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**ECBC-TR-282**

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

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## **PREFACE**

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## **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 were 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,<sup>1</sup> 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 $\mu$ m particle size. (reversed phase chromatography column)  |
| Flow rate:            | 0.25 mL/min.  |
| Mobile phase:         | Gradient<br>0-10 min.: 97.5% DI water, 2.5% acetonitrile, 1% acetic acid<br>20 min.: 50% DI water, 50% acetonitrile, 0.5% acetic acid<br>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 $\mu$ L  |
| 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.

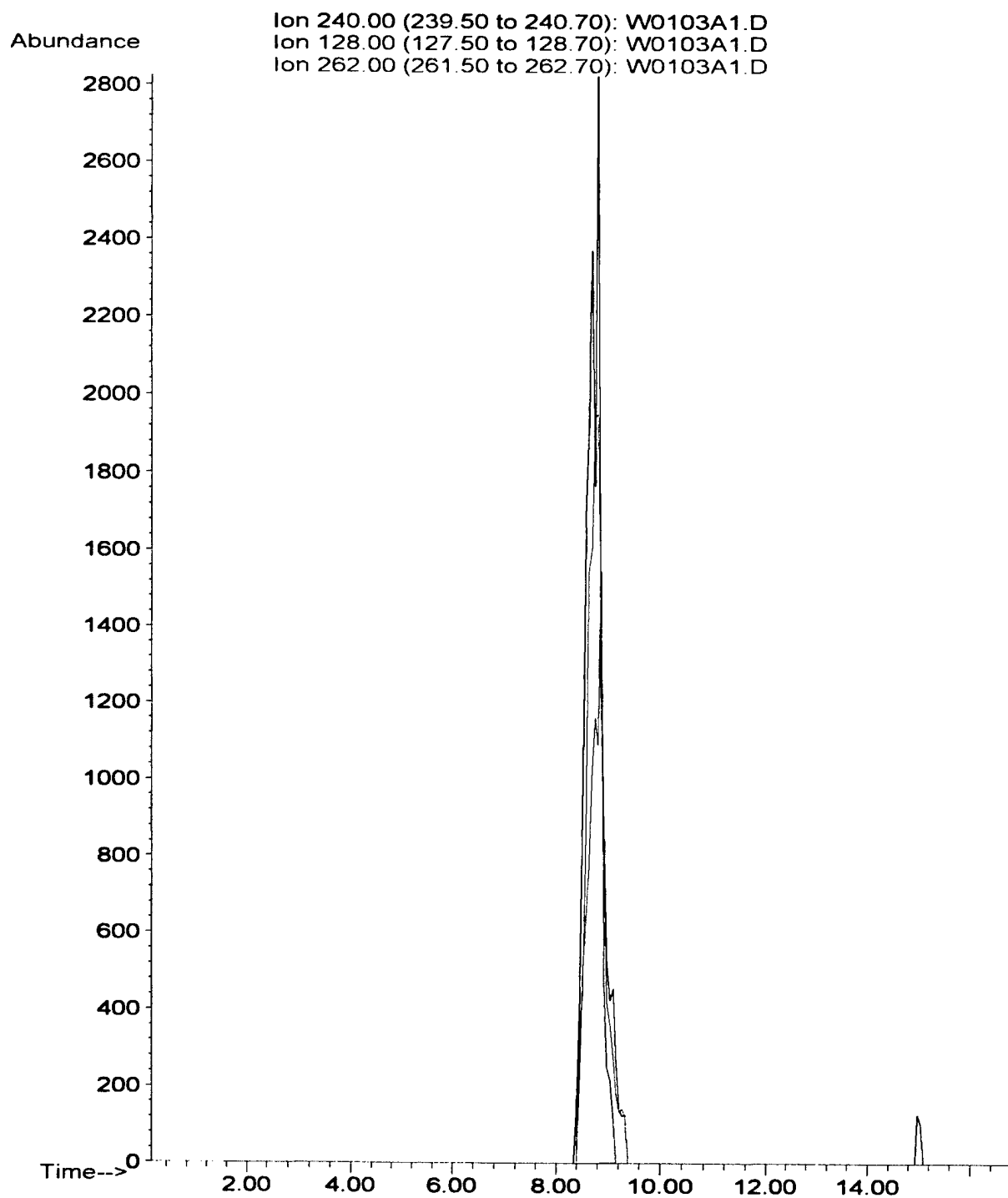
A weak anion exchange column, a Synchronpak AX-100, was used by itself and in series with a C18 column. No improvement in chromatography was observed. This column is also more advantageous for normal phase chromatography, but it was not studied in detail.

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.

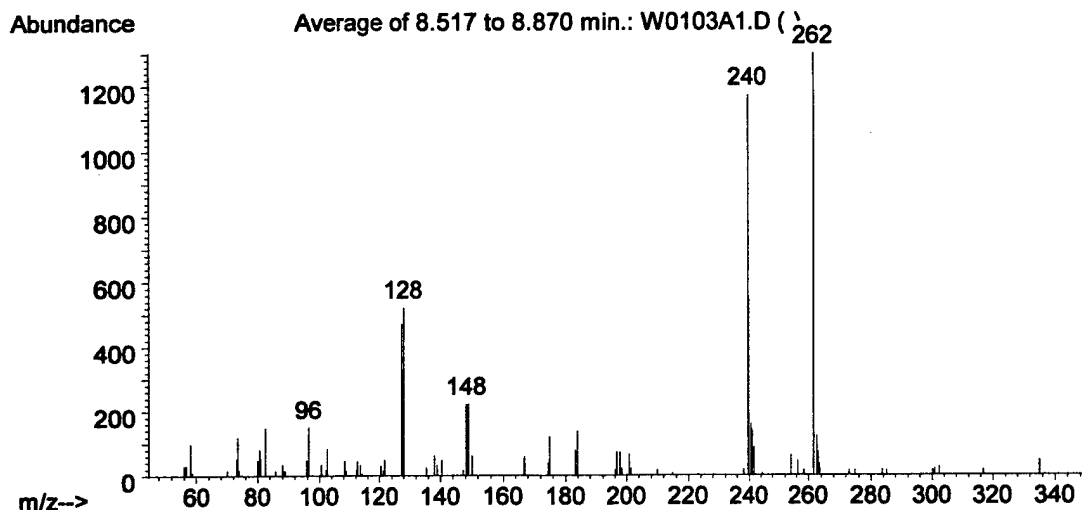
Other mobile phases were attempted. Increasing the acetonitrile concentration to 97% produced a longer retention time for EA-2192. However, the peak became broader, so sensitivity and quantitation accuracy were not as good. Mobile phases of methanol or isopropanol were not effective, because EA-2192 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.

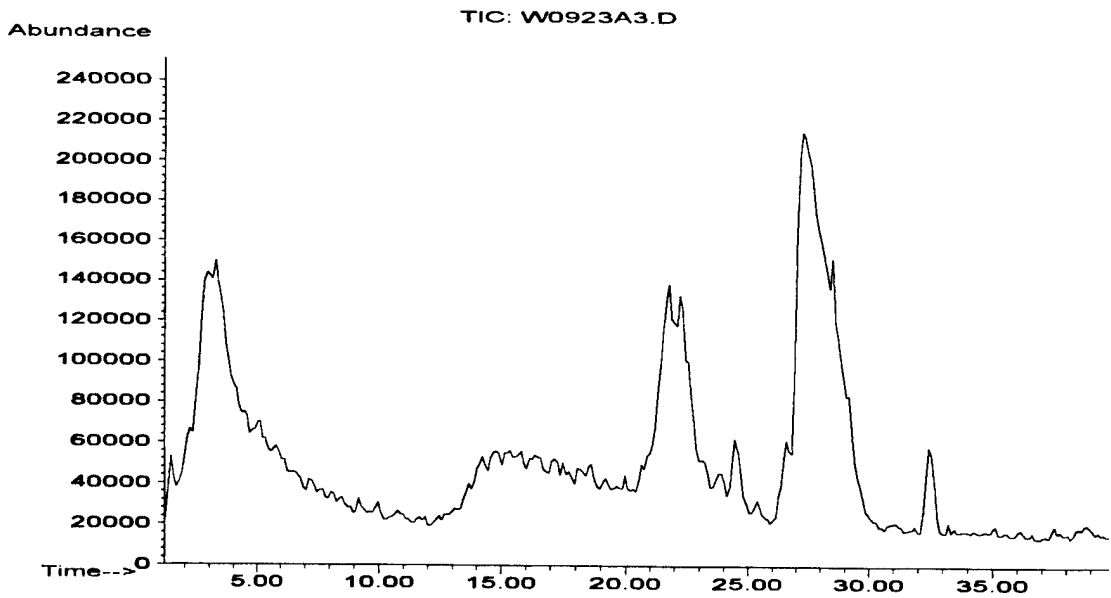
### 2.1.3 LC Mobile Phase

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.

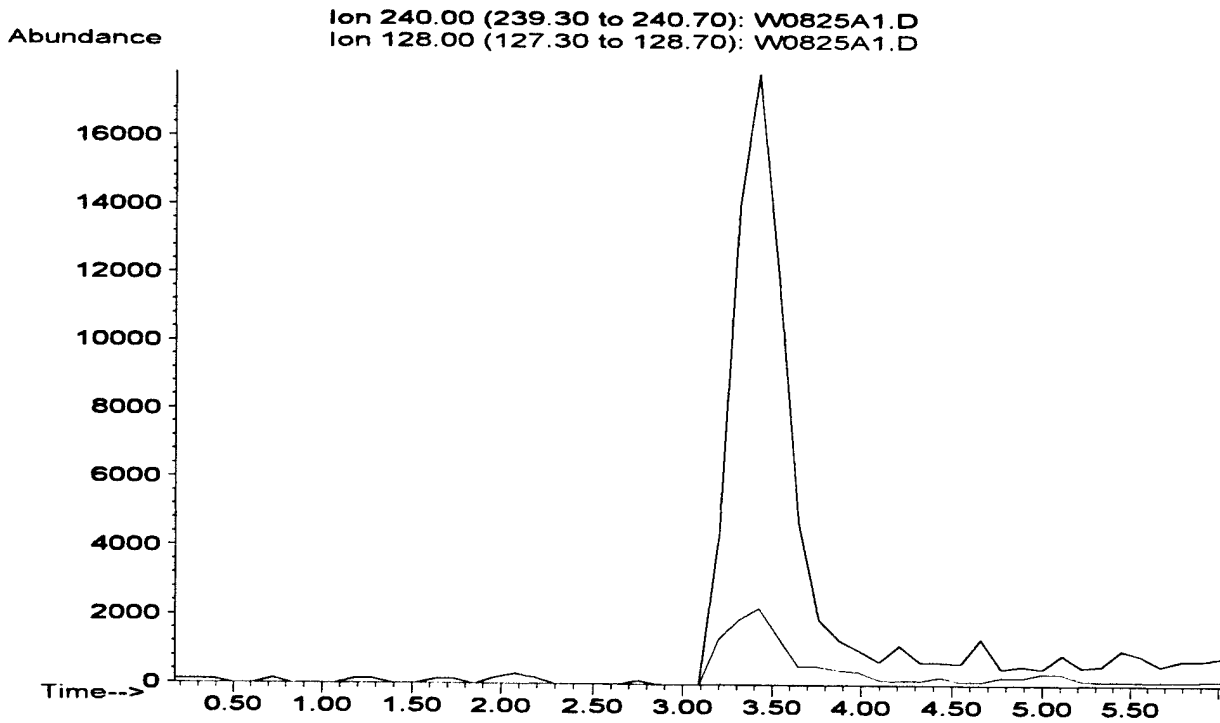
The pH of the aqueous phase can be adjusted. EA-2192 is soluble in aqueous solution at any pH. There are other compounds in the decontamination solution that are more soluble in acidic solution and less soluble in basic solution, so the pH adjustment can be used to modify the chromatography of those 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.

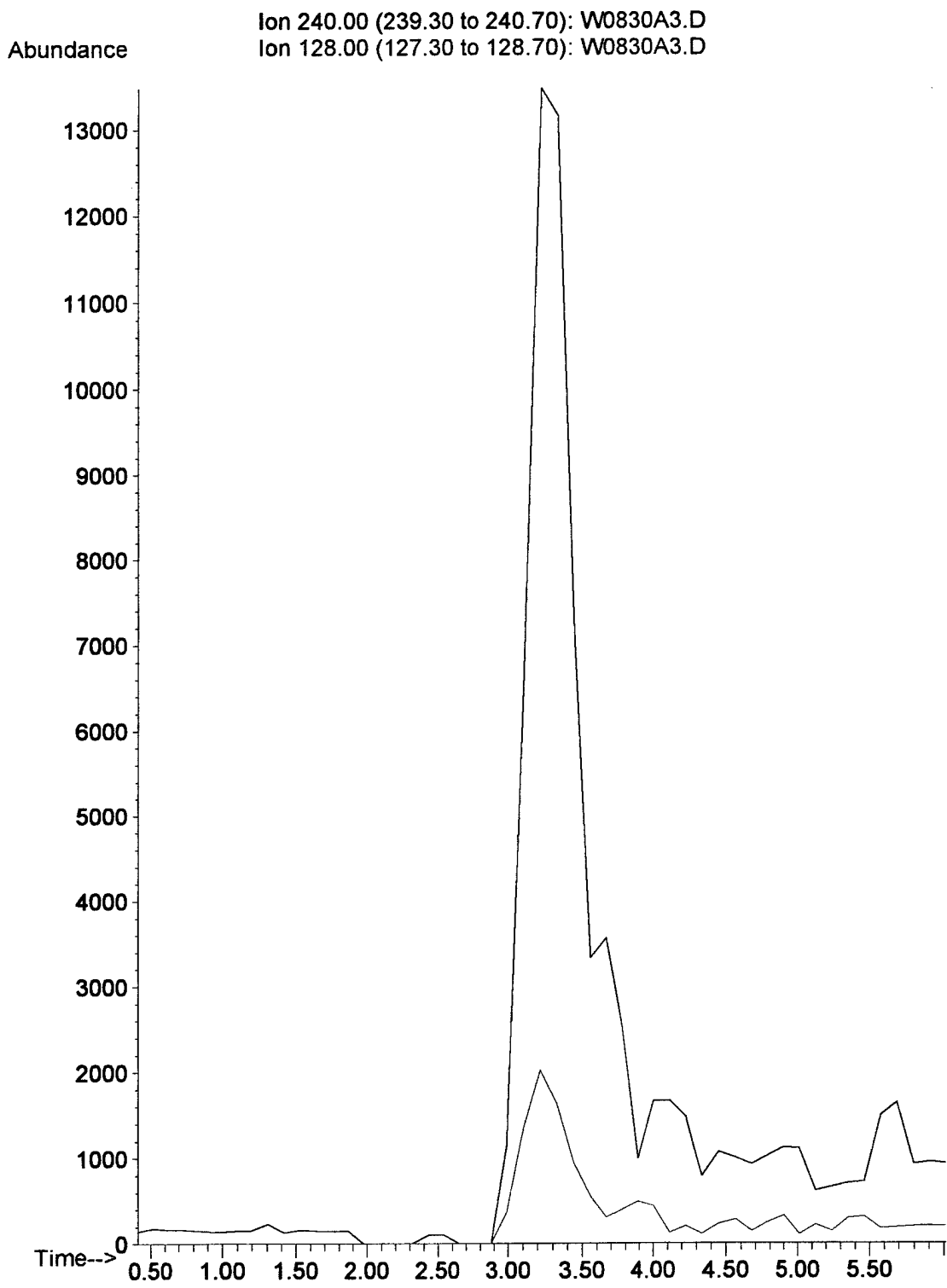
Figure 2-4 shows the chromatogram of a standard of EA-2192 in acidic mobile phase (1% acetic acid in DI water), using ESI. Figure 2-5 shows a chromatogram in basic mobile phase (pH 8, adjusted using ammonium hydroxide)



**Figure 2-3.** Typical LC Chromatogram with a Total Ion Chromatogram (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  $\mu$ L/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  $\mu$ L/min or so.

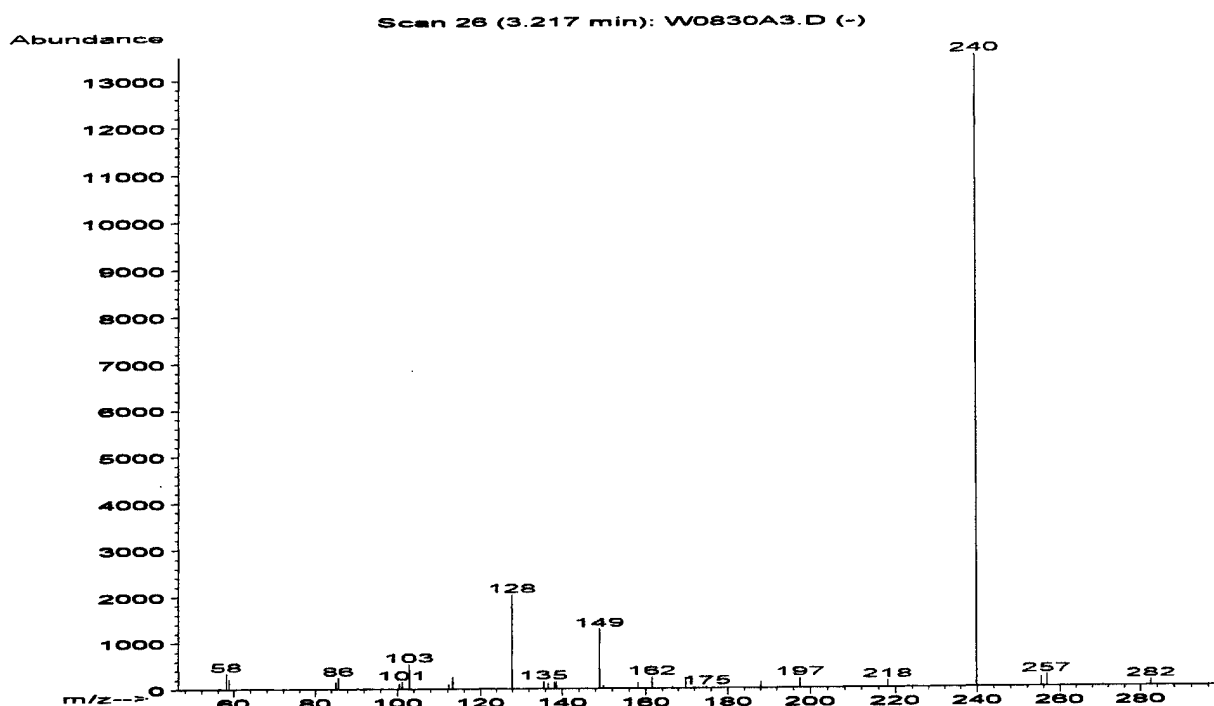
The long-term stability of ESI for the quantitative analysis of decontamination solutions is acceptable. In general, analysis of samples with high salt content can be difficult with atmospheric pressure ionization methods (either ESI or APCI), since the salt tends to build up in the ion source and affect 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.<sup>2</sup> 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.

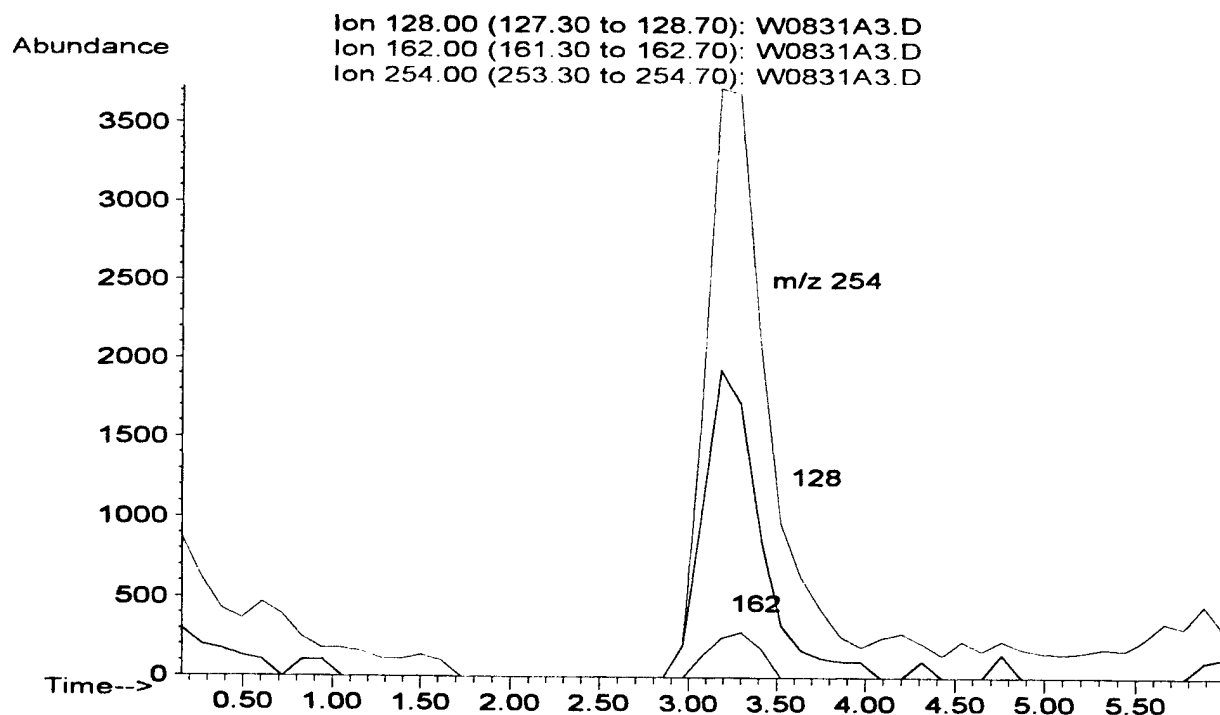


**Figure 2-6.** Positive Ion ESI Mass Spectrum of EA-2192 from a Standard Solution, Using a 5989A Instrument. The  $[M+H]^+$  peak is at  $m/z$  240.

### 2.1.5 Post-Column Derivatization

To improve the performance of APCI, a method of postcolumn derivatization was studied.<sup>2</sup> 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 TPAH [trimethylphenyl ammonium hydroxide] was used. A commercial 0.1 M solution in methanol (Fluka) was added at 5-10  $\mu\text{L}/\text{min}$  to the LC flow of 250  $\mu\text{L}/\text{min}$ . 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.<sup>3</sup> 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  $\mu\text{L}$  quantity of sample was pulled into the injector loop from an autosampler vial, followed by 100-150 $\mu\text{L}$  of basic water, and then 5-10  $\mu\text{L}$  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.

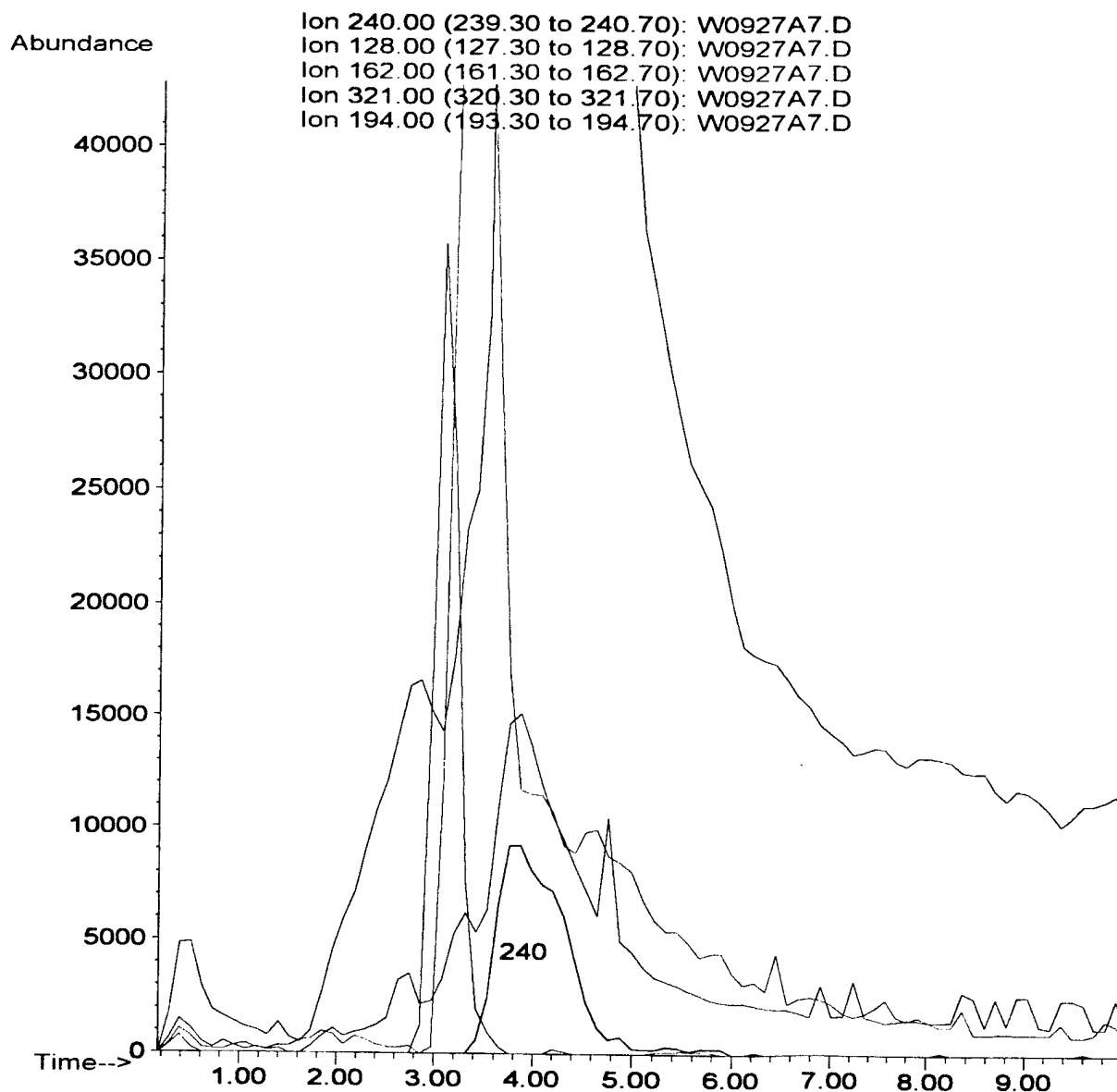
This technique improved the shape of the chromatographic peak significantly. Figure 2-8 shows the chromatograph of EA-2192 which is eluted off the column using the syringe programming method. This approach still requires validation work, but it appears to greatly improve the chromatography of EA-2192 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  $\mu\text{L}$ , 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 preferred 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           |

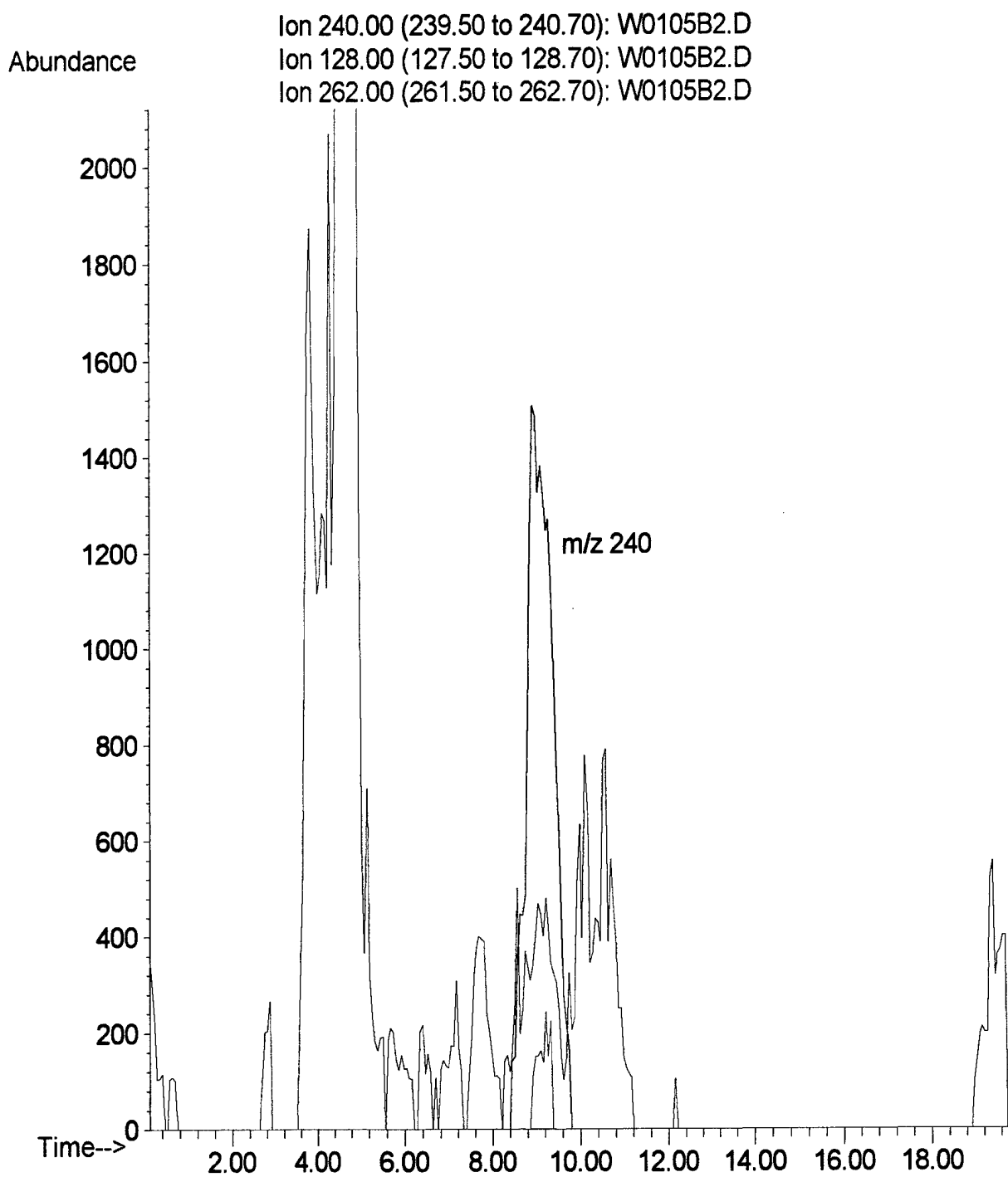
#### 2.1.9.1 Extraction from MMD Hydrolysate

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/H<sub>2</sub>O/MeOH before injection of 10  $\mu$ L of the diluted sample on the LC/MS. Higher injection volumes, even of the diluted sample, tend to degrade the chromatographic resolution.

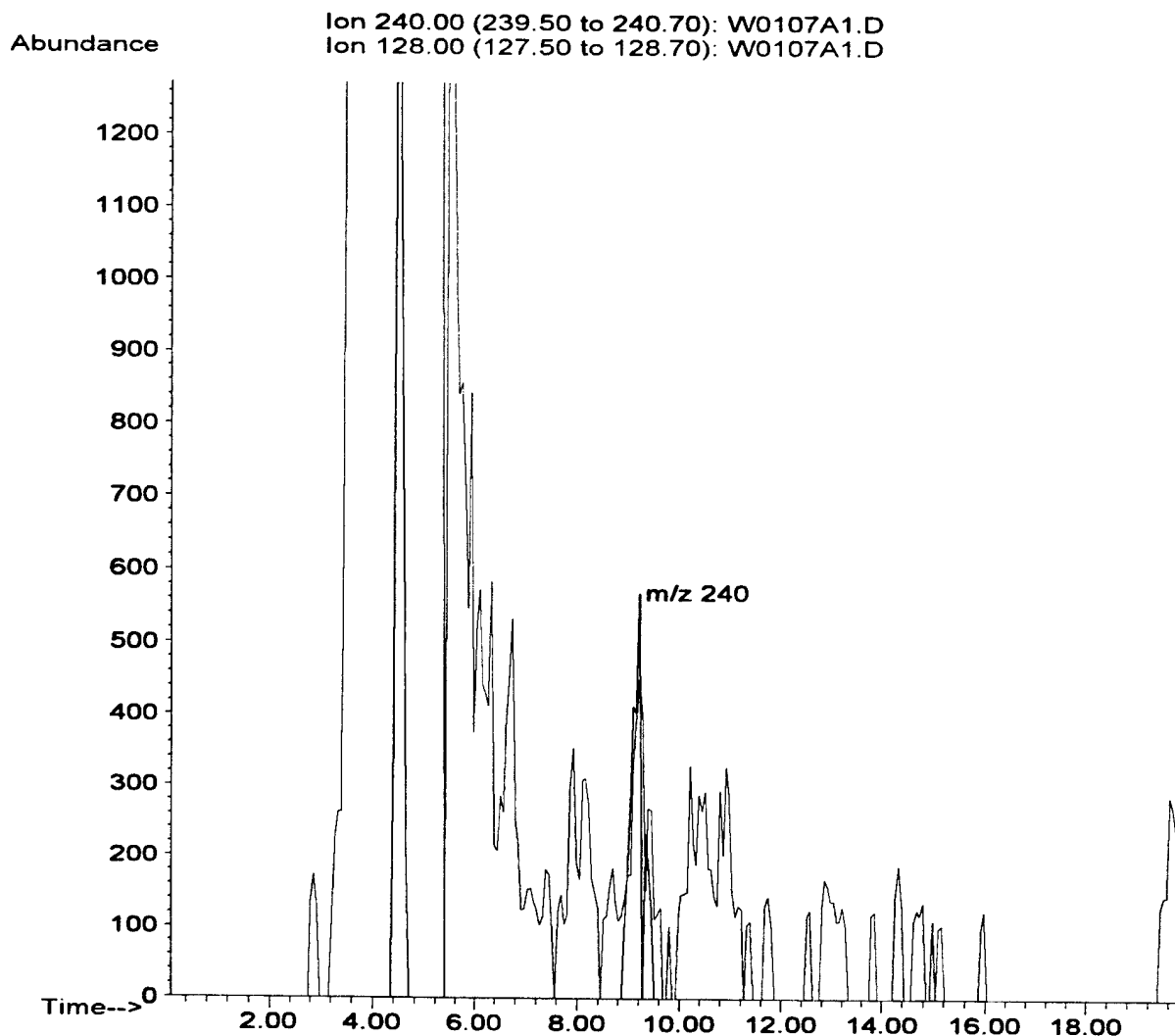
This spiked hydrolysate sample was then extracted with methylene chloride or chloroform. EA-2192 was observed in the extract after the hydrolysate solution was saturated with NaCl. A chromatogram of the extract is shown in Figure 2-10. The  $m/z$  240 peak can be identified, but it is weak and not strongly 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.<sup>1,4</sup> 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.

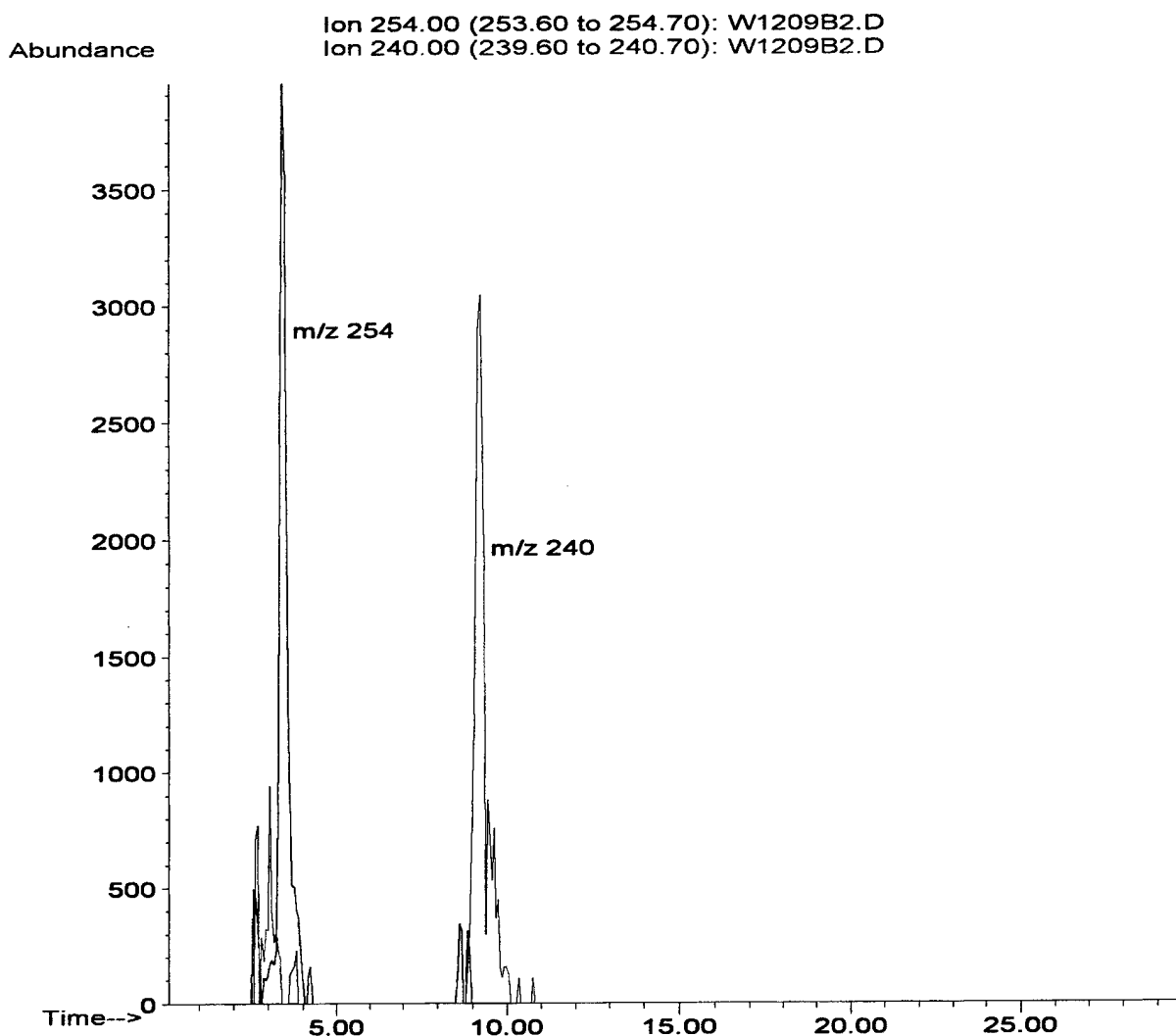


**Figure 2-9.** LC/MS Chromatogram of MMD Hydrolysate Spiked with 40 ppm EA-2192. The pH was adjusted to pH1, and the sample was diluted by about 1:20 in ACN/H<sub>2</sub>O/MeOH before injection on the LC/MS.



**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  $\mu$ L) 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  $\mu$ L 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  $\mu$ g/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  $\mu$ L of the standard give a spike of 0.1  $\mu$ g 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  $\mu$ L acetonitrile, then 3  $\times$  200  $\mu$ L 95% DI water.
2. Pipet 300  $\mu$ L of diluted, acidified decontamination solution (spiked if necessary) on cartridge.
3. Elute liquid to waste.
4. Pipet 300  $\mu$ L of 95% DI water, 5% acetonitrile on cartridge. Elute to sample A.
5. Pipet 300  $\mu$ L 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 $^{31}\text{P}$ -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  $^{31}\text{P}$ -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  $14 \pm 7$   $\mu\text{g/g}$  of EA-2192.

**Table 2-3: Quantitative  $^{31}\text{P}$ -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       | $1.70 \pm 0.02$                     |
| O-(2-amino)ethyl methylphosphonic acid | AEMPA        | NA <sup>1</sup> | $1.55 \pm 0.04$                     |
| Methylphosphonic acid                  | MPA          | 993-13-5        | $0.34 \pm 0.01$                     |

<sup>1</sup> 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  $^1\text{H}$ ,  $^{31}\text{P}$ , and  $^{13}\text{C}$  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  $\mu$ sec, 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  $\text{CDCl}_3$ . The signal-to-noise ratio (SNR) was measured after tuning and shimming the instrument. The SNR should exceed 125:1 for the  $^1\text{H}$  NMR spectrum, although this target may be modified based on past performance of the instrument. A 0.1% solution of triphenylphosphate in  $\text{D}_2\text{O}$  was used to determine the instrument performance of the QNP probe. The SNR should exceed 30:1 for the  $^{31}\text{P}$  NMR spectrum.

A standard of EA-2192 was prepared by placing 600  $\mu\text{L}$  of  $\text{D}_2\text{O}$  into a vial, then adding 38  $\mu\text{L}$  of 400  $\mu\text{g}/\text{ml}$  EA 2192 stock solution (stock solution prepared in 2-propanol), and 36  $\mu\text{L}$  of 2-propanol. The sample was then mixed, and transferred to a 5 mm NMR tube. This standard is 25  $\mu\text{g}/\text{ml}$  EA-2192, and contains 12% (v/v) of 2-propanol.

The unspiked neutralent sample was prepared by adding 600  $\mu\text{L}$  of neutralent into a vial, adding 2 drops of  $\text{D}_2\text{O}$  as a lock solvent, then adding 75  $\mu\text{L}$  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  $\mu\text{L}$  of neutralent into a vial, adding 2 drops of  $\text{D}_2\text{O}$  as a lock solvent, then adding 400  $\mu\text{g}/\text{ml}$  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  $^1\text{H}$  and  $^{31}\text{P}$  NMR spectra.

The spectra obtained from the analysis of a 25  $\mu\text{g/ml}$  EA-2192 standard in  $\text{D}_2\text{O}$  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.

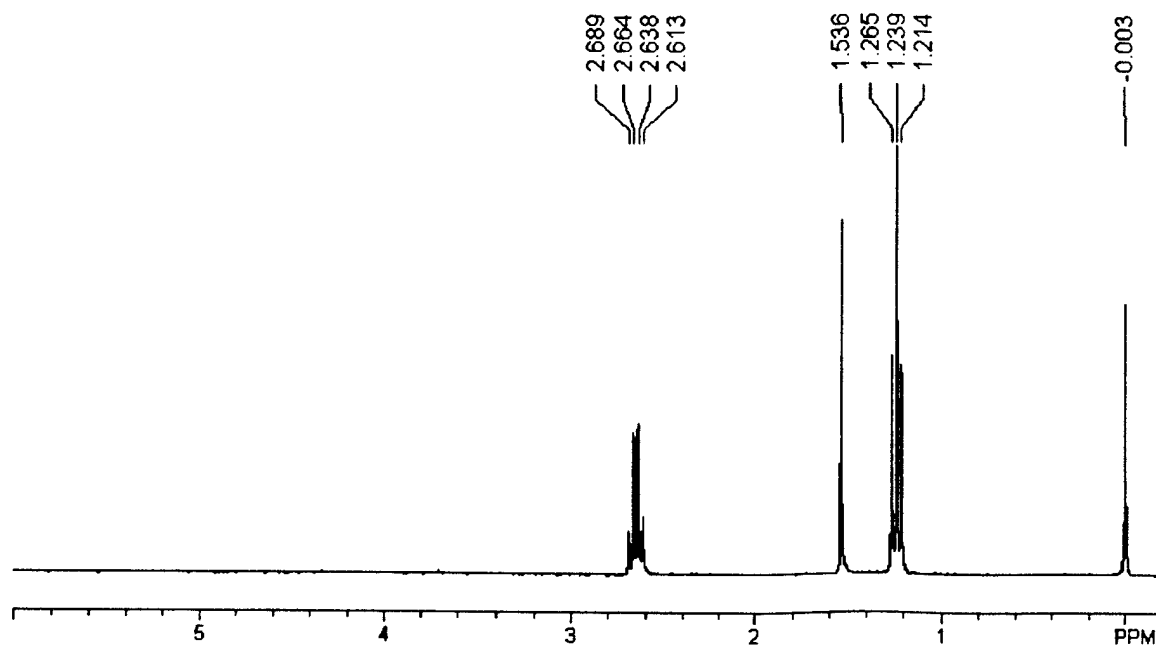
The spectra obtained from the analysis of unspiked neutralent is attached as Figure 2-15. There is a peak at a chemical shift of 43.312 ppm, with an SNR of approximately 13:1. This is the only peak in the chemical shift region where EA-2192 is observed in the standard solution. The spectra obtained from the analysis of neutralent spiked to 50  $\mu\text{g/ml}$  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  $\mu\text{g/ml}$ , 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  $\mu\text{g/ml}$  EA-2192 standard analyzed by  $^{31}\text{P}$  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  $\mu\text{g/ml}$ .

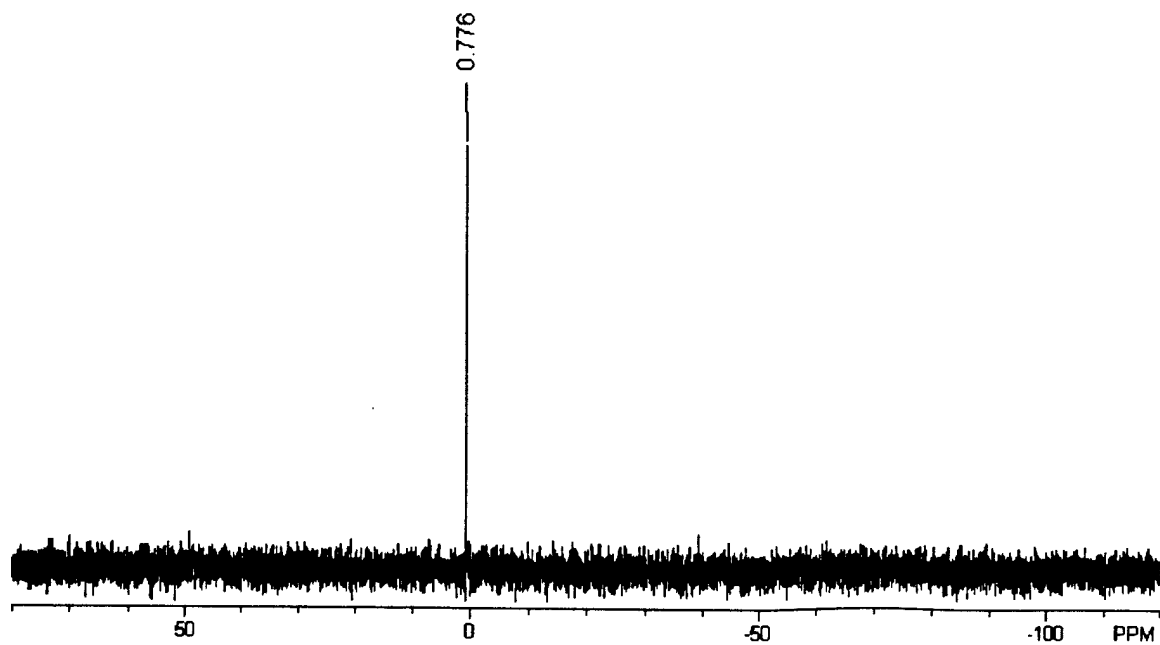
The established  $^{31}\text{P}$ -NMR method used to analyze this sample matrix is not suitable for quantitative analysis of EA-2192 in this sample matrix at the target action level of 50  $\mu\text{g/ml}$ . The technique of NMR is best suited to quantitation of bulk (>0.1 wt%) components, and to structural 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

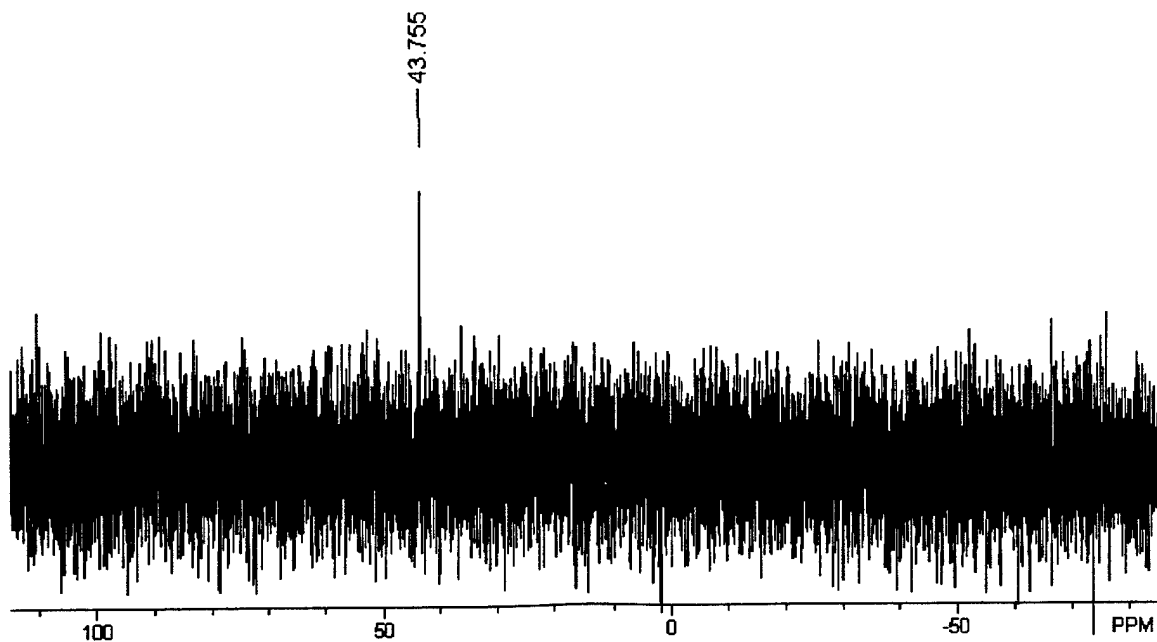
One of the problems with the previous NMR study was that old decontamination solution was used. In the solution, many low concentration phosphorus containing compounds were formed over time by slow reactions. These extra compounds made it difficult to positively identify the EA-2192 peak.



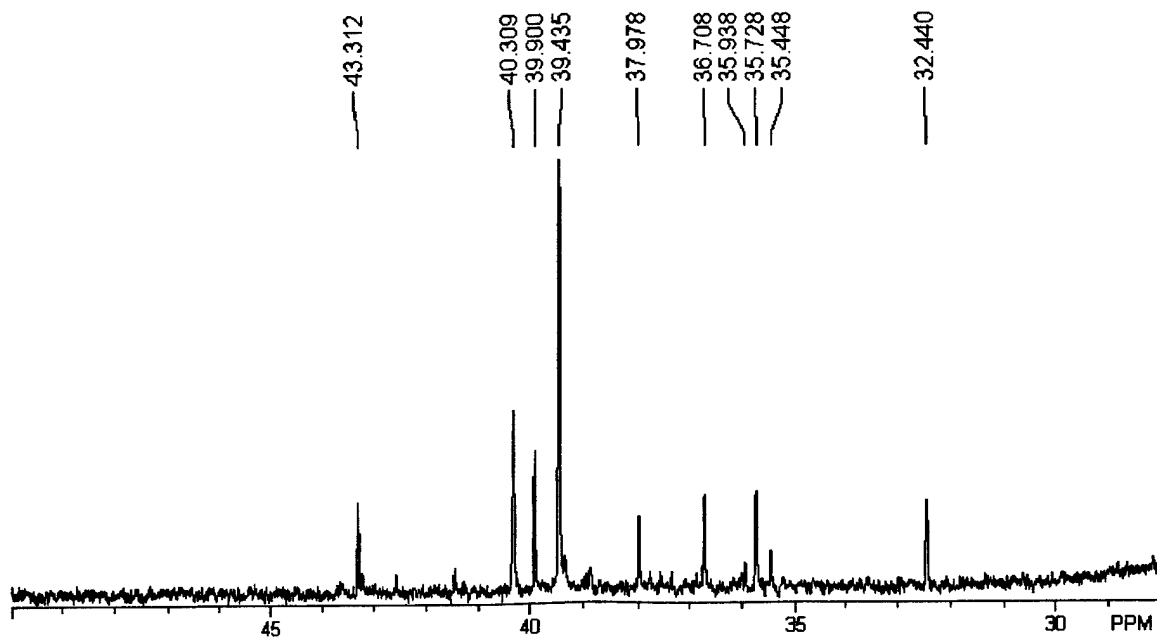
**Figure 2-12.**  $^1\text{H}$  NMR Spectrum of the 0.1% Ethylbenzene Performance Check Standard. The SNR was determined to be 141:1.



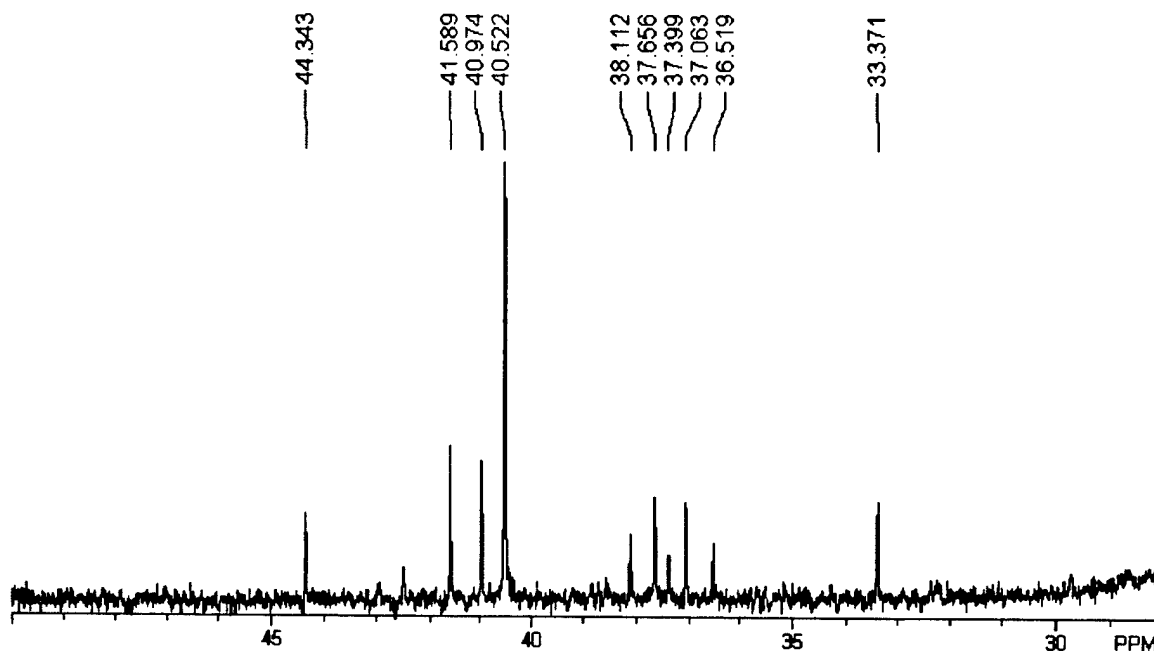
**Figure 2-13.**  $^{31}\text{P}$  NMR Spectrum of the 0.1% Triphenylphosphate Performance Check Standard. The SNR was determined to be 33:1.



**Figure 2-14.**  $^{31}\text{P}$  NMR Spectrum of the 25  $\mu\text{g/ml}$  EA-2192 Standard in  $\text{D}_2\text{O}$ . The SNR was determined to be 4:1.



**Figure 2-15.**  $^{31}\text{P}$  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.



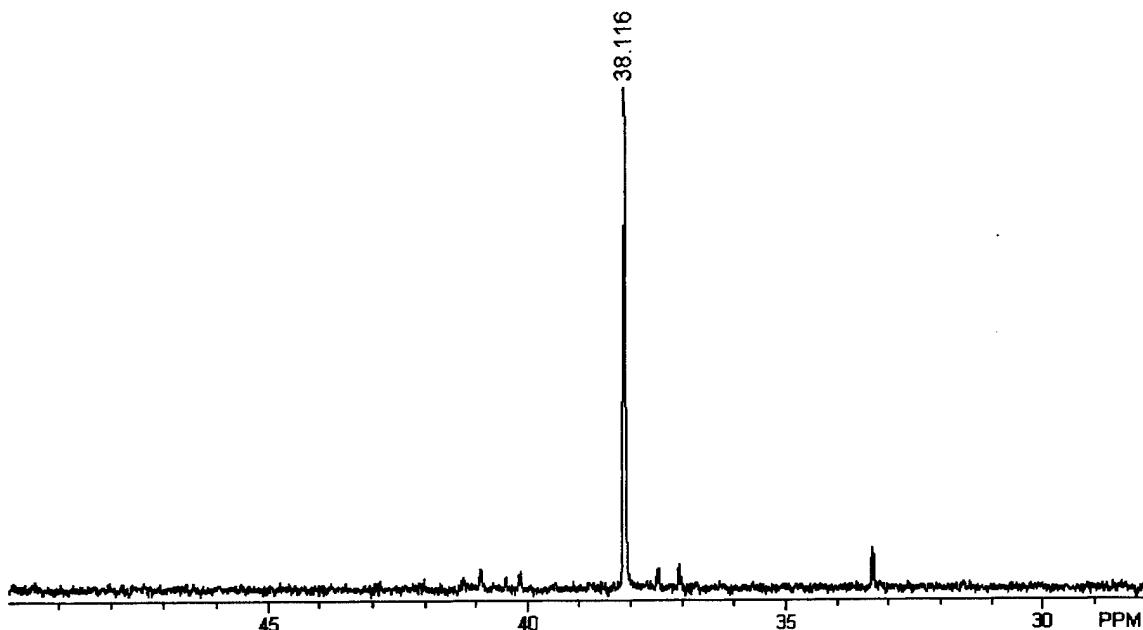
**Figure 2-16.** <sup>31</sup>P NMR Spectrum of the Neutralent Spiked to 50 µg/ml EA-2192. The peak (at a shift of 44.343 ppm) in the chemical shift region of EA-2192 was determined to have an SNR of 13:1. There are shifts in the peaks that are between 35 to 40 ppm.

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 than 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.

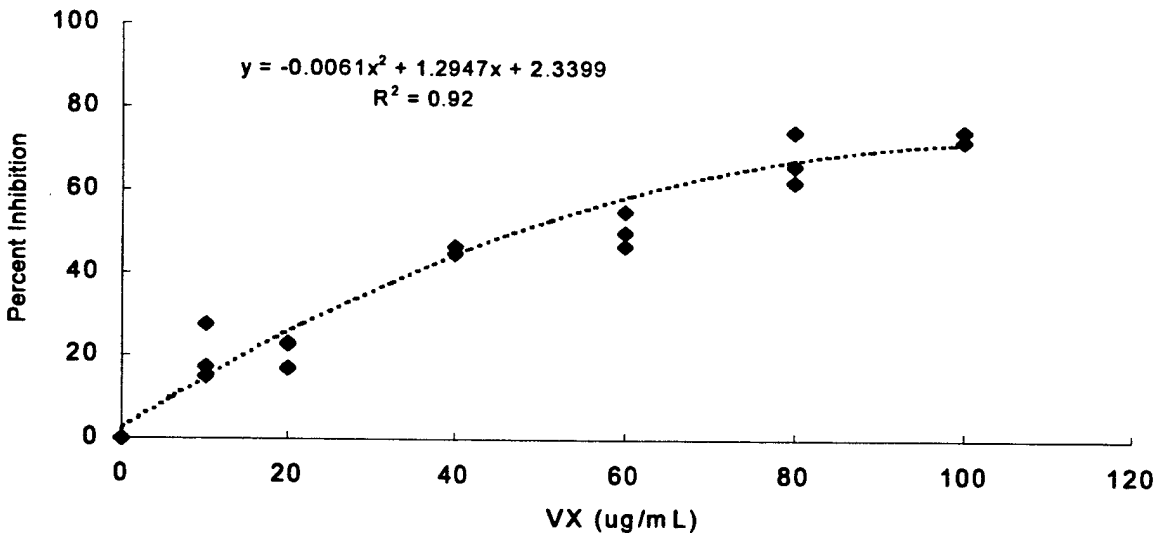


**Figure 2-17.** Expanded Section of a  $^{31}\text{P}$  NMR Spectrum of the New MMD Reaction Matrix Neutralent Solution Spiked to 100  $\mu\text{g}/\text{ml}$  EA-2192.

Five different reactor runs (MEA/NaOH chemistry) were analyzed for residual EA-2192 approximately one month after the reactions took place. 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  $\mu\text{g}/\text{g}$  (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  $\mu\text{g}/\text{g}$ ) in the VX/caustic hydrolysates produced in the MMD-1 program.<sup>5,6</sup> 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  $\mu\text{g}/\text{g}$  (ppm) VX. However, EA-2192, up to 100  $\mu\text{g}/\text{g}$  (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  $\mu\text{g}/\text{g}$  (ppm) VX would be successful.



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

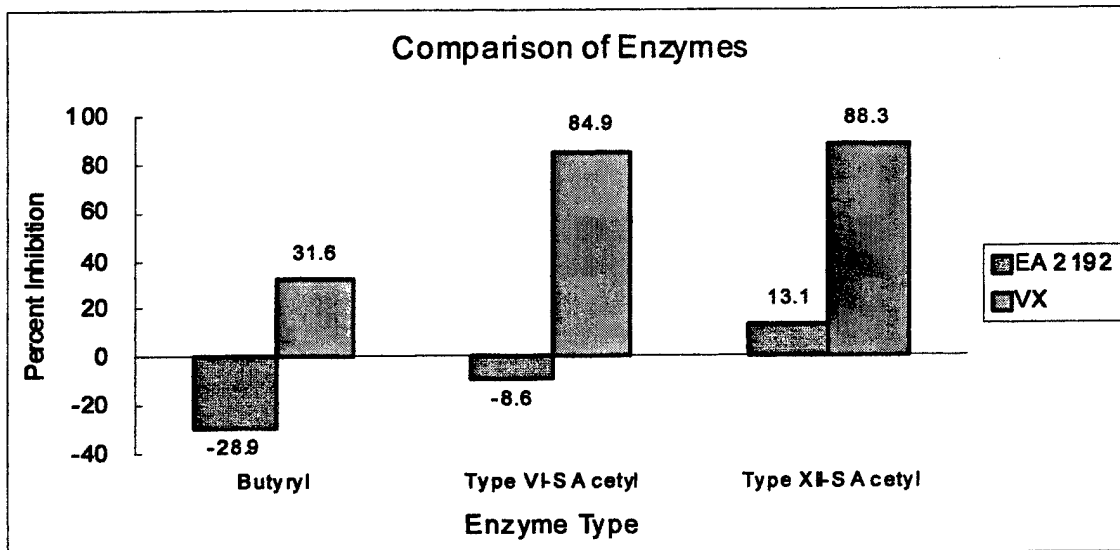
An experiment was conducted to compare three different cholinesterase enzymes, to determine the way both VX and EA-2192 inhibited them. In all cases, the spike level was 50 µg/g (ppm) for both VX and EA 2192. These results are illustrated in Figure 2-19. The negative percent inhibition values are indicative 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**

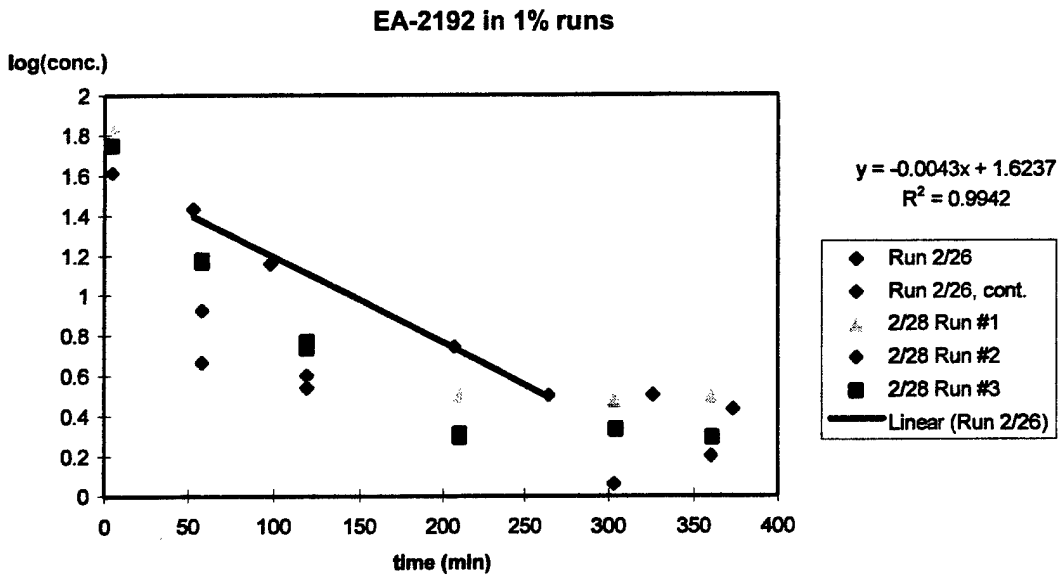
Figure 3-1 shows the data for the log of the concentration of EA-2192 as a function of reaction time for the four 1% VX runs.

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  $-0.00572 \pm 0.0010$  (RSD: = 17.7%).

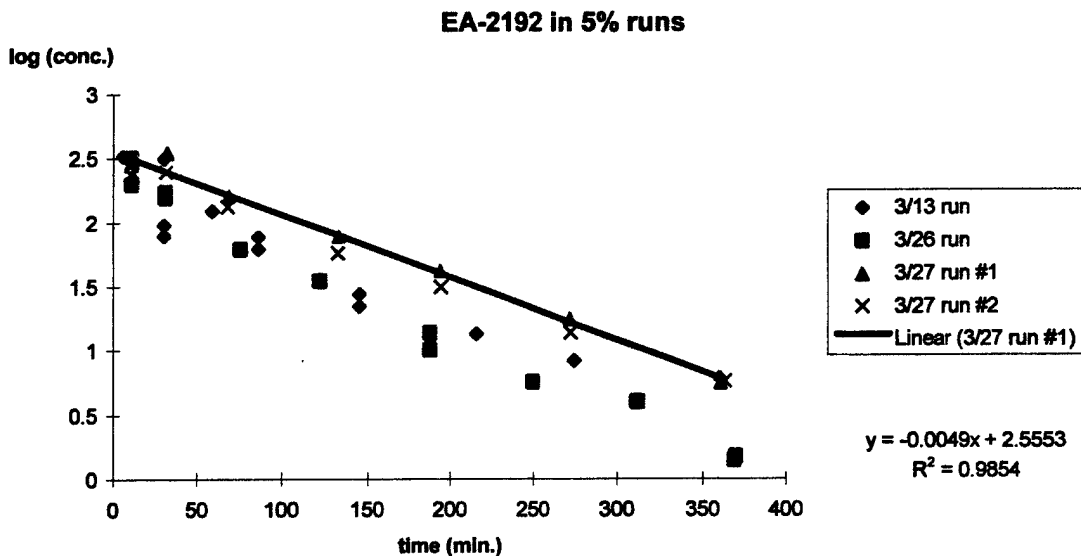
The highest concentration of EA-2192 that was measured for these runs is 70 ppm, as measured 5 min. after mixing the reagents. The EA-2192 arises in part from impurity in the VX, and in part from formation of EA-2192 in the decontamination reaction from the VX. In all cases, the concentration was below 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  $-0.00515 \pm 0.00064$  (%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**

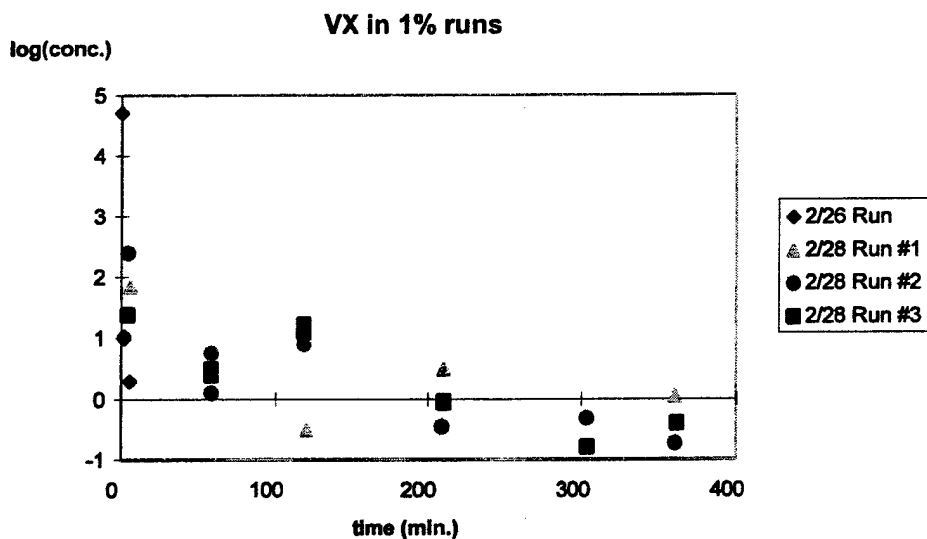
The concentration of VX was analyzed for these runs using the same LC/MS procedure. Most of the VX is destroyed in 5-15 min., which was also observed in the trial runs using the other lot of VX. Because of these preliminary results, the analytical method was not validated for VX detection, although the 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.

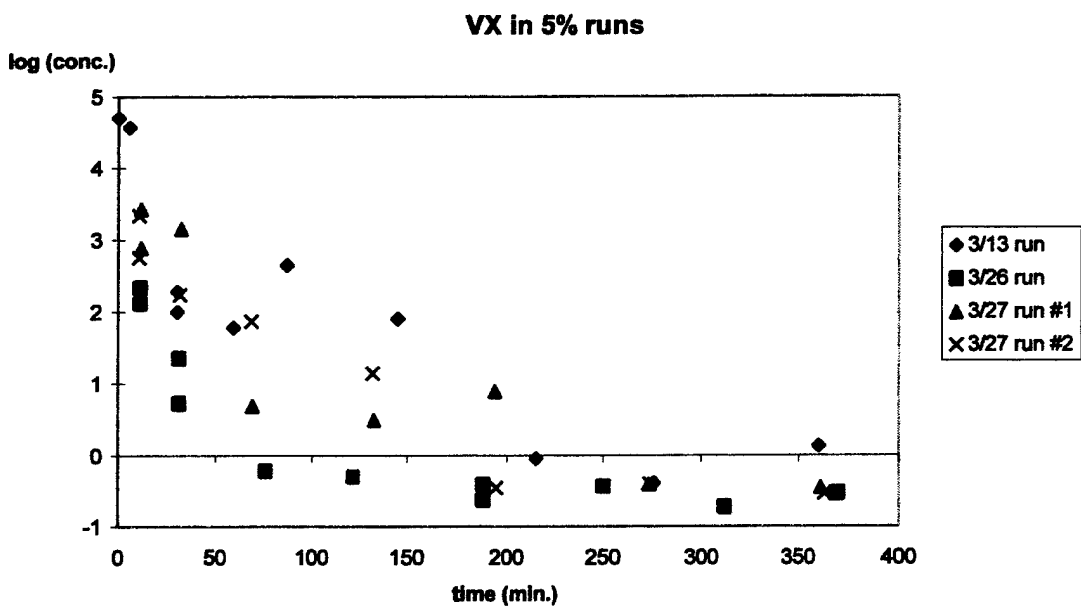
Figure 3-3 shows the log of the concentration of VX for the 1% VX runs. Figure 3-4 shows the log of the concentration of VX for the 5% VX runs.

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.<sup>6</sup> 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**

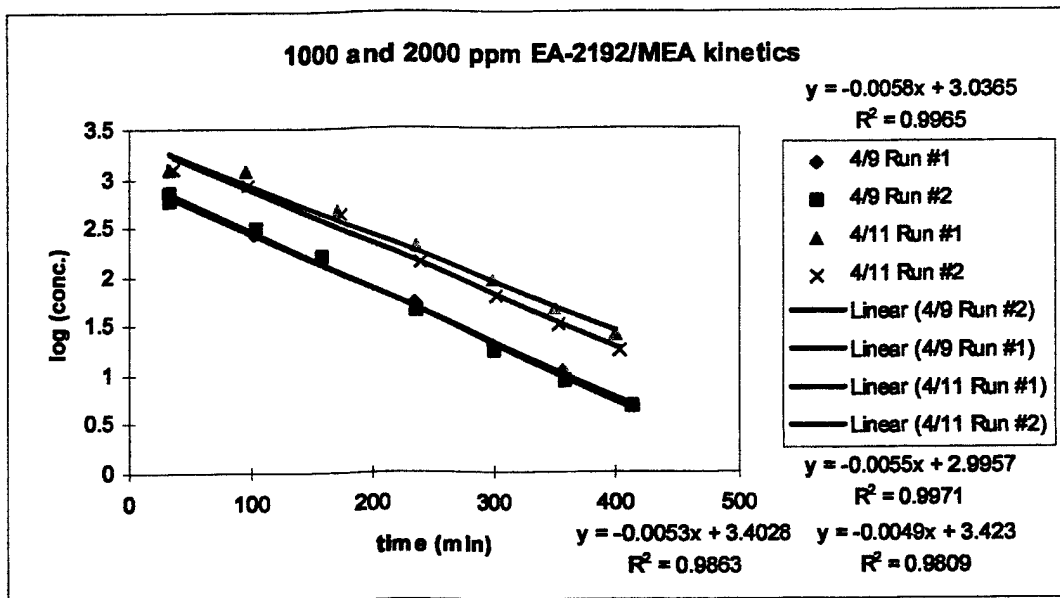
A worst case scenario can be developed as follows. Literature results indicate that if VX is decontaminated with aqueous caustic solution, the VX is converted to 10-20% EA-2192.<sup>7,8</sup> It seems unlikely that munitions that are full of purified EA-2192 would be encountered, so EA-2192 from decontaminated 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  $-0.00537 \pm 0.0038$  (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  $-0.00515 \pm 0.00064$  (%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:

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

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**

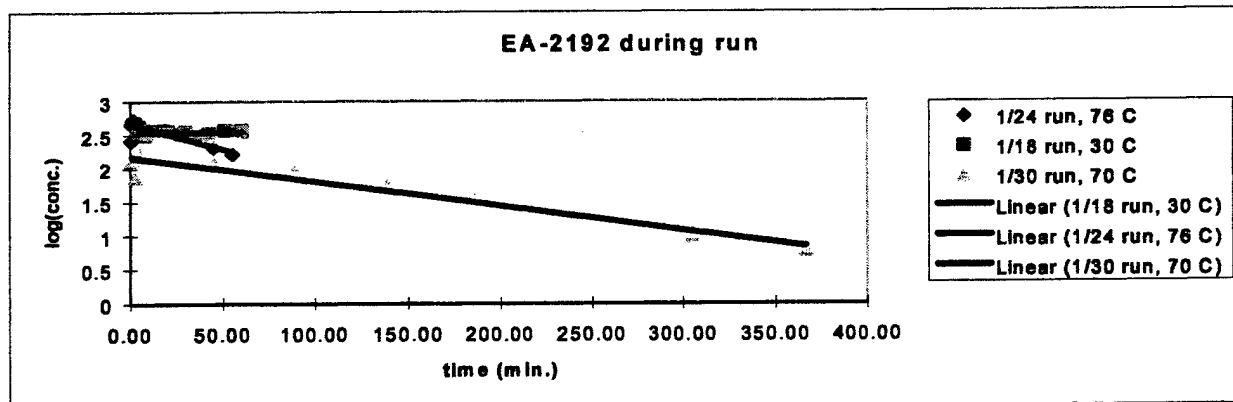
The first preliminary experiments were done to study the kinetics of the decrease in the amount of EA-2192 in MMD decontamination solution as a function of time. Experiments were done at ambient temperature, about 25°C, and at a higher temperature than that specified for the MMD-1 reactor, about 70°C. 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 <sup>31</sup>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.



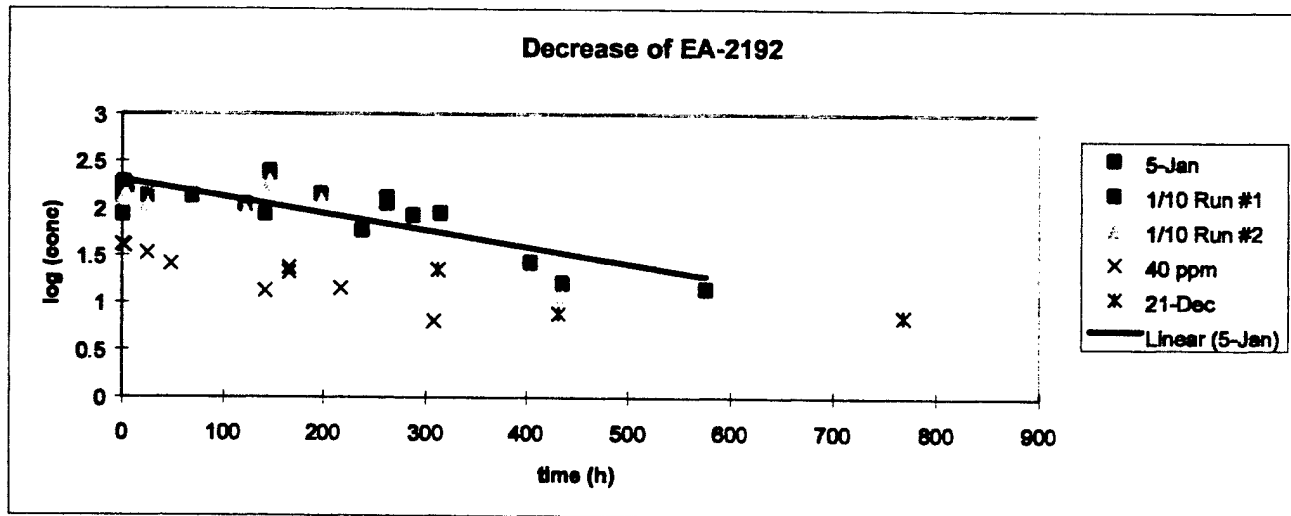
**Figure 3-6.** Log of the EA-2192 Concentration vs. Time, in Minutes. The runs on 1/24 and 1/30 are at elevated temperature, and the run on 1/18 was at room temperature. On the log scale, 50 ppm corresponds to 1.69 on the y-axis.

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.

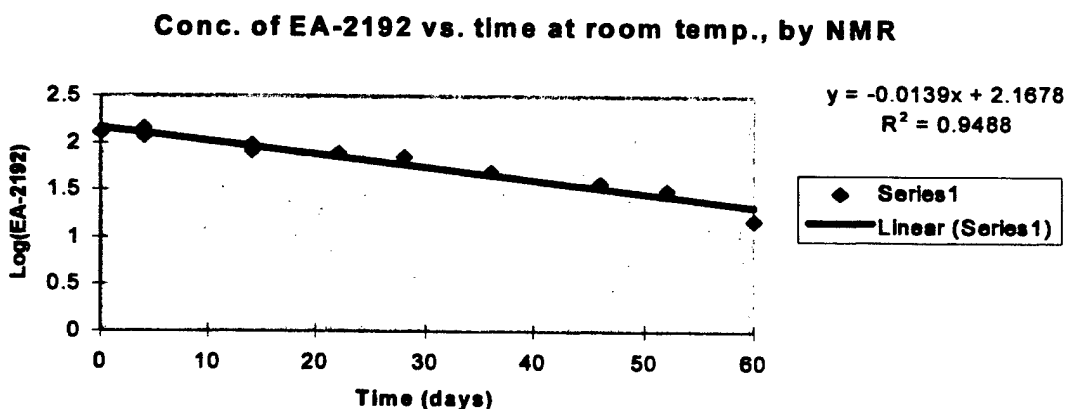
Runs at room temperature were done to determine the stability of EA-2192 in the solutions in ambient storage conditions. At room temperature (about 20-25°C) the decrease was slow, as shown in Figure 3-7. The time required to decrease below 50 ppm was 300-400 hours, or 12-16 days. The data is noisy because 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 <sup>31</sup>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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**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. =  $3.51e-15 * (\text{signal})^2 + 4.47e-8 * \text{signal}$

CCV

| file    | sample. | Signal (240) | calc.conc.<br>(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,<br>linear fit) | found con.<br>(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

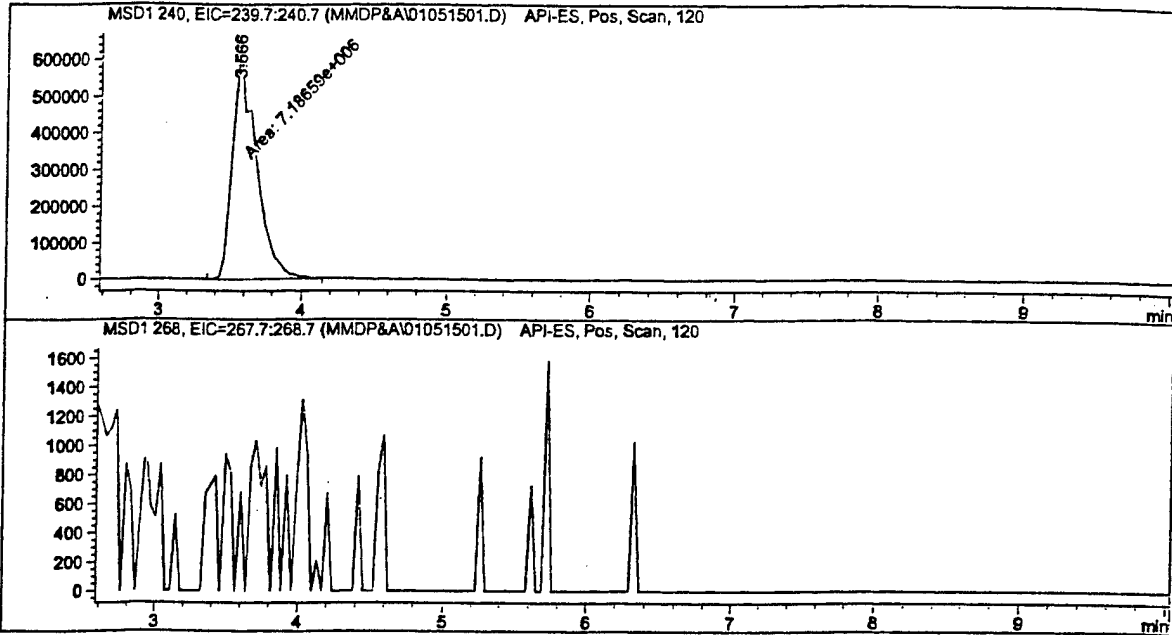
$$\text{conc.} = 3.41\text{e-}15 * (\text{signal})^2 + 4.44\text{e-}8 * \text{signal}$$

CCV

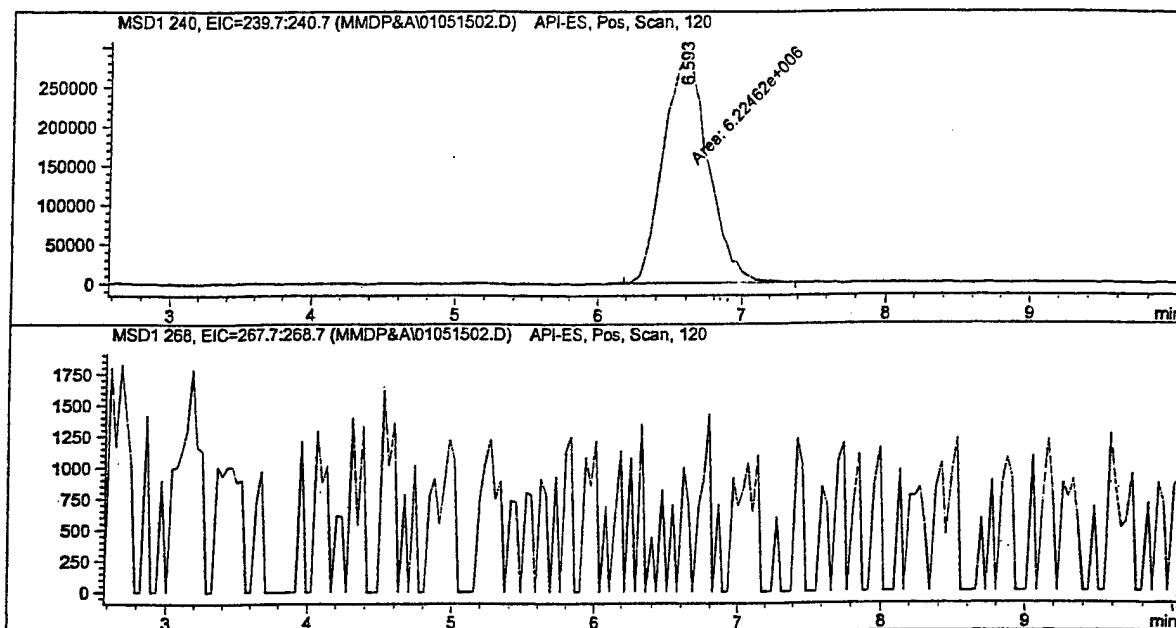
| file    | sample. | Signal (240) | calc.conc.<br>(linear) | calc (poly) |
|---------|---------|--------------|------------------------|-------------|
| 1051601 | 400     | 6.00E+06     | 415.0894               | 0.38916     |
| 1051602 | 400     | 6.21E+06     | 429.1541               | 0.407228  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

(5/16 Run)

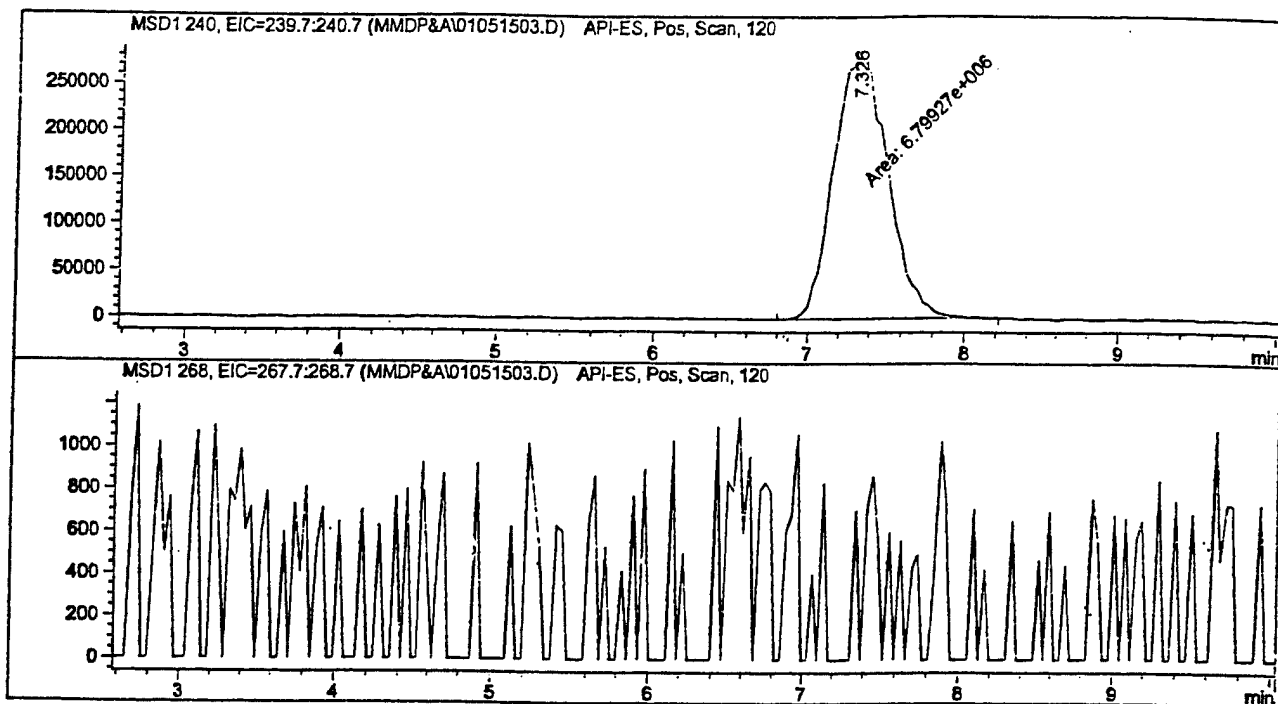
| Filename | Sample ID | signal     | found con.<br>(ppb, linear fit) | found con.<br>(ppm, poly. fit) | Conc. (ppb) | LC r.t   |
|----------|-----------|------------|---------------------------------|--------------------------------|-------------|----------|
| 1051604  | NB114P89A | 8.60E+04   | 19.00103                        | 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    |          |



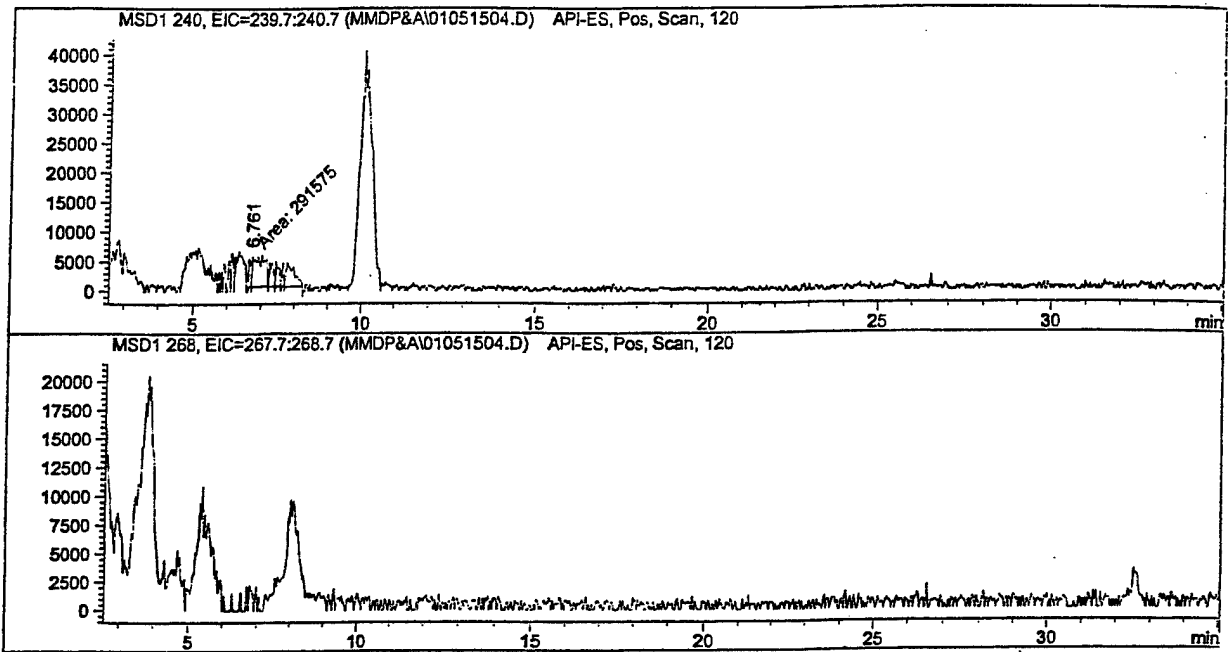
\*\*\* End of Report \*\*\*



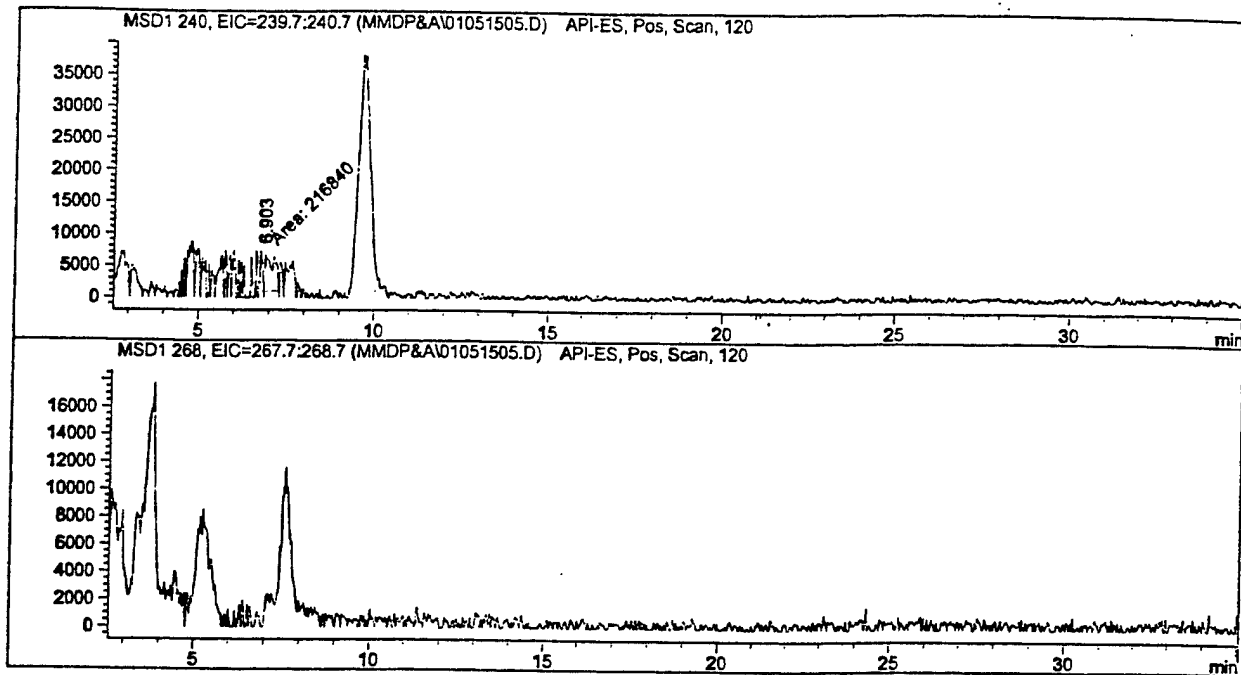
\*\*\* End of Report \*\*\*



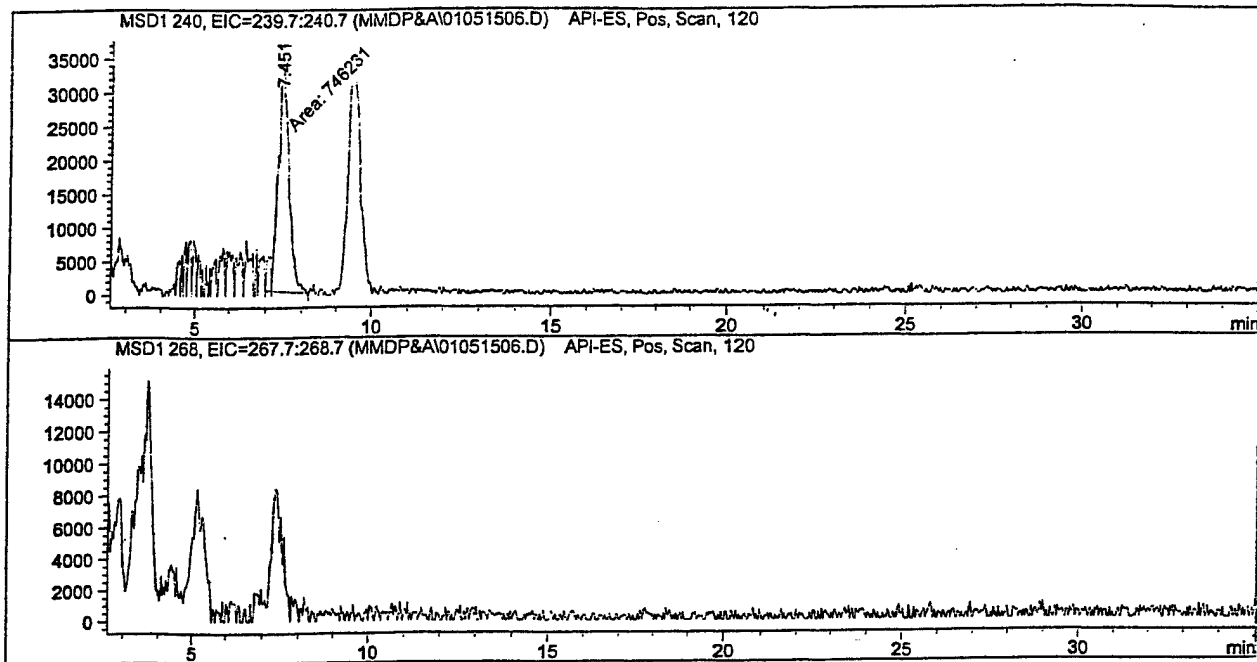
\*\*\* End of Report \*\*\*



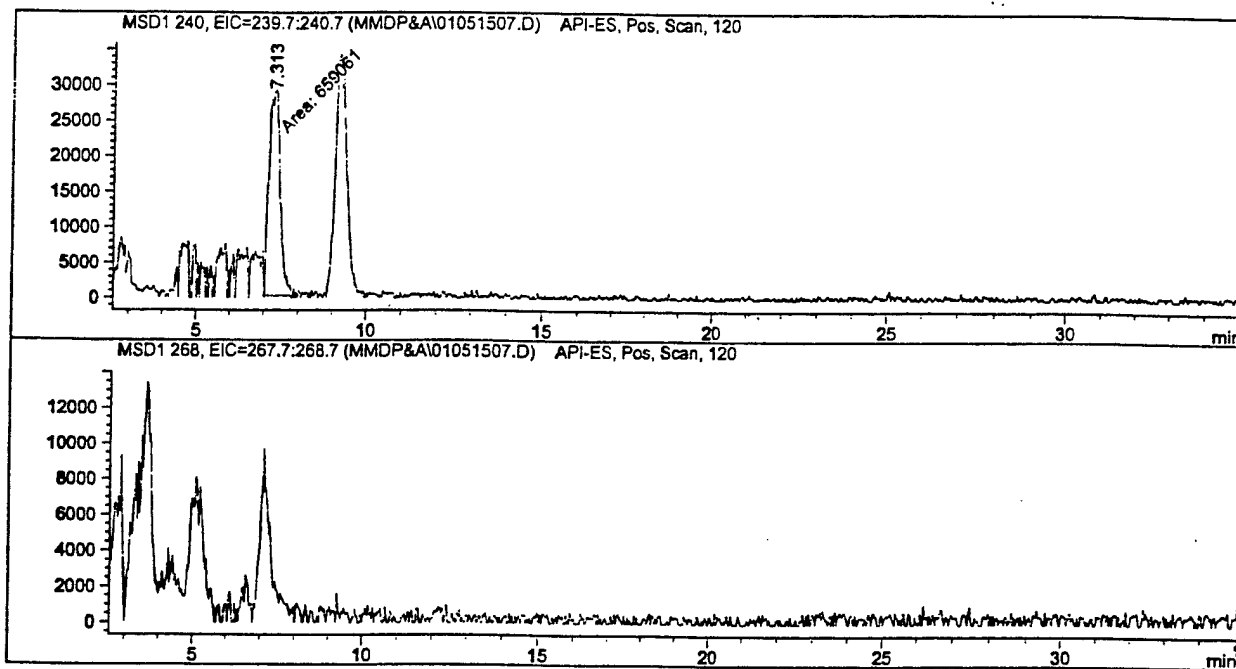
\*\*\* End of Report \*\*\*



\*\*\* End of Report \*\*\*

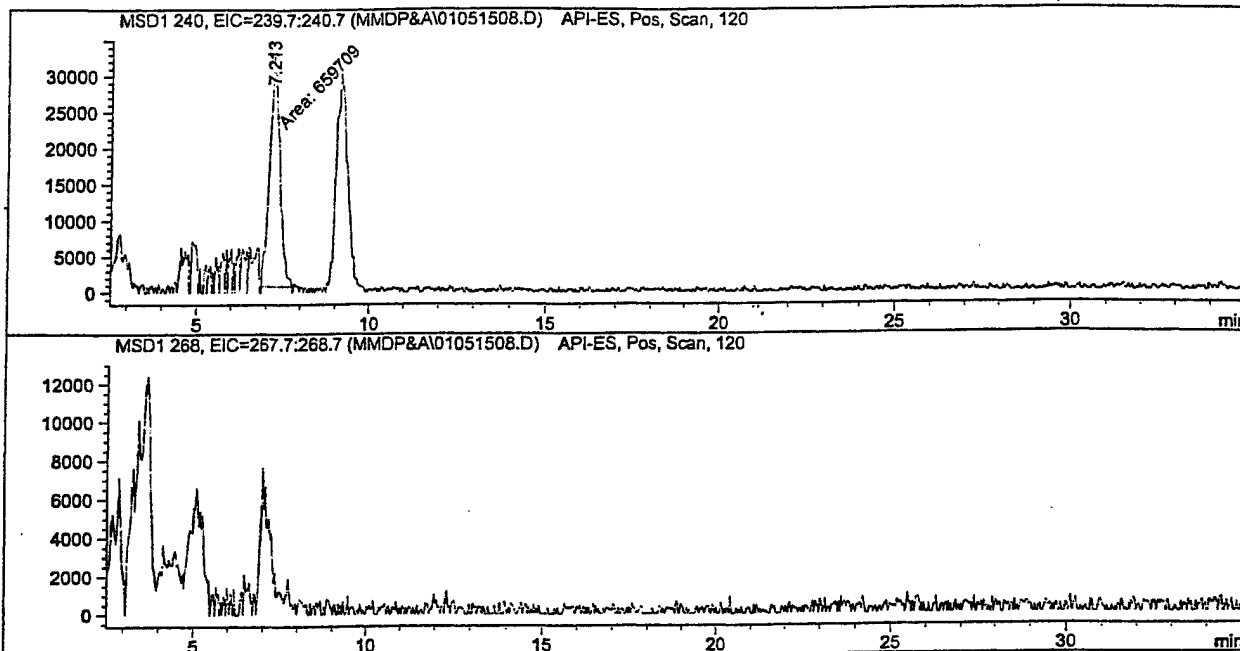


\*\*\* End of Report \*\*\*

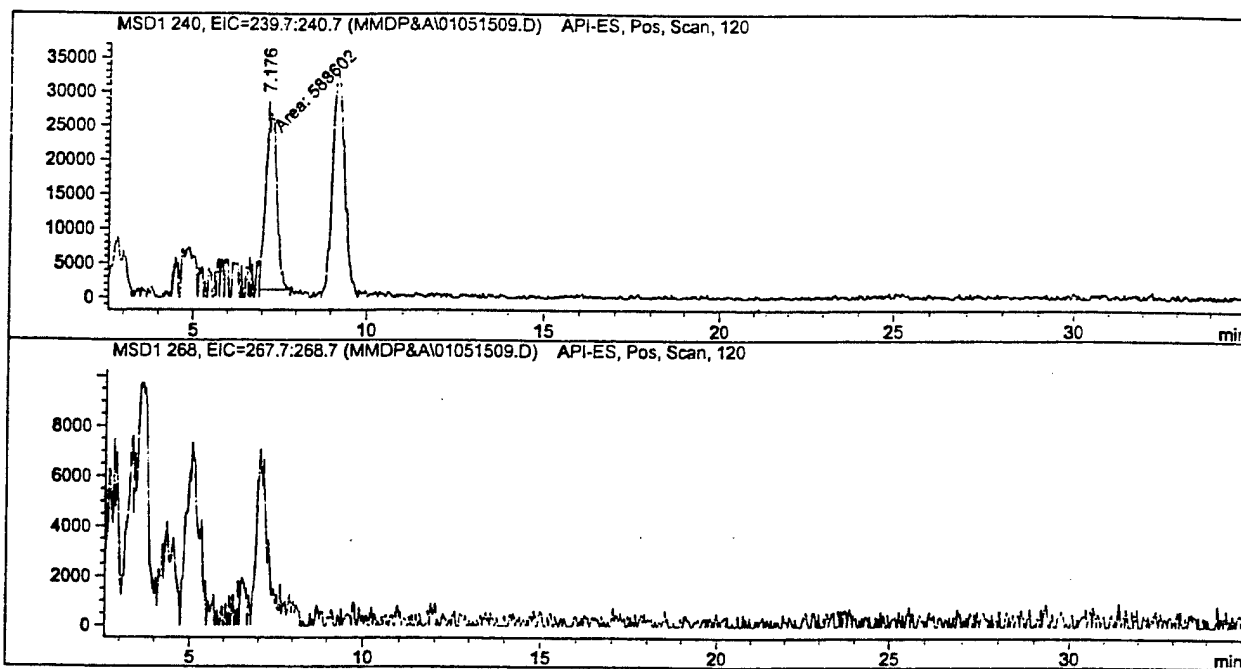


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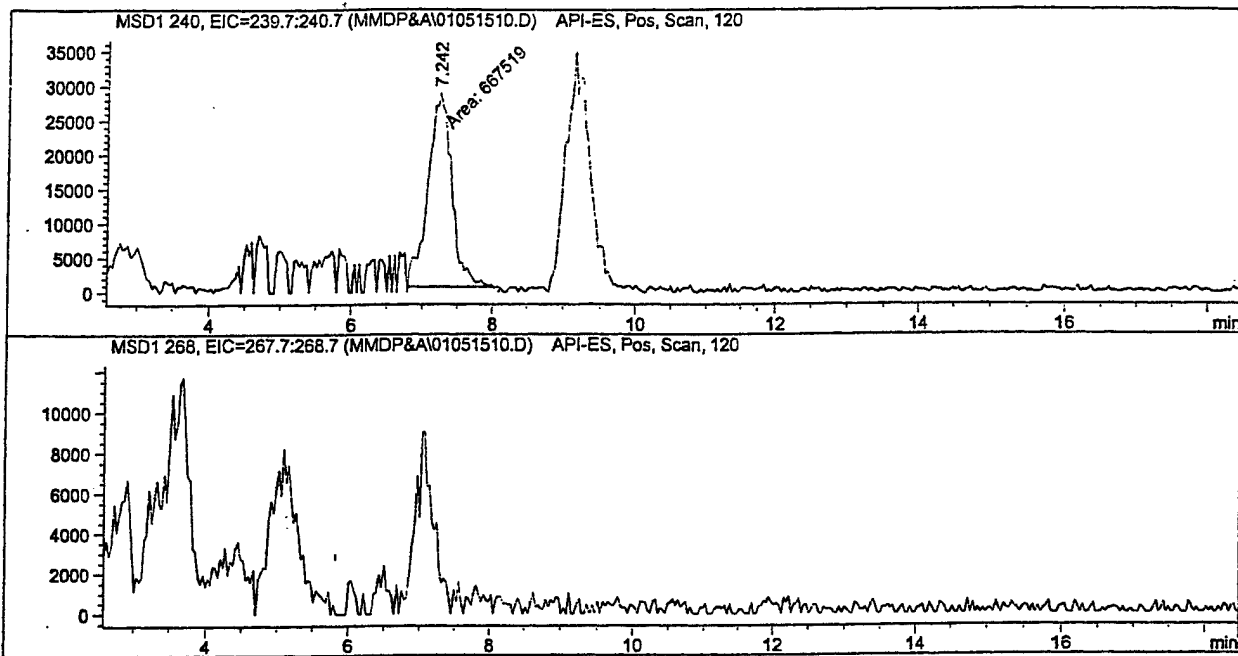




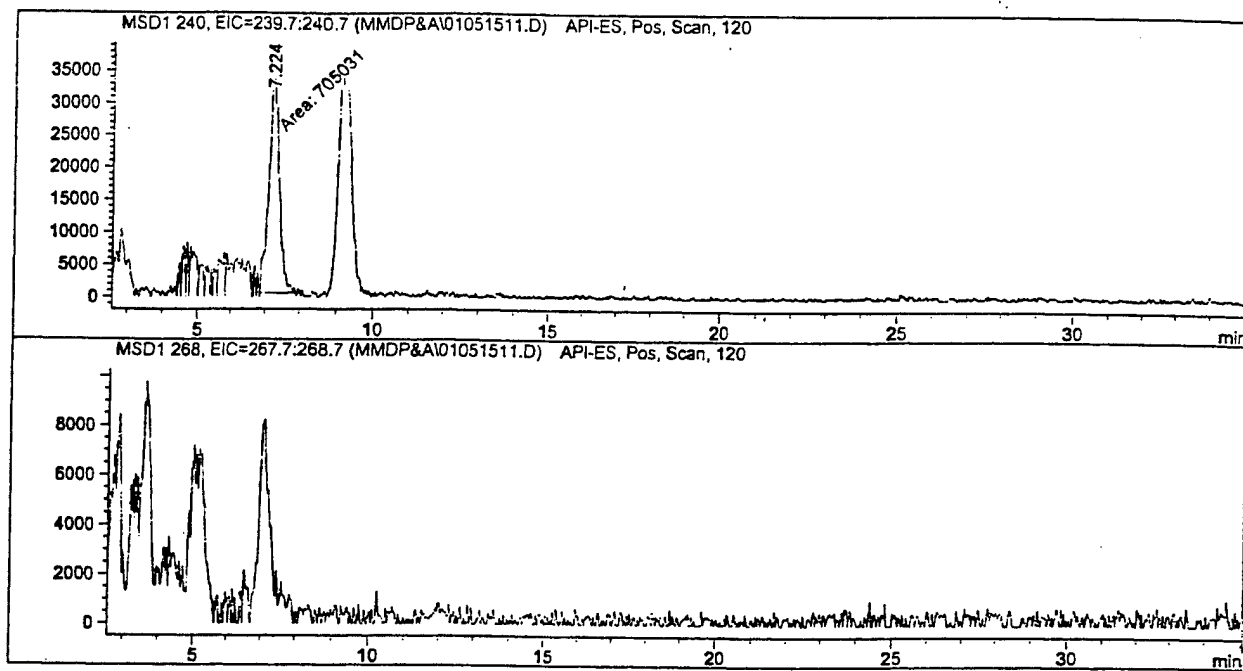
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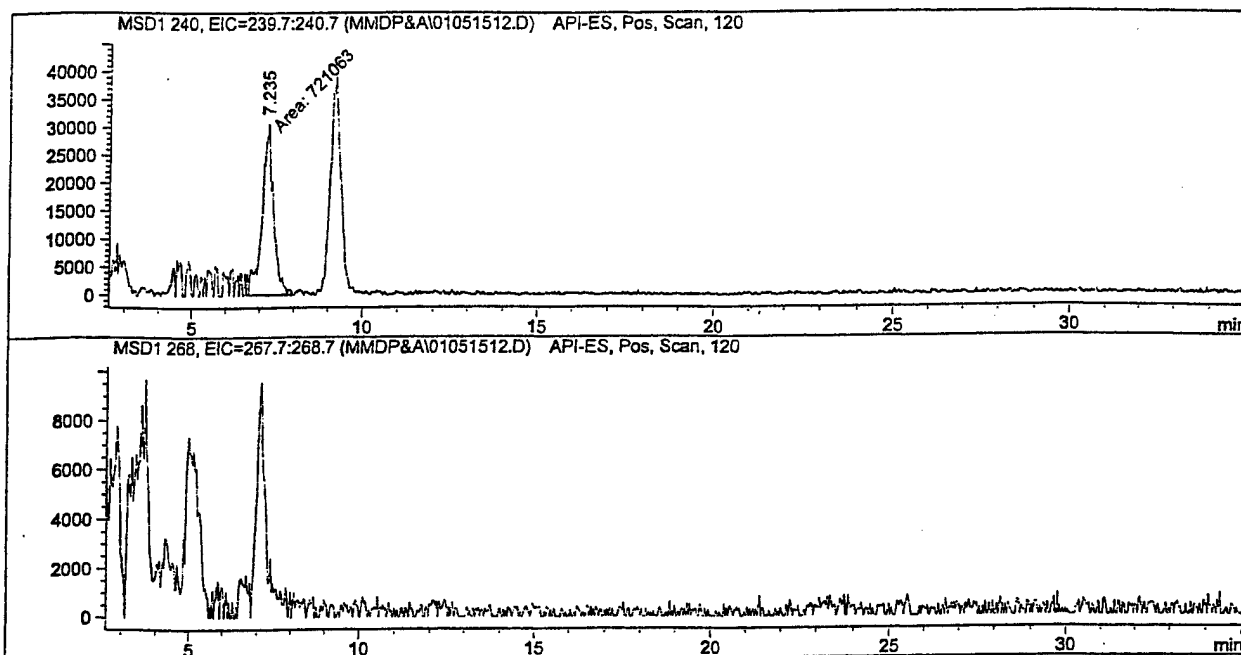
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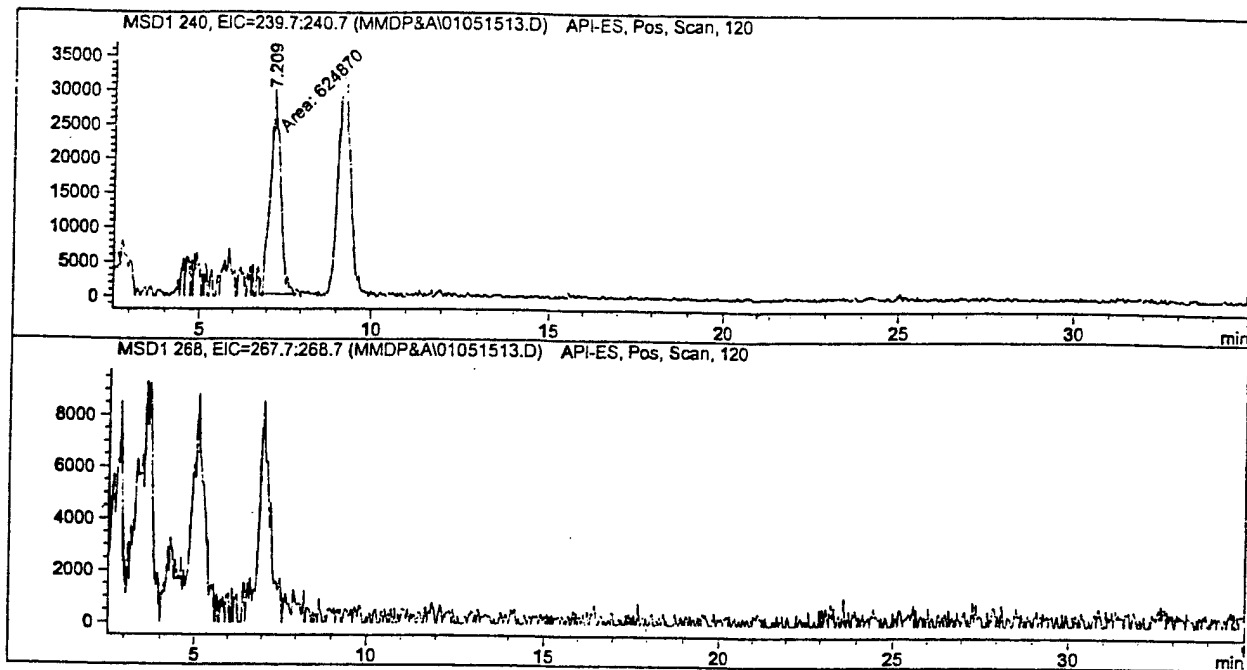
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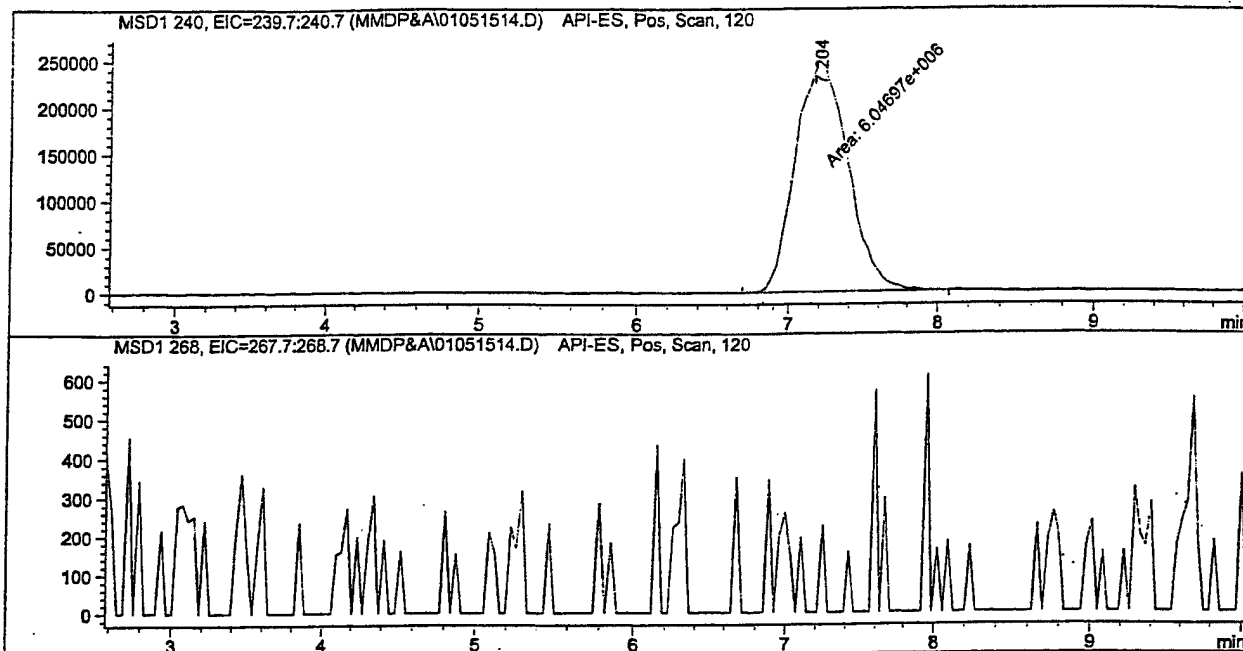
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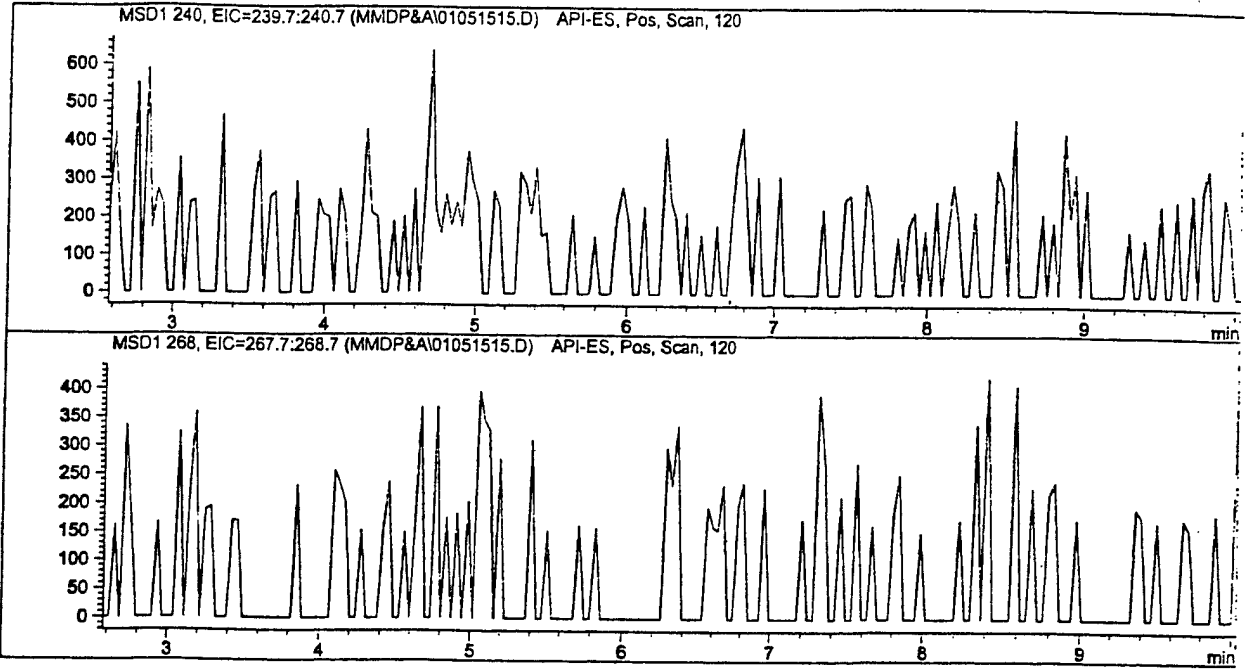
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\*\*\* End of Report \*\*\*

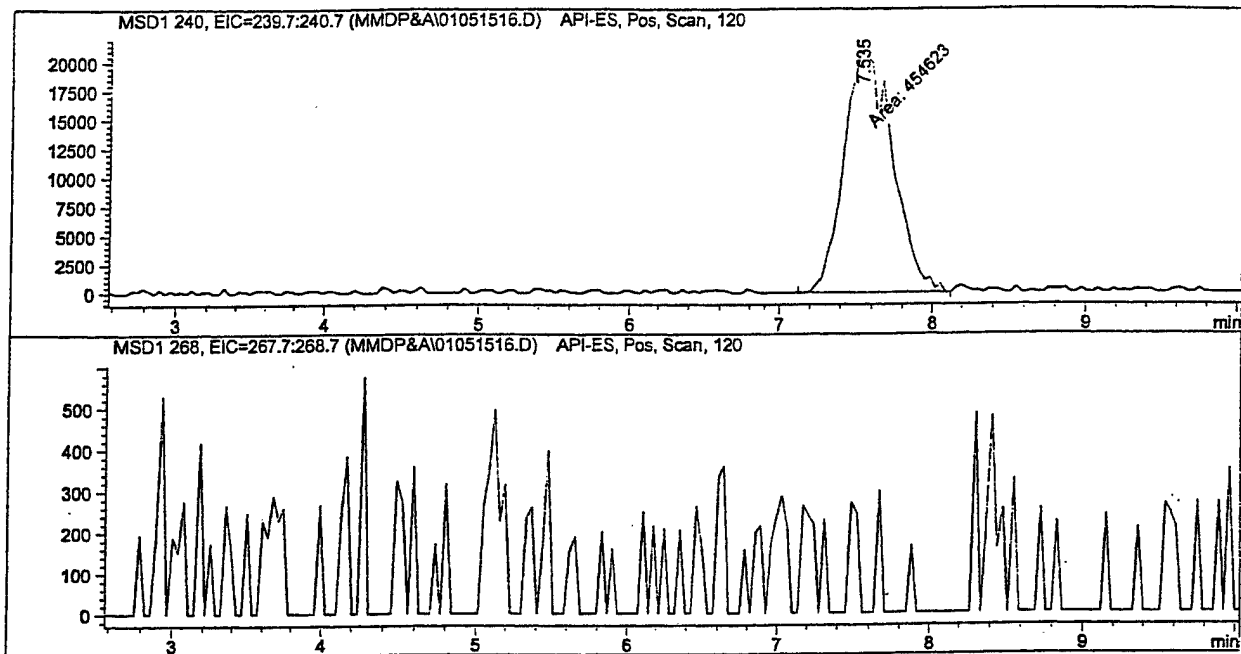


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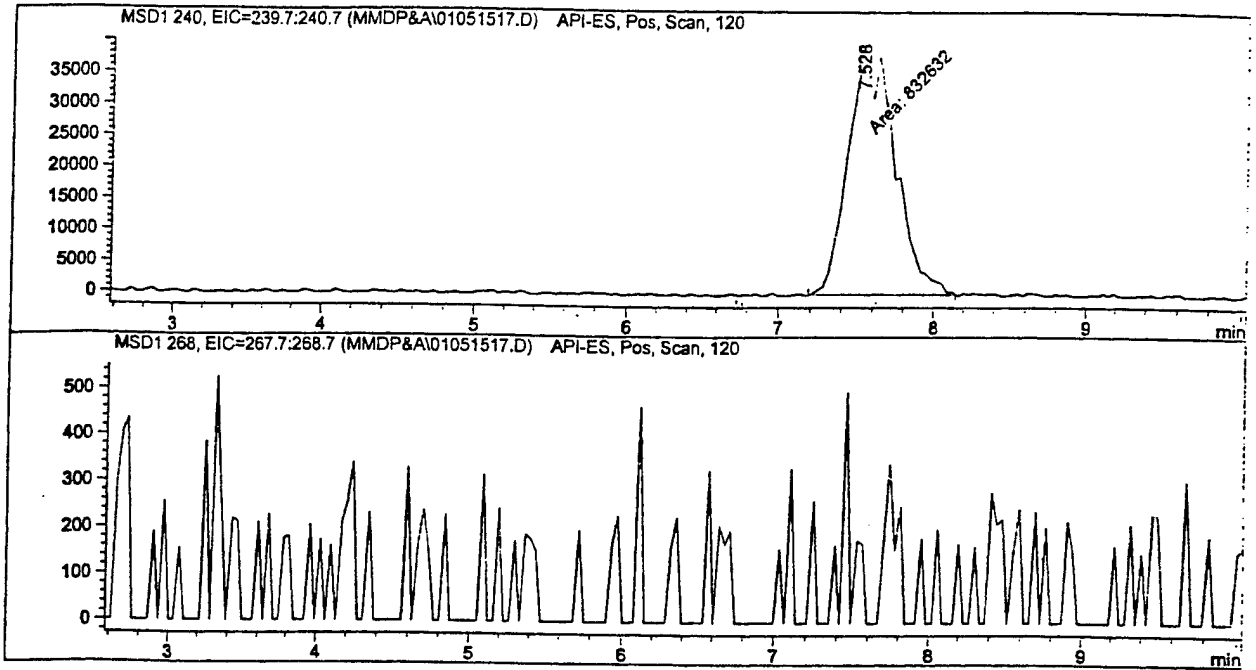


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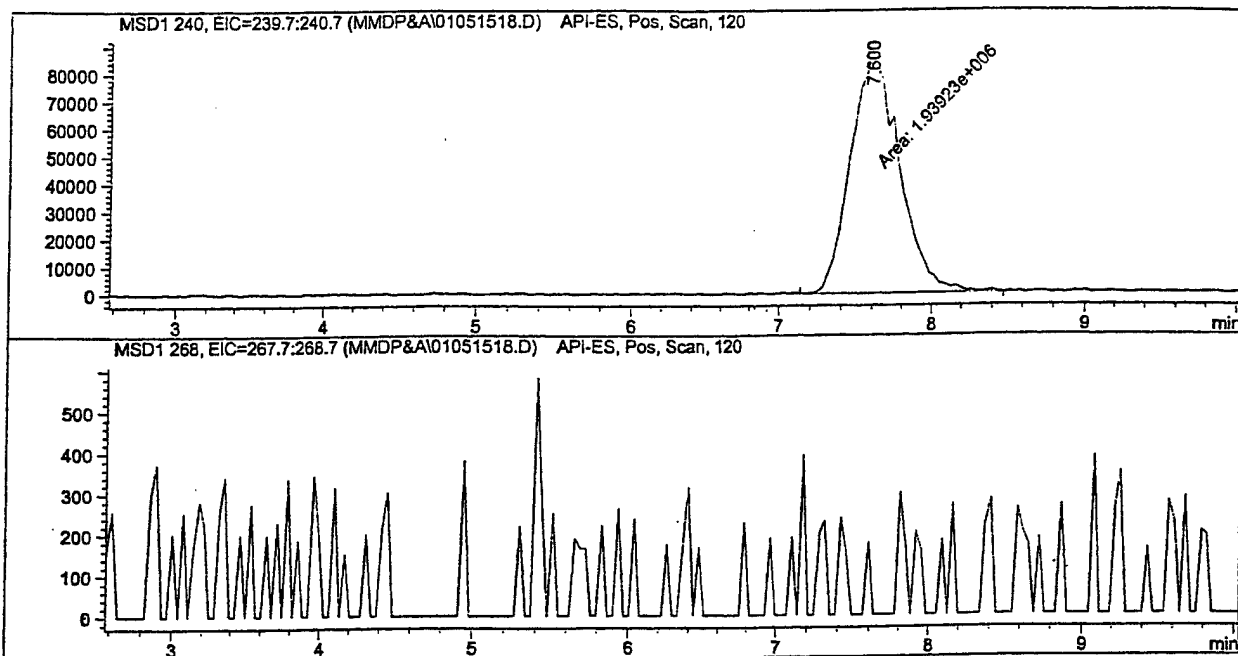




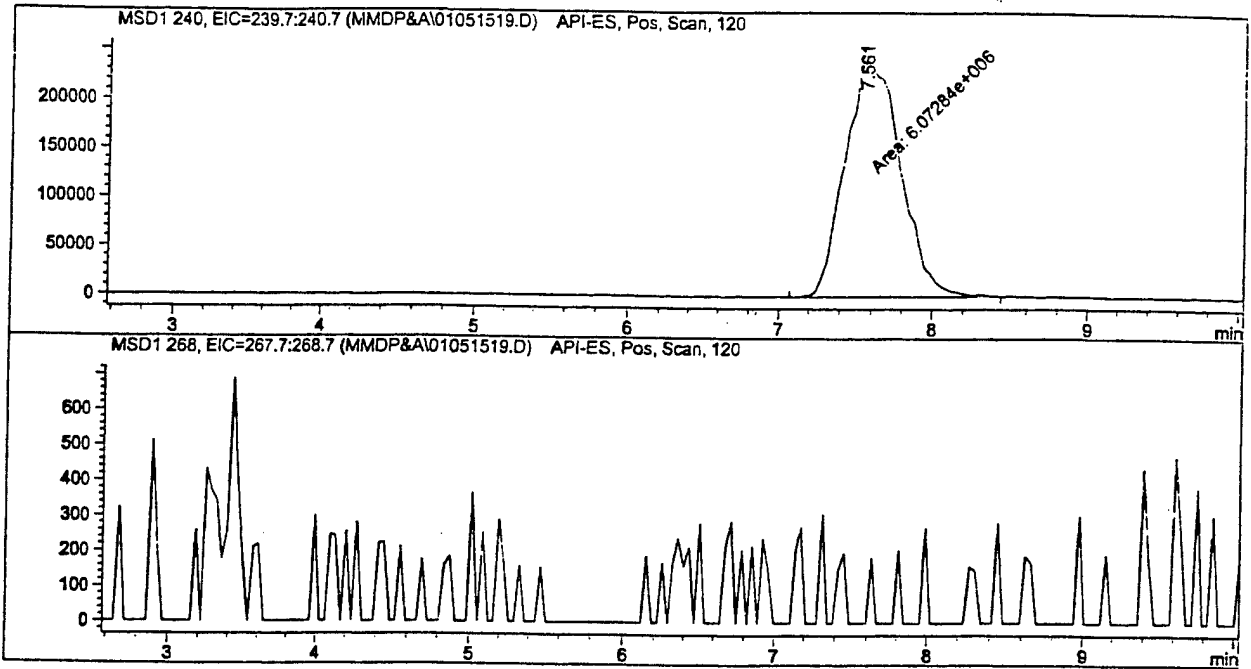
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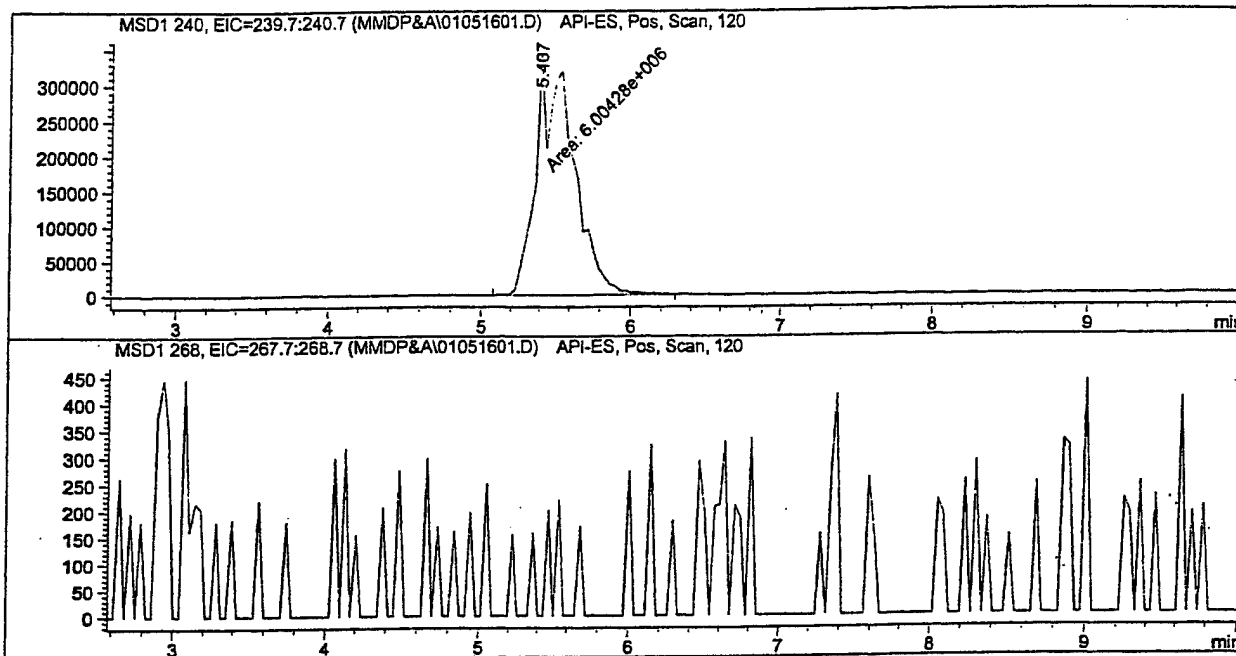
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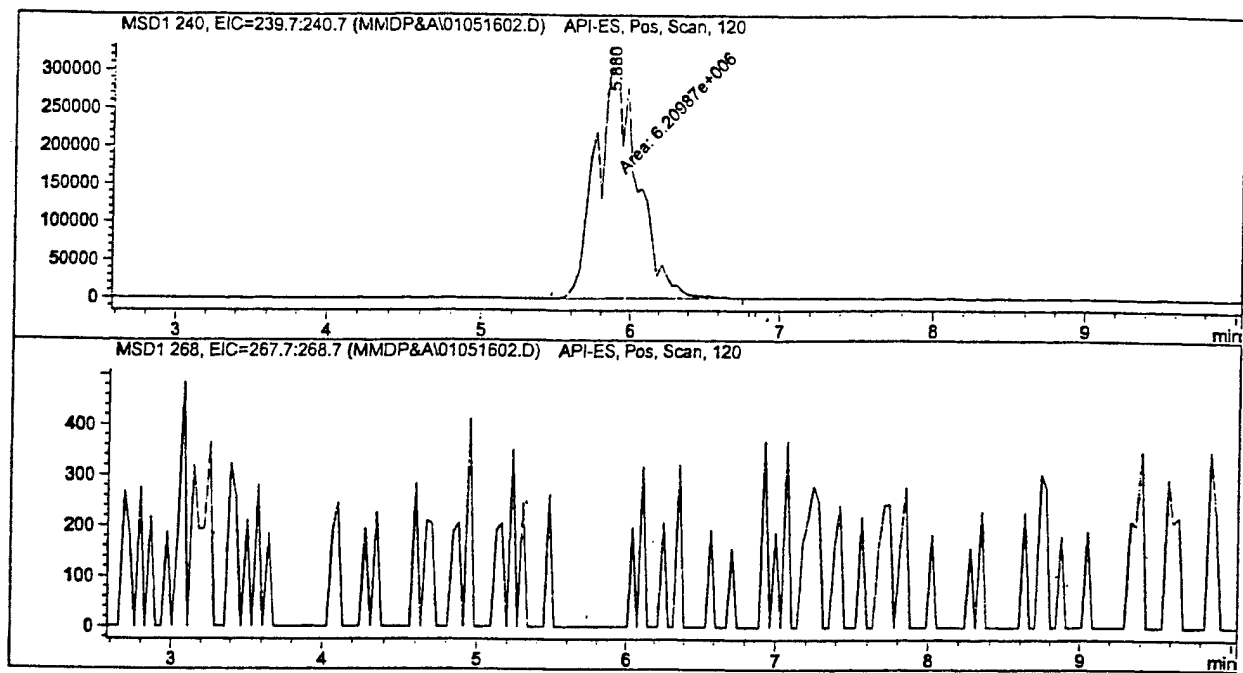
\*\*\* End of Report \*\*\*



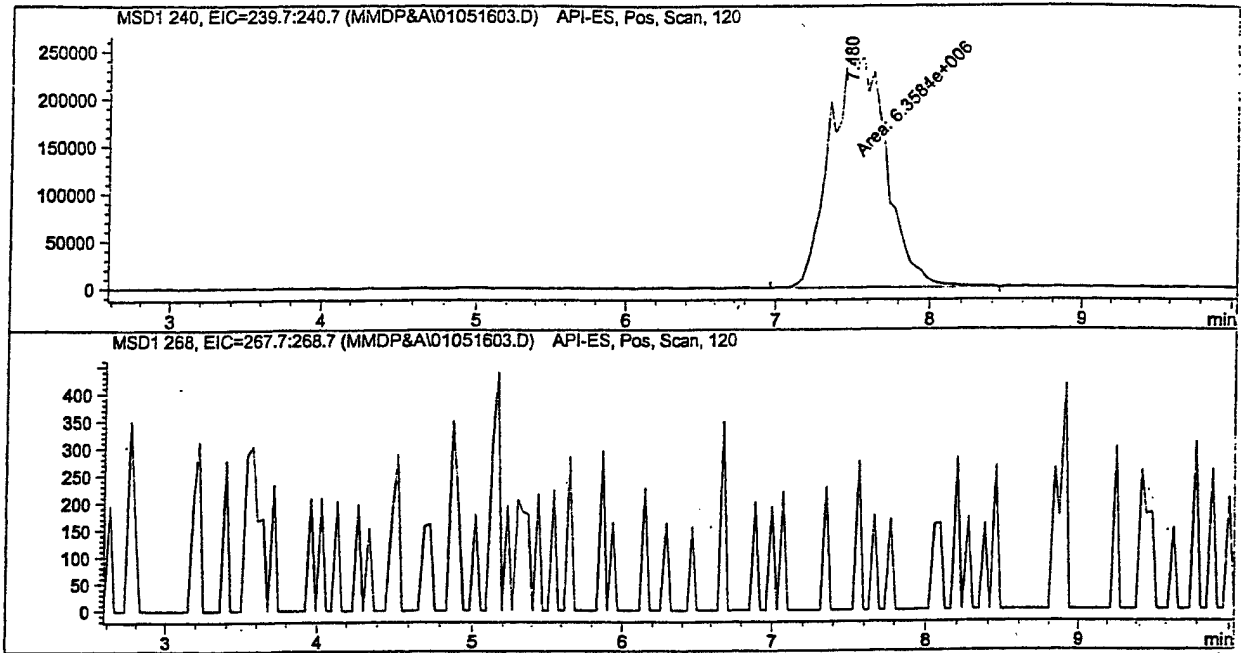
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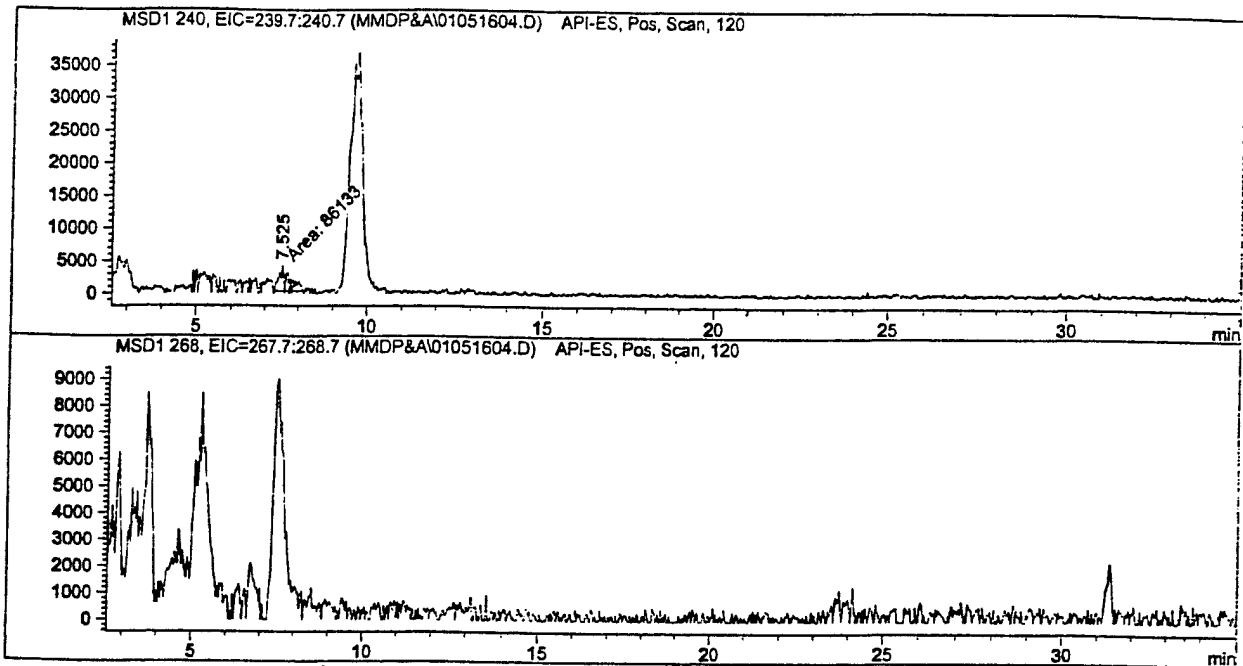
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\*\*\* End of Report \*\*\*

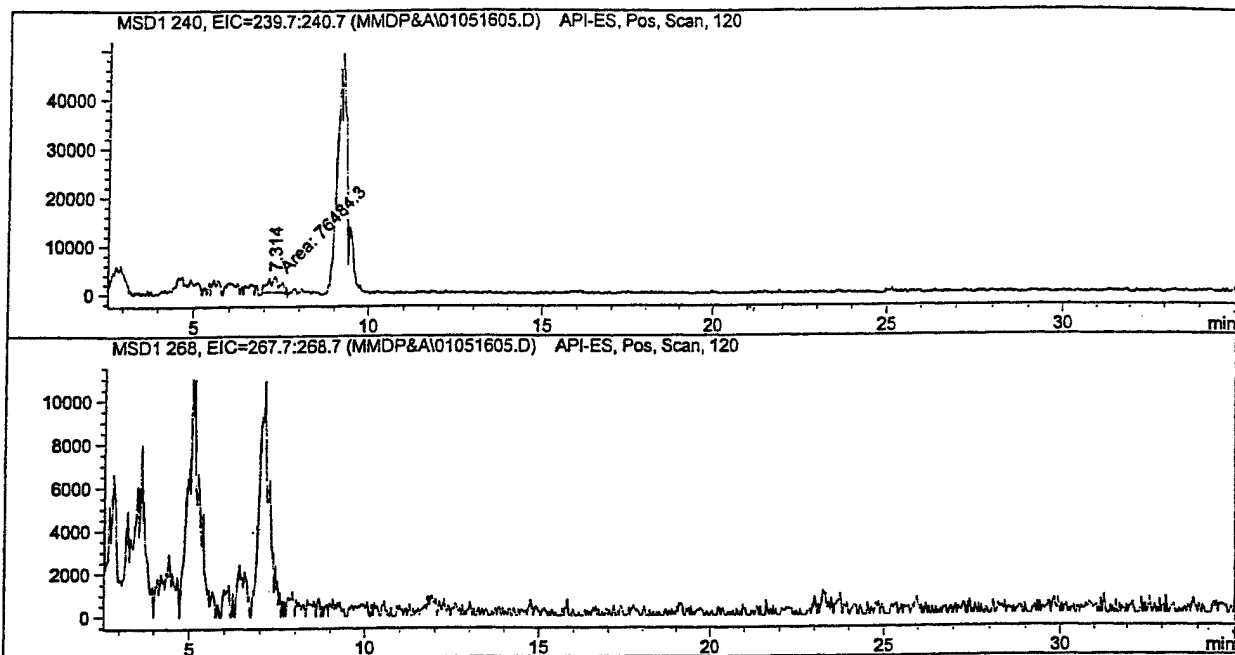


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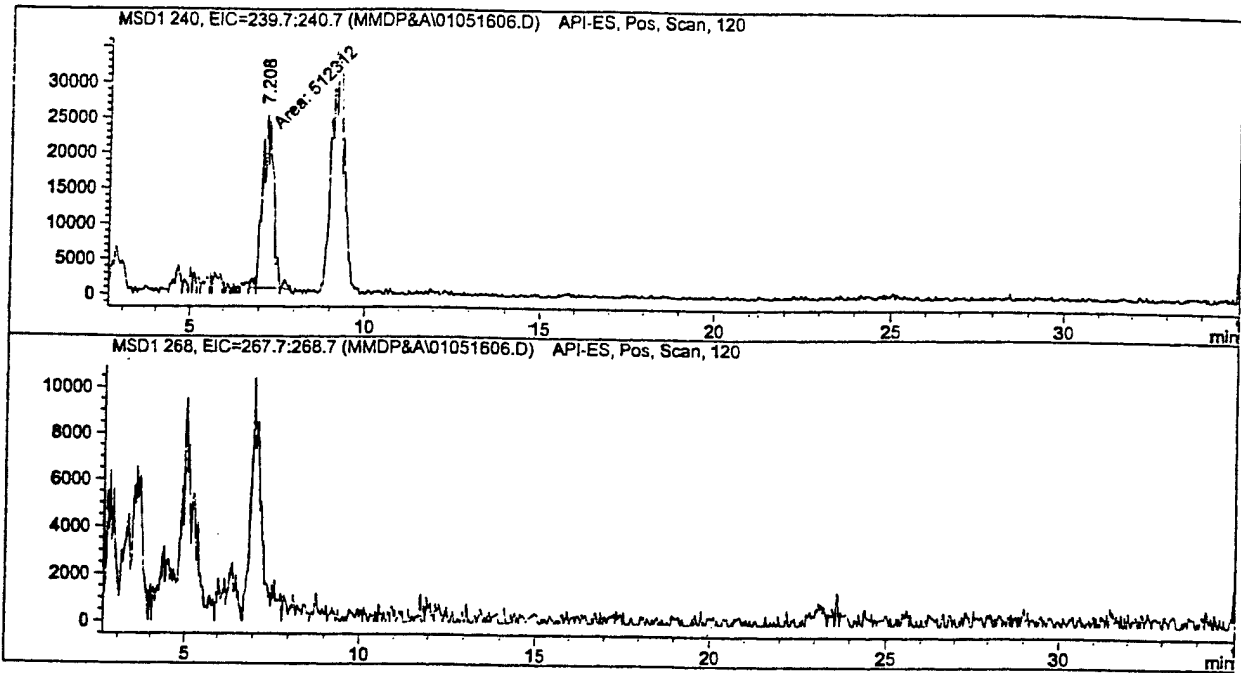


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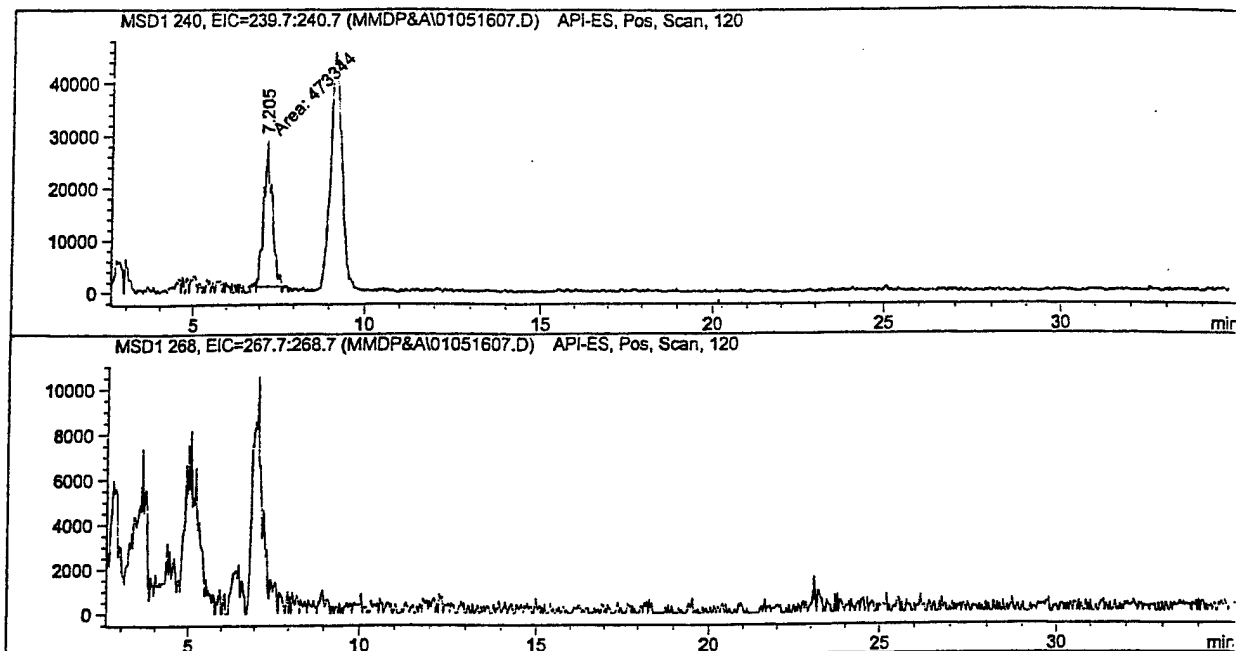




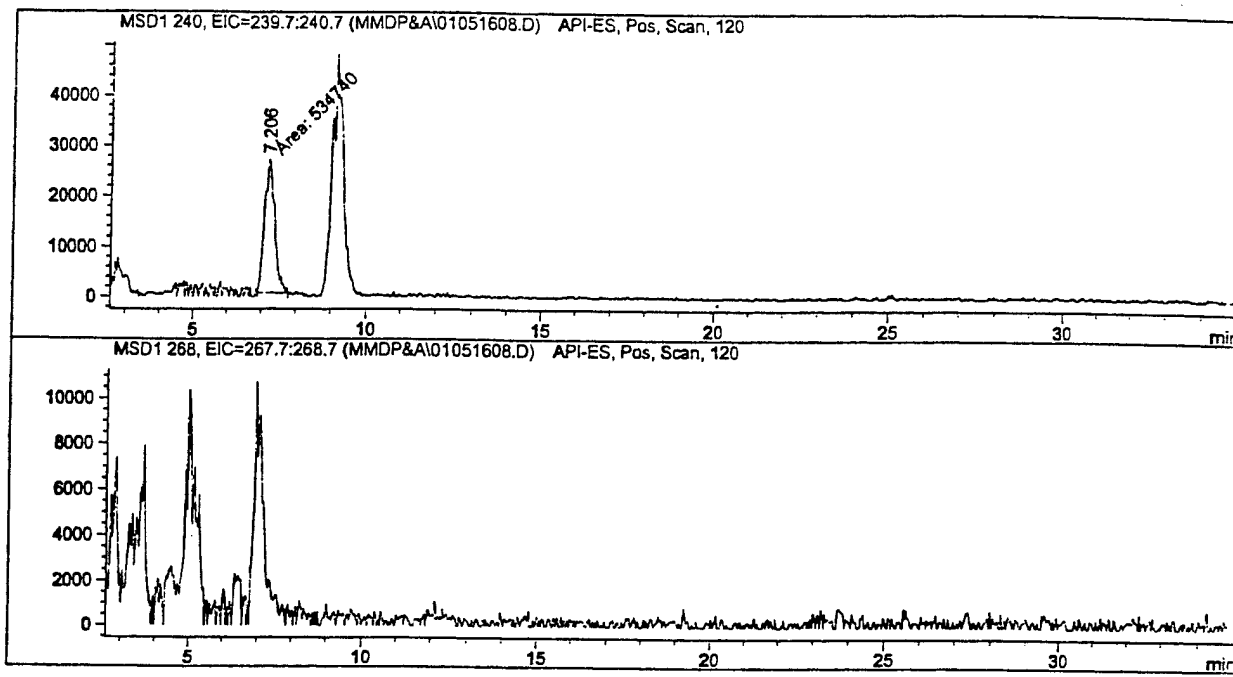
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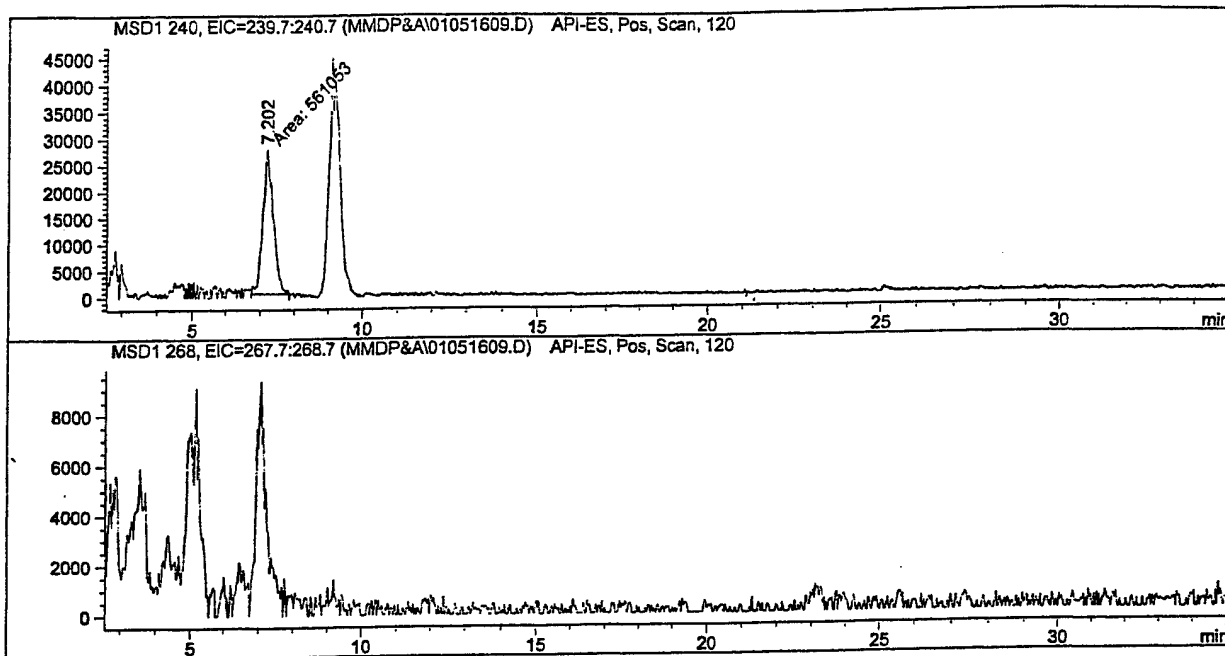
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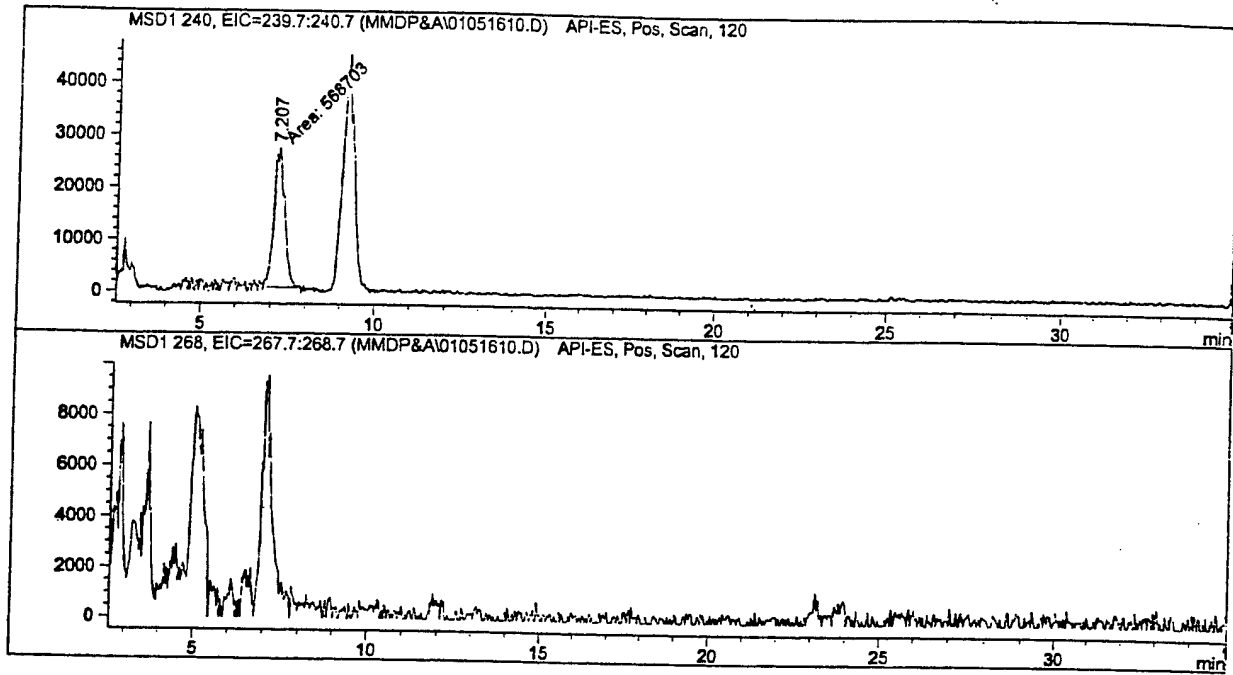
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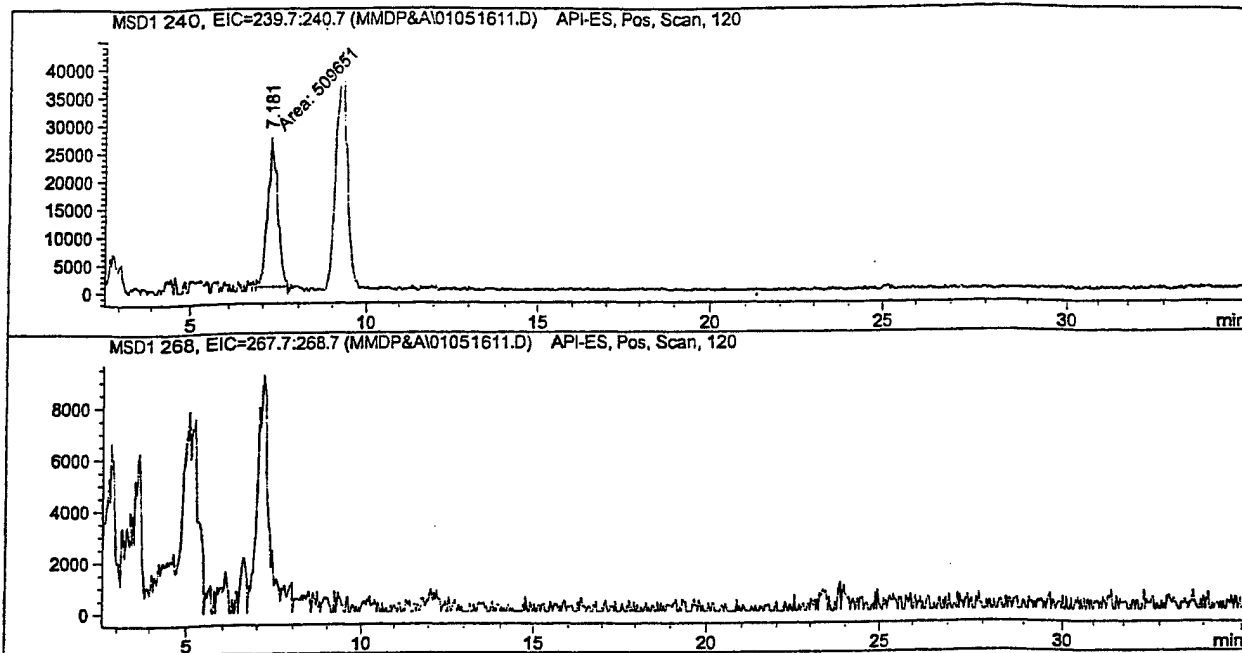
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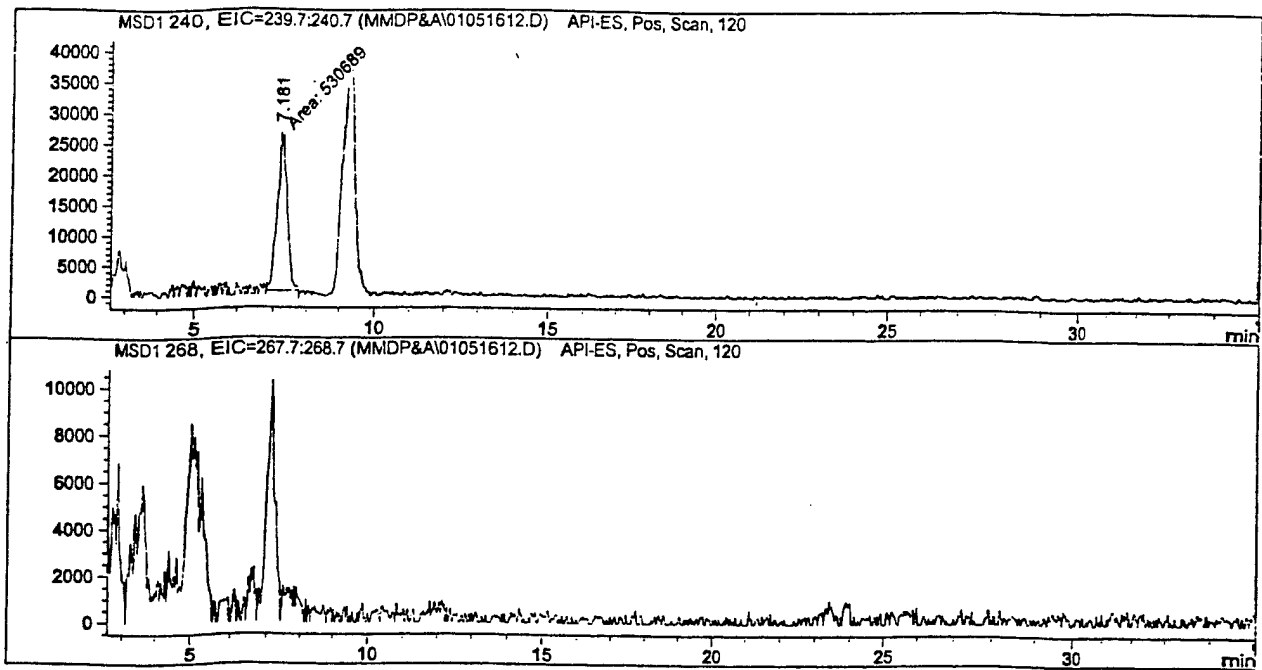
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\*\*\* End of Report \*\*\*

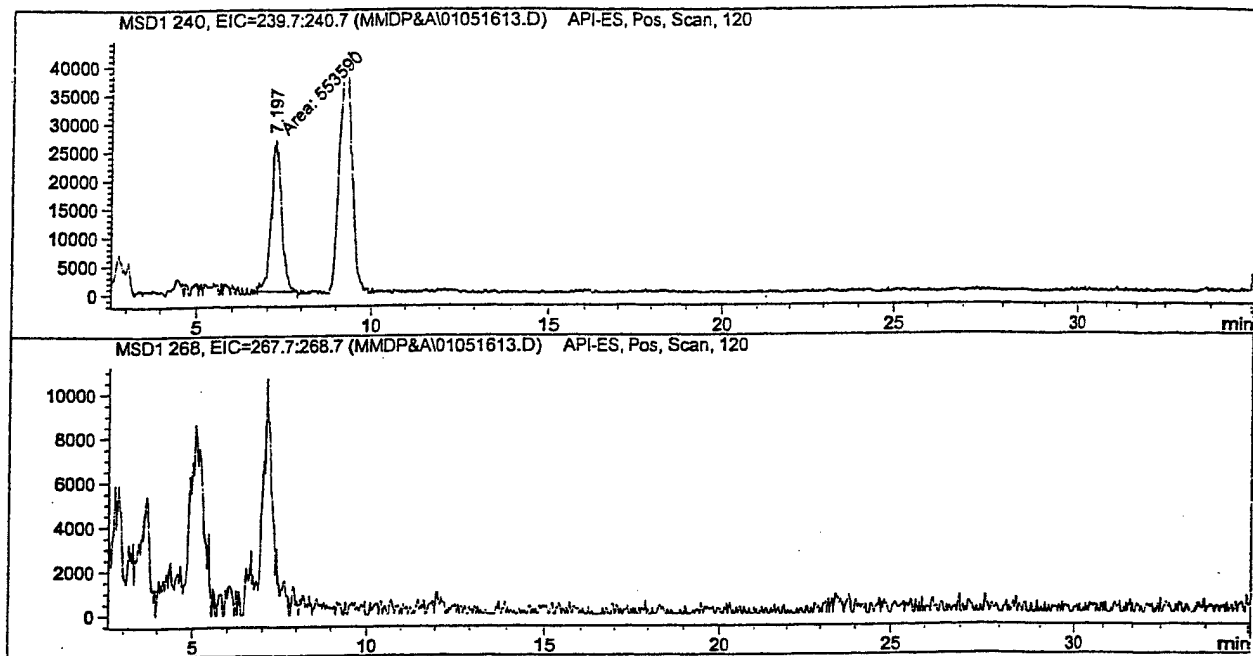


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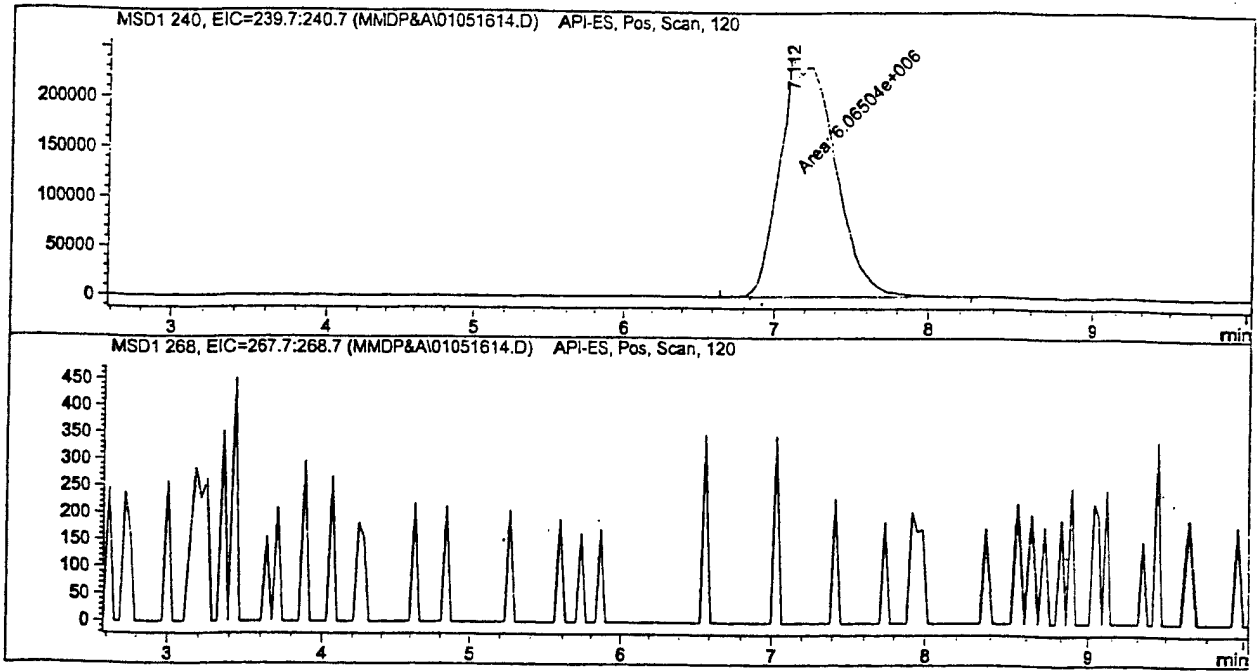


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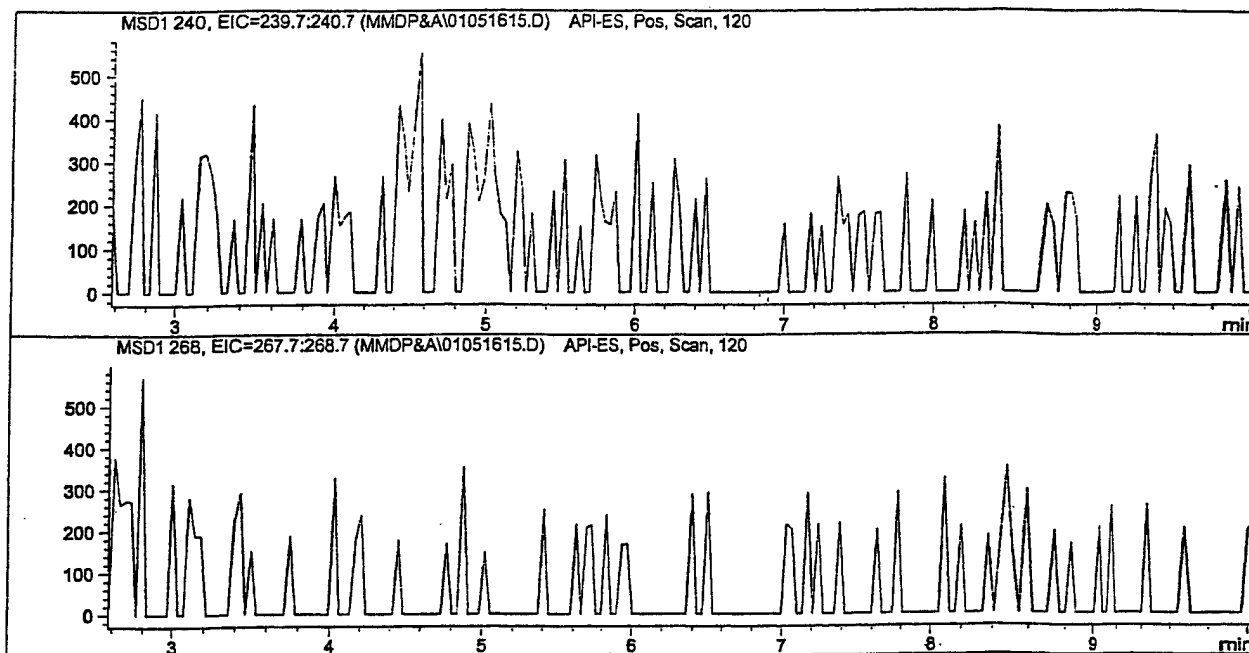




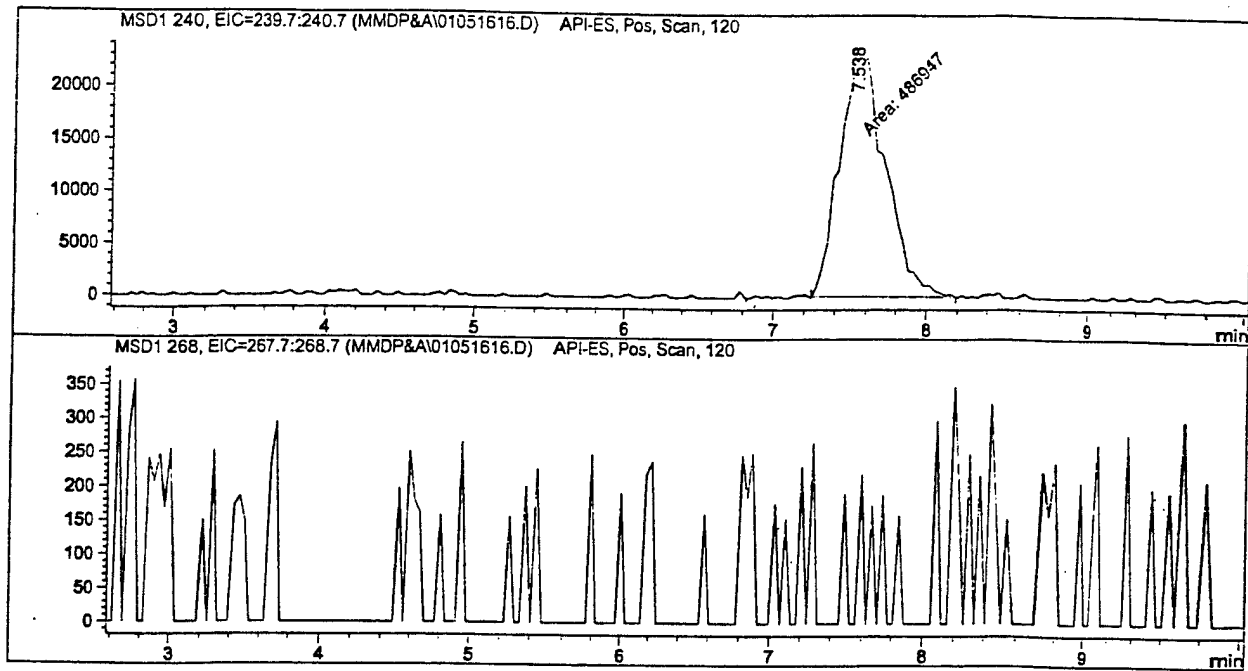
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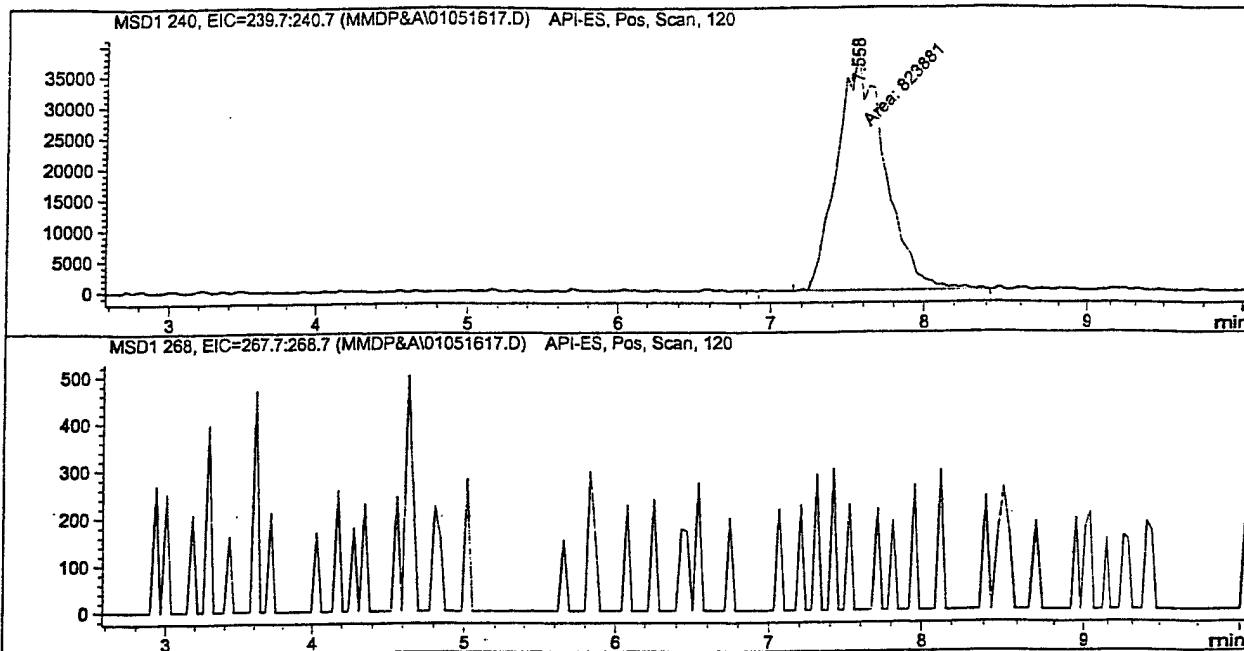
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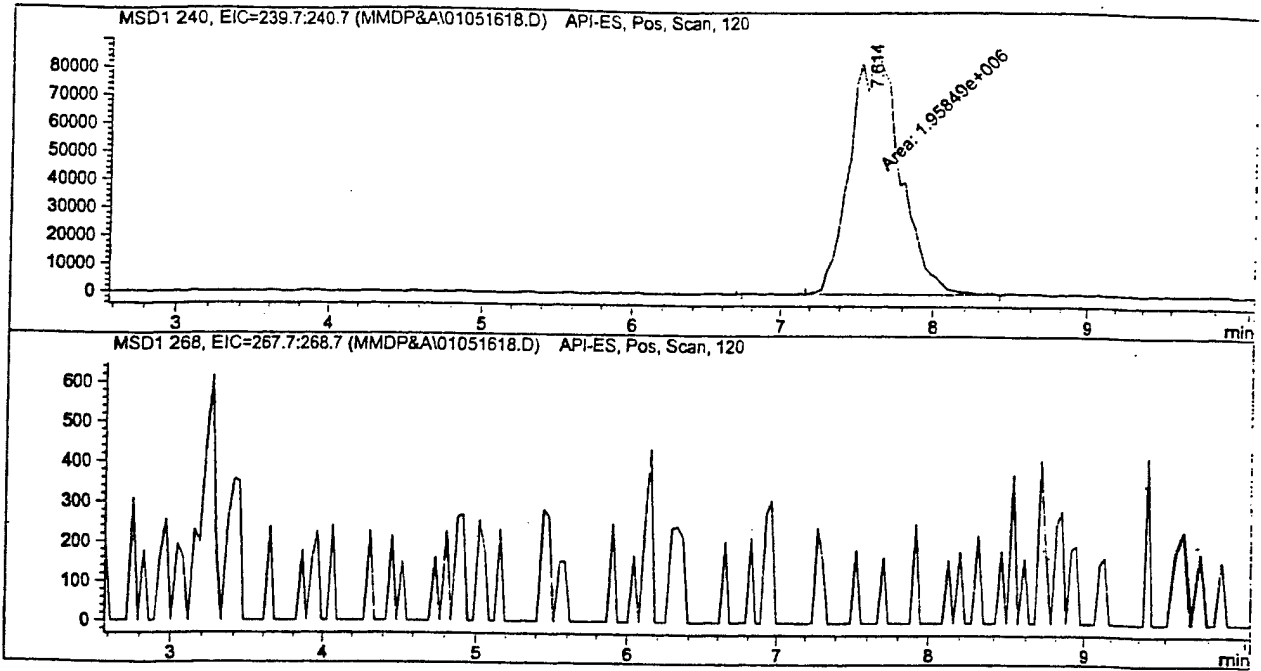
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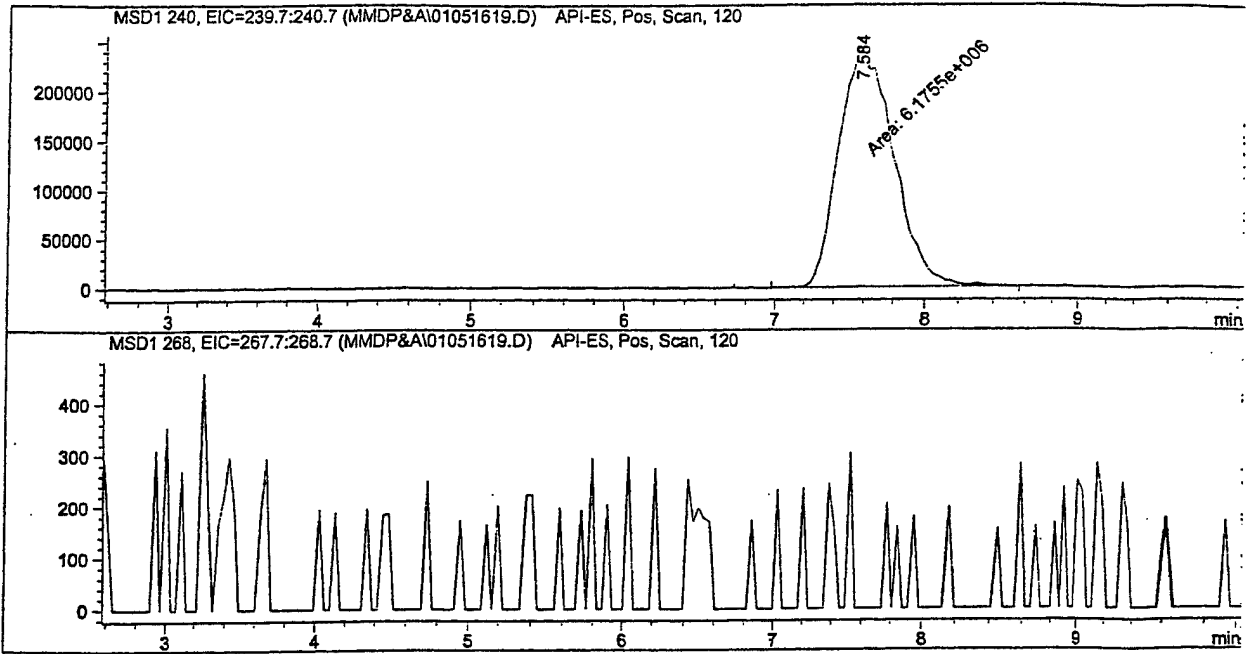
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**APPENDIX B**

**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                                |       |  |
| <b>Ave.</b>                 | 4.72E+05         |   |       |  |
| <b>Std dev.</b>             | 87759.96663      |   |       |  |
| <b>%RSD</b>                 | 18.59%           |   |       |  |
| <b>MDL (ppb)</b>            | 557.4473671      |   |       |  |
| <b>MDL (ng)</b>             | 13.93618418      |   |       |  |
| <b>Recovered amt. (ppb)</b> | 2.35             | (assume zero intercept)                 |       |  |
| <b>Sample Conc. (ppb)</b>   | 113              | (Corrected for dilution of sample only) |       |  |
| <b>Recovery (%)</b>         | 2.08%            |   |       |  |
| <b>EA-2192 Calibration</b>  |                  |   |       |  |
| 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                                      |       |  |
|                             | <b>slope</b>     | 4.97917E-06                             |       |  |
|                             | <b>intercept</b> | -2.072844987                            |       |  |
|                             | <b>corr.</b>     | 0.991789633                             |       |  |

| <b>CCV Results</b> | <b>file</b> | <b>signal</b> | <b>Calc. conc.</b> | <b>Percent</b> |
|--------------------|-------------|---------------|--------------------|----------------|
| 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%           |
|                    |             |               |                    |                |
|                    |             |               |                    |                |



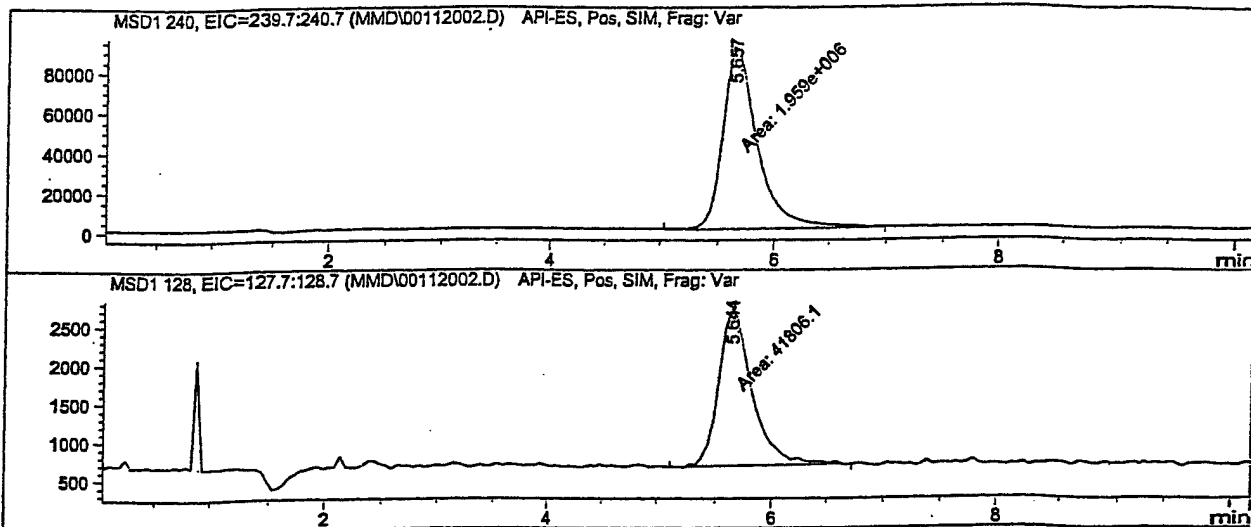
```

=====
Injection Date : 11/20/2000 12:15:50 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

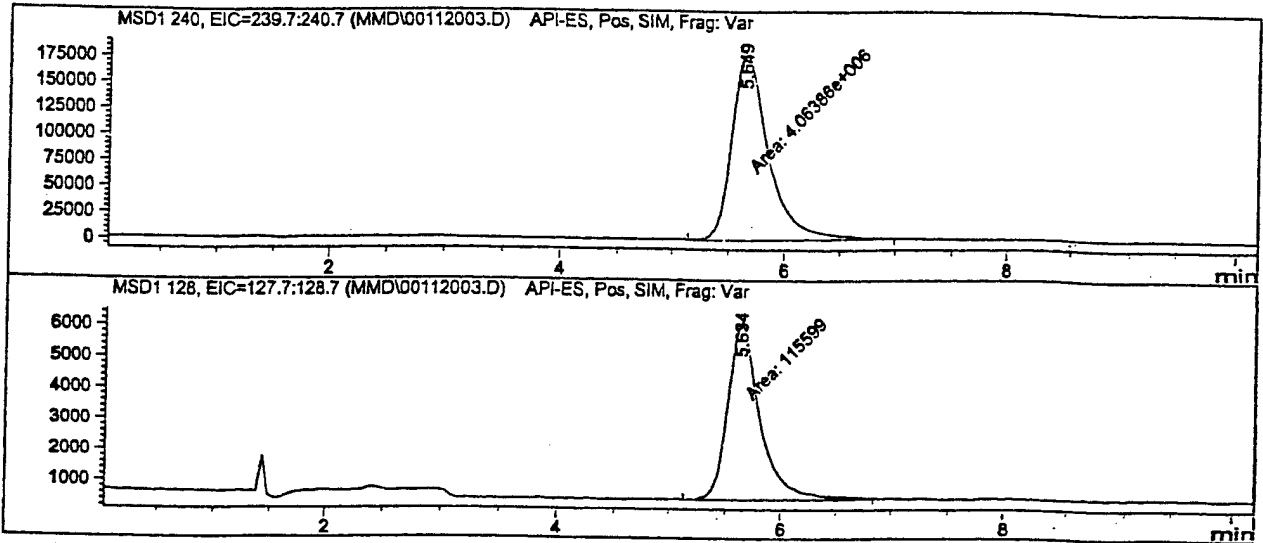
| Peak # | RetTime [min] | Type | Width [min] | Area      | Height     | Area %   |
|--------|---------------|------|-------------|-----------|------------|----------|
| 1      | 5.644         | MM   | 0.3382      | 4.18061e4 | 2060.07715 | 100.0000 |

Totals : 4.18061e4 2060.07715

```

=====
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      | 1.15599e5 | 5816.12891 | 100.0000 |

Totals : 1.15599e5 5816.12891

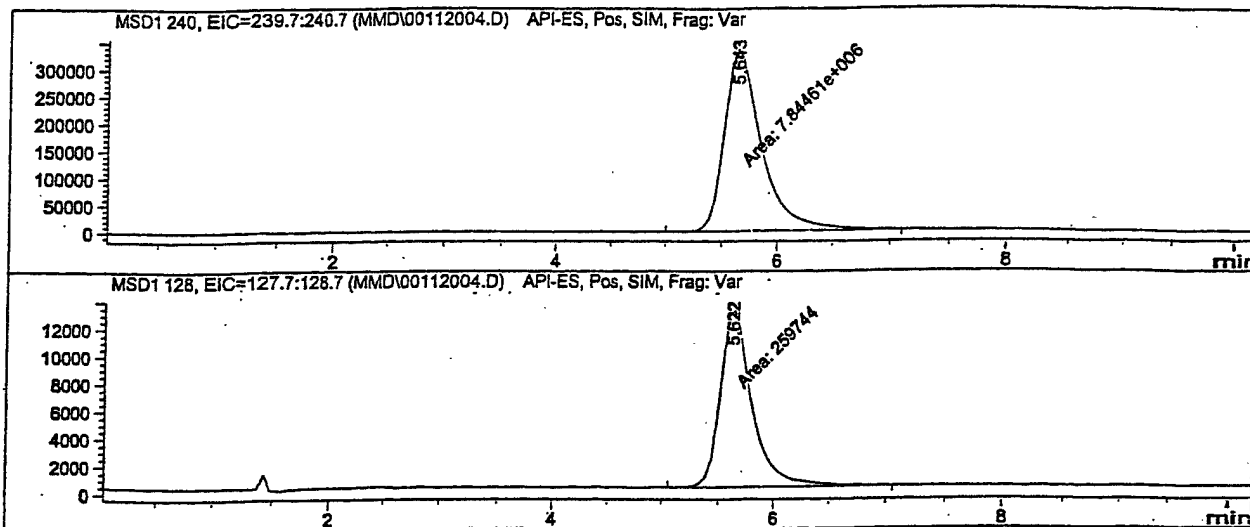
```

=====
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

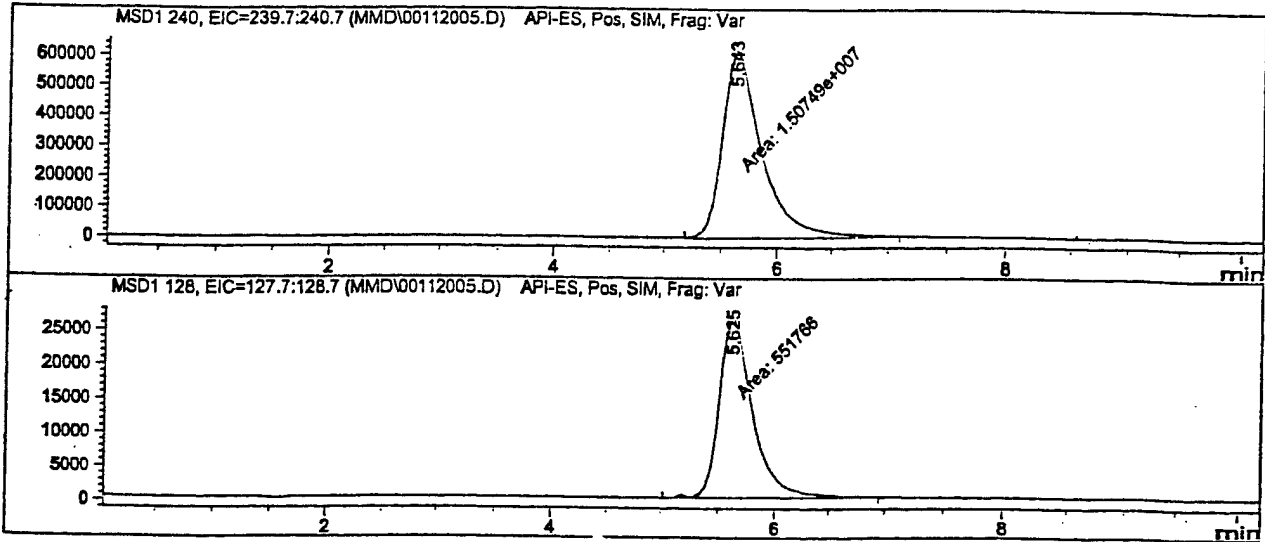
```

=====
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

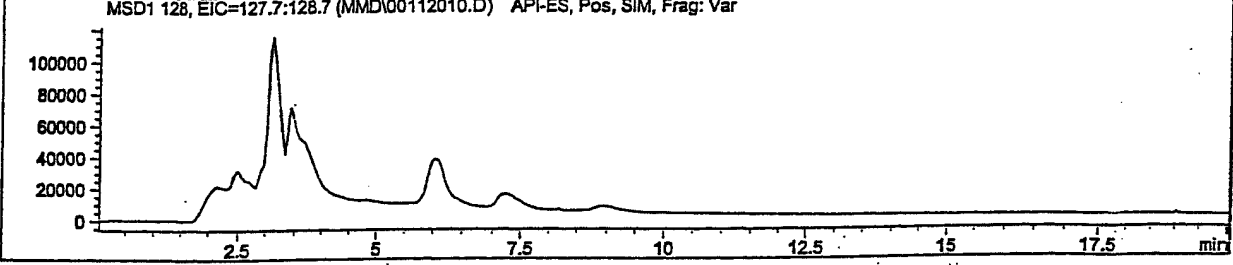
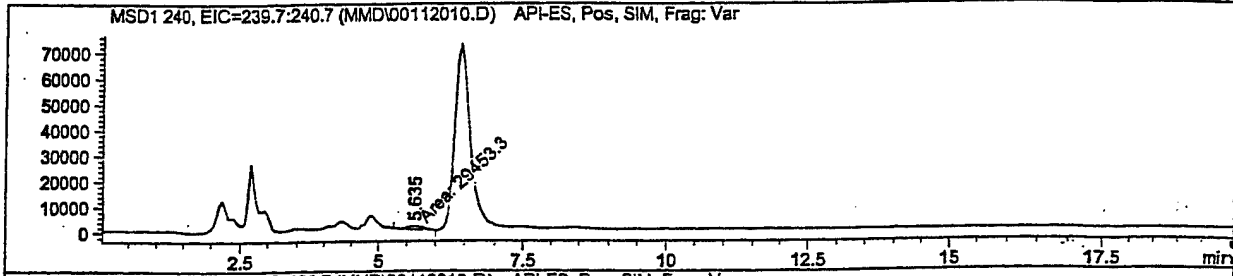


```

=====
Injection Date : 11/20/2000 1:49:05 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 [min] | Type | Width [min] | Area      | Height     | Area %   |
|--------|---------------|------|-------------|-----------|------------|----------|
| 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

```

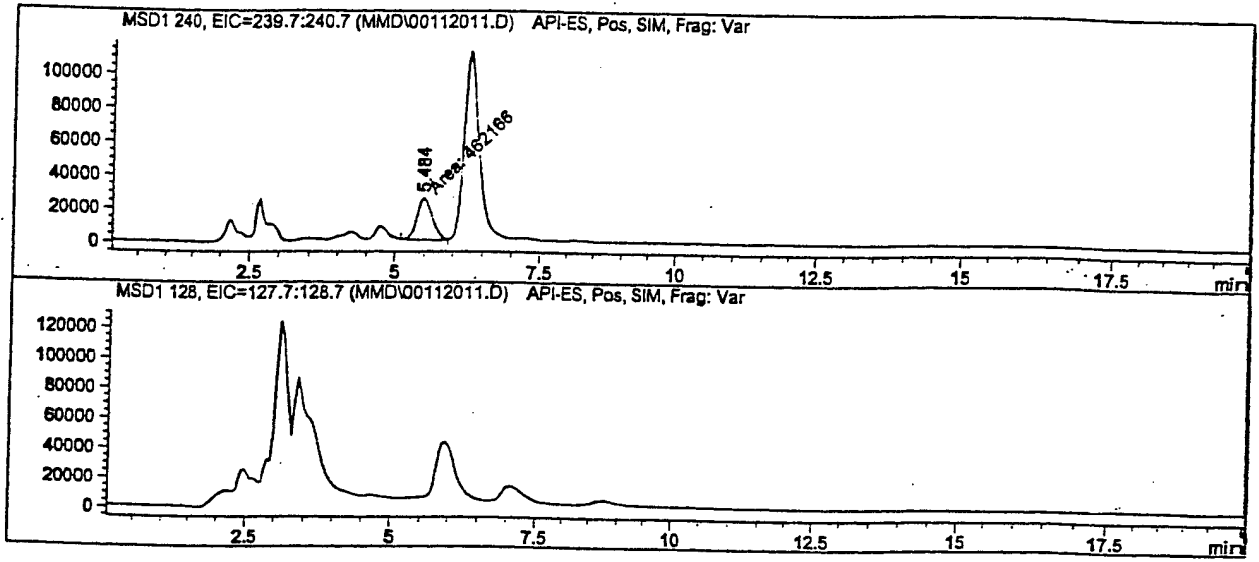
=====
*** End of Report ***

```

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

Sample Name: NB114P36D

=====  
Injection Date : 11/20/2000 2:48:07 PM                   Seq. Line : 1  
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      | 5.484         | MM   | 0.3020      | 4.62166e5 | 2.55063e4 | 100.0000 |

Totals :                                   4.62166e5 2.55063e4

Signal 2: MSD1 128, EIC=127.7:128.7

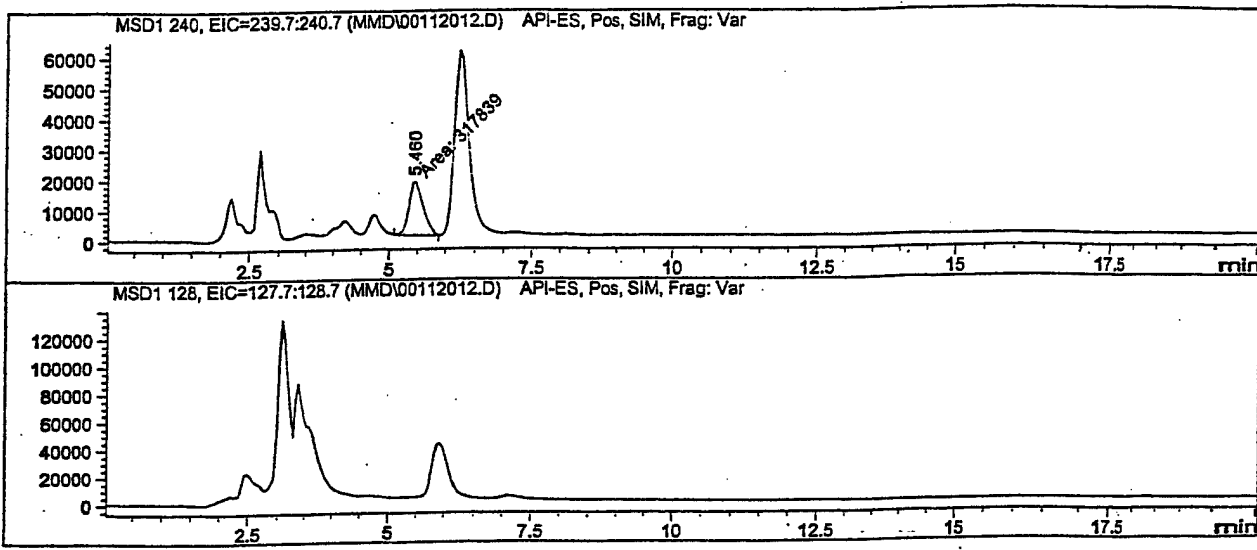
=====  
\*\*\* End of Report \*\*\*

```

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

Totals :                                    3.17839e5 1.78684e4

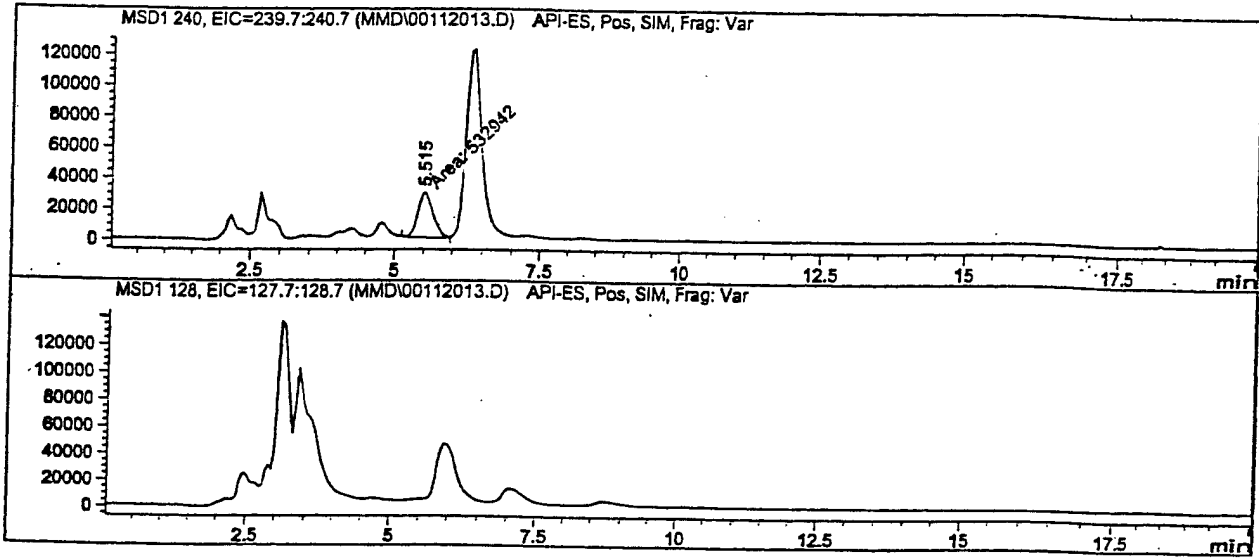
Signal 2: MSD1 128, EIC=127.7:128.7

=====  
 \*\*\* End of Report \*\*\*

```

=====
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

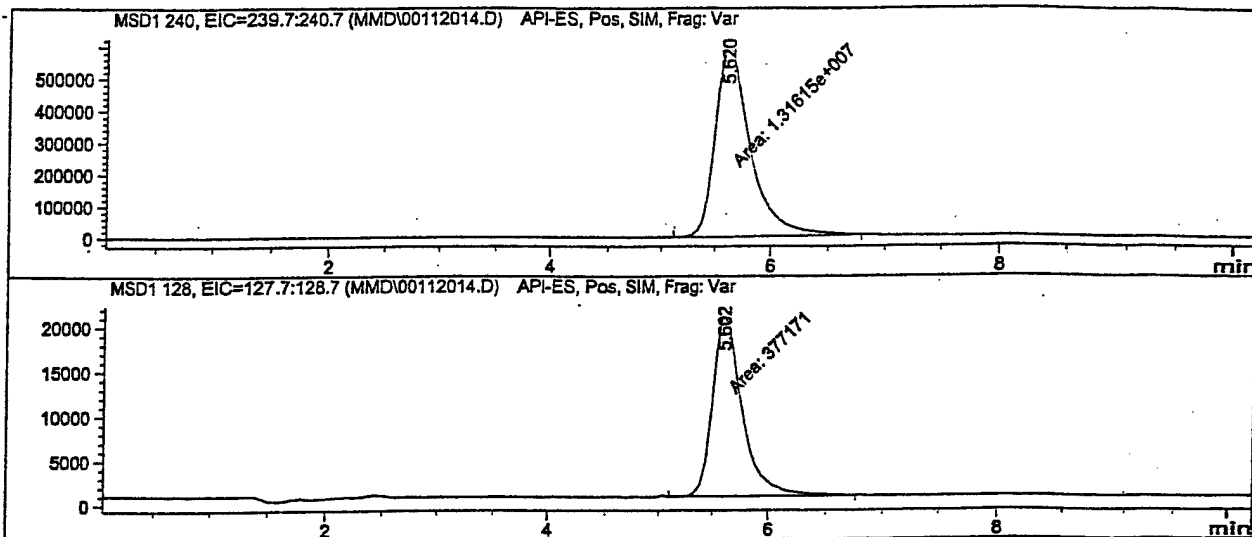
Signal 2: MSD1 128, EIC=127.7:128.7

=====  
 \*\*\* End of Report \*\*\*

```

=====
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

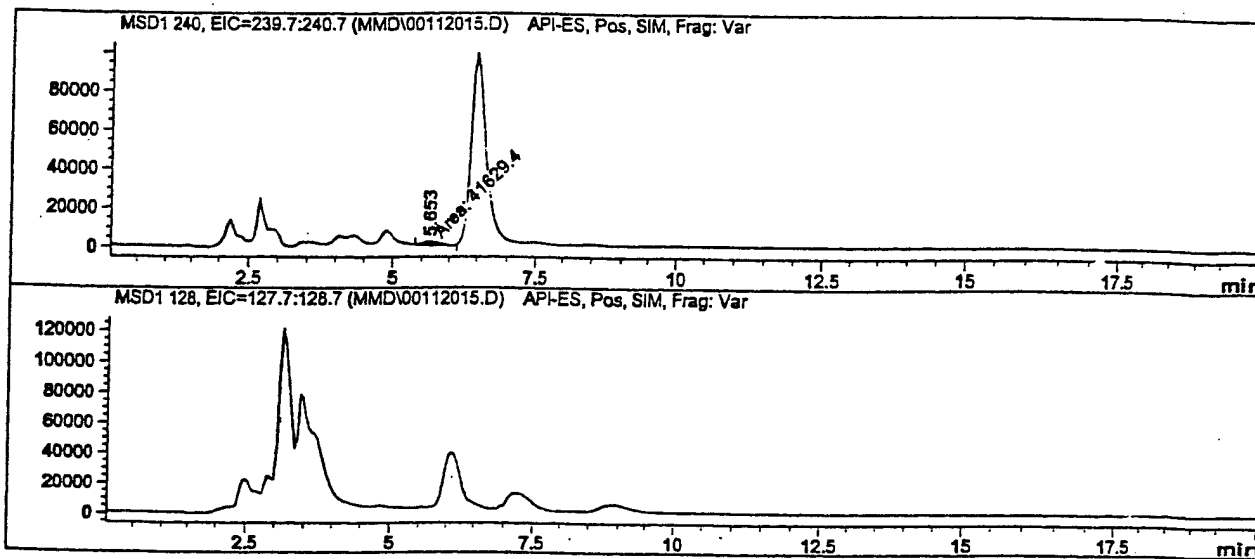
| Peak # | RetTime [min] | Type | Width [min] | Area      | Height    | Area %   |
|--------|---------------|------|-------------|-----------|-----------|----------|
| 1      | 5.602         | MM   | 0.3018      | 3.77171e5 | 2.08291e4 | 100.0000 |

Totals :                            3.77171e5   2.08291e4

=====  
 \*\*\* End of Report \*\*\*

```

=====
Injection Date : 11/20/2000 5:06:25 PM      Seq. Line : 5
Sample Name    : NB114936B                  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

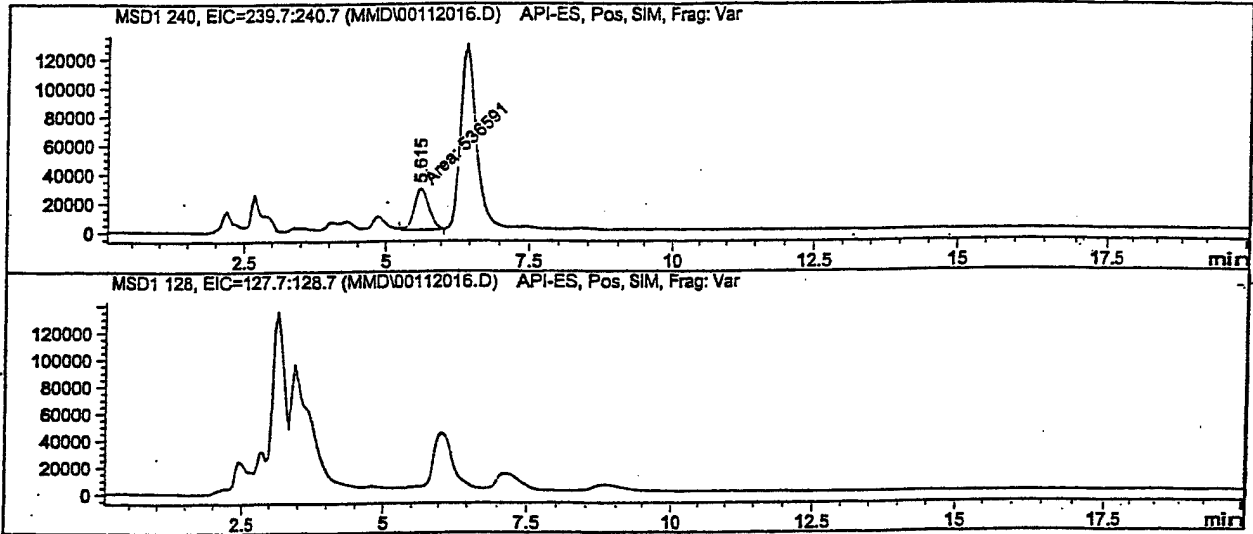
=====  
 \*\*\* End of Report \*\*\*

```

=====
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

```

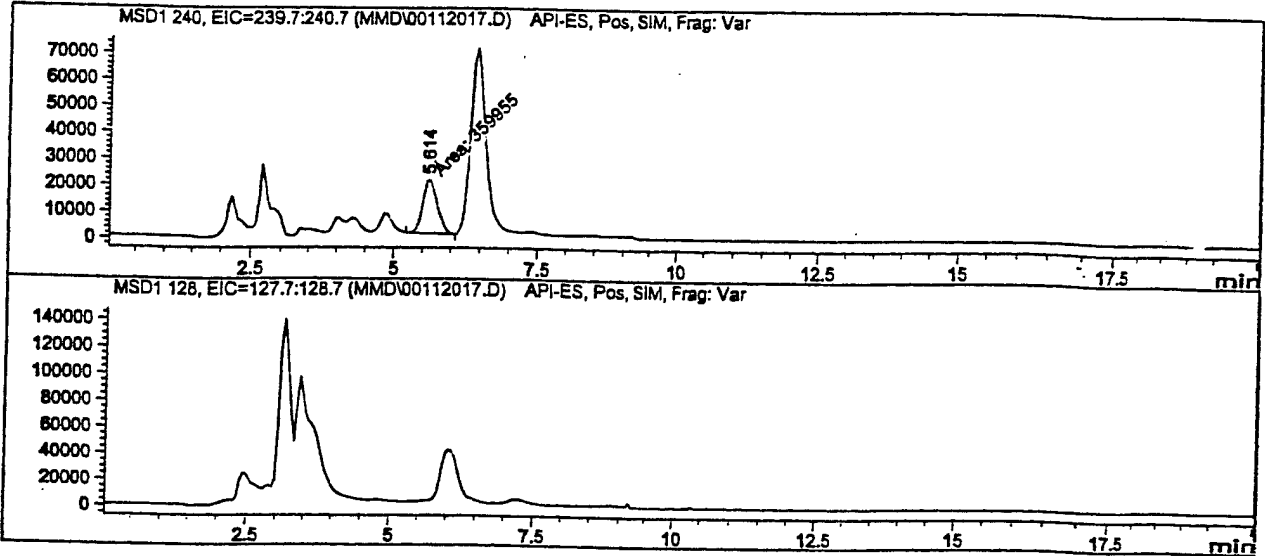
=====
*** End of Report ***

```

```

=====
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 \*\*\*



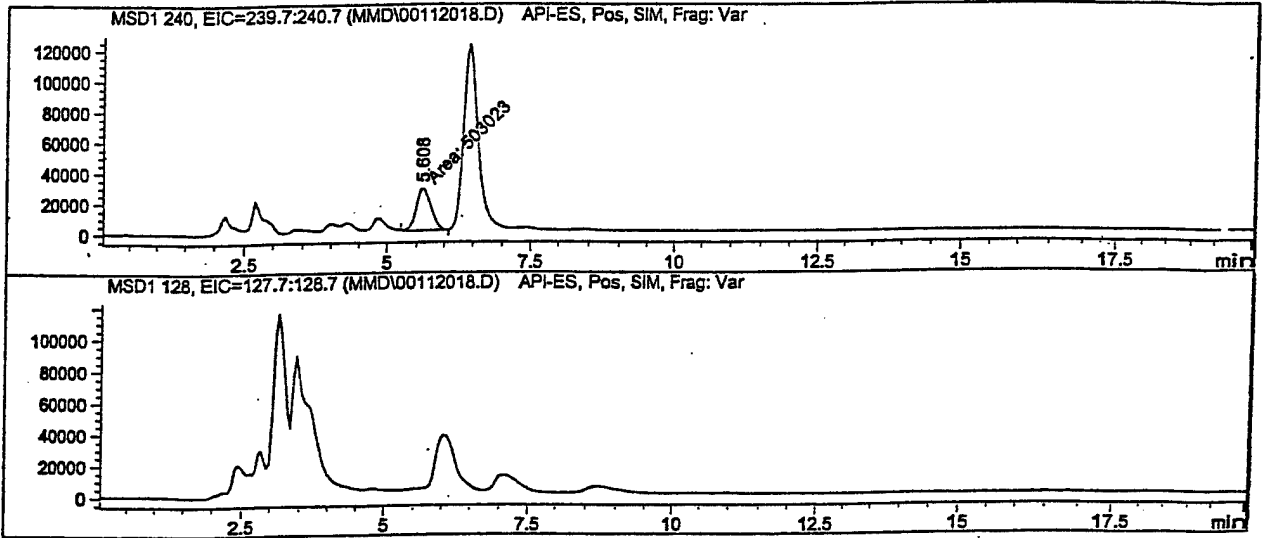
```

=====
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

```

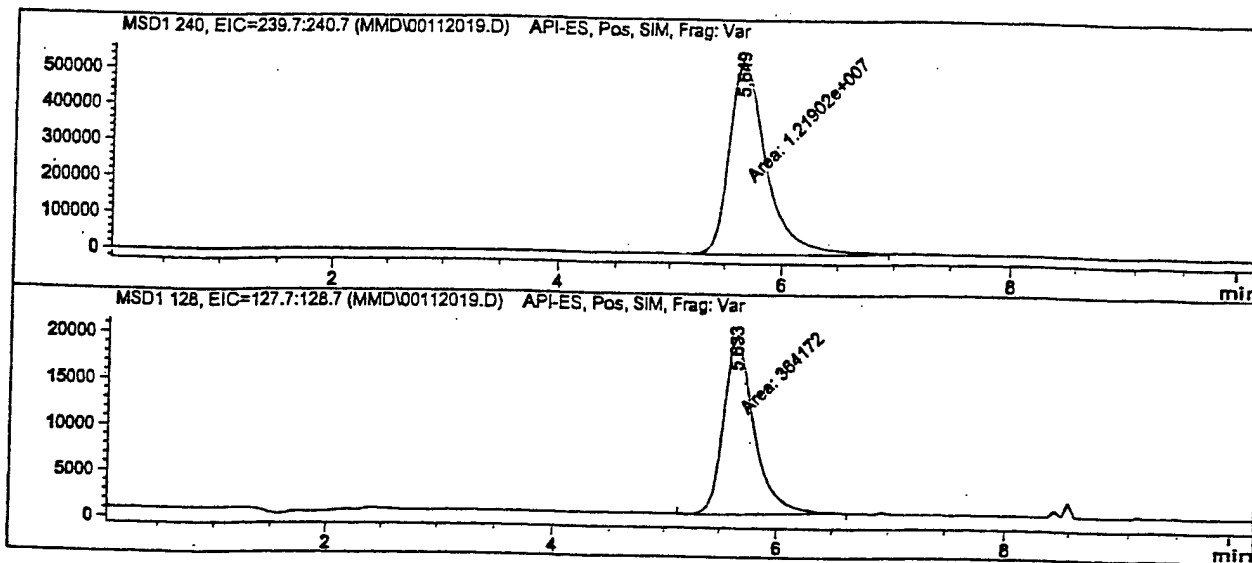
=====
*** End of Report ***

```

```

=====
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

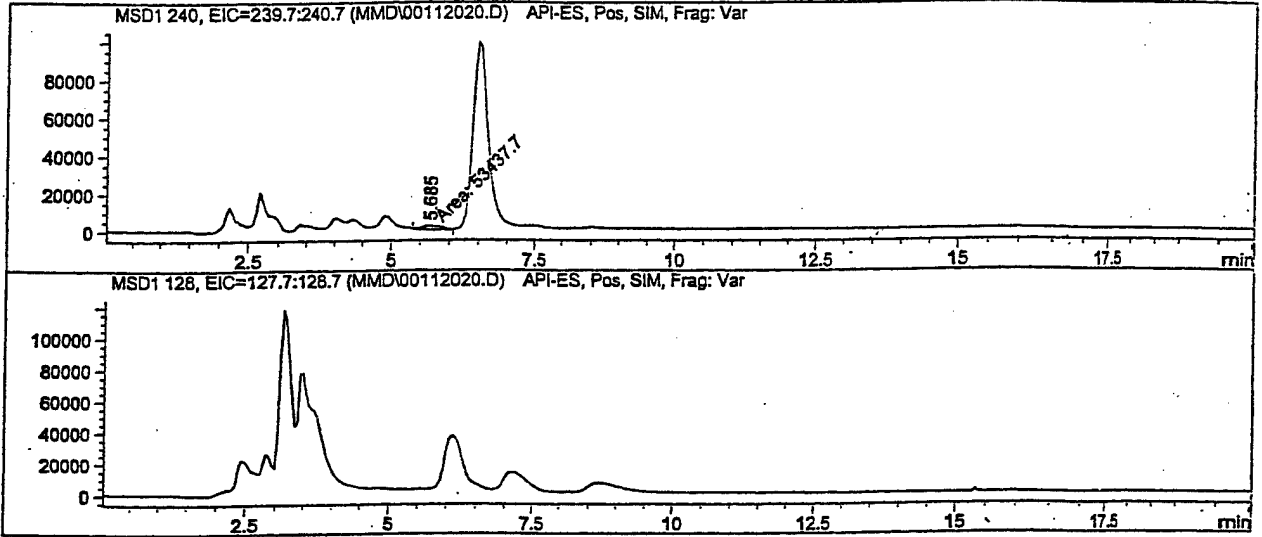
\*\*\* End of Report \*\*\*

```

=====
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 [min] | Type | Width [min] | Area      | Height     | Area %   |
|--------|---------------|------|-------------|-----------|------------|----------|
| 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

```

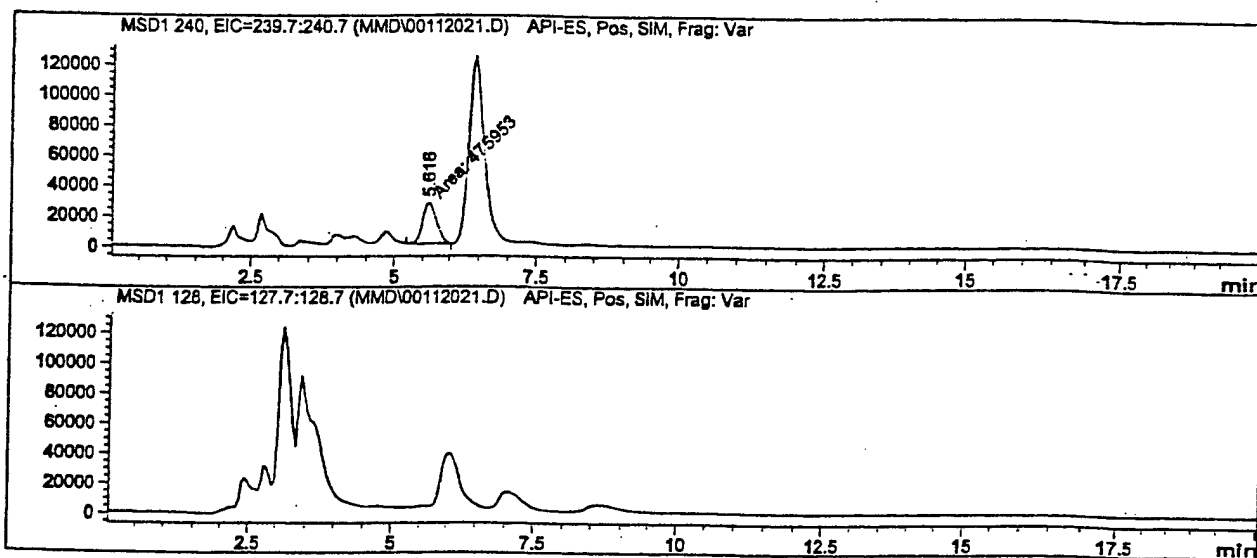
=====
*** End of Report ***
=====

```

```

=====
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 \*\*\*

```

=====
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
=====

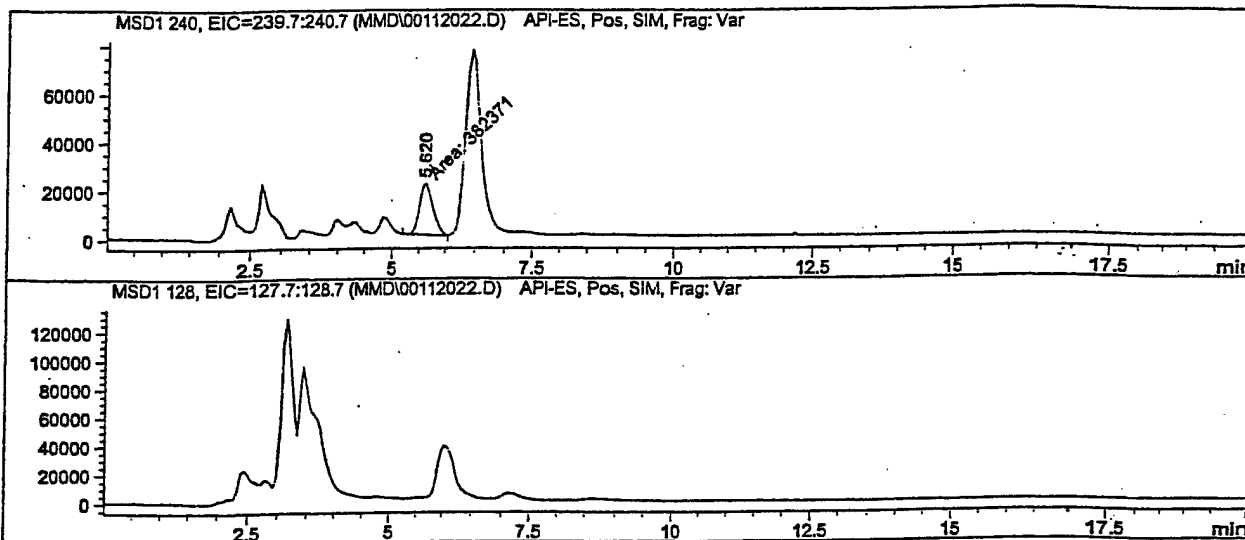
```

```

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

Totals : 3.82371e5 2.14461e4

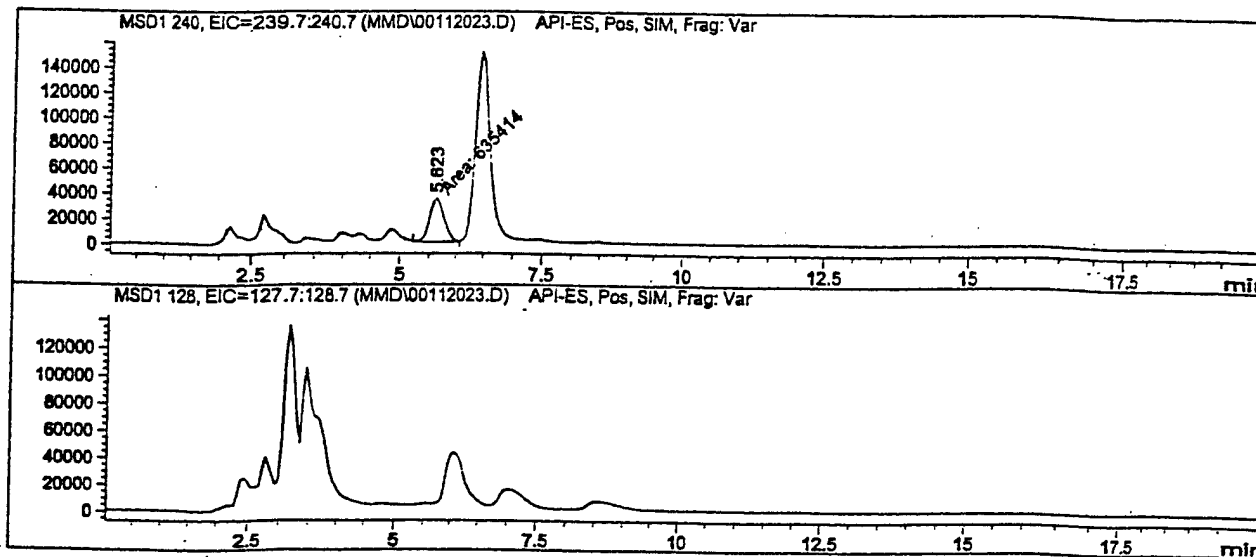
Signal 2: MSD1 128, EIC=127.7:128.7

\*\*\* End of Report \*\*\*

```

=====
Injection Date : 11/20/2000 10:12:26 PM      Seq. Line : 13
Sample Name    : NB114936H                    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

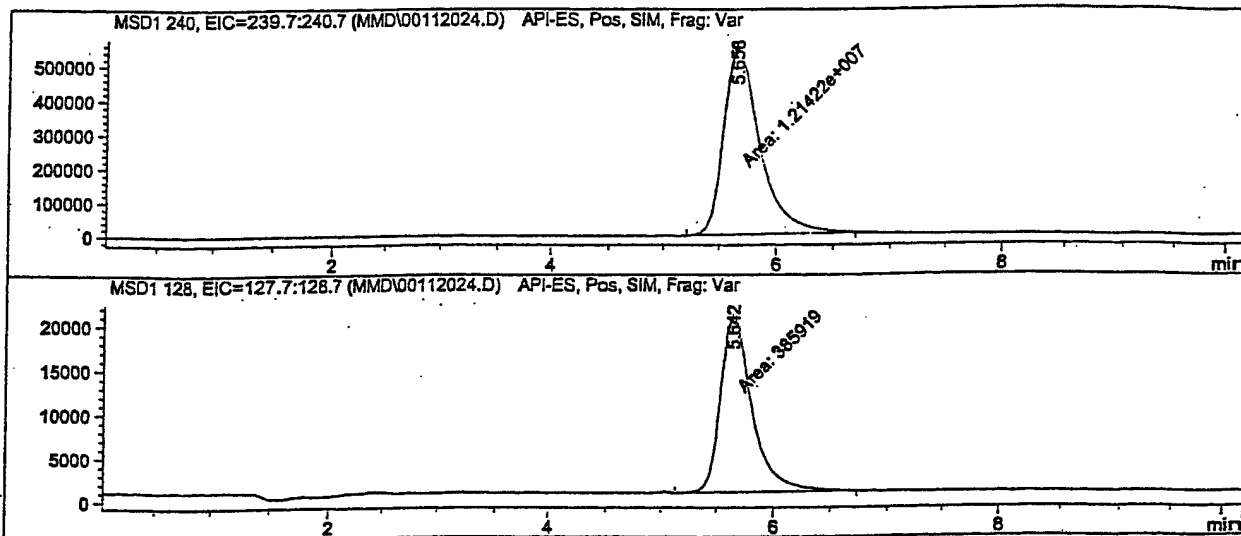
Signal 2: MSD1 128, EIC=127.7:128.7

=====  
 \*\*\* End of Report \*\*\*

```

=====
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

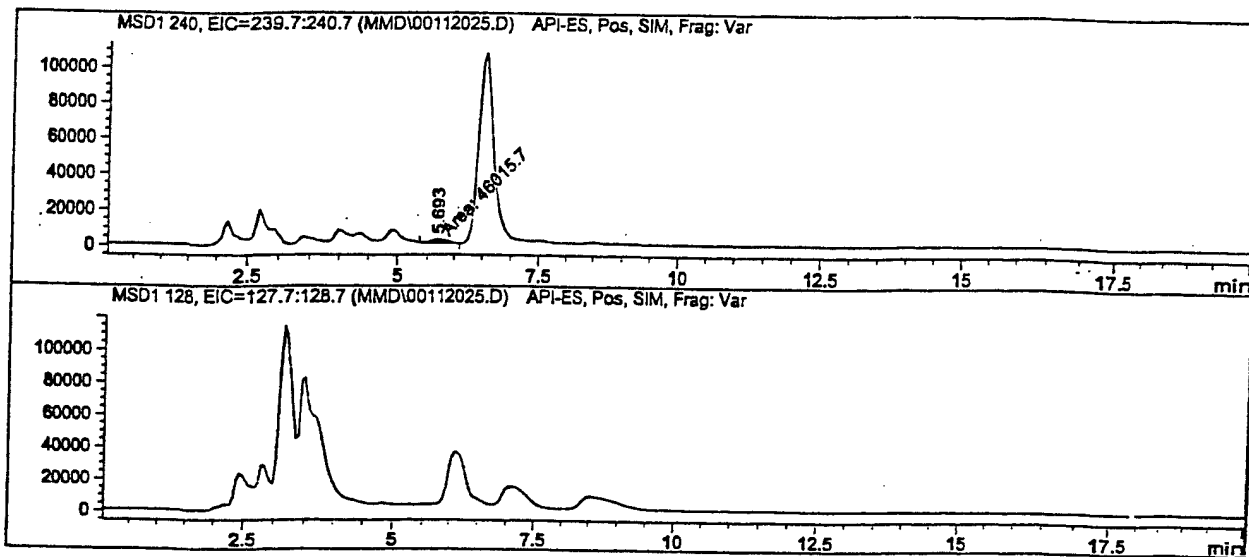
| Peak # | RetTime [min] | Type | Width [min] | Area      | Height    | Area %   |
|--------|---------------|------|-------------|-----------|-----------|----------|
| 1      | 5.642         | MM   | 0.3150      | 3.85919e5 | 2.04201e4 | 100.0000 |

Totals :                    3.85919e5  2.04201e4

=====  
 \*\*\* End of Report \*\*\*

```

=====
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 \*\*\*

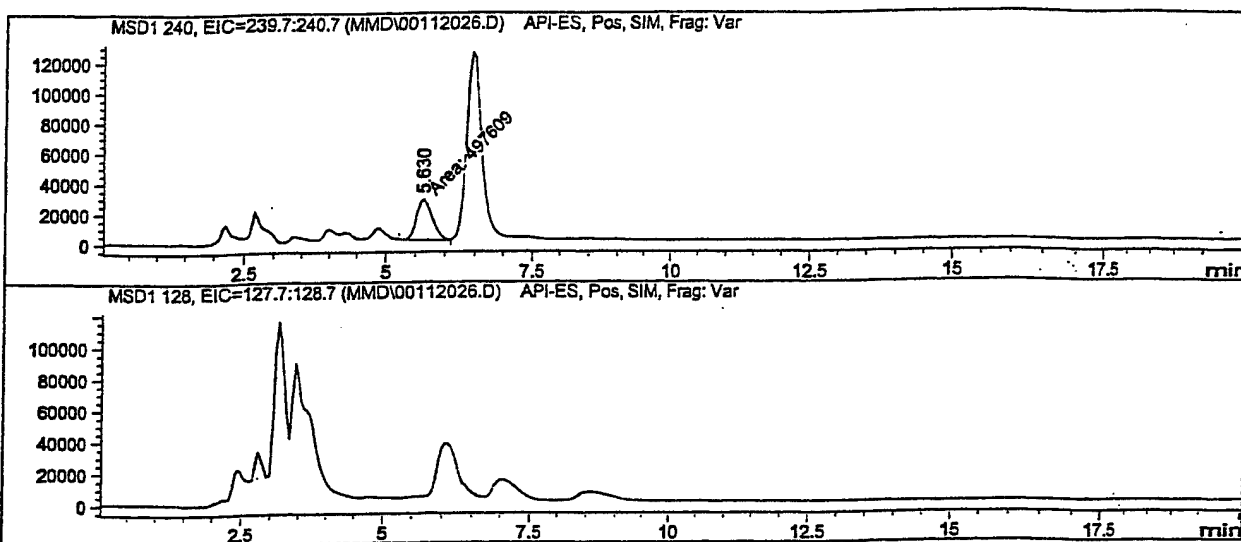


```

=====
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

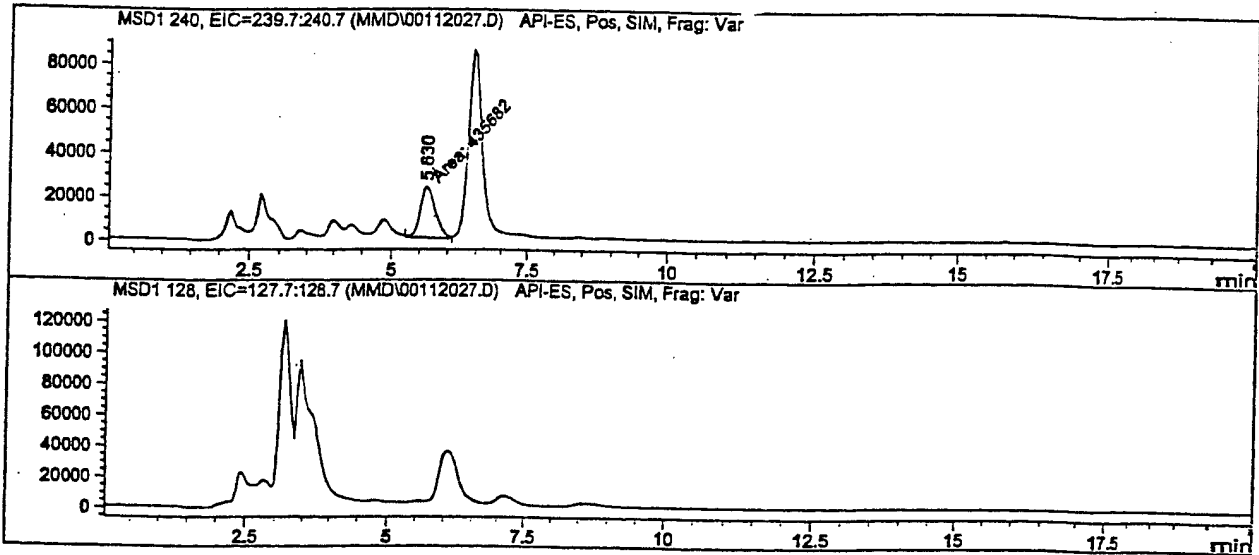
Signal 2: MSD1 128, EIC=127.7:128.7

=====  
 \*\*\* End of Report \*\*\*

```

=====
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

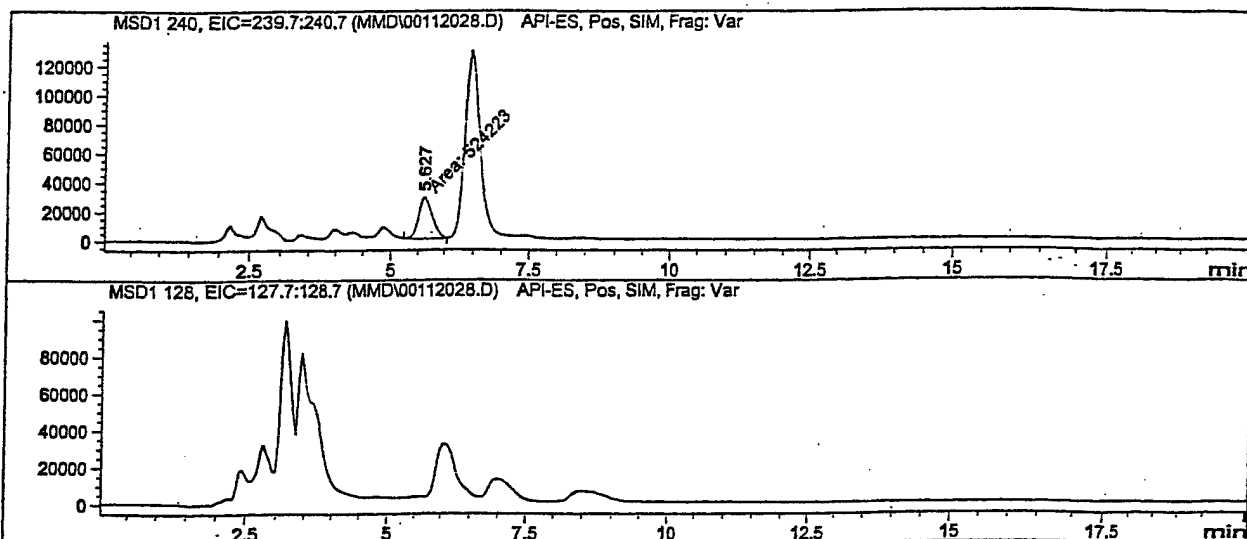
Signal 2: MSD1 128, EIC=127.7:128.7

\*\*\* End of Report \*\*\*

```

=====
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

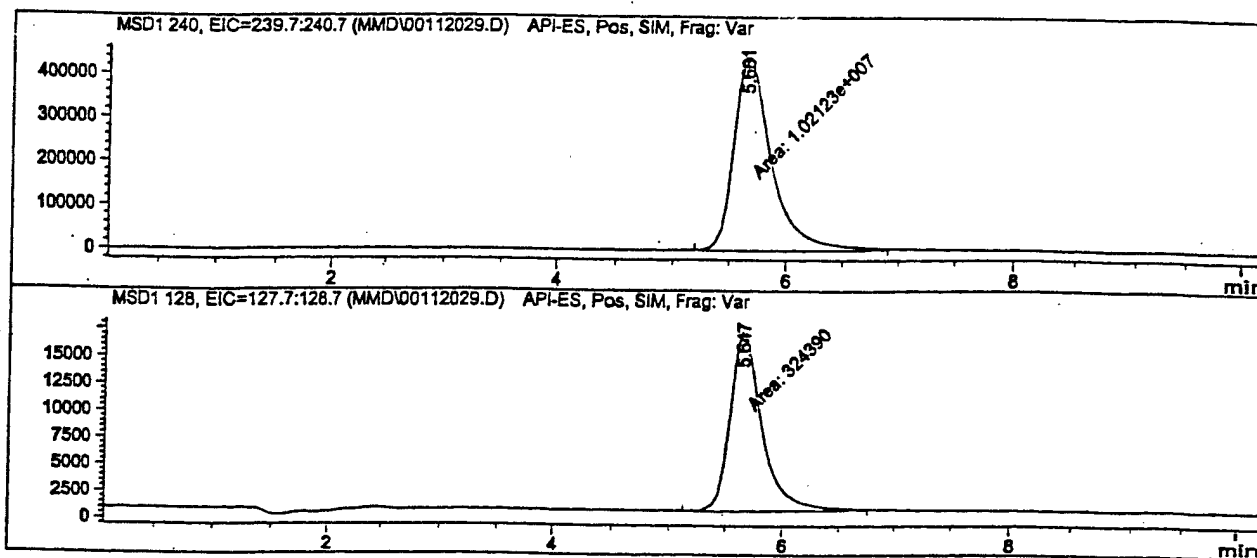
=====  
 \*\*\* End of Report \*\*\*

```

=====
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

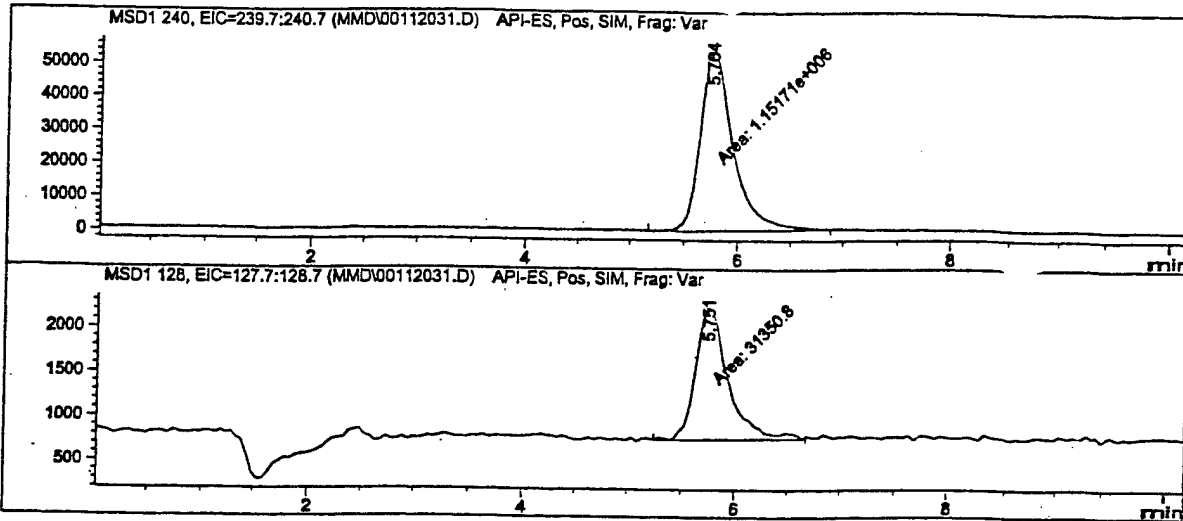
=====  
 \*\*\* End of Report \*\*\*



```

=====
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 ***

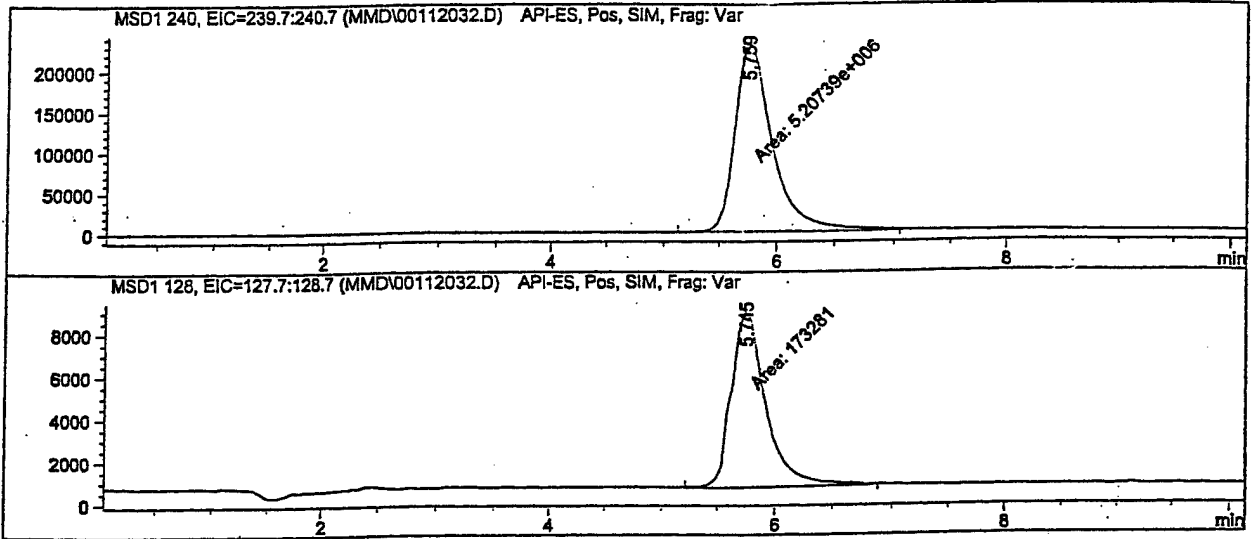
```

```

=====
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

```

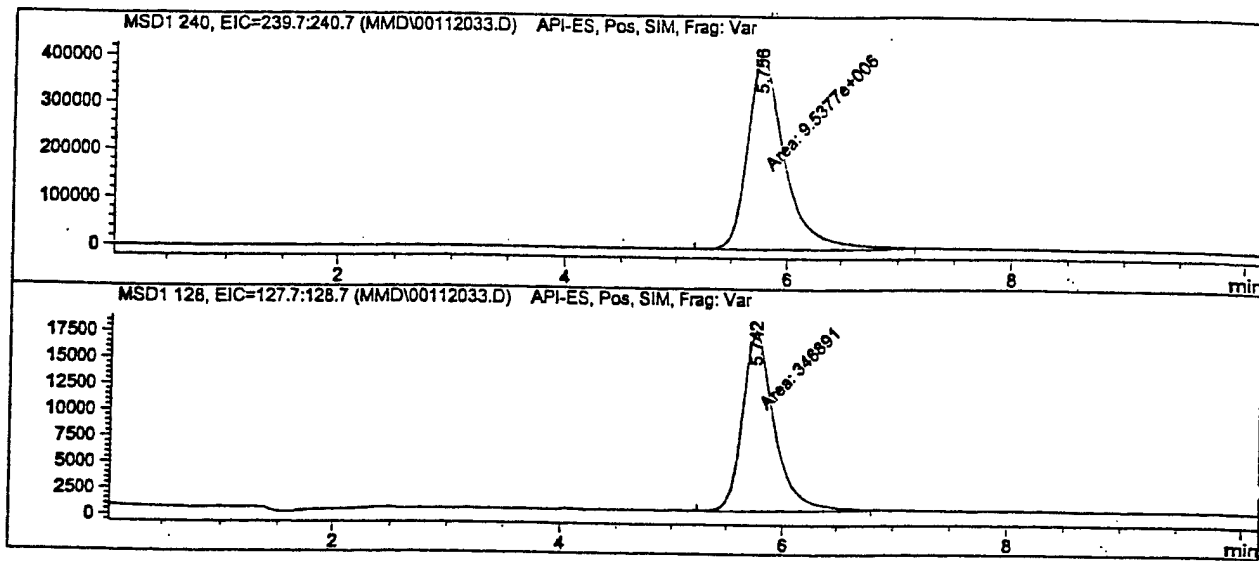
=====
*** End of Report ***

```

```

=====
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 \*\*\*

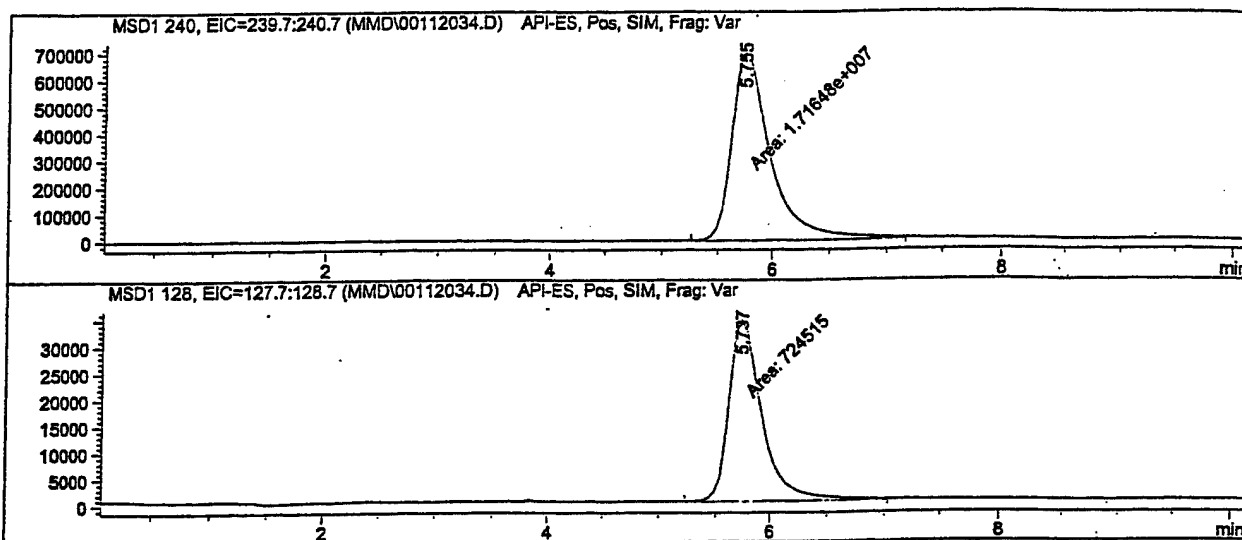


```

=====
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

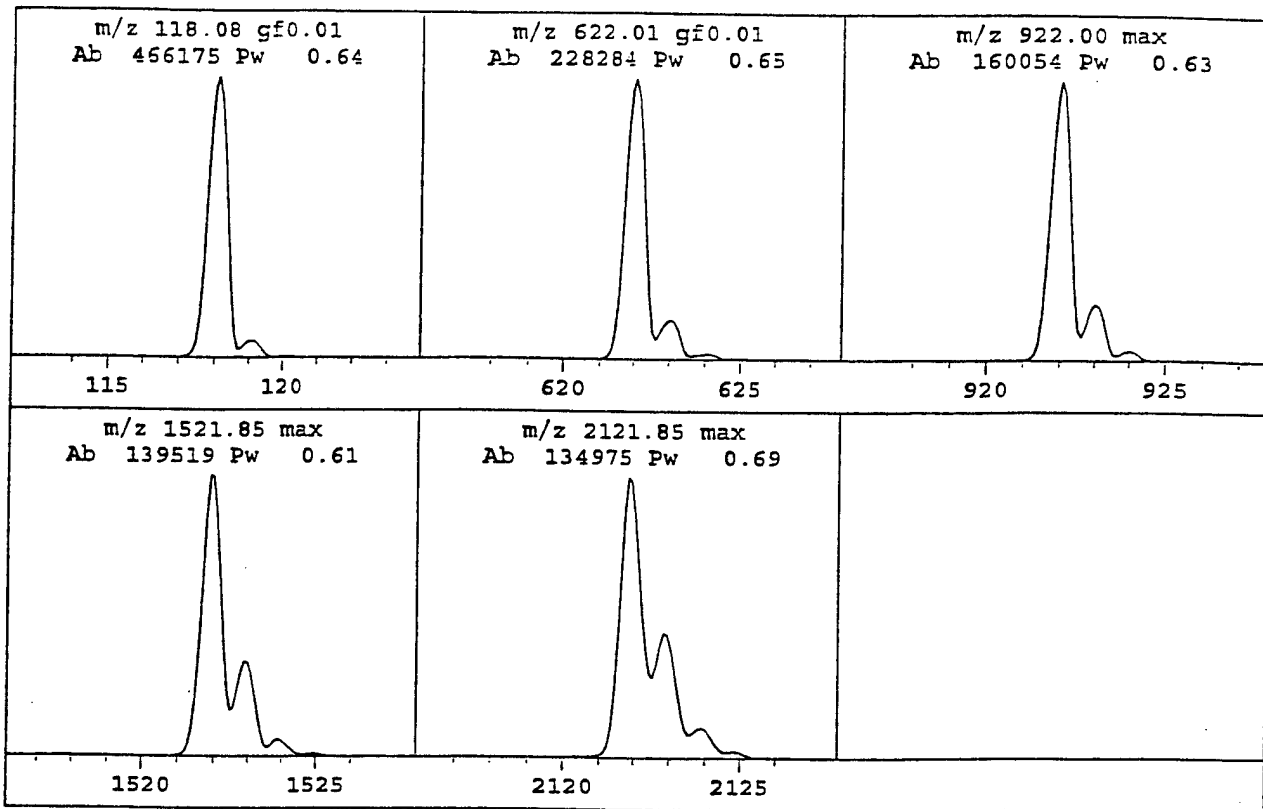
| Peak # | RetTime [min] | Type | Width [min] | Area      | Height    | Area %   |
|--------|---------------|------|-------------|-----------|-----------|----------|
| 1      | 5.737         | MM   | 0.3488      | 7.24515e5 | 3.46145e4 | 100.0000 |

Totals :                      7.24515e5   3.46145e4

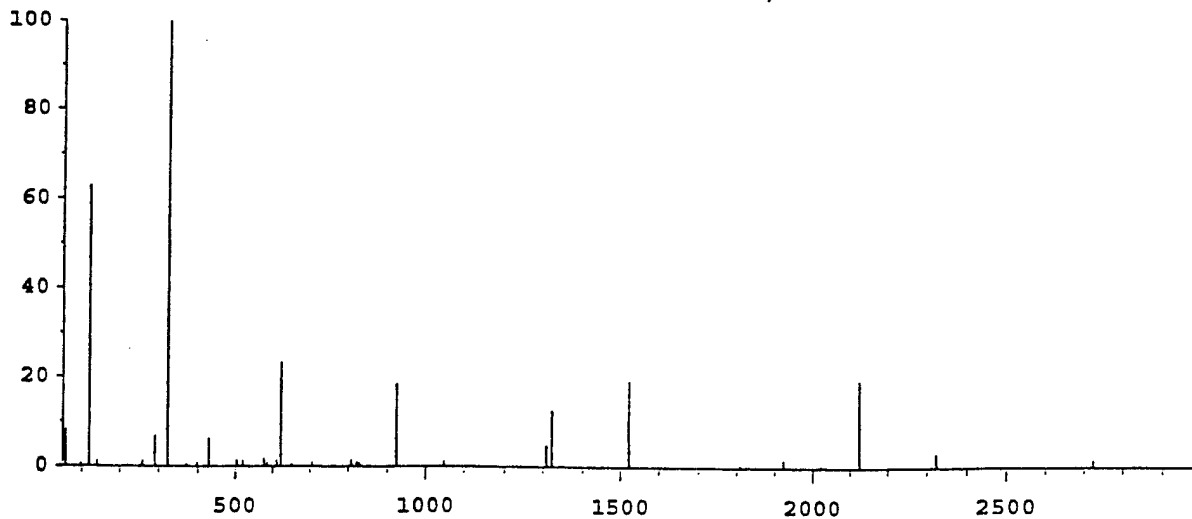
=====  
 \*\*\* End of Report \*\*\*

**BLANK**

**APPENDIX C**  
**ELECTROSPRAY AUTOTUNE REPORT FOR THE LC/MSD**



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

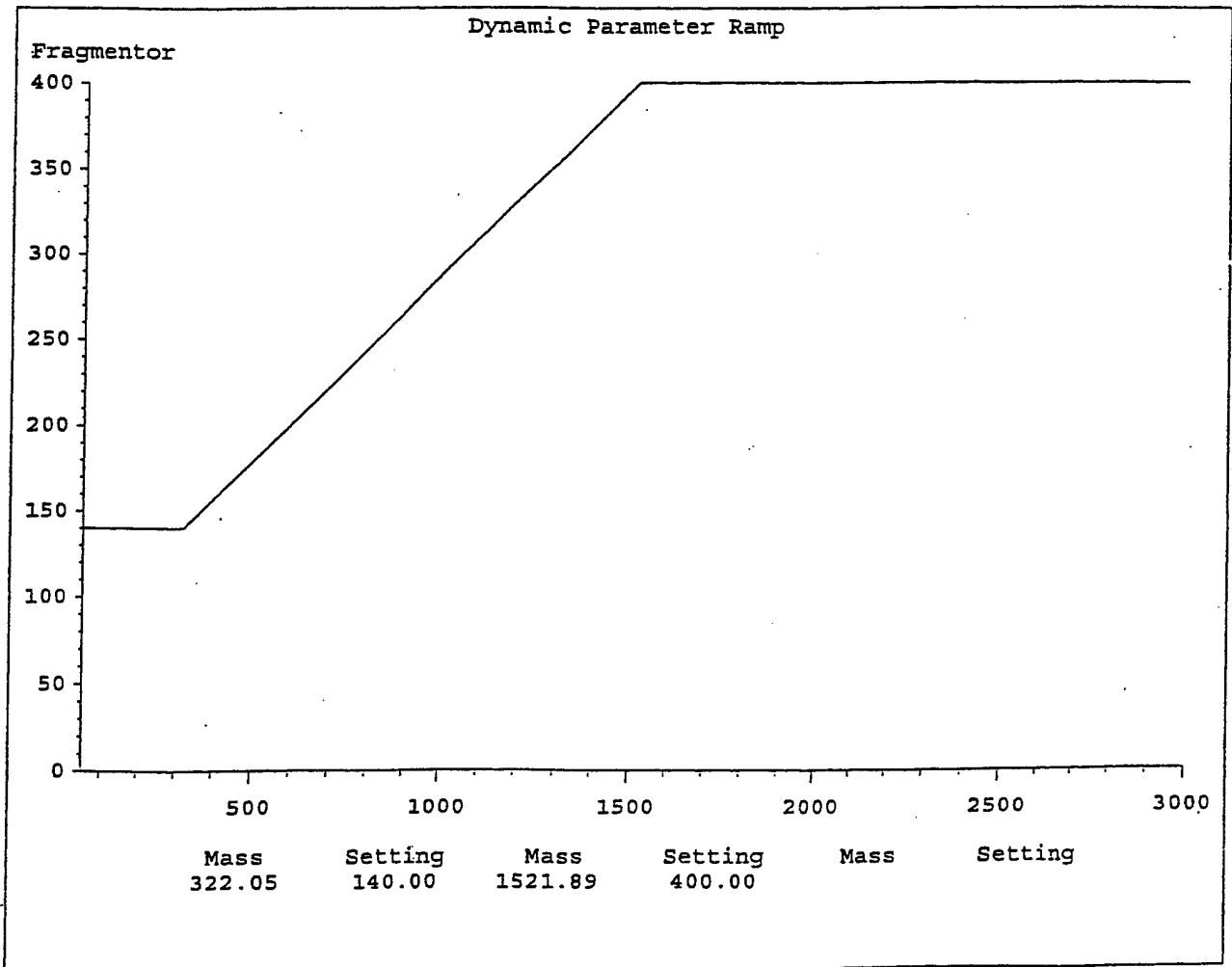
                Energy    3.0
                OpolPeak  300
                OpolKnee  300
Gain       1.0 QuadDC    0.00

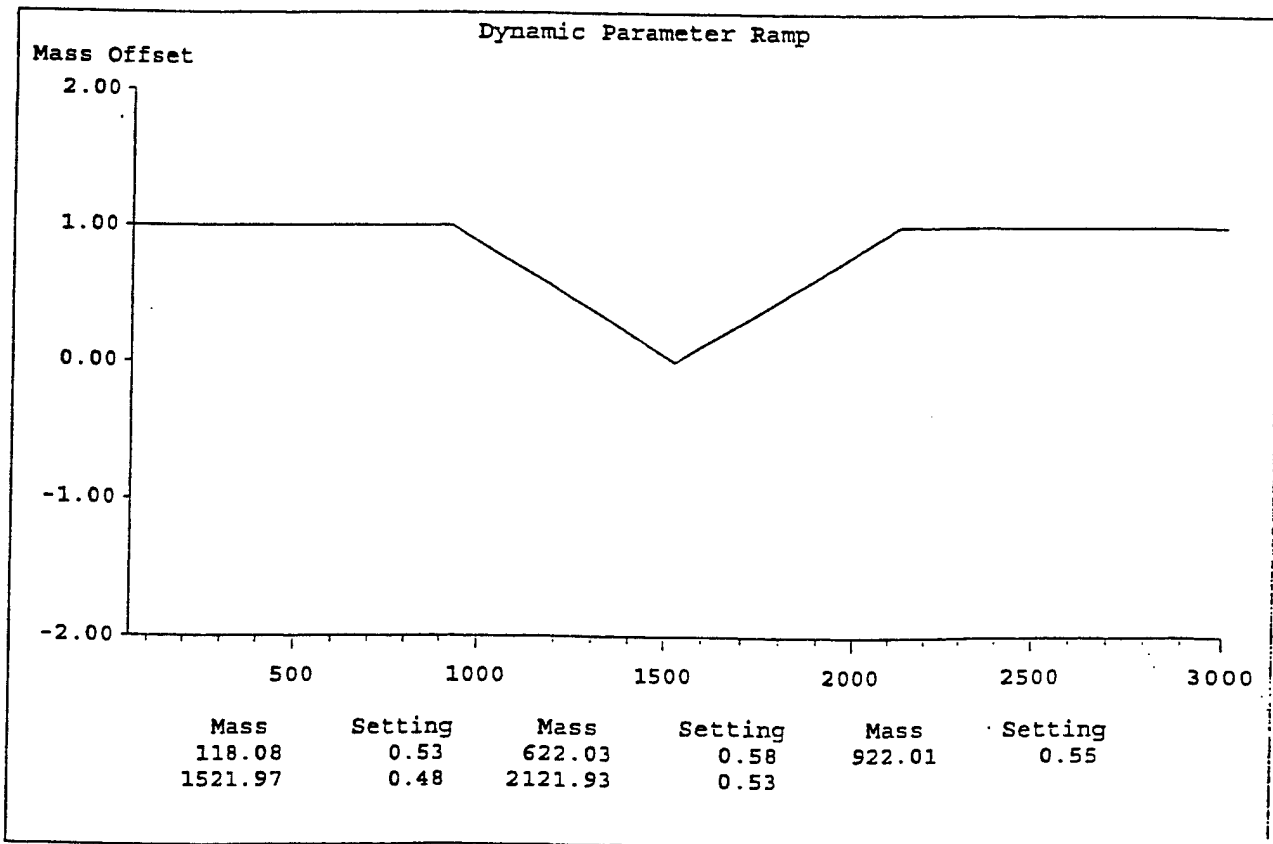
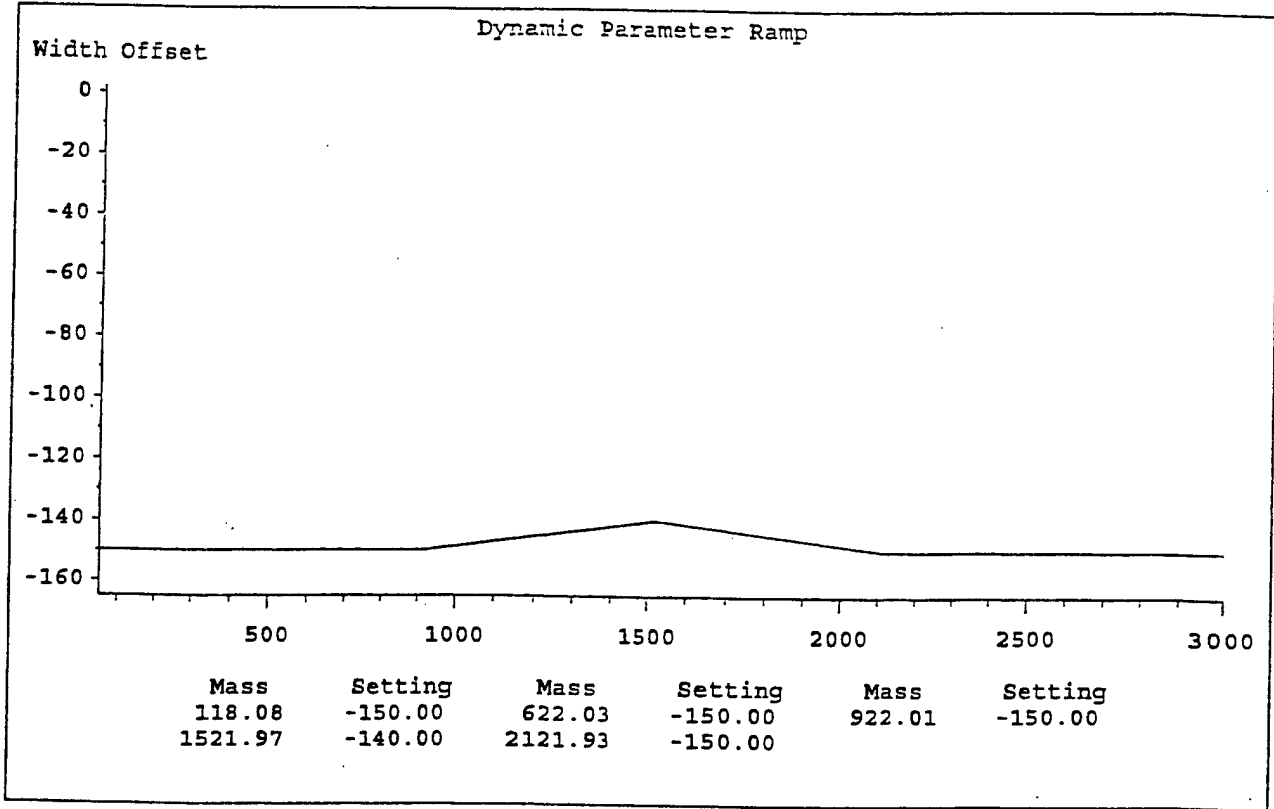
EMV        2508 Samples   8
VCap       4000 Averages  1
                StepSize  0.10

Status:
CapCur    47 DryingGas  6.0
ChamCur   0.48 Gas Temp  300

Quad Temp  99 Neb Pres   50

```





**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.<br>(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. =  $5.11e-15 * (\text{signal})^2 + 7.56e-8 * \text{signal}$

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   |

**Cal Curve 3/26**

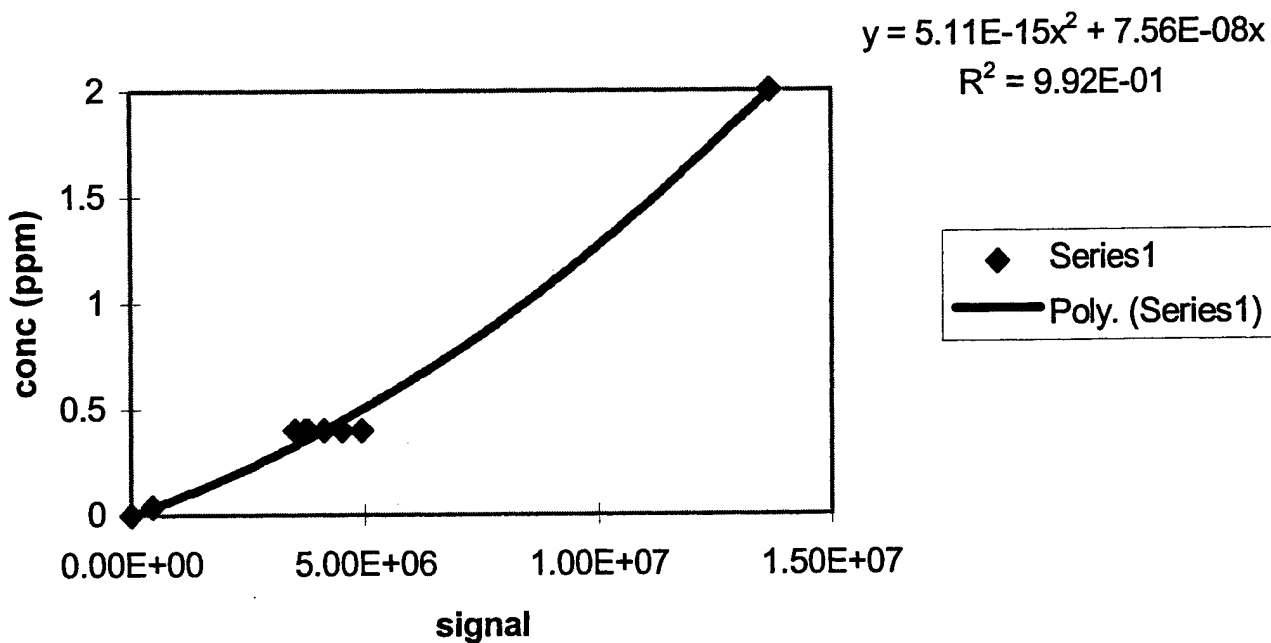
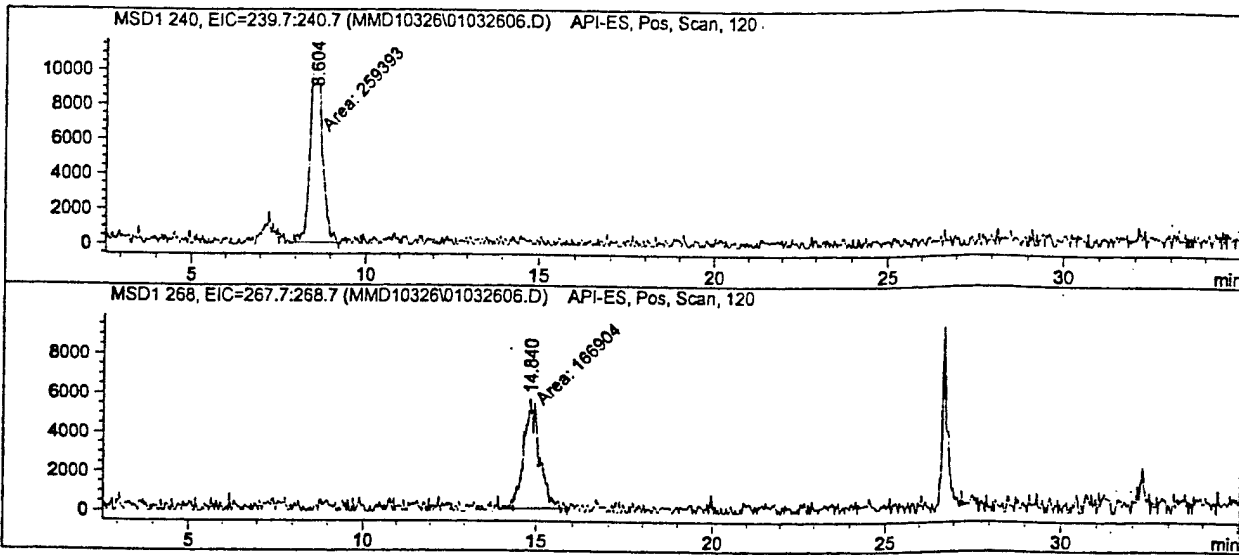


Figure D-1. Calibration Curve for EA-2192 using LC/MS, Using a Polynomial Fit with Forced Zero Intercept. The point for the 1 ppm standard was excluded because it was significantly off of the calibration curve.

```

=====
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 |

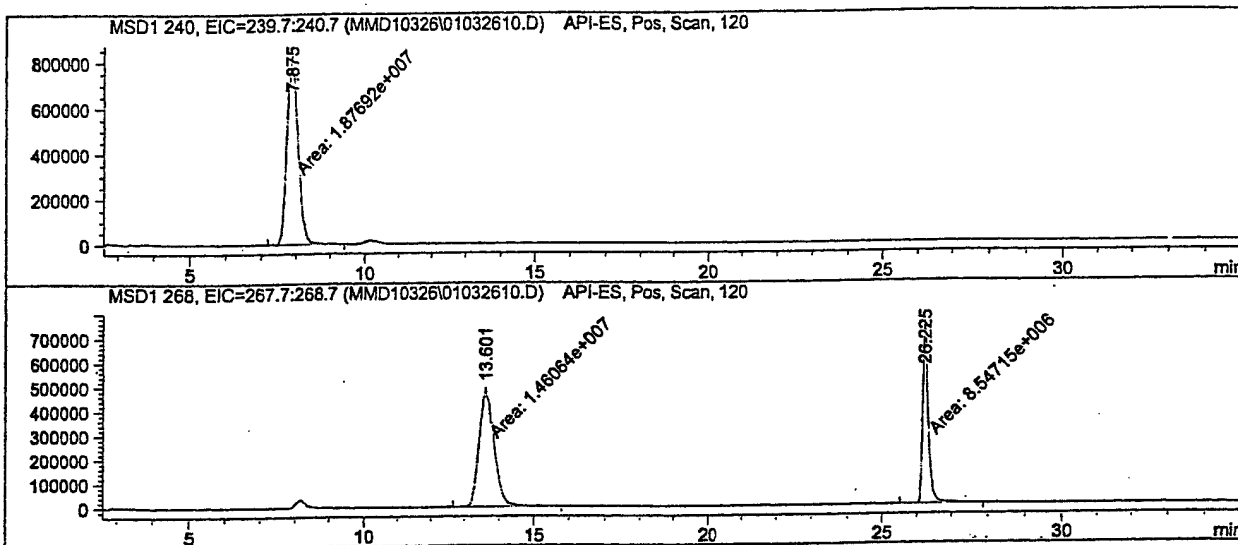
Totals :                    1.66904e5 5663.35010

=====  
 \*\*\* End of Report \*\*\*

```

=====
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

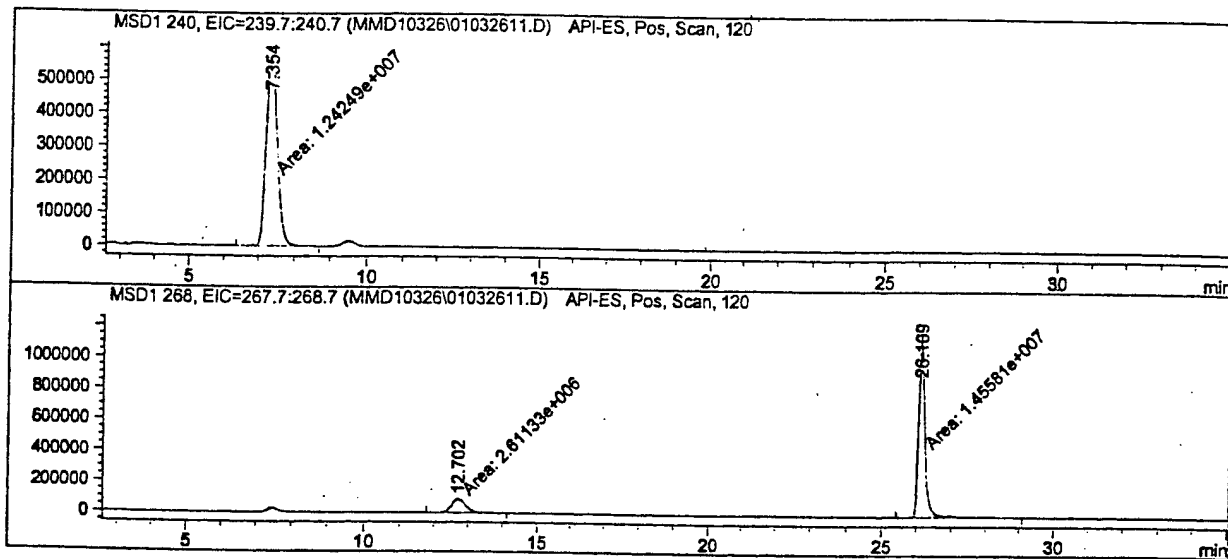
| Peak # | RetTime [min] | Type | Width [min] | Area      | Height    | Area %  |
|--------|---------------|------|-------------|-----------|-----------|---------|
| 1      | 13.601        | MM   | 0.4877      | 1.46064e7 | 4.99119e5 | 63.0849 |
| 2      | 26.225        | MM   | 0.1826      | 8.54715e6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

Totals :                                    2.31535e7   1.27940e6

```

=====
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

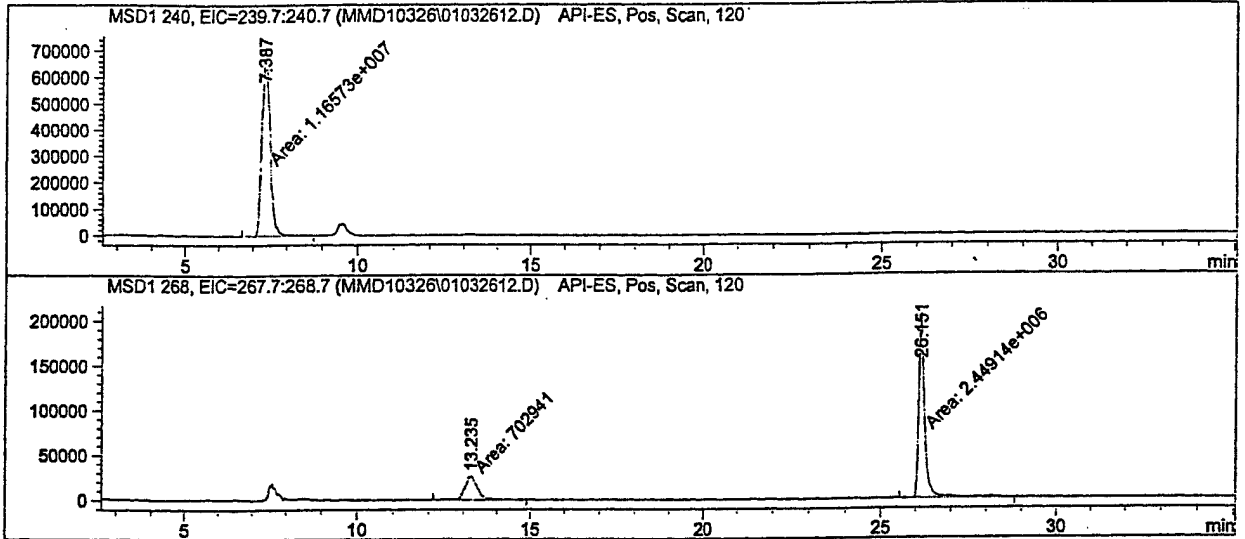
| Peak # | RetTime [min] | Type | Width [min] | Area      | Height    | Area %  |
|--------|---------------|------|-------------|-----------|-----------|---------|
| 1      | 12.702        | MM   | 0.4652      | 2.61133e6 | 9.35620e4 | 15.2091 |
| 2      | 26.109        | MM   | 0.1988      | 1.45581e7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

Totals :                      1.71694e7  1.31387e6

```

=====
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

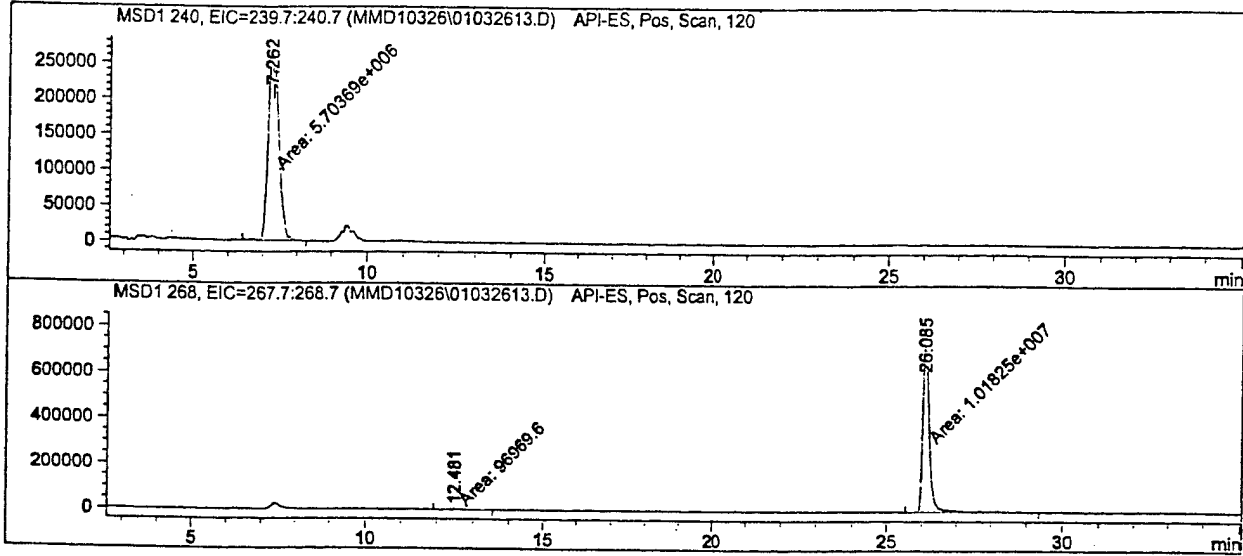
| Peak # | RetTime [min] | Type | Width [min] | Area      | Height    | Area %  |
|--------|---------------|------|-------------|-----------|-----------|---------|
| 1      | 13.235        | MM   | 0.4485      | 7.02941e5 | 2.61225e4 | 22.3008 |
| 2      | 26.151        | MM   | 0.1953      | 2.44914e6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

Totals :                    3.15209e6  2.35166e5

```

=====
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

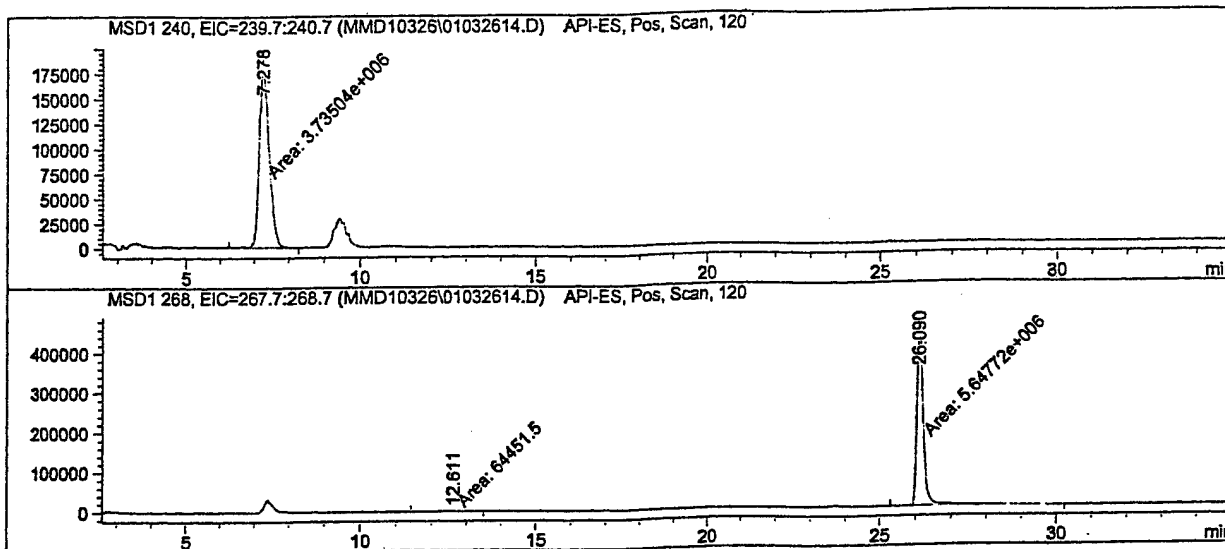
| Peak # | RetTime [min] | Type | Width [min] | Area      | Height     | Area %  |
|--------|---------------|------|-------------|-----------|------------|---------|
| 1      | 12.481        | MM   | 0.4905      | 9.69696e4 | 3294.71582 | 0.9433  |
| 2      | 26.085        | MM   | 0.2041      | 1.01825e7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

Totals :                                    1.02795e7   8.34968e5

```

=====
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

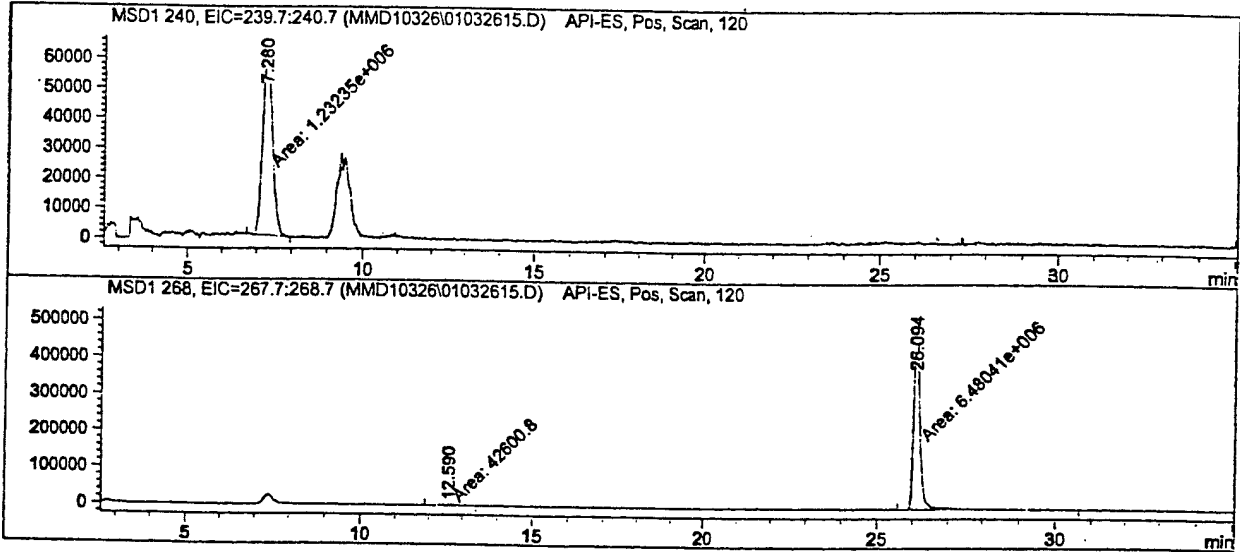
| Peak # | RetTime [min] | Type | Width [min] | Area      | Height     | Area %  |
|--------|---------------|------|-------------|-----------|------------|---------|
| 1      | 12.611        | MM   | 0.4728      | 6.44515e4 | 2272.05347 | 1.1283  |
| 2      | 26.090        | MM   | 0.1927      | 5.64772e6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

Totals :                            5.71218e6  4.90807e5

```

=====
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 |

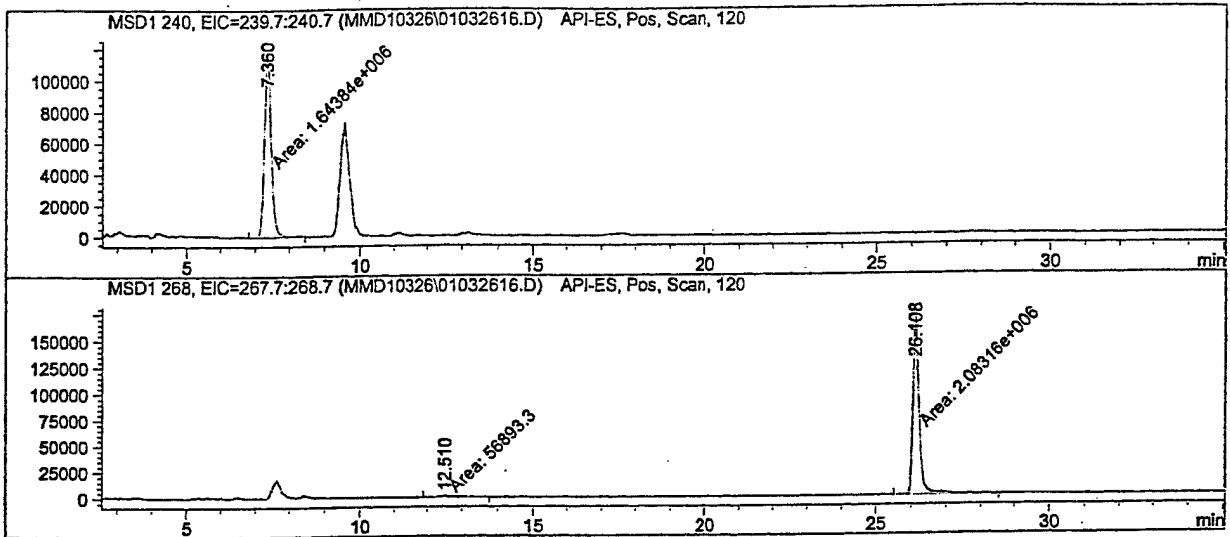
Totals :                            6.52301e6  5.18615e5



```

=====
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 [min] | Type | Width [min] | Area      | Height    | Area %   |
|--------|---------------|------|-------------|-----------|-----------|----------|
| 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

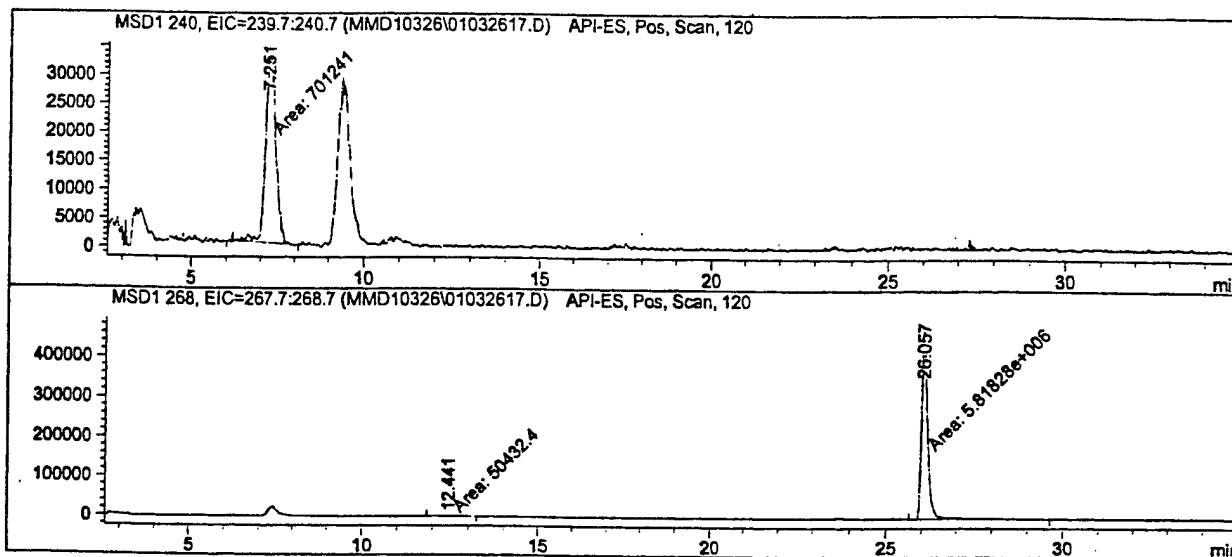
| Peak # | RetTime [min] | Type | Width [min] | Area      | Height     | Area %  |
|--------|---------------|------|-------------|-----------|------------|---------|
| 1      | 12.510        | MM   | 0.5801      | 5.68932e4 | 1634.66650 | 2.6585  |
| 2      | 26.108        | MM   | 0.1974      | 2.08316e6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

Totals : 2.14005e6 1.77545e5

```

=====
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

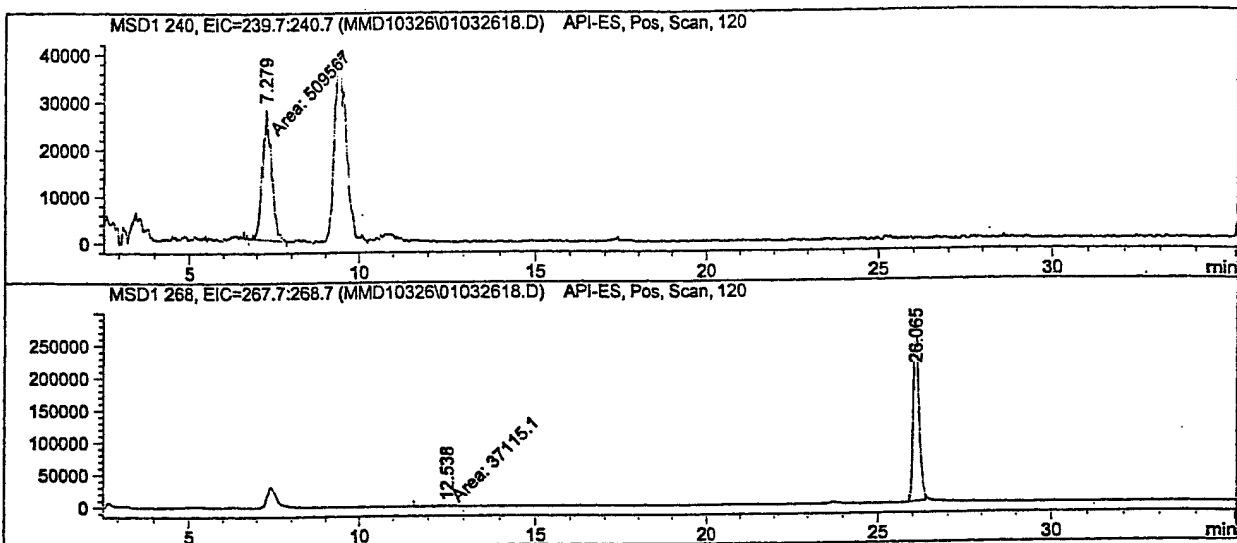
| Peak # | RetTime [min] | Type | Width [min] | Area      | Height     | Area %  |
|--------|---------------|------|-------------|-----------|------------|---------|
| 1      | 12.441        | MM   | 0.4797      | 5.04324e4 | 1752.39929 | 0.8593  |
| 2      | 26.057        | MM   | 0.1952      | 5.81828e6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

Totals :                    5.86872e6  4.98514e5

```

=====
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

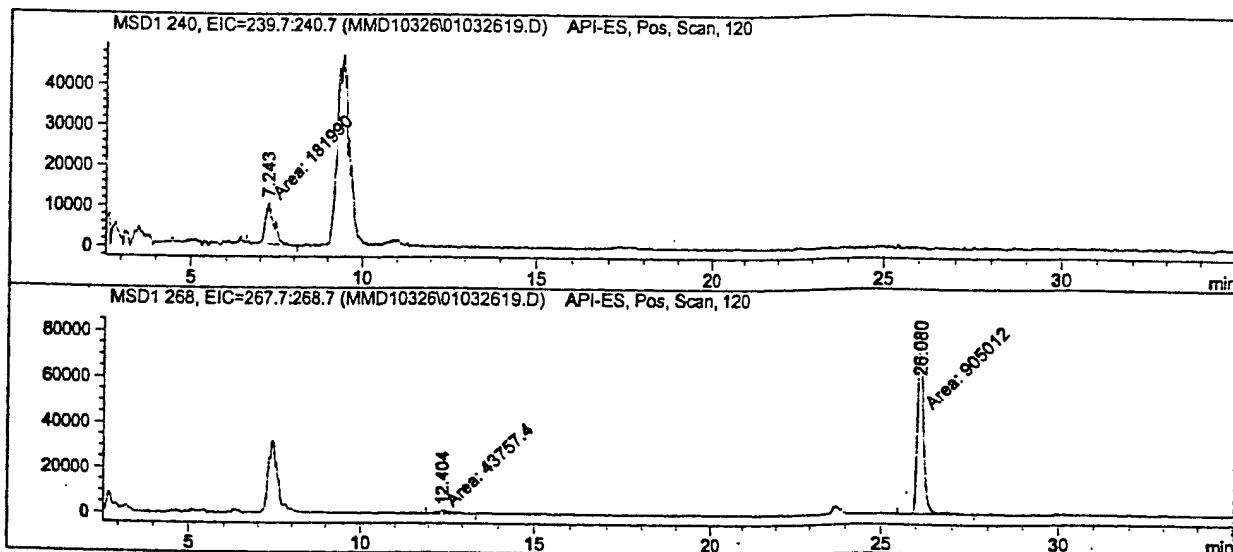
| Peak # | RetTime [min] | Type | Width [min] | Area      | Height     | Area %  |
|--------|---------------|------|-------------|-----------|------------|---------|
| 1      | 12.538        | MM   | 0.4234      | 3.71151e4 | 1461.04126 | 1.1833  |
| 2      | 26.065        | BB   | 0.1578      | 3.09945e6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

Totals :                      3.13656e6    2.88377e5

```

=====
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 |

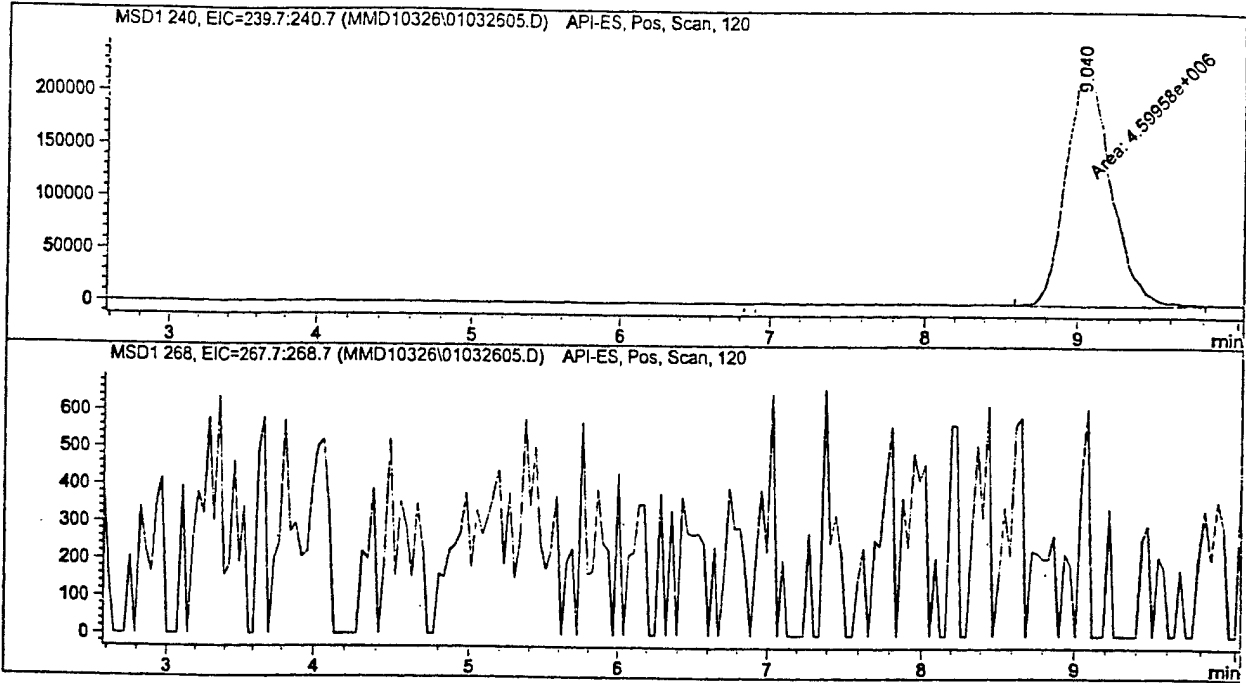
Totals :                    1.81990e5  1.03480e4

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





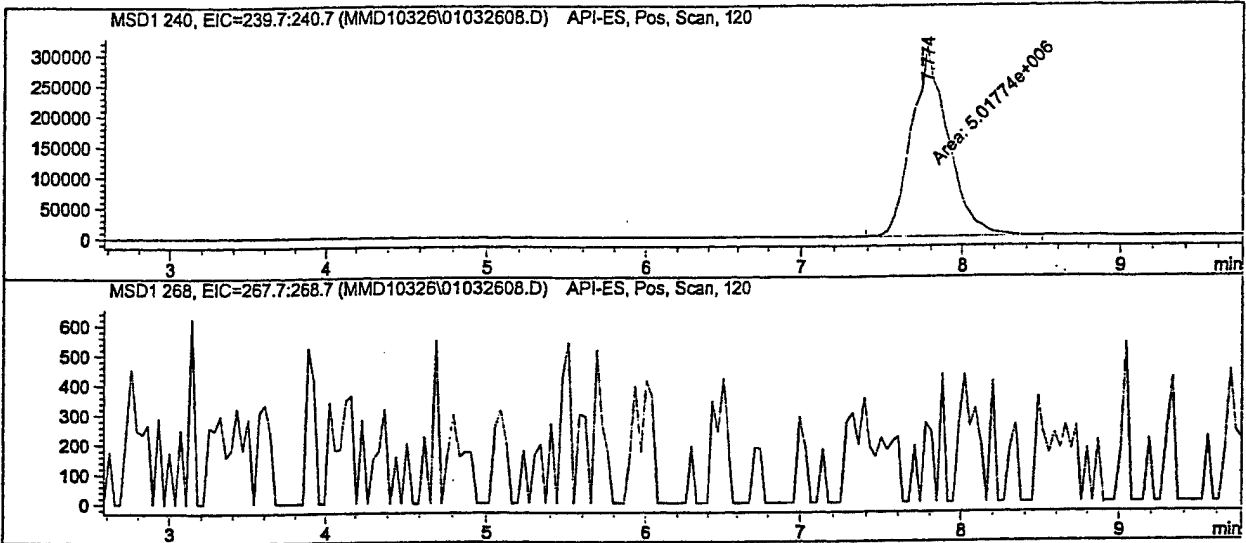
\*\*\* End of Report \*\*\*

```

=====
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 [min] | Type | Width [min] | Area      | Height    | Area %   |
|--------|---------------|------|-------------|-----------|-----------|----------|
| 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 ***
=====

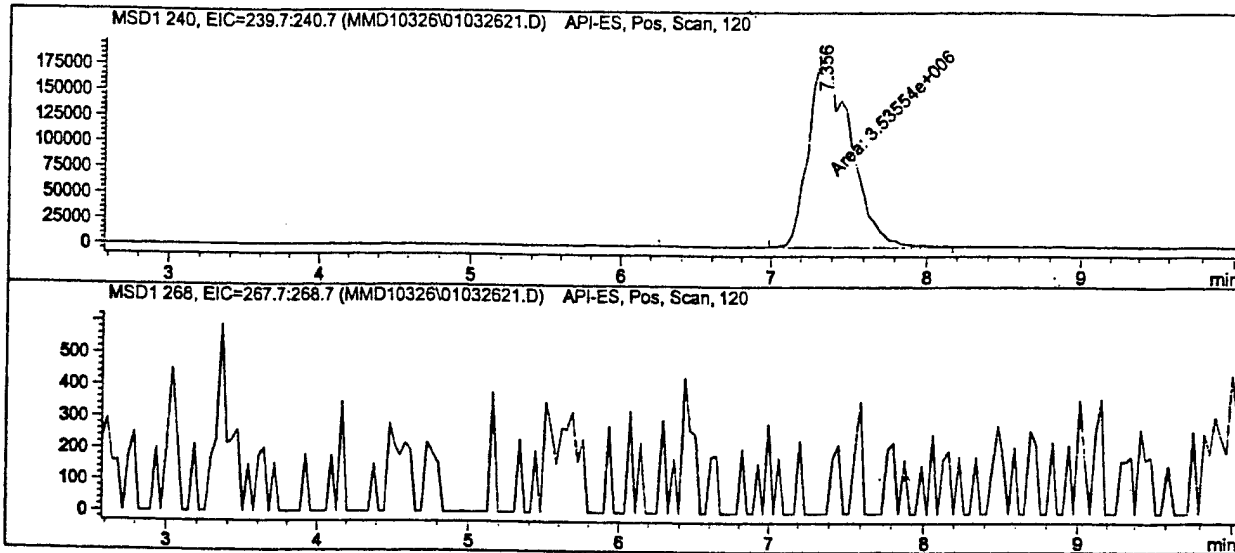
```

```

=====
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 ***

```

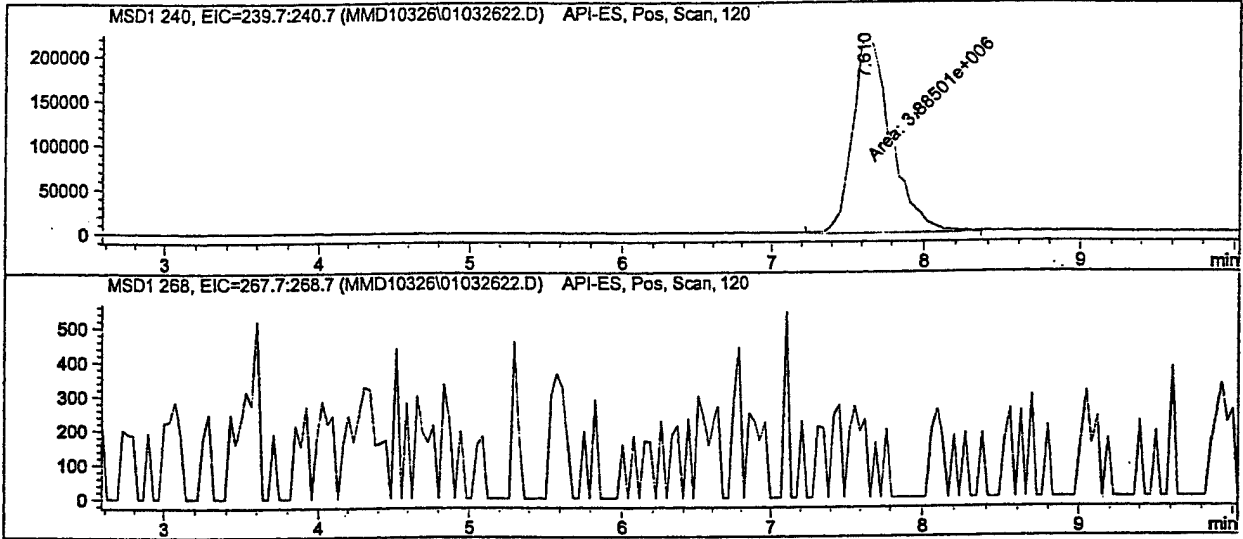


```

=====
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 ***
=====

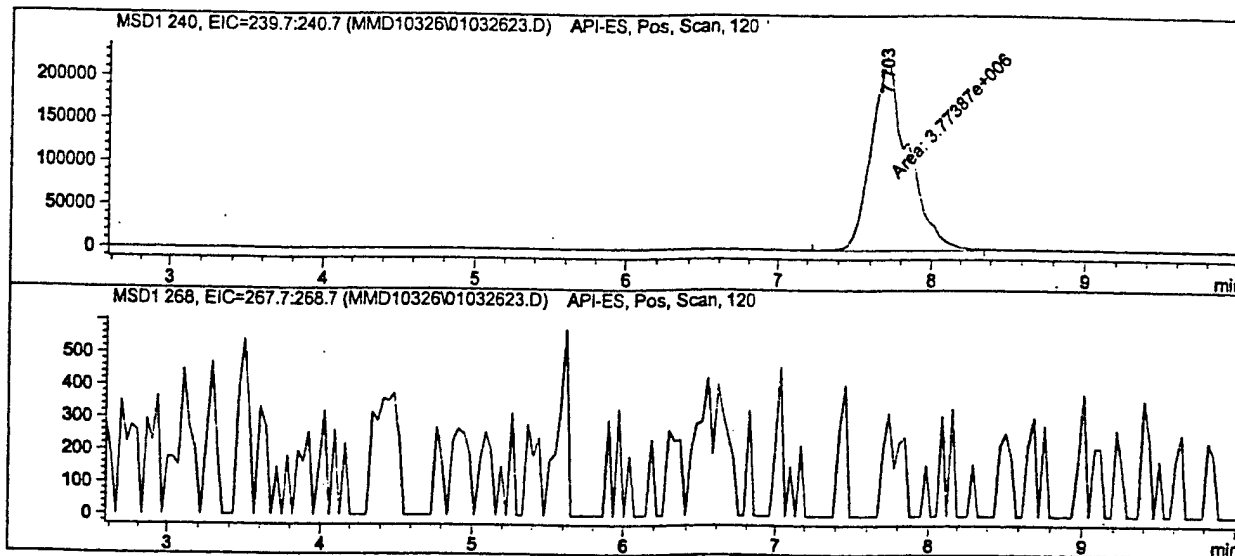
```

```

=====
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
```

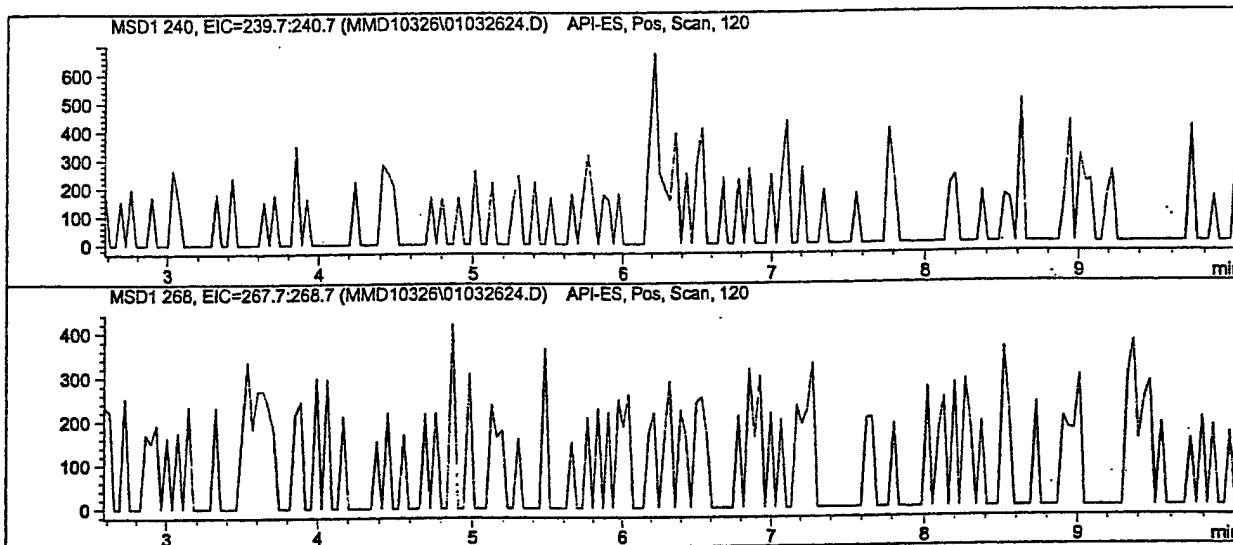
Signal 2: MSD1 268, EIC=267.7:268.7

```

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

```

=====  
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

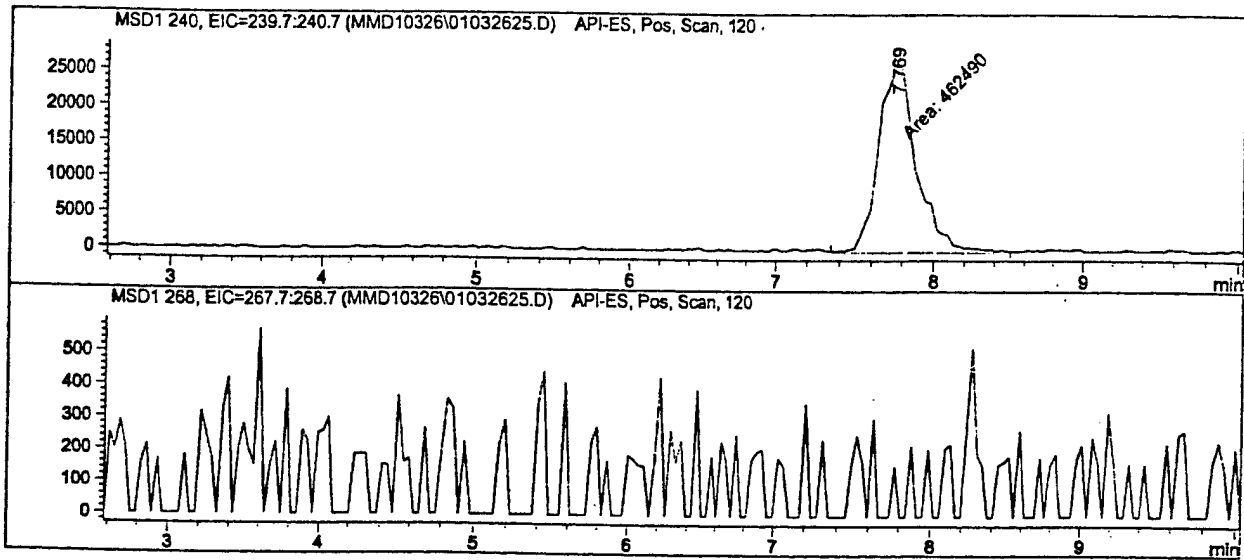
No peaks found

=====  
\*\*\* End of Report \*\*\*  
=====

```

=====
Injection Date : 3/27/2001 2:57:31 AM      Seq. Line : 16
Sample Name    : 40 ppb 2192                Location  : Vial 2
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      | 7.769         | MM   | 0.2756      | 4.62490e5 | 2.79662e4 | 100.0000 |

Totals :                    4.62490e5  2.79662e4

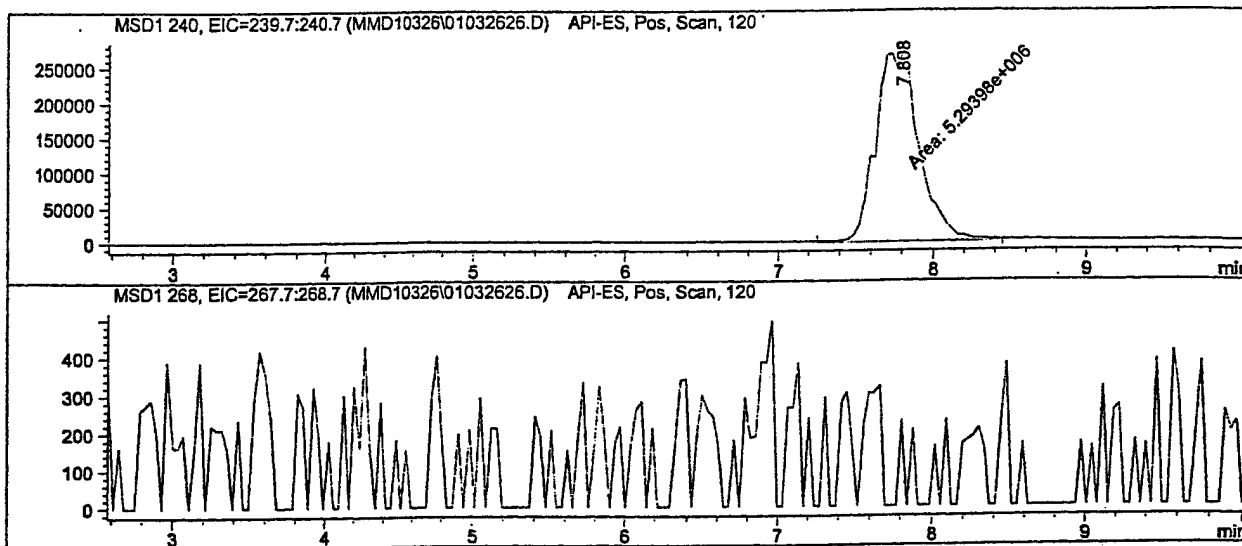
Signal 2: MSD1 268, EIC=267.7:268.7

\*\*\* End of Report \*\*\*

```

=====
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

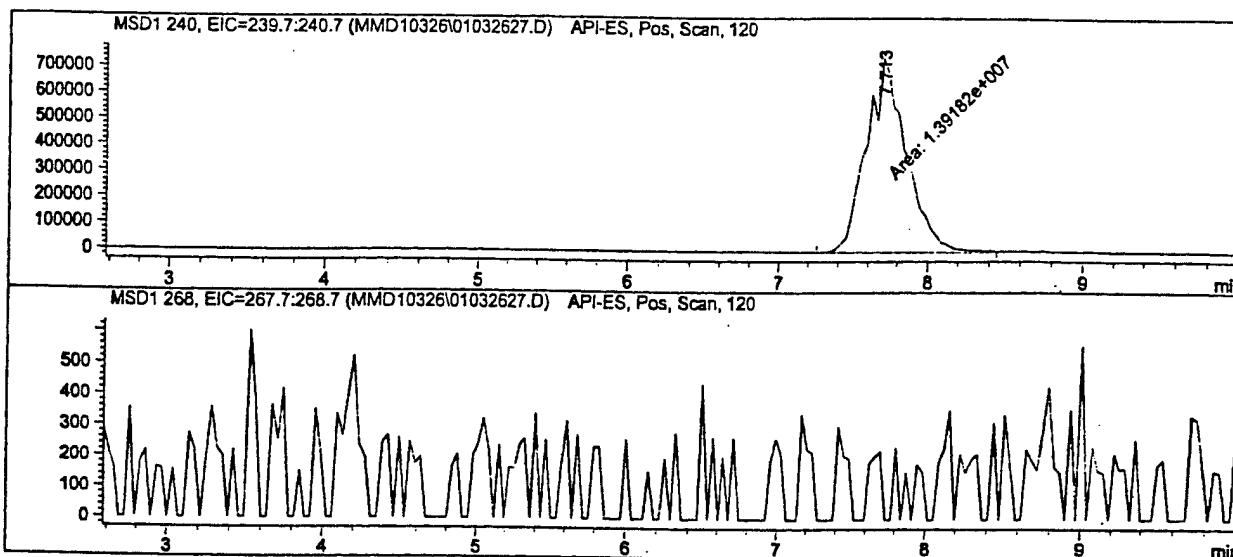
=====  
 \*\*\* End of Report \*\*\*

```

=====
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
```

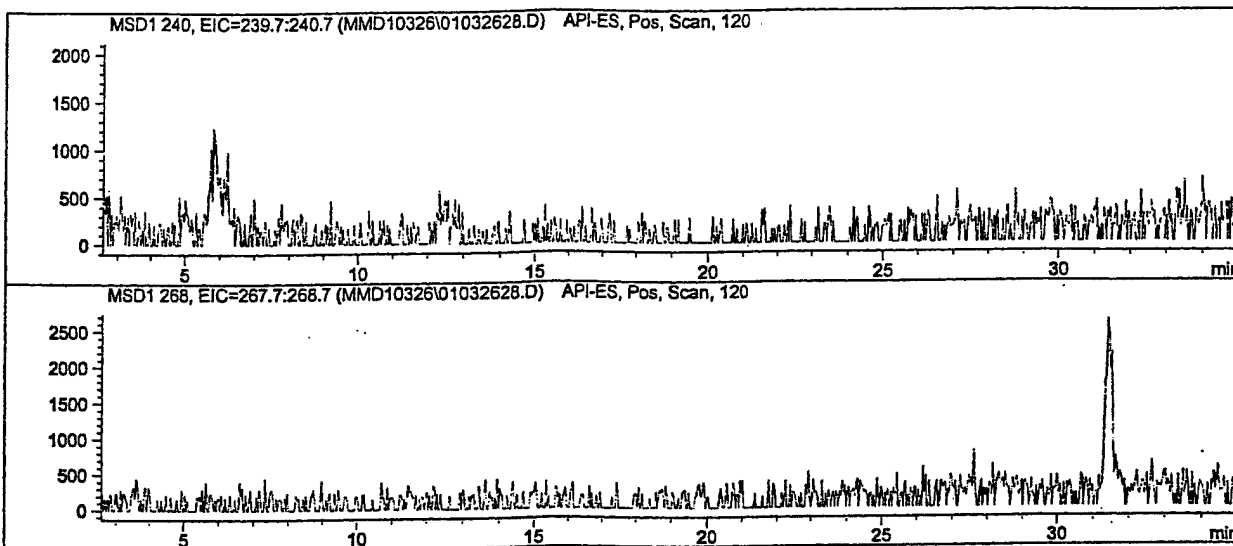
Signal 2: MSD1 268, EIC=267.7:268.7

```

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

```

=====  
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

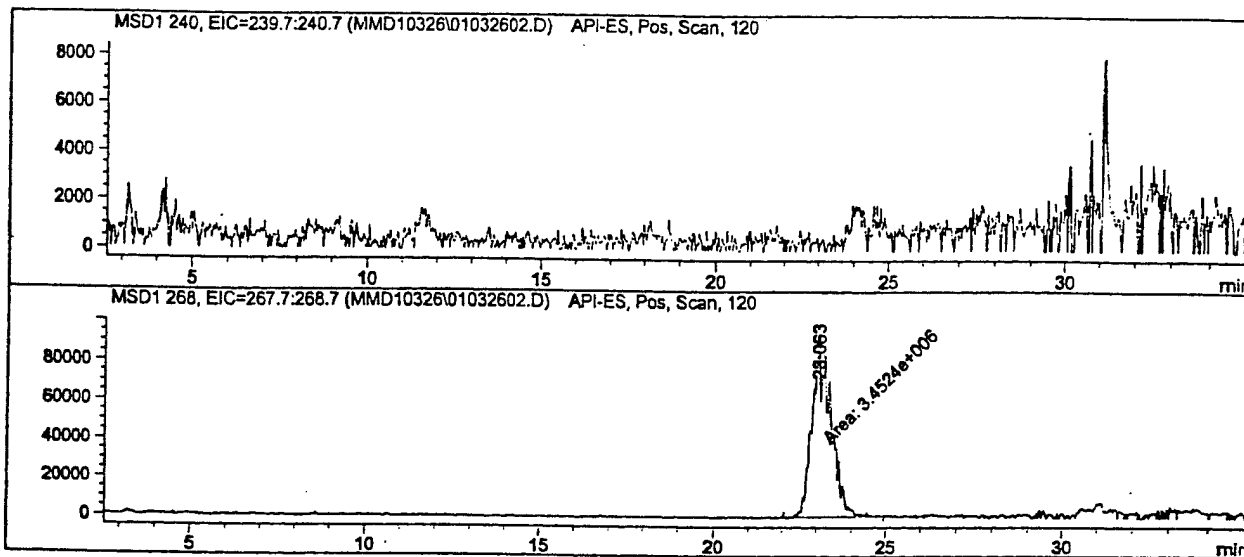
No peaks found

=====  
\*\*\* End of Report \*\*\*  
=====

```

=====
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 [min] | Type | Width [min] | Area      | Height    | Area %   |
|--------|---------------|------|-------------|-----------|-----------|----------|
| 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  $^{31}\text{P}$  NMR**

Tabulated data:

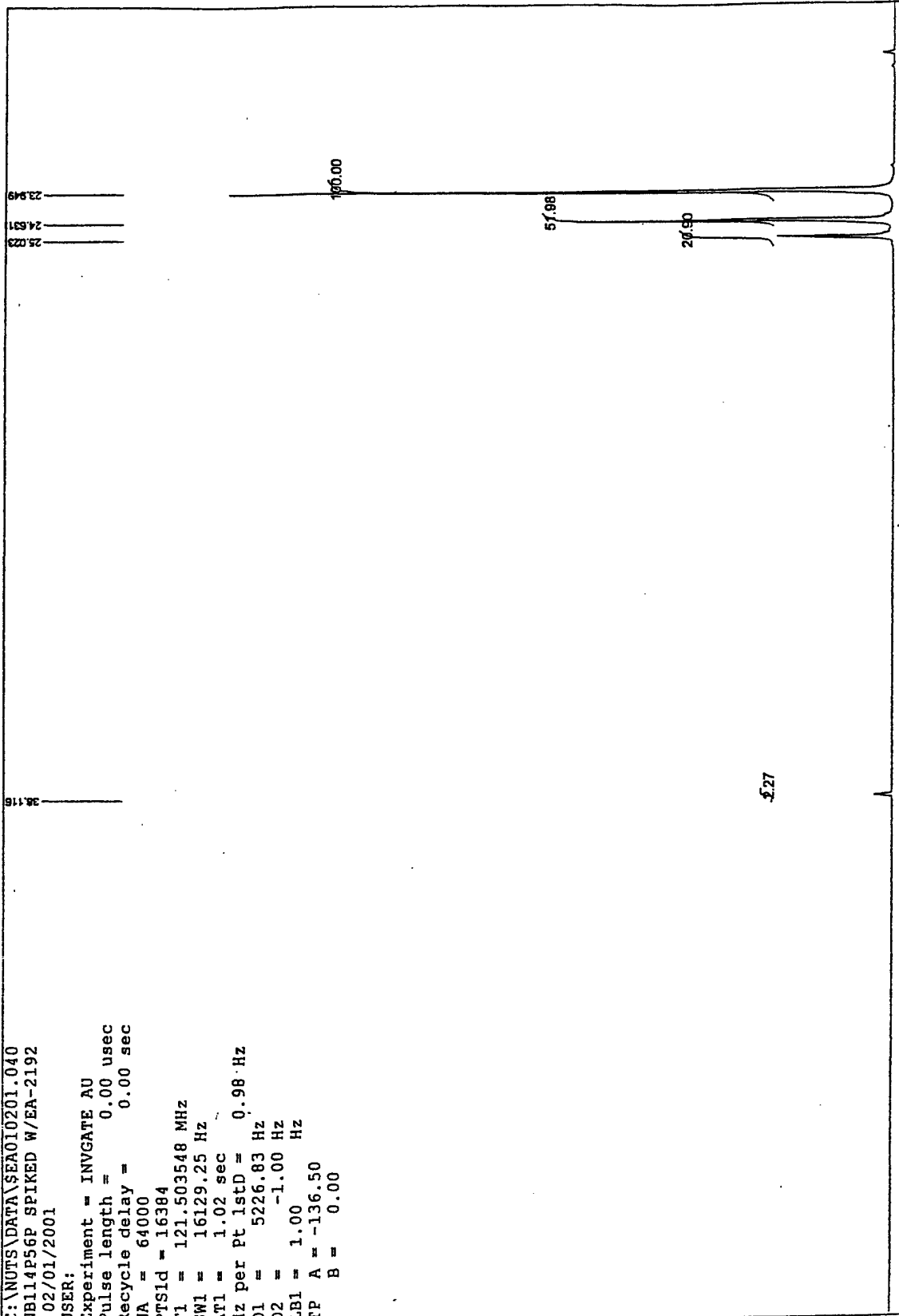
| Date      | Data File    | # of Days | Integral |       |       |        | Combined Area | EA2192/<br>Combined Area |
|-----------|--------------|-----------|----------|-------|-------|--------|---------------|--------------------------|
|           |              |           | 1        | 2     | 3     | 4      |               |                          |
| 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/<br>Combined Area | log EA2192/<br>Combined Area | Norm. to<br>Comb.<br>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\SEA010201.040  
NB114P56P SPIKED W/EA-2192  
02/01/2001

USER:

Experiment = INVGATE AU  
Pulse length = 0.00 usec  
Recycle delay = 0.00 sec  
NA = 64000  
PTSID = 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



|                            |              |                           |                         |
|----------------------------|--------------|---------------------------|-------------------------|
| NB114P56P SPIKED W/EA-2192 |              | USER: -- DATE: 02/01/2001 |                         |
| FL: 121.504                | SW1: 16129   | OF1: 5226.8               | PTSID: 16384            |
| EX: INVGATE AU             | PW: 0.0 usec | NA: 64000                 | LR: 1.0                 |
|                            | PD: 0.0 sec  |                           | WinNuts - SEA010201.040 |

C:\NUTS\DATA\SEA010201.045  
 NB114P56P WITH D2O INSERT AND H3PO4

USER:

Experiment = INVGATE AU  
 Pulse length = 0.00 usec  
 Recycle delay = 0.00 sec

NA = 128

PTSId = 16384

F1 = 121.503548 MHz

SW1 = 16129.25 Hz

AT1 = 1.02 sec

Hz per Pt 1stD = 0.98 Hz

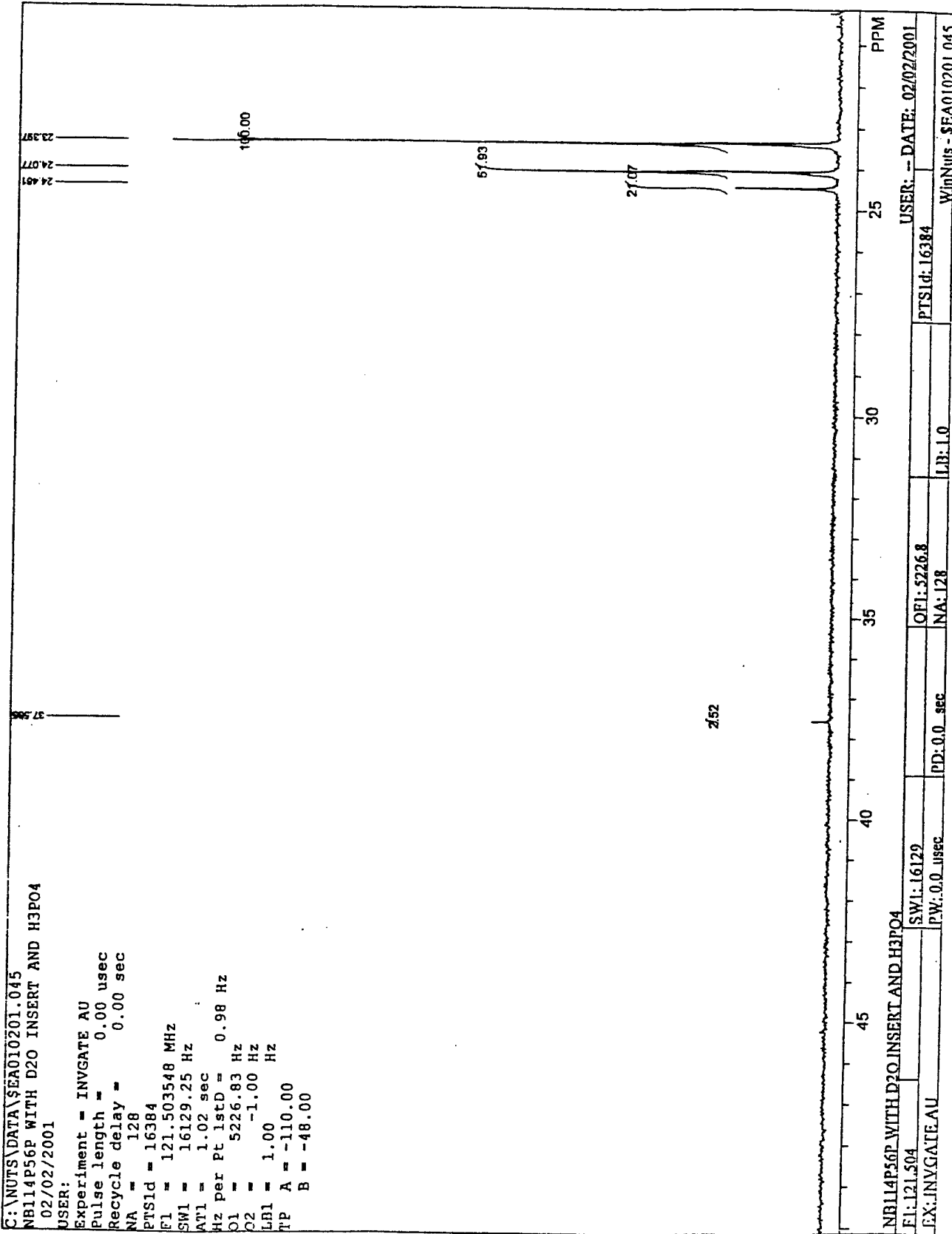
O1 = 5226.83 Hz

O2 = -1.00 Hz

LR1 = 1.00

TP A = -110.00

B = -48.00



NB114P56P WITH D2O INSERT AND H3PO4

F1: 121.504

SW1: 16129

PW: 0.0 usec

EX: INVGATE AU

PD: 0.0 sec

QF1: 5226.8

NA: 128

IR: 1.0

PTSId: 16384

USER: -- DATE: 02/02/2001

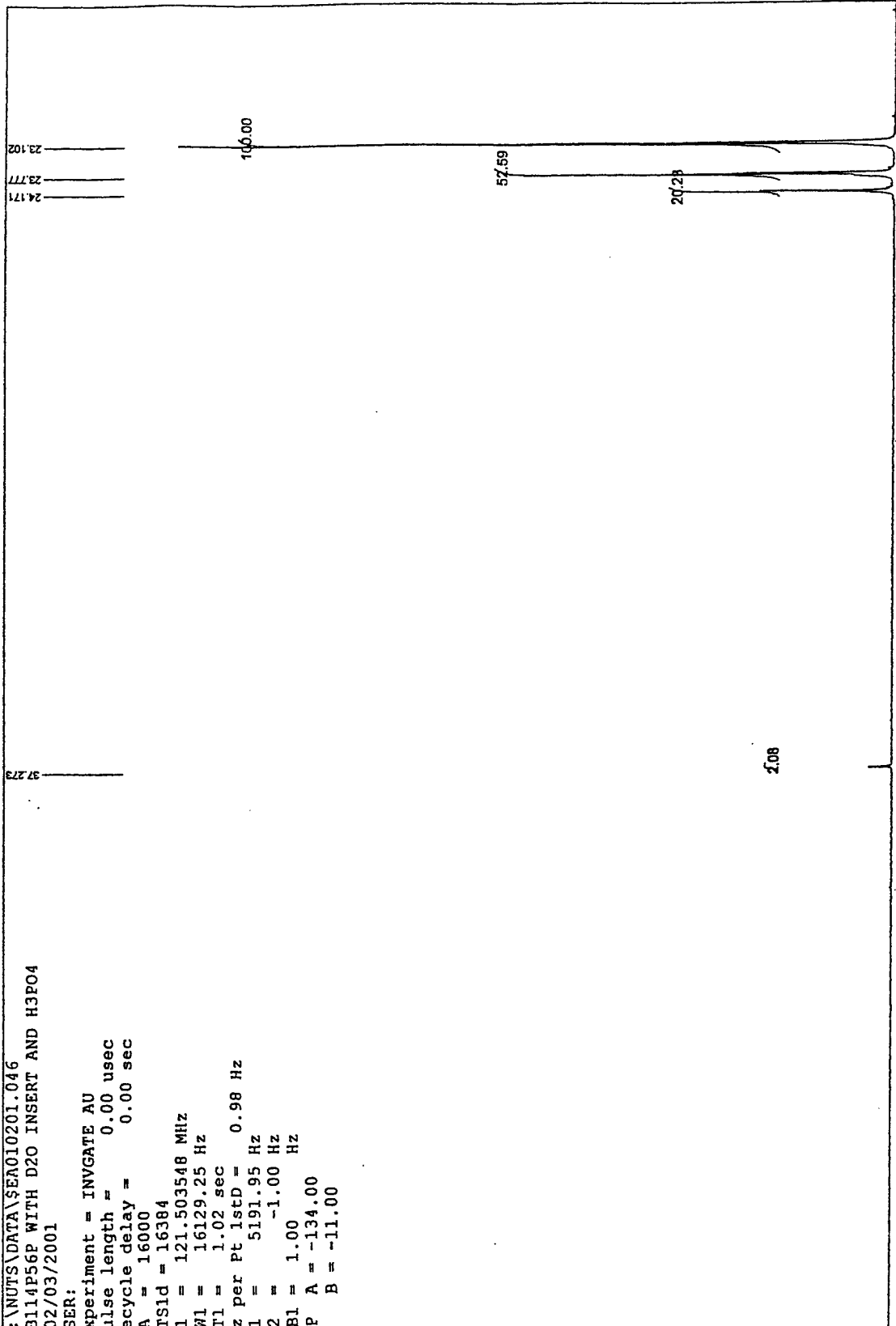
WinNuts - SEA010201.045

C:\NUTS\DATA\SEA010201.046  
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  
PTSId = 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  
TP A = -134.00  
B = -11.00

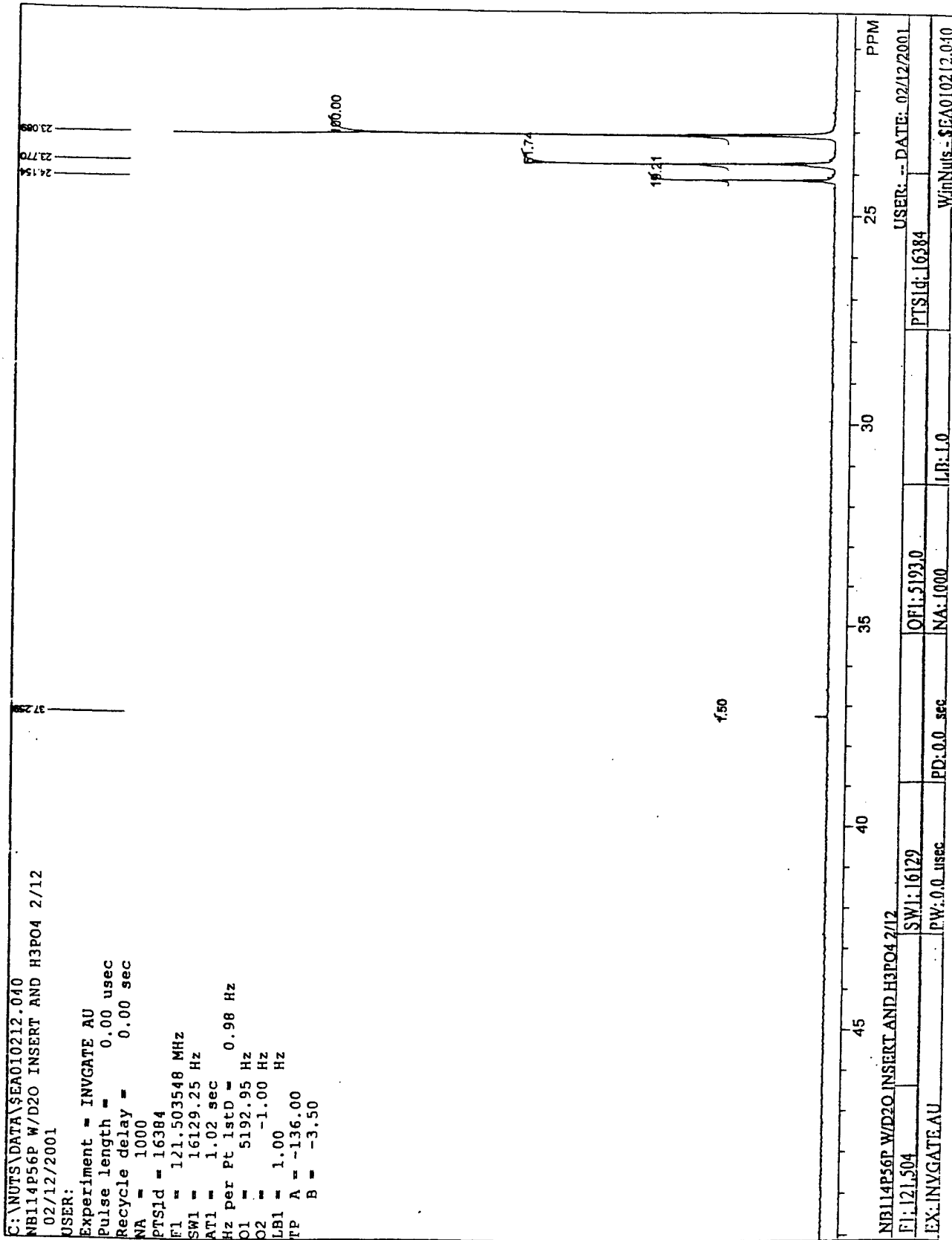


|                                     |             |             |              |                        |                  |
|-------------------------------------|-------------|-------------|--------------|------------------------|------------------|
| 45                                  | 40          | 35          | 30           | 25                     | PPM              |
| NB114P56P WITH D2O INSERT AND H3PO4 |             |             |              |                        |                  |
| F1: 121.504                         | SW1: 16129  | OF1: 5192.0 | PTSId: 16384 | USER: --               | DATE: 02/03/2001 |
| EX: INVGATE AU                      | PD: 0.0 sec | NA: 16000   | LB: 1.0      | W/nJus - SEA010201.046 |                  |

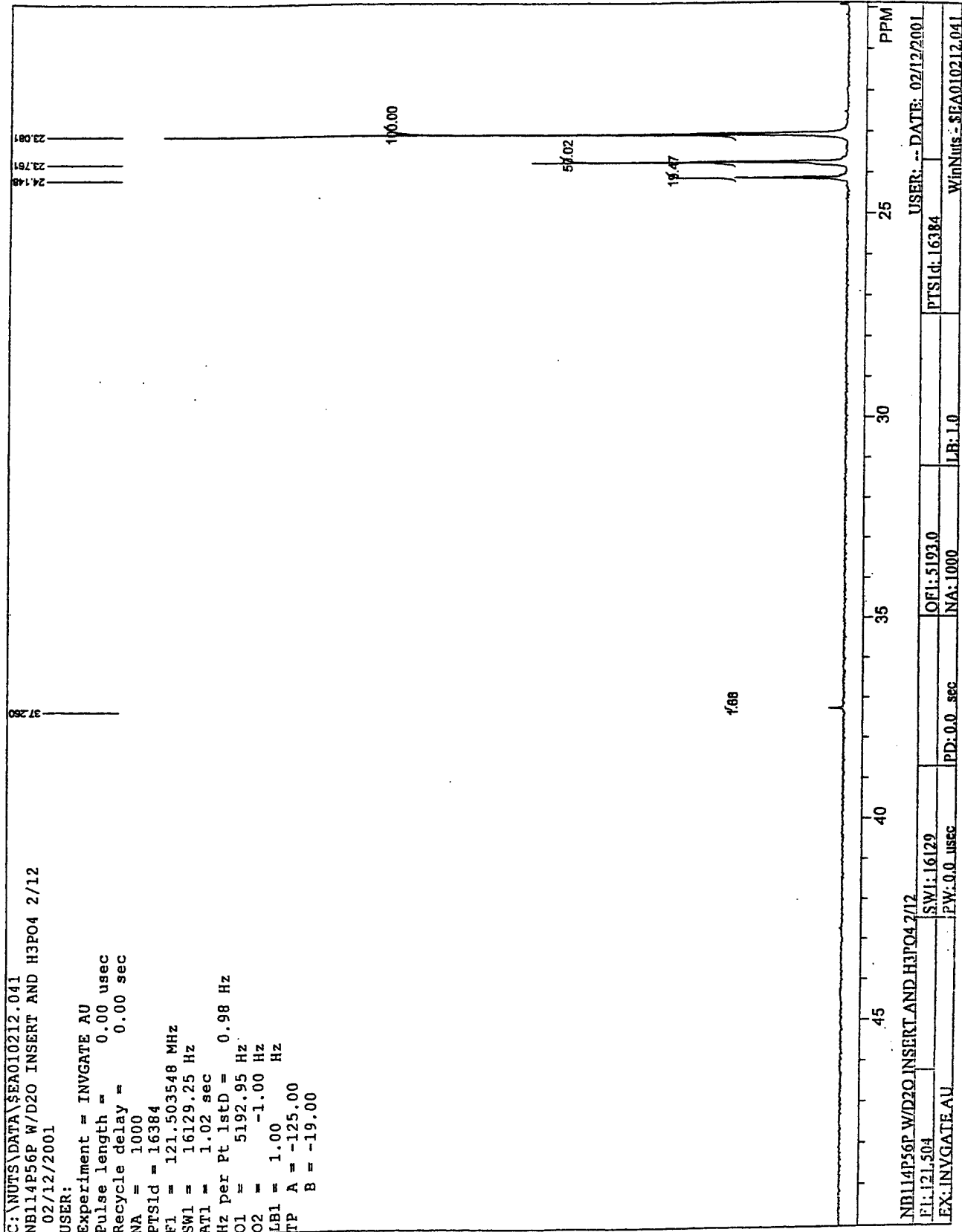
C:\NUTS\DATA\SEA010212.040  
 NB114P56P W/D20 INSERT AND H3PO4 2/12  
 02/12/2001

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  
 TP A = -136.00  
 B = -3.50



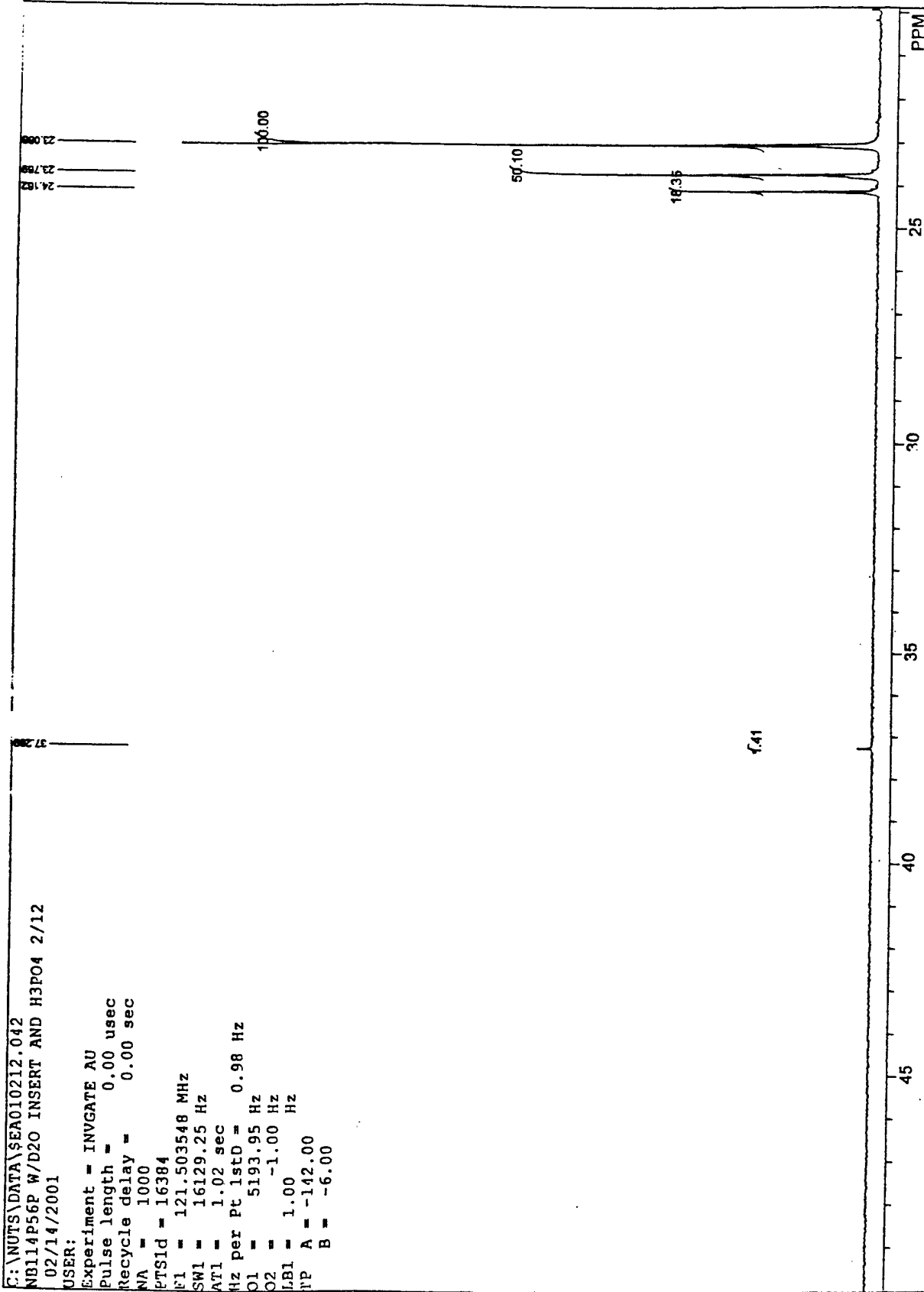
NB114P56P W/D20 INSERT AND H3PO4 2/12  
 F1: 121.504  
 EX: INVGATE AU  
 USER: -- DATE: 02/12/2001  
 PTS1d: 16384  
 OF1: 5192.0  
 NA: 1000  
 LR: L0  
 PD: 0.0 sec  
 PW: 0.0 usec  
 WinNuts = SEA010212.040



C:\NUTS\DATA\SEA010212.042  
 NB114P56P W/D20 INSERT AND H3PO4 2/12  
 02/14/2001

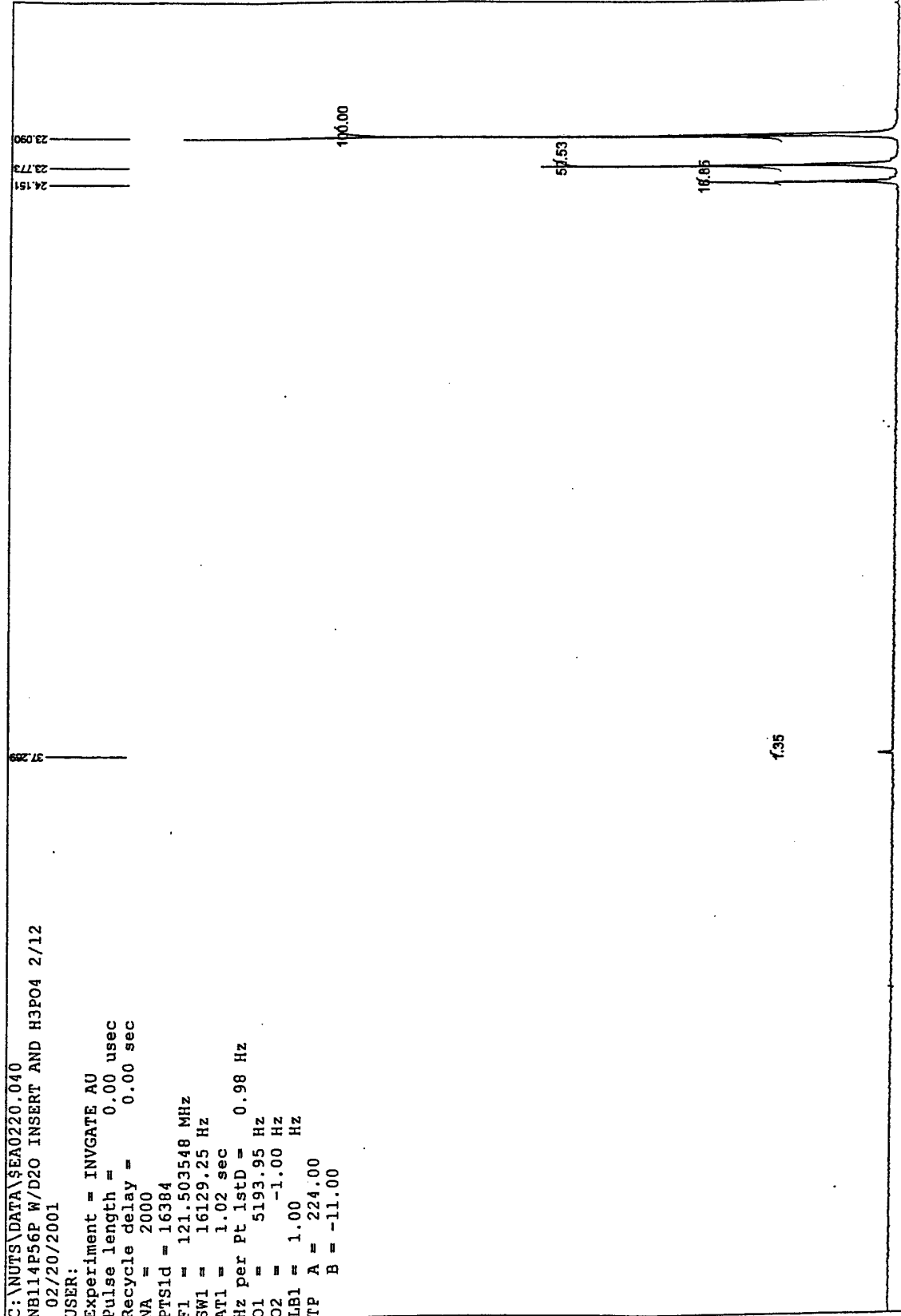
USER:

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  
 WP A = -142.00  
 B = -6.00



|                                       |              |                           |                         |
|---------------------------------------|--------------|---------------------------|-------------------------|
| NB114P56P W/D20 INSERT AND H3PO4 2/12 |              | USER: -- DATE: 02/14/2001 |                         |
| FI: 121.504                           | SW1: 16129   | OFl: 5194.0               | PTS1d: 16384            |
| EX: INVGATE AU                        | PW: 0.0 usec | NA: 1000                  | WinNuts - SEA010212.042 |
|                                       | PD: 0.0 sec  | LR: 1.0                   |                         |





C:\NUTS\DATA\SEA0220.040  
 NB114F56P W/D20 INSERT AND H3F04 2/12  
 02/20/2001  
 USER:  
 Experiment = INVGATE AU  
 Pulse length = 0.00 usec  
 Recycle delay = 0.00 sec  
 NA = 2000  
 PTSId = 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

|                                       |              |                           |              |
|---------------------------------------|--------------|---------------------------|--------------|
| NB114F56P W/D20 INSERT AND H3F04 2/12 |              | USER: -- DATE: 02/20/2001 |              |
| F1: 121.504                           | SW1: 16129   | OFF: 5194.0               | PTSId: 16384 |
| EX: INVGATE.AU                        | PW: 0.0 usec | NA: 2000                  | LB: 1.0      |
|                                       | PD: 0.0 sec  | WinUnits - \$EA0220.040   |              |

C:\NUTS\DATA\SEA010226.040  
 NB114P56P W/D20 INSERT AND H3PO4 2/12  
 02/26/2001

USER:

Experiment = INVGATE AU  
 Pulse length = 0.00 usec  
 Recycle delay = 0.00 sec  
 NA = 2000  
 PtsId = 16384  
 F1 = 121.503548 MHz  
 SW1 = 16129.25 Hz  
 AT1 = 1.02 sec  
 Hz per Pt lstd = 0.98 Hz  
 O1 = 5193.95 Hz  
 O2 = -1.00 Hz  
 LB1 = 1.00 Hz  
 TP A = 215.50  
 B = 0.00

37.278

24.151  
 23.774  
 23.082

170.00

49.89

18.28

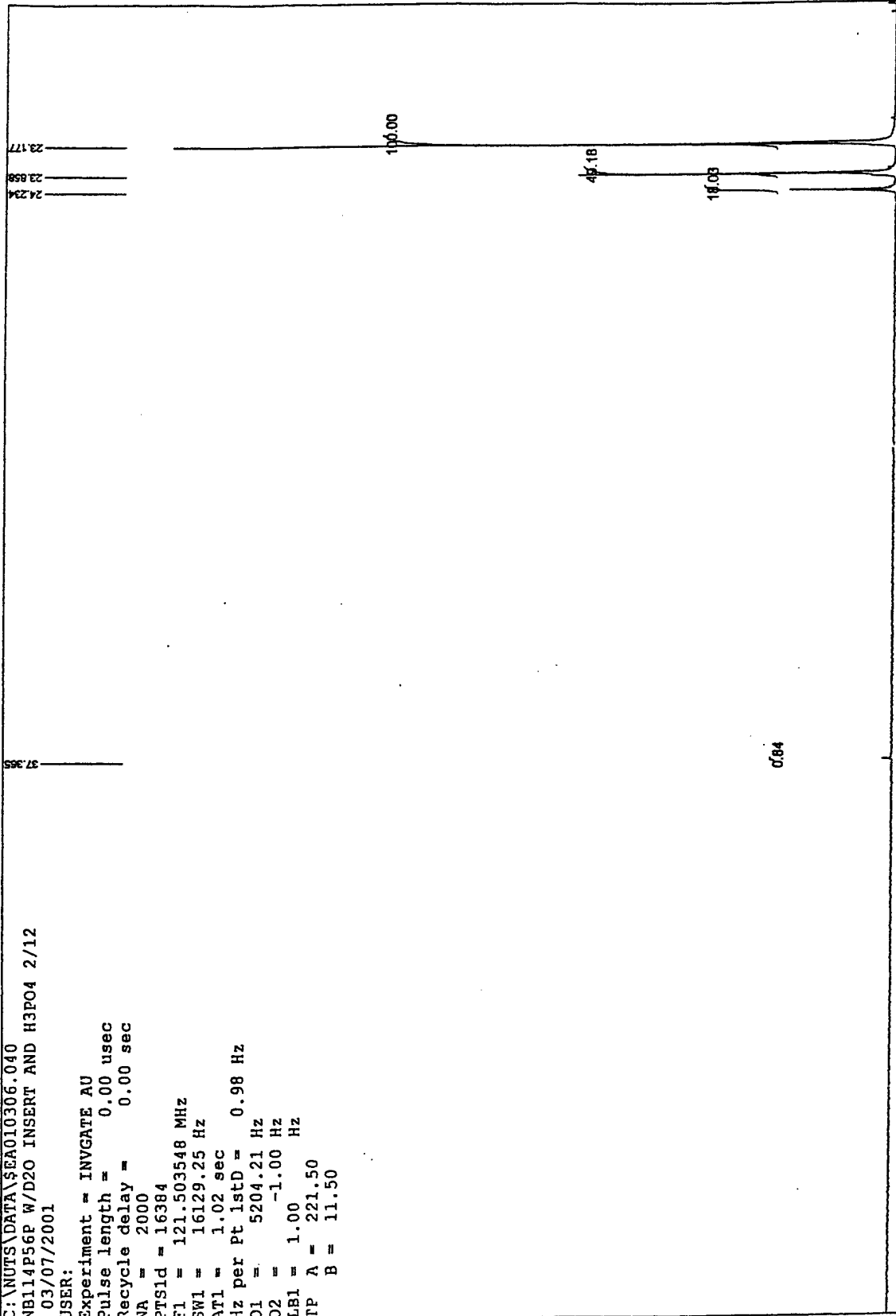
f.22

45 40 35 30 25 PPM

NB114P56P W/D20 INSERT AND H3PO4 2/12  
 F1: 121.504 SW1: 16129 OF1: 5194.0 USER: -- DATE: 02/26/2001  
 EX: INVGATE AU PW: 0.0 usec PD: 0.0 sec NA: 2000 PTSId: 16384 LB: 1.0  
 WinNuts - SEA010226.040

C:\NUTS\DATA\SEA010306.040  
 NB114P56P W/D20 INSERT AND H3PO4 2/12  
 03/07/2001

USER:  
 Experiment = INVGATE AU  
 Pulse length = 0.00 usec  
 Recycle delay = 0.00 sec  
 NA = 2000  
 PTSId = 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  
 LBI = 1.00 Hz  
 TP A = 221.50  
 B = 11.50

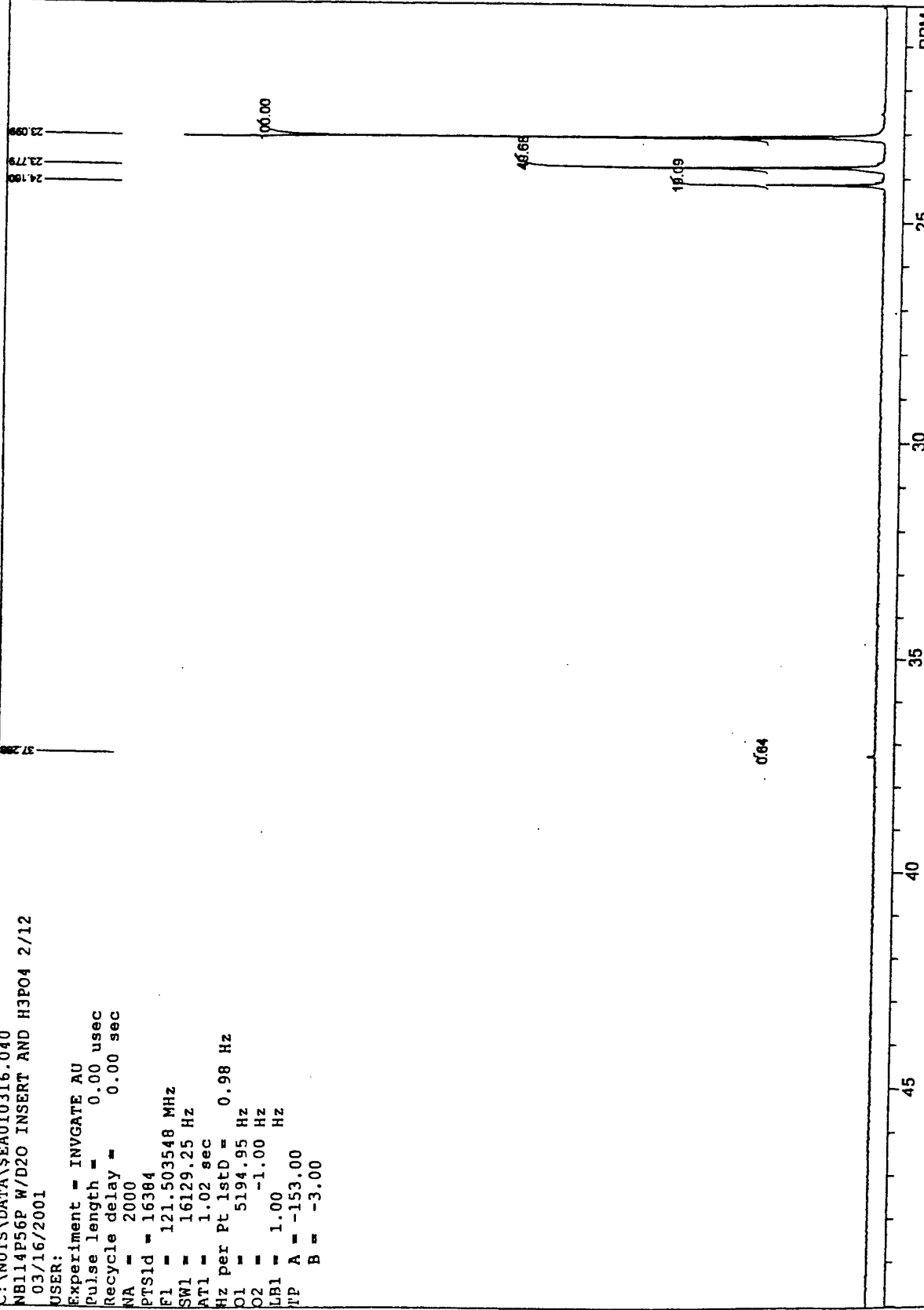


|                                       |    |             |    |                           |     |
|---------------------------------------|----|-------------|----|---------------------------|-----|
| 45                                    | 40 | 35          | 30 | 25                        | PPM |
| NB114P56P W/D20 INSERT AND H3PO4 2/12 |    |             |    |                           |     |
| F1: 121.504                           |    | ORI: 5204.2 |    | USER: -- DATE: 03/07/2001 |     |
| EX: INVGATE AU                        |    | NA: 2000    |    | PTSId: 16384              |     |
| P.W: 0.0 usec                         |    | PD: 0.0 sec |    | LB: 1.0                   |     |
| WinNuts - SEA010306.040               |    |             |    |                           |     |

C:\NUTS\DATA\SEA010316.040  
 NB114P56P W/D20 INSERT AND H3PO4 2/12  
 03/16/2001

USER:

Experiment = INVGATE AU  
 Pulse length = 0.00 usec  
 Recycle delay = 0.00 sec  
 NA = 2000  
 PTSld = 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  
 FP A = -153.00  
 B = -3.00

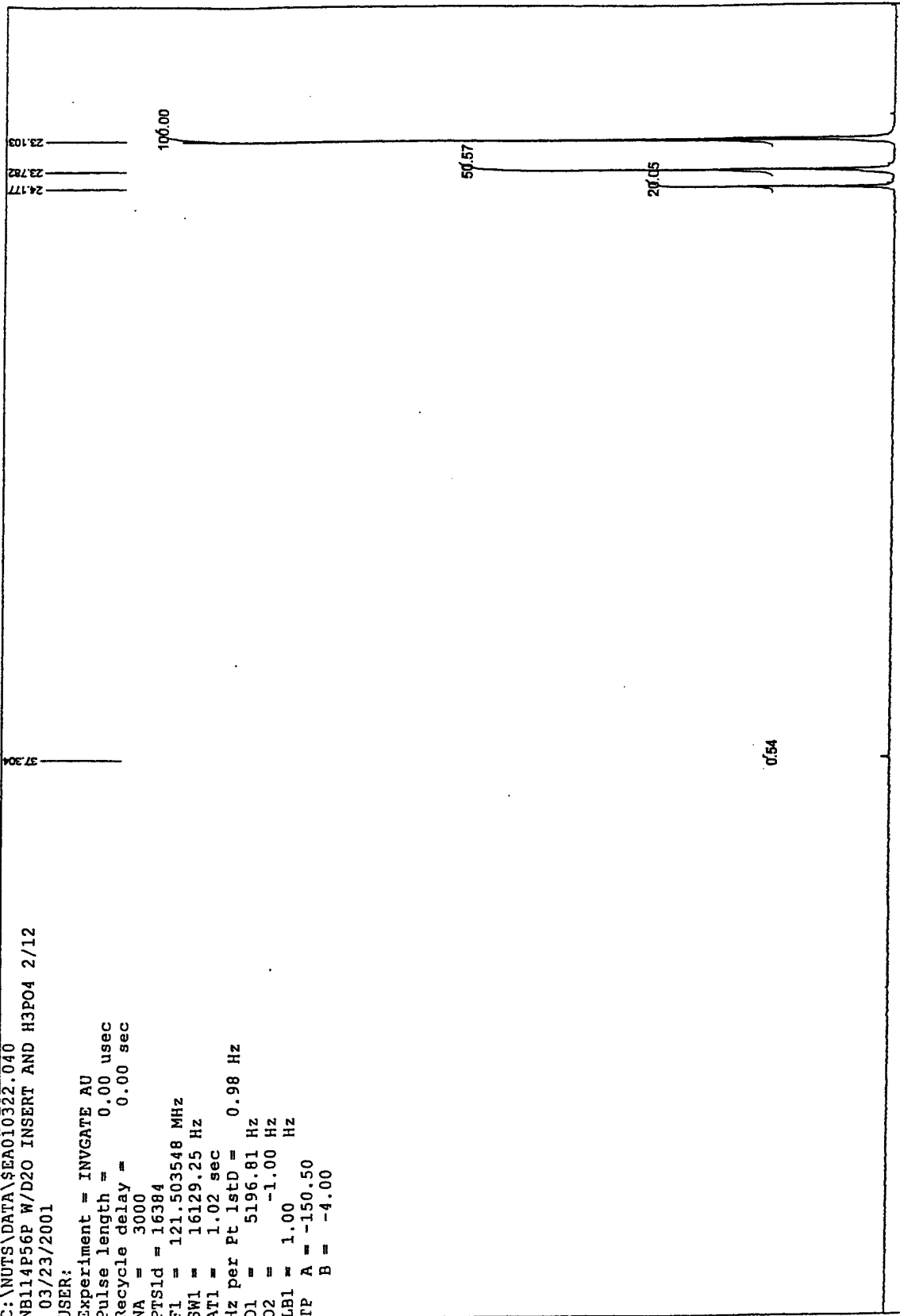


EX: INVGATE.AU  
 F1: 121.504  
 NB114P56P W/D20 INSERT AND H3PO4 2/12  
 SW1: 16129  
 OF1: 5195.0  
 PW: 0.0 usec  
 NA: 2000  
 PD: 0.0 sec  
 LB: 1.0  
 PTSld: 16384  
 USER: -- DATE: 03/16/2001  
 WinNuis: SEA010316.040

C:\NUTS\DATA\SEA010322.040  
 NB114P56P W/D20 INSERT AND H3PO4 2/12  
 03/23/2001

USER:

Experiment = INVGATE AU  
 Pulse length = 0.00 usec  
 Recycle delay = 0.00 sec  
 NA = 3000  
 PTSId = 16384  
 F1 = 121.503548 MHz  
 SW1 = 16129.25 Hz  
 AT1 = 1.02 sec  
 Hz per Pt lstd = 0.98 Hz  
 O1 = 5196.81 Hz  
 O2 = -1.00 Hz  
 LB1 = 1.00 Hz  
 TP A = -150.50  
 B = -4.00

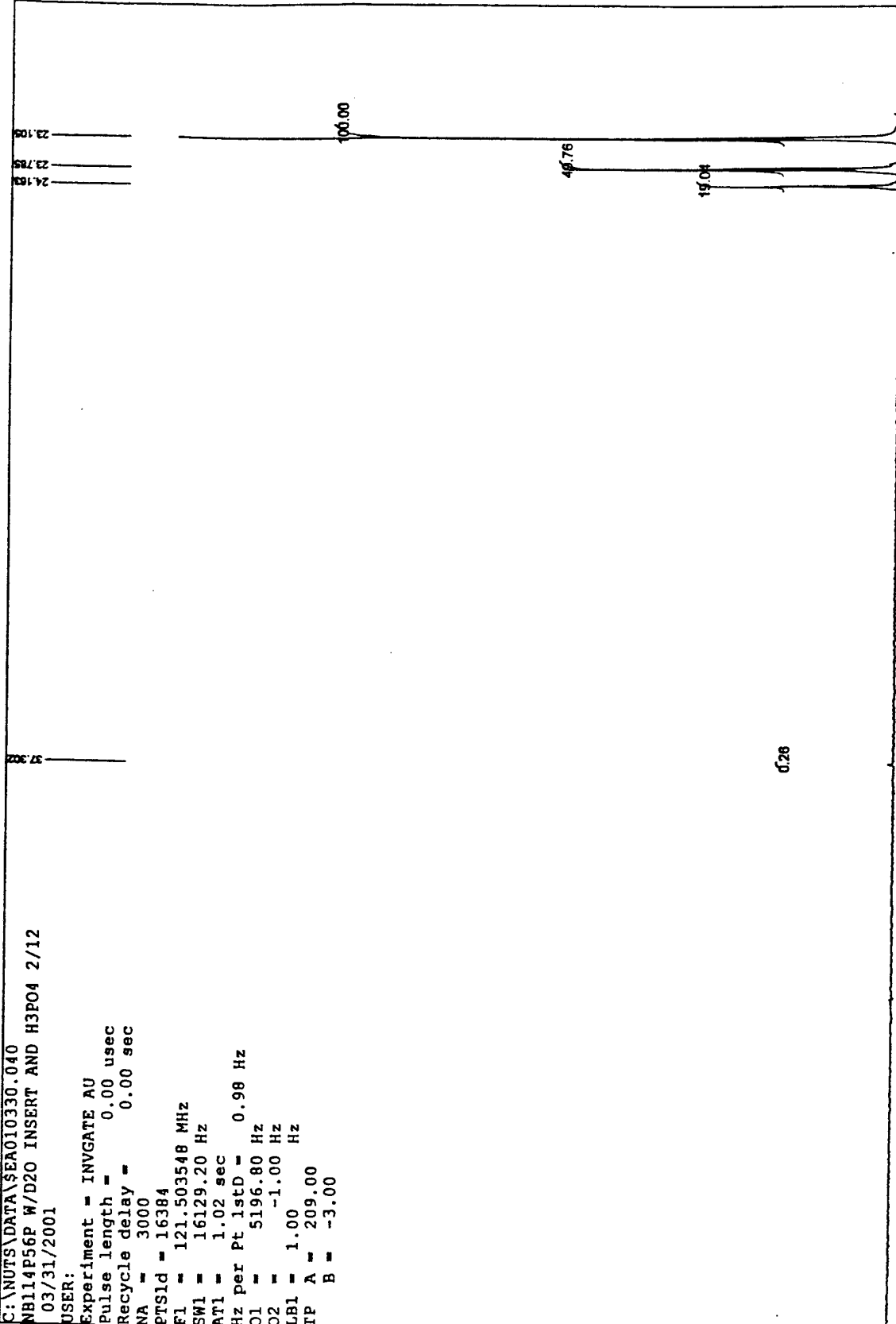


|                                       |              |                           |                         |
|---------------------------------------|--------------|---------------------------|-------------------------|
| NB114P56P W/D20 INSERT AND H3PO4 2/12 |              | USER: -- DATE: 03/23/2001 |                         |
| F1: 121.504                           | SW1: 16129   | QF1: 5196.8               | PTSId: 16384            |
| EX: INVGATEAU                         | PW: 0.0 usec | NA: 3000                  | LB: 1.0                 |
|                                       | PD: 0.0 sec  |                           | WinNuts - SEA010322.040 |

C:\NUTS\DATA\SEA010330.040  
 NB114P56P W/D20 INSERT AND H3PO4 2/12  
 03/31/2001

USER:

Experiment = INVGATE AU  
 Pulse length = 0.00 usec  
 Recycle delay = 0.00 sec  
 NA = 3000  
 PTSld = 16384  
 F1 = 121.503548 MHZ  
 SW1 = 16129.20 Hz  
 AT1 = 1.02 sec  
 Hz per Pt lstd = 0.98 Hz  
 O1 = 5196.80 Hz  
 O2 = -1.00 Hz  
 LB1 = 1.00 Hz  
 TP A = 209.00  
 B = -3.00



|                                       |              |                           |                         |
|---------------------------------------|--------------|---------------------------|-------------------------|
| NB114P56P W/D20 INSERT AND H3PO4 2/12 |              | USER: -- DATE: 03/31/2001 |                         |
| F1: 121.504                           | SW1: 16129   | OF1: 5196.8               | PTSld: 16384            |
| EX: INVGATE AU                        | PW: 0.0 usec | NA: 3000                  | LB: 1.0                 |
|                                       |              | PD: 0.0 sec               | WinNuts - SEA010330.040 |