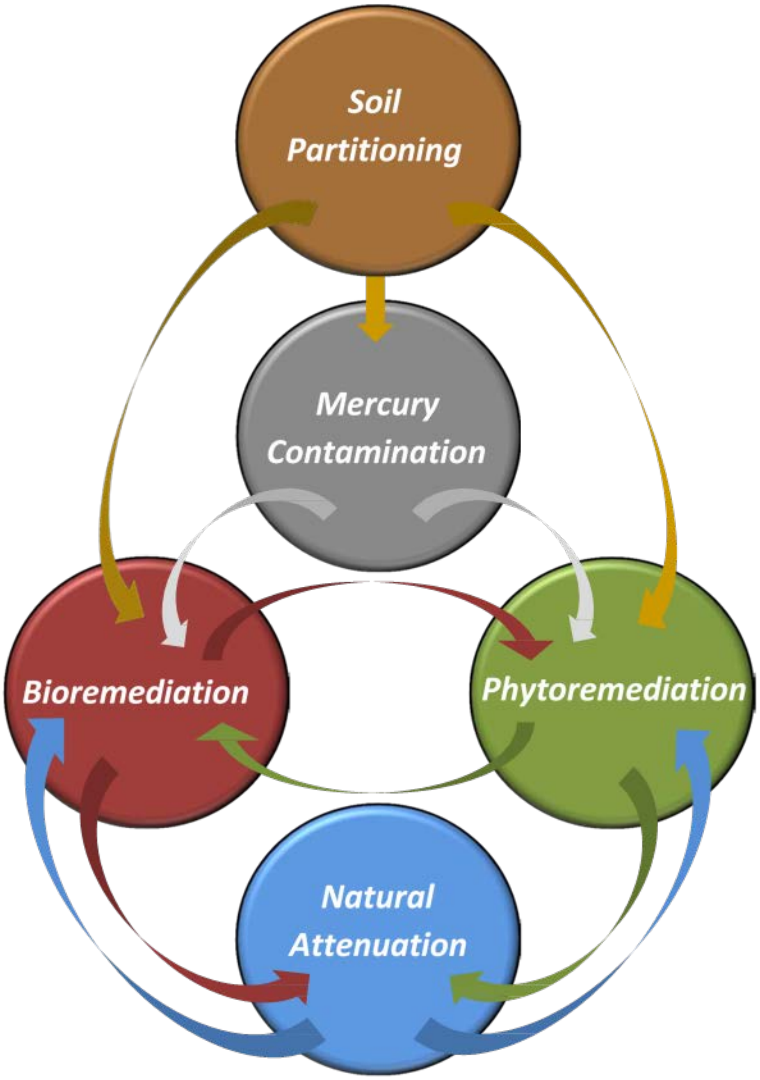
Soil Treatability Studies Summary Report

Soil Treatability Studies
Area IV Santa Susana Field Laboratory
Ventura County, California



September 2015

CDM Smith

Soil Treatability Studies Summary Report
Area IV Santa Susana Field Laboratory
Ventura County, California

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September 2015
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Ventura County, California

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Prepared by: Keegan L. September 14, 2015
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Acronyms

AOC	Administrative Order of Consent
Cal Poly	California Polytechnic State University
COIs	contaminants of interest
dioxins	polychlorinated dibenzo dioxins
DOE	U.S. Department of Energy
DTSC	Department of Toxic Substances Control
EDTA	ethylenediaminetetraacetic acid
EFHs	Extractable Fuel Hydrocarbons
ft bgs	feet below ground surface
LUT	Look-up Table
mg/kg	milligrams per kilogram
mm	millimeters
PAHs	polycyclic aromatic hydrocarbons
PCBs	polychlorinated biphenyls
Sandia	Sandia National Laboratories
SSFL	Santa Susana Field Laboratory
TPH	total petroleum hydrocarbons
UCR	University of California-Riverside

Section 1

Introduction

In accordance with the Administrative Order of Consent (AOC) signed by the U.S. Department of Energy (DOE) with the California Department of Toxic Substances Control (DTSC), DOE conducted a series of soil treatability studies using soil from Area IV of the Santa Susana Field Laboratory (SSFL). The objective of the soil treatability studies was to evaluate onsite soil treatment technologies that could reduce the volume of contaminated soil that otherwise would need to be excavated and transported from Area IV.

DOE's first step in conducting the soil treatability studies was engaging Sandia National Laboratories (Sandia) to review literature and inquire with practicing remediation professionals on soil treatability studies conducted throughout the world to determine which soil treatment technologies may be applicable to contaminants in soil at Area IV. The contaminants of interest (COIs) investigated by the treatability studies were, generally, metals (primarily mercury, silver, lead), polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), polychlorinated dibenzo dioxins (dioxins), and total petroleum hydrocarbons (TPH).

Based on Sandia's literature review and inquiry with professionals, Sandia recommended that DOE consider six soil study options (Sandia 2012) for the COIs:

- Mercury valence state determination
- Soil partitioning
- Bioremediation
- Phytoremediation
- Natural attenuation
- Thermal treatment

Section 2

Background

Mercury has unique chemical properties that allow it to exist in the environment in different chemical states (also termed valence states) – as a liquid metal, as a salt, or as a complex with other compounds. The chemical state of mercury in soil is important to know for soil treatment considerations because as a metal it can be removed from soil through thermal treatment, as a soluble salt it can be absorbed in soil by microbes and plant roots, and as an insoluble complex only soil washing may be effective in removing mercury from soil.

Soil partitioning evaluates within which of the various particle size ranges the contaminants may be found. Soil is composed of particles ranging in size from a fraction of a millimeter (clay, silt, and fine sand) to coarse sand and gravel sizes. Typically soil contaminants preferentially adsorb to the smaller soil particles (termed "fines"). If this is the case, it may be possible to separate larger soil particles from the smaller particles through the use of sieves. If the contaminants are primarily found on the smaller soil particles, then the separation can reduce the volume of soil to be transported from Area IV. The Area IV soil is primarily of sandstone origin so it consists of predominantly larger soil particle sizes. The types and sizes of soil particles that contaminants are adsorbed to is also important for biological remediation considerations. In order for microbes and plant roots to absorb contaminants, the contaminants must be soluble or found in soil moisture. If the contaminants are strongly adsorbed to smaller soil particles, the contaminants may not be soluble and thus are not available to microbes and plant roots. Soil partitioning is also the first step in evaluating whether soil washing (removing contaminants from soil particles using chemicals and mechanical mixing) would be an effective treatment technology.

Bioremediation employs microorganisms (e.g., bacteria) and fungi to use the contaminants as an energy source or to chemically convert the contaminants to less toxic forms. How the microorganisms can convert the contaminants is highly dependent on the amount of oxygen and types of bacteria/fungi in the soil. Some microorganisms can only live in soil with oxygen, while others can only live in the absence of oxygen. And, as indicated in the paragraph below, there can be a symbiotic relationship between microorganisms and plant roots.

Phytoremediation is the use of plants to uptake (remove from soil) and/or degrade contaminants. The contaminants either are metabolized (converted to carbon dioxide, water, and salts) by the plants, incorporated into plant tissue, or volatilized from the plants into the atmosphere. In order for the contaminants to be absorbed into plant roots, the contaminants must be biologically available, usually meaning the contaminants must be in a soluble form. In some instances, a symbiotic relationship between plants and microbes exists; microorganisms in the soil can make some contaminants more available to plant roots, and the roots can release compounds making chemicals more available to microorganisms.

Natural attenuation is the use of physical, chemical, and biological processes to reduce concentrations of contaminants in soil. All organic compounds (meaning compounds with carbon as a basis) will eventually degrade into basic elements such as carbon dioxide, water, and mineral salts. However, the rate of chemical degradation can vary greatly depending on contaminant properties (e.g., volatility, solubility, and degree of chlorination) and soil type and conditions. Degradation rates can range from months to years depending upon the aforementioned variables. Knowing rates of degradation is important not only for site monitoring considerations, but also for understanding the feasibility of enhancing degradation of contaminants in soil.

Thermal treatment is the practice of either heating soil to drive off volatile chemicals (fuels, solvents, mercury) or incinerating soil to destroy highly stable chlorinated chemicals. Thermal treatment can involve placement of thermal probes into the soil to heat it, or excavation of the soil for placement in incinerators for burning.

Section 3

Treatability Studies Discussion

Based on the recommendations made by Sandia, DOE elected to conduct five soil treatability studies:

- Mercury chemical (valence) state determination
- Soil partitioning
- Bioremediation
- Phytoremediation
- Natural attenuation

Thermal treatment was not chosen to be studied at this time.

The selected soil treatment technologies are interrelated and provide supporting information for each other. For example, knowing the mercury chemical state is necessary to determine whether mercury is amenable to soil washing, bioremediation, phytoremediation, and/or thermal treatment. The soil partitioning study results indicate whether contaminants could be available to plants, microorganisms, or fungi for bioremediation and phytoremediation studies. All of the studies are related to the biological, chemical, and/or physical aspects of natural attenuation. Therefore, the findings of each study determine the natural attenuation processes present at Area IV and their rates of COI reductions.

At the suggestion of the community, DOE contracted two local universities to conduct the soil treatability studies. Researchers at California Polytechnic State University (Cal Poly) conducted the bioremediation, phytoremediation, and natural attenuation studies; researchers from the University of California-Riverside (UCR) conducted the mercury and soil partitioning study. The goal of the studies conducted by the universities was to determine whether any of the technologies could reduce soil concentrations to meet cleanup levels identified in the AOC soil Look-up Table (LUT), or could reduce the volume of contaminated soil requiring excavation and transportation from Area IV.

To describe the relationships, background, and purposes of the studies, DOE developed a Master Work Plan (CDM Smith, 2013). Cal Poly developed separate work plans for the bioremediation study (Nelson et al., 2015a), phytoremediation study (Nelson et al., 2015d), and natural attenuation study (Nelson et al., 2014 and 2015c). UCR developed separate work plans for the mercury study (Liu, 2015) and soil partitioning study (Matsumoto and Martin, 2015).

The treatability studies determined that attaining the AOC soil cleanup levels (LUT values) would require significant time and effort, if the LUT values could be achieved at all, through the identified treatment options. This determination is due in part to the combination of the highly weathered state of the contaminants (i.e., the more easily degradable contaminants have already been degraded), contaminant chemical state (e.g., mercury valence state), and the nature of the AOC cleanup levels. The results of the five individual treatability studies are summarized below.

More detailed descriptions of each study's purpose, design and methodology, results, and conclusions can be found in the individual treatability study work plans and study reports (see References Section for full citations).

3.1 Mercury Contamination Study Conclusions

The mercury contamination treatability study was designed to determine the current valence states and spatial distribution of mercury in Area IV soils, and assess potential mercury remediation technologies. The results are reported in Liu (2015). Soil samples were collected from four locations within Area IV, and these samples were then analyzed to determine the concentration of each mercury valence state. Mercury concentration information from previous soil sampling events was also reviewed to aid in the interpretation of the spatial distribution of total mercury at Area IV.

In three of the four locations sampled specifically for this study, mercury concentrations were highest in surface soils (0 and 1.5 feet below ground surface [ft bgs]) and decreased with increasing soil depth. Total mercury exceeded its AOC LUT value at these locations. The sample location not exhibiting the trend of mercury concentration decreasing with depth (STS-73-SA6) had been excavated as part of a soil removal action prior to the commissioning of these soil treatability studies.

No elemental vapor phase mercury was detected in any of the samples. Methyl mercury was detected only in trace amounts at two locations. Approximately 12 percent of all mercury found in the soil samples was in a chemical form that is soluble (mobile) and thus potentially bioavailable. This would suggest that soil washing, bioremediation, and phytoremediation, theoretically, could be viable treatment options to remove the mobile fractions of mercury from these surface soils. However, the immobile fraction of mercury at the same locations is still above the mercury LUT value. This means that bioremediation and phytoremediation alone would likely not be able to achieve mercury LUT values for Area IV soil. The phytoremediation study saw no increase of mercury in plant tissue samples under controlled greenhouse conditions.

In deeper soils, the majority of mercury exists in the immobile, elemental form that is tightly bound to soil particles. Additional testing of potential alternative cleanup approaches would be required to determine the feasibility of any treatment methods for these immobile fractions.

Figure 1 presents the location of the samples collected for the Mercury Contamination Study.

Service Layer Credits: Source: Esri, DigitalGlobe, GeoEye, i-cubed, USDA, USGS, AEX, Getmapping, Aerogrid, IGN, IGP, swisstopo, and the GIS User Community.

Legend

- ○ Mercury Study Sample Location
- ⬚ NE Buffer Zone Cleanup Responsibility
- ⬚ Site Area

Mercury Treatability Study Sample Locations

Santa Susana Field Laboratory
Ventura County, California
Figure 1

CDM Smith

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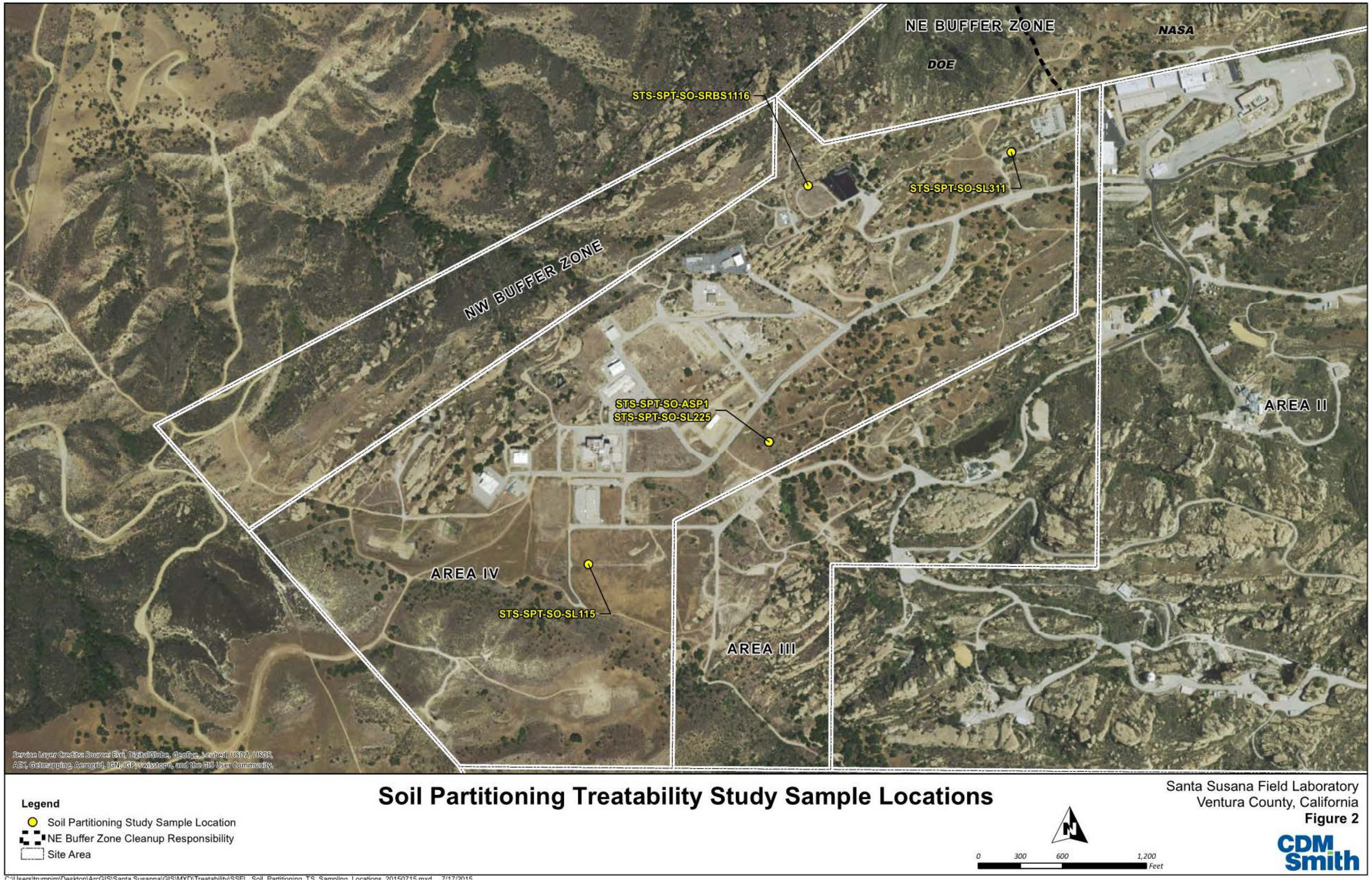
3.2 Soil Partitioning Study Conclusions

The soil partitioning study was designed to determine the soil particle size range(s) where contaminants are found. The results are reported in Matsumoto and Martin (2015). Surface soil samples (0.0 to 0.5 or 0.5 to 1.5 ft bgs) were collected from four Area IV locations. At one location, three distinct depth intervals were sampled (0.0 to 0.5, 0.5 to 1.5, and 2.0 to 3.0 ft bgs). Each sample was subsequently separated into one of four particle size ranges (greater than 2.0 millimeters [mm], 0.425 to 2.0 mm, 0.075 to 0.425 mm, and less than 0.075 mm) via mechanical sieving. Each size fraction and an aliquot of the whole un-sieved soil for each sample were then analyzed for the COIs to determine if the COIs were preferentially found in a certain soil particle size range.

The soil partitioning study concluded that it may be possible to reduce the removal depth of contaminated soil for offsite disposal or onsite treatment, as the COI concentrations generally decreased with increasing depth in the soil column (suggesting that downward migration through the soil of the contaminants is a slow process). However, the potential for reducing the volume of contaminated soil requiring treatment or disposal via particle size separation is minimal. COI concentrations were found to exceed the LUT values in many of the soil size fractions. *Ex-situ* soil washing is a potentially applicable remedial technology due to dominance of coarse material and sands in Area IV soils, but further laboratory testing would be required to determine the strength of the chemical washing agents (solvents, surfactants, acids, etc.) required for COI removal, to determine the agent's effects on the soil, and to determine proper waste disposal procedures for any generated waste stream. It is likely that multiple washing agents would be required for COI removal, resulting in sterilization of the soil. This sterilization would likely require the addition of significant soil amendments to restore the soil biological function.

Figure 2 presents the location of the samples collected for the Soil Partitioning Study.



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Soil Partitioning Treatability Study Sample Locations

Santa Susana Field Laboratory
Ventura County, California
Figure 2

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3.3 Bioremediation Study Conclusions

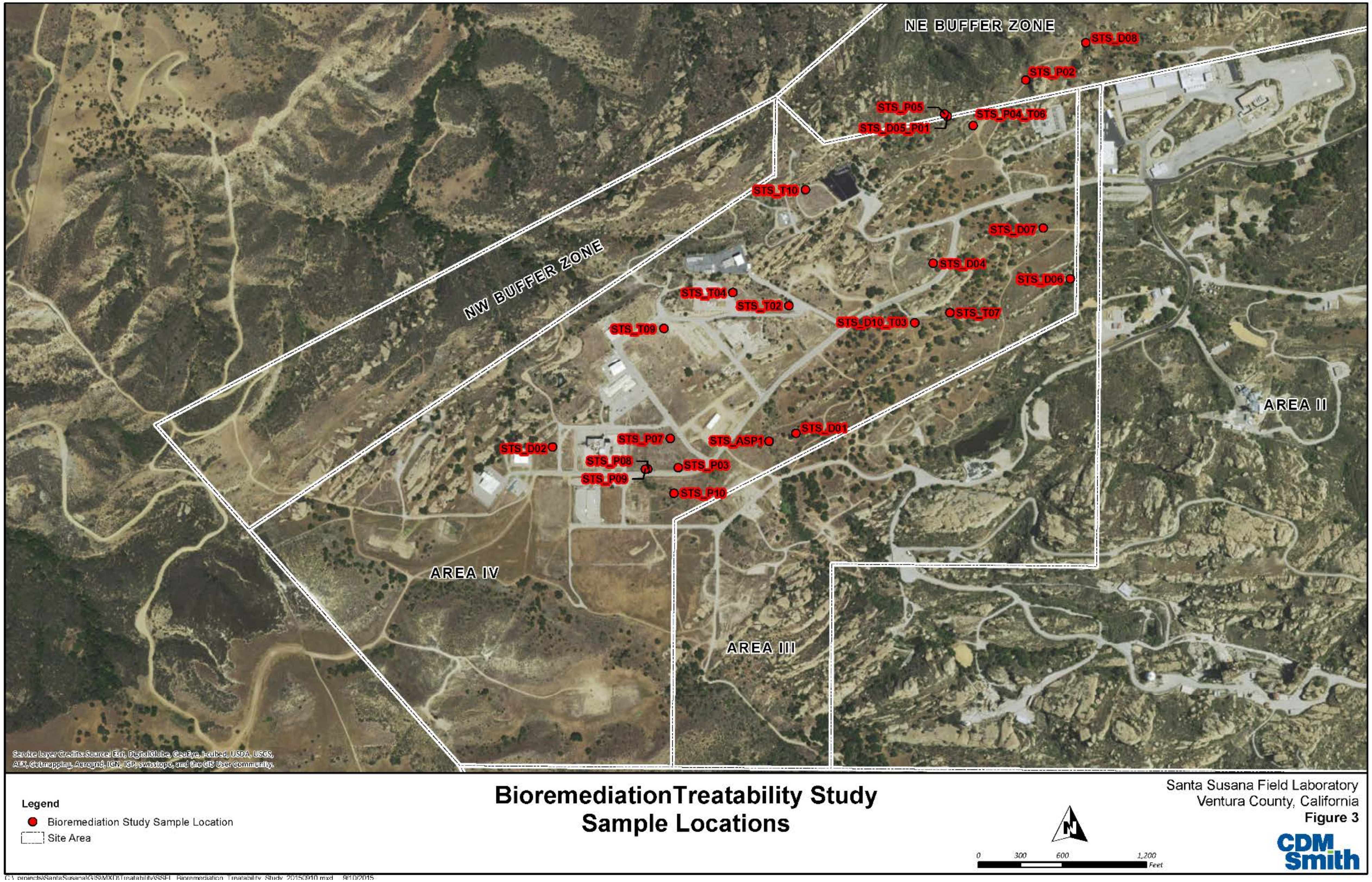
The bioremediation treatability study was conducted in two phases. Both phases are reported in Nelson et al. (2015a). Phase 1 involved the collection of soil samples from across Area IV that had a range of COI concentrations. These samples were then cultured and/or analyzed to determine if the microbial organisms already present in Area IV soils are capable of degrading the COIs. Phase 2 involved collecting Area IV soils for use in controlled laboratory microcosms to measure biodegradation rates under typical site conditions and to estimate the effectiveness of biostimulation (e.g., adding fertilizers) and bioaugmentation (e.g., adding organisms) for increasing COI degradation rates.

Phase 1 results identified 21 unique microorganism species in Area IV soils, including 14 bacteria and 7 fungi. These isolated microorganisms included three strains of the fungi *Phanerochaete chrysosporium*; bacterial species *Arthrobacter, Streptomyces, Micromonospora,* and *Variovorax*; and six strains of the bacteria *Pseudomonas*. Ten of the bacteria and three of the fungi that were isolated are known to be degraders of the COIs or come from a genus that contains known COI degraders.

Phase 2 results suggested slow biodegradation rates for the organic COIs. Slight decreases in organic COI concentrations were observed over the 8-month laboratory microcosm incubation period, but decreases were not statistically significant at 95% confidence. Biostimulation with fertilizer and bulking agents was not effective at increasing organic COI degradation rates, and biostimulation with surfactants had limited effect. Bioaugmentation with the white-rot fungi *Phanerochaete chrysosporium* was determined to have potential for degradation of dioxins.

The Phase 2 conclusions also suggested that the organic COIs at Area IV are highly weathered and strongly adsorbed to soil particles. Most of the readily biodegradable compounds have likely already biodegraded or volatilized, and the remaining compounds are likely to be sequestered in the soil, reducing their bioavailability. Therefore, if bioremediation were considered as a remedial technology, it would primarily be for those areas with low COI concentrations, low risk of COI exposure to humans, and where significant time for degradation could be allowed.

Figure 3 presents the location of the samples collected for the Bioremediation Study.



BioremediationTreatability Study
Sample Locations

Santa Susana Field Laboratory
Ventura County, California
Figure 3

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3.4 Phytoremediation Study Conclusions

The phytoremediation study was conducted in two phases. Phase 1 involved the collection of plant roots, above ground stems and foliage, and associated soils to determine which plant species presently growing in Area IV may contribute to the phytoremediation of the COIs. Nine species of plants were sampled during Phase 1. Phase 2 involved growing three candidate plant species identified during Phase 1 in greenhouse microcosms to measure COI uptake/degradation rates and test the effects of additives (e.g., chelating agents, fertilizer) on uptake/degradation rates. Both phases are reported in Nelson et al. (2015d).

Phase 1 results showed that Extractable Fuel Hydrocarbons (EFHs) were observed in the roots and foliage of all plant species. However, the EFHs observed in plant tissues appeared to be phytogenic (i.e., produced by plants). PAHs were also detected in the roots of most plant species but, similar to EFHs, the PAHs detected in some species appeared to be phytogenic.

PCB uptake was not observed for any Phase 1 plant species; however, PCBs were not detected in the soils associated with Palmer's Goldenbush or Purple Needlegrass specimens. Chlorinated dioxins were found in Blue Elderberry, Palmer's Goldenbush, Yerba Santa, and Purple Needlegrass tissues.

Mercury uptake was not observed for any species; however, mercury was below the detection limits in soil associated with Palmer's Goldenbush, Narrowleaf Milkweed, and Purple Needlegrass. Silver was observed in the roots of all plant species except for Palmer's Goldenbush and Purple Needlegrass. Summer Mustard was the only species that showed uptake of silver into the foliage. This uptake of silver into the foliage was at much lower concentrations than the silver concentration present in the soil.

For the Phase 2 greenhouse microcosms, Coyote Brush (*Baccharis pilularis*) and Mule Fat (*Baccharis salicifolia*) were selected for study as they both showed uptake of most of the COIs during Phase 1. A grass species had to be selected without field screening due to growing-season constraints, and Purple needlegrass (*Nassella pulchra*) was chosen because it is native to Area IV and known for its resilience.

A comparison of Phase 2 planted and unplanted microcosm results indicated that EFH degradation rates via rhizostimulation of soil microbes are slow over the first 85 days. Final EFH concentrations at 211 days were inconclusive because of an apparent anomaly in EFH measurement between different contract analytical laboratories used for the 85-day test analysis and 211-day analysis. Therefore, 12-month samples were sent to the first analytical laboratory (same as the 85-day test). The resulting petroleum hydrocarbon concentrations fell in a similar concentration range as the initial and second analyses.

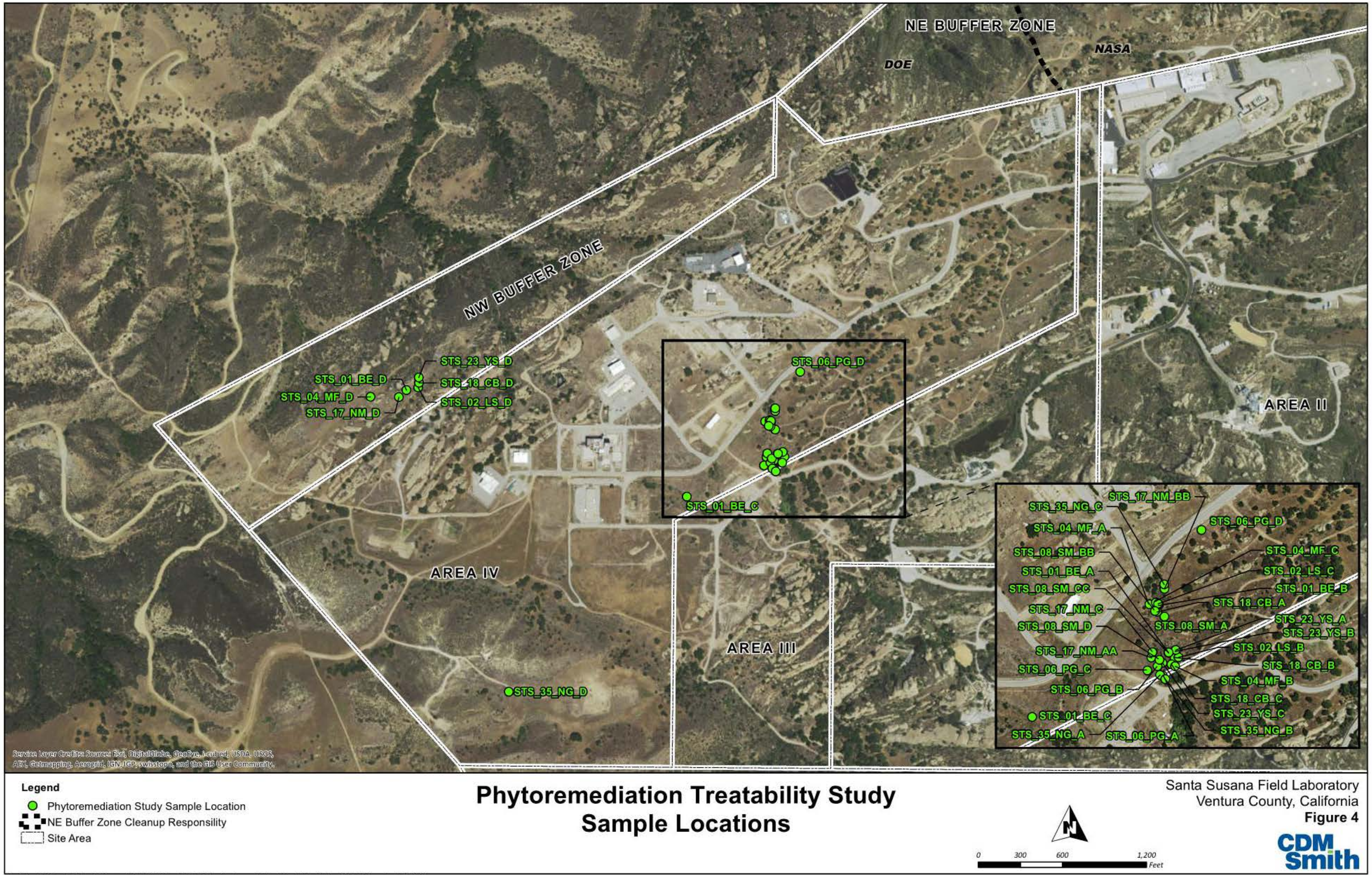
As a follow-up effort to these treatability efforts, university researchers investigated, in part, the inherent difficulties of accurately measuring petroleum hydrocarbons at low concentrations (Nelson et al., 2015b). The study's authors observed that it is difficult to accurately quantify petroleum hydrocarbons at the concentrations that were present in their Area IV soil samples (100 to 300 milligrams per kilogram [mg/kg]). As such, the study's authors concluded that reliable hydrocarbon measurement near the 5 mg/kg LUT value for TPH would be nearly impossible to achieve for Area IV. Hydrocarbon concentrations are calculated by integrating the area under numerous overlapping gas chromatograph data peaks, and thus a baseline has to be drawn underneath the peaks for the integration. The study author's stated that drifting baselines, non-zero signal at the end of the elution time, and non-zero blanks all can make the concentration calculation inaccurate at low hydrocarbon concentrations.

No significant reductions of PAH, silver, or mercury soil concentrations were observed for any of the Phase 2 microcosms. Amending the soil by adding the chelation agent ethylenediaminetetraacetic acid (EDTA) did not improve mercury uptake.

Soil PCB concentrations decreased by 13 to 15 percent (with significant variability of observed PCB concentrations) over 7 months for soil microcosms planted with purple needlegrass and coyote brush, relative to sterile controls. Since no PCBs were observed in the plant tissue of these microcosms, the mechanism for this PCB reduction appears to be stimulation of soil bacteria. Soil dioxin concentrations decreased by 18 to 20 percent over 7 months for soil microcosms planted with coyote brush. Rhizostimulation again appears to be the operative mechanism.

As with bioremediation, phytoremediation could be useful in conjunction with site restoration in areas with low COI concentrations, low risk of COI exposure to humans, and where significant time for degradation could be allowed.

Figure 4 presents the location of the samples collected for the Phytoremediation Study.



Phytoremediation Treatability Study
Sample Locations

Santa Susana Field Laboratory
Ventura County, California
Figure 4

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3.5 Natural Attenuation Study Conclusions

The natural attenuation study was conducted in two phases. Phase 1 primarily consisted of a literature review of natural attenuation processes to determine which Area IV COIs are amenable to natural degradation processes. The literature review results were then used to estimate the time that would be required for natural attenuation of these COIs to reach LUT values, based on current Area IV soil COI concentrations. Phase 1 also examined soil COI concentrations from locations resampled over a period of time in order to estimate site-specific natural attenuation rates. All of these results were presented in a Phase 1 natural attenuation study report (Nelson et al., 2014).

Figure 5 presents the location of the samples collected for the Natural Attenuation Study.

Phase 2 involved using findings from the companion bioremediation and phytoremediation treatability studies to further develop site-specific COI degradation rates under typical site conditions. The Phase 1 natural attenuation study report was then updated with the findings from Phase 2 (Nelson et al., 2015c).

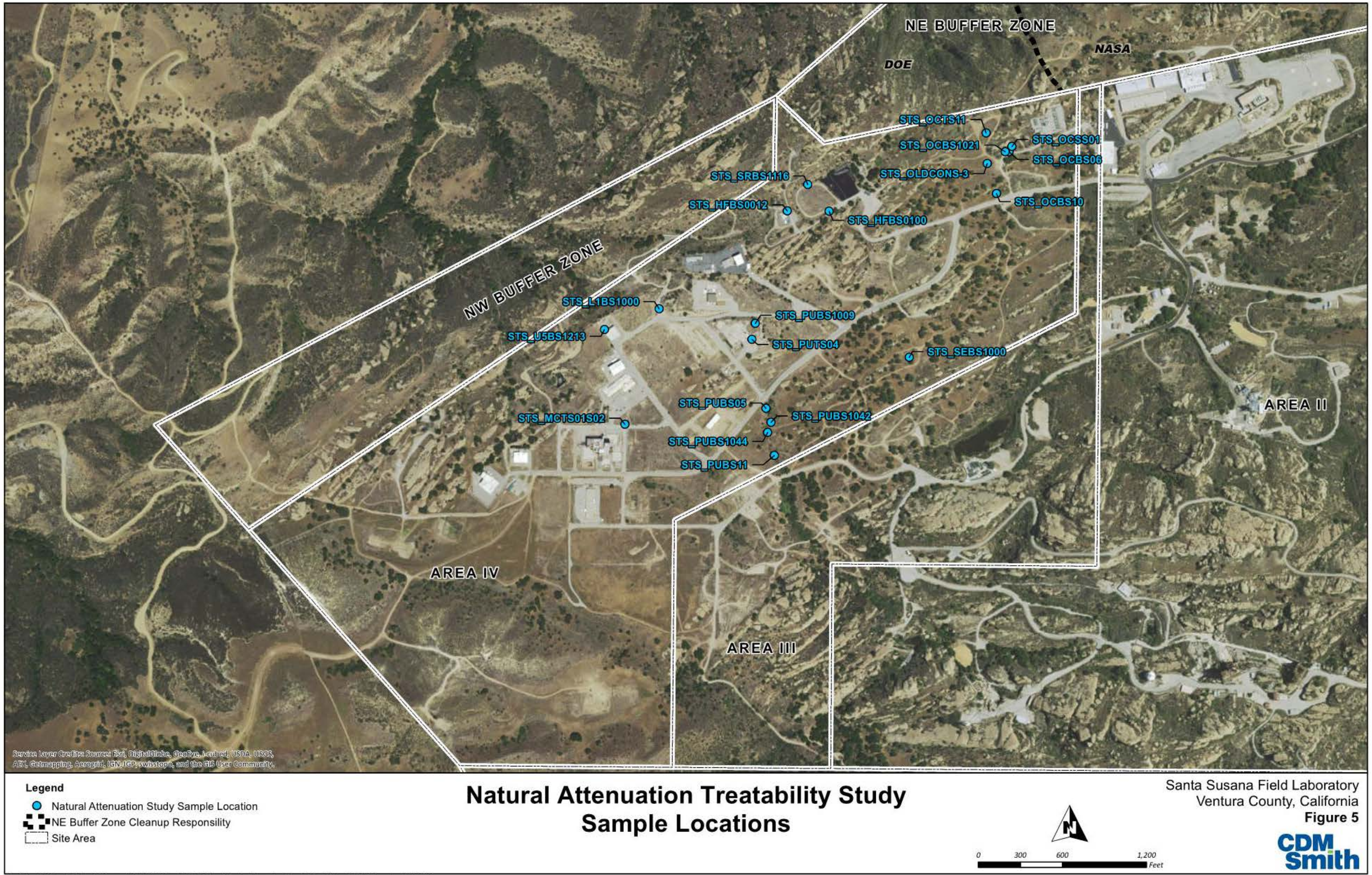
The findings of the Phase 1 literature review (Nelson et al., 2014) suggest all organic COIs in Area IV soils will eventually biodegrade, but some COIs will degrade slowly. Predicted times for Area IV soil COI concentrations to reach LUT values, as determined by the natural attenuation study Phase 1 literature review, are as follows:

- Petroleum hydrocarbons: 0.42 to 69 years
- PAHs: 5 to 15 years
- PCBs: Undetermined (half-life = 40 years)
- Chlorinated dioxin/furans: 1 to 50 years

The broad range of predicted natural attenuation rates were attributed to the variances in the initial concentrations and biodegradation rates reported in the literature.

Phase 2 analyses of the companion bioremediation and phytoremediation studies suggested longer estimated remediation times than those predicted by the published literature. Conclusions regarding Area IV-specific natural attenuation rates, as determined by the review of the bioremediation and phytoremediation treatability study results, are provided further below.

This study ultimately concluded that natural attenuation at Area IV is expected to take on the order of decades to reach current AOC LUT values for the various COIs. These predicted remediation times are partially the result of the current clean-up goals to reach background COI levels and the result of COI weathering into the soil matrix, rendering the contaminants generally unavailable to biological or phytoremediation potential. The natural attenuation study also concluded that much shorter remediation times would be expected if clean-up goals were set similar to those set for typical past industrial sites (Nelson et al., 2015b)(see Attachment A).



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Natural Attenuation Treatability Study
Sample Locations

Figure 5

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3.5.1 EFH Natural Attenuation Conclusions

More than a 40 percent reduction in EFH concentrations in 126 days was observed for one of the three unamended bioremediation Phase 2 microcosms. However, little or no EFH was observed for the other two microcosms. These results indicate the variability in the efficacy of hydrocarbon bioremediation.

Naturally occurring organic matter could possibly be misinterpreted as petroleum hydrocarbons during chemical analyses. As stated above, Nelson et al. (2015b) determined that it is difficult to accurately quantify petroleum hydrocarbons at the concentrations that were present in their Area IV soil samples (see Attachment A). The study also concluded that reliable hydrocarbon measurement near the 5 mg/kg LUT value would be nearly impossible to achieve.

3.5.2 PAH Natural Attenuation Conclusions

Phase 2 bioremediation microcosm experiments did not show significant PAH biodegradation for the unamended controls. Phytoremediation study results also indicated little or no PAH biodegradation in microcosm soils with plants. Together, these results suggest limited PAH degradation under natural conditions, and that the PAHs remaining in Area IV soils have low biodegradability. This limited PAH degradation may be a result of PAHs being weathered and sequestered in the soil matrix, limiting their availability to microorganisms.

3.5.3 PCB Natural Attenuation Conclusions

Bacterial PCB degradation requires anaerobic dechlorination, and anaerobic conditions were not observed in Area IV soils. This suggests that bacterial dechlorination of PCBs will not be a viable mechanism for PCB degradation. However, fungal biodegradation of PCBs is possible under aerobic conditions, and three strains of the white-rot fungi *Phanerochaete chrysosporium* reportedly capable of biodegrading PCBs were isolated from Area IV soils. PCB degradation via white-rot fungi may be a potential remedial measure.

PCB concentrations decreased over the bioremediation microcosm incubation period, but these decreases were not statistically significant at the 95% confidence level from the sterile controls. In the phytoremediation study, soil PCB concentrations also decreased for soil microcosms planted with purple needlegrass and coyote brush. No PCBs were observed in plant tissue, so the mechanism for PCB concentration reduction appears to be stimulation of microorganisms in the soil.

3.5.4 Dioxin Natural Attenuation Conclusions

Only small decreases in dioxin concentrations were observed in the unamended bioremediation soil microcosms, suggesting slow dioxin biodegradation under natural attenuation conditions. The bioremediation microcosms augmented with the fungi *Phanerochaete chrysosporium* exhibited decreases in chlorinated dioxin concentrations more than in unamended microcosms. Some decreases in soil dioxin concentrations were observed in the planted phytoremediation microcosms. This suggests some potential, albeit limited, for dioxin remediation via phytoremediation processes.

Section 4

Summarized Conclusions

Results from the Cal Poly and UCR soil treatability studies identified the following conclusions:

- The COIs present in Area IV soils appear to be highly weathered. The easily degraded COIs have likely already been degraded.
- Due to their state of weathering, the COIs are fairly recalcitrant and are not likely readily amenable to natural attenuation, bioremediation, or phytoremediation processes.
- It will take considerable time (possibly decades in some cases) to achieve current LUT values for organic COIs via natural attenuation, bioremediation, or phytoremediation processes.
- It is likely that bioremediation or phytoremediation processes cannot achieve current LUT values, regardless of duration, for mercury as the immobile fraction of mercury present in many samples is, by itself, above the mercury LUT value.
- The potential for reducing the volume of contaminated soil requiring disposal or treatment via size separation is minimal.
- *Ex-situ* soil washing is a potential remedial technology for Area IV soils; however, further study would be required to determine the required strength of the extraction fluid and its effects on the soil.
- University researchers determined that naturally occurring organic matter could possibly be misinterpreted as petroleum hydrocarbons during laboratory chemical analyses.

Section 5

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Attachment A – Chemical Characterization of Residual Fuel Hydrocarbons in Soils at the Santa Susana Field Laboratory

Chemical Characterization of Residual Fuel Hydrocarbons in Soils at the Santa Susana Field Laboratory

Environmental Remediation Services for Environmental Compliance for Area IV

FINAL REPORT

July 31, 2015

Submitted to the US Department of Energy via CDM Smith

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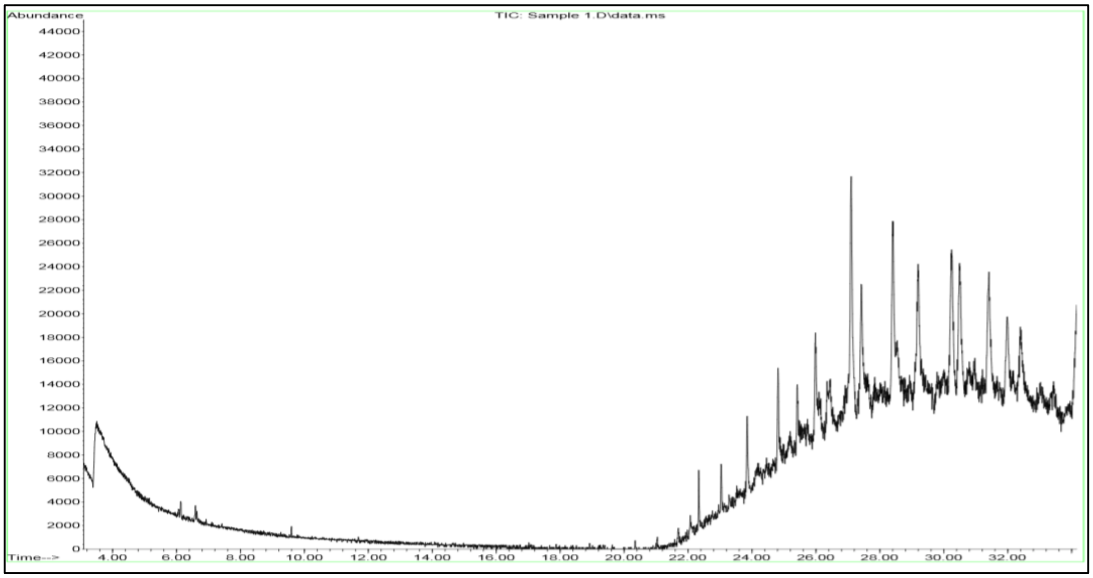

Executive Summary

Five soil samples were collected from Area IV of the former Santa Susana Field Laboratory (SSFL) and analyzed using gas chromatography/mass spectroscopy (GC/MS) to determine the chemical constituents of the material being characterized as total petroleum hydrocarbon (TPH). One objective of this study was to determine to what extent natural organic material (NOM) contributes to the TPH signal. Another objective was to investigate the use of silica gel cleanup as a method to remove polar NOM before TPH analysis to provide a more accurate measure of actual petroleum hydrocarbons. A third issue which ended up being addressed in this study was the inherent difficulties of accurately measuring TPH at low levels.

Results of the GC/MS spectral analyses suggest that the contamination of Area IV soils being characterized as TPH consists of high molecular weight hydrocarbons, suggestive of residual-range oil (RRO). These hydrocarbons are mostly highly-branched and cyclic hydrocarbons which are known to be slow to biodegrade. PAHs are also present in the soils, as well as some heterocyclic compounds. Many of the branched hydrocarbons and PAHs are partially oxidized. Also detected in the soil samples were organic acids such as oleic acid and oxalic acid, suggestive of NOM. This NOM fraction accounted for about 5 to 8% of the total TPH.

Silica gel cleanup by EMAX Laboratory removed 5 to 40% of the TPH in the five soil samples. Mixed results were obtained in the Cal Poly lab, with silica gel cleanup actually increasing the TPH for three of the five samples. GC/MS analysis showed that silica gel cleanup removed some of the polar organic compounds, but not all of them. Thus, the silica gel approach may underestimate the contribution of NOM to TPH measurement. PAHs and large branched hydrocarbons were not removed by the silica gel cleanup.

An important observation during this study is that it is difficult to accurately quantify TPH at the very low soil TPH concentrations of the Area IV soils used in this study (100 to 300 mg/kg). TPH concentrations of soil samples sent to two different contract laboratories as part of the earlier bioremediation study differed by nearly an order of magnitude. Reliable TPH measurement near background TPH levels or near the 5 mg/kg look-up table value for Area IV would be nearly impossible. At these very low TPH concentrations several factors can significantly impact the calculation of TPH concentrations from GC data, making the calculated concentrations unreliable. TPH concentrations are calculated by integrating the area under numerous overlapping peaks, and thus a baseline has to be drawn underneath the peaks for the integration. Drifting baselines, non-zero signal at the end of the elution time, and non-zero blanks all can make the calculation of TPH inaccurate at low TPH concentrations.

The look-up table value published by the DTSC for TPH is 5 mg/kg, which is 20 times lower than any state standard for RRO, and 1,000 times lower than typical values used by many states. Most states have now started using risk-based clean-up goals. It is beyond the scope of this study to argue for a change in clean-up goals, however, this study has demonstrated that there are clear technical problems with measuring TPH concentrations at such low levels.

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1.0 Introduction

Cal Poly recently completed three soil treatability studies to support remediation efforts for contaminated soil in Area IV of the former Santa Susana Field Laboratory (SSFL). The purpose of these studies was to determine the viability of using *in-situ* remediation techniques, such as bioremediation, phytoremediation and/or natural attenuation to reduce contaminant concentrations and thus reduce the volume of soil needing to be excavated and hauled off-site. In the process of conducting this research, Cal Poly researchers noted difficulties accurately measuring total petroleum hydrocarbon (TPH) concentrations at the very low levels specified by the Department of Toxic Substances and Control (DTSC) for cleanup goals and also discovered the presence of natural organic material (NOM) in the soil samples which was inadvertently contributing to TPH quantification. To address these issues, Cal Poly was given the charge to investigate the chemical composition of the material in the soil being quantified as TPH, and test methods such as silica gel cleanup for providing more accurate TPH measurement.

Possible anomalies in the measurement of TPH were elucidated in the analysis of the final soil samples for a bioremediation microcosm experiment which was part of the bioremediation study. Initial soil samples collected during this experiment and those collected after 126 days of incubation were analyzed for TPH by EMAX, an EPA-certified contract laboratory. Final samples collected after 244 days of incubation were analyzed for TPH by a different contract laboratory – Lancaster Laboratory. TPH concentrations reported by Lancaster Laboratory were 5 to 10 times higher than those reported by EMAX for earlier samples. Both laboratories use an EPA-approved method for gas chromatographic (GC) analysis. Since such an increase in TPH concentration didn’t make sense, additional final samples were then analyzed by EMAX, and the TPH concentrations were reported to be similar to those measured earlier. This suggested that different lab techniques may be resulting in widely different measurements of TPH. In the current research, possible reasons for these discrepancies were explored, such as GC integration problems caused by drifting baselines and interference from the presence of NOM. Ordinarily these problems are not expected to be significant, but at the very low TPH concentrations of the samples from Area IV, these problems may have significantly impacted the analysis of TPH.

Another interesting finding at the end of the bioremediation study was the identification of natural oils, such as oleic acid and palmitic acid, in a soil sample from the end of the soil microcosm experiment. A preliminary chemical analysis using gas chromatography/mass spectrometry (GC/MS) indicated significant amounts of natural organic material (NOM) which is likely to be counted as total petroleum hydrocarbon (TPH) and/or extractable fuel hydrocarbons (EFH) in standard tests for petroleum hydrocarbons. From the GC/MS analysis of one of the final bioremediation soil samples, the larger peaks in the chromatogram were associated with organic acids, while the smaller peaks were attributed to weathered petroleum hydrocarbons (Figure 1-1). The interference from NOM could potentially result in reported TPH/EFH soil concentrations higher than that attributable to hydrocarbons from petroleum-based origins. It is therefore important to 1) understand the actual composition of the material thought to be petroleum hydrocarbons so that clean-up goals can be set accordingly, and 2) develop analytical methods to independently quantitate petroleum hydrocarbons with minimal interference from NOM in the soil.

A common method to remove polar organic compounds from soil extracts prior to TPH analyses is a cleanup procedure using silica gel fractionation. However, it is not certain if this method is accurate for removing NOM (Mujis and Jonker, 2009). Thus, another objective of this study was to investigate the use of silica gel cleanup for Area IV soils, specifically to determine if this methods effectively removes NOM before TPH analysis.

To address the issues described above, a series of laboratory investigations of soils from SSFL were conducted. TPH quantitation methods were tested and chemical composition was investigated for five fresh soil samples collected from Area IV to provide representative samples for testing the relative quantities of TPH and NOM in the soil. Cal Poly researchers worked with CDM Smith staff to identify the five sites for soil collection. The specific objectives listed below were addressed by this research:

1. Characterize chemical constituents in the five soil samples using GC/MS to analyze for NOM, as well as hydrocarbons and PAHs.

2. Quantify NOM relative to petroleum hydrocarbons.

3. Evaluate the standard silica gel cleanup protocol to separate NOM from petroleum hydrocarbons – determine if silica gel cleanup removes the NOM in Area IV soils and leaves intact the petroleum hydrocarbons and PAHs.

4. Interpret site-wide TPH/EFH analytical results with silica gel prep.

This study also examined the look-up table values for TPH set by the DTSC in terms of the technical feasibility of accurate TPH measurement at these low concentrations. The DTSC created a lookup table with cleanup goals for each of the soil contaminants at Area IV, and set a lookup table value for TPH of 5 mg/kg for the C15-C20 equivalent carbon range. However, the DSTC stated that “for locations where TPH is the sole contaminant, a cleanup strategy will be considered based on the findings of the soil treatability study” (DTSC 2013). One of the objectives of this follow-on study is therefore to make conclusions regarding the measurement of TPH which may inform future clean-up standards for TPH at Area IV.

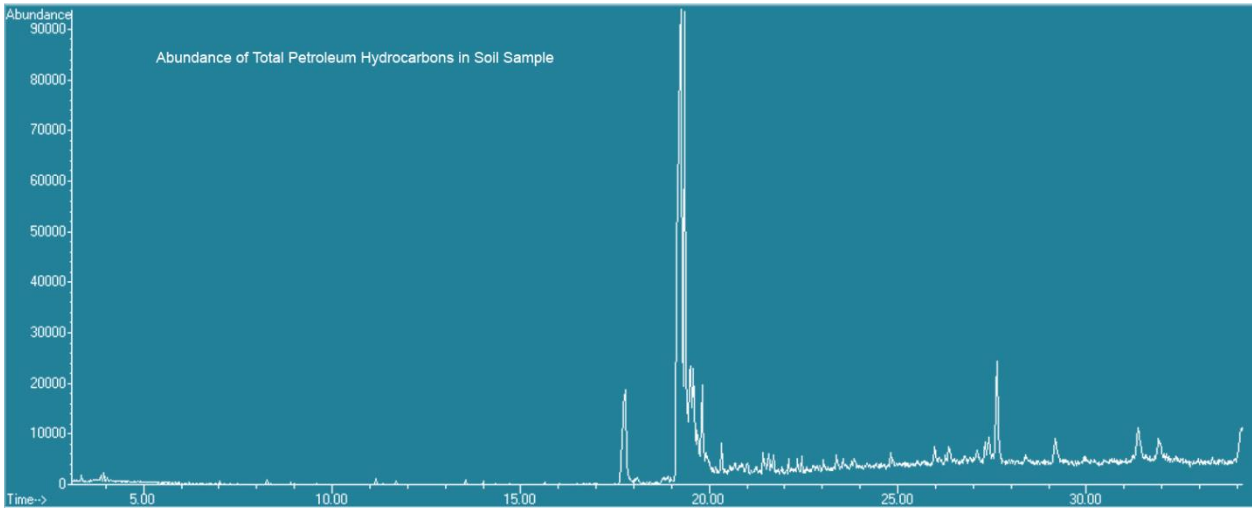

Figure 1-1. Analysis of a soil sample from the end of the bioremediation microcosm experiment in the soil treatability study.

2.0 Background

2.1 Regulatory TPH clean-up levels

The USEPA originally exempted petroleum hydrocarbons from CERCLA regulation, and as a result states developed their own requirements for clean-up levels. These requirements were usually presented as concentration of TPH, and ranged from 10 to over 10,000 mg TPH/kg soil (TPHCWG, 1998). The Total Petroleum Hydrocarbon Criteria Working Croup (TPHCWG) was formed in 1993 based on this observation that widely different clean-up requirements were being used by states at sites that were contaminated with petroleum compounds. The TPHCWG members recognized that these numerical standards were not based on scientific assessment of risk, and therefore recommended a new approach utilizing appropriate fraction-specific toxicity data. More recently, the State of Alaska conducted a survey of all 50 states and Canada and found that most states were by then using risk-based cleanup levels, but still found a wide range of TPH cleanup levels (ADEC 2013). For residual range oil (RRO), which is typical of SSFL soil contamination, the cleanup levels varied from 99 mg/kg (Texas) to 10,000 mg/kg (Utah). In comparison, the look-up table value established by the DTSC for Area IV at SSFL is only 5 mg/kg for TPH in the C15-C20 equivalent carbon range (DTSC 2011). However, as stated above, the DSTC noted that the final cleanup strategy should be based on the findings of the soil treatability study. In this final phase of the soil treatability studies, it is thus important to determine if the 5 mg/kg cleanup level can be measured accurately.

2.2 GC/MS analysis of petroleum hydrocarbons

TPH is most often measured using gas chromatography with a flame ionization detector (GC/FID). However, gas chromatography with a mass spectrometer detector was used in the present study to provide more detailed identification of specific compounds in the unresolved complex mixture typically observed for petroleum compounds in Area IV soils. The MS detector allows for identification of specific compounds by comparison of mass spectra to a library of 250,000 specific compounds. However, mass spectroscopy has some limitations, and it should be used with caution for identification of compounds in complex mixtures such as petroleum products (TPHCWG, 1998). It should also be noted that GC/FID is better for quantifying TPH than GC/MS because the response of an FID is proportional to the mass of hydrocarbon present regardless of the type of hydrocarbon (e.g., aromatic, aliphatic or olefin). In contrast, the MS detector may have different responses for different types of hydrocarbon compounds (TPHCWG, 1998). In this study, the GC/MS was primarily used for diagnostic purposes to roughly characterize the residual “TPH” and determine if some of the compounds eluting with the TPH are actually natural organic material.

2.3 Silica gel treatment to remove lipids

An important limitation of the standard method for measuring TPH is that the entire chromatogram of the unresolved complex mixture is integrated, and thus non-petroleum hydrocarbons, such as soil or sediment organic matter (i.e., degradation products of plants and animals), or lipids may be inadvertently included in the measurement of TPH. If not properly removed, these compounds can cause an overestimation of TPH concentrations, as they will contribute to the overall detector response by FID or MS. Therefore, "appropriate sample clean-up is of great importance when determining TPH concentrations in environmental matrixes" (Mujis and Jonker, 2009). The most common cleanup technique used on extracts for TPH analyses is silica-gel cleanup using EPA Method SW-846 3630 (TPHCWG, 1998). Mujis and Jonker (2009) evaluated this silica gel cleanup method and found 84% TPH recovery and 59% removal of lipids from Dutch sediment samples.

3.0 Methodology

3.1 Soil collection

Fresh soil samples were collected from five locations of Area IV at the SSFL site (Table 3-1). Soil sample collection was conducted by Hazardous Waste Operations and Emergency Response (HAZWOPER)-certified field personnel per 29 CFR 1910.120. Soils were collected with decontaminated stainless steel instruments. Splits of each sample were sent to Cal Poly and EMAX Laboratory.

Table 3-1. Legend for soil sample identification

Soil Sample Number	Sample ID
1	5D-612
2	5D-885
3	01-BE-B
4	02-LS-C
5	18-B-A

3.2 Extraction and TPH analytical methods

Extraction:

The TPH extraction protocol was adapted from EPA Standard Method #3550. Ten to 25-g samples of each soil sample were placed in a 100-mL sample bottle. Approximately 30 mL of methylene chloride (MeCl) was added to the sample bottle containing the soil, and the sample was sonicated for 3 min at 60,000 Hz using a Sonifier 250 (Branson Ultrasonics Corp., Danbury, Connecticut). Sodium sulfate (Na2SO4) was added to a 24-cm (diameter) 802 Fluted Grade Whatman Inc. filter mounted in a glass funnel to remove water from the extracts. Na2SO4 was also added directly to the sample bottle. The extract was poured through the fluted filter and then through a Millipore API 04200 glass fiber filter into a test tube. Another 25 mL of MeCl was added to the soil sample. This additional solvent was then sonicated and filtered as described above and added to the previous 25 mL of MeCl for a total extract volume of 50 mL.

Each sample extract was transferred to a Turbovap® tube and concentrated using a Turbovap® nitrogen evaporation system in a room temperature (20 - 25°C) water bath to an approximate volume of 2 mL. The sample was transferred into a 10-mL graduated cylinder, and the volume was brought up to 5 mL with additional MeCl. These extracts were poured into two GC vials with crimp caps for GC analysis.

GC/MS analysis:

Samples from each extract were run through an Agilent Technologies 6890N Gas Chromatograph (splitless inlet) with an Agilent 5975B inert Mass Selective Detector. A 50-m fused silica column 250-µm in diameter was used (Agilent Catalog #19091S-433) in the chromatograph. Samples were automatically loaded using an Agilent 7683B Series Injector capable of holding eight GC vials, two solvent vials, and a waste vial. The sample injection volume was 2 µL from a 10-µL syringe. To ensure no samples were cross-contaminated, the injector was programed to rinse the syringe twice with methylene chloride before taking sample extract from the GC vial. The temperature ramped from 45°C to 275°C at a rate of 12°C per minute and was then held at 275°C for the remainder of the 34-min run. The GC front inlet was pressurized to 12.26 psi at a temperature of 200°C. Helium was used as the carrier gas.

Each chromatogram was integrated over the appropriate range of elution times, and the resulting area was used to calculate the TPH concentration in the solvent extract using a calibration curve derived from known standards.

Standard curves:

Calibration curves were prepared using SAE-30 motor oil, because the apparent equivalent carbon range matched that of the soil sample extracts. For the motor oil standards, 0.0250 g of SAE 30 motor oil was weighed out in a small beaker and rinsed into a 50-mL volumetric flask using MeCl. The flask was then filled to volume with methylene chloride, for a final solution with 5000-mg/L motor oil in MeCl. Standard dilutions were then prepared from the 5000-mg/L stock solution. A sample calibration curve is shown in Figure 3-1.

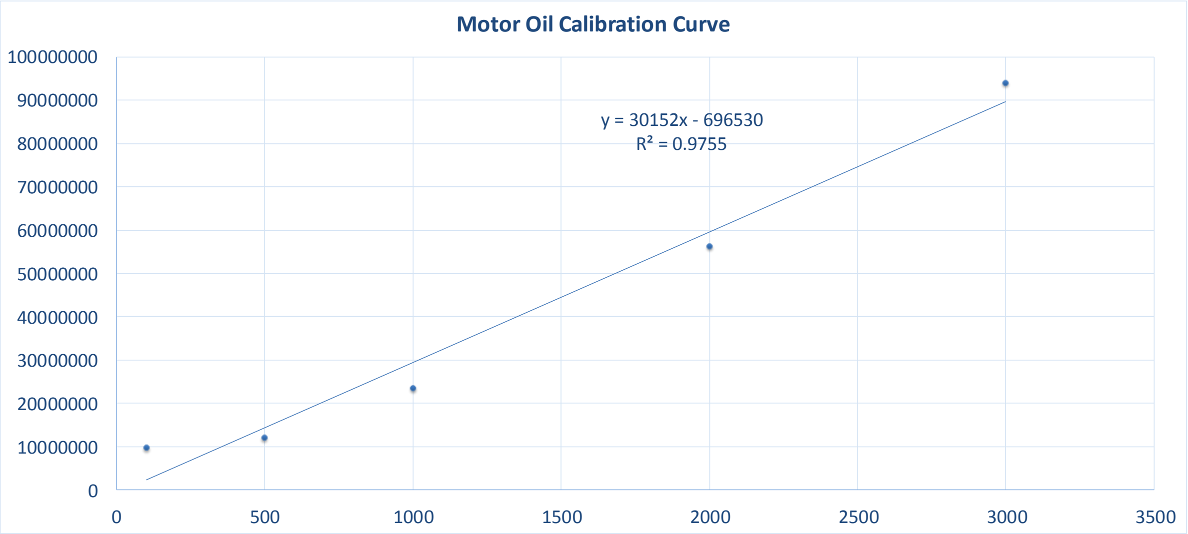

Figure 3-1. Sample calibration curve for TPH measurement in MeCl extracts.

3.3 Silica gel technique

The MeCl extracts from each soil sample (before concentrating) were run on a silica gel column as a preparatory method to remove polar compounds. Chromatographic-grade silica gel (100-200 mesh, Fisher Scientific S817-1) was baked overnight at 130°C and placed in a 10 in x 10.5 mm ID glass chromatographic column (Ace Glass, Inc 5906-05). After rinsing the column with MeCl, it was filled with MeCl and a glass wool plug was pushed into the bottom with a glass rod. The column was packed with 11.0 g ± 0.1 g of activated silica gel as a slurry. The slurry was created by adding approximately 5 mL of methylene chloride. The slurry was transferred, with rinsing, into the column containing glass wool. The sample in MeCl was poured through the column, and the eluent collected in a glass flask. The sample was carefully transferred to a Turbovap® tube and concentrated using a Turbovap® nitrogen evaporation system in a room temperature (20 - 25°C) water bath to an approximate volume of 2 mL. The sample was transferred into a 10-mL graduated cylinder, and the volume was brought to 5 mL with additional MeCl. The samples were stored in the freezer and analyzed by GC/MS as described above.

4.0 Results

4.1 TPH analysis of five soil samples

Results of TPH analyses for duplicate soil samples in the Cal Poly Lab by GC/MS are compared to TPH results reported by EMAX using GC/FID in Table 4-1 (detailed results from EMAX are in Appendix A). High variability was observed between the Cal Poly duplicate analyses, with relative standard deviations ranging from 13 to 82%. The variation between Cal Poly TPH results and those of EMAX was even greater, with percent differences ranging from -5% to +256%. Note that the difference between TPH concentrations previously measured by Lancaster Laboratories (another commercial laboratory) and EMAX (as described in Section 2.4) were much greater than the differences between TPH concentrations measured by Cal Poly and EMAX. For Sample 1, the TPH concentration measured by EMAX was non-detect, but it was 56 and 208 mg/kg for replicate analyses at Cal Poly conducted on two different soil aliquots of Sample 1.

The high variability in measured TPH concentrations may be attributed to soil heterogeneity as well as inherent difficulties of measuring such low TPH concentrations. Soil Sample 1 contained small rocks about 2-8 mm in size, and the presence of these rocks could result in soil aliquots with varying amounts of fine soil for which TPH concentrations are expected to be highest. Also, what appeared to be small tar balls had been previously observed in bulk soil collected for the bioremediation and phytoremediation soil treatability studies. These small tar balls would be expected to cause large differences in TPH concentrations measured for samples containing tar balls vs. sample aliquots with no tar balls.

Several difficulties were encountered during the GC quantification of TPH at the low soil concentrations of these samples. First, the petroleum compounds appear to be of very high molecular weight, in the residual range oil (RRO) range (see for example Figure 4-1A below). Since the equivalent carbon range was so high, the baseline of the GC did not return to zero at the end of each run. This makes it very difficult to decide where to set the baseline for integration of total peak area for the unresolved peaks in the chromatogram. Second, the detector signal for the solvent (methylene chloride) was significant, causing a wide solvent shoulder extending 10 to 15 minutes into the chromatogram. Third, solvent blanks often exhibited peak areas similar to those of the low concentration samples and the low-concentration standards. Fourth, the r-squared for the standard curve was only about 0.97, which is lower than the value of 0.99 typically observed for standards with higher concentrations. At higher TPH concentrations all of these effects would be insignificant, but at very low TPH concentrations these effects could result in poor reproducibility and possibly even anomalous results.

Table 4.1. TPH results from Cal Poly Lab (GC/MS) compared to TPH results of EMAX commercial laboratory

Sample Number	Sample ID	TPH Concentration (mg/kg)						Difference between Cal Poly and EMAX (%)
		EMAX	Cal Poly Lab					
			Rep 1	Rep 2	Average	Std. Dev.	Rel. Std. Dev. (%)	
1	5D-612	ND	208	56	132	108	82	+ ∞
2	5D-885	50	212	41	127	121	95	+154
3	01-BE-B	170	147	176	161	21	13	-5
4	02-LS-C	17	34	87	60	37	62	+256
5	18-B-A	190	219	316	268	69	26	+41

4.2 Effect of silica gel cleanup on TPH concentrations

Chromatograms for GC/MS analysis of the five soil samples with and without silica gel cleanup are shown in Appendix B, Figures B-1 through B-5. There were no obvious specific peaks removed by the silica gel clean-up in these chromatograms. However, a more detailed analysis of peaks associated with NOM is given below in Section 4.3.

TPH concentrations measured with and without silica gel cleanup are shown in Table 4.1. For the EMAX analyses, the TPH concentrations were 5 to 40% lower after silica gel cleanup. This suggests that polar NOM may have been removed by the silica gel cleanup method, but this is difficult to prove based on the Cal Poly chromatograms in Figures 4-1 through 4-5. For the Cal Poly analyses, the silica gel cleanup gave mixed results, with somewhat higher TPH concentrations observed after silica gel cleanup for 3 out of the 5 samples. One Cal Poly sample was the same with and without silica gel cleanup, and one sample decreased in TPH concentration after silica gel cleanup.

Table 4.2. Effect of silica gel cleanup on measured TPH concentrations by EMAX and Cal Poly

Soil Sample	Soil Sample Location	TPH Concentration (mg/kg)			
		EMAX Results		Cal Poly Results	
		Before Silica	After Silica	Before Silica	After Silica
1	5D-612	-	-	208	209
2	5D-885	50	43	212	99
3	5B- 01-BE-B	170	150	147	168
4	5B-02-LS-C	17	10	34	101
5	5B-18-CB-A	190	180	219	262

4.3 Identification and quantification of NOM and petroleum compounds

Specific compounds eluting in the GC analyses of the soil samples with and without silica gel cleanup were identified using their MS spectra. The Agilent GC/MS software has a library of 250,000 compounds used to match mass spectra to the peaks identified in the chromatograms. The software provides an estimate of the probability of a good match between the compounds eluting from the GC column and the stored model compounds in the library. A summary of the compounds observed in each chromatogram is given in Table 4-3, and chemical structures of 64 different potential compounds are shown in Appendix C along with their mass spectra. Match probabilities are shown in Table 4.3. It is important to remember that the mass spectra are not necessarily perfect matches for specific compounds, but this analysis provides a general snapshot of the types of compounds detected in the soil samples which are being counted as part of the "TPH."

Many of the compounds identified by their mass spectra were highly branched and cyclic hydrocarbons (Appendix C, Figures C- 13, 30, 33, 34, 39-42, 44, 45, 56-58, 60, 61 and 63). Such compounds are typical of RRO because these hydrocarbons are resistant to biodegradation. A variety of PAHs were also identified by their mass spectra (Appendix C, Figures C- 3, 12, 15-17, 20-22, 25, 26, 37 and 52). Many of the hydrocarbons identified were partially oxidized (Appendix C, Figures C- 2, 5, 14, 18, 19, 23, 24, 27-29, 31, 43, 62 and 64). There were also organic acids identified, such as n-decanoic acid (Figure C-35), stearic acid (Figure C-48), and oleic acid (Figure C-49). These organic acids could be naturally-occurring organic compounds from plant sources (NOM) or degradation products of hydrocarbons. Other oxidized compounds observed which could be considered NOM include nonanal (Figure C-46) and an oxalic acid derivative (Figure C-50). There were also some heterocylic compounds identified (Figures C- 4, 6, 8, 51, 53 and 54), but these were relatively rare. A couple of silicon-based compounds were observed (Figures C- 9-11) which may be coming from the column packing.

From the summary of compounds found in Table 4.3, it can be seen that only a few compounds, such as oleic acid, were removed by the silica gel cleanup. Oxalic acid derivatives were partially removed by the silica gel cleanup. PAHs and branched/cyclic hydrocarbons were not removed by the silica gel cleanup. It can be cautiously concluded that the silica gel cleanup removes part of the NOM, but not all of it, and that compounds typically associated with TPH, such as hydrocarbons and PAHs are not removed.

The quantitative contribution of NOM to the total TPH measured was estimated by integrating peaks of compounds thought to be NOM. About 3 to 8% of the TPH was attributed to NOM using this method (Table 4.4). For Soil Samples 1-3, the silica gel cleanup did not reduce the contributions of NOM, but for Soil Samples 4 and 5 all of the NOM appeared to be removed by the silica gel cleanup.

Table 4-3. Summary of common compounds identified in soil extracts using mass spectroscopy – with and without silica gel cleanup. Figure numbers refer to mass spectra and chemical structures shown in Appendix C.

	Sample 1, no Silica Gel Prep				**Sample 1, Silica Cleanupp**		
Elution Time	Compound	Concentration (mg/kg)	Probability (%)	Elution Time	Compound	Concentration (mg/kg)	Probability (%)
22.33	Sulfurous Acid/Oxalic Acid	5.64	8.5	6.14	Cyclopentane	1.73	55
23.04, 23.85	Oxalic Acid	6.67	30	6.64	Branched Cyclohexane (*Figure C-33)*	0.32	26.3
25.41	Large oxidized compound (*Figures C-58, C-62*)	6.03	35	7.3, 8.89, 10.2	Branched Heptane *(Figures C-39, 40, 63)*	3.38	55
27.1	28-Nor-1 7B(H)-hopane	9.12	53.3	22.33	Oxalic Acid	0.81	15
27.42	Large Organic Compound, Cl poss (*Figure C-59)*	6.66	10	23.85	Branched Tetracontane/ Oxalic Acid	1.06	6/ 3.62
28.4-32.4	Large Cyclic Organics, Cl & Br poss (Figure C-57)	46.53	5 to 10	25.4	Large Oxidized Organic *(Figures 58, 62)*	0.85	50
				27.1	28-Nor-1 7B(H)-hopane	3.29	15
				27.4	Heptacosane	2.52	16
	Sample 2, no Silica Gel Prep				**Sample 2, Silica Cleanup**		
Elution Time	Compound	Concentration (mg/kg)	Probability (%)	Elution Time	Compound	Concentration (mg/kg)	Probability (%)
6.6	C8H17N2O (*Figure C-6)*	7.32	91.8	7.3	Branched Heptane (*Figures C-39, 40, 63)*	7.93	35
9.59	Decamethyl Cyclopentasiloxane	7.25	63.1	9.59	Quinolinedione	6.00	79
28.1	Oxalic Acid	10.43	50	22.35, 24.7, 27.4	Oxalic Acid	7.13	30
29.48	Bacchotricuneatinc	7.39	50				
23.5-30	Piperidinone	10.73	10 to 25				
	Sample 3, no Silica Gel Prep				**Sample 3, Silica Cleanup**		
Elution Time	Compound	Concentration (mg/kg)	Probability (%)	Elution Time	Compound	Concentration (mg/kg)	Probability (%)
9.59	Chloro ethanol acetate	8.17	25	7.3, 8.89, 10.2, 11.4	Branched Heptane	7.81	55
18.67	Cyclic Octatomic Sulfur	8.32	98.3	18.67	Cyclic Octoatomic Sulfur	5.68	97
18.81,19.24	Pyrene	11.12	65	18.81, 19.24	Pyrene/ Fluyoranthene	9.98	50
21.65-21.75	PAHs	10.55	50	21.65-21.75	PAHs (*Figures C-3, 15, 16, 17, 20, etc.)*	10.86	75
24.0-25.1	Benzo-k-fluoranthene	11.13	33	24-25.1	Benzo-k-Fluoranthene	10.18	35
26.5-28.5	oxalic acid/octadecanoic acid	13.50	10 to 20				
	Sample 4, no Silica Gel Prep				**Sample 4, Silica Cleanup**		
Elution Time	Compound	Concentration (mg/kg)	Probability (%)	Elution Time	Compound	Concentration (mg/kg)	Probability (%)
21.69	2,4,6,8 Tetramethyl-Undecene	3.79	44.4	7.29	Heptane, 2,2,4,6,6-pentamethyl	8.96	42.3
22.34	Oleic Acid	3.92	9.71	7.87, 8.18	Methylene Choride	6.07	80
24	Benzo-e-pyrene	3.88	34.2	8.89, 10.19, 11.4	Dimethyl Heptane	6.74	30.6
24.71, 24.85, 25.11	Benzo-k-fluoranthene/Perylene	5.25	30.5	17.08	Phthalic Acid	5.88	~40
28.4-30.22	Benzo-ghi-perylene	6.20	65.6-77.1	17.87-17.9	Ethaneperoxic Acid	5.85	76.2
				22.33	Hexylundecylester Sulfurous Acid	6.00	49-54.5
				23.99-25.11	Benzo-k-fluoranthene	17.44	75.5
				29.0-30.2	Benzo-ghi-perylene	11.23	67.8
				26.5-27.1	Cyclic hydroxy-ethyl compound	7.26	50
	Sample 5, no Silica Gel Prep				**Sample 5, Silica Cleanup**		
Elution Time	Compound	Concentration (mg/kg)	Probability (%)	Elution Time	Compound	Concentration (mg/kg)	Probability (%)
16.47	Fluorene	7.70	56	7.3	Branched Heptane (*Figures 39, 40, 63)*	7.96	36.7
18.82.19.24	Fluoranthene/Pyrene	13.79	50	16.47	Fluorene	6.13	54
21.65,21.71	Triphenylene (PAHs)	23.24	40	18.82,19.24	Pyrene/ fluoranthene	9.58	50
24-25.1	Benzo-k-fluoranthene	28.86	20	21.65-21.75	PAHs (*Figures 3, 15, 16, 17, 20, etc.)*	21.57	40
29-30.2	Benzo-ghi-pyrylene	9.99	60-70	24,24.7,24.85	Benzo-k-fluoranthene	23.43	50
				29-30.2	Benzo-ghi-perylene	9.18	60-70

Table 4.4. Estimated Contribution of NOM to Measured TPH of the Soil Samples With and Without Silica Gel Cleanup.

	Without Silica Cleanup		With Silica Cleanup	
Soil Sample	Concentration NOM (mg/kg)	Percent of TPH	Concentration NOM (mg/kg)	Percent of TPH
1	9.3	4.03	9.0	4.02
2	16.8	6.89	9.0	7.14
3	9.1	4.95	6.6	3.45
4	4.0	7.94	0.0	0
5	7.4	2.93	0.0	0

4.4 Site-wide implications

Important implications of this study which are applicable to interpretation of site-wide TPH soil concentrations can be drawn from the results of the silica gel cleanup investigation and the general problems identified for TPH analysis of Area IV soils with low TPH concentrations. Results of this study show that the silica gel cleanup is an appropriate tool for increasing the accuracy of TPH measurements. Even more significant are the findings that accurate measurement of TPH concentrations is very difficult at low concentrations.

The silica gel cleanup used by EMAX for many TPH analyses site-wide can significantly lower TPH concentrations. Based on the GC/MS analyses reported here, this cleanup technique is appropriate because it appears to remove only polar organic compounds and not PAHs or petroleum hydrocarbons. Further, these results suggest that the removal of such polar compounds may not be complete, and therefore actual TPH concentrations should be even lower than those determined with the silica gel cleanup.

The GC/MS chromatograms show that the residual petroleum compounds in Area IV soils exhibit very high molecular weight consistent with residual-range organics (RRO). The RRO is similar to asphalt (bitumen). Such high molecular weight material gives GC signals outside the normal C-8 to C-40 equivalent carbon range, and this complicates TPH measurement because it is difficult to integrate the total unresolved complex mixture when the final signal is not zero – this is because it is difficult to decide what background level to use for an integrating baseline. See for example the chromatogram for Sample 1 (Appendix B, Figure B-1A) which shows a relatively high GC signal at the end of the GC run.

Setting TPH clean-up levels near background levels is problematic because of the inherent difficulties of measuring TPH concentrations near background concentrations. At these low TPH concentrations, effects such as drifting baselines, TPH in sample blanks, and solvent fronts become significant. These effects are ordinarily insignificant when measuring higher TPH concentrations. As described in Section 2.1, clean-up levels for RRO in the U.S. vary from

99 mg/kg in Texas to 10,000 mg/kg in Utah (ADEC 2013). States typically set their cleanup goals at 5,000 mg/kg for soils. Thus, the look-up table values for TPH at Area IV are about 20 times lower than the lowest state standard and 1,000 times lower than typical values.

Another observation from the bioremediation study is that the petroleum compounds sometimes appear to be concentrated in small tar balls in the soil as opposed to homogeneously dispersed in the soil. Such tar balls were observed in the bulk soil collected from Area IV for the bioremediation study using laboratory microcosms as well as the phytoremediation study. The presence of these tar balls would be expected to cause high variability of TPH measurements because one subsample may contain a tar ball while another does not.

5.0 Conclusions

The GC/MS spectral analyses suggest that the remaining TPH contamination of Area IV soils consists of high molecular weight hydrocarbons as expected for RRO or asphalt. These hydrocarbons are mostly highly-branched and cyclic hydrocarbons which are known to be slow to biodegrade. PAHs are also present in the soils, as well as some heterocyclic compounds. Many of the branched hydrocarbons and PAHs appeared to be partially oxidized. Also detected in the soil samples were organic acids such as oleic acid and oxalic acid, suggestive of NOM. Such compounds also can be degradation products of hydrocarbon biodegradation. This NOM fraction accounted for about 5 to 8% of the total TPH. NOM appeared to account for a larger fraction of the TPH in our preliminary analysis of a soil from the bioremediation microcosm experiment.

Silica gel cleanup by EMAX Laboratory removed 5 to 40% of the TPH in the five soil samples. Mixed results were obtained in the Cal Poly lab, with silica gel cleanup actually increasing the TPH for three of the five samples. The silica gel cleanup was found to remove some of the polar organic compounds, but not all of them. Thus, the silica gel approach may underestimate the contribution of NOM to TPH measurement. PAHs and large branched hydrocarbons were not removed by the silica gel cleanup.

An important observation during this study is that it is difficult to accurately quantify TPH at the very low soil TPH concentrations of the Area IV soils used in this study (100 to 300 mg/kg). Reliable TPH measurement near background TPH levels or near the 5 mg/kg look-up table value for Area IV would be nearly impossible. At these very low TPH concentrations several factors can significantly impact the calculation of TPH concentrations from GC data, making the calculated concentrations unreliable. TPH concentrations are calculated by integrating the area under numerous overlapping peaks, and thus a baseline has to be drawn underneath the peaks for the integration. Drifting baselines, non-zero signal at the end of the elution time, and non-zero blanks all can make the calculation of TPH inaccurate at low TPH concentrations. At higher TPH concentrations, these effects become much less significant.

The look-up table value published by the DTSC for TPH is 5 mg/kg, which is 20 times lower than any state standard for RRO, and 1,000 times lower than typical values used by many states. Most states have now gone to risk-based clean-up goals (ADEC, 2011). It is beyond the scope of this study to argue for a change in clean-up goals, however, this study has demonstrated that there are clearly technical problems with measuring TPH concentrations at such low levels.

6.0 References

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Appendix A: Detailed EMAX TPH results for five soil samples showing equivalent carbon ranges.

All analyses conducted on April 22, 2015.

Table A-1: EMAX results for TPH analyses without silica gel clean-up

Soil Sample ID	Percent moisture	Silica Cleanup?	Equivalent Carbon Range	TPH Concentration (mg/kg)
SSFL-EFH-SO-612-SA5D-0.5-1.0	7.7	NO	(C12-C14)	
SSFL-EFH-SO-612-SA5D-0.5-1.0	7.7	NO	(C15-C20)	
SSFL-EFH-SO-612-SA5D-0.5-1.0	7.7	NO	(C21-C30)	
SSFL-EFH-SO-612-SA5D-0.5-1.0	7.7	NO	(C30-C40)	
SSFL-EFH-SO-612-SA5D-0.5-1.0	7.7	NO	(C8-C11)	
SSFL-EFH-SO-612-SA5D-0.5-1.0	7.7	NO	TOTAL (C8-C40)	
SSFL-EFH-SO-885-SA5D-0.5-1.0	4.7	NO	(C12-C14)	
SSFL-EFH-SO-885-SA5D-0.5-1.0	4.7	NO	(C15-C20)	
SSFL-EFH-SO-885-SA5D-0.5-1.0	4.7	NO	(C21-C30)	13
SSFL-EFH-SO-885-SA5D-0.5-1.0	4.7	NO	(C30-C40)	37
SSFL-EFH-SO-885-SA5D-0.5-1.0	4.7	NO	(C8-C11)	
SSFL-EFH-SO-885-SA5D-0.5-1.0	4.7	NO	TOTAL (C8-C40)	50
SSFL-EFH-SO-01-BE-B-SA5B-0.5-1.0	3.4	NO	(C12-C14)	
SSFL-EFH-SO-01-BE-B-SA5B-0.5-1.0	3.4	NO	(C15-C20)	2.9
SSFL-EFH-SO-01-BE-B-SA5B-0.5-1.0	3.4	NO	(C21-C30)	77
SSFL-EFH-SO-01-BE-B-SA5B-0.5-1.0	3.4	NO	(C30-C40)	91
SSFL-EFH-SO-01-BE-B-SA5B-0.5-1.0	3.4	NO	(C8-C11)	
SSFL-EFH-SO-01-BE-B-SA5B-0.5-1.0	3.4	NO	TOTAL (C8-C40)	170
SSFL-EFH-SO-02-LS-C-SA5B-0.5-1.0	8.1	NO	(C12-C14)	
SSFL-EFH-SO-02-LS-C-SA5B-0.5-1.0	8.1	NO	(C15-C20)	
SSFL-EFH-SO-02-LS-C-SA5B-0.5-1.0	8.1	NO	(C21-C30)	4
SSFL-EFH-SO-02-LS-C-SA5B-0.5-1.0	8.1	NO	(C30-C40)	13
SSFL-EFH-SO-02-LS-C-SA5B-0.5-1.0	8.1	NO	(C8-C11)	
SSFL-EFH-SO-02-LS-C-SA5B-0.5-1.0	8.1	NO	TOTAL (C8-C40)	17
SSFL-EFH-SO-18-CB-A-SA5B-0.5-1.0	2.9	NO	(C12-C14)	
SSFL-EFH-SO-18-CB-A-SA5B-0.5-1.0	2.9	NO	(C15-C20)	7.8
SSFL-EFH-SO-18-CB-A-SA5B-0.5-1.0	2.9	NO	(C21-C30)	100
SSFL-EFH-SO-18-CB-A-SA5B-0.5-1.0	2.9	NO	(C30-C40)	83
SSFL-EFH-SO-18-CB-A-SA5B-0.5-1.0	2.9	NO	(C8-C11)	
SSFL-EFH-SO-18-CB-A-SA5B-0.5-1.0	2.9	NO	TOTAL (C8-C40)	190

Table A-2: EMAX results for TPH analyses with silica gel clean-up

Soil Sample ID	Percent moisture	Silica Cleanup?	Equivalent Carbon Range	TPH Concentration (mg/kg)
SSFL-EFH-SO-612-SA5D-0.5-1.0	7.7	YES	(C12-C14)	
SSFL-EFH-SO-612-SA5D-0.5-1.0	7.7	YES	(C15-C20)	
SSFL-EFH-SO-612-SA5D-0.5-1.0	7.7	YES	(C21-C30)	
SSFL-EFH-SO-612-SA5D-0.5-1.0	7.7	YES	(C30-C40)	
SSFL-EFH-SO-612-SA5D-0.5-1.0	7.7	YES	(C8-C11)	
SSFL-EFH-SO-612-SA5D-0.5-1.0	7.7	YES	TOTAL (C8-C40)	
SSFL-EFH-SO-885-SA5D-0.5-1.0	4.7	YES	(C12-C14)	
SSFL-EFH-SO-885-SA5D-0.5-1.0	4.7	YES	(C15-C20)	
SSFL-EFH-SO-885-SA5D-0.5-1.0	4.7	YES	(C21-C30)	21
SSFL-EFH-SO-885-SA5D-0.5-1.0	4.7	YES	(C30-C40)	22
SSFL-EFH-SO-885-SA5D-0.5-1.0	4.7	YES	(C8-C11)	
SSFL-EFH-SO-885-SA5D-0.5-1.0	4.7	YES	TOTAL (C8-C40)	43
SSFL-EFH-SO-01-BE-B-SA5B-0.5-1.0	3.4	YES	(C12-C14)	
SSFL-EFH-SO-01-BE-B-SA5B-0.5-1.0	3.4	YES	(C15-C20)	
SSFL-EFH-SO-01-BE-B-SA5B-0.5-1.0	3.4	YES	(C21-C30)	73
SSFL-EFH-SO-01-BE-B-SA5B-0.5-1.0	3.4	YES	(C30-C40)	73
SSFL-EFH-SO-01-BE-B-SA5B-0.5-1.0	3.4	YES	(C8-C11)	
SSFL-EFH-SO-01-BE-B-SA5B-0.5-1.0	3.4	YES	TOTAL (C8-C40)	150
SSFL-EFH-SO-02-LS-C-SA5B-0.5-1.0	8.1	YES	(C12-C14)	
SSFL-EFH-SO-02-LS-C-SA5B-0.5-1.0	8.1	YES	(C15-C20)	
SSFL-EFH-SO-02-LS-C-SA5B-0.5-1.0	8.1	YES	(C21-C30)	3.3
SSFL-EFH-SO-02-LS-C-SA5B-0.5-1.0	8.1	YES	(C30-C40)	6.9
SSFL-EFH-SO-02-LS-C-SA5B-0.5-1.0	8.1	YES	(C8-C11)	
SSFL-EFH-SO-02-LS-C-SA5B-0.5-1.0	8.1	YES	TOTAL (C8-C40)	10
SSFL-EFH-SO-18-CB-A-SA5B-0.5-1.0	2.9	YES	(C12-C14)	
SSFL-EFH-SO-18-CB-A-SA5B-0.5-1.0	2.9	YES	(C15-C20)	7.2
SSFL-EFH-SO-18-CB-A-SA5B-0.5-1.0	2.9	YES	(C21-C30)	82
SSFL-EFH-SO-18-CB-A-SA5B-0.5-1.0	2.9	YES	(C30-C40)	86
SSFL-EFH-SO-18-CB-A-SA5B-0.5-1.0	2.9	YES	(C8-C11)	
SSFL-EFH-SO-18-CB-A-SA5B-0.5-1.0	2.9	YES	TOTAL (C8-C40)	180

Appendix B. Cal Poly chromatograms of soil extracts with and without silica gel cleanup

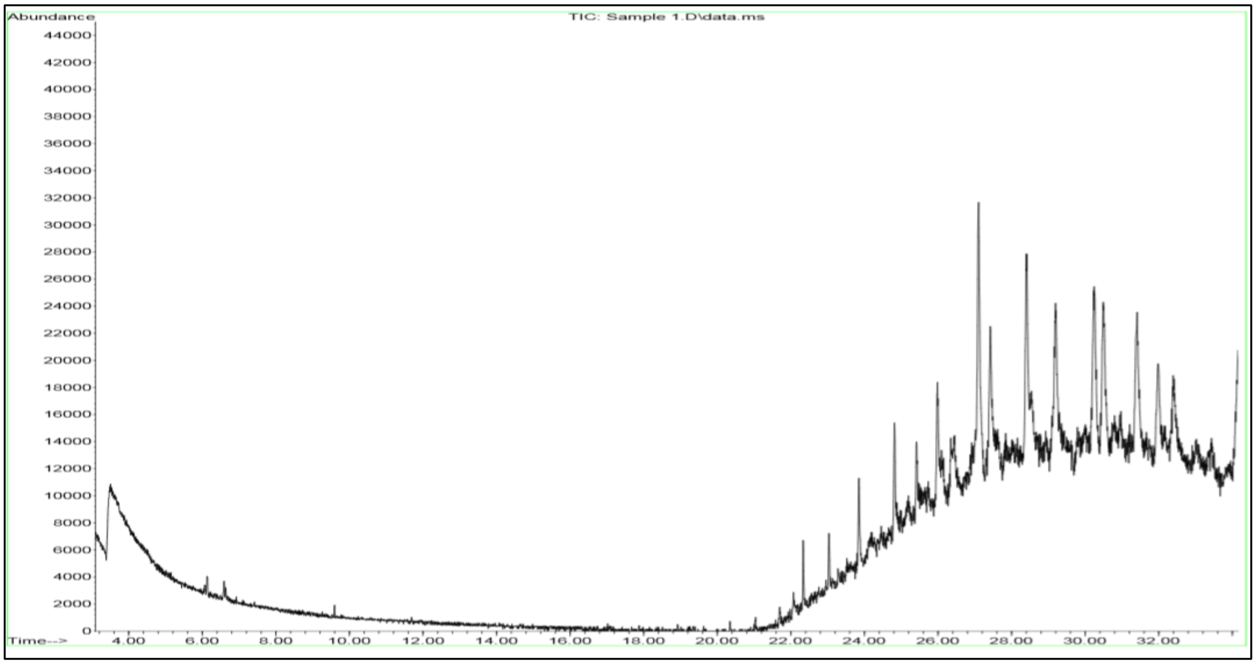


Figure B-1A: Sample 1 (5D-612) with no silica gel prep.

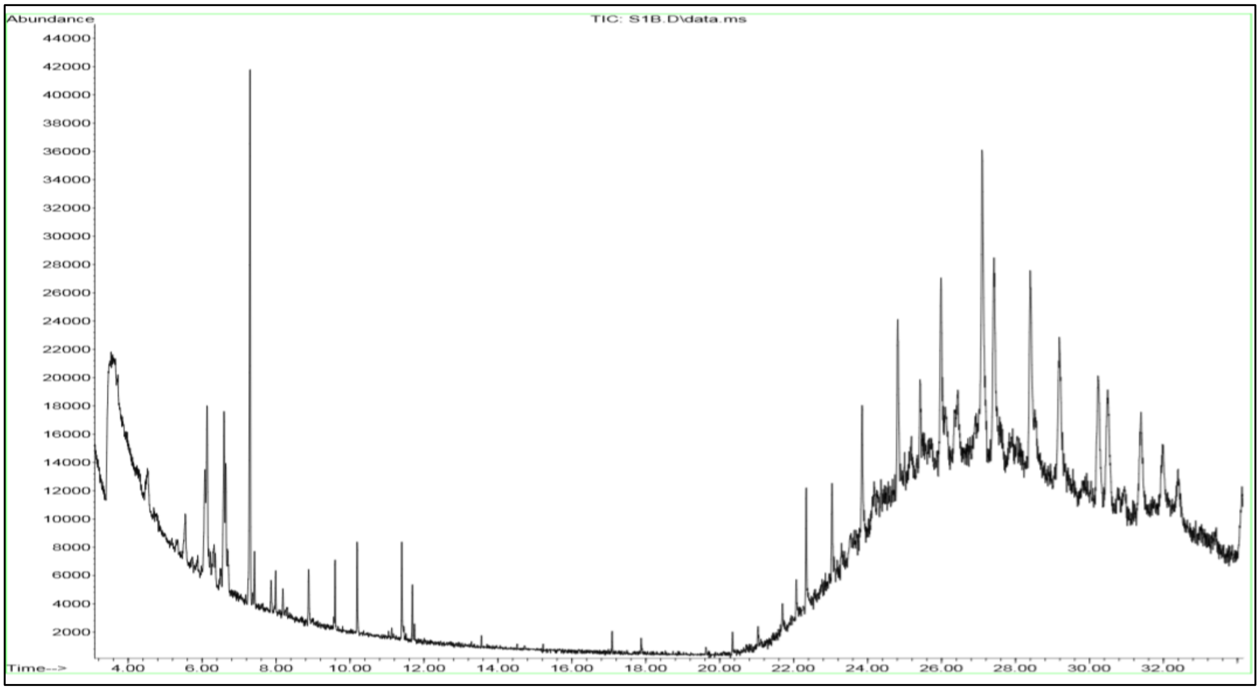


Figure B-1B: Sample 1 (5D-612) with silica gel prep.

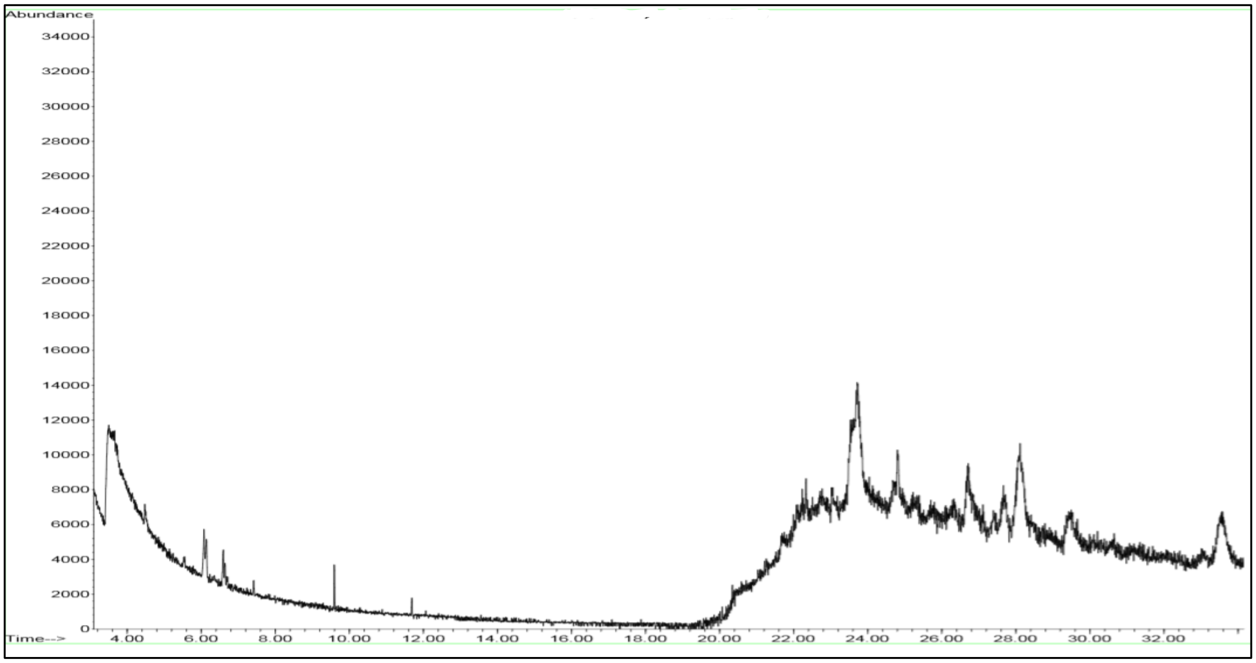


Figure B-2A: Sample 2 (5D-885) without silica gel prep.

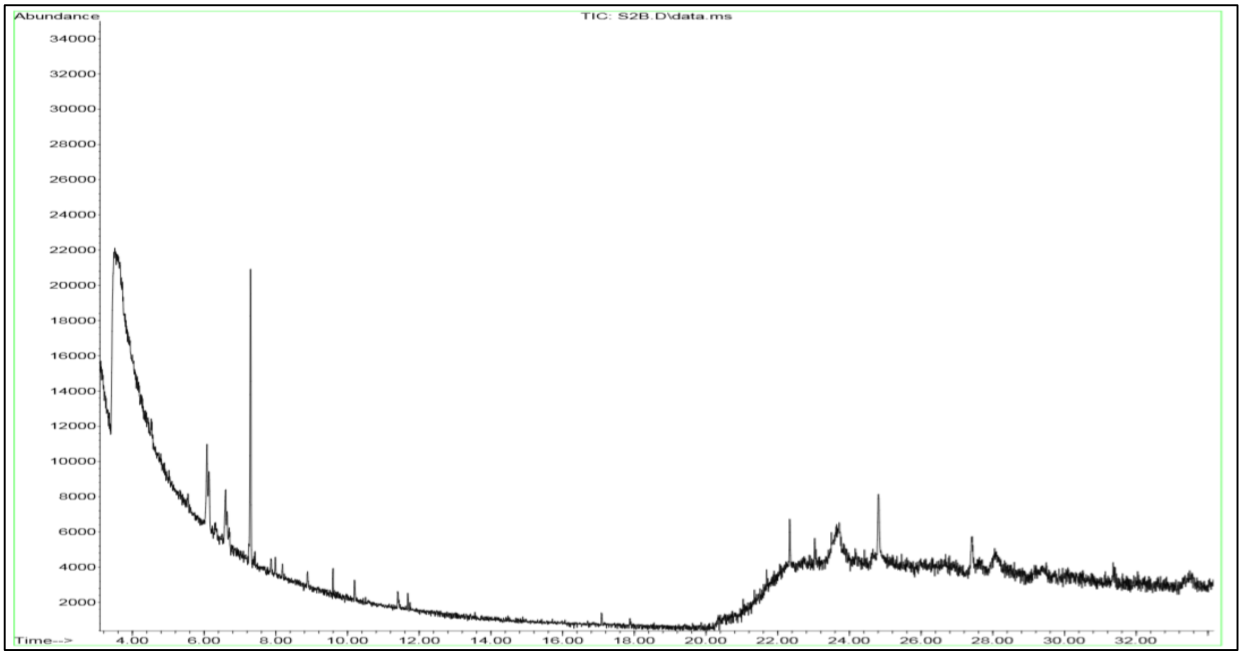


Figure B-2B: Sample 2 (5D-885) with silica gel prep.

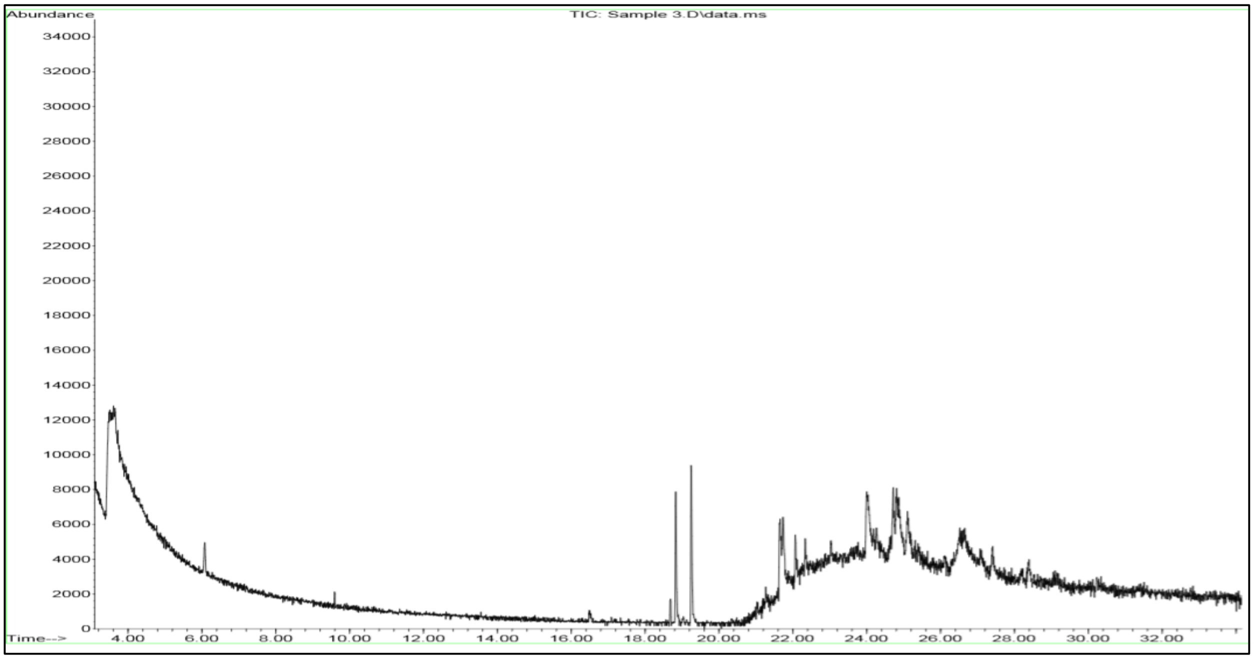


Figure B-3A: Sample 3 (01-BE-B) without silica gel prep.

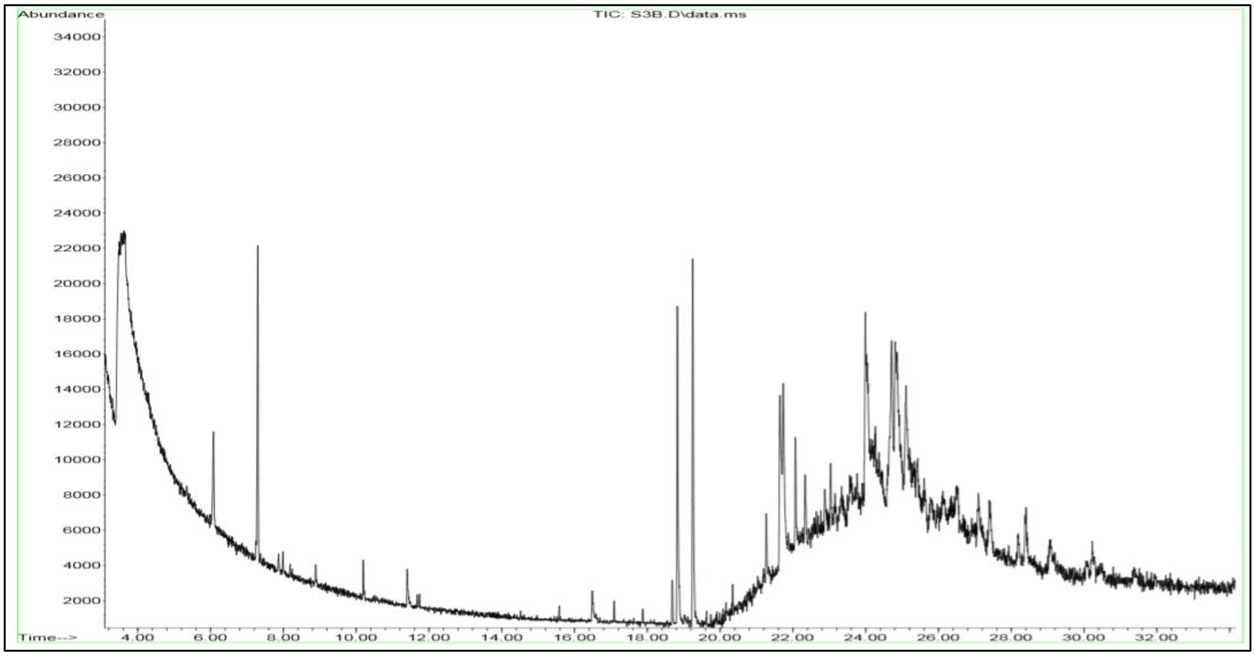


Figure B-3B: Sample 3 (01-BE-B) with silica gel prep.

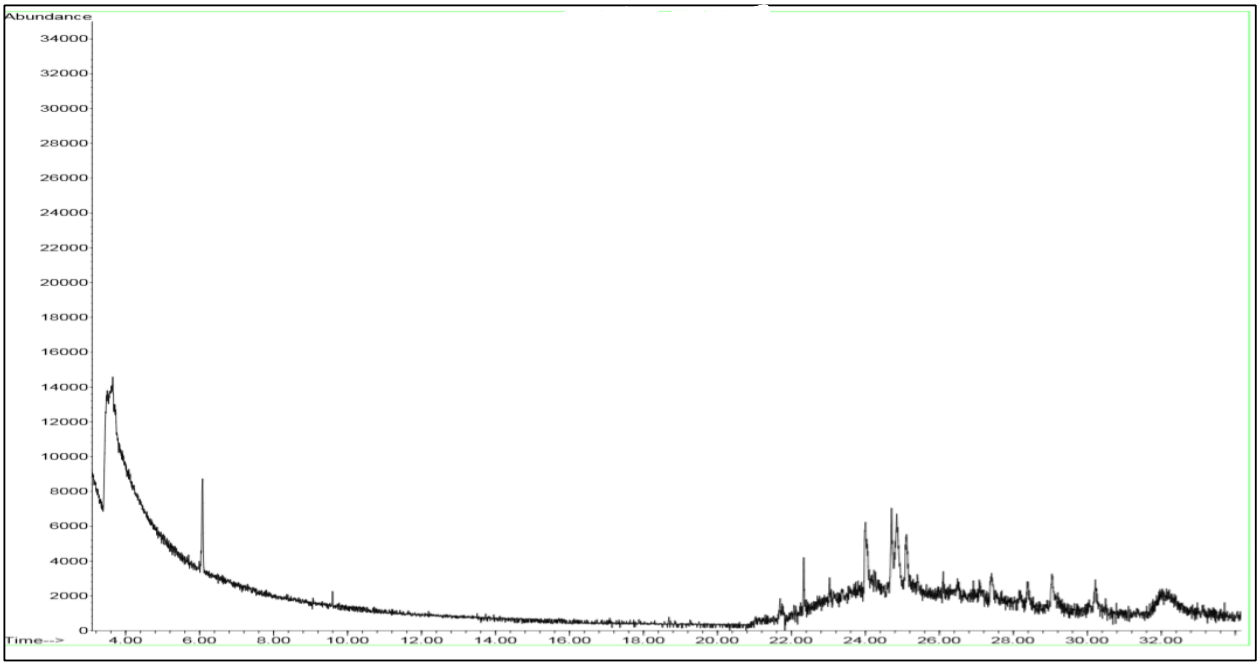


Figure B-4A: Sample 4 (02-LS-C) without silica gel prep.

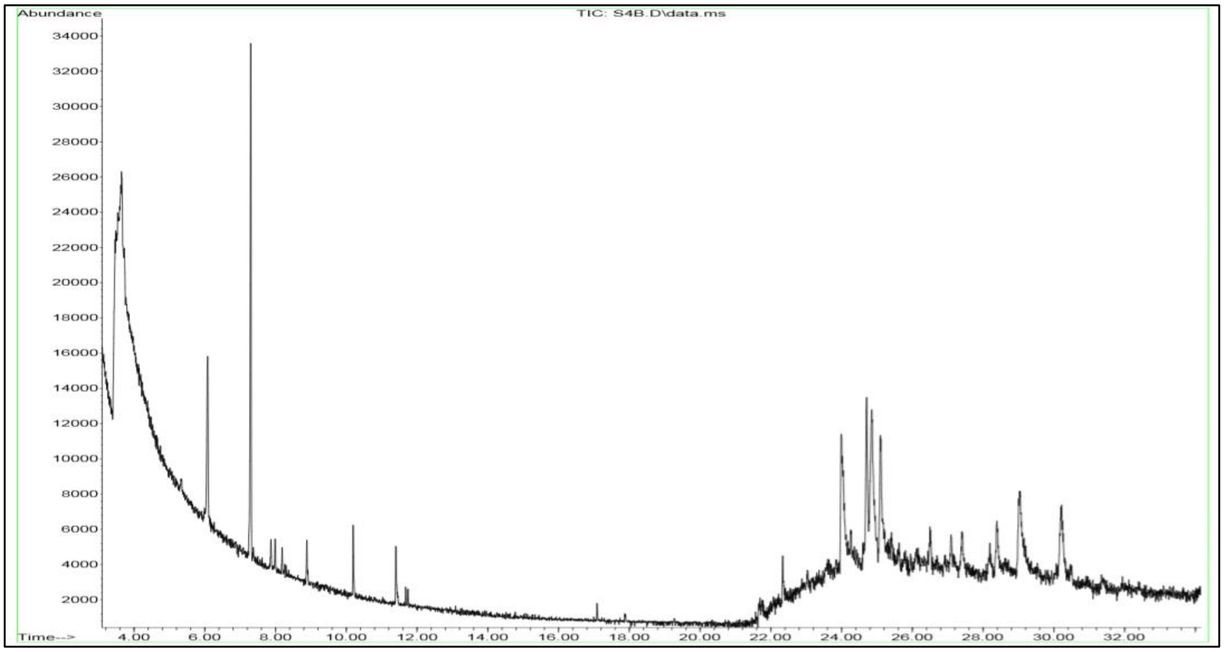


Figure B-4B: Sample 4 (02-LS-C) with silica gel prep.

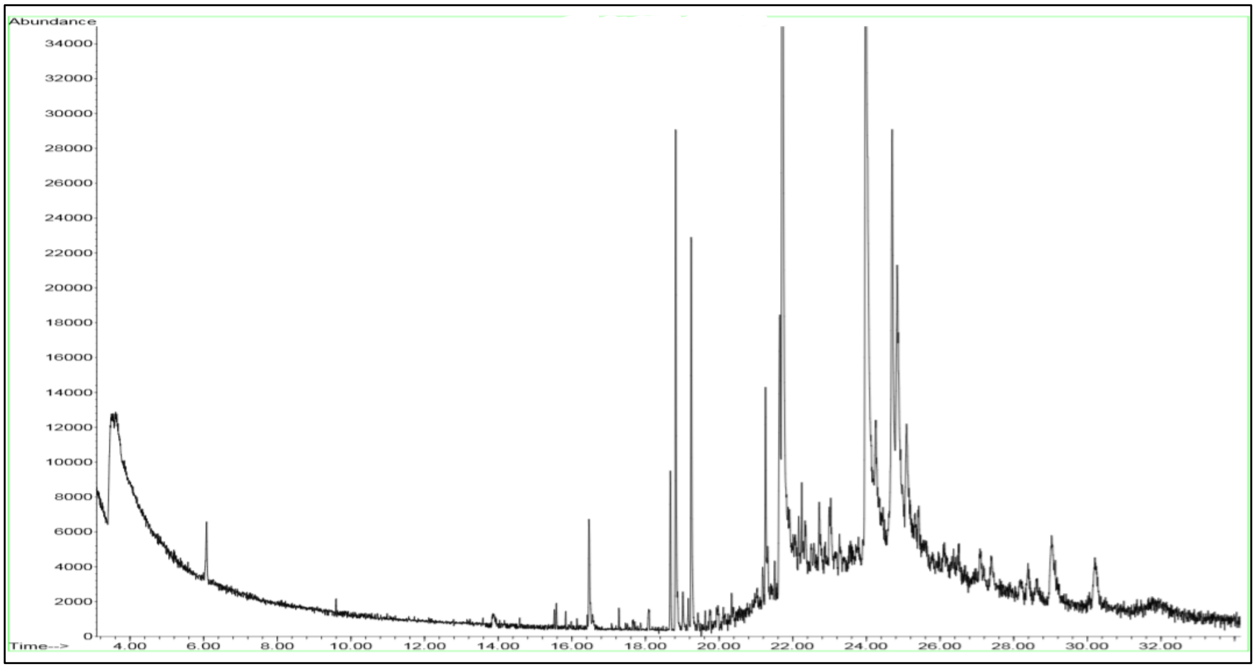


Figure B-5A: Sample 5 (18-B-A) without silica gel prep.

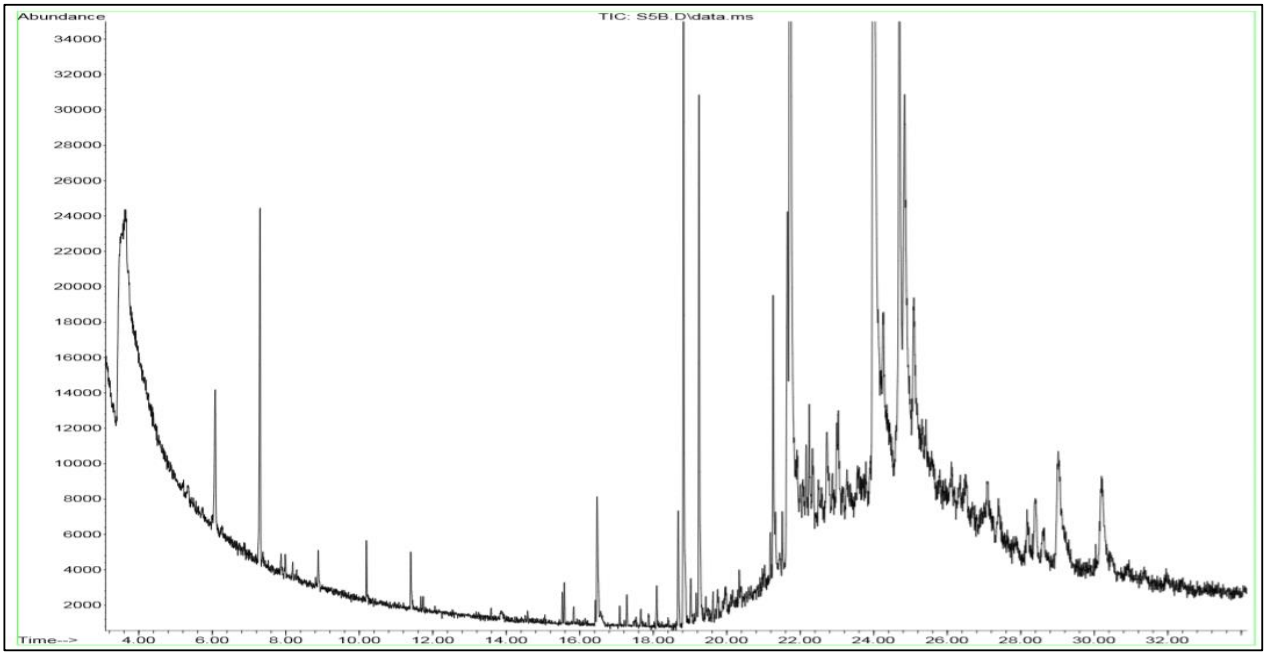
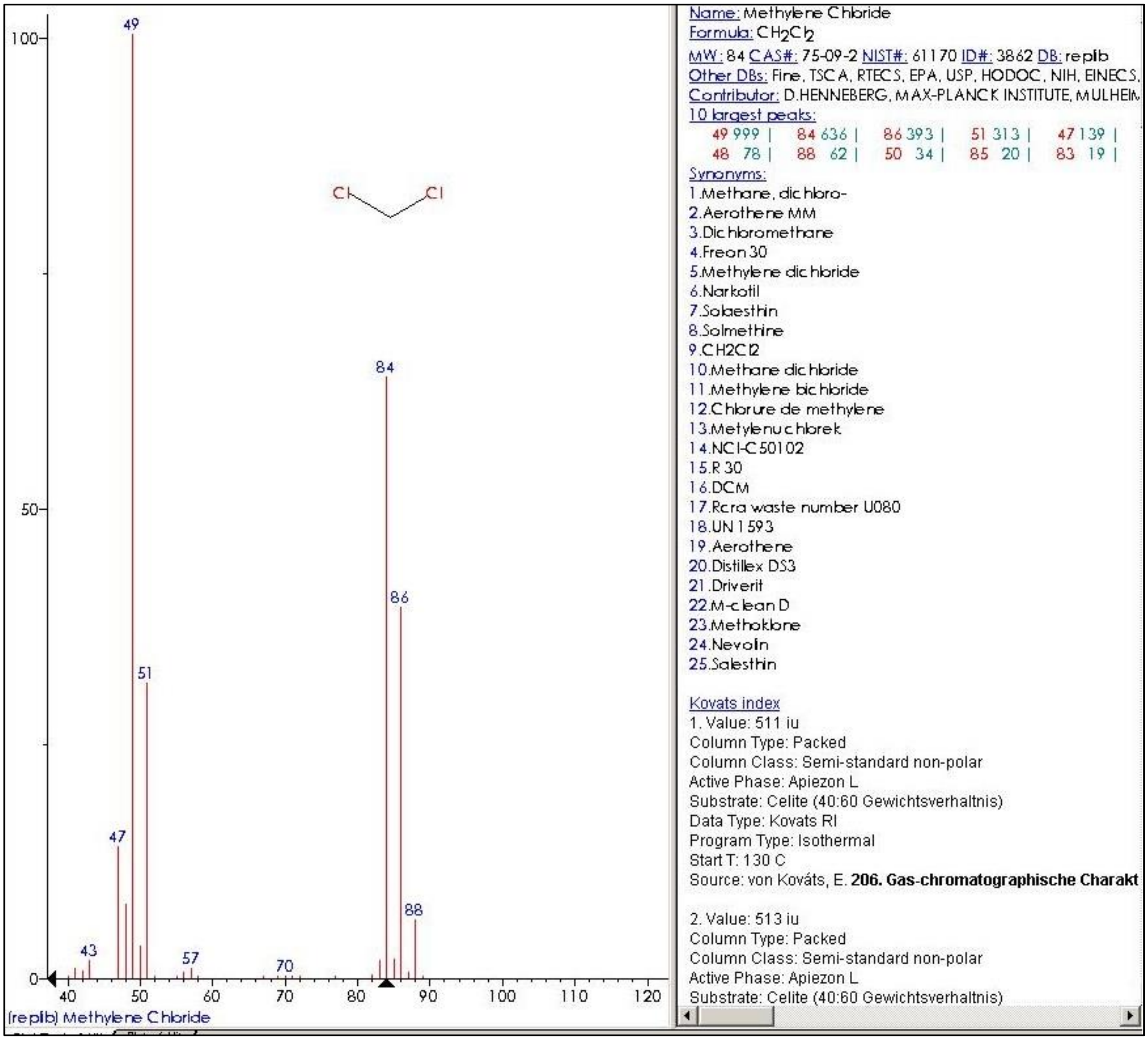


Figure B-5B: Sample 5 (18-B-A) with silica gel prep.

Appendix C. Mass spectral identification of specific compounds in the soil samples.

Appendix: Compound Figures



Name: Methylene Chloride
Formula: CH_2Cl_2
MW: 84 CAS#: 75-09-2 NIST#: 61170 ID#: 3862 DB: replib
Other DBs: Fine, TSCA, RTECS, EPA, USP, HODOC, NIH, EINECS,
Contributor: D.HENNEBERG, MAX-PLANCK INSTITUTE, MULHEI
10 largest peaks:

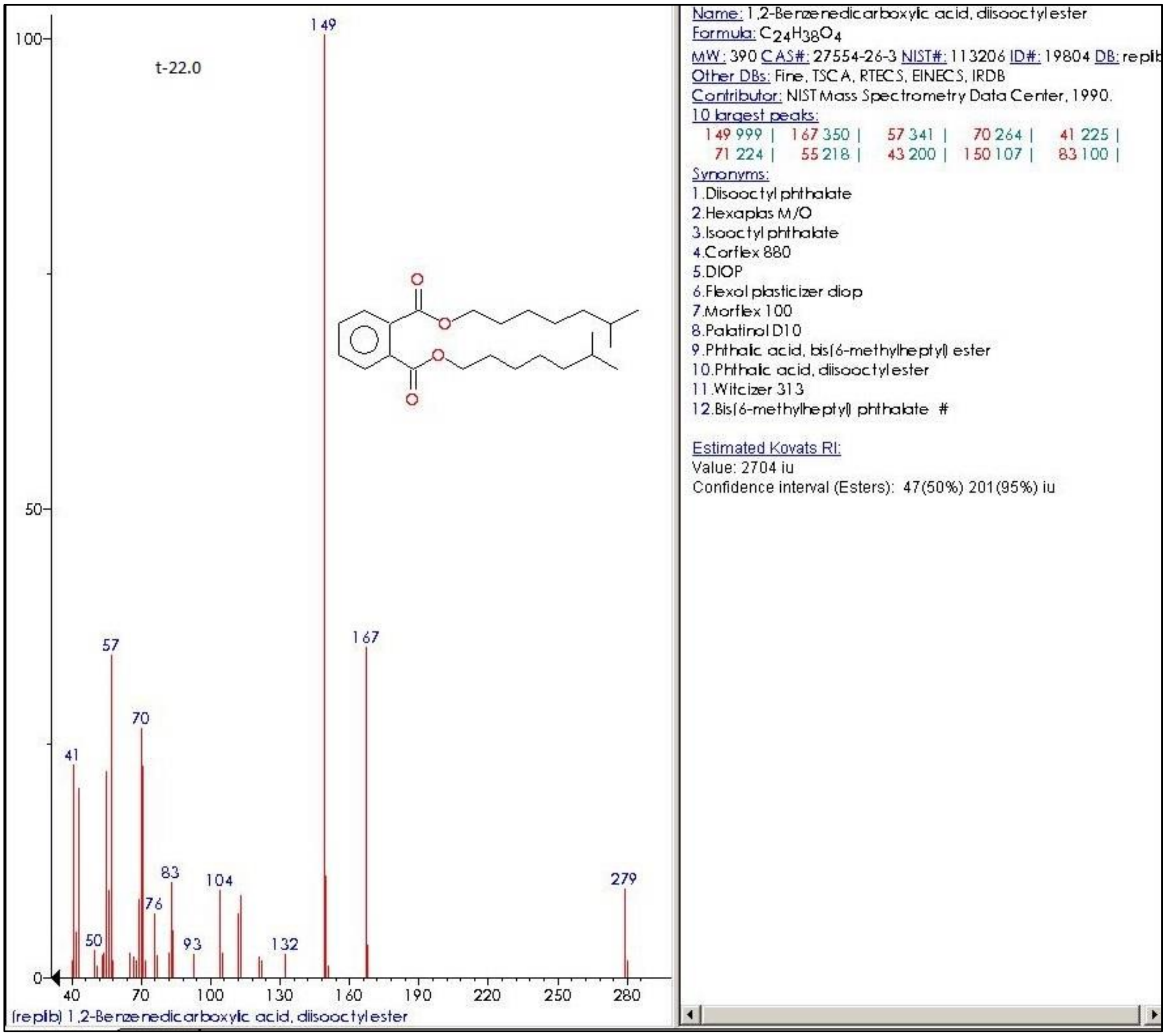
```
49 999 |   84 636 |   86 393 |   51 313 |   47 139 |
48  78 |   88  62 |   50  34 |   85  20 |   83  19 |
```

Synonyms:
1.Methane, dichloro-
2.Aerothene MM
3.Dichloromethane
4.Freon 30
5.Methylene dichloride
6.Narkotil
7.Solaesthin
8.Solmethine
9.CH2Cl2
10.Methane dichloride
11.Methylene bichloride
12.Chlorure de methylene
13.Metylenu chlorek
14.NCI-C50102
15.R 30
16.DCM
17.Rcra waste number U080
18.UN 1593
19.Aerothene
20.Distillex DS3
21.Driverit
22.M-clean D
23.Methoklone
24.Nevolin
25.Salesthin

Kovats index
1. Value: 511 iu
Column Type: Packed
Column Class: Semi-standard non-polar
Active Phase: Apiezon L
Substrate: Celite (40:60 Gewichtsverhaltnis)
Data Type: Kovats RI
Program Type: Isothermal
Start T: 130 C
Source: von Kováts, E. **206. Gas-chromatographische Charakt**

2. Value: 513 iu
Column Type: Packed
Column Class: Semi-standard non-polar
Active Phase: Apiezon L
Substrate: Celite (40:60 Gewichtsverhaltnis)

Figure C-1

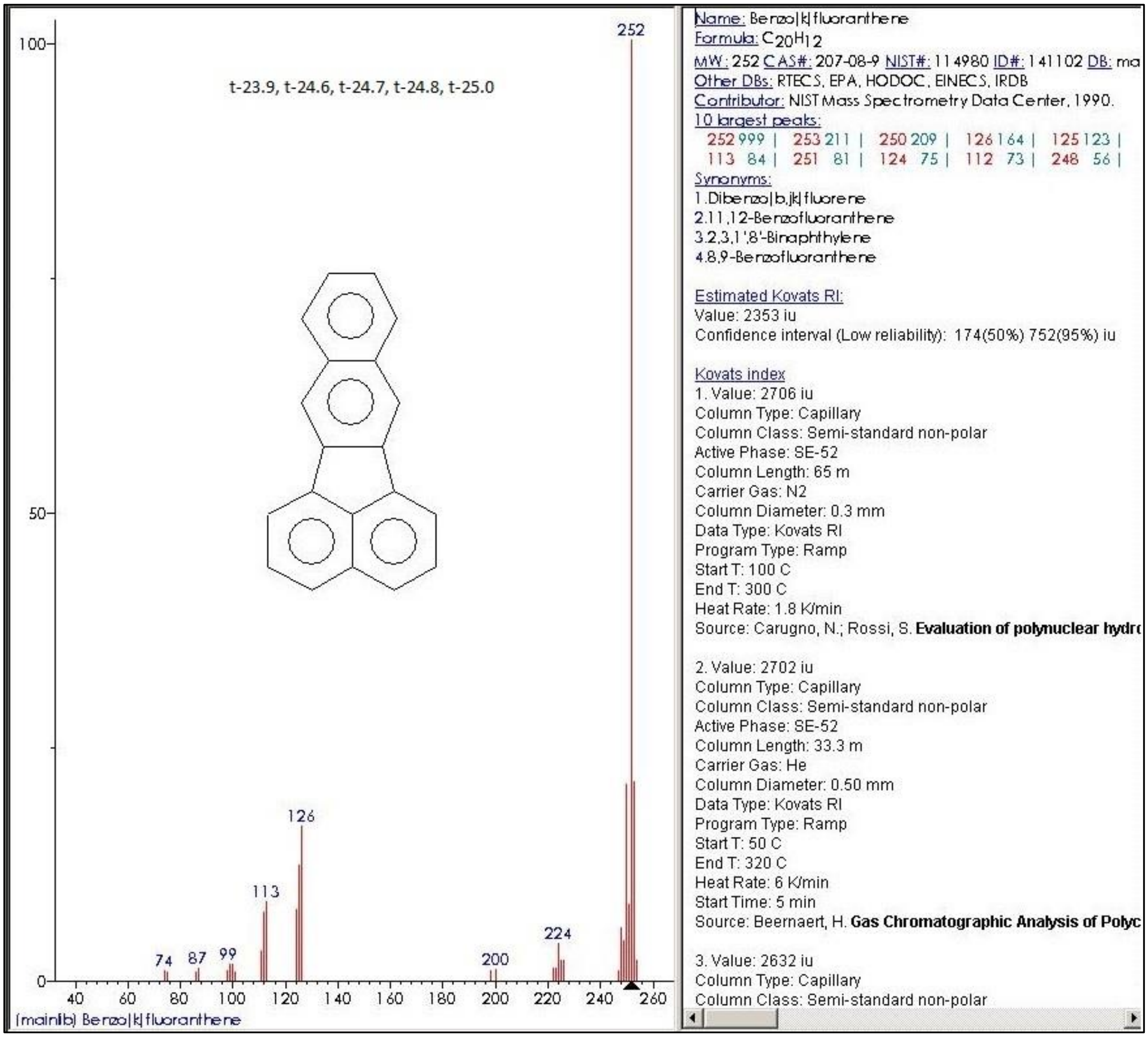


Name: 1,2-Benzenedicarboxylic acid, diisooctyl ester
Formula: $C_{24}H_{38}O_4$
MW: 390 CAS#: 27554-26-3 NIST#: 113206 ID#: 19804 DB: replib
Other DBs: Fine, TSCA, RTECS, EINECS, IRDB
Contributor: NIST Mass Spectrometry Data Center, 1990.
10 largest peaks:
149 999 | 167 350 | 57 341 | 70 264 | 41 225 |
71 224 | 55 218 | 43 200 | 150 107 | 83 100 |
Synonyms:
1.Diisooctyl phthalate
2.Hexaplas M/O
3.Isooctyl phthalate
4.Corflex 880
5.DIOP
6.Flexol plasticizer diop
7.Morflex 100
8.Palatinol D10
9.Phthalic acid, bis(6-methylheptyl) ester
10.Phthalic acid, diisooctyl ester
11.Witcizer 313
12.Bis(6-methylheptyl) phthalate #

Estimated Kovats RI:
Value: 2704 iu
Confidence interval (Esters): 47(50%) 201(95%) iu

(replib) 1,2-Benzenedicarboxylic acid, diisooctyl ester

Figure C-2



Name: Benzo[k]fluoranthene
Formula: $C_{20}H_{12}$
MW: 252 CAS#: 207-08-9 NIST#: 114980 ID#: 141102 DB: ma
Other DBs: RTECS, EPA, HODOC, EINECS, IRDB
Contributor: NIST Mass Spectrometry Data Center, 1990.
10 largest peaks:
252 999 | 253 211 | 250 209 | 126 164 | 125 123 |
113 84 | 251 81 | 124 75 | 112 73 | 248 56 |
Synonyms:
1.Dibenzo[b,jk]fluorene
2.11,12-Benzofluoranthene
3.2,3,1',8'-Binaphthylene
4.8,9-Benzofluoranthene

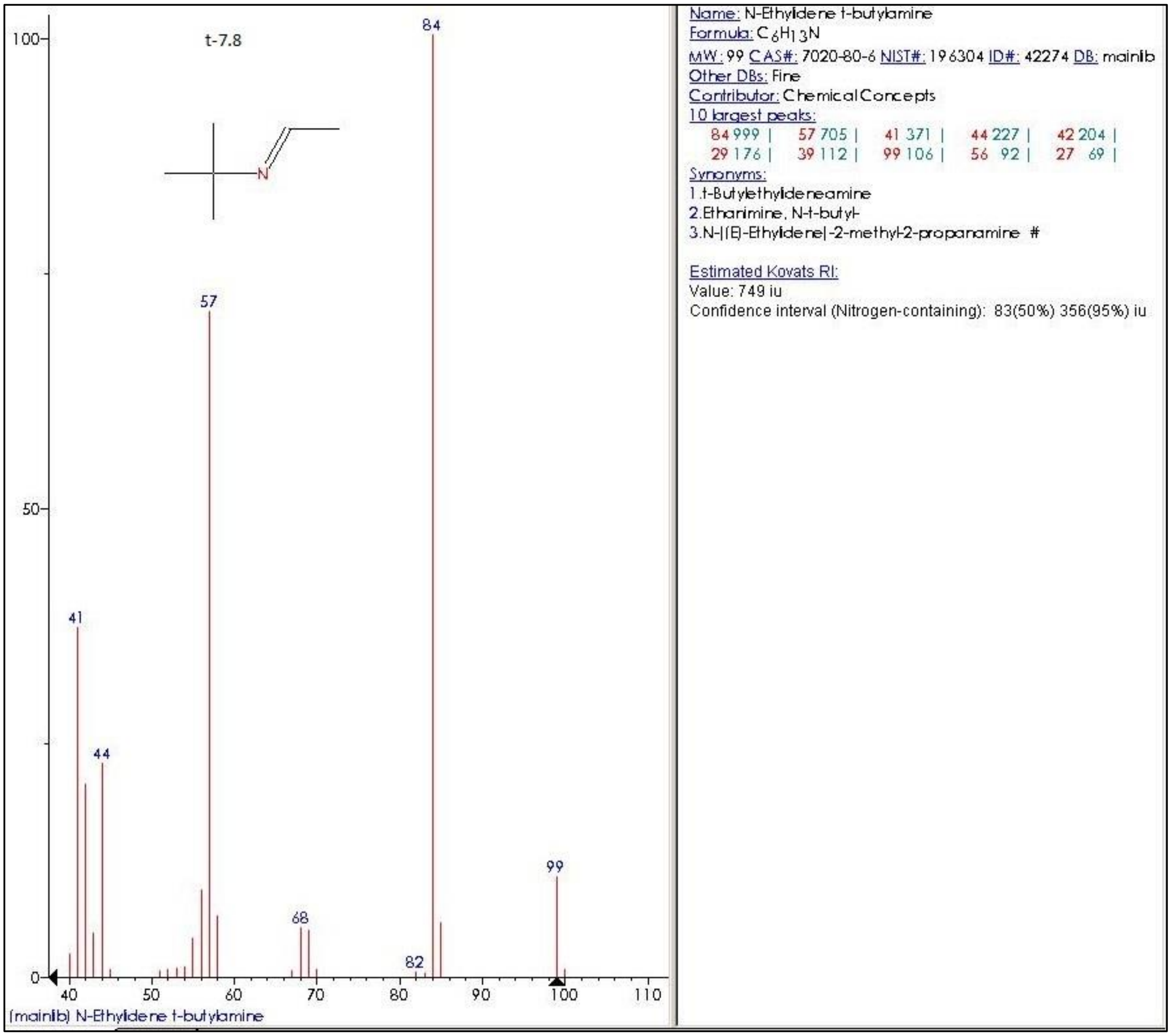
Estimated Kovats RI:
Value: 2353 iu
Confidence interval (Low reliability): 174(50%) 752(95%) iu

Kovats index
1. Value: 2706 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Column Length: 65 m
Carrier Gas: N2
Column Diameter: 0.3 mm
Data Type: Kovats RI
Program Type: Ramp
Start T: 100 C
End T: 300 C
Heat Rate: 1.8 K/min
Source: Carugno, N.; Rossi, S. **Evaluation of polynuclear hydro**

2. Value: 2702 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Column Length: 33.3 m
Carrier Gas: He
Column Diameter: 0.50 mm
Data Type: Kovats RI
Program Type: Ramp
Start T: 50 C
End T: 320 C
Heat Rate: 6 K/min
Start Time: 5 min
Source: Beernaert, H. **Gas Chromatographic Analysis of Polyc**

3. Value: 2632 iu
Column Type: Capillary
Column Class: Semi-standard non-polar

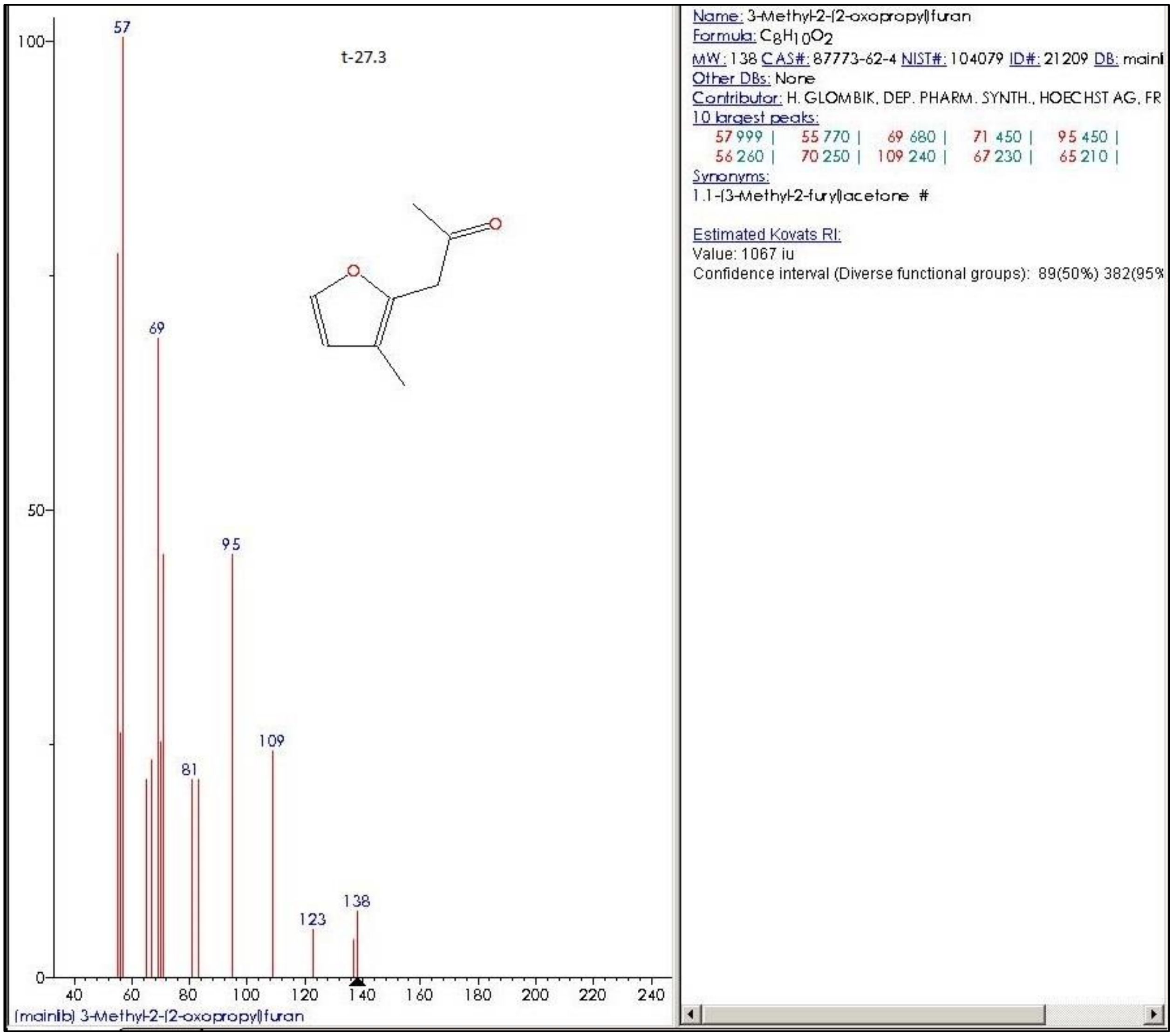
Figure C-3



Name: N-Ethylidene t-butylamine
Formula: $C_6H_{13}N$
MW: 99 CAS#: 7020-80-6 NIST#: 196304 ID#: 42274 DB: mainlib
Other DBs: Fine
Contributor: Chemical Concepts
10 largest peaks:
84 999 | 57 705 | 41 371 | 44 227 | 42 204 |
29 176 | 39 112 | 99 106 | 56 92 | 27 69 |
Synonyms:
1.t-Butylethylideneamine
2.Ethanimine, N-t-butyl-
3.N-[(E)-Ethylidene]-2-methyl-2-propanamine #

Estimated Kovats RI:
Value: 749 iu
Confidence interval (Nitrogen-containing): 83(50%) 356(95%) iu

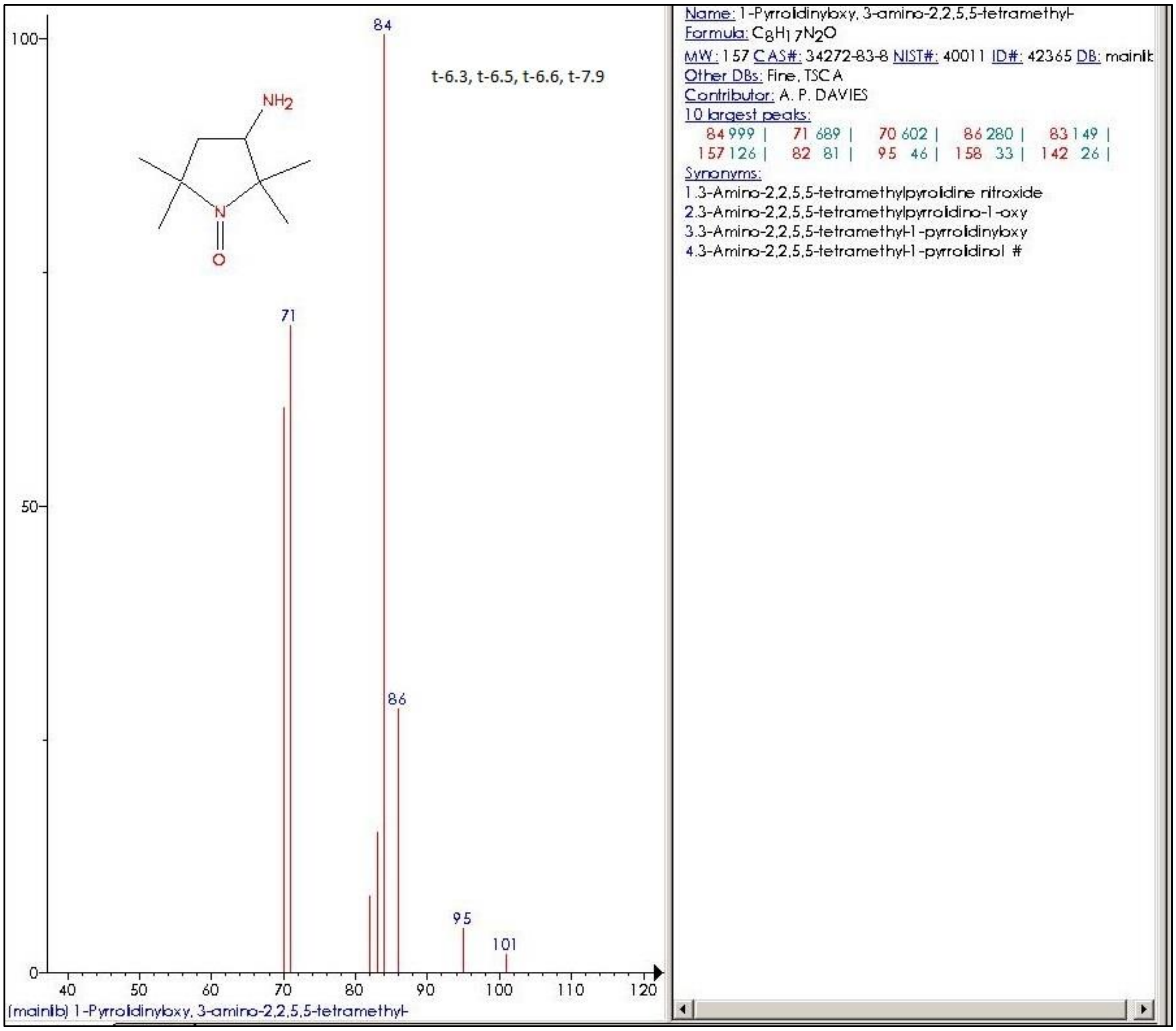
Figure C-4



Name: 3-Methyl-2-(2-oxopropyl)furan
Formula: $C_8H_{10}O_2$
MW: 138 CAS#: 87773-62-4 NIST#: 104079 ID#: 21209 DB: mainl
Other DBs: None
Contributor: H. GLOMBIK, DEP. PHARM. SYNTH., HOECHST AG, FR
10 largest peaks:
57 999 | 55 770 | 69 680 | 71 450 | 95 450 |
56 260 | 70 250 | 109 240 | 67 230 | 65 210 |
Synonyms:
1.1-(3-Methyl-2-furyl)acetone #

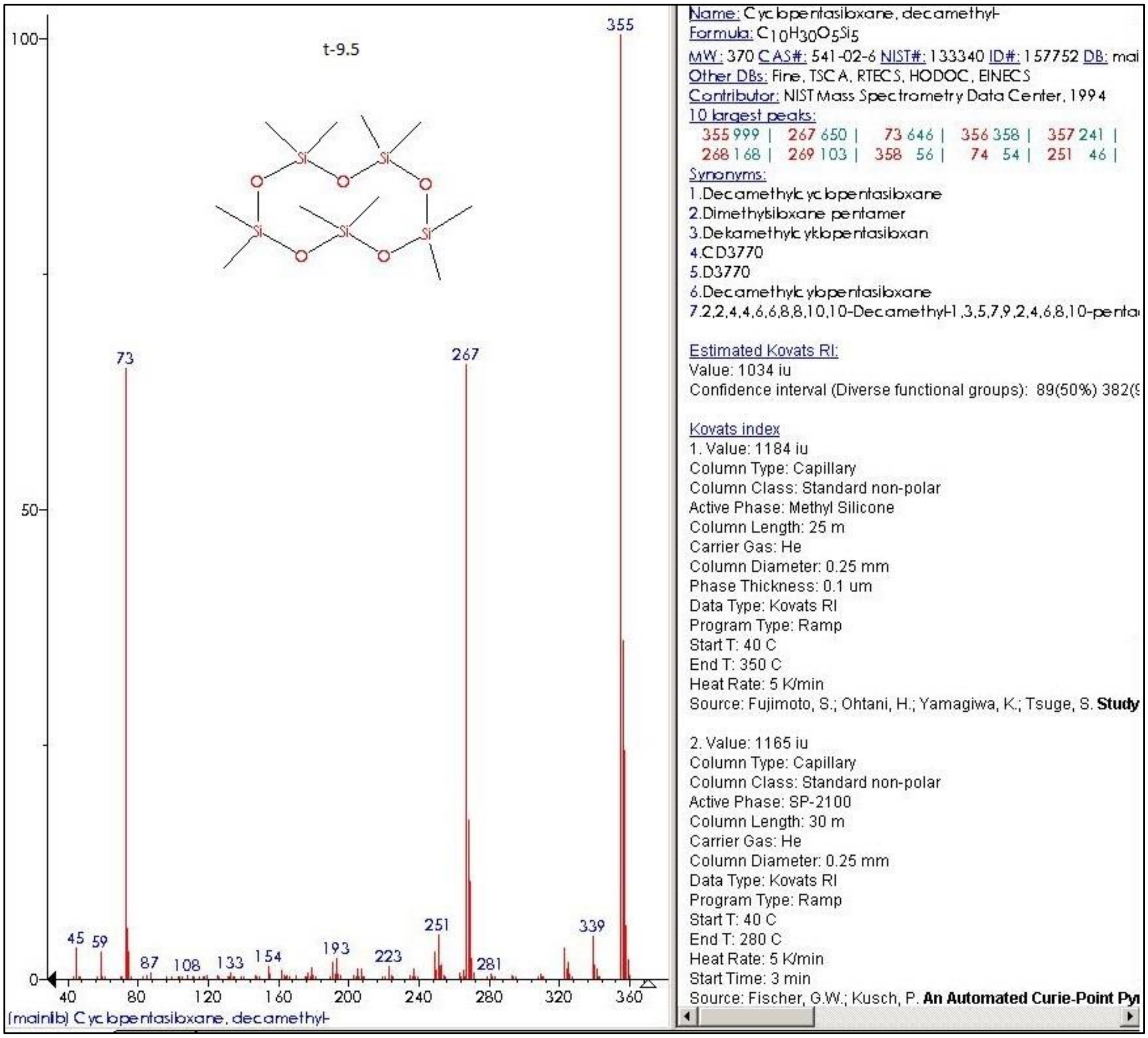
Estimated Kovats RI:
Value: 1067 iu
Confidence interval (Diverse functional groups): 89(50%) 382(95%

Figure C-5



Name: 1-Pyrrolidinyloxy, 3-amino-2,2,5,5-tetramethyl-
Formula: $C_8H_{17}N_2O$
MW: 157 CAS#: 34272-83-8 NIST#: 40011 ID#: 42365 DB: mainlib
Other DBs: Fine, TSCA
Contributor: A. P. DAVIES
10 largest peaks:
84 999 | 71 689 | 70 602 | 86 280 | 83 149 |
157 126 | 82 81 | 95 46 | 158 33 | 142 26 |
Synonyms:
1.3-Amino-2,2,5,5-tetramethylpyrolidine nitroxide
2.3-Amino-2,2,5,5-tetramethylpyrrolidino-1-oxy
3.3-Amino-2,2,5,5-tetramethyl-1-pyrrolidinyloxy
4.3-Amino-2,2,5,5-tetramethyl-1-pyrrolidinol #

Figure C-6



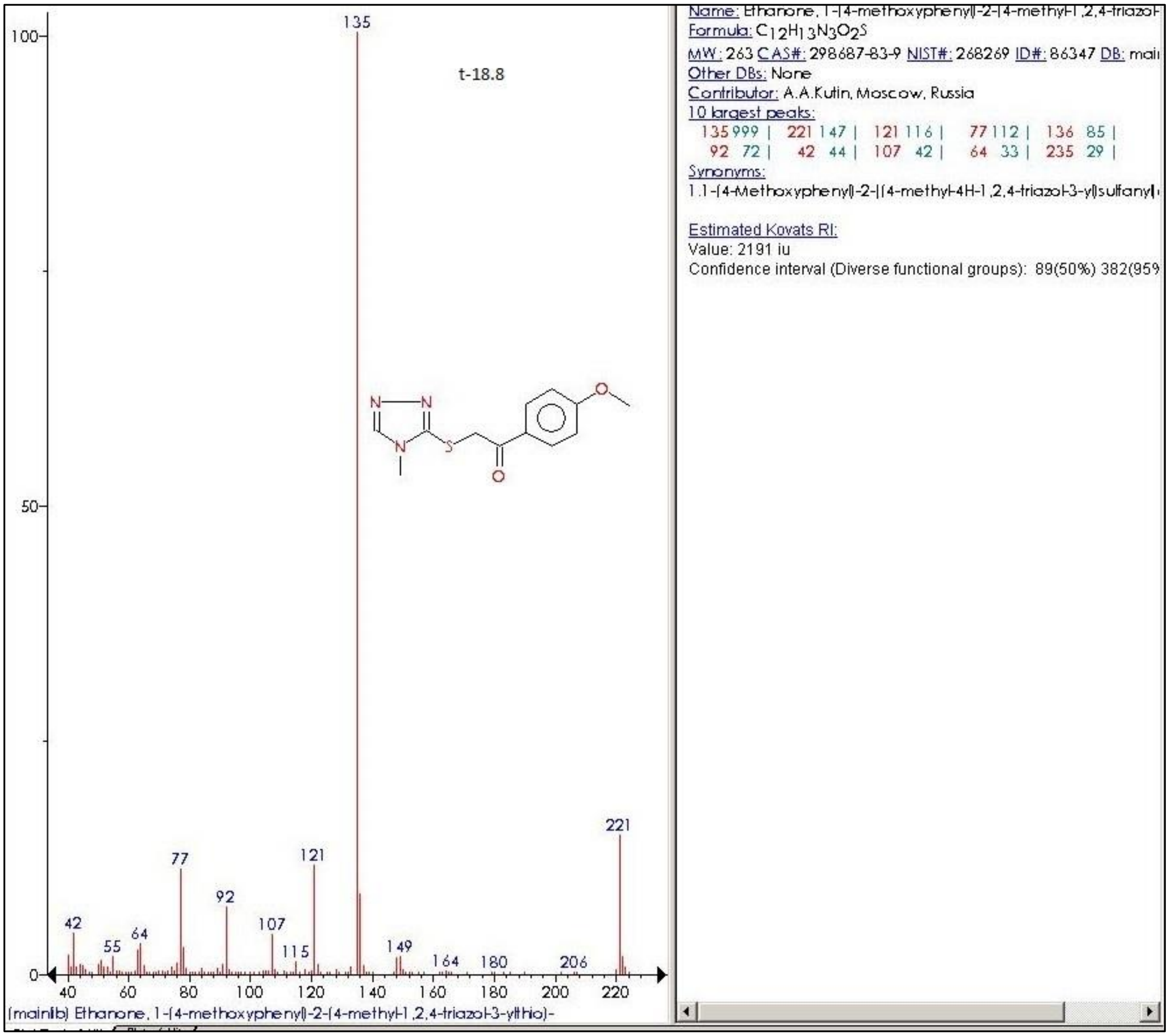
Name: Cyclopentasiloxane, decamethyl-
Formula: $C_{10}H_{30}O_5Si_5$
MW: 370 CAS#: 541-02-6 NIST#: 133340 ID#: 157752 DB: mai
Other DBs: Fine, TSCA, RTECS, HODOC, EINECS
Contributor: NIST Mass Spectrometry Data Center, 1994
10 largest peaks:
355 999 | 267 650 | 73 646 | 356 358 | 357 241 |
268 168 | 269 103 | 358 56 | 74 54 | 251 46 |
Synonyms:
1.Decamethylcyclopentasiloxane
2.Dimethylsiloxane pentamer
3.Dekamethylcyklopentasiloxan
4.CD3770
5.D3770
6.Decamethylcylopentasiloxane
7.2,2,4,4,6,6,8,8,10,10-Decamethyl-1,3,5,7,9,2,4,6,8,10-penta

Estimated Kovats RI:
Value: 1034 iu
Confidence interval (Diverse functional groups): 89(50%) 382(

Kovats index
1. Value: 1184 iu
Column Type: Capillary
Column Class: Standard non-polar
Active Phase: Methyl Silicone
Column Length: 25 m
Carrier Gas: He
Column Diameter: 0.25 mm
Phase Thickness: 0.1 um
Data Type: Kovats RI
Program Type: Ramp
Start T: 40 C
End T: 350 C
Heat Rate: 5 K/min
Source: Fujimoto, S.; Ohtani, H.; Yamagiwa, K.; Tsuge, S. **Study**

2. Value: 1165 iu
Column Type: Capillary
Column Class: Standard non-polar
Active Phase: SP-2100
Column Length: 30 m
Carrier Gas: He
Column Diameter: 0.25 mm
Data Type: Kovats RI
Program Type: Ramp
Start T: 40 C
End T: 280 C
Heat Rate: 5 K/min
Start Time: 3 min
Source: Fischer, G.W.; Kusch, P. **An Automated Curie-Point Py**

Figure C-7

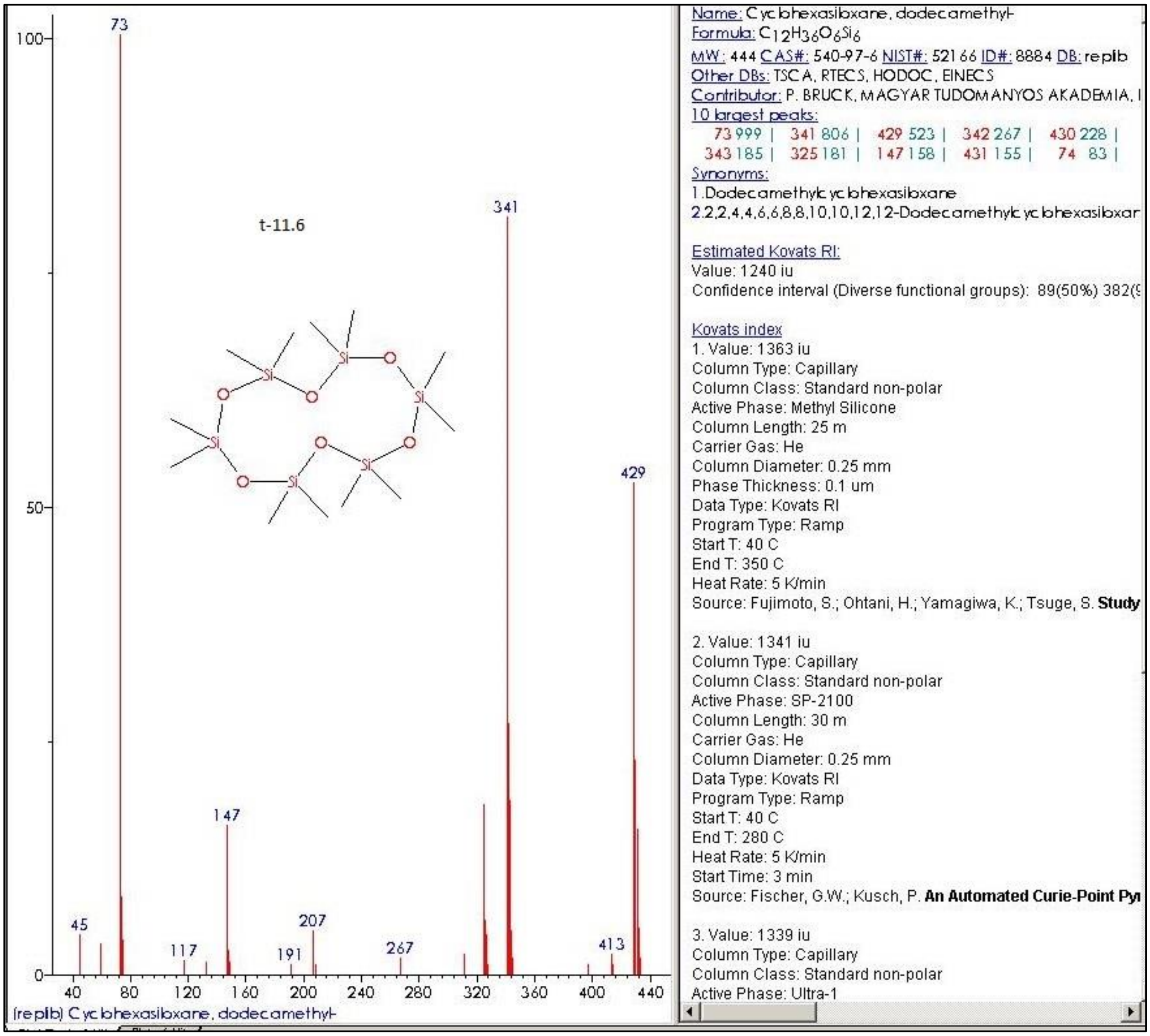


Name: Ethanone, 1-(4-methoxyphenyl)-2-(4-methyl-1,2,4-triazol-
Formula: $C_{12}H_{13}N_3O_2S$
MW: 263 CAS#: 298687-83-9 NIST#: 268269 ID#: 86347 DB: mai
Other DBs: None
Contributor: A.A.Kutin, Moscow, Russia
10 largest peaks:
135 999 | 221 147 | 121 116 | 77 112 | 136 85 |
92 72 | 42 44 | 107 42 | 64 33 | 235 29 |
Synonyms:
1.1-(4-Methoxyphenyl)-2-((4-methyl-4H-1,2,4-triazol-3-yl)sulfanyl

Estimated Kovats RI:
Value: 2191 iu
Confidence interval (Diverse functional groups): 89(50%) 382(95

(mainlib) Ethanone, 1-(4-methoxyphenyl)-2-(4-methyl-1,2,4-triazol-3-ylthio)-

Figure C-8



(replib) Cyclohexasiloxane, dodecamethyl-

Name: Cyclohexasiloxane, dodecamethyl-
Formula: $C_{12}H_{36}O_6Si_6$
MW: 444 CAS#: 540-97-6 NIST#: 52166 ID#: 8884 DB: replib
Other DBs: TSCA, RTECS, HODOC, EINECS
Contributor: P. BRUCK, MAGYAR TUDOMANYOS AKADEMIA, I
10 largest peaks:
73 999 | 341 806 | 429 523 | 342 267 | 430 228 |
343 185 | 325 181 | 147 158 | 431 155 | 74 83 |
Synonyms:
1.Dodecamethylcyclohexasiloxane
2.2,2,4,4,6,6,8,8,10,10,12,12-Dodecamethylcyclohexasiloxar

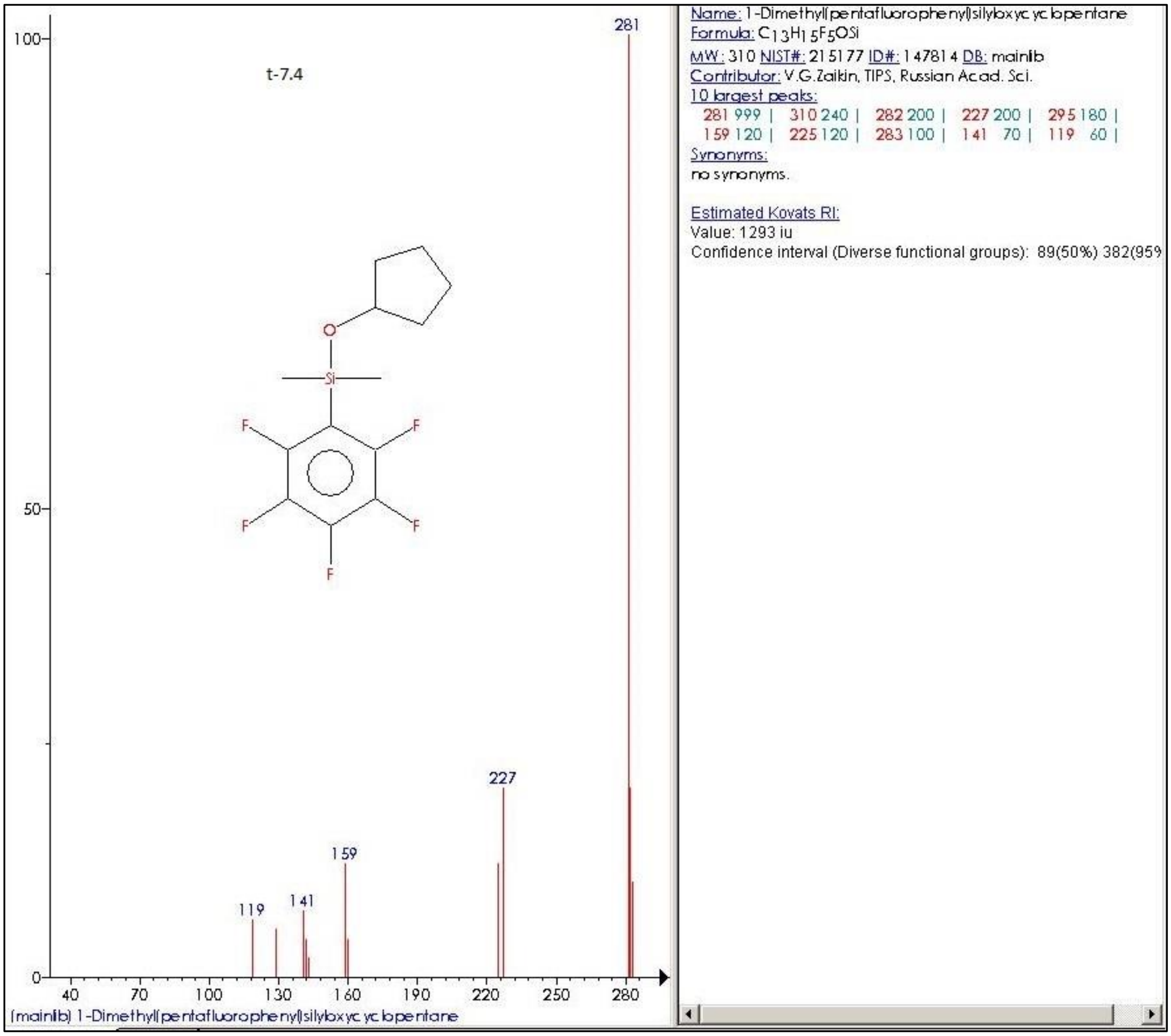
Estimated Kovats RI:
Value: 1240 iu
Confidence interval (Diverse functional groups): 89(50%) 382(

Kovats index
1. Value: 1363 iu
Column Type: Capillary
Column Class: Standard non-polar
Active Phase: Methyl Silicone
Column Length: 25 m
Carrier Gas: He
Column Diameter: 0.25 mm
Phase Thickness: 0.1 um
Data Type: Kovats RI
Program Type: Ramp
Start T: 40 C
End T: 350 C
Heat Rate: 5 K/min
Source: Fujimoto, S.; Ohtani, H.; Yamagiwa, K.; Tsuge, S. **Study**

2. Value: 1341 iu
Column Type: Capillary
Column Class: Standard non-polar
Active Phase: SP-2100
Column Length: 30 m
Carrier Gas: He
Column Diameter: 0.25 mm
Data Type: Kovats RI
Program Type: Ramp
Start T: 40 C
End T: 280 C
Heat Rate: 5 K/min
Start Time: 3 min
Source: Fischer, G.W.; Kusch, P. **An Automated Curie-Point Py**

3. Value: 1339 iu
Column Type: Capillary
Column Class: Standard non-polar
Active Phase: Ultra-1

Figure C-9



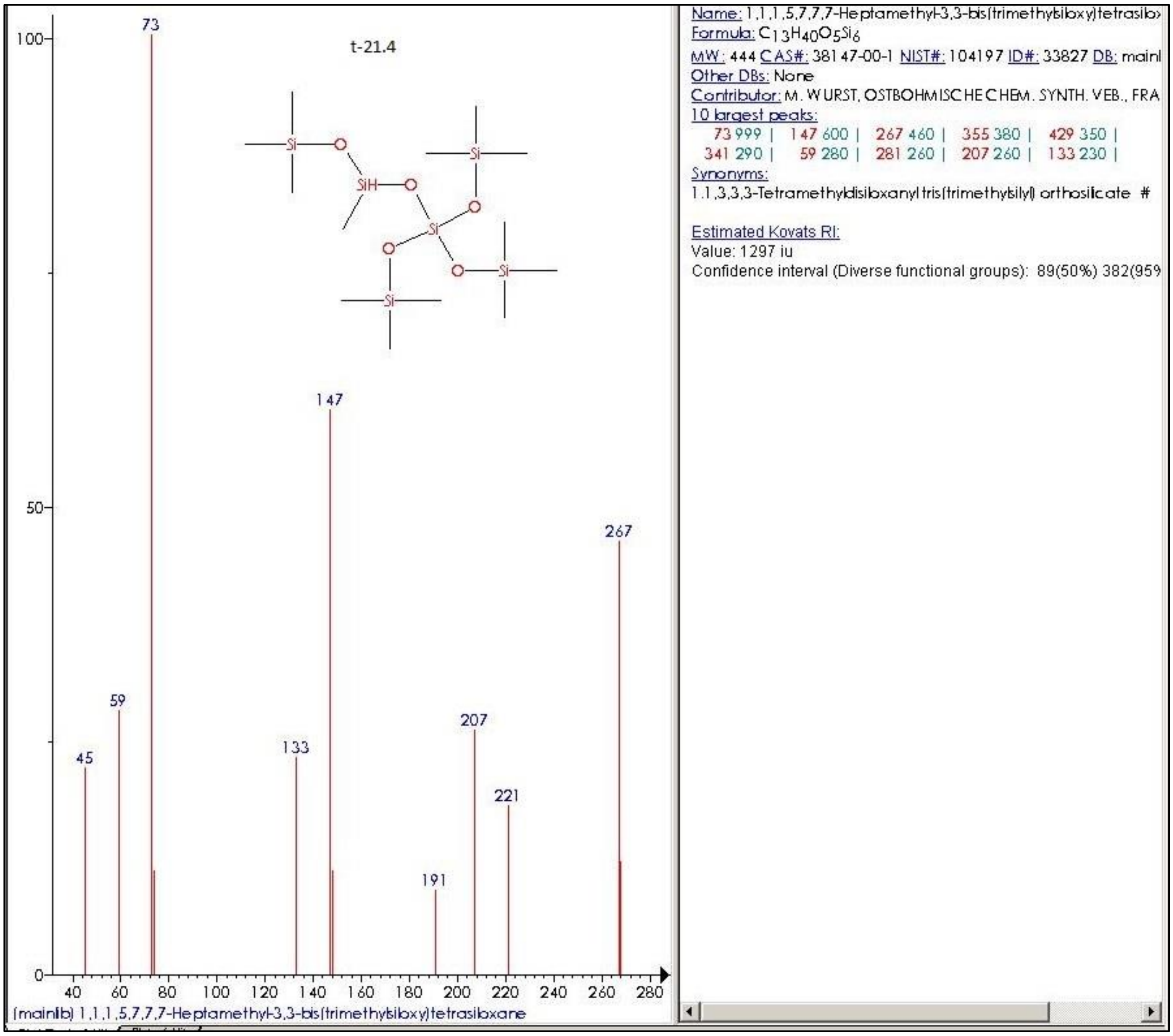
Name: 1-Dimethyl(pentafluorophenyl)silyloxycyclopentane
Formula: $C_{13}H_{15}F_5OSi$
MW: 310 NIST#: 215177 ID#: 147814 DB: mainlib
Contributor: V.G.Zaikin, TIPS, Russian Acad. Sci.
10 largest peaks:

```
  281 999 |   310 240 |   282 200 |   227 200 |   295 180 |
  159 120 |   225 120 |   283 100 |   141  70 |   119  60 |
```

Synonyms:
no synonyms.

Estimated Kovats RI:
Value: 1293 iu
Confidence interval (Diverse functional groups): 89(50%) 382(959

Figure C-10

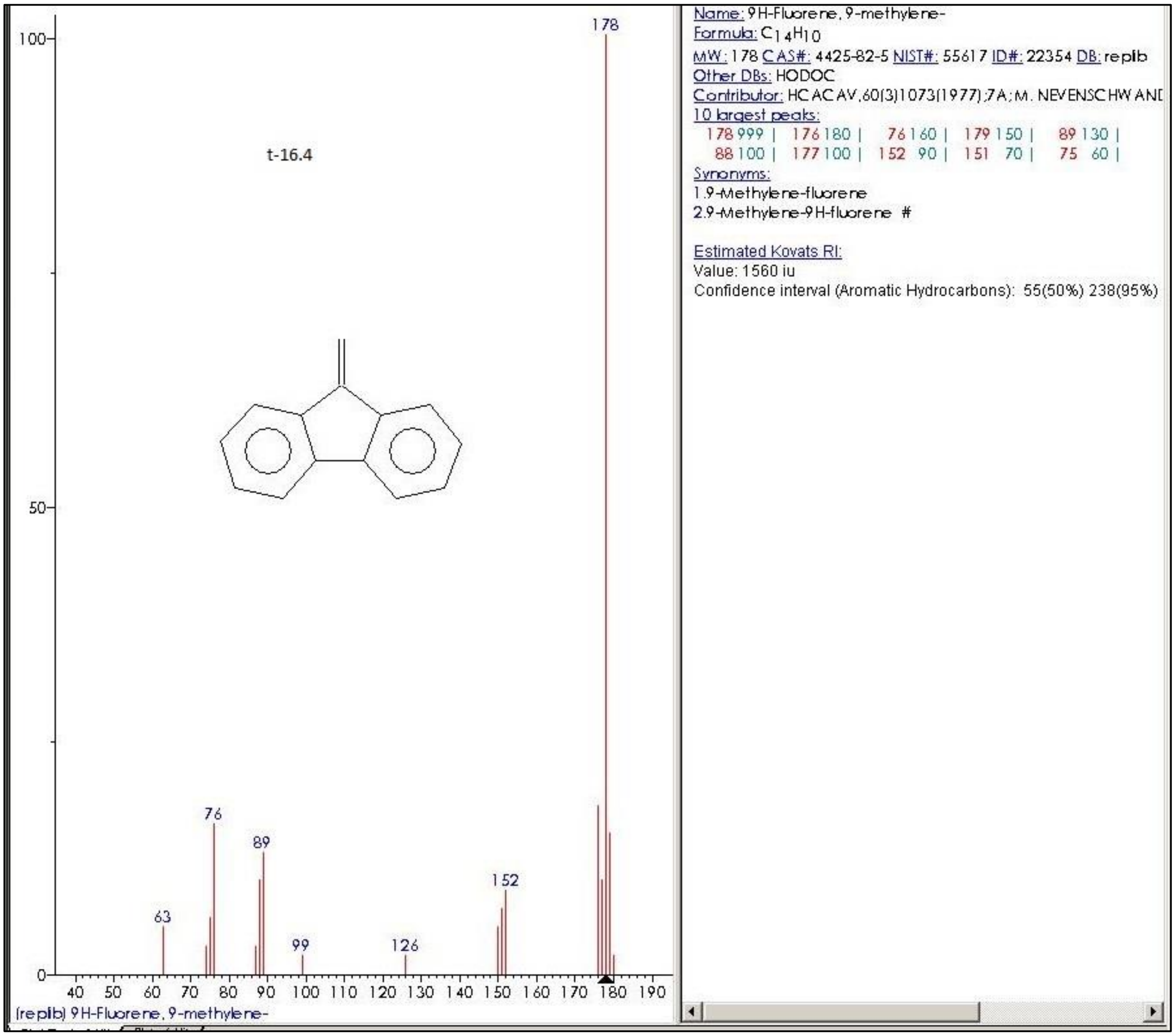


(mainlib) 1,1,1,5,7,7,7-Heptamethyl-3,3-bis(trimethylsiloxy)tetrasiloxane

Name: 1,1,1,5,7,7,7-Heptamethyl-3,3-bis(trimethylsiloxy)tetrasilo
Formula: $C_{13}H_{40}O_5Si_6$
MW: 444 CAS#: 38147-00-1 NIST#: 104197 ID#: 33827 DB: mainl
Other DBs: None
Contributor: M. WURST, OSTBOHMISCHE CHEM. SYNTH. VEB., FRA
10 largest peaks:
73 999 | 147 600 | 267 460 | 355 380 | 429 350 |
341 290 | 59 280 | 281 260 | 207 260 | 133 230 |
Synonyms:
1,1,3,3,3-Tetramethyldisiloxanyl tris(trimethylsilyl) orthosilicate #

Estimated Kovats RI:
Value: 1297 iu
Confidence interval (Diverse functional groups): 89(50%) 382(95

Figure C-11



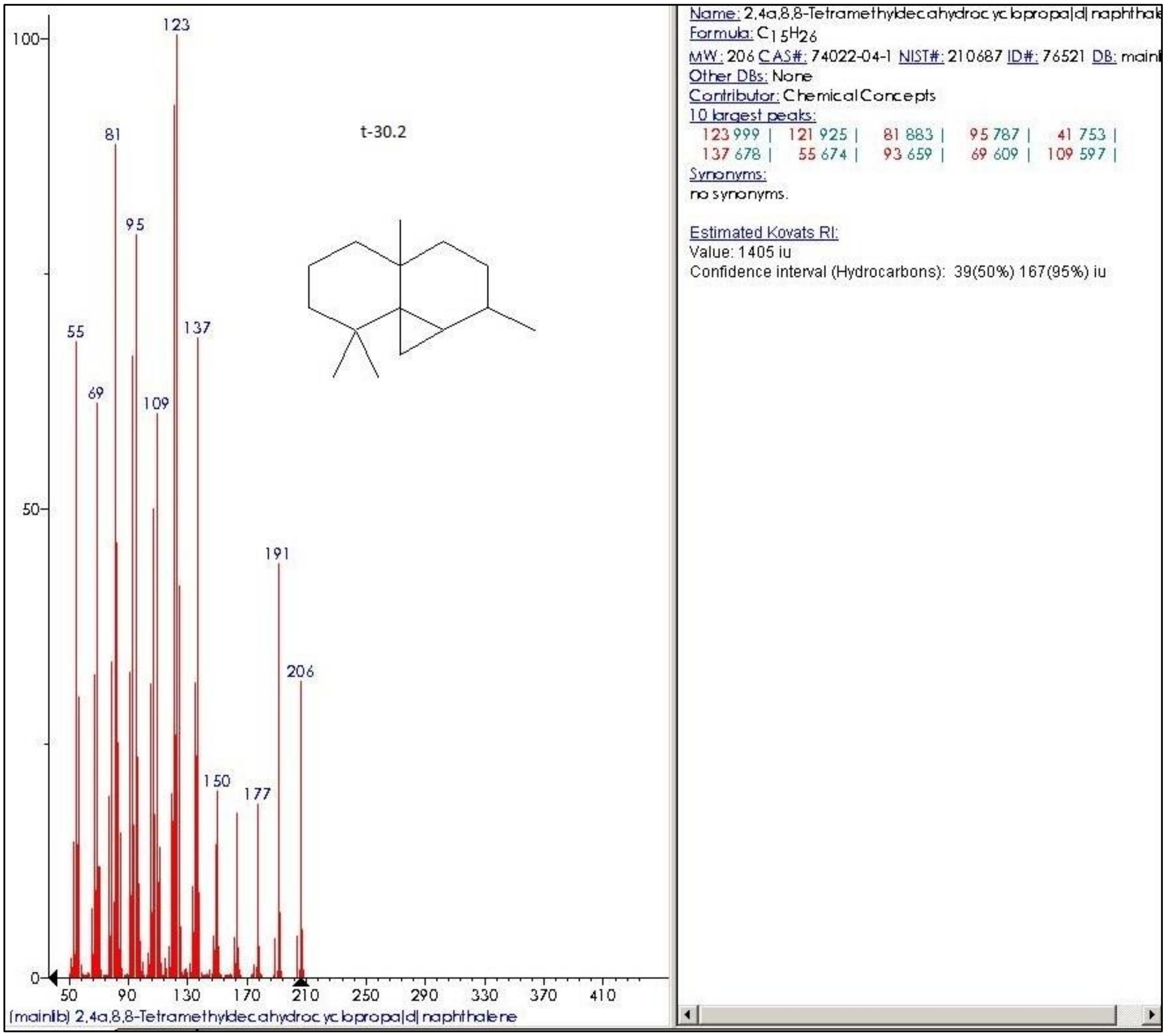
Name: 9H-Fluorene, 9-methylene-
Formula: $C_{14}H_{10}$
MW: 178 CAS#: 4425-82-5 NIST#: 55617 ID#: 22354 DB: replib
Other DBs: HODOC
Contributor: HCACAV,60(3)1073(1977);7A; M. NEVENSCHWAN
10 largest peaks:

```
  178 999 |  176 180 |   76 160 |  179 150 |   89 130 |
   88 100 |  177 100 |  152  90 |  151  70 |   75  60 |
```

Synonyms:
1.9-Methylene-fluorene
2.9-Methylene-9H-fluorene #

Estimated Kovats RI:
Value: 1560 iu
Confidence interval (Aromatic Hydrocarbons): 55(50%) 238(95%)

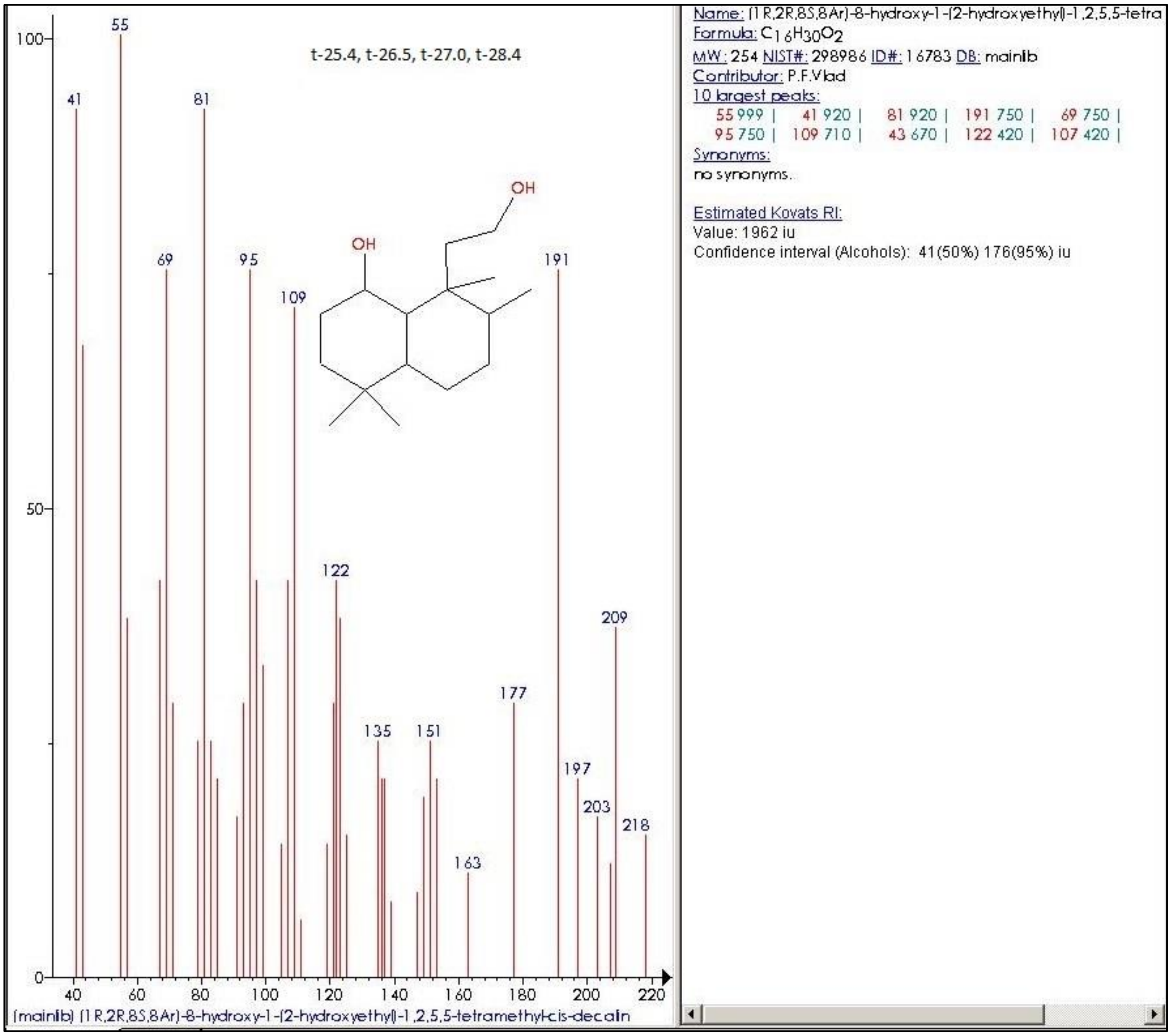
Figure C-12



Name: 2,4a,8,8-Tetramethyldecahydrocyclopropa[d]naphthal
Formula: $C_{15}H_{26}$
MW: 206 CAS#: 74022-04-1 NIST#: 210687 ID#: 76521 DB: mainl
Other DBs: None
Contributor: Chemical Concepts
10 largest peaks:
123 999 | 121 925 | 81 883 | 95 787 | 41 753 |
137 678 | 55 674 | 93 659 | 69 609 | 109 597 |
Synonyms:
no synonyms.

Estimated Kovats RI:
Value: 1405 iu
Confidence interval (Hydrocarbons): 39(50%) 167(95%) iu

Figure C-13

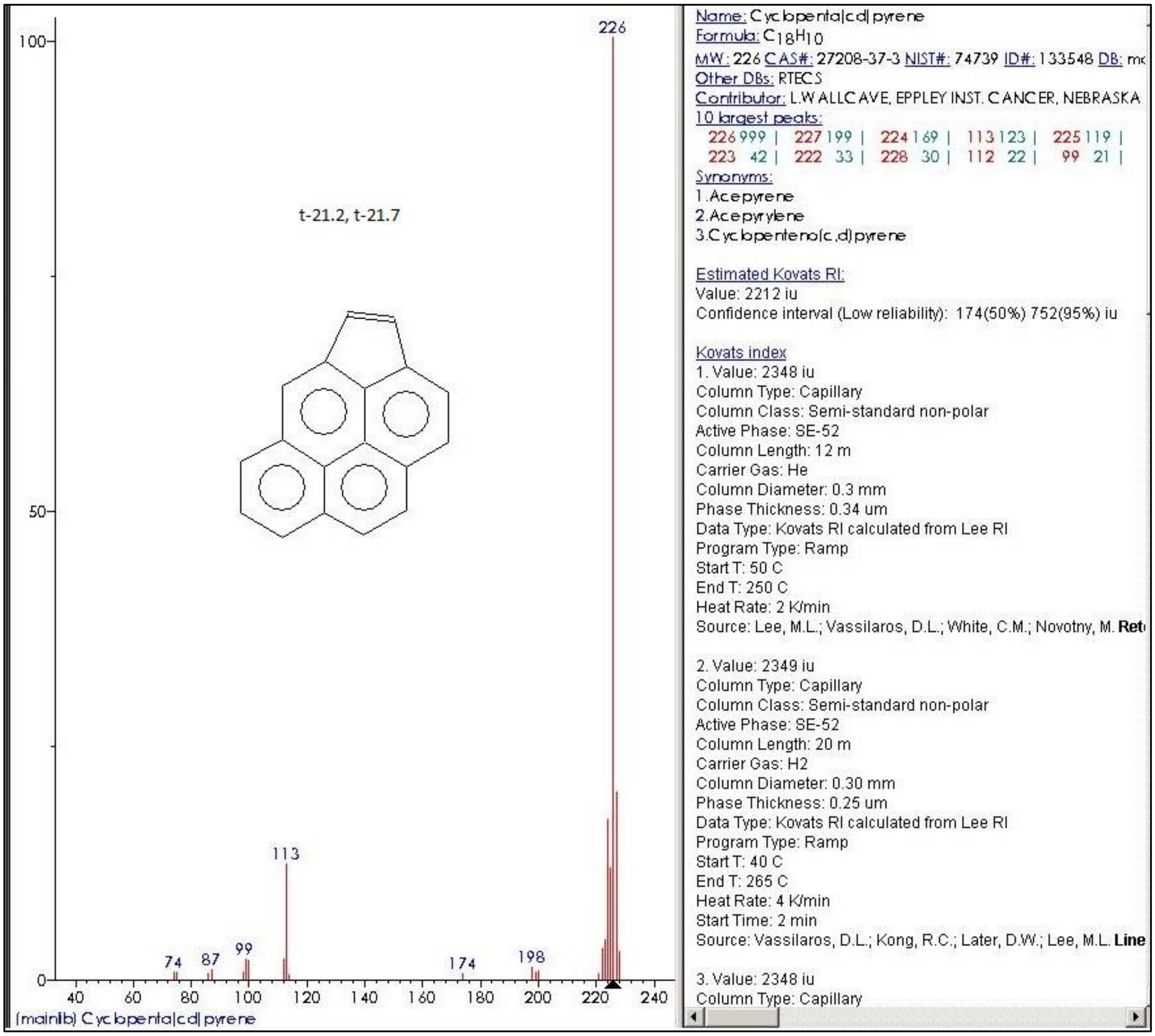


(mainlib) (1R,2R,8S,8Ar)-8-hydroxy-1-(2-hydroxyethyl)-1,2,5,5-tetramethyl-cis-decalin

Name: (1R,2R,8S,8Ar)-8-hydroxy-1-(2-hydroxyethyl)-1,2,5,5-tetra
Formula: $C_{16}H_{30}O_2$
MW: 254 NIST#: 298986 ID#: 16783 DB: mainlib
Contributor: P.F.Vlad
10 largest peaks:
55 999 | 41 920 | 81 920 | 191 750 | 69 750 |
95 750 | 109 710 | 43 670 | 122 420 | 107 420 |
Synonyms:
no synonyms.

Estimated Kovats RI:
Value: 1962 iu
Confidence interval (Alcohols): 41(50%) 176(95%) iu

Figure C-14



Name: Cyclopenta[cd]pyrene
Formula: $C_{18}H_{10}$
MW: 226 CAS#: 27208-37-3 NIST#: 74739 ID#: 133548 DB: m
Other DBs: RTECS
Contributor: L.WALLCAVE, EPPLEY INST. CANCER, NEBRASKA
10 largest peaks:
226 999 | 227 199 | 224 169 | 113 123 | 225 119 |
223 42 | 222 33 | 228 30 | 112 22 | 99 21 |
Synonyms:
1.Acepyrene
2.Acepyrylene
3.Cyclopenteno(c,d)pyrene

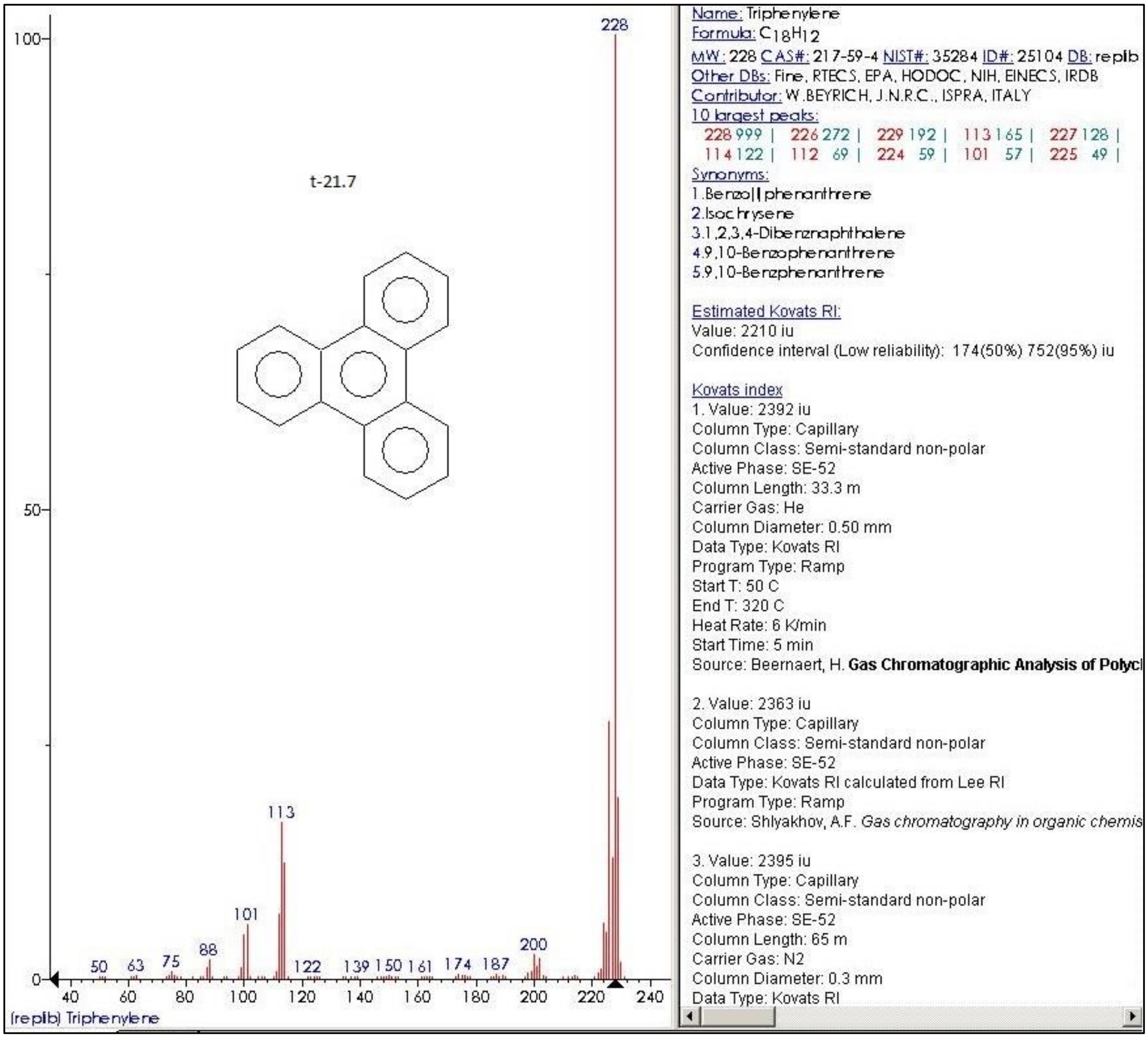
Estimated Kovats RI:
Value: 2212 iu
Confidence interval (Low reliability): 174(50%) 752(95%) iu

Kovats index
1. Value: 2348 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Column Length: 12 m
Carrier Gas: He
Column Diameter: 0.3 mm
Phase Thickness: 0.34 um
Data Type: Kovats RI calculated from Lee RI
Program Type: Ramp
Start T: 50 C
End T: 250 C
Heat Rate: 2 K/min
Source: Lee, M.L.; Vassilaros, D.L.; White, C.M.; Novotny, M. **Ret**

2. Value: 2349 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Column Length: 20 m
Carrier Gas: H2
Column Diameter: 0.30 mm
Phase Thickness: 0.25 um
Data Type: Kovats RI calculated from Lee RI
Program Type: Ramp
Start T: 40 C
End T: 265 C
Heat Rate: 4 K/min
Start Time: 2 min
Source: Vassilaros, D.L.; Kong, R.C.; Later, D.W.; Lee, M.L. **Line**

3. Value: 2348 iu
Column Type: Capillary

Figure C-15



Name: Triphenylene
Formula: $C_{18}H_{12}$
MW: 228 CAS#: 217-59-4 NIST#: 35284 ID#: 25104 DB: replib
Other DBs: Fine, RTECS, EPA, HODOC, NIH, EINECS, IRDB
Contributor: W.BEYRICH, J.N.R.C., ISPRA, ITALY
10 largest peaks:
228 999 | 226 272 | 229 192 | 113 165 | 227 128 |
114 122 | 112 69 | 224 59 | 101 57 | 225 49 |
Synonyms:
1.Benzo[l]phenanthrene
2.Isochrysene
3.1,2,3,4-Dibenznaphthalene
4.9,10-Benzophenanthrene
5.9,10-Benzphenanthrene

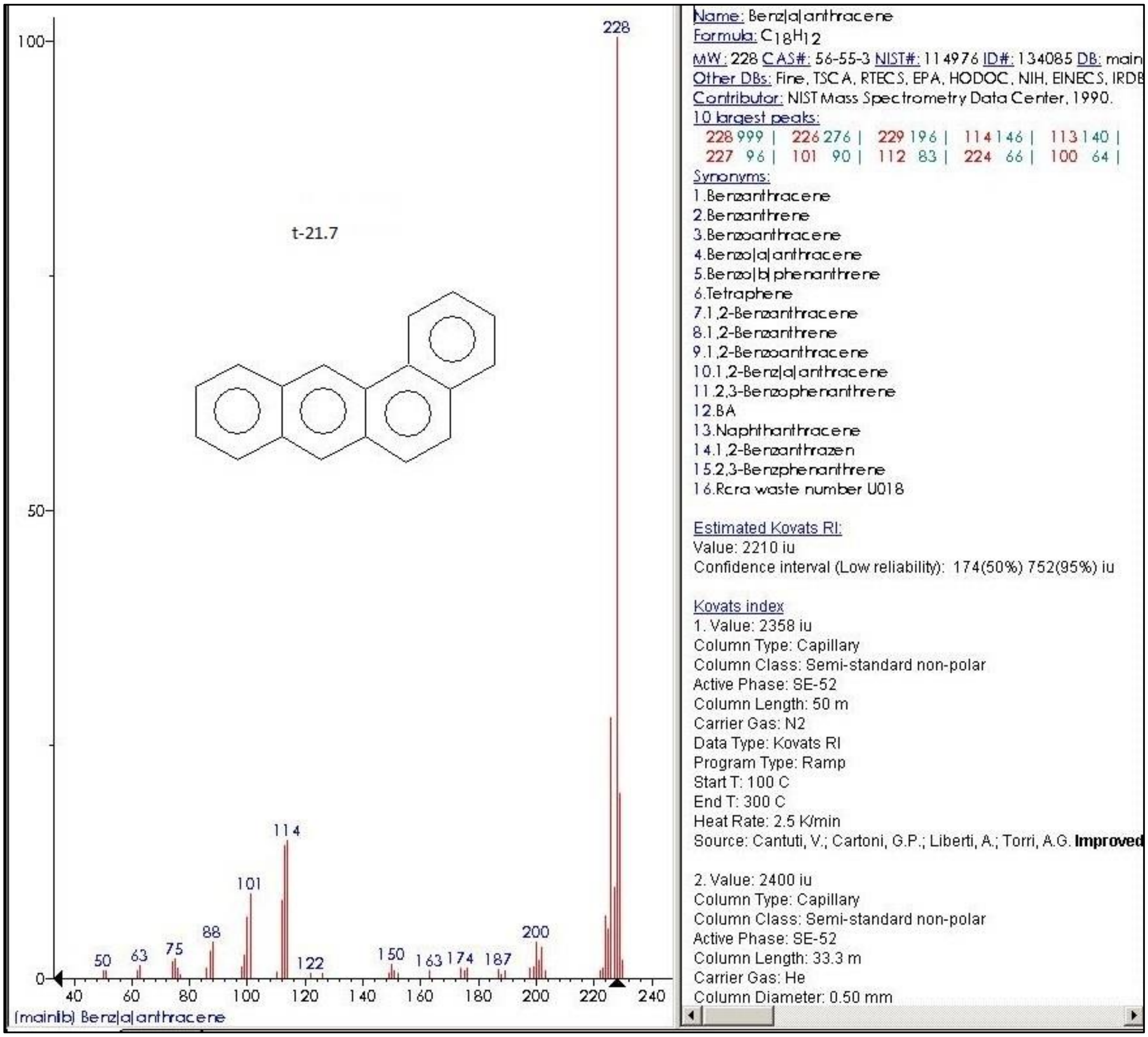
Estimated Kovats RI:
Value: 2210 iu
Confidence interval (Low reliability): 174(50%) 752(95%) iu

Kovats index
1. Value: 2392 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Column Length: 33.3 m
Carrier Gas: He
Column Diameter: 0.50 mm
Data Type: Kovats RI
Program Type: Ramp
Start T: 50 C
End T: 320 C
Heat Rate: 6 K/min
Start Time: 5 min
Source: Beernaert, H. **Gas Chromatographic Analysis of Polycl**

2. Value: 2363 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Data Type: Kovats RI calculated from Lee RI
Program Type: Ramp
Source: Shlyakhov, A.F. *Gas chromatography in organic chemis*

3. Value: 2395 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Column Length: 65 m
Carrier Gas: N2
Column Diameter: 0.3 mm
Data Type: Kovats RI

Figure C-16



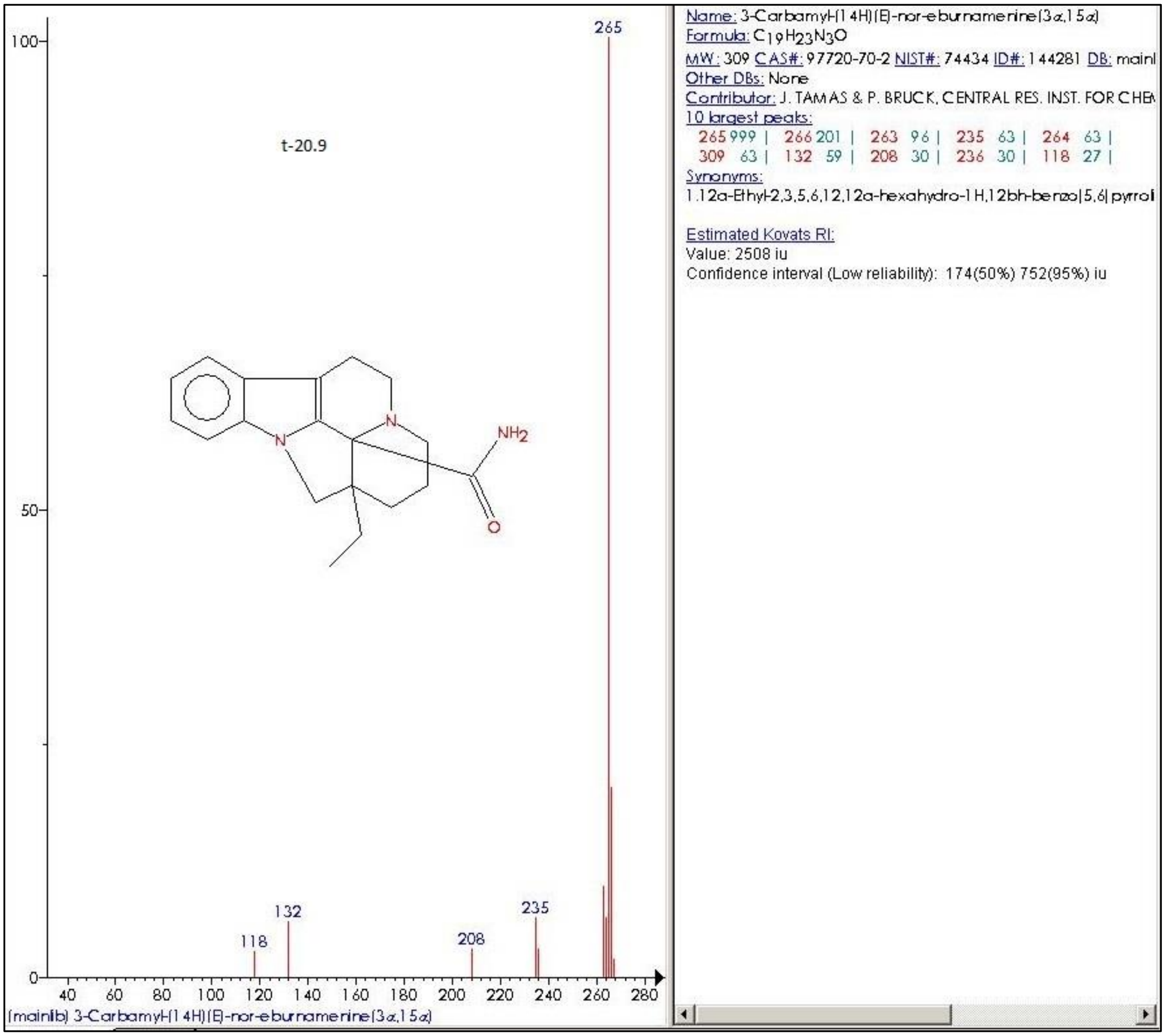
Name: Benz|a|anthracene
Formula: $C_{18}H_{12}$
MW: 228 CAS#: 56-55-3 NIST#: 114976 ID#: 134085 DB: main
Other DBs: Fine, TSCA, RTECS, EPA, HODOC, NIH, EINECS, IRDB
Contributor: NIST Mass Spectrometry Data Center, 1990.
10 largest peaks:
228 999 | 226 276 | 229 196 | 114 146 | 113 140 |
227 96 | 101 90 | 112 83 | 224 66 | 100 64 |
Synonyms:
1.Benzanthracene
2.Benzanthrene
3.Benzoanthracene
4.Benzo|a|anthracene
5.Benzo|b|phenanthrene
6.Tetraphene
7.1,2-Benzanthracene
8.1,2-Benzanthrene
9.1,2-Benzoanthracene
10.1,2-Benz|a|anthracene
11.2,3-Benzophenanthrene
12.BA
13.Naphthanthracene
14.1,2-Benzanthrazen
15.2,3-Benzphenanthrene
16.Rcra waste number U018

Estimated Kovats RI:
Value: 2210 iu
Confidence interval (Low reliability): 174(50%) 752(95%) iu

Kovats index
1. Value: 2358 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Column Length: 50 m
Carrier Gas: N2
Data Type: Kovats RI
Program Type: Ramp
Start T: 100 C
End T: 300 C
Heat Rate: 2.5 K/min
Source: Cantuti, V.; Cartoni, G.P.; Liberti, A.; Torri, A.G. **Improved**

2. Value: 2400 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Column Length: 33.3 m
Carrier Gas: He
Column Diameter: 0.50 mm

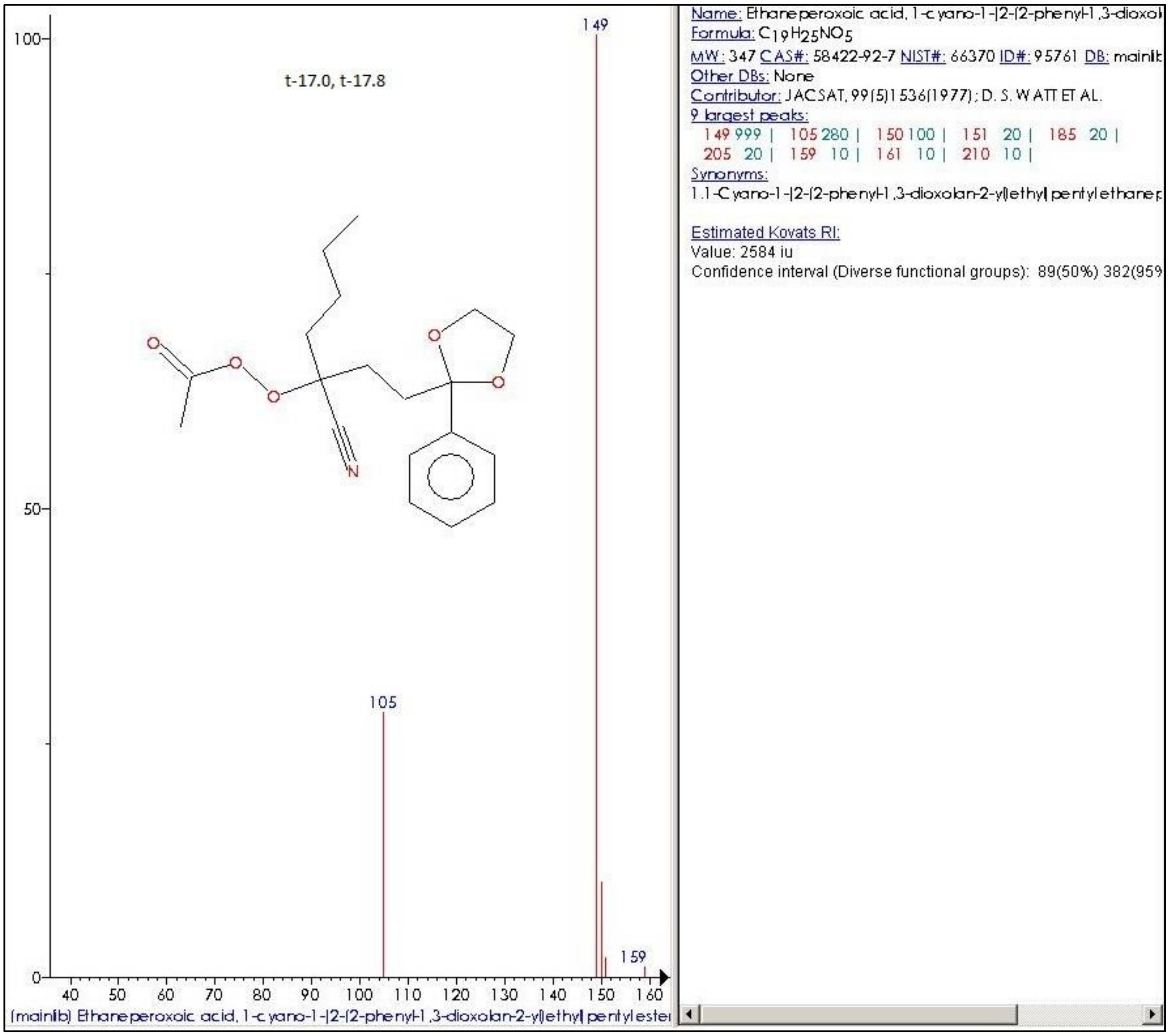
Figure C-17



Name: 3-Carbamyl-(14H)(E)-nor-eburnamenine(3α,15α)
Formula: $C_{19}H_{23}N_3O$
MW: 309 CAS#: 97720-70-2 NIST#: 74434 ID#: 144281 DB: mainl
Other DBs: None
Contributor: J. TAMAS & P. BRUCK, CENTRAL RES. INST. FOR CHEM
10 largest peaks:
265 999 | 266 201 | 263 96 | 235 63 | 264 63 |
309 63 | 132 59 | 208 30 | 236 30 | 118 27 |
Synonyms:
1.12a-Ethyl-2,3,5,6,12,12a-hexahydro-1H,12bh-benzo[5,6]pyrroli

Estimated Kovats RI:
Value: 2508 iu
Confidence interval (Low reliability): 174(50%) 752(95%) iu

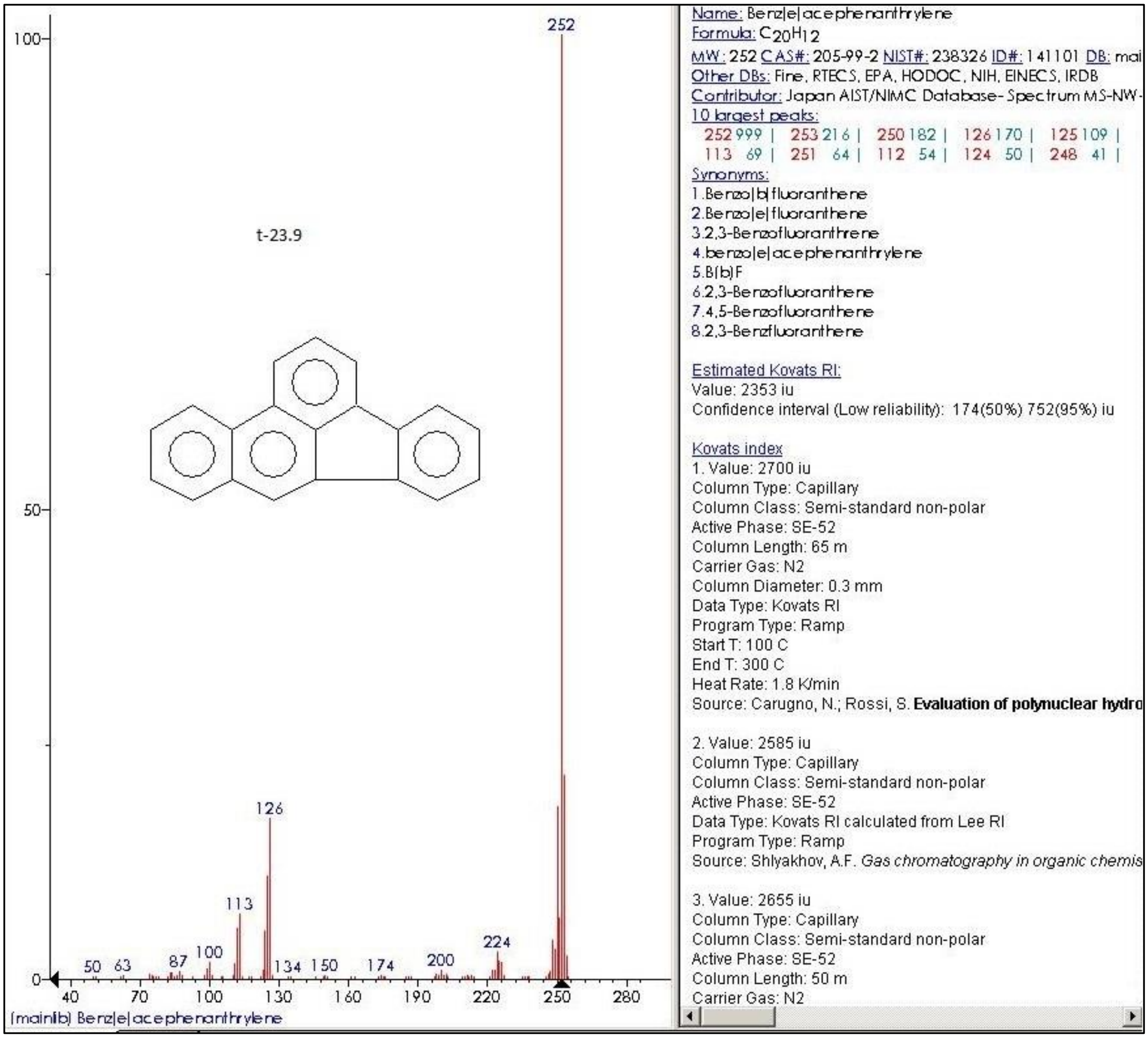
Figure C-18



Name: Ethaneperoxoic acid, 1-cyano-1-[2-(2-phenyl-1,3-dioxol
Formula: $C_{19}H_{25}NO_5$
MW: 347 CAS#: 58422-92-7 NIST#: 66370 ID#: 95761 DB: mainli
Other DBs: None
Contributor: JACSAT, 99(5)1536(1977); D. S. WATT ET AL.
9 largest peaks:
149 999 | 105 280 | 150 100 | 151 20 | 185 20 |
205 20 | 159 10 | 161 10 | 210 10 |
Synonyms:
1.1-Cyano-1-[2-(2-phenyl-1,3-dioxolan-2-yl)ethyl] pentyl ethane

Estimated Kovats RI:
Value: 2584 iu
Confidence interval (Diverse functional groups): 89(50%) 382(95

Figure C-19



(mainlib) Benz|e|acephenanthrylene

Name: Benz|e|acephenanthrylene
Formula: $C_{20}H_{12}$
MW: 252 CAS#: 205-99-2 NIST#: 238326 ID#: 141101 DB: mai
Other DBs: Fine, RTECS, EPA, HODOC, NIH, EINECS, IRDB
Contributor: Japan AIST/NIMC Database- Spectrum MS-NW-
10 largest peaks:
252 999 | 253 216 | 250 182 | 126 170 | 125 109 |
113 69 | 251 64 | 112 54 | 124 50 | 248 41 |
Synonyms:
1.Benzo|b|fluoranthene
2.Benzo|e|fluoranthene
3.2,3-Benzofluoranthrene
4.benzo|e|acephenanthrylene
5.B(b)F
6.2,3-Benzofluoranthene
7.4,5-Benzofluoranthene
8.2,3-Benzfluoranthene

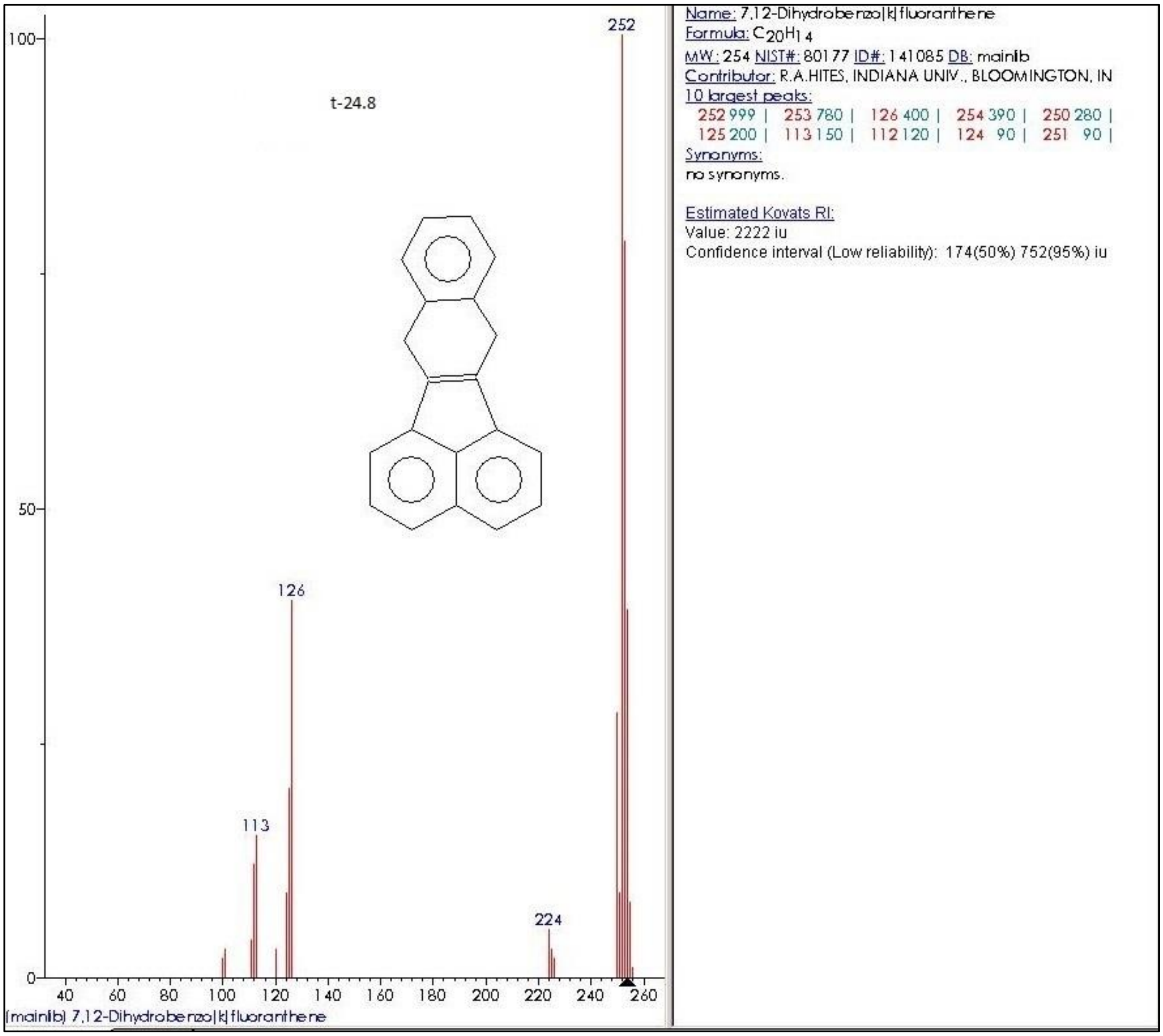
Estimated Kovats RI:
Value: 2353 iu
Confidence interval (Low reliability): 174(50%) 752(95%) iu

Kovats index
1. Value: 2700 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Column Length: 65 m
Carrier Gas: N2
Column Diameter: 0.3 mm
Data Type: Kovats RI
Program Type: Ramp
Start T: 100 C
End T: 300 C
Heat Rate: 1.8 K/min
Source: Carugno, N.; Rossi, S. **Evaluation of polynuclear hydro**

2. Value: 2585 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Data Type: Kovats RI calculated from Lee RI
Program Type: Ramp
Source: Shlyakhov, A.F. *Gas chromatography in organic chemis*

3. Value: 2655 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Column Length: 50 m
Carrier Gas: N2

Figure C-20



Name: 7,12-Dihydrobenzo[k]fluoranthene

Formula: $C_{20}H_{14}$

MW: 254 NIST#: 80177 ID#: 141085 DB: mainlib

Contributor: R.A.HITES, INDIANA UNIV., BLOOMINGTON, IN

10 largest peaks:

252 999 | 253 780 | 126 400 | 254 390 | 250 280 |
125 200 | 113 150 | 112 120 | 124 90 | 251 90 |

Synonyms:

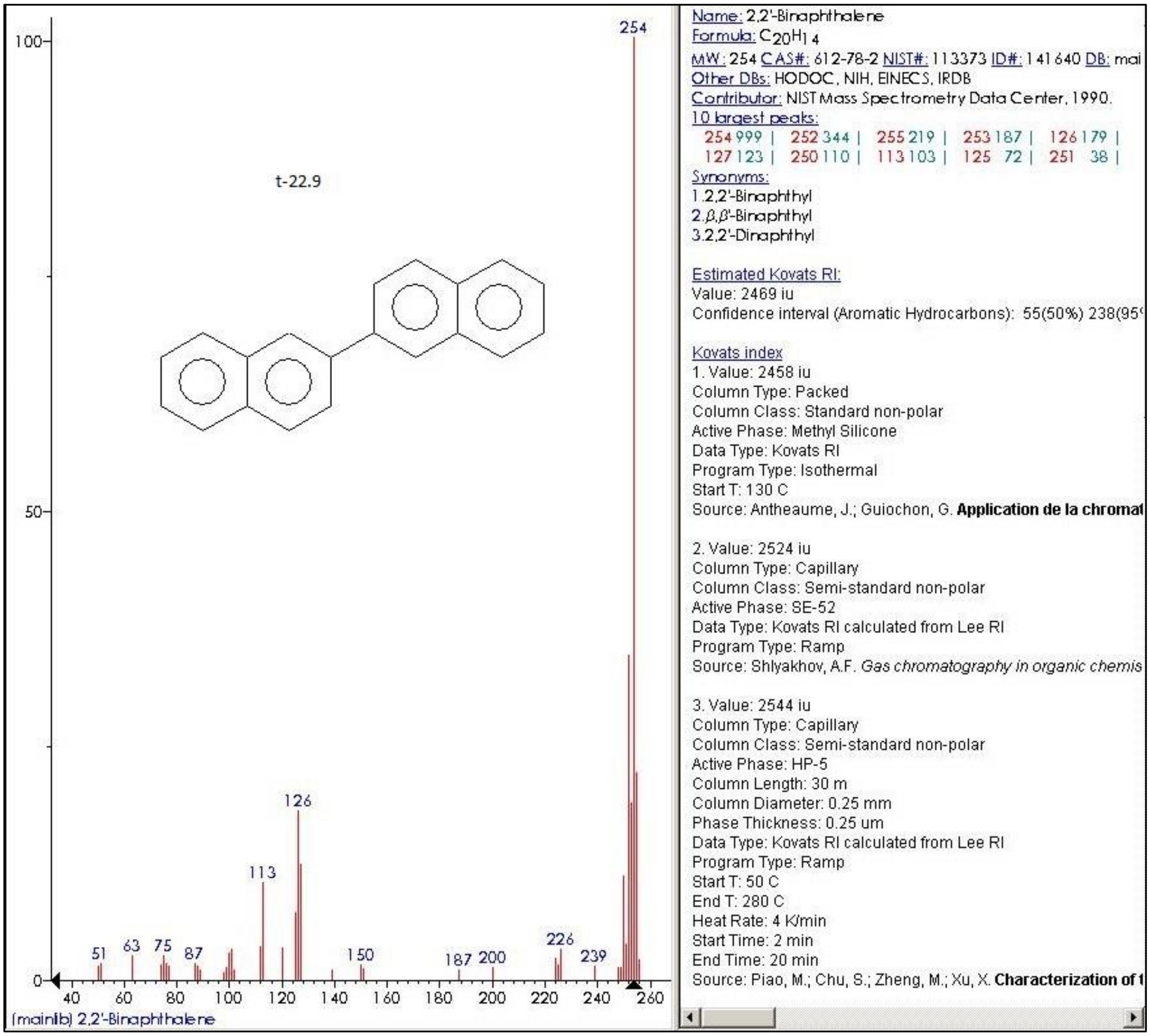
no synonyms.

Estimated Kovats RI:

Value: 2222 iu

Confidence interval (Low reliability): 174(50%) 752(95%) iu

Figure C-21



Name: 2,2'-Binaphthalene
Formula: $C_{20}H_{14}$
MW: 254 CAS#: 612-78-2 NIST#: 113373 ID#: 141640 DB: mai
Other DBs: HODOC, NIH, EINECS, IRDB
Contributor: NIST Mass Spectrometry Data Center, 1990.
10 largest peaks:
254 999 | 252 344 | 255 219 | 253 187 | 126 179 |
127 123 | 250 110 | 113 103 | 125 72 | 251 38 |
Synonyms:
1.2,2'-Binaphthyl
2.β,β'-Binaphthyl
3.2,2'-Dinaphthyl

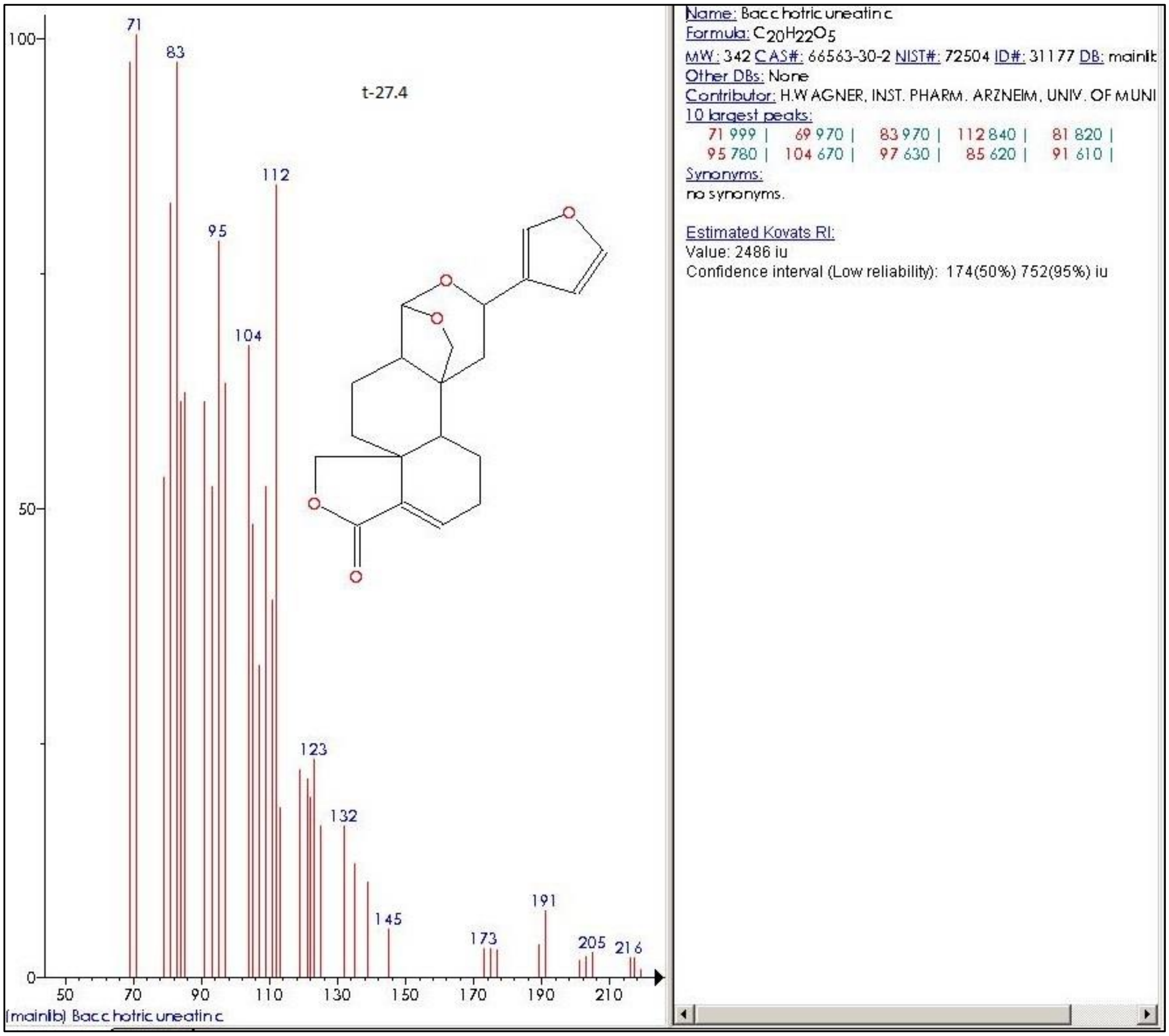
Estimated Kovats RI:
Value: 2469 iu
Confidence interval (Aromatic Hydrocarbons): 55(50%) 238(95

Kovats index
1. Value: 2458 iu
Column Type: Packed
Column Class: Standard non-polar
Active Phase: Methyl Silicone
Data Type: Kovats RI
Program Type: Isothermal
Start T: 130 C
Source: Antheaume, J.; Guiochon, G. **Application de la chromat**

2. Value: 2524 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Data Type: Kovats RI calculated from Lee RI
Program Type: Ramp
Source: Shlyakhov, A.F. *Gas chromatography in organic chemis*

3. Value: 2544 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: HP-5
Column Length: 30 m
Column Diameter: 0.25 mm
Phase Thickness: 0.25 um
Data Type: Kovats RI calculated from Lee RI
Program Type: Ramp
Start T: 50 C
End T: 280 C
Heat Rate: 4 K/min
Start Time: 2 min
End Time: 20 min
Source: Piao, M.; Chu, S.; Zheng, M.; Xu, X. **Characterization of**

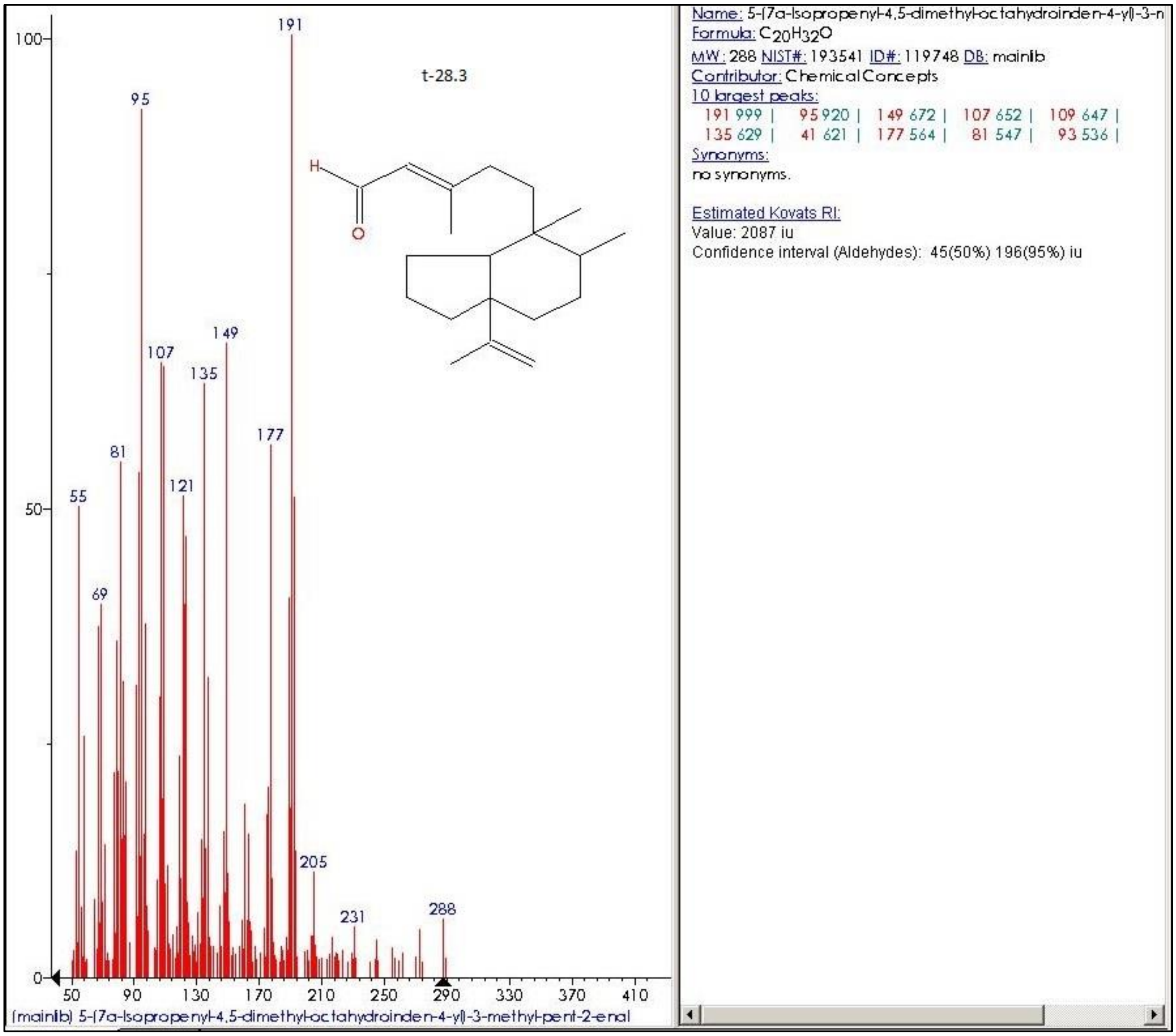
Figure C-22



Name: Bacchotricuneatin c
Formula: $C_{20}H_{22}O_5$
MW: 342 CAS#: 66563-30-2 NIST#: 72504 ID#: 31177 DB: mainli
Other DBs: None
Contributor: H.WAGNER, INST. PHARM. ARZNEIM, UNIV. OF MUNI
10 largest peaks:
71 999 | 69 970 | 83 970 | 112 840 | 81 820 |
95 780 | 104 670 | 97 630 | 85 620 | 91 610 |
Synonyms:
no synonyms.

Estimated Kovats RI:
Value: 2486 iu
Confidence interval (Low reliability): 174(50%) 752(95%) iu

Figure C-23

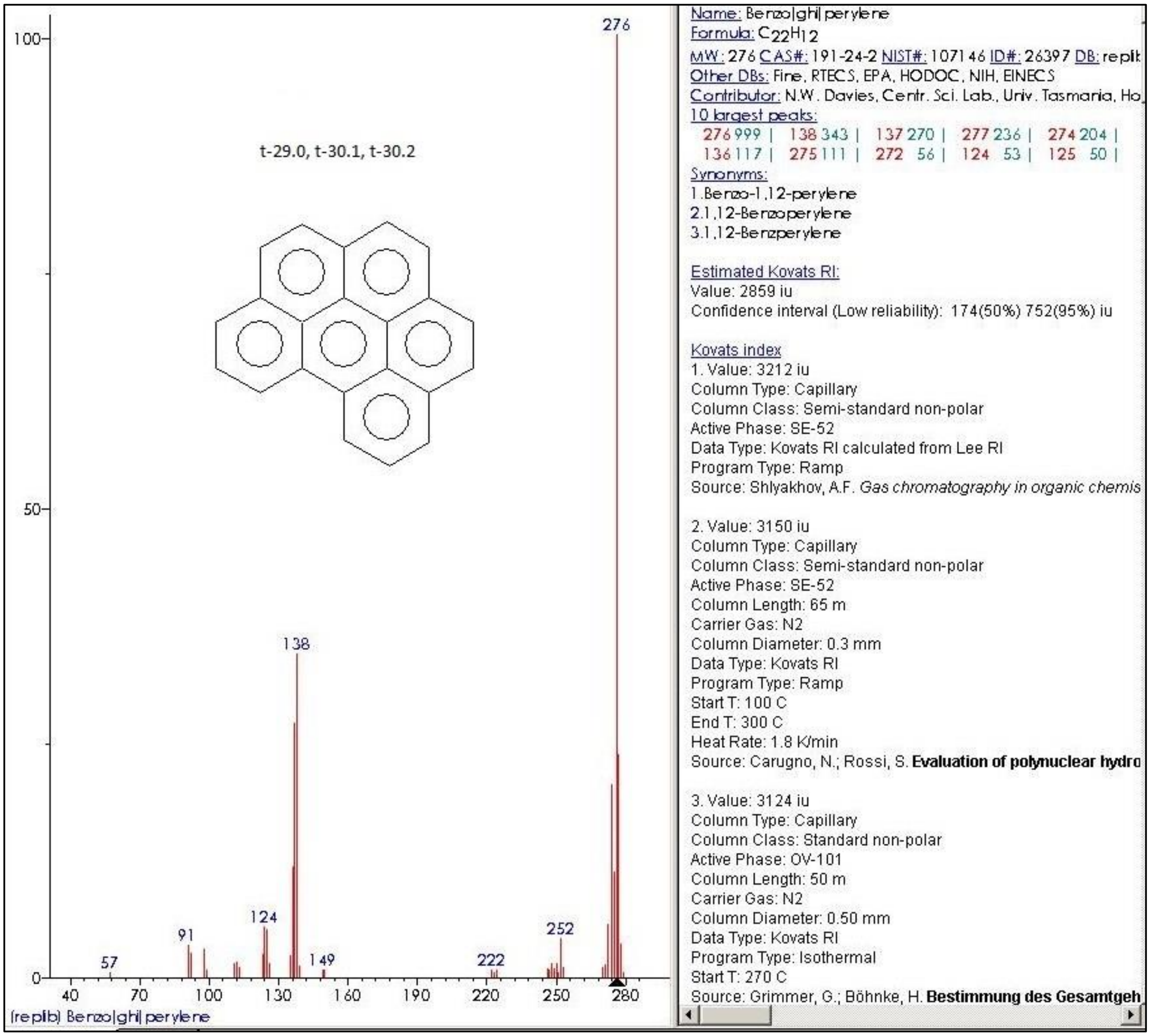


(mainlib) 5-(7a-Isopropenyl-4,5-dimethyl-octahydroinden-4-yl)-3-methyl-pent-2-enal

Name: 5-(7a-Isopropenyl-4,5-dimethyl-octahydroinden-4-yl)-3-n
Formula: $C_{20}H_{32}O$
MW: 288 NIST#: 193541 ID#: 119748 DB: mainlib
Contributor: Chemical Concepts
10 largest peaks:
191 999 | 95 920 | 149 672 | 107 652 | 109 647 |
135 629 | 41 621 | 177 564 | 81 547 | 93 536 |
Synonyms:
no synonyms.

Estimated Kovats RI:
Value: 2087 iu
Confidence interval (Aldehydes): 45(50%) 196(95%) iu

Figure C-24



Name: Benzo[ghi]perylene
Formula: $C_{22}H_{12}$
MW: 276 CAS#: 191-24-2 NIST#: 107146 ID#: 26397 DB: replib
Other DBs: Fine, RTECS, EPA, HODOC, NIH, EINECS
Contributor: N.W. Davies, Centr. Sci. Lab., Univ. Tasmania, Ho
10 largest peaks:
276 999 | 138 343 | 137 270 | 277 236 | 274 204 |
136 117 | 275 111 | 272 56 | 124 53 | 125 50 |
Synonyms:
1.Benzo-1,12-perylene
2.1,12-Benzoperylene
3.1,12-Benzperylene

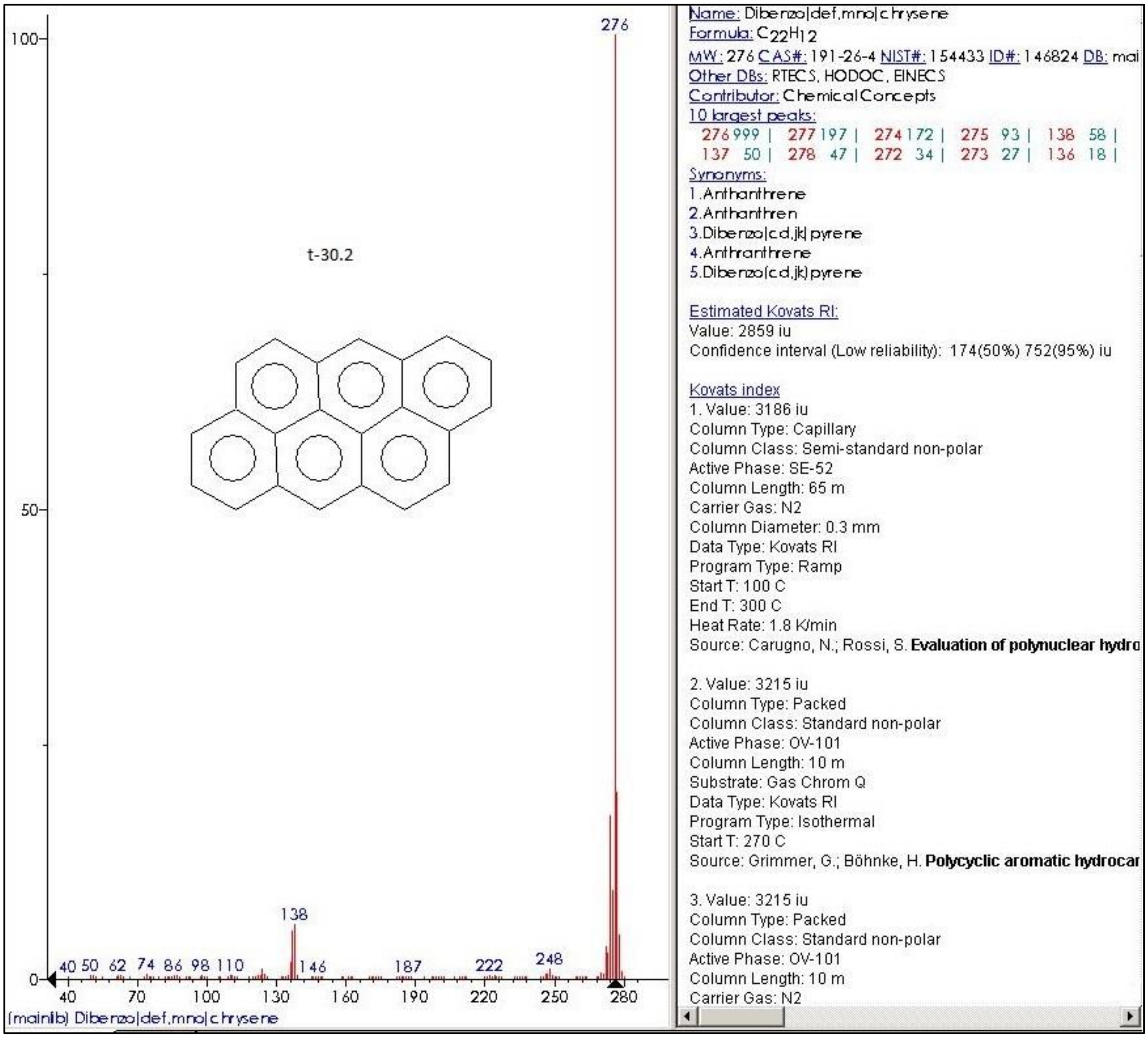
Estimated Kovats RI:
Value: 2859 iu
Confidence interval (Low reliability): 174(50%) 752(95%) iu

Kovats index
1. Value: 3212 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Data Type: Kovats RI calculated from Lee RI
Program Type: Ramp
Source: Shlyakhov, A.F. *Gas chromatography in organic chemis*

2. Value: 3150 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Column Length: 65 m
Carrier Gas: N2
Column Diameter: 0.3 mm
Data Type: Kovats RI
Program Type: Ramp
Start T: 100 C
End T: 300 C
Heat Rate: 1.8 K/min
Source: Carugno, N.; Rossi, S. **Evaluation of polynuclear hydro**

3. Value: 3124 iu
Column Type: Capillary
Column Class: Standard non-polar
Active Phase: OV-101
Column Length: 50 m
Carrier Gas: N2
Column Diameter: 0.50 mm
Data Type: Kovats RI
Program Type: Isothermal
Start T: 270 C
Source: Grimmer, G.; Böhnke, H. **Bestimmung des Gesamtgeh**

Figure C-25



Name: Dibenzo|def,mno|chrysene
Formula: $C_{22}H_{12}$
MW: 276 CAS#: 191-26-4 NIST#: 154433 ID#: 146824 DB: mai
Other DBs: RTECS, HODOC, EINECS
Contributor: Chemical Concepts
10 largest peaks:
276 999 | 277 197 | 274 172 | 275 93 | 138 58 |
137 50 | 278 47 | 272 34 | 273 27 | 136 18 |
Synonyms:
1.Anthanthrene
2.Anthanthren
3.Dibenzo|cd,jk|pyrene
4.Anthranthrene
5.Dibenzo(cd,jk)pyrene

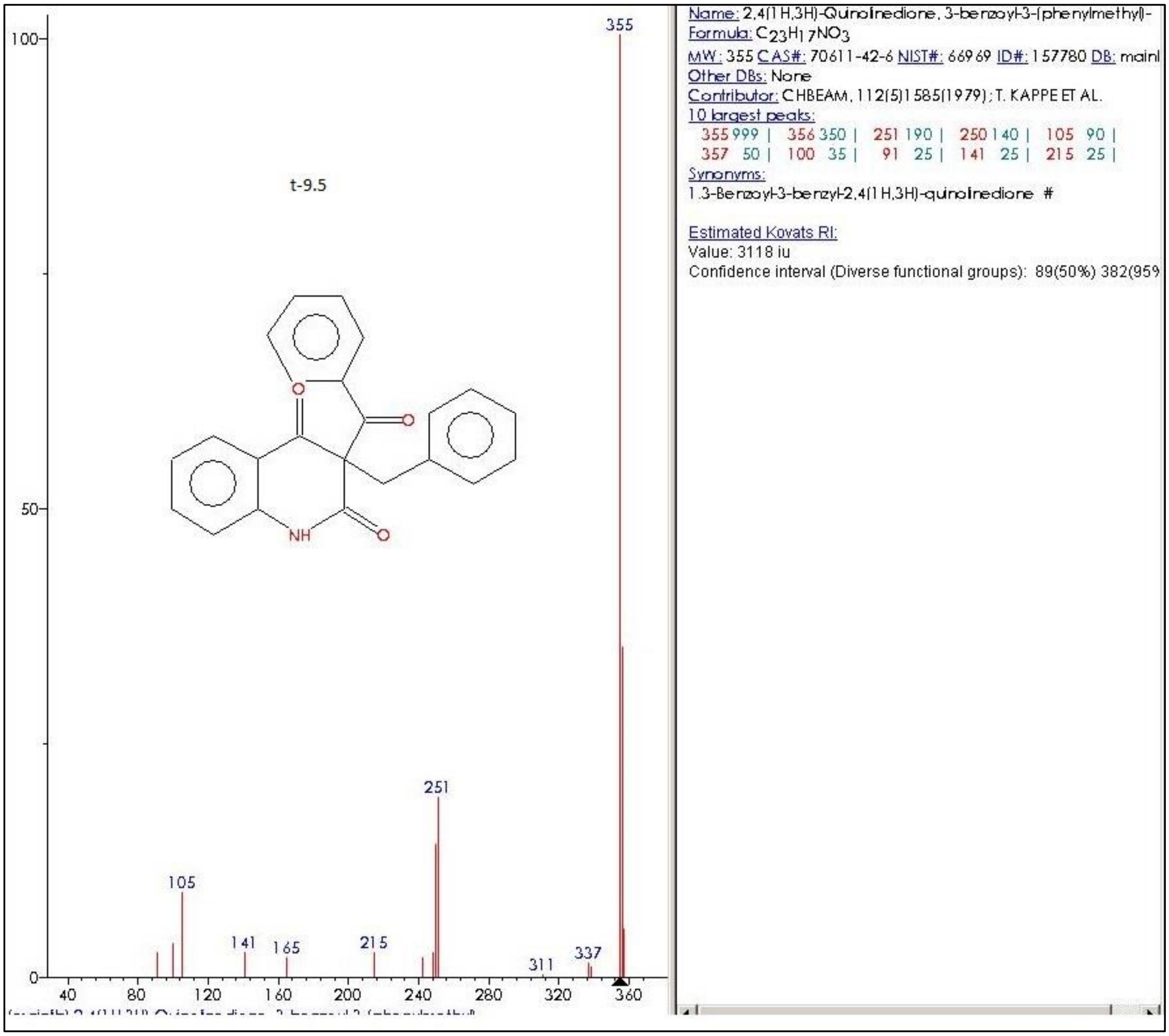
Estimated Kovats RI:
Value: 2859 iu
Confidence interval (Low reliability): 174(50%) 752(95%) iu

Kovats index
1. Value: 3186 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Column Length: 65 m
Carrier Gas: N2
Column Diameter: 0.3 mm
Data Type: Kovats RI
Program Type: Ramp
Start T: 100 C
End T: 300 C
Heat Rate: 1.8 K/min
Source: Carugno, N.; Rossi, S. **Evaluation of polynuclear hydro**

2. Value: 3215 iu
Column Type: Packed
Column Class: Standard non-polar
Active Phase: OV-101
Column Length: 10 m
Substrate: Gas Chrom Q
Data Type: Kovats RI
Program Type: Isothermal
Start T: 270 C
Source: Grimmer, G.; Böhnke, H. **Polycyclic aromatic hydrocar**

3. Value: 3215 iu
Column Type: Packed
Column Class: Standard non-polar
Active Phase: OV-101
Column Length: 10 m
Carrier Gas: N2

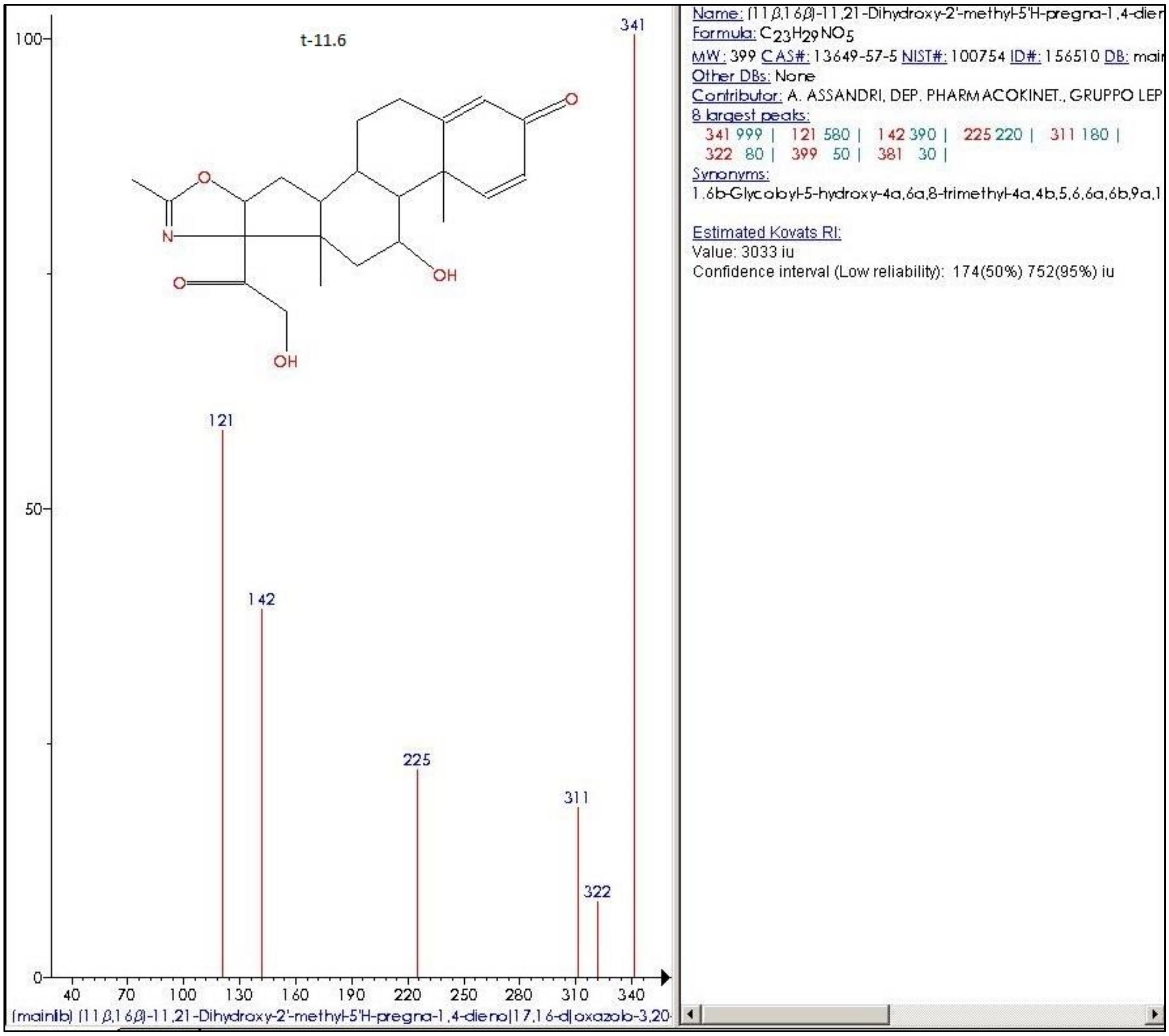
Figure C-26



Name: 2,4(1H,3H)-Quinolinedione, 3-benzoyl-3-(phenylmethyl)-
Formula: $C_{23}H_{17}NO_3$
MW: 355 CAS#: 70611-42-6 NIST#: 66969 ID#: 157780 DB: mainl
Other DBs: None
Contributor: CHBEAM, 112(5)1585(1979); T. KAPPE ET AL.
10 largest peaks:
355 999 | 356 350 | 251 190 | 250 140 | 105 90 |
357 50 | 100 35 | 91 25 | 141 25 | 215 25 |
Synonyms:
1.3-Benzoyl-3-benzyl-2,4(1H,3H)-quinolinedione #

Estimated Kovats RI:
Value: 3118 iu
Confidence interval (Diverse functional groups): 89(50%) 382(959

Figure C-27

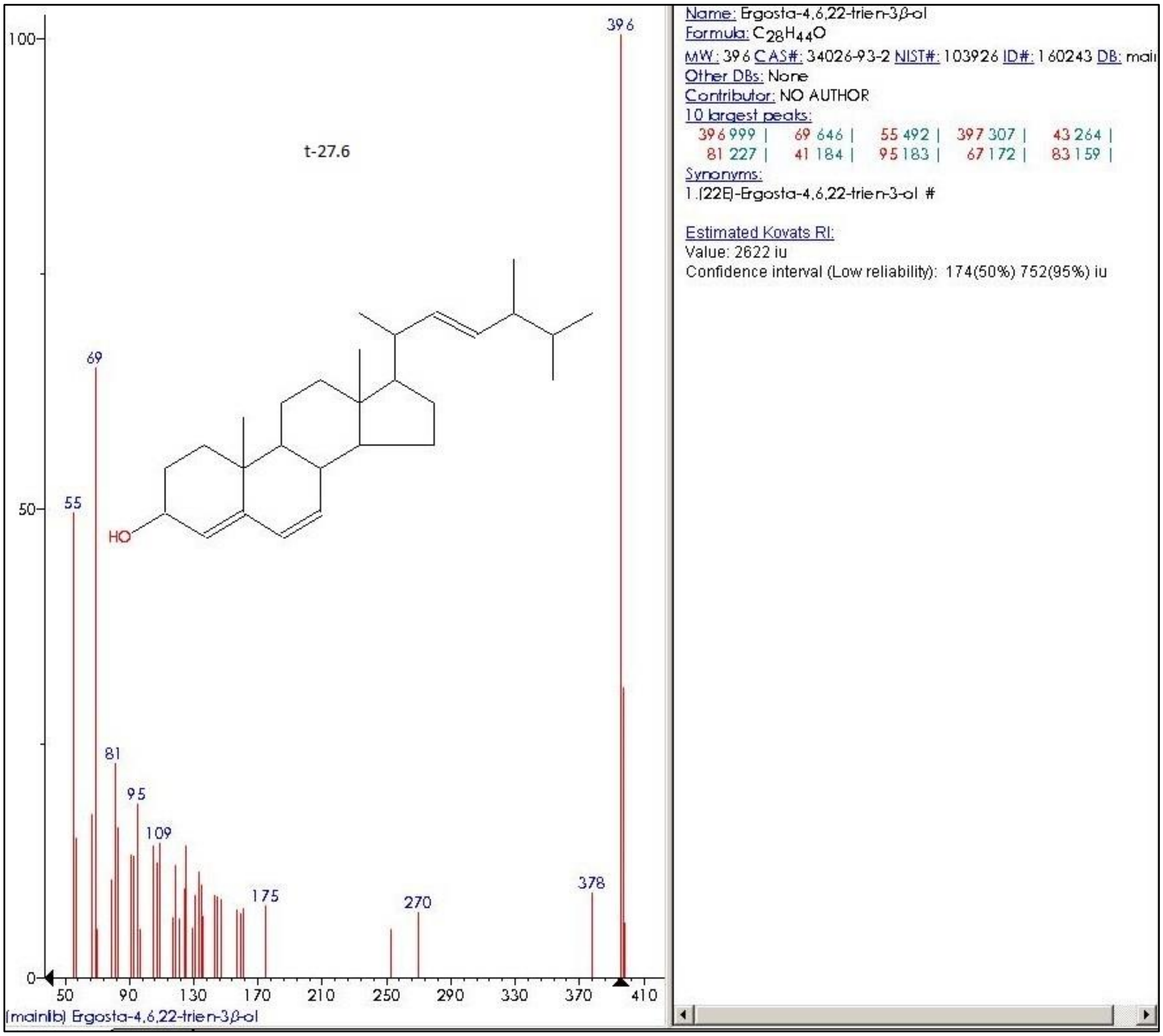


[mainlib] (11β,16β)-11,21-Dihydroxy-2'-methyl-5'H-pregna-1,4-dieno[17,16-d]oxazole-3,20-

Name: (11β,16β)-11,21-Dihydroxy-2'-methyl-5'H-pregna-1,4-dien
Formula: $C_{23}H_{29}NO_5$
MW: 399 CAS#: 13649-57-5 NIST#: 100754 ID#: 156510 DB: mai
Other DBs: None
Contributor: A. ASSANDRI, DEP. PHARMACOKINET., GRUPPO LEP
8 largest peaks:
341 999 | 121 580 | 142 390 | 225 220 | 311 180 |
322 80 | 399 50 | 381 30 |
Synonyms:
1,6b-Glycoloyl-5-hydroxy-4a,6a,8-trimethyl-4a,4b,5,6,6a,6b,9a,1

Estimated Kovats RI:
Value: 3033 iu
Confidence interval (Low reliability): 174(50%) 752(95%) iu

Figure C-28

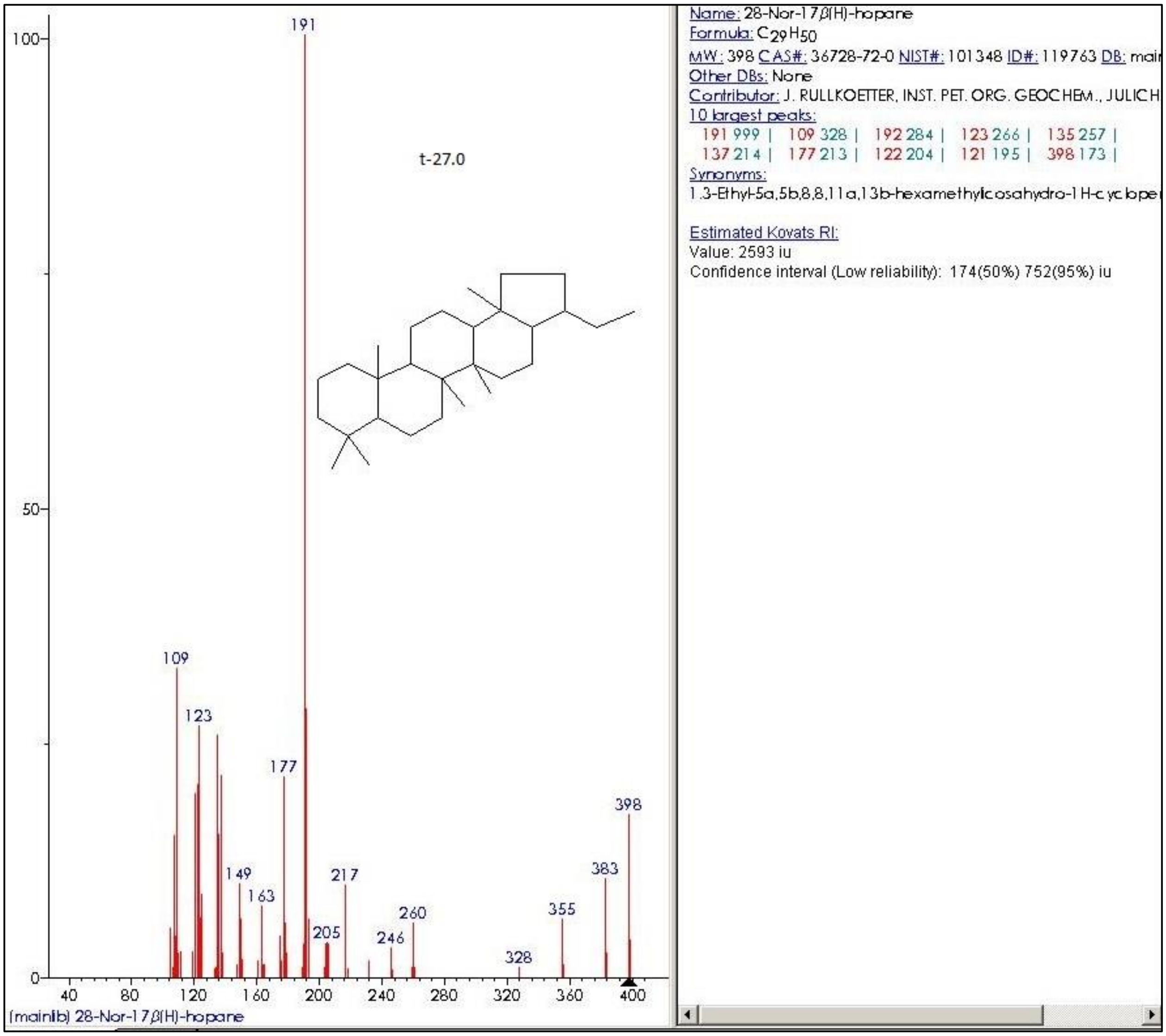


(mainlib) Ergosta-4,6,22-trien-3β-ol

Name: Ergosta-4,6,22-trien-3β-ol
Formula: $C_{28}H_{44}O$
MW: 396 CAS#: 34026-93-2 NIST#: 103926 ID#: 160243 DB: mai
Other DBs: None
Contributor: NO AUTHOR
10 largest peaks:
396 999 | 69 646 | 55 492 | 397 307 | 43 264 |
81 227 | 41 184 | 95 183 | 67 172 | 83 159 |
Synonyms:
1.(22E)-Ergosta-4,6,22-trien-3-ol #

Estimated Kovats RI:
Value: 2622 iu
Confidence interval (Low reliability): 174(50%) 752(95%) iu

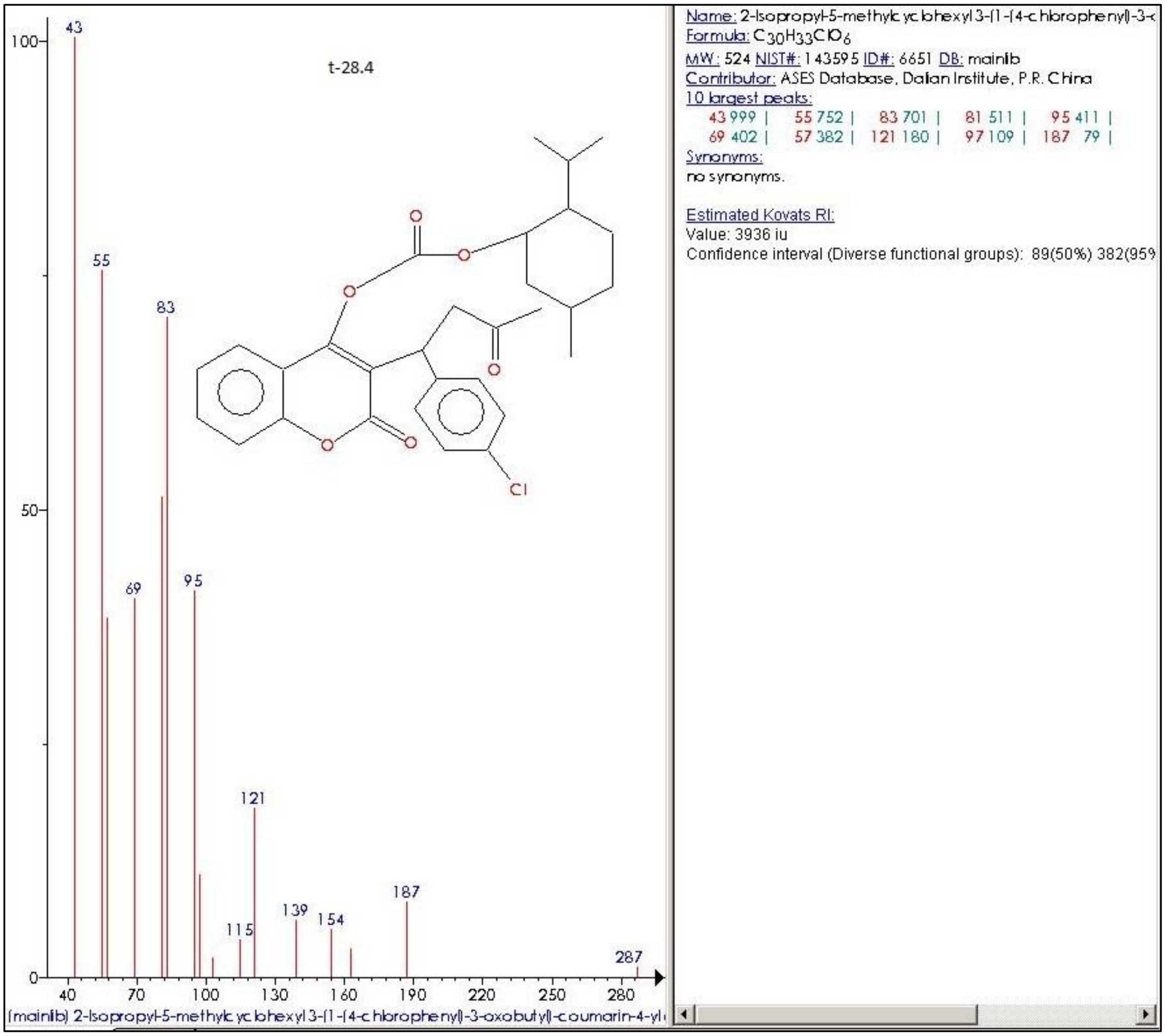
Figure C-29



Name: 28-Nor-17β(H)-hopane
Formula: $C_{29}H_{50}$
MW: 398 CAS#: 36728-72-0 NIST#: 101348 ID#: 119763 DB: mai
Other DBs: None
Contributor: J. RULLKOETTER, INST. PET. ORG. GEOCHEM., JULICH
10 largest peaks:
191 999 | 109 328 | 192 284 | 123 266 | 135 257 |
137 214 | 177 213 | 122 204 | 121 195 | 398 173 |
Synonyms:
1.3-Ethyl-5a,5b,8,8,11a,13b-hexamethylcosahydro-1H-cyclope

Estimated Kovats RI:
Value: 2593 iu
Confidence interval (Low reliability): 174(50%) 752(95%) iu

Figure C-30



Name: 2-Isopropyl-5-methylcyclohexyl 3-(1-(4-chlorophenyl)-3-

Formula: $C_{30}H_{33}ClO_6$

MW: 524 NIST#: 143595 ID#: 6651 DB: mainlib

Contributor: ASES Database, Dalian Institute, P.R. China

10 largest peaks:

```
43 999 |   55 752 |   83 701 |   81 511 |   95 411 |
69 402 |   57 382 |  121 180 |   97 109 |  187  79 |
```

Synonyms:

no synonyms.

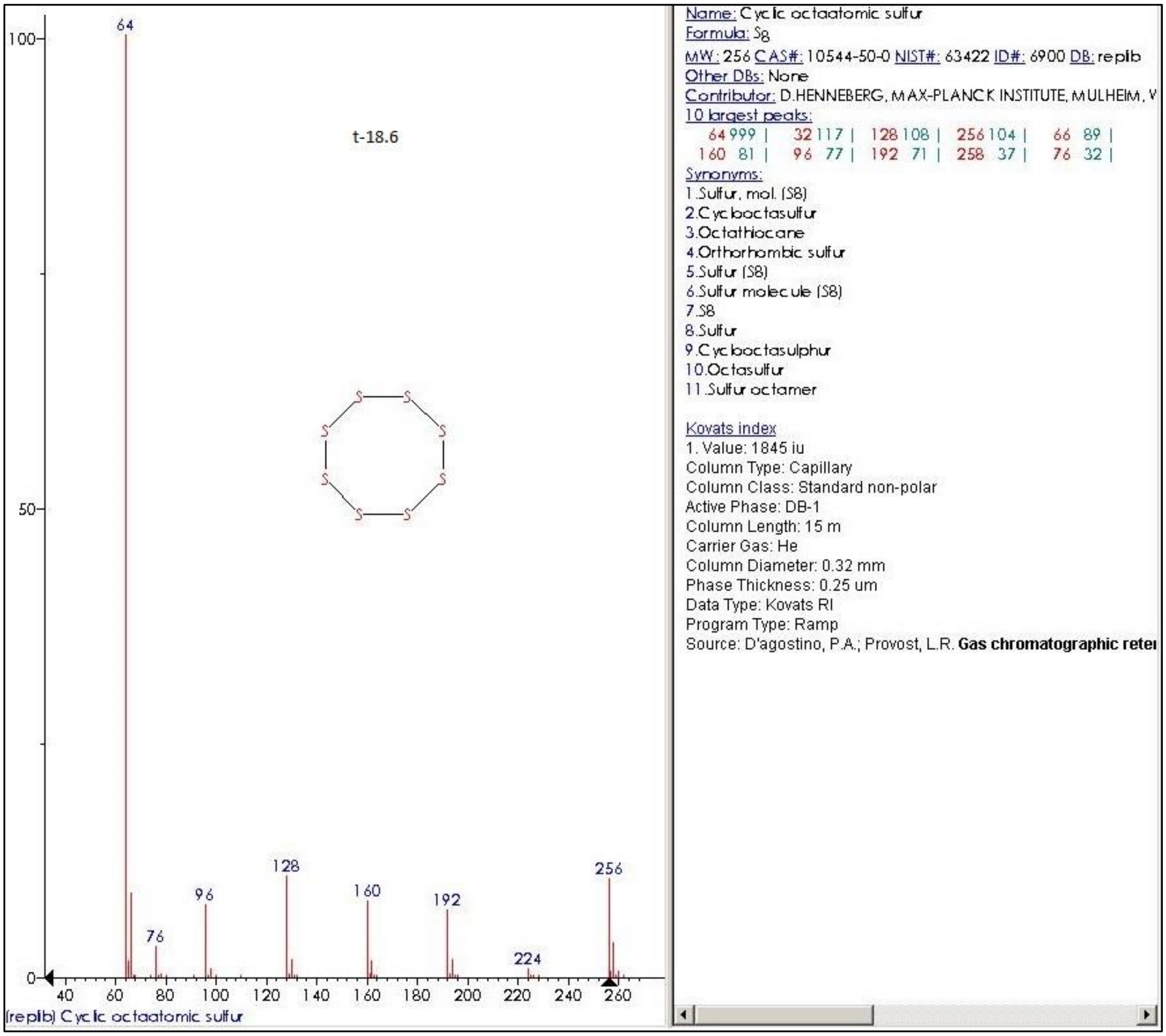
Estimated Kovats RI:

Value: 3936 iu

Confidence interval (Diverse functional groups): 89(50%) 382(95

(mainlib) 2-Isopropyl-5-methylcyclohexyl 3-(1-(4-chlorophenyl)-3-oxobutyl)-coumarin-4-yl

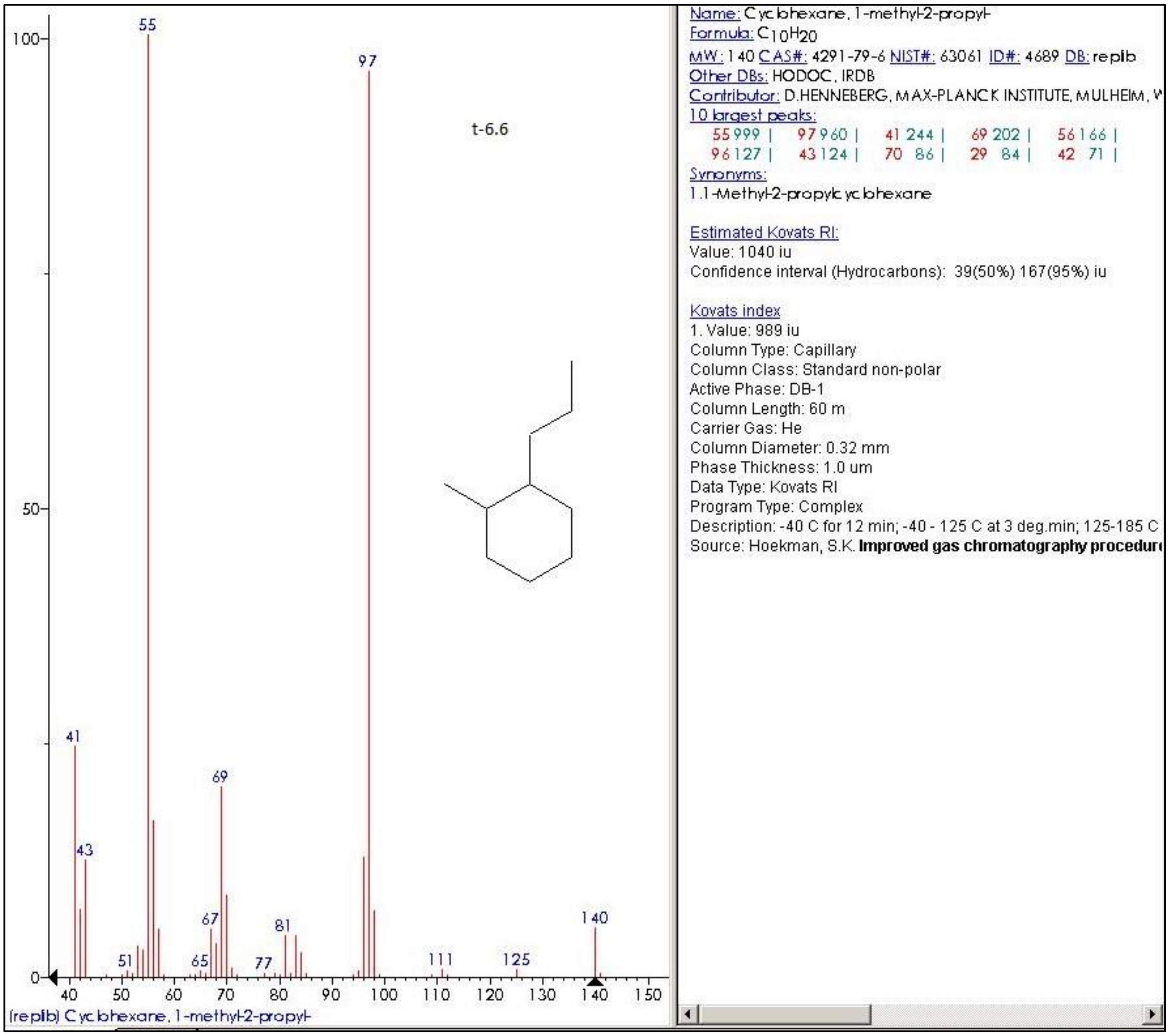
Figure C-31



Name: Cyclic octaatomic sulfur
Formula: S_8
MW: 256 CAS#: 10544-50-0 NIST#: 63422 ID#: 6900 DB: replib
Other DBs: None
Contributor: D.HENNEBERG, MAX-PLANCK INSTITUTE, MULHEIM, V
10 largest peaks:
64 999 | 32 117 | 128 108 | 256 104 | 66 89 |
160 81 | 96 77 | 192 71 | 258 37 | 76 32 |
Synonyms:
1.Sulfur, mol. (S8)
2.Cyclooctasulfur
3.Octathiocane
4.Orthorhombic sulfur
5.Sulfur (S8)
6.Sulfur molecule (S8)
7.S8
8.Sulfur
9.Cyclooctasulphur
10.Octasulfur
11.Sulfur octamer

Kovats index
1. Value: 1845 iu
Column Type: Capillary
Column Class: Standard non-polar
Active Phase: DB-1
Column Length: 15 m
Carrier Gas: He
Column Diameter: 0.32 mm
Phase Thickness: 0.25 um
Data Type: Kovats RI
Program Type: Ramp
Source: D'agostino, P.A.; Provost, L.R. **Gas chromatographic reter**

Figure C-32

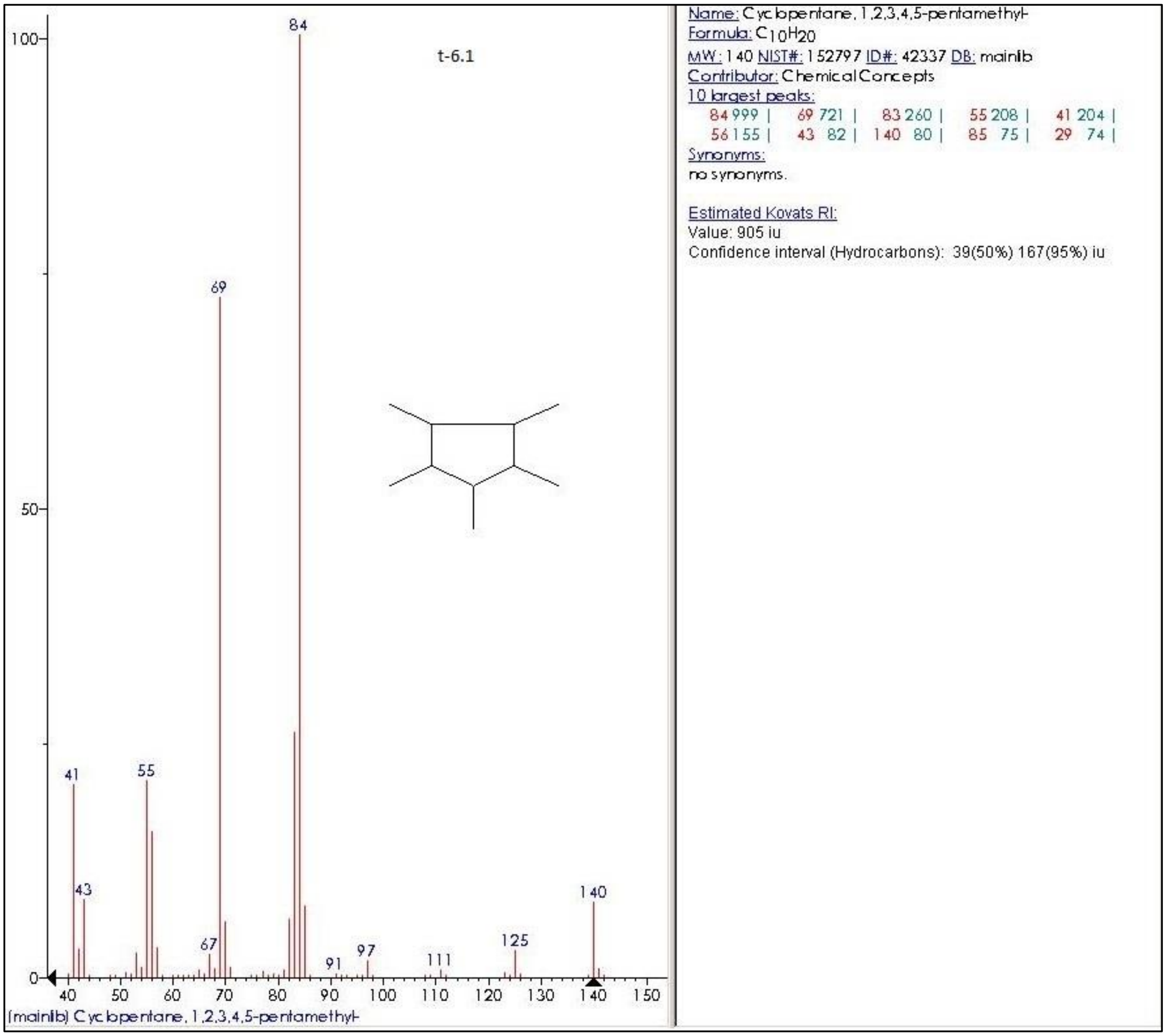


Name: Cyclohexane, 1-methyl-2-propyl-
Formula: $C_{10}H_{20}$
MW: 140 CAS#: 4291-79-6 NIST#: 63061 ID#: 4689 DB: replib
Other DBs: HODOC, IRDB
Contributor: D.HENNEBERG, MAX-PLANCK INSTITUTE, MULHEIM,
10 largest peaks:
55 999 | 97 960 | 41 244 | 69 202 | 56 166 |
96 127 | 43 124 | 70 86 | 29 84 | 42 71 |
Synonyms:
1.1-Methyl-2-propylcyclohexane

Estimated Kovats RI:
Value: 1040 iu
Confidence interval (Hydrocarbons): 39(50%) 167(95%) iu

Kovats index
1. Value: 989 iu
Column Type: Capillary
Column Class: Standard non-polar
Active Phase: DB-1
Column Length: 60 m
Carrier Gas: He
Column Diameter: 0.32 mm
Phase Thickness: 1.0 um
Data Type: Kovats RI
Program Type: Complex
Description: -40 C for 12 min; -40 - 125 C at 3 deg.min; 125-185 C
Source: Hoekman, S.K. **Improved gas chromatography procedure**

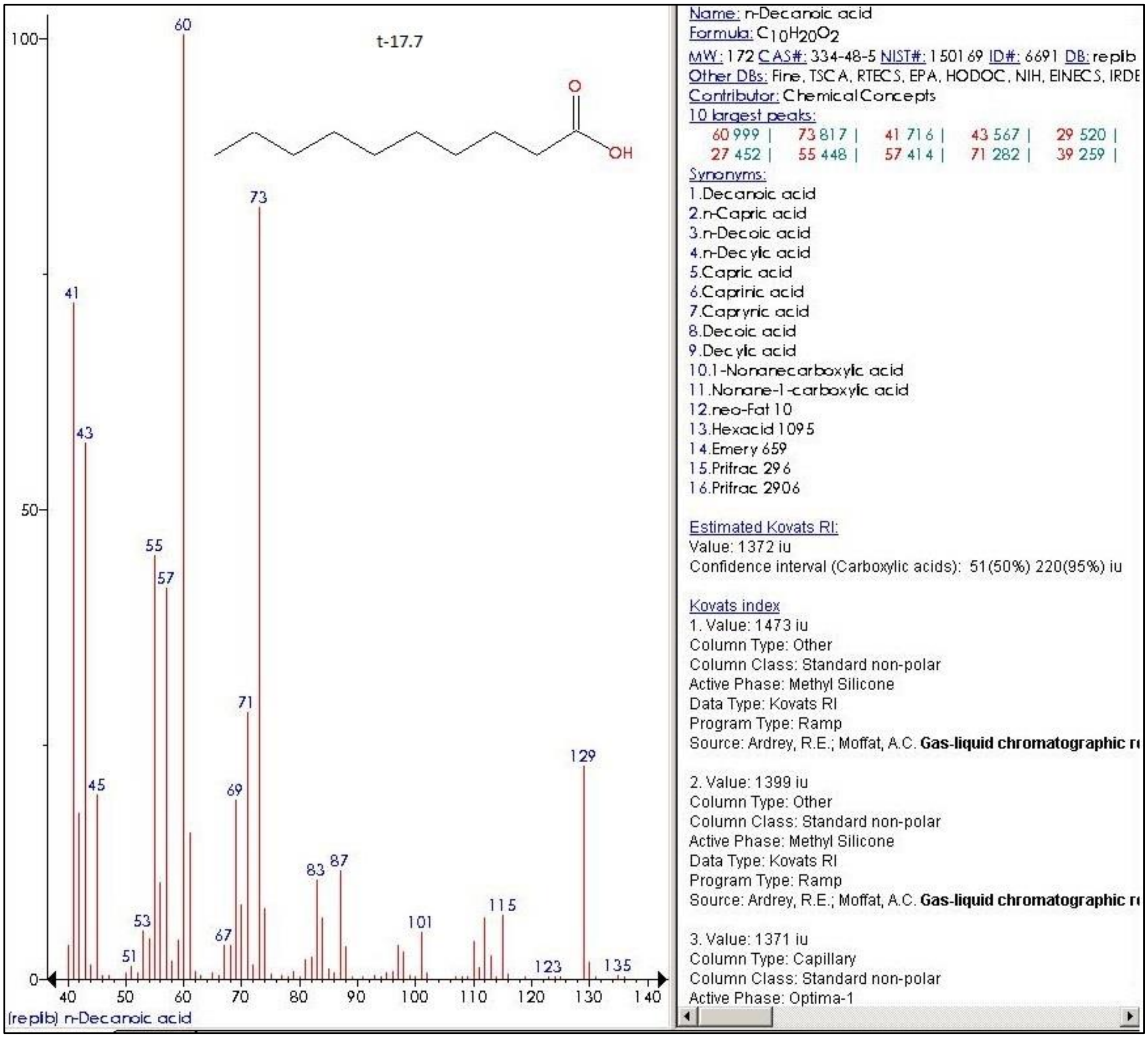
Figure C-33



Name: Cyclopentane, 1,2,3,4,5-pentamethyl-
Formula: $C_{10}H_{20}$
MW: 140 NIST#: 152797 ID#: 42337 DB: mainlib
Contributor: Chemical Concepts
10 largest peaks:
84 999 | 69 721 | 83 260 | 55 208 | 41 204 |
56 155 | 43 82 | 140 80 | 85 75 | 29 74 |
Synonyms:
no synonyms.

Estimated Kovats RI:
Value: 905 iu
Confidence interval (Hydrocarbons): 39(50%) 167(95%) iu

Figure C-34



Name: n-Decanoic acid
Formula: $C_{10}H_{20}O_2$
MW: 172 CAS#: 334-48-5 NIST#: 150169 ID#: 6691 DB: replib
Other DBs: Fine, TSCA, RTECS, EPA, HODOC, NIH, EINECS, IRDE
Contributor: Chemical Concepts
10 largest peaks:
60 999 | 73 817 | 41 716 | 43 567 | 29 520 |
27 452 | 55 448 | 57 414 | 71 282 | 39 259 |
Synonyms:
1.Decanoic acid
2.n-Capric acid
3.n-Decoic acid
4.n-Decylic acid
5.Capric acid
6.Caprinic acid
7.Caprynic acid
8.Decoic acid
9.Decylic acid
10.1-Nonanecarboxylic acid
11.Nonane-1-carboxylic acid
12.neo-Fat 10
13.Hexacid 1095
14.Emery 659
15.Prifrac 296
16.Prifrac 2906

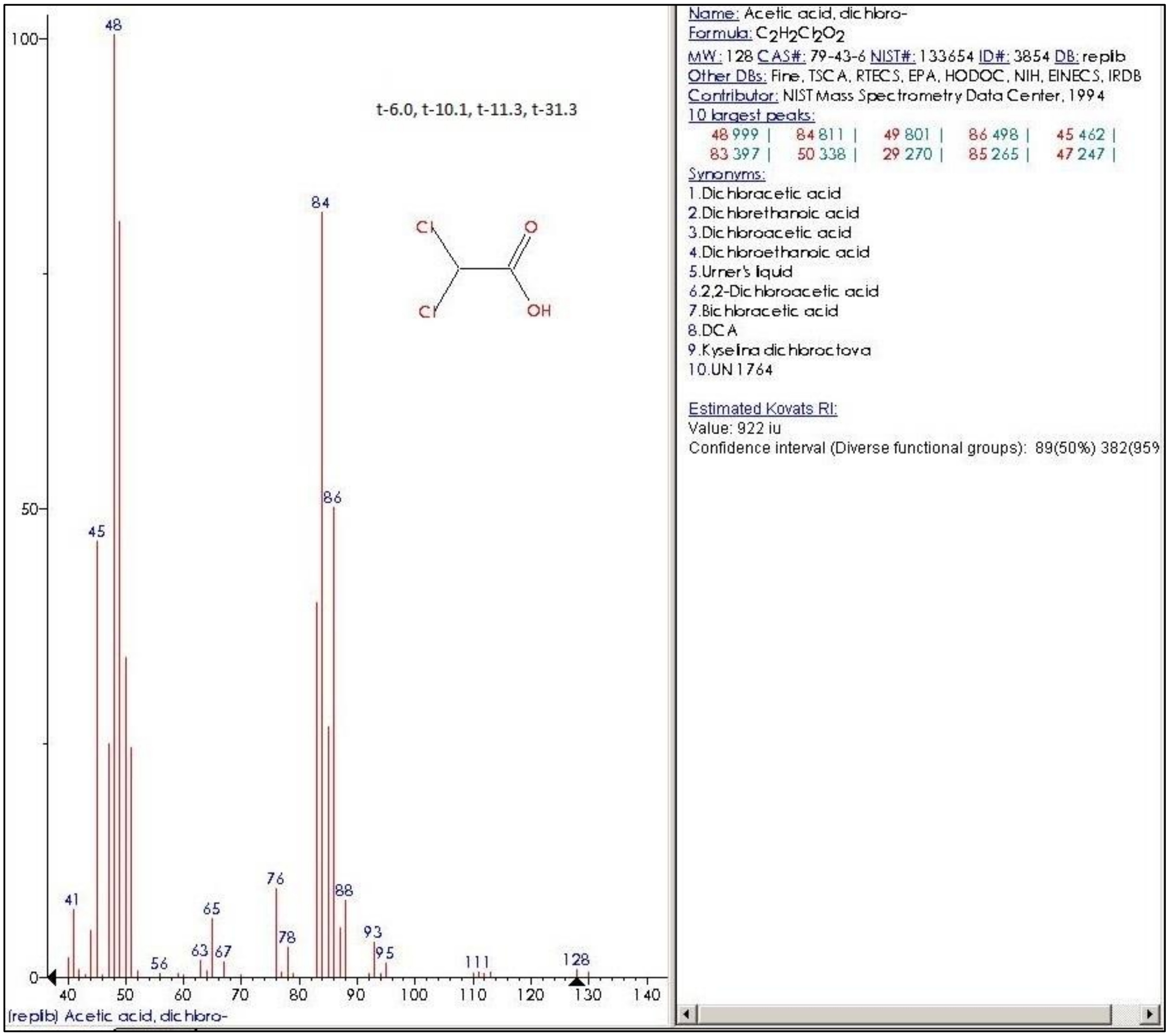
Estimated Kovats RI:
Value: 1372 iu
Confidence interval (Carboxylic acids): 51(50%) 220(95%) iu

Kovats index
1. Value: 1473 iu
Column Type: Other
Column Class: Standard non-polar
Active Phase: Methyl Silicone
Data Type: Kovats RI
Program Type: Ramp
Source: Ardrey, R.E.; Moffat, A.C. **Gas-liquid chromatographic r**

2. Value: 1399 iu
Column Type: Other
Column Class: Standard non-polar
Active Phase: Methyl Silicone
Data Type: Kovats RI
Program Type: Ramp
Source: Ardrey, R.E.; Moffat, A.C. **Gas-liquid chromatographic r**

3. Value: 1371 iu
Column Type: Capillary
Column Class: Standard non-polar
Active Phase: Optima-1

Figure C-35

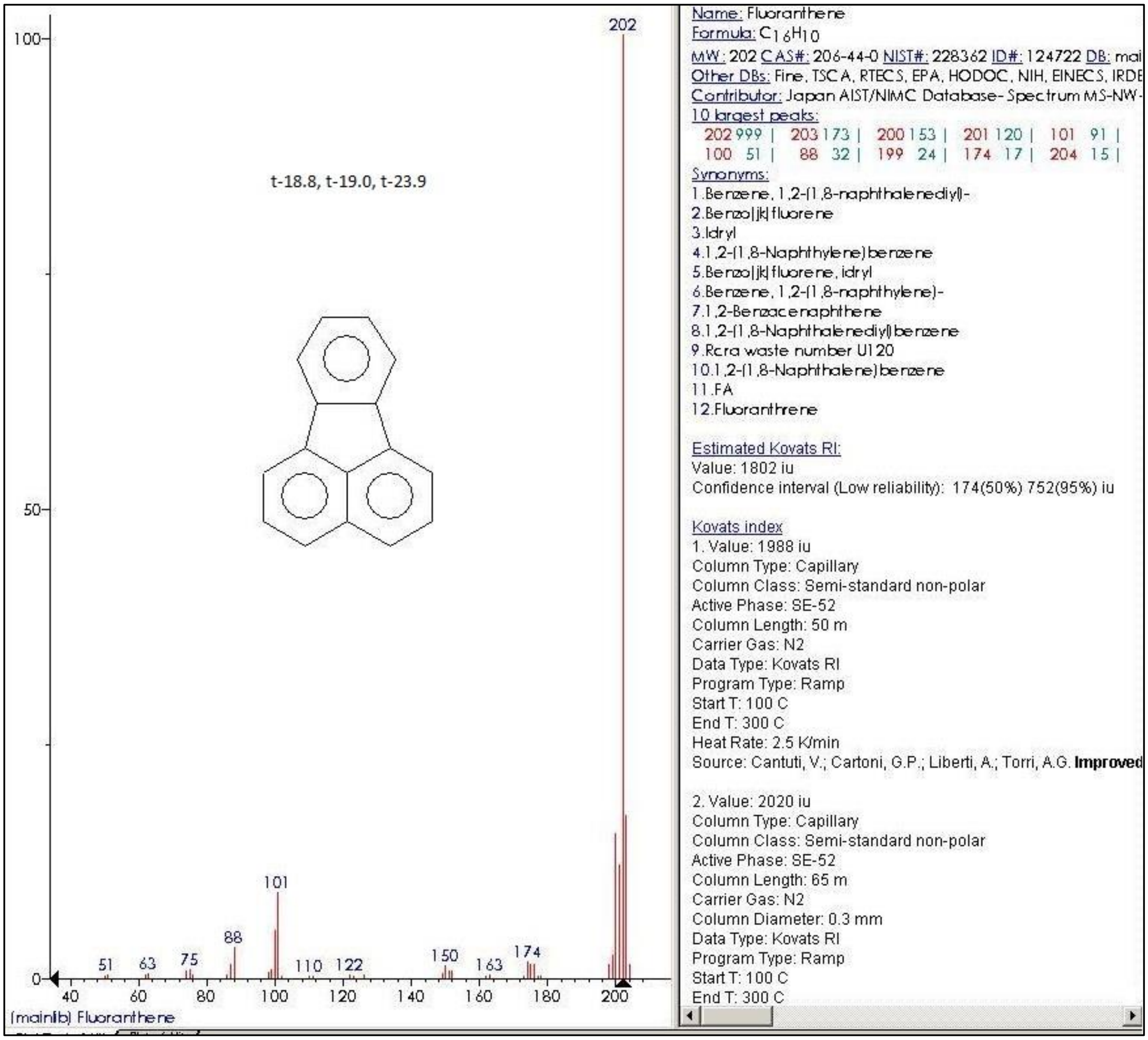


t-6.0, t-10.1, t-11.3, t-31.3

Name: Acetic acid, dichloro-
Formula: $C_2H_2Cl_2O_2$
MW: 128 CAS#: 79-43-6 NIST#: 133654 ID#: 3854 DB: replib
Other DBs: Fine, TSCA, RTECS, EPA, HODOC, NIH, EINECS, IRDB
Contributor: NIST Mass Spectrometry Data Center, 1994
10 largest peaks:
48 999 | 84 811 | 49 801 | 86 498 | 45 462 |
83 397 | 50 338 | 29 270 | 85 265 | 47 247 |
Synonyms:
1.Dichloracetic acid
2.Dichlorethanoic acid
3.Dichloroacetic acid
4.Dichloroethanoic acid
5.Urner's liquid
6.2,2-Dichloroacetic acid
7.Bichloracetic acid
8.DCA
9.Kyselina dichloroctova
10.UN 1764

Estimated Kovats RI:
Value: 922 iu
Confidence interval (Diverse functional groups): 89(50%) 382(95

Figure C-36



t-18.8, t-19.0, t-23.9

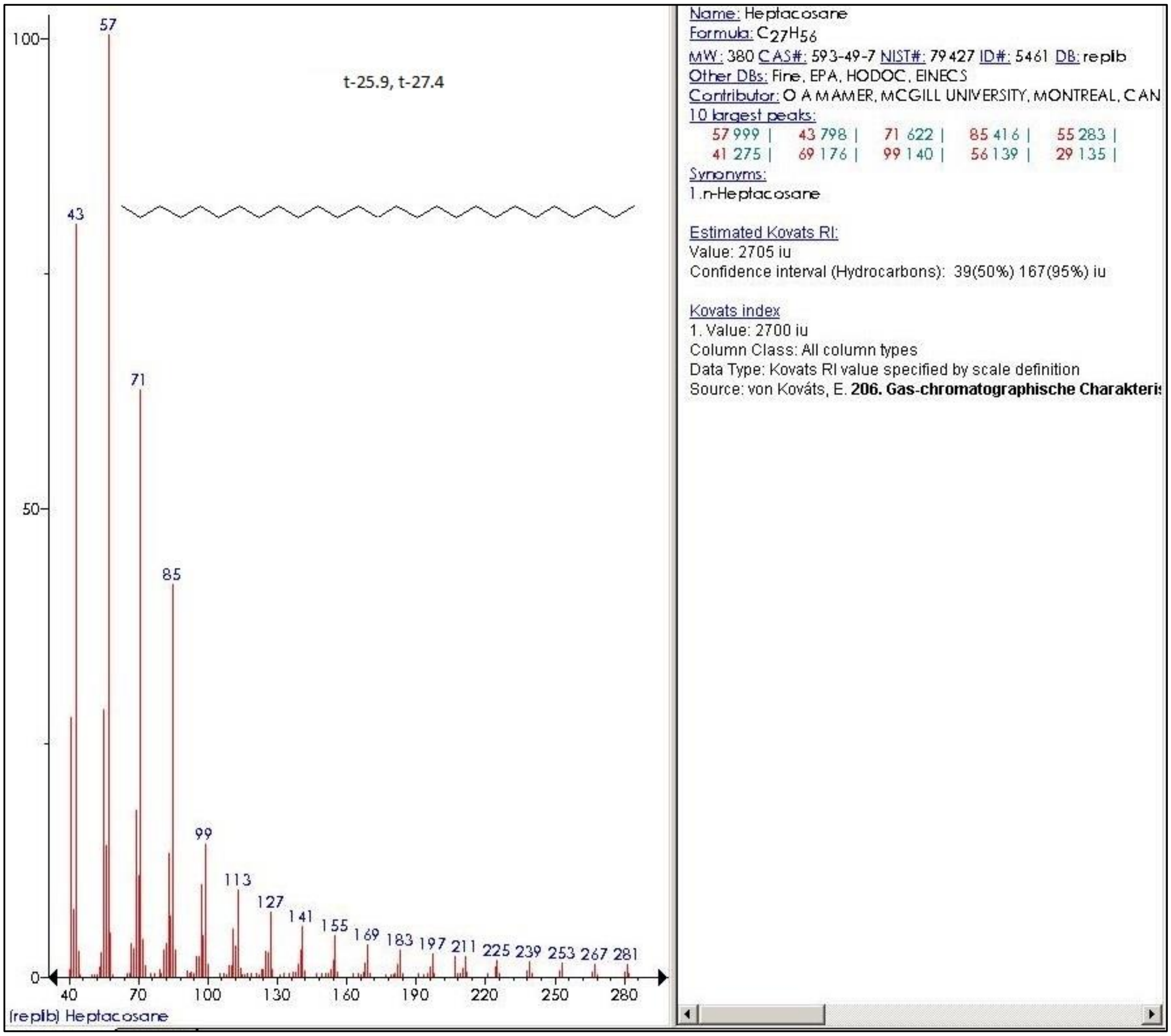
Name: Fluoranthene
Formula: $C_{16}H_{10}$
MW: 202 CAS#: 206-44-0 NIST#: 228362 ID#: 124722 DB: mai
Other DBs: Fine, TSCA, RTECS, EPA, HODOC, NIH, EINECS, IRDE
Contributor: Japan AIST/NIMC Database- Spectrum MS-NW-
10 largest peaks:
202 999 | 203 173 | 200 153 | 201 120 | 101 91 |
100 51 | 88 32 | 199 24 | 174 17 | 204 15 |
Synonyms:
1.Benzene, 1,2-(1,8-naphthalenediyl)-
2.Benzo[jk]fluorene
3.Idryl
4.1,2-(1,8-Naphthylene)benzene
5.Benzo[jk]fluorene, idryl
6.Benzene, 1,2-(1,8-naphthylene)-
7.1,2-Benzacenaphthene
8.1,2-(1,8-Naphthalenediyl)benzene
9.Rcra waste number U120
10.1,2-(1,8-Naphthalene)benzene
11.FA
12.Fluoranthrene

Estimated Kovats RI:
Value: 1802 iu
Confidence interval (Low reliability): 174(50%) 752(95%) iu

Kovats index
1. Value: 1988 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Column Length: 50 m
Carrier Gas: N2
Data Type: Kovats RI
Program Type: Ramp
Start T: 100 C
End T: 300 C
Heat Rate: 2.5 K/min
Source: Cantuti, V.; Cartoni, G.P.; Liberti, A.; Torri, A.G. **Improved**

2. Value: 2020 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Column Length: 65 m
Carrier Gas: N2
Column Diameter: 0.3 mm
Data Type: Kovats RI
Program Type: Ramp
Start T: 100 C
End T: 300 C

Figure C-37

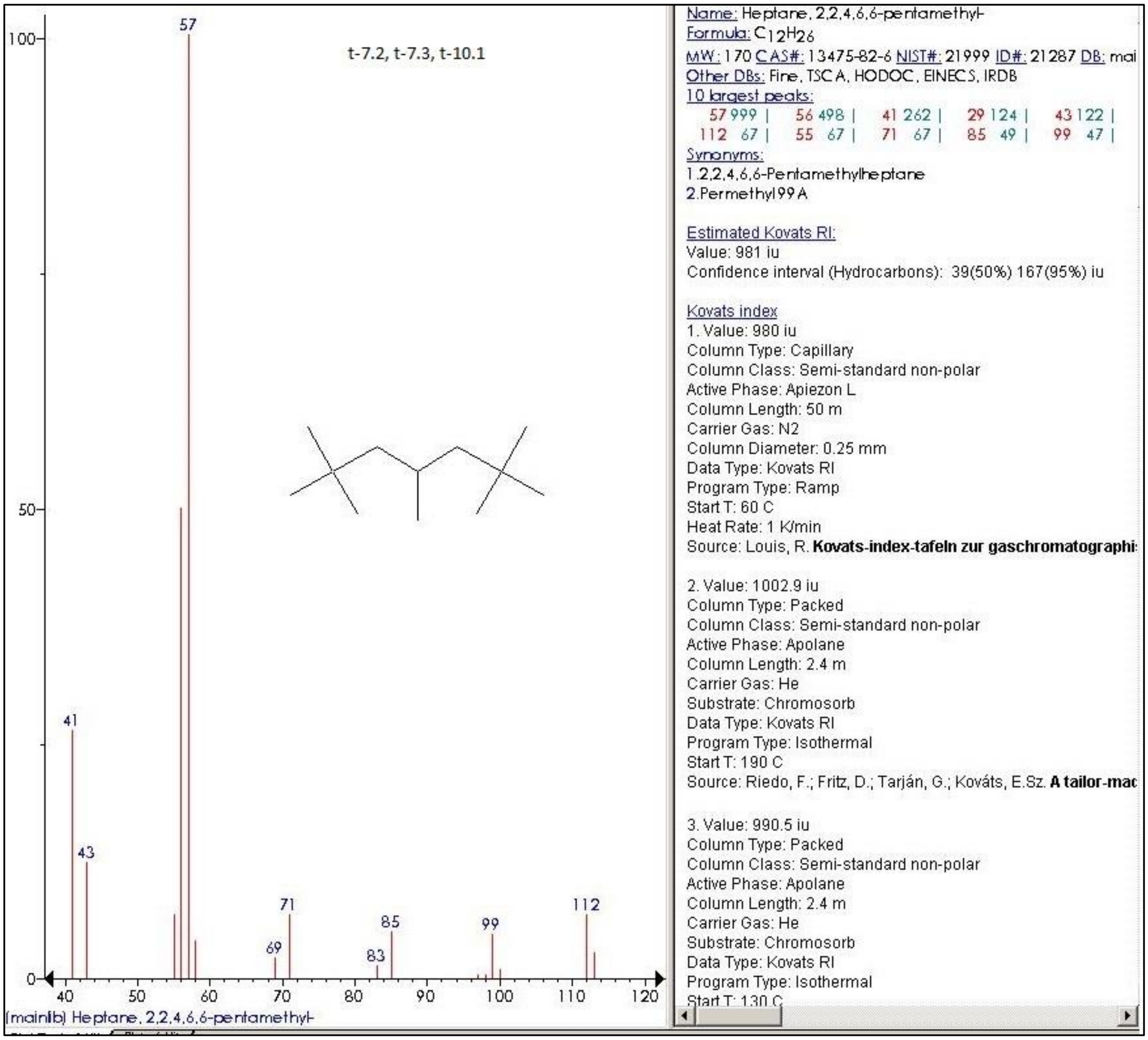


Name: Heptacosane
Formula: $C_{27}H_{56}$
MW: 380 CAS#: 593-49-7 NIST#: 79427 ID#: 5461 DB: replib
Other DBs: Fine, EPA, HODOC, EINECS
Contributor: O A MAMER, MCGILL UNIVERSITY, MONTREAL, CAN
10 largest peaks:
57 999 | 43 798 | 71 622 | 85 416 | 55 283 |
41 275 | 69 176 | 99 140 | 56 139 | 29 135 |
Synonyms:
1.n-Heptacosane

Estimated Kovats RI:
Value: 2705 iu
Confidence interval (Hydrocarbons): 39(50%) 167(95%) iu

Kovats index
1. Value: 2700 iu
Column Class: All column types
Data Type: Kovats RI value specified by scale definition
Source: von Kováts, E. **206. Gas-chromatographische Charakteris**

Figure C-38



t-7.2, t-7.3, t-10.1

(mainlib) Heptane, 2,2,4,6,6-pentamethyl-

Name: Heptane, 2,2,4,6,6-pentamethyl-
Formula: $C_{12}H_{26}$
MW: 170 CAS#: 13475-82-6 NIST#: 21999 ID#: 21287 DB: mai
Other DBs: Fine, TSCA, HODOC, EINECS, IRDB
10 largest peaks:
57 999 | 56 498 | 41 262 | 29 124 | 43 122 |
112 67 | 55 67 | 71 67 | 85 49 | 99 47 |
Synonyms:
1.2,2,4,6,6-Pentamethylheptane
2.Permethyl99A

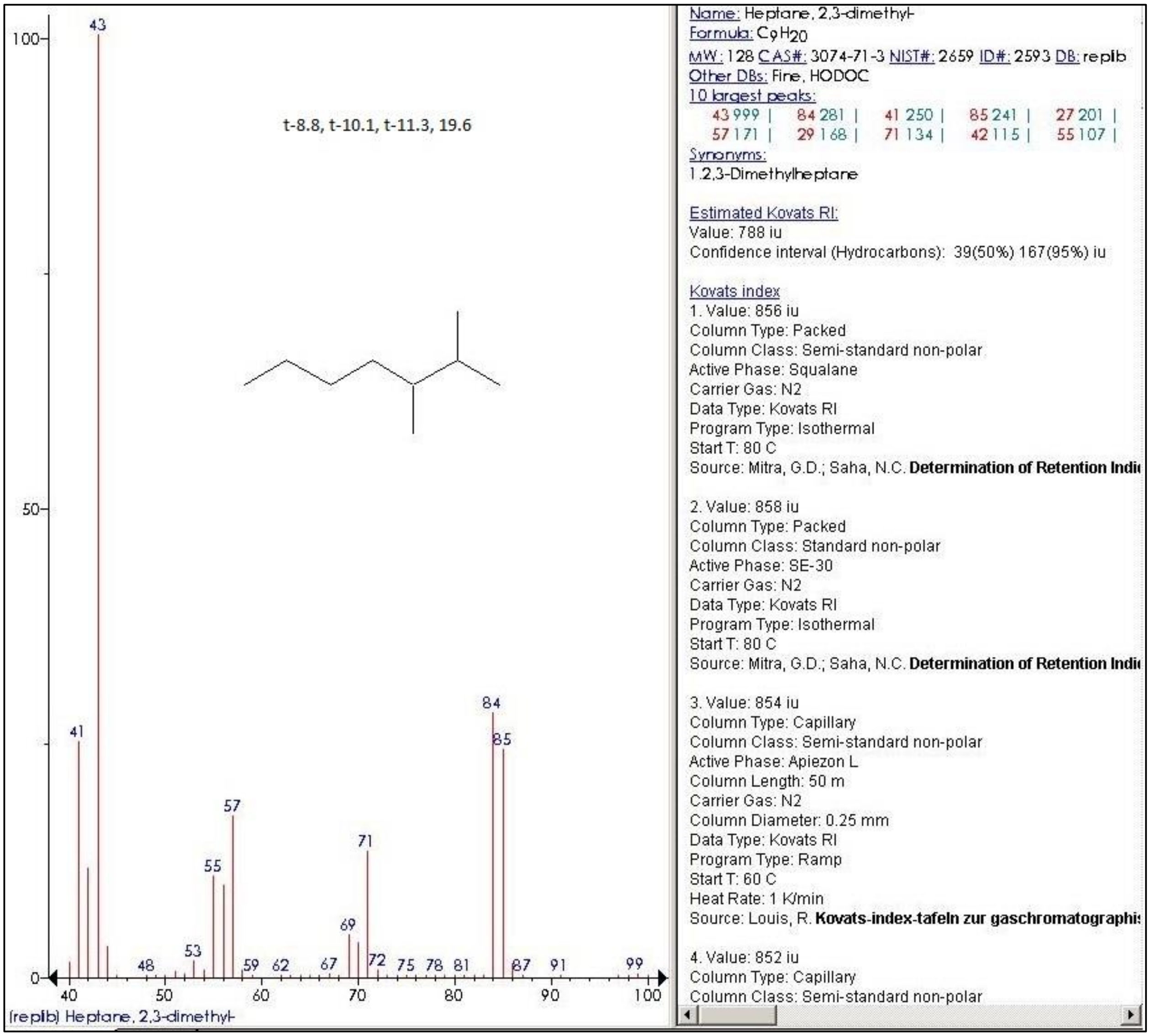
Estimated Kovats RI:
Value: 981 iu
Confidence interval (Hydrocarbons): 39(50%) 167(95%) iu

Kovats index
1. Value: 980 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: Apiezon L
Column Length: 50 m
Carrier Gas: N2
Column Diameter: 0.25 mm
Data Type: Kovats RI
Program Type: Ramp
Start T: 60 C
Heat Rate: 1 K/min
Source: Louis, R. **Kovats-index-tafeln zur gaschromatographi**

2. Value: 1002.9 iu
Column Type: Packed
Column Class: Semi-standard non-polar
Active Phase: Apolane
Column Length: 2.4 m
Carrier Gas: He
Substrate: Chromosorb
Data Type: Kovats RI
Program Type: Isothermal
Start T: 190 C
Source: Riedo, F.; Fritz, D.; Tarján, G.; Kováts, E.Sz. **A tailor-mac**

3. Value: 990.5 iu
Column Type: Packed
Column Class: Semi-standard non-polar
Active Phase: Apolane
Column Length: 2.4 m
Carrier Gas: He
Substrate: Chromosorb
Data Type: Kovats RI
Program Type: Isothermal
Start T: 130 C

Figure C-39



t-8.8, t-10.1, t-11.3, 19.6

Name: Heptane, 2,3-dimethyl-
Formula: C_9H_{20}
MW: 128 CAS#: 3074-71-3 NIST#: 2659 ID#: 2593 DB: replib
Other DBs: Fine, HODOC
10 largest peaks:
43 999 | 84 281 | 41 250 | 85 241 | 27 201 |
57 171 | 29 168 | 71 134 | 42 115 | 55 107 |
Synonyms:
1.2,3-Dimethylheptane

Estimated Kovats RI:
Value: 788 iu
Confidence interval (Hydrocarbons): 39(50%) 167(95%) iu

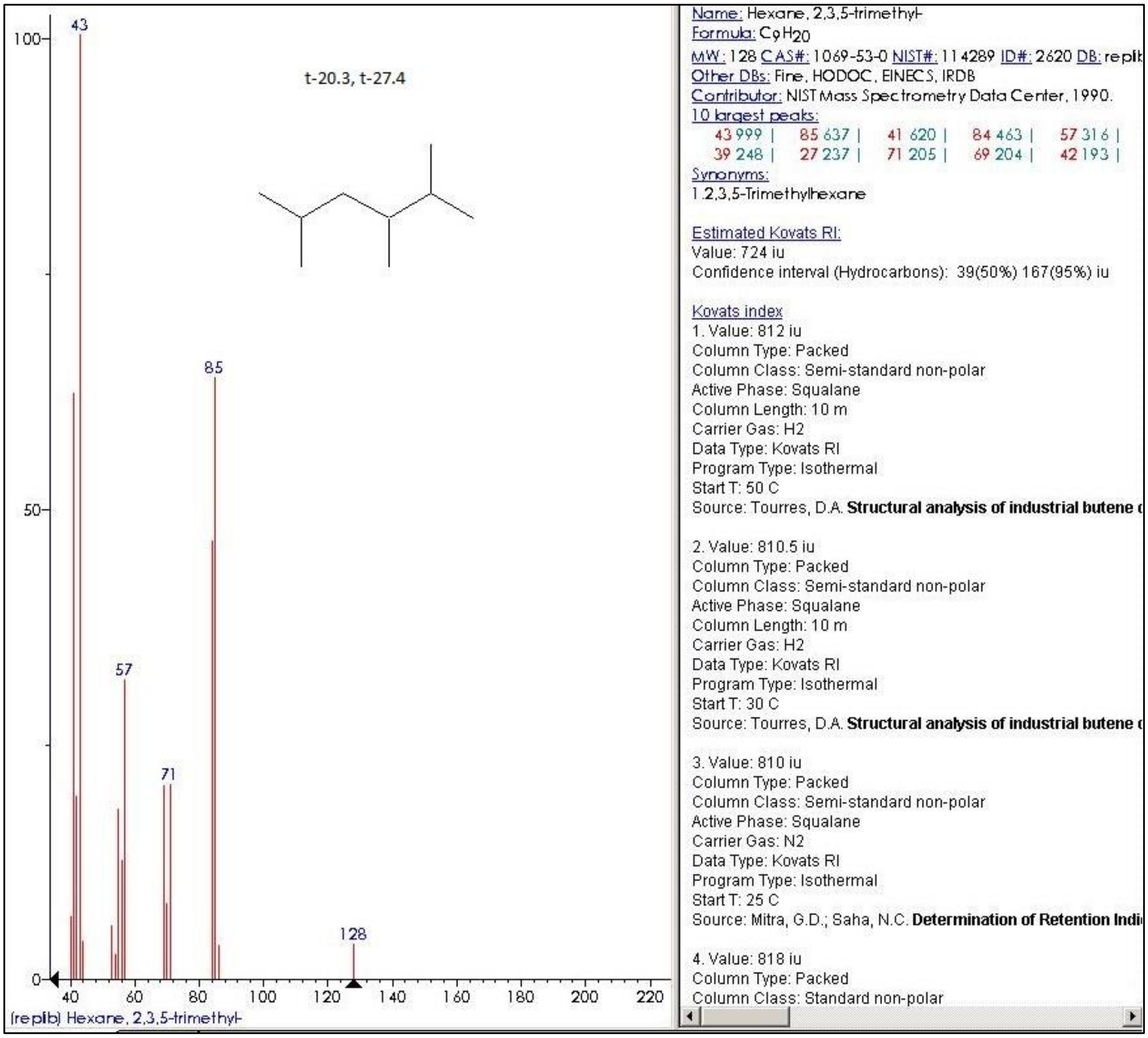
Kovats index
1. Value: 856 iu
Column Type: Packed
Column Class: Semi-standard non-polar
Active Phase: Squalane
Carrier Gas: N2
Data Type: Kovats RI
Program Type: Isothermal
Start T: 80 C
Source: Mitra, G.D.; Saha, N.C. **Determination of Retention Indi**

2. Value: 858 iu
Column Type: Packed
Column Class: Standard non-polar
Active Phase: SE-30
Carrier Gas: N2
Data Type: Kovats RI
Program Type: Isothermal
Start T: 80 C
Source: Mitra, G.D.; Saha, N.C. **Determination of Retention Indi**

3. Value: 854 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: Apiezon L
Column Length: 50 m
Carrier Gas: N2
Column Diameter: 0.25 mm
Data Type: Kovats RI
Program Type: Ramp
Start T: 60 C
Heat Rate: 1 K/min
Source: Louis, R. **Kovats-index-tafeln zur gaschromatographis**

4. Value: 852 iu
Column Type: Capillary
Column Class: Semi-standard non-polar

Figure C-40]



Name: Hexane, 2,3,5-trimethyl-
Formula: C_9H_{20}
MW: 128 CAS#: 1069-53-0 NIST#: 114289 ID#: 2620 DB: repli
Other DBs: Fine, HODOC, EINECS, IRDB
Contributor: NIST Mass Spectrometry Data Center, 1990.
10 largest peaks:
43 999 | 85 637 | 41 620 | 84 463 | 57 316 |
39 248 | 27 237 | 71 205 | 69 204 | 42 193 |
Synonyms:
1.2,3,5-Trimethylhexane

Estimated Kovats RI:
Value: 724 iu
Confidence interval (Hydrocarbons): 39(50%) 167(95%) iu

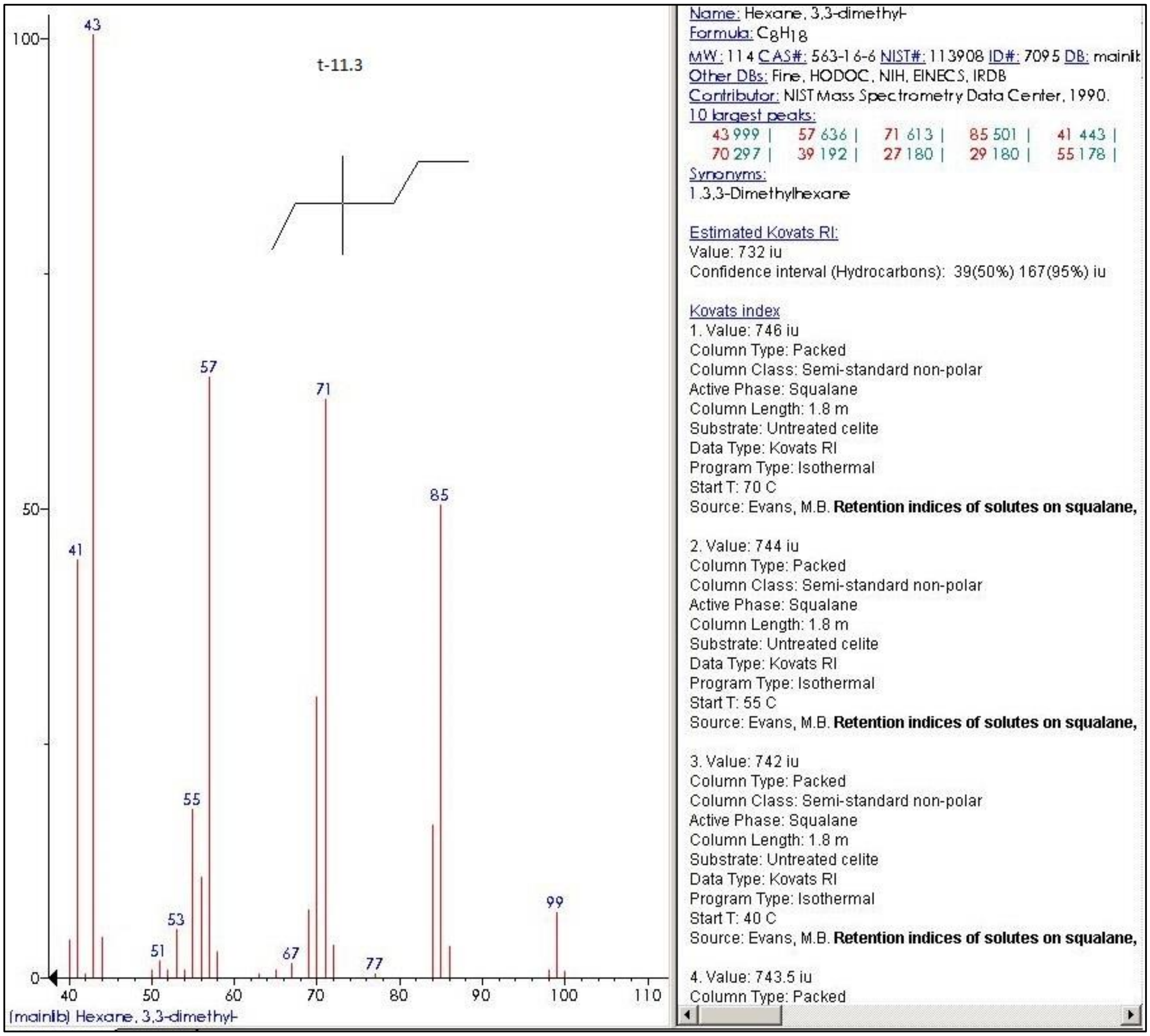
Kovats index
1. Value: 812 iu
Column Type: Packed
Column Class: Semi-standard non-polar
Active Phase: Squalane
Column Length: 10 m
Carrier Gas: H2
Data Type: Kovats RI
Program Type: Isothermal
Start T: 50 C
Source: Tourres, D.A. **Structural analysis of industrial butene**

2. Value: 810.5 iu
Column Type: Packed
Column Class: Semi-standard non-polar
Active Phase: Squalane
Column Length: 10 m
Carrier Gas: H2
Data Type: Kovats RI
Program Type: Isothermal
Start T: 30 C
Source: Tourres, D.A. **Structural analysis of industrial butene**

3. Value: 810 iu
Column Type: Packed
Column Class: Semi-standard non-polar
Active Phase: Squalane
Carrier Gas: N2
Data Type: Kovats RI
Program Type: Isothermal
Start T: 25 C
Source: Mitra, G.D.; Saha, N.C. **Determination of Retention Indi**

4. Value: 818 iu
Column Type: Packed
Column Class: Standard non-polar

Figure C-41



Name: Hexane, 3,3-dimethyl-
Formula: C_8H_{18}
MW: 114 CAS#: 563-16-6 NIST#: 113908 ID#: 7095 DB: mainlib
Other DBs: Fine, HODOC, NIH, EINECS, IRDB
Contributor: NIST Mass Spectrometry Data Center, 1990.
10 largest peaks:
43 999 | 57 636 | 71 613 | 85 501 | 41 443 |
70 297 | 39 192 | 27 180 | 29 180 | 55 178 |
Synonyms:
1.3,3-Dimethylhexane

Estimated Kovats RI:
Value: 732 iu
Confidence interval (Hydrocarbons): 39(50%) 167(95%) iu

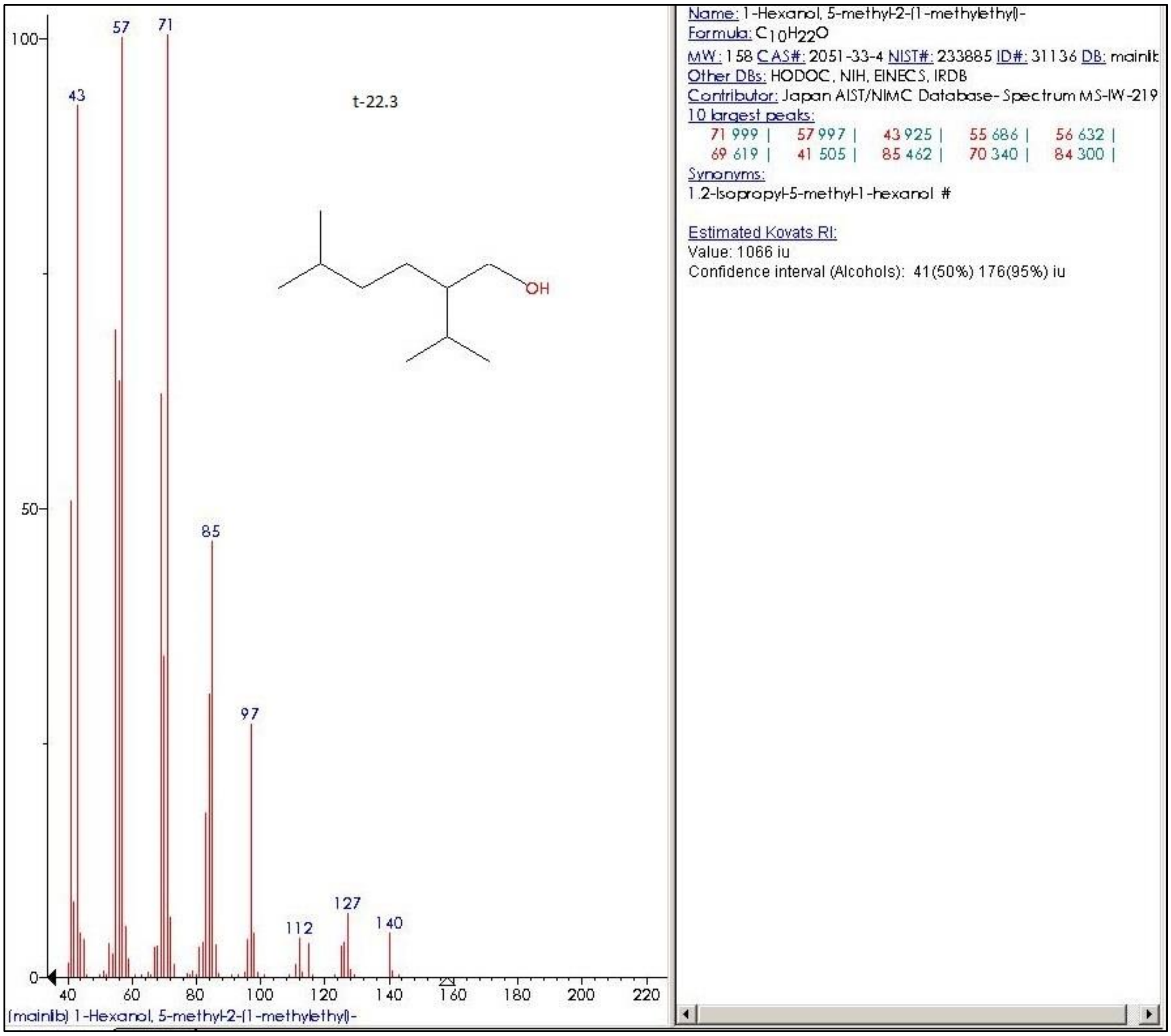
Kovats index
1. Value: 746 iu
Column Type: Packed
Column Class: Semi-standard non-polar
Active Phase: Squalane
Column Length: 1.8 m
Substrate: Untreated celite
Data Type: Kovats RI
Program Type: Isothermal
Start T: 70 C
Source: Evans, M.B. **Retention indices of solutes on squalane,**

2. Value: 744 iu
Column Type: Packed
Column Class: Semi-standard non-polar
Active Phase: Squalane
Column Length: 1.8 m
Substrate: Untreated celite
Data Type: Kovats RI
Program Type: Isothermal
Start T: 55 C
Source: Evans, M.B. **Retention indices of solutes on squalane,**

3. Value: 742 iu
Column Type: Packed
Column Class: Semi-standard non-polar
Active Phase: Squalane
Column Length: 1.8 m
Substrate: Untreated celite
Data Type: Kovats RI
Program Type: Isothermal
Start T: 40 C
Source: Evans, M.B. **Retention indices of solutes on squalane,**

4. Value: 743.5 iu
Column Type: Packed

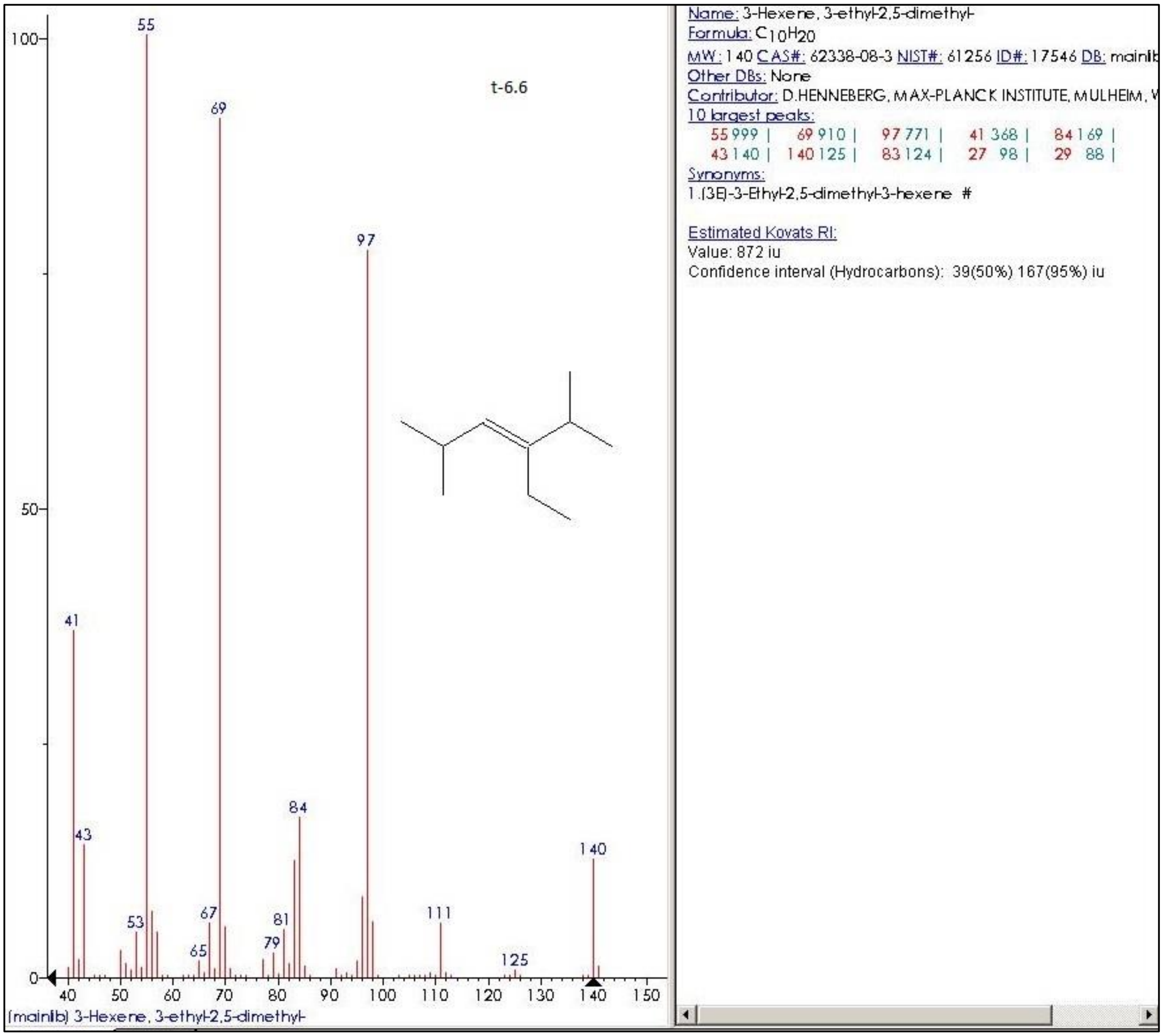
Figure C-42



Name: 1-Hexanol, 5-methyl-2-(1-methylethyl)-
Formula: $C_{10}H_{22}O$
MW: 158 CAS#: 2051-33-4 NIST#: 233885 ID#: 31136 DB: mainlib
Other DBs: HODOC, NIH, EINECS, IRDB
Contributor: Japan AIST/NIMC Database- Spectrum MS-IW-219
10 largest peaks:
71 999 | 57 997 | 43 925 | 55 686 | 56 632 |
69 619 | 41 505 | 85 462 | 70 340 | 84 300 |
Synonyms:
1.2-Isopropyl-5-methyl-1-hexanol #

Estimated Kovats RI:
Value: 1066 iu
Confidence interval (Alcohols): 41(50%) 176(95%) iu

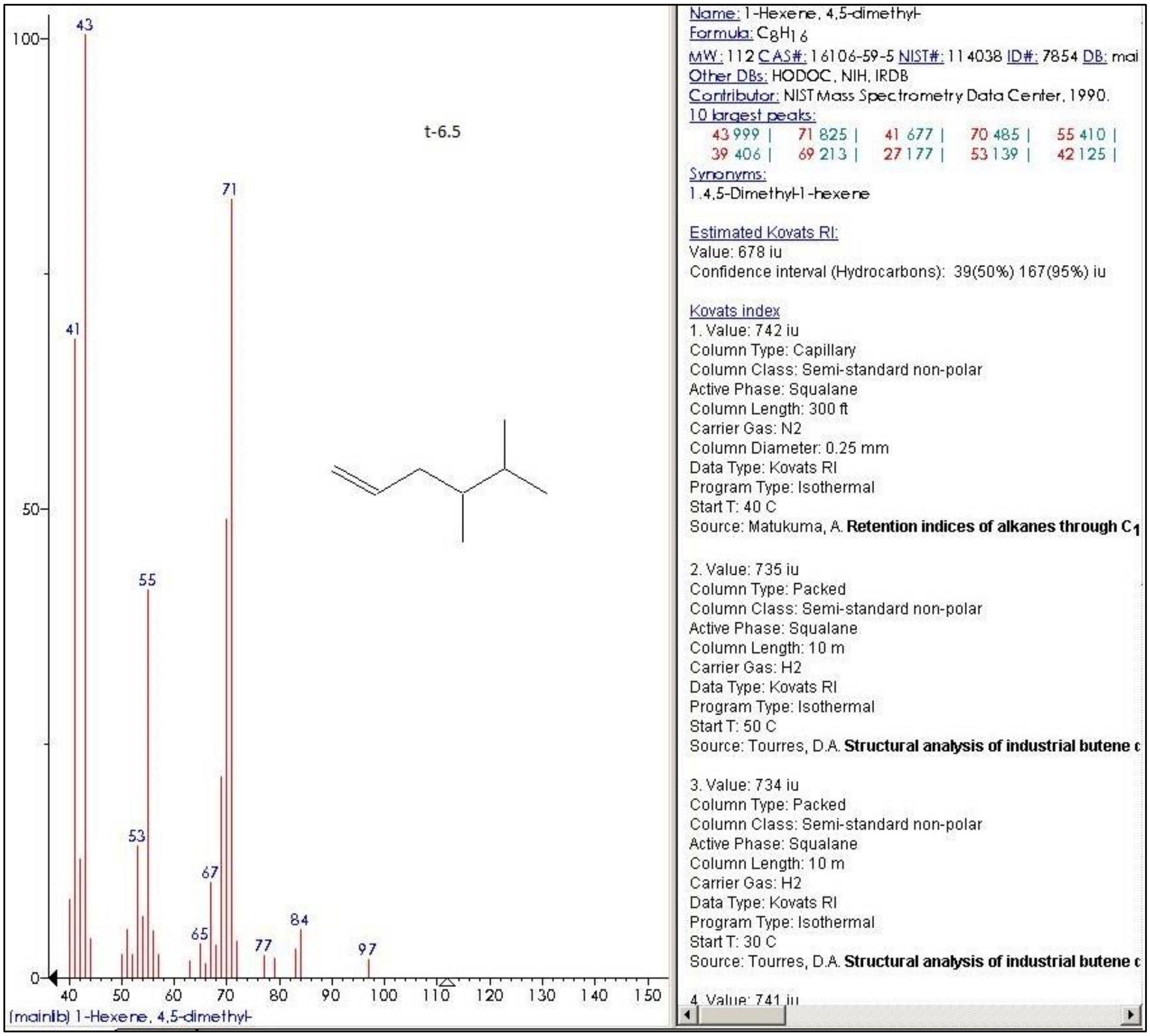
Figure C-43



Name: 3-Hexene, 3-ethyl-2,5-dimethyl-
Formula: $C_{10}H_{20}$
MW: 140 CAS#: 62338-08-3 NIST#: 61256 ID#: 17546 DB: mainli
Other DBs: None
Contributor: D.HENNEBERG, MAX-PLANCK INSTITUTE, MULHEIM,
10 largest peaks:
55 999 | 69 910 | 97 771 | 41 368 | 84 169 |
43 140 | 140 125 | 83 124 | 27 98 | 29 88 |
Synonyms:
1.(3E)-3-Ethyl-2,5-dimethyl-3-hexene #

Estimated Kovats RI:
Value: 872 iu
Confidence interval (Hydrocarbons): 39(50%) 167(95%) iu

Figure C-44



Name: 1-Hexene, 4,5-dimethyl-
Formula: C_8H_{16}
MW: 112 CAS#: 16106-59-5 NIST#: 114038 ID#: 7854 DB: mai
Other DBs: HODOC, NIH, IRDB
Contributor: NIST Mass Spectrometry Data Center, 1990.
10 largest peaks:
43 999 | 71 825 | 41 677 | 70 485 | 55 410 |
39 406 | 69 213 | 27 177 | 53 139 | 42 125 |
Synonyms:
1.4,5-Dimethyl-1-hexene

Estimated Kovats RI:
Value: 678 iu
Confidence interval (Hydrocarbons): 39(50%) 167(95%) iu

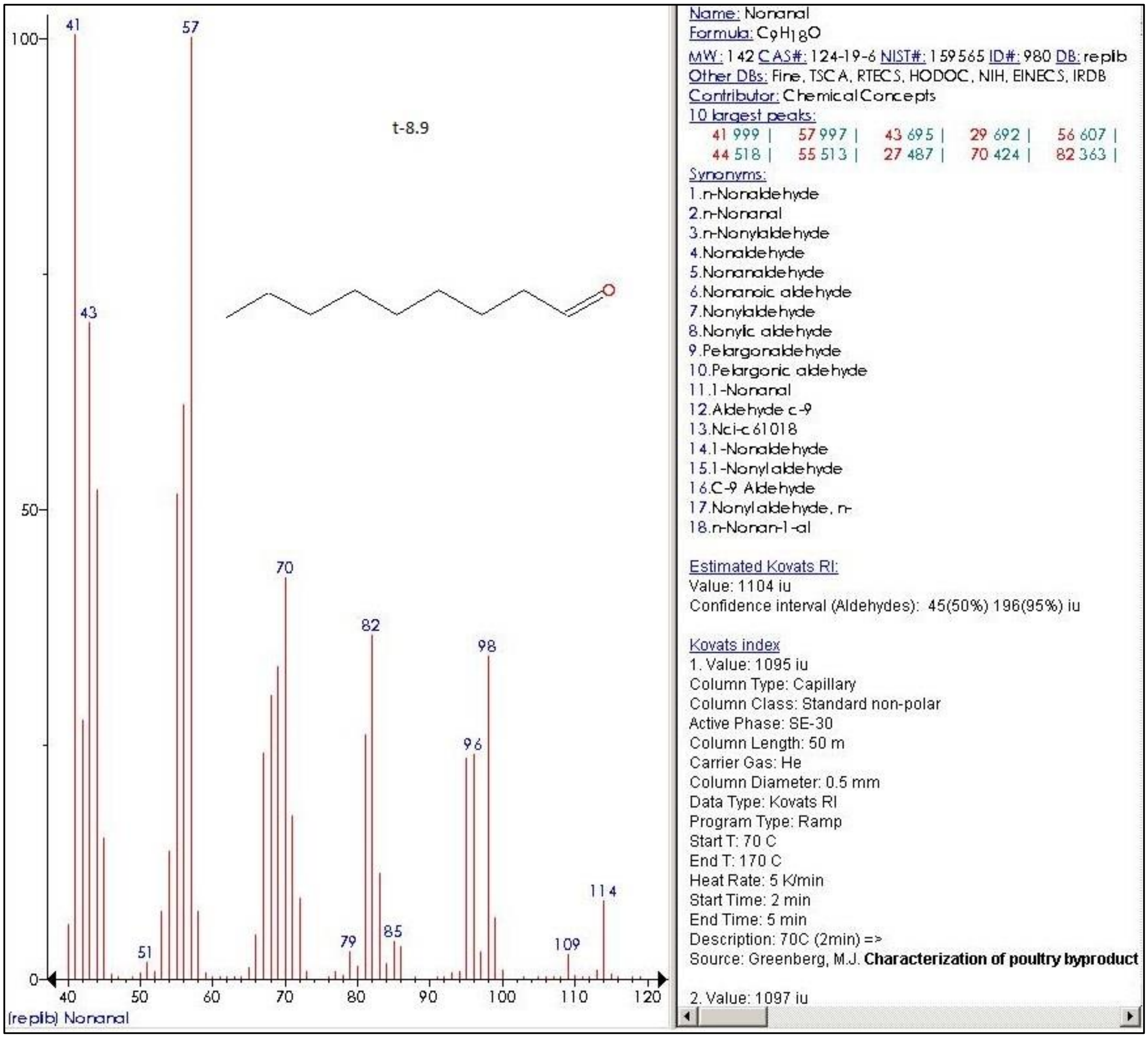
Kovats index
1. Value: 742 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: Squalane
Column Length: 300 ft
Carrier Gas: N2
Column Diameter: 0.25 mm
Data Type: Kovats RI
Program Type: Isothermal
Start T: 40 C
Source: Matukuma, A. **Retention indices of alkanes through C1**

2. Value: 735 iu
Column Type: Packed
Column Class: Semi-standard non-polar
Active Phase: Squalane
Column Length: 10 m
Carrier Gas: H2
Data Type: Kovats RI
Program Type: Isothermal
Start T: 50 C
Source: Tourres, D.A. **Structural analysis of industrial butene c**

3. Value: 734 iu
Column Type: Packed
Column Class: Semi-standard non-polar
Active Phase: Squalane
Column Length: 10 m
Carrier Gas: H2
Data Type: Kovats RI
Program Type: Isothermal
Start T: 30 C
Source: Tourres, D.A. **Structural analysis of industrial butene c**

4. Value: 741 iu

Figure C-45



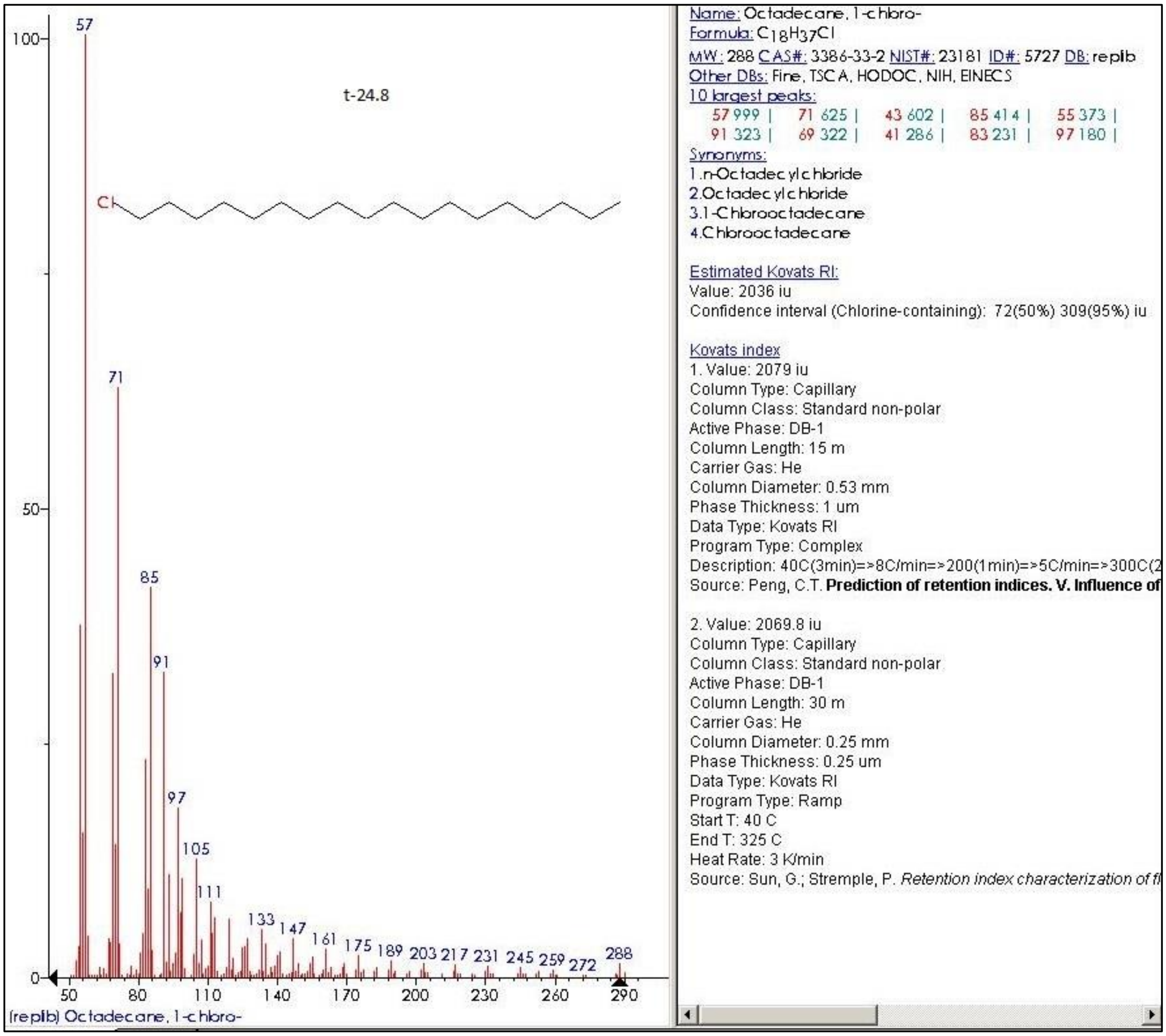
Name: Nonanal
Formula: $C_9H_{18}O$
MW: 142 CAS#: 124-19-6 NIST#: 159565 ID#: 980 DB: replib
Other DBs: Fine, TSCA, RTECS, HODOC, NIH, EINECS, IRDB
Contributor: Chemical Concepts
10 largest peaks:
41 999 | 57 997 | 43 695 | 29 692 | 56 607 |
44 518 | 55 513 | 27 487 | 70 424 | 82 363 |
Synonyms:
1.n-Nonaldehyde
2.n-Nonanal
3.n-Nonylaldehyde
4.Nonaldehyde
5.Nonanaldehyde
6.Nonanoic aldehyde
7.Nonylaldehyde
8.Nonylic aldehyde
9.Pelargonaldehyde
10.Pelargonic aldehyde
11.1-Nonanal
12.Aldehyde c-9
13.Nci-c61018
14.1-Nonaldehyde
15.1-Nonyl aldehyde
16.C-9 Aldehyde
17.Nonyl aldehyde, n-
18.n-Nonan-1-al

Estimated Kovats RI:
Value: 1104 iu
Confidence interval (Aldehydes): 45(50%) 196(95%) iu

Kovats index
1. Value: 1095 iu
Column Type: Capillary
Column Class: Standard non-polar
Active Phase: SE-30
Column Length: 50 m
Carrier Gas: He
Column Diameter: 0.5 mm
Data Type: Kovats RI
Program Type: Ramp
Start T: 70 C
End T: 170 C
Heat Rate: 5 K/min
Start Time: 2 min
End Time: 5 min
Description: 70C (2min) =>
Source: Greenberg, M.J. **Characterization of poultry byproduct**

2. Value: 1097 iu

Figure C-46



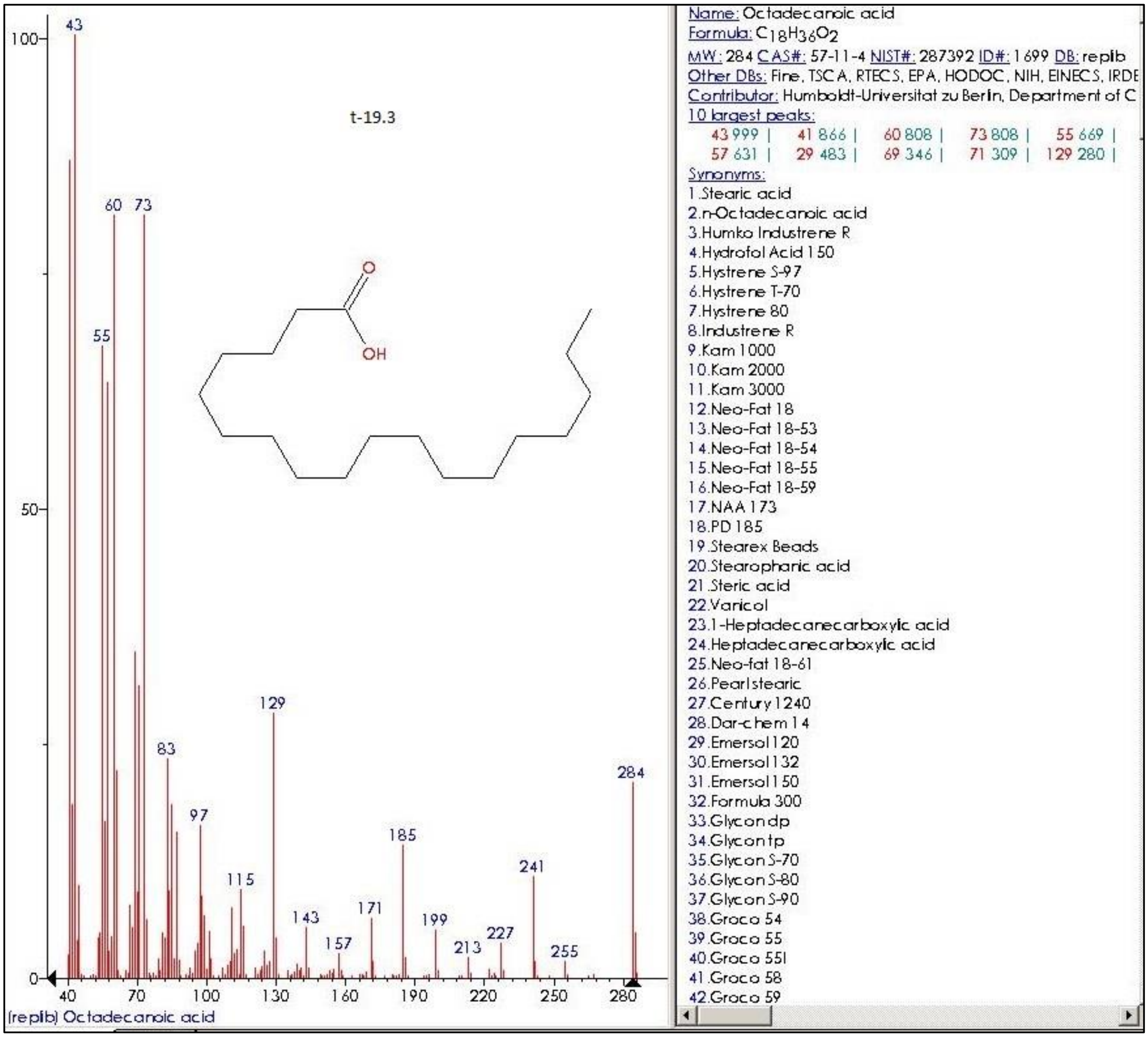
Name: Octadecane, 1-chloro-
Formula: $C_{18}H_{37}Cl$
MW: 288 CAS#: 3386-33-2 NIST#: 23181 ID#: 5727 DB: replib
Other DBs: Fine, TSCA, HODOC, NIH, EINECS
10 largest peaks:
57 999 | 71 625 | 43 602 | 85 414 | 55 373 |
91 323 | 69 322 | 41 286 | 83 231 | 97 180 |
Synonyms:
1.n-Octadecylchloride
2.Octadecylchloride
3.1-Chlorooctadecane
4.Chlorooctadecane

Estimated Kovats RI:
Value: 2036 iu
Confidence interval (Chlorine-containing): 72(50%) 309(95%) iu

Kovats index
1. Value: 2079 iu
Column Type: Capillary
Column Class: Standard non-polar
Active Phase: DB-1
Column Length: 15 m
Carrier Gas: He
Column Diameter: 0.53 mm
Phase Thickness: 1 um
Data Type: Kovats RI
Program Type: Complex
Description: 40C(3min)=>8C/min=>200(1min)=>5C/min=>300C(2
Source: Peng, C.T. **Prediction of retention indices. V. Influence of**

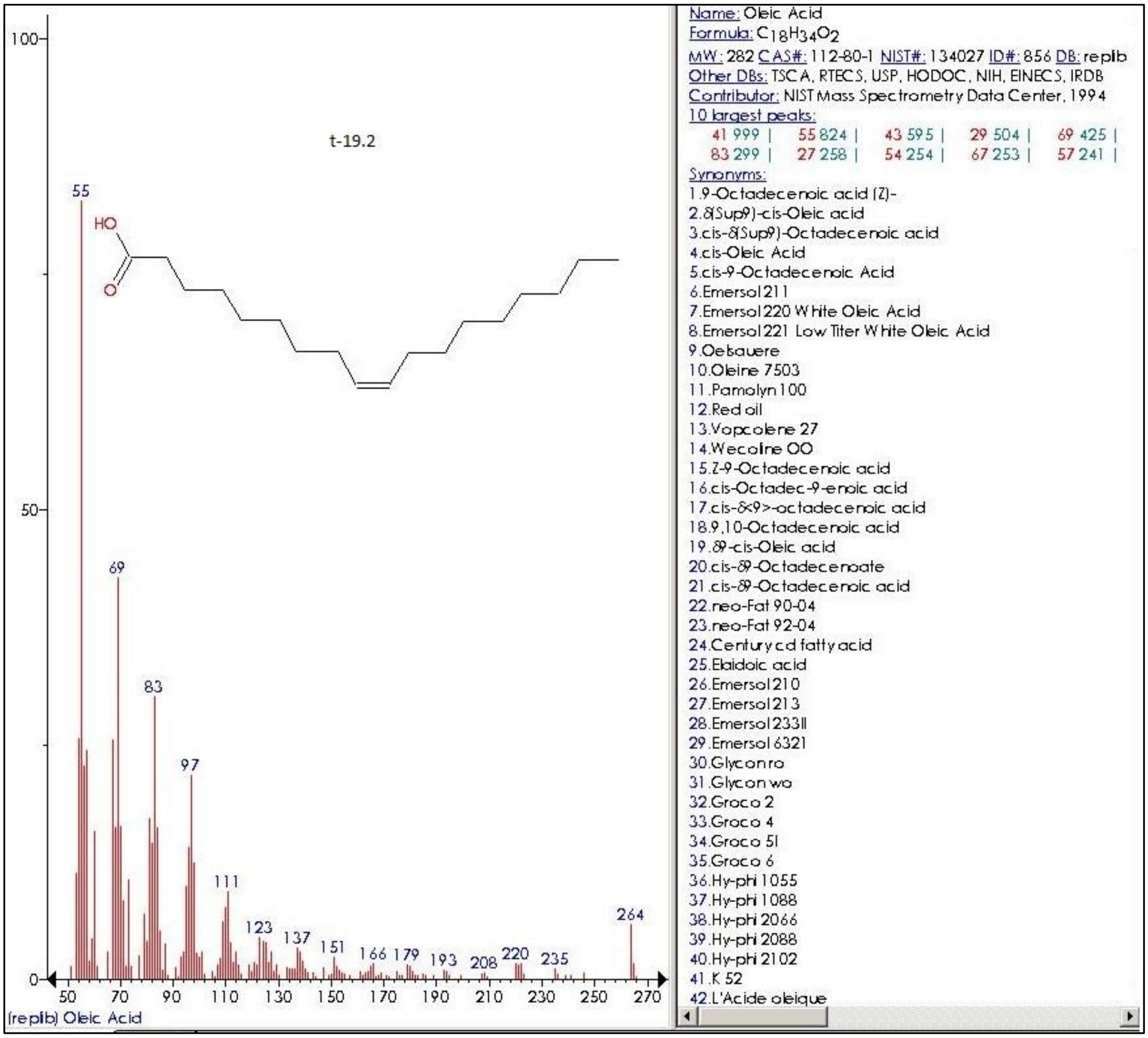
2. Value: 2069.8 iu
Column Type: Capillary
Column Class: Standard non-polar
Active Phase: DB-1
Column Length: 30 m
Carrier Gas: He
Column Diameter: 0.25 mm
Phase Thickness: 0.25 um
Data Type: Kovats RI
Program Type: Ramp
Start T: 40 C
End T: 325 C
Heat Rate: 3 K/min
Source: Sun, G.; Stremple, P. *Retention index characterization of f*

Figure C-47



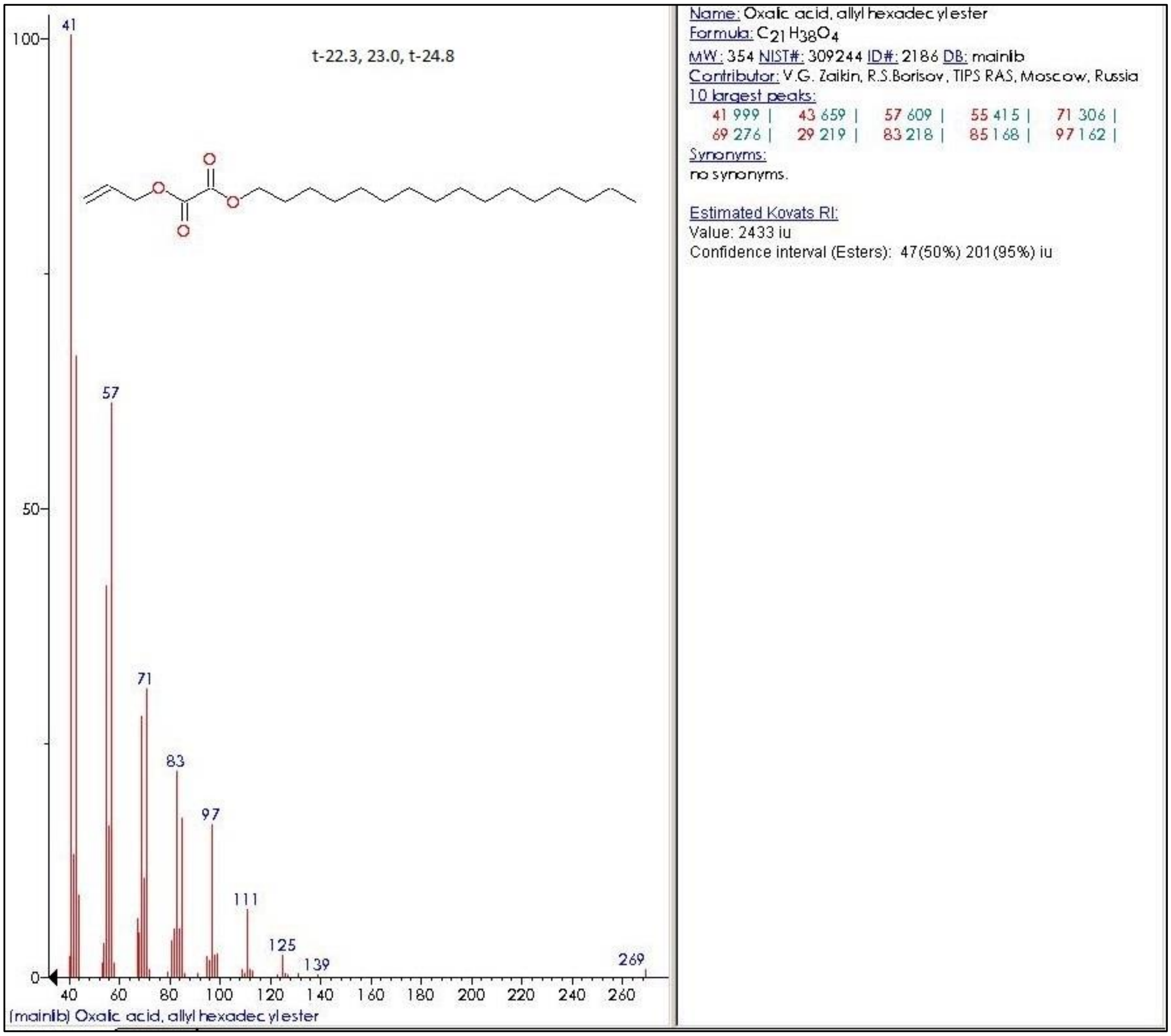
Name: Octadecanoic acid
Formula: $C_{18}H_{36}O_2$
MW: 284 CAS#: 57-11-4 NIST#: 287392 ID#: 1699 DB: replib
Other DBs: Fine, TSCA, RTECS, EPA, HODOC, NIH, EINECS, IRDE
Contributor: Humboldt-Universitat zu Berlin, Department of C
10 largest peaks:
43 999 | 41 866 | 60 808 | 73 808 | 55 669 |
57 631 | 29 483 | 69 346 | 71 309 | 129 280 |
Synonyms:
1.Stearic acid
2.n-Octadecanoic acid
3.Humko Industrene R
4.Hydrofol Acid 150
5.Hystrene S-97
6.Hystrene T-70
7.Hystrene 80
8.Industrene R
9.Kam 1000
10.Kam 2000
11.Kam 3000
12.Neo-Fat 18
13.Neo-Fat 18-53
14.Neo-Fat 18-54
15.Neo-Fat 18-55
16.Neo-Fat 18-59
17.NAA 173
18.PD 185
19.Stearex Beads
20.Stearophanic acid
21.Steric acid
22.Vanicol
23.1-Heptadecanecarboxylic acid
24.Heptadecanecarboxylic acid
25.Neo-fat 18-61
26.Pearl stearic
27.Century 1240
28.Dar-chem 14
29.Emersol 120
30.Emersol 132
31.Emersol 150
32.Formula 300
33.Glycon dp
34.Glycon tp
35.Glycon S-70
36.Glycon S-80
37.Glycon S-90
38.Groco 54
39.Groco 55
40.Groco 55l
41.Groco 58
42.Groco 59

Figure C-48



Name: Oleic Acid
Formula: $C_{18}H_{34}O_2$
MW: 282 CAS#: 112-80-1 NIST#: 134027 ID#: 856 DB: replib
Other DBs: TSCA, RTECS, USP, HODOC, NIH, EINECS, IRDB
Contributor: NIST Mass Spectrometry Data Center, 1994
10 largest peaks:
41 999 | 55 824 | 43 595 | 29 504 | 69 425 |
83 299 | 27 258 | 54 254 | 67 253 | 57 241 |
Synonyms:
1.9-Octadecenoic acid (Z)-
2.δ(Sup9)-cis-Oleic acid
3.cis-δ(Sup9)-Octadecenoic acid
4.cis-Oleic Acid
5.cis-9-Octadecenoic Acid
6.Emersol 211
7.Emersol 220 White Oleic Acid
8.Emersol 221 Low Titer White Oleic Acid
9.Oelsauere
10.Oleine 7503
11.Pamolyn 100
12.Red oil
13.Vopcolene 27
14.Wecoline OO
15.Z-9-Octadecenoic acid
16.cis-Octadec-9-enoic acid
17.cis-δ<9>-octadecenoic acid
18.9,10-Octadecenoic acid
19.δ9-cis-Oleic acid
20.cis-δ9-Octadecenoate
21.cis-δ9-Octadecenoic acid
22.neo-Fat 90-04
23.neo-Fat 92-04
24.Century cd fatty acid
25.Elaidoic acid
26.Emersol 210
27.Emersol 213
28.Emersol 233ll
29.Emersol 6321
30.Glycon ro
31.Glycon wo
32.Groco 2
33.Groco 4
34.Groco 5l
35.Groco 6
36.Hy-phi 1055
37.Hy-phi 1088
38.Hy-phi 2066
39.Hy-phi 2088
40.Hy-phi 2102
41.K 52
42.L'Acide oleique

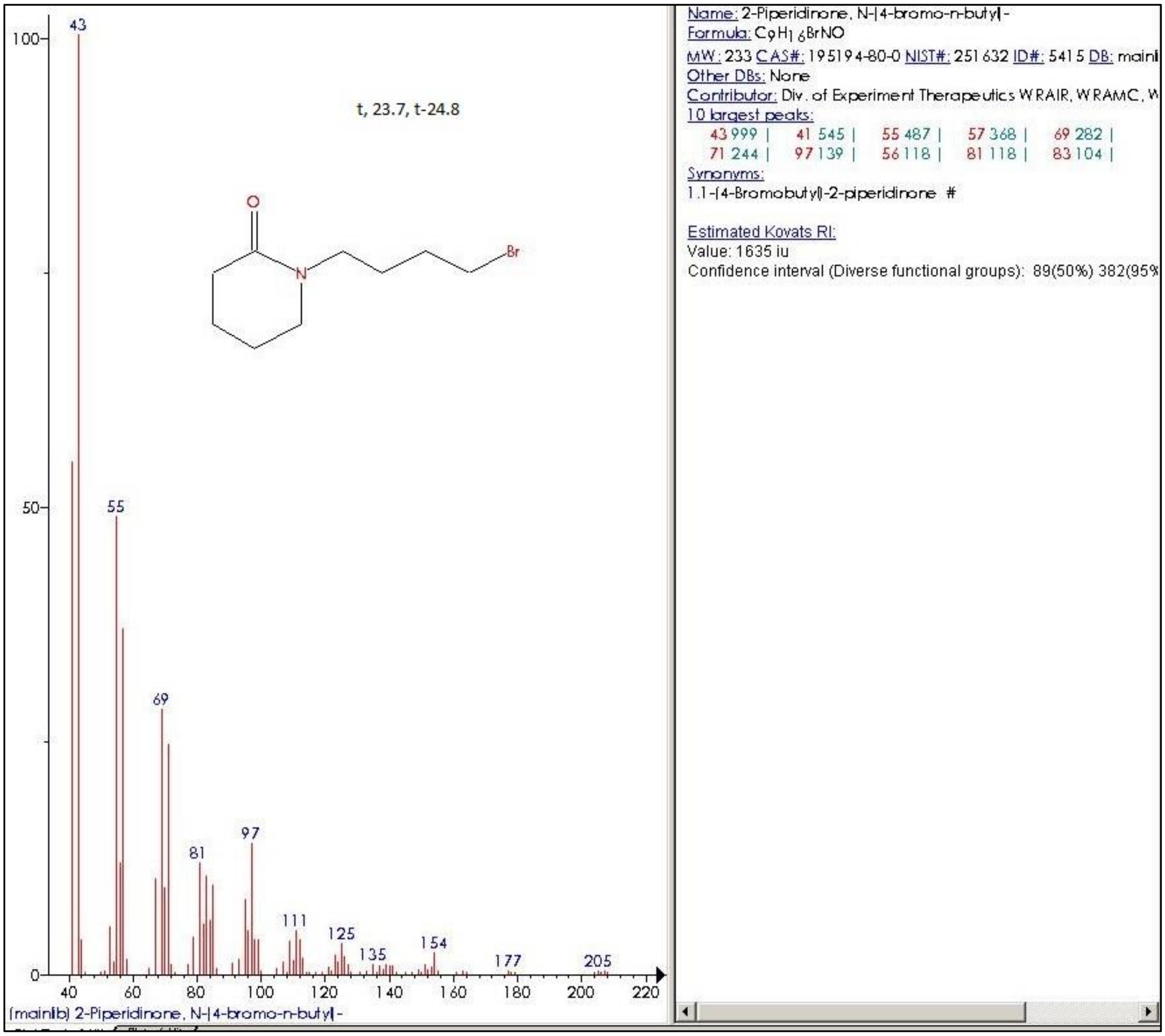
Figure C-49



Name: Oxalic acid, allyl hexadecyl ester
Formula: $C_{21}H_{38}O_4$
MW: 354 NIST#: 309244 ID#: 2186 DB: mainlib
Contributor: V.G. Zaikin, R.S.Borisov, TIPS RAS, Moscow, Russia
10 largest peaks:
41 999 | 43 659 | 57 609 | 55 415 | 71 306 |
69 276 | 29 219 | 83 218 | 85 168 | 97 162 |
Synonyms:
no synonyms.

Estimated Kovats RI:
Value: 2433 iu
Confidence interval (Esters): 47(50%) 201(95%) iu

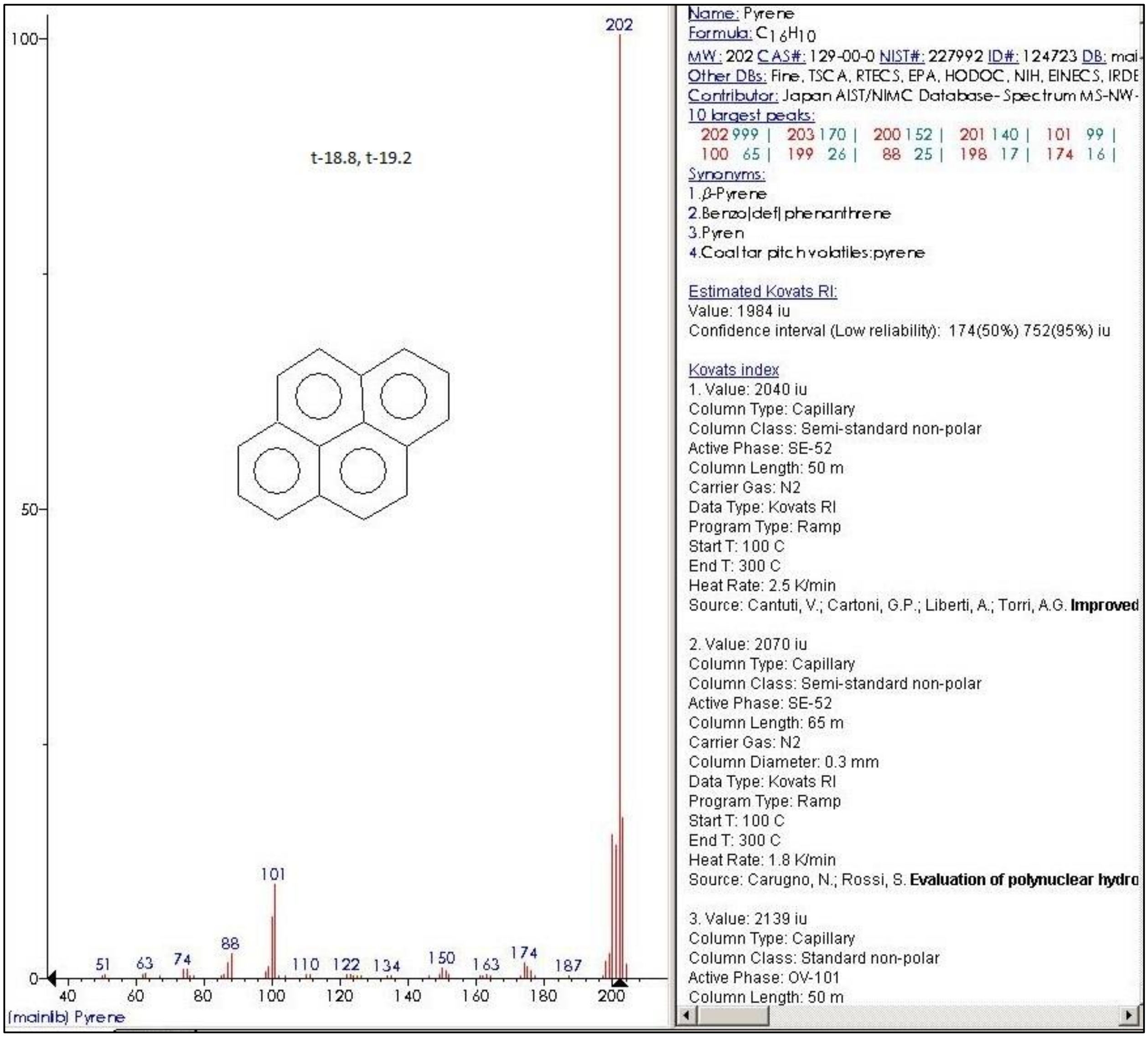
Figure C-50



Name: 2-Piperidinone, N-(4-bromo-n-butyl)-
Formula: $C_9H_{16}BrNO$
MW: 233 CAS#: 195194-80-0 NIST#: 251632 ID#: 5415 DB: mainl
Other DBs: None
Contributor: Div. of Experiment Therapeutics WRAIR, WRAMC, W
10 largest peaks:
43 999 | 41 545 | 55 487 | 57 368 | 69 282 |
71 244 | 97 139 | 56 118 | 81 118 | 83 104 |
Synonyms:
1.1-(4-Bromobutyl)-2-piperidinone #

Estimated Kovats RI:
Value: 1635 iu
Confidence interval (Diverse functional groups): 89(50%) 382(95%

Figure C-51



Name: Pyrene
Formula: $C_{16}H_{10}$
MW: 202 CAS#: 129-00-0 NIST#: 227992 ID#: 124723 DB: mai
Other DBs: Fine, TSCA, RTECS, EPA, HODOC, NIH, EINECS, IRDE
Contributor: Japan AIST/NIMC Database- Spectrum MS-NW-
10 largest peaks:
202 999 | 203 170 | 200 152 | 201 140 | 101 99 |
100 65 | 199 26 | 88 25 | 198 17 | 174 16 |
Synonyms:
1.β-Pyrene
2.Benzo|def| phenanthrene
3.Pyren
4.Coal tar pitch volatiles:pyrene

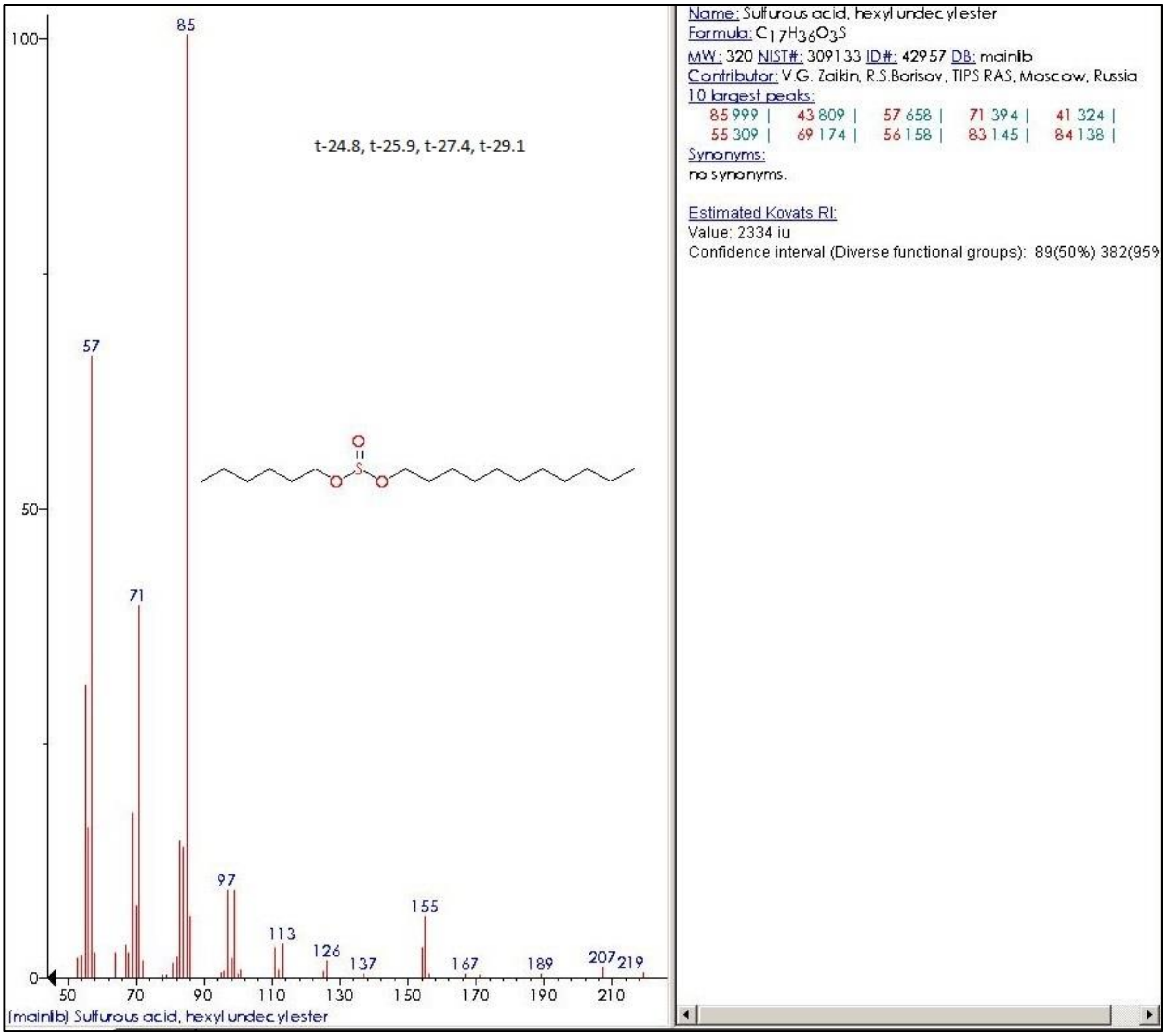
Estimated Kovats RI:
Value: 1984 iu
Confidence interval (Low reliability): 174(50%) 752(95%) iu

Kovats index
1. Value: 2040 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Column Length: 50 m
Carrier Gas: N2
Data Type: Kovats RI
Program Type: Ramp
Start T: 100 C
End T: 300 C
Heat Rate: 2.5 K/min
Source: Cantuti, V.; Cartoni, G.P.; Liberti, A.; Torri, A.G. **Improved**

2. Value: 2070 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: SE-52
Column Length: 65 m
Carrier Gas: N2
Column Diameter: 0.3 mm
Data Type: Kovats RI
Program Type: Ramp
Start T: 100 C
End T: 300 C
Heat Rate: 1.8 K/min
Source: Carugno, N.; Rossi, S. **Evaluation of polynuclear hydro**

3. Value: 2139 iu
Column Type: Capillary
Column Class: Standard non-polar
Active Phase: OV-101
Column Length: 50 m

Figure C-52



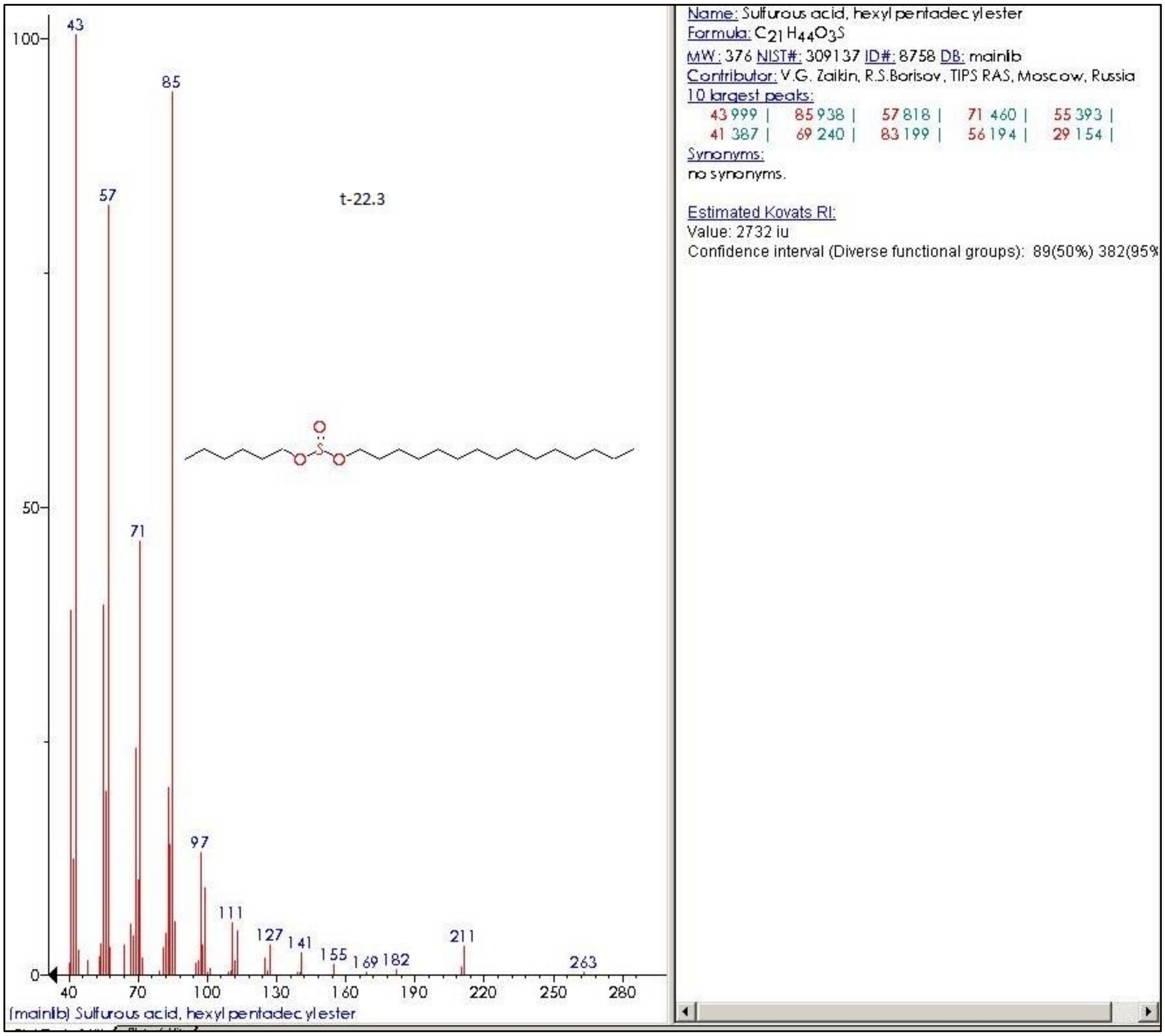
t-24.8, t-25.9, t-27.4, t-29.1

(mainlib) Sulfurous acid, hexyl undec yl ester

Name: Sulfurous acid, hexyl undec yl ester
Formula: $C_{17}H_{36}O_3S$
MW: 320 NIST#: 309133 ID#: 42957 DB: mainlib
Contributor: V.G. Zaikin, R.S.Borisov, TIPS RAS, Moscow, Russia
10 largest peaks:
85 999 | 43 809 | 57 658 | 71 394 | 41 324 |
55 309 | 69 174 | 56 158 | 83 145 | 84 138 |
Synonyms:
no synonyms.

Estimated Kovats RI:
Value: 2334 iu
Confidence interval (Diverse functional groups): 89(50%) 382(959

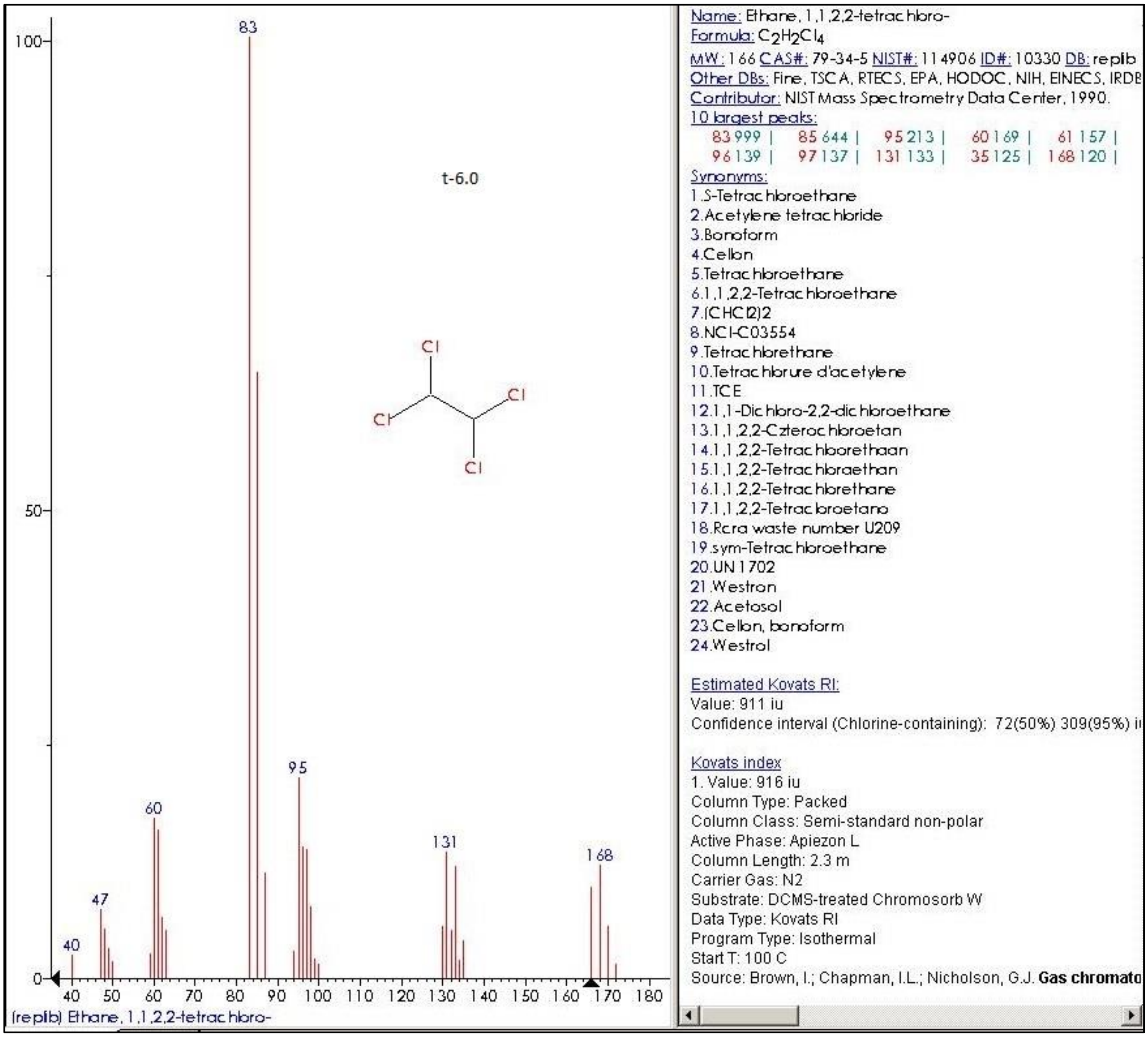
Figure C-53



Name: Sulfurous acid, hexyl pentadec yl ester
Formula: $C_{21}H_{44}O_3S$
MW: 376 NIST#: 309137 ID#: 8758 DB: mainlib
Contributor: V.G. Zaikin, R.S.Borisov, TIPS RAS, Moscow, Russia
10 largest peaks:
43 999 | 85 938 | 57 818 | 71 460 | 55 393 |
41 387 | 69 240 | 83 199 | 56 194 | 29 154 |
Synonyms:
no synonyms.

Estimated Kovats RI:
Value: 2732 iu
Confidence interval (Diverse functional groups): 89(50%) 382(95%

Figure C-54

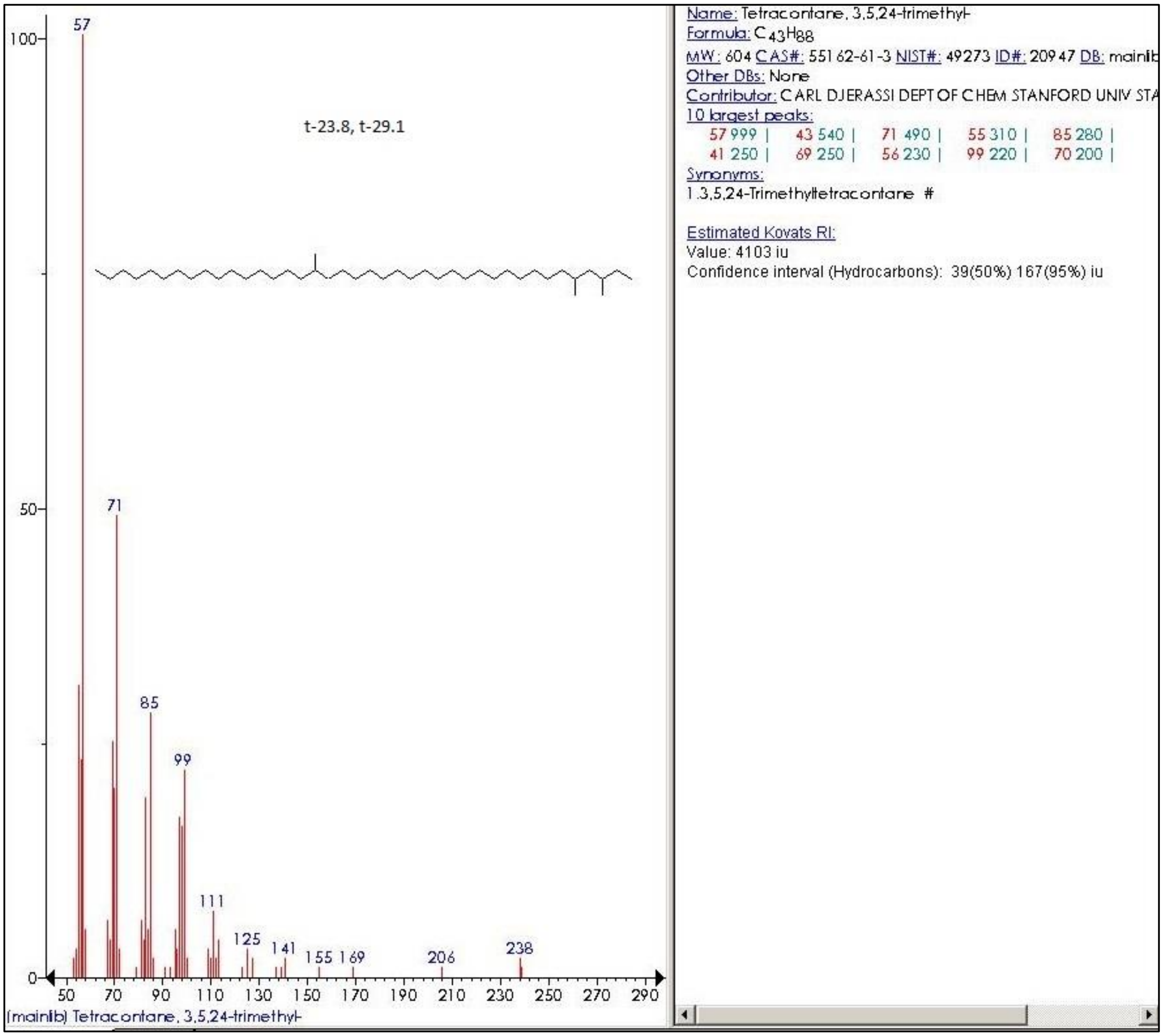


Name: Ethane, 1,1,2,2-tetrachloro-
Formula: $C_2H_2Cl_4$
MW: 166 CAS#: 79-34-5 NIST#: 114906 ID#: 10330 DB: replib
Other DBs: Fine, TSCA, RTECS, EPA, HODOC, NIH, EINECS, IRDB
Contributor: NIST Mass Spectrometry Data Center, 1990.
10 largest peaks:
83 999 | 85 644 | 95 213 | 60 169 | 61 157 |
96 139 | 97 137 | 131 133 | 35 125 | 168 120 |
Synonyms:
1.S-Tetrachloroethane
2.Acetylene tetrachloride
3.Bonoform
4.Cellon
5.Tetrachloroethane
6.1,1,2,2-Tetrachloroethane
7.(CHCl2)2
8.NCI-C03554
9.Tetrachlorethane
10.Tetrachlorure d'acetylene
11.TCE
12.1,1-Dichloro-2,2-dichloroethane
13.1,1,2,2-Czterochloroetan
14.1,1,2,2-Tetrachloorethaan
15.1,1,2,2-Tetrachloraethan
16.1,1,2,2-Tetrachlorethane
17.1,1,2,2-Tetracloroetano
18.Rcra waste number U209
19.sym-Tetrachloroethane
20.UN 1702
21.Westron
22.Acetosol
23.Cellon, bonoform
24.Westrol

Estimated Kovats RI:
Value: 911 iu
Confidence interval (Chlorine-containing): 72(50%) 309(95%) i

Kovats index
1. Value: 916 iu
Column Type: Packed
Column Class: Semi-standard non-polar
Active Phase: Apiezon L
Column Length: 2.3 m
Carrier Gas: N2
Substrate: DCMS-treated Chromosorb W
Data Type: Kovats RI
Program Type: Isothermal
Start T: 100 C
Source: Brown, I.; Chapman, I.L.; Nicholson, G.J. **Gas chromato**

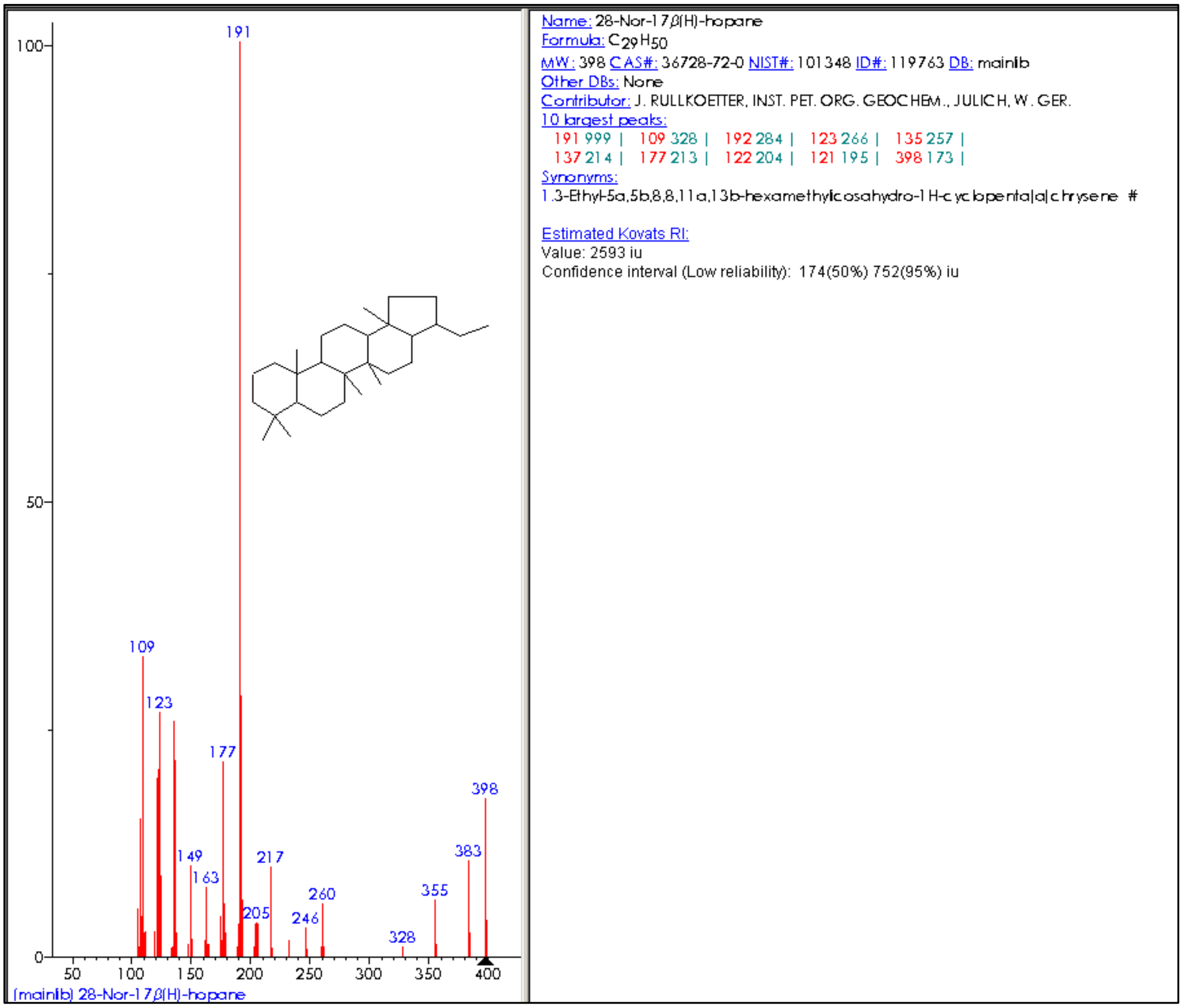
Figure C-55



Name: Tetracontane, 3,5,24-trimethyl-
Formula: $C_{43}H_{88}$
MW: 604 CAS#: 55162-61-3 NIST#: 49273 ID#: 20947 DB: mainlib
Other DBs: None
Contributor: CARL DJERASSI DEPT OF CHEM STANFORD UNIV STA
10 largest peaks:
57 999 | 43 540 | 71 490 | 55 310 | 85 280 |
41 250 | 69 250 | 56 230 | 99 220 | 70 200 |
Synonyms:
1.3,5,24-Trimethyltetracontane #

Estimated Kovats RI:
Value: 4103 iu
Confidence interval (Hydrocarbons): 39(50%) 167(95%) iu

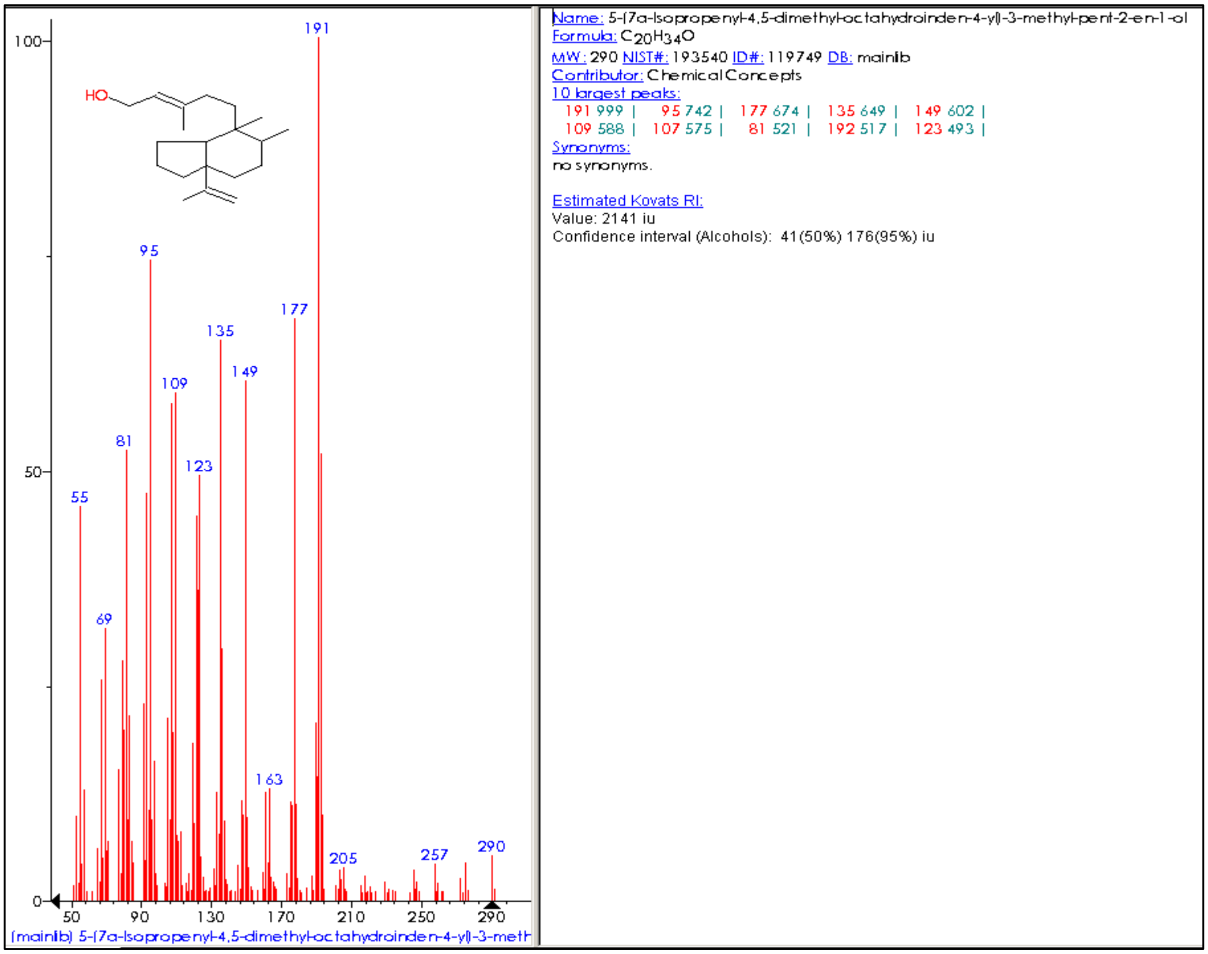
Figure C-56



Name: 28-Nor-17β(H)-hopane
Formula: $C_{29}H_{50}$
MW: 398 CAS#: 36728-72-0 NIST#: 101348 ID#: 119763 DB: mainlib
Other DBs: None
Contributor: J. RULLKOETTER, INST. PET. ORG. GEOCHEM., JULICH, W. GER.
10 largest peaks:
191 999 | 109 328 | 192 284 | 123 266 | 135 257 |
137 214 | 177 213 | 122 204 | 121 195 | 398 173 |
Synonyms:
1.3-Ethyl-5a,5b,8,8,11a,13b-hexamethylicosahydro-1H-cyclopenta[a]chrysene #

Estimated Kovats RI:
Value: 2593 iu
Confidence interval (Low reliability): 174(50%) 752(95%) iu

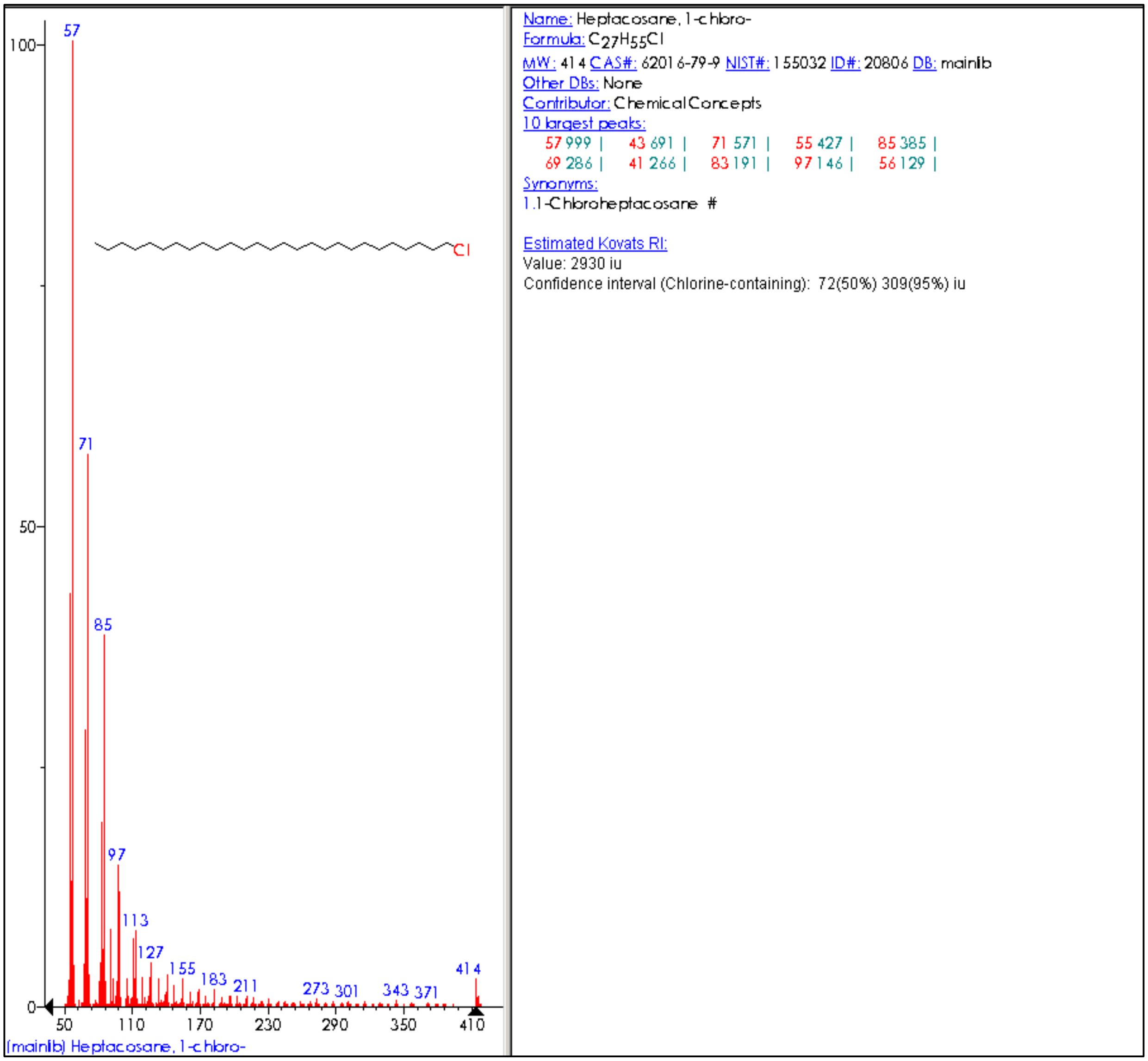
Figure C-57



Name: 5-(7a-Isopropenyl-4,5-dimethyl-octahydroinden-4-yl)-3-methyl-pent-2-en-1-ol
Formula: $C_{20}H_{34}O$
MW: 290 NIST#: 193540 ID#: 119749 DB: mainlib
Contributor: Chemical Concepts
10 largest peaks:
191 999 | 95 742 | 177 674 | 135 649 | 149 602 |
109 588 | 107 575 | 81 521 | 192 517 | 123 493 |
Synonyms:
no synonyms.

Estimated Kovats RI:
Value: 2141 iu
Confidence interval (Alcohols): 41(50%) 176(95%) iu

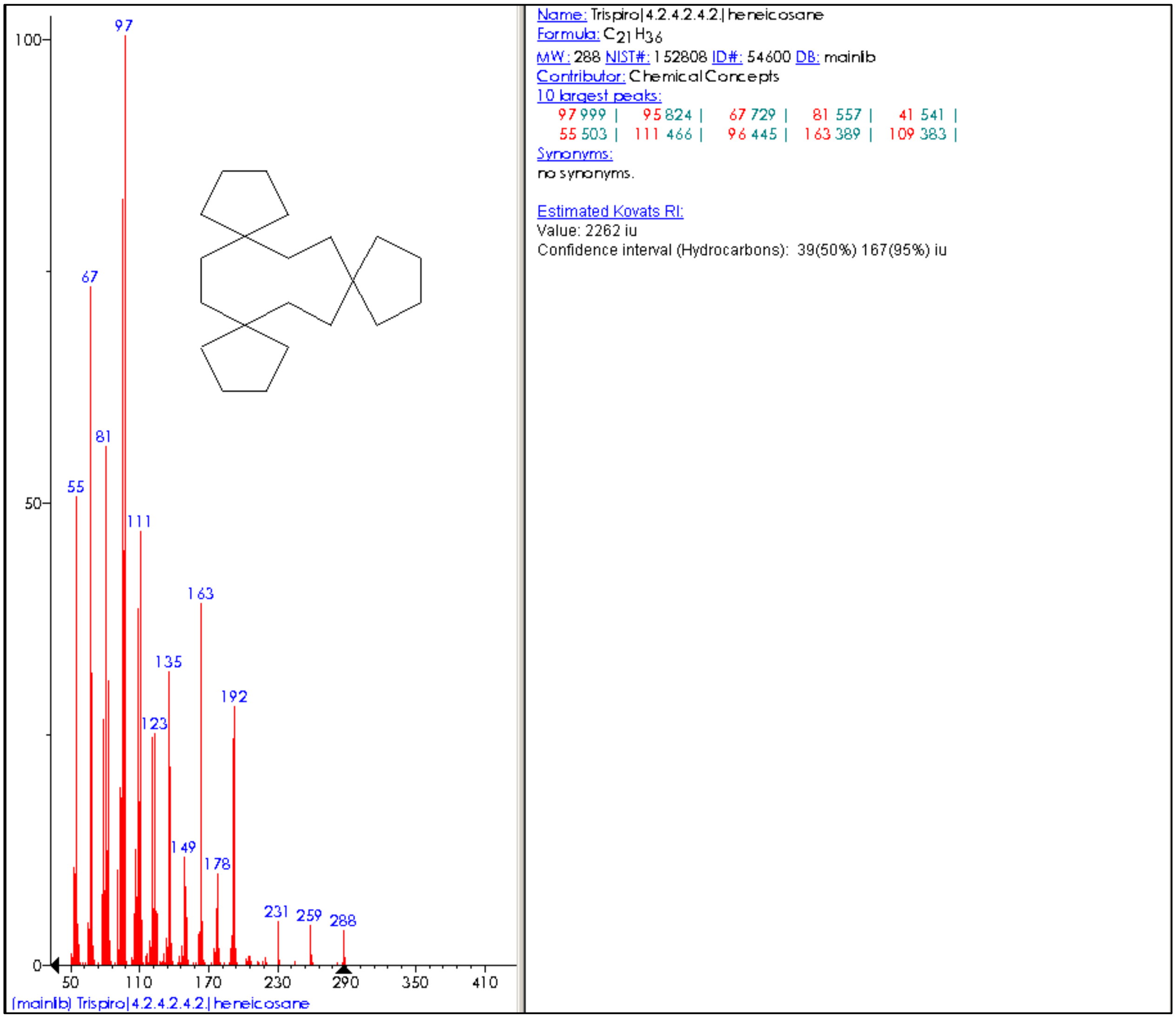
Figure C-58



Name: Heptacosane, 1-chloro-
Formula: $C_{27}H_{55}Cl$
MW: 414 CAS#: 62016-79-9 NIST#: 155032 ID#: 20806 DB: mainlib
Other DBs: None
Contributor: Chemical Concepts
10 largest peaks:
57 999 | 43 691 | 71 571 | 55 427 | 85 385 |
69 286 | 41 266 | 83 191 | 97 146 | 56 129 |
Synonyms:
1.1-Chloroheptacosane #

Estimated Kovats RI:
Value: 2930 iu
Confidence interval (Chlorine-containing): 72(50%) 309(95%) iu

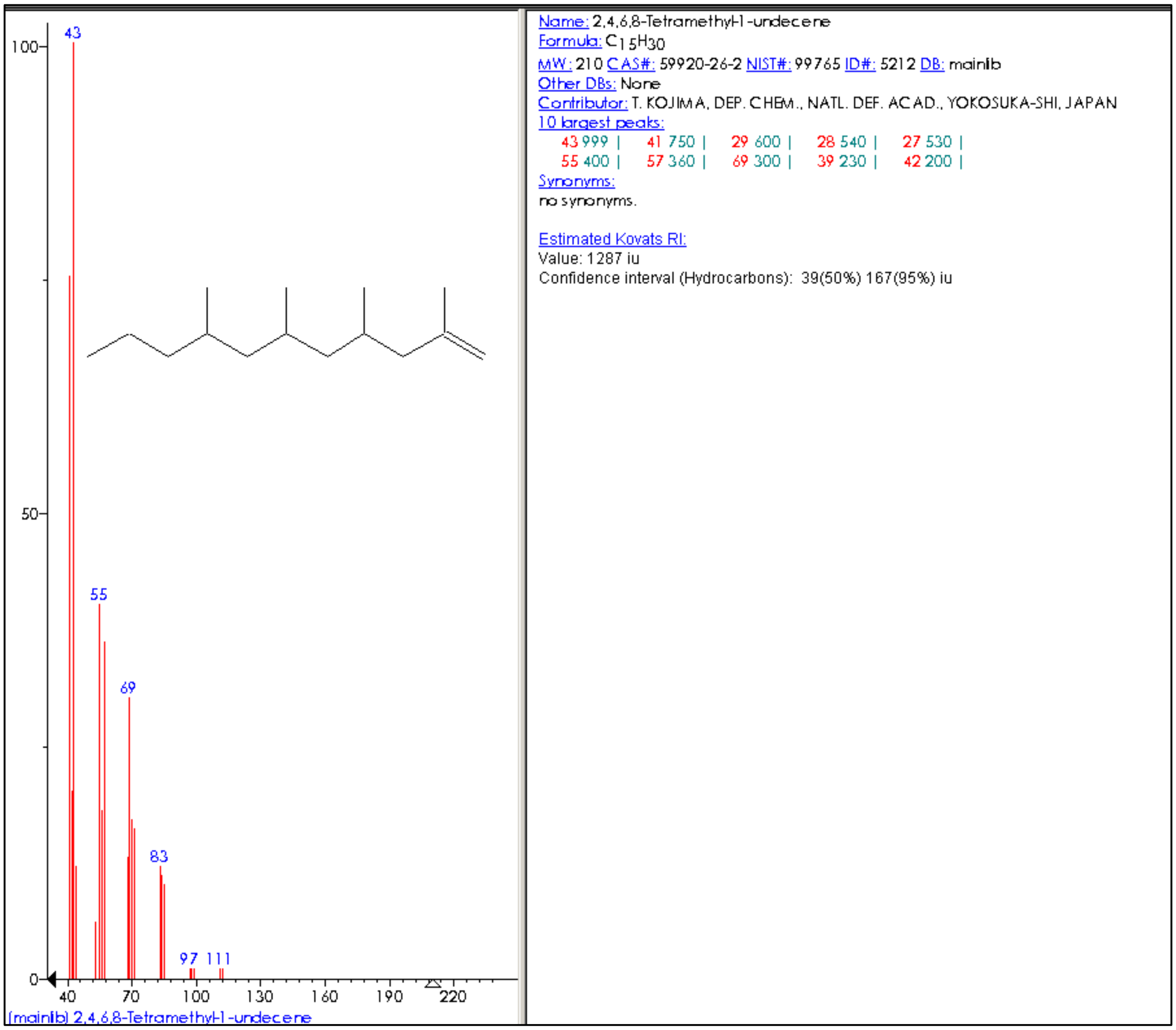
Figure C-59



Name: Trispiro|4.2.4.2.4.2.|heneicosane
Formula: $C_{21}H_{36}$
MW: 288 NIST#: 152808 ID#: 54600 DB: mainlib
Contributor: Chemical Concepts
10 largest peaks:
97 999 | 95 824 | 67 729 | 81 557 | 41 541 |
55 503 | 111 466 | 96 445 | 163 389 | 109 383 |
Synonyms:
no synonyms.

Estimated Kovats RI:
Value: 2262 iu
Confidence interval (Hydrocarbons): 39(50%) 167(95%) iu

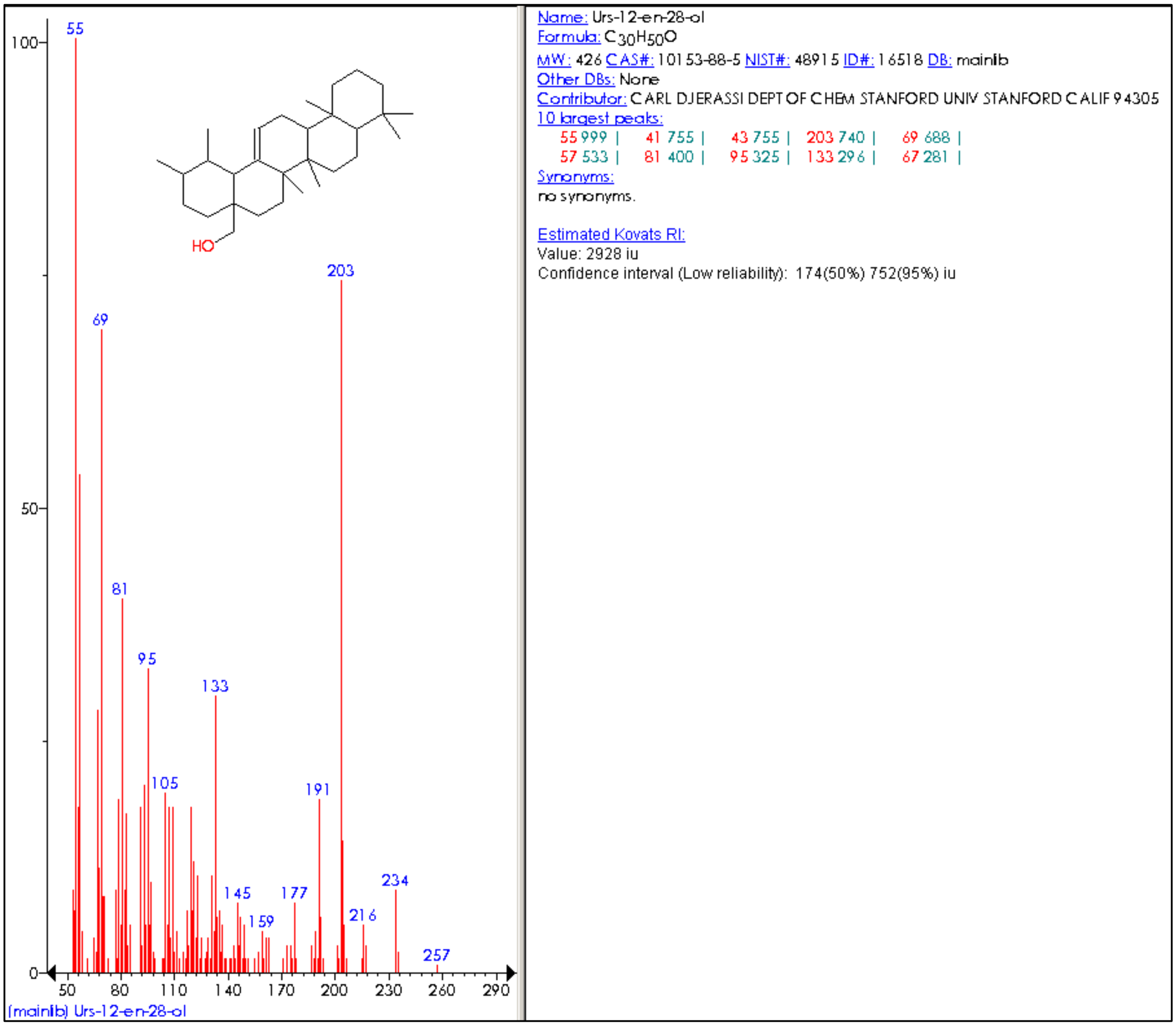
Figure C-60



Name: 2,4,6,8-Tetramethyl-1-undecene
Formula: $C_{15}H_{30}$
MW: 210 CAS#: 59920-26-2 NIST#: 99765 ID#: 5212 DB: mainlib
Other DBs: None
Contributor: T. KOJIMA, DEP. CHEM., NATL. DEF. ACAD., YOKOSUKA-SHI, JAPAN
10 largest peaks:
43 999 | 41 750 | 29 600 | 28 540 | 27 530 |
55 400 | 57 360 | 69 300 | 39 230 | 42 200 |
Synonyms:
no synonyms.

Estimated Kovats RI:
Value: 1287 iu
Confidence interval (Hydrocarbons): 39(50%) 167(95%) iu

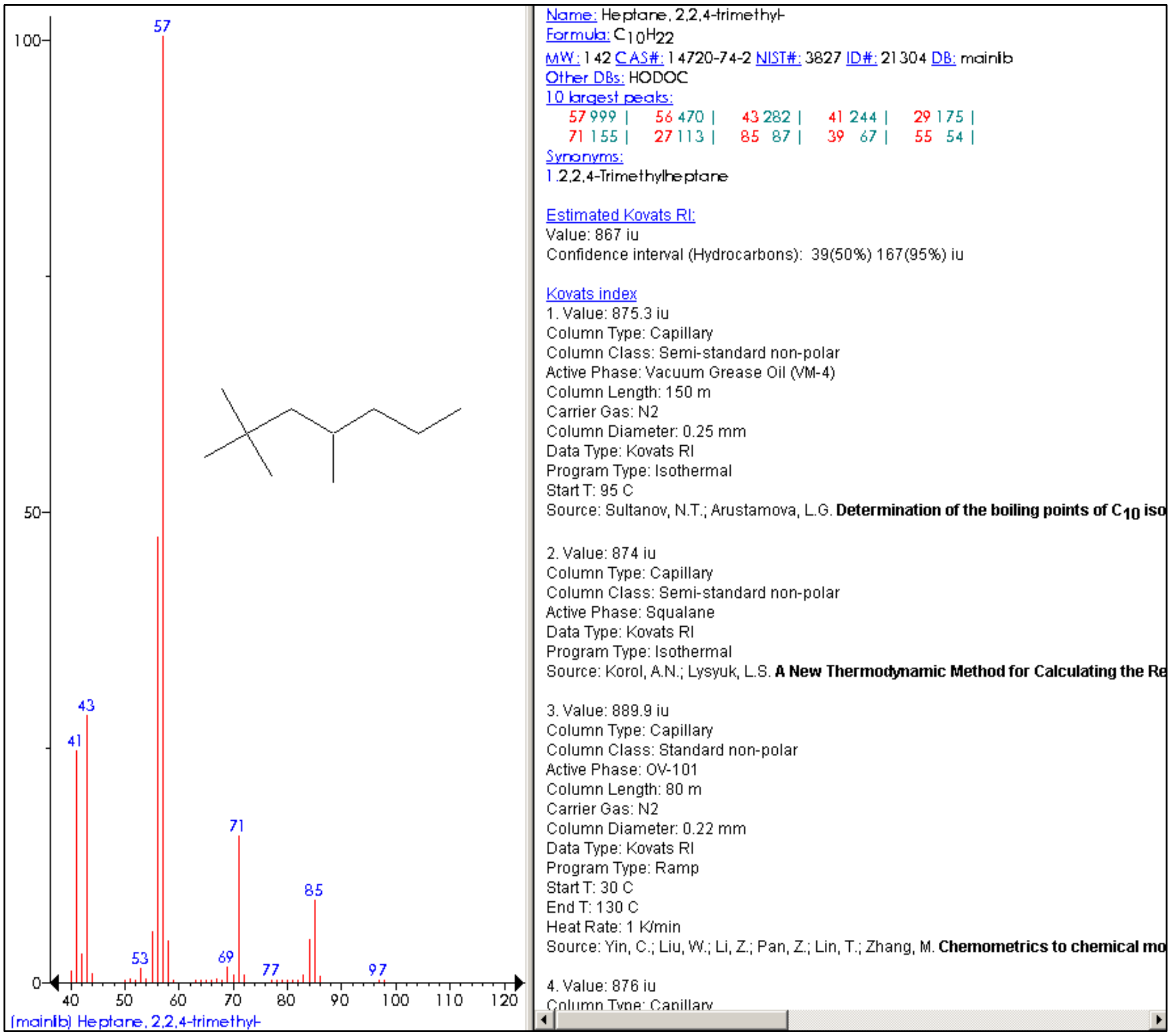
Figure C-61



Name: Urs-12-en-28-ol
Formula: $C_{30}H_{50}O$
MW: 426 CAS#: 10153-88-5 NIST#: 48915 ID#: 16518 DB: mainlib
Other DBs: None
Contributor: CARL DJERASSI DEPT OF CHEM STANFORD UNIV STANFORD CALIF 94305
10 largest peaks:
55 999 | 41 755 | 43 755 | 203 740 | 69 688 |
57 533 | 81 400 | 95 325 | 133 296 | 67 281 |
Synonyms:
no synonyms.

Estimated Kovats RI:
Value: 2928 iu
Confidence interval (Low reliability): 174(50%) 752(95%) iu

Figure C-62



Name: Heptane, 2,2,4-trimethyl-
Formula: $C_{10}H_{22}$
MW: 142 CAS#: 14720-74-2 NIST#: 3827 ID#: 21304 DB: mainlib
Other DBs: HODOC
10 largest peaks:
57 999 | 56 470 | 43 282 | 41 244 | 29 175 |
71 155 | 27 113 | 85 87 | 39 67 | 55 54 |
Synonyms:
1.2,2,4-Trimethylheptane

Estimated Kovats RI:
Value: 867 iu
Confidence interval (Hydrocarbons): 39(50%) 167(95%) iu

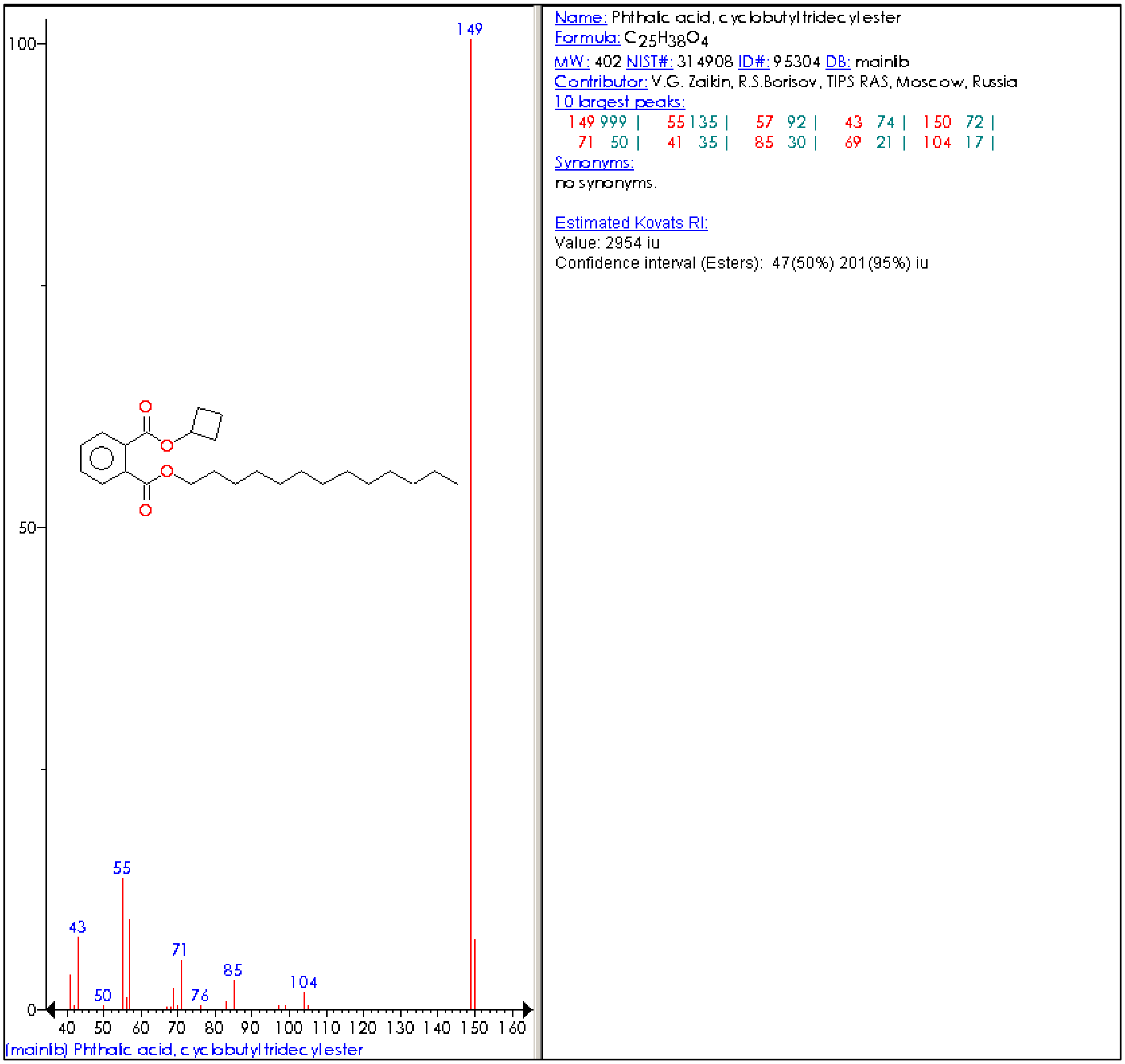
Kovats index
1. Value: 875.3 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: Vacuum Grease Oil (VM-4)
Column Length: 150 m
Carrier Gas: N2
Column Diameter: 0.25 mm
Data Type: Kovats RI
Program Type: Isothermal
Start T: 95 C
Source: Sultanov, N.T.; Arustamova, L.G. **Determination of the boiling points of C_{10} iso**

2. Value: 874 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: Squalane
Data Type: Kovats RI
Program Type: Isothermal
Source: Korol, A.N.; Lysyuk, L.S. **A New Thermodynamic Method for Calculating the Re**

3. Value: 889.9 iu
Column Type: Capillary
Column Class: Standard non-polar
Active Phase: OV-101
Column Length: 80 m
Carrier Gas: N2
Column Diameter: 0.22 mm
Data Type: Kovats RI
Program Type: Ramp
Start T: 30 C
End T: 130 C
Heat Rate: 1 K/min
Source: Yin, C.; Liu, W.; Li, Z.; Pan, Z.; Lin, T.; Zhang, M. **Chemometrics to chemical mo**

4. Value: 876 iu
Column Type: Capillary

Figure C-63



Name: Phthalic acid, cyclobutyl tridecyl ester
Formula: $C_{25}H_{38}O_4$
MW: 402 NIST#: 314908 ID#: 95304 DB: mainlib
Contributor: V.G. Zaikin, R.S.Borisov, TIPS RAS, Moscow, Russia
10 largest peaks:
149 999 | 55 135 | 57 92 | 43 74 | 150 72 |
71 50 | 41 35 | 85 30 | 69 21 | 104 17 |
Synonyms:
no synonyms.

Estimated Kovats RI:
Value: 2954 iu
Confidence interval (Esters): 47(50%) 201(95%) iu

Figure C-64