Greenpeace Research Laboratories Analytical Results 2017-01

Analysis of wastewaters and associated sediments collected from two industrial parks in China for the presence of metals and organic chemical contaminants

Melissa Wang, Iryna Labunska, Kevin Brigden, and David Santillo

Introduction

Six samples of wastewater and three samples of sediment were received from Greenpeace East Asia for analysis at the Greenpeace Research Laboratories between 22nd August 2016 and 29th September 2016. According to documentation supplied, the first batch of samples was collected from discharge pipes exiting from the territory of Qilu Chemical Industrial Park facilities (Zibo, Shandong Province, China) on 16th August 2016. The main facilities operating at this location are associated with petrochemical, chlor-alkali and coal-related chemical industries. The second batch of samples was collected from Lianyungang Chemical Industrial Park (Duigougang Town, Jiangsu Province, China) between 19th and 20th September 2016. The construction of Lianyungang Industrial Park started in 2003[1] and was the only provincial-level industrial park in Norther Jiangsu Province as approved by the government in May 2006[2]. Pesticides, dyes, pharmaceutical products and biochemicals are among its major products[3].

Details of the samples received are provided in Table 1a, together with GPS coordinates for the sample collection locations in Table 1b.

All samples were analysed quantitatively for the presence of a range of metals. Concentrations of metals in both whole and filtered water were determined in order to distinguish between metals associated with suspended matter and those present in dissolved form in the water. All samples were also analysed qualitatively for the presence of semi-volatile (solvent-extractable) organic compounds, and separately for the presence of volatile organic compounds (VOCs), including quantitative analysis for a range of common VOC environmental contaminants.

Greenpeace Research Laboratories
School of Biosciences
Innovation Centre Phase 2
Rennes Drive
University of Exeter
Exeter EX4 4RN, UK

[1]Guannan Town Year Book, 2004, Yu Hongjing (ed), 灌南县地方志编纂委员会, Jilin People Publishing House, p. 251.

[2] Lianyungang Year Book, 2016, Lv Weiguang, 江苏连云港化工产业园_概况, p. 138.

[3].Lianyungang Chemical Industrial Park website, http://js.zhaoshang.net/yuanqu/detail/4045/intro. Accessed 28.03.2017

Sample code	Sample type	Date of sampling	Location	Description
Area 1 - Qilu Chemical Industrial Park				
CN16001	Wastewater	2016.08.16	Point of the three pipelines are on the territory of industrial park, and beside the centralised WWTP. Pipes discharge wastewater openly into an underground collector/pipeline. The origin of the pipes is unknown, as is the final fate of the mixed wastewater.	Sample collected from the 1st of the three pipes; sample has strong smell.
CN16002	Wastewater	2016.08.16		Sample collected from the 3rd of the three pipes; discharge has faster flow than from 1st pipe; sample has strong smell.
Area 2 - Lianyungang Chemical Industrial Park				
CN16003	Wastewater	2016.09.19	Small River running through the Industrial park. Sampling was done at the point between a steel factory and a chemical factory. River is crossed by a wastewater channel at a point approx. 300m SW from the sampling point.	Sample has green-yellow colour and strong smell.
CN16004	Sediment	2016.09.19		Sample consists of black viscous mud; sample has strong smell.
CN16005	Wastewater	2016.09.19	Wastewater channel running through the Industrial park. Wastewater from both ends of the channel is discharged into GuanHe River that later meets the Yellow Sea (approx. 8km to the north). Sampling was done at the northern end of the two wastewater discharge points into the GuanHe River, adjacent to a sluice gate.	Brown colour sample; high suspended solids; collected at low tide; sample has strong smell.
CN16006	Sediment	2016.09.19		Sample consists of dark brown mud; collected at high tide; sample has strong smell.
CN16007	Wastewater	2016.09.20		Yellow-green colour sample; high suspended solids; sample has strong smell.
CN16008	Sediment	2016.09.20		Sample consists of brown viscous mud.
CN16009	Wastewater	2016.09.20		Yellow-green colour sample; white foam observed during sampling; sample has strong smell.

Table 1a. Description of the samples collected at Qilu Chemical Industrial Park and Lianyungang Chemical Industrial Park.

Sample code	N			E		
	degree (º)	minutes (')	seconds ('')	degree (º)	minutes (')	seconds ('')
CN16001	36	47	26.0	118	11	39.9
CN16002						
CN16003	34	23	6.9	119	46	49.7
CN16004						
CN16005	34	23	58.8	119	46	46.8
CN16006						
CN16007						
CN16008						
CN16009						

Table 1b: GPS coordinated of sample collection locations

Materials and methods

All samples were collected in pre-cleaned glass bottles and kept cold and dark before shipment to our laboratory in the UK for analysis.

Heavy metal concentrations were determined for all samples by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) following acid digestion and using appropriate certified reference materials in addition to intra-laboratory standards. For water samples from Area 2, both the total concentrations in the whole (unfiltered) sample and the concentrations of dissolved forms in a filtered sample were determined separately for each sample. Water samples from Area 1 did not contain any visible suspended solids and therefore only the total concentrations in the whole (unfiltered) samples were determined. Hexavalent chromium concentrations in water samples were determined colourimetrically using a diphenylcarbazide method.

Semi-volatile organic compounds were isolated from samples using solid phase extraction (SPE) with ethyl acetate, pentane and toluene (for water samples) or Accelerated Solvent Extraction (ASE) with a mixture of pentane and acetone (for sediment samples). Extracted compounds were subsequently identified as far as possible using gas chromatography/mass spectrometry (GC/MS) operated in both SCAN and SIM modes. A multiresidue LC-HRMS analysis for traces of 274 pesticides was also conducted for all the samples.

Volatile organic chemicals (VOCs) were identified and quantified in all wastewater samples as received (with anhydrous sodium sulfate added to partition VOCs into headspace) using GC/MS with HeadSpace sample introduction technique.

More detailed descriptions of the sample preparation and analytical procedures are presented in the Appendix 1, including a list of VOCs quantified in the samples, method detection limit (MDL) and limits of quantification (LOQ) (Tables 9a&b).

Results and Discussion

The results for the samples are outlined in the following sections, divided into areas in which the samples were collected. For each section, the concentrations of metals and metalloids in whole waters (dissolved and suspended metals), in filtered (dissolved metals) waters, and/or in sediments are reported, in Tables 2, 5 and 6. In addition, the quantification of certain VOCs in wastewater samples are summarized in tables 3 and 7. Groups of organic chemicals identified in individual samples are summarised in Tables 4 and 8. A full list of organic chemicals identified in each sample is provided in Appendix 2.

In some cases, concentrations of metals and metalloids were below limits of quantification for the analytical methods employed in this study. These are shown in the results tables as '<xx' where xx is the method quantification limit for the individual metal or metalloid.

Area 1 - Qilu Chemical Industry Park

Qilu Chemical Industry Park was set up in 2002, with an overall planning area of 48 square kilometers.[4] By the end of 2015, 224 enterprises of various sectors of chemical industry (e.g., petrochemical, chlor-alkali, and coal-related industries) operated in this park.[5] The major products are derived from oil refining and coal gasification as well as from manufacture of polymers and fine chemicals.[6]

Three parallel pipes located next to each other and running beside the territory of the centralized WWTP of the Qilu Chemical Industry Park were identified during sampling trip. However, the origin of the wastewater discharging via these pipes into underground pipeline/reservoir is unclear. Likewise, the further fate of these wastewaters is also unknown. No discharge was observed from one of the pipes at the sampling time, hence, wastewater samples CN16001 & CN16002 were collected directly from each of the two operating pipes.

Metals

Neither water sample from Area 1 contained any visible suspended solids, and therefore the total concentrations in the whole (unfiltered) samples were determined, but the concentrations of dissolved forms in a filtered sample were not separately determined for each sample.

With one exception (zinc in CH16002), the concentrations of the quantified metals in both samples of wastewater collected in this area indicated that they were not notably contaminated with these metals, with little sign of elevation in concentrations above typical background concentrations for uncontaminated surface freshwaters. The concentration of zinc in CH16002 was 747 µg/l (0.747 mg/l), while background concentrations for uncontaminated surface freshwaters are typically below 50 µg/l (ATSDR 2005a, Salomons & Forstner 1984).

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[4]Qilu Chemical Industry Park website, http://www.qcip.gov.cn/about.php?id=1. Accessed 01.11.2016.
[5] Bureau Commerce of Zibo City
[6] Qilu Chemical Industry Park website, http://www.qcip.gov.cn/news.php?sort=2. Accessed 01.11.2016.

Metal	CN16001	CN16002
	Wastewater	
Antimony	1.0	<0.2
Arsenic	14	2.0
Beryllium	<0.01	<0.01
Cadmium	0.14	0.39
Chromium	0.5	2.4
Chromium (VI)	<50	<50
Cobalt	0.2	0.4
Copper	1	19
Iron	80	289
Lead	2.3	1.4
Manganese	32	13
Mercury	0.08	<0.05
Nickel	2.0	1.9
Selenium	2	2
Vanadium	3.4	4.7
Zinc	18	747

Table 2: Concentrations of metals and metalloids (μg/l) in whole samples CN16001 & CN16002.

Organic chemical contaminants

Both wastewater effluent samples collected at this location contained complex mixtures of organic chemical contaminants, of which a significant proportion in each case could not be identified. In the case of sample CN16001, unidentifiable compounds made up the majority (more than 60%) of the 86 compounds isolated. Among the 33 chemicals that could be identified in this sample, 13 (almost 40%) were chlorinated chemicals, including several chloromethyl butene compounds, chlorinated benzenes and a variety of volatile chlorinated methanes, ethanes and ethenes. A single organophosphorus chemical, a tributyl ester of phosphoric acid, was also indentified, as well as a number of phthalate esters.

Of particular note was the very high concentration of the volatile chlorinated compound trichloromethane, or chloroform, which, at more than 1.6 mg/l, undoubtedly contributed to the strong smell associated with the sample at the time of collection. The concentration of chloroform in this sample exceeds the maximum allowed discharge concentration (i.e. the most permissive standard) specified in table 4 of GB8978-1996 Integrated waste water discharge standard, which is for waste water samples collected at the discharge point of facilities established after January 1st, 1998[7]. Its presence at such high concentrations is a strong indicator of wastewater discharge from industrial chlorine chemistry processes, either from deliberate use of chloroform as a solvent, or its generation as a by-product from the manufacture of other chlorinated compounds or, perhaps, final treatment of complex wastewaters with chlorine for disinfection purposes. Chloroform is toxic by

[7] GB8978-1996 Integrated wastewater discharge standard of the People's Republic of China, Ministry of Environmental Protection of the People's Republic of China, 1996. http://kjs.mep.gov.cn/hjbhbz//bzwb/shjbh/swrwpfbz/199801/W020061027521858212955.pdf

inhalation, in contact with skin and if swallowed, with the liver and kidney being the primary organs affected, and is reasonably anticipated to be a human carcinogen.

Compound	CN16001	CN16002
	Wastewater	
Ethene, 1,1-dichloro-	<2	3
Methane, dichloro-	<2	N.D.
Ethane, 1,1-dichloro-	<2	<2
Ethene, 1,2-dichloro-, cis-	N.D.	<2
Chloroform	1646	<2
Methane, tetrachloro-	2	<2
Benzene	N.D.	<2
Ethane, 1,2-dichloro-	2	<2
Ethene, trichloro-	3	<2
Propane, 1,2-dichloro-	<2	N.D.
Methane, bromodichloro-	168	<2
Toluene	<2	<2
1,1,2-trichloroethane	47	4
Ethene, tetrachloro-	<2	<2
Methane, dibromochloro-	116	<2
Benzene, chloro-	<2	N.D.
o-Xylene	N.D.	<2
Styrene	<2	<2
Bromoform	23	<2
1,2-dichlorobenzene	<2	N.D.
1,2,4-trichlorobenzene-	<2	N.D.

Table 3. Quantification results of several VOCs (µg/L) identified in sample CN16001& CN16002.

Note:

1. *N.D.: not detected in sample*
2. *In cases in which undiluted extracts gave results outside the calibration range, the results reported are those following dilution, though in these cases, reported results could be underestimates of the concentrations in the original samples as a result of loss of VOCs through volatilization during the dilution process*

Sample code	**CN16001**	**CN16002**
Sample type	ww	ww
Number of compounds isolated	86	33
Number of compounds identified	33	22
% of compounds identified	38%	67%
	Number of compounds (and % of total identified)	
Chlorinated	13 (39%)	9 (41%)
Brominated	1 (3%)	1 (5%)
Mixed halogenated	2 (6%)	2 (9%)
Organophosphorous	1 (3%)	nd

Table 4: Summary of key organic chemical groups isolated from wastewater (ww) samples collected at Qilu Chemical Industry Park and identified to a high degree of reliability.

Area 2 - Lianyungang Chemical Industrial Park

Lianyungang Industry Park was set up in the year of 2003 and has an overall planning area of 30 square kilometers. At present, there are more than 100 enterprises operating in this industrial area. Lianyungang Chemical Industrial Park was originally designated for pesticide production and currently it has four industrial sectors: pesticide, dyes, medicinal and biochemical industry.[8]

The channel running through the Lianyungang Industrial Park receives wastewater from plants located on its territory. This wastewater channel discharges into the GuanHe River, which later meets the Yellow Sea. There is also a small river running through the Industrial Park (roughly west to east) and through local village. This small river is crossed by the wastewater channel (which runs roughly perpendicular to the river, north to south). Hence, water from this small river is mixed with water and effluents entering from the wastewater channel. All watercourses at this location experience variable directions and rates of flow due to tidal processes.

Two samples, water CN16003 and sediment CN16004, were collected from this small river about 300m away from the point of its crossing with the wastewater channel. Due to low accessibility to wastewater discharge pipes from individual enterprises located within this area, three water samples (CN16005, CN16007, and CN16009) and two sediment samples (CN16006, and CN16008) were collected directly from the wastewater discharge channel at its northern end, close to the point at which it joins with the GuanHe River, at different times and states of the tide.

Metals

The concentrations of the quantified metals in all samples of wastewater collected in this area indicated that they were not notably contaminated with these metals, with little sign of elevation in concentrations above typical background concentrations for uncontaminated surface freshwaters (Salomons & Forstner 1984).

For one sediment sample (CN16004), the concentrations of certain metals were somewhat higher than concentrations typically found in uncontaminated freshwater sediments; by approximately 2 times for nickel, 3 times for chromium and lead, and 5 times for copper and zinc (ATSDR 2004, 2005a & b, 2008; Salomons & Forstner 1984).

[8] Lianyungang Chemical Industrial Park website, http://js.zhaoshang.net/yuanqu/detail/4045/intro.

Metals	CN16003		CN16005		CN16007		CN16009	
	F	W	F	W	F	W	F	W
	Wastewater							
Antimony	1.4	1.4	2.6	2.1	2.6	2.7	2.8	4.0
Arsenic	5.8	8.2	25.8	684	8.3	14.6	8.1	31.2
Beryllium	<0.1	<0.1	<0.1	35	<0.1	1	<0.1	3
Cadmium	0.1	0.3	0.1	7.7	<0.1	0.3	0.2	1.4
Chromium	2.0	14.9	0.8	5940	2.1	41.4	2.8	191
Chromium (VI)	<50	-	<50	-	<50	-	<50	-
Cobalt	1.6	2.6	2.5	569	1.6	7.3	1.5	24.0
Copper	9.5	73.2	1.3	1610	3.2	24.3	5.3	104
Lead	1.1	11.2	0.9	961	0.1	11.2	0.1	39.5
Manganese	265	394	3290	41500	1540	1650	1430	2290
Mercury	<0.2	<0.2	<0.2	6.1	<0.2	<0.2	<0.2	0.3
Nickel	22.3	33.0	5.9	1420	9.4	23.4	8.1	68.9
Selenium	1.2	1.7	0.6	86.3	0.7	1.1	<0.5	3.8
Vanadium	16.3	34.3	3.82	1930	5.49	46.0	8.44	164
Zinc	20.6	115	3.1	5420	21.8	69.5	15.5	214

Table 5: Concentrations of metals and metalloids (μg/l) in in filtered (F) and whole (W) wastewater samples of CN16003, CN16005, CN16007, CN16009.

Metals	CN16004	CN16006	CN16008
	Sediment		
Antimony	1.00	0.32	0.26
Arsenic	20.3	15.3	14.1
Beryllium	1.48	1.12	0.92
Cadmium	0.72	0.20	0.18
Chromium	250	43.2	38.7
Chromium (VI)	-	-	-
Cobalt	15.4	13.5	12.2
Copper	297	39.7	34.0
Lead	80.4	23.4	19.5
Manganese	1370	684	686
Mercury	0.20	0.12	0.12
Nickel	115	32.8	29.5
Selenium	2.28	2.24	2.00
Vanadium	179	45.6	44.2
Zinc	556	103	97.6

Table 6: Concentrations of metals and metalloids (mg/kg dry weight) in sediment samples of CN16004, CN16006, CN16008.

Organic chemical contaminants

Compound	Samples			
	CN16003	CN16005	CN16007	CN16009
	Wastewater			
Ethene, 1,1-dichloro-	N.D	<5	<5	<5
Carbon disulfide	<5	<5	<5	<5
Methane, dichloro-	<5	943	794	1000
ethene, 1,2-dichloro-, cis-	N.D	<5	N.D	<5
chloroform	20	43	51	65
Cyclohexane	69	<5	<5	6
Methane, tetrachloro-	N.D	N.D	<5	<5
benzene	N.D	166	175	279
Ethane, 1,2-dichloro-	34	710	1200	1630
ethene, trichloro-	N.D	<5	<5	<5
propane, 1,2-dichloro-	<5	<5	<5	<5
cyclohexane, methyl-	N.D	<5	<5	<5
toluene	<5	90	139	197
1,1,2-trichloroethane	N.D	<5	<5	<5
Ethene, tetrachloro-	<5	<5	<5	<5
benzene, chloro-	<5	101	69	90
benzene, ethyl-	7	7	<5	5
m- p-xylene	19	16	10	12
o--xylene	8	28	26	45
bromoform	N.D	N.D	<5	<5
benzene, isopropyl-	N.D	<5	<5	<5
1,3-dichlorobenzene	<5	13	15	20
1,4-dichlorobenzene	<5	<5	<5	<5
1,2-dichlorobenzene	<5	12	13	18
1,2,4-trichlorobenzene-	N.D	5	<5	5

Table 7. Quantification results for several VOCs identified in sample CN16003b, CN16005b, CN16007b & CN16009b. (unit: µg/L).

Note:

1. *N.D.: not detected in sample*
2. *In cases in which undiluted extracts gave results outside the calibration range, the results reported are those following dilution, though in these cases, reported results could be underestimates of the concentrations in the original samples as a result of loss of VOCs through volatilization during the dilution process*

Sample code	**CN16003**	**CN16004**	**CN16005**	**CN16006**	**CN16007**	**CN16008**	**CN16009**
Sample type	ww	sed	ww	sed	ww	sed	ww
Number compounds isolated	74	127	151	94	136	39	134
Number compounds identified	41	82	71	53	73	25	75
% of compounds identified	55%	65%	47%	56%	54%	64%	56%
	Number of compounds (and % of total identified)						
Chlorinated	28 (68%)	21 (26%)	38 (54%)	19 (36%)	33 (45%)	9 (36%)	34 (45%)
Brominated	nd	nd	1 (1%)	2 (4%)	1 (1%)	2 (8%)	1 (2%)
Fluorinated	nd	3 (4%)	4 (6%)	nd	4 (5%)	1 (4%)	6 (8%)
Mixed halogenated	nd	2 (2%)	2 (3%)	1 (2%)	5 (7%)	1 (4%)	4 (5%)
Organophosphorous	nd	nd	1 (1%)	1 (2%)	3 (4%)	1 (4%)	2 (3%)

Table 8: Summary of key organic groups contaminants isolated from wastewater (ww) and sediment (sed) samples collected at Lianyungang Chemical Industry Park and identified to a high degree of reliability.

Analysis of the sample of water from the small river and of those from the wastewater channel revealed the presence of complex mixtures of contaminants in each case, with between 74 and 151 individual compounds resolved per sample, and with roughly only half (47-56%) of those being identified in each case. Of those chemicals identified in the waters from the small river and waste channel, more than half were either chlorinated, brominated or fluorinated.

In the case of the river water (CN16003), 28 (68%) of the 41 compounds identified in the water were chlorinated compounds, including chlorinated nitrobenzene derivatives, chlorinated pyridines and chlorinated toluenes, along with a variety of more volatile compounds. The sample of sediment collected from the same location (CN16004) contained 21 chlorinated chemicals, as well as 3 fluorinated compounds and a further 2 mixed halogenated chemicals, all indicative of the presence of wastes from industrial processes using chlorine and fluorine chemistry. A complex mix of benzene derivatives and polycyclic aromatic hydrocarbons (PAHs) was also present. Particularly prominent in this sediment sample were residues of the mixed chlorinated and fluorinated pesticide *lambda* – Cyhalothrin, a synthetic pyrethroid insecticide no longer approved for use within the European Union.

Among the diverse range of organic chemicals found in this sediment sample were a number of other pesticides, including another synthetic pyrethroid (Bifenthrin), which is particularly toxic to aquatic life, the carbamate herbicide Benthiocarb (a cholinesterase inhibitor), the insecticide Buprofezin, the carboximide herbicide Diflufenican, the oxidiazole herbicide Oxadiazon and 2 isomers of the fungicide Difenoconazole. Residues of the persistent chlorinated pesticide, Hexachlorobenzene, were also found; this compound was formerly used as a fungicide, but banned from use globally as a Persistent Organic Pollutant (POP) under the Stockholm Convention. Also found were residues of the chlorinated pyrimidine compound Fenclorim, used to reduce the toxicity of certain herbicides to non-target crops. While it cannot be ruled out that some of these residues may have arisen from the application of pesticides in upstream agricultural areas, a local manufacturing source would seem to be a more likely source. This is further supported by the presence of a number of chlorinated chemicals with close structural similarities to some of the pesticide active ingredients found, including pentachlorobenzene, chlorinated pyridines and pyrimidine.

The chemical characteristics of the three samples of wastewater and two associated sediments collected at the northernmost end of the channel that crosses and runs perpendicular to the small river showed many similarities to those of the river water and sediment described above, perhaps unsurprisingly given the link between the two water systems and the nature of the industrial manufacturing operations reported to be ongoing in the area. All three water samples (CN16005, 7 & 9), collected form the same location but at different times, contained more than 130 individual organic chemicals, of which only around half could be identified, around one quarter were chlorinated and between 4 and 6 of which were fluorinated in each case. Between 1 and 3 organophosphorus chemicals were also found in these samples.

In each sample, a number of chlorinated benzenes, chlorinated benzenamines, chlorinated phenols and chlorinated benzeneacetonitriles were particularly evident. Among the largest peaks in each sample trace were the compounds 2-methylbenzyl cyanide, 3-trifluoromethylbenzeneacetonitrile, 2,4-dichloro benzeneacetonitrile and dichlorobenzenamine.

In addition, all three of these samples contained notably high concentrations of several volatile organic compounds, including benzene (166-279 ug/l), toluene (90-197 ug/l), dichloromethane (794-1000 ug/l) and 1,2-dichloroethane, otherwise known as ethylene dichloride (710-1630 ug/l). This last chemical, EDC, is most commonly manufactured as an intermediate in the manufacture of vinyl chloride monomer (VCM), which in turn is used to manufacture the plastic polyvinyl chloride (PVC). It is a known carcinogen, as well as being harmful by inhalation and in contact with skin.

Several pesticide residues were also in evidence in these three wastewater samples, including:

- the three herbicides Oxadiazon, Metribuzin and Molinate in sample CN16005,
- the carcinogenic organochlorine herbicide Butachlor, the organophosphate insecticide Malathion and the herbicide Triallate in CN16007 and
- the insecticide Malathion and the herbicides Metribuzin and Triallate in CN16009.

The two sediment samples collected from the same location as these water samples (CN16006 and 8) also contained complex mixtures of hazardous organic chemical contaminants, including PAH derivatives, chlorinated and other substituted benzenes, chlorinated phenols and a number of pesticides. These pesticides included:

- in sample CN16006, the chlorinated synthetic pyrethroid insecticide *lambda*-Cyhalothrin, the fluorinated and chlorinated pyrethroid Bifenthrin, the insecticides Buprofezin and Chlorpyrifos, the herbicide Oxadiazon and the carbamate herbicide Triallate and
- in sample CN16008, the triazole fungicides Difenoconazole and Propiconazole, in addition to residues of Bifenthrin, Chlorpyrifos, Oxadiazon and Triallate.

Sample CN16006 was also found to contain residues of the persistent, chlorinated industrial chemical hexachlorobutadiene, manufactured as a solvent, but also known to be generated as a by-product of industrial chlorine chemistry.

Confirmatory pesticide analysis

All the samples were analysed in triplicate. Note that, although the analyses conducted were quantitative, concentrations reported below should be considered to be approximate only because it was not possible in this case to control fully for differences in the initial volume/weight of samples.

The 274 pesticide standard mix used for confirmatory analysis by LC-MS does not include residues for all of those pesticides identified through GC-MS analysis of the same samples, with the absence of lambda-cyhalothrin (which GC-MS analysis indicated was highly abundant in some samples) of particular note. Nevertheless, for those pesticides identified by GC-MS analysis that are included in the 274 residue mix employed in the confirmatory LC-MS analysis, there was relatively good overlap between the two methods, confirming e.g. the presence of Buprofezin, Benthiocarb and Difenoconazole in sediment sample CN16004, of Buprofezin and Chlorpyrifos (ethyl) in sediment CN16006 and of Chlorpyrifos (ethyl) in sediment CN16008. LC-MS analysis also confirmed the presence of residues of Butachlor in the water sample CN16007.

LC-MS analysis identified the presence of Buprofezin also in water sample CN16007 and sediment CN16008, and of Chlorpyrifos (ethyl) in sediment CN16004, residues that were not detectable in the GC-MS analysis of the same samples. Conversely, although residues of Difenoconazole and Propiconazole were identified in sediment CN16008 by GC-MS analysis, the presence of these compounds could not be confirmed through LC-MS analysis of the same samples. The reasons for these discrepancies are not currently known, though they could relate to differences in sensitivity of the two instruments, matrix effects and interferences, or a combination of these factors.

Prosulfocarb, was found in all the water samples from this site (CN16003, 05, 07 & 09), with the highest concentration of around 50ng/ml (ppb) in sample CN16005. This pesticide was also prominent in all of the sediment samples (CN16004, 06 & 08), with the highest concentration, in the range of 5900 ng/g (ppm), in sediment sample CN16004. In addition to the Buprofezin and Chlorpyrifos (ethyl) mentioned above, other pesticides were identified in these sediment samples, including Etofenprox, Hexythiazox and Fenazaquin, at maximum concentrations of around 60, 13 and 2 ng/g, respectively.

Conclusions

Taken together, the results of the four water and three sediment samples from the two different locations on the Lianyungang Chemical Industrial Park provide evidence for heavy contamination of both the small river and the perpendicular waste channel with complex mixtures of hazardous chemicals, many of them halogenated (chlorinated or fluorinated) and some highly persistent once they reach the environment. The mixtures of contaminants present include many chemicals typical of chlorinated and fluorinated chemistry used in the manufacture of pesticides, pharmaceuticals and specialist industrial chemicals, as well as of vinyl chloride (VCM) used in the production of PVC plastic. Residues of a total of 15 different pesticides, including herbicides, fungicides and insecticides, were found in total in this water system, including hexachlorobenzene, butachlor and chlorpyrifos. The presence of diverse organic compounds in the water, as well as the sediment, including high concentrations of a number of volatile organic compounds, strongly indicates substantial ongoing discharges of contaminated industrial wastewaters to these channels, rather than just reflecting historic inputs.

As both the small river and the perpendicular wastewater channel both connect to the GuanHe River and, ultimately, to the Yellow Sea, the presence in both water and sediments of such complex mixtures of toxic organic compounds, including a number of POPs and carcinogenic chemicals, is clearly cause for high concern in terms of both environmental pollution and public health. Despite being established less than 15 years ago, this industrial park has evidently already created a hotspot of toxic chemical pollution, and one that may be expected to continue to worsen over time unless action can be taken to prevent further discharges of hazardous wastes to the channels and to contain and clean-up the contaminated sediments. In the meantime, any attempt to assess the precise health and environmental risks associated with exposure to the chemicals in the water or sediment, or their subsequent contamination of fish and other river and seafood that may be harvested downstream, will clearly remain extremely difficult, if not impossible, when faced with such complex mixtures of chemical pollutants, especially given that chemicals can have different toxic effects singly and in combination and when a high proportion of which simply cannot be readily identified.

For more information please contact:

Melissa Wang, Iryna Labunska, Kevin Brigden, or David Santillo

References

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Appendix 1: Details of methodologies

Analysis for Volatile Organic Compounds (VOCs)

Methods

VOCs were analysed using an Agilent 7890B gas chromatograph with a Restek Rxi-624Sil column (30m, 0.25mm ID, 1.4µm film thickness) connected to an Agilent 7697A Headspace Sampler and linked to an Agilent 5977A MSD operated in EI mode. The GC oven temperature program included an initial temperature of $43^{0}C$ (held for 4min), rising to $55^{0}C$ at $5^{0}C/min$, and then to $210^{0}C$ at $15^{0}C/min$ (held for 2.5min). The carrier gas was helium, supplied at 1.5 ml/min.

From each sample:

1) 10ml portions of the sample was transferred into a 20ml headspace vial containing 3g of anhydrous sodium sulfate (analytical reagent grade). This sub-sample was analysed using GC-MS in total ion monitoring (SCAN) mode to identify as many of the volatile organic compounds as possible. Identification of compounds was carried out by matching spectra against the Wiley7N Library, employing expert judgment in order to avoid misidentifications. In addition, this sub-sample was also analysed with the GC-MS in selective ion monitoring (SIM) mode, in order to match the GC-MS spectra obtained against those of mixed standard preparations containing a range of volatile aromatic organic compounds and halogenated alkanes.

2) Two further 10ml portions were sub-sampled into a 20ml headspace vials containing 3g of anhydrous sodium sulfate (analytical reagent grade) and internal standards (Methane, bromochloro-, 1,2-Dichloroethane-d4, Toluene-d8, Chlorobenzene-d5). These sub-samples were subject to a duplicate quantitative analysis for selected halogenated VOCs (chlorinated, brominated and mixed), which had been detected in the samples through screening. Quantification was performed in SIM mode using 4 point calibration method; several calibration ranges were established to match as close as possible concentrations in the samples. If the concentration of a compound in a particular sample was over the calibration range, dilution was performed in order achieve reliable quantification

3) The remaining 20 ml of samples was stored in 20ml headspace vials as a back-up sub-sample.

Halogenated VOCs quantified in the water samples with method detection limit (MDL) & limits of quantification (LOQ) are presented in Table 9a and 9-b below.

Quality control

Method detection limits (MDL) were obtained using data of 7 replicas of standard solution analysis. A number of blanks of laboratory air capped at the time that sub-sampling had taken place were also analysed, alongside samples of the ultra pure reagent water which was used for the preparation of standard calibration solutions. The initial calibration curve for each compound of interest was verified immediately prior to sample analysis by analyzing a calibration standard at a concentration near the midpoint concentration for the calibration range of the GC-MS.

Compound	MDL (µg/L)	LOQ (µg/L)
Ethene, 1,1-dichloro-	1	2
Methane, dichloro-	0.3	2
Ethane, 1,1-dichloro-	0.5	2
Ethene, 1,2-dichloro-, cis-	0.5	2
Chloroform	0.5	2
Methane, tetrachloro-	2	2
Benzene	0.6	2
Ethane, 1,2-dichloro-	0.3	2
Ethene, trichloro-	1	2
Propane, 1,2-dichloro-	0.4	2
Methane, bromodichloro-	0.4	2
Toluene	0.3	2
1,1,2-trichloroethane	0.5	2
Ethene, tetrachloro-	2	2
Methane, dibromochloro-	0.3	2
Benzene, chloro-	0.3	2
o-xylene	0.9	2
Styrene	0.7	2
Bromoform	0.4	2
1,2-dichlorobenzene	0.5	2
1,2,4-trichlorobenzene	0.9	2

Table 9-a. Method detection limits (MDL) and limits of quantification (LOQ) for sample CN16001 and CN16002

Analysis for extractable organic compounds

Preparation

For water samples, 20 µg of deuterated naphthalene was added as an Internal Standard (IS) to each portion of sample that was subject to extraction. Water samples (400ml) were prepared using solid phase extraction technique with Dionex AutoTrace workstation, eluting with ethyl acetate followed by a mixture of pentane and toluene (95:5). Obtained extracts were concentrated to a volume of 3ml with a stream of clean nitrogen and cleaned up prior to analysis.

For solid samples, approximately 10 g of each sample (wet weight) was extracted employing an Accelerated Solvent Extraction (ASE) technique, using a Dionex ASE-350, with a mixture of pentane and acetone in a ratio of 3:1, and at a temperature of 100°C. Obtained extracts were concentrated to a volume of 3ml with a stream of clean nitrogen and cleaned up prior to analysis.

Compound	MDL (µg/L)	LOQ (µg/L)
Ethene, 1,1-dichloro-	3	5
Carbon disulfide	3	5
Methane, dichloro-	1	5
ethene, 1,2-dichloro-, cis-	1	5
chloroform	1	5
Cyclohexane	2	5
Methane, tetrachloro-	2	5
benzene	2	5
Ethane, 1,2-dichloro-	1	5
ethene, trichloro-	2	5
propane, 1,2-dichloro-	1	5
cyclohexane, methyl-	2	5
toluene	2	5
1,1,2-trichloroethane	1	5
Ethene, tetrachloro-	2	5
benzene, chloro-	1	5
benzene, ethyl-	1	5
m- p-xylene	2	10
o--xylene	1	5
bromoform	1	5
benzene, isopropyl-	1	5
1,3-dichlorobenzene	1	5
1,4-dichlorobenzene	1	5
1,2-dichlorobenzene	1	5
1,2,4-trichlorobenzene-	1	5

Table 9-b. Method detection limits (MDL) and limits of quantification (LOQ) for sample CN16003, CN16005, CN16007, CN16009 .

For the clean-up stage, each extract was shaken with 3ml isopropyl alcohol and 3ml TBA-reagent (mixture of 3% tetrabutylammonium hydrogen sulphate and 20% sodium sulphite in deionised water) and left to stand until the aqueous and organic phases had separated. The pentane phase was collected and eluted through a Florisil column, using a 95:5 pentane:toluene mixed eluent, and the cleaned extract concentrated to a final volume of 1ml as before. 20 µg of bromonaphthalene was added to each extract as a second IS prior to GC-MS analysis.

Analysis of semi-volatile organic chemicals

For the total organic compounds screening, samples were analysed using an Agilent 7890B GC with Restek Rxi-17Sil column (30m, 0.25mm ID, 0.25 µm film thickness) linked to an Agilent 5977A MSD operated in EI mode and interfaced with an Agilent Enhanced Chem Station data system. The GC oven temperature program employed was as follows: an initial temperature of 40^{0}C, raised to 260^{0}C at 10^{0}C/min, then to 295^{0}C at 50^{0}C/min (held for 15 min), and finally to 320^{0}C at 50^{0}C/min (held for 12 min). The carrier gas was helium, supplied at 1ml/min. Identification of compounds was carried out by matching spectra against both the Wiley 10N11 and Pesticides Libraries, using expert judgment as necessary in order to avoid misidentifications. Additionally, both the spectra and retention times of compounds isolated from the samples were matched against those obtained during GC-MS analysis of standard mixtures containing a range of chlorinated benzenes, phenols and pesticides, polychlorinated biphenyls (PCBs), phthalates, polycyclic aromatic hydrocarbons (PAHs) and aliphatic hydrocarbons.

Quality control

A number of extraction and solvent blanks were also analysed to ensure the detection of any possible contamination resulting from sample handling in the laboratory. Any background contaminants detected in blanks are subtracted from the chromatograms obtained for the samples before mass spectra are interpreted.

Analysis for pesticide residues

A 0.4 mL fraction of the GC analytical extracts obtained from each sample was blown down to dryness and reconstituted in 1 mL of MeOH before injection in the LC-MS system.

1 µL of the final extract of each sample was injected in a Dionex UltiMate 3000 LC system, Thermo Scientific (Hemel Hempstead, UK), furnished with a C18 Accucore aQ (100 x 2.1 mm; 2.6 µm) column. The mobile phase flowed at 0.3 mL/min with a gradient composition result of the mixture of A: water (2% MeOH, 0.1% formic acid and 5 mm ammonium formate) and B: MeOH (2% water, 0.1% formic acid and 5 mm ammonium formate) as follows: 0 min 2% B, 0.5 min 2% B, 7 min 70% B, 9-12 min 100% B.

The LC column flow was directed to an Orbitrap Q Exactive Focus, Thermo Scientific, equipped with a HESI-II source, a quadrupole, an HCD collision cell and the Orbitrap high-resolution mass analyser, where mass spectra were acquired in Full Scan and in MS^2, simultaneously. MS and MS/MS data was extracted with a small mass error window of 5 ppm, given the high resolution of the acquisition process, ensuring the unequivocal detection and quantification of the compounds.

As in the analysis done by GC-MS, solvent blanks were also analysed to ensure the detection of any possible contamination resulting from sample handling in the laboratory, confirming the absence of such interferences in these analyses

Analysis for metals

Preparation

Water samples: To obtain total metal concentrations, a representative portion of each whole water sample was acidified by the addition of concentrated nitric acid to give a final concentration of 5% v/v. In addition, for samples from Area 2, a portion of each whole sample was filtered through a 0.45 micron filter and then acidified in the same way to enable determination of dissolved metal concentrations. 25 ml of each acidified sample was digested firstly overnight at room temperature, then using microwave-assisted digestion with a CEM MARS Xpress system, with a temperature ramp to 180°C over 15 minutes followed by holding at 180°C for a further 15 minutes. Cooled digests were filtered and made up to 25 ml with deionised water.

Sediment samples: A representative portion of each sample was dried at 30°C to constant weight, homogenised, and then ground to a powder using a pestle and mortar. Approximately 0.25 g of the dried and ground sample was digested with 0.5 ml concentrated hydrochloric acid and 5 ml concentrated nitric acid, firstly overnight at room temperature, then using microwave-assisted digestion with a CEM MARS Xpress system, with a temperature ramp to 180°C over 20 minutes followed by holding at 180°C for a further 20 minutes. Following cooling, the digest was filtered and made up to 25 ml with deionised water.

Analysis

Prepared sample digests were analysed by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) using an Agilent 7900 Spectrometer utilizing a collision cell with helium as the collision gas to minimize polyatomic interferences. Multi-element standards, matrix matched to the samples, at concentrations of 1, 10, 100 and 1000 μg/l respectively, other than for mercury (1, 2, 5, 20 μg/l respectively) were used for instrument calibration. Analysis employed in-line addition of an internal standard mix at 100 μg/l (Scandium, Germanium, Yttrium, Indium and Terbium).

Concentrations of chromium (VI) were determined colorimetric for each water sample following filtration where necessary. 0.5 ml of a 1,5-diphenylcarbazide testing solution (freshly prepared from 0.4 g of 1,5-diphenylcarbazide, 20 ml acetone, 20 ml ethanol, 20 ml orthophosphoric acid solution and 20 ml of demineralised water) was added to 9.5 ml of each filtered sample. The solution was mixed and let to stand for 10 minutes to allow full colour development. Concentrations were determined using a spectrophotometer at 540 nm, correcting with a blank prepared from deionised water, using standards freshly prepared from potassium dichromate at concentrations of 0, 50, 100 and 200 μg/l respectively. Any sample exceeding the calibration range was diluted accordingly, in duplicate, and re-analysed.

Quality control

For each batch of water samples, one water sample was prepared for ICP analysis in duplicate and analysed to verify method reproducibility. Similarly, one sediment sample was prepared in duplicate. In addition, a blank sample was prepared and digested each of the water and sediment procedures. For water samples, two mixed metal quality control solution of 80 and 800 μg/l for each metal, other than mercury at 4 and 16 μg/l, were digested and analysed. For sediment samples, a certified reference material (CRM) sample was prepared in an identical manner; GBW07311, stream sediment certified by the China National Analysis Centre for Iron and Steel, Beijing, China. All control samples were prepared in an identical manor to the samples.

Calibration of the ICP-MS was validated by the use of quality control standards at 80 μg/l and 800 μg/l (4 μg/l and 16 μg/l for mercury) prepared in an identical manner but from different reagent stocks to the instrument calibration standards.

For chromium (VI) determination, one sample was prepared and analysed in duplicate, along with a blank sample, and a quality control solution of 100 μg/l prepared in an identical manor to the samples.

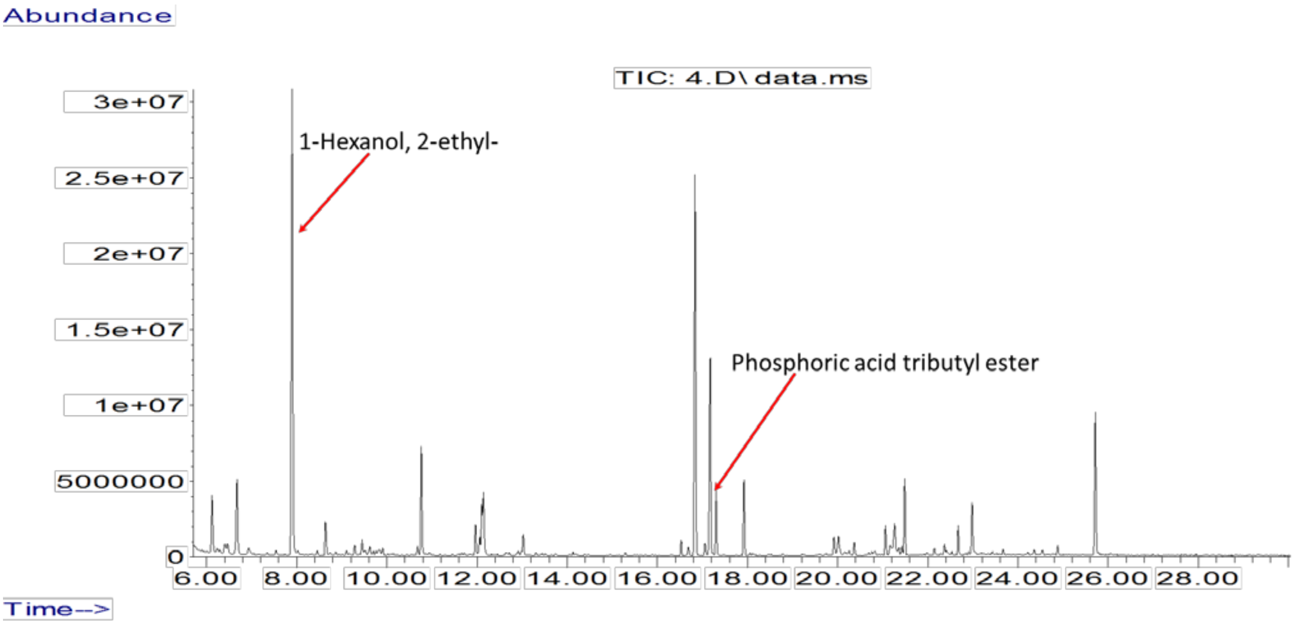
Further details of the methods employed can be provided on request.

Appendix 2: Detailed organic chemical analytical screening data

Detailed screening data arising from GC-MS analysis of each of the samples are presented below. These data list those semi-volatile organic compounds identified following solvent extraction, and for wastewater samples also included volatile organic compounds (VOCs) identified through separate headspace GC-MS analysis of sub-samples. Only those substances identified to greater than 90% quality match (following verification by expert interpretation) are listed here.

CN16001 (wastewater)

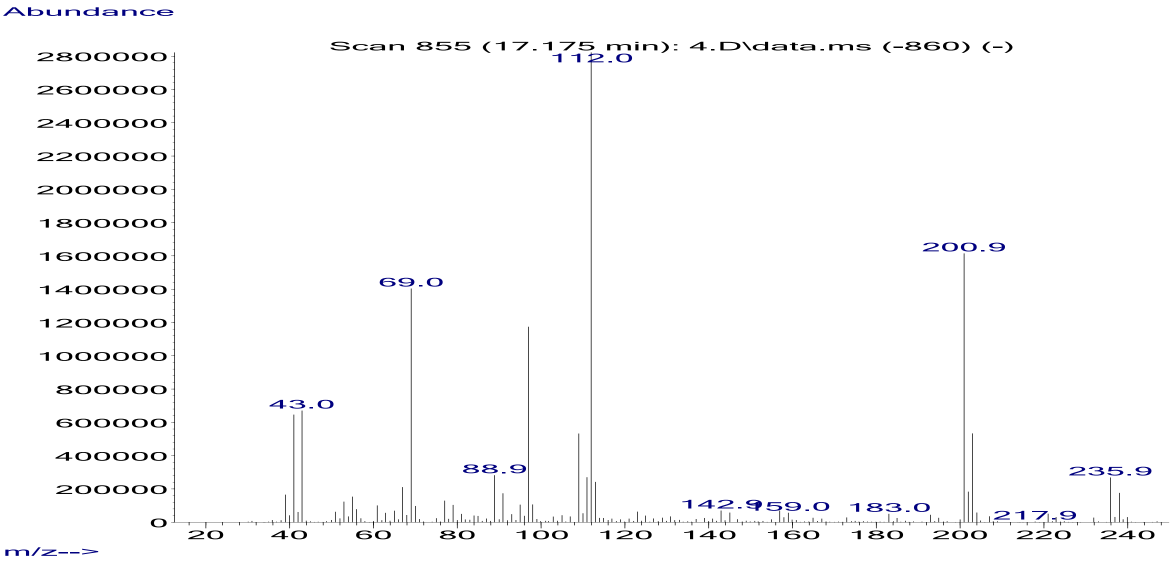
Number of semi-volatile compounds isolated: 43

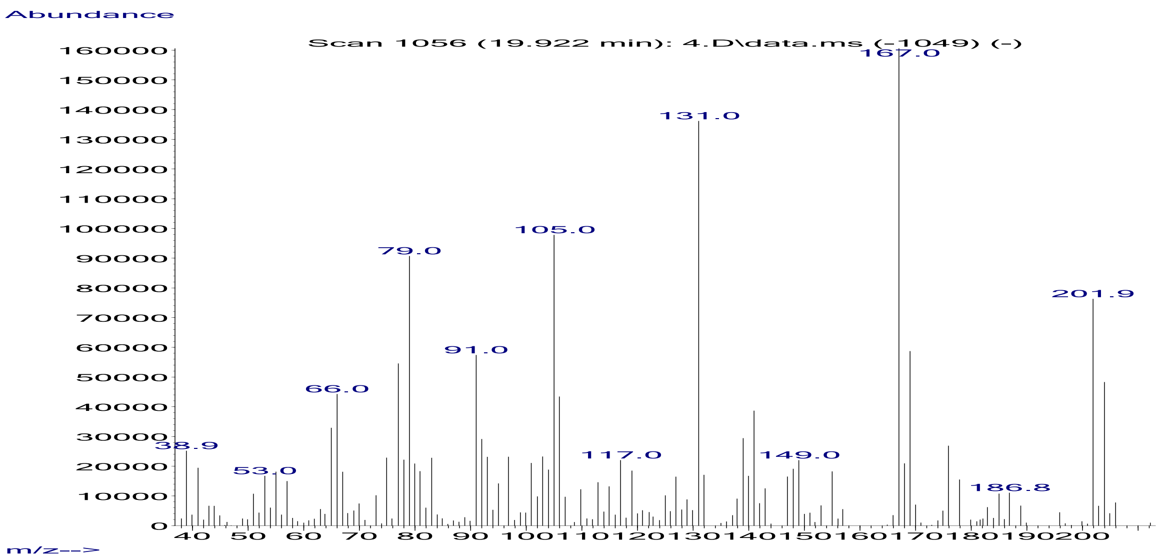


Compounds identified to better than 90%:

CAS#	Name
000084-66-2	1,2-Benzenedicarboxylic acid, diethyl ester
000084-74-2	1,2-Benzenedicarboxylic acid, dibutyl ester
000084-69-5	1,2-Benzenedicarboxylic acid, diisobutyl ester
000131-11-3	1,2-Benzenedicarboxylic acid, dimethyl ester
000104-76-7	1-Hexanol, 2-ethyl-
000140-29-4	Acetonitrile, phenyl-
003722-05-2	Cyclopropane, 1,1-dichloro-2-(chloromethyl)-
000126-73-8	Phosphoric acid tributyl ester

Note: chromatogram contained several compounds that cannot be identified to any degree of reliability. The fragmentation of these compounds is presented below.





Number of <u>volatile</u> compounds isolated: **43**

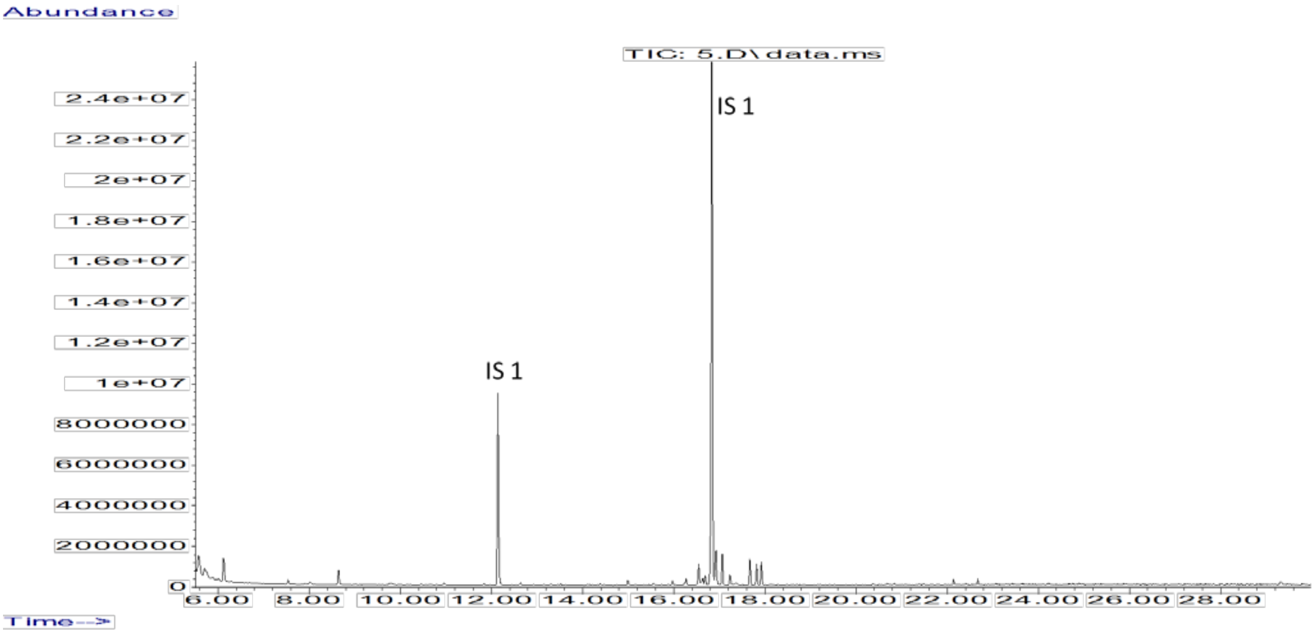
Compounds identified to better than 90%:

CAS#	**Name**
023378-11-2	1-Butene, 1-chloro-2-methyl-
023010-00-6	1-Butene, 1-chloro-3-methyl-
017773-64-7	1-Butene, 2-chloro-3-methyl-
000104-76-7	1-Hexanol, 2-ethyl-
000513-37-1	1-Propene, 1-chloro-2-methyl-
017773-65-8	2-Butene, 2-chloro-3-methyl-
000079-00-5	Ethane, 1,1,2-trichloro-
000107-06-2	Ethane, 1,2-dichloro-
000075-35-4	Ethene, 1,1-dichloro-
000079-01-6	Ethene, trichloro-

000075-27-4	Methane, bromodichloro-
000124-48-1	Methane, dibromochloro-
000075-25-2	Bromoform
000067-66-3	Chloroform
000078-82-0	Propanenitrile, 2-methyl-
000100-42-5	Styrene
000108-90-7	Benzene, chloro- (SIM)
000095-50-1	Benzene, 1,2-dichloro- (SIM)
000120-82-1	Benzene, 1,2,4-trichloro- (SIM)
000075-34-3	Ethane, 1,1-dichloro- (SIM)
000127-18-4	Ethene, tetrachloro- (SIM)
000075-09-2	Methane, dichloro- (SIM)
000056-23-5	Methane, tetrachloro- (SIM)
000078-87-5	Propane, 1,2-dichloro- (SIM)
000108-88-3	Toluene (SIM)

CN16002 (wastewater)

Number of semi-volatile compounds isolated: 15



Compounds identified to better than 90%:

CAS#	Name
000084-66-2	1,2-Benzenedicarboxylic acid, diethyl ester
019814-71-2	Benzene, 1,1'-oxybis[3-methyl-
004731-34-4	Benzene, 1-methyl-2-(2-methylphenoxy)-
003402-72-0	Benzene, 1-methyl-2-(4-methylphenoxy)-
051801-69-5	Benzene, 1-methyl-3-(4-methylphenoxy)-

Number of volatile compounds isolated: 18

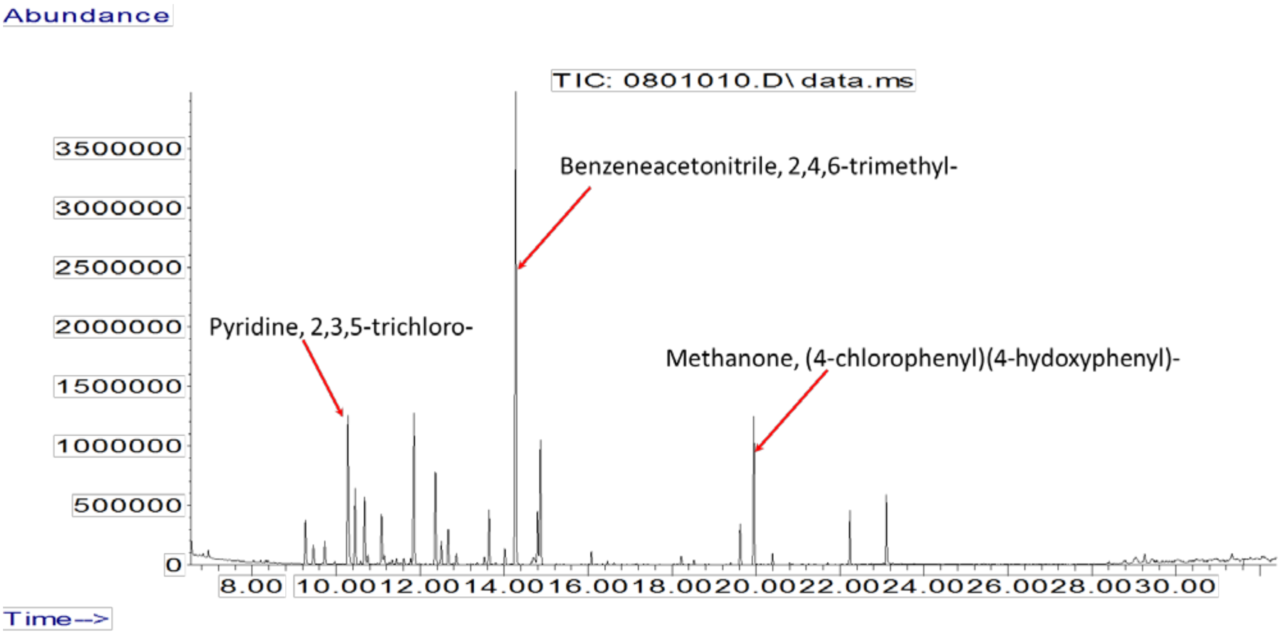
Compounds identified to better than 90%:

CAS#	Name
000078-78-4	Butane, 2-methyl-
000079-00-5	Ethane, 1,1,2-trichloro-
000075-35-4	Ethene, 1,1-dichloro-
000079-01-6	Ethene, trichloro-
000067-66-3	Chlorofom
000071-43-2	Benzene (SIM)
000075-34-3	Ethane, 1,1-dichloro- (SIM)
000107-06-2	Ethane, 1,2-dichloro- (SIM)
000156-59-2	Ethene, 1,2-dichloro-, cis- (SIM)
000127-18-4	Ethene, tetrachloro- (SIM)
000075-27-4	Methane, bromodichloro- (SIM)
000124-48-1	Methane, dibromochloro- (SIM)
000075-25-2	Bromoform (SIM)
000056-23-5	Methane, tetrachloro- (SIM)
000100-42-5	Styrene (SIM)

000108-88-3	Toluene (SIM)
000095-47-6	o-Xylene (SIM)

CN16003 (wastewater)

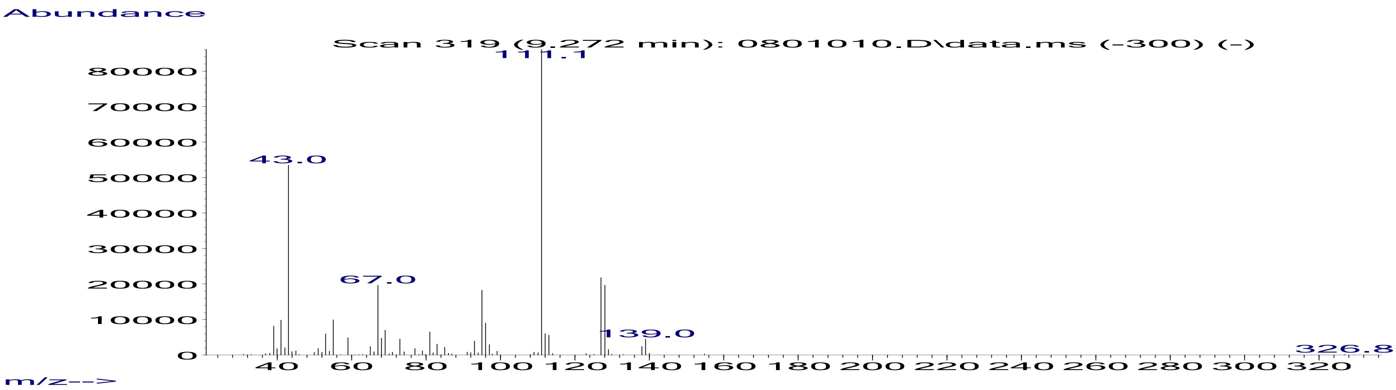
Number of semi-volatile compounds isolated: 43



Compounds identified to better than 90%:

CAS#	Name
001477-42-5	2-Benzothiazolamine, 4-methyl-
028249-77-6	Benthiocarb
000090-04-0	Benzenamine, 2-methoxy-
000000-00-0	Benzenamine, chloro-, 3 isomers
000088-73-3	Benzene, 1-chloro-2-nitro-
000100-00-5	Benzene, 1-chloro-4-nitro-
000100-29-8	Benzene, 1-ethoxy-4-nitro-
000091-23-6	Benzene, 1-methoxy-2-nitro-
000100-17-4	Benzene, 1-methoxy-4-nitro-
034688-71-6	Benzeneacetonitrile, 2,4,6-trimethyl-
002856-63-5	Benzeneacetonitrile, 2-chloro-
004170-90-5	Benzenemethanol, 2,4,6-trimethyl-
000090-98-2	Benzophenone, 4,4'-dichloro-
000084-66-2	Diethyl phthalate
042019-78-3	Methanone, (4-chlorophenyl)(4-hydoxyphenyl)-
019666-30-9	Oxadiazon
002402-79-1	Pyridine, 2,3,5,6-tetrachloro-
022963-62-8	Pyridine, 2,3,5,6-tetrachloro-4-methylthio-
016063-70-0	Pyridine, 2,3,5-trichloro-
002176-62-7	Pyridine, pentachloro-

Note: chromatogram contained several compounds that cannot be identified to any degree of reliability. The fragmentation of these compounds is presented below.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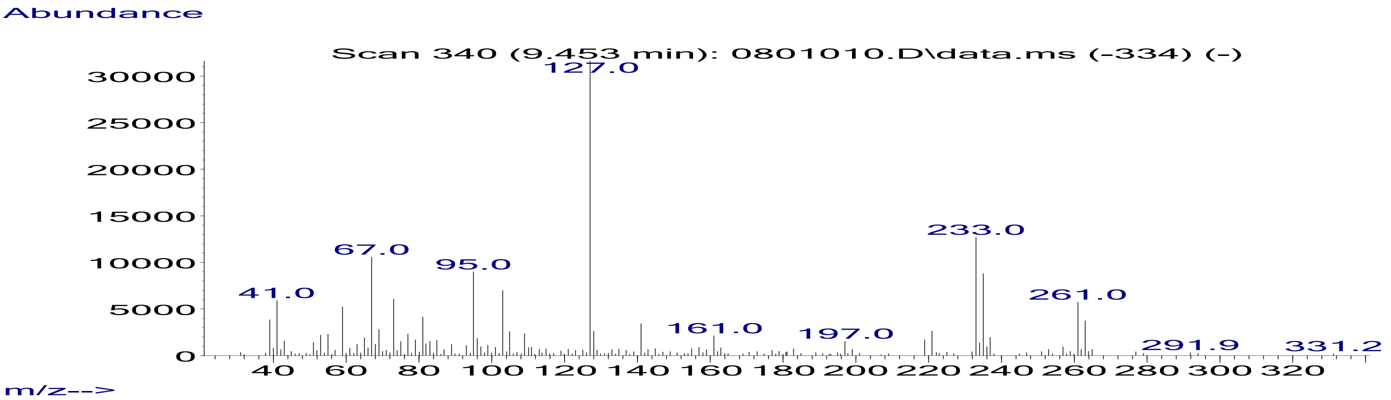
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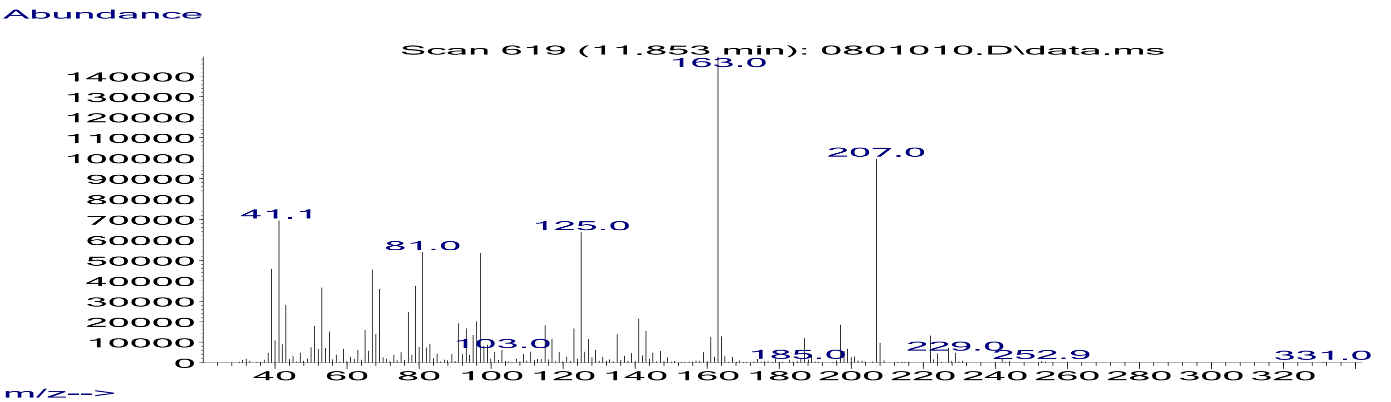
331.2
m/z-->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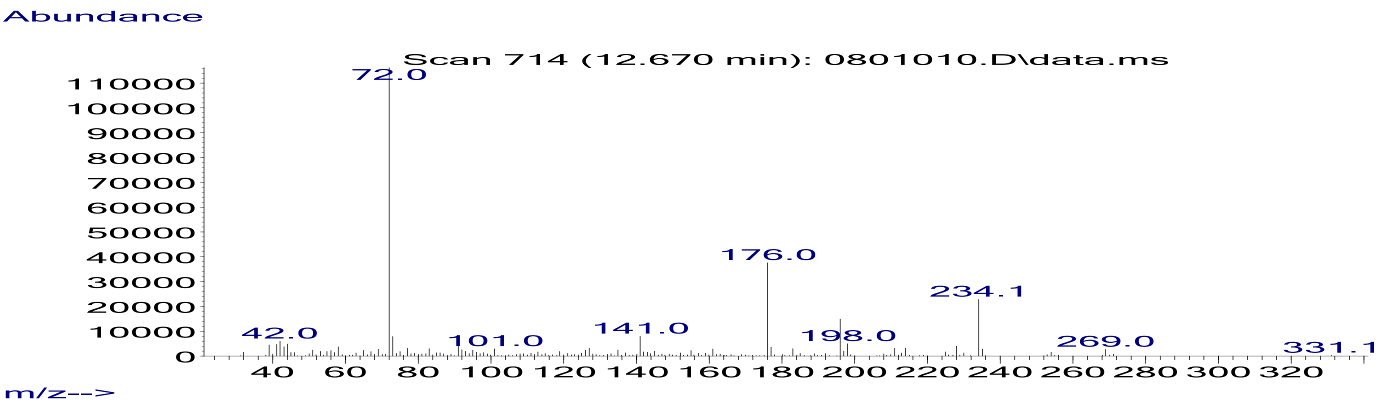
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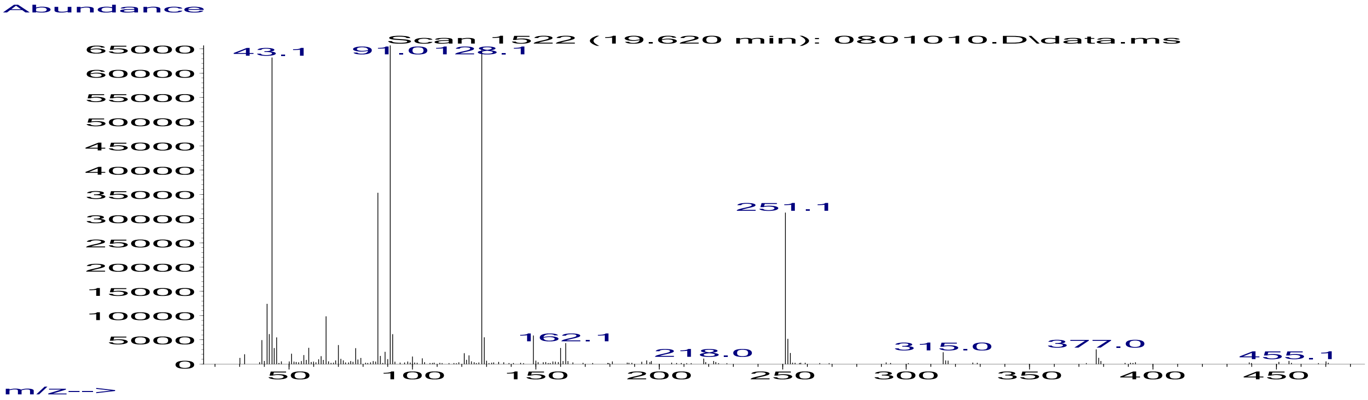
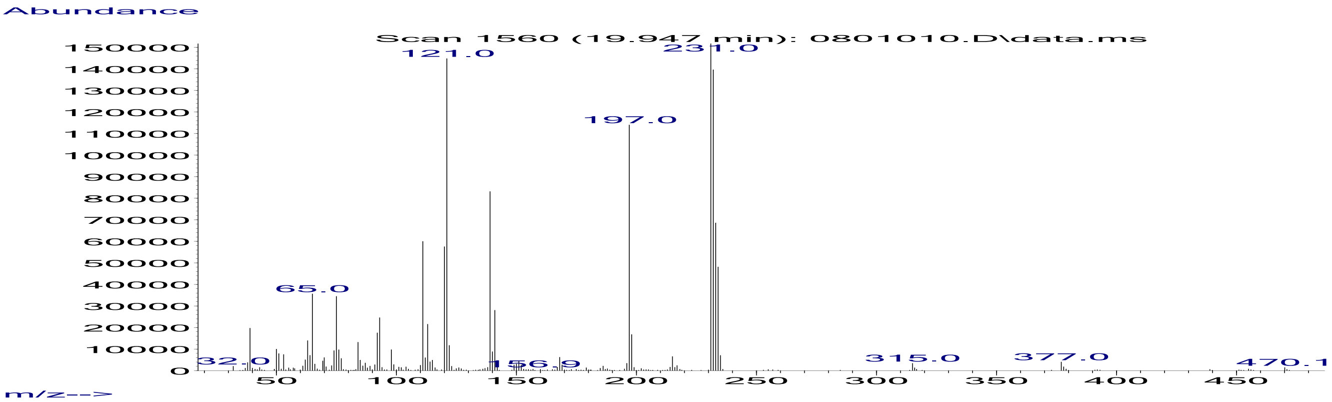
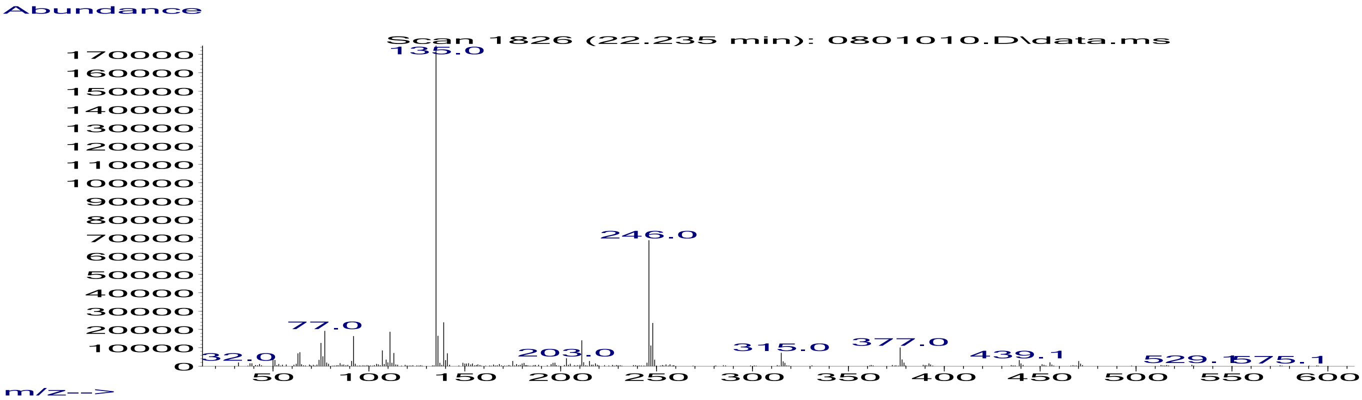






Number of volatile compounds isolated: **31**

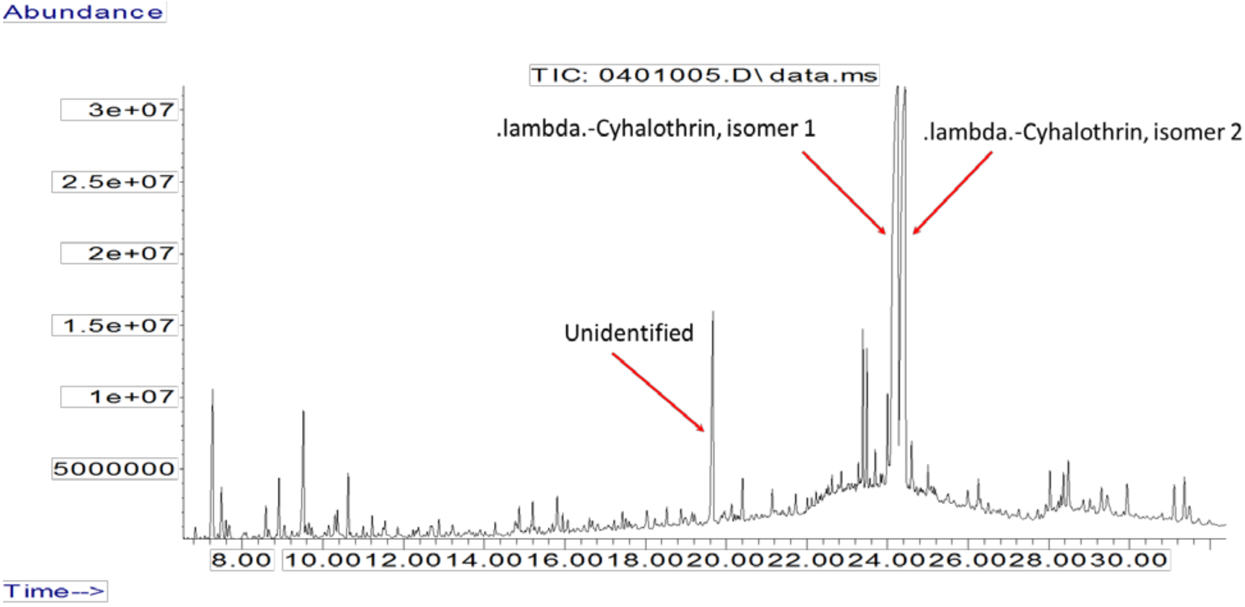
Compounds identified to better than 90%:

CAS#	**Name**
000095-50-1	Benzene, 1,2-dichloro-
000106-46-7	Benzene, 1,4-dichloro-
000100-41-4	Benzene, ethyl-
000000-00-0	Benzene, trimethyl-
000000-00-0	Chloroaniline
000067-66-3	Chloroform

000110-82-7	Cyclohexane
000107-06-2	Ethane, 1,2-dichloro-
000000-00-0	m-/p-Xylene
000095-47-6	o-Xylene
000078-87-5	Propane, 1,2-dichloro-
000000-00-0	Toluene, chloro- (2 isomers)
000000-00-0	Toluene, dichloro-
000000-00-0	Toluene, tert-butyl-
000000-00-0	Trichloropyridine
000108-90-7	Benzene, chloro- (SIM)
000541-73-1	Benzene, 1,3-dichloro- (SIM)
000075-15-0	Carbon disulfide (SIM)
000127-18-4	Ethene, tetrachloro- (SIM)
000075-09-2	Methane, dichloro- (SIM)
000108-88-3	Toluene (SIM)

CN16004 (Sediment)

Number of compounds isolated: 127

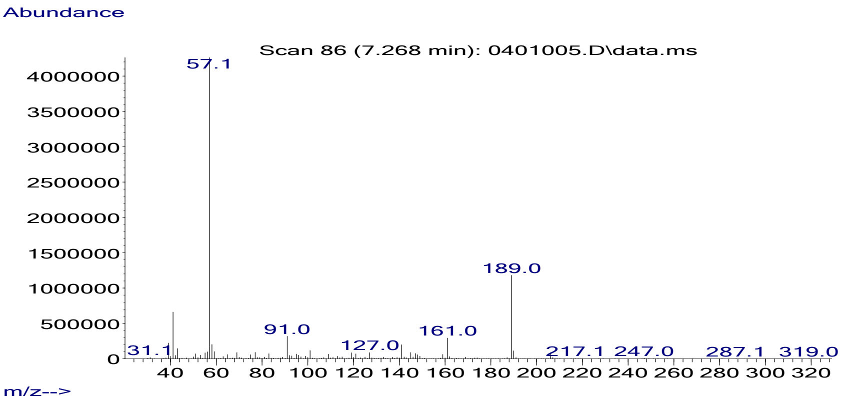


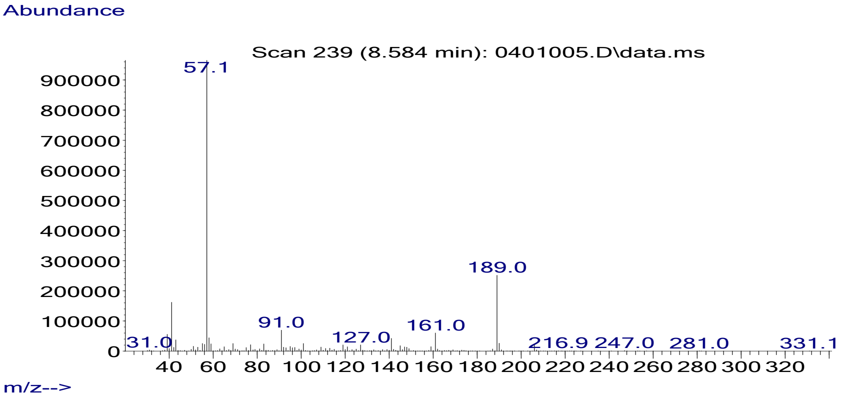
Compounds identified to better than 90%:

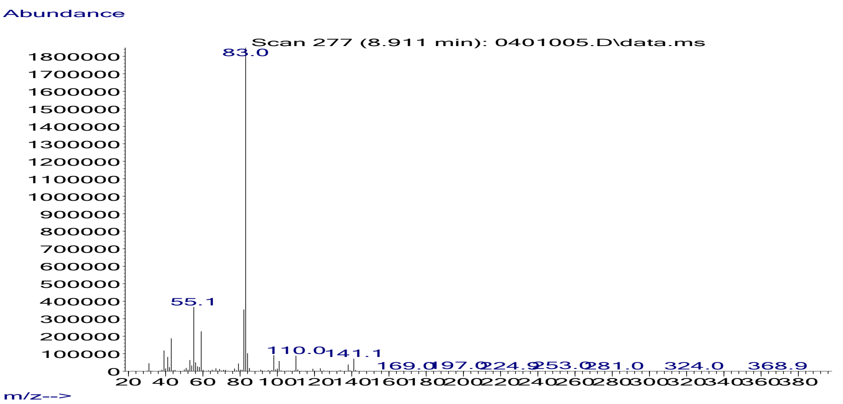
CAS#	Name
91465-08-6	.lambda.-Cyhalothrin, 2 isomers
000605-39-0	1,1'-Biphenyl, 2,2'-dimethyl-
6308-02-7	1,1,4,4-Tetramethyl-2-tetralone
000117-81-7	1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester
000084-69-5	1,2-Benzenedicarboxylic acid, diisobutyl ester
001599-67-3	1-Docosene
015220-85-6	1-Propene, 2-methyl-, tetramer
163064-79-7	1H-Indole, 2-cyclopropyl-3-phenyl-
100813-60-3	1H-Isoindole, 3-methoxy-4,7-dimethyl-
003075-84-1	2,2',5,5'-Tetramethyl-1,1'-biphenyl-
034688-71-6	2,4,6-Trimethylbenzyl cyanide
000719-22-2	2,5-Cyclohexadiene-1,4-dione, 2,6-bis(1,1-dimethylethyl)-
000504-20-1	2,5-Heptadien-4-one, 2, 6-dimethyl-
001477-42-5	2-Benzothiazolamine, 4-methyl-
053584-60-4	28-nor-17.alpha.(h)-Hopane
24279-39-8	4-Amino-3,5-dichlorobenzotrifluoride
000080-97-7	5.alpha.-Cholestan-3.beta.-ol
000084-65-1	9,10-Anthracenedione
000082-45-1	9,10-Anthracenedione, 1-amino-
000086-73-7	9H-Fluorene
000120-12-7	Anthracene
028249-77-6	Benthiocarb
039515-51-0	Benzaldehyde, 3-phenoxy-
000101-61-1	Benzenamine, 4,4'-methylenebis[N,N-dimethyl-
000095-79-4	Benzenamine, 5-chloro-2-methyl-
000000-00-0	Benzenamine, chloro-, 2isomers
000000-00-0	Benzenamine, dichloro-

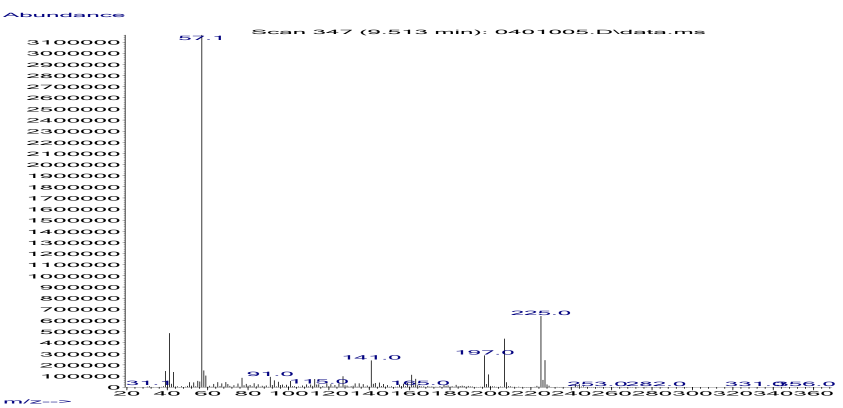
000121-69-7	Benzenamine, N,N-dimethyl-
019814-71-2	Benzene, 1,1'-oxybis[3-methyl-
001579-40-4	Benzene, 1,1'-oxybis[4-methyl-
002444-89-5	Benzene, 1,1'-oxybis[4-chloro-
000120-82-1	Benzene, 1,2,4-trichloro-
000095-50-1	Benzene, 1,2-dichloro-
006623-59-2	Benzene, 1,2-dichloro-4-ethyl-
001075-38-3	Benzene, 1-(1,1-dimethylethyl)-3-methyl-
000098-51-1	Benzene, 1-(1,1-dimethylethyl)-4-methyl-
000100-17-4	Benzene, 1-methoxy-4-nitro-
003402-72-0	Benzene, 1-methyl-2-(4-methylphenoxy)-
051801-69-5	Benzene, 1-methyl-3-(4-methylphenoxy)-
000615-60-1	Benzene, 4-chloro-1,2-dimethyl-
000118-74-1	Benzene, hexachloro-
000608-93-5	Benzene, pentachloro-
000700-12-9	Benzene, pentamethyl-
000000-00-0	Benzofuran, 2,2-dimethyl-5-t-butyl-2,3-dihydro-
082657-04-3	Bifenthrin
069327-76-0	Buprofezin
000057-88-5	Cholest-5-en-3-ol (3.beta.)-
113845-28-6	Cholest-5-en-3-ol, 23-ethyl-
119446-68-3	Difenoconazole, 2 isomers
083164-33-4	Diflufenican
003740-92-9	Fenclorim
000206-44-0	Fluoranthene
000593-49-7	Heptacosane
000629-78-7	Heptadecane
000000-00-0	Indole, 3-[2-(2-methylphenyl)ethenyl]-
000712-50-5	Methanone, cyclohexylphenyl-
000091-20-3	Naphthalene
077428-15-0	Naphthalene, 1,2,3,4-tetrahydro-5-methyl-1-(1-methylethyl)-
000090-12-0	Naphthalene, 1-methyl-
000091-57-6	Naphthalene, 2-methyl-
000000-00-0	Naphthalene, dimethyl-, 2 isomers
000630-03-5	Nonacosane
019666-30-9	Oxadiazon
000629-99-2	Pentacosane
000085-01-8	Phenanthrene
000483-65-8	Phenanthrene, 1-methyl-7-(1-methylethyl)-
000000-00-0	Phenanthrene, dimethyl-
000000-00-0	Phenanthrene, methyl-
000128-37-0	Phenol, 2,6-bis (1,1-dimethylethyl)-4-methyl-
000616-55-7	Phenol, 4,6-di(1,1-dimethylethyl)-2-methyl-
000129-00-0	Pyrene
000000-00-0	Pyridine, dichloro-
002176-62-7	Pyridine, pentachloro-
000000-00-0	Pyridine, tetrachloro-
000000-00-0	Pyridine, trichloro-, 2 isomers
000000-00-0	Pyrimidine,6-benzyl-2,4-dichloro-5-isopropyl-
000000-00-0	Quinoline, (chloromethyl)-
000602-09-5	[1,1'-Binaphthalene]-2,2'-diol

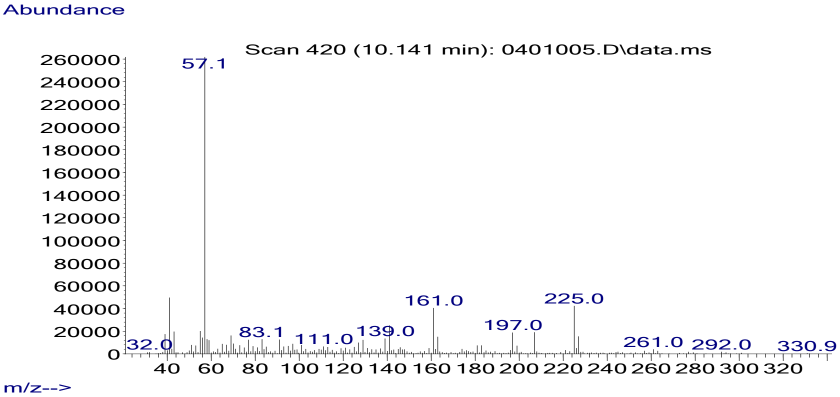
Note: chromatogram contained several compounds that cannot be identified to any degree of reliability. The fragmentation of these compounds is presented below.







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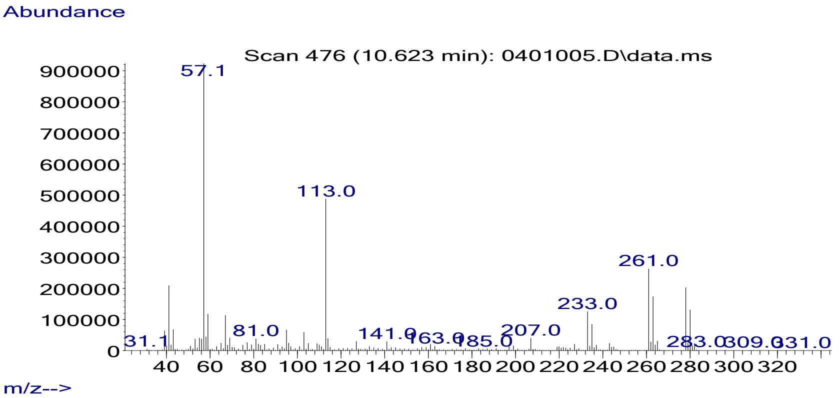
292.0
330.9
m/z-->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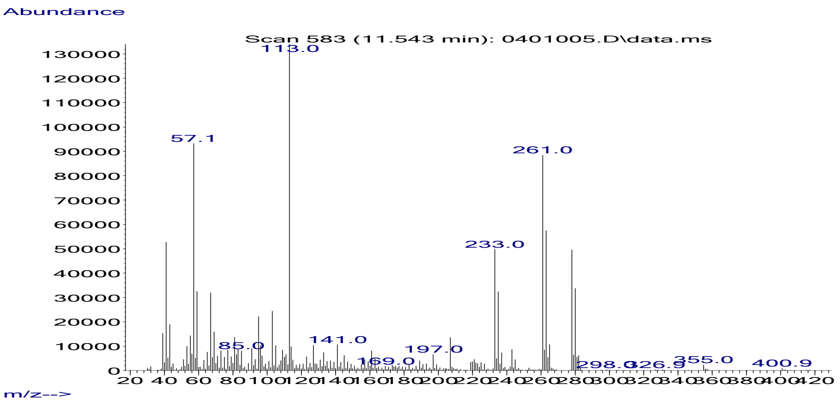
141.0
163.0
185.0
207.0
283.0
309.0
331.0
m/z-->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261.0
233.0
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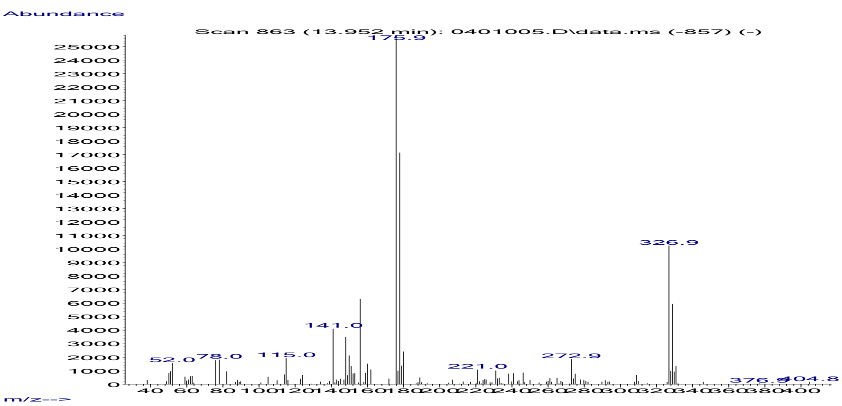
141.0
169.0
197.0
298.0
326.9
355.0
400.9
m/z-->


Abundance
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326.9
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52.0
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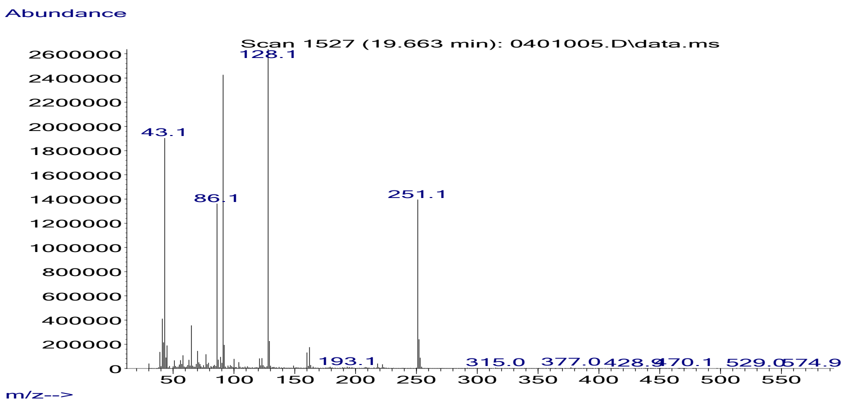
221.0
272.9
m/z-->



Abundance
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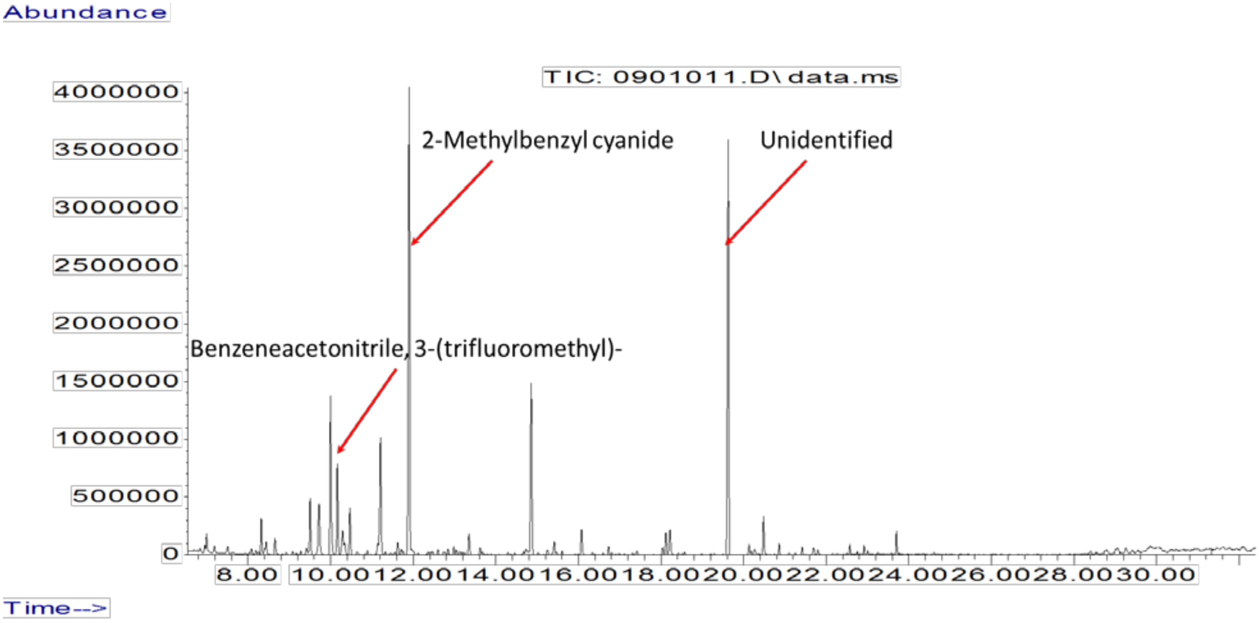
43.1
86.1
251.1
193.1
315.0
377.0
470.1
529.0
574.9
2600000
2400000
2200000
2000000
1800000
1600000
1400000
1200000
1000000
800000
600000
400000
200000
0
50 100 150 200 250 300 350 400 450 500 550
m/z-->

CN16005 (wastewater)

Number of semi-volatile compounds isolated: 97

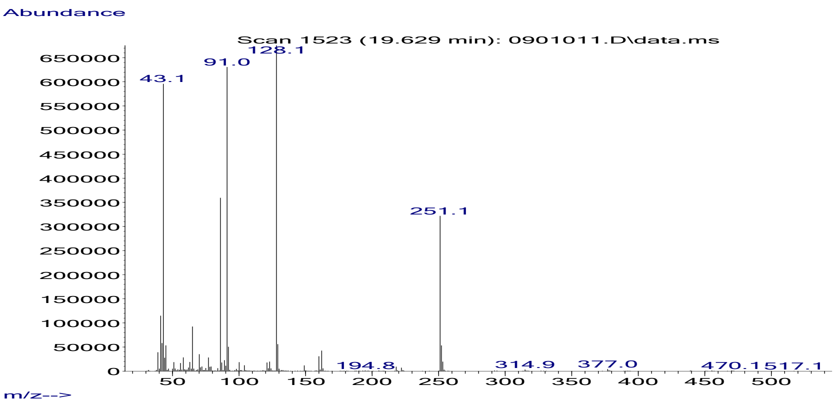


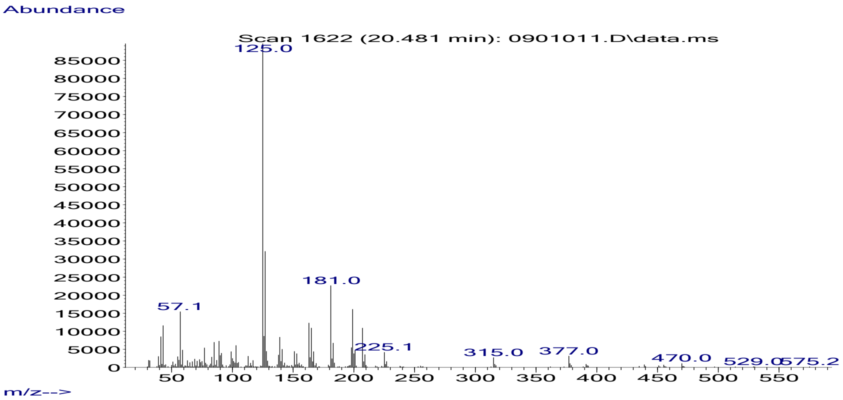
Compounds identified to better than 90%:

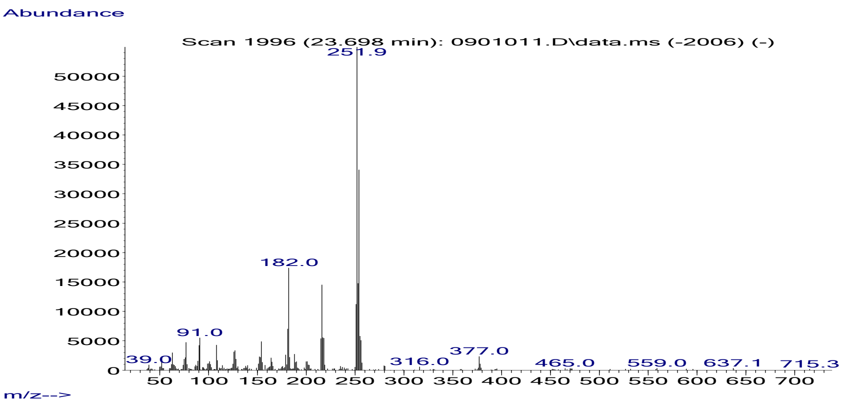
CAS#	Name
000131-11-3	1,2-Benzenedicarboxylic acid, dimethyl ester
000084-66-2	1,2-Benzenedicarboxylic acid, diethyl ester
000084-69-5	1,2-Benzenedicarboxylic acid, diisobutyl ester
000272-16-2	1,2-Benzisothiazole
000120-72-9	1H-Indole
001477-42-5	2-Benzothiazolamine, 4-methyl-
022364-68-7	2-Methylbenzyl cyanide
003218-49-3	3,4-Dichlorophenylacetonitrile
001563-38-8	7-Benzofuranol, 2,3-dihydro-2,2-dimethyl-
000608-31-1	Benzenamine, 2,6-dichloro-
000094-70-2	Benzenamine, 2-ethoxy-
000626-43-7	Benzenamine, 3,5-dichloro-
000087-60-5	Benzenamine, 3-chloro-2-methyl-
000106-47-8	Benzenamine, 4-chloro-
000095-79-4	Benzenamine, 5-chloro-2-methyl-
000095-50-1	Benzene, 1,2-dichloro-
000615-60-1	Benzene, 4-chloro-1,2-dimethyl-
002338-76-3	Benzeneacetonitrile, 3-(trifluoromethyl)-
000140-53-4	Benzeneacetonitrile, 4-chloro-
000100-47-0	Benzonitrile
000623-03-0	Benzonitrile, 4-chloro-
000615-22-5	Benzothiazole, 2-(methylthio)-
000000-00-0	Benzyl alcohol, (trifluoromethyl)-
000619-33-0	Ethane, 1,1-dichloro-2,2-diethoxy-
003740-92-9	Fenclorim

021087-64-9	Metribuzin
002212-67-1	Molinate
000120-83-2	Phenol, 2,4-dichloro-
001570-65-6	Phenol, 2,4-dichloro-6-methyl-
000128-37-0	Phenol, 2,6-bis(1,1-dimethylethyl)-
000098-17-9	Phenol, 3-(trifluoromethyl)-
001570-64-5	Phenol, 4-chloro-2-methyl-
002953-29-9	Phosphorodithioic acid, O,O,S-trimethyl ester
000078-67-1	Propanenitrile, 2,2'-azobis[2-methyl-
000000-00-0	Quinoline, chloromethyl-
019666-30-9	Oxadiazon
003140-93-0	Thiophene, 2,3-dibromo-

Note: chromatogram contained several compounds that cannot be identified to any degree of reliability. The fragmentation of these compounds is presented below.







Number of <u>volatile</u> compounds isolated: 54

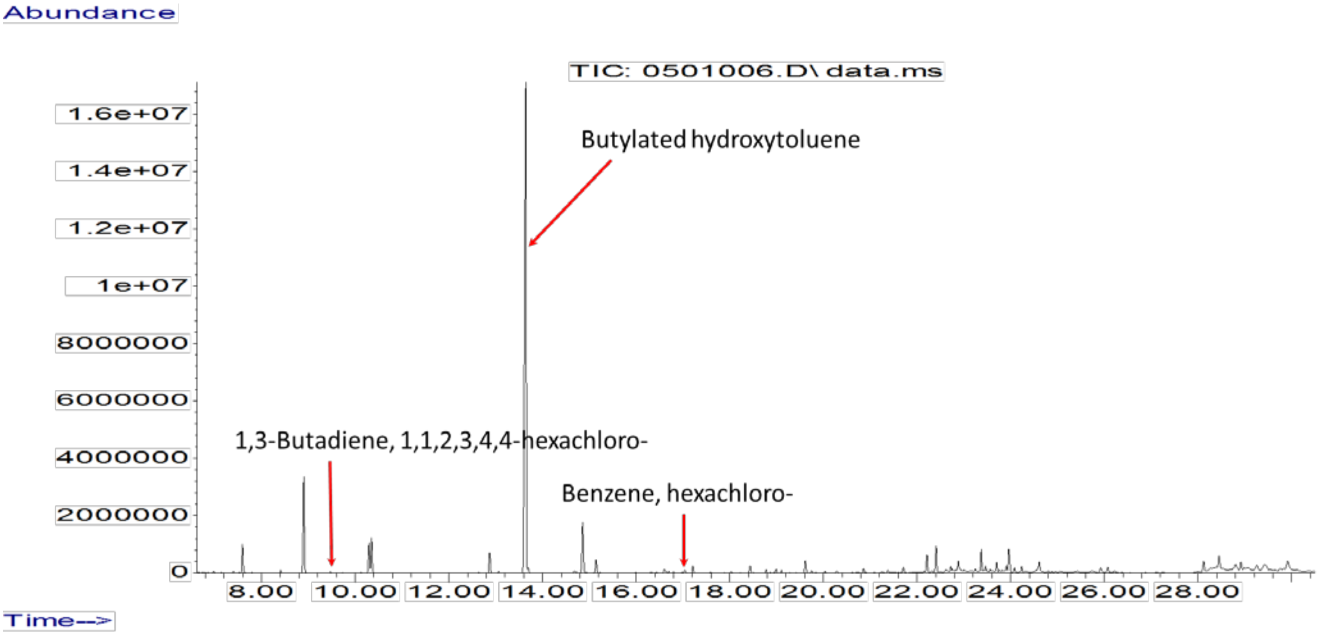
Compounds identified to better than 90%: 36

CAS#	Name
000000-00-0	4-Amino-3,5-dichlorobenzotrifluoride
000000-00-0	Benzenamine, dichloro-
000071-43-2	Benzene
000098-08-8	Benzene, (trifluoromethyl)-
000087-61-6	Benzene, 1,2,3-trichloro-
000120-82-1	Benzene, 1,2,4-trichloro-
000095-50-1	Benzene, 1,2-dichloro-
000541-73-1	Benzene, 1,3-dichloro-
000106-46-7	Benzene, 1,4-dichloro-
000000-00-0	Benzene, 1-chloro-methyl- (2 isomers)
000108-90-7	Benzene, chloro-
000100-41-4	Benzene, ethyl-
022364-68-7	Benzeneacetonitrile, 2-methyl-
000067-66-3	Chloroform
000000-00-0	Dichlorobenzotrifluoride
000075-18-3	Dimethyl sulfide
000121-44-8	Ethanamine, n,n-diethyl-
000079-00-5	Ethane, 1,1,2-trichloro-
000107-06-2	Ethane, 1,2-dichloro-
000127-18-4	Ethene, tetrachloro-
000079-01-6	Ethene, trichloro-
000000-00-0	m-/p-Xylene
000075-09-2	Methane, dichloro-
000095-47-6	o-Xylene
002176-62-7	Pyridine, pentachloro-
000108-88-3	Toluene
000000-00-0	Xylene, chloro- (2 isomers)
000098-82-8	Benzene, isopropyl- (SIM)
000075-15-0	Carbon disulfide (SIM)
000110-82-7	Cyclohexane (SIM)
000108-87-2	Cyclohexane, methyl- (SIM)

000075-35-4	Ethene, 1,1-dichloro- (SIM)
000156-59-2	Ethene, 1,2-dichloro-, cis- (SIM)
000078-87-5	Propane, 1,2-dichloro- (SIM)

CN16006 (Sediment)

Number of compounds isolated: 94

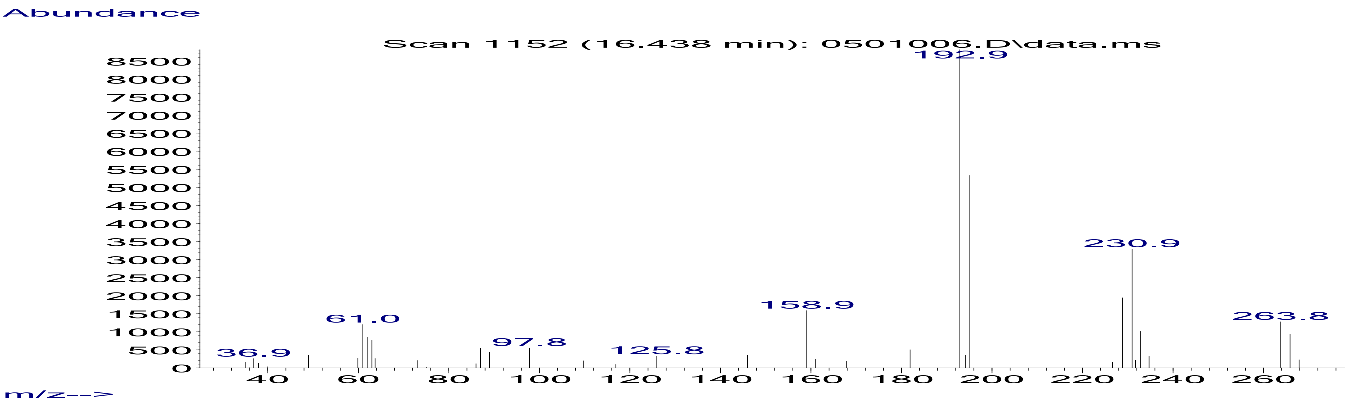


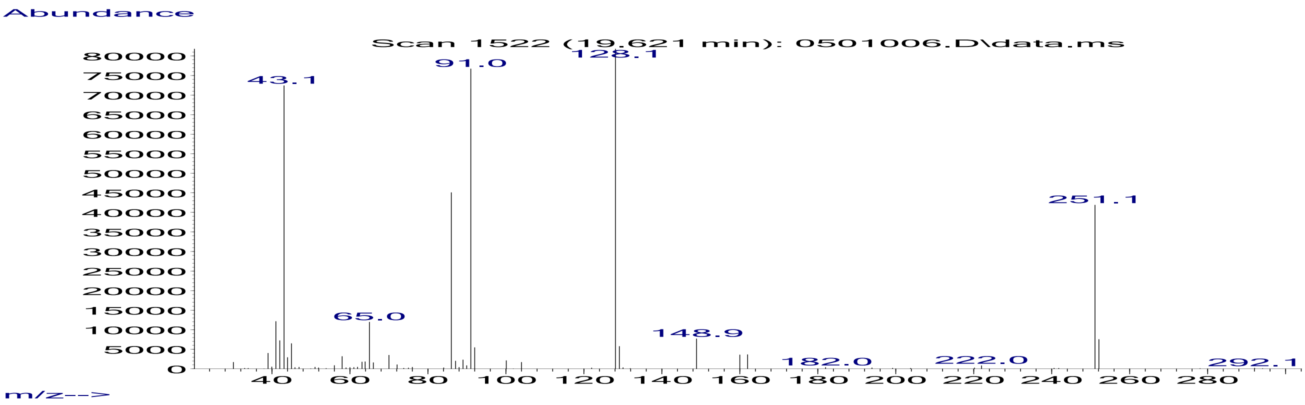
Compounds identified to better than 90%:

CAS#	Name
091465-08-6	.lambda.-Cyhalothrin, 2 isomers
006576-93-8	1,2,5-Trithiepane
000131-11-3	1,2-Benzenedicarboxylic acid, dimethyl ester
000084-66-2	1,2-Benzenedicarboxylic acid, diethyl ester
000117-81-7	1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester
000084-69-5	1,2-Benzenedicarboxylic acid, diisobutyl ester
001025-15-6	1,3,5-Triazine-2,4,6(1h,3h,5h)-trione, 1,3,5-tri-2-propenyl-
000087-68-3	1,3-Butadiene, 1,1,2,3,4,4-hexachloro-
000719-22-2	2,5-Cyclohexadiene-1,4-dione, 2,6-bis(1,1-dimethylethyl)-
010396-80-2	2,6-Di(t-butyl)-4-hydroxy-4-methyl-2,5-cyclohexadien-1-one
001477-42-5	2-Benzothiazolamine, 4-methyl-
000082-05-3	7H-benz[de]anthracen-7-one
014233-37-5	9,10-Anthracenedione, 1,4-bis[(1-methylethyl)amino]-
000082-45-1	9,10-Anthracenedione, 1-amino-
000081-49-2	9,10-Anthracenedione, 1-amino-2,4-dibromo-
000081-62-9	9,10-Anthracenedione, 1-amino-4-bromo-
000086-73-7	9H-Fluorene
000605-48-1	Anthracene, 9,10-dichloro-
000084-65-1	Anthraquinone
000082-38-2	Anthraquinone, 1-(methylamino)-
000095-76-1	Benzenamine, 3,4-dichloro-
004537-15-9	Benzene, (1-butylheptyl)-
002400-00-2	Benzene, (1-ethyldecyl)-
004536-86-1	Benzene, (1-propyloctyl)-
005216-32-0	Benzene, 1,1'-(1,2-dichloro-1,2-ethenediyl)bis-, (Z)-
000951-86-0	Benzene, 1,1'-(1,2-dichloro-1,2-ethenediyl)bis-, (E)-
019814-71-2	Benzene, 1,1'-oxybis[3-methyl-

002444-89-5	Benzene, 1,1'-oxybis[4-chloro-
000101-84-8	Benzene, 1,1’-oxybis-
002136-78-9	Benzene, 1,2,3,4,5-pentachloro-6-(chloromethyl)-
000541-73-1	Benzene, 1,3-dichloro-
051801-69-5	Benzene, 1-methyl-3-(4-methylphenoxy)-
013014-18-1	Benzene, 2,4-dichloro-1-(trichloromethyl)-
000118-74-1	Benzene, hexachloro-
000050-32-8	Benzo[a]pyrene
082657-04-3	Bifenthrin
003896-11-5	Bumetrizole
069327-76-0	Buprofezin
000128-37-0	Butylated hydroxytoluene
002921-88-2	Chlorpyrifos
013700-81-7	Ethane, 1,2-diphenyl-1,1,2,2-tetrachloro-
000206-44-0	Fluoranthene
013798-23-7	Hexathiane
019666-30-9	Oxadiazon
000085-01-8	Phenanthrene
063709-57-9	Phenol, 2,3-dichloro-6-(2,4-dichlorophenoxy)-
000120-83-2	Phenol, 2,4-dichloro-
006341-97-5	Phenol, 2,4-dichloro-, acetate
001570-65-6	Phenol, 2,4-dichloro-6-methyl-
000504-20-1	Phorone
002303-17-5	Triallate
000091-94-1	[1,1'-Biphenyl]-4,4'-diamine, 3,3'-dichloro-

Note: chromatogram contained several compounds that cannot be identified to any degree of reliability. The fragmentation of these compounds is presented below.

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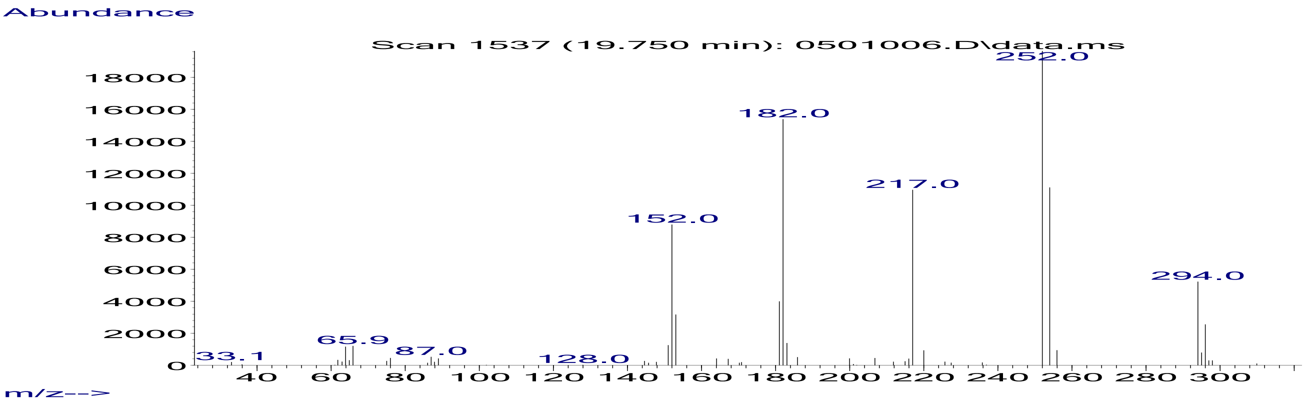
m/z-->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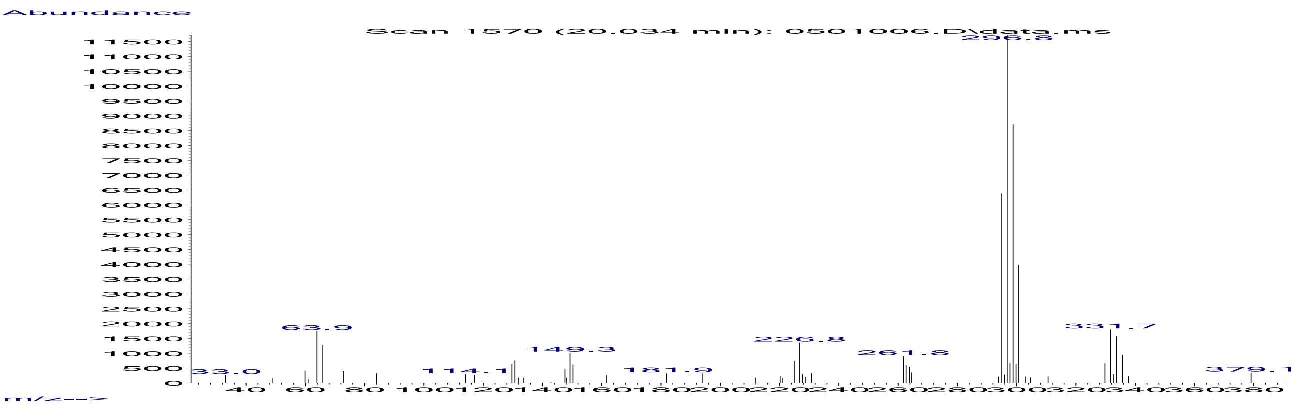
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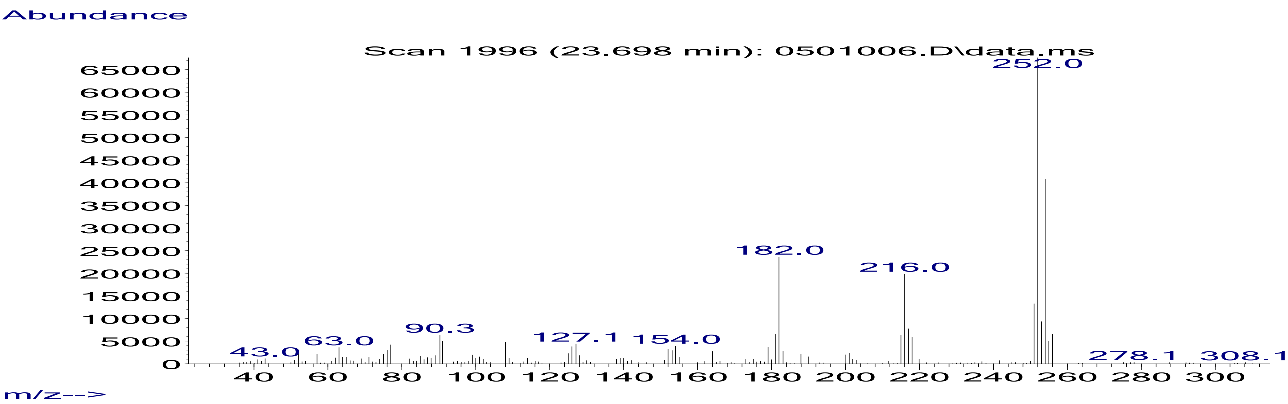
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63.0
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127.1
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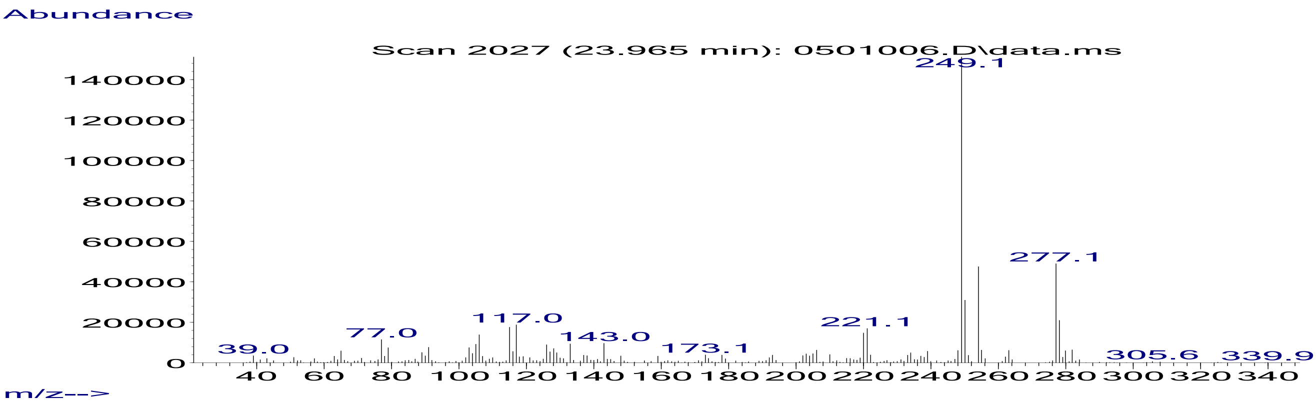
278.1
308.1
m/z-->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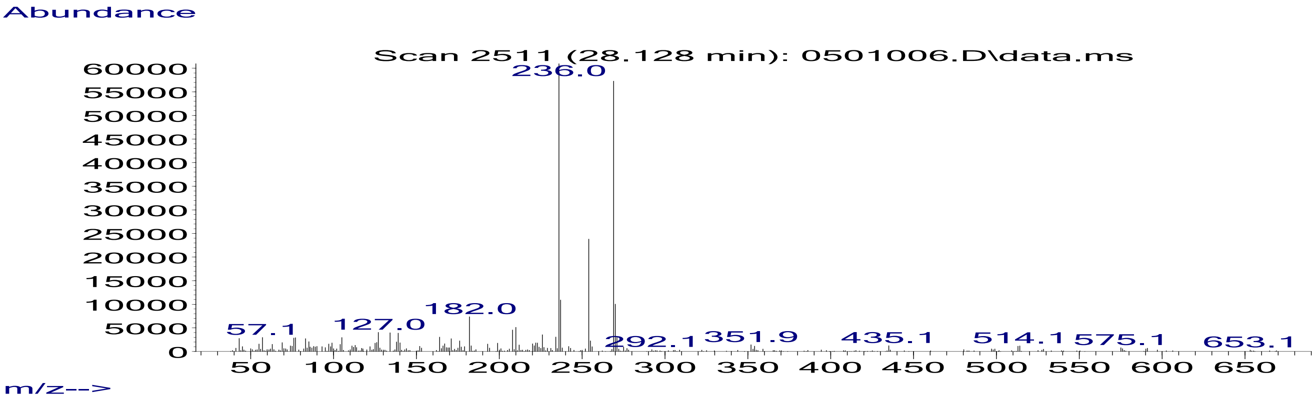
77.0
173.1
39.0
305.6
339.9
m/z-->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127.0
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514.1
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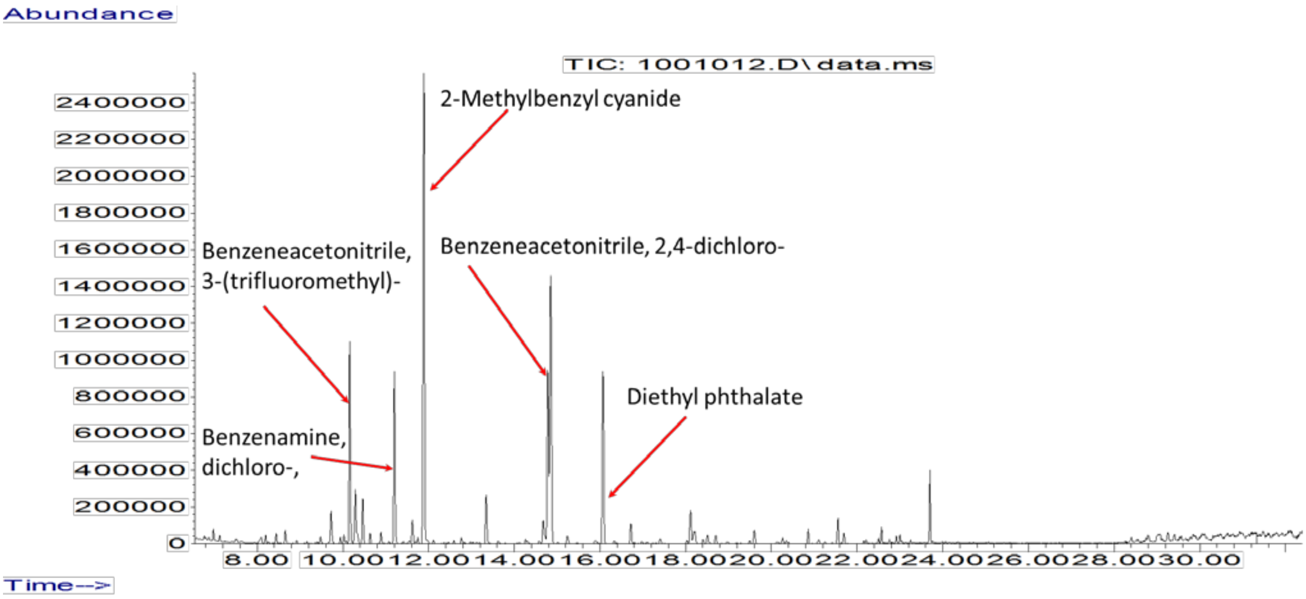
653.1
m/z-->

CN16007 (wastewater)

Number of semi-volatile compounds isolated: 84

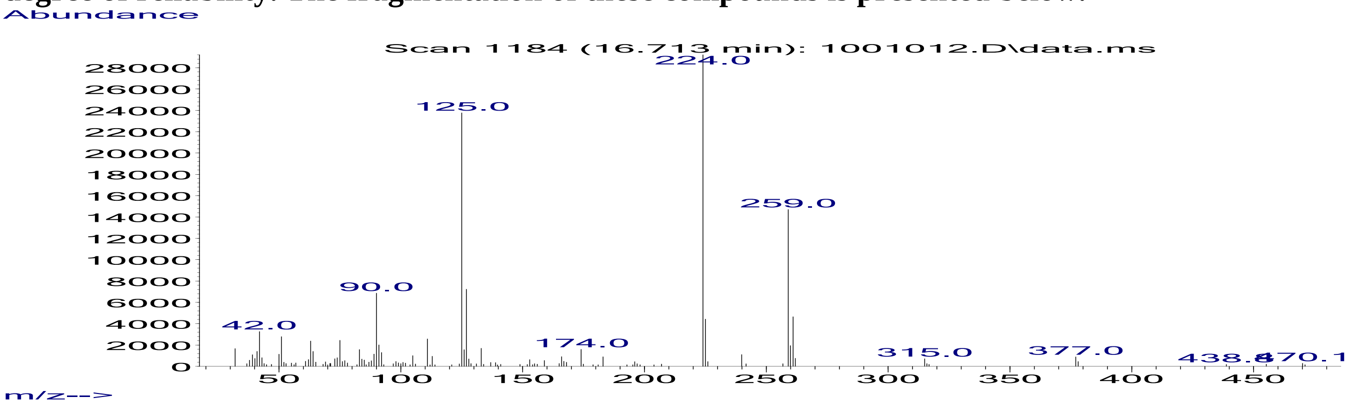


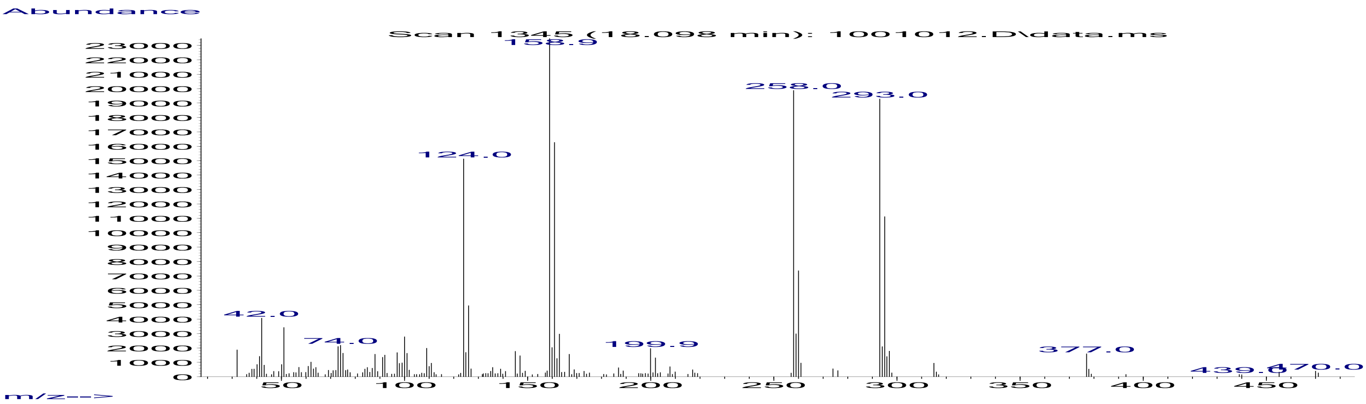
Compounds identified to better than 90%:

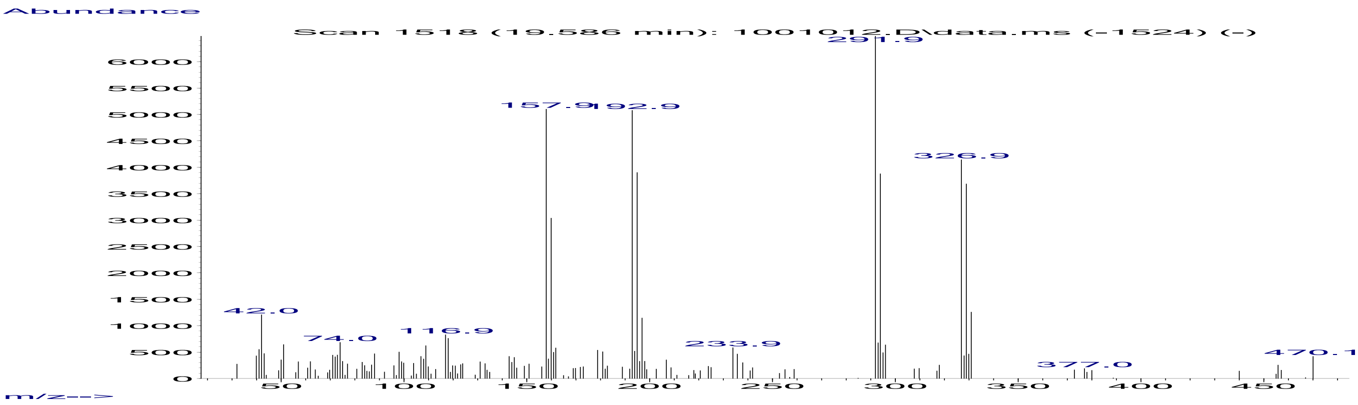
CAS#	Name
000131-11-3	1,2-Benzenedicarboxylic acid, dimethyl ester
000084-66-2	1,2-Benzenedicarboxylic acid, diethyl ester
000084-74-2	1,2-Benzenedicarboxylic acid, dibutyl ester
000084-69-5	1,2-Benzenedicarboxylic acid, diisobutyl ester
001477-42-5	2-Benzothiazolamine, 4-methyl-
022364-68-7	2-Methylbenzyl cyanide
000349-75-7	3-(Trifluoromethyl)benzyl alcohol
129716-11-6	4-Chloro-2-methylbenzyl alcohol
001563-38-8	7-Benzofuranol, 2,3-dihydro-2,2-dimethyl-
161957-61-5	Acetophenone, 3-bromo-2-fluoro-
024279-39-8	Aniline, 2,6-dichloro-4-trifluoromethyl-
000554-00-7	Benzenamine, dichloro-, 2 isomers
000121-69-7	Benzenamine, N,N-dimethyl-
000095-50-1	Benzene, 1,2-dichloro-
000091-16-7	Benzene, 1,2-dimethoxy-
000151-10-0	Benzene, 1,3-dimethoxy-
000705-29-3	Benzene, 1-(chloromethyl)-3-(trifluoromethyl)-
006306-60-1	Benzeneacetonitrile, 2,4-dichloro-
002338-76-3	Benzeneacetonitrile, 3-(trifluoromethyl)-
000140-53-4	Benzeneacetonitrile, 4-chloro-
000089-95-2	Benzenemethanol, 2-methyl-
000589-18-4	Benzenemethanol, 4-methyl-
000100-47-0	Benzonitrile
000766-84-7	Benzonitrile, 3-chloro-
000095-16-9	Benzothiazole
000615-22-5	Benzothiazole, 2-(methylthio)-

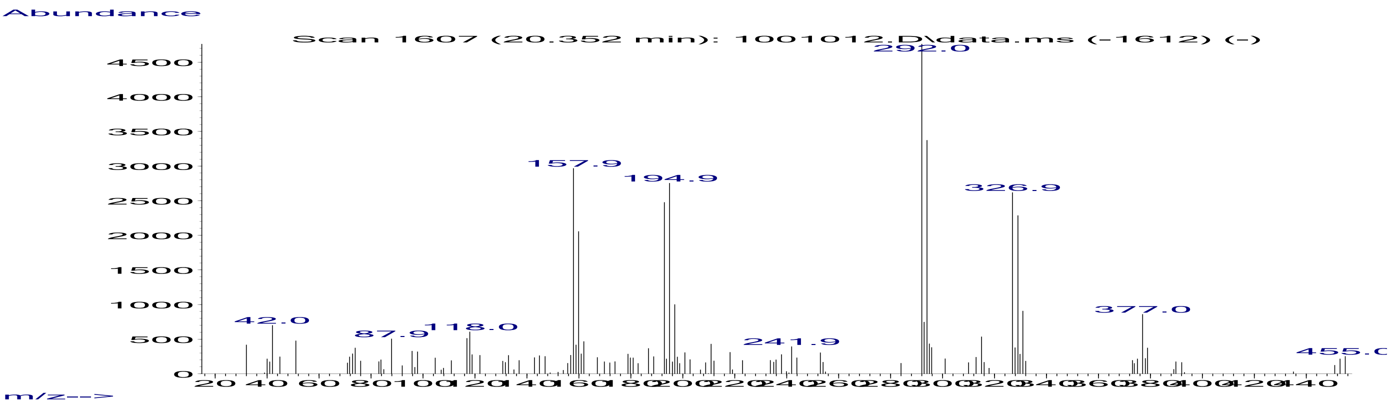
023184-66-9	Butachlor
003740-92-9	Fenclorim
000121-75-5	Malathion
000091-20-3	Naphthalene
026445-11-4	*o*-Xylene, chloro-
000120-83-2	Phenol, 2,4-dichloro-
001570-65-6	Phenol, 2,4-dichloro-6-methyl-
000087-64-9	Phenol, 2-chloro-6-methyl-
000501-92-8	Phenol, 4-propenyl-
002953-29-9	Phosphorodithioic acid, O,O,S-trimethyl ester
000298-06-6	Phosphorodithioic acid, O,O-diethyl ester
000078-67-1	Propanenitrile, 2,2'-azobis[2-methyl-
000000-00-0	Quinolone, (chloromethyl)-
002303-17-5	Triallate

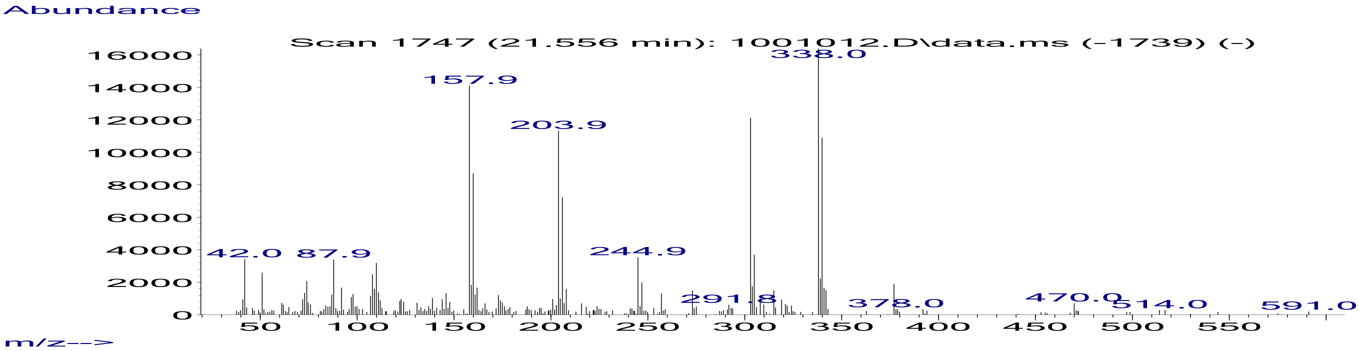
Note: chromatogram contained several compounds that cannot be identified to any degree of reliability. The fragmentation of these compounds is presented below.

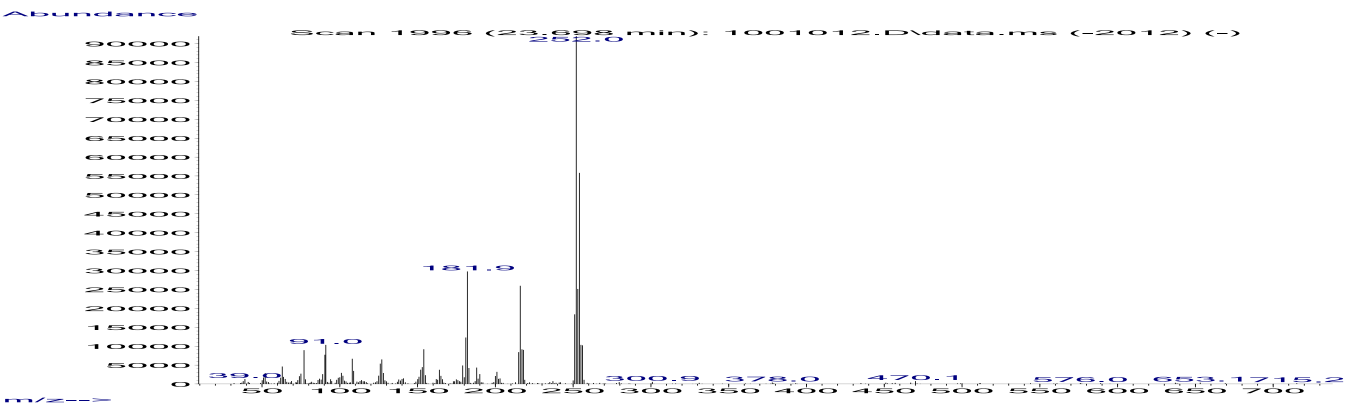





Abundance
Scan 1518 (19.586 min): 1001012.D\data.ms (-1524) (-)
291.9
157.9
192.9
326.9
42.0
74.0
116.9
233.9
377.0
470.1
m/z-->


Abundance
Scan 1607 (20.352 min): 1001012.D\data.ms (-1612) (-)
292.0
157.9
194.9
326.9
42.0
87.9
118.0
241.9
377.0
m/z-->


Abundance
Scan 1747 (21.556 min): 1001012.D\data.ms (-1739) (-)
338.0
157.9
203.9
42.0
87.9
244.9
291.8
378.0
470.0
514.0
591.0
m/z-->


Abundance
Scan 1996 (23.698 min): 1001012.D\data.ms (-2012) (-)
252.0
181.9
91.0
39.0
300.9
378.0
470.1
576.0
m/z-->

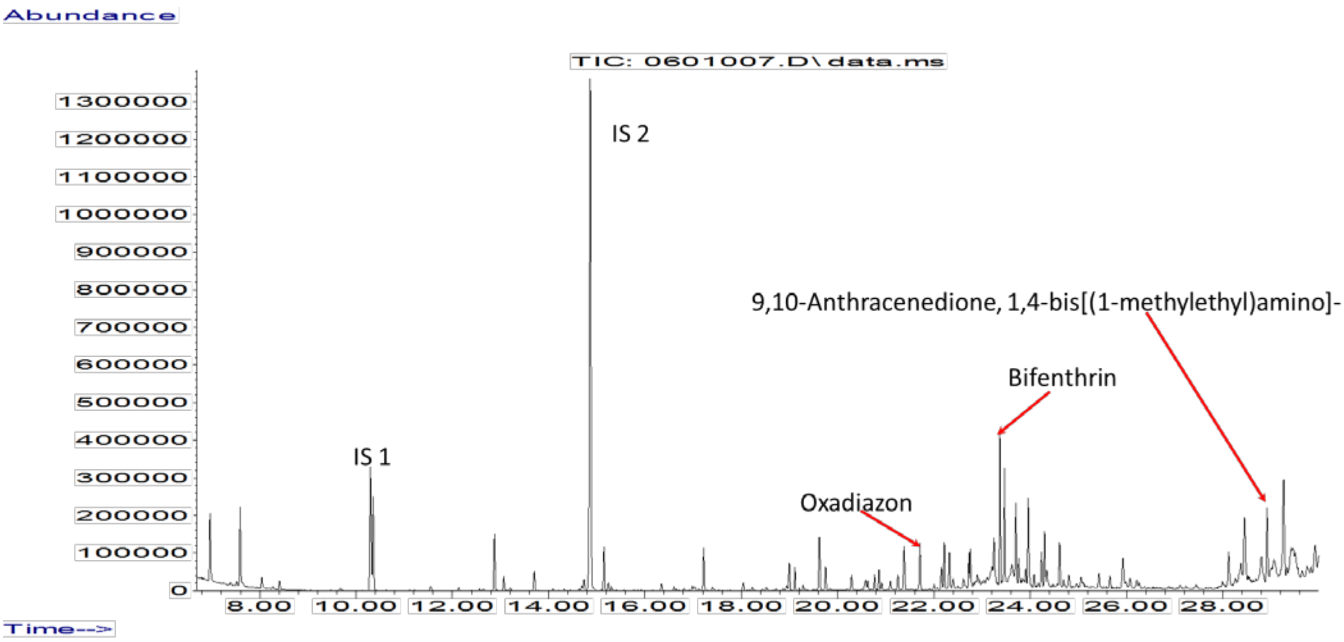
Number of volatile compounds isolated: **52**

Compounds identified to better than 90%:

CAS#	Name
000000-00-0	Benzenamine, dichloro-
000000-00-0	Benzenamine, difluoro-
000071-43-2	Benzene
000098-08-8	Benzene, (trifluoromethyl)-
000120-82-1	Benzene, 1,2,4-trichloro-
000095-50-1	Benzene, 1,2-dichloro-
000541-73-1	Benzene, 1,3-dichloro-
000106-46-7	Benzene, 1,4-dichloro-
000705-29-3	Benzene, 1-(chloromethyl)-3-(trifluoromethyl)-
000108-90-7	Benzene, chloro-
000100-41-4	Benzene, ethyl-
000067-66-3	Chloroform
000000-00-0	Dichlorobenzotrifluoride
000075-18-3	Dimethyl sulfide
000121-44-8	Ethanamine, n,n-diethyl-
000107-06-2	Ethane, 1,2-dichloro-
000127-18-4	Ethene, tetrachloro-
000079-01-6	Ethene, trichloro-
000000-00-0	m-/p-Xylene
000075-09-2	Methane, dichloro-
000095-47-6	o-Xylene
000108-88-3	Toluene
000000-00-0	Toluene, chloro- (2 isomers)
000000-00-0	Xylene, chloro- (2 isomers)
000098-82-8	Benzene, isopropyl- (SIM)
000075-25-2	Bromoform (SIM)
000075-15-0	Carbon disulfide (SIM)
000110-82-7	Cyclohexane (SIM)
000108-87-2	Cyclohexane, methyl- (SIM)
000079-00-5	Ethane, 1,1,2-trichloro- (SIM)
000075-35-4	Ethene, 1,1-dichloro- (SIM)
000056-23-5	Methane, tetrachloro- (SIM)
000078-87-5	Propane, 1,2-dichloro- (SIM)

CN16008 (sediment)

Number of compounds isolated: **39**

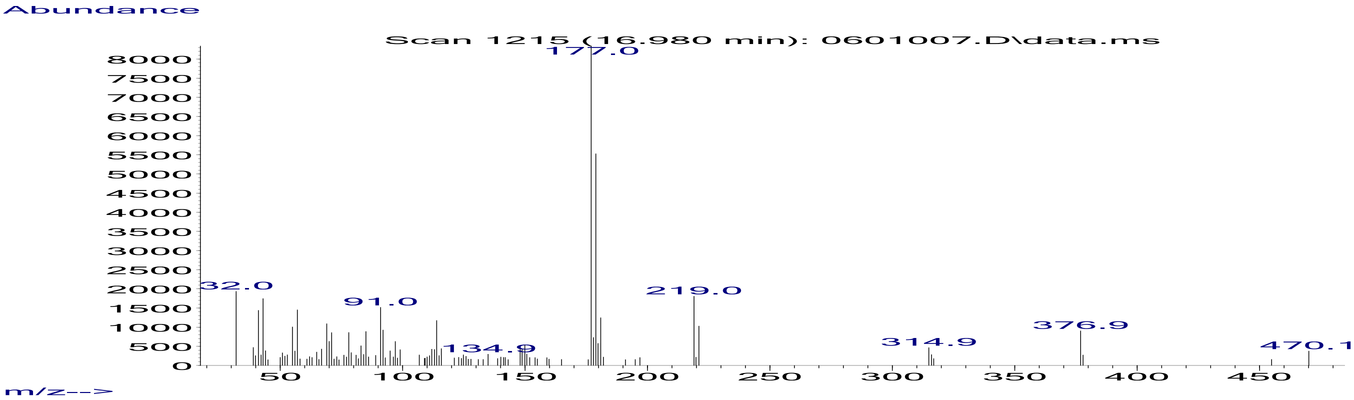


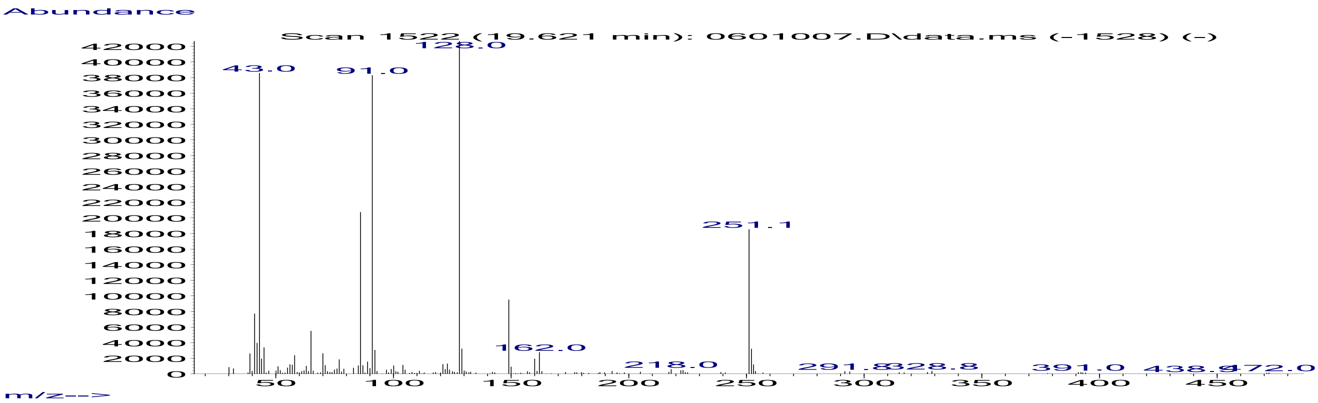
Compounds identified to better than 90%:

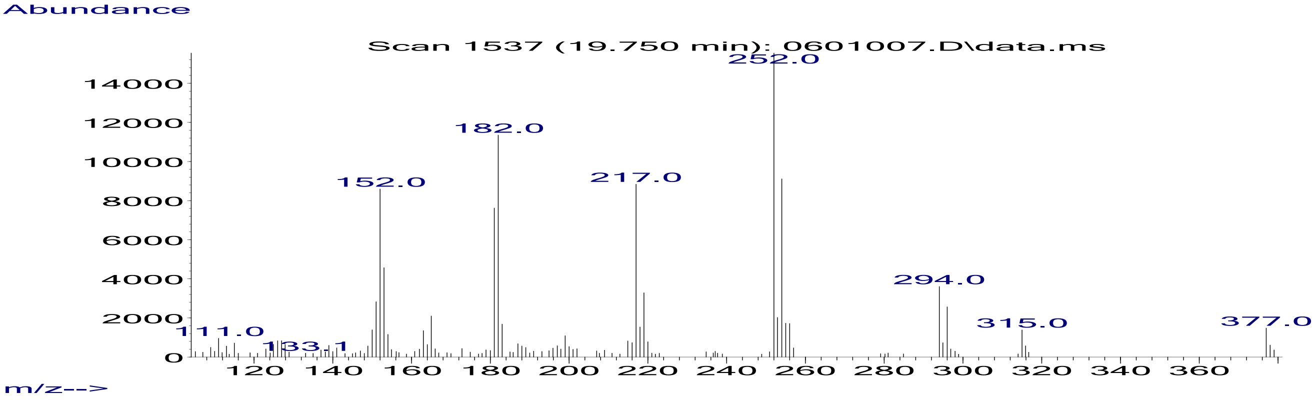
CAS#	Name
091465-08-6	.lambda.-Cyhalothrin
003075-84-1	1,1'-Biphenyl, 2,2',5,5'-tetramethyl-
000131-11-3	1,2-Benzenedicarboxylic acid, dimethyl ester
000117-81-7	1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester
000082-05-3	7H-Benz[de]anthracen-7-one
000084-65-1	9,10-Anthracene-dione
014233-37-5	9,10-Anthracenedione, 1,4-bis[(1-methylethyl)amino]-
000082-45-1	9,10-Anthracenedione, 1-amino-
000116-82-5	9,10-Anthracenedione, 1-amino-2-bromo-4-hydroxy-
000081-62-9	9,10-Anthracenedione, 1-amino-4-bromo-
000129-43-1	9,10-Anthracenedione, 1-hydroxy-
000000-00-0	Benzenamine, dichloro-
082657-04-3	Bifenthrin
003896-11-5	Bumetrizole
069327-76-0	Buprofezin
002921-88-2	Chlorpyrifos
000057-88-5	Cholest-5-en-3-ol (3.beta.)-
007334-33-0	Diazene, bis(2-chlorophenyl)-
119446-68-3	Difenoconazole
051661-17-7	Ethane, 1,2-bis(2,4,6-trichlorophenoxy)-

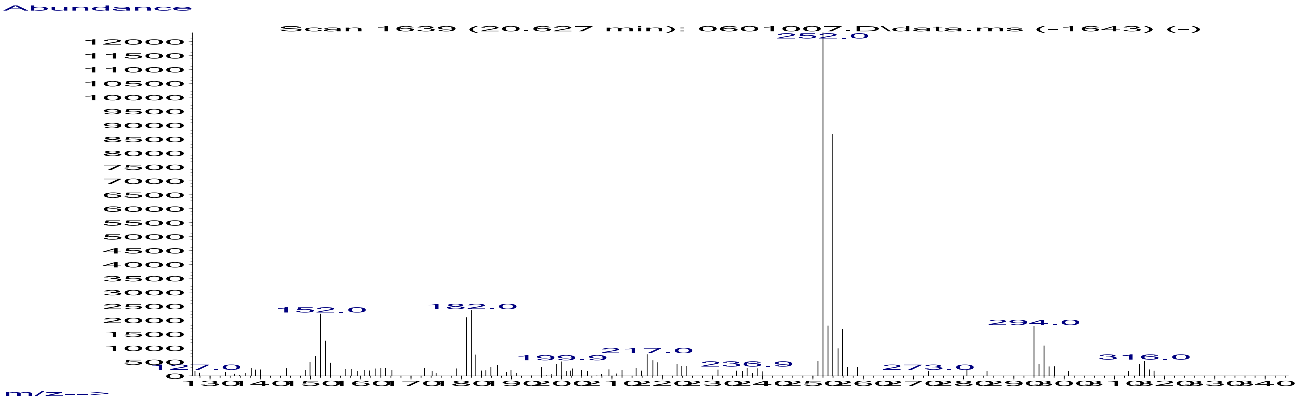
000206-44-0	Fluoranthene
019666-30-9	Oxadiazon
000085-01-8	Phenantherne
000088-06-2	Phenol, 2,4,6-trichloro-
060207-90-1	Propiconazole
002303-17-5	Triallate

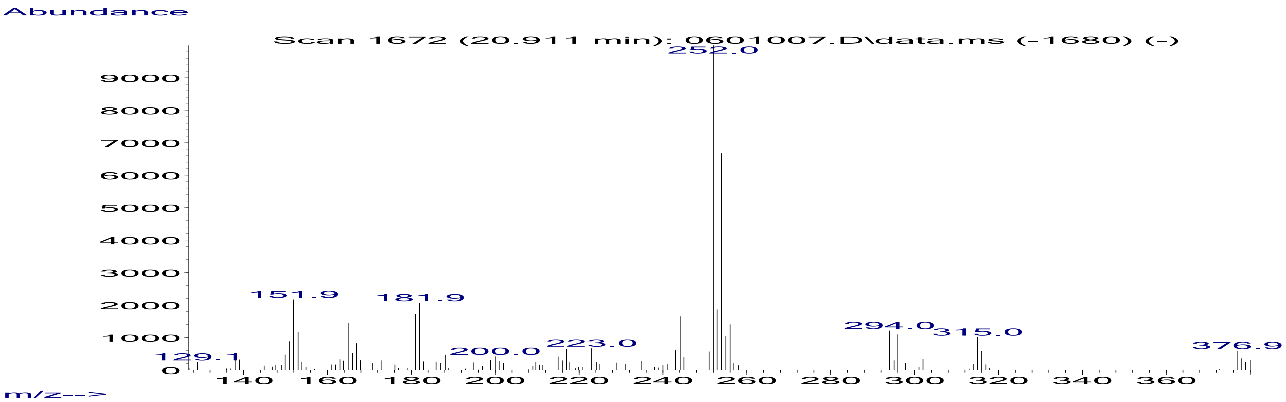
Note: chromatogram contained several compounds that cannot be identified to any degree of reliability. The fragmentation of these compounds is presented below.

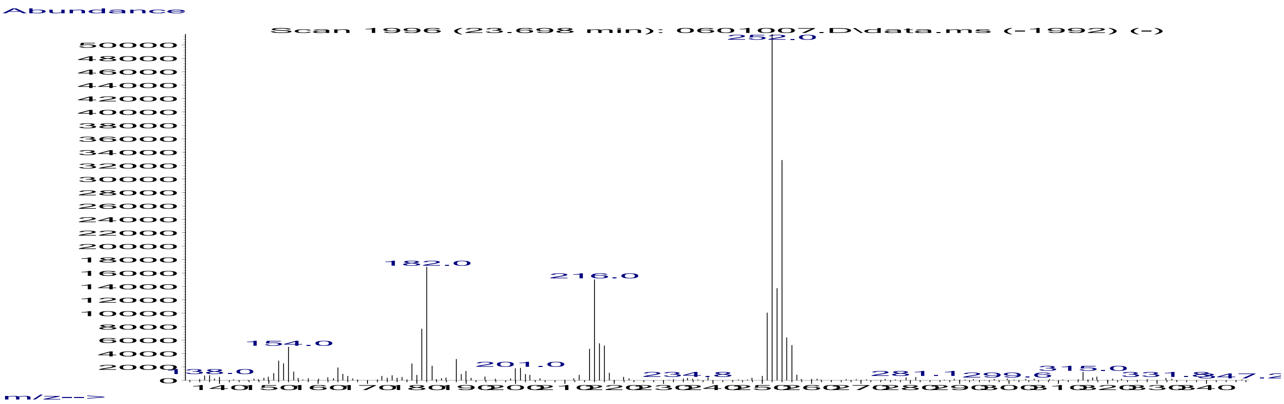


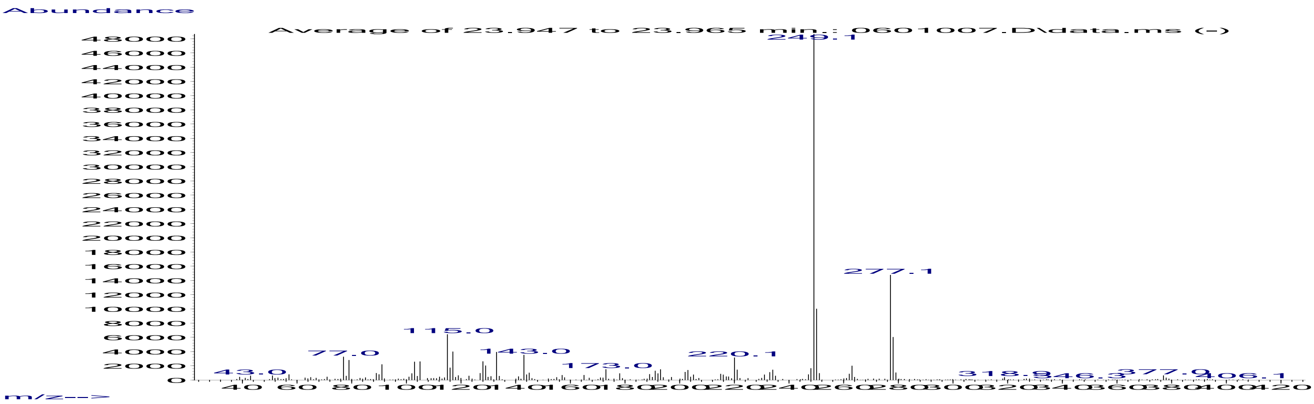


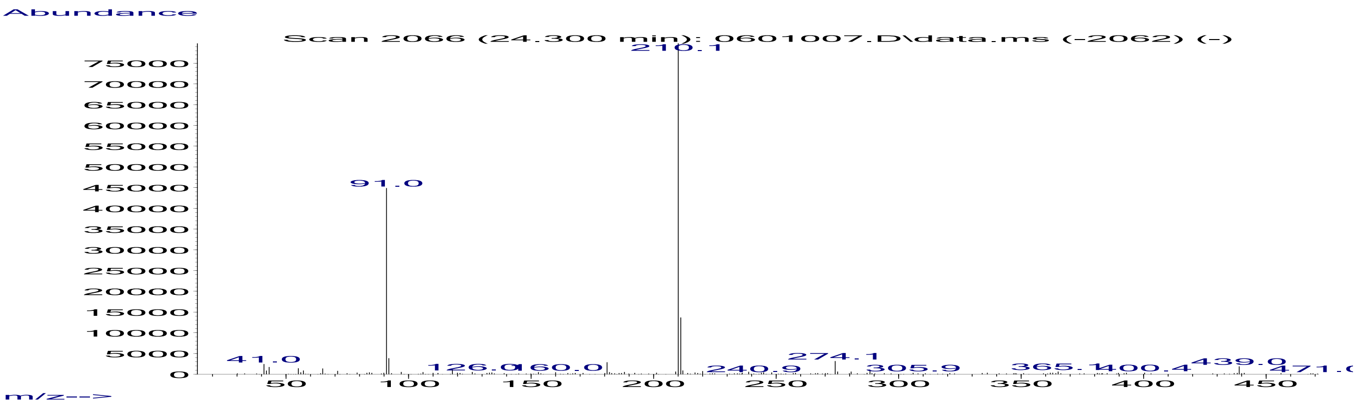

Abundance
Scan 1537 (19.750 min): 0601007.D\data.ms
252.0
182.0
152.0
217.0
294.0
315.0
377.0
111.0
133.1
m/z-->

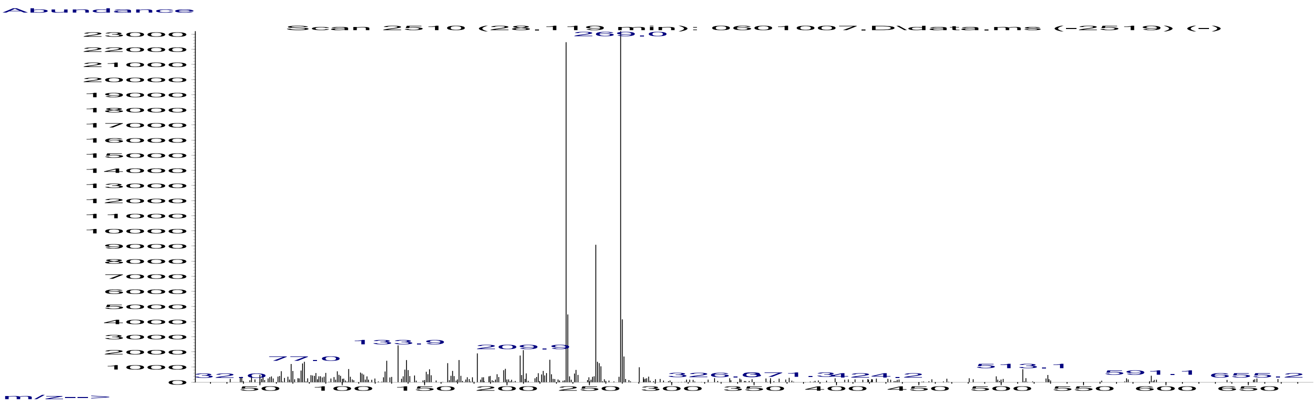

Abundance
Scan 1639 (20.627 min): 0601007.D\data.ms (-1643) (-)
252.0
152.0
182.0
199.9
217.0
236.9
273.0
294.0
316.0
127.0
m/z-->


Abundance
Scan 1672 (20.911 min): 0601007.D\data.ms (-1680) (-)
252.0
151.9
181.9
200.0
223.0
294.0
315.0
376.9
129.1
m/z-->

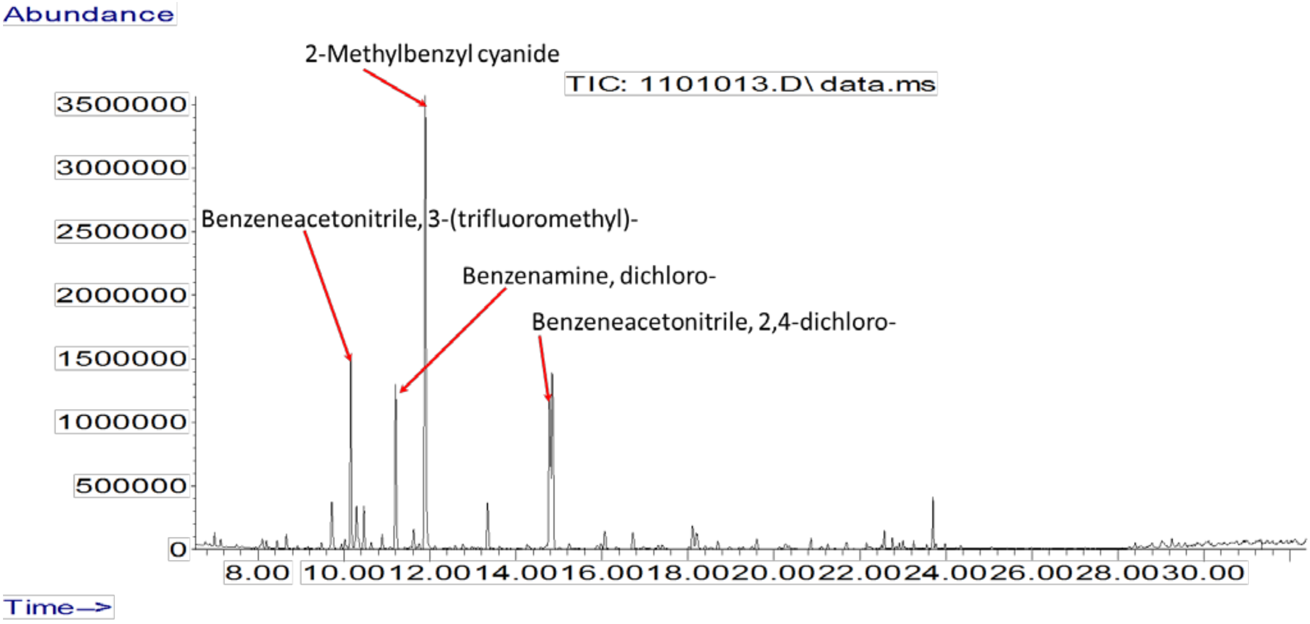

Abundance
Scan 1996 (23.698 min): 0601007.D\data.ms (-1992) (-)
252.0
182.0
216.0
154.0
201.0
138.0
234.8
315.0
m/z-->


Abundance
Average of 23.947 to 23.965 min.: 0601007.D\data.ms (-)
249.1
277.1
115.0
143.0
77.0
220.1
173.0
43.0
m/z-->


Abundance
Scan 2066 (24.300 min): 0601007.D\data.ms (-2062) (-)
210.1
91.0
41.0
274.1
240.9
305.9
439.0
m/z-->


Abundance
Scan 2510 (28.119 min): 0601007.D\data.ms (-2519) (-)
269.0
133.9
77.0
209.9
32.0
513.1
591.1
655.2
m/z-->

CN16009 (wastewater)



Number of semi-volatile compounds isolated: 81

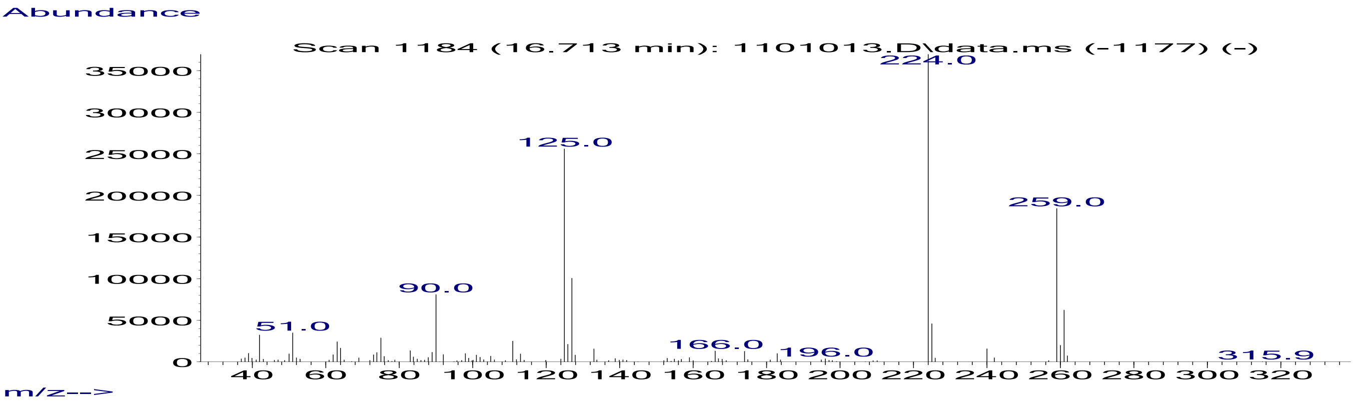
Compounds identified to better than 90%:

CAS#	Name
000084-66-2	1,2-Benzenedicarboxylic acid, diethyl ester
000272-16-2	1,2-Benzisothiazole
001477-42-5	2-Benzothiazolamine, 4-methyl-
022364-68-7	2-Methylbenzyl cyanide
024279-39-8	4-Amino-3,5-dichlorobenzotrifluoride
001563-38-8	7-Benzofuranol, 2,3-dihydro-2,2-dimethyl-
000000-00-0	Benzenamine, dichloro-, 2 isomers
000121-69-7	Benzenamine, N,N-dimethyl-
000095-50-1	Benzene, 1,2-dichloro-
000091-16-7	Benzene, 1,2-dimethoxy-
000541-73-1	Benzene, 1,3-dichloro-
000151-10-0	Benzene, 1,3-dimethoxy-
000705-29-3	Benzene, 1-(chloromethyl)-3-(trifluoromethyl)-
000615-60-1	Benzene, 4-chloro-1,2-dimethyl-
006306-60-1	Benzeneacetonitrile, 2,4-dichloro-
003038-47-9	Benzeneacetonitrile, 2-(trifluoromethyl)-
002338-76-3	Benzeneacetonitrile, 3-(trifluoromethyl)-
000140-53-4	Benzeneacetonitrile, 4-chloro-
000089-95-2	Benzenemethanol, 2-methyl-
000589-18-4	Benzenemethanol, 4-methyl-
000349-95-1	Benzenemethanol, 4-(trifluoromethyl)-
000100-47-0	Benzonitrile
000766-84-7	Benzonitrile, 3-chloro-
000000-00-0	Benzonitrile, difluoro-
000615-22-5	Benzothiazole, 2-(methylthio)-
003740-92-9	Fenclorim

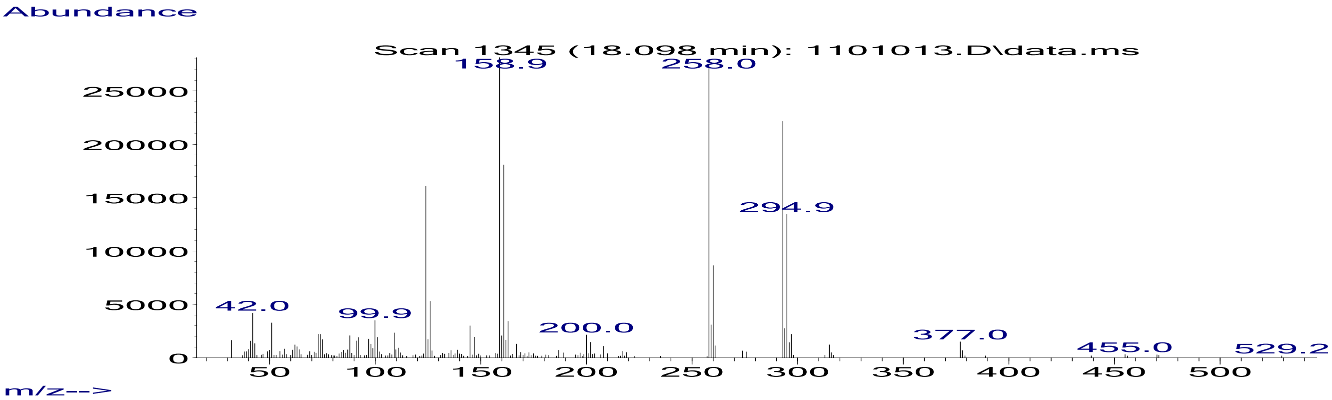
000121-75-5	Malathion
021087-64-9	Metribuzin
000091-20-3	Naphthalene
000120-83-2	Phenol, 2,4-dichloro-
001570-65-6	Phenol, 2,4-dichloro-6-methyl-
000095-57-8	Phenol, 2-chloro-
000095-48-7	Phenol, 2-methyl-
000106-44-5	Phenol, 4-methyl-
000501-92-8	Phenol, 4-propenyl-
000712-50-5	Phenyl cyclohexyl ketone
002953-29-9	Phosphorodithioic acid, O,O,S-trimethyl ester
000078-67-1	Propanenitrile, 2,2'-azobis[2-methyl-
000000-00-0	Quinoline, (chloromethyl)-
002303-17-5	Triallate

Note: chromatogram contained several compounds that cannot be identified to any degree of reliability. The fragmentation of these compounds is presented below.

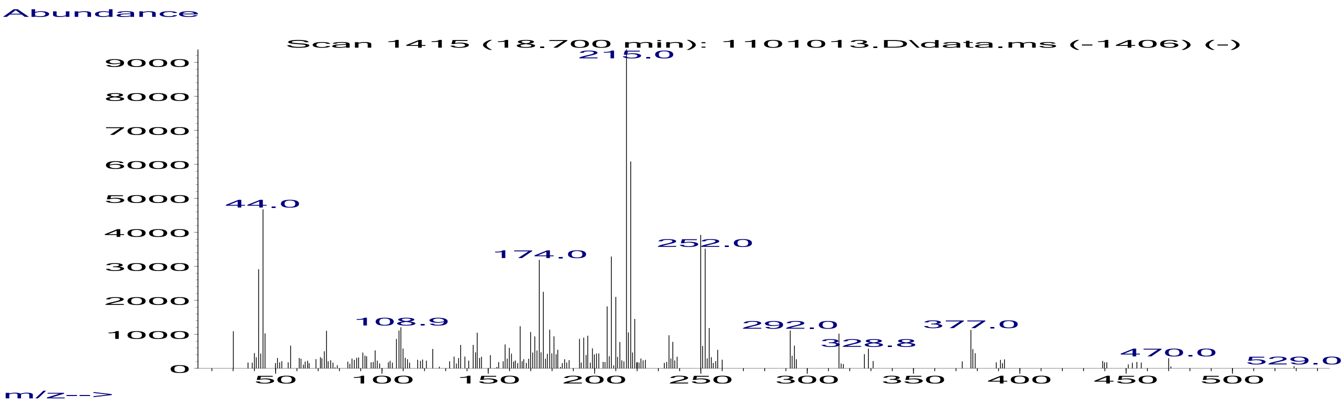
Unknown 1



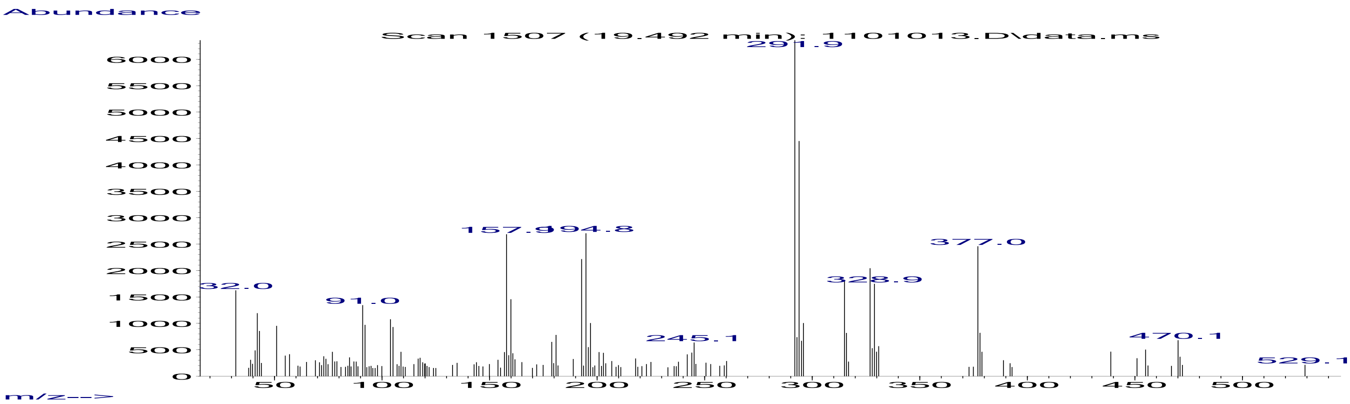
Unknown 2



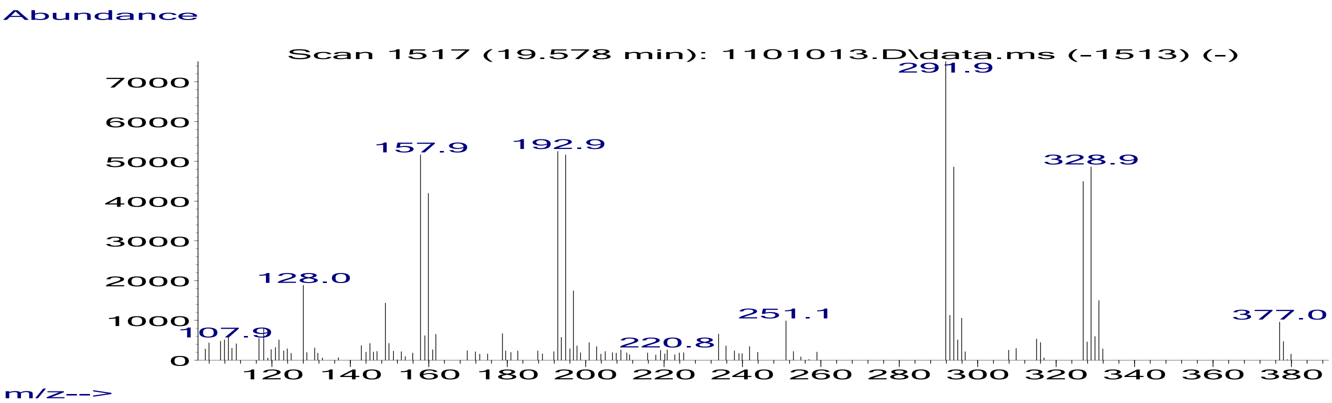
Unknown 3



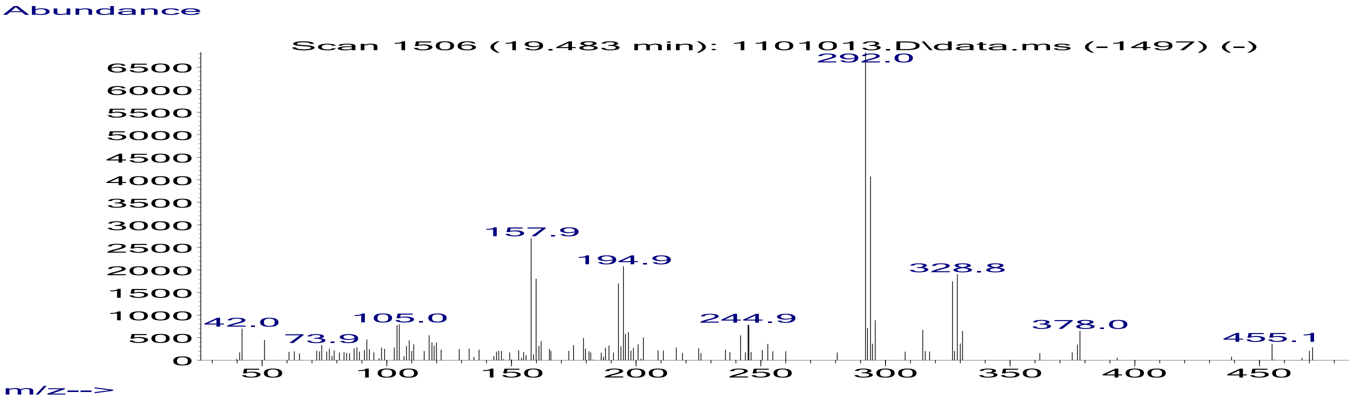
Unknown 4



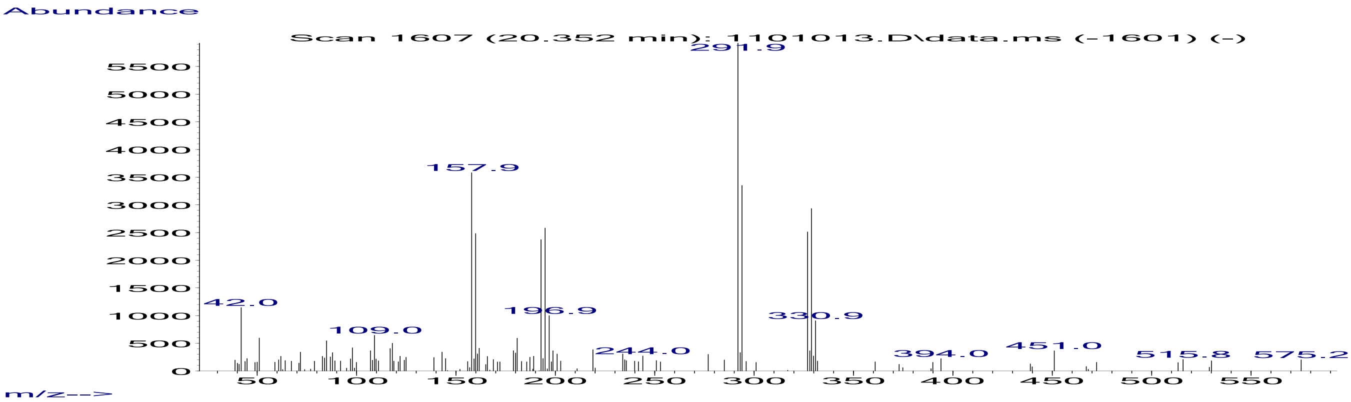
Unknown 5



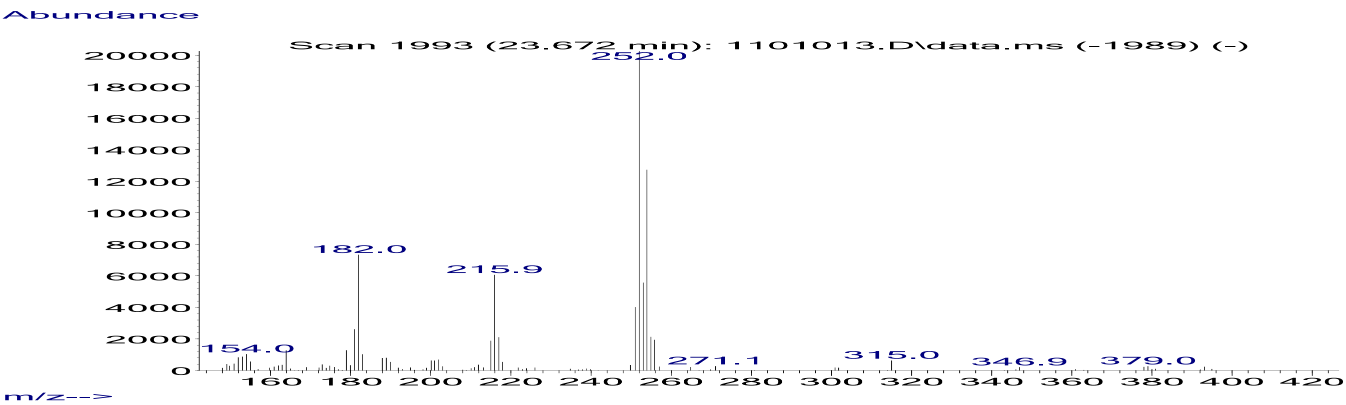
Unknown 6



Unknown 7



Unknown 8



Number of <u>volatile</u> compounds isolated: 53

Compounds identified to better than 90%:

CAS#	Name
000320-50-3	2,5-Dichlorobenzotrifluoride
000000-00-0	Benzenamine, dichloro-
000071-43-2	Benzene
000098-08-8	Benzene, (trifluoromethyl)-
000120-82-1	Benzene, 1,2,4-trichloro-
000095-50-1	Benzene, 1,2-dichloro-
000541-73-1	Benzene, 1,3-dichloro-
000106-46-7	Benzene, 1,4-dichloro-
000108-90-7	Benzene, chloro-
000100-41-4	Benzene, ethyl-
000000-00-0	Difluoroaniline
000075-18-3	Dimethyl sulfide
000624-92-0	Disulfide, dimethyl-
000107-06-2	Ethane, 1,2-dichloro-
000127-18-4	Ethene, tetrachloro-
000000-00-0	m-/p-Xylene
000075-09-2	Methane, dichloro-
000067-66-3	Methane, trichloro- (Chloroform)
000091-20-3	Naphthalene
000095-47-6	o-Xylene
000108-88-3	Toluene
000000-00-0	Toluene, chloro- (3 isomer)
000121-44-8	Triethylamine
000000-00-0	Trifluoromethylbenzyl chloride
000000-00-0	(Trifluoromethyl)phenylacetonitrile
000000-00-0	Xylene, chloro- (2 isomers)
000098-82-8	Benzene, isopropyl- (SIM)
000075-25-2	Bromoform (SIM)
000075-15-0	Carbon disulfide (SIM)
000110-82-7	Cyclohexane (SIM)
000108-87-2	Cyclohexane, methyl- (SIM)
000079-00-5	Ethane, 1,1,2-trichloro- (SIM)
000075-35-4	Ethene, 1,1-dichloro- (SIM)
000156-59-2	Ethene, 1,2-dichloro-, cis- (SIM)
000079-01-6	Ethene, trichloro- (SIM)
000056-23-5	Methane, tetrachloro- (SIM)
000078-87-5	Propane, 1,2-dichloro- (SIM)