Greenpeace Research Laboratories Analytical Results 2016-02

Analysis of water samples collected from deep drilled wells, open wells, a local drinking water company, settling ponds, tap water and an artificial lake, in Kerta Buana village, Kutai Karta Negara, Indonesia.

21.03.2016

Introduction

A total of 24 water samples were received from Greenpeace Southeast Asia for analysis at the Greenpeace Research Laboratories on 25th November 2015. According to documentation supplied, all samples were collected between 21st and 22nd November 2015 from a number of locations in Kerta Buana village, Kutai Karta Negara, Indonesia.

These samples included water collected from 12 open wells, and from 5 deep drilled wells in the village, which is to the northeast of an active coal mine of PT Kitadin, in Kutai Karta Negara, Indonesia.

In the north part of the village there is an artificial lake which, according to information supplied to us, was a coal mine pit (operated between 2004 until 2006) and has reportedly been used by PDAM, the local drinking water company in the north of Kerta Buana village, as their water source by 2007. One water sample was collected from the lake close to the PDAM water extraction point. Treated water was also sampled from PDAM before being distributed to consumers as drinking water. In addition, two water samples were collected from local villagers' taps, which were supplied by PDAM. Local villagers have reported that the quality of the water provided from these taps is occasionally poor.

The remaining three samples were collected from three settling ponds, reported to be related to coal washing processes.

Details of the samples received, as provided to us by Greenpeace South East Asia, 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, according to the methods outlined below.

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Sample code	Sample type	Sample Description (transcribed from the information provided with the samples as received)	Date & Time (Indonesian Western Time) of sampling	Analysis Conducted
IDN15001	Open Well Water	Depth of water: 0.2m; depth of well: 1.47m. Well water is used for gardening.	21/Nov/2015 09.47	SVOCs(qual), metals (quant)
IDN15002		Depth of water: 0.61m; depth of well: 3.69m. Water colour: dark. Smelly.	21/Nov/2015 14.49	
IDN15003		Depth of water: 0.2m; depth of well: 0.91m. Water colour: Light Brown. Well water is used for cleaning and washing, sometime for cooking.	21/Nov/2015 15.16	
IDN15004		Depth of water: 3.99m; depth of well: 5.2m. Water colour: light brown. Well water is used for washing and cleaning. Close to the deep drilling well IDN15014	22/Nov/2015 10.28	
IDN15005		Depth of water: 0.8 m; depth of well: 2.11m. Water colour: dark. Smelly.	21/Nov/2015 14.21	
IDN15006		Depth of water: 1.2 m; depth of well: 1.64m. Water colour: light dark. Well water is used by 6 household for daily used, and getting wider likes pond.	21/Nov/2015 11.14	
IDN15007		Depth of water: 4.5m; depth of well: 5.8m. Water from the well is used for washing and cleaning. Water colour: Light dark and sandy.	21/Nov/2015 13.06	
IDN15008		Depth of water: 3.45m; depth of well: 4m. The active coal mine is 100 m from the well. Well not in use.	21/Nov/2015 09.03	
IDN15009		Depth of water: 0.3m; depth of well: 2.85m. Well water is used by 3 houses.	21/Nov/2015 10.27	
IDN15010		Depth of water: 0.5m; depth of well: 1.85m. Water colour: soft green	21/Nov/2015 13.25	
IDN15011		Depth of water: 0.9m; depth of well: 3.95m. The water is not for drink, but for wash only.	21/Nov/2015 12.41	
IDN15012		Depth of water: 0.3m; depth of well: 2.95m. Lots of dead mosquitos larvae in the water.	22/Nov/2015 11.25	
IDN15013	Deep Drilled Well Water	Water color: light brown. The well is still used for gardening. Depth unknown.	21/Nov/2015 10.08	
IDN15014		Depth of well: 46m	22/Nov/2015 10.41	
IDN15015		Depth of well: 19m	21/Nov/2015 13.57	
IDN15016		Depth of well: 24m	22/Nov/2015 09.34	
IDN15017		Depth of well: 12m	22/Nov/2015 10.04	

IDN15018	Lake Water	The artificial lake was formed on the site of an ex coal mine pit	21/Nov/2015 16.04	
IDN15019	Drinking Water	After the Treatment by PDAM (Local Drinking Water Company), and before being distributed to costumers	21/Nov/2015 16.26	
IDN15020	Tap Water	Tap water from PDAM; reported by local villagers that the water quality was bad and cannot be used for cooking	21/Nov/2015 17.45	
IDN15021		Tap water from PDAM, reported by local villager that sometimes the water can cause irritation to their skin	22/Nov/2015 08.46	
IDN15022	Water from Settling Pond	This settling pond is in the southeast part of the village. The water will run to the village before end in Separi River.	22/Nov/2015 08.20	
IDN15023		This settling pond is to the south west of the village. The water will go to Mahakam River	21/Nov/2015 17.17	
IDN15024		This settling pond is to the south west of the village. Coal washing water are treated with $Ca(OH)_2$ to neutralize the acidity, before go to Mahakam River.	22/Nov/2015 12.02	

Table 1a: details of samples received and analysed at the Greenpeace Research Laboratories

Sample code	N degree (º)	E degree (º)
IDN15001	-0.30817	117.10078
IDN15002	-0.30952	117.10118
IDN15003	-0.30872	117.10176
IDN15004	-0.30409	117.10053
IDN15005	-0.31746	117.10871
IDN15006	-0.31597	117.10020
IDN15007	-0.31230	117.11008
IDN15008	-0.31772	117.09457
IDN15009	-0.30792	117.10048
IDN15010	-0.30980	117.11282
IDN15011	-0.31341	117.10882
IDN15012	-0.31187	117.10761
IDN15013	-0.30818	117.10076
IDN15014	-0.30434	117.10030
IDN15015	-0.30811	117.11200
IDN15016	-0.30708	117.09726
IDN15017	-0.31110	117.10358
IDN15018	-0.29106	117.10973
IDN15019	-0.28241	117.11637
IDN15020	-0.30413	117.10192
IDN15021	-0.30845	117.10110
IDN15022	-0.31886	117.11247
IDN15023	-0.31794	117.07819
IDN15024	-0.31691	117.07833

Table 1b: GPS coordinated of sample collection locations

Materials and methods

Concentrations of metals and metalloids were determined for all samples by ICP mass spectroscopy (ICP-MS) following acid digestion and using appropriate intra-laboratory standards. 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. Hexavalent chromium concentrations in filtered samples were determined colourimetrically using a diphenylcarbazide method.

Semi-volatile organic compounds were isolated from each sample using solid phase extraction (SPE) technique with ethyl acetate, pentane and toluene as solvents. Extracted compounds were subsequently identified as far as possible using gas chromatography/mass spectrometry (GC/MS). More detailed descriptions of the sample preparation and analytical procedures are presented in the Appendix 1.

Results and Discussion

The results for the samples are outlined below. The concentrations of metals and metalloids in filtered (dissolved metals) and in whole waters (dissolved and suspended metals) are reported in Tables 2a & 2b. In addition, the semi-volatile organic chemicals identified in individual samples are summarised in Table 3. 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 detection for the analytical methods employed in this study. These are shown in the results tables as '<xx' where xx is the method detection limit for the individual metal or metalloid.

Metals

Open well water samples

All but two (IDN15007 & IDN15009) of the open well samples contained high concentrations of particulate bound iron, over 1000 μg/l in the whole (unfiltered) sample, with sample IDN15012 having by far the highest concentration (53800 μg/l). Concentrations of dissolved iron in these samples were considerably lower, with all but one sample (IDN15008, 1130 μg/l) having below 1000 μg/l dissolved iron.

Concentrations of manganese in the waters from the open wells ranged from 1-4350 μg/l (dissolved manganese), with a notably high concentrations for one sample (IDN15001, 4530 μg/l). Data for the corresponding whole samples demonstrated that manganese was predominantly present in dissolved forms in these samples. The sample with the highest concentration (IDN15001) was collected close to other open wells from which water have far lower manganese concentrations (IDN15002, IDN15003 & IDN15009).

High concentrations of particulate bound aluminium, though not dissolved aluminium, were found for five open well water samples (IDN15002, IDN15003, IDN15005, IDN15010 and IDN15012; 1550-4260 μg/l). There was no clear link between the location from which each sample was collected & the presence of a high concentration of particulate bound aluminium.

One sample (IDN15012) also contained a somewhat higher concentration of particulate bound arsenic in the whole (unfiltered) sample (30.9 μg/l), though this was not reflected in the dissolved concentration in the filtered sample.

Deep drill well water samples

One of the samples from a deep drill well (IDN15013), located adjacent to an open well that was also sampled (IDN15001), did not contain notable concentrations of the quantified metals/metalloids. This

is in contrast to the sample from the open well at the same location (IDN15001), which contained far higher concentration of manganese and higher concentrations of both iron and cobalt.

The other sample from a deep drill well located adjacent to an open well that was also sampled (IDN15014) contained similar concentrations to those found in the sample from the open well at the same location (IDN15004), including relatively high concentrations of manganese and iron. The dissolved iron concentration was substantially higher in the deep drill water sample (IDN15014, 2090 μg/l) compared to the equivalent open well (IDN15004, 184 μg/l).

None of the three other samples from deep drill wells (IDN15015 - IDN15017) contained notable concentrations of the quantified metals/metalloids, with the exception of particulate bound iron in one whole (unfiltered) sample (IDN15015, 2820 μg/l) and to a lesser extent particulate bound aluminium in another whole (unfiltered) sample (IDN15017, 3147 μg/l)

Treated water samples

Neither the sample of treated water collected from the PDAM facility (IDN15019), nor from the piped network supplied by the PDAM facility (IDN15020 & IDN15021), contained notable concentrations of the quantified metals/metalloids, with the exception of aluminium in each of the whole (unfiltered) samples (745 to 2160 μg/l), with notably lower concentrations of dissolved aluminium in each case (110-170 μg/l). The presence of aluminium in suspended particles may result from the use of aluminium compounds in water treatment. In the European Union (EU), a limit for aluminium in drinking water is set at 200 μg/l (EU 1998); the dissolved aluminium concentrations in the samples are lower than the EU limit, though the total aluminium concentrations (which includes aluminium bound in suspended particles) are higher than the EU limit, being between 3.7 and 10.8 times the EU limit.

Surface water samples

The sample of water from a lake used as a water source by PDAM (IDN15018) did not contain any of the quantified metals/metalloids at concentrations above levels typically found in uncontaminated surface freshwater, and contained notably lower concentrations of aluminium in the whole (unfiltered) sample compared to the treated water samples (IDN15019 - IDN15021).

Likewise, the samples from the settling ponds (IDN15022- IDN15024) did not contain any of the quantified metals/metalloids at concentrations above levels typically found in uncontaminated surface freshwater, though two of these samples (IDN150232 & IDN15023) contained somewhat higher concentrations of aluminium and iron in the whole (unfiltered) sample as a result of these metals being associated with suspended matter within the samples.

Organic contaminants

After analysis with GC-MS operated under the SCAN mode, organic compounds were detected in 15 out of the 24 samples (62.5%). All of these 15 water samples contained the phthalate DEP (Diethyl Phthalate), while DMP (Dimethyl phthalate) were identified in 6 of these 15 samples. The likely origins of these contaminants are unknown in this case, though DEP is a widely used solvent and additive in a range of industrial and consumer products. Several alkanes and an alkene (1-Nonadecene) were identified in two open well water samples (IND15008 & IND15009) as well as in the treated-drinking-water sample collected from the local drinking water company PDAM (IDN15019).

All of the samples were also analyzed for PAHs using GC-MS operated in selected ion monitoring (SIM) mode, with the retention time and spectra of any relevant peaks being matched against those obtained using the same method for a mixed standard of 16 PAHs. Only one sample (IDN 15010) was found to contain a trace amount of the PAH pyrene (not detectable above background in SCAN mode), yielding a peak area just over 5 times that found in the corresponding method (extraction) blank. This is not indicative of substantive contamination of the water with this group of persistent organic compounds.

Sample code	IDN15001		IDN15002		IDN15003		IDN15004		IDN15005		IDN15006		IDN15007		IDN15008		IDN15009		IDN15010		IDN15011		IDN15012	
	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh
Aluminium	12	465	75	4260	129	3710	6	271	90	1550	16	106	17	129	46	465	32	398	110	2990	27	725	<5	2110
Antimony	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Arsenic	0.9	1.6	0.6	2.2	0.5	1.3	0.6	1.3	0.4	1.0	0.6	0.8	1.5	2.0	1.4	2.2	0.2	0.3	0.9	3.3	2.0	6.2	1.4	30.9
Barium	171	185	23	49	52	84	58	64	27	35	76	100	17	22	70	82	35	39	11	28	52	60	66	151
Beryllium	<0.02	0.03	<0.02	0.14	<0.02	0.10	<0.02	0.06	<0.02	0.05	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	0.36	0.41	<0.02	0.14	<0.02	<0.02	<0.02	0.08
Cadmium	0.06	0.07	0.02	0.06	0.08	0.13	0.27	0.31	0.12	0.15	0.02	0.07	0.04	0.07	0.10	0.10	0.06	0.07	0.05	0.11	0.03	0.44	0.02	0.12
Chromium	<0.1	0.9	0.1	6.4	0.3	6.0	<0.1	0.7	0.1	1.9	<0.1	0.6	<0.1	0.5	0.3	1.1	0.1	0.8	0.5	3.7	<0.1	1.2	<0.1	3.2
Chromium (VI)	<50	-	<50	-	<50	-	<50	-	<50	-	<50	-	<50	-	<50	-	<50	-	<50	-	<50	-	<50	-
Cobalt	13.9	13.7	0.8	2.4	3.1	6.0	7.0	7.4	0.6	1.7	<0.1	0.5	<0.1	0.3	2.7	5.2	6.1	6.4	<0.1	1.6	1.0	1.6	1.2	3.7
Copper	<0.5	2.4	<0.5	5.4	1.1	7.4	<0.5	2.6	0.6	3.9	<0.5	2.2	<0.5	2.4	2.0	4.8	<0.5	3.0	5.1	8.2	1.3	5.7	<0.5	4.4
Iron	105	6960	96	11100	183	6560	184	3370	164	2450	968	3690	119	481	1130	4510	25	867	803	10000	16	5380	380	53800
Lead	0.2	3.5	0.4	4.2	0.3	6.0	0.3	3.6	0.2	2.9	0.3	1.2	<0.1	0.6	1.4	2.2	0.2	1.2	0.6	7.8	<0.1	6.7	<0.1	2.8
Manganese	4530	4420	175	183	481	613	766	831	328	410	1	47	28	103	652	936	100	102	13	104	614	653	610	756
Mercury	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Molybdenum	0.2	0.2	0.3	0.4	0.1	0.3	<0.1	0.1	<0.1	0.2	<0.1	<0.1	0.4	0.5	0.5	0.6	<0.1	<0.1	0.5	0.5	1.1	1.2	<0.1	0.3
Nickel	7.6	7.7	<0.5	4.5	2.3	4.2	10.1	11.1	2.2	3.9	<0.5	1.0	1.8	2.5	4.7	9.0	5.3	7.8	2.7	5.0	0.9	2.1	1.5	6.2
Selenium	0.3	<0.1	0.1	0.6	<0.1	0.3	0.1	0.2	0.2	0.2	<0.1	<0.1	<0.1	0.2	<0.1	0.1	<0.1	0.1	0.3	0.5	<0.1	0.1	<0.1	0.3
Vanadium	0.06	1.32	0.36	14.1	0.45	10.7	0.07	0.84	0.38	3.60	1.57	2.42	0.53	0.64	0.71	2.11	0.15	0.84	1.07	8.06	0.04	1.74	0.03	4.94
Zinc	6	12	1	10	13	31	10	14	31	41	2	6	6	6	17	22	15	16	20	46	3	32	2	15

Table 2a: Concentrations of metals and metalloids (µg/l) in filtered (Filt.) and whole (Wh.) samples collected from open wells

Source	DD		DD		DD		DD		DD		LW		TW		PW		PW		SP		SP		SP	
adjacent to open well #	IDN15001		IDN15004		-		-		-		-		-		-		-		-		-		-	
Sample code	IDN15013		IDN15014		IDN15015		IDN15016		IDN15017		IDN15018		IDN15019		IDN15020		IDN15021		IDN15022		IDN15023		IDN15024	
	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh	Filt.	Wh
Aluminium	49	48	9	40	<5	9	28	27	11	314	35	239	170	1470	134	2160	110	745	161	1530	164	1470	145	477
Antimony	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.2	0.2
Arsenic	1.9	1.9	2.8	2.9	<0.1	<0.1	<0.1	<0.1	1.6	1.8	0.7	0.8	0.5	0.7	0.5	0.7	0.5	0.6	0.4	0.8	1.1	1.3	0.6	0.7
Barium	47	52	169	166	40	46	210	213	200	206	4	11	4	7	6	9	6	9	18	57	50	61	57	62
Beryllium	<0.02	<0.02	0.02	<0.02	<0.02	0.03	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	0.05	<0.02	<0.02
Cadmium	<0.01	0.02	<0.01	0.01	<0.01	0.01	<0.01	0.02	<0.01	0.01	0.02	0.1	<0.01	<0.01	<0.01	0.02	<0.01	<0.01	0.03	0.13	<0.01	0.04	0.01	0.02
Chromium	<0.1	0.3	0.1	0.4	<0.1	0.3	<0.1	0.3	<0.1	0.7	<0.1	0.7	<0.1	0.4	<0.1	0.5	<0.1	0.4	0.1	2.1	0.2	1.9	0.1	0.7
Chromium (VI)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Cobalt	0.1	0.1	1.4	1.3	<0.1	<0.1	<0.1	<0.1	<0.1	0.2	<0.1	0.2	<0.1	0.1	<0.1	0.2	<0.1	<0.1	<0.1	1.2	0.2	0.6	<0.1	0.2
Copper	0.6	2.2	<0.5	1.5	<0.5	1.6	<0.5	2.2	1.1	5.4	<0.5	2.3	<0.5	2.5	<0.5	2.2	<0.5	2	<0.5	4	<0.5	3.1	1	3.2
Iron	27	52	2090	2560	6	2820	200	234	8	377	6	201	<0.5	57	1	81	<0.5	39	22	1580	115	775	46	239
Lead	0.3	0.6	0.2	0.4	<0.1	0.4	0.2	0.4	<0.1	0.4	0.3	7.2	<0.1	0.3	0.1	0.8	<0.1	0.3	0.2	2	<0.1	1.2	<0.1	0.4
Manganese	1	1	382	389	155	163	3	3	66	70	5	257	25	81	2	123	0	31	13	115	1	16	1	13
Mercury	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Molybdenum	0.5	0.5	1.8	1.7	<0.1	<0.1	<0.1	<0.1	<0.1	0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.9	1	0.5	0.5	2.1	2
Nickel	<0.5	0.7	<0.5	0.8	<0.5	<0.5	0.7	1.3	2	6.3	<0.5	1.1	<0.5	1.2	<0.5	1.1	<0.5	0.8	<0.5	3	1.1	2.4	0.7	1.5
Selenium	0.2	0.1	<0.1	0.2	<0.1	<0.1		<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.1	0.3	0.2	0.1	0.2	0.2
Vanadium	0.04	0.04	0.45	0.51	0.02	0.04	0.02	0.03	0.16	0.48	1.39	1.85	1.34	1.41	1.25	1.59	1.14	1.22	0.36	3.29	1.21	3.21	1.17	1.56
Zinc	3	4	2	3	3	4	3	5	4	5	<1	4	<1	4	2	12	2	6	2	8	<1	7	5	12

Table 2b: Concentrations of metals and metalloids (μg/l) in filtered (Filt.) and whole (Wh.) samples collected from deep drill wells (DD), lake water (LW), settling ponds (SP), treated water at the PDAM facility (TW) and water piped from the PDAM facility (PW)

Sample Code	IDN 15001	IDN 15002	IDN 15003	IDN 15004	IDN 15005	IDN 15006	IDN 15007	IDN 15008	IDN 15009	IDN 15010	IDN 15011	IDN 15012
Sample Type	open well water											
Number of compounds isolated	6	6	5	0	4	1	0	12	2	2	1	1
Number of compounds identified to >90%	0	2	2	0	2	1	0	9	2	2	1	1
Percentage identified to >90%	0	33	40	N/A	50	100	N/A	75	100	100	100	100
Phthalates												
Diethyl Phthalate (DEP)		✓	✓		✓	✓		✓	✓	✓	✓	✓
Dimethyl phthalate (DMP)		✓	✓		✓							
Alkanes & alkenes								✓[a]	✓[b]			
PAH										✓[c]		

Sample Code	IDN 15013	IDN 15014	IDN 15015	IDN 15016	IDN 15017	IDN 15018	IDN 15019	IDN 15020	IDN 15021	IDN 15022	IDN 15023	IDN 15024
Sample Type	Deep Drilled Well Water					Lake Water	Drinking Water	Tap Water		Water from Settling Pond		
Number of compounds isolated	0	0	0	1	0	0	5	0	5	2	0	1
Number of compounds identified to >90%	0	0	0	1	0	0	3	0	2	2	0	1
Percentage identified to >90%	N/A	N/A	N/A	100	N/A	N/A	60	N/A	40	100	N/A	100
Phthalates												
Diethyl Phthalate (DEP)				✓			✓		✓	✓		✓
Dimethyl phthalate (DMP)							✓		✓	✓		
Alkanes & alkenes							✓[d]					
PAH												

a – docosane, eicosane, hexacosane, octacosane, nonacosane, pentacosane, tetracosane, tricosane

b – cyclotetracosane

c – pyrene at trace levels (could only be identified with GC-MS operated in selected ion monitoring (SIM) mode)

d - 1-Nonadecene

Table 3: summary of semi-volatile (solvent extractable) organic chemicals (SVOCs) present in samples.

For more information please contact:

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

Reference

EU (1998). Directive 1998/83/EC of the European Parliament and of the Council of 3 November 1998 on the quality of water intended for human consumption. http://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:31998L0083&from=EN

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. Water samples (500ml) 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 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

For the total organic compounds screening, samples were analysed using an Agilent 7890B GC with Restek Rxi-17Sil MS 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 Mass Hunter system. The GC oven temperature program employed was as follows: an initial temperature of 40^0C, raised to 260^0C at 10^0C/min, then to 295^0C at 50^0C/min (held for 15min), and finally to 320^0C at 50^0C/min (held for 12min). The carrier gas was helium, supplied at 1ml/min. Identification of compounds was carried out by matching spectra against the Wiley 7N Library, 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 polycyclic aromatic hydrocarbons (PAHs).

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 metals

Preparation

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 10% v/v. Separately, 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 20 minutes followed by

holding at 180°C for a further 20 minutes. Cooled digests were 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 (0.5, 5, 50 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).

Concentrations of chromium (VI) were determined colorimetric for each water sample following filtration. 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, 200 and 500 μg/l respectively. Any sample exceeding the calibration range was diluted accordingly, in duplicate, and re-analysed.

Quality control

Four samples were prepared for ICP analysis in duplicate and analysed to verify method reproducibility, along with a blank sample (10% v/v nitric acid in deionised water). 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. 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 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.

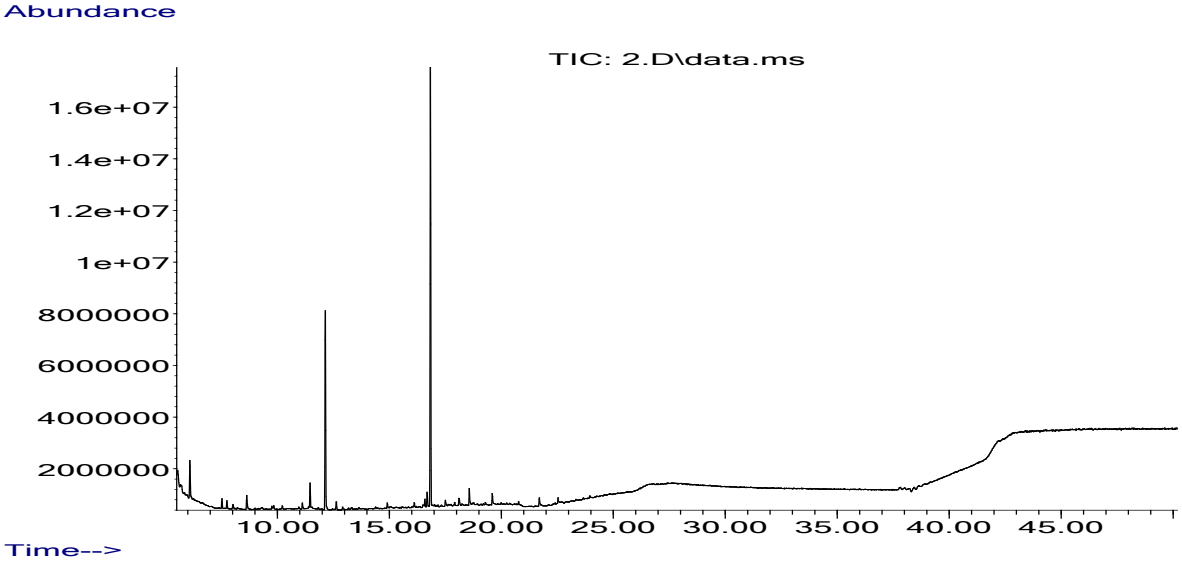
For chromium (VI) determination, two samples were prepared and analysed in duplicate, along with a blank sample, and two quality control solutions of 100 and 400 μg/l prepared in an identical manor to the samples.

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

Appendix 2: Detailed semi-volatile organic analytical screening data

Detailed screening data arising from GC-MS analysis of semi-volatile organic compounds fromsolvent extraction are presented below. Only those semi-volatile substances identified to greater than 90% quality match (following verification by expert interpretation) are listed here.

Sample code	IDN15001
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Open well water
Date & time	21/Nov/2015 09.47
Description	Depth of water: 0.2m; depth of well: 1.47m. Well water is used for gardening.



Number of compounds isolated: 6

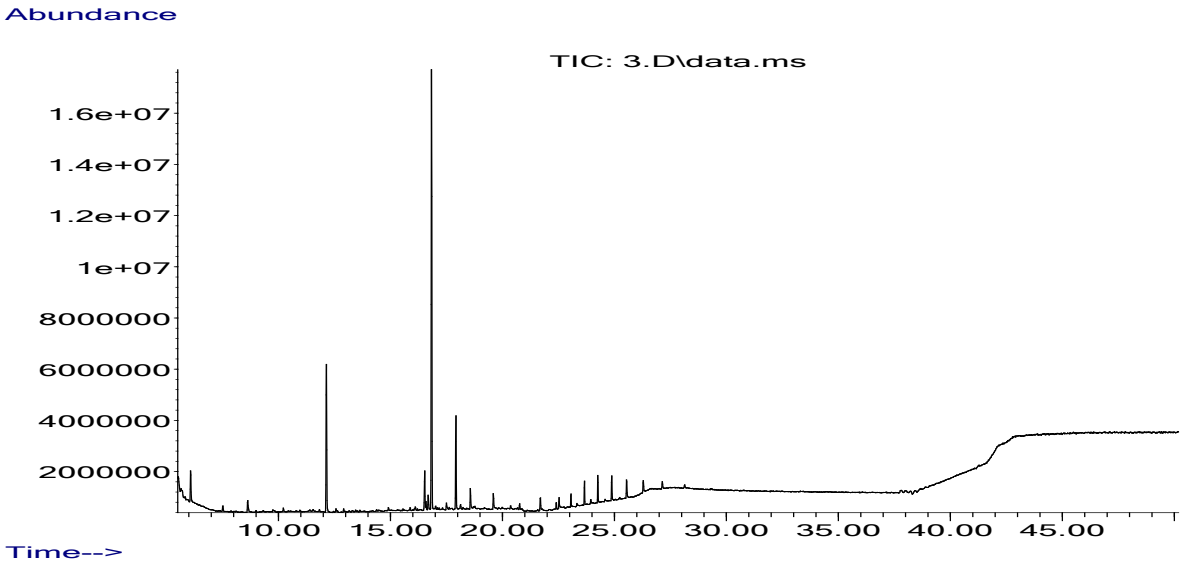
Compounds identified to better than 90%:

None

Compounds tentatively identified:

None

Sample code	IDN15002
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Open well water
Date & time	21/Nov/2015 14.49
Description	Depth of water: 0.61m; depth of well: 3.69m. Water colour: dark. Smelly.



Number of compounds isolated: 6

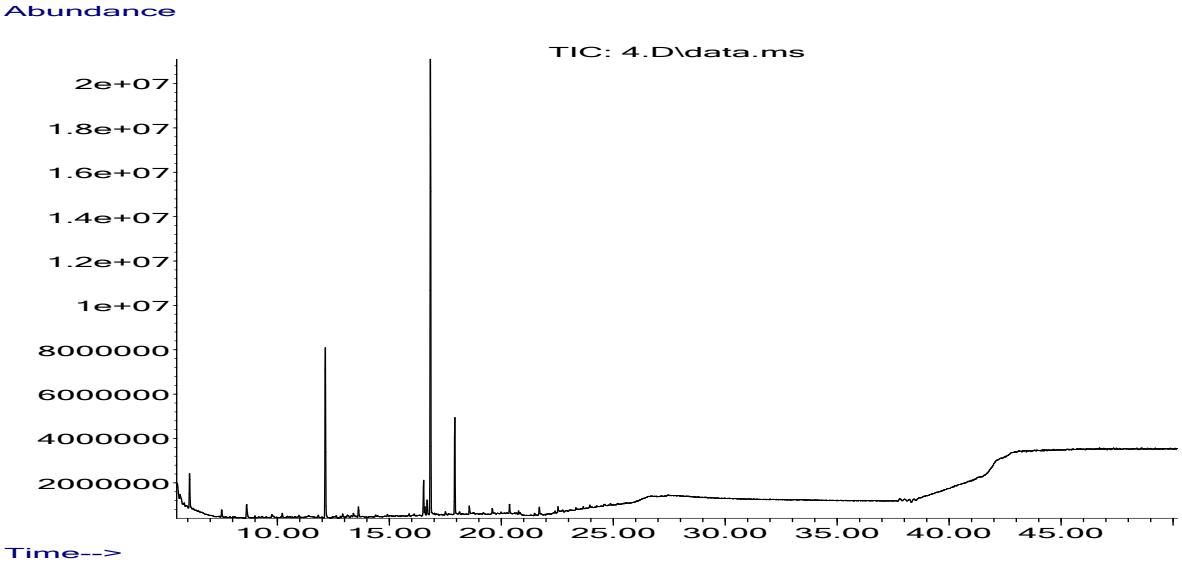
Compounds identified to better than 90%:

CAS#	**Name**
000084-66-2	1,2-Benzenedicarboxylic acid, diethyl ester
000131-11-3	1,2-Benzenedicarboxylic acid, dimethyl ester

Compounds tentatively identified:

None

Sample code	IDN15003
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Open well water
Date & time	21/Nov/2015 15.16
Description	Depth of water: 0.2m; depth of well: 0.91m. Water colour: Light Brown. Well water is used for cleaning and washing, sometime for cooking.



Number of compounds isolated: 5

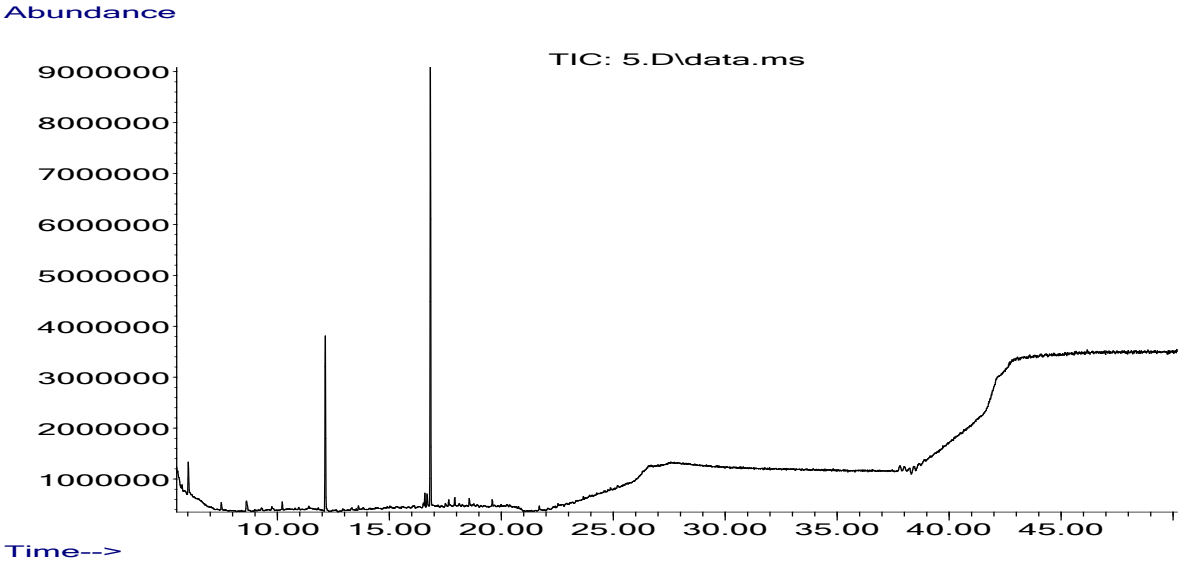
Compounds identified to better than 90%:

CAS#	**Name**
000131-11-3	1,2-Benzenedicarboxylic acid, dimethyl ester
000084-66-2	1,2-Benzenedicarboxylic acid, diethyl ester

Compounds tentatively identified:

None

Sample code	IDN15004
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Open well water
Date & time	22/Nov/2015 10.28
Description	Depth of water: 3.99m; depth of well: 5.2m. Water colour: light brown. Well water is used for washing and cleaning.



Number of compounds isolated: 0

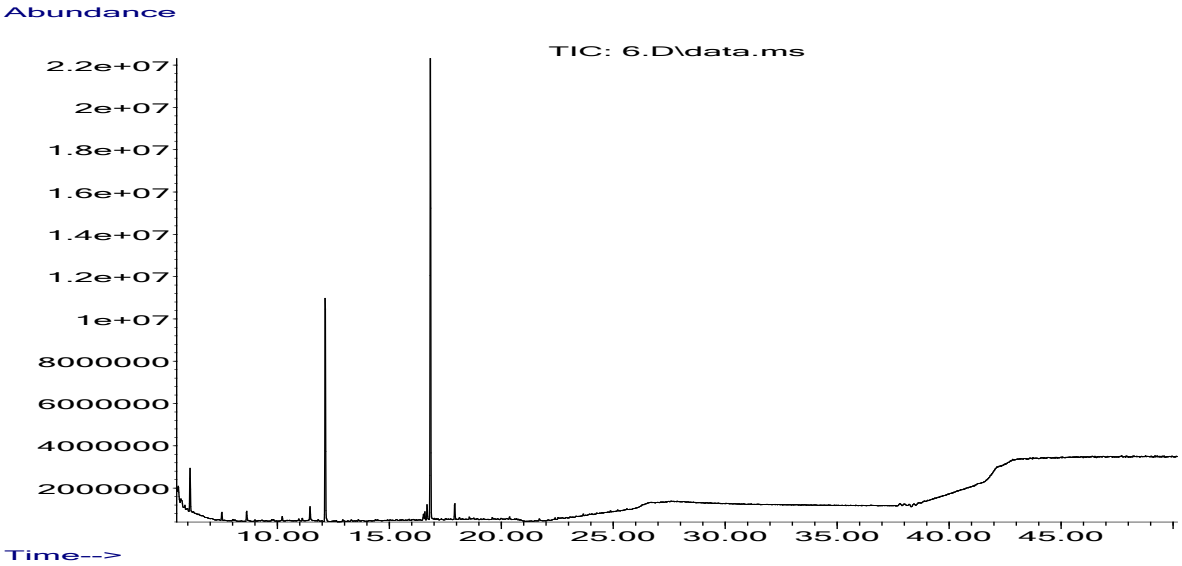
Compounds identified to better than 90%:

None

Compounds tentatively identified:

None

Sample code	IDN15005
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Open well water
Date & time	21/Nov/2015 14.21
Description	Depth of water: 0.8 m; depth of well: 2.11m. Water colour: dark. Smelly.



Number of compounds isolated: 4

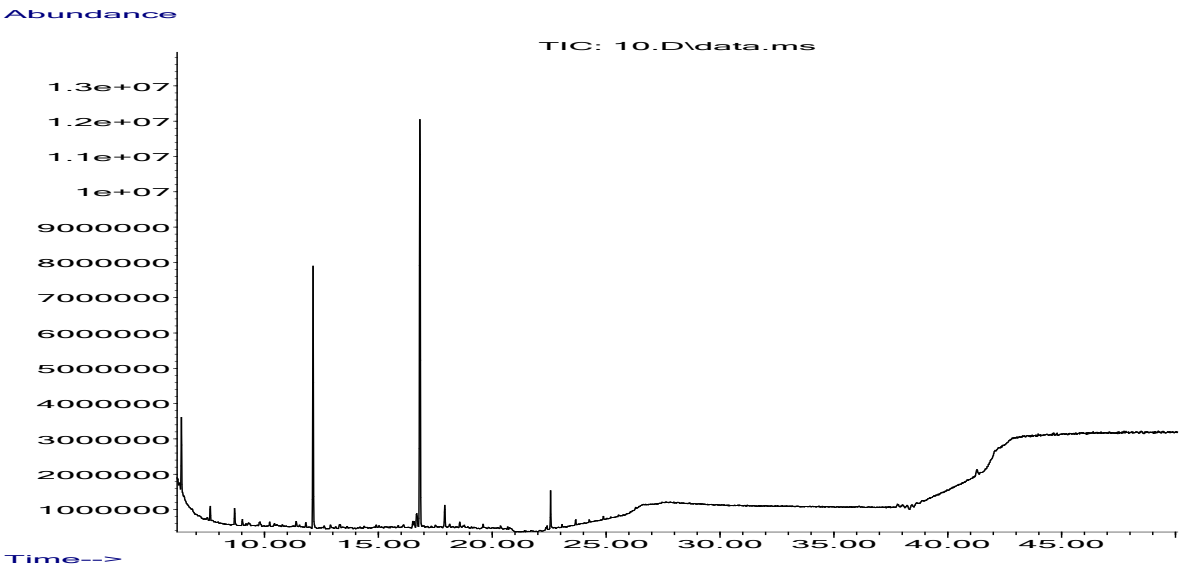
Compounds identified to better than 90%:

CAS#	**Name**
000131-11-3	1,2-Benzenedicarboxylic acid, dimethyl ester
000084-66-2	1,2-Benzenedicarboxylic acid, diethyl ester

Compounds tentatively identified:

None

Sample code	IDN15006
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Open well water
Date & time	21/Nov/2015 11.14
Description	Depth of water: 1.2 m; depth of well: 1.64m. Water colour: light dark. Well water is used by 6 household for daily used, and getting wider likes pond.



Number of compounds isolated: 1

Number of compounds isolated: 1

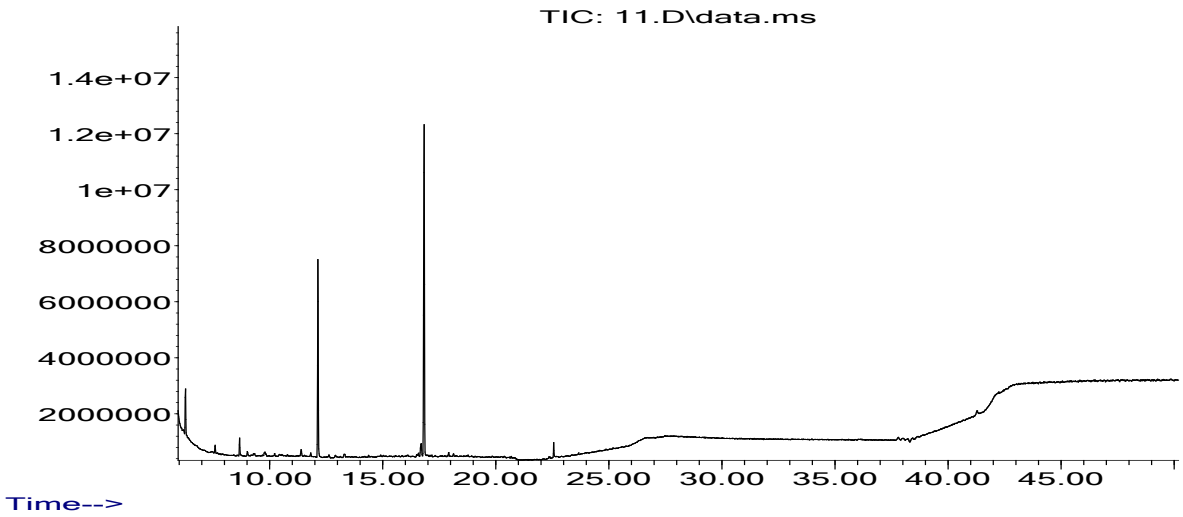
Compounds identified to better than 90%:

CAS#	**Name**
000084-66-2	1,2-Benzenedicarboxylic acid, diethyl ester

Compounds tentatively identified:

None

Sample code	IDN15007
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Open well water
Date & time	21/Nov/2015 13.06
Description	Depth of water: 4.5m; depth of well: 5.8m. Water from the well is used for washing and cleaning. Water colour: Light dark and sandy.



Number of compounds isolated: 0

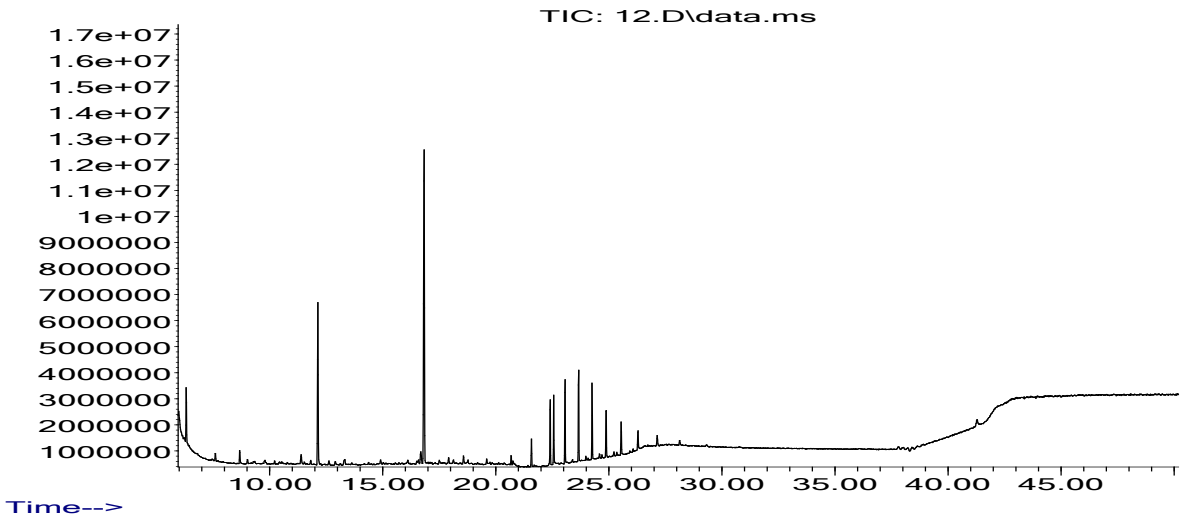
Compounds identified to better than 90%:

None

Compounds tentatively identified:

None

Sample code	IDN15008
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Open well water
Date & time	21/Nov/2015 09.03
Description	Depth of water: 3.45m; depth of well: 4m. The active coal mine is 100 m from the well. Well not in use.



Number of compounds isolated: 12

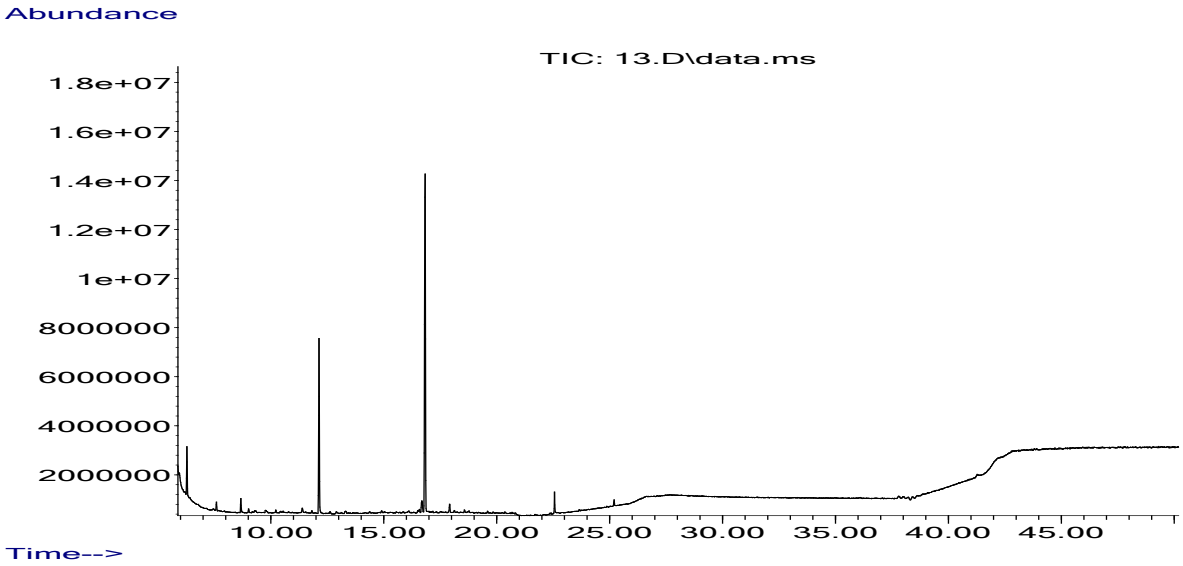
Compounds identified to better than 90%:

CAS#	**Name**
000084-66-2	1,2-Benzenedicarboxylic acid, diethyl ester
000629-97-0	Docosane
000112-95-8	Eicosane
000630-01-3	Hexacosane
000630-02-4	Octacosane
000630-03-5	Nonacosane
000629-99-2	Pentacosane
000646-31-1	Tetracosane
000638-67-5	Tricosane

Compounds tentatively identified:

None

Sample code	IDN15009
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Open well water
Date & time	21/Nov/2015 10.27
Description	Depth of water: 0.3m; depth of well: 2.85m. Well water is used by 3 houses.



Number of compounds isolated: 2

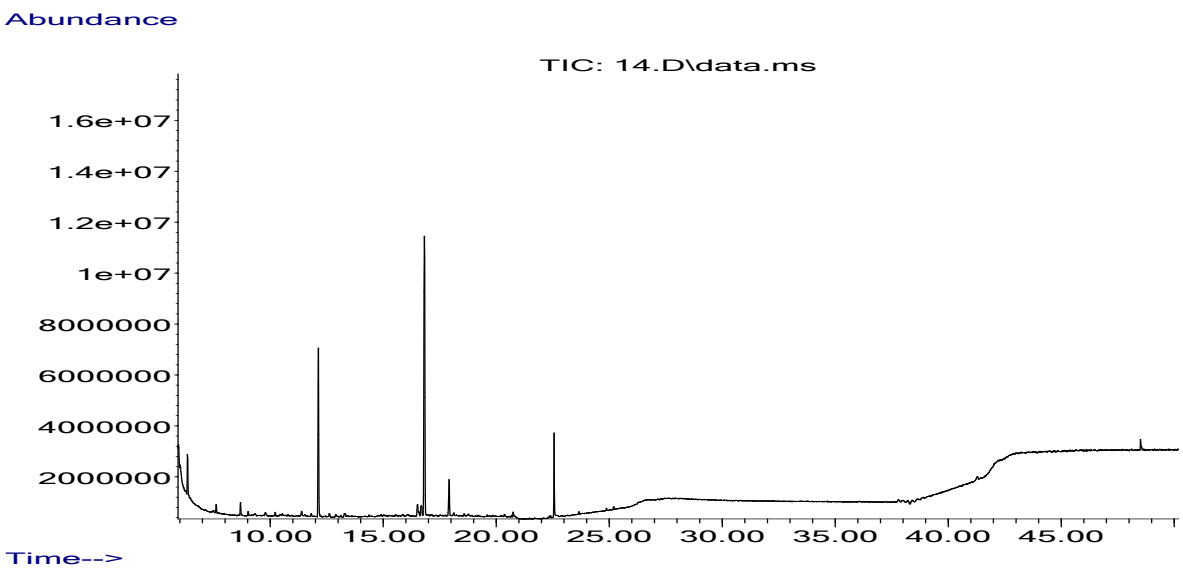
Compounds identified to better than 90%:

CAS#	**Name**
000297-03-0	Cyclotetracosane
000084-66-2	Diethyl phthalate

Compounds tentatively identified:

None

Sample code	IDN15010
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Open well water
Date & time	21/Nov/2015 13.25
Description	Depth of water: 0.5m; depth of well: 1.85m. Water colour: soft green



Number of compounds isolated: 2

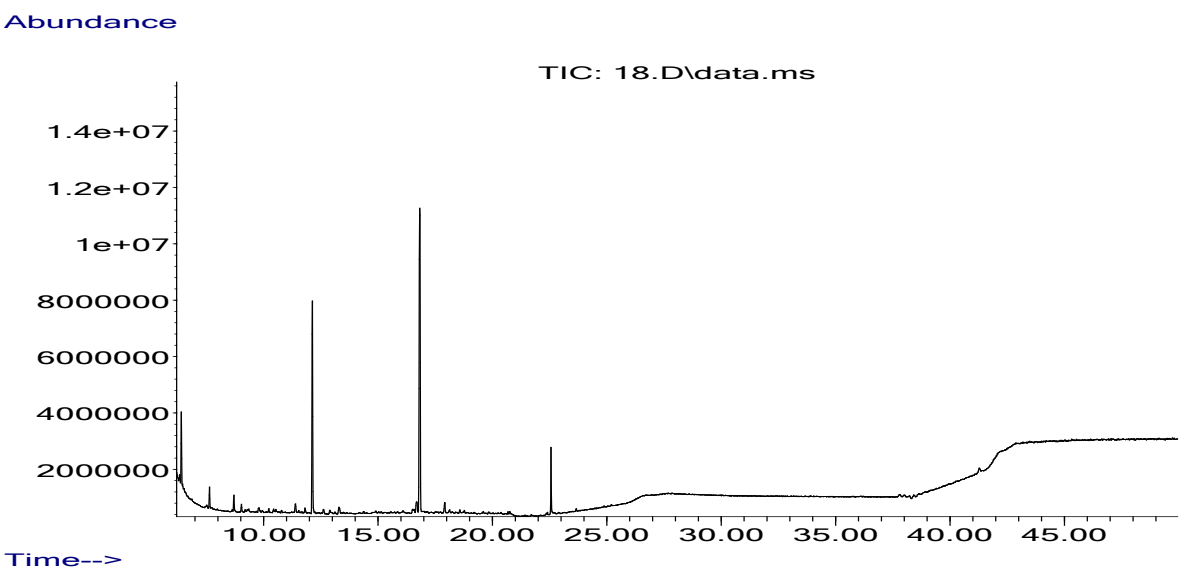
Compounds identified to better than 90%:

CAS#	**Name**
000084-66-2	Diethyl phthalate
000129-00-0	Pyrene (SIM)

Compounds tentatively identified:

None

Sample code	IDN15011
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Open well water
Date & time	21/Nov/2015 12.41
Description	Depth of water: 0.9m; depth of well: 3.95m. The water is not for drink, but for wash only.



Number of compounds isolated: 1

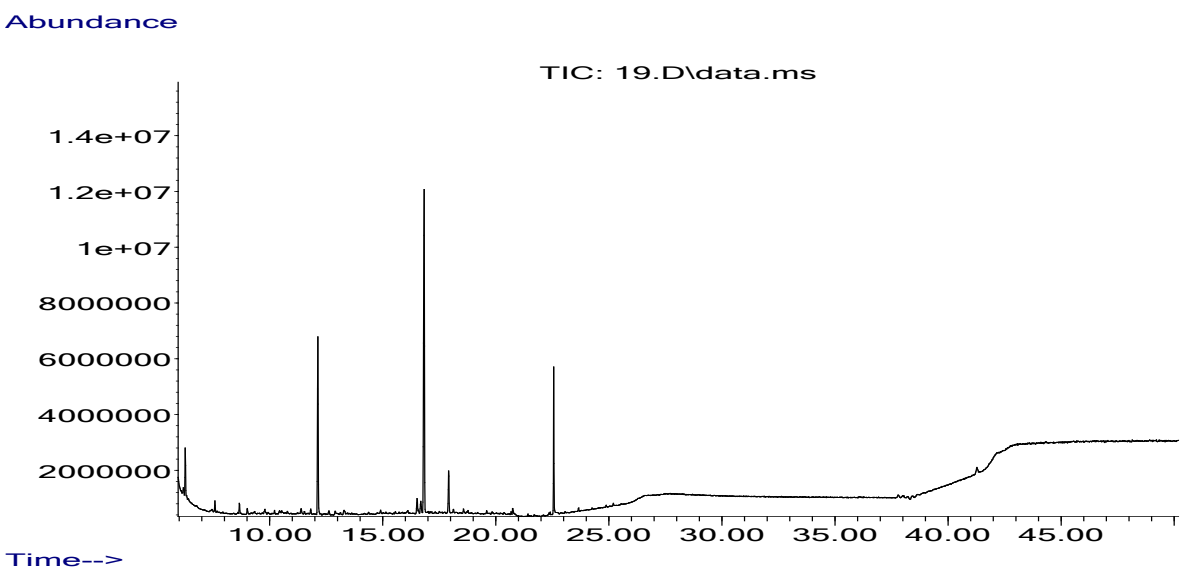
Compounds identified to better than 90%:

CAS#	**Name**
000084-66-2	Diethyl phthalate

Compounds tentatively identified:

None

Sample code	IDN15012
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Open well water
Date & time	22/Nov/2015 11.25
Description	Depth of water: 0.3m; depth of well: 2.95m. Lots of dead mosquitos larvae in the water.



Number of compounds isolated: 1

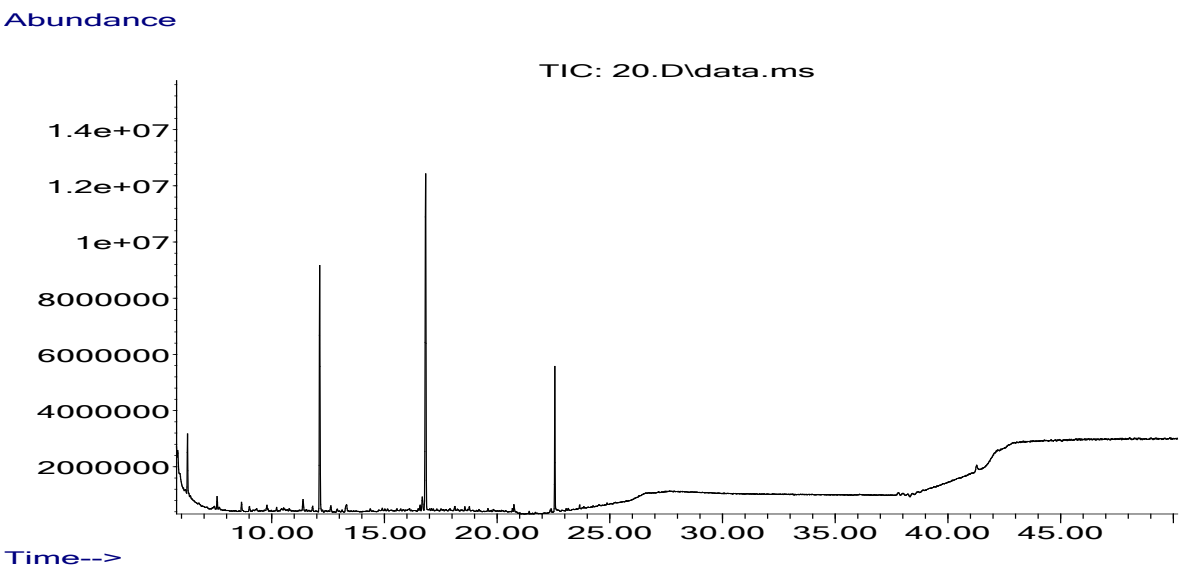
Compounds identified to better than 90%:

CAS#	**Name**
000084-66-2	Diethyl phthalate

Compounds tentatively identified:

None

Sample code	IDN15013
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Deep Drilled Well Water
Date & time	21/Nov/2015 10.08
Description	Water color: light brown. The well is still used for gardening. Depth unknown.



Number of compounds isolated: 0

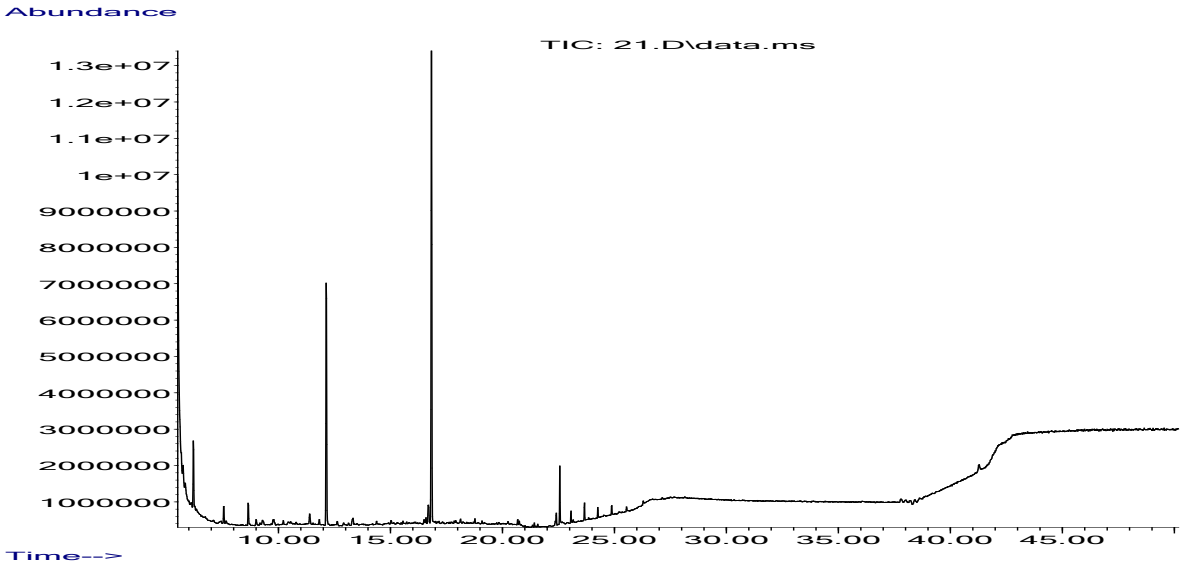
Compounds identified to better than 90%:

None

Compounds tentatively identified:

None

Sample code	IDN15014
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Deep Drilled Well Water
Date & time	22/Nov/2015 10.41
Description	Depth of well: 46m



Number of compounds isolated: 0

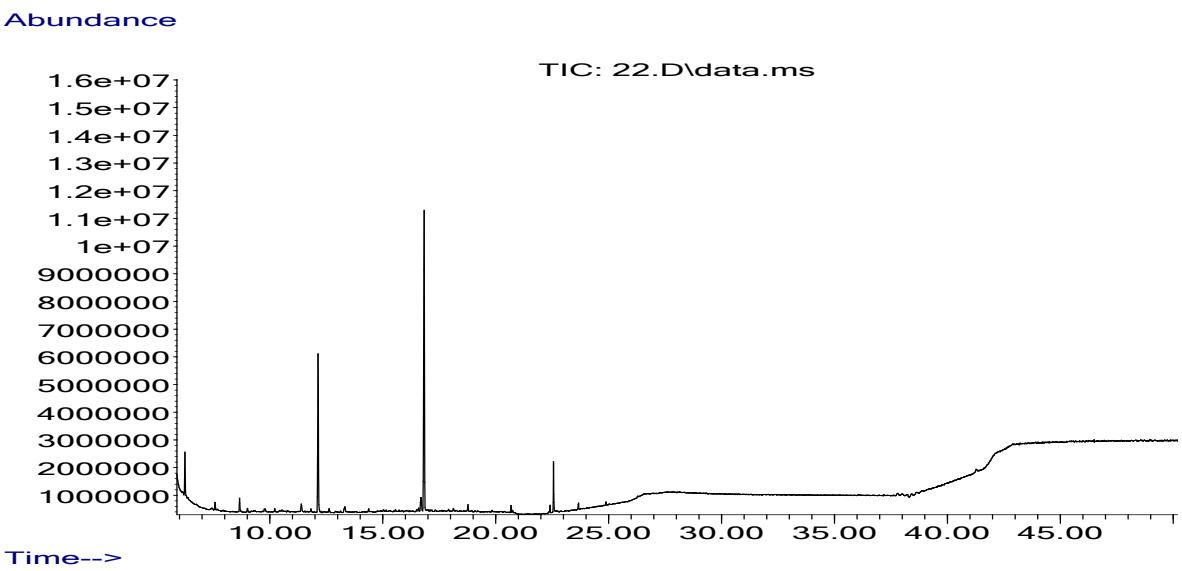
Compounds identified to better than 90%:

None

Compounds tentatively identified:

None

Sample code	IDN15015
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Deep Drilled Well Water
Date & time	21/Nov/2015 13.57
Description	Depth of well: 19m



Number of compounds isolated: 0

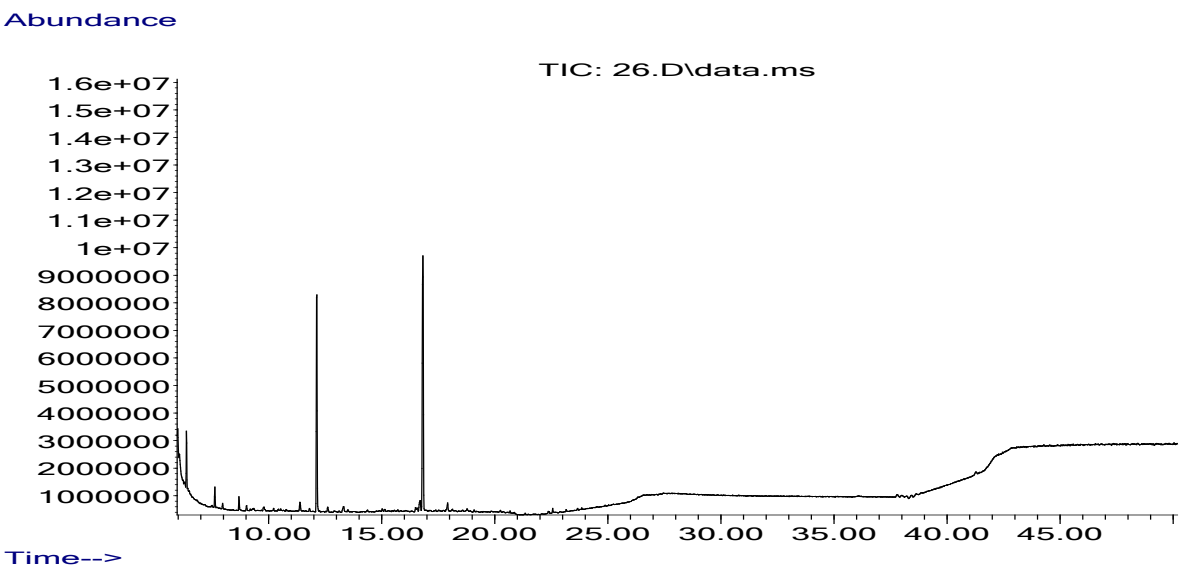
Compounds identified to better than 90%:

None

Compounds tentatively identified:

None

Sample code	IDN15016
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Deep Drilled Well Water
Date & time	22/Nov/2015 09.34
Description	Depth of well: 24m



Number of compounds isolated: 1

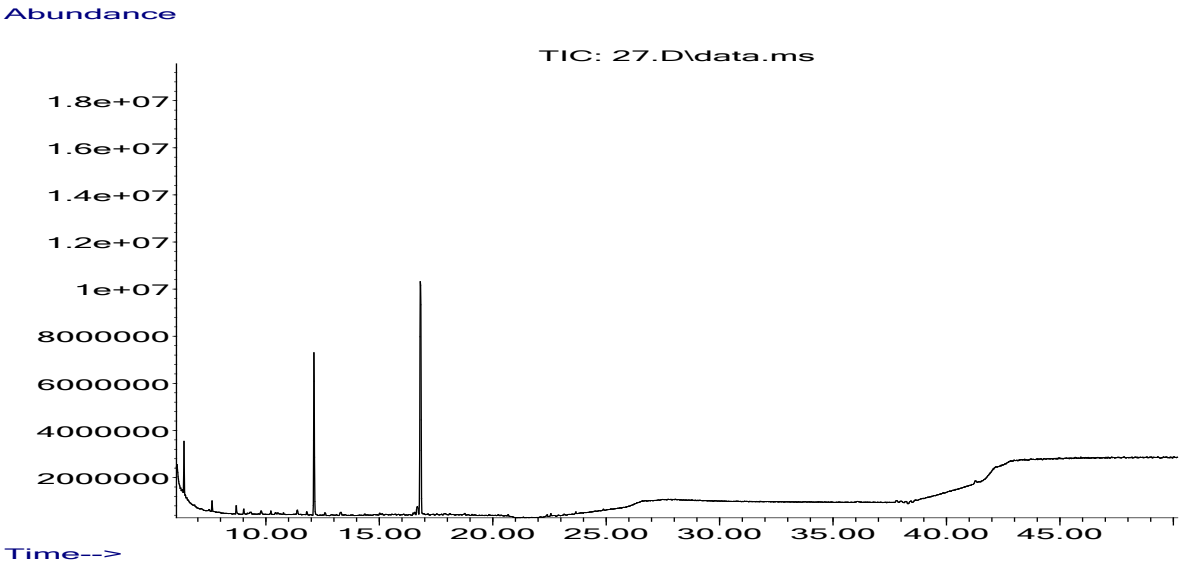
Compounds identified to better than 90%:

CAS#	**Name**
000084-66-2	Diethyl phthalate

Compounds tentatively identified:

None

Sample code	IDN15017
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Deep Drilled Well Water
Date & time	22/Nov/2015 10.04
Description	Depth of well: 12m



Number of compounds isolated: 0

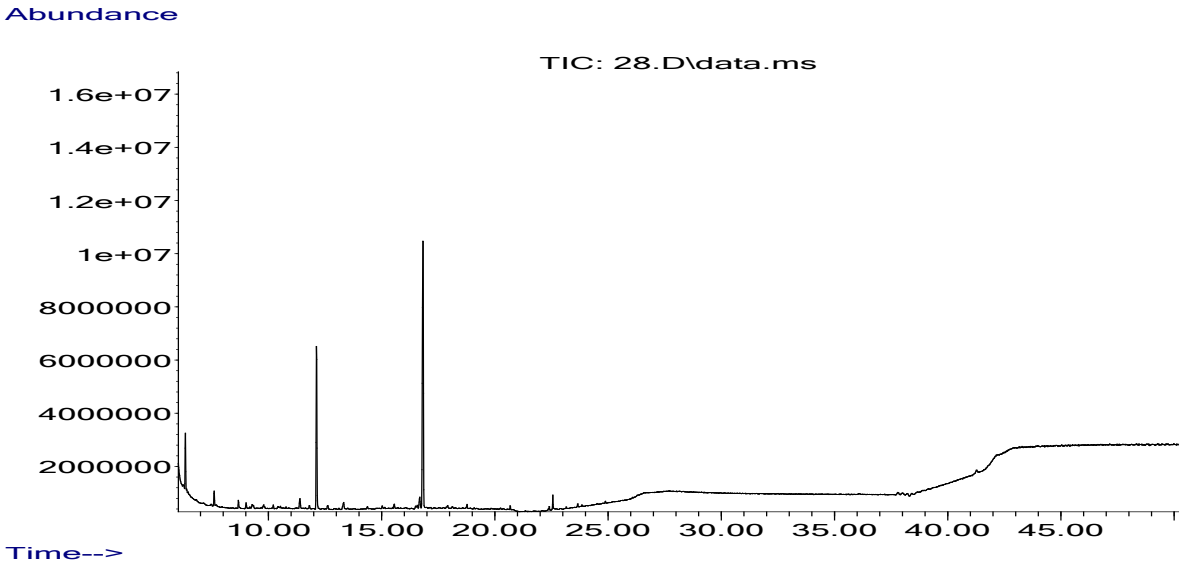
Compounds identified to better than 90%:

None

Compounds tentatively identified:

None

Sample code	IDN15018
Location	Northeast of Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Lake water
Date & time	21/Nov/2015 16.04
Description	The lake was formed on the site of an ex coal pit mine



Number of compounds isolated: 0

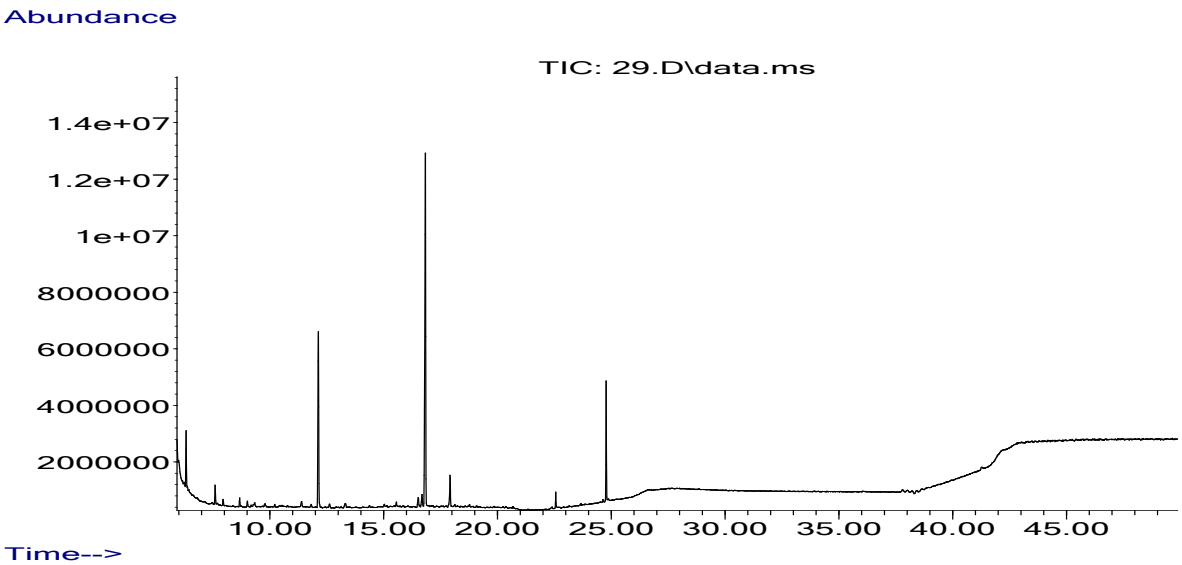
Compounds identified to better than 90%:

None

Compounds tentatively identified:

None

Sample code	IDN15019
Location	PDAM (Local Drinking Water Company), Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Treated Drinking water
Date & time	21/Nov/2015 16.26
Description	After the Treatment by PDAM (Local Drinking Water Company), and before being distributed to costumers.



Number of compounds isolated: 5

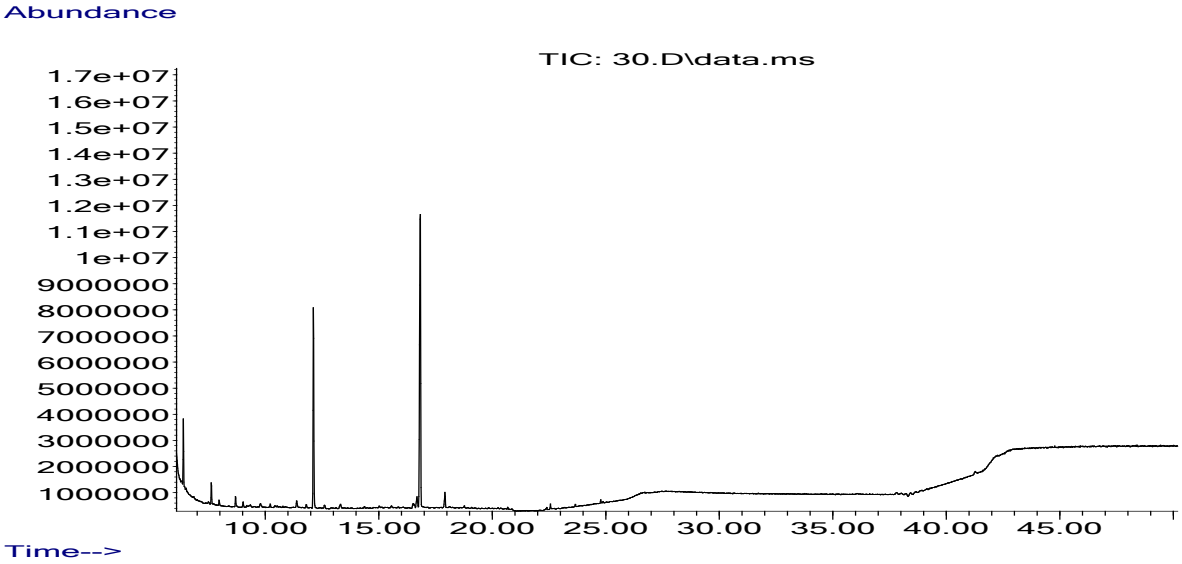
Compounds identified to better than 90%:

CAS#	**Name**
000131-11-3	1,2-Benzenedicarboxylic acid, dimethyl ester
000084-66-2	1,2-Benzenedicarboxylic acid, diethyl ester
018435-45-5	1-Nonadecene

Compounds tentatively identified:

None

Sample code	IDN15020
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Tap water
Date & time	21/Nov/2015 17.45
Description	Tap water from PDAM, reported by local villagers that the water quality are bad and cant used for cooking



Number of compounds isolated: 0

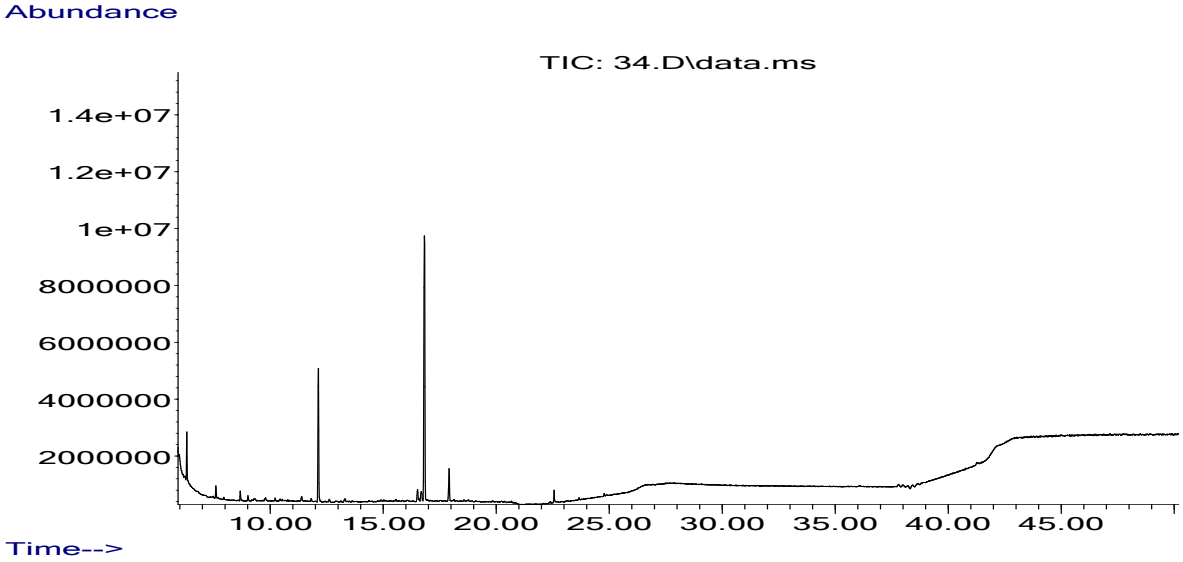
Compounds identified to better than 90%:

None

Compounds tentatively identified:

None

Sample code	IDN15021
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Tap water
Date & time	22/Nov/2015 08.46
Description	Tap water from PDAM, reported by local villager that the water can cause irritation to their skin some times



Number of compounds isolated: 5

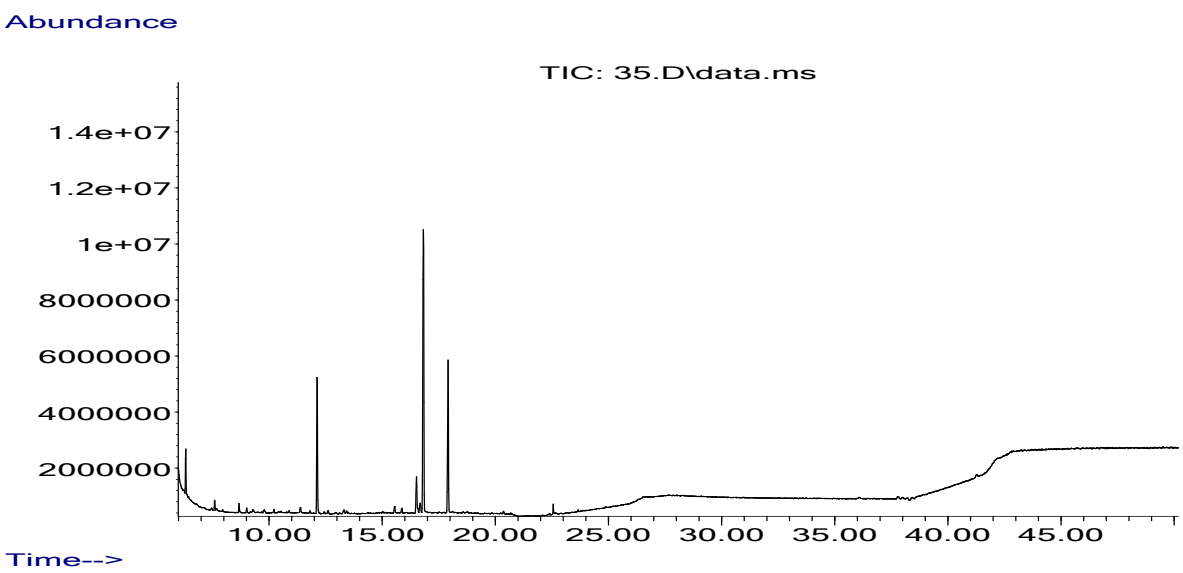
Compounds identified to better than 90%:

CAS#	**Name**
000131-11-3	1,2-Benzenedicarboxylic acid, dimethyl ester
000084-66-2	1,2-Benzenedicarboxylic acid, diethyl ester

Compounds tentatively identified:

None

Sample code	IDN15022
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Water from Settling Pond
Date & time	22/Nov/2015 08.20
Description	This settling pond is in the southeast part of the village. The water will run to the village before end in Separi River.



Number of compounds isolated: 2

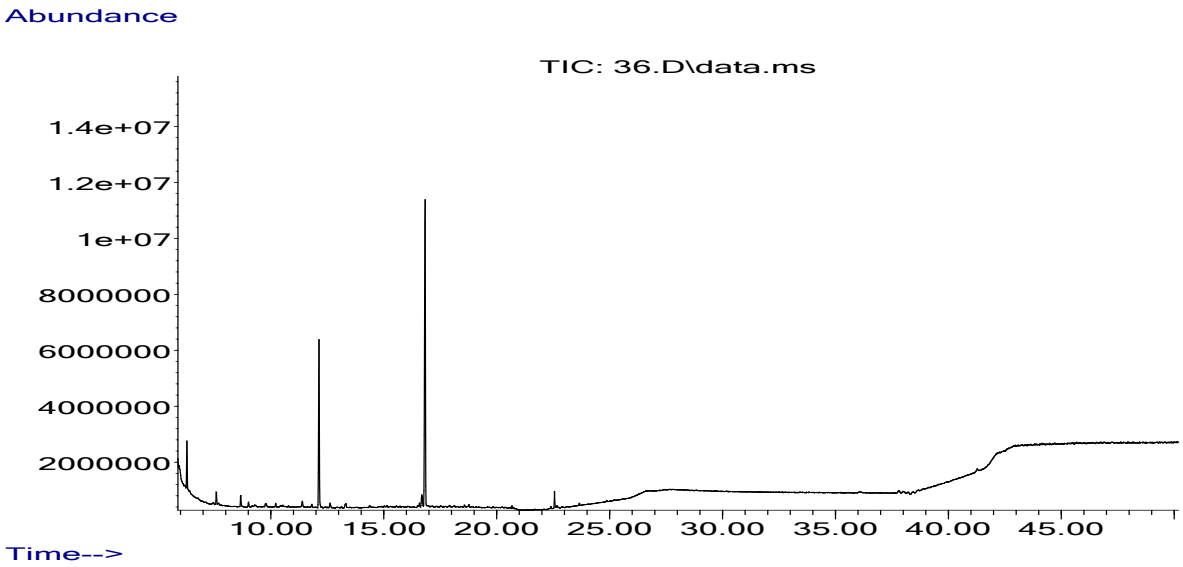
Compounds identified to better than 90%:

CAS#	**Name**
000131-11-3	1,2-Benzenedicarboxylic acid, dimethyl ester
000084-66-2	1,2-Benzenedicarboxylic acid, diethyl ester

Compounds tentatively identified:

None

Sample code	IDN15023
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Water from Settling Pond
Date & time	21/Nov/2015 17.17
Description	This settling pond is to the south west of the village. The water will go to Mahakam River



Number of compounds isolated: 0

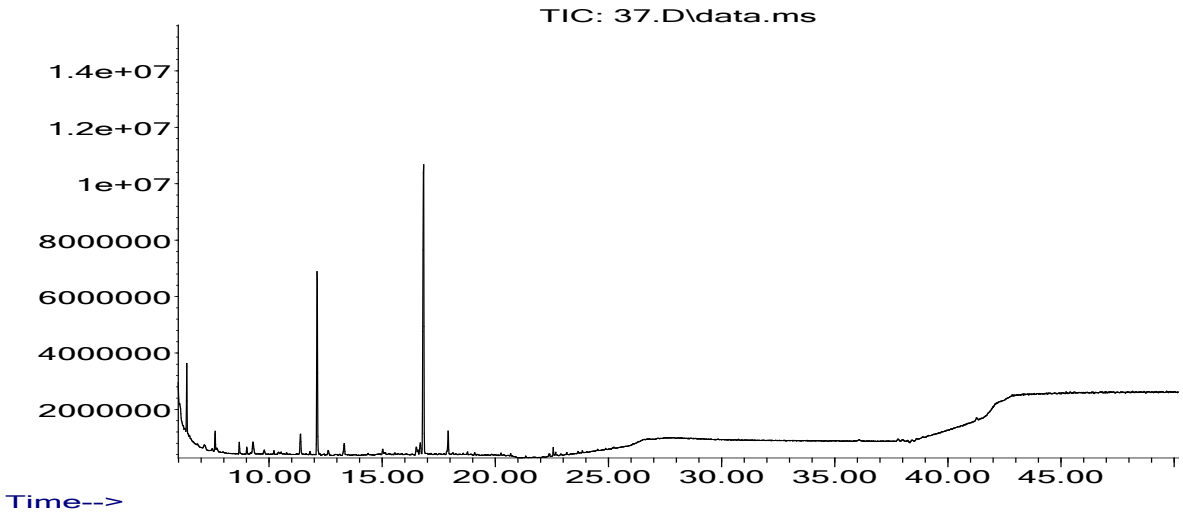
Compounds identified to better than 90%:

None

Compounds tentatively identified:

None

Sample code	IDN15024
Location	Kerta Buana Village, Kutai Karta Negara, Indonesia
Sample type	Water from Settling Pond
Date & time	22/Nov/2015 12.02
Description	This settling pond is to the south west of the village. Coal washing water are treated with $Ca(OH)_2$ to neutralize the acidity, before go to Mahakam River.



Number of compounds isolated: 1

Compounds identified to better than 90%:

CAS#	**Name**
000084-66-2	1,2-Benzenedicarboxylic acid, diethyl ester

Compounds tentatively identified:

None