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CHEMICAL ANALYSIS AND REACTION KINETICS OF EA-2192 IN DECONTAMINATION SOLUTION FOR THE MMD-1 PROJECT

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The studies reported include the development of analytical methods that are capable of identifying and determining the amount of EA-2192 [S-(2-diisopropylaminoethyl) methylphosphonothioic acid] in a decontamination solution. The rate of breakdown of EA-2192 in a mixture of monoethanolamine and caustic water was also determined.

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PREFACE

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CONTENTS

FIGURES

TABLES

CHEMICAL ANALYSIS AND REACTION KINETICS OF EA-2192 IN DECONTAMINATION SOLUTION FOR THE MMD-1 PROJECT

1.0 SUMMARY

EA-2192 [S-(2-diisopropylaminoethyl) methylphosphonothioic acid] is known to form from decontamination of VX in the presence of hydroxide. The current decontamination solution for the Munitions Management Device-1 (MMD-1) contains caustic water and monoethanolamine. As a result, some EA-2192 is formed, but not as much as decontamination in aqueous caustic solution.

A detailed study was done of the decontamination of EA-2192 in the MMD-1 decontamination solution. It was determined that EA-2192 will be destroyed to a concentration of less than 50 μg/mL in 5-6 hours of reaction time at 50-55°C, under typical operating conditions.

The studies included the development of an analytical method that was capable of detecting EA-2192 in decontamination solution. The best analytical method that was found used liquid chromatography/mass spectrometry. Several different sample preparation methods where characterized. Other detection methods were also studied, including gas chromatography/mass spectrometry with various sample preparation methods, and nuclear magnetic resonance.

Background

EA-2192 is a decontamination product of VX in the presence of hydroxide. EA-2192 is only about 2-3 times less toxic than VX. However, EA-2192 is a nonvolatile ionic compound which cannot be detected by traditional methods used for VX, including GC/MS analysis or AgF conversion to the G analog. Analytical methods were needed that were capable of determining the amount of EA-2192 in decontamination solutions to low concentrations.

In the initial studies of the MMD chemistry done in 1995-97,[1] the decontamination solutions were screened for EA-2192 using an analytical method using liquid chromatography/mass spectrometry with a Finnigan TSQ-7000 mass spectrometer. This instrument would be difficult to deploy on site.

Studies were done to develop methods that are appropriate for on-site analysis. Studies concentrated on using liquid chromatography/mass spectrometry (LC/MS). In recent years, several mass spectrometry vendors have developed bench-top LC/MS instruments that may be appropriate for field use. In particular, a LC/MSD made by Agilent was used for detection of EA-2192. In addition, gas chromatography/mass spectrometry (GC/MS) methods were investigated.

This report includes a discussion of the analytical methods that were developed for the analysis of EA-2192 in decontamination solutions in Section 2.0. In Section 3.0, the kinetic studies that were performed on the destruction of EA-2192 in the MMD decontamination solution are reported.

2.0 ANALYTICAL METHODS FOR DETECTION OF EA-2192 IN DECONTAMINATION SOLUTION

2.0.1 Chemical Agent Standard

A standard of EA-2192 was obtained from the CASARM program. The standard was received on 11 January 2000 as a dilute standard in isopropyl alcohol at a reported concentration of 412.11 µg/mL, with a 10 mL total quantity. The standard was diluted in the laboratory by 1:100 in DI water to make a working standard of 4 µg/mL. This standard was further diluted to make calibration standards.

2.0.2 Matrix and Other Chemicals

The MMD decontamination solution matrix was Sample Number MRCS-VX-4.5, generated as part of the MMD Project on 7 Mar 1996 in Room 210, Building E3300. The solution has been stored in a hood at room temperature. As a result, secondary reactions may have occurred that make the matrix more complex than a fresh solution would be.

When kinetic studies were begun, decontamination solution prepared from VX and monoethanolamine decontamination solution were generated. Some of these solutions were also used for method development or method validation, since they were considerably less complex than the 1996 solution.

Distilled, deionized water (18 MΩ) was obtained from a Barnstead (Dubuque, IA) Nanopure system. Acetonitrile was J. T. Baker HPLC Grade solvent. Acetic acid was glacial acetic acid from J. T. Baker.

2.1 Liquid Chromatography/Mass Spectrometry Method Development

Initial studies were done on a Hewlett Packard 5989A "MS Engine" mass spectrometer with an HP 1090 High Performance Liquid Chromatograph. The MS was equipped with either an electrospray ionization (ESI) or an atmospheric pressure chemical ionization (APCI) ion source, both of which were manufactured by Analytica of Branford (Branford, CT).

Final studies and kinetic measurements were made using an Agilent LC/MSD Model 1100. This is a newer model of instrument which fits on a benchtop. This instrument is 10-100 times more sensitive than the older 5989A.

Typical LC conditions used a 150 mm long by 2.1 mm i.d. column with a flow rate of 0.25 mL/min.

A number of experimental and instrumental conditions were optimized:

1. LC column
2. LC mobile phase
3. Sample Preparation
4. Ion source
5. Post-column derivatization
6. Injector programming
7. LC/MSD

2.1.1 Optimized LC/MS Method

The parameters that were found for the optimized LC/MS method are given in this section. The discussion of the optimization studies are given in the following sections.

The final, optimized conditions that were used for the analyses are as follows:

LC column:	Phenomenex Luna or Polar-RP column, 150 mm long by 2.0 mm i.d. column, and 4.0 µm particle size. (reversed phase chromatography column)
Flow rate:	0.25 mL/min.
Mobile phase:	Gradient 0-10 min.: 97.5% DI water, 2.5% acetonitrile, 1% acetic acid 20 min.: 50% DI water, 50% acetonitrile, 0.5% acetic acid 40 min.: return to 97.5% water to equilibrate column
Total Run Time:	60 min.
Flow splitting:	1:5 liquid flow to waste
MSD ion source:	Electrospray
Capillary voltage:	4000 V
Dry Gas flow:	9 L/min.
Nozzle gas pressure:	50 psig
Gas Temp.:	300°C
Other MSD conditions:	According to Autotune using Agilent ESI tune solution
Injection volume:	25 µL
Detection method:	SIM or Scan
SIM Detection ions:	240 (fragmenter = 120V), 128 (fragmenter = 200 V)
Sample Preparation:	Dilution of decontamination solution by a factor of 1:100 by volume in a solution of 10% acetic acid in water.

This method was evaluated using a two day, Class II P&A study. The P&A data is given in Appendix A. The results of the statistical analysis are given in Table 2-1. The MDL that is determined from the study is 7.59 ng/mL for the first day, and 4.6 ng/mL for the second day, uncorrected for dilution. Corrected for dilution of the samples by 1:100 during the sample preparation, the MDL results are 0.759 and 0.46 µg/mL in the original decontamination solution. The calibration data is given in Appendix A.

Table 2-1: Class II P&A Results for the Final EA-2192 Analytical Method for LC/MS. The First Column is in µg/mL, and the Second Column is in ng/mL

Day 1:

Ave. Spike recovery		0.0316	31.6
Std. Dev.		0.0025	2.53
MDL		0.0076	7.59
% recovery			79.02%
MDL, corr. for dilution		0.759	

Day 2:

Ave. Spike recovery		0.0245	24.5
Std. Dev.		0.0015	1.52
MDL		0.0046	4.6
% recovery			61.3%
MDL, corr. for dilution		0.46	

2.1.2 Best LC Column

In order to obtain acceptable LC results, it is necessary to find a column phase which retains the analyte. Then the conditions can be optimized to separate the analyte from other components and to find the best sensitivity. A good column is needed for separating EA-2192 from other components of the decontamination solution using chromatography.

The best column that has been found to date is a C18 column using reversed phase chromatography. The best brand of columns that has been used so far is a Phenomenex Luna column, 2.1 mm i.d. × 150 mm length × 4 µm particle size. However, all brands have not been tested, and any C18 column and some other reversed phase columns may also give acceptable results. Other columns that were used include the Agilent Zorbax Eclipse XDB-C18 column (Agilent part no. 993700.902), 2.1 mm i.d. by 150 mm length by 5 µm particle size.

Another type of LC column has been found on which EA-2192 has a great deal of retention for normal phase chromatography. The type of column is known as a HILIC column, for hydrophilic interaction chromatography. This is a type of normal phase chromatography (nonpolar solvent and polar solid phase) which was developed for analysis of polar molecules which are not retained in reversed phase chromatography. The phase of the column, sold by PolyLC, is polyhydroxyethyl aspartamide, which has charged and polar groups on the surface. This phase is appropriate for EA-2192, which is a very polar molecule which is a zwitterion over a range of pH.

This column provides a different type of separation of EA-2192 from other components of the decontamination solution, and it provides good chromatography for EA-2192. However, it is necessary to use an organic solvent for sample preparation. Acetonitrile (95% with 5% water) is an acceptable solvent for both chromatography and electrospray ionization.

A weak anion exchange column, a Synchropak AX-100, was used by itself and in series with a C18 column. No improvement in chromatography was observed. This column is also more advantageous for normal phase chromatography, but it was not studied in detail.

Several types of mobile phases were tested for the HILIC column. The retention of EA-2192 varied significantly for different solvents. A mobile phase mixture of 95% acetonitrile and 5% water gave good retention. Figure 2-1 shows a typical chromatogram of a 20 ppm standard solution. Figure 2-2 shows the mass spectrum for the EA-2192 peak.

One advantage of using 95% acetonitrile is that it is miscible with methylene chloride. Experiments were done to extract EA-2192 with methylene chloride or other nonpolar solvents. Most of these extracts could be analyzed by LC/MS to determine extraction efficiencies. This analysis allowed a separate determination of the extraction efficiency and the derivatization efficiency in the GC/MS method development.

Other mobile phases were attempted. Increasing the acetonitrile concentration to 97% produced a longer retention time for EA-2192. However, the peak became broader, so sensitivity and quantitation accuracy were not as good. Mobile phases of methanol or isopropanol were not effective, because EA-2192 had no retention on the column with these mobile phases.

The previous work showed that, for reversed phase chromatography, it is best to use 95-100% aqueous mobile phase. In comparison, the HILIC column allows isocratic rather than gradient runs, so the runs are shorter, but they must be in 95% organic solvent.

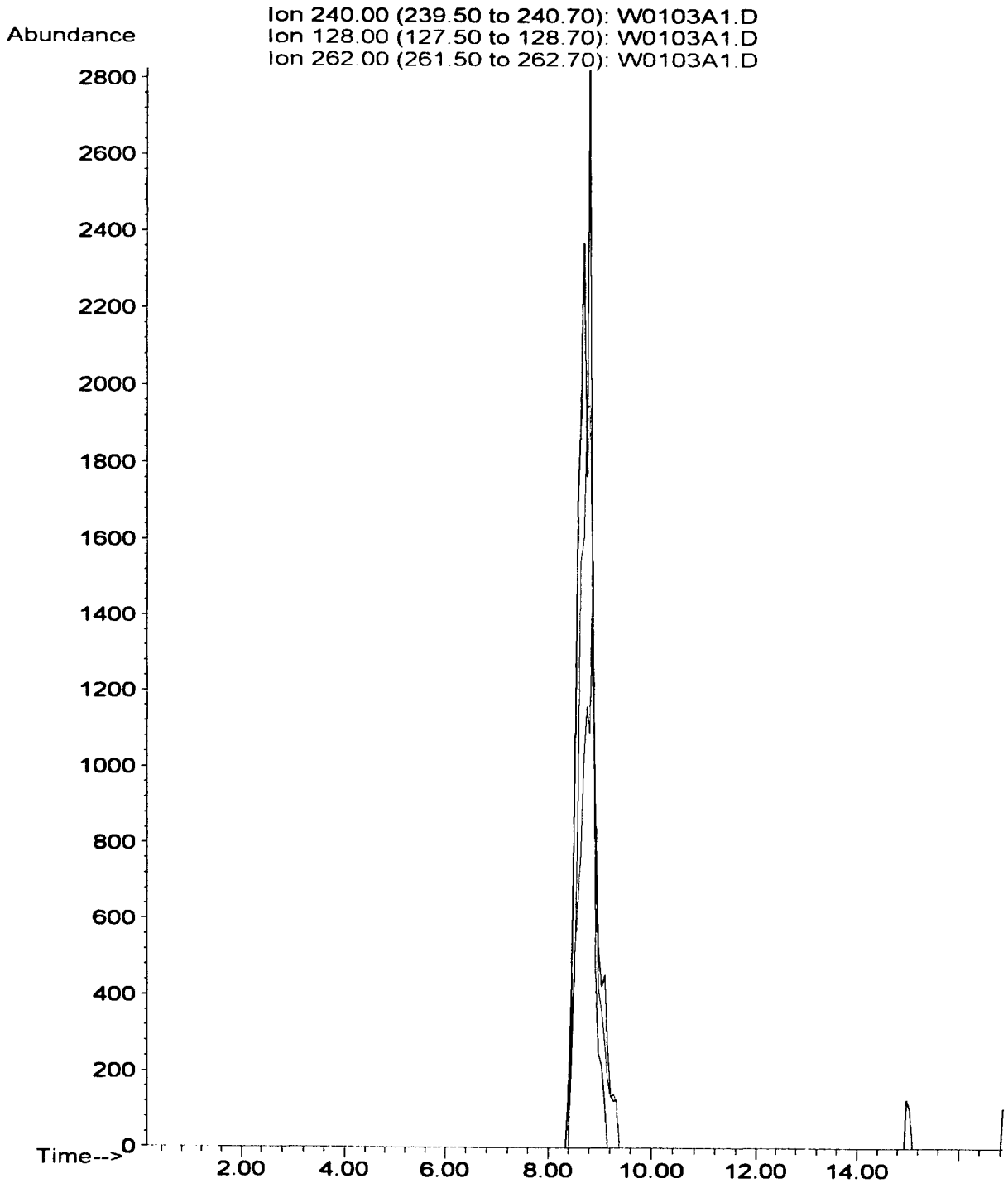

Figure 2-1. LC/MS Using the 5989A Instrument Showing an Extracted Ion Chromatogram of a Standard of 20 ppm of EA-2192 in Methylene Chloride. The Ion for *m/z* 240 is the $M+H^+$ ion, *m/z* 128 is a Fragment ion, and *m/z* 262 is a Sodium Adduct, $M+Na^+$.

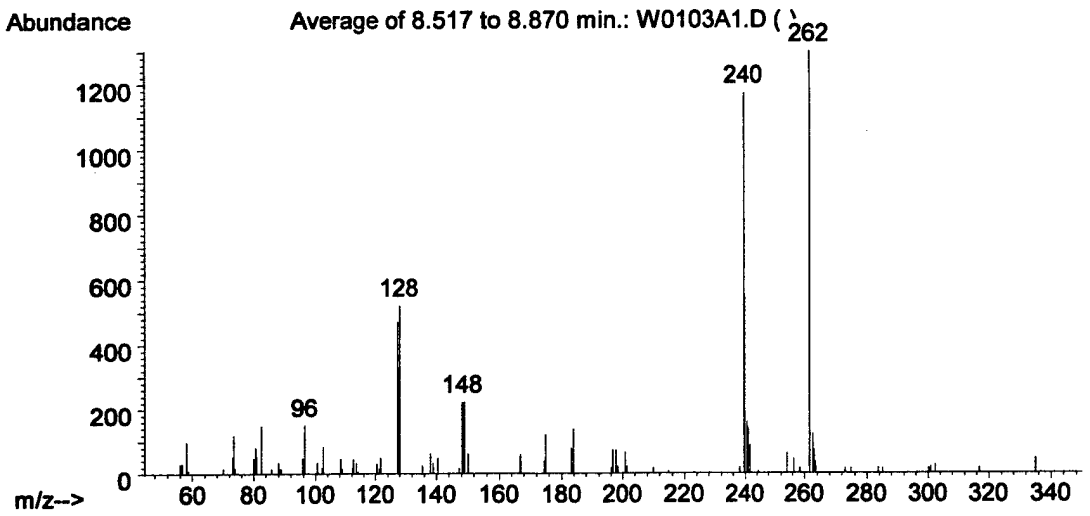

Figure 2-2. Mass Spectrum of EA-2192 Peak from the Chromatogram in Figure 2-1.

2.1.3 LC Mobile Phase

For reversed phase chromatography, it is best to use 95-100% aqueous mobile phase. Many types of C18 columns perform best with a small percentage of organic solvent, rather than 100% water, to prevent the column phase from becoming desolvated and becoming "matted down". It is necessary to use a gradient run after the EA-2192 elutes to remove from the column the other nonpolar compounds that are present in the decontamination solutions. Figure 2-3 shows a total ion chromatograph of a typical gradient run of a MMD decontamination solution, which shows many late eluting compounds.

The pH of the aqueous phase can be adjusted. EA-2192 is soluble in aqueous solution at any pH. There are other compounds in the decontamination solution that are more soluble in acidic solution and less soluble in basic solution, so the pH adjustment can be used to modify the chromatography of those compounds to resolve them from EA-2192. Overall, an acidic mobile phase is generally the most preferable. A solution of 1% acetic acid in water gives good results.

Since the detection is done by electrospray ionization, the ionization adds constraints to the type of mobile phase that can be used. The use of a nonvolatile acidic buffer should be avoided, since the nonvolatile salts tend to build up in the electrospray ion source and degrade the performance of the source. Acetic acid is volatile, so it does not tend to form salt deposits in the source, in the absence of samples with a high nonvolatile salt content.

Figure 2-4 shows the chromatogram of a standard of EA-2192 in acidic mobile phase (1% acetic acid in DI water), using ESI. Figure 2-5 shows a chromatogram in basic mobile phase (pH 8, adjusted using ammonium hydroxide)

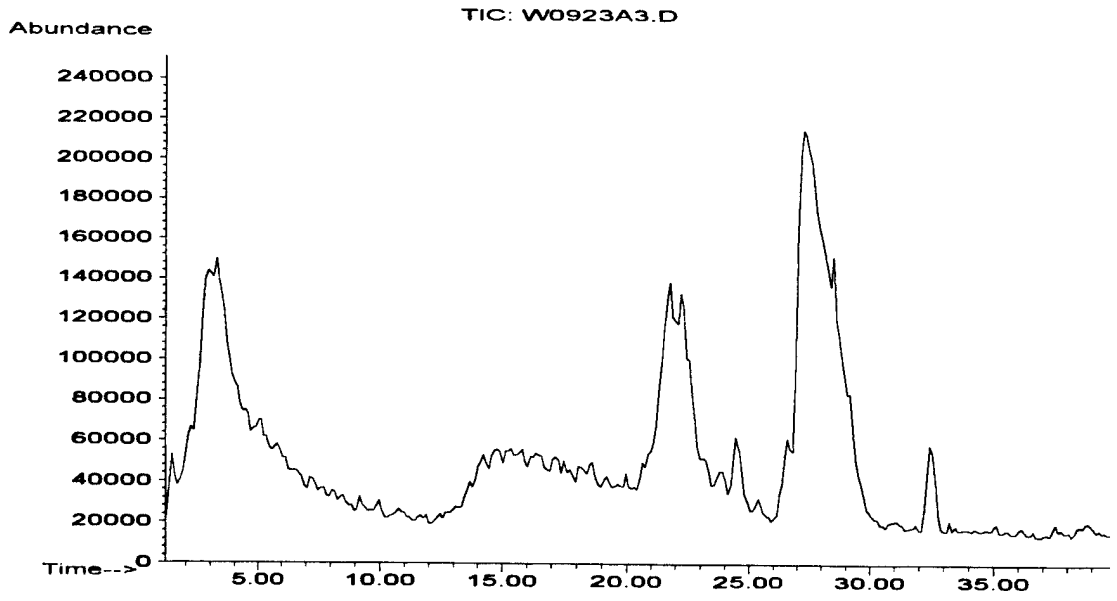

Figure 2-3. Typical LC Chromatogram with a Total Ion Chromatograph (TIC) Mass Spectrum Using Gradient Elution of a Acidified MMD Decontamination Solution. The gradient is from 100% Acidic Aqueous Solution (5 min.) to 52% Aqueous at 30 min. The Gradient is Necessary to Elute the Nonpolar Compounds from the Column.

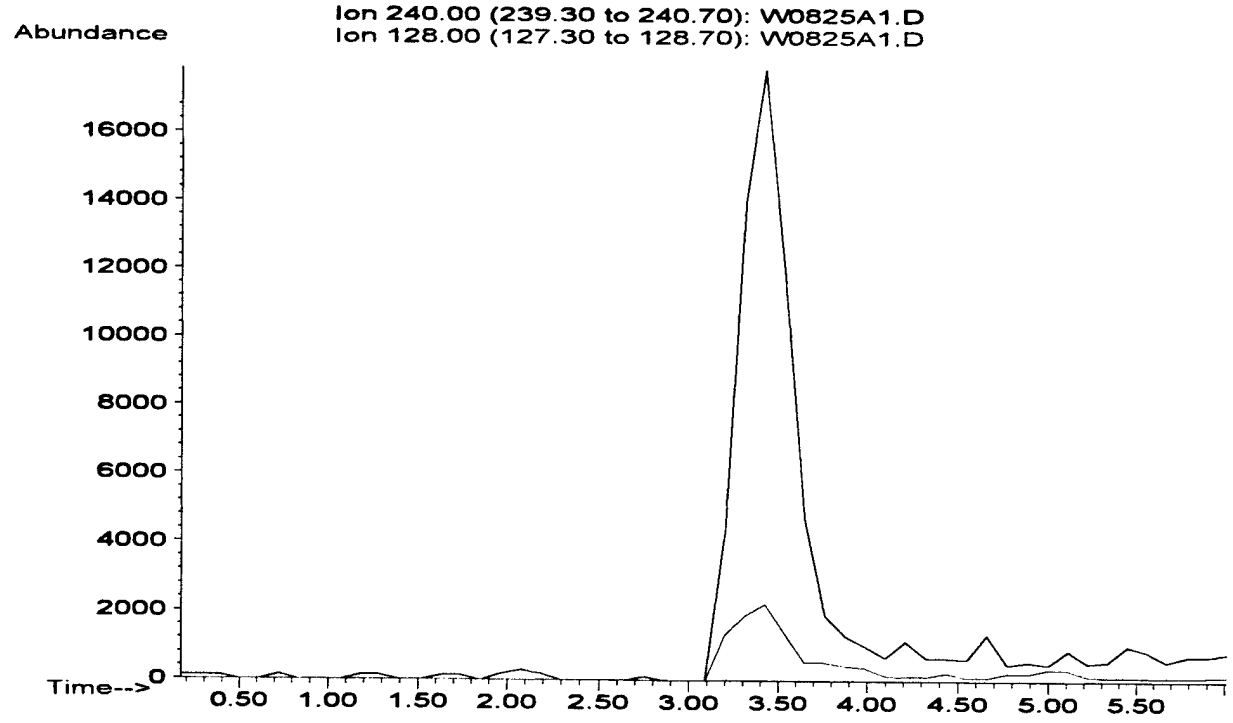

Figure 2-4. ESI Chromatogram from a 5989A Instrument of a 20 ppm EA-2192 Standard in Acidic Mobile Phase with 2.5% Acetonitrile and a Hypersil ODS Column.

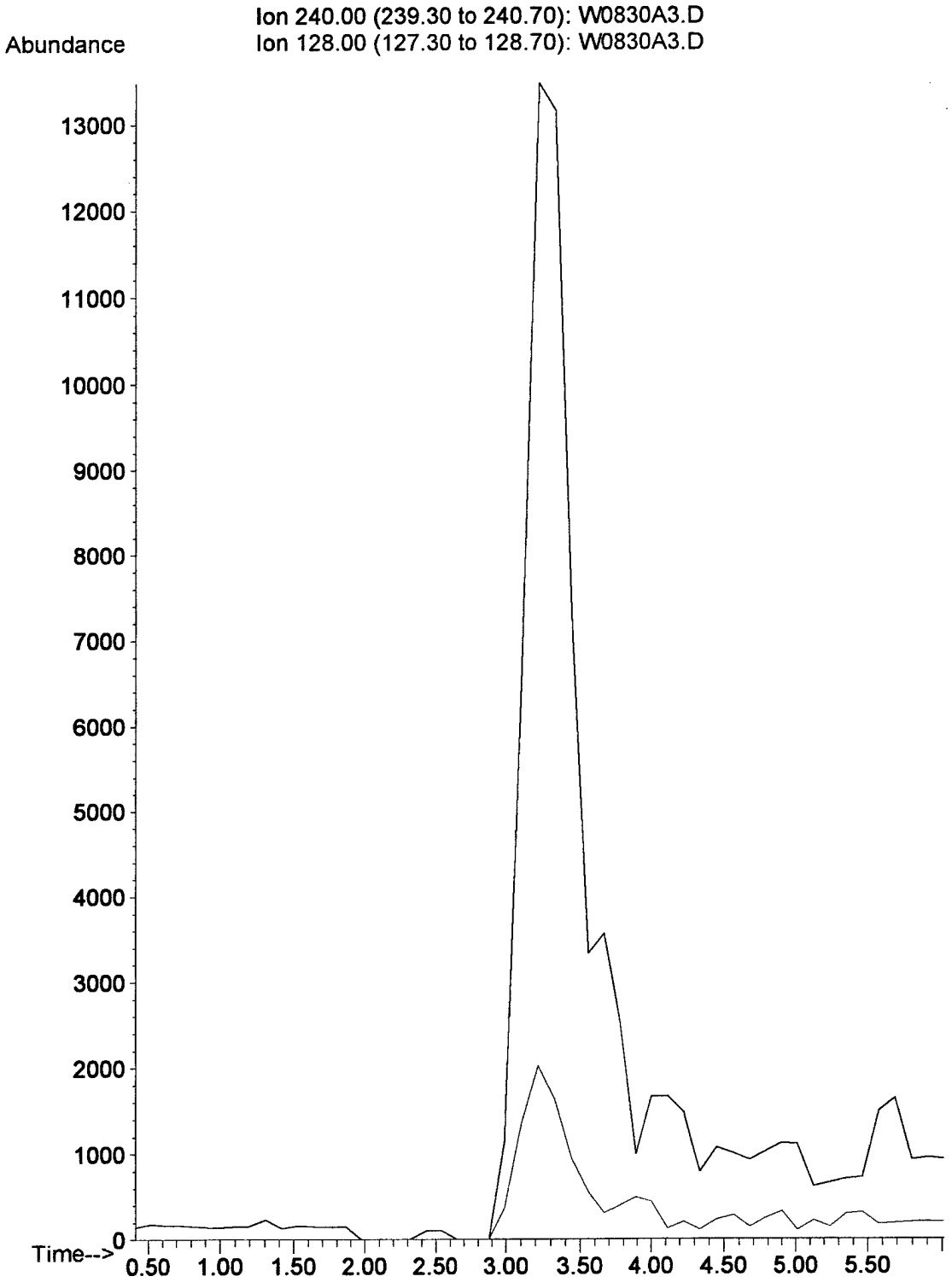

Figure 2-5. ESI Chromatogram of 20 ppm EA-2192 Standard in Basic Mobile Phase, with Postcolumn Addition of Acetic Acid to Improve Ionization.

using ESI. It was necessary to add postcolumn acetic acid to the basic mobile phase in order to observe good ionization signal with ESI. There is little change in the retention of EA-2192 over these conditions.

2.1.4 MSD Ion Source

A study was done to compare electrospray ionization (ESI) to atmospheric pressure chemical ionization (APCI) to ionize EA-2192 using the same instrument. The ion sources for these two techniques were available for both the 5989A and the LC/MSD.

ESI in positive ion mode produces a clean spectrum with a strong signal for the $[M+H]^+$ peak at *m/z* 240. There is a small amount of fragmentation to *m/z* 128, which can be used as a confirmation ion. The mass spectrum is shown in Figure 2-6. This method gives the best results that were observed.

A limitation of ESI is the low tolerance for high flow rates of aqueous solution. The best results were obtained by splitting the LC flow of 0.25 mL/min by about 1:5, so that about 50 µL/min of solution entered the source. Since ESI is a concentration sensitive method, the exact amount of splitting is not critical, as long as the flow into the source is <100 µL/min or so.

The long-term stability of ESI for the quantitative analysis of decontamination solutions is acceptable. In general, analysis of samples with high salt content can be difficult with atmospheric pressure ionization methods (either ESI or APCI), since the salt tends to build up in the ion source and affect the high voltage lenses. Newer models of ion sources, such as the one for the LC/MSD, are less subject to this type of problem. To minimize the cleaning that is required for the source, it is possible to divert the flow from the LC for the first few minutes of the run. For reversed phase chromatography, the salts and polar compounds elute in the void volume at the beginning of the run. By diverting the flow, these materials are not introduced into the source, so contamination is reduced.

Negative ion ESI was also attempted in both acidic and basic solutions. No useable signal was observed for EA-2192.

Electrospray ionization gave good results using 97.5% acetonitrile. With less than 2.5% water in the solution, however, the signal degraded.

APCI is often found to be more stable for aqueous mobile phases and less affected by high salt concentrations. APCI in positive ion mode of EA-2192 has the problem that the EA-2192 pyrolyzes in the source to form mostly VX-thiol [2-diisopropylaminoethanethiol], which forms a large *m/z* 162 signal.[2] A small *m/z* 240 signal is observed, but it can be unstable. Since the decontamination solution contains

m/z 162 signal for detection of EA-2192. The fragmentation can be decreased somewhat by decreasing the source vaporization temperature from 350-400°C, which is the typical operating temperature, to 250-300°C. At lower temperatures, the ionization becomes less efficient and the signal is less stable. For negative ion APCI, no signal was observed in either acidic or basic mobile phase.

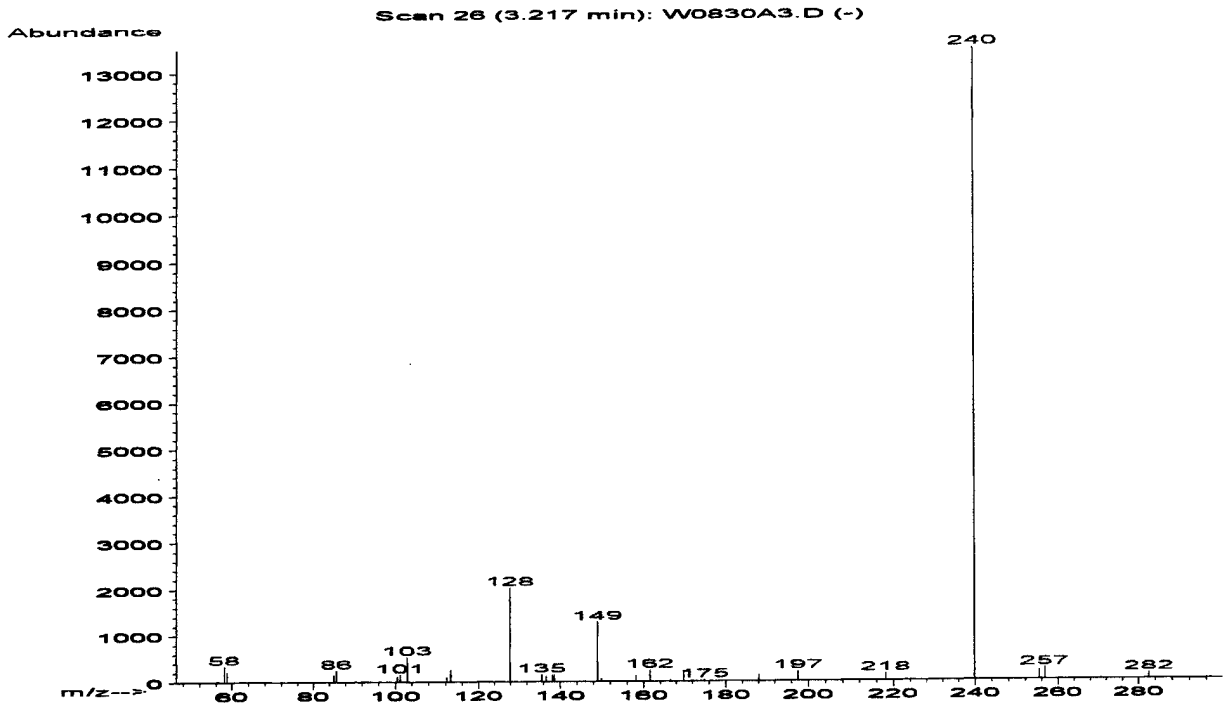

Figure 2-6. Positive Ion ESI Mass Spectrum of EA-2192 from a Standard Solution, Using a 5989A Instrument. The $[M+H]^+$ peak is at *m/z* 240.

2.1.5 Post-Column Derivatization

To improve the performance of APCI, a method of postcolumn derivatization was studied.[2] In this method, a reagent was added to the LC flow to methylate the EA-2192, forming the methyl analog of VX. The derivative could be detected as an $[M+H]^+$ by APCI with little thermal decomposition.

The reagent TMPAH [trimethylphenyl ammonium hydroxide] was used. A commercial 0.1 M solution in methanol (Fluka) was added at 5-10 µL/min to the LC flow of 250 µL/min. The $[M+H]^+$ ion at *m/z* 254 was observed. The chromatogram of a standard is shown in Figure 2-7. The signal is dominated by the $[M+H]^+$ ion, with some fragmentation to form the *m/z* 128, but the thermal decomposition to form the *m/z* 162 ion has been greatly reduced. The signal is fairly strong, although not quite as good as for ESI.

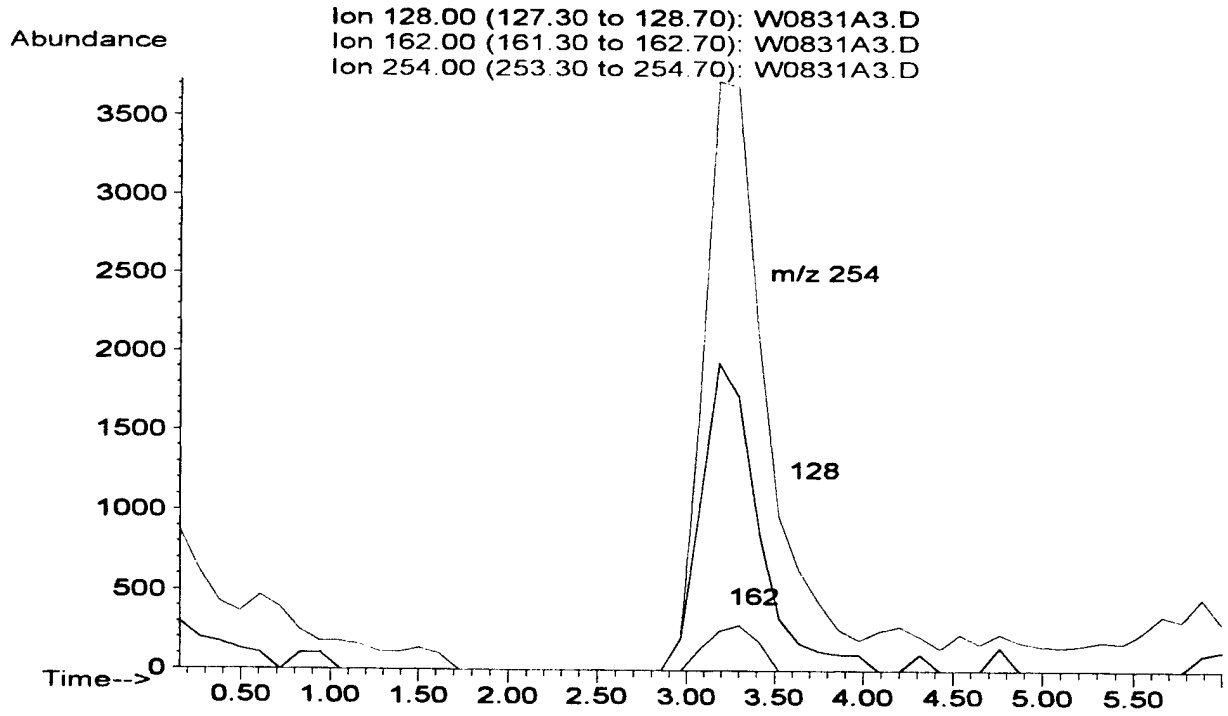

Figure 2-7. Positive Ion APCI Extracted Ion Chromatogram of EA-2192 Standard Using Postcolumn Derivatization with TMPAH to form Methylated Species, *m/z* 254.

The disadvantage of this method is that it requires the extra step of adding TMPAH using a syringe pump, which is not a standard approach for APCI. The excess TMPAH deposits in the source to form a residue on the source optics and corona needle, which must be cleaned at least daily. There is no long-term experience with this approach that is available to judge the reliability for routine operation, particularly for quantitative applications.

2.1.6 Injector Programming to Improve Peak Shape

In principle, the detection sensitivity should be improved by using less dilution of the sample during the sample preparation. In practice, for decontamination solutions which are undiluted, the EA-2192 peak can be very broad in chromatographs of the decontamination solution, and the chromatography can be poor. This is due to the poor solvent matching between the MEA matrix and the LC mobile phase, and by the suppression of ionization by MEA of the other analytes.

In work done in 1995 on a Finnigan TSQ-7000 mass spectrometer, it was found that the peak shapes for EA-2192 were very narrow.[3] The reason for this difference was traced to the difference in the HPLC hardware. The HPLC that was used with the TSQ was designed for low flow rates. It was programmed with a gradient

that began at 2 min into the run, before the EA-2192 eluted in aqueous mobile phase. The EA-2192 eluted on the leading edge of the organic solvent in the gradient, which allowed a very sharp peak to be obtained.

In contrast, if the HPLC is not optimized for low flow rates, a gradient does not begin until at least 10 min. into the run. In this case, the EA-2192 elutes during an isocratic portion of the run. The peak shape in this case is wider than for elution at the beginning of a gradient when running decontamination solutions.

A approach for addressing this problem was found. The syringe programming feature on the HPLC 1090 was used. A 25 µL quantity of sample was pulled into the injector loop from an autosampler vial, followed by 100-150µL of basic water, and then 5-10 µL of acetonitrile. These three components were pulled into the sample loop at the same time. This quantity of acetonitrile, injected after the sample, was enough to elute the EA-2192 off the column in a rapid, narrow peak.

This technique improved the shape of the chromatographic peak significantly. Figure 2-8 shows the chromatograph of EA-2192 which is eluted off the column using the syringe programming method. This approach still requires validation work, but it appears to greatly improve the chromatography of EA-2192 on the C18 column. It also decreases the dependence on the type of LC that is used. Figure 2-8 shows that several compounds co-elute with the EA-2192 using this method. This observation suggests that there may still be ionization suppression from competition of the different species in the ion source.

This method was not tested on the LC/MSD instrument. The instrument had better sensitivity to the EA-2192, so extra efforts, such as the use of syringe programming, were not necessary to obtain adequate signal. The chromatography and resolution was improved by diluting the sample by 1:100, and using the improved sensitivity of the mass spectrometer to detect the analyte. If even better sensitivity is required, this approach could be explored further.

2.1.7 5989A vs. LC/MSD

Early work was done on an HP 5989A mass spectrometer, which is too large for field work. This model is no longer sold by HP. The new model of LC/MS which is offered by Agilent (formerly HP) is called the LC/MSD. This system has significant improvements in sensitivity over the older 5989A.

Sensitivity comparisons were done between the 5989A and an LC/MSD Model SL. A series of standards of DIMP [diisopropyl methylphosphonate] were analyzed. The LC/MSD provided sensitivity to this compound using positive ion APCI which was up to 100 times higher than the 5989A, using comparable conditions.

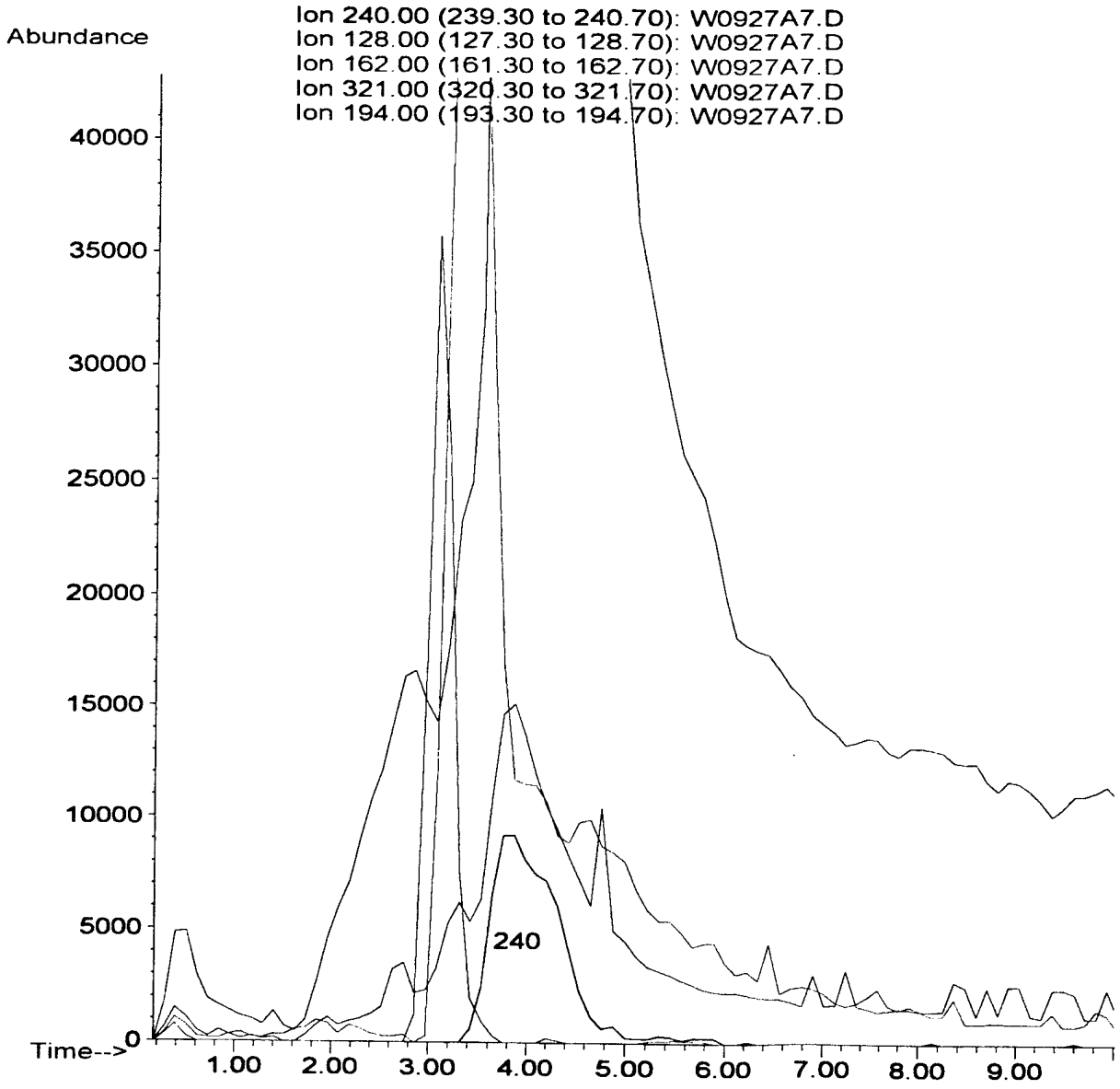

Figure 2-8. ESI Chromatogram of MMD Decontamination Solution Spiked to 40 ppm with EA-2192. EA-2192 is Eluted Using the Syringe Programming Method on the 5989A Instrument. This Approach was not Attempted on the LC/MSD.

2.1.8 Sample Preparation: Reversed Phase Chromatography

Since the decontamination samples are aqueous based, little sample preparation is necessary for reversed phase chromatography. The sample matrix is rather viscous, so dilution of the matrix makes it inject into the LC more accurately. The samples are basic, so acidifying them with acetic acid decreases the chance that they may cause column damage.

As a result, the sample preparation consists of

1. Add glacial acetic acid to the sample until it is acidic.
2. Add DI water or ammonium acetate buffer to dilute. With an LC/MSD, a total dilution of 1:100 gives good chromatographic performance and still gives adequate signal to give a detection limit of 1 ppm. With a 5989A, less dilution, down to 1:3 dilution, is required to achieve good sensitivity.

2.1.9 Sample Preparation: Normal Phase Chromatography and Solvent Extraction from Aqueous Solutions

The aqueous based samples can be run by normal phase chromatography on the HILIC column, but injection volumes must be small. If the volume is larger than about 5-10 µL, the peak shapes are distorted due to solvent effects.

If the hydrolysate sample is diluted in acetonitrile rather than water, the sample is matched to the mobile phase and the retention times are more reliable. However, the hydrolysate is not completely miscible with acetonitrile, so a liquid layer separates. The second phase is probably a high salt aqueous solution. The partition efficiency of the EA-2192 between the aqueous and acetonitrile phases was not determined. However, the two liquid phases can be combined by adding <10% methanol. This is not a preferred method for preparing the samples, though.

It was determined that some EA-2192 can be extracted from the hydrolysate using a liquid/liquid extraction with methylene chloride or other nonpolar solvents. Methylene chloride has an advantage over acetonitrile in that it can be analyzed more readily by GC/MS, although derivatization is necessary. Acetonitrile solutions have a higher water content that is not prefered for GC/MS analysis. The methylene chloride solutions can also be run directly by LC/MS using normal phase chromatography.

Methylene chloride and other solvents were compared to determine the best extraction efficiency. The extraction efficiencies determined by LC/MS are given in Table 2-2.

Methylene chloride and chloroform have about the same extraction efficiency. 1-Butanol has a significantly higher extraction efficiency. However, it has the disadvantage that it extracts about 10% water by volume from the aqueous phase, which makes it a disadvantage for GC/MS analysis. Less polar solvents, like toluene, hexane, and ethyl acetate, had very little extraction of EA-2192. Therefore, further work on solvent extraction concentrated on chloroform and methylene chloride.

Table 2-2: Extraction Efficiencies of EA-2192 from Aqueous Standard Solutions. Each Result is from one run of one solution. Signals for the LC/MS were compared between extract in the extraction solvent and a standard solution diluted in the same solvent (one point calibration).

Solvent	Recovery (%)
Ethyl Acetate	3
Toluene	0.5
Chloroform	29
Hexane	0
Methylene Chloride	20
1-Butanol	57

2.1.9.1 Extraction from MMD Hydrolysate

A decontamination solution sample was analyzed with the HILIC column using only dilution and pH correction. A peak for EA-2192 with a $M+H^+$ ion at *m/z* 240 was observed. A chromatogram is shown in Figure 2-9. The original MMD hydrolysate was spiked with 40 ppm EA-2192. The pH was adjusted to pH1, and the sample was diluted by about 1:20 in ACN/H2O/MeOH before injection of 10 µL of the diluted sample on the LC/MS. Higher injection volumes, even of the diluted sample, tend to degrade the chromatographic resolution.

This spiked hydrolysate sample was then extracted with methylene chloride or chloroform. EA-2192 was observed in the extract after the hydrolysate solution was saturated with NaCl. A chromatogram of the extract is shown in Figure 2-10. The *m/z* 240 peak can be identified, but it is weak and not strongly reproducible. Although the EA-2192 could be extracted, this approach was not optimized further.

2.1.9.2 Derivatization

In addition to the extraction studies, derivatization of EA-2192 to form the methyl analog of VX was studied. Derivatization of EA-2192 using (trimethylsilyl)diazomethane [2.0 M in hexanes, CAS RN 18107-18-1, purchased from Aldrich, Milwaukee, WI] has been reported.[1,4] This reagent is used for derivatization in sample preparation for GC analysis, so it has the advantage of potentially provided a method for the GC analysis of EA-2192.

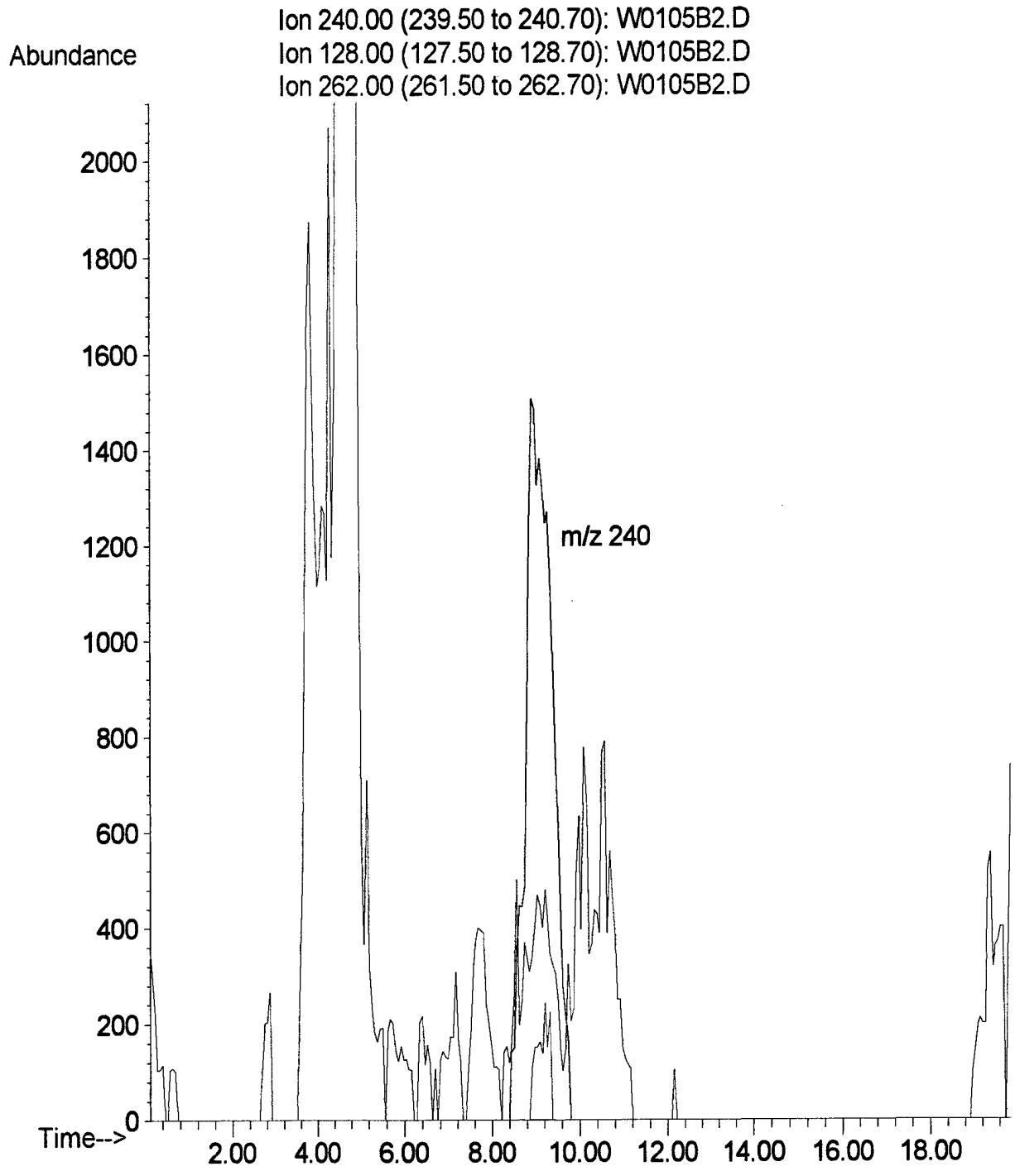

Figure 2-9. LC/MS Chromatogram of MMD Hydrolysate Spiked with 40 ppm EA-2192. The pH was adjusted to pH1, and the sample was diluted by about 1:20 in ACN/H2O/MeOH before injection on the LC/MS.

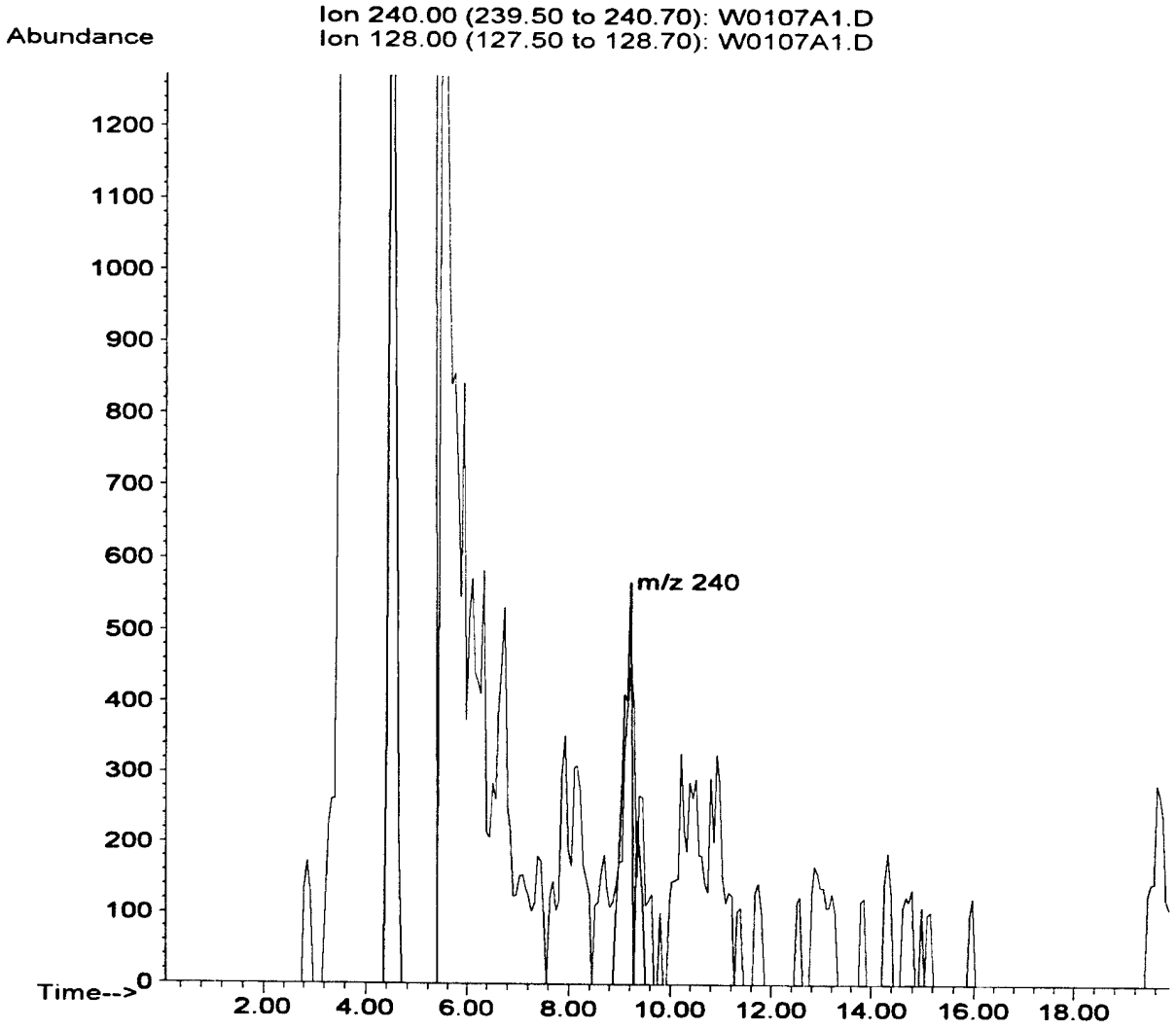

Figure 2-10. LC/MS Chromatogram from the 5989A Instrument of the Methylene Chloride Extract of a MMD Hydrolysate Spiked with 40 ppm of EA-2192. The *m/z* 240 peak can be identified, but it is weak and not reproducible.

The derivative must be prepared in a nonpolar solution of alcohol and a nonprotic solvent. This solution can be run on the HILIC column with the same LC conditions. Thus, the normal phase LC method allows the determination of both the methylated and unmethylated EA-2192 that is present in the extraction and derivatization solution. In a standard solution, it was observed that EA-2192 can be derivatized to 95% efficiency or better with this derivatizing reagent. Figure 2-11 shows a chromatogram of a sample that was only partially derivatized, in order to illustrate that both derivatized and underivatized analyte can be detected in the same sample.

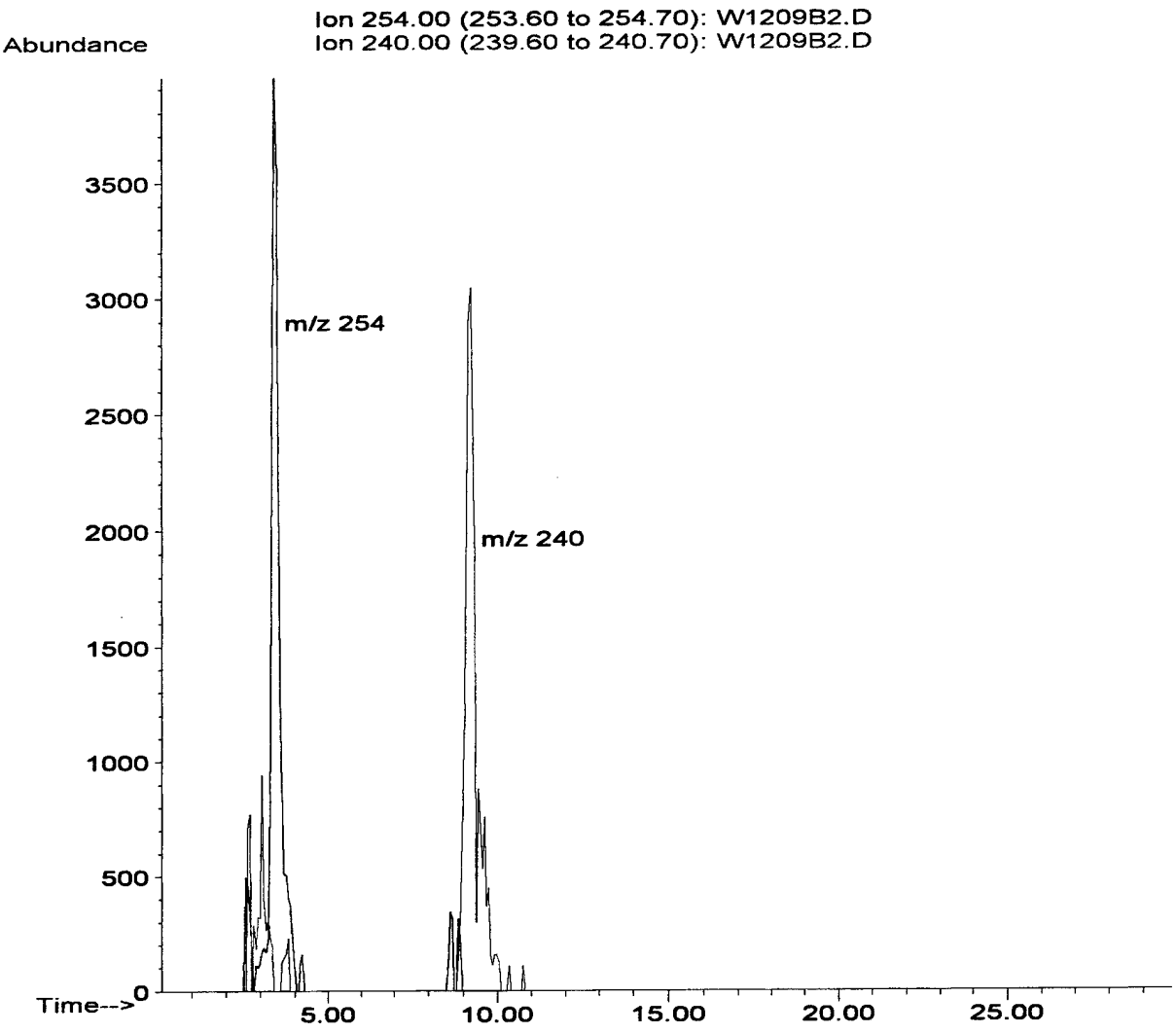

Figure 2-11. LC/MS Chromatogram from the 5989A Instrument of a Sample of Spiked Hydrolysate, Diluted in Acetonitrile, that was Partially Derivatized. The peak for *m/z* 254 is the methyl derivative of EA-2192, and the *m/z* 240 peak is underivatized EA-2192. This chromatogram illustrates that both of them can be detected in the same run.

It is more problematic to derivatize EA-2192 in the hydrolysate or the extract. There are always other compounds in higher concentrations that also react with the reagent, including the MEA that is added in excess as a decontaminating agent. A method for extracting and derivatizing the decontamination solution samples to detect EA-2192 was not found. As a result, there is not a method for screening of EA-2192 by GC methods in decontamination solutions.

2.1.10 Sample Preparation: Solid Phase Extraction (SPE) Method

Another approach that was tested was to use a Solid Phase Extraction method. This method was primarily for sample cleanup and to allow shorter LC chromatographic runs. Since reversed phase chromatography required a gradient elution, a considerable amount of time was spent flushing the column. With sample cleanup, an isocratic run could be used, which reduced the run time from about 60 min. to as little as 10 min. However, extra sample handling and labor was involved. An abbreviated P&A study was done with this method to test the reproducibility.

The approach that was used is as follows:

1. Weigh a quantity of about 0.1 g (100 µL) of decontamination solution matrix.
2. Add 1.0 mL DI water for total dilution of about 1:10, and weigh the solution.
3. Add 100 µL of glacial acetic acid, and weigh. Make sure the solution is a single phase and acidic.
4. Determine dilution of decontamination solution by weight ratio.

For spiked samples, spike a volume of the 4 µg/mL EA-2192 working standard that is appropriate for producing a 1 ppm by weight spike relative to the original decontamination solution. For a dilution which includes 0.1 g of decontamination solution, a spike of 25 µL of the standard give a spike of 0.1 µg of EA-2192.

2.1.10.1 Solid Phase Extraction (SPE):

SPE Cartridges: 3M Empore High Performance Extraction Disk Cartridges #4315 (SD), C18-SD (Octadecyl), (Phenomenex Part # AHO-4058), size 10 mm/6 mL.

Procedure:

1. Condition cartridge with 400 µL acetonitrile, then 3 × 200 µL 95% DI water.
2. Pipet 300 µL of diluted, acidified decontamination solution (spiked if necessary) on cartridge.
3. Elute liquid to waste.
4. Pipet 300 µL of 95% DI water, 5% acetonitrile on cartridge. Elute to sample A.
5. Pipet 300 µL of 95% DI water on cartridge. Elute to sample B.
6. Put elutions in separate autosampler vials with inserts, and run on LC/MSD.

Preliminary results showed that the analyte eluted from the cartridge with sample B, although a small amount of analyte was detected in sample A.

2.1.10.2 Precision and Accuracy Procedure

For this study, an abbreviated P&A procedure was used.

A total of three decontamination samples, spiked after acidification to 1 ppm by weight, were prepared by dilution, then prepared by following the SPE procedure. One blank sample was prepared identically. Each sample was analyzed in quadruplicate on the LC/MSD. Multiple analyses were done to test the reproducibility of the MSD detector and the stability of the samples over time. The twelve analyses were pooled to determine the %RSD and extraction efficiency.

The results of the abbreviated P&A study are given in Appendix B. They are grouped for each prepared sample. The actual order of analysis is given by the file number. The one blank and three samples were run together as a group, followed by a 40 ppb EA-2192 standard as a calibration verification. The group was repeated four times. A set of a solvent blank and four standard solutions in DI water were run before the samples, and also at the end of the samples.

Printouts of the chromatograms are given in Appendix B. Integrals are shown on the chromatograms. Integration was done manually by drawing a baseline, since the instrumental integration parameters were not optimized.

Appendix C shows the autotune report for the MSD, along with the instrumental parameters that were set by the autotune. The electrospray source conditions are part of the instrumental method, so they are different between the tune and the actual sample analysis. This is due to the difference in the solvent used for the tune solution compared to the LC mobile phase.

2.1.10.3 P&A Results

The performance of the LC/MSD was good in this study. The calibration curve correlation coefficient is 0.99, and the signal level is stable. One potential problem is the high signals for the calibration verification standards.

The matrix blank is largely free of interferences at the retention time of EA-2192. There is a small peak for the *m/z* 240 peak in the blank, but it is only about 10% of the signal of the spike. For lower spiking levels, this interference could be a problem. More significantly, the chromatograms show that other peaks with *m/z* 240 are observed which are not far from the EA-2192 peak in retention time. It may be advantageous to find chromatographic conditions in which these peaks are better separated from the analyte peak.

This MSD method only has one ion that is reliable for detecting the analyte. The chromatograms for the standards show a fragment ion at *m/z* 128, but the signal for this ion is considerably smaller than the *m/z* 240 ion signal. In the decon

sample, there are so many other compounds that have a *m/z* 128 peak that the signal is completely obscured, so this ion cannot be used for a confirmation ion. There don't appear to be any other fragment ions that can be used to confirm the analyte signal. It may be necessary to use an LC/MS/MS method, such as with an ion trap detector, to obtain additional confirmation of the analyte beyond the *m/z* 240 ion.

For the sample preparation conditions, the reproducibility is good. The three samples gave similar recoveries, and the overall %RSD is 18%. This is promising, although a full P&A study would be needed to determine the method recovery.

The major question associated with the sample preparation is the reason for the low absolute recovery. From the quantitation results, only 2% of the analyte is detected. The analyte is easily detected even with this low recovery. However, the confidence and detection limits could be improved with a higher recovery. Because of the low recovery, simple dilution of the samples by 1:100 is as effective as the SPE method.

The reason for the low recovery is not clear. It is possible that the analyte is not eluting from the SPE cartridge. However, extra elutions of solvent through the cartridge did not show that much additional analyte is recovered. Another possibility is that the ionization signal is suppressed, so that the signal strength is lower for the LC runs with the sample matrix than it is for the standards. It was observed that other compounds co-elute from the LC column with the analyte. Improving the LC conditions to prevent coelution may prevent the problem.

2.1.11 Conclusion

A number of studies were done to improve the sensitivity of LC/MS for determinations of EA-2192. The best results were found using LC/MS with a Phenomenex Luna C18 column or Polar-RP column, an acidic aqueous mobile phase, and electrospray ionization. An HP LC/MSD with an ESI source improved the detection limits for EA-2192 significantly to low ppm concentrations in the original decontamination solution.

2.2 NMR Method Development: Evaluation of an Established ^{31}P-NMR Method for the Analysis of EA-2192 in VX/Caustic/MEA Neutralents Produced in the MMD-1 Project

2.2.1 Introduction

This section summarizes the experiments examining the application of an established ^{31}P-NMR method for EA-2192. The established method was developed to analyze for EA-2192 in a decontamination solution produced from the reaction of VX

with 20% aqueous NaOH. The sample matrix examined in this study is a neutralent produced from the reaction of VX with a mixture of monoethanolamine (MEA) and 50% aqueous NaOH.

2.2.2 Sample Matrix

The sample matrix utilized in this experiment is an actual VX/NaOH/MEA neutralent produced by ECBC in March of 1996. This neutralization was performed in a glass reactor, using 368 g of VX, 3,370 g of MEA, and 562 g of 50% aqueous NaOH. This is a 47:1 molar ratio of MEA:VX. This neutralent is identified as MRCS-VX-4.4, and was extensively characterized by multiple chromatographic and spectroscopic techniques shortly after it was produced. This neutralent has been stored at ambient conditions, in a glass bottle, since it was generated. There were no visual signs of degradation observed, compared to what the neutralent looked like when it was originally produced. A summary of the bulk composition (of P containing components) of this sample when it was originally produced is included as Table 2-3. Additionally, analysis by LC/MS/MS at the time the neutralent was produced indicated this sample contained 14±7 μg/g of EA-2192.

Table 2-3: Quantitative ^{31}P-NMR Results from the Analysis of MRCS-VX-4.4.

Chemical Name	Abbreviation	CAS Number	Weight Percent with Error Range (%)
Ethyl methylphosphonic acid	EMPA	1832-53-7	1.70±0.02
O-(2-amino)ethyl methylphosphonic acid	AEMPA	NA[1]	1.55±0.04
Methylphosphonic acid	MPA	993-13-5	0.34±0.01

[1] The CAS number is not currently available.

2.2.3 Experimental Conditions

The NMR method is based on the property of nuclear spin. In a strong magnetic field, nuclei such as ^{1}H, ^{31}P, and ^{13}C with an odd spin (magnetic moment = 1/2) will segregate into two energy levels, with a slight population excess in the lower energy level. This produces a bulk sample magnetization which is aligned with the static magnetic field (B_0), and like a gyroscope in a gravitational field, the individual spinning nuclei and the vector which represents the total population of excess nuclei aligned with the magnetic field, precess around the axis of B_0. When this equilibrium magnetization is perturbed by the application of a radio frequency pulse, the sample magnetization is tipped away from B_0, usually referred to as the Z axis. Each type of nucleus precesses at a specific frequency (the Larmor frequency), which is a function

of the bulk magnetic field strength and intrinsic properties of that nucleus. Additionally, each nucleus will precess at a slightly different frequency depending on the chemical environment of the sample matrix. After a radio frequency pulse which induces precession is applied to the sample, the specific frequency of each nucleus is detected as it returns to equilibrium, and is plotted as a function of frequency. From this data, the structure of the various components in the sample can be inferred. Definitive determination of structure requires additional spectroscopic data (such as mass spectrometry), and/or spiking of the sample with an authentic standard of the target compound.

The experiments summarized in this report followed the procedures outlined in a report entitled "Nuclear Magnetic Resonance (NMR) Procedure for the Characterization of Products from the VX/NaOH Demilitarization Process" published by ECBC (Linda L. Szafraniec and William T. Beaudry, ERDEC-TR-481, March 1998). The analyses were conducted on a Bruker Avance 300 NMR spectrometer equipped with a 5 mm quattro nucleus probe (QNP). Data was acquired with a pulse length of 7.5 μsec, and an acquisition delay of 96 seconds. The pulse power was 0.0 dB, and the line broadening was 1 Hz. Each analysis took approximately 18 hours to complete.

Prior to sample analyses, instrument performance was monitored using a 0.1% ethylbenzene solution in $CDCl_3$. The signal-to-noise ratio (SNR) was measured after tuning and shimming the instrument. The SNR should exceed 125:1 for the ^{1}H NMR spectrum, although this target may be modified based on past performance of the instrument. A 0.1% solution of triphenylphosphate in D_2O was used to determine the instrument performance of the QNP probe. The SNR should exceed 30:1 for the ^{31}P NMR spectrum.

A standard of EA-2192 was prepared by placing 600 μL of D_2O into a vial, then adding 38 μL of 400 μg/ml EA 2192 stock solution (stock solution prepared in 2-propanol), and 36 μL of 2-propanol. The sample was then mixed, and transferred to a 5 mm NMR tube. This standard is 25 μg/ml EA-2192, and contains 12% (v/v) of 2-propanol.

The unspiked neutralent sample was prepared by adding 600 μL of neutralent into a vial, adding 2 drops of D_2O as a lock solvent, then adding 75 μL of 2-propanol. The sample was then mixed, and transferred to a 5 mm NMR tube. The spiked neutralent sample was prepared by adding 600 μL of neutralent into a vial, adding 2 drops of D_2O as a lock solvent, then adding 400 μg/ml EA-2192 stock solution (stock solution prepared in 2-propanol). The sample was then mixed, and transferred to a 5 mm NMR tube. In both the unspiked and spiked neutralent, each sample contained 12% (v/v) of 2-propanol, which matches the amount of 2-propanol contained in the EA-2192 standard.

2.2.4 Results

Sample spectra from the instrument performance check procedures are attached as Figures 2-12 and 2-13. Instrument performance exceeded the required SNR's for both the ^{1}H and ^{31}P NMR spectra.

The spectra obtained from the analysis of a 25 µg/ml EA-2192 standard in D_2O is attached as Figure 2-14. The EA-2192 peak is visualized at a chemical shift of 43.778 ppm, with an SNR of approximately 4:1.

The spectra obtained from the analysis of unspiked neutralent is attached as Figure 2-15. There is a peak at a chemical shift of 43.312 ppm, with an SNR of approximately 13:1. This is the only peak in the chemical shift region where EA-2192 is observed in the standard solution. The spectra obtained from the analysis of neutralent spiked to 50 µg/ml EA-2192 is attached as Figure 2-16. There is a peak at a chemical shift of 44.343 ppm, with an SNR of approximately 13:1. This is the only peak in the chemical shift region where EA-2192 is observed in the standard solution. These results indicate the analysis of EA-2192, at a level of 50 µg/ml, is not possible in this matrix using the referenced method.

Given the wide chemical shift range typically observed for EA-2192 as a function of pH, the peak in the neutralent sample for EA-2192 shifted relative to the standard solution (see next section). The 25 µg/ml EA-2192 standard analyzed by ^{31}P NMR yielded a peak with an SNR of 4:1, while the peak in the neutralent samples had an SNR of 13:1. Additional evidence suggesting the peak observed in the neutralent is not EA-2192, is that the peak did not increase when the neutralent was spiked with EA-2192 to a level of 50 µg/ml.

The established ^{31}P-NMR method used to analyze this sample matrix is not suitable for quantitative analysis of EA-2192 in this sample matrix at the target action level of 50 µg/ml. The technique of NMR is best suited to quantitation of bulk (>0.1 wt%) components, and to structural elucidation of bulk components. Although trace analysis can be performed by NMR, the technique is slow and insensitive, relative to chromatographic techniques such as LC/MS. The analysis of EA-2192 by NMR presents a particular challenge due to the large variability of its chemical shift, depending on the solvent utilized, and the sample matrix.

2.2.5 Additional NMR Studies

One of the problems with the previous NMR study was that old decontamination solution was used. In the solution, many low concentration phosphorus containing compounds were formed over time by slow reactions. These extra compounds made it difficult to positively identify the EA-2192 peak.

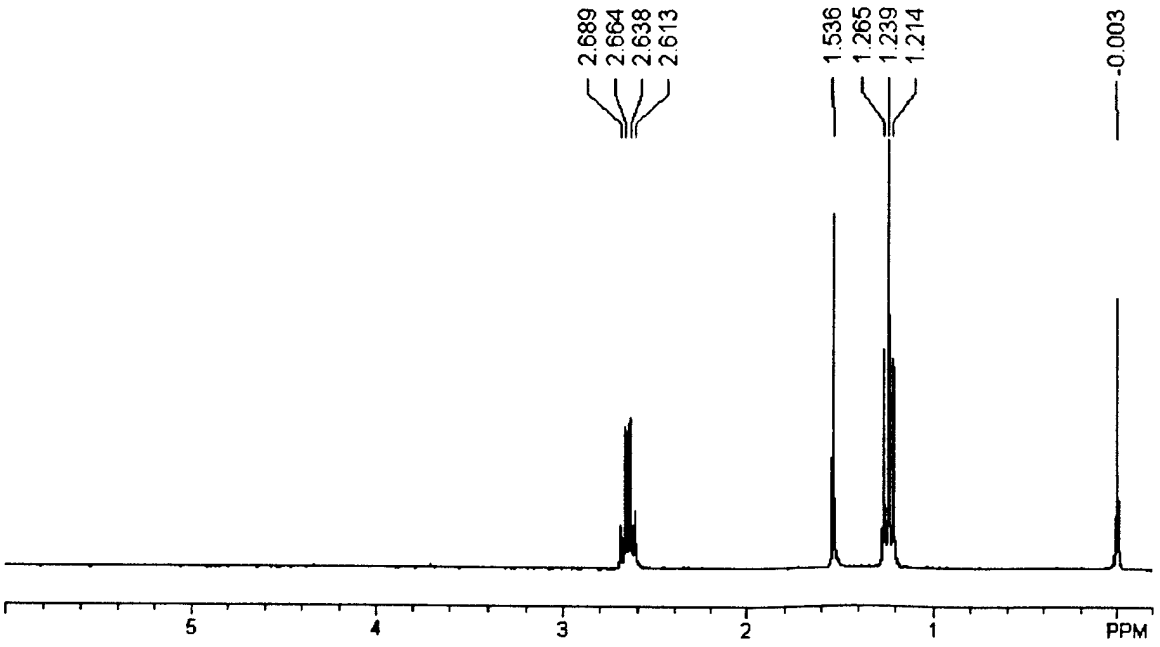

Figure 2-12. ^{1}H NMR Spectrum of the 0.1% Ethylbenzene Performance Check Standard. The SNR was determined to be 141:1.

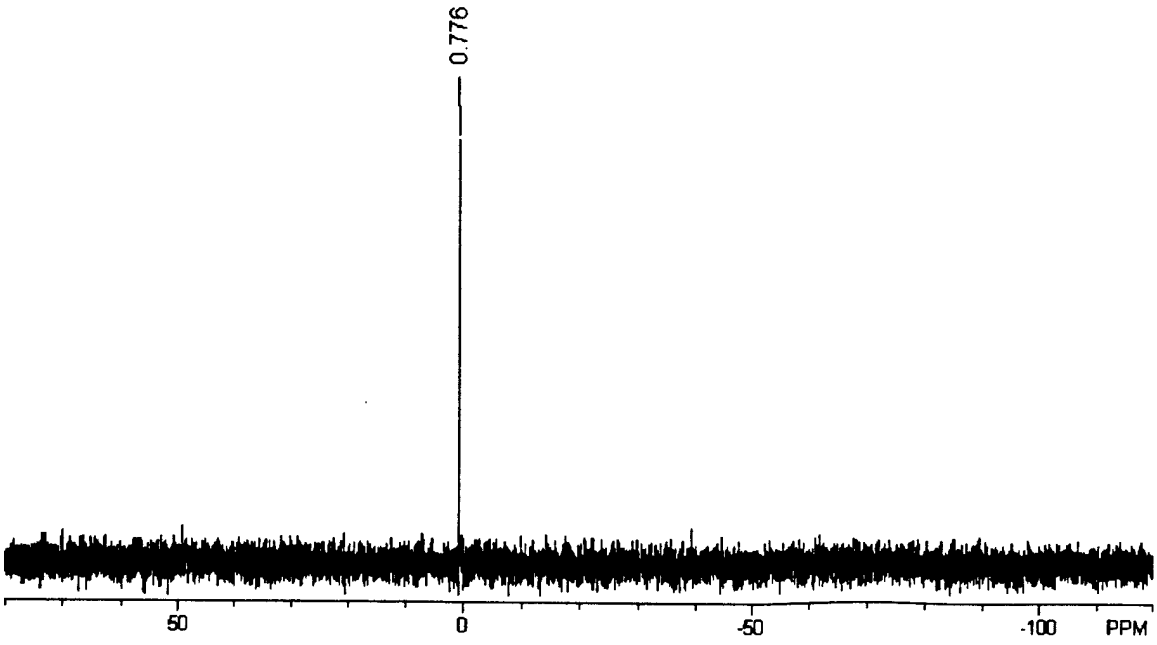

Figure 2-13. ^{31}P NMR Spectrum of the 0.1% Triphenylphosphate Performance Check Standard. The SNR was determined to be 33:1.

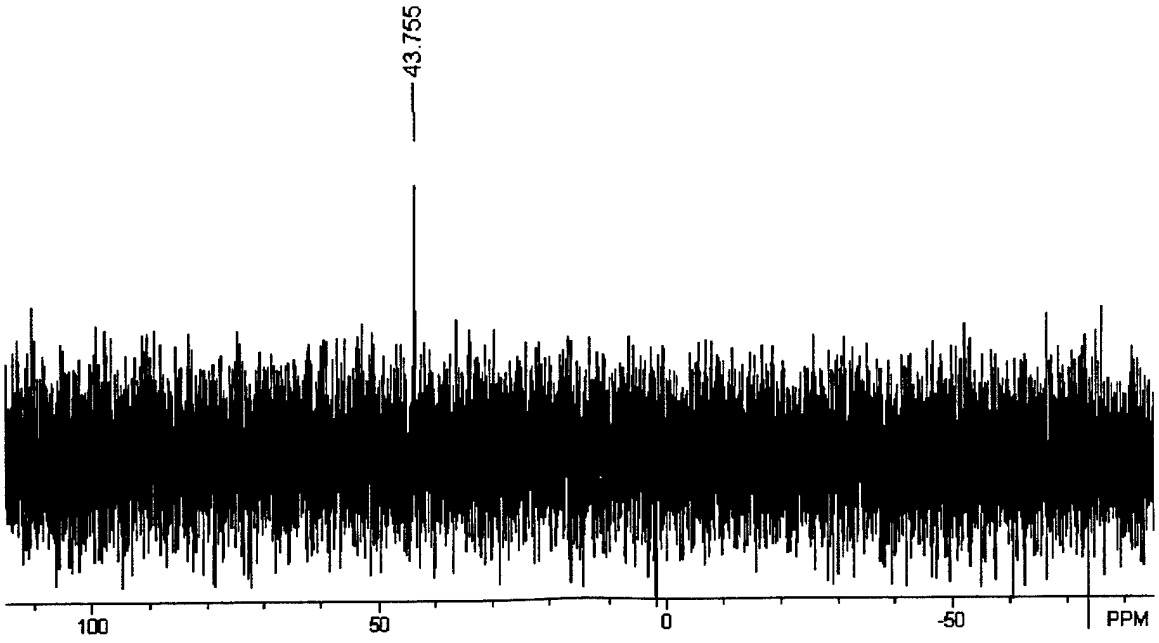

Figure 2-14. ^{31}P NMR Spectrum of the 25 µg/ml EA-2192 Standard in D_2O. The SNR was determined to be 4:1.

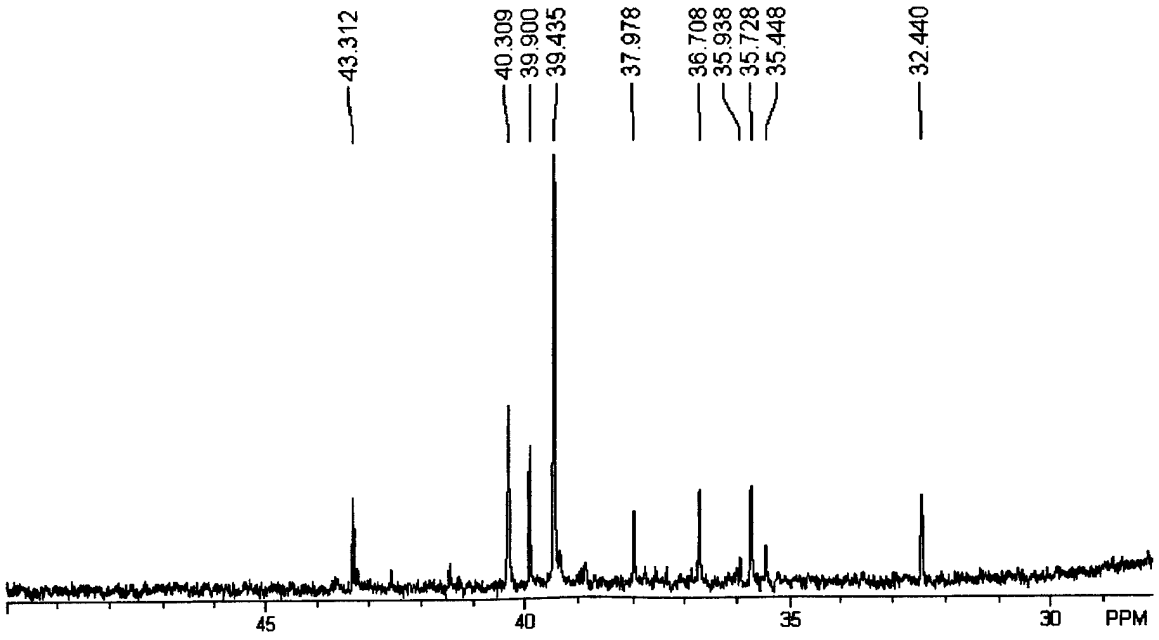

Figure 2-15. ^{31}P NMR Spectrum of the Unspiked Neutralent. The peak (at a shift of 43.312 ppm) in the chemical shift region of EA-2192 was determined to have an SNR of 13:1.

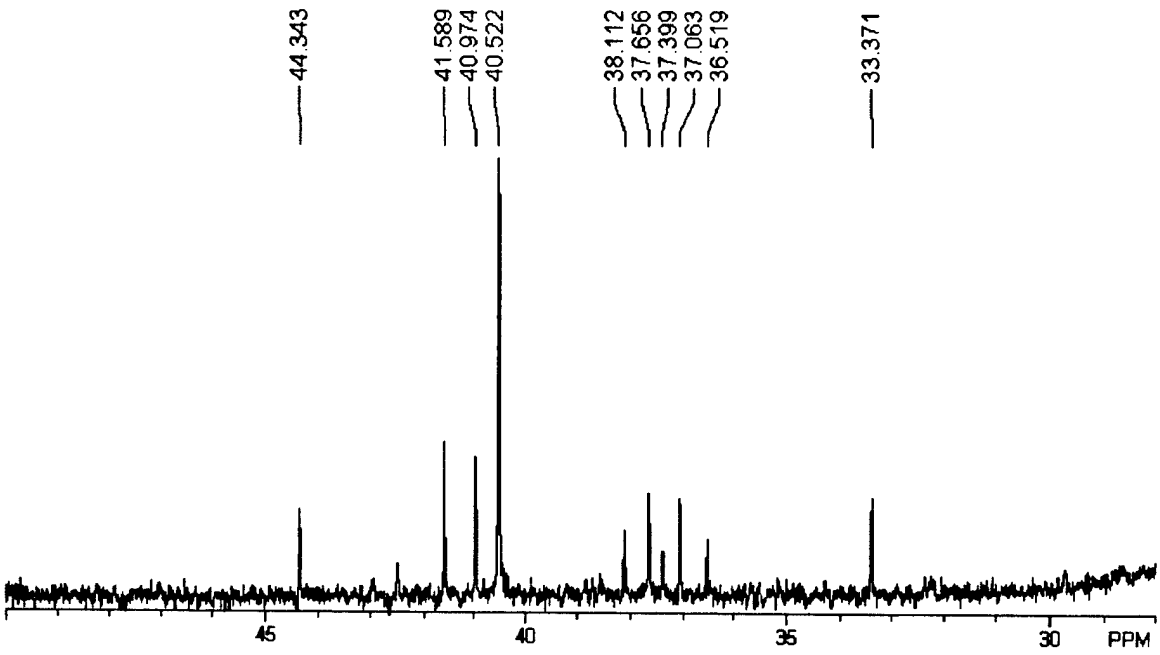

Figure 2-16. ^{31}P NMR Spectrum of the Neutralent Spiked to 50 µg/ml EA-2192. The peak (at a shift of 44.343 ppm) in the chemical shift region of EA-2192 was determined to have an SNR of 13:1. There are shifts in the peaks that are between 35 to 40 ppm.

During the kinetic runs that were done on EA-2192 (see section 3.0), some new decontamination solutions were generated that were much cleaner. A solution was spiked with EA-2192 to measure the reaction rate. Because the solution contained many fewer phosphorus compounds, it was possible to unequivocally identify the EA-2192 peak. It was assigned to a chemical shift of 38.1 ppm, which was considerably lower that that expected from the standard solution. The chemical shift changed due to the different pH of the decontamination solution compared to the standard solution.

Figure 2-17 shows the expanded view of the spectrum. The EA-2192 concentration was about 100 µg/mL. No studies were done to determine a detection limit for this solution.

2.3 Hazard Screening Method for EA-2192 Detection

The hazard characteristic screening approach can be used to screen decontamination solutions to provide additional confidence that the hazard (CHE inhibition) has been reduced to acceptable levels.

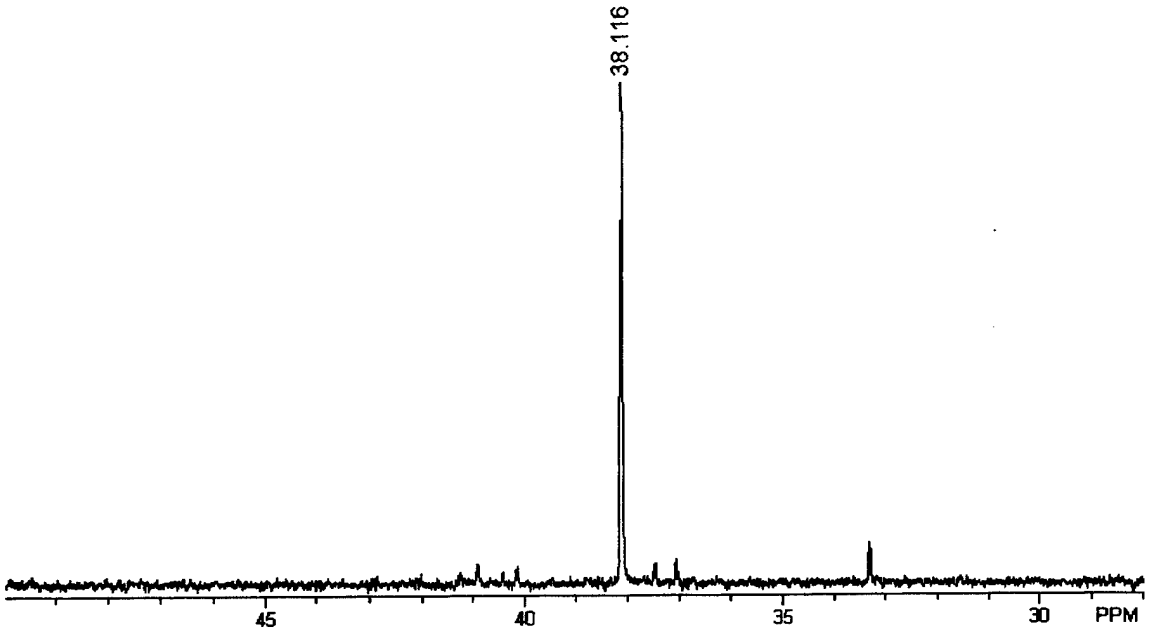

Figure 2-17. Expanded Section of a ^{31}P NMR Spectrum of the New MMD Reaction Matrix Neutralent Solution Spiked to 100 μg/ml EA-2192.

Five different reactor runs (MEA/NaOH chemistry) were analyzed for residual EA-2192 approximately one month after the reactions took place.[1] The samples were stored at room temperature prior to analysis. Analysis was by LC/MS/MS, with quantitation by the external standard approach. The EA-2192 concentrations ranged from 6 to 36 μg/g (ppm) in the original hydrolysate sample. The method utilized to analyze these samples has not been rigorously validated with this sample matrix.

The hazard screening approach for analyzing both VX and EA-2192 has been successfully validated for screening hydrolysates to below the RDT&E level (1,000 μg/g) in the VX/caustic hydrolysates produced in the MMD-1 program.[5,6] This approach is based on the inhibition of butyrylcholinesterase by VX and EA-2192. Measurement of the inhibition is based on simple colorimetric procedures.

Recent experiments have indicated this approach (with minor variations) can also be utilized to screen these hydrolysates to below 50 μg/g (ppm) VX. However, EA-2192, up to 100 μg/g (ppm), will not be visualized with this approach. The response of VX is non-linear, but reproducible. An example of the VX response curve is illustrated in Figure 2-18. Assuming the background inhibition in reactor hydrolysate is low, it is anticipated that a Class II P&A at a spike level of 40 μg/g (ppm) VX would be successful.

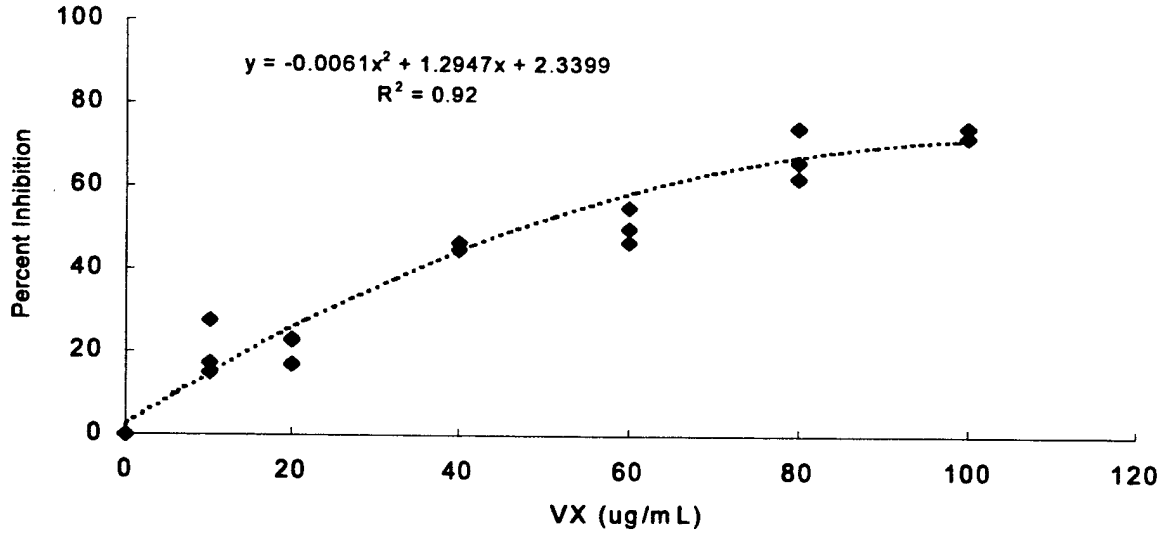

Figure 2-18. Response Curve for VX Inhibiting Butyrylcholinesterase. Each concentration was evaluated in triplicate.

An experiment was conducted to compare three different cholinesterase enzymes, to determine the way both VX and EA-2192 inhibited them. In all cases, the spike level was 50 µg/g (ppm) for both VX and EA 2192. These results are illustrated in Figure 2-19. The negative percent inhibition values are indicative of V_{max} values not significantly different from the unspiked samples. Note that only the bovine acetyl enzyme responded to EA-2192.

The following approach might enable EA-2192 to be distinguished from VX in these hydrolysate samples:

- Add the butyryl enzyme to the sample, and incubate. This should bind the VX, and any residual fluoride.
- Add the bovine acetyl enzyme, and the acetylthiocholine substrate, then incubate again.
- Visualize with Ellman's reagent, and measure V_{max}.

This approach would need to be demonstrated, then validated. If the approach worked, it appears likely that a Class II P&A at a spike level of 40 µg/g (ppm) EA-2192 would be successful.

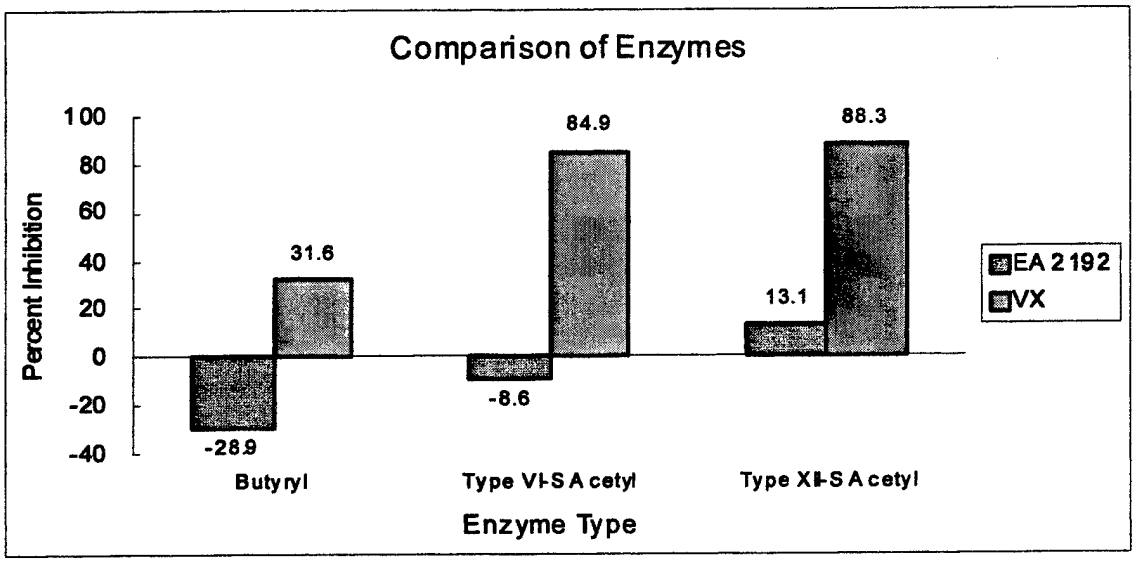

Figure 2-19. Comparison of the Inhibition of EA-2192 and VX on Three Different Enzymes. Each agent/enzyme combination was evaluated in triplicate.

3.0 KINETIC DETERMINATIONS OF VX AND EA-2192 DESTRUCTION IN DECONTAMINATION OF VX IN MEA DECONTAMINATION SOLUTIONS FOR THE MMD PROJECT

3.1 Summary

In accordance with the plan agreed to by PMNSCM, the following decontamination runs were done:

a) 4 kinetic runs using 1% by volume of VX in MMD decontamination solution (81% monoethanolamine, 9% water, 10% of 50% NaOH aqueous solution). The runs were done at 50-55°C.

b) 4 kinetic runs using 5% by volume VX in MMD decontamination solution. The runs were done at 50-55°C. The mixtures were sampled about every hour.

c) 4 kinetic runs using worst case estimated concentration of EA-2192 in MMD decontamination solution (81% monoethanolamine, 9% water, 10% of 50% NaOH solution). Two runs began with 1000 ppm EA-2192, and two began with 2000 ppm EA-2192. The runs were done at 50-55°C. The mixtures were sampled about every hour.

d) A few trial runs were done at an elevated temperature of 70°C.

e) Runs were done at ambient temperature, at 20-25°C. These runs were sampled about once a week for LC/MS analysis. Another run was analyzed by NMR about once a week. Results from these two analytical methods are in good agreement.

For analysis, the samples were diluted by 1:100 in 10% acetic acid/water, and analyzed by the optimized method for LC/MS. The mixture was analyzed for residual VX and EA-2192.

In all cases except the ambient temperature runs, the VX and EA-2192 were below the target concentration of 50 ppm in 200 min. or less. The EA-2192 reaction was much slower than the VX reaction rate, and it has a half-life of 50-70 min. at 50°C.

Preliminary experiments included test runs that were done at higher temperature. For those runs, a different lot of VX was used. There are some notable differences between the lots of VX that have been studied, and these differences will be pointed out in Section 3.3. However, the main conclusions of the study are not affected by the different lots of VX.

3.2 EA-2192 Results at 50°C

3.2.1 Runs of 1% VX by Volume in Decontamination Solution

Figure 3-1 shows the data for the log of the concentration of EA-2192 as a function of reaction time for the four 1% VX runs.

The slope is plotted for the first four points of the 2/26 run. The slopes for the rest of the runs are noisier, but they appear to be faster rates. The 2/26 run is the slowest (lowest slope), probably because the temperature was slightly lower. The slope is -0.0043 on the log plot for the slowest run. The average slope for all four runs is -0.00572 ± 0.0010 (RSD: = 17.7%).

The highest concentration of EA-2192 that was measured for these runs is 70 ppm, as measured 5 min. after mixing the reagents. The EA-2192 arises in part from impurity in the VX, and in part from formation of EA-2192 in the decontamination reaction from the VX. In all cases, the concentration was below 50 ppm within the first hour of the reaction.

3.2.2 Runs of 5% VX by Volume in Decontamination Solution

Figure 3-2 shows the data for the concentration of EA-2192 as a function of reaction time for the four 5% VX runs at 50-55°C.

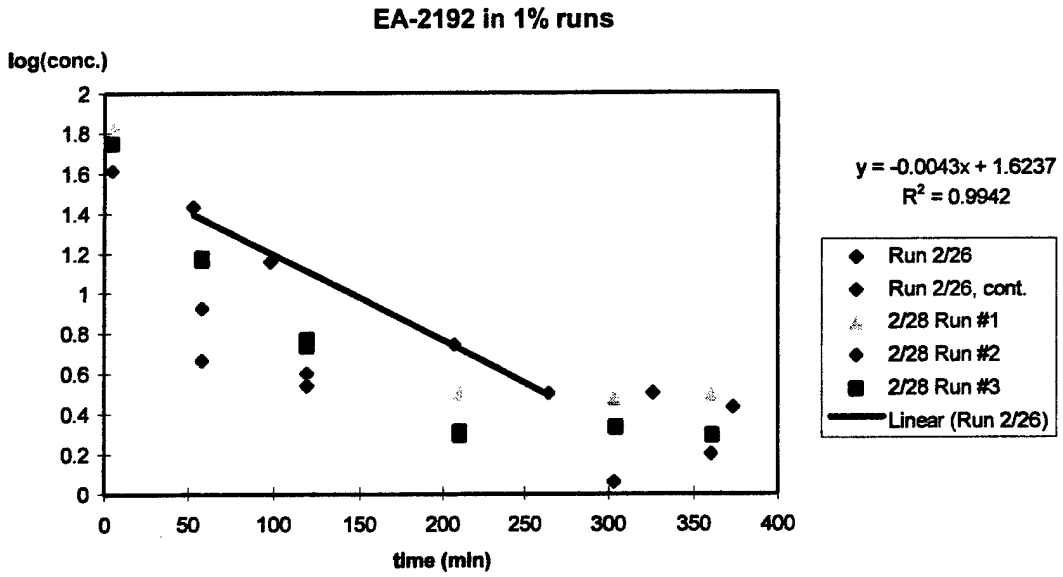

Figure 3-1. Log of EA-2192 Concentration as a Function of Time for Reaction Runs of 1% VX by Volume in the Decontamination Solution. The trend line is for the first four points of the 2/26 run.

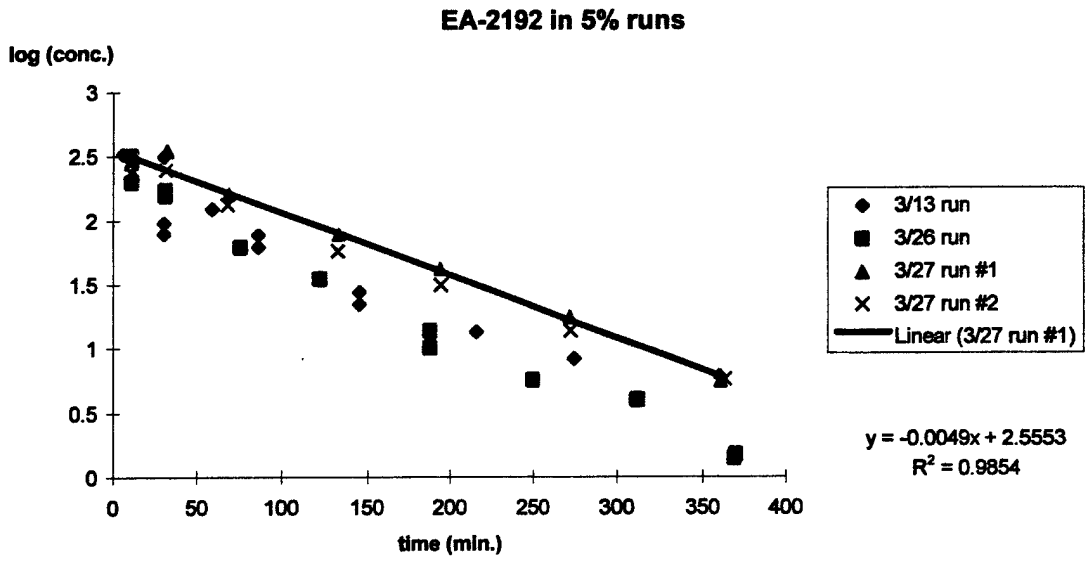

Figure 3-2. Log of EA-2192 Concentration as a Function of Time for Reaction Runs of 5% VX by Volume in the Decontamination Solution. The trend line is for the 3/27 run #1.

The trendline is for the 3/27 run #1, which is the slowest run. For all four runs, the average slope is -0.00515 ± 0.00064 (%RSD = 12%). These results are statistically the same as the results for the 1% VX runs.

The intercept of the plots increases as a function of the date of the run. This increase is probably due to extra hydrolysis of the VX standard from exposure to air due to the handling. The hydrolysis produces a higher initial concentration of EA-2192.

The data in Figure 3-1 and the data for the 3/13 run in Figure 3-2 show a leveling off at long times, so that there appears to be a residual amount of EA-2192 at long reaction times. This result is an analytical artifact. There was a small interference in the LC/MS chromatogram which overlapped with the peak for EA-2192. After 3/13, a different LC column was used which could resolve the EA-2192 from the interference. As a result, the EA-2192 concentration continues to decrease in a linear trend for the 3/26 and 3/27 data, as expected. Regardless of the interference, the apparent amount of EA-2192 which remains is much less than the target concentration of 50 ppm (1.699 on the log scale).

3.3 VX Results at 50°C

The concentration of VX was analyzed for these runs using the same LC/MS procedure. Most of the VX is destroyed in 5-15 min., which was also observed in the trial runs using the other lot of VX. Because of these preliminary results, the analytical method was not validated for VX detection, although the detection of VX was similar enough to detection of EA-2192 that the calibration validation using EA-2192 is expected to be applicable to VX.

However, unlike the preliminary runs using the first lot of VX, a second lot was used for the reaction kinetics runs. For these runs, a background signal of a residual amount of VX could be detected for considerably longer after the beginning of the run for the 50-55°C runs. The VX signal does not appear to be an interference.

Figure 3-3 shows the log of the concentration of VX for the 1% VX runs. Figure 3-4 shows the log of the concentration of VX for the 5% VX runs.

The measured VX concentration does not have a strong correlation with an exponential decrease, which was observed for the EA-2192 data. The VX concentration decreases to 1 ppm consistently by 200 min. (On the plot, 0 corresponds to a concentration of 1 ppm, and 1 corresponds to 10 ppm.)

It is not certain at the present time why this residual VX is observed. It is possible that this VX is observed as the result of an analytical artifact from acidifying the samples before analysis. It has been previously reported that acidification of MMD samples produces a signal corresponding to VX.[6] In any event, this residual VX is

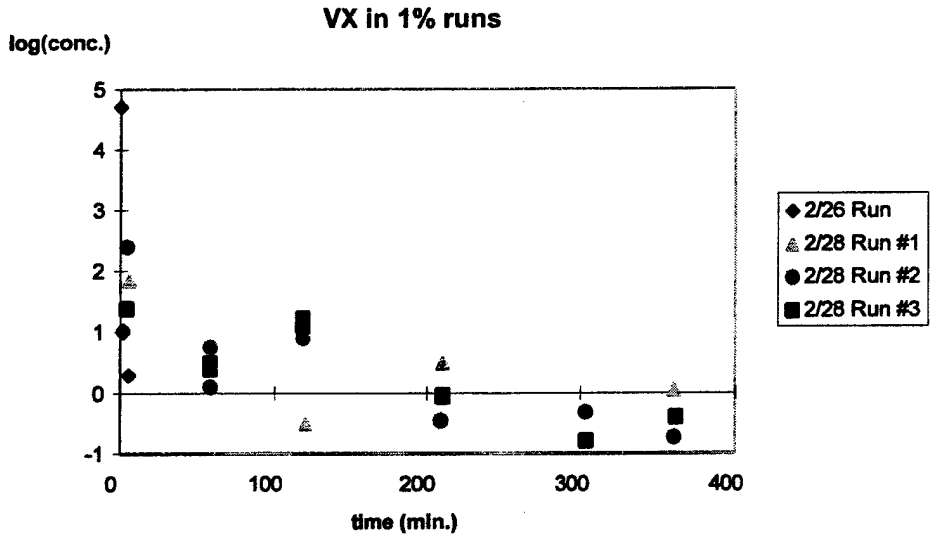

Figure 3-3. Log of the VX Concentration as a Function of Time, Starting with 1% by Volume of the VX in the Decontamination Solution.

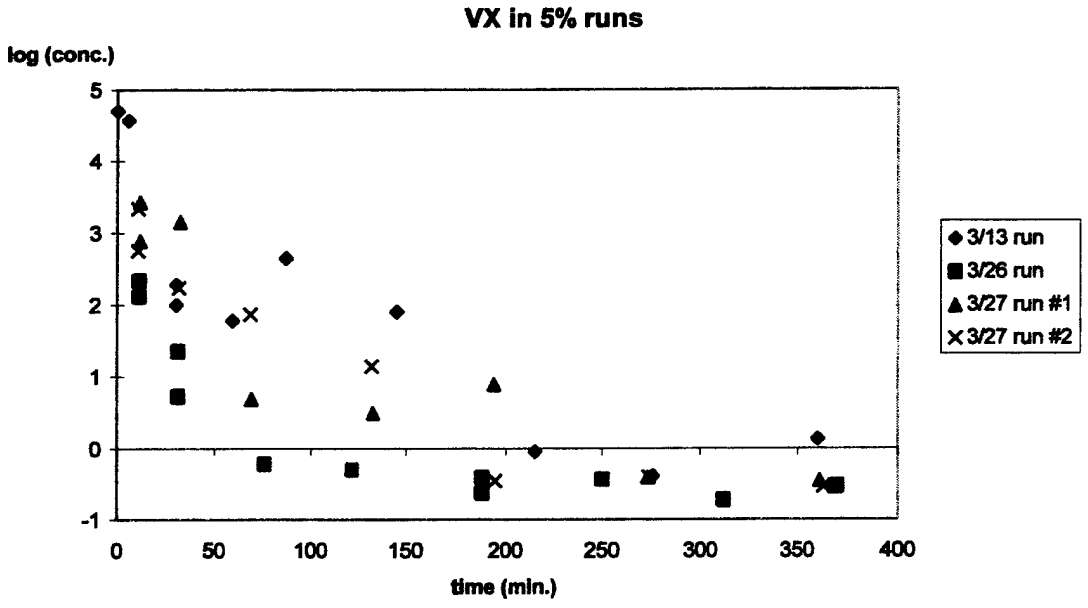

Figure 3-4. Log of the VX Concentration as a Function of Time, Starting with 5% by Volume of VX in the Decontamination Solution.

much less than the target concentration of 50 ppm. This artifact may be a result of the presence of a chemical stabilizer in the VX which was not present in the lot of VX that was used for the preliminary studies.

3.4 Determinations of 1000 ppm and 2000 ppm EA-2192 in MEA Decontamination Solutions (worst case)

Four kinetic runs were done using an estimated worst case concentrations of EA-2192 in MMD decontamination solution (81% monoethanolamine, 9% water, 10% of 50% NaOH solution). Two runs began with 1000 ppm EA-2192, and two began with 2000 ppm EA-2192. The runs were done at 50-55°C. The mixtures were sampled about every hour. The samples were diluted by 1:100 in 10% acetic acid/water, and analyzed by LC/MS. The mixture was analyzed for residual VX and EA-2192.

In all cases, the EA-2192 concentration was below the target concentration of 50 ppm in 400 min. or less. The EA-2192 reaction has a half-life of 56 min., which is consistent with previous measurements.

3.4.1 Worst Case Estimate

A worst case scenario can be developed as follows. Literature results indicate that if VX is decontaminated with aqueous caustic solution, the VX is converted to 10-20% EA-2192.[7,8] It seems unlikely that munitions that are full of purified EA-2192 would be encountered, so EA-2192 from decontaminated VX is likely to be the source of the highest concentration of EA-2192. As a result, a solution that contains 10-20% EA-2192 would be approximately the highest concentration of EA-2192 that would have to be decontaminated.

If 1% VX (or other munition contents) is added to the decon solution, and the maximum concentration of EA-2192 in the munition is 10%, then the concentration of EA-2192 in the decontamination solution after dilution would be 0.1%=1000 ppm.

3.4.2 Results

Figure 3-5 shows the data for the concentration of EA-2192 as a function of reaction time for the four runs at 50-55°C. Two runs on 4/9 began at 1000 ppm concentrations, and two runs on 4/11 began at 2000 ppm concentrations. Trendlines for all four runs are shown on the figure.

In order to perform these runs, a stock solution of 10% EA-2192 in isopropanol was prepared from neat, solid EA-2192. This stock solution was then diluted in the decontamination solution at time = 0 of the kinetic runs. No VX was added to the decontamination solution, and none was observed in the LC/MS analysis runs.

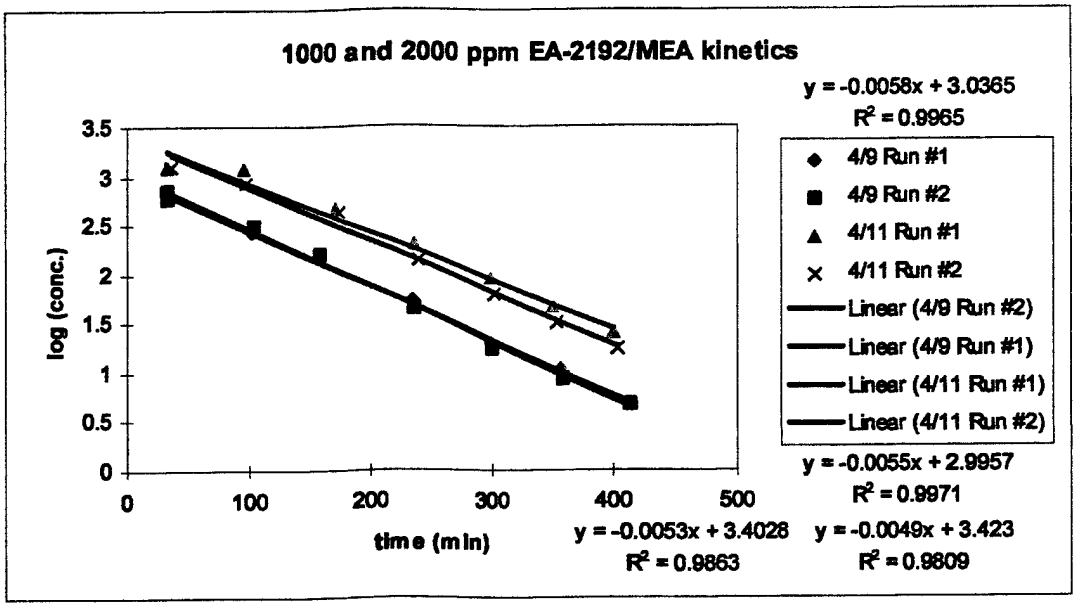

Figure 3-5. Log of EA-2192 Concentration as a Function of Time. The trend lines for all the runs are shown.

For the four runs, the average slope of the trendlines is -0.00537 ± 0.0038 (RSD 7.1%). The linearity of the plots is good. These results give a half-life for the reaction of 56.1 min. Even for the runs that start at 2000 ppm, the concentration of EA-2192 is less than 50 ppm by 400 min. (6.6 hours).

For comparison, the trendline for the 3/27 runs with 5% VX reagent gives an average slope of -0.00515 ± 0.00064 (%RSD = 12%). This slope is statistically identical to the latest runs.

Further runs can be done if needed to improve the knowledge base about this reaction.

3.4.3 Calculations of the Rate of Decrease of EA-2192 Concentration

It isn't known how much EA-2192 will be present in the actual VX that will be processed by the MMD project. A worst case scenario can be assumed for which 1000 ppm of EA-2192 is present in the decontamination solution at time = 0. The kinetics for this scenario were measured, as shown in the previous section.

If a different concentration of EA-2192 is present, it is possible to use the slope to calculate the approximate time required for this amount of EA-2192 to decrease to 50 ppm. A concentration of 1000 ppm is used for the sample calculation.

Let $y = \log(\text{conc. EA-2192}) = \log(1000) = 3$
for $x = \text{time} = 0$

So the loss of EA-2192 is given by the formula,

$$Y = -0.00537\,x + 3$$

Starting the reaction with a different concentration changes the intercept of the line, but not the slope. (This rate equation is appropriate for pseudo-first order kinetics, for which the concentration of MEA is in great excess compared to the concentration of EA-2192. At high enough concentrations of EA-2192, the rate would become nonexponential, and the loss of EA-2192 would become slower than the results indicated by this equation.)

Then the formula can be used to determine the amount of time required for the concentration to decrease to 50 ppm, for $y = \log(50) = 1.699$. Solving for time:

$$X = (y-3)/(-0.00537)$$
$$= 242 \text{ min.} = 4.0 \text{ hours}$$

So a concentration of 1000 ppm will be reduced to 50 ppm in 4 hours. For a higher concentration of EA-2192, the length of time is longer corresponding to the logarithmic function.

3.5 Ambient and Higher Temperature Reaction Studies

The first preliminary experiments were done to study the kinetics of the decrease in the amount of EA-2192 in MMD decontamination solution as a function of time. Experiments were done at ambient temperature, about 25°C, and at a higher temperature than that specified for the MMD-1 reactor, about 70°C. They were done with a slightly different decontamination solution composition than specified for the MMD reactor. The decontamination solution was 90% MEA with 10% by volume of 50 weight % NaOH in water solution. (The later runs were done using 90% of a 90/10 MEA/water solution, and 10% of 50 wt.% NaOH solution.) VX was added at a level of 1% by volume. Concentrations were measured using LC/MS and ^{31}P NMR.

Even at room temperature, the VX was below 50 ppm in 15-30 minutes. The kinetics for VX were so fast that it was difficult to determine accurate kinetic parameters using these experimental methods. At 70°C, the VX was less than 50 ppm in less than 5 min. As discussed previously, a different lot of VX was used for which the VX was completely eliminated, and no residual amounts of VX were detected at long times.

The EA-2192 concentration decreased much more slowly than the VX concentration. Figure 3-6 shows a plot of the log of the EA-2192 concentration vs. time in minutes after addition of VX. This run was done at 70°C (158°F). The slope of the

1/30 run trendline is -0.0037. The EA-2192 decreases below 50 ppm between 140 and 185 minutes. From the trendline, the concentration crosses 50 ppm at 144 min. This data is from only one run on 1/30 that went for 6 hours.

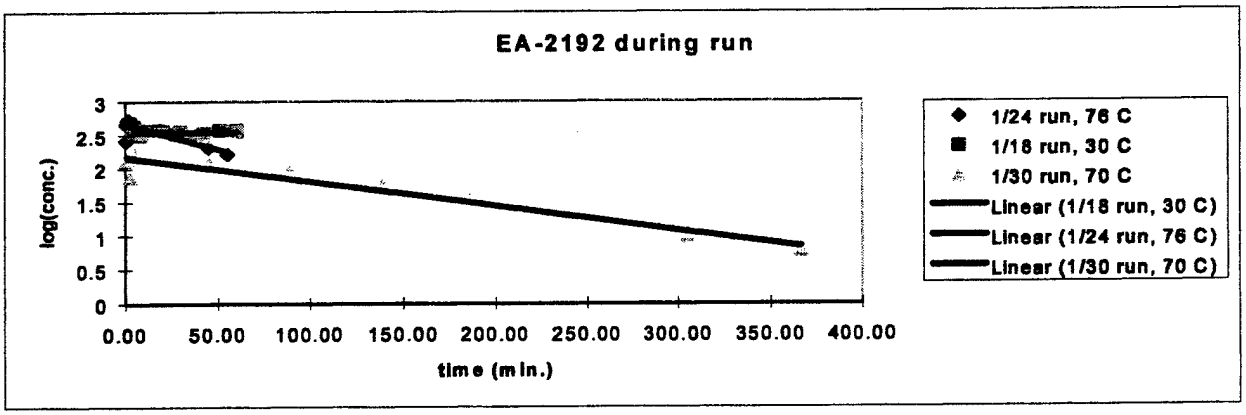

Figure 3-6. Log of the EA-2192 Concentration vs. Time, in Minutes. The runs on 1/24 and 1/30 are at elevated temperature, and the run on 1/18 was at room temperature. On the log scale, 50 ppm corresponds to 1.69 on the y-axis.

A preliminary run on 1/24, also shown in Figure 3-6, shows a similar trend, but it was only followed for one hour. Both of these runs show a rapid increase in EA-2192 concentration at short times as it is formed from the VX. The run on 1/18 was done at room temperature, at which virtually no change was observed in one hour.

Runs at room temperature were done to determine the stability of EA-2192 in the solutions in ambient storage conditions. At room temperature (about 20-25°C) the decrease was slow, as shown in Figure 3-7. The time required to decrease below 50 ppm was 300-400 hours, or 12-16 days. The data is noisy because it was taken on different days without recalibrating the instrument, and the LC/MS measurements had some variation. From the trendline for the 1/5 run only, the slope of the linear fit is -0.00189 (time units in hours) or -0.045 (time units in days), so the concentration of EA-2192 reaches 50 ppm at 333 hours, or 13.9 days.

The 40 ppm data on the figure is for a decontamination solution that was spiked with 40 ppm of EA-2192 but no VX. This solution shows a similar rate of decrease to the VX decontamination runs.

A similar study was done using detection by NMR of the EA-2192 peak. A sample of decontamination solution was spiked to about 125 μg/mL and followed for two months. The data is shown in Figure 3-8. The slope that was obtained for the linear fit in this case was -0.0139, in units of days. This result is about a factor of 3 times slower than the result by LC/MS, which may be due to slight differences in the storage temperature of the samples or the solution composition. This solution had 30% added isopropanol, which was necessary to decrease the viscosity of the solution for NMR analysis.

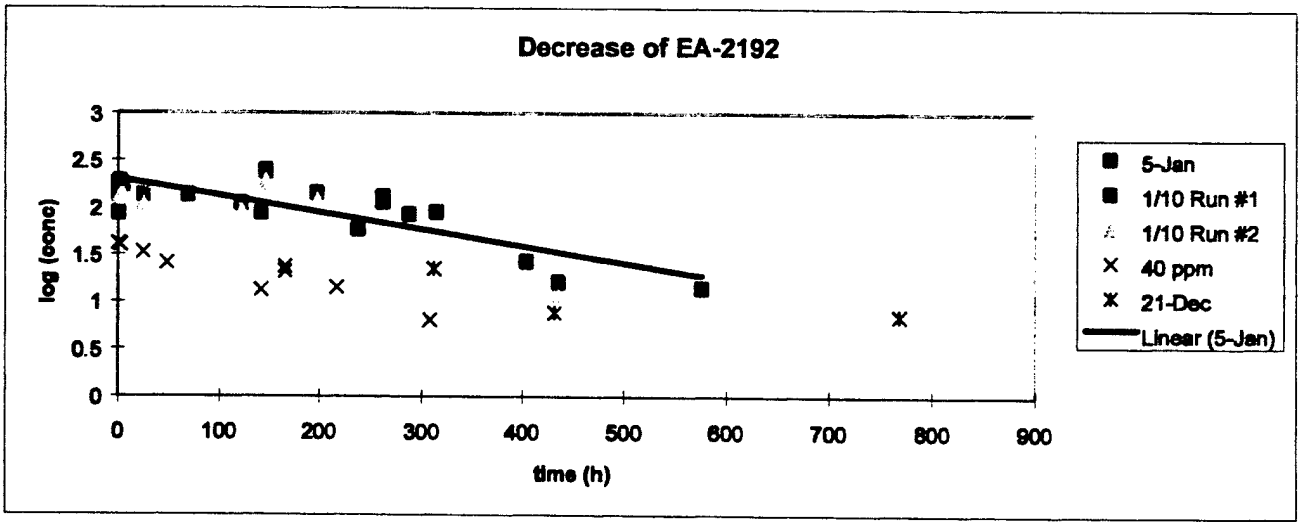

Figure 3-7. Log of EA-2192 Concentration vs. Time at Room Temperature, for Several Different Runs Which Began on the Dates Shown in the Legend. Note the time scale is in hours. The 40 ppm sample (X's) was a spike of EA-2192 in active decontamination solution with no VX added.

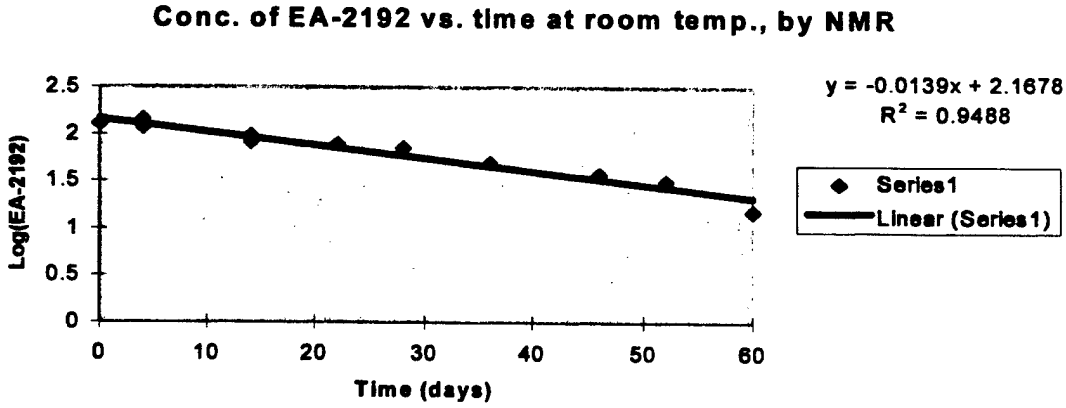

Figure 3-8. Log of the Concentration of EA-2192 vs. Time, in Days, at Room Temperature (20-25°C). Data was taken by ^{31}P NMR on a Bruker AC-300 instrument. The concentrations are not based on a calibration curve for EA-2192, but rather based on the ratio of EA-2192 signal to the total integrated phosphorus signal. The total signal was set to 1% by volume of VX, based on the original spike of VX in the decontamination solution. An additional spike of 125 ppm of EA-2192 was then also added to the active solution to provide enough EA-2192 concentration to detect.

3.6 Long-Term Reanalyses

Decontamination reaction runs were stored at ambient conditions and reanalyzed periodically by LC/MS up to 4 months after the reaction runs. There was no evidence of reformation of EA-2192 in the decontamination solutions during storage.

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LITERATURE CITED

1. H. D. Durst, ed., *Evaluation of the Chemistry which Supports VX Neutralization in the Munitions Management Device - 1 (MMD-1)*, Project Manager for Non-Stockpile Chemical Materiel, Aberdeen Proving Ground-Edgewood Area, MD, Oct. 1997.

2. W. R. Creasy, *J. Am. Soc. Mass Spectrom.* **1999,** *10,* 440-447.

3. W. Creasy, M.D. Brickhouse, K.M. Morrissey, B.R. Williams, L.S. Heykamp, et al., *Development of Analytical Methods for the Alternate Technologies Program: VX Caustic Decontamination Solutions*, EAI Report 58/96/004D1 submitted to the U. S. Army Chemical and Biological Defense Command, Aberdeen Proving Ground-Edgewood Area, MD, February 1996.

4. M. D. Crenshaw and D. B. Cummings, *Demonstration of (Trimethylsilyl)diazomethane for the derivatization of acids containing amino and hydroxyl groups*, presented at the ERDEC Scientific Conference on Chemical and Biological Defense Research, 17-20 November 1998, Aberdeen Proving Ground-Edgewood Area, MD.

5. K. M. Morrissey and T. R. Connell, *Hazard Characteristic Screening for Cholinesterase Inhibition by Neutralized VX Hydrolysates Produced in the MMD-1 Non-Stockpile Program*

6. K. M. Morrissey, B. R. Williams, J. L. Ruth, and J. R. Stuff, *Quantitative and Qualitative Gas Chromatographic Analysis of Reaction Masses Produced from Chemical Neutralization of Ethyl-S-2-Diisopropylaminoethyl Methylphosphonothioate with Monoethanolamine,* ECBC-TR-042, August 1999.

7. Y.-C. Yang, L. L. Szafraniec, W. T. Beaudry, and D. K. Rohrbaugh, *J. Am. Chem. Soc.* **1990,** *112,* 6621-6627.

8. Y.-C. Yang, L. L. Szafraniec, and W. T. Beaudry, *J. Org. Chem.* **1993,** *58,* 6964-6965.

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APPENDIX A

CHROMATOGRAPHIC DATA FROM THE CLASS II P&A OF SAMPLE PREPARATION OF EA-2192 IN MMD DECONTAMINATION SOLUTION

EA-2192 P&A in MMD Decon, 5/15/01-

Standards (5/15/01)

file	conc. (ppb)	240 signal	conc. (ppm)	Calc. (linear)	Calc. (2nd order)
1051514	400	6.05E+06	0.4	422.2489	0.39891
1051515	0	0.00E+00	0	13.03215	0
1051516	20	4.55E+05	0.02	43.80796	0.021065
1051517	40	8.32E+05	0.04	69.30791	0.03962
1051518	100	1.94E+06	0.1	144.1844	0.09987
1051519	400	6.07E+06	0.4	423.8046	0.400917

fit for first 5 points (0-400 ppb, linear)

slope	6.76391E-05
intercept	-13.0321547
corr coef.	0.998503781

Fit for all points, 2nd order polynomial
conc. =3.51e-15 * (signal)^2 + 4.47e-8* signal

CCV

file	sample.	Signal (240)	calc.conc. (linear)	calc (poly)
1051501	400	7.19E+06	499.3575	0.502846
1051502	400	6.22E+06	433.7475	0.41383
1051503	400	6.80E+06	472.9782	0.466262

(5/15 Run)

Filename	Sample ID	signal	found con. (ppb, linear fit)	found con. (ppm. poly. fit)	Conc. (ppb)	LC r.t
1051504	NB114P88A	2.91E+05	32.71514	0.013305	13.30493	
1051505	NB114P88B	2.17E+05	27.70984	0.009865	9.865182	
1051506	NB114P88C	7.46E+05	63.49094	0.0353	35.29957	7.45
1051507	NB114P88D	6.59E+05	57.60634	0.030982	30.98163	7.31
1051508	NB114P88E	6.60E+05	57.67398	0.031031	31.03096	7.21
1051509	NB114P88F	5.89E+05	52.8716	0.027546	27.54599	7.18
1051510	NB114P88G	6.67E+05	58.14745	0.031376	31.37646	7.24
1051511	NB114P88H	7.05E+05	60.71774	0.033258	33.25806	7.22
1051512	NB114P88J	7.21E+05	61.79996	0.034053	34.05334	7.23
1051513	NB114P88K	6.25E+05	55.30661	0.029309	29.30859	7.21
		Ave. Spike		0.031607	31.60682	7.25625
		Std. Dev.		0.002533	2.532536	0.086839
		MDL		0.007595	7.595076	
		% recovery			79.01706	

Standards (5/16/01)

file	conc. (ppb)	240 signal	conc. (ppm)	Calc. (linear)	Calc. (2nd order)
1051614	400	6.07E+06	0.4	419.7776	0.395149
1051615	0	0.00E+00	0	13.24121	0
1051616	20	4.86E+05	0.02	45.79091	0.022384
1051617	40	8.24E+05	0.04	68.42836	0.038901
1051618	100	1.96E+06	0.1	144.5116	0.100124
1051619	400	6.18E+06	0.4	427.1448	0.404628

fit for first 5 points (0-400 ppb, linear)

slope	6.69747E-05
intercept	-13.2412085
corr coef.	0.99845721
	9

Fit for all points, 2nd order polynomial
conc. =3.41e-15 * (signal)^2 + 4.44e-8* signal

CCV

file	sample.	Signal (240)	calc.conc. (linear)	calc (poly)
1051601	400	6.00E+06	415.0894	0.38916
1051602	400	6.21E+06	429.1541	0.407228
1051603	400	6.33E+06	437.191	0.417687

(5/16 Run)

Filename	Sample ID	signal	found con. (ppb, linear fit)	found con. (ppm. poly. fit)	Conc. (ppb)	LC r.t
1051604	NB114P89A	8.60E+04	19.00103	0.003844	3.84362	
1051605	NB114P89B	7.60E+04	18.33129	0.003394	3.394096	
1051606	NB114P89C	5.12E+05	47.53225	0.023627	23.62671	7.21
1051607	NB114P89D	4.73E+05	44.92024	0.021764	21.76412	7.2
1051608	NB114P89E	5.35E+05	49.07267	0.02473	24.73003	7.21
1051609	NB114P89F	5.61E+05	50.81401	0.025982	25.9816	7.2
1051610	NB114P89G	5.69E+05	51.34981	0.026368	26.36763	7.21
1051611	NB114P89H	5.10E+05	47.3983	0.023531	23.53094	7.18
1051612	NB114P89J	5.31E+05	48.80477	0.024538	24.53789	7.18
1051613	NB114P89K	5.54E+05	50.34519	0.025644	25.64418	7.2
		Ave. Spike		0.024523	24.52289	7.19875
		Std. Dev.		0.001523	1.52265	0.012464
		MDL		0.004566	4.566427	
		% recovery			61.30722	

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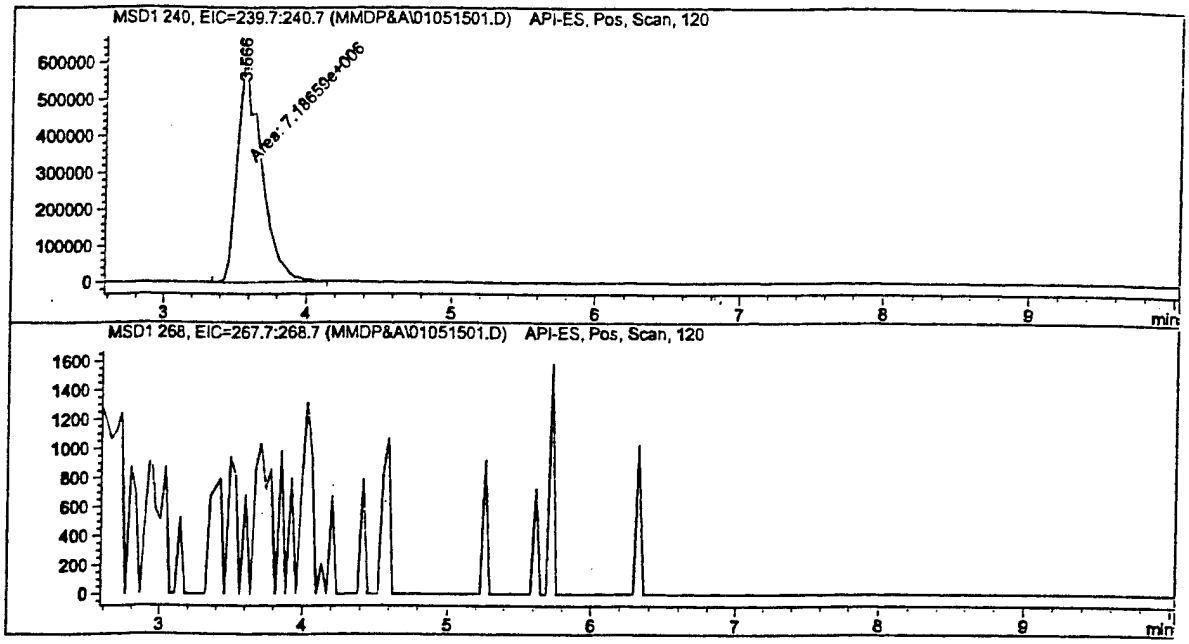

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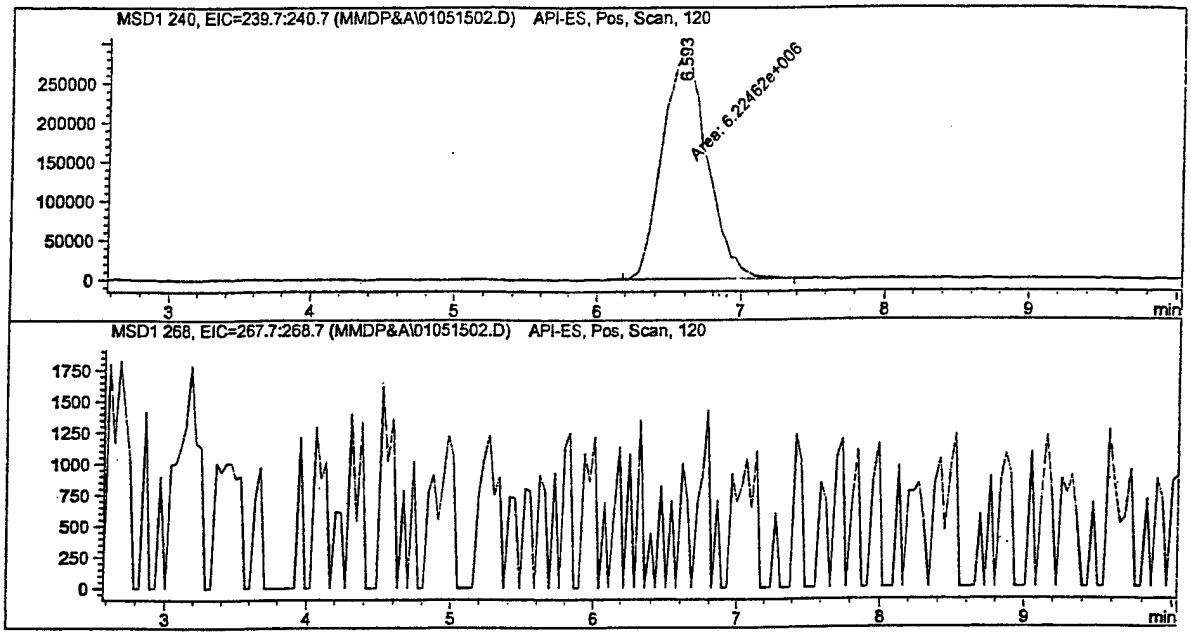

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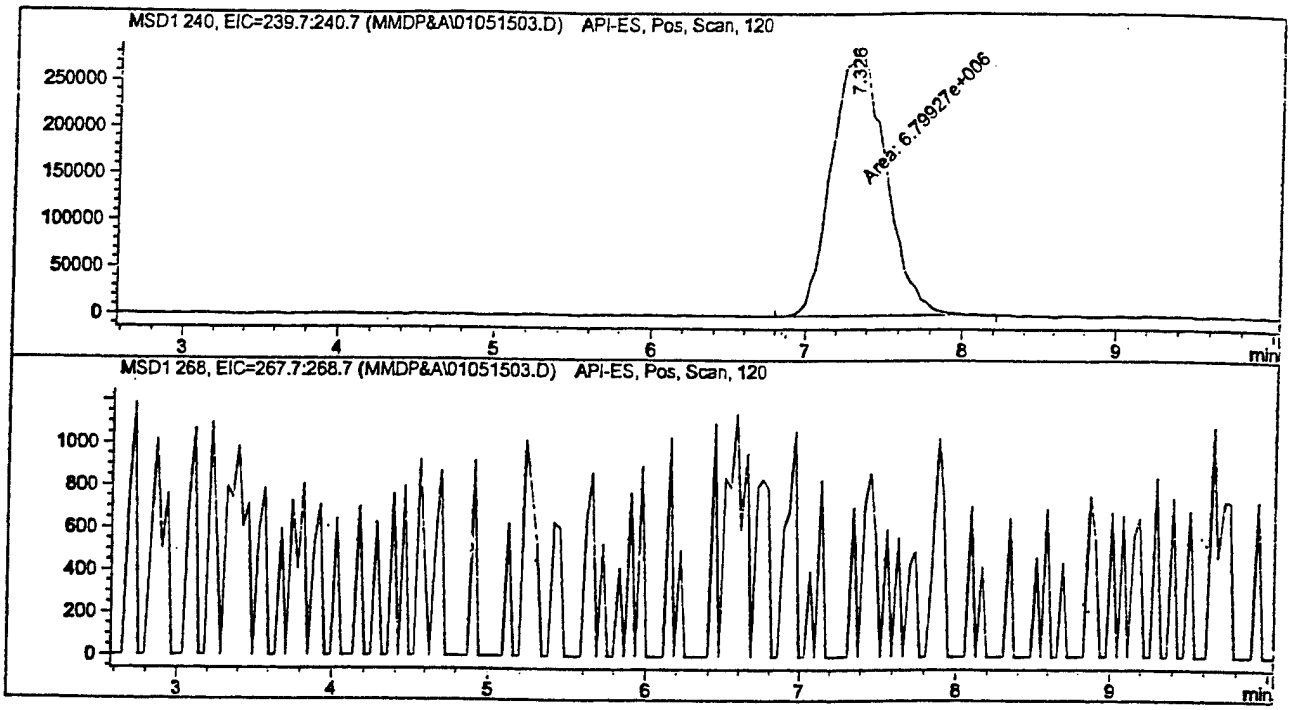

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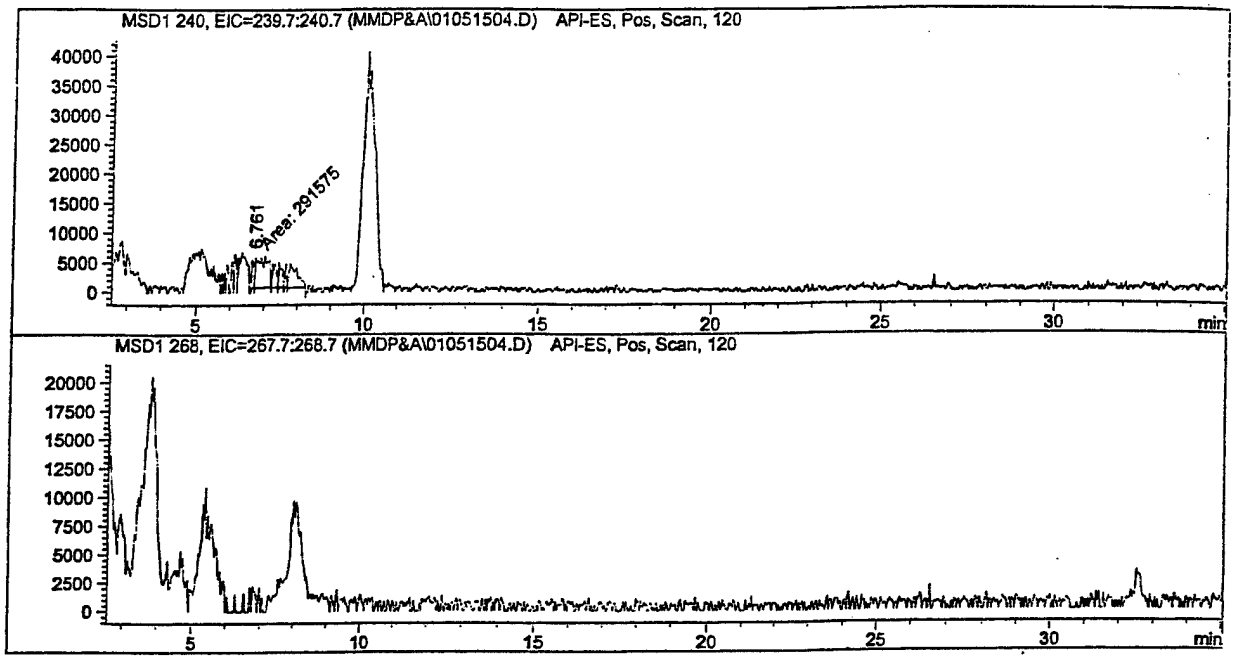

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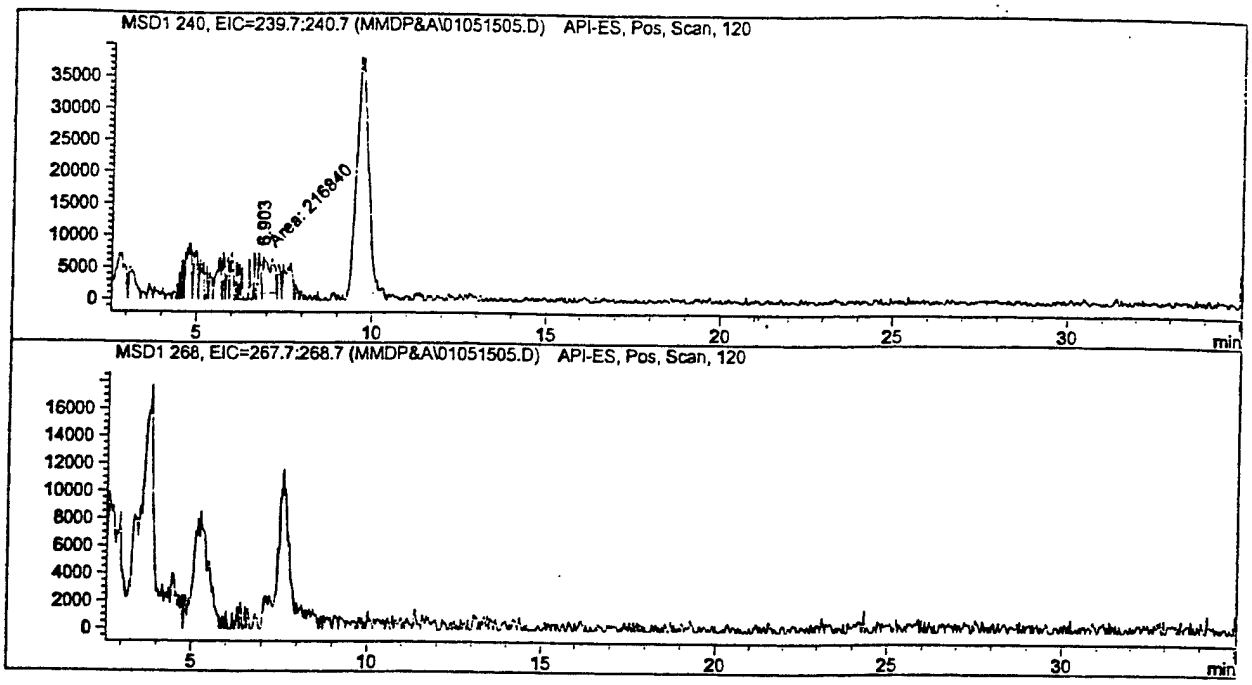

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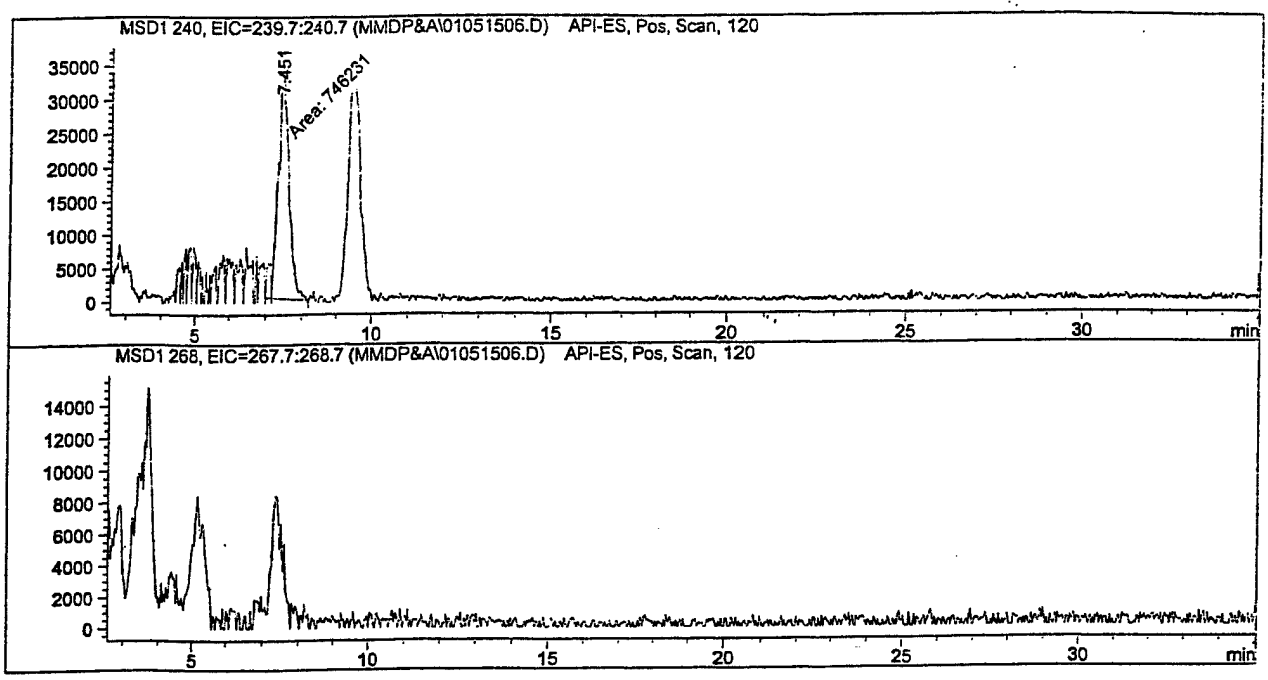

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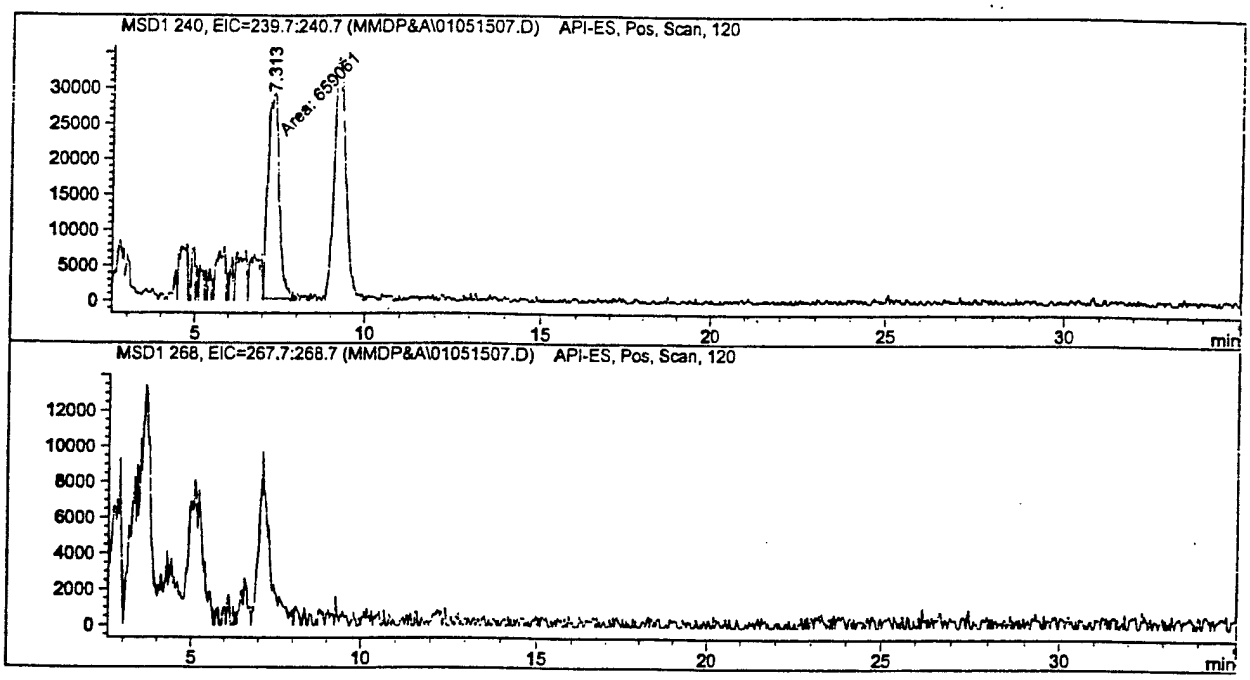

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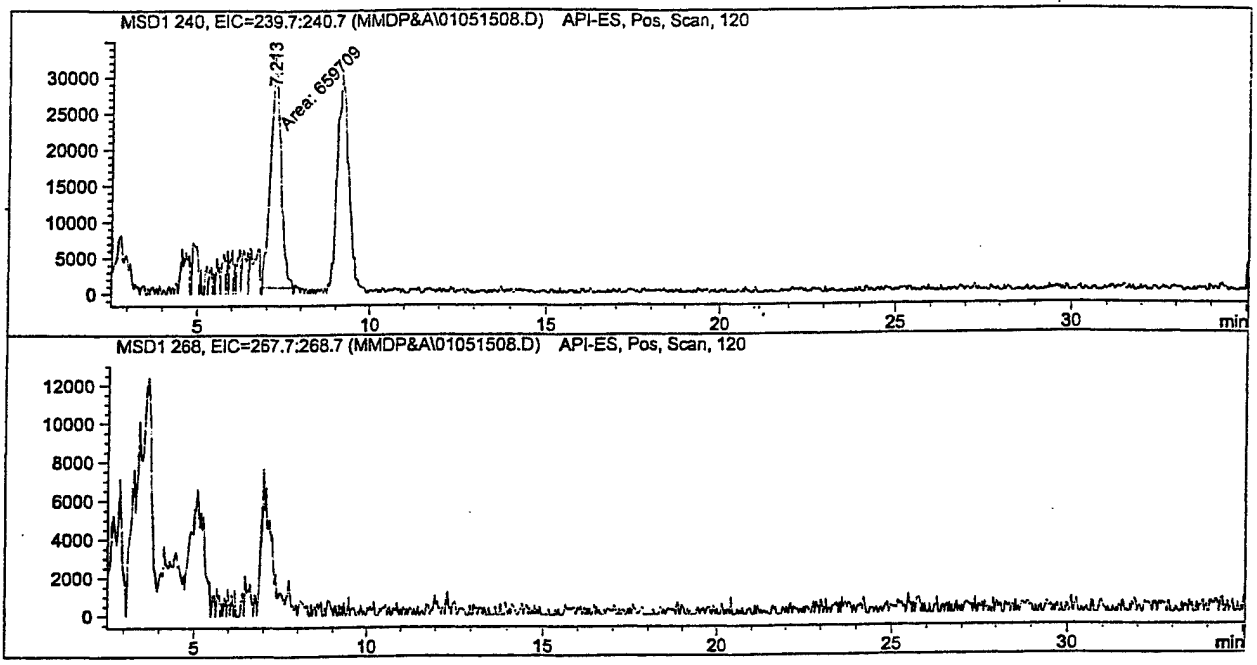

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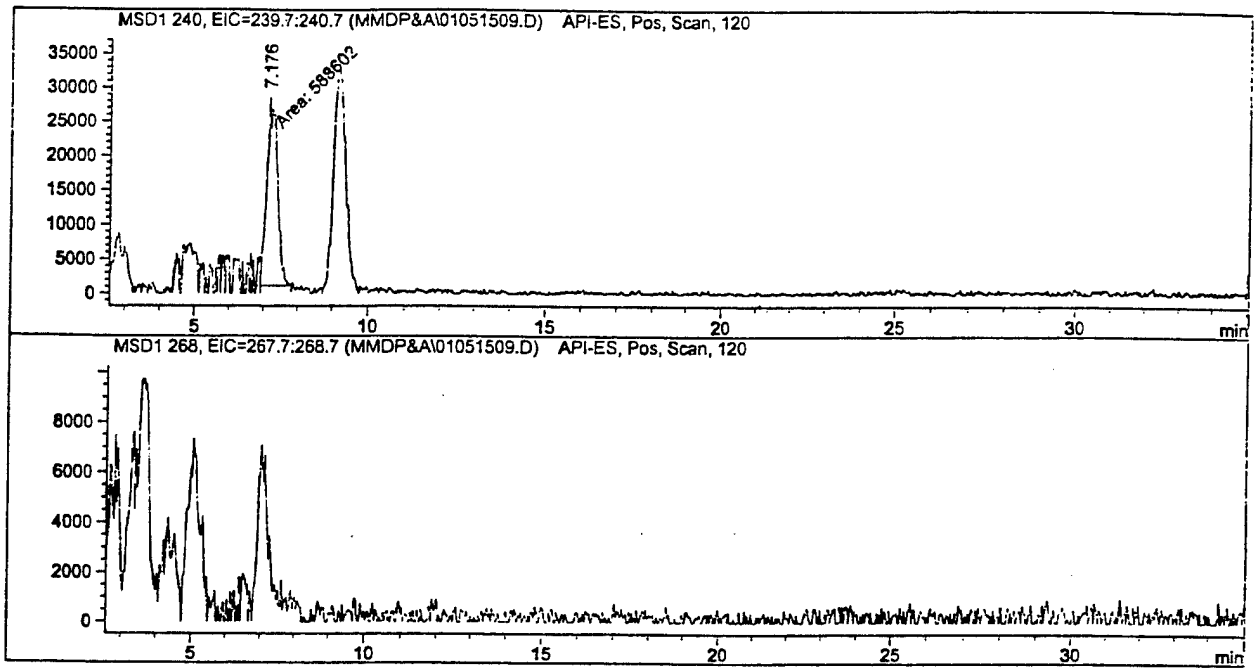
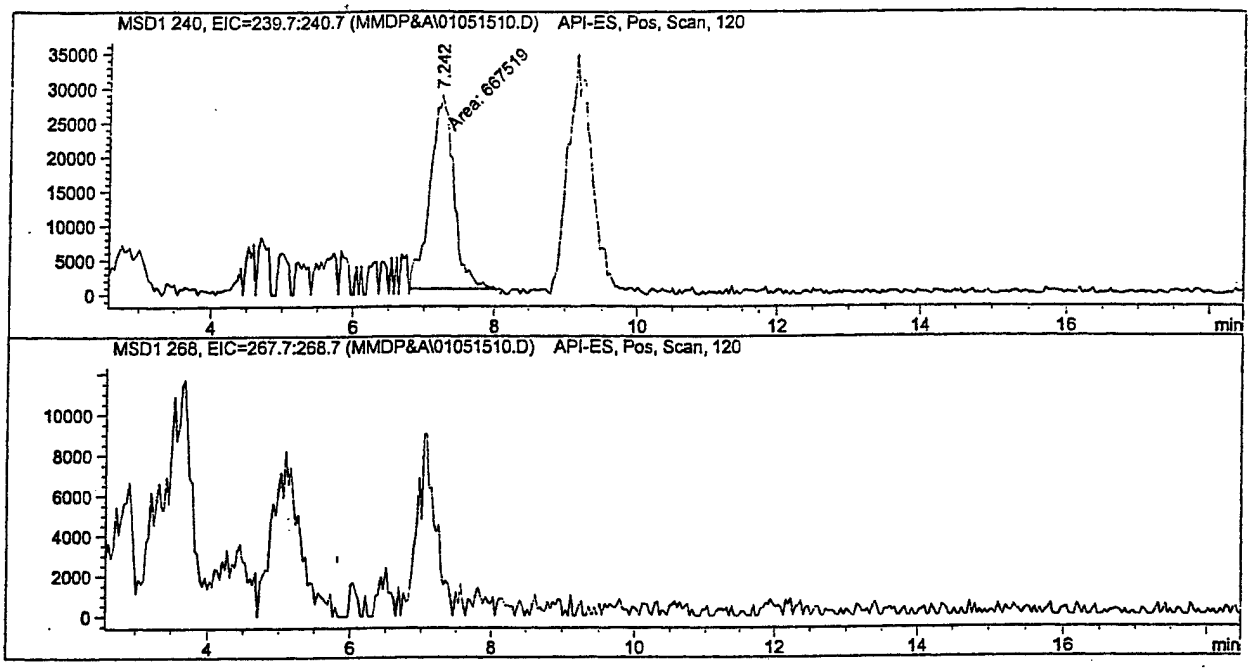
Data File C:\HPCHEM\1\DATA\MMDP&A\01051509.D Sample Name: NB114P88F



*** End of Report ***

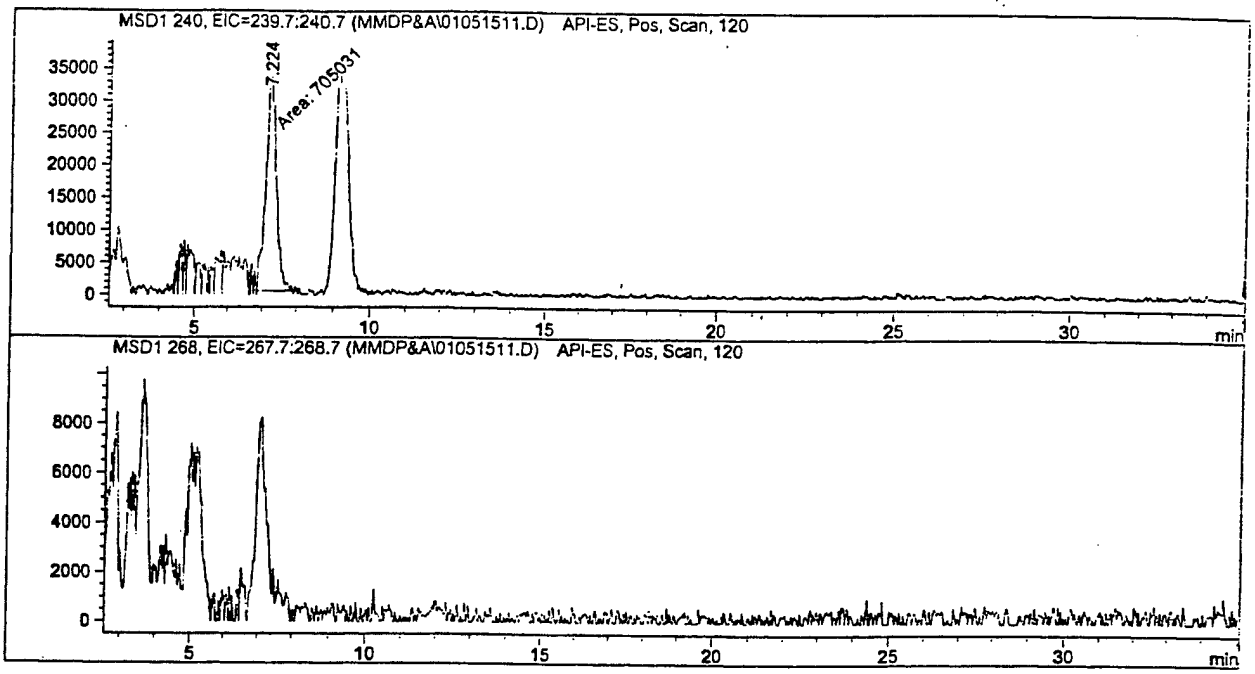
Data File C:\HPCHEM\1\DATA\MMDP&A\01051510.D Sample Name: NB114P88G



*** End of Report ***

Instrument 1 5/16/2001 1:13:21 PM wrc

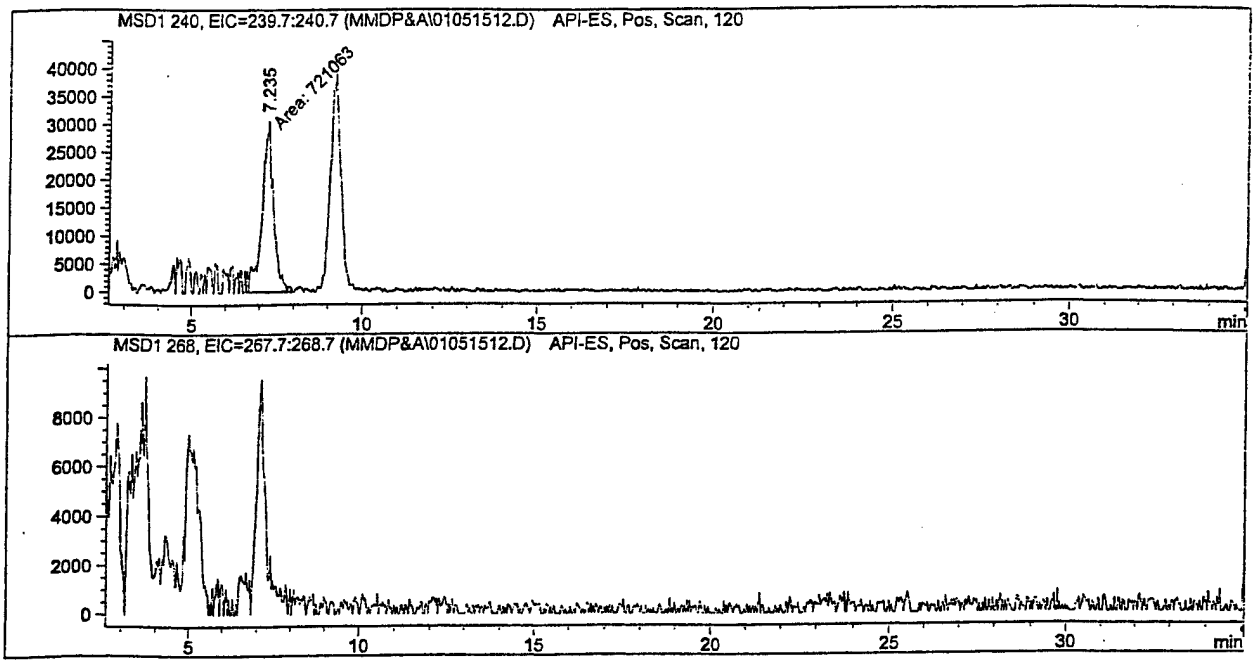
Data File C:\HPCHEM\1\DATA\MMDP&A\01051511.D Sample Name: NB114P88H



*** End of Report ***

Instrument 1 5/16/2001 1:04:31 PM wrc

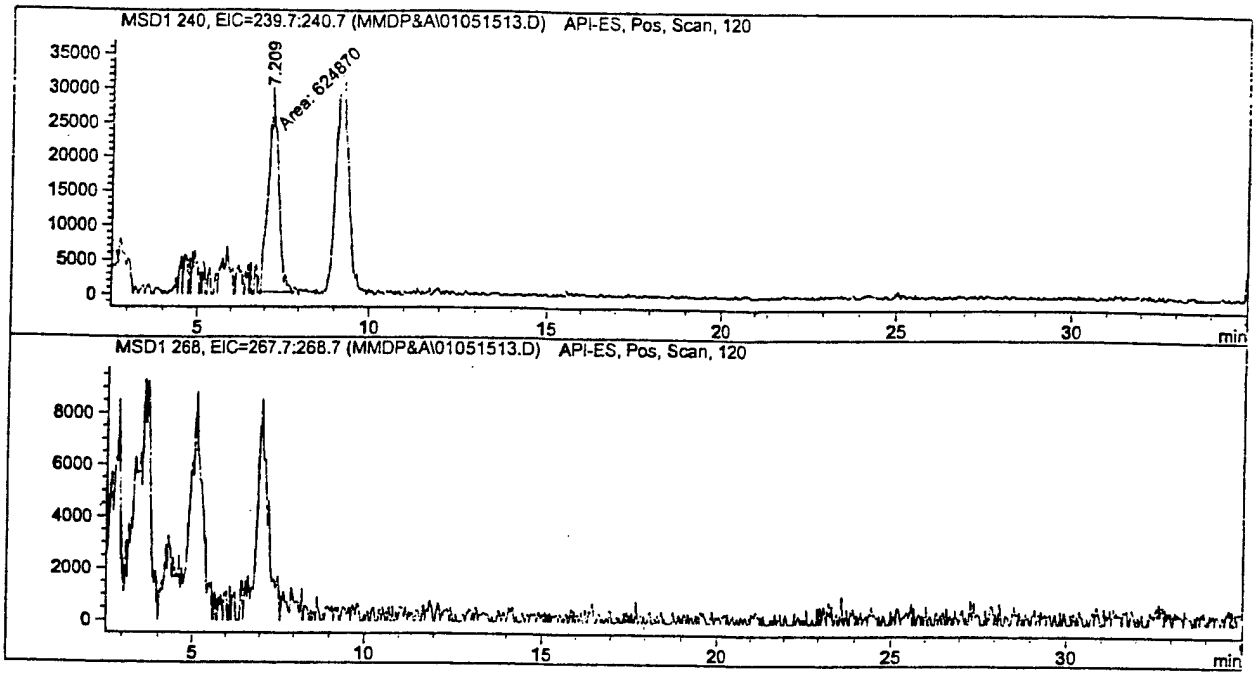
Data File C:\HPCHEM\1\DATA\MMDP&A\01051512.D Sample Name: NB114P88J



*** End of Report ***

Instrument 1 5/16/2001 1:05:29 PM wrc

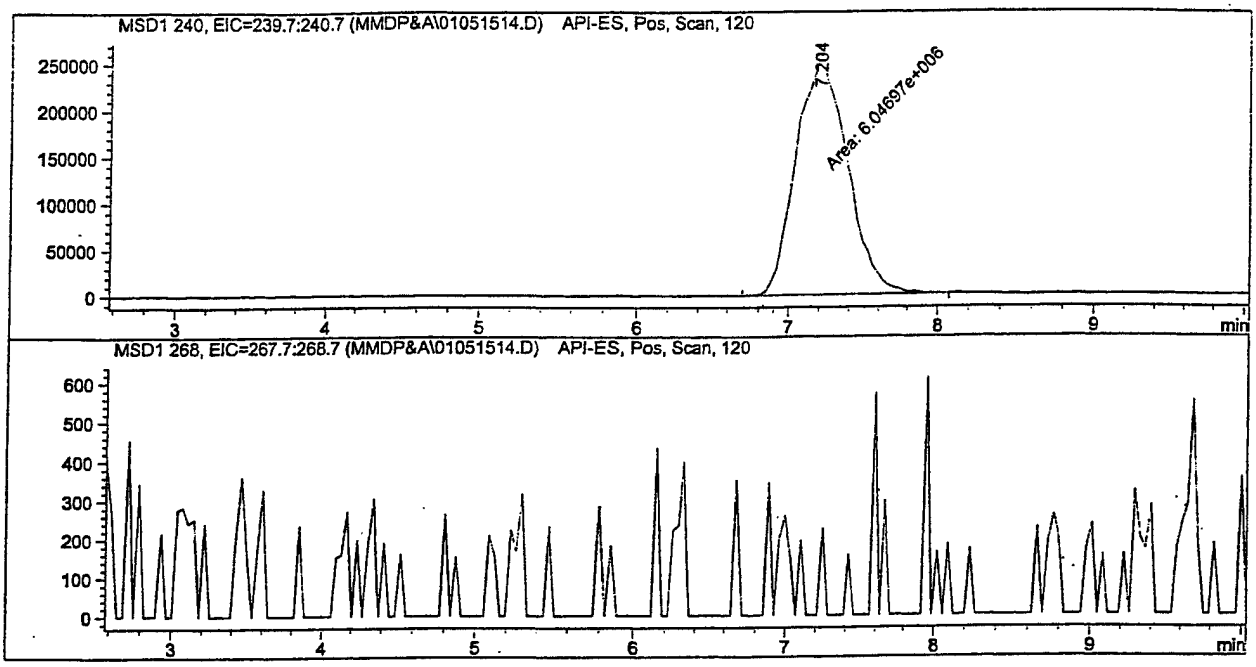
Data File C:\HPCHEM\1\DATA\MMDP&A\01051513.D Sample Name: NB114P88K



*** End of Report ***

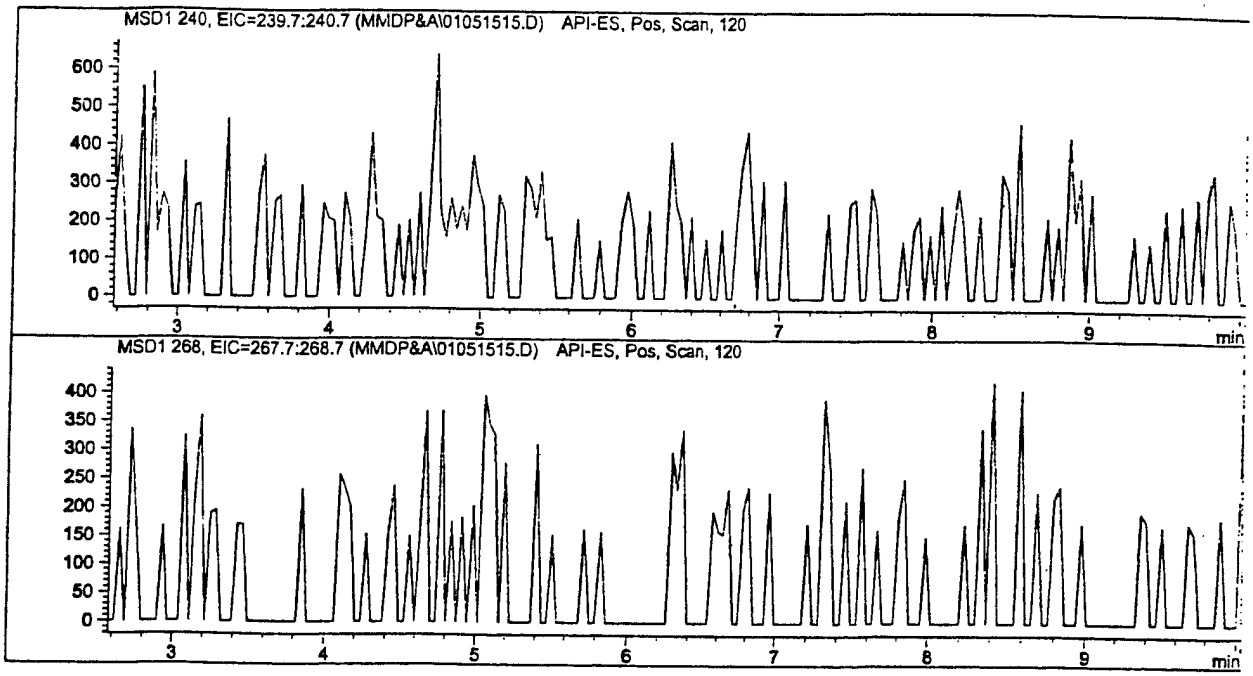
Instrument 1 5/16/2001 1:06:19 PM wrc

Data File C:\HPCHEM\1\DATA\MMDP&A\01051514.D Sample Name: 400 ppb



*** End of Report ***

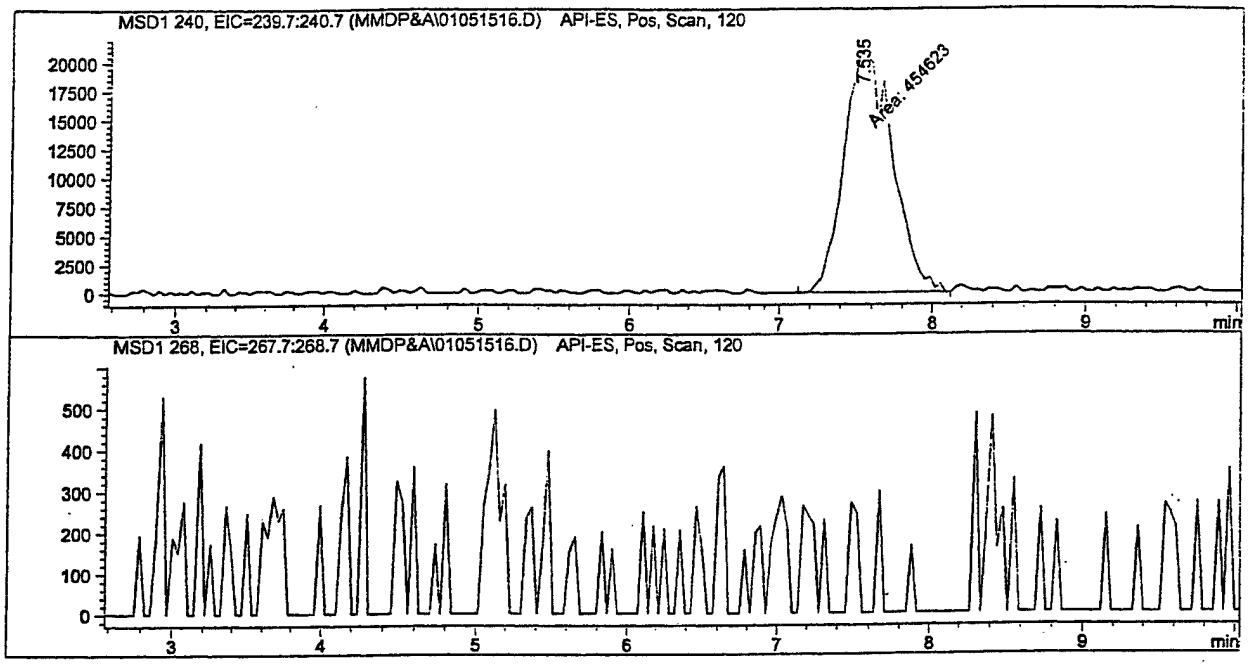
Data File C:\HPCHEM\1\DATA\MMDP&A\01051515.D Sample Name: blank



*** End of Report ***

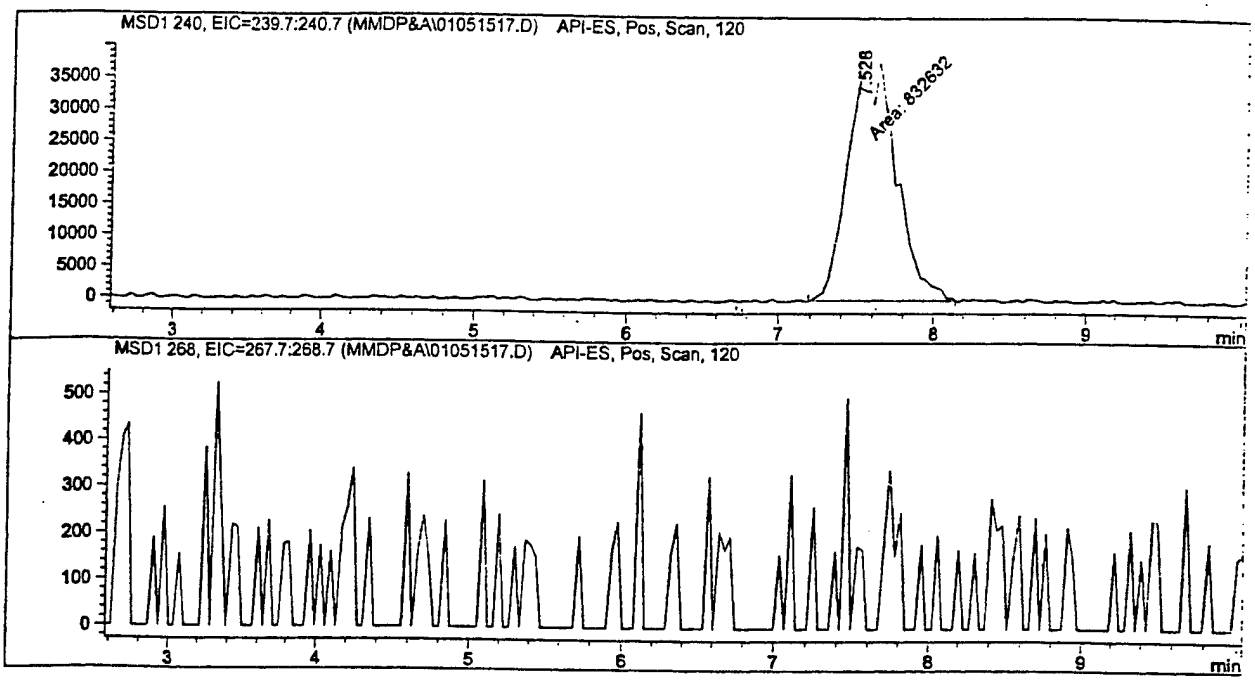
Instrument 1 5/16/2001 1:18:23 PM wrc

Data File C:\HPCHEM\1\DATA\MMDP&A\01051516.D Sample Name: 20 ppb



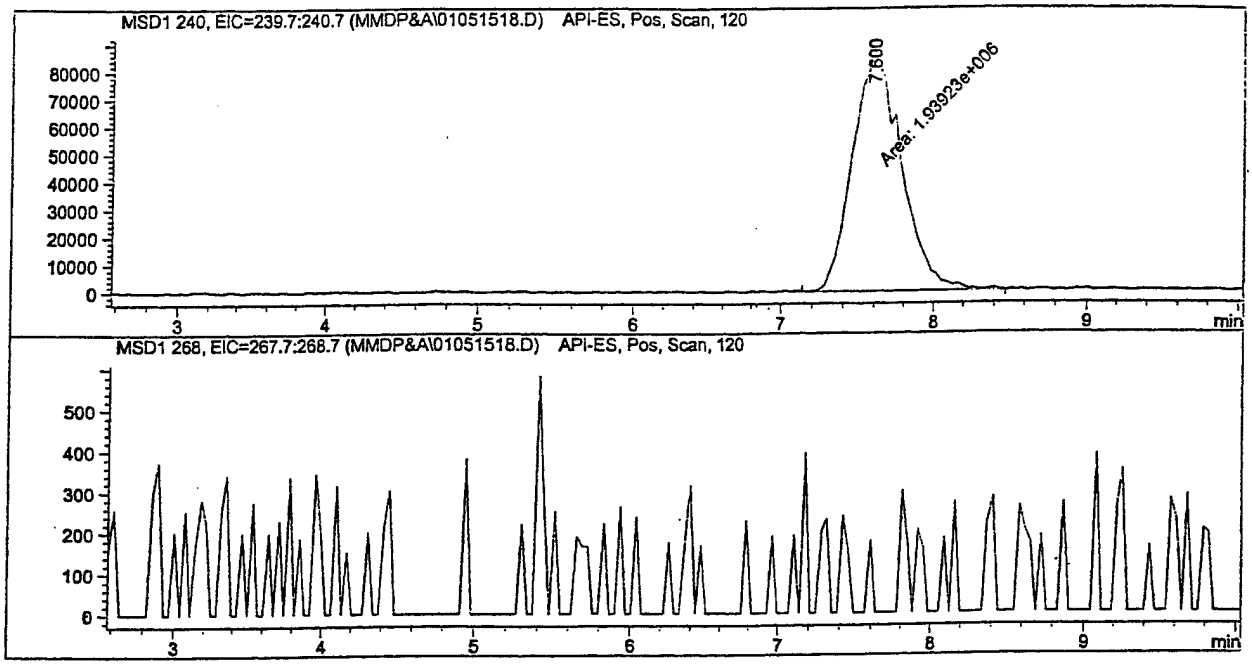
*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMDP&A\01051517.D Sample Name: 40 ppb



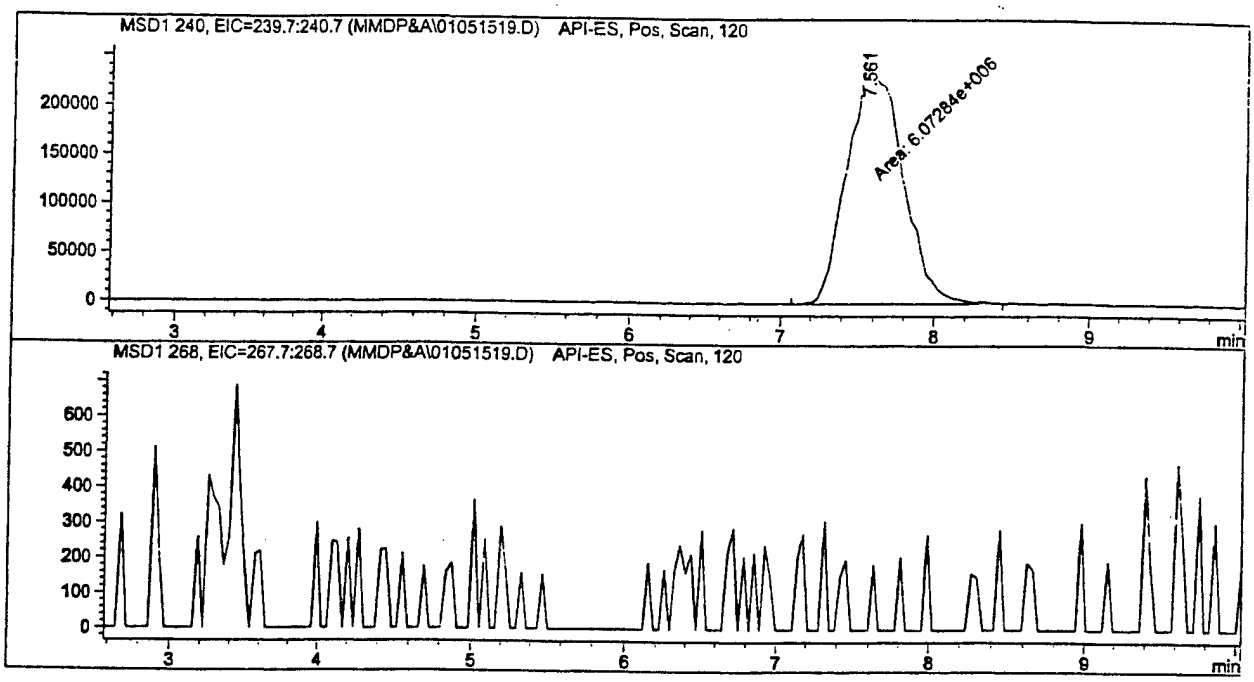
*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMDP&A\01051518.D Sample Name: 100 ppb



*** End of Report ***

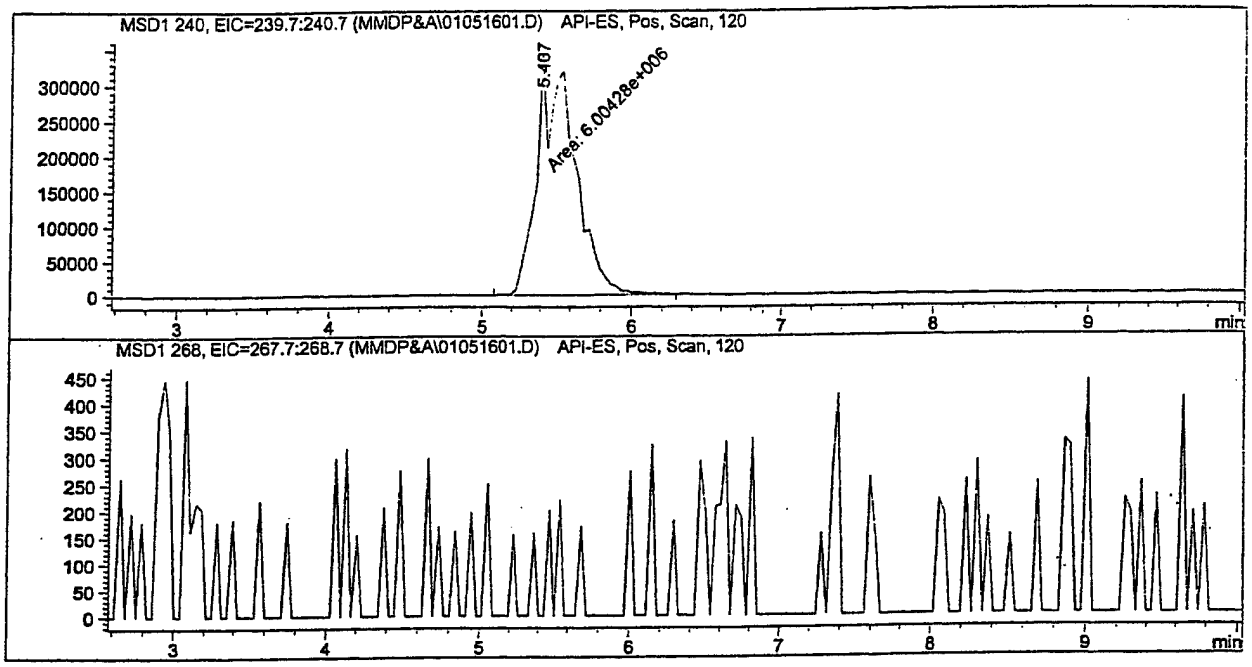
Data File C:\HPCHEM\1\DATA\MMDP&A\01051519.D Sample Name: 400 ppb



*** End of Report ***

Instrument 1 5/16/2001 1:21:50 PM wrc

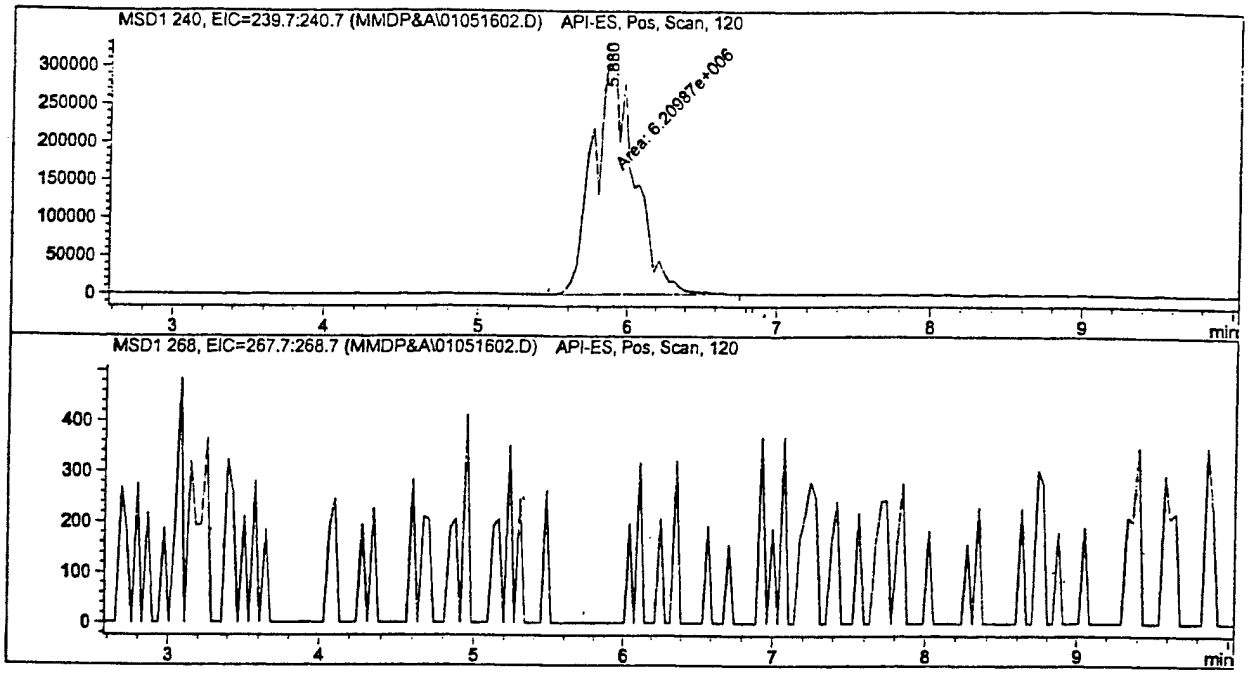
Data File C:\HPCHEM\1\DATA\MMDP&A\01051601.D Sample Name: 400 ppb 2192



*** End of Report ***

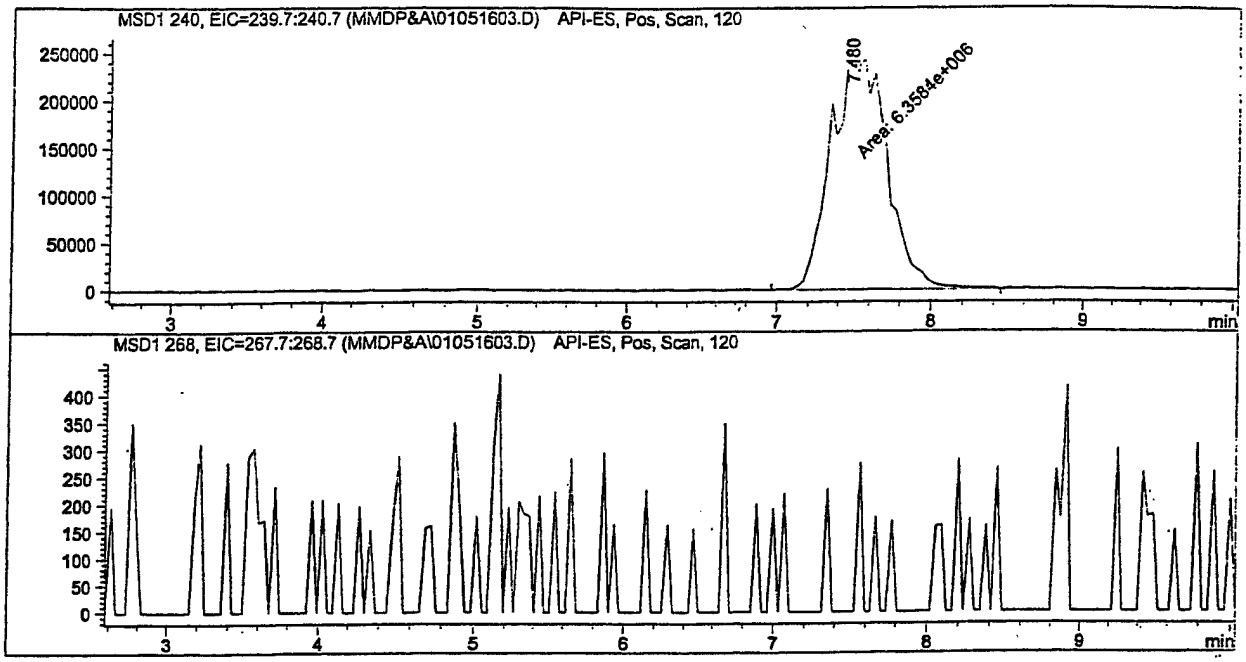
Instrument 1 5/17/2001 9:19:51 AM wrc

Data File C:\HPCHEM\1\DATA\MMDP&A\01051602.D Sample Name: 400 ppb 2192



*** End of Report ***

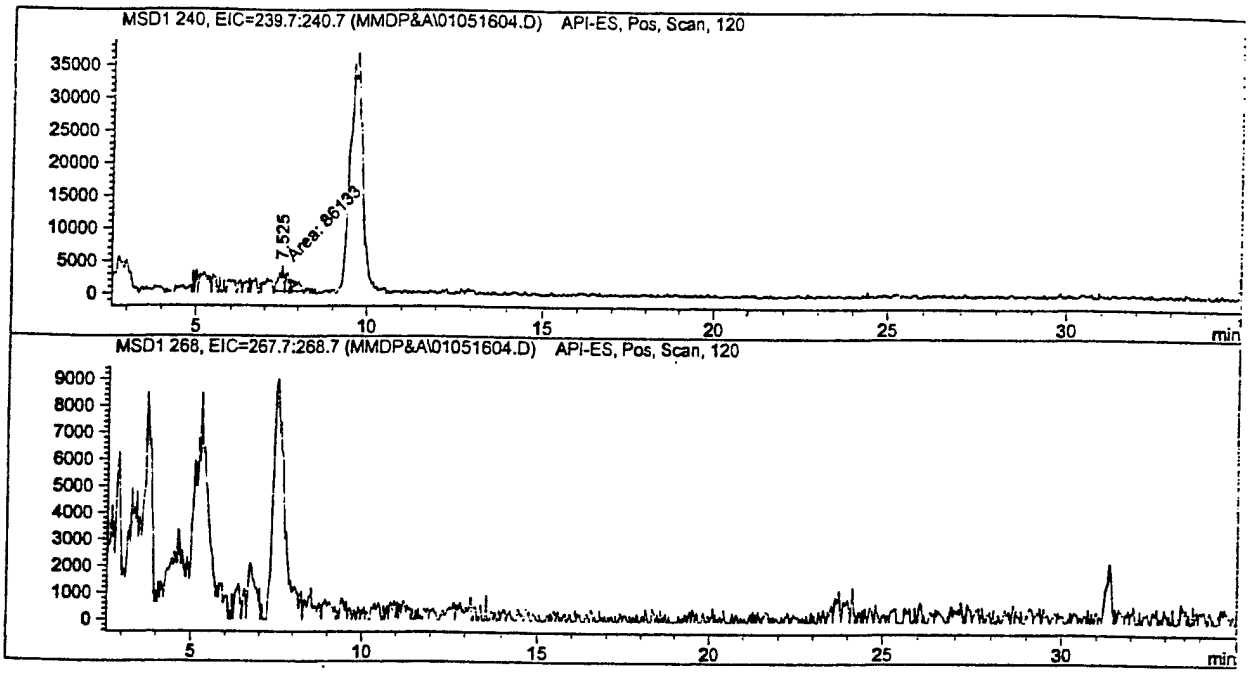
Data File C:\HPCHEM\1\DATA\MMDP&A\01051603.D .Sample Name: 400 ppb 2192



*** End of Report ***

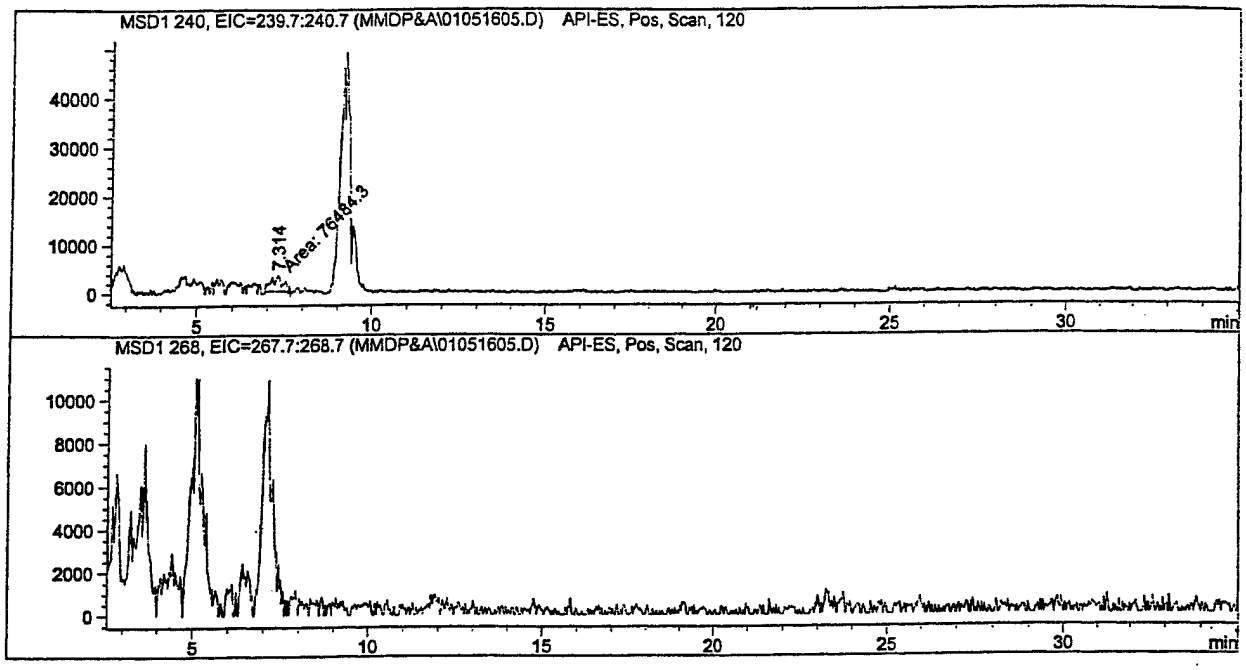
Instrument 1 5/17/2001 9:42:58 AM wrc

Data File C:\HPCHEM\1\DATA\MMDP&A\01051604.D Sample Name: NB114P89A



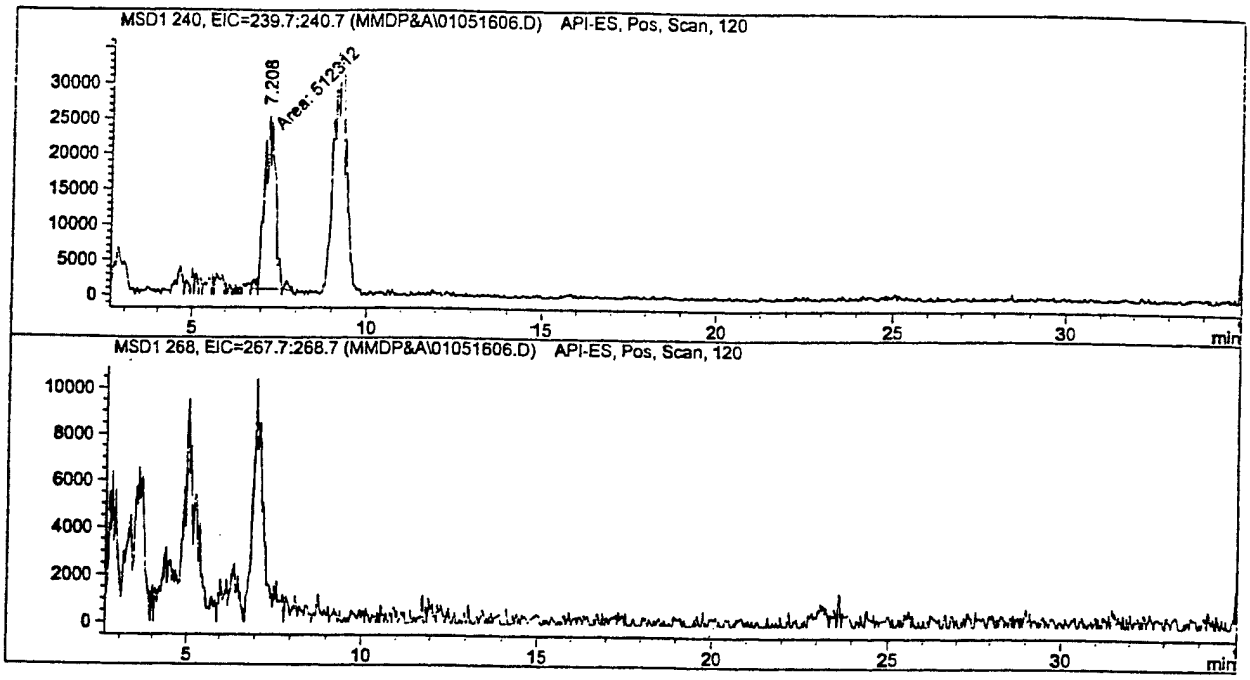
*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMDP&A\01051605.D Sample Name: NB114P89B

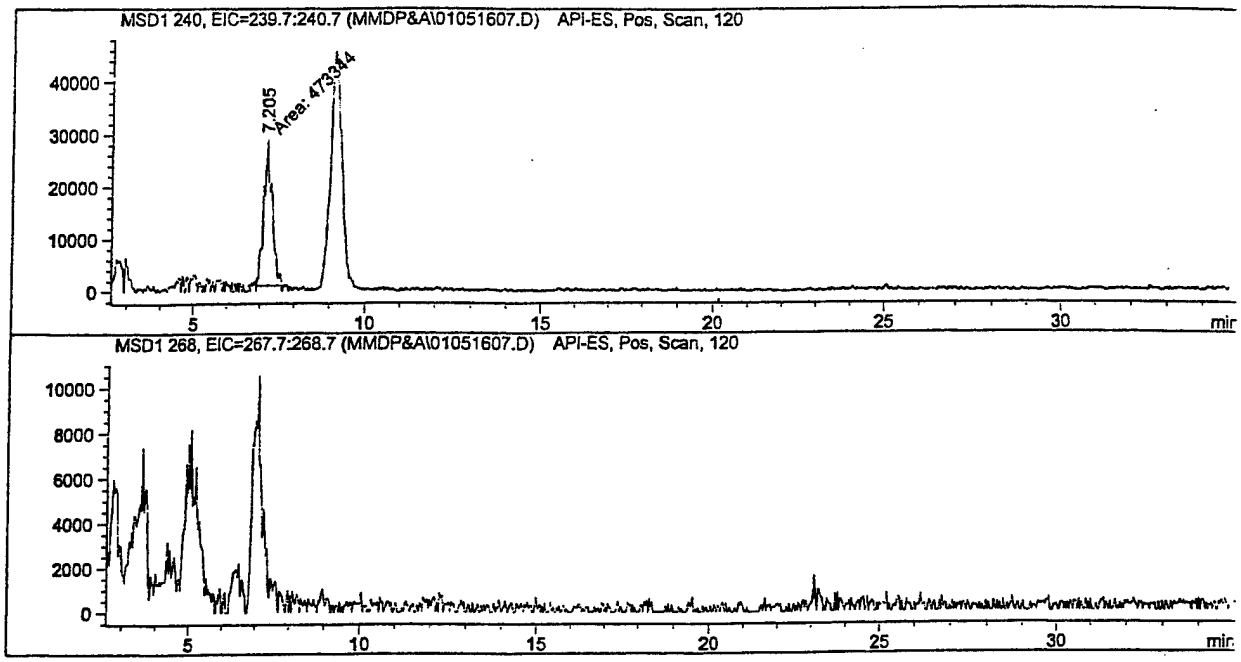


*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMDP&A\01051606.D Sample Name: NB114P89C

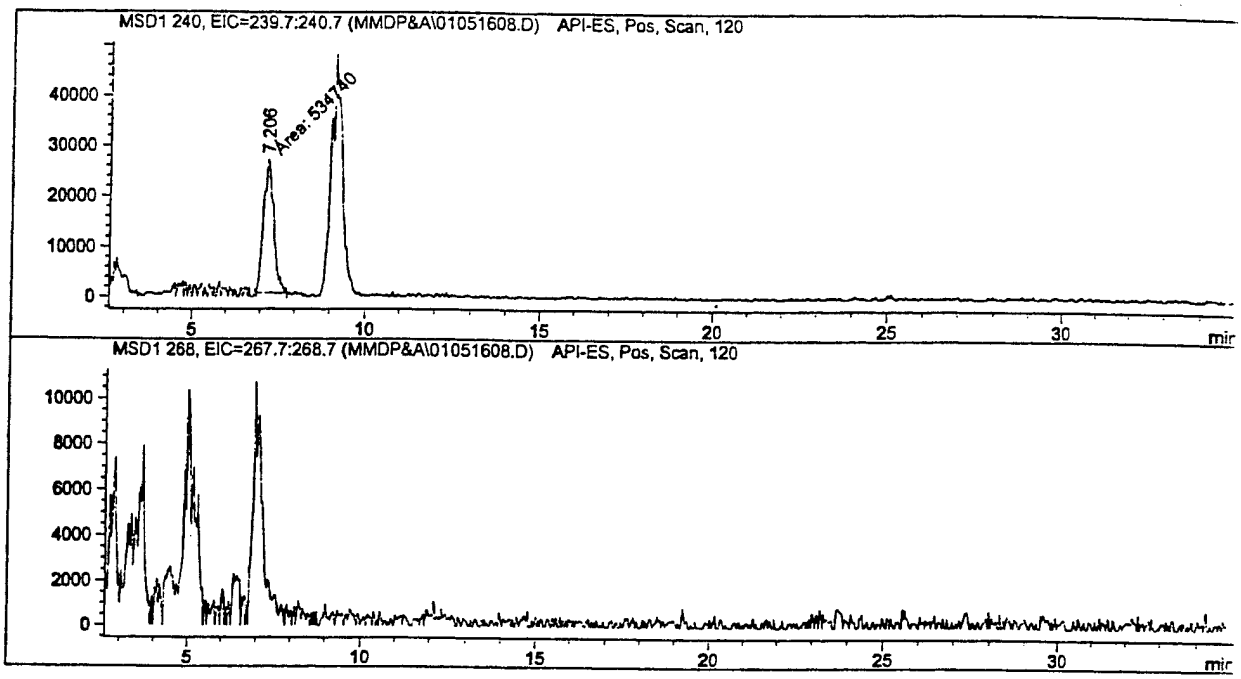


*** End of Report ***



*** End of Report ***

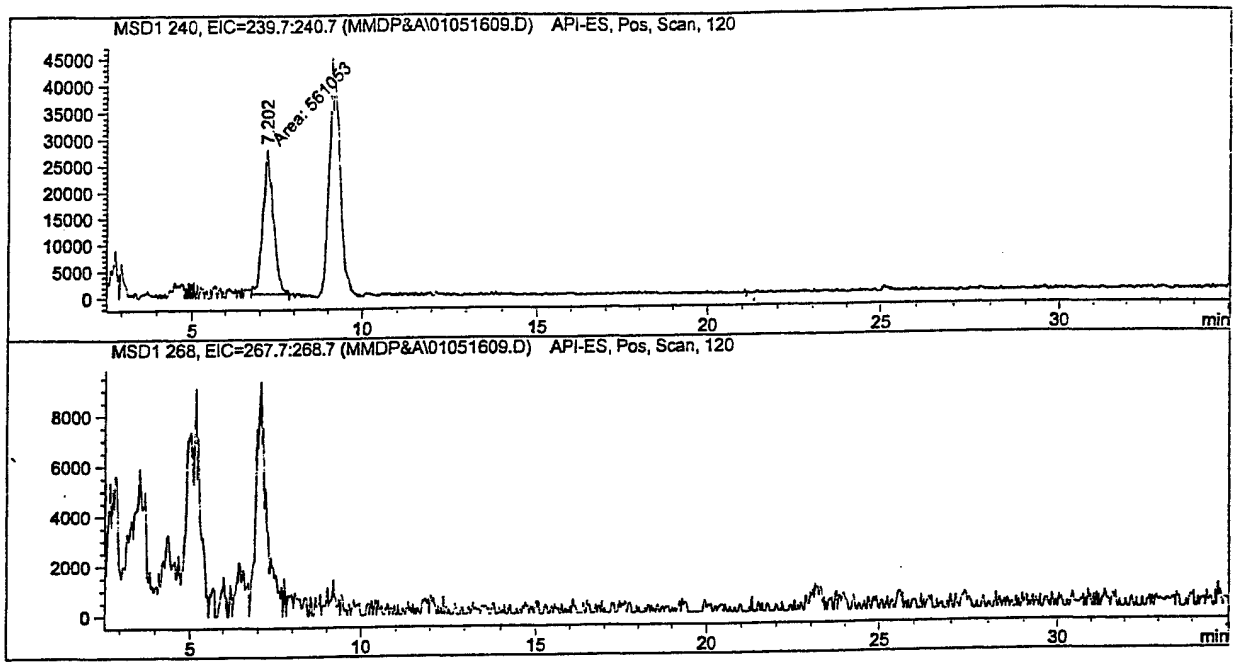
Data File C:\HPCHEM\1\DATA\MMDP&A\01051608.D Sample Name: NB114P89:



*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMDP&A\01051609.D

Sample Name: NB114P89F

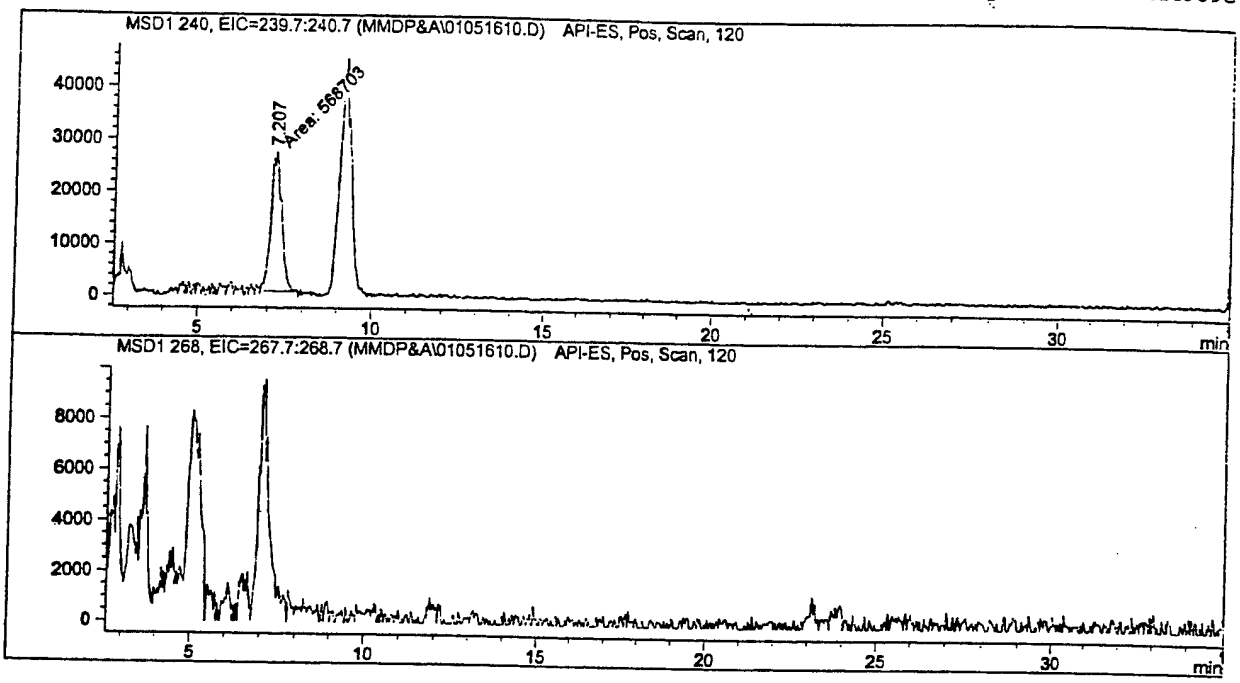


*** End of Report ***

Instrument 1 5/17/2001 9:27:56 AM wrc

Data File C:\HPCHEM\1\DATA\MMDP&A\01051610.D

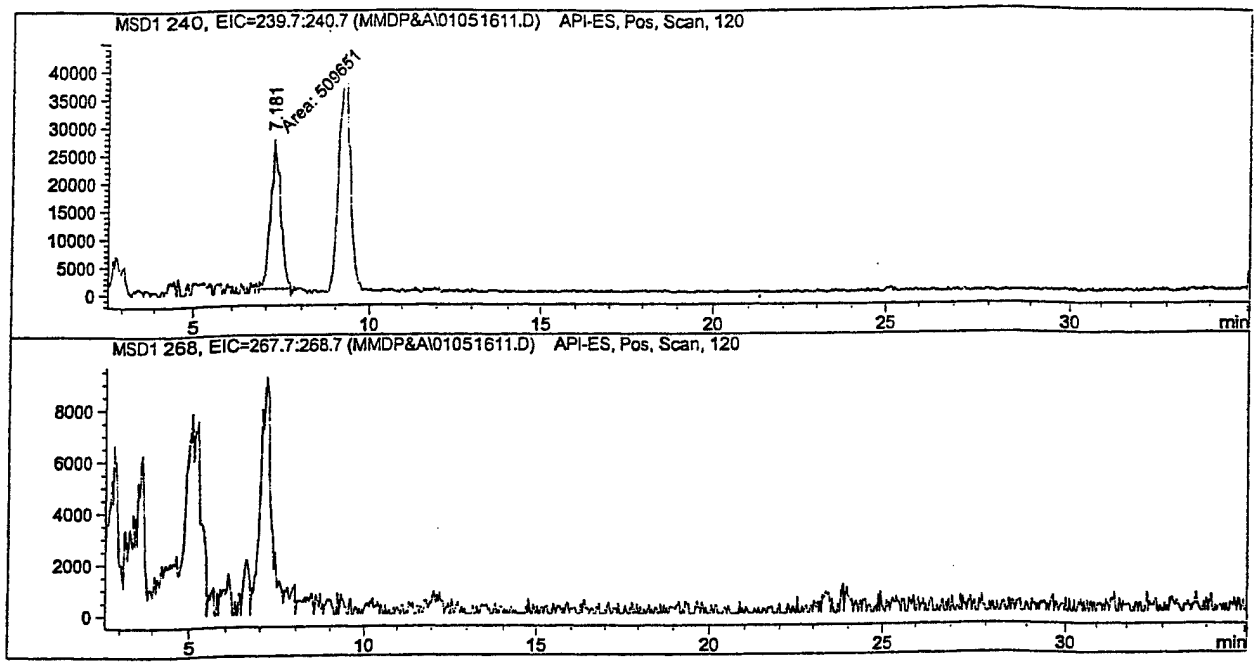
Sample Name: NB114P89G



*** End of Report ***

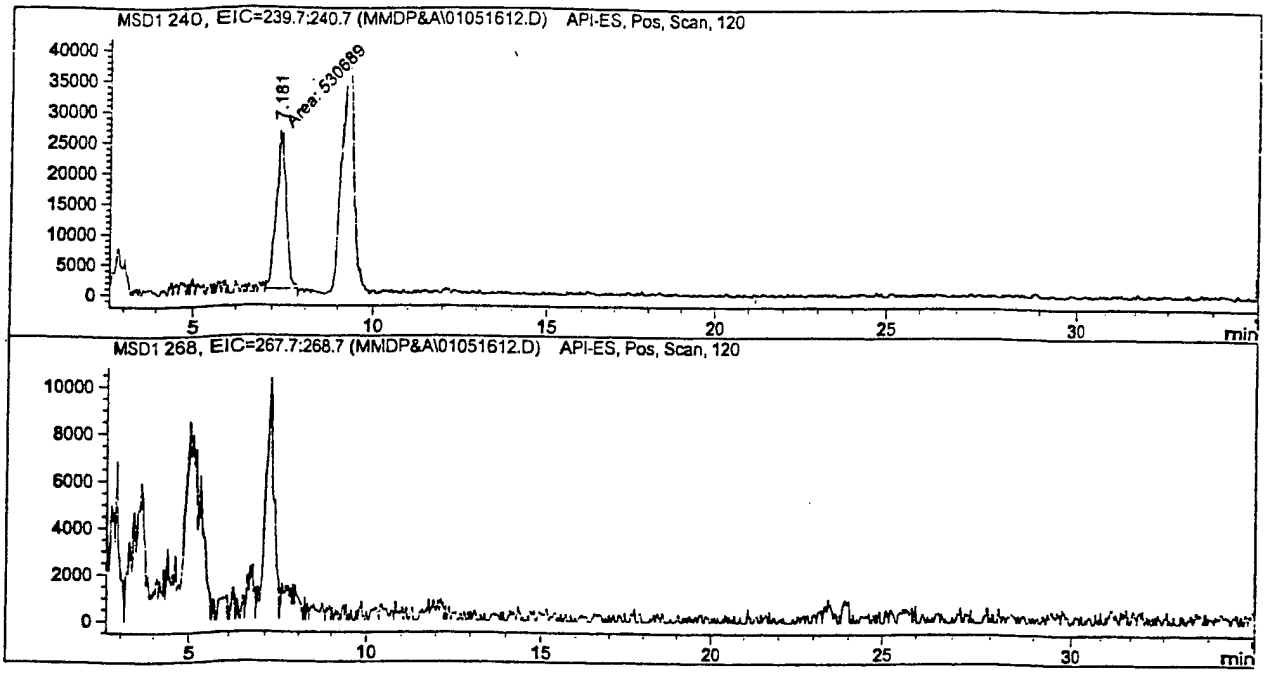
Instrument 1 5/17/2001 9:28:55 AM wrc

Data File C:\HPCHEM\1\DATA\MMDP&A\01051611.D Sample Name: NB114P89H



*** End of Report ***

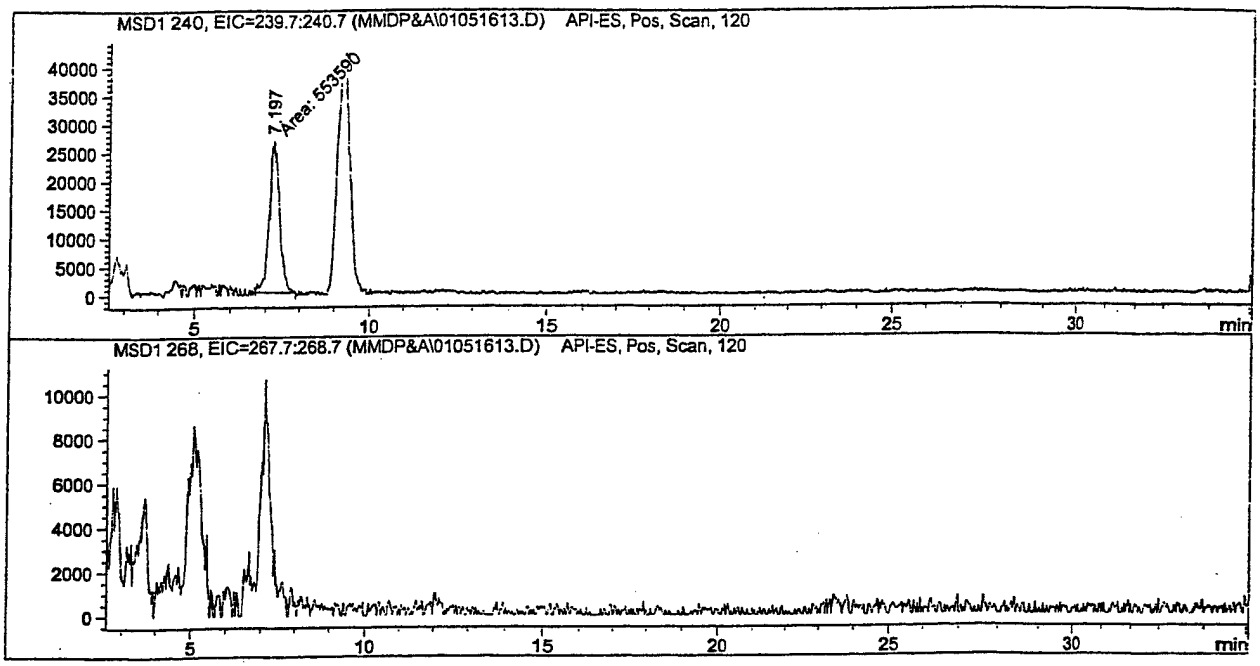
Data File C:\HPCHEM\1\DATA\MMDP&A\01051612.D Sample Name: NB114P89J



*** End of Report ***

Instrument 1 5/17/2001 9:30:41 AM wrc

Data File C:\HPCHEM\1\DATA\MMDP&A\01051613.D Sample Name: NB114P89K

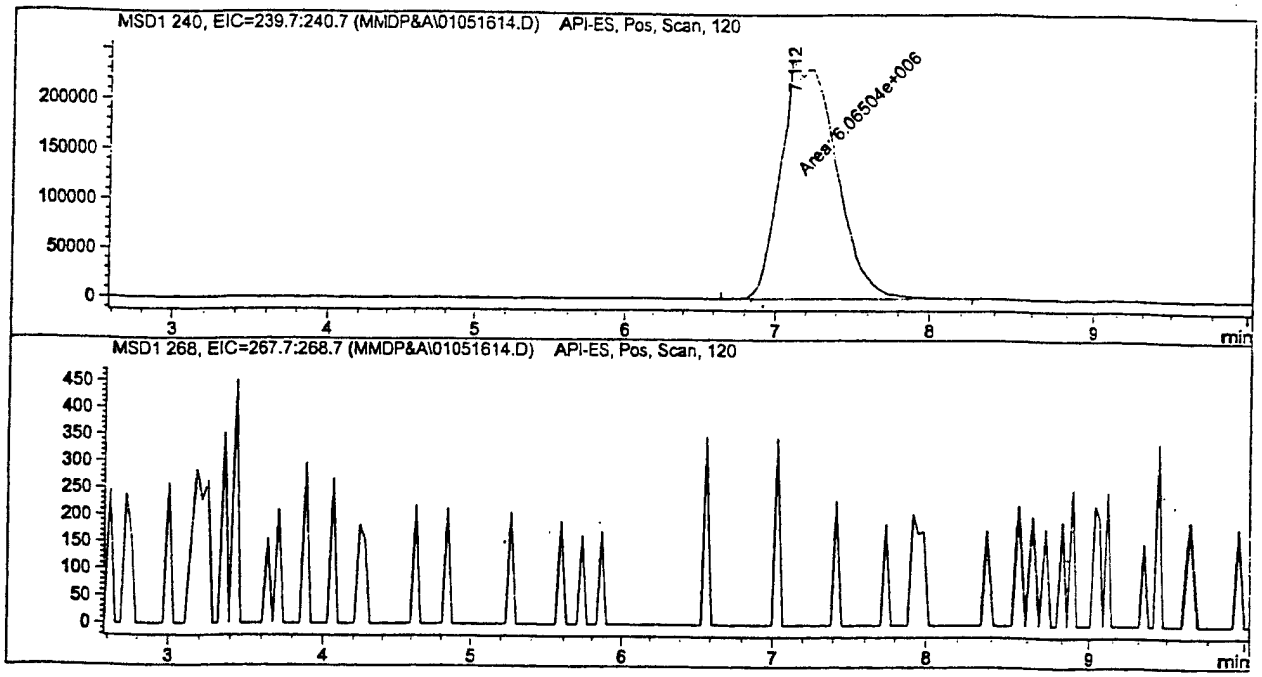


*** End of Report ***

Instrument 1 5/17/2001 9:31:38 AM wrc

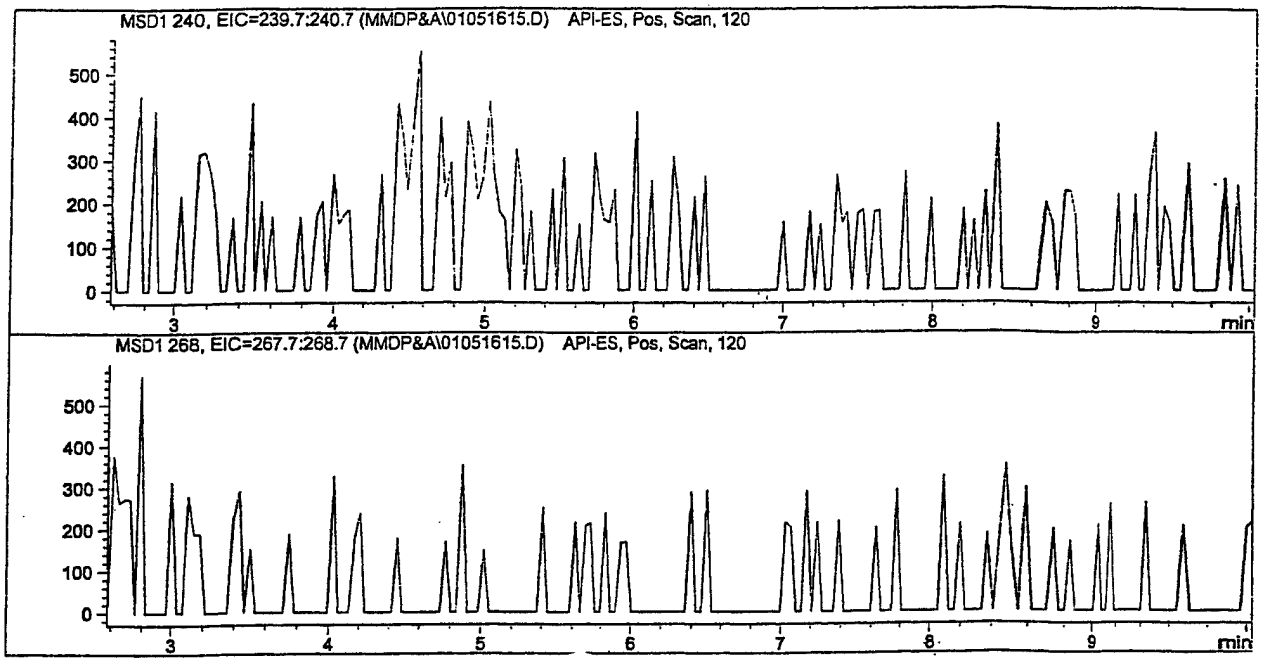
Data File C:\HPCHEM\1\DATA\MMDP&A\01051614.D

Sample Name: 400 ppb



*** End of Report ***

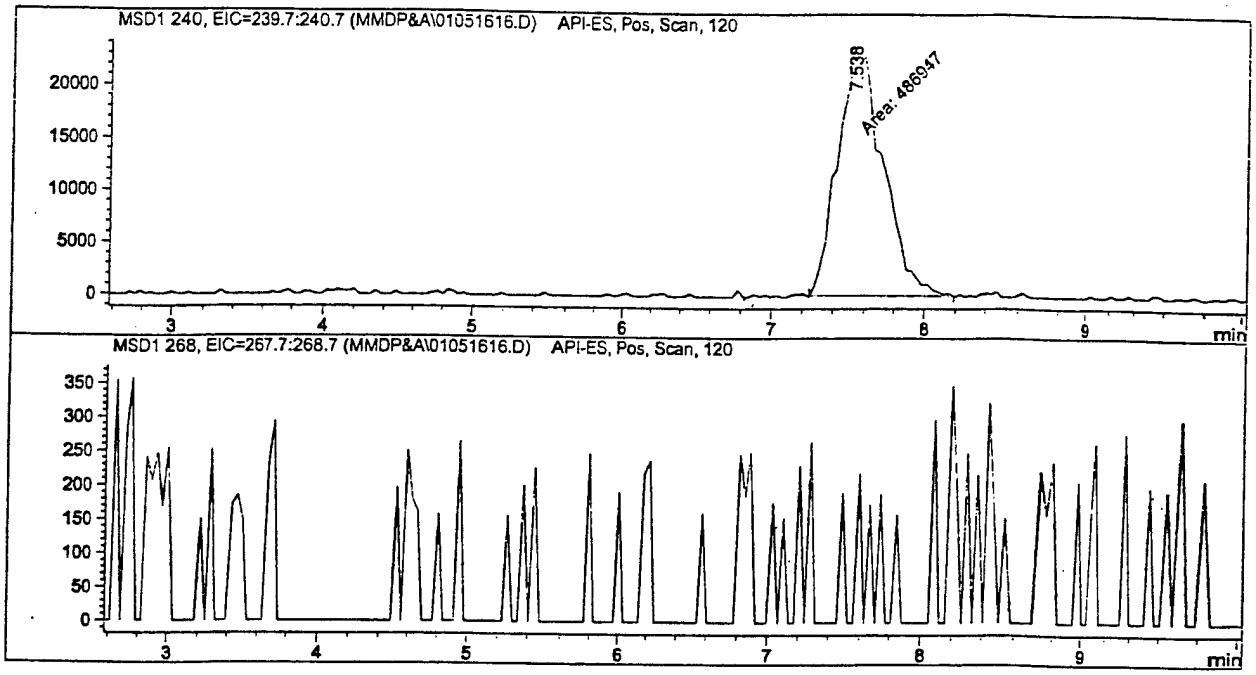
Data File C:\HPCHEM\1\DATA\MMDP&A\01051615.D Sample Name: blank



*** End of Report ***

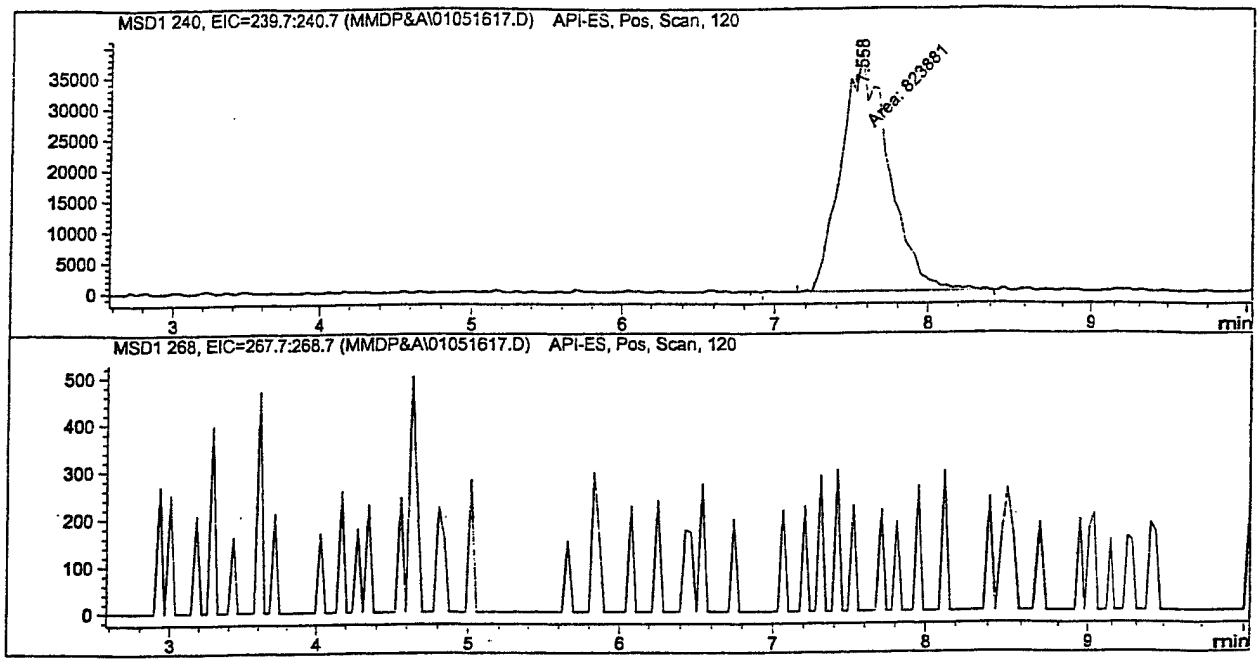
Data File C:\HPCHEM\1\DATA\MMDP&A\01051616.D

Sample Name: 20 ppb



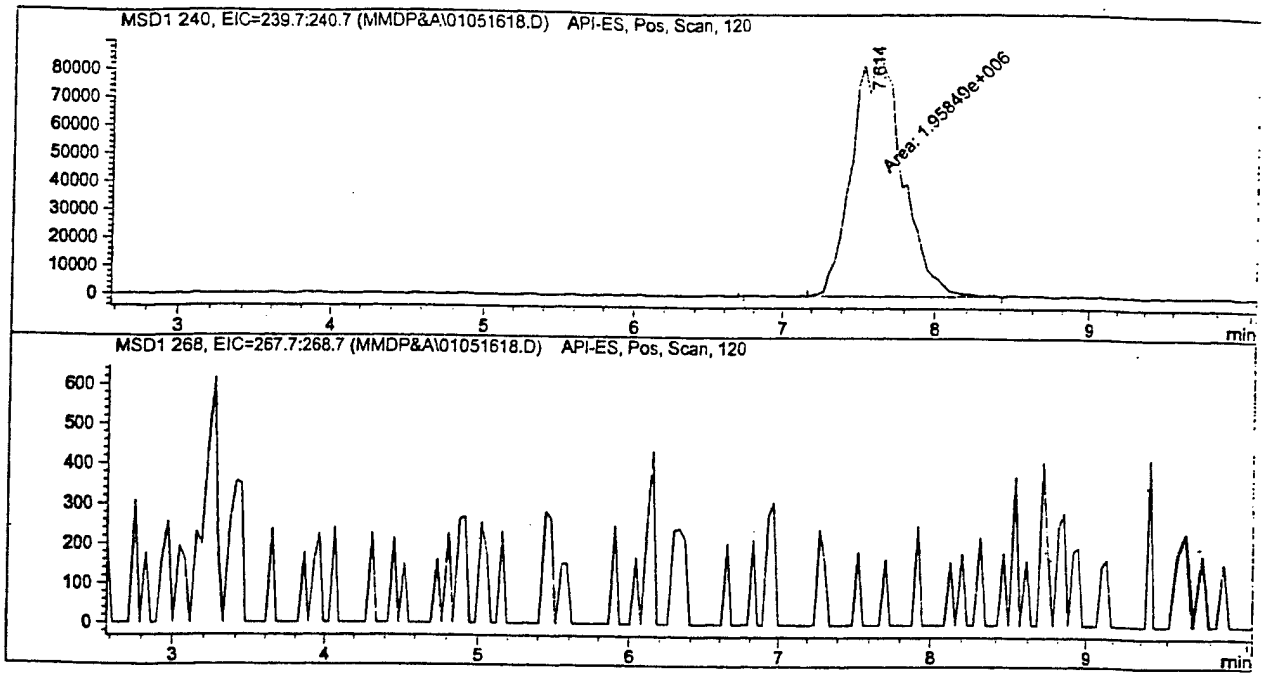
*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMDP&A\01051617.D Sample Name: 40 ppb



*** End of Report ***

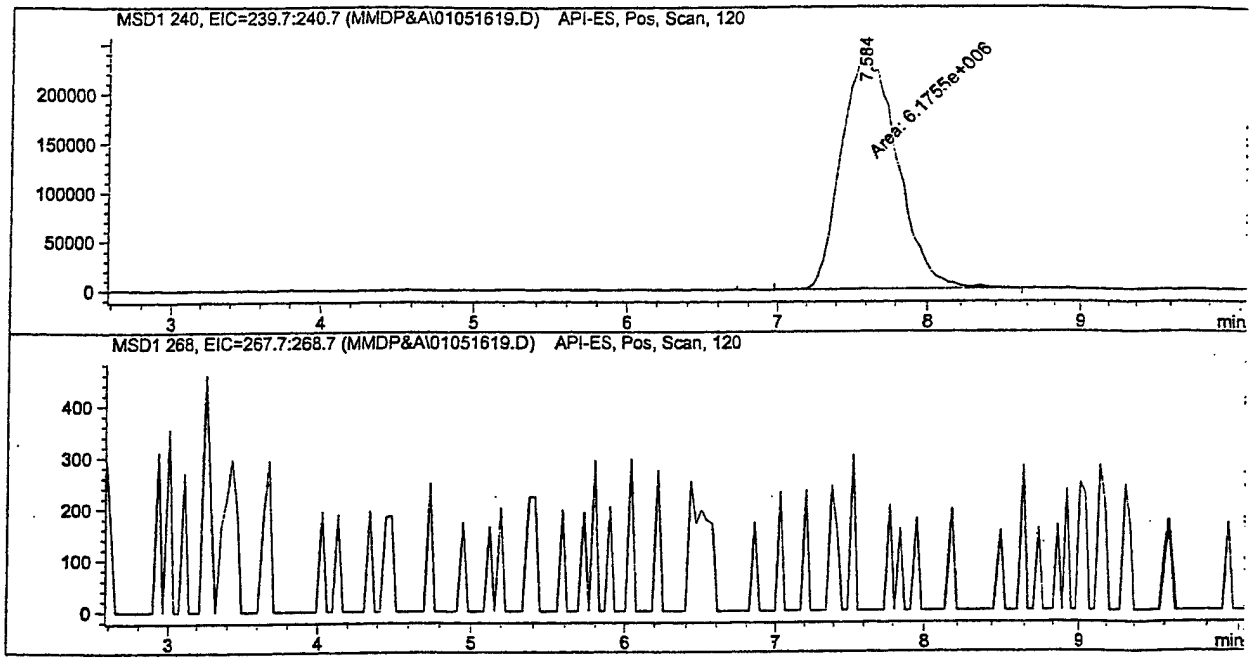
Data File C:\HPCHEM\1\DATA\MMDP&A\01051618.D Sample Name: 100 ppb



*** End of Report ***

Instrument 1 5/17/2001 9:40:15 AM wrc

Data File C:\HPCHEM\1\DATA\MMDP&A\01051619.D Sample Name: 400 ppb



*** End of Report ***

Instrument 1 5/17/2001 9:41:34 AM wrc

BLANK

APPENDIX B

CHROMATOGRAPHIC DATA FROM THE ABBREVIATED P&A OF SAMPLE PREPARATION OF EA-2192 IN MMD DECONTAMINATION SOLUTION USING SOLID PHASE EXTRACTION (SPE)

Table I: Tabulated results of the abbreviated P&A study of EA-2192 in MMD decontamination solution.

Sample	file	signal		
P36B (Blank)	00112010	29453	blank	
	00112015	41629	blank	
	00112020	53438	blank	
	00112025	46016	blank	
P36D (spike)	00112011	4.62E+05		
	00112016	5.37E+05		
	00112021	4.76E+05		
	00112026	4.98E+05		
P36F (spike)	00112012	3.18E+05		
	00112017	3.60E+05		
	00112022	3.82E+05		
	00112027	4.36E+05		
P36H (spike)	00112013	5.33E+05		
	00112018	5.03E+05		
	00112023	6.35E+05		
	00112028	5.24E+05		
Ave.	4.72E+05			
Std dev.	87759.96663			
%RSD	18.59%			
MDL (ppb)	557.4473671			
MDL (ng)	13.93618418			
Recovered amt. (ppb)	2.35	(assume zero intercept)		
Sample Conc. (ppb)	113	(Corrected for dilution of sample only)		
Recovery (%)	2.08%			
EA-2192 Calibration				
file	Signal	Conc.		
00112001	0.00E+00	0		
00112002	1.96E+06	4		
00112003	4.06E+06	20		
00112004	7.84E+06	40		
00112005	1.51E+07	80		
00112030	0.00E+00	0		
00112031	1.15E+06	4		
00112032	5.21E+06	20		
00112033	9.54E+06	40		
00112034	1.72E+07	80		
	slope	4.97917E-06		
	intercept	-2.072844987		
	corr.	0.991789633		

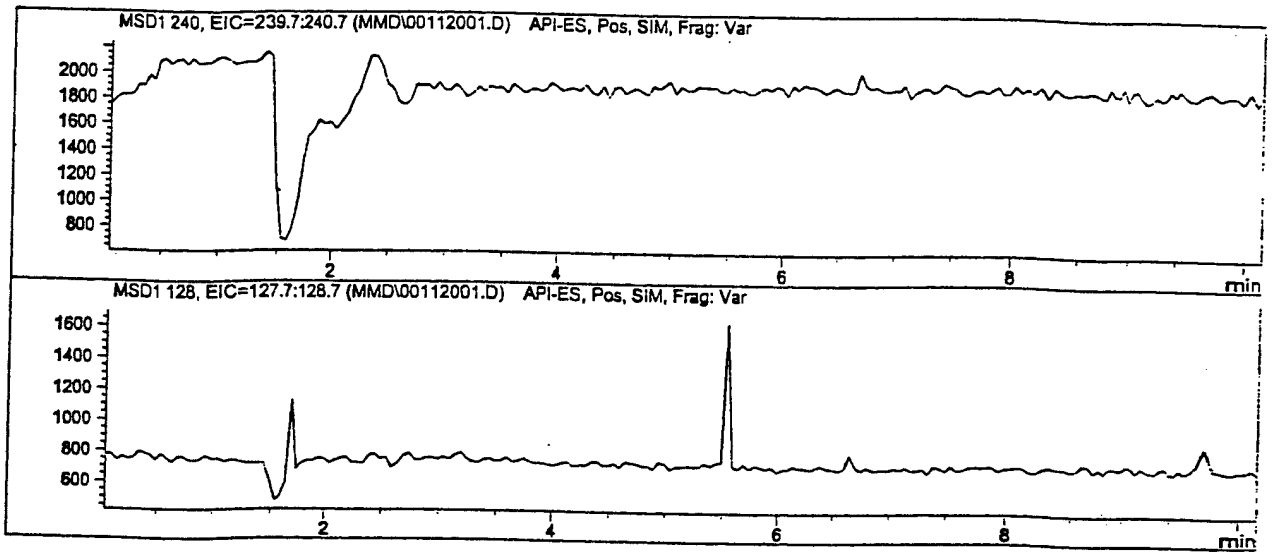
CCV Results	file	signal	Calc. conc.	Percent
40 ppb std.	00112014	1.32E+07	63.46054	159%
	00112019	1.22E+07	58.62427	147%
	00112024	1.21E+07	58.38527	146%
	00112029	1.02E+07	48.77596	122%

Data File C:\HPCHEM\1\DATA\MMD\00112001.D

Sample Name: H20 blank

Injection Date	: 11/20/2000 12:04:41 PM	Seq. Line	: 1
Sample Name	: H20 blank	Location	: Vial 4
Acq. Operator	: wrc	Inj	: 1
		Inj Volume	: 25 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ESPG005M.M		
Last changed	: 11/20/2000 12:01:31 PM by wrc (modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\ESPG005M.M		
Last changed	: 11/28/2000 9:12:07 AM by wrc (modified after loading)		

+ESI gradient method, reversed phase

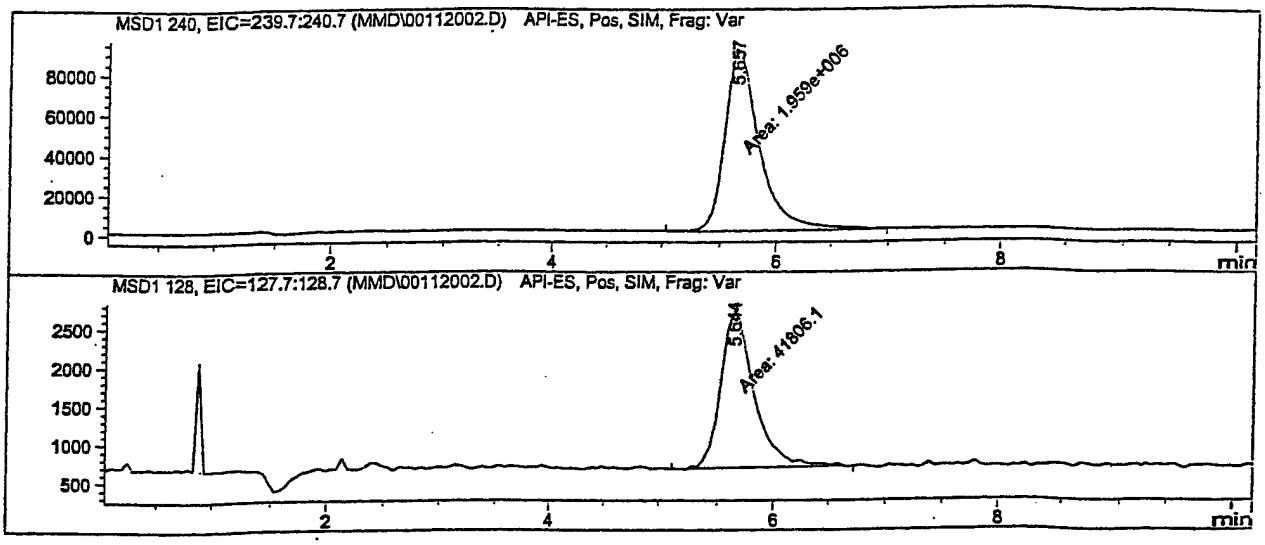


*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112002.D Sample Name: 4 ppb EA-2192

Injection Date : 11/20/2000 12:16:50 PM Seq. Line : 2
Sample Name : 4 ppb EA-2192 Location : Vial 5
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/20/2000 12:16:04 PM by wrc
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

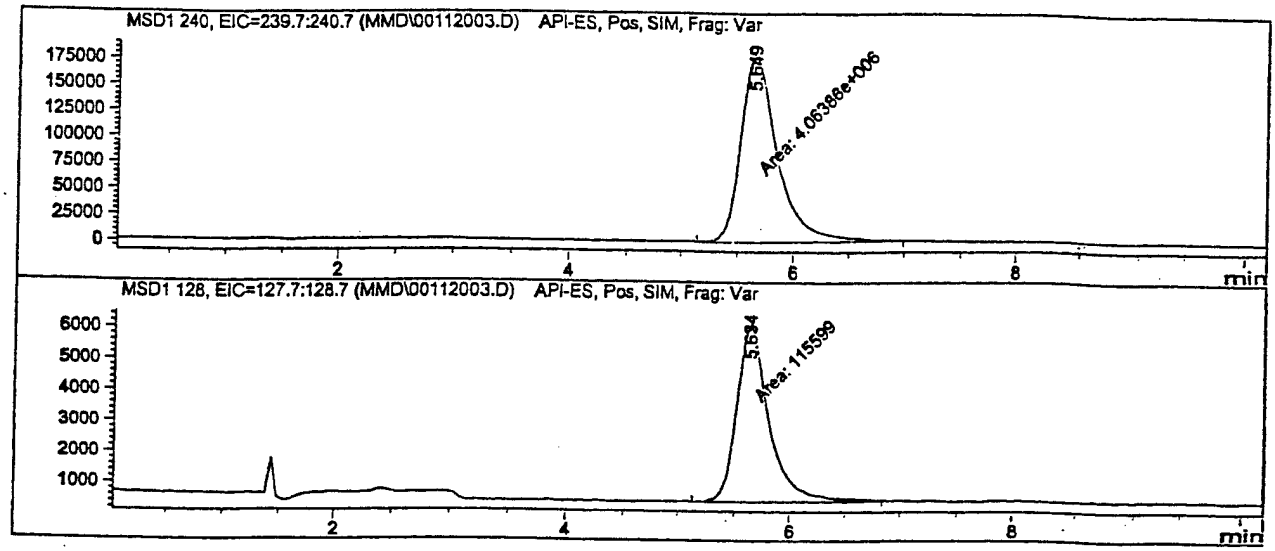
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.657	MM	0.3499	1.95900e6	9.33012e4	100.0000
Totals :				1.95900e6	9.33012e4	

Signal 2: MSD1 128, EIC=127.7:128.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.644	MM	0.3382	4.18061e4	2060.07715	100.0000
Totals :				4.18061e4	2060.07715	

Data File C:\HPCHEM\1\DATA\MMD\00112003.D Sample Name: 20 ppb EA-2192

Injection Date : 11/20/2000 12:28:59 PM Seq. Line : 3
Sample Name : 20 ppb EA-2192 Location : Vial 6
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl
Acq. Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/20/2000 12:28:12 PM by wrc
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.649	MM	0.3698	4.06386e6	1.83132e5	100.0000
Totals :				4.06386e6	1.83132e5	

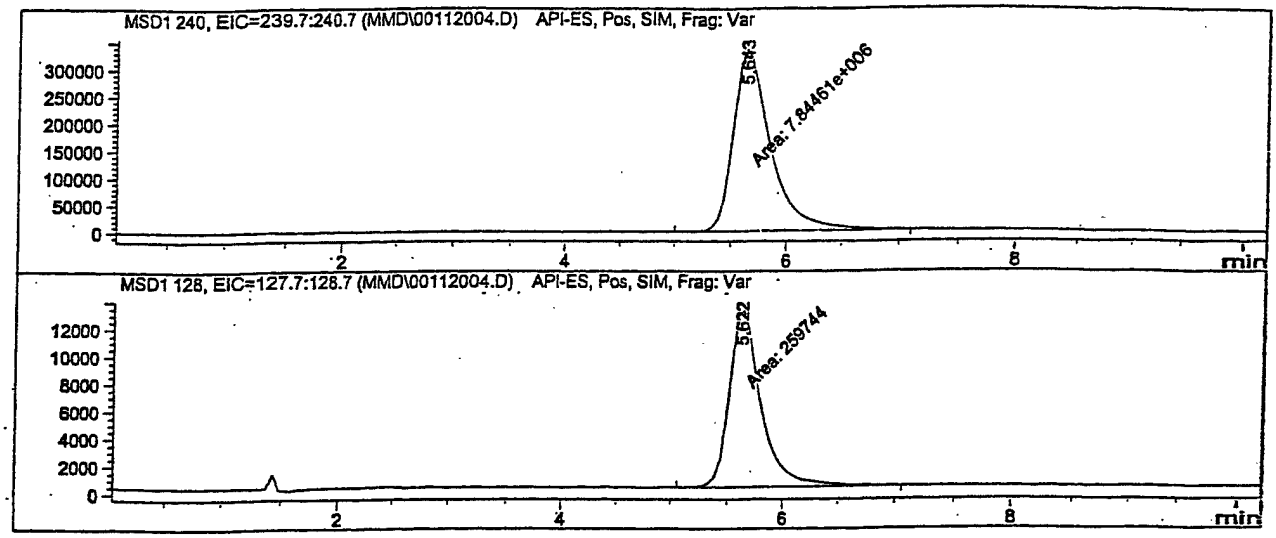
Signal 2: MSD1 128, EIC=127.7:128.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.634	MM	0.3313	1.15599e3	5816.12891	100.0000
Totals :				1.15599e3	5816.12891	

Instrument 1 11/28/2000 9:18:55 AM wrc

Data File C:\HPCHEM\1\DATA\MMD\00112004.D Sample Name: 40 ppb EA-2192

Injection Date : 11/20/2000 12:41:06 PM Seq. Line : 4
Sample Name : 40 ppb EA-2192 Location : Vial 7
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl
Acq. Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/20/2000 12:40:21 PM by wrc
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.643	MM	0.3843	7.84461e6	3.40205e5	100.0000
Totals :				7.84461e6	3.40205e5	

Signal 2: MSD1 128, EIC=127.7:128.7

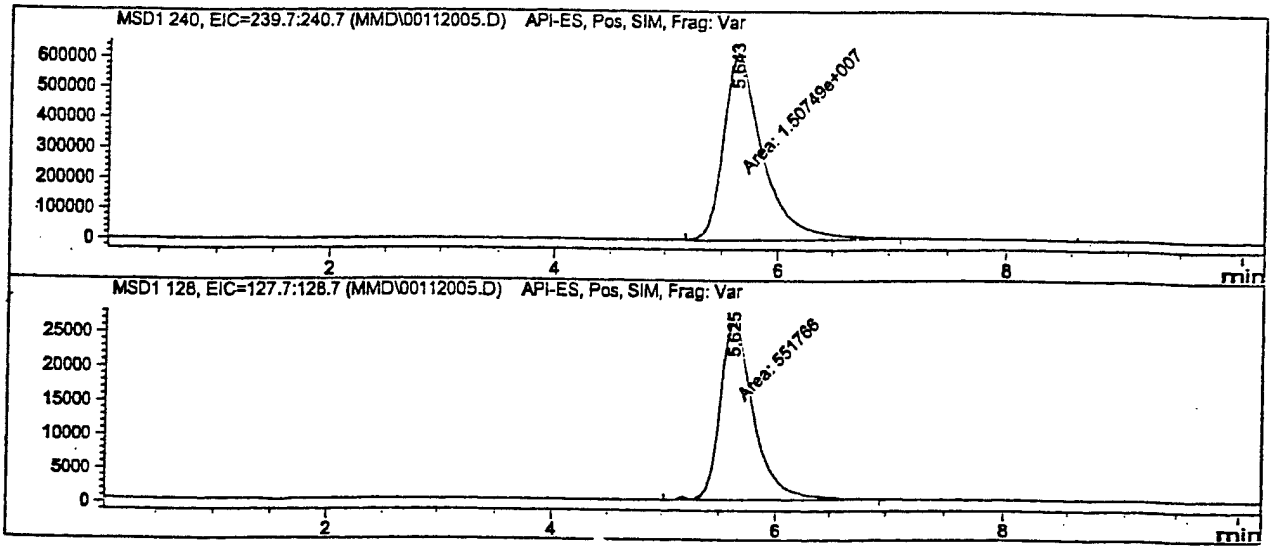
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.622	MM	0.3321	2.59744e5	1.30364e4	100.0000
Totals :				2.59744e5	1.30364e4	

Instrument 1 11/28/2000 9:21:21 AM wrc

Data File C:\HPCHEM\1\DATA\MMD\00112005.D Sample Name: 80 ppb EA-2192

Injection Date : 11/20/2000 12:53:15 PM Seq. Line : 5
Sample Name : 80 ppb EA-2192 Location : Vial 8
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/20/2000 12:52:28 PM by wrc
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

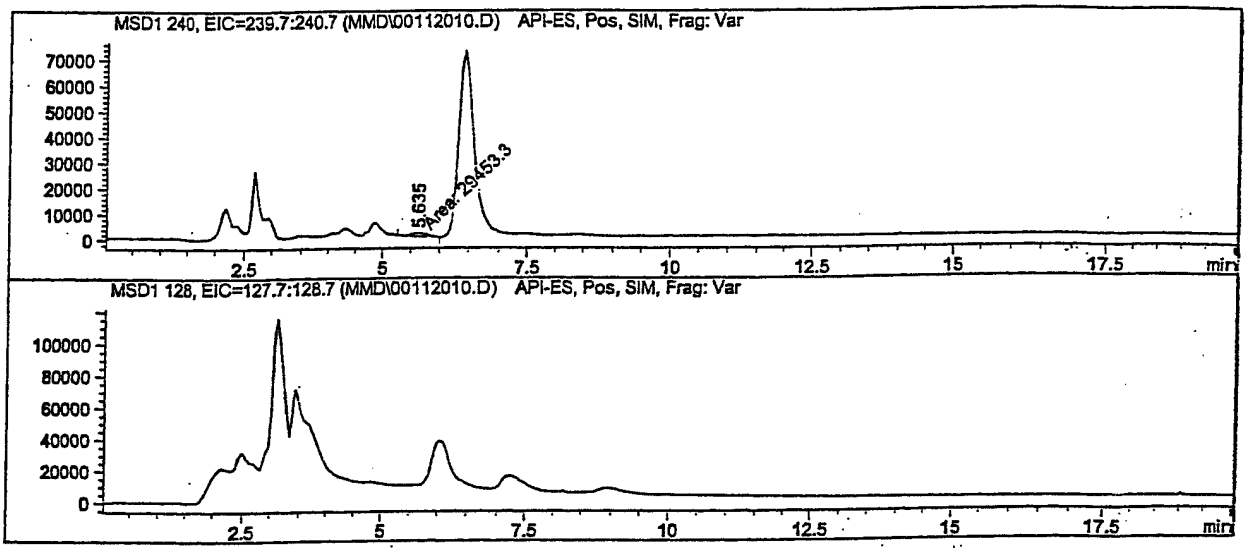
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.643	MM	0.3995	1.50749e7	6.28884e5	100.0000
Totals :				1.50749e7	6.28884e5	

Signal 2: MSD1 128, EIC=127.7:128.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.625	MM	0.3461	5.51766e5	2.65739e4	100.0000
Totals :				5.51766e5	2.65739e4	

Data File C:\HPCHEM\1\DATA\MMD\00112010.D Sample Name: NB114P36B

```
Injection Date  : 11/20/2000 1:49:06 PM        Seq. Line :   1
Sample Name     : NB114P36B                    Location : Vial 11
Acq. Operator   : wrc                             Inj :   1
                                               Inj Volume : 25 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed    : 11/20/2000 1:39:49 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed    : 11/28/2000 9:17:57 AM by wrc
                  (modified after loading)
+ESI gradient method, reversed phase
```




```
                    Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]                          %
----|-------|----|-------|----------|----------|--------|
   1  5.635 MM    0.3500 2.94533e4  1402.52393 100.0000

Totals :                  2.94533e4  1402.52393

Signal 2: MSD1 128, EIC=127.7:128.7

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

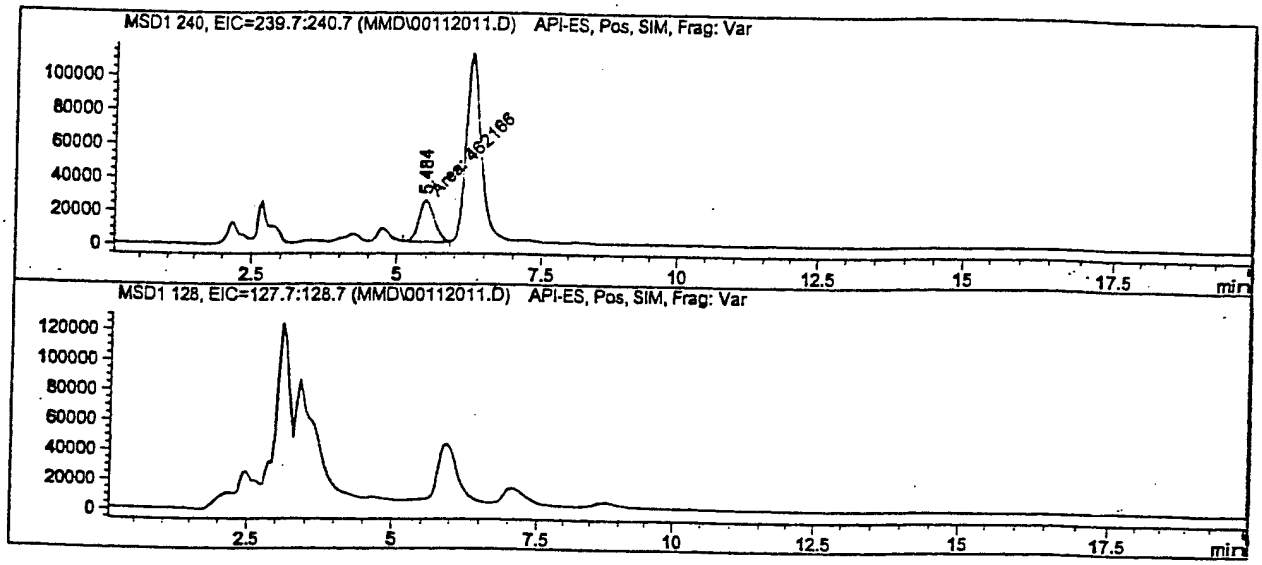
Data File C:\HPCHEM\1\DATA\MMD\00112011.D

Sample Name: NB114P36D

Injection Date : 11/20/2000 2:48:07 PM
Sample Name : NB114P36D
Acq. Operator : wrc

Seq. Line : 1
Location : Vial 12
Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/20/2000 2:27:31 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.484	MM	0.3020	4.62166e5	2.55063e4	100.0000
Totals :				4.62166e5	2.55063e4	

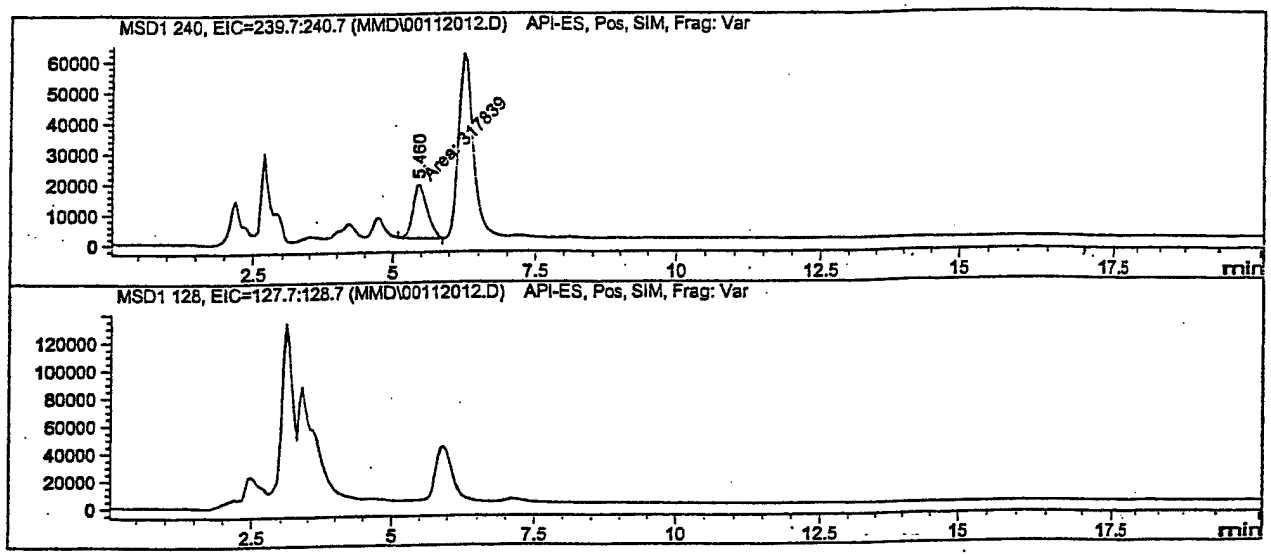
Signal 2: MSD1 128, EIC=127.7:128.7

*** End of Report ***

Instrument 1 11/28/2000 9:24:42 AM wrc

Data File C:\HPCHEM\1\DATA\MMD\00112012.D Sample Name: NB114P36F

```
Injection Date   : 11/20/2000 3:30:03 PM          Seq. Line :   2
Sample Name      : NB114P36F                      Location : Vial 13
Acq. Operator    : wrc                                Inj :   1
                                                Inj Volume : 25 µl
Acq. Method      : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed     : 11/20/2000 2:27:31 PM by wrc
Analysis Method  : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed     : 11/28/2000 9:17:57 AM by wrc
                   (modified after loading)
+ESI gradient method, reversed phase
```



Area Percent Report

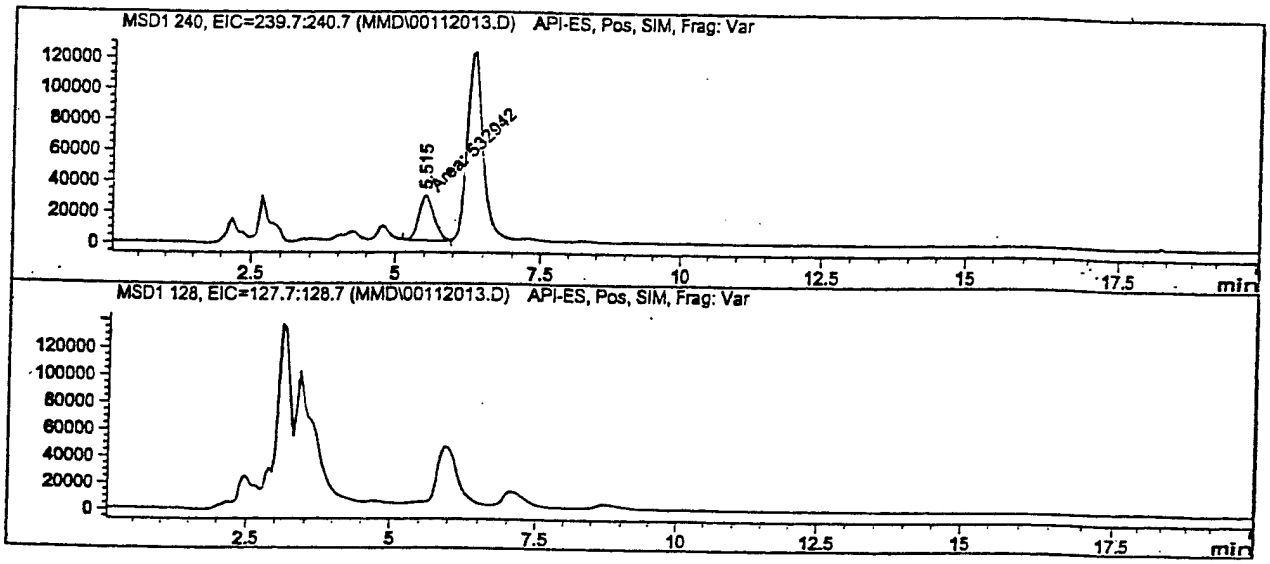
```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]                          %
----|-------|----|-------|----------|----------|--------|
   1   5.460 MM    0.2965 3.17839e5  1.78684e4  100.0000

Totals :                  3.17839e5  1.78684e4

Signal 2: MSD1 128, EIC=127.7:128.7
```

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112013.D Sample Name: NB114P36H

Injection Date : 11/20/2000 4:11:59 PM Seq. Line : 3
Sample Name : NB114P36H Location : Vial 14
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/20/2000 2:27:31 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.515	MM	0.3016	5.32941e5	2.94509e4	100.0000
Totals :				5.32942e5	2.94509e4	

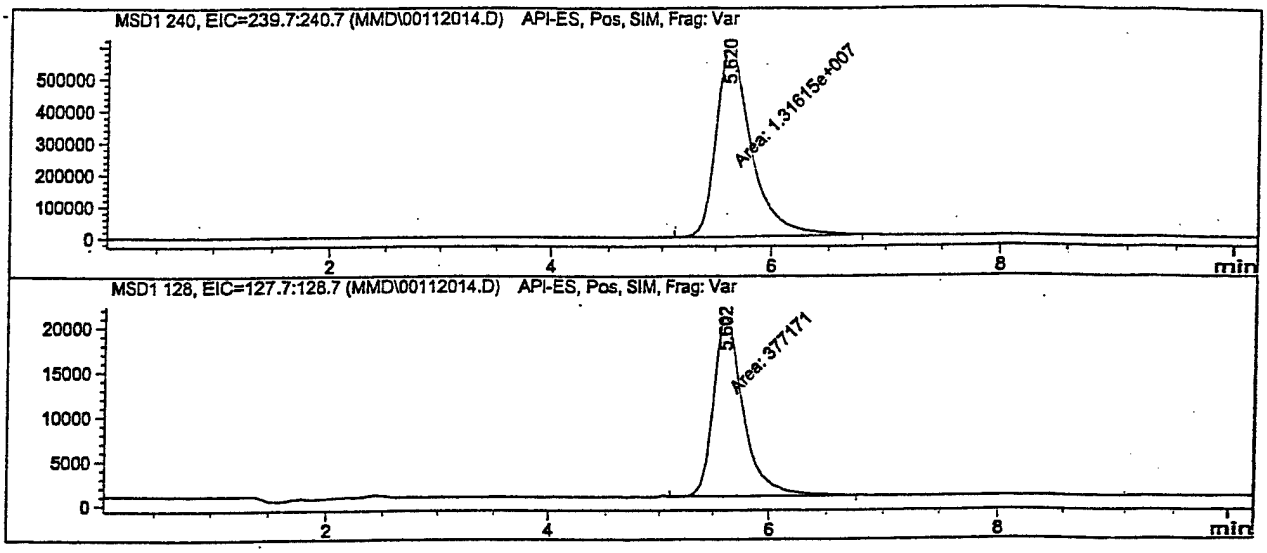
Signal 2: MSD1 128, EIC=127.7:128.7

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112014.D Sample Name: 40 ppb EA-2192

Injection Date : 11/20/2000 4:54:06 PM Seq. Line : 4
Sample Name : 40 ppb EA-2192 Location : Vial 7
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005S.M
Last changed : 11/20/2000 1:39:21 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.620	MM	0.3643	1.31615e7	6.02220e5	100.0000
Totals :				1.31615e7	6.02220e5	

Signal 2: MSD1 128, EIC=127.7:128.7

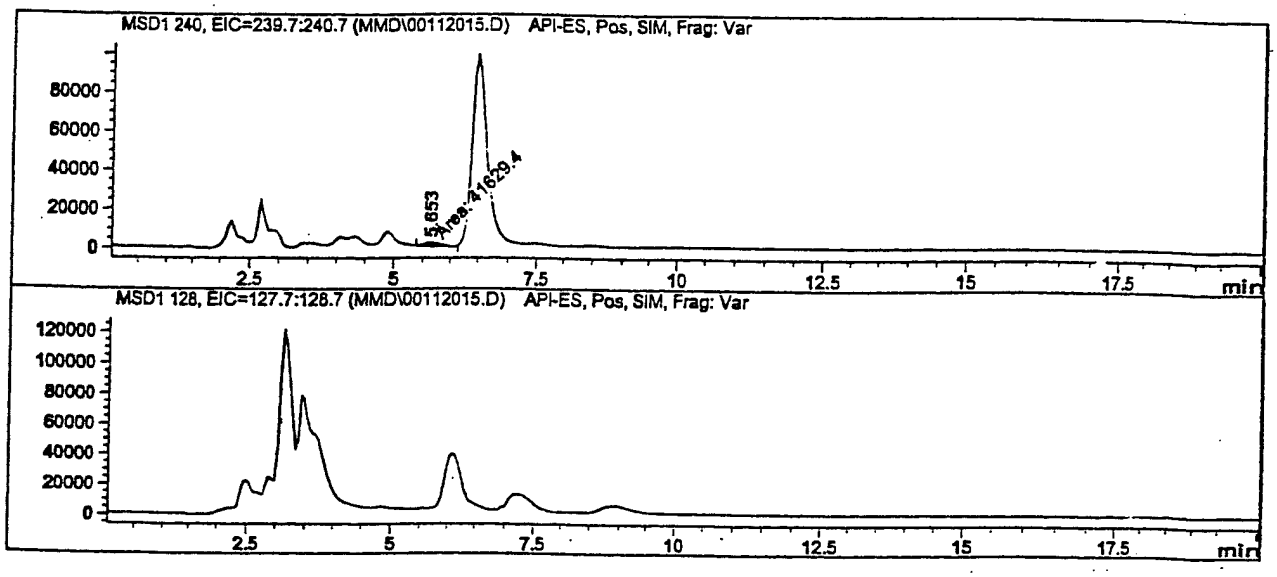
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.602	MM	0.3018	3.77171e5	2.08291e4	100.0000
Totals :				3.77171e5	2.08291e4	

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112015.D Sample Name: NB114P36B

Injection Date : 11/20/2000 5:06:25 PM Seq. Line : 5
Sample Name : NB114P36B Location : Vial 11
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/20/2000 2:27:31 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.653	MM	0.3603	4.16294e4	1925.53577	100.0000
Totals :				4.16294e4	1925.53577	

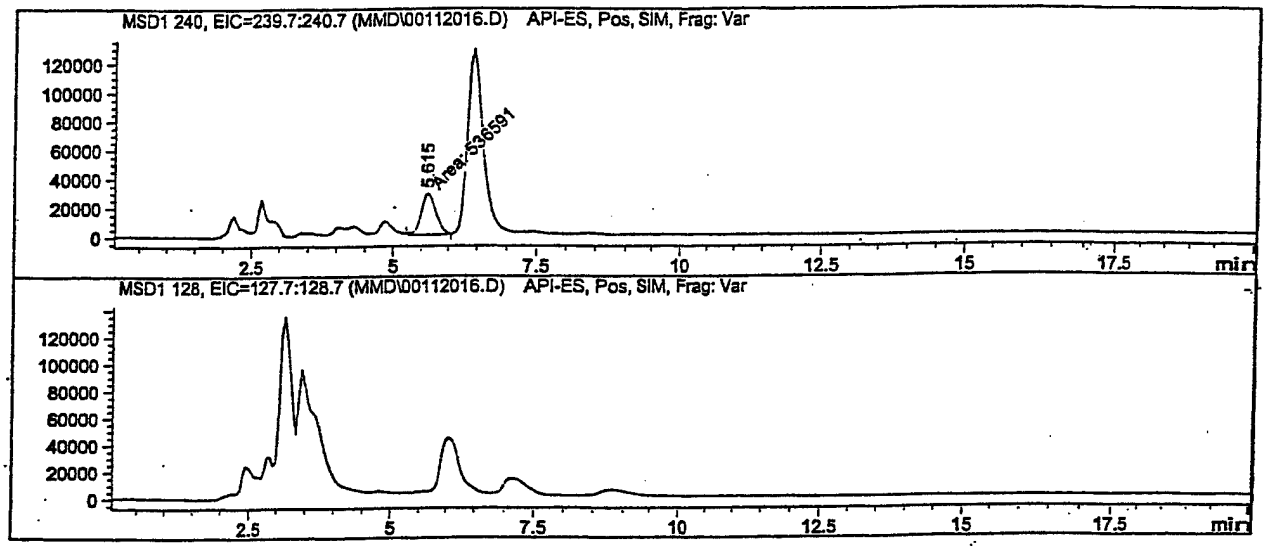
Signal 2: MSD1 128, EIC=127.7:128.7

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112016.D Sample Name: NB114P36D

Injection Date	: 11/20/2000 5:48:21 PM	Seq. Line	: 6
Sample Name	: NB114P36D	Location	: Vial 12
Acq. Operator	: wrc	Inj	: 1
		Inj Volume	: 25 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ESPG005M.M		
Last changed	: 11/20/2000 2:27:31 PM by wrc		
Analysis Method	: C:\HPCHEM\1\METHODS\ESPG005M.M		
Last changed	: 11/28/2000 9:17:57 AM by wrc (modified after loading)		

+ESI gradient method, reversed phase



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.615	MM	0.3102	5.36591e5	2.88301e4	100.0000
Totals :				5.36591e5	2.88301e4	

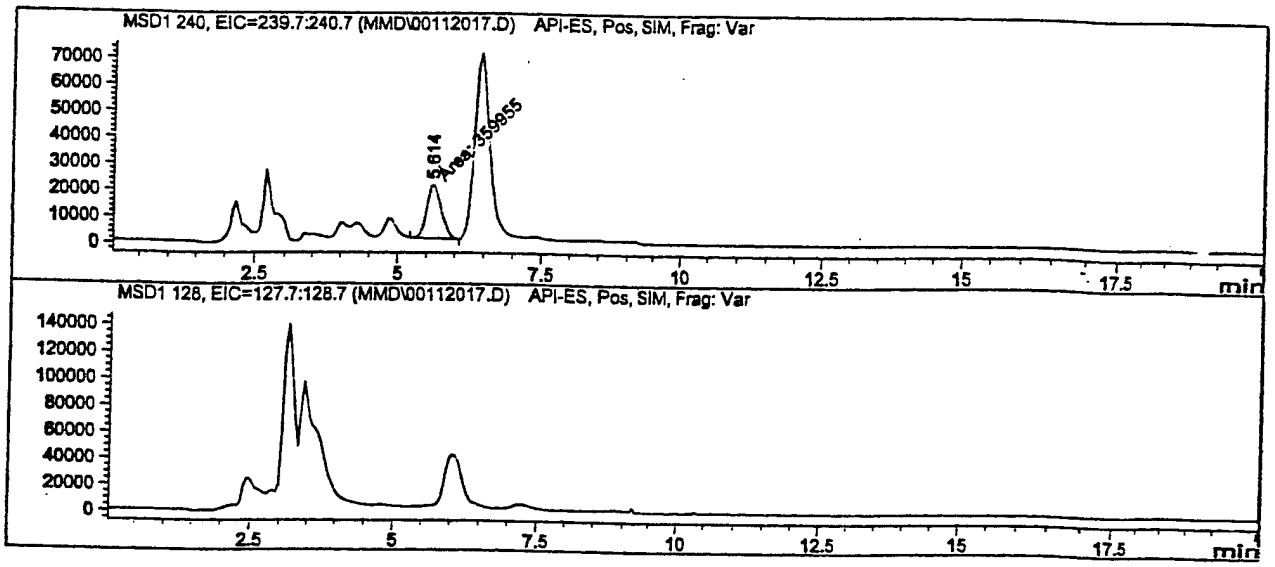
Signal 2: MSD1 128, EIC=127.7:128.7

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112017.D Sample Name: NB114P36F

Injection Date	: 11/20/2000 6:30:17 PM	Seq. Line	: 7
Sample Name	: NB114P36F	Location	: Vial 13
Acq. Operator	: wrc	Inj	: 1
		Inj Volume	: 25 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ESPG005M.M		
Last changed	: 11/20/2000 2:27:31 PM by wrc		
Analysis Method	: C:\HPCHEM\1\METHODS\ESPG005M.M		
Last changed	: 11/28/2000 9:17:57 AM by wrc (modified after loading)		

+ESI gradient method, reversed phase



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.614	MM	0.2939	3.59955e5	2.04154e4	100.0000
Totals :				3.59955e5	2.04154e4	

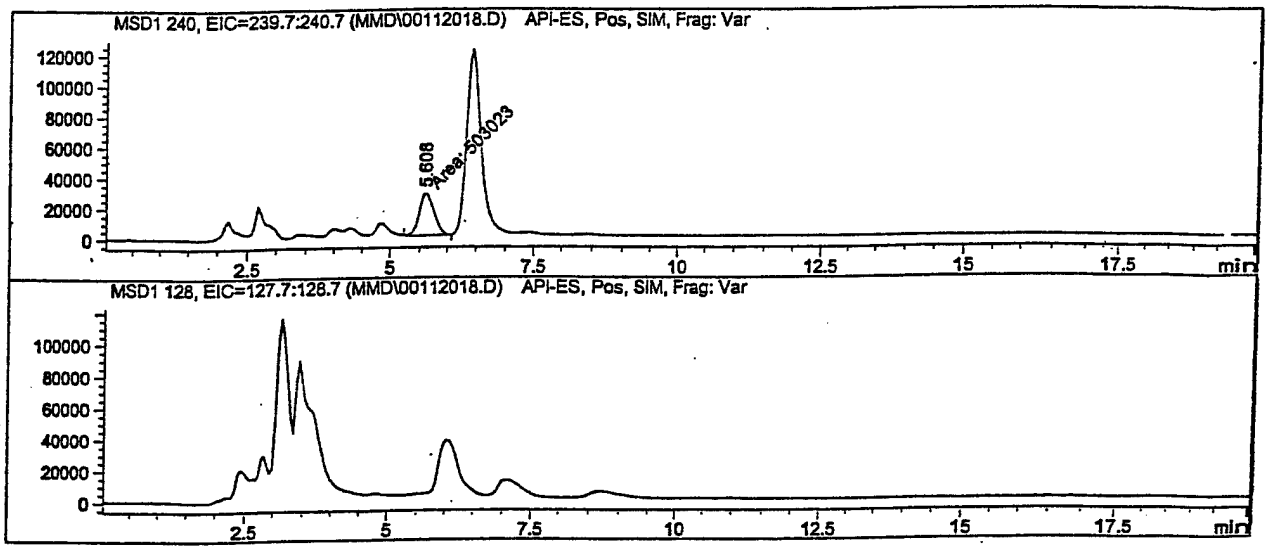
Signal 2: MSD1 128, EIC=127.7:128.7

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112018.D Sample Name: NB114P36H

Injection Date : 11/20/2000 7:12:12 PM Seq. Line : 8
Sample Name : NB114P36H Location : Vial 14
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/20/2000 2:27:31 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.608	MM	0.2941	5.03023e5	2.85081e4	100.0000
Totals :				5.03023e5	2.85081e4	

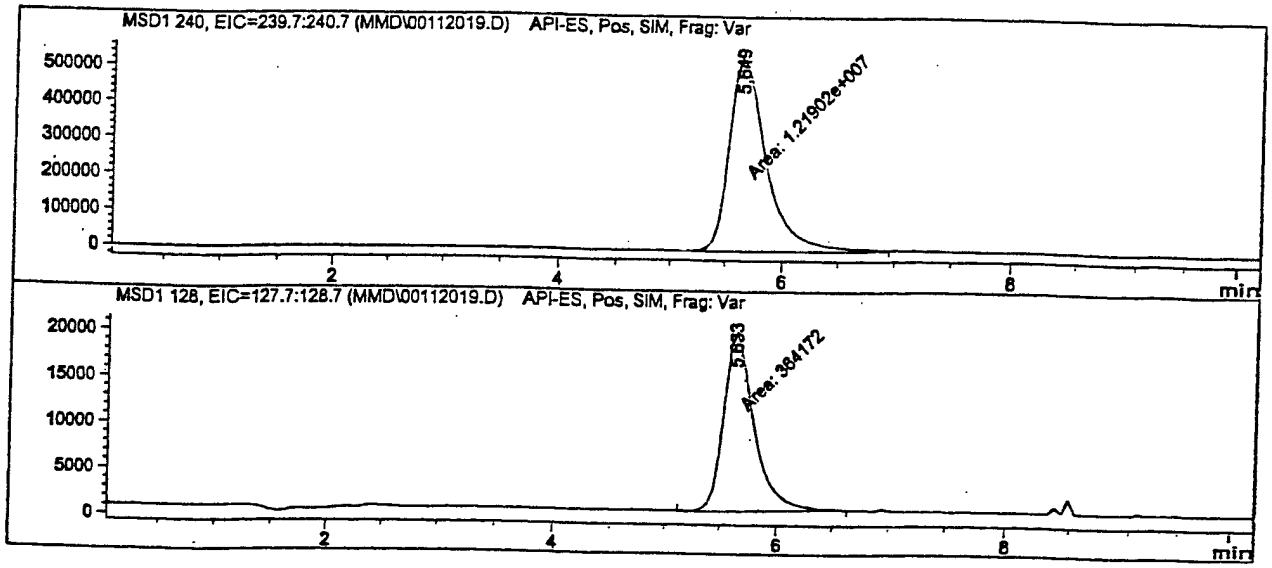
Signal 2: MSD1 128, EIC=127.7:128.7

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112019.D Sample Name: 40 ppb EA-2192

Injection Date : 11/20/2000 7:54:20 PM Seq. Line : 9
Sample Name : 40 ppb EA-2192 Location : Vial 7
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005S.M
Last changed : 11/20/2000 1:39:21 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.649	MM	0.3729	1.21902e7	5.44900e5	100.0000
Totals :				1.21902e7	5.44901e5	

Signal 2: MSD1 128, EIC=127.7:128.7

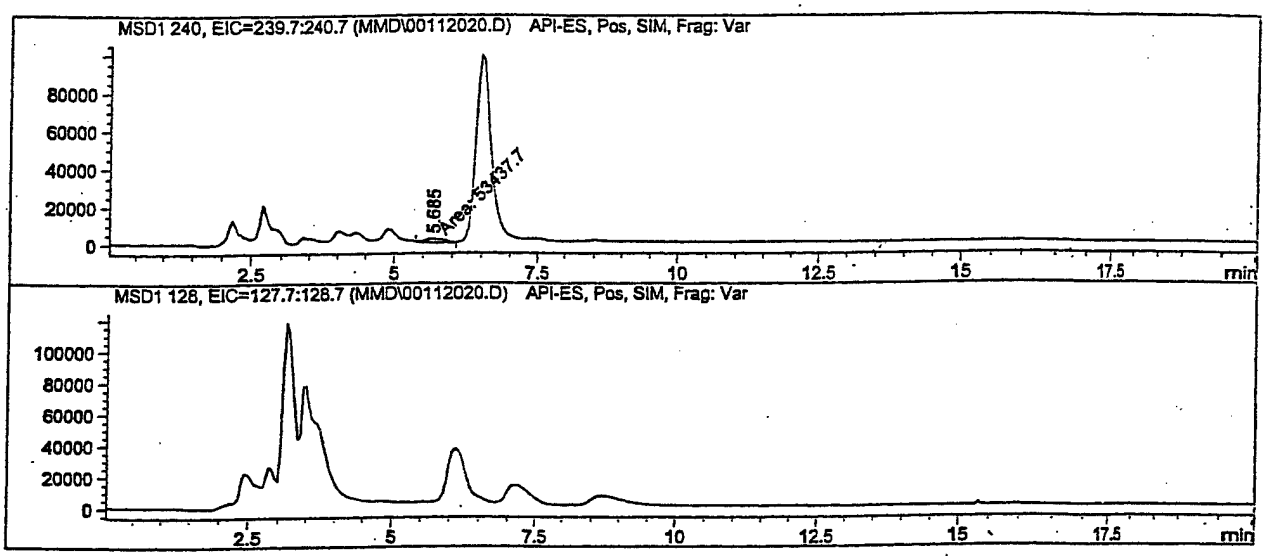
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.633	MM	0.3084	3.64172e5	1.96835e4	100.0000
Totals :				3.64172e5	1.96835e4	

*** End of Report ***

Instrument 1 11/28/2000 9:29:51 AM wrc

Data File C:\HPCHEM\1\DATA\MMD\00112020.D Sample Name: NB114P36B

```
Injection Date    : 11/20/2000 8:06:40 PM          Seq. Line :  10
Sample Name       : NB114P36B                       Location : Vial 11
Acq. Operator     : wrc                                 Inj :  1
                                                  Inj Volume : 25 µl
Acq. Method       : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed      : 11/20/2000 2:27:31 PM by wrc
Analysis Method   : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed      : 11/28/2000 9:17:57 AM by wrc
                    (modified after loading)
+ESI gradient method, reversed phase
```



Area Percent Report

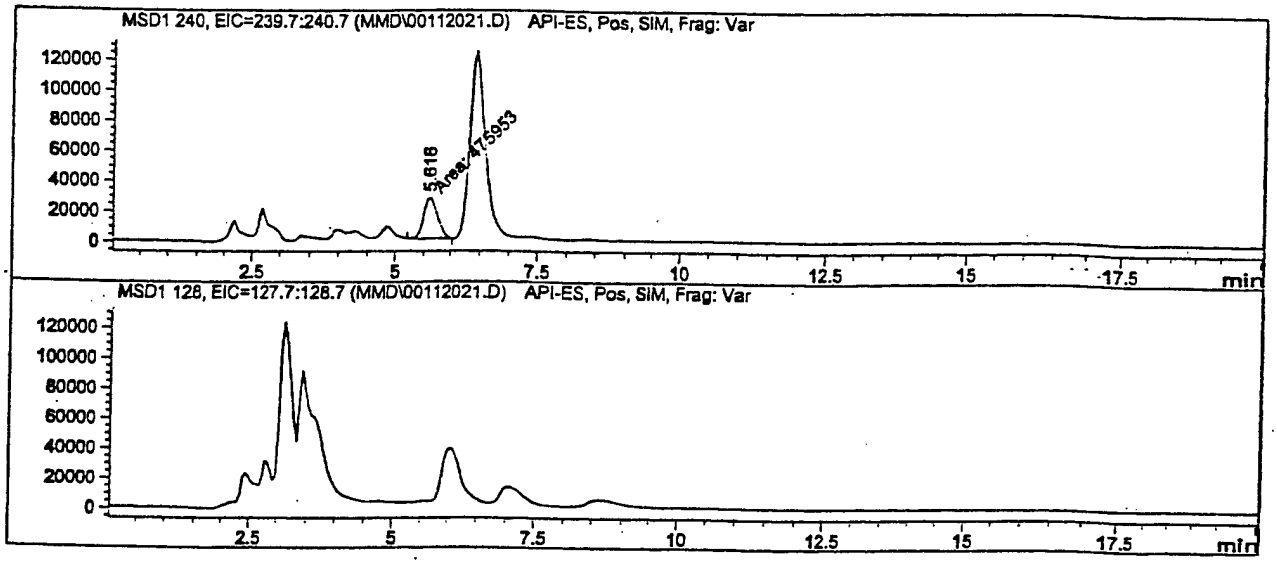
```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]                          %
----|-------|----|-------|----------|----------|--------|
   1   5.685 MM    0.4007 5.34377e4  2222.49707 100.0000

Totals :                  5.34377e4  2222.49707

Signal 2: MSD1 128, EIC=127.7:128.7
```

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112021.D Sample Name: NB114P36D

Injection Date : 11/20/2000 8:48:34 PM Seq. Line : 11
Sample Name : NB114P36D Location : Vial 12
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/20/2000 2:27:31 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.616	MM	0.2923	4.75953e5	2.71395e4	100.0000
Totals :				4.75953e5	2.71395e4	

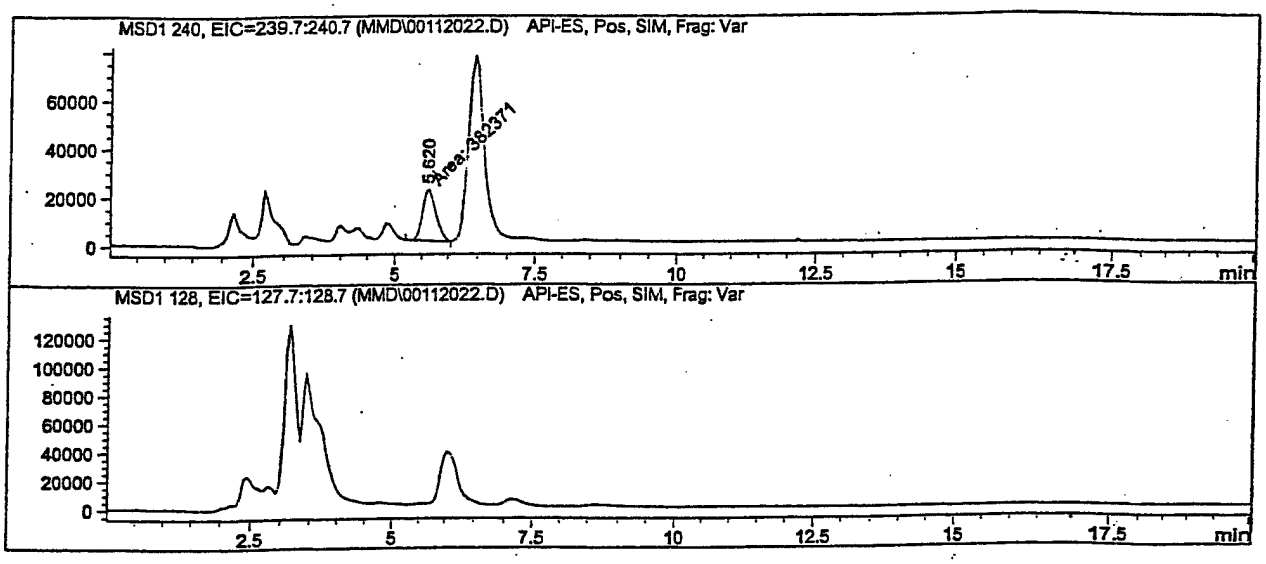
Signal 2: MSD1 128, EIC=127.7:128.7

*** End of Report ***

Instrument 1 11/28/2000 9:31:31 AM wrc Page 1 of 1

Data File C:\HPCHEM\1\DATA\MMD\00112022.D Sample Name: NB114P36F

```
Injection Date    : 11/20/2000 9:30:31 PM        Seq. Line :  12
Sample Name       : NB114P36F                    Location : Vial 13
Acq. Operator     : wrc                             Inj :   1
                                                 Inj Volume : 25 µl

Acq. Method       : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed      : 11/20/2000 2:27:31 PM by wrc
Analysis Method   : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed      : 11/28/2000 9:17:57 AM by wrc
                    (modified after loading)
+ESI gradient method, reversed phase
```



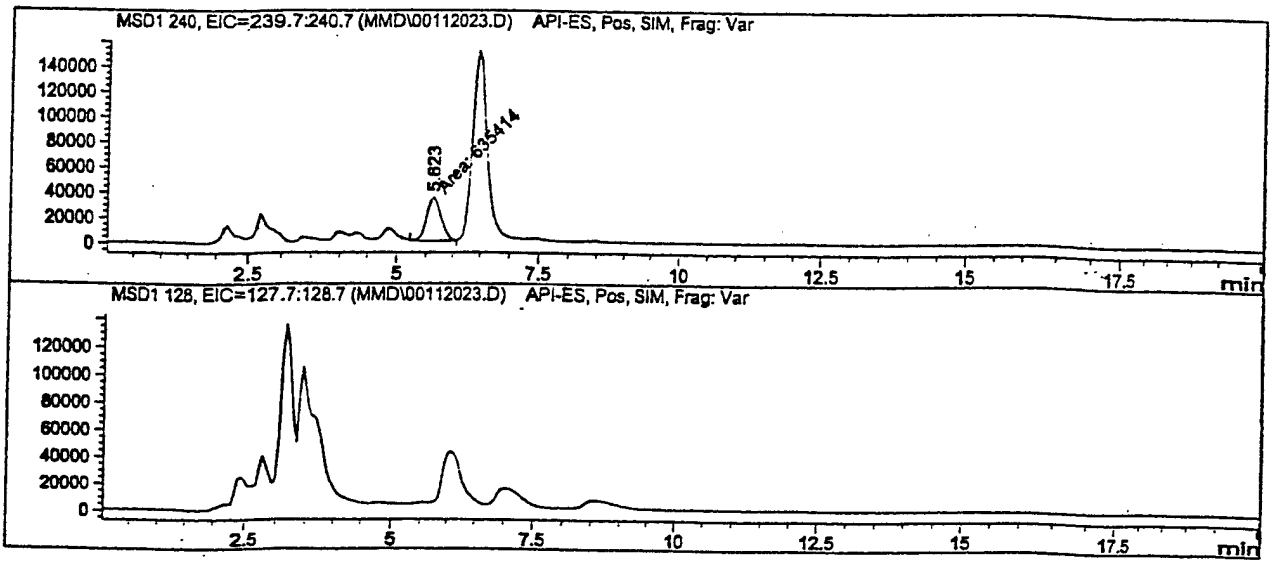
Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]                          %
----|-------|----|-------|----------|----------|--------|
   1   5.620 MM    0.2972 3.82371e5  2.14461e4  100.0000

Totals :                  3.82371e5  2.14461e4

Signal 2: MSD1 128, EIC=127.7:128.7
```

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112023.D Sample Name: NB114P36H

Injection Date : 11/20/2000 10:12:26 PM Seq. Line : 13
Sample Name : NB114P36H Location : Vial 14
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl
Acq. Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/20/2000 2:27:31 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.623	MM	0.3054	6.35414e5	3.46790e4	100.0000

Totals : 6.35414e5 3.46790e4

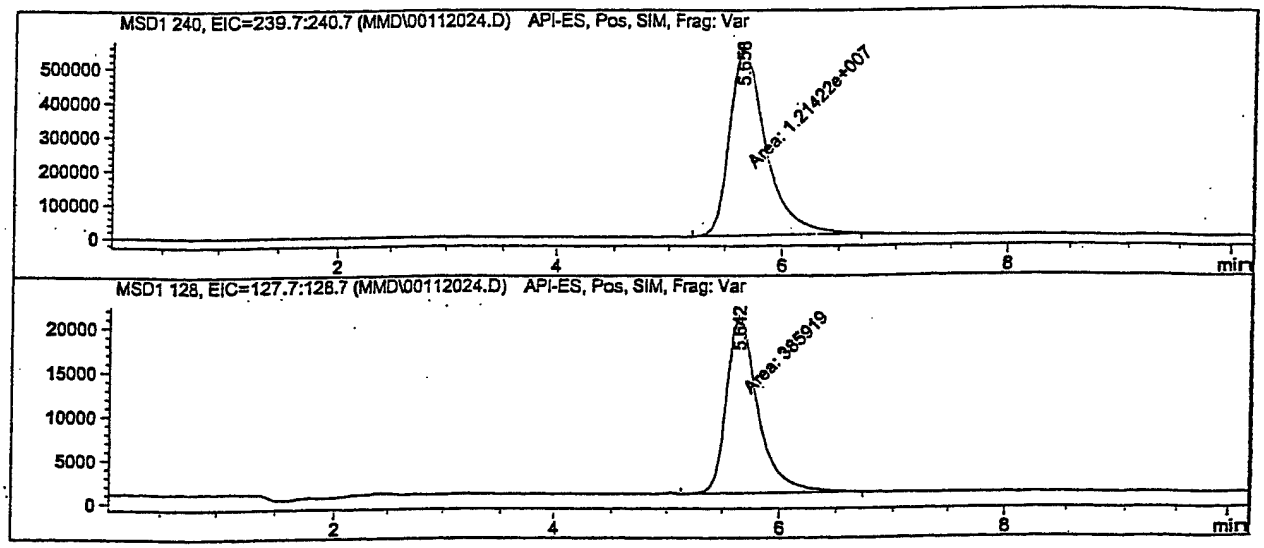
Signal 2: MSD1 128, EIC=127.7:128.7

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112024.D Sample Name: 40 ppb EA-2192

Injection Date : 11/20/2000 10:54:34 PM Seq. Line : 14
Sample Name : 40 ppb EA-2192 Location : Vial 7
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005S.M
Last changed : 11/20/2000 1:39:21 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.658	MM	0.3620	1.21422e7	5.59011e5	100.0000
Totals :				1.21422e7	5.59011e5	

Signal 2: MSD1 128, EIC=127.7:128.7

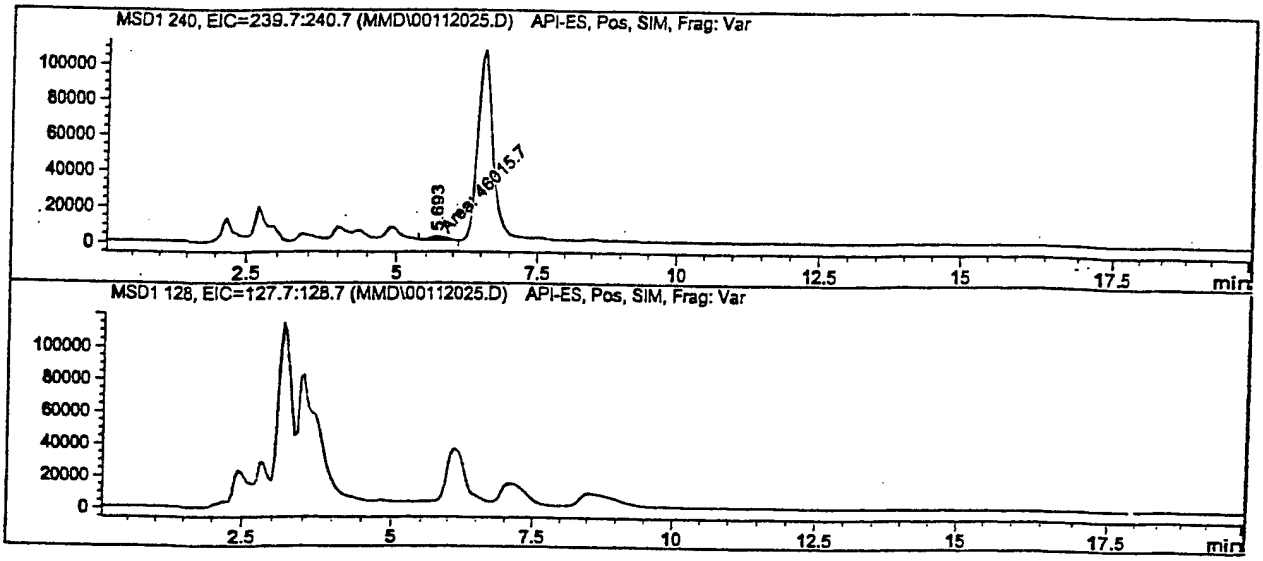
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.642	MM	0.3150	3.85919e5	2.04201e4	100.0000
Totals :				3.85919e5	2.04201e4	

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112025.D Sample Name: NB114P36B

Injection Date : 11/20/2000 11:06:53 PM Seq. Line : 15
Sample Name : NB114P36B Location : Vial 11
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/20/2000 2:27:31 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.693	MM	0.3623	4.60157e4	2116.61572	100.0000
Totals :				4.60157e4	2116.61572	

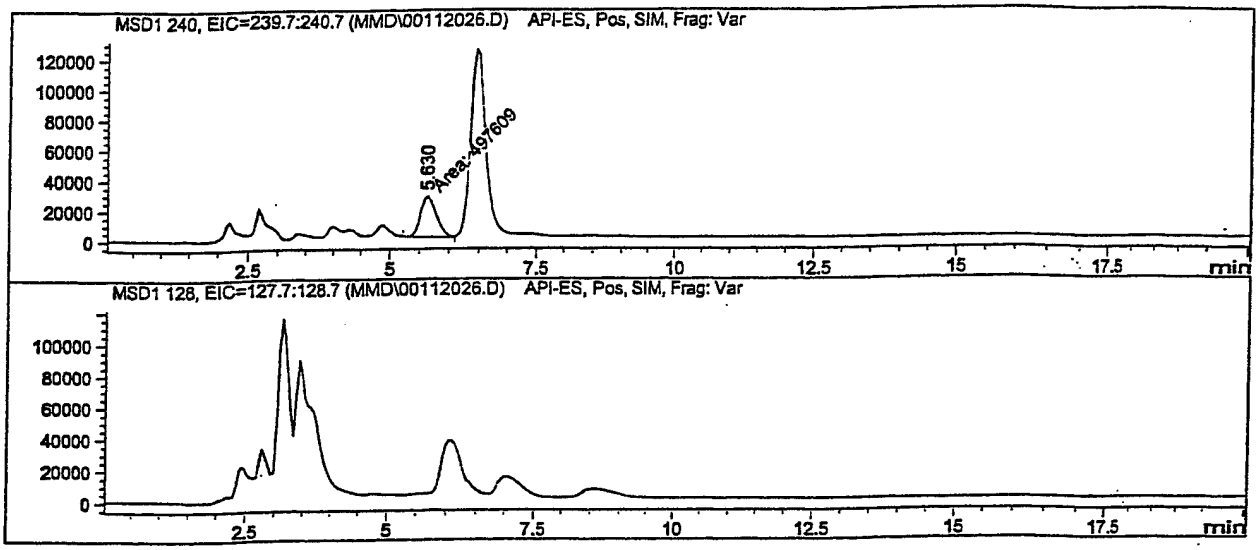
Signal 2: MSD1 128, EIC=127.7:128.7

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112026.D Sample Name: NB114P36D

Injection Date : 11/20/2000 11:48:49 PM Seq. Line : 16
Sample Name : NB114P36D Location : Vial 12
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/20/2000 2:27:31 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.630	MM	0.3021	4.97609e5	2.74505e4	100.0000
Totals :				4.97609e5	2.74505e4	

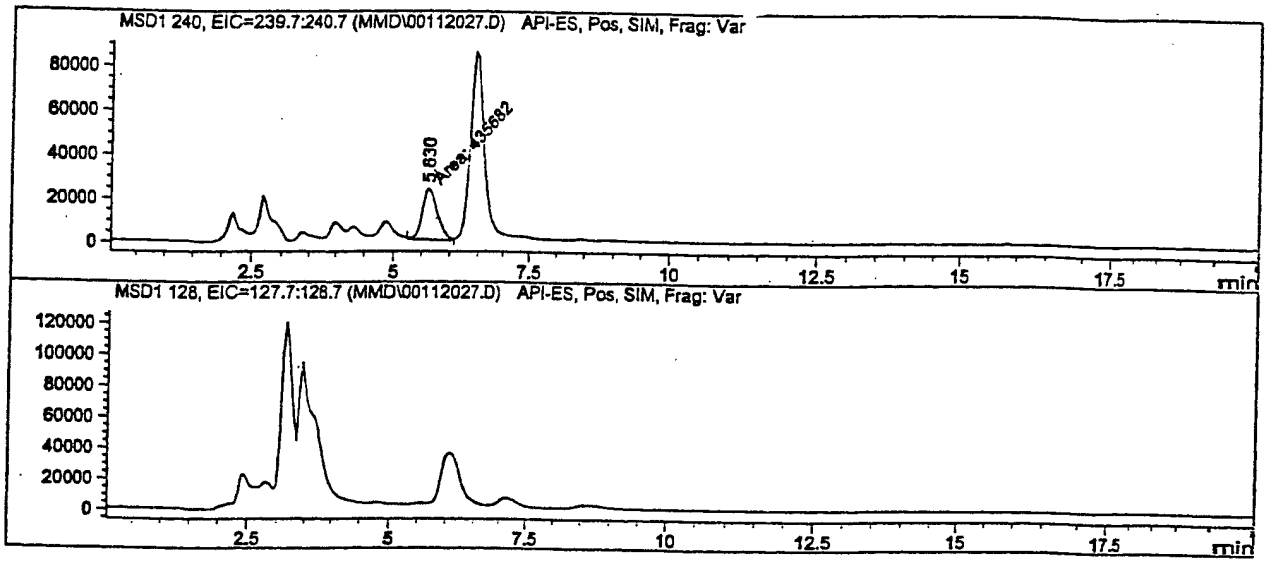
Signal 2: MSD1 128, EIC=127.7:128.7

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112027.D Sample Name: NB114P36F

Injection Date	: 11/21/2000 12:30:46 AM	Seq. Line	: 17
Sample Name	: NB114P36F	Location	: Vial 13
Acq. Operator	: wrc	Inj	: 1
		Inj Volume	: 25 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ESPG005M.M		
Last changed	: 11/20/2000 2:27:31 PM by wrc		
Analysis Method	: C:\HPCHEM\1\METHODS\ESPG005M.M		
Last changed	: 11/28/2000 9:17:57 AM by wrc (modified after loading)		

+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.630	MM	0.3071	4.35682e5	2.36473e4	100.0000
Totals :				4.35682e5	2.36473e4	

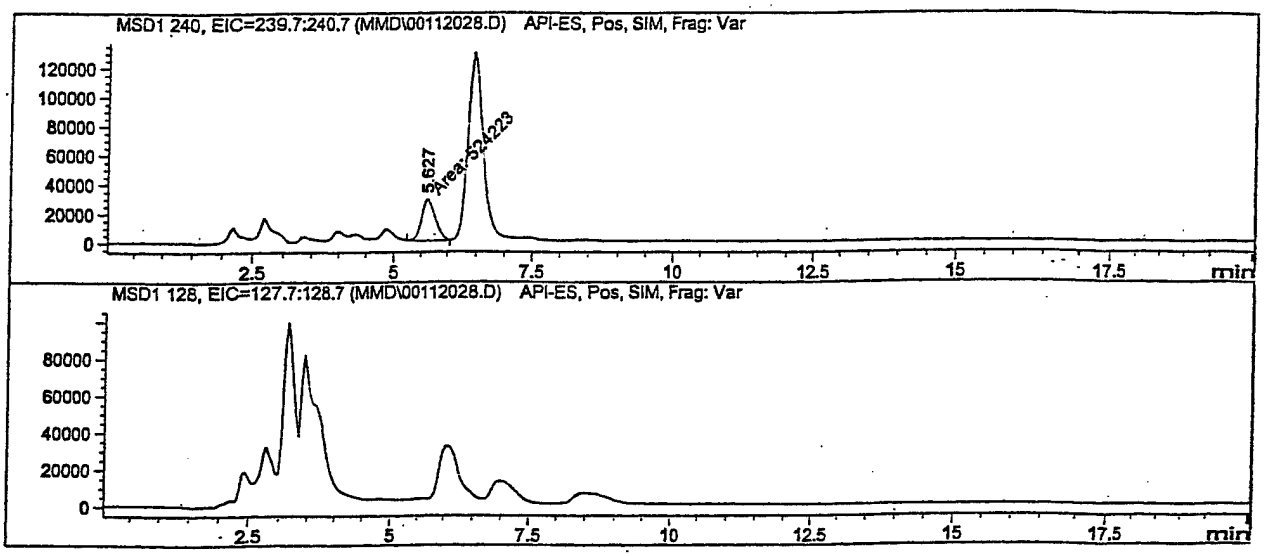
Signal 2: MSD1 128, EIC=127.7:128.7

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112028.D Sample Name: NB114P36H

Injection Date	: 11/21/2000 1:12:42 AM	Seq. Line	: 18
Sample Name	: NB114P36H	Location	: Vial 14
Acq. Operator	: wrc	Inj	: 1
		Inj Volume	: 25 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ESPG005M.M		
Last changed	: 11/20/2000 2:27:31 PM by wrc		
Analysis Method	: C:\HPCHEM\1\METHODS\ESPG005M.M		
Last changed	: 11/28/2000 9:17:57 AM by wrc (modified after loading)		

+ESI gradient method, reversed phase



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

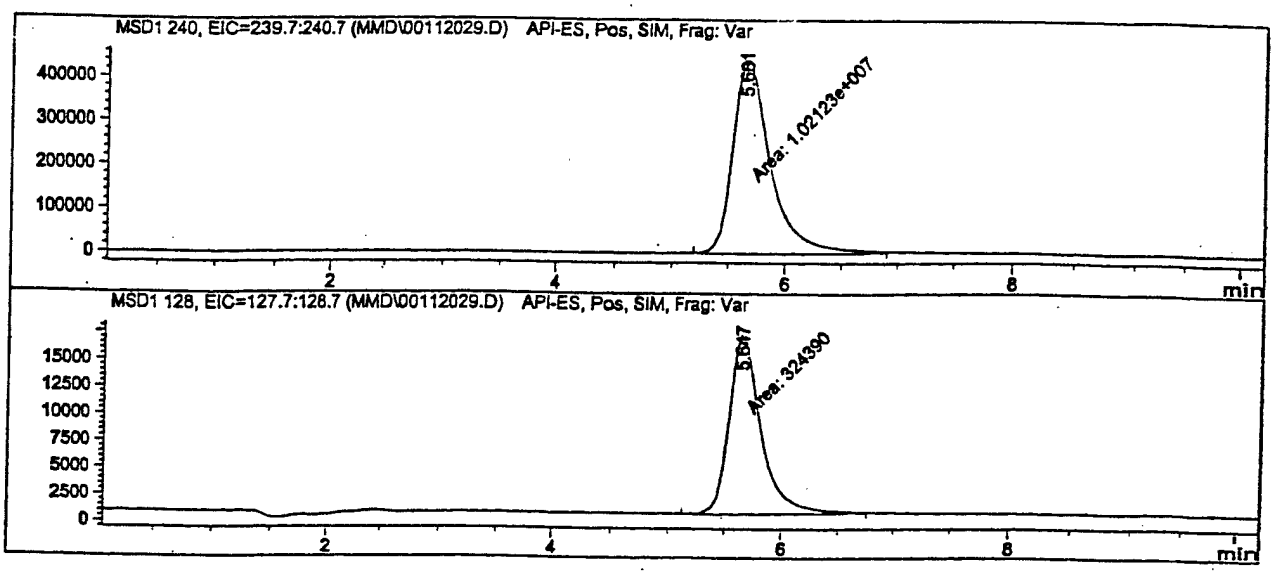
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.627	MM	0.3002	5.24223e5	2.91070e4	100.0000
Totals :				5.24223e5	2.91070e4	

Signal 2: MSD1 128, EIC=127.7:128.7

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112029.D Sample Name: 40 ppb EA-2192

Injection Date : 11/21/2000 1:54:49 AM Seq. Line : 19
Sample Name : 40 ppb EA-2192 Location : Vial 7
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl
Acq. Method : C:\HPCHEM\1\METHODS\ESPG005S.M
Last changed : 11/20/2000 1:39:21 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.661	MM	0.3769	1.02123e7	4.51612e5	100.0000
Totals :				1.02123e7	4.51612e5	

Signal 2: MSD1 128, EIC=127.7:128.7

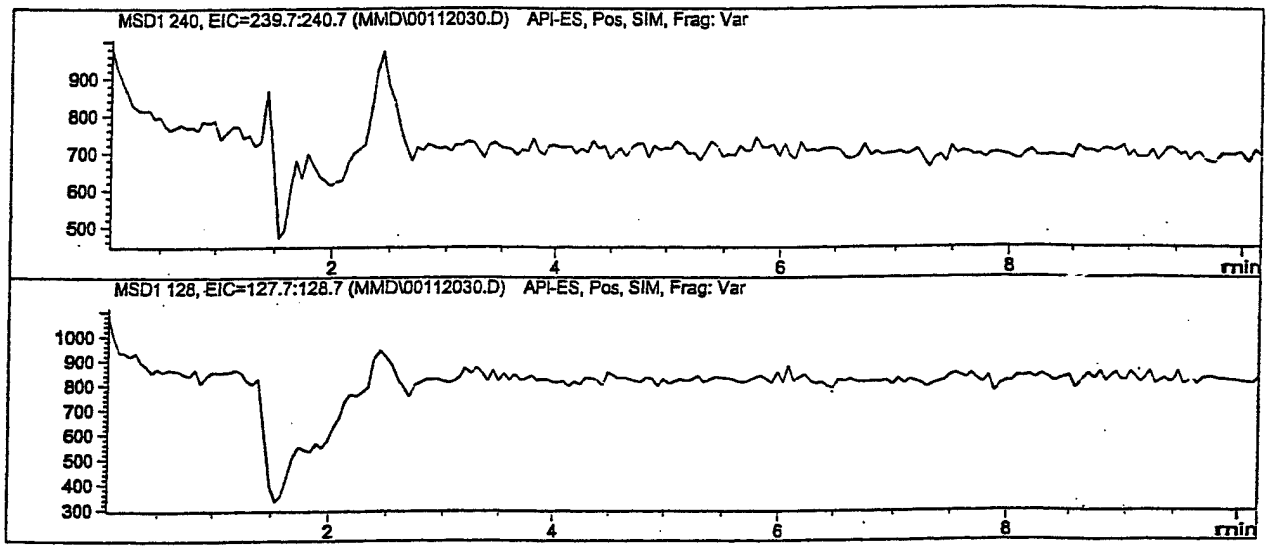
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.647	MM	0.3217	3.24390e5	1.68085e4	100.0000
Totals :				3.24390e5	1.68085e4	

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112030.D Sample Name: H20 blank

Injection Date : 11/21/2000 2:06:58 AM Seq. Line : 20
Sample Name : H20 blank Location : Vial 4
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005S.M
Last changed : 11/20/2000 1:39:21 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

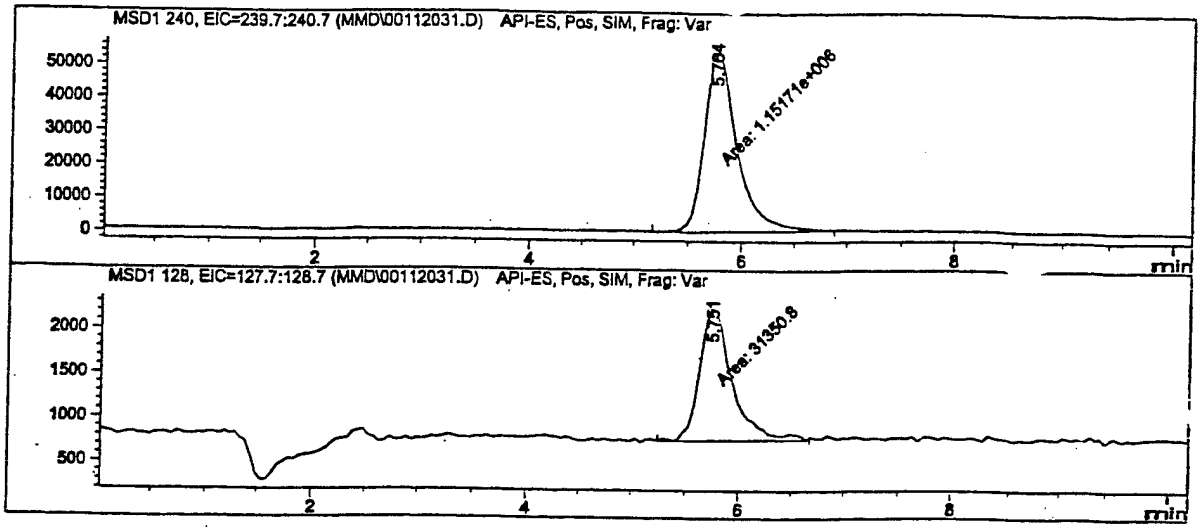
No peaks found

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112031.D Sample Name: 4 ppb EA-2192

Injection Date : 11/21/2000 2:19:06 AM Seq. Line : 21
Sample Name : 4 ppb EA-2192 Location : Vial 5
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005S.M
Last changed : 11/20/2000 1:39:21 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.764	MM	0.3480	1.15171e6	5.51647e4	100.0000
Totals :				1.15171e6	5.51647e4	

Signal 2: MSD1 128, EIC=127.7:128.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.751	MM	0.3443	3.13508e4	1517.53772	100.0000
Totals :				3.13508e4	1517.53772	

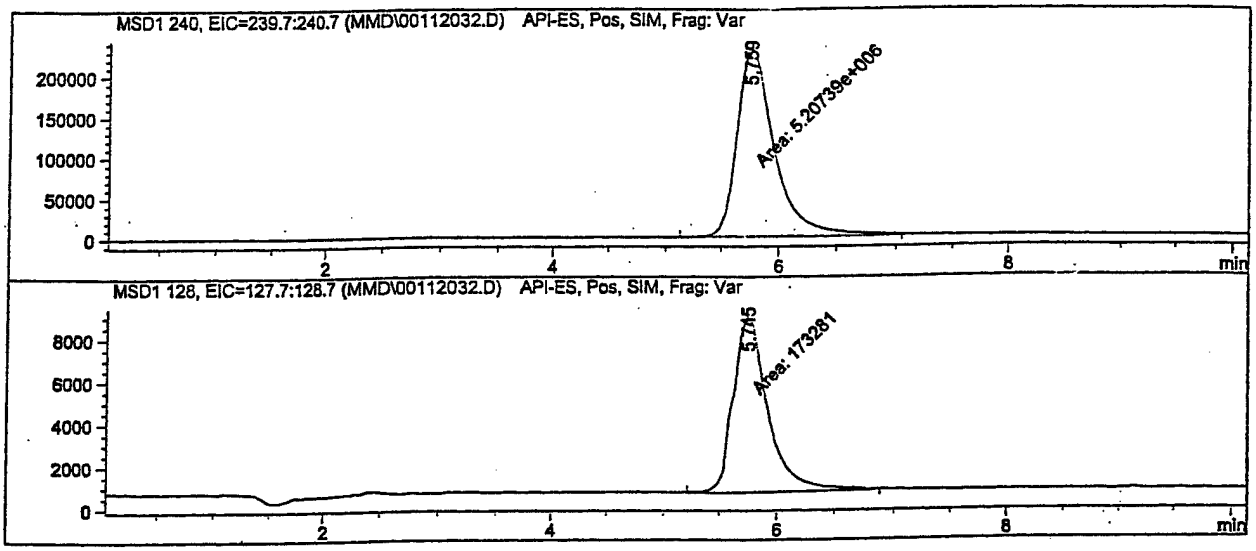
*** End of Report ***

Instrument 1 11/28/2000 9:38:09 AM wrc Page 1 of 1

Data File C:\HPCHEM\1\DATA\MMD\00112032.D Sample Name: 20 ppb EA-2192

Injection Date : 11/21/2000 2:31:14 AM Seq. Line : 22
Sample Name : 20 ppb EA-2192 Location : Vial 6
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005S.M
Last changed : 11/20/2000 1:39:21 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.759	MM	0.3659	5.20739e6	2.37175e5	100.0000
Totals :				5.20739e6	2.37175e5	

Signal 2: MSD1 128, EIC=127.7:128.7

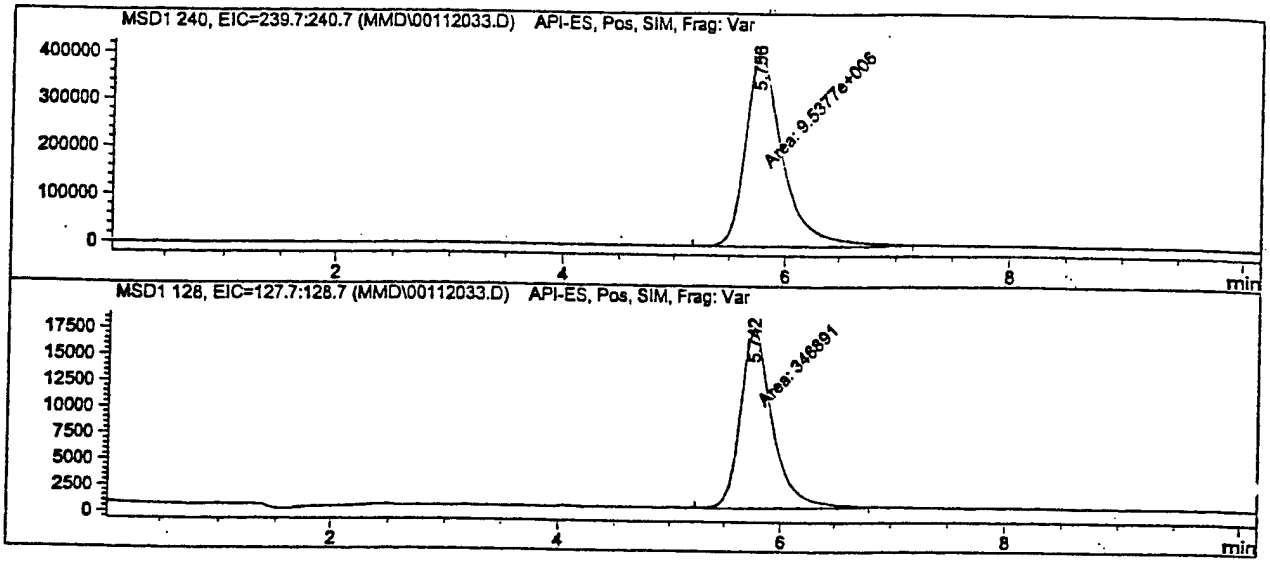
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.745	MM	0.3446	1.73281e5	8381.59570	100.0000
Totals :				1.73281e5	8381.59570	

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112033.D Sample Name: 40 ppb EA-2192

Injection Date : 11/21/2000 2:43:23 AM Seq. Line : 23
Sample Name : 40 ppb EA-2192 Location : Vial 7
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005S.M
Last changed : 11/20/2000 1:39:21 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.756	MM	0.3839	9.53769e6	4.14069e5	100.0000
Totals :				9.53770e6	4.14069e5	

Signal 2: MSD1 128, EIC=127.7:128.7

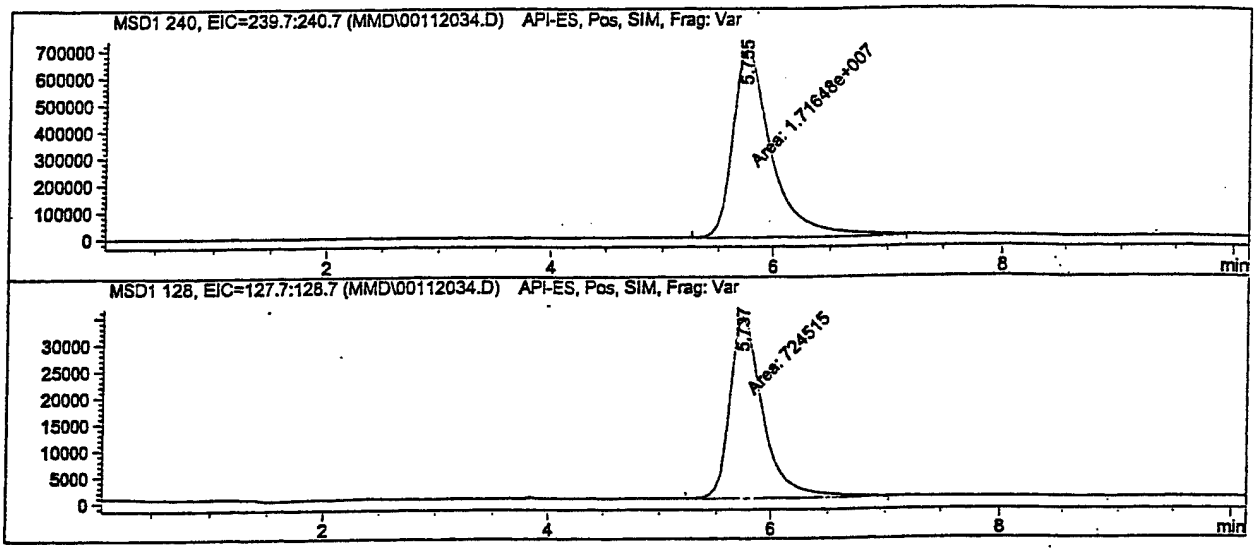
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.742	MM	0.3307	3.46891e5	1.74842e4	100.0000
Totals :				3.46891e5	1.74842e4	

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD\00112034.D Sample Name: 80 ppb EA-2192

Injection Date : 11/21/2000 2:55:33 AM Seq. Line : 24
Sample Name : 80 ppb EA-2192 Location : Vial 8
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG005S.M
Last changed : 11/20/2000 1:39:21 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG005M.M
Last changed : 11/28/2000 9:17:57 AM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.755	MM	0.3994	1.71648e7	7.16323e5	100.0000
Totals :				1.71648e7	7.16323e5	

Signal 2: MSD1 128, EIC=127.7:128.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	5.737	MM	0.3488	7.24515e5	3.46145e4	100.0000
Totals :				7.24515e5	3.46145e4	

*** End of Report ***

Instrument 1 11/28/2000 9:39:54 AM wrc

BLANK

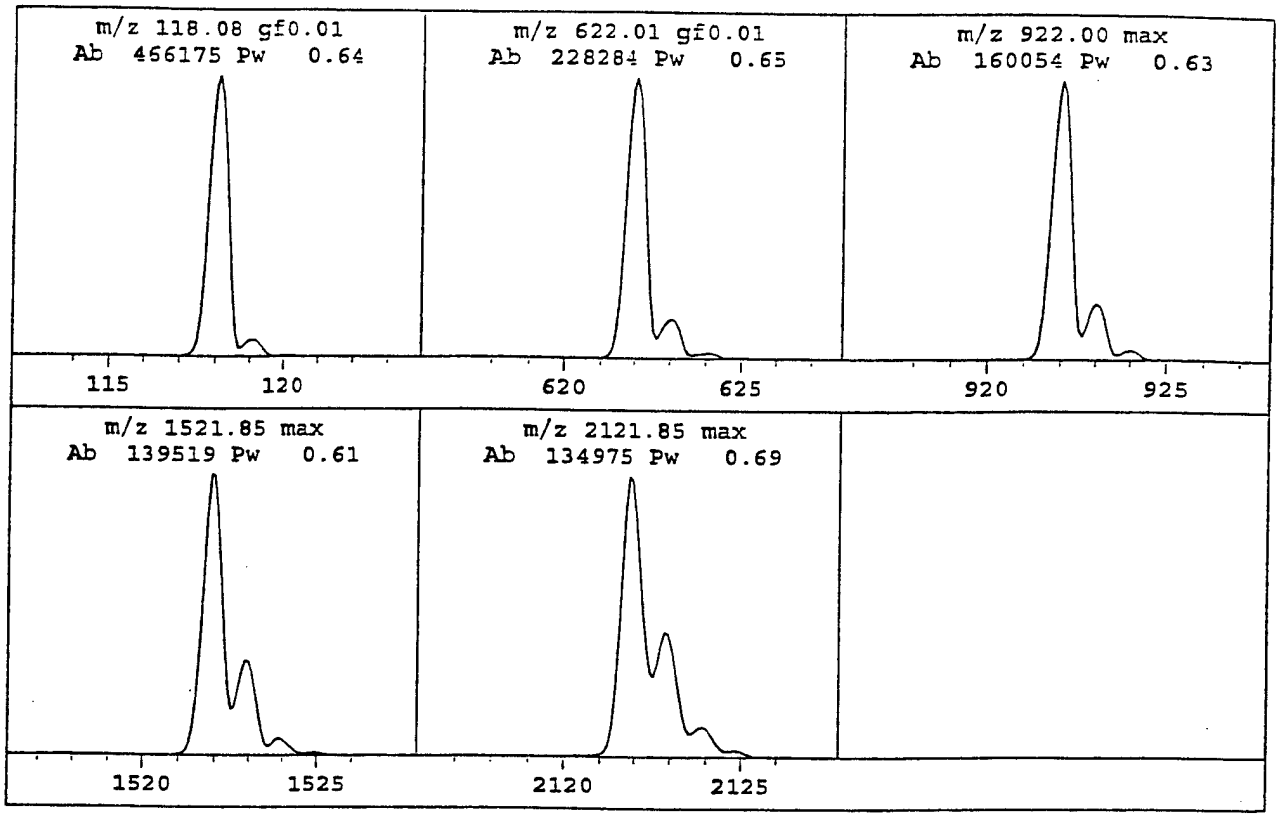
APPENDIX C

ELECTROSPRAY AUTOTUNE REPORT FOR THE LC/MSD

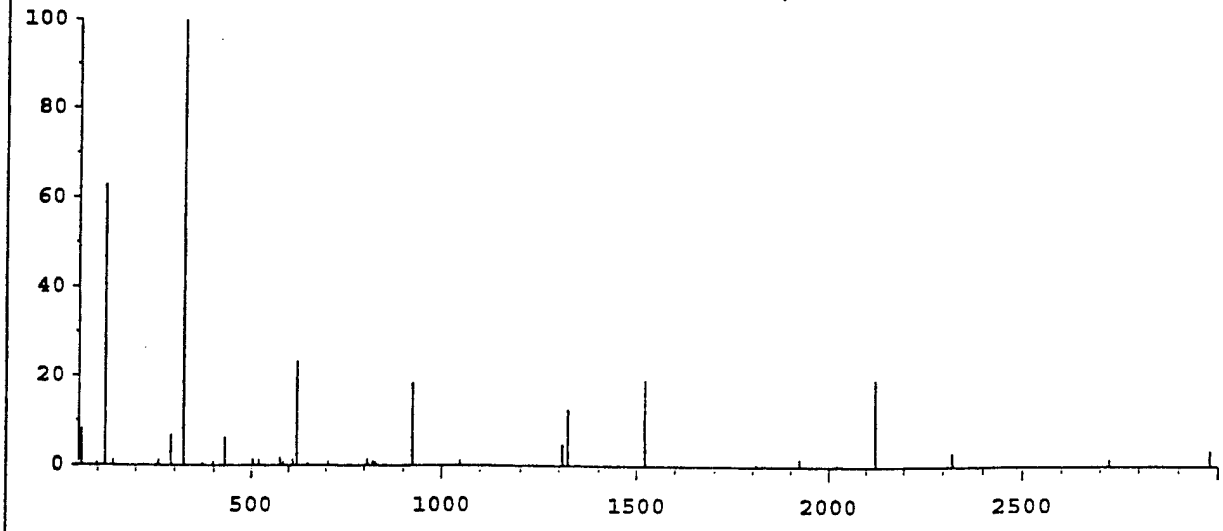
G1946: API_ES Positive mode

Instrument : Instrument 1
Mon Nov 20 10:42:36 2000

C:\HPCHEM\1\1946DTUN\ATUNES.TUN

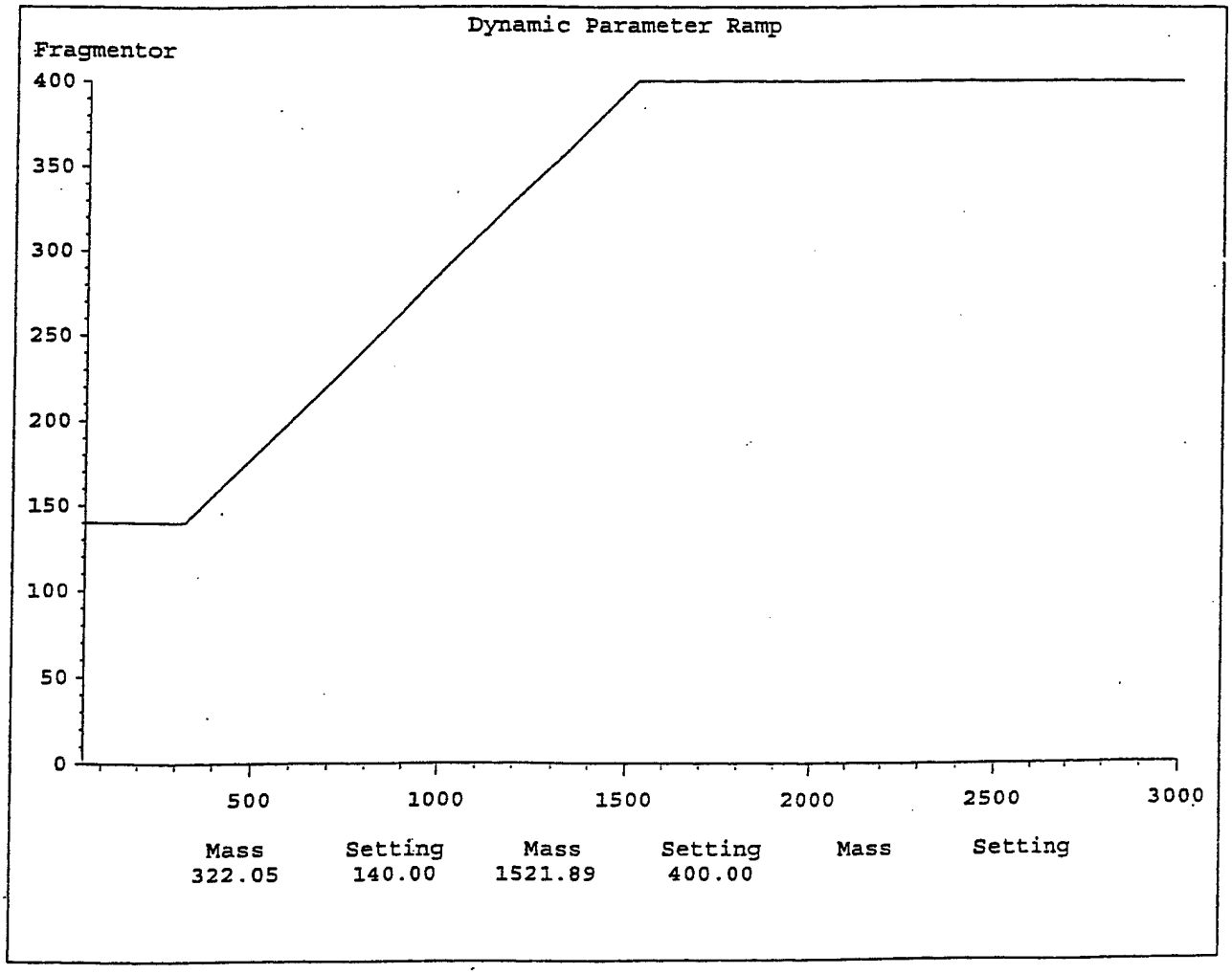


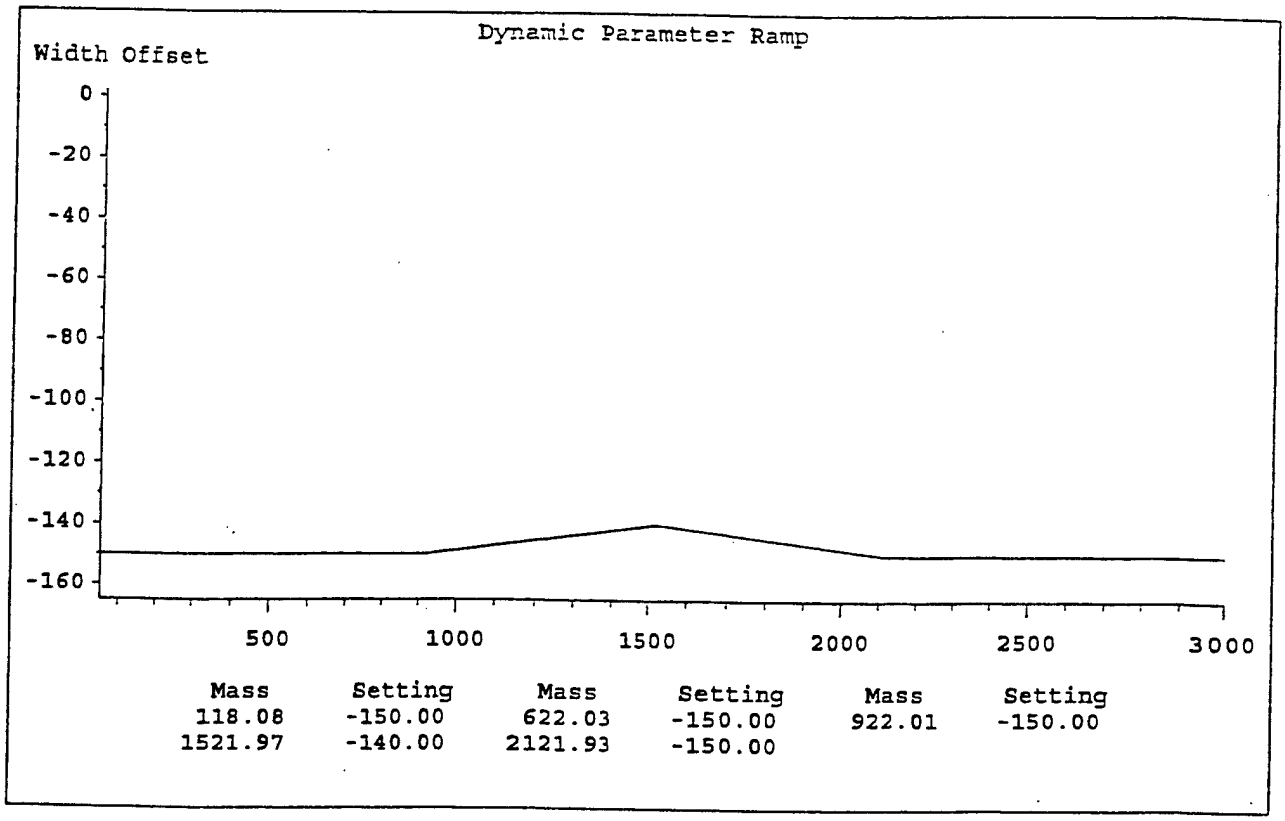
Scan: 50.00 - 3000.00 Samples: 8 Thresh: 0 Step: 0.10
29501 peaks Base: 322.10 Abundance: 754688 F Raw,Gauss 0.3



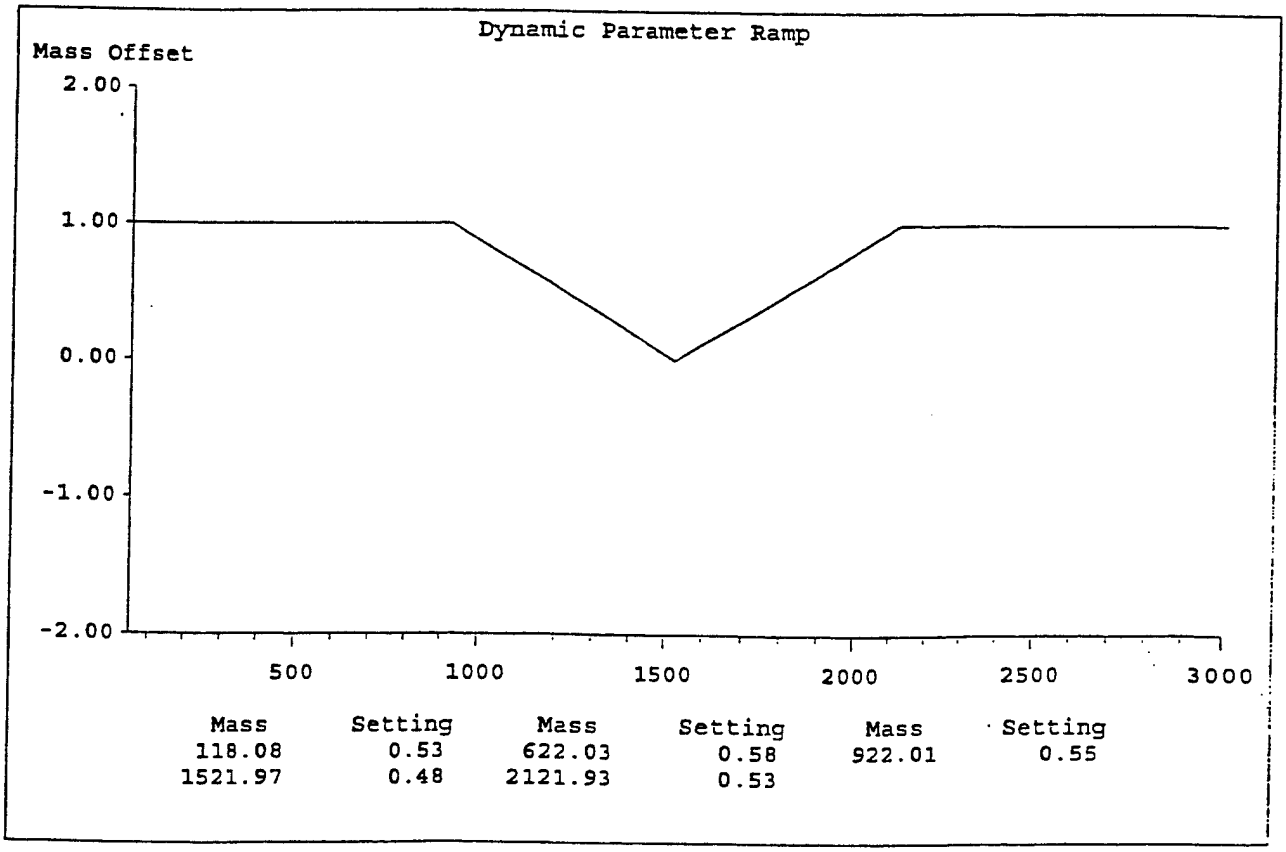
m/z gf	Abund	Rel Abund	Iso m/z	Iso Abund	Iso Ratio
118.09	475584	100.00	119.07	28200	5.93
622.02	177152	37.25	622.96	20496	11.57
922.00	140160	29.47	923.00	29264	20.88
1521.90	146496	30.80	1522.90	44160	30.14
2121.90	147264	30.96	2122.90	80600	54.73

Mode: API-ES		Polarity: POS	
Fragment	VAR	WidthGain	-251
Skim1	20	WidthOffs	VAR
Lens1	2.1	MassGain	2.95
Lens2DC	7.3	MassOffs	VAR
Iris	400		
		Energy	3.0
		OpolPeak	300
		OpolKnee	300
Gain	1.0	QuadDC	0.00
EMV	2508	Samples	8
VCap	4000	Averages	1
		StepSize	0.10
Status:			
CapCur	47	DryingGas	6.0
ChamCur	0.48	Gas Temp	300
Quad Temp	99	Neb Pres	50





Mass	Setting	Mass	Setting	Mass	Setting
118.08	-150.00	622.03	-150.00	922.01	-150.00
1521.97	-140.00	2121.93	-150.00		



Mass	Setting	Mass	Setting	Mass	Setting
118.08	0.53	622.03	0.58	922.01	0.55
1521.97	0.48	2121.93	0.53		

APPENDIX D

RAW DATA FOR EA-2192 DECONTAMINATION KINETICS FROM THE 3/26/01 RUN OF 5% VX IN DECONTAMINATION SOLUTION AT 50°C, USING LC/MS DETECTION

Tabulated data:

Standards (3/26/01)

file	conc. (ppb)	240 signal	conc. (ppm)	Calc. (linear)	Calc. (2nd order)
1032604	400	4.13E+06	0.4	738.1436507	399.388759
1032605	400	4.60E+06	0.4	806.3422792	455.8876
1032608	400	4.93E+06	0.4	854.2264226	496.906039
1032621	400	3.54E+06	0.4	652.5326064	331.660476
1032622	400	3.88E+06	0.4	701.8677845	370.255984
1032623	400	3.77E+06	0.4	685.9064033	357.639919
1032624	0	0.00E+00	0	138.8663407	0
1032625	40	4.62E+05	0.04	205.9041414	36.01789884
1032627	2000	1.39E+07	2	2155.804503	2038.1431
1032626	1000	5.29E+06	1	906.46367	542.922751

linear fit except 1 ppm point

slope	0.000145103
intercept	-138.8663407
corr coef.	0.981923758

Fit for all points, 2nd order polynomial

conc. =5.11e-15 * (signal)^2 + 7.56e-8* signal

3/26 run--EA-2192 results

file	sample	signal (240)	linear fit	poly. fit (ppm)	corr. for dil.	time (min.)	log(conc.)
1032606	NB114P73L	2.59E+05	176.4481	0.019923184	199.2318391	11.1	2.299359
1032610	NB114P73A	1.88E+07	2866.811	3.2273584	322.73584	11.1	2.508847
1032611	NB114P73B	1.24E+07	1938.149	1.7231536	172.31536	31.1	2.236324
1032612	NB114P73M	1.16E+07	1822.067	1.5645616	156.45616	31.1	2.194393
1032613	NB114P73C	5.70E+06	965.9561	0.5969439	59.69439	76	1.775934
1032614	NB114P73D	3.74E+06	681.5533	0.354220636	35.4220636	121.8333	1.549274
1032615	NB114P73E	1.23E+06	317.3436	0.100718919	10.0718919	188	1.003111
1032616	NB114P73N	1.64E+06	376.836	0.137727856	13.7727856	188	1.139022
1032617	NB114P73F	7.00E+05	240.4388	0.0554239	5.54239	249.8	0.743697
1032618	NB114P73G	5.10E+05	212.8691	0.039885111	3.9885111	311.6	0.600811
1032619	NB114P73H	1.82E+05	165.2752	0.013928464	1.392846364	369.1	0.143903
1032620	NB114P73P	2.03E+05	168.3223	0.015557378	1.555737799	370.3	0.191936

3/26 run--VX results using the same calibration curve

file	sample	signal (268)	linear fit	poly. fit (ppm)	corr. for dil.	time (min.)	log(conc.)
1032606	NB114P73L	1.67E+05	163.0986	0.012767713	127.6771279	11.1	2.106113
1032610	NB114P73A	1.46E+07	2257.377	2.1930076	219.30076	11.1	2.34104
1032611	NB114P73B	2.61E+06	517.5864	0.232125831	23.2125831	31.1	1.365723
1032612	NB114P73M	7.03E+05	240.8741	0.055672208	5.567220799	31.1	0.745638
1032613	NB114P73C	9.70E+04	152.9414	0.00738128	0.738127999	76	-0.13187
1032614	NB114P73D	6.40E+04	148.153	0.004859331	0.485933056	121.8333	-0.31342
1032615	NB114P73E	4.30E+04	145.1058	0.003260248	0.326024839	188	-0.48675
1032616	NB114P73N	5.70E+04	147.1372	0.004325802	0.432580239	188	-0.36393
1032617	NB114P73F	5.00E+04	146.1215	0.003792775	0.3792775	249.8	-0.42104
1032618	NB114P73G	3.70E+04	144.2352	0.002804196	0.280419559	311.6	-0.55219
1032619	NB114P73H	4.40E+04	145.2509	0.003336293	0.333629296	369.1	-0.47674
1032620	NB114P73P	4.10E+04	144.8156	0.00310819	0.310818991	370.3	-0.50749

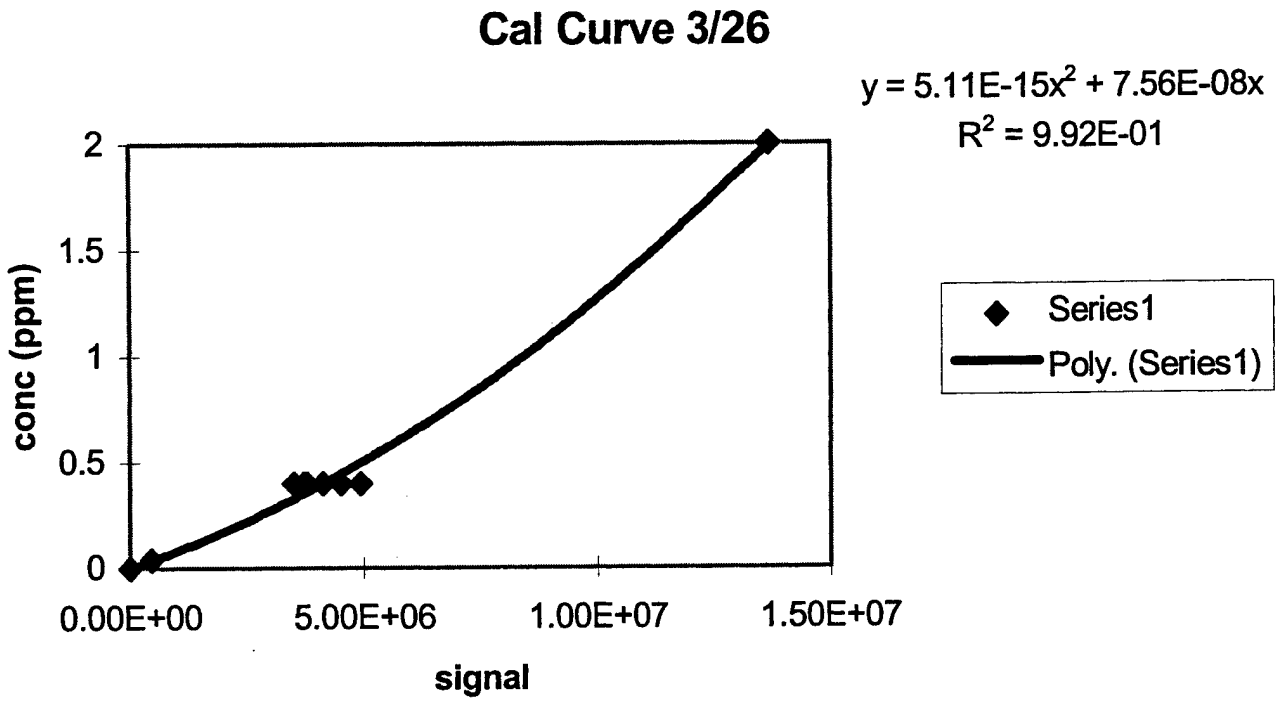
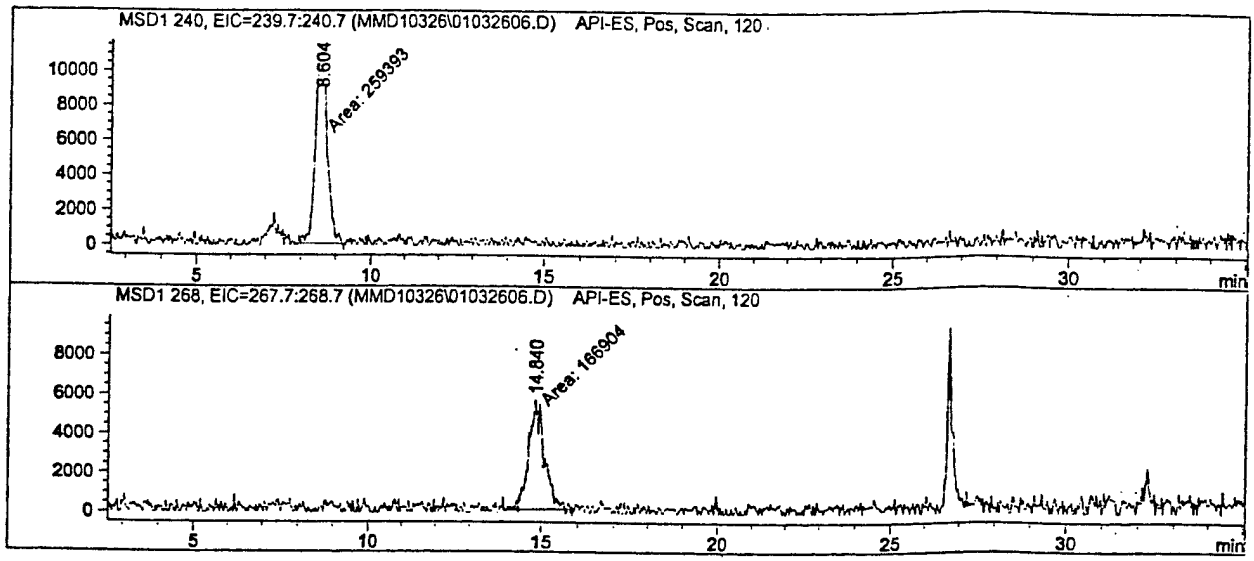


Figure D-1. Calibration Curve for EA-2192 using LC/MS, Using a Polynomial Fit with Forced Zero Intercept. The point for the 1 ppm standard was excluded because it was significantly off of the calibration curve.

Data File C:\HPCHEM\1\DATA\MMD10326\01032606.D Sample Name: NB114P73L

```
Injection Date  : 3/26/2001 3:27:18 PM          Seq. Line :   6
Sample Name     : NB114P73L                     Location : Vial 11
Acq. Operator   : wrc                              Inj :   1
                                                Inj Volume : 25 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 1/25/2001 10:15:20 AM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 7/2/2001 2:28:06 PM by wrc
                  (modified after loading)
+ESI gradient method, reversed phase
```



Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
```

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	8.604	MM	0.3833	2.59393e5	1.12787e4	100.0000
Totals :				2.59393e5	1.12787e4	

Signal 2: MSD1 268, EIC=267.7:268.7

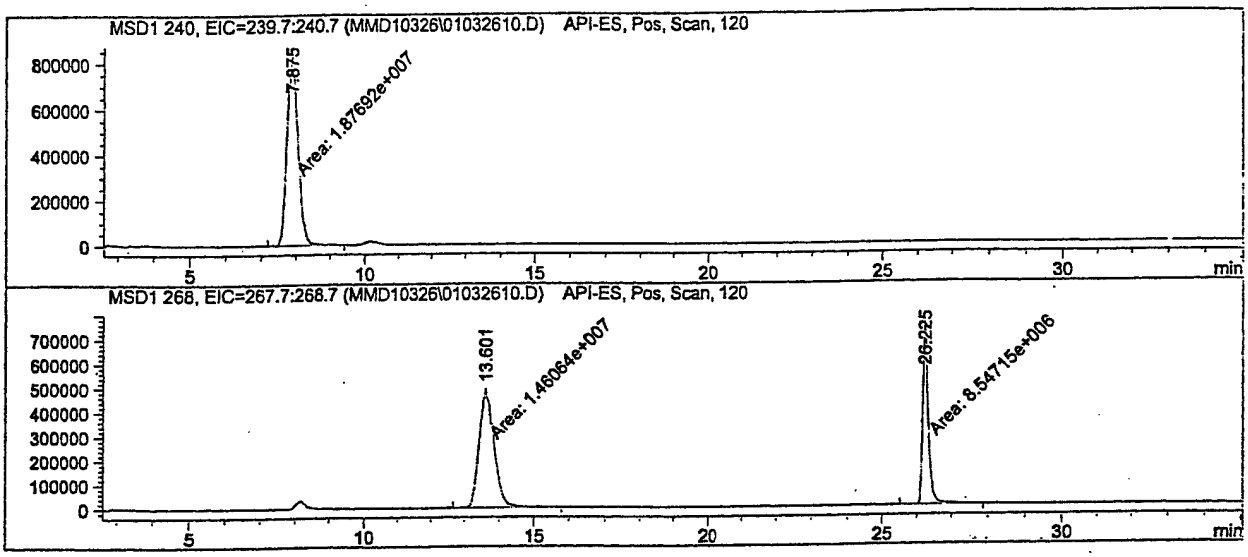
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	14.840	MM	0.4912	1.66904e5	5663.35010	100.0000
Totals :				1.66904e5	5663.35010	

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD10326\01032610.D Sample Name: NB114P73A

Injection Date	: 3/26/2001 4:39:40 PM	Seq. Line	: 1
Sample Name	: NB114P73A	Location	: Vial 12
Acq. Operator	: wrc	Inj	: 1
		Inj Volume	: 25 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ESPG012.M		
Last changed	: 1/25/2001 10:15:20 AM by wrc		
Analysis Method	: C:\HPCHEM\1\METHODS\ESPG012.M		
Last changed .	: 7/2/2001 2:28:06 PM by wrc (modified after loading)		

+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	7.875	MM	0.3739	1.87692e7	8.36549e5	100.0000
Totals :				1.87692e7	8.36549e5	

Signal 2: MSD1 268, EIC=267.7:268.7

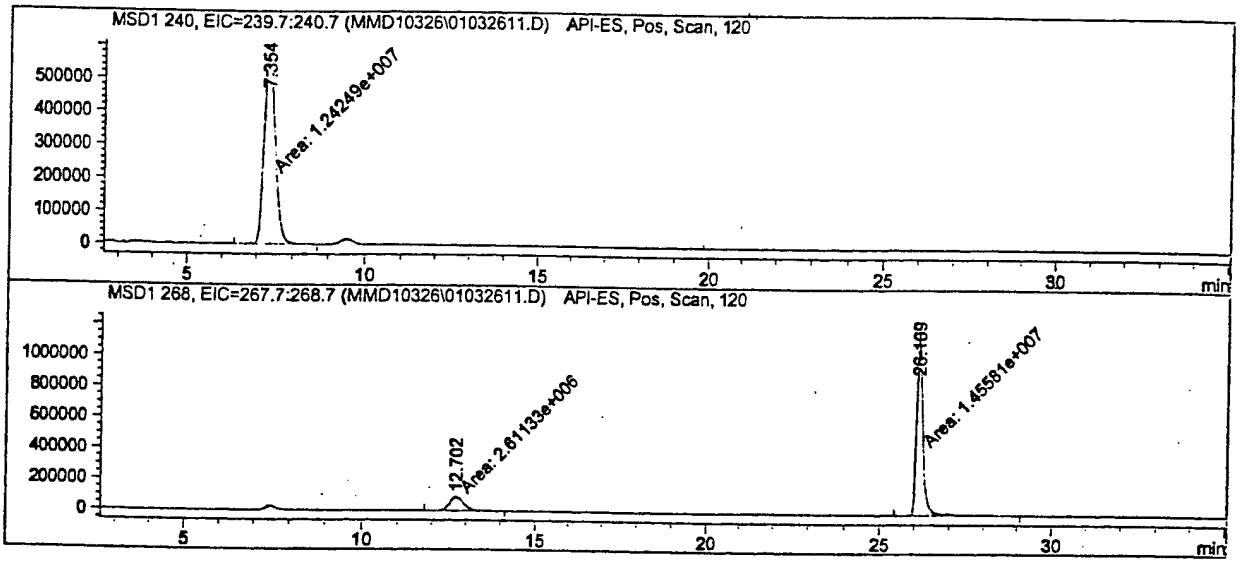
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	13.601	MM	0.4877	1.46064e7	4.99119e5	63.0849
2	26.225	MM	0.1826	8.54715e6	7.80281e5	36.9151
Totals :				2.31535e7	1.27940e6	

Instrument 1 7/2/2001 2:30:08 PM wrc

Data File C:\HPCHEM\1\DATA\MMD10326\01032611.D Sample Name: NB114P73B

Injection Date	: 3/26/2001 5:31:27 PM	Seq. Line	: 2
Sample Name	: NB114P73B	Location	: Vial 13
Acq. Operator	: wrc	Inj	: 1
		Inj Volume	: 25 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ESPG012.M		
Last changed	: 1/25/2001 10:15:20 AM by wrc		
Analysis Method	: C:\HPCHEM\1\METHODS\ESPG012.M		
Last changed	: 7/2/2001 2:28:06 PM by wrc (modified after loading)		

+ESI gradient method, reversed phase



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

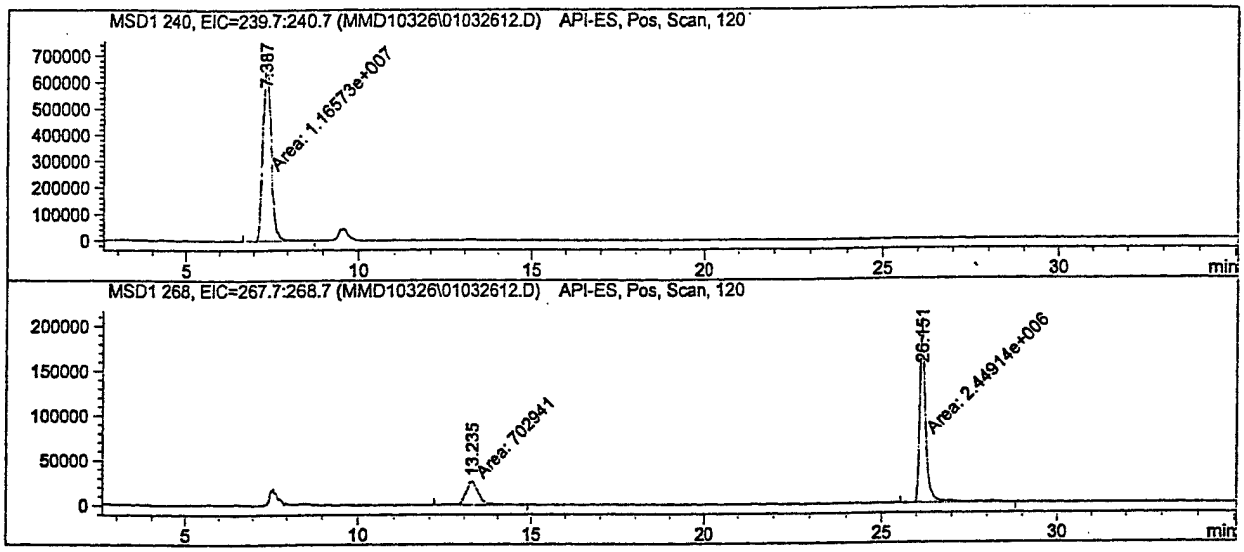
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	7.354	MM	0.3531	1.24249e7	5.86465e5	100.0000
Totals :				1.24249e7	5.86465e5	

Signal 2: MSD1 268, EIC=267.7:268.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	12.702	MM	0.4652	2.61133e6	9.35620e4	15.2091
2	26.109	MM	0.1988	1.45581e7	1.22031e6	84.7909
Totals :				1.71694e7	1.31387e6	

Data File C:\HPCHEM\1\DATA\MMD10326\01032612.D Sample Name: NB114P73M

```
Injection Date  : 3/26/2001 6:23:15 PM          Seq. Line :   3
Sample Name     : NB114P73M                     Location : Vial 14
Acq. Operator   : wrc                              Inj :   1
                                                Inj Volume : 25 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 1/25/2001 10:15:20 AM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 7/2/2001 2:28:06 PM by wrc
                  (modified after loading)
+ESI gradient method, reversed phase
```



Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
```

Signal 1: MSD1 240, EIC=239.7:240.7

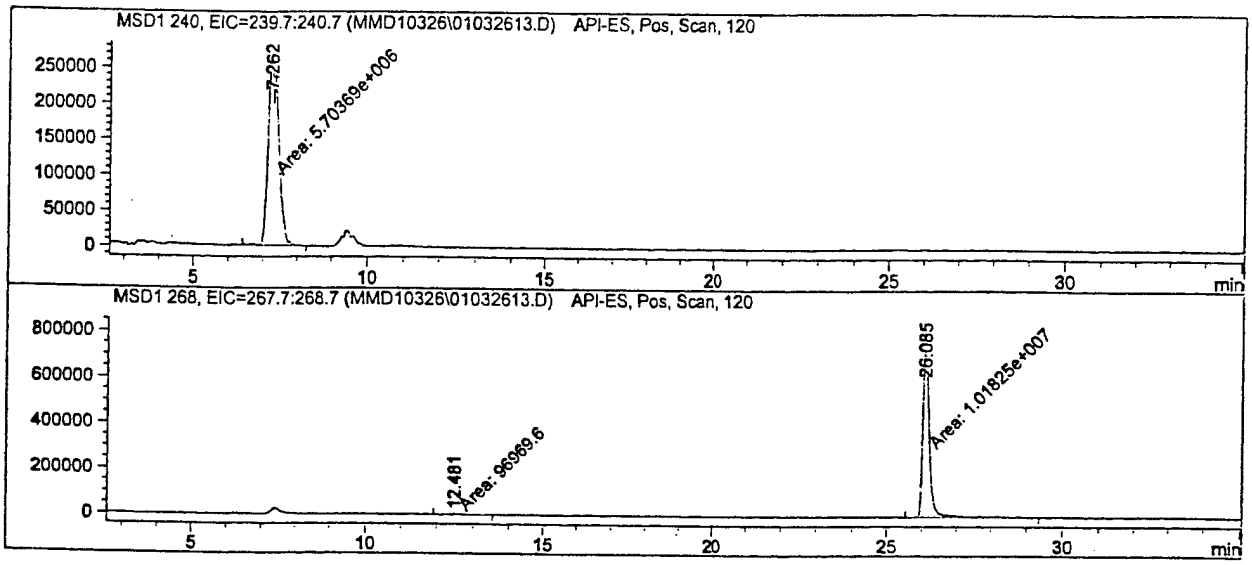
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	7.387	MM	0.2672	1.16573e7	7.27265e5	100.0000
Totals :				1.16573e7	7.27265e5	

Signal 2: MSD1 268, EIC=267.7:268.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	13.235	MM	0.4485	7.02941e5	2.61225e4	22.3008
2	26.151	MM	0.1953	2.44914e6	2.09043e5	77.6992
Totals :				3.15209e6	2.35166e5	

Data File C:\HPCHEM\1\DATA\MMD10326\01032613.D Sample Name: NB114P73C

```
Injection Date  : 3/26/2001 7:15:03 PM          Seq. Line :  4
Sample Name     : NB114P73C                    Location : Vial 15
Acq. Operator   : wrc                             Inj :  1
                                               Inj Volume : 25 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 1/25/2001 10:15:20 AM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 7/2/2001 2:28:06 PM by wrc
                  (modified after loading)
+ESI gradient method, reversed phase
```



Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
```

Signal 1: MSD1 240, EIC=239.7:240.7

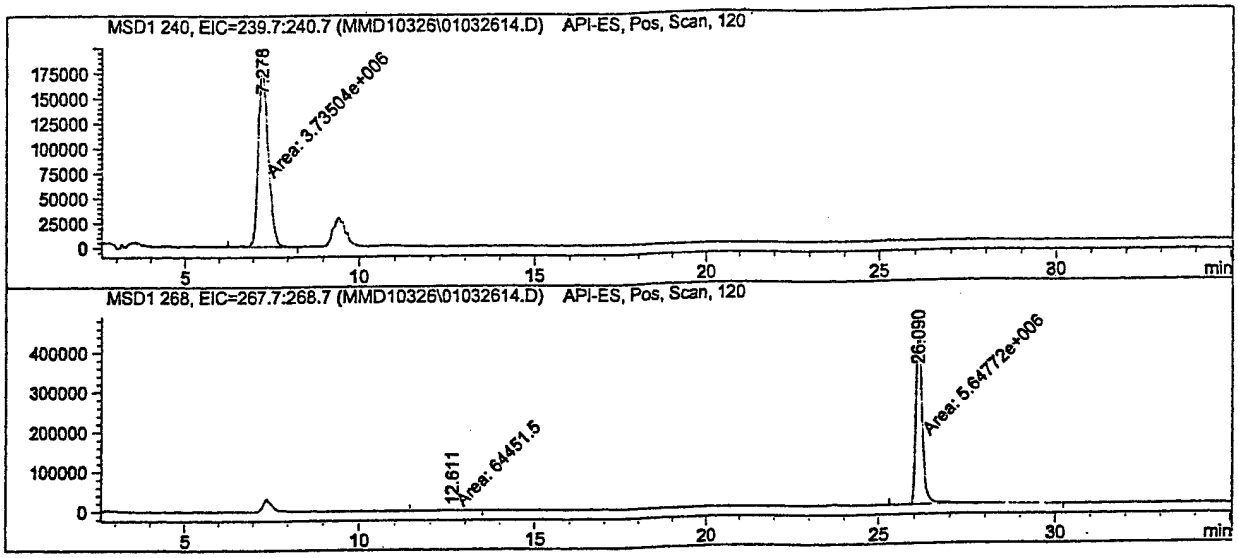
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	7.262	MM	0.3397	5.70369e6	2.79872e5	100.0000
Totals :				5.70369e6	2.79872e5	

Signal 2: MSD1 268, EIC=267.7:268.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	12.481	MM	0.4905	9.69696e4	3294.71582	0.9433
2	26.085	MM	0.2041	1.01825e7	8.31673e5	99.0567
Totals :				1.02795e7	8.34968e5	

Data File C:\HPCHEM\1\DATA\MMD10326\01032614.D Sample Name: NB114P73D

```
Injection Date  : 3/26/2001 8:06:50 PM          Seq. Line :   5
Sample Name     : NB114P73D                     Location : Vial 16
Acq. Operator   : wrc                              Inj :   1
                                                Inj Volume : 25 µl

Acq. Method     : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 1/25/2001 10:15:20 AM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 7/2/2001 2:28:06 PM by wrc
                  (modified after loading)
+ESI gradient method, reversed phase
```



Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
```

Signal 1: MSD1 240, EIC=239.7:240.7

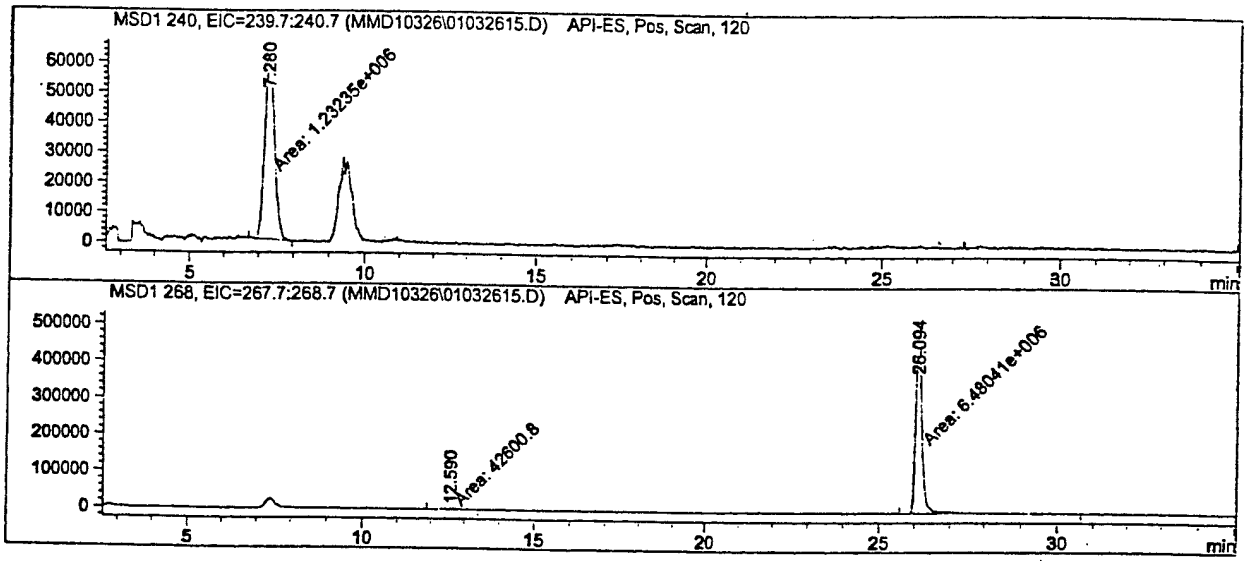
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	7.278	MM	0.3239	3.73504e6	1.92185e5	100.0000
Totals :				3.73504e6	1.92185e5	

Signal 2: MSD1 268, EIC=267.7:268.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	12.611	MM	0.4728	6.44515e4	2272.05347	1.1283
2	26.090	MM	0.1927	5.64772e6	4.88535e5	98.8717
Totals :				5.71218e6	4.90807e5	

Data File C:\HPCHEM\1\DATA\MMD10326\01032615.D Sample Name: NB114P73E

```
Injection Date  : 3/26/2001 8:58:38 PM          Seq. Line :   6
Sample Name     : NB114P73E                     Location : Vial 17
Acq. Operator   : wrc                              Inj :   1
                                                Inj Volume : 25 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 1/25/2001 10:15:20 AM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 7/2/2001 2:28:06 PM by wrc
                  (modified after loading)
+ESI gradient method, reversed phase
```



Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
```

Signal 1: MSD1 240, EIC=239.7:240.7

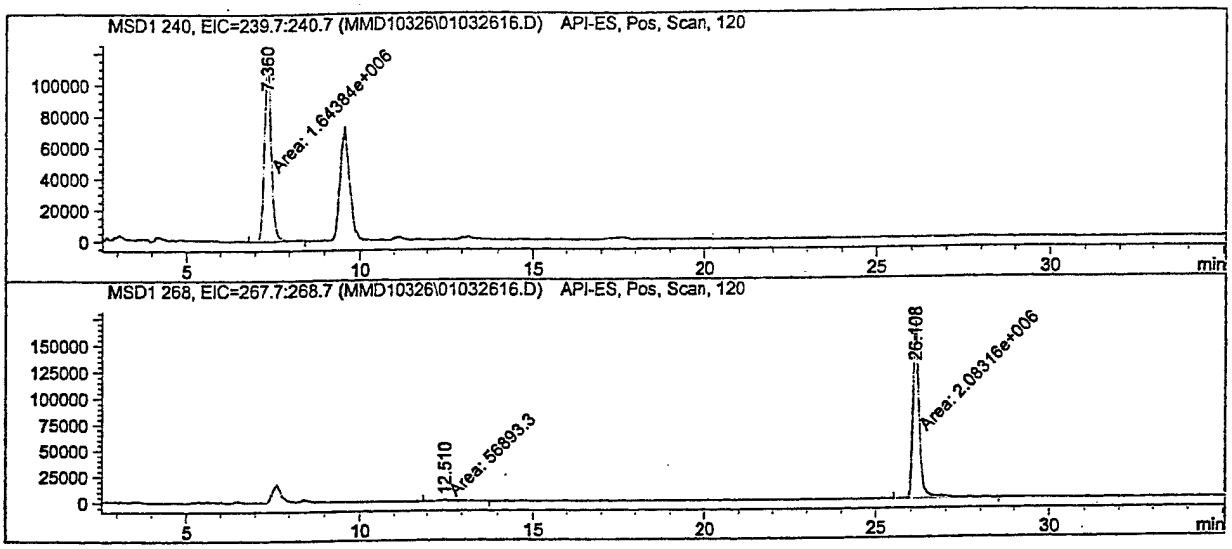
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	7.280	MM	0.3254	1.23235e6	6.31183e4	100.0000
Totals :				1.23235e6	6.31183e4	

Signal 2: MSD1 268, EIC=267.7:268.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	12.590	MM	0.5054	4.26008e4	1404.81860	0.6531
2	26.094	MM	0.2088	6.48041e6	5.17210e5	99.3469
Totals :				6.52301e6	5.18615e5	

Data File C:\HPCHEM\1\DATA\MMD10326\01032616.D Sample Name: NB114P73N

```
==========================================================================
Injection Date  : 3/26/2001 9:50:27 PM          Seq. Line :   7
Sample Name     : NB114P73N                      Location : Vial 18
Acq. Operator   : wrc                               Inj :   1
                                                Inj Volume : 25 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 1/25/2001 10:15:20 AM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 7/2/2001 2:28:06 PM by wrc
                  (modified after loading)
+ESI gradient method, reversed phase
==========================================================================
```



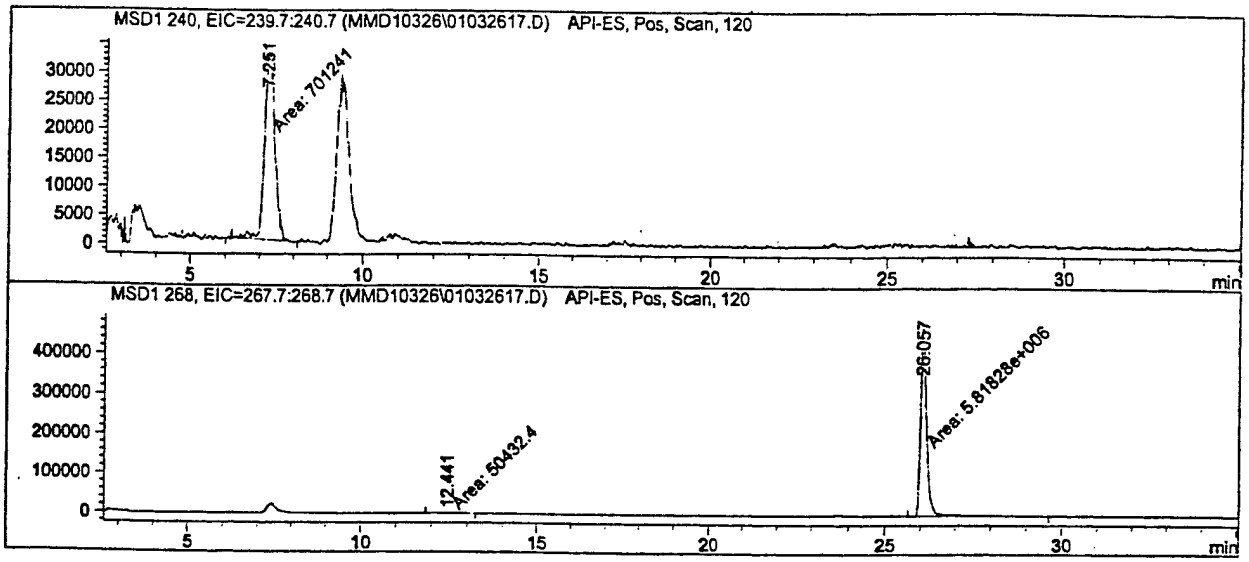

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]                          %
----|-------|----|-------|----------|----------|--------|
   1   7.360 MM    0.2233 1.64384e6  1.22668e5  100.0000

Totals :                  1.64384e6  1.22668e5


Signal 2: MSD1 268, EIC=267.7:268.7

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]                          %
----|-------|----|-------|----------|----------|--------|
   1  12.510 MM    0.5801 5.68932e4  1634.66650   2.6585
   2  26.108 MM    0.1974 2.08316e6  1.75910e5   97.3415

Totals :                  2.14005e6  1.77545e5

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

Instrument 1 7/2/2001 2:43:13 PM wrc

Data File C:\HPCHEM\1\DATA\MMD10326\01032617.D Sample Name: NB114P73F

Injection Date : 3/26/2001 10:42:17 PM Seq. Line : 8
Sample Name : NB114P73F Location : Vial 19
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed : 1/25/2001 10:15:20 AM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed : 7/2/2001 2:28:06 PM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

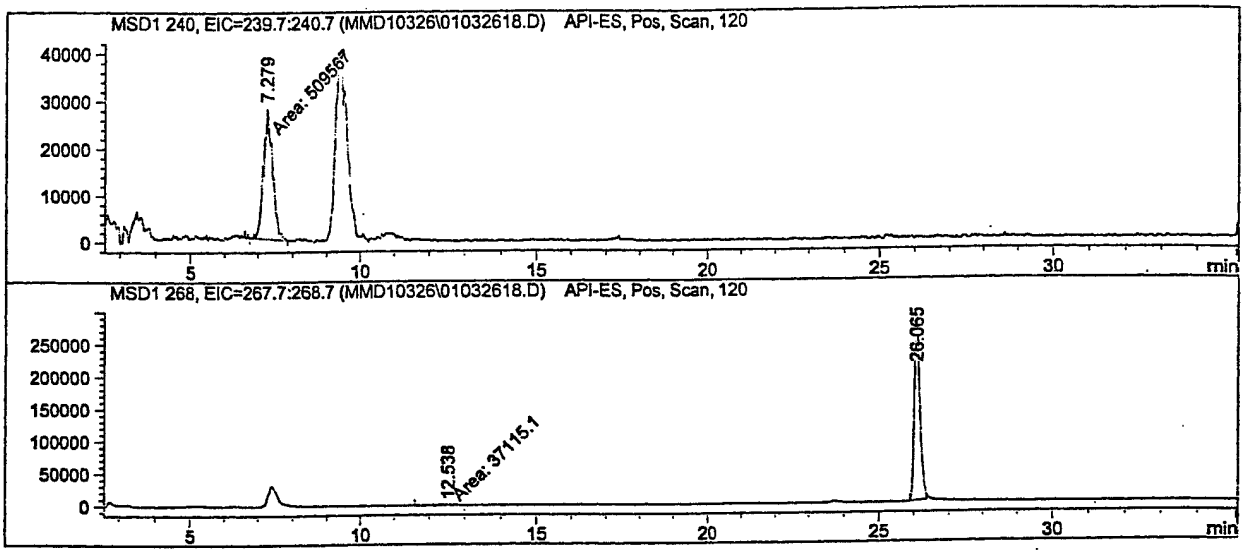
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	7.251	MM	0.3479	7.01241e5	3.35955e4	100.0000
Totals :				7.01241e5	3.35955e4	

Signal 2: MSD1 268, EIC=267.7:268.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	12.441	MM	0.4797	5.04324e4	1752.39929	0.8593
2	26.057	MM	0.1952	5.81828e6	4.96762e5	99.1407
Totals :				5.86872e6	4.98514e5	

Data File C:\HPCHEM\1\DATA\MMD10326\01032618.D Sample Name: NB114P73G

```
Injection Date  : 3/26/2001 11:34:06 PM        Seq. Line :   9
Sample Name     : NB114P73G                    Location : Vial 20
Acq. Operator   : wrc                             Inj :   1
                                               Inj Volume : 25 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 1/25/2001 10:15:20 AM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 7/2/2001 2:28:06 PM by wrc
                  (modified after loading)
+ESI gradient method, reversed phase
```



Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
```

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	7.279	MM	0.3010	5.09567e5	2.82158e4	100.0000
Totals :				5.09567e5	2.82158e4	

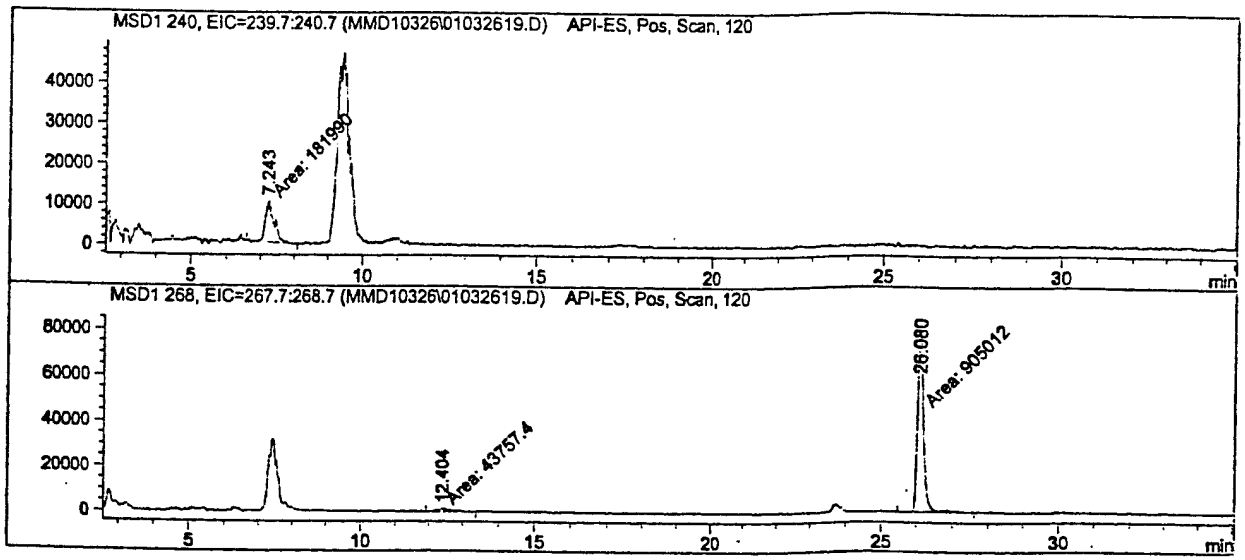
Signal 2: MSD1 268, EIC=267.7:268.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	12.538	MM	0.4234	3.71151e4	1461.04126	1.1833
2	26.065	BB	0.1578	3.09945e6	2.86916e5	98.8167
Totals :				3.13656e6	2.88377e5	

Instrument 1 7/2/2001 2:46:51 PM wrc

Data File C:\HPCHEM\1\DATA\MMD10326\01032619.D Sample Name: NB114P73H

```
Injection Date  : 3/27/2001 12:25:55 AM        Seq. Line :  10
Sample Name     : NB114P73H                    Location : Vial 21
Acq. Operator   : wrc                             Inj :   1
                                               Inj Volume : 25 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 1/25/2001 10:15:20 AM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 7/2/2001 2:28:06 PM by wrc
                  (modified after loading)
+ESI gradient method, reversed phase
```



Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
```

Signal 1: MSD1 240, EIC=239.7:240.7

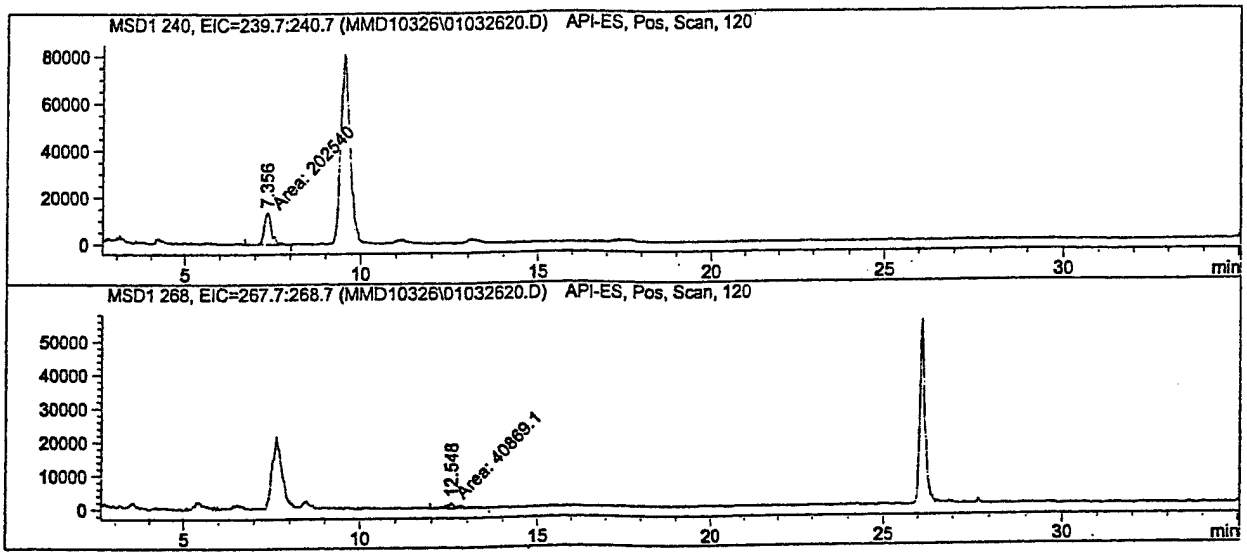
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	7.243	MM	0.2931	1.81990e5	1.03480e4	100.0000
Totals :				1.81990e5	1.03480e4	

Signal 2: MSD1 268, EIC=267.7:268.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	12.404	MM	0.4069	4.37574e4	1792.25208	4.6120
2	26.080	MM	0.1835	9.05012e5	8.22203e4	95.3880
Totals :				9.48769e5	8.40126e4	

Data File C:\HPCHEM\1\DATA\MMD10326\01032620.D Sample Name: NB114P73P

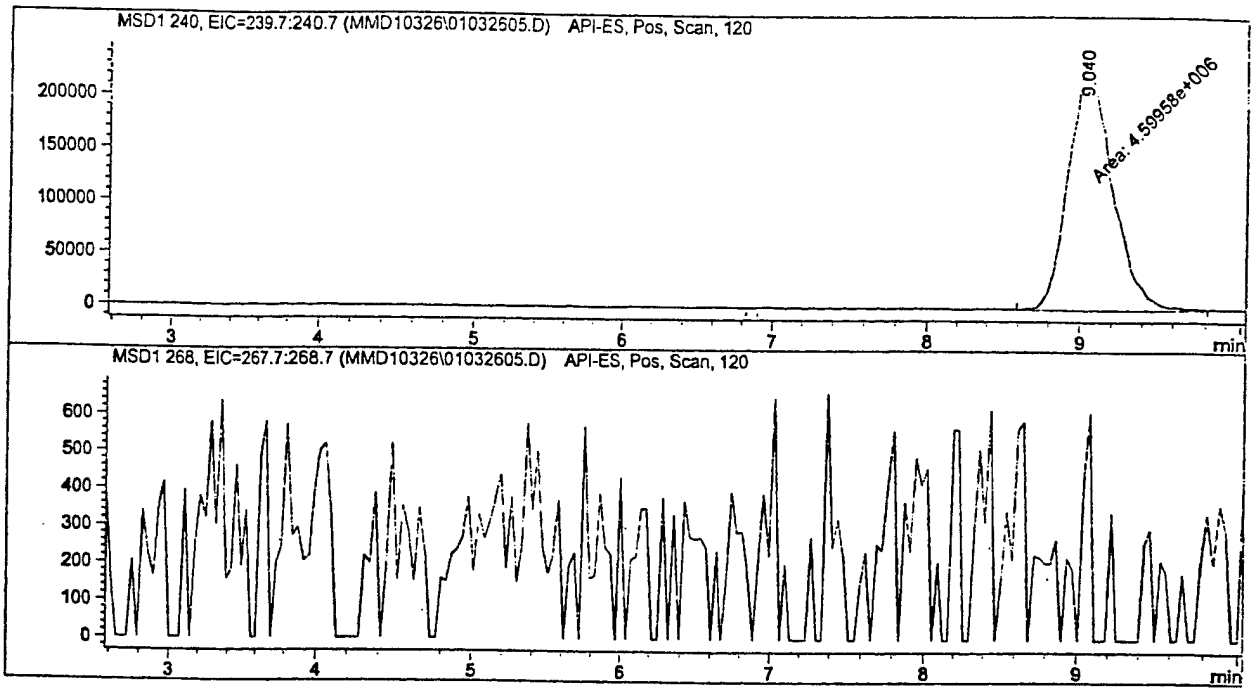
```
==========================================================================
Injection Date  : 3/27/2001 1:17:42 AM          Seq. Line :  11
Sample Name     : NB114P73P                     Location : Vial 22
Acq. Operator   : wrc                               Inj :   1
                                               Inj Volume : 25 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 1/25/2001 10:15:20 AM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed    : 7/2/2001 2:28:06 PM by wrc
                  (modified after loading)
+ESI gradient method, reversed phase
==========================================================================
```




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

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]                          %
----|-------|----|-------|----------|----------|--------|
   1  7.356 MM    0.2462 2.02540e5  1.37097e4  100.0000

Totals :                  2.02540e5  1.37097e4


Signal 2: MSD1 268, EIC=267.7:268.7

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]                          %
----|-------|----|-------|----------|----------|--------|
   1 12.548 MM    0.3531 4.08691e4  1928.84705 100.0000

Totals :                  4.08691e4  1928.84705

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

Instrument 1 7/2/2001 2:48:48 PM wrc

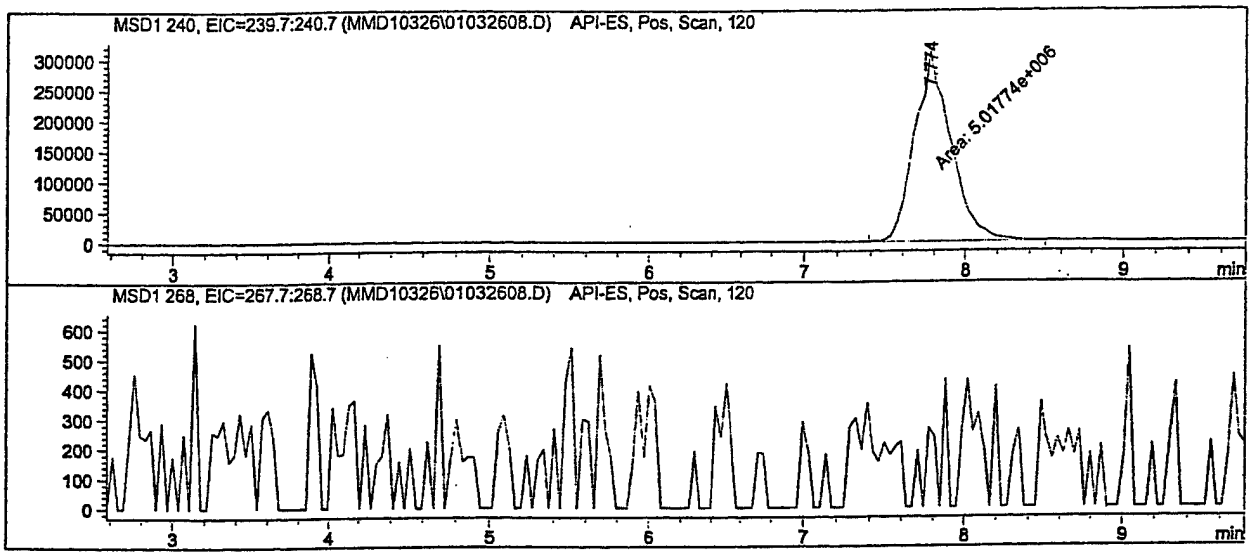
Data File C:\HPCHEM\1\DATA\MMD10326\01032605.D Sample Name: 400 ppb 2192



*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD10326\01032608.D Sample Name: 400 ppb 2192

```
Injection Date   : 3/26/2001 4:19:33 PM          Seq. Line :   7
Sample Name      : 400 ppb 2192                  Location : Vial 1
Acq. Operator    : wrc                               Inj :   1
                                                 Inj Volume : 25 µl
Acq. Method      : C:\HPCHEM\1\METHODS\ESPG011.M
Last changed     : 1/16/2001 12:28:53 PM by wrc
Analysis Method  : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed     : 7/2/2001 2:28:06 PM by wrc
                   (modified after loading)
+ESI gradient method, reversed phase
```



Area Percent Report

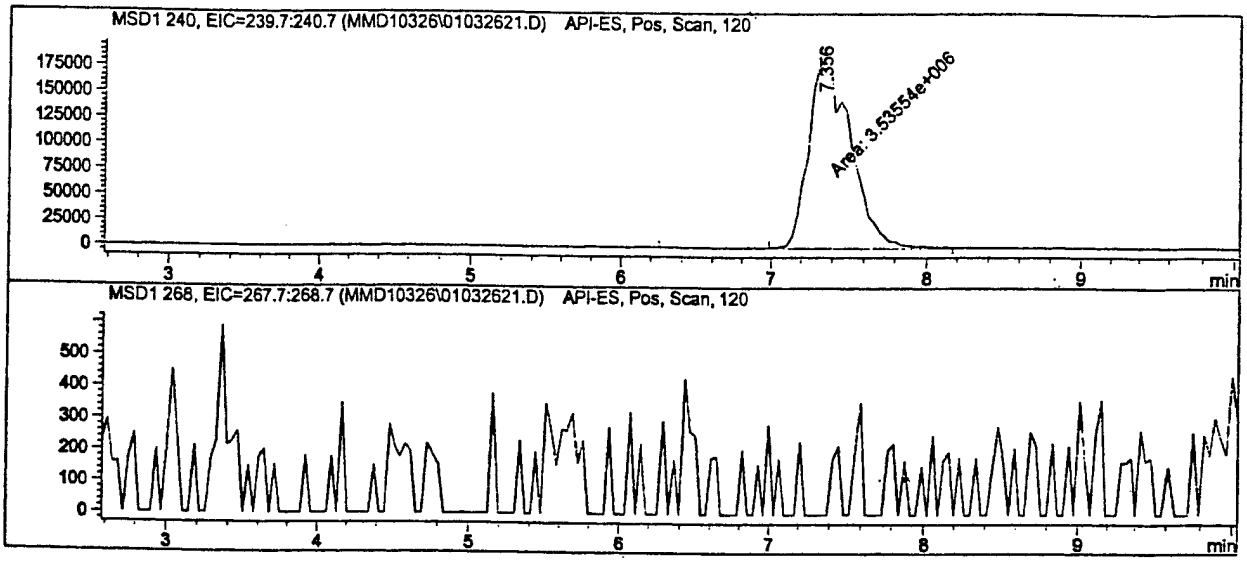
```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]                          %
----|-------|----|-------|----------|----------|--------|
   1   7.774 MM    0.2620 5.01774e6  3.19197e5  100.0000

Totals :                  5.01774e6  3.19197e5

Signal 2: MSD1 268, EIC=267.7:268.7
```

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD10326\01032621.D Sample Name: 400 ppb 2192

Injection Date : 3/27/2001 2:10:10 AM Seq. Line : 12
Sample Name : 400 ppb 2192 Location : Vial 1
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG011.M
Last changed : 1/16/2001 12:28:53 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed : 7/2/2001 2:28:06 PM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

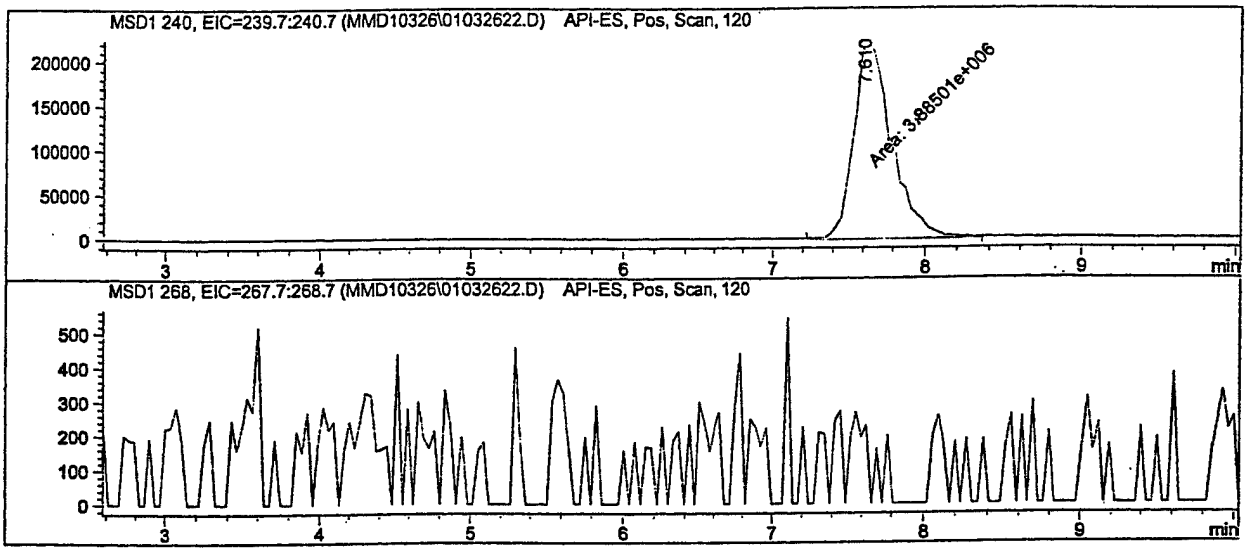
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	7.356	MM	0.3100	3.53554e6	1.90111e5	100.0000
Totals :				3.53554e6	1.90111e5	

Signal 2: MSD1 268, EIC=267.7:268.7

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD10326\01032622.D Sample Name: 400 ppb 2192

```
Injection Date    : 3/27/2001 2:22:00 AM          Seq. Line :  13
Sample Name       : 400 ppb 2192                   Location : Vial 1
Acq. Operator     : wrc                                Inj :   1
                                                 Inj Volume : 25 µl
Acq. Method       : C:\HPCHEM\1\METHODS\ESPG011.M
Last changed      : 1/16/2001 12:28:53 PM by wrc
Analysis Method   : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed      : 7/2/2001 2:28:06 PM by wrc
                    (modified after loading)
+ESI gradient method, reversed phase
```



Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
```

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	7.610	MM	0.2883	3.88501e6	2.24610e5	100.0000
Totals :				3.88501e6	2.24610e5	

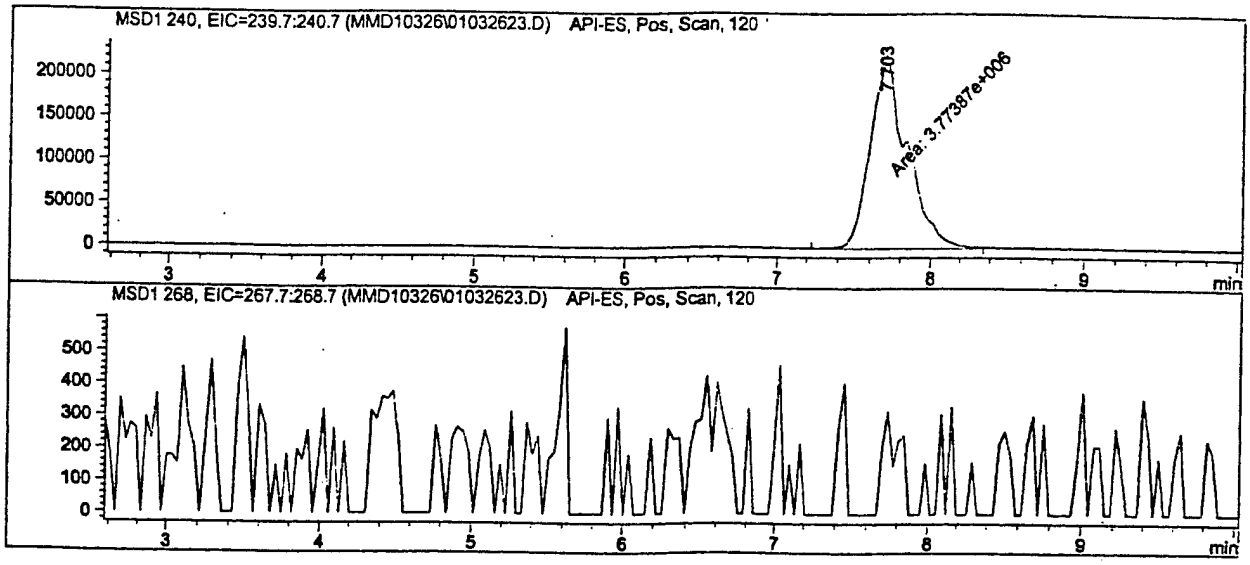
Signal 2: MSD1 268, EIC=267.7:268.7

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD10326\01032623.D Sample Name: 400 ppb 2192

Injection Date : 3/27/2001 2:33:50 AM Seq. Line : 14
Sample Name : 400 ppb 2192 Location : Vial 1
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG011.M
Last changed : 1/16/2001 12:28:53 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed : 7/2/2001 2:28:06 PM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	7.703	MM	0.2735	3.77387e6	2.29955e5	100.0000
Totals :				3.77387e6	2.29955e5	

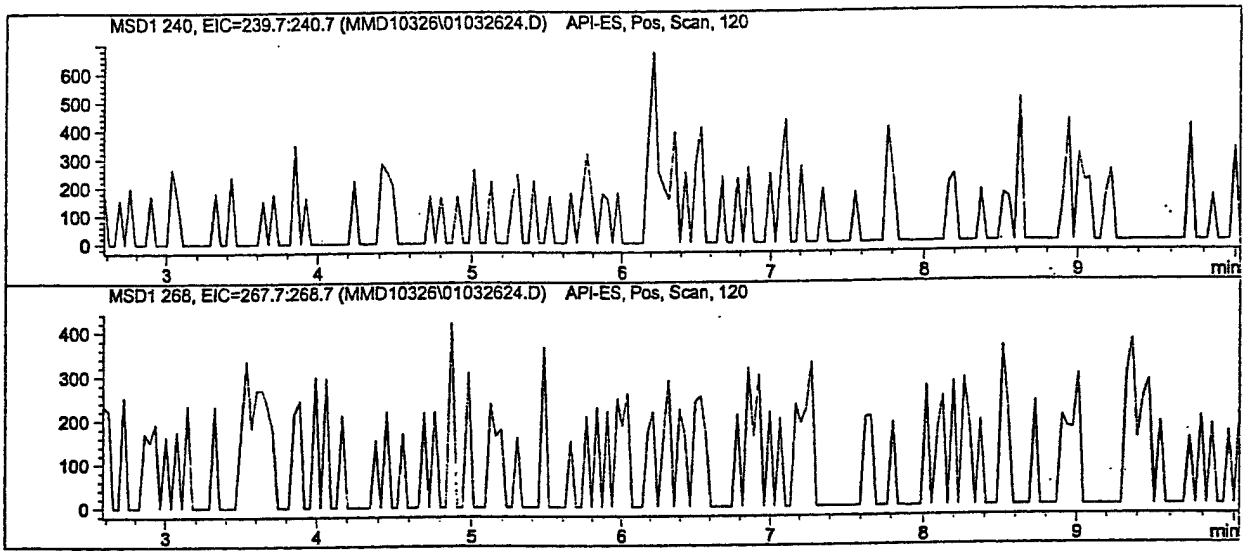
Signal 2: MSD1 268, EIC=267.7:268.7

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD10326\01032624.D Sample Name: blank

Injection Date	: 3/27/2001 2:45:41 AM	Seq. Line	: 15
Sample Name	: blank	Location	: Vial 41
Acq. Operator	: wrc	Inj	: 1
		Inj Volume	: 25 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ESPG011.M		
Last changed	: 1/16/2001 12:28:53 PM by wrc		
Analysis Method	: C:\HPCHEM\1\METHODS\ESPG012.M		
Last changed	: 7/2/2001 2:28:06 PM by wrc (modified after loading)		

+ESI gradient method, reversed phase



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

No peaks found

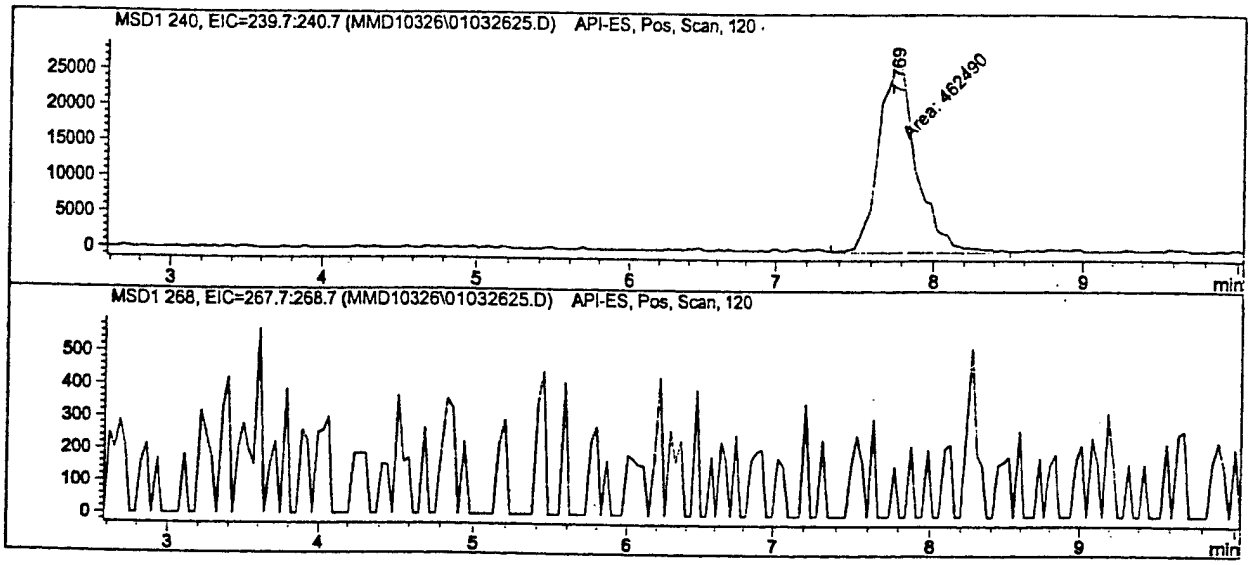
*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD10326\01032625.D Sample Name: 40 ppb 2192

Injection Date : 3/27/2001 2:57:31 AM
Sample Name : 40 ppb 2192
Acq. Operator : wrc

Seq. Line : 16
Location : Vial 2
Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG011.M
Last changed : 1/16/2001 12:28:53 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed : 7/2/2001 2:28:06 PM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

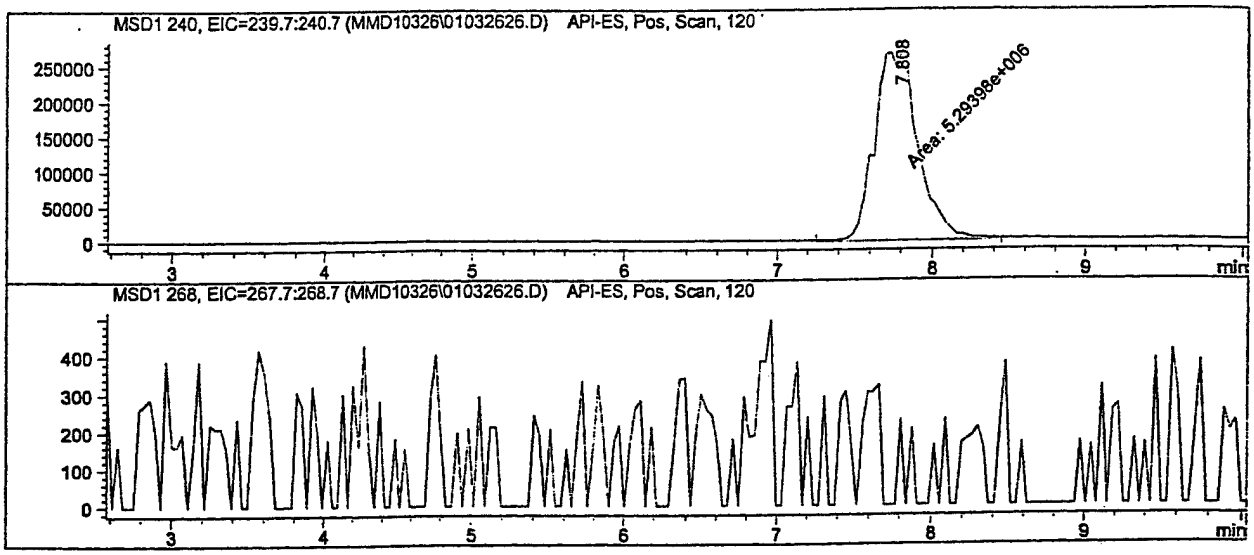
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	7.769	MM	0.2756	4.62490e5	2.79662e4	100.0000
Totals :				4.62490e5	2.79662e4	

Signal 2: MSD1 268, EIC=267.7:268.7

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD10326\01032626.D Sample Name: 1 ppm 2192

```
Injection Date   : 3/27/2001 3:09:22 AM          Seq. Line :  17
Sample Name      : 1 ppm 2192                    Location : Vial 3
Acq. Operator    : wrc                               Inj :   1
                                                 Inj Volume : 25 µl
Acq. Method      : C:\HPCHEM\1\METHODS\ESPG011.M
Last changed     : 1/16/2001 12:28:53 PM by wrc
Analysis Method  : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed     : 7/2/2001 2:28:06 PM by wrc
                   (modified after loading)
+ESI gradient method, reversed phase
```



Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
```

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	7.808	MM	0.3195	5.29398e6	2.76201e5	100.0000
Totals :				5.29398e6	2.76201e5	

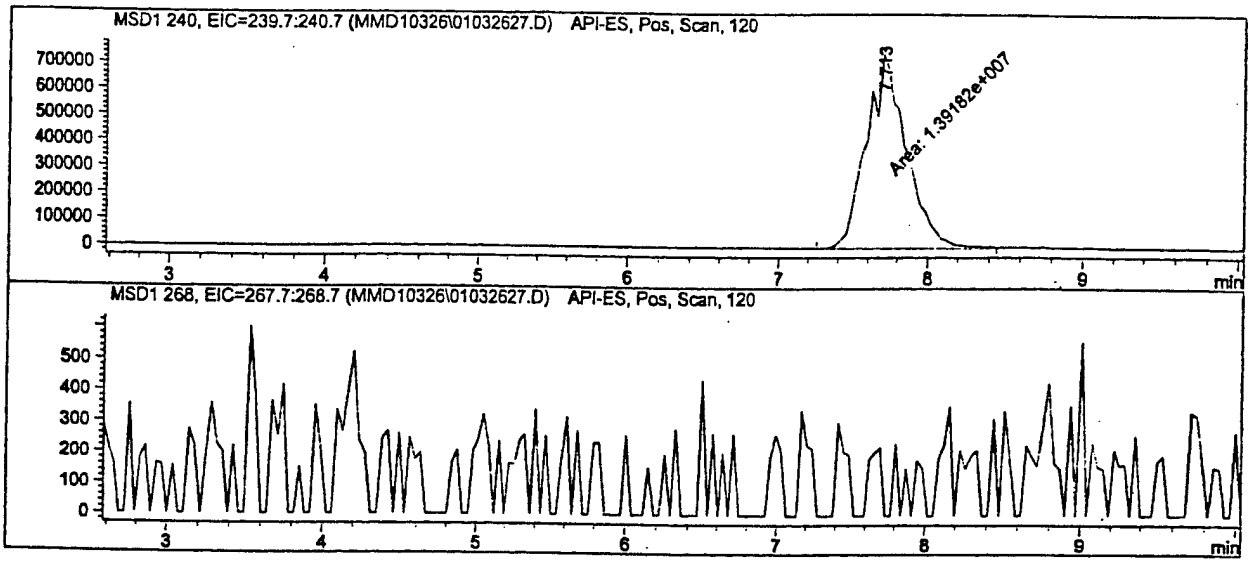
Signal 2: MSD1 268, EIC=267.7:268.7

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD10326\01032627.D Sample Name: 2 ppm 2192

Injection Date : 3/27/2001 3:21:12 AM Seq. Line : 18
Sample Name : 2 ppm 2192 Location : Vial 4
Acq. Operator : wrc Inj : 1
Inj Volume : 25 µl

Acq. Method : C:\HPCHEM\1\METHODS\ESPG011.M
Last changed : 1/16/2001 12:28:53 PM by wrc
Analysis Method : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed : 7/2/2001 2:28:06 PM by wrc
(modified after loading)
+ESI gradient method, reversed phase



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	7.713	MM	0.3002	1.39182e7	7.72625e5	100.0000
Totals :				1.39182e7	7.72625e5	

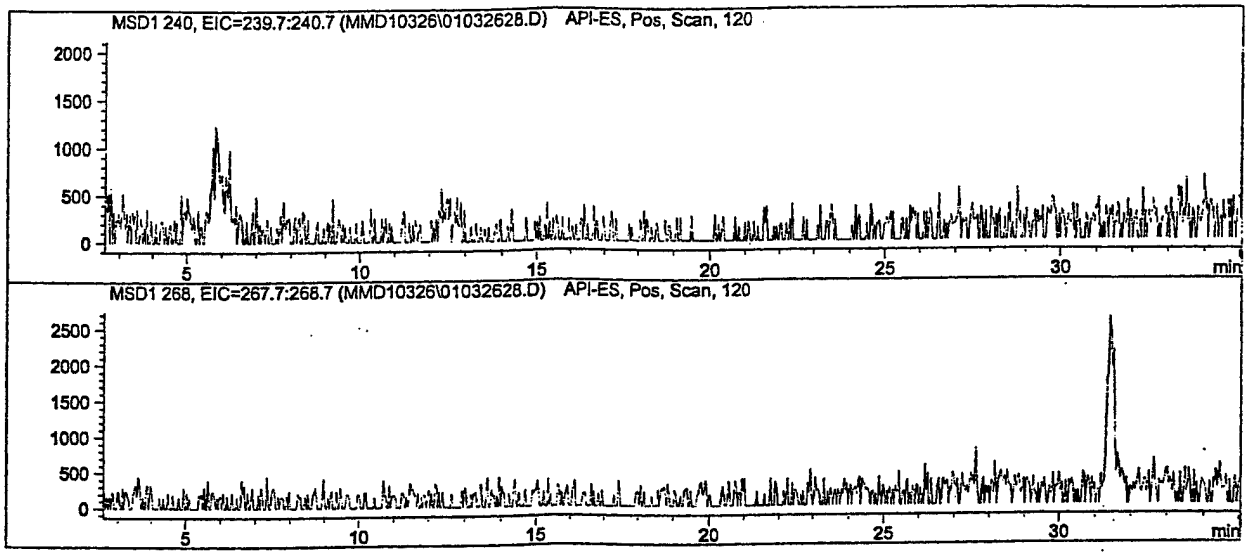
Signal 2: MSD1 268, EIC=267.7:268.7

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD10326\01032628.D Sample Name: blank

Injection Date	: 3/27/2001 3:33:34 AM	Seq. Line	: 19
Sample Name	: blank	Location	: Vial 41
Acq. Operator	: wrc	Inj	: 1
		Inj Volume	: 25 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ESPG012.M		
Last changed	: 1/25/2001 10:15:20 AM by wrc		
Analysis Method	: C:\HPCHEM\1\METHODS\ESPG012.M		
Last changed	: 7/2/2001 2:28:06 PM by wrc (modified after loading)		

+ESI gradient method, reversed phase



Area Percent Report

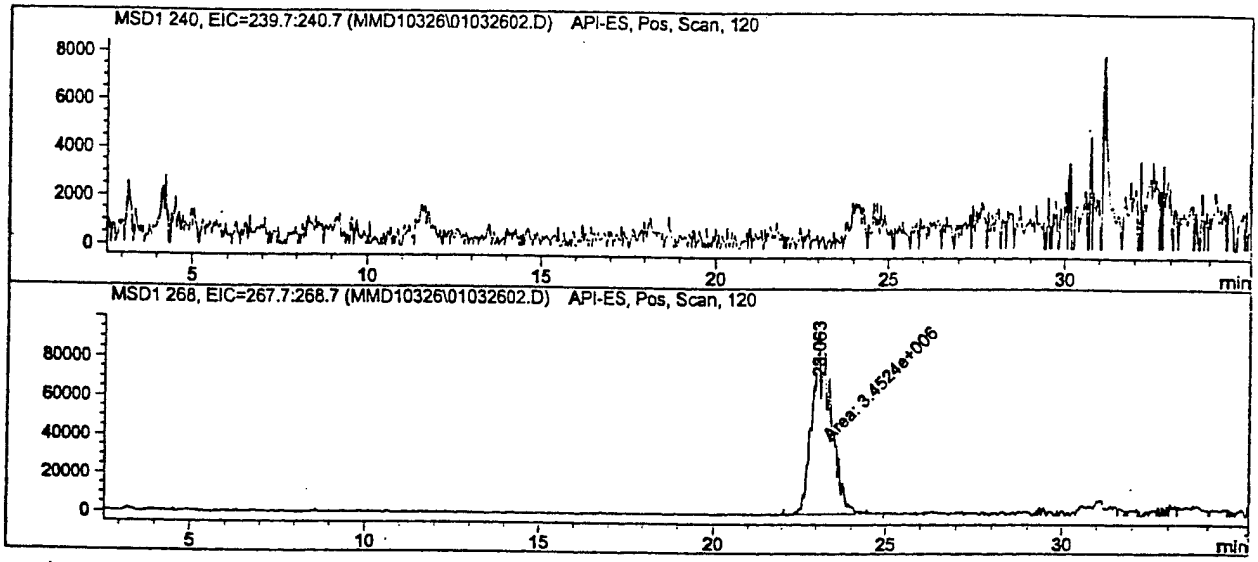
Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

No peaks found

*** End of Report ***

Data File C:\HPCHEM\1\DATA\MMD10326\01032602.D Sample Name: 200 ppb VX

```
Injection Date   : 3/26/2001 12:53:00 PM        Seq. Line :   2
Sample Name      : 200 ppb VX                   Location : Vial 7
Acq. Operator    : wrc                             Inj :   1
                                              Inj Volume : 25 µl
Acq. Method      : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed     : 1/25/2001 10:15:20 AM by wrc
Analysis Method  : C:\HPCHEM\1\METHODS\ESPG012.M
Last changed     : 7/2/2001 2:28:06 PM by wrc
                   (modified after loading)
+ESI gradient method, reversed phase
```




```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: MSD1 240, EIC=239.7:240.7

Signal 2: MSD1 268, EIC=267.7:268.7

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]                           %
----|-------|----|-------|----------|----------|--------|
   1  23.063 MM   0.5864 3.45240e6  9.81162e4  100.0000

Totals :                  3.45240e6  9.81162e4

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

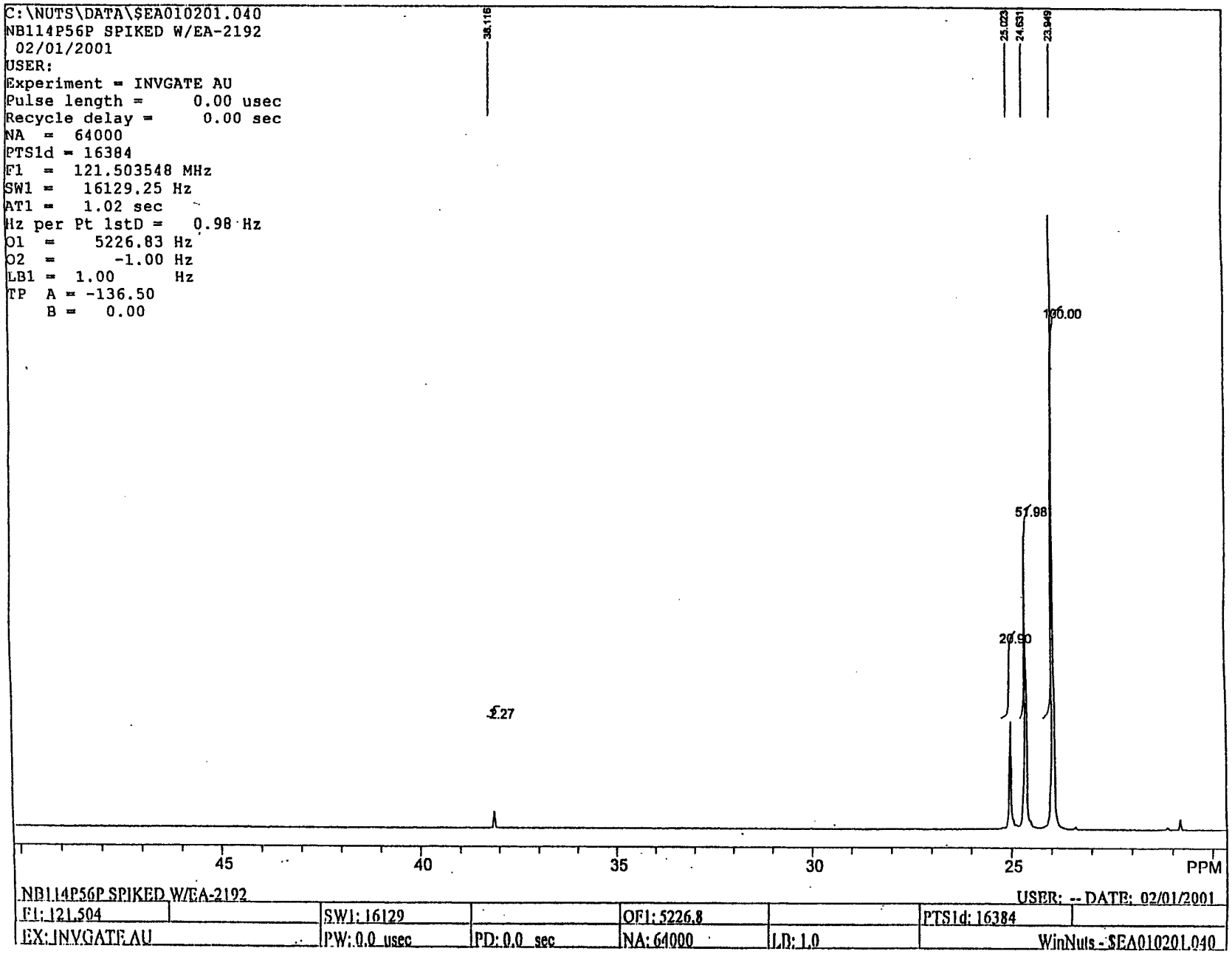
APPENDIX E

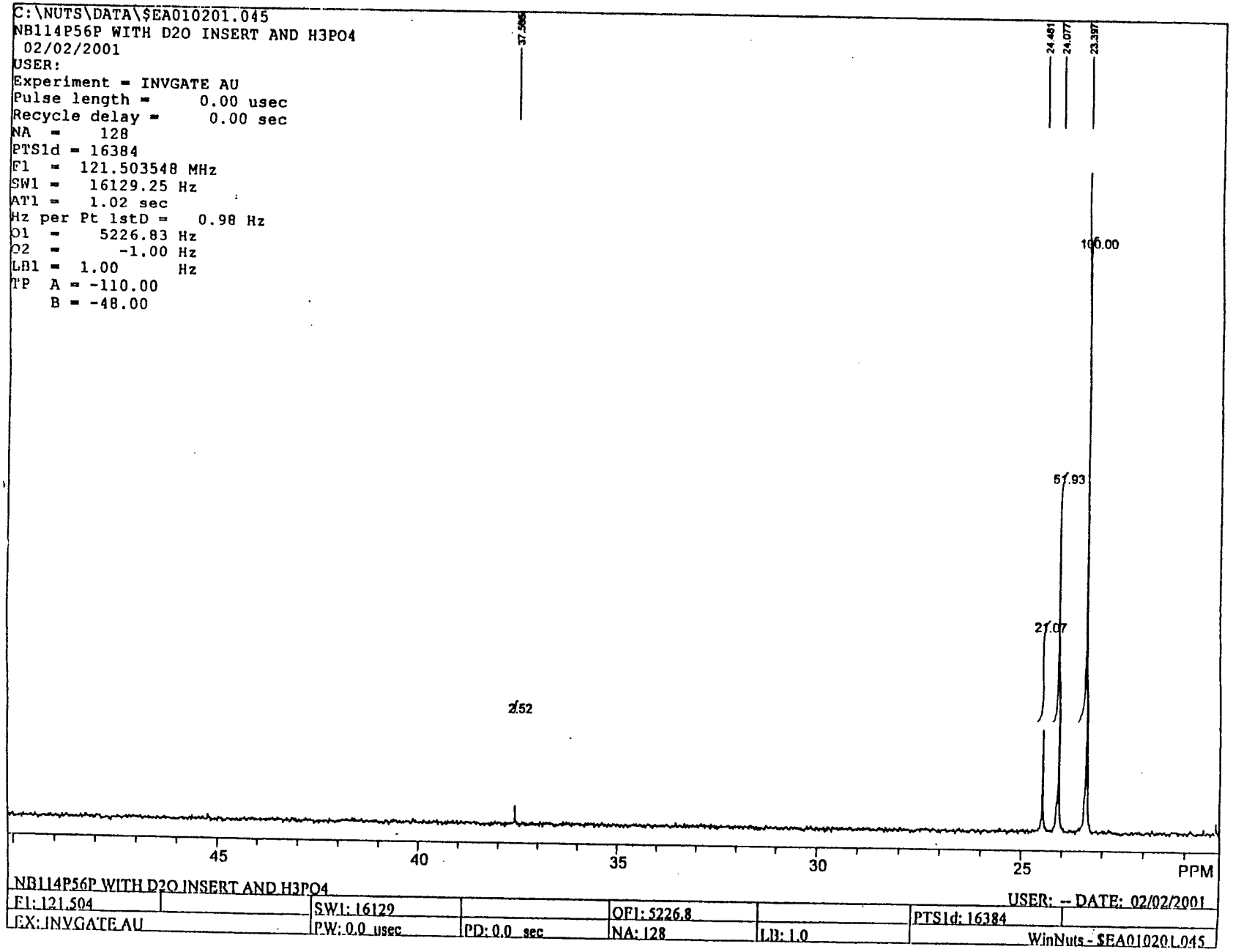
RAW DATA FOR EA-2192 DECONTAMINATION KINETICS FROM THE RUN OF 1% VX IN DECONTAMINATION SOLUTION, AT ROOM TEMPERATURE, BY ^{31}P NMR

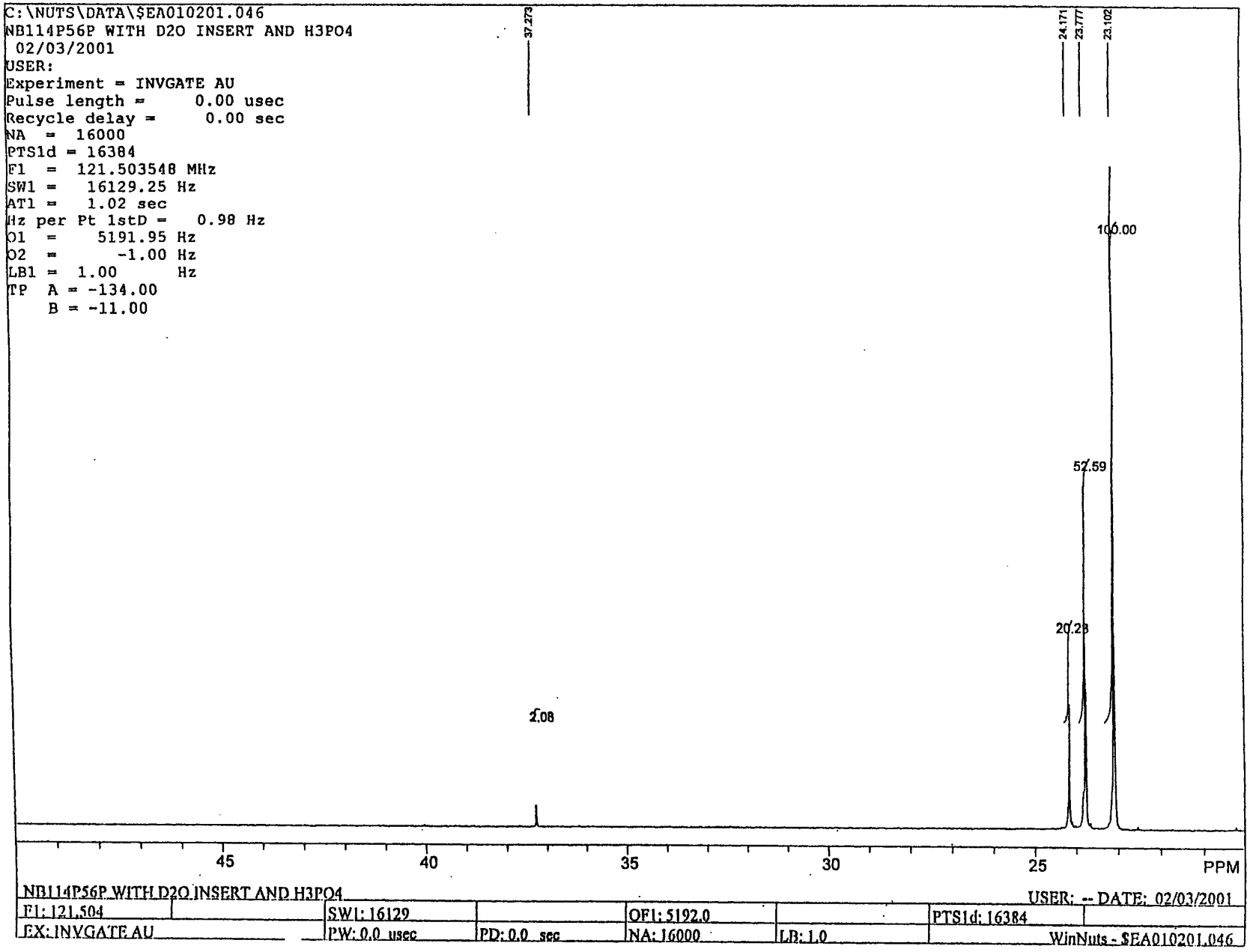
Tabulated data:

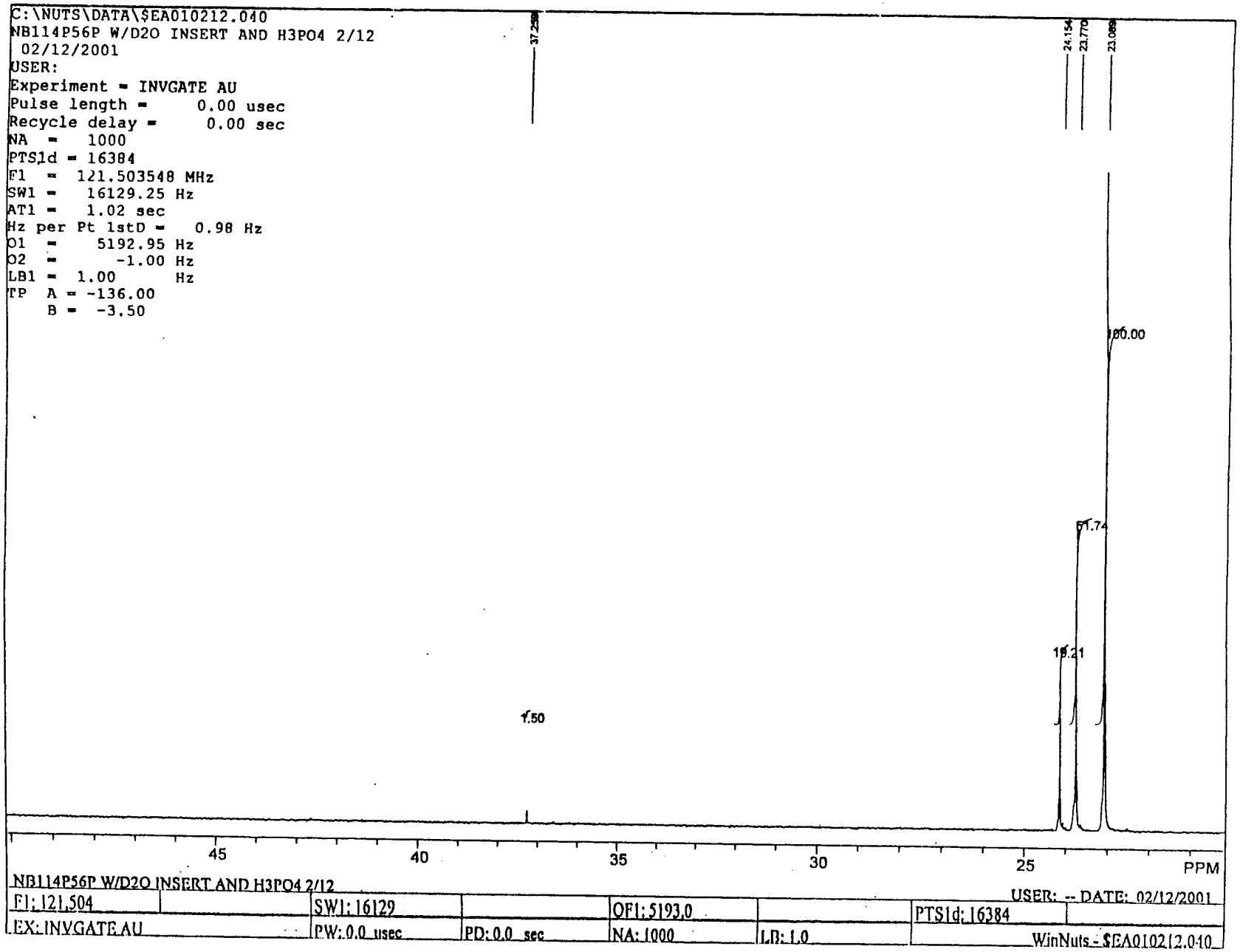
Date	Data File	# of Days	Integral 1	2	3	4	Combined Area	EA2192/ Combined Area
29-Jan-01	EA010201.040	0	2.27	20.90	51.98	100.00	175.15	0.013
2-Feb-01	EA010201.045	4	2.52	21.07	51.93	100.00	175.52	0.014
2-Feb-01	EA010201.046	4	2.08	20.23	52.59	100.00	174.90	0.012
12-Feb-01	EA010212.040	14	1.50	19.21	51.74	100.00	172.45	0.009
12-Feb-01	EA010212.041	14	1.68	19.47	50.02	100.00	171.17	0.010
12-Feb-01	EA010212.042	14	1.41	18.35	50.10	100.00	169.86	0.008
20-Feb-01	EA0220.040	22	1.35	18.85	50.53	100.00	170.73	0.008
26-Feb-01	EA010226.040	28	1.22	18.23	49.89	100.00	169.34	0.007
6-Mar-01	EA010306.040	36	0.84	18.03	49.18	100.00	168.05	0.005
16-Mar-01	EA010316.040	46	0.64	19.09	49.68	100.00	169.41	0.004
22-Mar-01	EA010322.040	52	0.54	20.05	50.57	100.00	171.16	0.003
30-Mar-01	EA010330.040	60	0.26	19.04	49.76	100.00	169.06	0.002

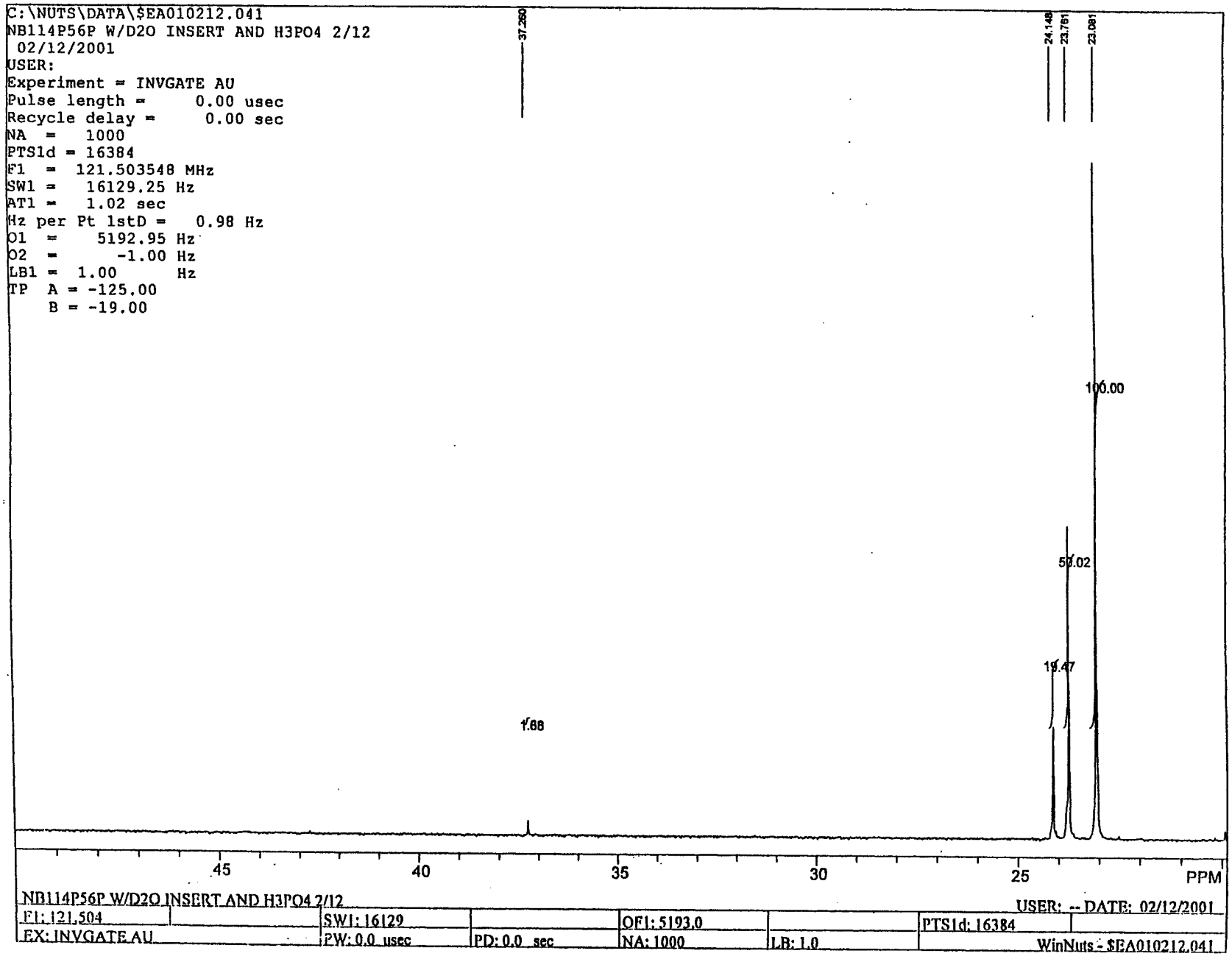
# of Days	Combined Area	EA2192/ Combined Area	log EA2192/ Combined Area	Norm. to Comb. Area=1%	log(norm.)
0	175.15	0.013	-1.887	129.60	2.112616
4	175.52	0.014	-1.843	143.57	2.157074
4	174.90	0.012	-1.925	118.93	2.075274
14	172.45	0.009	-2.061	86.98	1.939428
14	171.17	0.010	-2.008	98.15	1.991882
14	169.86	0.008	-2.081	83.01	1.919128
22	170.73	0.008	-2.102	79.07	1.898024
28	169.34	0.007	-2.142	72.04	1.8576
36	168.05	0.005	-2.301	49.99	1.698841
46	169.41	0.004	-2.423	37.78	1.577241
52	171.16	0.003	-2.501	31.55	1.498991
60	169.06	0.002	-2.813	15.38	1.186932

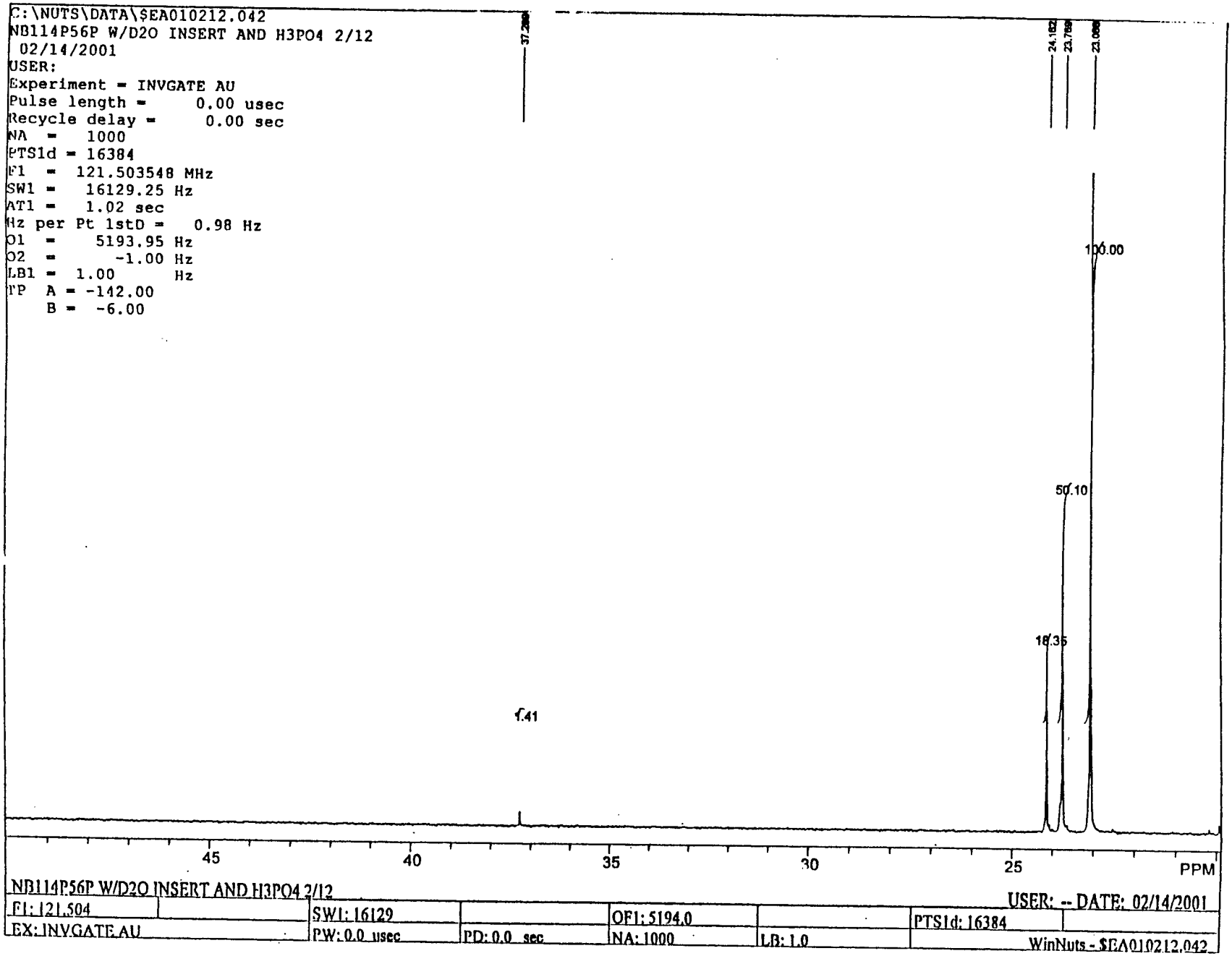

C:\NUTS\DATA\$EA010201.040
NB114P56P SPIKED W/EA-2192
02/01/2001
USER:
Experiment = INVGATE AU
Pulse length = 0.00 usec
Recycle delay = 0.00 sec
NA = 64000
PTS1d = 16384
F1 = 121.503548 MHz
SW1 = 16129.25 Hz
AT1 = 1.02 sec
Hz per Pt 1stD = 0.98 Hz
O1 = 5226.83 Hz
O2 = -1.00 Hz
LB1 = 1.00 Hz
TP A = -136.50
B = 0.00
38.116
25.023
24.631
23.949
100.00
51.98
20.90
2.27
45
40
35
30
25
PPM
NB114P56P SPIKED W/EA-2192
USER: -- DATE: 02/01/2001
F1: 121.504
SW1: 16129
OF1: 5226.8
PTS1d: 16384
EX: INVGATE.AU
PW: 0.0 usec
PD: 0.0 sec
NA: 64000
LD: 1.0
WinNuts - $EA010201.040

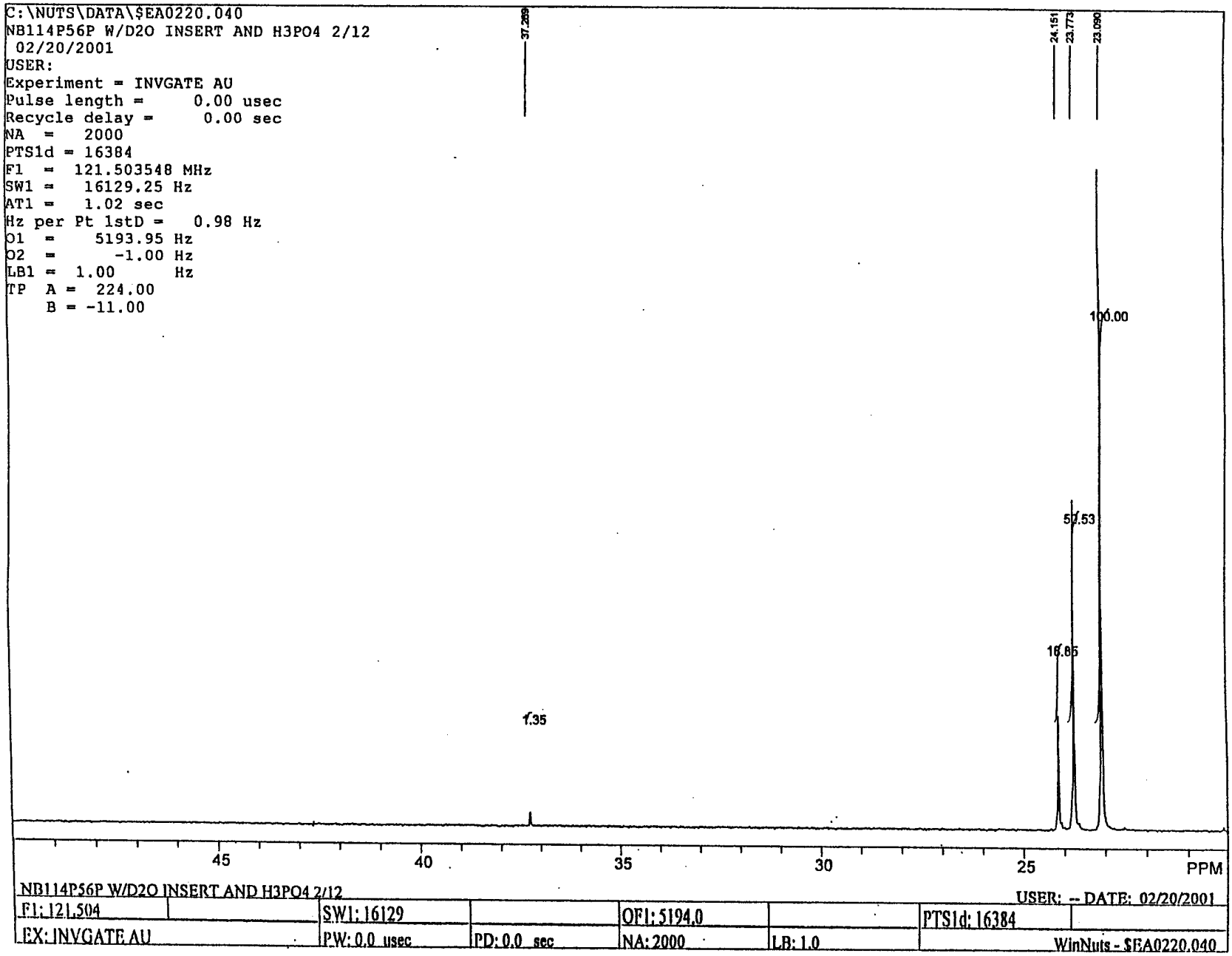

C:\NUTS\DATA\$EA010201.045
NB114P56P WITH D2O INSERT AND H3PO4
02/02/2001
USER:
Experiment = INVGATE AU
Pulse length = 0.00 usec
Recycle delay = 0.00 sec
NA = 128
PTS1d = 16384
F1 = 121.503548 MHz
SW1 = 16129.25 Hz
AT1 = 1.02 sec
Hz per Pt 1stD = 0.98 Hz
O1 = 5226.83 Hz
O2 = -1.00 Hz
LB1 = 1.00 Hz
TP A = -110.00
B = -48.00
37.595
24.491
24.077
23.397
100.00
51.93
21.07
2.52
45
40
35
30
25
PPM
NB114P56P WITH D2O INSERT AND H3PO4
USER: -- DATE: 02/02/2001
F1: 121.504
SW1: 16129
OF1: 5226.8
PTS1d: 16384
EX: INVGATE AU
PW: 0.0 usec
PD: 0.0 sec
NA: 128
LB: 1.0
WinNuts - $EA01020L045

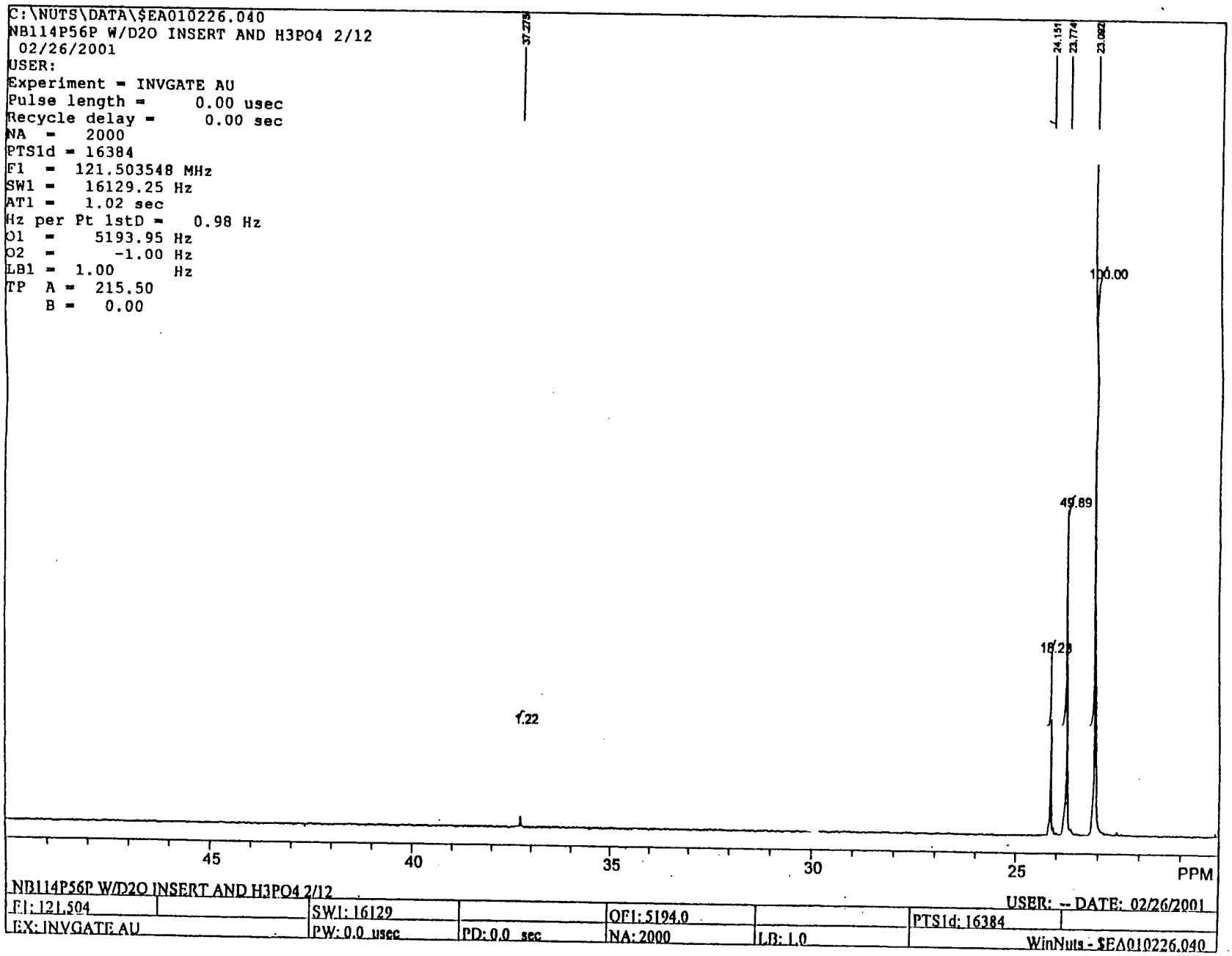

C:\NUTS\DATA\$EA010201.046
NB114P56P WITH D2O INSERT AND H3PO4
02/03/2001
USER:
Experiment = INVGATE AU
Pulse length = 0.00 usec
Recycle delay = 0.00 sec
NA = 16000
PTS1d = 16384
F1 = 121.503548 MHz
SW1 = 16129.25 Hz
AT1 = 1.02 sec
Hz per Pt 1stD = 0.98 Hz
O1 = 5191.95 Hz
O2 = -1.00 Hz
LB1 = 1.00 Hz
TP A = -134.00
B = -11.00
37.273
24.171
23.777
23.102
100.00
52.59
20.28
2.08
45
40
35
30
25
PPM
NB114P56P WITH D2O INSERT AND H3PO4
USER: -- DATE: 02/03/2001
F1: 121.504
SW1: 16129
OF1: 5192.0
PTS1d: 16384
EX: INVGATE AU
PW: 0.0 usec
PD: 0.0 sec
NA: 16000
LB: 1.0
WinNuts - $EA010201.046

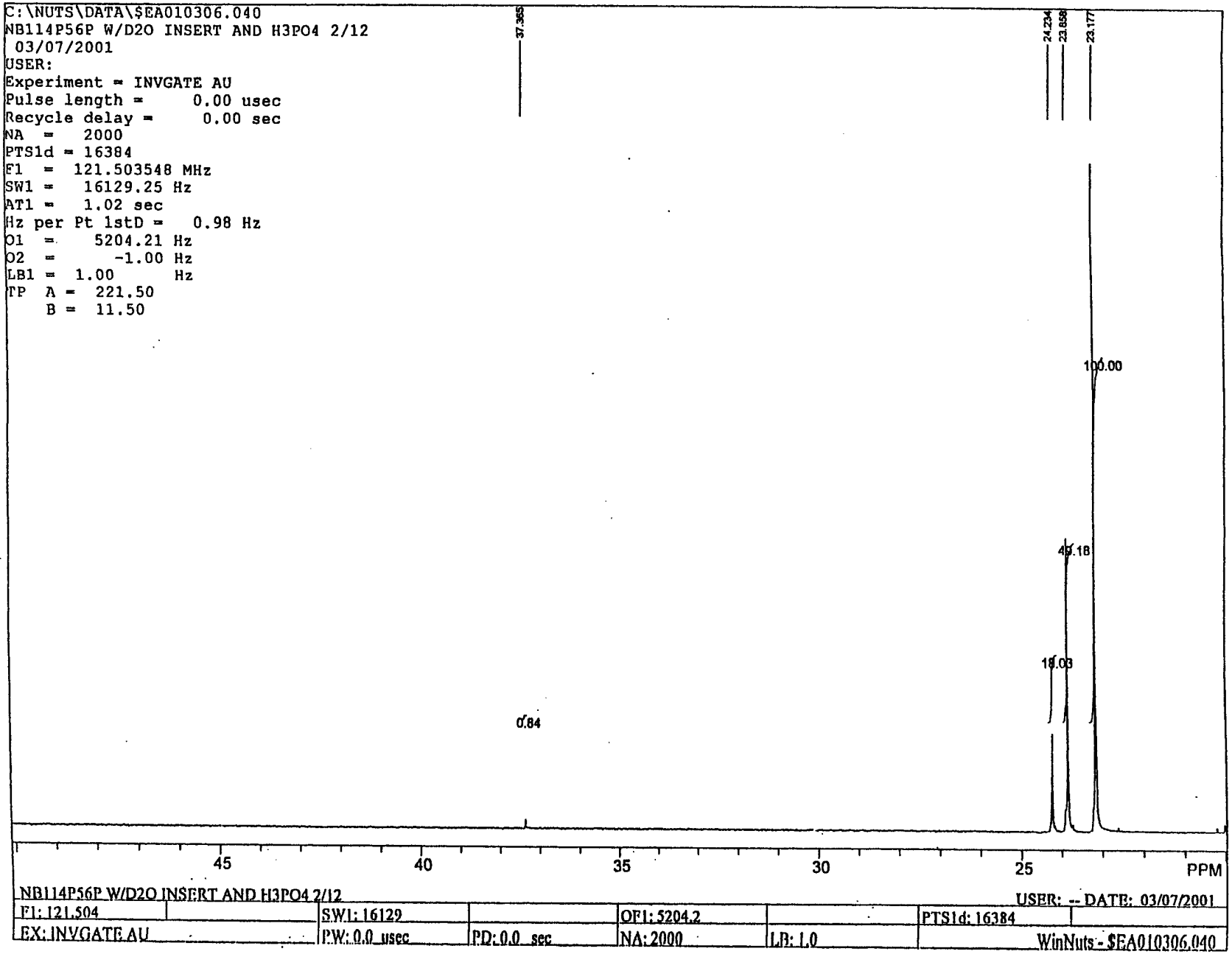

C:\NUTS\DATA\$EA010212.040
NB114P56P W/D2O INSERT AND H3PO4 2/12
02/12/2001
USER:
Experiment = INVGATE AU
Pulse length = 0.00 usec
Recycle delay = 0.00 sec
NA = 1000
PTS1d = 16384
F1 = 121.503548 MHz
SW1 = 16129.25 Hz
AT1 = 1.02 sec
Hz per Pt 1stD = 0.98 Hz
O1 = 5192.95 Hz
O2 = -1.00 Hz
LB1 = 1.00 Hz
TP A = -136.00
B = -3.50
37.258
24.154
23.770
23.089
100.00
51.74
19.21
1.50
45
40
35
30
25
PPM
NB114P56P W/D2O INSERT AND H3PO4 2/12
USER: -- DATE: 02/12/2001
F1: 121.504
SW1: 16129
OF1: 5193.0
PTS1d: 16384
EX: INVGATE.AU
PW: 0.0 usec
PD: 0.0 sec
NA: 1000
LB: 1.0
WinNuts - $EA010212.040

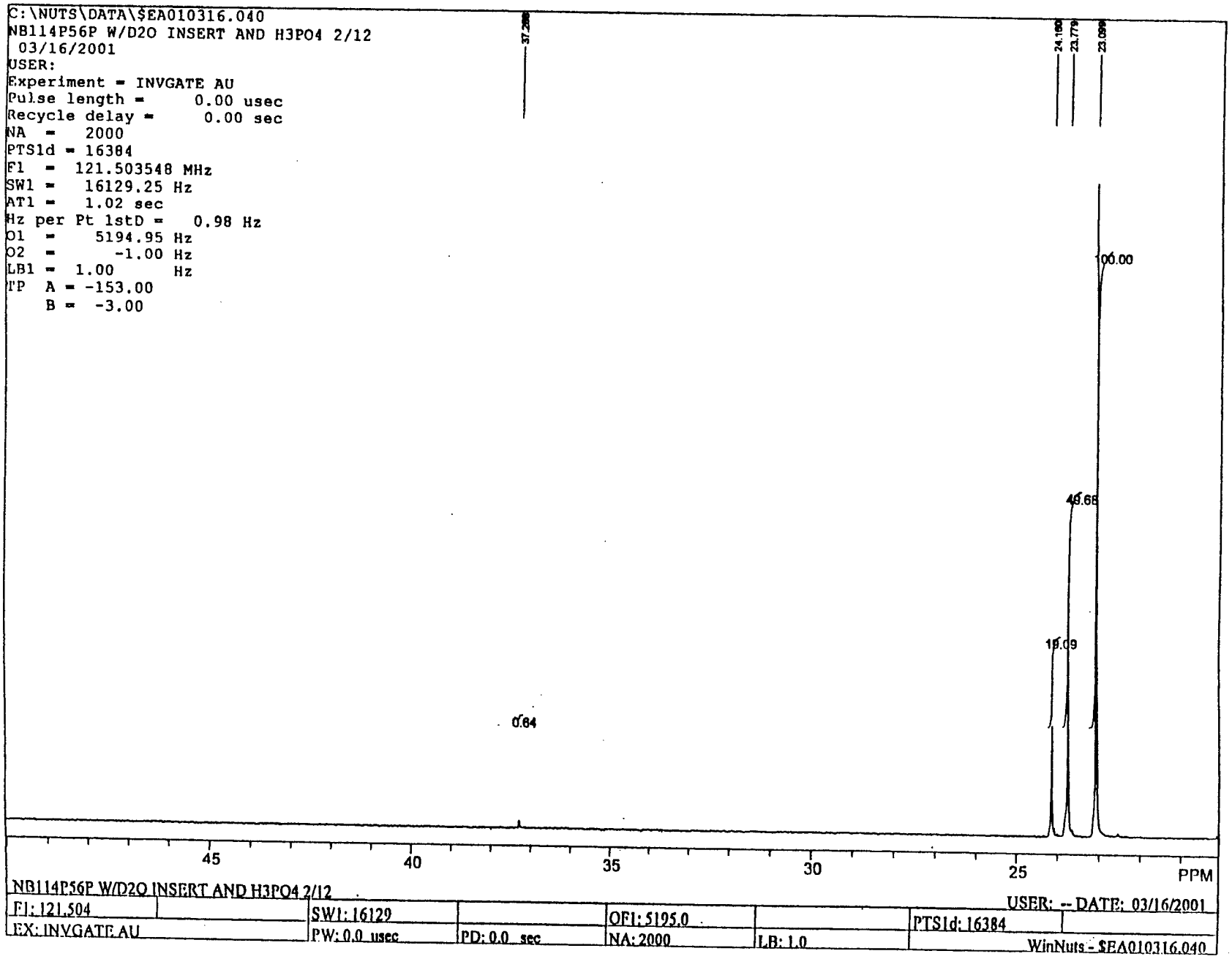

C:\NUTS\DATA\$EA010212.041
NB114P56P W/D2O INSERT AND H3PO4 2/12
02/12/2001
USER:
Experiment = INVGATE AU
Pulse length = 0.00 usec
Recycle delay = 0.00 sec
NA = 1000
PTS1d = 16384
F1 = 121.503548 MHz
SW1 = 16129.25 Hz
AT1 = 1.02 sec
Hz per Pt 1stD = 0.98 Hz
O1 = 5192.95 Hz
O2 = -1.00 Hz
LB1 = 1.00 Hz
TP A = -125.00
B = -19.00
37.260
24.148
23.761
23.081
100.00
50.02
19.47
1.68
45
40
35
30
25
PPM
NB114P56P W/D2O INSERT AND H3PO4 2/12
USER: -- DATE: 02/12/2001
F1: 121.504
SW1: 16129
OF1: 5193.0
PTS1d: 16384
EX: INVGATE.AU
PW: 0.0 usec
PD: 0.0 sec
NA: 1000
LB: 1.0
WinNuts - $EA010212.041

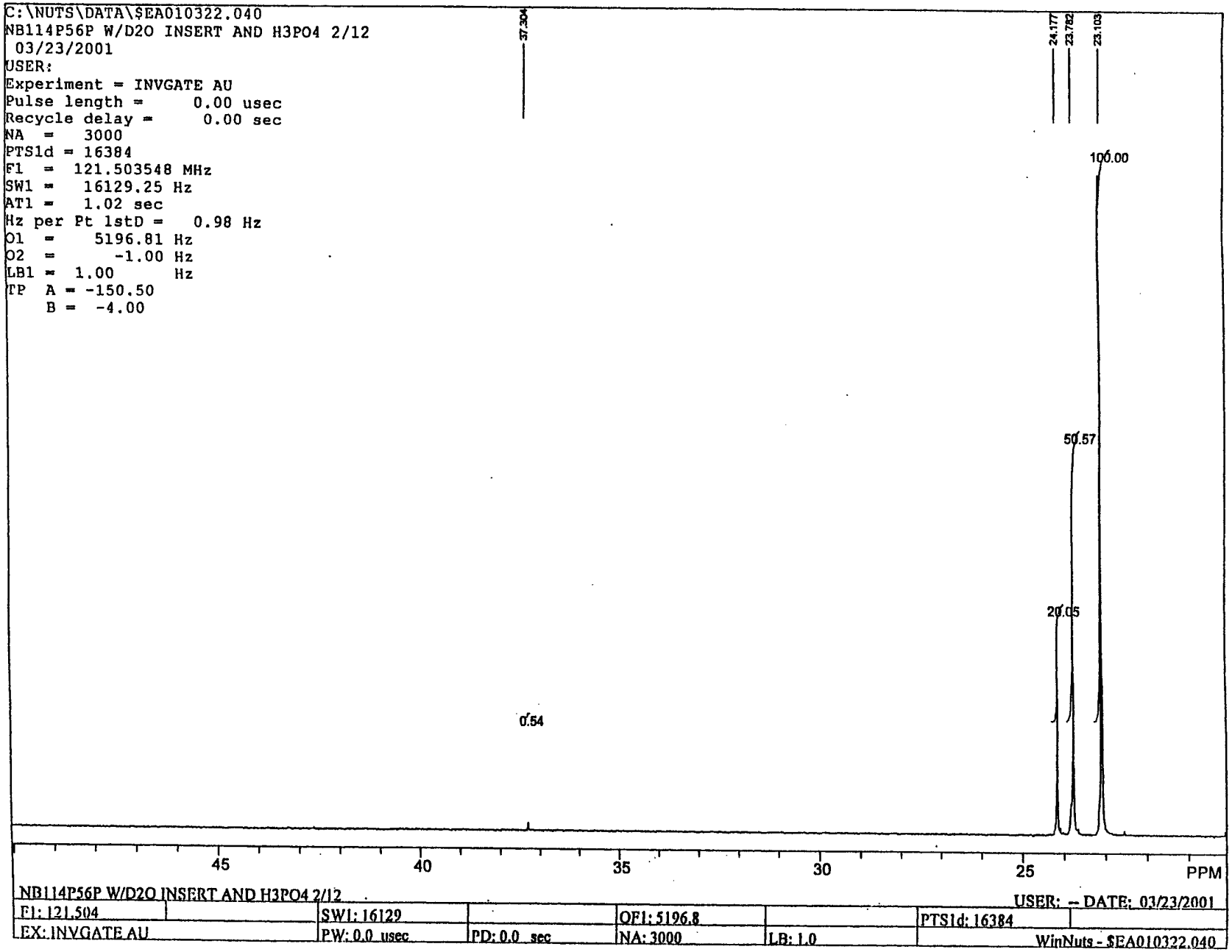

C:\NUTS\DATA\$EA010212.042
NB114P56P W/D2O INSERT AND H3PO4 2/12
02/14/2001
USER:
Experiment = INVGATE AU
Pulse length = 0.00 usec
Recycle delay = 0.00 sec
NA = 1000
PTS1d = 16384
F1 = 121.503548 MHz
SW1 = 16129.25 Hz
AT1 = 1.02 sec
Hz per Pt 1stD = 0.98 Hz
O1 = 5193.95 Hz
O2 = -1.00 Hz
LB1 = 1.00 Hz
TP A = -142.00
B = -6.00
24.182
23.759
23.069
100.00
50.10
18.35
1.41
45
40
35
30
25
PPM
NB114P56P W/D2O INSERT AND H3PO4 2/12
USER: -- DATE: 02/14/2001
F1: 121.504
SW1: 16129
OF1: 5194.0
PTS1d: 16384
EX: INVGATE.AU
PW: 0.0 usec
PD: 0.0 sec
NA: 1000
LB: 1.0
WinNuts - $EA010212.042

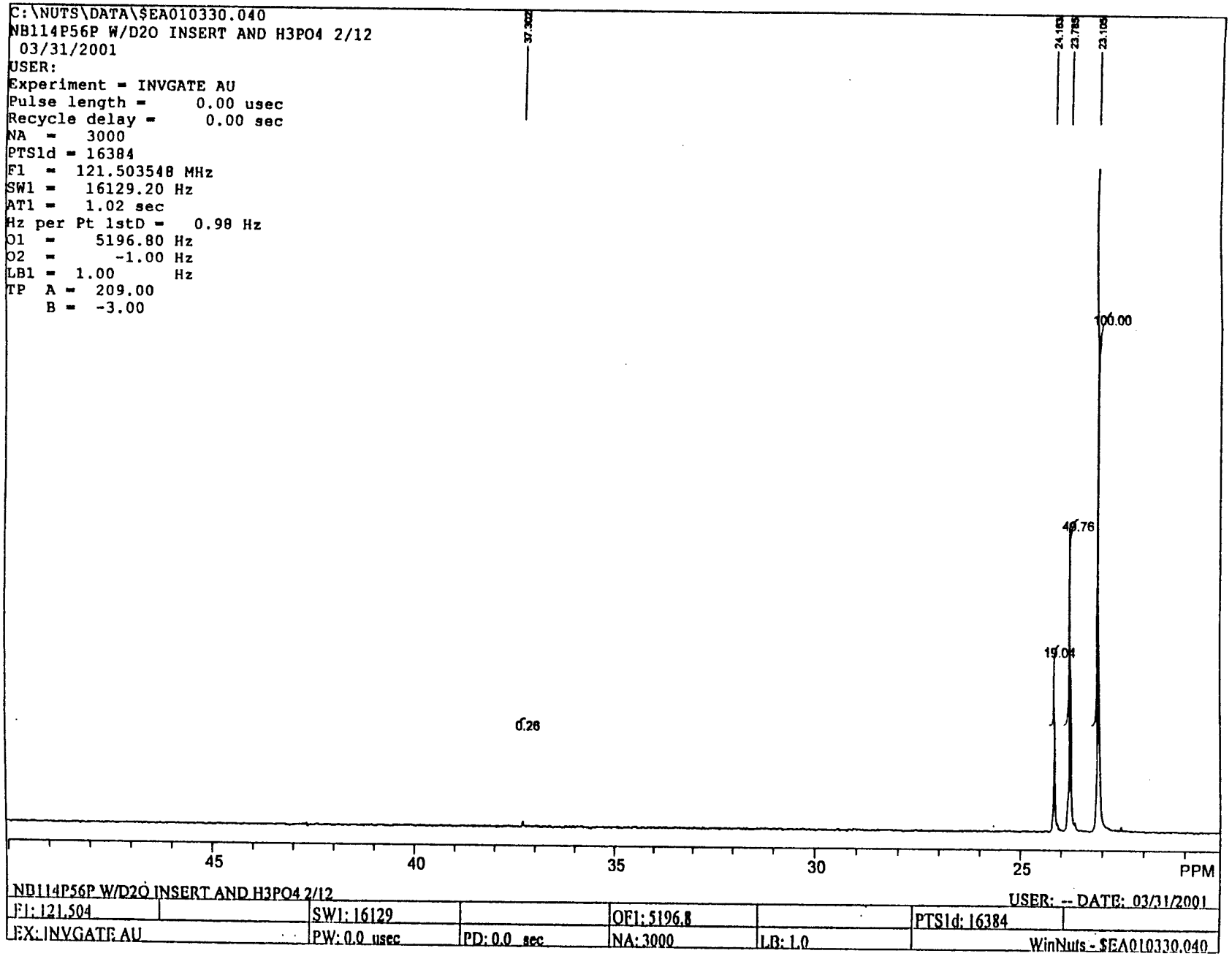

C:\NUTS\DATA\$EA0220.040
NB114P56P W/D2O INSERT AND H3PO4 2/12
02/20/2001
USER:
Experiment = INVGATE AU
Pulse length = 0.00 usec
Recycle delay = 0.00 sec
NA = 2000
PTS1d = 16384
F1 = 121.503548 MHz
SW1 = 16129.25 Hz
AT1 = 1.02 sec
Hz per Pt 1stD = 0.98 Hz
O1 = 5193.95 Hz
O2 = -1.00 Hz
LB1 = 1.00 Hz
TP A = 224.00
B = -11.00
37.269
24.151
23.773
23.090
100.00
50.53
16.86
1.35
45
40
35
30
25
PPM
NB114P56P W/D2O INSERT AND H3PO4 2/12
USER: -- DATE: 02/20/2001
F1: 121.504
SW1: 16129
OF1: 5194.0
PTS1d: 16384
EX: INVGATE AU
PW: 0.0 usec
PD: 0.0 sec
NA: 2000
LB: 1.0
WinNuts - $EA0220.040


C:\NUTS\DATA\$EA010226.040
NB114P56P W/D2O INSERT AND H3PO4 2/12
02/26/2001
USER:
Experiment = INVGATE AU
Pulse length = 0.00 usec
Recycle delay = 0.00 sec
NA = 2000
PTS1d = 16384
F1 = 121.503548 MHz
SW1 = 16129.25 Hz
AT1 = 1.02 sec
Hz per Pt 1stD = 0.98 Hz
O1 = 5193.95 Hz
O2 = -1.00 Hz
LB1 = 1.00 Hz
TP A = 215.50
B = 0.00
37.275
24.151
23.774
23.082
100.00
49.89
18.23
1.22
45
40
35
30
25
PPM
NB114P56P W/D2O INSERT AND H3PO4 2/12
USER: -- DATE: 02/26/2001
F1: 121.504
SW1: 16129
OF1: 5194.0
PTS1d: 16384
EX: INVGATE AU
PW: 0.0 usec
PD: 0.0 sec
NA: 2000
LB: 1.0
WinNuts - $EA010226.040


C:\NUTS\DATA\$EA010306.040
NB114P56P W/D2O INSERT AND H3PO4 2/12
03/07/2001
USER:
Experiment = INVGATE AU
Pulse length = 0.00 usec
Recycle delay = 0.00 sec
NA = 2000
PTS1d = 16384
F1 = 121.503548 MHz
SW1 = 16129.25 Hz
AT1 = 1.02 sec
Hz per Pt 1stD = 0.98 Hz
O1 = 5204.21 Hz
O2 = -1.00 Hz
LB1 = 1.00 Hz
TP A = 221.50
B = 11.50
37.355
24.234
23.858
23.177
100.00
45.18
18.03
0.84
45
40
35
30
25
PPM
NB114P56P W/D2O INSERT AND H3PO4 2/12
USER: -- DATE: 03/07/2001
F1: 121.504
SW1: 16129
OF1: 5204.2
PTS1d: 16384
EX: INVGATE AU
PW: 0.0 usec
PD: 0.0 sec
NA: 2000
LB: 1.0
WinNuts - $EA010306.040


C:\NUTS\DATA\$EA010316.040
NB114P56P W/D2O INSERT AND H3PO4 2/12
03/16/2001
USER:
Experiment = INVGATE AU
Pulse length = 0.00 usec
Recycle delay = 0.00 sec
NA = 2000
PTS1d = 16384
F1 = 121.503548 MHz
SW1 = 16129.25 Hz
AT1 = 1.02 sec
Hz per Pt 1stD = 0.98 Hz
O1 = 5194.95 Hz
O2 = -1.00 Hz
LB1 = 1.00 Hz
TP A = -153.00
B = -3.00
37.288
24.180
23.779
23.099
100.00
48.68
19.09
0.84
45
40
35
30
25
PPM
NB114P56P W/D2O INSERT AND H3PO4 2/12
USER: -- DATE: 03/16/2001
F1: 121.504
SW1: 16129
OF1: 5195.0
PTS1d: 16384
EX: INVGATE.AU
PW: 0.0 usec
PD: 0.0 sec
NA: 2000
LB: 1.0
WinNuts - $EA010316.040


C:\NUTS\DATA\$EA010322.040
NB114P56P W/D2O INSERT AND H3PO4 2/12
03/23/2001
USER:
Experiment = INVGATE AU
Pulse length = 0.00 usec
Recycle delay = 0.00 sec
NA = 3000
PTS1d = 16384
F1 = 121.503548 MHz
SW1 = 16129.25 Hz
AT1 = 1.02 sec
Hz per Pt 1stD = 0.98 Hz
O1 = 5196.81 Hz
O2 = -1.00 Hz
LB1 = 1.00 Hz
TP A = -150.50
B = -4.00
37.304
24.177
23.782
23.103
100.00
50.57
20.05
0.54
45
40
35
30
25
PPM
NB114P56P W/D2O INSERT AND H3PO4 2/12
USER: -- DATE: 03/23/2001
F1: 121.504
SW1: 16129
OF1: 5196.8
PTS1d: 16384
EX: INVGATE AU
PW: 0.0 usec
PD: 0.0 sec
NA: 3000
LB: 1.0
WinNuts - $EA010322.040


C:\NUTS\DATA\$EA010330.040
NB114P56P W/D2O INSERT AND H3PO4 2/12
03/31/2001
USER:
Experiment = INVGATE AU
Pulse length = 0.00 usec
Recycle delay = 0.00 sec
NA = 3000
PTS1d = 16384
F1 = 121.503548 MHz
SW1 = 16129.20 Hz
AT1 = 1.02 sec
Hz per Pt 1stD = 0.98 Hz
O1 = 5196.80 Hz
O2 = -1.00 Hz
LB1 = 1.00 Hz
TP A = 209.00
B = -3.00
37.302
24.163
23.785
23.109
100.00
49.76
19.04
0.26
45
40
35
30
25
PPM
NB114P56P W/D2O INSERT AND H3PO4 2/12
USER: -- DATE: 03/31/2001
F1: 121.504
SW1: 16129
OF1: 5196.8
PTS1d: 16384
EX: INVGATE AU
PW: 0.0 usec
PD: 0.0 sec
NA: 3000
LB: 1.0
WinNuts - $EA010330.040