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# Detection and Identification Techniques for Condom Residues in Sexual Assaults

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MASTER OF SCIENCE IN FORENSIC SCIENCE

By

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**DETECTION AND IDENTIFICATION TECHNIQUES FOR**

**CONDOM RESIDUES IN SEXUAL ASSAULTS**

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APPROVED FOR THE FORENSIC SCIENCE INSTITUTE

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## Abstract

This study investigated techniques used to detect and identify condom residues in sexual assaults. There were 10 condom brands/sub-brands analyzed, which were chosen based on the geographical locations of the manufacturers. Polarized light microscopy was implemented as an initial means of detecting condom residues by identifying common particulates added during production. It was found that starch was present in only 5 of the condom brands/sub-brands, and no other particulates were identified. These results led to the conclusion that this technique would not be effective as a general screen for the presence of condom residues. Gas chromatography/mass spectrometry (GC/MS), coupled with liquid-liquid extraction and later solid-phase microextraction (SPME) were explored with the intentions of building a database that could suggest a condom brand in the instance of an unknown source, i.e., from a criminal investigation. The foundation of this work was based on a protocol outlined in an unpublished work by Wolfgang Keil, Andrea Berzlanovich, and Robert Blackledge. Alkaline extractions were conducted on condom residues and in some instances, derivatization was performed. Analysis revealed that SPME, using a polyacrylate fiber, produced satisfactory results. This technique produced total ion chromatograms with distinct variations between condom brands and some of the sub-brands, while the mass spectra identified multiple components in the residues. Isotope-ratio mass spectrometry (IRMS) was also undertaken to determine if the carbon isotopic ratios of condom residues differed among brands. Three different ratios were observed, suggesting the possibility that manufacturers obtain their lubricants, polydimethylsiloxane, from different geographical sources.

# Introduction

## Research Problem

As DNA technology has advanced, sexual predators have become more savvy. The use of condoms by perpetrators in sexual assault cases has increased significantly over the past few decades (Blackledge R., 1996). Often, residues from condoms are left on victims, clothing, or bedding at crime scenes of this nature. Condom residues are classified as a type of trace evidence, thus in the same class with paint chips, fibers, and accelerants. Although common, this category of trace evidence is often overlooked because it is either undetected or believed to be of little use to investigators. At this time, local agencies do not have a simple method for detecting condom residues, and there is no current database in place in the forensic science community that can quickly classify or identify condom brands. In fact, there has been very little research conducted on products marketed in the United States.

## Literature Summary

The basic steps of condom production, excluding packaging, are formation, vulcanization, silicone washing treatment, powdering, and lubrication (Keil, 2007). The components added during the powdering and lubrication stages have been the primary target in past studies. The powders that are added largely consist of starch particles, roughly 2-32 μm in size. The majority of condoms are lubricated with a high molecular weight silicone-based substance, polydimethylsiloxane (PDMS), and there are a few lubricated with a water-soluble compound, polyethylene glycol (PEG). A small portion of condoms produced have a spermicide added during the lubrication step.

A variety of techniques have been implemented in the analysis of condom residue compositions. Some past experiments have combined several methods to identify multiple

components of the condoms examined, providing multi-level approaches to the assessment of these of products. Fourier transform infrared spectroscopy (FTIR) has been identified as the most common instrumentation used in most state laboratories for testing and comparison procedures of condom residues (Campbell & Gordon, 2007). Characterization of products commercially available in other countries has been undertaken, and several of the techniques that will be described have been shown to have advantages. Only recent works have focused on identifying chemical components found in condom residues produced from steps other than powdering and lubrication.

**Literature Deficiencies**

Though some precedent research has been conducted pertaining to the detection of condom residue components, there have been very few studies performed on products commercially available in the United States. Some instruments were not sensitive enough to detect residue samples in small quantities, and other methods that were studied are complex and not commonly accessible to most crime laboratories. These limitations make these approaches impractical for general use in forensic applications. Most of the studies that have been conducted to differentiate condom products have concentrated on the lubricant, due to its large contribution in condom residues. Currently, there is no indication that a practical method has been developed for differentiation between brands and sub-brands by analyzing the lubricant component alone. The biggest problem arises in the fact that the majority of condom products contain the same lubricant base, PDMS, which to date has not been conclusively distinguished across manufacturers. Due to the high molecular weight and low volatility of this silicone-based compound, complicated analytical methods or those that take an extended amount of time must be used, which make them impractical for routine casework in criminal laboratory settings. The

presence of a spermicide has played a role in differentiating condom manufacturers, but the majority of products do not contain a spermicide, so it cannot be used as method for individualization of a large number of products.

**Research Significance**

In instances where DNA cannot be found on the victim or at the crime scene, other evidence connecting the perpetrator to the offense is invaluable (Campbell & Gordon, 2007). Successful completion of this research can aid investigators in linking or excluding condom products found on or within the residence of a suspect in a sexual assault. The ability of forensic scientists to identify condom residues quickly is essential. Once these residues are identified, knowing their chemical composition and having access to a database to indicate or eliminate the source is crucial when trying to solve a sexual assault case promptly.

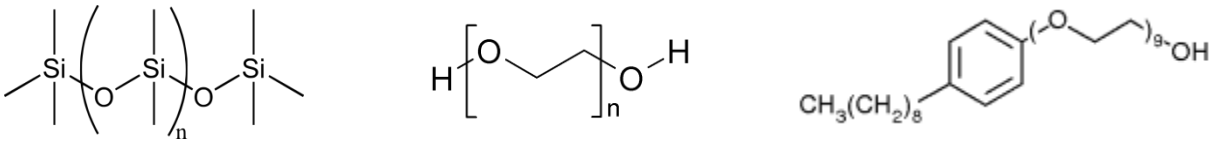
**Purpose of Study**

The initial element of this research was analyzing various brands and sub-brands of condoms under a polarized light microscope to determine which products contain starch particles or lycopodium spores. Depending on the commonality of these components, searching for the presence of these particulates could prove to be an ideal means of screening for condom residues. This data could also serve as a basic technique for differentiating condom manufacturers. GC/MS was utilized to uniquely characterize each condom. The total ion chromatogram and/or mass spectra for each product may then be catalogued to develop a searchable identification database, accessible to law enforcement agencies. The objective of this research was to develop a systematic method for detecting and identifying condom residue evidence in sexual assault cases.

## Literature Review

In determining the components found in condom residues, it is important to first understand the process in which condoms are fabricated and to know the chemical signatures that are left from each step (Keil, 2007). First, the rough condom is formed from latex collected from rubber trees. It then undergoes vulcanization. Vulcanization is the process of increasing the viscosity of the rubber into a more durable, less sticky material. The rough condom contains minute amounts of latex proteins, as well as dithiocarbamates and nitrosamines left from acceleration of the vulcanization step. The condoms are then washed with an aqueous silicone emulsion, containing lower molecular weight silicone oils within the slurry, which penetrate the rubber and act as a softener of the material. A fine powder coating is added to the condoms to keep the rolled-up latex from sticking, which allows the condom to be unrolled with ease. The particulates are composed primarily of cornstarch and polyethylene, with the concentration of starch being five times greater than polyethylene. Occasionally, the powder contains lycopodium spores, talc, and silica, added as a filler material (Blackledge & Vincenti, 1994) . Antioxidants and preservatives, added to retard the degradation of the latex, have also been detected in small quantities. Examples of these are Wingstay-L®, a butylated product of *p*-cresol and dicyclopentadiene, and Kathon CG, butylated hydroxytoluene (BHT), which kill bacteria (Keil, 2007). Most condoms have a lubricant added to the surface, which consists of either the water soluble compound, PEG, or the non-polar substance, PDMS, with the latter being the more common of the two. PEG and PDMS are often referred to as "wet" and "dry" lubricants, respectively (Blackledge R., 1996). The amount of lubricant added to the condom usually ranges from 150-300 mg (Keil, 2007). About 10% of lubricated condoms have the spermicide nonoxynol-9 added to them, which represents about 5-10% of the lubricant (Hollenbeck,

Siuzdak, & Blackledge, 1999; Maynard, Allwell, Roux, Dawson, & Royds, 2001). The lubricants and spermicide are illustrated in Figure 1. Last, some condom manufacturers add flavors, scents, and anesthetics, such as benzocaine or lidocaine, to the lubricant.




(a) (b) (c)

Figure 1. Structures of lubricants and spermicide (a) polydimethylsiloxane (PDMS) (b) polyethylene glycol (PEG) (c) nonoxynol-9.

Primarily, studies have focused on the lubricant coating of condoms. The instrumental approaches that have been employed include FTIR, GC/MS, pyrolysis gas chromatography/mass spectrometry (PyGC/MS), proton nuclear magnetic resonance ($^1$H-NMR) spectroscopy, desorption chemical ionization mass spectrometry (DCI/MS), and micellar electrokinetic capillary chromatography (MEKC) with ultraviolet absorbance detection (Blackledge & Vincenti, 1994; Burger, Dawson, Roux, Maynard, Doble, & Kirkbride, 2005; Campbell & Gordon, 2007; Conti, Dezzi, & Bianco, 1995; Lee, Brinch, Kannangara, Dawson, & Wilson, 2001; Maynard et al., 2001). FTIR, liquid chromatography/mass spectrometry (LC/MS), electrospray ionization/mass spectrometry (ESI/MS), and matrix assisted laser desorption ionization/mass spectrometry (MALDI/MS) have been techniques used to identify the spermicide nonoxynol-9 (Blackledge & Vincenti, 1994; Hollenbeck, et al., 1999; Maynard et al., 2001). There have been a couple of authors that examined the particulates from the powder coating found in trace residues. The techniques that were implemented were light microcopy, fluorescent light microscopy, polarized light microscopy (PLM), and Raman spectroscopy with Raman chemical imaging (Blackledge & Vincenti, 1994; Coyle & Anwar, 2009; Keil,

Berzlanovich, & Blackledge, n.d.; Maynard et al., 2001; Wolfe & Exline, 2003). Prior research conducted on condoms has been performed using techniques with single instruments, and others have used multiple methods on the same product to further differentiate the sample.

Robert D. Blackledge, a retired forensic chemist at the U.S. Naval Criminal Investigative Service (NCIS) Regional Forensic Laboratory in San Diego, California, was the first to examine condom lubricants and has produced the largest number of publications on the subject. Blackledge and Vincenti (1994) attempted to distinguish several different condom brands. They used polarized light microscopy to identify particulates added to the condom surfaces and found cornstarch, lycopodium, silica, and talc, which produced some discrimination among condom brands. The authors also extracted and detected PDMS and nonoxynol-9 by means of FTIR analysis. DCI/MS was implemented to try and differentiate the PDMS lubricant used by different manufacturers. The study was successful at detecting as little as 20 ng of PDMS in two actual case samples and had a fair capability of distinguishing the molecular weight distributions of PDMS compounds of varying viscosities.

In another publication, Blackledge (1995) used Fourier self-deconvolution (FSD) of FTIR spectra of condom lubricants to differentiate PDMS lubricants of varying viscosities. FSD is a method that can be used to determine the dimethyl to trimethyl (2ME/3ME) ratios by measuring the areas under their respective peaks. PDMS viscosity standards of 50, 100, 200, 350, and 500 CentiStokes (cSt) were obtained and their 2ME/3ME ratios measured. PDMS lubricant samples were taken from 10 different brands and examined using the FSD method. Their peak ratios were compared to known standards as a means of determining their approximate viscosities. The results revealed that this method could be used for determining the approximate chain lengths of the PDMS oligomers and thus differentiate condom brands. The

author indicated that further research should be conducted to determine if the viscosities of PDMS change with lot numbers or with elapsed time in the vaginal cavity.

Hollenbeck et al. (1999) chose then newer mass spectrometry techniques to identify the spermicide nonoxynol-9 in small traces and therefore provide evidence that a condom was used in a crime. The techniques of liquid chromatography/electrospray ionization mass spectrometry (LC/ESI-MS), nanoelectrospray ionization mass spectrometry (nanoESI-MS), and matrix assisted laser desorption ionization Fourier transform mass spectrometry (MALDI-FTMS) were all implemented. The methods were successful at detecting residues in low quantities from internal vaginal swabs taken post-coitus and an actual evidence sample. It was noted by the authors that these instruments are not commonly accessible to crime laboratories.

Conti et al. (1995) chose $^{1}$H-NMR spectroscopy, due to the instrument's sensitivity, to examine 47 condoms lubricated with PDMS on the market in Italy. The goal was to identify what the detection limits were for detecting PDMS. Experiments were performed on a variety of condoms, using the following techniques:

(a) Condom rubbed dry on a strip of skin approximately 5 cm long and 1 cm wide.

(b) Simple contact of 2 seconds on skin, having a surface area of 2 cm in diameter; with subsequent drying.

(c) Contact of 2 seconds on skin of arm, followed by washing with running water and drying.

The results confirmed the range for identifying PDMS as falling between 0.0428 and 0.0440 ppm in the proton NMR spectrum. This study also referred to two case histories in which $^{1}$H-NMR had been employed to analyze vaginal swabs. In the first instance, a sample was taken immediately following a sexual assault and showed a peak at 0.0424 ppm. In the other case, a swab was taken a number of hours after the attack, following two washings with water and a

third with a feminine hygiene detergent product. The $^{1}$H-NMR spectra revealed a peak at 0.0420 ppm, as well as other peaks representing organic substances. The authors indicated the need for further studies investigating the duration of PDMS on victims and clothing.

Lee et al. (2001) used both solution-state and solid-state NMR spectroscopic techniques to examine a representative set of 38 condom samples from 12 manufacturers marketed in Australia. For solid-state analysis, parts of the condom were clipped off and tested. It was determined that the solid-state method was not practical due to the commonality of the latex condom. For the solution-state, hexane was used to wash the condom samples. Of the 38 condoms tested, 15 could be differentiated by solution-state $^{1}$H-NMR. The remaining 23 were examined for variances in texture, color, and flavors. The results were that 33 of the 38 condoms examined in this study could be individualized by a combination of $^{1}$H-NMR and physical examination of the condom. A classification table and flow chart were created to identify the different chemical shifts seen with each product and the process for individualizing an unknown condom. It should be noted that this technique would only be useful if the actual condom that the perpetrator used in the crime could be located.

Maynard et al. (2001) performed several techniques commonly found in crime laboratories to try to differentiate a variety of condom lubricants, as well as personal and improvised lubricants, using fluorescence examination, FTIR, GC/MS, LC/MS, and PyGC/MS. The products consisted of 58 condoms, 22 personal lubricants, and 10 improvised lubricants, all marketed in Australia. Fluorescent light microscopy was used to examine smears from the product swabs to note morphology and the presence of particulates. Samples were then extracted using hexane and methanol and underwent FTIR analysis, identifying the presence of PDMS, PEG, and nonoxynol-9 in each product. GC/MS and PyGC/MS were used as confirmation tests

for PEG and PDMS, respectively. LC/MS was used to investigate any differences in the nonoxynol-9 structures, which produced no informative results. Out of 50 products examined, 11 were uniquely identified, and those remaining were classified into 9 groups. These results produced a recommended protocol to be used in Australia on unknown biological swabs from crime scenes. The author indicated that other analytical techniques may be more useful for discrimination purposes.

Campbell and Gordon (2007) attempted to establish a more sensitive and discriminating technique than FTIR for detecting lubricant evidence. PDMS and PEG, present in 38 condoms from all the major distributors and manufacturers available in New Zealand, were targeted. PDMS was analyzed using PyGC/MS, and PEG was detected using GC/MS directly from solution. The authors' hope was that they could not only devise a more sensitive method for detecting condom lubricants but that they could further differentiate the PDMS in condom lubricants. The thought was that by using high temperatures to break down the PDMS oligomers to cyclic dimethylsiloxane products, their respective pyrograms could be compared to look for variances in their peak ratios. Like past studies, the authors used hexane for the extraction of PDMS and methanol to extract PEG. The use of PyGC/MS and GC/MS for the detection of PDMS and PEG, respectively, proved to be significantly more sensitive methods than FTIR. PDMS was detected as low as 1 µg in standard solution and from clean swabs (not simulated case samples) using the PyGC/MS method. PEG was detected as low as 0.5 µg from standard solution and 50 µg from clean swabs using the GC/MS method. However, further discrimination between condom brands and sub-brands was not successful, because all produced similar pyrograms. These findings corroborated a prior publication by Kleinert and Weshler

(1980) from Bell Laboratories, which found that PDMS products of known varying viscosities could not be distinguished via comparison of their pyrograms.

Burger et al. (2005) analyzed 68 different condoms and personal lubricants marketed in Australia using MEKC with ultraviolet absorbance detection. The electropherograms were processed by principal component analysis (PCA) and classified with linear discriminate analysis. Of the 68 condoms analyzed, only 2 showed no detectable peaks in their electropherograms. Out of the 263 samples taken, 233 were able to be classified into an appropriate group using this method. Rough lubricant persistence tests were performed by swabbing an arm of a human subject or a piece of cloth rubbed with a freshly unrolled condom or a personal lubricant. The swabs were capable of being identified immediately after being rubbed on the surfaces but could not be successfully identified 30 minutes after contact. Although the technique described provides a quick and efficient method for classifying lubricants, the authors stated that it lacks the sensitivity needed to analyze trace amounts common in sexual assaults.

Raman spectroscopy and Raman chemical imaging were implemented by Wolfe and Exline (2003) to examine the components found on condom surfaces of several condom brands. They used Raman chemical imaging (RCI) to combine microscopy, digital imaging, and Raman spectroscopy. This provided both qualitative and quantitative information about the condom residue components. Pure dispersive spectra were obtained for lycopodium, PEG, PDMS, and nonoxynol-9. The lycopodium showed considerable fluorescence and unique surface morphology. PDMS and nonoxynol-9 appeared transparent, though the nonoxynol-9 contained bubbles resembling dark spheres in the liquid. Polarized light microscopy was also utilized to identify starch particles in some of the samples. Lubricant swabs from the various condom

brands, after being extracted and examined, were found to be accurately characterized by Raman spectroscopy. The structural quality and uniformity of the images were successfully used to differentiate brands of condoms. The authors suggested environmental effects and detection limits should be a further plan of study.

The impact of Raman spectroscopy on samples to be subjected to subsequent DNA analysis was explored by Coyle and Anwar (2009). Swabs taken from 47 condoms manufactured in the United Kingdom and 6 imported condoms were analyzed using Raman spectroscopy. This method revealed that 43 of the 47 condoms on the UK market (90%) were lubricated with PDMS. Of the 53 total samples swabbed, 11 exhibited near-infrared fluorescence. In this study of DNA analysis, 24 swabs were taken from known individuals, prepared with saliva, buccal scrapings, touch, and semen, and then analyzed using Raman spectroscopy. After extraction, quantification, and amplification of the swabs, the DNA profile of each sample was obtained. The results revealed that the impact of Raman spectroscopy on samples was not detrimental to later DNA analysis.

In a recent unpublished study, Keil et al. took a unique approach to individuate condom brands. They used light microscopy and GC/MS to analyze 54 condom brands available in Germany. Swabs of the unused condoms were smeared on a glass microscope slide, colored with hematoxylin-eosin staining, and examined for the presence of cornstarch, polyethylene, and lycopodium. The swabs then underwent an alkaline extraction, followed by derivatization, and were then subjected to GC/MS analysis. This was achieved following the steps outlined below:

1. The swab was washed with 5 mL of tris(hydroxymethyl)aminomethane (Tris) buffer of pH 7.5.

2. A 1 mL aliquot of the solution was combined with 1 mL ammonium buffer of pH 8.9 and 5 mL ether/ethyl acetate (1:1).
3. The organic layer was separated.
4. The solvent was evaporated.
5. Derivatization was performed with N,O-bis(trimethylsilyl)trifluoroacetamide with 1% trimethylchlorosilane (BSTFA + 1% TMCS).
6. A 1 µL injection was made into the GC/MS.

The goal was to detect unique components from the silicone washing treatment step that could individualize condom products. Although the chromatograms of each sample barely differed, the mass spectra of 5 to 11 of the peaks for each of the 54 condoms were filed into a database. Simulated case samples were then taken from 6 volunteer couples. After using a specific condom brand during intercourse, vaginal swabs were taken immediately after intercourse and at 1, 2, 3, 4, and 5 days post-coitus. The swabs were smeared on a glass microscope slide and examined via light microscopy in the same manner as the swabs taken from the unused condoms. The results revealed that starch particles, identified in all of the smears of the condom residues, and lycopodium spores, found in 4 of the brands, could be detected up to 4 days post-coitus. The presence of polyethylene particles was not found on any of the simulated sample swabs. The authors attributed this latter finding to the smaller concentrations of the substance in powder coatings, its small size, and lack of color or ability to be stained. The simulated sample swabs were then analyzed using GC/MS after performing the alkaline extraction process that was followed for the unused condoms. The samples were successfully matched back to the correct condom brand with 95% accuracy by using the database created from the unused condoms. Spectra were only obtainable from simulated sample swabs up to 1

day post-coitus. Although this work was unpublished, it was presented at an American Academy of Forensic Sciences meeting in 2003 and was later highlighted in a chapter written by Keil in a book edited by Blackledge (2007). Based on email communication, which is included in *Appendix A*, and personal conversations with Robert Blackledge, the conclusion is that to date, the protocol just described yields the best chance of unique differentiation among condom brands and the development of a usable database (R. Blackledge, personal communication, April 8, 2010).

A fairly new and simplified sampling technique coupled with GC/MS is becoming popular. The technique SPME, as a general approach to analysis of organics, was developed by Dr. Janusz Pawliszyn in 1992 at the University of Waterloo in Ontario, Canada. SPME utilizes a thin fiber made of fused silica that is protected by either a manual or automated stainless steel holder. The fiber is coated with an adsorbent polymer that extracts organic compounds in aqueous liquids or in the headspace of samples by chemical interactions or partitioning. The analyte may then be desorbed off the fiber in the injection port of a gas chromatograph. Various fiber coatings and thicknesses are available, thus facilitating tuning to the target analyte. On-fiber derivatization has been performed by exposing the SPME fiber to the vapors of a silylating reagent after extraction. One study used this process to analyze resveratrol in red wine. A Supelco® 85 µL polyacrylate fiber was utilized and is suitable for the extraction of polar semivolatiles. Extraction of the resveratrol from the wine was performed for 15 minutes while stirring at 400 rpm. The derivatization took place by inserting the fiber into the headspace of a 4 mL vial containing 5 µL of Sylon-BFT (BSTFA + 1% TMCS) reagent. Prior to derivatization, the silylating reagent was allowed to stand in the vial covered for 60 to 90 minutes to insure equilibrium (Stenerson, 2009). BSTFA + 1% TMCS acts by replacing active hydrogens with

trimethylsilyl groups. This in turn reduces the polarity and enhances the volatility of high molecular weight compounds or increases the molecular weight of very volatile compounds. This results in mass spectra that are more complex, making it easier to individuate specific compounds (Penton, 2005).

Another technique that has yet to be explored for the differentiation of condom residues is IRMS. This method is used to measure the mixture of stable isotopes found naturally in our environment and is usually utilized in the field of geology but has been implemented in environmental forensic casework. Recent studies have used IRMS to study the isotopic ratios in explosive residues. The results revealed that varying sources of triacetone triperoxide (TATP) and pentaerythritol tetranitrate (PETN) could be differentiated by measuring their carbon, oxygen, hydrogen, and their associated carbon and nitrogen isotopic ratios, respectively (Benson et al., 2009). In the field of environmental forensics, the measure of carbon isotopic ratios is common. This is due to the fact that various types of plants and the environment in which they exist determine the pathways they will use to photosynthesize. This will in turn produce organic matter of varying carbon isotopic ratios, depending on geographical location. The long-term decomposition of organic matter results in the formation of crude oils and other fossil fuels that have varying carbon isotopic signatures throughout the world. Carbon is made up of two stable isotopes $^{12}C$ and $^{13}C$, with the natural abundance of $^{12}C/^{13}C$ ratio being 99:1. When a stable carbon isotopic ratio is measured, it is then compared to a standard material (Pee Dee belemnite, or PDB). This is calculated by the following equation:

$$\delta^{13}C = (R_{sample}/R_{standard} - 1) \times 1000, \text{ where} \quad (1)$$

$$R = {}^{13}C/{}^{12}C \quad (2)$$

A measured sample will almost always be more depleted of the heavier $^{13}C$ isotope than the standard, and will therefore result in a negative number for $\delta^{13}C$, which is expressed in per mil (‰) (Philp & Jarde, 2007). Since PDMS, the common lubricant in condoms, is a synthetic product with petroleum-based components, it may be the case that condom manufacturers from varying geographical locations will use PDMS from different sources and will have variations in their carbon isotopic ratios. If this is true, then it might be possible to differentiate condom brands using IRMS.

## Materials and Methods

### Condom Selection

A representative set of condom brands and sub-brands was chosen from the list of registered medical devices in 2010 with the U.S. Food and Drug Administration (FDA). Many of the companies that are listed on the FDA's website do not actually manufacture condoms but only repackage, relabel, export, and develop formulas for products. There were five different brands selected by their manufacturing location, as well as sub-brands for some of the brands. This was done to determine if the geographical location of the manufacturer plays a factor in materials used in the production of condoms. This included two different brands made in the same country and a single brand made in two different countries. Table 1 lists the condom brands and sub-brands that were utilized in this study. A current list of FDA condom companies can be found in *Appendix F*.

Table 1

*Selected Condom Brands and Manufacturing Locations*

| Condom Brand | Manufacturer | Geographical Location |
|---|---|---|
| Durex Play Sensations Her Sensation | SSL Manufacturing, Ltd | Thailand |
| Durex Play Sensations Natural Feeling | SSL Manufacturing, Ltd | Thailand |
| Durex Play Sensations Tingling Pleasure | SSL Manufacturing, Ltd | Thailand |
| Durex Play Sensations Warming Pleasure | SSL Manufacturing, Ltd | Thailand |
| Kimono Select | Sagami Rubber Industries Co, Ltd | Japan |
| Lifestyles Contempo Bareback | Suretex, Ltd | Thailand |
| Lifestyles Contempo Luscious Flavors – Banana | Suretex Prophylactics (I), Ltd | India |
| Lifestyles Contempo Luscious Flavors – Strawberry | Suretex Prophylactics (I), Ltd | India |
| Lifestyles Contempo Luscious Flavors – Vanilla | Suretex Prophylactics (I), Ltd | India |
| Trojan Thintensity | Church & Dwight Co, Inc | United States |

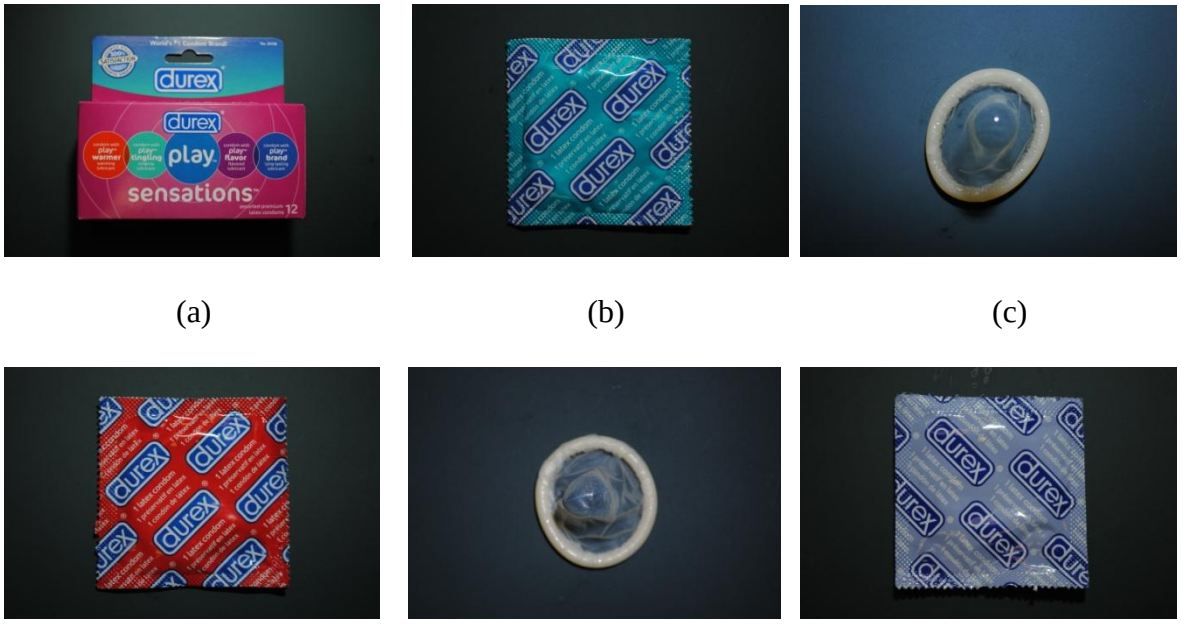
Each condom was photographed in its box, wrapper, and then by itself. This is illustrated in Figure 2 through Figure 6.


(a) (b) (c)

Figure 2. Trojan Thintensity (a) box (b) wrapper (c) condom.


(a) (b) (c)

Figure 3. Kimono Select (a) box (b) wrapper (c) condom.




(a) (b) (c)

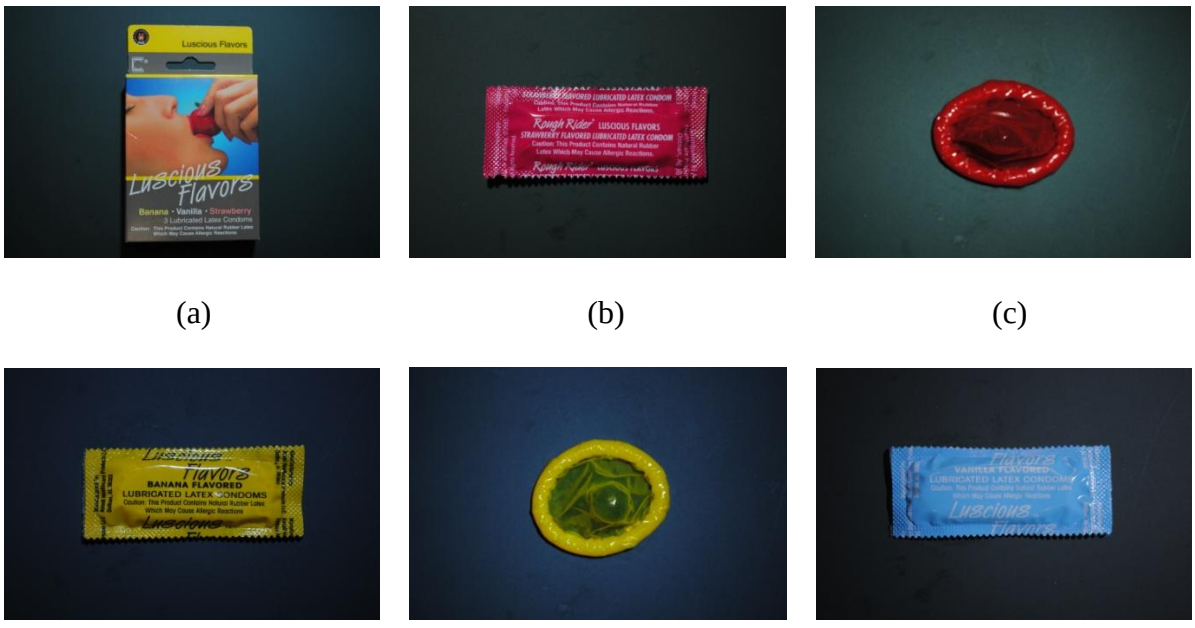
(d) (e) (f)


(g) (h) (i)

Figure 4. Durex Play Sensations (a) box (b) Tingling Pleasure wrapper (c) Tingling Pleasure condom (d) Warming Pleasure wrapper (e) Warming Pleasure condom (f) Her Sensation wrapper (g) Her Sensation condom (h) Natural Feeling wrapper (i) Natural Feeling condom.

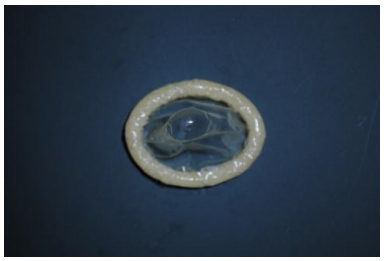

(a) (b) (c)

Figure 5. Lifestyles Contempo Bareback (a) box (b) wrapper (c) condom.




(a) (b) (c)

(d) (e) (f)



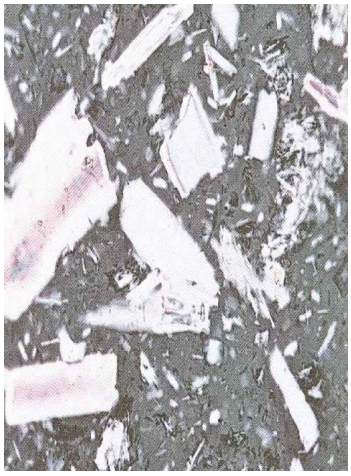
(g)

*Figure 6*. Lifestyles Contempo Luscious Flavors (a) box (b) Strawberry wrapper (c) Strawberry condom (d) Banana wrapper (e) Banana condom (f) Vanilla wrapper (g) Vanilla condom.

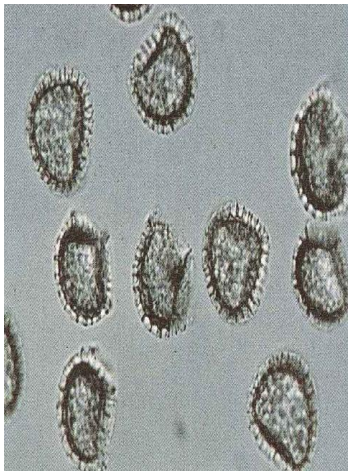
**Polarized Light Microscopy**

Each of the 10 condom brands/sub-brands was unrolled, and wooden cotton-tipped swabs were used to wipe the length of the outer surface of the condoms. The residues were then smeared onto separate microscope slides and cover slips were placed on top. The slides were examined under a PLM located in the Trace Evidence Unit of the Oklahoma State Bureau of Investigation (OSBI) laboratory. The samples were searched for the presence of cornstarch particles and lycopodium spores under plane-polarized light and under crossed polars. For the Kimono brand, a swab was also taken of the inside and compared to a swab taken from the outside surface. It was found that the smears had similar appearances to one another under both plane-polarized light and crossed polars. For those condoms that displayed Maltese cross interference patterns under crossed polars, the presence of starch was confirmed by staining with Lugol's solution ($I_2KI$). Iodine interacts with the coil structure of the polysaccharide, which results in blue-black staining of the starch. A few drops of the solution were allowed to flow under the coverslip from one end to the other, staining any starch particles that were present. It was found that when the Sigma Standard Fluka Lugol's solution, lot #BCBB3727, was diluted by 1:10 with distilled water, the starch was stained a shade lighter and was easier to view. In addition, there were several starch reference slides that were provided by the OSBI and

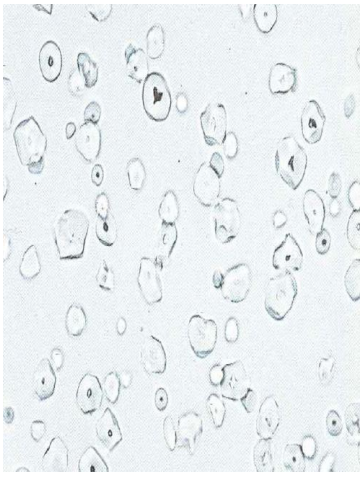
examined to use as a comparison to the slides of the condom residue smears. Figure 7 illustrates pictures of common condom particulates obtained from a microscopy atlas in the OSBI Trace Evidence Lab.



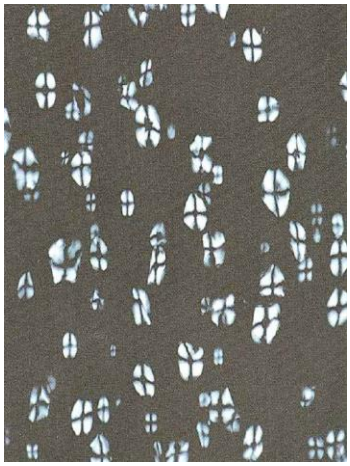
(a)



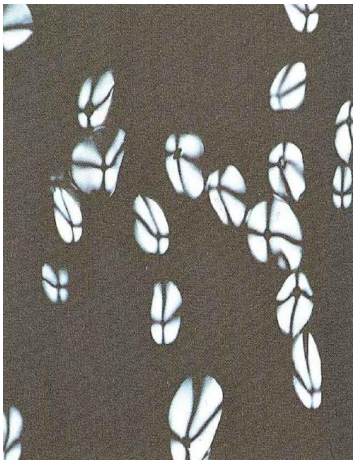
(b)



(c)



(d)



(e)

Figure 7. Common condom particulates (a) talc (b) lycopodium (c) starch (d) starch under crossed polars at low magnification (e) starch under crossed polars at high magnification.

**Gas Chromatography/Mass Spectrometry**

**Liquid-liquid extraction**. The following extraction steps were developed, using those outlined by the unpublished work by Keil et al. as a guide:

1. The condom was rinsed with 5 mL of Tris buffer (pH 7.5).
2. A 1 mL aliquot of the solution was transferred to a test tube and combined with 1 mL ammonium hydroxide buffer (pH 8.9) and 5 mL reagent grade ethyl ether/ethyl acetate (1:1).
3. The test tube was agitated for 2 minutes using both a vortex mixer and inverting the test tube manually.
4. The organic layer was removed from the top, transferred to 4 mL vial, and evaporated under nitrogen.
5. The residue was derivatized by adding BSTFA + 1% TMCS.
6. A 1 µL manual injection was made into the injection port of an H.P. 5890 Series II GC with an H.P. 5972 MS detector with a Varian VF-5MS, 25 m x 0.25 mm x 0.25 µm column, and run under the following conditions:

| | |
|---|---|
| GC injector temperature | 270°C |
| GC program | 100°C (hold 2 min) to 300°C (hold 5 min) |
| | Rate 20°C/min |
| Split ratio | Splitless |
| Manual injection | Mode: full scan |
| Scan range | 50.00-550.00 *m/z* |

Attempts were made to obtain the same results as Keil et al. As the steps detailed above are somewhat vague, the exact procedure used by the authors is unclear. Questions arising are: How much of the solvent was evaporated in Step 4? How much derivatizing agent was added in Step 5? How long was the solution allowed to stand? Was there a solvent added back to the derivatized wash before injecting onto the column? Why was each step performed?

Several attempts were made to contact the authors, Wolfgang Keil and Andrea Berzlanovich, to obtain additional details regarding their experimental procedure. The attempted correspondence met with no success until much later in the experimentation portion of this research, and even then some questions remained. Because of uncertainty related to the detail of the Keil/Berzlanovich protocol, steps were varied to determine what method produced the best results. The correspondence with these authors is detailed in *Appendix B.*

Initially, the steps outlined above were performed on a Durex Tingling Pleasure condom. The extract was placed in a round-bottom flask and evaporated to dryness. Approximately 1 mL BSTFA was added, along with 2 mL reagent grade methylene chloride. The solution was stirred for 15 minutes, and then a 1 µL injection was made onto a Varian VF-1ms 12 m x 0.2 mm x 0.33 µm GC column and ran under the following conditions:

| | |
|---|---|
| GC injector temperature | 230°C |
| GC program | 100°C (hold 2 min) to 250°C (hold 5 min) |
| | Rate 20°C/min |
| Split ratio | Splitless |
| Manual injection | Mode: full scan |
| Scan range | 50.00-550.00 *m/z* |

(The lower GC injector temperature was chosen to reduce column bleed, because the column that was used was not the same as that used by Keil, et al.) A few small peaks were observed on the total ion chromatogram (TIC) [Figure E1: DUXTPEX]. Since the peaks were in such small abundance, and the chromatogram looked nothing like those shown in the reference material, it was decided that each step should be performed in sequence to determine which step might have been performed improperly. As an initial explanation, all the steps were eliminated, except for

the Tris washing step. A 1 mL aliquot of the Tris buffer/Durex TP washing solution was mixed with 0.5 mL methanol. A 1 µl injection was made and run under the same conditions described above. There were no visible peaks observed on the TIC.

The second effort in this line of reasoning involved remodeling to a more amenable approach for use in crime labs. A Durex Tingling Pleasure condom was swabbed along the length of the outer surface. The swab was inserted into a test tube containing 1 mL Tris buffer and mixed by inversion for 1 minute. Next, 1 mL ammonium buffer was added, along with 5 mL of ether/ethyl acetate (1:1). The test tube was inverted, and bubbles were made by squeezing the pipette at the bottom of the test tube to create a thorough mixture. The organic layer was removed from the top and placed into a vial. A 1 mL aliquot was transferred to a GC vial and 0.5 mL HPLC grade methanol was added. The solution was mixed by inverting the tube, and a 1 µL injection was made and run under the same conditions described above. There were no visible peaks seen on the TIC [Figure E2: DUREXETH].

As a third experiment, a Kimono condom was swabbed along the length of the outer and inner surface to increase the amount of recovered residue. The tip of the swab, just above the cotton, was cut with a razor blade and placed into a vial. Then, 5 mL of Tris buffer were added, and the vial was vortexed for 2 minutes. A 1 mL aliquot was transferred to a test tube and combined with 1 mL ammonium buffer and 5 mL ether/ethyl acetate (1:1). The test tube was vortexed for 2 minutes, and the organic layer was transferred to a separate vial. The solvent was evaporated to dryness under nitrogen gas, and the vial was placed in a luke-warm bath to speed up the evaporation process. Then, 250 µL of BSTFA with 1% TMS were added to the residue, and the resulting solution was vortexed for 2 minutes. A 1 µL injection was made onto a new GC column Varian VF-5MS, 25 m x 0.25 mm x 0.25 µm, which was the same column used by

Keil et al., and ran under the same conditions [Figure E3: KIMONC]. The process was repeated for a Trojan condom [Figure E4: TROJNC]. The remaining derivatized Trojan extract was then allowed to stand for 24 hours and rerun through the GC/MS. A similar chromatogram to the 2 minute derivatization was obtained [Figure E5: TROJON]. In each of these runs, there was an initial large solvent peak observed from the BSTFA, which caused excessive tailing. This made the observation of some of the peaks on the chromatograms difficult. It was decided that a solvent delay should not be performed until the location of all vital peaks could be determined.

Two other runs were performed on the extract at carrier gas flow rates of 3 mL/min [Figure E6: TROJFR3] and 6 mL/min [Figure E7: TROJFR6] to see if this would decrease the tailing of the solvent. The results were that the variations in flow rate showed no significant improvement in the chromatograms.

Next, 1 mL of the Tris buffer wash of the Trojan condom that was previously prepared was extracted using the outlined steps. It was evaporated to dryness under nitrogen gas, and then 50 µL BSTFA was added (the amount of BSTFA was reduced in hopes that the solvent peak on the TIC would be smaller). The test tube was vortexed for 2 minutes, and crystals were seen forming. Therefore, 1 mL methylene chloride was added while vortexing until most of the solid was dissolved. A 1 µL injection was made on the column and run under the same conditions. There was still a large solvent peak on the chromatogram, and in fact, there were no other peaks observed [Figure E8: TROJ5D].

As the next experiment in this series, a 1 mL ammonium hydroxide buffer wash was performed on a Trojan condom. Another 2 mL ammonium hydroxide buffer were added to the test tube, along with 1 mL methylene chloride. The test tube was vortexed for 2 minutes, and then the organic layer was removed. The derivatizing step was bypassed, and a 1 µL injection of

the organic layer was made onto the column and run under the parameters described above [Figure E9: TROJMC]. There were large equidistance peaks observed on the TIC, which were all identified as heptasiloxane, and the tailing of the solvent was not as broad.

A Contempo Bareback condom was then rinsed and the usual extraction steps followed. The wash was derivatized with 150 µL BSTFA, and a 1 µL injection was made onto the column and ran under the same parameters [Figure E10: CONBBNC]. There were many peaks present on the TIC, with some still hidden by the large solvent peak. It was noted that the extract had some undissolved solids on the wall. The surface was scraped, and the vial was agitated for 2 minutes. The solids did not completely dissolve, so 500 µL of hexane were added and the vial agitated. Not all of the solids were dissolved, but still a 1 µL injection was made onto the column and run under the same conditions [Figure E11: CONBBHX]. The solvent peak was reduced on the TIC, as well as the abundance of the other peaks.

Next, a Contempo Bareback extraction was performed, and the solvent was evaporated to ~250 µL. The wash was derivatized with 50 µL BSTFA, and the vial was vortexed for 2 minutes. A 1 µL injection was made onto the column and run under the same conditions [Figure E12: CONBBEV].

Following on, a Kimono wash was made by rinsing the outside surface of the condom with 1 mL ammonium buffer into a beaker (the surface of the condom was washed, opposed to swabbing, in hopes that this would increase the amount of residue that was extracted). An additional 2 mL ammonium hydroxide buffer were added to the test tube, along with 1 mL methylene chloride. The organic layer was removed and placed in a separate vial after 2 minutes of vortexing. A 1 µL injection was made onto the column and run under the described conditions. [Figure E13: KIMMECL]. There were only two peaks present on the TIC. In an

effort to secure a stronger signature, a Kimono condom wash was performed by rinsing the outside surface with 1 mL ammonium hydroxide buffer into a beaker. The condom was then swirled around inside the beaker with an additional 2 mL ammonium hydroxide buffer. The wash was transferred to a test tube and combined with 5 mL ether/ethyl acetate (1:1). The test tube was agitated for 2 minutes, and the organic layer was removed and placed into a vial. A 1 µL injection was made onto the column and ran under the same parameters. The *m/z* range was changed from 50.00-550.00 to 50.00-400.00 to try and improve the abundance of peaks [Figure E14: KIMEAE]. The change in the *m/z* range appeared to have no effect. The Kimono extract was evaporated to ~100 µL and a 1 µL injection was made onto the column [Figure E15 through E18: KIMEVAP]. This procedure produced remarkable results with a chromatogram that had multiple peaks of various compounds.

**Solid-phase microextraction.** SPME was utilized as an alternative method to liquid-liquid extraction, though the steps outlined by Keil et al. were still used as a guide.

***Polydimethylsiloxane fiber.*** A Supelco® SPME fiber with a 7 µm polydimethylsiloxane (PDMS) bonded phase coating, which is designed for non-polar high molecular weight compounds, was the initial fiber applied in this study. The fiber, contained in a manual holder, was conditioned by inserting it into the injection port of the GC/MS at 230°C for 5 minutes. The previously described SPME method was then applied on the various condoms, using the following procedure:

1. The surface of the condom was rinsed with 1 mL ammonium hydroxide buffer (pH 8.9) into a beaker.
2. The extract was transferred to a 20 mL vial and diluted with 14 mL ammonium buffer.
3. The fiber was inserted into the solution while stirring for exactly 30 minutes.

4. The fiber was inserted into an H.P. 5890 Series II GC with an H.P. 5972 MS detector with a Varian VF-1ms 12 m x 0.2 mm x 0.33 µm column, allowed to desorb for 3 minutes, and then run under the following parameters.

| | |
|---|---|
| GC injector temperature | 230°C |
| GC program | 100°C (hold 2 min) to 250°C (hold 5 min) |
| | Rate 20°C/min |
| Split ratio | Splitless |
| Manual injection | Mode: full scan |
| Scan range | 50.00-550.00 *m/z* |

5. The fiber was conditioned after the run for use in a subsequent analysis by inserting it into the injection port of the GC/MS for 5 minutes at 230°C.

A negative control was performed by inserting the fiber into 15 mL ammonium hydroxide buffer solution for 30 minutes. The fiber was then allowed to desorb for 3 minutes in the injection port of the GC/MS at a temperature of 230°C. As expected, no peaks were seen on the TIC [Figure F1: BUFFER], illustrating the fiber itself was not degrading in the injection port, nor was the ammonium hydroxide buffer solution contaminated with organics.

Chromatograms and spectra were then obtained for the Trojan [Figure F2. TROJ1], Kimono [Figure F3. KIMO1], and Durex Tingling Pleasure [Figure F4. DUREXTP] washes.

*Derivatization*. On-fiber derivatization was also tested by means of SPME, using the steps outlined by Stenerson as a guide. The headspace of a 4 mL GC vial was saturated with derivatizing agent by adding 10 µL BSTFA + 1% TMCS and allowing it to stand covered at room temperature for ~30 minutes. The PDMS fiber was inserted into a Trojan wash solution for 30 minutes. The outside of the fiber was dried with a Kimwipe® and inserted into the headspace

of the BSTFA vial by piercing the plastic septum of the vial lid. The fiber was allowed to adsorb for 20 minutes and was then desorbed in the injection port of the column for 3 minutes and run under the parameters described above [Figure F5: TROJ_DER]. The process was thus repeated using a Durex Tingling Pleasure wash solution and ~1 hour 20 minutes headspace equilibrium time [Figure F6: DUTP_DER]. It was found that it was necessary to condition the fiber for 10 minutes between runs after on-fiber derivatization, as opposed to 5 minutes when no derivatization was performed.

*Tris Buffer.* SPME was also used to test washes performed using the Tris buffer solution. A Durex Tingling Pleasure condom was rinsed with 1 mL Tris buffer (pH 7.5) and 1 mL ammonium hydroxide buffer. The wash was diluted by adding 13 mL distilled water. The fiber was inserted into the solution for 30 minutes. The injection temperature of the GC/MS was increased to 270°C and the final temperature to 300°C to match the parameters of Keil, et al. The fiber was inserted into the injection port of the GC/MS for 3 minutes with subsequent analysis [Figure F7: DUTPTRIS]. Due to the low abundance of peaks on the TIC, a new SPME fiber was placed in the holder in order to rule out fiber deterioration as the cause for the low signal. Runs were performed on Kimono [Figure F8: KIMONO4] and Contempo Bareback [Figure F9: CONTBB] washes using the increased temperature conditions. Similar chromatograms were obtained with the increased temperatures, and some GC column bleed was observed as indicated by the presence of the increased height of the baseline on the TIC at higher temperatures.

***Polydimethylsiloxane/divinylbenzene fiber.*** A Supelco® SPME fiber with a 65 µm polydimethylsiloxane/divinylbenzene (PDMS/DVB) partially cross-linked coating, which is designed for volatile or polar organics, was next utilized. The fiber, contained in a manual

holder, was conditioned by inserting it into the injection port of the GC/MS fitted for 30 minutes at 250°C, following the conditioning protocol in the manufacturer's manual. The SPME method was then performed, implementing the following procedure:

1. The surface of the condom was rinsed with 0.5 mL of ammonium hydroxide buffer (pH 8.9) into a 30 mL beaker and the condom manipulated in the beaker to optimize the extraction of residue.
2. The inside of the condom wrapper was rinsed with an additional 0.5 mL ammonium hydroxide buffer into the same beaker.
3. The extract was diluted with 14 mL distilled water and transferred to a 20 mL vial.
4. The fiber was inserted into the solution while stirring for exactly 30 minutes.
5. The fiber was inserted into an H.P. 5890 Series II GC with an H.P. 5972 MS detector with a Varian VF-5MS, 25 m x 0.25 mm x 0.25 µm column, allowed to desorb for 3 minutes, and then run under the following parameters:

| | |
|---|---|
| GC Injector temperature | 270°C |
| GC program | 100°C (hold 2 min) to 300°C (hold 5 min) |
| | Rate 20°C/min |
| Split ratio | Splitless |
| Manual Injection | Mode: full scan |
| Scan range | 50.00-550.00 *m/z* |

6. The fiber was conditioned after the run by inserting it into the injection port for 10 minutes under the following parameters:

| | |
|---|---|
| GC Injector temperature | 270°C |
| GC program | 300°C |

Split ratio 1000:1

Chromatograms were obtained from washes of Trojan [Figure G1: TPDMSDVB], Kimono [Figure G2: KPDMSDVB], and Contempo Bareback [Figure G3: CBPDMDVB] condoms using the scheme described above and displayed significant differences.

***Polyacrylate fiber.*** A Supelco® SPME fiber with an 85 µm polyacrylate (PAC) partially cross-linked coating, which is designed for polar semivolatiles, was next utilized. The fiber, contained in a manual holder, was conditioned by inserting it into the injection port of the GC/MS for 1 hour at 280°C, following the conditioning protocol in the manufacturer's manual. The extraction procedure was then performed using the same steps as those that were followed using the PDMS/DVB fiber. The chromatograms obtained from washes of the Trojan [Figure H1 through Figure H4: TROJPAC], Kimono [Figure H5 through Figure H10: KIMPAC], and Contempo Bareback [Figure H11 through Figure H15: CONBBPAC] condoms displayed chromatograms similar to those obtained from the runs using the PDMS/DVB fiber. The PAC fiber was then used to analyze the remaining condom brands: Durex Warming Pleasure [Figure H16 through Figure H18: DURWPPAC], Durex Tingling Pleasure [Figure H19 through Figure H22: DURTPPAC], Durex Natural Feeling [Figure H23 through Figure 26: DURNFPAC], Durex Her Sensation [Figure H27 through Figure H32: DURHSPAC], Contempo Vanilla [Figure H33 through Figure H36: CONVPAC], Contempo Strawberry, [Figure H37 through Figure H40: CONSPAC], and Contempo Banana [Figure H41 through Figure H44: CONBPAC].

A negative control was prepared by inserting the fiber into a 20 mL vial containing 1 mL ammonium hydroxide buffer solution and 14 mL distilled water for 30 minutes while stirring. The fiber was then allowed to desorb for 3 minutes in the injection port of the GC/MS [Figure 45

and Figure H46: NCPAC]. There was only one insignificant peak observed on the TIC, identified as 3-(2,2-dimethyl-propyl)-3-methyl-2,2-diphenyl-oxetane.

The extraction process implementing the PAC fiber was repeated for some of the condom brands with boxes that displayed different lot numbers from those previously explored. This was performed to investigate lot-to-lot variability for the condom brands analyzed in this study. Unfortunately, the Contempo Luscious Flavors condoms were discontinued, so another lot of that brand (Vanilla, Strawberry, and Banana) could not be obtained. Several attempts were made to obtain different lot numbers for the Durex Play Sensations condoms (Tingling Pleasure, Her Sensation, Natural Feeling, and Warming Pleasure) but were unsuccessful. In more detail, this brand was ordered from two online stores, Condom Jungle and Under Cover Condoms, on three separate occasions, but all six condom boxes were from the same lot. Condom Jungle is located in Los Angeles, California, and Under Cover Condoms is located in Columbus, Ohio, so it seemed the distribution location was not the factor. Local stores were also searched, but this brand could not be located in an Oklahoma City Wal-Mart, Walgreens, or CVS. The condom samples for all of the runs that were performed using the PAC fiber and their corresponding lot numbers [Figure H47: TROJ2PAC, Figure H48: KIM2PAC, and Figure H49: CNBB2PAC] are listed in Table 2.

Table 2

*Condom Lot Numbers – SPME w/ PAC Fiber*

| Brand | GC/MS Run | Box Lot # | Wrapper Lot # |
|---|---|---|---|
| Trojan Thintensity | TROJPAC | DA9299GL3 | DA9299GL3 |
| | TROJ2PAC | DA0127GL6 | DA0127GL6 |
| Kimono Select | KIMPAC | 90656-9 | 90656-9 |
| | KIM2PAC | 00957-9 | 00957-9 |
| Contempo Bareback | CONBBPAC | 0912042916 | 0912042916 |
| | CNBB2PAC | 1006092516 | 1006092516 |
| Durex Warming Pleasure | DWPPAC | T9110 | TJR9120 |
| Durex Tingling Pleasure | DURTPPAC | T9110 | TRL9050 |
| Durex Her Sensation | DURHSPAC | T9110 | TK9008 |
| Durex Natural Feeling | DURNFPAC | T9110 | TJR9020 |
| Contempo Vanilla | CONVPAC | 0809752200 | 0609072816 |
| Contempo Strawberry | CONSPAC | 0809752200 | 0602052616 |
| Contempo Banana | CONBPAC | 0809752200 | 0607100616 |

***Swabbed samples.*** Last, a technique was developed for analyzing swabbed samples, which would be the type of evidence submitted to an actual crime laboratory. A Durex Tingling Pleasure condom was swabbed the length of the condom. The tip of the swab was cut in half (not including the wooden handle) with a razor blade and one half placed in a small test tube with 1 mL ammonium hydroxide buffer (pH 8.9). The test tube was vortexed for 2 minutes, transferred to a 20 mL vial, and diluted with 14 mL distilled water. The solution was adsorbed with a polyacrylate fiber for 30 minutes while stirring, desorbed on the column for 3 minutes,

and run through the GC/MS under the same parameters as the reference samples [Figure H50: DTPSBPAC]. There were very few peaks seen on the TIC, so the process was repeated using a 5 mL GC vial to hold the extract during the adsorption process. This allowed the fiber to be fully immersed without having to dilute with as much water. The chromatogram still had very few peaks [Figure H51: DTPSPAC2]. There were small pieces of cotton fibers seen sticking to the fiber during the extraction process that might have caused a limited amount of the extract to be adsorbed. Therefore, the process was repeated and the extract was filtered before adsorbing with the fiber. This time, the length of the condom was swabbed, as well as the inside of the wrapper (this step was performed to determine if the swab could retain additional residue). The swab was left whole and placed into the test tube. A 2 mL aliquot of ammonium hydroxide added to the test tube, and it was vortexed for 2 minutes. The extract was filtered into the vial, and then 2 mL distilled water were used to rinse the test tube and ran through the filter into the vial. The extract was adsorbed for 30 minutes while stirring, desorbed on the column for 3 min, and ran through the GC/MS [Figure H52: DURTPSPAC3]. This run produced larger peaks on the TIC. The process was then repeated with a Trojan condom [Figure H53: TROJSPAC].

**Isotope-Ratio Mass Spectrometry**

IRMS was conducted at the University of Oklahoma Conoco-Phillips School of Geology and Geophysics in the Sarkeys Energy Center by Dr. Richard Philp and Dr. Anne Warren. The samples analyzed were hexane washes of the various condoms examined in the current work, weighed to confirm that masses greater than 1 mg, which is the detection limit of the IRMS, had been secured. As the initial foray into this aspect of the study, a Durex Her Sensation condom was rinsed with 0.5 mL hexane into a 30 mL beaker and manipulated inside the beaker to obtain the maximum amount of residue possible. The extract was transferred to a

previously weighed GC vial, evaporated to dryness under nitrogen, and the vial reweighed. The residue weight was ~14 mg. The process was repeated with a Kimono condom, which resulted in a residue mass of ~35 mg. It was determined that the two different condom washes produced much more residue than needed to conduct the analysis, so it was assumed that each of the other condom brands would yield similar results.

A set of condom samples of the five brands were washed with Sigma-Aldrich Reagent Plus (≥99%) hexane. Each condom surface was washed with 0.5 mL hexane into a 30 mL beaker and then manipulated inside the beaker. The inside of the wrapper was then washed with 0.5 mL hexane into the same beaker. The washes were transferred to GC vials and then evaporated to ~50 µL. The condom samples were analyzed on a Finnigan MAT 253 stable isotope ratio mass spectrometer. The $\delta^{13}C$ bulk number for each sample, meaning the average $\delta^{13}C$ from all the carbon-contributing components of the condom residue, was measured. The condom samples that were analyzed and their corresponding lot numbers are listed in Table 3. A second run was performed on a second lot for the Contempo Bareback, Kimono, and Trojan brands. Analysis on different lot numbers from the initial runs was performed in order to explore lot-to-lot variability. Condom sub-brands were not analyzed, because the analysis is expensive and this effort is exploratory in nature. A second lot could not be obtained for the Durex Play Sensations, and the Contempo Luscious Flavors have been discontinued, thus additional units with different lot numbers are not available.

Table 3:

*Condom Lot Numbers - IRMS*

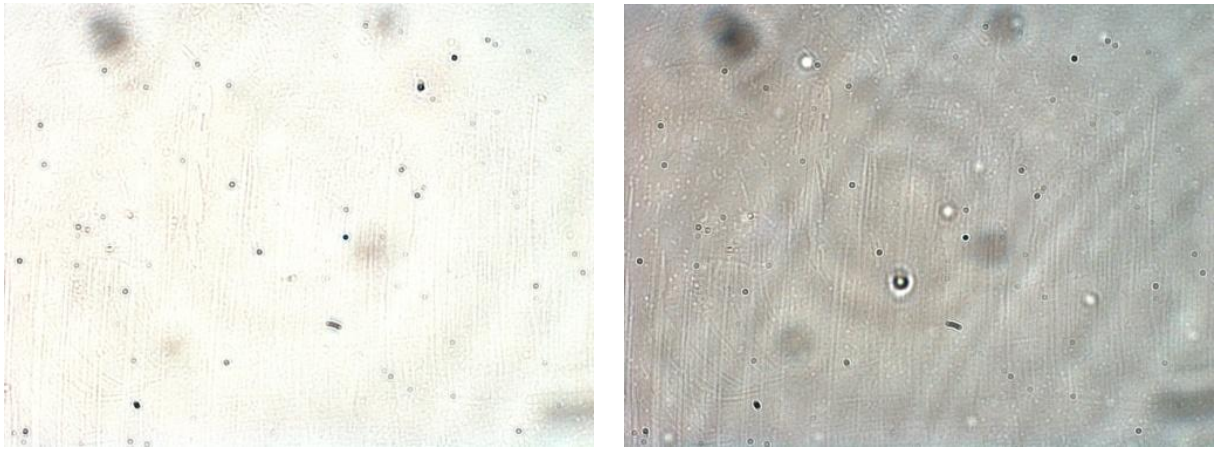
| Brand | IRMS Run | Box Lot # | Wrapper Lot # |
|---|---|---|---|
| Contempo Bareback | 1 | 0912042916 | 0912042916 |
| | 2 | 1006092516 | 1006092516 |
| Kimono Select | 1 | 90656-9 | 90656-9 |
| | 2 | 00957-9 | 00957-9 |
| Trojan Thintensity | 1 | DA9299GL3 | DA9299GL3 |
| | 2 | DA0127GL6 | DA0127GL6 |
| Durex Her Sensation | 1 | T9110 | TK9008 |
| | 2 | -------- | -------- |
| Contempo Banana | 1 | 0809752200 | 0607100616 |
| | 2 | -------- | -------- |

## Results and Discussion

### Polarized Light Microscopy

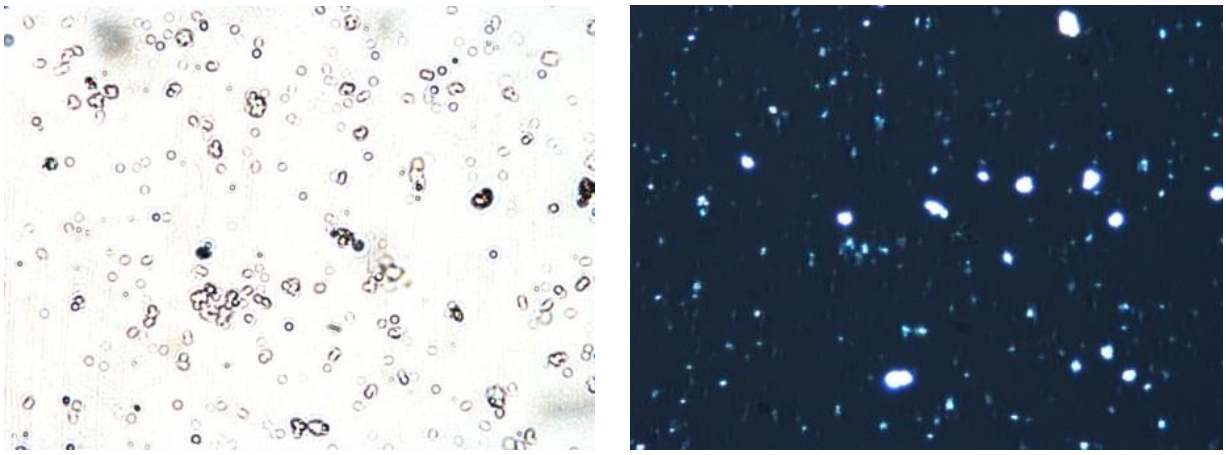
Under plane-polarized light, the Kimono, Durex, and Trojan brand smears had clear, round to irregular-shaped particulates. Under crossed polars, it was determined that these particles were birefringent. However, they did not display the Maltese cross patterns typical of cornstarch. Some of these smears are illustrated in Figure 8 through Figure 11. Each of the Contempo brand condom smears contained particulates consistent with starch. Figure 12 and Figure 14 are photographs of the Contempo Vanilla and Contempo Strawberry smears under crossed polars, respectively. Figure 15 and Figure 16 are of the Contempo Banana smear and illustrate the effects of staining starch with undiluted Lugol's solution. It was found that standard Lugol's solution turned the starch black, but it was anticipated that the solution would

turn the starch blue. Figure 17 and Figure 18 are of the Contempo Bareback smear stained with diluted Lugol's solution. As the photograph illustrates, a dilute solution of Lugol's produces stained particles of easier viewing.



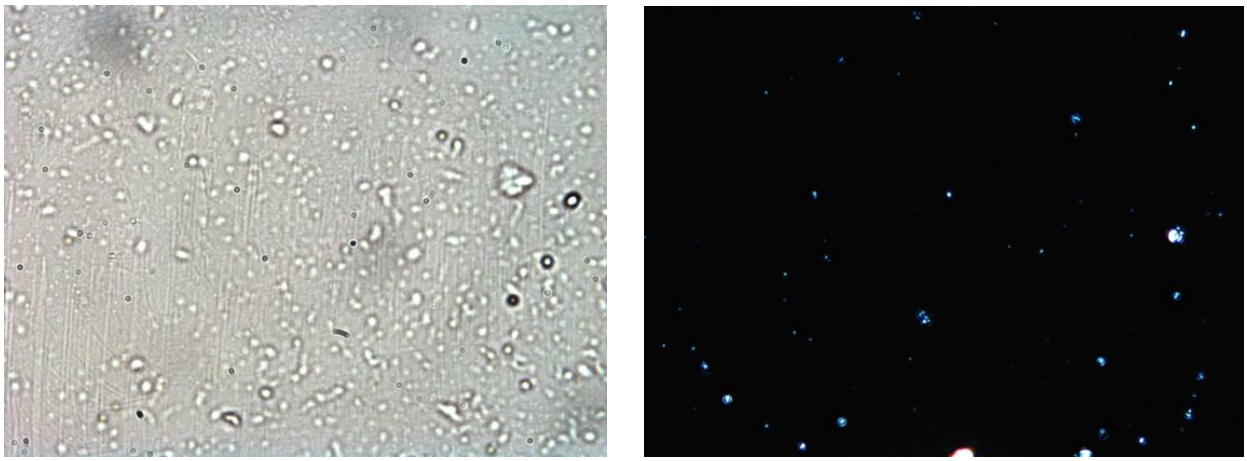
(a) (b)

Figure 8. Kimono smear under plane-polarized light (a) 40x (b) 63x.



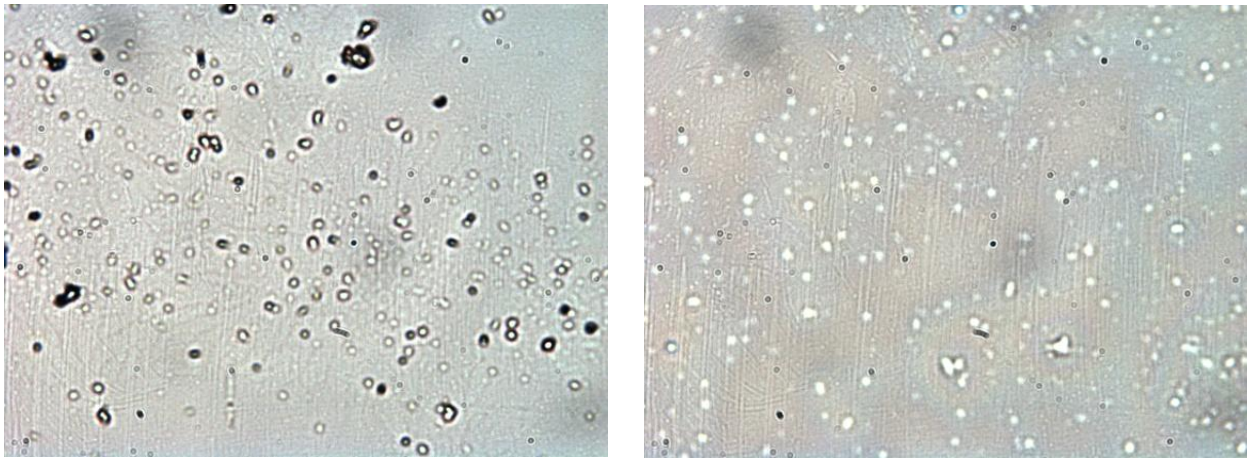
(a) (b)

Figure 9. Durex Warming Pleasure smear under (a) plane-polarized light 63x (b) crossed polars 40x.



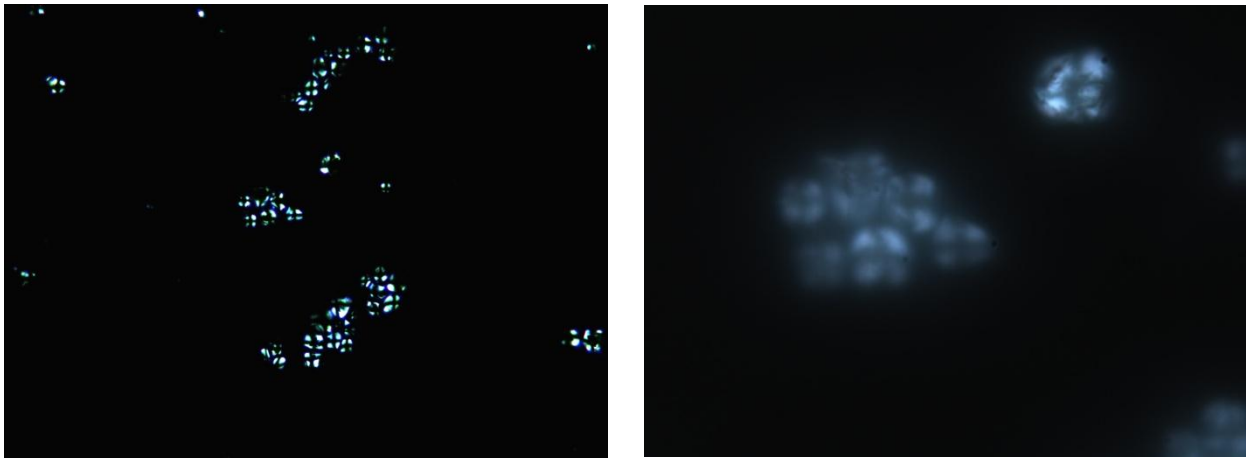
(a) (b)

Figure 10. Durex Tingling Pleasure smear under (a) plane-polarized light 63x (b) crossed polars 63x.



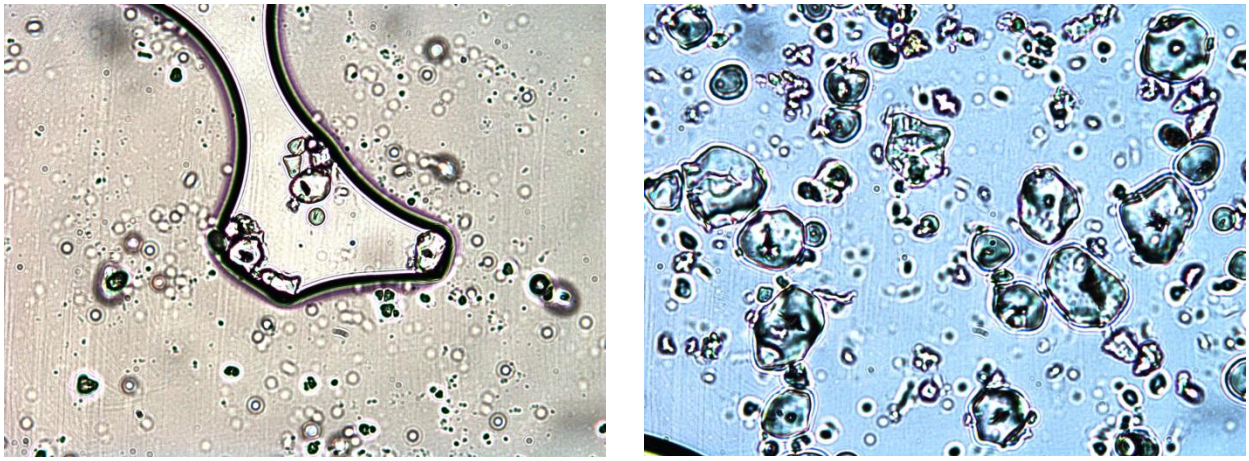
(a) (b)

Figure 11. Durex (a) Natural Feeling smear under plane-polarized light 63x (b) Her Sensation under plane-polarized light 63x.



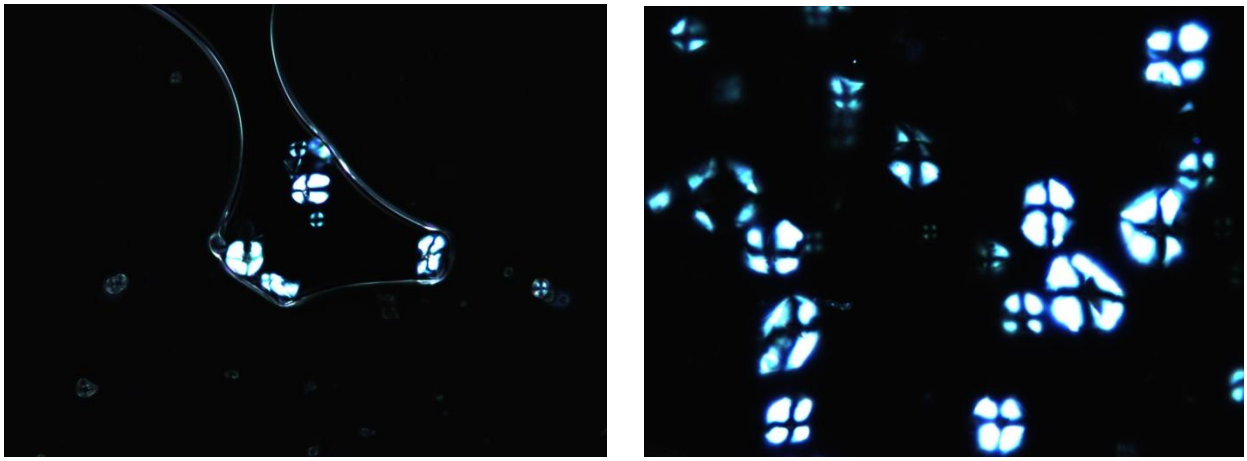
(a) (b)

Figure 12. Contempo Vanilla smear under crossed polars (a) 40x (b) 63x.



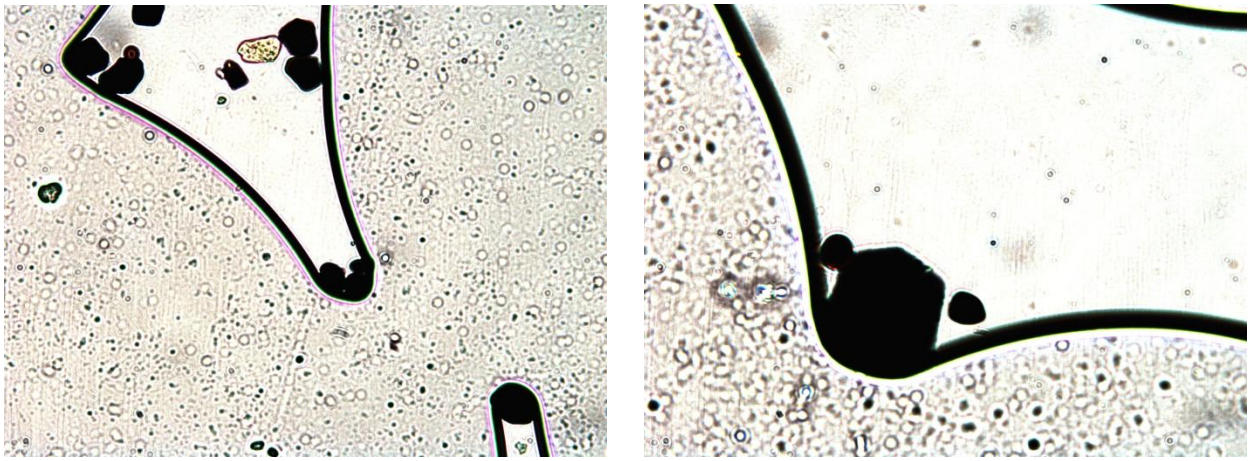
(a) (b)

Figure 13. Contempo Strawberry smear under plane-polarized light (a) 40x (b) 63x.



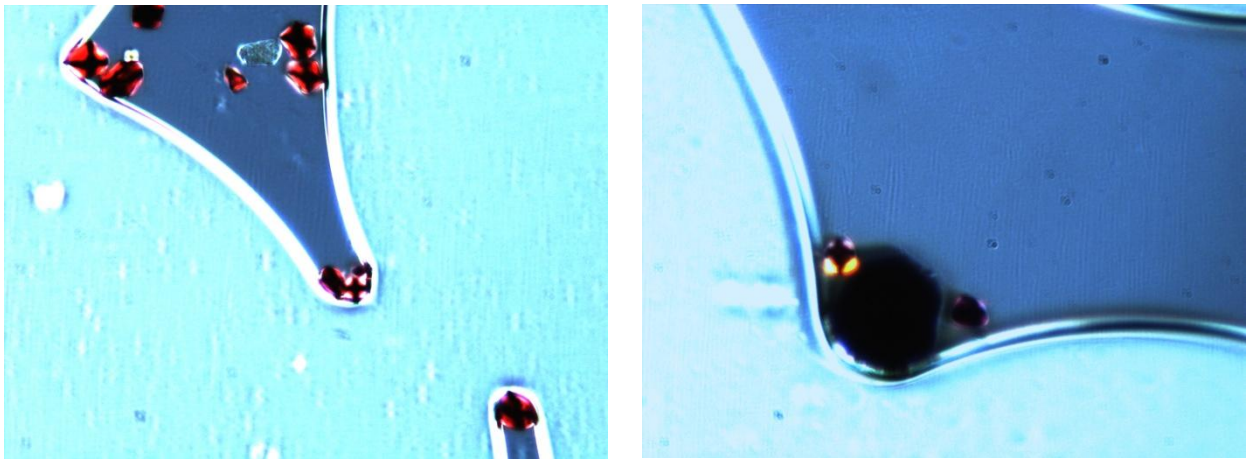
(a) (b)

Figure 14. Contempo Strawberry smear under crossed polars (a) 40x (b) 63x.



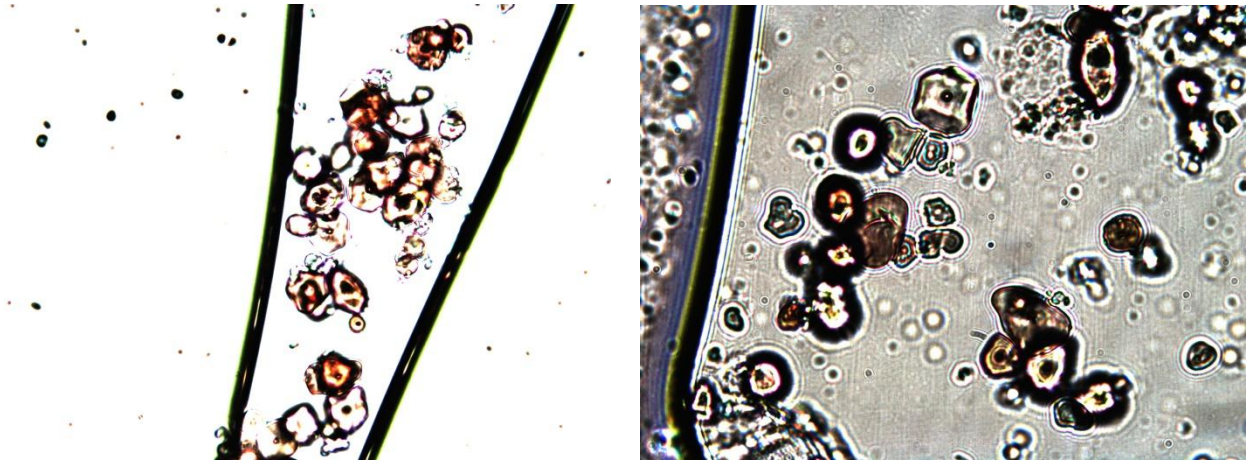
(a) (b)

Figure 15. Contempo Banana smear stained with Lugol's solution under plane-polarized light (a) 40x (b) 63x.



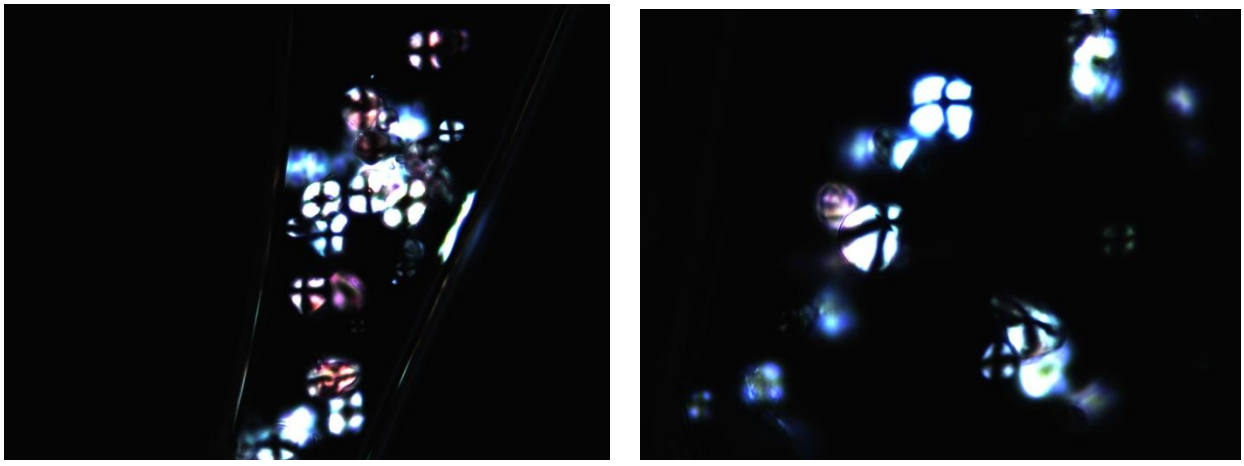
(a) (b)

Figure 16. Contempo Banana smear stained with Lugol's solution under crossed polars (a) 40x (b) 63x.



(a) (b)

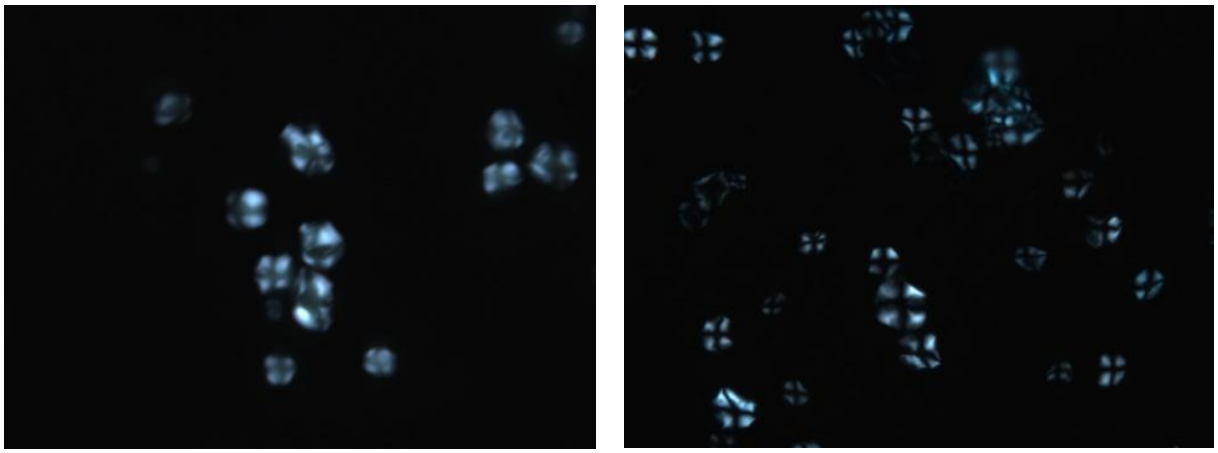
Figure 17. Contempo Bareback smear stained with Lugol's solution under plane-polarized light (a) 40x (b) 63x.



(a) (b)

Figure 18. Contempo Bareback smear stained with Lugol's solution under crossed polars (a) 40x (b) 63x.

The various starch reference samples had differences in appearance under crossed polars. The cornstarch particles appeared round to irregular in shape and displayed Maltese cross patterns. Sweet potato starch also had Maltese cross patterns but was much more perfectly round in appearance. Black wheat starch had an irregular-shaped pattern with variations in particle sizes. Bean starch displayed no Maltese cross pattern but appeared as solid bright orbs under crossed polars. The cornstarch reference slides, illustrated in Figure 7, are similar in appearance to the starch identified in the Contempo condom smears. The hazy appearance of the unmounted starch in Figure 19 shows the importance of mounting the condom residues in at least distilled water.



(a) (b)

Figure 19. Known starch particles under crossed polars (a) unmounted (b) mounted in Meltmount.

The forensic science literature has described cornstarch as the most common particulate used in the powdering step during condom manufacturing (Blackledge & Vincenti, 1994; Keil, 2007). Therefore, it was expected that more of the condom brands would have starch located in their residues. However, only the Contempo brands (one manufactured in India and the other in Thailand) had starch detected with the PLM, and none of the brands had lycopodium present. This suggests that the ingredients in the powdering step of condom production might have changed since the references were published. It may now be the case that searching for the presence of starch or lycopodium may no longer be a good means of screening for condom residues in sexual assaults. However, their presence may still be used as a tool for classifying condom brands. The colorless particles that were seen under plane-polarized light but did not display Maltese cross patterns under crossed polars could be polyethylene or silica, used as fillers for latex condoms (Keil, 2007). Unfortunately, the presence of these compounds cannot be confirmed with a polarized light microscope.

## Gas Chromatography/Mass Spectrometry

**Liquid-liquid extraction.** Numerous attempts were made to duplicate the alkaline liquid-liquid extraction technique described by Keil et al. The authors stated that the chromatograms of the 54 condoms they examined barely differed, but they were still able to store the mass spectra from 5 to 11 peaks of each chromatogram into a "data bank." They relayed that they were then able to analyze simulated case samples and correctly identify the brand with 95% accuracy using the "data bank." Though many variations to their method were performed in this work, the results described by these authors could not be replicated. Perhaps the principle problem was the large tailing of the solvent peak associated with the BSTFA derivatizing agent. When derivatization was performed, there was a significant amount of tailing up to 8 minutes on the chromatograms, likely hiding some of the underlying peaks. It was speculated that the Tris buffer was used by the authors as a preservative for possible DNA evidence that could be present on swabs in actual casework. The ammonium buffer was most likely added to further charge any DNA that is present, so as to enable separation of the relatively non-polar condom residues from biological materials. In theory, derivatization would then enable the condom residues to chromatograph better and create more individuality in their corresponding mass spectra. However, it was found that satisfactory results were obtained when the actual condom was rinsed with 1 mL ammonium hydroxide with no Tris buffer used, the extracting solvent was evaporated to ~100 µL, and no BSTFA was added. Multiple components of the residue were observed on the TIC [Figure E15], other than heptasiloxane, including one large peak around 8.010 minutes, identified as butylated hydroxytoluene [Figure E16], a small peak at 10.738 minutes, identified as *n*-hexadecanoic acid [Figure E17], and a peak at 11.782 minutes, identified as octadecanoic

acid [Figure E18]. Ultimately, the liquid-liquid extraction method was deferred for the simpler technique of SPME.

**Solid-phase microextraction.** The use of SPME was explored in lieu of liquid-liquid extraction, as it is a fairly new technique that is becoming more prevalent in laboratory settings. This extraction method is simple and takes up less time of hands-on work than liquid-liquid extraction (Pawliszyn, 1997). When the extraction steps were performed using the PDMS fiber, the chromatograms that were produced had equidistant peaks similar to those described by the reference. This is illustrated in Figure F2 through Figure F9. The peaks of different brands had similar retention times and mass spectra to one another. There were slight differences among condom brands in the intensities of some of the peaks in the mass spectra, but the major peaks were all identified as heptasiloxane. Though, it is most likely that the National Institute of Standards and Technology (NIST) mass spectral library is not broad enough to differentiate various lengths of siloxanes. The mass spectrometer does not scan above 500 *m/z*, so the high molecular weights of the siloxanes would cause the parent peaks on the spectra to be out of the range of the scale. Keil et al. stated that their molecular ion peaks were never identified, but their mass spectra looked very similar to that of heptasiloxane, with mass increments of 74 amu between major fragments. So, it is still uncertain how the authors were able to build a database that could differentiate brands by using this information alone.

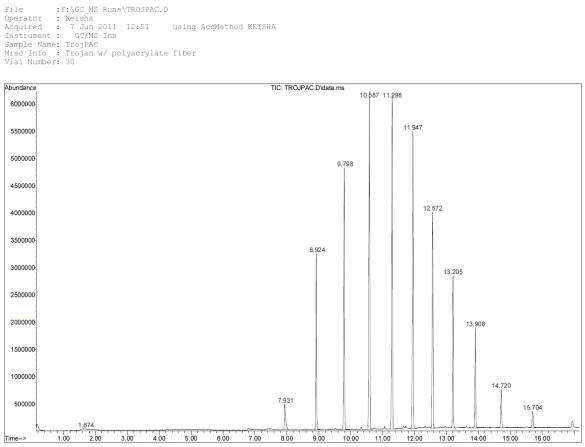
It was determined that the PDMS/DVB and the PAC SPME fibers produced similar results for extracting multiple components in condom residues. When the PAC fiber was implemented for the analysis of all ten of the condom brands and sub-brands, it was found that each of the five brands produced distinct chromatograms. Some of the condom sub-brands' spectra were also found to be unique. The Trojan brand produced a TIC that was similar to those

obtained for all the brands using the PDMS fiber [Figure H1]. There were equidistant peaks in a bell-shaped curve pattern similar to chromatograms obtained for the aliphatic carbons of gasoline. The peaks were all identified as heptasiloxane [Figure H2 through Figure H4]. Though as stated before, this is perhaps due to the library's inability to distinguish multiple siloxane chain lengths. Just as in gasoline, the peaks of siloxane oligomers would be expected to have higher retention times as the chain lengths increase. The Trojan condom was the only brand to display this pattern. The Kimono brand [Figure H5] had a large peak at 7.975 minutes, identified as butylated hydroxytoluene [Figure H6]. This was the only brand to have this compound present. There was also a peak at 10.717 minutes, identified as n-hexadecanoic acid [Figure H7], with the remaining equidistant peaks identified as heptasiloxane [Figure H8 through Figure H10]. The Contempo Bareback TIC [Figure H11] had an *n*-hexadecanoic acid peak at 10.719 minutes [Figure H14], an octadecadienoic acid peak at 11.664 minutes [Figure H15], and the remaining peaks were identified as heptasiloxane [Figure H12 & Figure H13]. The Durex sub-brands displayed unique chromatograms from one another, but some were similar. The Durex Warming Pleasure [Figure H16] exhibited an *n*-hexadecanoic acid peak at 10.721 minutes [Figure H17], an octadecanoic acid peak at 11.760 minutes [Figure H18], and no other significant peaks. The Durex Tingling Pleasure [Figure H19] only had peaks identified as heptasiloxane [Figure H20 through Figure H22], but the pattern of the TIC was not the same as that for the Trojan brand. Many of the peaks were equidistant from one another, but there was no bell-shaped curve. The Durex Natural Feeling [Figure H23] had a similar TIC to the Durex Warming Pleasure, with an *n*-hexadecanoic acid peak at 10.719 minutes [Figure H24], an octadecanoic acid peak at 11.758 minutes [Figure H25], but it also had a small peak at 15.500 minutes, identified as silane [Figure H26]. The Durex Her Sensation [Figure H27] displayed a

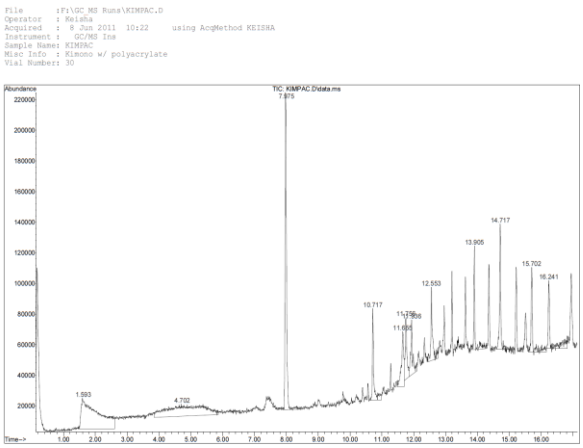
peak at 10.718, identified as undecanoic acid [Figure H28], a peak at 11.655, identified as octadecyne [Figure H29], a peak at 11.991, identified as butenol [Figure H30], and several peaks identified as nonamethyltetrasiloxane [Figure H31 & H32]. However, the low probabilities of the identifications of these compounds make it uncertain that these are the actual components present in the Durex Her Sensation condom residue. The Contempo Luscious Flavors sub-brands displayed chromatograms similar to one another. The Contempo Vanilla [Figure H33], Strawberry [Figure H37], and Banana [Figure H41] brands each had *n*-hexadecanoic peaks around 10.72 minutes. The Vanilla brand had an octadecanoic acid peak at 11.758 minutes [Figure H35]. Both the Strawberry and Banana brands had peaks near this retention time, identified as dimethoxybicyclononadione [Figure H39 & H43] but low levels of confidence were indicated in the library matches. The remaining peaks for the Strawberry and Banana brands were identified as heptasiloxane [Figure H40 & H44]. For the Vanilla brand, all the remaining peaks were identified as heptamethyltrisiloxane [Figure H36] but had significantly lower probabilities of being correct identifications than the heptasiloxane peaks in the chromatograms of the Strawberry and Banana brands. It appears the differences in the compound identifications might be due to variances in the abundance of the peaks. The similar appearance of the chromatograms and retention times of the peaks for the three flavored brands suggest that distinguishing them, using the described technique, would be difficult to achieve. These results are consistent with literature reports of the inability to distinguish brands that only have differences in the flavorings that were added (Keil, 2007). The negative control, using the polyacrylate fiber, produced only one insignificant peak, identified as 3-(2,2-dimethyl-propyl)-3-methyl-2,2-diphenyl-oxetane, at 11.982 minutes [Figure H45 & Figure H46]. The retention time of the peak does not correspond with any peaks of interest to the present analysis and does not

impact the level of confidence for the assessment of results. The TIC's for each of the condom brands are also illustrated in Figure 20.

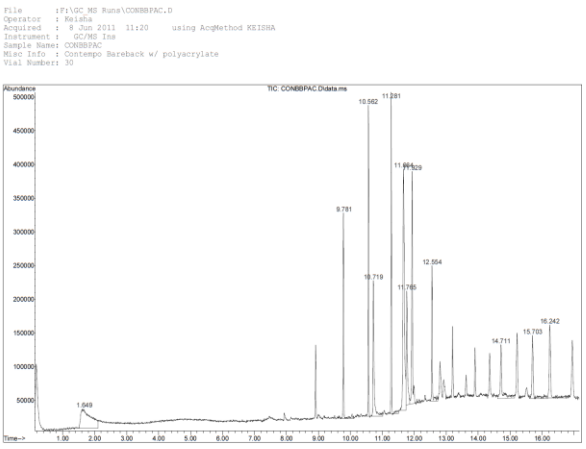
Butylated hydroxytoluene was previously determined by Keil (2007) to be an antioxidant added by manufacturers to kill bacteria. A computer search found that *n*-hexadecanoic acid (palmitic acid), octadecadienoic acid (linoleic acid), and octadecanoic acid (oleic acid) are all compounds used for the curing of rubber during the vulcanization process (Datta & Talma, 2002; Mowdood & Bharat, 1990). The scientific literature has stated that the major component in most condom residues is polydimethylsiloxane (Keil, 2007). However, this high molecular weight compound, which contains a mixture of oligomers of up to 20,000 amu, is non-volatile and unable to pass through the GC column (Campbell & Gordon, 2007). The peaks identified as heptasiloxane are most likely silicone-oils used in the washing step of condom production (R. Blackledge, personal communication, April 21, 2011).




(a)




(b)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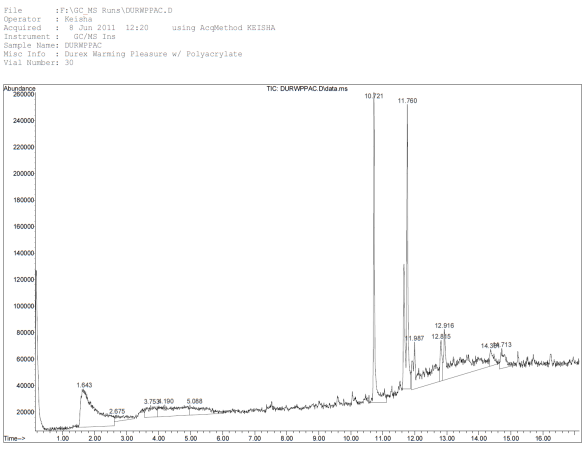
Operator : Keisha
Acquired : 8 Jun 2011 11:20 using AcqMethod KEISHA
Instrument : GC/MS Ins
Sample Name: CONBBPAC
Misc Info : Contempo Bareback w/ polyacrylate
Vial Number: 30
TIC: CONBBPAC.D\data.ms
Abundance
Time-->

(c)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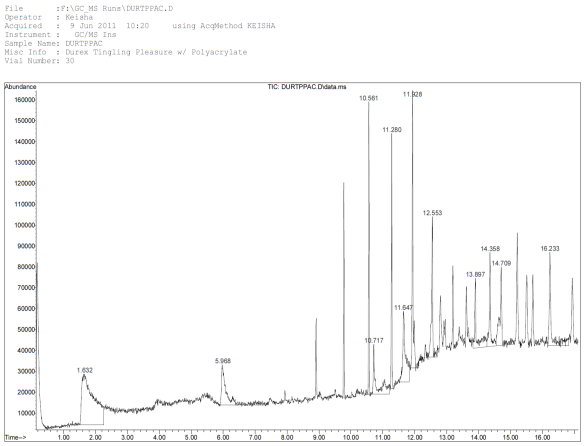
Operator : Keisha
Acquired : 8 Jun 2011 12:20 using AcqMethod KEISHA
Instrument : GC/MS Ins
Sample Name: DURWPPAC
Misc Info : Durex Warming Pleasure w/ Polyacrylate
Vial Number: 30
TIC: DURWPPAC.D\data.ms
Abundance
Time-->

(d)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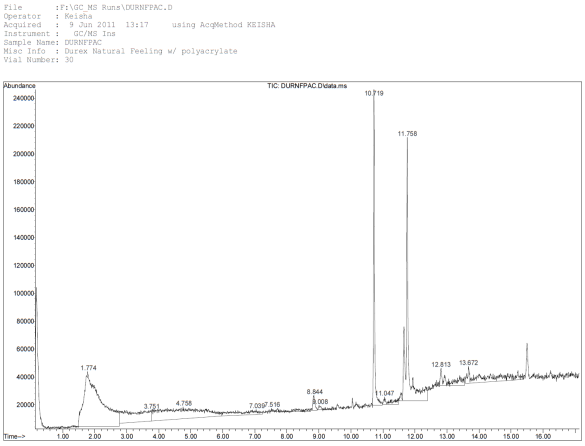
Acquired : 9 Jun 2011 10:20 using AcqMethod KEISHA
Instrument : GC/MS Ins
Sample Name: DURTPPAC
Misc Info : Durex Tingling Pleasure w/ Polyacrylate
Vial Number: 30
TIC: DURTPPAC.D\data.ms
Abundance
Time-->

(e)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Instrument : GC/MS Ins
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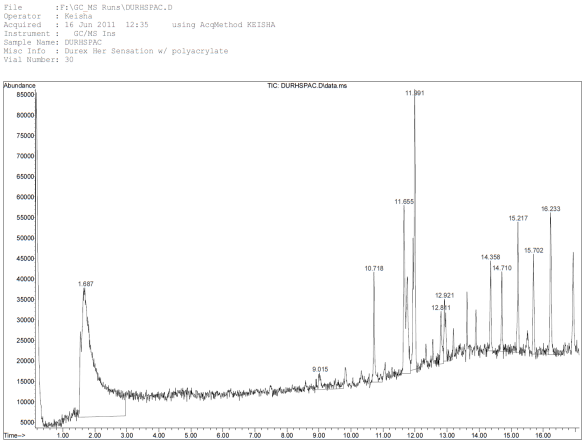
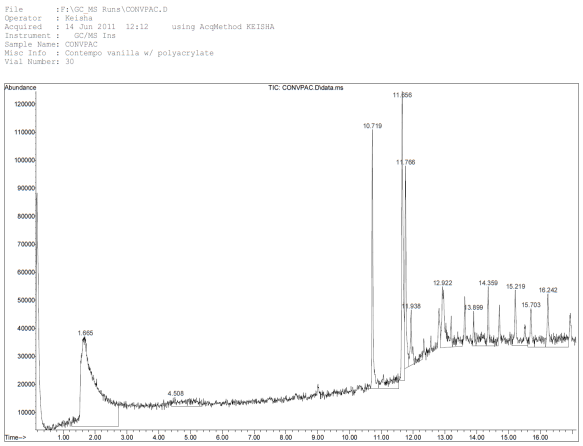
Misc Info : Durex Natural Feeling w/ polyacrylate
Vial Number: 30
TIC: DURNFPAC.D\data.ms
Abundance
Time-->

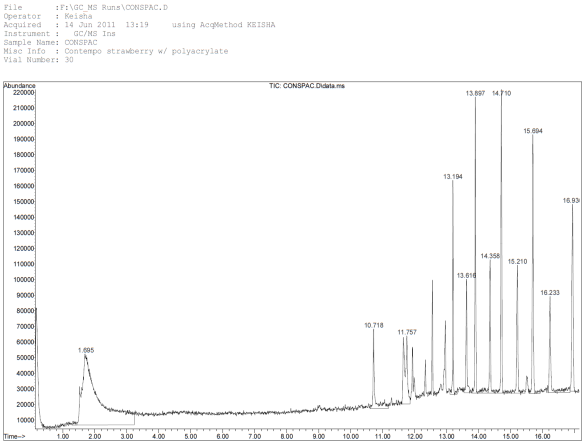
(f)



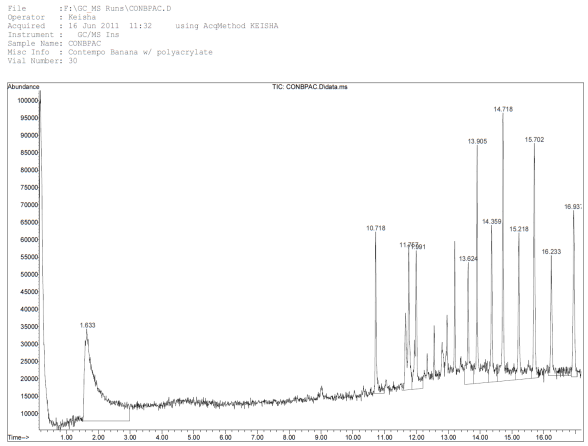

(g)




(h)




(i)

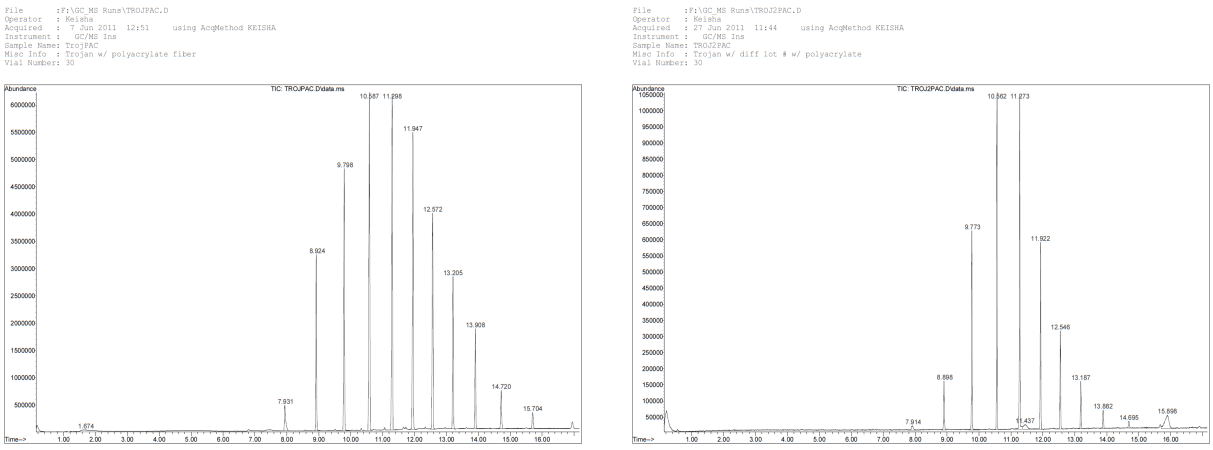



(j)

Figure 20. Total Ion Chromatograms with PAC Fiber (a) Trojan (b) Kimono (c) Contempo Bareback (d) Durex Warming Pleasure (e) Durex Tingling Pleasure (f) Durex Natural Feeling (g) Durex Her Sensation (h) Contempo Vanilla (i) Contempo Strawberry (j) Contempo Banana.

Although the intensities for some of the peaks in the chromatograms produced by the second lots of the Trojan [Figure H47], Kimono [Figure H48], and Contempo Bareback [Figure

H49] brands differed from those of the first lots [Figure H1, Figure H5, & Figure H11, respectively], the retention times of the major peaks were consistent between lots [Table 4 through Table 9]. This indicates a variation in the lot did not change any of the components that could be detected in the residues. However, the question still remains as to whether this would still be the case in the future, when the sources of components might be modified by manufacturers. The TIC's for the lot comparisons of these condom brands are illustrated in Figure 21 through Figure 23.




(a) (b)

Figure 21. Lot Comparison of Trojan Total Ion Chromatograms with PAC Fiber (a) Lot #DA9299GL3 (b) Lot # DA0127GL6.

Table 4:

*Retention Times- Trojan Lot #DA9299GL3*

Signal : TIC: TROJPAC.D\data.ms

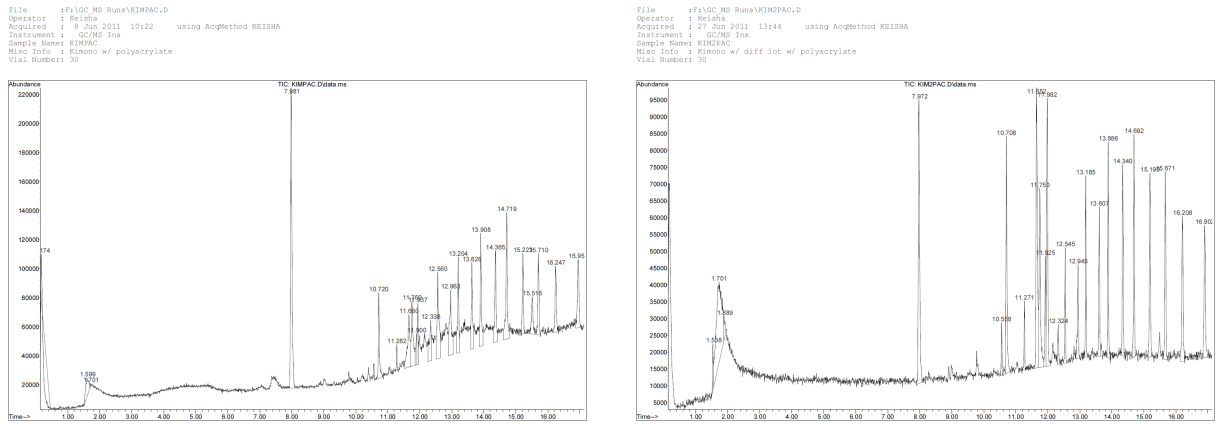
| peak # | R.T. min | first scan | max scan | last scan | PK TY | peak height |
|---|---|---|---|---|---|---|
| 1 | 7.939 | 988 | 999 | 1017 | BB | 474060 |
| 2 | 8.924 | 1113 | 1125 | 1134 | BB | 3222623 |
| 3 | 9.798 | 1224 | 1237 | 1247 | BB | 4648967 |
| 4 | 10.587 | 1326 | 1338 | 1348 | BB 2 | 6060490 |
| 5 | 11.306 | 1416 | 1430 | 1438 | BB | 6083263 |
| 6 | 11.955 | 1497 | 1513 | 1523 | BV | 5433380 |
| 7 | 12.572 | 1573 | 1592 | 1603 | BB | 3883639 |
| 8 | 13.212 | 1663 | 1674 | 1683 | BV | 2828460 |
| 9 | 13.908 | 1752 | 1763 | 1772 | BB | 1759890 |
| 10 | 14.720 | 1854 | 1867 | 1876 | BV | 697968 |
| 11 | 15.712 | 1976 | 1994 | 2004 | BB | 291993 |

Table 5:

*Retention Times – Trojan Lot # DA0127GL6*

Signal : TIC: TROJ2PAC.D\data.ms

| peak # | R.T. min | first scan | max scan | last scan | PK TY | peak height |
|---|---|---|---|---|---|---|
| 1 | 8.906 | 1113 | 1123 | 1133 | BB | 151899 |
| 2 | 9.781 | 1227 | 1235 | 1242 | BB | 579358 |
| 3 | 10.562 | 1327 | 1335 | 1341 | BB | 995691 |
| 4 | 11.273 | 1405 | 1426 | 1435 | BB | 974100 |
| 5 | 11.929 | 1501 | 1510 | 1515 | BV | 583343 |
| 6 | 12.546 | 1573 | 1589 | 1595 | BB | 292539 |
| 7 | 13.187 | 1661 | 1671 | 1680 | BB | 137377 |




(a) (b)

Figure 22. Lot Comparison of Kimono Total Ion Chromatograms with PAC Fiber (a) Lot #90656-9 (b) Lot # 00957-9.

Table 6:

*Retention Times – Kimono Lot #90656-9*

Signal : TIC: KIMPAC.D\data.ms

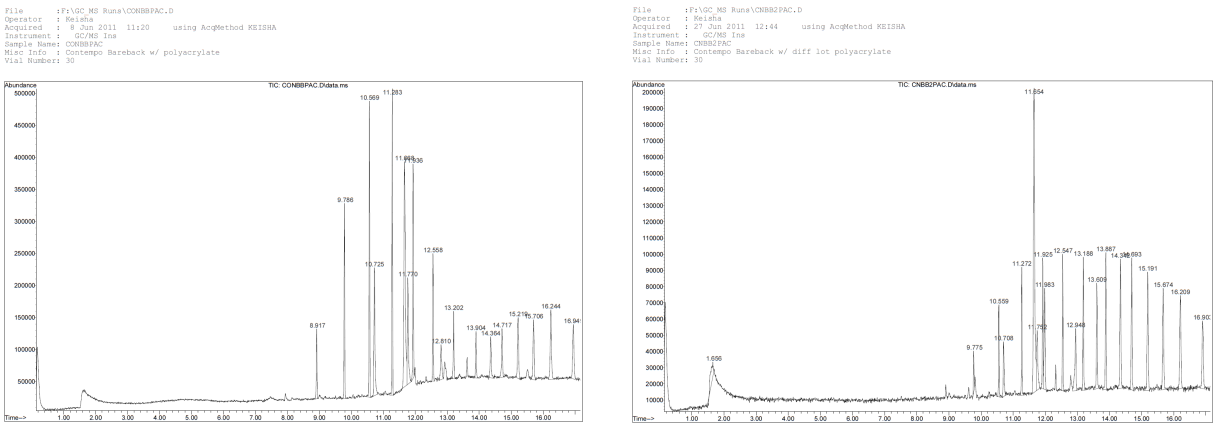
| peak # | R.T. min | first scan | max scan | last scan | PK TY | peak height |
|---|---|---|---|---|---|---|
| 1 | 0.171 | 3 | 5 | 41 | BV 2 | 31844 |
| 2 | 1.601 | 175 | 188 | 199 | PV 7 | 13044 |
| 3 | 1.702 | 199 | 201 | 202 | VV 2 | 2581 |
| 4 | 7.983 | 998 | 1005 | 1022 | PV | 205432 |
| 5 | 10.717 | 1347 | 1355 | 1370 | PV 3 | 57445 |
| 6 | 11.280 | 1419 | 1427 | 1431 | BV | 17363 |
| 7 | 11.663 | 1457 | 1476 | 1482 | VV 6 | 36080 |
| 8 | 11.756 | 1482 | 1488 | 1501 | VV 3 | 44843 |
| 9 | 11.897 | 1501 | 1506 | 1508 | VV 6 | 21155 |
| 10 | 11.936 | 1508 | 1511 | 1515 | VV | 40706 |
| 11 | 12.335 | 1551 | 1562 | 1567 | VV 2 | 27052 |
| 12 | 12.561 | 1584 | 1591 | 1602 | VV | 58518 |
| 13 | 12.960 | 1633 | 1642 | 1652 | VV 2 | 43220 |
| 14 | 13.202 | 1667 | 1673 | 1679 | VV | 64058 |
| 15 | 13.631 | 1723 | 1728 | 1735 | VV | 58347 |
| 16 | 13.905 | 1757 | 1763 | 1773 | VV | 75226 |
| 17 | 14.366 | 1814 | 1822 | 1829 | VV | 63744 |
| 18 | 14.717 | 1852 | 1867 | 1876 | VV | 85867 |
| 19 | 15.225 | 1925 | 1932 | 1937 | PV | 55391 |
| 20 | 15.514 | 1959 | 1969 | 1981 | VV 4 | 25330 |
| 21 | 15.709 | 1986 | 1994 | 2002 | VV | 56707 |
| 22 | 16.248 | 2051 | 2063 | 2071 | VV | 44852 |
| 23 | 16.951 | 2143 | 2153 | 2162 | VV 3 | 47118 |

Table 7:

*Retention Times – Kimono Lot #00957-9*

Signal : TIC: KIM2PAC.D\data.ms

| peak # | R.T. min | first scan | max scan | last scan | PK TY | peak height |
|---|---|---|---|---|---|---|
| 1 | 1.542 | 171 | 180 | 183 | PV 3 | 12884 |
| 2 | 1.698 | 183 | 200 | 223 | VV 3 | 24057 |
| 3 | 1.893 | 223 | 225 | 232 | VV 7 | 6494 |
| 4 | 7.971 | 994 | 1003 | 1017 | PV 2 | 83361 |
| 5 | 10.557 | 1329 | 1334 | 1340 | VV | 15053 |
| 6 | 10.705 | 1348 | 1353 | 1368 | VV | 68168 |
| 7 | 11.268 | 1418 | 1425 | 1431 | PV | 19469 |
| 8 | 11.650 | 1456 | 1474 | 1482 | VV 2 | 81972 |
| 9 | 11.752 | 1482 | 1487 | 1503 | VV 2 | 53217 |
| 10 | 11.924 | 1503 | 1509 | 1512 | VV | 32164 |
| 11 | 11.986 | 1512 | 1517 | 1529 | VV | 78186 |
| 12 | 12.322 | 1556 | 1560 | 1565 | VV 2 | 11465 |
| 13 | 12.541 | 1581 | 1588 | 1596 | VV | 32784 |
| 14 | 12.947 | 1632 | 1640 | 1645 | VV 2 | 27637 |
| 15 | 13.182 | 1661 | 1670 | 1677 | VV | 50712 |
| 16 | 13.603 | 1718 | 1724 | 1730 | VV | 42962 |
| 17 | 13.885 | 1753 | 1760 | 1771 | VV | 62320 |
| 18 | 14.338 | 1807 | 1818 | 1828 | BV | 55736 |
| 19 | 14.689 | 1856 | 1863 | 1870 | PV | 64132 |
| 20 | 15.189 | 1919 | 1927 | 1934 | PV | 53817 |
| 21 | 15.674 | 1981 | 1989 | 1996 | VV | 54642 |
| 22 | 16.205 | 2048 | 2057 | 2066 | VV | 42270 |
| 23 | 16.900 | 2136 | 2146 | 2158 | VV 3 | 38990 |




(a) (b)

Figure 23. Lot Comparison of Contempo Bareback Total Ion Chromatograms with PAC Fiber (a) Lot #0912042916 (b) Lot #1006092516.

Table 8:

*Retention Times – Contempo Bareback Lot #0912042916*

Signal : TIC: CONBBPAC.D\data.ms

| peak # | R.T. min | first scan | max scan | last scan | PK TY | peak height |
|---|---|---|---|---|---|---|
| 1 | 8.914 | 1107 | 1124 | 1131 | PV | 104246 |
| 2 | 9.789 | 1223 | 1236 | 1243 | PB | 290381 |
| 3 | 10.570 | 1320 | 1336 | 1341 | BV | 463418 |
| 4 | 10.726 | 1341 | 1356 | 1369 | PV | 199491 |
| 5 | 11.281 | 1416 | 1427 | 1432 | VV | 449699 |
| 6 | 11.672 | 1432 | 1477 | 1485 | PV 2 | 347627 |
| 7 | 11.773 | 1485 | 1490 | 1504 | VV 2 | 162370 |
| 8 | 11.937 | 1504 | 1511 | 1515 | PV | 331209 |
| 9 | 12.554 | 1579 | 1590 | 1596 | BV | 189586 |
| 10 | 12.812 | 1615 | 1623 | 1632 | PV 3 | 53774 |
| 11 | 13.203 | 1666 | 1673 | 1685 | BV | 105930 |
| 12 | 13.906 | 1758 | 1763 | 1775 | VB | 71552 |
| 13 | 14.367 | 1810 | 1822 | 1832 | VV | 62672 |
| 14 | 14.718 | 1854 | 1867 | 1876 | BV | 76132 |
| 15 | 15.218 | 1921 | 1931 | 1940 | BV | 94633 |
| 16 | 15.703 | 1982 | 1993 | 2002 | PV 2 | 89904 |
| 17 | 16.242 | 2053 | 2062 | 2076 | VV | 107506 |
| 18 | 16.945 | 2144 | 2152 | 2166 | BV | 83399 |

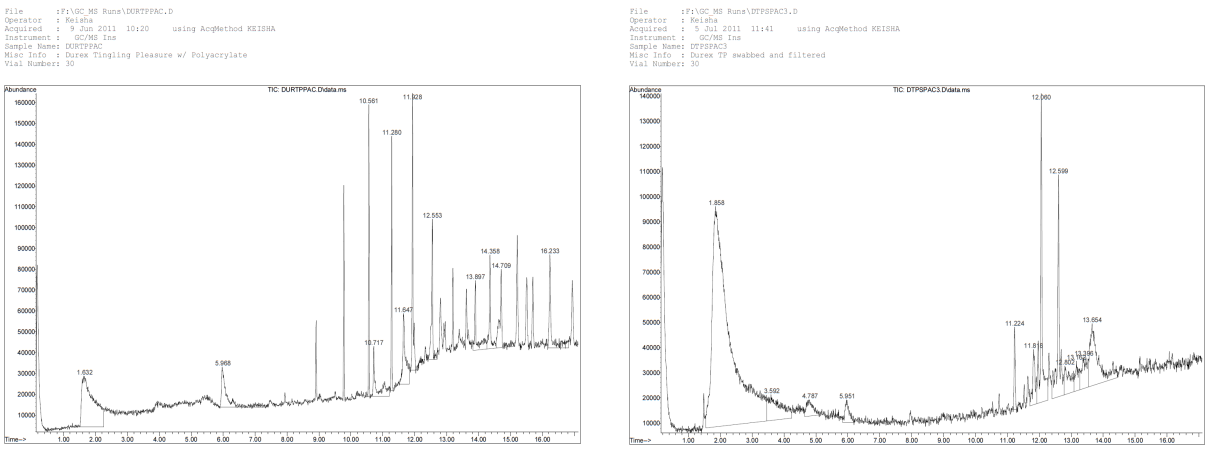
Table 9:

*Retention Times – Contempo Bareback Lot #1006092516*

Signal : TIC: CNBB2PAC.D\data.ms

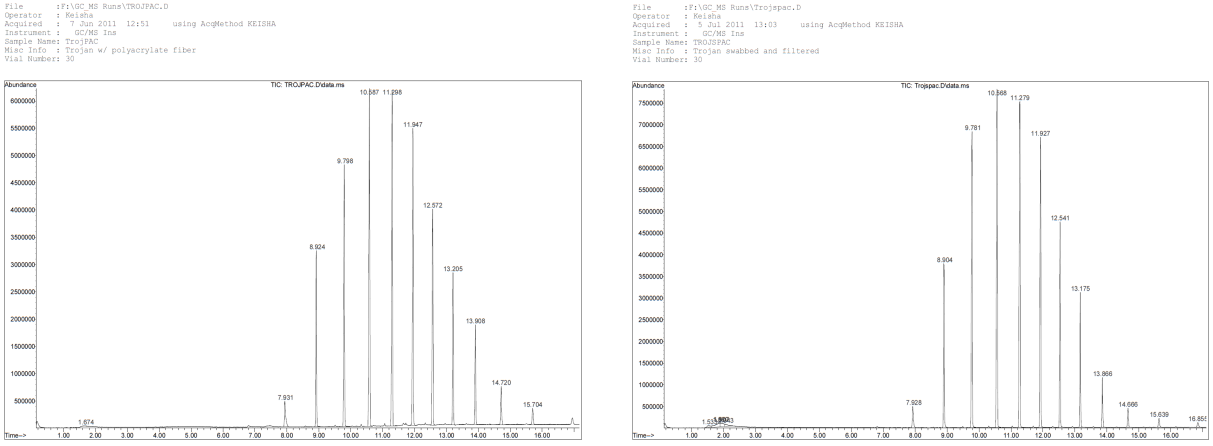
| peak # | R.T. min | first scan | max scan | last scan | PK TY | peak height |
|---|---|---|---|---|---|---|
| 1 | 1.653 | 171 | 194 | 198 | BV 8 | 7733 |
| 2 | 9.778 | 1228 | 1234 | 1237 | PV | 23455 |
| 3 | 10.559 | 1328 | 1334 | 1348 | BV | 56810 |
| 4 | 10.707 | 1348 | 1353 | 1363 | PV 2 | 33321 |
| 5 | 11.270 | 1407 | 1425 | 1434 | VV | 75458 |
| 6 | 11.653 | 1463 | 1474 | 1483 | BV 3 | 186670 |
| 7 | 11.754 | 1483 | 1487 | 1499 | VB 3 | 36397 |
| 8 | 11.926 | 1499 | 1509 | 1512 | BV | 81045 |
| 9 | 11.981 | 1512 | 1516 | 1533 | VB 2 | 60721 |
| 10 | 12.543 | 1580 | 1588 | 1595 | BB | 80025 |
| 11 | 12.949 | 1628 | 1640 | 1645 | VV 2 | 39197 |
| 12 | 13.192 | 1662 | 1671 | 1677 | VV | 77786 |
| 13 | 13.606 | 1711 | 1724 | 1732 | PV | 62939 |
| 14 | 13.887 | 1748 | 1760 | 1776 | PV | 82105 |
| 15 | 14.340 | 1808 | 1818 | 1829 | PV | 78367 |
| 16 | 14.692 | 1857 | 1863 | 1872 | PV | 79118 |
| 17 | 15.192 | 1912 | 1927 | 1934 | VV | 72123 |
| 18 | 15.676 | 1968 | 1989 | 2003 | VB | 61779 |
| 19 | 16.207 | 2041 | 2057 | 2065 | PV 2 | 56650 |
| 20 | 16.902 | 2136 | 2146 | 2160 | PV 2 | 41278 |

The Durex Tingling Pleasure condom that was swabbed, extracted, and filtered produced a chromatogram [Figure H52] that had some of the same peaks as the rinsed and extracted Durex Tingling Pleasure condom [Figure H19]. However, some of the peaks observed in the rinsed sample were not visible in the swabbed sample. The results of the Trojan swabbed sample were a chromatogram [Figure H53] displaying all the peaks present in the chromatogram of the Trojan rinsed sample [Figure H1]. This indicates that it is possible to obtain chromatograms from swabbed samples that can be matched to reference samples, but this procedure should be further developed to recover condom residue components which are in lower abundance. The comparisons of these TIC's are illustrated in Figure 24 and Figure 25.




(a) (b)

Figure 24. Comparison of Durex Tingling Pleasure Total Ion Chromatograms with PAC Fiber (a) Rinsed (b) Swabbed.




(a) (b)

Figure 25. Comparison of Trojan Total Ion Chromatograms with PAC Fiber (a) Rinsed (b) Swabbed.

**Isotope-ratio mass spectrometry.** The results of the IRMS analysis are listed in Table 10, and they reveal that the Contempo Luscious Flavors and Trojan brands have $\delta^{13}C$ values that are different from the other brands (Philp, personal communication, June 10, 2011). As mentioned before, based on the scientific literature, the primary component of condom residues is the lubricant. It can then be assumed that the bulk ratio of the carbon isotopes is mostly attributed to the PDMS, whose methyl groups originate from petroleum bodies. The results would then indicate that the PDMS came from three different sources of stable carbon isotopes. The two brands that are made in Thailand, Contempo Bareback and Durex Play Sensations, and the Kimono Select, which are made in Japan, have similar ratios. Geographically, Thailand and Japan are close to one another and could perhaps have the same petroleum body, so it is not surprising that these three brands have similar ratios. The Contempo Luscious Flavors, which is made in India, and the Trojan Thintensity brand, which is made in the United States, have distinct ratios from the other brands. Another interesting observation is that the two Contempo Bareback and Contempo Lucious Flavors brands, made by the same manufacturer but in two separate countries, have significantly different carbon isotopic values. This is indicative of the company obtaining PDMS from separate sources. The three brands that were analyzed using different lot numbers showed some variance in their $\delta^{13}C$ ratios. This could support the use of IRMS to distinguish lots of the same brand that have the inability to be differentiated by GC/MS alone. More runs would also need to be performed on specimens from a single lot to determine what the normal variance is between the $\delta^{13}C$ ratios of samples from the same carbon isotope source. The correspondence and results reported by the OU Geology Department can be found in *Appendix C*.

Table 10

*Isotopic Ratios*

| Brand | Manufacturing Location | $\delta^{13}C$ (‰) – Run 1 | $\delta^{13}C$ (‰) – Run 2 |
|---|---|---|---|
| Contempo Bareback | Thailand | –46.6 | –47.3 |
| Kimono Select | Japan | –46.8 | –45.3 |
| Trojan Thintensity | United States | –41.5 | –43.5 |
| Durex Play Sensations | Thailand | –47.9 | -------- |
| Contempo Luscious Flavors | India | –37.1 | -------- |

Note. The precision of the $\delta^{13}C$ values is +/- 0.1 ‰, and values with a difference of 1 ‰ or more are considered different (Philp, personal communication, August 11, 2011).

## Future Research

It is anticipated that peaks from the chromatograms produced by the GC/MS method, using the PAC SPME fiber, will be systematically entered into a database for each condom product. This will likely be created by the OSBI by cataloguing the information into the Spectral Library Identification and Classification Explorer (SLICE) program. This library will then need to be tested by entering an unknown sample to determine if it will match the correct condom brand with a high probability. The technique for analyzing swabbed samples also needs to be further developed. It appears samples with higher concentrations of residues can be correctly matched to the condom brand by chromatogram comparison, but for those samples with perhaps a limited abundance of residue that were explored in this exercise, not all of the components are exhibited on the TIC's. It is therefore more difficult to correctly identify a condom brand through chromatogram pattern matching alone. This could be resolved if the extraction process of specimen swabbings were improved. This should therefore be a future area of research.

This study did not attempt to use simulated case samples for any analysis. In an academic study, the use of volunteer couples to provide vaginal swabs post-coitus is not easily undertaken, and this subject has been extensively explored in the publications described herein. However, to prove the accuracy of the described technique for matching unknown condom brands to the correct brand stored in the database, the use of simulated case samples would need to be performed. Biological materials can have an impact on the extraction process and therefore the resulting chromatograms. This means that the procedure developed herein may need to be further altered to accommodate for this dimension.

Although there were 79 condom companies registered with the FDA in 2010, at the time the brands were chosen, only a representative subset of products were explored in this research. The brands were selected only by the geographical location of the manufacturer, rather than by consumer popularity. In the event that an actual condom database is formed, a much larger set of brands will need to be analyzed. At a minimum, all the brands marketed in the United States should be analyzed and entered into the library. An important point to note is that new products are introduced to the market annually. Also, the ingredients that a manufacturer uses can be expected to change over time. This suggests that a database supporting crime labs will need to be updated on continual basis.

Several attempts were made to contact Church and Dwight Co., Inc., the manufacturer of Trojan condoms, via phone, email, and the postal service. Since the research and development department of the company is located in New Jersey in the United States, it seemed practical to make a visit to their plant, and monies were allocated in the faculty grant supporting this research. The thought was that obtaining samples of the materials used in the production of condoms could greatly aide this research endeavor. However, Church and Dwight Co., Inc.

declined such a proposition, even after receiving a letter from the Director of the OSBI laboratory in support of such an exploratory visit. This is documented in *Appendix D*. Visits to condom manufacturers should be made in the future to obtain samples of components that are used in each step of production, so as to better understand and source an import of peaks on the chromatograms.

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# Appendix A

## Correspondence with Robert Blackledge

---

**Date:** Thursday, April 8, 2010 2:54 PM

**From:** Blackledge <[redacted]>

**To:** Keisha Jones <[redacted]>

**Subject:** Re: Thesis

---

Hi Keisha,

First, there already is an article in the Journal of Forensic Sciences on using PyGCMS to detect/characterize condom lubricants. The reference is: JFS Vol. 52 (2007), No. 3, pages 630-642, "Analysis of condom lubricants for forensic casework", Gareth P. Campbell and Amanda L. Gordon.

There are many aspects of this paper with which I am not in agreement, but I don't think it would be worthwhile to try to improve on their work. There is a different general approach to this question and that is to try to come up with an overall "signature" for the various components (lubricant, particulates, antioxidants, mold-release agents, spermicides, etc.) found in each brand. In Europe, Dr. Wolfgang Keil has had initial success with this approach, but there is much more that could be done.

I suggest that through the library at the University of Central Oklahoma you check out a copy of the book, FORENSIC ANALYSIS ON THE CUTTING EDGE: New Methods for Trace Evidence Analysis, Robert D. Blackledge, Editor, 2007, Wiley Interscience, ISBN 978-0-471-71644-0. Dr. Keil wrote Chapter 4, "Condom Trace Evidence in Sexual Assaults: Recovery and Characterization." Any analysis method that researchers come up with is of limited value if most crime labs don't have access to the instrumentation necessary. Today, just about every crime lab has: 1) a polarized light microscope (PLM); 2) an FTIR; 3) a GC/MS; 4) a computer with spread sheet software that could be used for creating a searchable database.

I'll be out of town from tomorrow morning until late the following Friday, but after that I'd be happy to talk with you about your thesis project.

Best regards,

Robert ("Bob") D. Blackledge
Forensic Chemist Consultant
[redacted]

Home phone: [redacted]
Home email: [redacted]

**Date:** Thursday, April 8, 2010 8:37 PM

**From:** Blackledge <[redacted]>

**To:** Keisha Jones <[redacted]>

**Subject:** Re: Thesis

Keisha,

It sounds like you have some good ideas and are off to a good start. The FSD
is part of the software that comes when you purchase the FTIR. It may vary slightly depending upon brand (Thermo Nicolet, Perkin Elmer, Hitachi, etc.) but they all do essentially the same thing. As far as fluorescence I'd be a bit surprised if it helps when you are just examining vaginal/anal cotton swabs. However, when examining things like undergarments, towels, and bedding it can be very useful in determining the best locations to take your
samples. What you need to keep in mind is that in all likelihood the condom lubricant components will not fluoresce. However, because of residues from optical brighteners present in detergents ("make your clothes whiter than white") the fabric items you examine for condom lubricant stains will fluoresce. Any stains from lubricants on these fabrics will partly tend to block any fluorescence. So rather than fluorescing (spots appearing brighter
and a certain color) the places where there are lubricant stains will have a
shadowy or darker appearance. This is not a useful property as far as distinguishing between different brands, but it does give you valuable information as far as the best places to take samples.

The most common condom lubricant, trimethoxy-terminated polydimethylsiloxane
(PDMS), has much too high a molecular weight range to first, go through a GC
column, and second, be characterized by the type of mass spectrometers typically found in crime labs that are primarily used for drug identification. However, after a latex condom is first formed many manufacturers rinse the condoms with a solution that may include a low molecular weight cyclic form of PDMS. Also present may be other low molecular weight molecules such as antioxidants (leave a rubber band on the dash board of your car a few days and notice how quickly it degrades from exposure to oxygen and UV rays from the sun). The additives used by each manufacturer are proprietary information, but by injecting a concentrated extract (say methanol for polar components and/or pentane for nonpolar components) into a GC/MS you may obtain 1) a total ion chromatogram (TIC) that is characteristic for that manufacturer, and 2) by putting your cursor on each peak in the TIC in turn, you can obtain the fragmentation pattern for each peak and then for each fragmentation pattern for each peak you can do a library search and possibly identify the various peaks in the TIC. With
the relative peak intensities of the various components and the fragmentation patterns for each peak you would have the beginning of

entries
you could make into a searchable database. In the Dr. Keil's chapter in my book he illustrates this.

I look forward to talking to you further about this. I'm a tour speaker for the American Chemical Society. On a tour last year I spoke at an ACS local section meeting in Lawton, Oklahoma. To this email I'll attach brief abstracts for a couple of my more popular talks. You might check with the Chemistry Dept. at the University of Central Oklahoma. Do they have invited speakers for department seminars? If they could just pay my travel expenses I'd be happy to be a visiting speaker for a seminar. This would also give us
a chance to meet and further discuss your thesis research. I also have a more general presentation on "Trace Evidence in Sexual Assaults" that I gave
last year before the Chem. Dept. at the Univ. of Texas at Arlington.

Best regards,

Bob Blackledge

---

**Date:** Wednesday, April 20, 2011 10:43 PM

**From:** Blackledge <[redacted]>

**To:** Keisha Jones <[redacted]>

**Subject:** Re: Thesis Research

---

Hi Keisha,

Sorry I didn't get back to you sooner, but the past two weeks I've been on speaking tours for the American Chemical Society. I'm sorry to hear your research is not going well. Dr. Wolfgang Keil used specific condom brands available in Germany. The results he obtained varied with the brand. If you are not using the same brands you will of course get different results although hopefully the results you get will be characteristic of the brands you examined. If you obtained and examined the same brands that Keil tested then perhaps in the intervening years the manufacturers have made production
changes.

Dr. Keil's e-mail address is: [redacted]

Best regards,

Bob Blackledge

---

**Date:** Thursday, April 21, 2011 5:53 PM

**From:** Blackledge <[redacted]>

**To:** Keisha Jones <[redacted]>

**Subject:** Re: Thesis Research

---

Keisha,

I retired in May 2006, so I no longer have access to a lab. I have never actually tried Dr. Keil's extraction/derivitization method. In the latex condom manufacturing process the newly-formed condoms on mandrels are dipped in a aqueous solution. This solution may contain a variety of ingredients. Some may be low molecular weight (therefore water soluble/miscible?) cyclic silicones or low molecular weight straight chain silicones that may have polar end groups (hydroxyl, etc.). There may be low molecular weight surfactants (have a non-polar branch and a polar branch), mold release agents (calcium carbonate), corn starch grains, and antioxidants (prevents or delays the oxidation/degradation of the latex). An antioxidant that I and others have seen in case work is CAS # 119-47-1 (it has various chemical
names). There are other phenolic antioxidants in use and they can generally be detected in both hexane and methanol extracts. In actual case work in the past I have found that a simple methanol extraction would recover the more polar ingredients and they could be identified simply by concentrating the methanol extract by partial evaporation and then injecting some of the concentrate onto a GC/MS. For example, that works with BHT (butylated hydroxytoluene) used as an antioxidant by some condom manufacturers. The silicones used as a lubricant have a much higher molecular weight that is well beyond the range of a GC/MS, so if you see silicones via GC/MS they are from the dipping solution rather than from the PDMS lubricant. Of course, for those latex condom brands that have a water-soluble lubricant (glycerol, PEG 300 or 400, etc.) you should see them with GC/MS.

The things that may be used in the manufacturing process of latex condoms are extremely varied. Following is a website that might be useful in providing possible ingredients to consider:

I:\condoms\(WO-2006-049627) LUBRICATED CONDOM.mht

In recent years there have been many changes in the latex condom industry with corporations being bought or merging with other corporations. In the past the corporation that made the Trojan brand was the most difficult for me to work with. They would never permit me direct contact with their chemists. I had to be working on a criminal case (not research) and I had to pass any questions I had to their lawyers who would then contact their chemists and then relay the information (stripped of anything proprietary) back to me. However, I believe they are one of the corporations that have been

sold/merged so perhaps they are now more cooperative. Ansell Inc. in Dothan, Alabama (LifeStyles condoms) has always been extremely helpful. Although he may have retired by now, my contact with Ansell was Lon McIlvain ( [redacted] ). Another contact at Ansell is Cindy Ingram [redacted] Contactss at Church & Dwight Co., Inc. (Trojan brand) are: [redacted] and [redacted] Another expert worth contacting is Russell D. Culp. He testified in a trial in LA where I also testified, but because we testified on different days we never actually met. Using cut and paste, below is something about him:

Russell D. Culp, Industry Consultant, with more than 25 years of experience in latex compound development, latex dipping technologies and research and development, helps Vystar's manufacturing clients integrate the proprietary Vytex™ natural rubber latex technology into their production lines. An active A.S.T.M. member, Mr. Culp served as the chairman of the A.S.T.M. D11.40 Subcommittee, which has responsibility for writing and reviewing standards for consumer rubber products (gloves, condoms, finger cots, etc.), for four years (1986-1990). He has been a consultant and held key technical services positions with Alatech Healthcare, LLC; Ansell, Incorporated; Baxter Healthcare; LMR International; and London International Group / Aladan Corporation. Mr. Culp holds a bachelor degree in biology from Troy State University.

I don't have his contact information but if you contact Vytex:

http://vytex.com/OurCompany/managementteam.aspx?team=1&pageid=OC2 and then click on "Contact Us" you should be able to track him down.

I hope this helps,

Bob Blackledge

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**Date:** Friday, May 13, 2011 2:33 PM

**From:** Blackledge <[redacted]>

**To:** Keisha Jones <[redacted]>

**Subject:** MS library of latex additives

---

Keisha,

At the below website it tells about an MS library for latex additives. I thought that this library might be useful in trying to identify some of the peaks in your MS spectra.

http://www.frontier-lab.com/techinfo/technote/pdf/PYA1-057E.pdf

Best regards,

Bob Blackledge

**Date:** Friday, April 22, 2011 1:37 PM

**From:** Blackledge <[redacted]>

**To:** Keisha Jones <[redacted]>

**Subject:** Re: Thesis Research

Keisha,

Although I could often distinguish between specific brands, since it would be impossible to have examined all the different brands I would never claim that the residues I examined had to have originated from a certain brand to the exclusion of all others.

Bob Blackledge

---

**Date:** Monday, June 13, 2011 1:29 AM

**From:** Blackledge <[redacted]>

**To:** Keisha Jones <[redacted]>

**Subject:** Re: MS library of latex additives

---

Try doing a Google Advanced search and enter the terms "hexadecanoic acid", "palmitic acid", and "rubber."

Do the same thing for "octadecadienoic acid."

Also see:
http://www.docstoc.com/docs/40639747/Tall-Oil-Fatty-Acid-Mixture-In-Rubber---Pat
ent-4895911

# Appendix B

## Correspondence with Wolfgang Keil et al.

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**Date:** Tuesday, March 8, 2011 1:43 PM

**From:** Andrea Berzlanovich <[redacted]>

**To:** Keisha Jones <[redacted]>

**Subject:** Re: Condom Trace Evidence Thesis

---

Sehr geehrte/r Absender/in!

Diese E-Mail-Adresse ist abgelaufen:

E-Mail-Adresse : [redacted]
Abgelaufen seit : 16.06.2008

E-Mails an diese Adresse werden nicht mehr abgerufen.

Mit freundlichen Grüßen,
Helpdesk des Zentralen Informatikdienstes
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Diese Nachricht wurde automatisch durch das Out-of-Office-Programm der
Universität Wien generiert. Ihre E-Mail wird jedoch normal zugestellt.
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This is an automatic reply generated by the Out of Office-Program at the
University of Vienna. However, your e-mail will be delivered normally.
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**Date:** Wednesday, June 1, 2011 5:17 AM

**From:** Wolfgang Keil <[redacted]>

**To:** Keisha Jones <[redacted]>

**Subject:** Your questions of April 13th

Hi Keisha,

please excuse that I did not report. I really have very little time.

Currently I'm at a conference in Slovakia and can try until next week to compile the details again.

Best regards,
Wolfgang

Prof.Dr.med. Wolfgang Keil
[redacted]

Tel. privat +[redacted]
Tel. dienstl.+[redacted]
Handy +[redacted]

**Date:** Friday, June 10, 2011 5:51 AM
**From:** Oevgueer, Birgit <[redacted]>
**To:** Keisha Jones <[redacted]>
**Subject:** Your request Thesis Research

Hi Keisha,

Prof. Keil forwarded your mail to our lab.

We did the extractions some years ago and after talking to my colleague next week (she was responsible for GC-MS) we definitely can answer your questions.

Have a nice weekend,

Bye for now,
Birgit

**********
Birgit Övgüer
**Institut für Rechtsmedizin**
Abteilung Toxikologie

[redacted]

Tel.: [redacted]
e-mail: [redacted]
**********

**Date:** Tuesday, July 12, 2011 10:45 AM
**From:** Oevgueer, Birgit <[redacted]>
**To:** Keisha Jones <[redacted]>
**Cc:** Keil, Wolfgang <[redacted]>
**Subject:** Your request

Hi Keisha,

finally, the answers to your questions are coming. We hope they'll help you.
If you need more information please feel free to contact us again.

Greetings from Germany,
Birgit

**1. Trisbuffer:** Neutral buffer is chosen as rinse solution for unused condoms.

**2. Ammonium-Buffer pH 8, 9:** We expected basic substances and pH 8, 9 is the common buffer used in screening methods.

**3. Derivatization:** Polar substances wouldn't pass the GC column, therefore a derivatization is necessary.

**4. Evaporation to dryness:** A constant ratio of BSTFA and Ethylacetat is essential, normally Vol % 1:1 (50µl BSTFA + 50µl Ethylacetat). It is almost impossible to evaporate to a certain amount; therefore we choose dryness to guaranty equal conditions.

**5. Identification:** Extracts of established condom brands are injected; the chromatograms are recorded to a library as "Fingerprints". The differences are: Numbers, height ratios, retention times, and distances of peaks. There is no special spectra registration.

Chromatograms of vaginal swab extracts are compared with library.

**********

Birgit Övgüer
**Institut für Rechtsmedizin**
Abteilung Toxikologie

[redacted]

[redacted]

Tel.: [redacted]
e-mail: [redacted]
**********

# Appendix C

## Correspondence with Dr. Richard Philp, et al.

**Date:** Friday, June 10, 2011 5:09 PM

**From:** Philp, Richard P. <[redacted]>

**To:** Thomas Jourdan <[redacted]>, Keisha Jones <[redacted]>

**Cc:** Tamiko Fakuda <[redacted]>

**Subject:** KEISHA JONES' PDMS FORENSIC EXPLORATION

Ok some interesting results here that you might like to see but do not get to
excited yet!!!!!!

1. Durex -47.91
2. Contempo -37.12
3. Kimwo -46.84
4. Trojan -41.51
5. Contempo -46.63

So basically great that 2 and 4 are different BUT how much variation is there
batch to batch and that is what you need to think about next. Regards RPP

**Date:** Tuesday, July 5, 2011 2:02 PM

**From:** Warren, Anne <[redacted]>

**To:** Keisha Jones <[redacted]>

**Subject:** RE: Re: KEISHA JONES' PDMS FORENSIC EXPLORATION

Hey Keisha,

I received an answer from Paul. He said that as those are bulk numbers, no
chromatograms can be recorded. So no print outs.

He is looking into the "contempo #2 and #5" problem.

Dr. Anne Warren - Geologist / Geochemist - University of Oklahoma - Sarkeys
Energy Center

---

**Date:** Thursday, July 7, 2011 10:58 AM

**From:** Philp, Richard P. <[redacted]>

**To:** Philp, Richard P. <[redacted]>, Thomas Jourdan <[redacted]>, Keisha Jones <[redacted]>

**Cc:** Tamiko Fakudo <[redacted]>, Warren, Anne <[redacted]>

**Subject:** RE: KEISHA JONES' PDMS FORENSIC EXPLORATION

---

OK the mystery is solved! Sample 2 Is the Banana and sample 5 is the Bareback.
Hope this helps and again apologies for the confusion. Regards Paul.

---

**Date:** Friday, July 8, 2011 11:21 AM
**From:** Philp, Richard P. <[redacted]>
**To:** Keisha Jones <[redacted]>
**Subject:** FW: Carbon Isotopes

---

Quick turn around this time. Paul

**From:** Maynard, Rick J.
**Sent:** Friday, July 08, 2011 10:11 AM
**To:** Philp, Richard P.
**Subject:** Carbon Isotopes

Carbon Isotopes Relative to the VPDB scale : Date: 7/8/11

| SAMPLE | DEL C13 |
|---|---|
| #1 Trojan | -43.49 |
| #2 Contempo Bareback | -47.26 |
| #3 Kimbo | -45.27 |

**Date:** Tuesday, August 9, 2011 3:04 PM

**From:** Warren, Anne <[redacted]>

**To:** Keisha Jones <[redacted]>, Philp, Richard P. <[redacted]>

**Subject:** answer to questions

Hi Keisha,

I would like to apologize if I am making here a mistake, but curiously the
questions you emailed few minutes ago strangely resemble to questions that
Tamiko may have asked to Paul Philp. Here are his answers.

Dr. Anne Warren - Geologist / Geochemist - University of Oklahoma - Sarkeys
Energy Center

_____
From: Philp, Richard P.
Sent: Tuesday, August 09, 2011 1:56 PM
To: Tamiko Fukuda
Cc: Warren, Anne
Subject: RE: Vistors at Goodyear Tire and Rubber Plant: August 10th by the OSBI
FSC, UCO FSI & OU concerning forensic tire study (08-05-2011)

Tamiko Basically the range of isotope values for oils world wide is in the range
of -20 to -35 permil. However you cannot really pinpoint specific parts of the
world that have specific values since it depends on source materials,
depositional environments etc. I think the chromatograms showing the isotope
numbers of some of the individual compounds in the pyrolysates are the important
thing and I thik you have that information.

The precision of the numbers for the individual compounds is about +/- .3per
mil. Typically if you have two compounds and their values differ by at least 1
per mil then you start to feel pretty confident that the compounds are coming
from different sources.

The rations reflect the relative proportions of the 13C/12C and these values

will vary depending on the origin or source of the compound. Basically the best
result is when two samples are isotopically different –if two samples are the
same then they could be from the same source or it could be a co-incidence since
there is only a finite range if isotope values.

Also you don’t want to use the isotope values on their own you also need to look
at the GC traces and see if those fingerprints are the same or different. Hopefully if they are different the isotope values will support that observation.  It is just one tool in the box you can use.

Hpoe this helps-I will make a PDF file of a review chapter I wrote that might
help with the background information.

Let me know if you have more questions.

**Date:** Thursday, August 11, 2011 7:15 PM

**From:** Philp, Richard P. <[redacted]>

**To:** Keisha Jones <[redacted]>, Warren, Anne <[redacted]>

**Cc:** Thomas Jourdan <[redacted]>

**Subject:** RE: answer to questions

OK I think there is some confusion here. First the numbers we gave you are the
bulk numbers and yes they are in per mil. I do not recall anything being reported to 4 significant figures so cannot address that comment. For the bulk
isotope numbers the precision is +/- 0.1 per mil. The value of R for the standard is a very small number like 0.0112356 or something I do not have that
in front of me right now. However that is the ratio of C13/C12 in the international standard that is not the delta 13C value. The delta 13C value
which is what you were given is expressed as ((Rstandard-R sample)/Rstandard)x1000.

Anne is correct with this method if you see differences of 1 per mil or more you
can be confident this is a real difference. This could be lot to lot or brand to
brand depending on what you are looking at.

So hopefully this clarification will help you interpret your results a little
more readily.

Regards Paul Philp

**Date:** Friday, August 12, 2011 11:08 AM

**From:** Warren, Anne <[redacted]>

**To:** Keisha Jones <[redacted]>, Philp, Richard P. <[redacted]>

**Subject:** RE: answer to questions

Hi,

We actually report 13C/12C values to 1 significant figure. We usually run one
sample two or three times in order to check any variability.
We usually report the average value and the standard deviation.
As you are writing a Thesis, your "correctors" may want see 2 significant figures even if it does not make any sense. For example, -46.63 per mil should
have been reported -46.6 per mil. You should write only -46.6 per mil , but
consider the +/- 0.1 per mil when analyzing your data.

Dr. Anne Warren - Geologist / Geochemist - University of Oklahoma - Sarkeys
Energy Center

# Appendix D

## Correspondence with Church and Dwight Co., Inc.

---

**Date:** Wednesday, April 27, 2011 11:08 AM

**From:** consumer.relations@churchdwight.com

**To:** Keisha Jones <[redacted]>

**Subject:** Reply from Web Form Regarding Trojan® Thintensity™ Condoms, Ref Number: 004753020C

---

Our ref: 004753020C
E-Mail Address: [redacted]

Dear Ms. Jones:

Thank you for visiting our web site regarding Trojan® Thintensity™ Condoms.

Regrettably we are unable to assist with your master's thesis. The information
you requested is considered proprietary and we are unable to provide it.

We wish you luck with your future endeavors.

Again, thank you for taking the time and having the interest to contact us.

Sincerely,

Caroline Reilly
Consumer Relations Representative

004753020C
Please do not reply to this email. If you would like to respond to this message,
please click on the link below.

http://www.econsumeraffairs.com/churchdwight/contactusfollowup.htm?F1=004753020C
&F2=USA&F3=805

---

---

**Date:** Tuesday, May 17, 2011 1:29 PM

**From:** consumer.relations@churchdwight.com

**To:** Keisha Jones <[redacted]>

**Subject:** Reply from Web Form Regarding Trojan® Thintensity™ Condoms, Ref Number: 004753020D

---

Our ref: 004753020D
E-Mail Address: [redacted]

Dear Ms. Jones:

We have received your follow-up email regarding Trojan® Thintensity™ Condoms.

As stated in our previous correspondence to you, specific sales, marketing and
quality information is considered proprietary. We are unable to assist you with
anything further.

Thank you again for contacting us at Church & Dwight Co., Inc.

We wish you luck in your future endeavors.

Sincerely,

Caroline Reilly
Consumer Relations Representative

004753020D
Please do not reply to this email. If you would like to respond to this message,
please click on the link below.

http://www.econsumeraffairs.com/churchdwight/contactusfollowup.htm?F1=004753020D
&F2=USA&F3=805

---

**Oklahoma State Bureau of Investigation**

STAN FLORENCE
*Director*

CHARLES D. CURTIS
*Deputy Director*

May 12, 2011

Caroline Reilly
Consumer Relations Representative
Church & Dwight Co., Inc.
469 North Harrison Street
Princeton, NJ 08543-5297

Dear Ms. Reilly,

The Oklahoma State Bureau of Investigation (OSBI) Laboratory has partnered with the University of Central Oklahoma's Forensic Science Institute (FSI) in the development of a forensic laboratory protocol in the area of sexual assault criminal investigations. As you may know, sexual predators having assimilated a rudimentary understanding of forensic DNA analysis have transitioned to the use of condoms in order to obscure their identities during these acts of violence. It has been hypothesized by some in the forensic community that, when used, condoms leave behind an informative forensic signature. FSI graduate student Keisha Jones has been dedicated to the development of an analytical protocol and supporting database of condom signatures as a Master of Science in Forensic Science thesis project.

Research has shown that many individuals who sexually assault victims will commit multiple attacks. The largest percentage of cases processed in the OSBI's Forensic Biology Unit involves sexual assaults, and we realize that a number of these assaults will have been committed by the same perpetrator. Our lab, therefore, understands the importance to law enforcement that they have testing procedures available to quickly identify individuals involved in sexual assaults. We believe that Ms. Jones' efforts to develop an appropriate protocol and database of condom signatures will benefit the forensic community as well as victims of rape.

The OSBI appreciates any assistance you may offer law enforcement in this endeavor to ultimately associate condom signatures developed from post-event swabs to particular products. The OSBI, FSI, and Ms. Jones are cognizant of your firm's proprietary interests, and we understand reluctance to allow access to sensitive information. However, we strongly believe that this forensic research could aid in solving crimes and possibly preventing future crimes. For this reason, we would welcome the opportunity to discuss ways in which we can accomplish our goals while preserving your proprietary interests.

Thank you for your time and consideration.

Respectfully,

Andrea Swiech
Division Director, Criminalistics
Oklahoma State Bureau of Investigation

HEADQUARTERS
6600 N. Harvey
Oklahoma City, OK 73116-7910
(405) 848-6724
Fax (405) 843-3804
TDD (405) 843-7303

# Appendix E

## Chromatograms and Mass Spectra for Liquid-Liquid Extraction

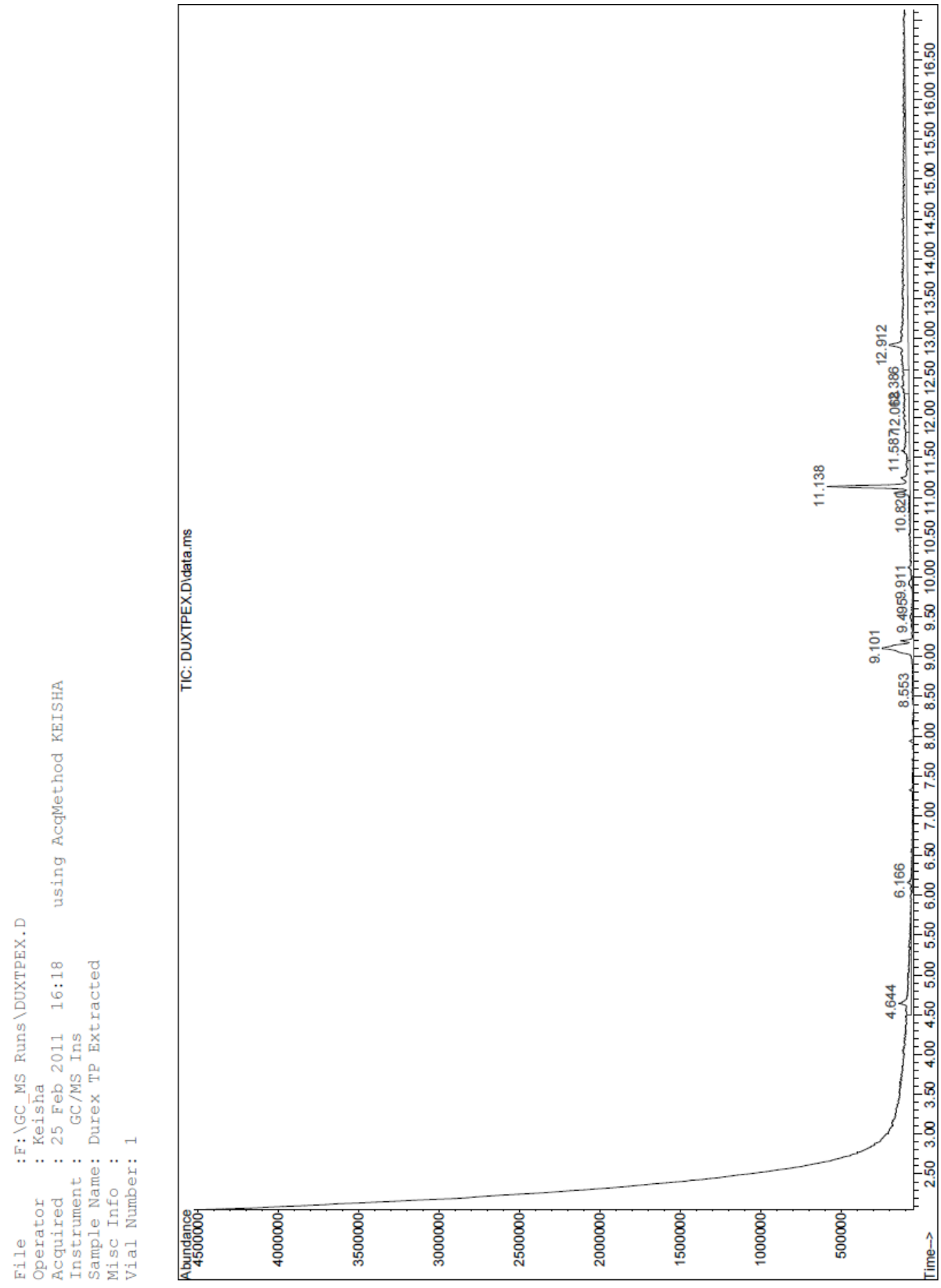


Figure E1. DUXTPEX TIC

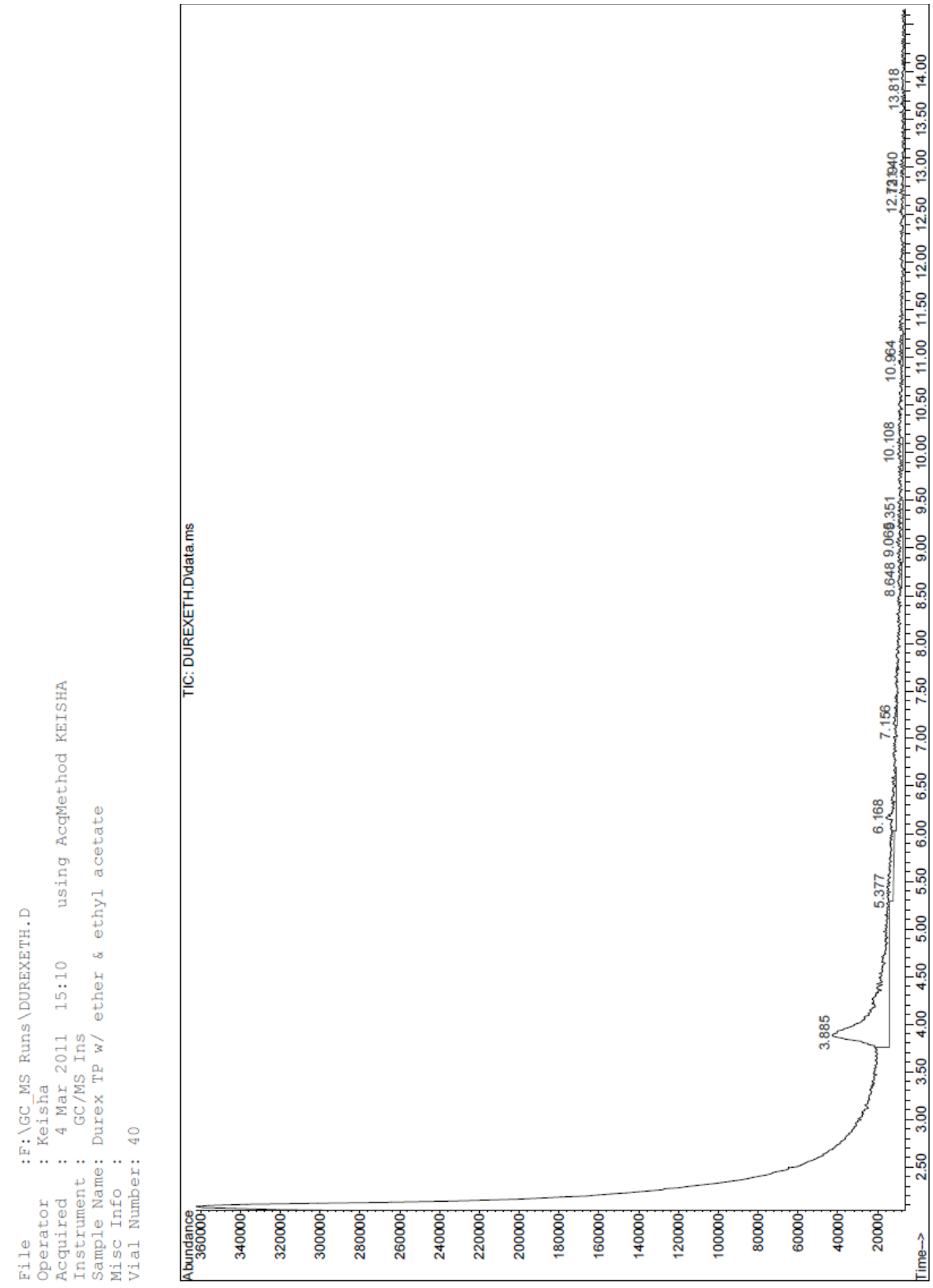


Figure E2. DUREXETH TIC

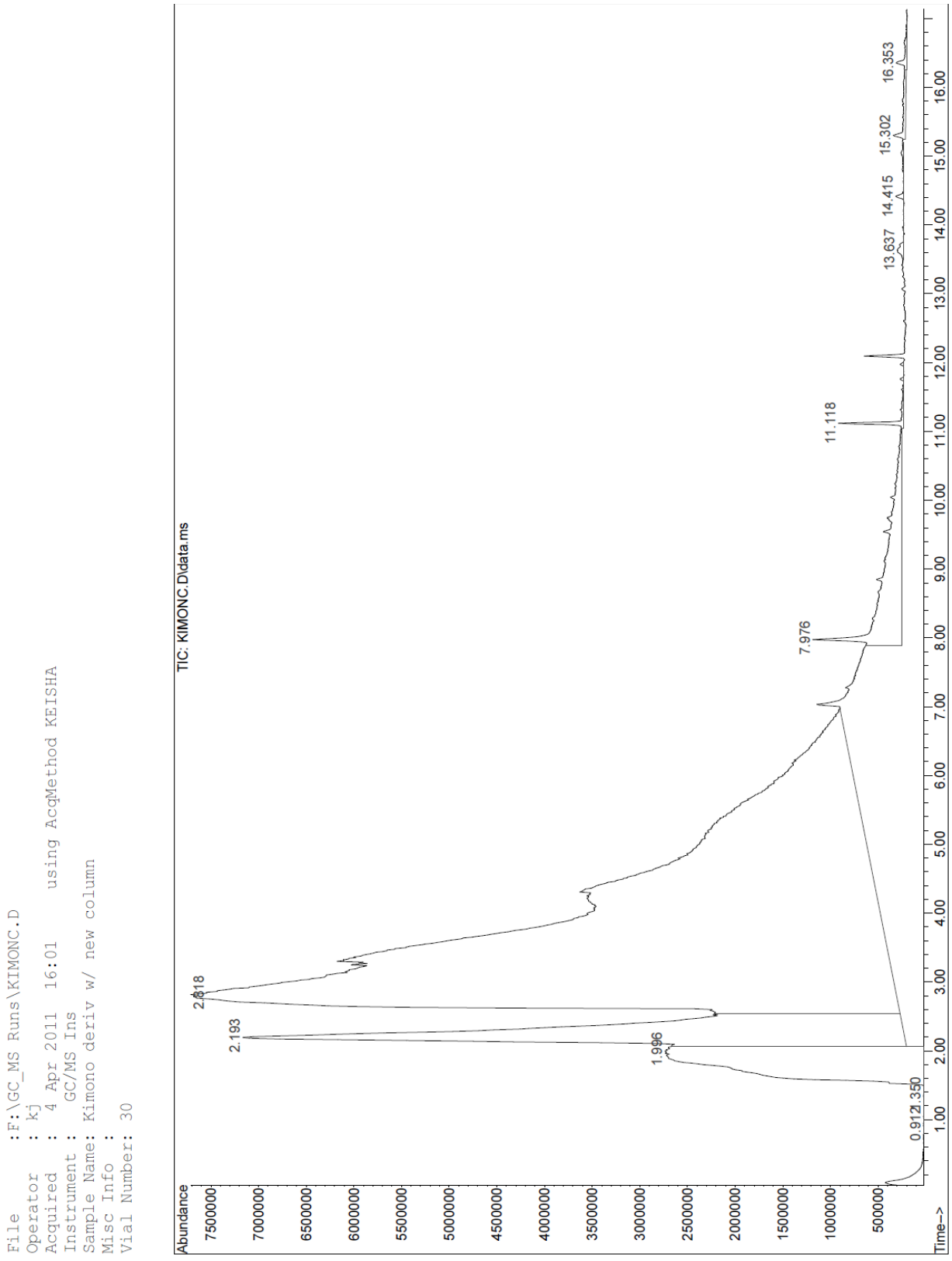


Figure E3. KIMONC TIC

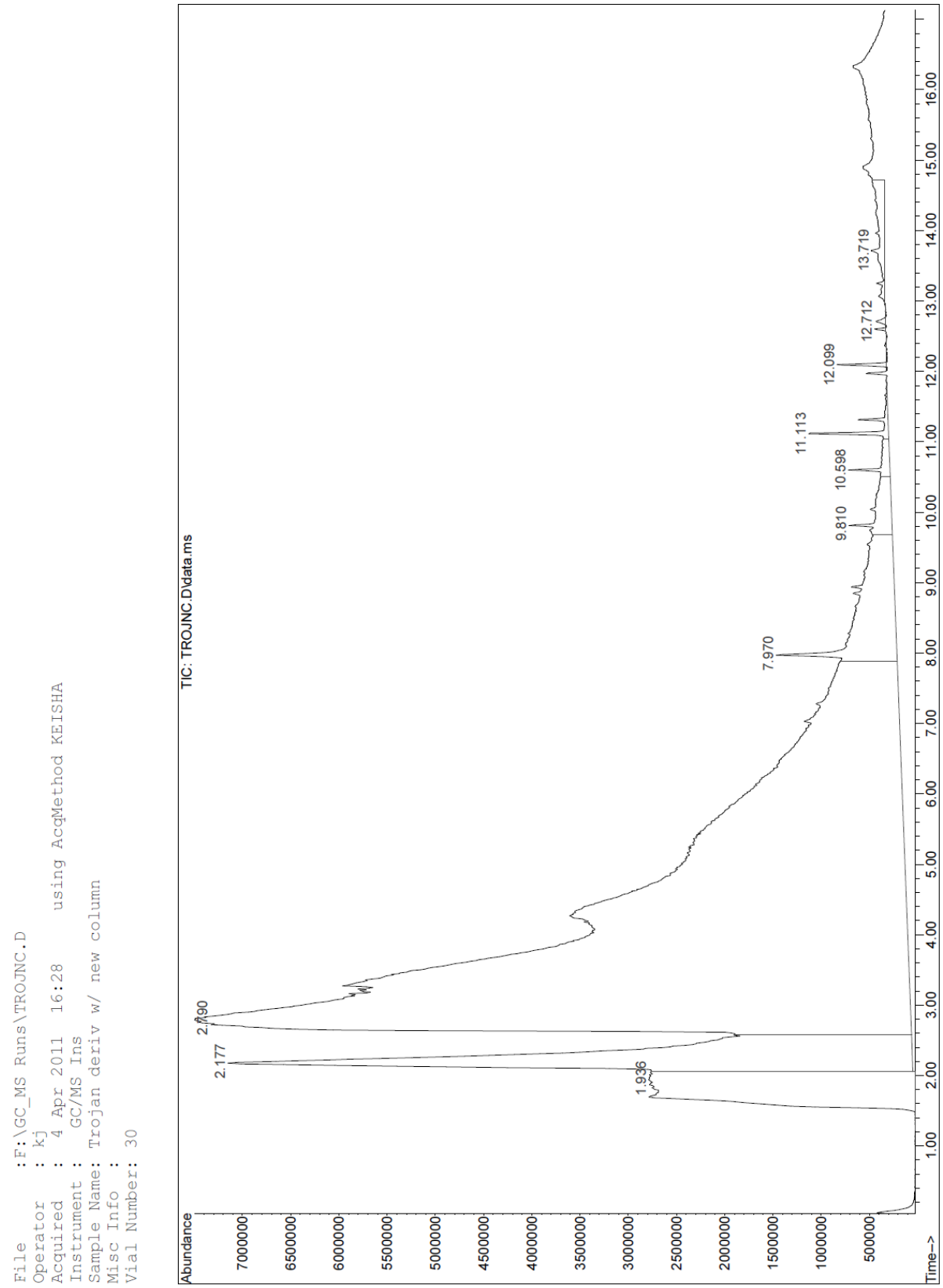


Figure E4. TROJNC TIC

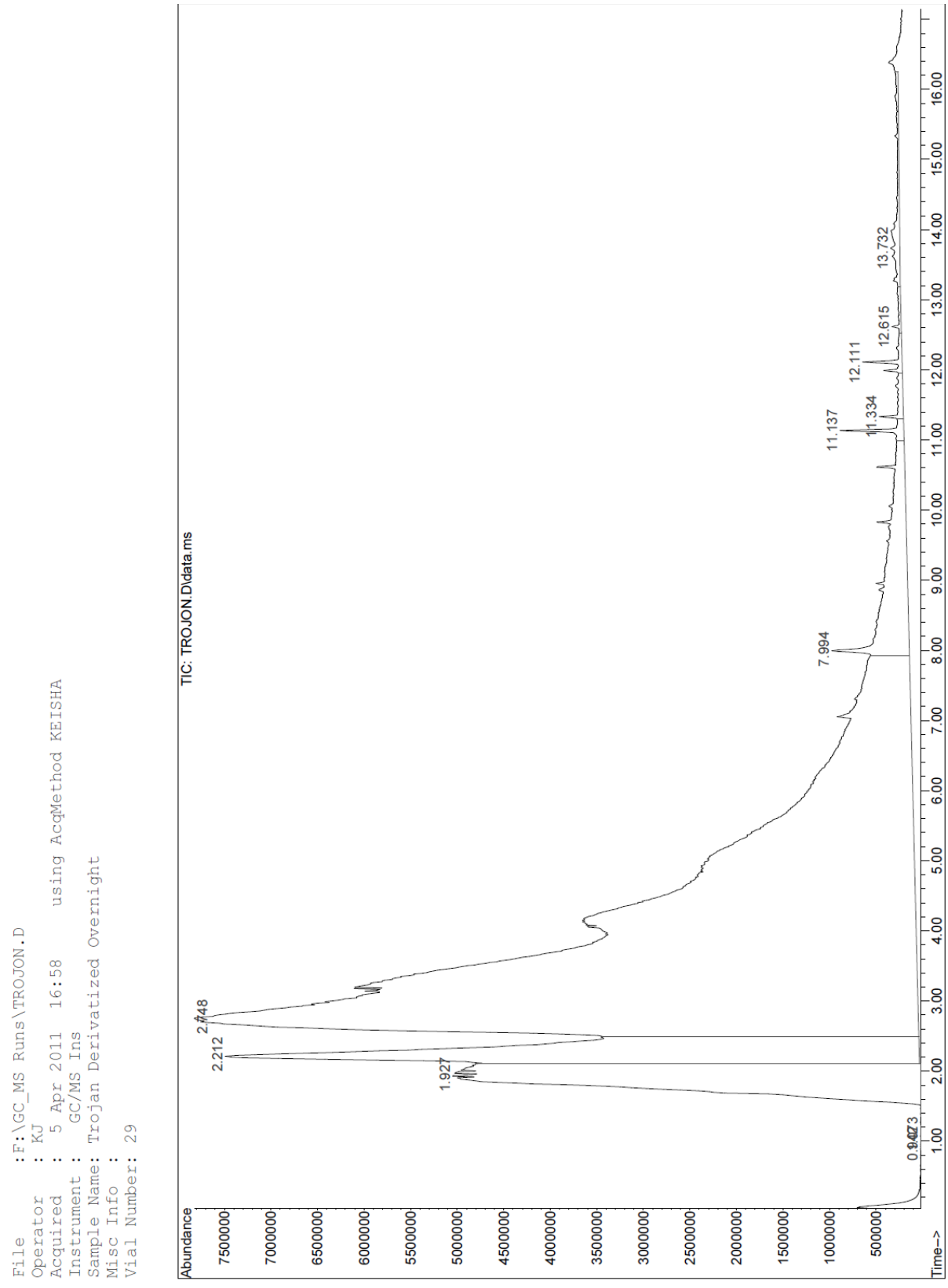


Figure E5. TROJON TIC

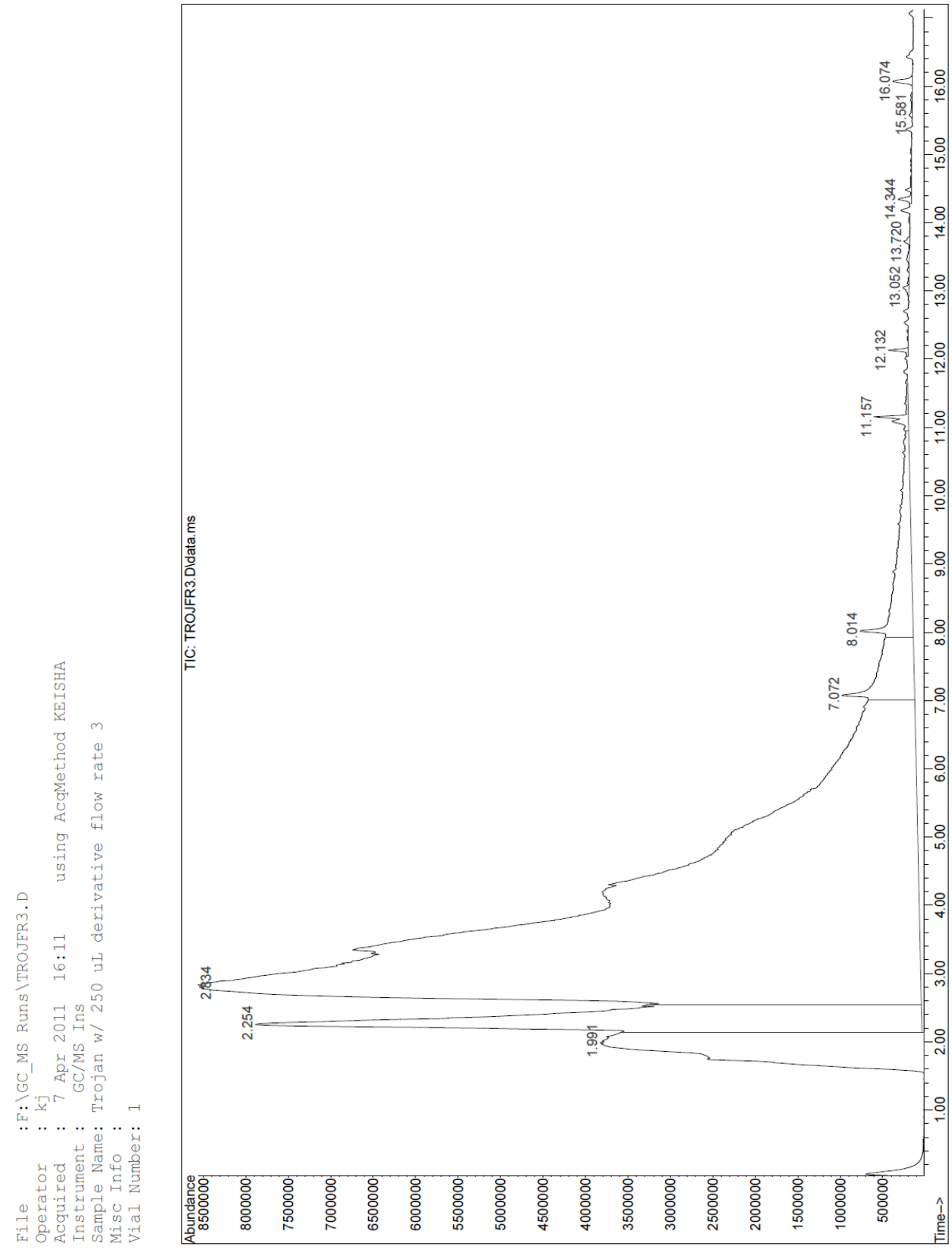


Figure E6. TROJFR3 TIC

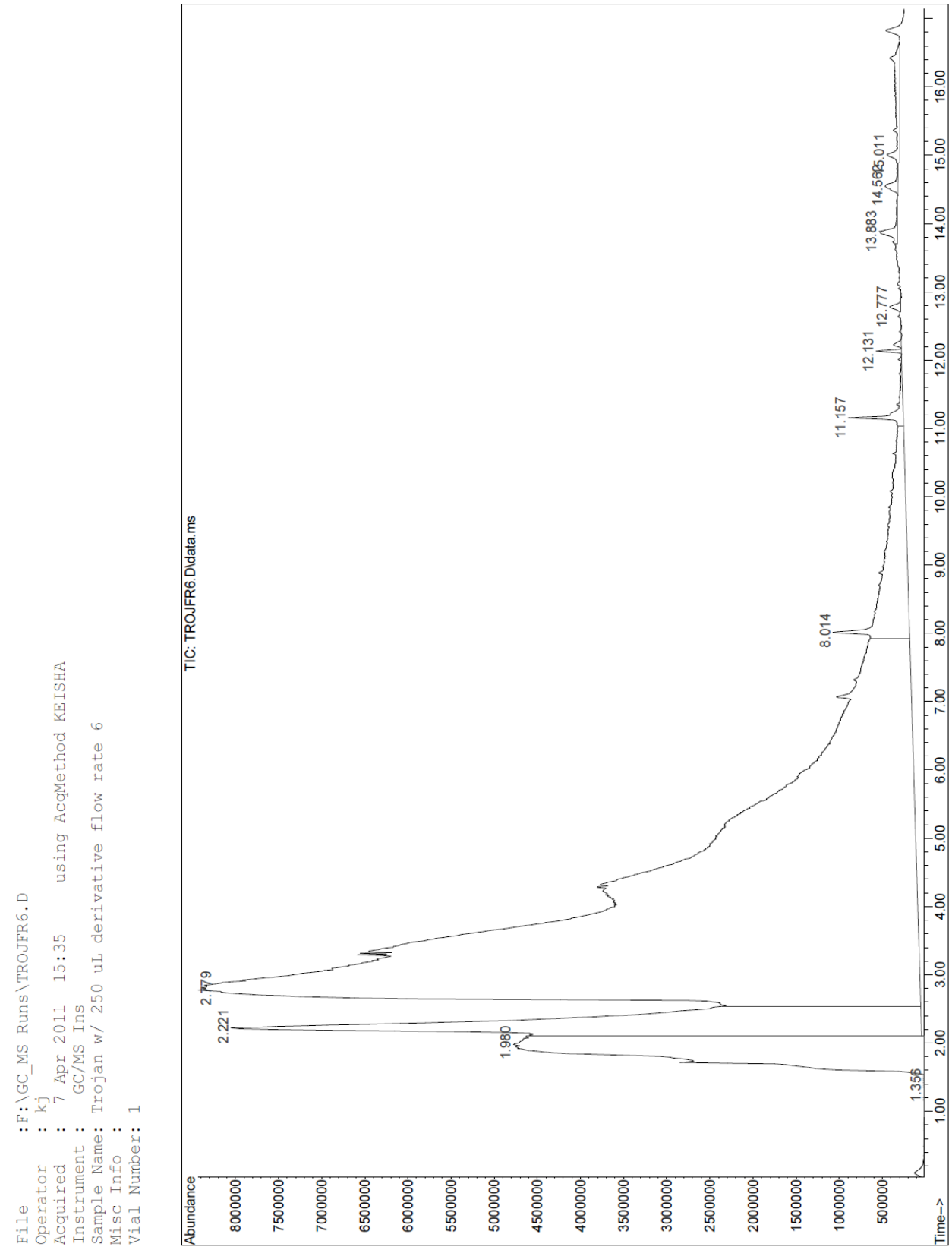


Figure E7. TROJFR6 TIC

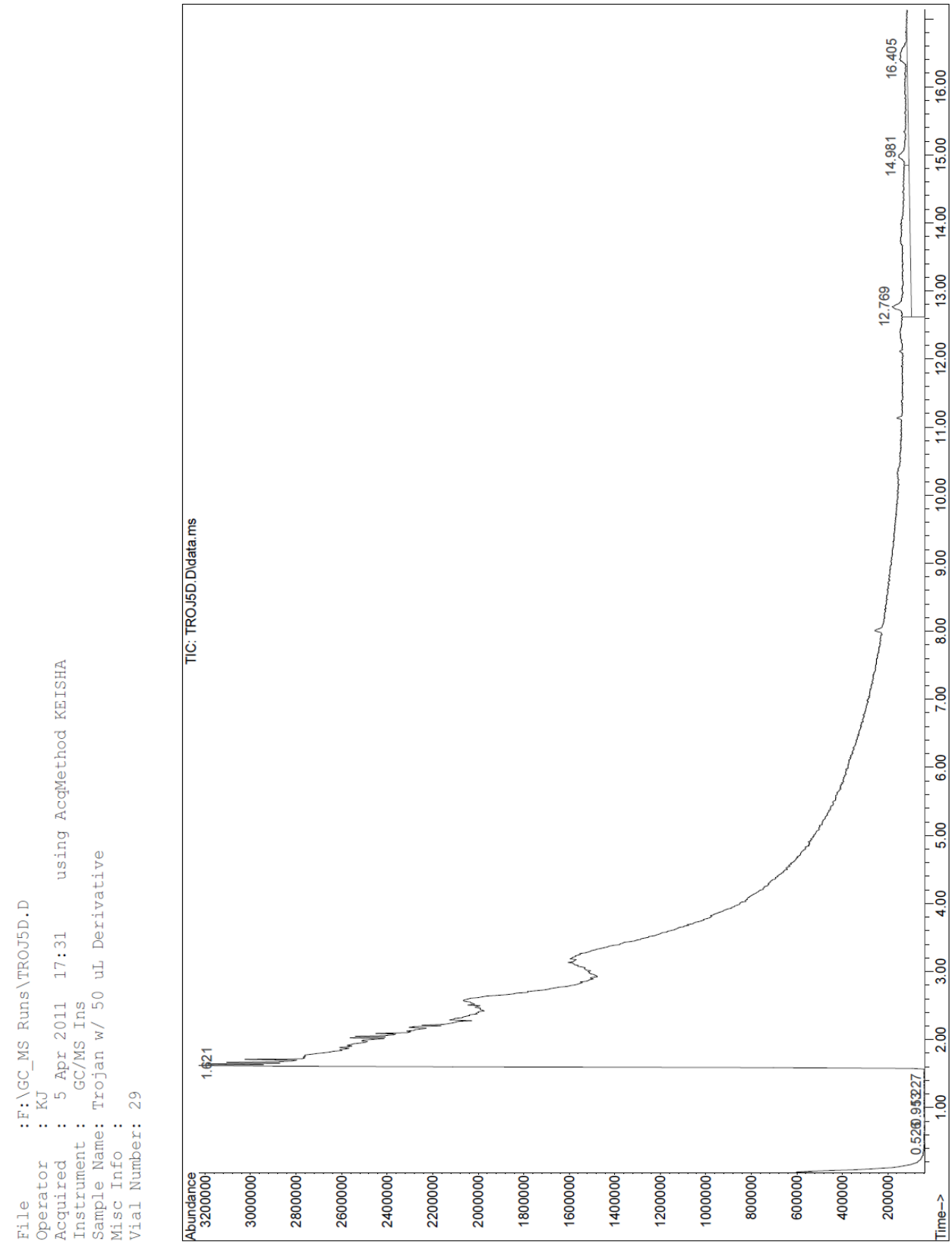


Figure E8. TROJ5D TIC

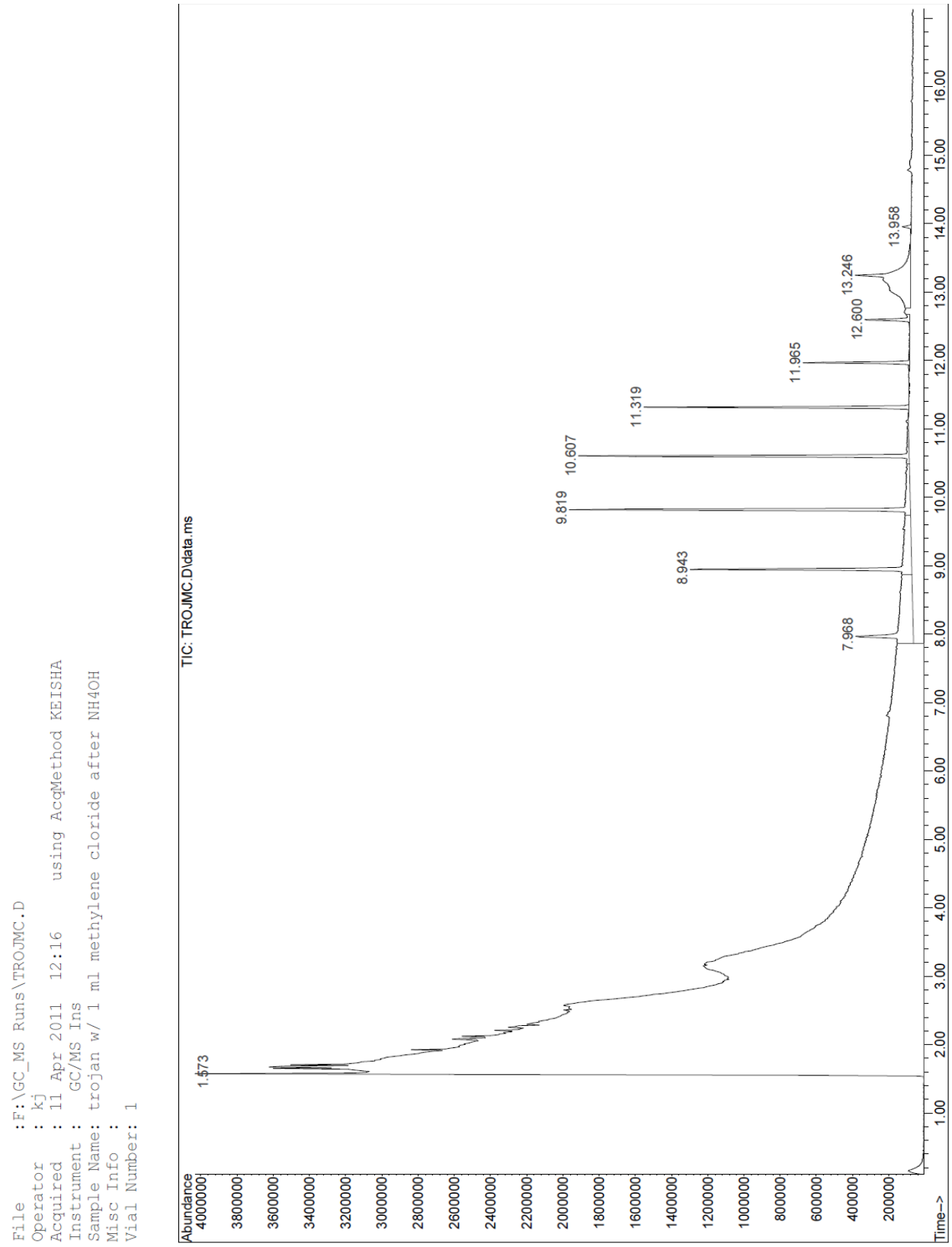


Figure E9. TROJMC TIC

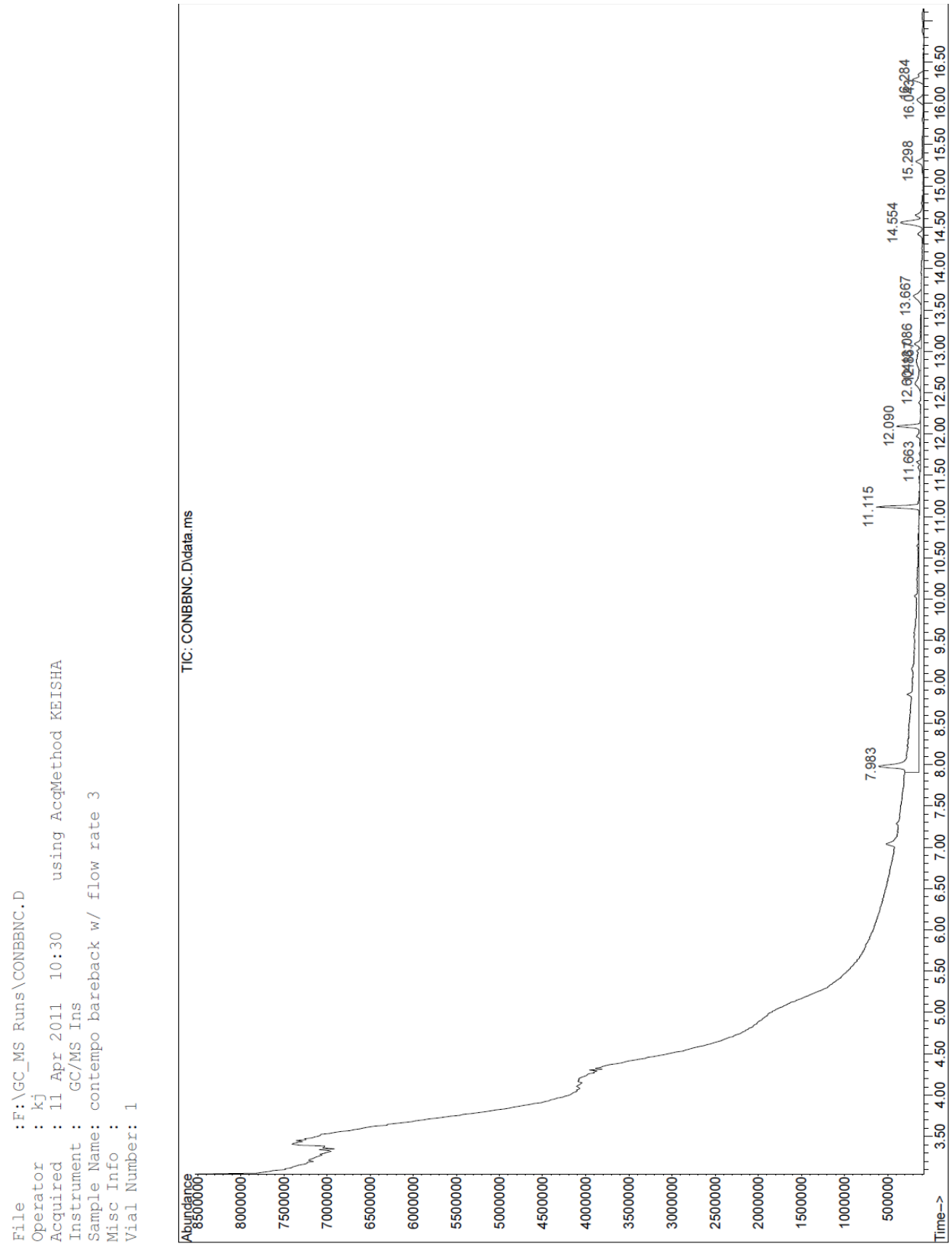


Figure E10. CONBBNC TIC

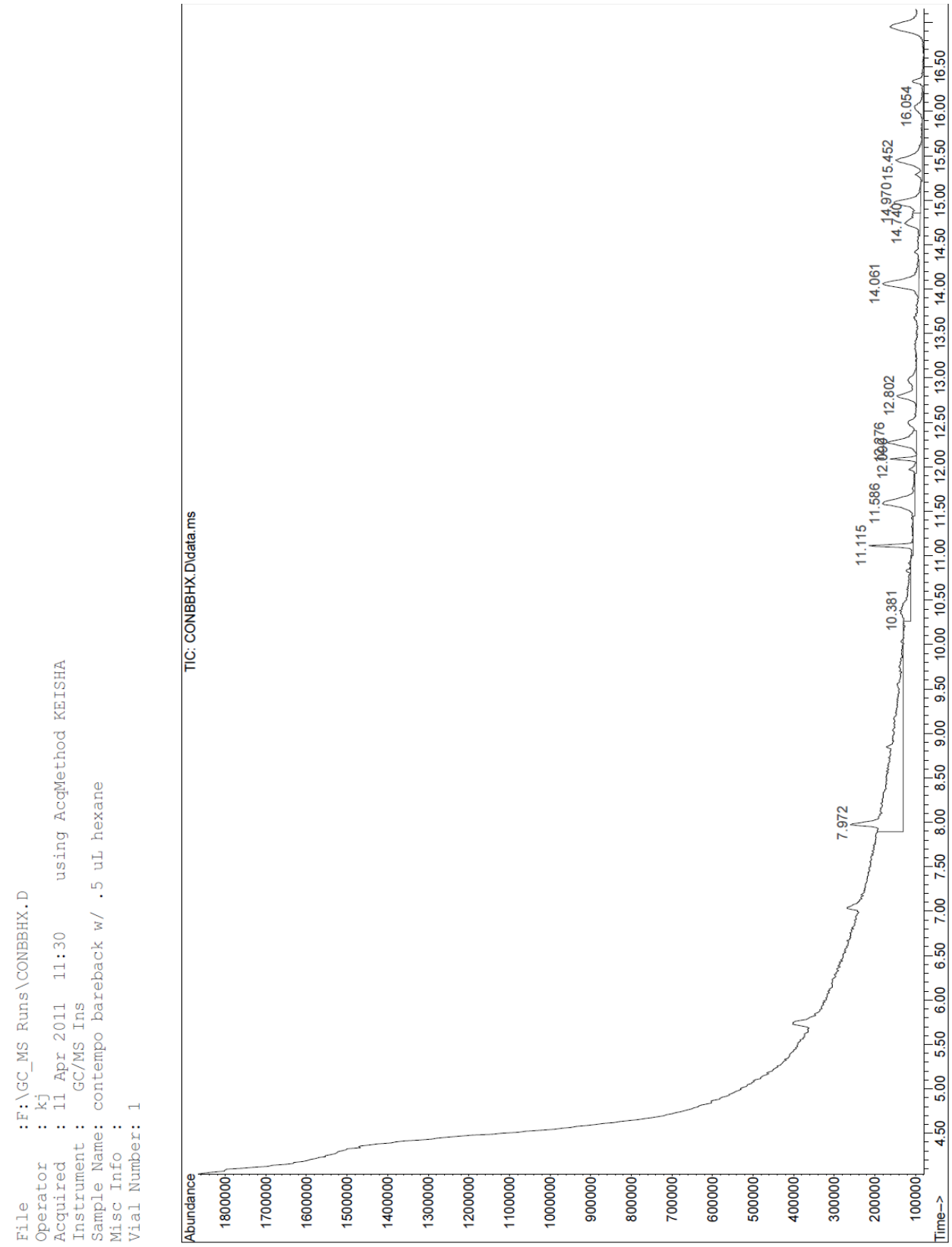


Figure E11. CONBBHX TIC

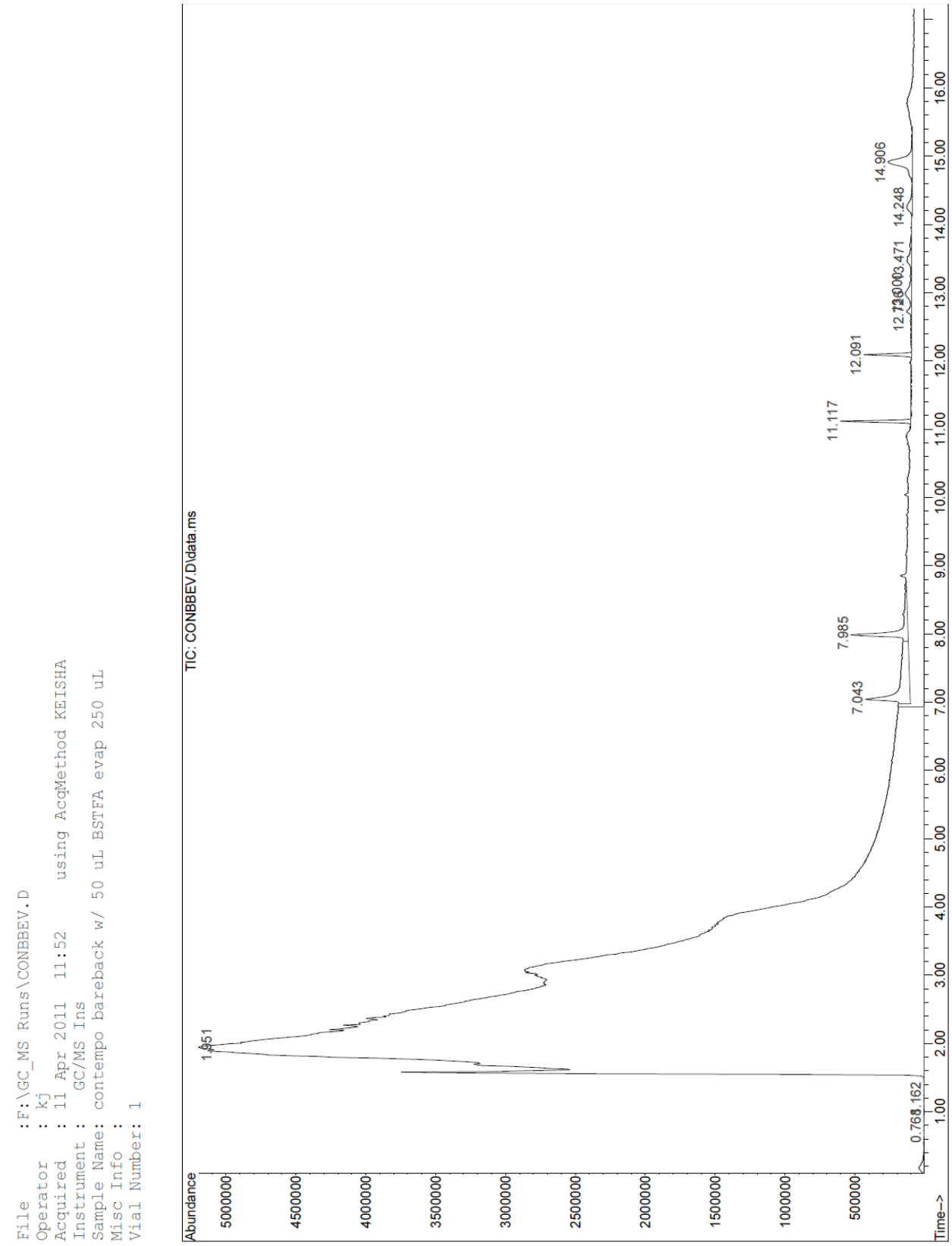


Figure E12. CONBBEV TIC

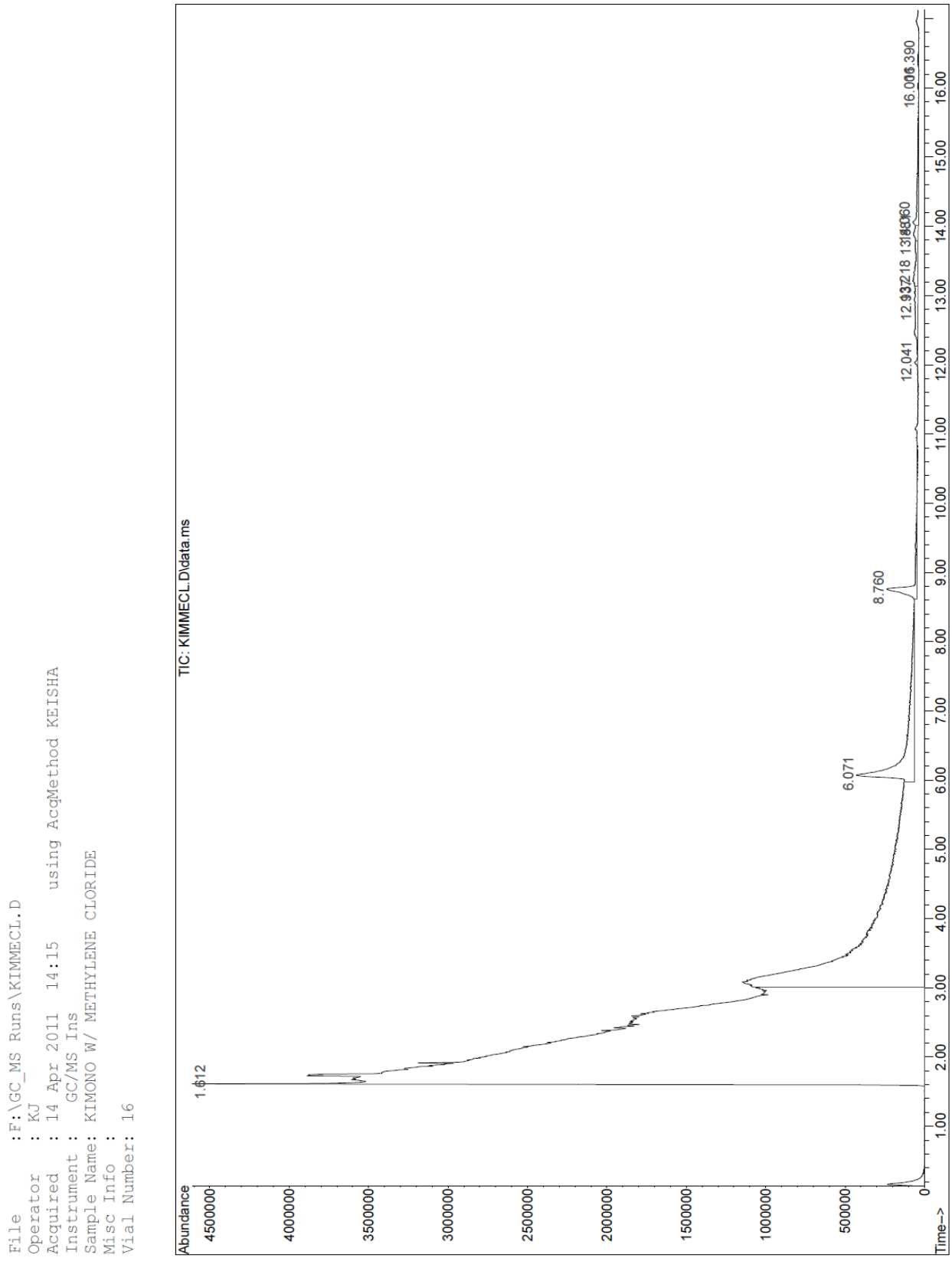


Figure E13. KIMMECL TIC

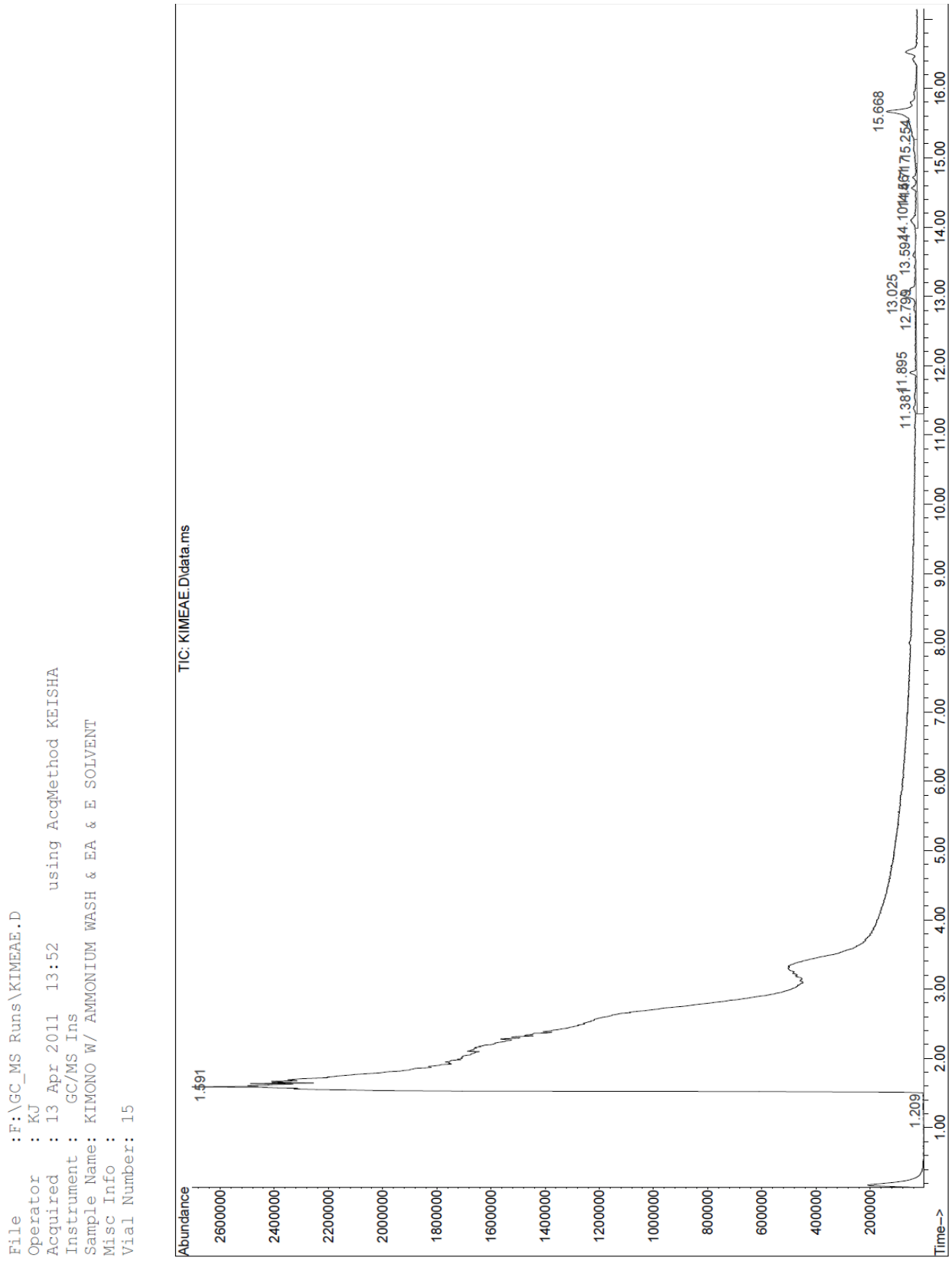


Figure E14. KIMEAE TIC

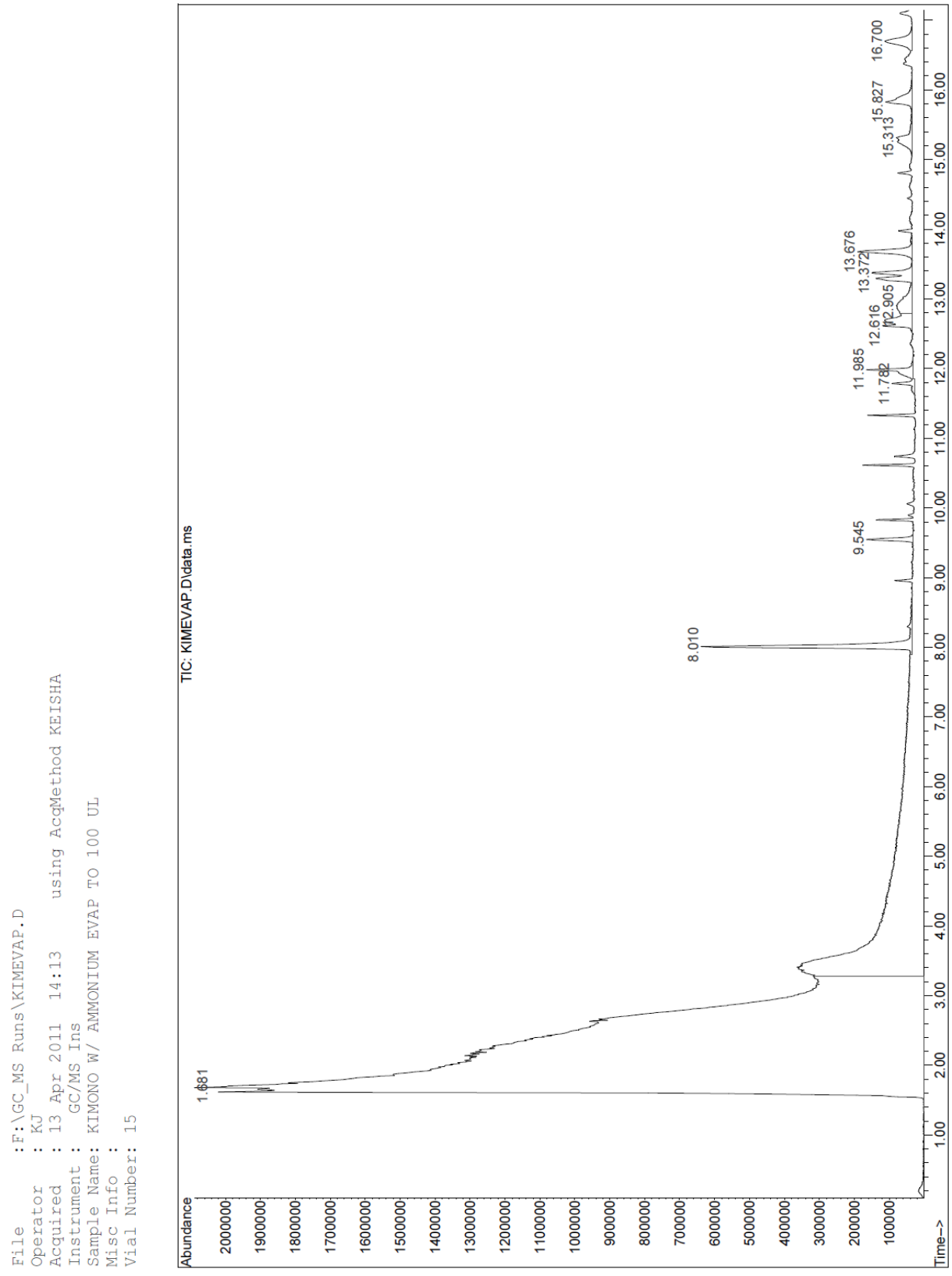


Figure E15. KIMEVAP TIC

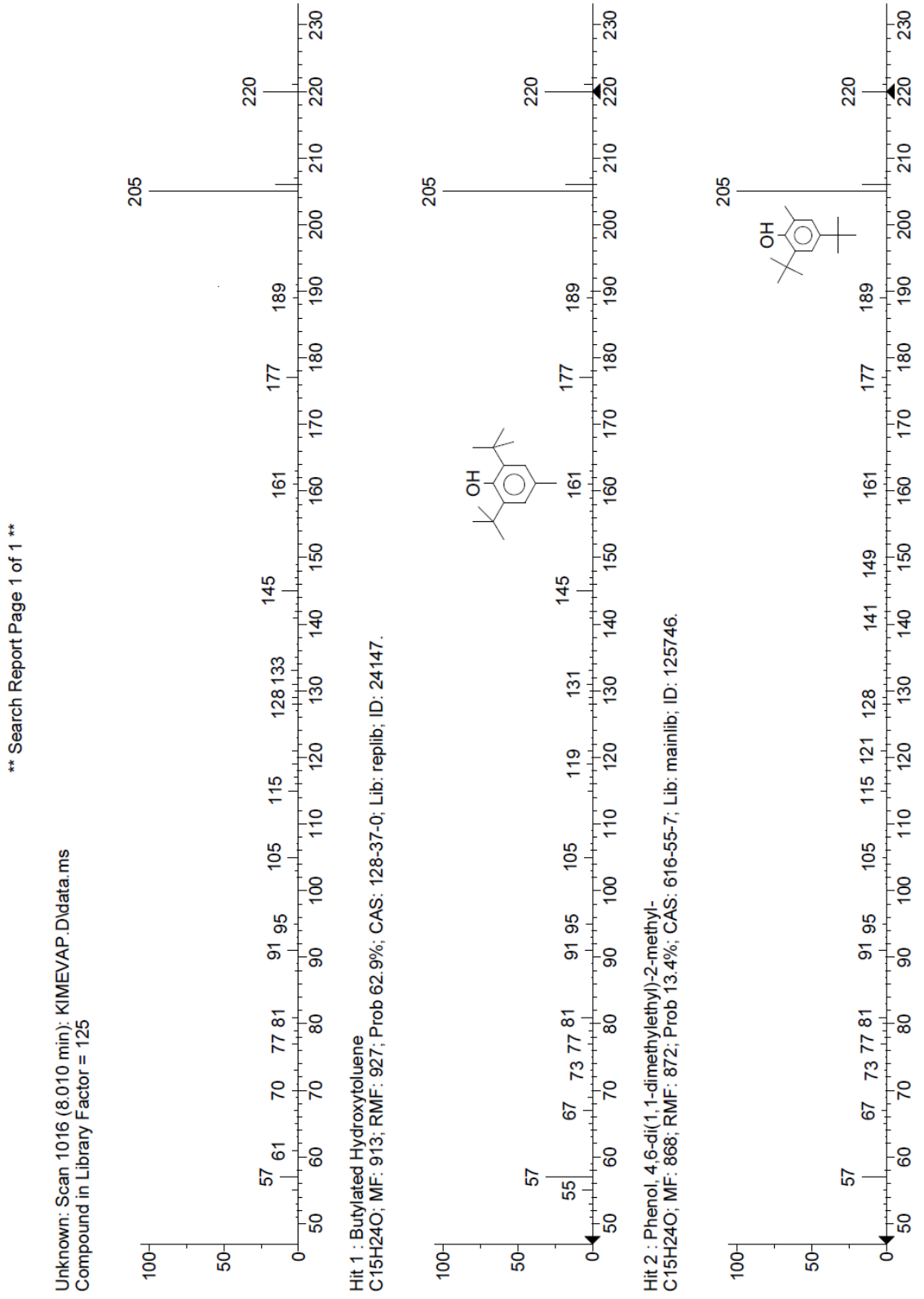


Figure E16. KIMEVAP RT 8.010 min MS

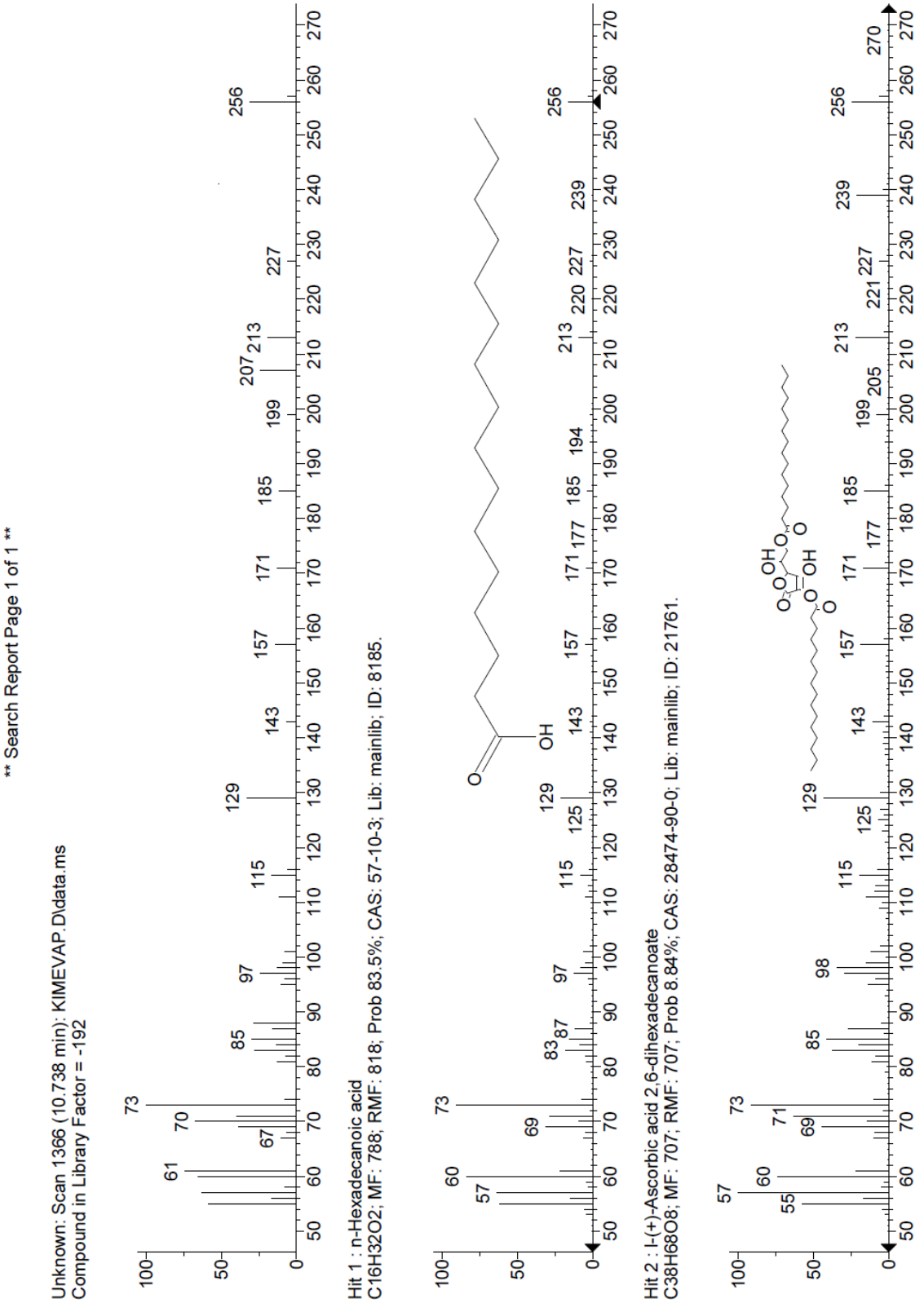


Figure E17. KIMEVAP RT 10.738 min MS

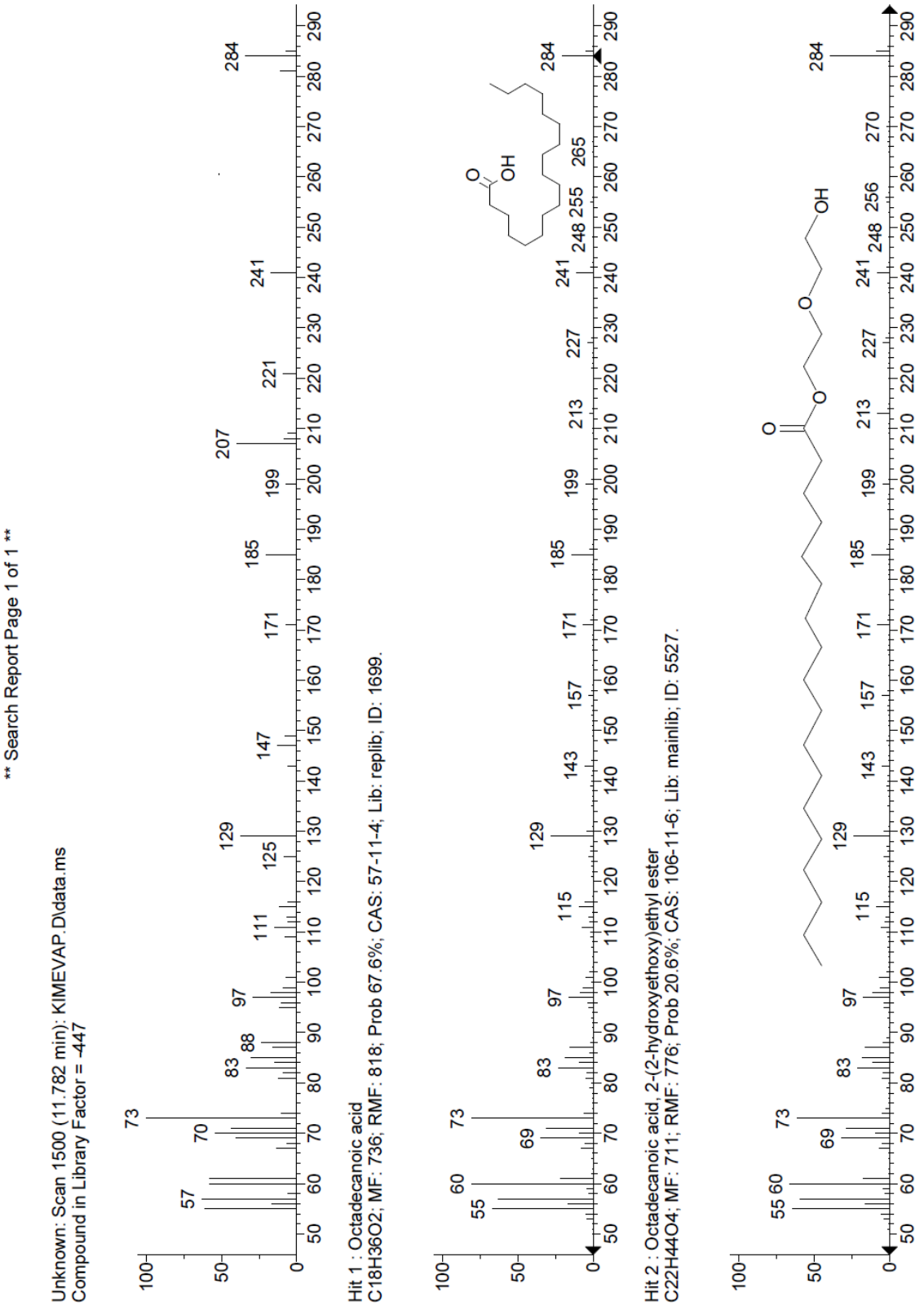


Figure E18. KIMEVAP RT 11.782 min MS

# Appendix F

## Chromatograms for PDMS SPME Fiber

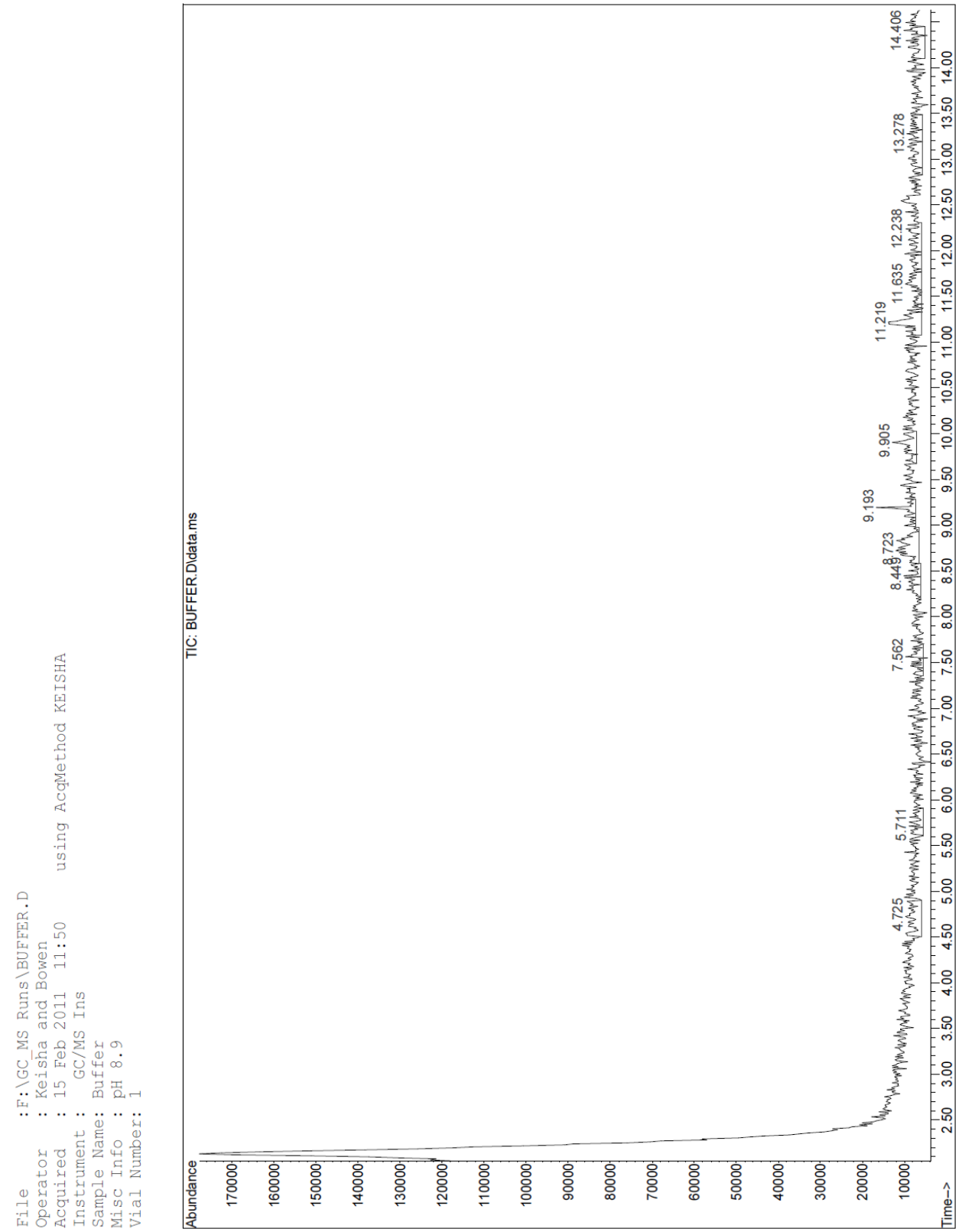


Figure F1. BUFFER TIC

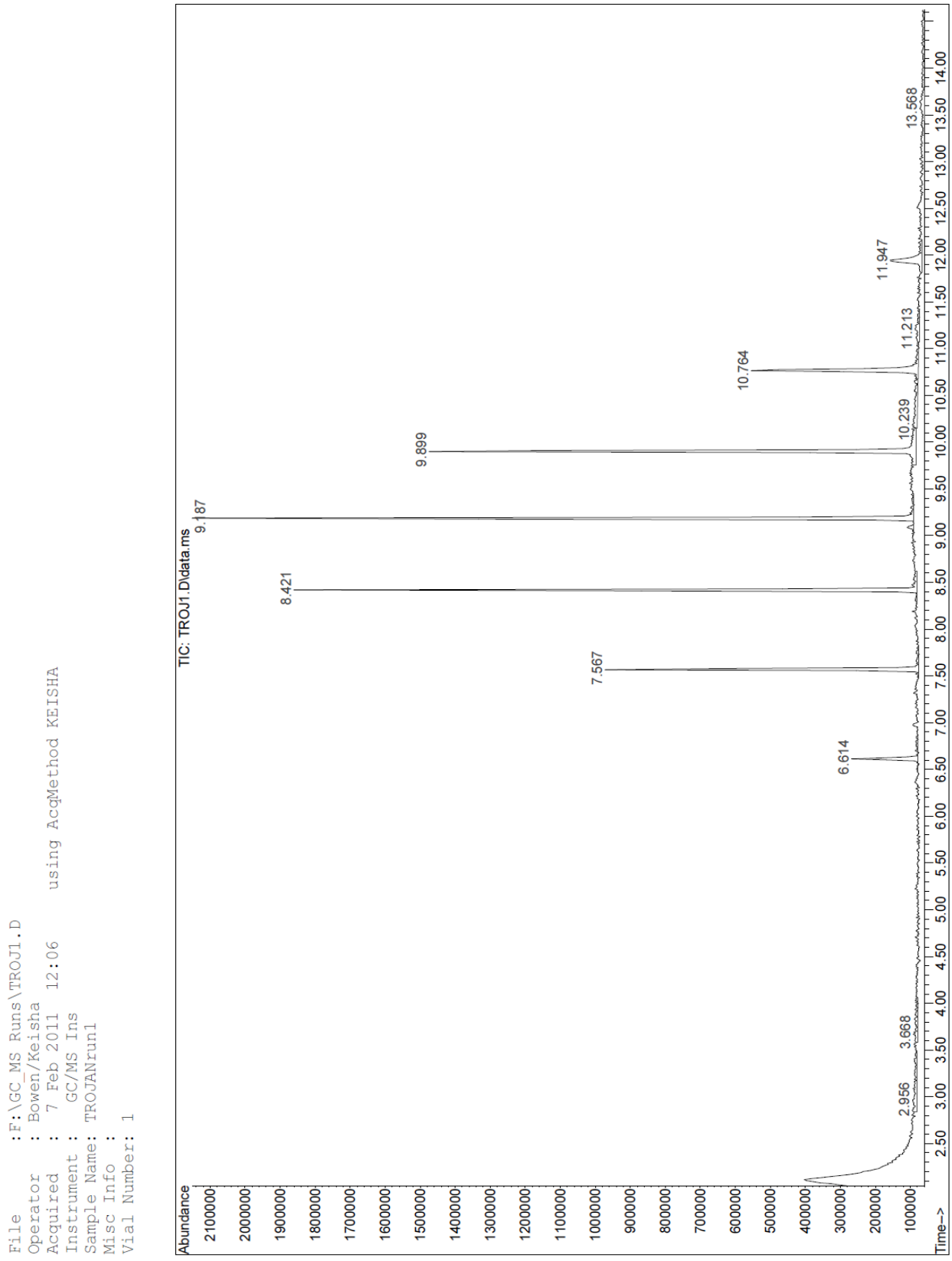


Figure F2. TROJ1 TIC

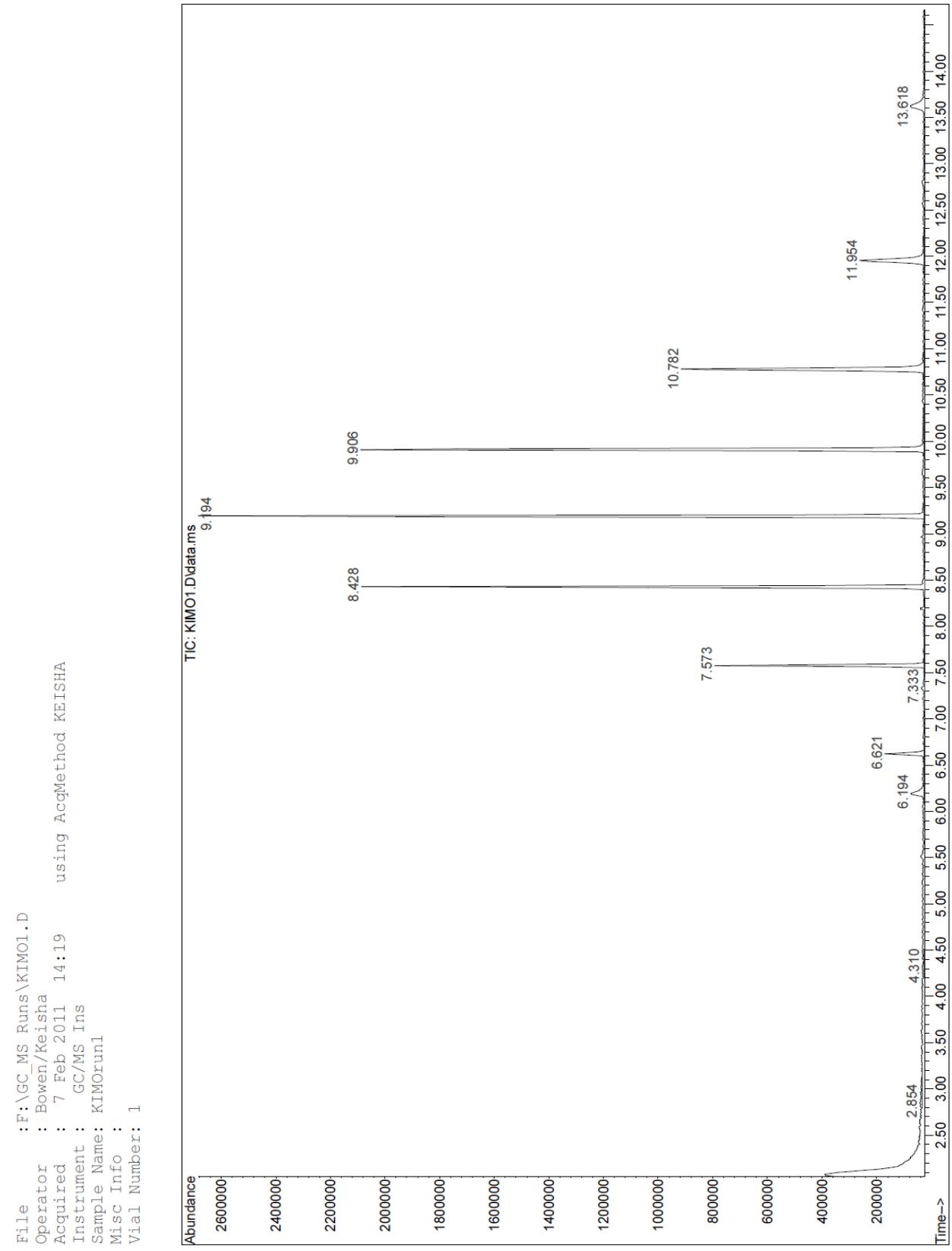


Figure F3. KIMO1 TIC

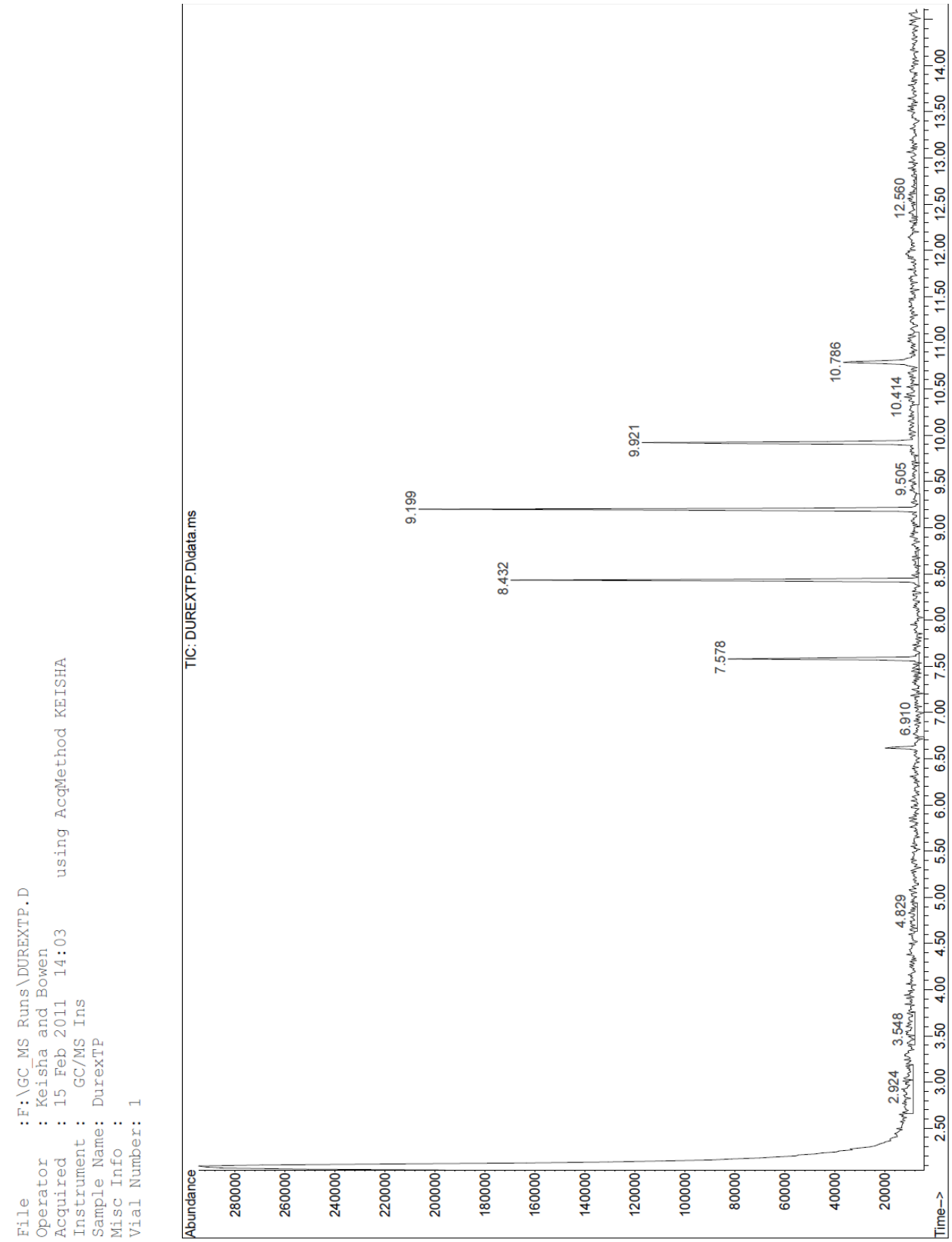


Figure F4. DUREXTP TIC

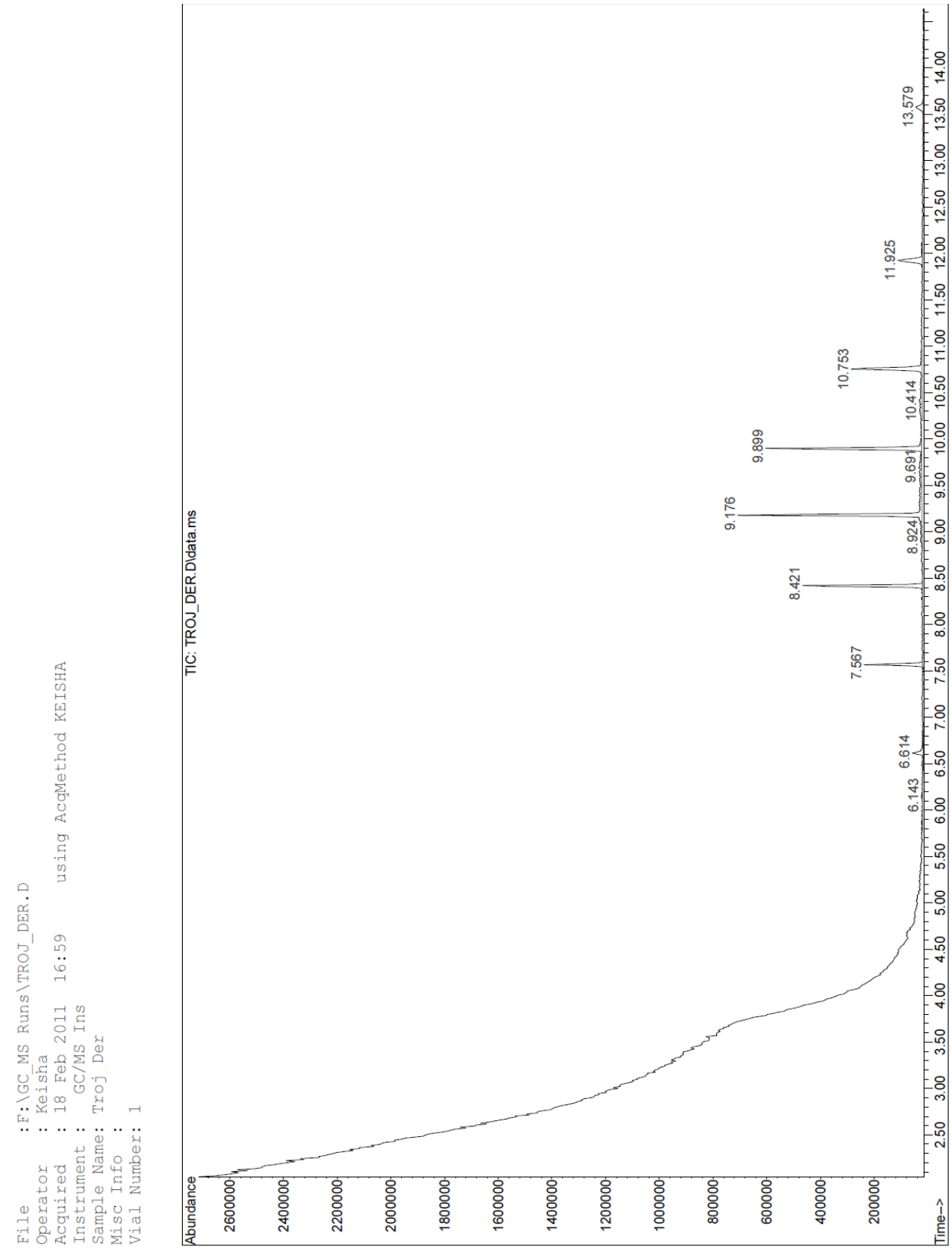


Figure F5. TROJ_DER TIC

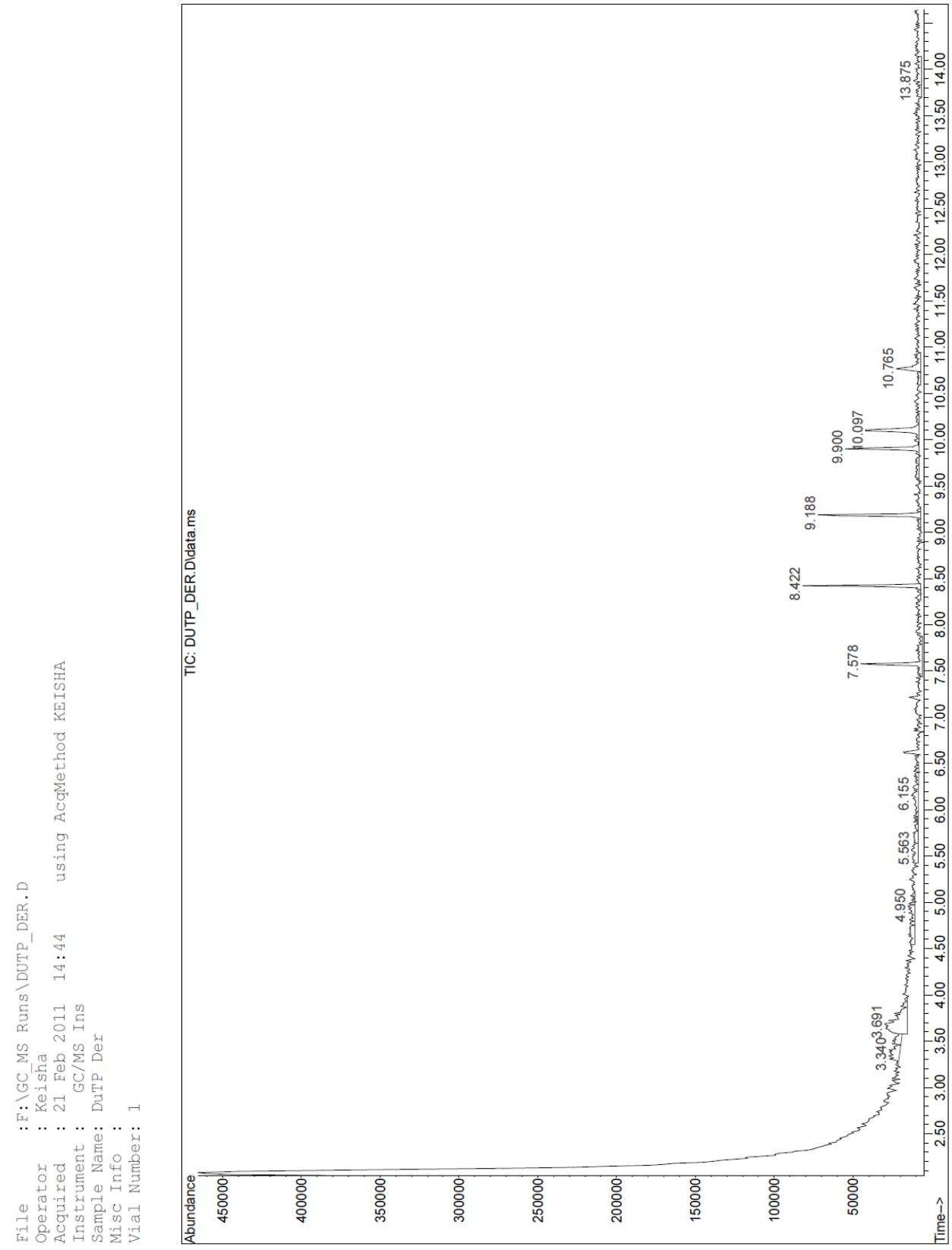


Figure F6. DUTP_DER TIC

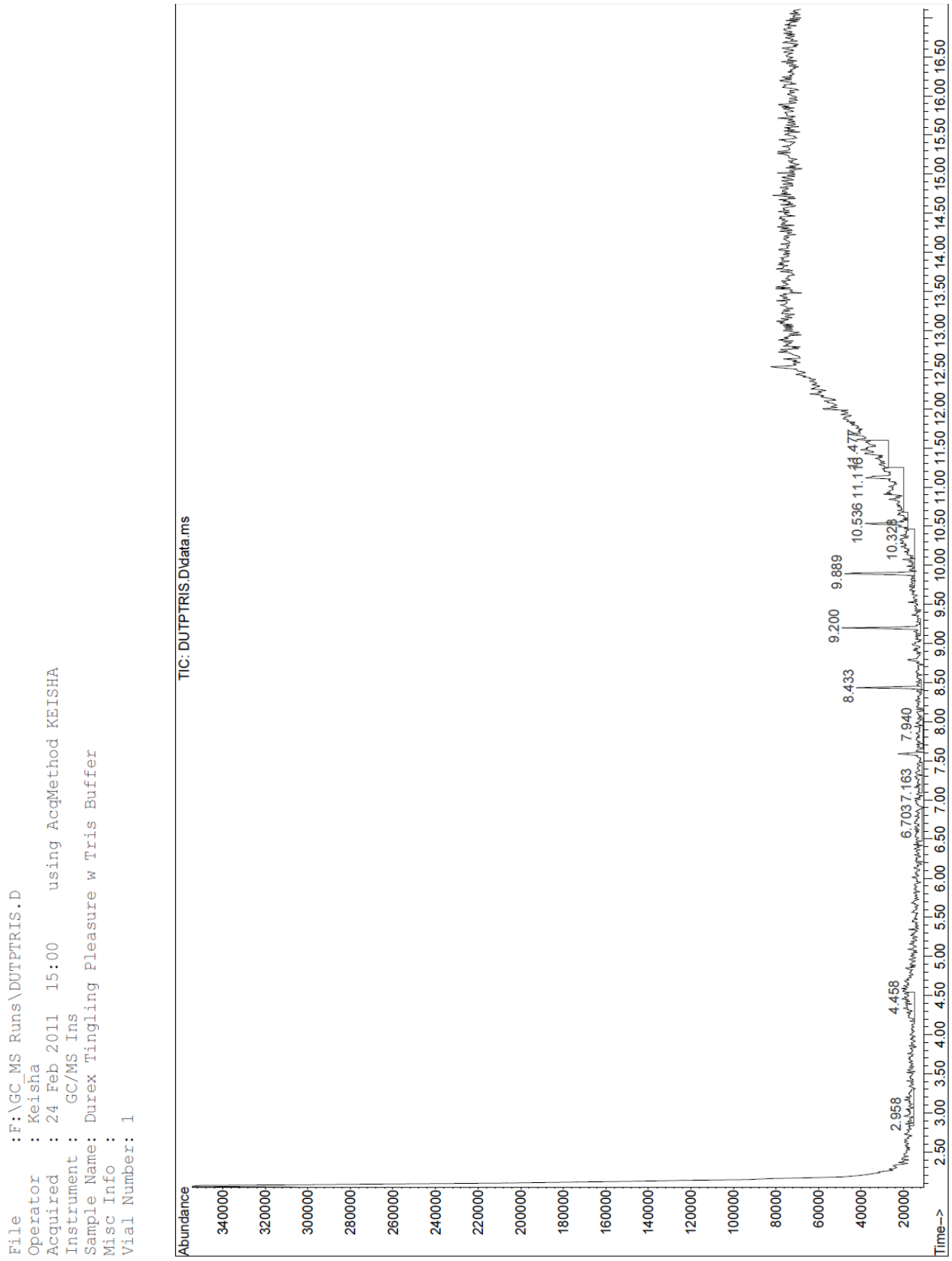


Figure F7. DUTPTRIS TIC

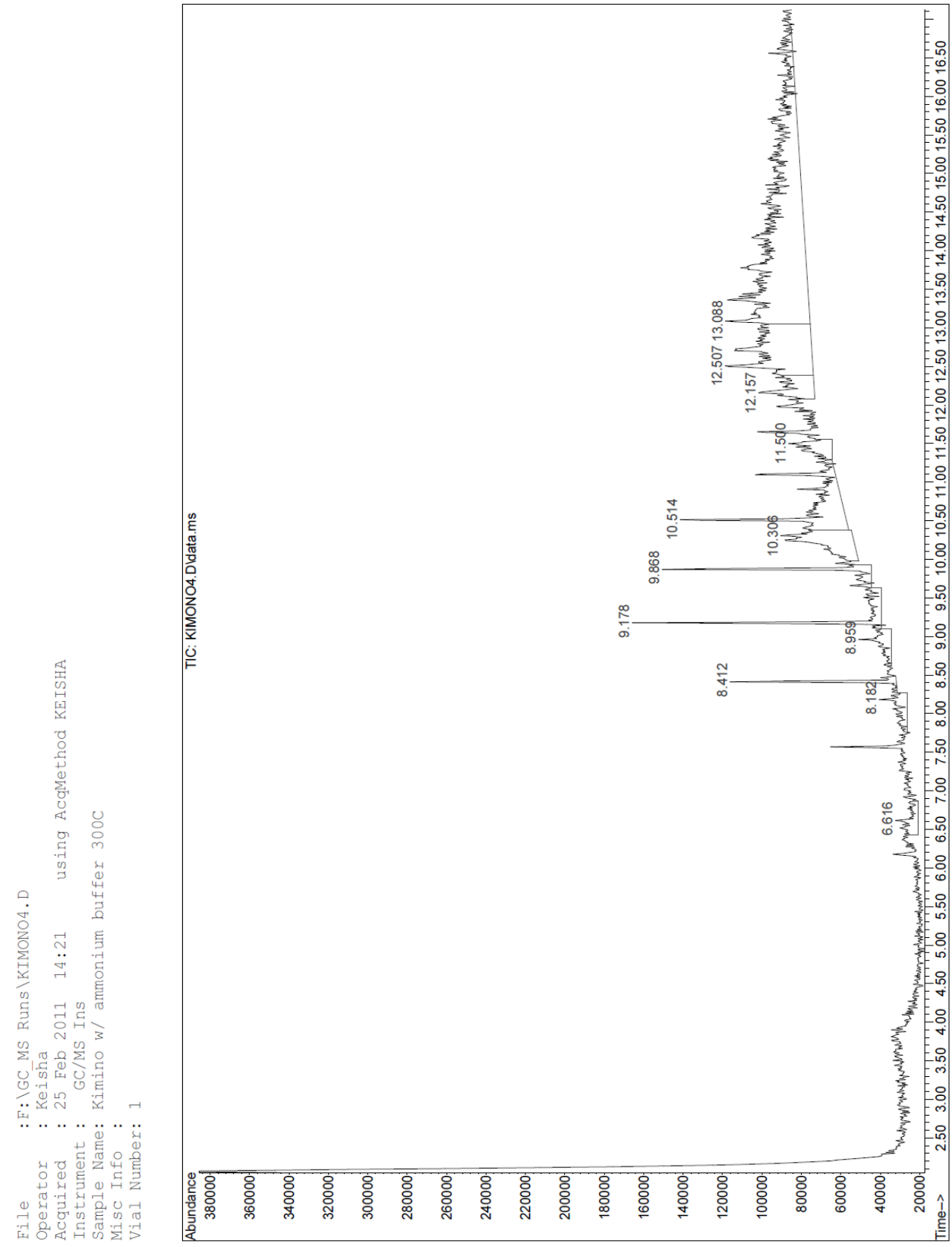


Figure F8. KIMONO4 TIC

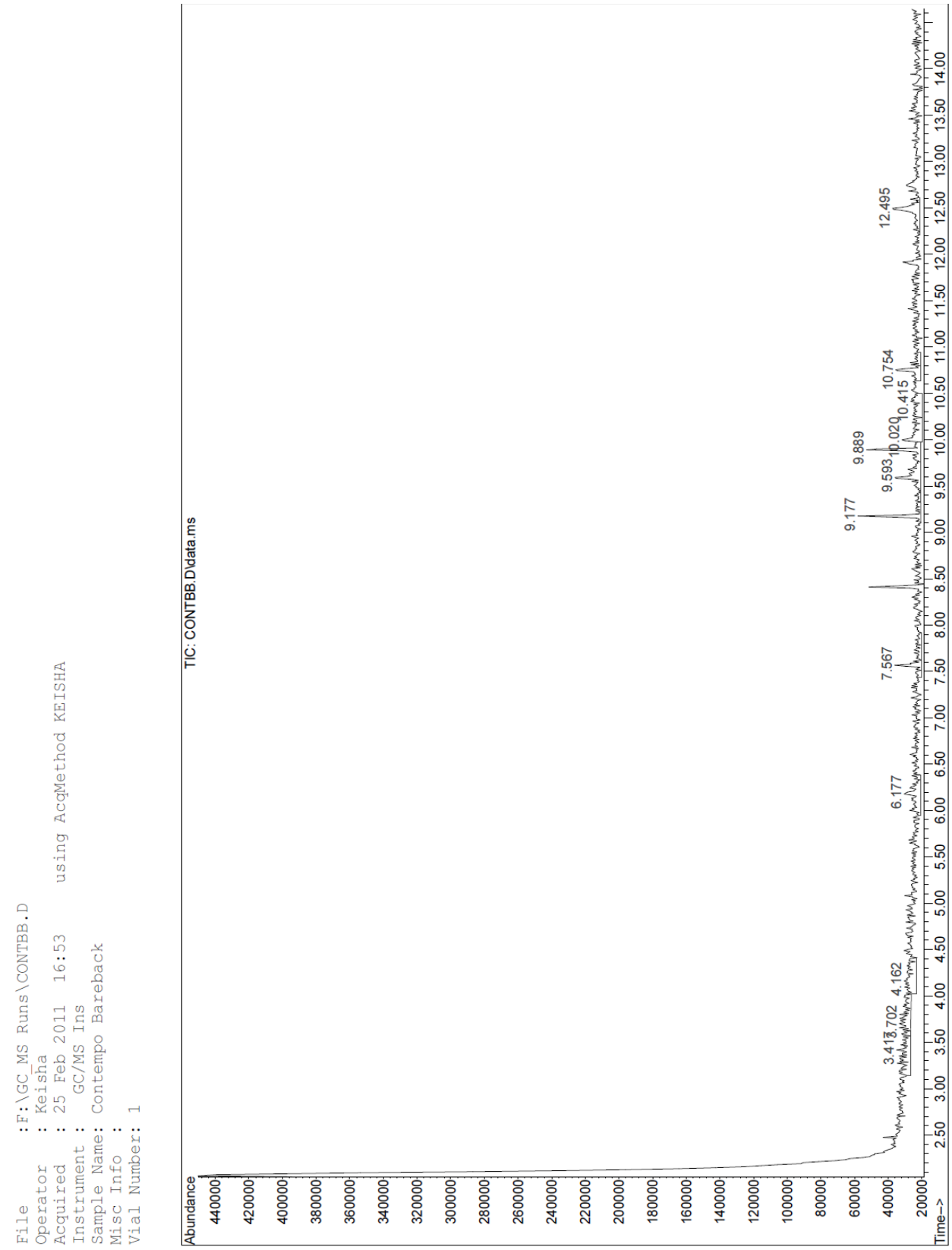


Figure F9. CONTBB TIC

# Appendix G

## Chromatograms for PDMS/DVB SPME Fiber

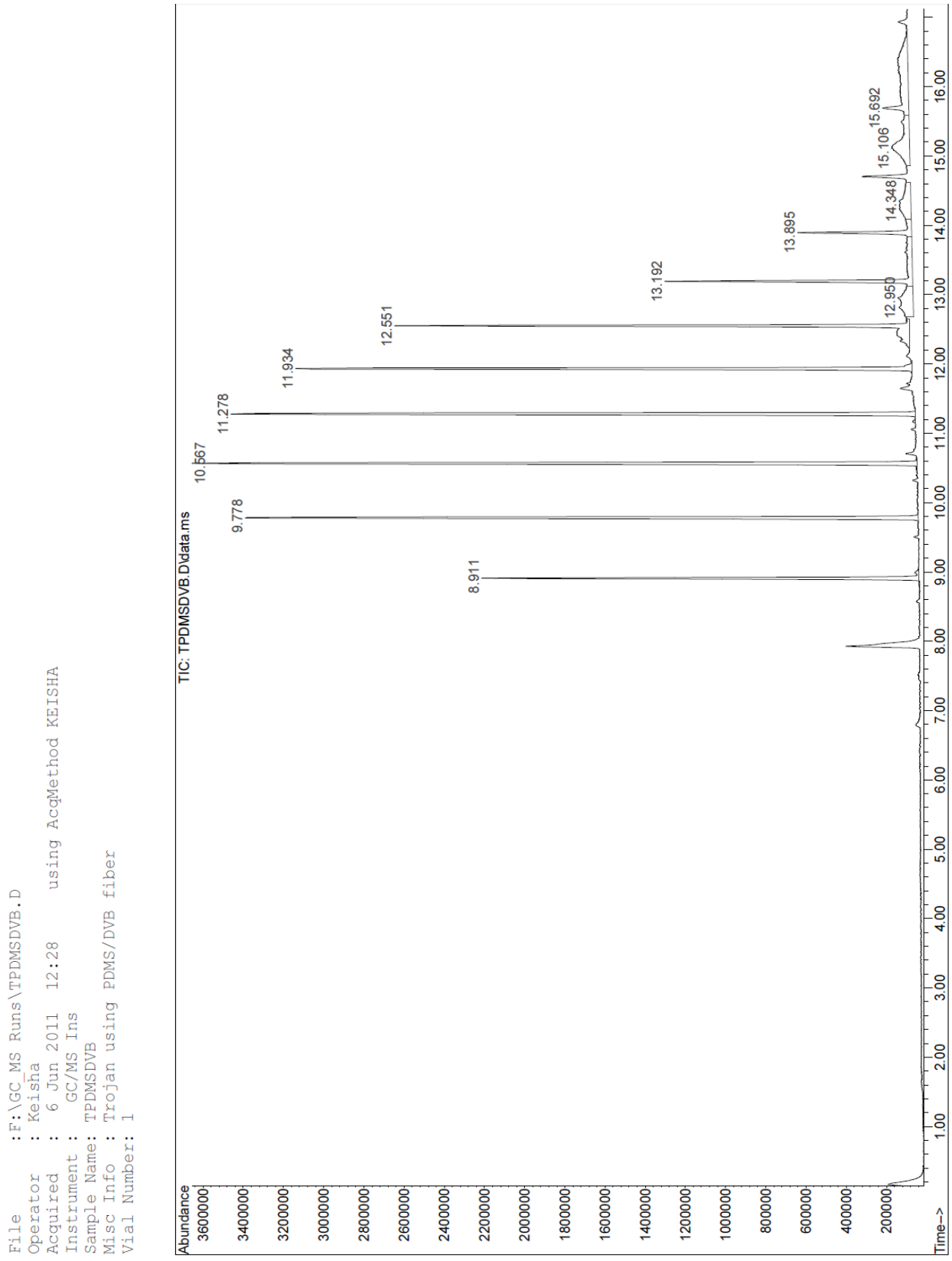


Figure G1. TPDMSDVB TIC

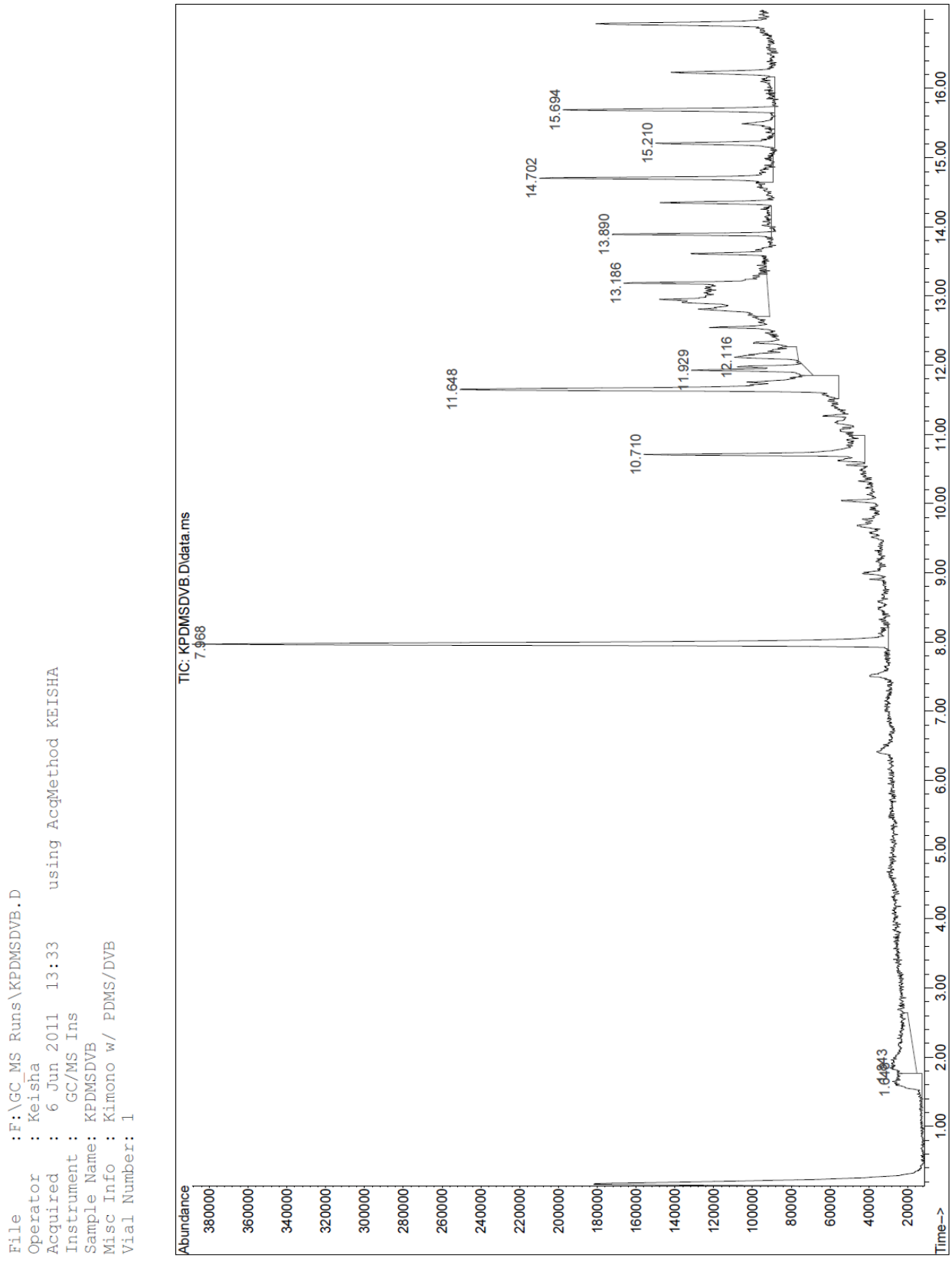


Figure G2. KPDMSDVB TIC

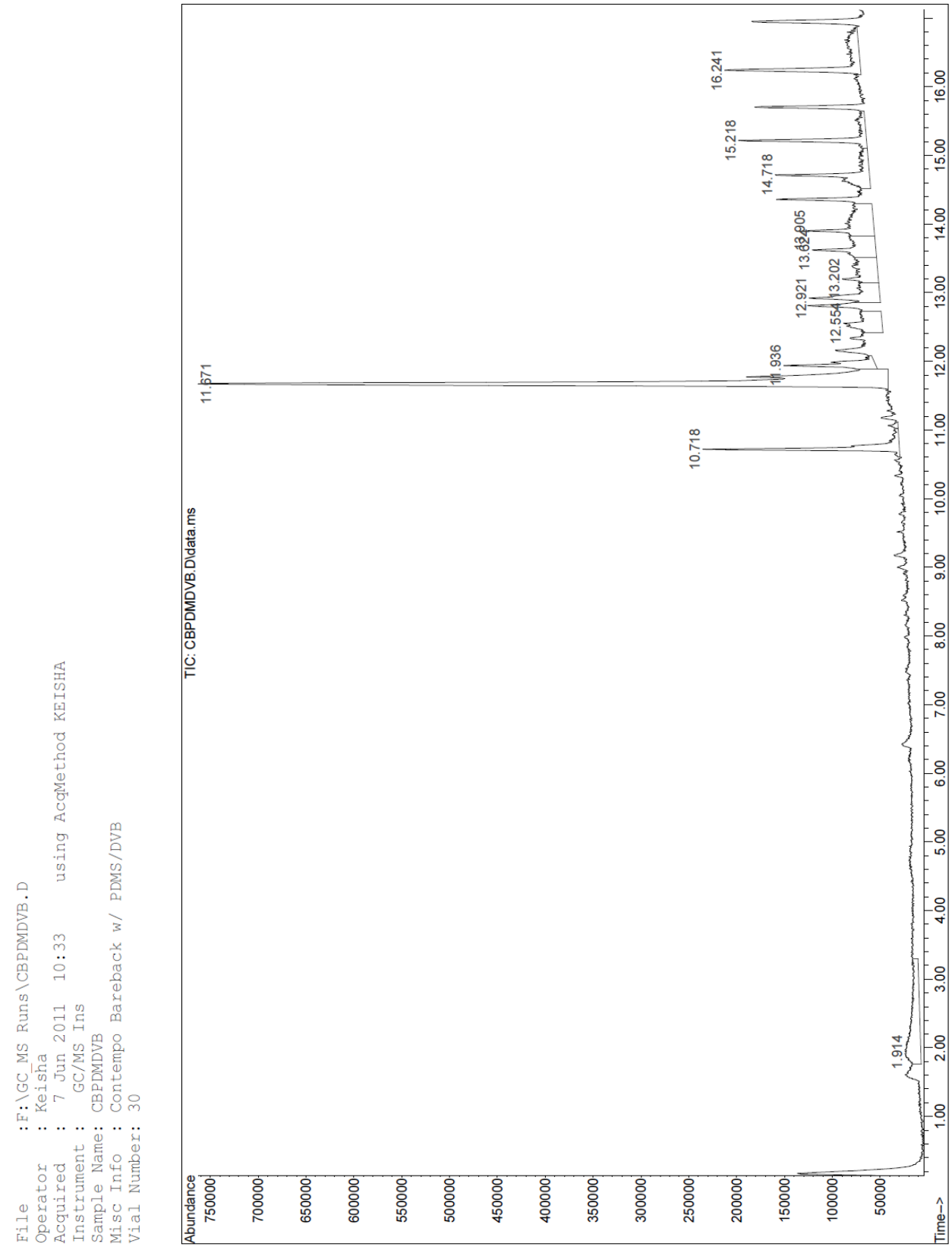


Figure G3. CBPDMDVB TIC

# Appendix H

## Chromatograms & Mass Spectra for PAC SPME Fiber

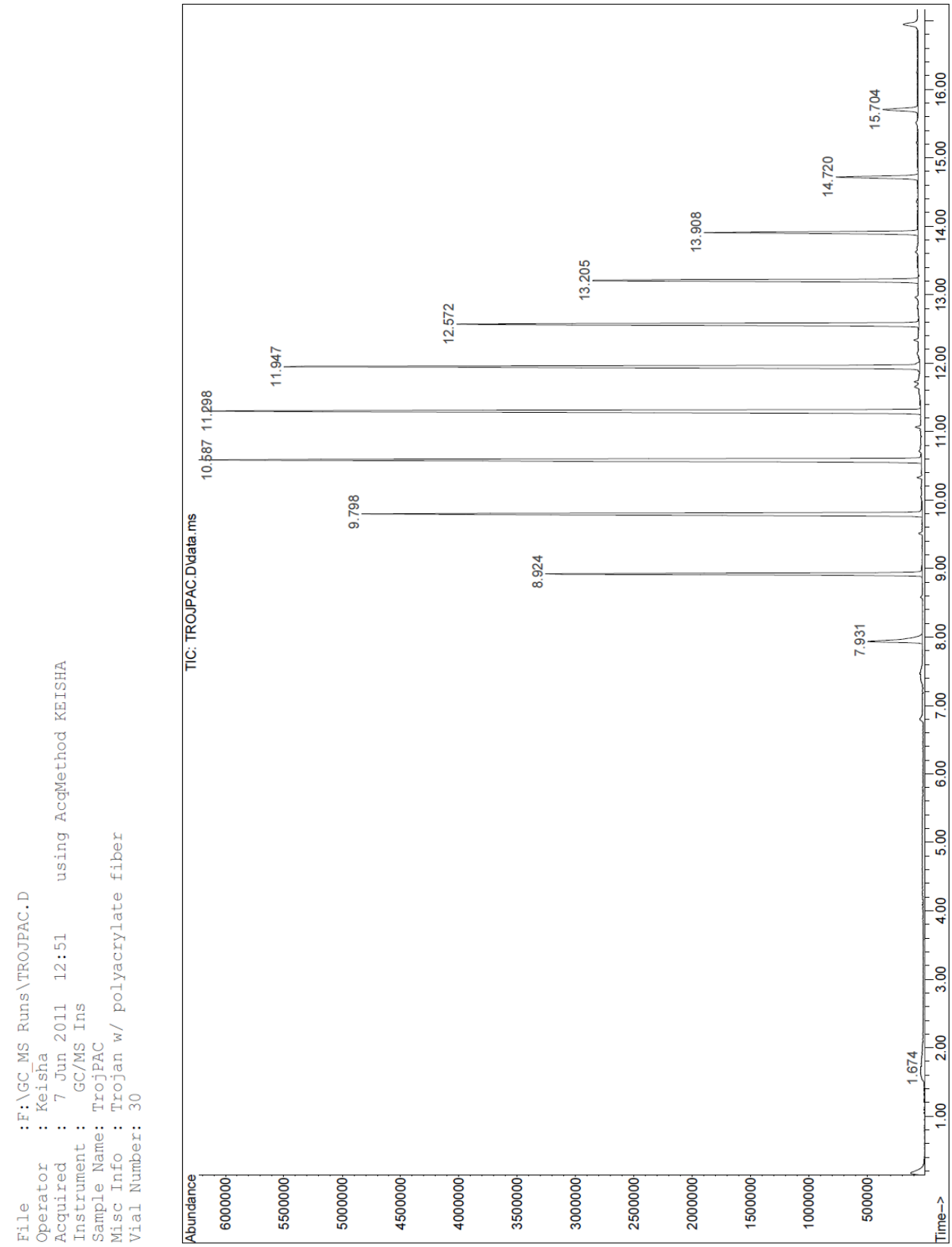


Figure H1. TROJPAC TIC

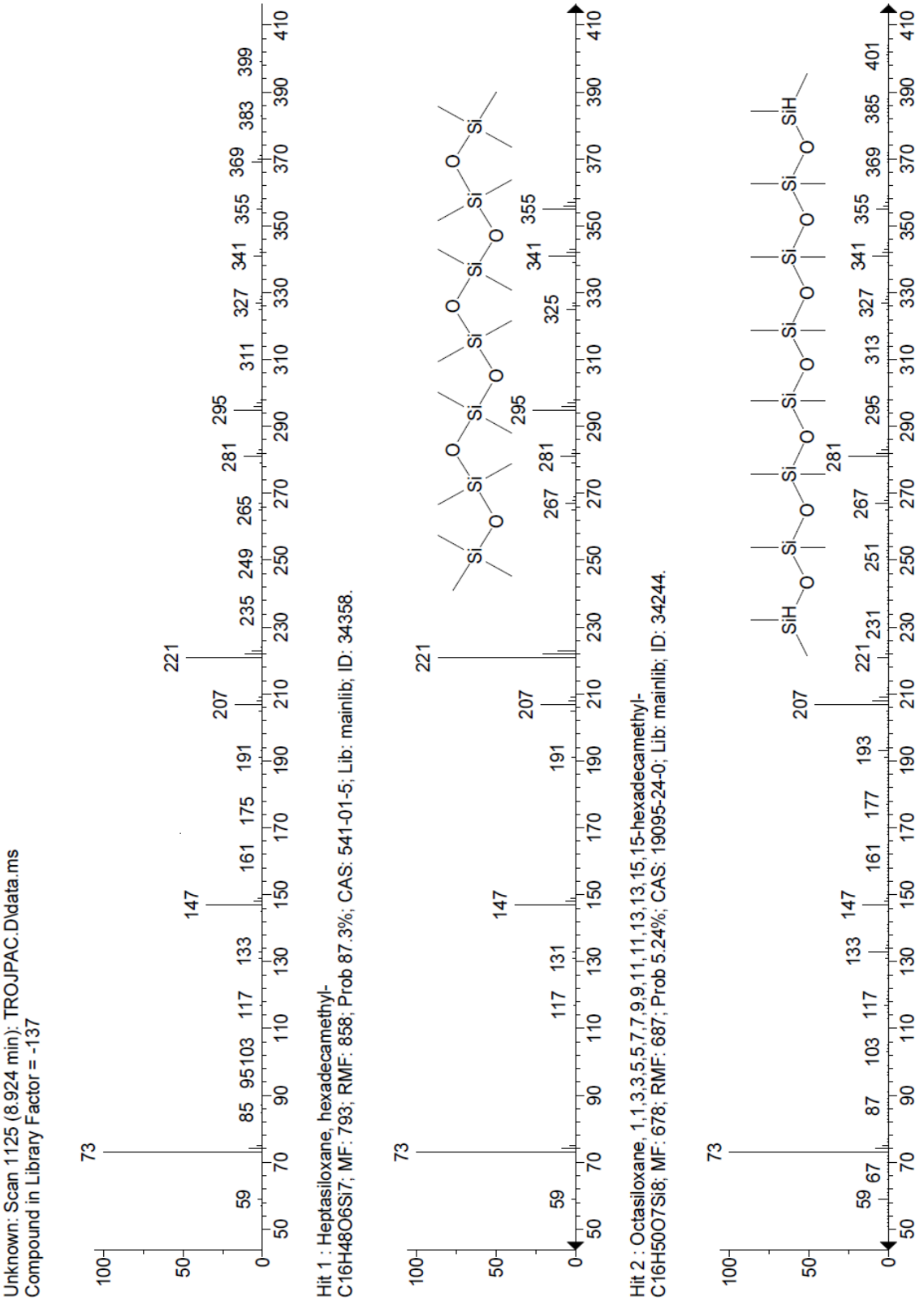


Figure H2. TROJPAC RT 8.924 min MS

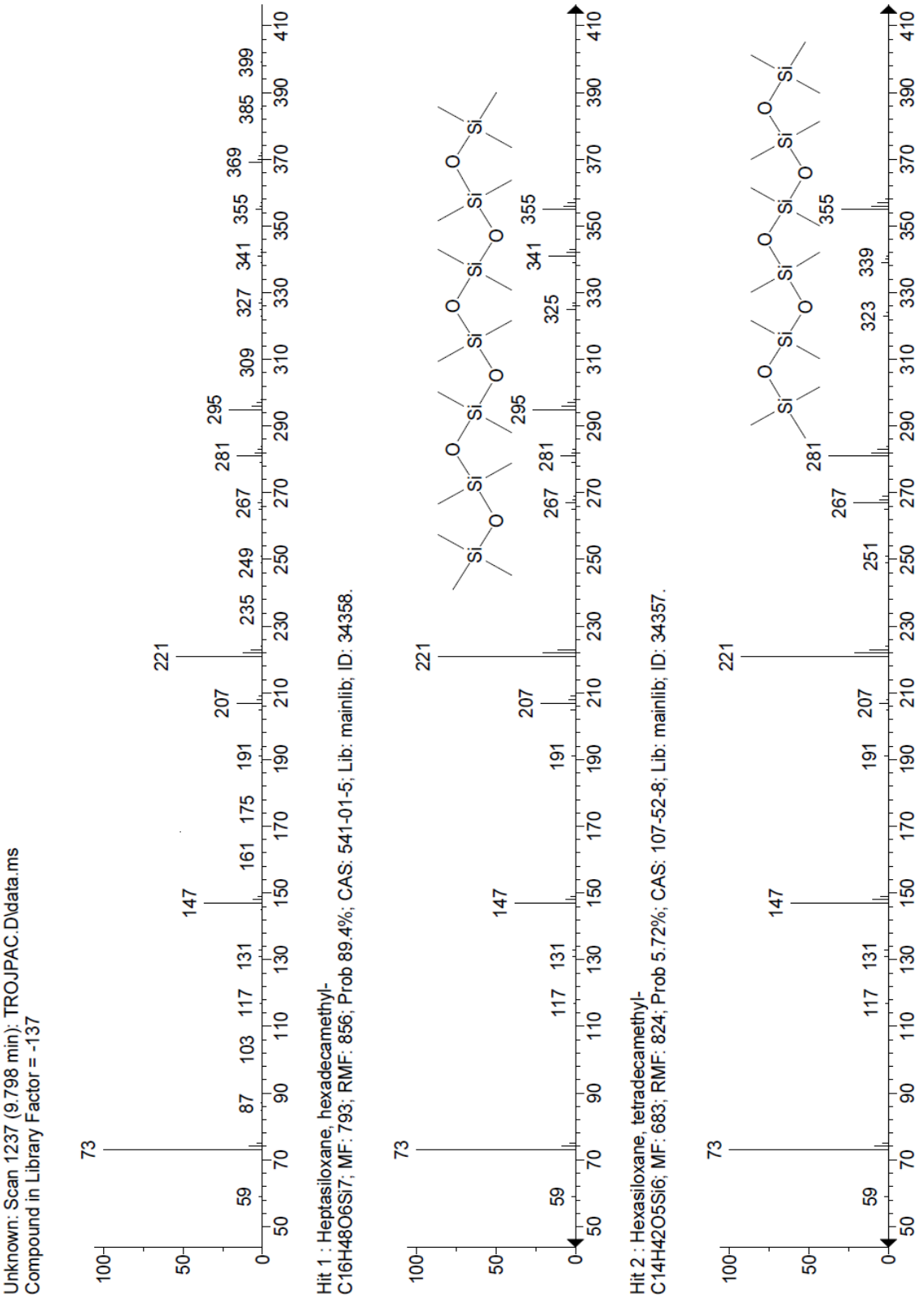


Figure H3. TROJPAC RT 9.798 min MS

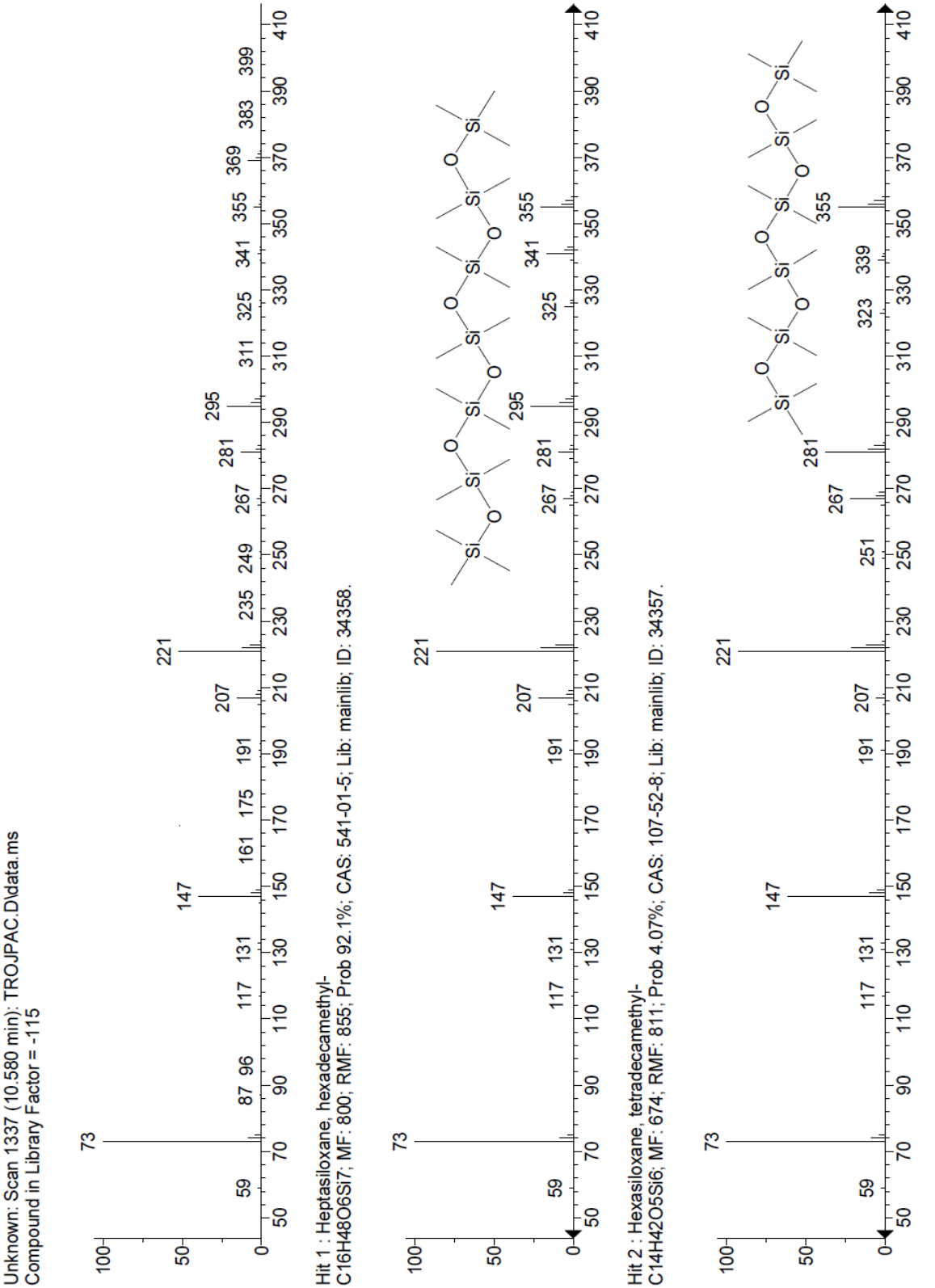


Figure H4. TROJPAC RT 10.580 min MS

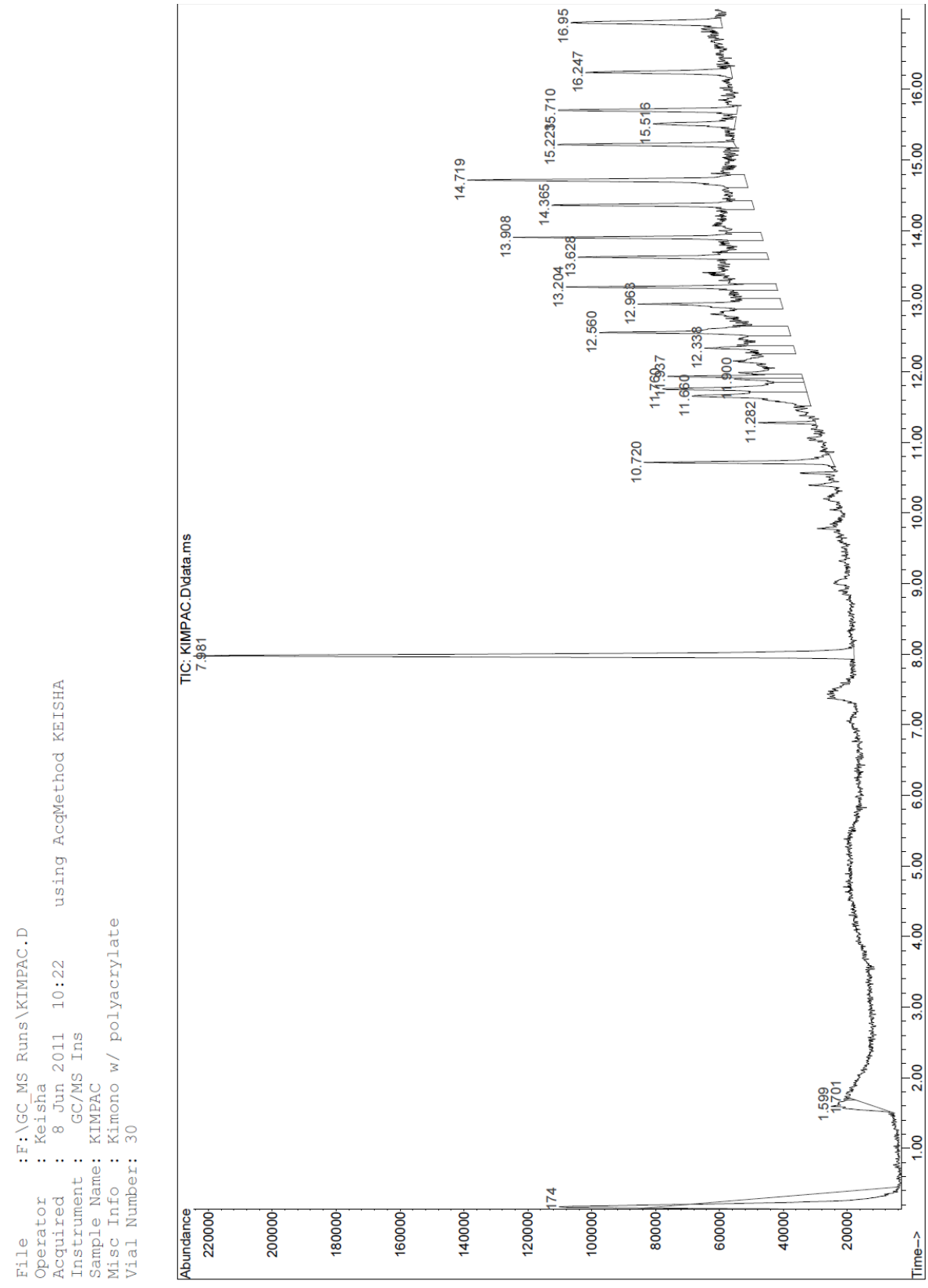


Figure H5. KIMPAC TIC

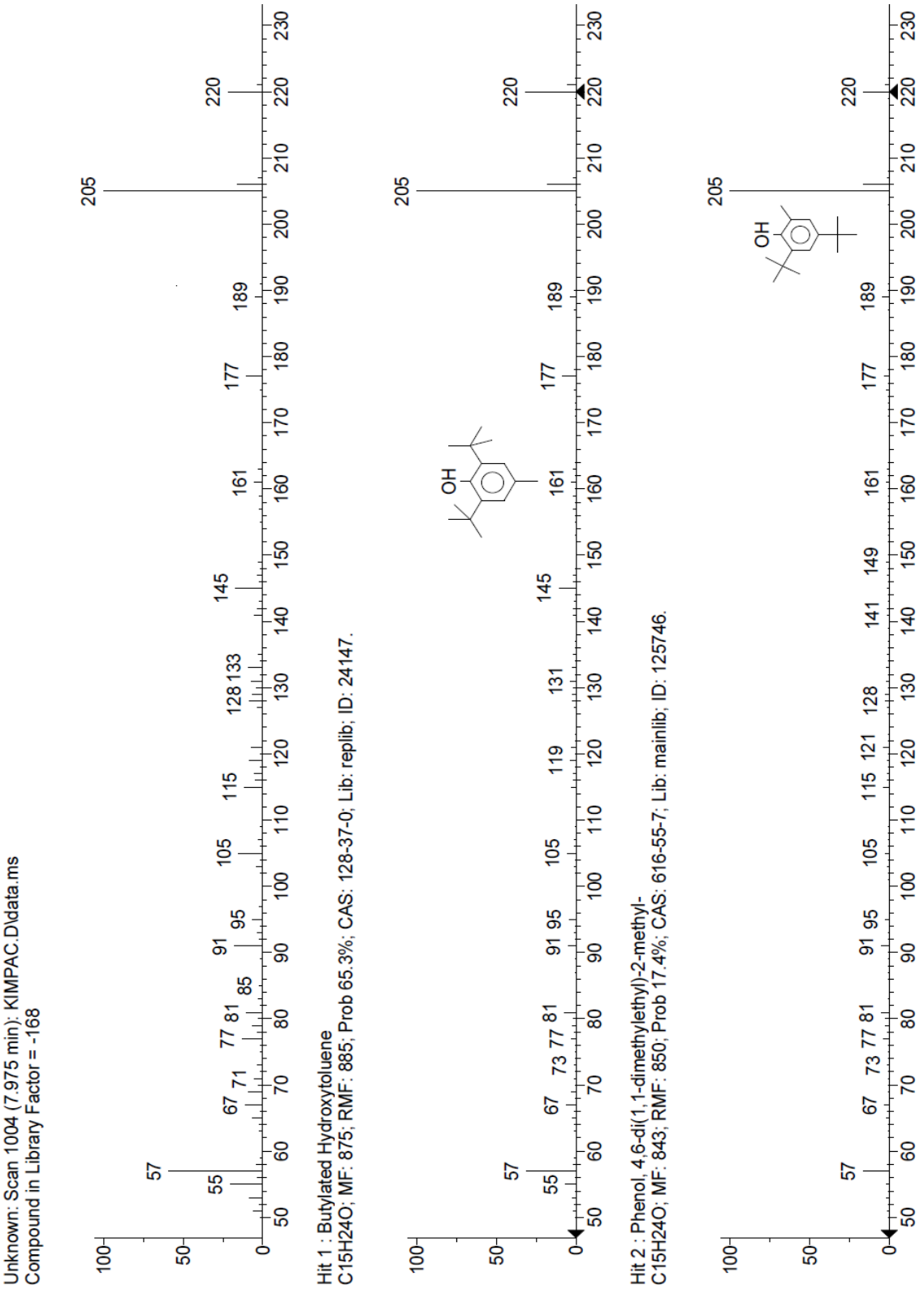


Figure H6. KIMPAC RT 7.975 min MS

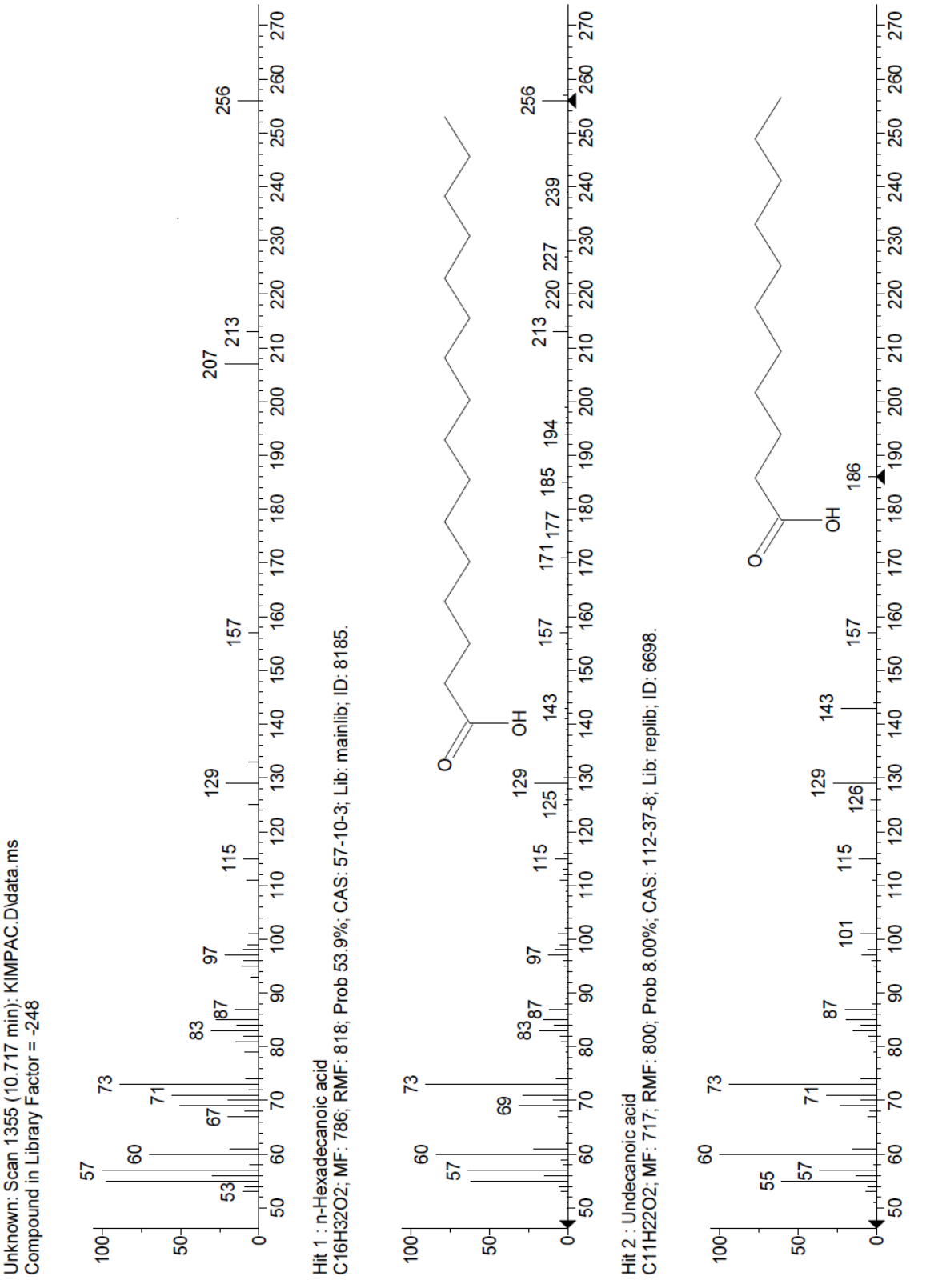


Figure H7. KIMPAC RT 10.717 min MS

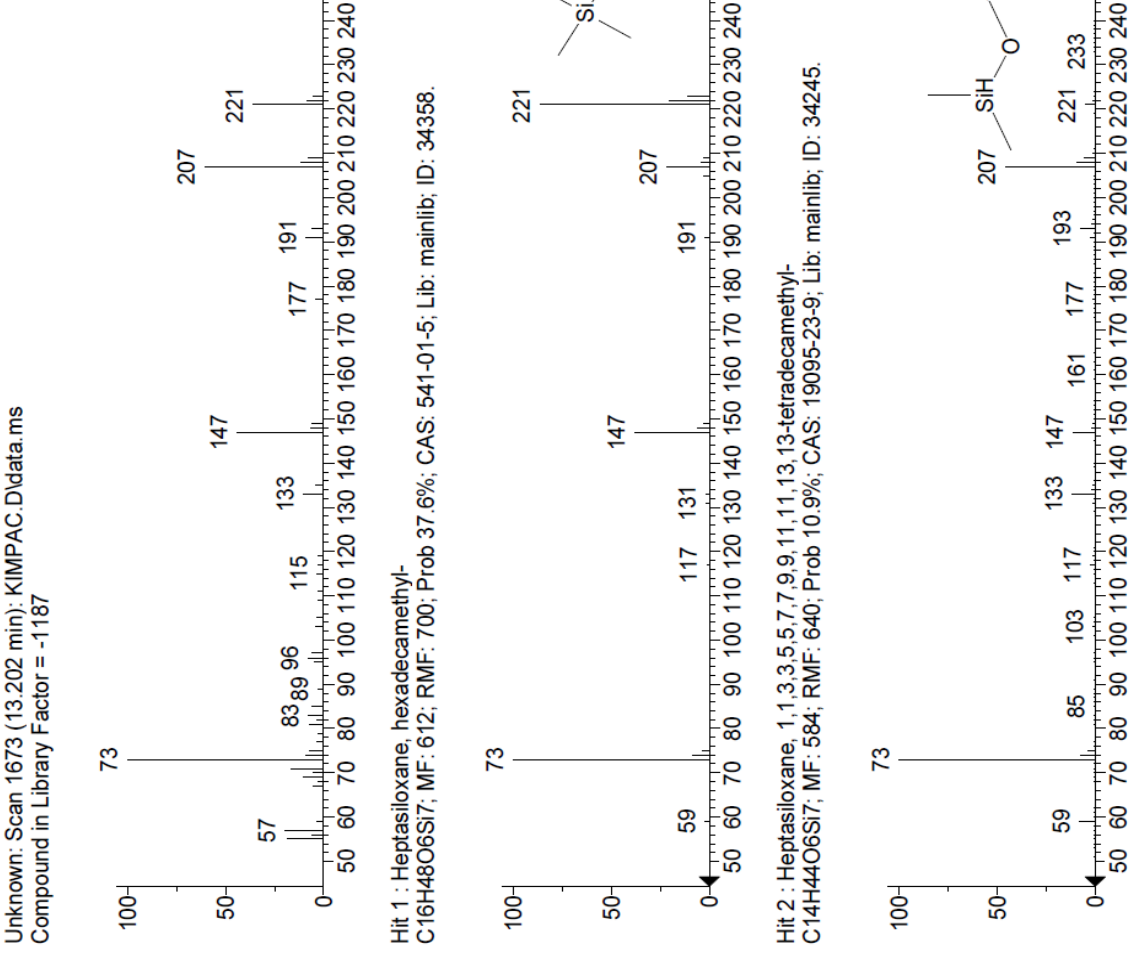


Figure H8. KIMPAC RT 13.202 min MS

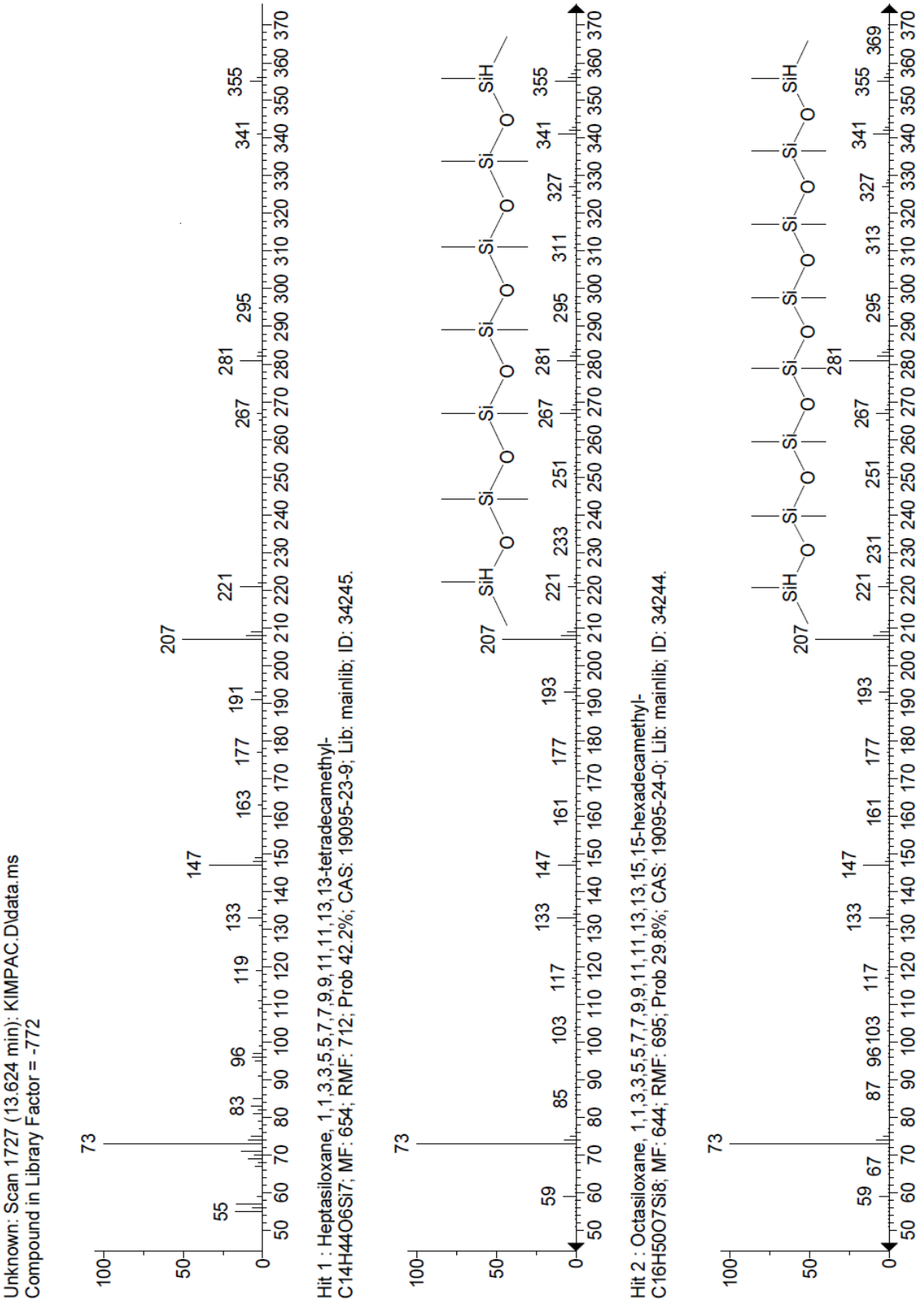


Figure H9. KIMPAC RT 13.624 min MS

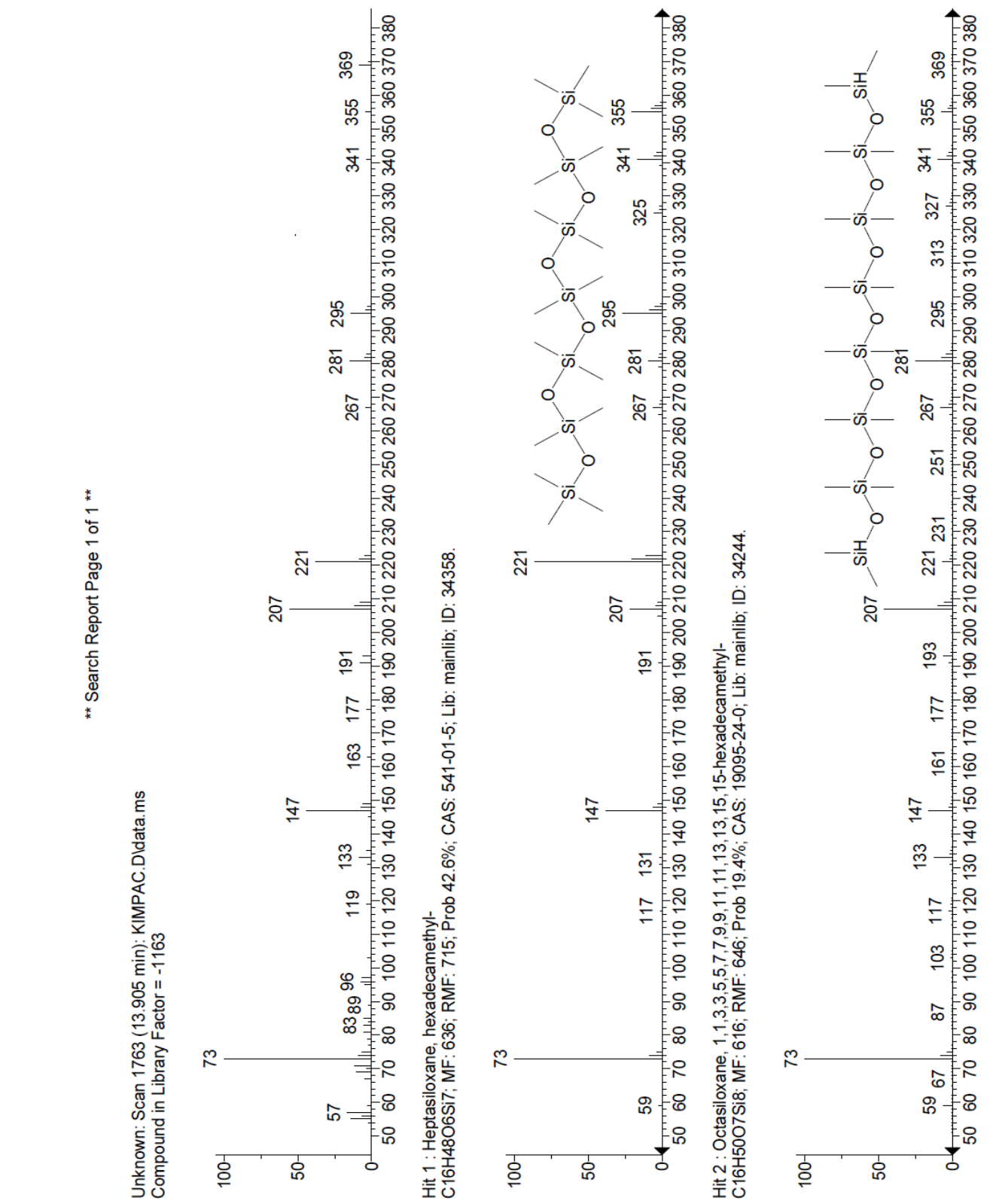


Figure H10. KIMPAC RT 13.905 min MS

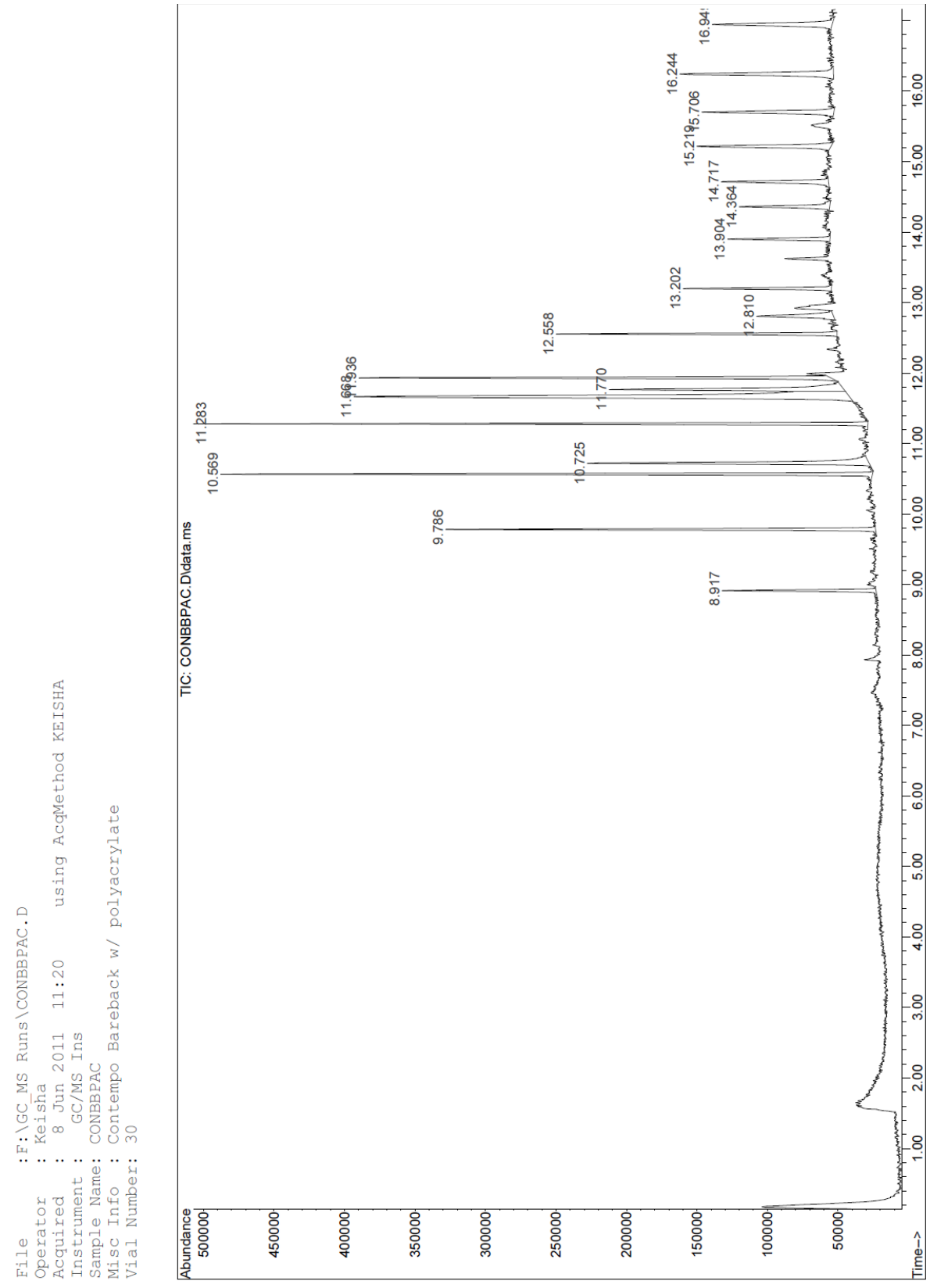


Figure H11. CONBBPAC TIC

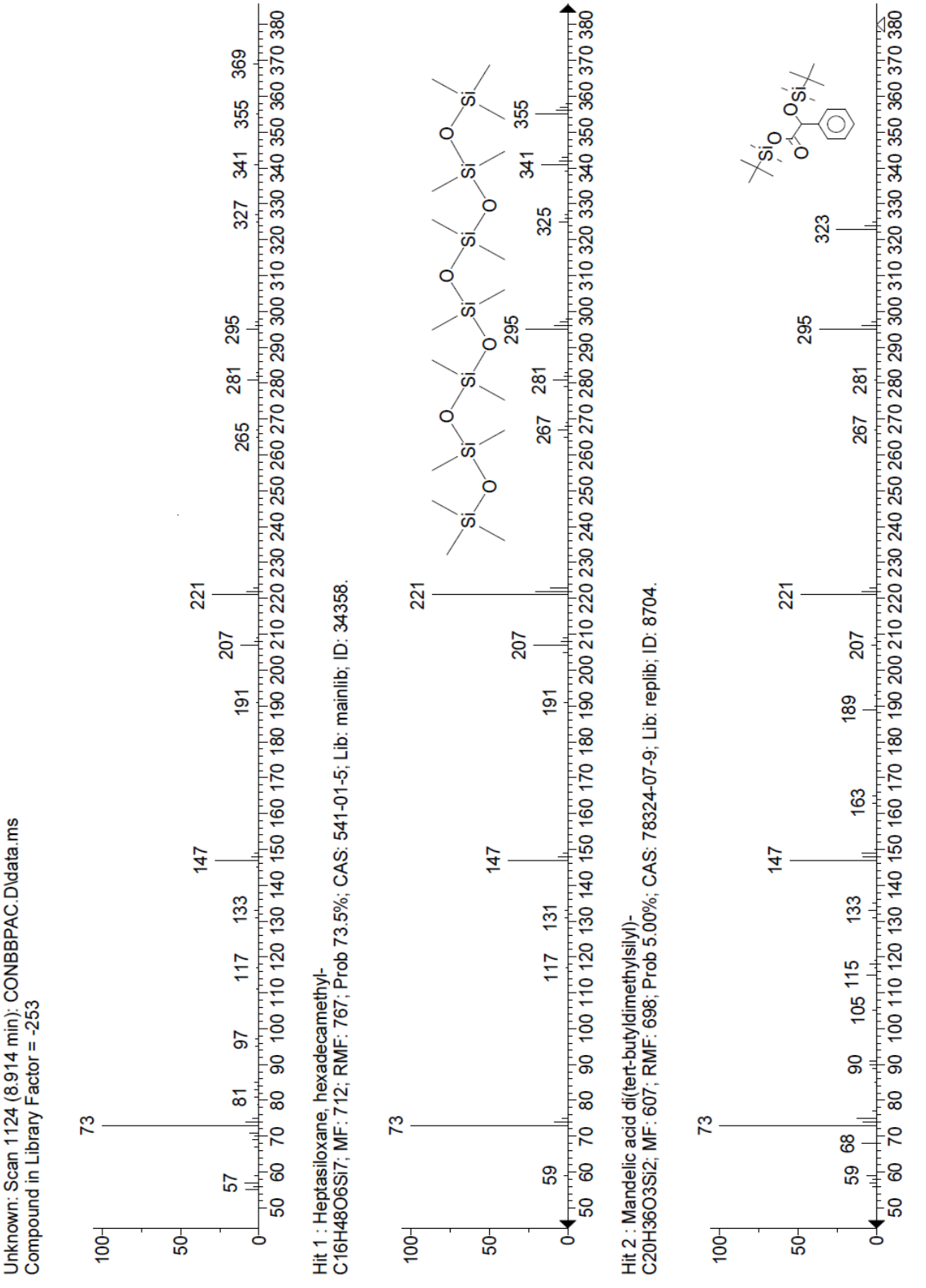


Figure H12. CONBBPAC RT 8.914 min MS

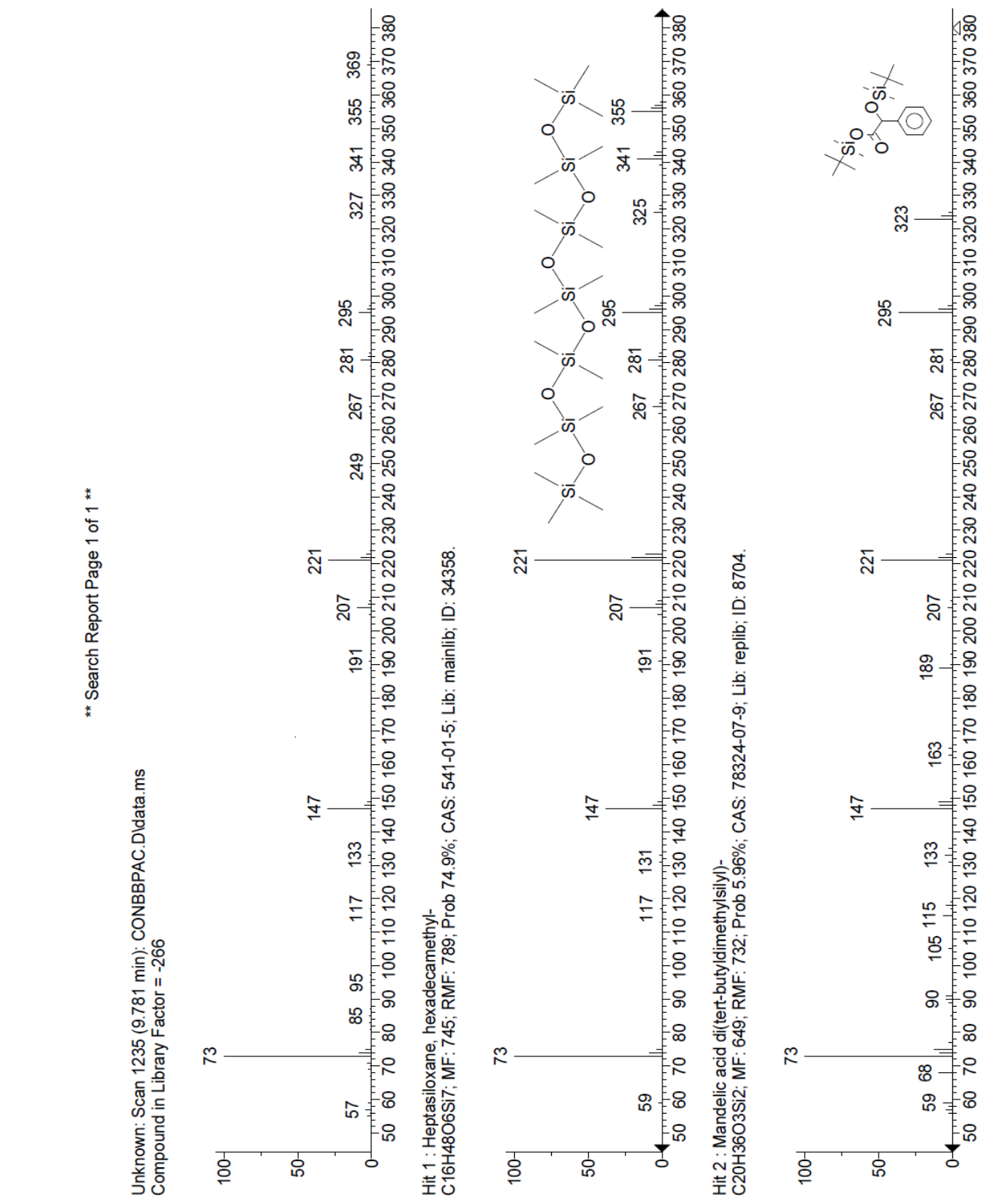


Figure H13. CONBBPAC RT 9.781 min MS

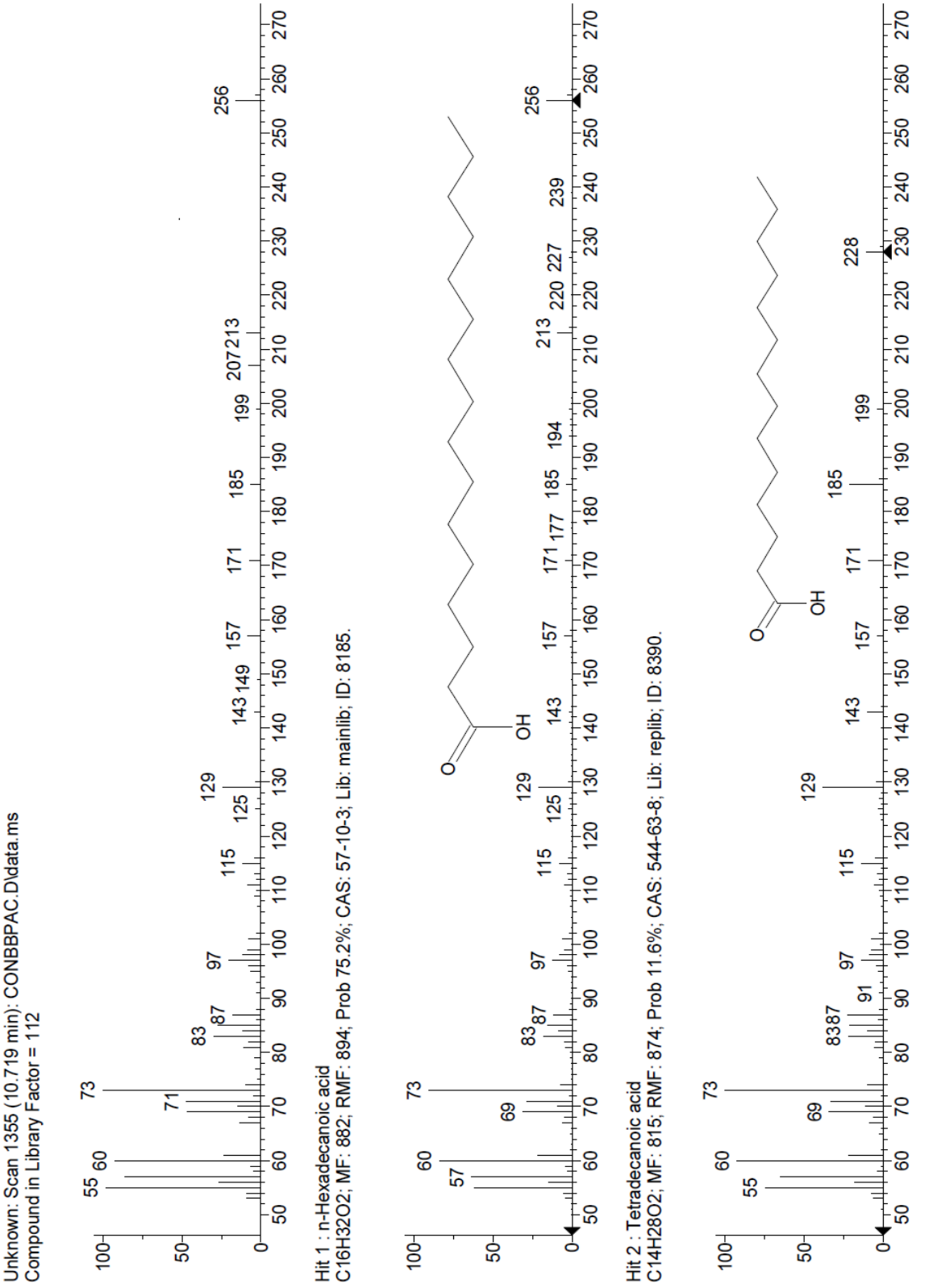


Figure H14. CONBBPAC RT 10.719 min MS

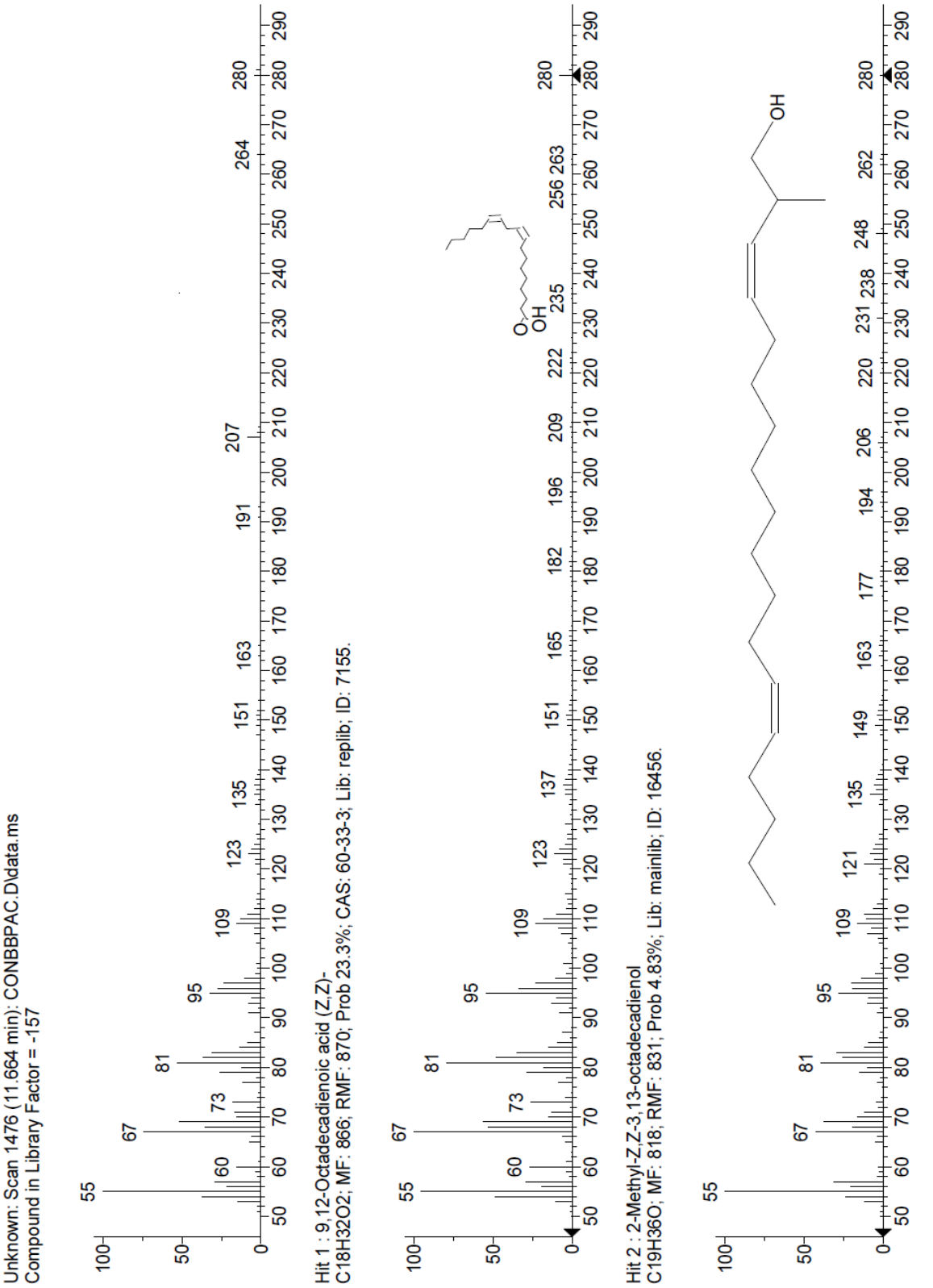


Figure H15. CONBBPAC RT 11.664 min MS

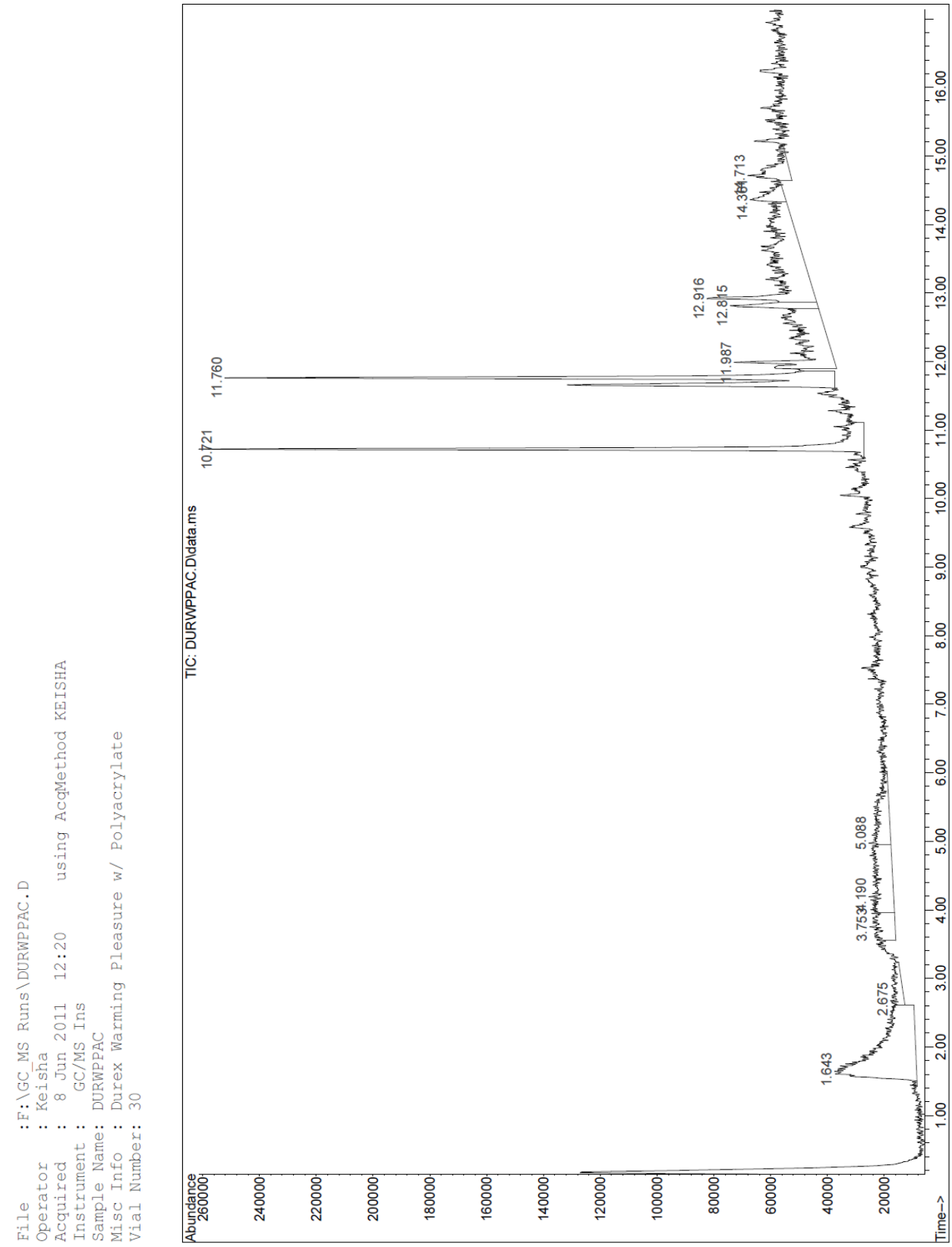


Figure H16. DURWPPAC TIC

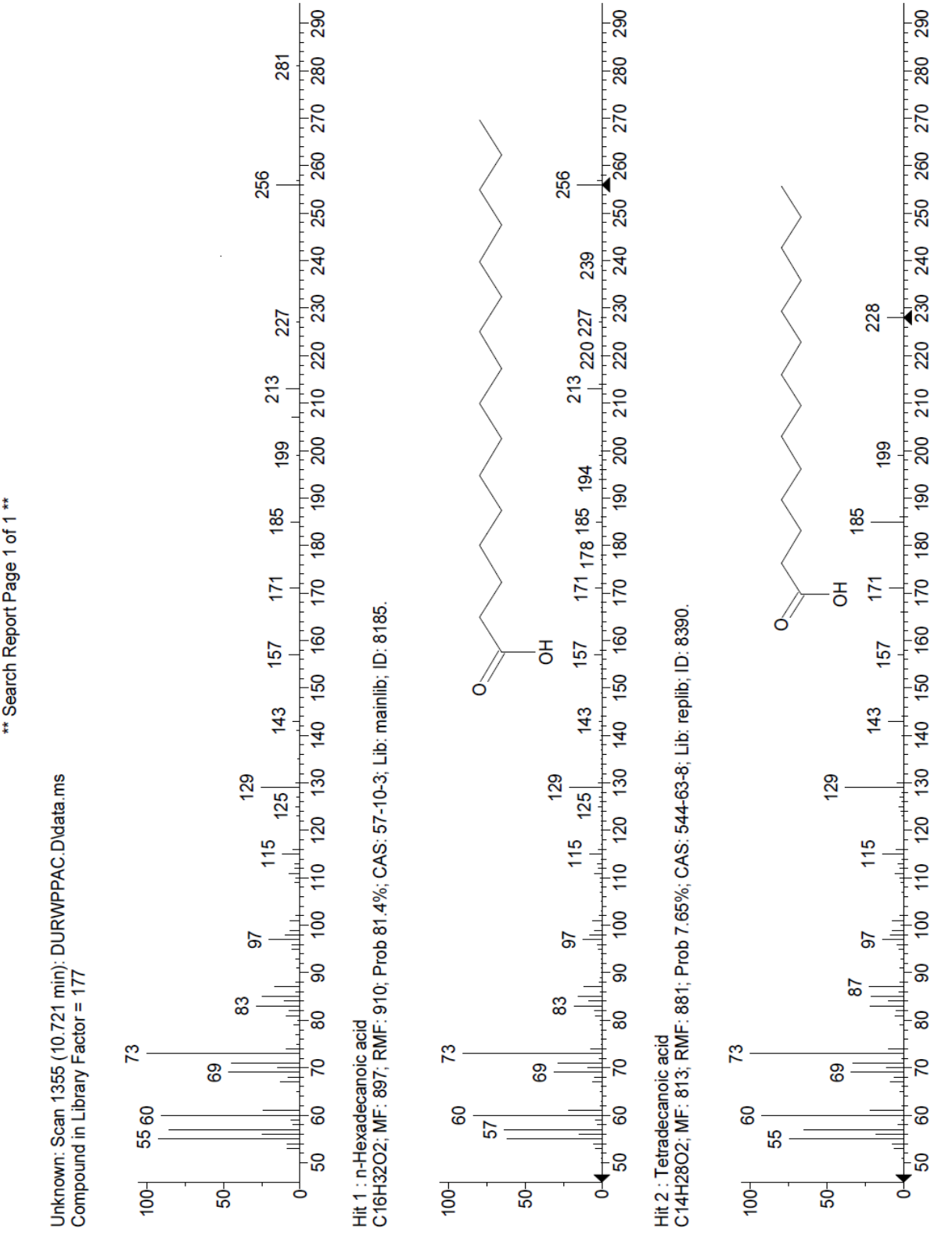


Figure H17. DURWPPAC RT 10.721 min MS

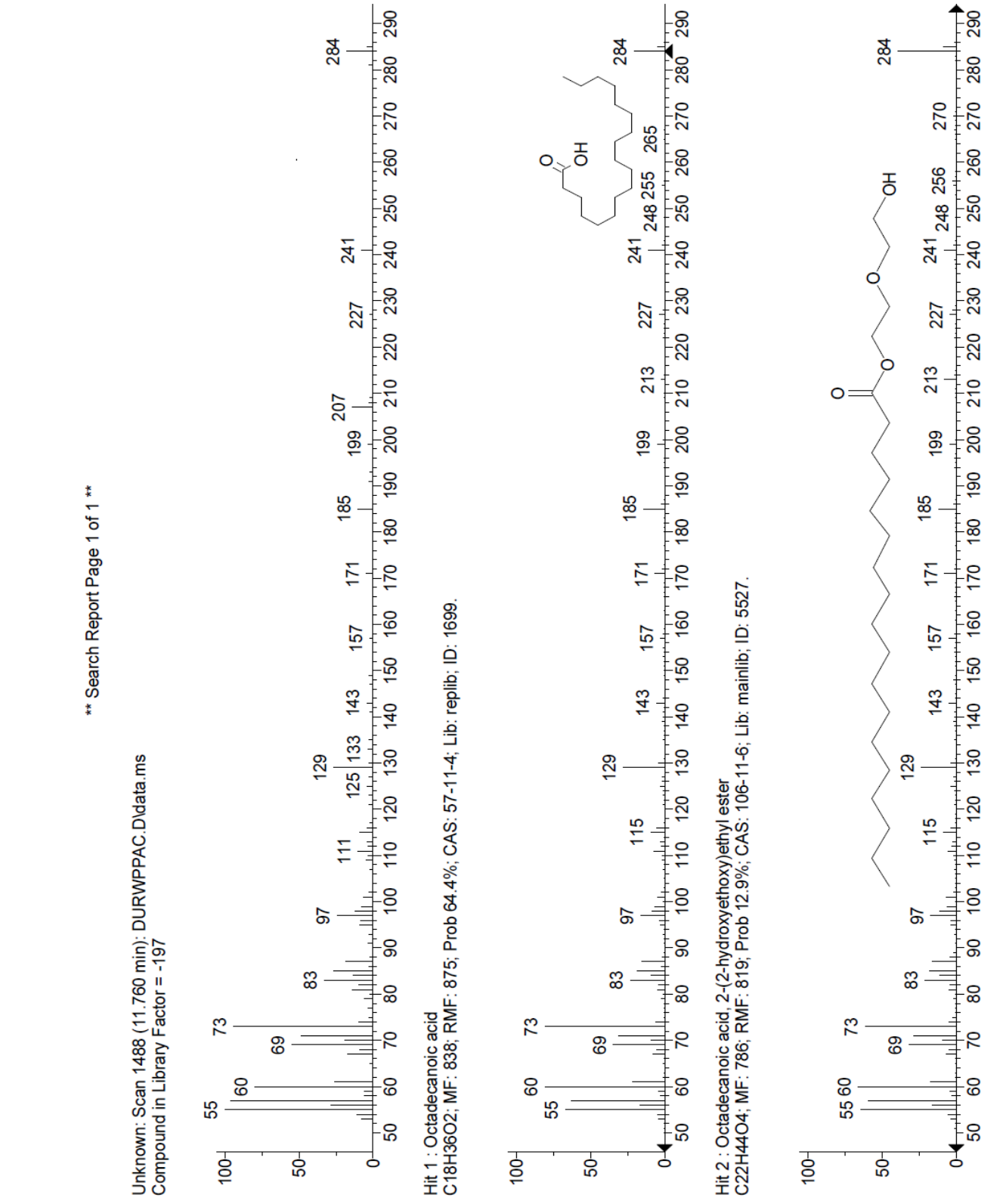


Figure H18. DURWPPAC RT 11.760 min MS

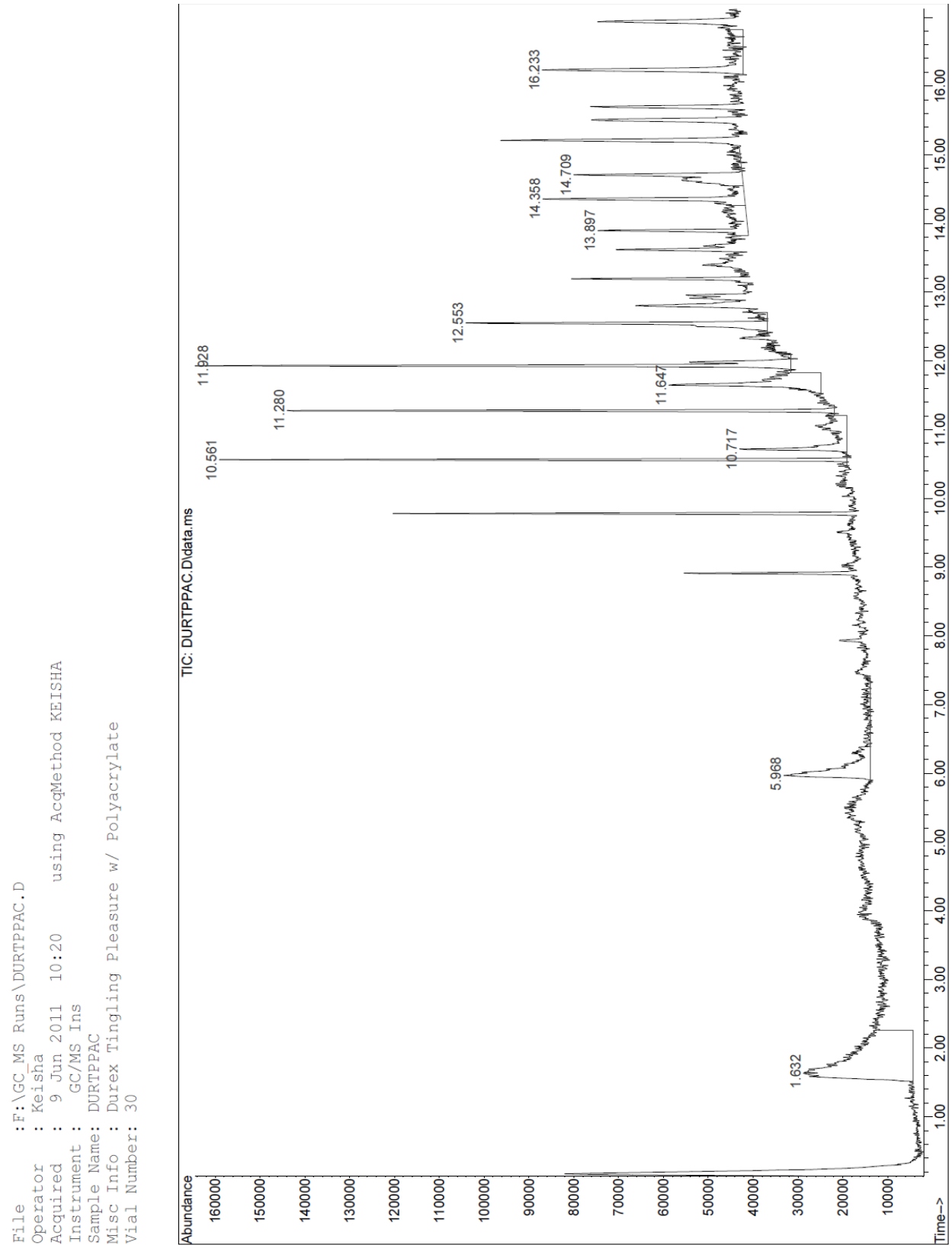


Figure H19. DURTPPAC TIC

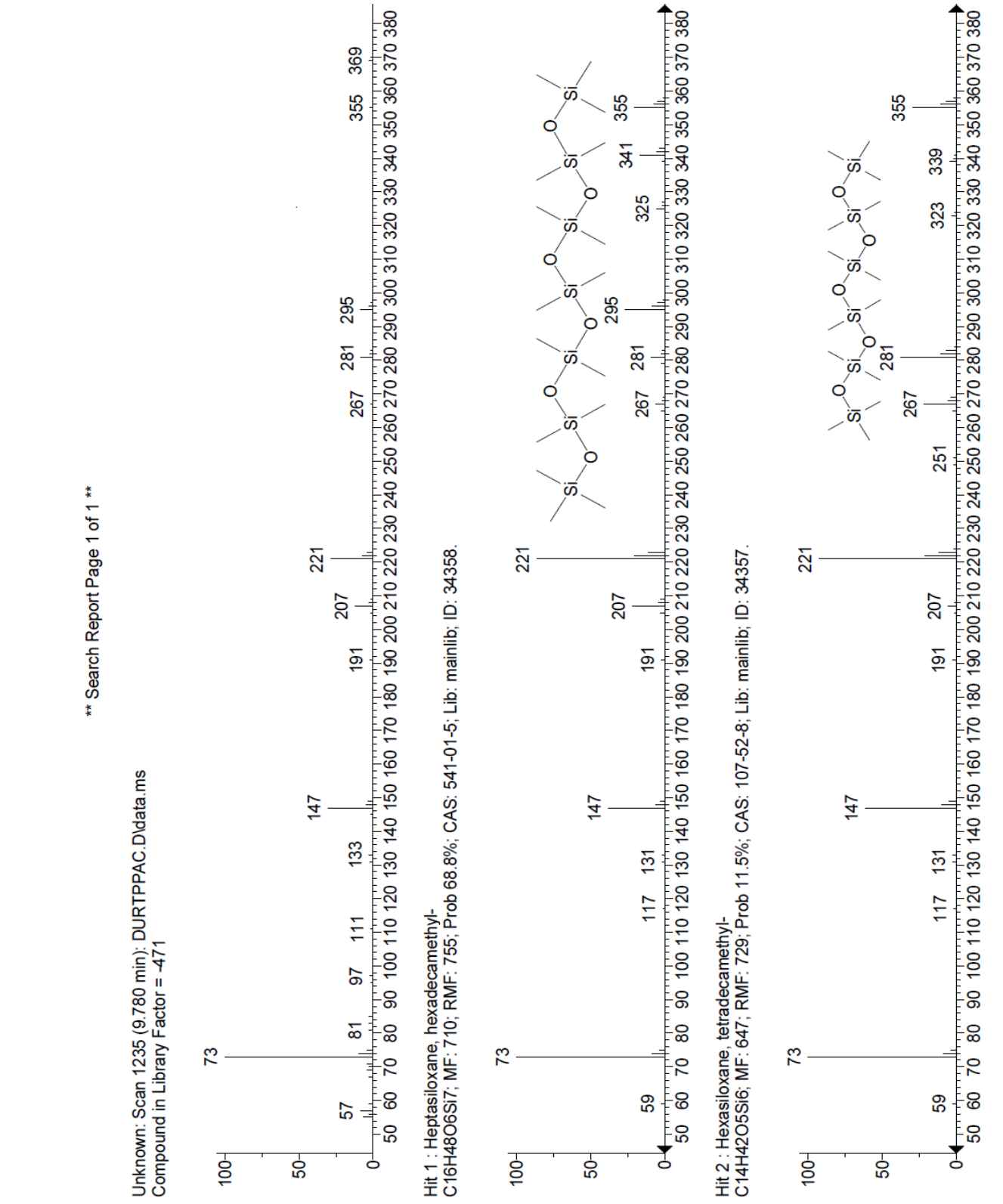


Figure H20. DURTPPAC RT 9.780 min MS

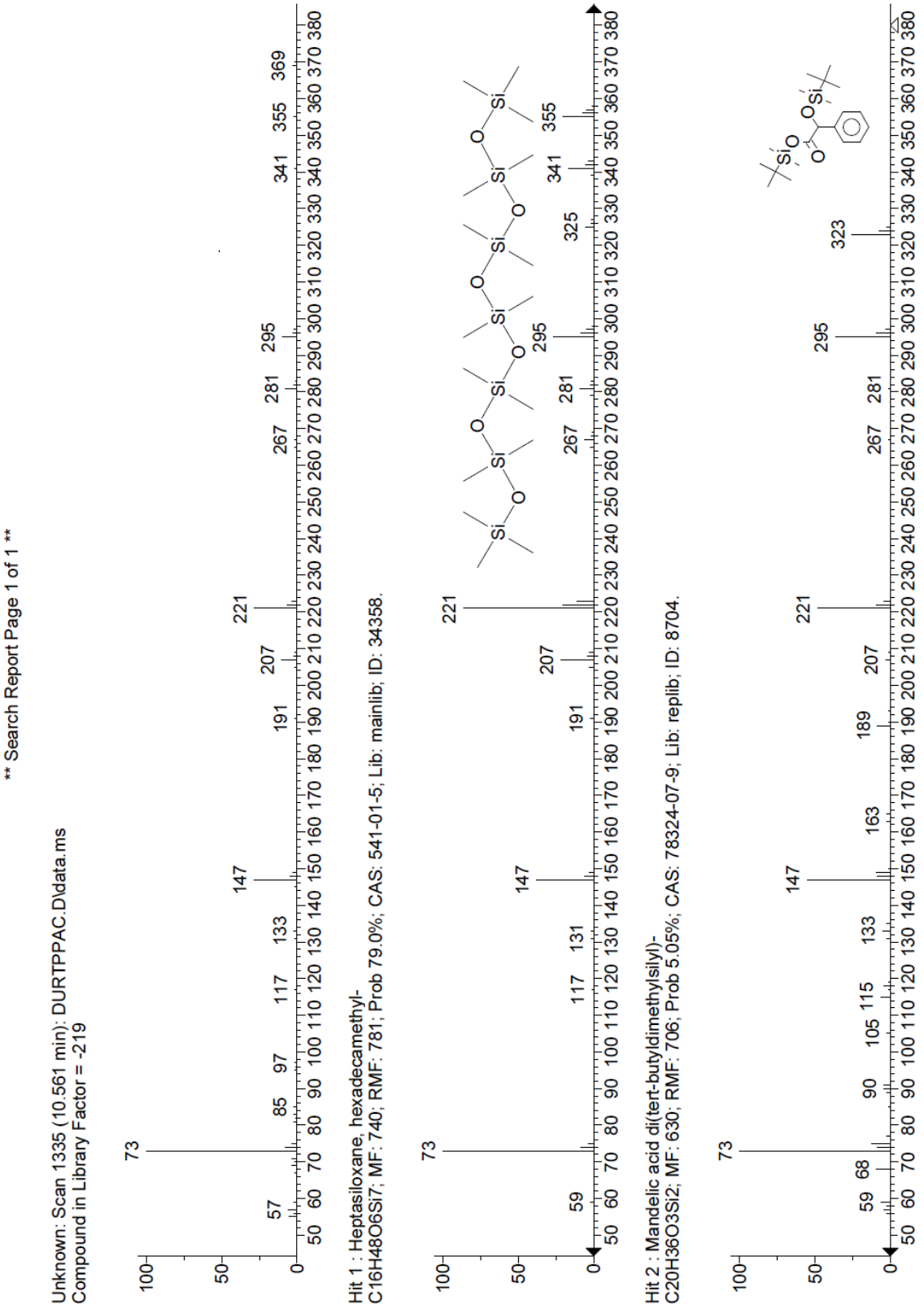


Figure H21. DURTPPAC RT 10.561 min MS

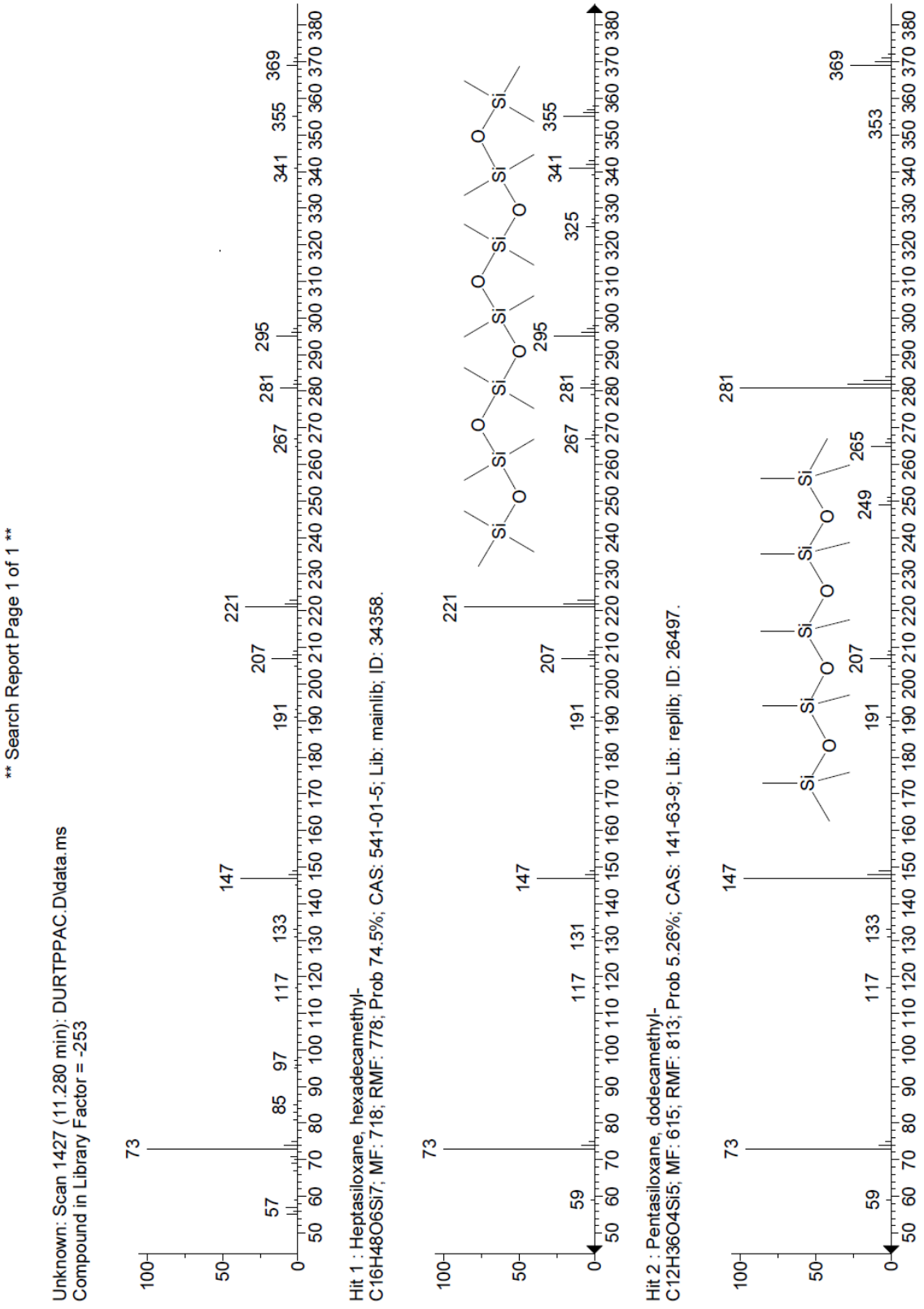


Figure H22. DURTPPAC RT 11.280 min MS

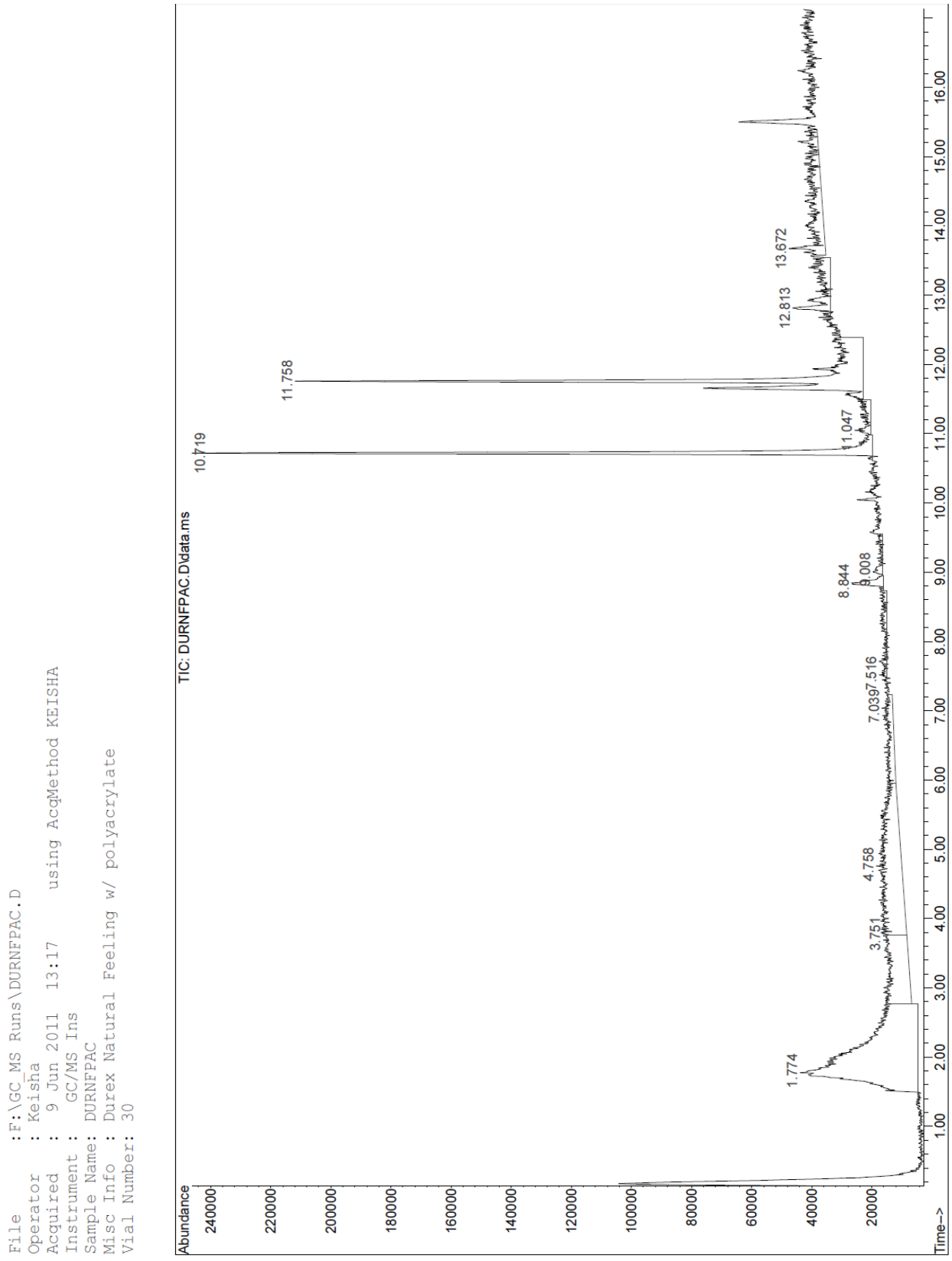


Figure H23. DURNFPAC TIC

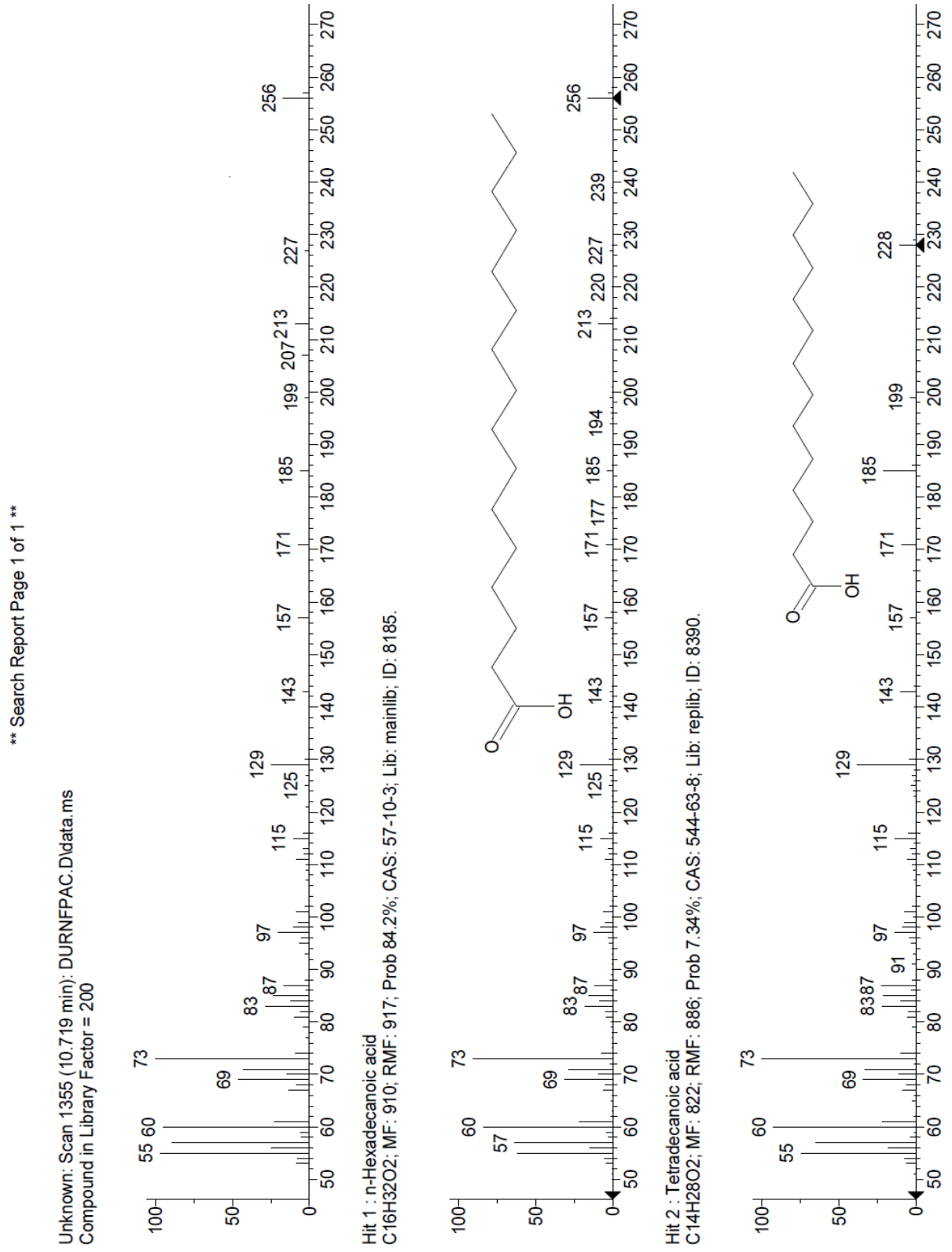


Figure H24. DURNFPAC RT 10.719 min MS

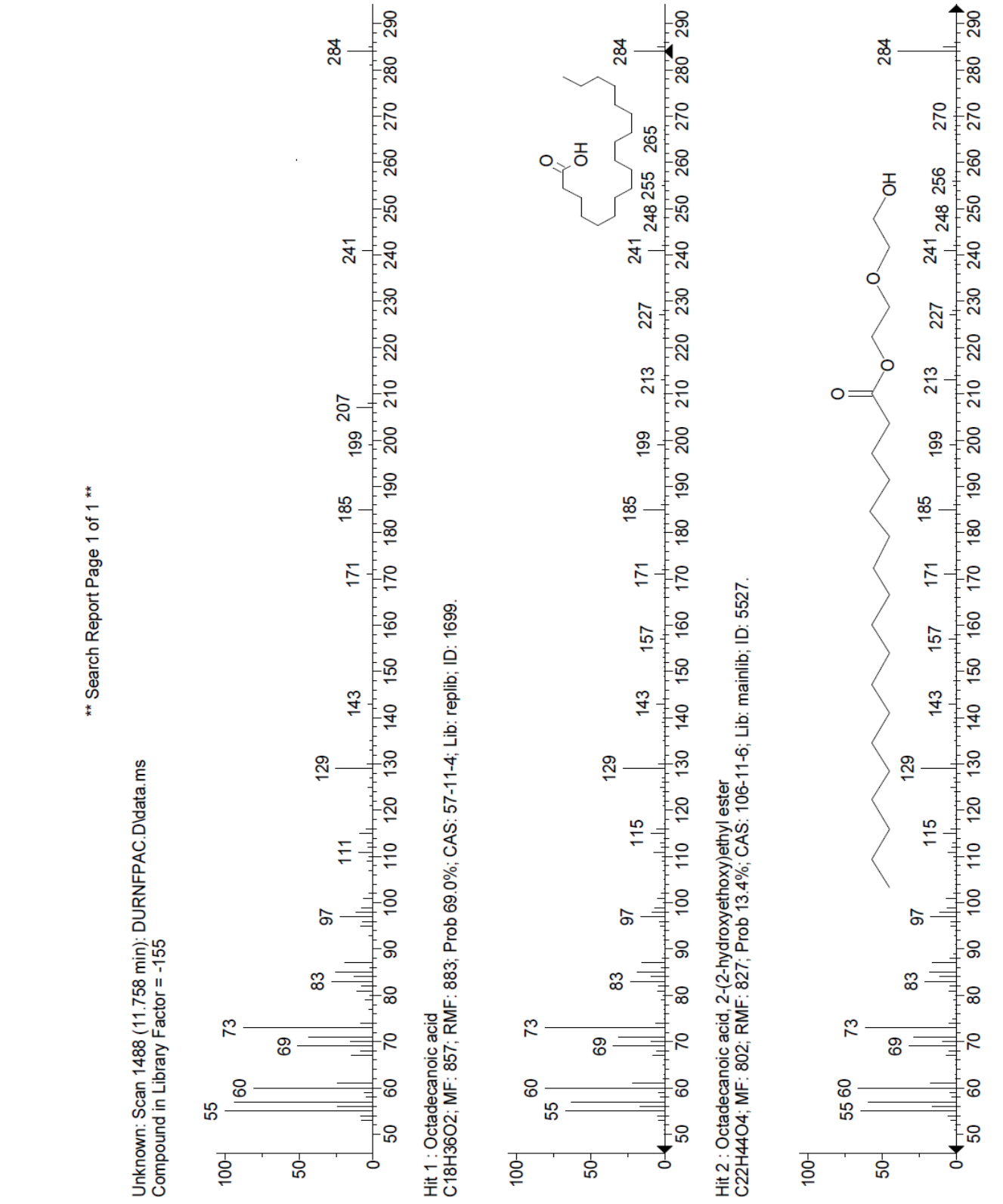


Figure H25. DURNFPAC RT 11.758 min MS

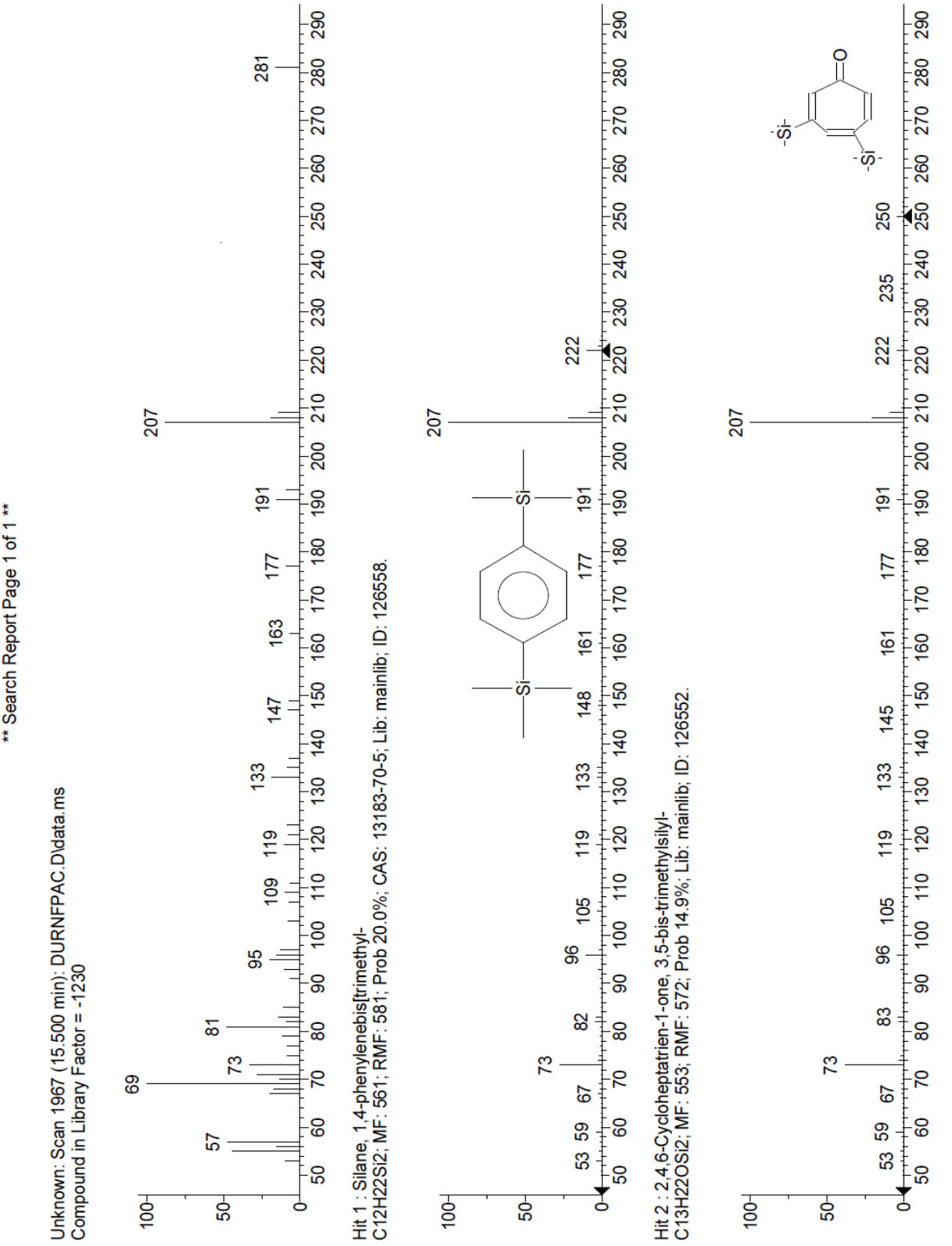


Figure H26. DURNFPAC RT 15.500 min MS

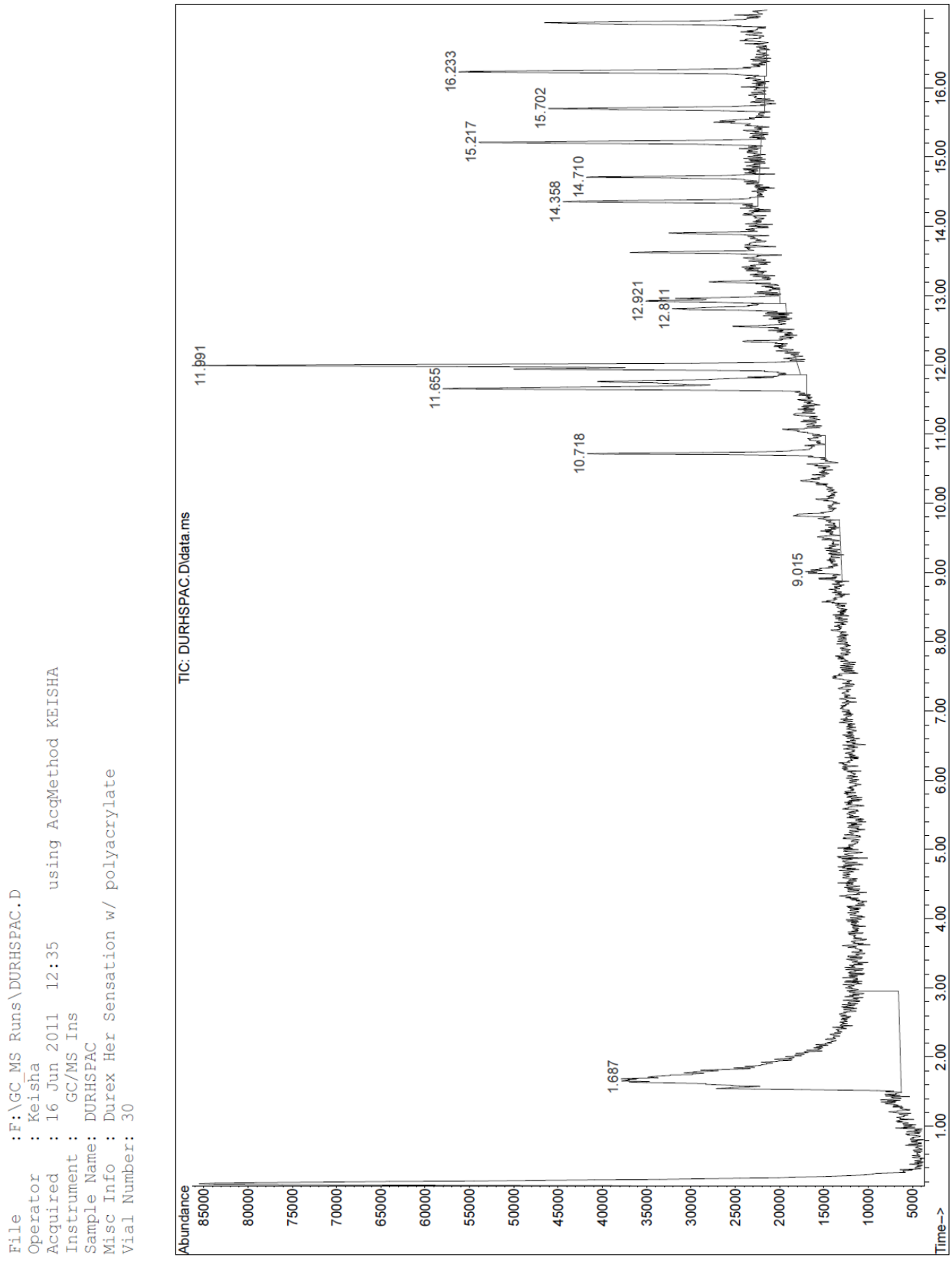


Figure H27. DURHSPAC TIC

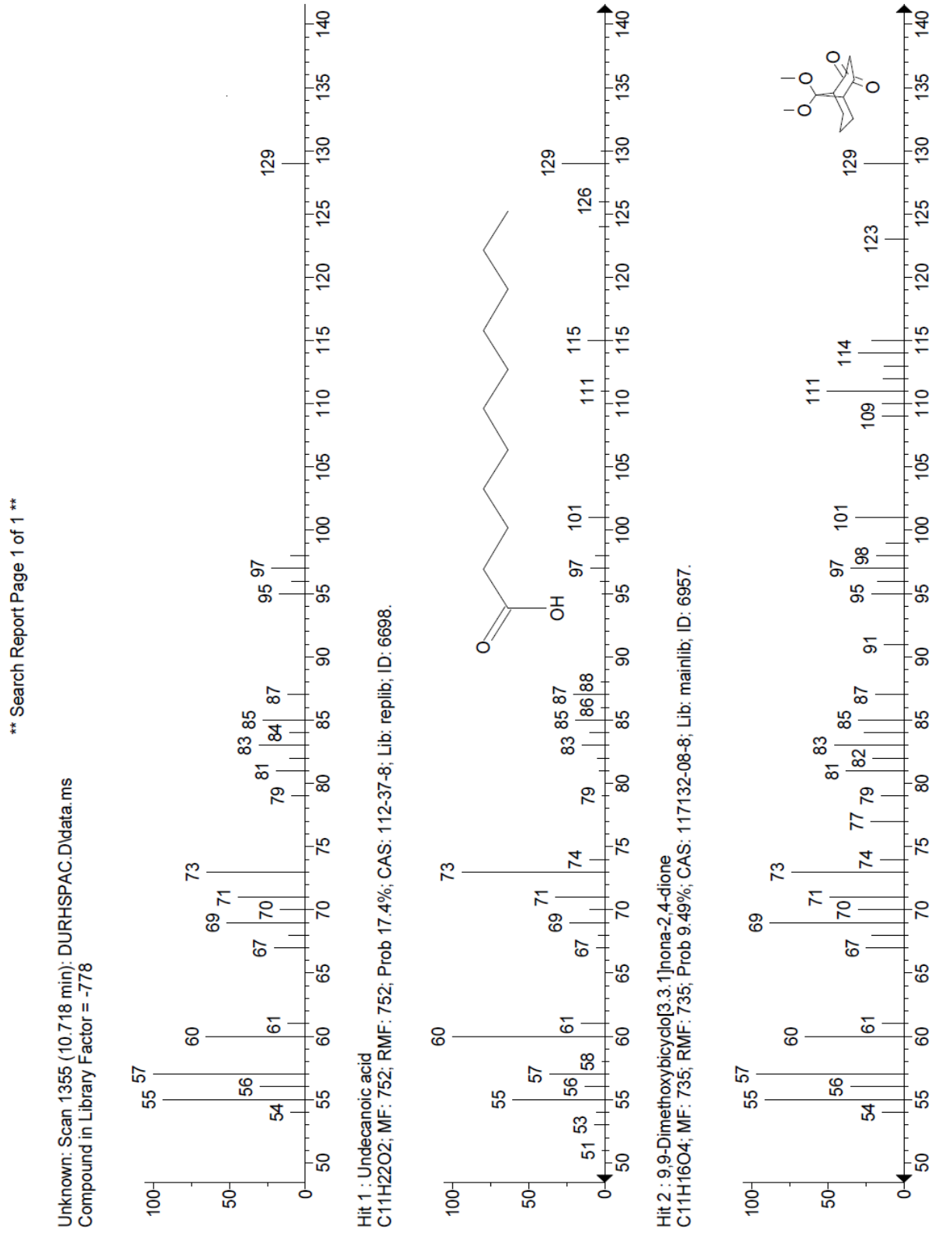


Figure H28. DURHSPAC RT 10.718 min MS

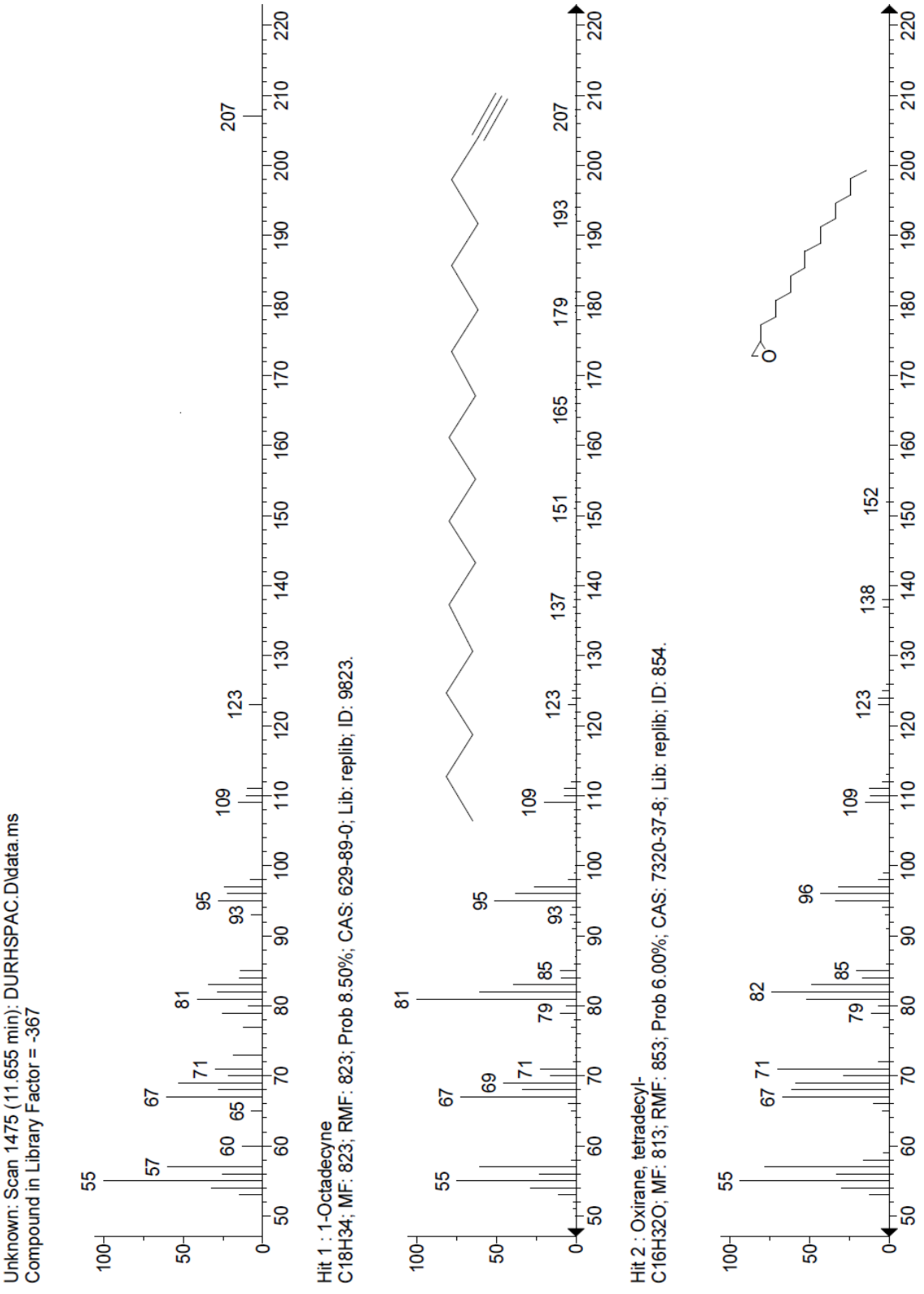


Figure H29. DURHSPAC RT 11.655 min MS

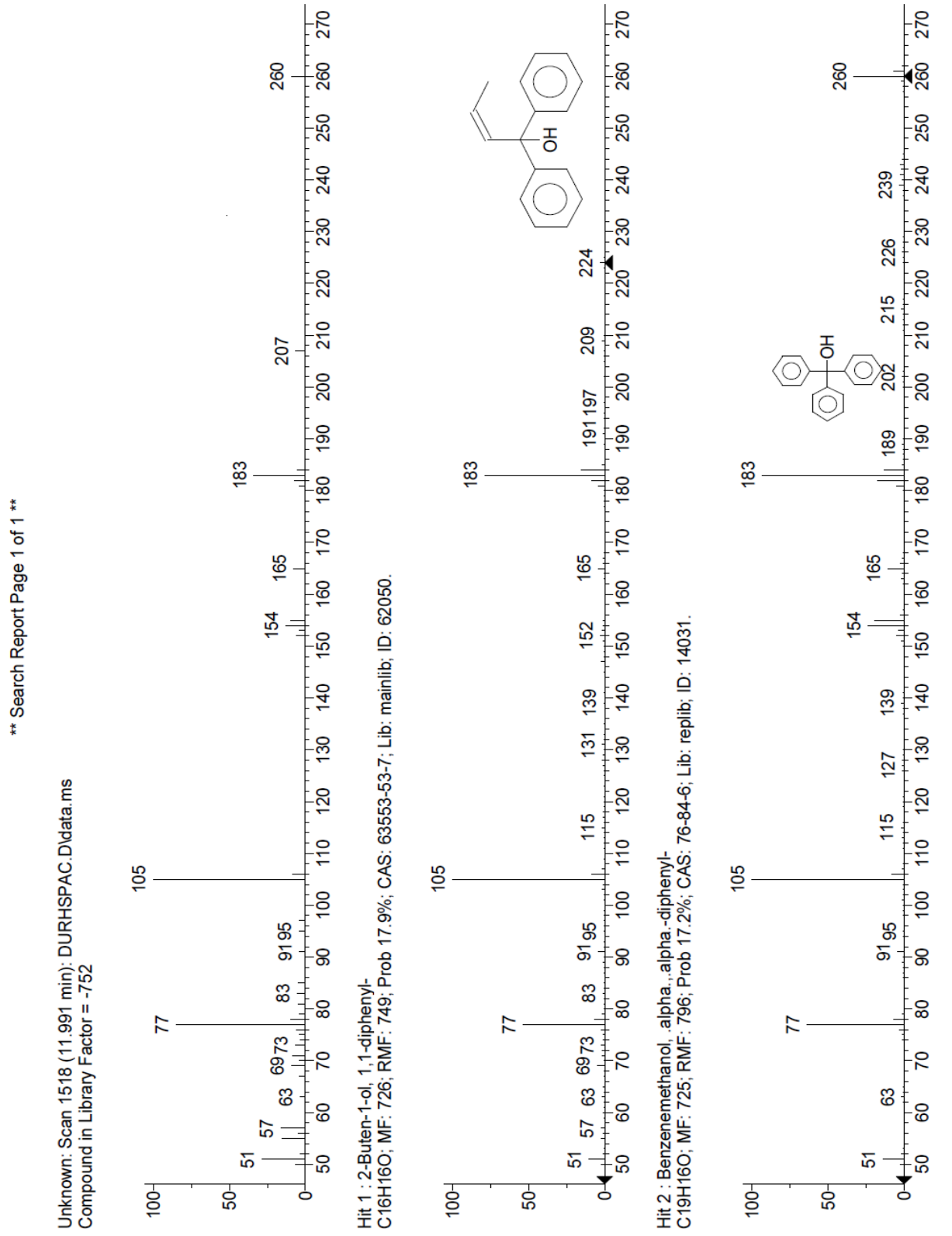


Figure H30. DURHSPAC RT 11.991 min MS

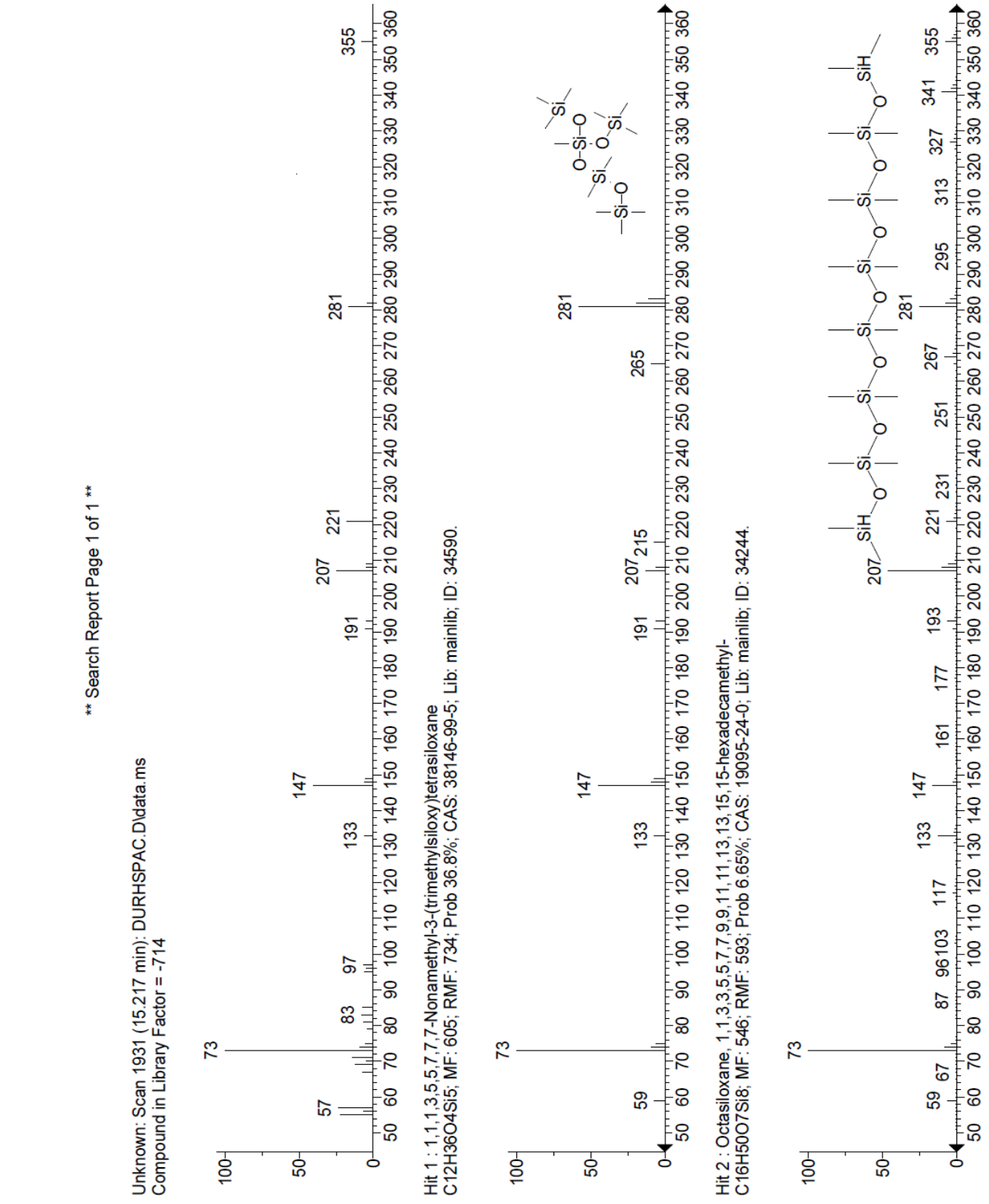


Figure H31. DURHSPAC RT 15.217 min MS

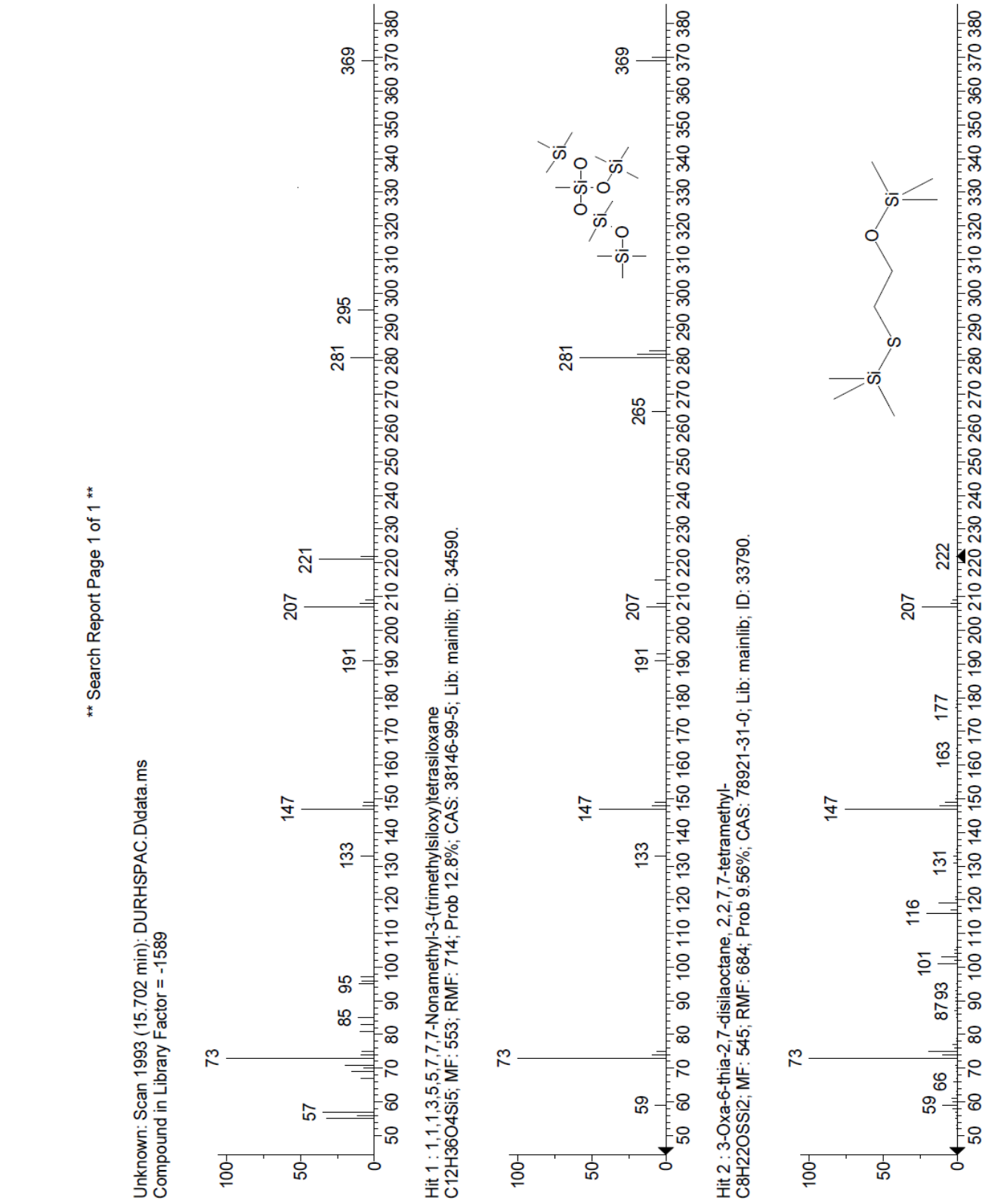


Figure H32. DURHSPAC RT 15.702 min MS

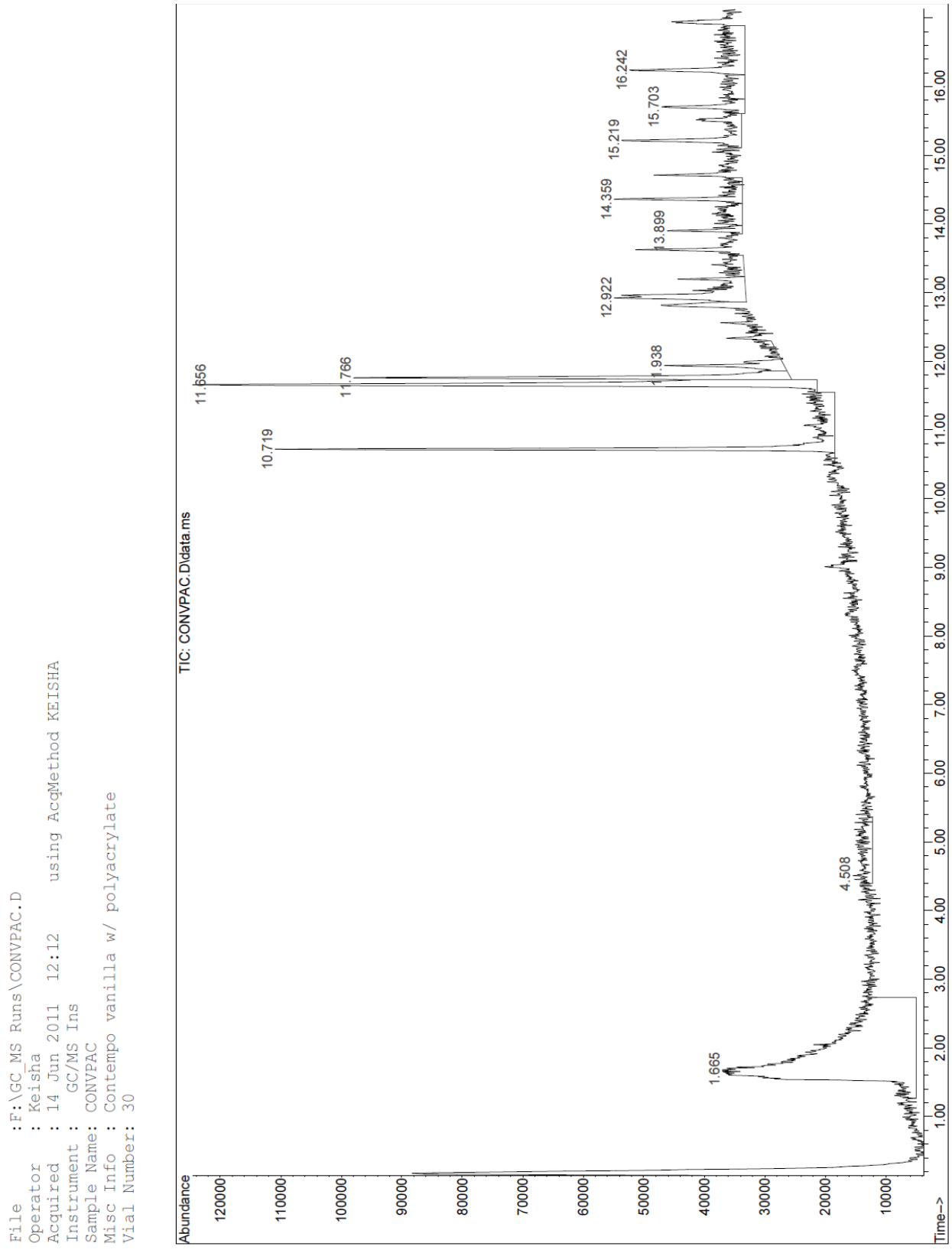


Figure H33. CONVPAC TIC

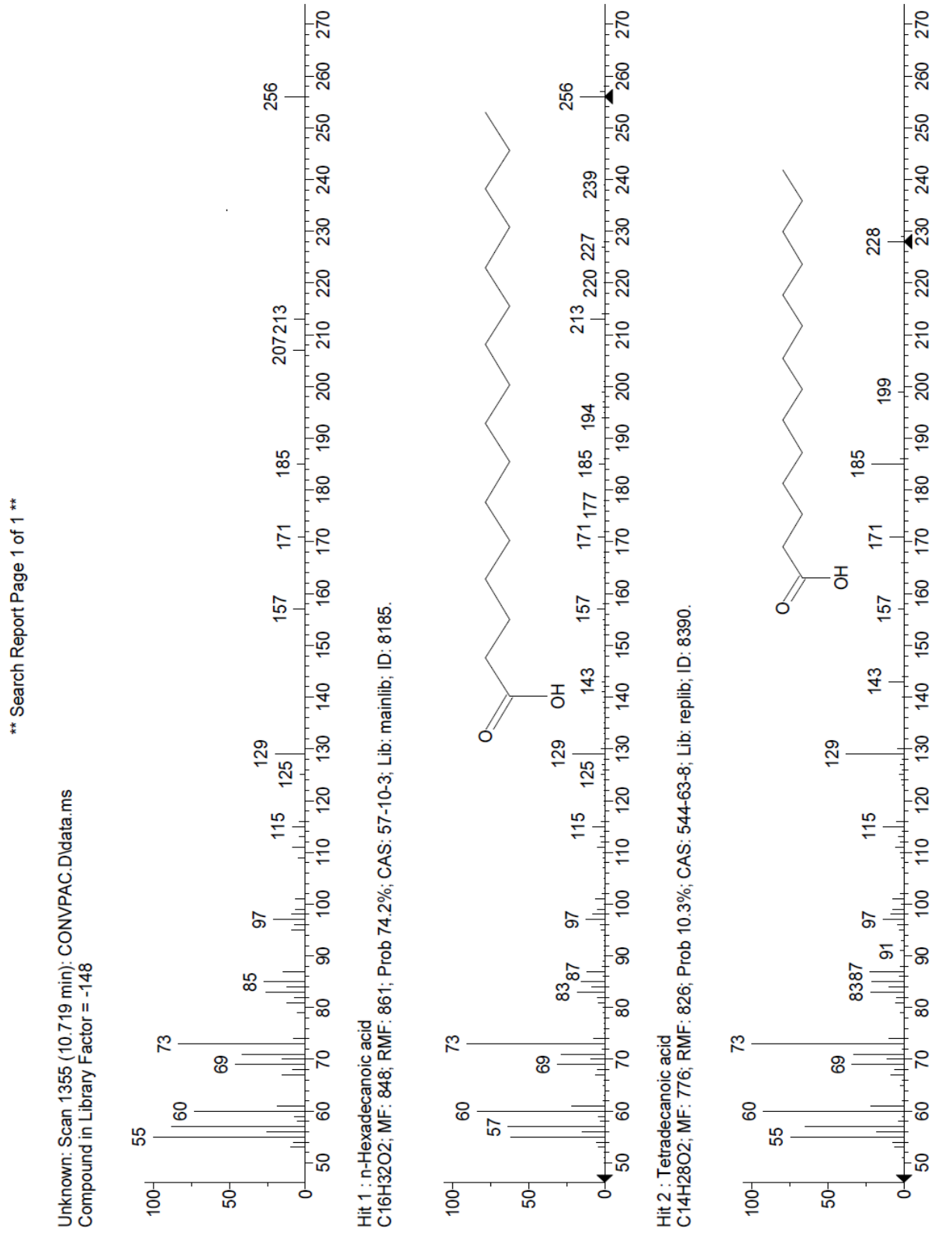


Figure H34. CONVPAC RT 10.719 min MS

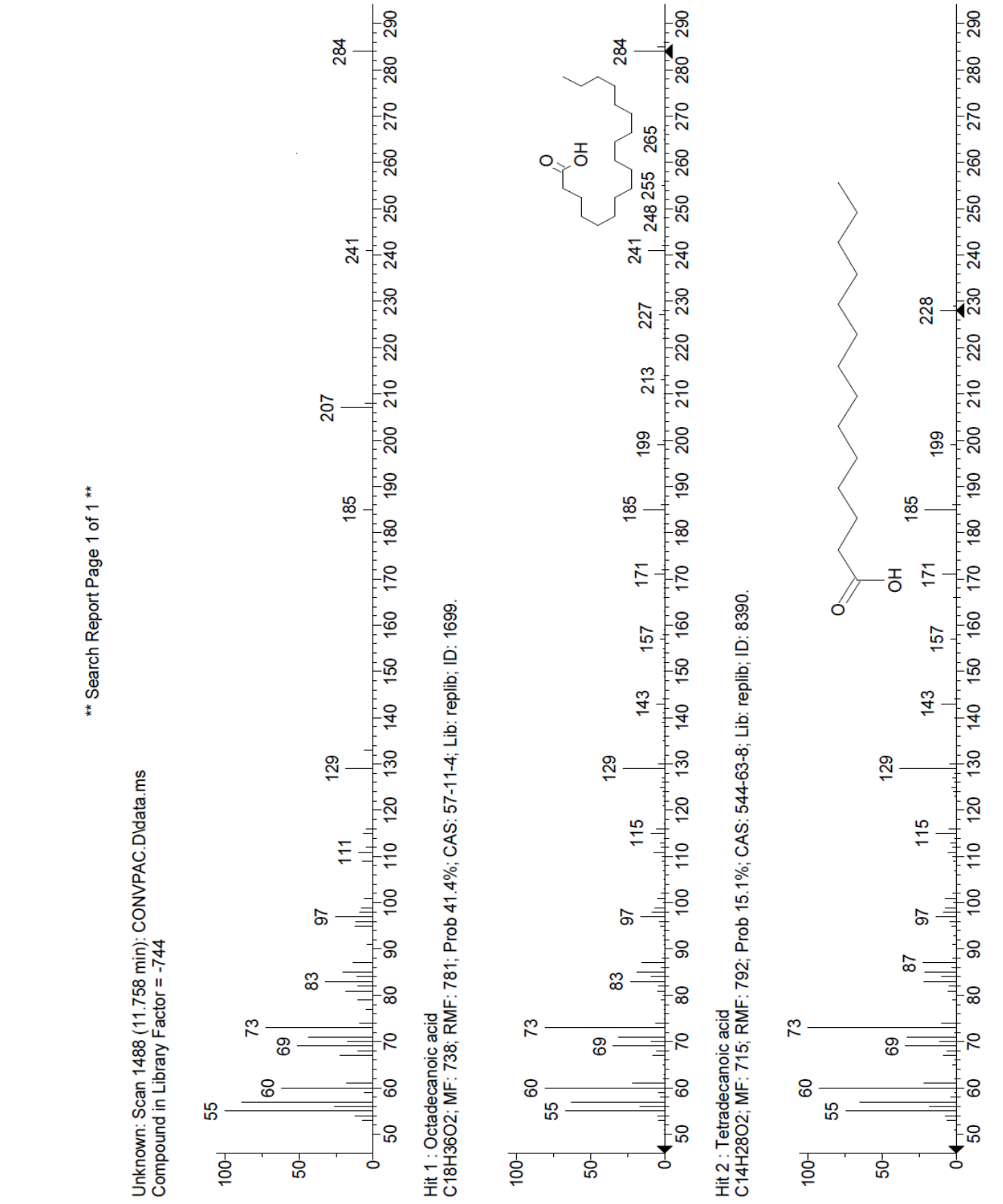


Figure H35. CONVPAC RT 11.758 min MS

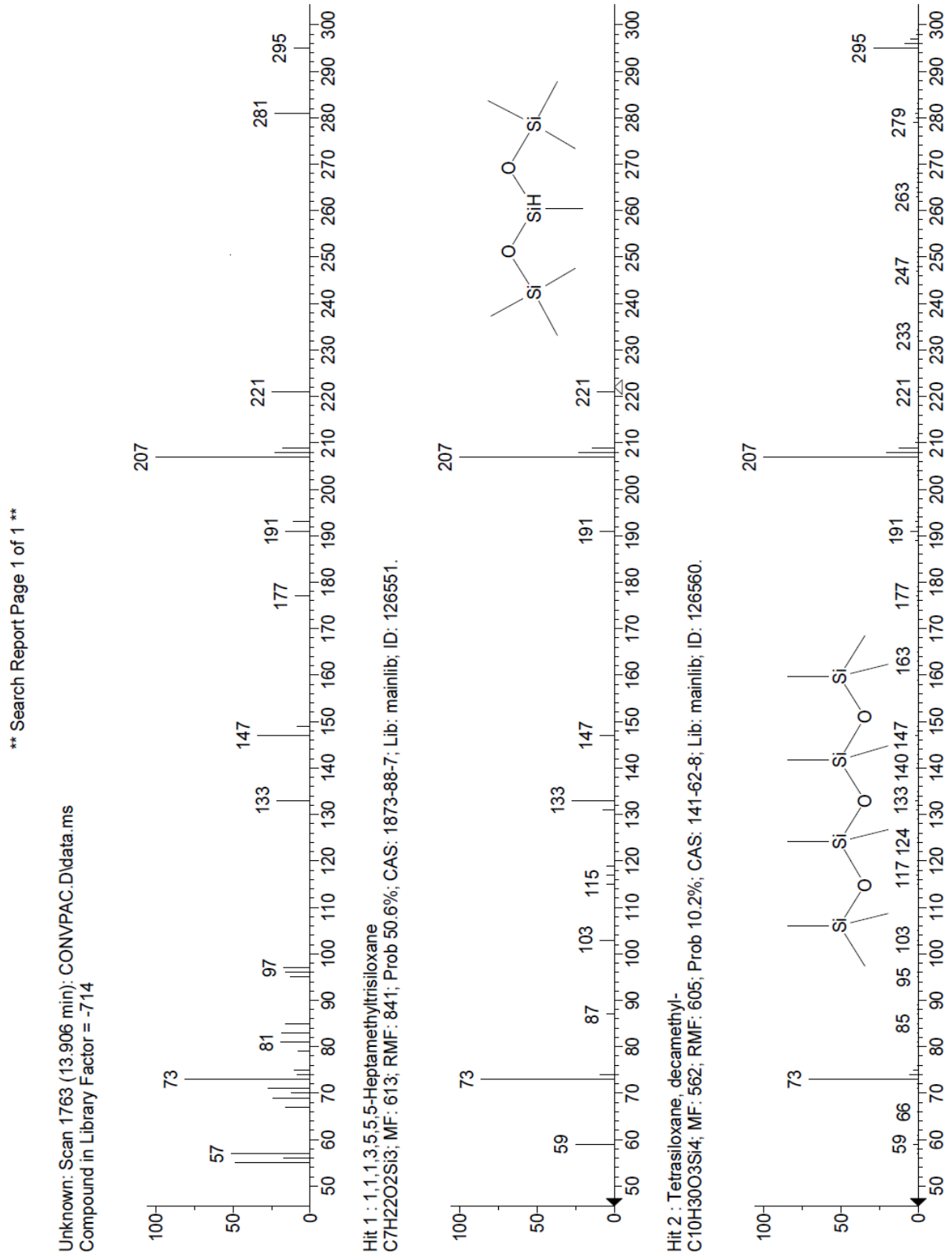


Figure H36. CONVPAC RT 13.906 min MS

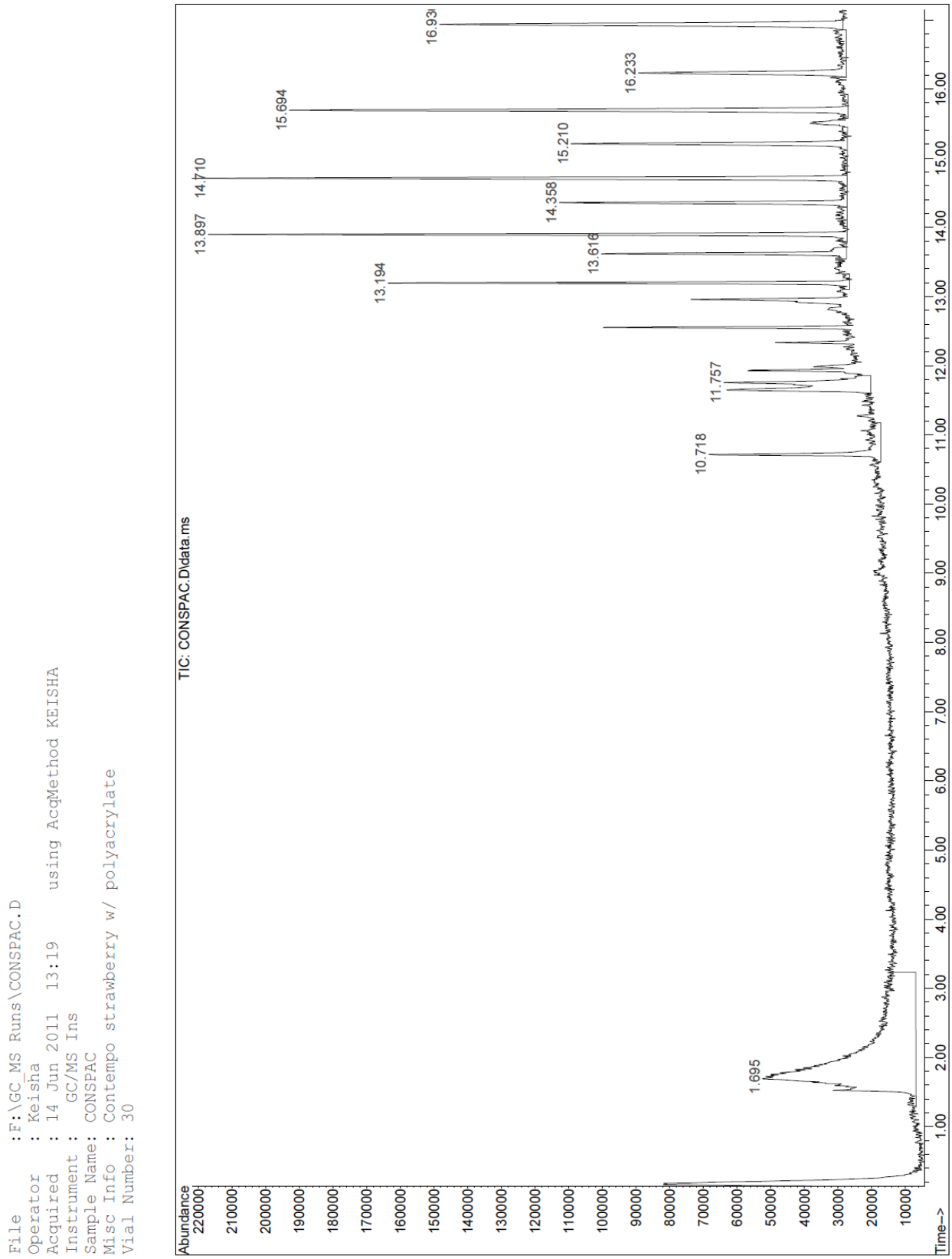


Figure H37. CONSPAC TIC

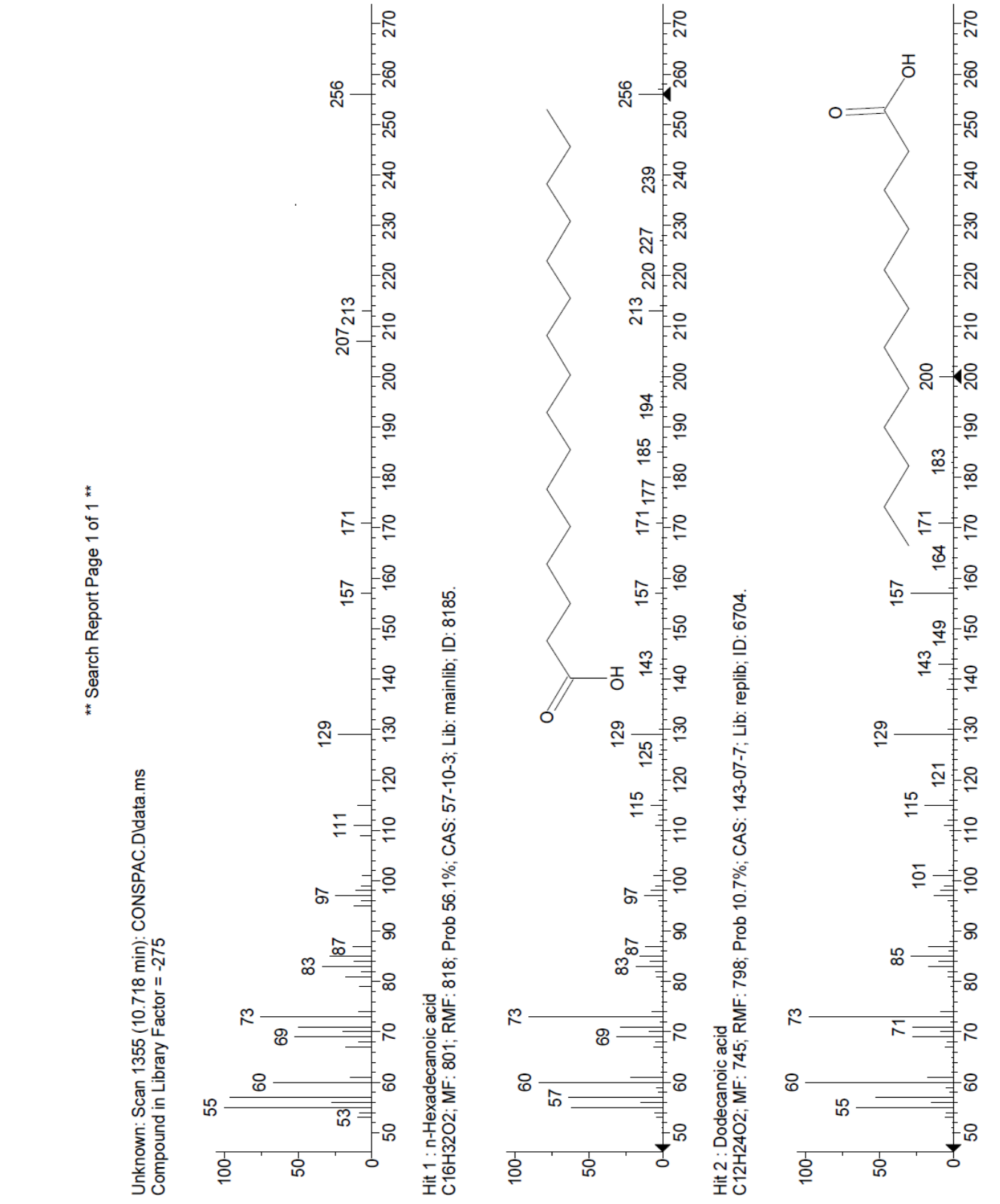


Figure H38. CONSPAC RT 10.718 min MS

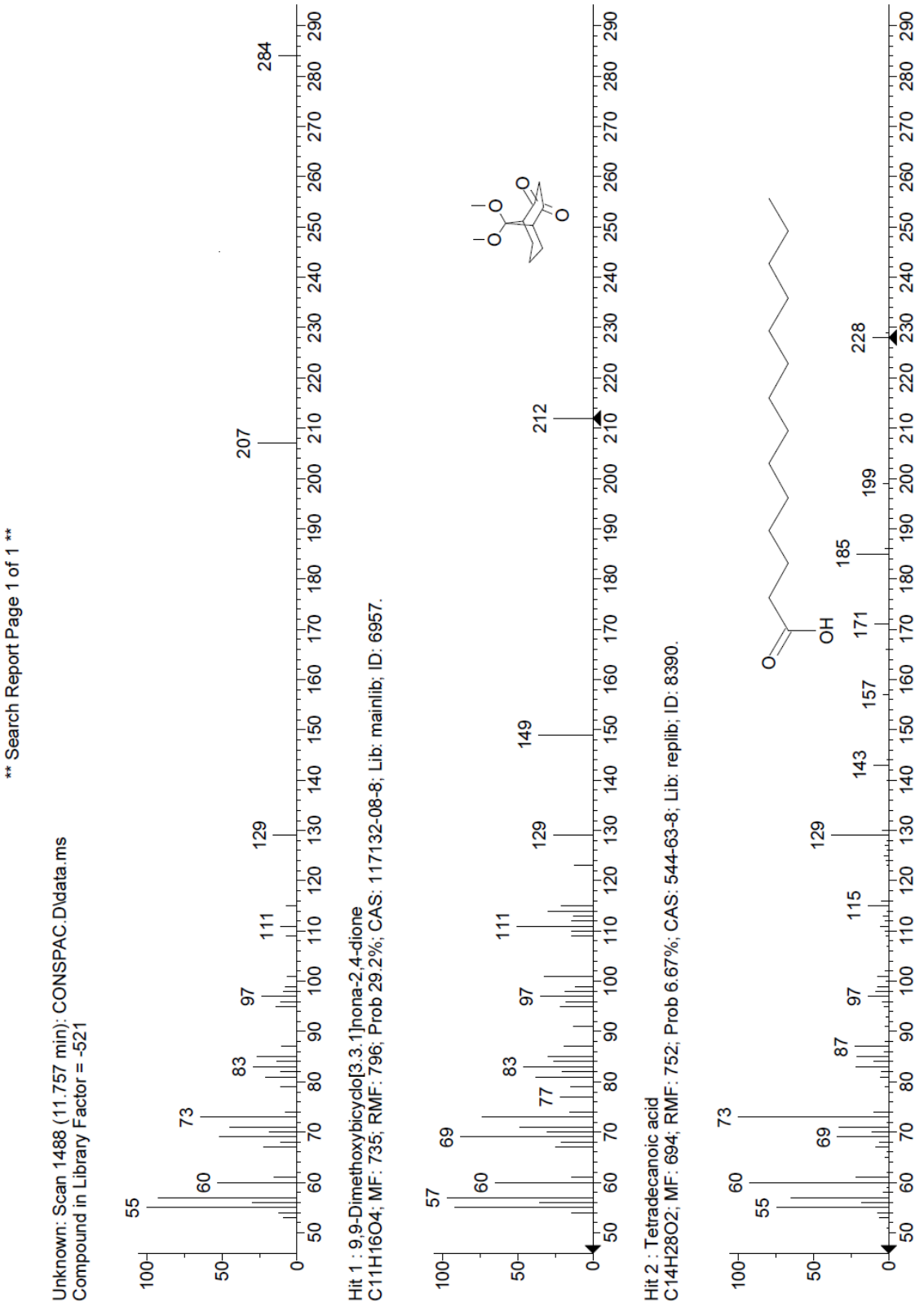


Figure H39. CONSPAC RT 11.757 min MS

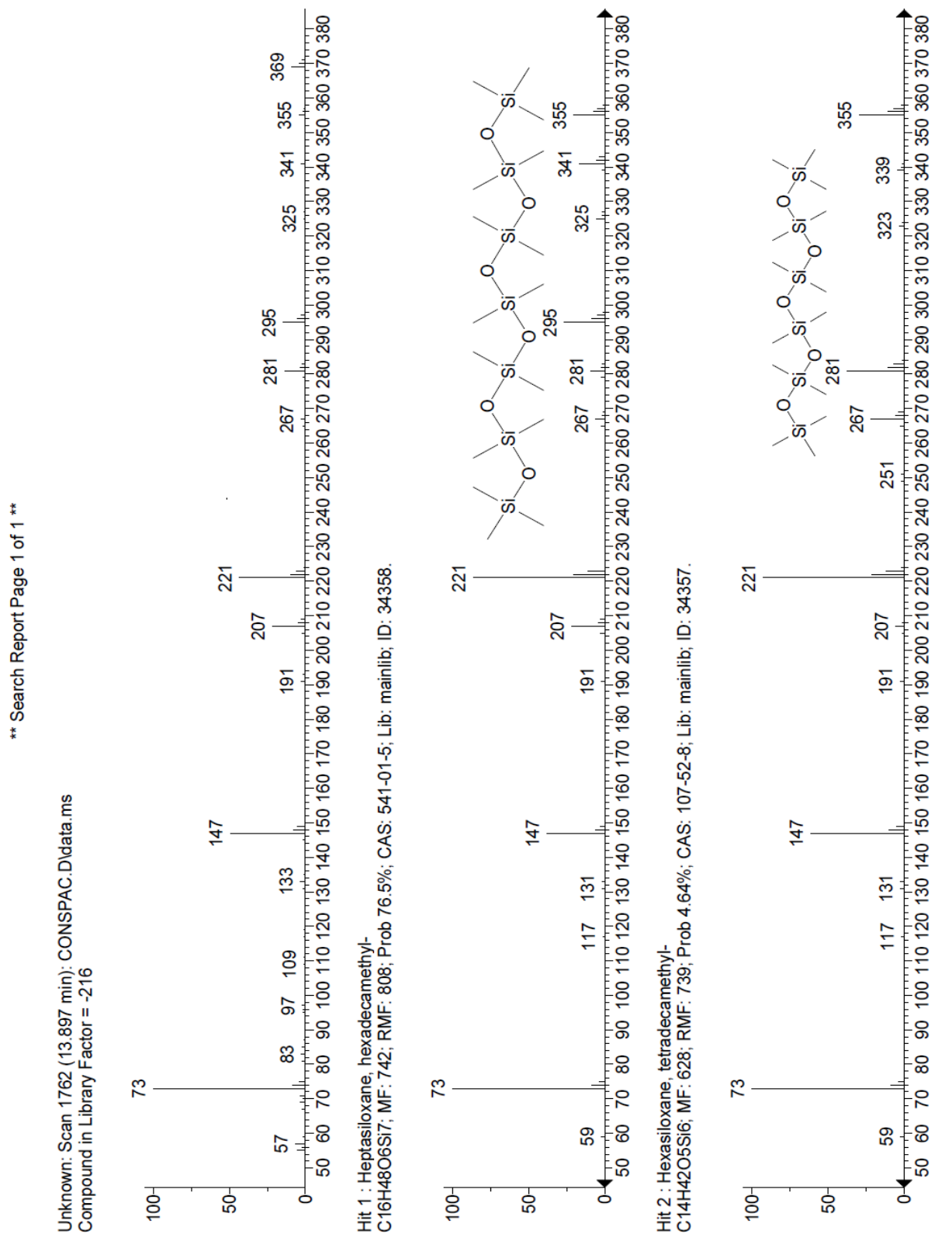


Figure H40. CONSPAC RT 13.897 min MS

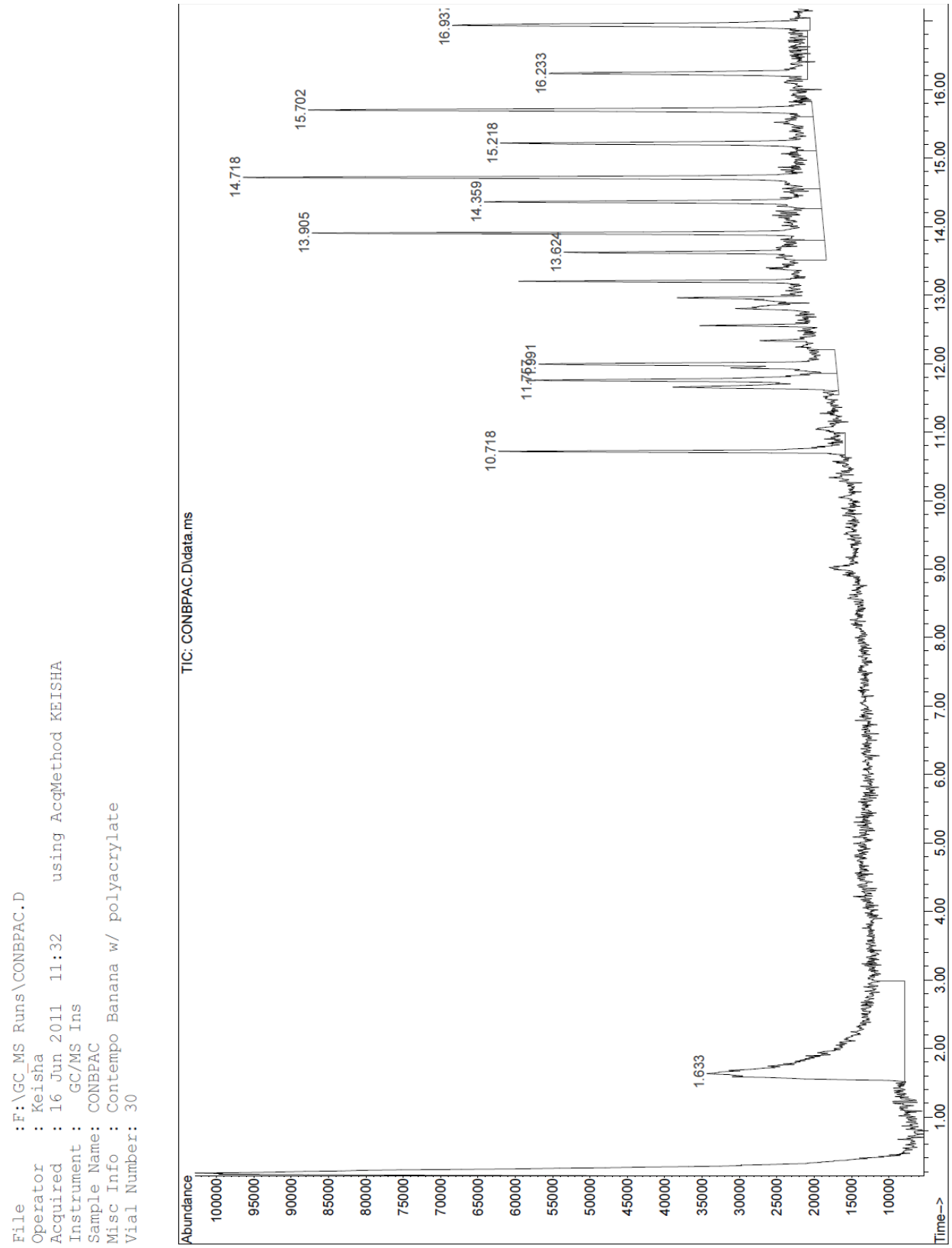


Figure H41. CONBPAC TIC

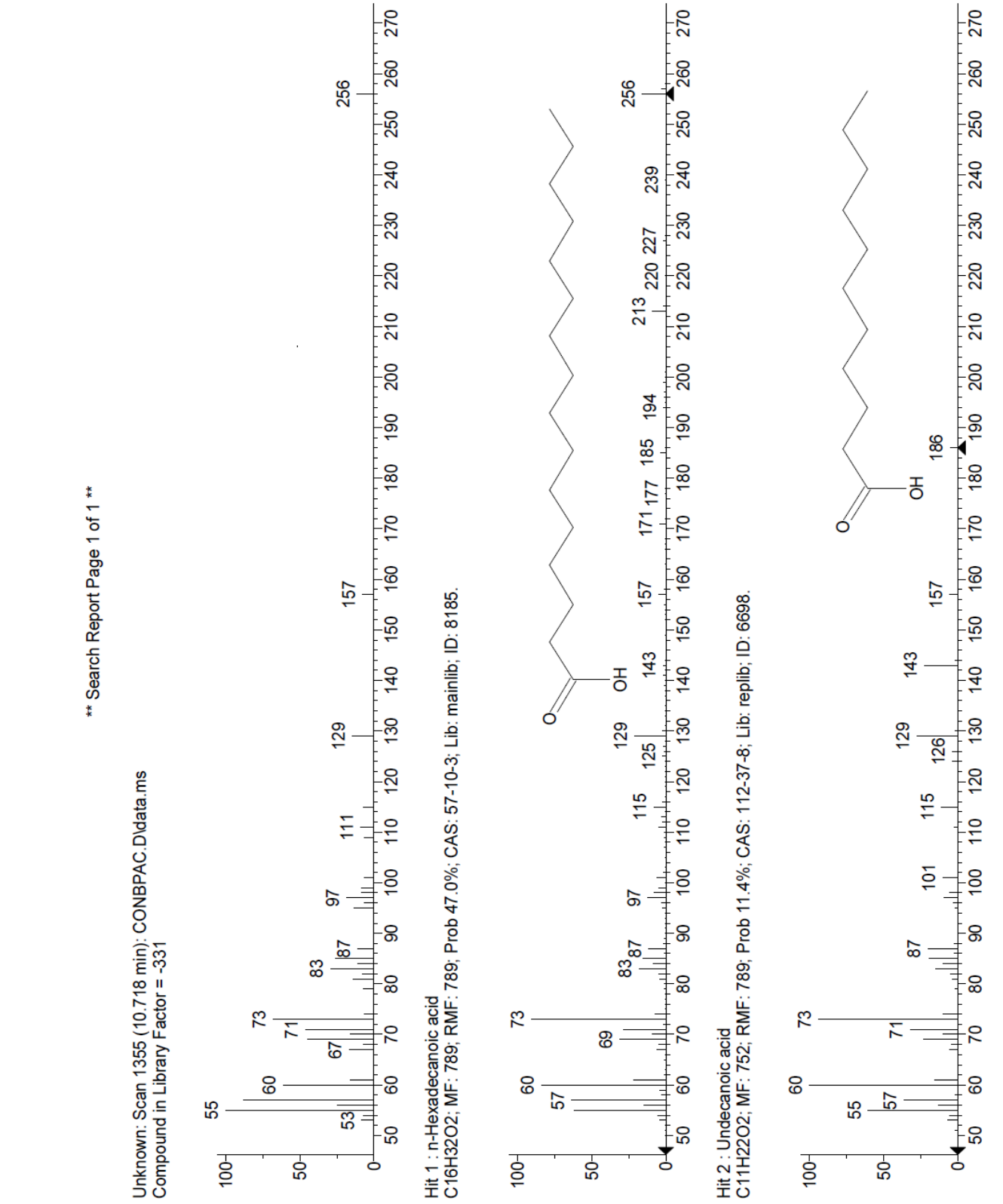


Figure H42. CONBPAC RT 10.718 min MS

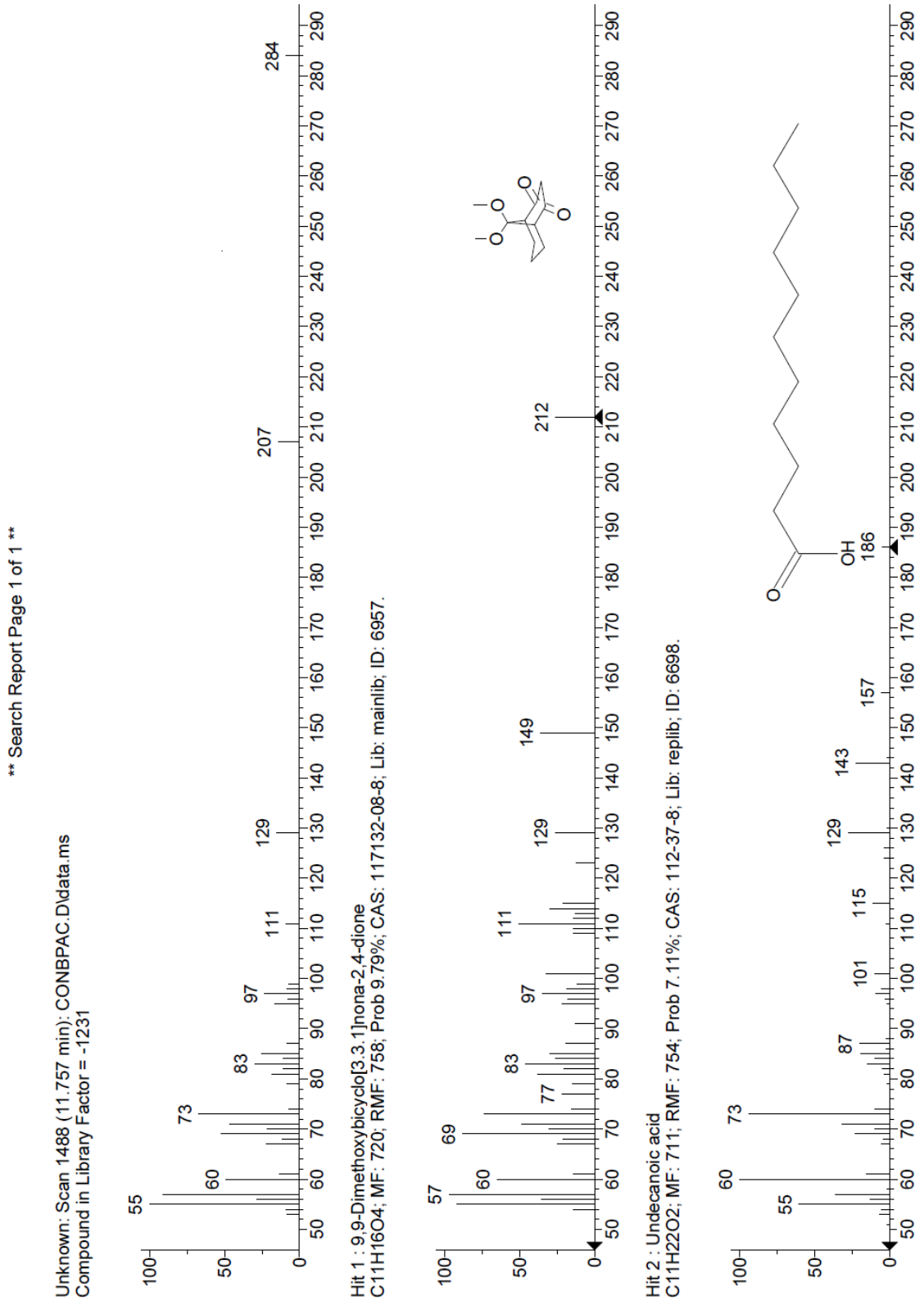


Figure H43. CONBPAC RT 11.757 min MS

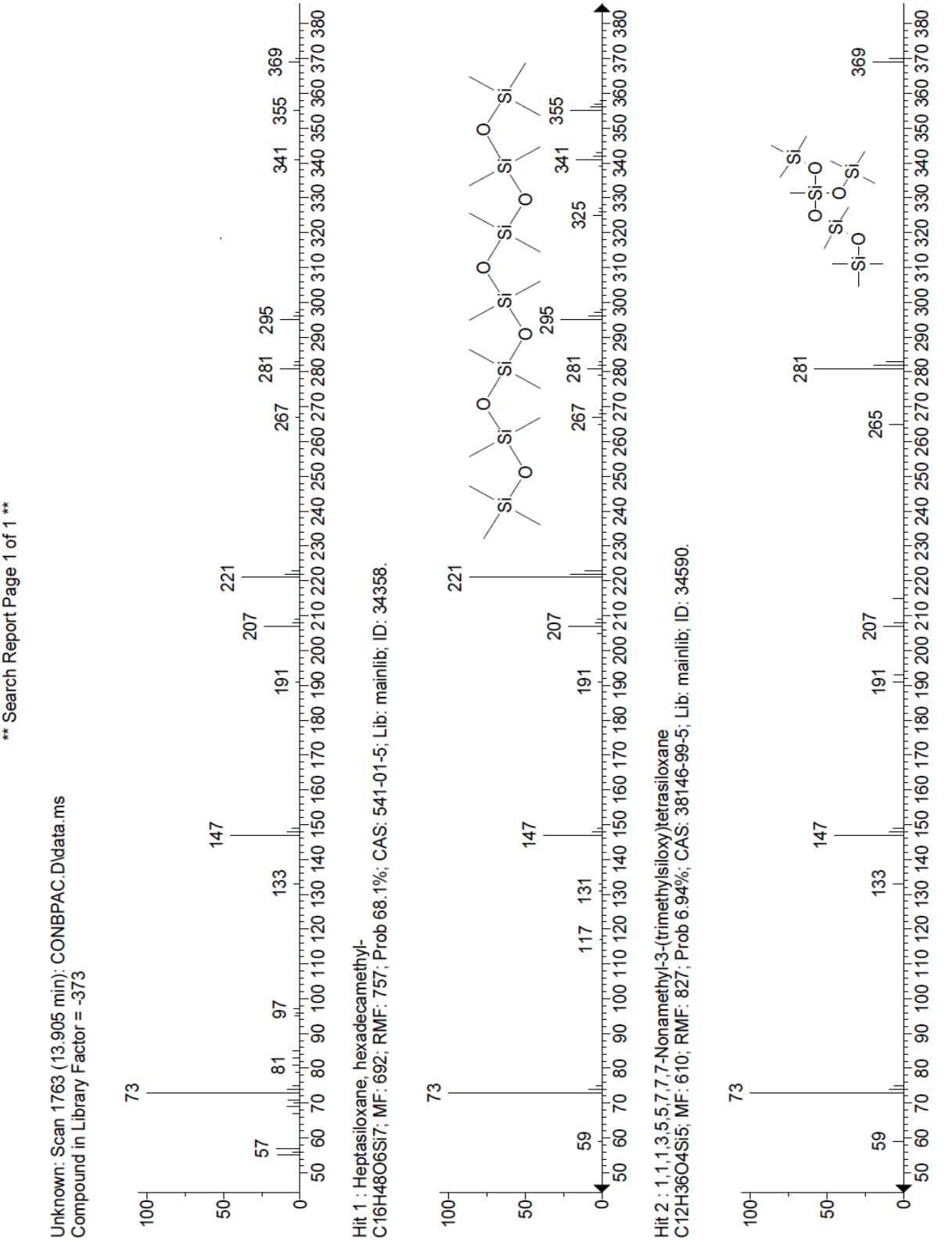


Figure H44. CONBPAC RT 13.905 min MS

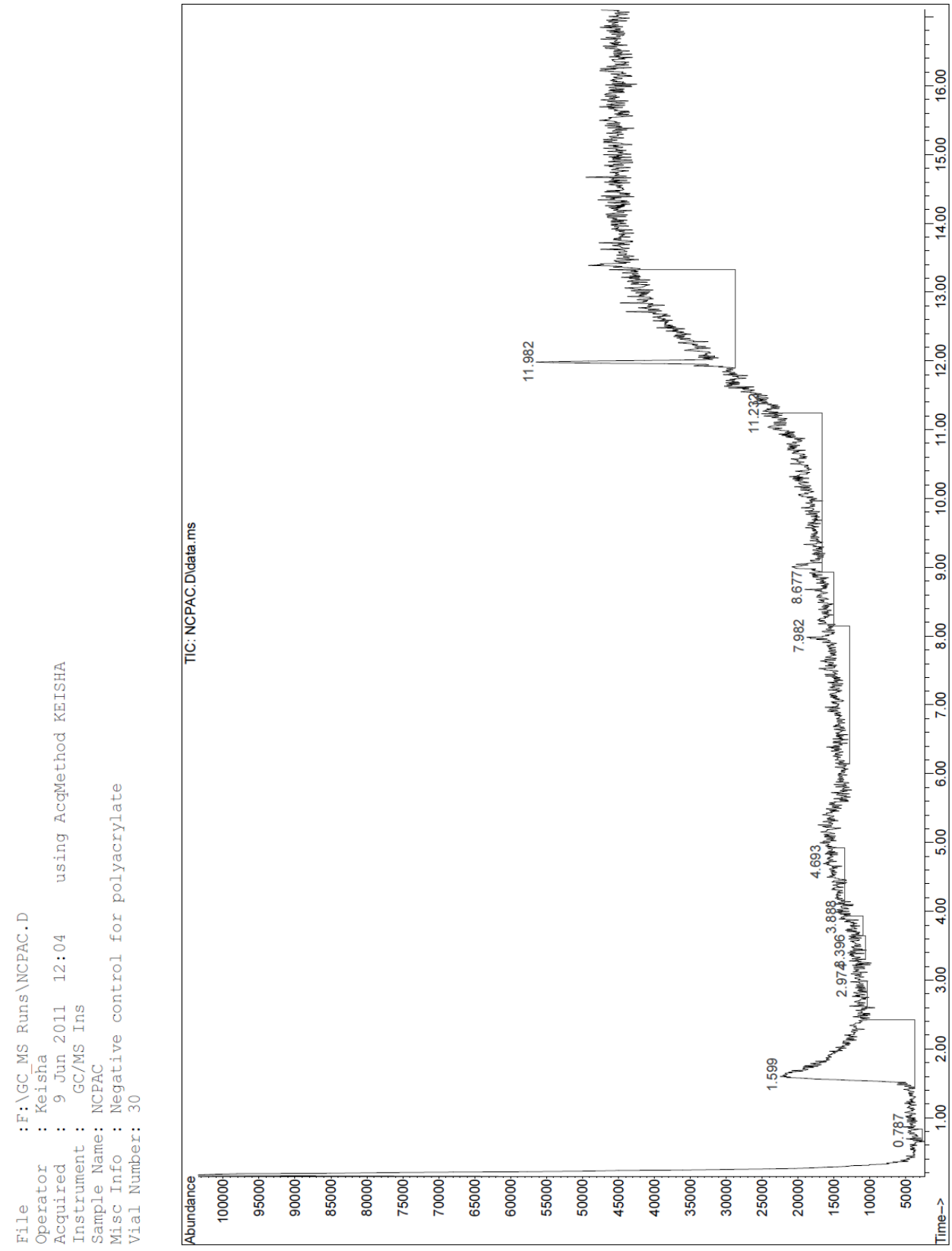


Figure H45. NCPAC TIC

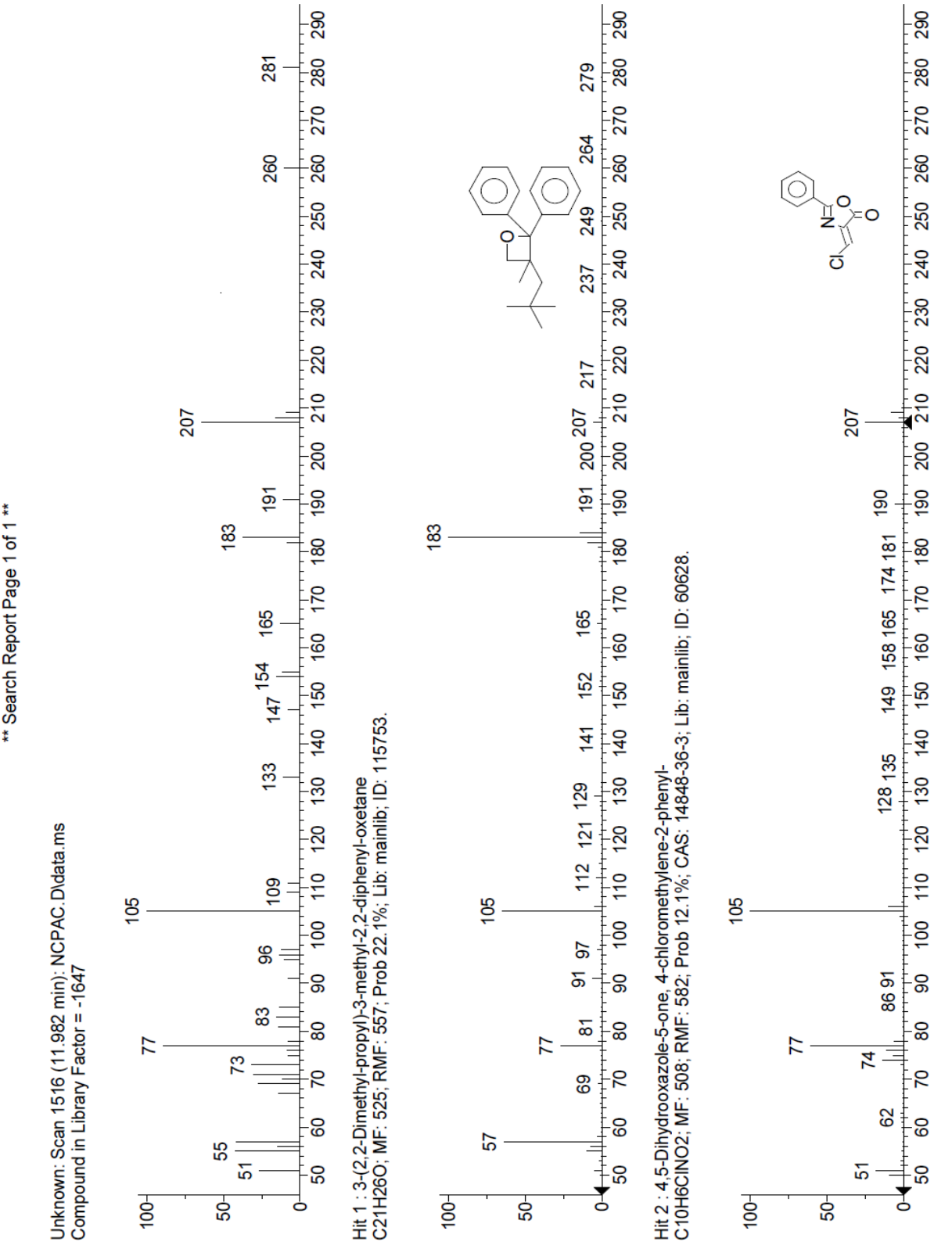


Figure H46. NCPAC RT 11.982 min MS

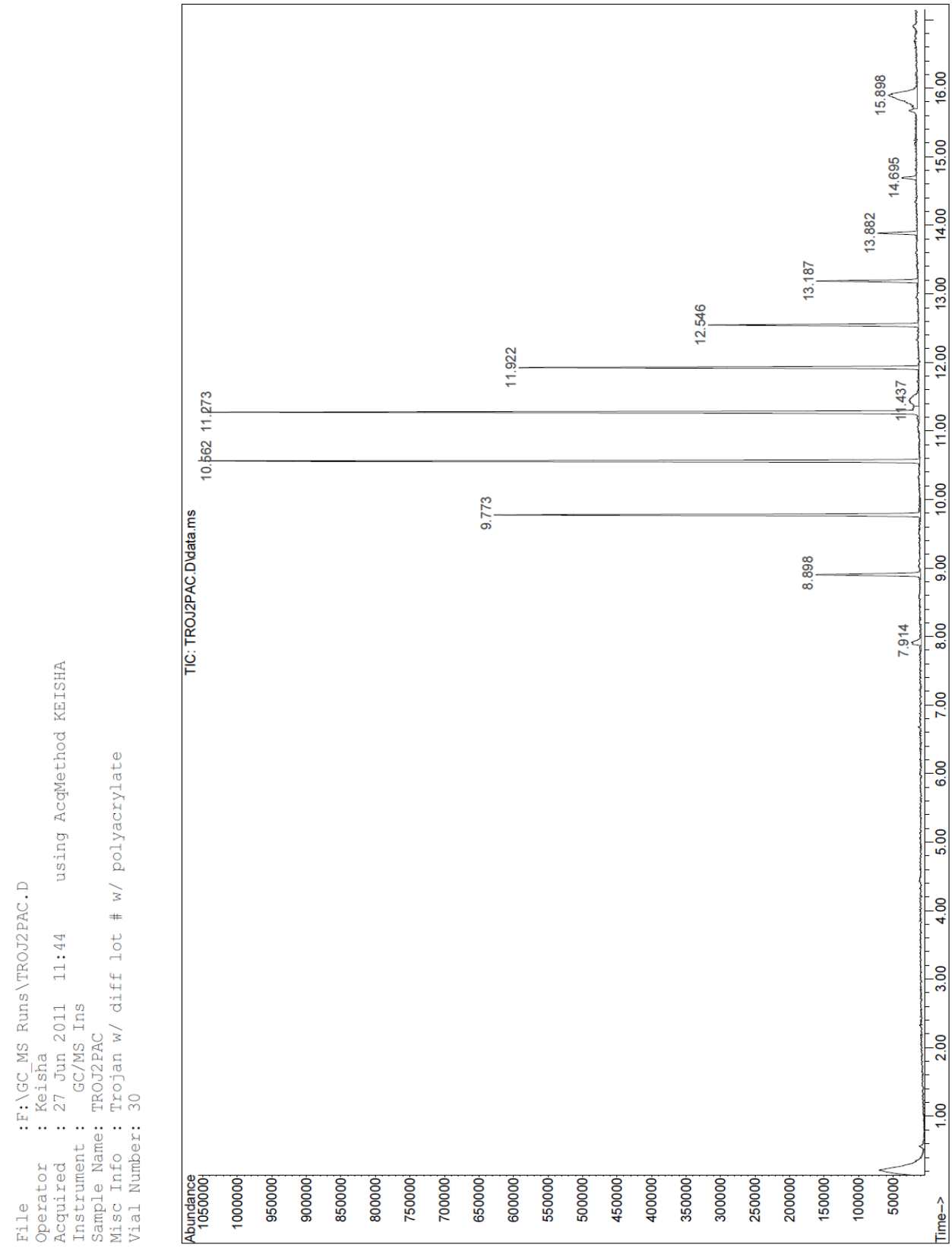


Figure H47. TROJ2PAC TIC

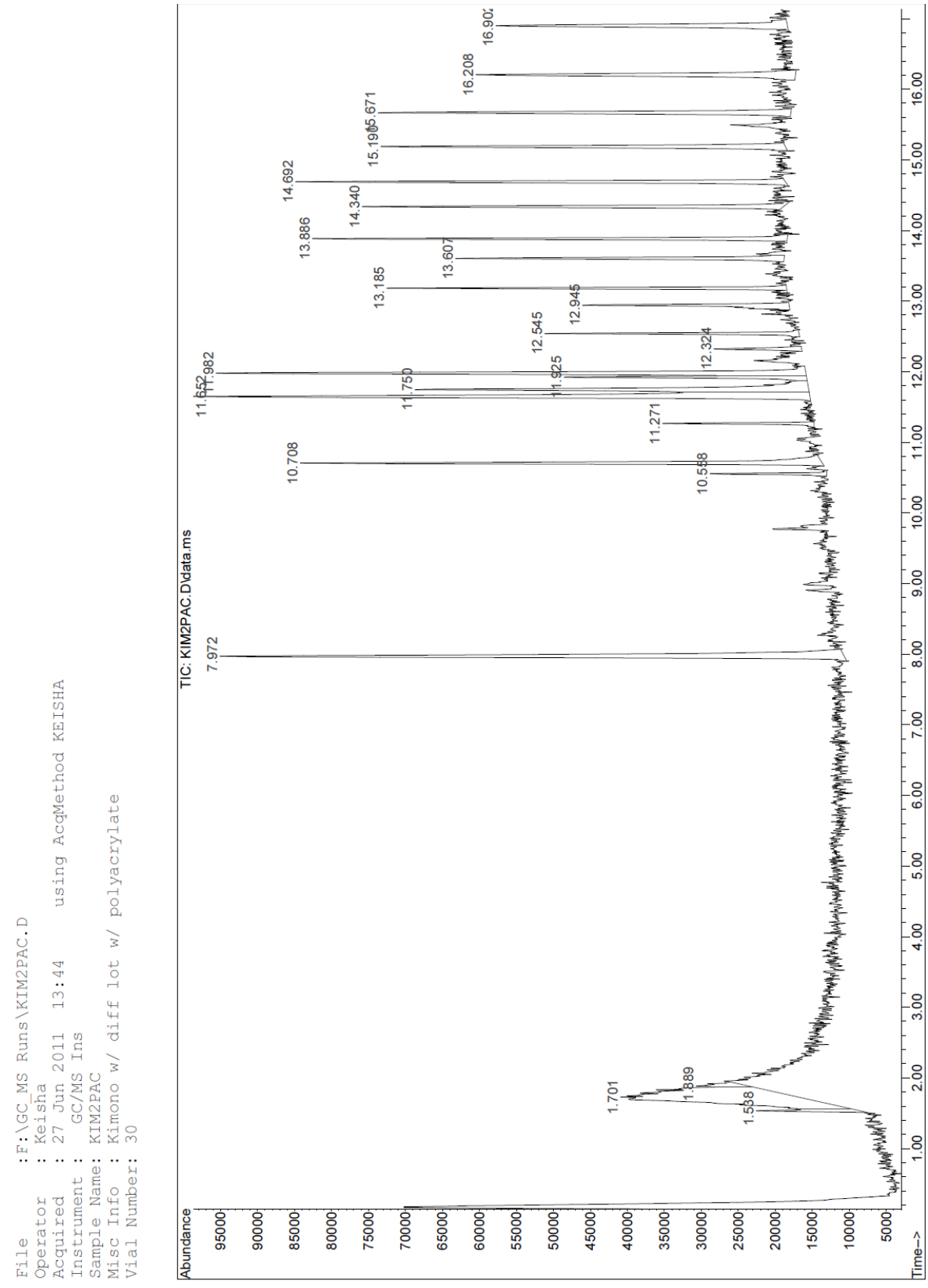


Figure H48. KIM2PAC TIC

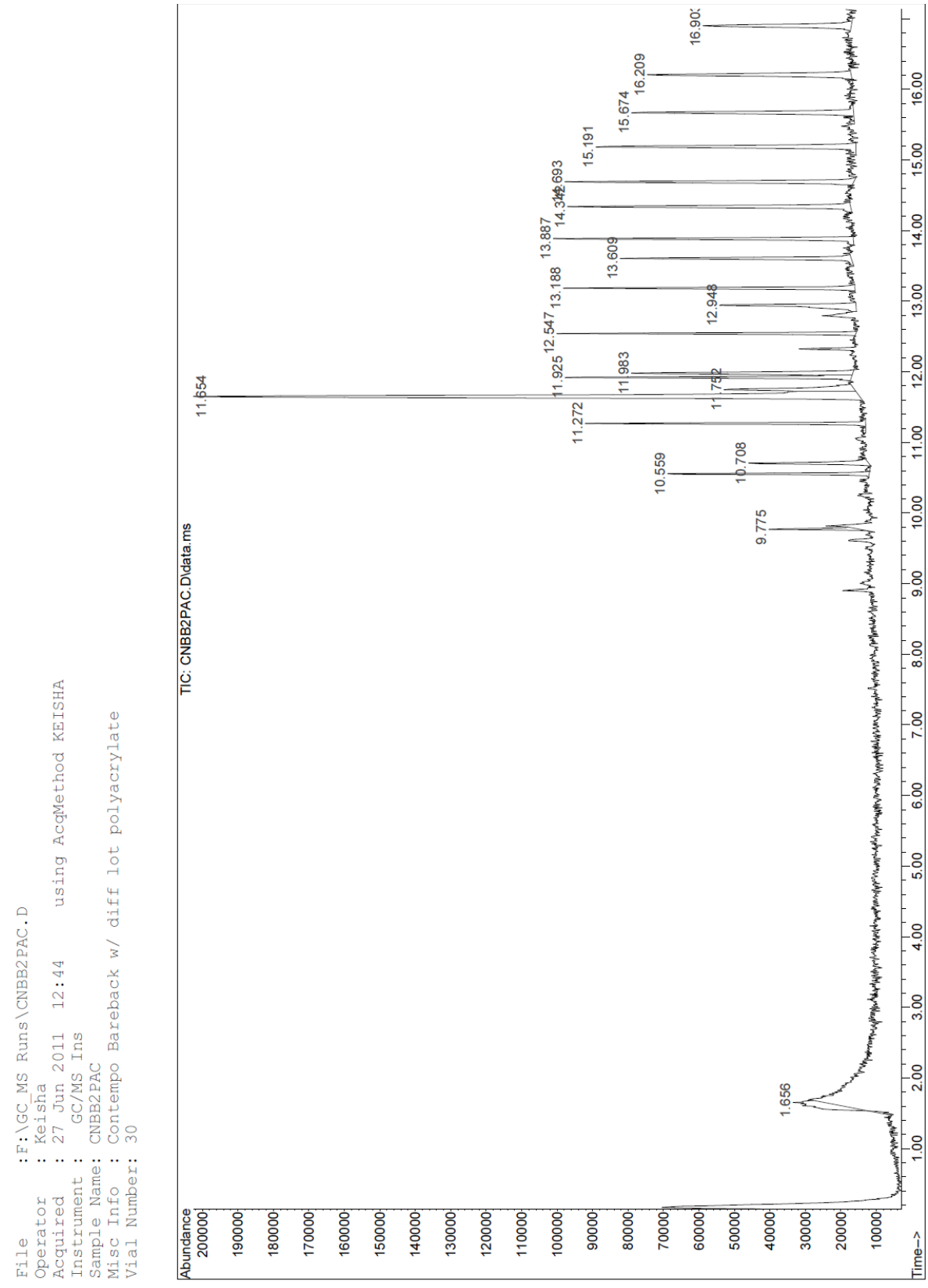


Figure H49. CNBB2PAC TIC

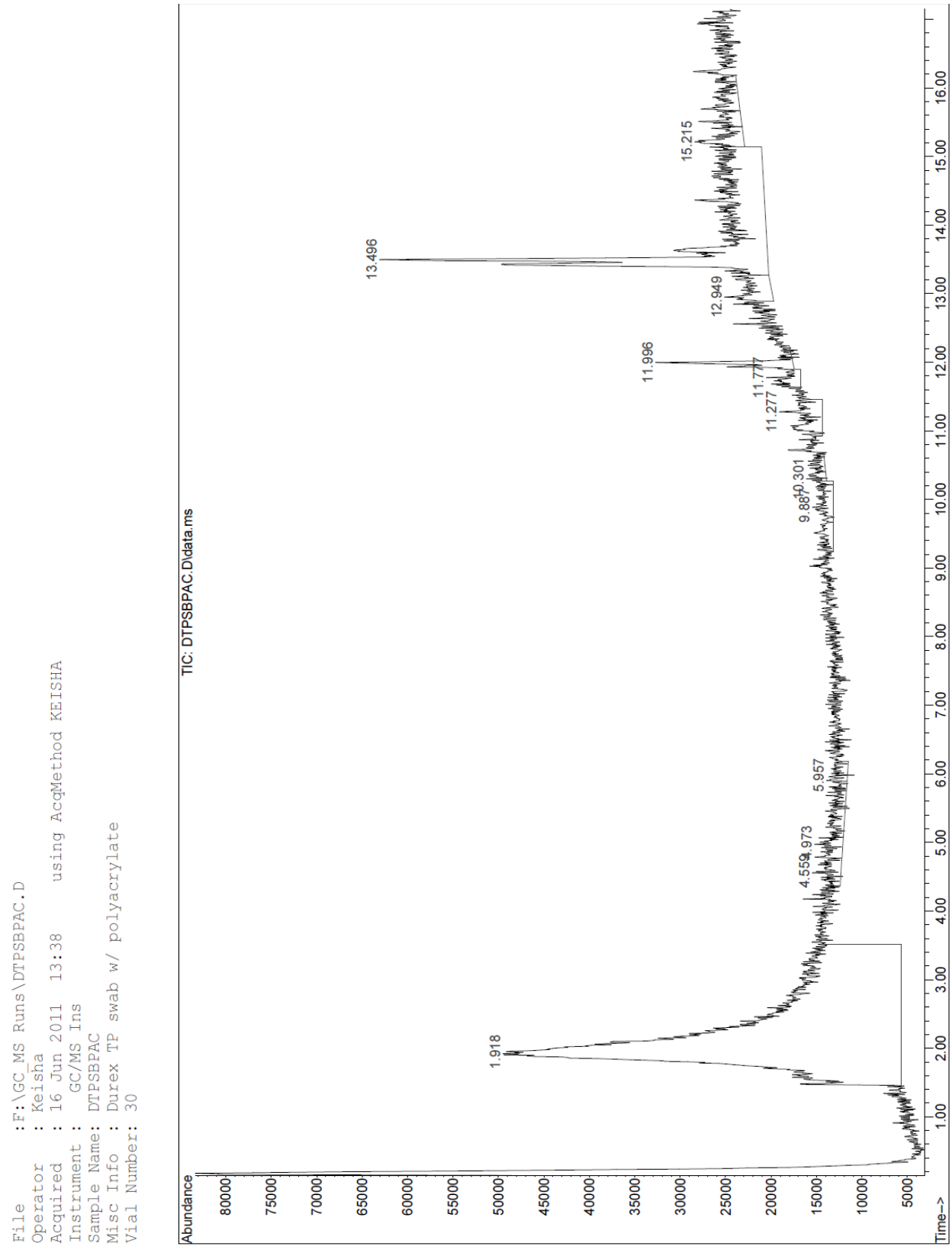


Figure H50. DTPSBPAC TIC

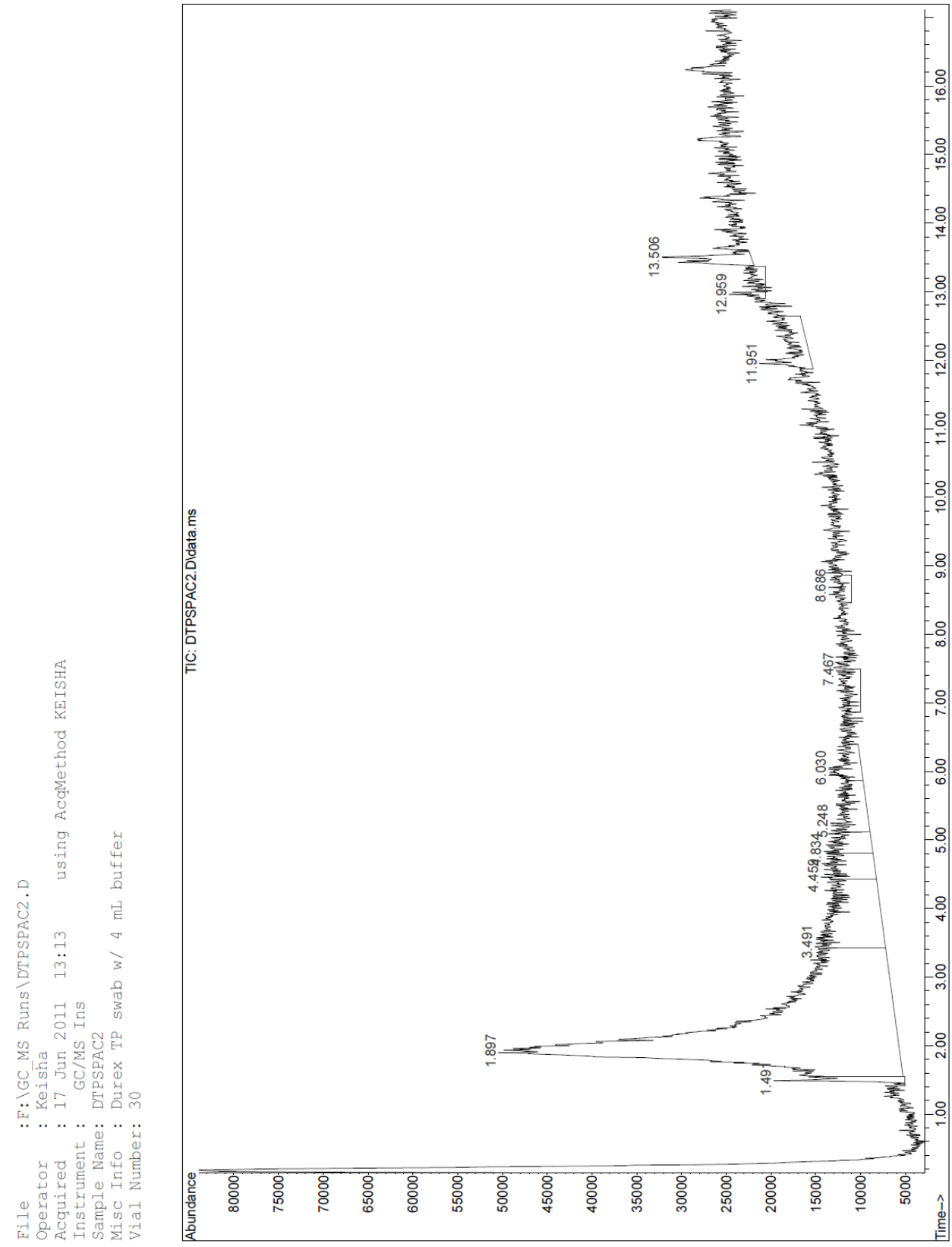


Figure H51. DTPSPAC2 TIC

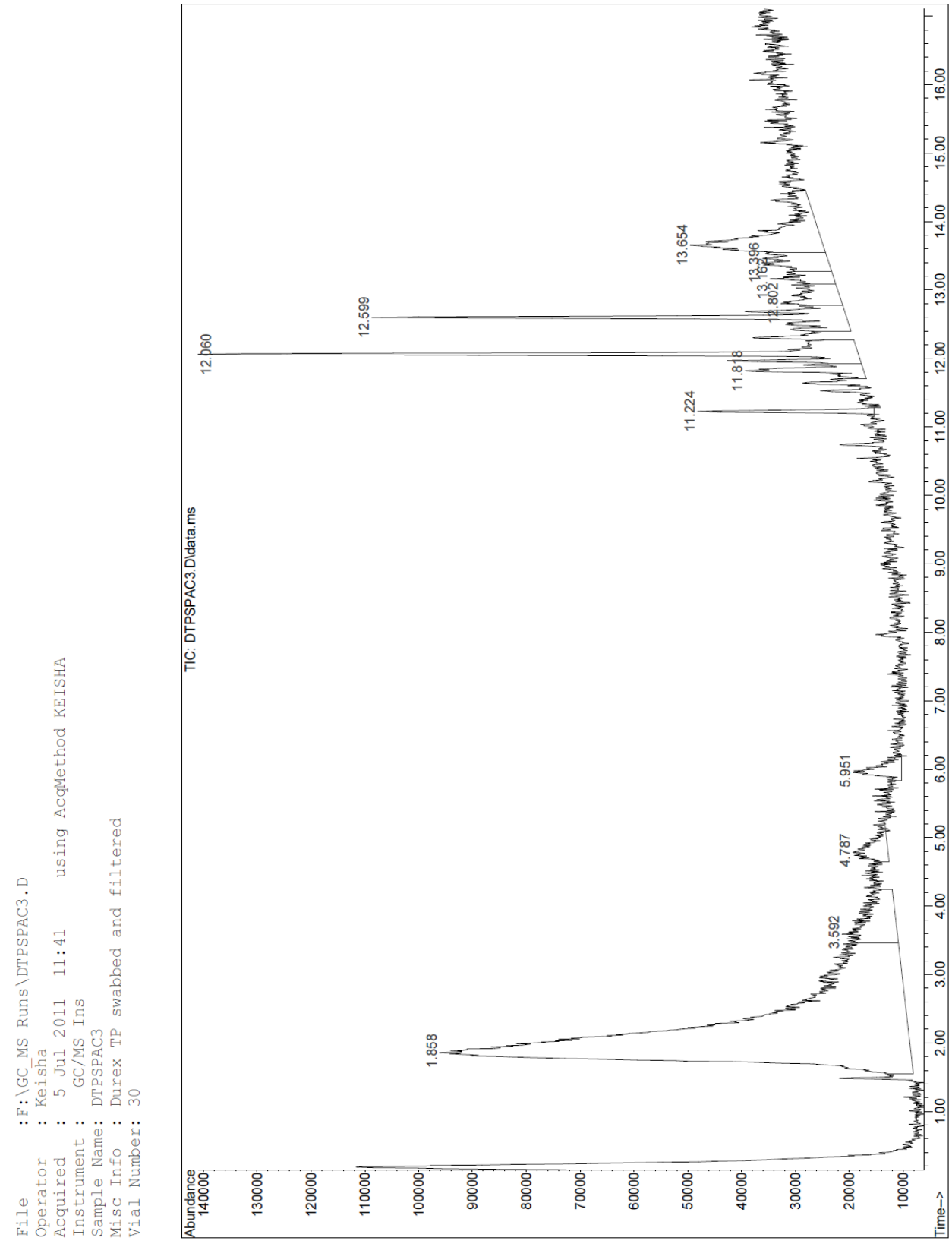


Figure H52. DTPSPAC3 TIC

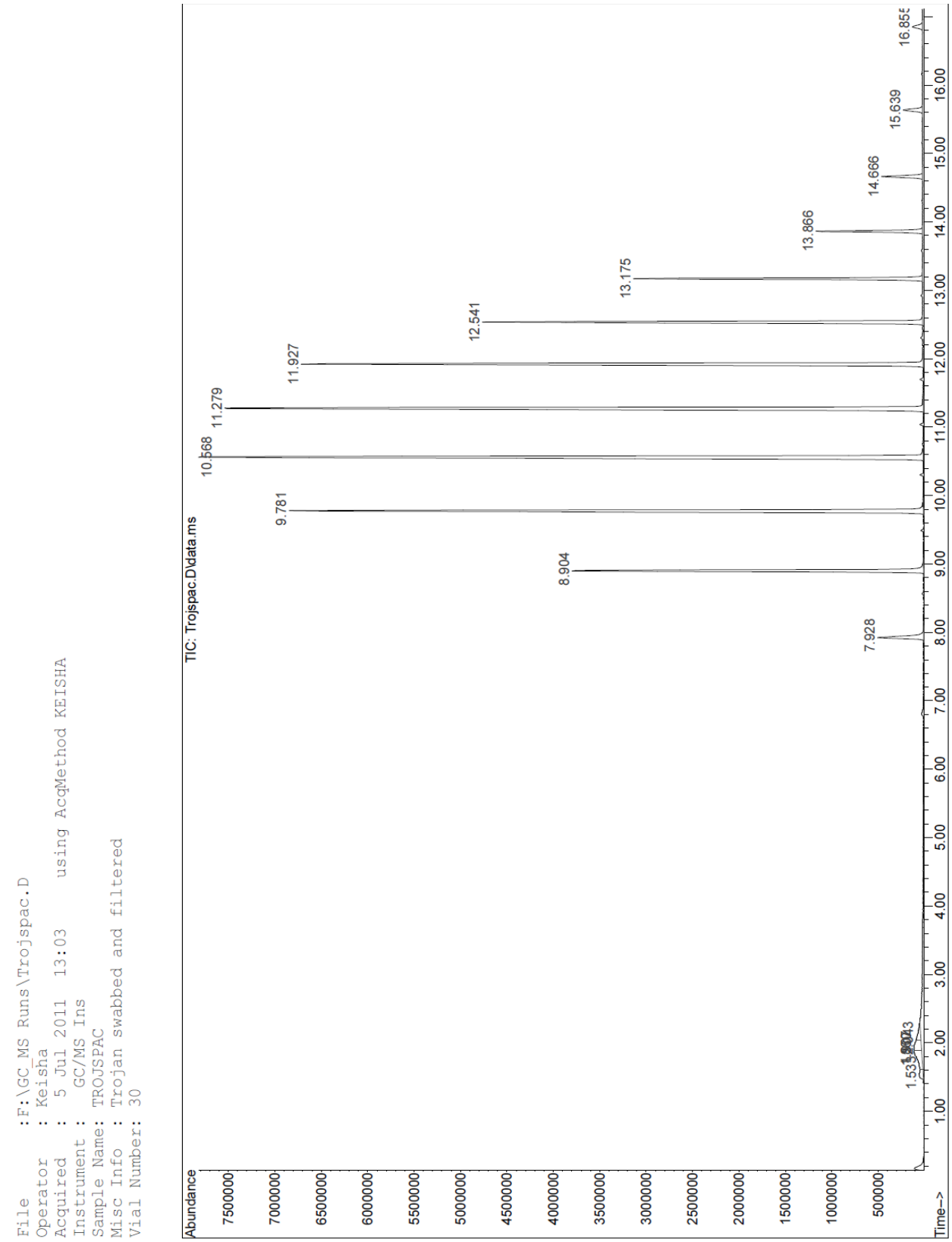


Figure H53. TROJSPAC TIC

# Appendix I

## 2011 FDA Registered Condom Companies

Table I1

*2011 FDA Registered Condom Companies*

| Business Name | Location | Nature of Business |
|---|---|---|
| ACP | FRANCE | Repackager/Relabeler |
| ADELPHIA DISCOUNT SERVICES INC. | NY/USA | Repackager/Relabeler |
| ANSELL HEALTHCARE PRODUCTS LLC | AL/USA | Specification Developer |
| BANDA STAR (DONG GUAN) ELECTRONICS CO., LTD. | CHINA | Repackager/Relabeler |
| BANDA STAR INDUSTRIAL LIMITED | HONG KONG, CHINA | Foreign Exporter |
| BARNETT INTL., CORP. | NC/USA | Repackager/Relabeler |
| BIOFILM, INC. | CA/USA | Manufacturer |
| BIZZY DIAMOND BV | NETHERLANDS | Foreign Exporter; Repackager/Relabeler |
| BRETHREN SERVICE CENTER | MD/USA | Packager/Relabeler |
| BRISAR INDUSTRIES | NJ/USA | Repackager/Relabeler |
| C.B. FLEET CO., INC. | VA/USA | Repackager/Relabeler |
| Caution Wear Corp | NH/USA | Repackager/Relabeler |
| CHURCH & DWIGHT CO., INC. | VA/USA | Manufacturer; Repackager/Relabeler |
| CHURCH & DWIGHT CO., INC. | NJ/USA | Specification Developer |
| CUPID LTD. | INDIA | Contract Manufacturer; Manufacturer; Specification Developer |
| DALIAN LATEX CO LTD | CHINA | Contract Manufacturer; Manufacturer |
| DAVRYAN LABORATORIES, INC. | OR/USA | Specification Developer |
| DONGKUK TRADING CO., LTD. | KOREA, REPUBLIC OF | Manufacturer |

| | | |
|---|---|---|
| DURDEN ENTERPRISES | GA/USA | Contract Manufacturer |
| Evofem Inc. (formerly d/b/a Instead, Inc.) | CA/USA | Specification Developer |
| Faria Limited LLC, dba Sheffield Pharmaceuticals | CT/USA | Manufacturer |
| FUJI LATEX CO., LTD. | JAPAN | Contract Manufacturer |
| GLOBAL PROTECTION CORP. | MA/USA | Repackager/Relabeler; Specification Developer |
| GLYDE HEALTH PTY LTD | AUSTRALIA | Foreign Exporter |
| GRAPHIC ARMOR, INC. | NC/USA | Repackager/Relabeler |
| Grove Medical,LLC | GA/USA | Manufacturer; Specification Developer |
| GUANGZHOU GUANGXIANG ENTERPRISES GROUP CO., LTD | CHINA | Contract Manufacturer; Manufacturer |
| GUILIN LATEX FACTORY | CHINA | Contract Manufacturer; Manufacturer |
| HANKOOK LATEX GONGUP CO., LTD. | KOREA, REPUBLIC OF | Manufacturer |
| HLL LIFECARE LIMITED | INDIA | Manufacturer |
| HR PHARMACEUTICALS, INC | PA/USA | Specification Developer |
| IDS MANUFACURING CO., LTD. | THAILAND | Contract Manufacturer |
| INDUS MEDICARE LIMITED | INDIA | Manufacturer |
| INNOLATEX (THAILAND) LIMITED | THAILAND | Manufacturer |
| INNOLATEX SDN. BHD | MALAYSIA | Manufacturer |
| J&J Healthcare Products Div McNeil-PPC, Inc. | NJ/USA | Specification Developer |
| J. KNIPPER AND COMPANY, INC. | NJ/USA | Repackager/Relabeler |
| J.K. ANSELL, LTD. | INDIA | Manufacturer |
| JUST PACKAGING INC. | NJ/USA | Repackager/Relabeler |

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| KARE KITS INC. | CANADA | Repackager/Relabeler |
| KAREX INDUSTRIES SDN BHD | MALAYSIA | Manufacturer |
| LINE ONE LABORATORIES INC. (USA) | CA/USA | Repackager/Relabeler |
| MANEXIM MULTICORP LTD. | CANADA | Repackager/Relabeler |
| MAPA GMBH | GERMANY | Manufacturer |
| MAYER LABORATORIES | CA/USA | Repackager/Relabeler; Specification Developer |
| NAKED INTERNATIONAL INC. | FL/USA | Specification Developer |
| NAVAJO MFG. CO. | CO/USA | Repackager/Relabeler |
| NO GLOVE NO LOVE LTD. | JAMAICA | Foreign Exporter |
| NRS GLOBAL PARTNERS SDN BHD | MALAYSIA | Contract Manufacturer; Manufacturer |
| Nulatex Sdn Bhd | MALAYSIA | Contract Manufacturer; Foreign Exporter; Manufacturer |
| OKAMOTO INDUSTRIES, INC. | JAPAN | Contract Manufacturer; Manufacturer |
| PARADISE MARKETING SERVICES | CA/USA | Repackager/Relabeler |
| PJUR GROUP LUXEMBOURG SA | LUXEMBOURG | Specification Developer |
| PLEASURE LATEX PRODUCTS SDN. BHD. | MALAYSIA | Contract Manufacturer; Foreign Exporter; Manufacturer |
| QINGDAO DOUBLE BUTTERFLY GROUP CO., LTD. | CHINA | Manufacturer |
| Qingdao London Durex Co., Ltd. | CHINA | Manufacturer |
| RFSU AB | SWEDEN | Manufacturer |
| RICHTER RUBBER TECHNOLOGY SDN. BHD. | MALAYSIA | Contract Manufacturer; Manufacturer |
| SAFERLIFE PRODUCTS CO.,LTD. | CHINA | Foreign Exporter |

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| SAGAMI MANUFACTURERS SDN. BHD. IPOH FACTORY | MALAYSIA | Foreign Exporter; Repackager/Relabeler |
| SAGAMI MANUFACTURERS SDN.BHD., BATU GAJAH FACTORY | MALAYSIA | Contract Manufacturer; Foreign Exporter; Manufacturer; Repackager/Relabeler |
| SAGAMI RUBBER INDUSTRIES CO., LTD. | JAPAN | Contract Manufacturer; Foreign Exporter; Manufacturer |
| SAN-MAR LABORATORIES, INC. | NY/USA | Contract Manufacturer |
| SHANTOU CITY KIN SENG PLASTIC CO., LTD. | CHINA | Repackager/Relabeler |
| Shenzhen Baoan Xixiang Item Plastic and Metal Factory | CHINA | Repackager/Relabeler |
| SILVER SPOON ENTERPRISE | CA/USA | Repackager/Relabeler |
| SOOKA INC. | CA/USA | Repackager/Relabeler |
| SSL AMERICAS DISTRIBUTION CENTER | SC/USA | Repackager/Relabeler |
| SSL INTERNATIONAL, PLC | UNITED KINGDOM | Specification Developer |
| SSL MANUFACTURING LTD. | THAILAND | Manufacturer |
| SURETEX PROPHYLACTICS (I), LTD. | INDIA | Manufacturer |
| SURETEX, LTD. | THAILAND | Manufacturer |
| SUZHOU COLOUR-WAY ENTERPRISE DEVELOPMENT CO.,LTD | CHINA | Manufacturer |
| TAKASO RUBBER PRODUCTS SDN BHD | MALAYSIA | Manufacturer |
| Thai Nippon Rubber Industry Co., Ltd. | THAILAND | Contract Manufacturer; Manufacturer |
| THAI NIPPON RUBBER INDUSTRY CO., LTD. | THAILAND | Contract Manufacturer; Foreign Exporter; Manufacturer |
| THE FEMALE HEALTH CO. | IL/USA | Specification Developer |
| THE FEMALE HEALTH CO. | UNITED KINGDOM | Manufacturer |

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| The Female Health Company (M) Sdn Bhd | MALAYSIA | Manufacturer |
| THE ORIGINAL CONDOM COMPANY | FRANCE | Foreign Exporter |
| TIMBAR PACKAGING AND DISPLAY | PA/USA | Repackager/Relabeler |
| TRIGG LABORATORIES, INC. | CA/USA | Repackager/Relabeler |
| TTK - LIG LTD. | INDIA | Manufacturer; Repackager/Relabeler |
| ULTRA-PAK, INC | SC/USA | Repackager/Relabeler |
| UNIDUS CORP. | KOREA, REPUBLIC OF | Manufacturer |
| UTAH MEDICAL PRODUCTS, INC. | UT/USA | Manufacturer |
| VAST RESOURCES INC. | CA/USA | Manufacturer |

Note. A number of the companies listed in this table do not actually manufacture condoms but only repackage, relabel, export, or develop formulas for products.