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ENVIRONMENTAL FORENSIC INVESTIGATION OF SUBSURFACE PIPES CONTAINING TAR RESIDUES NEAR A FORMER MGP IN ASHLAND, WI

- FINAL REPORT -

Submitted to:

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Spooner, WI 54801

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Prepared by:

Stephen Emsbo-Mattingly
Battelle Memorial Institute
397 Washington Street
Duxbury, MA 02332
(781) 934-0571

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1.0 OBJECTIVE

The purpose of this project was the detailed characterization of the hydrocarbons in non-aqueous phase liquid (NAPL), solid, and water samples collected from multiple subsurface pipes located between a former manufactured gas plant (MGP) site in Ashland, WI and selected down gradient locations proximate to the shoreline of Lake Superior. The data collected during this project provided the basis to determine whether the tar residue in the subsurface pipes was a source of elevated polycyclic aromatic hydrocarbons (PAH) measured in the selected down gradient locations. Battelle Memorial Institute (Battelle) conducted an environmental forensic investigation tailored for the analysis of MGP-derived tar and petroleum source signatures to satisfy these objectives. Its high-resolution chemical "fingerprinting" methods (GC/FID and GC/MS) focused on the aromatic and aliphatic composition of tar and petroleum thought to be present in the samples.

2.0 BACKGROUND

The Ashland MGP operated from the late 1800's to the 1940's as a manufactured gas plant using the carbureted water gas process. Coal, coke and/or petroleum feed stocks were presumably delivered to the facility via a nearby railroad line. A lumber mill that operated on the shore of Lake Superior was located topographically and hydrologically down gradient from the Ashland MGP site. This lumber facility generated a considerable quantity of wood scrap that, over the years, was distributed throughout the local coastline area. Approximately 100 feet of shoreline immediately down gradient from the Ashland MGP was filled since the MGP began operating.

Today, the subsurface soils, groundwater and sediments down gradient from the Ashland MGP are contaminated with PAH. Currently, it is not known if the PAH detected around the Lake Superior shoreline are from one or more sources. Past investigations at the site determined that the aliphatic and aromatic hydrocarbon composition of selected up and down gradient samples were significantly different, thereby suggesting the MGP operation was not the source of the PAH in the down gradient locations. However, this conclusion was not based on detailed (molecular level) "fingerprinting" data and was issued prior to the recent investigation of potential migration pathways leading from the former MGP toward Lake Superior.

This investigation revealed the presence of multiple subsurface pipes that potentially connected the former MGP with the former shoreline of Lake Superior. The buried steel and clay pipes were traced, uncovered, and sampled at different locations on three occasions. In July 2001, samples were collected from the down gradient section of the clay pipe and in the immediate vicinity of the discharge (seep) area. In addition, one monitoring well location above, and two monitoring well locations below the clay pipe were sampled (Figure 1). In September 2001, samples were collected from several up gradient sections of the buried clay pipe (Figure 1). Finally, in November 2001, samples were collected from two steel pipes of a different diameter that were located up gradient from the clay pipe and proximate to the former MGP site. Collectively, all of these samples were analyzed for the purpose of characterizing the hydrocarbon residues in and around the various pipes and comparing them to the water and solid samples collected down gradient from the pipes in the potential release areas.

3.0 METHODS

A summary of the methods for sample preservation and analysis at Battelle are presented in this section. Complete documentation of the chemical analysis described in this report, including a Quality Management Plan, a Quality Assurance Project Plan (QAPP), Standard Operating Procedures, chain-of-custody records, and raw and reduced data logs are archived at Battelle Memorial Institute in Duxbury, MA.

3.1 Sample Collection and Shipping

Representatives of Short Elliot Hendrickson, Inc. (SEH) and Wisconsin Department of Natural Resources (WDNR) collected and shipped sixteen samples in three shipments from July to November 2001. The matrices included NAPL, solid (soil and/or solidified organic material), and aqueous (water and/or sheens) residues. The samples were shipped by overnight courier in sample containers fitted with Teflon-lined caps. The three sample deliveries were received at Battelle at temperatures of < 4°C, 1.3°C and ambient temperature, respectively. The samples were received and stored according to the Battelle chain-of-custody procedures. The field chain of custody and sample receipt information is provided in Attachment 10.

The field sample identifications, locations, collection dates, and matrices for the samples studied are summarized in Table 1a. The field sample ID's were abbreviated (see Table 1b) to simplify the tables and figures used for data presentation. Also, in tables and figures throughout this report the samples are listed in order from approximately the most up gradient locations (i.e., nearest to Ashland MGP) to the most down gradient locations (i.e., nearest to Lake Superior).

3.2 Chemical Analysis

The samples were prepared and analyzed in accordance with published methods (Stout et al., 2002) and as described in the QAPP. A summary of the analytical parameters is presented in Table 1b.

3.2.1 Sample Preparation

A mass of solid sample (5 to 10 g) was fortified with surrogates, dried with sodium sulfate and serially shake extracted with dichloromethane (DCM). Less sample mass (0.05 g to 2 g) was used for samples with a large quantity of organic material. Approximately one liter of water (sometimes containing sheens) was extracted by separatory funnel using DCM. The sample extracts were concentrated by Kuderna-Danish and nitrogen blow down techniques. The NAPL samples were diluted to approximately 10 mg/ml in DCM. The surrogates (naphthalene-d8, phenanthrene-d10, and chrysene-d12) were quality control compounds added at the beginning of the method and used to document performance. The sample extracts and diluents were fortified with internal standards and split for GC/FID and GC/MS/SIM analyses (described below).

3.2.2 High Resolution Hydrocarbon Fingerprinting (GC/FID)

The sample extracts were analyzed using a high-resolution gas chromatograph equipped with a flame ionization detector (GC/FID). High-resolution hydrocarbon fingerprints were generated over a broad carbon range (C_9-C_{40}) that provided an overall assessment of the non-volatile hydrocarbons present in each sample, including the primary PAH analytes of concern. The GC/FID fingerprints for each field and QC sample are found in Attachment 3. These fingerprints provided information on the overall nature of the extractable hydrocarbons that were present, including the general PAH composition, variability, and degree of weathering. .

3.2.3 Diagnostic PAH and SVOC (GC/MS/SIM)

The sample extracts were also analyzed using a high-resolution gas chromatograph equipped with a mass spectrometer operated in the selected ion monitoring mode (GC/MS/SIM). The instrument was calibrated to allow for quantification of a broad range of 2- through 6-ring PAH, selected alkylated PAH homologues, selected sulfur-containing compounds (benzothiophenes and dibenzothiophenes), and other compounds useful for the identification of hydrocarbon sources in the environment. An inventory of the target compounds is found in Table 2, along with abbreviations used in selected figures in this report. The concentration of target compounds in the field samples are presented in Attachment 4. Collectively, the concentration of these target compounds are useful for qualitatively and quantitatively relating samples from the suspected source and release areas.

3.2.4 Biomarker Fingerprints (GC/MS/SIM)

Environmental forensic investigators have demonstrated that the presence and/or pattern of biomarkers can reveal information about the specific source(s) of petrogenic residues in the environment; e.g., petroleum or coal (Stout *et al.*, 2002). For this project, selected biomarker fingerprints were qualitatively generated using GC/MS/SIM that monitored for diagnostic fragment ions. These analyses were conducted on the same sample extracts prepared for the measurement of PAH. While the GC/MS was not calibrated for the full suite of biomarkers, several quality control measures for tuning, linearity and mass discrimination ensured that the acquired biomarker patterns were suitable for a qualitative comparison among the samples.

4.0 RESULTS

The high-resolution hydrocarbon fingerprints, quantitative PAH data, PAH histograms, and biomarker fingerprints are presented in Attachments 3 through 9. In the following sections, the data are described with the project objective in mind, i.e., determination of chemical relationships among the field samples.

4.1 High-Resolution Hydrocarbon Fingerprints

Resolved peaks representing the parent (non-alkylated) PAH, ranging from naphthalene to benzo(g,h,i)perylene, constituted the dominant feature of the high-resolution hydrocarbon fingerprints (Attachment 3). The distribution and predominance of parent PAH in each of the field samples was entirely consistent with those derived from MGP tar. (See the fingerprint for a coal tar reference material, ZL44CT, near the end of Attachment 3, for comparison). The hydrocarbon fingerprints reveal no obvious evidence for the presence of any other source of hydrocarbons in the samples studied.
The fingerprints do reveal that the primary variation among the field samples was in the degree of environmental weathering. The degree of weathering was most easily recognized by comparing the number and height of resolved peaks eluting before Androstane[1] (i.e., light or lower molecular weight PAH) relative to those eluting after Androstane (heavy or higher molecular weight PAH) (Figure 2). As expected given the greater susceptibility of lower molecular weight PAH to environmental weathering, those samples exhibiting a more highly weathered tar fingerprint were relatively depleted in lower molecular weight PAH and *vice versa* (Figure 2).

Although the samples did vary in the degree of weathering (Figure 2), overall they were only slightly-to-moderately weathered, as evidenced by the presence of light, lower molecular weight compounds in even the most highly weathered field samples (e.g. MW-7 in Figure 2d). Even though sample MW-7 exhibited

[1] The peaks labeled OTP (surrogate) and Androstane (internal standard) are quality control compounds that should otherwise be visually ignored for the interpretation of the hydrocarbon fingerprints.

Inspection of the PAH histograms illustrates a high degree of similarity among the samples collected from the pipes and those collected from the release areas (e.g., Figure 3). With one exception, all of the samples exhibited very similar patterns among the highest molecular weight (i.e. most recalcitrant) PAH. The recalcitrant, highest molecular weight PAH were functionally defined as 5- and 6-ring PAH (benzofluoranthenes through benzo(g,h,i)perylene; Table 2). The relative distributions among these PAH reflect the specific conditions of their formation; for example, the carburetion procedure/conditions and feedstock. The East Riser water sample deviated from the common pattern of heavy PAH and exhibited a PAH profile (and hydrocarbon fingerprint) that was consistent with the water-soluble fraction of MGP tar.

Some subtle differences among the samples were observed in the 4-ring PAH group (fluoranthene through chrysene in Table 2). Specifically, the samples from the steel pipe exhibited slightly lower relative abundances of parent to alkylated PAH within the 4-ring group. This difference was significant given the absence of severe PAH weathering (see hydrocarbon fingerprint discussion above); consequently, weathering of these stable analytes was unlikely. Instead this difference tends to indicate the presence of a slightly different type of tar in the steel pipe sampling locations (as compared to tar in the clay pipe locations). This slightly different type of tar in the steel pipe areas would appear to have been formed from a different or modified carburetion process and/or feedstock.

Obvious differences were observed in the PAH histograms among the 2- to 3-ring PAH (Figure 3). As expected, these differences were consistent with range of environmental weathering evident in the hydrocarbon fingerprints. While MGP tars formed differently can exhibit some source specific PAH distributions in this range (e.g., Figure A5-2 in Attachment 5), the potential effects of weathering in this molecular weight range complicates any interpretation. Nevertheless, close inspection of the PAH histograms reveals that the steel pipe samples contained a slightly higher abundance of dibenzofuran relative to fluorene than the samples from the clay pipe and down gradient release area. However, the samples from the clay pipe and the potential release area (MW-7 and TW-9) were very similar.

4.3 Principal Components Analysis of PAH Data

Principal components analysis (PCA) is a numerical analysis tool that is commonly used to explore the complex chemical differences or similarities among many samples. In this study, PCA was used as an exploratory tool to visualize the inter-sample and inter-variable relationships among the PAH dataset. It was performed by using the Ein*Sight software product (Version 4.0; Infometrix, Inc., Seattle, WA). PCA output includes a distribution of samples (e.g., pipe and release area samples) in n-dimensional space, where n was the number of input variables (e.g., PAH analytes or ratios). The Euclidean distance between sample points on the resulting factor score plots represented the variance captured in each principle component (PC). In simple terms, samples that cluster together are considered chemically similar and outliers are considered chemically distinct. A factor loading is simultaneously calculated for each variable (e.g., PAH analyte) contributing to each PC. A cross plot of the factor loadings for the corresponding PCs revealed the individual variables (PAH analytes or ratios) that were responsible for the variance in each PC. The PAH analyte concentrations or ratios were normalized prior to PCA in order to minimize the influence of concentration. Thus, the PCA focused on distinguishing the similarities and differences in PAH patterns irrespective of analyte concentration.

The chemical signatures of different pyrogenic materials (e.g. MGP tars) can be illustrated by comparing the relative content among functional groups that are preferentially pyrolyzed or condensed during industrial carburetion (Emsbo-Mattingly *et al.*, 2001). Useful parameters include the relative abundance of (1) PAH with variously alkylated hydrocarbon side-chains (e.g., parent PAH *versus* C2- and C3 alkylated PAH), (2) sulfur-containing PAH (e.g., dibenzothiophenes), and (3) 5-ring PAH with different ring structures. In general, ratios that capture the relative abundance of these functional groups, as well as

selected ratios among various 6-ring PAH of similar molecular weight, can be used to distinguish between different types of MGP wastes in the environment (Emsbo-Mattingly et al., 2001). Thus, selected and diagnostic ratios were calculated for each of the field samples. These ratios were used as input variables for PCA, allowing for the detailed comparison among the samples (Table 4).

The factor score plot of the first and second principal components is shown in Figure 4a. The corresponding factor loadings plot is shown in Figure 4b. Recall, samples that plot near to one another in the factor score plot are chemically similar, whereas those that plot apart are chemically distinct. The clustering of samples in the upper left hand quadrant of the factor score plot includes samples from the clay pipe and from the down gradient release area (Figure 4a). This clustering indicates that the MGP tar in the clay pipe samples was chemically comparable to the samples from the down gradient release area. For example, many of the samples collected from the clay pipe grouped closely with the down gradient sample TW-9 (Figure 4a). Thus, the clay pipe appears to be a likely source of PAH in samples collected from the down gradient release area.

PCA determined that the East Riser sample was unique among those studied (Figure 4a). Recall, this was the sample believed to contain the water-soluble fraction of MGP tar (see above). As might be expected, the factor loadings plot (Figure 4b) reveals the East Riser sample was enriched in parent PAH (relative to alkylated PAH), with a particular enrichment of light, low molecular weight parent PAH (i.e., naphthalene, phenanthrene, and anthracene). As revealed by the hydrocarbon fingerprint and PAH histogram, the PCA results reflect the unique presence of the water-soluble fraction of MGP tar in this sample.

PCA also revealed that the two samples (one a duplicate) of solidified tar collected around the clay pipe discharge were slightly depleted in light, low molecular weight PAH (Figure 4). These samples are slightly offset from the other clay pipe and discharge (water) samples. Environmental weathering has likely caused this depletion of light PAH in the solid discharge samples. The reduction of light PAH, combined with the elevated relative abundance of sulfur-containing PAH (dibenzothiophenes), distinguished MW-7 from the clay pipe samples (Figure 4). The reduction in light PAH in this sample was also attributed to weathering. Recall, the MW-7 sample was the most weathered sample studied (Figure 3). The relative enrichment of dibenzothiophenes in the MW-7 sample may also be due to weathering, or may indicate the trace presence of a localized release of uncombusted fuel and gas oil.

Finally, PCA revealed that the two steel pipe samples were comparable to one another, but exhibited slightly different distributions of diagnostic source ratios than those from the clay pipe (Figure 4a). This suggests that the MGP tar in the steel pipe area is distinct from the MGP tar from the clay pipe. Figure 4b shows that the MGP tar from the steel pipes were differentiated in part by the elevated ratio of dibenzofuran to fluorene and acenaphthylene to acenaphthene and a higher relative abundance of alkylated PAH.

4.4 Biomarker Fingerprints

Four categories of biomarker fingerprints were used to determine the type of petrogenic residues present in the samples. The categories included normal alkanes (as determined by m/z 85 mass chromatograms), alkylcyclohexanes (m/z 83), terpanes (m/z 191), and steranes (m/z 217). These fingerprints provided detailed chemical information that spans a wide molecular weight range. They can be found in Attachments 7 through 9. These fingerprints are useful in the detection and characterization of different petroleum (and perhaps coal) sources, as were used during the operational history of former MGPs. The normal alkanes and alkylcyclohexanes span almost the entire hydrocarbon range from nC9 to nC36. By contrast, the terpanes and steranes are generally found in crude oil and in heavier fuel and lubricating oils.

However, a great deal of information is available on the relationship of these recalcitrant analytes to the processes that generated the petroleum

The normal alkane and alkylcyclohexane fingerprints for the samples studied revealed four basic patterns (Figure 5; Attachment 7). First, the two samples from the steel pipe exhibited a relatively narrow range of normal alkanes in the nonane (nC9) to nonadecane (nC19) range (Figure 5a). Second, the East Riser sample featured trace, if any, normal alkanes amidst the high concentrations of light PAH (Figure 5b). However, trace levels were detected between nC22 and nC33. Third, the bulk of the clay pipe samples contained a wide range, middle distillate petroleum product(s) signature ranging from nC9 to nC27 with a maximum around nC12 to nC13 (e.g. Figure 5c). Fourth, the Solids Around the Discharge Pipe, MW-7, and TW-9 exhibited the same wide range petroleum product(s) signature that the clay pipe samples did, but this appears to be superimposed on another signature exhibiting elevated levels of isoprenoid hydrocarbons (e.g., Figure 5d). This pattern is consistent with a mixture of a relatively weathered petroleum product(s) enriched in the more recalcitrant isoprenoids and an unweathered (or less weathered) petroleum product(s) that is relatively enriched in normal alkanes.

It is notable that close inspection of the m/z 85 mass chromatograms (Attachment 7) reveals that the abundance of heptadecane relative to pristane (nC17/Pr) declined from its highest level at TW-13 (up gradient) to its lowest level in MW-7 (down gradient), while exhibiting intermediate though varying levels throughout the length of the clay pipe. The relative reduction of heptadecane with increasing distance down gradient would be consistent with increasing levels of biodegradation in at least some petroleum components and/or relative abundance of the more highly weathered petroleum component recognized in MW-7.

Inspection of the m/z 191 mass chromatograms (Attachment 8) revealed four terpane fingerprints patterns that are represented in Figure 6. First, the two steel pipe samples contained no terpanes above the analytical detection limit (Figure 6a). This finding is consistent with the previous observation that these samples contained a lighter and narrower range (nC9-nC19) petroleum product that would not be expected to contain these terpane biomarkers. Second, the majority of samples collected from the clay pipe and down gradient locations contained a full suite of terpane biomarkers (e.g., Figure 6b). Their presence (along with the n-alkane patterns described above) indicates the likely presence of a heavier fuel or gas oil in the clay pipe and down gradient samples. Third, the Pipe Sludge Center of Yard and Scrapings from Inside Discharge contained the pentacyclic triterpane, oleanane, which suggested that the petroleum in these samples originated from a separate source from, or contains a petroleum component that is distinct from, those observed in the clay pipe and discharge area. Fourth, the higher abundance of the biomarker Ts relative to Tm in sample MW-7 was considered characteristic of an independent source of petroleum. This interpretation was consistent with the localized elevation of dibenzothiophenes measured in MW-7 and supported the assertion that a second type of petroleum may have impacted this sampling location.

The low levels of steranes prevented a thorough interpretation.

5.0 CONCLUSIONS

Advanced chemical analyses were performed on each site sample. The results of these analyses generated the following conclusions:

- The high-resolution fingerprints indicated the presence of MGP tar in all the field samples.
- The distribution of PAH in all field samples was most consistent with a carbureted water gas tar.

- With the exception of the East Riser sample, the PAH histograms indicated that the samples were all very similar to one another regardless of their collection location.
- The East Riser sample was unique and appeared to be the water-soluble fraction of MGP tar. This pattern suggested that the aqueous dissolution of tar was an active weathering process within the pipeline system. Supporting this contention was the fact that the trace pattern of heavy, high molecular weight PAH in the East Riser sample matched other samples from the clay pipe system.
- Although the patterns of PAH were similar among the field samples, a detailed inspection of the PAH histograms and principal components analysis helped to differentiate the two, up gradient steel pipe samples from remaining down gradient field samples. This difference suggested that the MGP tar in the steel pipe area was distinct from that in the clay pipe system. The difference likely indicated that the tar in the steel pipe was produced under slightly different operating conditions and/or from a different feedstock.
- Differences in the MGP tar residues among the samples collected from the clay pipe and down gradient locations were attributed to environmental weathering. The heavy molecular weight portion of the tar in the clay pipe and down gradient samples, i.e., that portion least affected by weathering, was nearly identical and indicated a common source.
- While the more highly weathered PAH pattern of MW-7 still resembled the pattern in the clay pipe samples, the slightly higher relative abundance of sulfur-containing PAH and a diagnostic terpane biomarker pattern revealed the possible coexistence of uncombusted fuel or gas oil at this location.
- The petrogenic residues in the field samples were more variable than the MGP tar residues. The petrogenic residues could be qualitatively differentiated into five potential sources. Distinct petroleum patterns were detected in the steel pipe samples (relatively light weight), East Riser sample (trace level biomarkers similar to heavy weight range of the fuel oil measured in the other clay pipe samples), remaining clay pipe samples listed in Table 1 (wide range fuel or gas oil), MW-7 sample (similar to clay pipe samples with elevated an level of Ts relative to Tm), down gradient samples including Solids Around Discharge Pipe, MW-7, and TW-9 (elevated isoprenoid hydrocarbons overprinted on clay pipe pattern suggestive of mixed unweathered and weathered petroleum products).
- The varying abundance of heptadecane relative to pristane among the clay pipe and down gradient samples indicated that the weathering state of the petroleum in the clay pipe system may have been influenced by biodegradation, generally increasing in a down gradient direction.

The pyrogenic (PAH) and petrogenic (biomarkers) patterns independently identified two types of MGP tars in the field samples. The first type of tar was located in the steel pipes and the second type was located in both the clay pipe system and in the down gradient sampling stations. The petrogenic (biomarker) patterns also indicated the presence of residual petroleum refined from different crude oils. The weathering patterns observed included the dissolution of PAH (i.e., formation of a water-soluble fraction) and the biodegradation of petroleum. This finding did not exclude the possible existence of other weathering processes.

6.0 SUMMARY

Based on the conclusions stated in Section 5.0, this section proposes a conceptual site model that could account for the analyte distributions recorded in this report.

- During the operation of the former MGP, tar was transported via the subsurface pipes that were sampled during July, September, and November of 2001. The tar signatures detected in these pipes were consistent with one another and with the chemical signature of carbureted water gas

tars reported in the literature; for example, the ratio of fluoranthene to pyrene was less than 1.0 (Emsbo-Mattingly *et al.*, 2001; EPRI, 1993).

- The clay pipe(s) likely permitted the release of waste tar from an up gradient location near the former MGP in Ashland WI to approximately the former shoreline of Lake Superior. The samples collected down gradient of the pipes were chemically similar to the clay pipe samples. Consequently, the clay pipe samples and down gradient samples contain MGP tar(s) that likely originated from a similar carburetion process. The outer perimeter of this clay pipe signature extended minimally to the TW-9 sample collection station. Once the waste tar was discharged from the clay pipe, it may have migrated or been transported to proximate locations, like TW-9, around the former shoreline of Lake Superior as dictated by the developmental history of the shoreline area.
- Some variability existed in the relative abundance of the light constituents of these tars due to multiple weathering processes; for example, the East Riser sample, which was comprised of the water-soluble fraction of MGP tar, demonstrated that the dissolution of PAH from the tar had occurred and/or the declining abundance of heptadecane relative to pristane indicated the biodegradation of petroleum residue had occurred.
- Sample MW-7 differed chemically from the pipe samples in terms of its higher relative content sulfur-containing and alkylated PAH. A localized release of petroleum in the vicinity of MW-7 may have enriched these analytes. This sampling location may have been influenced by releases associated with gas oil supply transfers from the railroad.
- The tar samples collected in November 2001 from the 2" and 12" steel pipes were chemically distinct from samples collected from other locations and may reflect an alternate carburetion feed stock, modified MGP operating condition, or equipment upgrades that occurred commonly during the history of former MGPs (Emsbo-Mattingly *et al.*, 2001).
- The biomarker patterns varied occasionally within the clay pipe samples and among samples with similar PAH patterns. This finding suggested that the MGP used multiple sources of gas oil in the carburetor unit.

7.0 REFERENCES

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EPRI (1993). Chemical and Physical Characteristics of Tar Samples from Selected Manufactured Gas Plant (MGP) Sites. Electric Power Research Institute Publication TR-102-184.

Stout, S.A., Uhler, A.D, McCarthy, K.J. and Emsbo-Mattingly, S.D. (2002) Chemical Fingerprinting of Hydrocarbons. In: Introduction to Environmental Forensics, B. Murphy and R. Morrison, Eds. Academic Press.

Attachment 1

Figures

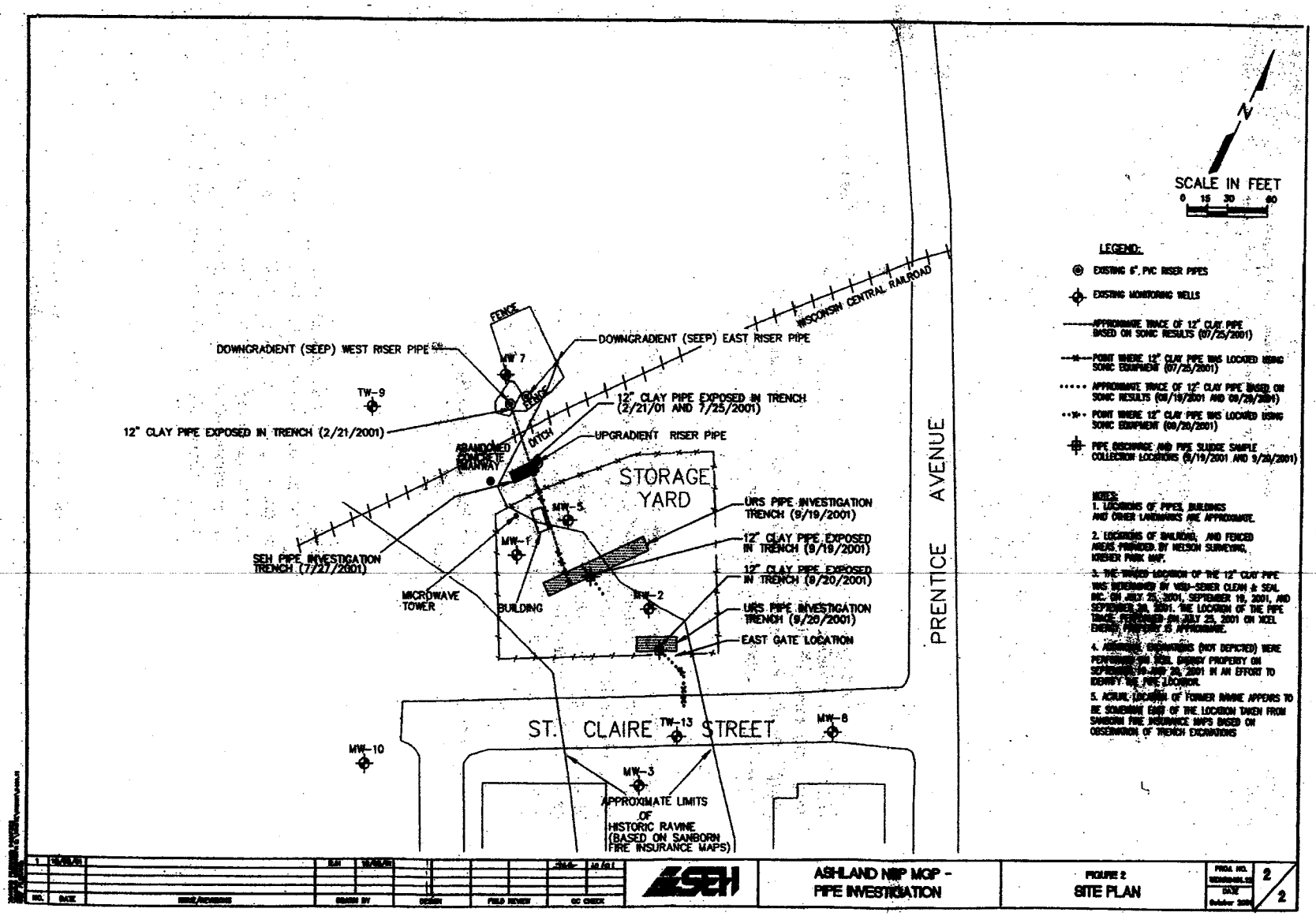

Figure 1. Site Map

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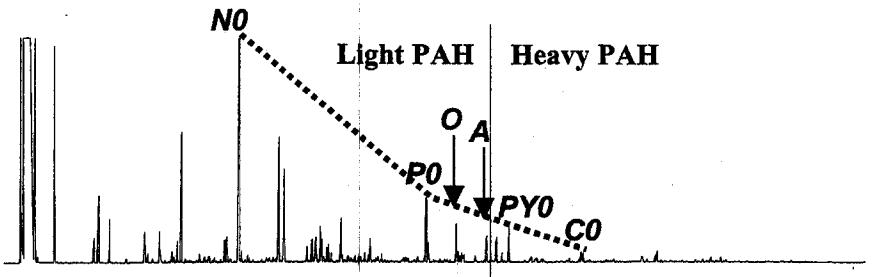
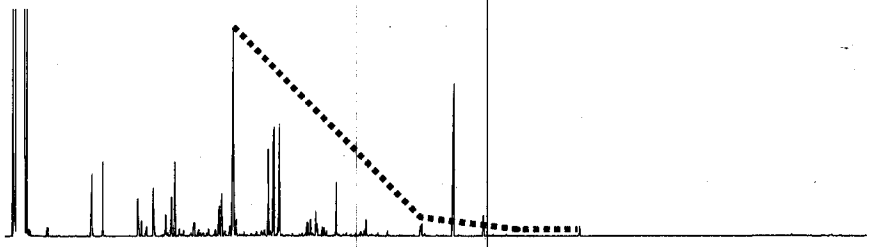
Figure 2.
High Resolution Fingerprints Illustrate Weathering Patterns

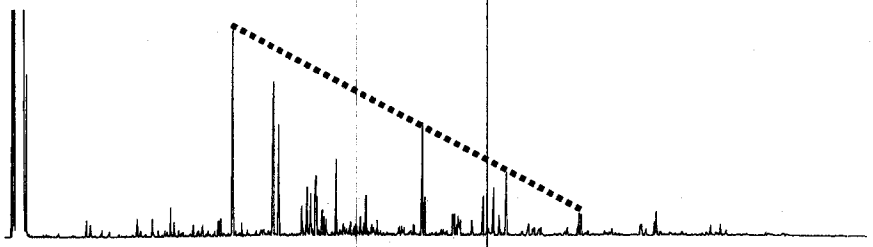
2a **TW-13**
Among Least Weathered of Field Samples
Light > Heavy PAH



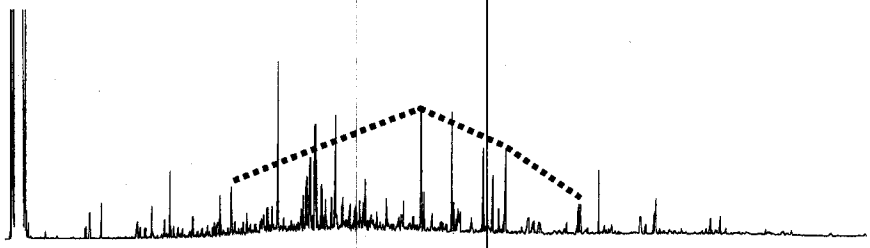
2b. **East Riser**
Water-soluble Fraction/ Drip Oil
Light >> Heavy PAH



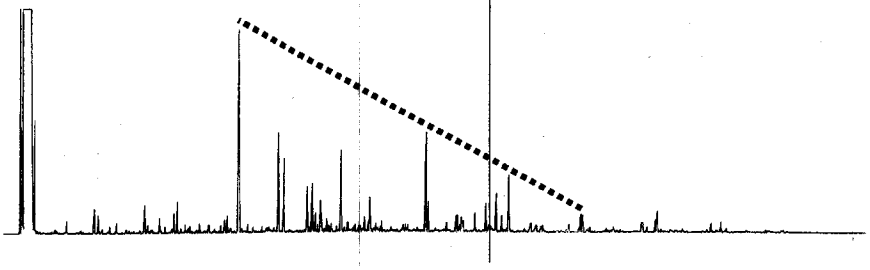
2c. **Pipe Discharge**
Light Weathering
Light > Heavy PAH
Like TW-13 with slightly less naphthalene relative to phenanthrene



2d. **MW-7**
Most weathered of Field Samples
Light > Heavy PAH
Naphthalene < Phenanthrene



2e. **TW-9**
Like Pipe Discharge



N0 – Naphthalene P0 – Phenanthrene PY0 – Pyrene C0 – Chrysene
A – Androstane (Internal Standard) O – ortho-Terphenyl (OTP) (Surrogate)

Figure 3.
PAH Histograms Illustrate Source and Weathering Patterns

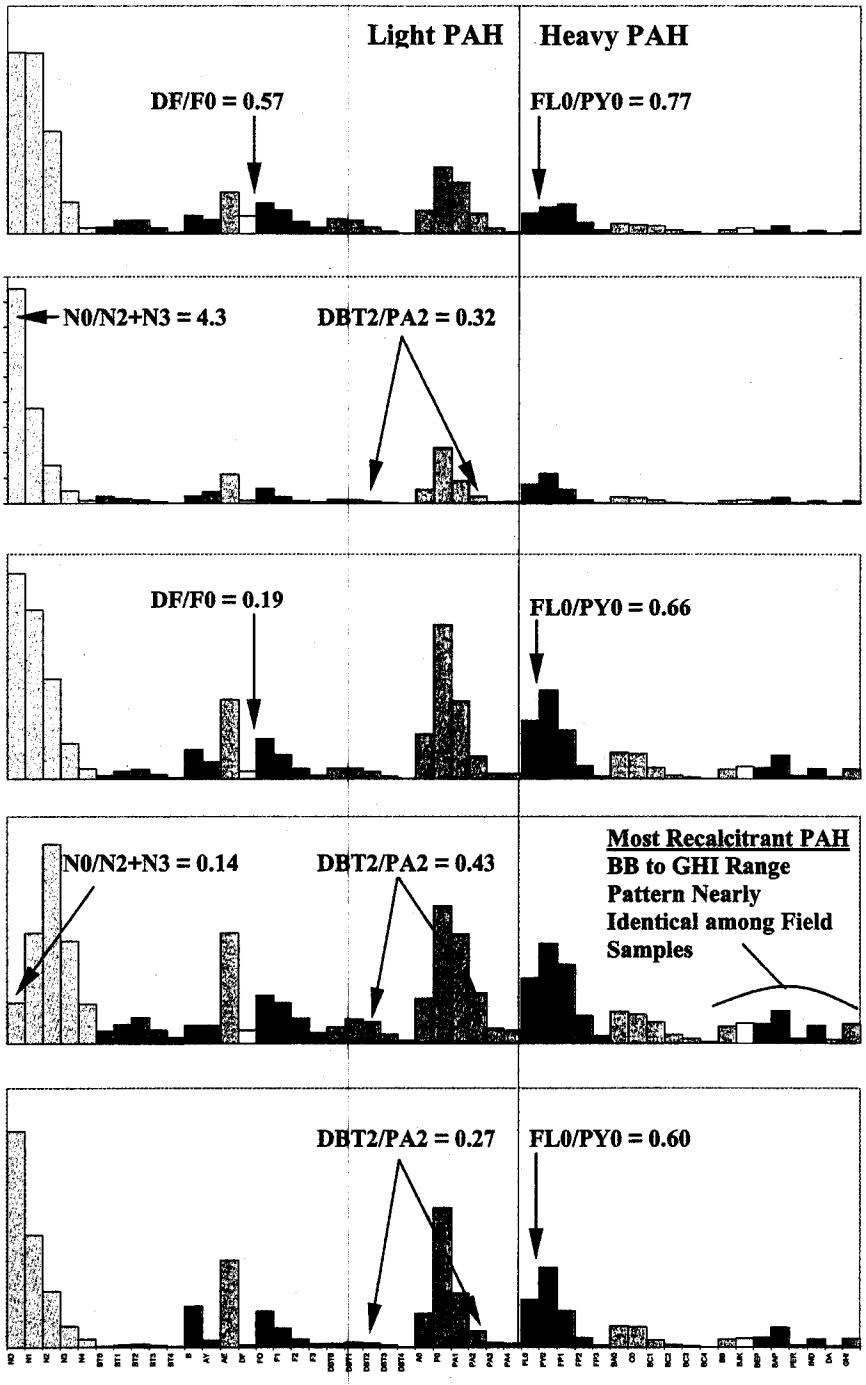
3a. **2" Pipe**
Slightly Different from Clay Pipe Pattern
LPAH/HPAH=5.6

3b. **TW-13**
Among Least Weathered of Field Samples
LPAH/HPAH=5.1

3c. **Pipe Discharge**
Light Weathering
LPAH/HPAH=3.0

3d. **MW-7**
Most weathered of Field Samples
LPAH/HPAH=2.4

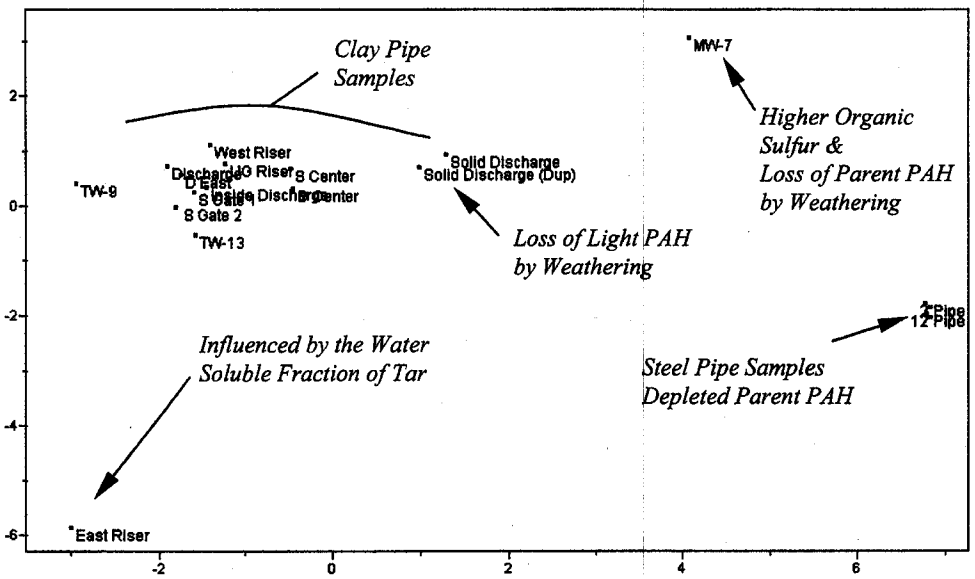
3e. **TW-9**
Like Pipe Discharge
LPAH/HPAH=3.0



LPAH = ΣNaphthalene through Phenanthrenes HPAH = ΣFluoranthene through Benzo(g,h,i)perylene (Attachment 4).
See Table 2 for key to target analyte abbreviations.

Figure 4.
Principal Components Analysis (PCA) Using Selected Source Ratios

4a. Sample Groupings (PCA Scores Plot). Principal components 1 (x-axis) and 2 (y-axis) contain 62% and 23% of the variability, respectively.



4b. Analyte Groupings (PCA Loadings Plot).

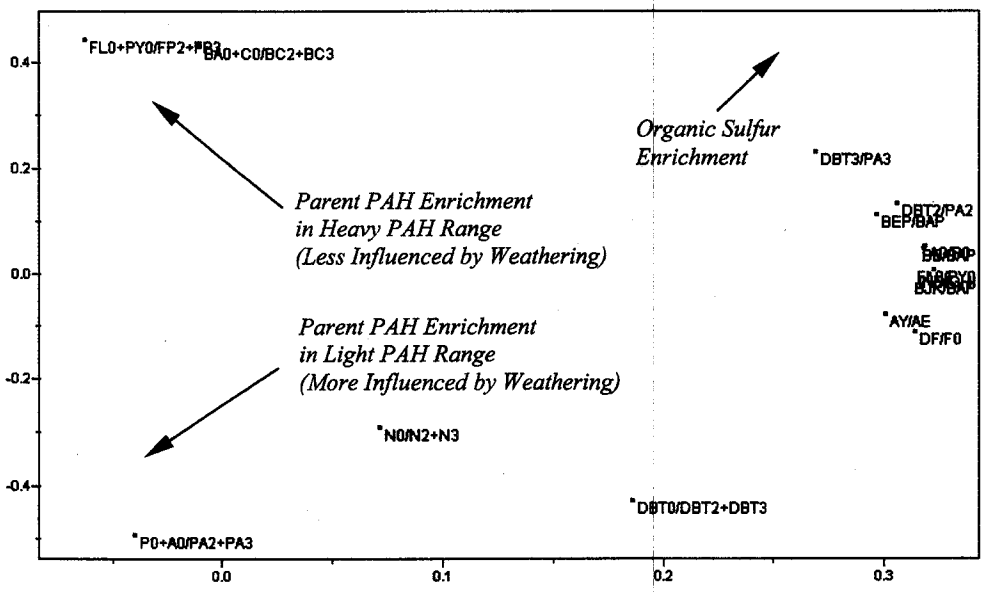
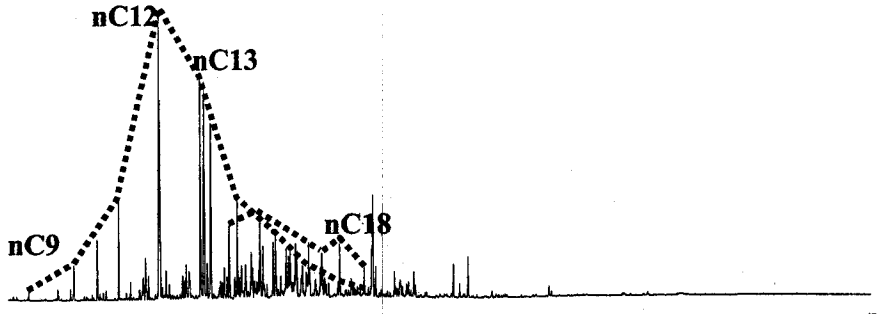
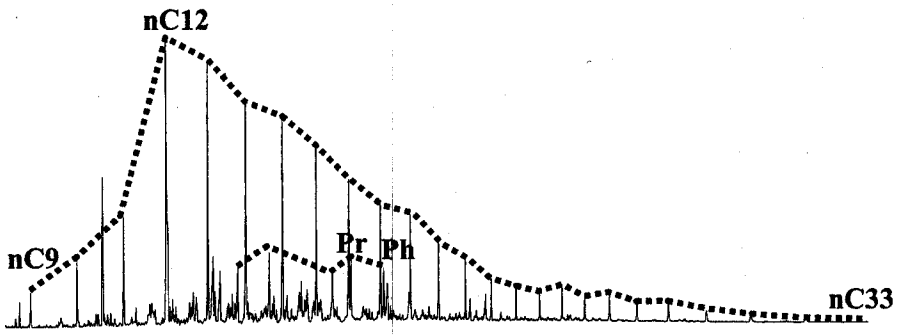


Figure 5.
Normal Alkane Biomarker Fingerprints Reveal Multiple Types of Petroleum

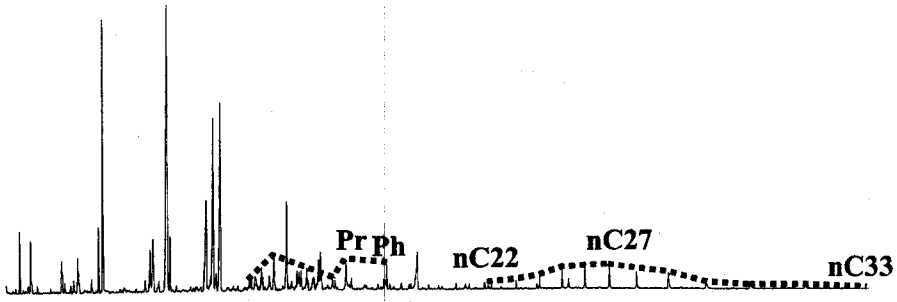
5a. **2" Pipe**
Lighter Weight,
Narrow Range Gas Oil
Weathering state unknown



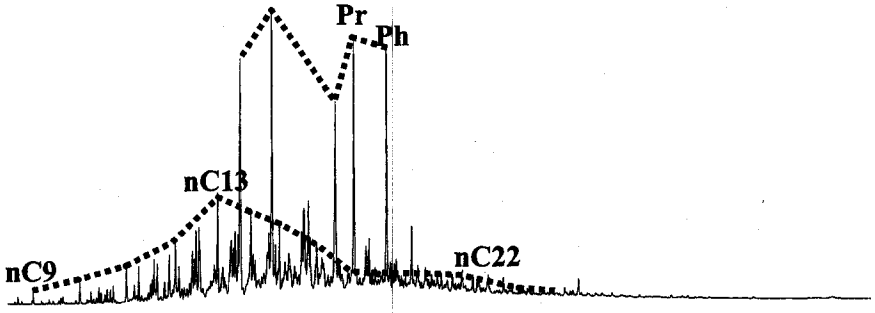
5b. **TW-13**
Wide Range Gas Oil
Relatively unweathered



5c. **East Riser**
Water Soluble Fraction of MGP tar
Light fraction of gas oil (nC9 to nC21) weathered
Heavy fraction of gas oil present



5d. **MW-7**
Mixture of Unweathered Gas Oil (nC9 to nC22) and Weathered Middle to Heavy Weight Petroleum Product (elevated isoprenoids)

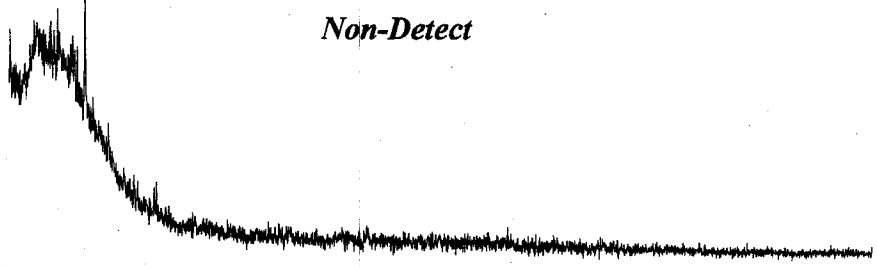


nC# = normal alkane containing # carbons in the chain (Blue Dash) peak to peak profile of the normal alkanes

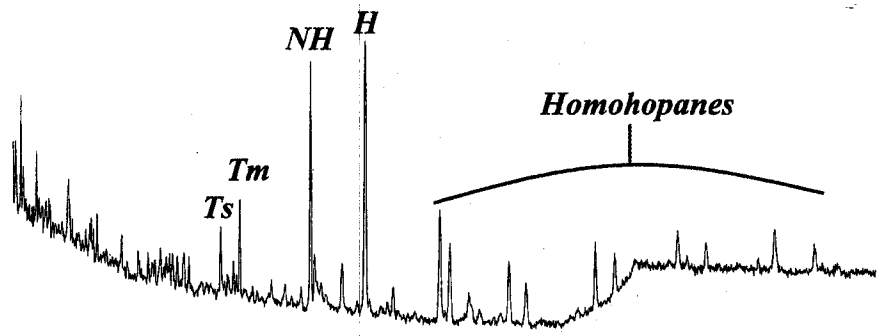
Pr = Pristane (isoprenoid hydrocarbon) Ph = Phytane (isoprenoid hydrocarbon) (Red Dash) peak to peak profile of selected isoprenoid hydrocarbons

Figure 6.
Terpane Biomarker Fingerprints Reveal Multiple Types of Petroleum

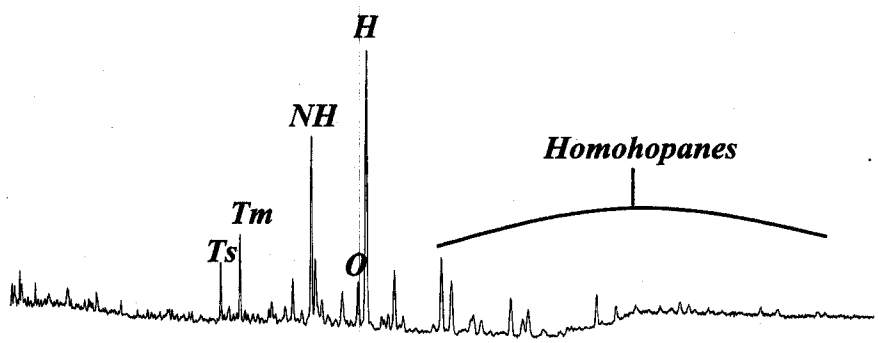
6a. **2" Pipe**
Lighter Weight,
Narrow Range Gas Oil
No Terpanes in Distillate



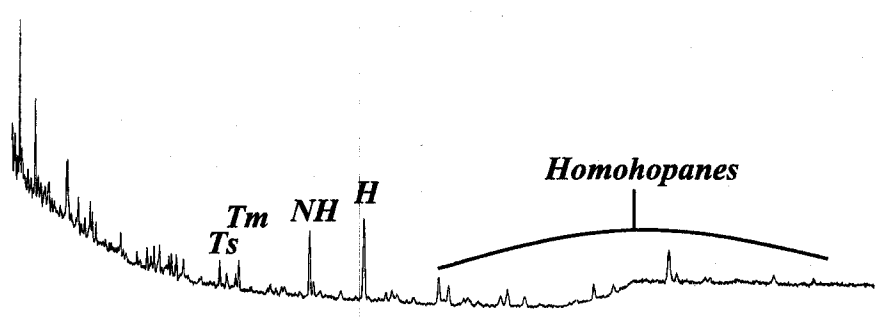
6b. **TW-13**
Wide Range Gas Oil
Relatively unweathered
Common pattern among field samples



6c. **East Riser**
Water Soluble Fraction of MGP tar
Biomarkers in heavy fraction of gas oil
Presence of oleanane indicates an additional petroleum source



6d. **MW-7**
Mixture of Unweathered Gas Oil and Weathered Middle to Heavy Weight Petroleum Product
Higher relative abundance of Ts suggests multiple petroleum sources



Ts = 18α(*H*),21β(*H*)-22,29,30-trisnorhopane Tm = 17α(*H*),21β(*H*)-22,29,30-trisnorhopane
NH = 17α(*H*),21β(*H*)-30-norhopane H = 17α(*H*),21β(*H*)-hopane O = 18α(*H*) & 18β(*H*)-oleanane

Attachment 2

Tables

Table 1.
Sample Identification, Collection and Analysis Summary

1a. Sample Collection Summary.

Field Sample ID	Location (Listed ~ Up to Down Gradient)	Collection Date	Matrix
2 Inch Steel Pipe	Steel Pipe Near Former MGP	11/12/01	Oil (NAPL)
12 Inch Steel Pipe	Steel Pipe Near Former MGP	11/12/01	Oil (NAPL)
TW-13	Between MGP and Clay Pipe	7/24/01	Water
Pipe Discharge East Yard Gate	Up Gradient Clay Pipe	9/20/01	Solid
Pipe Sludge East Yard Gate #1	Clay Pipe	9/20/01	Solid
Pipe Sludge East Yard Gate #2	Clay Pipe	9/20/01	Solid
Pipe Discharge Center Of Yard	Clay Pipe	9/19/01	Solid
Pipe Sludge Center of Yard	Clay Pipe	9/19/01	Solid
Upgradient Riser	Clay Pipe	7/24/01	Water
East Riser	Clay Pipe	7/24/01	Water
West Riser	Clay Pipe	7/24/01	Water
Scrapings From Inside Discharge	Clay Pipe	7/25/01	Solid
Pipe Discharge	Down Gradient Terminus of Clay Pipe	7/25/01	Water
Solids Around Discharge Pipe	Proximate Release Area	7/25/01	Solid
MW-7	Intermediate Release Area	7/24/01	Water
TW-9	Most Distant Release Area	7/24/01	Water

1b. Sample Identification and Analysis Summary.

Field Sample ID	Hydrocarbon Fingerprint	Quantitative PAH	Biomarker Fingerprint	Battelle ID	Abbreviation
2 Inch Steel Pipe	X	X	X	W9071	2" Pipe
12 Inch Steel Pipe	X	X	X	W9072	12" Pipe
TW-13	X	X	X	W5548	TW-13
Pipe Discharge East Yard Gate	X	X	X	W6841	D East
Pipe Sludge East Yard Gate #1	X	X	X	W6839	S Gate 1
Pipe Sludge East Yard Gate #2	X	X	X	W6840	S Gate 2
Pipe Discharge Center Of Yard	X	X	X	W6837	D Center
Pipe Sludge Center of Yard	X	X	X	W6838	S Center
Upgradient Riser	X	X	X	W5547	UG Riser
East Riser	X	X	X	W5544	East Riser
West Riser	X	X	X	W5543	West Riser
Scrapings From Inside Discharge	X	X	X	W5551	Inside Discharge
Pipe Discharge	X	X	X	W5549	Discharge
Solids Around Discharge Pipe	X	X	X	W5550	Solid Discharge
MW-7	X	X	X	W5545	MW-7
TW-9	X	X	X	W5546	TW-9

Table 2.
Primary PAH Analytes and Abbreviations

Target Analyte	Abbreviation	
	Individual (Large Histogram)	Group (Small Histogram)
Naphthalene	NO	N
C1-Naphthalenes	N1	
C2-Naphthalenes	N2	
C3-Naphthalenes	N3	
C4-Naphthalenes	N4	
Benzo(b)thiophene	BT0	BT
C1-Benzo(b)thiophenes	BT1	
C2-Benzo(b)thiophenes	BT2	
C3-Benzo(b)thiophenes	BT3	
C4-Benzo(b)thiophenes	BT4	
Biphenyl	B	B
Acenaphthylene	AY	Y
Acenaphthene	AE	E
Dibenzofuran	DF	D
Fluorene	FO	F
C1-Fluorenes	F1	
C2-Fluorenes	F2	
C3-Fluorenes	F3	
Dibenzothiophene	DBT0	DBT
C1-Dibenzothiophenes	DBT1	
C2-Dibenzothiophenes	DBT2	
C3-Dibenzothiophenes	DBT3	
C4-Dibenzothiophenes	DBT4	
Anthracene	A0	PA
Phenanthrene	P0	
C1-Phenanthrenes/Anthracenes	PA1	
C2-Phenanthrenes/Anthracenes	PA2	
C3-Phenanthrenes/Anthracenes	PA3	
C4-Phenanthrenes/Anthracenes	PA4	
Fluoranthene	FL0	FP
Pyrene	PY0	
C1-Fluoranthenes/Pyrenes	FP1	
C2-Fluoranthenes/Pyrenes	FP2	
C3-Fluoranthenes/Pyrenes	FP3	
Benz[a]anthracene	BA0	BC
Chrysene	C0	
C1-Chrysenes	BC1	
C2-Chrysenes	BC2	
C3-Chrysenes	BC3	
Benzo[b]fluoranthene	BB	B
Benzo[j/k]fluoranthene	BJK	K
Benzo[a]fluoranthene	BAF	A
Benzo[e]pyrene	BEP	E
Benzo[a]pyrene	BAP	A
Perylene	PER	P
Indeno[1,2,3-c,d]pyrene	IND	I
Dibenz[a,h]anthracene	DA	D
Benzo[g,h,i]perylene	GHI	B

Table 3.
Total PAH Concentrations (ppm)

Client Sample ID	Matrix	TPAH[1]	Units	Rank[2]	LPAH/HPAH Ratio[3]
2 Inch Steel Pipe	Oil	136000	mg/Kg	1	5.6
12 Inch Steel Pipe	Oil	97800	mg/Kg	2	5.0
TW-13	Aqueous	13.1	mg/L	14	5.1
Pipe Discharge East Yard Gate	Solid	76000	mg/Kg	3	3.2
Pipe Sludge East Yard Gate #1	Solid	16200	mg/Kg	6	3.5
Pipe Sludge East Yard Gate #2	Solid	24000	mg/Kg	5	4.1
Pipe Discharge Center Of Yard	Solid	496	mg/Kg	11	3.0
Pipe Sludge Center of Yard	Solid	919	mg/Kg	10	2.3
Upgradient Riser	Aqueous	39.1	mg/L	13	2.8
East Riser	Aqueous	0.576	mg/L	16	46
West Riser	Aqueous	172	mg/L	12	2.4
Scrapings From Inside Discharge	Solid	29400	mg/Kg	4	3.3
Pipe Discharge	Aqueous	2880	mg/L	8	3.0
Solids Around Discharge Pipe	Solid	1500	mg/Kg	9	2.6
MW-7	Aqueous	2.71	mg/L	15	2.4
TW-9	Aqueous	5520	mg/L	7	3.0

1 - Total PAH (TPAH) is the sum of PAH from naphthalene to benzo(g,h,i)perylene.
2 - Ranking based on highest to lowest TPAH concentration.
3 - Ratio of Light PAH (LPAH = ΣNaphthalene to Phenanthrene) to Heavy PAH (HPAH = ΣFluoranthene to Benzo(g,h,i)perylene) estimates the fraction of PAH that are potentially most subject to alteration by environmental weathering.

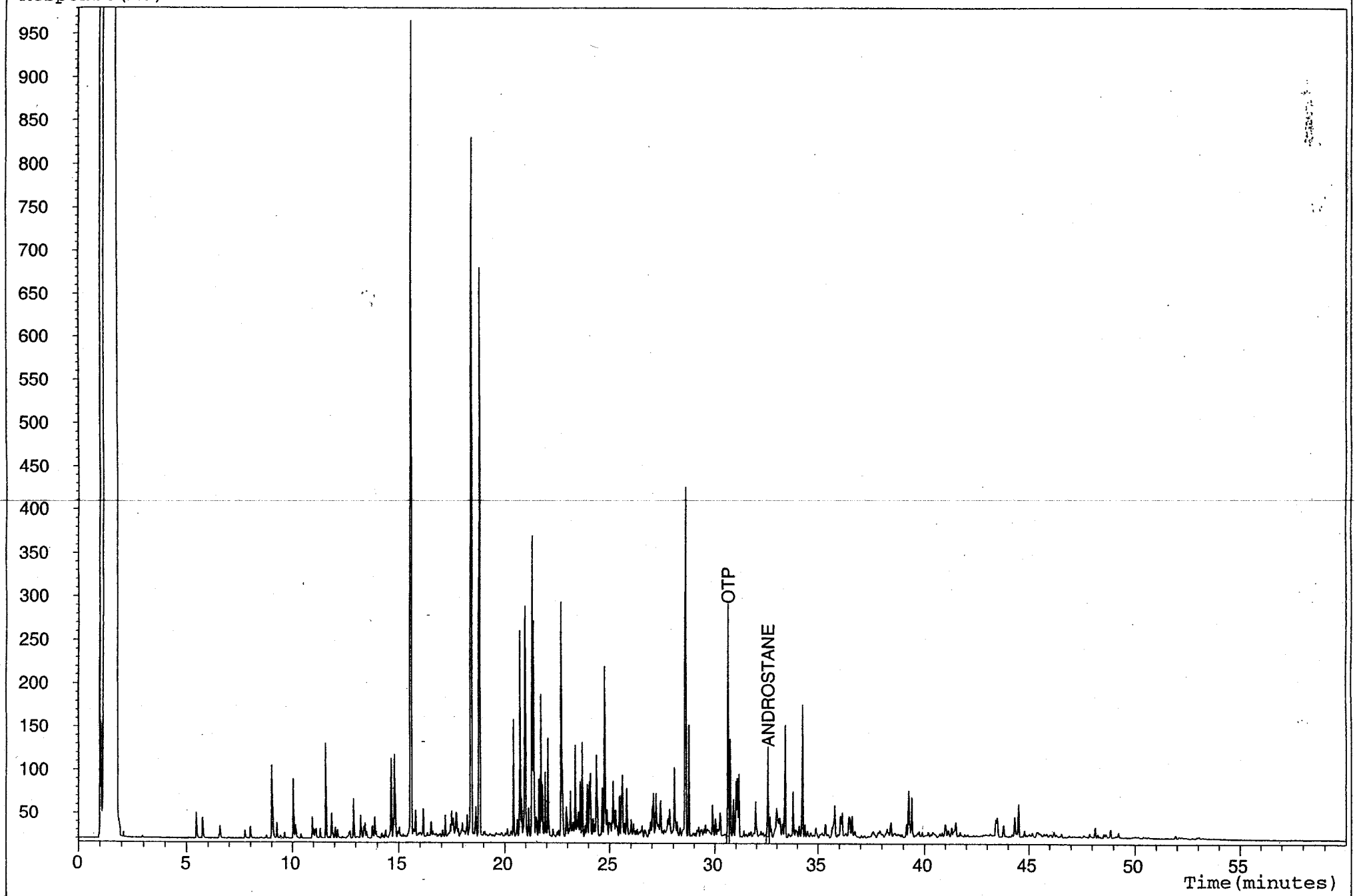
Table 4
Diagnostic Source Ratios Calculated as Input for PCA

Sample ID	AY/ AE	DF/ F0	A0/ P0	FL0/ PY0	BB/ BAP	BJK/ BAP	BEP/ BAP	IND/ GHI	DBT2/ PA2	DBT3/ PA3	N0/ N2+N3	P0+A0/ PA2+PA3	DBT0/ DBT2+DBT3
2"Pipe	0.33	0.57	0.35	0.77	0.49	0.75	0.45	1.18	0.33	0.38	1.34	3.43	1.67
12"Pipe	0.32	0.58	0.32	0.76	0.51	0.72	0.45	1.19	0.30	0.36	1.37	3.37	1.69
TW-13	0.39	0.21	0.25	0.64	0.50	0.64	0.56	0.97	0.32	0.49	4.30	7.89	1.22
D East	0.28	0.24	0.33	0.66	0.37	0.44	0.40	0.98	0.36	0.59	1.31	7.41	0.96
S Gate 1	0.20	0.29	0.33	0.68	0.38	0.45	0.39	1.03	0.33	0.51	1.25	7.07	1.70
S Gate 2	0.17	0.27	0.33	0.67	0.38	0.43	0.39	1.00	0.32	0.49	1.94	7.55	1.15
D Center	0.21	0.26	0.22	0.47	0.51	0.60	0.55	0.94	0.31	0.52	1.49	5.91	0.87
S Center	0.23	0.27	0.30	0.72	0.56	0.66	0.59	0.98	0.30	0.47	1.38	6.13	0.82
UG Riser	0.19	0.20	0.30	0.65	0.45	0.50	0.49	1.00	0.35	0.53	1.34	6.44	0.91
East Riser	0.17	0.32	0.25	0.72	0.56	0.65	0.68	0.84	0.34	0.52	3.71	23.77	7.44
West Riser	0.23	0.20	0.31	0.67	0.45	0.47	0.46	0.95	0.33	0.53	0.69	6.20	0.98
Inside Discharge	0.11	0.19	0.28	0.65	0.52	0.65	0.57	0.98	0.35	0.54	1.47	7.55	1.07
Discharge	0.21	0.19	0.29	0.66	0.41	0.51	0.47	1.03	0.32	0.51	1.52	7.00	1.03
Solid Discharge	0.21	0.34	0.27	0.66	0.56	0.64	0.61	0.91	0.38	0.57	0.65	5.12	0.85
MW-7	0.17	0.28	0.33	0.65	0.52	0.62	0.60	0.90	0.43	0.61	0.14	2.77	0.53
TW-9	0.08	0.07	0.24	0.60	0.41	0.45	0.50	0.94	0.27	0.48	2.77	8.24	0.58
Solid Discharge (Dup)	0.21	0.33	0.28	0.66	0.53	0.66	0.61	0.92	0.38	0.57	0.65	5.54	0.87
Average	0.22	0.28	0.29	0.66	0.48	0.58	0.51	0.99	0.34	0.51	1.61	7.14	1.43
Std Dev	0.08	0.13	0.04	0.07	0.07	0.11	0.09	0.09	0.04	0.06	1.07	4.58	1.59
CV	0.36	0.45	0.13	0.10	0.14	0.18	0.17	0.09	0.11	0.13	0.67	0.64	1.11

Attachment 3

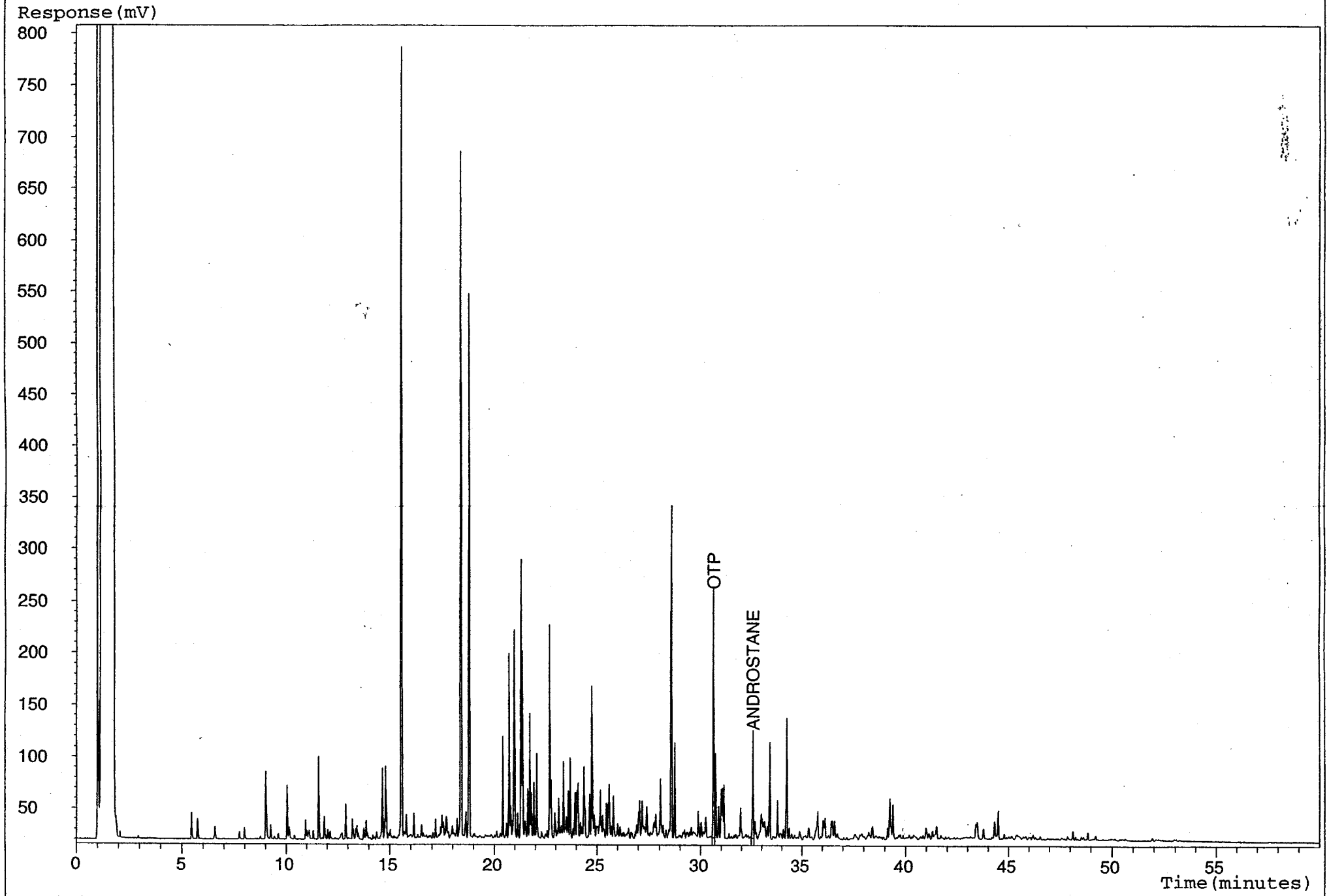
Gas Chromatographic Fingerprints

Analysis: sc1481,20,1 W9071-D 2 INCH STEEL PIPE Project: hydrocarbons
Instrument: chanl_14 Method: mc1421alk



Acquisition Time: 29 Nov 2001 at 03:32.19

Analysis: sc1481,21,1 W9072-D 12 INCH STEEL PIPE Project: hydrocarbons
Instrument: chanl_14 Method: mc1421alk

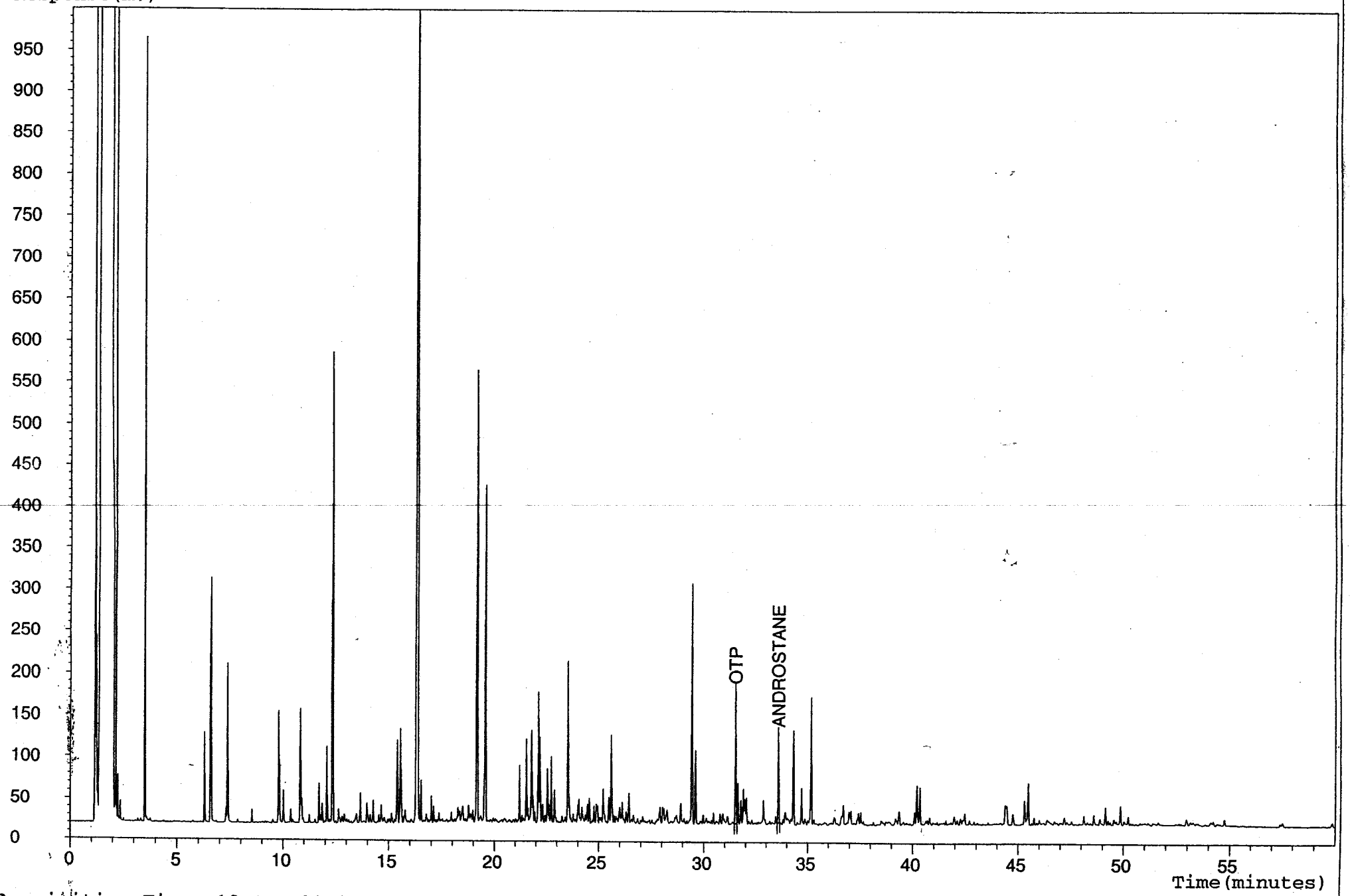


Acquisition Time: 29 Nov 2001 at 04:46.53

Analysis: sc1398,15,1
Instrument: chanl_13
Response(mV)

W5548-1-D

Project: hydrocarbons
Method: MC1352

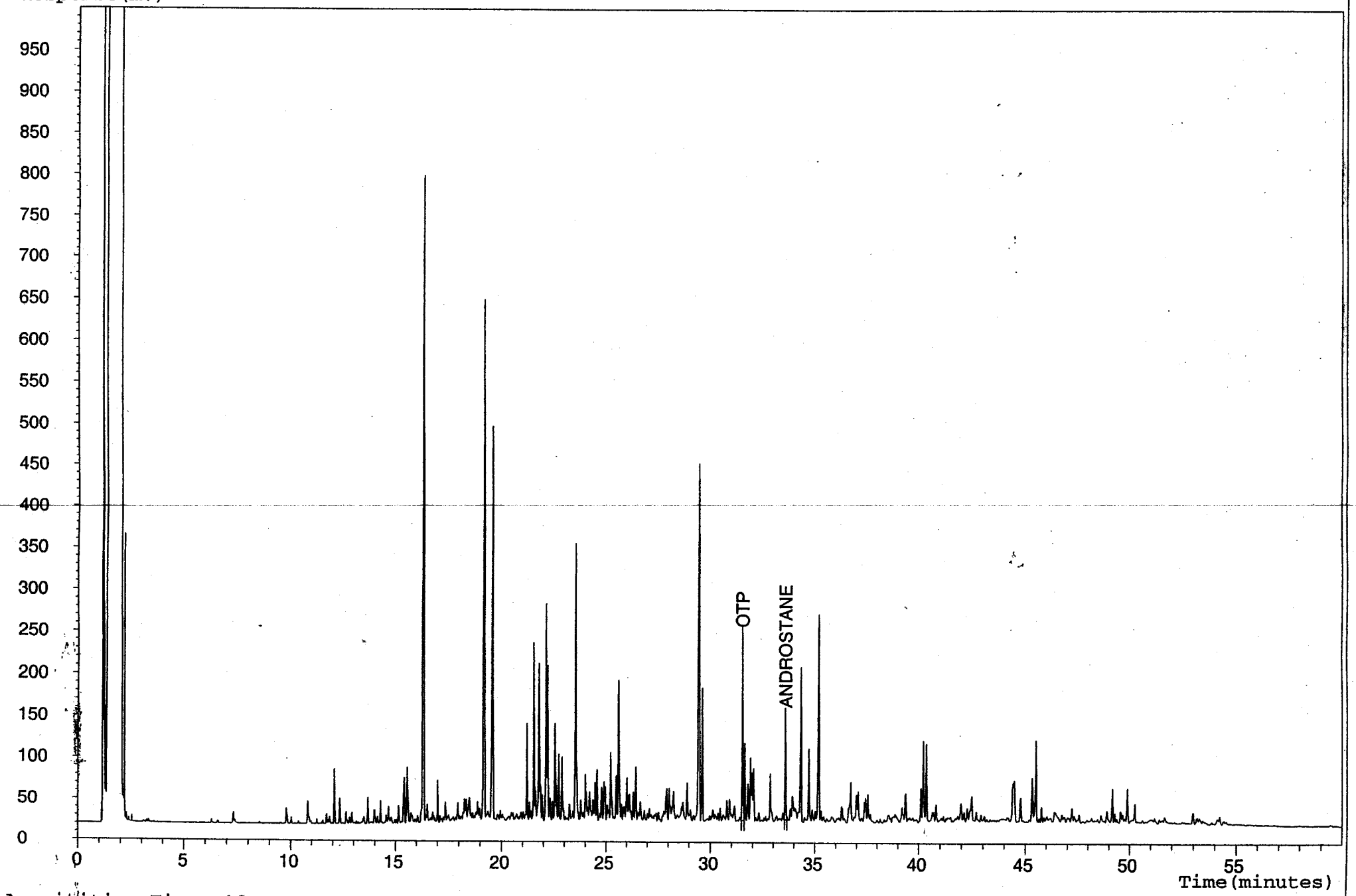


Acquisition Time: 18 Oct 2001 at 06:55.41

Analysis: sc1398,28,1
Instrument: chan1_13
Response(mV)

W6841-D

Project: hydrocarbons
Method: MC1352

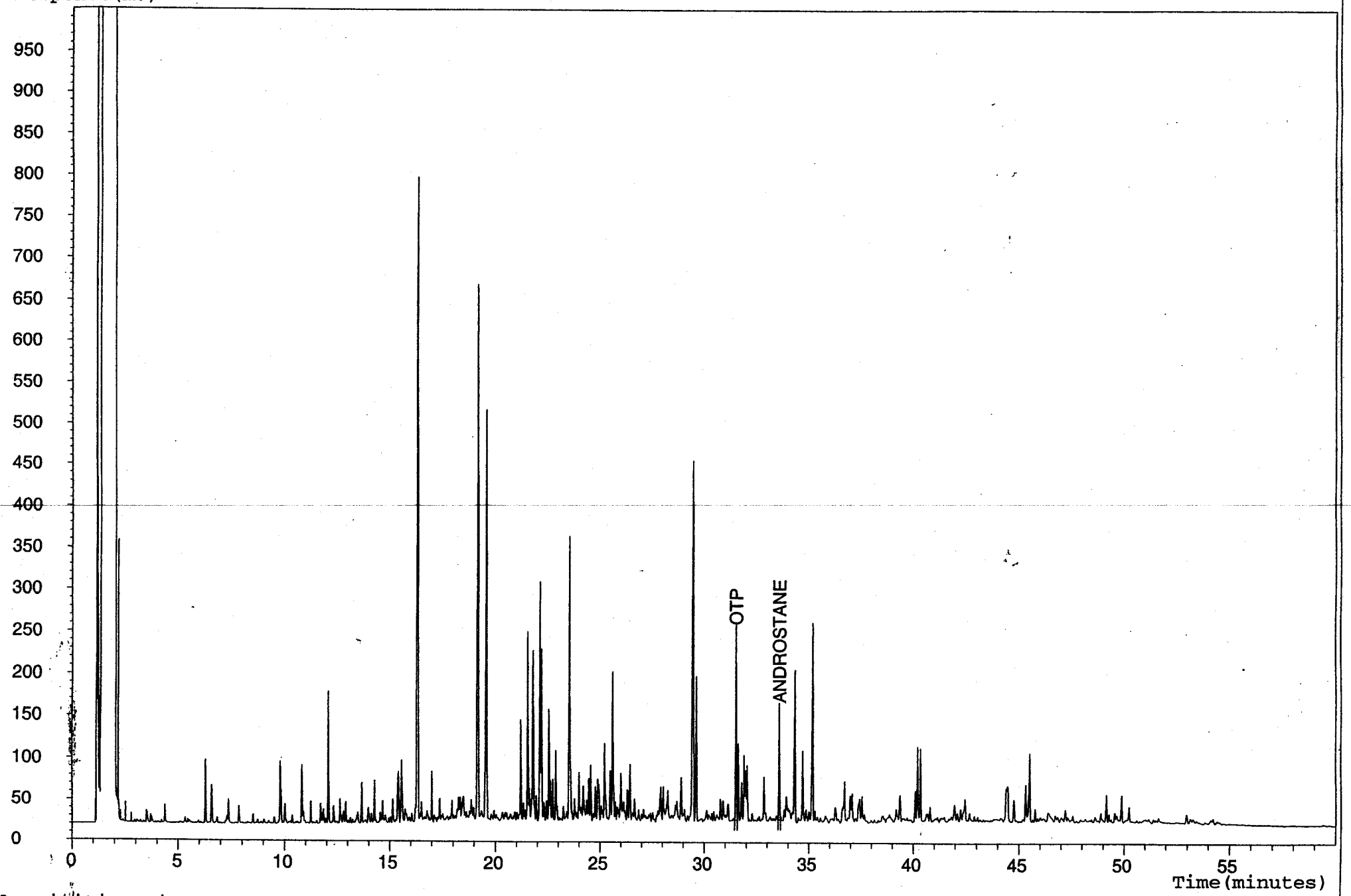


Acquisition Time: 18 Oct 2001 at 23:37.40

Analysis: sc1398,30,1
Instrument: chanl_13
Response(mV)

W6839-D

Project: hydrocarbons
Method: MC1352

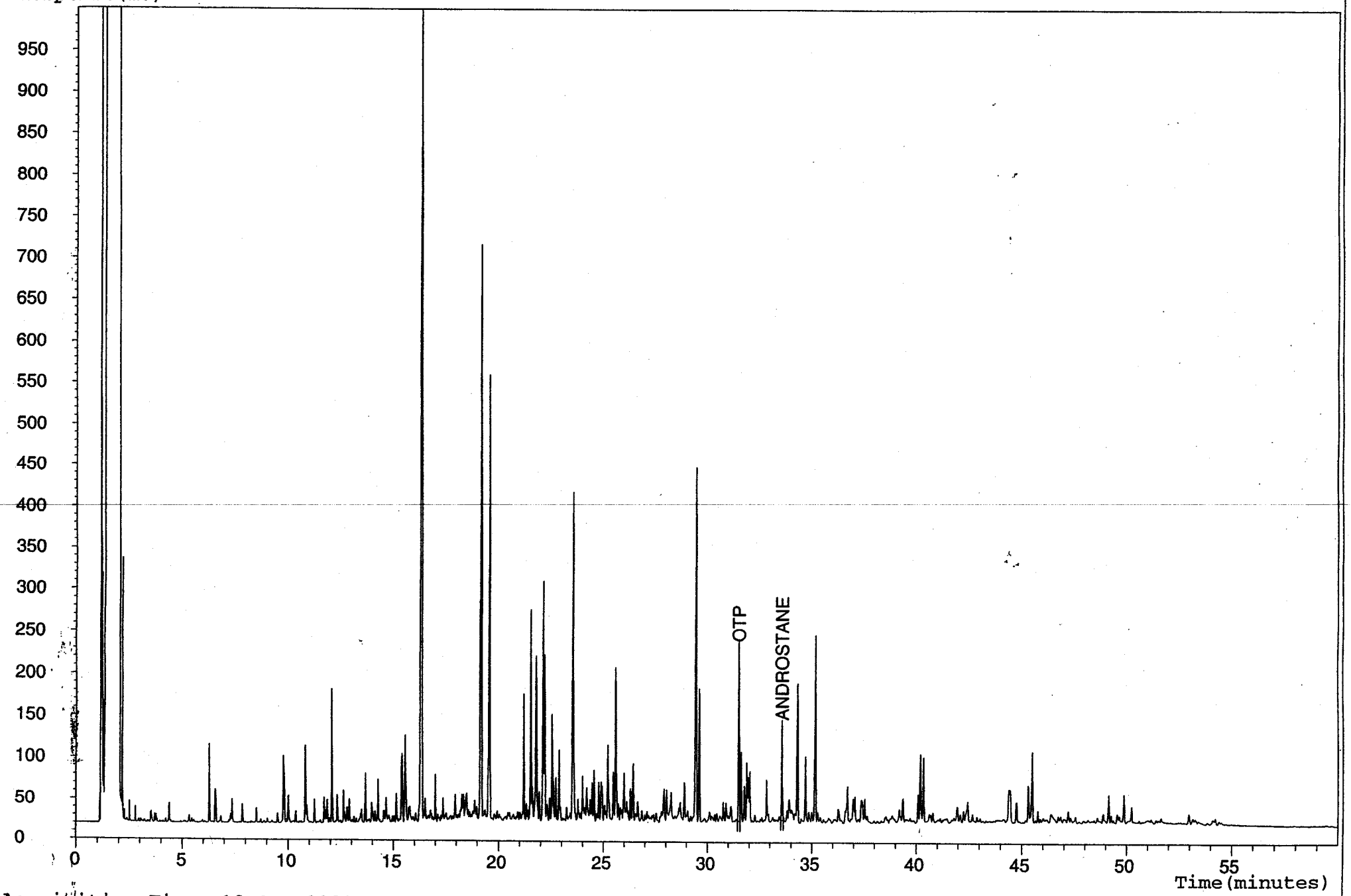


Acquisition Time: 19 Oct 2001 at 02:08.24

Analysis: sc1398,27,1
Instrument: chanl_13
Response(mV)

W6840-D

Project: hydrocarbons
Method: MC1352

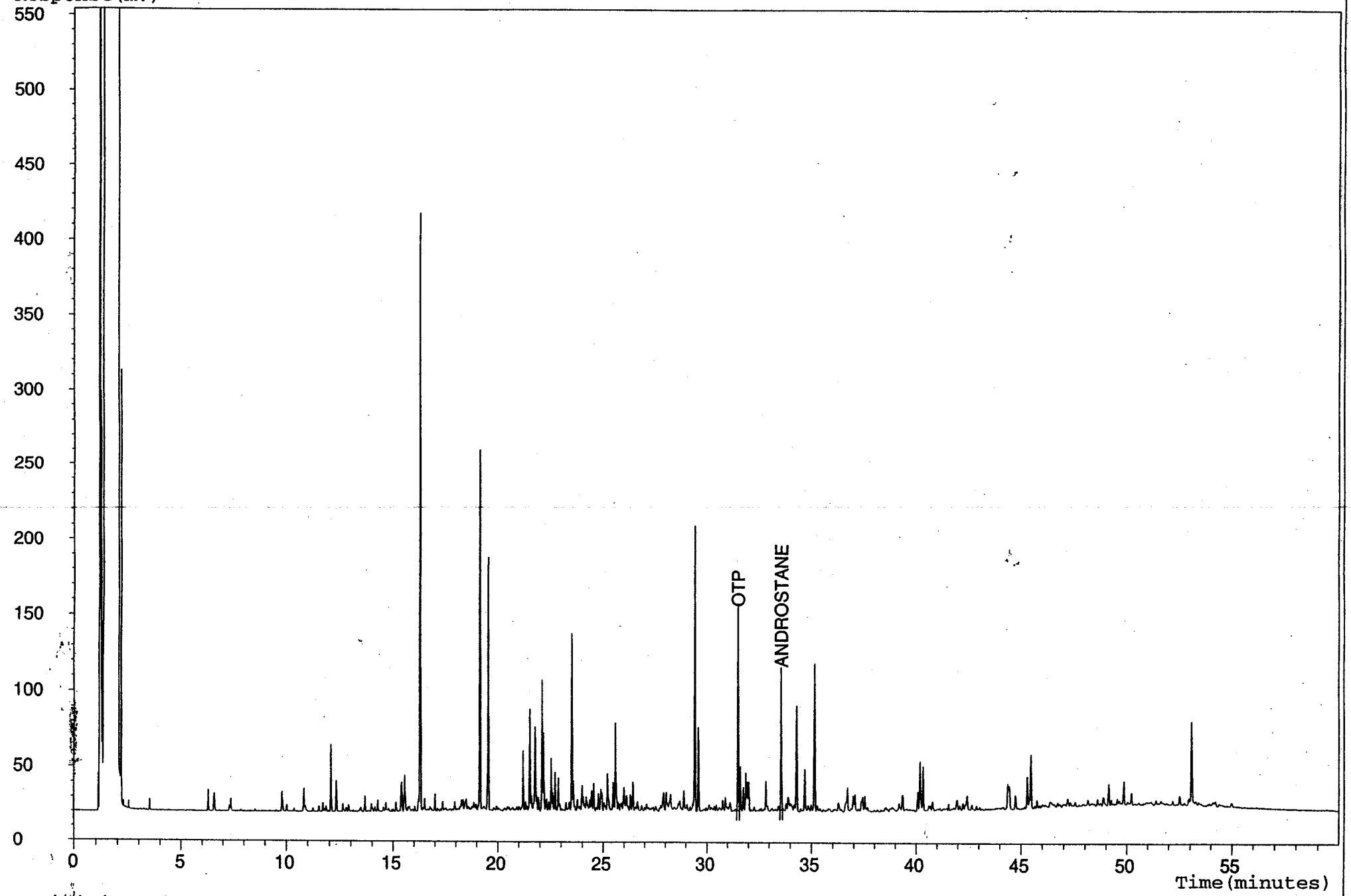


Acquisition Time: 18 Oct 2001 at 22:22.51

Analysis: sc1398,26,1
Instrument: chanl_13
Response(mV)

W6837-D

Project: hydrocarbons
Method: MC1352

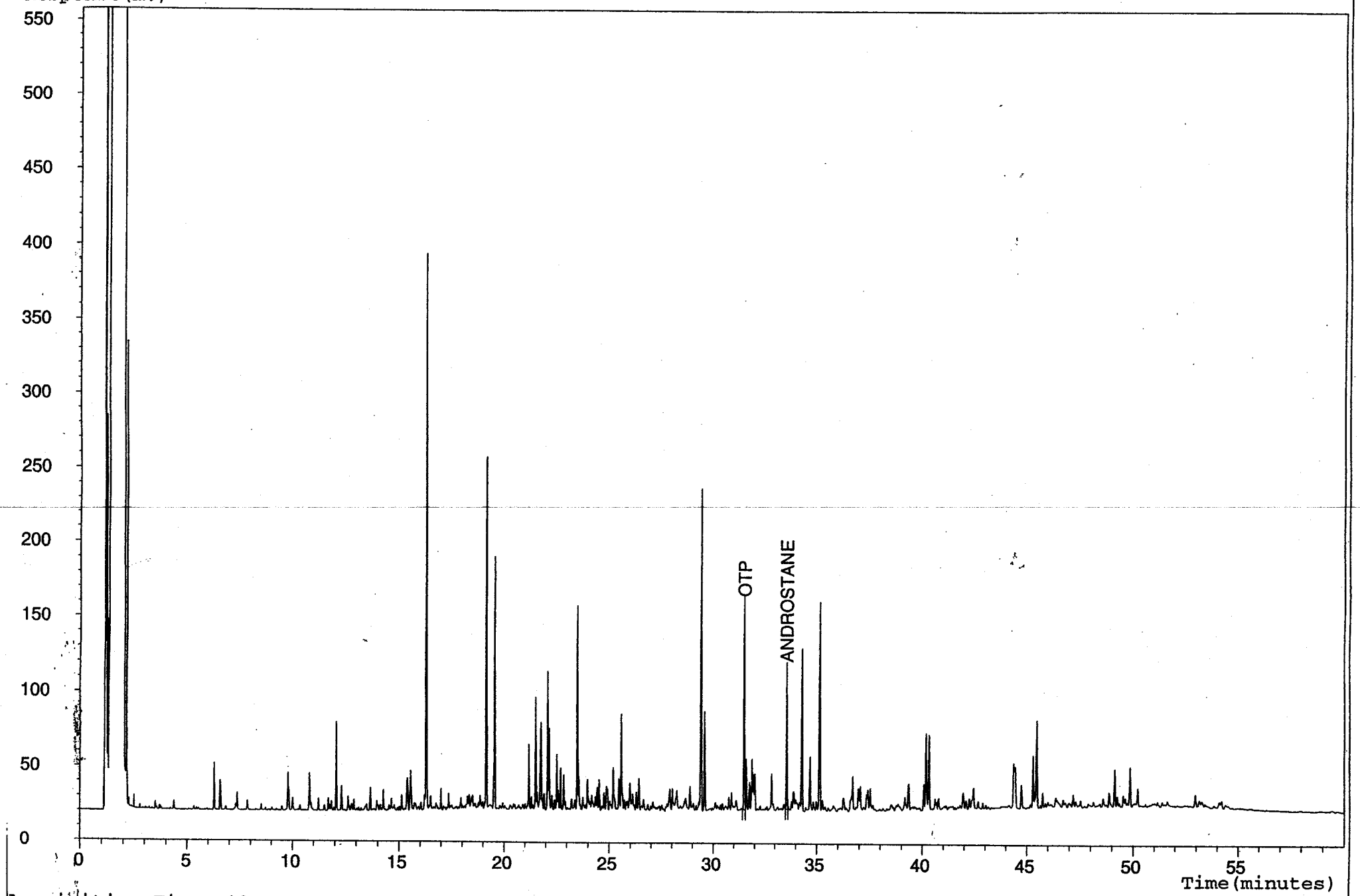


Acquisition Time: 18 Oct 2001 at 21:07.45

Analysis: sc1398,29,1
Instrument: chanl_13
Response(mV)

W6838-D

Project: hydrocarbons
Method: MC1352

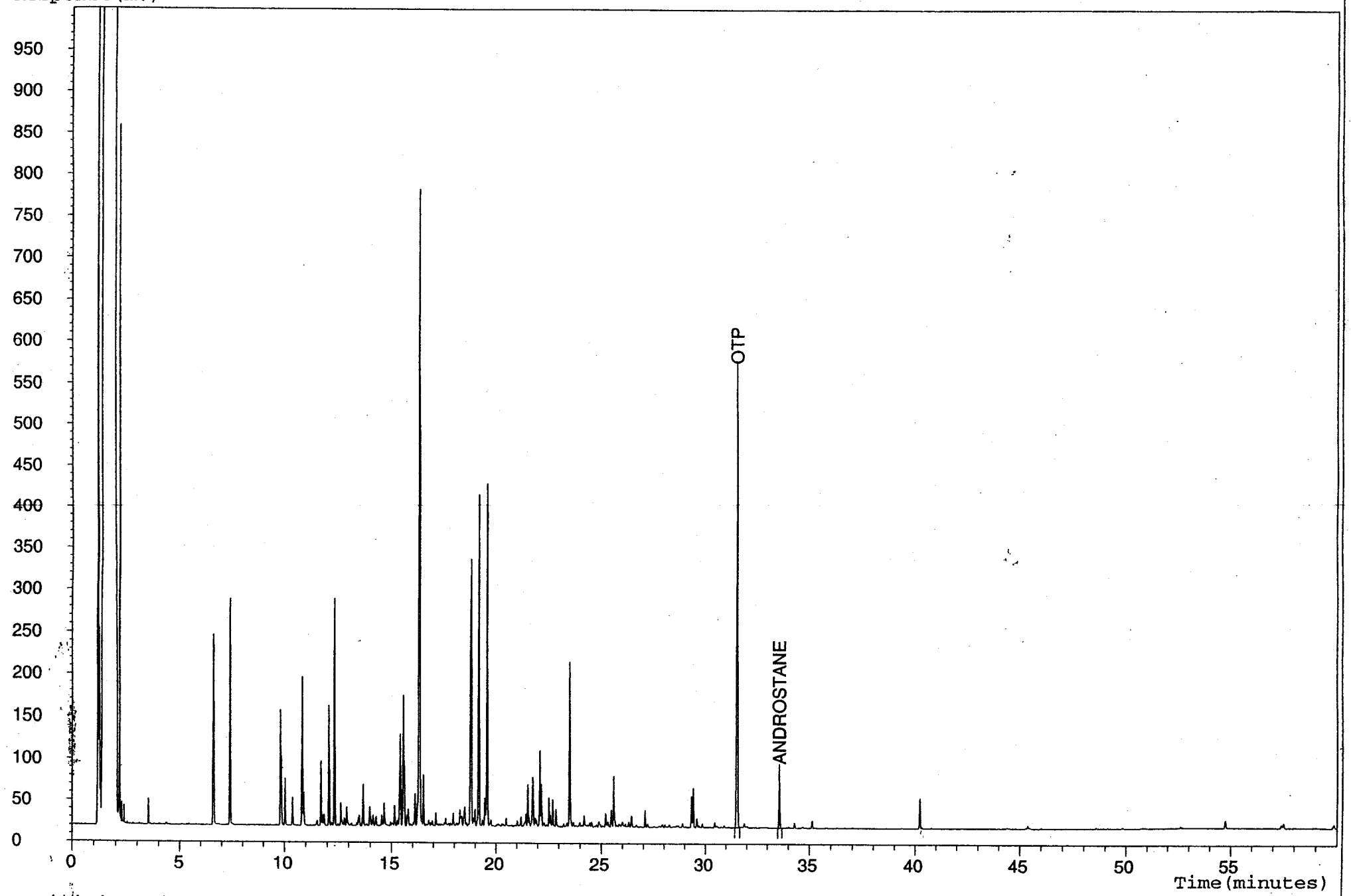


Acquisition Time: 19 Oct 2001 at 00:52.23

Analysis: sc1398,9,1
Instrument: chanl_13
Response(mV)

W5544-1

Project: hydrocarbons
Method: MC1352

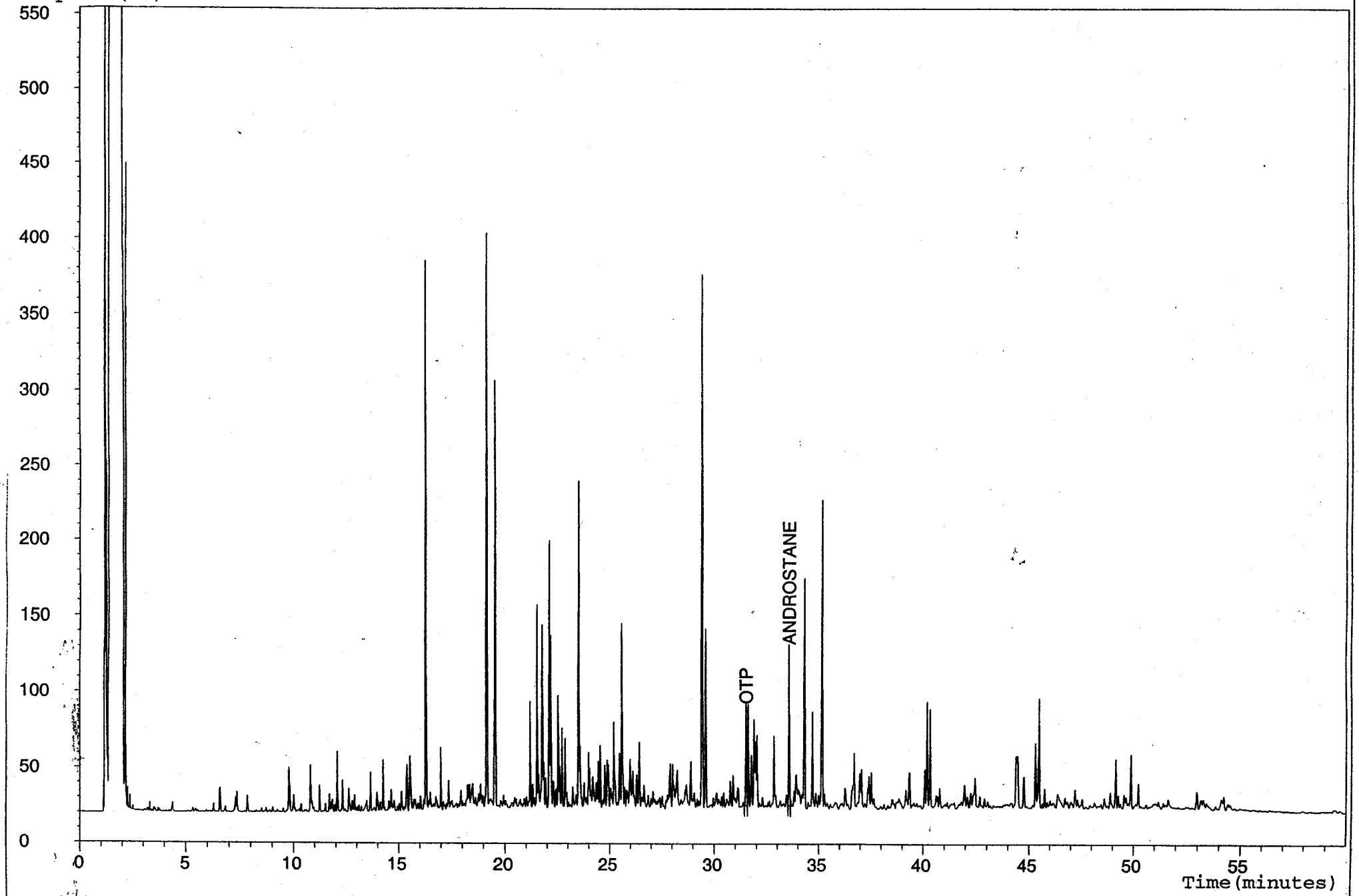


Acquisition Time: 17 Oct 2001 at 23:30.14

Analysis: sc1398,8,1
Instrument: chan1_13
Response(mV)

W5543-1-D

Project: hydrocarbons
Method: MC1352

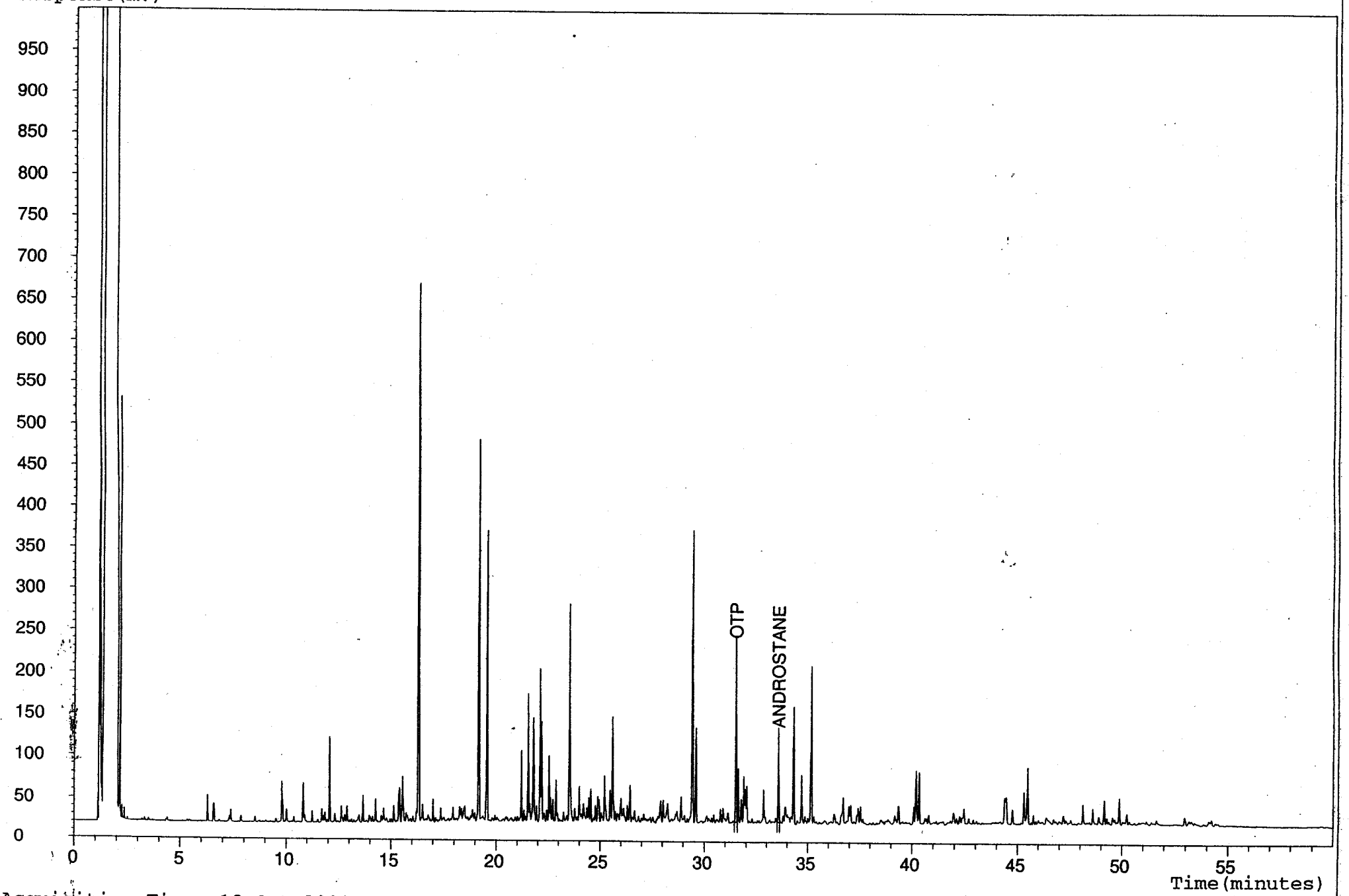


Acquisition Time: 17 Oct 2001 at 22:15.25

Analysis: sc1398,17,1
Instrument: chanl_13
Response(mV)

W5551-1-D

Project: hydrocarbons
Method: MC1352

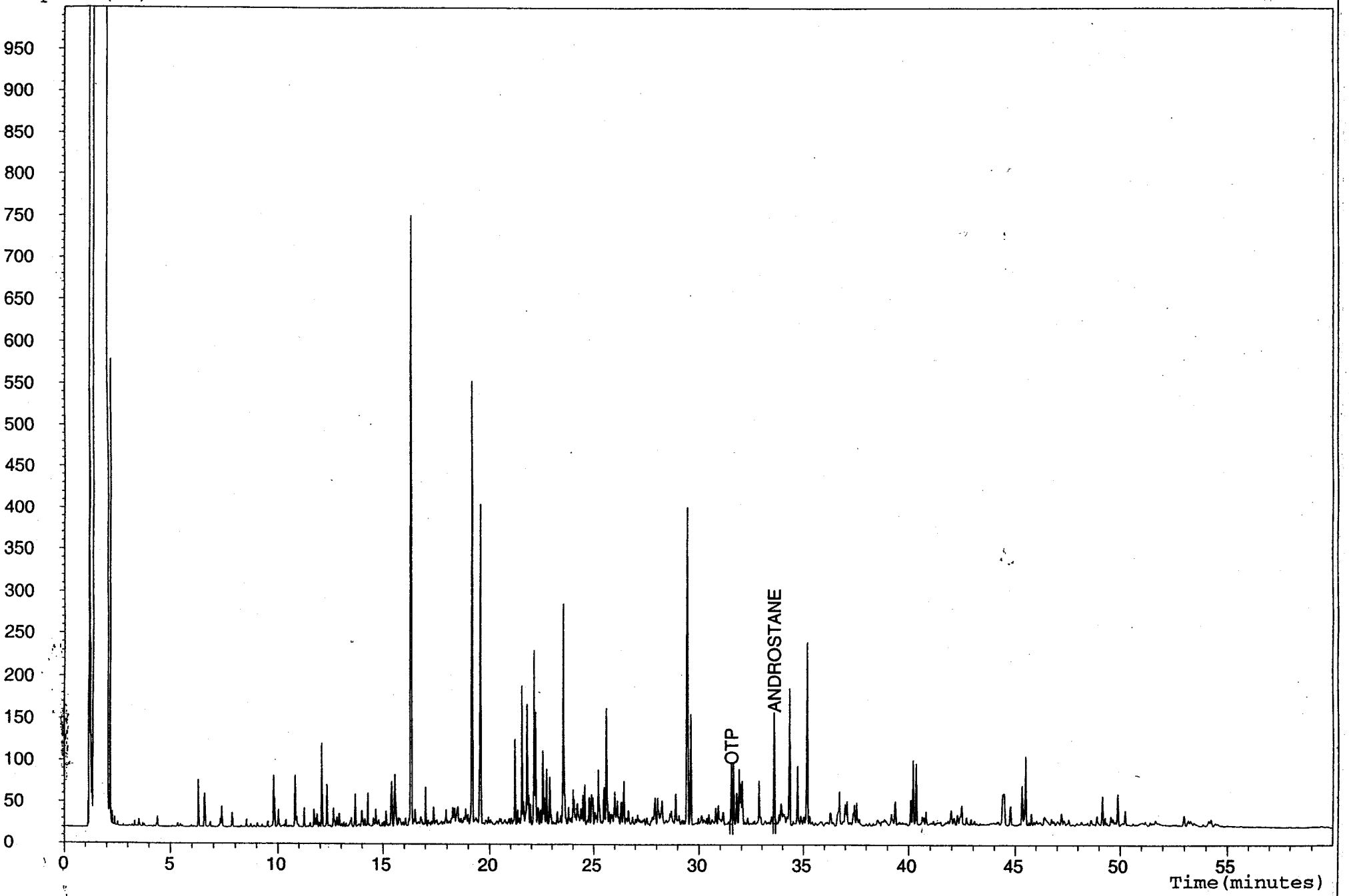


Acquisition Time: 18 Oct 2001 at 09:53.17

Analysis: sc1398,16,1
Instrument: chanl_13
Response(mV)

W5549-1-D

Project: hydrocarbons
Method: MC1352

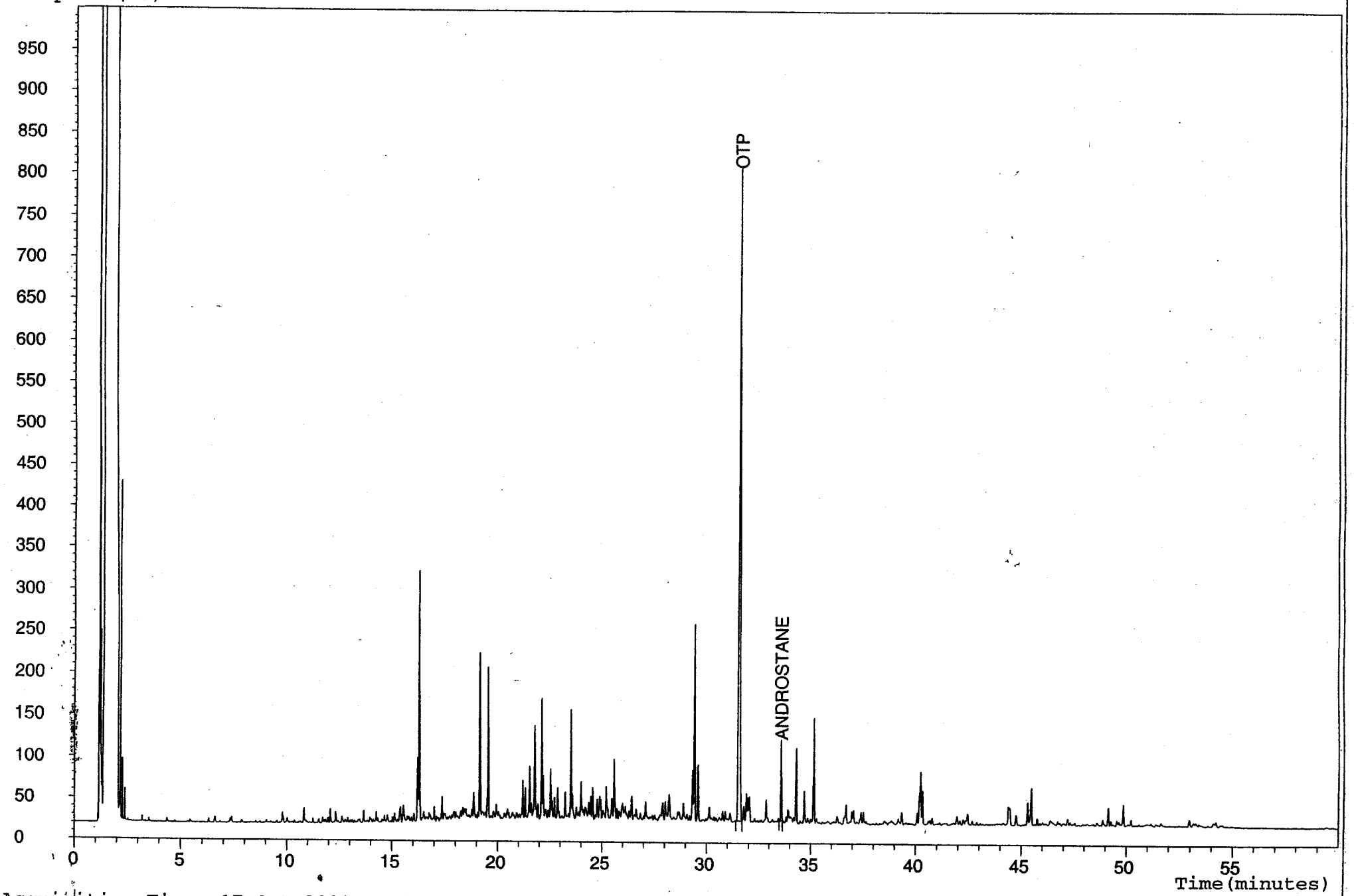


Acquisition Time: 18 Oct 2001 at 08:38.57

Analysis: sc1398,6,1
Instrument: chanl_13
Response(mV)

W5550-1-D

Project: hydrocarbons
Method: MC1352

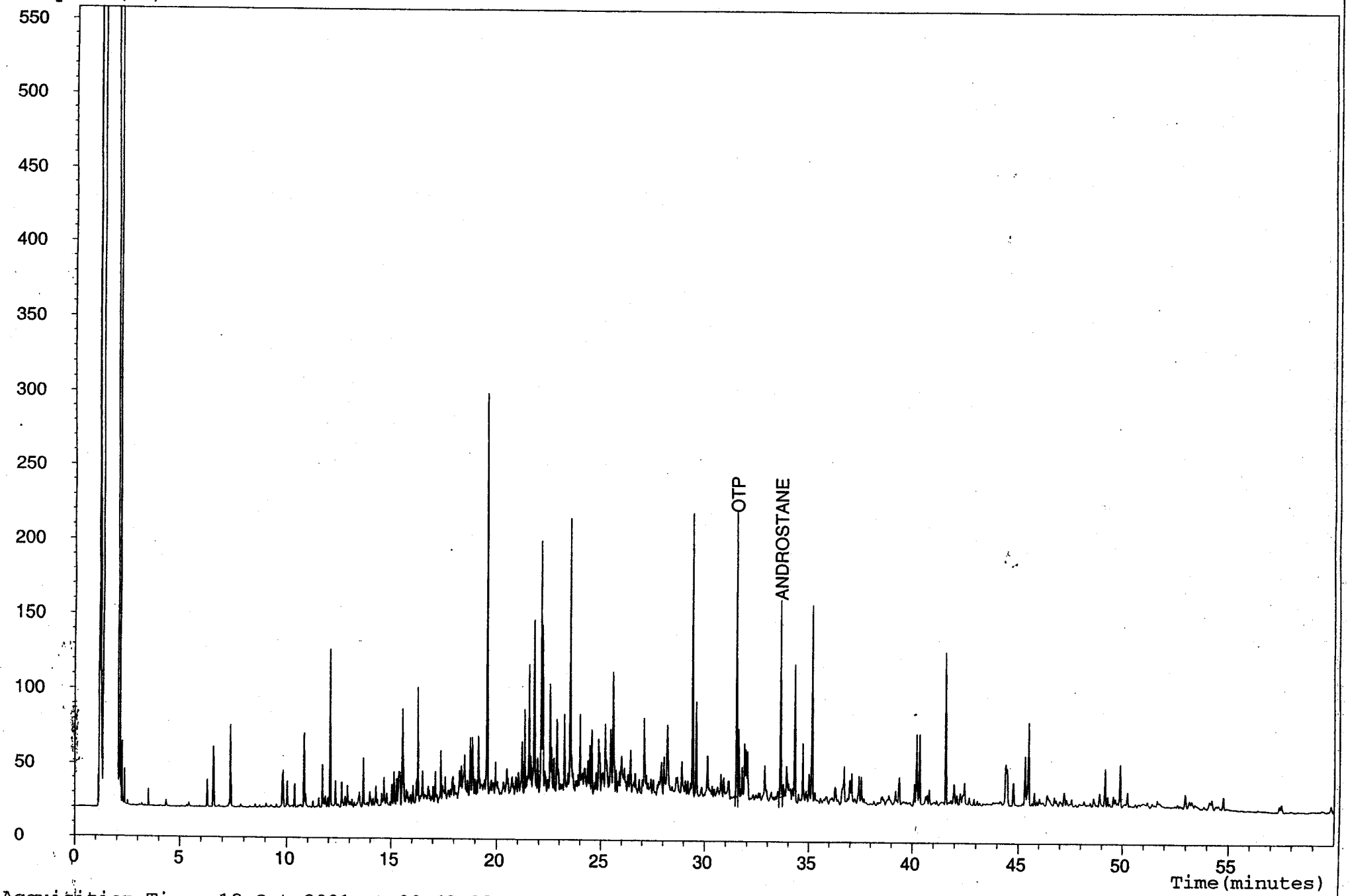


Acquisition Time: 17 Oct 2001 at 19:47.10

Analysis: sc1398,10,1
Instrument: chanl_13
Response(mV)

W5545-1-D

Project: hydrocarbons
Method: MC1352

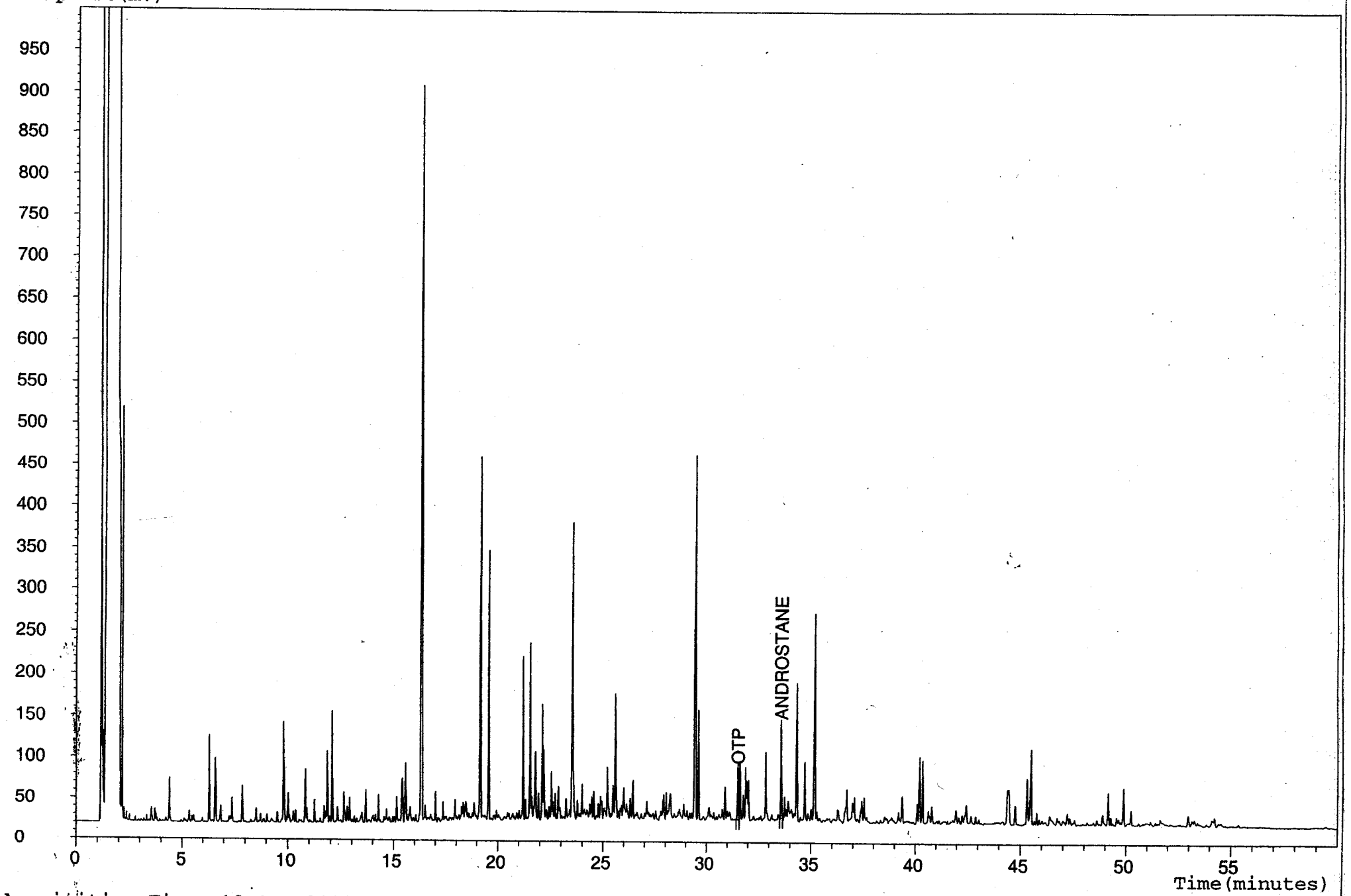


Acquisition Time: 18 Oct 2001 at 00:43.33

Analysis: sc1398,11,1
Instrument: chanl_13
Response(mV)

W5546-1-D

Project: hydrocarbons
Method: MC1352

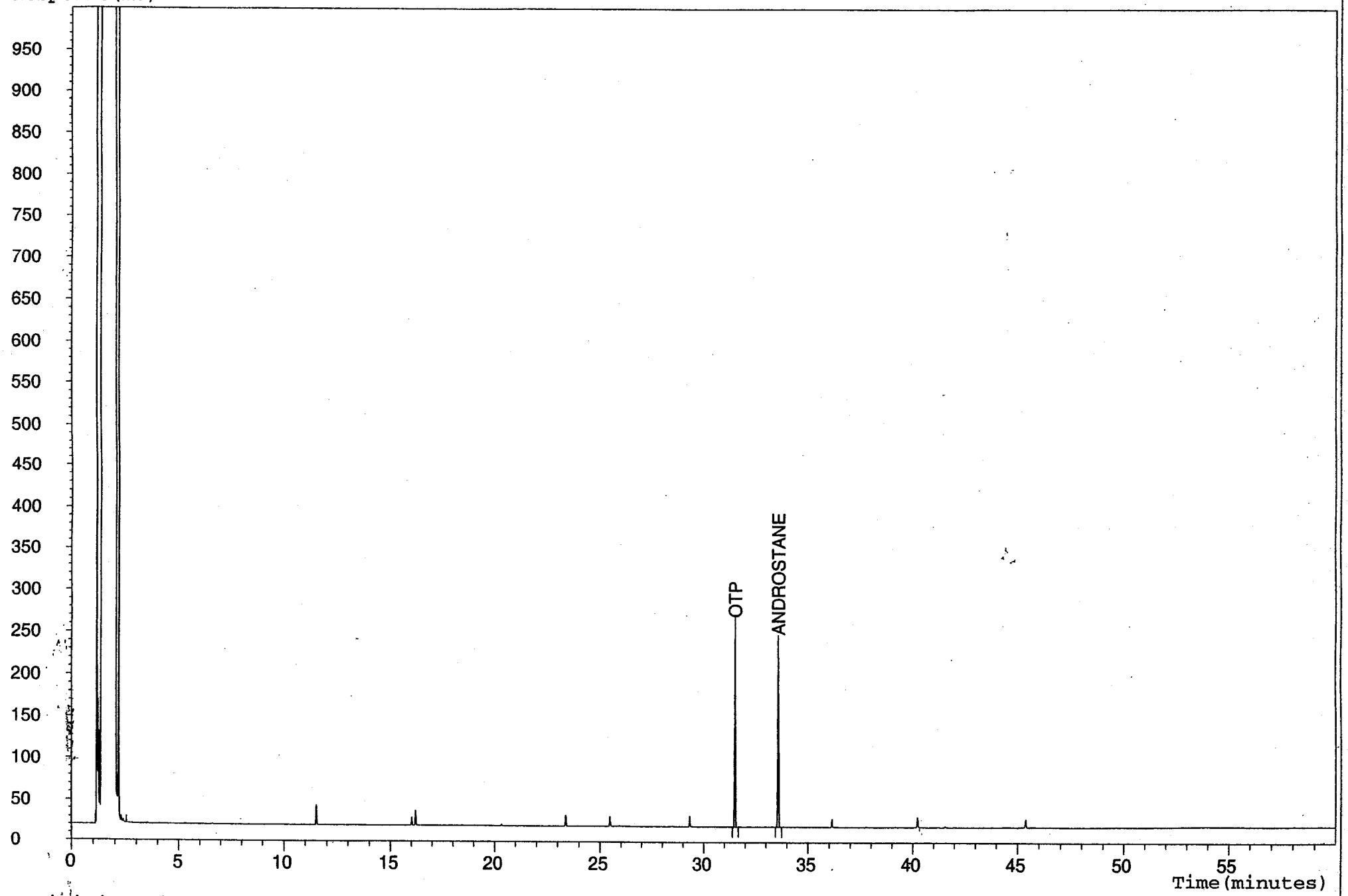


Acquisition Time: 18 Oct 2001 at 01:58.42

Analysis: sc1398,4,1
Instrument: chanl_13
Response(mV)

ZL25PB

Project: hydrocarbons
Method: MC1352

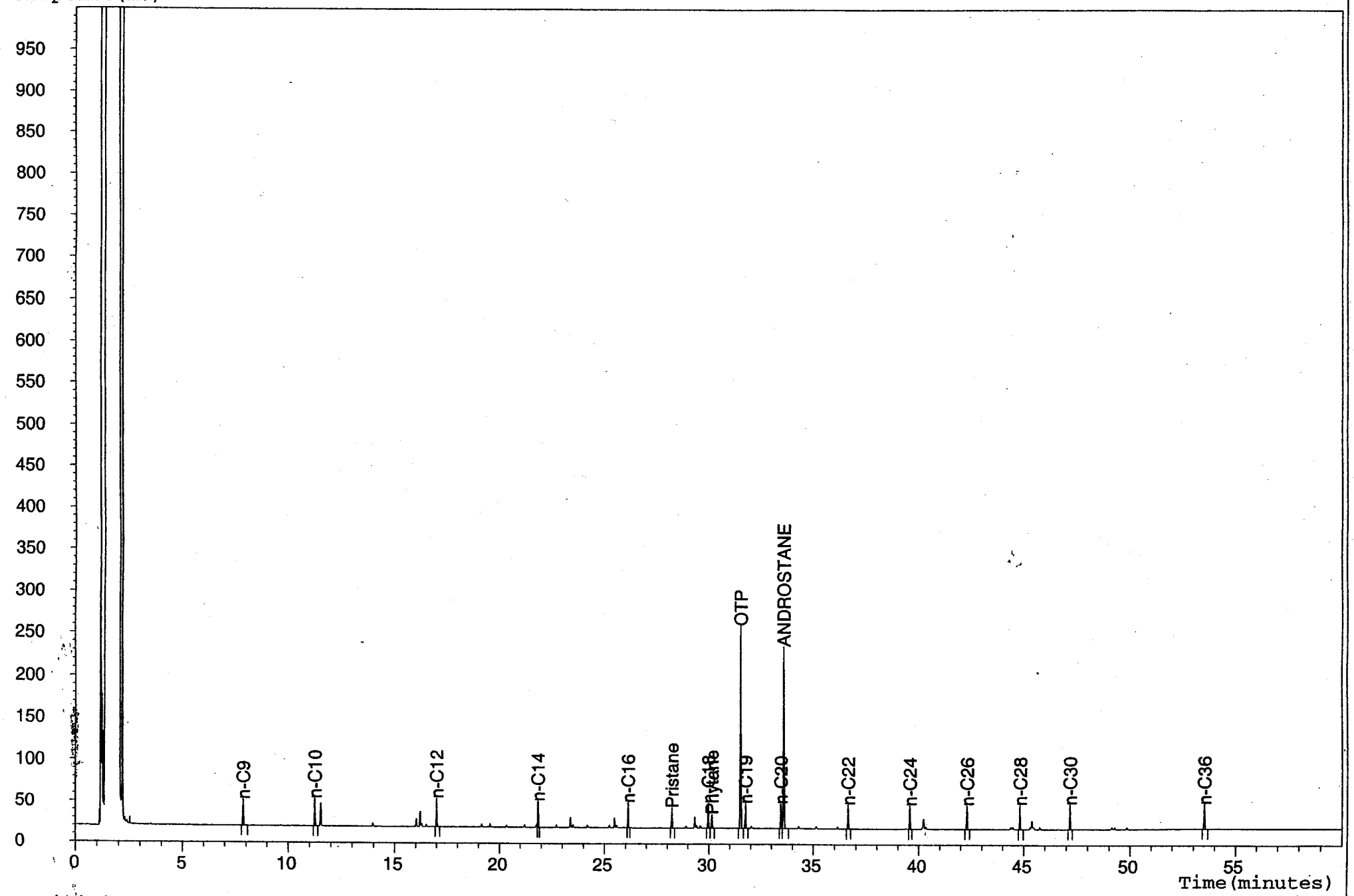


Acquisition Time: 17 Oct 2001 at 17:17.24

Analysis: sc1398,5,1
Instrument: chanl_13
Response(mV)

ZL26LCS

Project: hydrocarbons
Method: MC1352

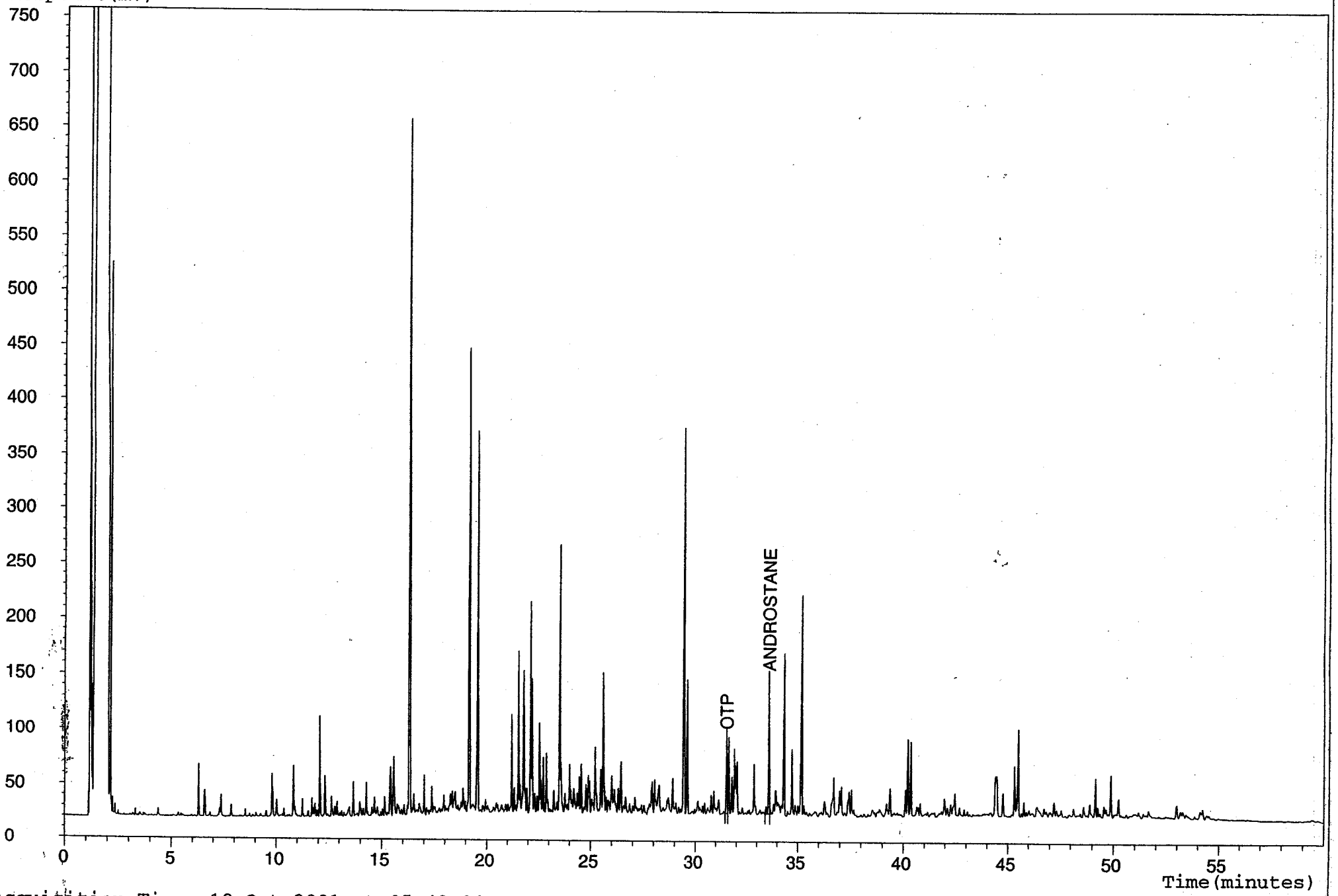


Acquisition Time: 17 Oct 2001 at 18:32.10

Analysis: sc1398,14,1
Instrument: chan1_13
Response(mV)

W5547-1-D

Project: hydrocarbons
Method: MC1352

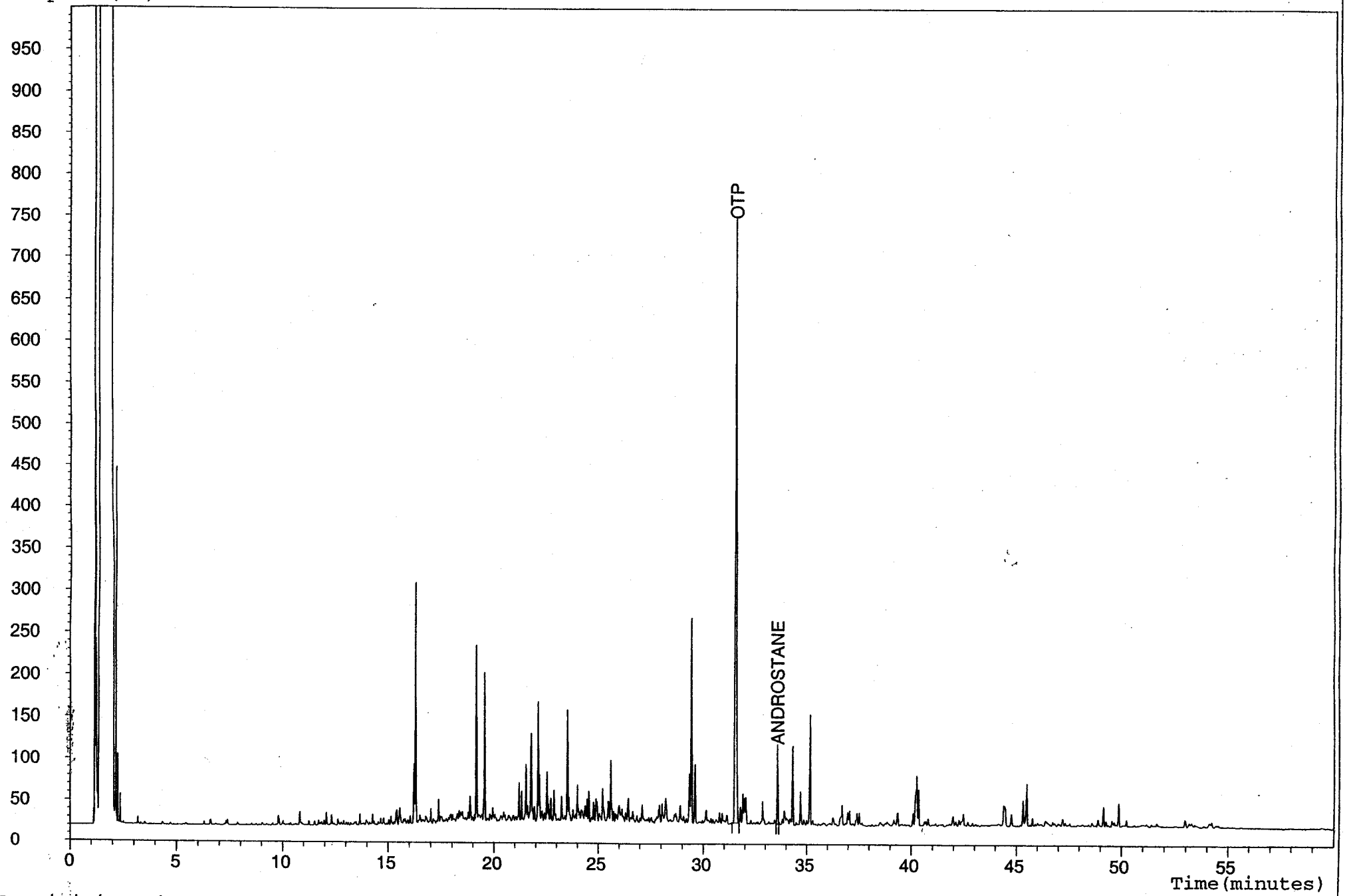


Acquisition Time: 18 Oct 2001 at 05:42.04

Analysis: sc1398,7,1
Instrument: chanl_13
Response(mV)

W5550DUP-1-D

Project: hydrocarbons
Method: MC1352

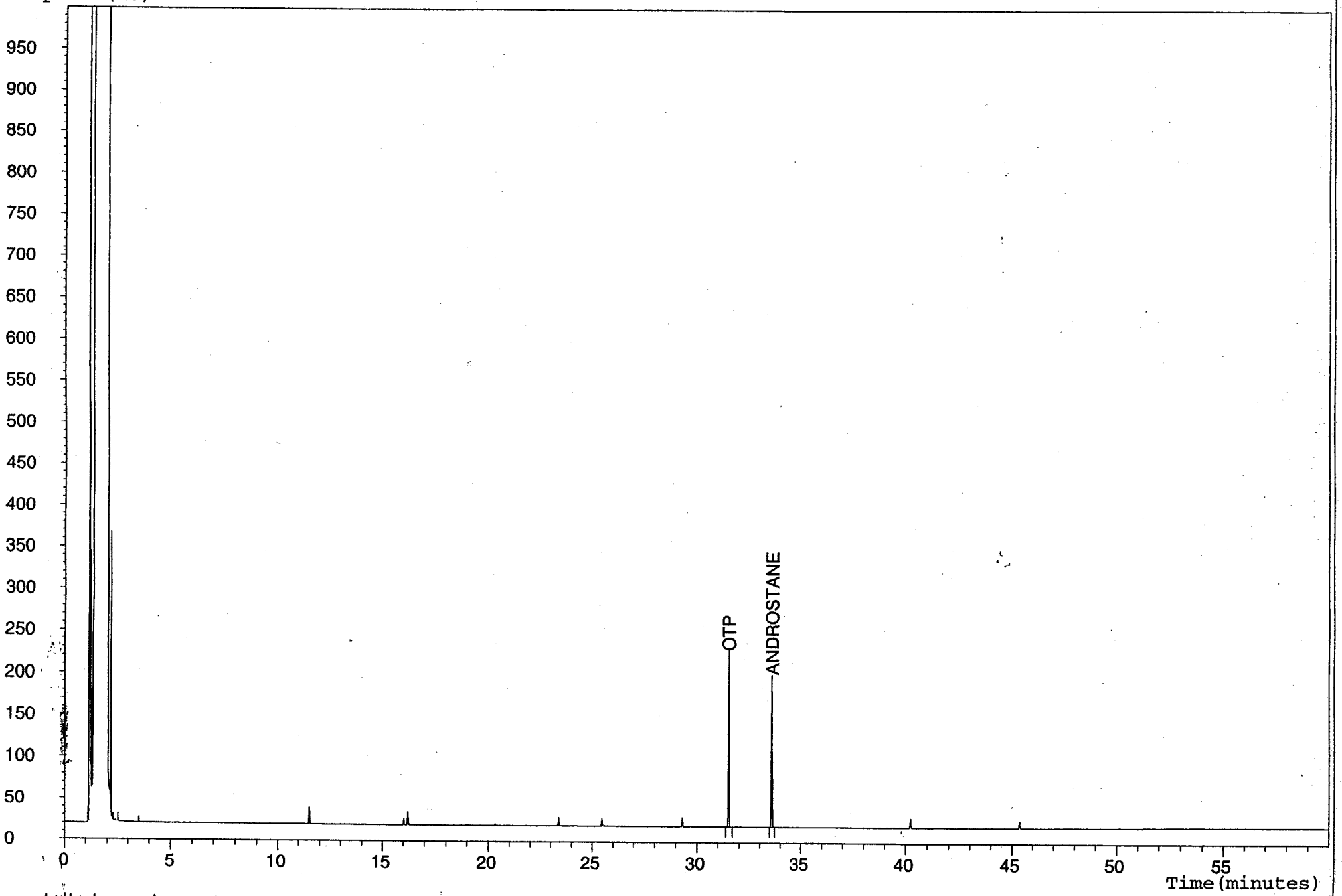


Acquisition Time: 17 Oct 2001 at 21:01.56

Analysis: sc1398,19,1
Instrument: chanl_13
Response(mV)

ZK90PB

Project: hydrocarbons
Method: MC1352

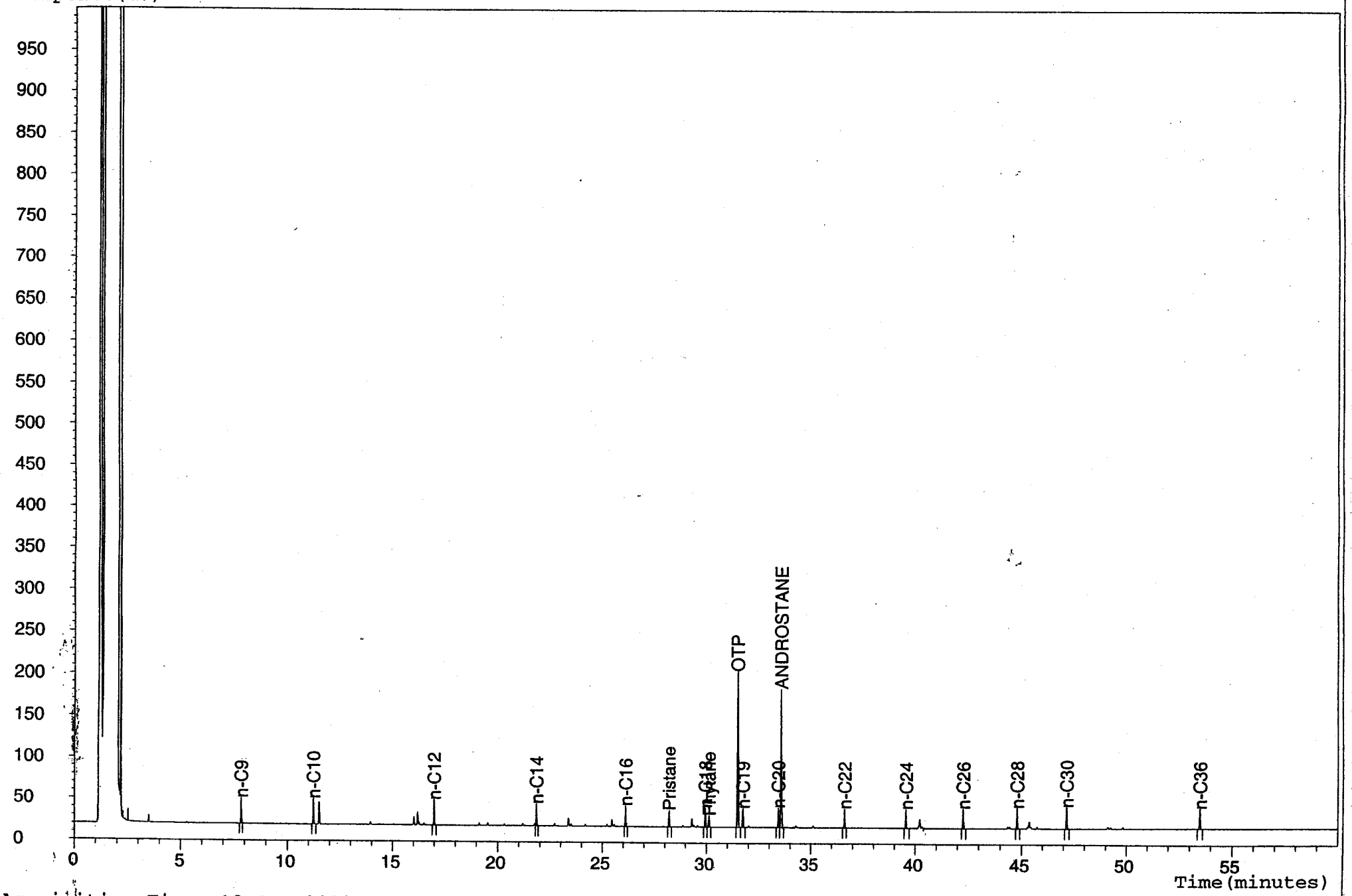


Acquisition Time: 18 Oct 2001 at 12:21.37

Analysis: sc1398,20,1
Instrument: chanl_13
Response(mV)

ZK91LCS

Project: hydrocarbons
Method: MC1352

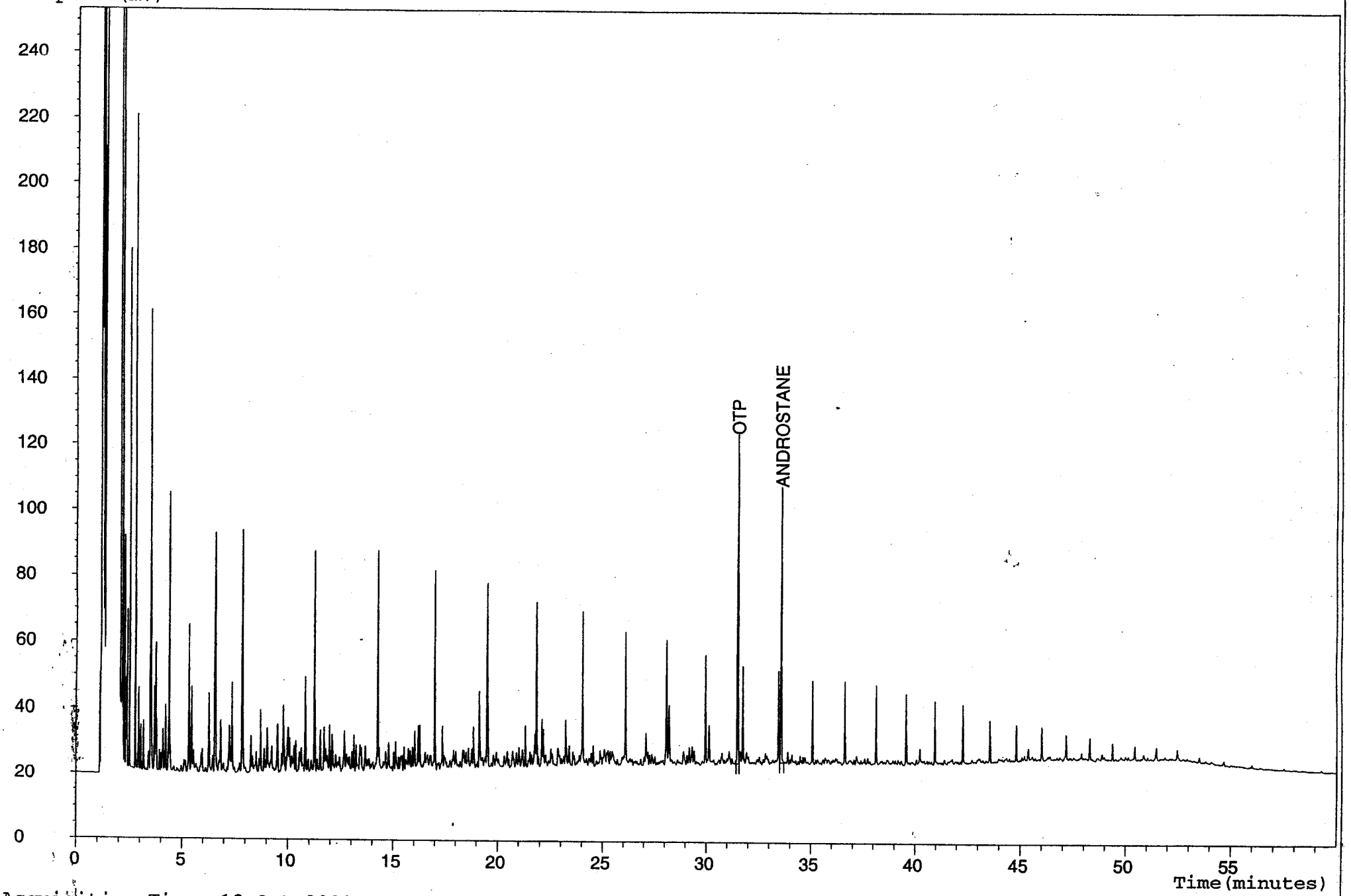


Acquisition Time: 18 Oct 2001 at 13:37.31

Analysis: sc1398,21,1
Instrument: chanl_13
Response(mV)

ZL42NSC

Project: hydrocarbons
Method: MC1352

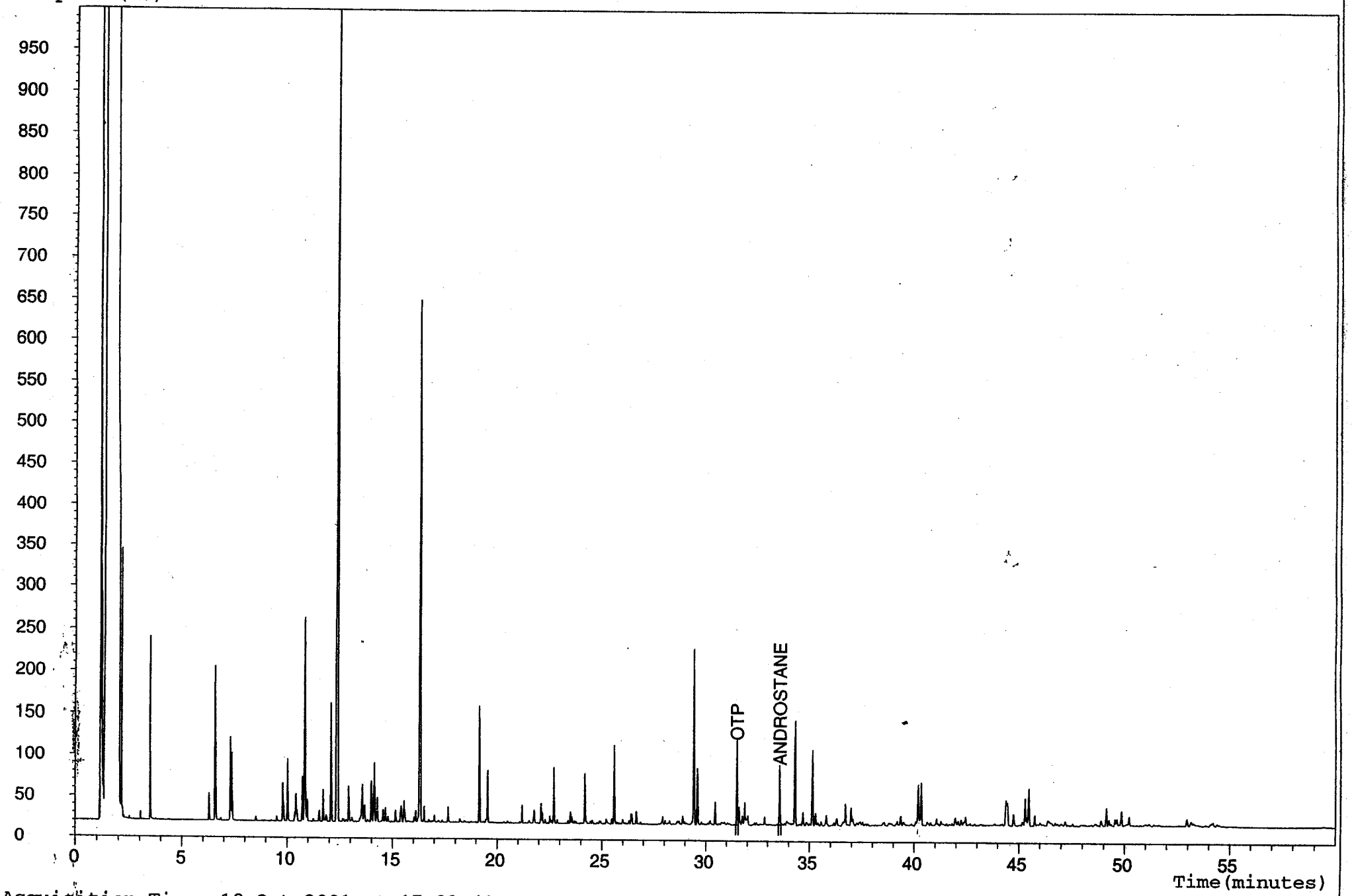


Acquisition Time: 18 Oct 2001 at 14:53.20

Analysis: sc1398,23,1
Instrument: chan1_13
Response(mV)

ZL44CT

Project: hydrocarbons
Method: MC1352

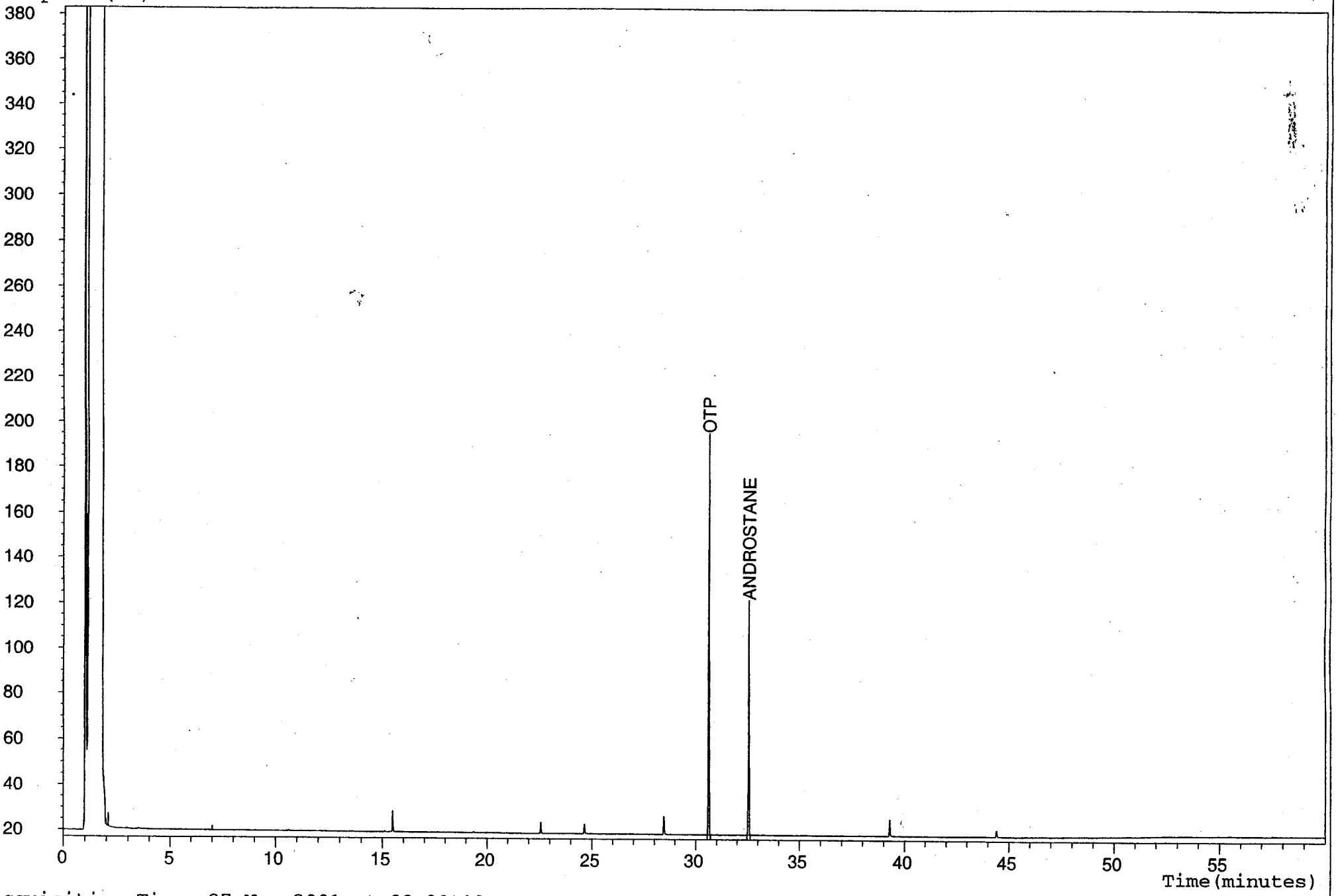


Acquisition Time: 18 Oct 2001 at 17:23.49

Analysis: sc1481,6,1
Instrument: chanl_14
Response(mV)

ZO16PB

Project: hydrocarbons
Method: mc1421alk



Acquisition Time: 27 Nov 2001 at 22:20.19

Attachment 4

Quantitative GC/MS Data

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Project Name Ashland MGP
Project Number N004602-0002

Concentrations of Polycyclic Armatic Hydrocarbons and Other Selected Analytes

Client Sample ID	2 Inch Steel Pipe	12 Inch Steel Pipe	TW-13	Pipe Discharge East Yard Gate
Battelle Sample ID	W9071-D	W9072-D	W5548-1-D	W6841-D
Matrix	Oil	Oil	Aqueous	Solid
Battelle Batch ID	01-610	01-610	01-550	01-546
Analytical Method	8270M	8270M	8270M	8270M
Collection Date	11/12/01	11/12/01	07/24/01	09/20/01
Receipt Date	11/14/01	11/14/01	07/27/01	09/21/01
Extraction Date	11/20/01	11/20/01	07/03/01	10/03/01
Analysis Date	11/23/01	11/23/01	10/25/01	10/22/01
Dilution Factor	10	10	9.53	2.5
% Moisture	NA	NA	NA	NA
Sample Size	0.0572 g	0.0594 g	1.1 L	0.0528 g
Units	mg/Kg	mg/Kg	ng/L	ug/Kg
Decalin	9	7	5400	25700
C1-Decalins	133	95	12200	65300
C2-Decalins	208	185	19500	130000
C3-Decalins	205	144	16000	128000
C4-Decalins	135	78	11400	102000
Benzo(b)thiophene	905	679	140000 D	169000
C1-Benzo(b)thiophenes	1760	1319	95800	450000
C2-Benzo(b)thiophenes	1828	1351	69100	564000
C3-Benzo(b)thiophenes	782	580	31200	291000
C4-Benzo(b)thiophenes	198	113	8210	96000
Naphthalene	23862 D	15512 D	4260000 D	9900000 D
C1-Naphthalenes	23804 D	15305 D	1660000 D	9880000 D
C2-Naphthalenes	13538 D	8545 D	749000	5480000
C3-Naphthalenes	4240 D	2747 D	242000	2060000
C4-Naphthalenes	785 D	479 D	61600	566000
Biphenyl	2417	1842	147000	1370000 D
Acenaphthylene	1818	1390	226000 D	1240000 D
Acenaphthene	5528	4278 D	574000 D	4450000 D
Dibenzofuran	2355	1808	60000	560000 D
Fluorene	4114	3136	290000 D	2290000 D
C1-Fluorenes	3151	2269	133000 D	1300000 D
C2-Fluorenes	1609	1135	60800	611000
C3-Fluorenes	861	1066	23900	260000
Anthracene	3084	2383	278000 D	2550000 D
Phenanthrene	8856	7369 D	1100000 D	7730000 D
C1-Phenanthrenes/Anthracenes	6816	5299	448000 D	4240000 D
C2-Phenanthrenes/Anthracenes	2709	2257	141000	1110000
C3-Phenanthrenes/Anthracenes	768	636	33700	278000
C4-Phenanthrenes/Anthracenes	228	155	36100	310000
Dibenzothiophene	1979	1541	75400	543000
C1-Dibenzothiophenes	1777	1366	71500	599000
C2-Dibenzothiophenes	894	683	45400	402000
C3-Dibenzothiophenes	294	231	16600	163000
C4-Dibenzothiophenes	78	53	4440	41900
Fluoranthene	2737	2159	377000 D	2940000 D
Pyrene	3560	2839 D	589000 D	4440000 D
C1-Fluoranthenes/Pyrenes	3985	3092	280000	2360000
C2-Fluoranthenes/Pyrenes	1496	1182	71900	689000
C3-Fluoranthenes/Pyrenes	517	462	16700	161000
Benz[a]anthracene	1350	1065	132000	1400000 D
Chrysene	1200	936	116000	1300000 D
C1-Chrysenes	1083	832	70500	545000
C2-Chrysenes	559	472	21900	186000
C3-Chrysenes	264	194	10600	87000
C4-Chrysenes	48	34 U	2820	U
Benzo[b]fluoranthene	511	416	61100	453000
Benzo[j/k]fluoranthene	793	584	77900	548000
Benzo[e]pyrene	473	362	67800	498000
Benzo[a]pyrene	1054	809	121000	1240000
Perylene	171	129	19700	148000
Indeno[1,2,3-c,d]pyrene	440	332	58400	473000
Dibenz[a,h]anthracene	116	88	13600	116000
Benzo[g,h,i]perylene	373	279	59900	482000
Total PAH	136000	97800	13100000	76000000
Surrogate Recoveries (%)				
Naphthalene-d8	76	81	80	103
Phenanthrene-d10	68	75	87	90
Chrysene-d12	72	80	81	80

Data Qualifiers
& - Excedes QC criteria
B - Analyte detected < 5x PB.
D - Result from higher dilution.
DO - Surrogate diluted out.
E - Result above calibration curve.
J - Result less than reporting limit.
ME - Matrix effect.
NA - Not applicable.
U - Undetected.

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Concentrations of Polycyclic Armatic Hydrocarbons and Other Selected Analytes

Project Name Ashland MGP
Project Number N004602-0002

Client Sample ID	Pipe Sludge East Yard Gate #1	Pipe Sludge East Yard Gate #2	Pipe Discharge Center Of Yard	Pipe Sludge Center of Yard
Battelle Sample ID	W6839-D	W6840-D	W6837-D	W6838-D
Matrix	Solid	Solid	Solid	Solid
Battelle Batch ID	01-546	01-546	01-546	01-546
Analytical Method	8270M	8270M	8270M	8270M
Collection Date	09/20/01	09/20/01	09/19/01	09/19/01
Receipt Date	09/21/01	09/21/01	09/21/01	09/21/01
Extraction Date	10/03/01	10/03/01	10/03/01	10/03/01
Analysis Date	10/23/01	10/23/01	10/23/01	10/22/01
Dilution Factor	100	125	3.33	10
% Moisture	NA	NA	NA	NA
Sample Size	10.021 g	10.0326 g	5.1851 g	10.0705 g
Units	ug/Kg	ug/Kg	ug/Kg	ug/Kg
Decalin	6000	5890	171	405
C1-Decalins	23500	30900	435	1210
C2-Decalins	37200	43200	800	1710
C3-Decalins	30900	38200	732	1590
C4-Decalins	19900	24900	642	1210
Benzo(b)thiophene	37800	57400	1730	2200
C1-Benzo(b)thiophenes	97100	142000	2760	3980
C2-Benzo(b)thiophenes	131000	179000	3370	5200
C3-Benzo(b)thiophenes	66100	86600	1910	3180
C4-Benzo(b)thiophenes	17400	22300	515	882
Naphthalene	2110000 D	4400000 D	76900 D	114000 D
C1-Naphthalenes	2180000 D	3350000 D	57600 D	90000 D
C2-Naphthalenes	1230000	1710000	37700	59200
C3-Naphthalenes	460000	556000	13900	23700
C4-Naphthalenes	114000	136000	3480	6050
Biphenyl	299000 D	533000 D	7980	12900
Acenaphthylene	190000 D	278000 D	5970	11200
Acenaphthene	969000 D	1630000 D	28400 D	49300 D
Dibenzofuran	138000 D	192000 D	2890	4390
Fluorene	480000 D	703000 D	11200	16000
C1-Fluorenes	295000 D	390000 D	8390	14000
C2-Fluorenes	127000	164000	4830	7540
C3-Fluorenes	43800	55600	1650	3650
Anthracene	558000 D	737000 D	11600	29900 D
Phenanthrene	1670000 D	2250000 D	53200 D	98200 D
C1-Phenanthrenes/Anthracenes	946000 D	1210000 D	22800	50200 D
C2-Phenanthrenes/Anthracenes	254000	313000	8750	16300
C3-Phenanthrenes/Anthracenes	61300	82800	2220	4600
C4-Phenanthrenes/Anthracenes	63900	76200	2070	5380
Dibenzothiophene	194000 D	162000	3380	5800
C1-Dibenzothiophenes	140000	169000	4410	7250
C2-Dibenzothiophenes	82700	100000	2740	4920
C3-Dibenzothiophenes	31400	40400	1150	2160
C4-Dibenzothiophenes	7920	10400	342	652
Fluoranthene	600000 D	789000 D	14300	47100 D
Pyrene	882000 D	1170000 D	30700 D	65400 D
C1-Fluoranthenes/Pyrenes	484000	638000	19300	36900
C2-Fluoranthenes/Pyrenes	137000	181000	5290	10400
C3-Fluoranthenes/Pyrenes	32000	42500	1200	2760
Benz[a]anthracene	277000 D	363000 D	8060	17000
Chrysene	255000 D	337000 D	6660	14400
C1-Chrysenes	104000	147000	4120	9040
C2-Chrysenes	41600	59500	1550	3520
C3-Chrysenes	19600	22800	713	1420
C4-Chrysenes	U	U	U	2.58 J
Benzo[b]fluoranthene	88200	118000	4040	9740
Benzo[j/k]fluoranthene	106000	135000	4710	11400
Benzo[e]pyrene	90100	121000	4330	10300
Benzo[a]pyrene	234000 D	314000 D	7890	17400
Perylene	28300	36300	1210	3060
Indeno[1,2,3-c,d]pyrene	84800	113000	3740	9780
Dibenz[a,h]anthracene	20400	26400	889	2340
Benzo[g,h,i]perylene	82300	113000	3970	9960
Total PAH	16200000	24000000	496000	919000
Surrogate Recoveries (%)				
Naphthalene-d8	102	102	109	109
Phenanthrene-d10	89	87	87	87
Chrysene-d12	80	76	95	94

Data Qualifiers
& - Excedes QC criteria
B - Analyte detected < 5x PB.
D - Result from higher dilution.
DO - Surrogate diluted out.
E - Result above calibration curve.
J - Result less than reporting limit.
ME - Matrix effect.
NA - Not applicable.
U - Undetected.

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Project Name Ashland MGP
Project Number N004602-0002

Concentrations of Polycyclic Armatic Hydrocarbons and Other Selected Analytes

Client Sample ID	Upgradient Riser	East Riser	West Riser	Scrapings From Inside Discharge
Battelle Sample ID	W5547-1-D	W5544-1	W5543-1-D	W5551-1-D
Matrix	Aqueous	Aqueous	Aqueous	Solid
Battelle Batch ID	01-550	01-550	01-550	01-550
Analytical Method	8270M	8270M	8270M	8270M
Collection Date	07/24/01	07/24/01	07/24/01	07/25/01
Receipt Date	07/27/01	07/27/01	07/27/01	07/27/01
Extraction Date	07/03/01	07/03/01	07/03/01	08/03/01
Analysis Date	10/25/01	10/25/01	10/25/01	10/25/01
Dilution Factor	34.95	1.05	149.6	32.64
% Moisture	NA	NA	NA	39.83
Sample Size	1.1 L	1.3 L	1.1 L	1.52 g
Units	ng/L	ng/L	ng/L	ug/Kg
Decalin	15700	98.6	69600	9500
C1-Decalins	95300	127	314000	40800
C2-Decalins	132000	99.6	451000	58100
C3-Decalins	106000	126	362000	48600
C4-Decalins	88800	U	311000	39600
Benzo(b)thiophene	108000	19300 D	295000	81600
C1-Benzo(b)thiophenes	219000	18400	840000	178000
C2-Benzo(b)thiophenes	268000	7660	1240000	214000
C3-Benzo(b)thiophenes	138000	1430	709000	101000
C4-Benzo(b)thiophenes	47500	165	232000	31600
Naphthalene	5200000 D	224000 D	12700000 D	4330000 D
C1-Naphthalenes	4250000 D	154000 D	17200000 D	3700000 D
C2-Naphthalenes	2830000	52900	13200000	2160000
C3-Naphthalenes	1060000	7540	5290000	777000
C4-Naphthalenes	336000	979	1510000	200000
Biphenyl	686000 D	3690	2340000	430000
Acenaphthylene	417000	9800	2160000	202000
Acenaphthene	2140000 D	59100 D	9420000 D	1890000 D
Dibenzofuran	210000	5190	998000	164000
Fluorene	1050000 D	16000	5050000 D	859000 D
C1-Fluorenes	681000 D	3570	3450000 D	468000 D
C2-Fluorenes	299000	676	1570000	203000
C3-Fluorenes	120000	372	566000	83500
Anthracene	1230000 D	3850	5880000 D	890000 D
Phenanthrene	4070000 D	15400	19000000 D	3210000 D
C1-Phenanthrenes/Anthracenes	2150000 D	2540	11200000 D	1600000 D
C2-Phenanthrenes/Anthracenes	650000	615	3230000	433000
C3-Phenanthrenes/Anthracenes	173000	195	786000	110000
C4-Phenanthrenes/Anthracenes	175000	198	821000	117000
Dibenzothiophene	292000	2330	1440000	227000
C1-Dibenzothiophenes	321000	584	1570000	229000
C2-Dibenzothiophenes	229000	211	1050000	152000
C3-Dibenzothiophenes	91800	102	418000	59600
C4-Dibenzothiophenes	25800	42.3	119000	16400
Fluoranthene	1590000 D	2530	7720000 D	1190000 D
Pyrene	2450000 D	3520	11600000 D	1820000 D
C1-Fluoranthenes/Pyrenes	1400000	1400	7220000	901000
C2-Fluoranthenes/Pyrenes	386000	434	1880000	251000
C3-Fluoranthenes/Pyrenes	92100	124	440000	60700
Benz[a]anthracene	730000 D	507	3740000 D	413000
Chrysene	707000 D	506	3510000 D	360000
C1-Chrysenes	335000	299	1660000	228000
C2-Chrysenes	126000	126	581000	76000
C3-Chrysenes	54700	86.6	244000	36600
C4-Chrysenes	11300	U	66800	9430
Benzo[b]fluoranthene	312000	326	1570000	198000
Benzo[j/k]fluoranthene	351000	376	1640000	246000
Benzo[e]pyrene	343000	391	1600000	216000
Benzo[a]pyrene	698000 D	579	3480000 D	381000
Perylene	96500	104	468000	65000
Indeno[1,2,3-c,d]pyrene	328000	356	1460000	192000
Dibenz[a,h]anthracene	68800	72.5	337000	43300
Benzo[g,h,i]perylene	328000	423	1540000	195000
Total PAH	39100000	576000	172000000	29400000
Surrogate Recoveries (%)				
Naphthalene-d8	38 &	66	90	81
Phenanthrene-d10	37 &	77	90	85
Chrysene-d12	37 &	74	94	72

Data Qualifiers
& - Excedes QC criteria
B - Analyte detected < 5x PB.
D - Result from higher dilution.
DO - Surrogate diluted out.
E - Result above calibration curve.
J - Result less than reporting limit.
ME - Matrix effect.
NA - Not applicable.
U - Undetected.

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Project Name Ashland MGP
Project Number N004602-0002

Concentrations of Polycyclic Armatic Hydrocarbons and Other Selected Analytes

Client Sample ID	Pipe Discharge	Solids Around Discharge Pipe	MW-7	TW-9
Battelle Sample ID	W5549-1-D	W5550-1-D	W5545-1-D	W5546-1-D
Matrix	Aqueous	Solid	Aqueous	Aqueous
Battelle Batch ID	01-550	01-550	01-550	01-550
Analytical Method	8270M	8270M	8270M	8270M
Collection Date	07/25/01	07/25/01	07/24/01	07/24/01
Receipt Date	07/27/01	07/27/01	07/27/01	07/27/01
Extraction Date	07/03/01	06/03/01	07/03/01	07/03/01
Analysis Date	10/25/01	10/24/01	10/25/01	10/25/01
Dilution Factor	2624.48	3.73	4.19	4199.16
% Moisture	NA	19.76	NA	NA
Sample Size	1.2 L	2.11 g	1.1 L	1.2 L
Units	ng/L	ug/Kg	ng/L	ng/L
Decalin	757000	928	1780	1090000
C1-Decalins	4780000	7760	14600	9170000
C2-Decalins	6120000	12100	23400	11800000
C3-Decalins	4960000	10300	23700	10300000
C4-Decalins	3630000	7940	21400	10100000
Benzo(b)thiophene	7270000	3390	19700	5480000
C1-Benzo(b)thiophenes	15800000	7280	29600	11800000
C2-Benzo(b)thiophenes	19300000	11600	40400	16300000
C3-Benzo(b)thiophenes	9230000	9220	21300	9970000
C4-Benzo(b)thiophenes	2830000	3930	9240	4290000
Naphthalene	412000000 D	136000 D	63500 D	1000000000 D
C1-Naphthalenes	339000000 D	131000 D	172000 D	521000000 D
C2-Naphthalenes	200000000	137000	308000	262000000
C3-Naphthalenes	71400000	71300	160000	98800000
C4-Naphthalenes	20700000	20500	61800	39000000
Biphenyl	58400000 D	21300	28400	193000000 D
Acenaphthylene	33600000	16000	28600	34000000
Acenaphthene	159000000 D	75300 D	172000 D	405000000 D
Dibenzofuran	15400000	10200	21000	11100000
Fluorene	80200000 D	29900	75200 D	168000000 D
C1-Fluorenes	48800000 D	29900	63500 D	89700000 D
C2-Fluorenes	21100000	15700	39500	39700000
C3-Fluorenes	7990000	6490	17100	16400000
Anthracene	89900000 D	43700 D	70100 D	158000000 D
Phenanthrene	309000000 D	160000 D	214000 D	645000000 D
C1-Phenanthrenes/Anthracenes	156000000 D	75600	170000 D	253000000 D
C2-Phenanthrenes/Anthracenes	45900000	31600	78500	75600000
C3-Phenanthrenes/Anthracenes	11100000	8210	24100	21900000
C4-Phenanthrenes/Anthracenes	10500000	7540	20400	18300000
Dibenzothiophene	20900000	14200	25700	17900000
C1-Dibenzothiophenes	21600000	18800	38000	23900000
C2-Dibenzothiophenes	14600000	12000	33500	20400000
C3-Dibenzothiophenes	5710000	4700	14800	10600000
C4-Dibenzothiophenes	1460000	1360	4700	3200000
Fluoranthene	117000000 D	63300 D	102000 D	221000000 D
Pyrene	178000000 D	95400 D	156000 D	371000000 D
C1-Fluoranthenes/Pyrenes	97200000	60600	123000	170000000
C2-Fluoranthenes/Pyrenes	25900000	19400	42900	44900000
C3-Fluoranthenes/Pyrenes	5820000	4840	12300	10300000
Benz[a]anthracene	53200000 D	26200	49900	100000000 D
Chrysene	50100000 D	22200	45600	95700000 D
C1-Chrysenes	22500000	16400	33400	34900000
C2-Chrysenes	7340000	6410	14300	12700000
C3-Chrysenes	3440000	3620	7950	6040000
C4-Chrysenes	869000	832	2530	1470000
Benzo[b]fluoranthene	19200000	13300	26400	38200000
Benzo[j/k]fluoranthene	24100000	15200	31400	42300000
Benzo[e]pyrene	21900000	14400	30500	47100000
Benzo[a]pyrene	46900000 D	23800	50700	94300000 D
Perylene	6200000	4440	7860	11700000
Indeno[1,2,3-c,d]pyrene	20400000	13800	27600	39700000
Dibenz[a,h]anthracene	4650000	3110	6230	8330000
Benzo[g,h,i]perylene	19800000	15100	30600	42200000
Total PAH	2880000000	1500000	2710000	5520000000
Surrogate Recoveries (%)				
Naphthalene-d8	DO	63	40	DO
Phenanthrene-d10	DO	73	44	DO
Chrysene-d12	DO	62	39 &	DO

Data Qualifiers
& - Exceedes QC criteria
B - Analyte detected < 5x PB.
D - Result from higher dilution.
DO - Surrogate diluted out.
E - Result above calibration curve.
J - Result less than reporting limit.
ME - Matrix effect.
NA - Not applicable.
U - Undetected.

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Instrument Control Checks

Project Name Ashland MGP
Project Number N004602-0002

Client Sample ID	True Value	SQD292 D0746			True Value			
Battelle Sample ID	FP83	FP83			FP15	FP15		
Battelle Batch ID		01-546/01-550				01-610		
Analytical Method		8270M				8270M		
Data File		D0746.D				D0746.D		
Collection Date		NA				NA		
Receipt Date		NA				NA		
Extraction Date		NA				NA		
Analysis Date		10/16/01				11/21/01		
Dilution Factor		1				1		
% Moisture		NA				NA		
Sample Size		1 uL				1 uL		
Units	ng/uL	ng/uL	% D	Q	ng/uL	ng/uL	% D	Q
Decalin		U				U		
C1-Decalins		U				U		
C2-Decalins		U				U		
C3-Decalins		U				U		
C4-Decalins		U				U		
Benzo(b)thiophene		U				U		
C1-Benzo(b)thiophenes		U				U		
C2-Benzo(b)thiophenes		U				U		
C3-Benzo(b)thiophenes		U				U		
C4-Benzo(b)thiophenes		U				U		
Naphthalene	1.00	1.14	13.8		1.00	1.06	5.8	
C1-Naphthalenes		U				U		
C2-Naphthalenes		U				U		
C3-Naphthalenes		U				U		
C4-Naphthalenes		U				U		
Biphenyl	1.00	1.1	9.7		1.00	1.04	3.7	
Acenaphthylene	1.00	1.1	9.5		1.00	0.97	-3.4	
Acenaphthene	1.00	1.12	11.5		1.00	1.03	2.6	
Dibenzofuran		U				U		
Fluorene	1.00	1.11	10.5		1.00	1.04	3.5	
C1-Fluorenes		U				U		
C2-Fluorenes		U				U		
C3-Fluorenes		U				U		
Anthracene	1.01	1.1	9.4		1.01	1.07	6.4	
Phenanthrene	1.01	1.11	10.2		1.01	1.05	4.3	
C1-Phenanthrenes/Anthracenes		U				U		
C2-Phenanthrenes/Anthracenes		U				U		
C3-Phenanthrenes/Anthracenes		U				U		
C4-Phenanthrenes/Anthracenes		U				U		
Dibenzothiophene		U				U		
C1-Dibenzothiophenes		U				U		
C2-Dibenzothiophenes		U				U		
C3-Dibenzothiophenes		U				U		
C4-Dibenzothiophenes		U				U		
Fluoranthene	1.00	1.13	12.5		1.00	1.02	1.6	
Pyrene	1.00	1.13	12.7		1.00	1.06	5.7	
C1-Fluoranthenes/Pyrenes		U				U		
C2-Fluoranthenes/Pyrenes		U				U		
C3-Fluoranthenes/Pyrenes		U				U		
Benz[a]anthracene	1.01	1.06	5.4		1.01	0.98	-2.5	
Chrysene	1.00	1.12	11.5		1.00	1.09	8.5	
C1-Chrysenes		U				U		
C2-Chrysenes		U				U		
C3-Chrysenes		U				U		
C4-Chrysenes		U				U		
Benzo[b]fluoranthene	1.00	1.06	5.6		1.00	1.01	0.6	
Benzo[j/k]fluoranthene	1.00	1.22	21.7		1.00	1.15	14.7	
Benzo[e]pyrene	1.03	1.13	9.8		1.03	1.09	5.9	
Benzo[a]pyrene	1.01	1.11	10.3		1.01	1.08	7.3	
Perylene	1.00	1.11	11.0		1.00	1.06	6.0	
Indeno[1,2,3-c,d]pyrene	1.00	1.03	2.7		1.00	0.94	-6.3	
Dibenz[a,h]anthracene	1.00	1.07	6.7		1.00	0.97	-3.2	
Benzo[g,h,i]perylene	1.00	1.07	6.6		1.00	0.96	-4.3	
Surrogate Recoveries (%)								
Naphthalene-d8		116				116		
Phenanthrene-d10		105				105		
Chrysene-d12		109				109		

Data Qualifiers
& - Excedes QC criteria
B - Analyte detected < 5x PB.
D - Result from higher dilution.
DO - Surrogate diluted out.
E - Result above calibration curve.
J - Result less than reporting limit.
ME - Matrix effect.
NA - Not applicable.
U - Undetected.

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Procedural Blanks

Project Name Ashland MGP
Project Number N004602-0002

Client Sample ID	Procedural Blank	Procedural Blank	Procedural Blank
Battelle Sample ID	ZK90PB	ZL25PB	ZO16PB
Matrix	Solid	Aqueous	Oil
Battelle Batch ID	01-546	01-550	01-610
Analytical Method	8270M	8270M	8270M
Data File	D0747.D	D0749.D	D0749.D
Collection Date	NA	NA	NA
Receipt Date	NA	NA	NA
Extraction Date	10/10/01	10/08/01	11/20/01
Analysis Date	10/16/01	10/16/01	11/22/01
Dilution Factor	1	1	1
% Moisture	NA	NA	NA
Sample Size	7.07 g	1 L	0.0511 g
Units	ug/Kg	ng/L	mg/kg
Decalin	U	U	U
C1-Decalins	U	U	U
C2-Decalins	U	U	U
C3-Decalins	U	U	U
C4-Decalins	U	U	U
Benzo(b)thiophene	U	U	U
C1-Benzo(b)thiophenes	U	U	U
C2-Benzo(b)thiophenes	U	U	U
C3-Benzo(b)thiophenes	U	U	U
C4-Benzo(b)thiophenes	U	U	U
Naphthalene	0.83 J	9.2 J	0.48 J
C1-Naphthalenes	U	2.06 J	0.48 J
C2-Naphthalenes	U	U	U
C3-Naphthalenes	U	U	U
C4-Naphthalenes	U	U	U
Biphenyl	U	U	U
Acenaphthylene	U	U	U
Acenaphthene	U	U	U
Dibenzofuran	U	U	U
Fluorene	U	U	U
C1-Fluorenes	U	U	U
C2-Fluorenes	U	U	U
C3-Fluorenes	U	U	U
Anthracene	U	U	U
Phenanthrene	U	U	U
C1-Phenanthrenes/Anthracenes	U	U	U
C2-Phenanthrenes/Anthracenes	U	U	U
C3-Phenanthrenes/Anthracenes	U	U	U
C4-Phenanthrenes/Anthracenes	U	U	U
Dibenzothiophene	U	U	U
C1-Dibenzothiophenes	U	U	U
C2-Dibenzothiophenes	U	U	U
C3-Dibenzothiophenes	U	U	U
C4-Dibenzothiophenes	U	U	U
Fluoranthene	U	0.40 J	U
Pyrene	U	1.69 J	U
C1-Fluoranthenes/Pyrenes	U	U	U
C2-Fluoranthenes/Pyrenes	U	U	U
C3-Fluoranthenes/Pyrenes	U	U	U
Benz[a]anthracene	U	U	U
Chrysene	U	U	U
C1-Chrysenes	U	U	U
C2-Chrysenes	U	U	U
C3-Chrysenes	U	U	U
C4-Chrysenes	U	U	U
Benzo[b]fluoranthene	U	U	U
Benzo[j/k]fluoranthene	U	U	U
Benzo[e]pyrene	U	U	U
Benzo[a]pyrene	U	U	U
Perylene	U	U	U
Indeno[1,2,3-c,d]pyrene	U	U	U
Dibenz[a,h]anthracene	U	U	U
Benzo[g,h,i]perylene	U	U	U
Total PAH	0.83	13.3	1.0 J
Surrogate Recoveries (%)			
Naphthalene-d8	118	109	109
Phenanthrene-d10	106	97	102
Chrysene-d12	108	109	100

Data Qualifiers
& - Excedes QC criteria
B - Analyte detected < 5x PB.
D - Result from higher dilution.
DO - Surrogate diluted out.
E - Result above calibration curve.
J - Result less than reporting limit.
ME - Matrix effect.
NA - Not applicable.
U - Undetected.

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Laboratory Control Samples

Project Name Ashland MGP
Project Number N004602-0002

Client Sample ID	True Value	Laboratory Control Spike			Laboratory Control Spike		
Battelle Sample ID		ZK91LCS			ZL26LCS		
Battelle Batch ID		01-546			01-550		
Analytical Method		8270M			8270M		
Data File		D0748.D			D0750.D		
Collection Date		NA			NA		
Receipt Date		NA			NA		
Extraction Date		10/10/01			10/08/01		
Analysis Date		10/16/01			10/16/01		
Dilution Factor		1			1		
% Moisture		NA			NA		
Sample Size		1			1		
Units	FN96	ng	%R	Q	ng	%R	Q
Decalin	538.2	670	124	&	585	109	
C1-Decalins		U			U		
C2-Decalins		U			U		
C3-Decalins		U			U		
C4-Decalins		U			U		
Benzo(b)thiophene	489.52	574	117		529	108	
C1-Benzo(b)thiophenes		U			U		
C2-Benzo(b)thiophenes		U			U		
C3-Benzo(b)thiophenes		U			U		
C4-Benzo(b)thiophenes		U			U		
Naphthalene	500	597	119		563	113	
C1-Naphthalenes		U			U		
C2-Naphthalenes		U			U		
C3-Naphthalenes		U			U		
C4-Naphthalenes		U			U		
Biphenyl	501.5	573	114		549	109	
Acenaphthylene	500	543	109		501	100	
Acenaphthene	500	570	114		544	109	
Dibenzofuran	500	576	115		543	109	
Fluorene	500	585	117		563	113	
C1-Fluorenes		U			U		
C2-Fluorenes		U			U		
C3-Fluorenes		U			U		
Anthracene	500	546	109		530	106	
Phenanthrene	500	531	106		521	104	
C1-Phenanthrenes/Anthracenes		U			U		
C2-Phenanthrenes/Anthracenes		U			U		
C3-Phenanthrenes/Anthracenes		U			U		
C4-Phenanthrenes/Anthracenes		U			U		
Dibenzothiophene	504.12	554	110		488	96.8	
C1-Dibenzothiophenes		U			U		
C2-Dibenzothiophenes		U			U		
C3-Dibenzothiophenes		U			U		
C4-Dibenzothiophenes		U			U		
Fluoranthene	510.75	547	107		563	110	
Pyrene	512.75	590	115		595	116	
C1-Fluoranthenes/Pyrenes		U			U		
C2-Fluoranthenes/Pyrenes		U			U		
C3-Fluoranthenes/Pyrenes		U			U		
Benz[a]anthracene	500	562	112		582	116	
Chrysene	500.25	555	111		593	118	
C1-Chrysenes		U			U		
C2-Chrysenes		U			U		
C3-Chrysenes		U			U		
C4-Chrysenes		U			U		
Benzo[b]fluoranthene	500	547	109		580	116	
Benzo[j/k]fluoranthene	500	610	122	&	660	132	&
Benzo[e]pyrene	514.6	567	110		590	115	
Benzo[a]pyrene	500	531	106		588	118	
Perylene	500.1	497	99.4		570	114	
Indeno[1,2,3-c,d]pyrene	500.25	483	96.6		541	108	
Dibenz[a,h]anthracene	500	481	96.2		524	105	
Benzo[g,h,i]perylene	500	506	101		570	114	
Surrogate Recoveries (%)							
Naphthalene-d8		114			105		
Phenanthrene-d10		94			92		
Chrysene-d12		104			110		

Data Qualifiers
& - Excedes QC criteria
B - Analyte detected < 5x PB.
D - Result from higher dilution.
DO - Surrogate diluted out.
E - Result above calibration curve.
J - Result less than reporting limit.
ME - Matrix effect.
NA - Not applicable.
U - Undetected.

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Laboratory Control Samples

Project Name Ashland MGP
Project Number N004602-0002

Client Sample ID	True Value	Laboratory Control Spike			Laboratory Control Spike		
Battelle Sample ID		ZO18LCS			ZO19LSCD		
Battelle Batch ID		01-610			01-610		
Analytical Method		8270M			8270M		
Data File							
Collection Date		NA			NA		
Receipt Date		NA			NA		
Extraction Date		11/20/01			11/20/01		
Analysis Date		11/22/01			11/22/01		
Dilution Factor		1			1		
% Moisture		NA			NA		
Sample Size		1			1		
Units	FQ74	ng	%R	Q	ng	%R	Q
Decalin		U			U		
C1-Decalins		U			U		
C2-Decalins		U			U		
C3-Decalins		U			U		
C4-Decalins		U			U		
Benzo(b)thiophene		U			U		
C1-Benzo(b)thiophenes		U			U		
C2-Benzo(b)thiophenes		U			U		
C3-Benzo(b)thiophenes		U			U		
C4-Benzo(b)thiophenes		U			U		
Naphthalene	2004	2150	107		2217	111	
C1-Naphthalenes		U			U		
C2-Naphthalenes		U			U		
C3-Naphthalenes		U			U		
C4-Naphthalenes		U			U		
Biphenyl	2006	2134	106		2202	110	
Acenaphthylene	2008	2082	104		2130	106	
Acenaphthene	2008	2172	108		2226	111	
Dibenzofuran		U			U		
Fluorene	2009	2214	110		2247	112	
C1-Fluorenes		U			U		
C2-Fluorenes		U			U		
C3-Fluorenes		U			U		
Anthracene	2011	2255	112		2325	116	
Phenanthrene	2014	2170	108		2267	113	
C1-Phenanthrenes/Anthracenes		U			U		
C2-Phenanthrenes/Anthracenes		U			U		
C3-Phenanthrenes/Anthracenes		U			U		
C4-Phenanthrenes/Anthracenes		U			U		
Dibenzothiophene		U			U		
C1-Dibenzothiophenes		U			U		
C2-Dibenzothiophenes		U			U		
C3-Dibenzothiophenes		U			U		
C4-Dibenzothiophenes		U			U		
Fluoranthene	2008	2138	106		2193	109	
Pyrene	2006	2223	111		2269	113	
C1-Fluoranthenes/Pyrenes		U			U		
C2-Fluoranthenes/Pyrenes		U			U		
C3-Fluoranthenes/Pyrenes		U			U		
Benz[a]anthracene	2011	2129	106		2145	107	
Chrysene	2009	2277	113		2319	115	
C1-Chrysenes		U			U		
C2-Chrysenes		U			U		
C3-Chrysenes		U			U		
C4-Chrysenes		U			U		
Benzo[b]fluoranthene	2007	2175	108		2222	111	
Benzo[j/k]fluoranthene	2005	2348	117		2393	119	
Benzo[e]pyrene	2058	2239	109		2315	112	
Benzo[a]pyrene	2013	2269	113		2319	115	
Perylene	2000	2238	112		2280	114	
Indeno[1,2,3-c,d]pyrene	2007	2147	107		2181	109	
Dibenz[a,h]anthracene	2005	2214	110		2240	112	
Benzo[g,h,i]perylene	2007	2117	105		2144	107	
Surrogate Recoveries (%)							
Naphthalene-d8		113			113		
Phenanthrene-d10		105			106		
Chrysene-d12		111			111		

Data Qualifiers
& - Excedes QC criteria
B - Analyte detected < 5x PB.
D - Result from higher dilution.
DO - Surrogate diluted out.
E - Result above calibration curve.
J - Result less than reporting limit.
ME - Matrix effect.
NA - Not applicable.
U - Undetected.

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Duplicate Precision

Project Name Ashland MGP
Project Number N004602-0002

Client Sample ID	Laboratory Control Spike		Laboratory Control Spike			
Battelle Sample ID	ZO18LCS		ZO19LSCD			
Battelle Batch ID	01-610		01-610			
Analytical Method	8270M		8270M			
Data File						
Collection Date	NA		NA			
Receipt Date	NA		NA			
Extraction Date	11/20/01		11/20/01			
Analysis Date	11/22/01		11/22/01			
Dilution Factor	1		1			
% Moisture	NA		NA			
Sample Size	1		1			
Units	ng		ng		RPD	Q
Decalin		U		U		
C1-Decalins		U		U		
C2-Decalins		U		U		
C3-Decalins		U		U		
C4-Decalins		U		U		
Benzo(b)thiophene		U		U		
C1-Benzo(b)thiophenes		U		U		
C2-Benzo(b)thiophenes		U		U		
C3-Benzo(b)thiophenes		U		U		
C4-Benzo(b)thiophenes		U		U		
Naphthalene	2150		2217		3.1	
C1-Naphthalenes		U		U		
C2-Naphthalenes		U		U		
C3-Naphthalenes		U		U		
C4-Naphthalenes		U		U		
Biphenyl	2134		2202		3.1	
Acenaphthylene	2082		2130		2.3	
Acenaphthene	2172		2226		2.5	
Dibenzofuran		U		U		
Fluorene	2214		2247		1.5	
C1-Fluorenes		U		U		
C2-Fluorenes		U		U		
C3-Fluorenes		U		U		
Anthracene	2255		2325		3.1	
Phenanthrene	2170		2267		4.4	
C1-Phenanthrenes/Anthracenes		U		U		
C2-Phenanthrenes/Anthracenes		U		U		
C3-Phenanthrenes/Anthracenes		U		U		
C4-Phenanthrenes/Anthracenes		U		U		
Dibenzothiophene		U		U		
C1-Dibenzothiophenes		U		U		
C2-Dibenzothiophenes		U		U		
C3-Dibenzothiophenes		U		U		
C4-Dibenzothiophenes		U		U		
Fluoranthene	2138		2193		2.5	
Pyrene	2223		2269		2.0	
C1-Fluoranthenes/Pyrenes		U		U		
C2-Fluoranthenes/Pyrenes		U		U		
C3-Fluoranthenes/Pyrenes		U		U		
Benzo[a]anthracene	2129		2145		0.7	
Chrysene	2277		2319		1.8	
C1-Chrysenes		U		U		
C2-Chrysenes		U		U		
C3-Chrysenes		U		U		
C4-Chrysenes		U		U		
Benzo[b]fluoranthene	2175		2222		2.1	
Benzo[j/k]fluoranthene	2348		2393		1.9	
Benzo[e]pyrene	2239		2315		3.3	
Benzo[a]pyrene	2269		2319		2.2	
Perylene	2238		2280		1.9	
Indeno[1,2,3-c,d]pyrene	2147		2181		1.6	
Dibenz[a,h]anthracene	2214		2240		1.2	
Benzo[g,h,i]perylene	2117		2144		1.3	
Total PAH	41691		42634		2.2	
Surrogate Recoveries (%)						
Naphthalene-d8	113		113		0.0	
Phenanthrene-d10	105		106		0.9	
Chrysene-d12	111		111		0.0	

Data Qualifiers
& - Excedes QC criteria
B - Analyte detected < 5x PB.
D - Result from higher dilution.
DO - Surrogate diluted out.
E - Result above calibration curve.
J - Result less than reporting limit.
ME - Matrix effect.
NA - Not applicable.
U - Undetected.

Attachment 5

Guidance for Reviewing PAH Histograms

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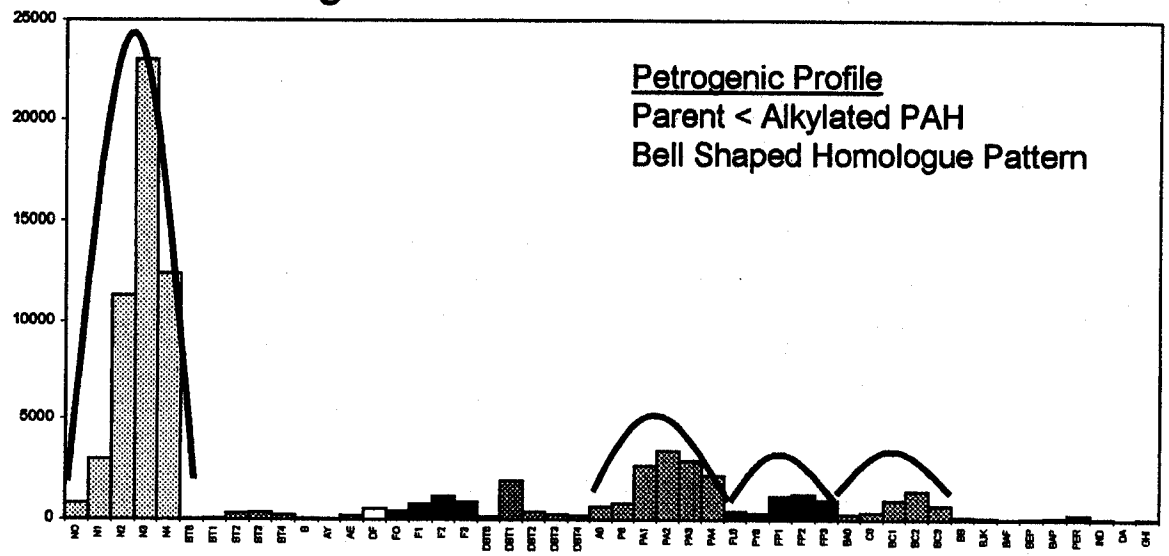
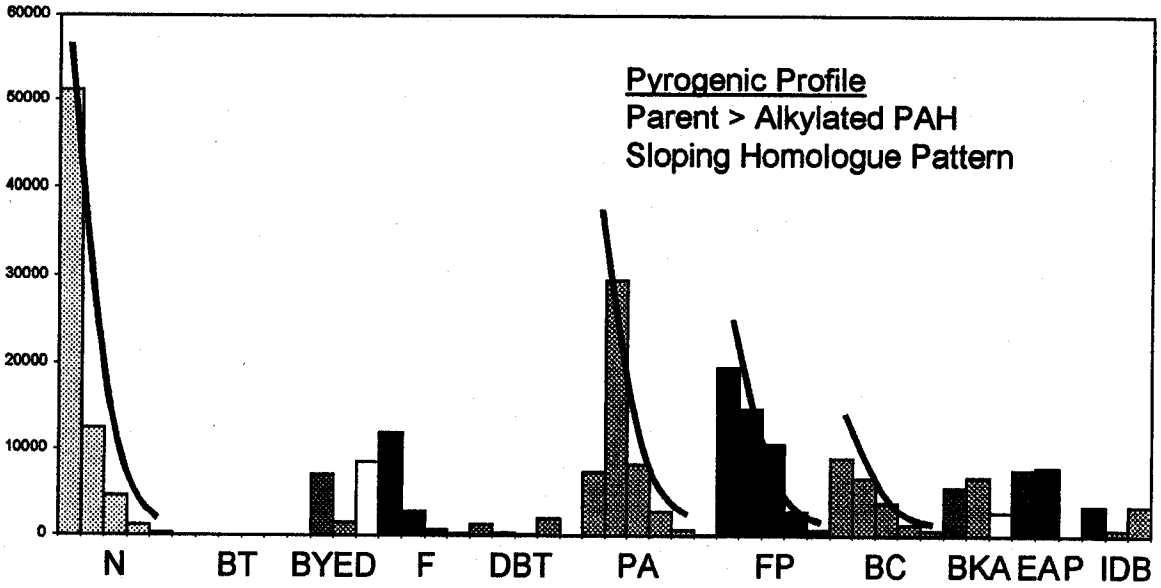
Figure A5-1. Example PAH Histograms of Petrogenic and Pyrogenic Reference Materials

High Volatile Bituminous Coal



Axis Label Mode: Individual Analyte Abbreviations

Coal Tar

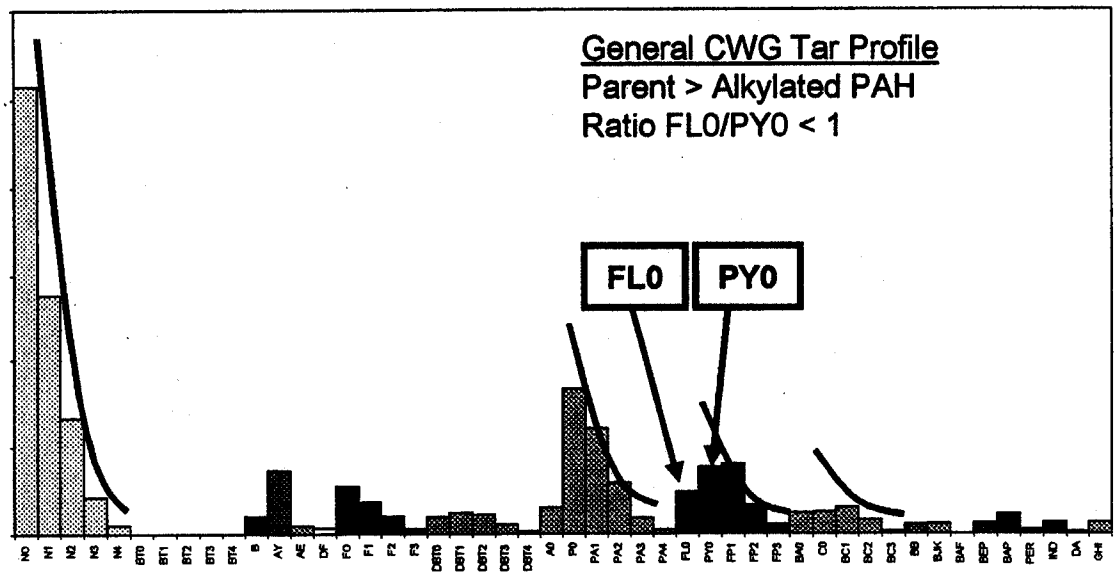


Axis Label Mode: Group Analyte Abbreviations

Reference: Emsbo-Mattingly, S.D., et al., 2001

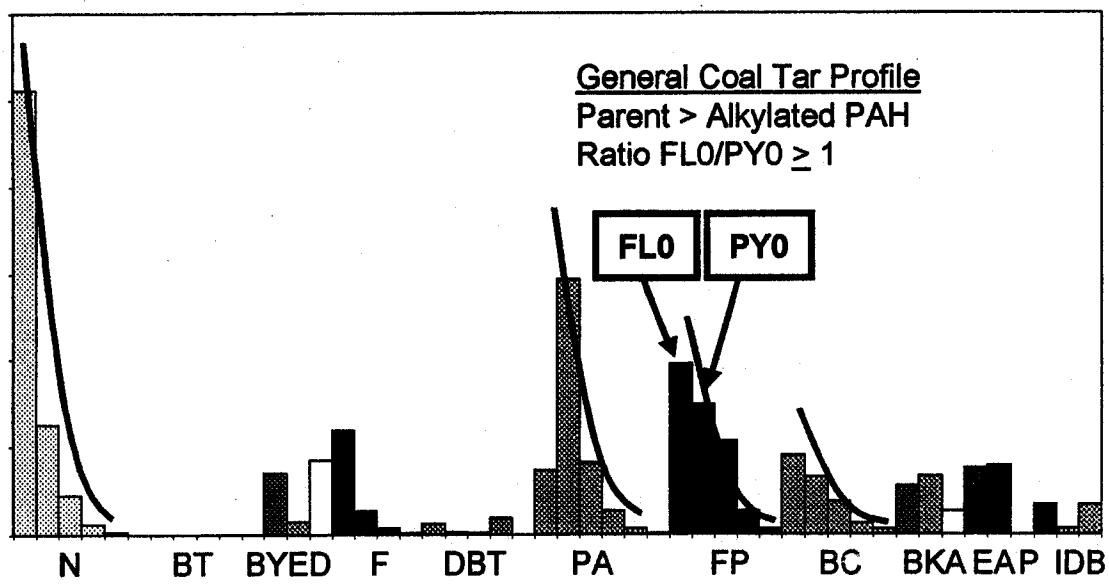
Figure A5-2.
Example PAH Histograms of Coal and CWG Tar Reference Materials

Carbureted Water Gas (CWG) Tar



Axis Label Mode: Individual Analyte Abbreviations

Coal Tar



Axis Label Mode: Group Analyte Abbreviations

Reference: Emsbo-Mattingly, S.D., et al., 2001

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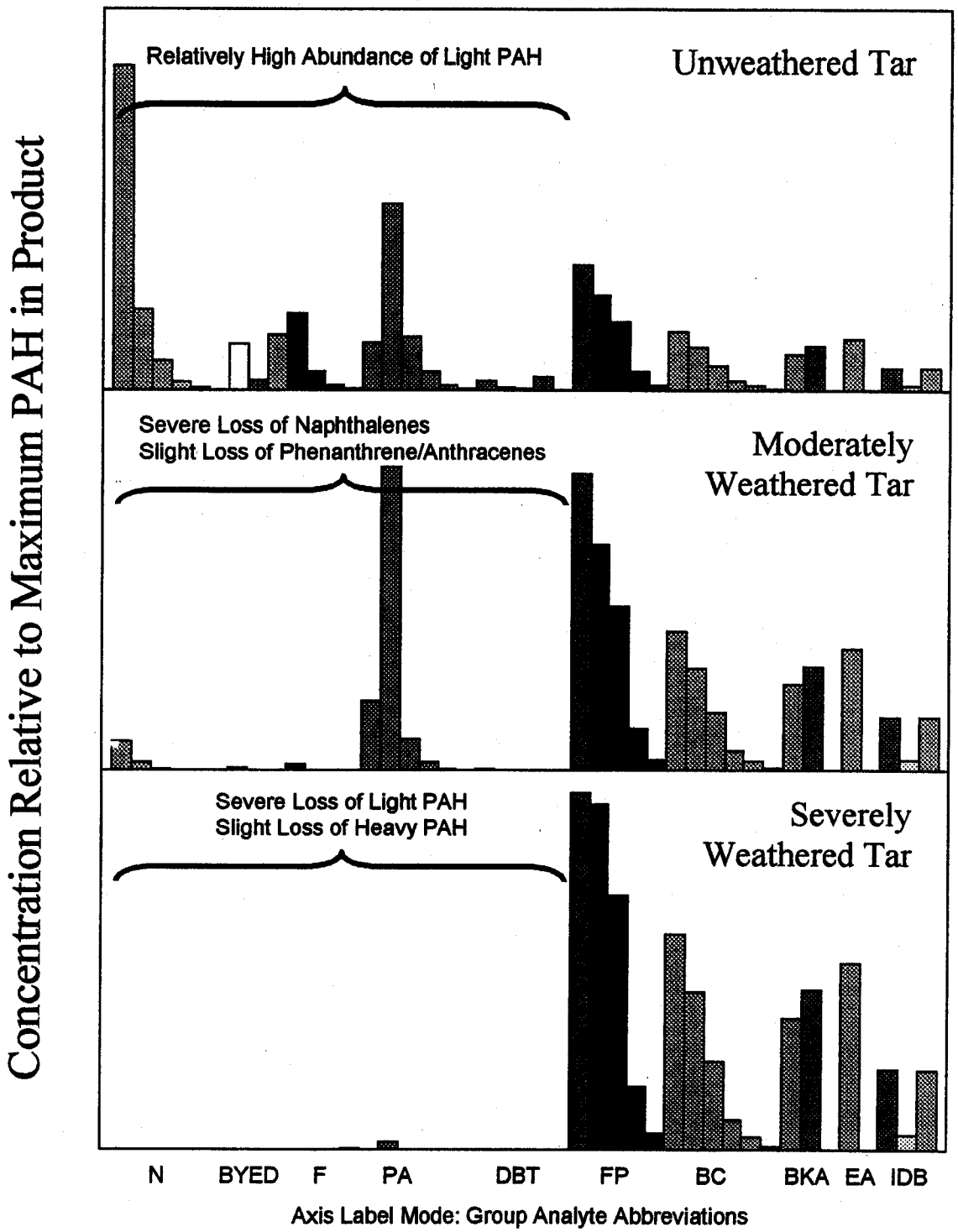
Figure A5-3.
Example PAH Histograms
Degradation of a Pyrogenic Signature



Reference: Emsbo-Mattingly, S.D., et al., 2001

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Table A5-1.
PAH Analytes Plotted on Histograms

Target Analyte	Abbreviation	
	Individual (Large Histogram)	Group (Small Histogram)
Naphthalene	NO	N
C1-Naphthalenes	N1	
C2-Naphthalenes	N2	
C3-Naphthalenes	N3	
C4-Naphthalenes	N4	
Benzo(b)thiophene	BT0	BT
C1-Benzo(b)thiophenes	BT1	
C2-Benzo(b)thiophenes	BT2	
C3-Benzo(b)thiophenes	BT3	
C4-Benzo(b)thiophenes	BT4	
Biphenyl	B	B
Acenaphthylene	AY	Y
Acenaphthene	AE	E
Dibenzofuran	DF	D
Fluorene	FO	F
C1-Fluorenes	F1	
C2-Fluorenes	F2	
C3-Fluorenes	F3	
Dibenzothiophene	DBT0	DBT
C1-Dibenzothiophenes	DBT1	
C2-Dibenzothiophenes	DBT2	
C3-Dibenzothiophenes	DBT3	
C4-Dibenzothiophenes	DBT4	
Anthracene	A0	PA
Phenanthrene	P0	
C1-Phenanthrenes/Anthracenes	PA1	
C2-Phenanthrenes/Anthracenes	PA2	
C3-Phenanthrenes/Anthracenes	PA3	
C4-Phenanthrenes/Anthracenes	PA4	
Fluoranthene	FL0	FP
Pyrene	PY0	
C1-Fluoranthenes/Pyrenes	FP1	
C2-Fluoranthenes/Pyrenes	FP2	
C3-Fluoranthenes/Pyrenes	FP3	
Benz[a]anthracene	BA0	BC
Chrysene	C0	
C1-Chrysenes	BC1	
C2-Chrysenes	BC2	
C3-Chrysenes	BC3	
Benzo[b]fluoranthene	BB	B
Benzo[j/k]fluoranthene	BJK	K
Benzo[a]fluoranthene	BAF	A
Benzo[e]pyrene	BEP	E
Benzo[a]pyrene	BAP	A
Perylene	PER	P
Indeno[1,2,3-c,d]pyrene	IND	I
Dibenz[a,h]anthracene	DA	D
Benzo[g,h,i]perylene	GHI	B

Attachment 6

PAH Histograms

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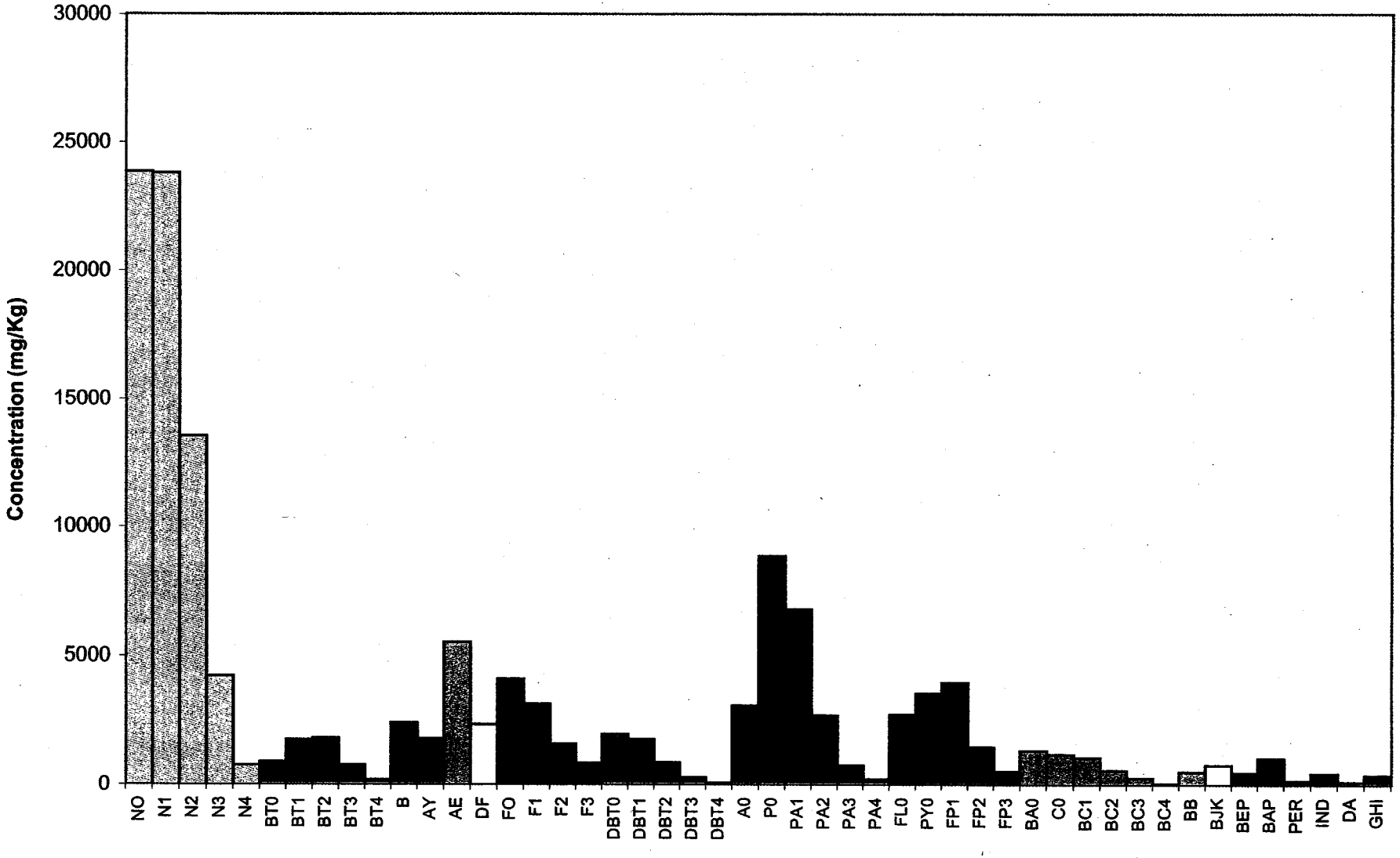
Alkylated PAH Histogram

Sample ID:

2 Inch Steel Pipe



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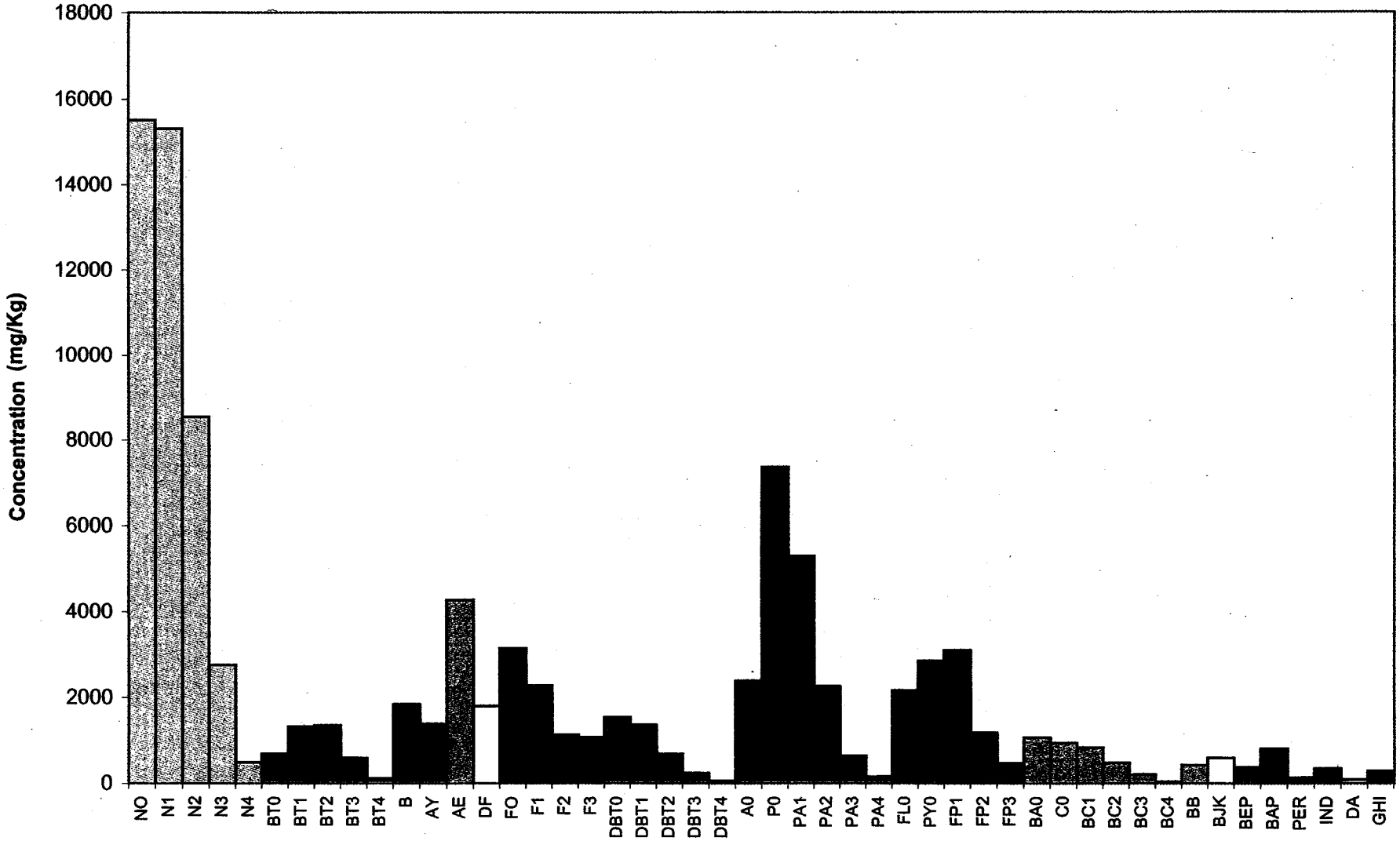
Alkylated PAH Histogram

Sample ID:

12 Inch Steel Pipe



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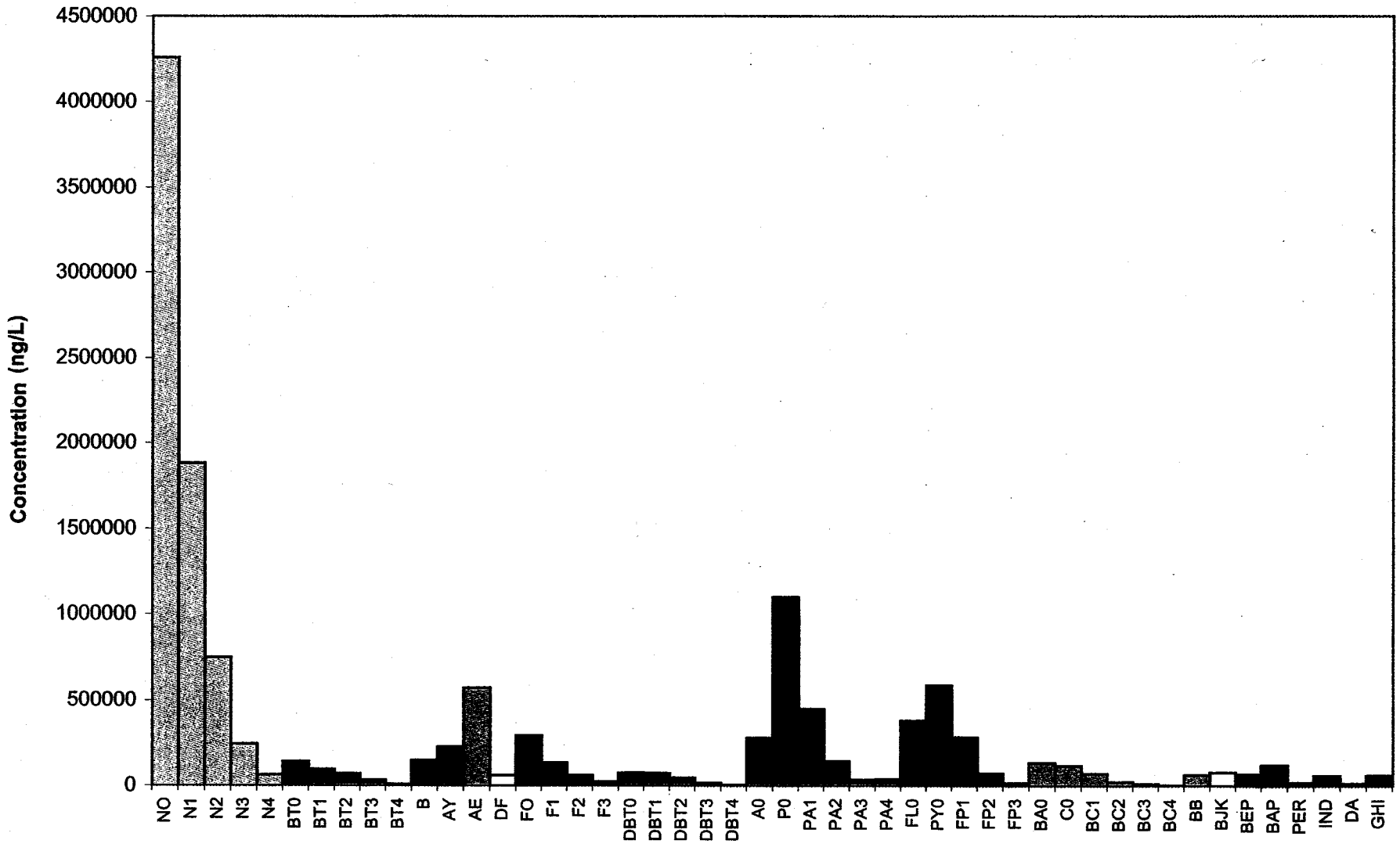
Alkylated PAH Histogram

Sample ID:
TW-13



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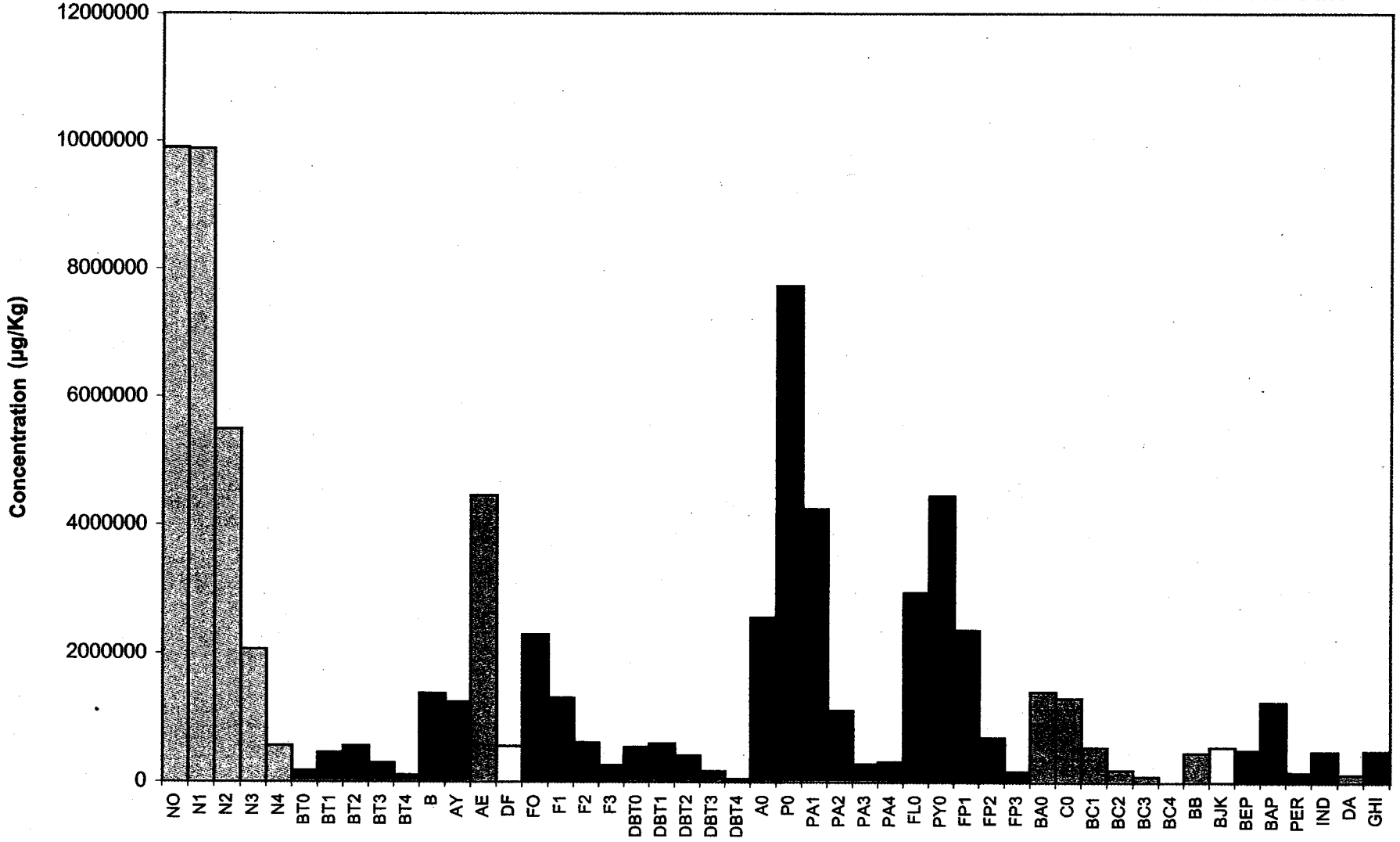
Alkylated PAH Histogram

Sample ID:
Pipe Discharge
East Yard Gate



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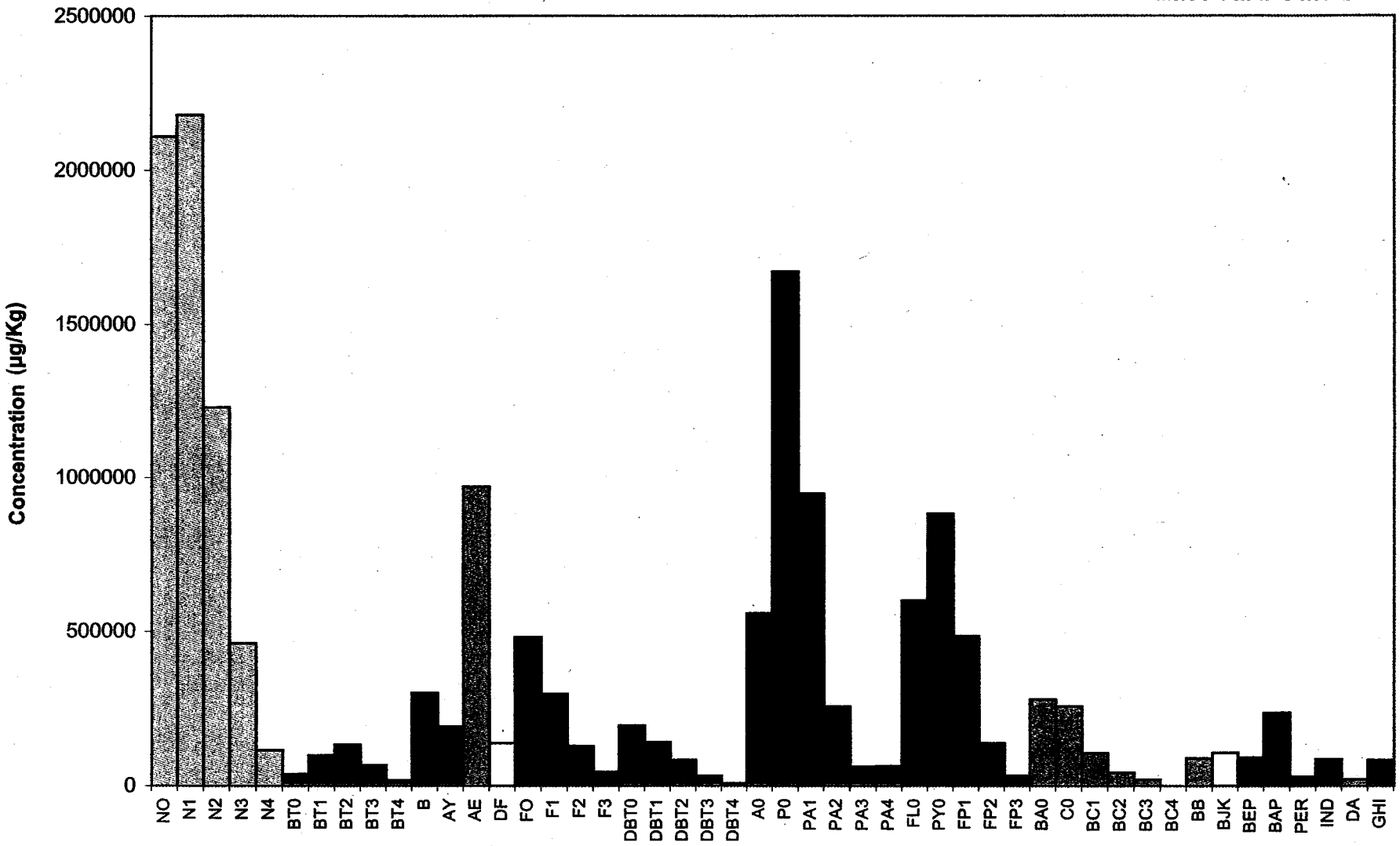
Alkylated PAH Histogram

Sample ID:
Pipe Sludge
East Yard Gate #1



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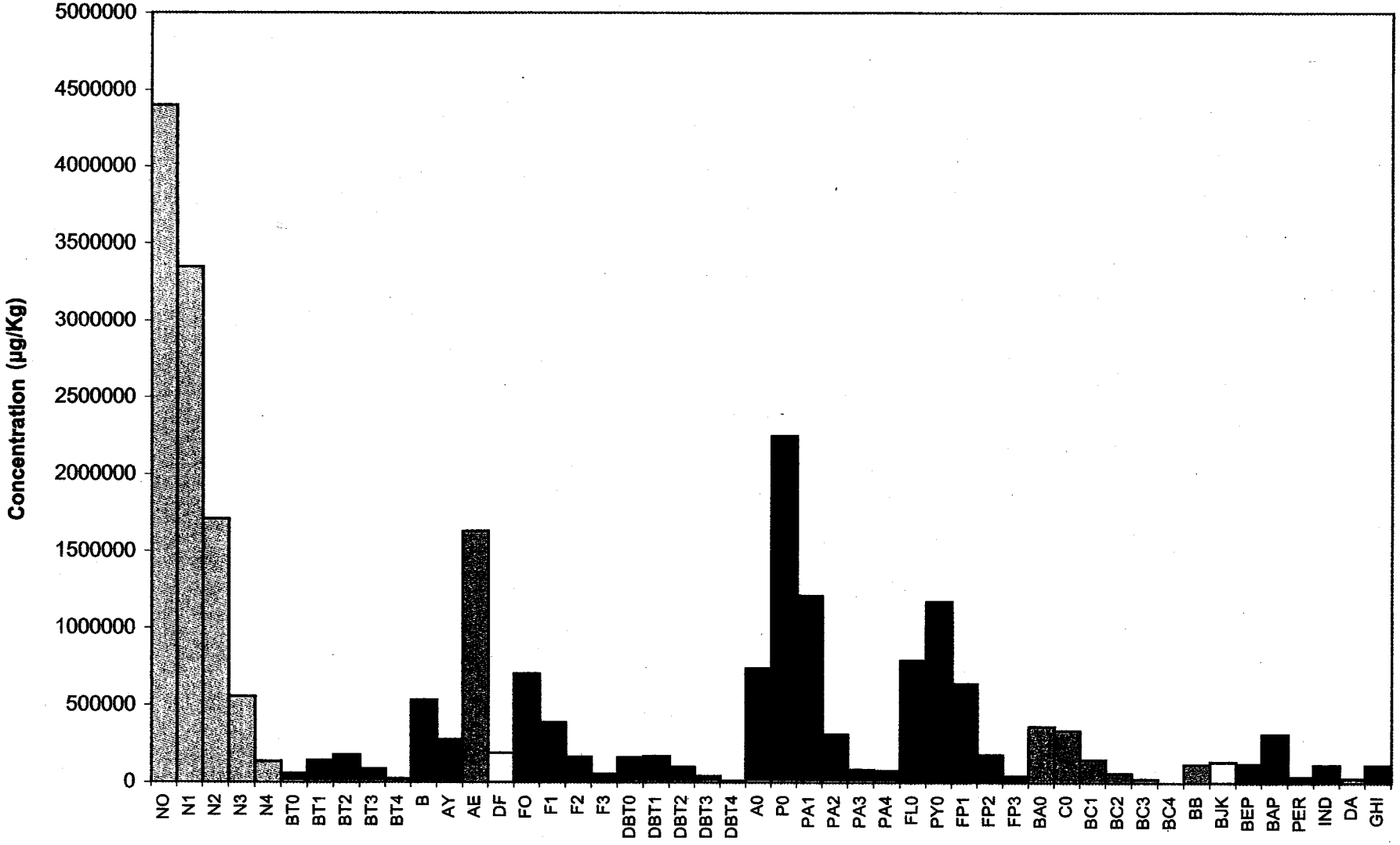
Alkylated PAH Histogram

Sample ID:
Pipe Sludge
East Yard Gate #2



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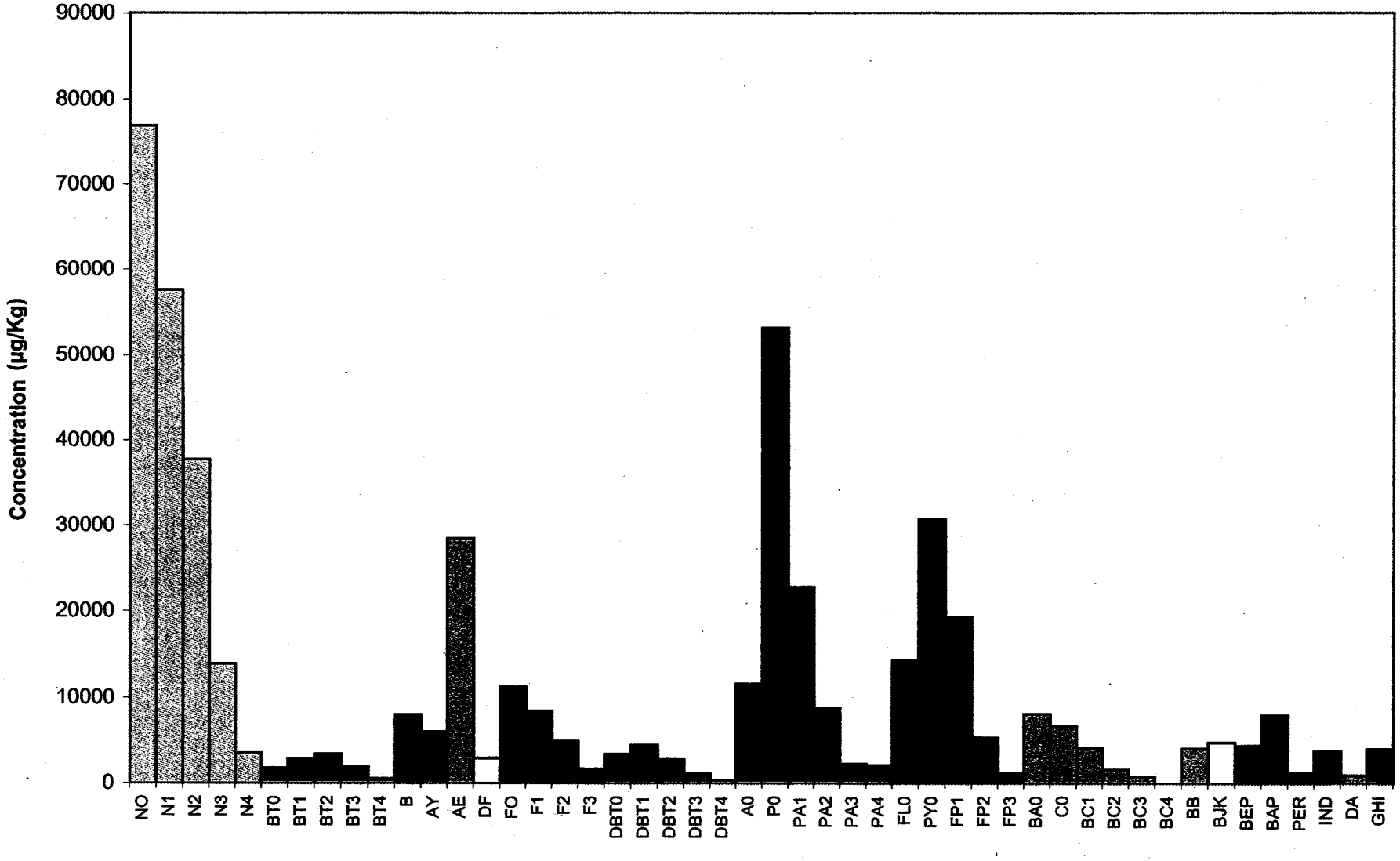
Alkylated PAH Histogram

Sample ID:
Pipe Discharge
Center Of Yard



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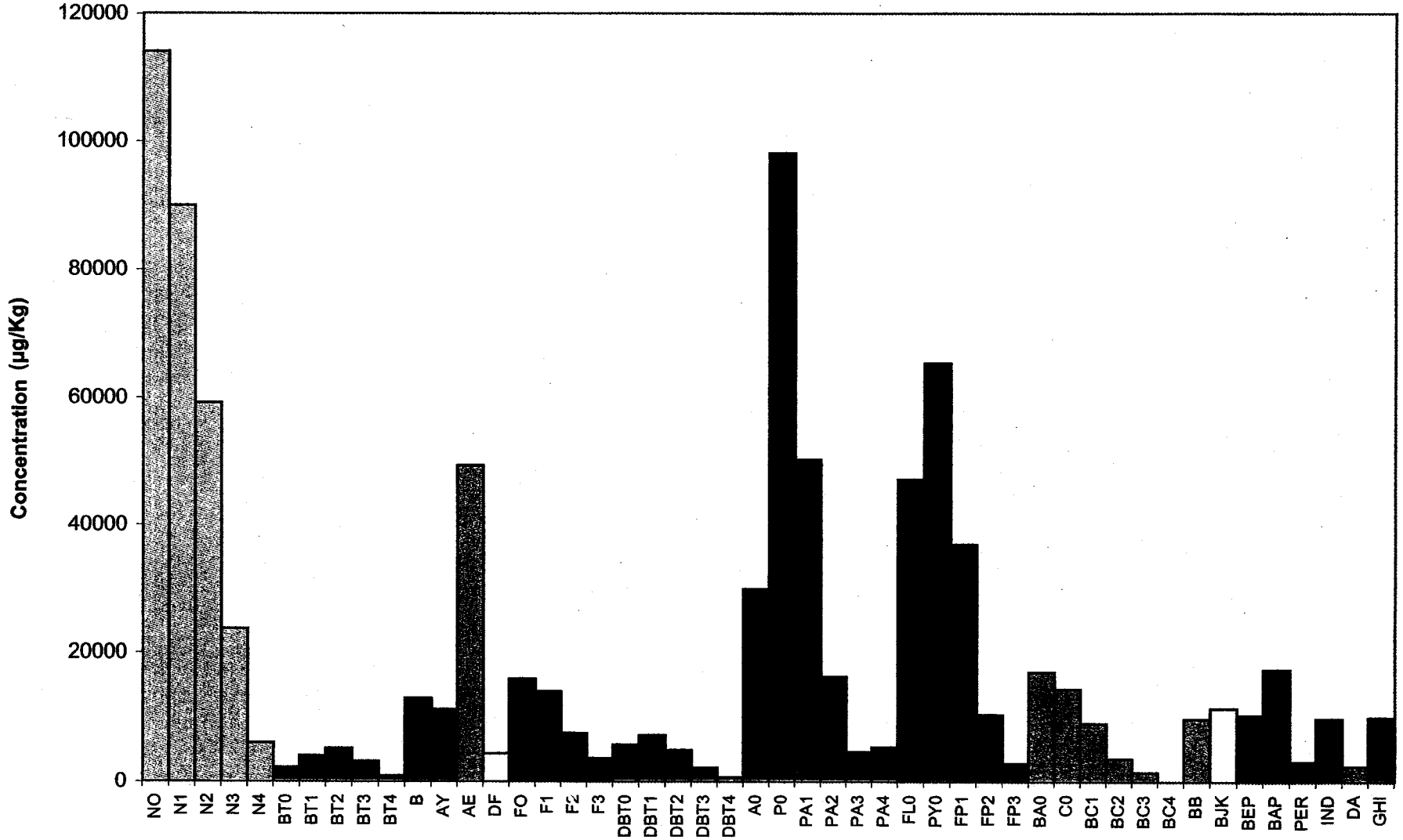
Alkylated PAH Histogram

Sample ID:
Pipe Sludge
Center of Yard



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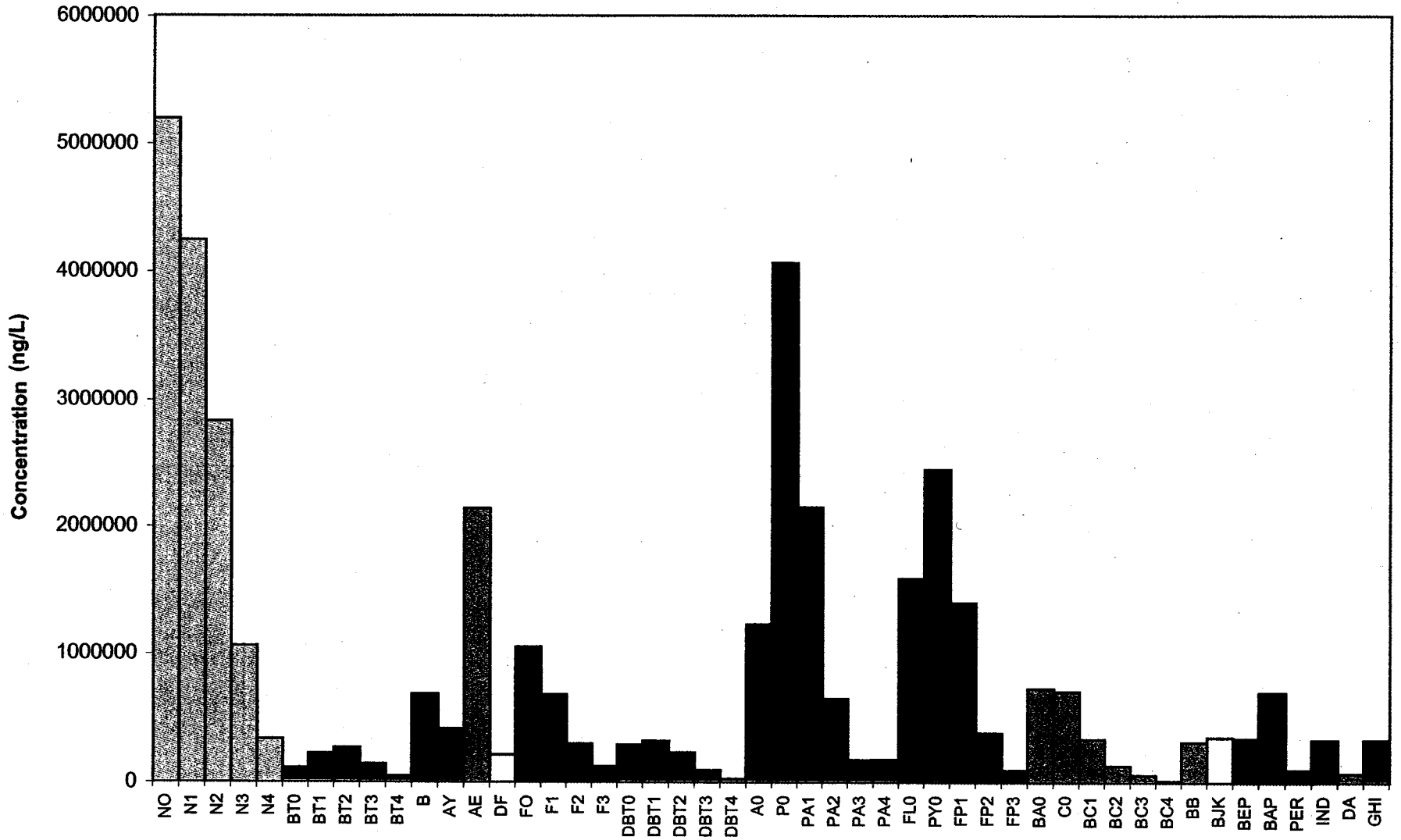
Alkylated PAH Histogram

Sample ID:
Upgradient Riser



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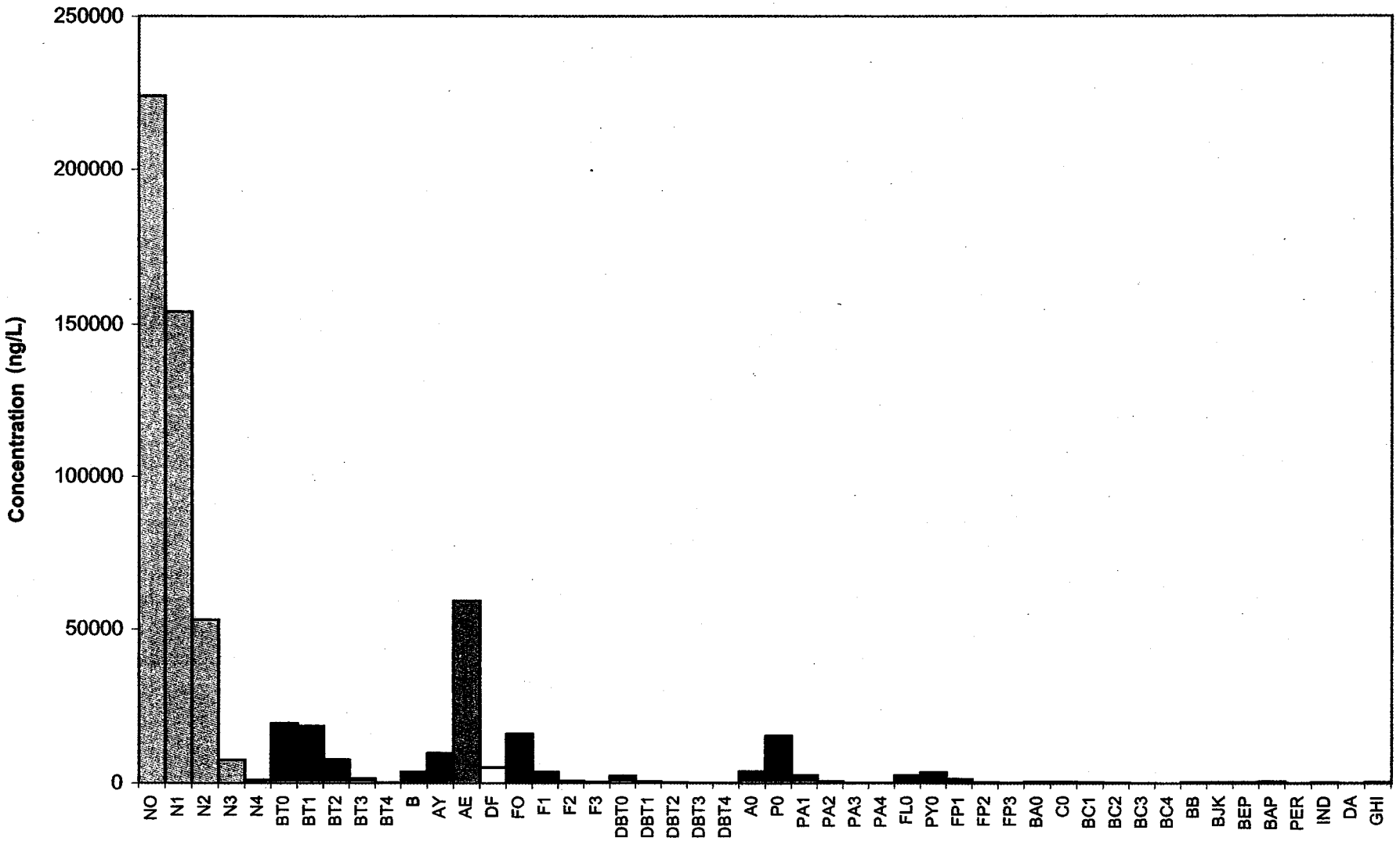
Alkylated PAH Histogram

Sample ID:
East Riser



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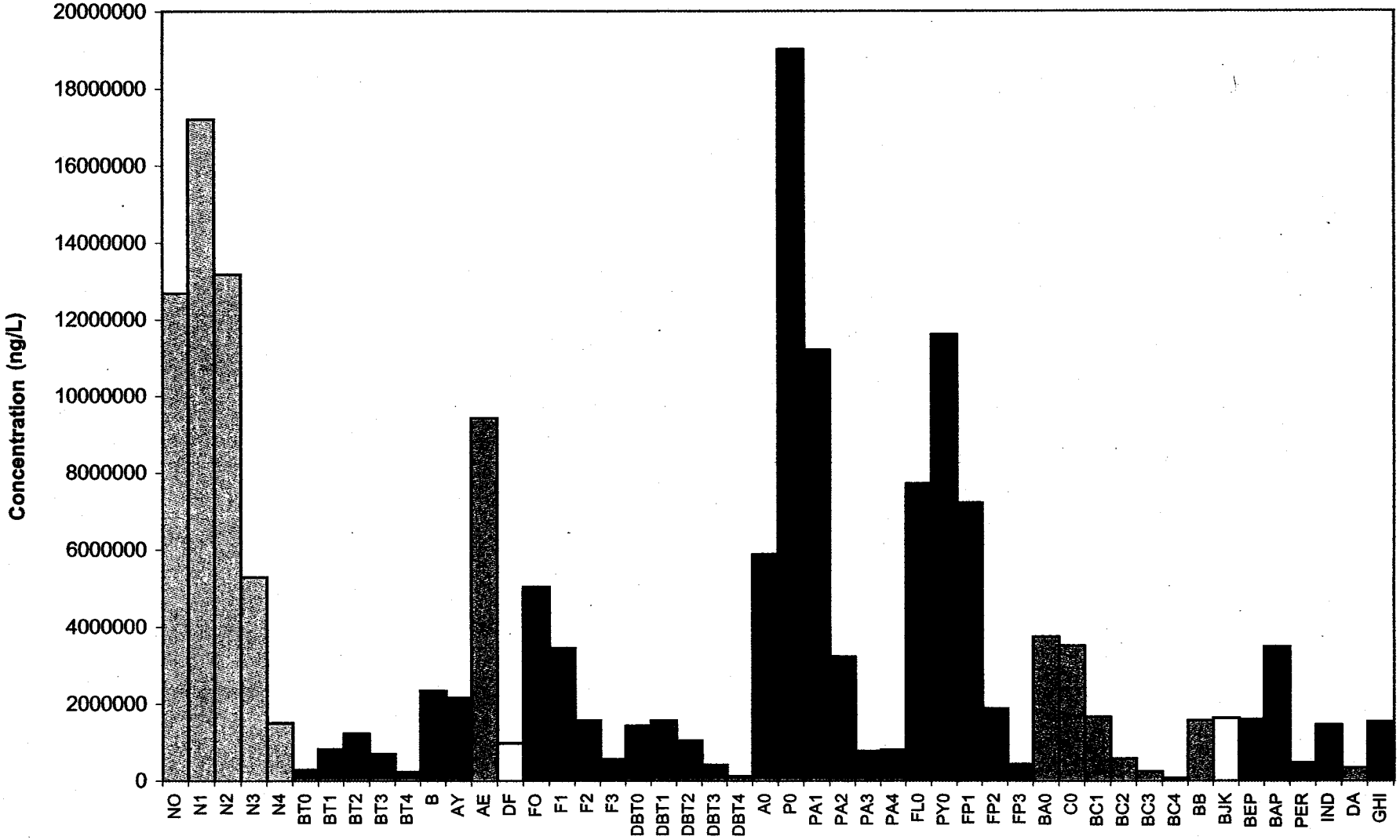
Alkylated PAH Histogram

Sample ID:
West Riser



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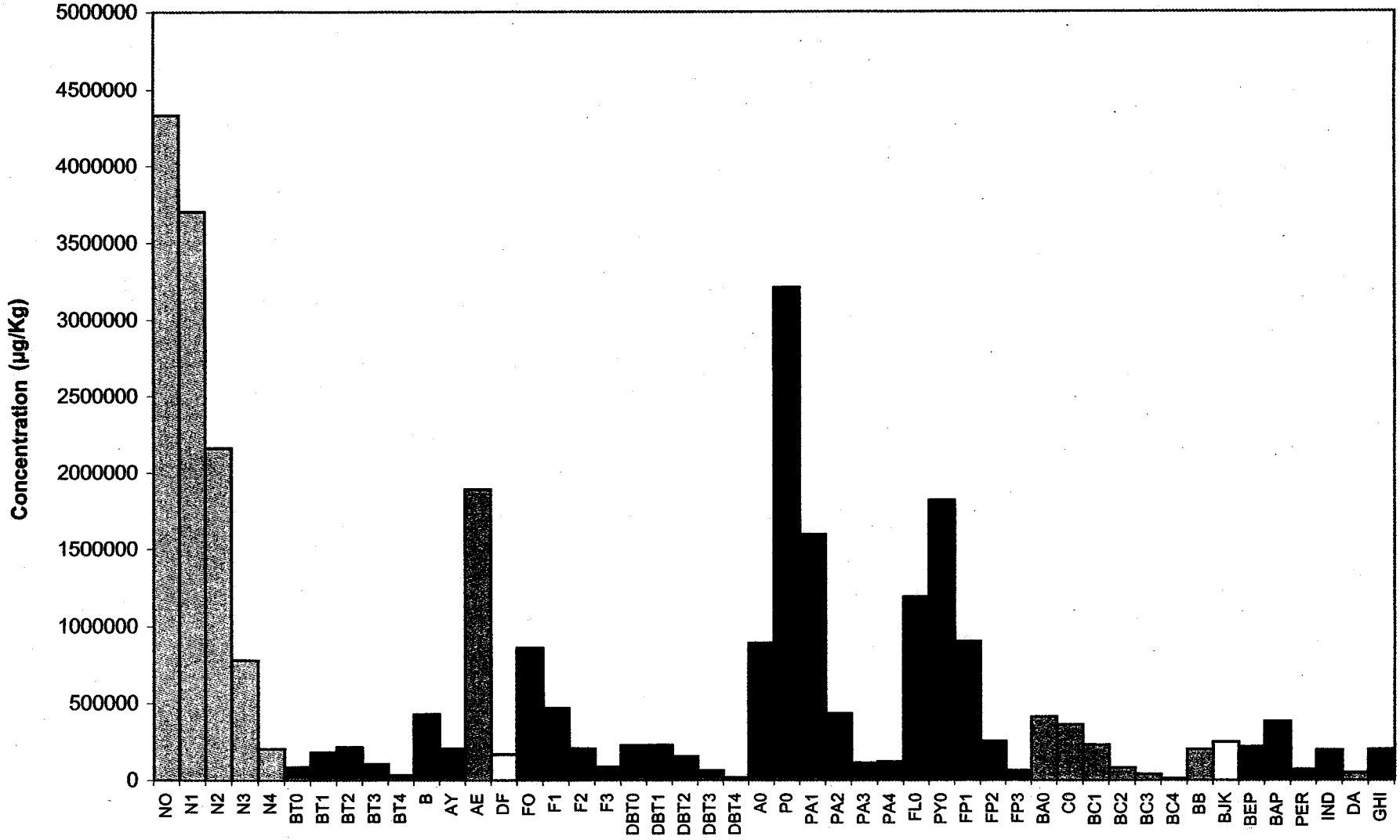
Alkylated PAH Histogram

Sample ID:
Scrapings From Inside Discharge



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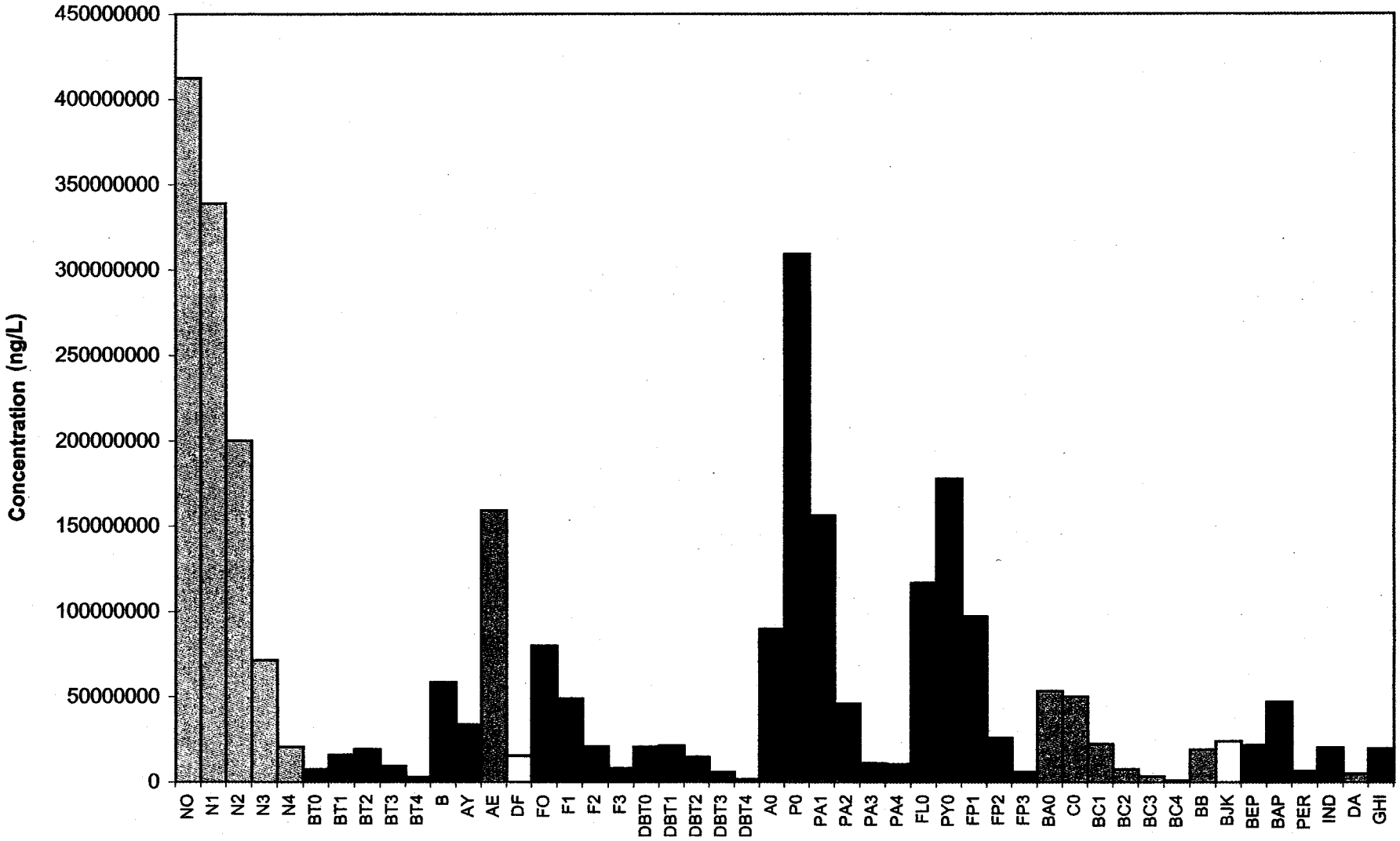
Alkylated PAH Histogram

Sample ID:
Pipe Discharge



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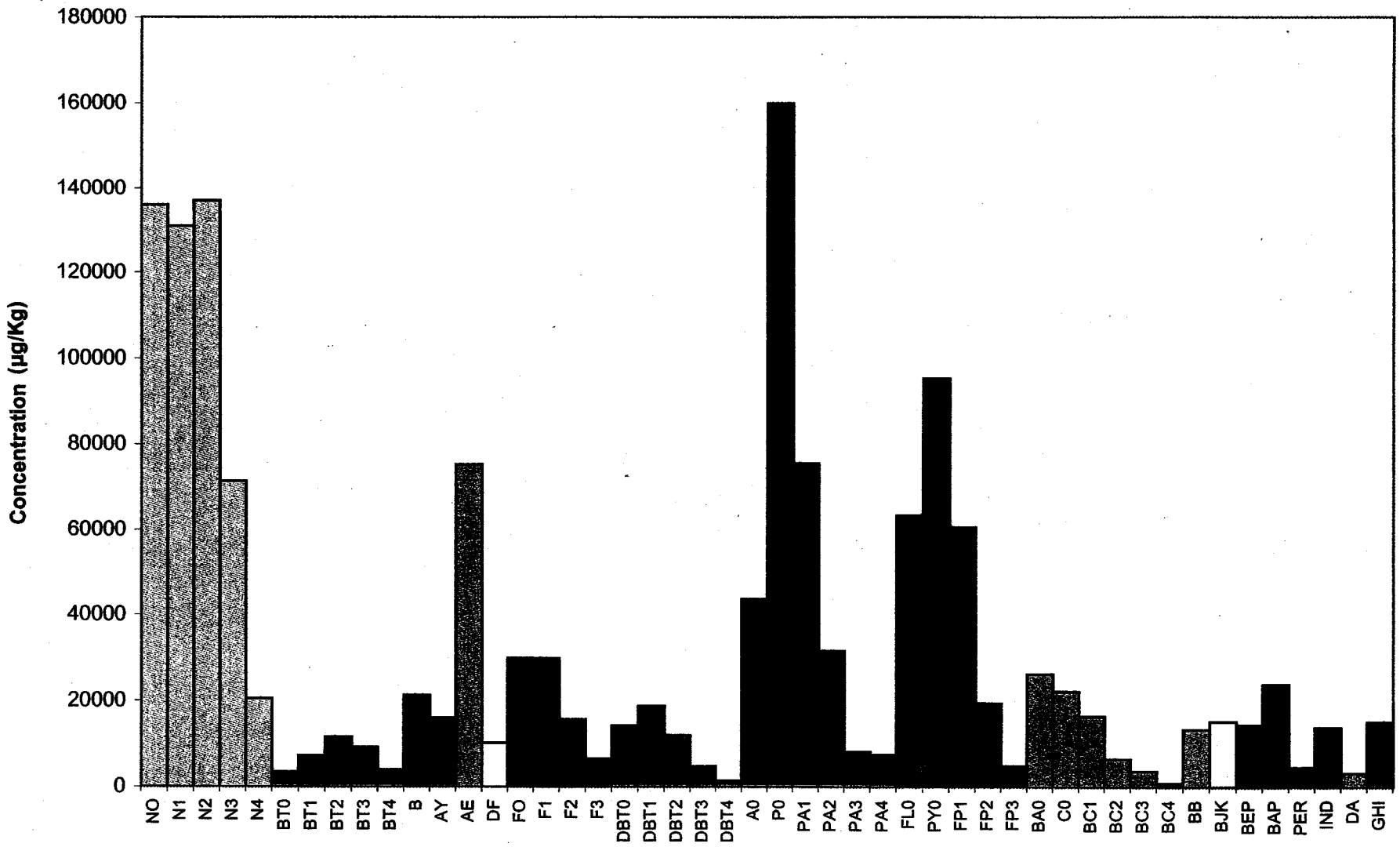
Alkylated PAH Histogram

Sample ID:
Solids Around Discharge Pipe



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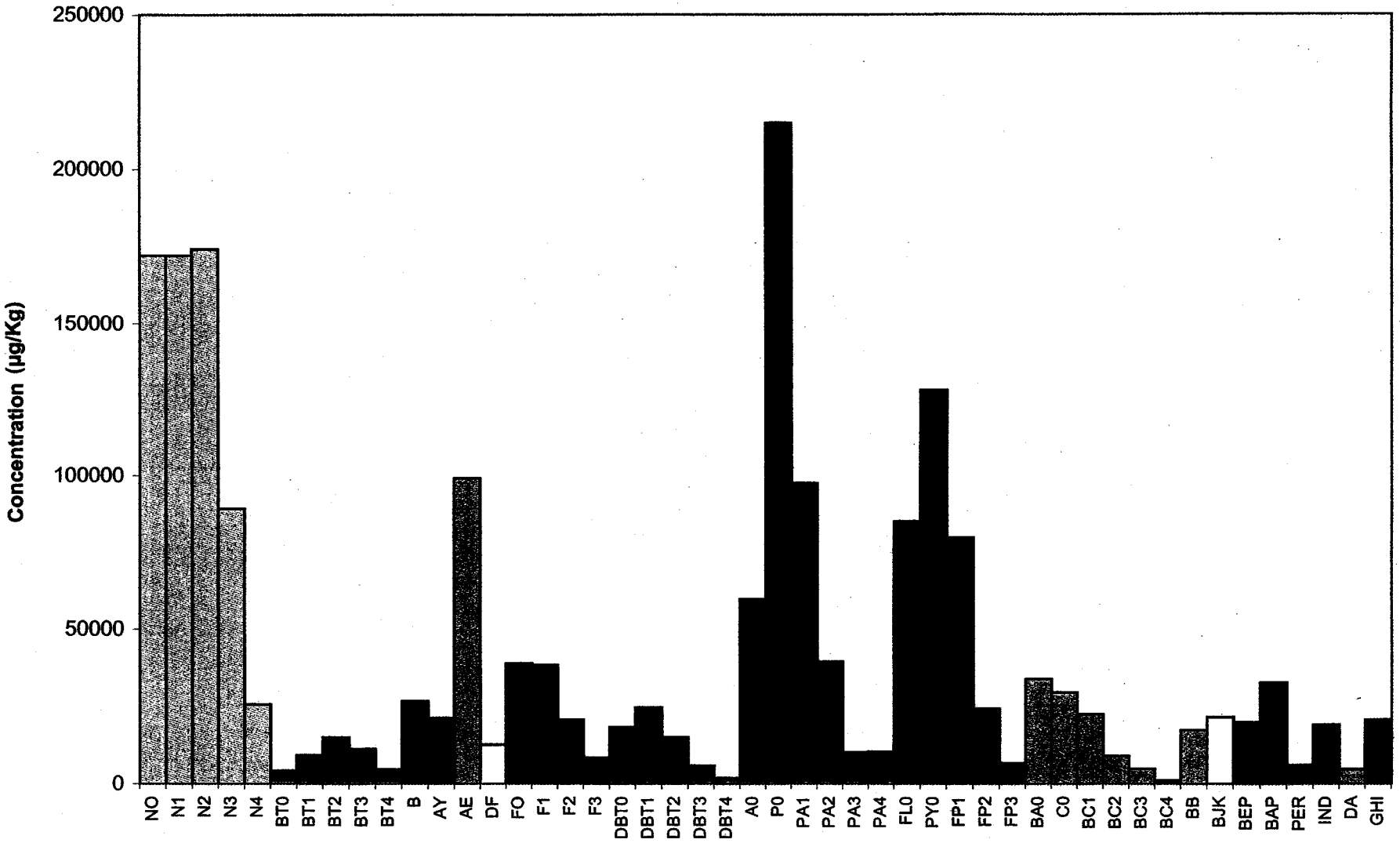
Alkylated PAH Histogram

Sample ID:
Solids around discharge pipe



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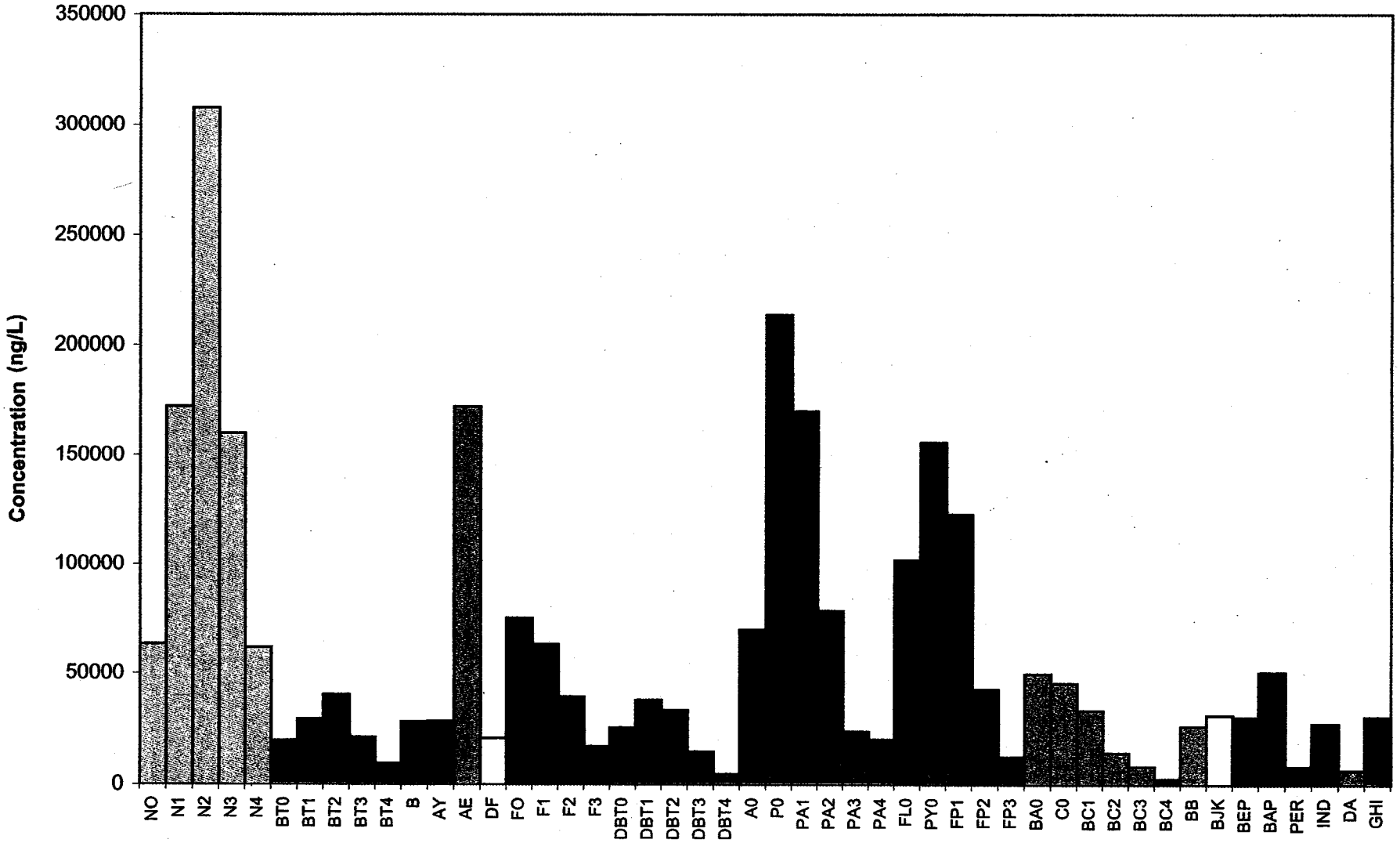
Alkylated PAH Histogram

Sample ID:
MW-7



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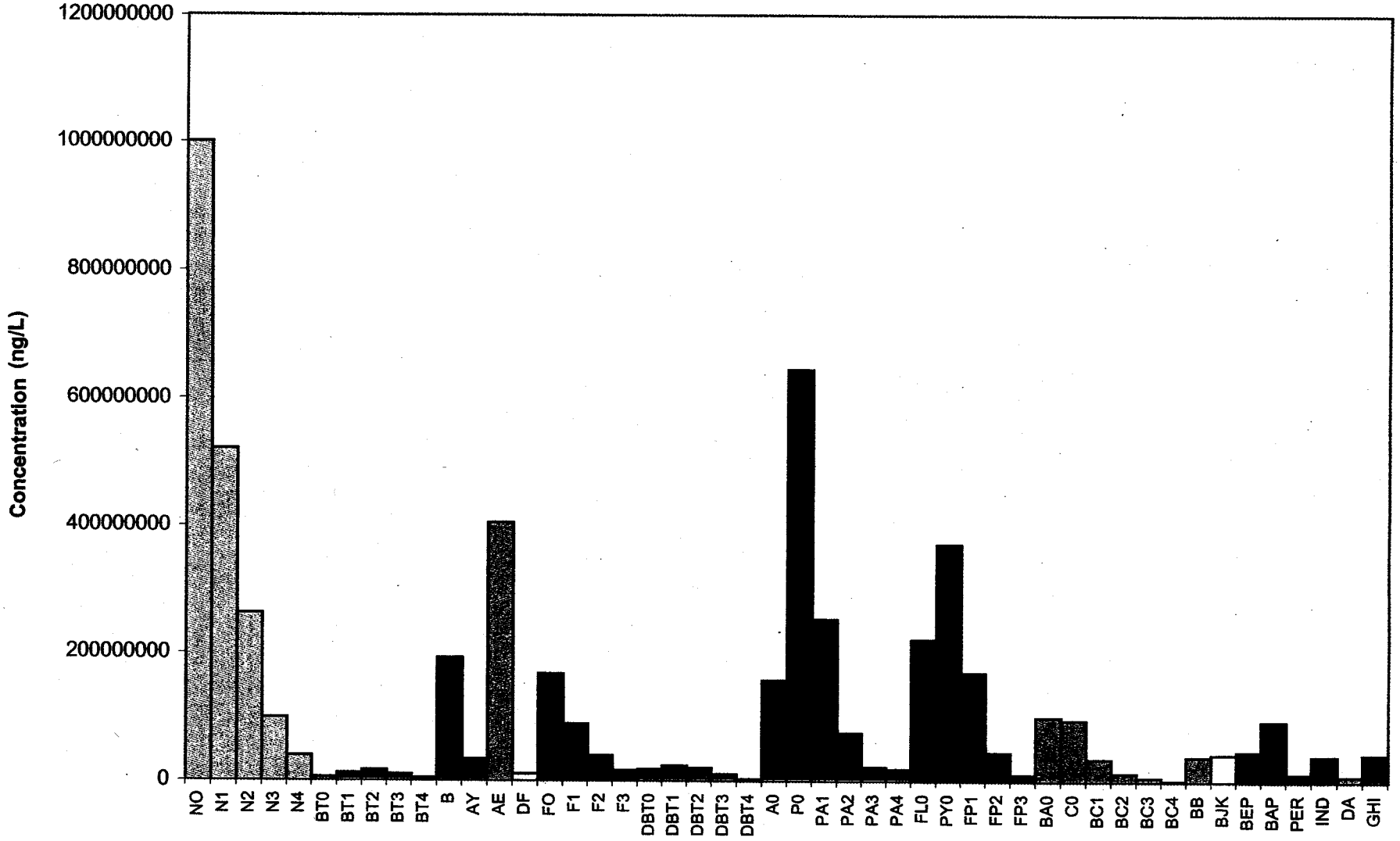
Alkylated PAH Histogram

Sample ID:
TW-9

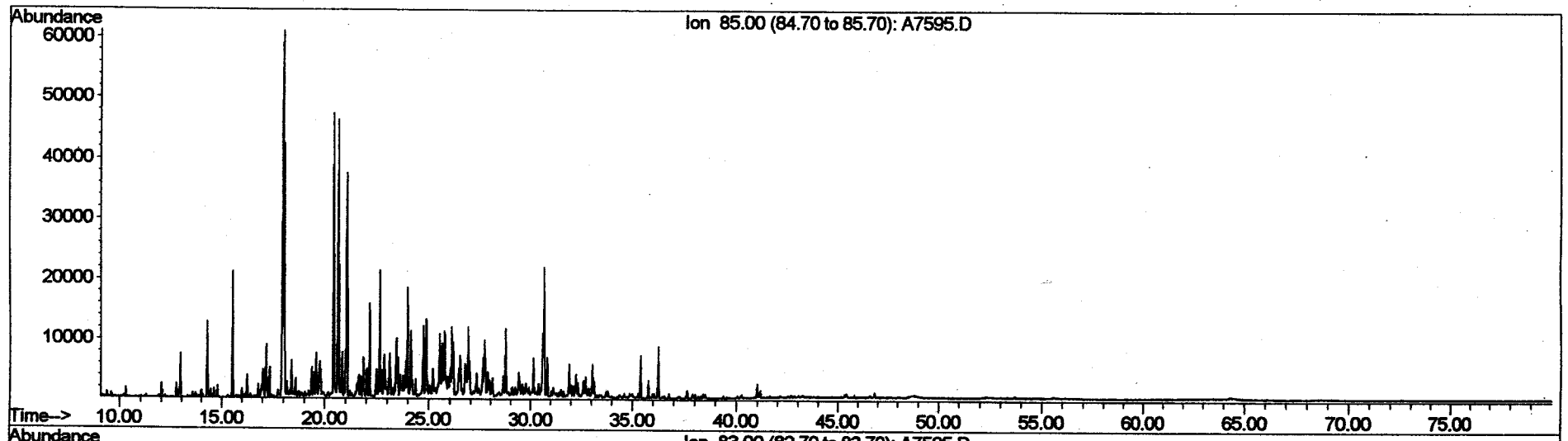


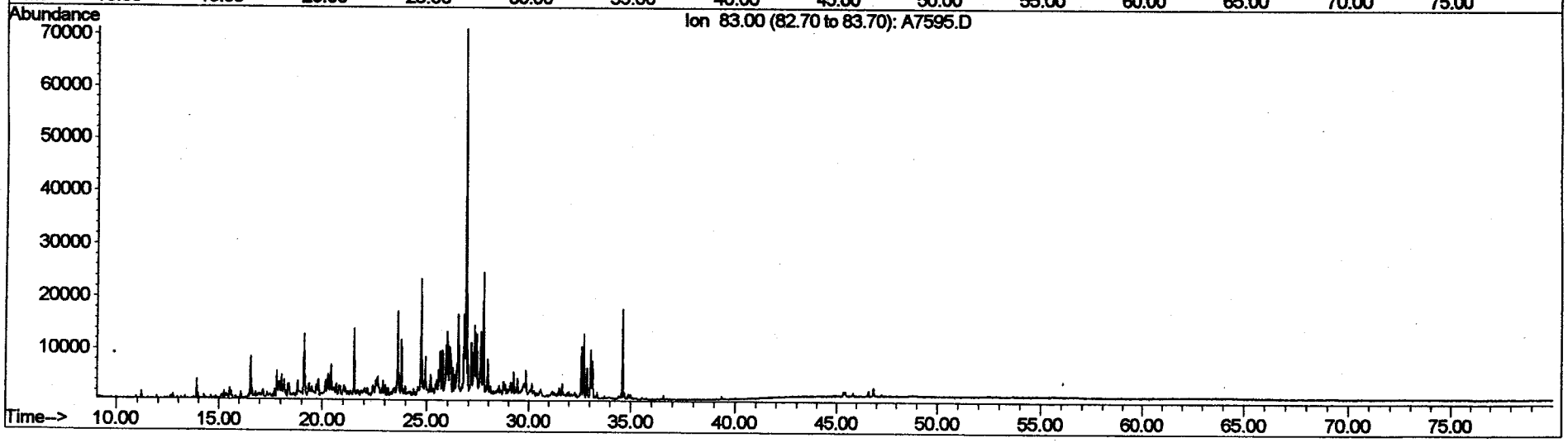
Attachment 7

Biomarker Fingerprints:

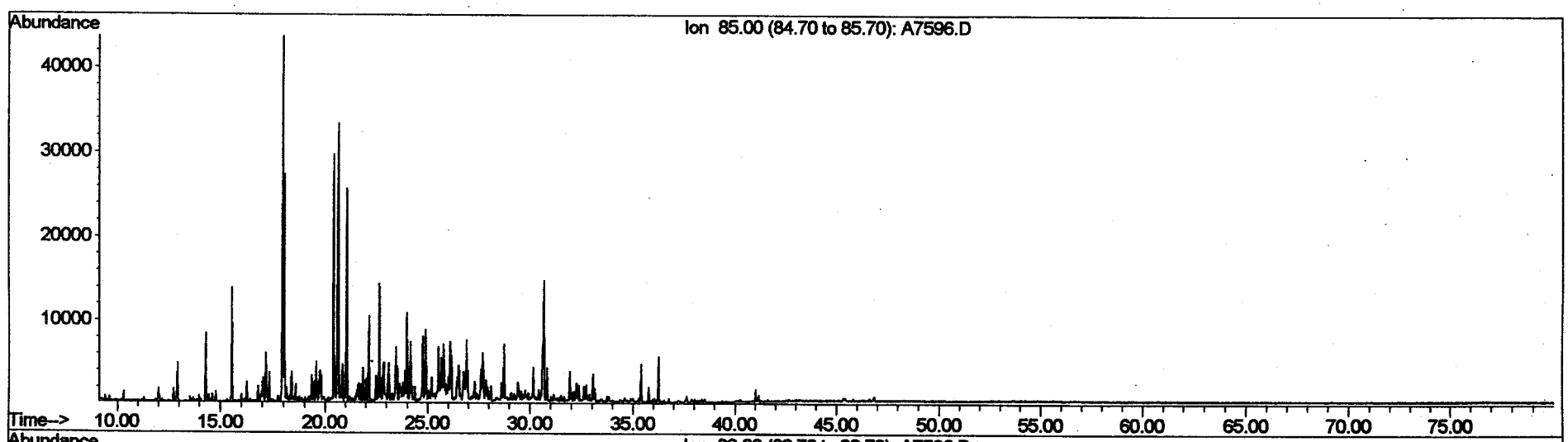
Normal Alkanes and Alkylcyclohexanes

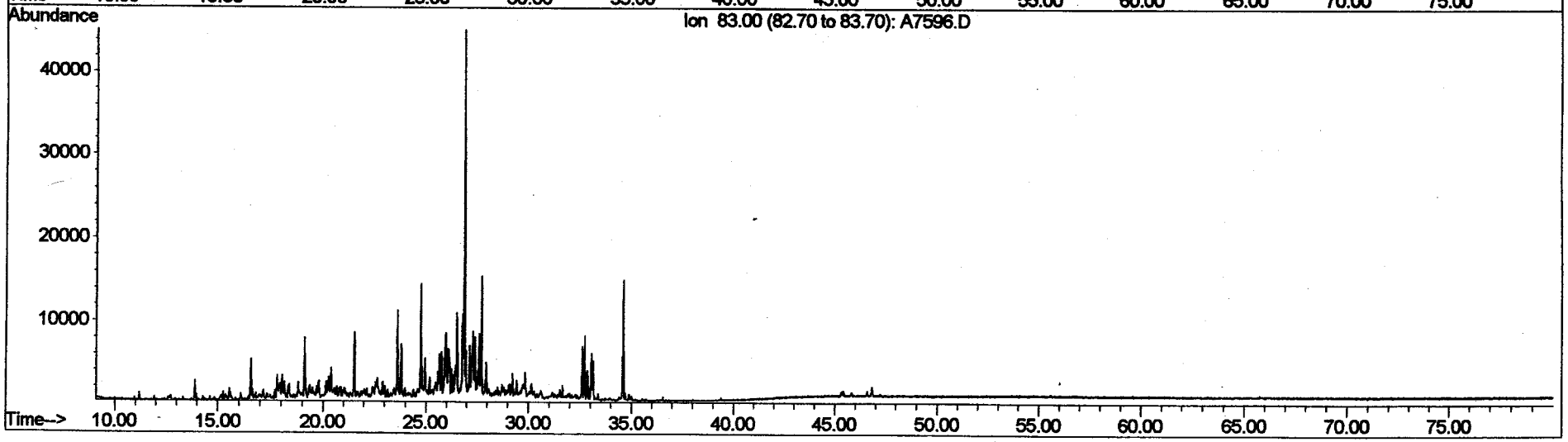
File : U:\A\DATA\SQA214\A7595.D
Operator : AC
Acquired : 17 Jan 2002 11:56 am using AcqMethod ACQASH
Instrument : GC/MS Ins
Sample Name: W9071
Misc Info : 2 Inch Steel Pipe
Vial Number: 2



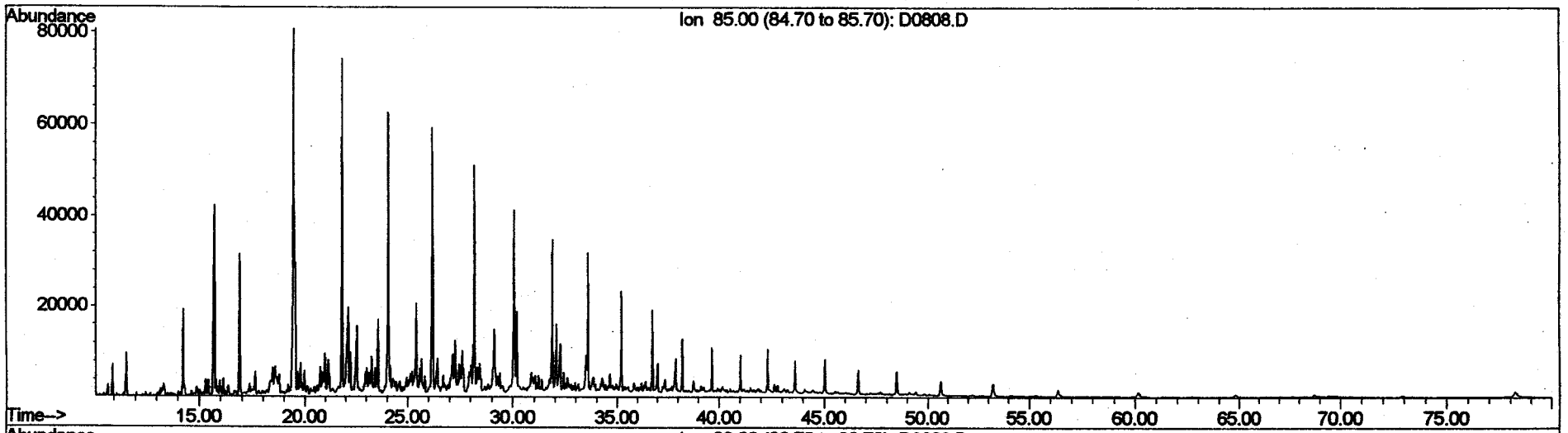


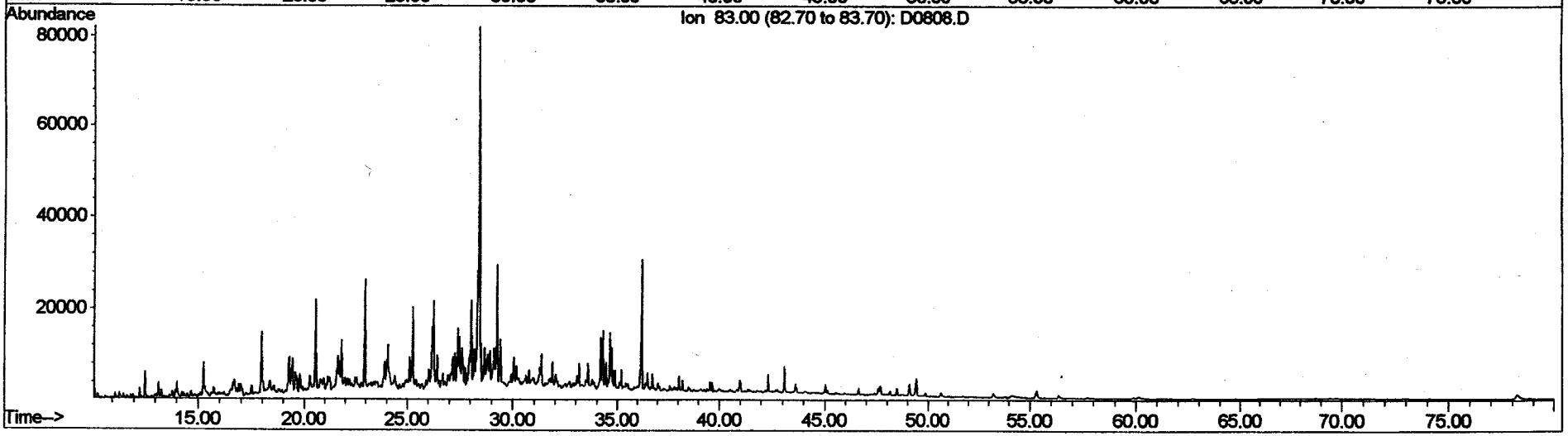
File : U:\A\DATA\SQA214\A7596.D
Operator : AC
Acquired : 17 Jan 2002 1:45 pm using AcqMethod ACQASH
Instrument : GC/MS Ins
Sample Name: W9072
Misc Info : 12 Inch Steel Pipe
Vial Number: 3



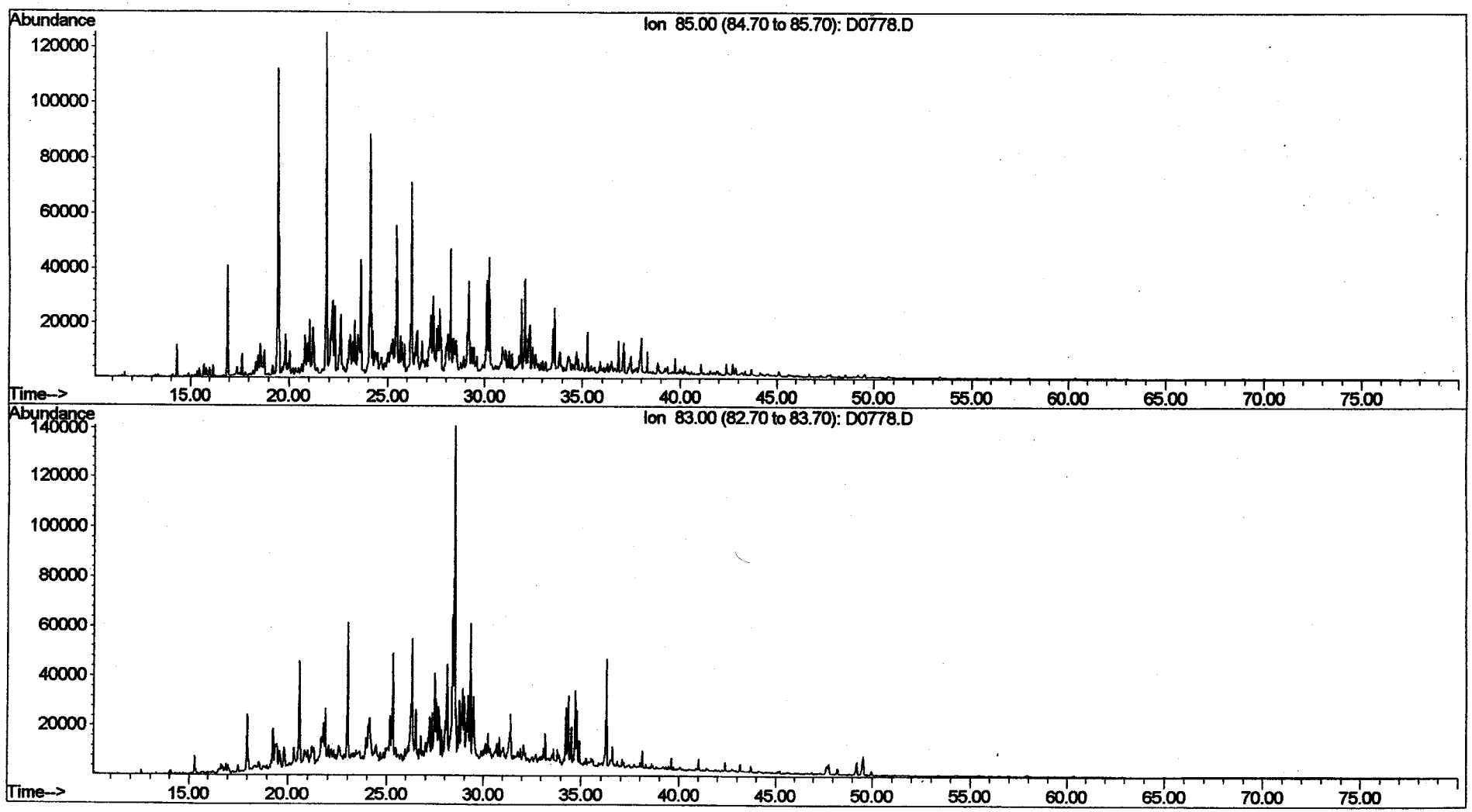


File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD295\D0808
Operator : SA
Acquired : 25 Oct 2001 12:39 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5548-1
Misc Info : TW-13
Vial Number: 19

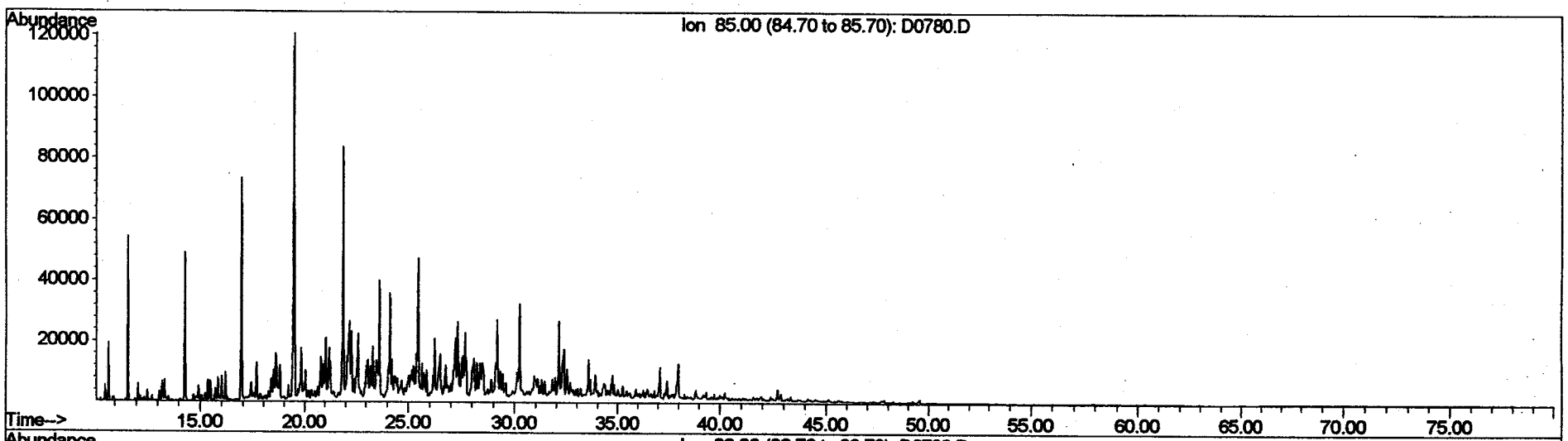


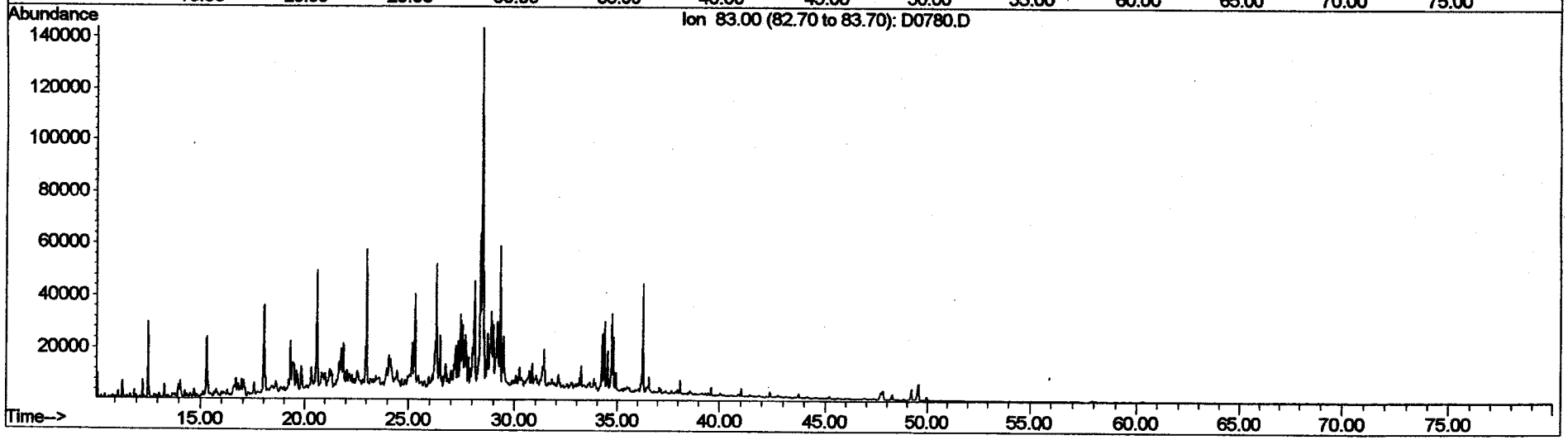


File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD294\D0778
Operator : SA
Acquired : 22 Oct 2001 9:21 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W6841
Misc Info : Pipe Discharge East Yard Gate
Vial Number: 4

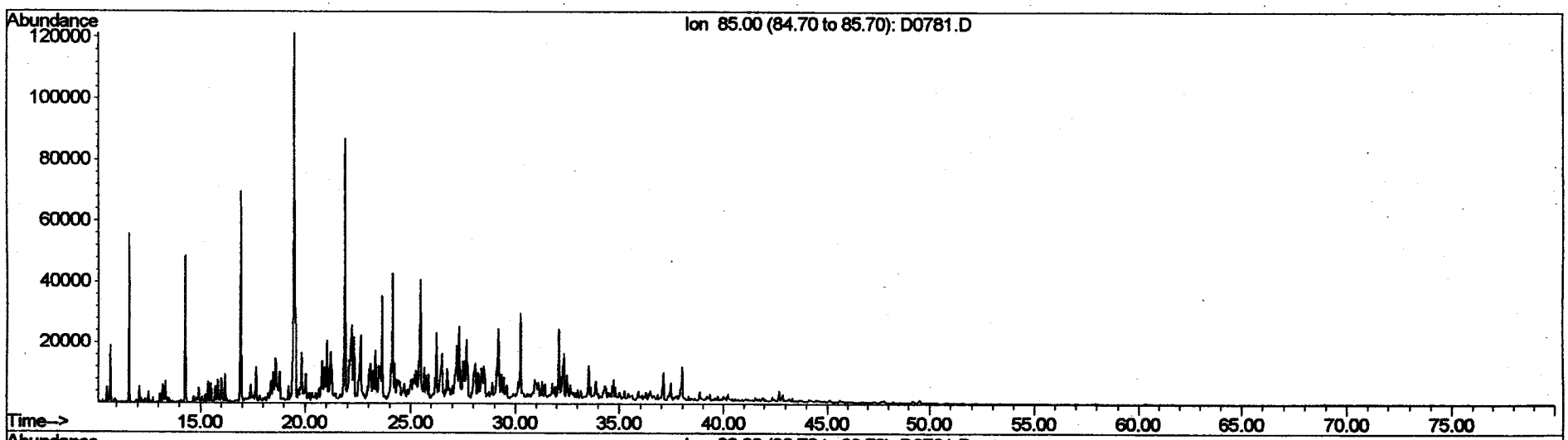


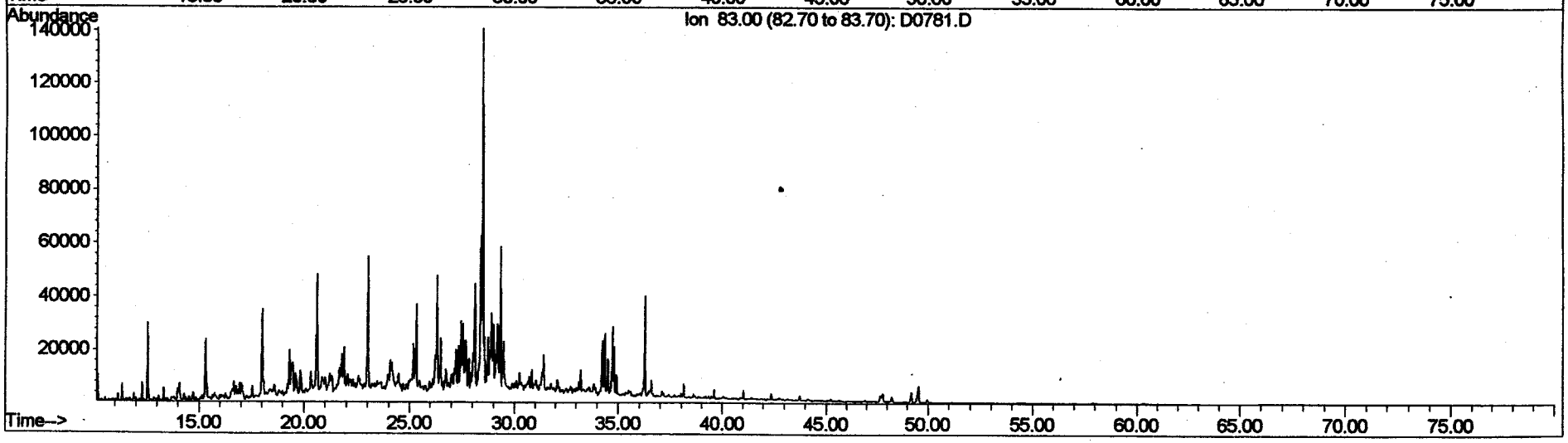
File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD294\D0780
Operator : SA
Acquired : 23 Oct 2001 12:33 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W6839
Misc Info : Pipe Sludge East Yard Gate #1
Vial Number: 6



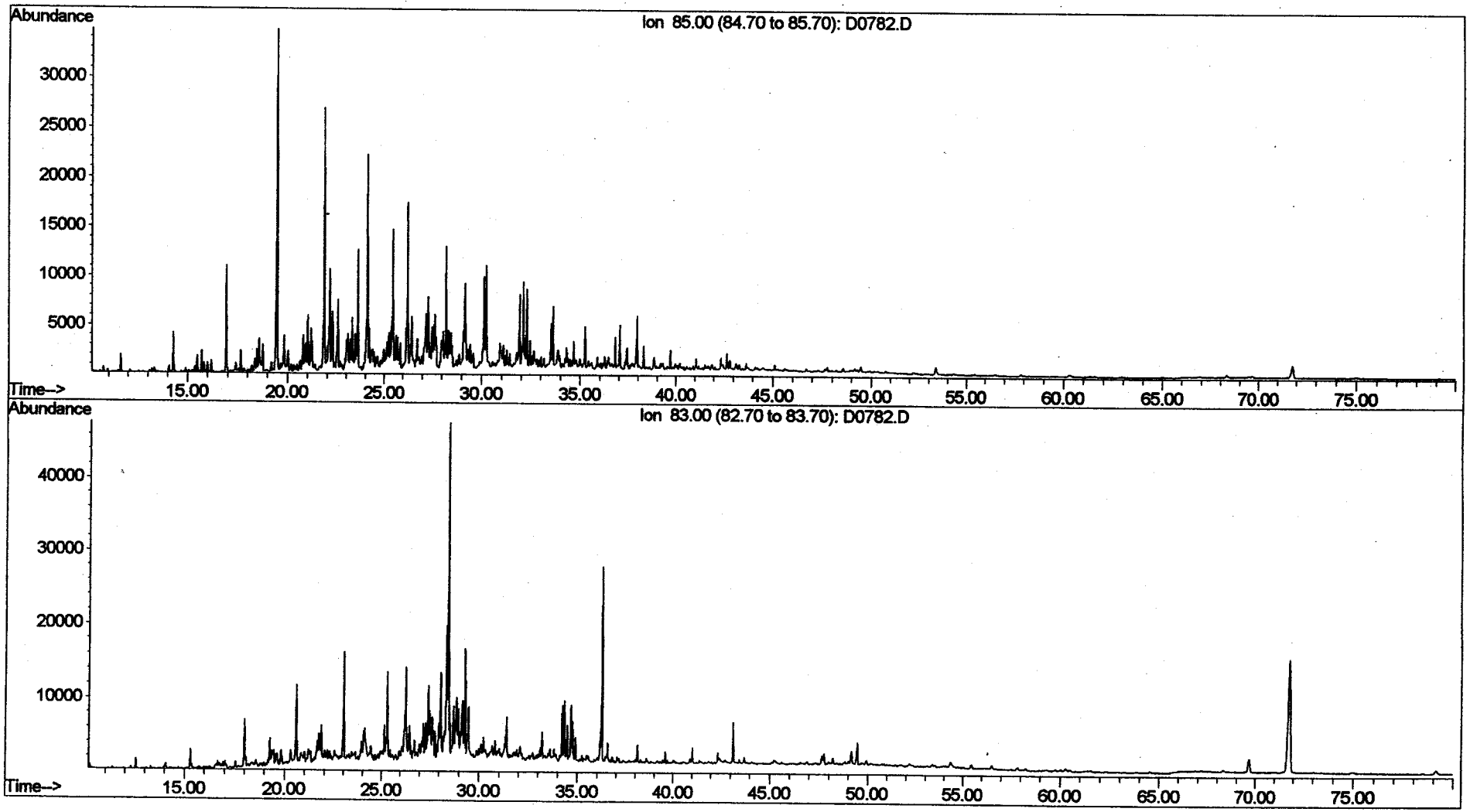


File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD294\D0781
Operator : SA
Acquired : 23 Oct 2001 2:06 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W6840
Misc Info : Pipe Sludge East Yard Gate #2
Vial Number: 7

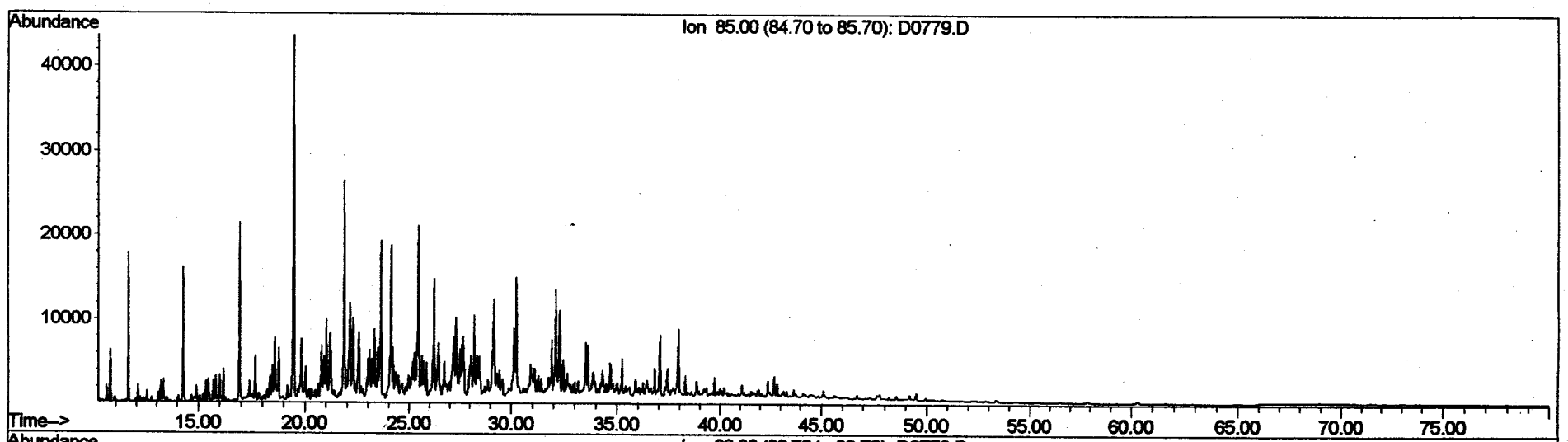


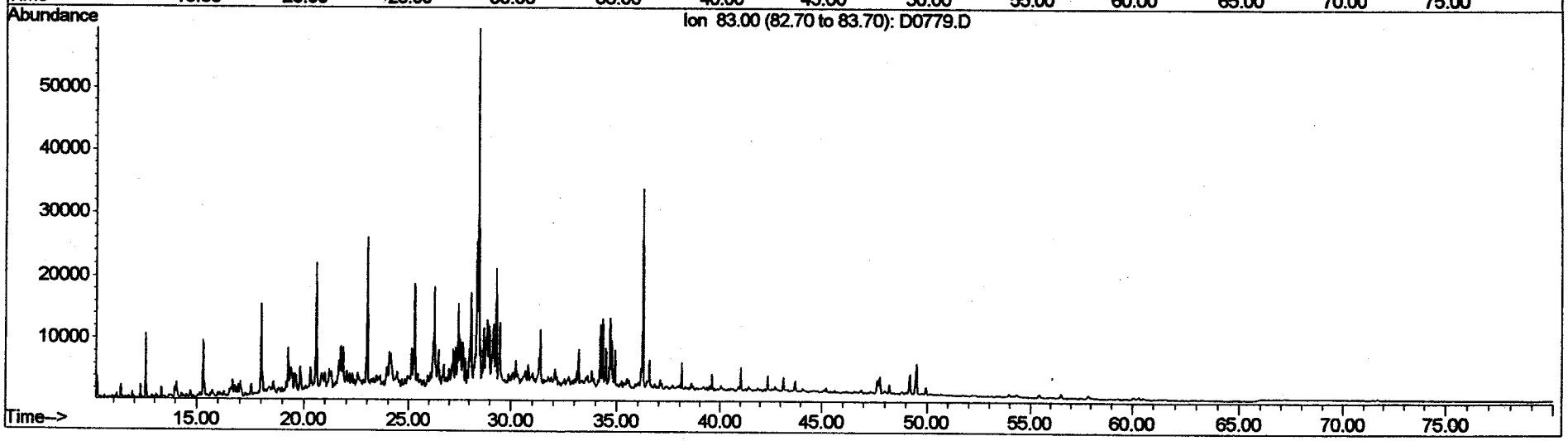


File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD294\D0782
Operator : SA
Acquired : 23 Oct 2001 3:40 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W6837
Misc Info : Pipe Discharge Center Of Yard
Vial Number: 8

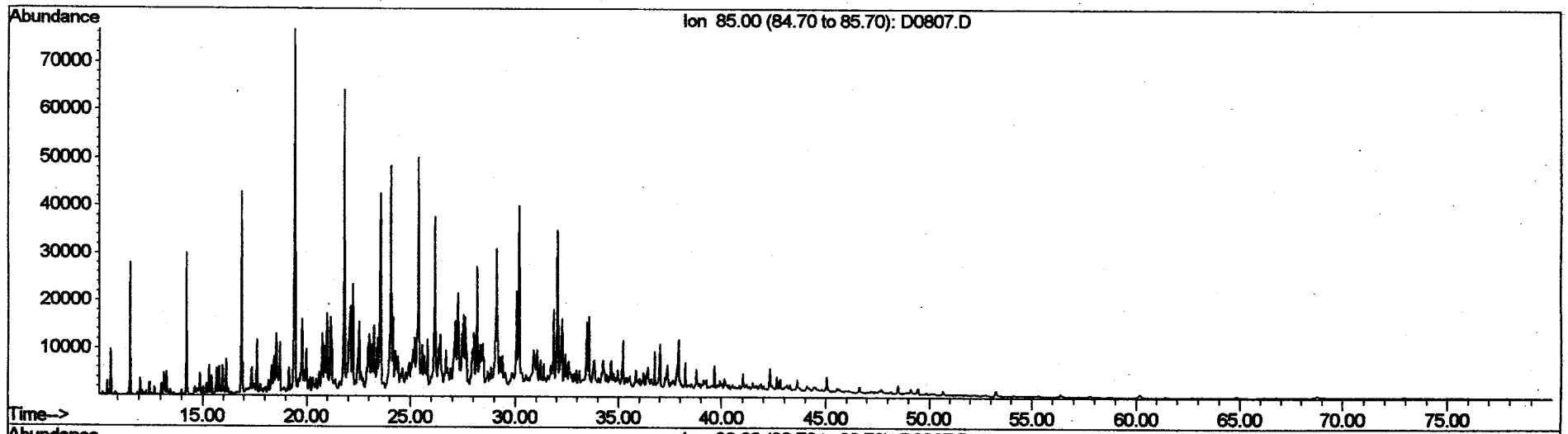


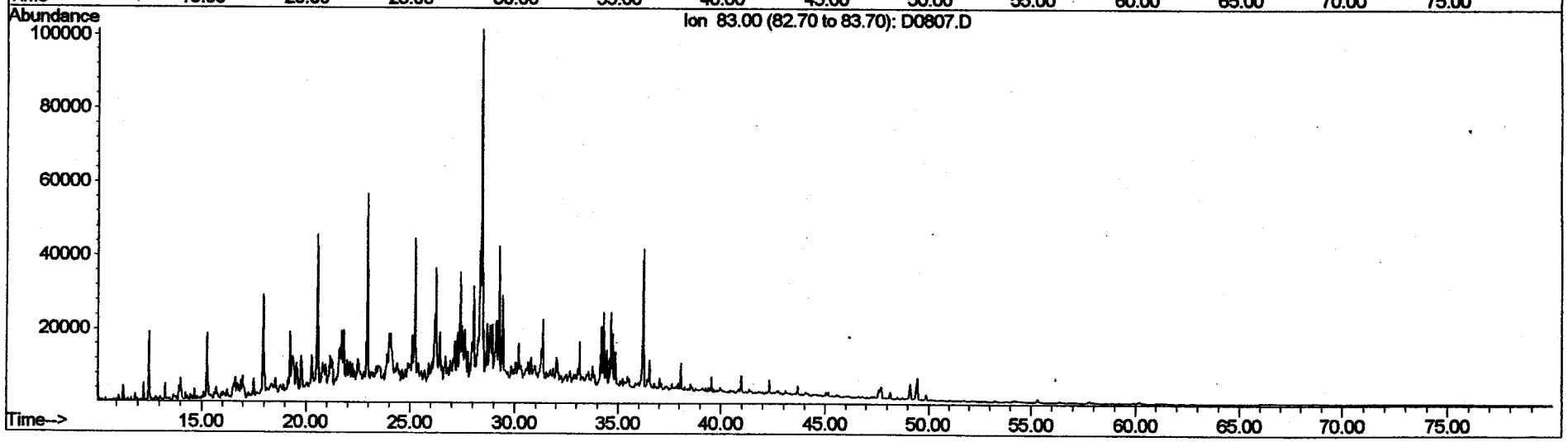
File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD294\D0779
Operator : SA
Acquired : 22 Oct 2001 10:56 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W6838
Misc Info : Pipe Sludge Center of Yard
Vial Number: 5



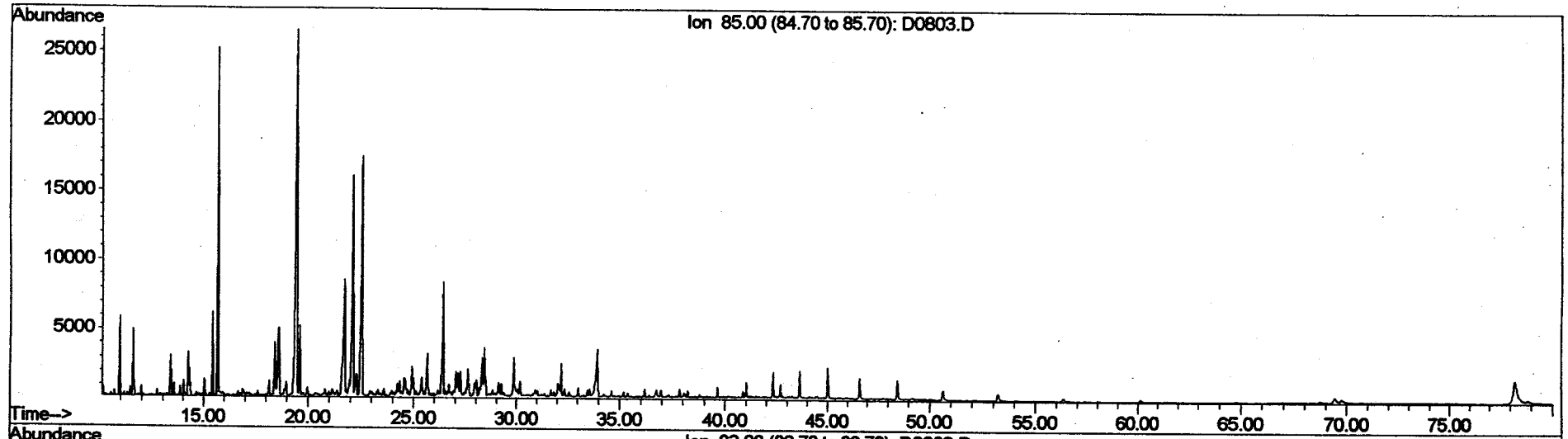


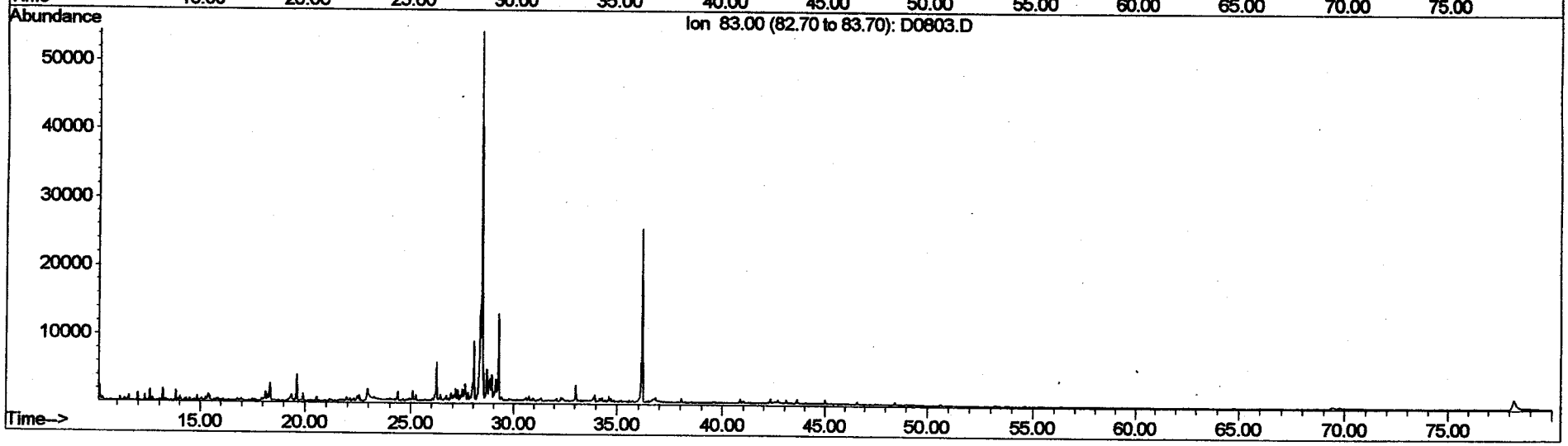
File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD295\D0807
Operator : SA
Acquired : 25 Oct 2001 11:01 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5547-1
Misc Info : Upgradient Riser
Vial Number: 18



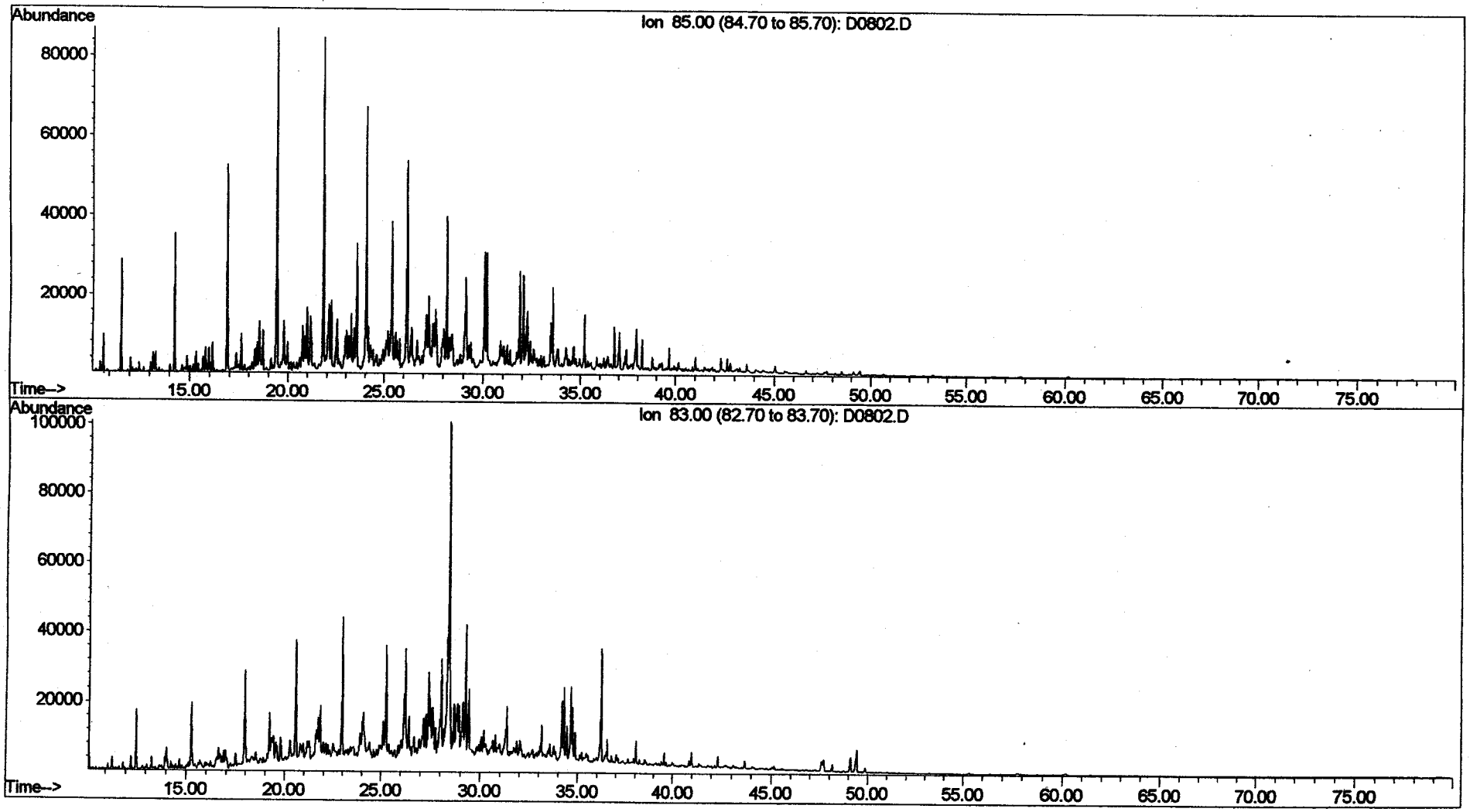


File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD295\D0803
Operator : SA
Acquired : 25 Oct 2001 4:14 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5544-1
Misc Info : East Riser
Vial Number: 14

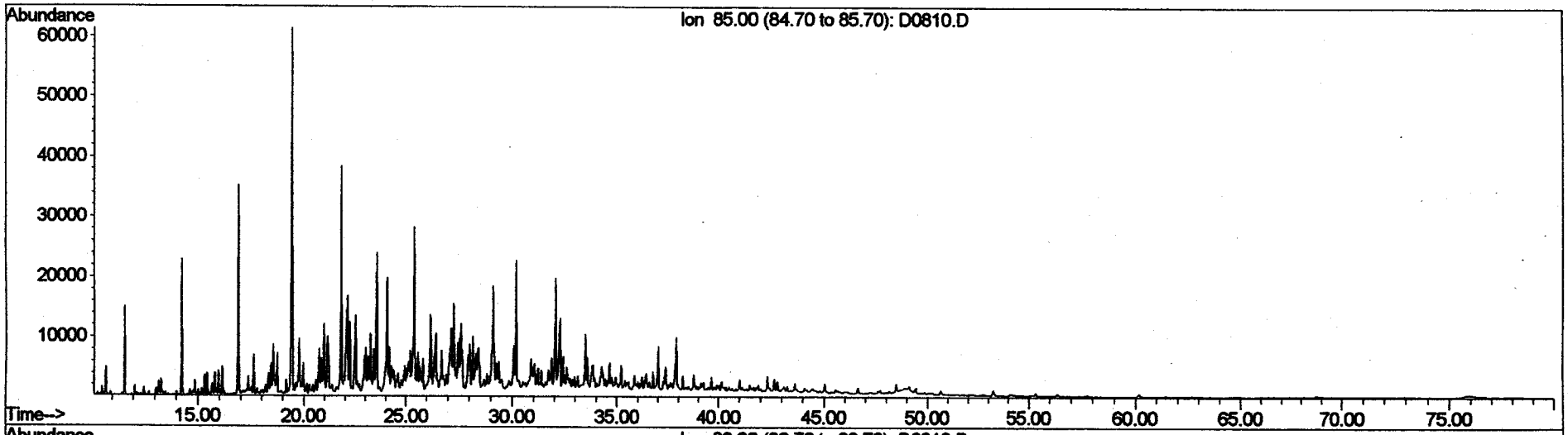


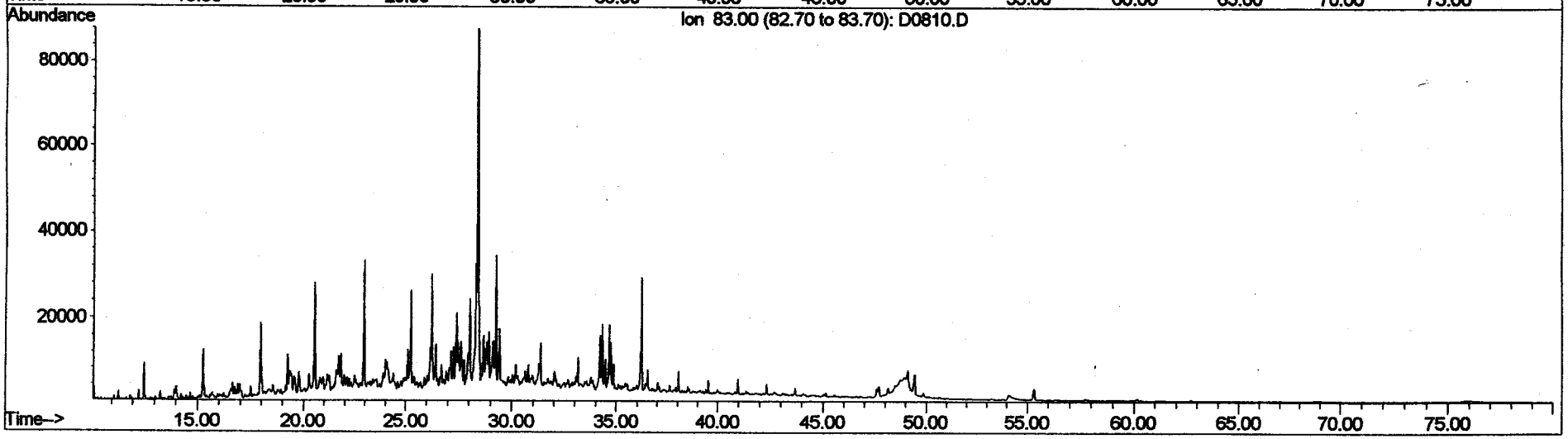


File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD295\D0802
Operator : SA
Acquired : 25 Oct 2001 2:32 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5543-1
Misc Info : West Risen
Vial Number: 13

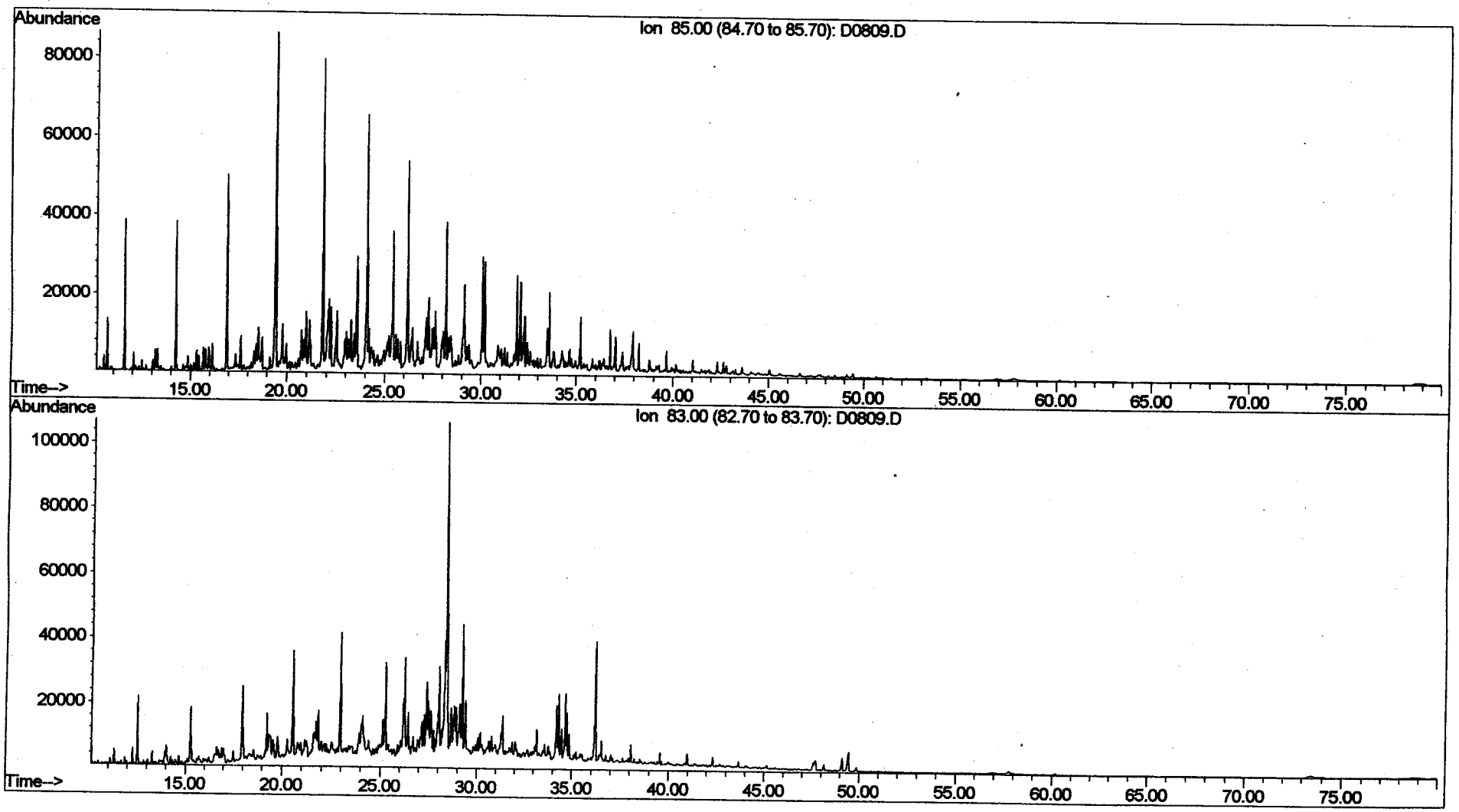


File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD295\D0810
Operator : SA
Acquired : 25 Oct 2001 4:01 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5551-1
Misc Info : Scrapings from inside discharge
Vial Number: 21

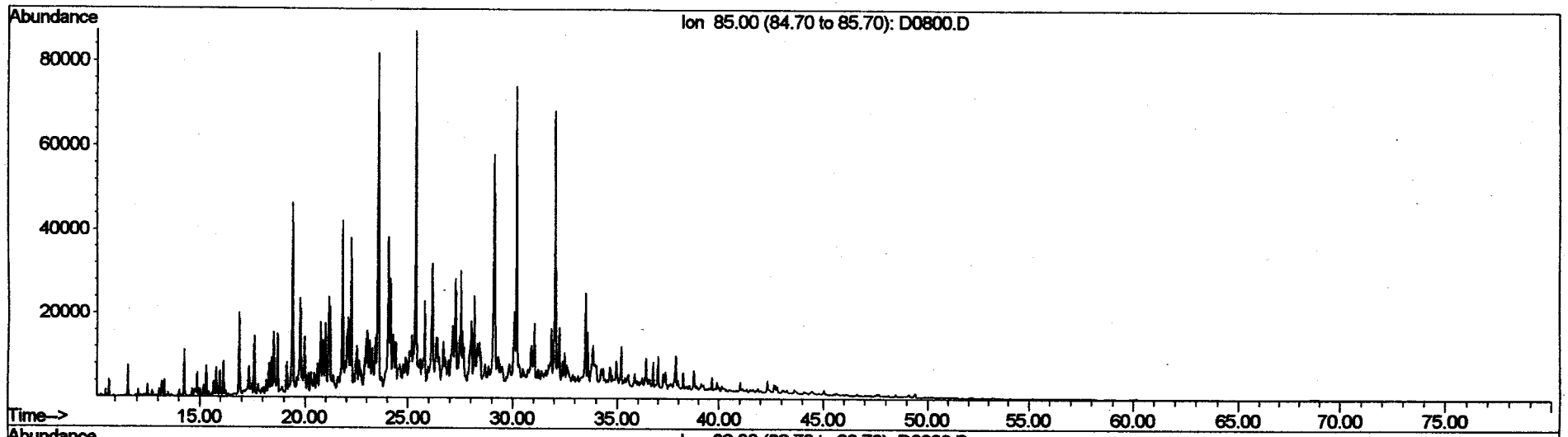


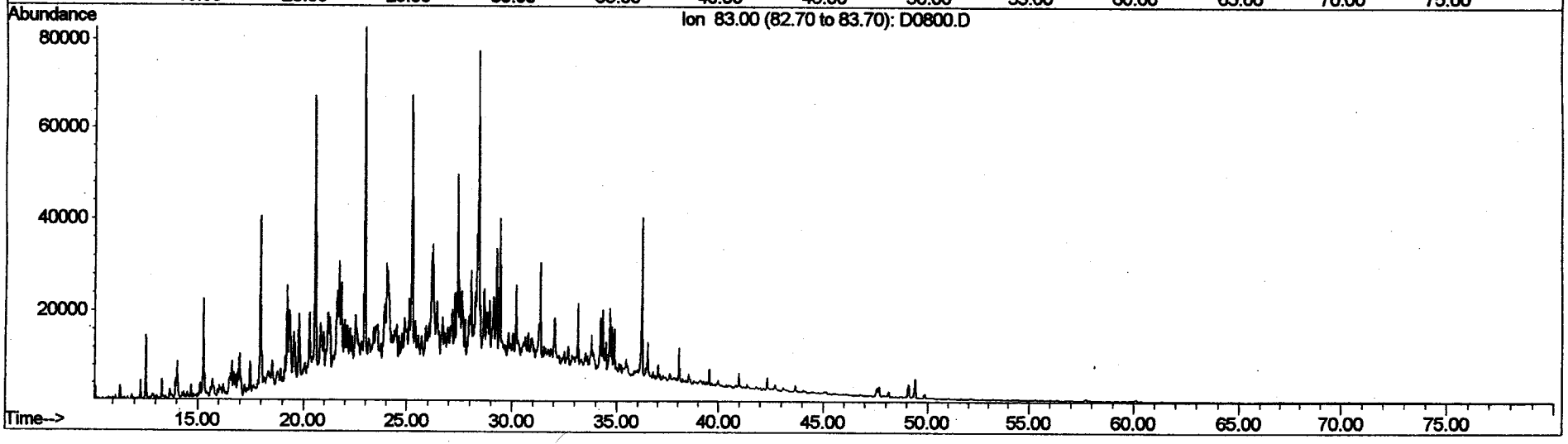


File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD295\D0809
Operator : SA
Acquired : 25 Oct 2001 2:18 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5549-1
Misc Info : Pipe Discharge
Vial Number: 20

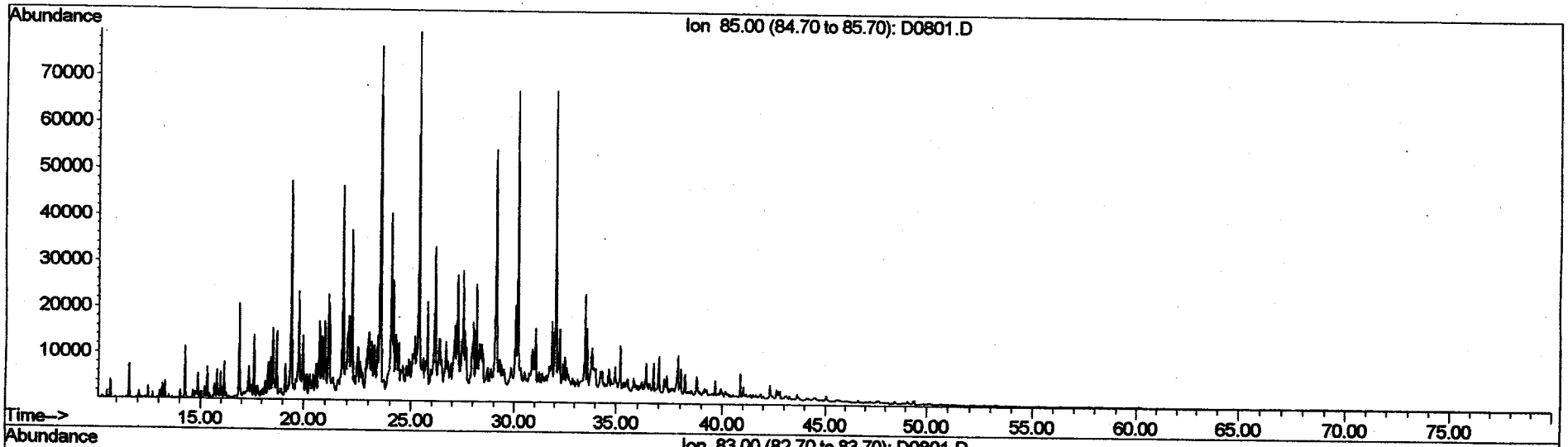


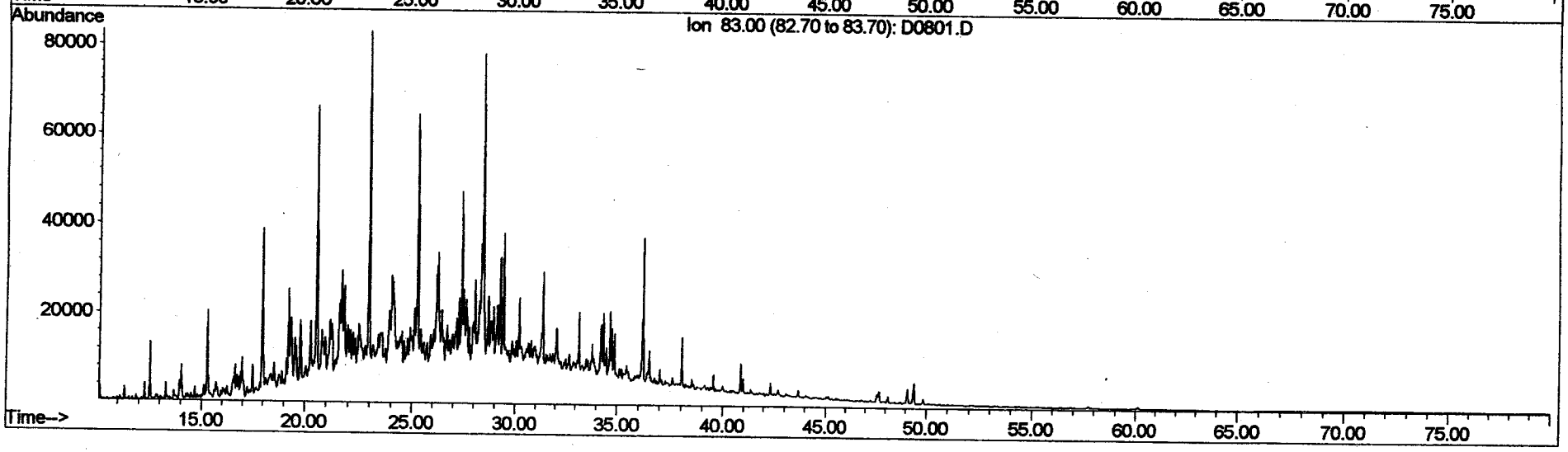
File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD295\D0800
Operator : SA
Acquired : 24 Oct 2001 11:23 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5550-1
Misc Info : Solids around discharge pipe
Vial Number: 11



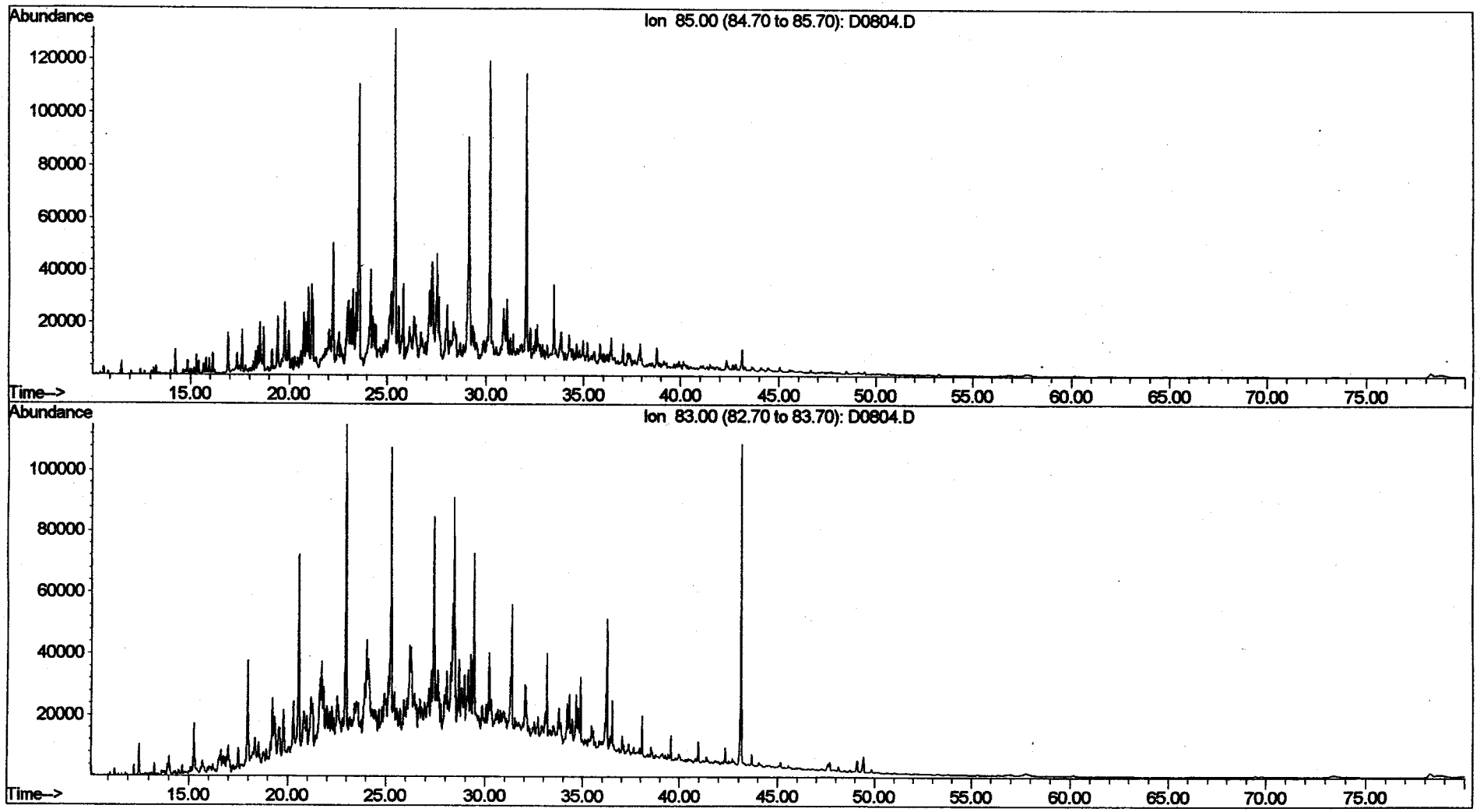


File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD295\D0801
Operator : SA
Acquired : 25 Oct 2001 12:57 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5550DUP-1
Misc Info : Solids around discharge pipe
Vial Number: 12

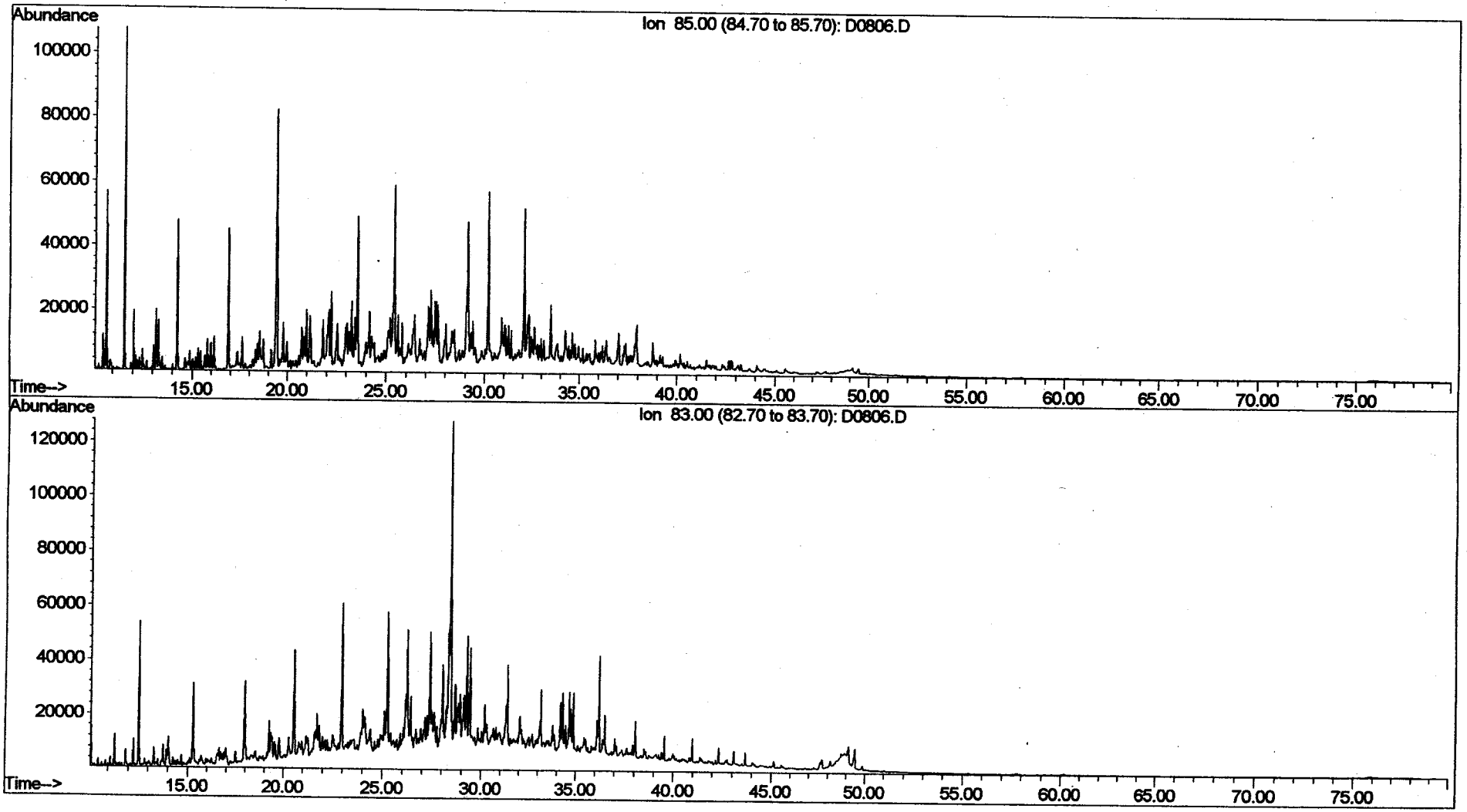




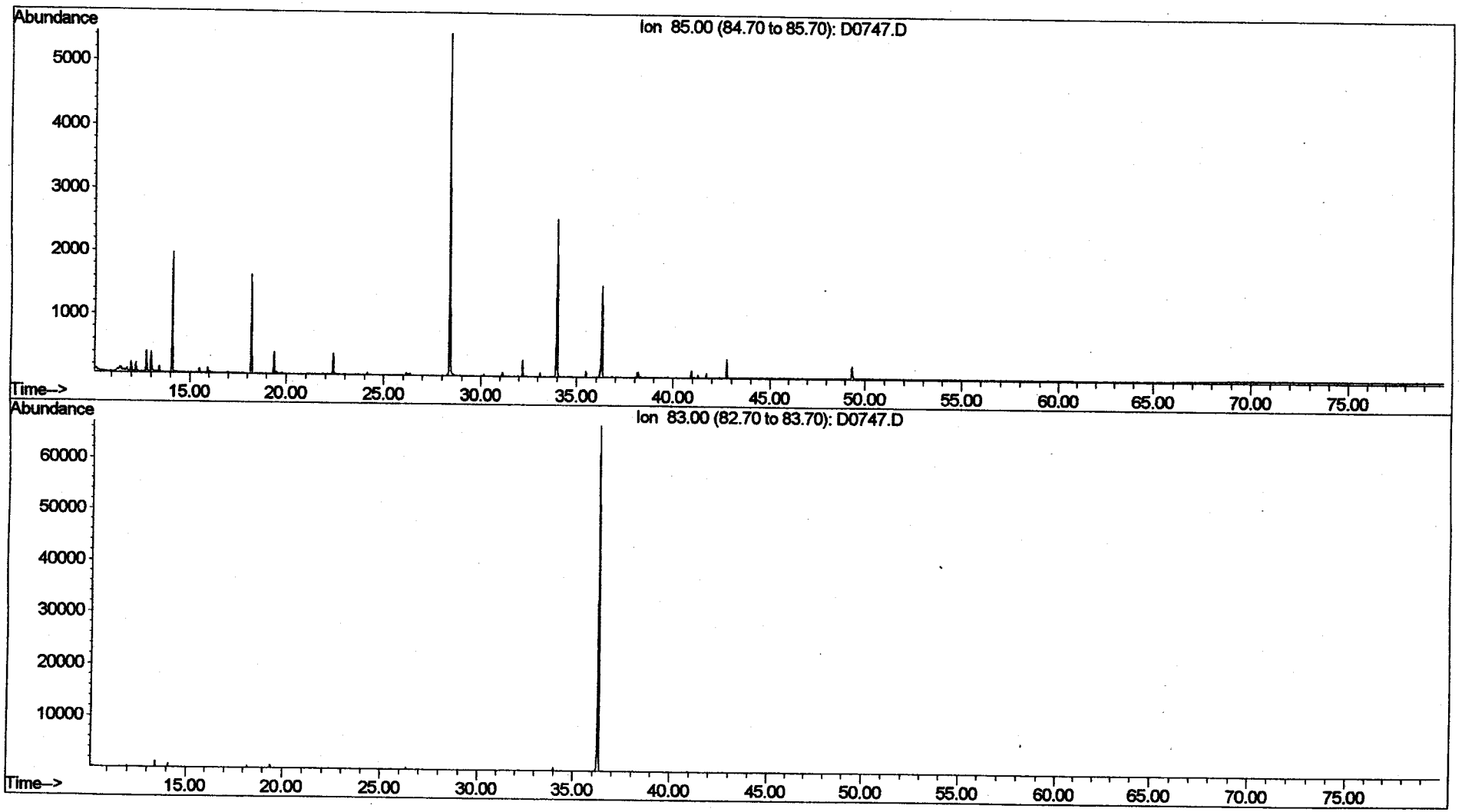
File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD295\D0804
Operator : SA
Acquired : 25 Oct 2001 5:59 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5545-1
Misc Info : MW-7
Vial Number: 15



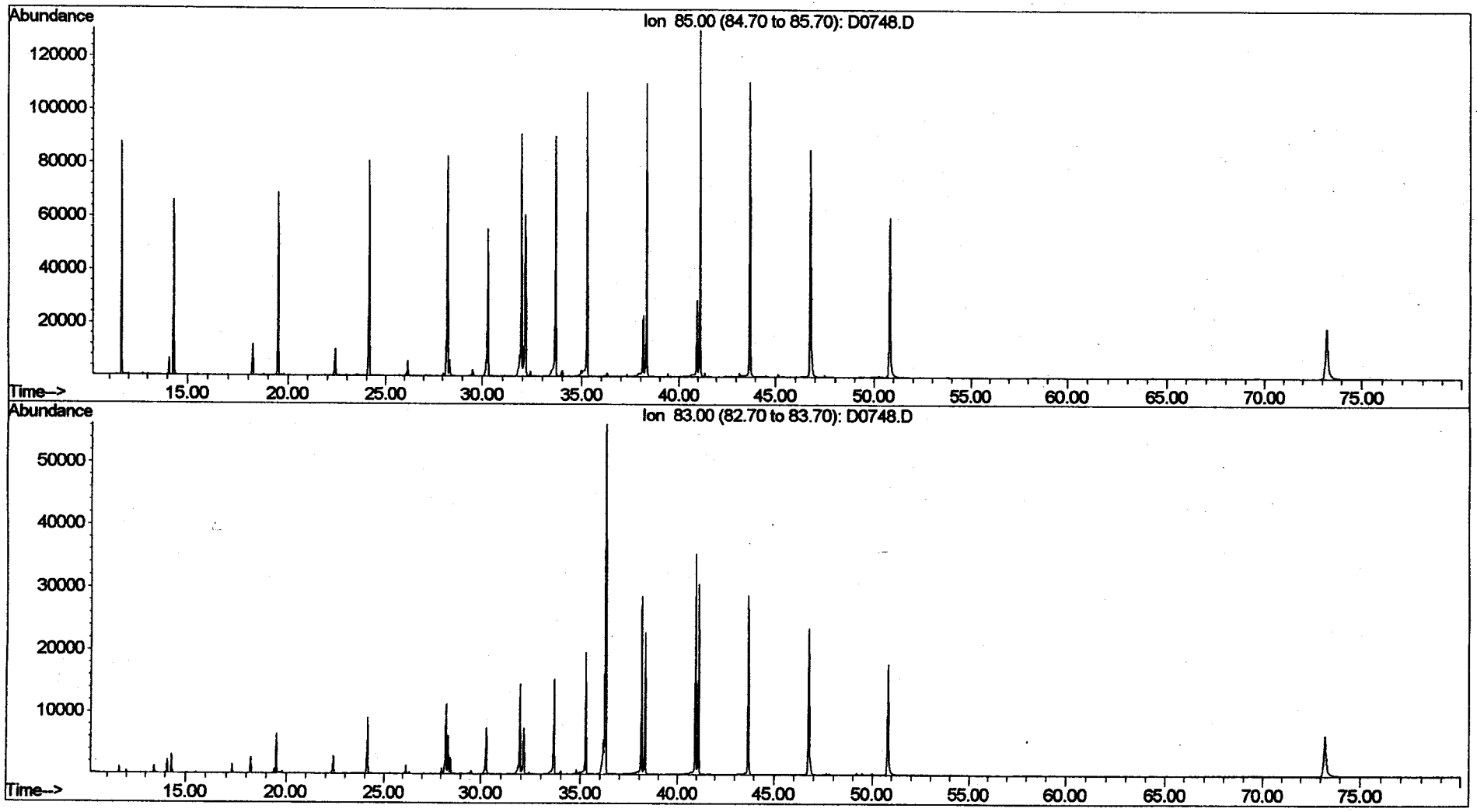
File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD295\D0806
Operator : SA
Acquired : 25 Oct 2001 9:19 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5546-1
Misc Info : TW-9
Vial Number: 17



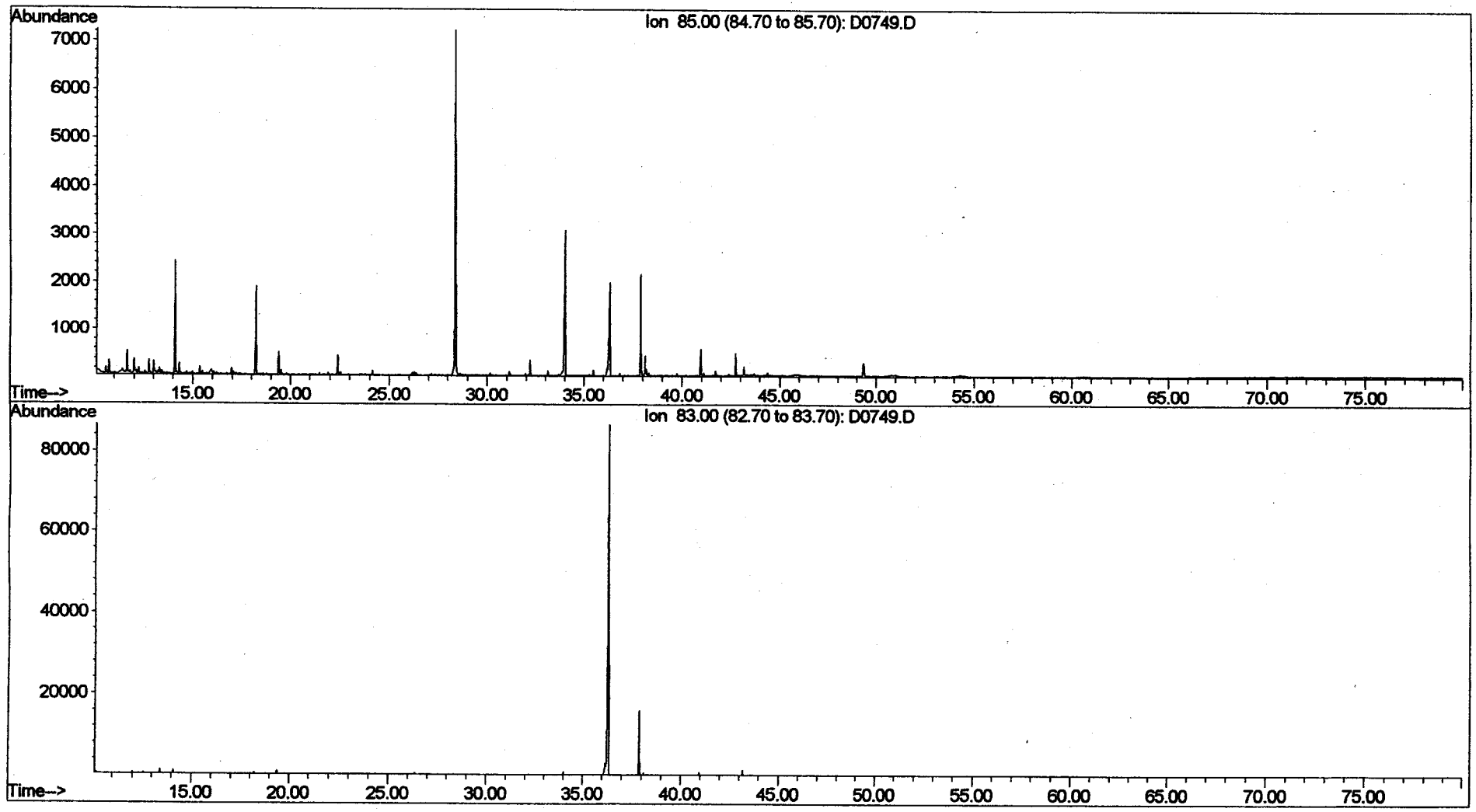
File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD292\D0747
Operator : SA
Acquired : 16 Oct 2001 8:56 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: ZK90PB
Misc Info : Procedural Blank
Vial Number: 10



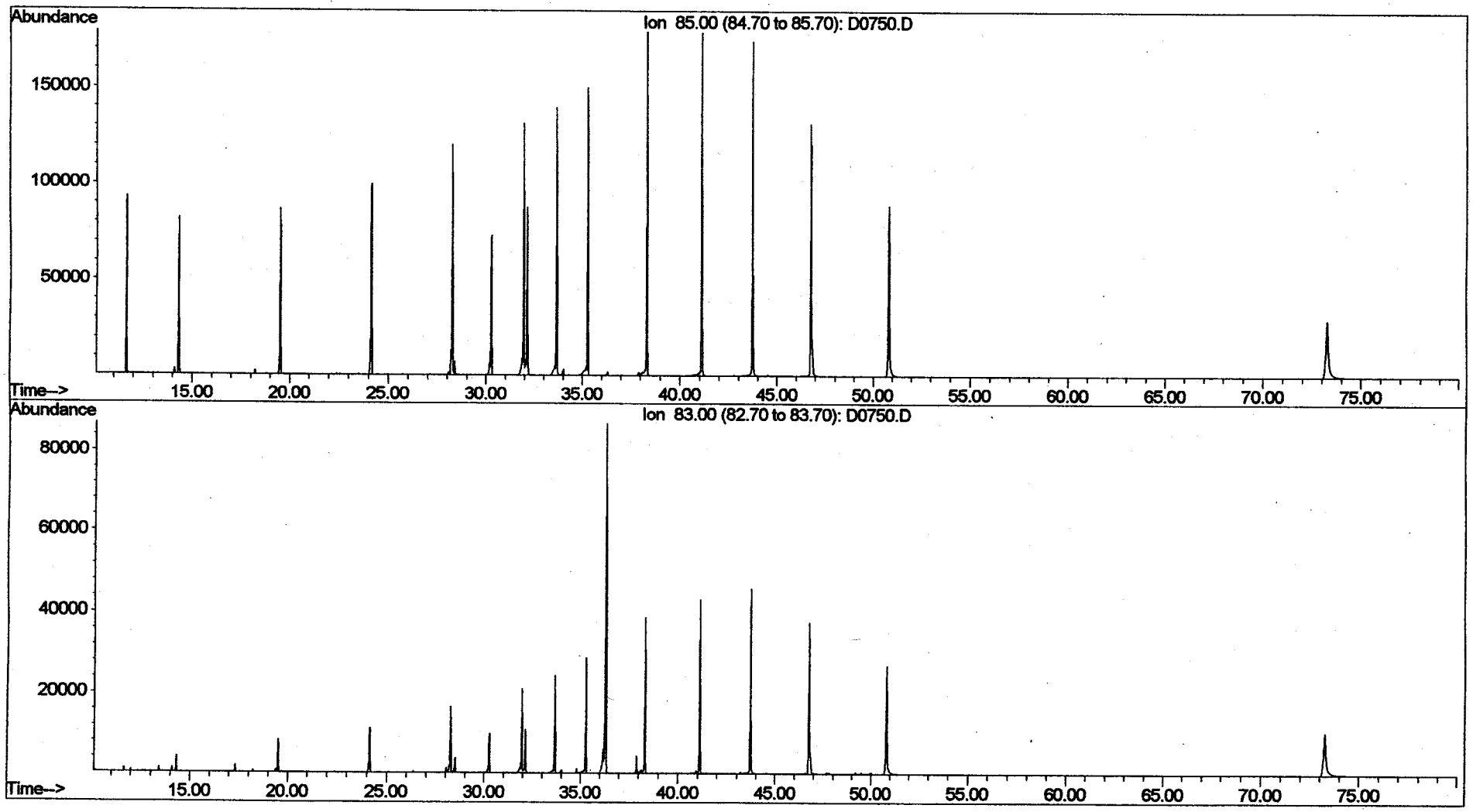
File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD292\D0748
Operator : SA
Acquired : 16 Oct 2001 10:28 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: ZK91LCS
Misc Info : Laboratory Control Spike
Vial Number: 11



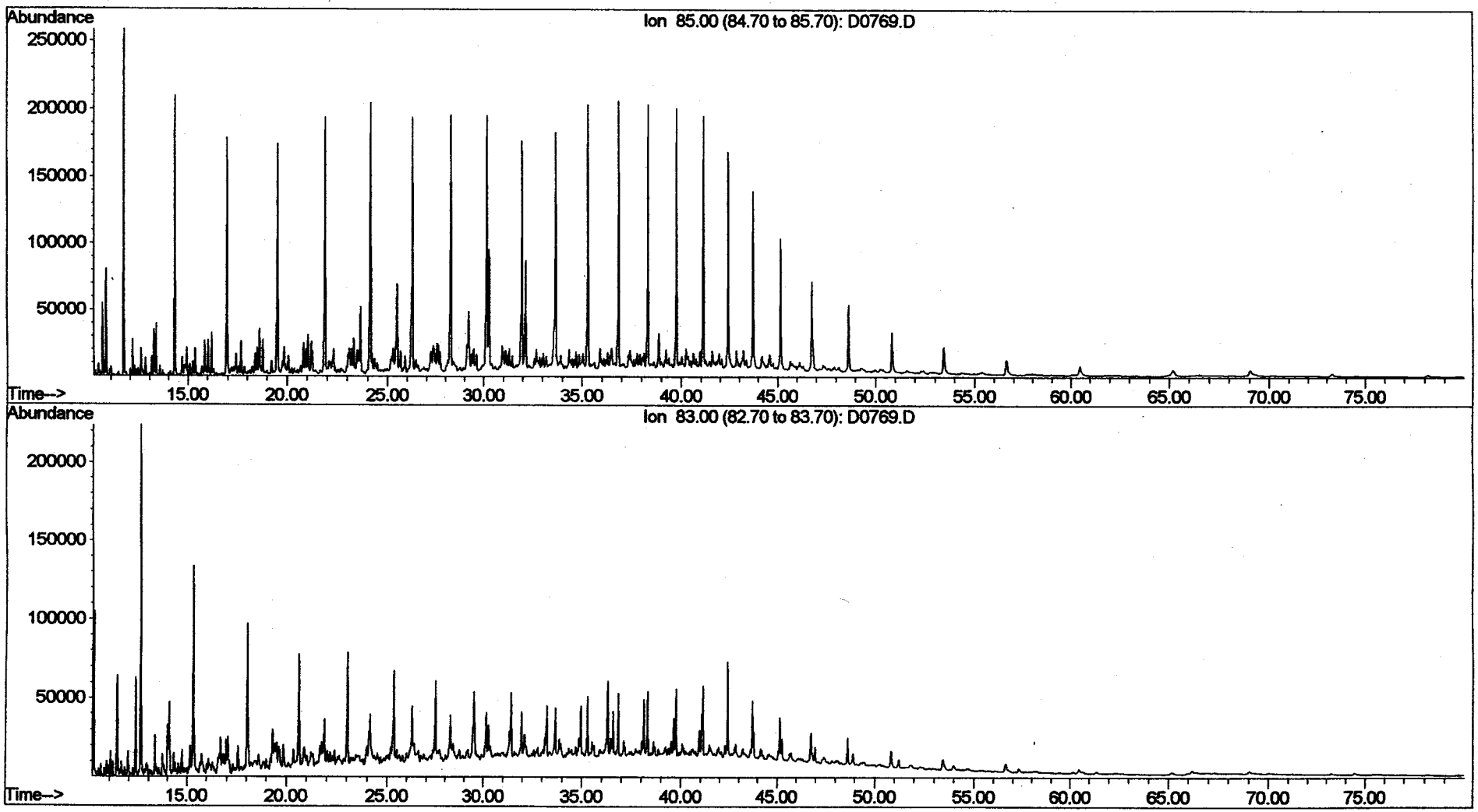
File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD292\D0749
Operator : SA
Acquired : 16 Oct 2001 12:01 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: ZL25PB
Misc Info : Procedural Blank
Vial Number: 12



File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD292\D0750
Operator : SA
Acquired : 16 Oct 2001 1:35 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: ZL26LCS
Misc Info : Laboratory Control Spike
Vial Number: 13



File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQD292\D0769
Operator : u
Acquired : 18 Oct 2001 11:10 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: ZL42NSC
Misc Info : North Slope Crude
Vial Number: 32

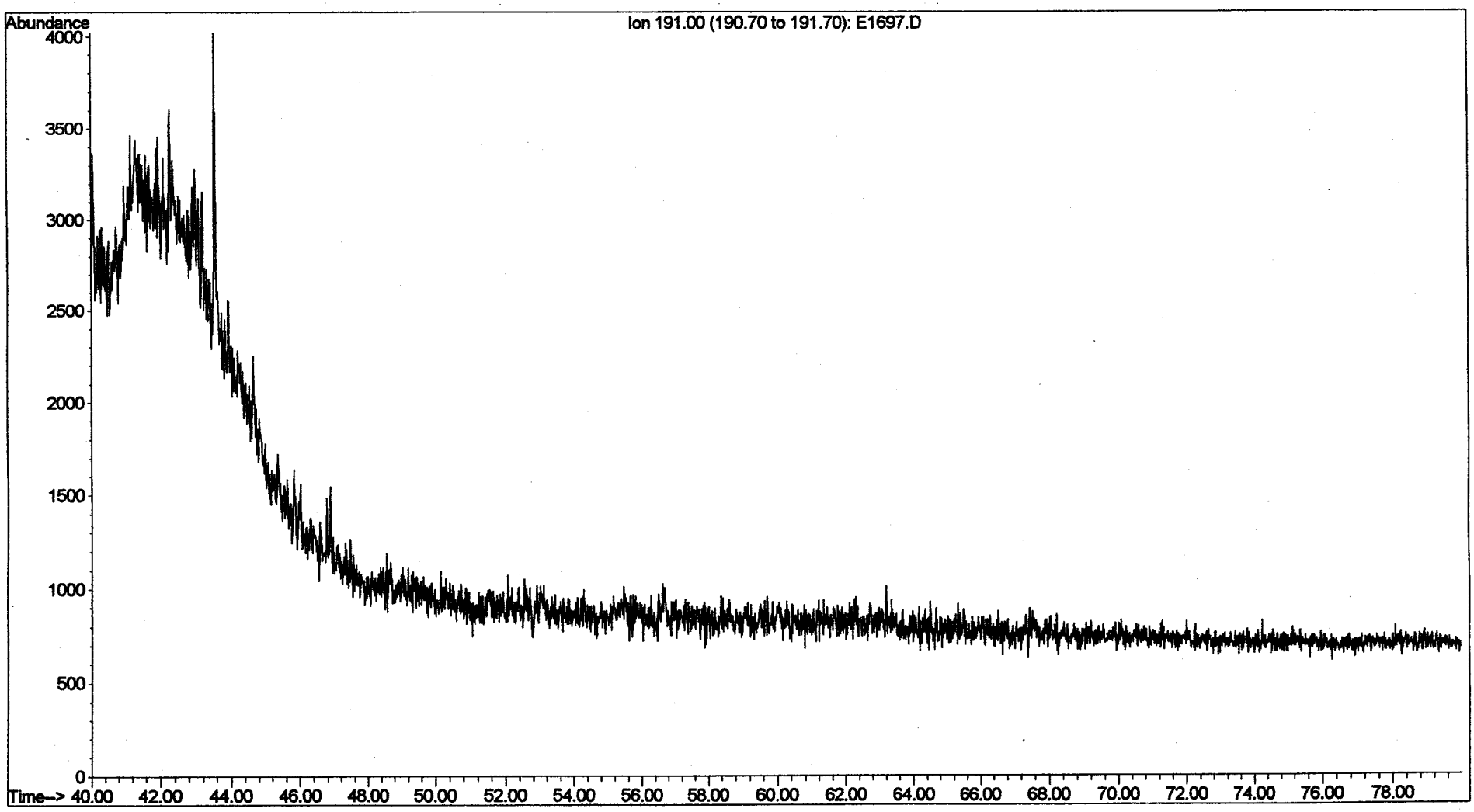


Appendix 8

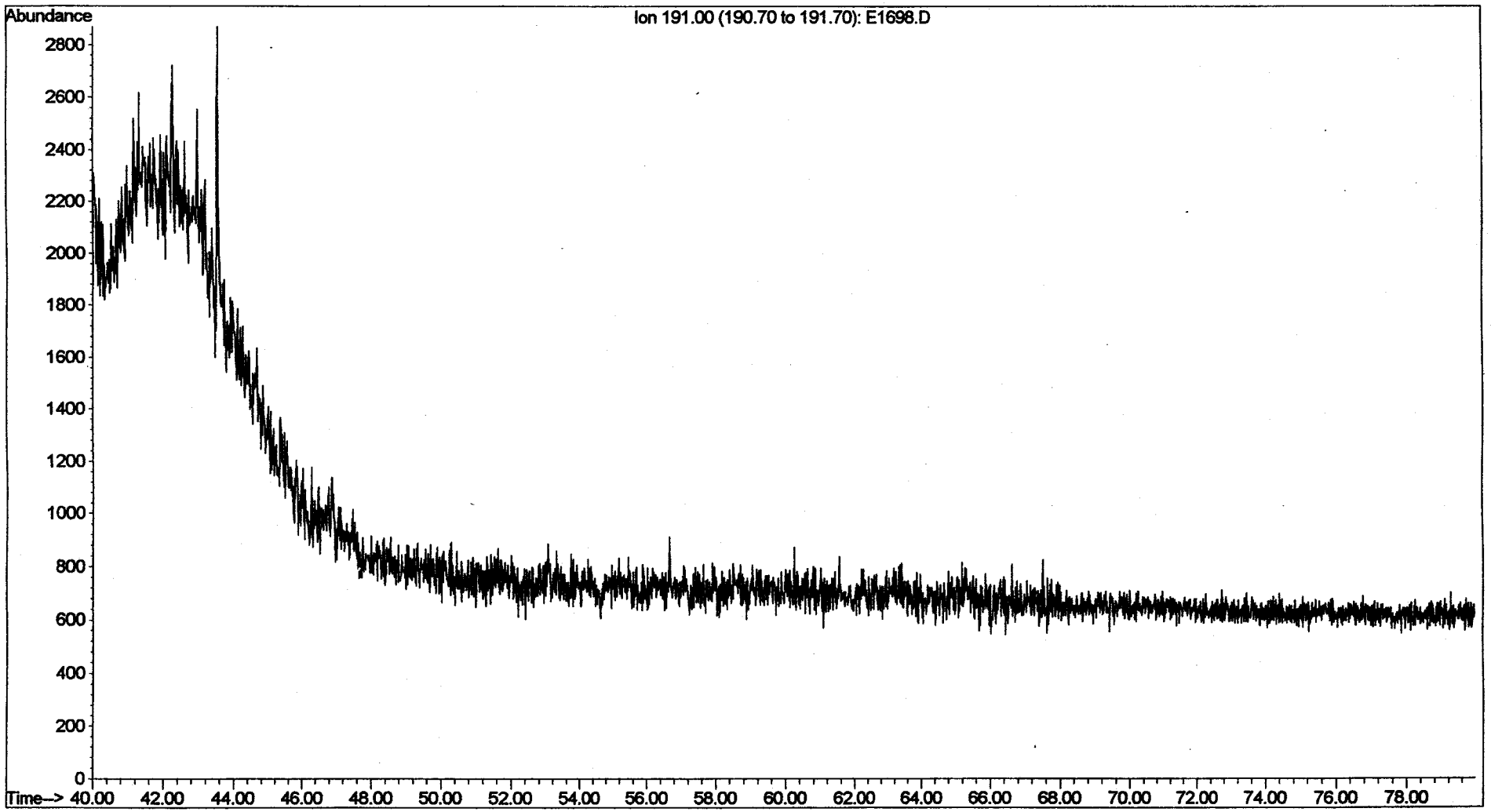
Biomarker Fingerprints:

Terpanes

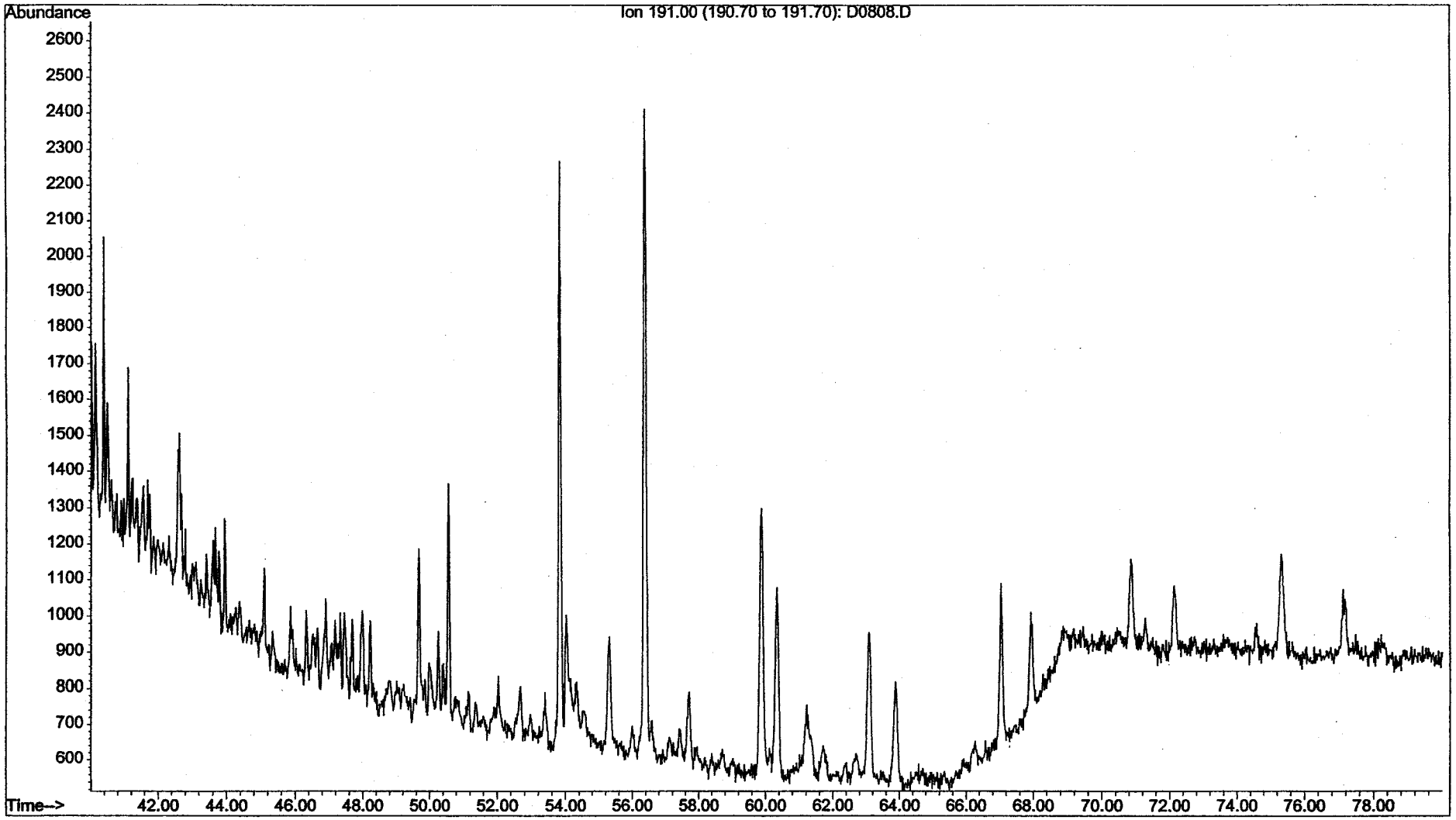
File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQE046\E1697
Operator : AC
Acquired : 23 Nov 2001 1:55 am using AcqMethod AQMETH6D
Instrument : GCMS-5
Sample Name: w9071
Misc Info : 2 Inch
Vial Number: 26



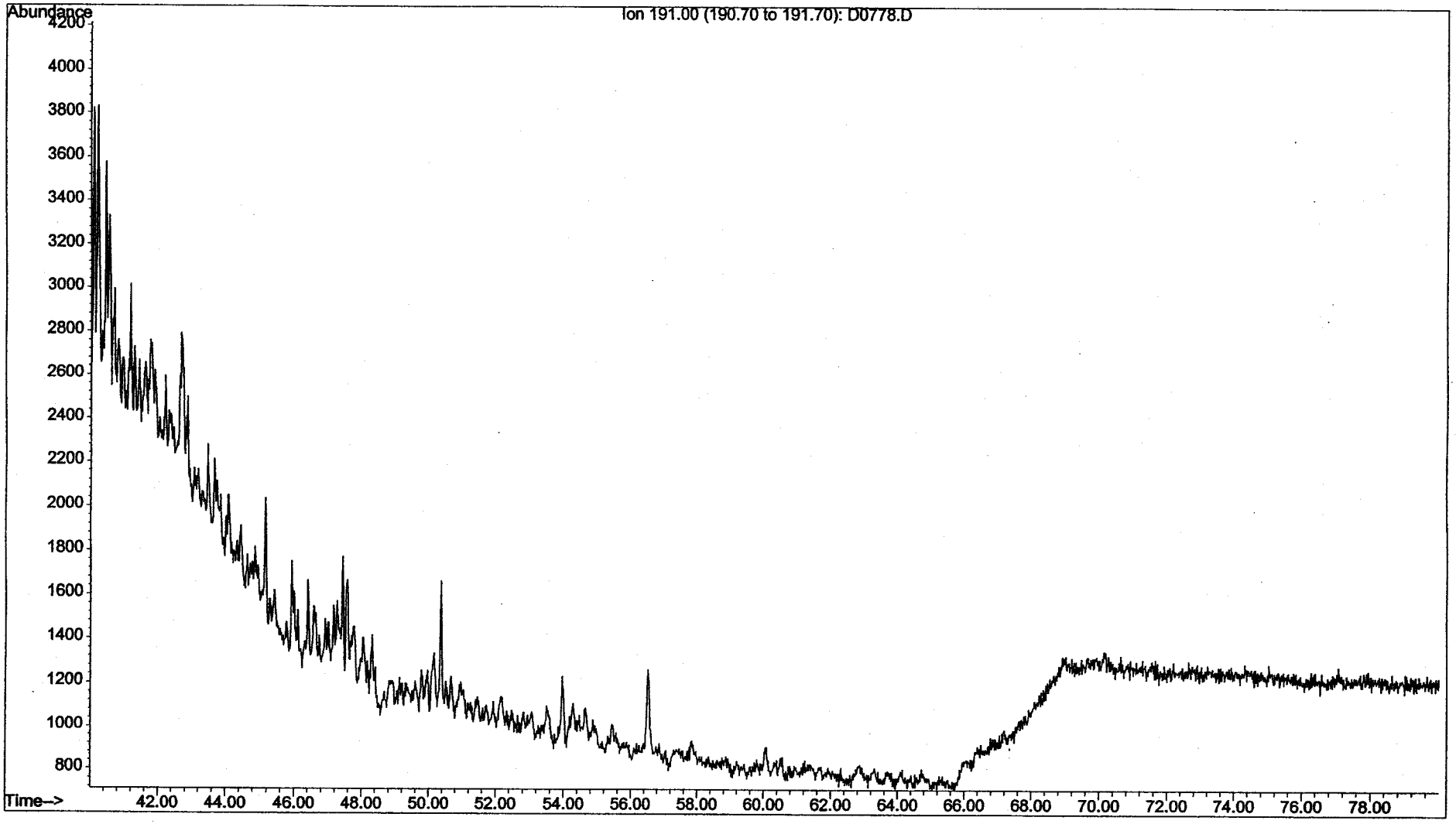
File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQE046\E1698
Operator : AC
Acquired : 23 Nov 2001 3:44 am using AcqMethod AQMETH6D
Instrument : GCMS-5
Sample Name: w9072
Misc Info : 12 Inch
Vial Number: 27



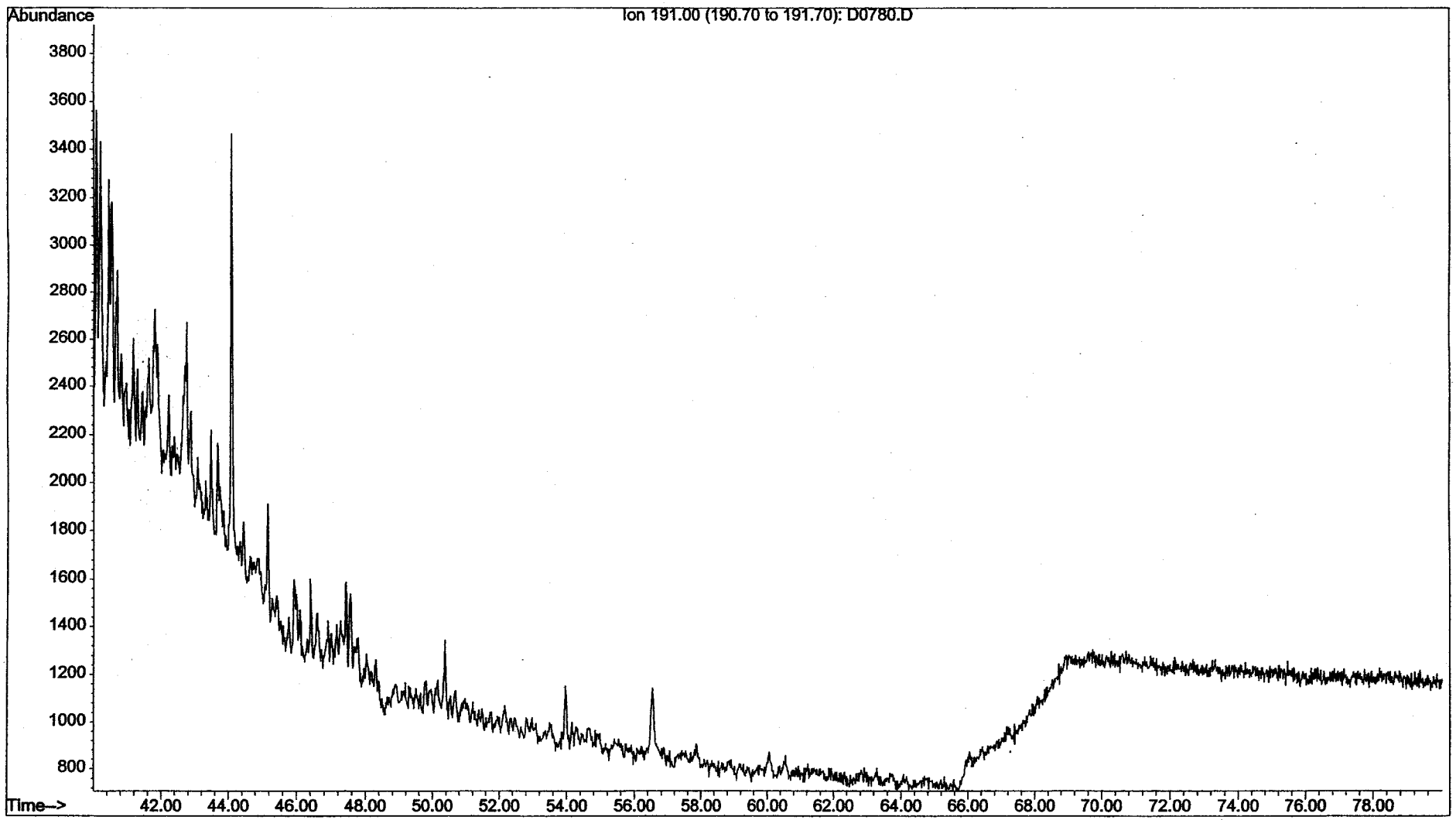
File : I:\D\DATA\SQD295\D0808.D
Operator : SA
Acquired : 25 Oct 2001 12:39 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5548-1
Misc Info : TW-13
Vial Number: 19



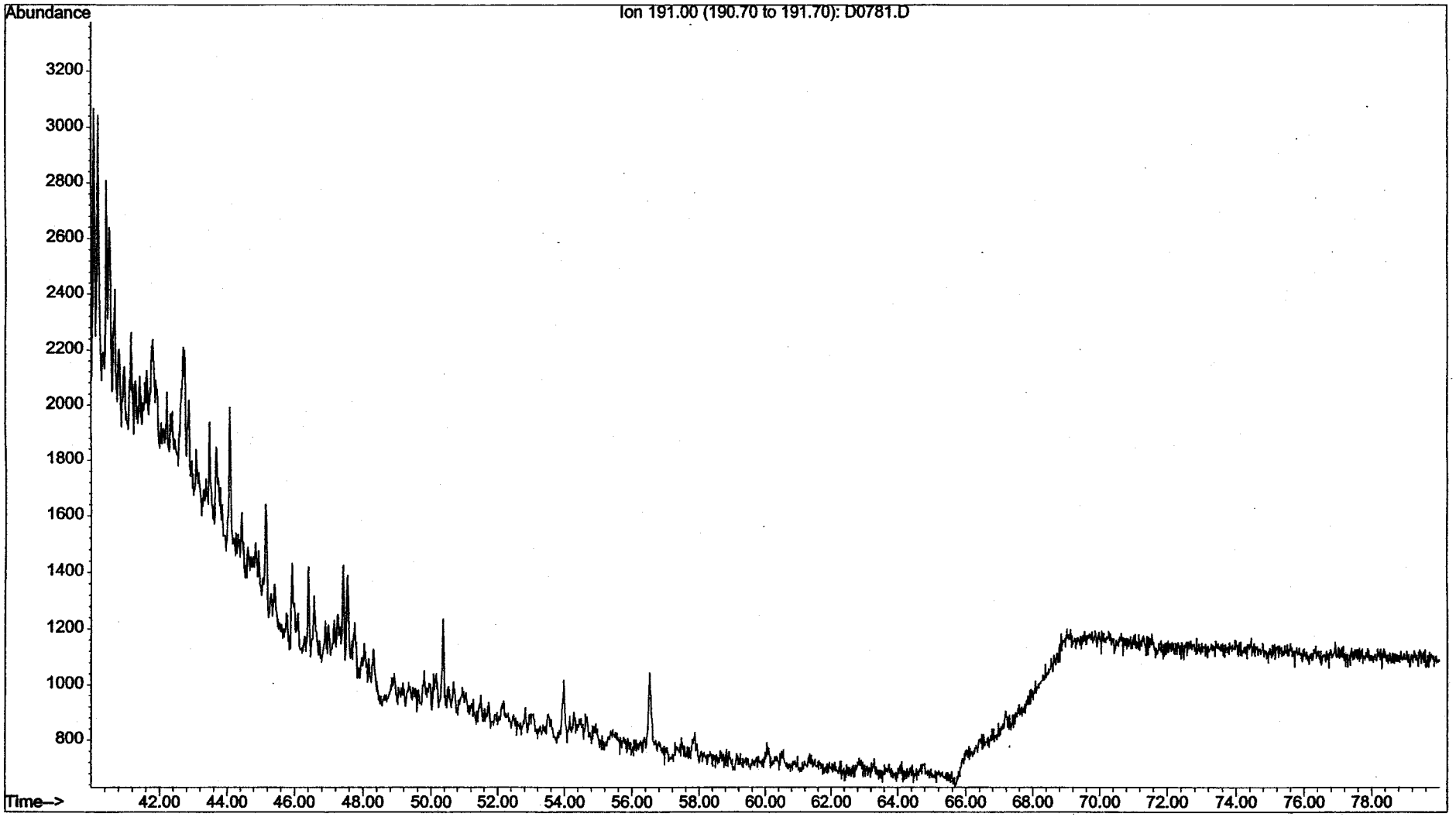
File : I:\D\DATA\SQD294\D0778.D
Operator : SA
Acquired : 22 Oct 2001 9:21 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W6841
Misc Info : Pipe Discharge East Yard Gate
Vial Number: 4



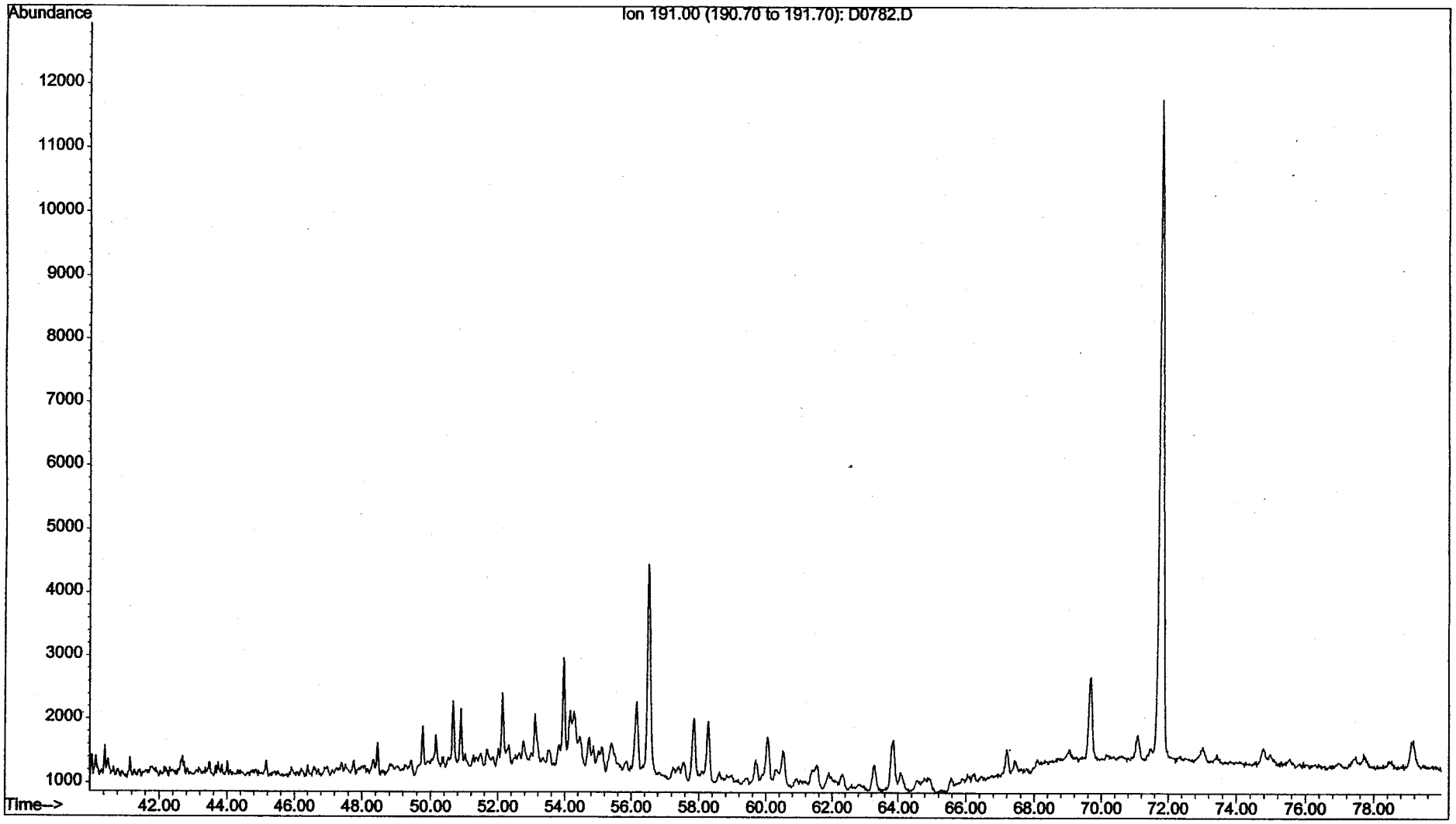
File : I:\D\DATA\SQD294\D0780.D
Operator : SA
Acquired : 23 Oct 2001 12:33 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W6839
Misc Info : Pipe Sludge East Yard Gate #1
Vial Number: 6



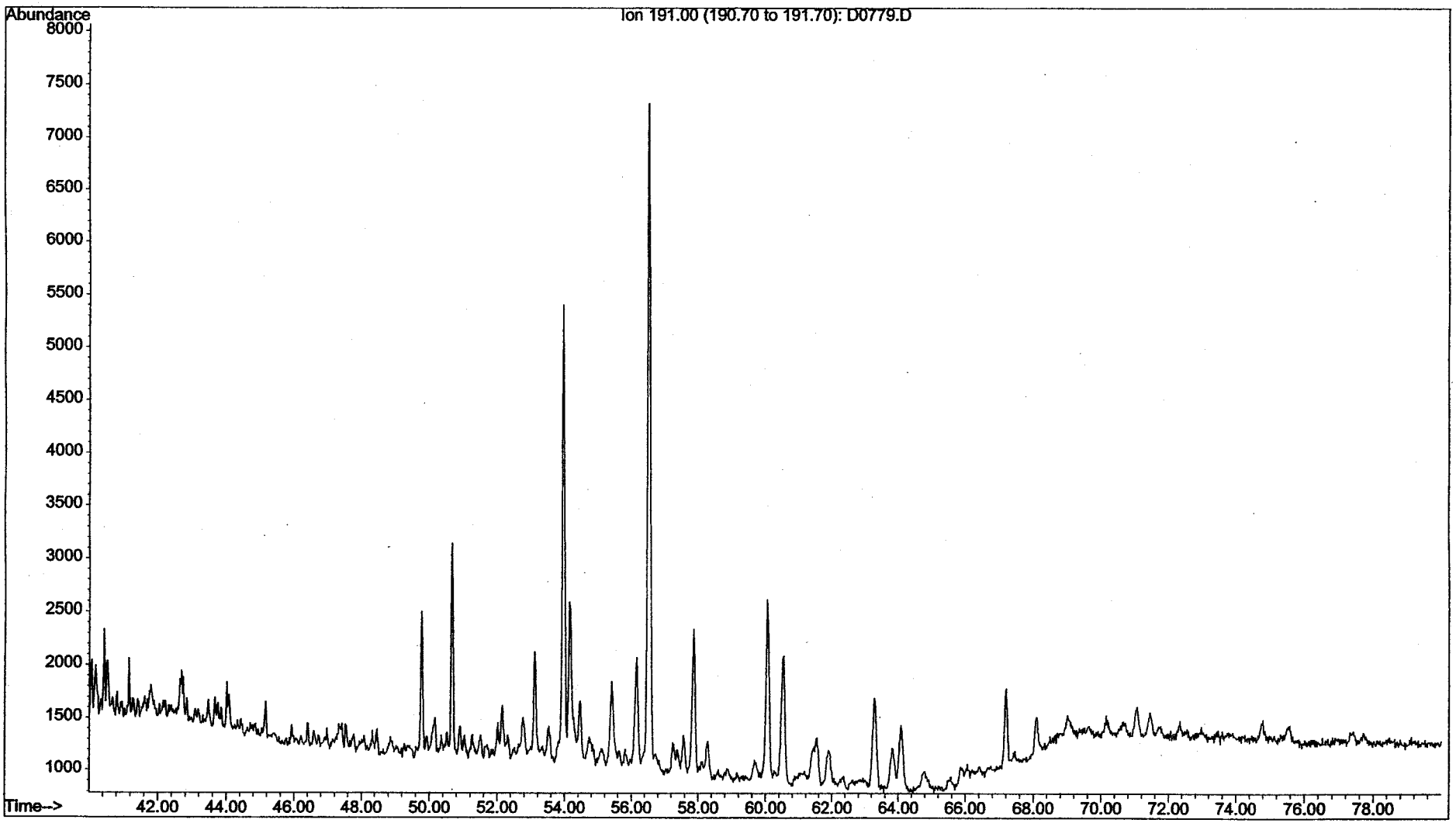
File : I:\D\DATA\SQD294\D0781.D
Operator : SA
Acquired : 23 Oct 2001 2:06 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W6840
Misc Info : Pipe Sludge East Yard Gate #2
Vial Number: 7



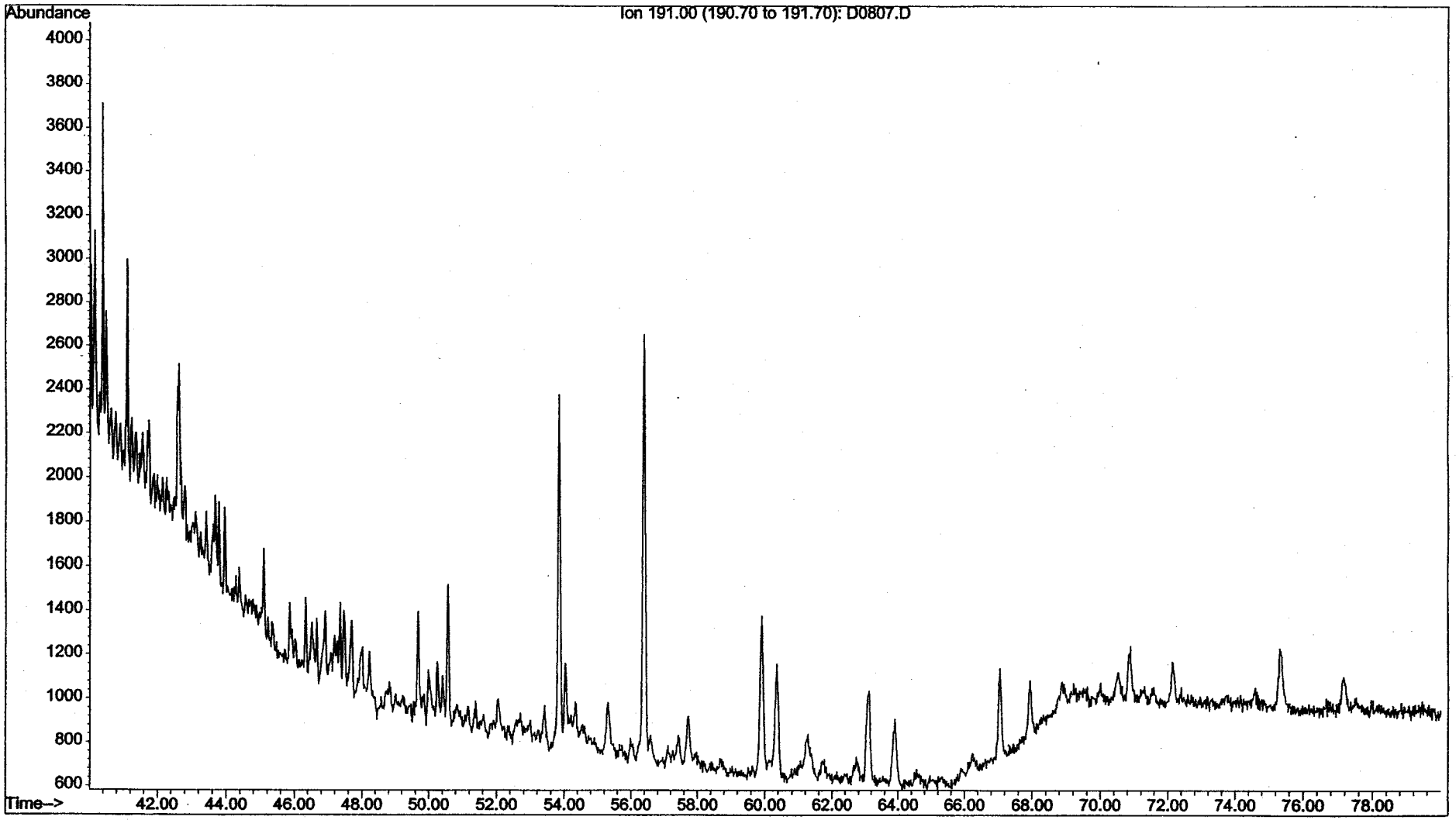
File : I:\D\DATA\SQD294\D0782.D
Operator : SA
Acquired : 23 Oct 2001 3:40 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W6837
Misc Info : Pipe Discharge Center Of Yard
Vial Number: 8



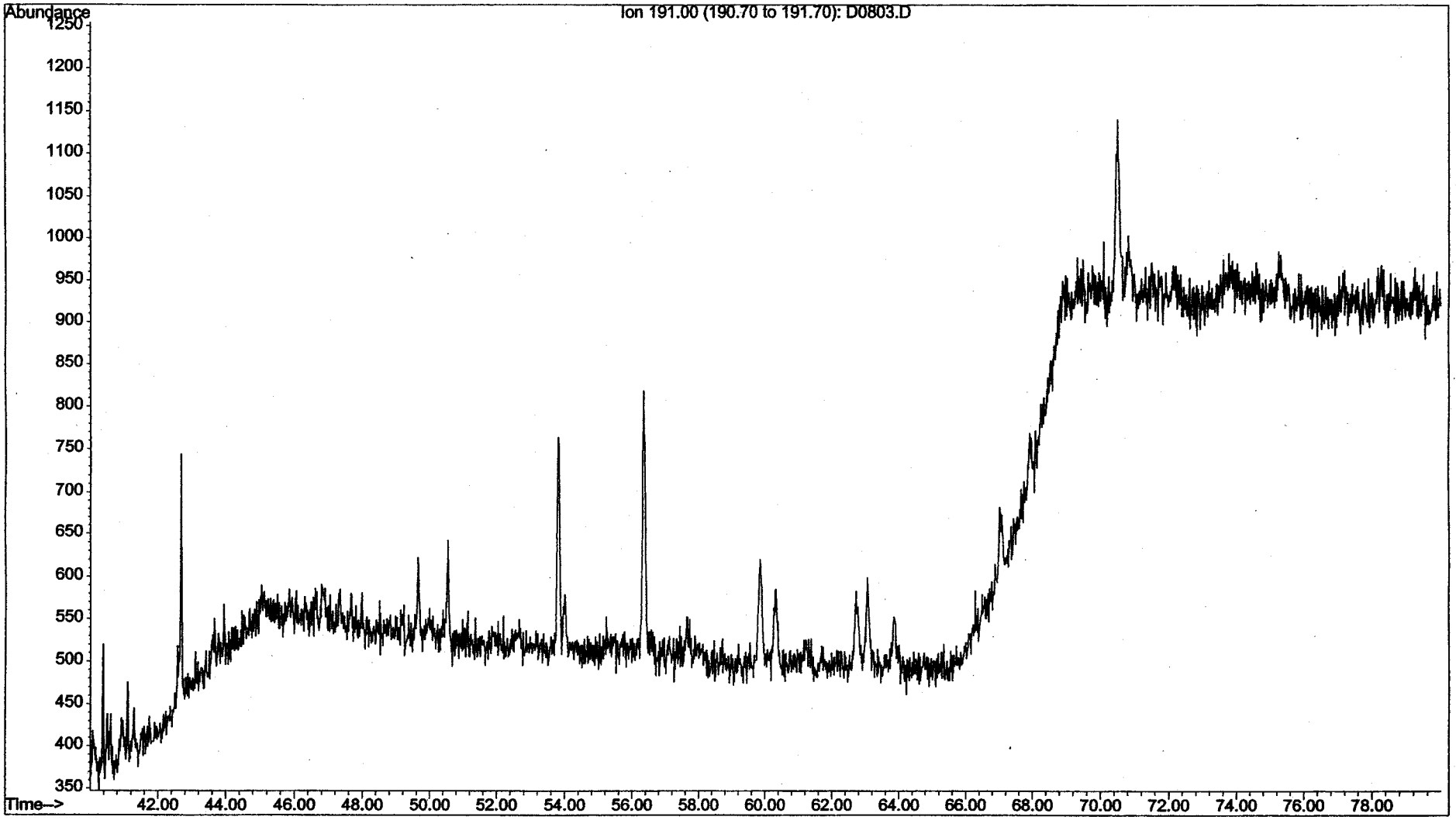
File : I:\D\DATA\SQD294\D0779.D
Operator : SA
Acquired : 22 Oct 2001 10:56 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W6838
Misc Info : Pipe Sludge Center of Yard
Vial Number: 5



File : I:\D\DATA\SQD295\D0807.D
Operator : SA
Acquired : 25 Oct 2001 11:01 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5547-1
Misc Info : Upgradient Riser
Vial Number: 18

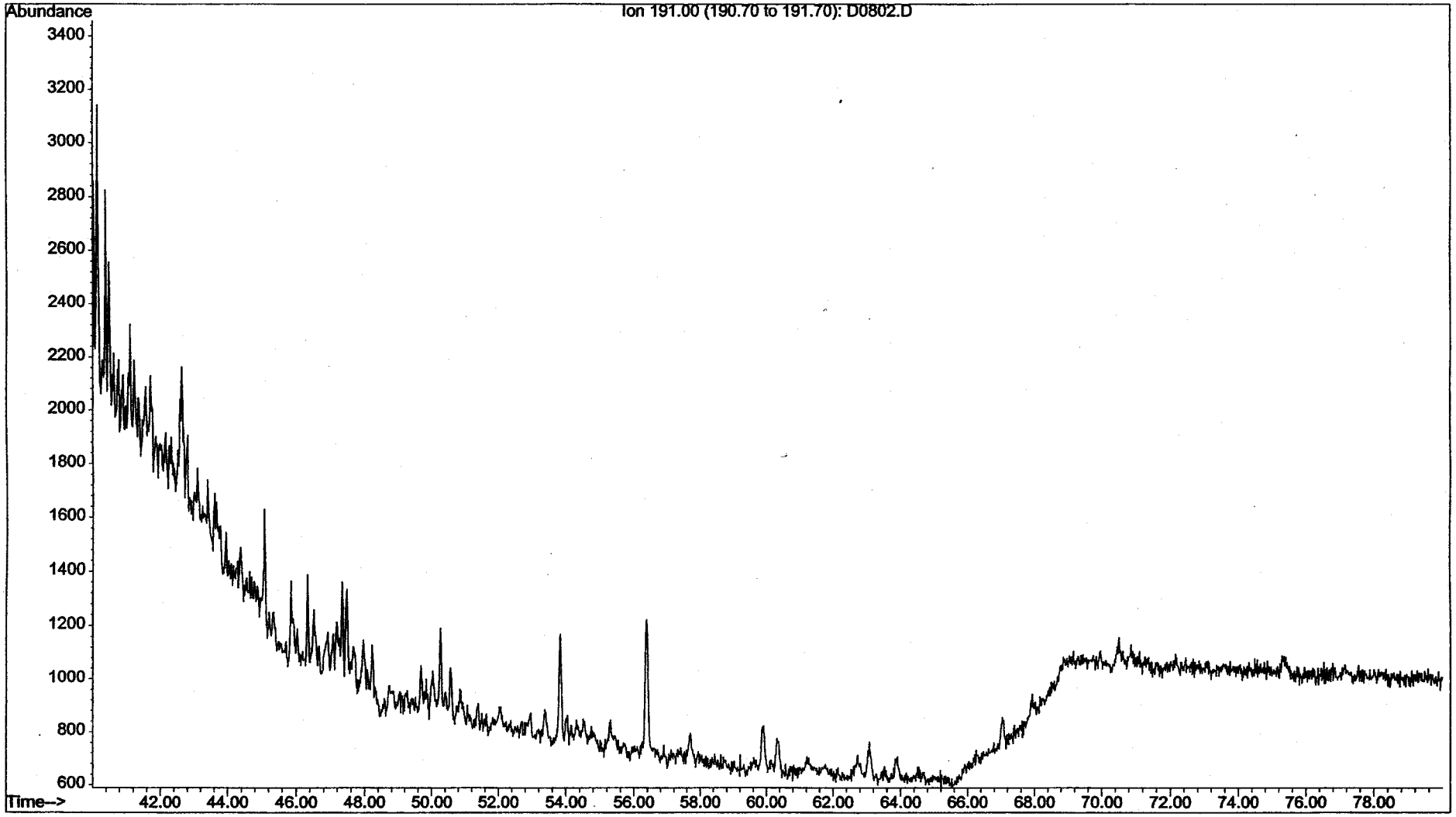


File : I:\D\DATA\SQD295\D0803.D
Operator : SA
Acquired : 25 Oct 2001 4:14 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5544-1
Misc Info : East Riser
Vial Number: 14

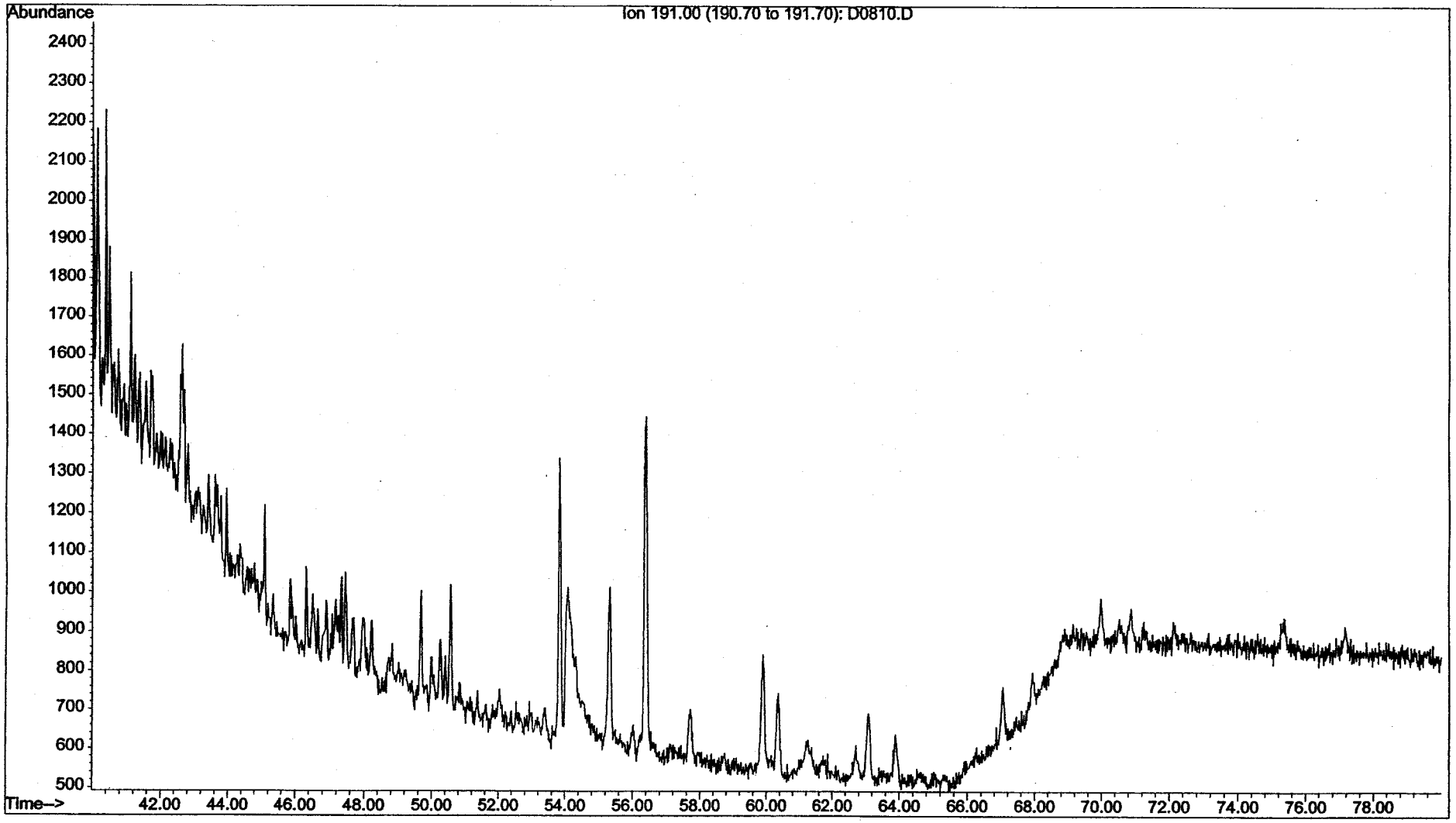


00298

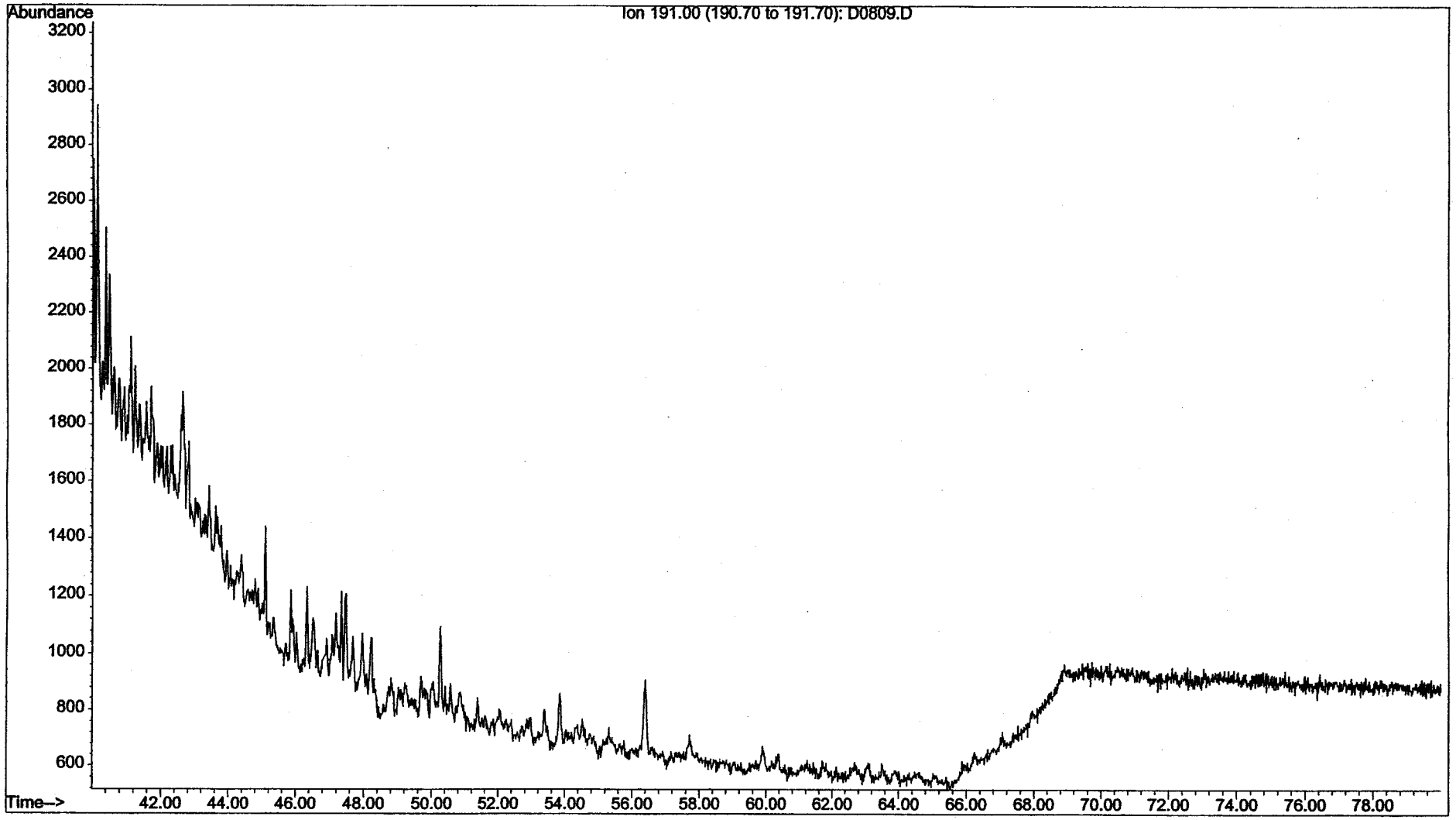
File : I:\D\DATA\SQD295\D0802.D
Operator : SA
Acquired : 25 Oct 2001 2:32 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5543-1
Misc Info : West Risen
Vial Number: 13



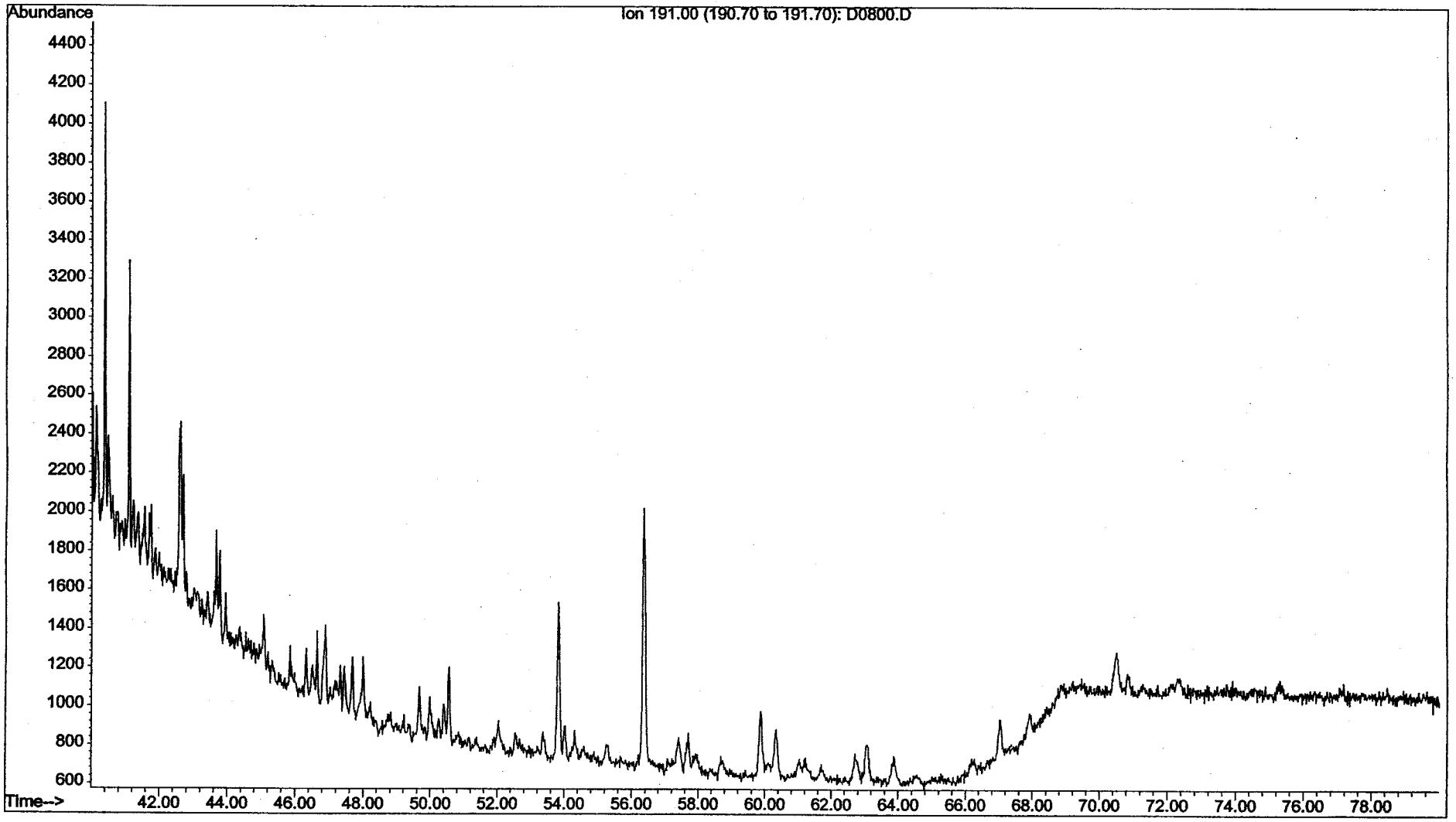
File : I:\D\DATA\SQD295\D0810.D
Operator : SA
Acquired : 25 Oct 2001 4:01 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5551-1
Misc Info : Scrapings from inside discharge
Vial Number: 21



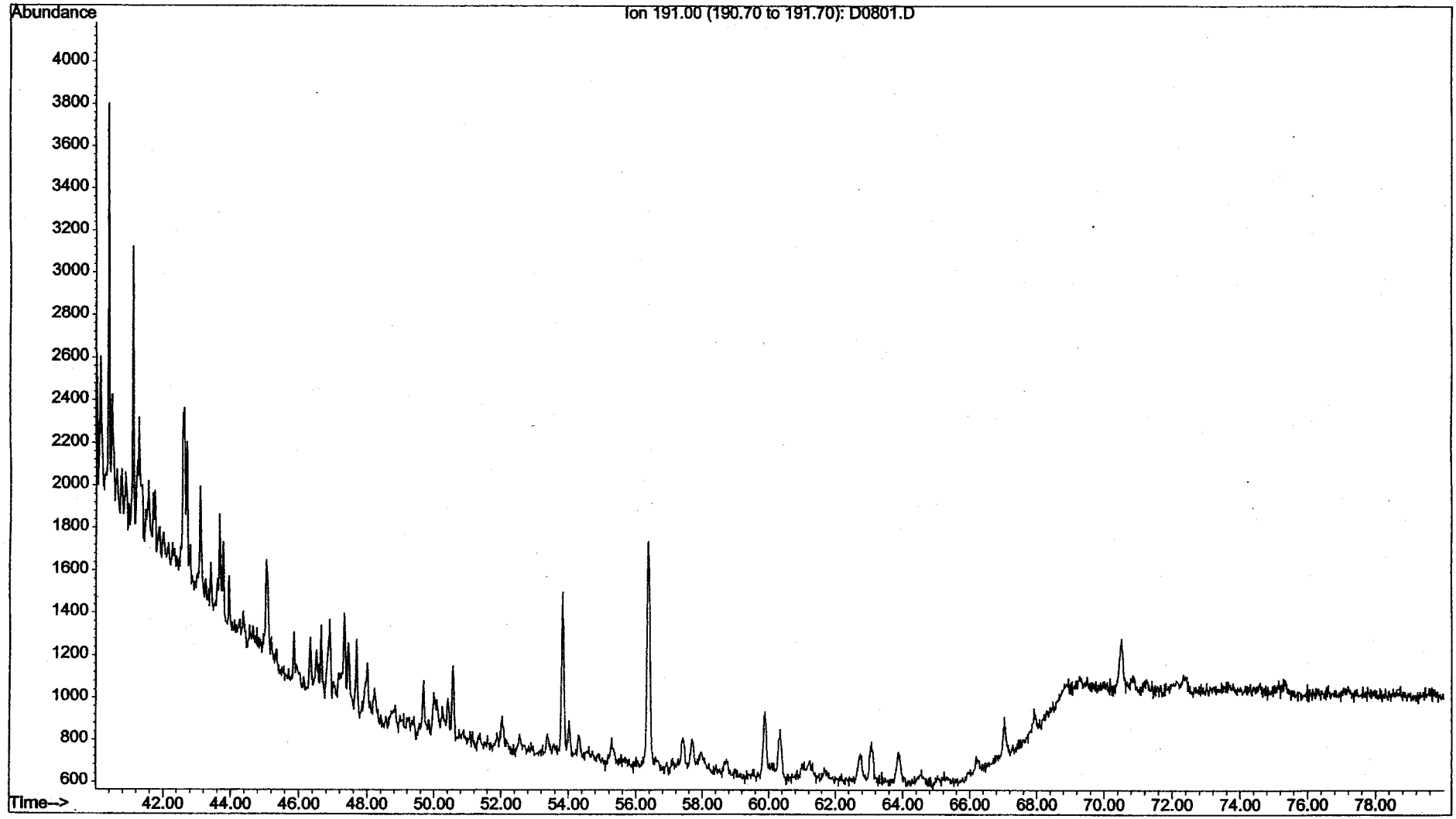
File : I:\D\DATA\SQD295\D0809.D
Operator : SA
Acquired : 25 Oct 2001 2:18 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5549-1
Misc Info : Pipe Discharge
Vial Number: 20



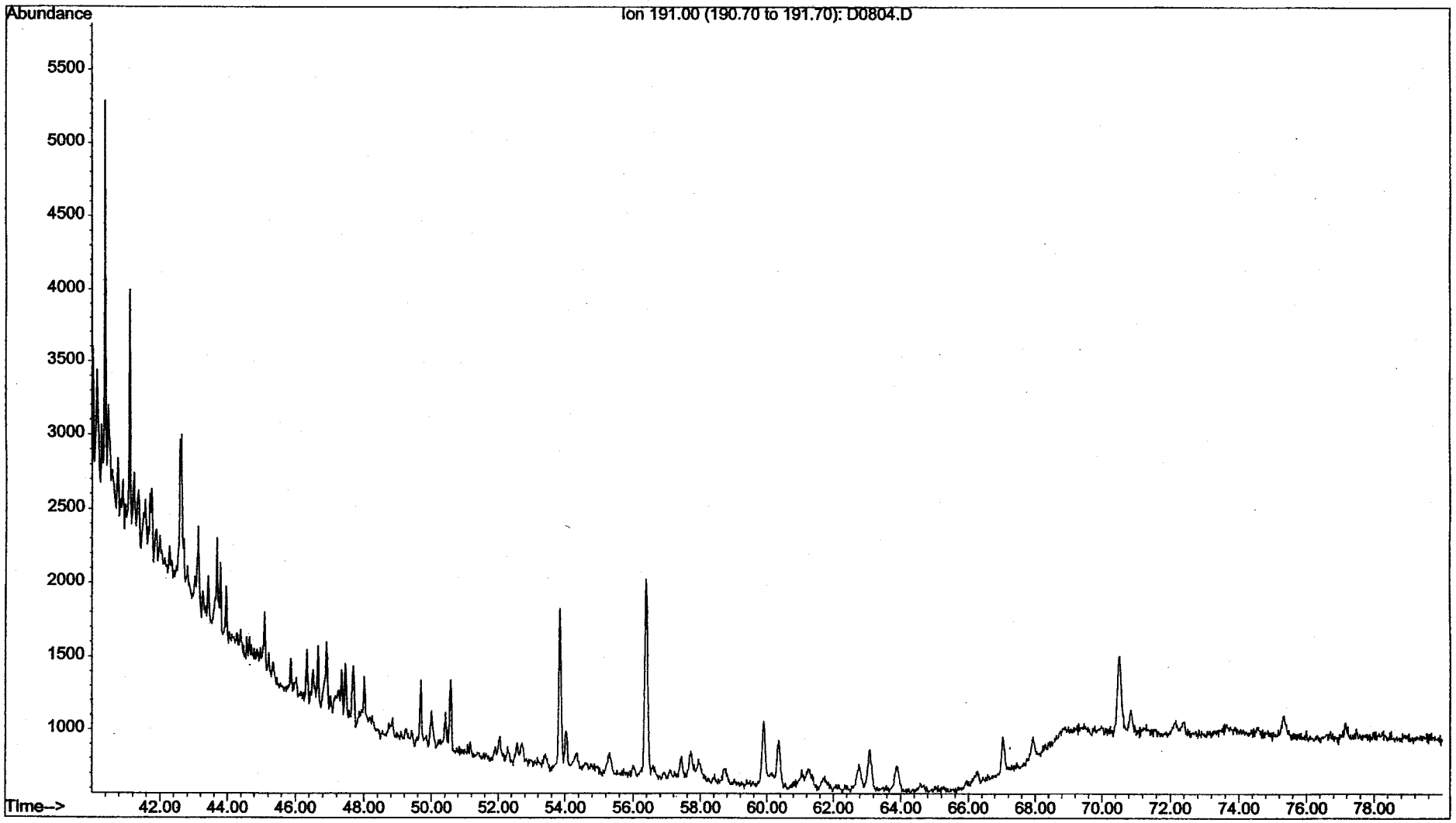
File : I:\D\DATA\SQD295\D0800.D
Operator : SA
Acquired : 24 Oct 2001 11:23 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5550-1
Misc Info : Solids around discharge pipe
Vial Number: 11



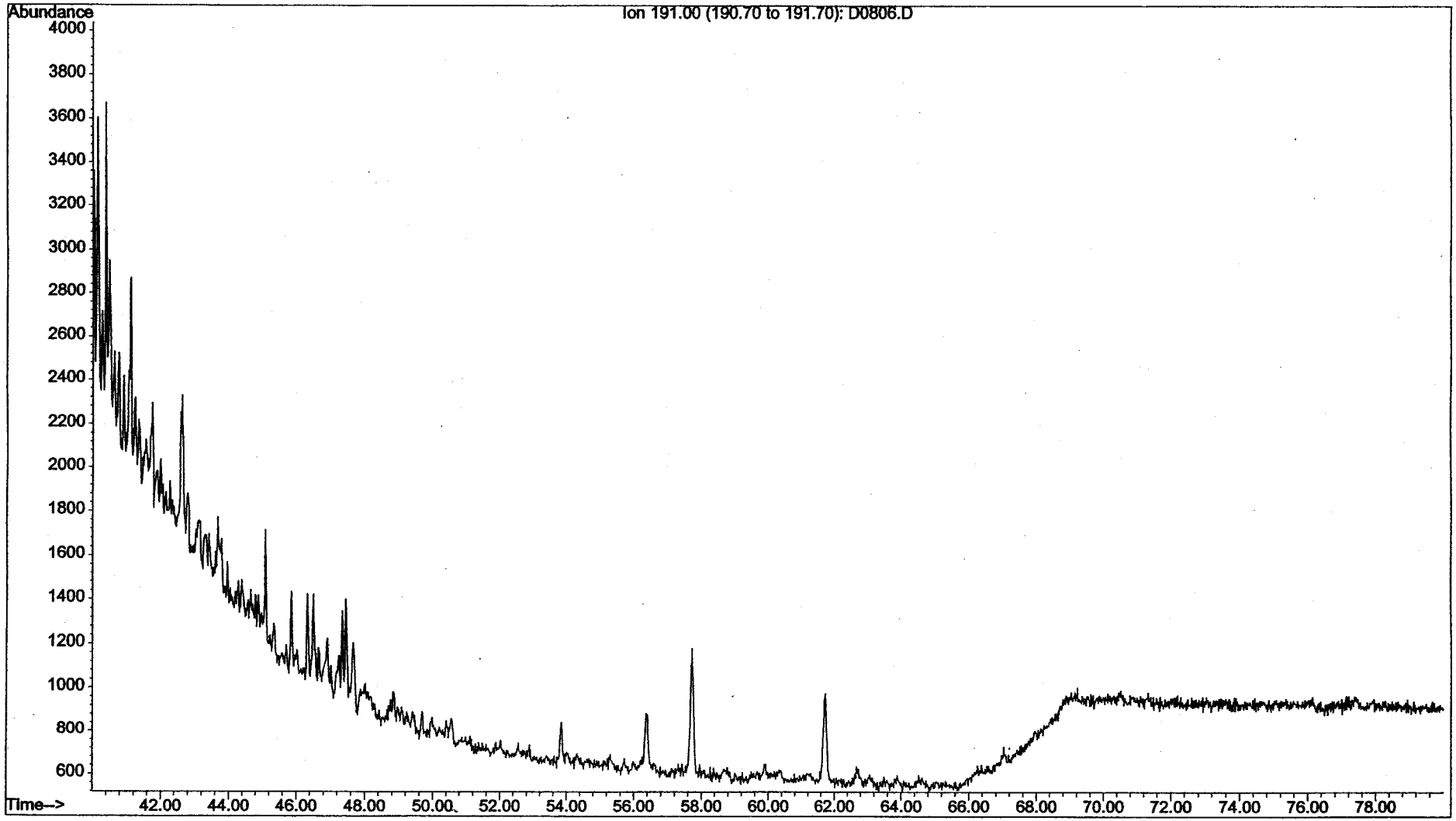
File : I:\D\DATA\SQD295\D0801.D
Operator : SA
Acquired : 25 Oct 2001 12:57 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5550DUP-1
Misc Info : Solids around discharge pipe
Vial Number: 12



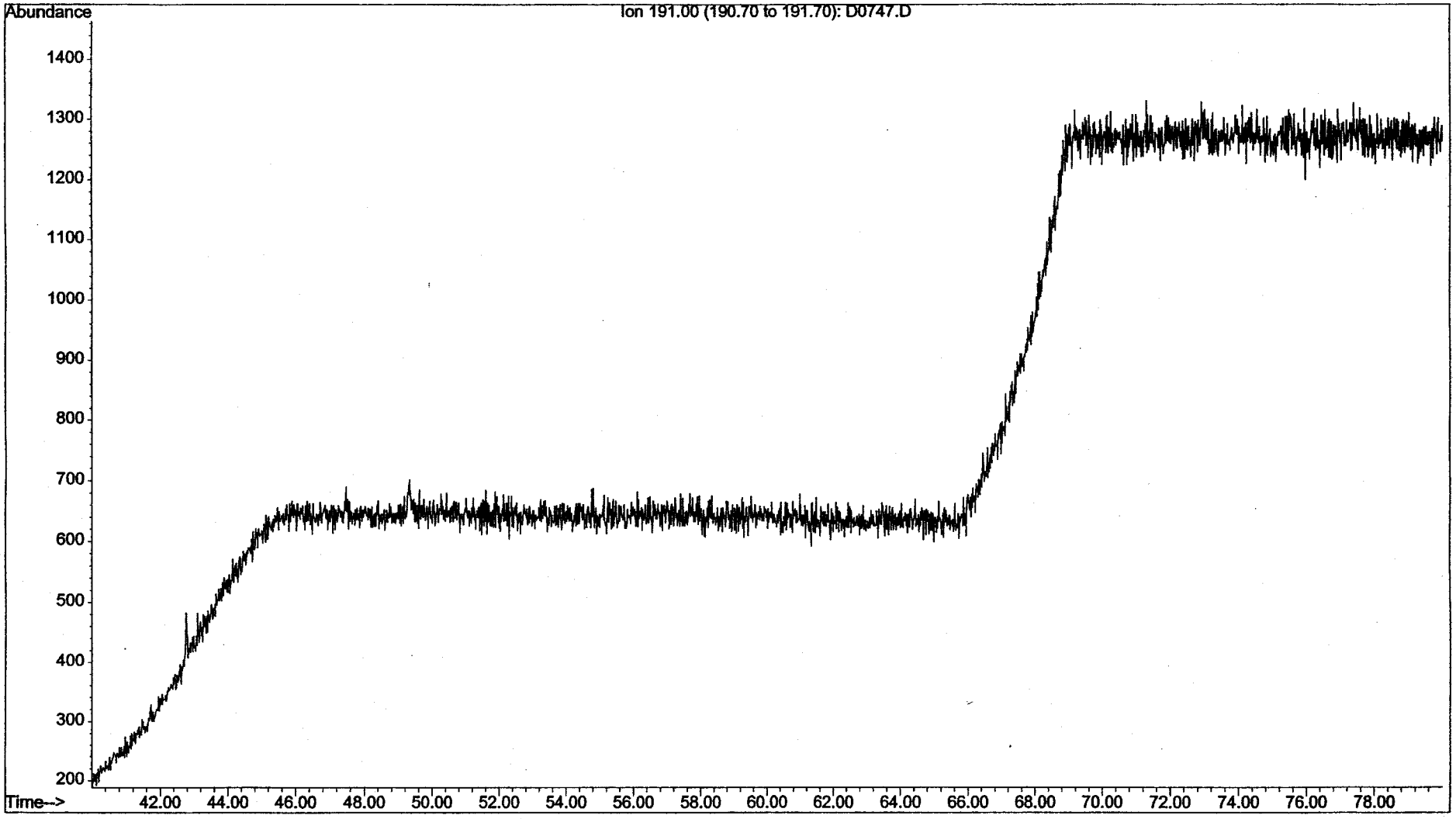
File : I:\D\DATA\SQD295\D0804.D
Operator : SA
Acquired : 25 Oct 2001 5:59 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5545-1
Misc Info : MW-7
Vial Number: 15



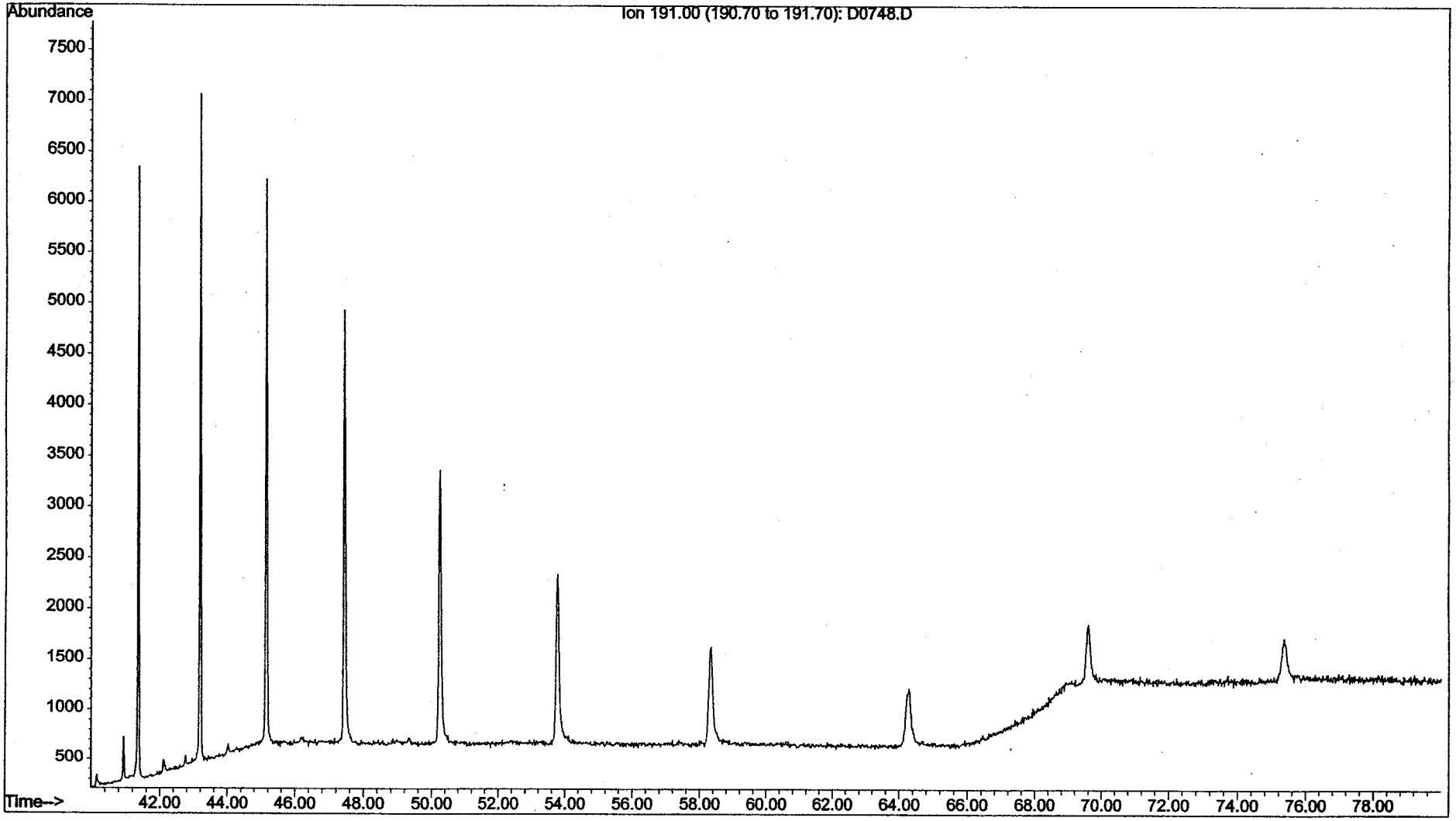
File : I:\D\DATA\SQD295\D0806.D
Operator : SA
Acquired : 25 Oct 2001 9:19 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5546-1
Misc Info : TW-9
Vial Number: 17



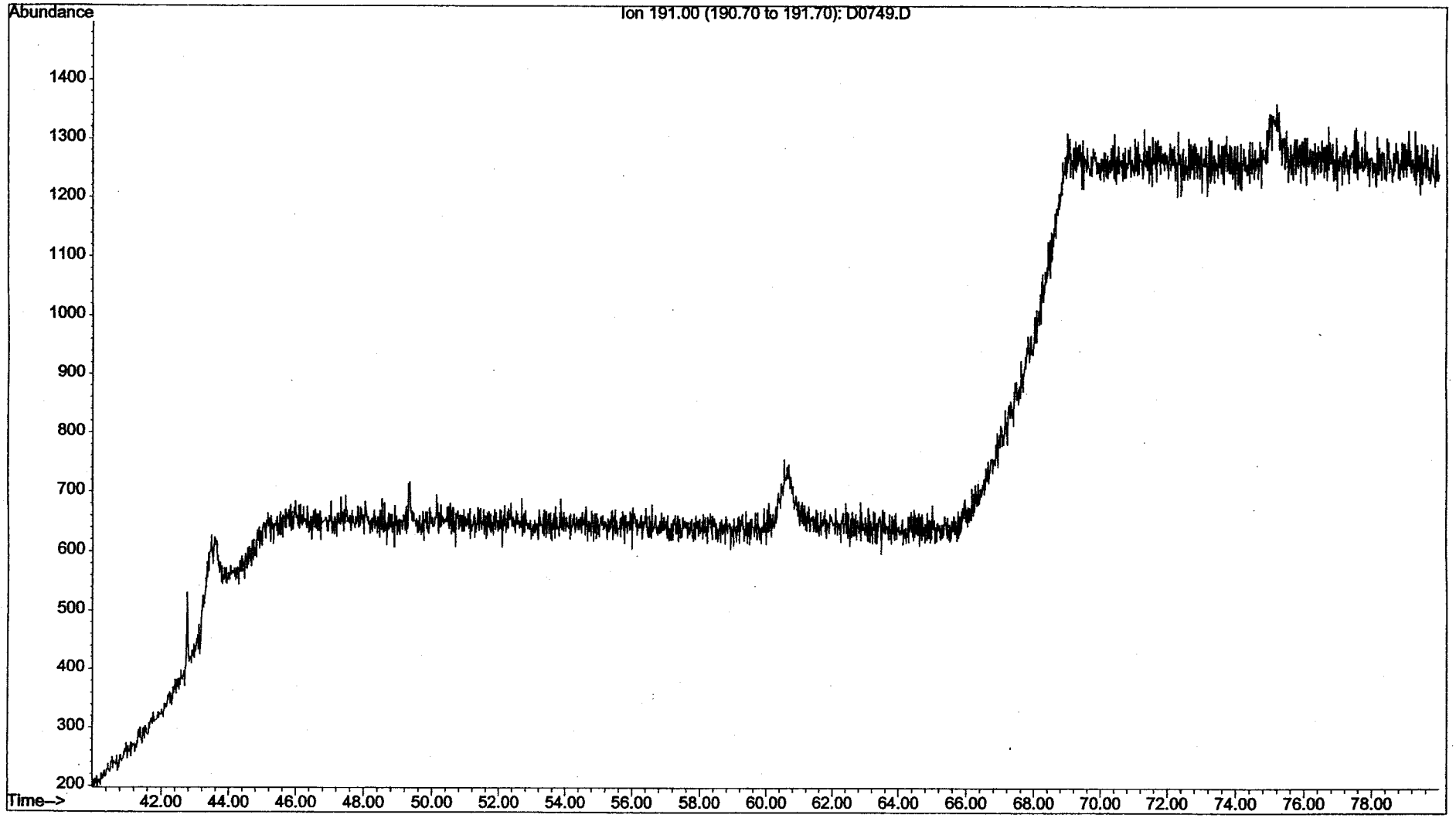
File : I:\D\DATA\SQD292\D0747.D
Operator : SA
Acquired : 16 Oct 2001 8:56 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: ZK90PB
Misc Info : Procedural Blank
Vial Number: 10



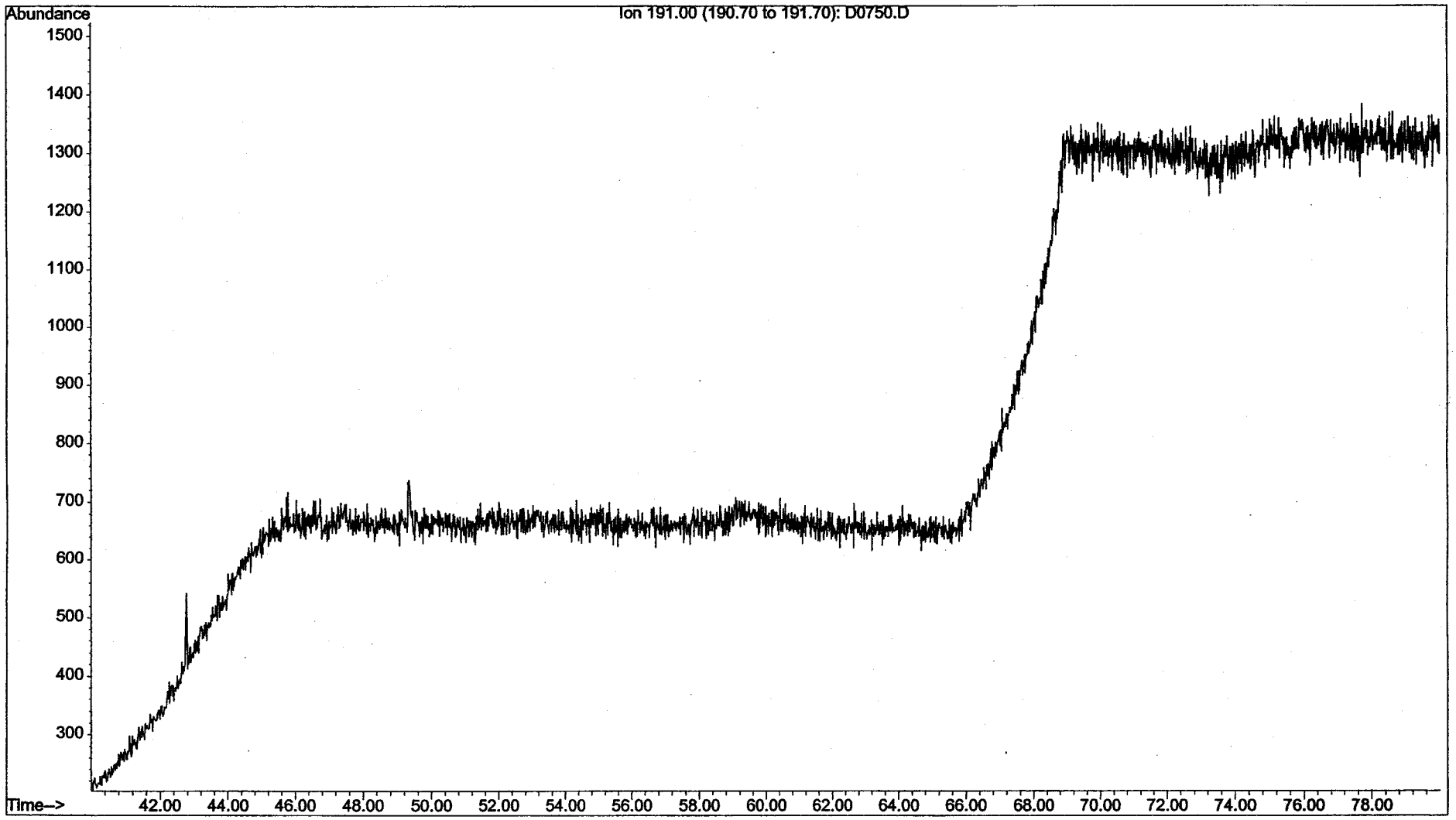
File : I:\D\DATA\SQD292\D0748.D
Operator : SA
Acquired : 16 Oct 2001 10:28 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: ZK91LCS
Misc Info : Laboratory Control Spike
Vial Number: 11



File : I:\D\DATA\SQD292\D0749.D
Operator : SA
Acquired : 16 Oct 2001 12:01 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: ZL25PB
Misc Info : Procedural Blank
Vial Number: 12



File : I:\D\DATA\SQD292\D0750.D
Operator : SA
Acquired : 16 Oct 2001 1:35 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: ZL26LCS
Misc Info : Laboratory Control Spike
Vial Number: 13

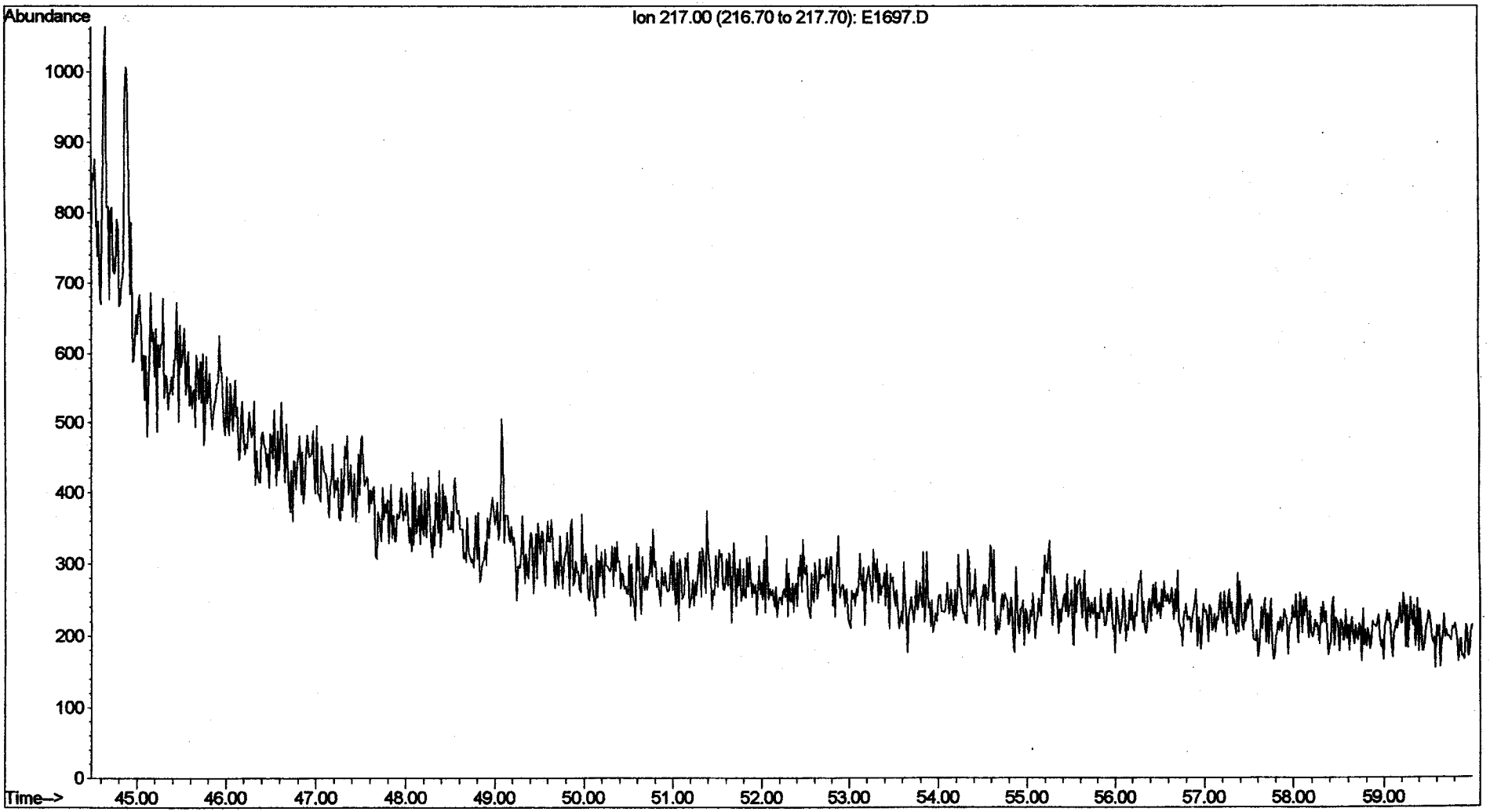


Appendix 9

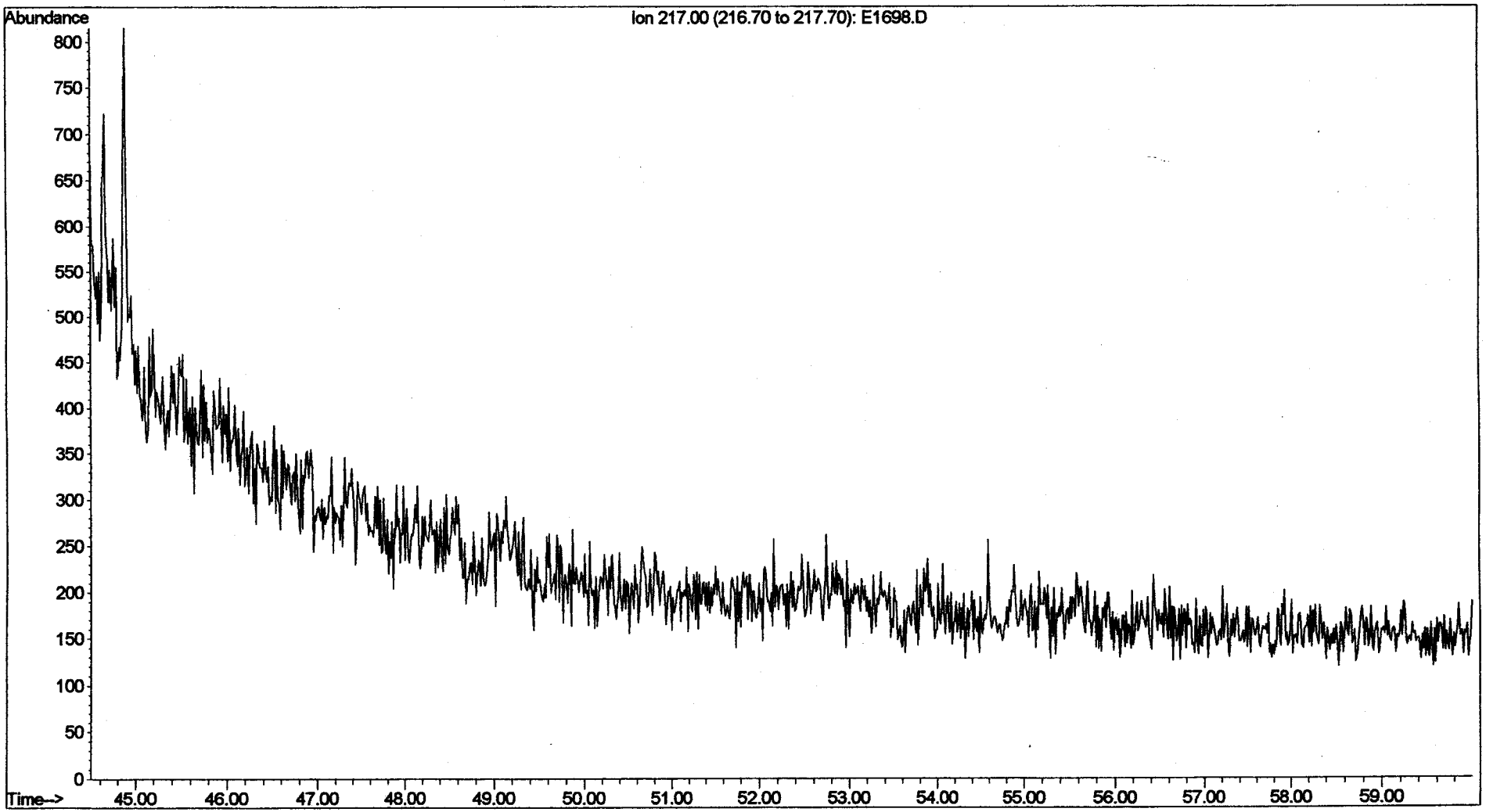
Biomarker Fingerprints:

Steranes

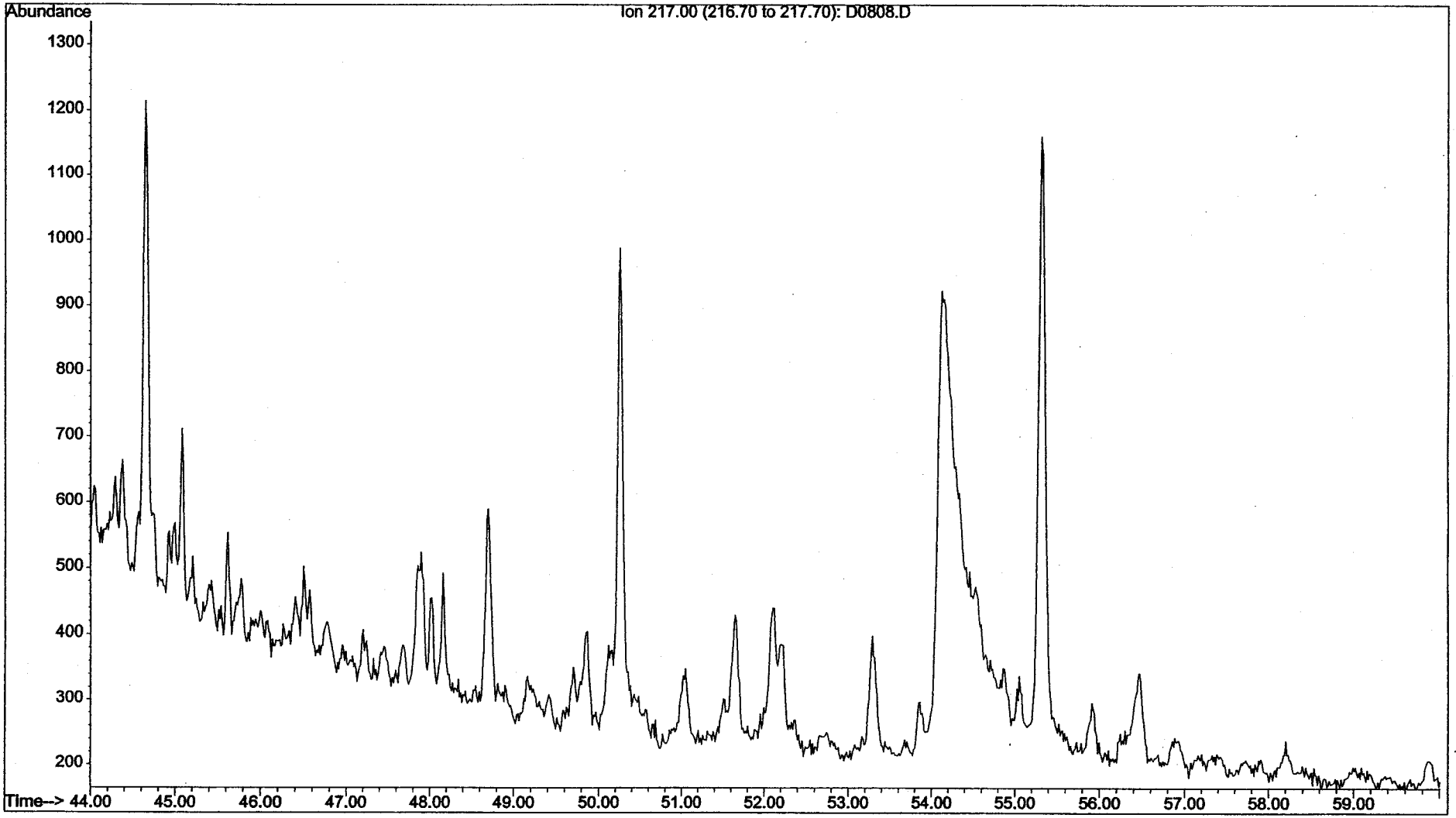
File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQE046\E1697
Operator : AC
Acquired : 23 Nov 2001 1:55 am using AcqMethod AQMETH6D
Instrument : GCMS-5
Sample Name: w9071
Misc Info : 2 Inch Steel Pipe
Vial Number: 26



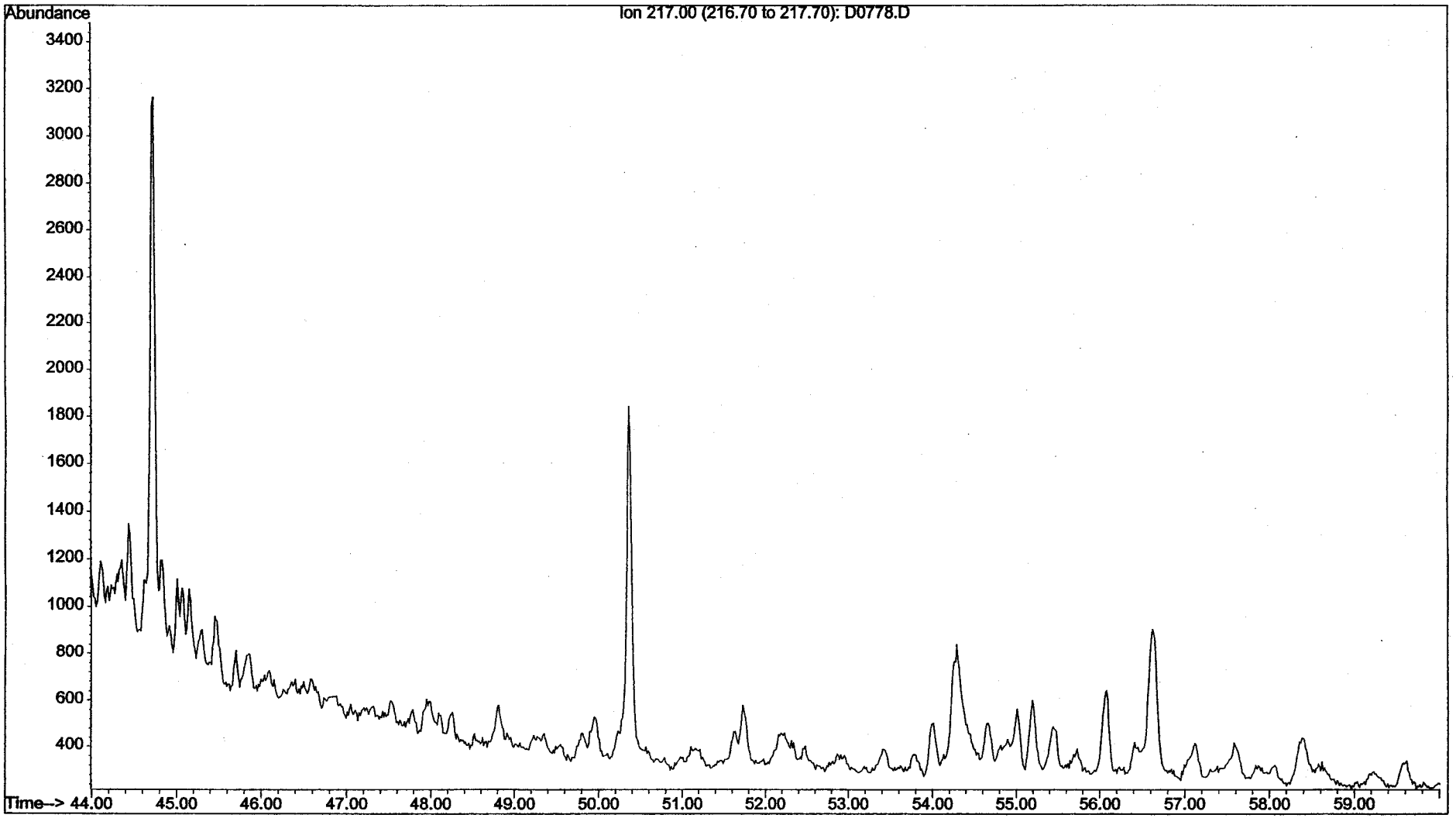
File : C:\ACTIVE\SM2000\PROJECTS\WDNRAS~1\BATTEL~1\PAHGCM~1\SQE046\E1698
Operator : AC
Acquired : 23 Nov 2001 3:44 am using AcqMethod AQMETH6D
Instrument : GCMS-5
Sample Name: w9072
Misc Info : 12 Inch Steel Pipe
Vial Number: 27



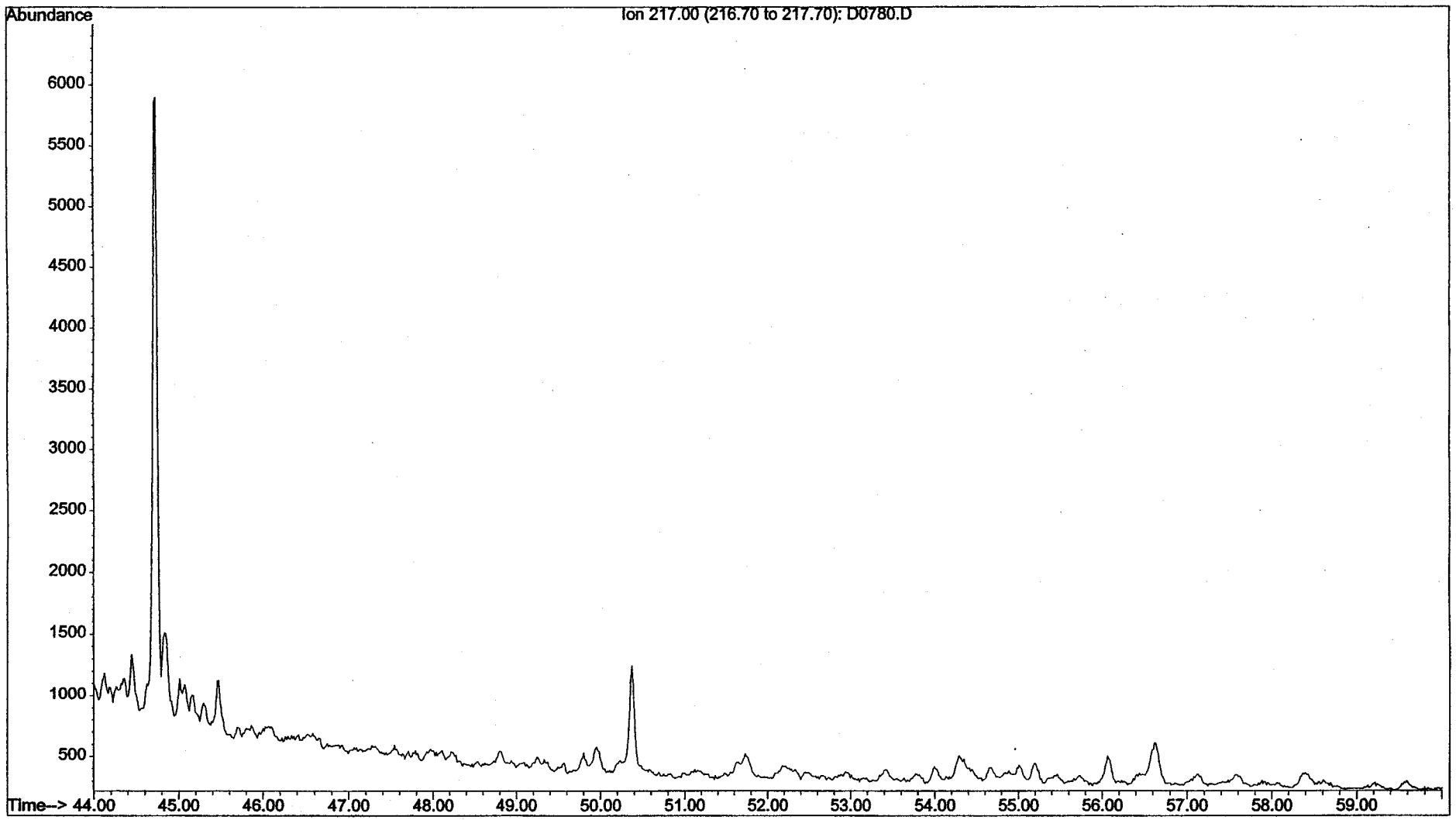
File : I:\D\DATA\SQD295\D0808.D
Operator : SA
Acquired : 25 Oct 2001 12:39 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5548-1
Misc Info : TW-13
Vial Number: 19



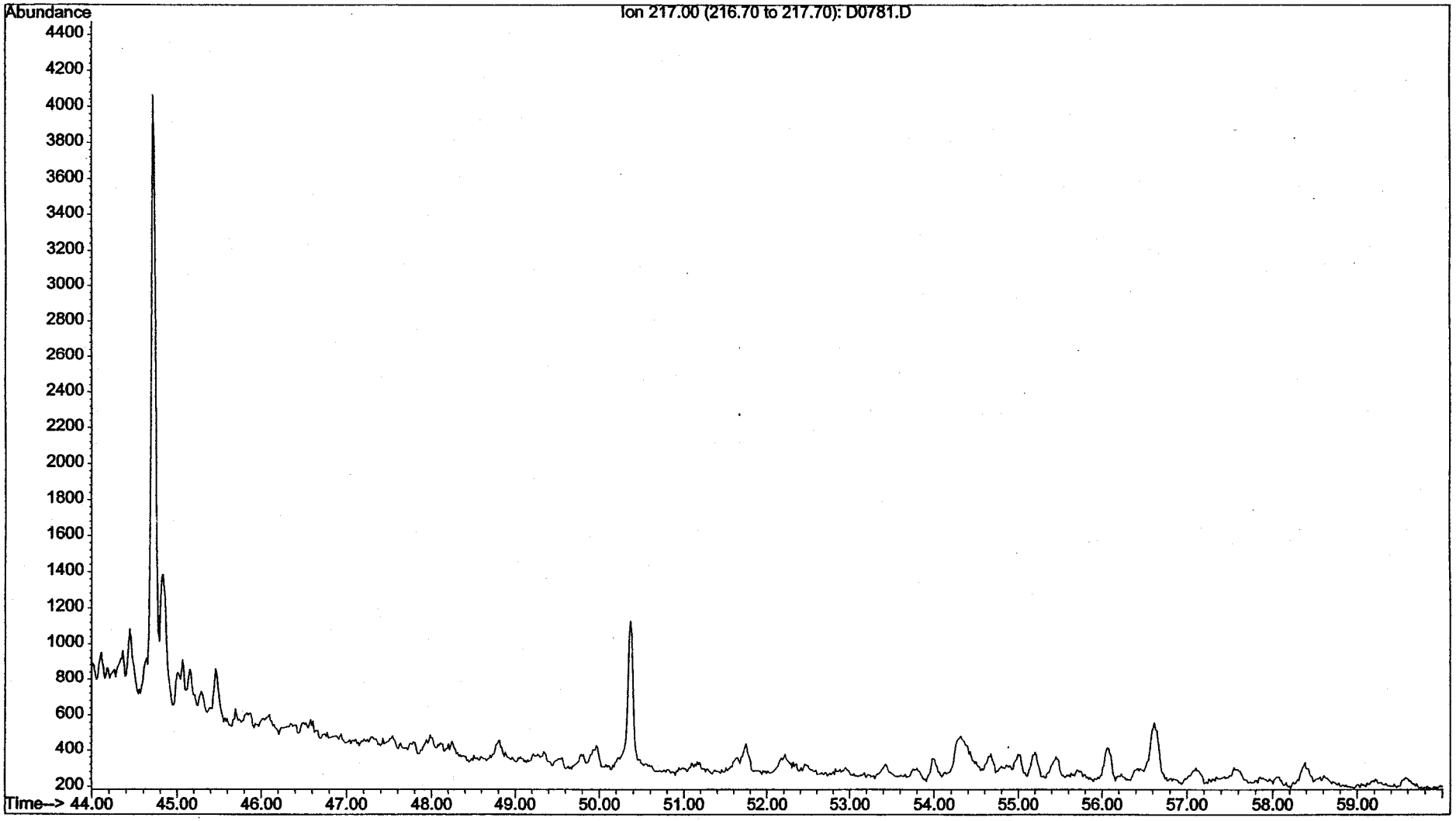
File : I:\D\DATA\SQD294\D0778.D
Operator : SA
Acquired : 22 Oct 2001 9:21 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W6841
Misc Info : Pipe Discharge East Yard Gate
Vial Number: 4



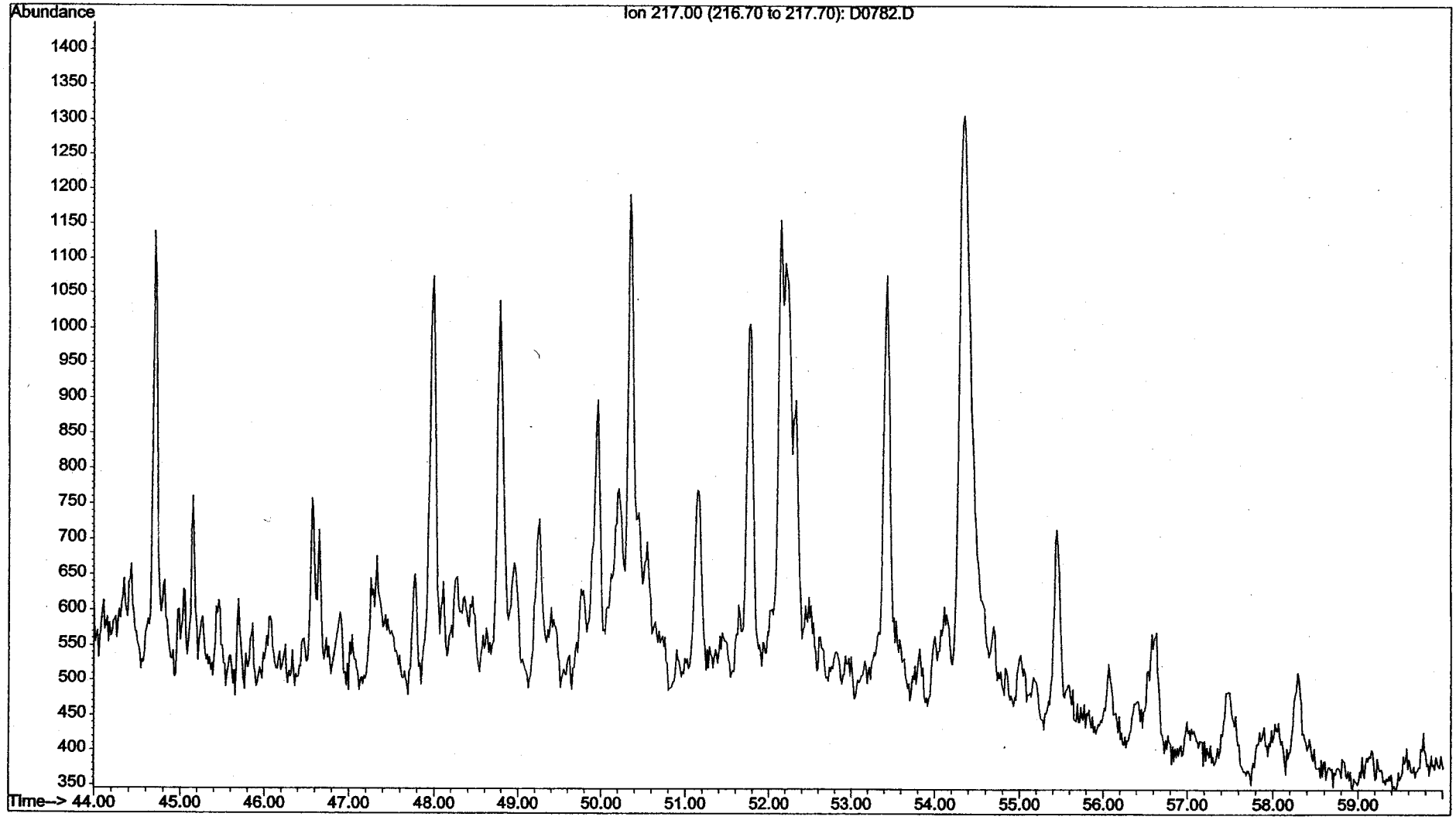
File : I:\D\DATA\SQD294\D0780.D
Operator : SA
Acquired : 23 Oct 2001 12:33 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W6839
Misc Info : Pipe Sludge East Yard Gate #1
Vial Number: 6



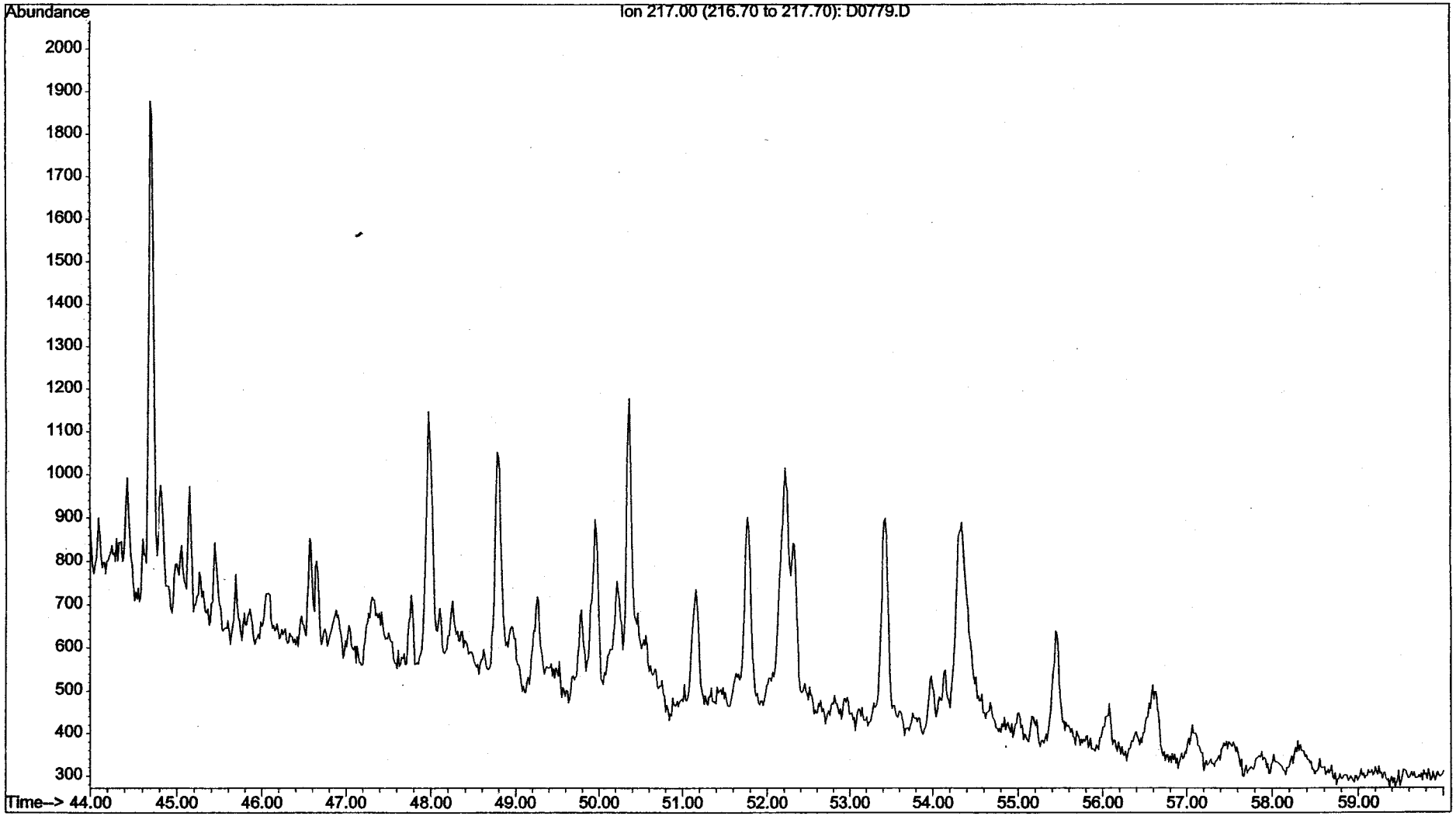
File : I:\D\DATA\SQD294\D0781.D
Operator : SA
Acquired : 23 Oct 2001 2:06 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W6840
Misc Info : Pipe Sludge East Yard Gate #2
Vial Number: 7



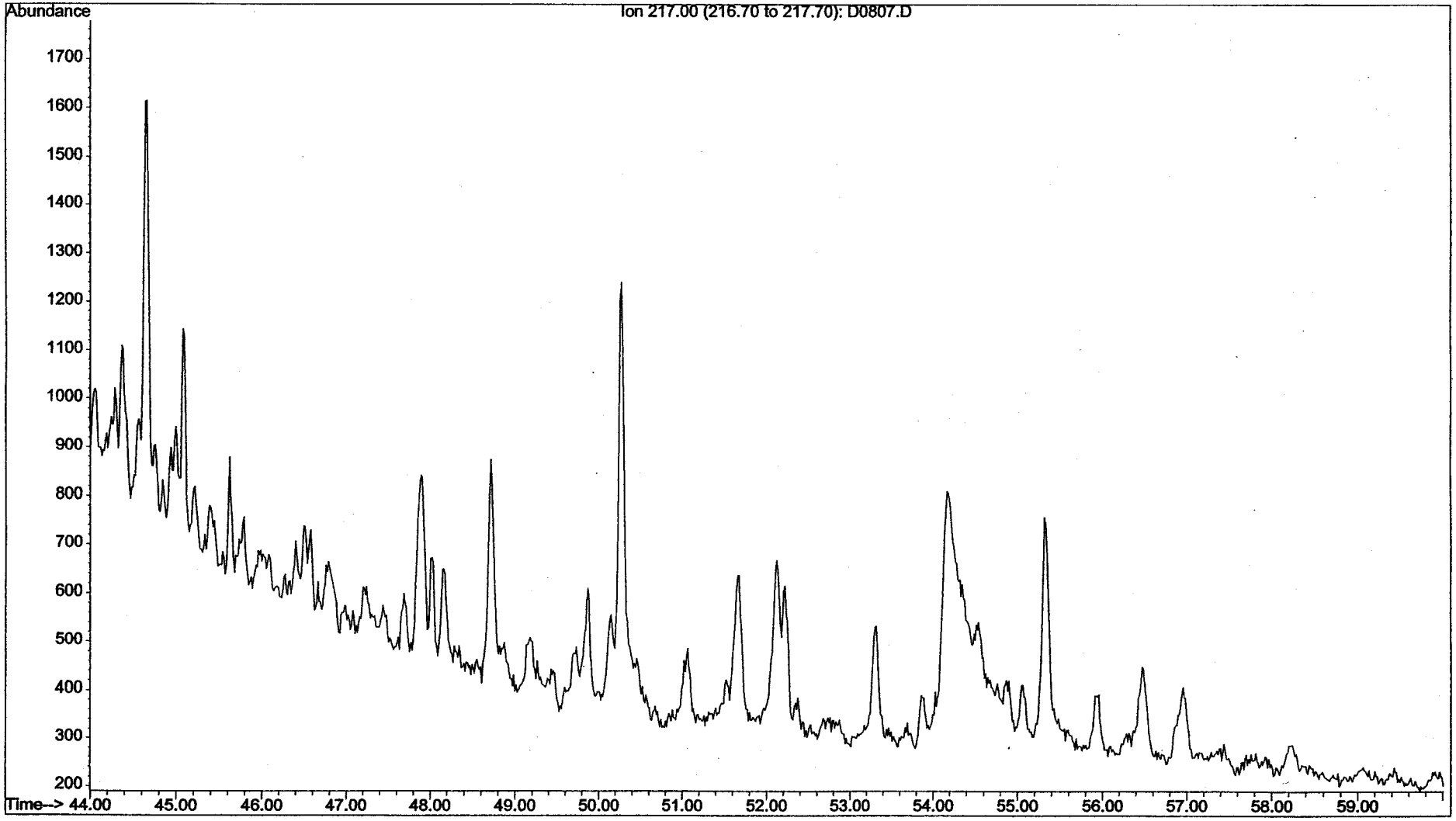
File : I:\D\DATA\SQD294\D0782.D
Operator : SA
Acquired : 23 Oct 2001 3:40 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W6837
Misc Info : Pipe Discharge Center Of Yard
Vial Number: 8



File : I:\D\DATA\SQD294\D0779.D
Operator : SA
Acquired : 22 Oct 2001 10:56 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W6838
Misc Info : Pipe Sludge Center of Yard
Vial Number: 5

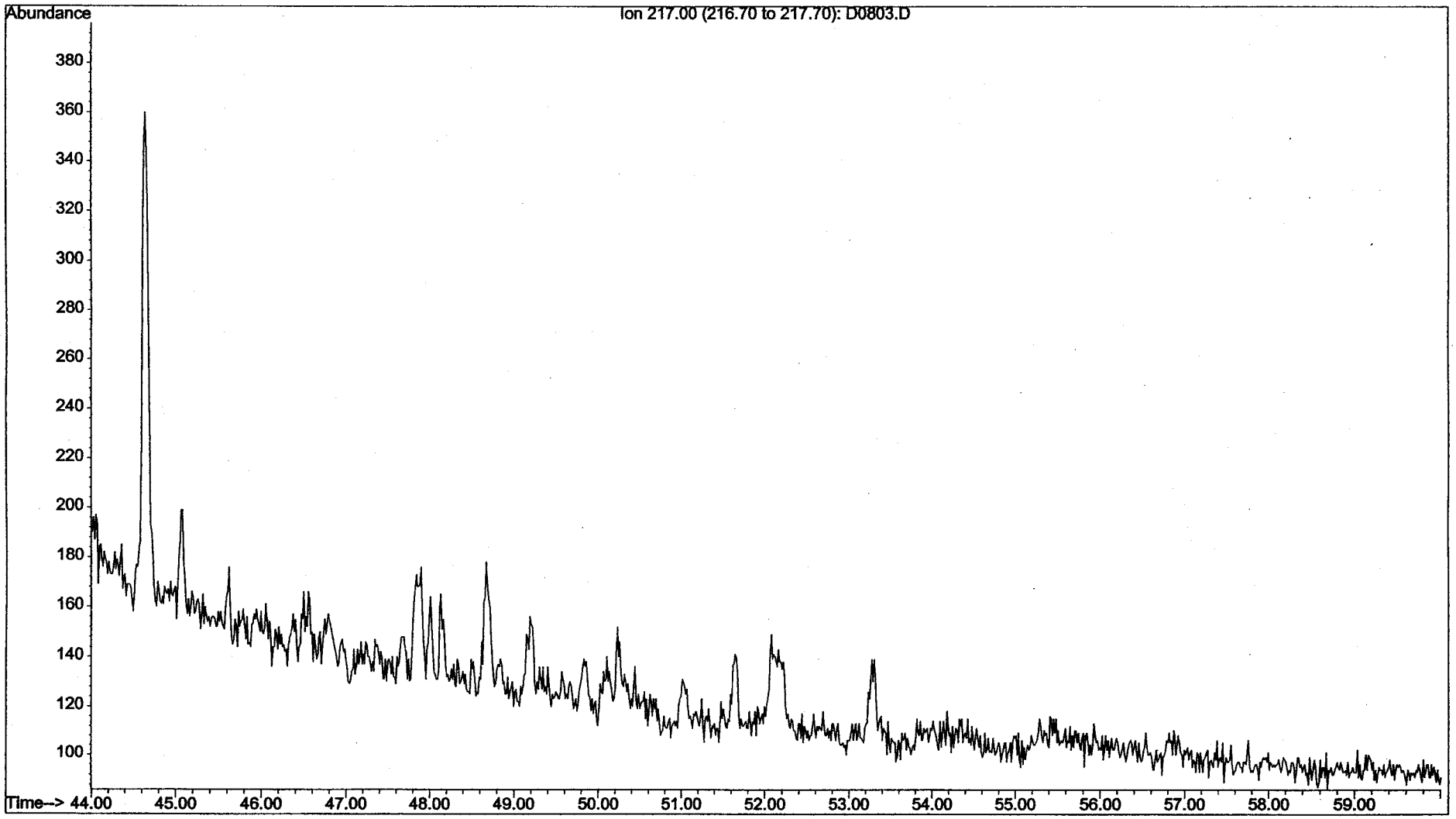


File : I:\D\DATA\SQD295\D0807.D
Operator : SA
Acquired : 25 Oct 2001 11:01 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5547-1
Misc Info : Upgradient Riser
Vial Number: 18

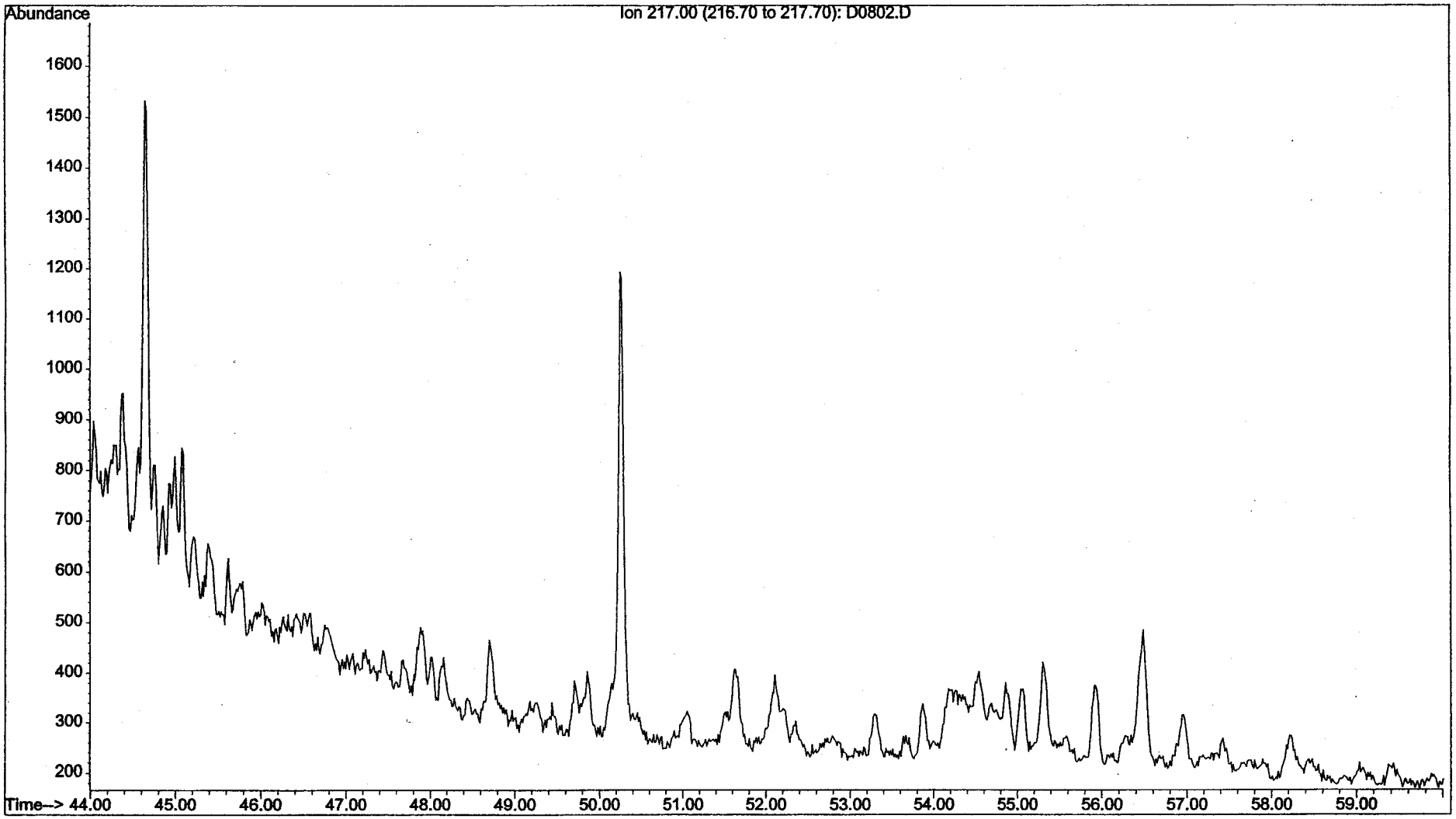


00324

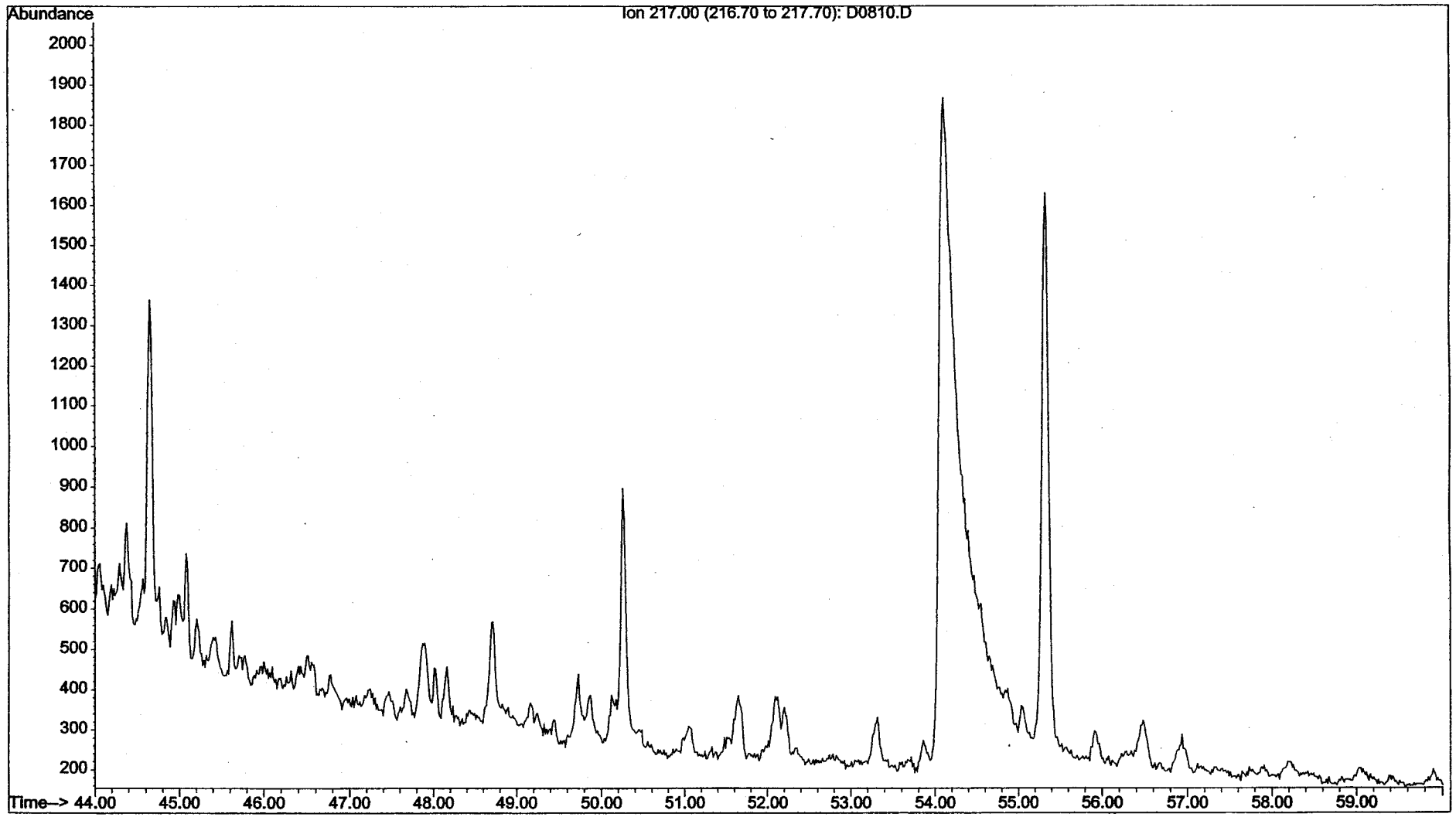
File : I:\D\DATA\SQD295\D0803.D
Operator : SA
Acquired : 25 Oct 2001 4:14 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5544-1
Misc Info : East Riser
Vial Number: 14



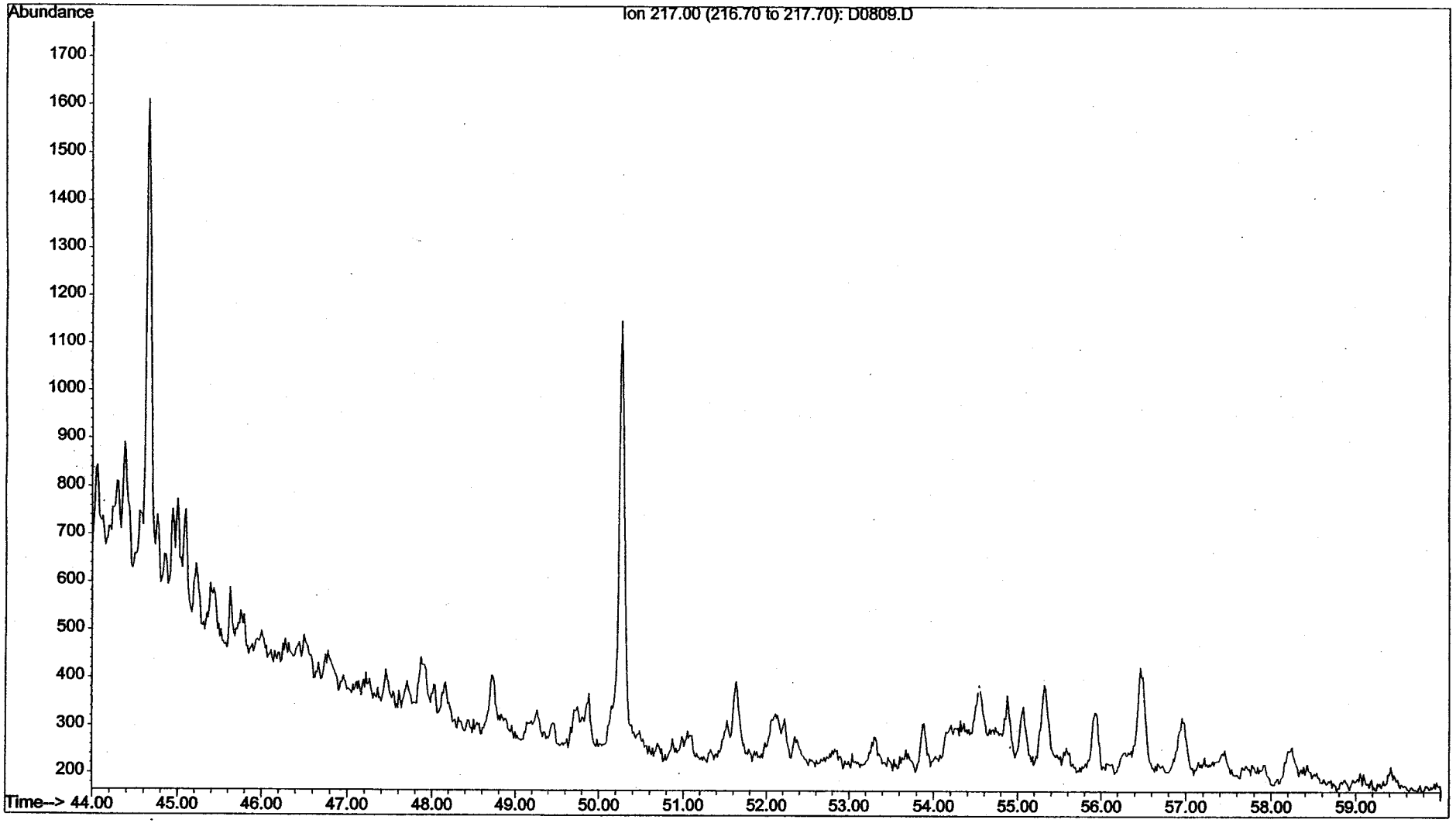
File : I:\D\DATA\SQD295\D0802.D
Operator : SA
Acquired : 25 Oct 2001 2:32 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5543-1
Misc Info : West Risen
Vial Number: 13



File : I:\D\DATA\SQD295\D0810.D
Operator : SA
Acquired : 25 Oct 2001 4:01 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5551-1
Misc Info : Scrapings from inside discharge
Vial Number: 21

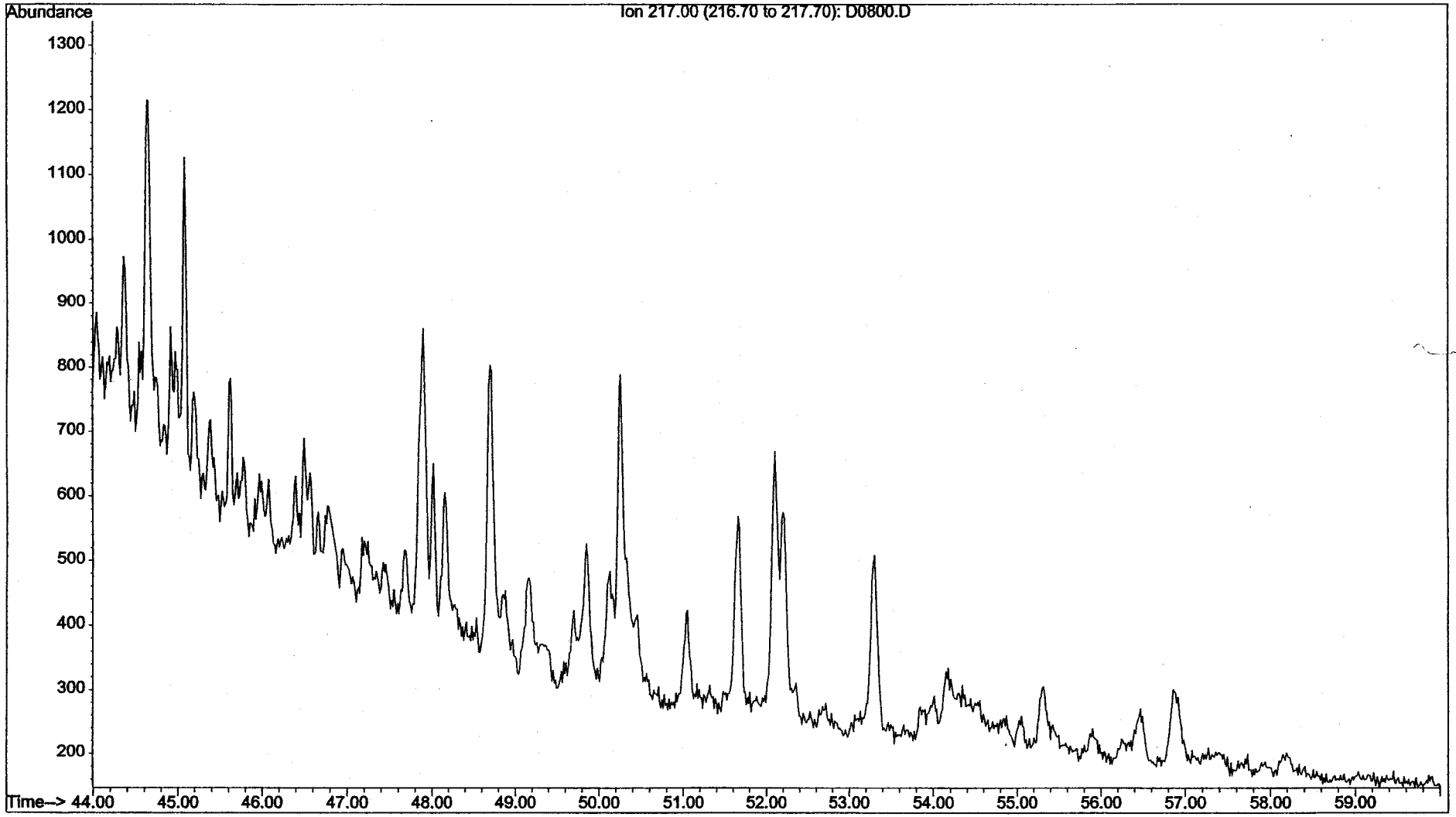


File : I:\D\DATA\SQD295\D0809.D
Operator : SA
Acquired : 25 Oct 2001 2:18 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5549-1
Misc Info : Pipe Discharge
Vial Number: 20

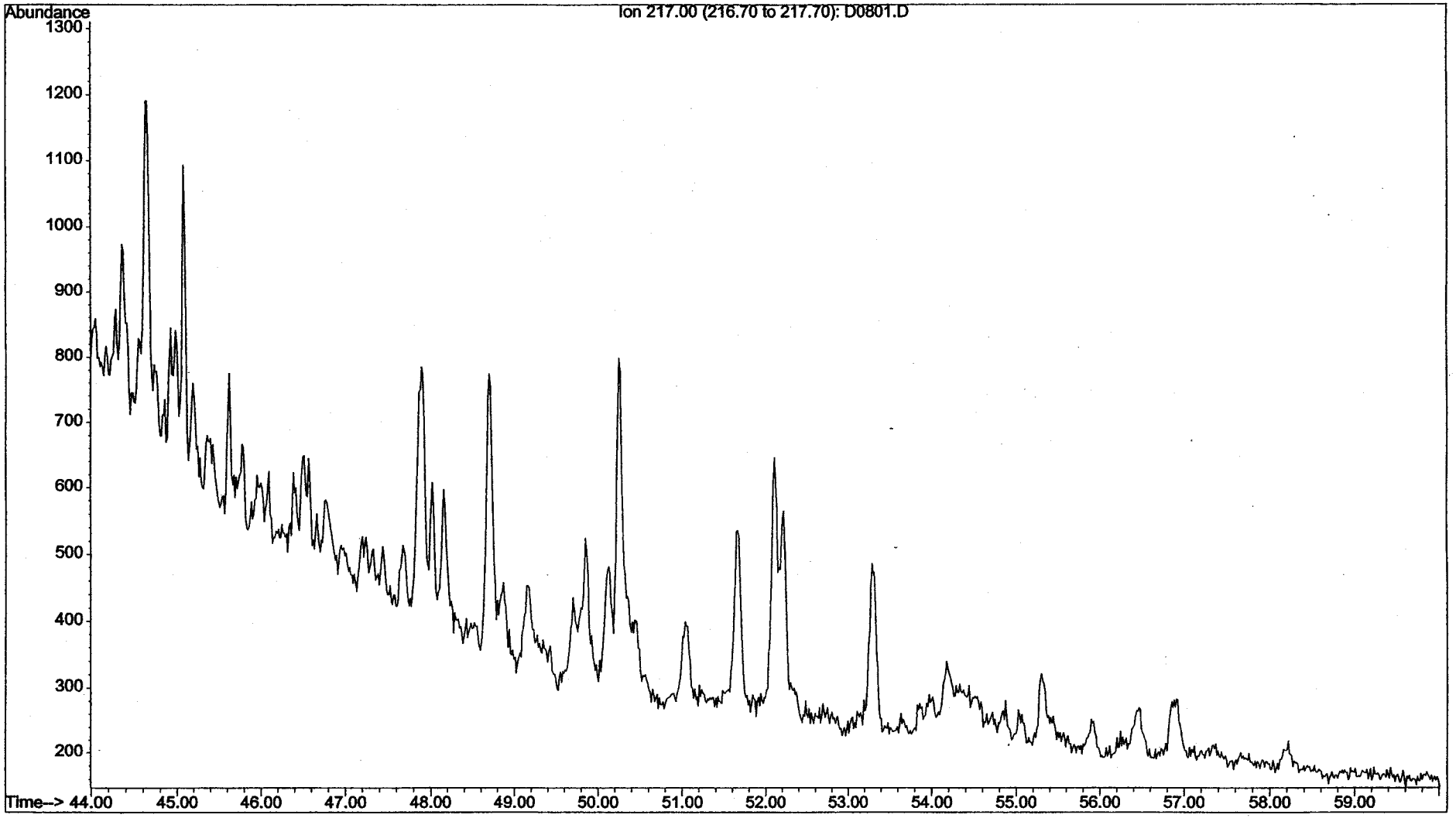


00326

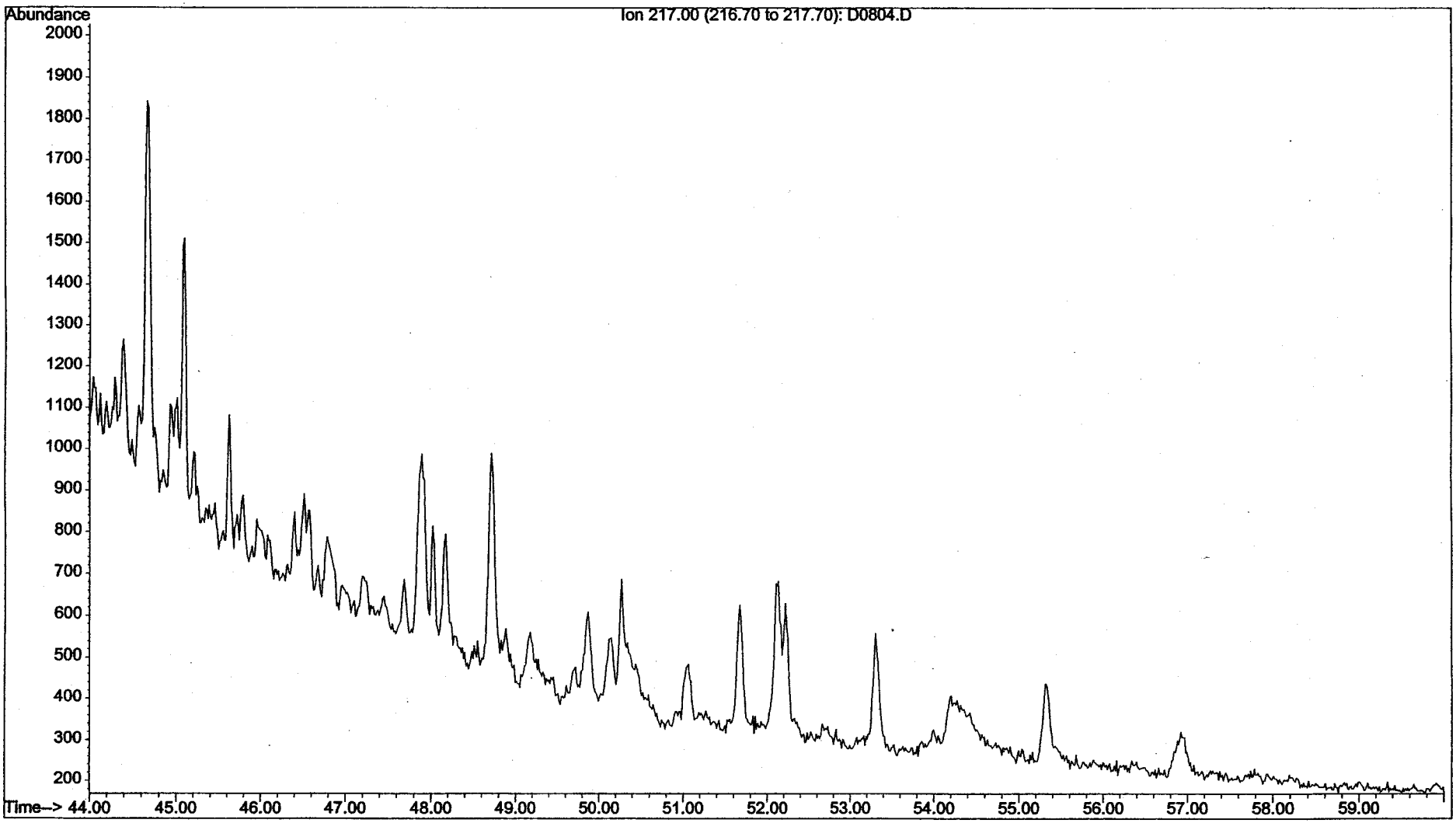
File : I:\D\DATA\SQD295\D0800.D
Operator : SA
Acquired : 24 Oct 2001 11:23 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5550-1
Misc Info : Solids around discharge pipe
Vial Number: 11



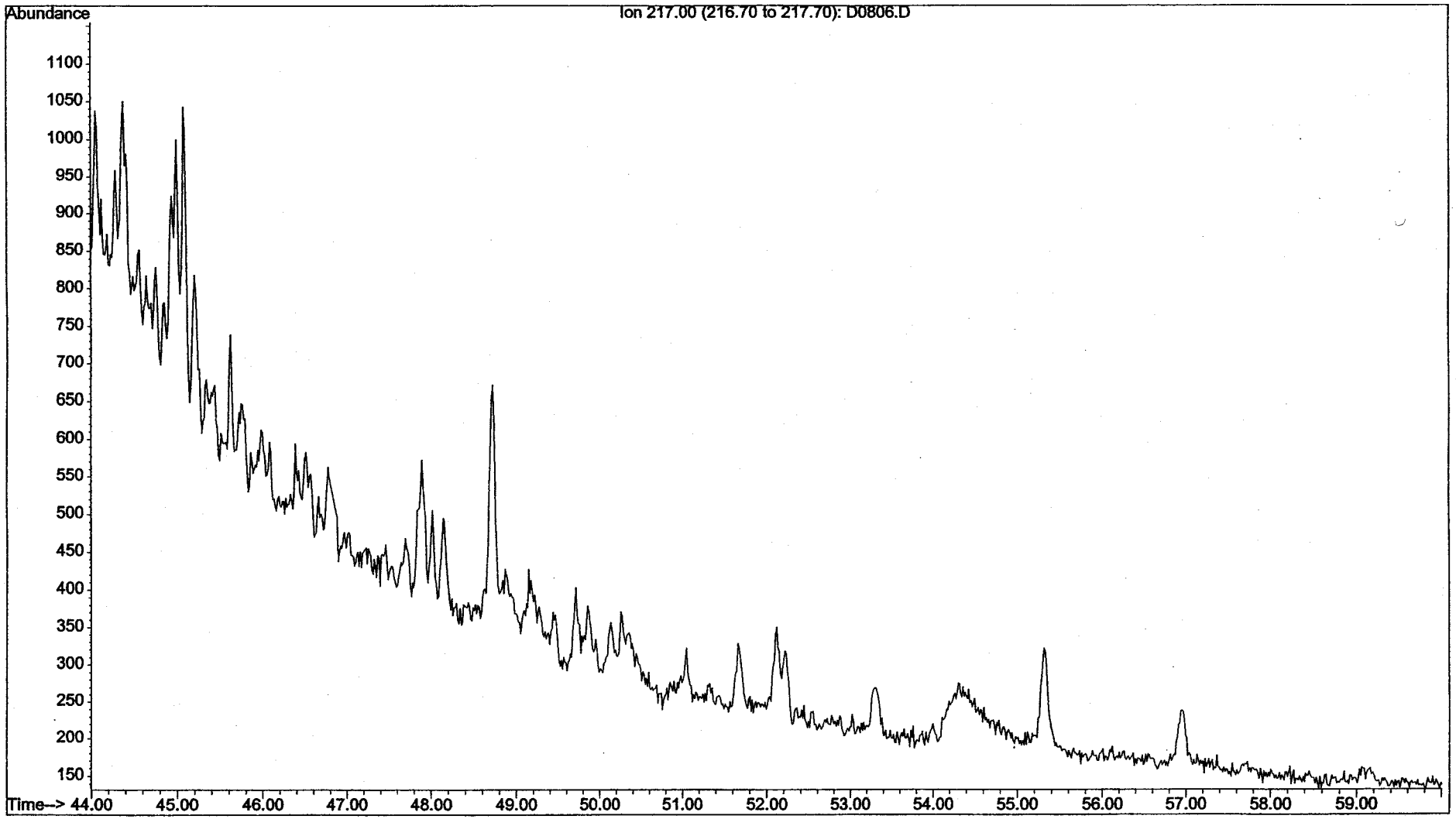
File : I:\D\DATA\SQD295\D0801.D
Operator : SA
Acquired : 25 Oct 2001 12:57 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5550DUP-1
Misc Info : Solids around discharge pipe
Vial Number: 12



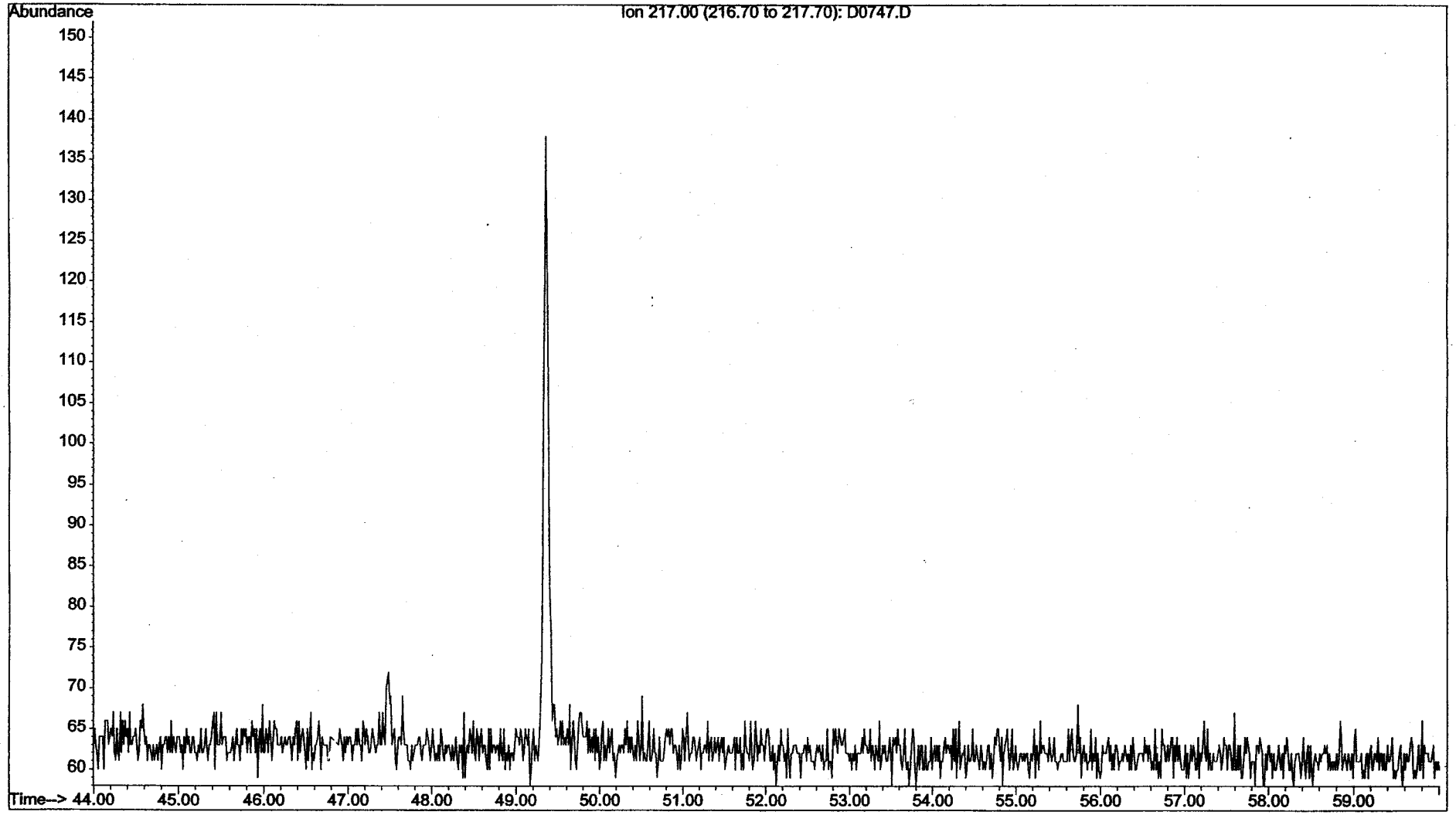
File : I:\D\DATA\SQD295\D0804.D
Operator : SA
Acquired : 25 Oct 2001 5:59 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5545-1
Misc Info : MW-7
Vial Number: 15



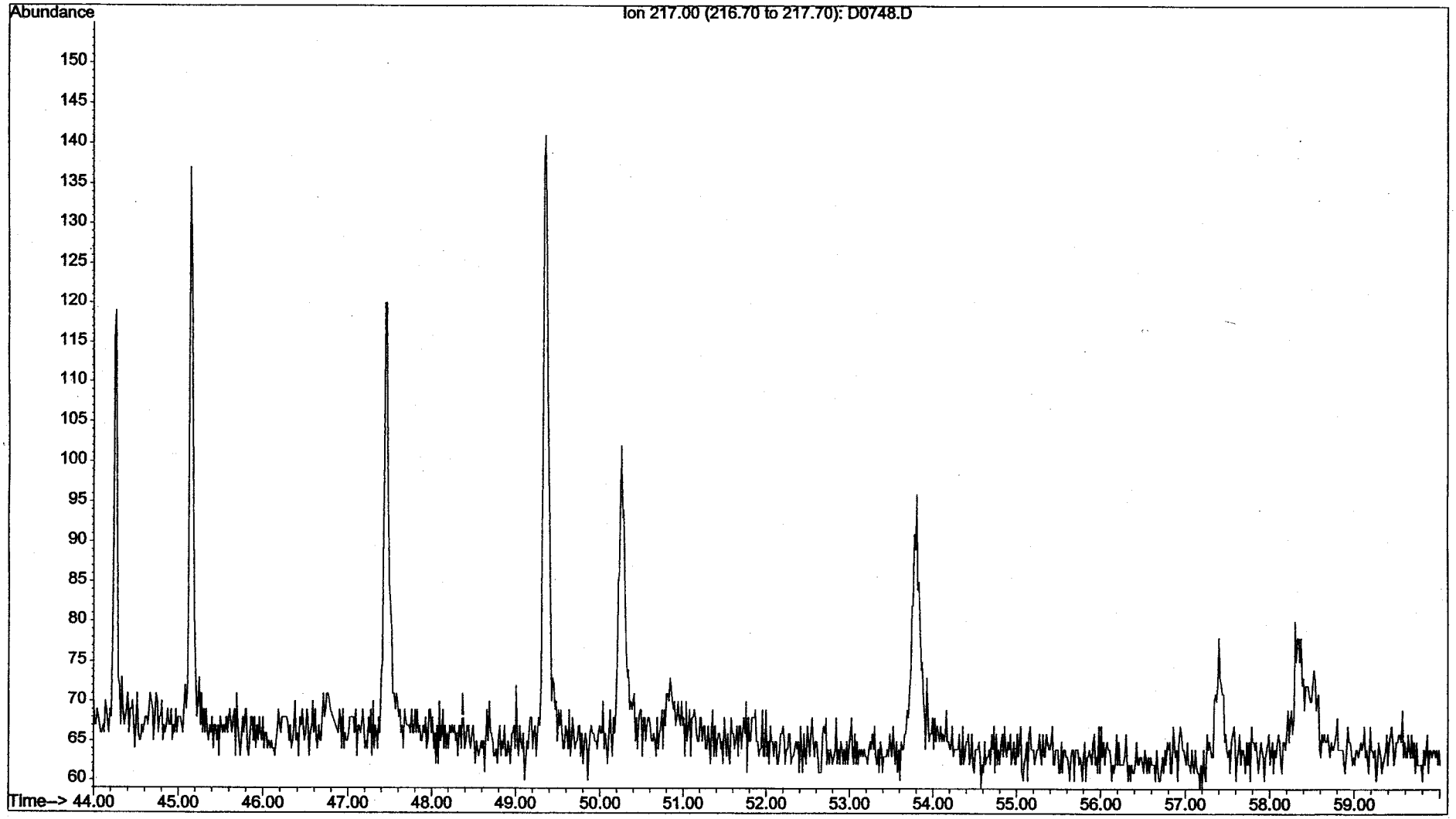
File : I:\D\DATA\SQD295\D0806.D
Operator : SA
Acquired : 25 Oct 2001 9:19 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: W5546-1
Misc Info : TW-9
Vial Number: 17



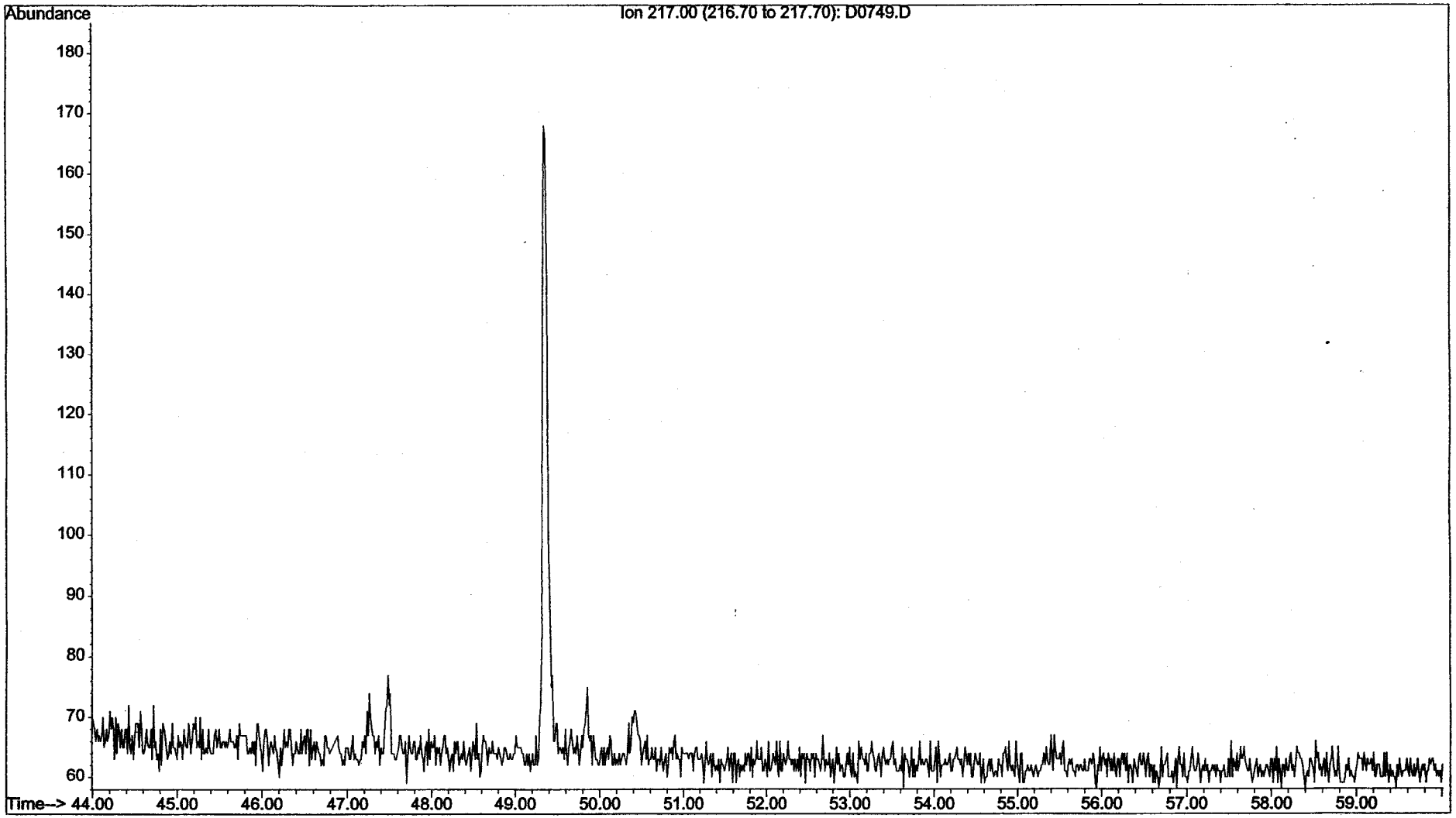
File : I:\D\DATA\SQD292\D0747.D
Operator : SA
Acquired : 16 Oct 2001 8:56 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: ZK90PB
Misc Info : Procedural Blank
Vial Number: 10



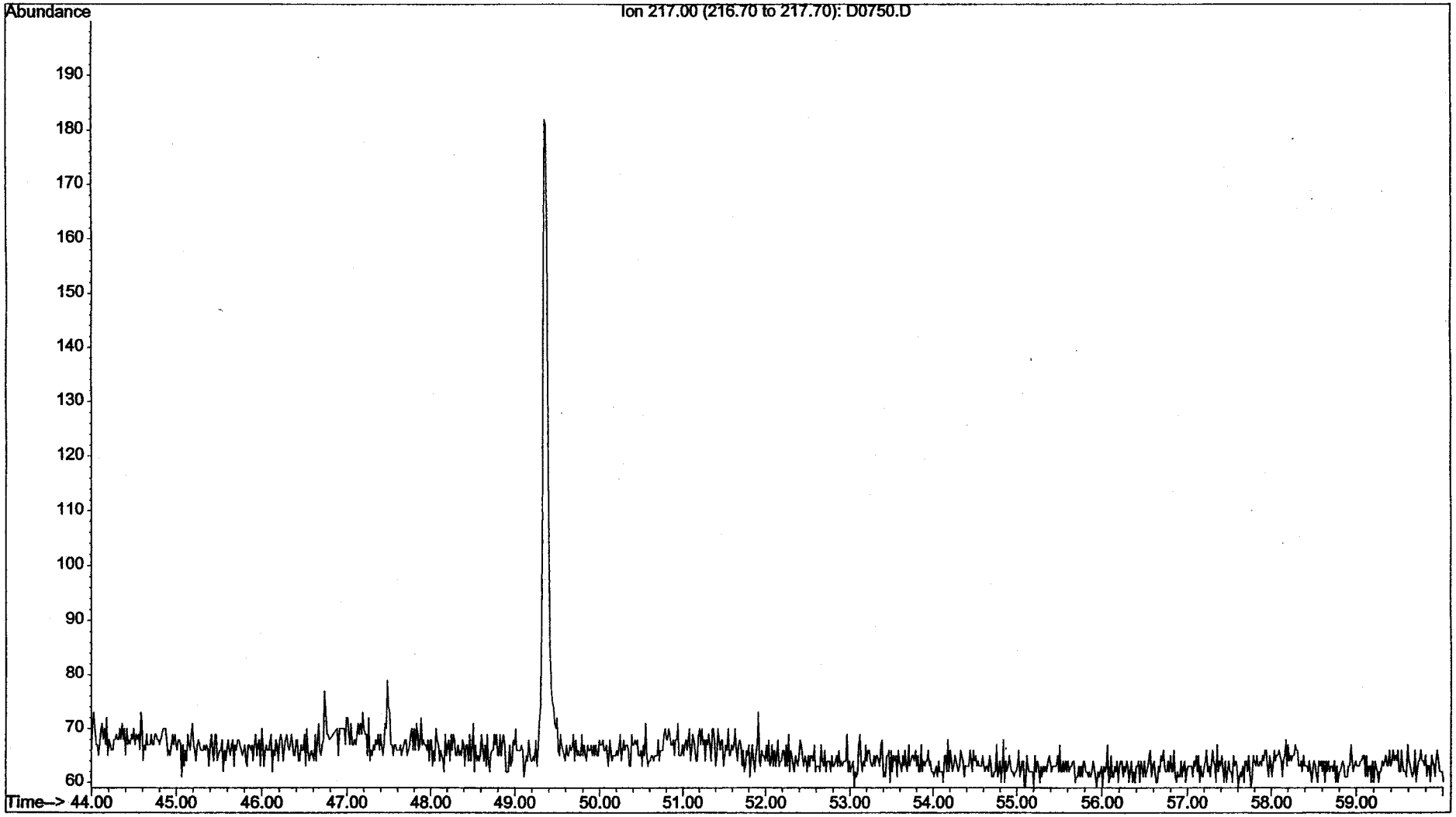
File : I:\D\DATA\SQD292\D0748.D
Operator : SA
Acquired : 16 Oct 2001 10:28 am using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: ZK91LCS
Misc Info : Laboratory Control Spike
Vial Number: 11



File : I:\D\DATA\SQD292\D0749.D
Operator : SA
Acquired : 16 Oct 2001 12:01 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: ZL25PB
Misc Info : Procedural Blank
Vial Number: 12



File : I:\D\DATA\SQD292\D0750.D
Operator : SA
Acquired : 16 Oct 2001 1:35 pm using AcqMethod SINTEF1C
Instrument : GC/MS Ins
Sample Name: ZL26LCS
Misc Info : Laboratory Control Spike
Vial Number: 13



Attachment 10

Chain of Custody

ATTACHMENT 4a
Battelle Duxbury Operations
Sample Receipt Form

Project Number: _____ Client: SEA
Received by: JDH Date/Time Received: 7/27/01 10:45AM IR 8/8/01
No. of Shipping Containers 1

SHIPMENT

Method of Delivery: FED Ex Commercial Carrier (Air bill No. 827065712101)
_____ Hand Delivered
_____ US Mail (RPS No. _____)

COC Forms: ✓ Shipped with samples _____ No forms

Cooler(s)\Box(es) were sealed with: ✓ Tape _____ Custody Seals _____ (Other specify)
Were the seals intact for each shipping container? ✓ Yes _____ No _____ NA
If NO, see Sample Custody Corrective Action Form

SAMPLES

Sample Labels: ✓ Sample labels agree with COC forms
_____ Discrepancies (see Sample Custody Corrective Action Form)*

Container Seals: ✓ Tape _____ Custody Seals _____ (Other specify)
✓ Seals intact for each shipping container
_____ Seal broken (list impacted samples):

Condition of Samples: ✓ Sample containers intact
_____ Sample containers broken/leaking (see Sample Custody Corrective Action Form)*

IR 8/8/01

Temperature upon receipt (°C): Below 4° C Temperature blank used _____ Yes ✓ No
(Note: If temperature upon receipt differs from required conditions, list impacted samples):

Samples Acidified? ___ Yes ✓ No ___ Unknown

Initial pH 5 - 9? (Y/N): NA *If no, individual sample adjustments on the Auxiliary Sample Receipt Form.*

Total Residual Chlorine Present? (water) (Y/N): NA
If yes, individual sample adjustments on the Auxiliary Sample Receipt Form.

Head Space <1% in samples for water VOC analysis NA Yes _____ No
Individual sample deviations listed below.

Sample Containers:
Samples returned in PC-grade jars? ___ Yes ✓ No ___ Unknown / Lot No. _____

Storage Location: FIRST FLOOR WET ROOM BDO IDs Assigned: WS543 - WS551

Holding Times:	Water	Sediment	Tissue

Additional Comments:

Samples logged in by: [signature] Date/Time: 7/27/01 1:00PM IR 8/8/01
* Must also be noted on the C-O-C.

Battelle
. . . Putting Technology To Work

Chain of Custody

Proj. No	Proj. Name
WIONR 9401.07	Ashland NSP Site

SAMPLERS: Signature: Dave [signature]

ANALYSIS REQUESTED → "NUMBER OF CONTAINERS"

DATE	TIME	BATTELLE ID	CLIENT ID	SAMPLE DESCRIPTION	PEST	PCB	TPH FINGERPRINT	PAH	VOA	TBT	METALS NAPLC	OTHER Nappe-Fingerprinting	ACIDIFIED	PRESERVED	Total Number of Containers
7/24/01	1:40 pm	W5543		West Riser	*Liquid							✓			
	1:50 pm	W5544		East Riser								✓			
	2:00 pm	W5545		MW-7								✓			
	2:15 pm	W5546		TW-9								✓			
	2:30 pm	W5547		Upgradient Riser								✓			
	1:30 pm	W5548		TW-13								✓			
7/25/01	3:30 pm	W5549		Pipe Discharge								✓			
	4:00 pm	W5550		Solids around Discharge Pipe	*Solid							✓			
	4:20 pm	W5551		Scrapings from Inside Discharge Pipe	*Solid							✓			

Relinquished by:	Date/Time		Received by:	Date/Time	
Dave [signature]	7/26/01	11:00	[signature]	7/27/01	10:45 am
Relinquished by:	Date/Time		Received by:	Date/Time	

Comments:
Please call w/any questions or comments.
Cy Ingraham 715.720.6231 or Dave Etheridge 715.720.6208

0109

LAB COPY

BATTELLE Laboratory Sample Login Report

Project # : N004602-0002 *Receive Date :* 7/27/01 10:45:00 AM

Client : SEH *Logged in By :* JHATCH

Collection Date	Login Date	Lab ID	Client Sample ID	#Containers	Matrix	Preservative	Storage Location	Login Comments
7/24/01 1:40:00 PM	7/27/01 10:38:41 AM	W5543	WEST RISER	3	WATER	NONE	FIRST FLOOR COLD ROOM	
7/24/01 1:50:00 PM	7/27/01 10:58:37 AM	W5544	EAST RISER	3	WATER	NONE	FIRST FLOOR COLD ROOM	
7/24/01 2:00:00 PM	7/27/01 11:01:48 AM	W5545	MW-7	3	WATER	NONE	FIRST FLOOR COLD ROOM	
7/24/01 2:15:00 PM	7/27/01 11:12:50 AM	W5546	TW-9	3	WATER	NONE	FIRST FLOOR COLD ROOM	
7/24/01 2:30:00 PM	7/27/01 11:17:47 AM	W5547	UPGRADIENT RISER	3	WATER	NONE	FIRST FLOOR COLD ROOM	
7/24/01 1:30:00 PM	7/27/01 11:22:14 AM	W5548	TW-13	3	WATER	NONE	FIRST FLOOR COLD ROOM	
7/25/01 3:30:00 PM	7/27/01 11:31:12 AM	W5549	PIPE DISCHARGE	3	WATER	NONE	FIRST FLOOR COLD ROOM	
7/25/01 4:00:00 PM	7/27/01 11:50:02 AM	W5550	SOLIDS AROUND DISCH	1	SOIL	NONE	FIRST FLOOR COLD ROOM	
7/25/01 4:20:00 PM	7/27/01 1:01:34 PM	W5551	SCRAPING FROM INSID	1	SOIL	NONE	FIRST FLOOR COLD ROOM	

ATTACHMENT 4a
Battelle Duxbury Operations
Sample Receipt Form

Project Number: _____ Client: ASHLAND
Received by: [illegible] Date/Time Received: 9/21/01 12:00PM
No. of Shipping Containers 1

SHIPMENT

Method of Delivery: FED EX Commercial Carrier (Air bill No. 8261868605)
_____ Hand Delivered
_____ US Mail (RPS No. _____)

COC Forms: ✓ Shipped with samples _____ No forms

Cooler(s)\Box(es) were sealed with: ✓ Tape _____ Custody Seals _____ (Other specify)
Were the seals intact for each shipping container? ✓ Yes _____ No _____ NA
If NO, see Sample Custody Corrective Action Form

SAMPLES

Sample Labels: ✓ Sample labels agree with COC forms
_____ Discrepancies (see Sample Custody Corrective Action Form)*

Container Seals: ✓ Tape _____ Custody Seals _____ (Other specify)
✓ Seals intact for each shipping container
_____ Seal broken (list impacted samples):

Condition of Samples: ✓ Sample containers intact
_____ Sample containers broken/leaking (see Sample Custody Corrective Action Form)*

Temperature upon receipt (°C): 1.3°C Temperature blank used _____ Yes ✓ No
(Note: If temperature upon receipt differs from required conditions, list impacted samples):

Samples Acidified? ___ Yes ✓ No ___ Unknown

Initial pH 5 - 9? (Y/N): NA *If no, individual sample adjustments on the Auxiliary Sample Receipt Form.*

Total Residual Chlorine Present? (water) (Y/N): NA
If yes, individual sample adjustments on the Auxiliary Sample Receipt Form.

Head Space <1% in samples for water VOC analysis NA Yes _____ No
Individual sample deviations listed below.

Sample Containers:
Samples returned in PC-grade jars? ___ Yes ✓ No ___ Unknown / Lot No. _____

Storage Location: FIRST FLOOR COLD ROOM BDO IDs Assigned: W6837-W6841

Holding Times:	Water	Sediment	Tissue

Additional Comments:

Samples logged in by: [signature] Date/Time: 9/21/01 3:00PM
* Must also be noted on the C-O-C.

State of Wisconsin
Department of Natural Resources

EN CHEM INC.

CHAIN OF CUSTODY RECORD
~~LUST PROGRAM~~
Based on Form 4400-151 Rev. 4-93

Note: Use of this form is voluntary but is requested by the Department pursuant to ch. NR 149, NR 500-540, NR 158 and NR 419, Wis.Adm.Code. Personally identifiable information will be used for no other purpose.

Sample Collector(s): John Guhl	Title/Work Station/Company: Short Elliott Hendrickson Inc.	Telephone Number (include area code): 715.720.6225
~~Property Owner~~ Client: WDNR	Property Address: Ashland - Xcel Energy Storage Yard	Telephone Number (include area code):

I hereby certify that I received, properly handled and disposed of these samples as noted below:

Relinquished By (Signature): [signature]	Date/Time: 9/20/01 4:00 pm	Received By (Signature): [signature] 9/21/01 12:00
Relinquished By (Signature):	Date/Time:	Received By (Signature):
Relinquished By (Signature):	Date/Time:	Received for EN CHEM by (Signature):

LABORATORY USE ONLY

Temperature of temperature blank:
If samples were received on ice and there was ice remaining, you may report the temperature as received on ice. If all of the ice was melted, the temperature of the melt may be substituted for a temperature blank.

Field ID Number	Date Collected	Time Collected	Sample Type 1	Device	Preserv. Type	Field Screening	Location/Description (see footnote 2)	Analysis Type	Lab ID Number	No/Type of Containers	Cracked /broken	Improp. Sealed	Good Cond.	Other Comments
Pipe Discharge Center of Yard	9-19-01	1:00 pm			None	—	Pipe Discharge (water - NAPL) Center of Storage Yard	Fingerprint	W6837					
Pipe Sludge Center of Yard	9-19-01	1:00 pm			None	—	Pipe Sludge - Center of Yard	"	W6838					
Pipe Sludge East Yard Gate #1	9-20-01	9:00 am			None	—	Pipe Sludge - East Yard Gate #1	"	W6839					
Pipe Sludge East Yard Gate #2	9-20-01	11:00 am			None	—	Pipe Sludge - East Yard Gate #2	"	W6840					
Pipe Discharge East Yard Gate	9-20-01	11:00 am			None	—	Pipe Discharge - East Yard Gate	"	W6841					

FOOTNOTES
1. specify groundwater, surface water, soil, leachate, sludge, etc.
2. sample description must clearly correlate the sample ID to the sampling location.

QTA#: _____ En Chem Project#: _____

ANALYSIS CODES

1. GRO	5. DRO	9. Free Liquids	13. BETX
2. PVOC	6. PAH	10. pH	14. Protocol D1-GRO
3. Lead	7. Flashpoint	11. TCLP-Benzene	15. Protocol D1-DRO
4. 8021	8. Percent Solids	12. TCLP-Lead	16. 8260

BILLING ADDRESS:
Wisconsin Department of Nat. Resources
810 W. Maple
Spooner, WI
Attn: Jamie Dunn

Job Name/Number: Ashland / NSP Coal Gas Site
Job Description: Clay Pipe Investigation - Xcel Energy Storage Yard

012

BATTELLE Laboratory Sample Login Report

Project # : PENDING

Client : ASHLAND

Receive Date : 9/21/01 12:00:00 PM

Logged in By : JHATCH

Validated 9/26/01 RB

Collection Date	Login Date	Lab ID	Client Sample ID	#Containers	Matrix	Preservative	Storage Location	Login Comments
9/19/01 1:00:00 PM	9/21/01 2:42:40 PM	W6837	PIPE DISCHARGE CENT	1	WATER	NONE	FIRST FLOOR COLD ROOM	
9/19/01 1:00:00 PM	9/21/01 2:56:06 PM	W6838	PIPE SLUDGE CENTER	1	WATER	NONE	FIRST FLOOR COLD ROOM	
9/20/01 9:00:00 AM	9/21/01 2:58:43 PM	W6839	PIPE SLUDGE EAST #1	1	WATER	NONE	FIRST FLOOR COLD ROOM	
9/20/01 11:00:00 AM	9/21/01 3:01:20 PM	W6840	PIPE SLUDGE EAST #2	1	WATER	NONE	FIRST FLOOR COLD ROOM	
9/20/01 11:00:00 AM	9/21/01 3:07:23 PM	W6841	PIPE DISCHARGE EAST	1	WATER	NONE	FIRST FLOOR COLD ROOM	

ATTACHMENT 4a
Battelle Duxbury Operations
Sample Receipt Form

Project Number: N004602-0002 Client: ASHLAND
Received by: JDH Date/Time Received: 11/14/01 10:20 AM
No. of Shipping Containers 1

SHIPMENT

Method of Delivery: FED EX Commercial Carrier (Air bill No. 826186860556)
____ Hand Delivered
____ US Mail (RPS No. ____)

COC Forms: ✓ Shipped with samples ____ No forms

Cooler(s)\Box(es) were sealed with: ✓ Tape ____ Custody Seals ____ (Other specify)
Were the seals intact for each shipping container? ✓ Yes ____ No ____ NA
If NO, see Sample Custody Corrective Action Form

SAMPLES

Sample Labels: ✓ Sample labels agree with COC forms
____ Discrepancies (see Sample Custody Corrective Action Form)*

Container Seals: ✓ Tape ____ Custody Seals ____ (Other specify)
✓ Seals intact for each shipping container
____ Seal broken (list impacted samples);

Condition of Samples: ✓ Sample containers intact
____ Sample containers broken/leaking (see Sample Custody Corrective Action Form)*

Temperature upon receipt (°C): AMBIENT Temperature blank used ____ Yes ✓ No
(Note: If temperature upon receipt differs from required conditions, list impacted samples):

Samples Acidified? ____ Yes ✓ No ____ Unknown

Initial pH 5 - 9? (Y/N): NA *If no, individual sample adjustments on the Auxiliary Sample Receipt Form.*

Total Residual Chlorine Present? (water) (Y/N): NA
If yes, individual sample adjustments on the Auxiliary Sample Receipt Form.

Head Space <1% in samples for water VOC analysis NA Yes ____ No
Individual sample deviations listed below.

Sample Containers:
Samples returned in PC-grade jars? ____ Yes ✓ No ____ Unknown / Lot No. ____

Storage Location: FIRST FLOOR COLD ROOM BDO IDs Assigned: W9071-W9072

Holding Times:	Water	Sediment	Tissue

Additional Comments:

Samples logged in by: [signature] Date/Time: 11/14/01 3:00PM
* Must also be noted on the C-O-C.

BATTELLE

~~TestAmerica~~ INCORPORATED

~~Watertown Division~~ ~~502 Commerce Drive~~ ~~Watertown, WI 53094~~ ~~Phone: 920-261-1660~~ ~~Fax: 920-261-6120~~

To assist us in using the proper analytical methods, is this work being conducted for regulatory purposes?
Compliance Monitoring _____

Client Name: WDNR Client #: _____
Address: 810 W. MAPLE ST.
City/State/Zip Code: SPOONER, WI 54801
Project Manager: JAMES R. DUNN
Telephone Number: 715.635.4049 Fax: _____
Sampler Name: (Print Name) JOHN E. GUHL
Sampler Signature: John E. Guhl (SEH)

Project Name: ASHLAND - NSP COAL GAS SITE
Project #: WIDNR9401
Site/Location ID: ASHLAND State: WI
Report To: JAMES R. DUNN
Invoice To: JAMES R. DUNN
Quote #: _____ PO#: _____

TAT
X Standard
___ Rush (surcharges may apply)
Date Needed: _____
Fax Results: Y N

SAMPLE ID	Date Sampled	Time Sampled	G = Grab, C = Composite	Field Filtered	Matrix: SL - Sludge DW - Drinking Water GW - Groundwater S - Soil/Solid WW - Wastewater Specify Other	Preservation & # of Containers: HNO_3	HCl	NaOH	H_2SO_4	Methanol	None	Other (Specify)	Analyze For: FINGERPRINT ANALYSIS								BDD ID	REMARKS
2-Inch Steel Pipe	11/12/01	10:30	G								2		/								W9071	NAPL FROM INSIDE PIPE
12-Inch Steel Pipe	11/12/01	2:00	G								2		/								W9072	WATER/NAPL FROM INSIDE PIPE

QC Deliverables
___ None
___ Level 2 (Batch QC)
___ Level 3
___ Level 4
Other: _____

Special Instructions:

LABORATORY COMMENTS:
Init Lab Temp:
Rec Lab Temp:
Custody Seals: Y N N/A
Bottles Supplied by TestAmerica: Y N
Method of Shipment:

Relinquished By	Date	Time	Received By	Date	Time
John E. Guhl	11/13/01	12:00	Jim DV	11/14/01	10:20

BATTELLE Laboratory Sample Login Report

Project # : N004602-0002 ***Receive Date :*** 11/14/01 10:20:00 AM

Client : ASHLAND ***Logged in By :*** JHATCH

Collection Date	*Login Date*	*Lab ID*	*Client Sample ID*	*#Containers*	*Matrix*	*Preservative*	*Storage Location*	*Login Comments*
11/12/01 10:30:00 AM	11/14/01 2:26:08 PM	W9071	2 INCH STEEL PIPE	1	SLUDGE	NONE	FIRST FLOOR COLD ROOM	
11/12/01 2:00:00 PM	11/14/01 2:57:07 PM	W9072	12 INCH STEEL PIPE	1	SLUDGE	NONE	FIRST FLOOR COLD ROOM	