Greenpeace Research Laboratories Analytical Results 2019-05

Organic contaminants and metals in samples of water, soil, sediment and plastic from waste dumpsites in Malaysia

November 2019

Introduction

A total of 21 samples (16 of surface water, 2 of shredded plastic, 1 burned plastic, 1 soil and 1 sediment) were received from Greenpeace Malaysia for analysis at the Greenpeace Research Laboratories (GRL) on 22nd August 2019. According to documentation supplied, all samples were collected from several sites at which plastic waste is stored or disposed of during one of two visits to the sites, the first between 29-31st July 2019, and the second between the 6th and 7th August 2019.

The locations from which the samples were collected are shown in Figure 1, with descriptions provided in Table 1a, together with GPS coordinates for the sample collection location in Table 1b. A map showing sampling locations is given in Figure 1. As shown in Table 1, some of the 21 samples were duplicate or triplicate sets of identical samples (i.e. collected at the same time and from the same locations), and as such some individual samples were not analysed.

Materials and methods

Concentrations of metals and metalloids were determined for the samples by ICP mass spectrometry (MS) following acid digestion, using appropriate certified reference samples. For water samples, a portion of each sample was filtered through a 0.45 micron filter prior to acidification to enable quantification of dissolved metals in each sample.

The two samples of shredded plastic were each composed of a heterogeneous mix of different plastic fragments. For each sample, the concentrations of metals and metalloids were analysed in three separate, non-identical, subsamples to determine the variation in concentrations within the heterogeneous samples.

Semi-volatile organic compounds (sVOCs) were isolated from samples using Accelerated Solvent Extraction (ASE) system with a mixture of pentane, ethylacetate, and ethanol (ratio 6:3:1). Extracted compounds were subsequently identified as far as possible using gas chromatography/mass spectrometry (GC/MS) operated in simultaneous SCAN/SIM modes. Volatile organic chemicals (VOCs) were identified in samples as received (with no pre-treatment) using GC/MS with HeadSpace sample introduction technique.

More detailed descriptions of the sample preparation and analytical procedures are presented in Appendix 1.

Sample code		Sample type	Collection date	Sample location
Klang, Selangor, Kapar - Jalan Pasan Malam				
MY19001	Duplicate set	water	29.07.2019	Creek next to plastics recycling facility
MY19002[a]		water	29.07.2019	
Sungai Petani unregulated dumpsite				
MY19003	Triplicate set	water	31.07.2019	Sungai Muda River at edge of dumpsite.
MY19004[a]		water	31.07.2019	
MY19005[b]		water	31.07.2019	
MY19006	Single sample	soil	31.07.2019	Sungai Muda river bank, adjacent to river water sample
Pulau Indah dumpsite, Klang				
MY19007	Single sample	ash/BP	06.08.2019	partially burned material from dumpsite
MY19008	Single sample	Sed	06.08.2019	Fish pond close to plastic waste dumpsite and plastic waste recycling factories
MY19009	Triplicate set	water	06.08.2019	Fish pond, as MY19008
MY19010[a]		water	06.08.2019	
MY19011[b]		water	06.08.2019	
MY19012	Triplicate set	water	06.08.2019	Channel flowing into fish pond (MY19009), close to plastic waste dumpsite & downstream of 1 large & several smaller plastic waste recycling factories
MY19013[a]		water	06.08.2019	
MY19014[b]		water	06.08.2019	
MY19015	Triplicate set	water	06.08.2019	Small ditch in front of dumpsite
MY19016[a]		water	06.08.2019	
MY19017[b]		water	06.08.2019	
Pulau Indah, Sungai Chandong				
MY19018	Duplicate set	water	07.08.2019	Channel at waste burning site
MY19019[a]		water	07.08.2019	
Jenjarom dumpsite, Sri Cheeding				
MY19020	Single sample	SP	07.08.2019	Surface layer on ground at former unregulated dumpsite
MY19021	Single sample	SP	07.08.2019	Surface layer on ground at former unregulated dumpsite

Table 1A: Details of surface water, soil, sediment (Sed), ash/burned plastic (BP) and shredded plastic (SP) samples from plastic waste dumpsites in Malaysia received and analysed at the Greenpeace Research Laboratories. a- sample not analysed; b-sample collected in VOC sampling bottle and analysed for VOCs

Sample code	N degree (º)	E degree (º)
MY19001	3.161950	101.438367
MY19003	5.542767	100.584868
MY19006	5.542767	100.584868
MY19007	2.98419	101.345649
MY19008	2.9851	101.346100
MY19009	2.9851	101.346100
MY19012	2.98427	101.346639
MY19015	2.98400	101.346900
MY19018	2.98111	101.36111
MY19020	2.89250	101.56390
MY19021	2.89250	101.56390

Table 1b: GPS coordinates of sample collection locations.

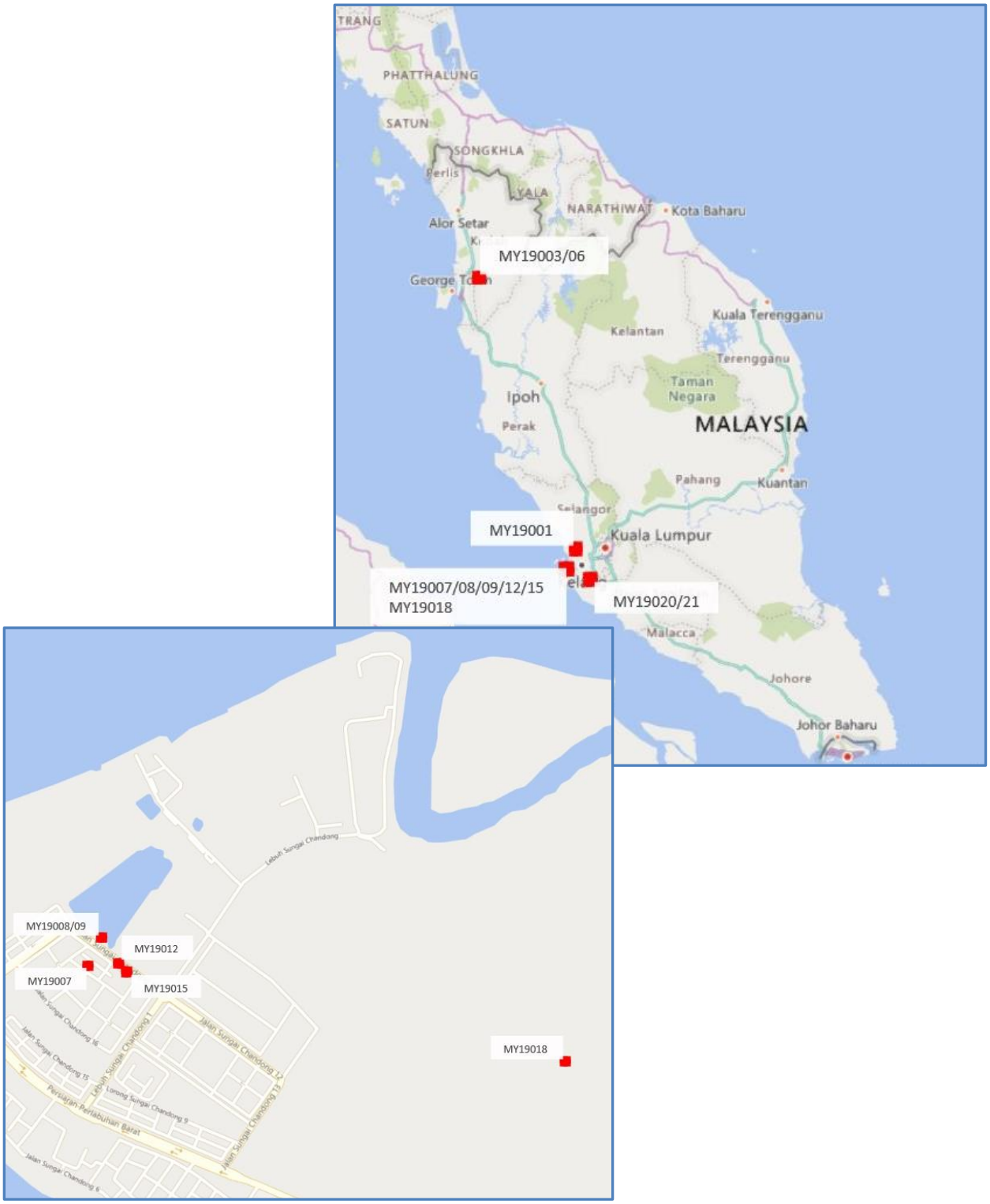


Figure. 1 Locations of sampling points, with expanded section for samples MY19007- MY19018

Results and Discussion

The results for the samples are outlined in the following sections, based on the sites from which they were collected. The concentrations of metals and metalloids are reported in Tables 2a (shredded plastic), 2b (soil, sediment and burned plastic) and 2c (water). The individual organic chemicals identified (where possible) in each sample through forensic mass spectrometry screening techniques are summarised in Table 3.

More detailed lists of the organic chemicals identified in each sample are provided in Appendix 2, along with their corresponding total ion chromatographs.

Jenjarom dumpsite, Sri Cheeding

The 2 samples of shredded plastic collected from the material covering the ground at the dumpsite (MY19020 & MY19021) contained relatively high concentrations of a number of metals and metalloids. For both samples, concentrations of copper, lead and zinc were above 1000 mg/kg (0.1% by weight). Though lower, concentrations of cadmium (9.5-36.9 mg/kg) were also noteworthy as this metal is toxic and commonly found in the environment at very low concentrations, typically below 1 mg/kg in soils and sediments (Alloway 1990, ATSDR 2012, Salomons & Forstner 1984).

Other concentrations of note were those of antimony (58.7-238 mg/kg) and tin (163-214 mg/kg) in both samples, and molybdenum in MY19021 (225-539 mg/kg). In addition, all subsamples contained relatively low levels of mercury (0.26-0.33 mg/kg). Though far lower than the concentrations of many other metals and metalloids, mercury is a highly toxic metal which is typically found in the environment at very low concentrations.

Despite being composed of heterogeneous mixes of different plastic fragments, for each of the 2 samples the concentrations of each metal/metalloid were reasonably similar between each of the 3 subsamples, with the highest concentration for each metal/metalloid in a subsample being only 1-2 times that of the lowest concentration, other than for arsenic, cadmium and molybdenum (2-4 times).

Sample code	MY19020					MY19021				
subsample	/1	/2	/3	median	max	/1	/2	/3	median	max
Aluminium	15400	15000	16600	15400	16600	16900	14700	27300	16900	27300
Antimony	92.2	70.2	58.7	70.2	92.2	63.6	95.6	128	95.6	128
Arsenic	11.8	4.2	3.3	4.2	11.8	4.0	7.0	14.7	7.0	14.7
Barium	232	215	247	232	247	474	283	222	283	474
Beryllium	0.1	0.1	0.1	0.1	0.1	0.1	<0.1	0.1	0.1	0.1
Cadmium	9.52	13.8	36.9	13.8	36.9	20.7	16.7	14	16.7	20.7
Chromium	23.6	54	31.4	31.4	54	56.7	38.7	53.3	53.3	56.7
Cobalt	2.98	2.83	3.63	2.98	3.63	3.76	3.06	3.28	3.28	3.76
Copper	6830	3340	6340	6340	6830	5170	4700	9780	5170	9780
Iron	2350	2150	3310	2350	3310	2850	1550	1530	1550	2850
Lead	2940	2520	2940	2940	2940	4670	3440	3780	3780	4670
Manganese	58.7	60.7	71.5	60.7	71.5	53	79.8	41	53.0	79.8
Mercury	0.33	0.26	0.31	0.31	0.33	0.29	0.28	0.29	0.29	0.29
Molybdenum	127	36.7	92.2	92.2	127	226	225	539	226	539
Nickel	27.7	31.5	37.2	31.5	37.2	26.5	39.1	24.9	26.5	39.1
Strontium	27.6	23.5	29.7	27.6	29.7	39.5	27.9	39.5	39.5	39.5
Tin	193	163	206	193	206	183	214	206	206	214
Titanium	4.8	9.6	8.4	8.4	9.6	13.6	6.7	7	7.0	13.6
Vanadium	3.24	3.10	3.74	3.24	3.74	4.75	3.04	2.65	3.04	4.75
Zinc	2640	2130	3600	2640	3600	3920	2620	4390	3920	4390

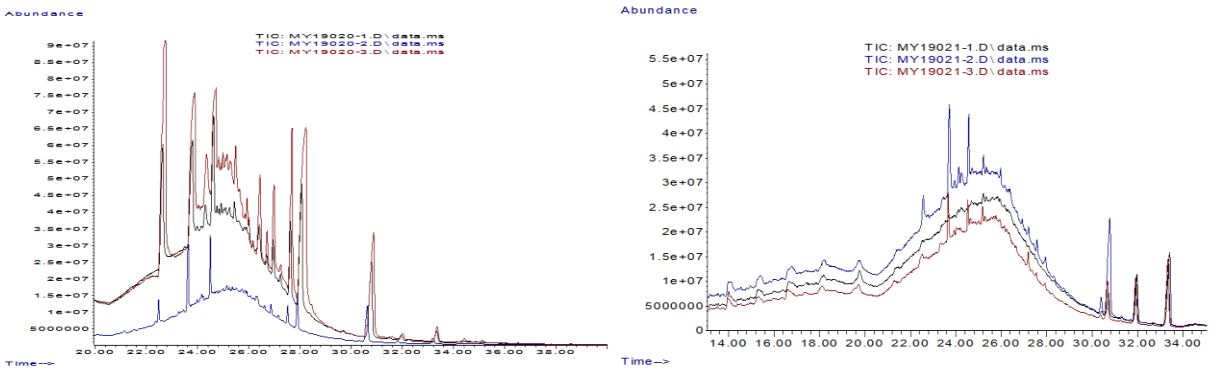
Table 2a: Concentrations of metals and metalloids (mg/kg dry weight) in subsamples of shredded plastic from the Jenjarom dumpsite in Malaysia, together with median and maximum values for each sample

Similarly, the differences between the 2 samples (MY19020 & MY19021) were relatively small. The mean concentrations for all metals other than molybdenum in sample MY19020 were between 0.7 and 1.3 times their respective values for MY19021.

Given that the concentrations of metal and metalloids were analysed in heterogeneous mixtures, it is possible that some of the individual plastic fragments within a mixture contain considerably higher concentrations than those reported for the subsamples.

A wide range of metals and metalloids, including those identified in the plastic samples, are present in various types of plastics, either remaining from manufacturing processes, or additives intentionally incorporated into plastic formulations such as fillers, stabilisers, pigments or flame retardants (Hahladakis *et al*. 2018, Jaffe & East 2007, Matthews 1996).

In a similar way to the metals analysis of these two shredded plastic samples, analysis of organic chemicals was carried out in triplicate for each sample. The profiles of isolated organic compounds were similar for each set of triplicates and differ only slightly in the apparent relative abundance of the compounds – see Figure below.



Both samples (MY19020 & MY19021) contained various flame retardants (FRs) including a range of highly brominated diphenyl ethers (PBDEs): from hepta- to decabrominated congeners in sample MY19020, and from octa- to decabrominated congeners in sample MY19021. Both samples also contained isomers of the highly chlorinated FR Dechlorane. In addition, sample MY19020 contained two additional brominated FRs: 1,2-bis(2,4,6-tribromophenoxy)ethane (BTBPE) and decabromodiphenyl ethane (DBDPE). Plasticizers were also identified in both samples, including two phthalate esters (di(2-ethylhexyl) 1,2-benzenedicarboxylate, and diundecyl 1,2-benzenedicarboxylate) and one terephthalate (di(2-ethylhexyl) 1,4-benzenedicarboxylate). Sample MY19021 alsocontained several long chain aliphatic hydrocarbons and 1,6-dimethyl-4-(1-methylethyl) naphthalene, a polycyclic aromatic hydrocarbon (PAH).

Pulau Indah

The sample of burned plastic and ash (MY19007) contained a very similar range of metals and metalloids at high concentrations as those found in the shredded plastic samples from the Jenjarom dumpsite (MY19020 & MY19021). Concentrations of antimony, cadmium and zinc in the burned

plastic and ash (MY19007) were similar to those found in the shredded plastic, while those of lead and tin were 3-5 times lower than median shredded plastic sample concentrations. Concentrations of copper and molybdenum in MY19007 were above typically levels in uncontaminated environmental matrices such as soil, though between 20 to 30 times lower than concentrations in the shredded plastic samples.

Sample code	MY19006	MY19007	MY19008
Site	Sungai Petani	Pulau Indah	
Location	river bank	dumpsite	fishpond
Type	soil	ash/burned plastic	sediment
Aluminium	23200	6560	55200
Antimony	0.5	50.6	73.1
Arsenic	16.5	14.1	18.3
Barium	123	652	26.3
Beryllium	0.8	0.3	0.3
Cadmium	0.27	11.4	0.44
Chromium	29.4	84.4	151
Cobalt	16.1	10.1	2.3
Copper	48.3	224	2920
Iron	13700	18000	5030
Lead	18	710	33.3
Manganese	488	211	1050
Mercury	<0.1	0.3	0.1
Molybdenum	0.79	7.96	4.69
Nickel	10.8	85.3	754
Strontium	52.1	34.5	116
Tin	4.56	65.2	8.48
Titanium	186	198	71
Vanadium	24.1	11.4	14.3
Zinc	133	4880	198

Table 2b: Concentrations of metals and metalloids (mg/kg dry weight) in samples of soil, sediment or ash/burned plastic from dumpsites in Malaysia

For metals and metalloids bound within a plastic, burning of the plastic can expose them to environmental weathering, and convert the metals and metalloids into different chemical forms which can be more mobile in the environment.

Though composed of a different type of material, comparison of the composition of the burned plastic and ash sample with concentrations in uncontaminated soil can provide useful context given the location from where this sample was collected (surface layer on ground). Antimony, cadmium, lead and zinc concentrations were between 10 and 50 times higher than the upper end of the concentration ranges typically found in uncontaminated soil, while those of copper, tin and molybdenum were between 4 and 7 times their respective upper uncontaminated soil concentrations (Alloway 1990, ATSDR 2004a, 2005b, 2012, 2019, Salomons & Forstner 1984).

In addition to the metals and metalloids, 22 organic compounds were reliably identified in the burnt plastic sample MY19007, mainly represented by long chain aliphatic hydrocarbons, which are common products of plastics thermal decomposition (Simoneit et al., 2005; Tulashie et al., 2019). Three further chemicals identified in this sample (1,1':3',1''-terphenyl, 5'-phenyl-; benzene, 1,1'-(1,3-propanediyl)bis-; and 1H-indole, 2-methyl-3-phenyl-) can be formed during plastics pyrolysis (Hamidi, 2013; Miskolczi and Ateş, 2016; Tulashie *et al*., 2019). In addition, the eight polycyclic aromatic hydrocarbons (PAHs) that were identified as trace contaminants in this sample may also result from incomplete plastics burning.

Sample code	MY19001	MY19003	MY19009	MY19012	MY19015	MY19018
Site	Klang, Selangor, Kapar - Jalan Pasan Malam	Sungai Petani	Pulau Indah			Pulau Indah, Sungai Chandong
Location	creek	Sungai Muda river	fish pond	channel to fish pond	ditch by dumpsite	channel at site
Aluminium	42	574	33	22	25	6
Antimony	1.6	<0.3	9.4	20.3	0.7	0.6
Arsenic	2.3	1.0	3.0	<0.5	6.9	1.8
Barium	47.0	16.8	12.3	59.0	20.4	70.4
Beryllium	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3
Cadmium	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Chromium	1.5	0.4	0.7	<0.1	0.2	0.5
Cobalt	0.6	<0.1	<0.1	0.9	0.1	0.2
Copper	0.9	0.9	14	165	7.2	0.9
Iron	2210	308	<4	<4	270	11
Lead	0.8	0.4	<0.1	0.2	1.3	<0.1
Manganese	178	1.4	5.9	2100	56.1	414
Mercury	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Molybdenum	8.0	0.2	5.0	0.4	2.0	93.2
Nickel	1.7	0.6	139	1720	1.7	1.0
Strontium	77.3	14.5	4460	157	62.8	503
Tin	0.6	<0.2	0.4	0.5	<0.2	<0.2
Titanium	2	10	<1	<1	<1	<1
Vanadium	1.3	0.7	0.6	<0.2	1.0	0.4
Zinc	2	<2	3	7	39	<2

Table 2c: Concentrations of metals and metalloids (µg/l) in filtered samples of surface water from plastic waste dumpsites in Malaysia

Water from a fish pond located close to the plastic waste dumpsite and a plastic waste recycling factory (MY19009) contained elevated concentrations of antimony (9.4 µg/l), nickel (139 µg/l), and to a lesser extent copper (14 µg/l). Background concentration of soluble metals in uncontaminated surface waters can vary significantly between locations. Levels are typically below 1 µg/l for antimony, below 20 µg/l for nickel and below 10 µg/l for copper, though copper and nickel concentrations can often be far lower in some locations (ATSDR 2004a, ATSDR 2005a, ATSDR 2019, Flem *et al*. 2018).

Water from a channel flowing into this fish pond (MY19012) also contained elevated concentrations of antimony, copper and nickel. The concentration of antimony was over double that in the fish pond water (MY19007), while those of copper and nickel were 12 times higher.

The channel water also contained a high concentration of manganese (2100 µg/l), a metal which is typically found in uncontaminated surface water at concentrations below 200 µg/l, and often far lower (Barceloux 1999, Flem *et al*. 2018). An elevated manganese concentration was not, however, found in the fish pond water (MY19009). Conversely, the fish pond water (MY19009), though not the channel water (MY19012), contained strontium at 4460 µg/l, a metal for which uncontaminated surface water concentrations are typically below 1000 µg/l, and often below 100 µg/l (ATSDR 2004b, Flem *et al*. 2018).

In contrast, water collected from a small ditch in front of dumpsite (MY19015) did not contain notable concentrations of any metals or metalloids.

None of the organic compounds identified in the burned plastic (MY19007) were identified in the water sample MY19012 collected from the channel leading to the fish pond. However, the other two water samples from this dumpsite, MY19009 (fish pond) and MY19015 (ditch), contain the following organic compounds that could potentially originate from plastics:

- TPPO, a flame retardant, was detected in both samples (and also in water sample MY19003 from the Sungai Muda River);
- 3-[1-(4-Cyano-1,2,3,4-tetrahydronaphthyl)]propanenitrile, one of the isomers of styrene-acrylonitrile trimer, was detected in both MY19009 and MY19012;
- Triphenylphosphine sulphide, a related compound to phosphorous-containing flame retardant TPPO, which is also used in organic synthesis and as a raw material in manufacture of flame retardants, catalysts, biocides, and extractants (Weferling *et al*., 2016) – detected in MY19009 only;
- 9-Octadecenamide, (Z)-, also called oleamide, was detected in sample MY19009 only. This chemical has a variety of industrial uses including as a slip agent, a lubricant, and a corrosion inhibitor. Oleamide has been found to leach out of plastics in laboratory experiments (Weferling *et al*., 2016).

No volatile organic compound were found in a separate sample of the ditch water (MY19017).

In freshwater environments, metals and metalloids, along with many organic chemicals which show poor water solubility, commonly accumulate in sediment over time. Sediment from the fish pond (MY19008) contained elevated concentrations of antimony, copper and nickel (Salomons & Forstner 1984), reflecting their high concentrations in the fish pond water (MY19009). Compared to typical background concentration ranges for freshwater sediments, the concentrations of antimony and copper in the sediment were over 50 times the upper end of their respective background ranges for freshwater sediments, while that of nickel was over 10 times the upper background concentration (ATSDR 2004a, 2005a, 2019, Salomons & Forstner 1984)

Sample Code	MY19003	MY19007	MY19008	MY19009	MY19015	MY19018	MY19020[a]	MY19021[a]
Sample type	**w**	**s**	**s**	**w**	**w**	**w**	**s**	**s**
STATISTICS FOR ISOLATED AND IDENTIFIED COMPOUNDS								
Number isolated	4	44	15	42	6	1	48	27
Number identified to >90%	2	22	6	4	2	1	19	20
Percentage identified to >90% (%)	50	50	40	10	33	100	40	74
NAME OF COMPOUNDS AND GROUPS IDENTIFIED TO >90%								
Aliphatic hydrocarbons	-	11	-	-	-	-	-	4
PAHs & derivatives	-	8b	1, 4[b]	-	-	-	-	1
PBDEs	-	-	-	-	-	-	12[b]	10[b]
Dechlorane	-	-	-	-	-	-	2	2
BTBPE	-	-	-	-	-	-	1	-
DBDPE	-	-	-	-	-	-	1	-
Phthalates	-	-	-	-	-	-	3	3
Decanedioic acid, dibutyl ester	1	-	-	-	-	-	-	-
Triphenylphosphine oxide (TPPO)	1	-	-	1	1	-	-	-
Triphenylphosphine sulfide	-	-	-	1	-	-	-	-
3-[1-(4-Cyano-1,2,3,4-tetrahydronaphthyl)]propanenitrile	-	-	-	1	1	-	-	-
9-Octadecenamide, (z)-	-	-	-	1	-	-	-	-
7,9-Di-tert-butyl-1-oxaspiro(4,5)deca-6,9-diene-2,8-dione	-	-	-	-	-	1	-	-
1,1':3',1''-Terphenyl, 5'-phenyl-	-	1	-	-	-	-	-	-
1H-Indole, 2-methyl-3-phenyl-	-	1	-	-	-	-	-	-
Benzene, 1,1'-(1,3-propanediyl)bis-	-	1	1	-	-	-	-	-

Table 3: Summary of results of organic compounds analysis in samples determined by GC/MS. w- water sample; s – solid sample; a – results for three replicates combined; b – compounds identified in selective monitoring mode (SIM) only; – not detected. Note: data for samples MY19001, MY19006, and MY19012 are not included in this table due to the absence of reliably identified compounds

Other than nickel, the metals and metalloids found in the fish pond and channel at high concentrations were also present at relatively high concentrations in plastic samples (MY19007, 20, 21). The source of the nickel in the fish pond may be independent of plastics recycling or disposal. Similarly, this may also be the case for manganese and strontium given the complexity of their distribution in water samples from the fish pond and the channel that flows into it.

Some of the same compounds that were found in the burned plastic sample (MY19007) were also identified in the fish pond sediment, but with fewer examples in the fish pond sediment. Those chemicals present in both the sediment and the burned plastic were 5 PAHs and 1,1'-(1,3-propanediyl)bis-benzene. None of these were identified in water from the fish pond (MY19009) nor the channel (MY19012), probably relating to their relatively low water solubility.

Samples of both the water in the fish pond (MY 19011) and water in the channel flowing to this pond (MY19014) contained a range of volatile organic compounds, including cyclohexane and a number of chlorinated alkenes, albeit at trace levels that could only be identified in the sensitive selective ion monitoring (SIM) mode on the GC-MS. The most distinguishable peaks in the channel water sample

(MY19014) were those for benzene, toluene, and o-xylene, which along with the identified m- and/or p- xylene, are part of the compound group referred to as BTEX, important petrochemical materials while also a group of common Products of Incomplete Combustion (PICs) (Zhang et.al. 2017).

Overall, therefore, there were some similarities between the chemicals identified in samples from the fish pond and channel and those identified in the burned plastic sample collected from the nearby dumpsite. Nevertheless, given the industrial nature of the zone from which these samples were collected, the possibility cannot be excluded that other industrial sources of these contaminants upstream from the pond, including other plastics storage and recycling facilities in the area, might be important contributors to the contamination found in the channel and pond. Further investigations would be necessary in order to try to confirm likely sources and their relative contributions.

Pulau Indah, Sungai Chandong

Water collected from a channel at this waste burning site (MY19018) contained a high concentration of molybdenum (93.2 µg/l), notably higher than in other local surface waters samples (MY19001 & MY19003), and above typical levels uncontaminated surface water levels (which are usually below 10 µg/l and often far lower) (Flem *et al*. 2018, Smedley & Kinniburgh 2017). A relatively high molybdenum concentration was also found in one of the shredded plastic samples (MY19021).

Only one organic compound was reliably identified in this water sample, 7,9-di-tert-butyl-1-oxaspiro(4,5)deca-6,9-diene-2,8-dione, which is a degradation product of antioxidant Irganox 1010 that is used for stabilizing polymers, particularly polyethylene and polypropylene (Félix et al., 2012; Lagacé et al., 2017; Simoneit et al., 2005)

Sungai Petani

River water from the Sungai Muda River at edge of dumpsite (MY19003) did not contain the quantified metals and metalloids at concentrations above typical ranges for uncontaminated surface waters, other than aluminium (Flem *et al*. 2018, Salomons & Forstner 1984).

For the sample of soil collected from the river bank at the same location (MY19006), the concentrations of metals and metalloids were well within the broad 'typical' ranges reported for uncontaminated soils globally (Alloway 1990, Salomons & Forstner 1984, Ure & Berrow 1982).

A limited number of compounds were isolated from both the river water and soil samples (4 and 9 compounds, respectively), but it was only possible to reliably identify compounds in the water sample MY19003. Those identified were represented by dibutyl ester of decanedioic acid and triphenyl phosphine oxide (TPPO). The former compound, also called as dibutyl sebacate, is a plasticizer used in a variety of plastics (Sheftel 2000) including those intended for contact with food and drink (Lahimer *et al* 2013). TPPO is organophosphorus compound that is used in organic synthesis and also can be used as a flame retardant (Liaw *et al*., 2017; Weferling *et al*., 2016).

No VOC compounds were identified in the water sample from the Sungai Muda River at edge of dumpsite (MY19005)

Klang, Selangor, Kapar - Jalan Pasan Malam

The creek water (MY19001) did not contain the quantified metals and metalloids at concentrations above typical ranges for uncontaminated surface waters, other than iron and to a lesser extent manganese (Flem *et al*. 2018, Salomons & Forstner 1984). No organic compounds were isolated from this sample.

Overall, this investigation has demonstrated that shredded plastic disposed of at some dumpsites in Malaysia contain a range of metals, metalloids and organic chemicals, including persistent organic pollutants (POPs), and are likely to be contaminating the surrounding environments during their storage or processing/recycling. In locations where such plastics are burned, the post-burning residues can contain many of these contaminants, some in forms that are likely to be more mobile compared to the source plastics, as well as additional chemicals generated during the combustion. There is evidence that surface waters adjacent to, or downstream of, some of the plastics disposal or processing sites investigated in this study are contaminated with chemicals which may have originated from the plastics at these sites or equivalent operations in the same areas.

References

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Appendix 1: Details of methodologies

Analysis for extractable organic compounds

Preparation

20 µg of deuterated naphthalene was added as an Internal Standard (IS) to each portion of sample that was subject to extraction.

Solid sample extraction: 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, ethyl acetate and ethanol in a ratio of 6:3:1, and at a temperature of 100^{o}C. Obtained extracts were concentrated to a volume of 3ml with a stream of clean nitrogen and cleaned up prior to analysis.

For the clean-up stage, each extract was vortexed for 1 min with 3 ml of concentrated sulfuric acid. The pentane phase was collected and eluted through a Florisil column, using a 95:5 pentane:toluene mixed eluent resulting in about 50ml of the extract. The cleaned extract was concentrated to a final volume of 1ml. 20 µg of Bromonaphthalene was added to each extract as a second IS prior to GC-MS analysis.

Water sample extraction: 500ml of sample were prepared using solid phase extraction technique with Dionex AutoTrace workstation, eluting solvents were 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.

GC/MS analysis

For the total organic compounds screening, samples were analysed using an Agilent 6890 Series II GC with Restek Rtx-17Sil column (30m, 0.25mm ID, 0.25 µm film thickness) linked to an Agilent 5975B Inert MSD operated in EI mode and interfaced with an Agilent Enhanced Chem Station data system. Total Ion chromatograms (TIC) were obtained simultaneously with the chromatograms of target compounds using Selective Ion Monitoring (SIM) mode. 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 5 min), then to 325^{0}C at 50^{0}C /min (held for 4 min), finally raised to 330^{0}C at 50^{0}C/min. The carrier gas was helium, supplied at 1ml/min. Identification of compounds was carried out by matching spectra obtained during analysis against both the Wiley W10N11 and Pesticides Libraries, and against spectra obtained for target compounds (see Table A1) using expert judgment as necessary in order to avoid misidentifications.

Retention time, min	Compound Name	Ions monitored
4.78	Benzene, 1,3-dichloro-	**146, 148, 111**
4.95	Benzene, 1,4-dichloro-	146, 148, 111
5.29	Benzene, 1,2-dichloro-	146, 148, 111
5.37	Ethane, hexachloro-	**117, 119,** 201
6.05	Benzene, 1,3,5-trichloro-	**180, 182,** 184
6.54	Butadiene, hexachloro-	**225, 227,** 223
6.64	Benzene, 1,2,4-trichloro-	180, 182, 184
7.03	Naphthalene	**128,** 129, 127
7.08	Benzene, 1,2,3-trichloro-	180, 182, 184
7.62	Hexachlorcyclopentadiene	**239, 235**
8.02	Benzene, 1,2,3,5-tetrachloro-	**216, 214,** 218
8.07	Benzene, 1,2,4,5-tetrachloro-	216, 214, 218
8.82	Benzene, 1,2,3,4-tetrachloro-	216, 214, 218
9.10	Naphthalene, 1-chloro-	**162, 127,** 164
10.34	Acenaphthylene	**152, 151,** 153
10.37	Dimethyl phthalate	**163,** 164, 77
10.43	Benzene, pentachloro-	**250, 252,** 248
10.48	Acenaphthene	**153, 154,** 152
10.61	4-tert-octyl phenol	**135,** 136, 134
11.11	Diethyl phthalate	**149,** 150, 177
11.14	Fluorene	**166, 165**
11.18-12.00	Nonylphenol, mix of isomeris	135, **121,** 149, 107, 163
11.90	Benzene, hexachloro-	**284, 286,** 282
13.36	Di-iso-butyl phthalate	149, 150 223
13.54	Phenanthrene	178, 176, 179
13.62	Anthracene	178, 176, 179
14.78	Di-n-butyl phthalate	149, 150
15.48	Bis(4-methyl-2-pentyl) phthalate isom. 1	149, 167, 150
15.54	Bis(4-methyl-2-pentyl) phthalate isom. 2	149, 167, 150
16.69	Bis(2-methoxyethyl) phthalate	**59, 58,** 149
17.32	Di-n-pentyl phthalate	**149,** 150, 237
17.85	Fluoranthene	**202,** 200, 101
17.98	Bis(2-ethoxyethyl) phthalate	**72, 45,** 149
19.00	Pyrene	202, 200, 101
20.17	Di-n-hexyl phthalate	149, 150, 251
20.37	BDE-17	**246, 248, 406**
20.85	BDE-28	246, 248, 406
21.69	PBEB	**485, 487, 500**
21.75	p,p'-DDT	**235, 237, 165**
22.21	Butyl benzyl phthalate	**149, 91, 150**
22.49	Bis(2-ethylhexyl)phthalate	149, **167,** 150
22.85	Bis(2-butoxyethyl) phthalate	149, **85,** 193
23.65	Bis(2-ethylhexyl) terephthalate	**70,** 149, 167
23.73	Dicyclohexyl phthalate	149, 167, 150

Retention time, min	Compound Name	Ions monitored
23.84	Benz[a]Anthracene	**228,** 226, 114
23.93	HBB	**225,550,554**
24.08	BDE-47	**326, 486,** 484
24.06	Chrysene	**228,** 226, 113
24.21	Di-n-octyl phthalate	**149, 150,** 167
24.39	BDE-66	**326, 324,** 328
25.34	Di-n-nonyl phthalate	149, 150, 167
25.56	BDE-100	**406, 404, 564**
25.95	BDE-99	404, 406, 564
26.25	Benzo[b]Fluoranthene	**252, 250, 253**
26.30	Benzo[k]Fluoranthene	252, 250, 253
27.00	BDE-85	404, 406, 564
27.17	BDE-154	**484, 482, 644**
27.15	Benzo[a]Pyrene	**252, 250, 126**
27.85	BDE-153	484, 482, 644
29.58	BDE-138	484, 486, 482
30.42	Indeno[123-cd]pyrene	**276, 277**
30.45	Dibenzo[a,h]anthracene	**278, 279**
31.43	BDE-183	**564, 562**, 722
31.76	Benzo[ghi]Perylene	276, 277
32.10	BTBPE	357,359,355
36.52	BDE-197	321, 322, 642
43.30	BDE-207	360, 361, 722
56.88	BDE-209	400, 399, 799
69.57	DBDPE	485,487,483

Table A1. List of target compounds monitored during SIM GC/MS analysis and their retention times.

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. Also, the field blank (unused sampling bottle of the type used to collect water samples) was checked for the presence of organic contaminants. For this, the bottle was filled with ultrapure deionised water and allowed to stand for 4 days, after which the water was treated as all other water samples. For comparison, a similar procedure was conducted with a sampling bottle chemically cleaned at GRL to check for potential contamination during the sample extraction process. Any background contaminants detected in blanks were subtracted from the chromatograms obtained for the samples before mass spectra interpretation.

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..

A 10ml portion from each water sample was sub-sampled into a 20ml headspace vial. They were analysed with the GC-MS in total ion monitoring (SCAN) mode to identify as many of the volatile organic compounds present 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 at the same time 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.

Quality control

A number of blanks of laboratory air and deionized water capped at the time that sub-sampling had taken place were analysed to confirm that any BTEX compounds identified were from the samples. Any background contaminants detected in blanks were subtracted from the chromatograms obtained for the samples before mass spectra were interpreted.

Analysis for metals

Preparation

For water samples, a portion of each sample was filtered through a 0.45 micron filter and then acidified by the addition of concentrated nitric acid to give a final concentration of 5% v/v. 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^{o}C over 20 minutes followed by holding at 180^{o}C for a further 20 minutes. Cooled digests were filtered prior to analysis.

For soil, sediment & ash/burned plastic samples, a representative portion of each sample was air dried to constant weight, homogenised, sieved through a 2mm mesh and then ground to a powder using a pestle and mortar. Approximately 0.25 g of the ground sample was accurately weighed and digested with 5.0 ml concentrated nitric acid and 0.5 ml concentrated hydrochloric acid, firstly overnight at room temperature then using microwave-assisted digestion with a CEM MARS Xpress system with temperature ramping: heating to 180^{o}C over 20 minutes, held at 180^{o}C for 20 minutes, heating to 210^{o}C over 20 minutes, held at 210^{o}C for 20 minutes. Following cooling, each digest solution was filtered and made up to 25 ml with deionised water. Prior to analysis, each digest solution was diluted 1:4 using deionised water.

For shredded plastic samples, a representative portion of each sample was air dried to constant weight and homogenised as far as possible. Three separate subsamples of approximately 1 g were accurately weighed from each sample digested with 10 ml concentrated nitric acid and 1.0 ml concentrated hydrochloric acid, firstly overnight at room temperature then using microwave-assisted digestion with a CEM MARS Xpress system with temperature ramping: heating to 100^{o}C over 10 minutes, held at 100^{o}C for 120 minutes, cooled and excess pressure released; heating to 125^{o}C over 10 minutes, held at 125^{o}C for 20 minutes, cooled and excess pressure released; heating to 150^{o}C over 10 minutes, held at 150^{o}C for 20 minutes, cooled and excess pressure released. 2 ml of hydrogen peroxide (30%) was added for 1 hour at room temperature, followed by heating to 170^{o}C over 10 minutes, held at 170^{o}C for 20 minutes, cooled and excess pressure released; heating to 200^{o}C over 10 minutes, held at 200^{o}C for 20 minutes. Following cooling, each digest solution was filtered and made up to 25 ml with deionised water. Prior to analysis, each digest solution was diluted 1:8 using 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, 1000 and 5000 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 1000 μg/l (Scandium, Germanium, Yttrium, Indium and Terbium). Any sample exceeding the calibration range was diluted accordingly, in duplicate, and re-analysed.

Quality control

The field blank (unused bottle of the type used to collect water samples) was filled with deionised water and allowed to stand for 4 days, after which the water was treated as all other water samples. No metals or metalloids were detected in the water stored in the field blank bottle, other than a trace of manganese (5 μg/l).

One water sample and one soil sample were prepared for ICP analysis in duplicate and analysed to verify method reproducibility. A blank sample was also prepared with each set of samples (water, solids, shredded plastic). For water samples, a mixed metal quality control solution of 80 μg/l for each metal, other than mercury at 4 μg/l, was digested and analysed in an identical manner. To check method efficiency for solid samples, two certified reference material (CRM) were prepared in an identical manner; GBW07406 (NCS DC73324), soil reference material certified by the China National Analysis Centre for Iron and Steel, Beijing, China; EC681K, low density polyethylene certified by the Institute for Reference Materials and Measurements (IRMM).

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.

Further details on analytical procedures and quality controls can be provided on request.

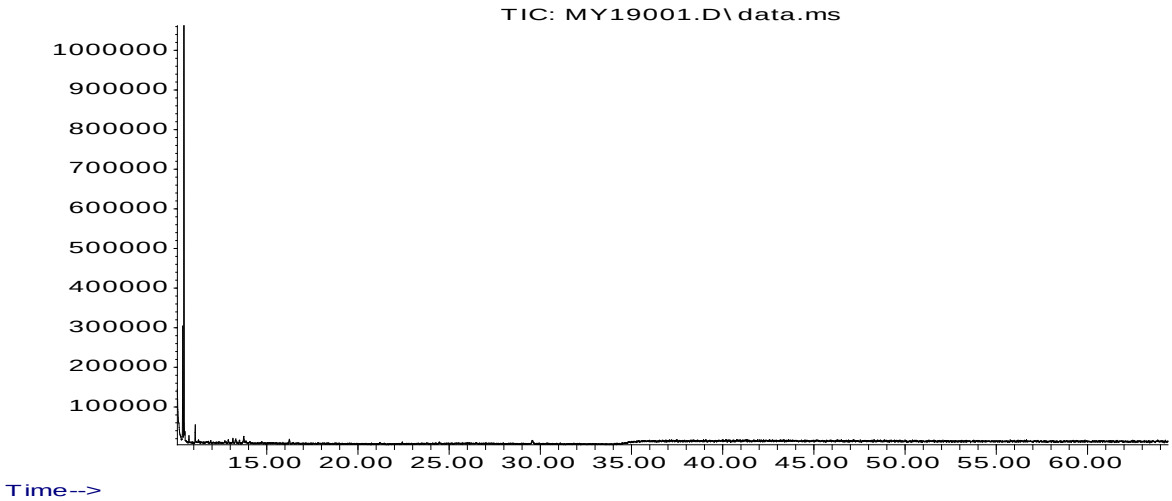
Appendix 2: Detailed semi-volatile organic (sVOCs) and volatile organic (VOCs) chromatograms and analytical screening data

Chromatograms and detailed screening data arising from GC-MS analysis of all samples are presented below.

Note: Some compounds have been identified only at trace levels using Selective Ion Monitoring (SIM) method, which is indicated below next to the name of such compounds.

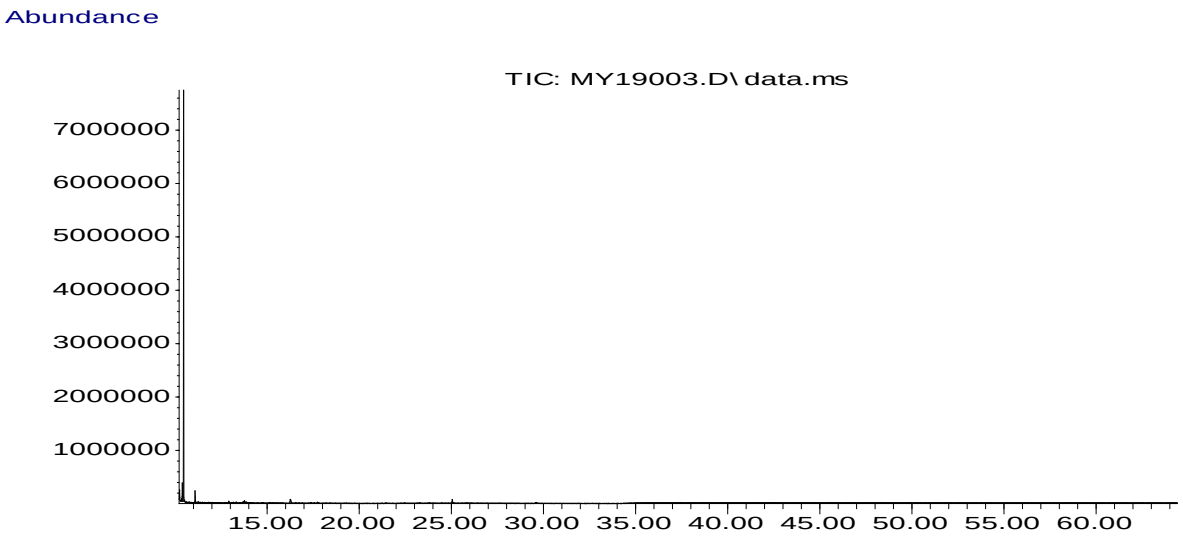
Semi-volatile organic analysis results (sVOCs)

Sample code	MY19001
Location	Klang, Selangor, Kapar - Jalan Pasan Malam
Sample type	Water (sVOC)
Date	29.07.2019
Description	Creek by plastics recycling facility



Number of compounds isolated: None

Sample code	MY19003
Location	Sungai Petani unregulated dumpsite
Sample type	Water (sVOC)
Date	31.07.2019
Description	Sungai Muda River at edge of dumpsite.



Number of compounds isolated: 4

Compounds identified to better than 90%:

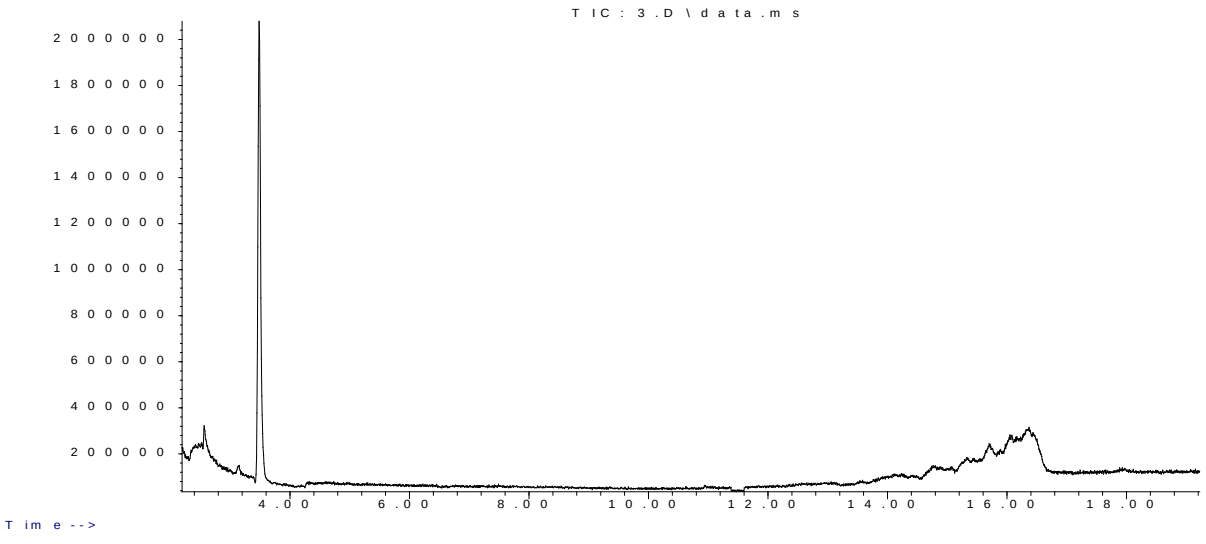
1. **In total ion chromatogram (TIC)**

CAS#	**Name**
000109-43-3	Decanedioic acid, dibutyl ester
000791-28-6	Phosphine oxide, triphenyl-

2. **In selective ion monitoring mode (SIM)**

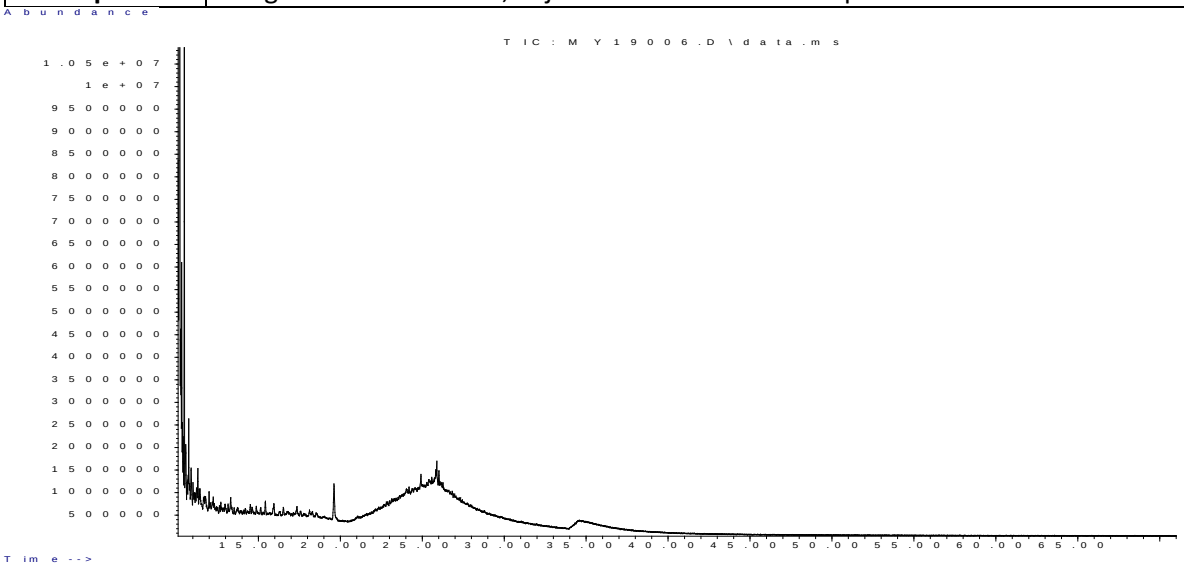
None

Sample code	MY19005
Location	Sungai Petani unregulated dumpsite
Sample type	Water (VOC)
Date	31.07.2019
Description	Sungai Muda River at edge of dumpsite.



Number of compounds isolated: 0

Sample code	MY19006
Location	Sungai Petani unregulated dumpsite
Sample type	Soil (sVOC)
Date	31.07.2019
Description	Sungai Muda river bank, adjacent to river water sample



Number of compounds isolated: 9

Compounds identified to better than 90%:

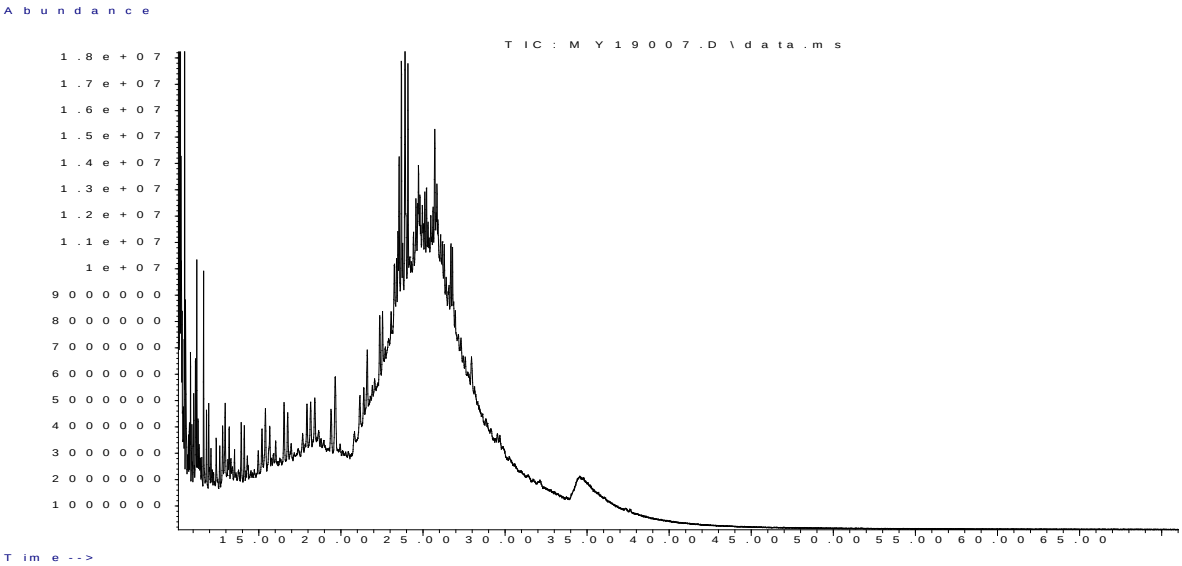
1. **In total ion chromatogram (TIC)**

None

2. **In selective ion monitoring mode (SIM)**

None

Sample code	MY19007
Location	Pulau Indah dumpsite, Klang
Sample type	Ash/BP (sVOC)
Date	06.08.2019
Description	partially burned material from unregulated dumpsite



Number of compounds isolated: 44

Compounds identified to better than 90%:

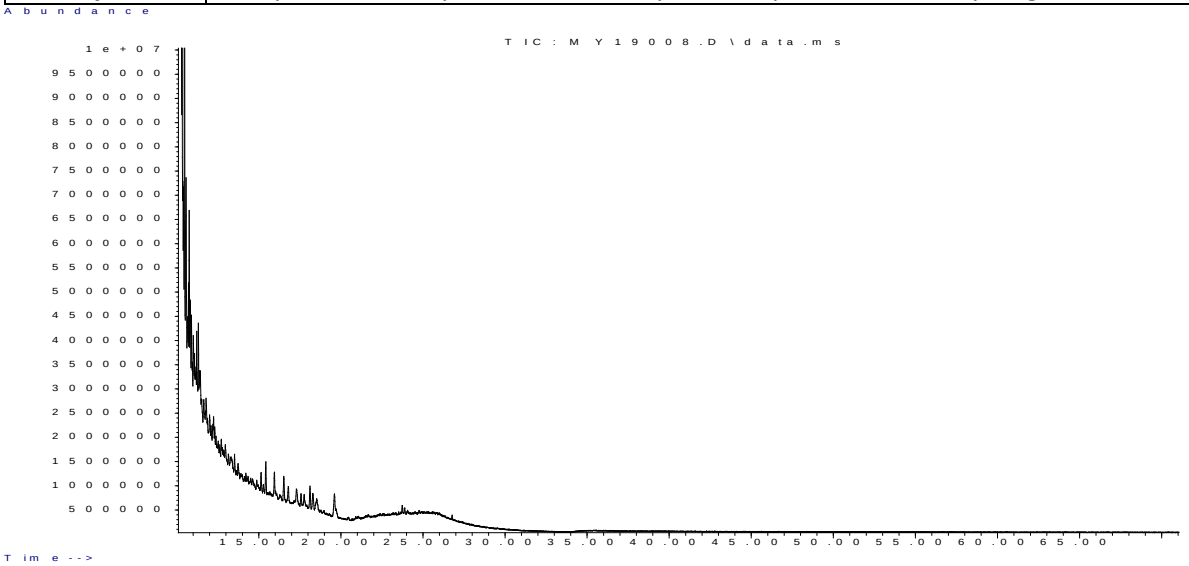
2. In total ion chromatogram (TIC)

CAS#	Name
000612-71-5	1,1':3',1''-Terphenyl, 5'-phenyl-
018835-33-1	1-Hexacosene
018435-45-5	1-Nonadecene
004757-69-1	1H-Indole, 2-methyl-3-phenyl-
027519-02-4	9-Tricosene, (z)-
001081-75-0	Benzene, 1,1'-(1,3-propanediyl)bis-
000629-92-5	Nonadecane
000112-95-8	Eicosane
000629-94-7	Heneicosane
000629-97-0	Docosane
000638-67-5	Tricosane
000646-31-1	Tetracosane
000629-99-2	Pentacosane
630-02-4	Octacosane

3. In selective ion monitoring mode (SIM)

000086-73-7	9H-Fluorene
000085-01-8	Phenanthrene
000129-00-0	Pyrene
000206-44-0	Fluoranthene
000207-08-9	Benzo[k]fluoranthene
000205-99-2	Benzo[b]fluoranthene
000050-32-8	Benzo[a]pyrene
000191-24-2	Benzo[ghi]perylene

Sample code	MY19008
Location	Pulau Indah dumpsite, Klang
Sample type	Sediment (sVOC)
Date	06.08.2019
Description	Fish pond close to plastic waste dumpsite and plastic waste recycling factories



Number of compounds isolated: 15

Compounds identified to better than 90%:

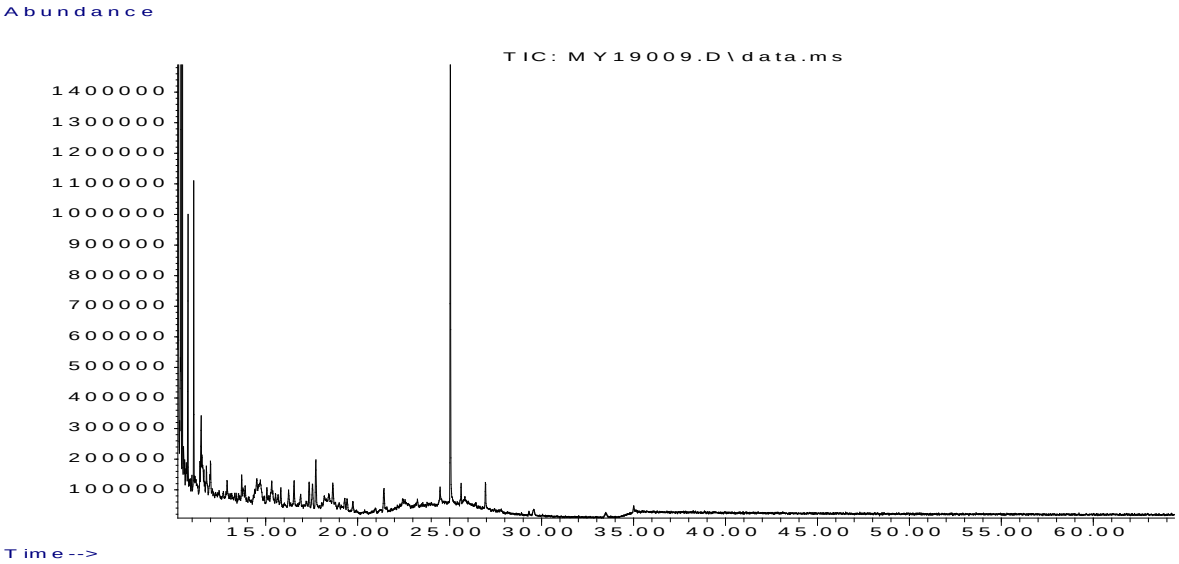
1. **In total ion chromatogram (TIC)**

CAS#	**Name**
001081-75-0	Benzene, 1,1'-(1,3-propanediyl)bis-
000085-01-8	Phenanthrene

2. **In selective ion monitoring mode (SIM)**

000050-32-8	Benzo[a]pyrene
000218-01-9	Chrysene
000206-44-0	Fluoranthene
000129-00-0	Pyrene

Sample code	MY19009
Location	Pulau Indah dumpsite, Klang
Sample type	Water (sVOC)
Date	06.08.2019
Description	Fish pond, as MY19008



Number of compounds isolated: 42

Compounds identified to better than 90%:

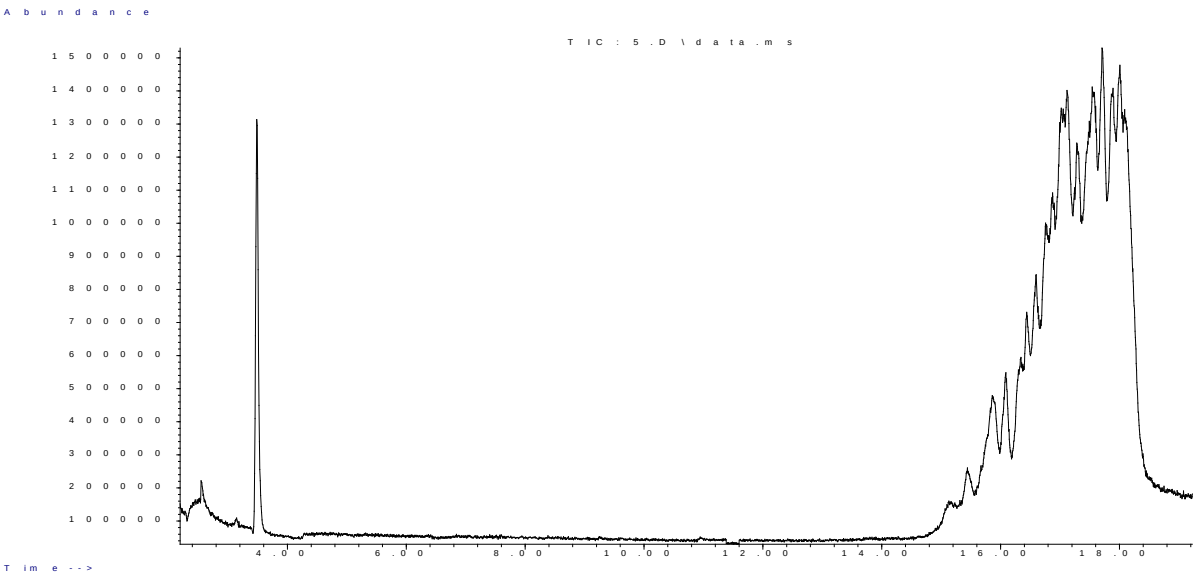
1. **In total ion chromatogram (TIC)**

CAS#	**Name**
057964-40-6	3-[1-(4-Cyano-1,2,3,4-tetrahydronaphthyl)]propanenitrile
000301-02-0	9-Octadecenamide, (z)-
000791-28-6	Phosphine oxide, triphenyl-
003878-45-3	Triphenylphosphine sulfide

2. **In selective ion monitoring mode (SIM)**

None

Sample code	MY19011
Location	Pulau Indah dumpsite, Klang
Sample type	Water (VOC)
Date	06.08.2019
Description	Fish pond, as MY19008

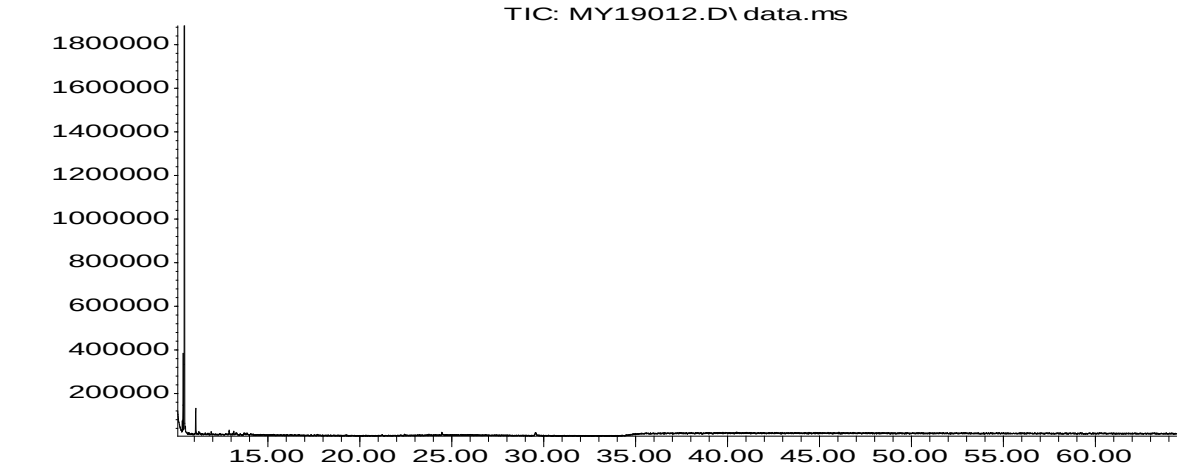


Number of compounds isolated: 25

Compounds identified to better than 90%:

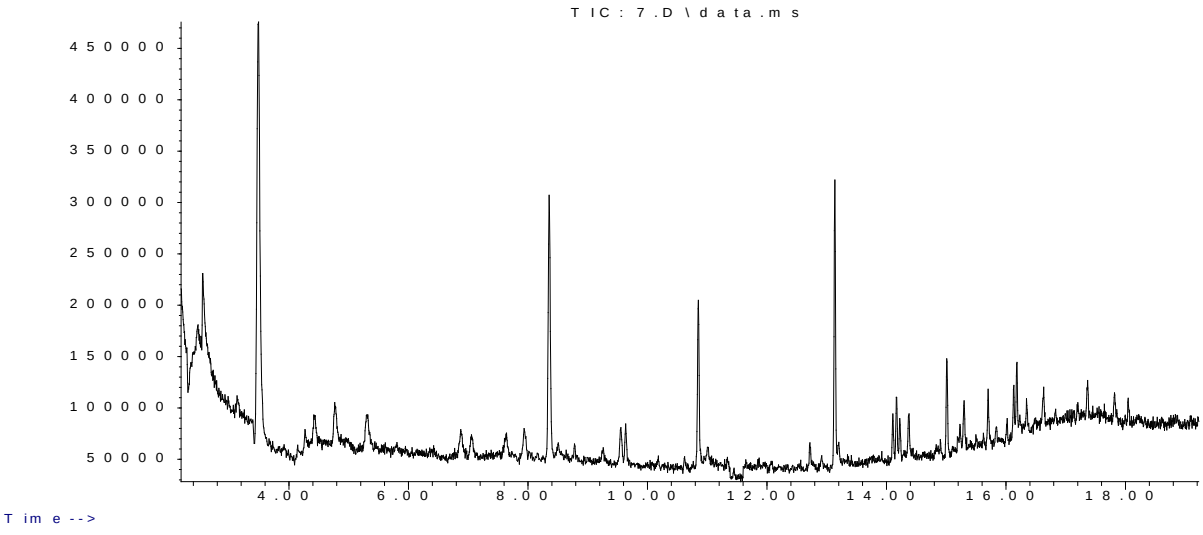
CAS#	**Name**
000110-82-7	Cyclohexane (SIM)
000156-59-2	Ethene, 1,2-dichloro-,cis- (SIM)
000079-01-6	Ethene, trichloro- (SIM)
000127-18-4	Ethene, tetrachloro- (SIM)
000075-09-2	Methane, dichloro- (SIM)

Sample code	MY19012
Location	Pulau Indah dumpsite, Klang
Sample type	Water (sVOC)
Date	06.08.2019
Description	Channel flowing into fish pond (MY19009), close to plastic waste dumpsite & downstream of 1 large & several smaller plastic waste recycling factories



Number of compounds isolated: 0

Sample code	MY19014
Location	Pulau Indah dumpsite, Klang
Sample type	Water (VOC)
Date	06.08.2019
Description	Channel flowing into fish pond (MY19009), close to plastic waste dumpsite & downstream of 1 large & several smaller plastic waste recycling factories

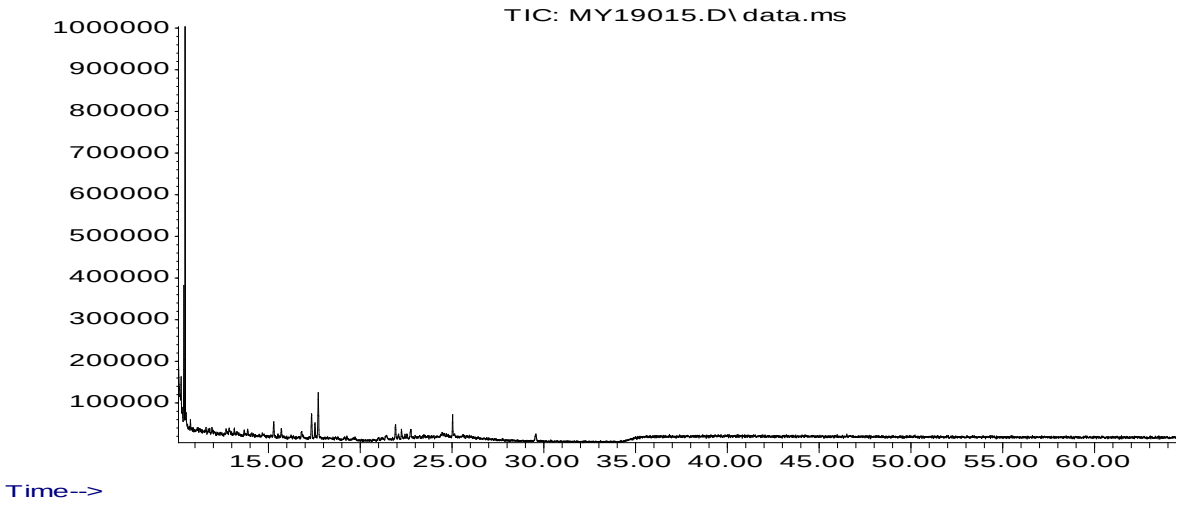


Number of compounds isolated: **25**

Compounds identified to better than 90%:

CAS#	Name
000071-43-2	Benzene
026545-61-9	Benzene, bis(trifluoromethyl)-
000156-59-2	Ethene, 1,2-dichloro-,cis-
000108-38-3	M- / p- xylene
000095-47-6	O-xylene
000108-88-3	Toluene
000075-15-0	Carbon disulphide (SIM)
000110-82-7	Cyclohexane (SIM)
000108-87-2	Cyclohexane, methyl- (SIM)
000156-60-5	Ethene, 1,2-dichloro-, trans- (SIM)
000079-01-6	Ethene, trichloro- (SIM)
000127-18-4	Ethene, tetrachloro- (SIM)
000075-09-2	Methane, dichloro- (SIM)

Sample code	MY19015
Location	Pulau Indah dumpsite, Klang
Sample type	Water (sVOC)
Date	06.08.2019
Description	Small ditch in front of dumpsite



Number of compounds isolated: **6**

Compounds identified to better than 90%:

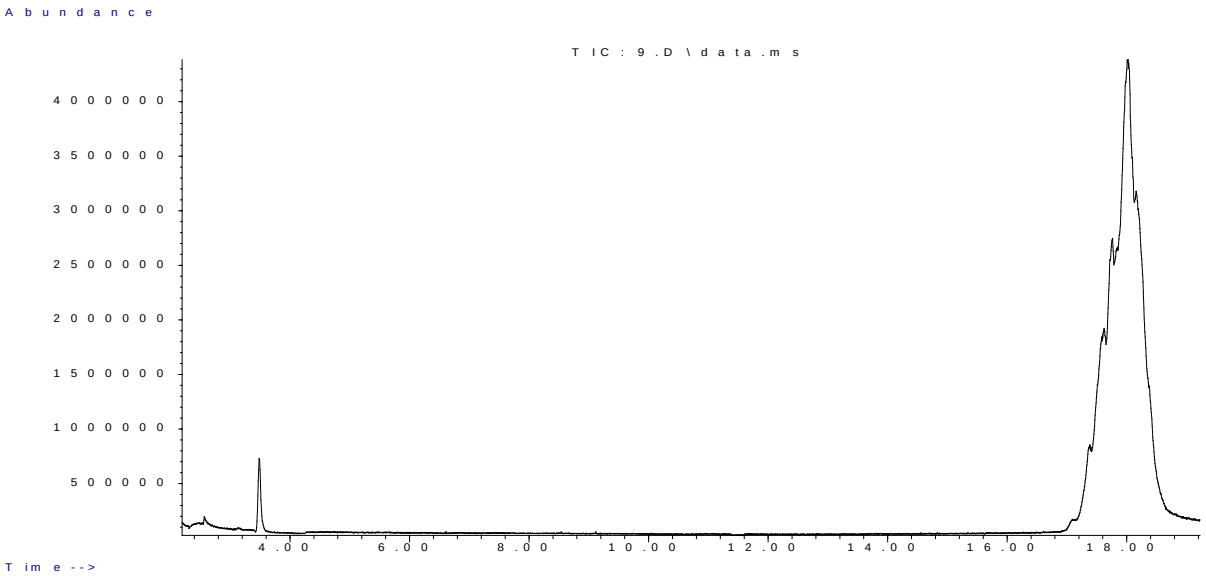
1. **In total ion chromatogram (TIC)**

CAS#	Name
057964-40-6	3-[1-(4-Cyano-1,2,3,4-tetrahydronaphthyl)]propanenitrile
000791-28-6	Phosphine oxide, triphenyl-

2. **In selective ion monitoring mode (SIM)**

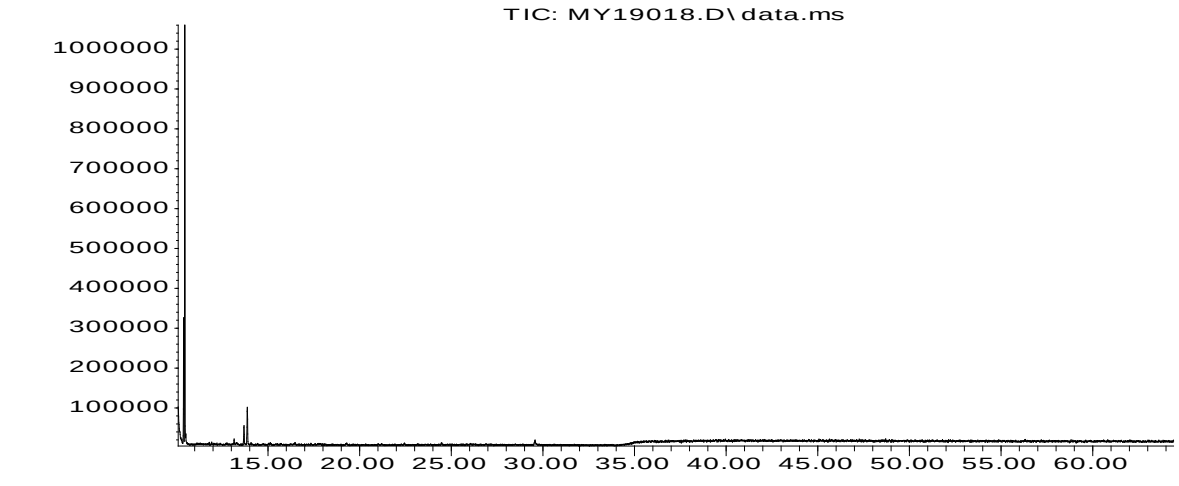
None

Sample code	MY19017
Location	Pulau Indah dumpsite, Klang
Sample type	Water (VOC)
Date	06.08.2019
Description	Small ditch in front of dumpsite



Number of compounds isolated: 0

Sample code	MY19018
Location	Pulau Indah, Sungai Chandong
Sample type	Water (sVOC)
Date	07.08.2019
Description	Channel at waste burning site



Number of compounds isolated: 1

Compounds identified to better than 90%:

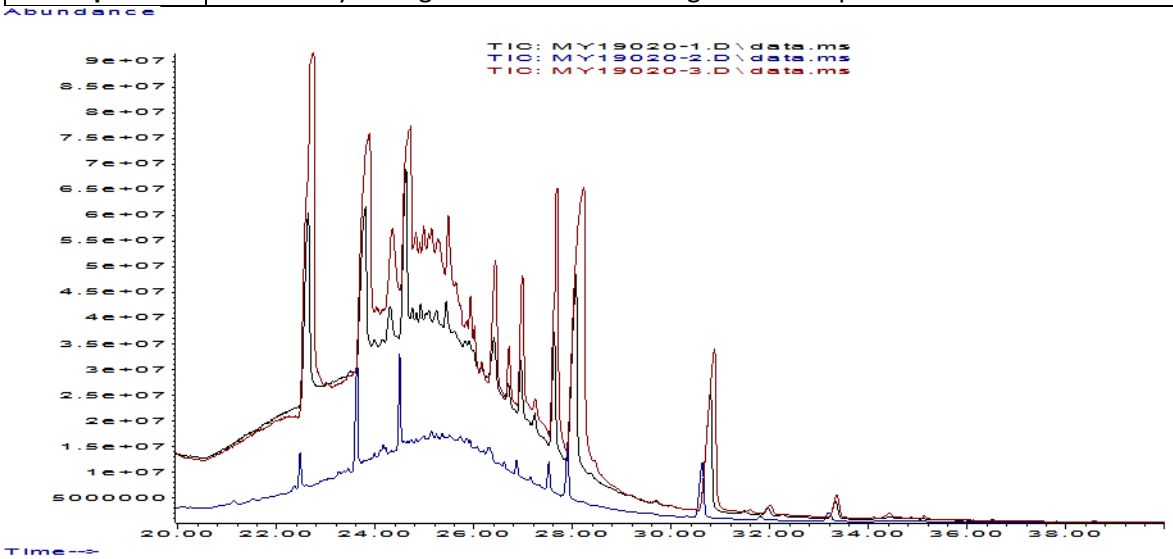
1. **In total ion chromatogram (TIC)**

CAS#	Name
082304-66-3	7,9-Di-tert-butyl-1-oxaspiro(4,5)deca-6,9-diene-2,8-dione

2. In selective ion monitoring mode (SIM)

None

Sample code	MY19020 (results for three replicates combined)
Location	Jenjarom dumpsite, Sri Cheeding
Sample type	shredded plastic (sVOC)
Date	07.08.2019
Description	Surface layer on ground at former unregulated dumpsite



Number of compounds isolated: up to 48 (in MY19020 replicate 3)

Compounds identified to better than 90%:

1. In total ion chromatogram (TIC)

CAS#	Name
000117-81-7	1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester
003648-20-2	1,2-Benzenedicarboxylic acid, diundecyl ester
006422-86-2	1,4-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester
037853-59-1	1,2-bis(2,4,6-tribromophenoxy)ethane (BTBPE)
000000-00-0	Dechlorane, 2 isomers*

2. In selective ion monitoring mode (SIM)

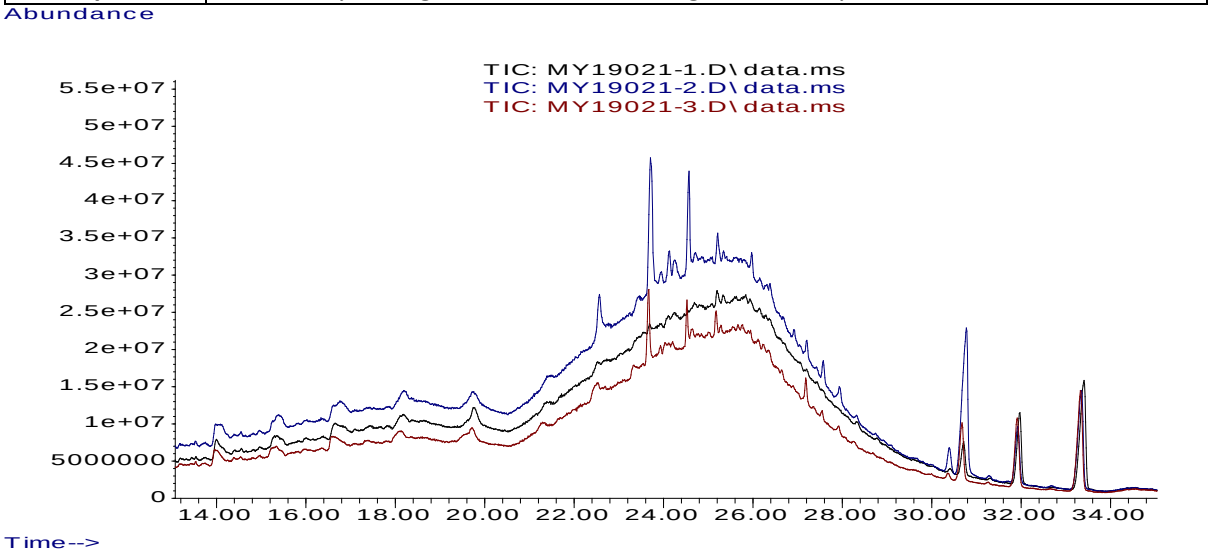
CAS#	Name
084852-53-9	Decabromodiphenyl ethane (DBDPE)**
000000-00-0	Diphenyl ether, heptabromo-, up to 5 isomers including BDE-183
000000-00-0	Diphenyl ether, octabromo-, up to 3 isomers including BDE-197
000000-00-0	Diphenyl ether, nonabromo-, 3 isomers including BDE-206,207&208
000000-00-0	Diphenyl ether, decabromo- (BDE-209)

* The isolation of two isomers of Dechlorane from the sample strongly suggest the presence of Dechlorane Plus flame retardant.

** Identified only in one of the triplicates.

Note: Triplicates contained mainly identical compounds and differ in their abundance (see below). Chromatograms also contained several unidentified peaks that showed fragmentation characteristic for polyhalogenated compounds.

Sample code	MY19021 (results for three replicates combined)
Location	Jenjarom dumpsite, Sri Cheeding
Sample type	shredded plastic (sVOC)
Date	07.08.2019
Description	Surface layer on ground at former unregulated dumpsite



Number of compounds isolated: **up to 27 (in MY19021 replicate 2)**

Compounds identified to better than 90%:

1. **In total ion chromatogram (TIC)**

CAS#	**Name**
000117-81-7	1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester
003648-20-2	1,2-Benzenedicarboxylic acid, diundecyl ester
006422-86-2	1,4-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester
000000-00-0	Dechlorane, 2 isomers*
000483-78-3	Naphthalene, 1,6-dimethyl-4-(1-methylethyl)-
000629-92-5	Nonadecane
000112-95-8	Eicosane
000629-94-7	Heneicosane
000629-97-0	Docosane

2. **In selective ion monitoring mode (SIM)**

CAS#	**Name**
000000-00-0	Diphenyl ether, octabromo-, up to 6 isomers including BDE-197
000000-00-0	Diphenyl ether, nonabromo-, 3 isomers (BDE-206, 207 & 208)
000000-00-0	Diphenyl ether, decabromo- (BDE-209)

* The isolation of two isomers of Dechlorane from the sample strongly suggest the presence of Dechlorane Plus flame retardant.

Note: Triplicates contained mainly identical compounds and differ in their abundance (see below). Chromatograms also contained several unidentified peaks that showed fragmentation characteristic for polyhalogenated compounds.