factor: 1

System ID: Solv Blk
Client Sample ID: na
Project Number: 01.05935.03.001
Instrument: LCMS1
Date Received: 10/07/02
Date Extracted: 10/16/02
Date Analyzed: 10/16/02
Final Volume (mL) : 4

CAS No.	Compound	ug/mL
50782-69-9	VX	0.063 U

Date Reporting Qualifiers

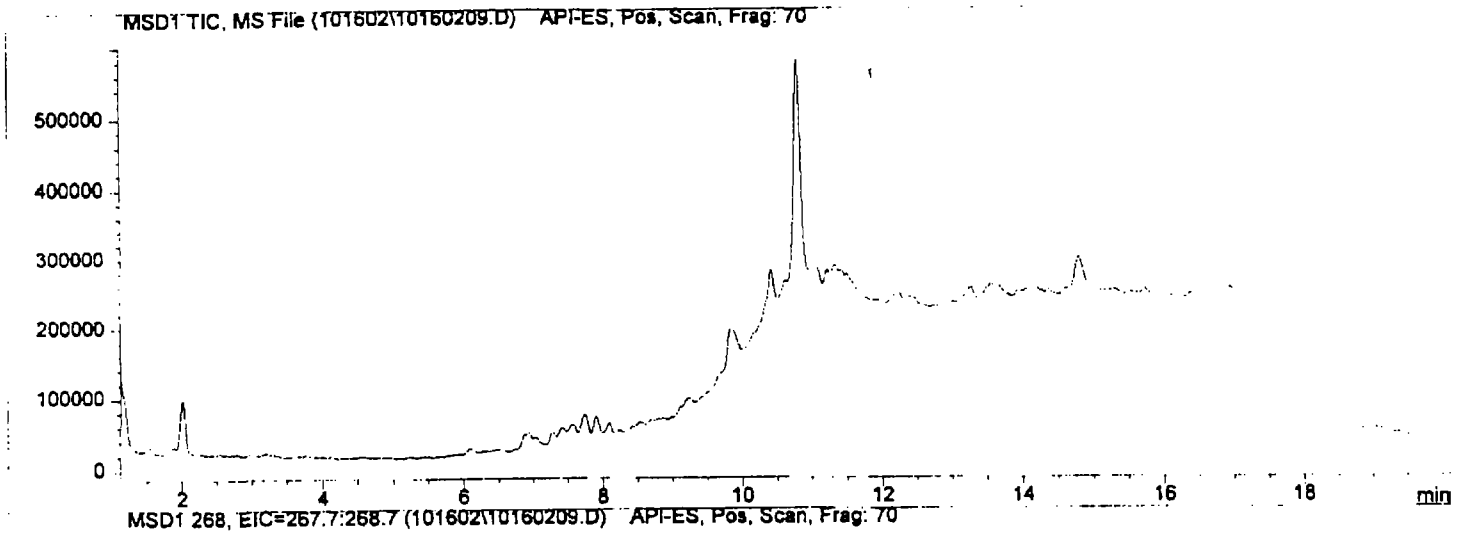
B This flag is used when the analyte is found in the blank as well as in the sample.
E This flag indicates compounds whose concentrations exceed the calibration range.
J Indicates an estimated value.
U Indicates compound was analyzed for, but not detected. Report the minimum detection limit for the sample with U (e.g. 10U) based on necessary concentration dilution action.

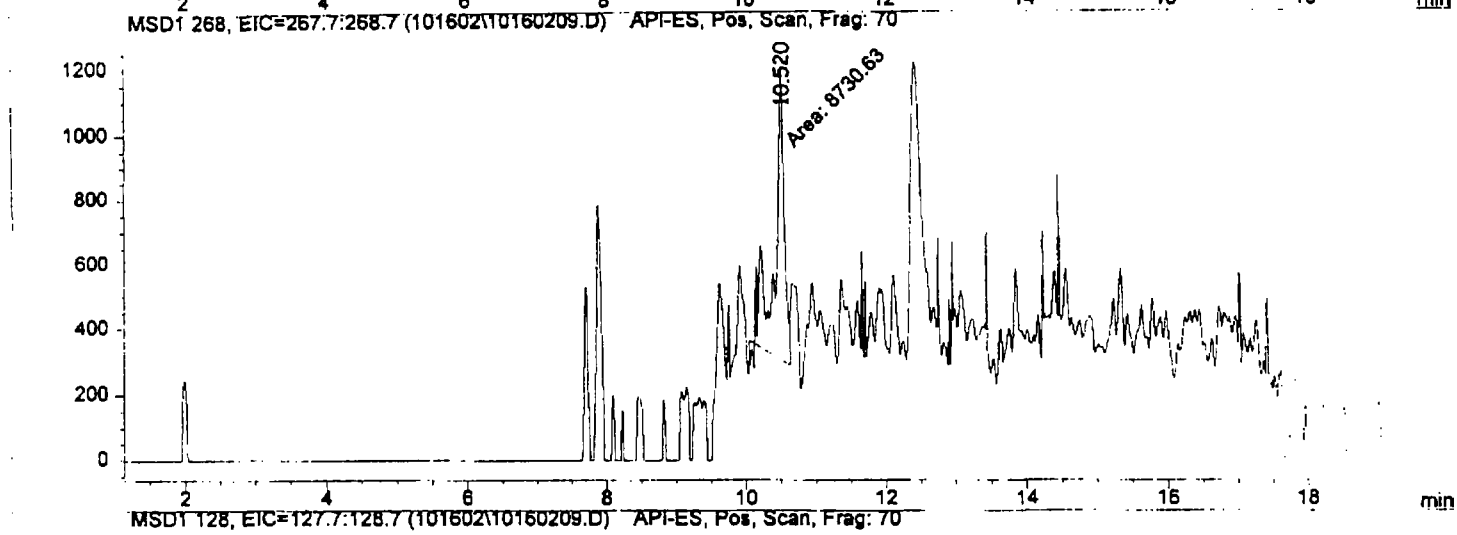
Data File C:\HPCHEM\1\DATA\101602\10160209.D

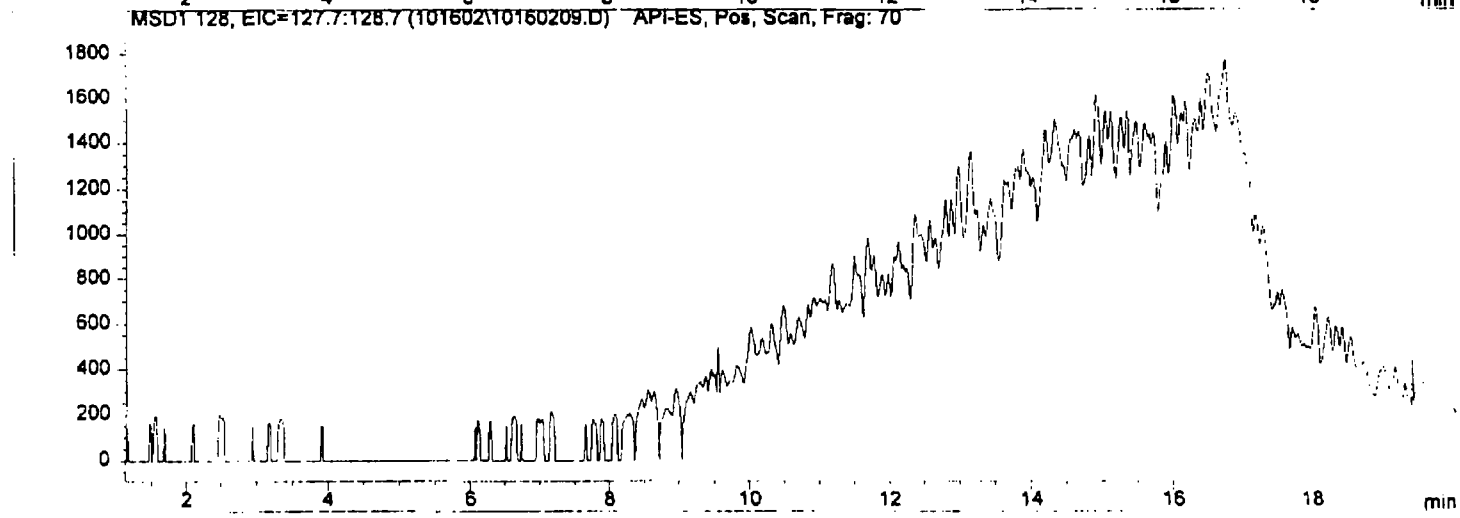
Sample Name: SOLV BLK

Injection Date	: 10/16/2002 7:26:39 PM	Seq. Line	: 8
Sample Name	: SOLV BLK	Location	: Vial 8
Acq. Operator	: GAA	Inj	: 1
		Inj Volume	: 50 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_FS.M		
Last changed	: 10/11/2002 5:12:48 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX1016.M		
Last changed	: 10/17/2002 10:54:32 AM by GAA		

010032







010033

Injection Date : 10/16/2002 7:26:39 PM
Sample Name : SOLV BLK
Acq. Operator : GAA

Seq. Line : 8
Location : Vial 8
Inj : 1
Inj Volume : 50 µl

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:54:32 AM by GAA

External Standard Report

Sorted By : Signal
Calib. Data Modified : Thursday, October 17, 2002 10:52:50 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime [min]	Type	Area	Amt/Area	Amount [ug/mL]	Grp	Name
7.093		-	-	-		VX

Totals : 0.00000

1 Warnings or Errors :

Warning : Calibrated compound(s) not found

Area Percent Report

Sorted By : Signal
Calib. Data Modified : Thursday, October 17, 2002 10:52:50 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 TIC, MS File

Uncalibrated Peaksn.a.

Signal 2: MSD1 268, EIC=267.7:268.7

Peak #	RetTime [min]	Type	Width [min]	Area	Area %	Name
1	7.093		0.0000	0.00000	0.0000	VX

Totals : 0.00000 0.0000

Uncalibrated Peaks:

Peak #	RetTime [min]	Type	Width [min]	Area	Area %	Name
1	10.520	MM	0.1602	8730.62598	100.0000	?

Uncalib. totals : 8730.62598 100.0000

SOUTHWEST RESEARCH INSTITUTE

VX

Results Data Reporting Form

010034

Client: JACADS
Case: JACADS
SDG: 213854
Matrix: Liquid
Filename: 10160210
Injection Vol (uL): 50
Dilution Factor: 2

System ID: Solv Blk MS
Client Sample ID: n/a
Project Number: 01.05935.03.001
Instrument: LCMS1
Date Received: 10/07/02
Date Extracted: 10/16/02
Date Analyzed: 10/16/02
Final Volume (mL) : 4

CAS No.	Compound	ug/mL
50782-69-9	VX	0.526

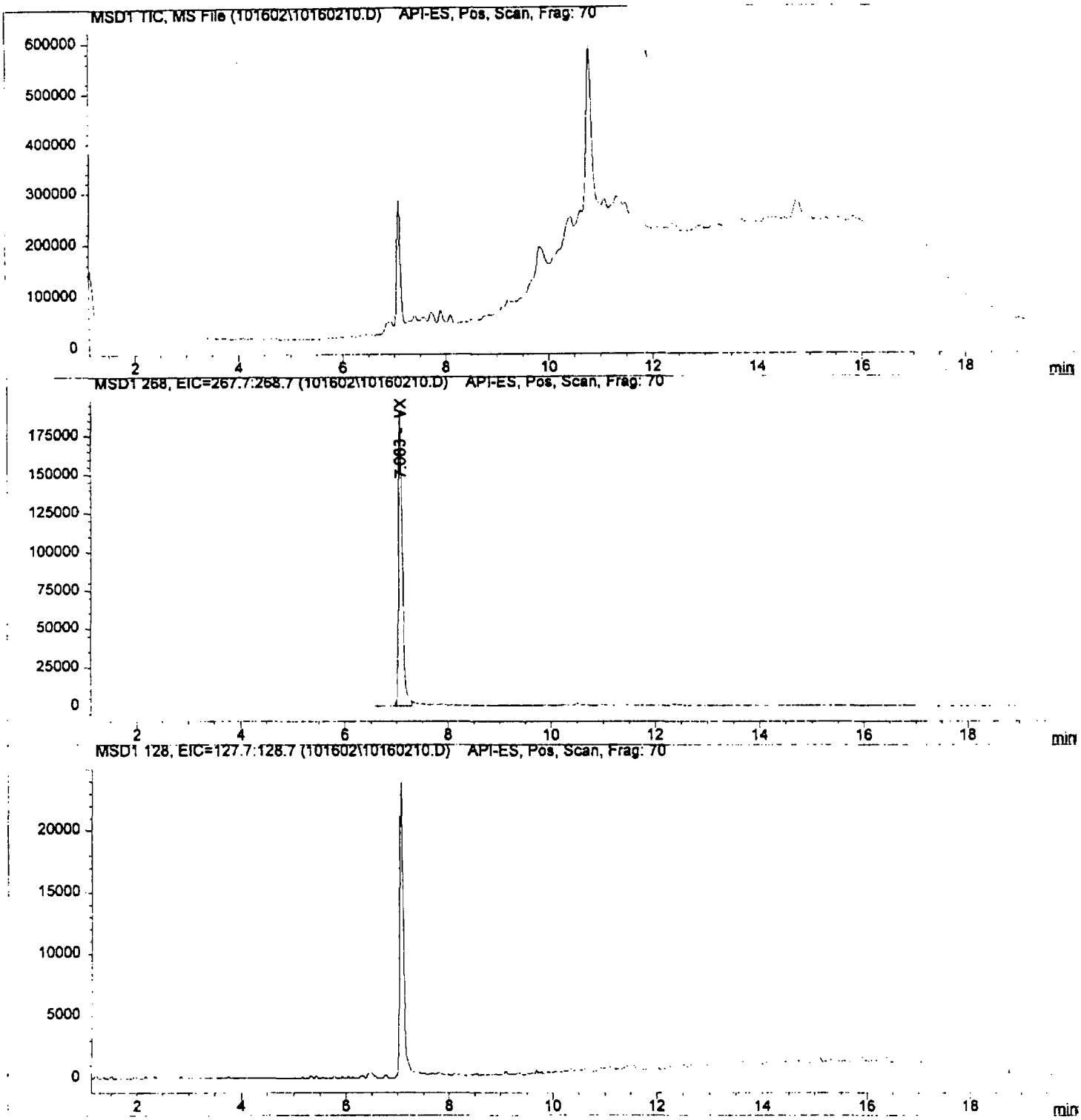
Data Reporting Qualifiers

B This flag is used when the analyte is found in the blank as well as in the sample.
E This flag indicates compounds whose concentrations exceed the calibration range.
J Indicates an estimated value.
U Indicates compound was analyzed for, but not detected. Report the minimum detection limit for the sample with U (e.g. 10U) based on necessary concentration dilution action.

Data File C:\HPCHEM\1\DATA\101602\10160210.D Sample Name: SOLV BLK MS 2X

Injection Date	: 10/16/2002 7:48:29 PM	Seq. Line :	9
Sample Name	: SOLV BLK MS 2X	Location :	Vial 9
Acq. Operator	: GAA	Inj :	1
		Inj Volume :	50 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_FS.M		
Last changed	: 10/11/2002 5:12:48 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX1016.M		
Last changed	: 10/17/2002 10:54:32 AM by GAA		

010035



010036

Injection Date : 10/16/2002 7:48:29 PM
Sample Name : SOLV BLK MS 2X
Acq. Operator : GAA

Seq. Line : 9
Location : Vial 9
Inj : 1
Inj Volume : 50 µl

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:54:32 AM by GAA

External Standard Report

Sorted By : Signal
Calib. Data Modified : Thursday, October 17, 2002 10:52:50 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime [min]	Type	Area	Amt/Area	Amount [ug/mL]	Grp	Name
7.083	BV	9.43532e5	5.57006e-7	5.25553e-1		VX

Totals : 5.25553e-1

*** End of Report ***

SOUTHWEST RESEARCH INSTITUTE

010037

VX

Results Data Reporting Form

Client: JACADS
Case: JACADS
SDG: 213854
Matrix: Solid
Filename: 10160212
Injection Vol (uL): 50
Dilution Factor: 2
Sample Wt (g): 2.017

System ID: 213854 MS
Client Sample ID: Tray 153 (021002-001)
Project Number: 01.05935.03.001
Instrument: LCMS1
Date Received: 10/07/02
Date Extracted: 10/16/02
Date Analyzed: 10/16/02
Final Volume (mL) : 4

CAS No.	Compound	ug/g
50782-69-9	VX	12.101

Data Reporting Qualifiers

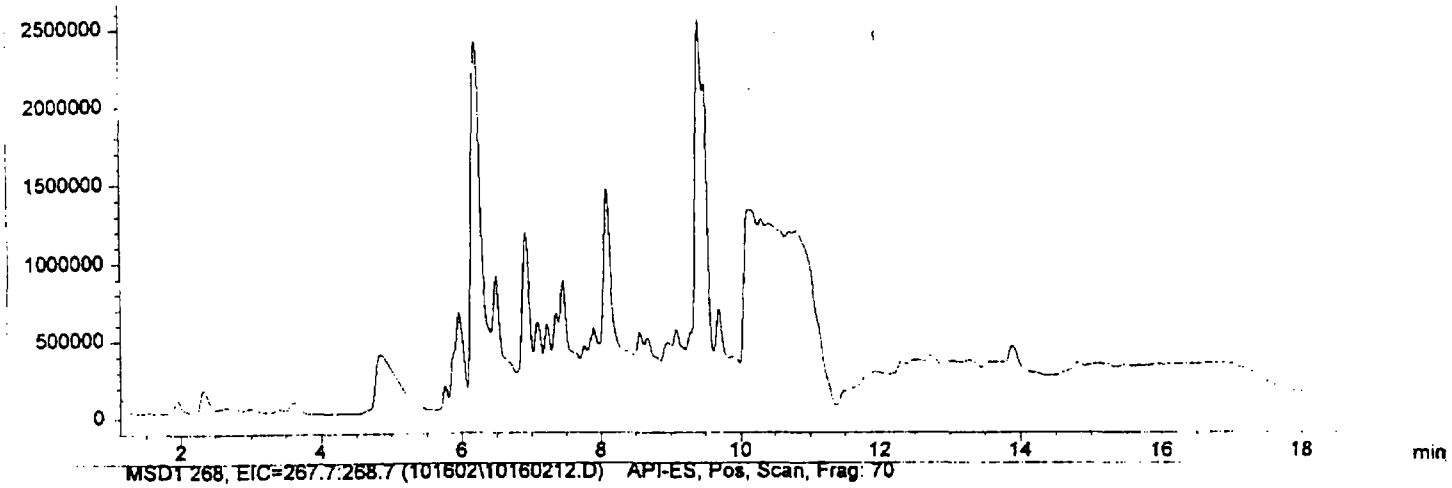
B This flag is used when the analyte is found in the blank as well as in the sample.
E This flag indicates compounds whose concentrations exceed the calibration range.
J Indicates an estimated value.
U Indicates compound was analyzed for, but not detected. Report the minimum detection limit for the sample with U (e.g. 10U) based on necessary concentration dilution action.

Data File C:\HPCHEM\1\DATA\101602\10160212.D Sample Name: 213854 MS 2X

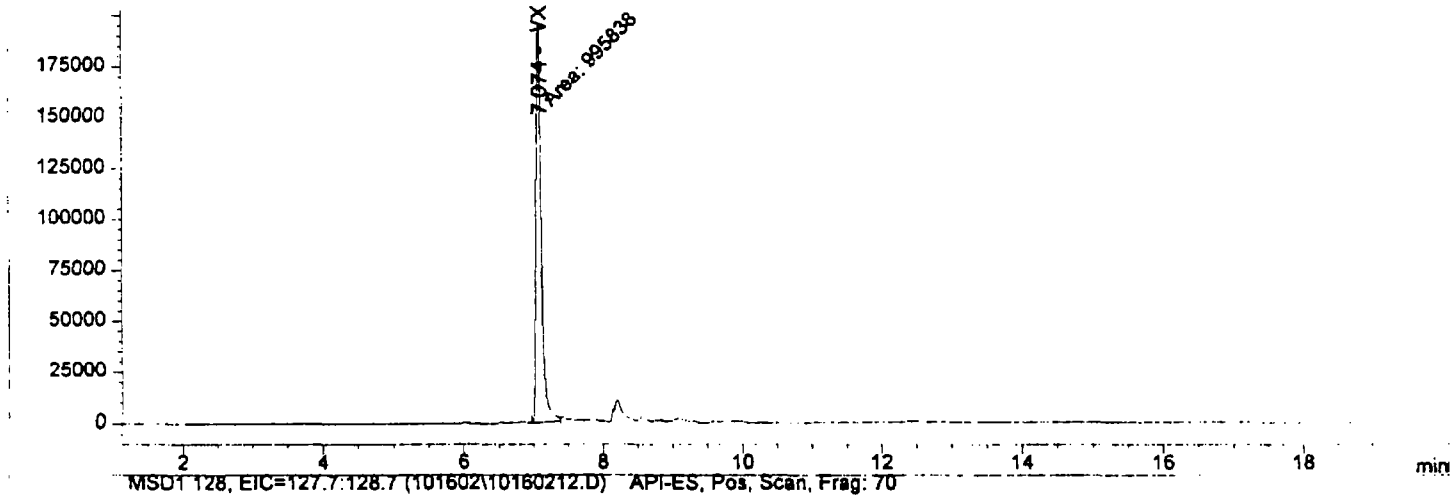
Injection Date	: 10/16/2002 8:32:19 PM	Seq. Line	: 11
Sample Name	: 213854 MS 2X	Location	: Vial 11
Acq. Operator	: GAA	Inj	: 1
		Inj Volume	: 50 µl
Acq. Method	: C:\HPCHEM\1\METHODS\VX_FS.M		
Last changed	: 10/11/2002 5:12:48 PM by GAA		
Analysis Method	: C:\HPCHEM\1\METHODS\VX1016.M		
Last changed	: 10/17/2002 10:54:32 AM by GAA		

010038

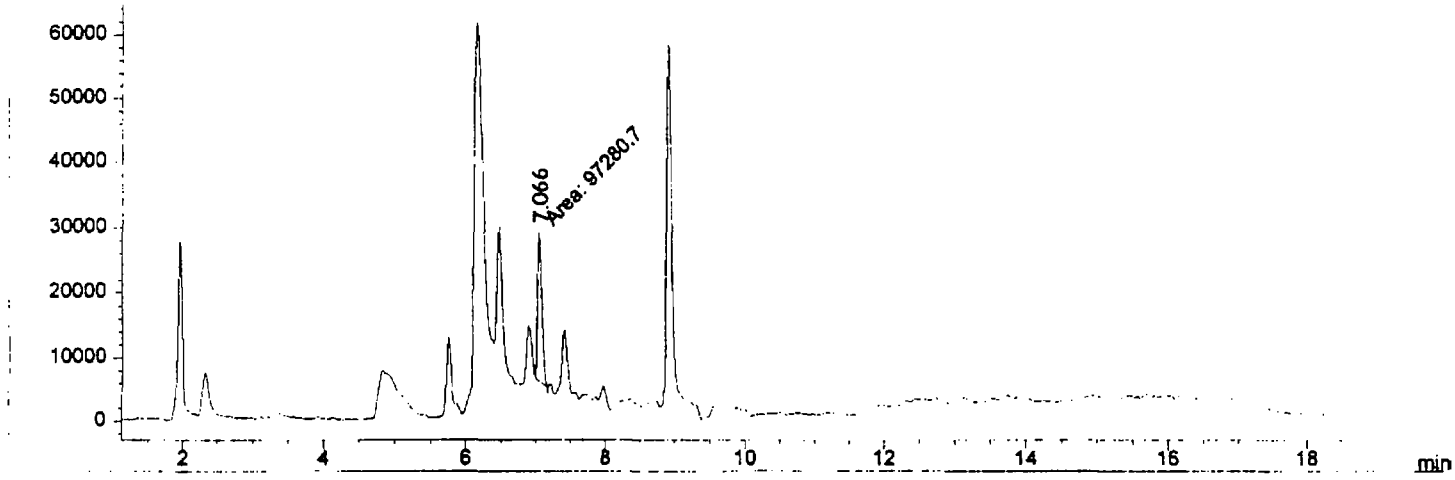
MSD1 TIC, MS File (101602\10160212.D) API-ES, Pos, Scan, Frag: 70



MSD1 268, EIC=267.7:268.7 (101602\10160212.D) API-ES, Pos, Scan, Frag: 70



MSD1 128, EIC=127.7:128.7 (101602\10160212.D) API-ES, Pos, Scan, Frag: 70



Injection Date : 10/16/2002 8:32:19 PM
Sample Name : 213854 MS 2X
Acq. Operator : GAA

Seq. Line : 11
Location : Vial 11
Inj : 1
Inj Volume : 50 µl

010039

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:54:32 AM by GAA

External Standard Report

Sorted By : Signal
Calib. Data Modified : Thursday, October 17, 2002 10:52:50 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 2: MSD1 268, EIC=267.7:268.7

RetTime [min]	Type	Area	Amt/Area	Amount [ug/mL]	Grp	Name
7.074	MM	9.95838e5	5.57022e-7	5.54704e-1		VX

Totals : 5.54704e-1

*** End of Report ***

SOUTHWEST RESEARCH INSTITUTE

VX

Results Data Reporting Form

Client: JACADS
Case: JACADS
SDG: 213854
Matrix: Solid
Filename: 10160214
Injection Vol (uL): 50
Dilution Factor: 2
Sample Wt (g): 2.012

System ID: 213854 MSD
Client Sample ID: Tray 153 (021002-001)
Project Number: 01.05935.03.001
Instrument: LCMS1
Date Received: 10/07/02
Date Extracted: 10/16/02
Date Analyzed: 10/16/02
Final Volume (mL) : 4

CAS No.	Compound	ug/g
50782-69-9	VX	12.804

Data Reporting Qualifiers

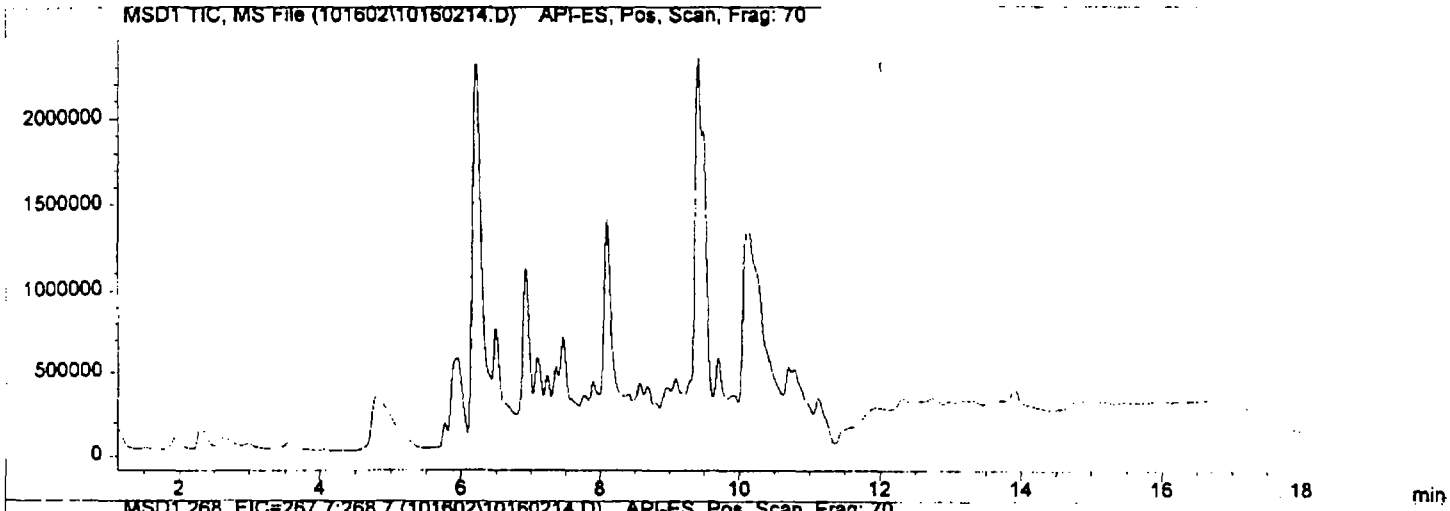
B This flag is used when the analyte is found in the blank as well as in the sample.
E This flag indicates compounds whose concentrations exceed the calibration range.
J Indicates an estimated value.
U Indicates compound was analyzed for, but not detected. Report the minimum detection limit for the sample with U (e.g. 10U) based on necessary concentration dilution action.

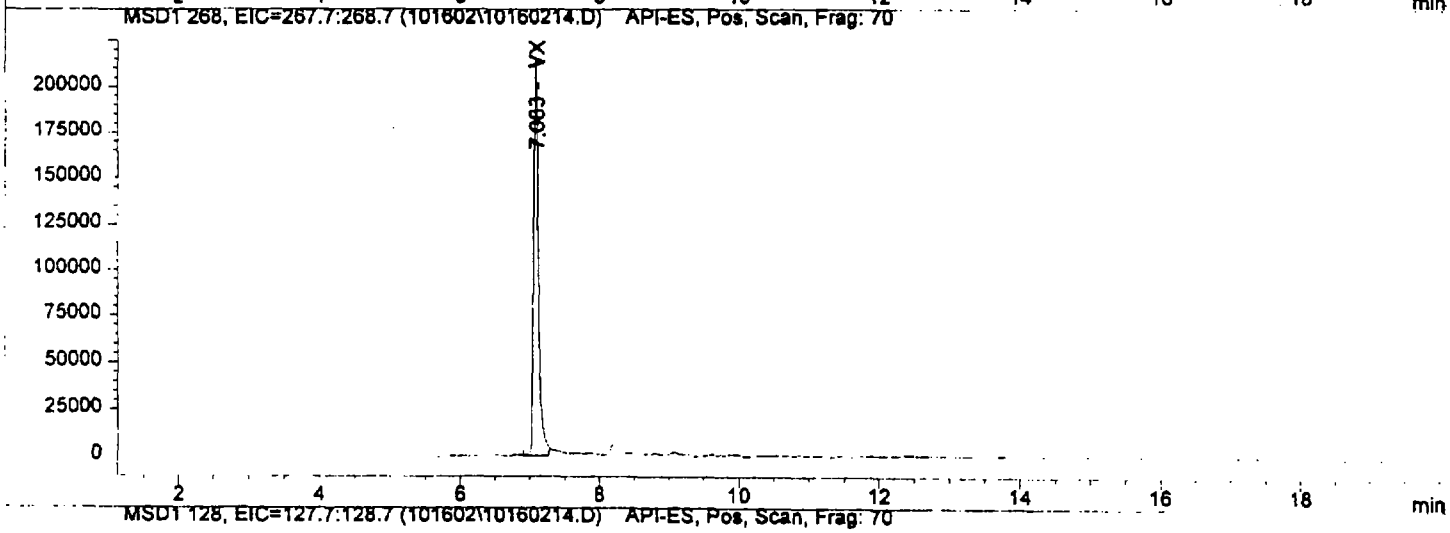
Data File C:\HPCHEM\1\DATA\101602\10160214.D Sample Name: 213854 MSD 2X

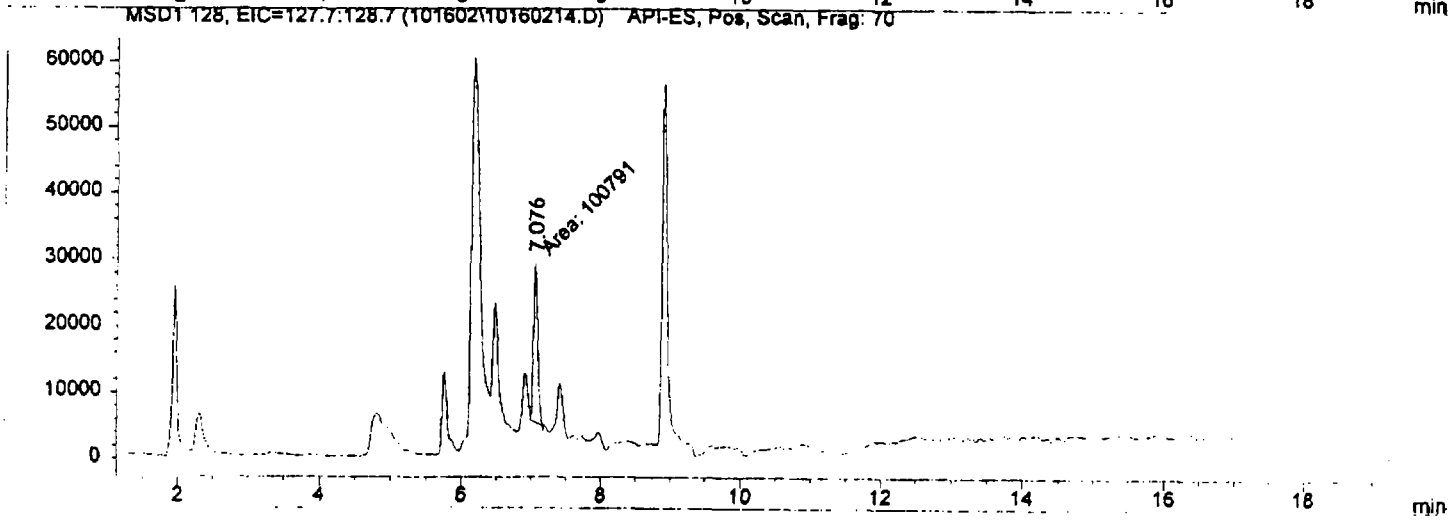
Injection Date	: 10/16/2002 9:16:06 PM	Seq. Line	: 13
Sample Name	: 213854 MSD 2X	Location	: Vial 13
Acq. Operator	: GAA	Inj	: 1
		Inj Volume	: 50 µl

010041

Acq. Method : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed : 10/17/2002 10:54:32 AM by GAA







010042

```
Injection Date  : 10/16/2002 9:16:06 PM          Seq. Line :  13
Sample Name     : 213854 MSD 2X                   Location : Vial 13
Acq. Operator   : GAA                                 Inj :   1
                                                Inj Volume : 50 µl
Acq. Method     : C:\HPCHEM\1\METHODS\VX_FS.M
Last changed    : 10/11/2002 5:12:48 PM by GAA
Analysis Method : C:\HPCHEM\1\METHODS\VX1016.M
Last changed    : 10/17/2002 10:54:32 AM by GAA
```

External Standard Report

```
Sorted By              :      Signal
Calib. Data Modified   :      Thursday, October 17, 2002 10:52:50 AM
Multiplier             :      1.0000
Dilution               :      1.0000

Signal 2: MSD1 268, EIC=267.7:268.7
```

RetTime [min]	Type	Area	Amt/Area	Amount [ug/mL]	Grp	Name
7.083	VV	1.05105e6	5.57038e-7	5.85475e-1		VX

Totals : 5.85475e-1

*** End of Report ***

APPENDIX D

IITRI DATA PACKAGE FOR GC/IT/MS

Classification

C 31763

Unclassified

This is Book No. — of this Project

IIT RESEARCH INSTITUTE
CHICAGO, ILLINOIS 60616

10-24-02 Changed to misc 246-0-2008

Project No. Misc. Venture No. 001-001 Contract No.

Sponsor Washington Dismil

Subject Analyses of Rock Samples

Section Supervisor Harsh Dev Project Leader

Logbook Issued to Yirgalem Abebe Date Issued 10-7-02

Microfilm Record

Page	to	Page	Date	Roll No.

Unclassified

THIS LOGBOOK IS THE PROPERTY OF IIT RESEARCH INSTITUTE

C-o-C #681

CHAIN OF CUSTODY RECORD

United Engineers & Constructors
A Raytheon Company
Western Operations

PROJ. NO.	PROJ. NAME: JACADS - PMCSD / WDC					NO. OF SAMPLE CONTAINERS	SAMPLE TAG NUMBERS							REMARKS
SAMPLERS: (SIGNATURE) consolidated by ECP														
SAMPLE STATION NO.	2002 DATE	TIME	DESIGNATE COMP	DESIGNATE GRAB	STATION DESCRIPTION									
MAY 153	02OCT	AM		X	SDS Sludge (Solid)	1	02H002-002							sent per SwRI's request

1. RELINQUISHED BY: (SIGNATURE)	DATE/TIME	RECEIVED BY: (SIGNATURE & COMPANY)	2. RELINQUISHED BY: (SIGNATURE)	DATE/TIME	RECEIVED BY: (SIGNATURE & COMPANY)
[signature]	2002 03OCT 0730	[signature] RTSC	MILES OLA	Oct 3 02 12:00	10/7/02 [signature] IITRI
3. RELINQUISHED BY: (SIGNATURE)	**DATE/TIME**	**RECEIVED BY: (SIGNATURE & COMPANY)**	**4. RELINQUISHED BY: (SIGNATURE)**	**DATE/TIME**	**RECEIVED BY: (SIGNATURE & COMPANY)**
[signature]	2002 07OCT 0850	[signature] IITRI 10-7-02			
5. RELINQUISHED BY: (SIGNATURE)	**DATE/TIME**	**6. RECEIVED FOR LABORATORY BY: (SIGNATURE & COMPANY)**	**REMARKS:**		**DATE/TIME**

DISTRIBUTION: 1 - WHITE 2 - BLUE 3 - GREEN 4 - YELLOW 5 - PINK 6 - GOLDENROD

A10891951

CHEMICAL AGENT CUSTODIAL STOCK RECORD

Container No: _____ Project No. _____ Agent Type Sludge IITRI Lot No: Y. Abdhu

Room No: CAF Hood No: _____ Container Type, No. Ea: _____

Date	Document Number (logbook & page)/Project No./Purpose	Vial No.	Receipt	Issue	Balance	Signature
10-7-02	C-o-c #681, JACADS-PMCSD/WDC		50g		50g	Y. Abdhu
	SDS Sludge (Solid), For VX Analysis					
10-15-02	Logbook C31763, p.4, 246-0-2008-001-001		50g	10g	40g	Y. Abdhu
10-17-02	Logbook C31763, p- , 246-0-2008-001-001		40g	4g	36g	Y. Abdhu

FORM 2

PROJECT LOGBOOK

C 31763 1

Book Page

Project Sample reciept Date 10-7-02

Signature Y. Abrha

* (Ash sample ~~from~~ from Raytheon Demil Co was 2850 PAA St Ste 100 P.O. Box 17843, Honolulu HI 96817 ~~SWRI~~ YA (Re) 10-7-02 was recieved on 10-7-02

50 g & stored on the XDS storage Refrigerator in Sorting Lab.

Tag # or Label of the Can LAWIC 153 B

"Environmentally Hazardous substance, solid sample was designated on the side of the container by the sender."

Note :-

The sample is from JACADS-PMCSD/WDC

SDS Sludge (solid) 150 g

ID # C-O-C # 681

* Ultrasonic Extractor of Model CPX # 500

Program - Time - 3 min

Temp - 22 °C

Pulse - 0.5 sec on 0.5 sec off

Amplitude = 70 %

Read and Understood by me

Signature _____ Date _____

Signature _____ Date _____

PROJECT LOGBOOK C 31763 2
Book Page

Subject Ultrasonic Extraction Date 10-14-02

Signature Y. Abrha

A mixture of solvent $MeCl_2$ 90% & IPA 10% made in 2000mL vol flask by adding 200mL of IPA then dilute to vol with $MeCl_2$ to the mark.

IPA used Fishers, optima lot # 011554

$MeCl_2$ used Fishers GC Resolve lot # 011758

Triethylamine used, Fluka Chemika 90340

SOP-C-Safety-204-00 was followed with some deviations

1. Amount of sample used at the beginning 5 gm instead of 25g
2. Two time sonication using 20mL each instead of 3 times with 100mL & make basic by adding triethylamine (pH 4)
3. Concentrate by blowing with nitrogen
4. Analyze with GC/ITMS

Calibration of GC/ITMS was done/run Std's Range 0.046 to 1.83 µg/mL VX std.

Two blank or Glassware blank & Two Glassware blank spike was done.

Balance # 89 logbook C 31643 P-14

Na_2SO_4 anhydrous from disicator was used of

Sample 1 wt of the beaker = 27.4089 g

wt of beaker + Na_2SO_4 = 32.4157 g

5.0065 g

Sample 2. wt of the beaker = 30.1740

wt of beaker + Na_2SO_4 = 35.1932

5.0192 g

Sample 3. wt of the beaker

wt of the beaker + Na_2SO_4

(RE) 10-14-02 YA

Read and Understood by me

Signature [signature] Date 10/22/02

Signature Date

Y-Anthra
C 31763
Back of P-2
10-14-02

1 mL each of std prepared on 10-03-02
Logbook C 31742 p-5 was taken on auto sampler vials &
made to run into GC/ITMS.
XVX-100302-1 upto XVX-100302-6
1.83 µg/mL " 0.046 µg/mL

Print Date: 15 Oct 2002 10:51:28

Calibration Block Report

Run File:	c:\saturnws\data\2002\october\101402#1\xvx100302-1, 1.8ug10-14-02001.sms		
Instrument Method:	c:\... vs092502-mz268.2-0.42v-pil	Acquisition Date	10/14/02 8:27 PM
Recalc Method:	c:\... vs-calib-dsael-10-14-02.mi	Last Calibration.	10/15/02 10:16 AM
Sample List:		Compound Table Updated.	10/15/02 10:37 AM
Sequence:		Detector:	2000 Mass Spec
tion Name:	Saturn GC/MS Workstation	Workstation Version:	Version 5.52
easurement.	Area	Calibration Type.	External Standard Analysis

Retention Time (min)	Peak Name	Cal. Method	X3	X2	X	C	r2	Cal. Range	No. of Points	Fit Codes
28.613	VX	2 IG None		96236.5	213859.3	-1769.64	0.999124	1-6	6	

Curve Fit Codes	Origin Point Codes	Regression Weighting Codes		Fit Codes
1 linear	IN include	None	1/nX	1 curve
2 quadratic	IG ignore	1/n	1/X2	2 origin
3 cubic	FO force	1/X	1/nX2	3 coefficient

Ret. Time: 28.613 min. Peak Name: VX

Level	Amount	Replicate No.	Response	Avg. Response	Std. Dev.	% RSD	RF	Avg. RF
1	1.830000	1	712854	712854.1	#		389537.8	
2	0.913000	1	268616	268615.9	#		294216.6	
3	0.457000	1	118646	118646.4	#		259628.3	
4	0.182000	1	34097	34097.8	#		199987.8	
5	0.091000	1	10577	10576.5	#		116335.4	
6	0.046000	1	3476	3476.6	#		75578.7	239194.5

= Too few points to calculate.

Peak Name	Level	Rep.	Injection Date/Time	Run Files
VX	1	1	10/14/02 9:27 PM	c:\... \october\101402#1\xvx100302-1, 1.8ug10-14-02001.sms
	2	1	10/14/02 10:11 PM	c:\... \2002\october\101402#1\xvx100302-2,0.91ug10-14-02.sms
	3	1	10/14/02 10:55 PM	c:\... \2002\october\101402#1\xvx100302-3,0.46ug10-14-02.sms
	4	1	10/14/02 11:39 PM	c:\... \2002\october\101402#1\xvx100302-4,0.18ug10-14-02.sms
	5	1	10/15/02 12:23 AM	c:\... \2002\october\101402#1\xvx100302-5,.091ug10-15-02.sms
	6	1	10/15/02 1:06 AM	c:\... \2002\october\101402#1\xvx100302-6,.046ug10-15-02.sms

Project Ultrasonic Extraction Date 10-14-02
Continued Signature Y. Abotha

Sample 3 wt of the beaker - 27.6245 g
wt of the beaker + Na_2SO_4 32.6858 g
5.0613 g

Sample 4 wt of the beaker = 29.6906 g
wt of the beaker + Na_2SO_4 = 34.7277 g
5.0371 g

The ultrasonic extracts was concentrated by blowing to 10 mL then 1 mL of the sample was added with 80 µL of DEAET and analysed by GC/ITMS.

DEAET Std. was prepared as follows.
Balance # 307 Cal check C 31651 p-11
DEAET of 4-11-02 made in House logbook C31703 p-8

wt of 25 mL Vol flask = 23.4383
wt of 25 mL Vol flask + DEAET = 23.9884
wt of DEAET = 0.5501 g

0.5501 g of DEAET stock in a 25 ml flask was diluted to Vol with hexane & labeled as DEAET 10-14-02
final concentration 22.0 mg/mL

Sample ID of extract

XVX-101402-US1
XVX-101402-US2
XVX-101402-US3 } Spike with 25 µL of 91.3 µg/mL VX on the solid anhydrous Na_2SO_4.
XVX-101402-US4 }

Read and Understood by me

Signature [signature] Date 10/22/02
Signature Date

Y. Abthen
C31763
back of P-3
10-14-02

Varian Star Workstation - 3400 SampleList Tue Oct 15 13:36:37 2002

SampleList: C:\saturnws\Data\2002\October\10101301\VZ101401.smp

Created: Mon Oct 14 17:01:54 2002
Modified Tue Oct 15 08:11:49 2002

Line	Sample Name	Injection Mode	AutoLink	Vial	Inject Volume
1			C:\saturnws\Methods\VZ-2303\VZ2302S3-Aq3A6.3-8.63V-PIT-200-BT-0.65-MOFF-Purge323-25,Multi"S3V-133V 0:1		
2	VZ3-013303-6,spk6-	Below	none	1	1.0
	Notes: VZ3-11-07-31A,spike, 5.0ug/mL VZ, MAE extract:Bin-SM2,30ml 31mm/0.1um,				
	Notes: 100C,5min-240C@6C/m-315C@25C/m,5min,0.43VZE,PIT2300msec/0.65cscns/sec,MOFF;m/s2A3 3.1750V,S-TRAP5170				
3	VZ-3-013303-5,spk6-	Below	none	1	1.0
	Notes: VZ3-11-07-31A,spike, 5.0ug/mL VZ, MAE extract:Bin-SM2,30ml 31mm/0.1um,				
	Notes: 100C,5min-240C@6C/m-315C@25C/m,5min,0.43VZE,PIT2300msec/0.65cscns/sec,MOFF,m/s2A3 3.1750V,S-TRAP5170				
4	VZ-100322-1, 1.0ug	Below	none	3	1.0
	Notes: 1.0ug/mLVZ Std. Bin-SM2 30M x 0.35mm ID, 1.0um, 100C,5min - 240C 6C/m - 300C				
	Notes: 025C/m,5min,0.42VZE,PIT2300msec,0.65cscns/sec,MOFF,m/s2A3.3,1750V,S-TRAP5170C,Sngl-CH				
5	VZ-100322-1, 1.0ug	Below	none	3	1.0
	Notes: 1.0ug/mLVZ Std. Bin-SM2.30M x 0.35mm ID, 1.0um, 100C,5min - 240C 6C/m - 300C				
	Notes: 025C/m 5min,0.42VZE,PIT2300msec;0.65cscns/sec,MOFF,m/s2A3.3,1750V,S-TRAP5170C,Sngl-CH				
6	VZ100303-3.0.91ug	Below	none	3	1.0
	Notes: 0.91ug/mLVZ Std.Bin-SM2.30M x 0.35mm ID, 1.0um, 100C,5min - 240C 6C/m - 300C				
	Notes: 025C/m,5min,0.42V-ZE,PIT2300msec;0.65cscns/sec,MOFF,m/s2A3.3;1750V,TS-TRAP5170C;Sngl-CH				
7	VZ100303-3.0.45ug	Below	none	4	1.0
	Notes: 0.457ug/mLVZ Std.Bin-SM2.30M x 0.35mm ID, 1.0um, 100C,5min - 240C 6C/m - 300C				
	Notes: 025C/m,5min,0.42VZE,PIT2300msec;0.65cscns/sec,MOFF,m/s2A3.3,1750V,S-TRAP5170C,Sngl-CH				
8	VZ100303-4.0 1ug	Below	none	5	1.0
	Notes: 0.101ug/mLVZ Std.Bin-SM2.30M x 0.35mm ID, 1.0um, 100C,5min - 240C 6C/m - 300C				
	Notes: 025C/m,5min,0.42VZE,PIT2300msec;0.65cscns/sec,MOFF,m/s2A3.3,1750V,S-TRAP5170C;Sngl-CH				
9	VZ100303-5..051ug	Below	none	6	1.0
	Notes: 0.051ug/mLVZ Std Bin-SM2 30M 0.35mm ID, 1.0um; 100C,5min - 240C 6C/m - 300C				
	Notes: 025C/m,5min,0.42VZE,PIT2300msec;0.65cscns/sec;MOFF;m/s2A3.3;1750V,S-TRAP5170C;Sngl-CH				
10	VZ100303-6..046ug	Below	none	7	1.0
	Notes: 0.046ug/mLVZ Std.Bin-SM2.30M 0.35mm ID, 1.0um, 100C,5min - 240C 6C/m - 300C				
	Notes: 025C/m,5min,0.42VZE,PIT2300msec;0.65cscns/sec;MOFF;m/s2A3.3,1750V,S-TRAP5170C;Sngl-CH				
11	VZ100303-3,0.46ug	Below	none	8	1.0
	Notes: 0.457ug/mLVZ Std.Bin-SM2.30M x 0.35mm ID, 1.0um, 100C,5min - 240C 6C/m - 300C				
	Notes: 025C/m 5min;0.42VZE,PIT2300msec;0.65cscns/sec,MOFF/m/s2A3 3;1750V,S-TRAP5170C,Sngl-CH				
12	VZ-131493-MB-1	Below	none	9	1.0
	Notes: Methylene Chloride/IPA Sonicator Blank - BEAST,Bin3MS;S-TRAP;SM017519				
13	VZ-101401-MB-2	Below	none	9	1.0
	Notes: Methylene Chloride/IPA Sonicator Blank - BEAST,Bin3MS;S-TRAP;SM017519				
14	VZ-101402-VZ-2	Below	none	10	1.0
	Notes: Method Spike,0.57ug/mL VZ Spike into by NaSO4 - BEAST, Bin3MS,S-TRAP;SM017519V				
15	VZ-101402-MB-4	Below	none	11	1.0
	Notes: Method Spike;0.57ug/mL VZ Spike into Sg NaSO4 - BEAST, Bin3MS;S-TRAP,SM017519V				
16	VZ100303-3,0.45ug	Below	none	6	1.0
	Notes: 0.457ug/mLVZ Std.Bin-SM2.30M 0.35mm ID, 1.0um, 100C,5min - 240C 6C/m - 300C				
	Notes: 025C/m,5min;0.42VZE,PIT2300msec;0.65cscns/sec,MOFF,m/s2A3.3,1750V,S-TRAP5170C,Sngl-CH				
17			C:\saturnws\methods\Standby.mth		

Directory for Data Files: c:\saturnws\data\2002\oct2002\10101301

Data File Names: Auto

Create and update a new SampleList: c:\saturnws\data\2002\october\10101301\vz101401.rcl

Subject Ultrasonic Extraction on Ash samples. Date 10-15-02
Signature Y. Abotha

Balance # 307 Cal ck C31651 P-1

5 g of Na_2SO_4 & 5 g of Ash sample mixed together

wt of Ash of JACADS lot # LAWIC 153 B

Sample 1A wt of the beaker & conical centrifuge tube = 45.0534 g
wt of Beaker & C. tube + Ash = 50.1195 g
wt of Ash = 5.0661 g

Sample 2A wt of the beaker & conical C. tube = 44.9740 g
wt of the beaker & C. tube + Ash = 50.0563 g
wt of Ash = 5.0823 g

Weight of Na_2SO_4 Anhydrous,
Balance # Cal ck C31643 P-14

For Sample 1 wt of the beaker = 27.4149 g
wt of the Beaker + Na_2SO_4 = 32.5370 g
wt of Na_2SO_4 = 5.1221 g

Sample 2 wt of the beaker = 30.1767 g
wt of the Beaker + Na_2SO_4 = 35.2142 g
wt of Na_2SO_4 = 5.0375 g

Sample 3 wt of the beaker = 27.4182 g
wt of the Beaker + Na_2SO_4 = 32.4416 g
wt of Na_2SO_4 = 5.0234 g

Sample 4 wt of the beaker = 29.6927 g
wt of the Beaker + Na_2SO_4 = 34.7790 g
wt of Na_2SO_4 = 5.0863 g

Read and Understood by me
Signature Dr Miller Date 10/22/02
Signature Date

Subject Date 10-15-02

Contin. Signature Y. Abrha

Sample 1 & 2 of Anhydrous Na_2SO_4 was spiked with 50 µL of 9.13 µg/mL VX (XVX-100302-C) (i.e. Glassware blank spike) one by one & left to dry for 5 min then sonicated the same was as the procedure in page 2 of this logbook

- The difference from the previous one was the sample was ~~belowed~~ to 2 mL & 100 µL of it was taken for analysis. The 100 µL of the sample was added 4 µL of DEAET 10-14-02. Second difference the amount of spike level is low in this case than the previous case.

Sample ID # given

see logbook C31742 P-4

XVX-101502-US-1 } spike with 50 µL of 9.13 µg/mL VX
XVX-101502-US-2 } (XVX-100302-C)

Ash samples Sample 1A mixed with Sample 3 Na_2SO_4 &
Sample 2A mixed with Sample 4 Na_2SO_4

The mixture of Ash & Na_2SO_4 was sonicated the same way as the blank samples but after decoming the solution the sample was centrifuged further to seperate the solid from the liquid soln.

The liquid soln was concentrated to 5 mL using or by blowing Nitrogen stream to 40 mL solution

Read and Understood by me

Signature [signature] Date 10/22/02

Signature Date

Y. Abshire
C31763
back of P-5
10-15-02

****

Varian Star Workstation - Sample Sequence Tue Oct 15 15:13:32 2002

Sequence: d:\saturnws\data\2002\October\10151401\VS10151201.seq

Created Tue Oct 15 14:35:26 2002
Modified Tue Oct 15 15:12:47 2002

****

Line	Sample Name	Injection Method	Solution	Vial	Inject Volume
1			C:\saturnws\Methods\VS-2002\VS2002223-mr366.S-S 63V-P27-230-ST-3 61-ASPP-Purge313-E1;Multi7929v-2310v.mth		
2	XVS-112002-1.spAS-	Below	none	1	1.0
	Notes: XVS-11-27-01A.spine, 0.60mg/mL VS BAS extract:Bis-SMB.30MAS.35mm:0.1ml. Notes: 100C,5ml x-Prep30C/m-250C0250C/m 5min,0.42VMS.P17:30Prime:0 60arcme/sec.mPP;m/v265.2,17025V,0-TRAP0:70				
3	XVS112002-1.0.4mg	Below	none	1	1.0
	Notes: 0 60mg/mLVS Std.Bis-SMB 30ma 0 21mm ID, 0.0um, 100C,5min - 240C 30C/m - 320C Notes: 0250C/m,5min,0 42VMS.P17:30Prime:0.60arcme/sec.mPP;m/v366 3.17025V-0-TRAP0:70C.Exp1-GV				
4			C:\saturnws\Methods\VS-2002\VS2002223-mr366 3-S.63V-P27-230-ST-3 61-ASPP-3.Purge313-E1.Multi7929v-2310v-V2Sim] mth		
5	Vs-101522-US-1-	Below	none	1	1.0
	Notes: Method Spike. 5ing/g VS Spike into 1g MeOHs - BEAST. 0ca4ME,S-TRAP,E=:1750V				
6	Vs-101522-US-2-	Below	none	1	1.0
	Notes: Method Spike. 5ing/g VS Spike into 1g MeOHs - BEAST. 0ca4ME,S-TRAP,E=:1750V				
7	Vs-101522-US-3-	Below	none	1	1.0
	Notes: JACADS Ash Sample No. LAW101520. 1g - BEAST. Ultrasonic Extraction. no EAE clean-up. Notes: 0ca4ME,S-TRAP,E=:1750V				
8	Vs-101522-US-4-	Below	none	1	1.0
	Notes: JACADS Ash Sample No LAW101520. 1g - BEAST. Ultrasonic Extraction. no EAE clean-up. Notes: 0ca4ME,S-TRAP,E=:750V				
9	XVs100202-2.0 4mg	Below	none	1	1.0
	Notes: 0 60mg/mLVS Std.Bis-SMB.30ma 0.21mm ID, 0.0um, 100C,5min - 240C 30C/m - 320C Notes: 0250C/m,5min,0 42:VS.P17:30Prime:0.60arcme/sec.mPP;m/v366.2/1750V;0-TRAP0170C;Exp1-GV				
10			C:\saturnws\Methods\Standby.mth		

Directory for Data Files: d:\saturnws\data\2002\October\10151201

Data File Name: %%d

Create and update a new Sequence: d:\saturnws\data\2002\October\10151201\vs101501-1.seq.

Subject Ultrasonication Date 10-15-02
Cont. Signature Y-Abrha

The concentrated sample (5mL) was filtered by using a syringe attached to a filter (Gelman Laboratory GHP Acrodisc® 25mm Syringe Filter with 0.45 µm GHP Membrane Lot # A421012412)
Then 100 µL of the sample was withdrawn & 4 µL of DEAET added to it for analysis by GC/ITMS.

Sample ID #

XVX-101502-US-3
XVX-101502-US-4

10-16-02 YA
Since the area count is high or beyond our calibration curve the above sample was diluted by taking 10 ml (Re) YA 1 mL & dilute in 10 mL Vol. flask with $MeCl_2$.

Sample ID #
XVX-101602-US-3D (
XVX-101602-US-4D

1 mL of each sample was taken in autosampler & 40 µL of DEAET added to it then analysed by GC/ITMS

Calibration run made
XVX-100302-1 - 1.81 µg/mL (1 mL)
XVX-100302-2 - 0.91 µg/mL (1 mL)
XVX-100302-3 - 0.46 µg/mL (1 mL)
XVX-100302-4 - 0.18 µg/mL (1 mL)
XVX-100302-5 - 0.041 µg/mL (1 mL)
XVX-100302-6 - 0.046 µg/mL (1 mL)

The result on the ~~ba~~ back of the next page

Read and Understood by me
Signature [signature] Date 10/22/02
Signature Date

Y. Abthen
C31763
back 7P-6
10-15-02

Varian Star Workstation - 2000 SampleList Wed Oct 16 10:05:33 2001

SampleList: C:\saturnws\data\2002\october\101602\VS101602.smp

Created: Wed Oct 16 10:06:33 2002
Modified: Wed Oct 16 10:08:22 2002

Line	Sample Name	Injection Number	Method	AutoLink	Vial	Inject Volume
1				C:\saturnws\methods\VS-2002\VS101602-02 PubTOxan,Multi75ug-100ug-VBInj.mth		
2	VS-[illegible]	below	none	1		1.0
			Notes: JACADS Ash Sample No. LA91C1530. 5g + DEAET. Final Volume = 5mL. Ultrasonic Extraction. no NAS			
			Notes: cleanup. Ds:ZMS;S-TRAP ZM:17SEV = ZEST. EI Full Scan m/z 50-450amu			
3				C:\saturnws\methods\VS-2002\VS101602-02 PubTOxan,Multi75ug-100ug-VBInj.mth		
4	VS-101202-VS-4-	below	none	2		1.0
			Notes: JACADS Ash Sample No. LA91C1530. 5g + DEAET. Ultrasonic Extraction. no NAS cleanup			
			Notes: Ds:ZMS;S-TRAP,ZM:17SEV			
5				C:\saturnws\methods\VS-2002\VS101602-02 [illegible]		
6	EVS\100202-3.0 ug	below	none	3		1.0
			Notes: 0.457ug/mLVS Std.Ds:-SMS 30% 0.25mm ID, 1.0um; 180C,5min - 240C 05C/m - 280C			
			Notes: 035C/m,5min,0.42VES,PIT:2200nsec,0.65scans/sec,mnPP;m/z360.3,1750V,S-TRAP:190C,Sngl-SM			
7	EVS\100202-3.0 ug	below	none	5		1.0
			Notes: 0.457ug/mLVS Std,JACADS Ash Sample No. LA91C1530. 5g + DEAET. Ultrasonic Extraction. no NAS			
			Notes: Cleanup. Initial Volume = 5mL. diluted 1:10 w/CH2Cl2. Ds:ZMS.S-TRAP,ZM:17SEV			
8	VS-101202-VS-2-	below	none	4		1.0
			Notes: JACADS Ash Sample No. LA91C1530. 5g + DEAET. Ultrasonic Extraction. no NAS cleanup. Initial Volume			
			Notes: = 5mL. diluted 1:20 w/CH2Cl2. Ds:ZMS.S-TRAP.ZM:17SEV			
9	MeCl2 Blank	below	none	5		1.0
			Notes: Methylene Chloride Blank			
10	VS-101202-VS-4B-	below	none	6		1.0
			Notes: JACADS Ash Sample No. LA91C1530. 5g + DEAET. Ultrasonic Extraction. no NAS cleanup. Initial Volume			
			Notes: = 5mL. diluted 1:20 w/CH2Cl2. Ds:ZMS S-TRAP,ZM:17SEV			
11	EVS\100202-3.0 ug	below	none	7		1.0
			Notes: 0.457ug/mLVS Std.Ds:-SMS 30% 0.25mm ID, 1 Dum. 180C,5min - 240C 05C/m - 280C			
			Notes: 035C/m,5min,0.42VES,PIT:2200nsec,0.65scans/sec;mnPP;m/z360.3,1750V,S-TRAP:190C,Sngl-SM			
12	EVS\100202-1. 0 ug	below	none	8		1.0
			Notes: 0.152ug/mLVS Std.Ds:-SMS 30% 0.25mm ID, 1.0um. 180C,5min - 240C 05C/m - 280C			
			Notes: 035C/m,5min,0.42VES,PIT:2200nsec,0.65scans/sec;mnPP,m/z360.3,1750V,S-TRAP:190C,Sngl-SM			
13	EVS\100202-1. 0 ug	below	none	9		1.0
			Notes: 1.52ug/mLVS Std. Ds:-SMS 30% 0.25mm ID, 1.0um. 180C,5min - 240C 05C/m - 280C			
			Notes: 035C/m,5min,0.42VES,PIT:2200nsec,0.65scans/sec;mnPP,m/z360.3,1750V,S-TRAP:190C,Sngl-SM			
14	EVS\100202-1.5.0ug	below	none	7		1.0
			Notes: 4.913ug/mLVS Std.Ds:-SMS 30% 0.25mm ID, 1.0um; 180C,5min - 240C 05C/m - 280C			
			Notes: 035C/m,5min,0.42VES,PIT:2200nsec,0.65scans/sec;mnPP;m/z360.3,1750V,TS-TRAP:190C,Sngl-SM			
15	EVS\100202-1.0.05ug	below	none	8		1.0
			Notes: 0.037ug/mLVS Std.Ds:-SMS.30% 0.25mm ID, 1.0um; 180C,5min - 240C 05C/m - 280C			
			Notes: 035C/m,5min,0.42VES,PIT:2200nsec;1 65scans/sec,mnPP,m/z360.3,1750V,S-TRAP:190C;Sngl-SM			
16	EVS\100202-0.0.10ug	below	none	9		1.0
			Notes: 0.152ug/mLVS Std.Ds:-SMS.30% 0.25mm ID, 1.0um. 180C,5min - 240C 05C/m - 280C			
			Notes: 035C/m,5min,0.42VES PIT:2200nsec,0.65scans/sec.mnPP,m/z360 3.1750V,S-TRAP:190C,Sngl-SM			
17	EVS\100202-0.0.05ug	below	none	10		1.0
			Notes: 0.051ug/mLVS Std Ds:-SMS 30% 0.25mm ID, 1.0um. 180C,5min - 240C 05C/m - 280C			
			Notes: 035C/m,5min,0.42VES.PIT:2200nsec;1.65scans/sec;mnPP;m/z360.3,1750V,S-TRAP:190C,Sngl-SM			
18	EVS\100202-0 .045ug	below	none	11		1.0
			Notes: 0.045ug/mLVS Std.Ds:-SMS.30% 0.25mm ID, 1.0um; 180C,5min - 240C 05C/m - 280C			
			Notes: 035C/m,5min,0 42VES.PIT:2200nsec.0 65scans/sec;mnPP,m/z360.3,1750V,S-TRAP:190C,Sngl-SM			
19	EVS\100202-0.0 05ug	below	none	12		1.0
			Notes: 0.051ug/mLVS Std.Ds:-SMS 30% 0.25mm ID, 1.0um; 180C,5min - 240C 05C/m - 280C			
			Notes: 035C/m,5min,0 42VES.PIT:2200nsec;0 65scans/sec;mnPP,m/z360 3.1750V,S-TRAP:190C,Sngl-SM			
20	VS-101202-VS-1-	below	none	13		1.0
			Notes: Method Spike Plug/g VS Spike Into 5g Na2SO4. DEAET added pre-analysis; Ds:ZMS,S-TRAP.ZM:17SEV			
21	VS-101202-VS-2-	below	none	14		1.0
			Notes: Method Spike. Plug/g VS Spike Into 5g Na2SO4; DEAET added pre-analysis, Ds:ZMS,S-TRAP;ZM:17SEV			
22	VS-101202-VS-3B-	below	none	6		1.0
			Notes: JACADS Ash Sample No. LA91C1530. 5g + DEAET. Ultrasonic Extraction. no NAS cleanup. Initial Volume			
			Notes: = 5mL. diluted 1:10 w/CH2Cl2. Ds:ZMS/S-TRAP;ZM:17SEV			
23	VS-101202-VS-4B-	below	none	5		1.0
			Notes: JACADS Ash Sample No. LA91C1530. 5g + DEAET. Ultrasonic Extraction. no NAS cleanup. Initial Volume			
			Notes: = 5mL. diluted 1:10 w/CH2Cl2. Ds:ZMS.S-TRAP;ZM:17SEV			
24	CH2Cl2 Blank	below	none	6		1.0
			Notes: CH2Cl2 Blank. Ds:-SMS;30%0.25mm ID, 1.0um; 180C,5min - 240C 05C/m-280C			
			Notes: 035C/m.5min,0.42VES;PIT:2200nsec; 0 65scans/sec,mnPP,m/z360 3;1750V; S-TRAP:190C			
25	EVS\100202-1.0.05ug	below	none	8		1.0
			Notes: 0.051ug/mLVS Std.Ds:-SMS.30%0.25mm ID, 1.0um; 180C,5min - 240C 05C/m - 280C			
			Notes: 035C/m,5min,0.42VES;PIT:2200nsec,0 65scans/sec,mnPP,m/z360.3,1750V,S-TRAP:190C,Sngl-SM			
26				C:\saturnws\methods\Standby.mth		

Directory for Data Files: c:\saturnws\data\2002\october\101602\

Data File Names: ext4

Create and update a new Resultlist: c:\saturnws\data\2002\october\101602\vs101602-1.rsl

PROJECT LOGBOOK C 31763 7
Book Page

Subject Ultrasonic Extraction on Ash samples. Date 10-17-02
Signature Y. Abrha

Two blank (unspike) of Ash sample & Two spike Ash samples were made as follows.
- SOP-C-Saftley-204-01 was followed with deviations stated on page 2 of this logbook.

The amount of sample Ash (LAWIC 153 B) used was approximately 1g each. i.e.

Balance # 89 logbook (calck) C 31643 P-14

Sample 1

wt of beaker + Cent tube with cap = 39.9625 g
wt of beaker + C. tube with cap + Ash = 41.0628 g
wt of Ash = 1.1003 g

mixed with
wt of beaker = 26.7790 g
wt of beaker + Na_2SO_4 = 31.8319 g
wt of Na_2SO_4 = 5.0529 g

Sample 2

wt of beaker + Cent. tube with cap = 42.8513 g
wt of beaker + C tube with cap + Ash = 43.8517 g
wt of Ash = 1.0004 g

mixed with
wt of beaker = 29.8026 g
wt of beaker + Na_2SO_4 = 34.8962 g
wt of Na_2SO_4 = 5.0936 g

Read and Understood by me
Signature [signature] Date 10/22/02
Signature Date

Y-Abhan
C31763 P-7
back of P-7
10-17-02

Print Date: 21 Oct 2002 16:45:00

Calibration Block Report

Run File:	c:\saturnws\data\2002\october\101602#1\xvs100302-1, 1.8ug10-16-02.sms		
Instrument Method:	c:\... vs092502-mz208.2-0.42v-pil	Acquisition Date:	10/16/02 6:27 PM
Recalc Method:	c:\... vs-calib-dead-10-16-02.mi	Last Calibration:	10/21/02 4:34 PM
Sample List:		Compound Table Updated:	10/21/02 6:42 PM
Sequence:		Detector:	2000 Mass Spec
Workstation Name:	Saturn GC/MS Workstation	Workstation Version:	Version 3.32
Peak Measurement:	Area	Calibration Type:	External Standard Analysis

Retention Time (min)	Peak Name	Cal Method	X3	X2	X	C	r2	Cal Range	No. of Points	Edit Codes
28.812	VX	2 IG None		343619.5	1056865.5	24310.9	0.997597	1-6	6	

Curve Fit Codes	Origin Point Codes	Regression Weighting Codes		Edit Codes
1 linear	IN include	None	1/nX	1 curve
2 quadratic	IG ignore	1/n	1/X2	2 origin
3 cubic	FO force	1/X	1/nX2	3 coefficient

Ret. Time: 28.812 min. Peak Name: VX

Level	Amount	Replicate No.	Response	Avg. Response	Std. Dev.	% RSD	RF	Avg. RF
1	1.820000	1	3119859	3119858.5	#		1704840.7	
2	0.913000	1	1213277	1213276.6	#		1328890.1	
3	0.457000	1	647717	647716.8	#		1417323.5	
4	0.183000	1	294563	294563.2	#		1609634.7	
5	0.091000	1	73654	73654.0	#		809384.9	
6	0.046000	1	40986	40985.6	#		890991.3	1293510.8

= Too few points to calculate.

Peak Name	Level	Rep.	Injection Date/Time	Run Files
VX	1	1	10/16/02 7:12 PM	c:\... \october\101602#1\xvs100302-1, 1.8ug10-16-02001.sms
	2	1	10/16/02 7:57 PM	c:\... \2002\october\101602#1\xvs100302-2,0.91ug10-16-02.sms
	3	1	10/16/02 8:42 PM	c:\... \october\101602#1\xvs100302-3,0.46ug10-16-02003.sms
	4	1	10/16/02 9:27 PM	c:\... \2002\october\101602#1\xvs100302-4,0.18ug10-16-02.sms
	5	1	10/16/02 10:12 PM	c:\... \2002\october\101602#1\xvs100302-5,.09ug10-16-02.sms
	6	1	10/16/02 10:57 PM	c:\... \2002\october\101602#1\xvs100302-6,.046ug10-16-02.sms

Subject Ultrasonication Date 10-17-02
Continued Signature Y. Abrha

Sample 3.
wt of beaker + Centri tube with cap = 42.1677 g
wt of beaker + C. tube with cap + Ash = 43.2237 g
wt of Ash = 1.0560 g

The ash was spiked with 30 µL of 91.3 µg/mL VX (XVX-100302-B) then dried for 5 min and mixed with Na_2SO_4 (anhydrous)

wt of beaker = 28.9948 g
wt of beaker + Na_2SO_4 = 34.0201 g
wt of Na_2SO_4 = 5.0253 g

Sample 4.
wt of beaker + Centrifuge tube with cap = 41.3532 g
wt of beaker + Centrifuge tube with cap + Ash = 42.4465 g
wt of Ash = 1.0933 g

The Ash was spiked with 30 µL of 91.3 µg/mL VX (XVX-100302-B) then dried for 5 min and mixed with Na_2SO_4 (anhydrous)

wt of Beaker = 27.7386 g
wt of beaker + Na_2SO_4 = 32.7930 g
wt of Na_2SO_4 = 5.0544 g

All the samples were sonicated two times with 20 mL of 90% $MeCl_2$ / 10% IPA, then the extracts were combined and centrifuged for 5 min then blowed with stream of N_2 to 5 mL. 1 mL of each sample was taken & added 40 µL of DEAET then Analyzed by GC/ITMS.

Read and Understood by me
Signature [signature] Date 10/22/02
Signature Date

Subject Ultrasonication & Analysis Date 10-17-02
Contin Signature Y. Abrha

Sample ID #

Sample #1 XVX-101702-US-1 } unspike
#2 XVX-101702-US-2 }
#3 XVX-101702-US-3 } Spiked 30µL of ~~91.3~~ 91.3 µg/mL VX (XVX-100302-B) 10-17-02 see C31742 p-4
#4 XVX-101702-US-4 } to 1 g of ASL YA

Recording error 10-17-02
* ~~Another~~ Naming error YA 10-17-02
~~Sample XVX-101502-US-3D, 1mL of it spiked with 8µL DEAET~~
~~XVX-101502-US-4D, 1mL of it " " "~~
~~Sample ID #~~
~~XVX-101502-US-3DS, 1mL of it spiked with 8µL of DEAET & 8µ~~

To check the VX window for conformation we spike on the samples XVX-101602-3D & 4D with 30µL of 91.3µg/mL (XVX-100302-B), spike label equals 2.74 µg

Sample ID # XVX-101602-US-3DS1
XVX-101602-US-4DS1

Since the spike level was high we take 1mL each of the dilute sample i.e from samples XVX-101602-3D & 4D then spiked lower level

i.e. Sample ID XVX-101602-US-3D
XVX-101602-US-3DS2 (spiked with 8µL of 91.3 µg/mL)
XVX-101602-US-4D
XVX-101602-US-4DS2 (spiked with 8µL of 91.3 µg/mL)

Read and Understood by me

Signature [signature] Date 10/22/02
Signature Date

YA
C31763
back up-9
10-17-02

Varian Star Workstation - 3400 Sampleliist Thu Oct 17 14:40:47 2002

Sampleliist: G:\saturnws\data\2002\October\101702\VM101702L.smp

Created: Thu Oct 17 14:40:33 2002
Modified: Thu Oct 17 14:44:34 2002

Line	Sample Name	Injection Method	AutoLoadA	Vial	Inject Volume
1					
2	EVE195333-1.0 4ug	below	none	2	1.0
	Notes: 1 417ug/mLVE Std.Mix-SPH-3Hz 0 31um ID. 1.0um; 200C.5min - 310C 05C/m - 320C Notes: 025C/m.5min;0.02VEE.PIT3100usec;0 61econs/sec;noPP.m/3360.3,17500V;S-TRAP/1170C.Sngl-GS				
3	EVE-101703-VS-3001-	below	none	3	1.0
	Notes: JACADS Ash Sample No. LAW1C1535. 5g + DEAET, Ultrasonic Extraction, no HAS cleanup. Initial Volume Notes: = 5mL diluted 1:10 w/CH2Cl2.2 5ug VE Spike into dilution Mx4SMS.S-TRAP,SM317500V				
4	EVE-101703-VS-4001-	below	none	4	1.0
	Notes: JACADS Ash Sample No. LAW1C1535. 5g + DEAET, Ultrasonic Extraction. no HAS Cleanup. Initial Volume Notes: = 5mL. diluted 1:10 w/CH2Cl2.2 5ug VE Spike into dilution Mx4SMS;S-TRAP,SM317500V				
5	CH2Cl2 Blank	below	none	5	1.0
	Notes: CH2Cl2 Blank. Rtx-5MS 30m0 25um ID. 1 0um. 140C.5min - 240C 05C/m-250C Notes: 025C/m.5min;0.02VEE.PIT3100usec; 0 61econs/sec.noPP.m/3360 3,17500V; S-TRAP/1170C				
6	EVE101703-3.0 4ug	below	none	6	1.0
	Notes: 5 417ug/mLVE Std.Mix-SPH-3Hz 0 31um ID. 1 0um. 100C.5min - 240C 05C/m - 300C Notes: 015C/m.5min.3 41VEE.PIT3100usec.3 61econs/sec;noPP.m/3360.3,17500V.S-TRAP/1170C.Sngl-GS				
7	EVE-101703-VS-3001-	below	none	6	1.0
	Notes: JACADS Ash Sample No. LAW1C1535. 5g + DEAET, Ultrasonic Extraction, no HAS Cleanup. Initial Volume Notes: = 5mL. diluted 1:10 w/CH2Cl2.				
8	EVE-101703-VS-2001-	below	none	7	1.0
	Notes: JACADS Ash Sample No. LAW1C1535. 5g + DEAET Ultrasonic Extraction, no HAS Cleanup. Initial Volume Notes: = 5mL. diluted 1 10 w/CH2Cl2.2.5ug VE Spike into dilution Mx4SMS.S-TRAP,SM317500V				
9	EVE-101703-VS-1001-	below	none	8	1.0
	Notes: JACADS Ash Sample No LAW1C1535 5g + DEAET; Ultrasonic Extraction, no HAS Cleanup Initial Volume Notes: = 5mL. diluted 1 10 w/CH2Cl2. Mx4SMS.S-TRAP,SM317500V				
10	EVE-101703-VS-0002-	below	none	9	1.0
	Notes: JACADS Ash Sample No. LAW1C1535. 5g + DEAET, Ultrasonic Extraction, no HAS cleanup: Initial Volume Notes: = 5mL. diluted 1:10 w/CH2Cl2.0 5ug VE Spike into dilution Mx4SMS.S-TRAP,SM317500V				
11	CH2Cl2 Blank	below	none	0	1.0
	Notes: CH2Cl2 Blank. Rtx-1MS.30m0.25um ID. 1.0um. 100C.5min - 240C 05C/m-120C Notes: 015C/m.5min.0.02VEE.PIT3100usec. 0.61econs/sec;noPP.m/3360.3,17500V; S-TRAP/1170C				
12	EVE170303-1. 1 5ug	below	none	10	1.0
	Notes: 1.5ug/mLVE Std. Rtx-1MS.30M x 0.25um ID. 1.0um. 100C.5min - 240C 05C/m - 310C Notes: 015C/m.5min;0 42VEE.PIT3100usec.0.65econs/sec;noPP.m/3360.3,17500V,S-TRAP/1170C.Sngl-GS				
13	EVE100307-1. 1 0ug	below	none	10	1.0
	Notes: 1.0ug/mLVE Std. Rtx-1MS:30M x 0.25um ID. 1 0um. 100C.5min - 240C 05C/m - 240C Notes: 015C/m.5min.0 42VEE.PIT3100usec;0 65econs/sec;noPP.m/3360 3,17500V,S-TRAP/1170C.Sngl-GS				
14	EVE100303-1.0 91ug	below	none	11	1.0
	Notes: 0.91ug/mLVE Std.Rtx-5MS:30M x 0.25um ID. 1.0um. 100C.5min - 240C 05C/m - 320C Notes: 015C/m.5min.0.41VEE.PIT3100usec.0.65econs/sec.noPP.m/3360.3,17500V;S-TRAP/1170C.Sngl-GS				
15	EVE100330-1.0 4ug	below	none	5	1.0
	Notes: 0.457ug/mLVE Std.Rtx-5MS.30M 0.25um ID. 1.0um. 100C.5min - 240C 05C/m - 310C Notes: 025C/m.5min;0.42VEE;PIT3100usec.0.65econs/sec.noPP;m/3360.3,17500V.S-TRAP/1170C.Sngl-GS				
16	EVE100303-4.0.10ug	below	none	12	1.0
	Notes: 4 10ug/mLVE Std.Rtx-5MS.30M 0.25um ID. 1 0um. 100C.5min - 240C 05C/m - 310C Notes: 015C/m.5min;0.42VEE;PIT3100usec.3 61econs/sec.noPP;m/3360.3,17500V.S-TRAP/1170C.Sngl-GS				
17	EVE100309-1.,051ug	below	none	13	1.0
	Notes: 0.051ug/mLVE Std Rtx-5MS MS/2)Mx 0 25um ID. 1.0um; 100C.5min - 240C 05C/m - 307C Notes: 025C/m.5min;0.42VEE.PIT3100usec.0.65econs/sec;noPP.m/3360.3,17500V.S-TRAP/1170C.Sngl-GS				
18	EVE100332-1.,.04ug	below	none	14	1.0
	Notes: 0 04ug/mLVE Std.Rtx-5MS 30Mx 0.25um ID. 1.0um. 100C.5min - 240C 05C/m - 300C Notes: 025C/m.5min;0.42VEE.PIT3100usec.0.65econs/sec,noPP.m/3360.3,17500V.S-TRAP/1170C;Sngl-GS				
19	EVE100332-2.0.04ug	below	none	5	1.0
	Notes: 0.457ug/mLVE Std.Rtx-5MS.30Mx 0 25um ID. 1.0um. 100C.5min - 240C 05C/m - 300C Notes: 025C/m.5min.0 42VEE.PIT3100usec.0 65econs/sec;noPP.m/3360.3,17500V.S-TRAP/1170C.Sngl-GS				
20	EVE-101703-VS-1-	below	none	15	1.0
	Notes: JACADS Ash Sample No LAW1C1535. 5g + 5g NaSO4; Ultrasonic Extraction, no HAS cleanup; Final Volume Notes: = 5mL + DEAET; Unspiked; Mx4SMS.S-TRAP,SM317500V				
21	EVE-101703-VS-2-	below	none	16	1.0
	Notes: JACADS Ash Sample No LAW1C1535. 5g + 5g NaSO4. Ultrasonic Extraction. no HAS cleanup; Final Volume Notes: = 5mL + DEAET, Unspiked, Mx4SMS.S-TRAP,SM317500V				
22	EVE-101703-VS-3-	below	none	17	1.0
	Notes: JACADS Ash Sample No. LAW1C1535. 5g + 5g NaSO4; Ultrasonic Extraction, no HAS cleanup, Final Volume Notes: = 5mL + DEAET; 2.5ug/g VE Spike Mx4SMS.S-TRAP,SM317500V				
23	EVE-101703-VS-4-	below	none	18	1.0
	Notes: JACADS Ash Sample No. LAW1C1535. 5g + 5g NaSO4; Ultrasonic Extraction. no HAS cleanup Final Volume Notes: = 5mL + DEAET; 2.5ug/g VE Spike. Mx4SMS.S-TRAP,SM317500V				
24	CH2Cl2 Blank	below	none	0	1.0
	Notes: CH2Cl2 Blank. Rtx-1MS.30M0 25um ID. 1 0um. 100C.5min - 240C 05C/m-200C Notes: 015C/m.5min;0 42VEE.PIT3100usec. 0 61econs/sec;noPP.m/3360.3.17500V. S-TRAP/1170C				
25	EVE101332-2.0 4ug	below	none	5	1.0
	Notes: 5 457ug/mLVE Std.Rtx-5MS:30Mx 0 25um ID. 1 0um. 100C.5min - 240C 05C/m - 260C Notes: 015C/m.5min.0.42VEE.PIT3100usec.0 61econs/sec;noPP.m/3360.3,17500V;S-TRAP/1170C;Sngl-GS				
26	EVE101331-1.0 4ug	below	none	0	1.0
	Notes: 0 457ug/mLVE Std.Rtx-5MS:30Mx 0 25um ID. 1 0um 100C.5min - 240C 05C/m - 240C Notes: 015C/m.5min;0.42VEE.PIT3100usec.0 61econs/sec;noPP.m/3360 3,17500V,S-TRAP/1170C;Sngl-GS				
27			C:\saturnws\methods\Sunday.mth		

Directory for Data Files: G:\saturnws\data\2002\october\101702

Data File Name: %d%

Create and update a new Sampleliist. G:\saturnws\data\2002\october\101702\VM101702-1.sml

PROJECT LOGBOOK

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Book Page

Project: Calibration of Std Cont.

Date: 10-17-02

Signature: Y. Abbine

Calibration of Std was made range 1.83 µg/mL to 0.046 µg/mL VX std with DEAET

1 mL each of the standards was transferred to auto sampler vials.

XVX-100302-1 1.83 µg/mL
XVX-100302-2 0.91 "
XVX-100302-3 0.46 "
XVX-100302-4 0.18 "
XVX-100302-5 0.091 "
XVX-100302-6 0.046 "

Print Date: 21 Oct 2002 18:43:18

Calibration Block Report

Page 1 21 Oct 2002 18:43:14

Run File:	c:\saturnws\data\2002\october\101702#1\xvx100302-3,0.46ug10-17-02.sms		
Instrument Method:	c:\... vx092502-mz268 2-0.42v-pit	Acquisition Date:	10/17/02 2:52 PM
Recalc Method:	c:\... vx-calib-deaet-10-17-02.mt	Last Calibration:	10/18/02 1:02 PM
Sample List:		Compound Table Updated:	10/21/02 7:23 PM
Sequence:		Detector:	2000 Mass Spec
Workstation Name:	Saturn GC/MS Workstation	Workstation Version:	Version 5.52
Peak Measurement:	Area	Calibration Type:	External Standard Analysis

Retention Time (min)	Peak Name	Cal. Method	X3	X2	X	C	r2	Cal. Range	No. of Points	Edit Codes
28.612	VX	2 IG None		204171.5	1286284.6	5592.58	0.997614	1-6	6	

Curve Fit Codes
1 linear
2 quadratic
3 cubic

Origin Point Codes
IN include
IG ignore
FO force

Regression Weighting Codes
None 1/nX
1/n 1/X2
1/X 1/nX2

Edit Codes
1 curve
2 origin
3 coefficient

Ret. Time: 28.612 min. Peak Name: VX

Level	Amount	Replicate No.	Response	Avg. Response	Std. Dev.	% RSD	RF	Avg. RF
1	1.830000	1	3054257	3054256.5	#		1668992.4	
2	0.913000	1	1281206	1281205.5	#		1403281.9	
3	0.457000	1	735341	735341.1	#		1609061.6	
4	0.183000	1	251252	251252.4	#		1372844.0	
5	0.091000	1	99121	99120.7	#		1089238.1	
6	0.046000	1	45644	45643.7	#		992253.3	1355946.9

= Too few points to calculate.

Peak Name	Level	Rep.	Injection Date/Time	Run Files
VX	1	1	10/17/02 11:16 PM	c:\... \october\101702#1\xvx100302-1, 1.8ug10-17-02001.sms
	2	1	10/18/02 12:01 AM	c:\... \2002\october\101702#1\xvx100302-2,0.91ug10-18-02.sms
	3	1	10/18/02 12:48 AM	c:\... \2002\october\101702#1\xvx100302-3,0.46ug10-18-02.sms
	4	1	10/18/02 1:31 AM	c:\... \2002\october\101702#1\xvx100302-4,0.18ug10-18-02.sms
	5	1	10/18/02 2:16 AM	c:\... \2002\october\101702#1\xvx100302-5,.091ug10-18-02.sms
	6	1	10/18/02 3:01 AM	c:\... \2002\october\101702#1\xvx100302-6,.046ug10-18-02.sms

Read and Understood by me

Signature: [signature] Date: 10/22/02

Signature: _____ Date: _____

YA
10-17-02
C31763
Back of P-10

File Cross reference logbook sample ID & MS ID#

Sample Identification		Analyzed concentration (µg/mL)	Final Volume (mL)	VX Spike Level
Sample ID No.	ITMS File ID No.			
Samples prepared on 10-14-02				
XVX-101402-US-1	VX-101402-US-1-10-15-02	0	10	
XVX-101402-US-2	VX-101402-US-2-10-15-02	0	10	
XVX-101402-US-3	VX-101402-US-3-10-15-02	0.295	10	0.457ug/g
XVX-101402-US-4	VX-101402-US-4-10-15-02	0.109	10	0.457ug/g
Samples prepared on 10-15-02				
XVX-101502-US-1	VX-101502-US-1-10-15-02 -1-10-17-02	0.260 0.200	2	0.091ug/g
XVX-101502-US-2	VX-101502-US-2-10-15-02 -2-10-17-02	0.327 0.219	2	0.091ug/g
XVX-101502-US-3	VX-101502-US-3-10-15-02	NC	5	
XVX-101502-US-4	VX-101502-US-4-10-16-02	NC	5	
Samples XVX-101502-3 and -4 were diluted 1:10 with $MeCl_2$ on 10-16-02				
XVX-101602-US-3D	VX-101502-US-3D-10-16-02 VX-101502-US-3D-10-17-02	0.368 0.375	1:10	
XVX-101602-US-4D	VX-101502-US-4D-10-16-02 VX-101502-US-4D-10-17-02	0.295 0.365	1:10	
XVX-101602-US-3DS1 [a]	VX-101502-US-3DS-10-17-02	NC	1:10	2.7ug/mL
XVX-101602-US-4DS1 [a]	VX-101502-US-4DS-10-17-02	NC	1:10	2.7ug/mL
XVX-101602-US-3D	VX-101602-US-3D1-10-17-02	0.302	1:10	
XVX-101602-US-3DS2 [a]	VX-101702-US-3DS2-10-17-02	1.02	1:10	0.73ug/mL
XVX-101602-US-4D	VX-101602-US-4D1-10-17-02	0.285	1:10	
XVX-101602-US-4DS2 [a]	VX-101702-US-4DS2-10-17-02	1.01	1:10	0.73ug/mL
Samples prepared on 10-17-02				
XVX-101702-US-1	VX-101702-US-1-10-18-02	0.84	5	
XVX-101702-US-2	VX-101702-US-2-10-18-02	0.78	5	
XVX-101702-US-3	VX-101702-US-3-10-18-02	1.41	5	2.7ug/g
XVX-101702-US-4	VX-101702-US-4-10-18-02	1.46	5	2.7ug/g

Note: [a] The 1mL of the 5-gram diluted ash extracts were spiked with VX to assist in confirming the presence of VX

Y. Arthur
10-22-02
C 31763

Logbook Sample ID & ITMS File ID No
identifications.

Sample Identification		Analyzed concentration (µg/mL)	Final Volume (mL)	VX Spike Level
Sample ID No.	ITMS File ID No.			
Samples prepared on 10-14-02				
XVX-101402-US-1	VX-101402-US-1-10-15-02	0	10	
XVX-101402-US-2	VX-101402-US-2-10-15-02	0	10	
XVX-101402-US-3	VX-101402-US-3-10-15-02	0.295	10	0.457ug/g
XVX-101402-US-4	VX-101402-US-4-10-15-02	0.109	10	0.457ug/g
Samples prepared on 10-15-02				
XVX-101502-US-1	VX-101502-US-1-10-15-02 -1-10-17-02	0.260 0.200	2	0.091ug/g
XVX-101502-US-2	VX-101502-US-2-10-15-02 -2-10-17-02	0.327 0.219	2	0.091ug/g
XVX-101502-US-3	VX-101502-US-3-10-15-02	NC	5	
XVX-101502-US-4	VX-101502-US-4-10-16-02	NC	5	
Samples XVX-101502-3 and -4 were diluted 1:10 with $MeCl_2$ on 10-16-02				
XVX-101602-US-3D	VX-101502-US-3D-10-16-02 VX-101502-US-3D-10-17-02	0.368 0.375	1:10	
XVX-101602-US-4D	VX-101502-US-4D-10-16-02 VX-101502-US-4D-10-17-02	0.295 0.365	1:10	
XVX-101602-US-3DS1 [a]	VX-101502-US-3DS-10-17-02	NC	1:10	2.7ug/mL
XVX-101602-US-4DS1 [a]	VX-101502-US-4DS-10-17-02	NC	1:10	2.7ug mL
XVX-101602-US-3D	VX-101602-US-3D1-10-17-02	0.302	1:10	
XVX-101602-US-3DS2 [a]	VX-101702-US-3DS2-10-17-02	1.02	1:10	0.73ug/mL
XVX-101602-US-4D	VX-101602-US-4D1-10-17-02	0.285	1:10	
XVX-101602-US-4DS2 [a]	VX-101702-US-4DS2-10-17-02	1.01	1:10	0.73ug/mL
Samples prepared on 10-17-02				
XVX-101702-US-1	VX-101702-US-1-10-18-02	0.84	5	
XVX-101702-US-2	VX-101702-US-2-10-18-02	0.78	5	
XVX-101702-US-3	VX-101702-US-3-10-18-02	1.41	5	2.7ug/g
XVX-101702-US-4	VX-101702-US-4-10-18-02	1.46	5	2.7ug/g

Note: [a] The 1mL of the 5-gram diluted ash extracts were spiked with VX to assist in confirming the presence of VX

PROJECT LOGBOOK

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ubject Data Summary Report

Date 10-22-02

Signature Y. Abrha

Data Summary of the Ash Samples

Sample Identification		VX Concentration (µg/mL)		Recovery %
Sample ID No.	ITMS File ID No.	Analyzed (µg/mL)	Sample (µg/g)	
Samples prepared on 10-14-02				
XVX-101402-US-1	VX-101402-US-1-10-15-02	0		
XVX-101402-US-2	VX-101402-US-2-10-15-02	0		
XVX-101402-US-3	VX-101402-US-3-10-15-02	0.295	2.95	129.1
XVX-101402-US-4	VX-101402-US-4-10-15-02	0.109	1.09	47.7
Samples prepared on 10-15-02				
XVX-101502-US-1	VX-101502-US-1-10-15-02	0.260	0.52	114.3
	-1-10-17-02	0.200	0.40	87.9
XVX-101502-US-2	VX-101502-US-2-10-15-02	0.327	0.654	143.7
	-2-10-17-02	0.219	0.438	96.3
XVX-101502-US-3	VX-101502-US-3-10-15-02	NC		
XVX-101502-US-4	VX-101502-US-4-10-16-02	NC		
Samples XVX-101502-3 and -4 were diluted 1:10 with $MeCl_2$ on 10-16-02				
XVX-101602-US-3D	VX-101502-US-3D-10-16-02	0.368	3.68	
	VX-101502-US-3D-10-17-02	0.375	3.75	
XVX-101602-US-4D	VX-101502-US-4D-10-16-02	0.295	2.95	
	VX-101502-US-4D-10-17-02	0.365	3.65	
XVX-101602-US-3DS1 [a]	VX-101502-US-3DS-10-17-02	NC		
XVX-101602-US-4DS1 [a]	VX-101502-US-4DS-10-17-02	NC		
XVX-101602-US-3D	VX-101602-US-3D1-10-17-02	0.302	3.02	
XVX-101602-US-3DS2 [a]	VX-101702-US-3DS2-10-17-02	1.02	10.2	98.3
XVX-101602-US-4D	VX-101602-US-4D1-10-17-02	0.285	2.85	
XVX-101602-US-4DS2 [a]	VX-101702-US-4DS2-10-17-02	1.01	10.1	99.3
Samples prepared on 10-17-02				
XVX-101702-US-1	VX-101702-US-1-10-18-02	0.84	4.20	
XVX-101702-US-2	VX-101702-US-2-10-18-02	0.78	3.90	
XVX-101702-US-3	VX-101702-US-3-10-18-02	1.41	7.05	111.1
XVX-101702-US-4	VX-101702-US-4-10-18-02	1.46	7.30	119.0

Note: [a] The 1mL of the 5-gram diluted ash extracts were spiked with VX to assist in confirming the presence of VX

Read and Understood by me

Signature [signature] Date 10/22/02

Signature _____ Date _____

Print Date: ...

PROJECT LOGBOOK

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Book Page

ject Standard preparation cont. Date 10-3-02

Signature Y. Abrha

VX stds. preparation

VX Working Std.

Std ID	VX Stock Std (µg/mL)	Vol (mL) of VX Stock used	Vol (mL) of DEAET Stock used	Final Vol (mL)	Final VX Conc. (µg/mL)	Final DEAET Conc. (mg/mL)
XVX-100302-1	B (91.3)	1.0	2	50	1.83	0.898
XVX-100302-2	B (91.3)	1.0	4	100	0.91	0.898
XVX-100302-3	B (91.3)	0.5	4	100	0.46	0.898
XVX-100302-4	C (9.13)	1.0	2	50	0.18	0.898
XVX-100302-5	C (9.13)	0.5	2	50	0.091	0.898
XVX-100302-6	C (9.13)	0.5	4	100	0.046	0.898

Std ID	VX Stock Std (µg/mL)	Vol (mL) of VX Stock used	Vol (mL) of Enhancement Stock used	Final Vol (mL)	Final VX Conc. (µg/mL)	Final Enhancement Conc. (mg/mL)
XVX-100302-1e	B (91.3)	1.0	2	50	1.83	0.713
XVX-100302-2e	B (91.3)	1.0	4	100	0.91	0.713
XVX-100302-3e	B (91.3)	0.5	4	100	0.46	0.713
XVX-100302-4e	C (9.13)	1.0	2	50	0.18	0.713
XVX-100302-5e	C (9.13)	0.5	2	50	0.091	0.713
XVX-100302-6e	C (9.13)	0.5	4	100	0.046	0.713

Note: All transfers made using vol. pipettes into volumetric flasks all are diluted to vol with hexane

Note: Two HAE extractions using 1.0N & 0.1N HCl acids & 0.363N & 0.0363N HCl acid was made their chromatograms are two different & their recovery is good with less concentrated acid used.

Read and Understood by me

Signature Chantel Hamm Date 10-25-02

Signature _____ Date _____

YA
C31763 P-3
10-14-02

****

Varian Star Workstation - 8400 SampleList Tue Oct 15 10:24:37 2002

SampleList: C:\saturnws\Data\2002\October\10140281\VX10140281.smp

1: Mon Oct 14 19:08:54 2002
.ied: Tue Oct 15 06:11:40 2002

****

Line	Sample Name	Injection Notes	AutoLink	Vial	Inject Volume
1			C:\saturnws\Methods\VX-2002\VX092502-mz268.2-0.42V-PIT-200-ST-0.65-noPP-Purge@20-EI;Multi1750v-300v.mth		
2	XVX-050902-6,spk6-	below	none	1	1.0
	Notes	XVX-11-07-01A,spike,.046ug/mL VX; HAE extract:Rtx-5MS:30Mx0.25mmID,1um;			
	Notes	100C,5min-240C@5C/m-280C@25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170			
3	XVX-050902-6,spk6-	below	none	1	1.0
	Notes	XVX-11-07-01A,spike,.046ug/mL VX; HAE extract:Rtx-5MS:30Mx0.25mmID,1um;			
	Notes	100C,5min-240C@5C/m-280C@25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170			
4	XVX100302-1, 1.8ug	below	none	2	1.0
	Notes	1.83ug/mLVX Std, Rtx-5MS:30M x 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C			
	Notes	@25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN			
5	XVX100302-1, 1.8ug	below	none	2	1.0
	Notes	1.83ug/mLVX Std, Rtx-5MS:30M x 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C			
	Notes	@25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN			
6	XVX100302-2,0.91ug	below	none	3	1.0
	Notes	0.911ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C			
	Notes	@25C/m,5min;0.42V-EE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;TS-RAP@170C;Sngl-GN			
7	XVX100302-3,0.46ug	below	none	4	1.0
	Notes	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C			
	Notes	@25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN			
8	XVX100302-4,0.18ug	below	none	5	1.0
	Notes	0.183ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C			
	Notes	@25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN			
9	XVX100302-5,.091ug	below	none	6	1.0
	Notes	0.091ug/mLVX Std Rtx-5Sil MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C			
	Notes	@25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN			
10	XVX100302-6,.046ug	below	none	7	1.0
	Notes	0.046ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C			
	Notes	@25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN			
11	XVX100302-3,0.46ug	below	none	4	1.0
	Notes	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C			
	Notes	@25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN			
12	VX-101402-US-1	below	none	8	1.0
	Notes	Methylene Chloride/IPA Sonicator Blank + DEAET;RtxSMS;S-TRAP;EM@1750V			
13	VX-101402-US-2	below	none	9	1.0
	Notes	Methylene Chloride/IPA Sonicator Blank + DEAET;RtxSMS;S-TRAP;EM@1750V			
14	VX-101402-US-3	below	none	10	1.0
	Notes	Method Spike:.457ug/mL VX Spike into Sg NaSO4 + DEAET; RtxSMS;S-TRAP;EM@1750V			
15	VX-101402-US-4	below	none	11	1.0
	Notes	Method Spike:.457ug/mL VX Spike into Sg NaSO4 + DEAET; RtxSMS;S-TRAP;EM@1750V			
16	XVX100302-3,0.46ug	below	none	4	1.0
	Notes	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C			
	Notes	@25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN			
17			C:\saturnws\Methods\Standby.mth		

Directory for Data Files: c:\saturnws\data\2002\october\10140281

Data File Names: %%d

Create and update a new RecalcList: c:\saturnws\data\2002\october\10140281\vx101402-1.rcl.

PROJECT LOGBOOK C 31726 20
Book Page

Subject VX stockstandard Date 7-25-02

Signature Tiffany Parrish

Prepare VX stock std from CASARM

VX: casarm GCBC VX-U-2128-CTF-N can VX14 Lot#
VX1-22-98 Vial # 150

IPA: 2-Propanol Aldrich Lot # T1 16467P1

Balance # 70 Daily Calibration ck C31665 p6

Initial vial = 22.046 g
Final vial = 22.023 g
amount removed = 0.023 g
Remaining VX in vial = 8.74 g

Balance # 307 Daily calibration ck C31651 p8

25mL vol flask = 23.2871 g
VX + flask = 23.3108 g
VX = 0.0237 g

23.7mg VX was transfered to a 25mL vol flask and diluted to vol w/ IPA
Labeled XVX 7-25-02
Final conc = 0.948 mg/mL VX
Purity of VX = 96.3%
Purity corrected conc = 0.913 mg/mL VX

Placed in 3 vials labeled XVX 7-25-02 A-C and stored in can XVX 166 in CAF dilute refrigerator

Read and Understood by me

Signature Sarah O'Brien Date 8-1-02

Signature Date

****

Varian Star Workstation - RecalcList Tue Oct 15 10:29:06 2002

lcList: C:\saturnws\Data\2002\October\101402#1\VX101402-1.RCL

Created: Mon Oct 14 19:12:05 2002
Modified: Tue Oct 15 10:29:02 2002

****

Line	Sample Type	Sample Name	Data File	Inj.
1	Analysis	XVX-050902-6,spk6-	XVX-050902-6,spk6-10-14-02.SMS	1
2	Analysis	XVX-050902-6,spk6-	XVX-050902-6,spk6-10-14-02001.SMS	1
3	Analysis	XVX100302-1, 1.8ug	XVX100302-1, 1.8ug10-14-02.SMS	1
4	Analysis	XVX100302-1, 1.8ug	XVX100302-1, 1.8ug10-14-02001.SMS	1
5	Analysis	XVX100302-2,0.91ug	XVX100302-2,0.91ug10-14-02.SMS	1
6	Analysis	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-14-02.SMS	1
7	Analysis	XVX100302-4,0.18ug	XVX100302-4,0.18ug10-14-02.SMS	1
8	Analysis	XVX100302-5,.091ug	XVX100302-5,.091ug10-15-02.SMS	1
9	Analysis	XVX100302-6,.046ug	XVX100302-6,.046ug10-15-02.SMS	1
10	Analysis	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-15-02.SMS	1
11	Analysis	VX-101402-US-1	vx-101402-us-1-10-15-02.sms	1
12	Analysis	VX-101402-US-2	vx-101402-us-2-10-15-02.sms	1
13	Analysis	VX-101402-US-3	vx-101402-us-3-10-15-02.sms	1
14	Analysis	VX-101402-US-4	vx-101402-us-4-10-15-02.sms	1
15	Analysis	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-15-02001.SMS	1

Print Date: 15 Oct 2002 10:51:28

Calibration Block Report

Run File:	c:\saturnws\data\2002\october\101402#1\xvx100302-1, 1.8ug10-14-02001.sms		
Instrument Method:	c:\... vx092502-mz268.2-0.42v-pit	Acquisition Date:	10/14/02 9:27 PM
Recalc Method:	c:\... vx-calib-deaet-10-14-02.mt	Last Calibration:	10/15/02 10:16 AM
Sample List:		Compound Table Updated:	10/15/02 10:37 AM
Sequence:		Detector:	2000 Mass Spec
Workstation Name:	Saturn GC/MS Workstation	Workstation Version:	Version 5.52
Peak Measurement:	Area	Calibration Type:	External Standard Analysis

Retention Time (min)	Peak Name	Cal. Method	X3	X2	X	C	r2	Cal. Range	No. of Points	Edit Codes
28.812	VX	2 IG None		96226.5	213859.3	-1749.64	0.999124	1-6	6	

Curve Fit Codes	Origin Point Codes		Regression Weighting Codes		Edit Codes
1 linear	IN	include	None	1/nX	1 curve
2 quadratic	IG	ignore	1/n	1/X2	2 origin
3 cubic	FO	force	1/X	1/nX2	3 coefficient

Ret. Time: 28.812 min. Peak Name: VX

Level	Amount	Replicate No.	Response	Avg. Response	Std. Dev.	% RSD	RF	Avg. RF
1	1.830000	1	712854	712854.1	#		389537.8	
2	0.913000	1	268620	268619.9	#		294216.8	
3	0.457000	1	118646	118646.4	#		259620.2	
4	0.183000	1	54898	54897.8	#		299987.8	
5	0.091000	1	10577	10576.5	#		116225.6	
6	0.046000	1	3477	3476.6	#		75578.7	239194.5

= Too few points to calculate.

Peak Name	Level	Rep.	Injection Date/Time	Run Files
VX	1	1	10/14/02 9:27 PM	c:\... \october\101402#1\xvx100302-1, 1.8ug10-14-02001.sms
	2	1	10/14/02 10:11 PM	c:\... \2002\october\101402#1\xvx100302-2,0.91ug10-14-02.sms
	3	1	10/14/02 10:55 PM	c:\... \2002\october\101402#1\xvx100302-3,0.46ug10-14-02.sms
	4	1	10/14/02 11:39 PM	c:\... \2002\october\101402#1\xvx100302-4,0.18ug10-14-02.sms
	5	1	10/15/02 12:23 AM	c:\... \2002\october\101402#1\xvx100302-5,.091ug10-15-02.sms
	6	1	10/15/02 1:06 AM	c:\... \2002\october\101402#1\xvx100302-6,.046ug10-15-02.sms

Print Date: 15 Oct 2002 10:21:49

Calibration Curve Report

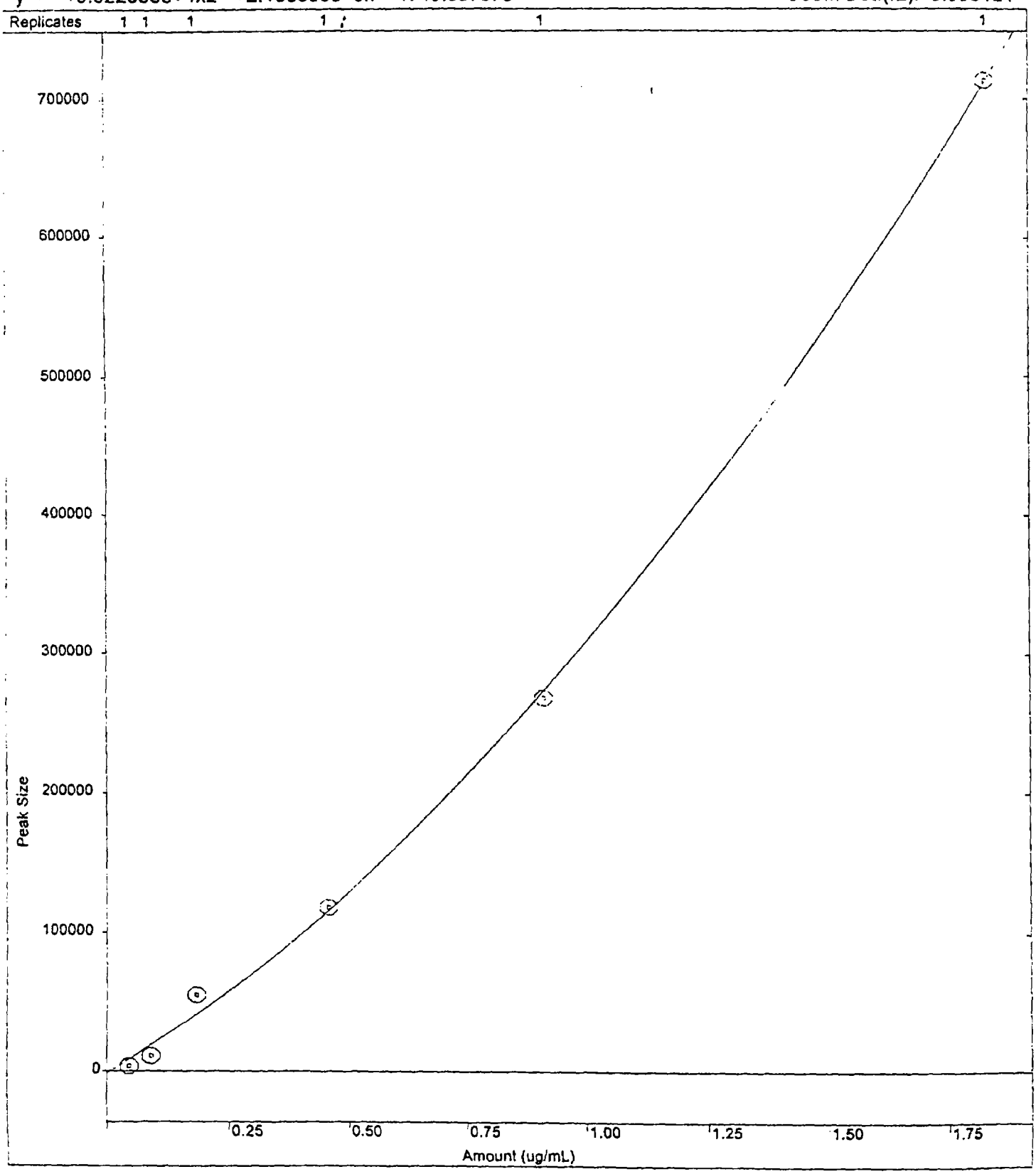
YA C 31763 back p-3

e: c:\saturnws\methods\vx-2002\vx-calib-deaet-10-14-02-m.mth
tector: 2000 Mass Spec, Address: 40

VX

Curve Fit: Quadratic, Origin: Ignore, Weight: None
y = +9.622655e+4x2 +2.138593e+5x -1749.637573

Resp. Fact. RSD: 50.04%
Coeff. Det.(r2): 0.999124

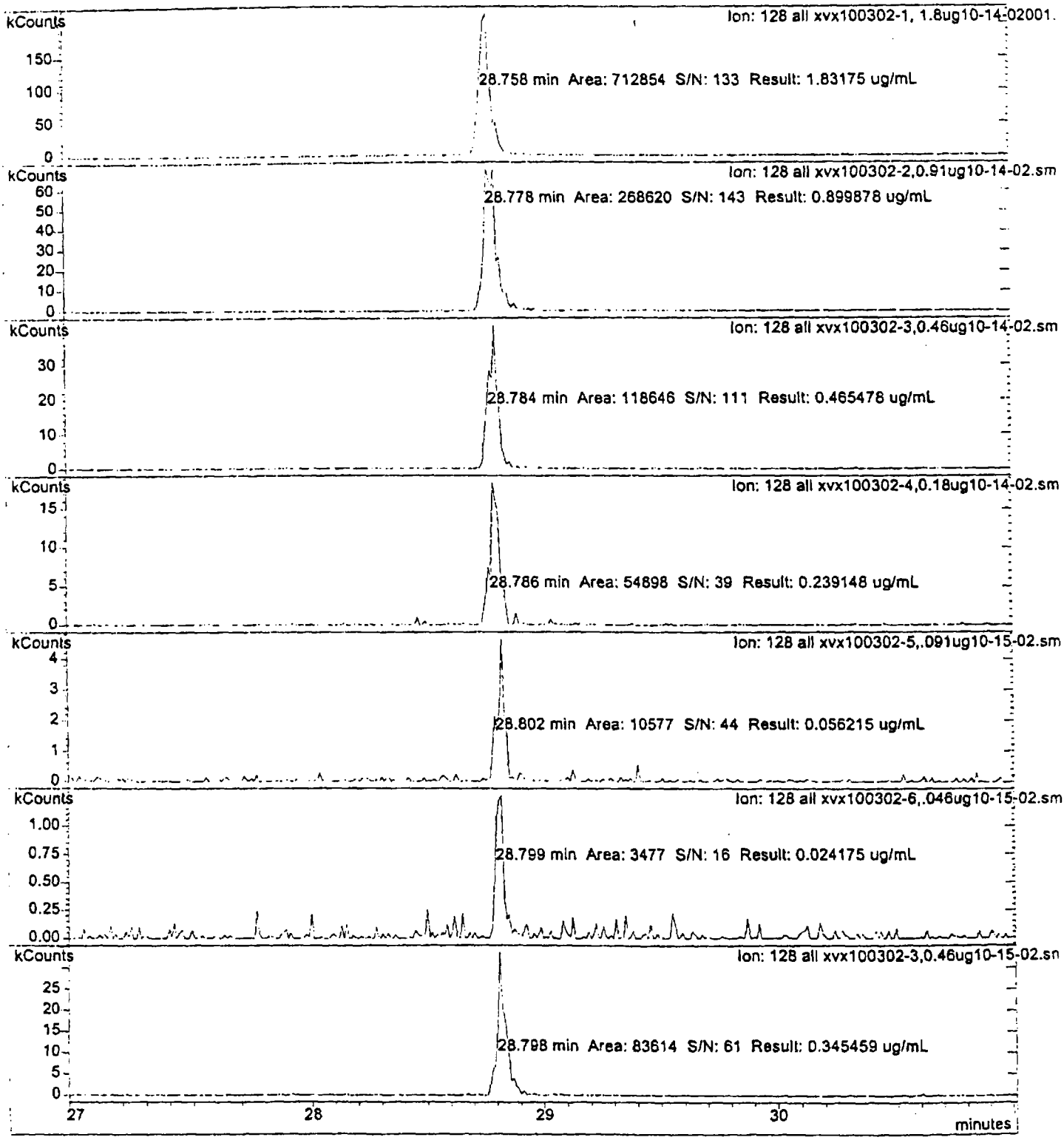


Print Date: 15 Oct 2002 10:37:14

Yingzhu S - Abbie
C31763
back p-3
10-14-02

Chromatogram Plots

Plot 1: c:\... \xvx100302-1, 1.8ug10-14-02001.sms Ion: 128 all
Plot 2: c:\... \101402#1\xvx100302-2,0.91ug10-14-02.sms Ion: 128 all
Plot 3: c:\... \101402#1\xvx100302-3,0.46ug10-14-02.sms Ion: 128 all
Plot 4: c:\... \101402#1\xvx100302-4,0.18ug10-14-02.sms Ion: 128 all
Plot 5: c:\... \101402#1\xvx100302-5,.091ug10-15-02.sms Ion: 128 all
Plot 6: c:\... \101402#1\xvx100302-6,.046ug10-15-02.sms Ion: 128 all
Plot 7: c:\... \101402#1\xvx100302-3,0.46ug10-15-02.sms Ion: 128 all

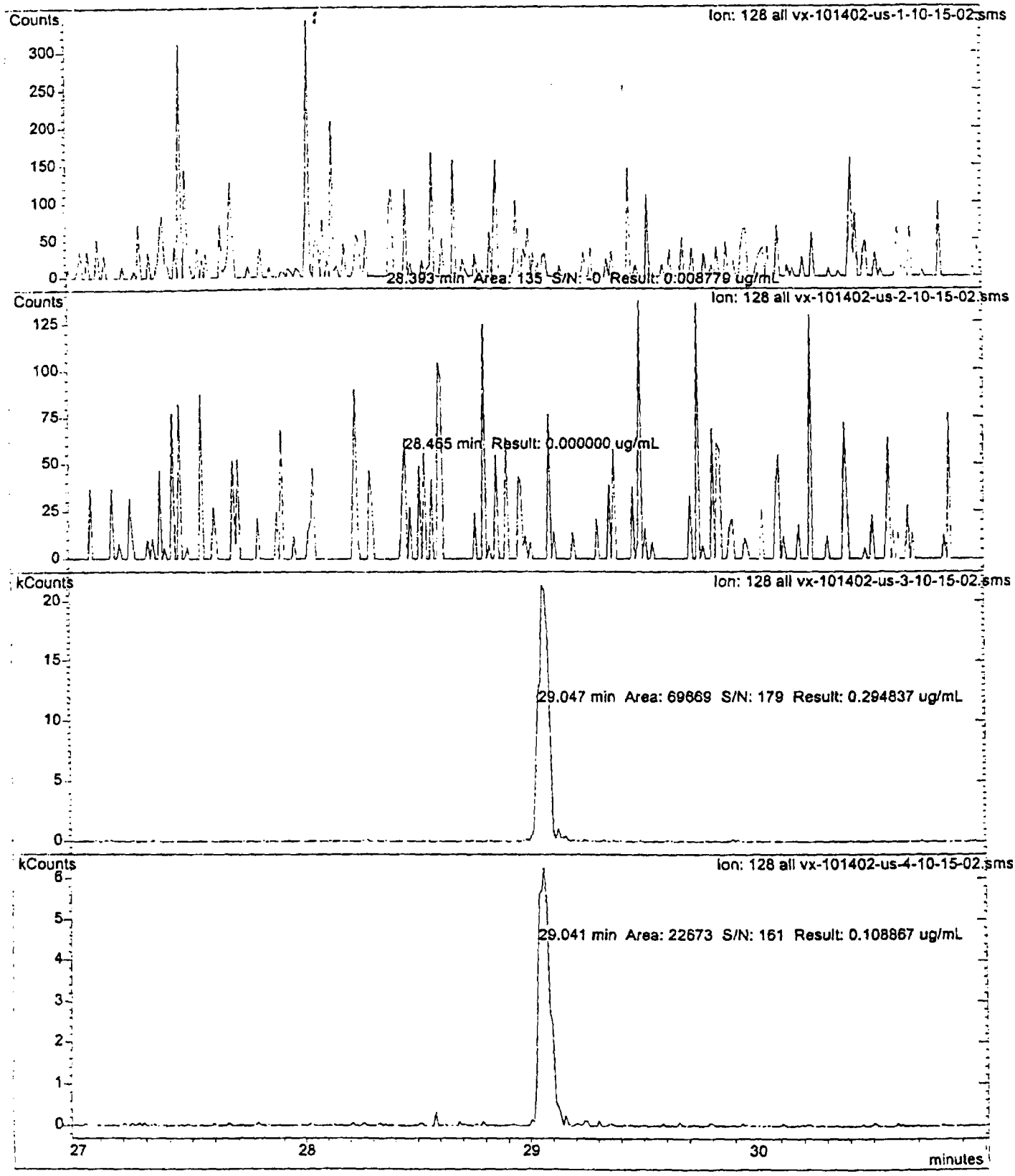


Print Date: 15 Oct 2002 10:57:03

Xingtus - Abstr
C31763 Lab p.3
10/14/02

Chromatogram Plots

Plot 1: c:\... \101402#1\vx-101402-us-1-10-15-02.sms Ion: 128 all
Plot 2: c:\... \101402#1\vx-101402-us-2-10-15-02.sms Ion: 128 all
Plot 3: c:\... \101402#1\vx-101402-us-3-10-15-02.sms Ion: 128 all
Plot 4: c:\... \101402#1\vx-101402-us-4-10-15-02.sms Ion: 128 all



Print Date: 15 Oct 2002 10:45:20

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-1, 1.8ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-1, 1.8ug	Acquisition Date:	10/14/02 9:27 PM
Vial:	2	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	1.83ug/mLVX Std, Rtx-5MS:30M x 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans /sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \october\101402#1\xvx100302-1, 1.8ug10-14-02001.sms		

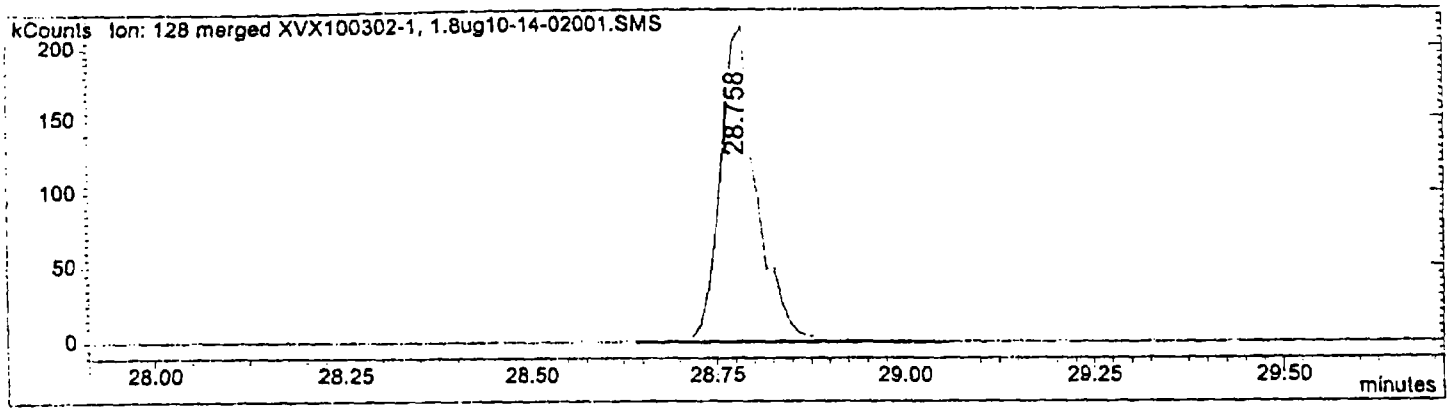
YA
C31763 BP
10-14-02

Analyte: VX

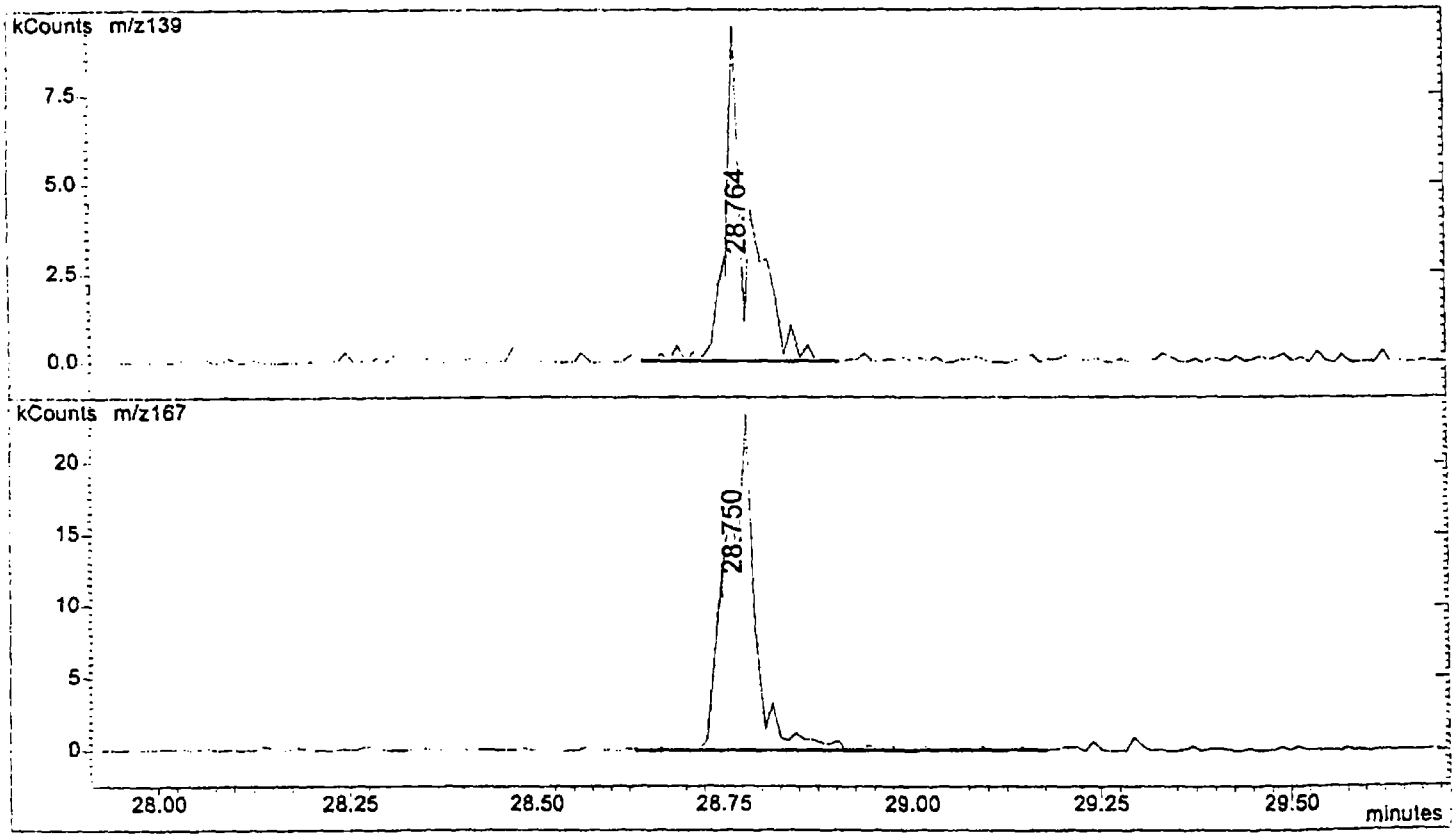
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.758 min.	28.812 min.	28.112 - 29.512 min.	712854	Failed

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.764 min.	2.83%	2.0%	0.6% - 3.4%	20176	Pass
167	28.750 min.	8.67%	4.6%	1.4% - 7.8%	61797	Fail

Concentration: 1.832 ug/mL



Sample: VX



Segment: 4 Time(min): 23.979-32.984 Scans: 1421-2261 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 15 Oct 2002 10:48:04

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-2,0.91ug10-1

Saturn 2000 No.1

ample ID:	XVX100302-2,0.91ug	Acquisition Date:	10/14/02 10:11 PM
Vial:	3	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	0.913ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42V-EE;PIT@200usec;0.65scans /sec;noPP;m/z268.2;1750V;TS-RAP@170C;Sngl-GN		
Data File:	c:\... \2002\octpber\101402#1\xvx100302-2,0.91ug10-14-02.sms		

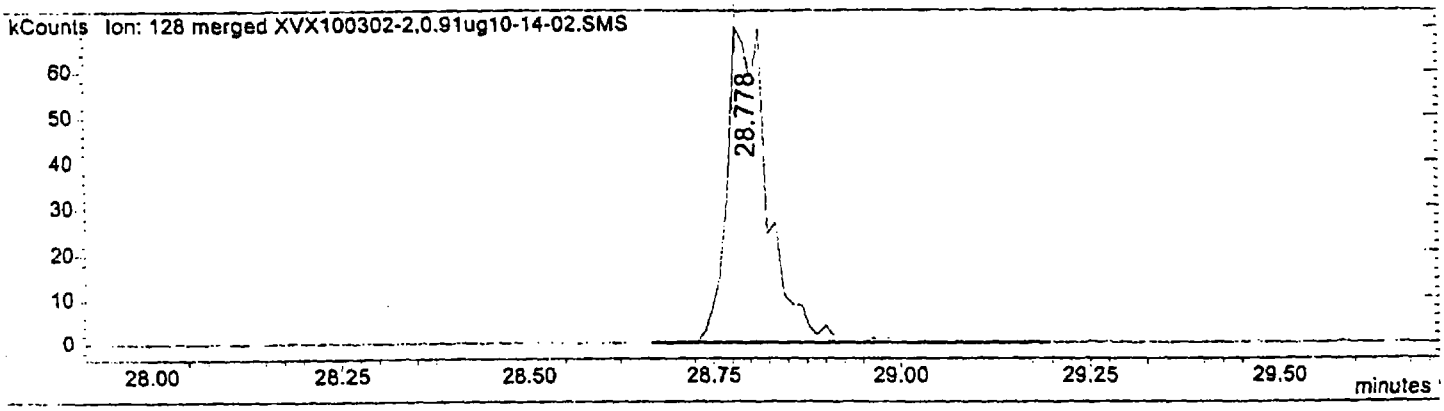
YA C31763 B-A3 10-14-02

Analyte: VX

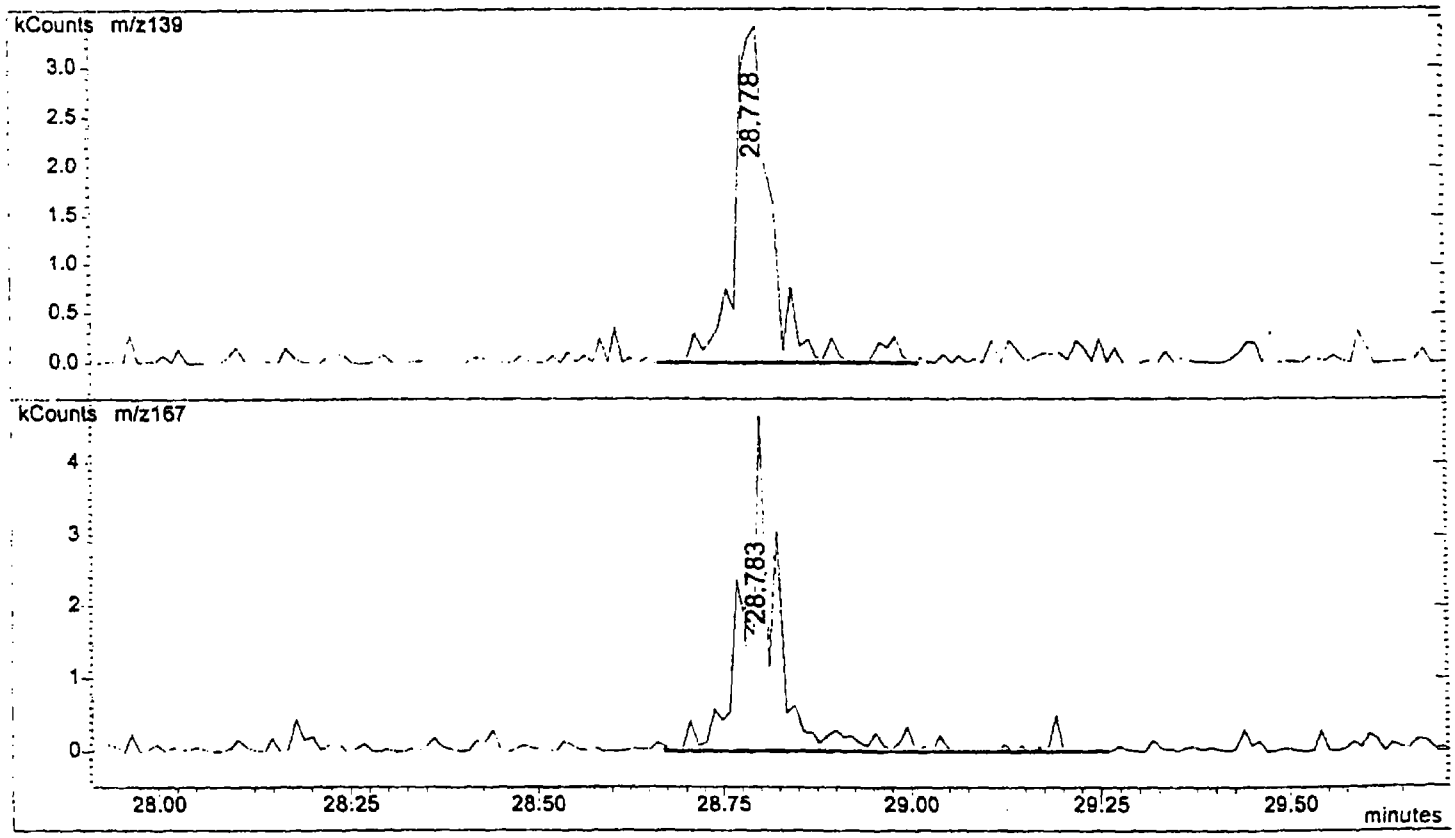
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.778 min.	28.812 min.	28.112 - 29.512 min.	268620	Failed

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.778 min.	4.14%	2.0%	0.6% - 3.4%	11112	Fail
167	28.783 min.	5.06%	4.6%	1.4% - 7.8%	13599	Pass

Concentration: 0.900 ug/mL



Sample: VX



Segment: 4 Time(min): 23.996-32.980 Scans: 1422-2260 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 15 Oct 2002 10:48:16

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

ample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/14/02 10:55 PM
Vial:	4	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \2002\october\101402#1\xvx100302-3,0.46ug10-14-02.sms		

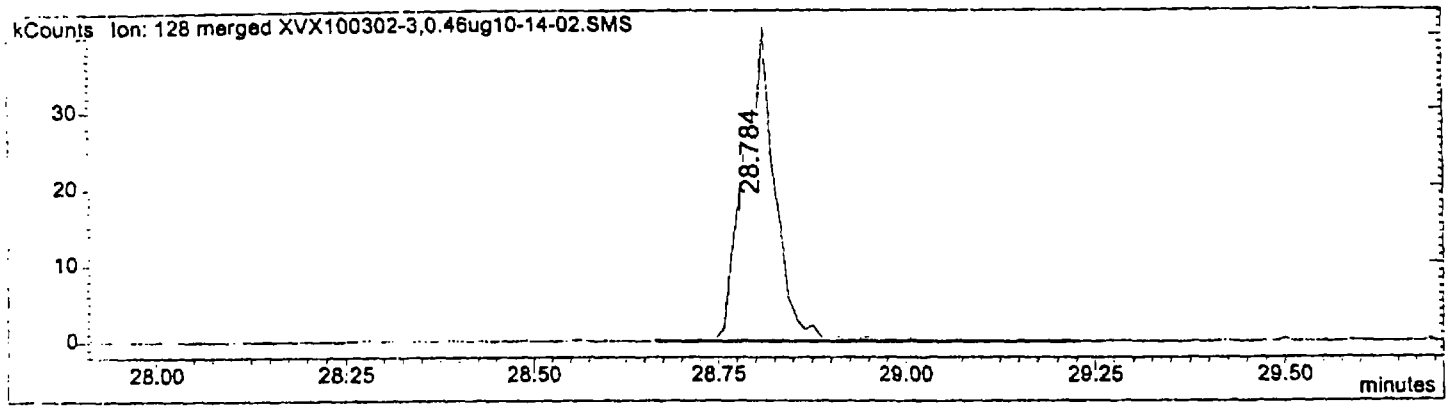
YA
C31763 B-P-3
10-14-02

Analyte: VX

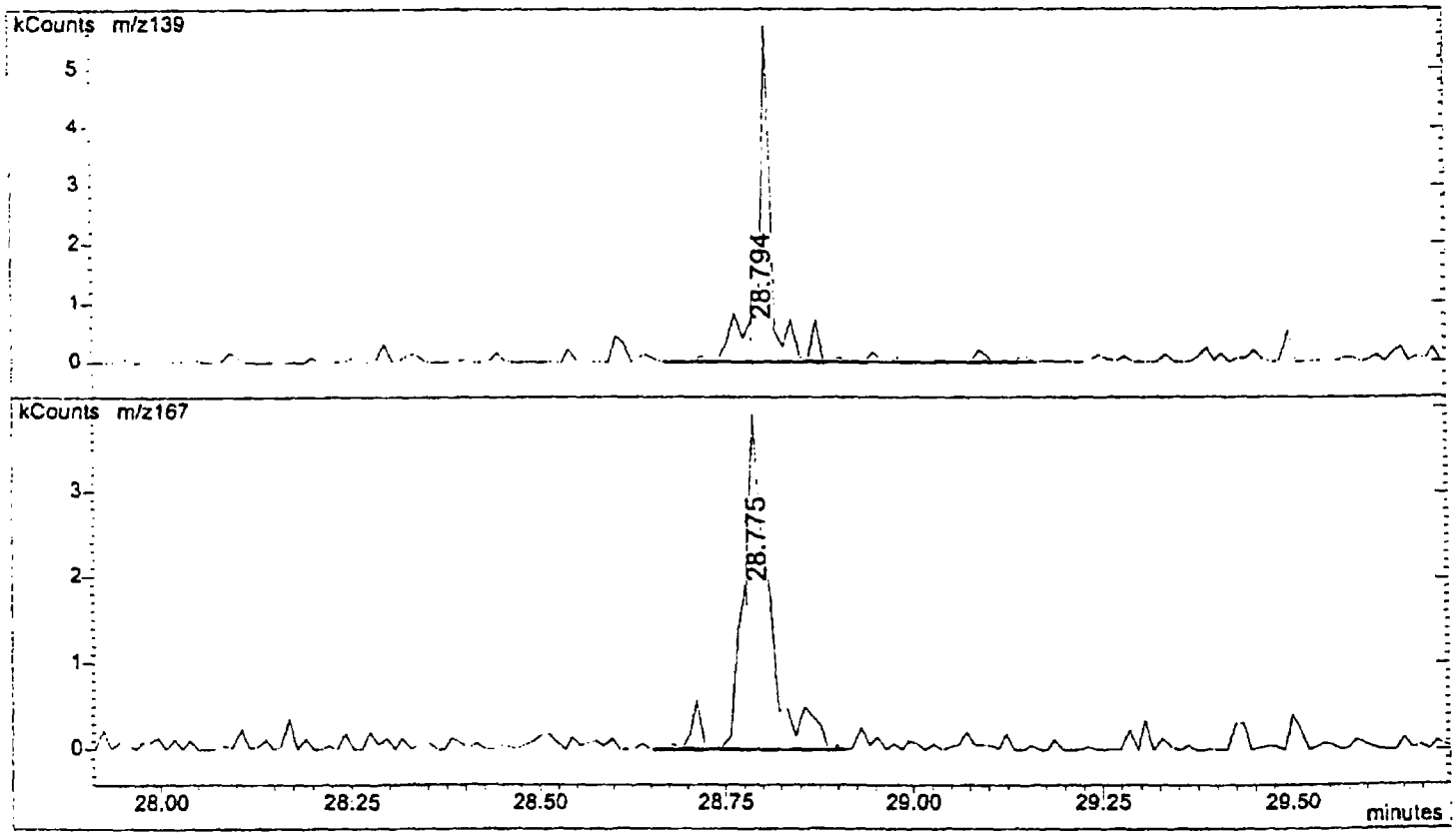
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.784 min.	28.812 min.	28.112 - 29.512 min.	118646	Failed

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.794 min.	6.74%	2.0%	0.6% - 3.4%	8001	Fail
167	28.775 min.	8.01%	4.6%	1.4% - 7.8%	9499	Fail

Concentration: 0.465 ug/mL



Sample: VX



Segment: 4 Time(min): 23.990-32.986 Scans: 1422-2260 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 15 Oct 2002 10:48:28

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-4,0.18ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-4,0.18ug	Acquisition Date:	10/14/02 11:39 PM
Vial:	5	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	0.183ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \2002\octpber\101402#1\xvx100302-4,0.18ug10-14-02.sms		

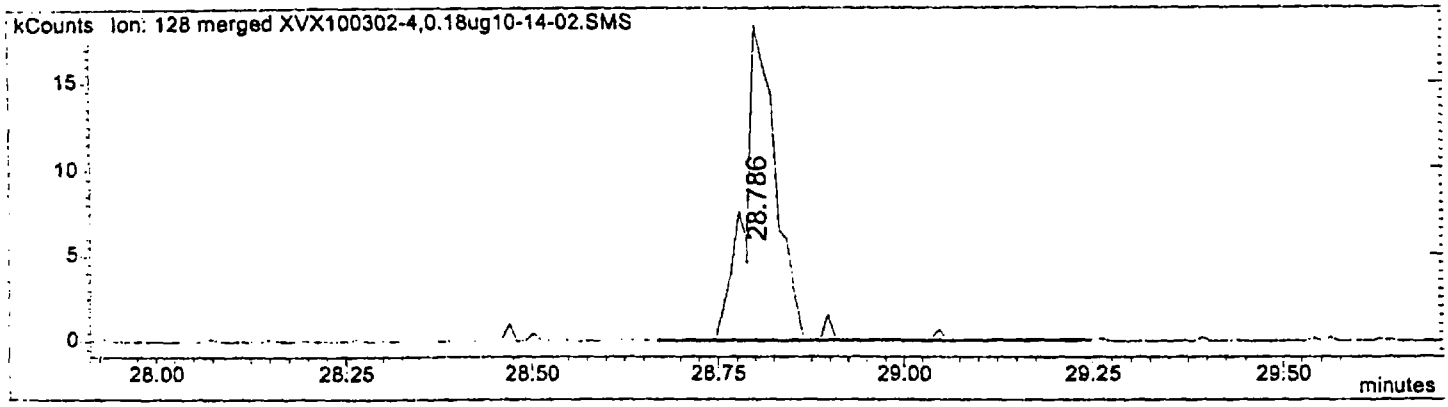
Angela S. Abbott
C31763 bulk
10-14-02

Analyte: VX

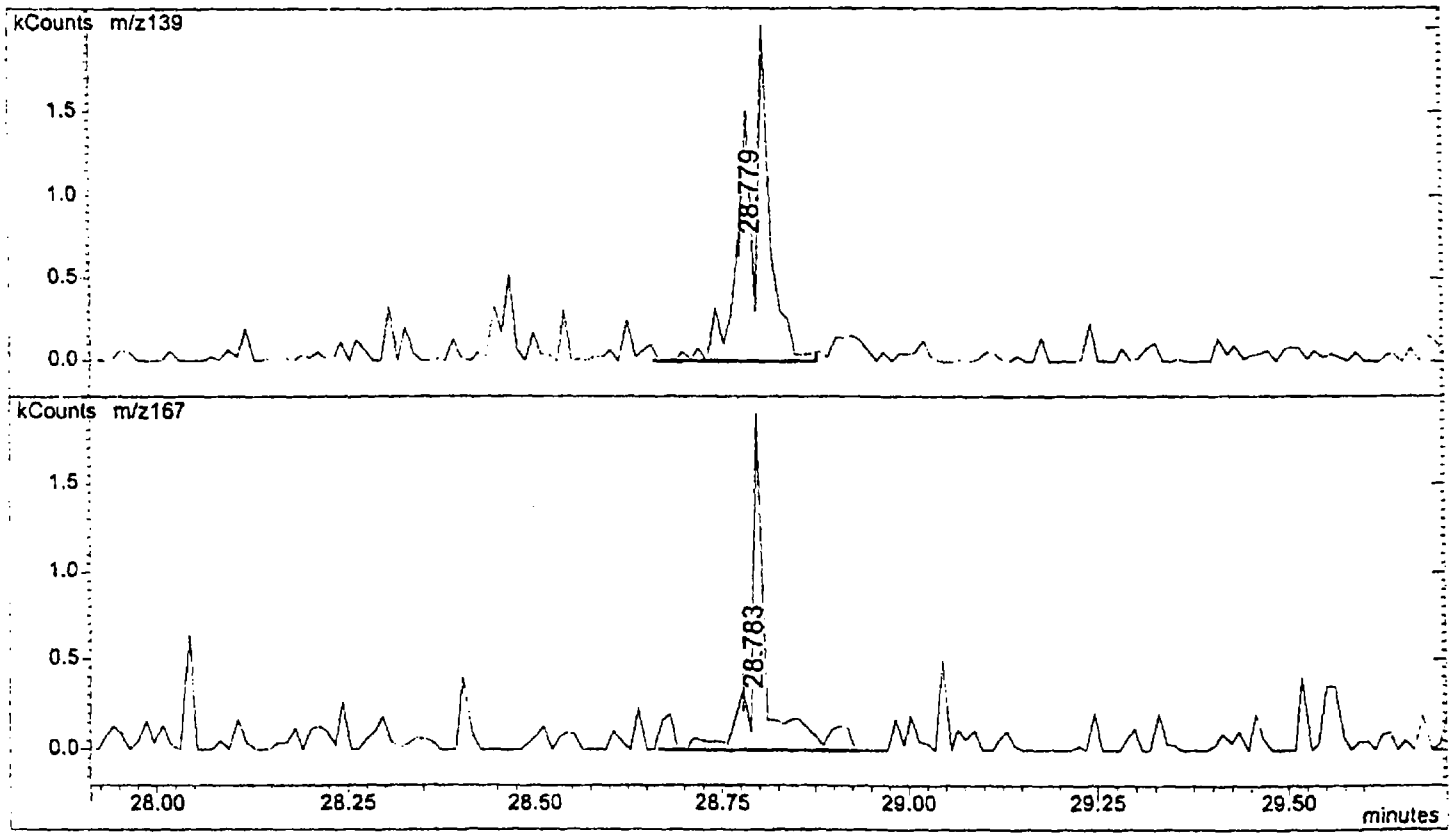
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.786 min.	28.812 min.	28.112 - 29.512 min.	54898	Failed

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.779 min.	7.97%	2.0%	0.6% - 3.4%	4375	Fail
167	28.783 min.	5.35%	4.6%	1.4% - 7.8%	2936	Pass

Concentration: 0.239 ug/mL



Sample: VX



Segment: 4 Time(min): 24.001-32.985 Scans: 1422-2260 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 15 Oct 2002 10:48:39

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-5,.091ug10-1

Saturn 2000 No.1

ample ID:	XVX100302-5,.091ug	Acquisition Date:	10/15/02 12:23 AM
Vial:	6	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	0.091ug/mLVX Std Rtx-5Sil MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \2002\october\101402#1\xvx100302-5,.091ug10-15-02.sms		

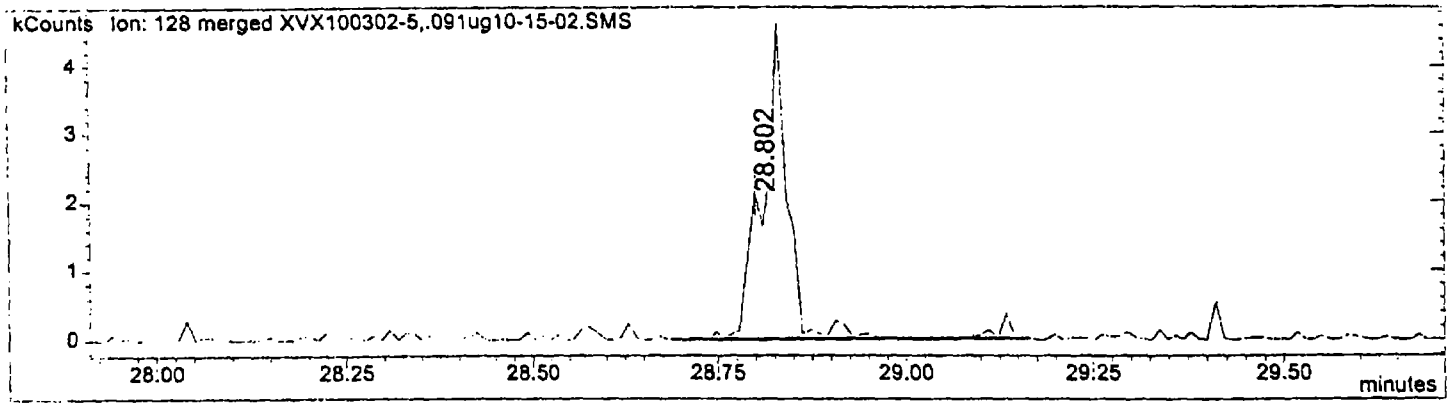
Target SAbb
C31763 Bud
10-14-02

Analyte: VX

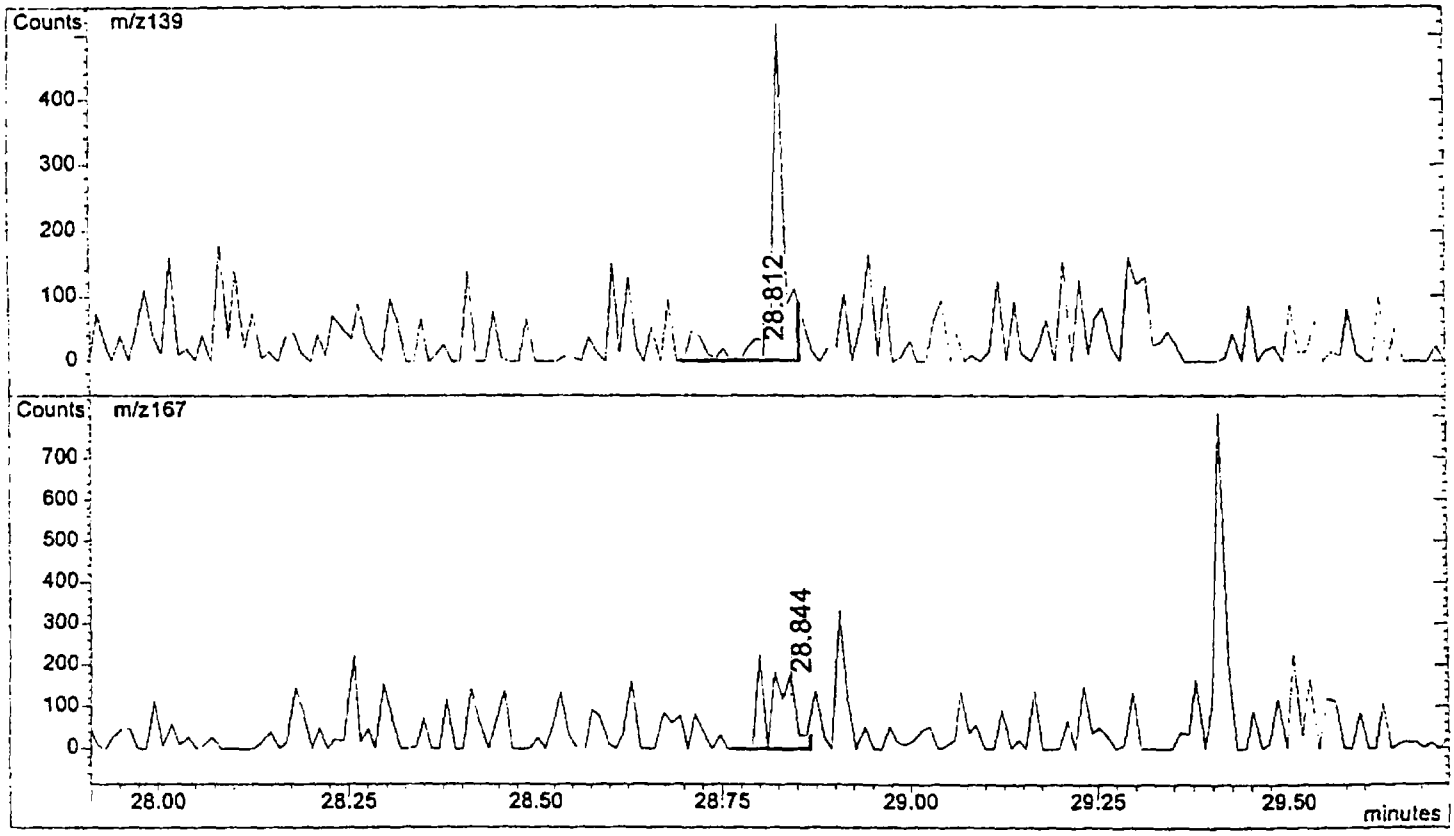
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.802 min.	28.812 min.	28.112 - 29.512 min.	10577	Failed

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.812 min.	5.98%	2.0%	0.6% - 3.4%	633	Fail
167	28.844 min.	4.65%	4.6%	1.4% - 7.8%	492	Pass

Concentration: 0.056 ug/mL



Sample: VX



Segment: 4 Time(min): 23.996-32.982 Scans: 1422-2261 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 15 Oct 2002 10:35:08

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-6,.046ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-6,.046ug	Acquisition Date:	10/15/02 1:06 AM
Vial:	7	Measurement Type:	Area
Method:	c:\saturnws\methods\vx-2002\vx-callb-deaet-10-14-02-m.mth		
Inj. Sample Notes:	0.046ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \2002\october\101402#1\xvx100302-6,.046ug10-15-02.sms		

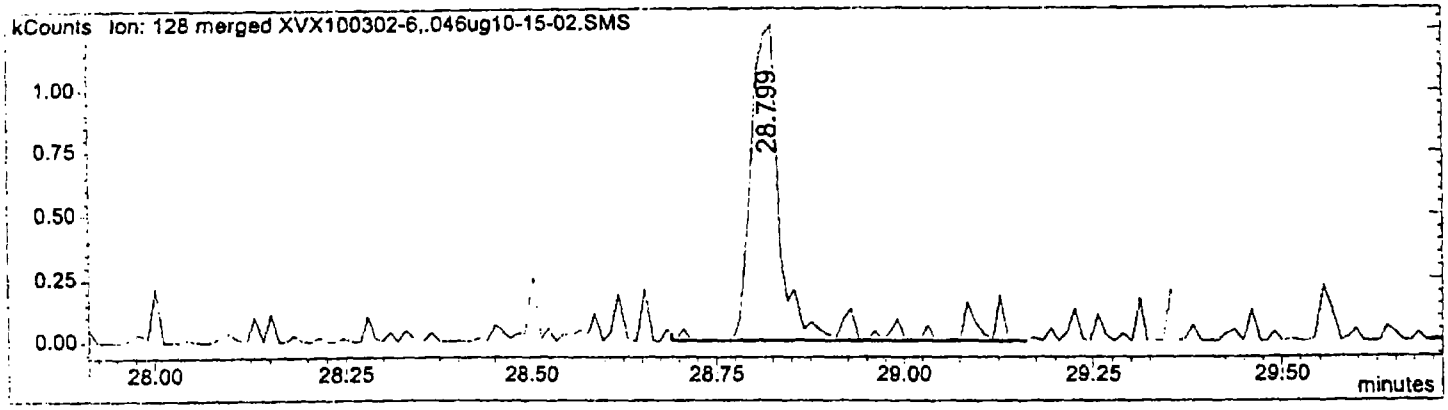
Y. Abraham C31763 Exhibit 10-14-02

Analyte: VX

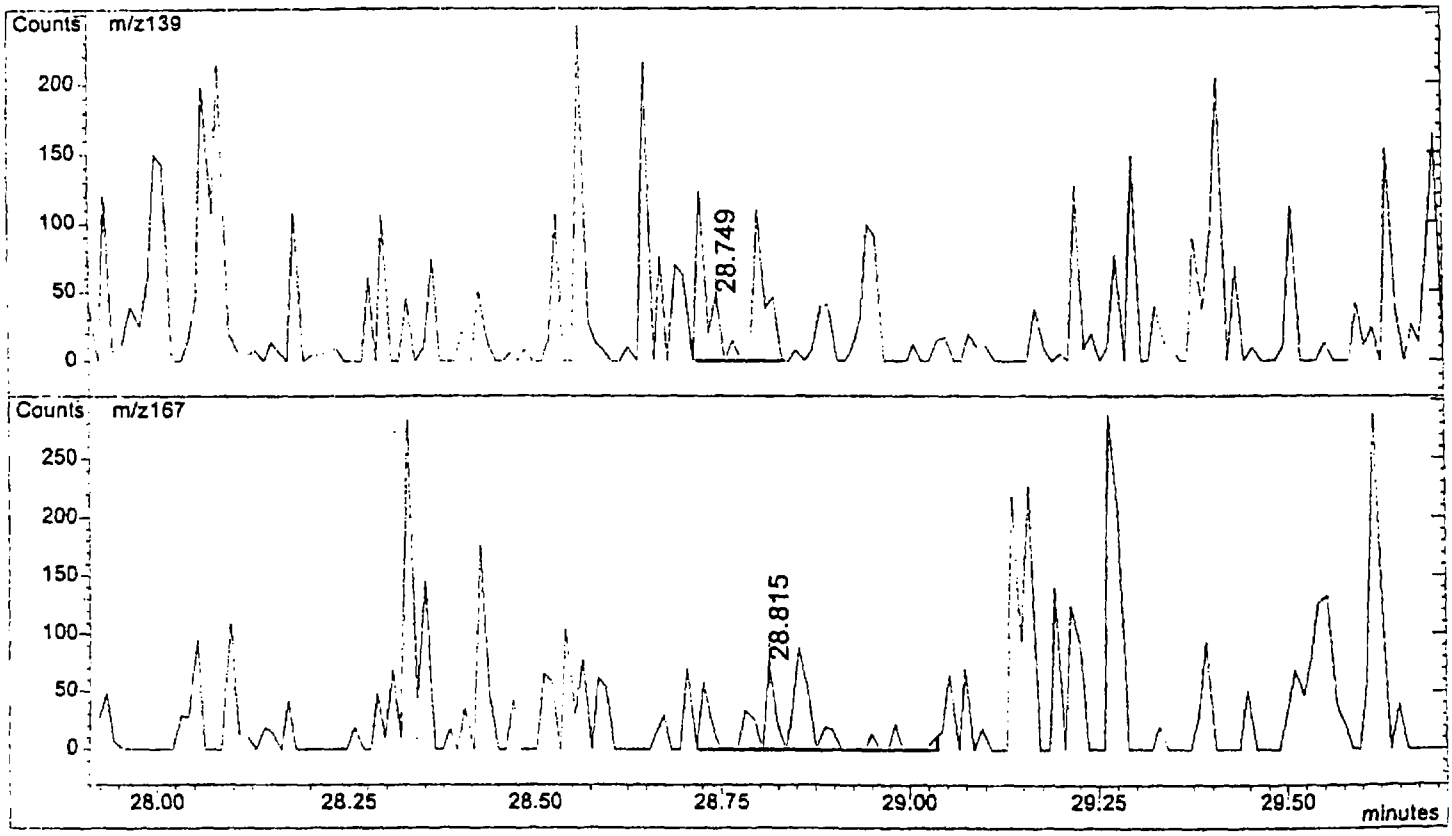
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.799 min.	28.812 min.	28.112 - 29.512 min.	3477	Failed

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.749 min.	7.10%	2.0%	0.6% - 3.4%	247	Fail
167	28.815 min.	9.03%	4.6%	1.4% - 7.8%	314	Fail

Concentration: 0.024 ug/mL



Sample: VX



Segment: 4 Time(min): 23.989-32.987 Scans: 1422-2265 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 15 Oct 2002 10:49:01

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/15/02 1:50 AM
Vial:	4	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \2002\october\101402#1\xvx100302-3,0.46ug10-15-02.sms		

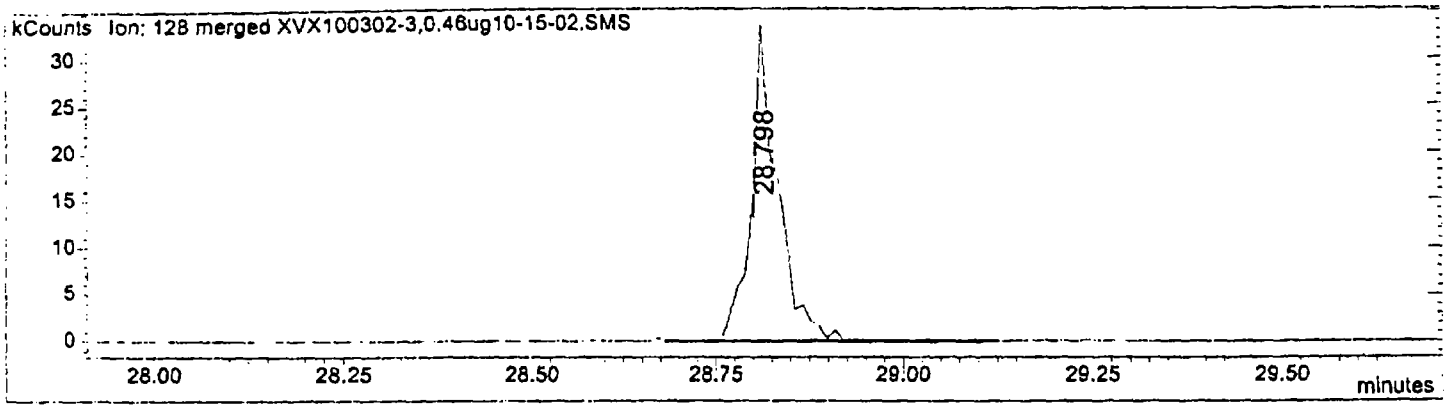
Y. Abrahe
C31763 Back
10-14-02

Analyte: VX

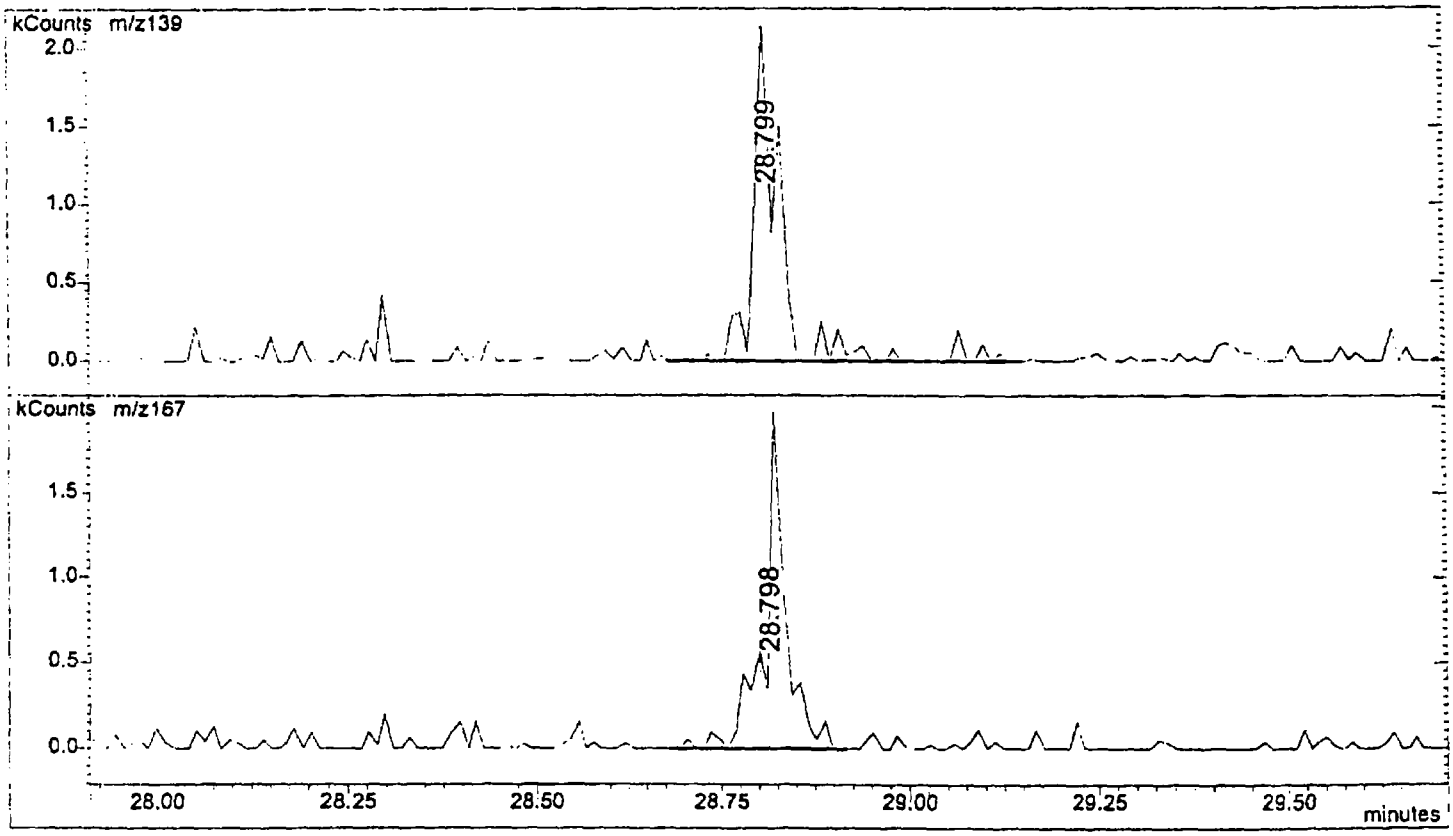
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.798 min.	28.812 min.	28.112 - 29.512 min.	83614	Failed

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.799 min.	5.27%	2.0%	0.6% - 3.4%	4403	Fail
167	28.798 min.	4.64%	4.6%	1.4% - 7.8%	3882	Pass

Concentration: 0.345 ug/mL



Sample: VX



Segment: 4 Time(min): 23.994-32.984 Scans: 1422-2261 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 15 Oct 2002 10:49:17

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-1, 1.8ug10-1

Saturn 2000 No.1

ample ID:	XVX100302-1, 1.8ug	Acquisition Date:	10/14/02 8:43 PM
Vial:	2	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	1.83ug/mLVX Std, Rtx-5MS:30M x 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans /sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \2002\octqber\101402#1\xvx100302-1, 1.8ug10-14-02.sms		

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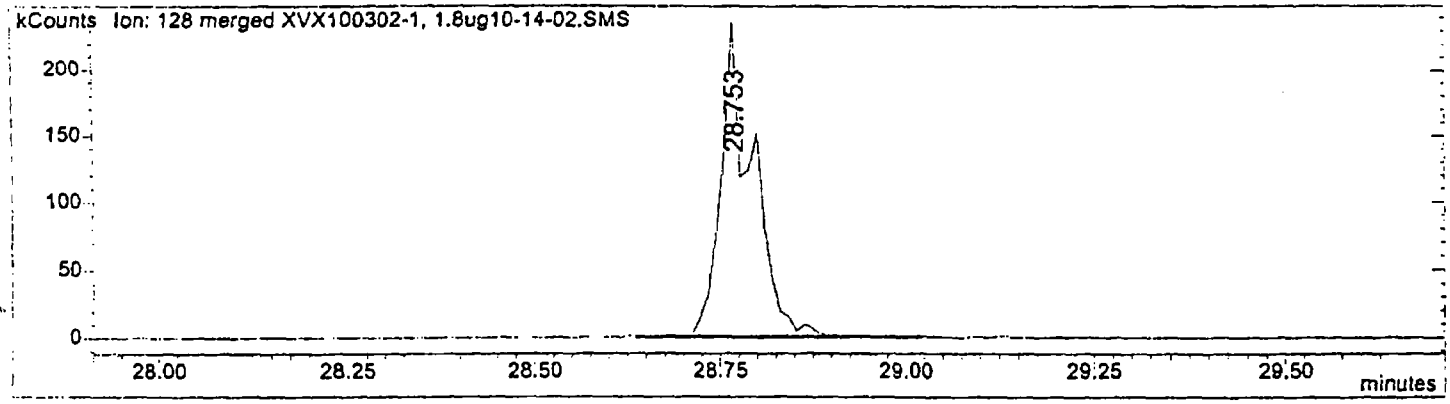
C31763
bad P-3
10-14-02

Analyte: VX

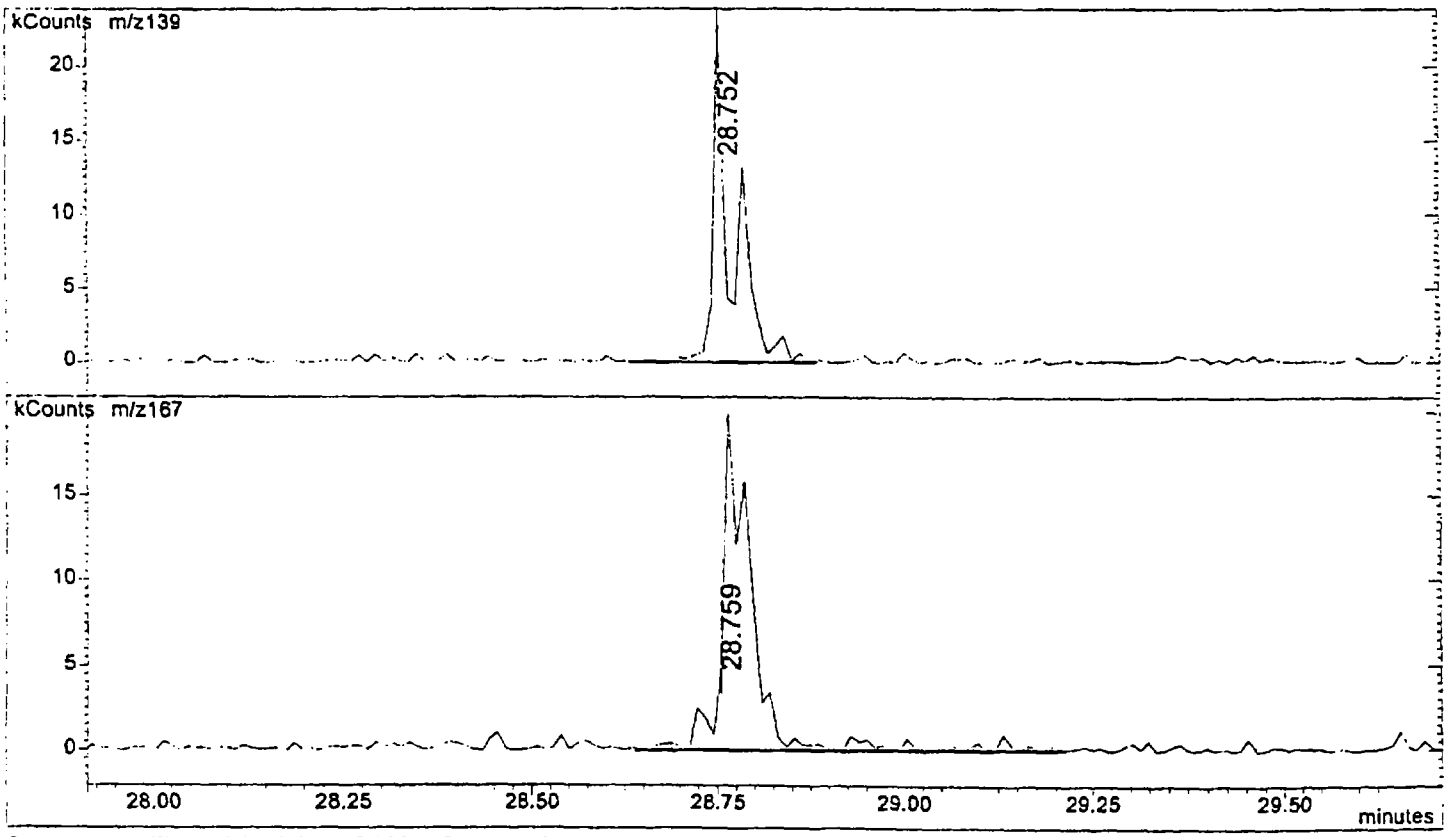
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.753 min.	28.812 min.	28.112 - 29.512 min.	707507	Failed

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.752 min.	5.82%	2.0%	0.6% - 3.4%	41155	Fail
167	28.759 min.	7.33%	4.6%	1.4% - 7.8%	51830	Pass

Concentration: 1.822 ug/mL



Sample: VX



Segment: 4 Time(min): 23.996-32.982 Scans: 1421-2259 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 15 Oct 2002 10:49:30

Ion Ratio Report for Target Compound #1 from vx-101402-us-1-10-15-02.sms

Saturn 2000 No.1

Sample ID:	VX-101402-US-1	Acquisition Date:	10/15/02 2:34 AM
Trial:	8	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	Methylene Chloride/IPA Sonicator Blank + DEAET;Rtx5MS;S-TRAP ;EM@1750V		
Data File:	c:\... \2002\october\101402#1\vx-101402-us-1-10-15-02.sms		

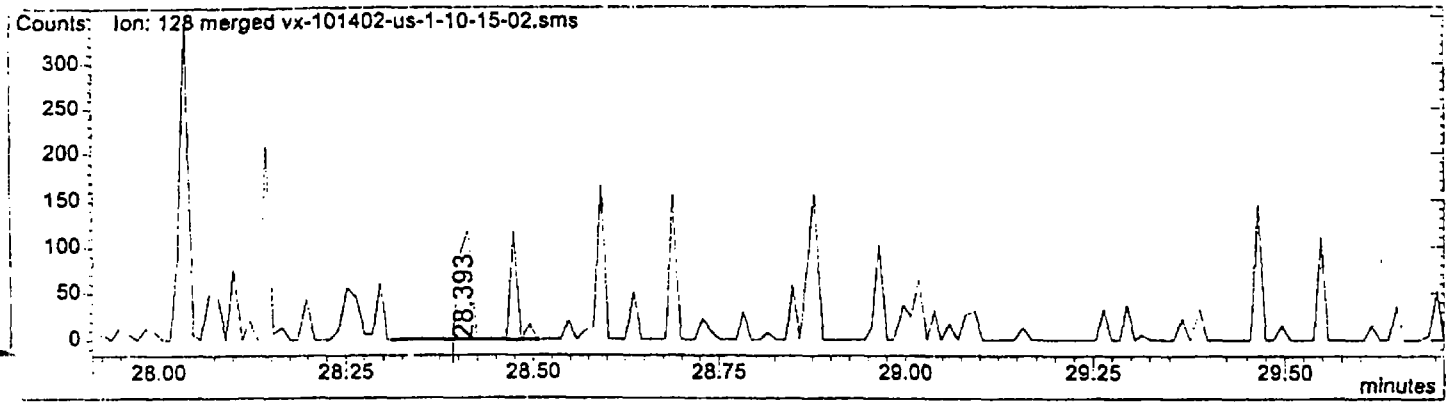
Yiqz S Abdullah
C31763 Rev R-3
10-14-02

Analyte: VX

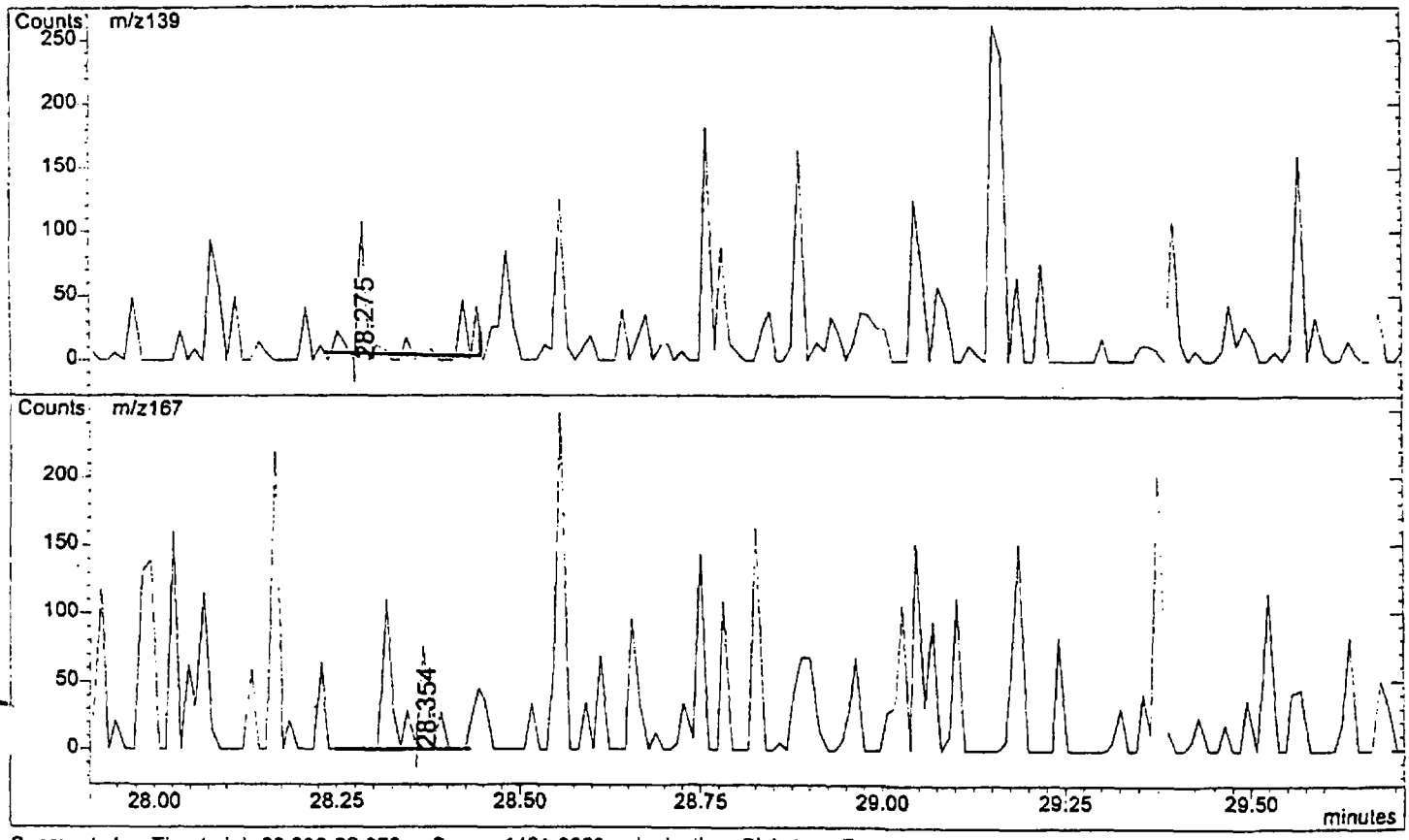
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.393 min.	28.812 min.	28.112 - 29.512 min.	135	Failed

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.275 min.	88.91%	2.0%	0.6% - 3.4%	120	Fail
167	28.354 min.	131.45%	4.6%	1.4% - 7.8%	178	Fail

Concentration: 0.009 ug/mL



Sample: VX



Segment: 4 Time(min): 23.996-32.979 Scans: 1421-2263 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 15 Oct 2002 10:49:43

Ion Ratio Report for Target Compound #1 from vx-101402-us-2-10-15-02.sms

Saturn 2000 No.1

Sample ID:	VX-101402-US-2	Acquisition Date:	10/15/02 3:18 AM
Vial:	9	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	Methylene Chloride/IPA Sonicator Blank + DEAET;Rtx5MS;S-TRAP ;EM@1750V		
Data File:	c:\... \2002\october\101402#1\vx-101402-us-2-10-15-02.sms		

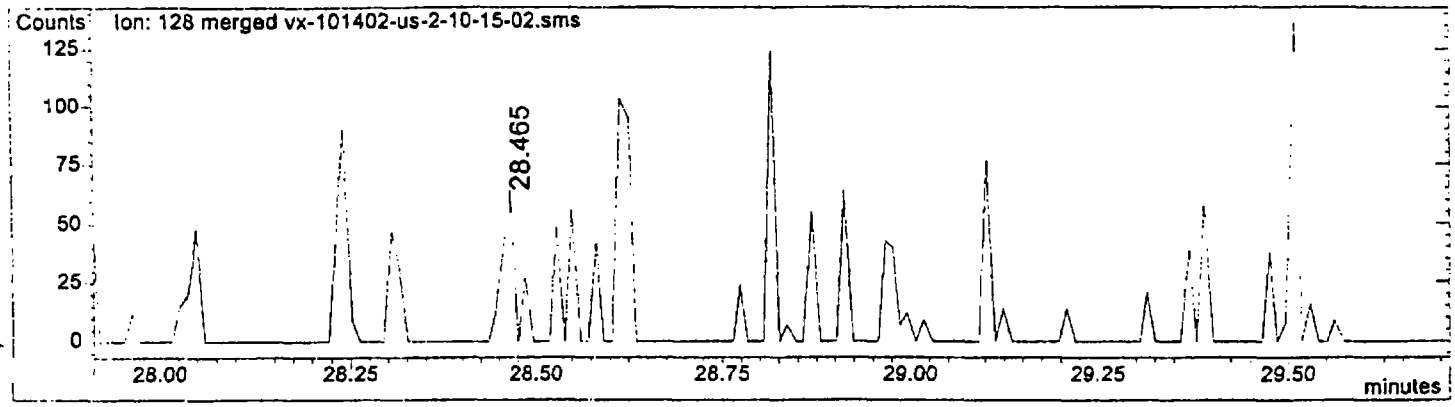
C31763 P-3
10-14-02

Analyte: VX

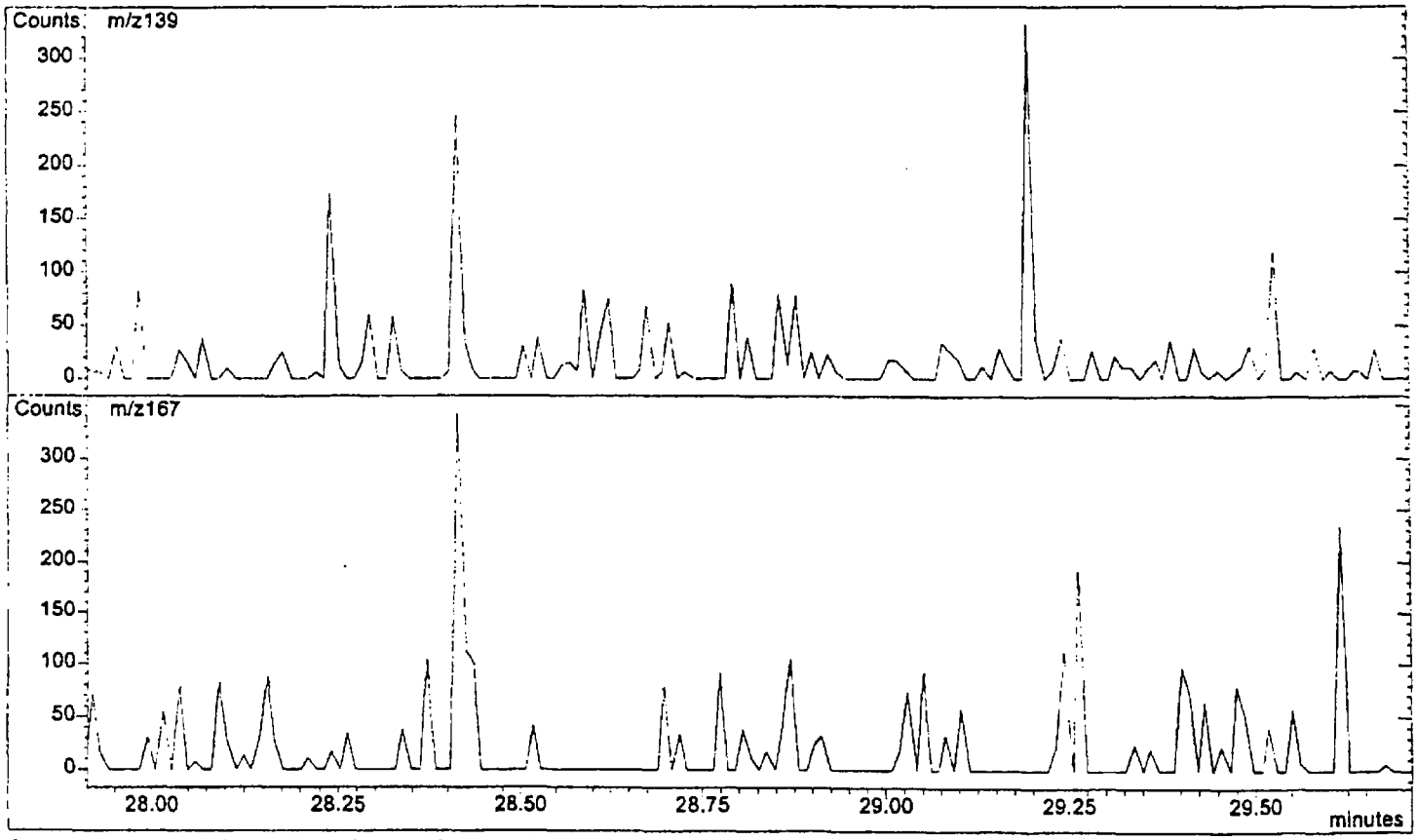
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.465 min.	28.812 min.	28.112 - 29.512 min.	0	Missing

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	0.000 min.	0.00%	2.0%	0.6% - 3.4%	0	Fail
167	0.000 min.	0.00%	4.6%	1.4% - 7.8%	0	Fail

Concentration: 0.000 ug/mL



Sample: VX



Segment: 4 Time(min): 23.989-32.977 Scans: 1421-2264 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 15 Oct 2002 10:50:04

Ion Ratio Report for Target Compound #1 from vx-101402-us-3-10-15-02.sms

Saturn 2000 No.1

Sample ID:	VX-101402-US-3	Acquisition Date:	10/15/02 4:02 AM
Vial:	10	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	Method Spike:457ug/mL VX Spike into 5g NaSO4 + DEAET; Rtx5MS ;S-TRAP;EM@1750V		
Data File:	c:\... \2002\october\101402#1\vx-101402-us-3-10-15-02.sms		

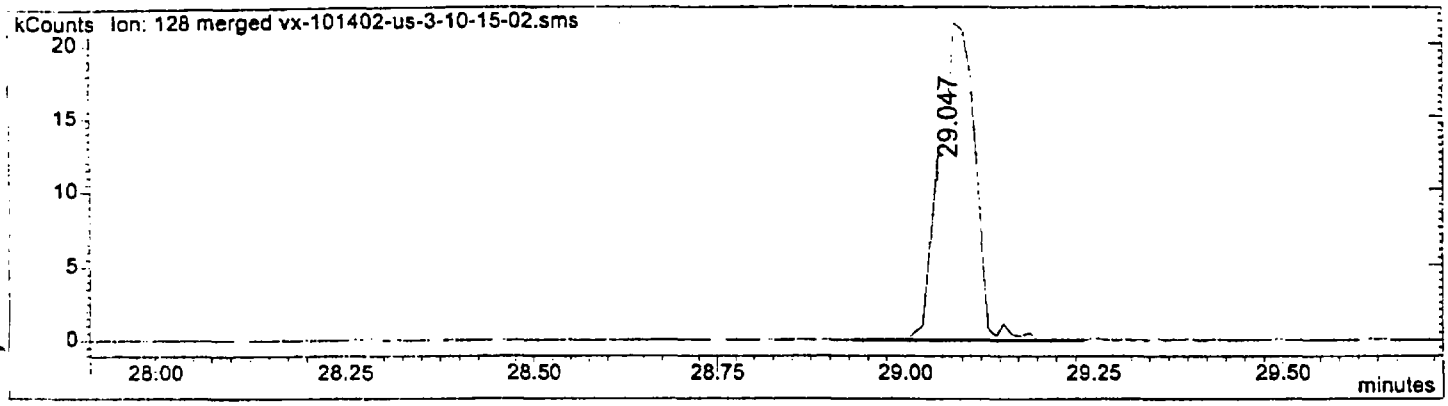
Y. Arthur
C31763 Backf-3
10-14-02

Analyte: VX

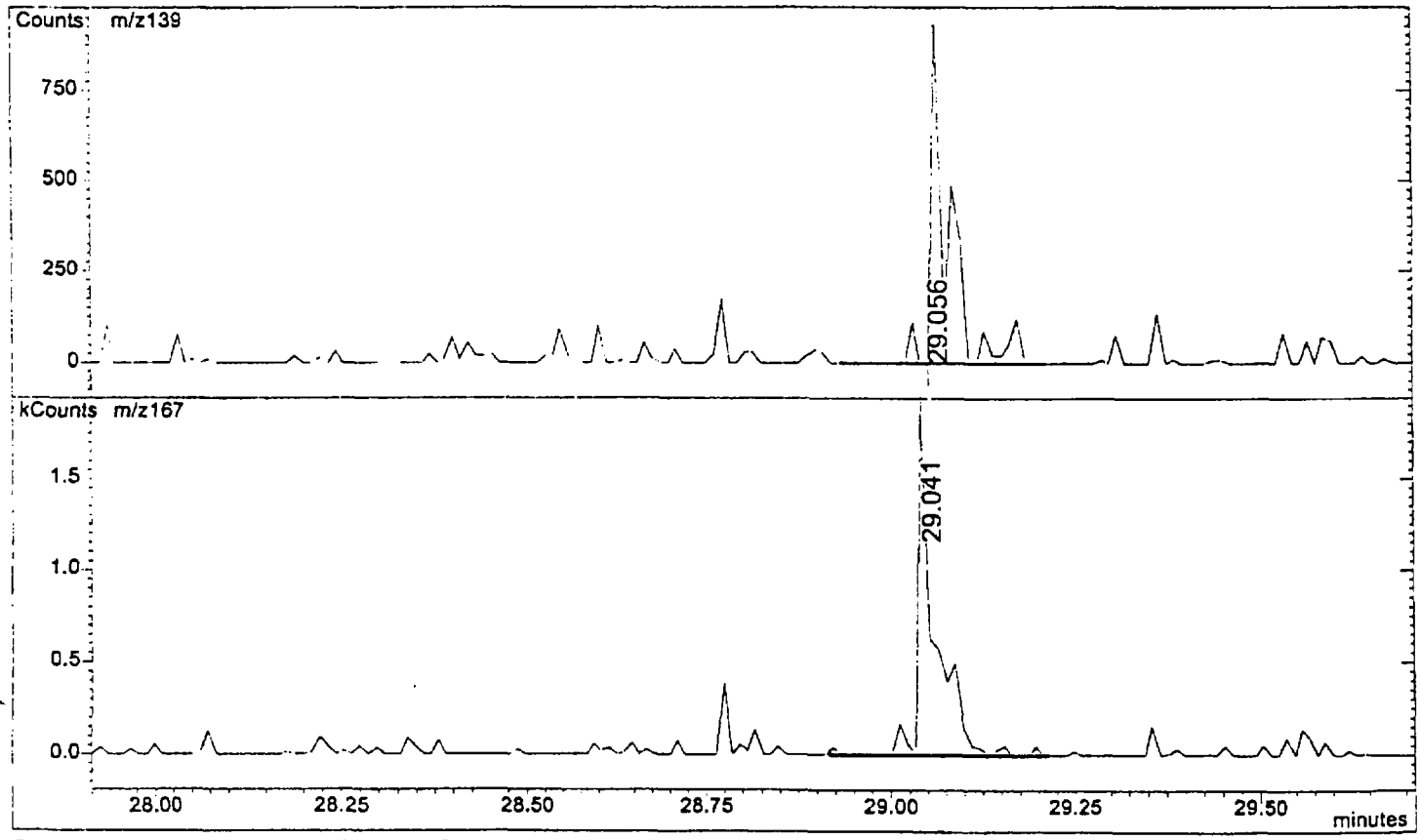
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	29.047 min.	28.812 min.	28.112 - 29.512 min.	69669	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	29.056 min.	1.92%	2.0%	0.6% - 3.4%	1340	Pass
167	29.041 min.	4.28%	4.6%	1.4% - 7.8%	2985	Pass

Concentration: 0.295 ug/mL



Sample: VX



Segment: 4 Time(min): 23.979-32.978 Scans: 1421-2265 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 15 Oct 2002 10:50:20

Ion Ratio Report for Target Compound #1 from vx-101402-us-4-10-15-02.sms

Saturn 2000 No.1

Sample ID:	VX-101402-US-4	Acquisition Date:	10/15/02 4:46 AM
ial:	11	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	Method Spike:457ug/mL VX Spike into 5g NaSO4 + DEAET; Rtx5MS ;S-TRAP;EM@1750V		
Data File:	c:\... \2002\october\101402#1\vx-101402-us-4-10-15-02.sms		

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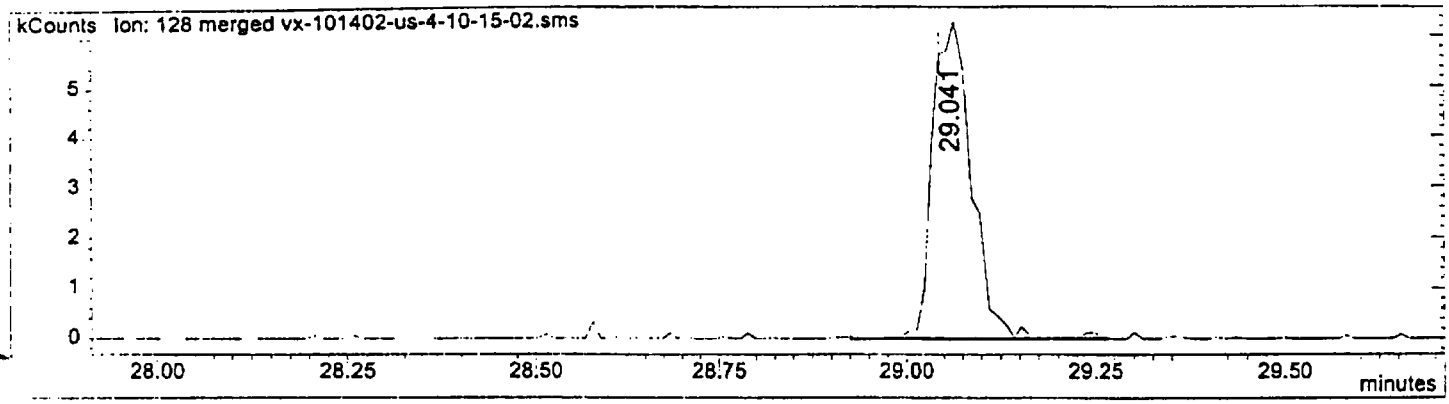
C 31763 book P-3
10-14-02

Analyte: VX

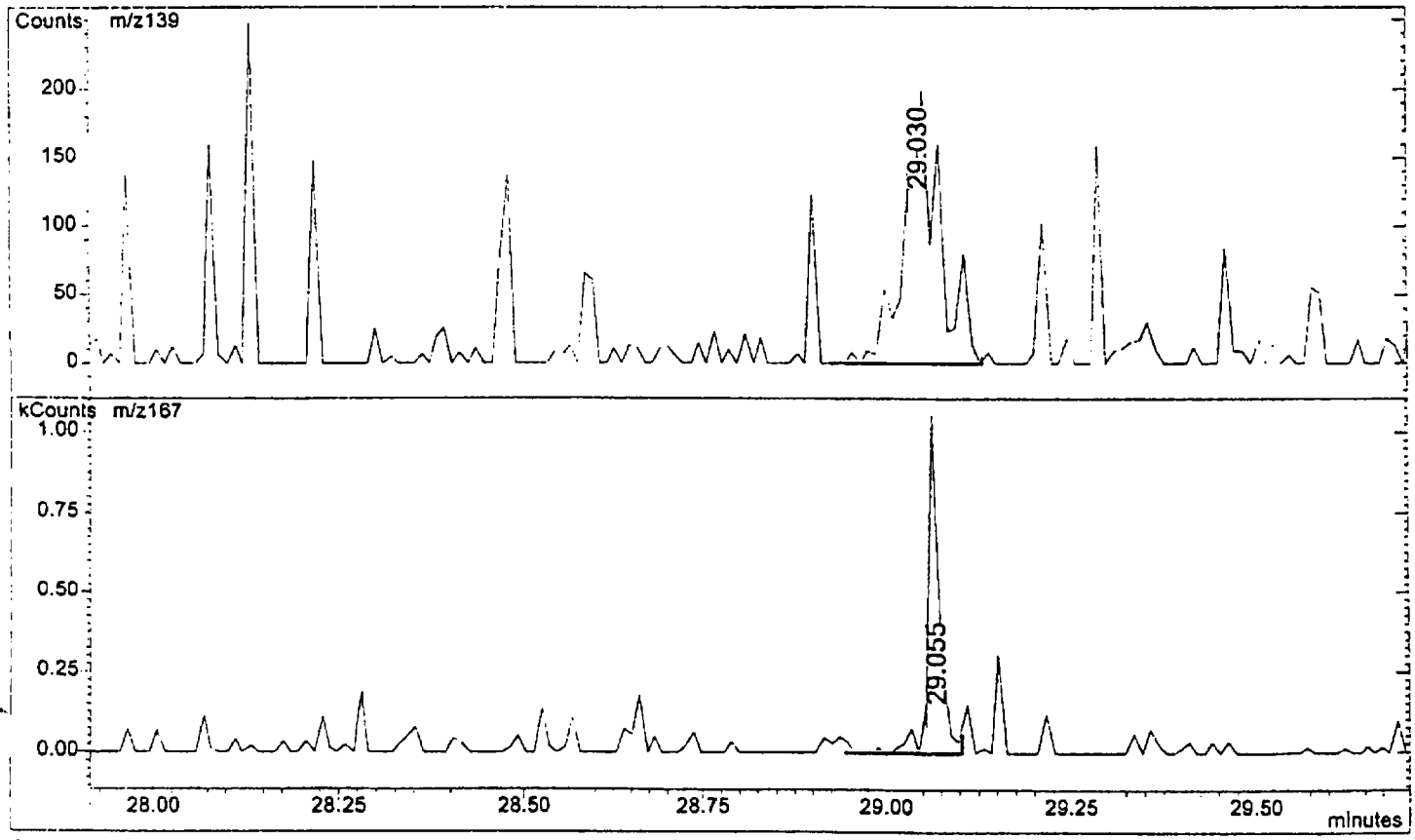
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	29.041 min.	28.812 min.	28.112 - 29.512 min.	22673	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	29.030 min.	2.84%	2.0%	0.6% - 3.4%	644	Pass
167	29.055 min.	5.04%	4.6%	1.4% - 7.8%	1144	Pass

Concentration: 0.109 ug/mL



Sample: VX



Segment: 4 Time(min): 23.994-32.978 Scans: 1421-2265 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 15 Oct 2002 10:50:30

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/15/02 5:29 AM
ial:	4	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \october\101402#1\xvx100302-3,0.46ug10-15-02001.sms		

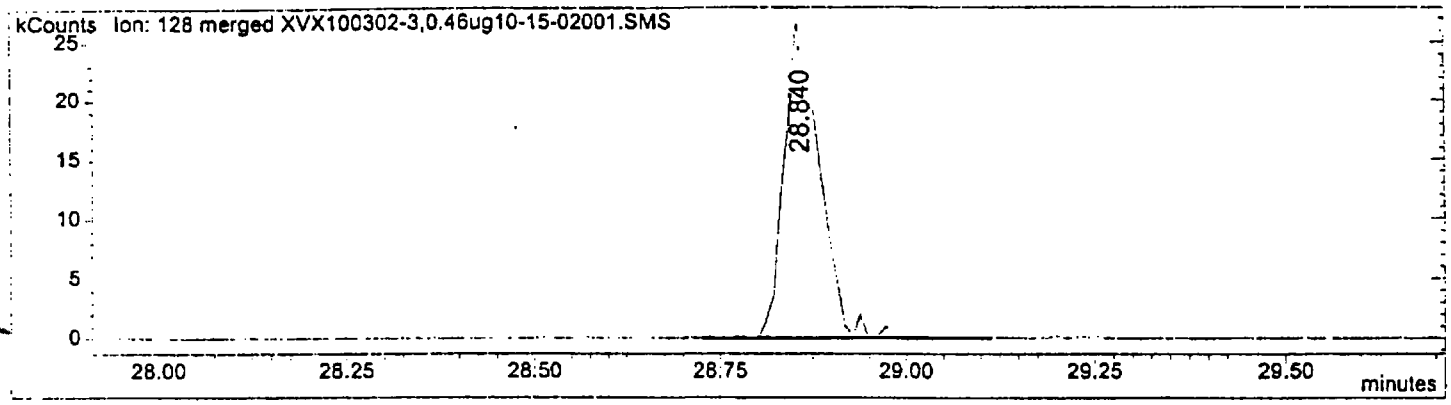
Y. Abraha
C31763 lot P-3
10-14-02

Analyte: VX

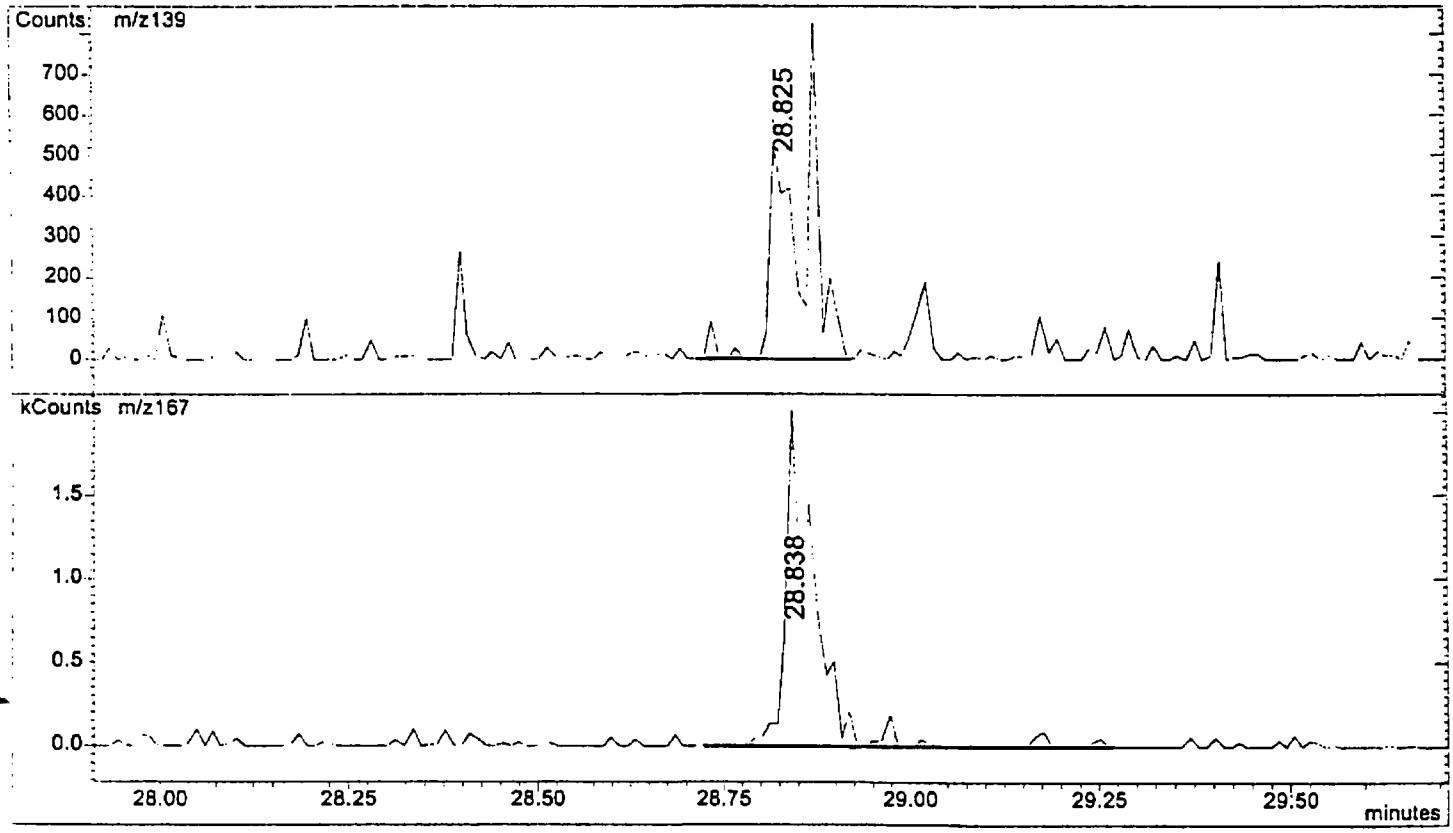
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.840 min.	28.812 min.	28.112 - 29.512 min.	86185	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.825 min.	2.27%	2.0%	0.6% - 3.4%	1959	Pass
167	28.838 min.	5.70%	4.6%	1.4% - 7.8%	4908	Pass

Concentration: 0.355 ug/mL



Sample: VX



Segment: 4 Time(min): 23.989-32.984 Scans: 1422-2269 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 15 Oct 2002 10:51:06

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/15/02 5:29 AM
al:	4	Measurement Type:	Area
ethod:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \october\101402#1\xvx100302-3,0.46ug10-15-02001.sms		

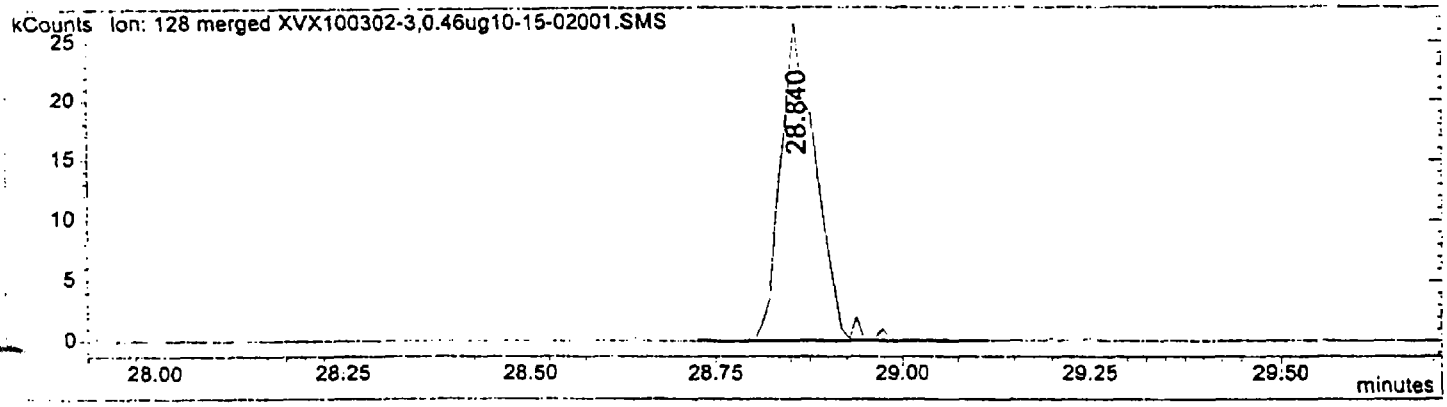
Y. Abraha
C 31763 back p. 3
10-14-02

Analyte: VX

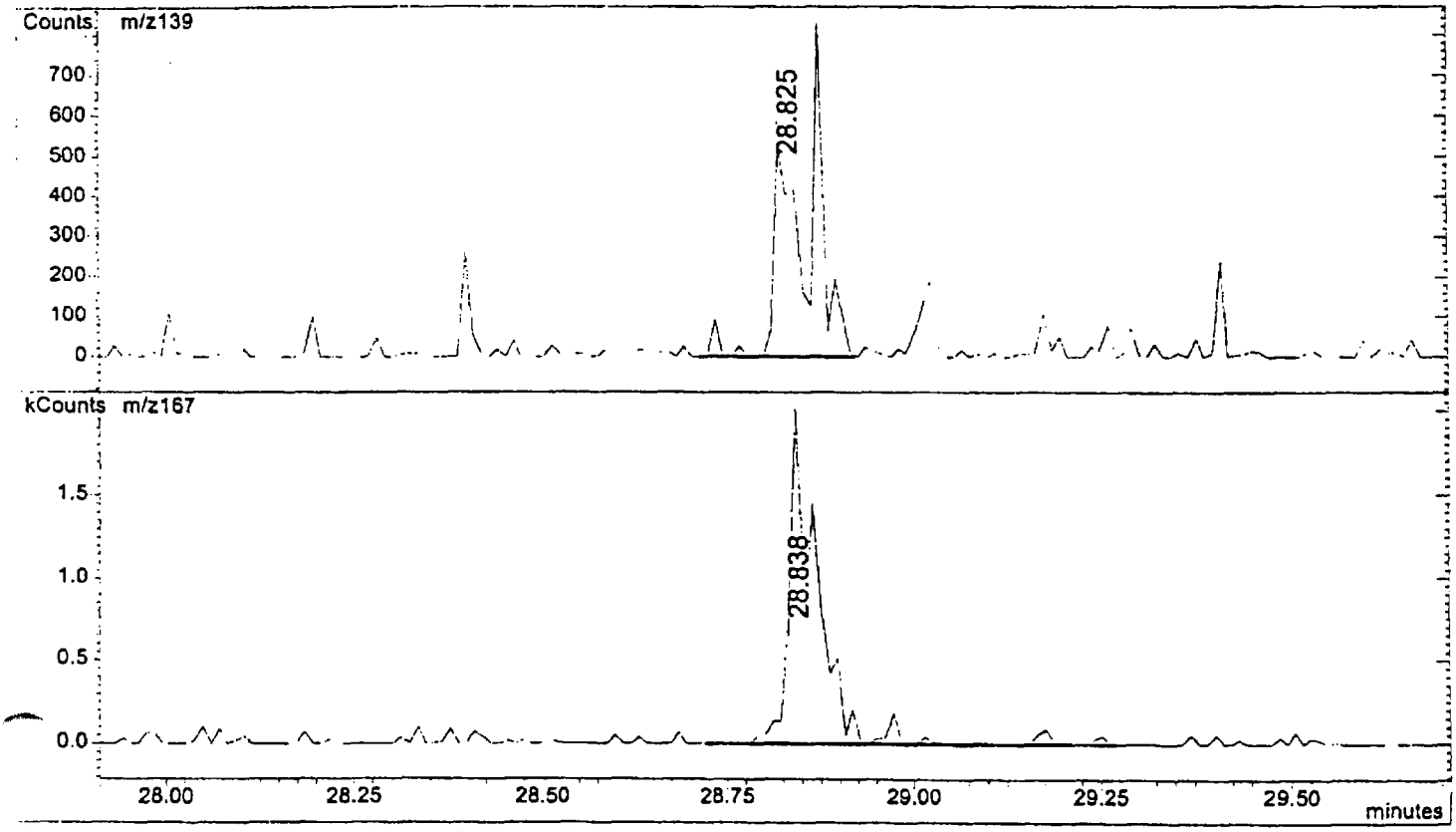
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.840 min.	28.812 min.	28.112 - 29.512 min.	86185	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.825 min.	2.27%	2.0%	0.6% - 3.4%	1959	Pass
167	28.838 min.	5.70%	4.6%	1.4% - 7.8%	4908	Pass

Concentration: 0.355 ug/mL



Sample: VX



Segment: 4 Time(min): 23.989-32.984 Scans: 1422-2269 Ionization: CI Auto Preparation: MS/MS Channels: 1

YA
C31763 back P-6
10-15-02

Varian Star Workstation - 8400 SampleList Tue Oct 15 19:13:52 2002

SampleList: C:\saturnws\Data\2002\October\10150281\VX10150281.smp

: Tue Oct 15 10:35:26 2002
ed: Tue Oct 15 19:13:47 2002

Line	Sample Name	Injection Notes	AutoLink	Vial	Inject Volume
1			C:\saturnws\Methods\VX-2002\VX092502-m2268.2-0.42V-PIT-200-ST-0.65-noPP-Purge020+EI;Mult1750v+300v.mth		
2	XVX-050902-6,spk6-	below	none	1	1.0
	Notes XVX-11-07-01A,spike,.046ug/mL VX; HAE extract:Rtx-5MS:30Mx0.25mmID.1um;				
	Notes 100C,5min-240C@5C/m-280C@25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2,1750V;S-TRAP@170				
3	XVX100302-3,0.46ug	below	none	2	1.0
	Notes 0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
4			C:\saturnws\Methods\VX-2002\VX092502-m2268.2-0.42V-PIT-200-ST-0.65-noPP-Purge020+EI;Mult1750v+300v+UDInj.mth		
5	VX-101502-US-1-	below	none	3	1.0
	Notes Method Spike: 91ng/g VX Spike into 5g NaSO4 + DEAET; RtxSMS;S-TRAP;EM@1750V				
6	VX-101502-US-2-	below	none	4	1.0
	Notes Method Spike: 91ng/g VX Spike into 5g NaSO4 + DEAET; RtxSMS;S-TRAP;EM@1750V				
7	VX-101502-US-3-	below	none	5	1.0
	Notes JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultasonic Extraction, no HAE cleanup;				
	Notes RtxSMS;S-TRAP;EM@1750V				
8	VX-101502-US-4-	below	none	6	1.0
	Notes JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultasonic Extraction, no HAE cleanup;				
	Notes RtxSMS;S-TRAP;EM@1750V				
9	XVX100302-3,0.46ug	below	none	2	1.0
	Notes 0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
10			C:\saturnws\Methods\Standby.mth		

Directory for Data Files: c:\saturnws\data\2002\october\10150281

Data File Names: %s%d

Create and update a new RecalcList: c:\saturnws\data\2002\october\10150281\vx101502-1.rcl.

****

Varian Star Workstation - RecalcList Wed Oct 16 11:07:31 2002

.cList: C:\saturnws\Data\2002\October\101502#1\VX101502-1.RCL

Created: Wed Oct 16 10:38:48 2002
Modified: Wed Oct 16 11:06:53 2002

****

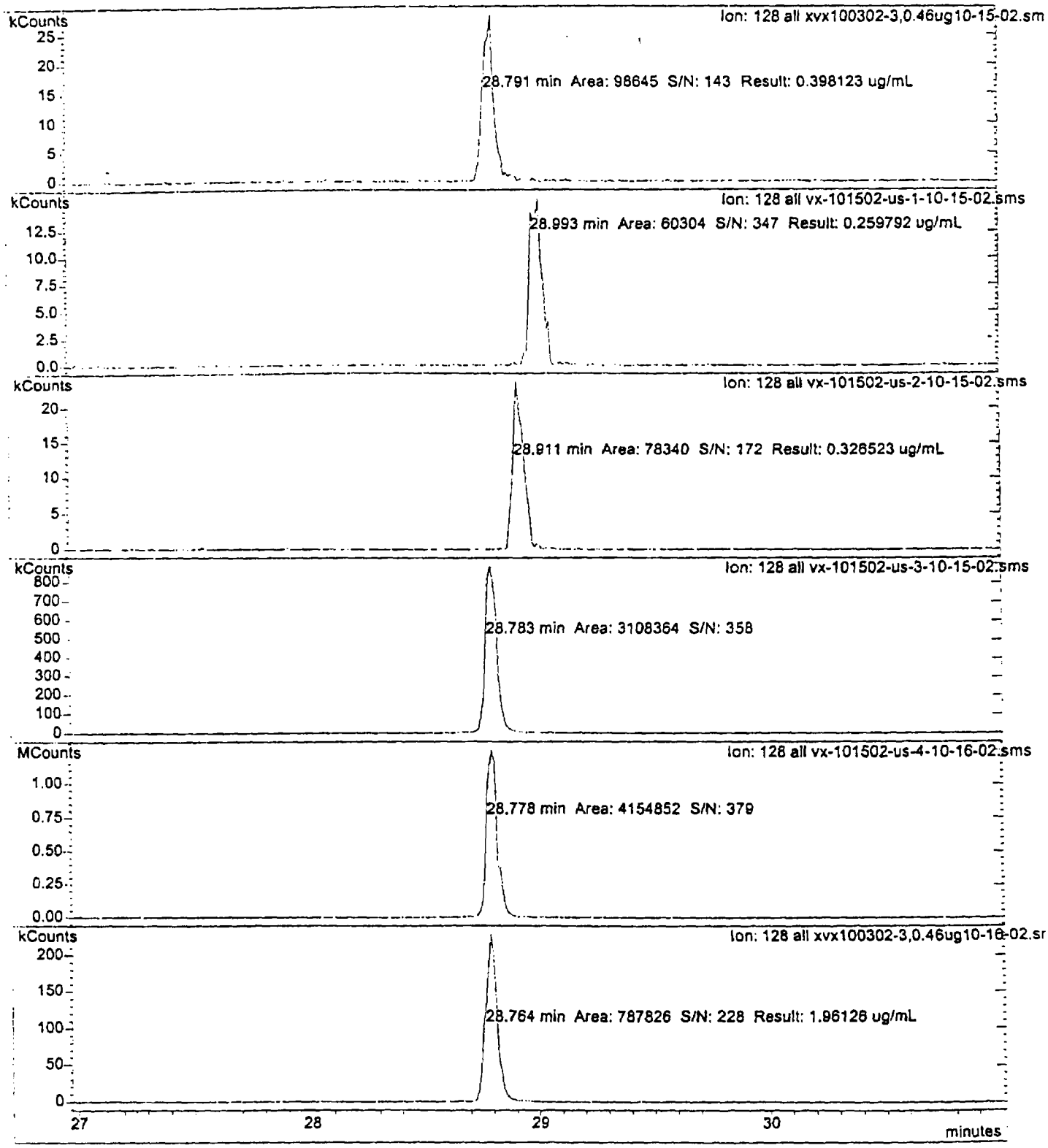
Line	Sample Type	Sample Name	Data File
1	Analysis	XVX-050902-6,spk6-	XVX-050902-6,spk6-10-15-02.SMS
2	Analysis	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-15-02.SMS
3	Analysis	VX-101502-US-1-	VX-101502-US-1-10-15-02.SMS
4	Analysis	VX-101502-US-2-	VX-101502-US-2-10-15-02.SMS
5	Analysis	VX-101502-US-3-	VX-101502-US-3-10-15-02.SMS
6	Analysis	VX-101502-US-4-	VX-101502-US-4-10-16-02.SMS
7	Analysis	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-16-02.SMS

Print Date: 16 Oct 2002 11:11:24

Y. Arthur
C31763 book P-6
10-15-02

Chromatogram Plots

Plot 1: c:\... \101502#1\xvx100302-3,0.46ug10-15-02.sms Ion: 128 all
Plot 2: c:\... \101502#1\vx-101502-us-1-10-15-02.sms Ion: 128 all
Plot 3: c:\... \101502#1\vx-101502-us-2-10-15-02.sms Ion: 128 all
Plot 4: c:\... \101502#1\vx-101502-us-3-10-15-02.sms Ion: 128 all
Plot 5: c:\... \101502#1\vx-101502-us-4-10-16-02.sms Ion: 128 all
Plot 6: c:\... \101502#1\xvx100302-3,0.46ug10-16-02.sms Ion: 128 all



Print Date: 16 Oct 2002 11:08:01

Ion Ratio Report for Target Compound #1 from x:\... xvx-050902-6,spk6-10-1

Saturn 2000 No.1

ample ID:	XVX-050902-6,spk6-	Acquisition Date:	10/15/02 7:35 PM
Vial:	1	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	XVX-11-07-01A,spike,.046ug/mL VX; HAE extract:Rtx-5MS:30Mx0.25mmID,1um; 100C,5min-240C@5C/m-280C@25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C		
Data File:	c:\... \2002\october\101502#1\xvx-050902-6,spk6-10-15-02.sms		

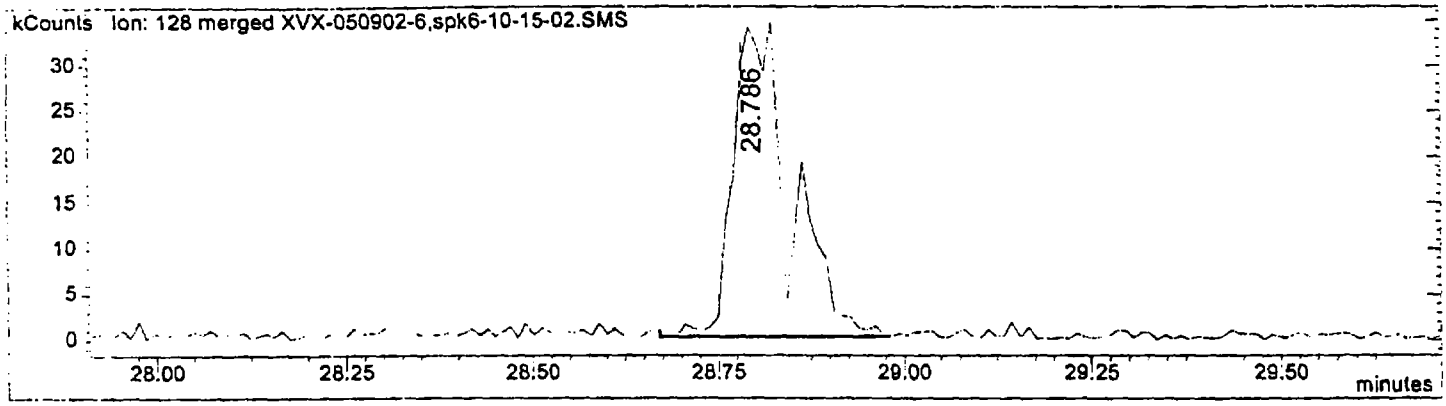
Y Abrahm
C31763 Bmk p.c
10-15-02

Analyte: VX

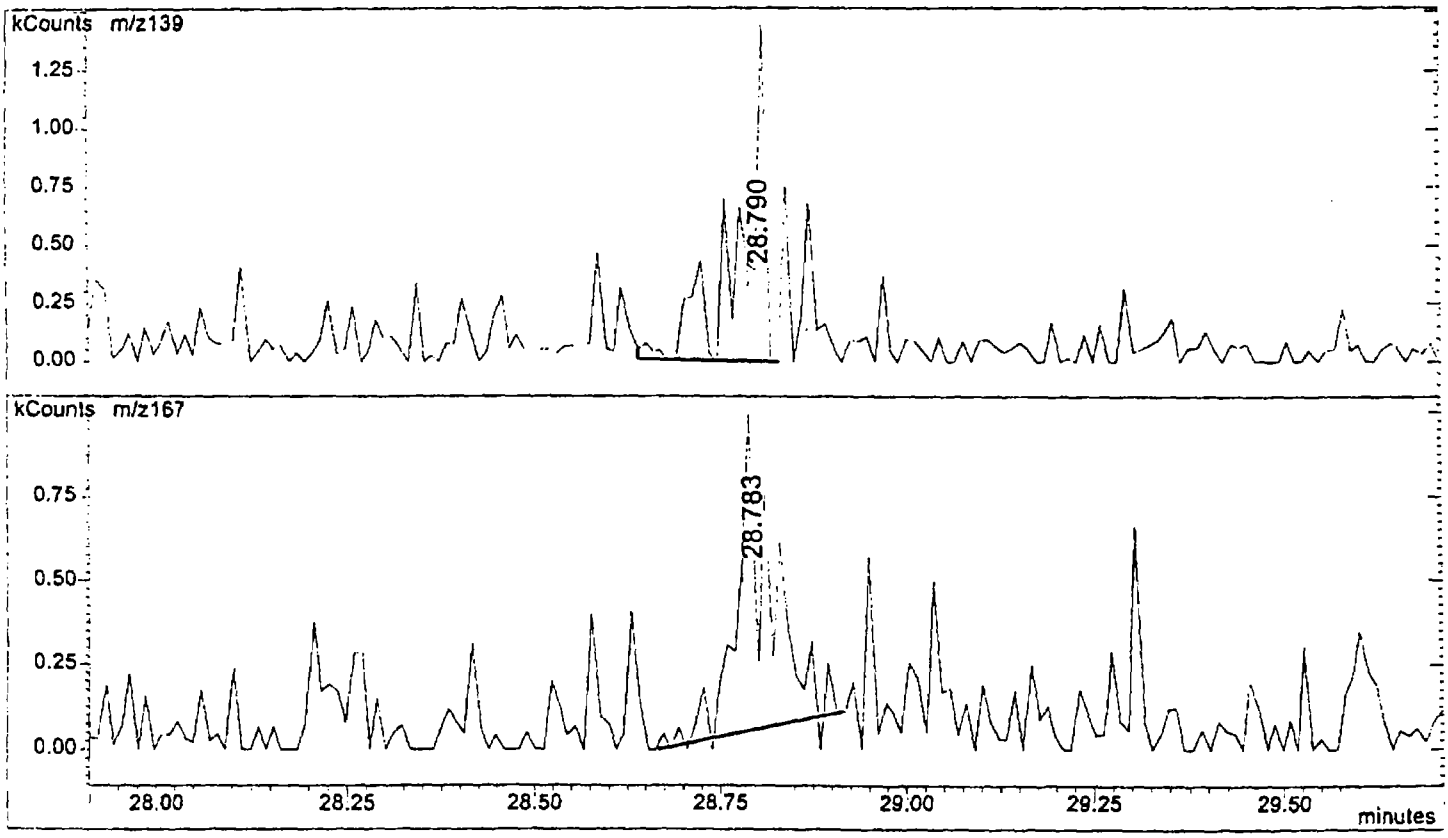
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.786 min.	28.812 min.	28.112 - 29.512 min.	181424	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.790 min.	1.72%	2.0%	0.6% - 3.4%	3124	Pass
167	28.783 min.	1.65%	4.6%	1.4% - 7.8%	2993	Pass

Concentration: 0.660 ug/mL



Sample: VX



Segment: 4 Time(min): 23.979-32.989 Scans: 1422-2274 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 16 Oct 2002 11:08:16

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

ımple ID:	XVX100302-3,0.46ug	Acquisition Date:	10/15/02 8:18 PM
ıal:	2	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \2002\october\101502#1\xvx100302-3,0.46ug10-15-02.sms		

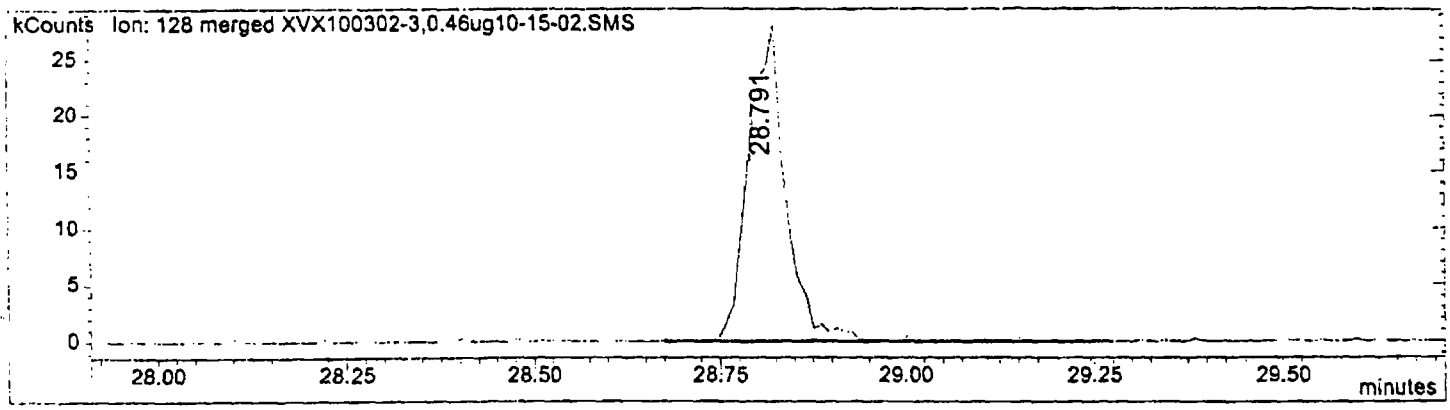
Y. Abitha
C31763 back P.6
10-15-02

Analyte: VX

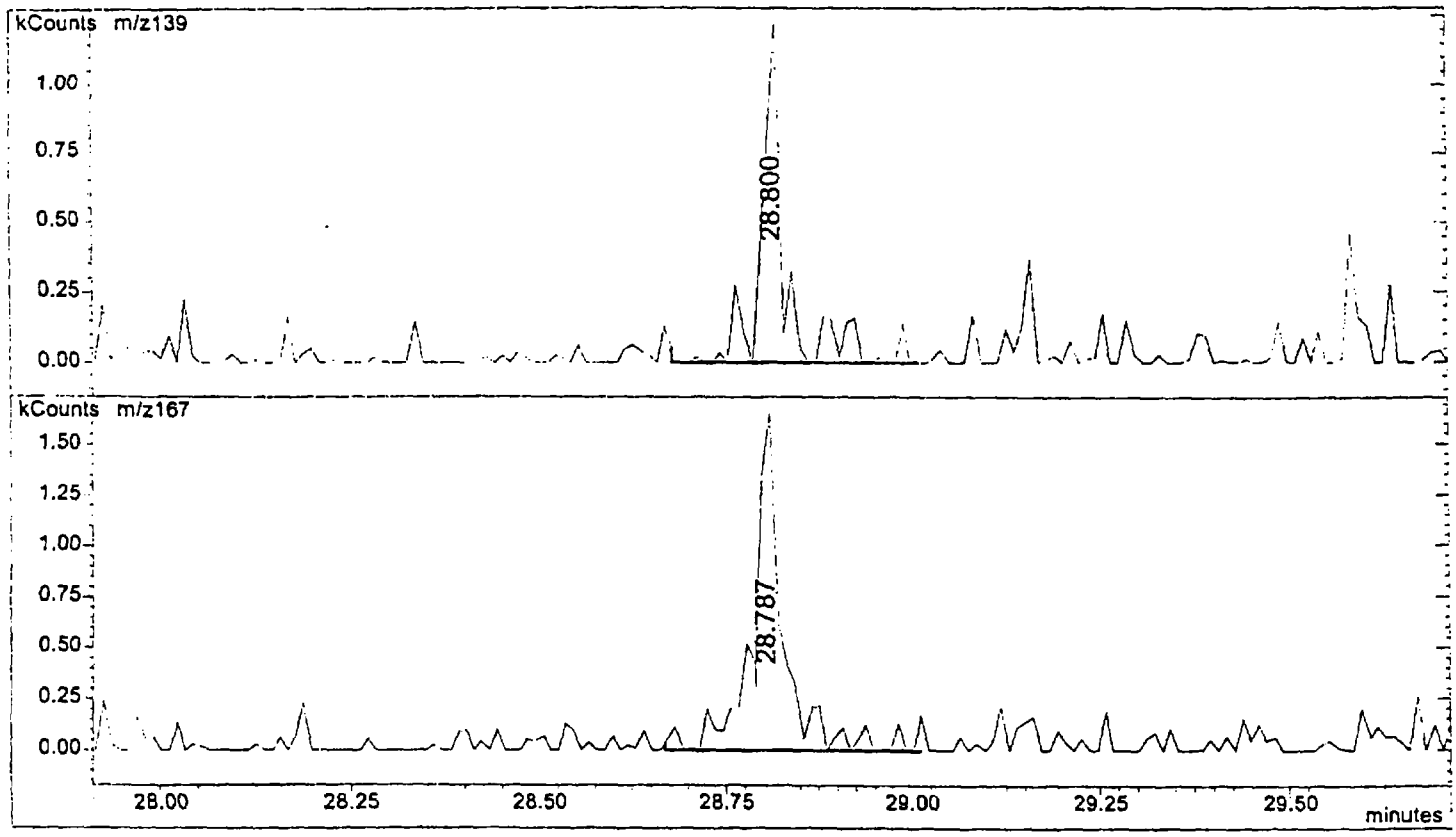
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.791 min.	28.812 min.	28.112 - 29.512 min.	98645	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.800 min.	2.23%	2.0%	0.6% - 3.4%	2198	Pass
167	28.787 min.	4.75%	4.6%	1.4% - 7.8%	4681	Pass

Concentration: 0.398 ug/mL



Sample: VX



Segment: 4 Time(min): 23.994-32.984 Scans: 1423-2270 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 16 Oct 2002 11:08:29

Ion Ratio Report for Target Compound #1 from VX-101502-US-1-10-15-02.SMS

Saturn 2000 No.1

Sample ID:	VX-101502-US-1-	Acquisition Date:	10/15/02 9:59 PM
Vial:	3	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	Method Spike: 91ng/g VX Spike into 5g NaSO4 + DEAET; Rtx5MS ;S-TRAP;EM@1750V		
Data File:	c:\... \2002\october\101502#1\vx-101502-us-1-10-15-02.sms		

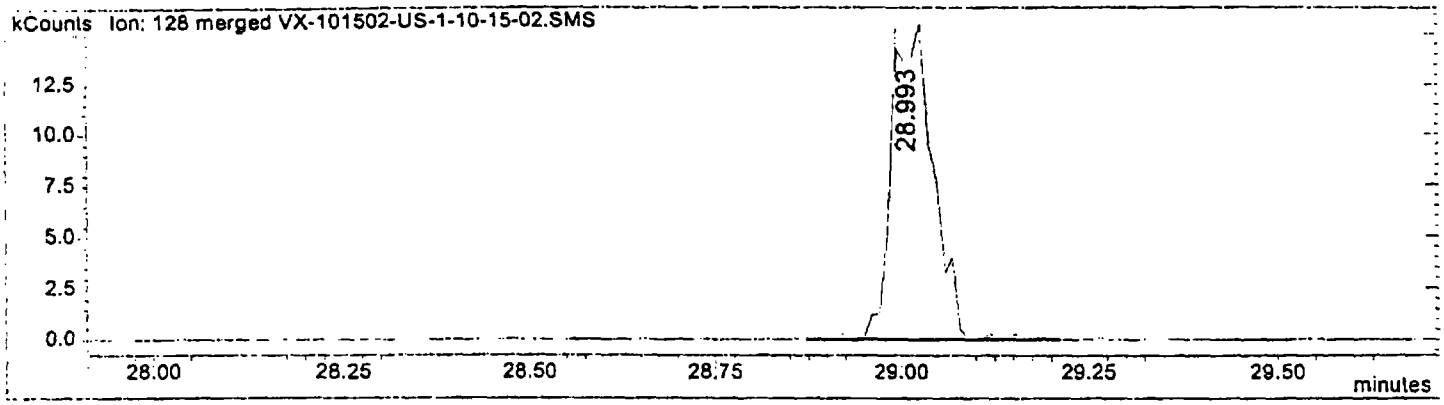
Y. Arthur
C31763 bulk Pb
10-15-02

Analyte: VX

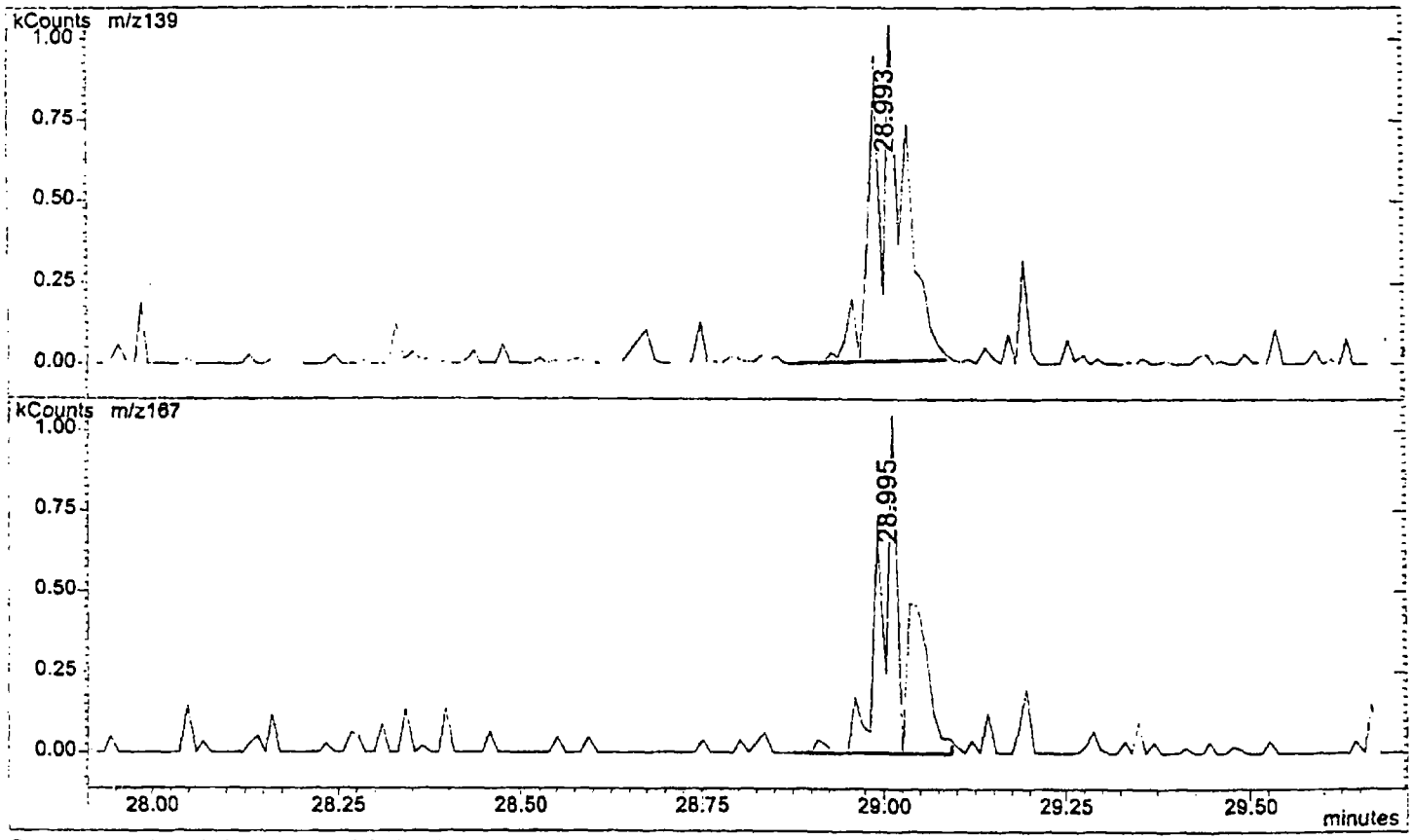
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.993 min.	28.812 min.	28.112 - 29.512 min.	60304	Failed

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.993 min.	4.95%	2.0%	0.6% - 3.4%	2985	Fail
167	28.995 min.	4.16%	4.6%	1.4% - 7.8%	2510	Pass

Concentration: 0.260 ug/mL



Sample: VX



Segment: 4 Time(min): 23.979-32.984 Scans: 1422-2280 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 16 Oct 2002 11:08:45

Ion Ratio Report for Target Compound #1 from VX-101502-US-2-10-15-02.SMS

Saturn 2000 No.1

Sample ID:	VX-101502-US-2-	Acquisition Date:	10/15/02 10:45 PM
Vial:	4	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	Method Spike: 91ng/g VX Spike into 5g NaSO4 + DEAET; Rtx5MS ;S-TRAP;EM@1750V		
Data File:	c:\... \2002\october\101502#1\vx-101502-us-2-10-15-02.sms		

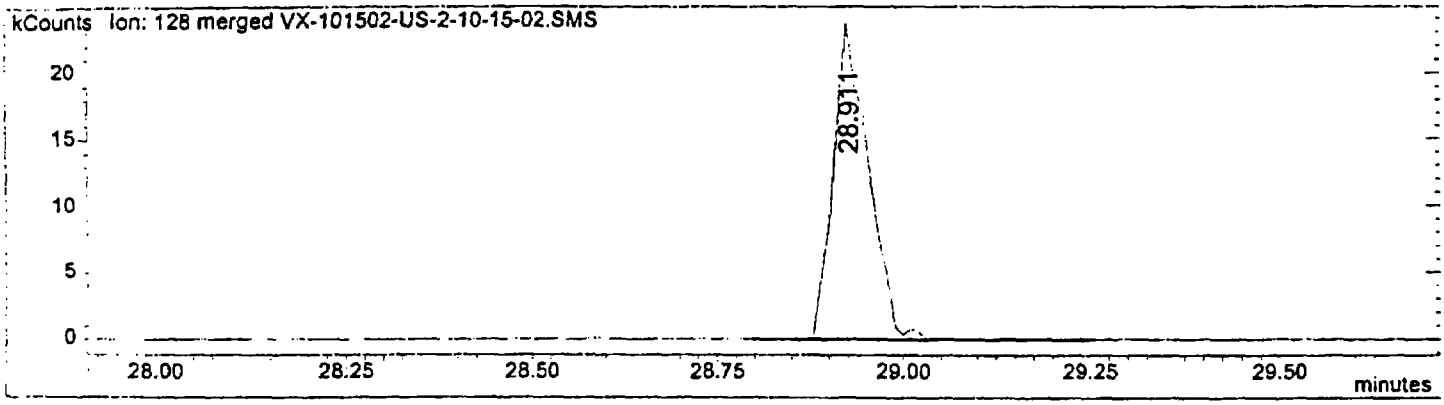
Y. Abrahm
C31263 Book P-6
10-15-02

Analyte: VX

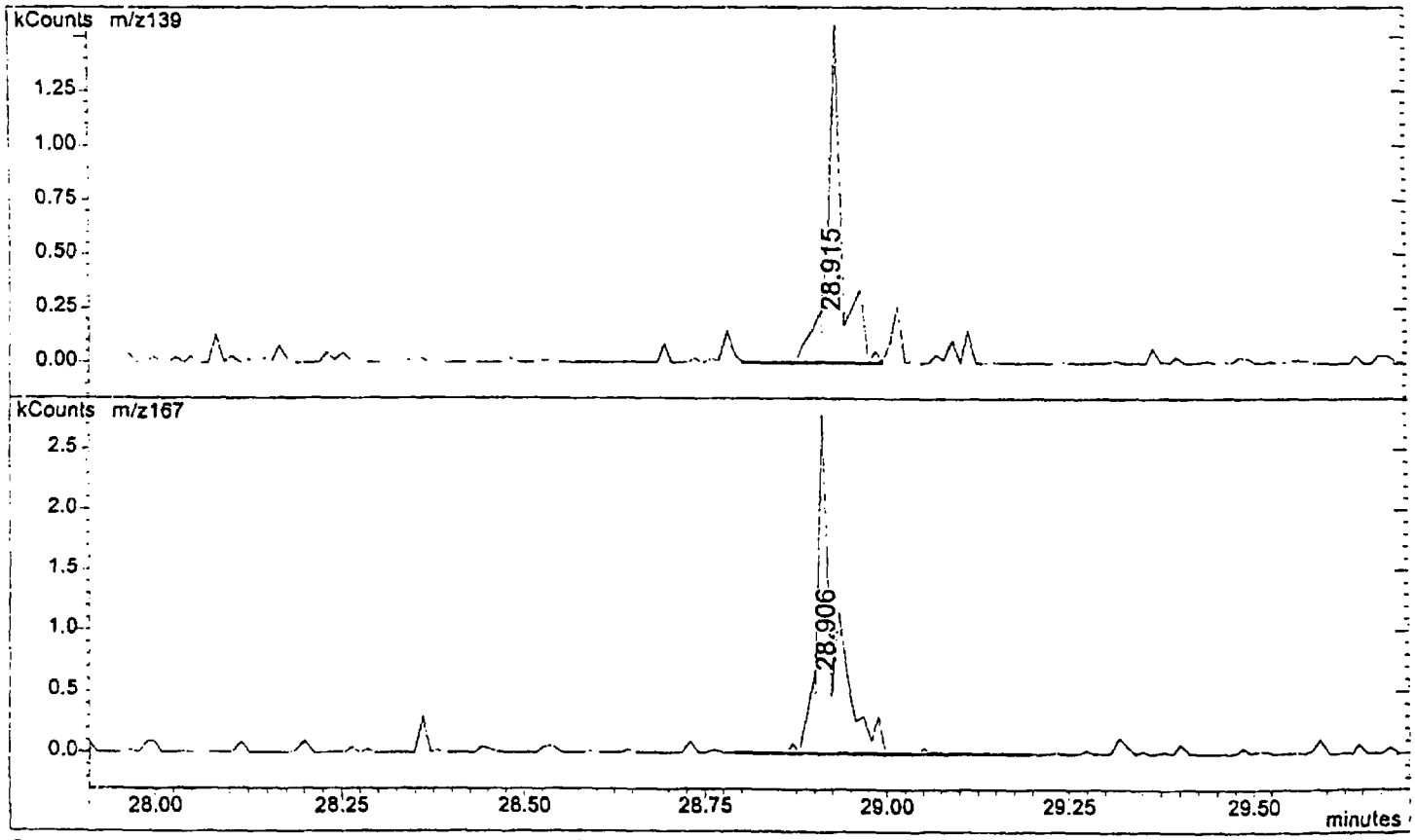
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.911 min.	28.812 min.	28.112 - 29.512 min.	78340	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.915 min.	2.91%	2.0%	0.6% - 3.4%	2282	Pass
167	28.906 min.	5.98%	4.6%	1.4% - 7.8%	4686	Pass

Concentration: 0.327 ug/mL



Sample: VX



Segment: 4 Time(min): 23.991-32.983 Scans: 1422-2266 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 16 Oct 2002 11:09:14

Ion Ratio Report for Target Compound #1 from VX-101502-US-3-10-15-02.SMS

Saturn 2000 No.1

Sample ID:	VX-101502-US-3-	Acquisition Date:	10/15/02 11:30 PM
Vial:	5	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultasonic Extraction, no HAE cleanup; Rtx5MS;S-TRAP;EM@1750V		
Data File:	c:\... \2002\october\101502#1\vx-101502-us-3-10-15-02.sms		

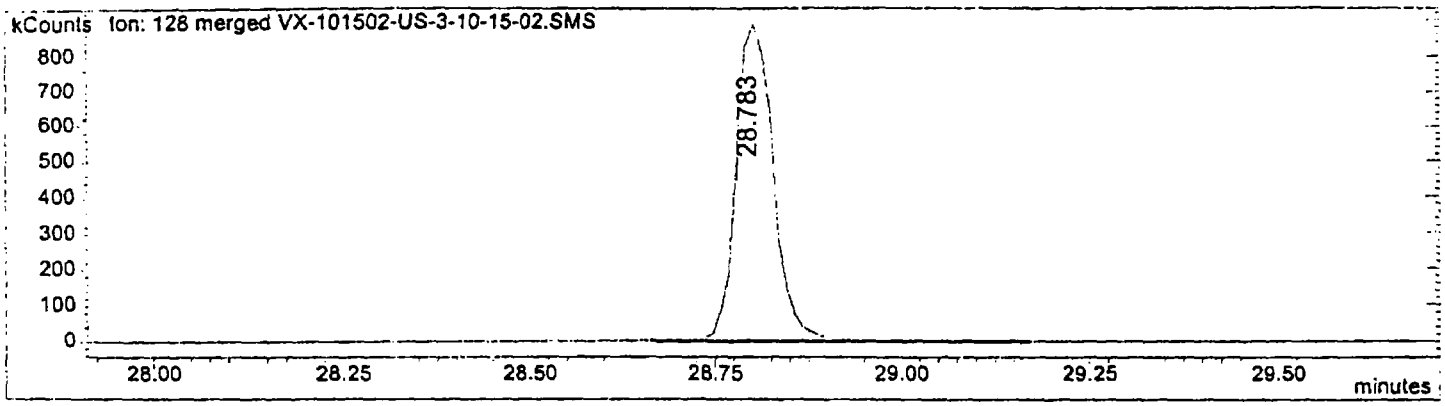
Y-Arthur
C31765 Lab P-6
10-15-02

Analyte: VX

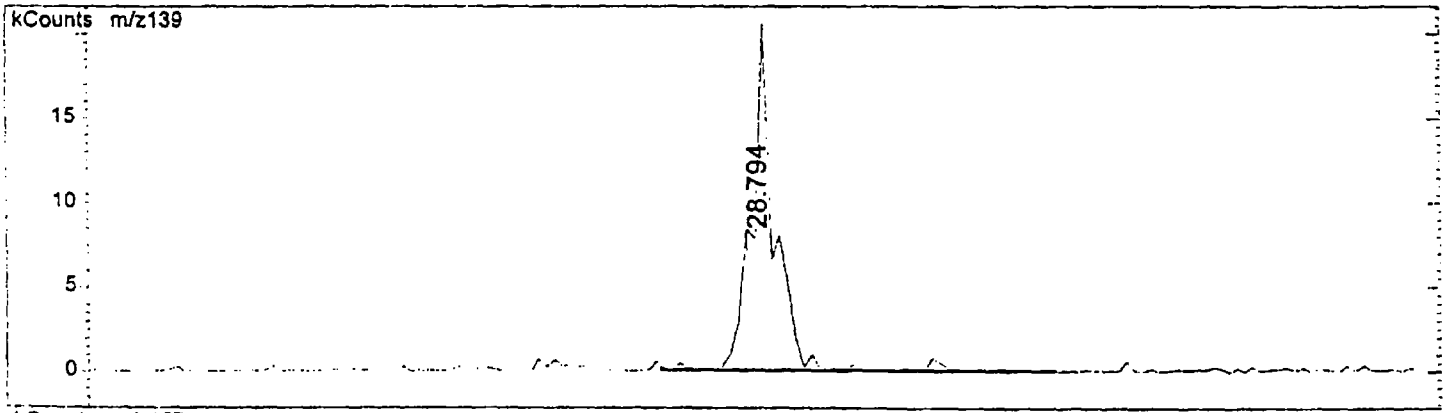
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.783 min.	28.812 min.	28.112 - 29.512 min.	3108364	Identified

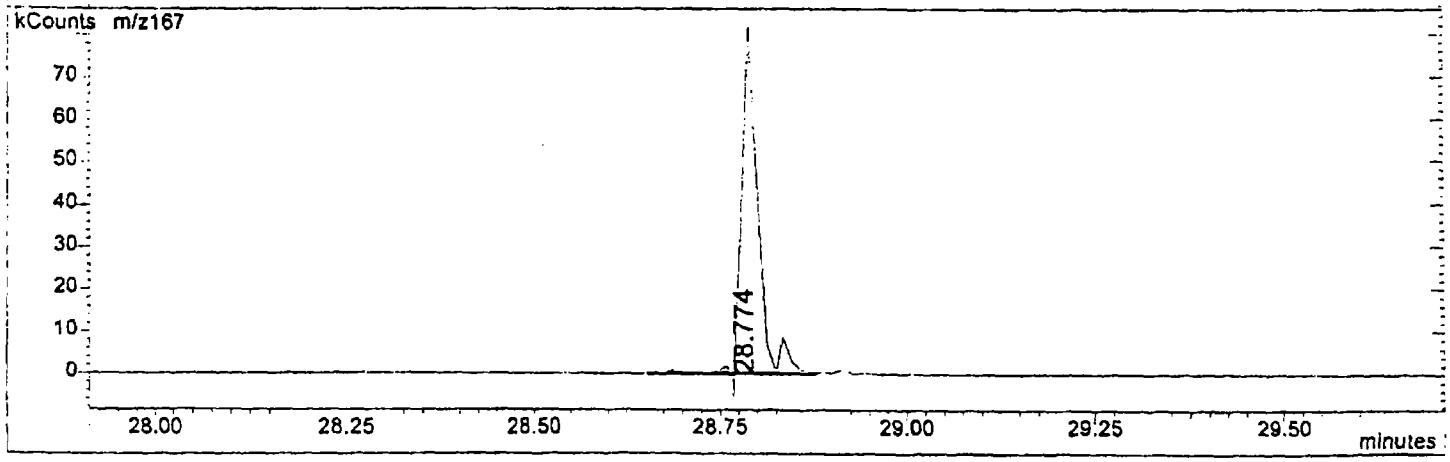
Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.794 min.	1.40%	2.0%	0.6% - 3.4%	43506	Pass
167	28.774 min.	4.03%	4.6%	1.4% - 7.8%	125115	Pass

Concentration: N/A



Sample: VX





Segment: 4 Time(min): 23.985-32.986 Scans: 1420-2263 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 16 Oct 2002 11:09:25

Ion Ratio Report for Target Compound #1 from VX-101502-US-4-10-16-02.SMS

Saturn 2000 No.1

ample ID:	VX-101502-US-4-	Acquisition Date:	10/16/02 12:15 AM
ial:	6	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultasonic Extraction, no HAE cleanup; Rtx5MS;S-TRAP;EM@1750V		
Data File:	c:\... \2002\october\101502#1\vx-101502-us-4-10-16-02.sms		

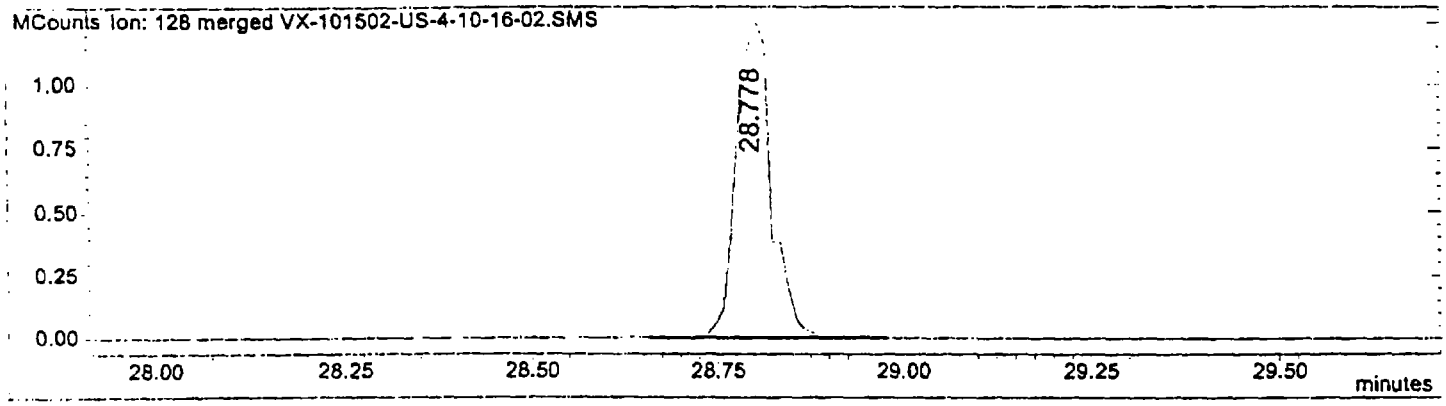
Y. Abrahe
C31763 back P-6
10-15-02

Analyte: VX

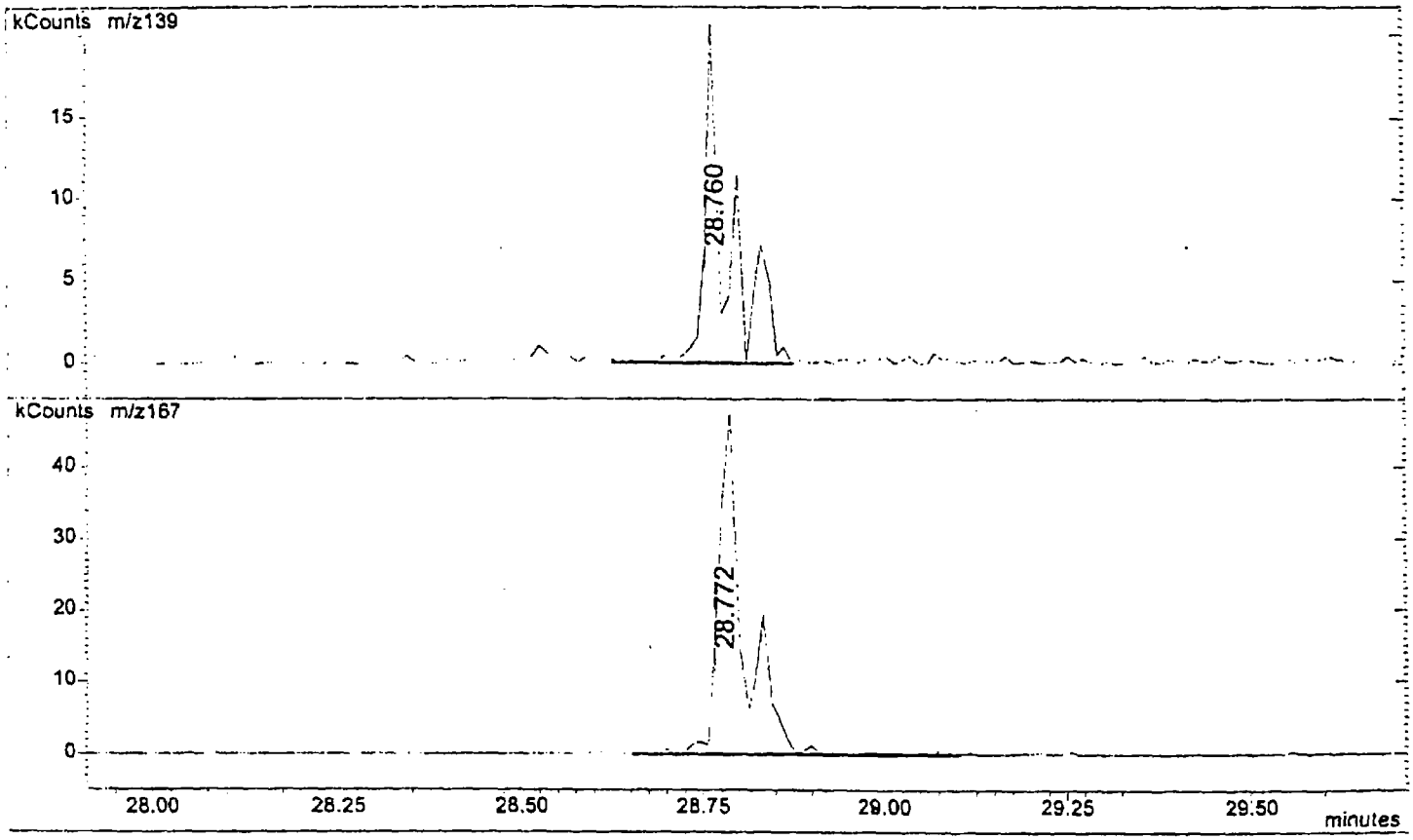
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.778 min.	28.812 min.	28.112 - 29.512 min.	4154852	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.760 min.	1.08%	2.0%	0.6% - 3.4%	44963	Pass
167	28.772 min.	2.77%	4.6%	1.4% - 7.8%	115252	Pass

Concentration: N/A



Sample: VX



Segment: 4 Time(min): 23.995-32.987 Scans: 1422-2262 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 16 Oct 2002 11:09:38

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/16/02 1:00 AM
Vial:	2	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\vx-calib-deaet-10-14-02.mth		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \2002\october\101502#1\xvx100302-3,0.46ug10-16-02.sms		

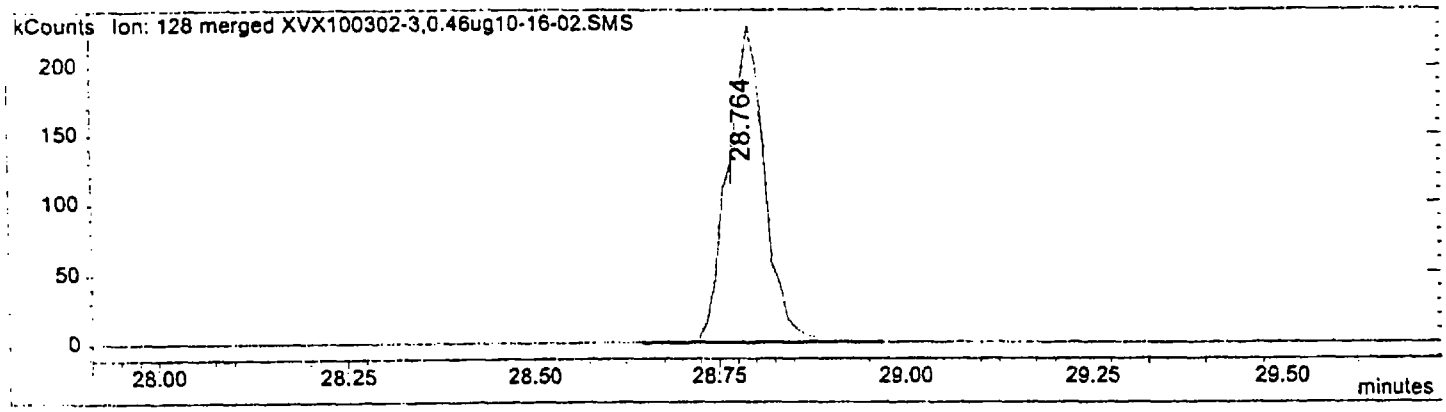
Y. Arthur
C 31763 book p-6
10-15-02

Analyte: VX

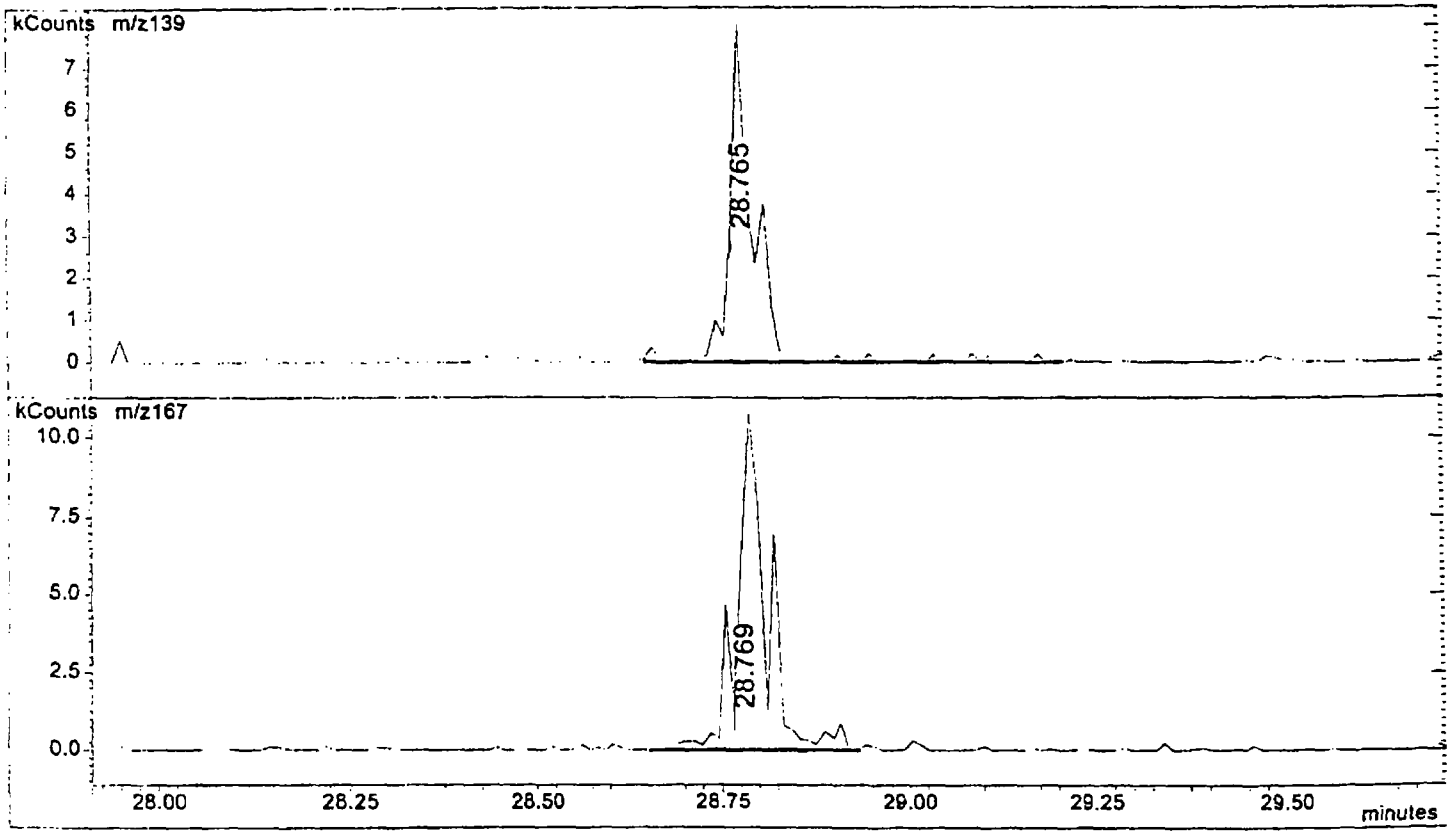
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.764 min.	28.812 min.	28.112 - 29.512 min.	787826	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.765 min.	2.18%	2.0%	0.6% - 3.4%	17169	Pass
167	28.769 min.	3.77%	4.6%	1.4% - 7.8%	29691	Pass

Concentration: 1.961 ug/mL



Sample: VX



Segment: 4 Time(min): 23.978-32.986 Scans: 1420-2260 Ionization: CI Auto Preparation: MS/MS Channels: 1

Y. Abtha
C31763 Back P-6
10-16-02

Varian Star Workstation - 3400 SampleList Wed Oct 16 18:05:59 2002

SampleList: C:\saturnws\Data\2002\October\10160281\VX10160281.smp

Wd: Wed Oct 16 10:06:33 2002
Mod: Wed Oct 16 18:05:53 2002

Line	Sample Name	Injection Notes	AutoLink	Vial	Inject Volume
1			C:\saturnws\Methods\VX-2002\VX101602-EI FullScan;Mult1750v+100v-UDInj.mth		
2	VX-101502-US-3-	below	none	1	1.0
	Notes JACADS Ash Sample No. LAWIC1538, 5g + DEAET; Final Volume = 5mL; Ultasonic Extraction, no HAE				
	Notes cleanup; RtxSMS;S-TRAP;EM@1750V + 100V; EI Full Scan m/z 50-650amu				
3			C:\saturnws\Methods\VX-2002\VX101603-EI FullScan;Mult1750v+300v-UDInj1.mth		
4	VX-101502-US-4-	below	none	2	1.0
	Notes JACADS Ash Sample No. LAWIC1538, 5g + DEAET; Ultasonic Extraction, no HAE cleanup;				
	Notes RtxSMS;S-TRAP;EM@1750V				
5			C:\saturnws\Methods\VX-2002\VX092502-mz268.2-0.42V-PIT-200-ST-0.65-noPP-Purge@20+EI;Mult1750v+300v-UDInj1.mth		
6	XVX100302-3,0.46ug	below	none	3	1.0
	Notes 0.457ug/mLVX Std,Rtx-SMS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
7	XVX100302-3,0.46ug	below	none	3	1.0
	Notes 0.457ug/mLVX Std,JACADS Ash Sample No. LAWIC1538, 5g + DEAET; Ultrasonic Extraction, no HAE				
	Notes cleanup; Initial Volume = 5mL, diluted 1:10 w/CH2Cl2, RtxSMS;S-TRAP;EM@1750V				
8	VX-101502-US-3D-	below	none	4	1.0
	Notes JACADS Ash Sample No. LAWIC1538, 5g + DEAET; Ultrasonic Extraction, no HAE cleanup; Initial Volume				
	Notes = 5mL, diluted 1:10 w/CH2Cl2, RtxSMS;S-TRAP;EM@1750V				
9	MeCl2 Blank	below	none	0	1.0
	Notes Methylene Chloride Blank				
10	VX-101502-US-4D-	below	none	5	1.0
	Notes JACADS Ash Sample No. LAWIC1538, 5g + DEAET; Ultrasonic Extraction, no HAE cleanup; Initial Volume				
	Notes = 5mL, diluted 1:10 w/CH2Cl2, RtxSMS;S-TRAP;EM@1750V				
11	XVX100302-3,0.46ug	below	none	3	1.0
	Notes 0.457ug/mLVX Std,Rtx-SMS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
12	XVX100302-1, 1.8ug	below	none	6	1.0
	Notes 1.83ug/mLVX Std, Rtx-SMS:30M x 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
13	XVX100302-1, 1.8ug	below	none	6	1.0
	Notes 1.83ug/mLVX Std, Rtx-SMS:30M x 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
14	XVX100302-2,0.91ug	below	none	7	1.0
	Notes 0.913ug/mLVX Std,Rtx-SMS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42V-EE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;TS-RAP@170C;Sngl-GN				
15	XVX100302-3,0.46ug	below	none	8	1.0
	Notes 0.457ug/mLVX Std,Rtx-SMS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
16	XVX100302-4,0.18ug	below	none	9	1.0
	Notes 0.183ug/mLVX Std,Rtx-SMS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
17	XVX100302-5,.091ug	below	none	10	1.0
	Notes 0.091ug/mLVX Std Rtx-5&11 MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
18	XVX100302-6,.046ug	below	none	11	1.0
	Notes 0.046ug/mLVX Std,Rtx-SMS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
19	XVX100302-3,0.46ug	below	none	8	1.0
	Notes 0.457ug/mLVX Std,Rtx-SMS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
20	VX-101502-US-1-	below	none	12	1.0
	Notes Method Spike: 91ng/g VX Spike into 5g NaSO4; DEAET added pre-analysis; RtxSMS;S-TRAP;EM@1750V				
21	VX-101502-US-2-	below	none	13	1.0
	Notes Method Spike: 91ng/g VX Spike into 5g NaSO4; DEAET added pre-analysis; RtxSMS;S-TRAP;EM@1750V				

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  22     VX-101502-US-3D-     below      none      4       1.0
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                Notes  JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extraction, no HAE cleanup; Initial Volume
                Notes  = 5mL, diluted 1:10 w/CH2Cl2, Rtx5MS;S-TRAP;EM@1750V
-----  ------------------  --------  --------  -----  ---------
   3     VX-101502-US-4D-     below      none      5       1.0
        ------------------  --------  --------  -----  ---------
                Notes  JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extraction, no HAE cleanup; Initial Volume
                Notes  = 5mL, diluted 1:10 w/CH2Cl2, Rtx5MS;S-TRAP;EM@1750V
-----  ------------------  --------  --------  -----  ---------
  24         CH2Cl2 Blank     below      none      0       1.0
        ------------------  --------  --------  -----  ---------
                Notes  CH2Cl2 Blank, Rtx-5MS:30Mx0.25mm ID,  1.0um; 100C,5min - 240C @5C/m-280C
                Notes  @25C/m,5min;0.42VEE;PIT@200usec; 0.65scans/sec;noPP;m/z268.2;1750V; S-TRAP@170C
-----  ------------------  --------  --------  -----  ---------
  25    XVX100302-3,0.46ug     below      none      8       1.0
        ------------------  --------  --------  -----  ---------
                Notes  0.457ug/mLVX Std,Rtx-5MS:30Mx0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C
                Notes  @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN
-----  ------------------  --------  --------  -----  ---------
  26                                C:\saturnws\Methods\Standby.mth
-----  ------------------  --------  --------  -----  ---------

Directory for Data Files: c:\saturnws\data\2002\october\101602#1

        Data File Names: %s%d


Create and update a new RecalcList: c:\saturnws\data\2002\october\101602#1\vx101602-1.rcl.
```

****

Varian Star Workstation - RecalcList Thu Oct 17 09:17:56 2002

.lcList: C:\saturnws\Data\2002\October\101602#1\VX101602-1.rcl

Created: Wed Oct 16 10:15:04 2002
Modified: Thu Oct 17 09:17:53 2002

****

Y. Abdur
C31763 Back P-6
10-16-02

Line	Sample Type	Sample Name	Data File
1	Analysis	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-16-02.SMS
2	Analysis	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-16-02001.SMS
3	Analysis	VX-101502-US-3-	vx-101502-us-3d-10-16-02.sms
4	Analysis	MeCl2 Blank	mecl2 blank-10-16-02.sms
5	Analysis	VX-101502-US-4-	vx-101502-us-4d-10-16-02.sms
6	Analysis	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-16-02002.SMS
7	Analysis	XVX100302-1, 1.8ug	XVX100302-1, 1.8ug10-16-02.SMS
8	Analysis	XVX100302-1, 1.8ug	XVX100302-1, 1.8ug10-16-02001.SMS
9	Analysis	XVX100302-2,0.91ug	XVX100302-2,0.91ug10-16-02.SMS
10	Analysis	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-16-02003.SMS
11	Analysis	XVX100302-4,0.18ug	XVX100302-4,0.18ug10-16-02.SMS
12	Analysis	XVX100302-5,.091ug	XVX100302-5,.091ug10-16-02.SMS
13	Analysis	XVX100302-6,.046ug	XVX100302-6,.046ug10-16-02.SMS
14	Analysis	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-16-02004.SMS
15	Analysis	VX-101502-US-1-	VX-101502-US-1-10-17-02.SMS
16	Analysis	VX-101502-US-2-	VX-101502-US-2-10-17-02.SMS
17	Analysis	VX-101502-US-3-	vx-101502-us-3d-10-17-02.sms
18	Analysis	VX-101502-US-4-	vx-101502-us-4d-10-17-02.sms
19	Analysis	CH2Cl2 Blank	ch2cl2 blank-10-17-02.sms
20	Analysis	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-17-02.SMS
21	Analysis	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-17-02001.SMS

Print Date: 21 Oct 2002 16:45:00

Calibration Block Report

Run File:	c:\saturnws\data\2002\october\101602#1\xvx100302-1, 1.8ug10-16-02.sms		
Instrument Method:	c:\... vx092502-mz268.2-0.42v-pit	Acquisition Date:	10/16/02 6:27 PM
Recalc Method:	c:\... vx-calib-deaet-10-16-02.mt	Last Calibration:	10/21/02 4:34 PM
Sample List:		Compound Table Updated:	10/21/02 6:42 PM
Sequence:		Detector:	2000 Mass Spec
Workstation Name:	Saturn GC/MS Workstation	Workstation Version:	Version 5.52
Peak Measurement:	Area	Calibration Type:	External Standard Analysis

Retention Time (min)	Peak Name	Cal. Method	X3	X2	X	C	r2	Cal. Range	No. of Points	Edit Codes
28.812	VX	2 IG None	343619.5	1056865.5	24310.9		0.997597	1-6	6	

Curve Fit Codes	Origin Point Codes		Regression Weighting Codes		Edit Codes
1 linear	IN	include	None	1/nX	1 curve
2 quadratic	IG	ignore	1/n	1/X2	2 origin
3 cubic	FO	force	1/X	1/nX2	3 coefficient

Ret. Time: 28.812 min. Peak Name: VX

Level	Amount	Replicate No.	Response	Avg. Response	Std. Dev.	% RSD	RF	Avg. RF
1	1.830000	1	3119859	3119858.5	#		1704840.7	
2	0.913000	1	1213277	1213276.6	#		1328890.1	
3	0.457000	1	647717	647716.8	#		1417323.5	
4	0.183000	1	294563	294563.2	#		1609634.7	
5	0.091000	1	73654	73654.0	#		809384.9	
6	0.046000	1	40986	40985.6	#		890991.1	1293510.8

= Too few points to calculate.

Peak Name	Level	Rep.	Injection Date/Time	Run Files
VX	1	1	10/16/02 7:12 PM	c:\... \october\101602#1\xvx100302-1, 1.8ug10-16-02001.sms
	2	1	10/16/02 7:57 PM	c:\... \2002\october\101602#1\xvx100302-2,0.91ug10-16-02.sms
	3	1	10/16/02 8:42 PM	c:\... \october\101602#1\xvx100302-3,0.46ug10-16-02003.sms
	4	1	10/16/02 9:27 PM	c:\... \2002\october\101602#1\xvx100302-4,0.18ug10-16-02.sms
	5	1	10/16/02 10:12 PM	c:\... \2002\october\101602#1\xvx100302-5,.091ug10-16-02.sms
	6	1	10/16/02 10:57 PM	c:\... \2002\october\101602#1\xvx100302-6,.046ug10-16-02.sms

Print Date: 21 Oct 2002 16:44:36

Calibration Curve Report

e: c:\saturnws\methods\vx-2002\vx-calib-deaet-10-16-02.mth

etector: 2000 Mass Spec, Address: 40

Y. Adrian
C31763 Buck P-6
10-16-02

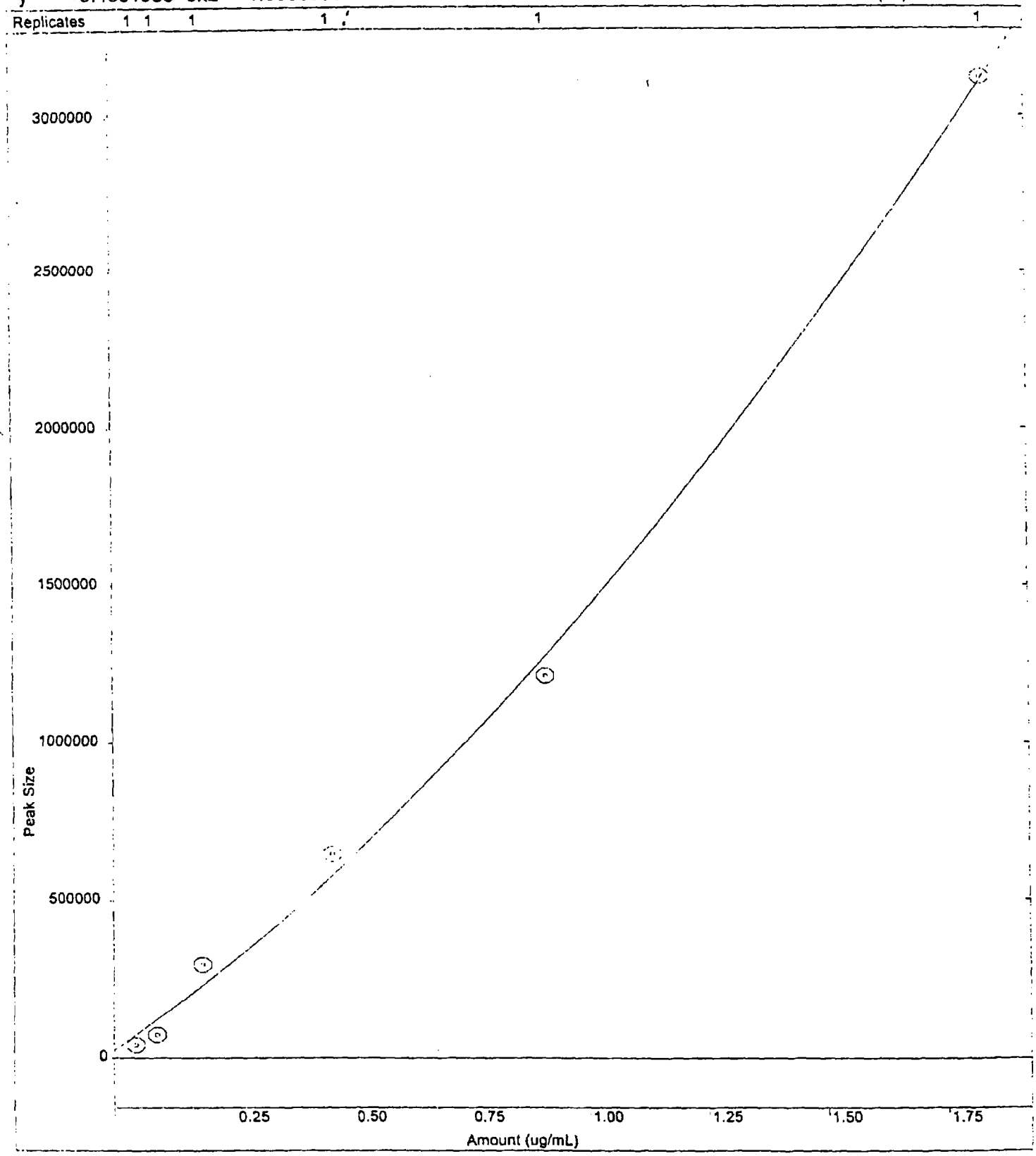
VX

Curve Fit: Quadratic, Origin: Ignore, Weight: None

y = +3.436195e+5x2 +1.056866e+6x +2.431092e+4

Resp. Fact. RSD: 28.55%

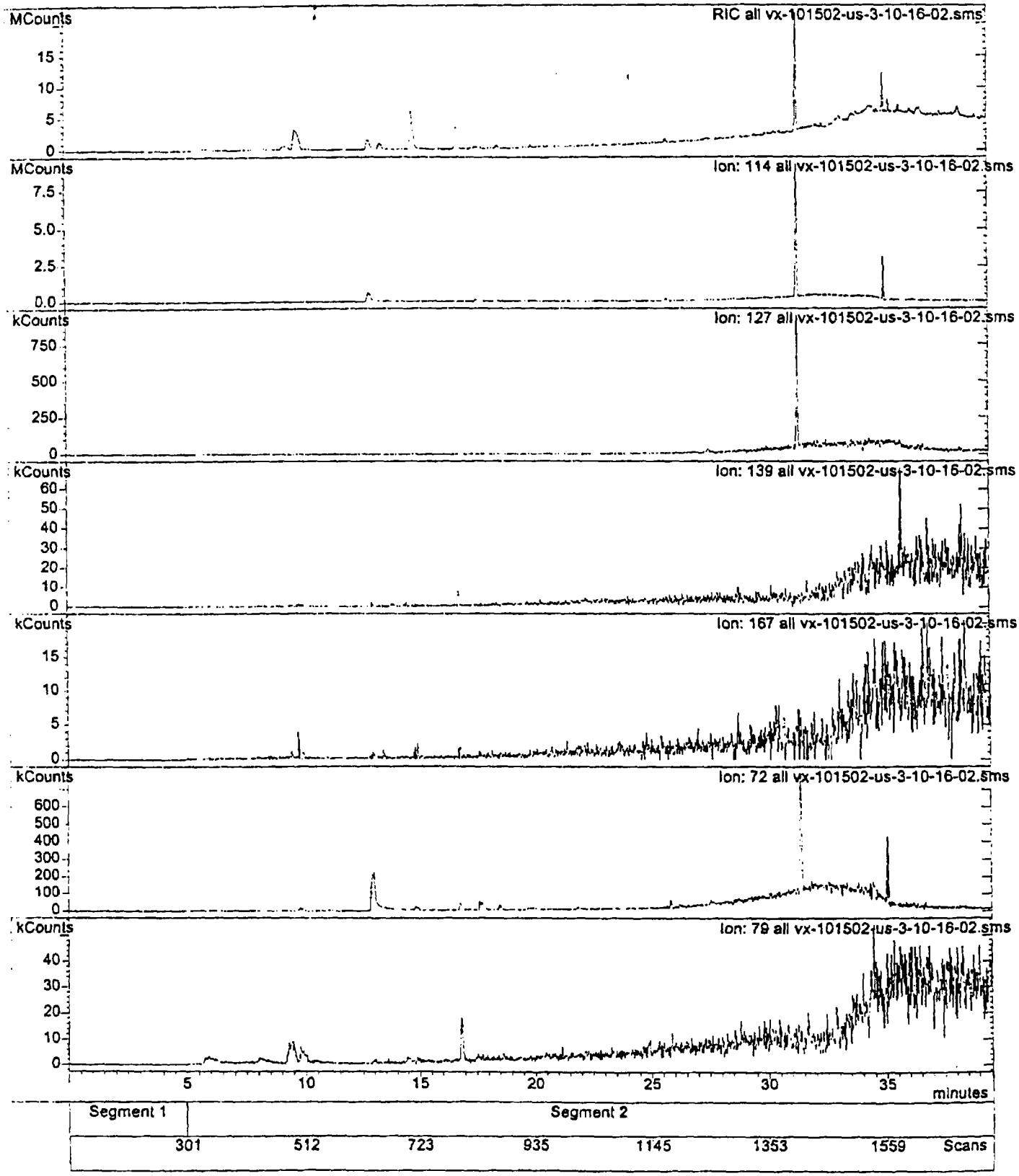
Coeff. Det.(r2): 0.997597



Print Date: 17 Oct 2002 09:26:58

Chromatogram Plots

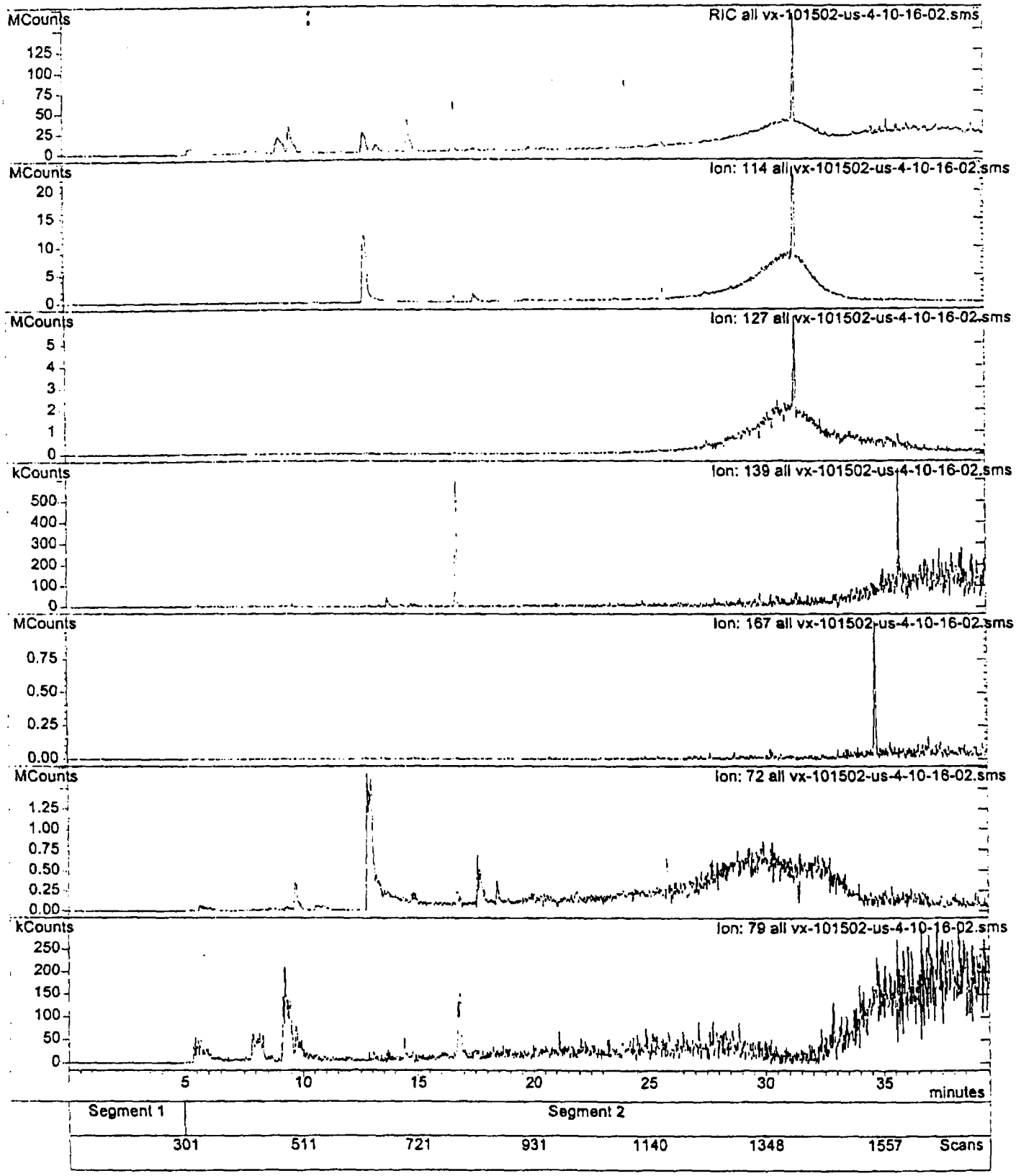
File: c:\satúrnws\data\2002\october\101602#1\vx-101502-us-3-10-16-02.sms
Sample: VX-101502-US-3- Operator: MM
Scan Range: 1 - 1747 Time Range: 0.00 - 39.56 min. Date: 10/16/02 10:17 AM
Sample Notes: JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Final Volume = 5mL;



Print Date: 17 Oct 2002 09:27:41

Chromatogram Plots

File: c:\saturnws\data\2002\october\101602#1\vx-101502-us-4-10-16-02.sms
Sample: VX-101502-US-4- Operator: MM
Scan Range: 1 - 1747 Time Range: 0.00 - 39.56 min. Date: 10/16/02 11:07 AM
Sample Notes: JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultasonic Extractio

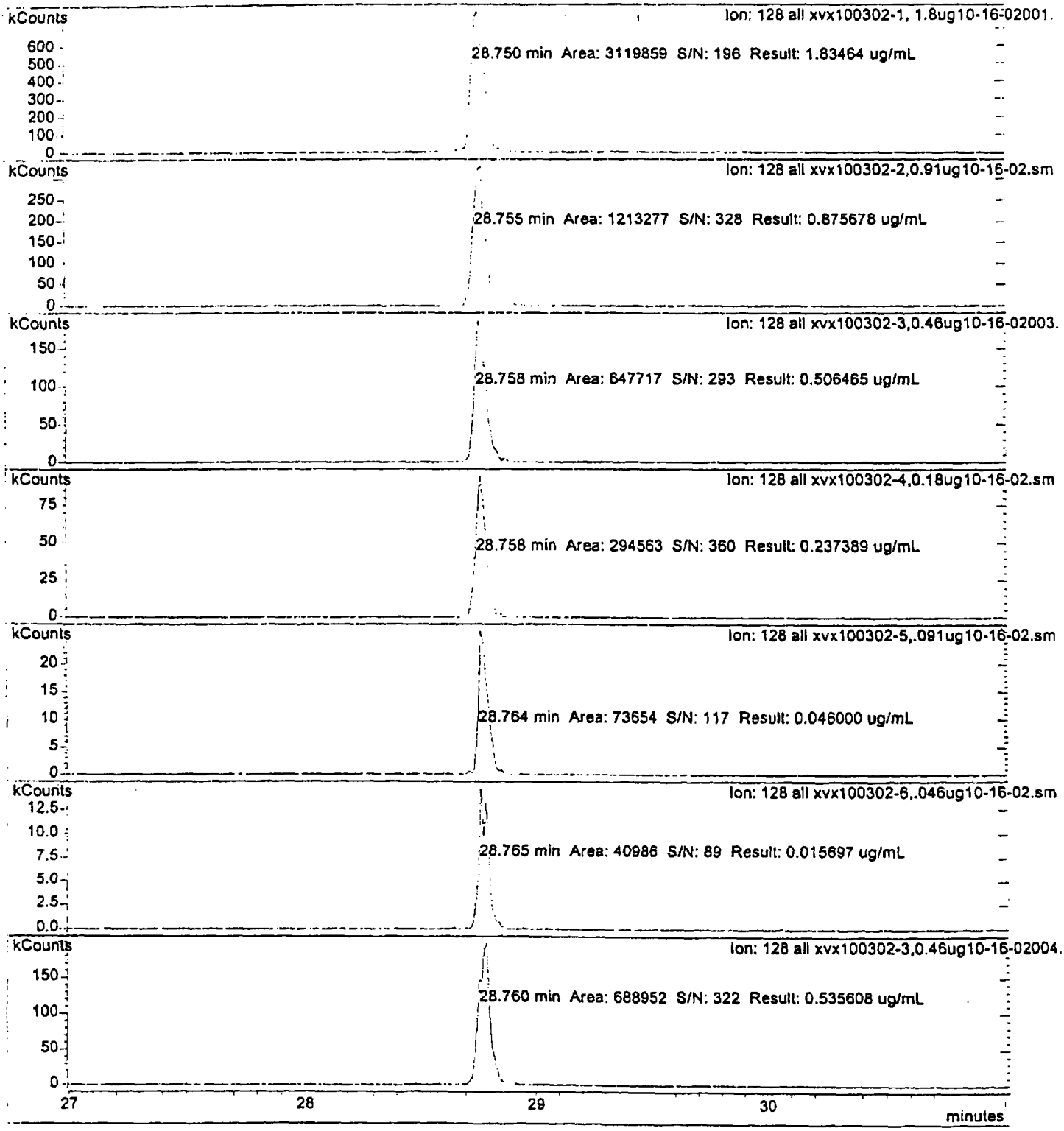


Print Date: 21 Oct 2002 17:27:49

Y Abrahm
C31763 Back P-6
10-16-02

Chromatogram Plots

Plot 1: c:\... \xvx100302-1, 1.8ug10-16-02001.sms Ion: 128 all
Plot 2: c:\... \101602#1\xvx100302-2,0.91ug10-16-02.sms Ion: 128 all
Plot 3: c:\... \xvx100302-3,0.46ug10-16-02003.sms Ion: 128 all
Plot 4: c:\... \101602#1\xvx100302-4,0.18ug10-16-02.sms Ion: 128 all
Plot 5: c:\... \101602#1\xvx100302-5,.091ug10-16-02.sms Ion: 128 all
Plot 6: c:\... \101602#1\xvx100302-6,.046ug10-16-02.sms Ion: 128 all
Plot 7: c:\... \xvx100302-3,0.46ug10-16-02004.sms Ion: 128 all

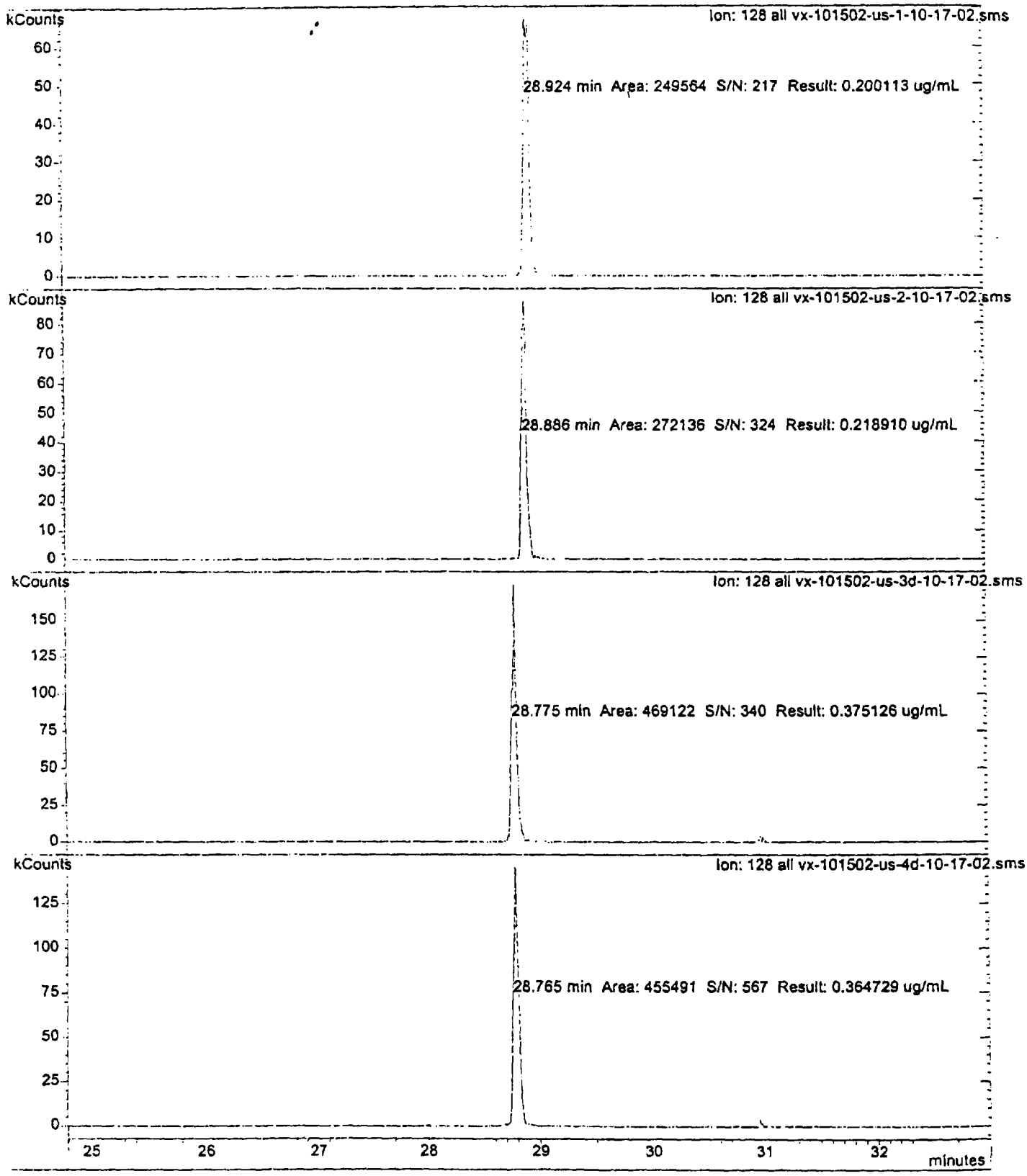


Print Date: 21 Oct 2002 17:15:40

Y. Abthu
C31763 Book. P. 6
10-16-02

Chromatogram Plots

Plot 1: c:\... \101602#1\vx-101502-us-1-10-17-02.sms Ion: 128 all
Plot 2: c:\... \101602#1\vx-101502-us-2-10-17-02.sms Ion: 128 all
Plot 3: c:\... \101602#1\vx-101502-us-3d-10-17-02.sms Ion: 128 all
Plot 4: c:\... \101602#1\vx-101502-us-4d-10-17-02.sms Ion: 128 all



Print Date: 21 Oct 2002 18:14:59

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/16/02 12:59 PM
Vial:	3	Measurement Type:	Area
Data File:	c:\... \2002\october\101602#1\xvx100302-3,0.46ug10-16-02.sms		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

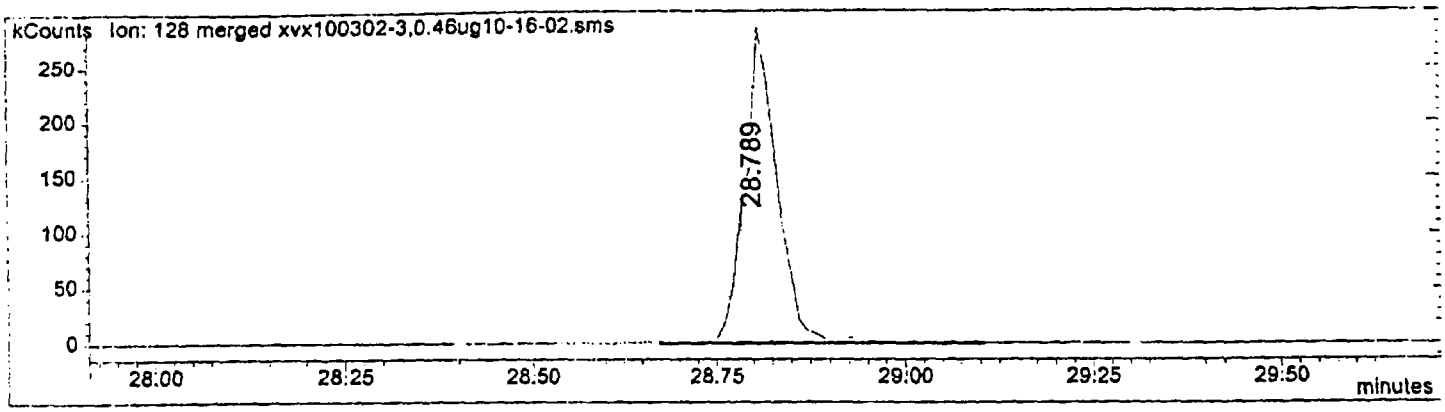
Y. Abrahan
C31763 Bk p-6
10-16-02

Analyte: VX

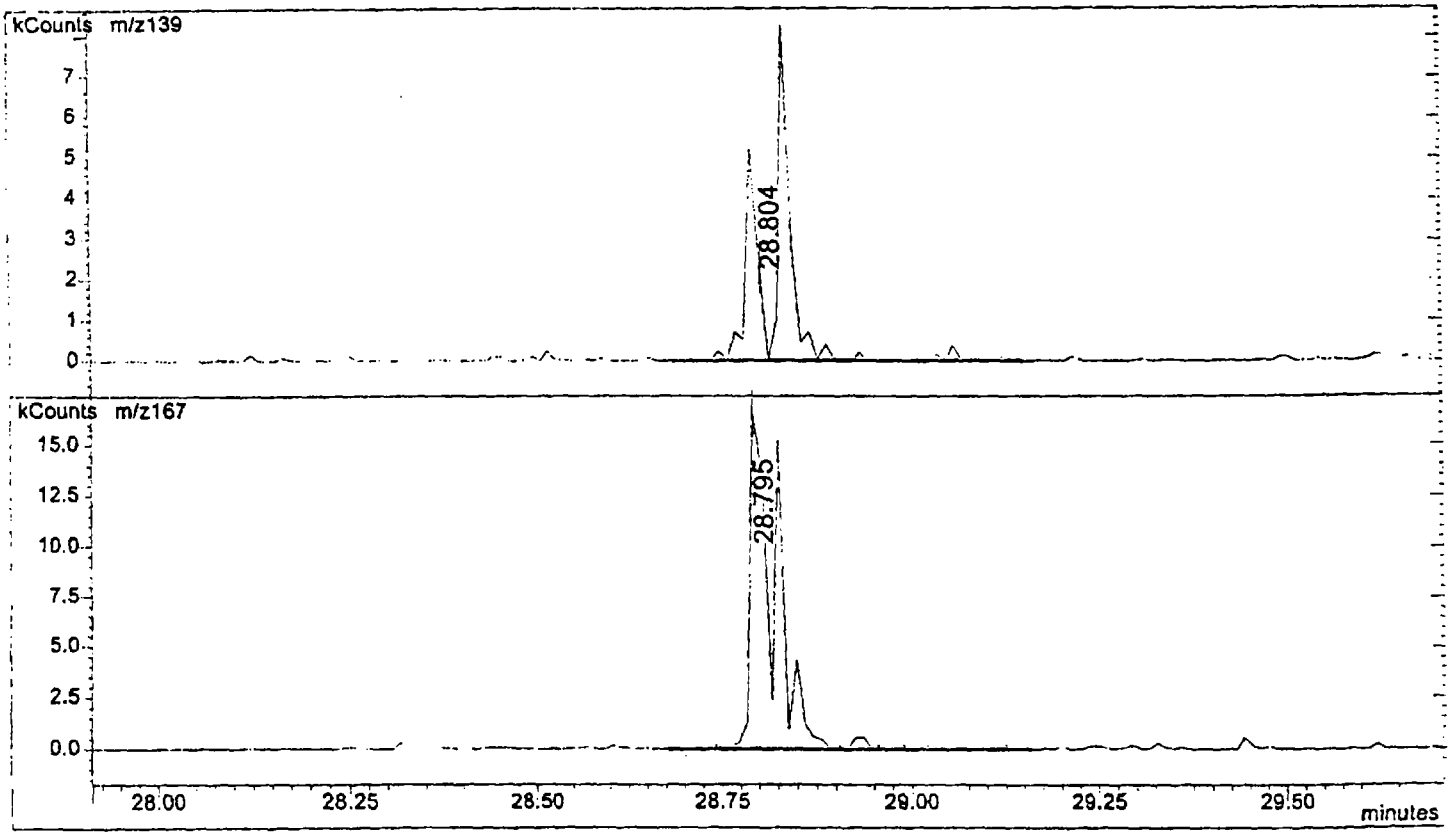
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.789 min.	28.812 min.	28.112 - 29.512 min.	858353	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.804 min.	1.82%	2.0%	0.6% - 3.4%	15583	Pass
167	28.795 min.	4.48%	4.6%	1.4% - 7.8%	38421	Pass

Concentration: 0.651 ug/mL



Sample: VX



Segment: 4 Time(min): 23.998-32.985 Scans: 1422-2271 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:16:12

Ion Ratio Report for Target Compound #1 from vx-101502-us-3d-10-16-02.sms

Saturn 2000 No.1

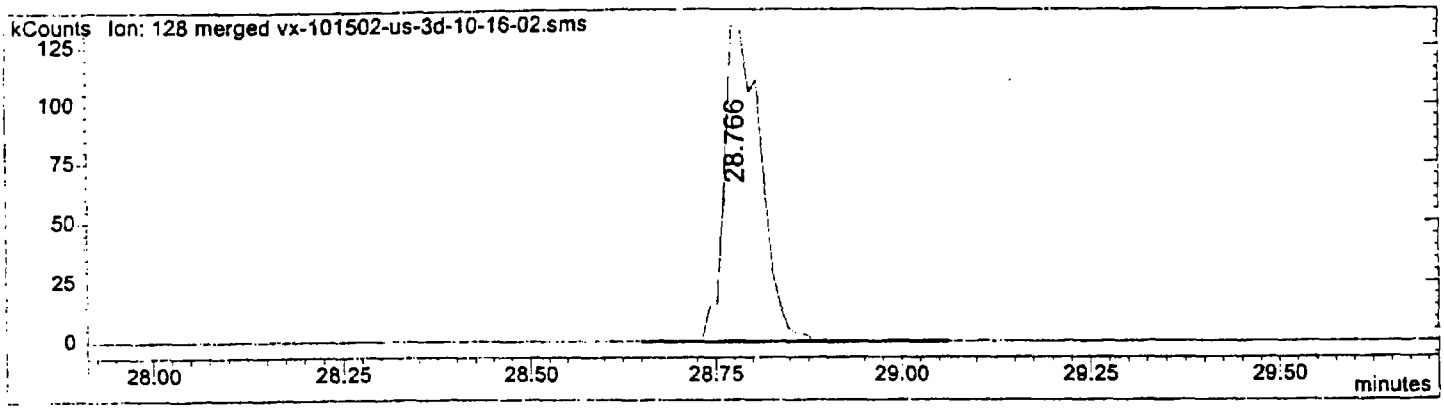
ample ID:	VX-101502-US-3-	Acquisition Date:	10/16/02 2:29 PM
Vial:	4	Measurement Type:	Area
Data File:	c:\... \2002\october\101602#1\vx-101502-us-3d-10-16-02.sms		
Inj. Sample Notes:	JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extr action, no HAE cleanup; Initial Volume = 5mL, diluted 1:10 w /CH2Cl2, Rtx5MS;S-TRAP;EM@1750V		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

Analyte: VX

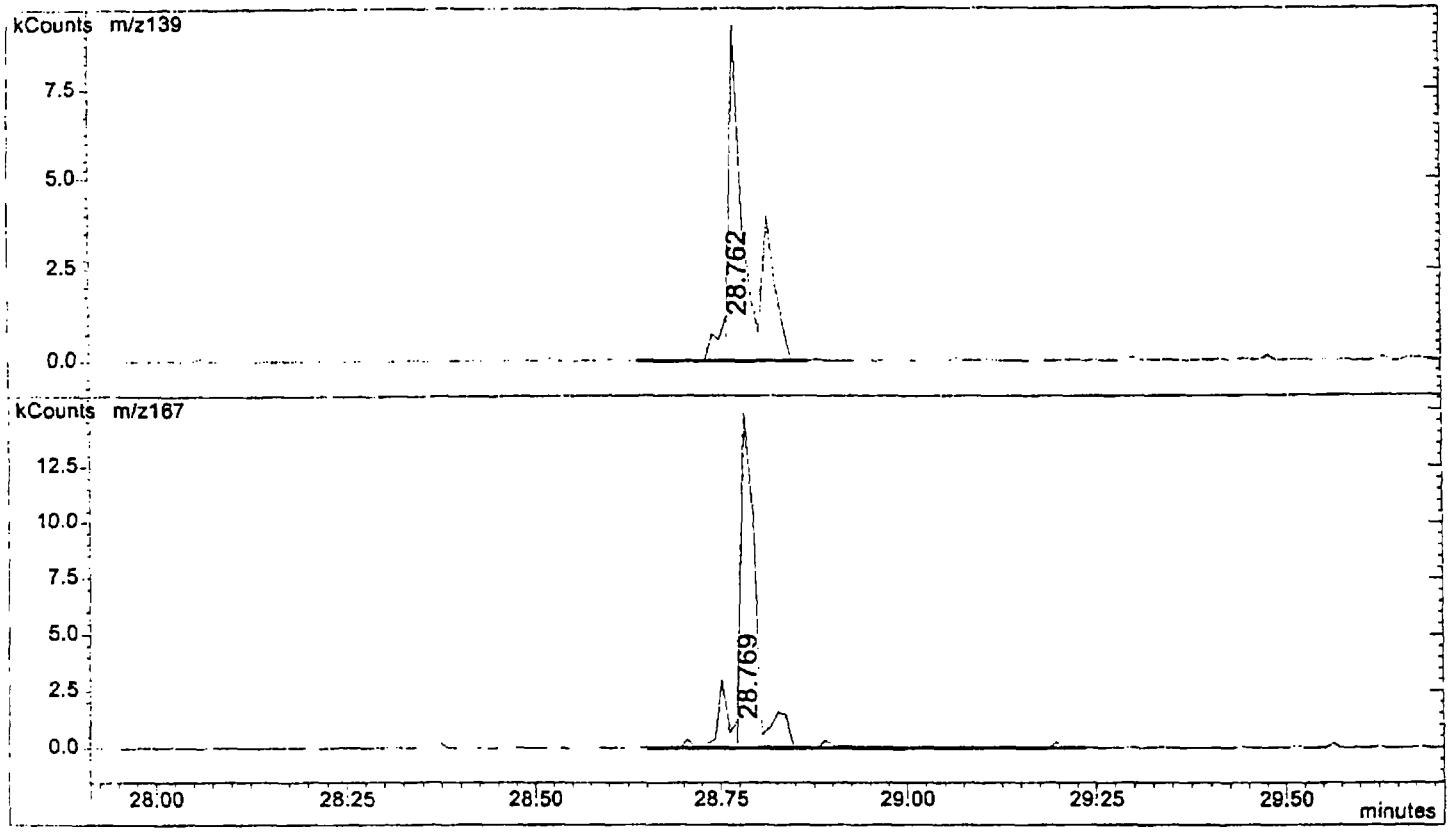
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.766 min.	28.812 min.	28.112 - 29.512 min.	460378	Failed

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.762 min.	3.51%	2.0%	0.6% - 3.4%	16156	Fail
167	28.769 min.	5.23%	4.6%	1.4% - 7.8%	24084	Pass

Concentration: 0.368 ug/mL



Sample: VX



Segment: 4 Time(min): 23.991-32.985 Scans: 1421-2266 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:16:29

Ion Ratio Report for Target Compound #1 from mecl2 blank-10-16-02.sms

Saturn 2000 No.1

ample ID:	MeCl2 Blank	Acquisition Date:	10/16/02 3:14 PM
Vial:	0	Measurement Type:	Area
Data File:	c:\... \data\2002\october\101602#1\mecl2 blank-10-16-02.sms		
Inj. Sample Notes:	Methylene Chloride Blank		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

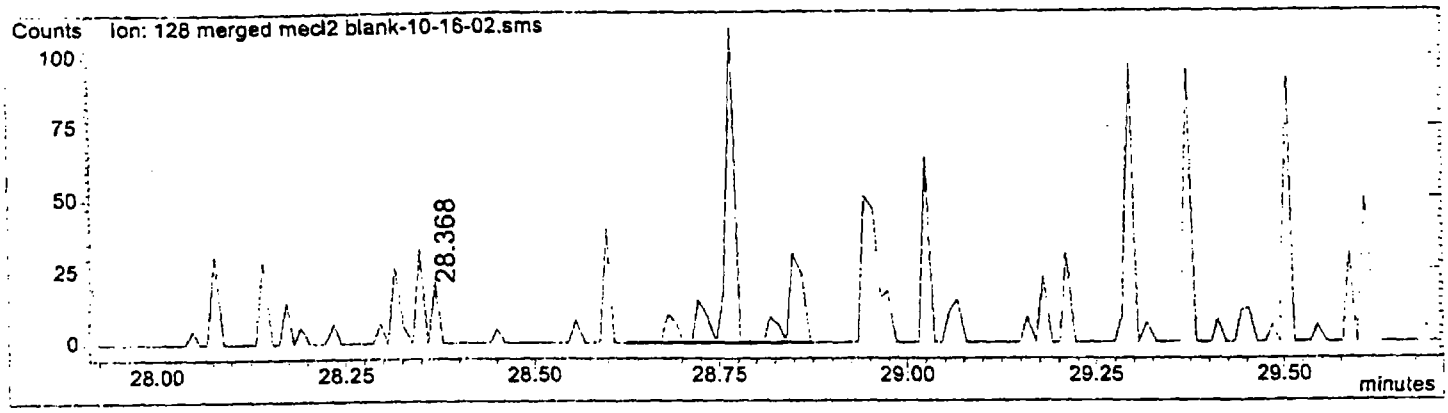
Y. Arthur
C31763 Back P-6
10-16-02

Analyte: VX

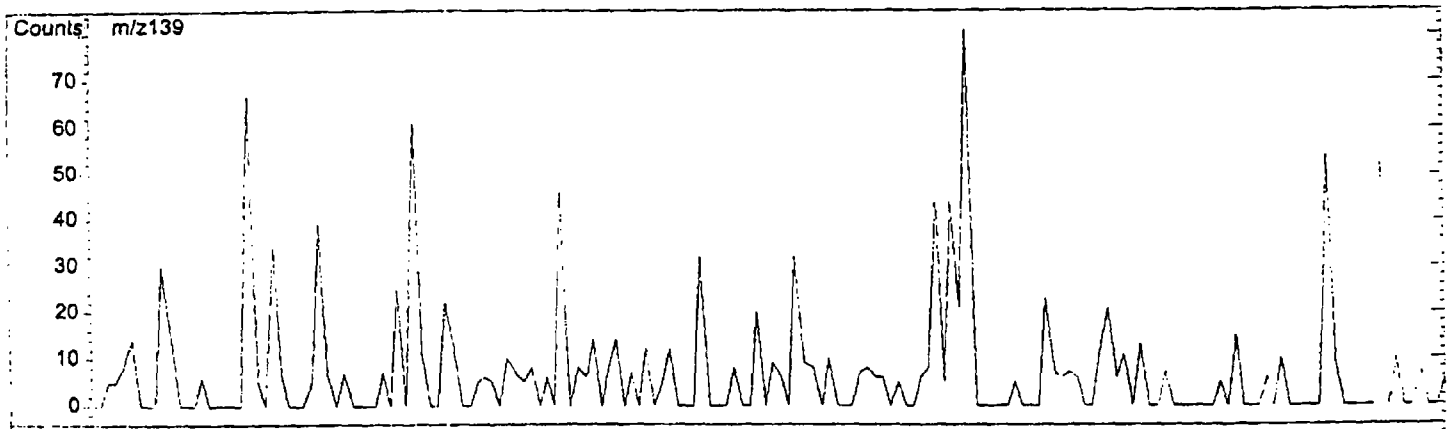
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.368 min.	28.812 min.	28.112 - 29.512 min.	0	Missing

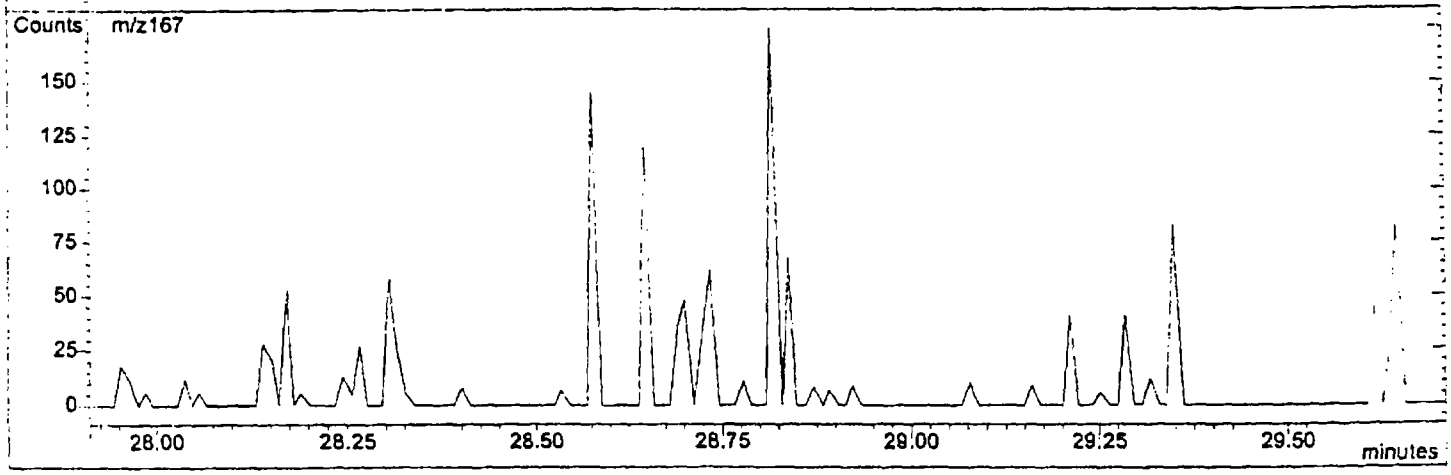
Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	0.000 min.	0.00%	2.0%	0.6% - 3.4%	0	Fail
167	0.000 min.	0.00%	4.6%	1.4% - 7.8%	0	Fail

Concentration: 0.000 ug/mL



Sample: VX





Segment: 4 Time(min): 23.992-32.980 Scans: 1423-2285 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:16:52

Ion Ratio Report for Target Compound #1 from vx-101502-us-4d-10-16-02.sms

Saturn 2000 No.1

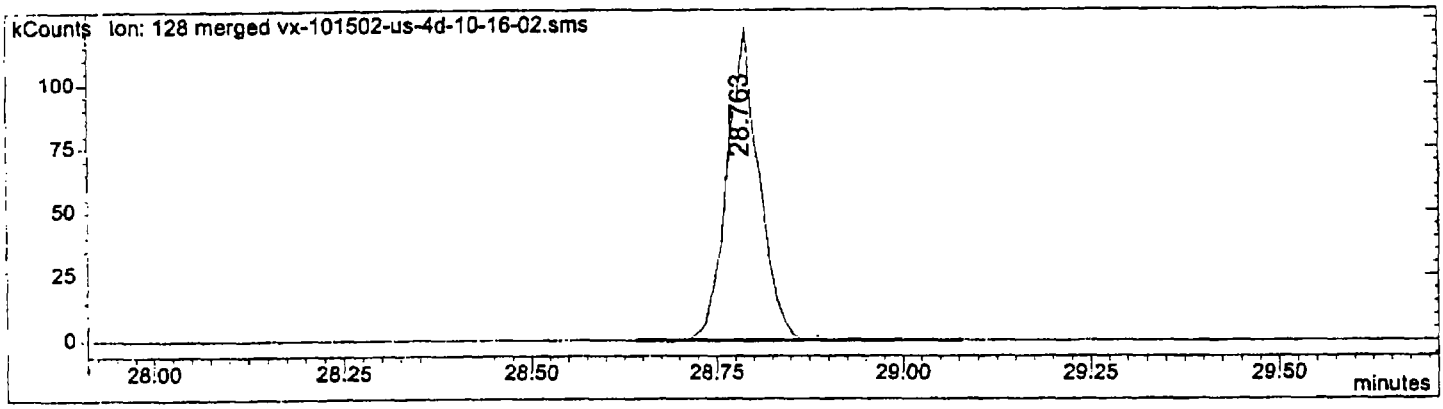
ample ID:	VX-101502-US-4-	Acquisition Date:	10/16/02 3:59 PM
Vial:	5	Measurement Type:	Area
Data File:	c:\... \2002\october\101602#1\vx-101502-us-4d-10-16-02.sms		
Inj. Sample Notes:	JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extr action, no HAE cleanup; Initial Volume = 5mL, diluted 1:10 w /CH2Cl2, Rtx5MS;S-TRAP;EM@1750V		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

Analyte: VX

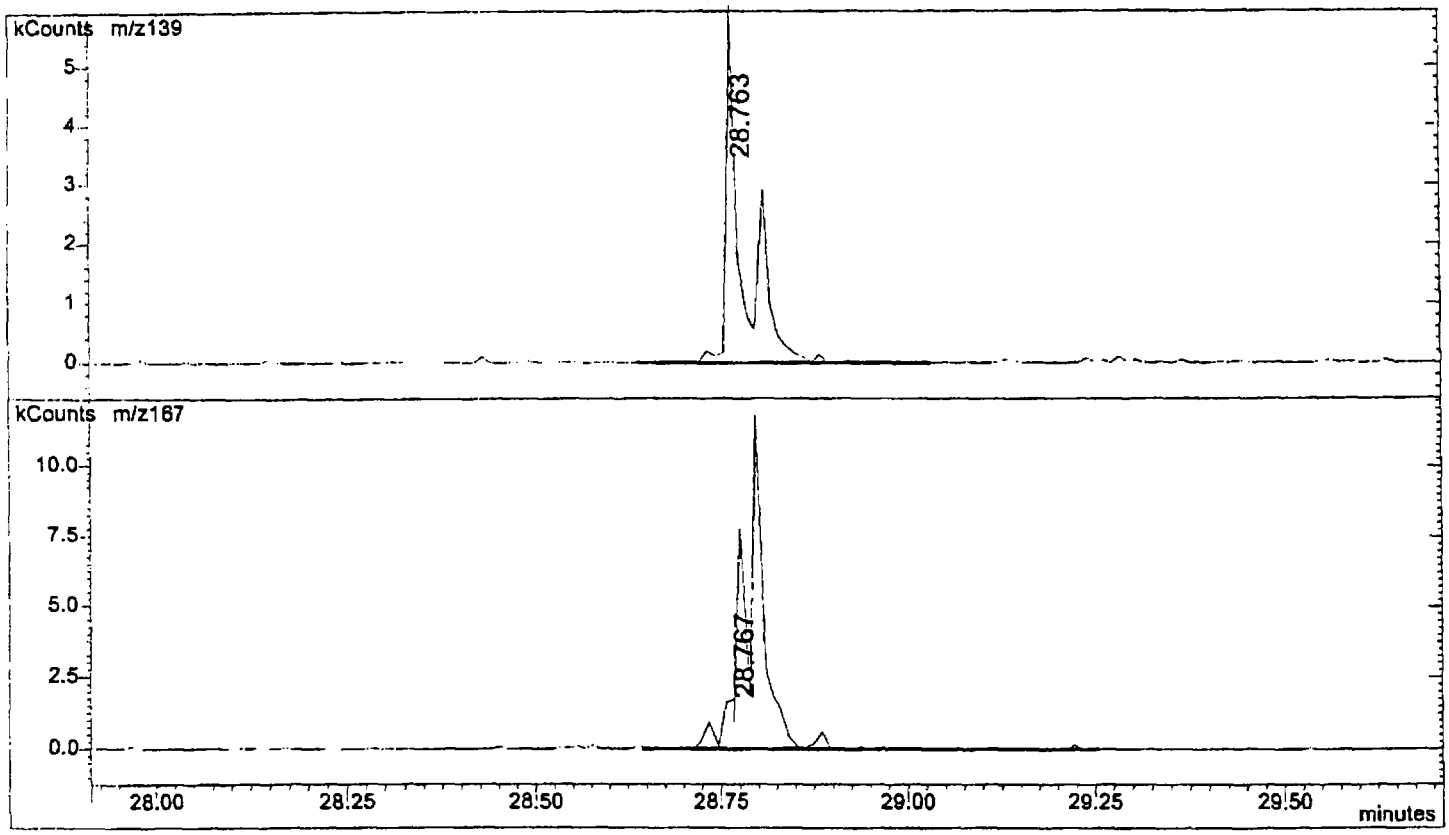
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.763 min.	28.812 min.	28.112 - 29.512 min.	366358	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.763 min.	2.48%	2.0%	0.6% - 3.4%	9072	Pass
167	28.767 min.	5.95%	4.6%	1.4% - 7.8%	21806	Pass

Concentration: 0.295 ug/mL



Sample: VX



Segment: 4 Time(min): 23.981-32.987 Scans: 1423-2282 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:17:47

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

ample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/16/02 4:44 PM
Vial:	3	Measurement Type:	Area
Data File:	c:\... \october\101602#1\xvx100302-3,0.46ug10-16-02002.sms		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

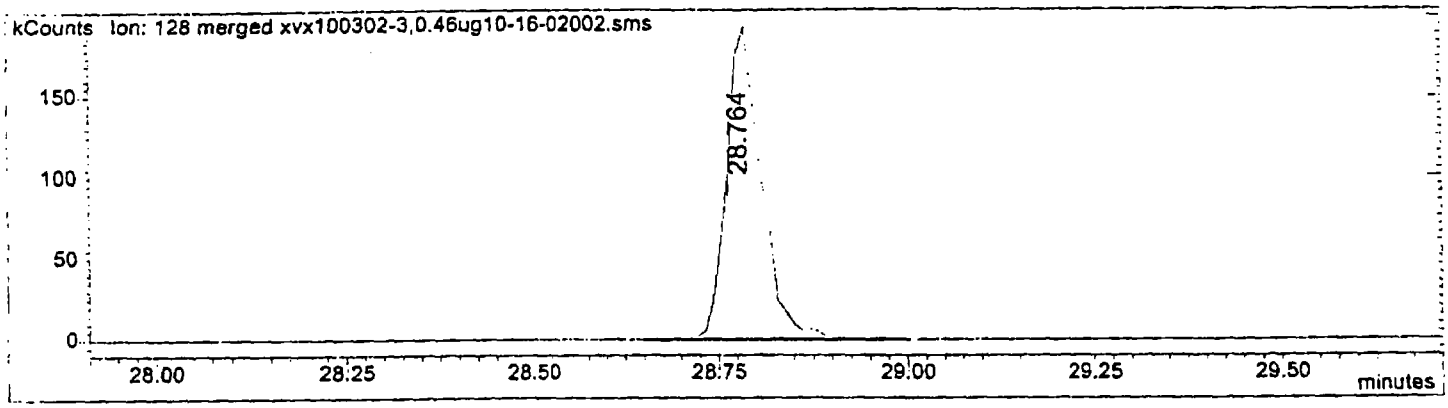
Y. Arthur
C31763 [illegible]
10-16-02

Analyte: VX

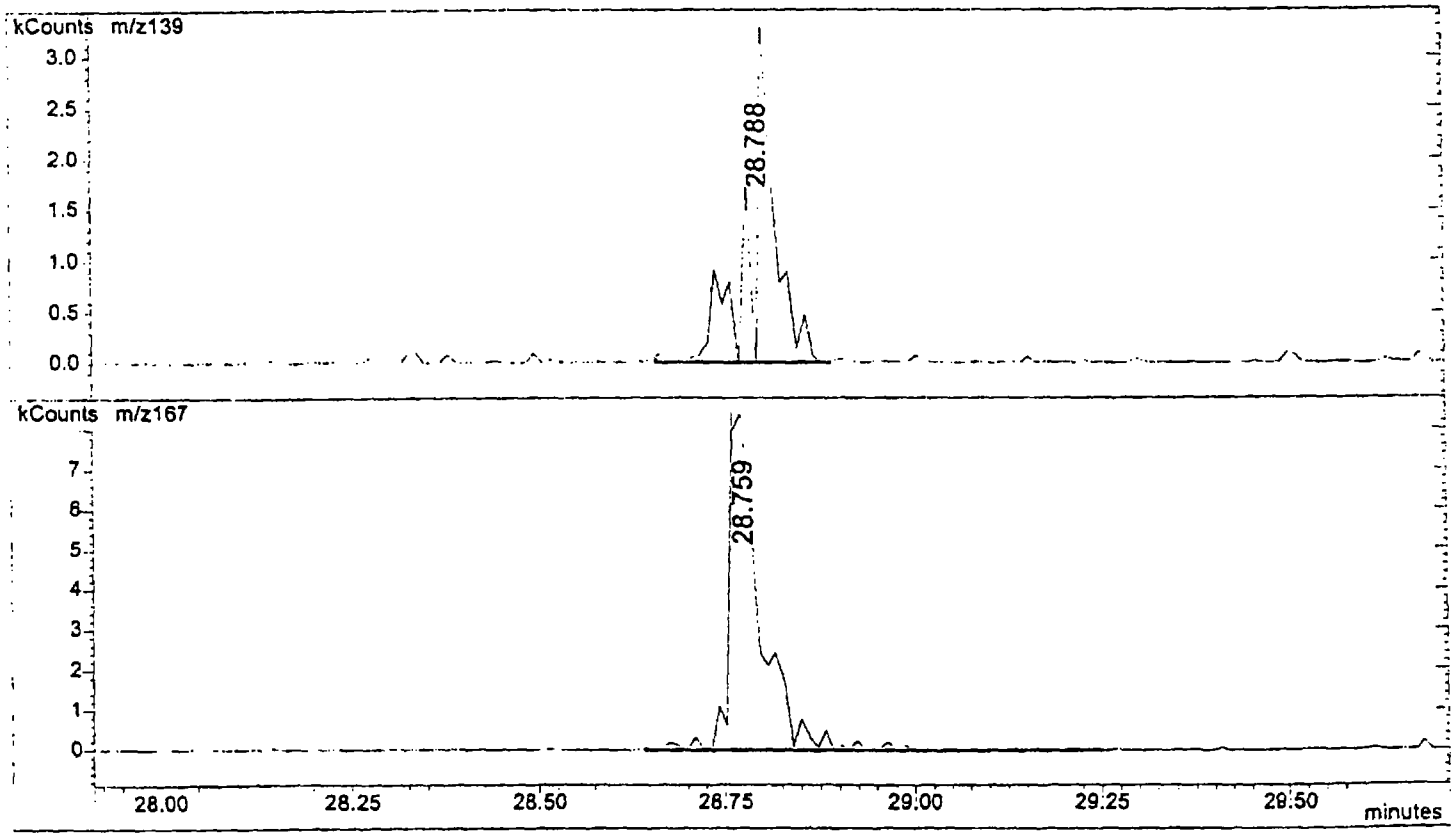
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.764 min.	28.812 min.	28.112 - 29.512 min.	628731	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.788 min.	1.22%	2.0%	0.6% - 3.4%	7644	Pass
167	28.759 min.	3.70%	4.6%	1.4% - 7.8%	23286	Pass

Concentration: 0.493 ug/mL



Sample: VX



Segment: 4 Time(min): 23.999-32.985 Scans: 1423-2282 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:19:58

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-1, 1.8ug10-1

Saturn 2000 No.1

ample ID:	XVX100302-1, 1.8ug	Acquisition Date:	10/16/02 6:27 PM
Vial:	6	Measurement Type:	Area
Data File:	c:\... \2002\october\101602#1\xvx100302-1, 1.8ug10-16-02.sms		
Inj. Sample Notes:	1.83ug/mLVX Std, Rtx-5MS:30M x 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans /sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

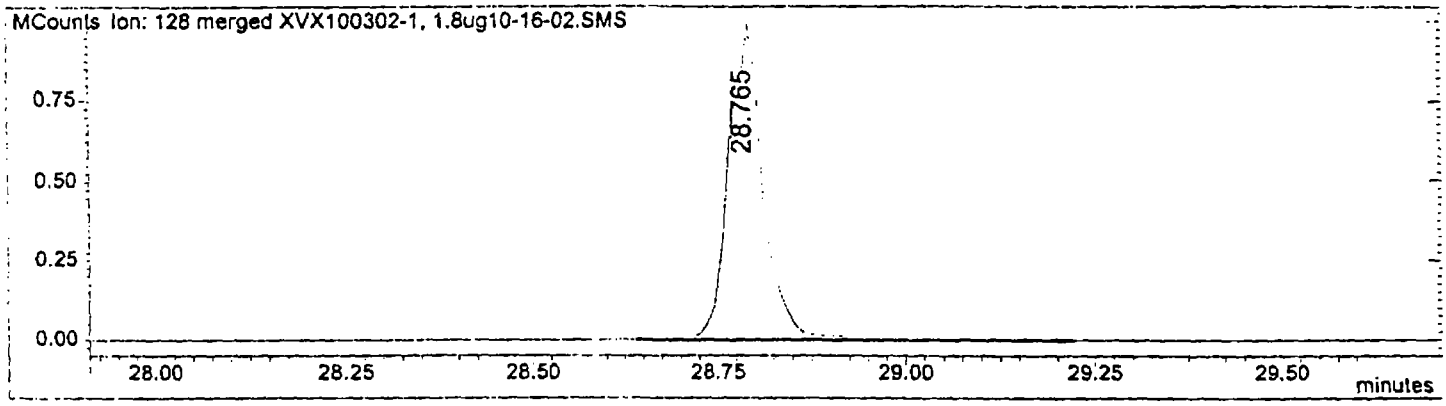
Y-Abthu
C 31763 Bulk P-6
10-16-02

Analyte: VX

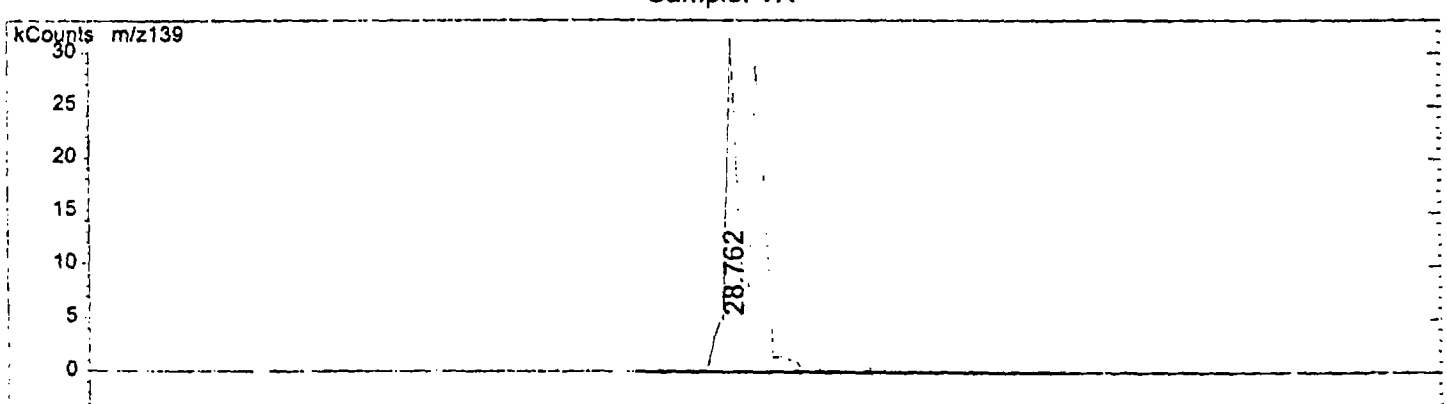
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.765 min.	28.812 min.	28.112 - 29.512 min.	3185279	Identified

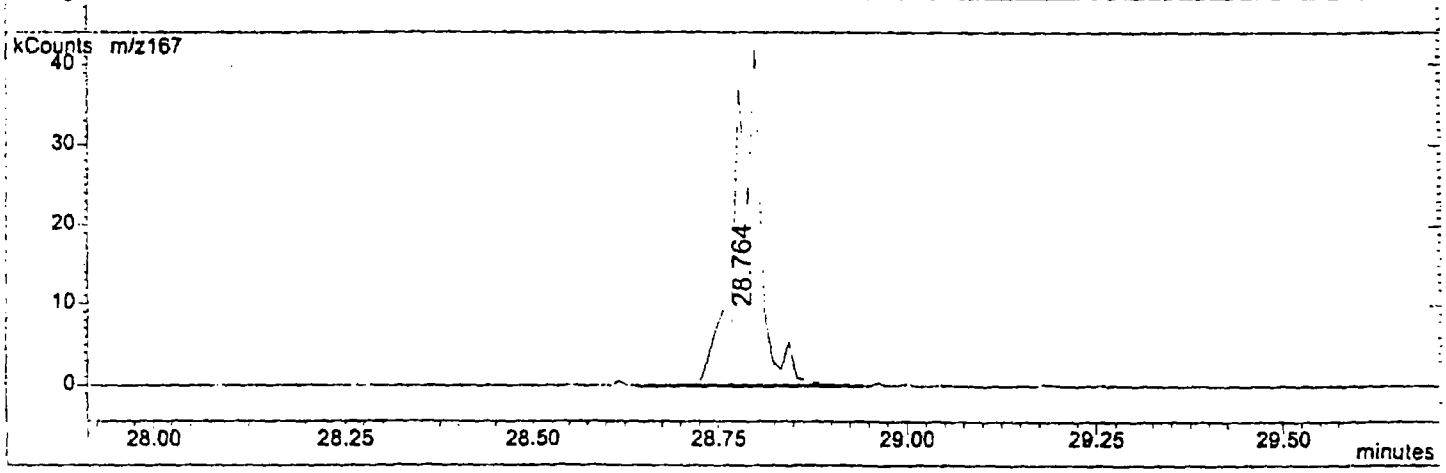
Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.762 min.	2.36%	2.0%	0.6% - 3.4%	75047	Pass
167	28.764 min.	3.20%	4.6%	1.4% - 7.8%	101796	Pass

Concentration: 1.863 ug/mL



Sample: VX





Segment: 4 Time(min): 23.998-32.982 Scans: 1423-2282 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:20:14

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-1, 1.8ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-1, 1.8ug	Acquisition Date:	10/16/02 7:12 PM
Vial:	6	Measurement Type:	Area
Data File:	c:\... \october\101602#1\xvx100302-1, 1.8ug10-16-02001.sms		
Inj. Sample Notes:	1.83ug/mLVX Std, Rtx-5MS:30M x 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans /sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

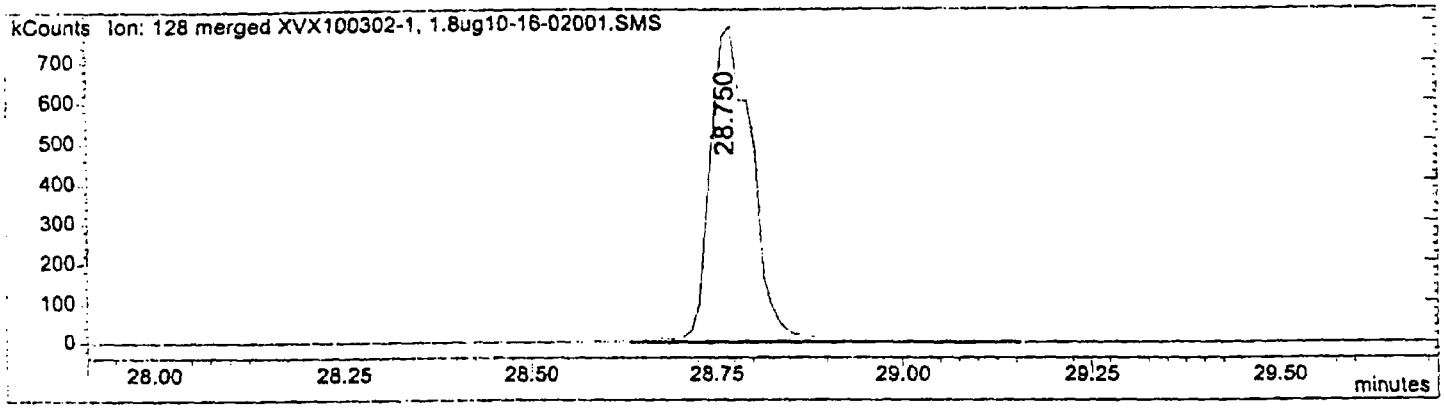
Y-Abthu
C31763 Bad P.
10-16-02

Analyte: VX

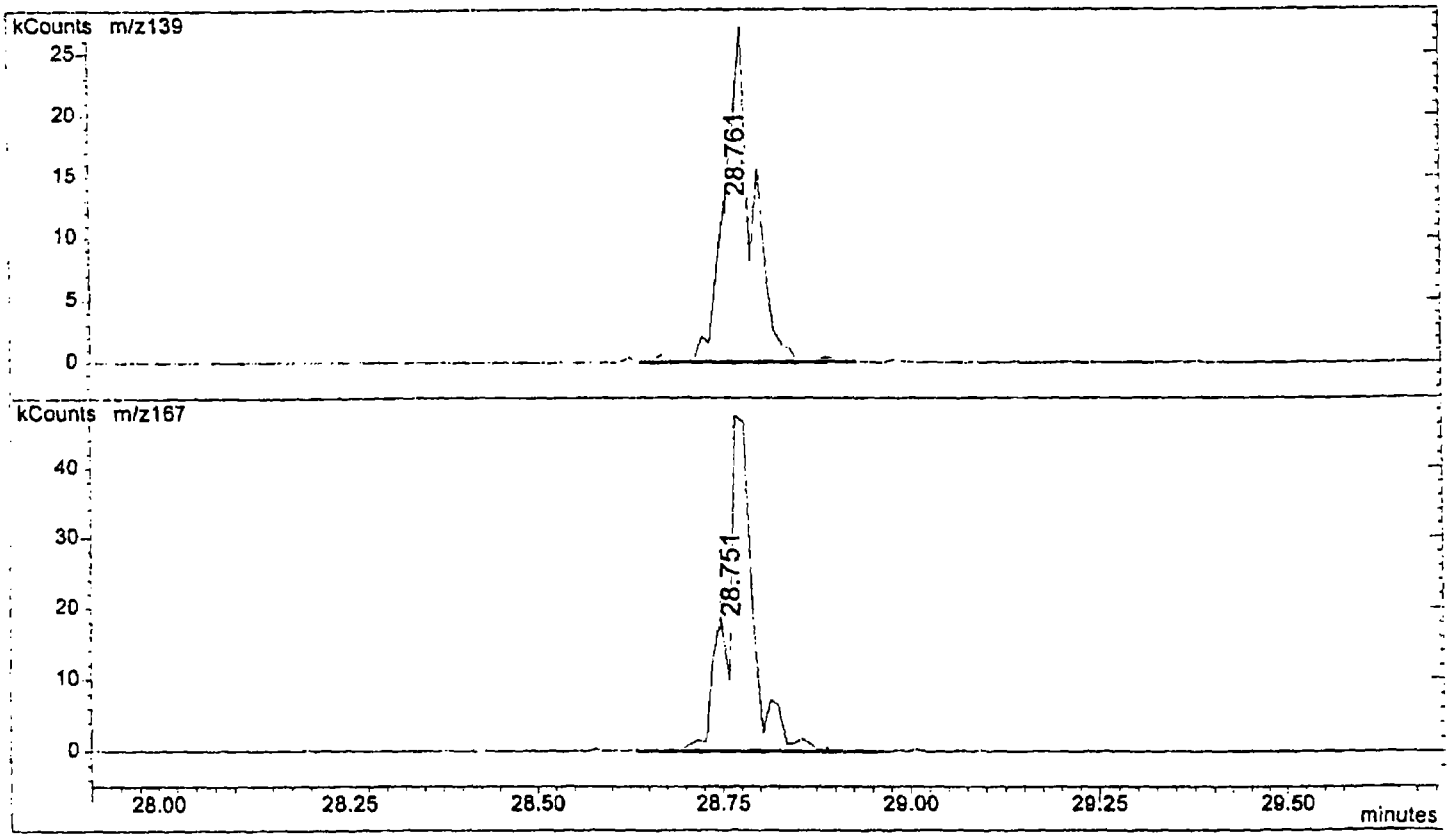
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.750 min.	28.812 min.	28.112 - 29.512 min.	3119859	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.761 min.	2.30%	2.0%	0.6% - 3.4%	71832	Pass
167	28.751 min.	3.84%	4.6%	1.4% - 7.8%	119774	Pass

Concentration: 1.835 ug/mL



Sample: VX



Segment: 4 Time(min): 23.985-32.987 Scans: 1423-2284 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:22:02

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-2,0.91ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-2,0.91ug	Acquisition Date:	10/16/02 7:57 PM
Vial:	7	Measurement Type:	Area
Data File:	c:\... \2002\october\101602#1\xvx100302-2,0.91ug10-16-02.sms		
Inj. Sample Notes:	0.913ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42V-EE;PIT@200usec;0.65scans /sec;noPP;m/z268.2;1750V;TS-RAP@170C;Sngl-GN		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

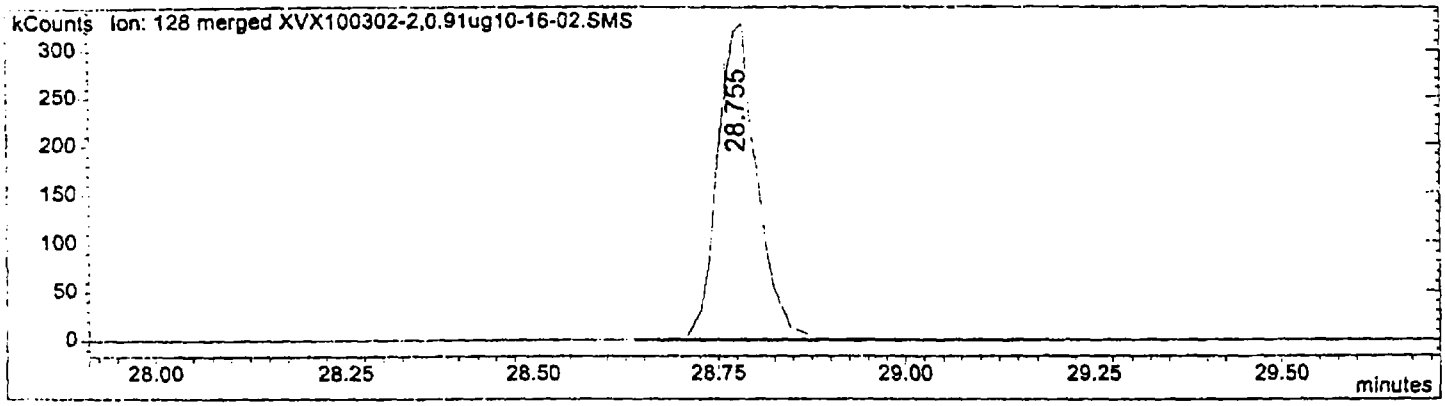
Y. Arthur
C31663 Bot P-6
10-16-02

Analyte: VX

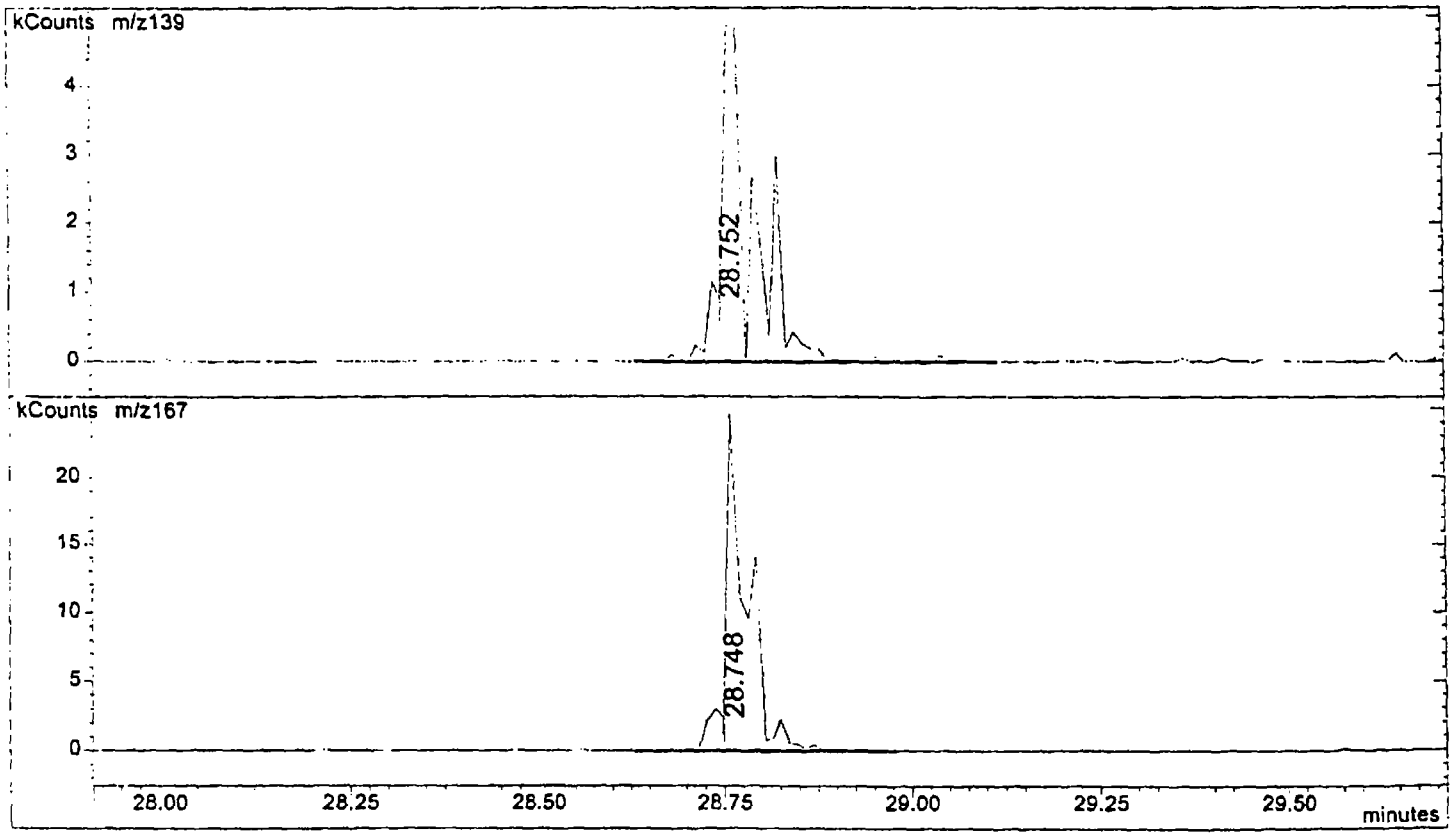
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.755 min.	28.812 min.	28.112 - 29.512 min.	1213277	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.752 min.	1.18%	2.0%	0.6% - 3.4%	14351	Pass
167	28.748 min.	4.08%	4.6%	1.4% - 7.8%	49459	Pass

Concentration: 0.876 ug/mL



Sample: VX



Segment: 4 Time(min): 23.992-32.981 Scans: 1422-2272 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:30:02

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/16/02 8:42 PM
Vial:	8	Measurement Type:	Area
Data File:	c:\... \october\101602#1\xvx100302-3,0.46ug10-16-02003.sms		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

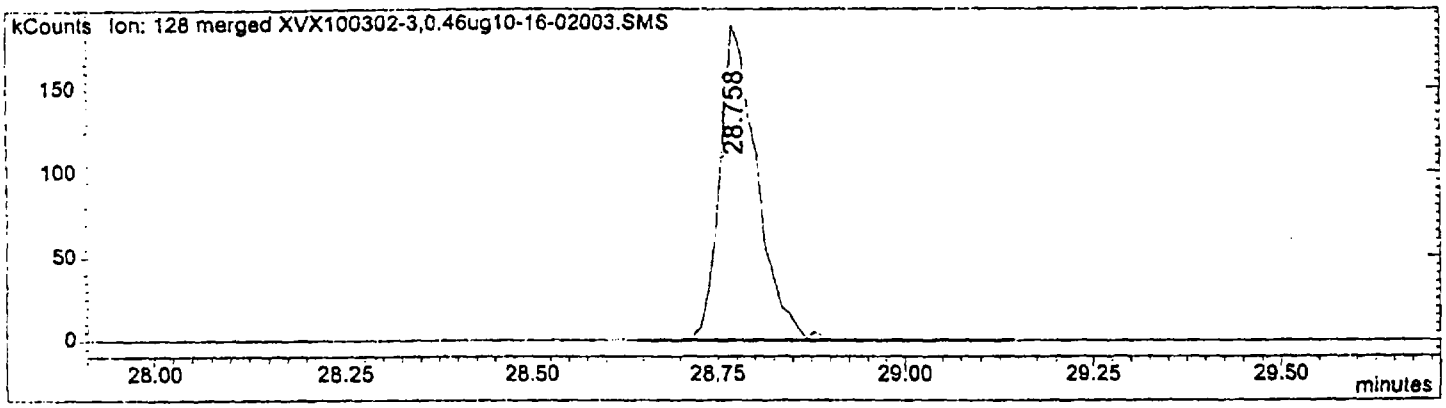
X-Abstr
C31763 Back P-
10-16-02

Analyte: VX

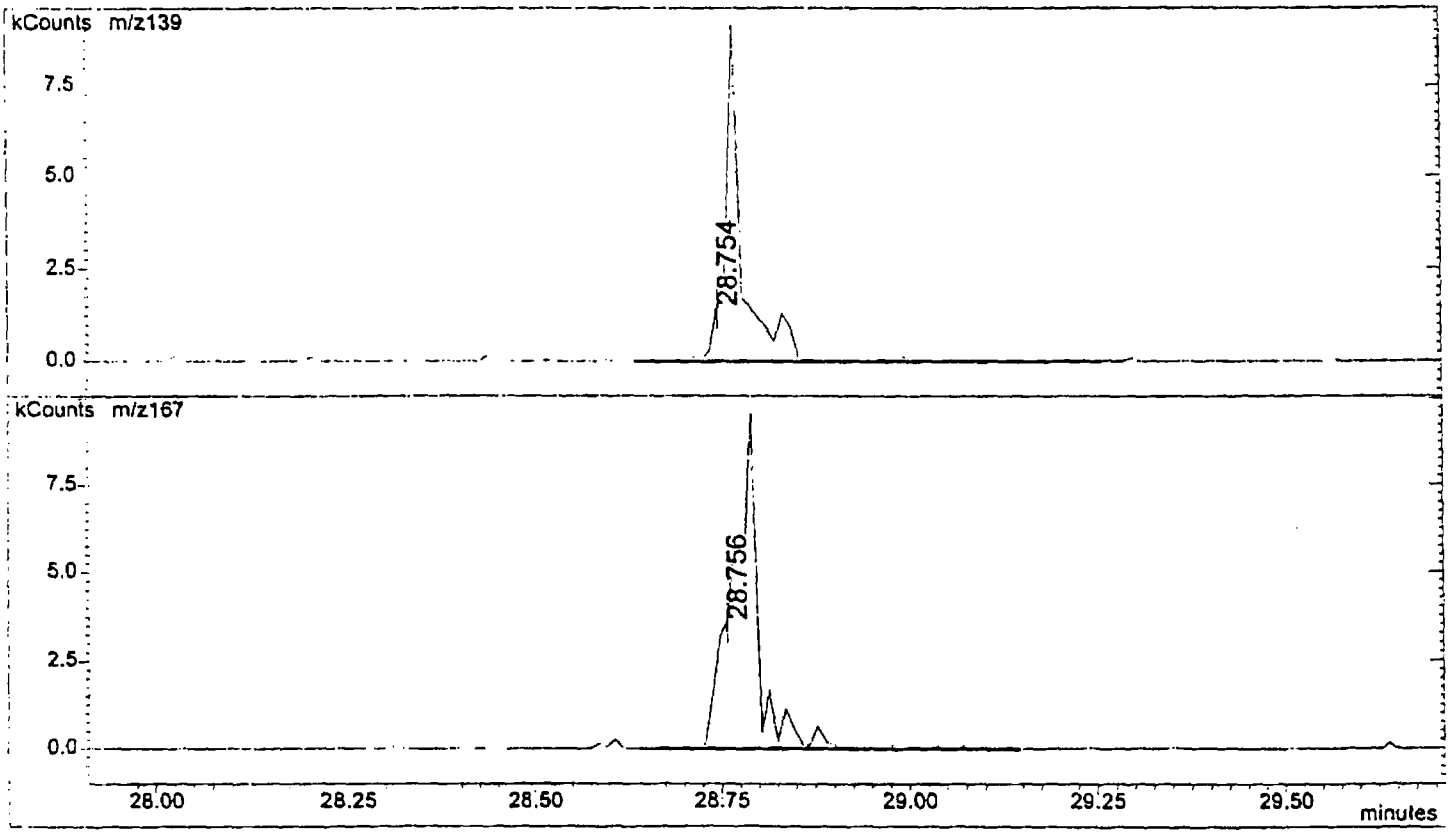
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.758 min.	28.812 min.	28.112 - 29.512 min.	647717	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.754 min.	2.24%	2.0%	0.6% - 3.4%	14531	Pass
167	28.756 min.	3.49%	4.6%	1.4% - 7.8%	22581	Pass

Concentration: 0.506 ug/mL



Sample: VX



Segment: 4 Time(min): 23.996-32.987 Scans: 1422-2272 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:30:19

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-4,0.18ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-4,0.18ug	Acquisition Date:	10/16/02 9:27 PM
Vial:	9	Measurement Type:	Area
Data File:	c:\... \2002\october\101602#1\xvx100302-4,0.18ug10-16-02.sms		
Inj. Sample Notes:	0.183ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

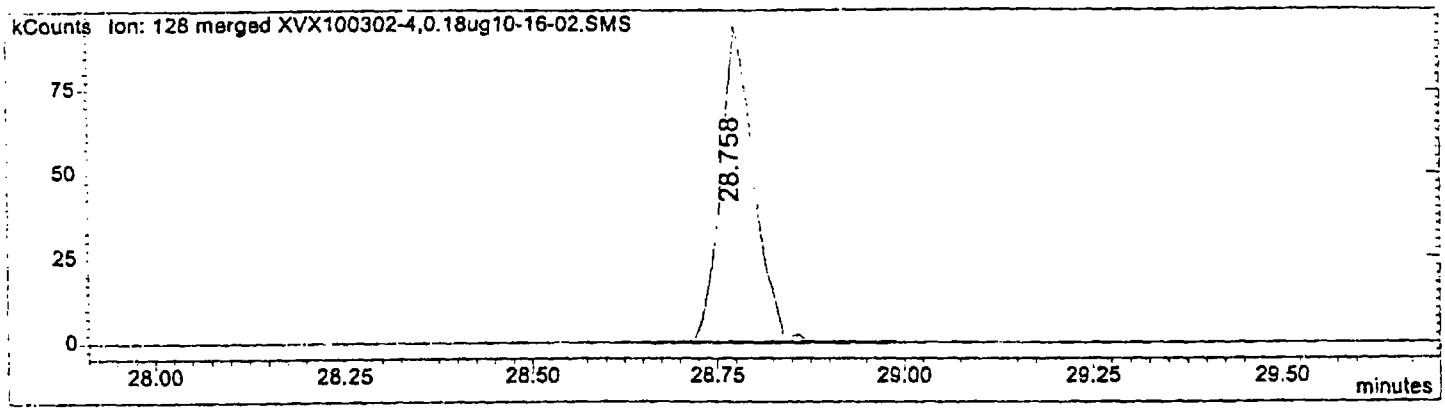
Y-Arthur
C 31763 Batch P-6
10-16-02

Analyte: VX

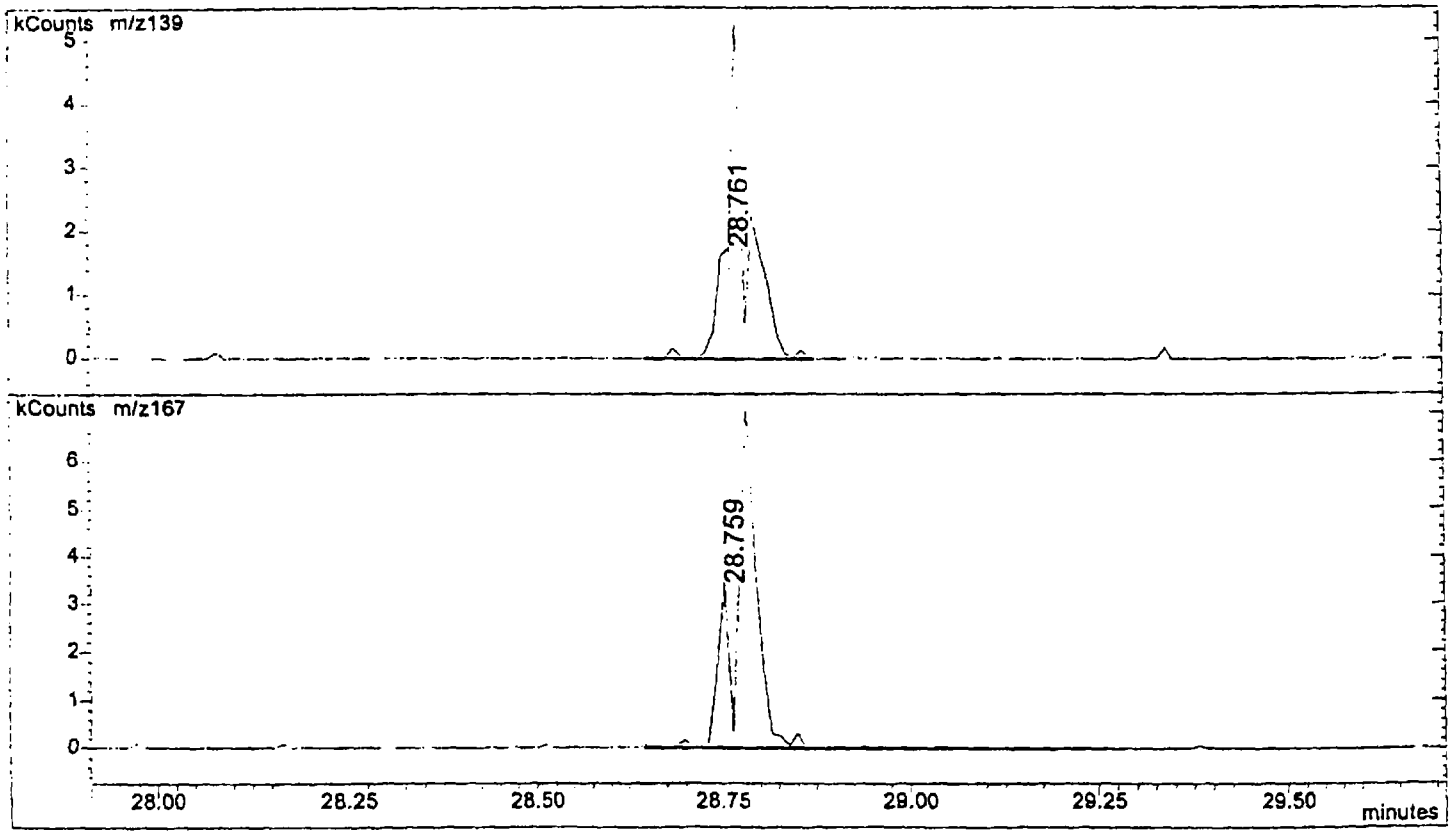
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.758 min.	28.812 min.	28.112 - 29.512 min.	294563	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.761 min.	3.36%	2.0%	0.6% - 3.4%	9900	Pass
167	28.759 min.	4.94%	4.6%	1.4% - 7.8%	14544	Pass

Concentration: 0.237 ug/mL



Sample: VX



Segment: 4 Time(min): 23.993-32.986 Scans: 1422-2275 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:30:39

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-5,.091ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-5,.091ug	Acquisition Date:	10/16/02 10:12 PM
Vial:	10	Measurement Type:	Area
Data File:	c:\... \2002\october\101602#1\xvx100302-5,.091ug10-16-02.sms		
Inj. Sample Notes:	0.091ug/mLVX Std Rtx-5Sil MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

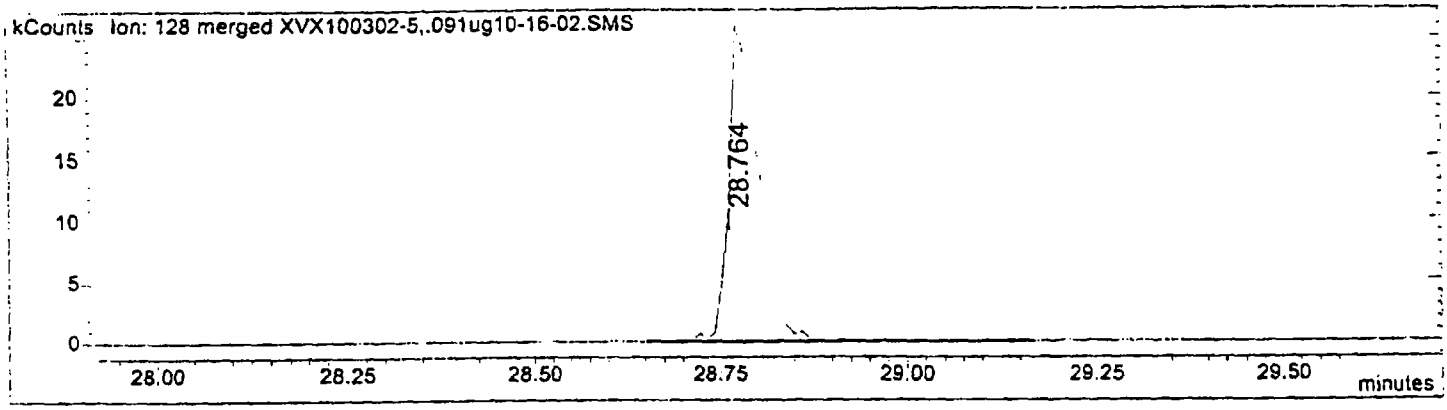
Y-Anthrax
C31763 Bot P-6
10-16-02

Analyte: VX

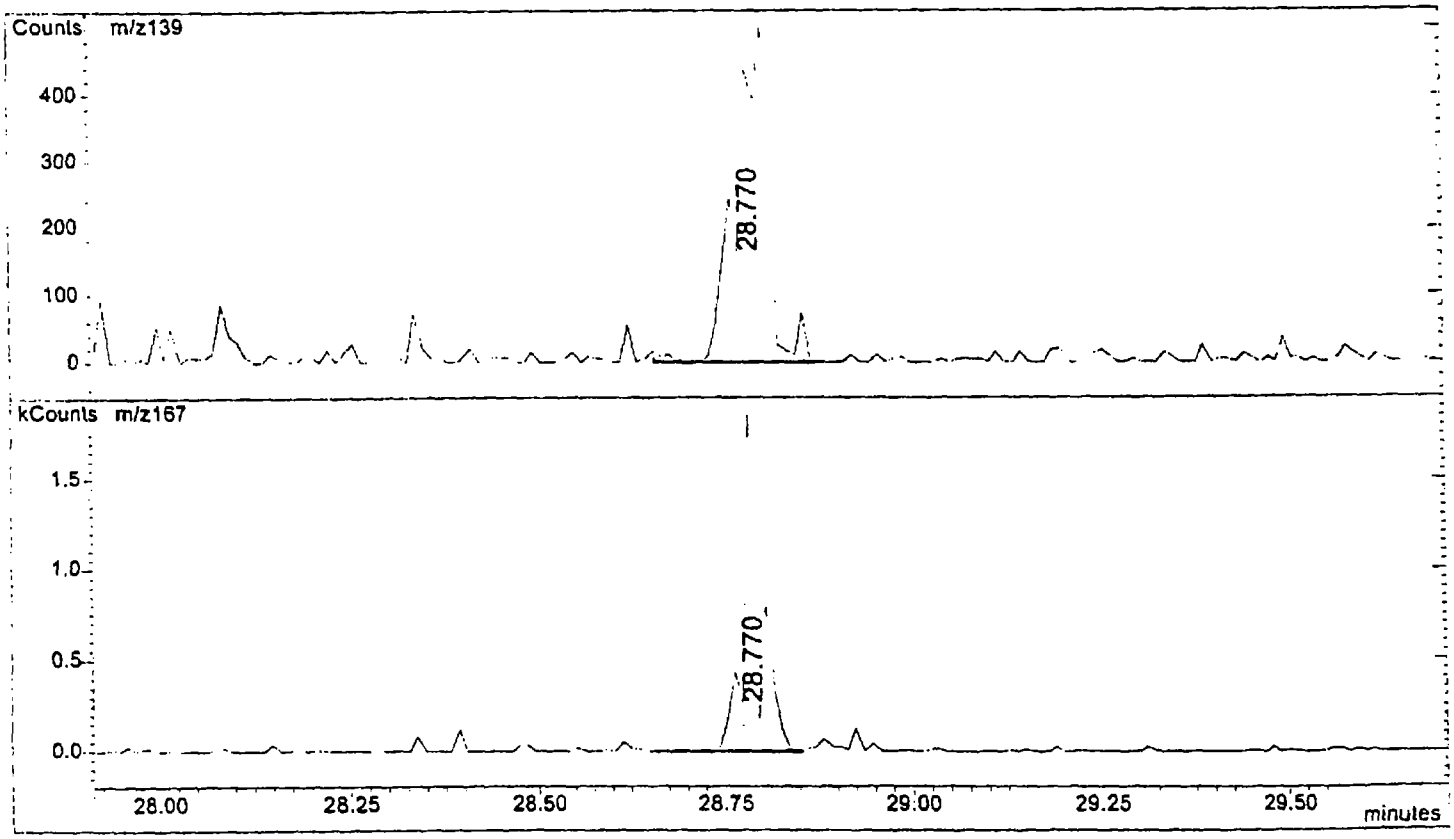
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.764 min.	28.812 min.	28.112 - 29.512 min.	73654	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.770 min.	2.10%	2.0%	0.6% - 3.4%	1545	Pass
167	28.770 min.	3.69%	4.6%	1.4% - 7.8%	2717	Pass

Concentration: 0.046 ug/mL



Sample: VX



Segment: 4 Time(min): 23.995-32.987 Scans: 1422-2274 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:30:52

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-6,.046ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-6,.046ug	Acquisition Date:	10/16/02 10:57 PM
Vial:	11	Measurement Type:	Area
Data File:	c:\... \2002\october\101602#1\xvx100302-6,.046ug10-16-02.sms		
Inj. Sample Notes:	0.046ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

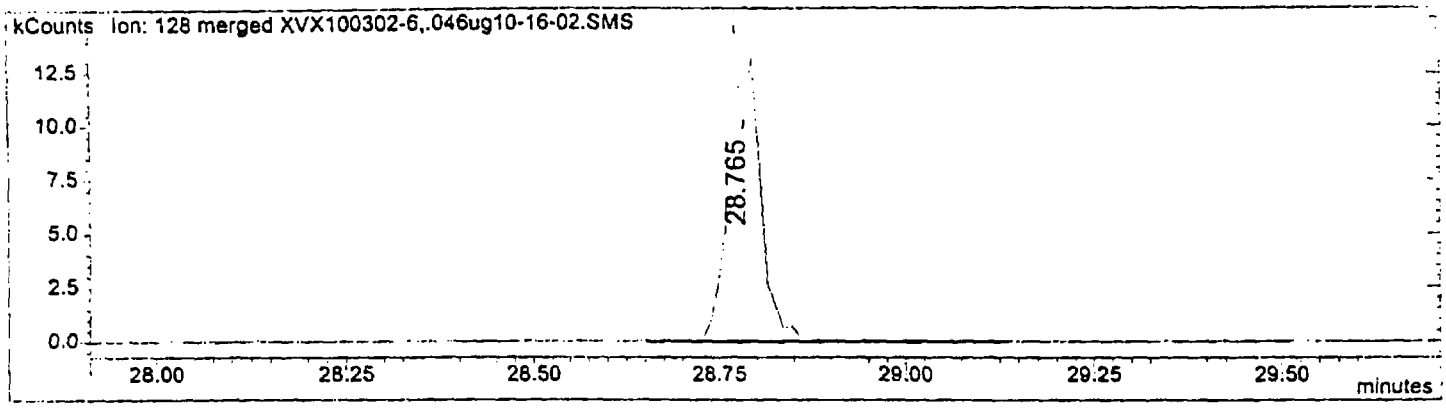
Y-Anthes
C31763 Book P-1
10-16-02

Analyte: VX

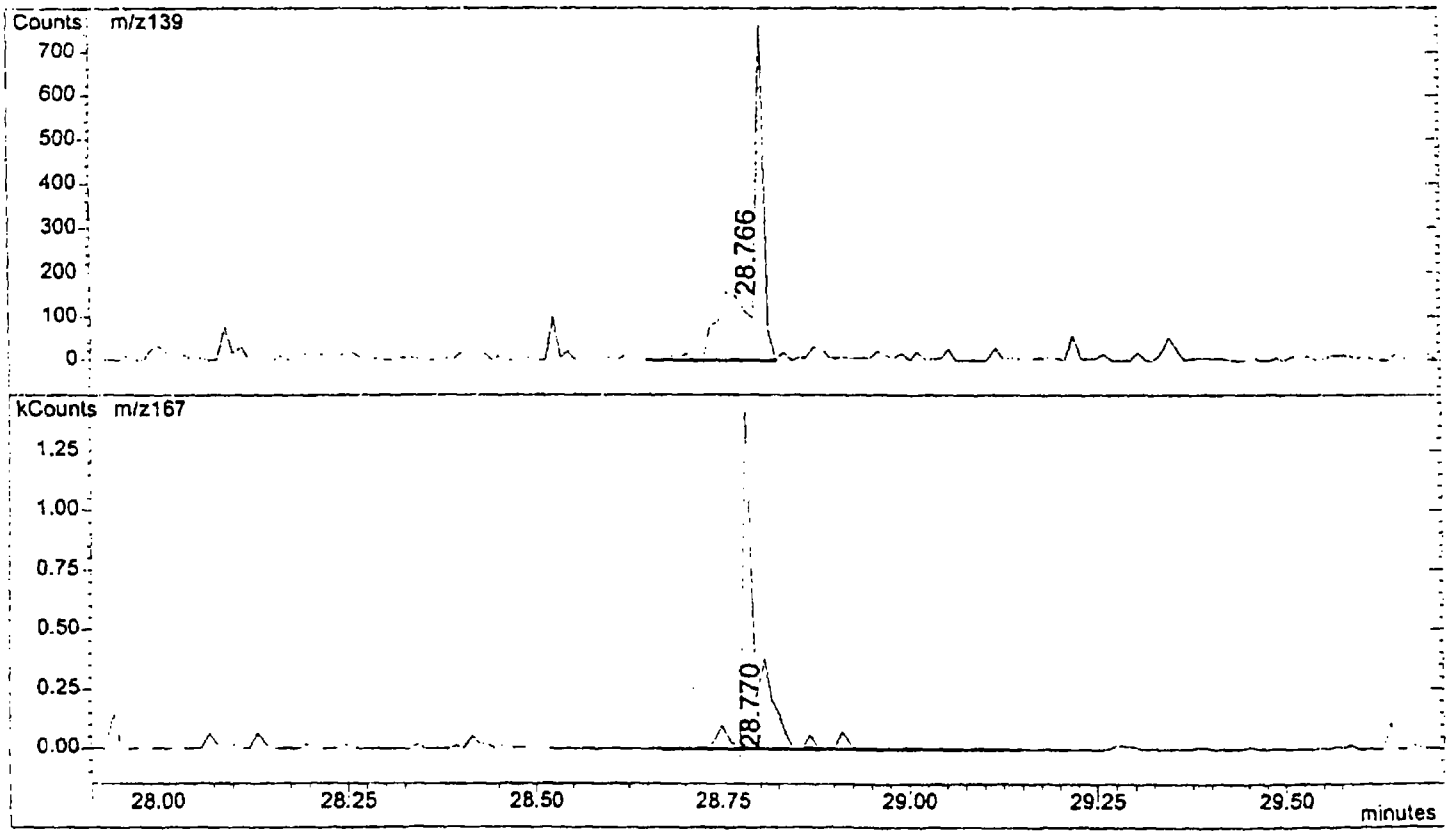
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.765 min.	28.812 min.	28.112 - 29.512 min.	40986	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.766 min.	2.45%	2.0%	0.6% - 3.4%	1006	Pass
167	28.770 min.	4.39%	4.6%	1.4% - 7.8%	1798	Pass

Concentration: 0.016 ug/mL



Sample: VX



Segment: 4 Time(min): 23.996-32.982 Scans: 1422-2276 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:31:10

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

ample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/16/02 11:42 PM
Vial:	8	Measurement Type:	Area
Data File:	c:\... \october\101602#1\xvx100302-3,0.46ug10-16-02004.sms		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

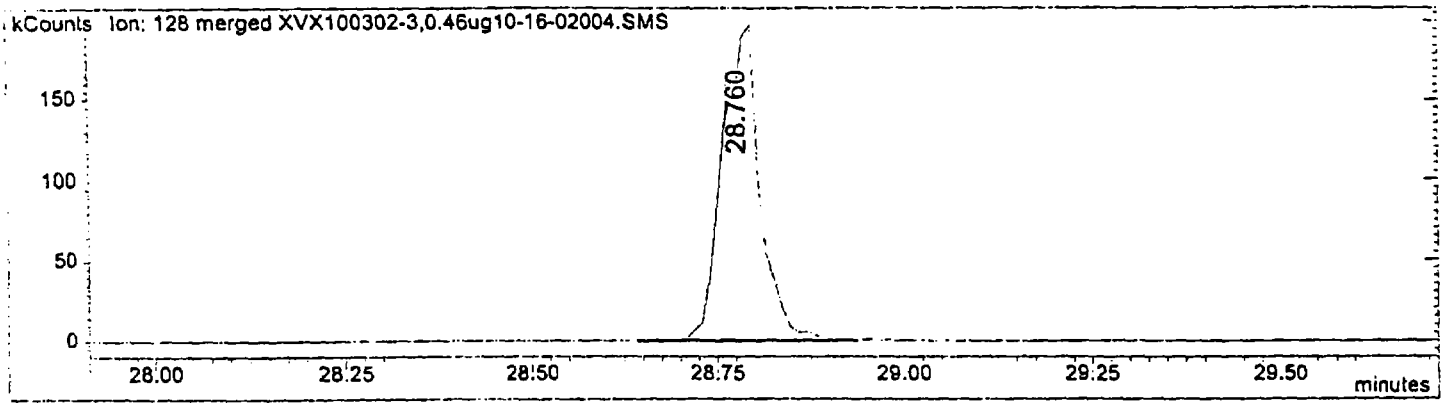
Y. Abrhe
C31763 Book P6
10-16-02

Analyte: VX

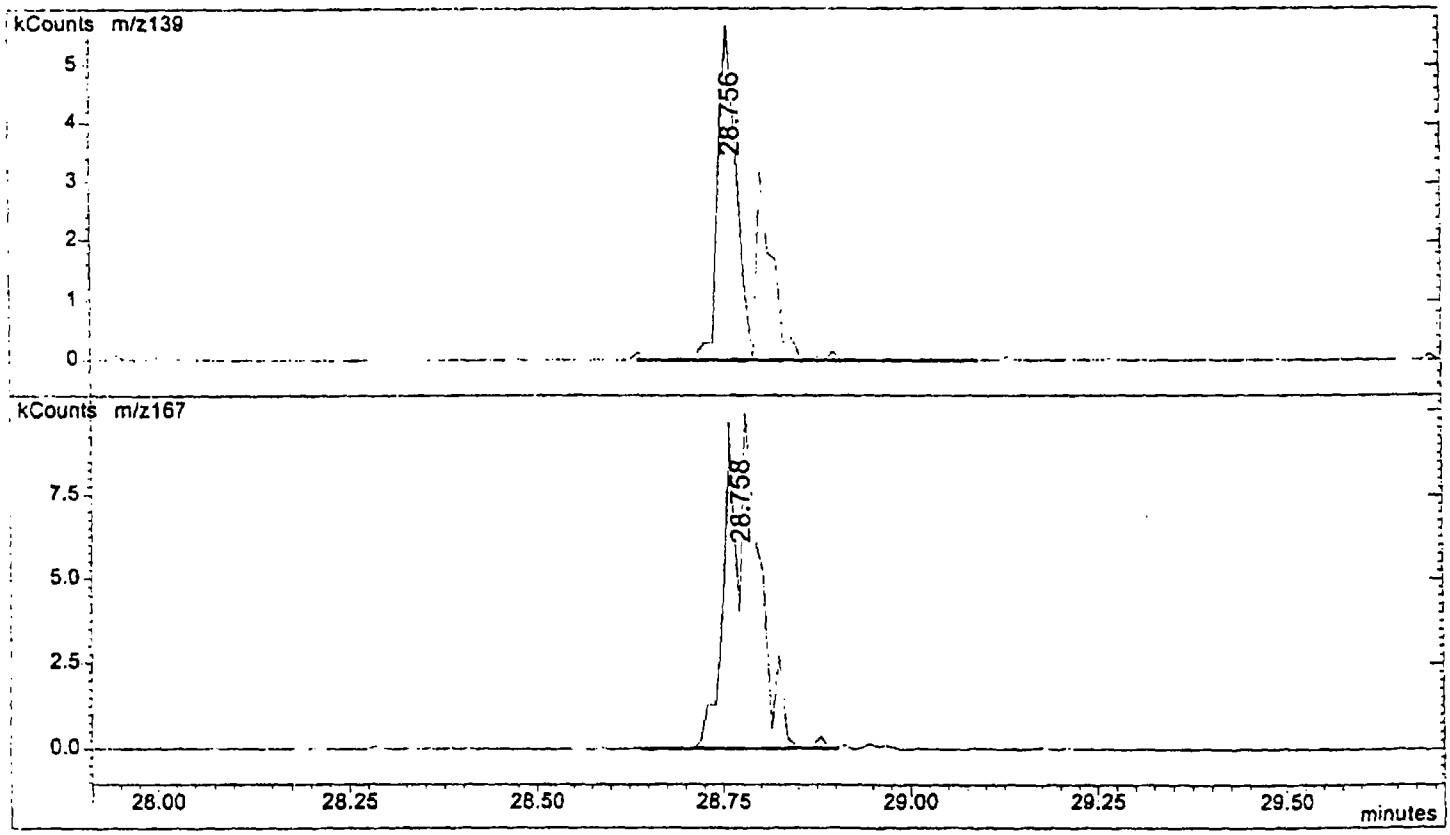
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.760 min.	28.812 min.	28.112 - 29.512 min.	688952	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.756 min.	2.11%	2.0%	0.6% - 3.4%	14510	Pass
167	28.758 min.	4.20%	4.6%	1.4% - 7.8%	28934	Pass

Concentration: 0.536 ug/mL



Sample: VX



Segment: 4 Time(min): 23.991-32.982 Scans: 1422-2272 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:31:28

Ion Ratio Report for Target Compound #1 from VX-101502-US-1-10-17-02.SMS

Saturn 2000 No.1

ample ID:	VX-101502-US-1-	Acquisition Date:	10/17/02 12:27 AM
Vial:	12	Measurement Type:	Area
Data File:	c:\... \2002\october\101602#1\vx-101502-us-1-10-17-02.sms		
Inj. Sample Notes:	Method Spike: 91ng/g VX Spike into 5g NaSO4; DEAET added pr e-analysis; Rtx5MS;S-TRAP;EM@1750V		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

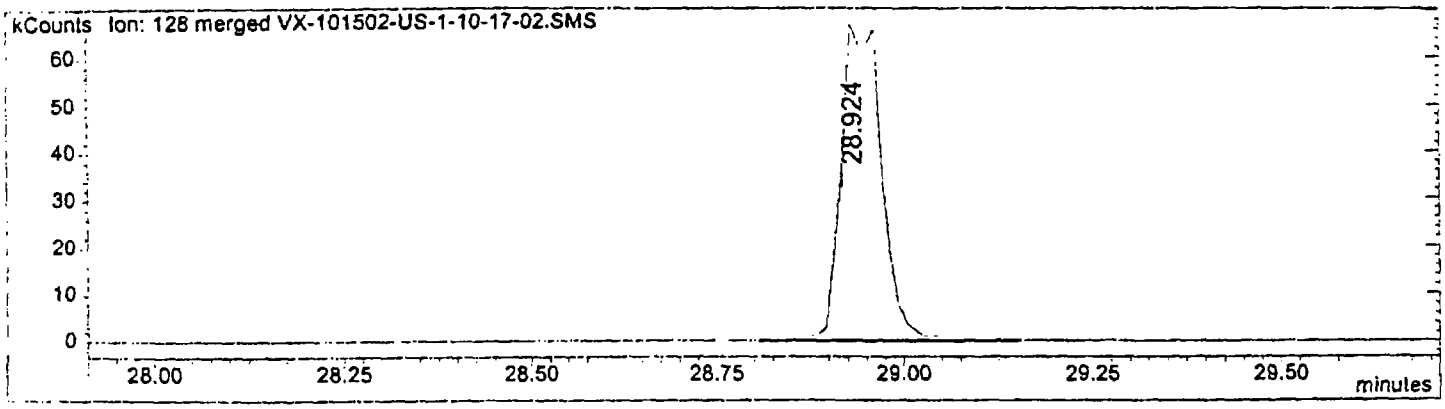
Y-Abitha
C31763 Bulk P.L
10-16-02

Analyte: VX

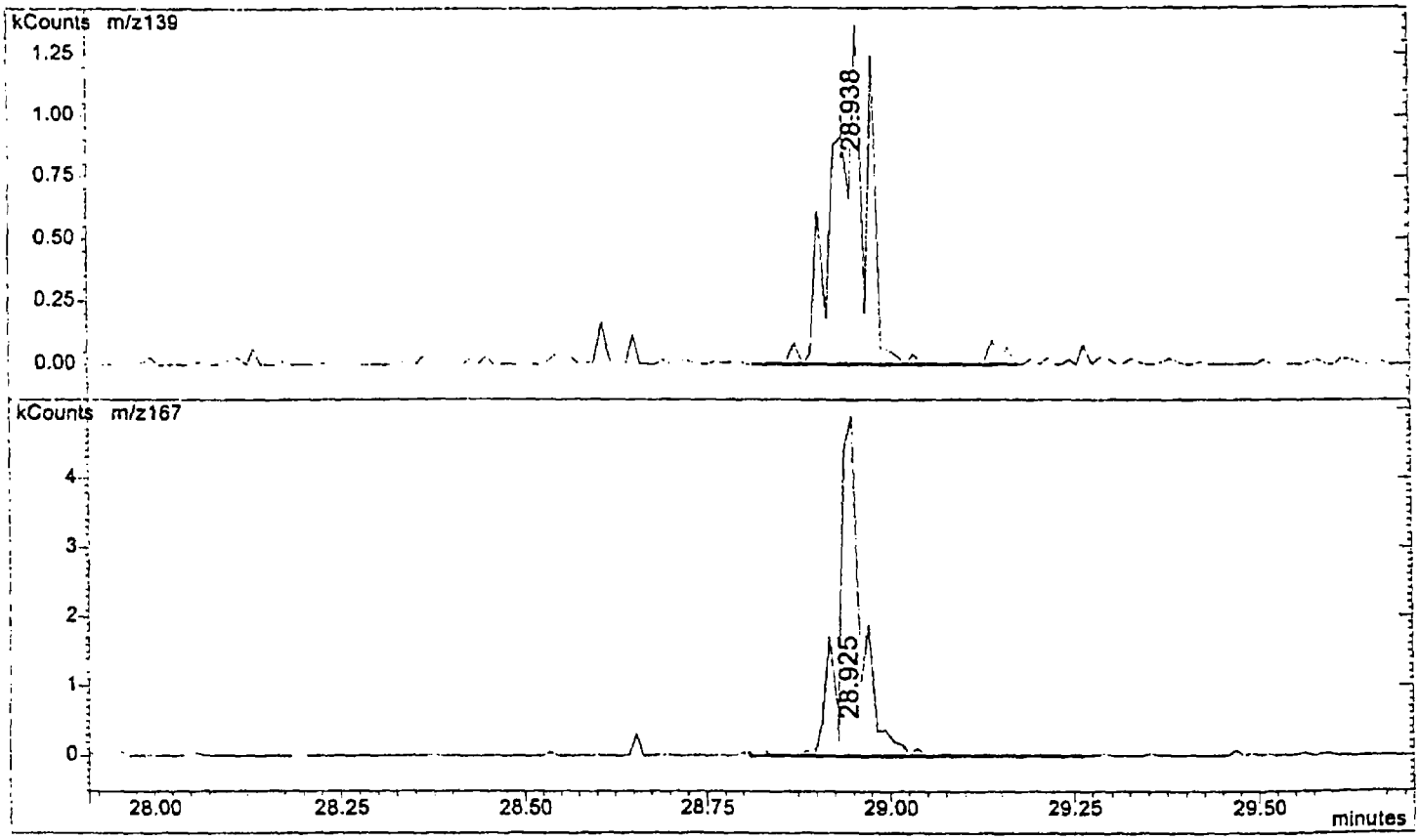
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.924 min.	28.812 min.	28.112 - 29.512 min.	249564	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.938 min.	1.74%	2.0%	0.6% - 3.4%	4345	Pass
167	28.925 min.	4.21%	4.6%	1.4% - 7.8%	10507	Pass

Concentration: 0.200 ug/mL



Sample: VX



Segment: 4 Time(min): 23.987-32.987 Scans: 1421-2271 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:31:44

Ion Ratio Report for Target Compound #1 from VX-101502-US-2-10-17-02.SMS

Saturn 2000 No.1

ample ID:	VX-101502-US-2-	Acquisition Date:	10/17/02 1:12 AM
Vial:	13	Measurement Type:	Area
Data File:	c:\... \2002\october\101602#1\vx-101502-us-2-10-17-02.sms		
Inj. Sample Notes:	Method Spike: 91ng/g VX Spike into 5g NaSO4; DEAET added pr e-analysis; Rtx5MS;S-TRAP;EM@1750V		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

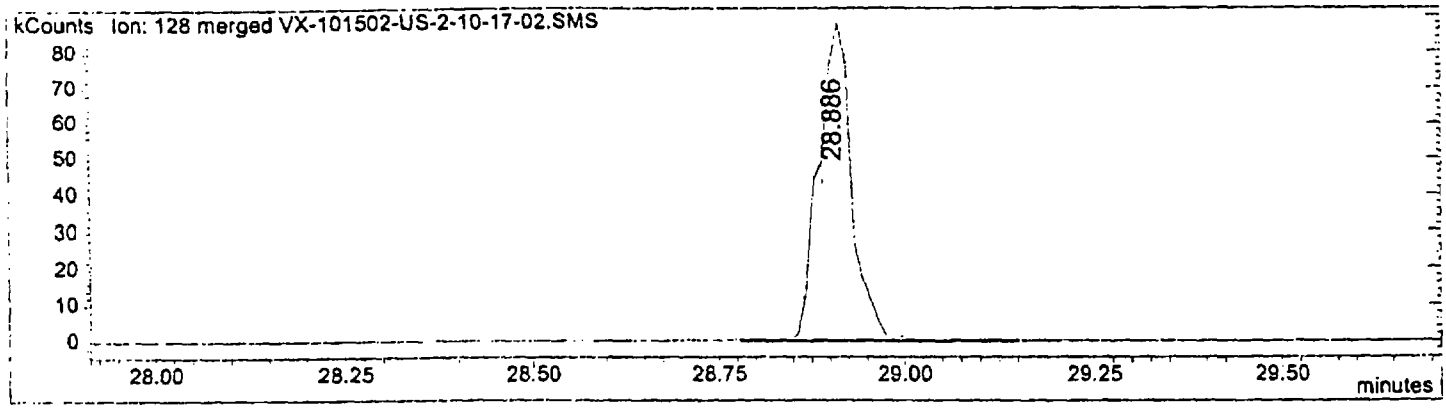
Y-Abstract
C31763 Back P.
10-16-02

Analyte: VX

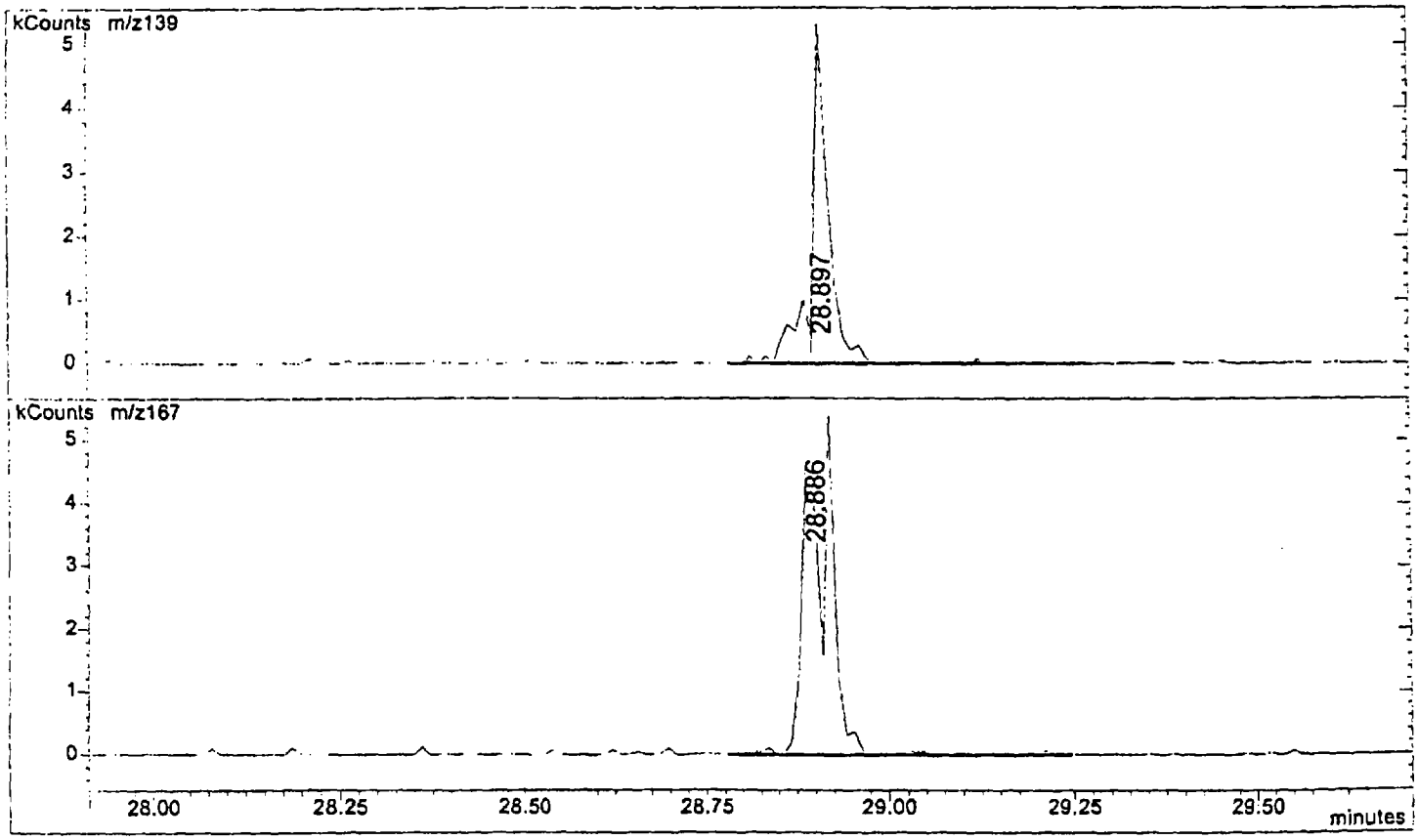
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.886 min.	28.812 min.	28.112 - 29.512 min.	272136	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.897 min.	3.25%	2.0%	0.6% - 3.4%	8849	Pass
167	28.886 min.	4.58%	4.6%	1.4% - 7.8%	12457	Pass

Concentration: 0.219 ug/mL



Sample: VX



Segment: 4 Time(min): 23.978-32.986 Scans: 1421-2269 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:32:17

Ion Ratio Report for Target Compound #1 from vx-101502-us-3d-10-17-02.sms

Saturn 2000 No.1

ample ID:	VX-101502-US-3-	Acquisition Date:	10/17/02 1:57 AM
Vial:	4	Measurement Type:	Area
Data File:	c:\... \2002\october\101602#1\vx-101502-us-3d-10-17-02.sms		
Inj. Sample Notes:	JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extr action, no HAE cleanup; Initial Volume = 5mL, diluted 1:10 w /CH2Cl2, Rtx5MS;S-TRAP;EM@1750V		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

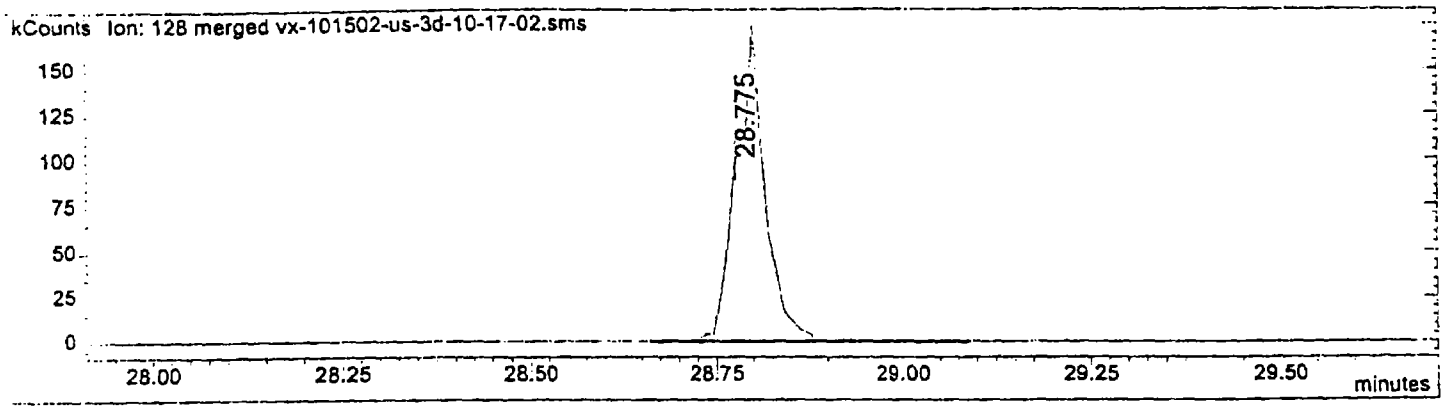
Y. Abdha
C31763 Bach P-6
10-16-02

Analyte: VX

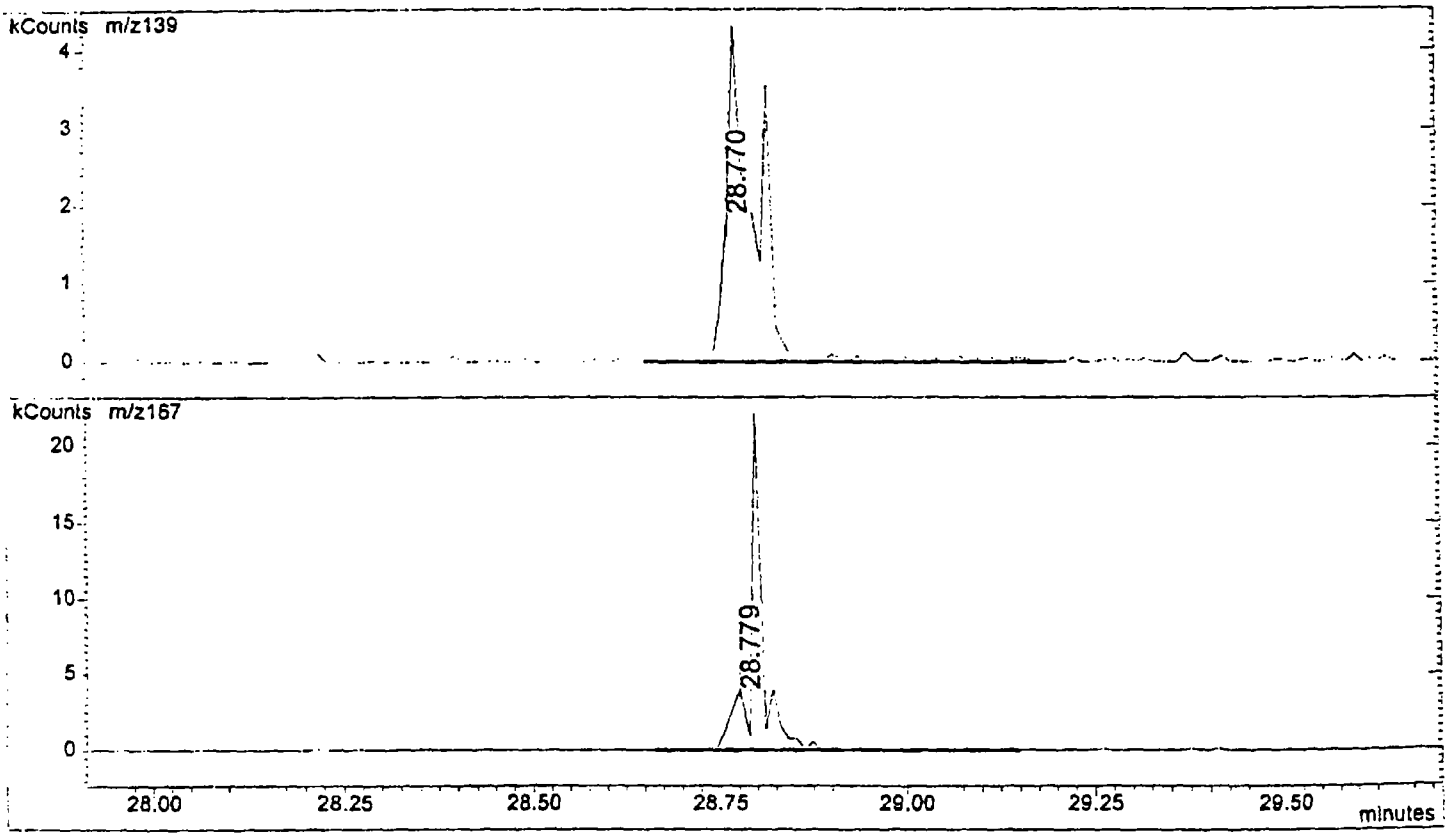
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.775 min.	28.812 min.	28.112 - 29.512 min.	469122	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.770 min.	2.33%	2.0%	0.6% - 3.4%	10945	Pass
167	28.779 min.	5.55%	4.6%	1.4% - 7.8%	26013	Pass

Concentration: 0.375 ug/mL



Sample: VX



Segment: 4 Time(min): 23.996-32.988 Scans: 1422-2270 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:32:47

Ion Ratio Report for Target Compound #1 from vx-101502-us-4d-10-17-02.sms

Saturn 2000 No.1

ample ID:	VX-101502-US-4-	Acquisition Date:	10/17/02 2:42 AM
Vial:	5	Measurement Type:	Area
Data File:	c:\... \2002\october\101602#1\vx-101502-us-4d-10-17-02.sms		
Inj. Sample Notes:	JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extraction, no HAE cleanup; Initial Volume = 5mL, diluted 1:10 w /CH2Cl2, Rtx5MS;S-TRAP;EM@1750V		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

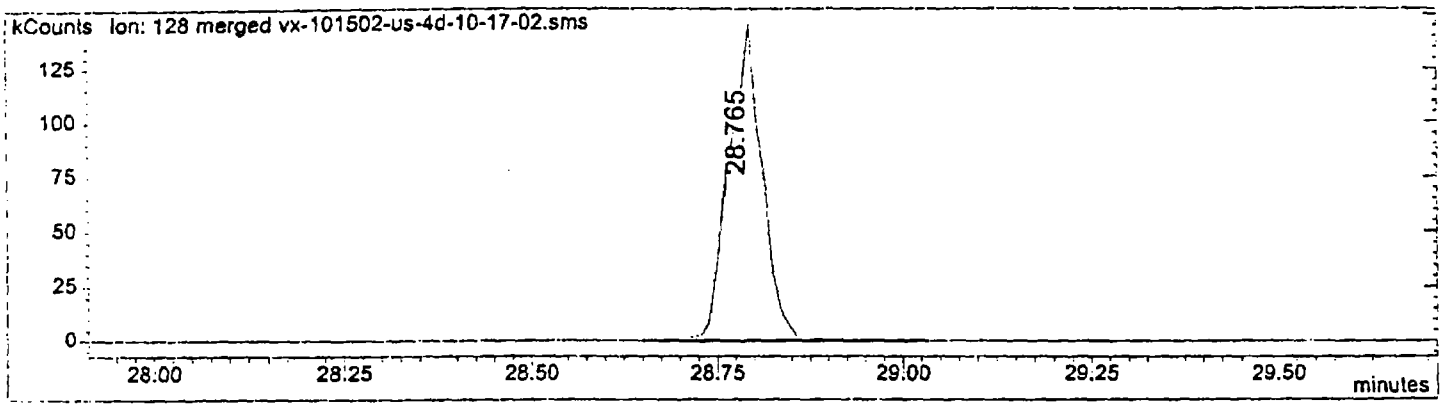
X. Arthur
C31763 Bulp. b
10-16-02

Analyte: VX

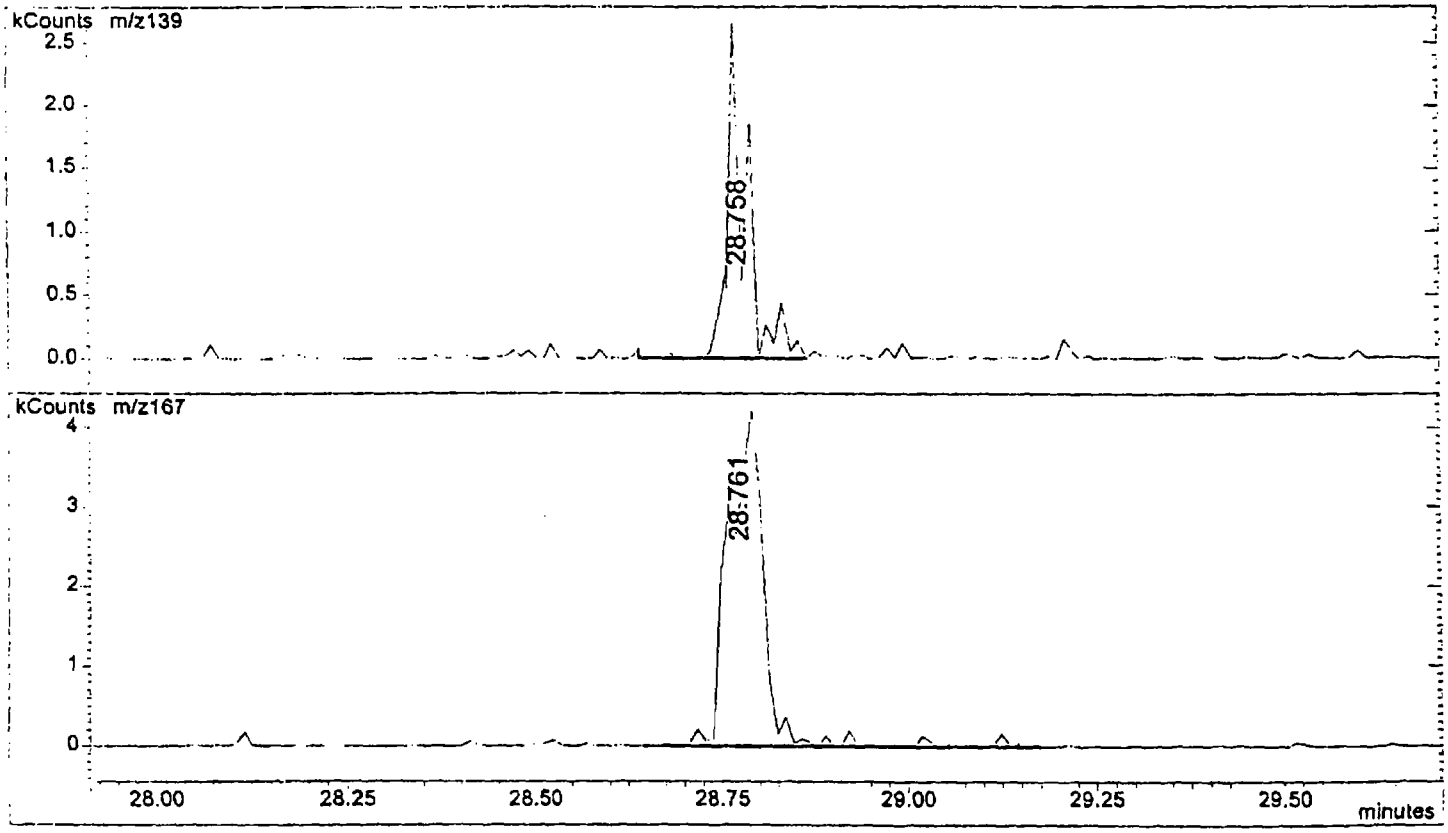
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.765 min.	28.812 min.	28.112 - 29.512 min.	455491	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.758 min.	1.04%	2.0%	0.6% - 3.4%	4721	Pass
167	28.761 min.	2.94%	4.6%	1.4% - 7.8%	13394	Pass

Concentration: 0.365 ug/mL



Sample: VX



Segment: 4 Time(min): 23.997-32.986 Scans: 1422-2271 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:52:52

Ion Ratio Report for Target Compound #1 from ch2cl2 blank-10-17-02.sms

Saturn 2000 No.1

ample ID:	CH2Cl2 Blank	Acquisition Date:	10/17/02 3:27 AM
al:	0	Measurement Type:	Area
Data File:	c:\... \data\2002\october\101602#1\ch2cl2 blank-10-17-02.sms		
Inj. Sample Notes:	CH2Cl2 Blank, Rtx-5MS:30Mx0.25mm ID, 1.0um; 100C,5min - 240 C @5C/m-280C @25C/m,5min;0.42VEE;PIT@200usec; 0.65scans/sec; noPP;m/z268.2;1750V; S-TRAP@170C		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

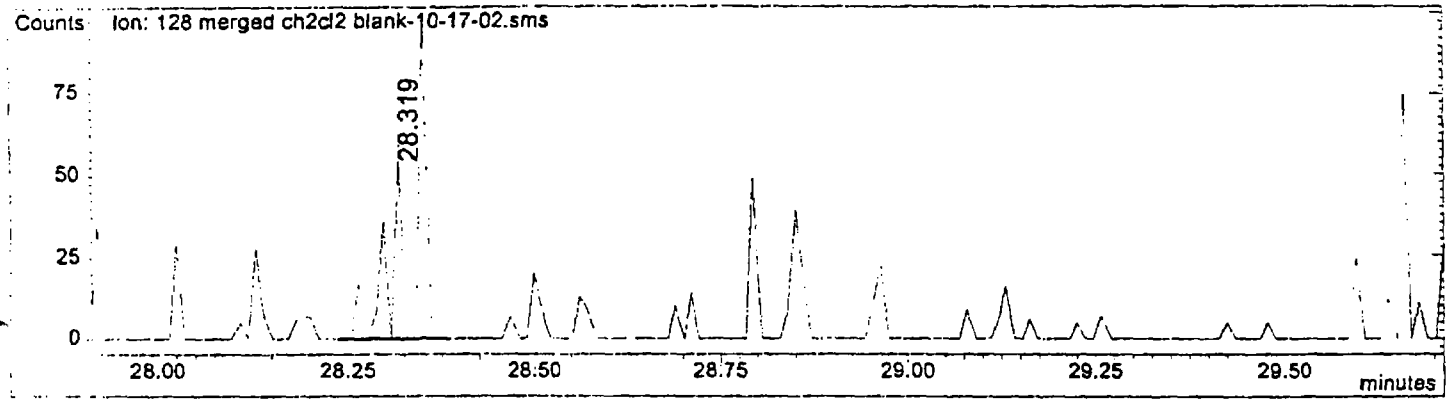
Y-Abstract
C31763 Bad P.
10-16-02

Analyte: VX

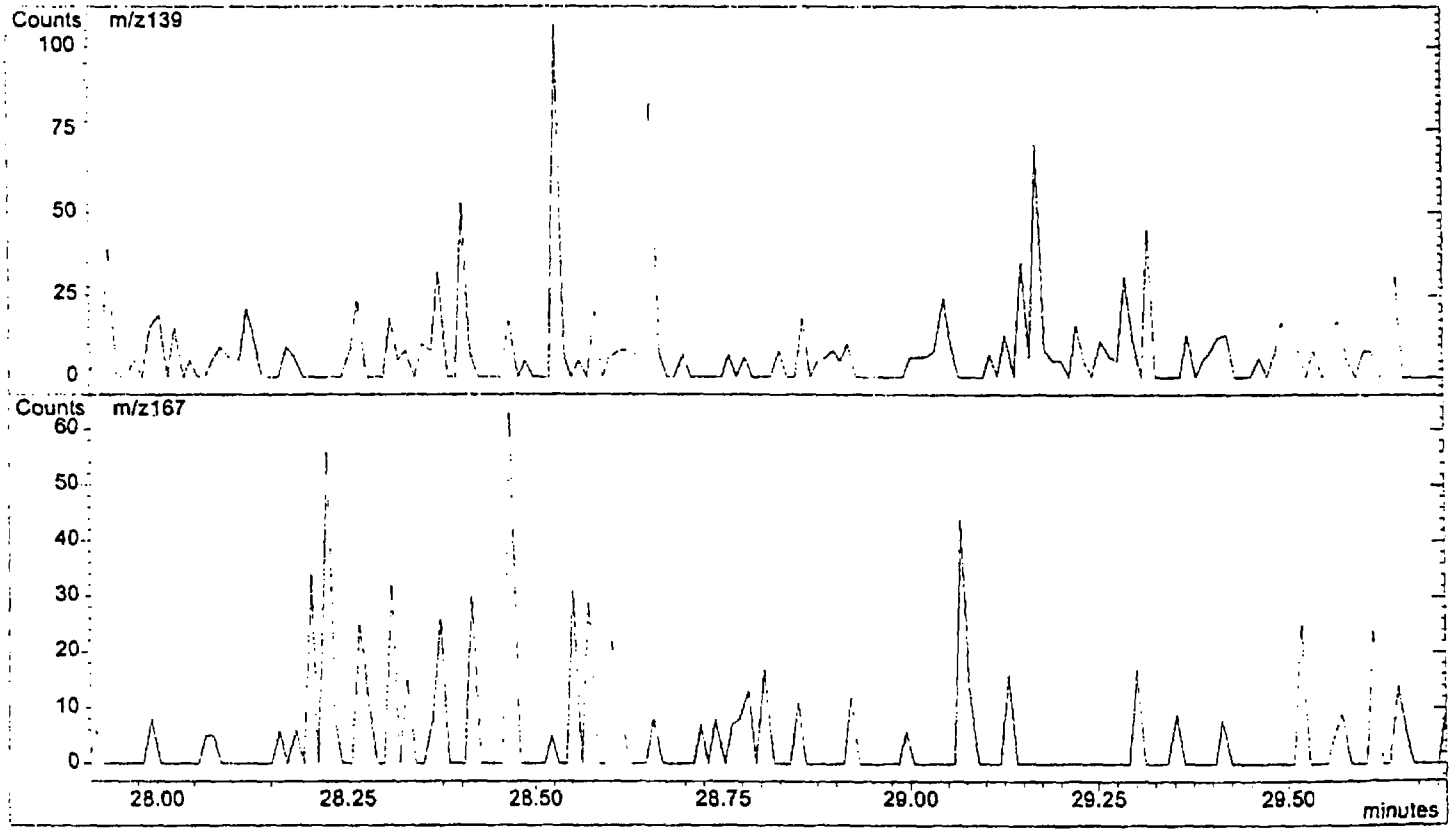
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.319 min.	28.812 min.	28.112 - 29.512 min.	0	Missing

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	0.000 min.	0.00%	2.0%	0.6% - 3.4%	0	Fail
167	0.000 min.	0.00%	4.6%	1.4% - 7.8%	0	Fail

Concentration: 0.000 ug/mL



Sample: VX



Segment: 4 Time(min): 23.993-32.986 Scans: 1422-2273 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:53:31

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/17/02 4:12 AM
al:	8	Measurement Type:	Area
Data File:	c:\... \2002\october\101602#1\xvx100302-3,0.46ug10-17-02.sms		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

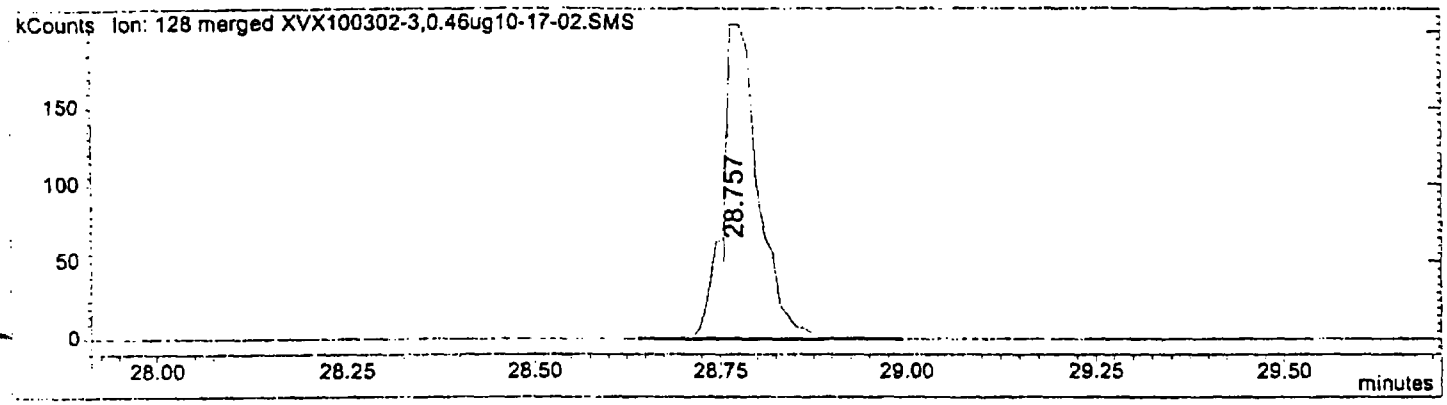
X-Author
C31763 Lot P-6
10-16-02

Analyte: VX

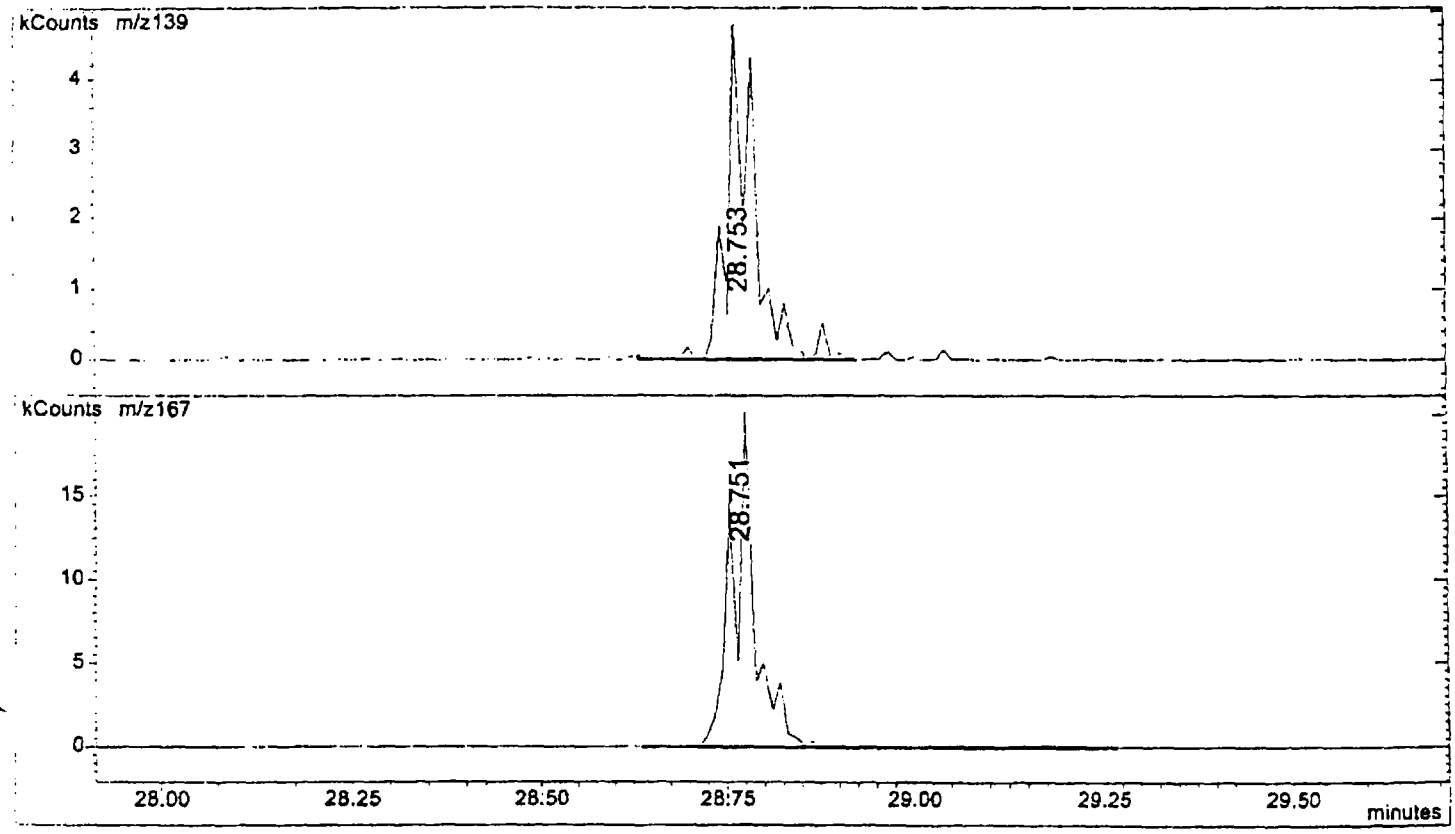
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.757 min.	28.812 min.	28.112 - 29.512 min.	692113	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.753 min.	1.75%	2.0%	0.6% - 3.4%	12143	Pass
167	28.751 min.	6.11%	4.6%	1.4% - 7.8%	42283	Pass

Concentration: 0.538 ug/mL



Sample: VX



Segment: 4 Time(min): 23.993-32.987 Scans: 1422-2276 Ionization: CI Auto Preparation: MS/MS Channels: 1

Varian Star Workstation - 8400 SampleList Thu Oct 17 14:44:47 2002

SampleList: C:\saturnws\Data\2002\October\10170281\VX10170281.smp

i: Thu Oct 17 14:44:33 2002
ed: Thu Oct 17 14:44:34 2002

X. Arthur
C3 #63 Book P-9
10-17-02

Line	Sample Name	Injection Notes	AutoLink	Vial	Inject Volume
1			C:\saturnws\Methods\VX-2002\VX092502-ms268.2-0.42V-PIT-200-ST-0.65-noPP-Purge@20-EI;Multi1750v+300v-UDInj1.mth		
2	XVX100302-3,0.46ug	below	none	2	1.0
	Notes 0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
3	XVX-101502-US-3DS1-	below	none	3	1.0
	Notes JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extraction, no HAE cleanup; Initial Volume				
	Notes = 5mL, diluted 1:10 w/CH2Cl2,2.74ug VX Spike into dilution RtxSMS;S-TRAP;EM@1750V				
4	XVX-101502-US-4DS1-	below	none	4	1.0
	Notes JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extraction, no HAE cleanup; Initial Volume				
	Notes = 5mL, diluted 1:10 w/CH2Cl2,2.74ug VX Spike into dilution RtxSMS;S-TRAP;EM@1750V				
5	CH2Cl2 Blank	below	none	8	1.0
	Notes CH2Cl2 Blank, Rtx-5MS:30Mx0.25mm ID, 1.0um; 100C,5min - 240C @5C/m-280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec; 0.65scans/sec;noPP;m/z268.2;1750V; S-TRAP@170C				
6	XVX100302-3,0.46ug	below	none	5	1.0
	Notes 0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
7	XVX-101602-US-3D1-	below	none	6	1.0
	Notes JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extraction, no HAE cleanup; Initial Volume				
	Notes = 5mL, diluted 1:10 w/CH2Cl2; RtxSMS;S-TRAP;EM@1750V				
8	XVX-101702-US-3DS2-	below	none	7	1.0
	Notes JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extraction, no HAE cleanup; Initial Volume				
	Notes = 5mL, diluted 1:10 w/CH2Cl2,8.73ug VX Spike into dilution RtxSMS;S-TRAP;EM@1750V				
9	XVX-101602-US-4D1-	below	none	8	1.0
	Notes JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extraction, no HAE cleanup; Initial Volume				
	Notes = 5mL, diluted 1:10 w/CH2Cl2; RtxSMS;S-TRAP;EM@1750V				
10	XVX-101702-US-4DS2-	below	none	9	1.0
	Notes JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extraction, no HAE cleanup; Initial Volume				
	Notes = 5mL, diluted 1:10 w/CH2Cl2,8.73ug VX Spike into dilution RtxSMS;S-TRAP;EM@1750V				
11	CH2Cl2 Blank	below	none	0	1.0
	Notes CH2Cl2 Blank, Rtx-5MS:30Mx0.25mm ID, 1.0um; 100C,5min - 240C @5C/m-280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec; 0.65scans/sec;noPP;m/z268.2;1750V; S-TRAP@170C				
12	XVX100302-1, 1.8ug	below	none	10	1.0
	Notes 1.83ug/mLVX Std, Rtx-5MS:30M x 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
13	XVX100302-1, 1.8ug	below	none	10	1.0
	Notes 1.83ug/mLVX Std, Rtx-5MS:30M x 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
14	XVX100302-2,0.91ug	below	none	11	1.0
	Notes 0.913ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42V-EE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;TS-RAP@170C;Sngl-GN				
15	XVX100302-3,0.46ug	below	none	5	1.0
	Notes 0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
16	XVX100302-4,0.18ug	below	none	12	1.0
	Notes 0.183ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
17	XVX100302-5,.091ug	below	none	13	1.0
	Notes 0.091ug/mLVX Std Rtx-5Sil MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
18	XVX100302-6,.046ug	below	none	14	1.0
	Notes 0.046ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
19	XVX100302-3,0.46ug	below	none	5	1.0
	Notes 0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C				
	Notes @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN				
20	XVX-101702-US-1-	below	none	15	1.0

	Notes	JACADS Ash Sample No. LAWIC153B, 1g + 5g NaSO4; Ultrasonic Extraction, no NAE cleanup; Final Volume			
	Notes	= 5mL + DEAET; Unspiked; Rtx5MS;S-TRAP;EM@1750V			
21	XVX-101702-US-2-	below	none	16	1.0
	Notes	JACADS Ash Sample No. LAWIC153B, 1g + 5g NaSO4; Ultrasonic Extraction, no NAE cleanup; Final Volume			
	Notes	= 5mL + DEAET; Unspiked; Rtx5MS;S-TRAP;EM@1750V			
22	XVX-101702-US-3-	below	none	17	1.0
	Notes	JACADS Ash Sample No. LAWIC153B, 1g + 5g NaSO4; Ultrasonic Extraction, no NAE cleanup; Final Volume			
	Notes	= 5mL + DEAET; 3.74ug/g VX Spike; Rtx5MS;S-TRAP;EM@1750V			
23	XVX-101702-US-4-	below	none	18	1.0
	Notes	JACADS Ash Sample No. LAWIC153B, 1g + 5g NaSO4; Ultrasonic Extraction, no NAE cleanup; Final Volume			
	Notes	= 5mL + DEAET; 3.74ug/g VX Spike; Rtx5MS;S-TRAP;EM@1750V			
24	CH2Cl2 Blank	below	none	0	1.0
	Notes	CH2Cl2 Blank, Rtx-5MS:30Mx0.25mm ID, 1.0um; 100C,5min - 240C @5C/m-280C			
	Notes	@25C/m,5min;0.42VEE;PIT@200usec; 0.65scans/sec;noPP;m/z268.2;1750V; S-TRAP@170C			
25	XVX100302-3,0.46ug	below	none	5	1.0
	Notes	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C			
	Notes	@25C/m,5min;0.43VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN			
26	XVX100302-3,0 46ug	below	none	5	1.0
	Notes	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C			
	Notes	@25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN			
27		C:\saturnws\Methods\Standby.mth			

Directory for Data Files: c:\saturnws\data\2002\october\10170281

Data File Names: %%d

Create and update a new RecalcList: c:\saturnws\data\2002\october\10170281\vx101702-1.rcl.

****

Varian Star Workstation - RecalcList Fri Oct 18 13:22:55 2002

lcList: C:\saturnws\Data\2002\October\101702#1\VX101702-1.RCL

Created: Thu Oct 17 14:46:51 2002
Modified: Fri Oct 18 13:22:46 2002

****

Line	Sample Type	Sample Name	Data File	Inj.
1	Verification	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-17-02.SMS	1
2	Analysis	VX-101502-US-3DS-	VX-101502-US-3DS-10-17-02.SMS	1
3	Analysis	VX-101502-US-4DS-	VX-101502-US-4DS-10-17-02.SMS	1
4	Analysis	CH2Cl2 Blank	CH2Cl2 Blank10-17-02.SMS	1
5	Verification	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-17-02001.SMS	1
6	Analysis	VX-101602-US-3D1-	VX-101602-US-3D1-10-17-02.SMS	1
7	Analysis	VX-101702-US-3DS2-	VX-101702-US-3DS2-10-17-02.SMS	1
8	Analysis	VX-101602-US-4D1-	VX-101602-US-4D1-10-17-02.SMS	1
9	Analysis	VX-101702-US-4DS2-	VX-101702-US-4DS2-10-17-02.SMS	1
10	Analysis	CH2Cl2 Blank	CH2Cl2 Blank10-17-02001.SMS	1
11	Analysis	XVX100302-1, 1.8ug	XVX100302-1, 1.8ug10-17-02.SMS	1
12	Analysis	XVX100302-1, 1.8ug	XVX100302-1, 1.8ug10-17-02001.SMS	1
13	Analysis	XVX100302-2,0.91ug	XVX100302-2,0.91ug10-18-02.SMS	1
14	Analysis	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-18-02.SMS	1
15	Analysis	XVX100302-4,0.18ug	XVX100302-4,0.18ug10-18-02.SMS	1
16	Analysis	XVX100302-5,.091ug	XVX100302-5,.091ug10-18-02.SMS	1
17	Analysis	XVX100302-6,.046ug	XVX100302-6,.046ug10-18-02.SMS	1
18	Verification	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-18-02001.SMS	1
19	Analysis	VX-101702-US-1	vx-101702-us-1-10-18-02.sms	1
20	Analysis	VX-101702-US-2	vx-101702-us-2-10-18-02.sms	1
21	Analysis	VX-101702-US-3	vx-101702-us-3-10-18-02.sms	1
22	Analysis	VX-101702-US-4	vx-101702-us-4-10-18-02.sms	1
23	Analysis	CH2Cl2 Blank	CH2Cl2 Blank10-18-02.SMS	1
24	Verification	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-18-02002.SMS	1
25	Verification	XVX100302-3,0.46ug	XVX100302-3,0.46ug10-18-02003.SMS	1

Print Date: 21 Oct 2002 18:43:18

Calibration Block Report

Run File:	c:\saturnws\data\2002\october\101702#1\xvx100302-3,0.46ug10-17-02.sms		
Instrument Method:	c:\... vx092502-mz268.2-0.42v-pit	Acquisition Date:	10/17/02 2:52 PM
Recalc Method:	c:\... vx-calib-deaet-10-17-02.mt	Last Calibration:	10/18/02 1:02 PM
Sample List:		Compound Table Updated:	10/21/02 7:23 PM
Sequence:		Detector:	2000 Mass Spec
Workstation Name:	Saturn GC/MS Workstation	Workstation Version:	Version 5.52
Peak Measurement:	Area	Calibration Type:	External Standard Analysis

Retention Time (min)	Peak Name	Cal. Method	X3	X2	X	C	r2	Cal. Range	No. of Points	Edit Codes
28.812	VX	2 IG None		204171.5	1286284.6	5592.58	0.997614	1-6	6	

Curve Fit Codes	Origin Point Codes		Regression Weighting Codes		Edit Codes
1 linear	IN	include	None	1/nX	1 curve
2 quadratic	IG	ignore	1/n	1/X2	2 origin
3 cubic	FO	force	1/X	1/nX2	3 coefficient

Ret. Time: 28.812 min. Peak Name: VX

Level	Amount	Replicate No.	Response	Avg. Response	Std. Dev.	% RSD	RF	Avg. RF
1	1.830000	1	3054257	3054256.5	#		1668992.6	
2	0.913000	1	1281206	1281205.5	#		1403291.9	
3	0.457000	1	735341	735341.1	#		1609061.6	
4	0.183000	1	251252	251252.4	#		1372964.0	
5	0.091000	1	99121	99120.7	#		1089238.1	
6	0.046000	1	45644	45643.7	#		992253.3	1355966.9

= Too few points to calculate.

Peak Name	Level	Rep.	Injection Date/Time	Run Files
VX	1	1	10/17/02 11:16 PM	c:\... \october\101702#1\xvx100302-1, 1.8ug10-17-02001.sms
	2	1	10/18/02 12:01 AM	c:\... \2002\october\101702#1\xvx100302-2,0.91ug10-18-02.sms
	3	1	10/18/02 12:46 AM	c:\... \2002\october\101702#1\xvx100302-3,0.46ug10-18-02.sms
	4	1	10/18/02 1:31 AM	c:\... \2002\october\101702#1\xvx100302-4,0.18ug10-18-02.sms
	5	1	10/18/02 2:16 AM	c:\... \2002\october\101702#1\xvx100302-5,.091ug10-18-02.sms
	6	1	10/18/02 3:01 AM	c:\... \2002\october\101702#1\xvx100302-6,.046ug10-18-02.sms

Print Date: 21 Oct 2002 18:42:32

Calibration Curve Report

e: c:\saturnws\methods\vx-2002\vx-calib-deaet-10-17-02.mth

tector: 2000 Mass Spec, Address: 40

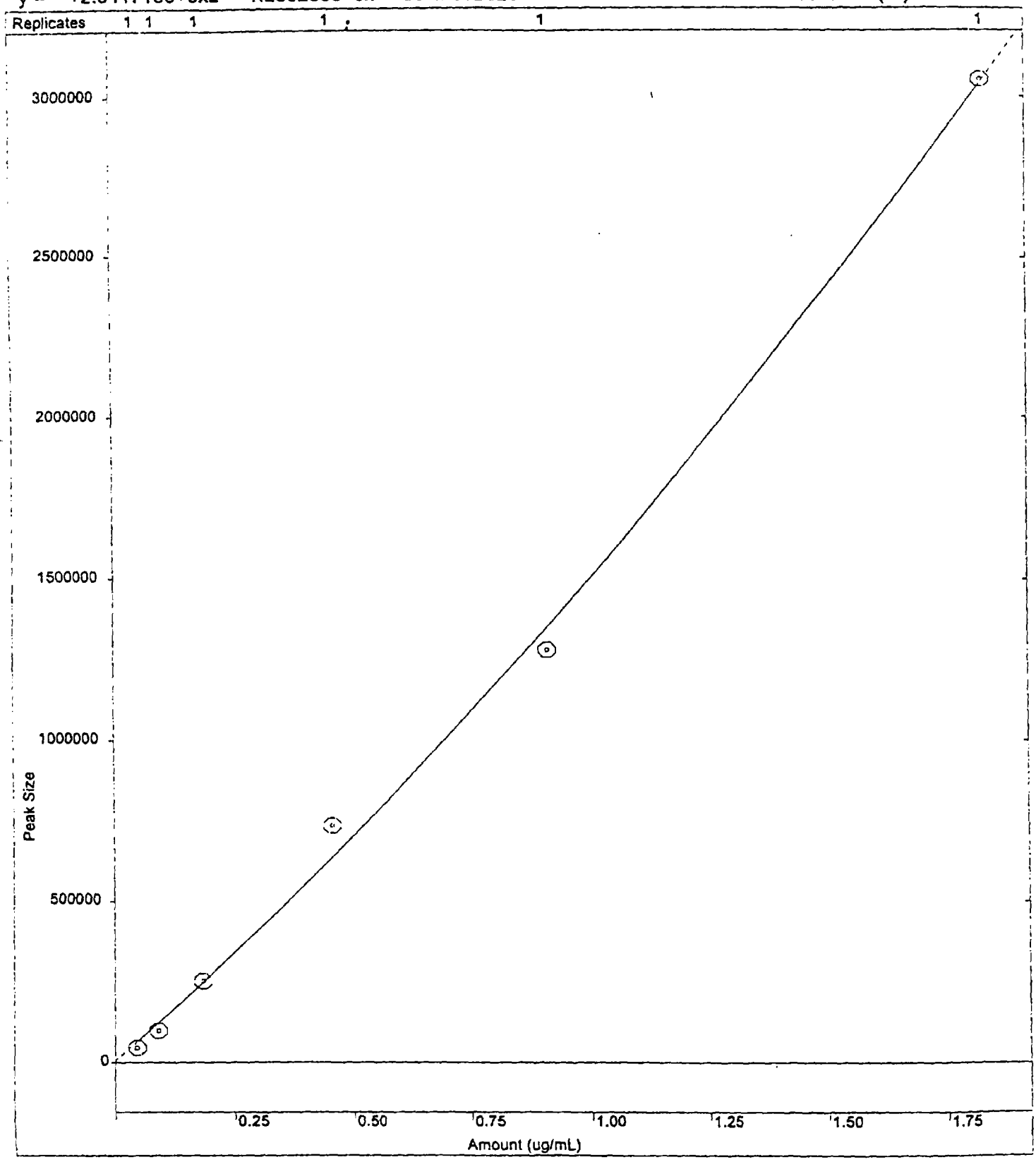
VX

Curve Fit: Quadratic, Origin: Ignore, Weight: None

y = +2.041715e+5x2 +1.286285e+6x +5592.582520

Resp. Fact. RSD: 20.01%

Coeff. Det.(r2): 0.997614

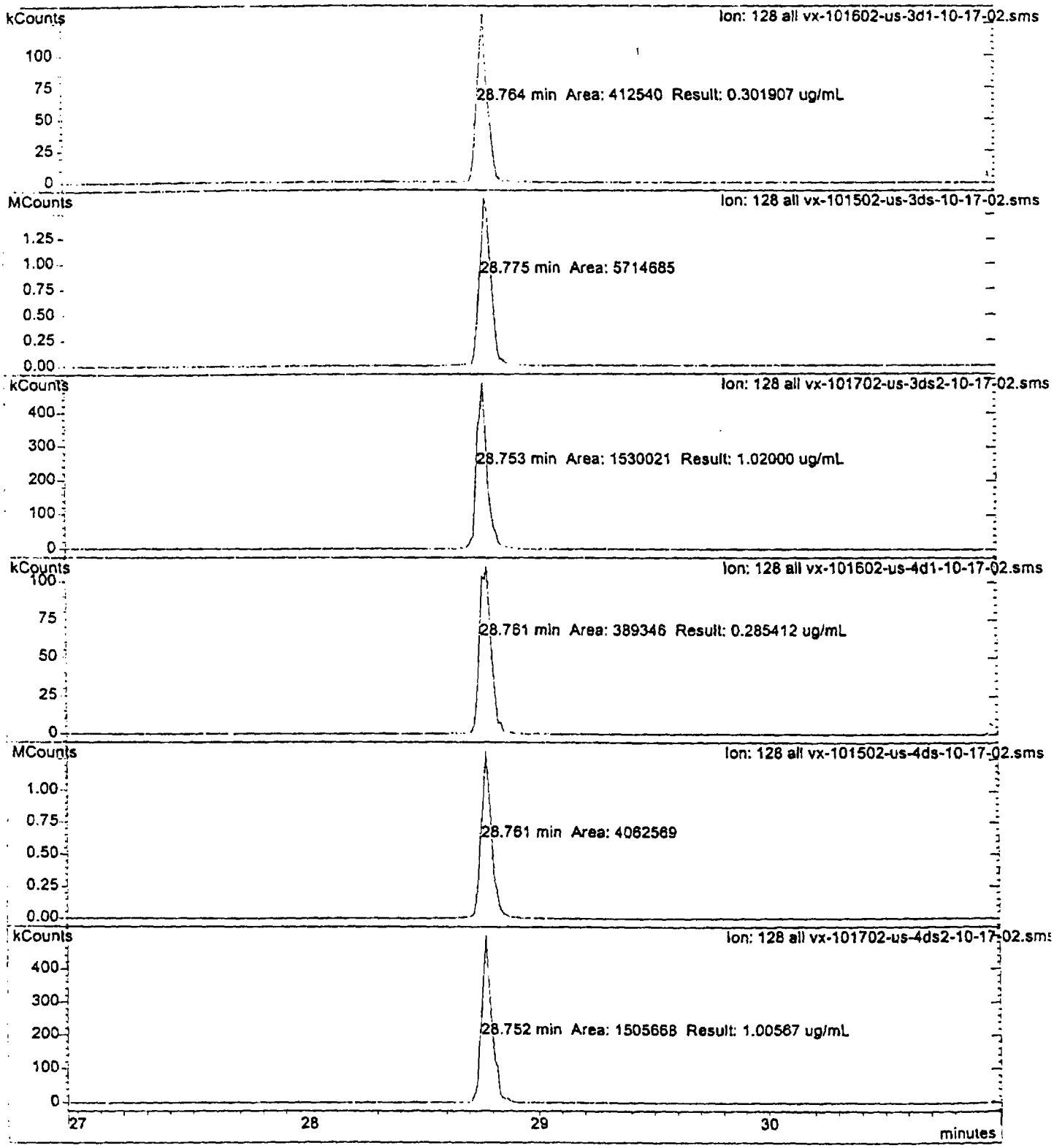


Print Date: 21 Oct 2002 20:32:10

Y. Abshu
C31763 [illegible] P-4
10-17-02

Chromatogram Plots

Plot 1: c:\... \101702#1\vx-101602-us-3d1-10-17-02.sms Ion: 128 all
Plot 2: c:\... \101702#1\vx-101502-us-3ds-10-17-02.sms Ion: 128 all
Plot 3: c:\... \101702#1\vx-101702-us-3ds2-10-17-02.sms Ion: 128 all
Plot 4: c:\... \101702#1\vx-101602-us-4d1-10-17-02.sms Ion: 128 all
Plot 5: c:\... \101702#1\vx-101502-us-4ds-10-17-02.sms Ion: 128 all
Plot 6: c:\... \101702#1\vx-101702-us-4ds2-10-17-02.sms Ion: 128 all

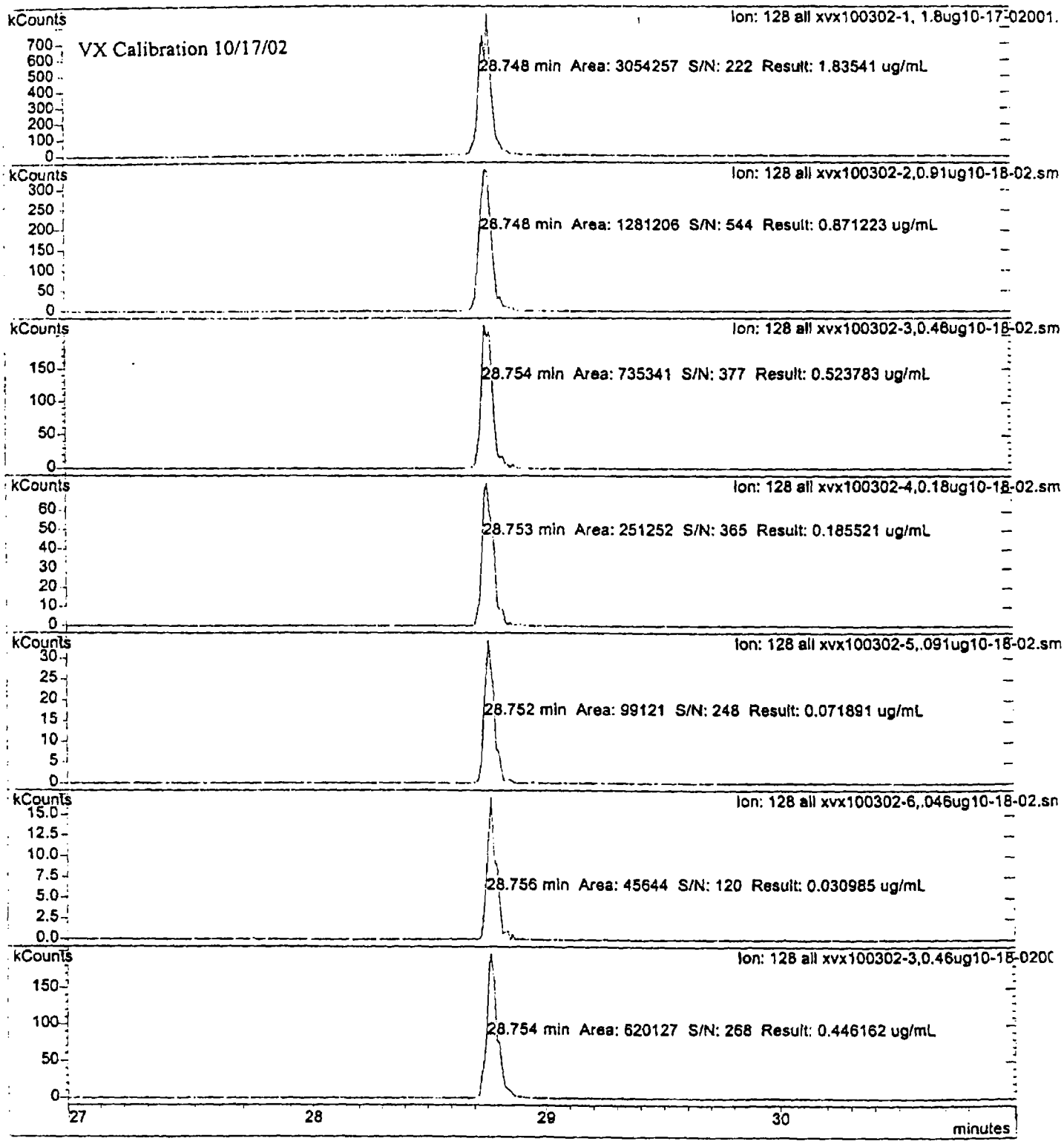


Print Date: 21 Oct 2002 19:13:18

Y. Abrahe
C31763 Back-P-4
10-17-02

Chromatogram Plots

Plot 1: c:\... \xvx100302-1, 1.8ug10-17-02001.sms Ion: 128 all
Plot 2: c:\... \101702#1\xvx100302-2,0.91ug10-18-02.sms Ion: 128 all
Plot 3: c:\... \101702#1\xvx100302-3,0.46ug10-18-02.sms Ion: 128 all
Plot 4: c:\... \101702#1\xvx100302-4,0.18ug10-18-02.sms Ion: 128 all
Plot 5: c:\... \101702#1\xvx100302-5,.091ug10-18-02.sms Ion: 128 all
Plot 6: c:\... \101702#1\xvx100302-6,.046ug10-18-02.sms Ion: 128 all
Plot 7: c:\... \xvx100302-3,0.46ug10-18-02001.sms Ion: 128 all

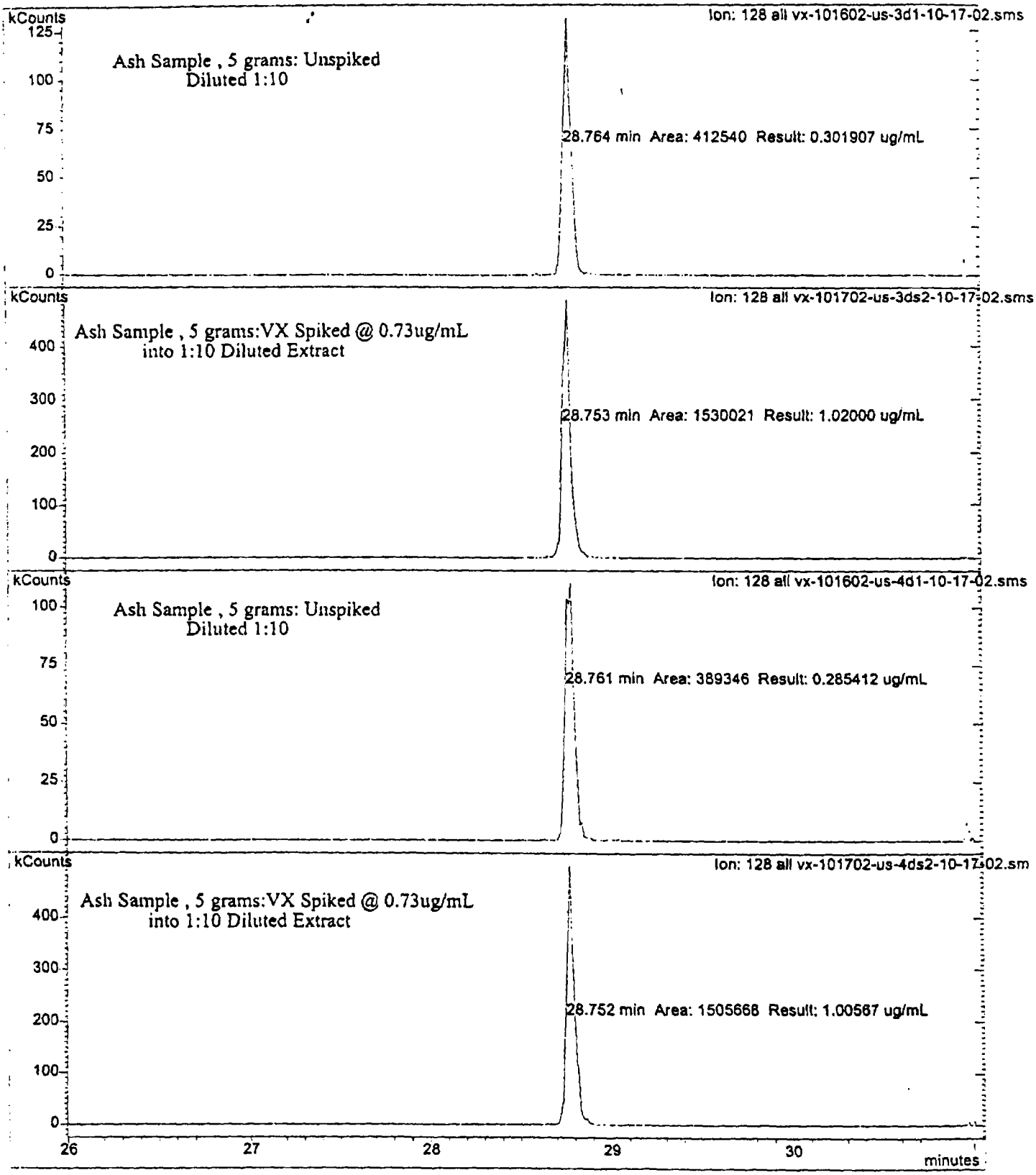


Print Date: 22 Oct 2002 19:09:24

Y. Arthur
C31763 Back P-4
10-17-02

Chromatogram Plots

Plot 1: c:\... \101702#1\vx-101602-us-3d1-10-17-02.sms Ion: 128 all
Plot 2: c:\... \101702#1\vx-101702-us-3ds2-10-17-02.sms Ion: 128 all
Plot 3: c:\... \101702#1\vx-101602-us-4d1-10-17-02.sms Ion: 128 all
Plot 4: c:\... \101702#1\vx-101702-us-4ds2-10-17-02.sms Ion: 128 all

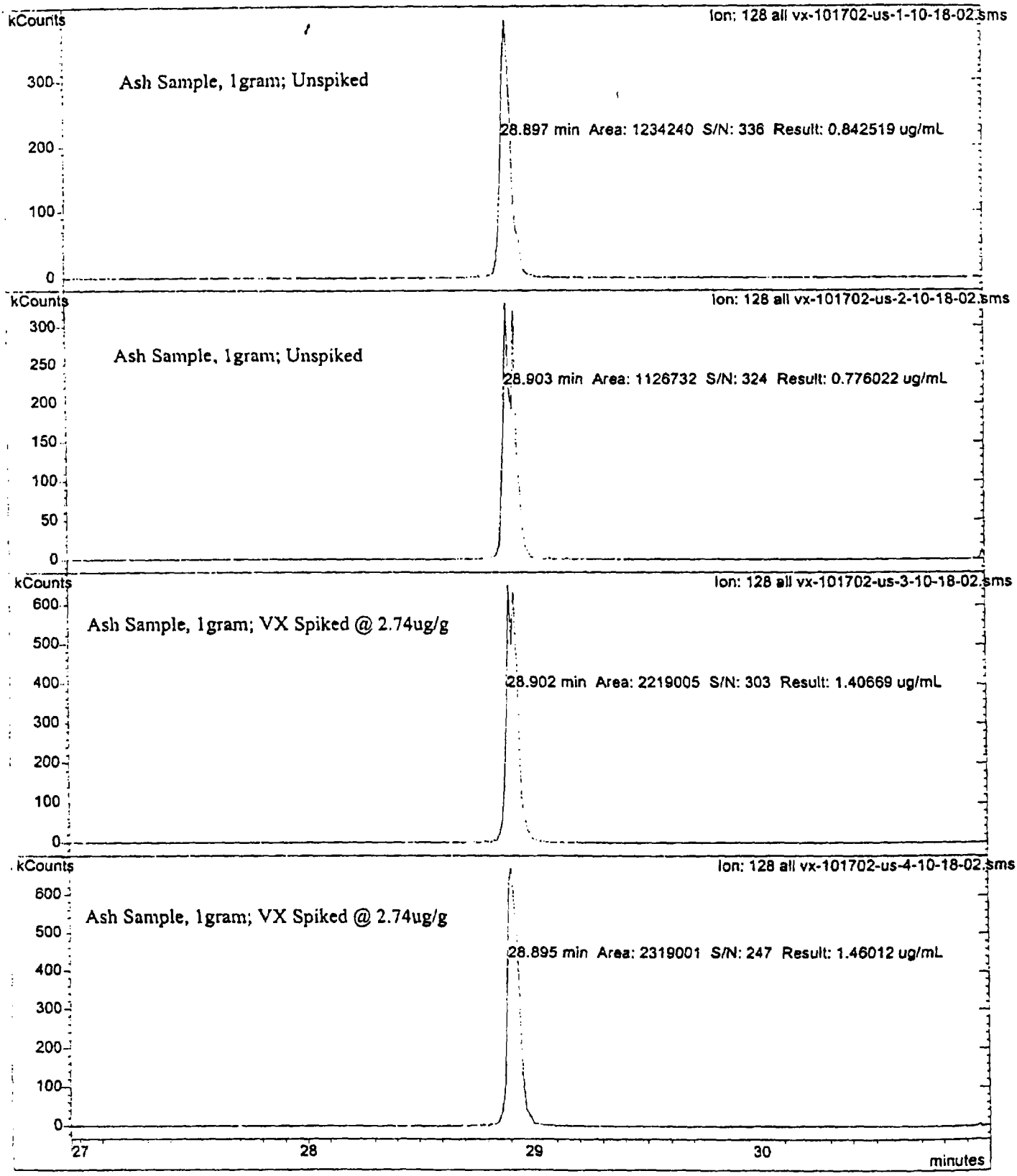


Print Date: 21 Oct 2002 19:17:00

Y. Arthur
C31763 Bi P-9
10-17-02

Chromatogram Plots

Plot 1: c:\... \101702#1\vx-101702-us-1-10-18-02.sms Ion: 128 all
Plot 2: c:\... \101702#1\vx-101702-us-2-10-18-02.sms Ion: 128 all
Plot 3: c:\... \101702#1\vx-101702-us-3-10-18-02.sms Ion: 128 all
Plot 4: c:\... \101702#1\vx-101702-us-4-10-18-02.sms Ion: 128 all



Print Date: 21 Oct 2002 18:47:45

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

ample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/17/02 2:52 PM
Vial:	2	Measurement Type:	Area
Data File:	c:\...\2002\october\101702#1\xvx100302-3,0.46ug10-17-02.sms		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-16-02.mth		

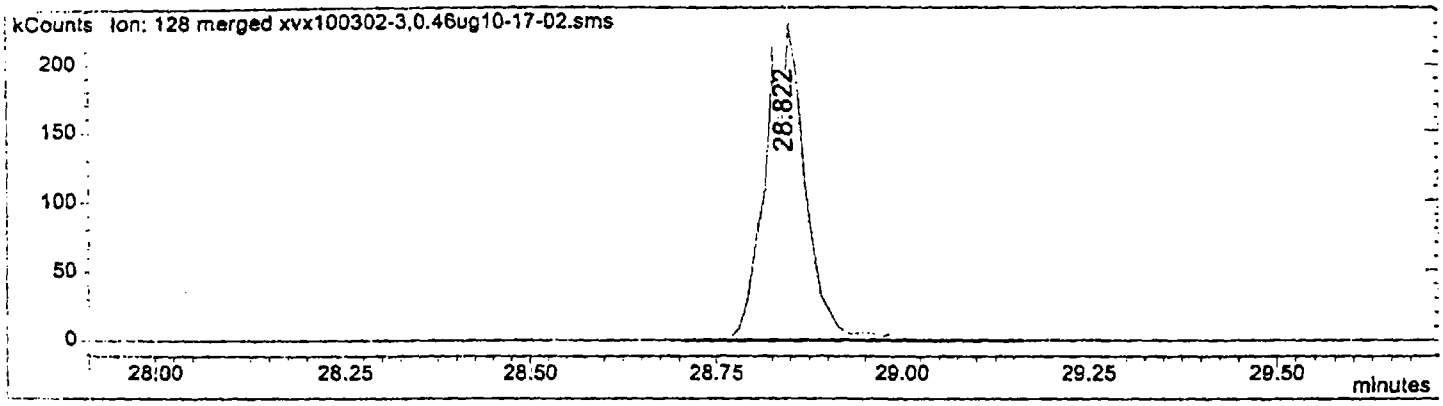
Y-Abstract
C 31763 B-P-4
10-17-02

Analyte: VX

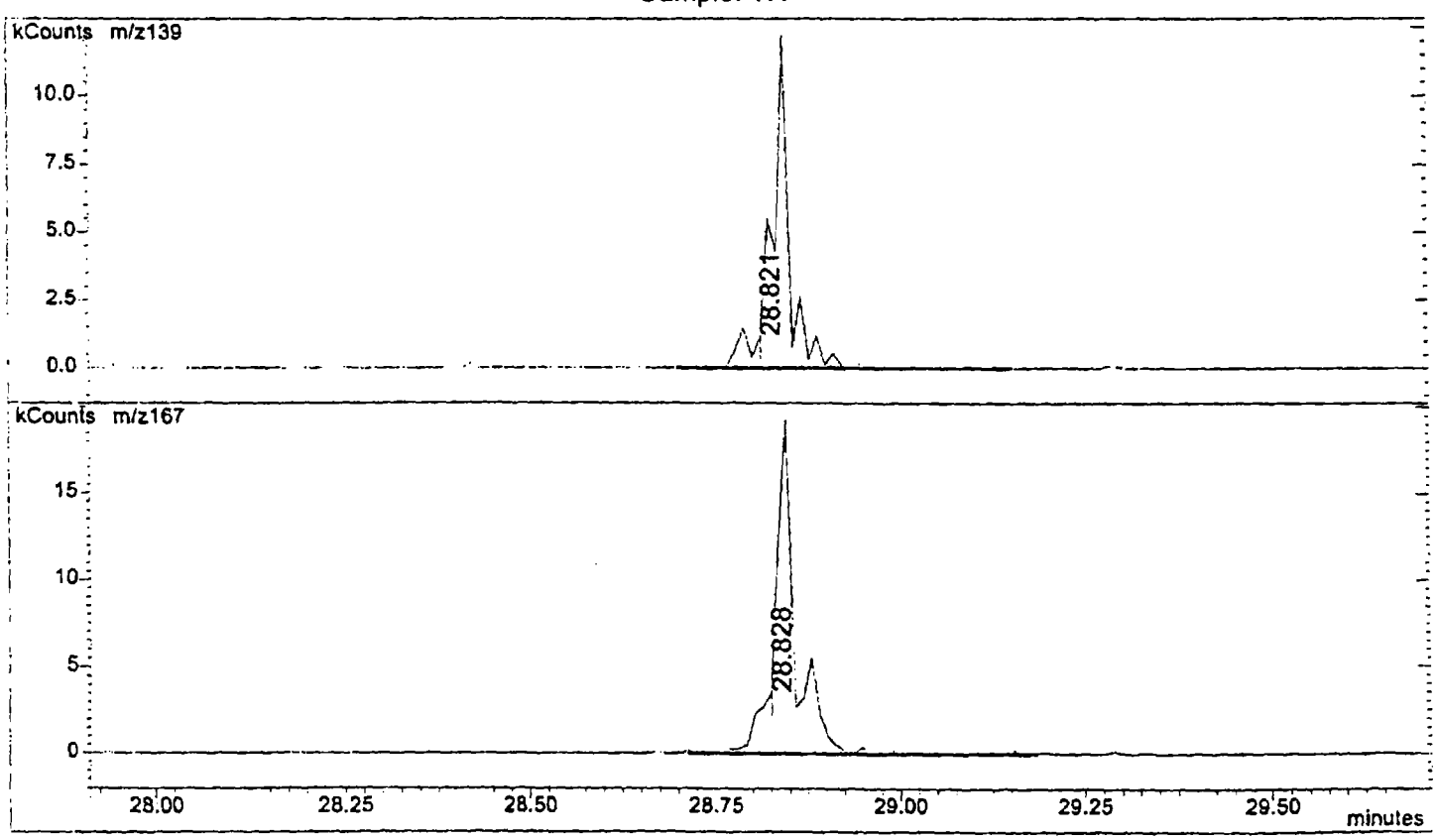
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.822 min.	28.812 min.	28.112 - 29.512 min.	824759	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.821 min.	2.55%	2.0%	0.6% - 3.4%	21000	Pass
167	28.828 min.	4.63%	4.6%	1.4% - 7.8%	38200	Pass

Concentration: 0.629 ug/mL



Sample: VX



Segment: 4 Time(min): 23.996-32.988 Scans: 1422-2243 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:48:40

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/17/02 2:52 PM
Vial:	2	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \2002\october\101702#1\xvx100302-3,0.46ug10-17-02.sms		

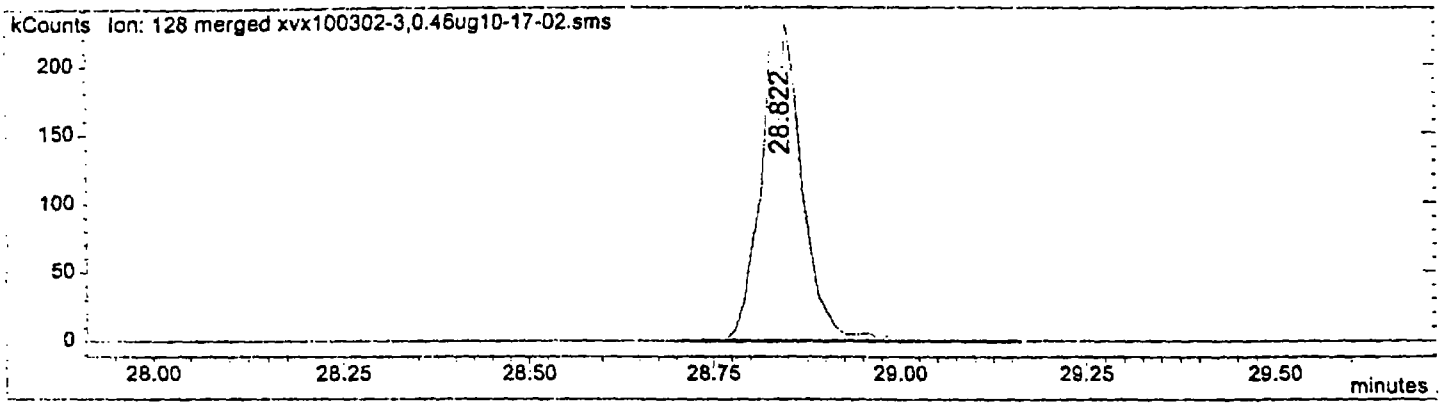
Y. Arthur
C31863 B:P-9
10-17-02

Analyte: VX

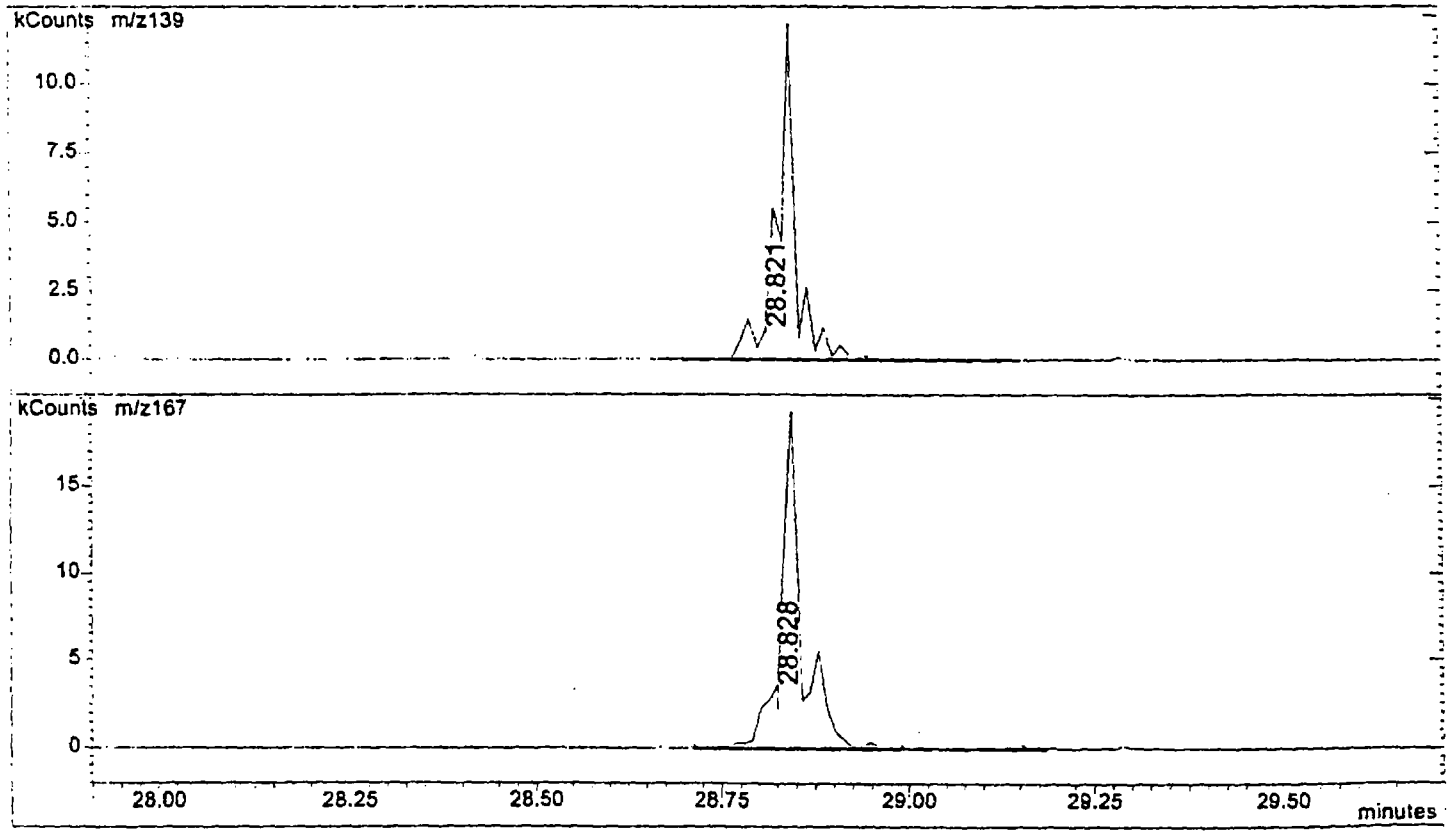
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.822 min.	28.812 min.	28.112 - 29.512 min.	824759	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.821 min.	2.55%	2.0%	0.6% - 3.4%	21000	Pass
167	28.828 min.	4.63%	4.6%	1.4% - 7.8%	38200	Pass

Concentration: 0.583 ug/mL



Sample: VX



Segment: 4 Time(min): 23.996-32.988 Scans: 1422-2243 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:49:23

Ion Ratio Report for Target Compound #1 from vx-101502-us-3ds-10-17-02.sms

Saturn 2000 No.1

ample ID:	VX-101502-US-3DS-	Acquisition Date:	10/17/02 3:37 PM
Vial:	3	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extr action, no HAE cleanup; Initial Volume = 5mL, diluted 1:10 w /CH2Cl2,2.74ug VX Spike into dilution Rtx5MS;S-TRAP;EM@1750V		
Data File:	c:\... \2002\october\101702#1\vx-101502-us-3ds-10-17-02.sms		

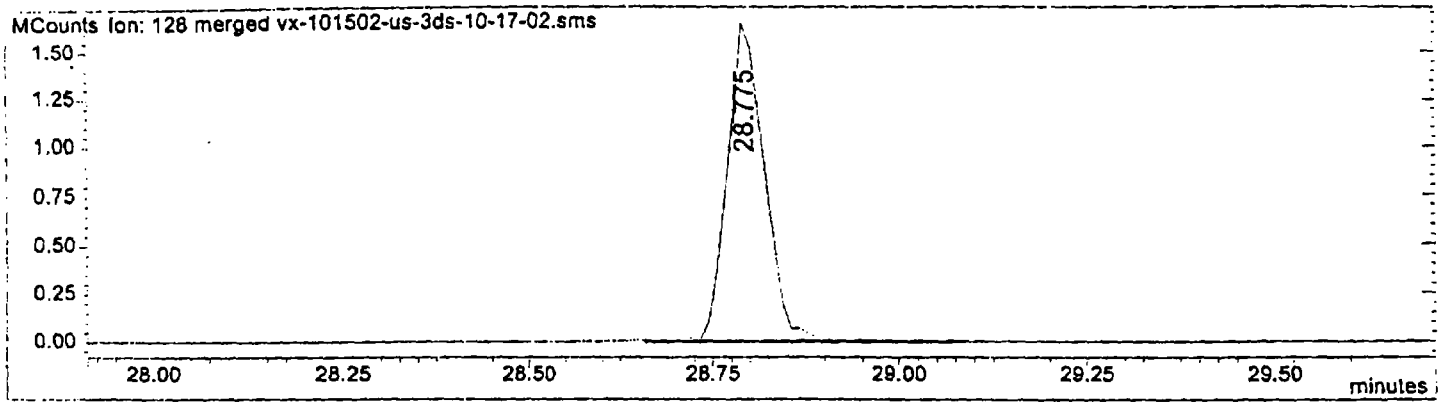
Y-Abstract
C 31763B+P-1
10-17-02

Analyte: VX

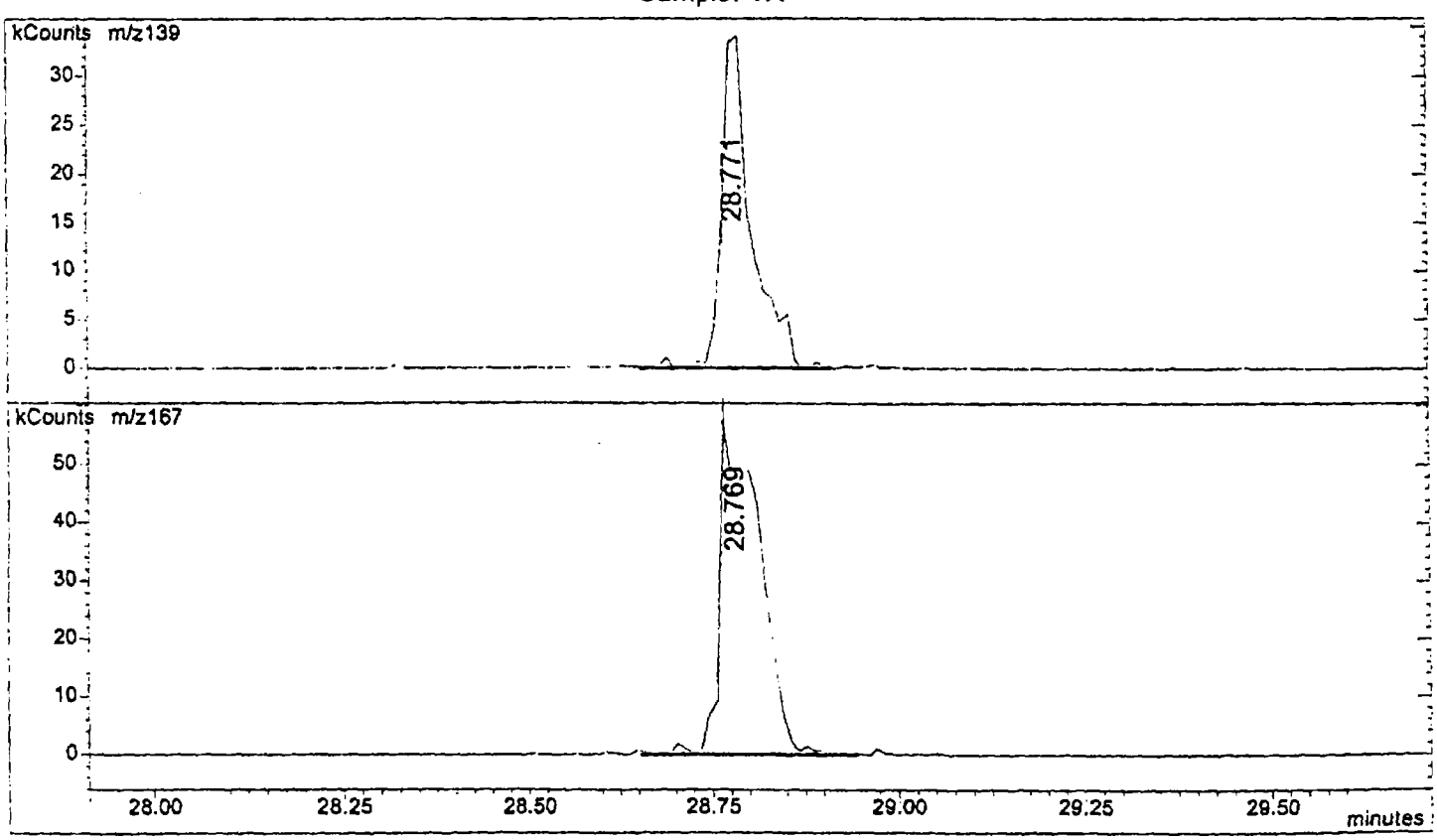
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.775 min.	28.812 min.	28.112 - 29.512 min.	5714685	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.771 min.	1.69%	2.0%	0.6% - 3.4%	96426	Pass
167	28.769 min.	3.80%	4.6%	1.4% - 7.8%	217088	Pass

Concentration: N/A



Sample: VX



Segment: 4 Time(min): 23.996-32.988 Scans: 1421-2274 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:50:20

Ion Ratio Report for Target Compound #1 from vx-101502-us-4ds-10-17-02.sms

Saturn 2000 No.1

ample ID:	VX-101502-US-4DS-	Acquisition Date:	10/17/02 4:22 PM
Vial:	4	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extr action, no HAE cleanup; Initial Volume = 5mL, diluted 1:10 w /CH2Cl2,2.74ug VX Spike into dilution Rtx5MS;S-TRAP;EM@1750V		
Data File:	c:\... \2002\october\101702#1\vx-101502-us-4ds-10-17-02.sms		

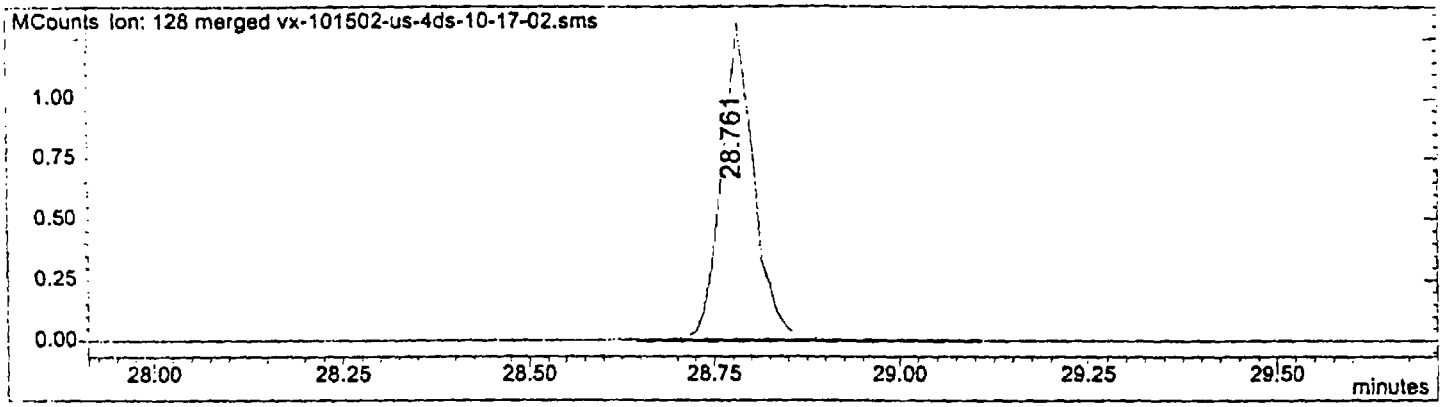
Y-Arthur
C31763 Exp.
10-17-02

Analyte: VX

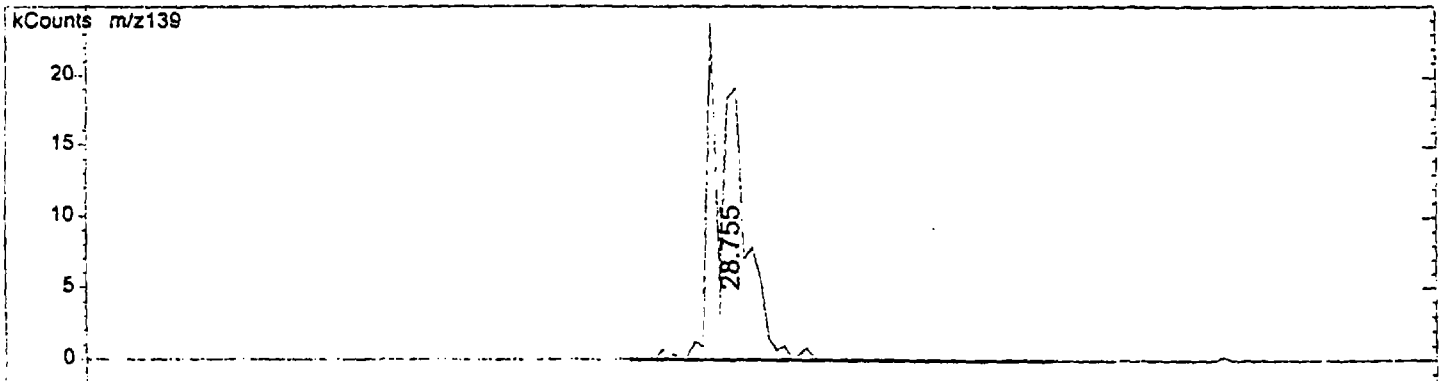
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.761 min.	28.812 min.	28.112 - 29.512 min.	4062569	Identified

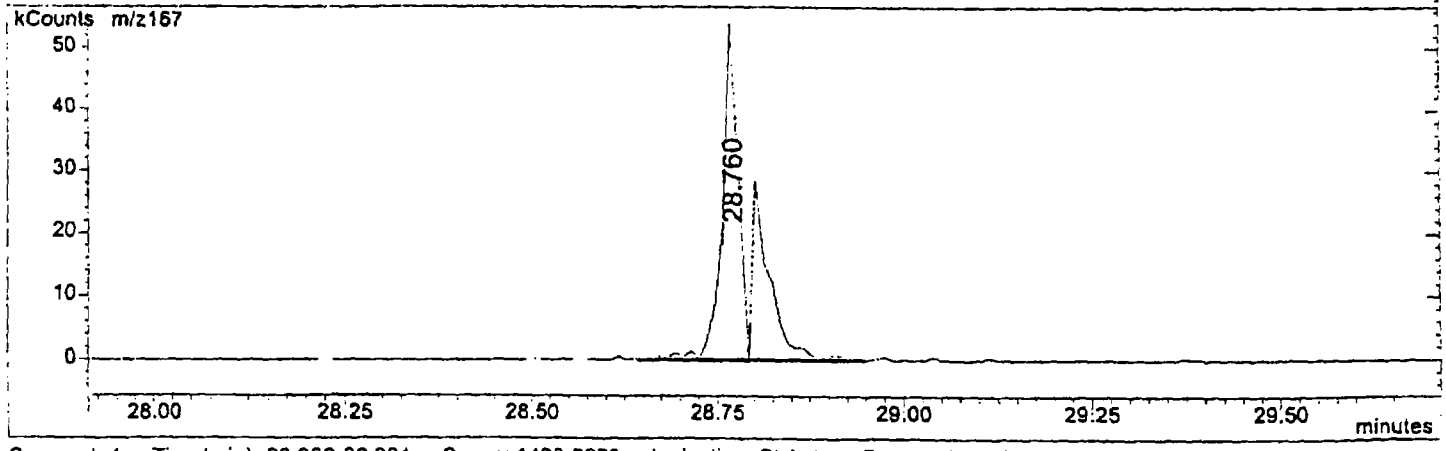
Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.755 min.	1.60%	2.0%	0.6% - 3.4%	64935	Pass
167	28.760 min.	3.10%	4.6%	1.4% - 7.8%	125939	Pass

Concentration: N/A



Sample: VX





Segment: 4 Time(min): 23.992-32.984 Scans: 1423-2279 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:50:34

Ion Ratio Report for Target Compound #1 from ch2cl2 blank-10-17-02.sms

Saturn 2000 No.1

Sample ID:	CH2Cl2 Blank	Acquisition Date:	10/17/02 5:07 PM
Vial:	0	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	CH2Cl2 Blank, Rtx-5MS:30Mx0.25mm ID, 1.0um; 100C,5min - 240 C @5C/m-280C @25C/m,5min;0.42VEE;PIT@200usec; 0.65scans/sec; noPP;m/z268.2;1750V; S-TRAP@170C		
Data File:	c:\... \data\2002\october\101702#1\ch2cl2 blank-10-17-02.sms		

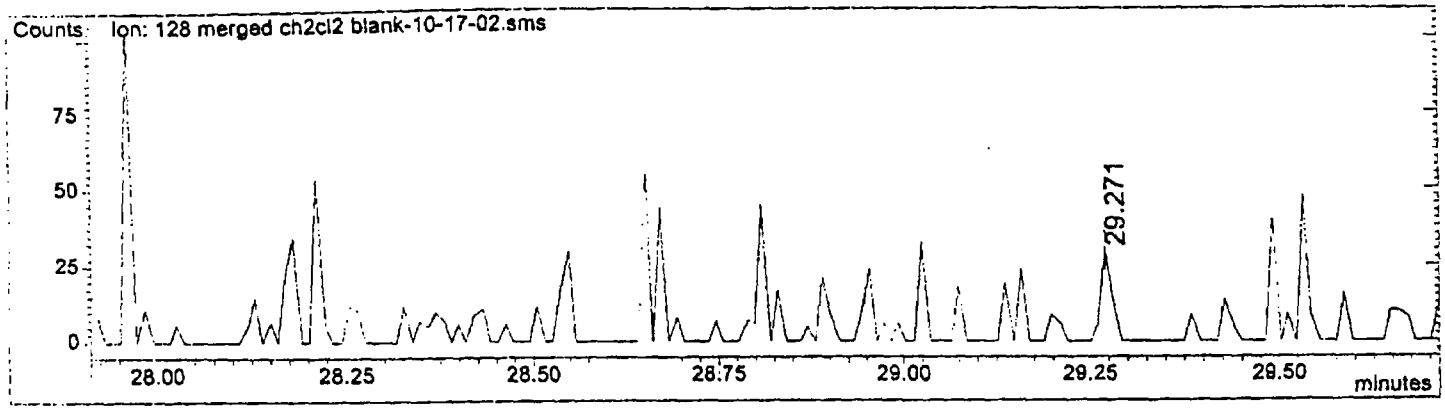
X-Abthm
C31763 Book P
10-17-02

Analyte: VX

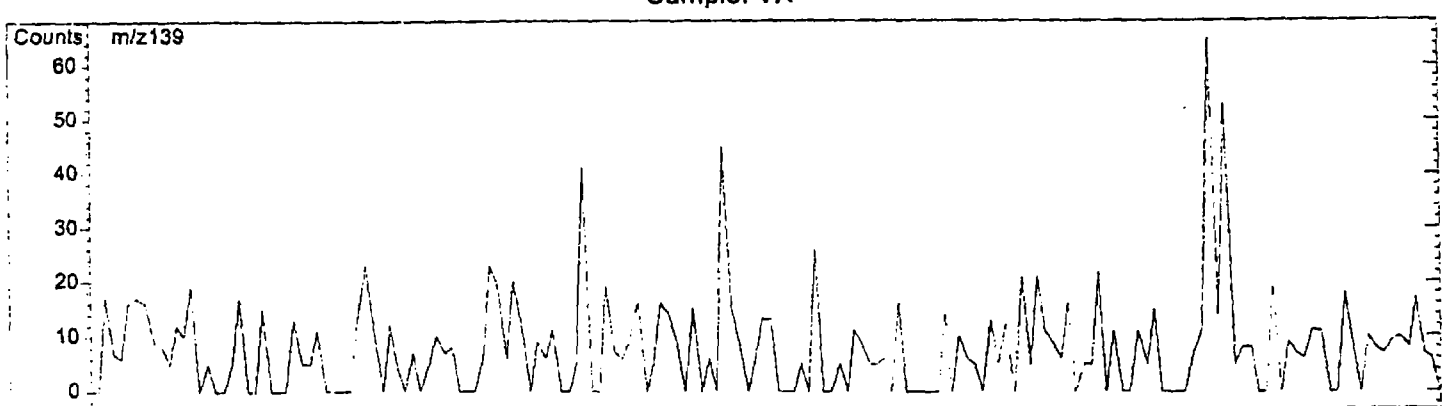
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	29.271 min.	28.812 min.	28.112 - 29.512 min.	0	Missing

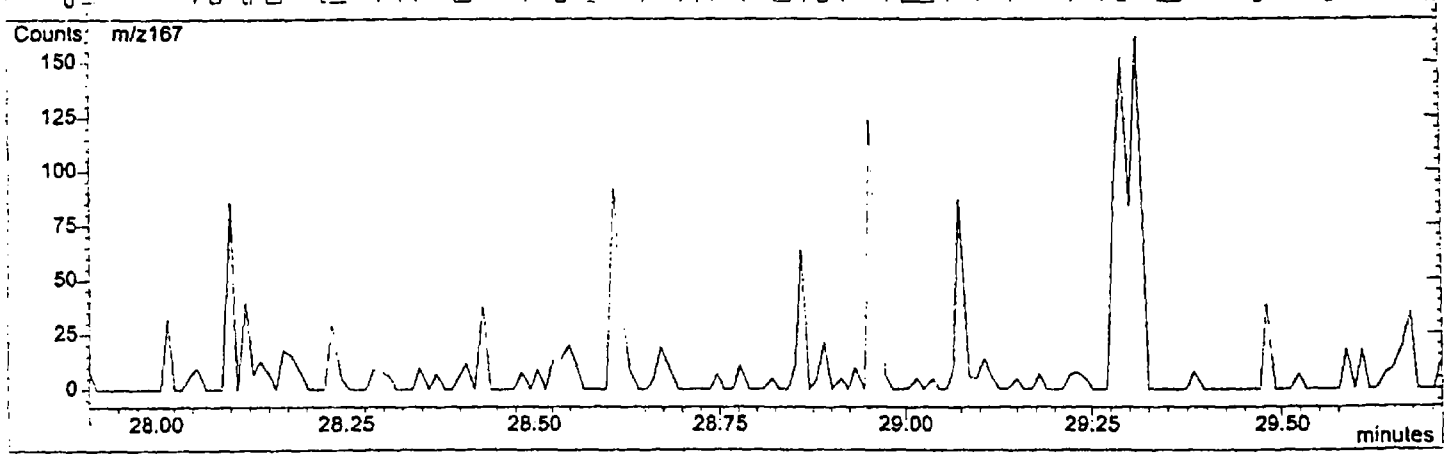
Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	0.000 min.	0.00%	2.0%	0.6% - 3.4%	0	Fail
167	0.000 min.	0.00%	4.6%	1.4% - 7.8%	0	Fail

Concentration: 0.000 ug/mL



Sample: VX





Segment: 4 Time(min): 23.981-32.987 Scans: 1423-2288 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:50:47

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

ample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/17/02 5:53 PM
Vial:	5	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \october\101702#1\xvx100302-3,0.46ug10-17-02001.sms		

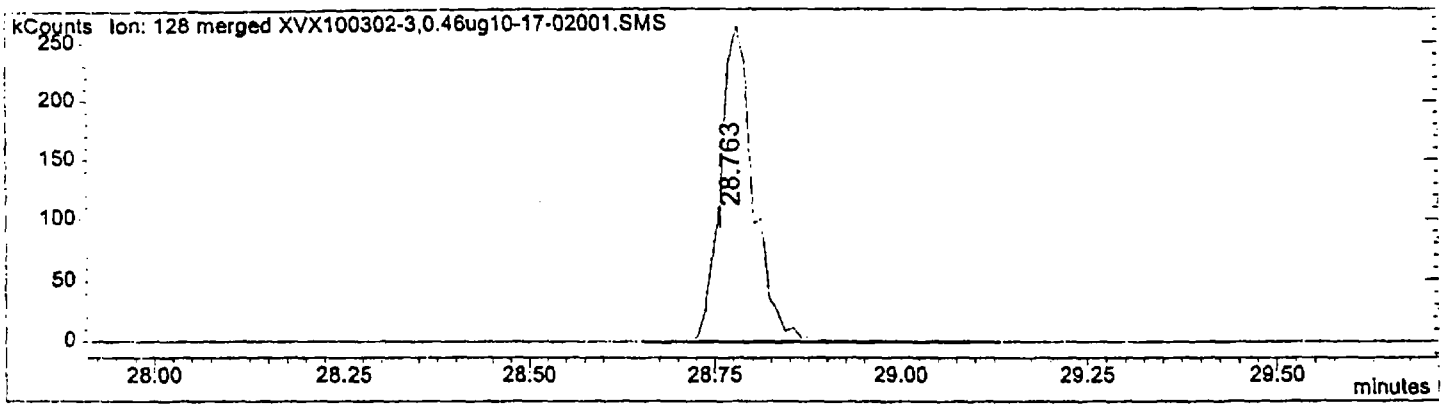
C31765B-P-9
10-17-02

Analyte: VX

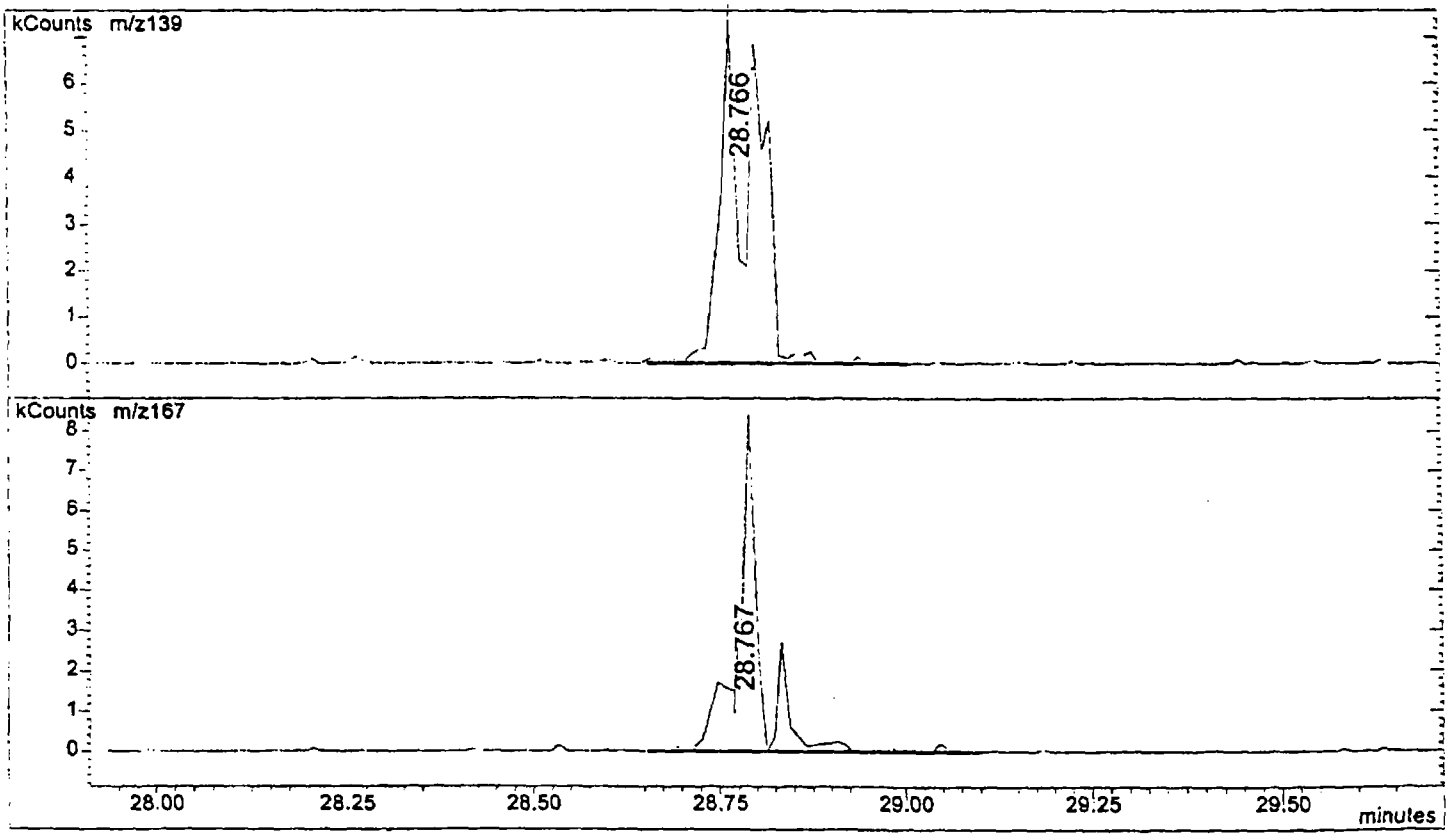
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.763 min.	28.812 min.	28.112 - 29.512 min.	813888	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.766 min.	2.87%	2.0%	0.6% - 3.4%	23348	Pass
167	28.767 min.	2.09%	4.6%	1.4% - 7.8%	17016	Pass

Concentration: 0.576 ug/mL



Sample: VX



Segment: 4 Time(min): 23.982-32.985 Scans: 1418-2272 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 18:51:01

Ion Ratio Report for Target Compound #1 from VX-101602-US-3D1-10-17-02.SMS

Saturn 2000 No.1

ample ID:	VX-101602-US-3D1-	Acquisition Date:	10/17/02 6:45 PM
Vial:	6	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extr action, no HAE cleanup; Initial Volume = 5mL, diluted 1:10 w /CH2Cl2; Rtx5MS;S-TRAP;EM@1750V		
Data File:	c:\... \2002\october\101702#1\vx-101602-us-3d1-10-17-02.sms		

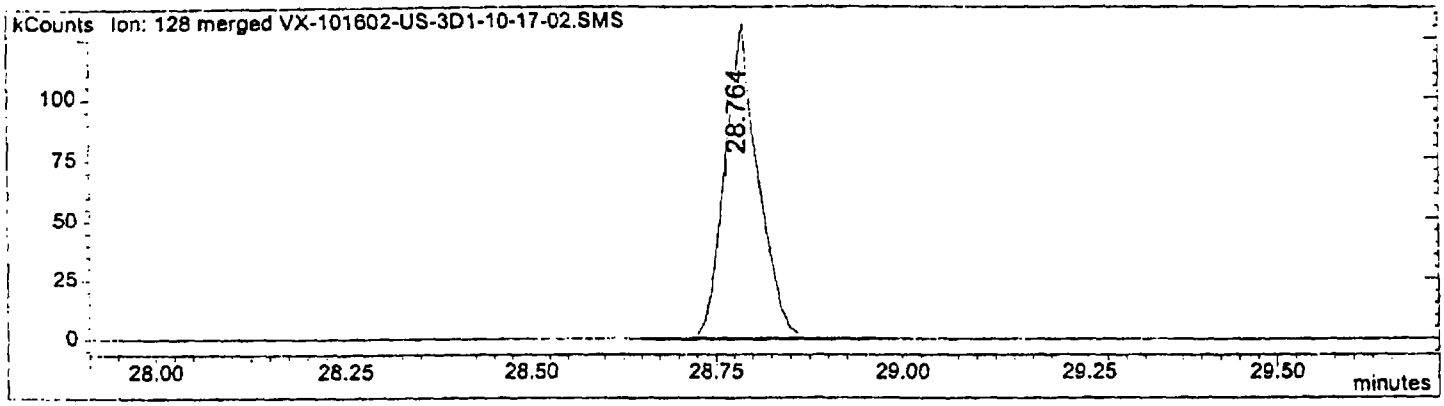
Y Atkins
C31763 Book p-11
10-17-02

Analyte: VX

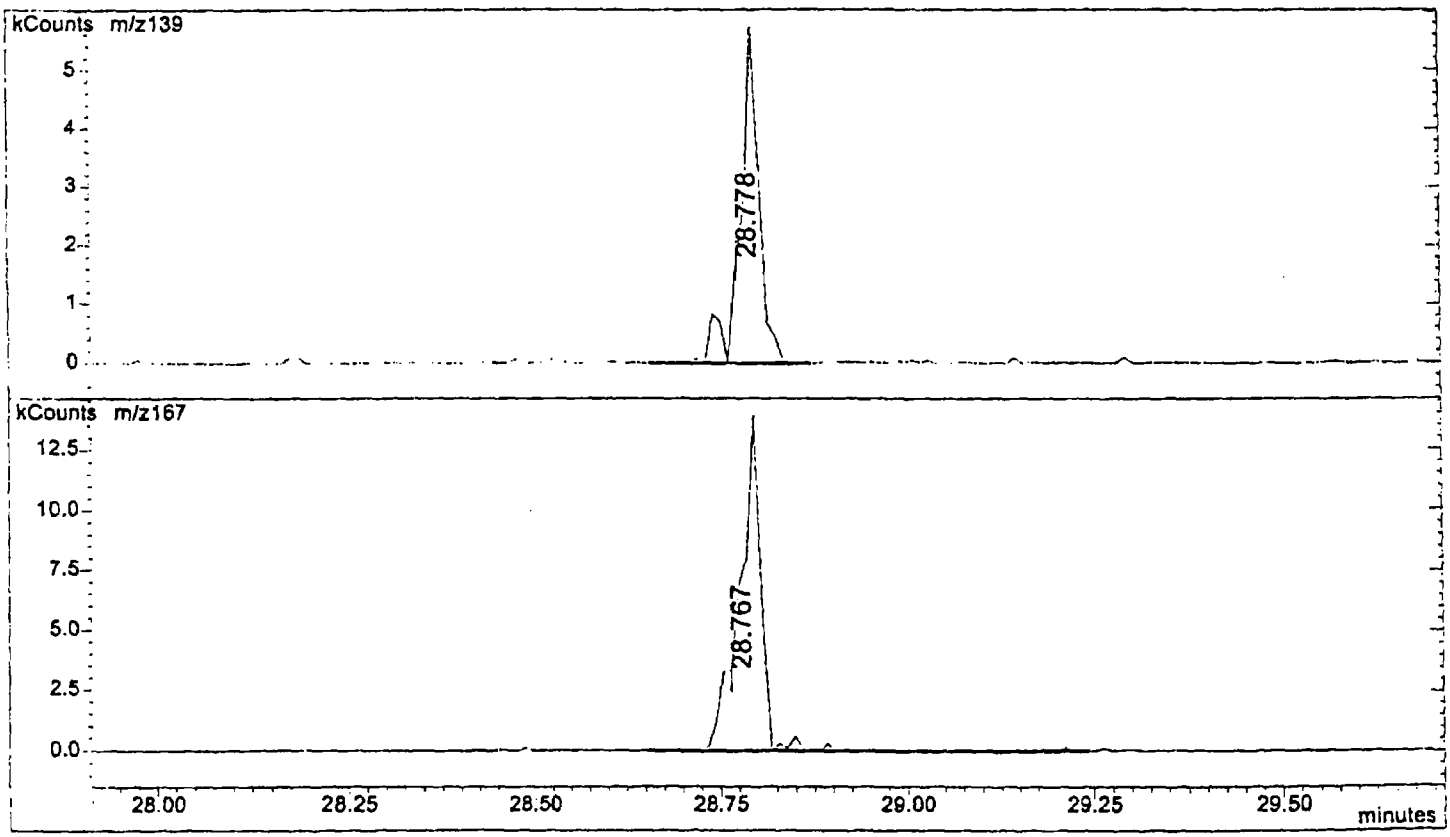
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.764 min.	28.812 min.	28.112 - 29.512 min.	412540	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.778 min.	2.47%	2.0%	0.6% - 3.4%	10209	Pass
167	28.767 min.	6.69%	4.6%	1.4% - 7.8%	27619	Pass

Concentration: 0.302 ug/mL



Sample: VX



Segment: 4 Time(min): 23.989-32.983 Scans: 1423-2285 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:02:43

Ion Ratio Report for Target Compound #1 from v:\... vx-101702-us-3ds2-10-1

Saturn 2000 No.1

Sample ID:	VX-101702-US-3DS2-	Acquisition Date:	10/17/02 7:30 PM
Vial:	7	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extr action, no HAE cleanup; Initial Volume = 5mL, diluted 1:10 w /CH2Cl2,0.73ug VX Spike into dilution Rtx5MS;S-TRAP;EM@1750V		
Data File:	c:\... \2002\october\101702#1\vx-101702-us-3ds2-10-17-02.sms		

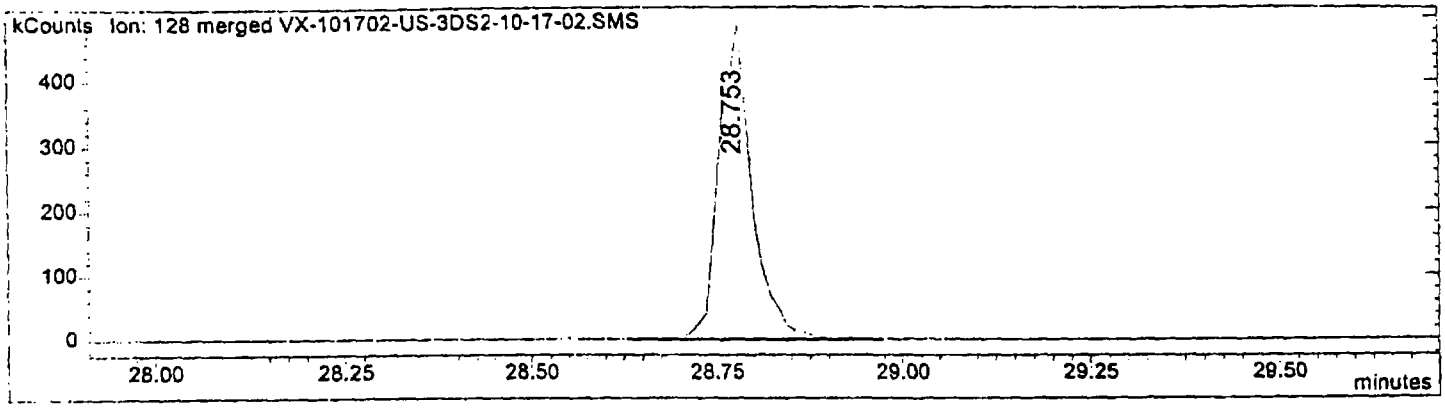
C31263 B-P-4
10-17-02

Analyte: VX

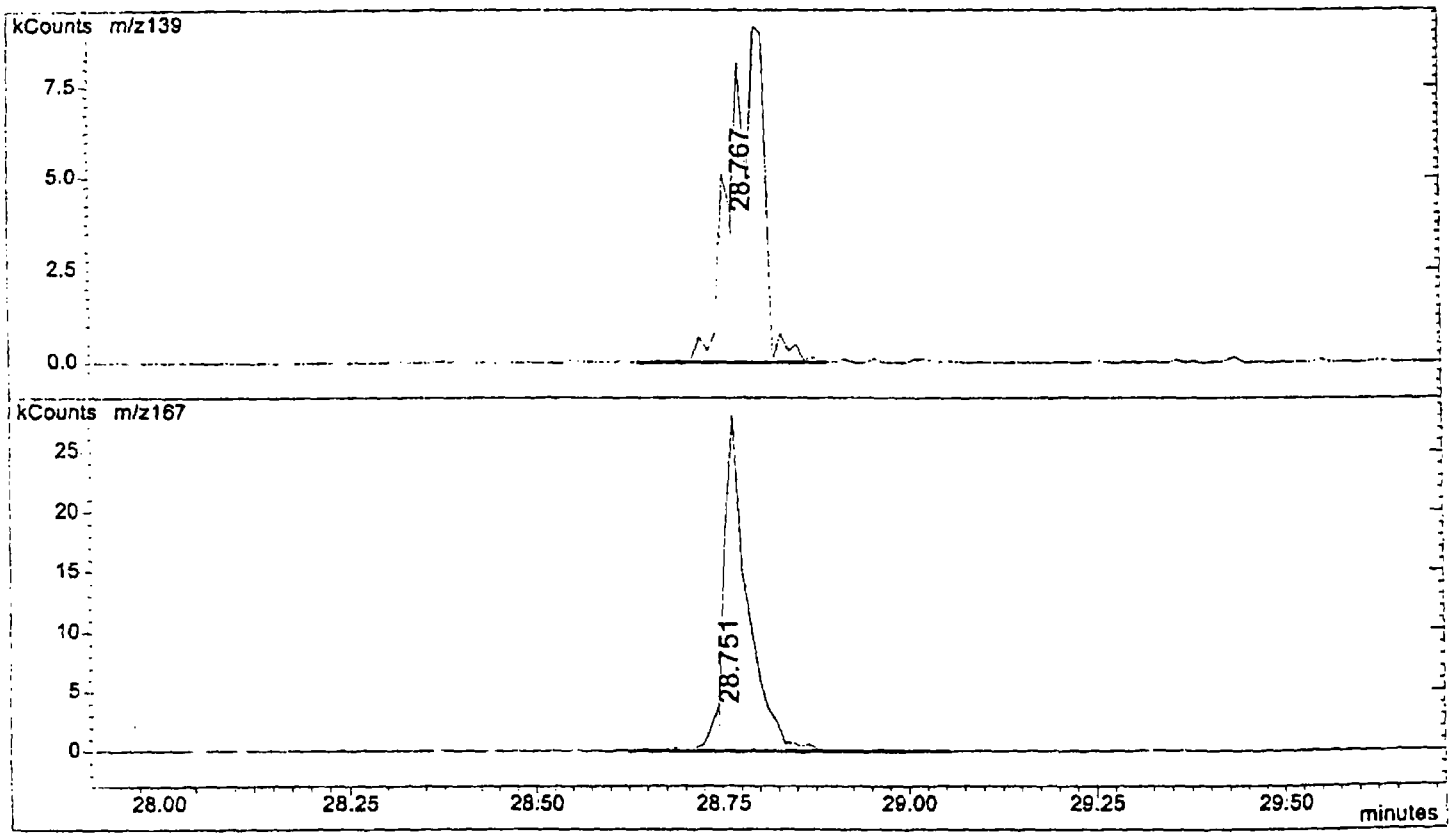
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.753 min.	28.812 min.	28.112 - 29.512 min.	1530021	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.767 min.	1.89%	2.0%	0.6% - 3.4%	28913	Pass
167	28.751 min.	4.11%	4.6%	1.4% - 7.8%	62823	Pass

Concentration: 1.020 ug/mL



Sample: VX



Segment: 4 Time(min): 23.986-32.988 Scans: 1423-2280 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:03:14

Ion Ratio Report for Target Compound #1 from VX-101602-US-4D1-10-17-02.SMS

Saturn 2000 No.1

Sample ID:	VX-101602-US-4D1-	Acquisition Date:	10/17/02 8:16 PM
Vial:	8	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extraction, no HAE cleanup; Initial Volume = 5mL, diluted 1:10 w /CH2Cl2; Rtx5MS;S-TRAP;EM@1750V		
Data File:	c:\... \2002\october\101702#1\vx-101602-us-4d1-10-17-02.sms		

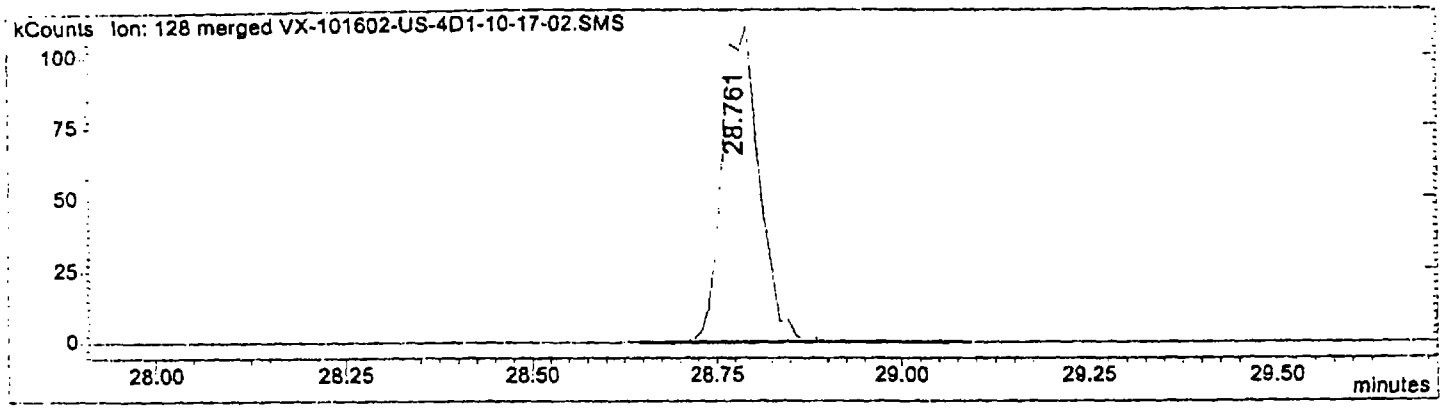
Y. Abdhu
C31763 Bk p.4
10-17-02

Analyte: VX

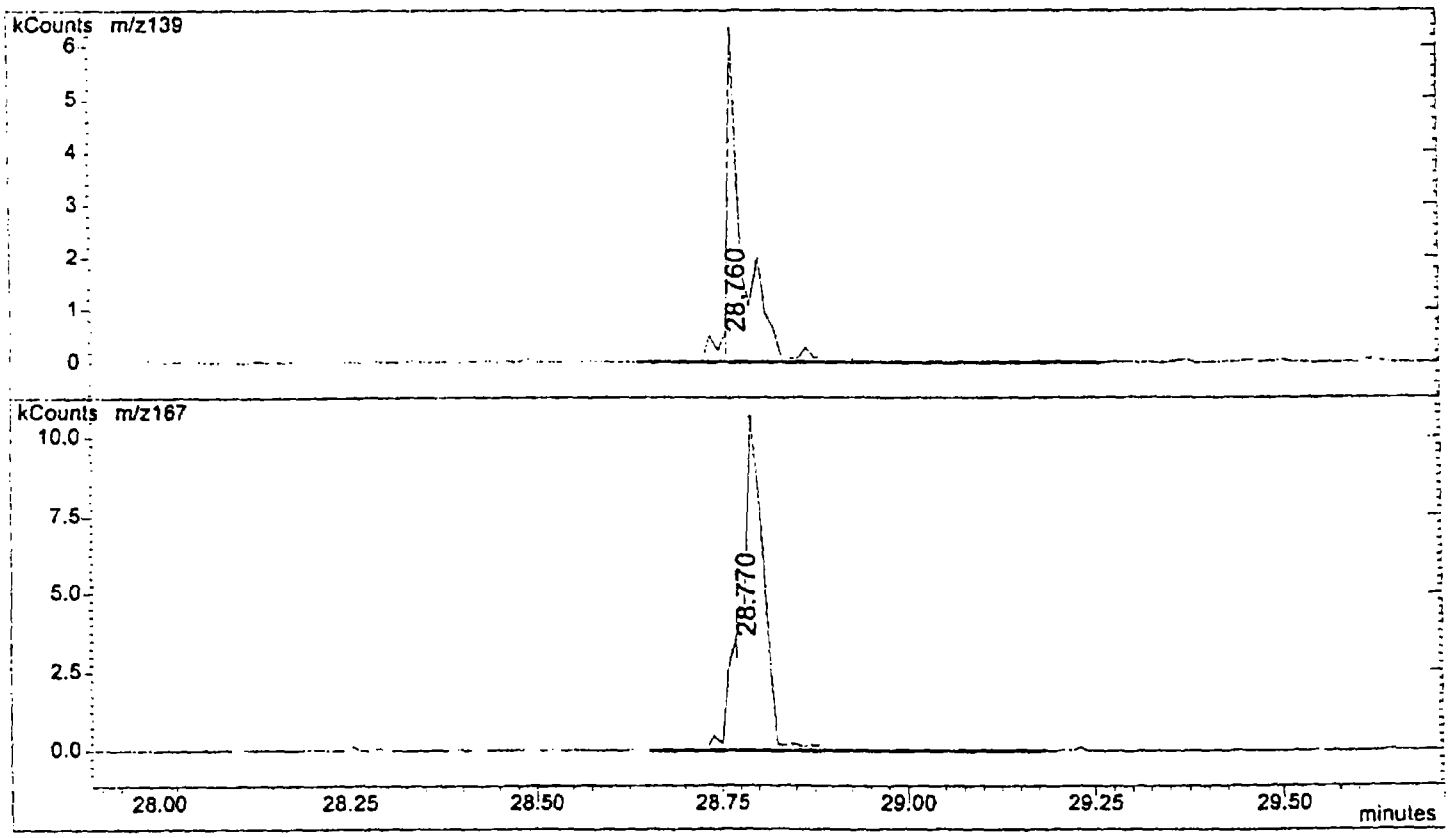
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.761 min.	28.812 min.	28.112 - 29.512 min.	389346	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.760 min.	2.53%	2.0%	0.6% - 3.4%	9835	Pass
167	28.770 min.	5.93%	4.6%	1.4% - 7.8%	23091	Pass

Concentration: 0.285 ug/mL



Sample: VX



Segment: 4 Time(min): 23.984-32.981 Scans: 1423-2275 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:03:44

Ion Ratio Report for Target Compound #1 from v:\... vx-101702-us-4ds2-10-1

Saturn 2000 No.1

Sample ID:	VX-101702-US-4DS2-	Acquisition Date:	10/17/02 9:00 PM
Vial:	9	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	JACADS Ash Sample No. LAWIC153B, 5g + DEAET; Ultrasonic Extraction, no HAE cleanup; Initial Volume = 5mL, diluted 1:10 w /CH2Cl2,0.73ug VX Spike into dilution Rtx5MS;S-TRAP;EM@1750V		
Data File:	c:\... \2002\october\101702#1\vx-101702-us-4ds2-10-17-02.sms		

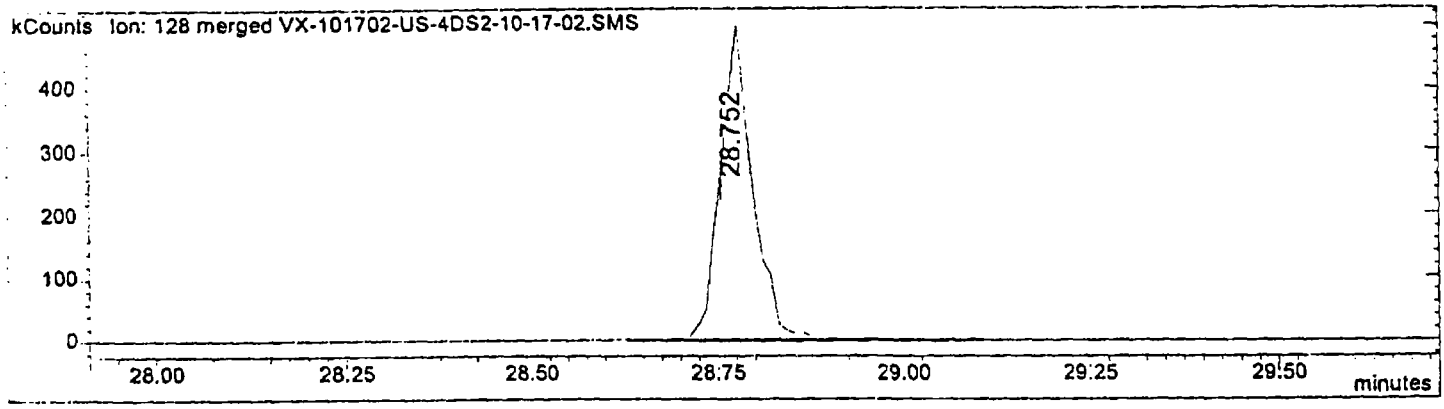
Y. Abitha
C 31763 Back P.
10-17-02

Analyte: VX

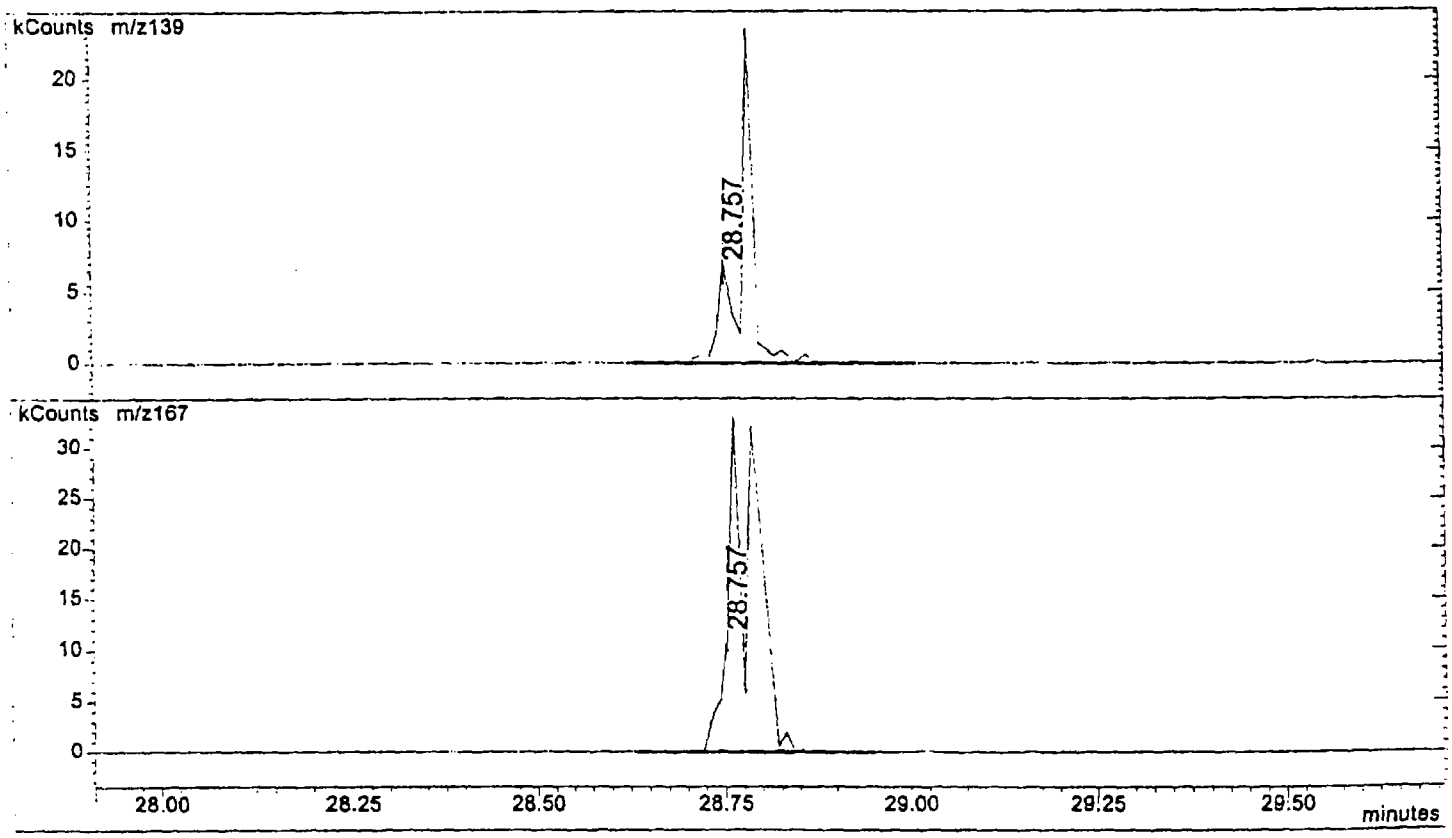
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.752 min.	28.812 min.	28.112 - 29.512 min.	1505668	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.757 min.	2.03%	2.0%	0.6% - 3.4%	30554	Pass
167	28.757 min.	5.74%	4.6%	1.4% - 7.8%	86401	Pass

Concentration: 1.006 ug/mL



Sample: VX



Segment: 4 Time(min): 23.999-32.988 Scans: 1422-2267 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:04:04

Ion Ratio Report for Target Compound #1 from ch2cl2 blank-10-17-02001.sms

Saturn 2000 No.1

ample ID:	CH2Cl2 Blank	Acquisition Date:	10/17/02 9:46 PM
Vial:	0	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	CH2Cl2 Blank, Rtx-5MS:30Mx0.25mm ID, 1.0um; 100C,5min - 240 C @5C/m-280C @25C/m,5min;0.42VEE;PIT@200usec; 0.65scans/sec; noPP;m/z268.2;1750V; S-TRAP@170C		
Data File:	c:\... \2002\october\101702#1\ch2cl2 blank-10-17-02001.sms		

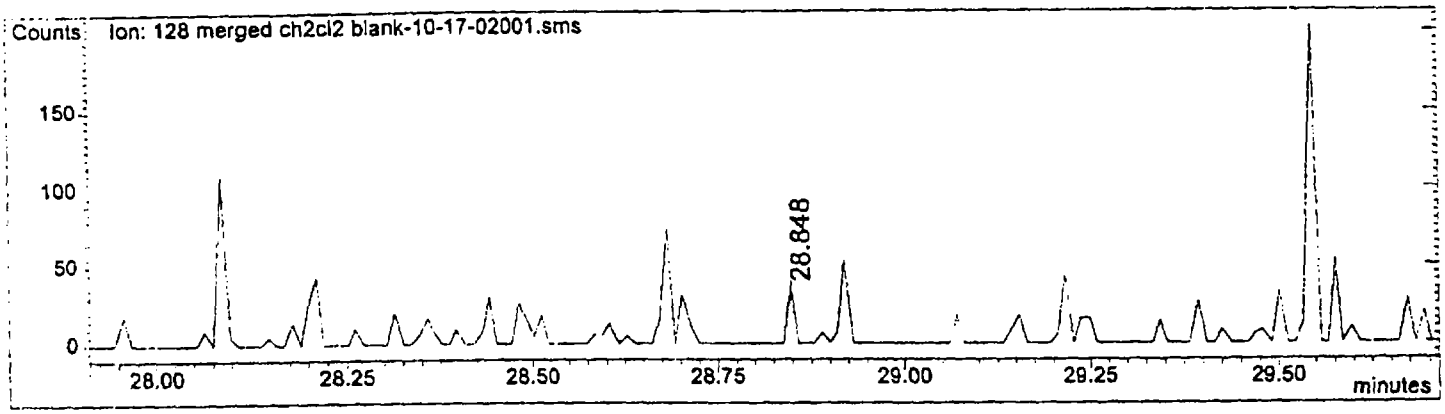
X-Abstract
C31763 Blank P4
10-17-02

Analyte: VX

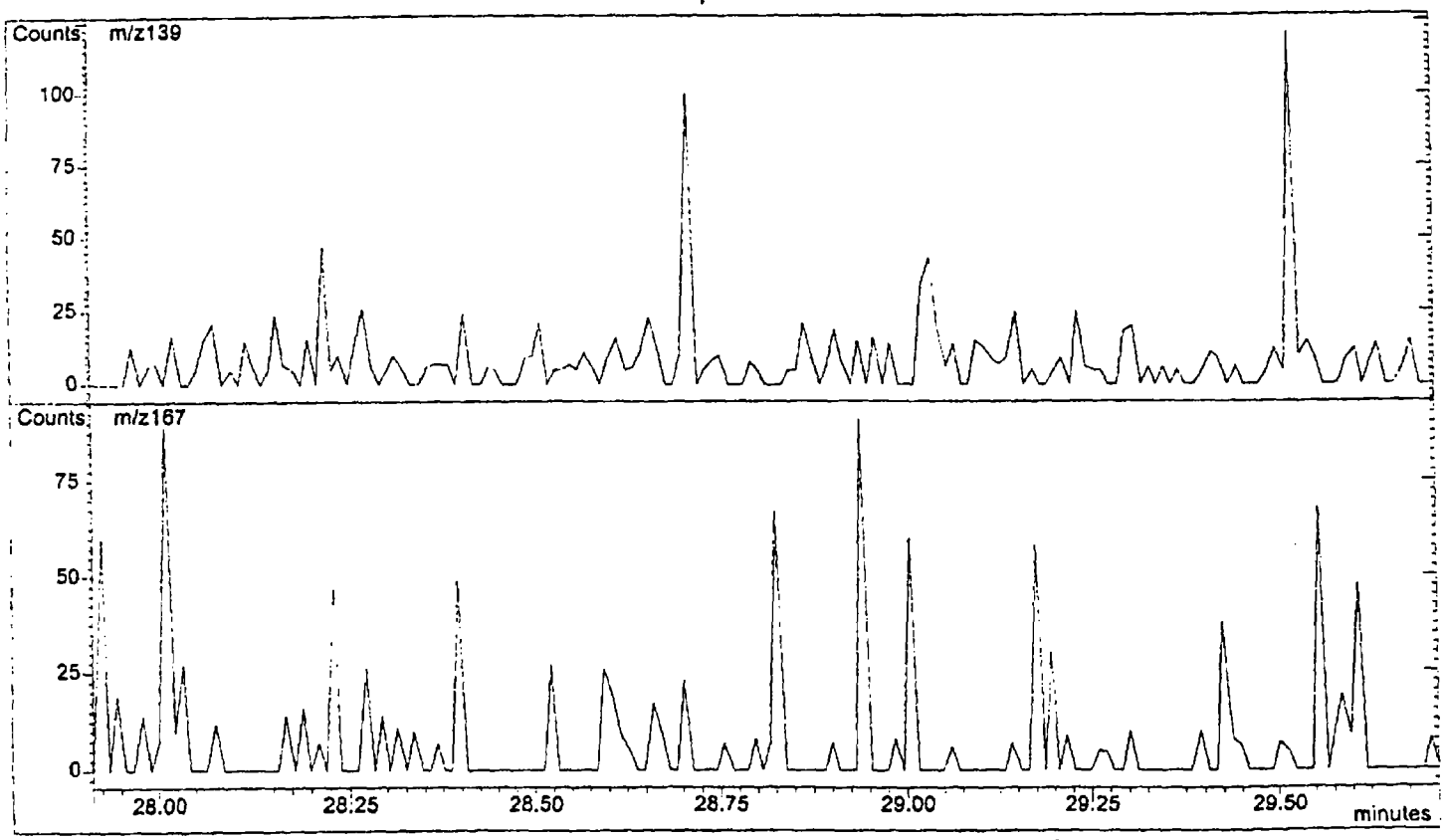
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.848 min.	28.812 min.	28.112 - 29.512 min.	0	Missing

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	0.000 min.	0.00%	2.0%	0.6% - 3.4%	0	Fail
167	0.000 min.	0.00%	4.6%	1.4% - 7.8%	0	Fail

Concentration: 0.000 ug/mL



Sample: VX



Segment: 4 Time(min): 23.998-32.987 Scans: 1422-2273 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:04:30

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-1, 1.8ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-1, 1.8ug	Acquisition Date:	10/17/02 10:31 PM
Vial:	10	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	1.83ug/mLVX Std, Rtx-5MS:30M x 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans /sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \2002\october\101702#1\xvx100302-1, 1.8ug10-17-02.sms		

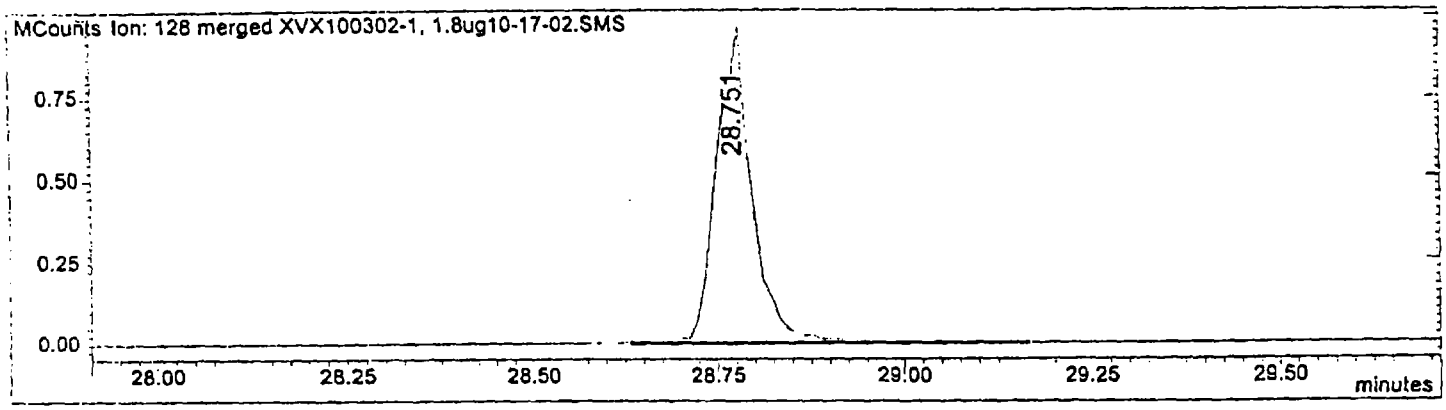
Y. Arthur
C31763 p-9
10-22-02

Analyte: VX

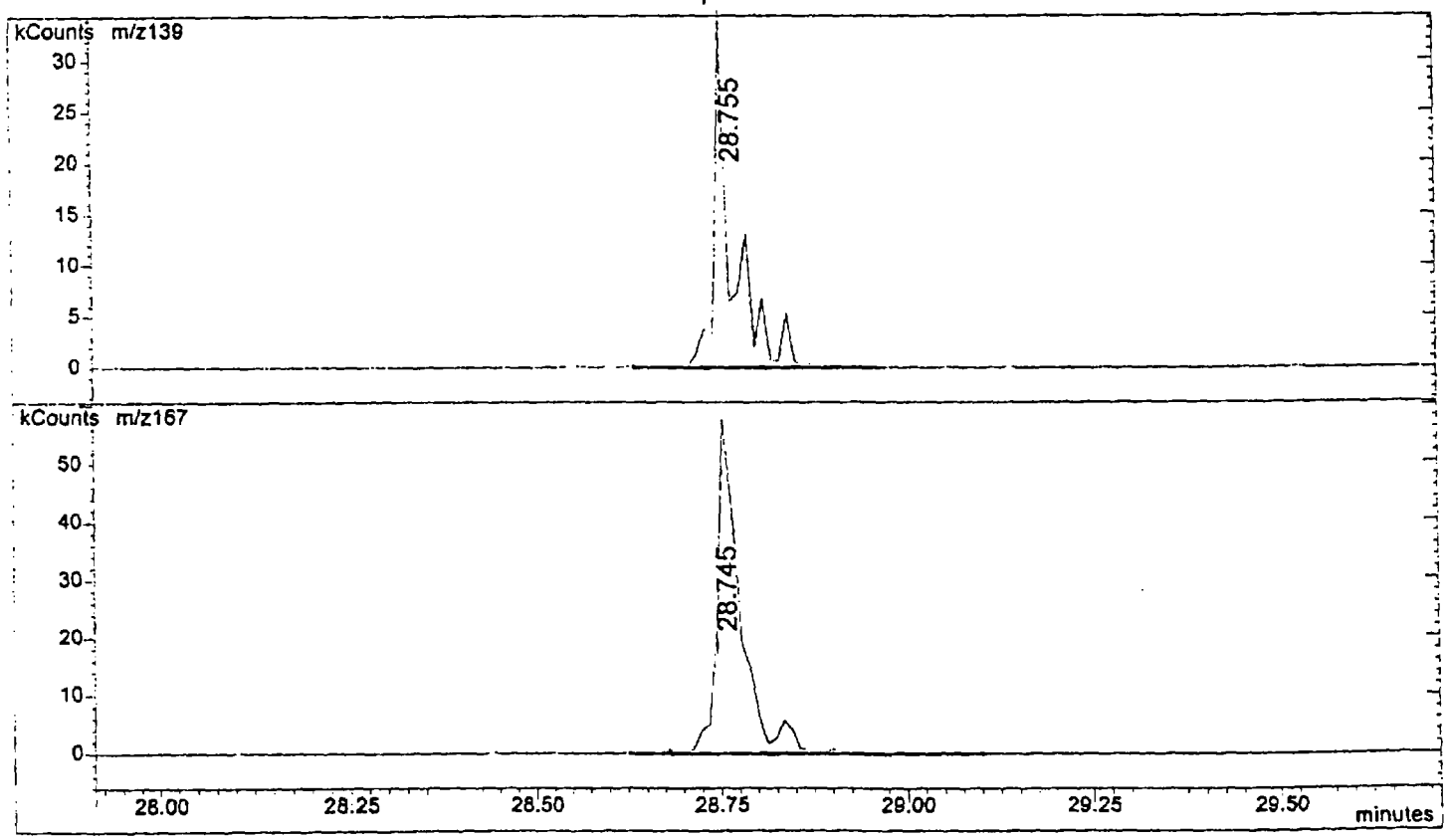
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.751 min.	28.812 min.	28.112 - 29.512 min.	3226284	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.755 min.	1.78%	2.0%	0.6% - 3.4%	57438	Pass
167	28.745 min.	3.94%	4.6%	1.4% - 7.8%	127274	Pass

Concentration: 1.919 ug/mL



Sample: VX



Segment: 4 Time(min): 23.997-32.980 Scans: 1422-2268 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:04:43

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-1, 1.8ug10-1

Saturn 2000 No.1

ample ID:	XVX100302-1, 1.8ug	Acquisition Date:	10/17/02 11:16 PM
Vial:	10	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	1.83ug/mLVX Std, Rtx-5MS:30M x 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans /sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \october\101702#1\xvx100302-1, 1.8ug10-17-02001.sms		

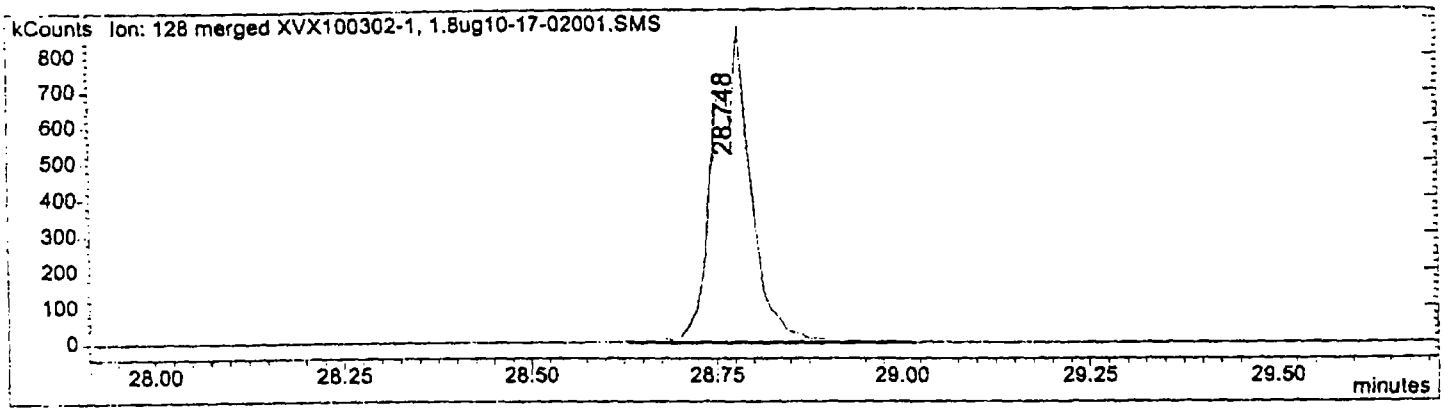
Y. Arthur
C 31763 B P-9
10-17-02

Analyte: VX

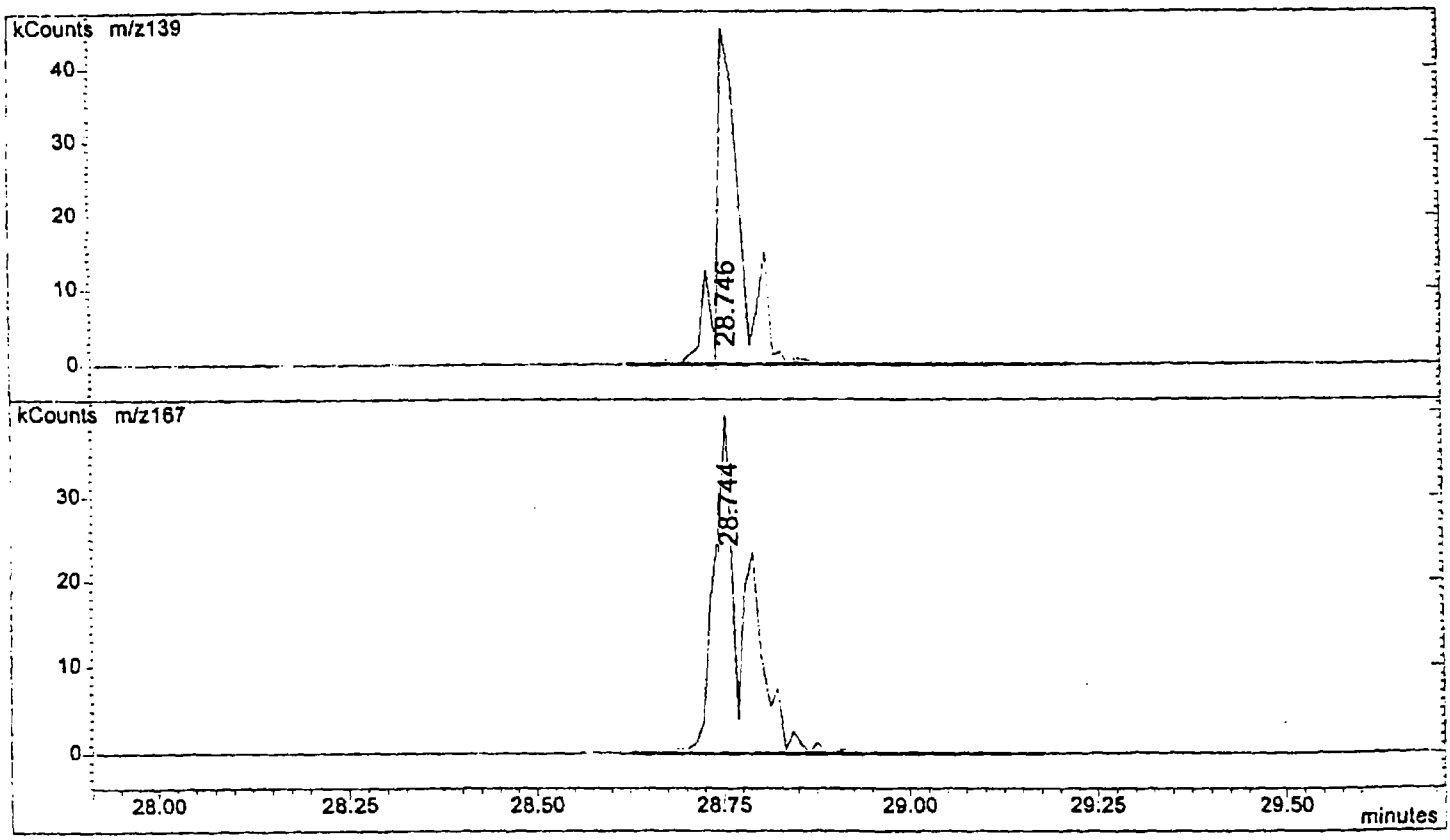
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.748 min.	28.812 min.	28.112 - 29.512 min.	3054257	Failed

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.746 min.	3.43%	2.0%	0.6% - 3.4%	104874	Fail
167	28.744 min.	3.71%	4.6%	1.4% - 7.8%	113214	Pass

Concentration: 1.835 ug/mL



Sample: VX



Segment: 4 Time(min): 23.989-32.987 Scans: 1421-2270 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:04:58

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-2,0.91ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-2,0.91ug	Acquisition Date:	10/18/02 12:01 AM
Vial:	11	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	0.913ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42V-EE;PIT@200usec;0.65scans /sec;noPP;m/z268.2;1750V;TS-RAP@170C;Sngl-GN		
Data File:	c:\... \2002\october\101702#1\xvx100302-2,0.91ug10-18-02.sms		

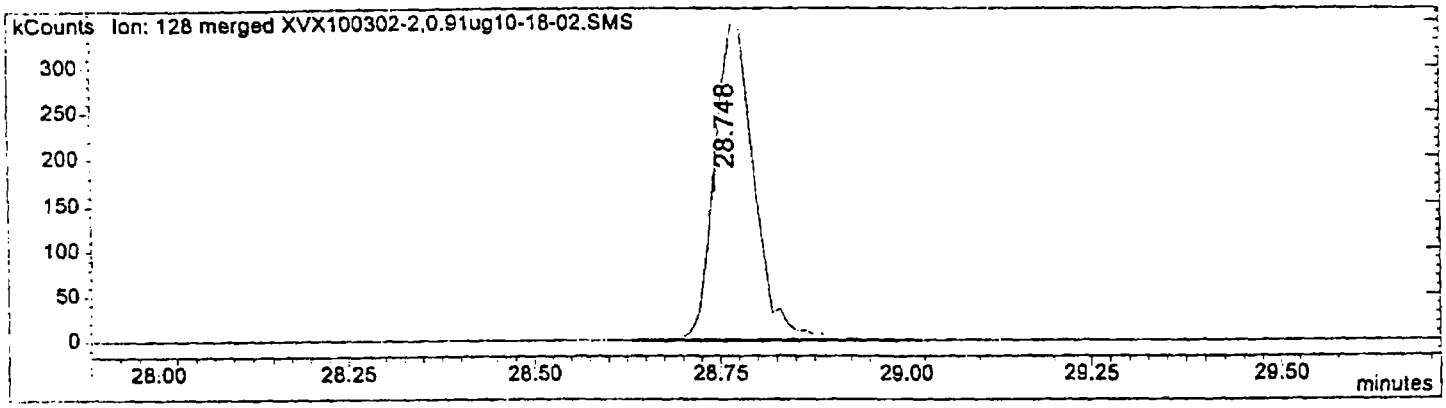
Y. Abshu
C31763 Bk P:
10-17-02

Analyte: VX

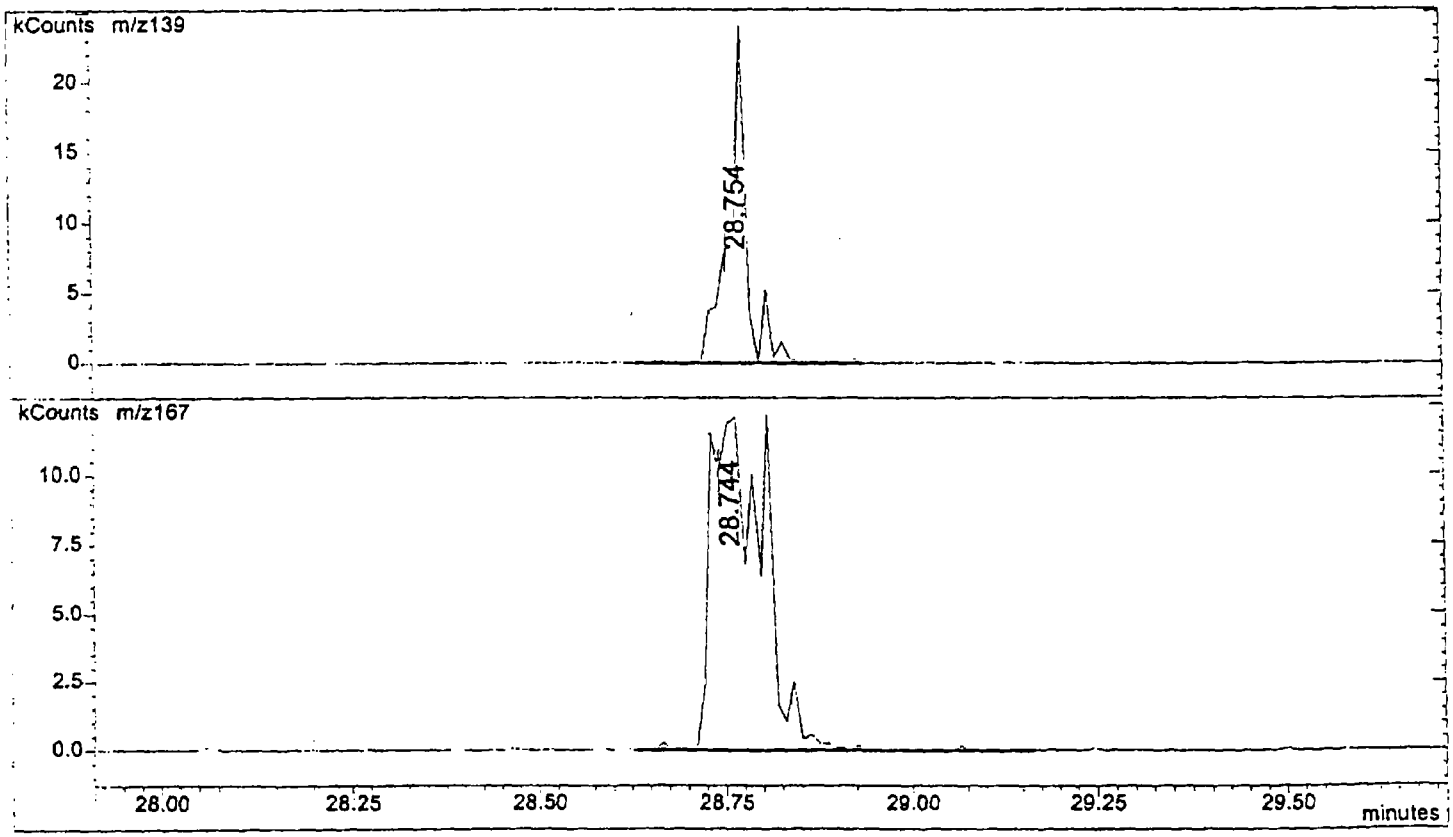
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.748 min.	28.812 min.	28.112 - 29.512 min.	1281206	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.754 min.	3.10%	2.0%	0.6% - 3.4%	39659	Pass
167	28.744 min.	4.61%	4.6%	1.4% - 7.8%	59017	Pass

Concentration: 0.871 ug/mL



Sample: VX



Segment: 4 Time(min): 23.998-32.980 Scans: 1422-2271 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:05:11

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/18/02 12:46 AM
Vial:	5	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \2002\october\101702#1\xvx100302-3,0.46ug10-18-02.sms		

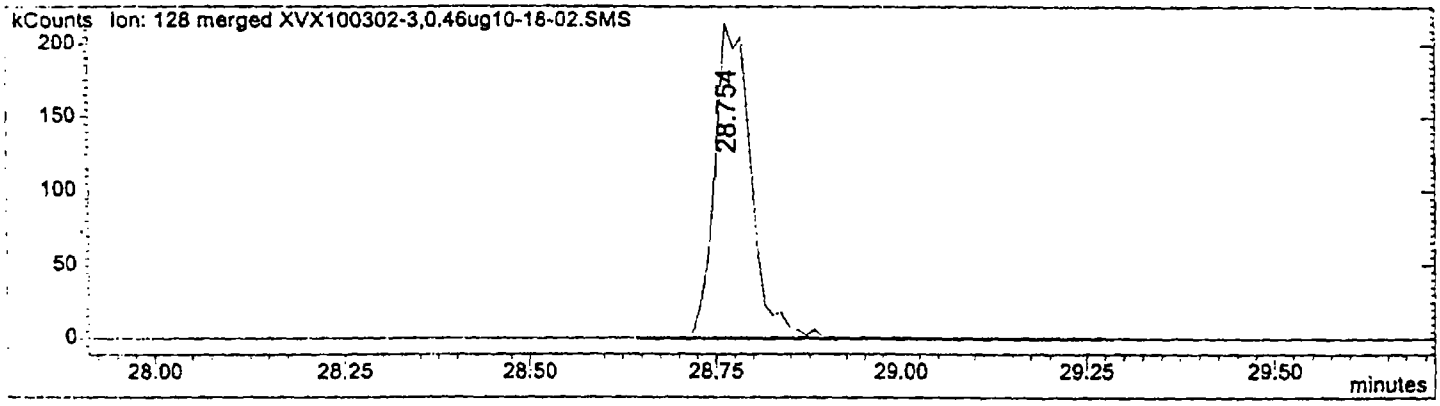
Y. Abitha
C31763 Bulp.
10-17-02

Analyte: VX

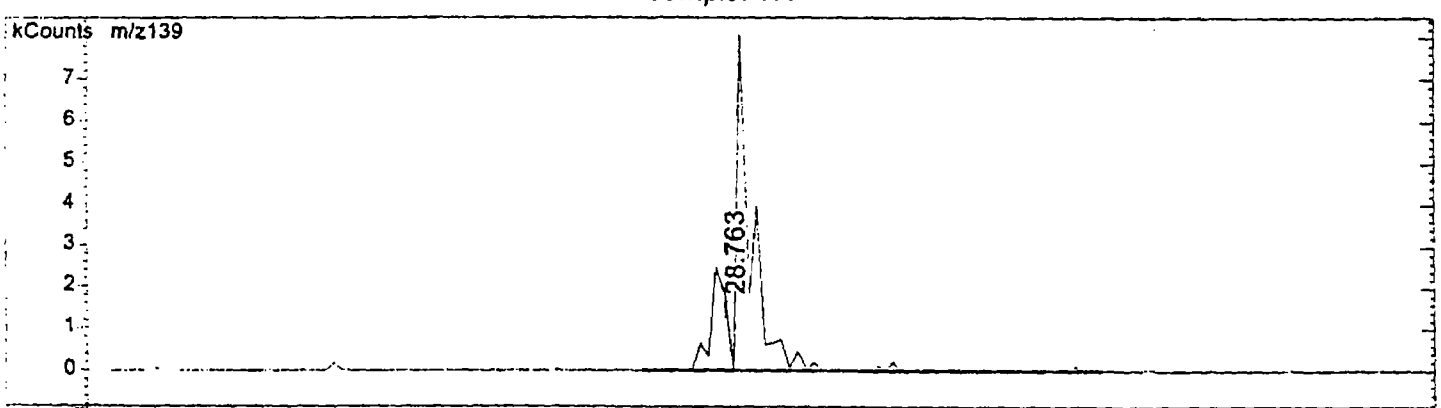
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.754 min.	28.812 min.	28.112 - 29.512 min.	735341	Identified

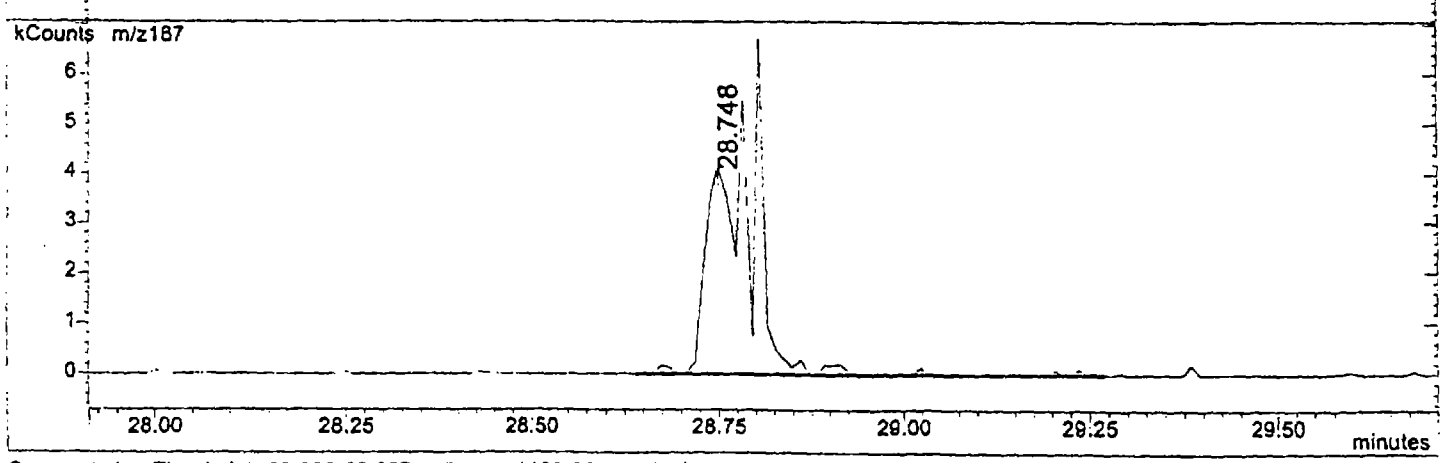
Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.763 min.	2.01%	2.0%	0.6% - 3.4%	14803	Pass
167	28.748 min.	2.83%	4.6%	1.4% - 7.8%	20847	Pass

Concentration: 0.524 ug/mL



Sample: VX





Segment: 4 Time(min): 23.996-32.987 Scans: 1422-2273 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:05:21

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-4,0.18ug10-1

Saturn 2000 No:1

Sample ID:	XVX100302-4,0.18ug	Acquisition Date:	10/18/02 1:31 AM
Vial:	12	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	0.183ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \2002\octqber\101702#1\xvx100302-4,0.18ug10-18-02.sms		

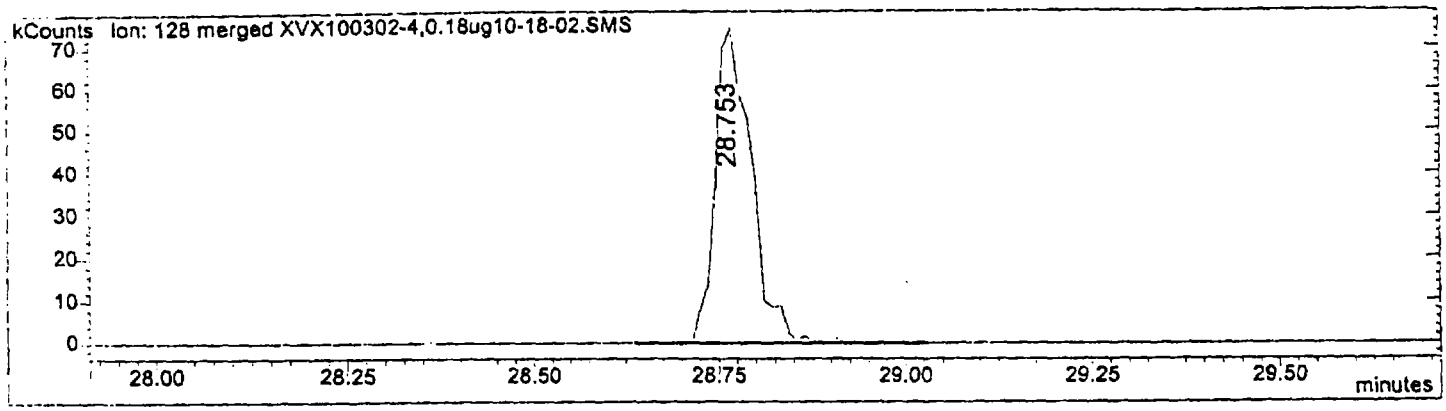
Y. Arthur
C31763 Rec'd P-4
10-17-02

Analyte: VX

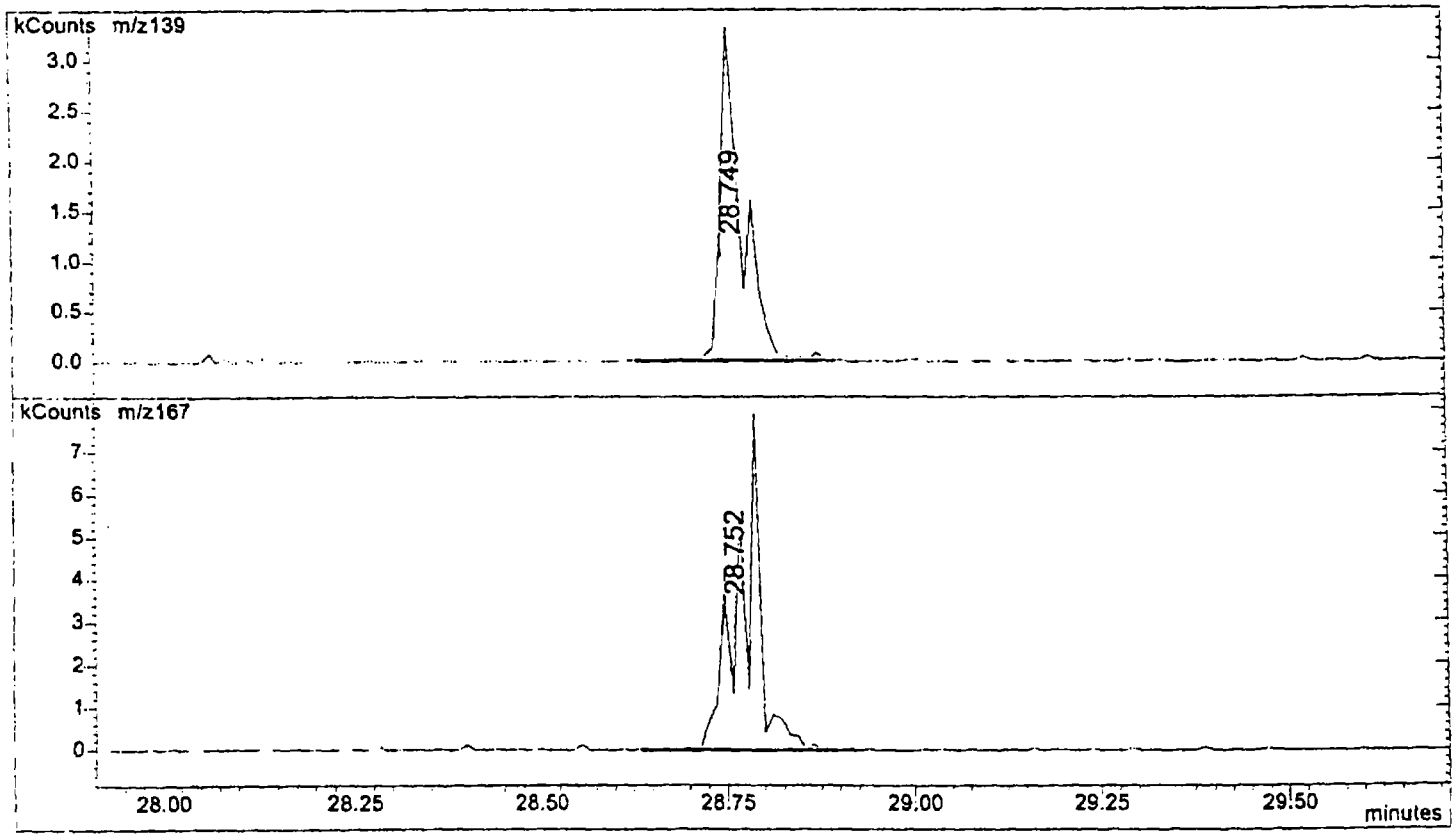
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.753 min.	28.812 min.	28.112 - 29.512 min.	251252	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.749 min.	2.63%	2.0%	0.6% - 3.4%	6609	Pass
167	28.752 min.	6.22%	4.6%	1.4% - 7.8%	15632	Pass

Concentration: 0.186 ug/mL



Sample: VX



Segment: 4 Time(min): 23.993-32.989 Scans: 1422-2277 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:05:34

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-5,.091ug10-1

Saturn 2000 No.1

ample ID:	XVX100302-5,.091ug	Acquisition Date:	10/18/02 2:16 AM
Vial:	13	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	0.091ug/mLVX Std Rtx-5Sil MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \2002\october\101702#1\xvx100302-5,.091ug10-18-02.sms		

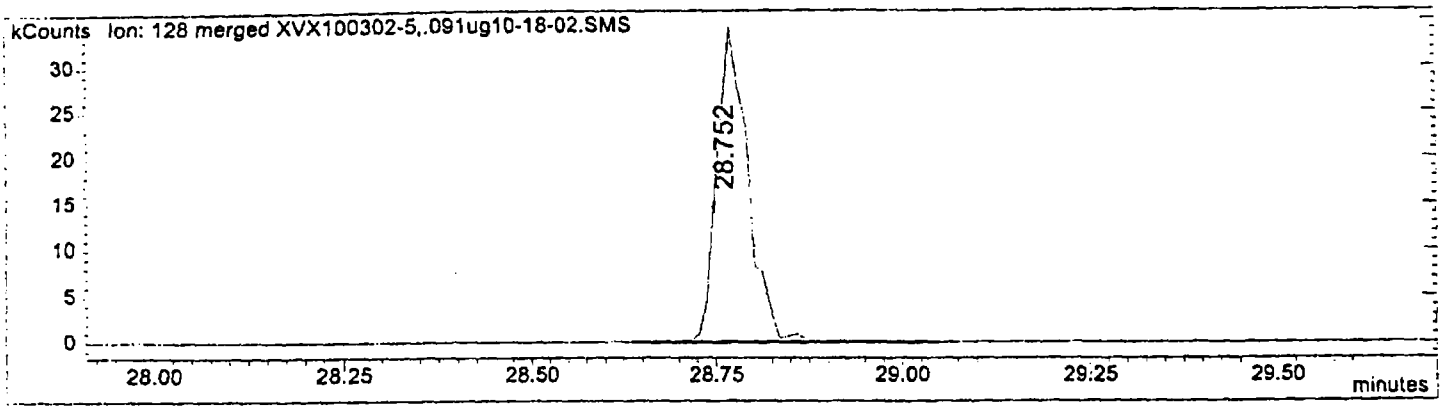
Y-[illegible]
C31763B[illegible]P-4
10-17-02

Analyte: VX

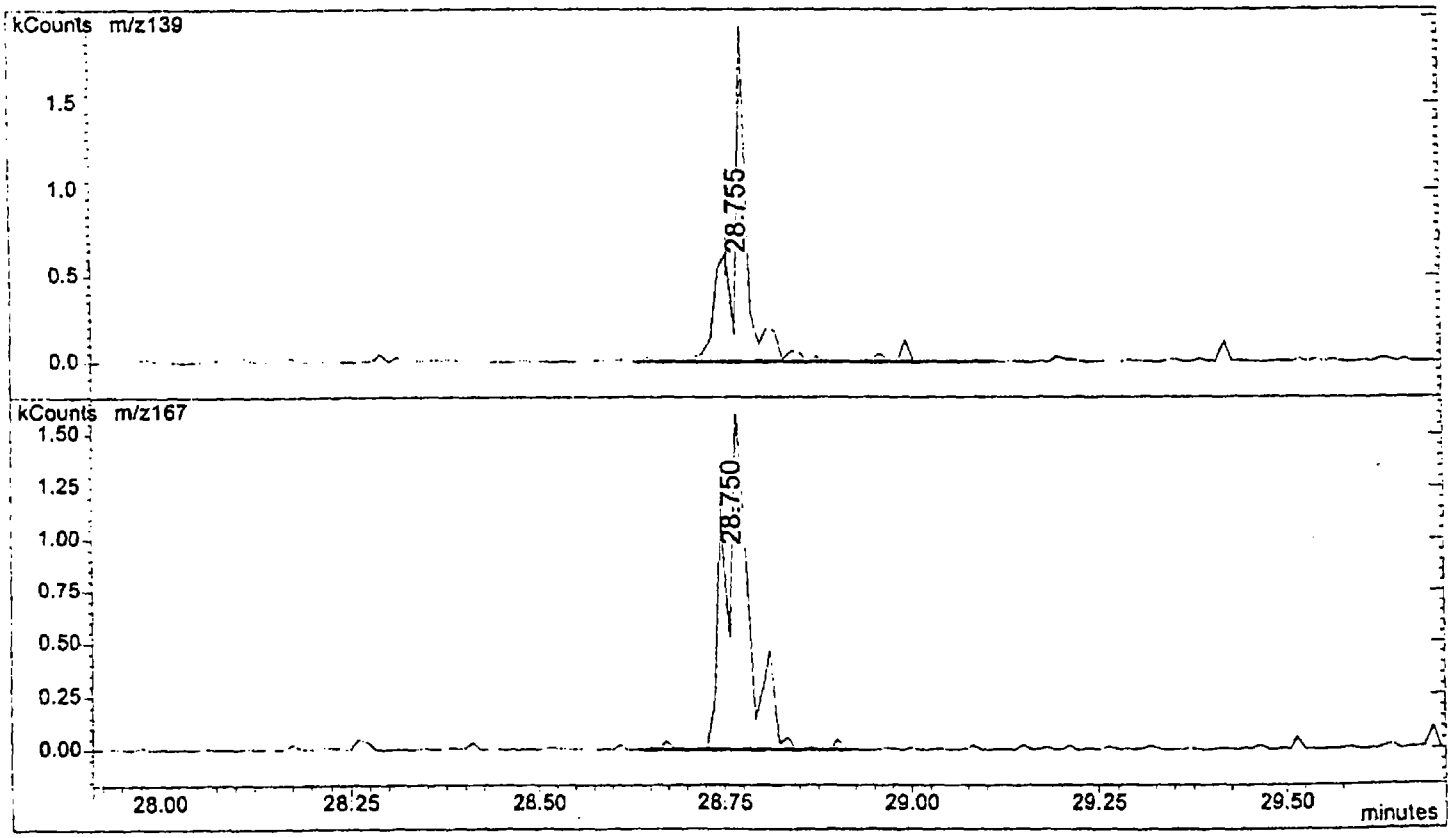
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.752 min.	28.812 min.	28.112 - 29.512 min.	99121	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.755 min.	2.86%	2.0%	0.6% - 3.4%	2838	Pass
167	28.750 min.	3.61%	4.6%	1.4% - 7.8%	3580	Pass

Concentration: 0.072 ug/mL



Sample: VX



Segment: 4 Time(min): 23.994-32.986 Scans: 1422-2274 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:05:49

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-6,.046ug10-1

Saturn 2000 No.1

ample ID:	XVX100302-6,.046ug	Acquisition Date:	10/18/02 3:01 AM
Vial:	14	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	0.046ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \2002\october\101702#1\xvx100302-6,.046ug10-18-02.sms		

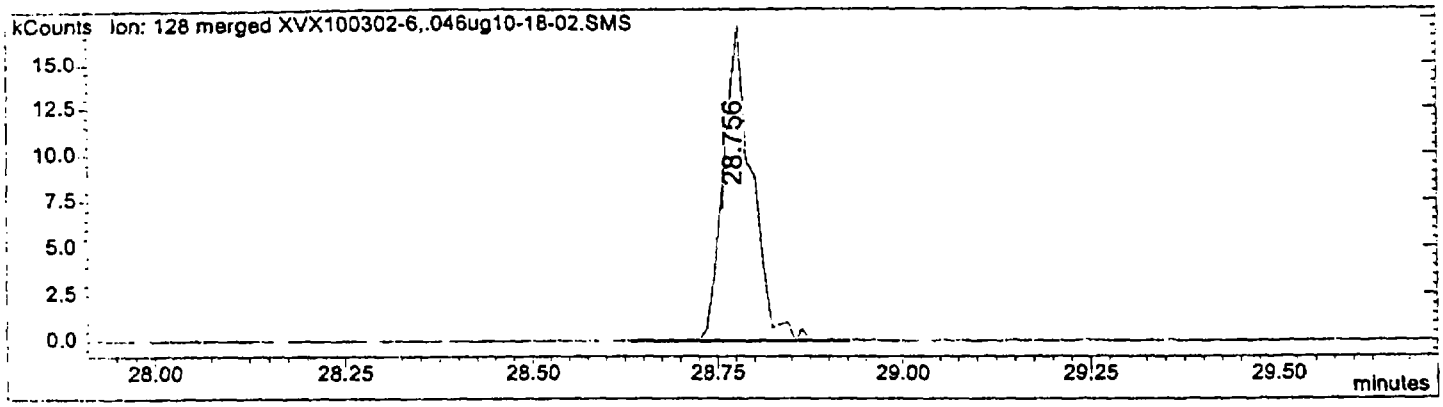
Y. Arthur
C31763 Book P-
10-17-02

Analyte: VX

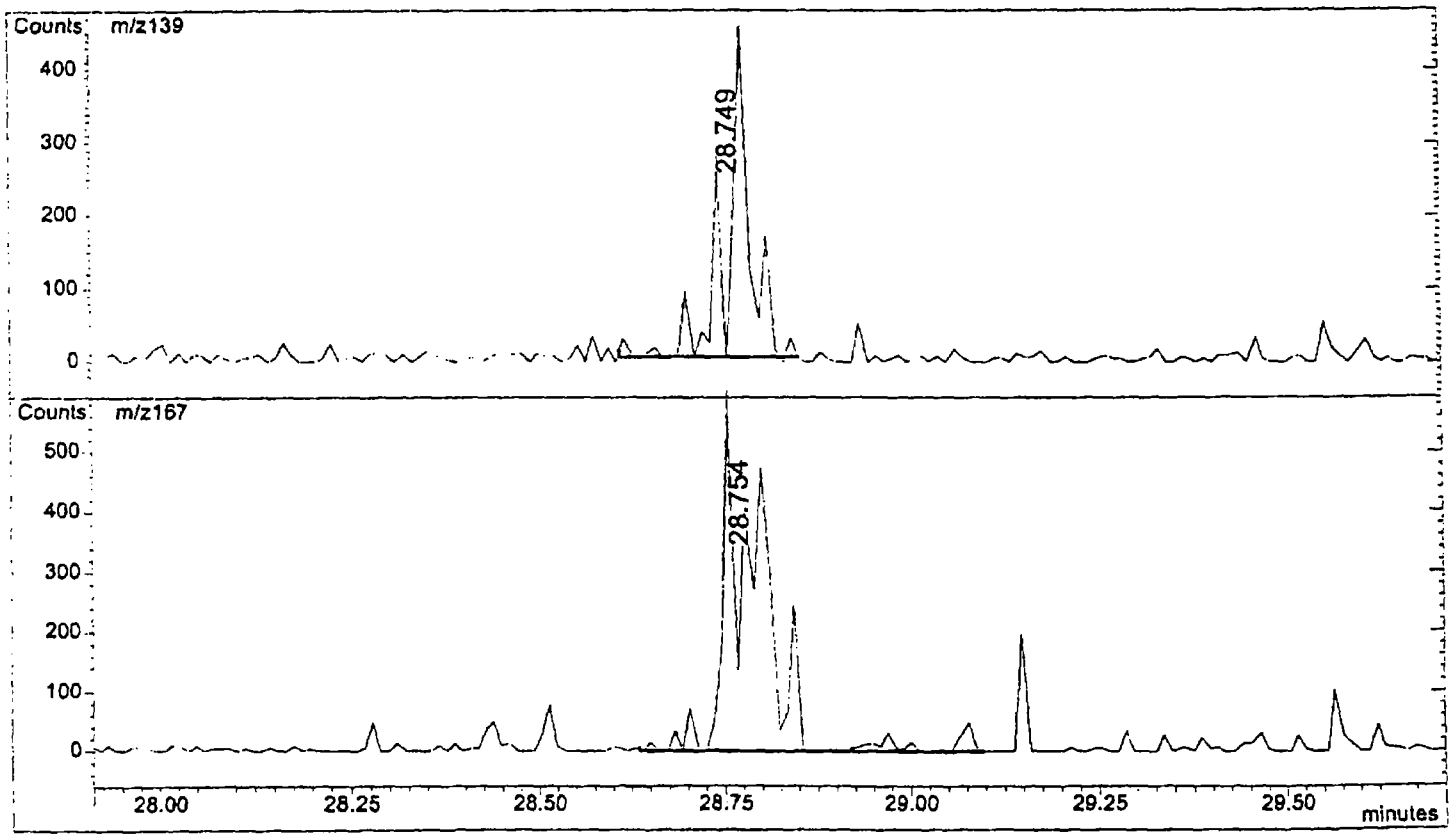
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.756 min.	28.812 min.	28.112 - 29.512 min.	45644	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.749 min.	2.09%	2.0%	0.6% - 3.4%	952	Pass
167	28.754 min.	4.03%	4.6%	1.4% - 7.8%	1840	Pass

Concentration: 0.031 ug/mL



Sample: VX



Segment: 4 Time(min): 23.992-32.979 Scans: 1422-2276 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:06:01

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

ample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/18/02 3:46 AM
Vial:	5	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \october\101702#1\xvx100302-3,0.46ug10-18-02001.sms		

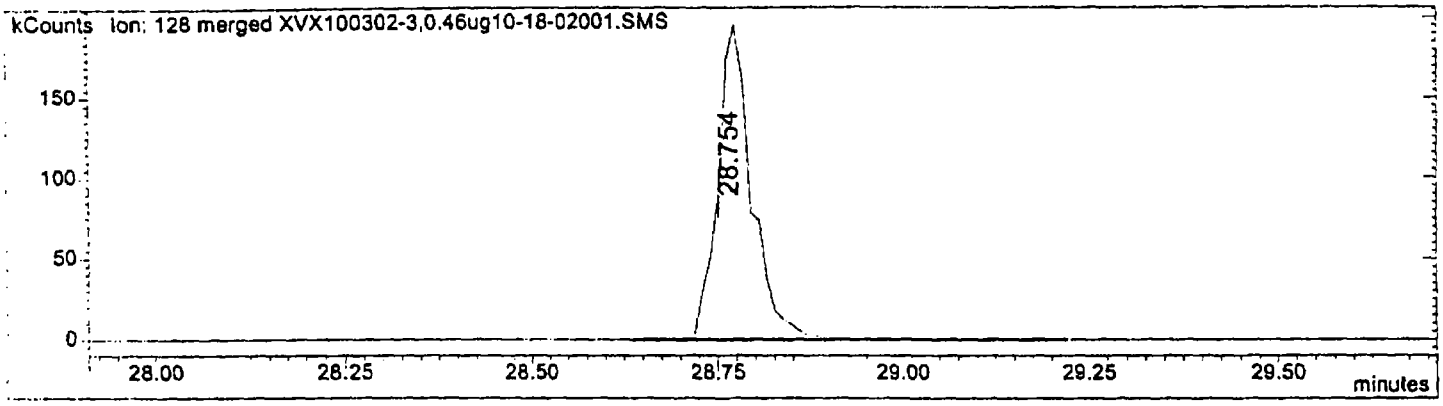
Y-Abbott
C31763 Back P-
10-17-02

Analyte: VX

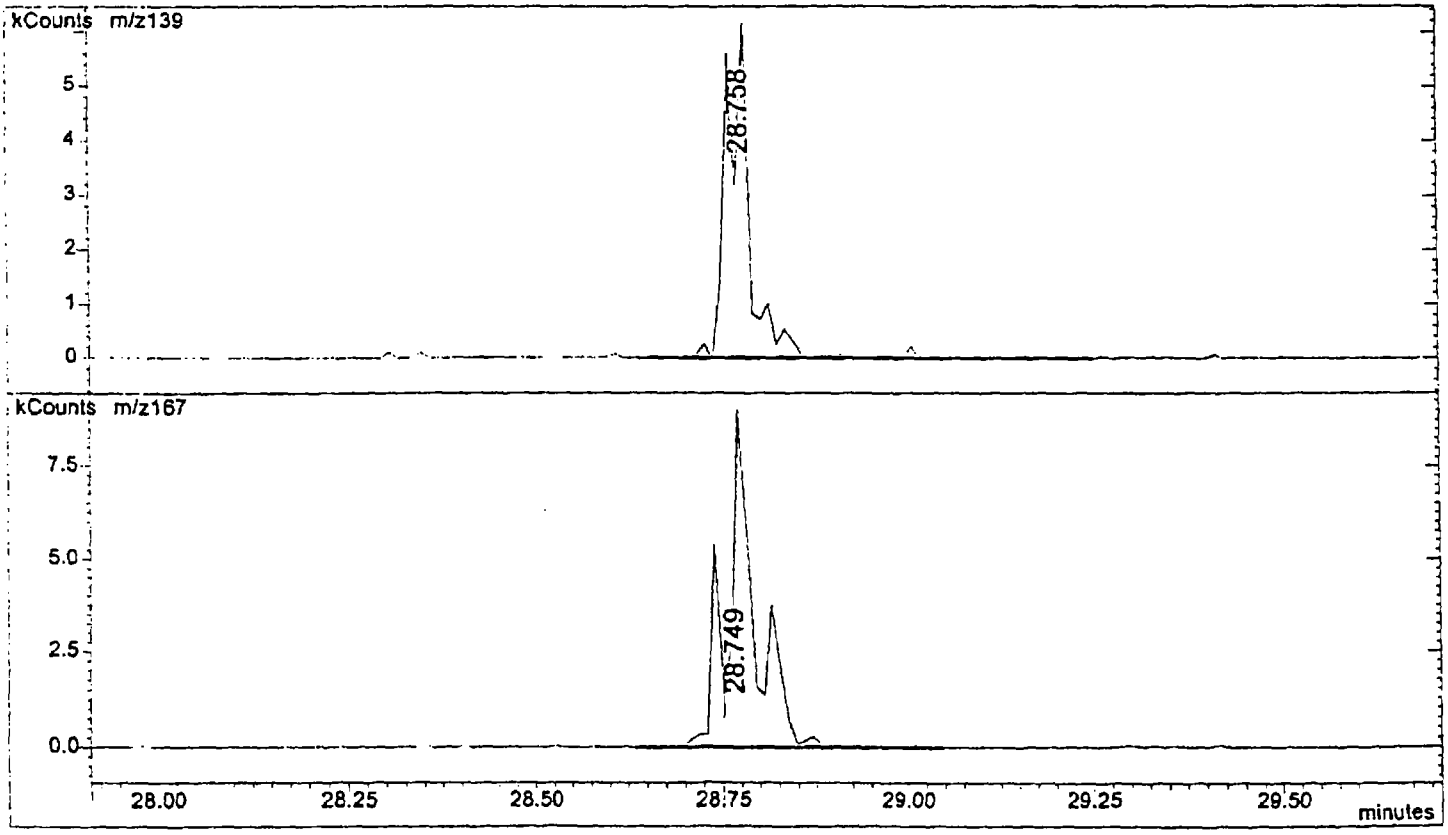
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.754 min.	28.812 min.	28.112 - 29.512 min.	620127	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.758 min.	2.10%	2.0%	0.6% - 3.4%	13019	Pass
167	28.749 min.	3.70%	4.6%	1.4% - 7.8%	22929	Pass

Concentration: 0.446 ug/mL



Sample: VX



Segment: 4 Time(min): 23.999-32.979 Scans: 1422-2273 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:06:19

Ion Ratio Report for Target Compound #1 from vx-101702-us-1-10-18-02.sms

Saturn 2000 No.1

ample ID:	VX-101702-US-1	Acquisition Date:	10/18/02 4:31 AM
al:	15	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	JACADS Ash Sample No. LAWIC153B, 1g + 5g NaSO4; Ultrasonic E xtraction, no HAE cleanup; Final Volume = 5mL + DEAET; Unspi ked; Rtx5MS;S-TRAP;EM@1750V		
Data File:	c:\... \2002\october\101702#1\vx-101702-us-1-10-18-02.sms		

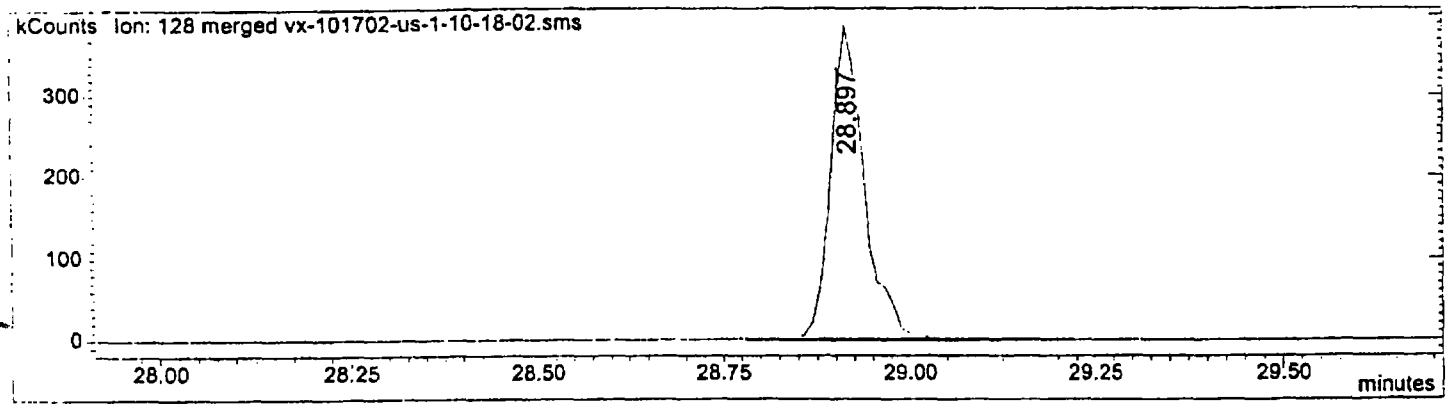
Y. Ahsher
C31763 Bk-1-9
10-17-02

Analyte: VX

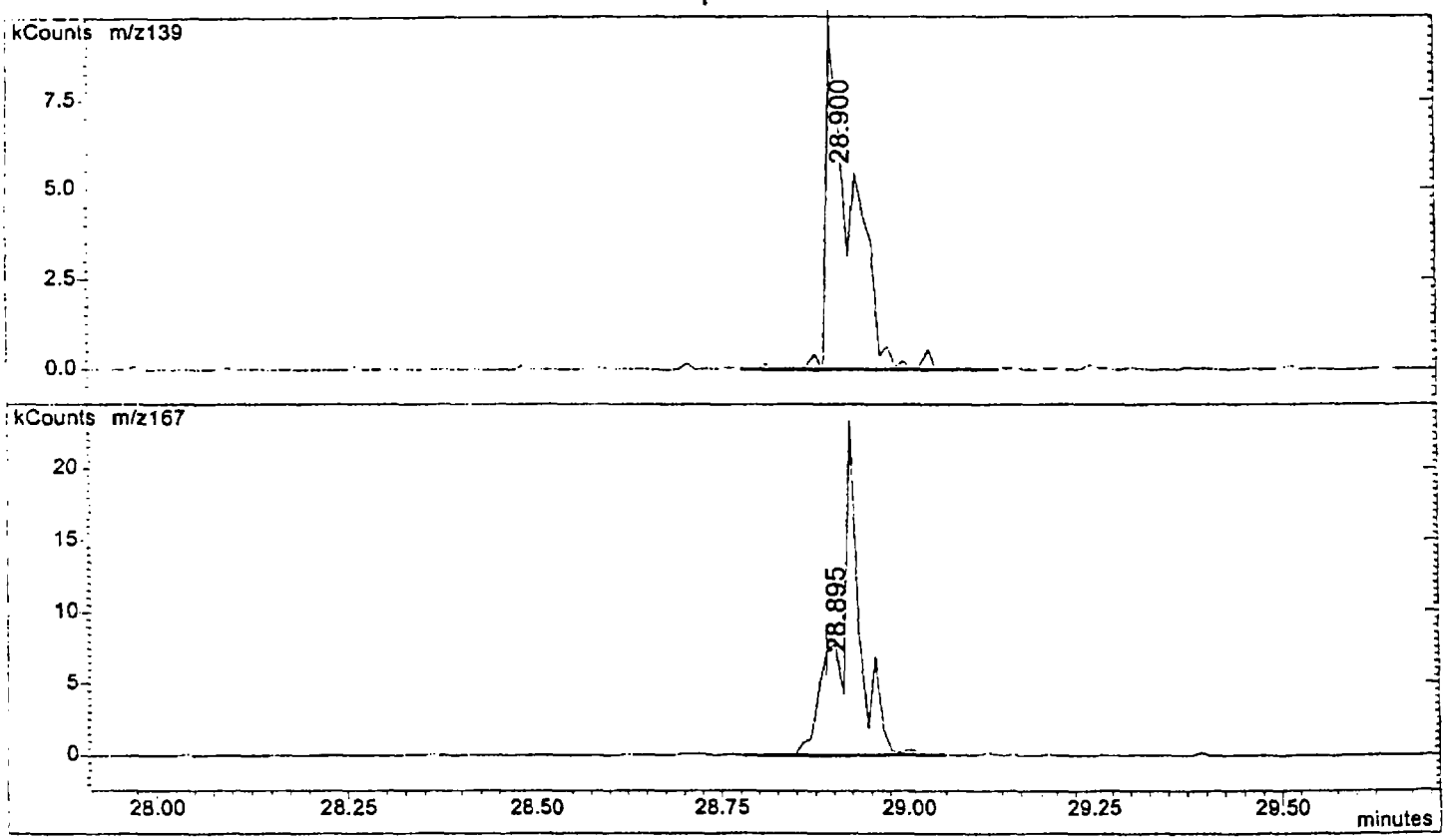
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.897 min.	28.812 min.	28.112 - 29.512 min.	1234240	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.900 min.	1.91%	2.0%	0.6% - 3.4%	23610	Pass
167	28.895 min.	3.66%	4.6%	1.4% - 7.8%	45222	Pass

Concentration: 0.843 ug/mL



Sample: VX



Segment: 4 Time(min): 23.985-32.985 Scans: 1421-2270 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:06:30

Ion Ratio Report for Target Compound #1 from vx-101702-us-2-10-18-02.sms

Saturn 2000 No.1

ample ID:	VX-101702-US-2	Acquisition Date:	10/18/02 5:16 AM
ial:	16	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	JACADS Ash Sample No. LAWIC153B, 1g + 5g NaSO4; Ultrasonic E xtraction, no HAE cleanup; Final Volume = 5mL + DEAET; Unspi ked; Rtx5MS;S-TRAP;EM@1750V		
Data File:	c:\... \2002\october\101702#1\vx-101702-us-2-10-18-02.sms		

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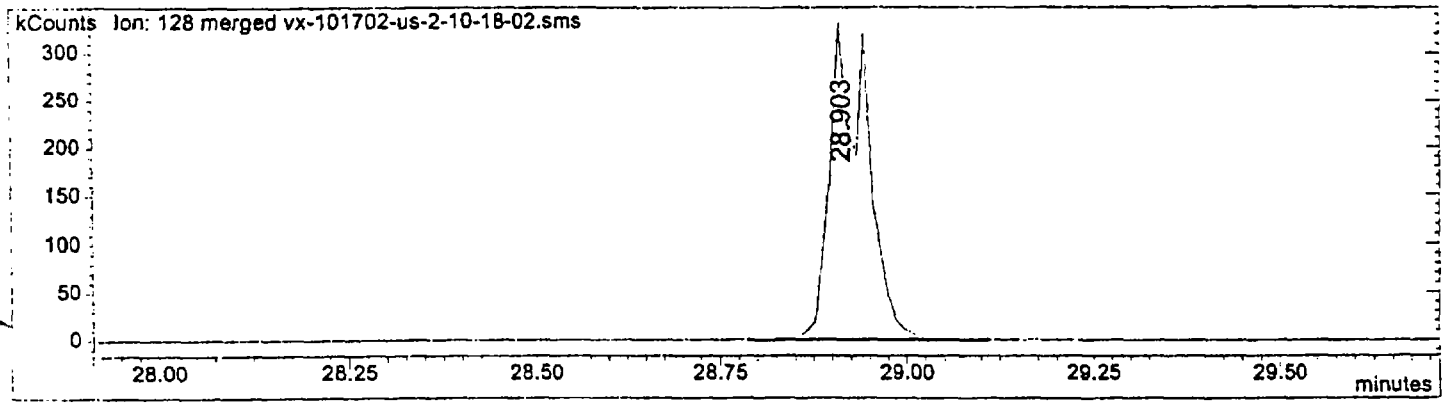
C31763 [illegible] P-4
10-17-02

Analyte: VX

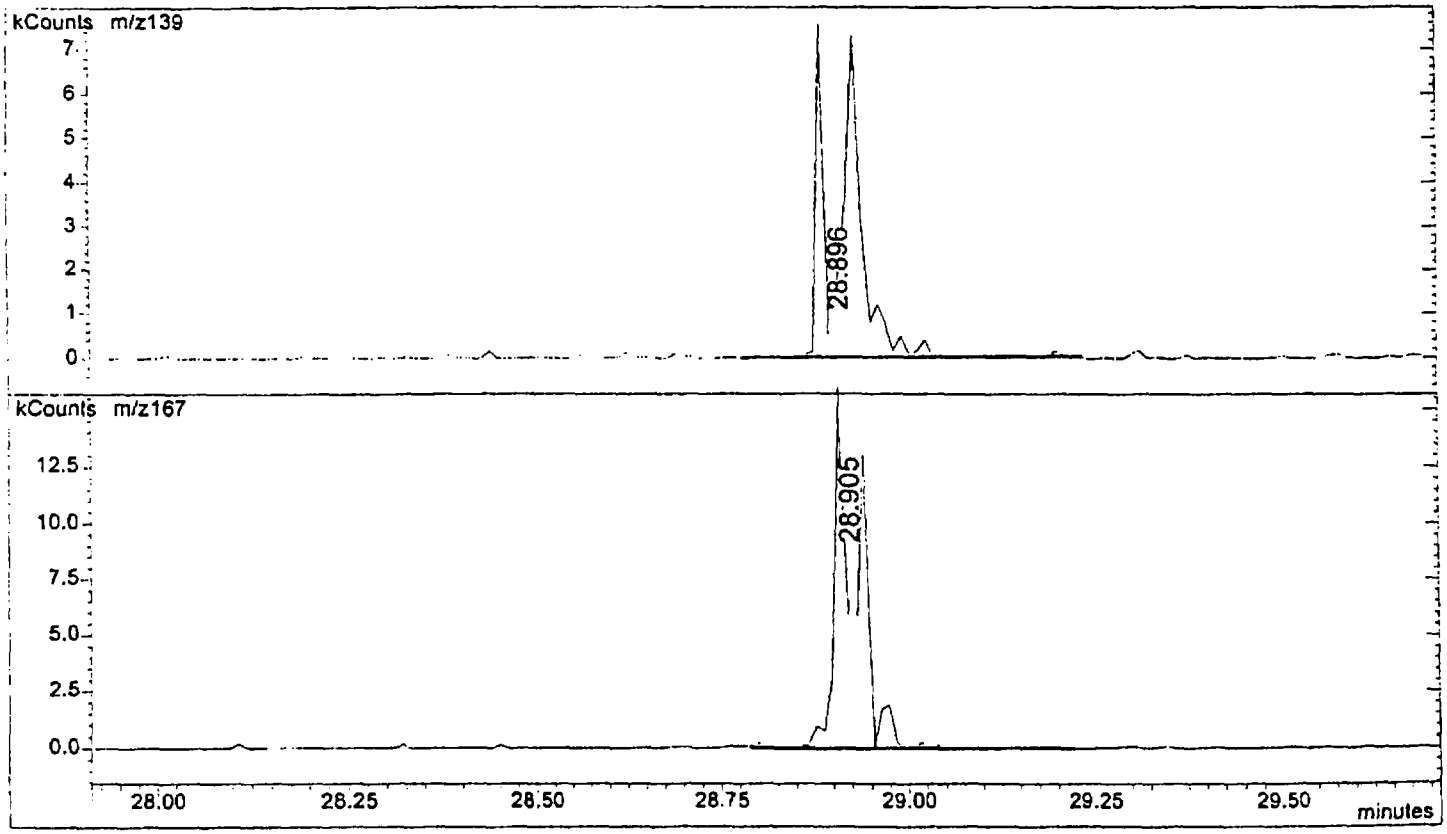
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.903 min.	28.812 min.	28.112 - 29.512 min.	1126732	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.896 min.	1.62%	2.0%	0.6% - 3.4%	18273	Pass
167	28.905 min.	2.92%	4.6%	1.4% - 7.8%	32926	Pass

Concentration: 0.776 ug/mL



Sample: VX



Segment: 4 Time(min): 23.981-32.981 Scans: 1421-2262 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:06:40

Ion Ratio Report for Target Compound #1 from vx-101702-us-3-10-18-02.sms

Saturn 2000 No.1

ample ID:	VX-101702-US-3	Acquisition Date:	10/18/02 6:01 AM
ial:	17	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	JACADS Ash Sample No. LAWIC153B, 1g + 5g NaSO4; Ultrasonic E xtraction, no HAE cleanup; Final Volume = 5mL + DEAET; 2.74u g/g VX Spike; Rtx5MS;S-TRAP;EM@1750V		
Data File:	c:\... \2002\october\101702#1\vx-101702-us-3-10-18-02.sms		

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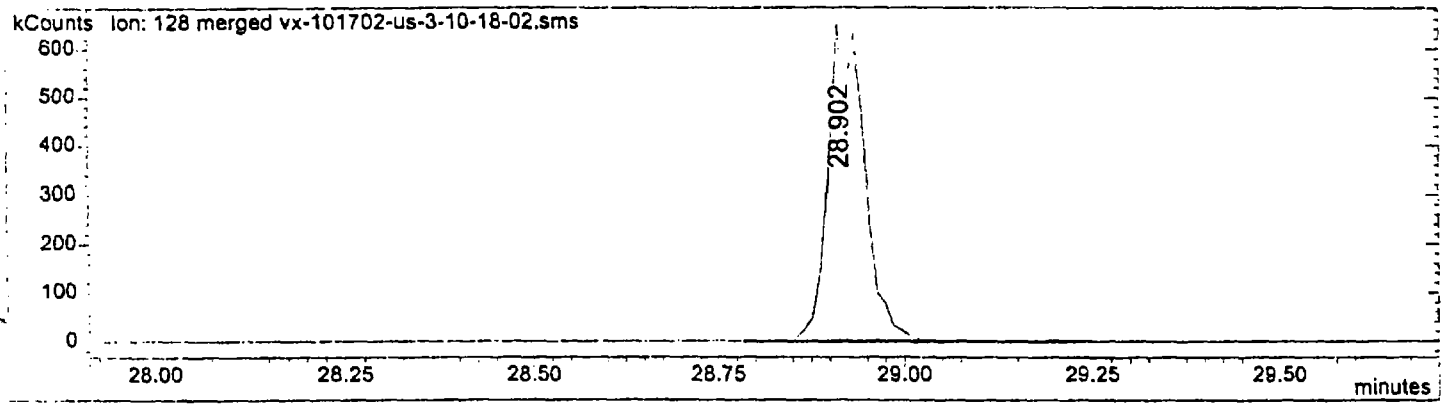
C31763 Bak p-9
10-17-02

Analyte: VX

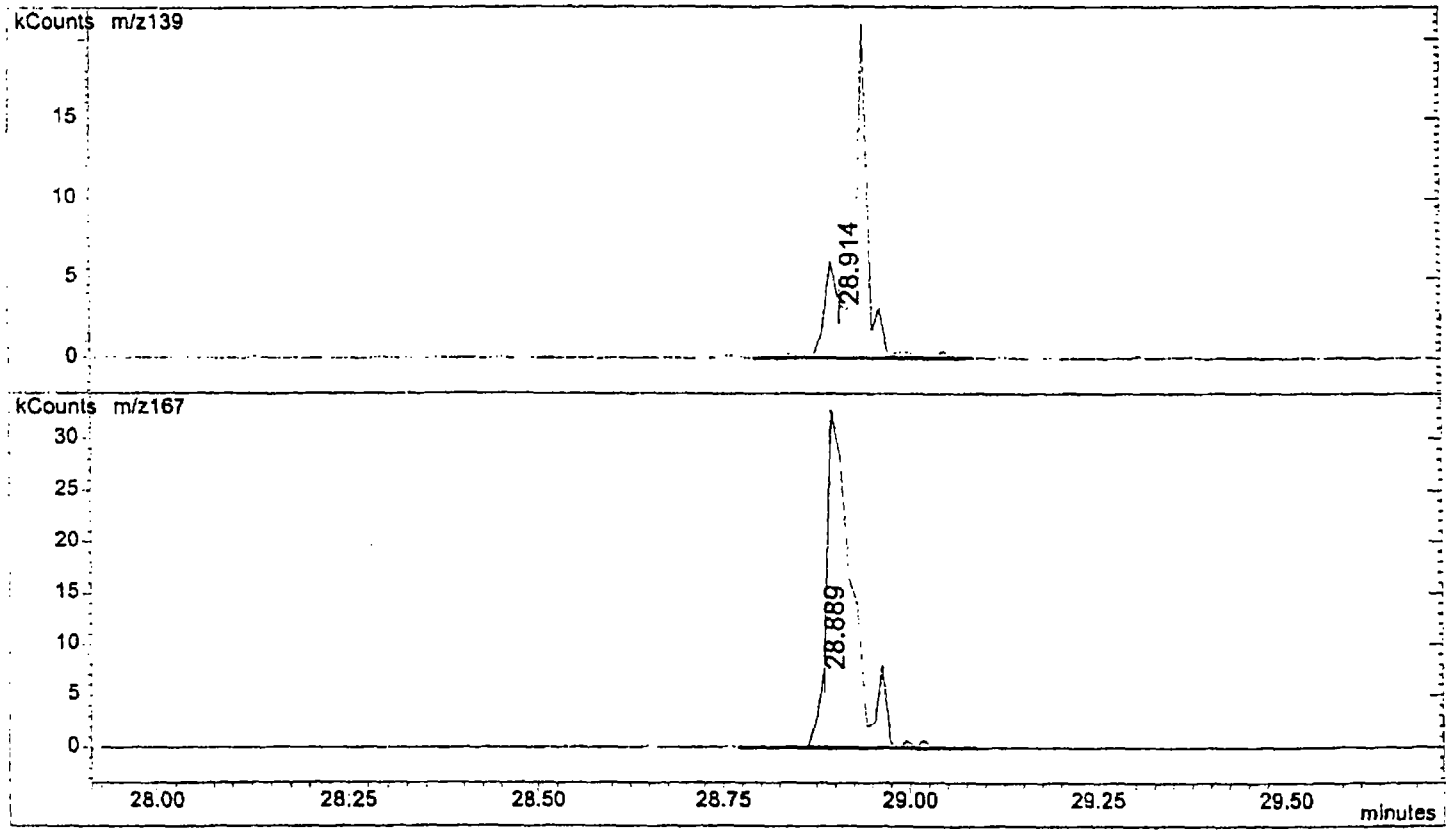
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.902 min.	28.812 min.	28.112 - 29.512 min.	2219005	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.914 min.	1.44%	2.0%	0.6% - 3.4%	31941	Pass
167	28.889 min.	3.54%	4.6%	1.4% - 7.8%	78471	Pass

Concentration: 1.407 ug/mL



Sample: VX



Segment: 4 Time(min): 23.999-32.980 Scans: 1422-2262 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:06:53

Ion Ratio Report for Target Compound #1 from vx-101702-us-4-10-18-02.sms

Saturn 2000 No.1

Sample ID:	VX-101702-US-4	Acquisition Date:	10/18/02 6:46 AM
Vial:	18	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	JACADS Ash Sample No. LAWIC153B, 1g + 5g NaSO4; Ultrasonic Extraction, no HAE cleanup; Final Volume = 5mL + DEAET; 2.74ug/g VX Spike; Rtx5MS;S-TRAP;EM@1750V		
Data File:	c:\... \2002\october\101702#1\vx-101702-us-4-10-18-02.sms		

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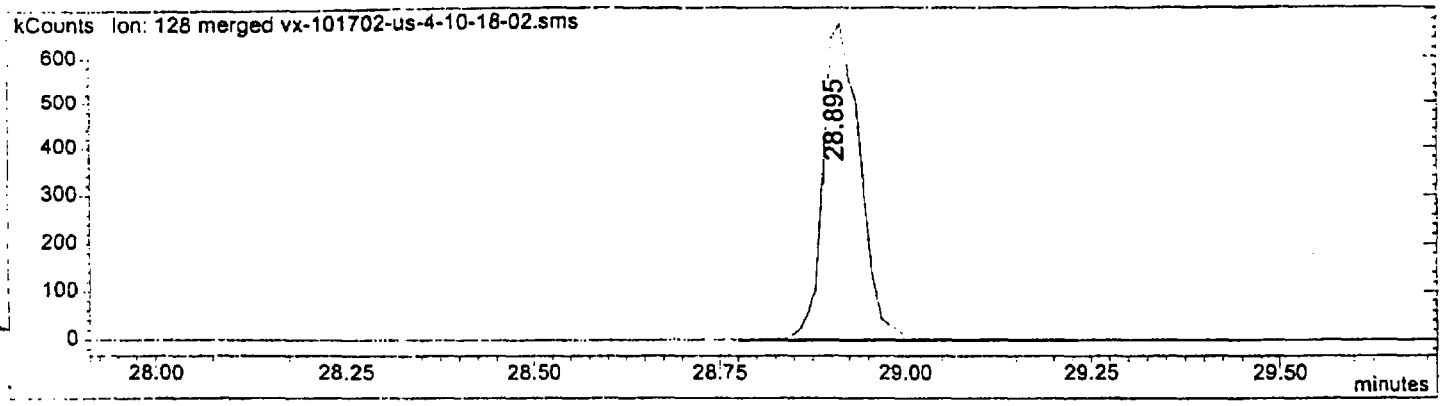
C31763 Bulk P-4
10-17-02

Analyte: VX

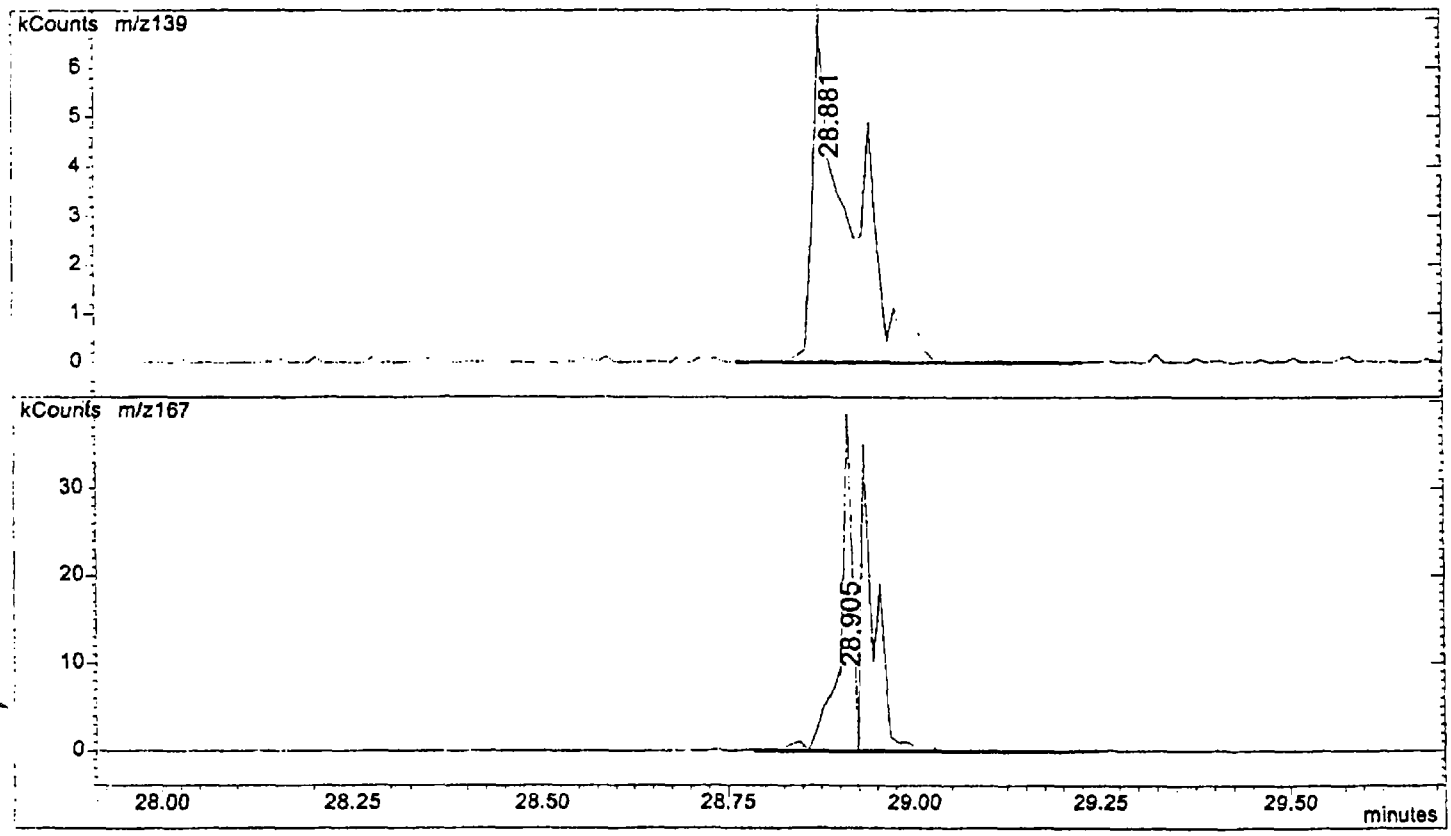
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.895 min.	28.812 min.	28.112 - 29.512 min.	2319001	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.881 min.	1.05%	2.0%	0.6% - 3.4%	24375	Pass
167	28.905 min.	3.86%	4.6%	1.4% - 7.8%	89469	Pass

Concentration: 1.460 ug/mL



Sample: VX



Segment: 4 Time(min): 23.997-32.983 Scans: 1422-2263 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:07:04

Ion Ratio Report for Target Compound #1 from ch2cl2 blank-10-18-02.sms

Saturn 2000 No.1

Sample ID:	CH2Cl2 Blank	Acquisition Date:	10/18/02 7:31 AM
ial:	0	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	CH2Cl2 Blank, Rtx-5MS:30Mx0.25mm ID, 1.0um; 100C,5min - 240 C @5C/m-280C @25C/m,5min;0.42VEE;PIT@200usec; 0.65scans/sec; noPP;m/z268.2;1750V; S-TRAP@170C		
Data File:	c:\... \data\2002\october\101702#1\ch2cl2 blank-10-18-02.sms		

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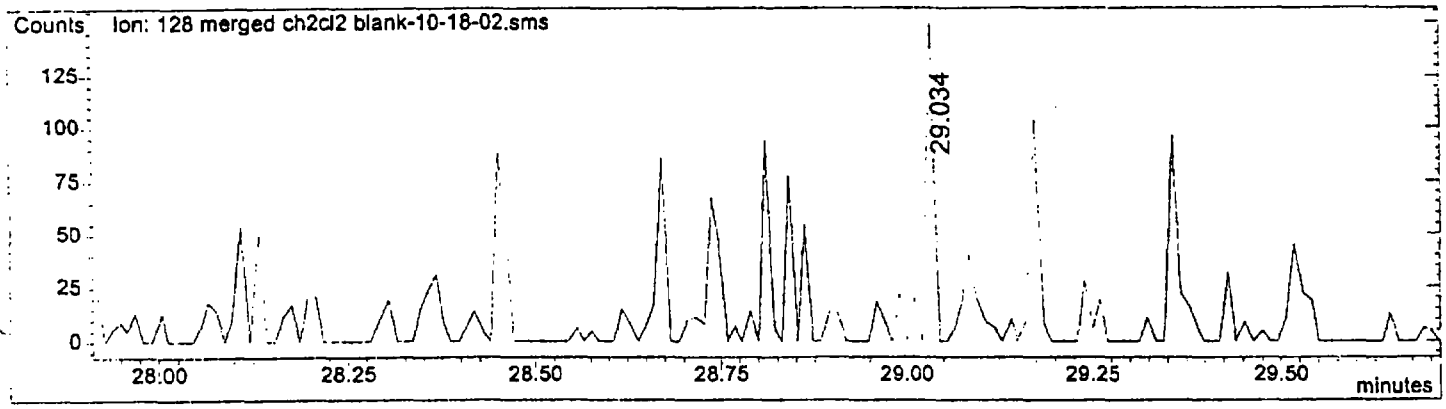
C31763-Book p-
10-17-02

Analyte: VX

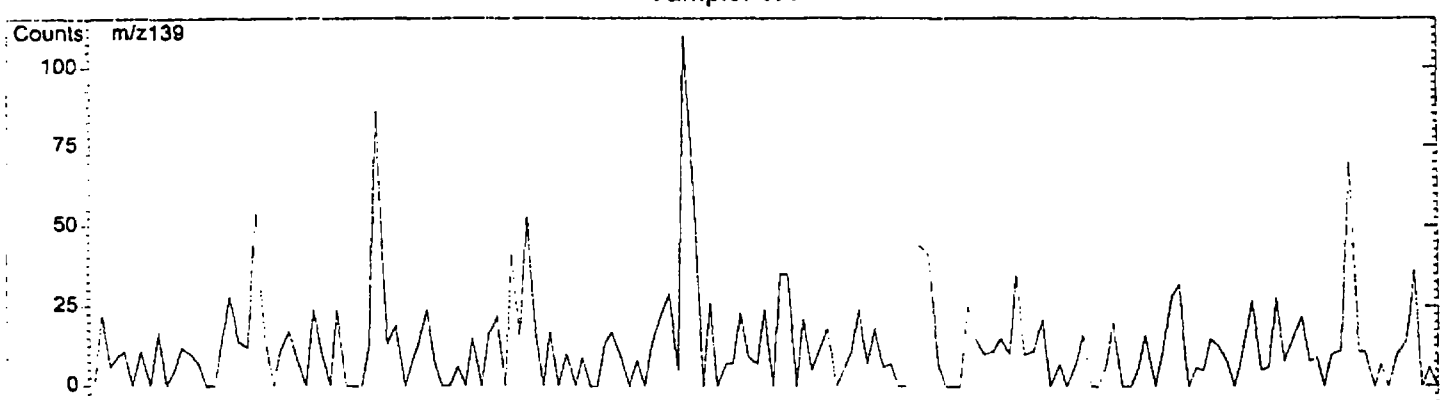
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	29.034 min.	28.812 min.	28.112 - 29.512 min.	0	Missing

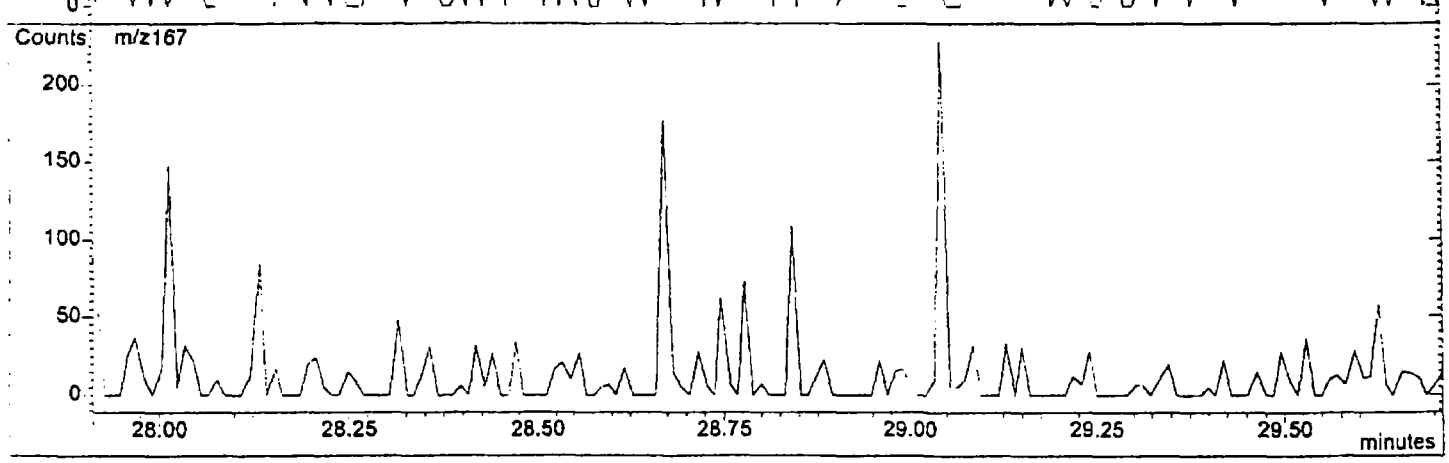
Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	0.000 min.	0.00%	2.0%	0.6% - 3.4%	0	Fail
167	0.000 min.	0.00%	4.6%	1.4% - 7.8%	0	Fail

Concentration: 0.000 ug/mL



Sample: VX





Segment: 4 Time(min): 23.987-32.988 Scans: 1421-2265 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:07:20

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/18/02 8:17 AM
al:	5	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \october\101702#1\xvx100302-3,0.46ug10-18-02002.sms		

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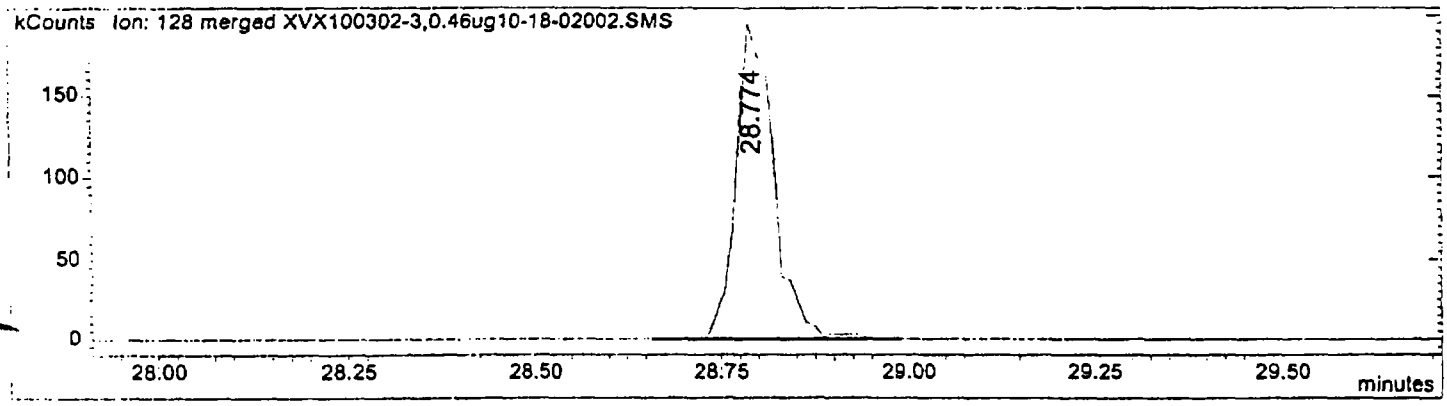
C31763 Book P.
10-17-02

Analyte: VX

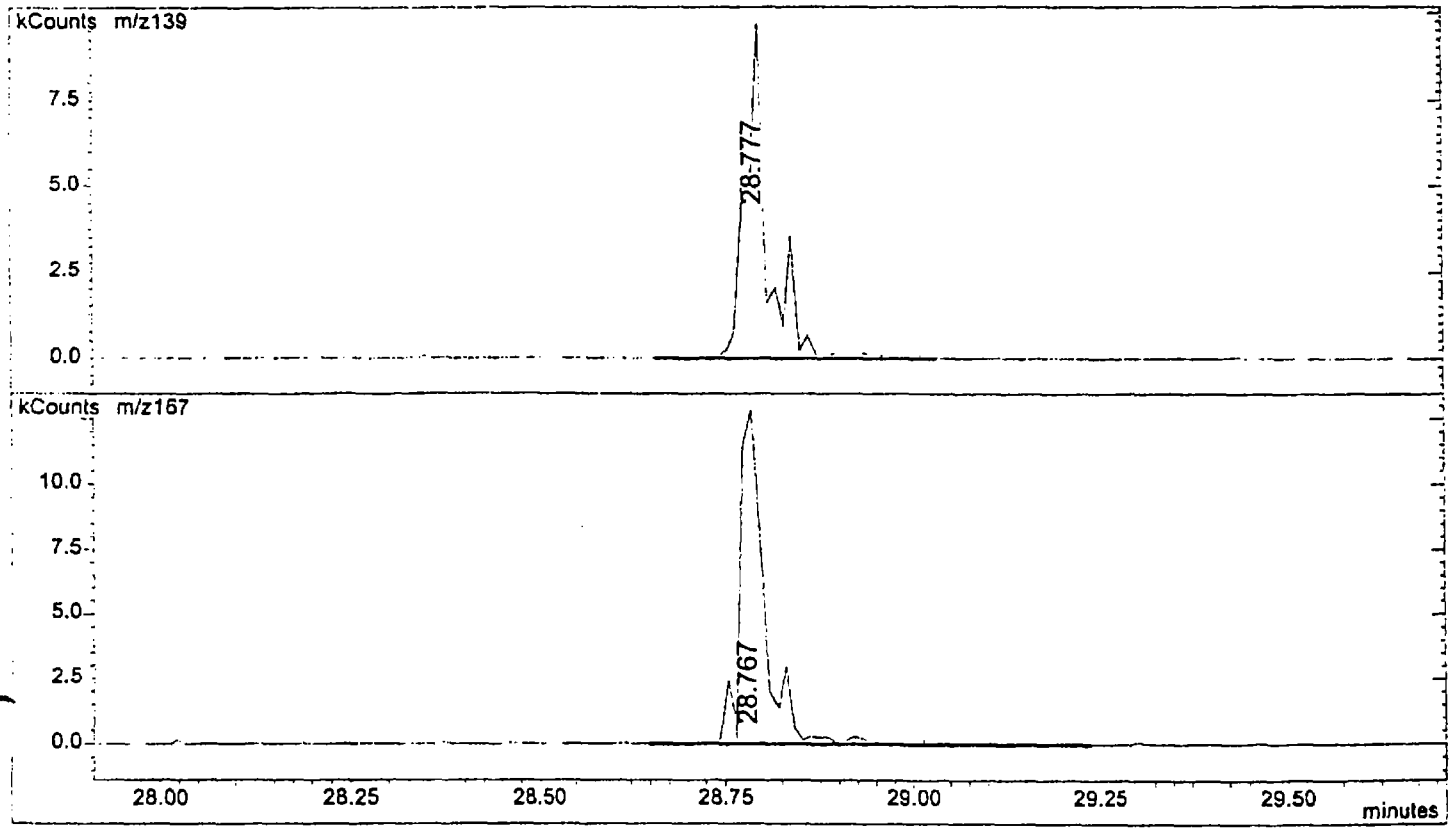
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.774 min.	28.812 min.	28.112 - 29.512 min.	685199	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.777 min.	2.84%	2.0%	0.6% - 3.4%	19493	Pass
167	28.767 min.	4.31%	4.6%	1.4% - 7.8%	29564	Pass

Concentration: 0.490 ug/mL



Sample: VX



Segment: 4 Time(min): 23.995-32.986 Scans: 1421-2266 Ionization: CI Auto Preparation: MS/MS Channels: 1

Print Date: 21 Oct 2002 19:07:32

Ion Ratio Report for Target Compound #1 from x:\... xvx100302-3,0.46ug10-1

Saturn 2000 No.1

Sample ID:	XVX100302-3,0.46ug	Acquisition Date:	10/18/02 9:02 AM
Vial:	5	Measurement Type:	Area
Method:	C:\saturnws\Methods\VX-2002\VX-calib-deaet-10-17-02.mth		
Inj. Sample Notes:	0.457ug/mLVX Std,Rtx-5MS:30Mx 0.25mm ID, 1.0um; 100C,5min - 240C @5C/m - 280C @25C/m,5min;0.42VEE;PIT@200usec;0.65scans/sec;noPP;m/z268.2;1750V;S-TRAP@170C;Sngl-GN		
Data File:	c:\... \october\101702#1\xvx100302-3,0.46ug10-18-02003.sms		

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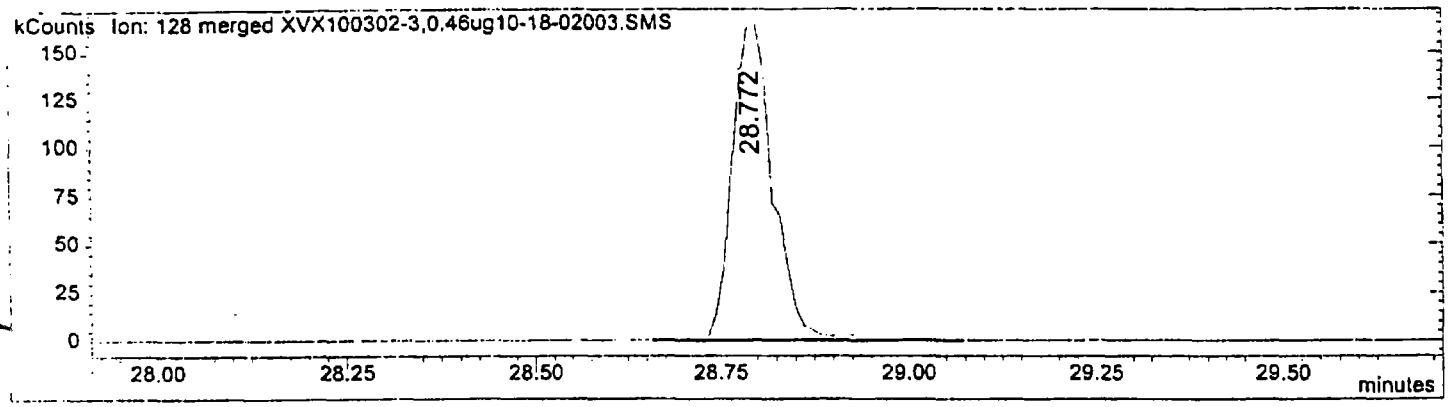
10-17-02

Analyte: VX

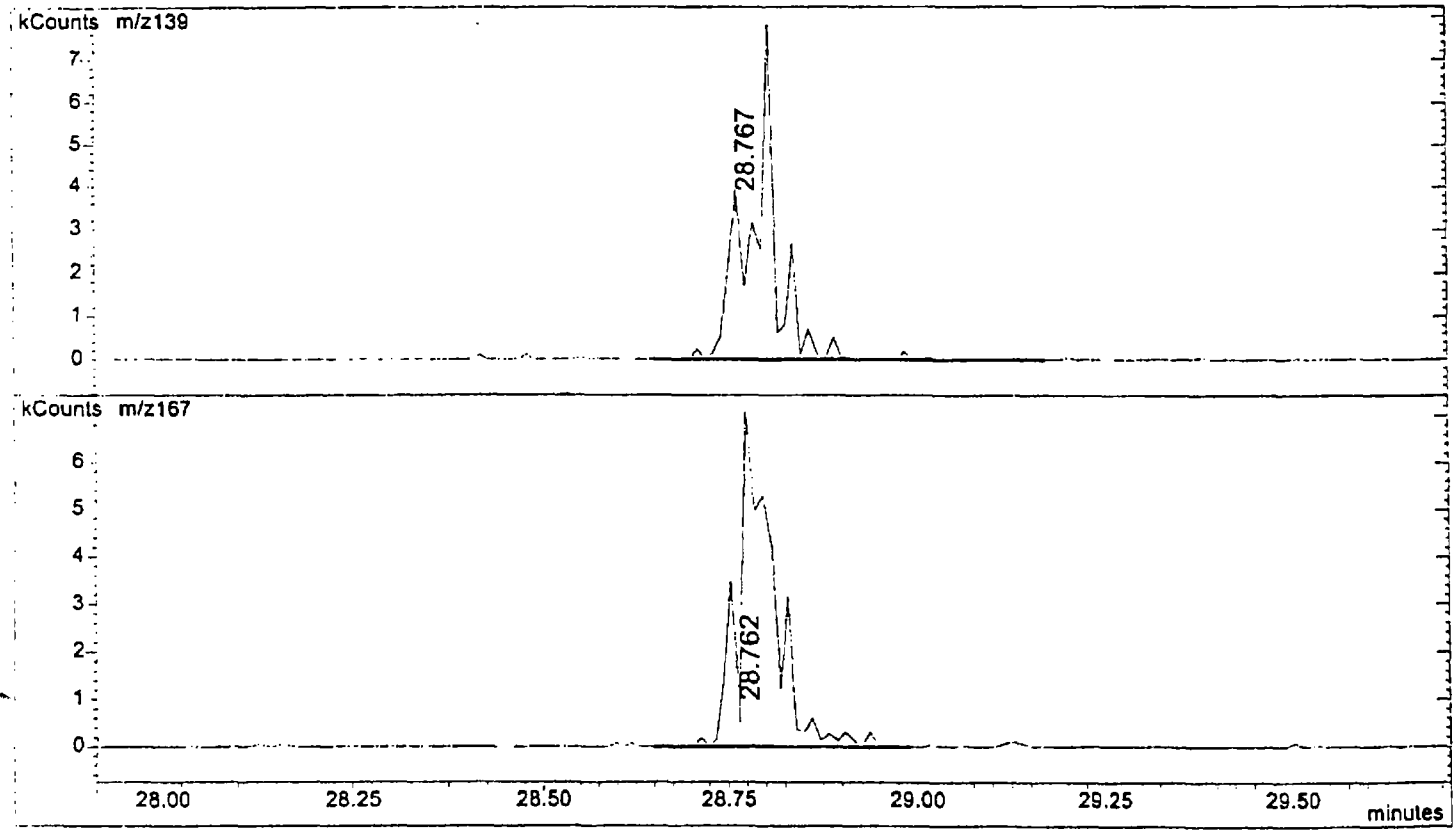
Ion	Actual RT	Specified RT	RT Time Range	Abundance	Status
128	28.772 min.	28.812 min.	28.112 - 29.512 min.	629319	Identified

Ion	Actual RT	Actual %	Specified %	% Range	Abundance	Status
139	28.767 min.	2.87%	2.0%	0.6% - 3.4%	18088	Pass
167	28.762 min.	3.64%	4.6%	1.4% - 7.8%	22902	Pass

Concentration: 0.452 ug/mL



Sample: VX



Segment: 4 Time(min): 23.988-32.984 Scans: 1423-2284 Ionization: CI Auto Preparation: MS/MS Channels: 1

Autotune Saturn 2000 #1; 10/14/02; MM/YA

17:15:24 Auto Tune: Started
17:15:24 Air/Water Check: Started
17:15:51 Air Check: Acceptable Level Found (28 Width: 0.6 m/z)
17:15:51 Water Check: Acceptable Level Found (19/18 Ratio: 3.1 %)
17:15:51 Air/Water Check: Completed - No Problems Found
17:15:51 RF Full Scale Adj: Started
17:16:01 RF Full Scale Adj: Centered on Mass 69 at 67.49 (Setting: 242)
17:16:10 RF Full Scale Adj: Centered on Mass 414 at 417.40 (Setting: 202)
17:16:15 RF Full Scale Adj: Centered on Mass 614 at 614.43 (Setting: 199)
17:16:15 RF Full Scale Adj: Setting is OK (Setting: 199, Mass: 614, Apex: 614.1)
17:16:15 RF Full Scale Adj: Completed
17:16:16 Multi-Point Mass Cal: Started
17:16:23 Multi-Point Mass Cal: Found Calibration Mass 28 at 27.60
17:16:30 Multi-Point Mass Cal: Found Calibration Mass 69 at 68.44
17:16:34 Multi-Point Mass Cal: Found Calibration Mass 131 at 130.40
17:16:46 Multi-Point Mass Cal: Found Calibration Mass 264 at 263.49
17:17:05 Multi-Point Mass Cal: Found Calibration Mass 414 at 413.75
17:17:25 Multi-Point Mass Cal: Found Calibration Mass 464 at 463.80
17:17:49 Multi-Point Mass Cal: Found Calibration Mass 502 at 501.76
17:18:16 Multi-Point Mass Cal: Found Calibration Mass 614 at 614.08
17:18:17 Multi-Point Mass Cal: Calibration is OK (Slope: 6.257, Std Dev: 0.055)
17:18:17 Multi-Point Mass Cal: Completed
17:18:17 Trap Function Calib: Started
17:18:21 Trap Function Calib: Calibrating at Mass 69
17:19:02 Trap Function Calib: Calibrating at Mass 131
17:20:11 Trap Function Calib: Mass 69 Calibrated (Freq: 260.100 kHz)
17:20:11 Trap Function Calib: Mass 131 Calibrated (Freq: 257.600 kHz)
17:20:11 Trap Function Calib: Completed
17:20:11 Auto Tune: Completed

Autotune Saturn 2000 #1; 10/15/02; MM/YA

10:03:15 Auto Tune: Started
10:03:15 Air/Water Check: Started
10:03:42 Air Check: Acceptable Level Found (28 Width: 0.6 m/z)
10:03:42 Water Check: Acceptable Level Found (19/18 Ratio: 4.6 %)
10:03:43 Air/Water Check: Completed - No Problems Found
10:03:43 RF Full Scale Adj: Started
10:03:53 RF Full Scale Adj: Centered on Mass 69 at 67.50 (Setting: 241)
10:04:02 RF Full Scale Adj: Centered on Mass 414 at 417.35 (Setting: 201)
10:04:07 RF Full Scale Adj: Centered on Mass 614 at 614.37 (Setting: 198)
10:04:07 RF Full Scale Adj: Setting is OK (Setting: 198, Mass: 614, Apex: 614.0)
10:04:07 RF Full Scale Adj: Completed
10:04:08 Multi-Point Mass Cal: Started
10:04:14 Multi-Point Mass Cal: Found Calibration Mass 28 at 27.61
10:04:21 Multi-Point Mass Cal: Found Calibration Mass 69 at 68.45
10:04:26 Multi-Point Mass Cal: Found Calibration Mass 131 at 130.41
10:04:38 Multi-Point Mass Cal: Found Calibration Mass 264 at 263.47
10:04:56 Multi-Point Mass Cal: Found Calibration Mass 414 at 413.69
10:05:17 Multi-Point Mass Cal: Found Calibration Mass 464 at 463.75
10:05:40 Multi-Point Mass Cal: Found Calibration Mass 502 at 501.72
10:06:08 Multi-Point Mass Cal: Found Calibration Mass 614 at 614.01
10:06:09 Multi-Point Mass Cal: Calibration is OK (Slope: 6.257, Std Dev: 0.054)
10:06:09 Multi-Point Mass Cal: Completed
10:06:09 Trap Function Calib: Started
10:06:14 Trap Function Calib: Calibrating at Mass 69
10:06:56 Trap Function Calib: Calibrating at Mass 131
10:08:05 Trap Function Calib: Mass 69 Calibrated (Freq: 260.100 kHz)
10:08:05 Trap Function Calib: Mass 131 Calibrated (Freq: 257.600 kHz)
10:08:05 Trap Function Calib: Completed
10:08:06 Auto Tune: Completed

Autotune Saturn 2000 #1; 10/16/02; MM/YA

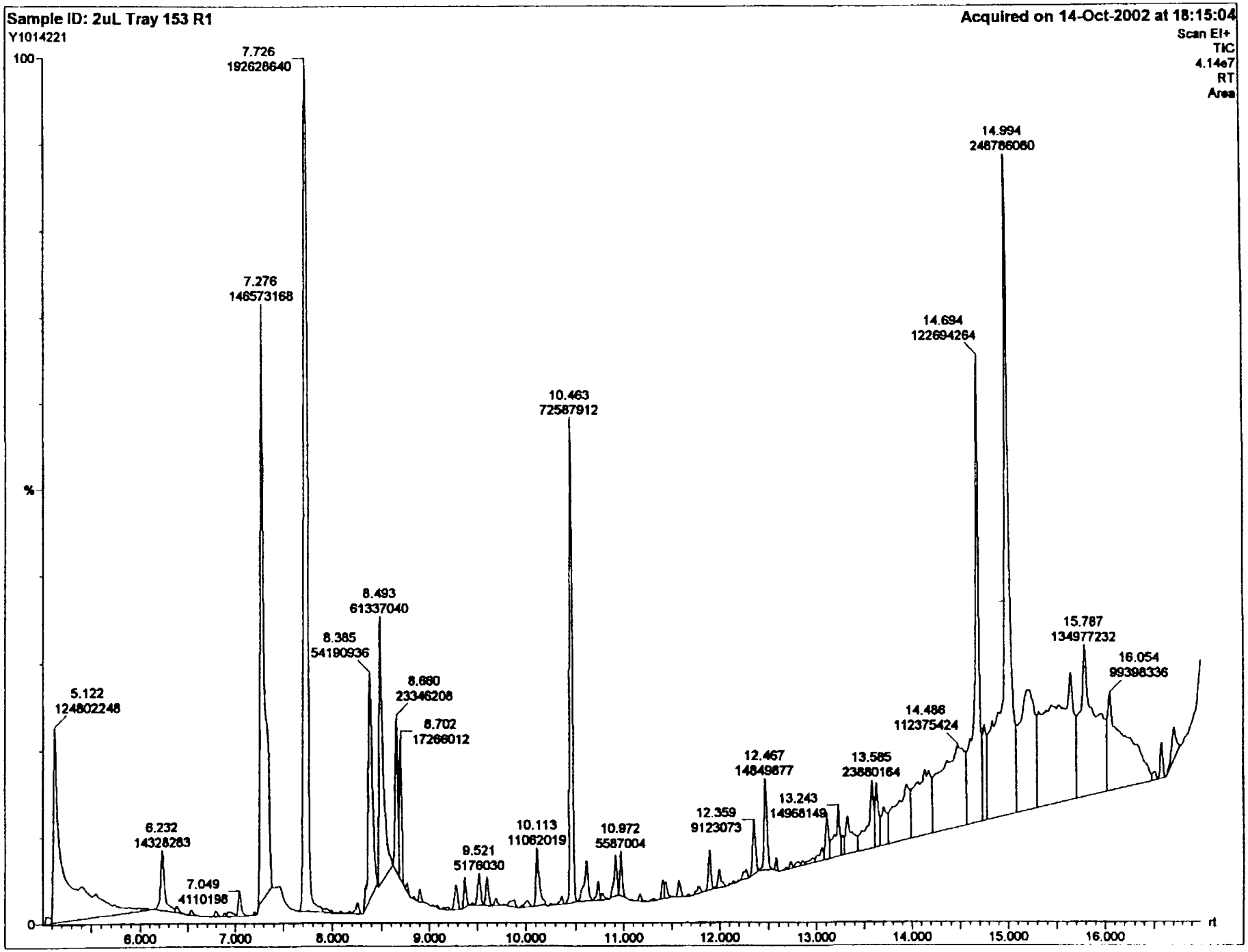
10:03:15 Auto Tune: Started
10:03:15 Air/Water Check: Started
10:03:42 Air Check: Acceptable Level Found (28 Width: 0.6 m/z)
10:03:42 Water Check: Acceptable Level Found (19/18 Ratio: 4.6 %)
10:03:43 Air/Water Check: Completed - No Problems Found
10:03:43 RF Full Scale Adj: Started
10:03:53 RF Full Scale Adj: Centered on Mass 69 at 67.50 (Setting: 241)
10:04:02 RF Full Scale Adj: Centered on Mass 414 at 417.35 (Setting: 201)
10:04:07 RF Full Scale Adj: Centered on Mass 614 at 614.37 (Setting: 198)
10:04:07 RF Full Scale Adj: Setting is OK (Setting: 198, Mass: 614, Apex: 614.0)
10:04:07 RF Full Scale Adj: Completed
10:04:08 Multi-Point Mass Cal: Started
10:04:14 Multi-Point Mass Cal: Found Calibration Mass 28 at 27.61
10:04:21 Multi-Point Mass Cal: Found Calibration Mass 69 at 68.45
10:04:26 Multi-Point Mass Cal: Found Calibration Mass 131 at 130.41
10:04:38 Multi-Point Mass Cal: Found Calibration Mass 264 at 263.47
10:04:56 Multi-Point Mass Cal: Found Calibration Mass 414 at 413.69
10:05:17 Multi-Point Mass Cal: Found Calibration Mass 464 at 463.75
10:05:40 Multi-Point Mass Cal: Found Calibration Mass 502 at 501.72
10:06:08 Multi-Point Mass Cal: Found Calibration Mass 614 at 614.01
10:06:09 Multi-Point Mass Cal: Calibration is OK (Slope: 6.257, Std Dev: 0.054)
10:06:09 Multi-Point Mass Cal: Completed
10:06:09 Trap Function Calib: Started
10:06:14 Trap Function Calib: Calibrating at Mass 69
10:06:56 Trap Function Calib: Calibrating at Mass 131
10:08:05 Trap Function Calib: Mass 69 Calibrated (Freq: 260.100 kHz)
10:08:05 Trap Function Calib: Mass 131 Calibrated (Freq: 257.600 kHz)
10:08:05 Trap Function Calib: Completed
10:08:06 Auto Tune: Completed

Autotune Saturn 2000 #1; 10/17/02; MM/YA

13:27:35 Auto Tune: Started
13:27:36 Air/Water Check: Started
13:28:03 Air Check: Acceptable Level Found (28 Width: 0.6 m/z)
13:28:04 Water Check: Acceptable Level Found (19/18 Ratio: 6.5 %)
13:28:04 Air/Water Check: Completed - No Problems Found
13:28:04 RF Full Scale Adj: Started
13:28:13 RF Full Scale Adj: Centered on Mass 69 at 67.50 (Setting: 240)
13:28:20 RF Full Scale Adj: Centered on Mass 414 at 417.26 (Setting: 201)
13:28:27 RF Full Scale Adj: Centered on Mass 614 at 614.33 (Setting: 198)
13:28:27 RF Full Scale Adj: Setting is OK (Setting: 198, Mass: 614, Apex: 614.0)
13:28:27 RF Full Scale Adj: Completed
13:28:28 Multi-Point Mass Cal: Started
13:28:35 Multi-Point Mass Cal: Found Calibration Mass 28 at 27.61
13:28:42 Multi-Point Mass Cal: Found Calibration Mass 69 at 68.45
13:28:46 Multi-Point Mass Cal: Found Calibration Mass 131 at 130.40
13:28:58 Multi-Point Mass Cal: Found Calibration Mass 264 at 263.47
13:29:16 Multi-Point Mass Cal: Found Calibration Mass 414 at 413.68
13:29:37 Multi-Point Mass Cal: Found Calibration Mass 464 at 463.74
13:30:00 Multi-Point Mass Cal: Found Calibration Mass 502 at 501.68
13:30:28 Multi-Point Mass Cal: Found Calibration Mass 614 at 613.98
13:30:29 Multi-Point Mass Cal: Calibration is OK (Slope: 6.257, Std Dev: 0.054)
13:30:29 Multi-Point Mass Cal: Completed
13:30:29 Trap Function Calib: Started
13:30:33 Trap Function Calib: Calibrating at Mass 69
13:31:14 Trap Function Calib: Calibrating at Mass 131
13:32:24 Trap Function Calib: Mass 69 Calibrated (Freq: 260.100 kHz)
13:32:24 Trap Function Calib: Mass 131 Calibrated (Freq: 257.600 kHz)
13:32:24 Trap Function Calib: Completed
13:32:25 Auto Tune: Completed

APPENDIX E

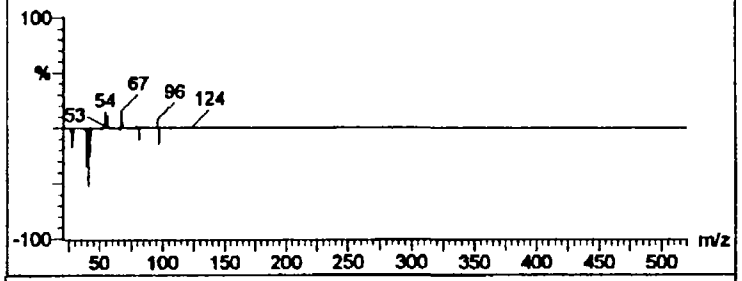
TENTATIVELY IDENTIFIED COMPOUNDS


Sample ID: 2uL Tray 153 R1
Y1014221
Acquired on 14-Oct-2002 at 18:15:04
Scan EI+
TIC
4.14e7
RT
Area
100
%
0
7.726
192628640
7.276
146573168
14.994
248786060
14.694
122694264
10.463
72587912
8.493
61337040
8.385
54190936
15.787
134977232
16.054
99398336
5.122
124802248
8.660
23346208
8.702
17268012
14.486
112375424
12.467
14849877
13.585
23880164
13.243
14968149
12.359
9123073
6.232
14328283
10.113
11062019
10.972
5587004
9.521
5176030
7.049
4110198
6.000
7.000
8.000
9.000
10.000
11.000
12.000
13.000
14.000
15.000
16.000
rt

Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

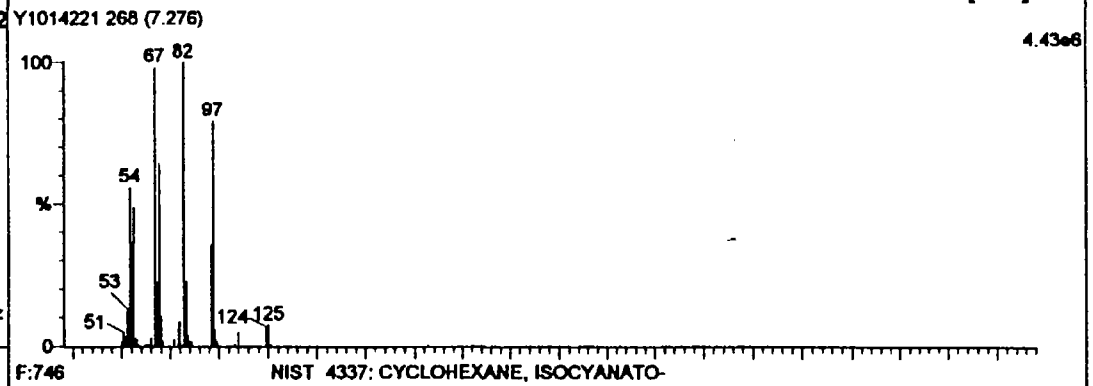
Hit	Compound Name	FOR	rev
1	CYCLOHEXANE, ISOCYANATO-	746	752
2	CYCLOHEPTANE, CHLORO-	632	790
3	5-BUTYL-2-METHYLPYRROLINE	576	583
4	1,1'-BICYCLOHEXYL, 2-METHYL-, CIS-	566	585
5	1,1'-BICYCLOHEXYL, 2-METHYL-, TRANS-	562	581
6	CYCLOHEXANE, 1-(CYCLOHEXYLMETHYL)-4-METHYL-, CIS-	561	578
7	1,1'-BICYCLOHEPTYL	521	541
8	1-(1-CYANOCYCLOHEXYL)ETHYLAMINE	517	637
9	HEXANE, 1,6-DICYCLOHEXYL-	511	528
10	2H-CYCLOPENTACYCLOOCTEN-2-ONE, DECAHYDRO-3A-METHYL-, TRANS-	511	522
11	CYCLOHEXANE, 1,1'-(1,5-PENTANEDIYL)BIS-	510	526
12	CYCLOHEPTANEMETHANOL	506	534
13	CYCLOHEXANE, 1,1'-(1,3-PROPANEDIYL)BIS-	498	516
14	9-BORABICYCLO 3.3.1 NONANE, 9-HYDROXY-	490	509
15	PENTYLENETETRAZOLE(METRAZOL)	483	637
16	CYCLOHEXANEHEXANOL	480	500
17	CYCLOHEXANE, 1-ETHYL-4-METHYL-, CIS-	468	496
18	CYCLOHEXANONE, HYDRAZONE	460	487
19	8-NONENOIC ACID	457	471
20	1-(1-CYANOCYCLOPENTYL)ETHYLAMINE	420	659

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 746, Reverse Fit: 752**
Y1014221 268 (7.276) Cm (268-4337)

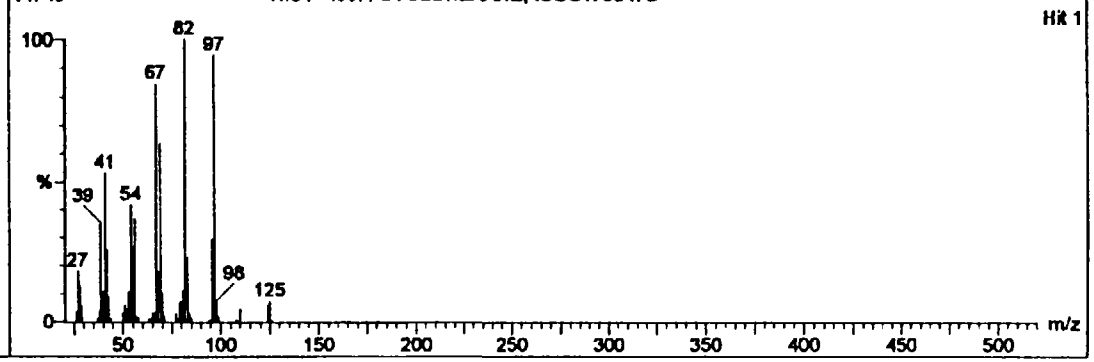


Compound Name: CYCLOHEXANE, ISOCYANATO-
Synonym:
Molecular Weight: 125
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 752
Y1014221 268 (7.276)



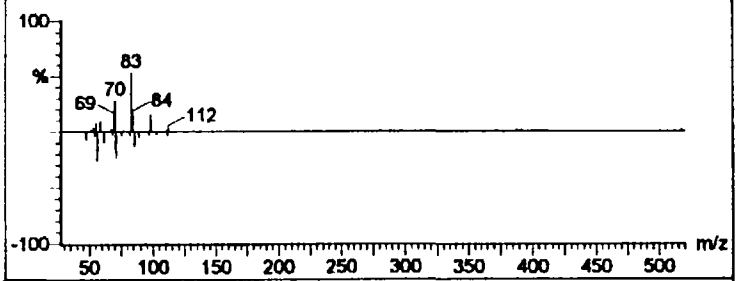
F:746 NIST 4337: CYCLOHEXANE, ISOCYANATO- Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

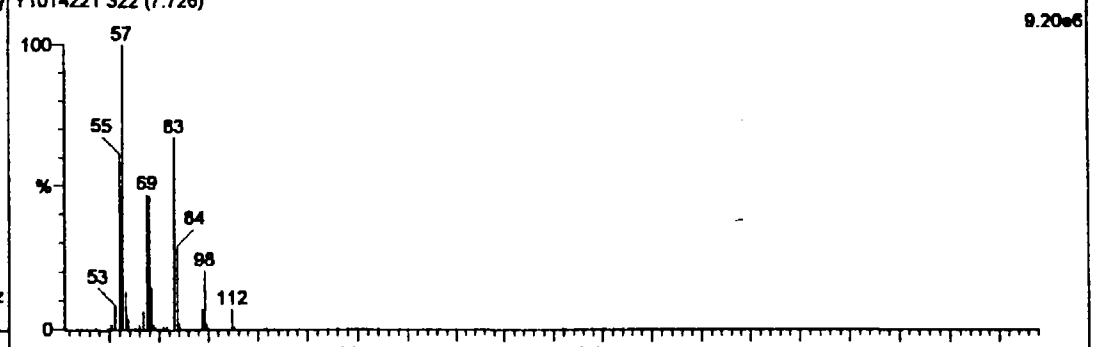
Hit	Compound Name	FOR	rev
1	DODECANE, 1-FLUORO-	700	707
2	HEPTANE, 1,1'-OXYBIS-	657	675
3	CYCLOPROPANE, 1-(2-METHYLBUTYL)-1-(1-METHYLPROPYL)-	645	658
4	SILACYCLOPENT-3-ENE, 3-METHYL-	599	621
5	CYCLOHEXANE, 1,5-DIETHYL-2,3-DIMETHYL-	589	603
6	CYCLOPROPANE, 1-METHYL-1-(1-METHYLETHYL)-2-NONYL-	588	601
7	4-UNDECENE, 3-METHYL-, (Z)-	588	599
8	5-ETHYL-1-NONENE	587	600
9	2-UNDECENE, 9-METHYL-, (E)-	575	586
10	4-OCTENE, 2,3,6,7-TETRAMETHYL-	572	640
11	CYCLOPROPANE, 1-BUTYL-1-METHYL-2-PROPYL-	570	582
12	5,8-DIETHYL-6-DODECANOL	564	574
13	TRIDECANOL	562	589
14	1-UNDECENE, 9-METHYL-	559	568
15	5-UNDECENE, 9-METHYL-, (Z)-	557	570
16	4-UNDECENE, 9-METHYL-, (Z)-	553	564
17	CYCLOPROPANE, 1-(1,2-DIMETHYLPROPYL)-1-METHYL-2-NONYL-	550	581
18	TETRACONTANE, 3,5,24-TRIMETHYL-	542	555
19	DECANEDIOIC ACID, DIDECYL ESTER	539	567
20	5-EICOSENE, (E)-	539	553

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST — **Forward Fit: 700, Reverse Fit: 707**
Y1014221 322 (7.726) Cm (322-19931)

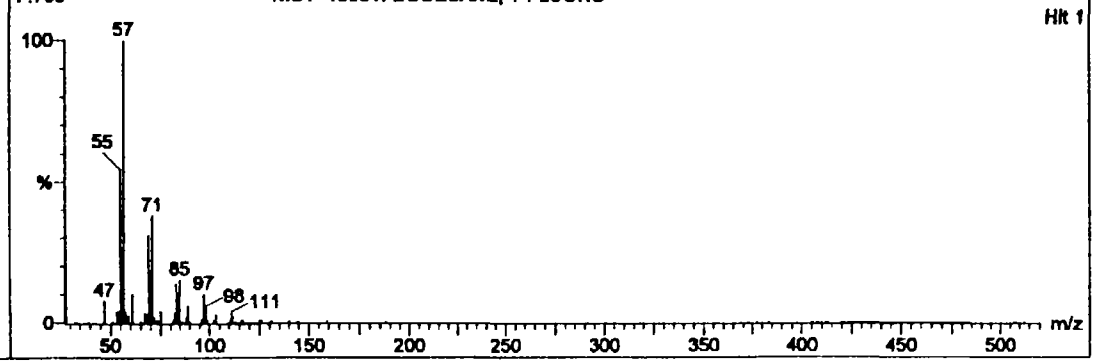


Compound Name: DODECANE, 1-FLUORO-
Synonym:
Molecular Weight: 188
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 707
Y1014221 322 (7.726)



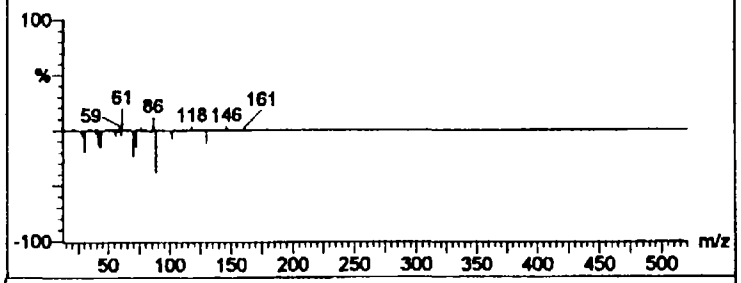
F:700 — NIST 19931: DODECANE, 1-FLUORO- — Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

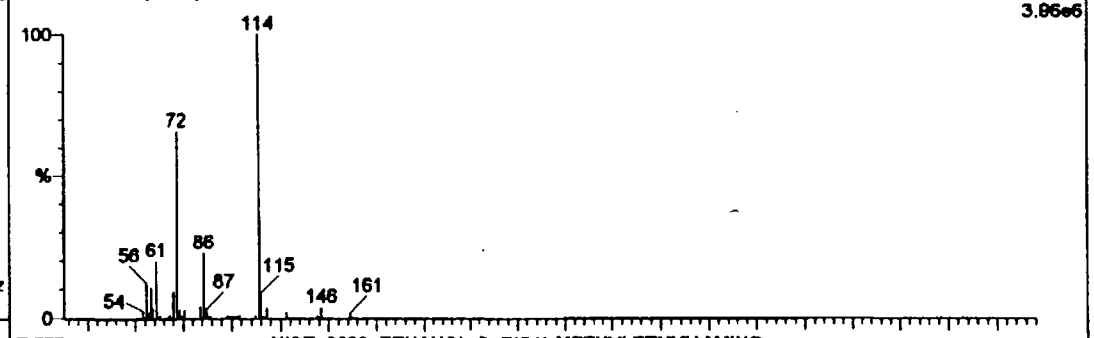
Hit	Compound Name	FOR	rev
1	ETHANOL, 2- BIS(1-METHYLETHYL)AMINO -	555	640
2	OXAZOLIDINE, 3-ETHYL-2,2-DIMETHYL-	511	612
3	2-IMIDAZOLIDINONE, 1,3-DIMETHYL-	501	586
4	PENTANAMIDE, 2-(DIMETHYLAMINO)-4-METHYL-N- 2-METHYL-1- 3,3A,11,12,13,14,15,15A-OCTAHYDRO-12,15-DIOX	468	600
5	METHIMAZOLE REF-PMS-P358	467	638
6	3-(DIISOPROPYLAMINO)-1,2-PROPANEDIOL	456	492
7	PROPANIDID REF-PMS-P503	455	606
8	N-ETHYLAMPHETAMINE ACETATE	455	649
9	2,4-IMIDAZOLIDINEDIONE, 3-METHYL-	448	791
10	2-PROPANAMINE, N-ETHYL-N-(1-METHYLETHYL)-	439	528
11	3-DIPROPYLAMINO-1,2-PROPANEDIOL	417	485
12	N-BUTYL-TERT-BUTYLAMINE	417	520
13	1,2,3-CYCLOHEXANETRIOL, (1.ALPHA.,2.ALPHA.,3.BETA.)-(.+/-.)-	406	445
14	SILACYCLOOCTAN-5-ONE, 1,1-DIMETHYL-	405	435
15	OXETHAZAINE	402	536

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 555, Reverse Fit: 640**
Y1014221 401 (8.385) Cm (401-8639)

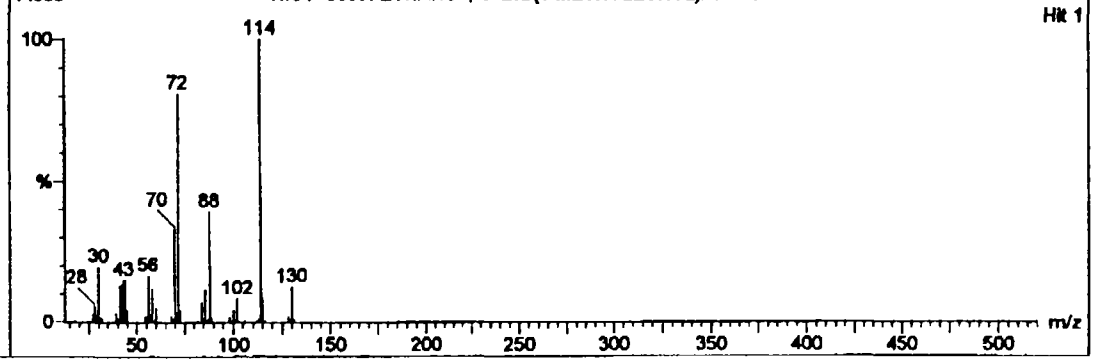


Compound Name: ETHANOL, 2- BIS(1-METHYLETHYL)AMINO -
Synonym:
Molecular Weight: 145
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 640
Y1014221 401 (8.385)



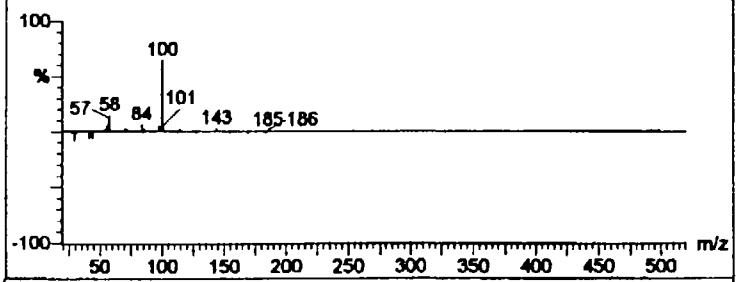
F:555 NIST 8639: ETHANOL, 2- BIS(1-METHYLETHYL)AMINO - Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

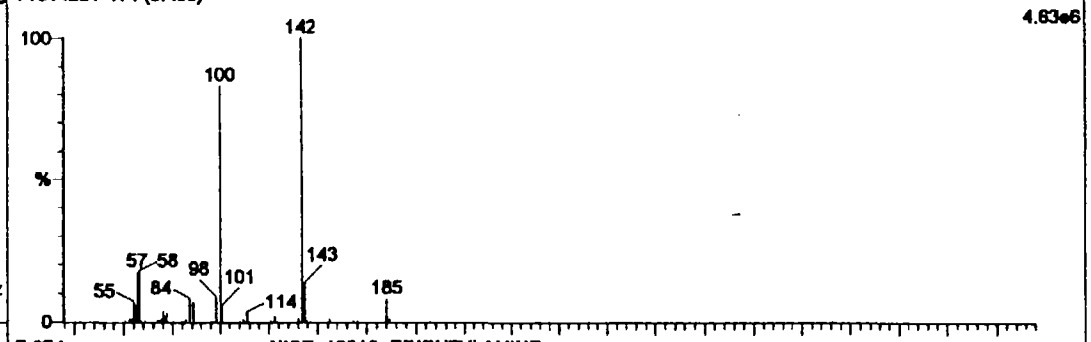
Hit	Compound Name	FOR	rev
1	TETRA-N-BUTYLAMMONIUM BROMIDE	695	717
2	TRIBUTYLAMINE	674	695
3	TETRA-N-BUTYLAMMONIUM IODIDE	631	819
4	TETRABUTYLAMMONIUM FLUORIDE	621	636
5	1,3-PROPANEDIAMINE, N,N-DIBUTYL-	621	652
6	ACETAMIDE, N,N-BIS(1-METHYLPROPYL)-	601	727
7	BUTALAMINE REF-PMS-P61	593	690
8	TETRA-N-BUTYLAMMONIUM CHLORIDE	576	706
9	ETHANOL, 2-(DIBUTYLAMINO)-	570	593
10	2-PROPANOL, 1-(DIBUTYLAMINO)-	566	592
11	2-DIBUTYLAMINOETHYLAMINE	531	562
12	BENZYLTRIBUTYLAMMONIUM BROMIDE	445	464
13	N,N-2-TRIETHYLHEXAMIDE	442	464
14	2(1H)-PYRIMIDINONE, 3,4-DIHYDRO-1-METHYL-4-THIOXO-	430	464
15	LEUCINOCAINE REF-PMS-P327	430	558
16	METHYL 4,6-DI-O-ACETYL-2,3-DIACETAMIDO-2,3-DIDEOXY-.BETA.-D-GLUCOPYRANOSIDE	419	508

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 674, Reverse Fit: 695**
Y1014221 414 (8.493) Cm (414-19216)

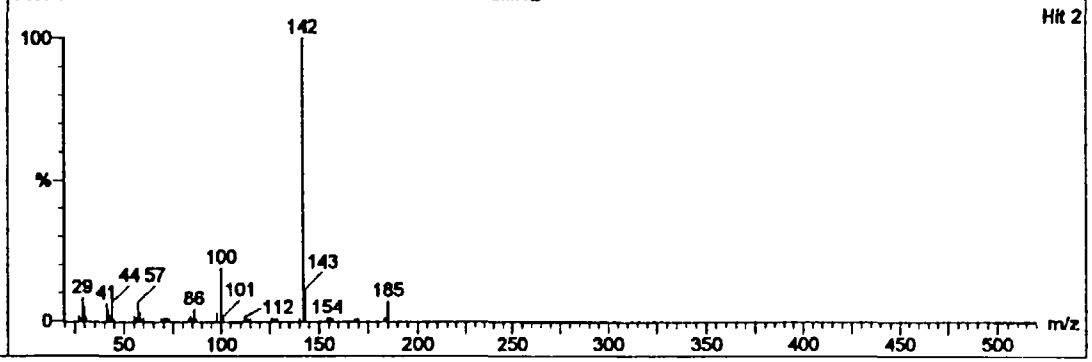


Compound Name: TRIBUTYLAMINE
Synonym:
Molecular Weight: 185
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 695
Y1014221 414 (8.493)



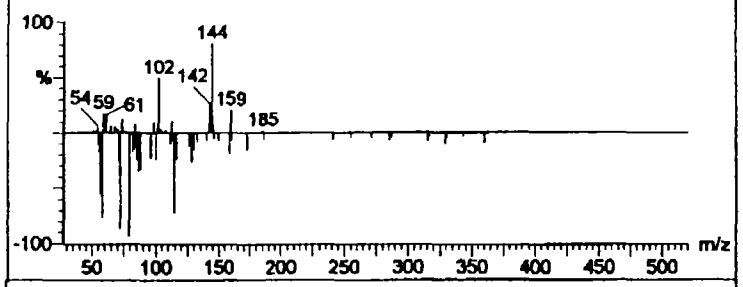
F:674 NIST 19216: TRIBUTYLAMINE — Hit 2



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

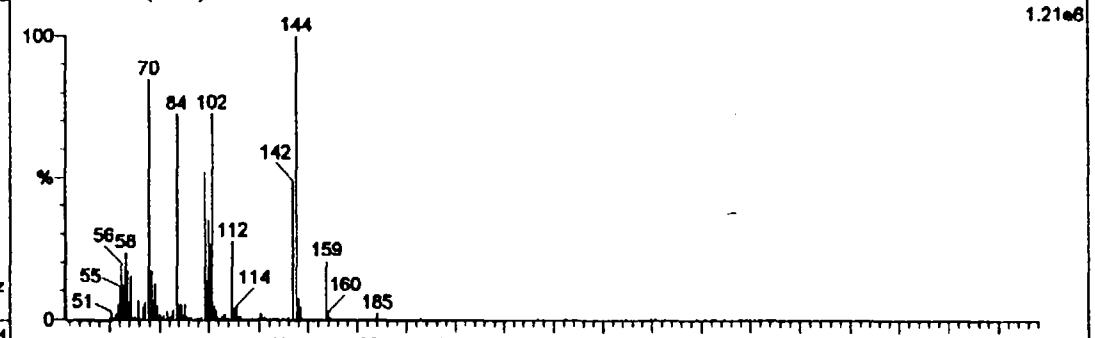
Hit	Compound Name	FOR	rev
1	5,8,15,18,23-PENTAOXA-1,12-DIAZABICYCLO 10.8.5 -PENTACOZANE	471	563
2	4,7,14,20-TETRAOXA-1,10-DIAZABICYCLO 8.7.5 DOCOZANE	415	508
3	5,8,15,20-TETHAOXA-1,12 DIAZABICYCLO 10,5,5 -DOCOZANE	405	516
4	4,7,13,16,22-PENTAOXA-1,10-DIAZABICYCLO 8.8.7 -PENTACOSANE	403	517

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 471, Reverse Fit: 563**
Y1014221 434 (8.660) Cm (434-50332)



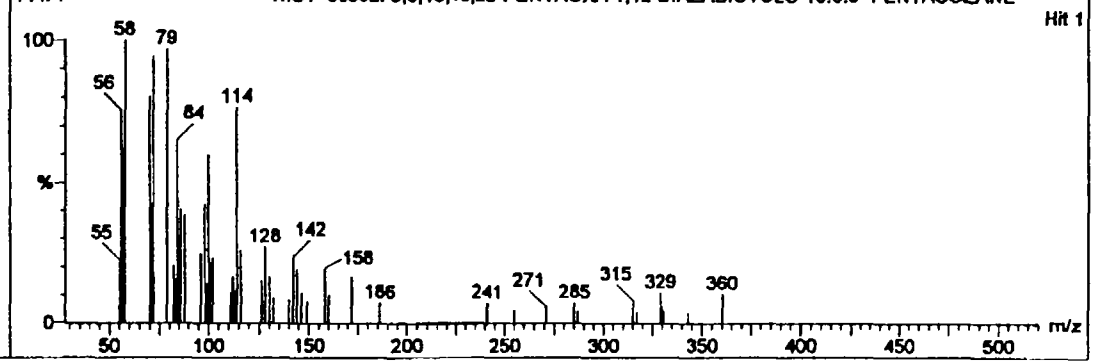
Compound Name: 5,8,15,18,23-PENTAOXA-1,12-DIAZABICYCLO 1
Synonym:
Molecular Weight: 360
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 563
Y1014221 434 (8.660)



F:471 NIST 50332: 5,8,15,18,23-PENTAOXA-1,12-DIAZABICYCLO 10.8.5 -PENTACOZANE

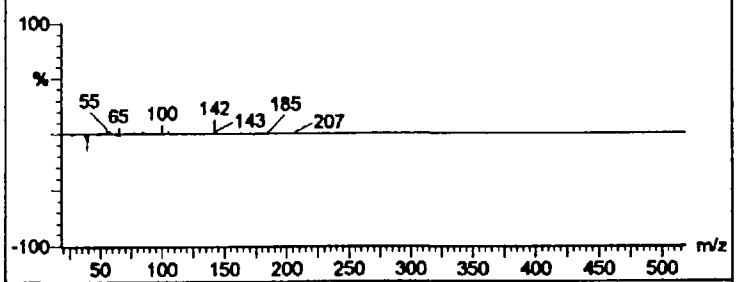
Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

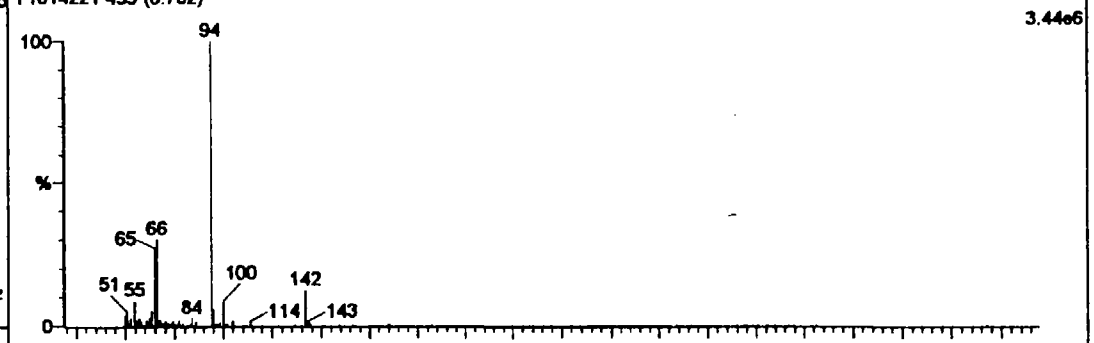
Hit	Compound Name	FOR	rev
1	PHOSPHONIC ACID, (P-HYDROXYPHENYL)-	662	805
2	CARBAMIC ACID, BUTYLMETHYL-, PHENYL ESTER	642	828
3	BENZENESULFONIC ACID, 4-HYDROXY-	631	763
4	PHENOL	583	705
5	BENZENE, (2-METHYLPROPOXY)-	549	671
6	CARBONIC ACID, BUTYL PHENYL ESTER	527	697
7	BENZENE, (1,1-DIMETHYLETHOXY)-	519	664
8	BENZENE, ETHOXY-	510	605
9	BUTANOIC ACID, 4-PHENOXY-	495	619
10	BENZENE, (2-BUTENYLOXY)-	478	585
11	3-PHENOXYPROPIONIC ACID	473	600
12	ACETIC ACID, PHENYL ESTER	463	562
13	BENZENE, (4-CHLOROBUTOXY)-	462	582
14	PROPANOIC ACID, 2,2-DIMETHYL-, PHENYL ESTER	462	590
15	PROPANOIC ACID, PHENYL ESTER	458	620
16	CARBAMIC ACID, PHENYL-, PHENYL ESTER	442	578
17	CARBAMIC ACID, METHYL-, PHENYL ESTER	442	540
18	CARBAMIC ACID, PHENYL ESTER	437	546
19	BENZENE, (2-CHLOROETHOXY)-	420	517

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 662, Reverse Fit: 805**
Y1014221 439 (8.702) Cm (439-16041)

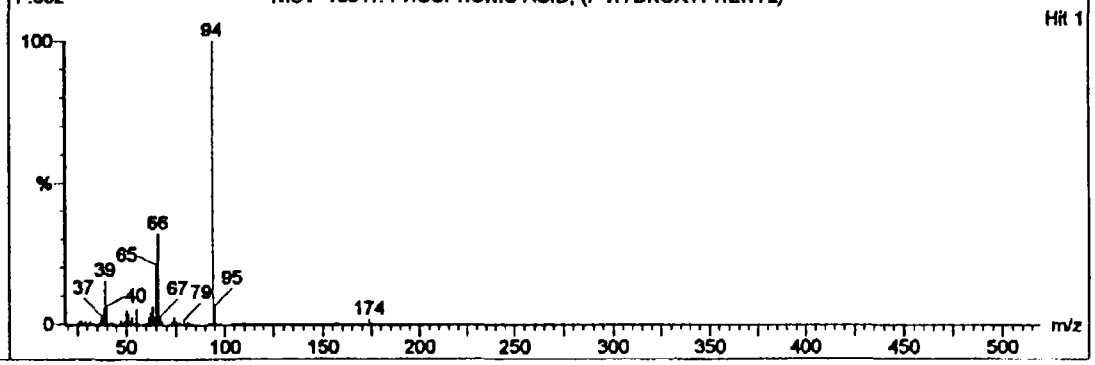


Compound Name: PHOSPHONIC ACID, (P-HYDROXYPHENYL)-
Synonym:
Molecular Weight: 174
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 805
Y1014221 439 (8.702)



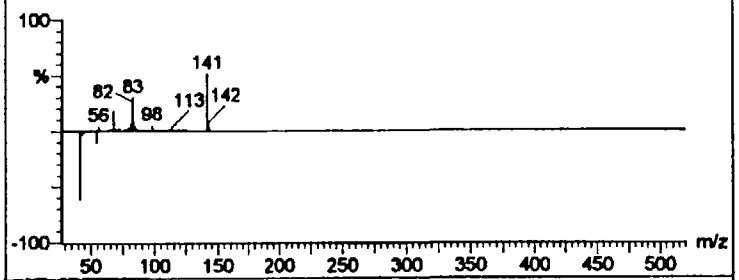
F:662 NIST 16041: PHOSPHONIC ACID, (P-HYDROXYPHENYL)- Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

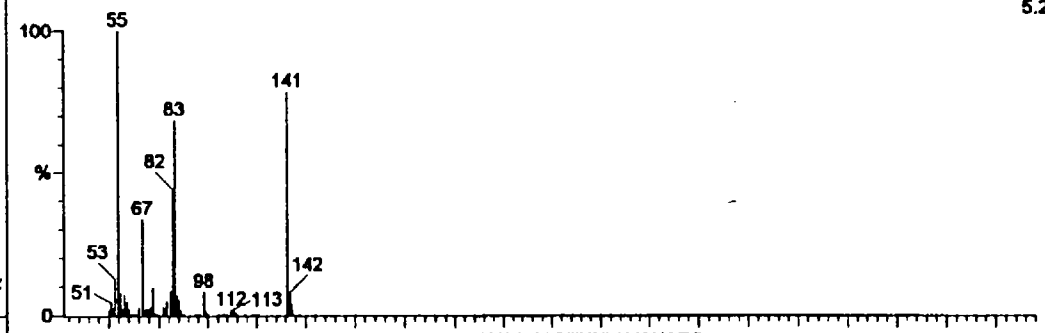
Hit	Compound Name	FOR	rev
1	CYCLOHEXANE, ISOTHIOCYANATO-	709	723
2	THIOUREA, N,N'-DICYCLOHEXYL-	512	525
3	3-PENTENOIC ACID, 3-ETHYL-, METHYL ESTER	507	537
4	2-FURANCARBOXALDEHYDE, 5-NITRO-	496	550
5	DIAZENE, 1-CYCLOHEXYL-2-METHOXY-, 1-OXIDE	492	623
6	CYCLOHEXANE, 2-PROPENYL-	480	701
7	METHYPRYLON DESMETHYL-ARTEFACT	467	644
8	METHANONE, DICYCLOHEXYL-	466	640
9	3-HEXENOIC ACID, 3-METHYL-, METHYL ESTER	462	491
10	2,4(1H,3H)-PYRIMIDINEDIONE, 1,3-DIMETHYL-	460	491
11	3-HEXENOIC ACID, 2-METHYL-, METHYL ESTER	456	595
12	CYCLOHEXANE, 1,1'-METHYLENEBIS-	448	626
13	ANODENDRINE, IODIDE, METHYL ESTER	448	505
14	CYCLOPENTANE, 1-METHYL-2-(2-PROPENYL)-, TRANS-	429	612
15	CYCLOHEXANEACETIC ACID	427	574
16	2-SEC-BUTYL-4,6-DINITROPHENYL 3-METHYLCROTONATE (BINAPACRYL)	426	682
17	2H,7H-PYRANO 2,3-B PYRAN, HEXAHYDRO-	418	567
18	CYCLOHEXANAMINE, N-METHYL-N-NITROSO-	418	450
19	TROPINE	414	448
20	PENTYLENETETRAZOLE(METRAZOL)	411	654

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 709, Reverse Fit: 723**
Y1014221 650 (10.463) Cm (650-7677)

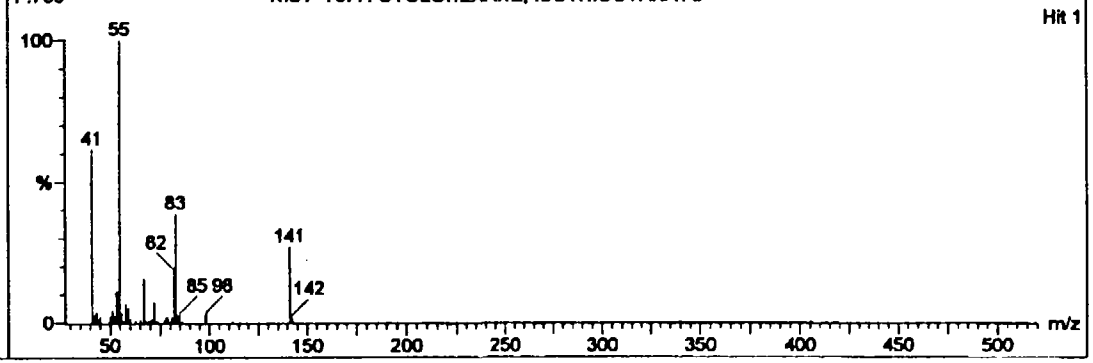


Compound Name: CYCLOHEXANE, ISOTHIOCYANATO-
Synonym:
Molecular Weight: 141
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 723
Y1014221 650 (10.463) 5.20e6



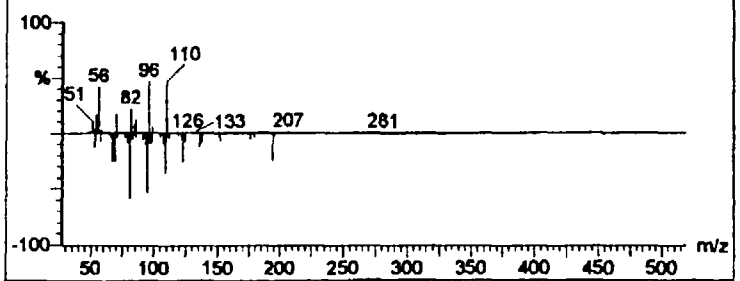
F:709 NIST 7677: CYCLOHEXANE, ISOTHIOCYANATO- Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

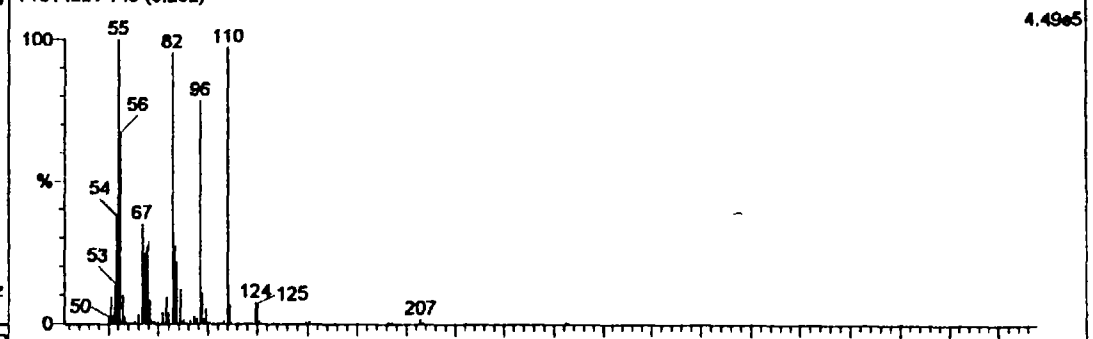
Hit	Compound Name	FOR	rev
1	2(1H)-BENZOCYCLOOCTENONE, DECAHYDRO-10A-METHYL-, TRANS-	601	637
2	5-BUTYL-2-METHYLPYRROLINE	601	623
3	7-AZABICYCLO 4.1.0 HEPTANE, 1-METHYL-	594	638
4	OCTANENITRILE	583	619
5	(Z)6-PENTADECEN-1-OL	559	592
6	2-CYCLOHEXEN-1-ONE, 4,5-DIMETHYL-	552	594
7	ETHANAMINE, N-CYCLOPENTYLIDENE-	550	641
8	OLEYL ALCOHOL	544	585
9	CYCLOHEXANE, ISOCYANATO-	544	568
10	ETHYL (1H)-1,2,4-TRIAZOLE-1-ACETATE	539	673
11	DECANENITRILE	533	612
12	CYCLOHEXANE, 1,1'-(1-METHYL-1,3-PROPANEDIYL)BIS-	532	567
13	CYCLOPENTADECANOL-	530	562
14	1,13-TETRADECADIENE	530	565
15	1,10-DICYANODECANE	530	564
16	HEPTANENITRILE, 4-ACETYL-	530	557
17	UNDECANENITRILE, 11-BROMO-	529	567
18	CYCLOPENTANEPROPANOIC ACID, 2-OXO-, METHYL ESTER	528	591
19	11-HEXADECEN-1-OL, (Z)-	527	568
20	1,12-DODECANEDIOL	525	559

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 601, Reverse Fit: 637**
Y1014221 143 (6.232) Cm (143-21390)

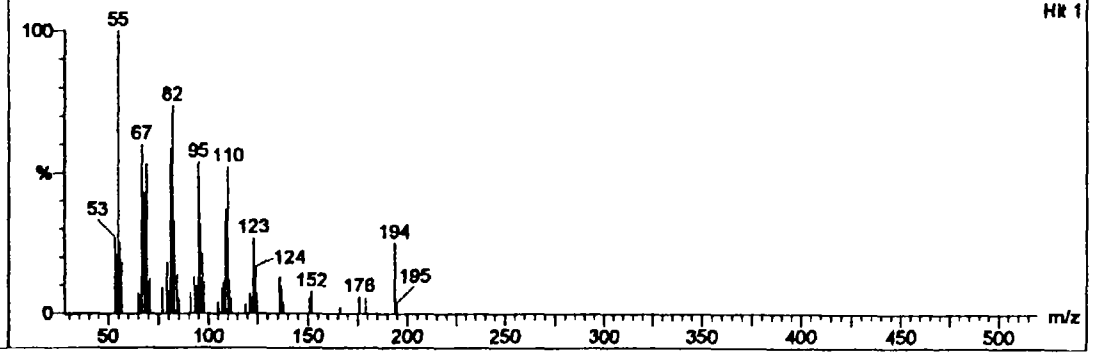


Compound Name: 2(1H)-BENZOCYCLOOCTENONE, DECAHYDRO
Synonym:
Molecular Weight: 194
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 637
Y1014221 143 (6.232)



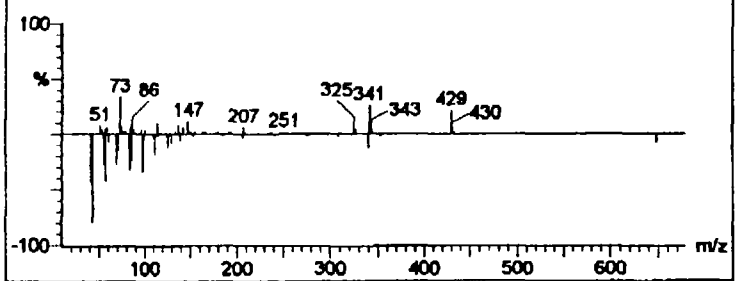
F:601 NIST 21390: 2(1H)-BENZOCYCLOOCTENONE, DECAHYDRO-10A-METHYL-, TRANS- Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

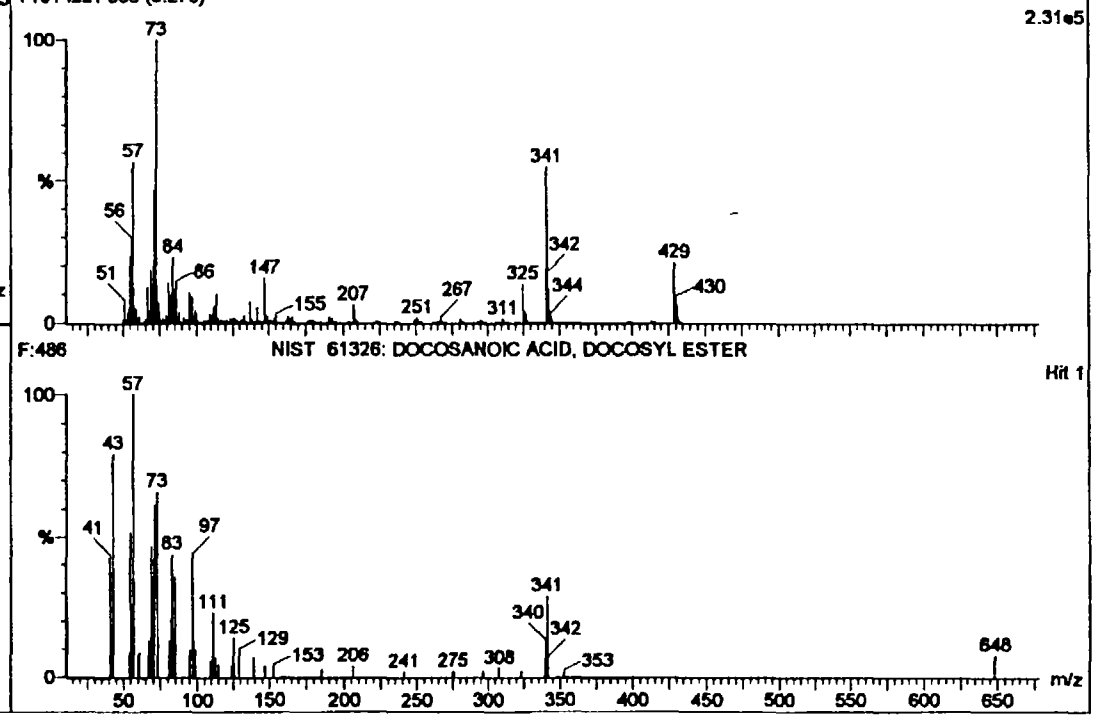
Hit	Compound Name	FOR	rev
1	DOCOSANOIC ACID, DOCOSYL ESTER	486	643

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 486, Reverse Fit: 643**
Y1014221 508 (9.279) Cm (508-61326)



Compound Name: DOCOSANOIC ACID, DOCOSYL ESTER
Synonym:
Molecular Weight: 648
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 643
Y1014221 508 (9.279)

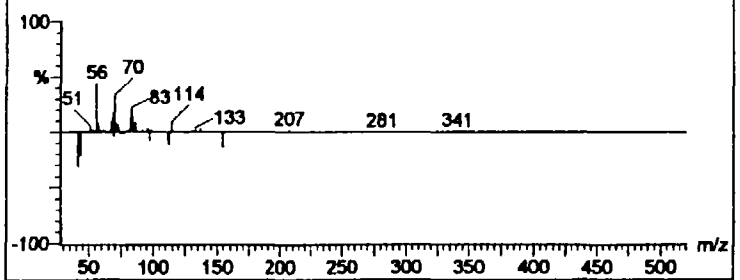


Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

Hit	Compound Name	FOR	rev
1	CYCLOPROPANE, 1-BUTYL-1-METHYL-2-PROPYL-	722	783
2	DODECANE, 1-FLUORO-	691	733
3	CYCLOBUTANE, 3-HEXYL-1,1,2-TRIMETHYL-, CIS-	677	734
4	4-UNDECENE, 5-METHYL-, (Z)-	673	724
5	5-UNDECENE	673	727
6	CYCLOPROPANE, 1-METHYL-1-(2-METHYLPROPYL)-2-NONYL-	663	720
7	4-UNDECENE, 4-METHYL-, (Z)-	663	705
8	CYCLOPROPANE, 1,2-DIBUTYL-	663	730
9	CYCLODECANE, METHYL-	663	718
10	CYCLOPROPANE, 1,1-DIMETHYL-2-NONYL-	660	734
11	CYCLOOCTANE, 1,5-DIMETHYL-	659	715
12	4-UNDECENE, 4-METHYL-	658	723
13	8-HEPTADECENE, 8-METHYL-, (E)-	651	706
14	3-UNDECENE, 5-METHYL-	648	707
15	CYCLOPENTANE, 1-HEXYL-3-METHYL-	647	698
16	4-UNDECENE, 6-METHYL-	647	703
17	5-OCTADECENE, (E)-	644	692
18	7-HEXADECENE, (Z)-	643	687
19	4-UNDECENE, 3-METHYL-, (Z)-	641	688
20	5-TETRADECENE, (E)-	640	692

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST — **Forward Fit: 722, Reverse Fit: 783**

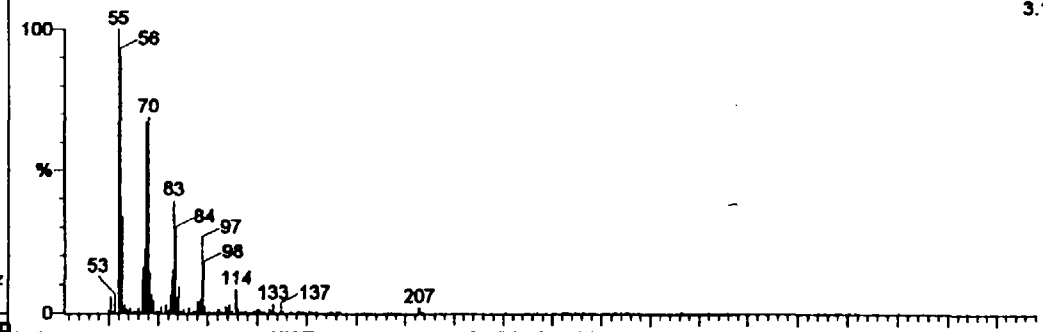
Y1014221 519 (9.371) Cm (519-11034)



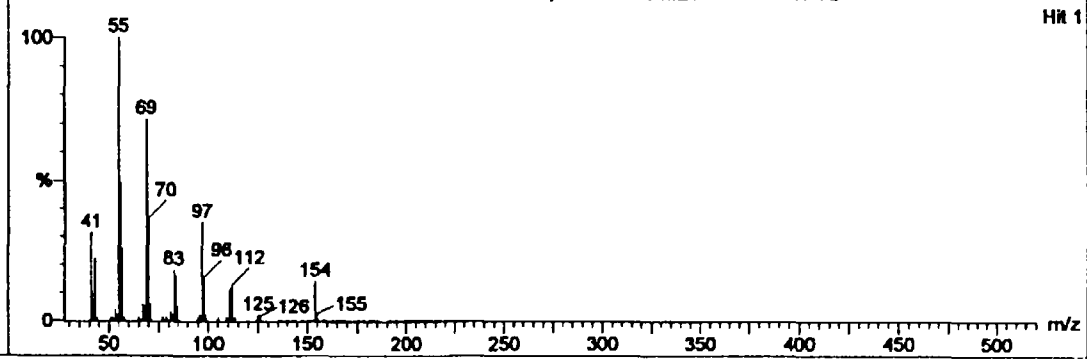
Compound Name: CYCLOPROPANE, 1-BUTYL-1-METHYL-2-PROP
Synonym:
Molecular Weight: 154
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 783

Y1014221 519 (9.371) — 3.17e5



F:722 — NIST 11034: CYCLOPROPANE, 1-BUTYL-1-METHYL-2-PROPYL- — Hit 1

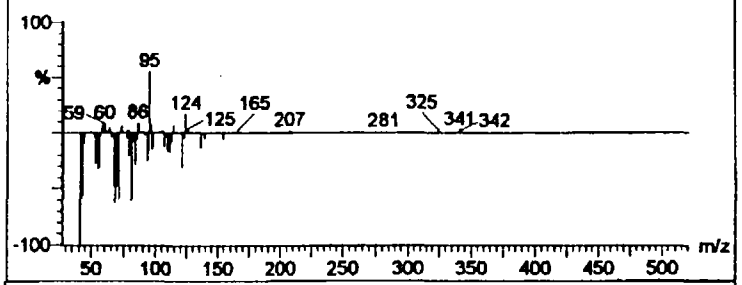


Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

Hit	Compound Name	FOR	rev
1	CYCLOHEXANOL, 5-METHYL-2-(1-METHYLETHENYL)-	635	715
2	CYCLOHEXANOL, 5-METHYL-2-(1-METHYLETHENYL)-, 1R-(1.ALPHA.,2.BETA.,5.ALPHA.) -	597	680
3	6-OCTENAL, 3,7-DIMETHYL-, (R)-	547	630
4	BICYCLO 3.1.1 HEPTAN-3-OL, 2,6,6-TRIMETHYL-, 1S-(1.ALPHA.,2.BETA.,3.ALPHA.,5.ALPHA.) -	523	598
5	2,10-DODECADIEN-1-OL, 3,7,11-TRIMETHYL-, (E)-(.+/-.)-	511	614
6	2,3,6-TRIMETHYLHEPT-3-EN-1-OL	507	610
7	1,3-HEXADIENE, 3-ETHYL-2-METHYL-, (Z)-	505	603
8	R(+)5,7-DIMETHYL-1,6-OCTADIENE	500	599
9	8,11,14-DOCOSATRIENOIC ACID, METHYL ESTER	497	608
10	BICYCLO 3.1.1 HEPTAN-3-OL, 2,6,6-TRIMETHYL-, 1R-(1.ALPHA.,2.BETA.,3.ALPHA.,5.ALPHA.) -	491	562
11	1,1'-BICYCLOPENTYL -1-OL	487	588
12	BICYCLO 3.1.1 HEPTAN-3-OL, 2,6,6-TRIMETHYL-, (1.ALPHA.,2.BETA.,3.ALPHA.,5.ALPHA.)-	483	556
13	BICYCLO 3.1.1 HEPTAN-2-ONE, 3,6,6-TRIMETHYL-	479	609
14	2(1H)-NAPHTHALENONE, OCTAHYDRO-4A,7,7-TRIMETHYL-, CIS-	476	570
15	5,9-TRIDECADIEN-1-OL, 10-PROPYL-, ACETATE	474	608
16	AMORPHANE-B	462	622
17	10-UNDECENOYL CHLORIDE	461	589
18	1,1'-BICYCLOHEXYL, 2-PROPYL-, TRANS-	456	553
19	BICYCLO 2.2.1 HEPTAN-2-ONE, 1,7,7-TRIMETHYL-, (1S)-	456	628
20	1,1'-BICYCLOHEXYL, 2-PROPYL-, CIS-	448	543

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 635, Reverse Fit: 715**

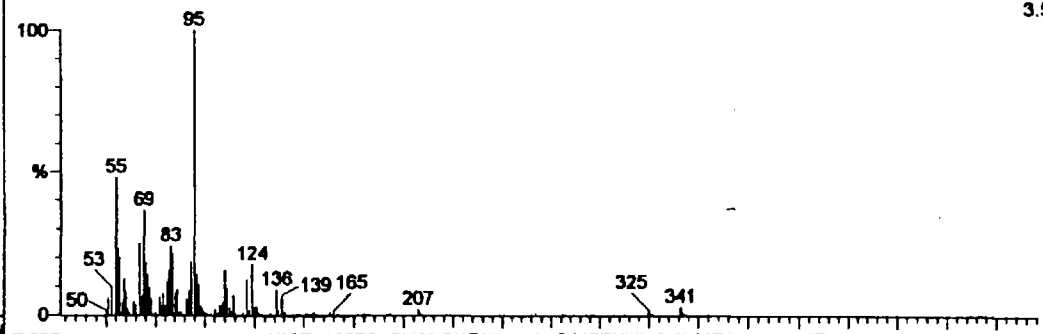
Y1014221 537 (9.521) Cm (537-10972)



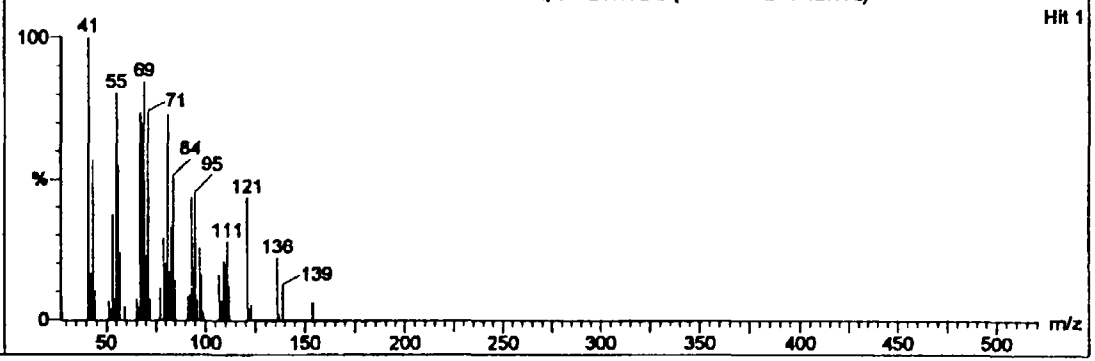
Compound Name: CYCLOHEXANOL, 5-METHYL-2-(1-METHYLETH
Synonym:
Molecular Weight: 154
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 715

Y1014221 537 (9.521) — 3.52e5



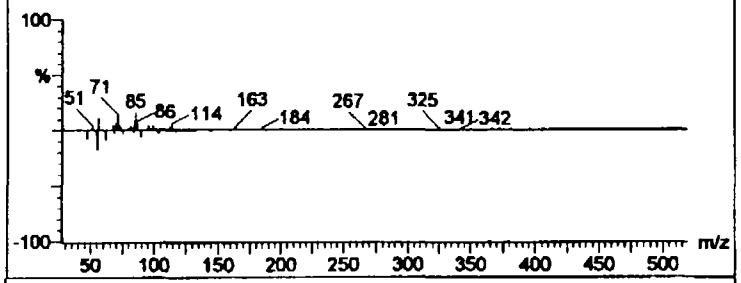
F:635 — NIST 10972: CYCLOHEXANOL, 5-METHYL-2-(1-METHYLETHENYL)- — Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

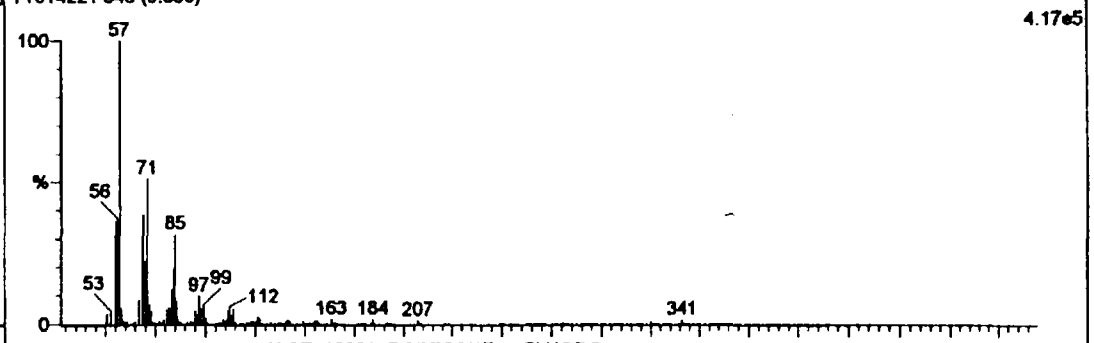
Hit	Compound Name	FOR	rev
1	DODECANE, 1-FLUORO-	774	835
2	DECANEDIOIC ACID, DIDECYL ESTER	651	736
3	TRIDECANOL	629	698
4	1-OCTANOL, 2-BUTYL-	624	675
5	DECANE, 1,1'-OXYBIS-	611	657
6	NONADECANE	609	663
7	NONANE, 4,5-DIMETHYL-	600	643
8	1-DODECANOL, 2-METHYL-, (S)-	598	646
9	1-DECANOL, 2-ETHYL-	579	628
10	DECANE, 1-(ETHENYLOXY)-	577	622
11	HEXYL OCTYL ETHER	558	639
12	HEXADECANE, 1-METHOXY-13-METHYL-	535	584
13	DECANE, 2,5,6-TRIMETHYL-	529	629
14	PYRROLIDINE, 1-(1,6-DIOXOOCTADECYL)-	524	576
15	1-UNDECENE, 4-METHYL-	509	548
16	UNDECANE, 5,6-DIMETHYL-	500	561
17	DECANE, 2,4,6-TRIMETHYL-	498	562
18	HEPTANE, 2,2,3,4,6,6-HEXAMETHYL-	497	603
19	1-UNDECENE, 8-METHYL-	497	538
20	OCTANE, 2,4,6-TRIMETHYL-	496	544

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 774, Reverse Fit: 835**
Y1014221 546 (9.596) Cm (546-19931)

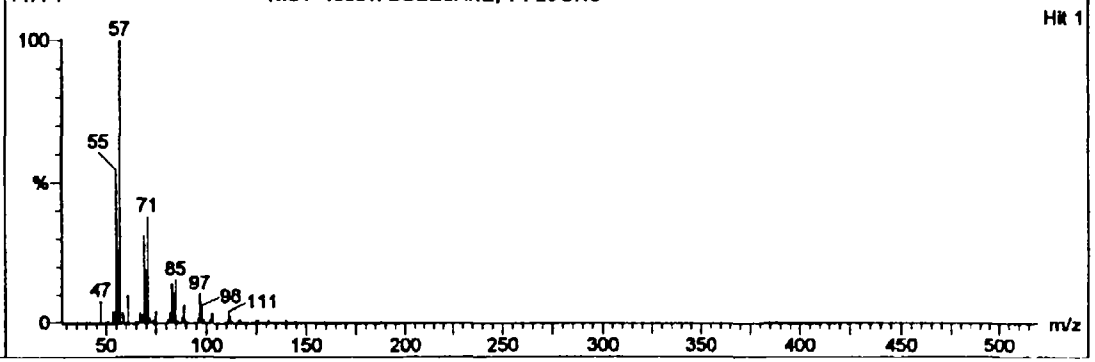


Compound Name: DODECANE, 1-FLUORO-
Synonym:
Molecular Weight: 188
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 835
Y1014221 546 (9.596)



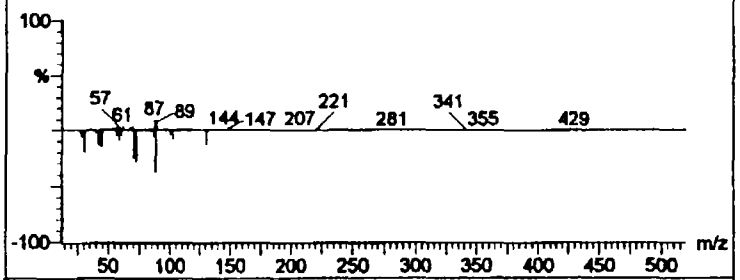
F:774 NIST 19931: DODECANE, 1-FLUORO- Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

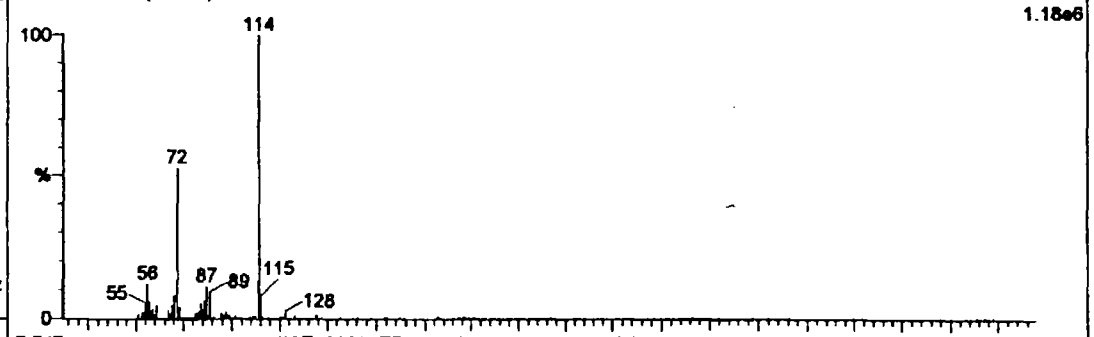
Hit	Compound Name	FOR	rev
1	ETHANOL, 2- BIS(1-METHYLETHYL)AMINO -	547	609
2	METHIMAZOLE REF-PMS-P358	530	744
3	OXAZOLIDINE, 3-ETHYL-2,2-DIMETHYL-	513	615
4	DIETHYLPROPION-N-DESETHYL,ACETATE	486	657
5	PENTANAMIDE, 2-(DIMETHYLAMINO)-4-METHYL-N- 2-METHYL-1- 3,3A,11,12,13,14,15,15A-OCTAHYDRO-12,15-DIOX	485	681
6	DIETHYLPROPION-N-DESETHYL, ACETATE	485	656
7	PHENADOXONE REF-PMS-P452	446	887
8	3-(DIISOPROPYLAMINO)-1,2-PROPANEDIOL	423	519
9	N-ETHYLAMPHETAMINE ACETATE	417	600
10	2-PROPANAMINE, N-ETHYL-N-(1-METHYLETHYL)-	413	497

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 547, Reverse Fit: 609**
Y1014221 608 (10.113) Cm (608-8639)

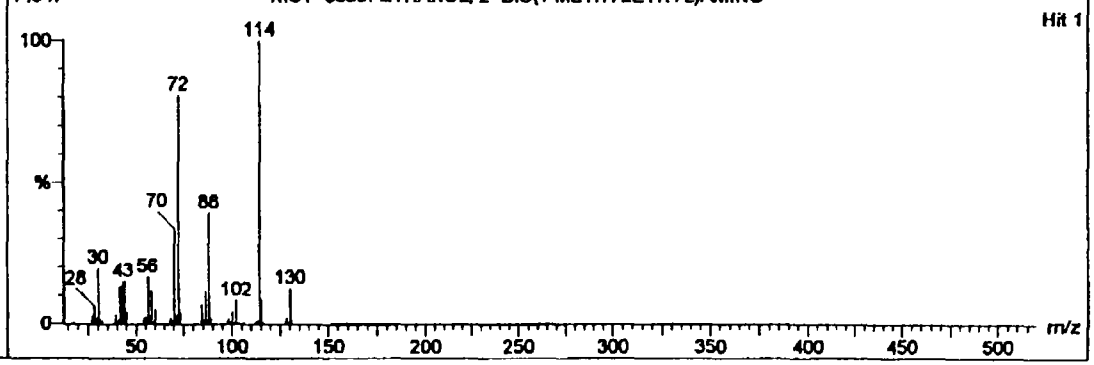


Compound Name: ETHANOL, 2- BIS(1-METHYLETHYL)AMINO -
Synonym:
Molecular Weight: 145
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 609
Y1014221 608 (10.113)



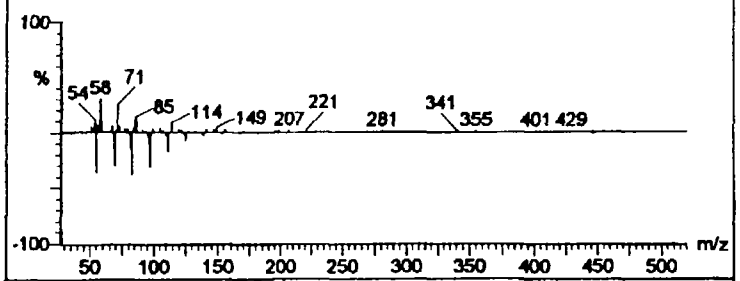
F:547 NIST 8639: ETHANOL, 2- BIS(1-METHYLETHYL)AMINO - Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

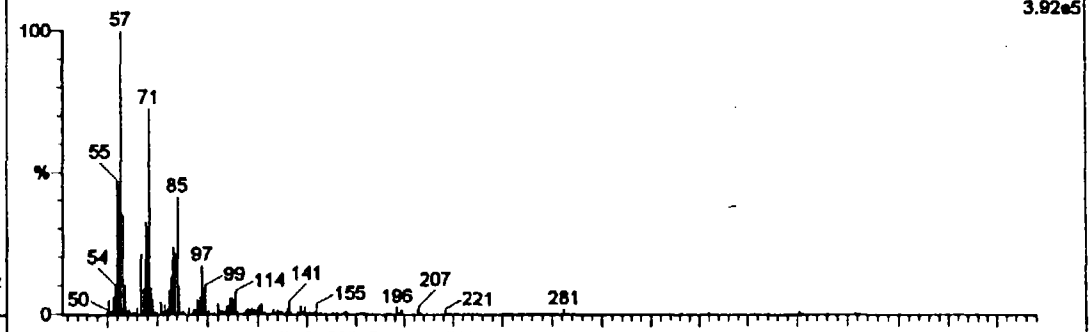
Hit	Compound Name	FOR	rev
1	1-DOTRIACONTANOL	762	866
2	DODECANE, 1-FLUORO-	749	817
3	TETRACONTANE, 3,5,24-TRIMETHYL-	688	752
4	TRITETRACONTANE	686	782
5	OXIRANE, (DODECYLOXY)METHYL -	675	728
6	1-DECANOL, 2-HEXYL-	664	720
7	DECANEDIOIC ACID, DIDECYL ESTER	642	743
8	HEPTADECANE, 2,6,10,15-TETRAMETHYL-	625	705
9	DECANE, 1,1'-OXYBIS-	624	687
10	1-OCTADECANETHIOL	619	678
11	1-OCTANOL, 2-BUTYL-	613	677
12	HEXADECANE, 1-(ETHENYLOXY)-	610	669
13	PENTATRIACONTANE	609	680
14	CYCLOOCTACOSANE	609	703
15	HEPTANE, 1,1'-OXYBIS-	607	700
16	BICYCLO 3.2.1 OCTAN-2-ONE, 1-(1-PROPENYL)-	599	800
17	10-METHYLNONADECANE	596	658
18	NONADECANE	595	659
19	DOCOSANE	591	670
20	1-DODECANOL, 2-METHYL-, (S)-	591	646

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 762, Reverse Fit: 866**
Y1014221 668 (10.613) Cm (668-57795)

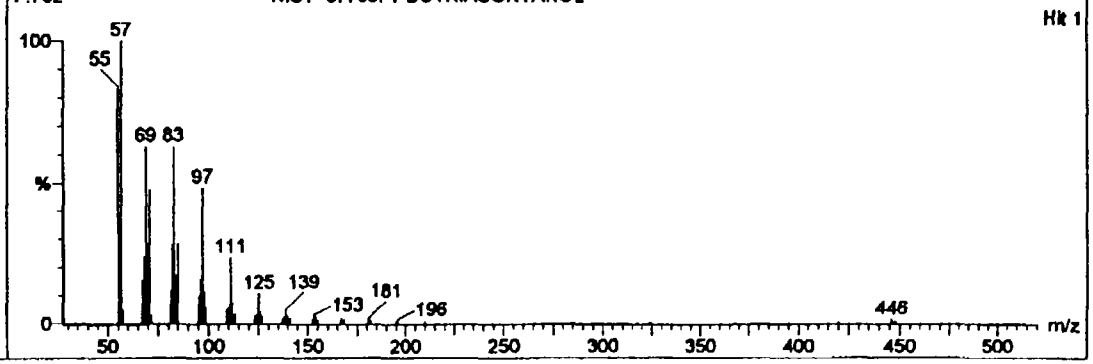


Compound Name: 1-DOTRIACONTANOL
Synonym:
Molecular Weight: 466
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 866
Y1014221 668 (10.613) 3.92e5



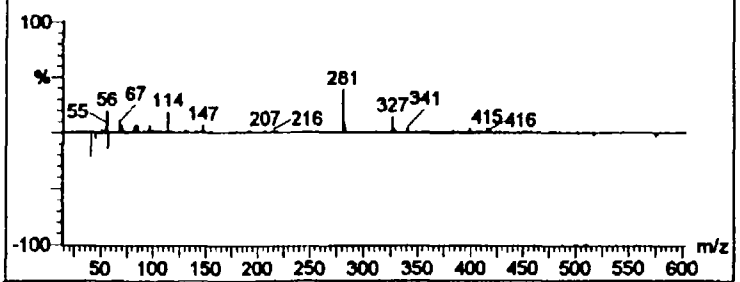
F:762 NIST 57795: 1-DOTRIACONTANOL Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

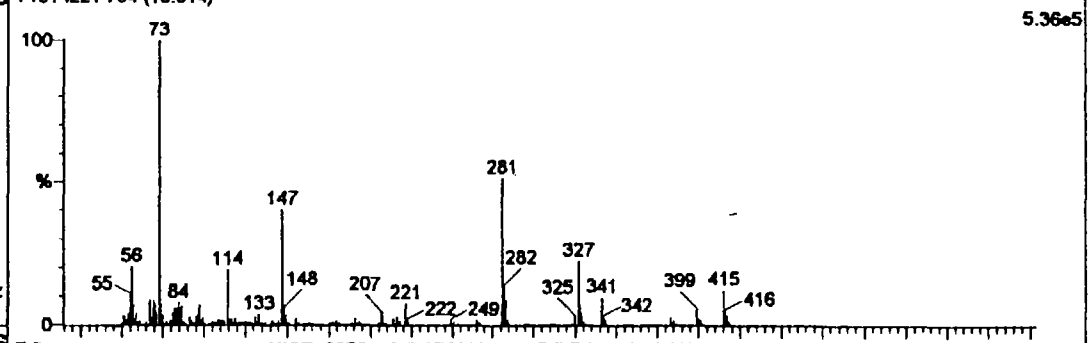
Hit	Compound Name	FOR	rev
1	3-BUTOXY-1,1,1,7,7,7-HEXAMETHYL-3,5,5-TRIS(TRIMETHYLSILOXY)TETRASILOXANE	549	765
2	3-ISOPROPOXY-1,1,1,7,7,7-HEXAMETHYL-3,5,5-TRIS(TRIMETHYLSILOXY)TETRASILOXANE	526	772
3	3-ETHOXY-1,1,1,7,7,7-HEXAMETHYL-3,5,5-TRIS(TRIMETHYLSILOXY)TETRASILOXANE	516	781
4	TRISILOXANE, 1,1,1,5,5,5-HEXAMETHYL-3,3-BIS (TRIMETHYLSILYL)OXY -	419	780
5	1,1,1,3,5,7,9,11,11,11-DECAMETHYL-5-(TRIMETHYLSILOXY)HEXASILOXANE	418	756
6	1,1,1,3,5,5,7,7,7-NONAMETHYL-3-(TRIMETHYLSILOXY)TETRASILOXANE	403	793

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 549, Reverse Fit: 765**
Y1014221 704 (10.914) Cm (704-60704)

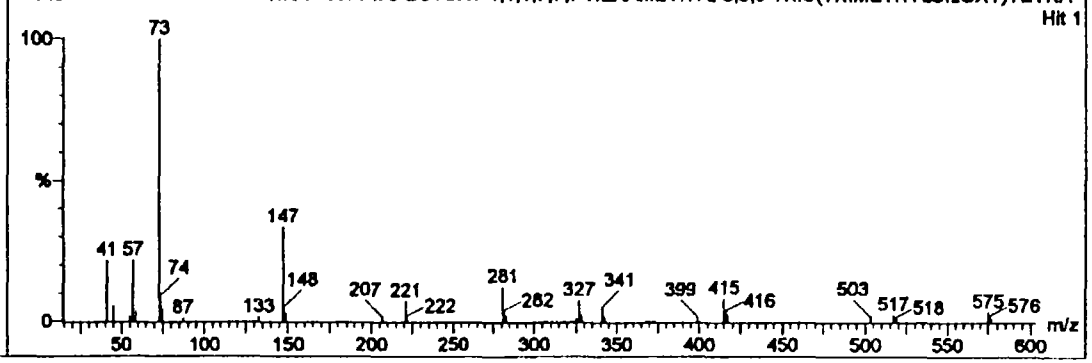


Compound Name: 3-BUTOXY-1,1,1,7,7,7-HEXAMETHYL-3,5,5-TRIS
Synonym:
Molecular Weight: 590
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 765
Y1014221 704 (10.914)



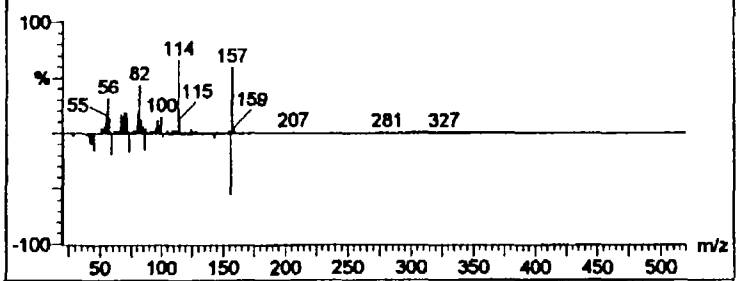
F:549 NIST 60704: 3-BUTOXY-1,1,1,7,7,7-HEXAMETHYL-3,5,5-TRIS(TRIMETHYLSILOXY)TETRA
Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

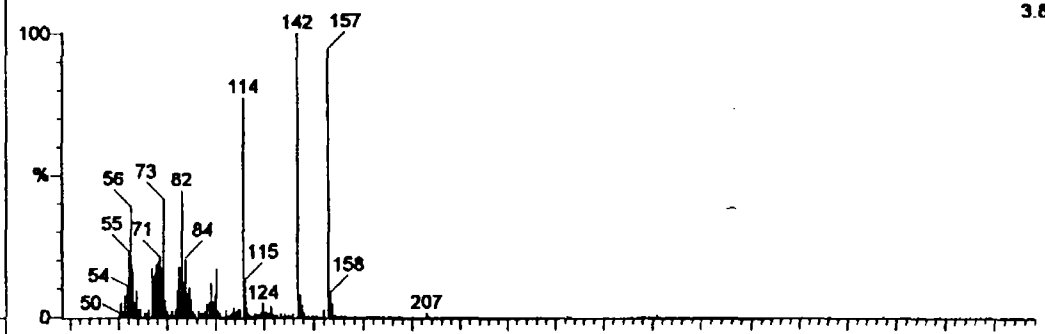
Hit	Compound Name	FOR	rev
1	PIPERIDINE, 1-(TRIMETHYLSILYL)-	466	558
2	.ALPHA.-D-ARABINO-HEXOPYRANOSE, 2-DEOXY-, TETRAACETATE	438	519
3	METHYL 3-O-ACETYL-2,6-DIDEOXY-2-(N-METHYLACETAMIDO)-4-O-METHYL-D-GALACTOPYRANOSIDE	415	522
4	4(1H)-PYRIMIDINONE, 2,3-DIHYDRO-5-METHYL-2-THIOXO-	408	666
5	3-PIPERIDINOL, 1-ACETYL-6-METHYL-	404	458

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 466, Reverse Fit: 558**
Y1014221 711 (10.972) Cm (711-11716)

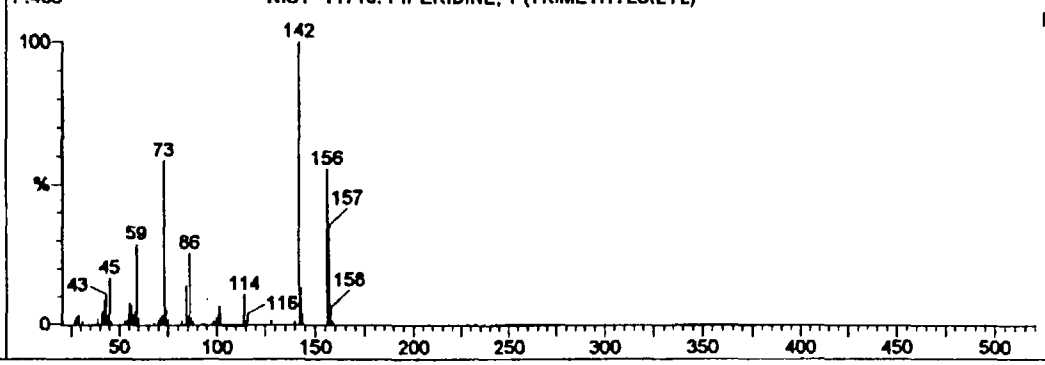


Compound Name: PIPERIDINE, 1-(TRIMETHYLSILYL)-
Synonym:
Molecular Weight: 157
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 558
Y1014221 711 (10.972) 3.83e5



F:466 NIST 11716: PIPERIDINE, 1-(TRIMETHYLSILYL)- Hit 1

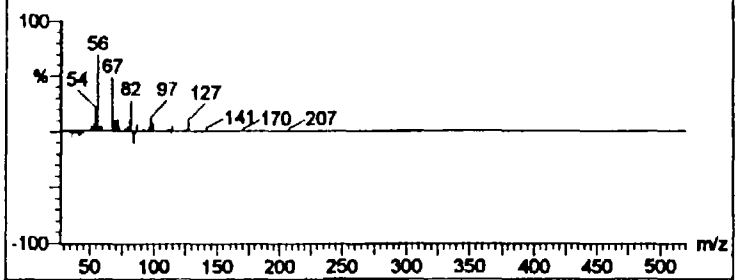


Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

Hit	Compound Name	FOR	rev
1	2-ETHYLPIPERIDINE	660	715
2	PIPERIDINE, 2-PROPYL-, (S)-	545	721
3	CONIINE	538	712
4	2-CYCLOHEXYLPIPERIDINE	530	618
5	7-TETRADECENE	478	535
6	CYCLOHEPTANONE, 2-METHYL-	476	510
7	5.ALPHA.-PREGNAN-3.BETA.-AMINE, N,N-DIMETHYL-	472	573
8	ETHANAMINE, N-PENTYLIDENE-	472	596
9	PYRIMIDIN-2,4-DIONE, 1,2,3,4-TETRAHYDRO-5-METHYL-1- 2-HYDROXYMETHYL-3-DIMETHYLAMINO TETRAHYDROFUR-5	466	509
10	1-METHYL-2-(4-METHYLPENTYL)CYCLOPENTANE	466	524
11	TRANS-1-METHYL-2-(4-METHYLPENTYL)CYCLOPENTANE	465	523
12	CYCLOPENTANE, 1-HEXYL-3-METHYL-	454	505
13	1-OXASPIRO 4.4 NONAN-4-ONE, 2-ISOPROPYL-	450	505
14	ETHANONE, 1-(2,2-DIMETHYLCYCLOPENTYL)-	450	482
15	1-HENEICOSYL FORMATE	444	516
16	OXONANE	434	486
17	7-HEPTADECENE, 1-CHLORO-	433	508
18	2-PROPENOIC ACID, 1,6-HEXANEDIYL ESTER	425	466
19	CYCLOPENTANE, 1-ETHENYL-3-ETHYL-2-METHYL-	421	490
20	TRANS-2-UNDECEN-1-OL	420	478

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 660, Reverse Fit: 715**

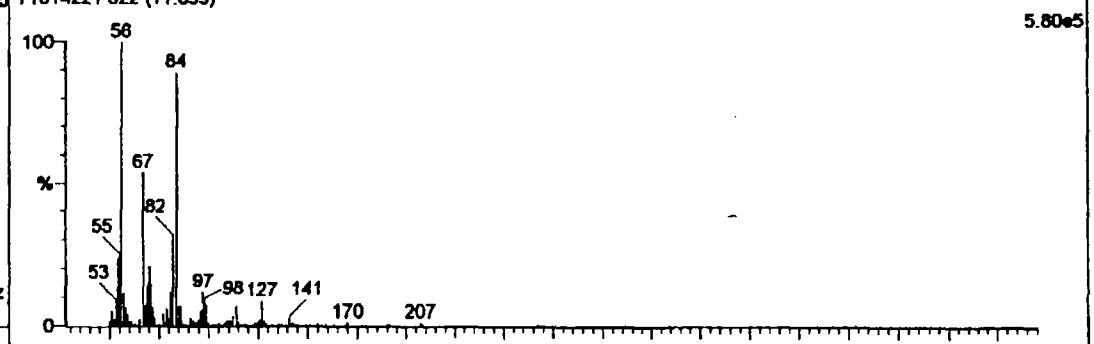
Y1014221 822 (11.899) Cm (822-2789)



Compound Name: 2-ETHYLPIPERIDINE
Synonym:
Molecular Weight: 113
structure fou

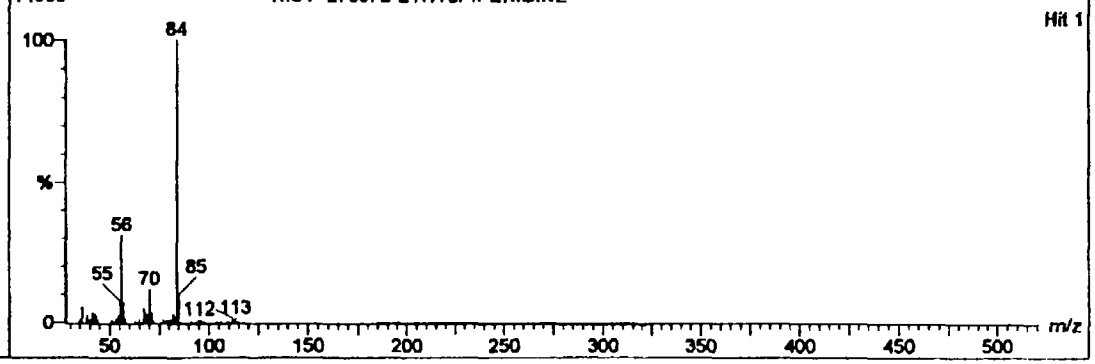
Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 715

Y1014221 822 (11.899)



F:660 NIST 2789: 2-ETHYLPIPERIDINE

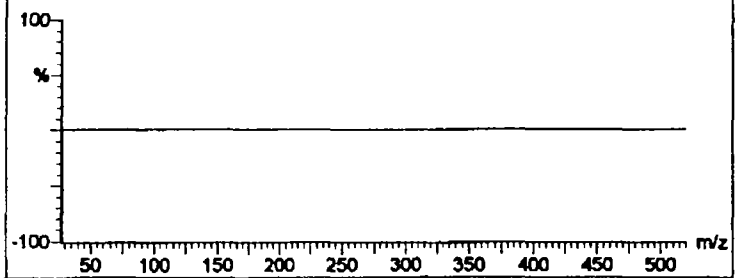
Hit 1



Sample ID: 2uL Tray 153 R1 **Acquired 18:15:04 at 18:15:04**

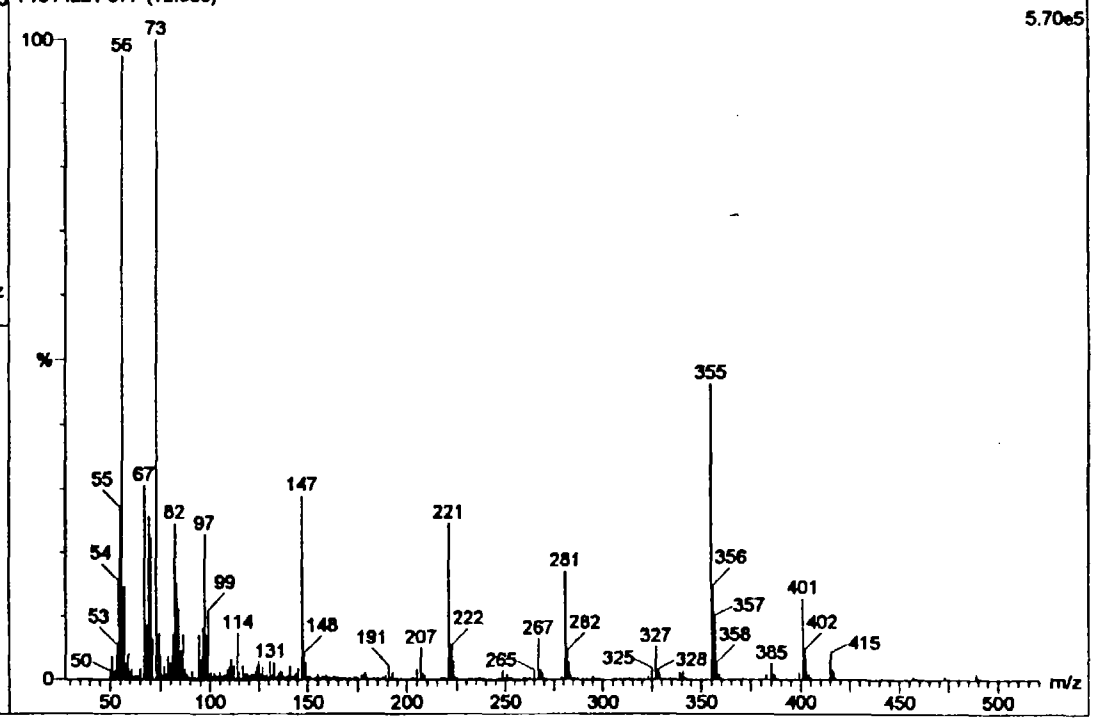
Hit	Compound Name	FOR	rev

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 660, Reverse Fit: 715**
0 (0.000)



Compound Name: 2-ETHYLPIPERIDINE
Synonym:
Molecular Weight: 113
structure fou

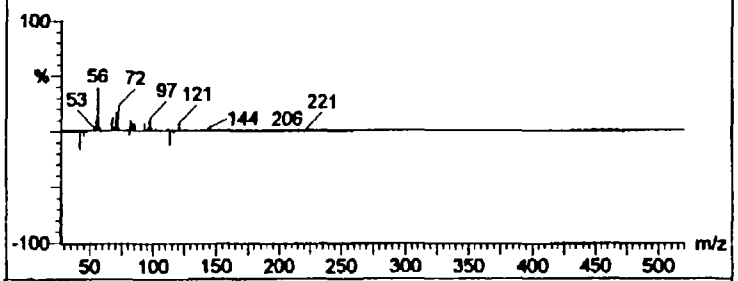
Sample ID: 2uL Tray 153 R1 **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 715
Y1014221 877 (12.359)



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

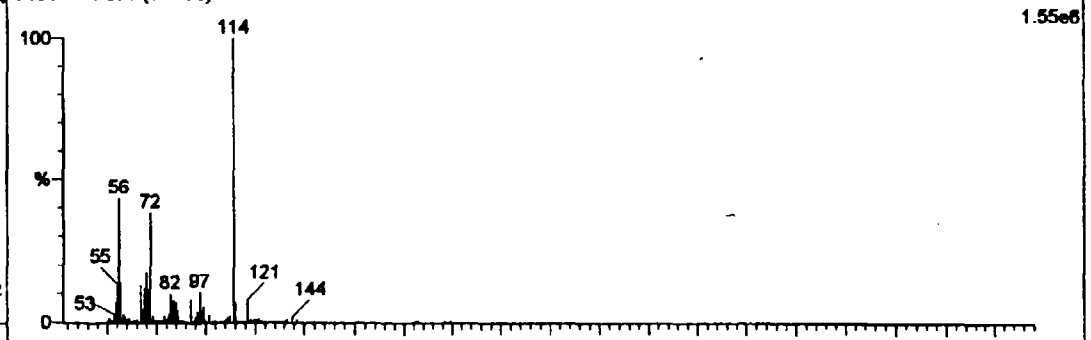
Hit	Compound Name	FOR	rev
1	METHIMAZOLE REF-PMS-P358	450	714

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: LIBTX **Forward Fit: 450, Reverse Fit: 714**
Y1014221 891 (12.476) Cm (891-1758)

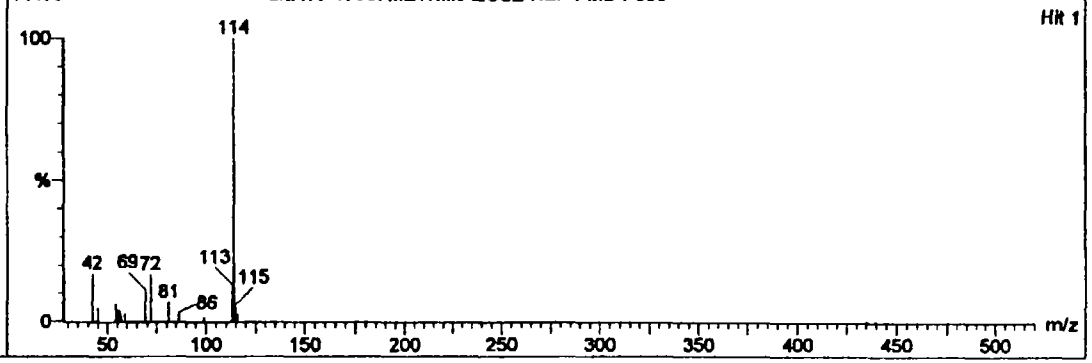


Compound Name: METHIMAZOLE REF-PMS-P358
Synonym:
Molecular Weight: 114
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 714
Y1014221 891 (12.476)



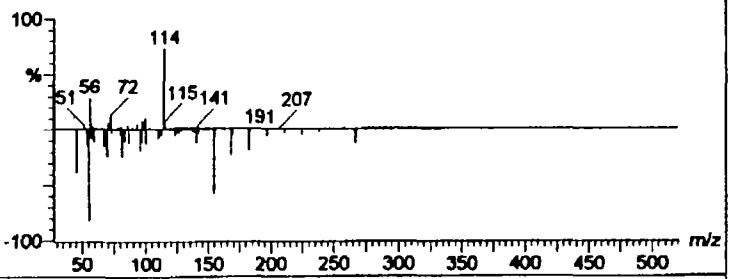
F:450 LIBTX 1758: METHIMAZOLE REF-PMS-P358 Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

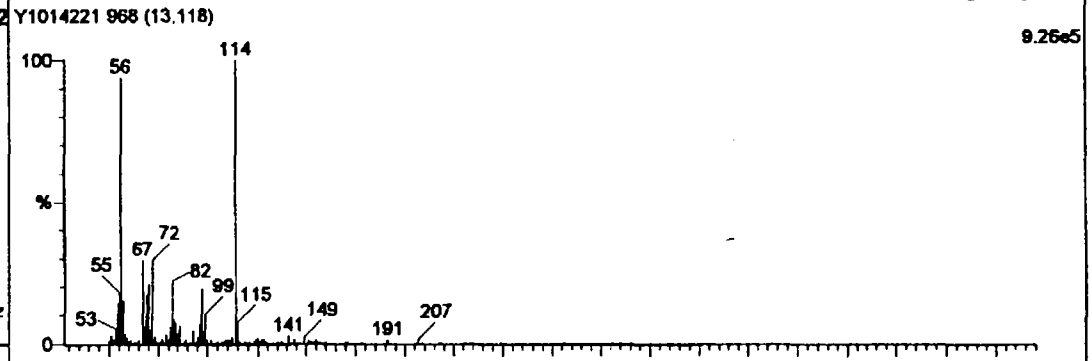
Hit	Compound Name	FOR	rev
1	9-OCTADECEN-1-AMINE	525	592
2	1-DOTRIACONTANOL	471	696
3	2-PROPENOIC ACID, 3-(DIMETHYLAMINO)-3-(METHYLAMINO)-, METHYL ESTER	452	543
4	CYCLOPROPANEACETIC ACID, 2-HEXYL-	448	510
5	1,2-CYCLOHEXANEDIAMINE	445	485
6	(1R,2R)-(-)-1,2-DIAMINOCYCLOHEXANE	442	484
7	2-PROPENOIC ACID, 3-(DIETHYLAMINO)-3-(DIMETHYLAMINO)-, METHYL ESTER	438	536
8	13-OXABICYCLO 10.1.0 TRIDECANE	434	597
9	2-PENTENAMIDE, N-(2-OXO-3-PIPERIDINYL)-	429	508
10	(1S,2S)-(+)-1,2-DIAMINOCYCLOHEXANE	415	459
11	2,3,6-TRIMETHYLHEPT-6-EN-1-OL	414	555
12	CYCLODECANE, METHYL-	413	583
13	CYCLOPENTANE, 1-HEXYL-3-METHYL-	408	606
14	AZIRIDINE, 1-ACETYL-2-METHYL-	404	583
15	1-HEXADECANOL	402	563
16	7-TETRADECENE	400	604
17	10-METHYLOXACYCLODECAN-2-ONE	400	456
18	METHIMAZOLE REF-PMS-P358	400	640

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 525, Reverse Fit: 592**
Y1014221 968 (13.118) Cm (968-37212)

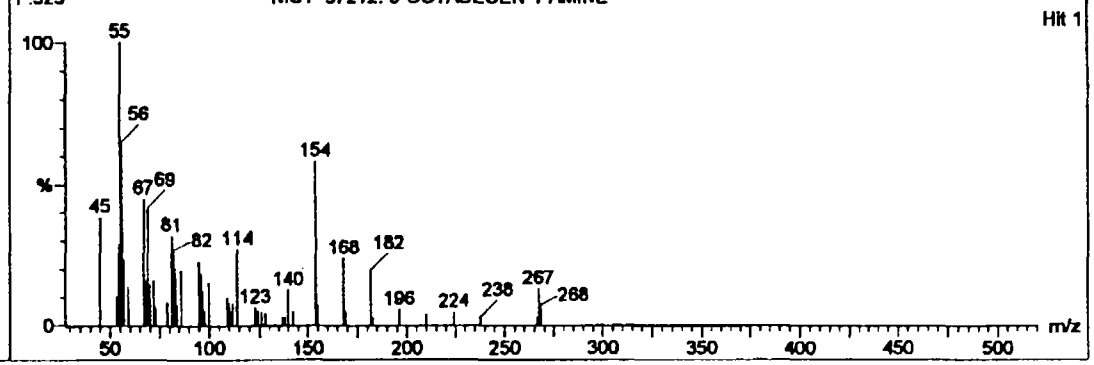


Compound Name: 9-OCTADECEN-1-AMINE
Synonym:
Molecular Weight: 267
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 592
Y1014221 968 (13.118)



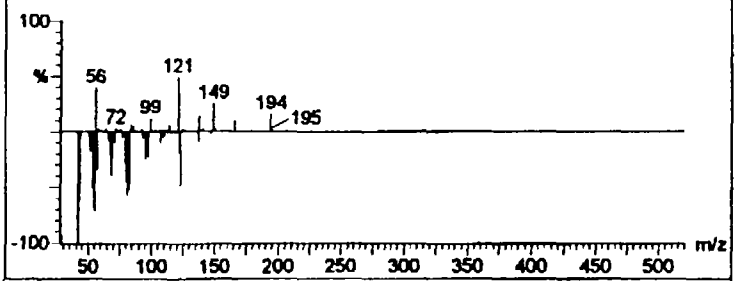
F:525 NIST 37212: 9-OCTADECEN-1-AMINE — Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

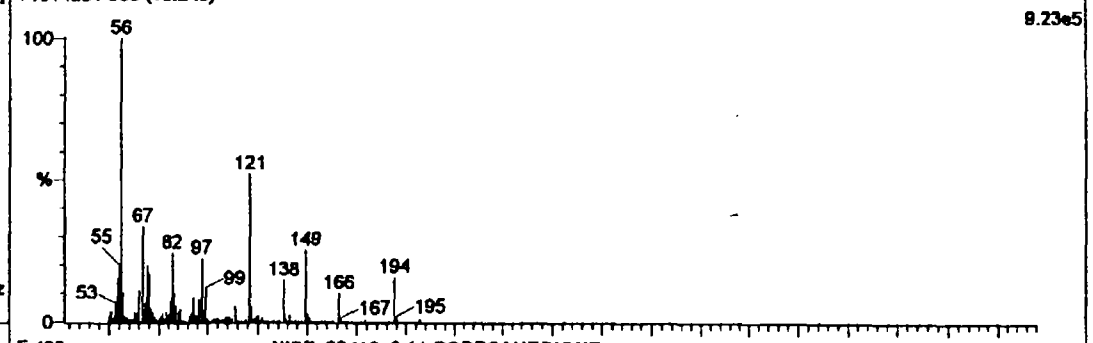
Hit	Compound Name	FOR	rev
1	2,11-DODECANEDIONE	438	557
2	IRON, TRICARBONYL(.ETA.3-2-PROPENYL)-2-PROPENYL-	432	557
3	CYCLOHEXANE, 1,1'-(1-METHYL-1,3-PROPANEDIYL)BIS-	422	497
4	CYCLOHEXANE, ISOCYANATO-	421	609
5	CYCLOPENTANE, 1-HEXYL-3-METHYL-	410	567
6	CYCLOHEXANE, 1-(CYCLOHEXYLMETHYL)-4-METHYL-, TRANS-	406	517
7	9-BORABICYCLO 3.3.1 NONANE, 9-HYDROXY-	403	572
8	CYCLOHEXANE, 1-(CYCLOHEXYLMETHYL)-4-METHYL-, CIS-	400	510

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 438, Reverse Fit: 557**
Y1014221 983 (13.243) Cm (983-22410)



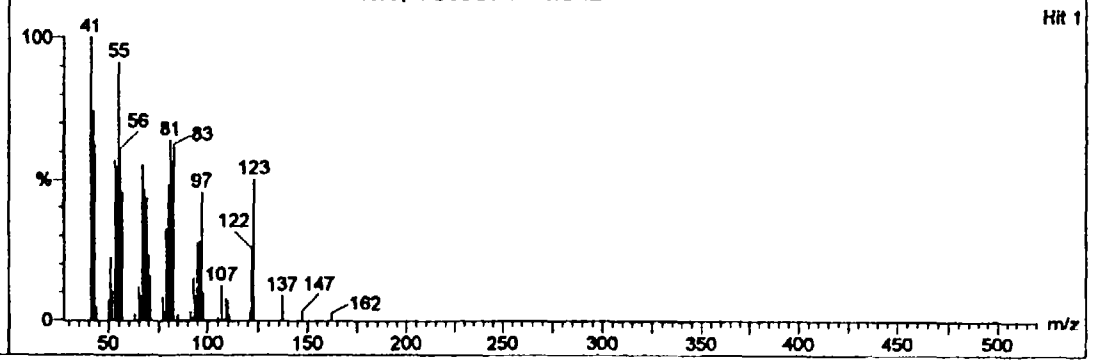
Compound Name: 2,11-DODECANEDIONE
Synonym:
Molecular Weight: 198
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 557
Y1014221 983 (13.243)



F:438 NIST 22410: 2,11-DODECANEDIONE

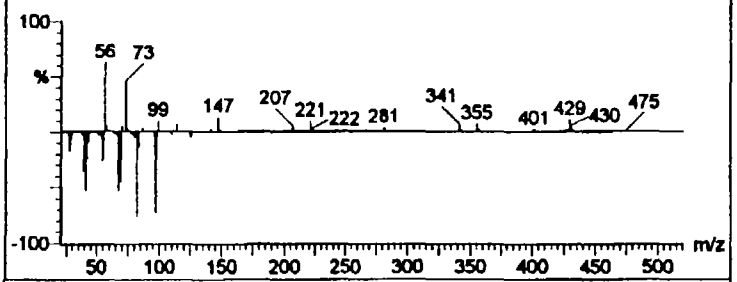
Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

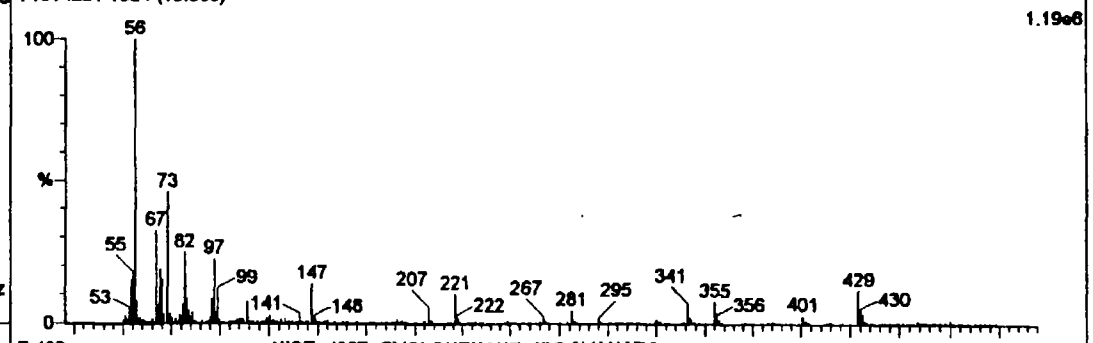
Hit	Compound Name	FOR	rev
1	CYCLOHEXANE, ISOCYANATO-	409	600

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 409, Reverse Fit: 600**
Y1014221 1024 (13.585) Cm (1024-4337)

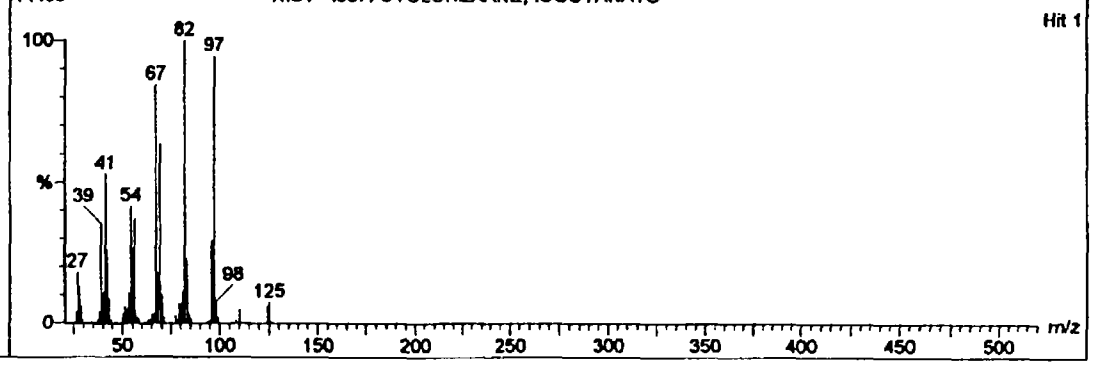


Compound Name: CYCLOHEXANE, ISOCYANATO-
Synonym:
Molecular Weight: 125
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 600
Y1014221 1024 (13.585)



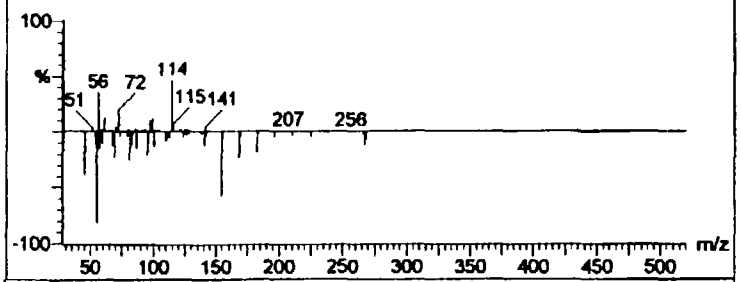
NIST 4337: CYCLOHEXANE, ISOCYANATO-



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

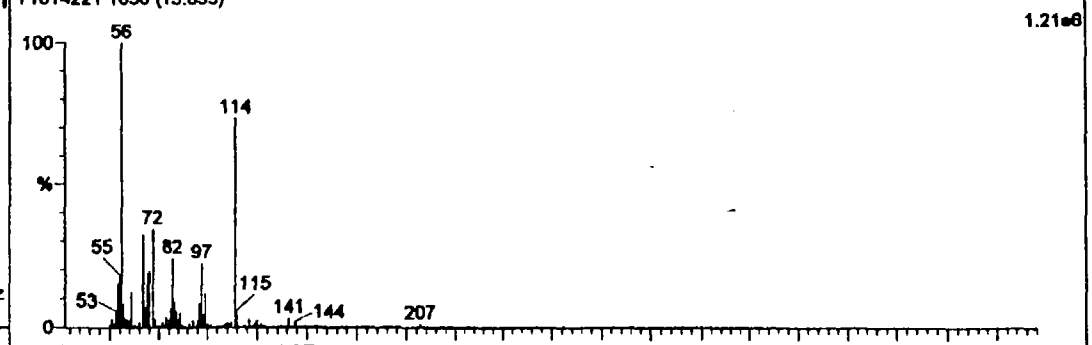
Hit	Compound Name	FOR	rev
1	9-OCTADECEN-1-AMINE	527	601
2	CYCLOPROPANEACETIC ACID, 2-HEXYL-	453	531
3	1-DOTRIACONTANOL	441	624
4	(1R,2R)-(-)-1,2-DIAMINOCYCLOHEXANE	435	487
5	13-OXABICYCLO 10.1.0 TRIDECANE	431	566
6	1,2-CYCLOHEXANEDIAMINE	431	483
7	2-PENTENAMIDE, N-(2-OXO-3-PIPERIDINYL)-	424	515
8	2-PROPENOIC ACID, 3-(DIMETHYLAMINO)-3-(METHYLAMINO)-, METHYL ESTER	424	527
9	2-PROPENOIC ACID, 3-(DIETHYLAMINO)-3-(DIMETHYLAMINO)-, METHYL ESTER	423	534
10	2,3,6-TRIMETHYLHEPT-6-EN-1-OL	420	538
11	CYCLOHEXANE, ISOCYANATO-	416	614
12	CYCLODECANE, METHYL-	409	550
13	(1S,2S)-(+)-1,2-DIAMINOCYCLOHEXANE	404	457
14	CYCLOPENTANE, 1-HEXYL-3-METHYL-	403	583
15	13-OCTADECENAL, (Z)-	400	583

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 527, Reverse Fit: 601**
Y1014221 1030 (13.635) Cm (1030-37212)

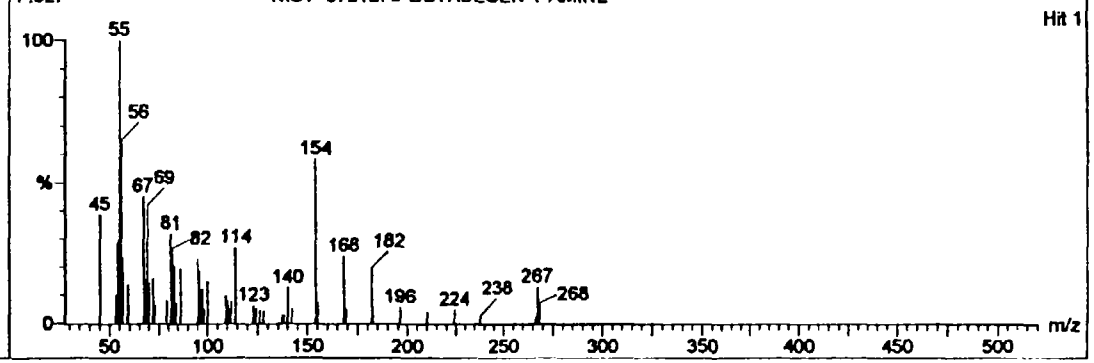


Compound Name: 9-OCTADECEN-1-AMINE
Synonym:
Molecular Weight: 267
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 601
Y1014221 1030 (13.635)



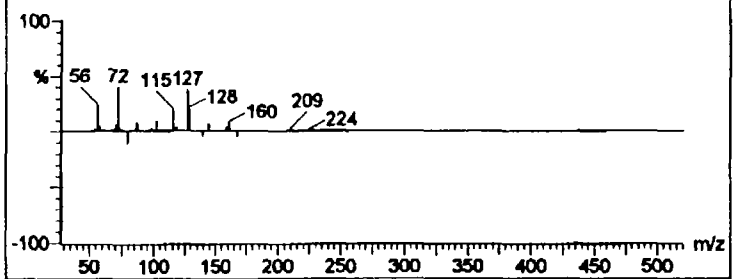
F:527 NIST 37212: 9-OCTADECEN-1-AMINE Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

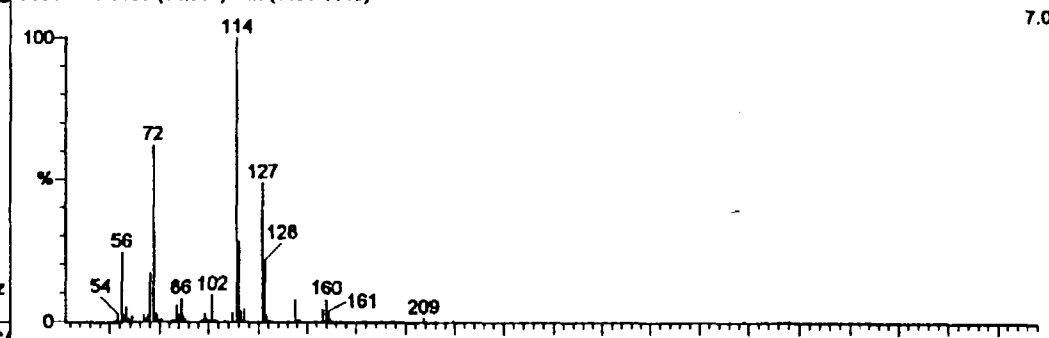
Hit	Compound Name	FOR	rev
1	PHOSPHONOTHIOIC ACID, METHYL-, O- 2- BIS(1-METHYLETHYL)AMINO ETHYL O-ETHYL ESTER	522	808
2	SILACYCLOOCTAN-5-ONE, 1,1-DIMETHYL-	496	558
3	4,6,9,11-TETRAMETHYL-1-OXA-4,6,9,11-TETRAAZACYCLOTRIDECANE-5,10-DIONE	486	536
4	ETHANOL, 2- BIS(1-METHYLETHYL)AMINO -	484	625
5	PROPRANOLOL (INDERAL)	474	572
6	TRIMECRYTON	465	629
7	PHOSPHONIC ACID, METHYL-, 2- BIS(1-METHYLETHYL)AMINO ETHYL ETHYL ESTER	450	715
8	1,1,4,7,10,10-HEXAMETHYLTRIETHYLENETETRAMINE	447	494
9	OXAZOLIDINE, 3-ETHYL-2,2-DIMETHYL-	440	602
10	2-PROPENOIC ACID, 3-(DIMETHYLAMINO)-3-(METHYLAMINO)-, METHYL ESTER	417	495
11	3-(DIISOPROPYLAMINO)-1,2-PROPANEDIOL	409	544

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 522, Reverse Fit: 808**
Y1014221 1193 (14.994) Cm (1-37113); Cm (1193-1113)

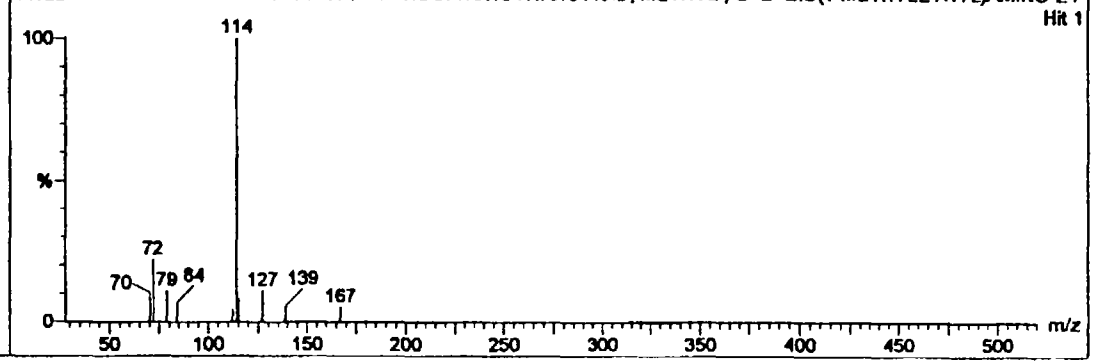


Compound Name: PHOSPHONOTHIOIC ACID, METHYL-, O- 2- BIS(
Synonym:
Molecular Weight: 267
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 808
Y1014221 1193 (14.994) Cm (1193-1113)



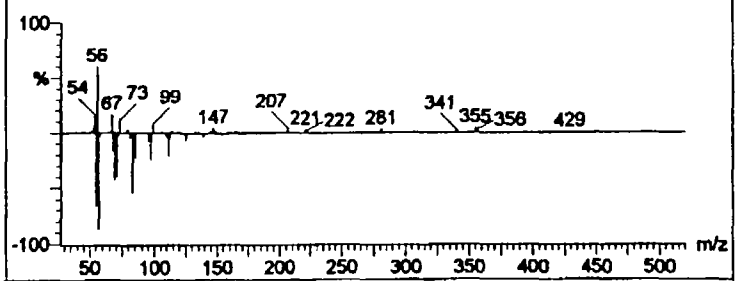
F:522 NIST 37113: PHOSPHONOTHIOIC ACID, METHYL-, O- 2- BIS(1-METHYLETHYL)AMINO ET
Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

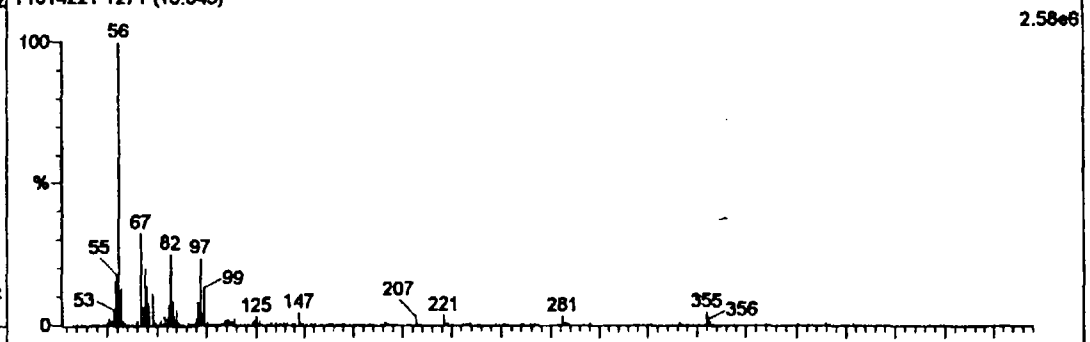
Hit	Compound Name	FOR	rev
1	1-DOTRIACONTANOL	596	722
2	ISOHEPTADECANOL	534	623
3	CYCLOHEXANE, ISOCYANATO-	531	607
4	9-TRICOSENE, (Z)-	529	598
5	CYCLOPROPANEACETIC ACID, 2-HEXYL-	526	575
6	CYCLOPENTANE, 1-HEXYL-3-METHYL-	521	591
7	1-HENEICOSYL FORMATE	520	606
8	CYCLOPENTANE, 1,2-DIBUTYL-	520	585
9	1-HEXADECANOL	516	571
10	7-TETRADECENE	516	584
11	CYCLODECANE, METHYL-	516	598
12	13-OCTADECENAL, (Z)-	515	583
13	CYCLOTETRADECANE	515	586
14	1-METHYL-2-(4-METHYLPENTYL)CYCLOPENTANE	514	600
15	TRANS-1-METHYL-2-(4-METHYLPENTYL)CYCLOPENTANE	514	600
16	CIS-2-METHYL-7-OCTADECENE	508	576
17	CYCLOPENTADECANE	507	576
18	1-PENTADECANOL	505	580
19	1-HEPTADECANOL	504	579
20	CYCLOPENTANE, 1-PENTYL-2-PROPYL-	504	567

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 596, Reverse Fit: 722**
Y1014221 1271 (15.645) Cm (1271-57795)

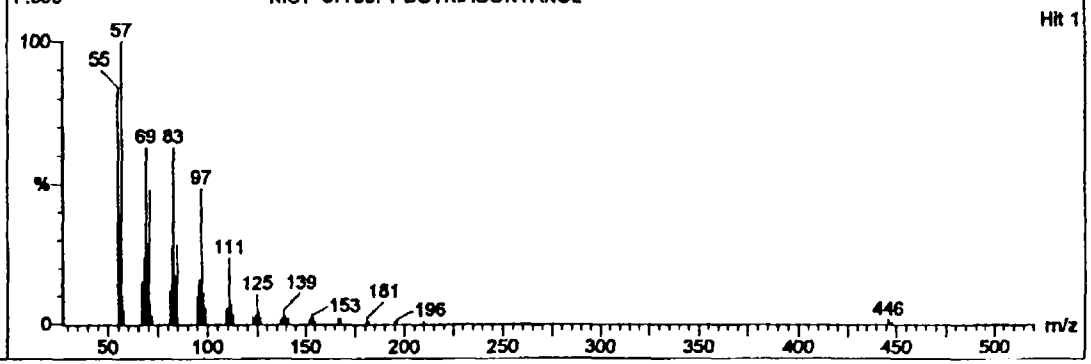


Compound Name: 1-DOTRIACONTANOL
Synonym:
Molecular Weight: 466
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 722
Y1014221 1271 (15.645)



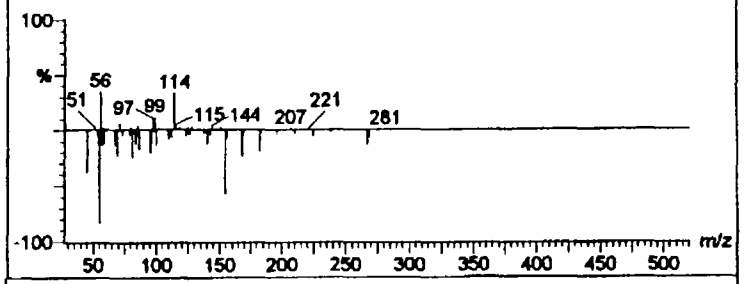
F:596 NIST 57795: 1-DOTRIACONTANOL — Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

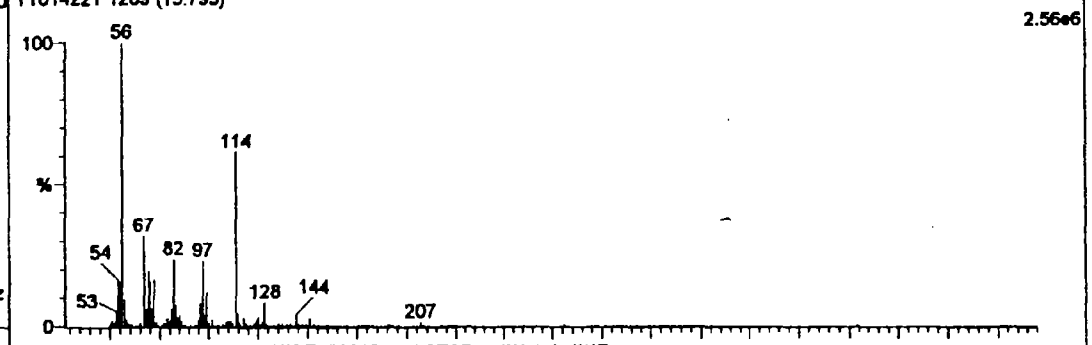
Hit	Compound Name	FOR	rev
1	9-OCTADECEN-1-AMINE	525	600
2	1-DOTRIACONTANOL	507	696
3	CYCLOPROPANEACETIC ACID, 2-HEXYL-	476	535
4	CYCLOHEXANE, ISOCYANATO-	459	618
5	(1R,2R)-(-)-1,2-DIAMINOCYCLOHEXANE	459	517
6	7-TETRADECENE	447	584
7	2-PROPENOIC ACID, 3-(DIETHYLAMINO)-3-(DIMETHYLAMINO)-, METHYL ESTER	445	552
8	2,3,6-TRIMETHYLHEPT-6-EN-1-OL	445	565
9	CYCLODECANE, METHYL-	445	581
10	1,2-CYCLOHEXANEDIAMINE	445	505
11	CYCLOPENTANE, 1-HEXYL-3-METHYL-	439	588
12	CYCLOPENTANE, 1,2-DIBUTYL-	437	583
13	1-METHYL-2-(4-METHYLPENTYL)CYCLOPENTANE	435	600
14	TRANS-1-METHYL-2-(4-METHYLPENTYL)CYCLOPENTANE	435	599
15	1-HEXADECANOL	433	559
16	13-OXABICYCLO 10.1.0 TRIDECANE	433	568
17	ISOHEPTADECANOL	432	606
18	CYCLOPENTADECANE	432	557
19	CYCLOTETRADECANE	432	583
20	9-TRICOSENE, (Z)-	430	583

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 525, Reverse Fit: 600**
Y1014221 1289 (15.795) Cm (1289-37212)

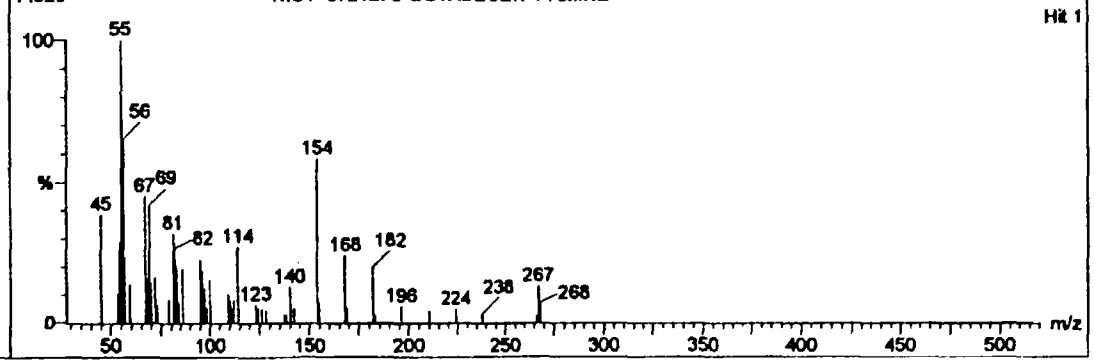


Compound Name: 9-OCTADECEN-1-AMINE
Synonym:
Molecular Weight: 267
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 600
Y1014221 1289 (15.795)



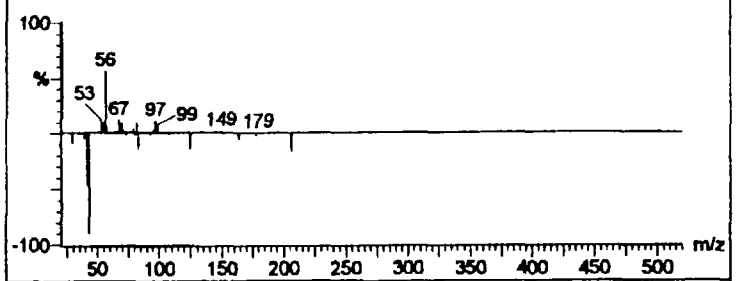
F:525 NIST 37212: 9-OCTADECEN-1-AMINE Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

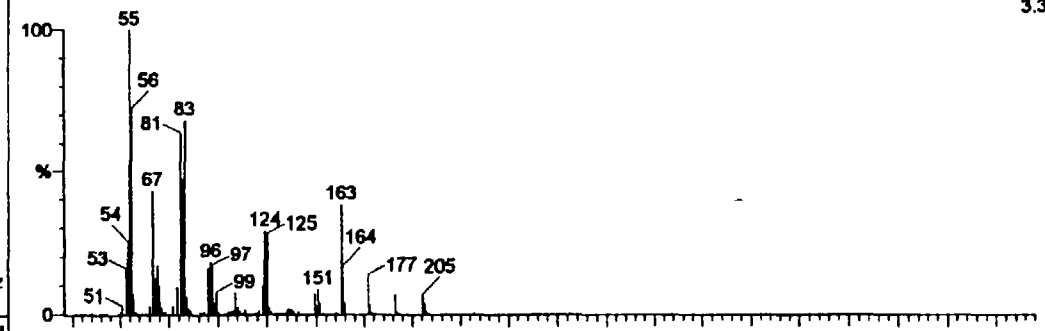
Hit	Compound Name	FOR	rev
1	CYCLOHEXANAMINE, N,N'-METHANETETRAYLBIS-	717	755
2	CYCLOHEXANE, (2,2-DIMETHYLCYCLOPENTYL)-	638	697
3	1,1'-BICYCLOHEXYL, 2-PROPYL-, TRANS-	576	669
4	1,1'-BICYCLOHEXYL, 2-PROPYL-, CIS-	572	665
5	1,1'-BICYCLOHEXYL, 2-(1-METHYLETHYL)-, TRANS-	553	648
6	2-CYCLOHEXEN-1-ONE, 4,5-DIMETHYL-	549	691
7	CYCLOHEXANE, 1,1'-PROPYLIDENEBIS-	547	659
8	1H-INDENE, OCTAHYDRO-	540	647
9	1,1'-BICYCLOHEXYL, 2-(1-METHYLETHYL)-, CIS-	537	631
10	4-CYCLOHEXYL-1-BUTANOL	530	703
11	CYCLOHEXANE, 1,1'-(1-METHYL-1,2-ETHANEDIYL)BIS-	526	635
12	1,1'-BICYCLOHEXYL -2-OL, 5-(1,1-DIMETHYLETHYL)-	525	611
13	CIS-9,10-EPOXYOCTADECAN-1-OL	513	629
14	8-METHYLOCTAHYDROCOUMARIN	507	617
15	CYCLOPENTANOL, 3,7-DIMETHYL-	496	676
16	9-BORABICYCLO 3.3.1 NONANE, 9-METHOXY-	492	584
17	1,1'-BICYCLOHEXYL -4-ONE	490	574
18	CYCLOHEXANEHEXANOL	489	619
19	8,8-DIMETHYLSPIRO(4.5)UNDECANE-6,10-DIONE	487	598
20	CYCLOHEXANE, 1,1'-(1-METHYLETHYLIDENE)BIS-	486	590

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 717, Reverse Fit: 755**
Y1014221 1157 (14.694) Cm (1157-24388)

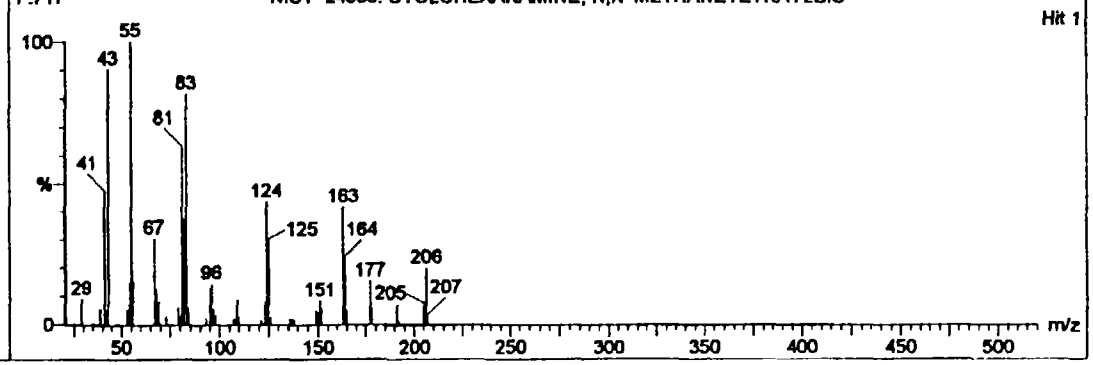


Compound Name: CYCLOHEXANAMINE, N,N'-METHANETETRAYL
Synonym:
Molecular Weight: 206
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 755
Y1014221 1157 (14.694) 3.32e6



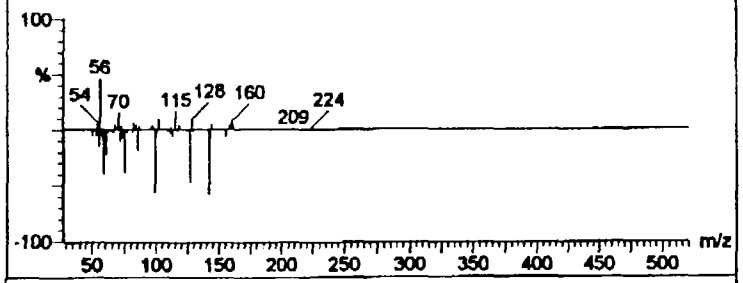
F:717 NIST 24388: CYCLOHEXANAMINE, N,N'-METHANETETRAYLBIS- Hit 1



Sample ID: 2uL Tray 153 R1 — **Acquired 18:15:04 at 18:15:04**

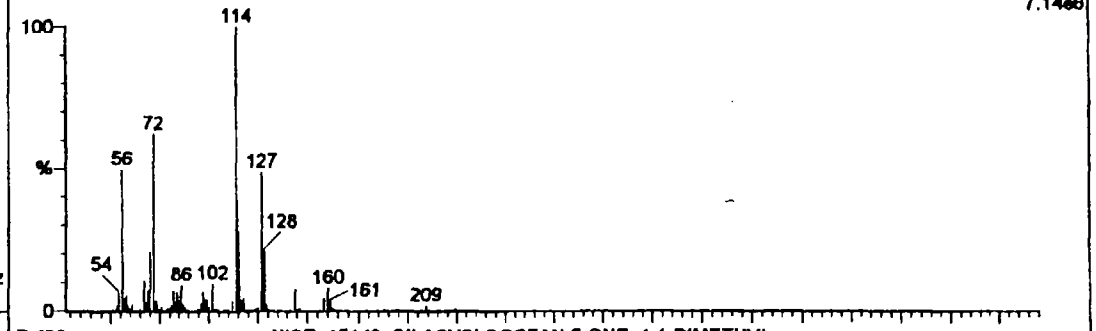
Hit	Compound Name	FOR	rev
1	SILACYCLOOCTAN-5-ONE, 1,1-DIMETHYL-	488	556
2	4,6,9,11-TETRAMETHYL-1-OXA-4,6,9,11-TETRAAZACYCLOTRIDECANE-5,10-DIONE	480	568
3	PHOSPHONOTHIOIC ACID, METHYL-, O- 2- BIS(1-METHYLETHYL)AMINO ETHYL O-ETHYL ESTER	462	833
4	2-PROPENOIC ACID, 3-(DIMETHYLAMINO)-3-(METHYLAMINO)-, METHYL ESTER	458	546
5	ETHANOL, 2- BIS(1-METHYLETHYL)AMINO -	451	592
6	TRIMECRYTON	444	646
7	PROPRANOLOL (INDERAL)	439	574
8	1,1,4,7,10,10-HEXAMETHYLTRIETHYLENETETRAMINE	436	490
9	OXAZOLIDINE, 3-ETHYL-2,2-DIMETHYL-	421	592

Sample ID: 2uL Tray 153 R1
Acquired: 14-Oct-2002 at 18:15:04
Library: NIST **Forward Fit: 488, Reverse Fit: 556**
Y1014221 1193 (14.994) Cm (1193-15148)

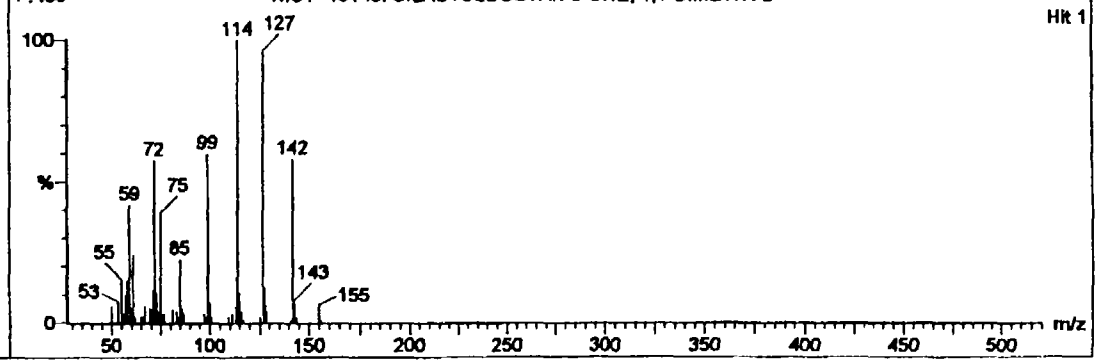


Compound Name: SILACYCLOOCTAN-5-ONE, 1,1-DIMETHYL-
Synonym:
Molecular Weight: 170
structure fou

Sample ID: 2uL Tray 153 R1 — **Acquired on 14-Oct-2002 at 18:15:04**
Reverse fit factor [REV]: 556
Y1014221 1193 (14.994) 7.14e6



F:488 NIST 15148: SILACYCLOOCTAN-5-ONE, 1,1-DIMETHYL- Hit 1



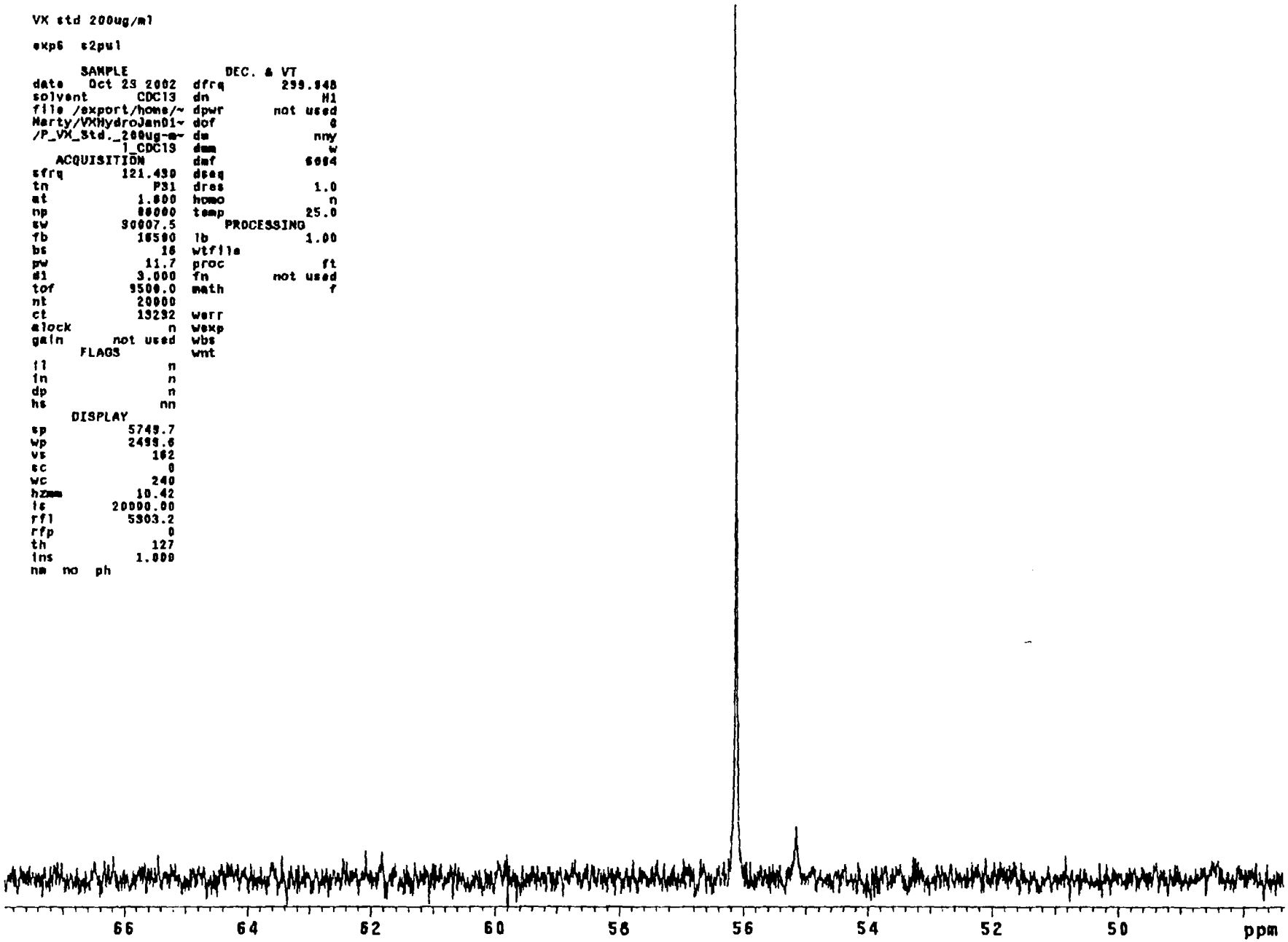
APPENDIX F

NMR DATA

VX std 200ug/ml

exp6 s2pul

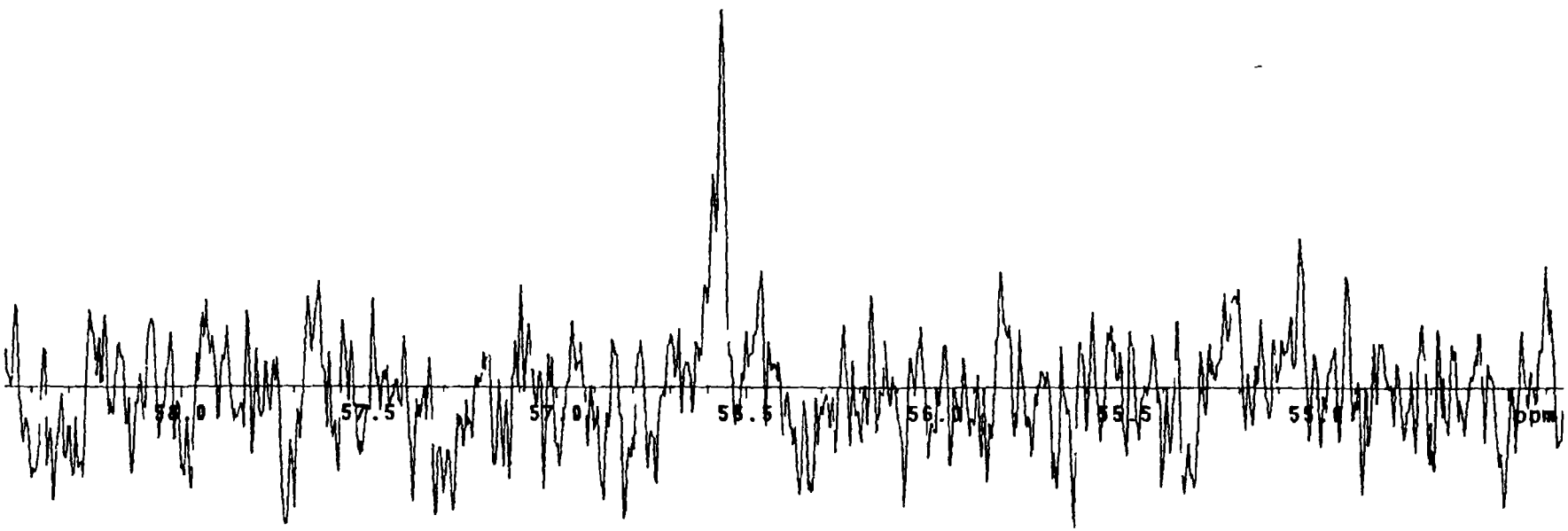
SAMPLE		DEC. & VT	
date	Oct 23 2002	dfrq	299.948
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	not used
	Marty/VXHydroJan01~	dof	0
	/P_VX_Std._200ug-m~	dm	nny
	1_CDCl3	dmm	w
ACQUISITION		dmf	6064
sfrq	121.430	dseq	
tn	P31	dres	1.0
at	1.800	homo	n
np	80000	temp	25.0
sw	30007.5	PROCESSING	
fb	16500	lb	1.00
bs	16	wtfile	
pw	11.7	proc	ft
d1	3.000	fn	not used
tof	9500.0	math	f
nt	20000		
ct	13292	werr	
alock	n	wexp	
gain	not used	wbs	
FLAGS		wnt	
il	n		
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	5749.7		
wp	2499.6		
vs	162		
sc	0		
wc	240		
hzmm	10.42		
is	20000.00		
rfl	5303.2		
rfp	0		
th	127		
ins	1.000		
nm no ph			



Extraction of 4 g of Ash

exp5 s2pul

SAMPLE		DEC. & VT	
date	Oct 24 2002	dfrq	299.948
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	not used
Marty/VXHydroJan01~		dof	0
/P_Ash_Extract_4g		dm	nny
ACQUISITION		dmm	w
sfrq	121.430	dmf	8094
tn	P31	dseq	
at	1.800	dres	1.0
np	86000	homo	n
sw	30007.5	temp	25.0
fb	16500	PROCESSING	
bs	16	lb	1.00
pw	11.7	wtfile	
d1	3.000	proc	ft
tof	9500.0	fn	not used
nt	20000	math	f
ct	13568		
alock	n	werr	
gain	not used	wexp	
FLAGS		wbs	
il	n	wnt	
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	8800.0		
wp	499.6		
vs	162		
sc	0		
wc	240		
hzmm	2.08		
is	354.40		
rfl	5304.1		
rfp	0		
th	20		
ins	1.000		
nm no ph			

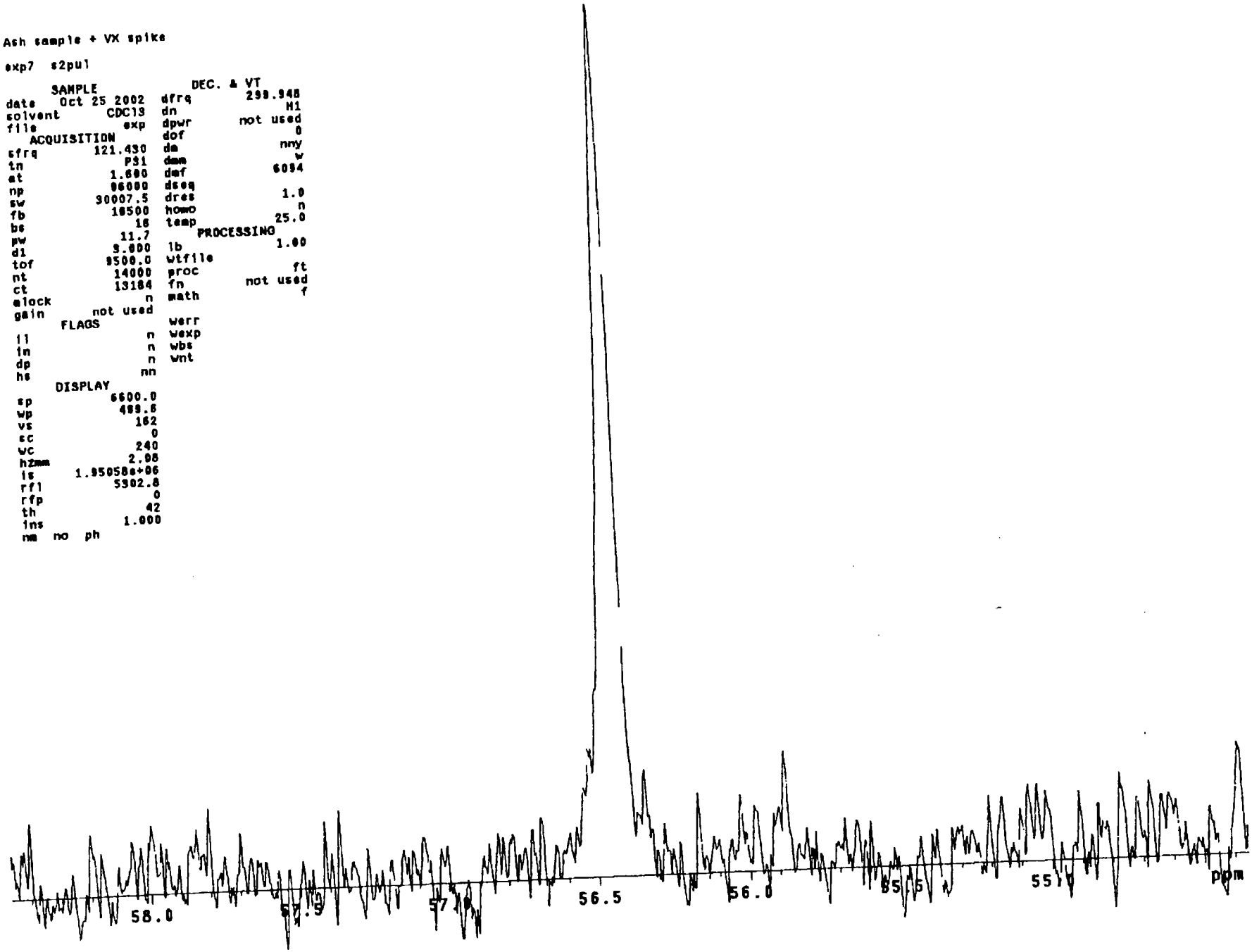


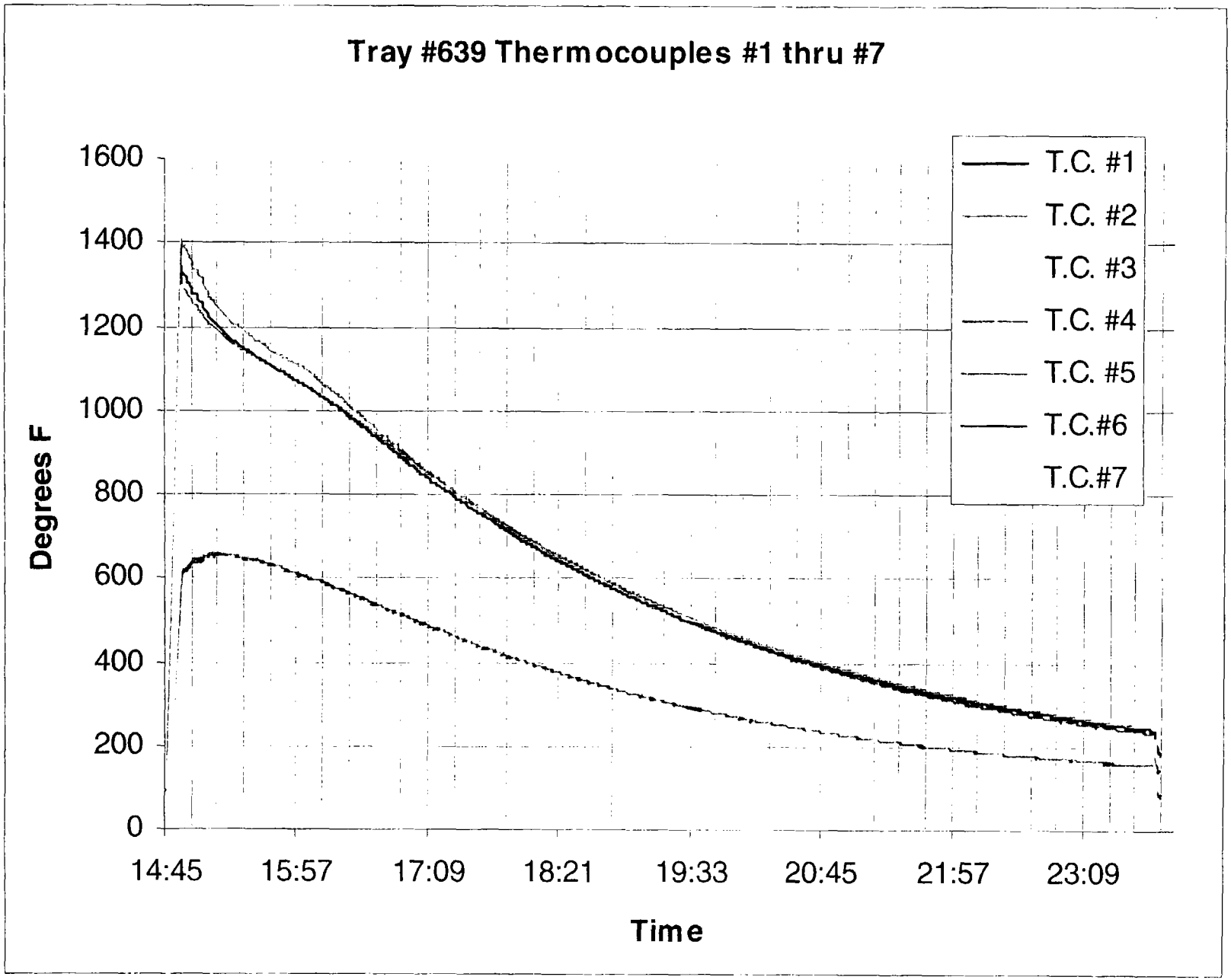
Ash sample + VX spike

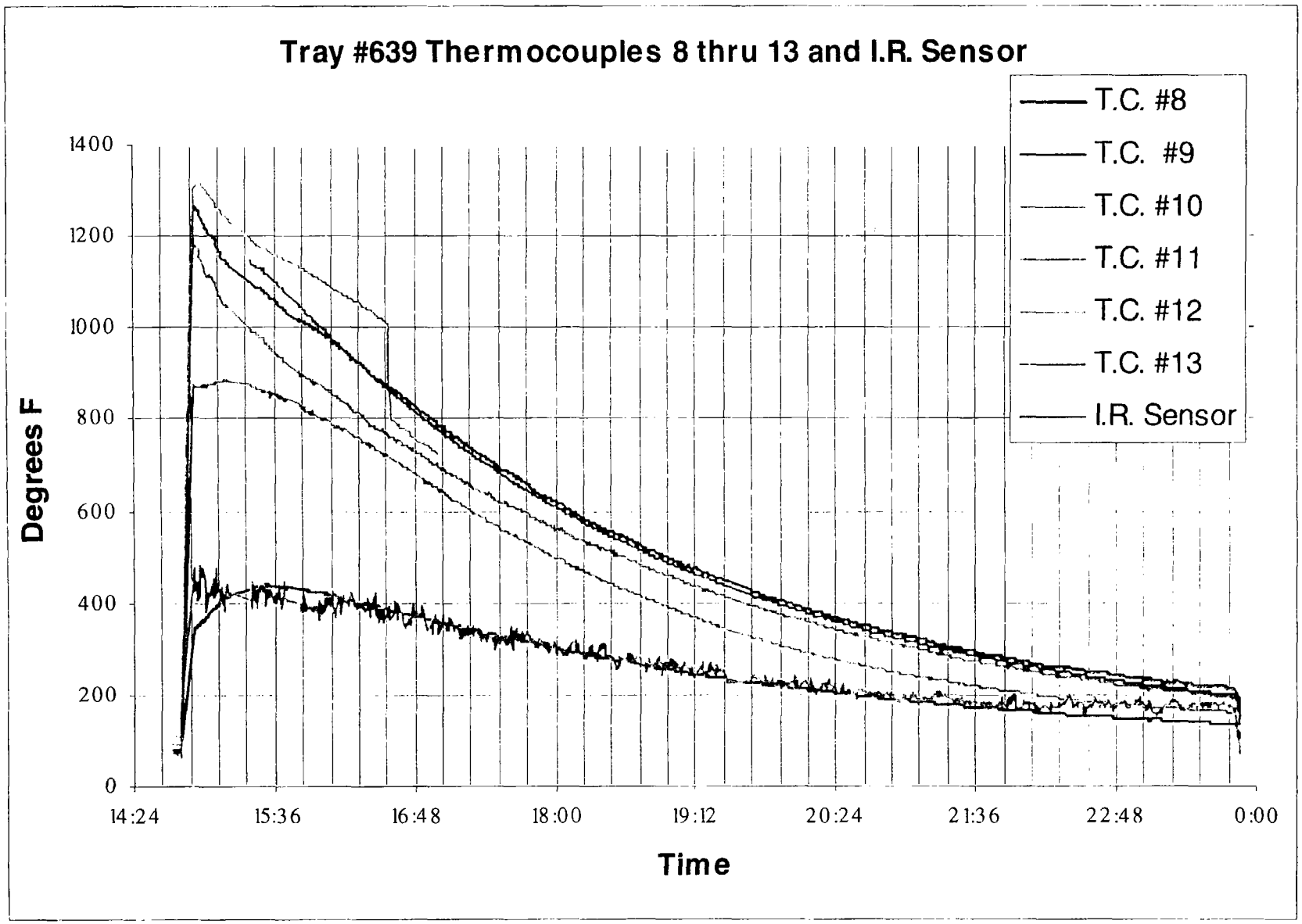
exp7 s2pul

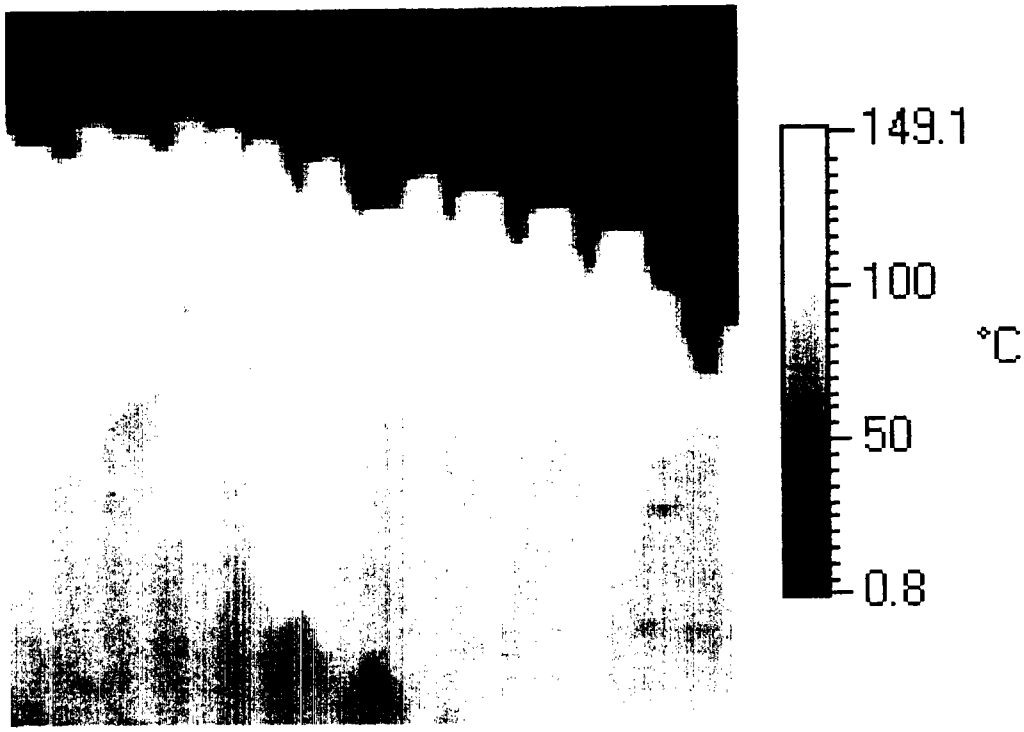
SAMPLE	
date	Oct 25 2002
solvent	CDCl3
file	exp
ACQUISITION	
sfrq	121.430
tn	P31
at	1.600
np	96000
sw	30007.5
fb	18500
bs	16
pw	11.7
d1	9.000
tof	9500.0
nt	14000
ct	13184
alock	n
gain	not used
FLAGS	
il	n
in	n
dp	n
hs	nn
DISPLAY	
sp	6600.0
wp	499.6
vs	162
sc	0
wc	240
hzmm	2.98
is	1.95058e+06
rfl	5302.8
rfp	0
th	42
ins	1.000
nm	no ph

DEC. & VT	
dfrq	299.948
dn	H1
dpwr	not used
dof	0
dm	nny
dmm	w
def	6094
dseq	
dres	1.0
homo	n
temp	25.0
PROCESSING	
lb	1.00
wtfile	
proc	ft
fn	not used
math	f
werr	
wexp	
wbs	
wnt	

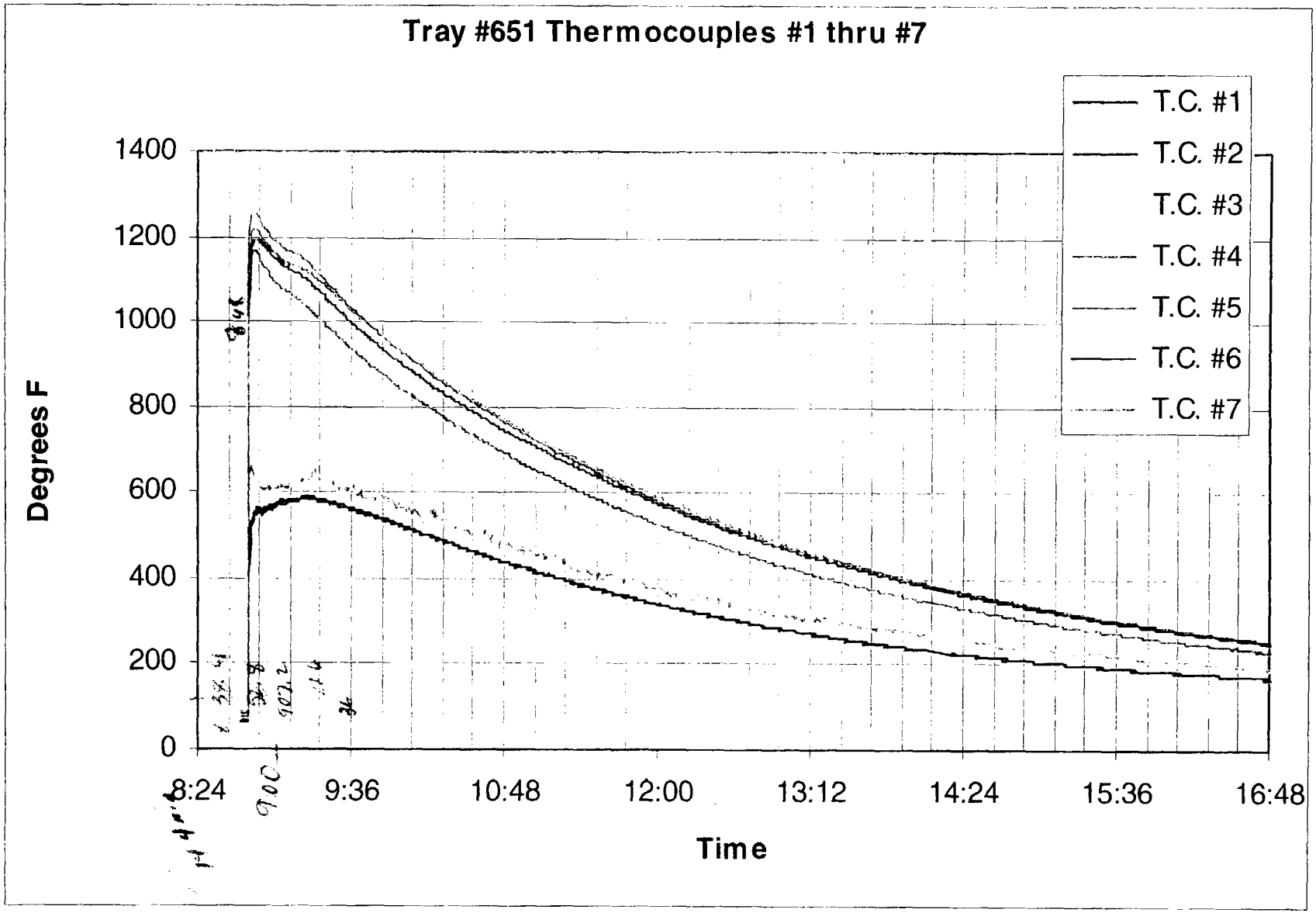


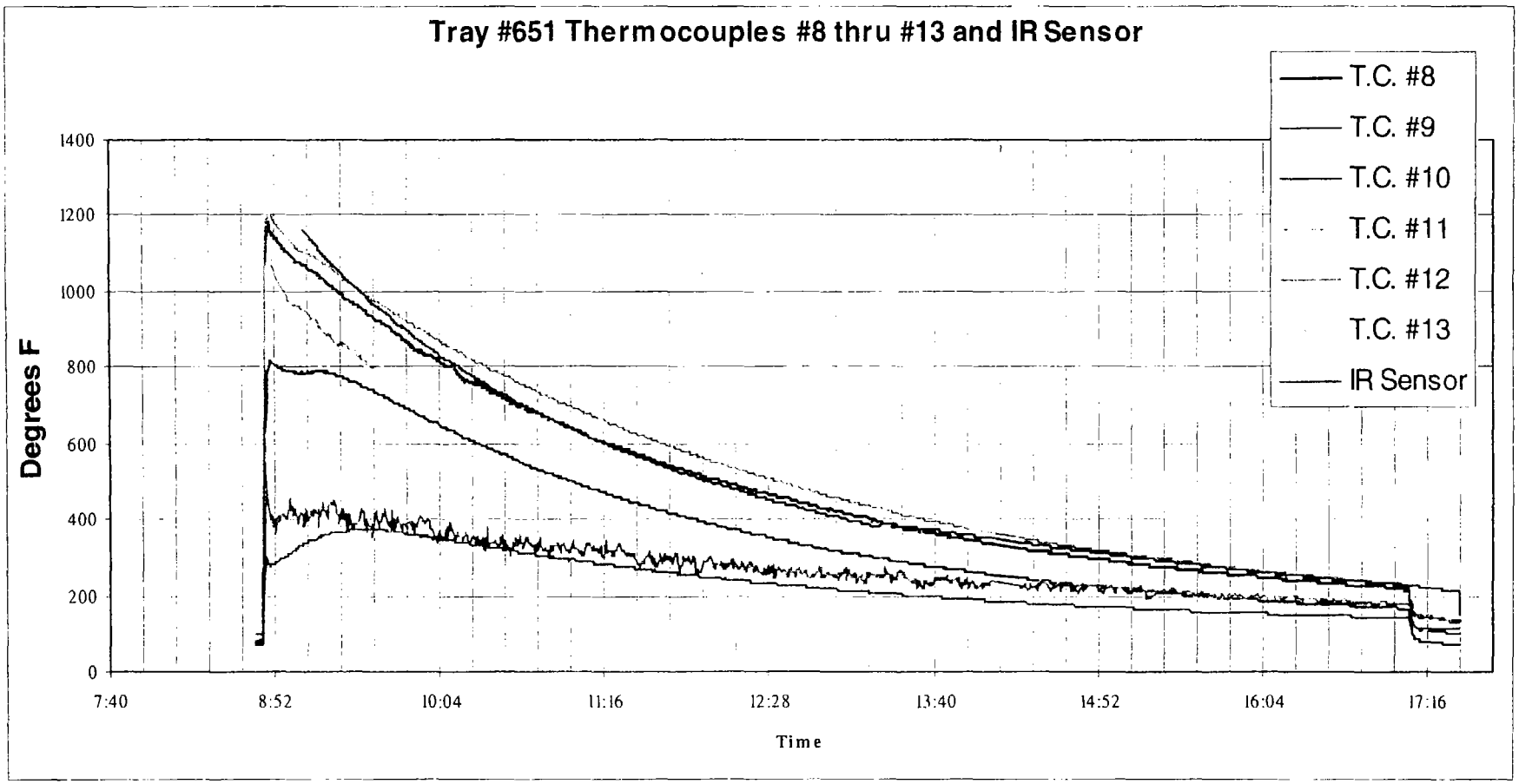

Tray #639 Thermocouples #1 thru #7
Degrees F
1600
1400
1200
1000
800
600
400
200
0
14:45
15:57
17:09
18:21
19:33
20:45
21:57
23:09
Time
T.C. #1
T.C. #2
T.C. #3
T.C. #4
T.C. #5
T.C.#6
T.C.#7

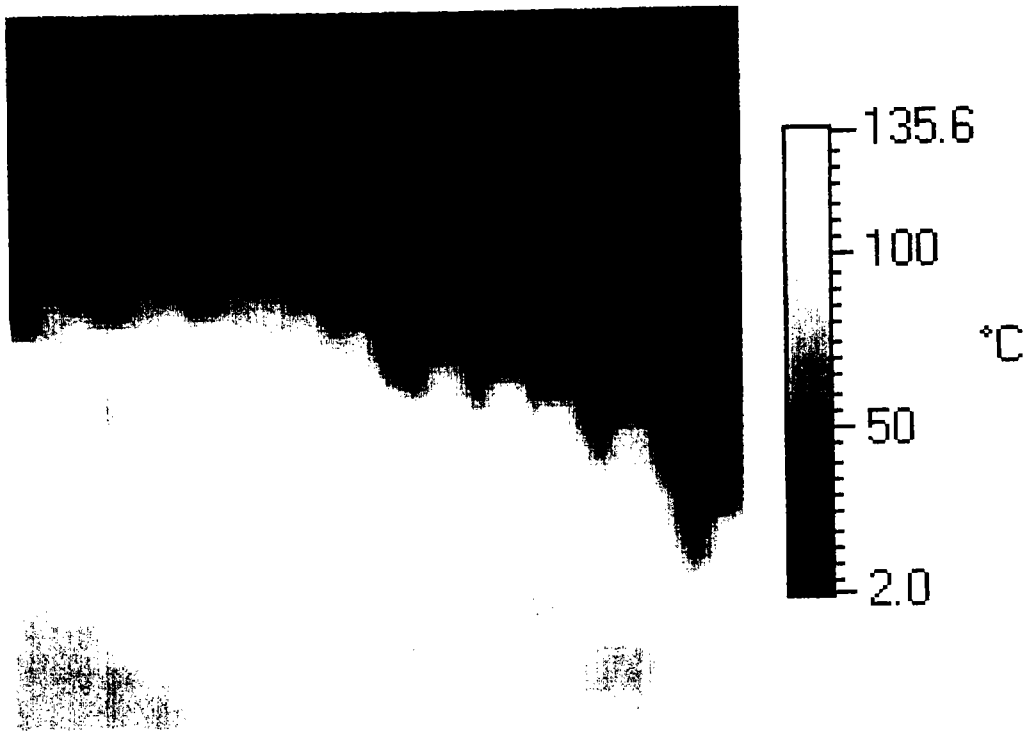

Tray #639 Thermocouples 8 thru 13 and I.R. Sensor
T.C. #8
T.C. #9
T.C. #10
T.C. #11
T.C. #12
T.C. #13
I.R. Sensor
Degrees F
1400
1200
1000
800
600
400
200
0
14:24
15:36
16:48
18:00
19:12
20:24
21:36
22:48
0:00
Time



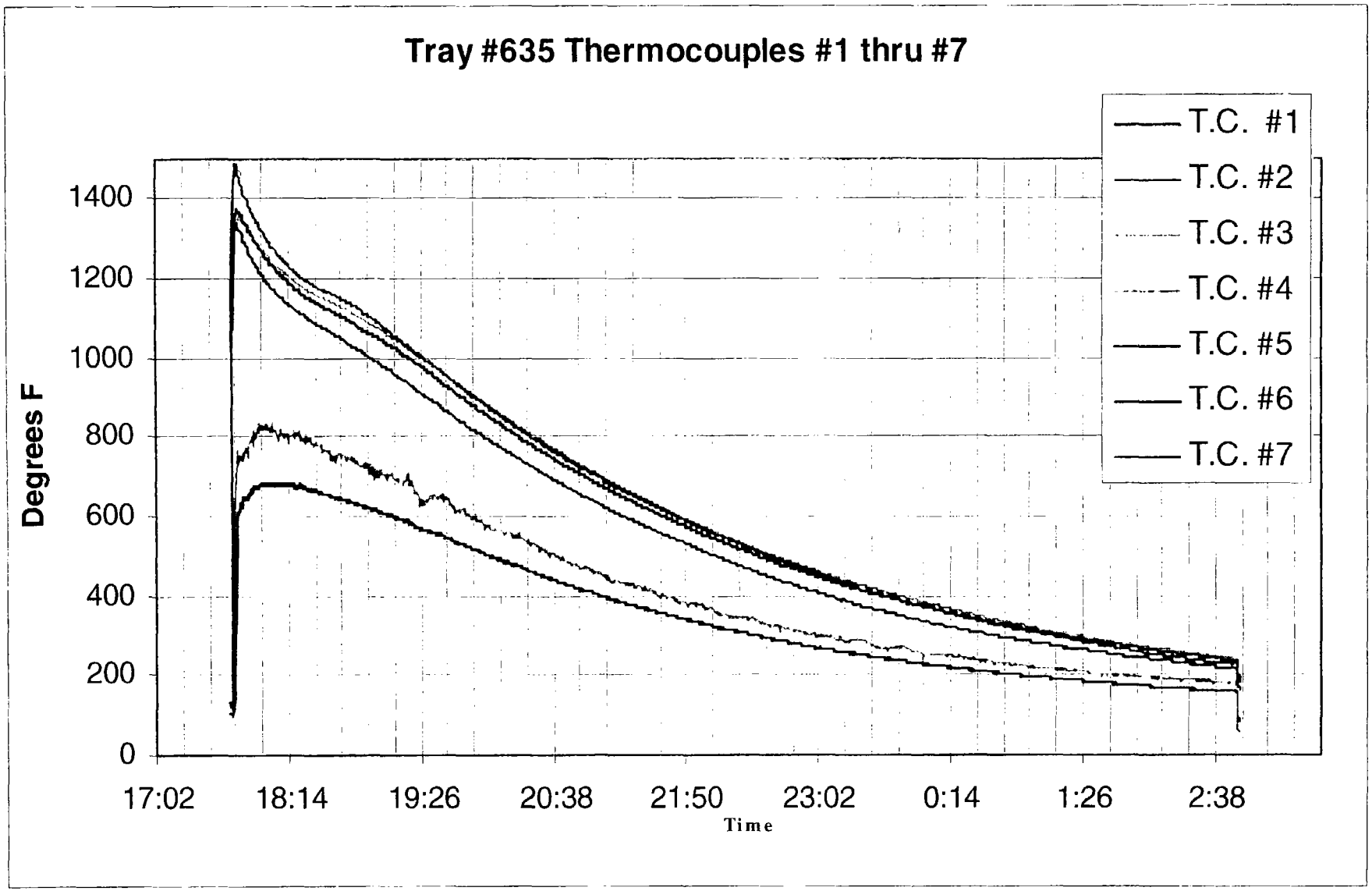
Name: C:\SNAPVIEW\ir0006.isi
Date: Thursday, January 09, 2003
Time: 1:02:39 AM
Location: Tray #639
Target:
User:
Comments:

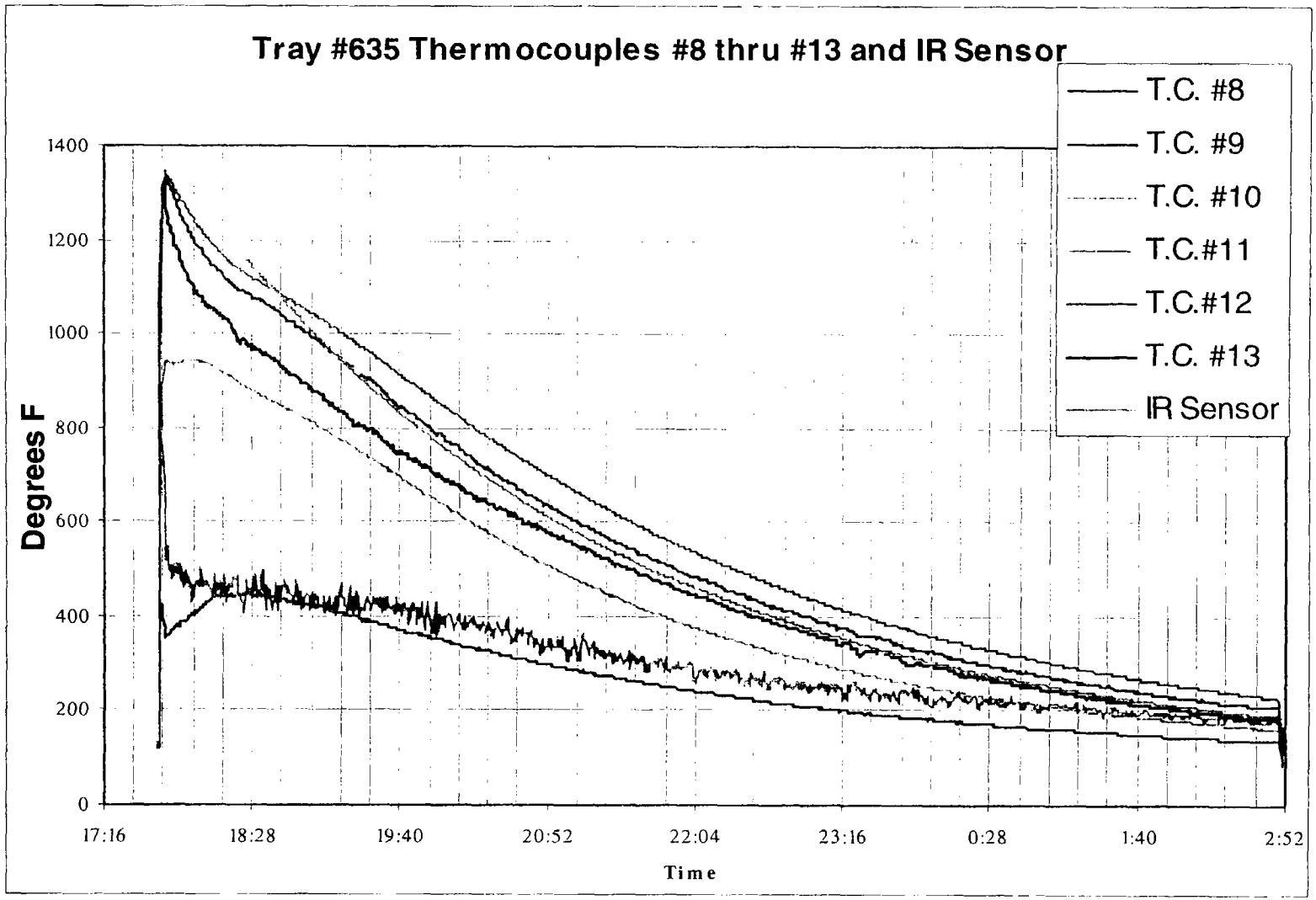

Tray #651 Thermocouples #1 thru #7
T.C. #1
T.C. #2
T.C. #3
T.C. #4
T.C. #5
T.C. #6
T.C. #7
Degrees F
1400
1200
1000
800
600
400
200
0
8:24
9:36
10:48
12:00
13:12
14:24
15:36
16:48
Time


Tray #651 Thermocouples #8 thru #13 and IR Sensor
T.C. #8
T.C. #9
T.C. #10
T.C. #11
T.C. #12
T.C. #13
IR Sensor
Degrees F
1400
1200
1000
800
600
400
200
0
7:40
8:52
10:04
11:16
12:28
13:40
14:52
16:04
17:16
Time



Name: C:\SNAPVIEW\ir0020.isi
Date: Thursday, January 09, 2003
Time: 6:42:32 PM
Location: Tray #651
Target:
User:
Comments:


Tray #635 Thermocouples #1 thru #7
T.C. #1
T.C. #2
T.C. #3
T.C. #4
T.C. #5
T.C. #6
T.C. #7
Degrees F
1400
1200
1000
800
600
400
200
0
17:02
18:14
19:26
20:38
21:50
23:02
0:14
1:26
2:38
Time


Tray #635 Thermocouples #8 thru #13 and IR Sensor
T.C. #8
T.C. #9
T.C. #10
T.C.#11
T.C.#12
T.C. #13
IR Sensor
Degrees F
1400
1200
1000
800
600
400
200
0
17:16
18:28
19:40
20:52
22:04
23:16
0:28
1:40
2:52
Time

F

APPENDIX F

TOCDF Testing with 155 SETH Projectiles

Metal Parts Furnace Discharge Airlock Thermal Test (cooling rate) Report

Prepared by Larry Davis

Feb. 21, 2003

MPF Discharge Airlock Thermal Test (cooling rate) Report

Introduction

Following the MPF Discharge Airlock Agent Sampling Incident at Johnston Island, concerns have been expressed that high air temperatures in the MPF Discharge Airlock (DAL) do not allow for proper sampling of Chemical Agents. Temperatures in the Discharge Airlock and at sample point locations in the air duct exiting the Discharge Airlock were unknown prior to this test. To properly determine these temperatures, it was necessary to install temporary thermocouples capable of measuring the temperatures in these areas of concern.

Three munition trays containing 48 Seth projectiles (155mm) were used for this test. Trays with 48 projectiles (155mm) presented the worst case scenario for cool down rates in the Discharge Airlock at TOCDF because this load has the highest metal mass (6894 lbs) and is processed at the highest temperatures (1600°F) in the MPF Primary Chamber. The 155mm projectile tray has 12 rows of projectiles with 4 projectiles on each row. The Test Trays were processed through Primary Chamber of the Metal Parts Furnace at projectile processing temperatures (1600°F) prior to entering the Discharge Airlock. Temperature data was then gathered while hot trays were in the Discharge Airlock.

Materials and Methods

A hand held optical pyrometer was used to measure DAL skin temperatures. One infrared sensor and a total of 13 type K thermocouples were used to measure temperatures within the DAL and DAL Exhaust Duct. The thermocouples and infrared sensor were wired into a Data Logger and the data was downloaded to Lap Top Computer. The locations of the thermocouples and infrared sensor are shown on attached Sketch MPF-TC-124-1 rev 1.

Results and Discussion

The Test was started on 1-8-03 and completed on 1-10-03. Thermocouples #4 and #5 were of the greatest interest because they indicated the highest temperatures and required the longest time to cool to 259°F. Thermocouple #4 was located near the DAL exit door approximately 2 inches above the first row of projectiles. Thermocouple #5 hung down between the projectiles, approximately 5 inches from the top of the projectiles near the 8th row of projectiles. Thermocouple #1 was inserted inside the DAL exhaust duct at the VX sampling probe location, approximately 7 ½ feet up the DAL exhaust duct.

Tray # 639 was the first tray to enter the DAL and was in the primary chamber for 5 hrs and 45min due to conveyor malfunctions. It took thermocouple #1, 1hr 14min to cool to 600°F and 5 hrs 21 min to cool to 259°F. It took thermocouple #4, 3 hrs 56 min to cool to 600°F and 8 hrs 29 min to cool to 259°F. It took thermocouple #5, 3 hrs 52 min to cool to 600°F and 8 hrs 12 min to cool to 259°F. Thermocouple #12 failed

2 hrs and 10 min after tray #639 entered the DAL and was repaired after the tray exited the DAL.

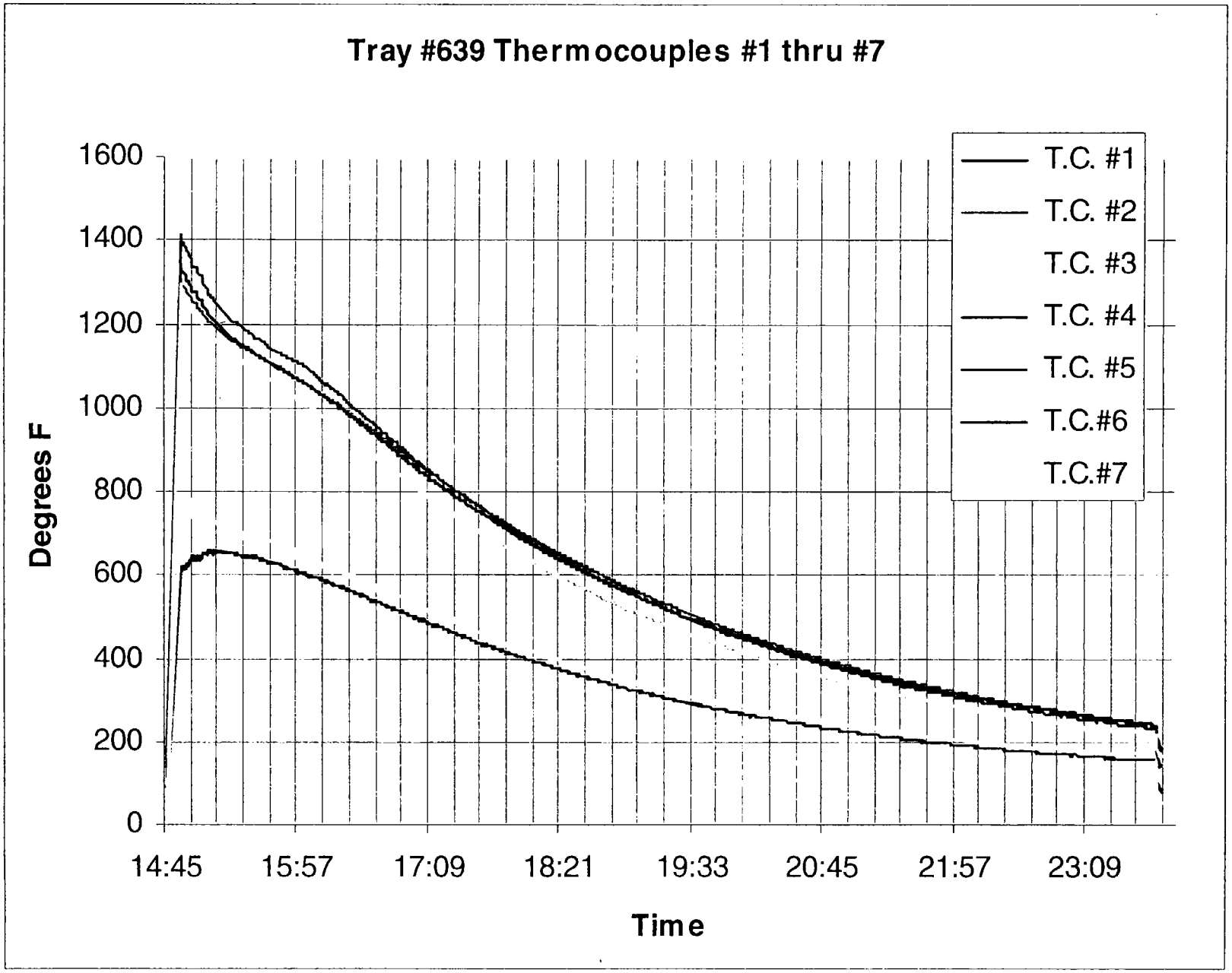
Tray # 651 was the second tray to enter the DAL and was in the primary chamber for 1 hr 15 min. Thermocouple # 1 reached a maximum temperature of 584.3°F and took 4 hrs 37 min to cool to 259°F. It took thermocouple #4, 3 hrs 6 min to cool to 600°F and 7 hrs 48 min to cool to 259°F. It took thermocouple #5, 3 hrs 3 min to cool to 600°F and 7 hrs 38 min to cool to 259°F. Thermocouple #13 failed, 47 min after Tray #651 entered the DAL.

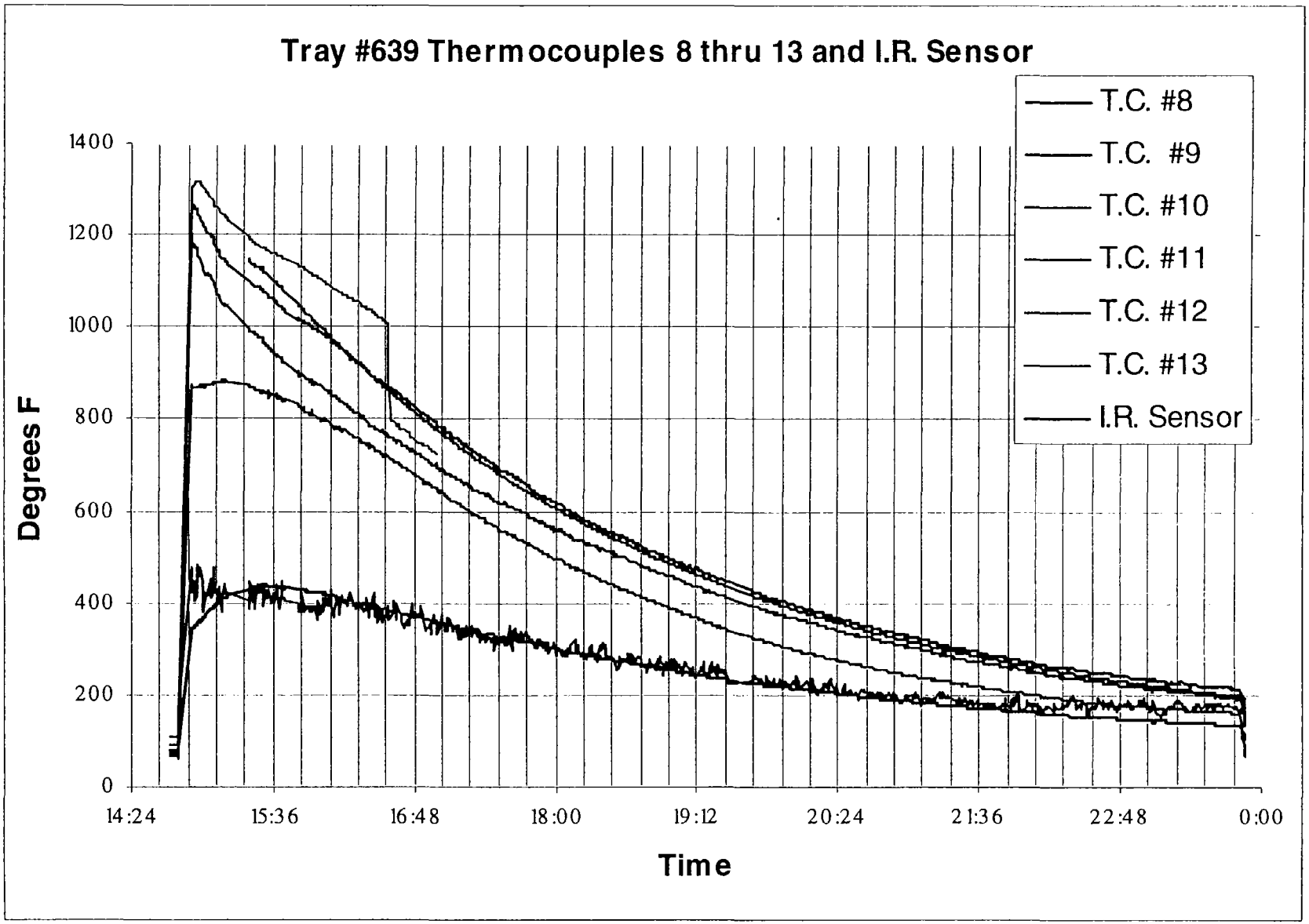
Tray # 635 was the third tray to enter the DAL and was in the primary chamber for 9 hr 37min prior to entering the DAL. Tray #635 entered zone one just after tray #651 entered zone two and remained in the primary chamber until tray # 651 was able to exit the DAL. It took thermocouple #1, 1 hr 26 min to cool to 600°F and 5 hrs 28 min to cool to 259°F. It took thermocouple #4, 4 hrs 2 min to cool to 600°F and 8 hrs 32 min to cool to 259°F. It took thermocouple #5, 4 hrs 0 min to cool to 600°F and 8 hrs 11 min to cool to 259°F.

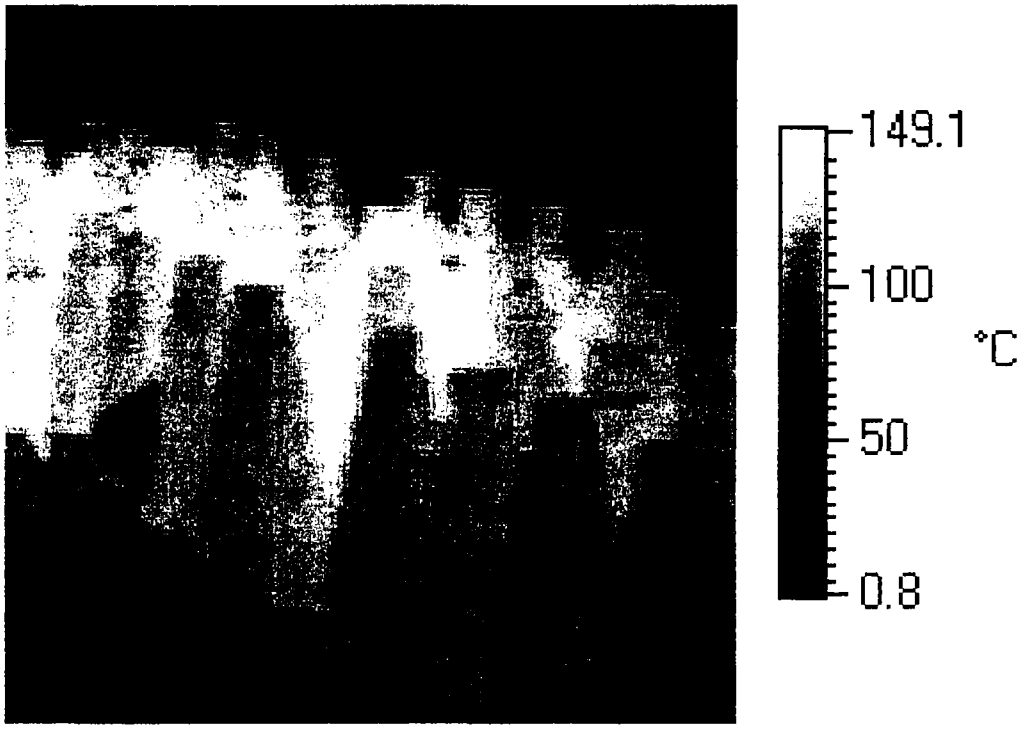
An infrared camera was used to capture thermal images of all three test trays when they exited the Discharge Airlock. Temperatures of projectiles in the center of the trays were approximately 50 °F higher than those projectiles on the outer perimeter of the tray. See attached Infrared photograph of test tray.

Airflow measurements were taken on the inlet air duct to the DAL using a hot wire anemometer for trays #639 and #651. The average inlet airflow for tray #639 was 214.7 SCFM. Two sets of measurements were taken for tray # 651, their averages were 221.2 SCFM and 208.8 SCFM.

The Skin temperatures of the DAL were taken while tray #651 was cooling within the DAL. The highest temperature recorded on the roof of the DAL was 126°F, for the South Wall 131°F was the highest, and for the North Wall 130°F was the highest. The highest Ambient temperature recorded was 60°F.


Tray #639 Thermocouples #1 thru #7
Degrees F
1600
1400
1200
1000
800
600
400
200
0
14:45
15:57
17:09
18:21
19:33
20:45
21:57
23:09
Time
T.C. #1
T.C. #2
T.C. #3
T.C. #4
T.C. #5
T.C.#6
T.C.#7


Tray #639 Thermocouples 8 thru 13 and I.R. Sensor
T.C. #8
T.C. #9
T.C. #10
T.C. #11
T.C. #12
T.C. #13
I.R. Sensor
Degrees F
1400
1200
1000
800
600
400
200
0
14:24
15:36
16:48
18:00
19:12
20:24
21:36
22:48
0:00
Time



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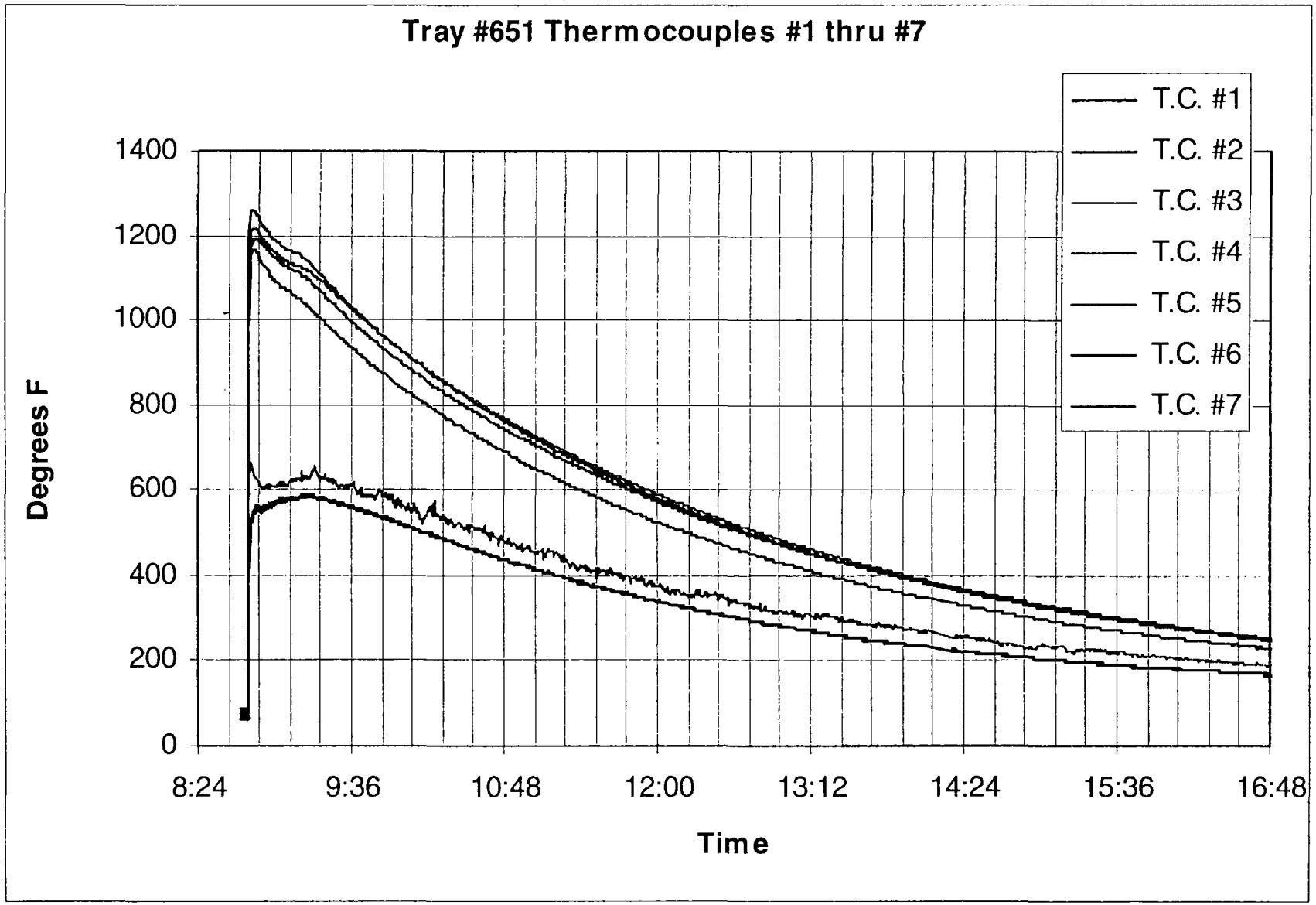
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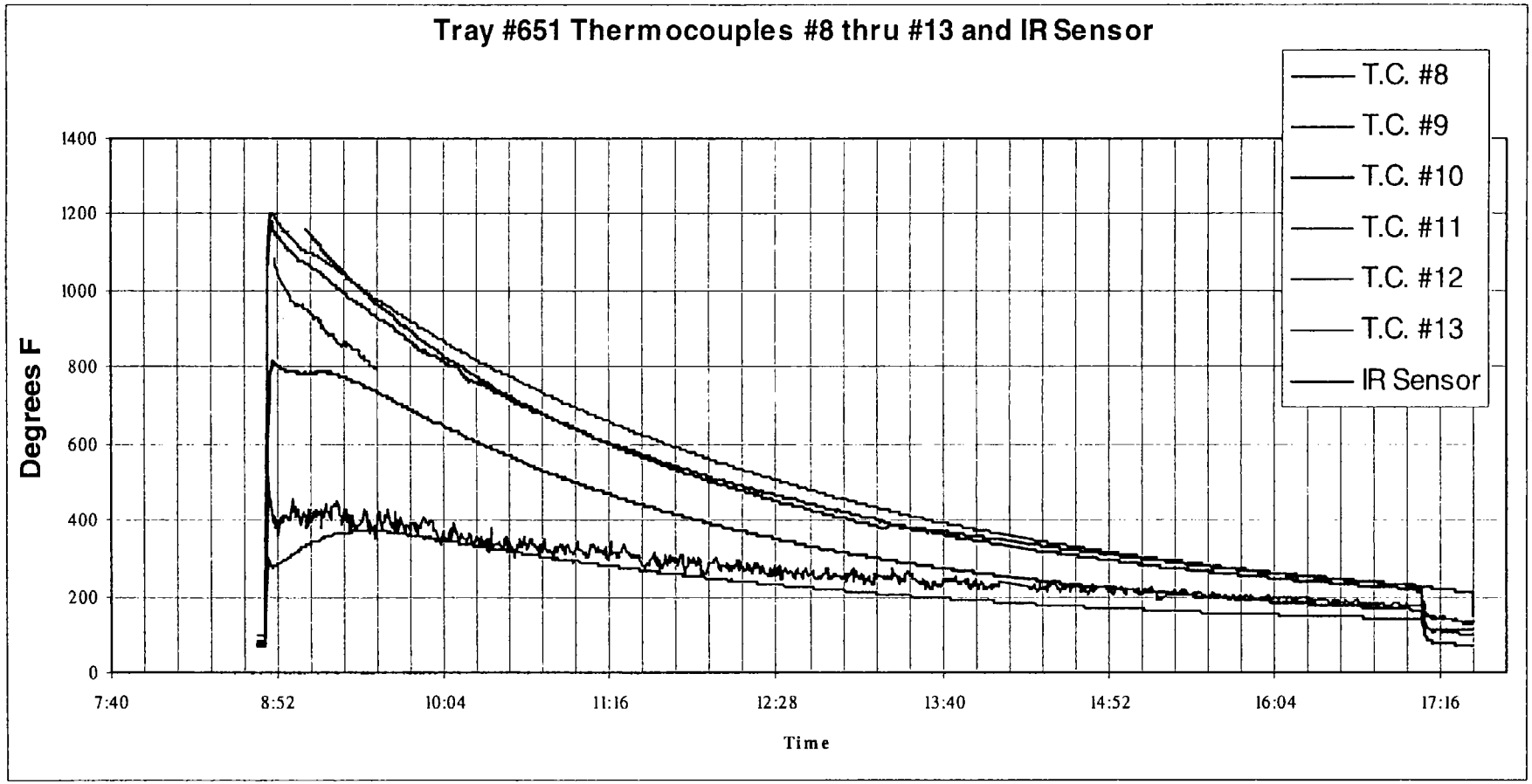
T.C. #2
T.C. #3
T.C. #4
T.C. #5
T.C. #6
T.C. #7
Degrees F
1400
1200
1000
800
600
400
200
0
8:24
9:36
10:48
12:00
13:12
14:24
15:36
16:48
Time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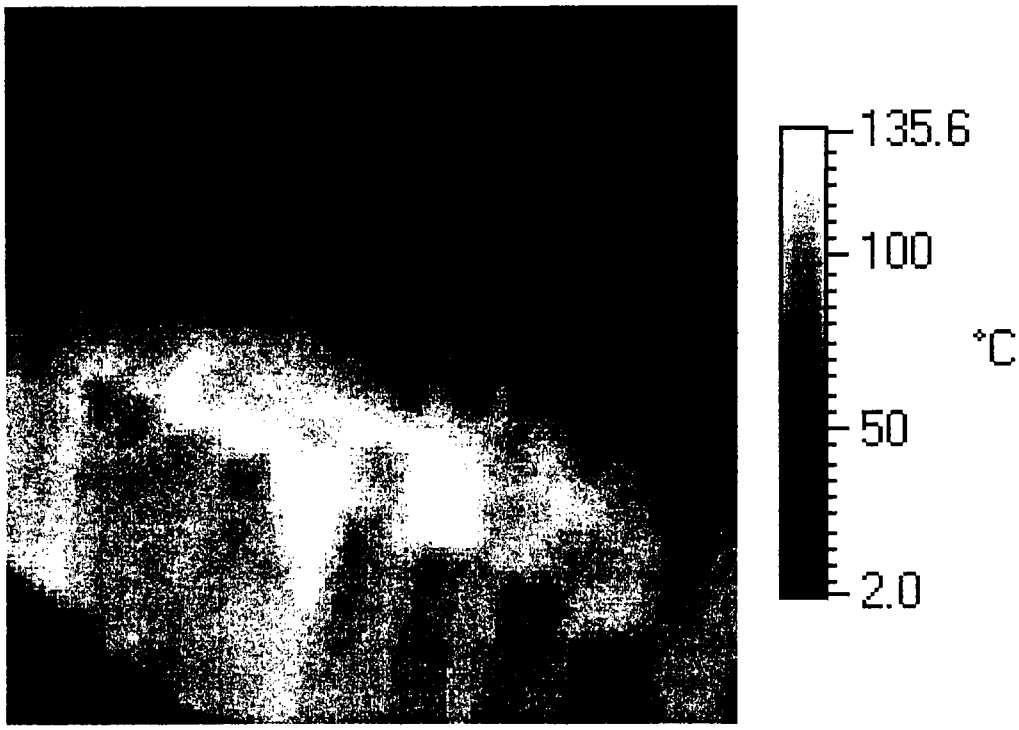
T.C. #8
T.C. #9
T.C. #10
T.C. #11
T.C. #12
T.C. #13
IR Sensor
Degrees F
1400
1200
1000
800
600
400
200
0
7:40
8:52
10:04
11:16
12:28
13:40
14:52
16:04
17:16
Time



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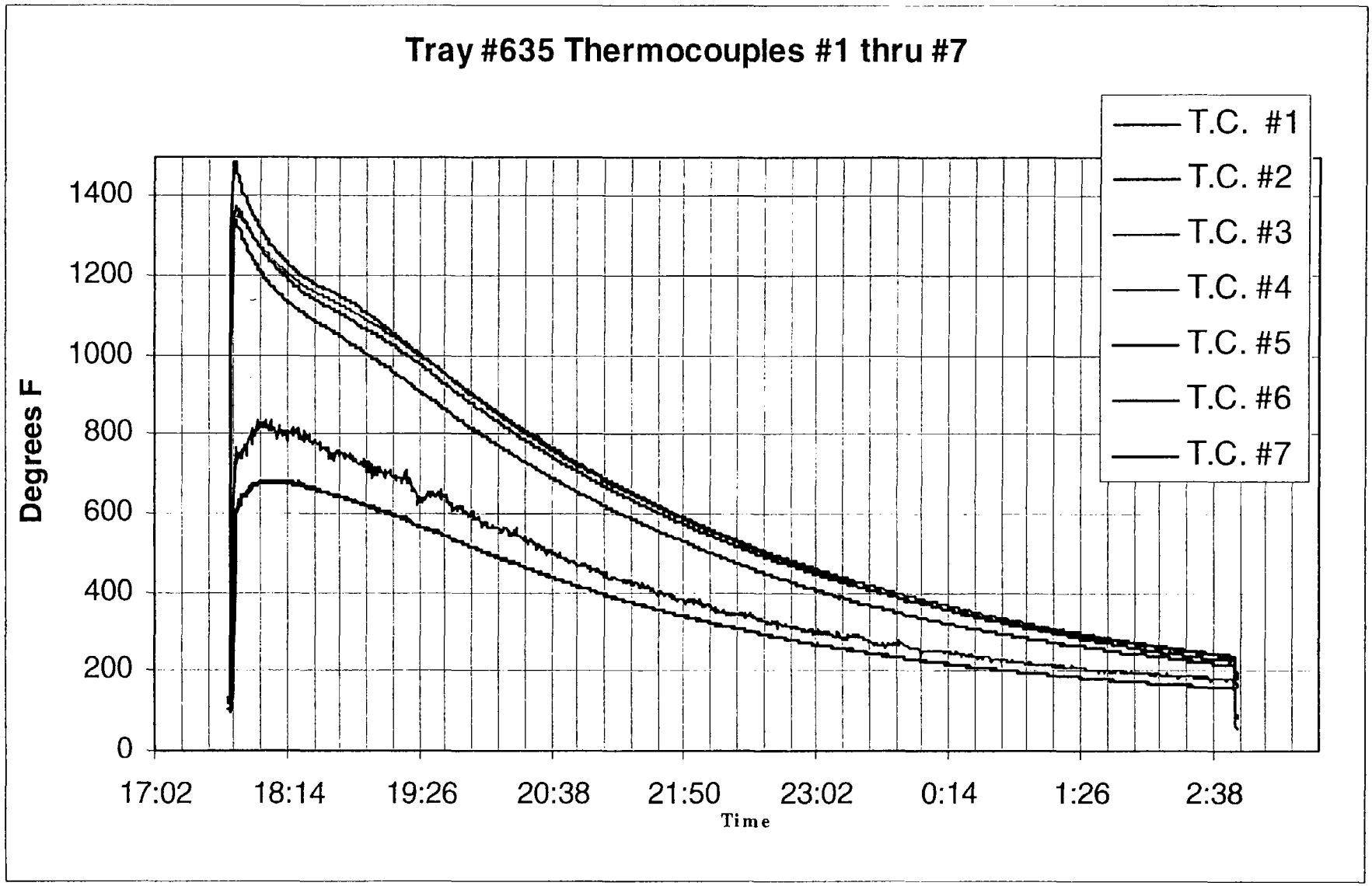
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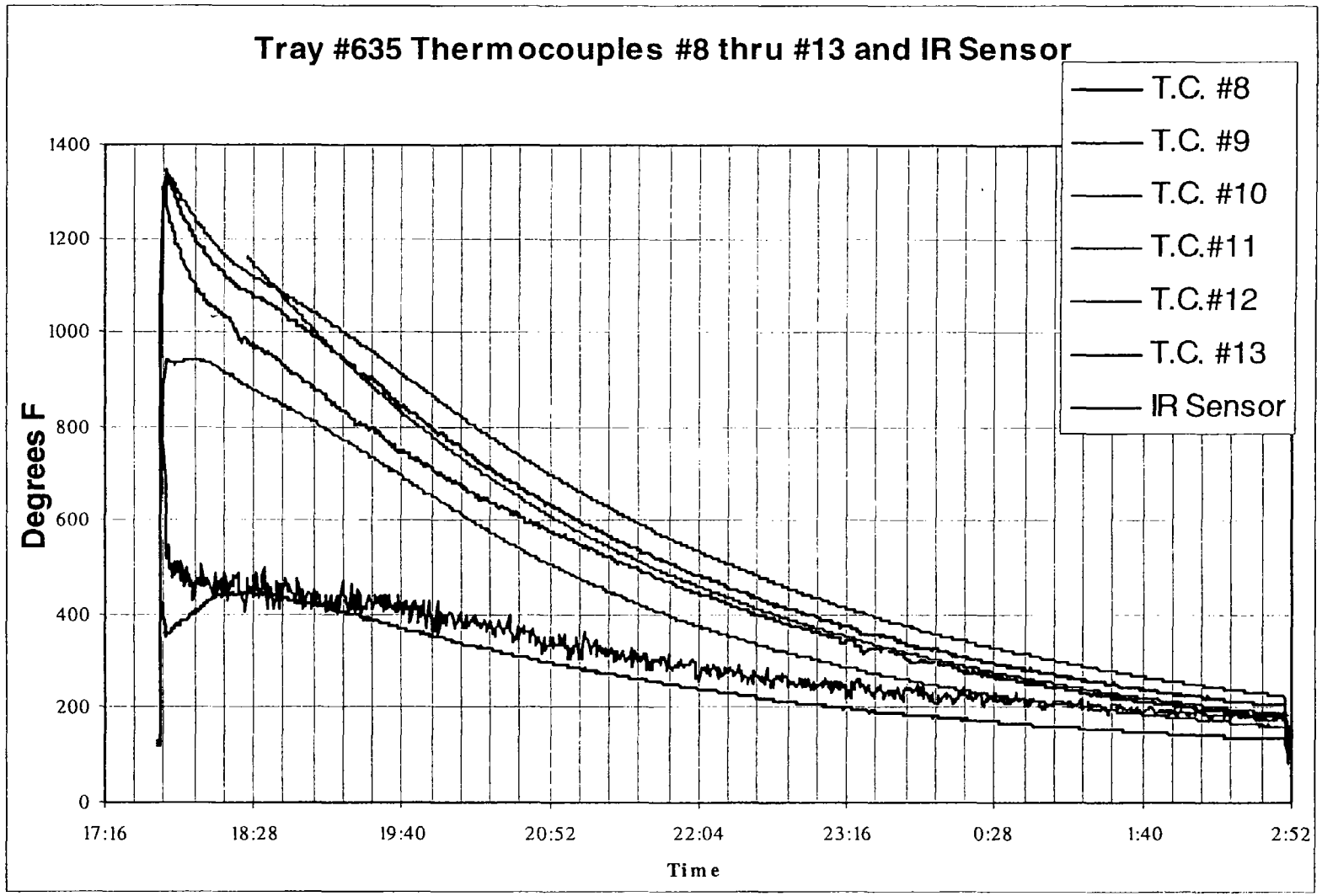
T.C. #1
T.C. #2
T.C. #3
T.C. #4
T.C. #5
T.C. #6
T.C. #7
Degrees F
1400
1200
1000
800
600
400
200
0
17:02
18:14
19:26
20:38
21:50
23:02
0:14
1:26
2:38
Time


Tray #635 Thermocouples #8 thru #13 and IR Sensor
T.C. #8
T.C. #9
T.C. #10
T.C.#11
T.C.#12
T.C. #13
IR Sensor
Degrees F
1400
1200
1000
800
600
400
200
0
17:16
18:28
19:40
20:52
22:04
23:16
0:28
1:40
2:52
Time

TAB
G

APPENDIX G

University of Dayton Research Institute Report

Modeling of the Incinerability of Chemical Agents GB and VX

Final Report Prepared by:

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In response to a verbal and written request from:

Ms. Lisa Douglas
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3580 Morrisville Road
Anniston, AL 36201

January 24, 2003

1. Background

The destruction efficiency (DE) of principal organic hazardous constituents (POHCs) is dominated by the temperature, time, fuel (waste)/air mixing, and fuel/air stoichiometry (excess air) experienced by the POHCs in the high temperature zones of incinerators (Dellinger, et al., 1991). Numerous calculations and experiments have shown that emissions of undestroyed, residual POHCs are kinetically, not thermodynamically controlled (Tsang and Shaub, 1982; Trenholm, et al., 1984; Dellinger, et al. 1991). As a result, accurate assessment of POHC emissions require thermal stability testing and cannot be accurately modeled based on thermodynamic equilibrium calculations.

Simple conceptual and more complex computer models indicate that gas-phase residence time and temperature in the post-flame zones of incinerators control the relative emissions of most POHCs (Clark, et al., 1984; Dellinger, et al., 1986; Dellinger, et al. 1991). This is because all molecules entering the flame zone of an incinerator are destroyed completely to thermodynamic endproducts and only the minute fraction escaping the flame zone is actually emitted from the facility. Once in the post-flame zone, gas-phase thermal decomposition reactivity in the presence of the major gas-phase constituents of this zone control the rate of POHC destruction and formation and destruction of products of incomplete combustion (PICs).

If all POHCs in a given waste stream are volatilized at approximately the same rate, they will experience the same post-flame gas-phase residence time, temperature, and stoichiometry history (relative concentrations of POHC, oxygen, and other major gas-phase constituents as the POHCs traverse this zone). This means that gas-phase thermal stability of POHCs (as determined under a standardized set of conditions) may be used to predict their relative incinerability. The temperature for 99% destruction at 2.0 seconds gas-phase residence time, [T_{99} (2)(°C)] has been used previously to rank the thermal stability of POHCs (Taylor, et al., 1990). Other residence times or levels of destruction may be used to develop a ranking. However, laboratory data indicate that although absolute POHC DEs are dependent upon time and temperature, relative DEs are largely insensitive to these parameters (Dellinger, et al., 1984; Graham, et al., 1986; Taylor and Dellinger, 1988). On the other hand, stoichiometry has been shown to be a significant variable in determining relative stability (Graham, et al., 1986; Taylor and Dellinger, 1988; Taylor, et al., 1991).

Experimental and theoretical considerations suggest that various flame zone failure modes exist that may cause residual POHCs to be emitted from a facility. The most prominent of these are thermal quenching and waste/air mixing failure modes. Even though a facility may be operating under nominal excess air conditions, poor waste/air mixing or thermal quenching zones due to poor heat transfer at incinerator surfaces will result in conditions where the rate of POHC destruction is low and PIC formation is favored. Consequently, it is believed that gas-phase thermal stability as characterized under oxygen-starved conditions is an effective predictor of POHC relative incinerability.

The UDRI thermal stability-based incinerability ranking was initially published in 1990 with further development published in 1991 (Taylor, et al. 1990; Dellinger, et al., 1991). The US-EPA has evaluated the UDRI gas-phase thermal decomposition kinetic rankings on both the pilot

and full-scale as a basis for determining POHC incinerability. Pilot-scale studies (Carroll, et al., 1992) of an eleven-component hazardous waste mixture under thermal failure and worst-case conditions (encompassing three failure-promoting conditions resulting in lower kiln-exit temperature, larger charge mass, and lower H/Cl ratio than the baseline set of conditions) both produced statistically significant correlations between product emission concentrations and their gas-phase thermal stability rankings. For the thermal failure tests, correlations above the 99% confidence interval were observed. Full-scale studies (Dellinger, et al., 1993) of a seven-component hazardous waste mixture indicated that thermal failure and waste/air mixing failures also produced statistically significant correlations. Based on median destruction and removal efficiencies (DREs), the data indicated that both the mixing and thermal failure modes produced statistically significant correlations between product emission concentrations and their gas-phase thermal stability rankings.

Statement of Work:

In order to respond accurately to a comment made during a public meeting, modeling of the chemical agents GB and VX need to be performed and compared to the UDRI class rankings. As such, it is prudent to the use the same facility that developed the original list so that our compounds our compared on an equal basis.

UDRI will perform molecular orbital theory modeling to determine the class ranking of chemical agents GB and VX. These chemicals were not modeled in the original class ranking used by EPA and state environmental regulators to determine the thermal stability of hazardous waste constituents. The original ranking list was prepared by UDRI and is used nationwide as the industry standard for ranking chemicals by class on their thermal stability.

UDRI will also review the REI report entitled "Chemical Agent Decomposition Rates at Temperatures anticipated in the Discharge Airlock." UDRI will participate in a conference call scheduled for Tuesday, January 14th, 2003 to discuss the different calculational approaches used. UDRI will provide a brief synopsis of the conference call and any resolutions reached.

Following this conference call, it was learned that REI had performed similar calculations in related work performed for the U.S. Army. The purpose of this report is to submit revised calculations in light of the recent work that was performed by REI.

Results and Conclusions:

Arrhenius parameters for GB and VX thermal decomposition were derived from molecular orbital modeling calculations (Density Functional Theory, B3LYP/6-31G(d), using the Gaussian 98 computer code). The results of the calculations are summarized in Tables 1 and 2 for GB and VX, respectively. All of the calculations were performed by UDRI, with the exception of the retro-ene pathway for VX. This pathway was recently calculated by Joe Bozzelli's research group as part of a U.S. Army sponsored study (Montgomery, et al., 2002).

The results indicate T_{99} (2) values for GB and VX of 457 and 515°C, respectively. Given these values, both of these compounds fall into class 5 of the UDRI thermal stability index. The

results indicate that neither of these two compounds exhibit significant thermal stability (UDRI thermal stability class rankings of 1 or 2). These results are consistent with previous EPA-sponsored thermal stability tests studies of structurally similar pesticide and herbicide materials (Tirey et al., 1993).

Further information regarding the calculations is available upon request.

Table 1. Arrhenius Parameters for GB Thermal Decomposition

Pathway	A (1/s)	n	Ea (cal/mole)
Retro-ene – Propene elimination	1.16E13	-0.58	41,870
P-C bond rupture	2.54E17	0	86,600
P-O bond rupture	5.04E17	0	95,600

Table 2. Arrhenius Parameters for VX Thermal Decomposition

Pathway	A (1/s)	n	Ea (cal/mole)
Retro-ene – Propene elimination	5.36E6	1.65	42,570
P-S bond rupture	1.69E18	0	65,200
S-C bond rupture	1.08E18	0	66,600

References:

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TAB
H

APPENDIX H

EPA Region IX Approval for JACADS to Elevate Temperature

MEMORANDUM OF RECORD

This is to document on 21 January 2003 U.S. Environmental Protection Agency (U.S. EPA) Representative, Stacy Braye and Department of Health and Human Services (DHHS) Representative, John Decker witnessed the modifications to the Discharge Airlock Monitoring configuration. JACADS has provided U.S. EPA Region IX information as follows:

(a) Briefing, 27 August 2002, San Francisco, CA, Subject: 12 August 2002 MPF DAL Incident.
(b) Memorandum, Response to U.S. EPA Request for Information Pursuant to Section 3007 of Resource Conservation and Recovery Act (RCRA), dated 21 November 2002.
(c) Memorandum, Status of Supplemental Reviews Based Upon the 12 August 2002 Event, dated 13 November 2002.
(d) Briefing, 13 January 2003, San Francisco, CA. Subject: 12 August 2002 MPF DAL Incident.
(e) Site Visit, 21 January 2003, Braye and Decker.

Based upon all information received to date, JACADS is authorized to proceed as follows:

(1) JACADS is authorized to resume processing all waste permissible under the JACADS RCRA Permit.
(2) JACADS may return to sampling MPF generated waste in accordance with the requirements of the Waste Analysis Plan or other applicable section of the JACADS RCRA Permit.
(3) The Discharge Airlock monitoring process was acceptable and may commence monitoring at 600 degrees.

1/21/03

Stacy ~~L.~~ Braye
JACADS Project Manager
U.S. EPA Region IX