

Greenpeace Research Laboratories Analytical Results 2021-02

Screening of sediments collected close to decommissioned fish farms in Scotland for metals and organic contaminants

June 2021, 24 pp.

Introduction

7 samples of marine sediment collected in the vicinity of three decommissioned salmon farms in NW Scotland (two locations in Loch Ewe and one in Loch Toridon) and one control site (also in Loch Ewe) were delivered to our laboratory for analysis in December 2020. According to information provided to us in the form of an expedition report, all samples had been collected by divers between the dates of 09 and 17 November 2020. The samples were contained in aluminium screw-capped containers in which they were originally collected from the seafloor by the dive team before being chilled and then frozen for transport and storage. Details of the samples received are provided in Table 1, together with GPS coordinates and water depths. All samples were stored at -20 C in our laboratory prior to sub-sampling and analysis

Project sample code	GRL sample code	Sampling location	GPS	Depth (m)	Analyses conducted
004	NGP20001	Old farm 1 (FS0206 – Loch Ewe Poolewe) - centre	57.81 N / 5.65 W	22	Metals (quantitative), semi-volatile organic compounds (sVOCs, qualitative), pesticides mix (semi-quantitative), pharmaceutical mix (qualitative)
005	NGP20002	Old farm 1 (FS0206 – Loch Ewe Poolewe) - 10m N	57.81 N / 5.65 W	22	
007	NGP20003	Control 1 - centre	57.794 N / 5.648 W	22	
019	NGP20004	Old farm 2 (FS0729 – Aultbea) - 10m N	57.817 N / 5.588 W	34	
021	NGP20005	Old farm 2 (FS0729 – Aultbea) - centre	57.817 N / 5.588 W	34	
038	NGP20006	Old farm 3 (FS0049 – Camas an Eilean) centre	57.558 N / 5.751 W	24	
040	NGP20007	Old farm 3 (FS0049 – Camas an Eilean) - 10m N	57.558 N / 5.751 W	24	

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

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Materials & Methods

In January 2021, all 7 samples were thawed in a refrigerator at 6 C overnight, before decanting off the water layer to leave the settled sediments undisturbed. These wet sediments were then homogenised by mixing thoroughly by hand using a pre-cleaned stainless steel spatula before taking sub-samples for a range of subsequent analyses outlined in the final column of Table 1 above.

Metals

A representative portion of each sample was air dried to constant weight, sieved through a 2mm mesh, and then ground to a powder using a pestle and mortar. Approximately 0.25 g of each 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 using a CEM MARS Xpress system, with temperature ramping, heating to 180 C over 20 minutes, held at 180 C for 20 minutes, heating to 200 C over 20 minutes and finally held at 200 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.

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, were used for instrument calibration, at concentrations of 1, 10, 100 and 1000 µg/l respectively, other than for mercury (1, 2, 5, 20µg/l respectively). 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. 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.

For quality control purposes, one sample was prepared for ICP analysis in duplicate and analysed to verify method reproducibility, along with a blank. To check the method efficiency, two certified reference material (CRM) samples were prepared in an identical manner; LGC6187, leachable metals in river sediment, and LGC6156, extractable metals in harbour sediment, both certified by the Laboratory of the Government Chemist, UK.

Semi-volatile organics (sVOCs)

Sub-samples of approximately 10 g of each sample (wet weight) were collected from each homogenised sample into clean 100ml glass Duran bottles, before adding 20 µg of deuterated naphthalene as an Internal Standard (IS) to each bottle and mixing thoroughly with a clean stainless steel spatula. Each sub-sample was then 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 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.

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 C, raised to 260 C at 10 C/min, then to 295 C at 50 C/min (held for 5 min), then to 325 C at 50 C /min (held for 4 min), finally raised to 330 C at 50 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.

Pesticides & pharmaceuticals

Sub-samples of 10g (wet weight) were collected from each homogenised sample and mixed with 5 ml deionised water before adding 15 ml of acetonitrile (HPLC grade) and performing a standardised QuEChERS extraction (Schenck & Hobbs 2004). The liquid phase was then centrifuged for 5 minutes at 3000 rpm to remove any residues of sediment before collecting and filtering the supernatant through a Nylon syringe filter (17mm diameter, 0.2 µm pore size) prior to analysis. Extracts were analyzed qualitatively for the presence of a mix of 251 pesticide and 79 veterinary drug components (see Appendix 1 for a complete list of those compounds analysed for) using liquid chromatography (LC) - mass spectrometry (MS) (LC-ESI-Q-Orbitrap-MS) with the following parameters:

- Gradient chromatographic conditions (with eluents “A”= water+2% methanol+0.1% formic acid+5mM ammonium formate, “B”= methanol+2%water+0.1% formic acid+5mM ammonium formate)
- Separation was carried out on Accucore aQ C18 column
- MS analysis was carried out both positive and negative ionisation mode
- Mass range was: 80-1000 Da

In case of the pesticides, semi-quantitative determination was carried out using external calibration standards, with a check for extraction efficiency using additional sub-samples of a reference river sediment spiked with known concentrations of the pesticide mix. The limit of quantification for this method varies from substance to substance across the full list of 251 pesticides, but was generally at or below 15 µg/kg (ppb) in wet sediment. Limits of detection (i.e. ability to distinguish the presence of a substance without quantification) would be significantly lower than this, meaning that if such substances were present in the sediments even at low ppb levels, they should at least be identified as peaks with a significant signal to noise ratio.

Results

Metals

Concentrations of the range of metals and metalloids for which the samples were analysed are reported in table 2.

Sample		Old farm 1 (centre)	Old farm 1 (10m N)	Control 1	Old farm 2 (10m N)	Old farm 2 (centre)	Old farm 3 (centre)	Old farm 3 (10m N)	
GRL sample code		NGP20001	NGP20002	NGP20003	NGP20004	NGP20005	NGP20006	NGP20007	
Project sample code		004	005	007	019	021	038	040	
Metal/metalloid									LOD
Arsenic	As	3.5	3.4	2.5	7.2	9.1	2.8	3.5	<0.1
Beryllium	Be	0.36	0.31	0.27	0.58	0.68	0.28	0.42	<0.02
Barium	Ba	35.3	33.9	19.8	53.7	68.3	24.1	39.3	<0.2
Cadmium	Cd	0.08	0.08	0.09	0.24	0.27	0.09	0.11	<0.02
Chromium	Cr	21.9	25.5	16.5	27.6	32.2	18.4	18.2	<0.1
Cobalt	Co	3.05	3.00	2.27	4.48	4.54	2.51	3.02	<0.02
Copper	Cu	4.2	3.8	3.4	103	184	3.1	11.2	<0.2
Lead	Pb	7.9	7.2	9.6	15.4	17.2	10.0	10.5	<0.1
Manganese	Mn	136	136	99	173	167	110	101	<0.1
Mercury	Hg	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Molybdenum	Mo	0.2	0.2	0.2	0.6	2.1	0.2	0.4	<0.1
Nickel	Ni	9.4	10.1	7.4	14.5	15.1	7.8	9.8	<0.2
Selenium	Se	1.1	1.0	1.0	1.5	1.5	1.1	1.2	<0.2
Tin	Sn	0.2	0.5	0.4	1.1	1.1	0.3	0.3	<0.1
Titanium	Ti	278	326	247	45	90	247	133	<1
Vanadium	V	30.7	28.9	21.2	45.9	50.7	24.0	24.6	<0.1
Zinc	Zn	22	20	21	67	126	24	31	<1

Table 2: concentrations of metals and metalloids in sediment samples, expressed as mg/kg dry weight of sediment

Samples from the Old farm 1 site, Loch Ewe Poolewe (NGP2001 & NGP2002) and from the Old farm 3 site, Camas an Eilean (NGP2006 & NGP2007) had concentrations of all metals and metalloids that were similar to those in the sample from the control site (NGP2003). In contrast, samples from the Old farm 2 site, Aultbea (NGP2004 & NGP2005) had notably higher concentrations in some cases.

The sample collected at the centre of the Old farm 2 site (NGP2005) yielded a concentration of copper over 50 times that of the control sediment, as well as concentrations of molybdenum and zinc that were 11 and 6 times the control value, respectively. Similarly, the sample collected 10m north of that site (NGP2004) also had an elevated concentration of copper (approximately 30 times that of the control sediment) and, to a lesser extent, of molybdenum and zinc.

Both samples from this site also contained a number of other metals and metalloids at concentrations 2-3 times their respective control concentrations, including arsenic, beryllium, barium, cadmium, tin and vanadium.

It is difficult to define typical background concentrations for metals in coastal marine sediments as sources and concentrations can vary considerably around the coast of the UK depending on local geology and proximity to industrial sources. Perhaps the best reference source remains that of Stevenson (2001),

which collates data on metals in many thousands of marine sediment samples collected by the British Geological Survey over many studies. Compared to the summary statistics presented in that paper, the concentrations of zinc in sediments collected from the control site, the Old farm 1 site and the Old farm 3 site were all in the range of the median (26 mg/kg) for the approximately 9000 sediment samples collated by Stevenson (2001), and slightly below the average (39 mg/kg) for the same large sample set. In contrast, the concentrations of zinc in the two samples collected at the Old farm 2 site were well above the median and mean values cited by Stevenson (2001), and higher also than the 75th percentile (though still well below the maximum reported value of 885 mg/kg).

Median and mean copper concentrations tend to be far lower than those of zinc in marine sediments, with an average of around 4 mg/kg (close to those concentrations found in samples from the control site, the Old farm 1 site and the Old farm 3 site). Against that “typical” background, the levels of copper in the two samples from the Old farm 2 site stand out as containing very high levels of copper, with that at the centre of Old farm 2 site (184 mg/kg) approaching the maximum value of 247 mg/kg reported by Stevenson (2001) for sediments collected around the UK in BGS surveys. High concentrations of copper and zinc, among other metals, have previously been reported in sediments collected beneath caged salmon aquaculture farms (see e.g. review by Dean *et al.* 2007 and analyses conducted in seven Scottish sea lochs by Russell *et al.* 2011), as a result of the presence of these elements at elevated concentrations in salmon feed (including food supplements and preserving agents) and/or the use of copper-based formulations in treating net pens to prevent biofouling. Although it is not possible to conclude for sure that the high levels of zinc and especially of copper in samples from Old farm 2 site arise from such sources, it is clearly a strong possibility which deserves further investigation.

It may also be valuable to analyse more of the sediments collected in the vicinity of Old farm 2 site in order to determine the wider extent of metal contamination. In that context, however, Russell *et al.* (2011) have already reported concentrations of both copper and zinc in sediments collected from beneath and in the wider vicinity of salmon aquaculture cages in a number of Scottish sea lochs, three of them on the West coast and including Loch Ewe, which could provide a baseline for comparison with the current samples. For example, that study reported mean and median values for copper in sediments of 35.8 and 18.6 mg/kg dry weight respectively, in most cases above the values determined in our study but with the exception of the two samples from the Old farm 2 site (which were well above those averages and, in the case of the sample from the centre of the site, approaching the maximum value of copper reported for all sites investigated by Russell *et al.* (2011).

Semi-volatile organics (sVOCs)

Complete lists of the semi-volatile organic compounds identified in the qualitative GC-MS screening analysis are included in Appendix 2, along with the accompanying GC chromatograms. In all cases, the two most prominent peaks visible are for the internal standards deuterated naphthalene and brominated naphthalene which are added to the samples and extracts respectively for quality control purposes. The additional smaller peaks include substances extracted from the samples themselves and a small number of substances that were also present as low-level contaminants in the extraction blanks and which were therefore excluded from the results.

Overall these qualitative screening results indicate that the sediments sampled were not heavily contaminated with semi-volatile organic compounds. Only between 12 and 19 individual compounds could be distinguished as substances originating from the sediment samples themselves, and of those the majority were polycyclic aromatic hydrocarbons (PAHs), sometimes present only at trace levels (identified only in selective ion monitoring, or SIM, mode). As well as being components of crude and some refined oil-based products, PAHs are commonly formed as products of incomplete combustion either from natural (e.g. forest or brush fires) or anthropogenic (e.g. engine emissions) sources. Their common presence across all seven of the samples analysed in the current study, including the control site, could arise from inputs from boat engines over time, but could also arise either from deposition from land or sea-based sources or through sediment transport from other areas. Their presence in these samples in and of itself does not provide strong evidence for a predominance of local anthropogenic sources. Further efforts at source reconciliation would require more extensive sampling and targeted quantitative analysis for PAHs (which was not possible in the context of the current study) but the value of this is questionable given that the levels of contamination appear to be relatively low (through semi-quantitative comparison with the peak areas for the internal standards).

Three of the samples - Control site 1 (NGP20003), Old farm 2 centre (NGP20005) and Old farm 3 10m N (NGP20007) – also contained traces of linear and branched alkanes, which could also have either or both natural and/or anthropogenic origins. Sample NGP20005 contained a prominent peak for elemental sulphur, which almost certainly relates to the presence of naturally occurring sulphur compounds in sediments that are slightly anoxic (and could relate to the fact that the sediments from the Old farm 2 site were collected from deeper water than at the other sites). Both sediments from Old farm site 3 contained the brominated compound 5-bromo-1H-indole, along with a non-brominated indole, compounds that are known to be produced naturally by macroalgae under certain conditions (Mandrekar *et al.* 2019).

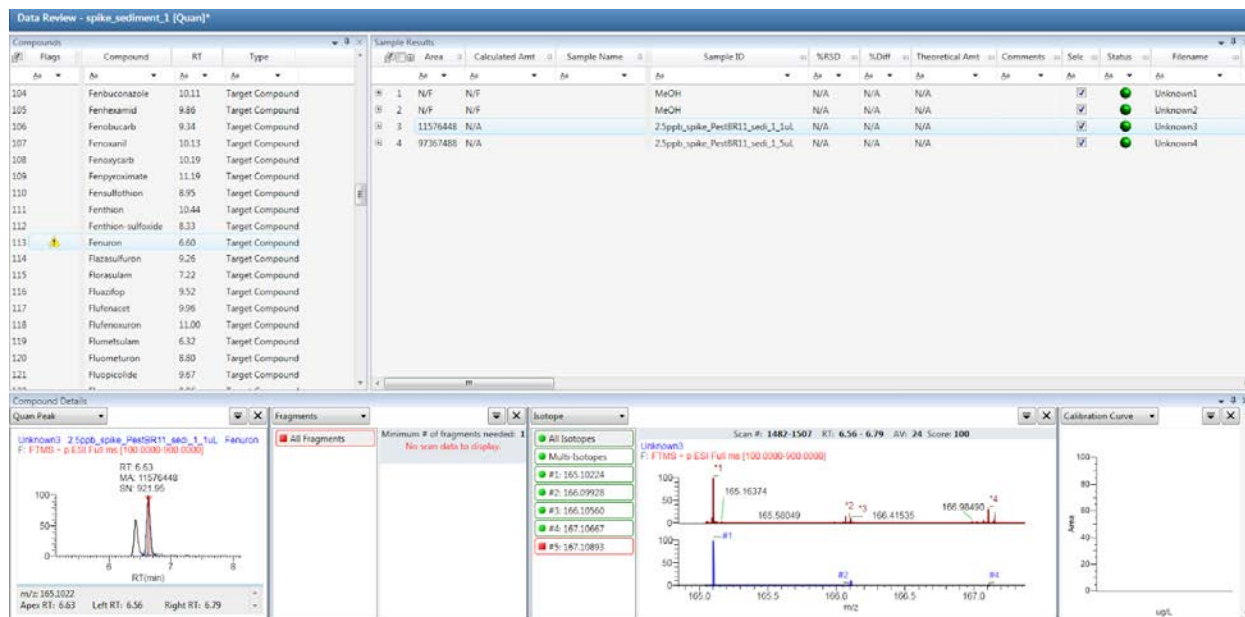
Pesticides & pharmaceuticals

None of the extracted samples contained detectable residues any of the pesticides or veterinary drugs listed in Appendix 1. Percentage recoveries of pesticides from the spiked reference river sediment again varied from substance to substance, but were generally greater than 80%, indicating that the QuEChERS extraction method applied is efficient in extracting these residues from sediments in which they are present and reaffirming that no such residues were present at levels above the limits of detection in any of the sediment samples analysed in this study.

One significant chromatographic peak was present in all the extracted sediment samples, with an exact mass of molecular ion (M+H) of 165.1022, which is identical to that of the once widely used but now obsolete pre-emergence herbicide fenuron. In order to verify this provisional identity, a separate sub-sample of sediment NGP20001 (project sample code 004) was spiked with an external standard solution of fenuron to a concentration of 5 ppb in the sample. Figure 1 below shows two distinct peaks with different retention times on the column in the LC-MS instrument, the first being that of the spiked fenuron standard and the second that of the unknown compound. This spiked samples analysis confirmed that the compound found in the sediment samples with an accurate molecular mass of 165.1022 is nevertheless not fenuron but another, so far unidentified compound. It was not possible within the limits of the current

study to determine the identity of this compound, but its presence was a characteristic common to all the sediment samples analysed is perhaps suggestive of it being a naturally occurring component of the sediments in the region (though the possibility that it is a ubiquitous anthropogenic contaminant of undetermined nature and origin cannot be excluded).

Figure 1: sediment_1 spiked with 5 ppb fenuron:



Concluding remarks

Overall, the results of the analyses we have been able to conduct on these seven sediments, which are a subset of a larger set of samples collected from the sites listed, indicate that there may well be evidence of some historical contamination with metals (especially copper but also zinc) at one of the three former salmon farm sites investigated (designated as Old farm 2, at Aultbea). This is perhaps worthy of further examination, including the analysis of the remaining sediment samples collected from this site and, perhaps, a greater number of samples from the associated control site (though it is worth first examining closely the data available from Russell *et al.* (2011), especially as they included transect samples from Loch Ewe).

In contrast, we were not able to find evidence of significant levels of contamination with synthetic organic compounds (including a wide range of pesticides and pharmaceutical compounds) or of other persistent organic compounds that may have both natural and anthropogenic sources. Although PAHs were found in all seven samples, the levels of these compounds present in these sediments do not appear from our qualitative analysis to be worthwhile investigating further, especially as there were also no clear distinctions between the patterns of contamination at any of the samples analysed in this study, including the control site.

The possibility cannot be excluded that synthetic compounds (such as biocides or pharmaceuticals) other than those specifically analysed for in this investigation may be present in these sediments. However, further targeted analysis would need to be informed by more specific indications of the chemicals that may have been in use at the farms while they were active. If such information arises in the future, the nature of the data files acquired by the mass spectrometer on the LC-MS system is such that it will be possible to search the data from the analyses already conducted for residues of those additional compounds based initially on their published accurate mass and isotopic signatures. If significant peaks matching those characteristics were subsequently found, it would then be necessary to obtain certified standards for those compounds in order to check also for a retention time match (as was carried out to confirm or, in this case, exclude the presence of fenuron in the current study).

It should also be noted that sediments underlying the farms at the time they were active may since have been moved by currents or storm events or become buried with fresh sediment over time, such that some contaminants that may initially have been detectable may now have been dispersed or persist only in deeper layers of sediment. The presence of high levels of copper and zinc at Old farm 2 site provides some indications that mixing and/or burial of sediments may have been more limited at that site, but it is not possible to draw firm conclusions on that based on the analyses conducted to date.

For more information please contact:

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Appendix 1a: Examined pesticides by HPLC-MS technique in positive measurement mode

1	Acetamiprid	41	Chlorantraniliprole
2	Acibenzolar-S-methyl	42	Chlorbromuron
3	Alachlor	43	Chlorfenvinphos, B-
4	Aldicarb-sulfone	44	Chloridazon
5	Allethrin	45	Chloroxuron
6	Ametryn	46	Chlorpyrifos
7	Aminocarb	47	Chlortoluron
8	Ancymidol	48	Cinosulfuron
9	Anilofos	49	Clomazone
10	Aramite	50	Clothianidin
11	Atrazine	51	Coumaphos
12	Avermectin B1a	52	Crotoxyphos_M+NH4
13	Azaconazole	53	Cumyluron
14	Azamethiphos	54	Cyanazine
15	Azinphos-ethyl	55	Cyazofamid
16	Azinphos-methyl	56	Cycloate
17	Azoxystrobin	57	Cycluron
18	Bendiocarb	58	Cyflufenamid
19	Benodanil	59	Desmedipham_M+NH4
20	Benoxacor	60	Desmethyl primicarb
21	Bensulfuron-methyl	61	Desmetryn
22	Benzoximate	62	Diclobutrazol
23	Benzoylprop-ethyl	63	Diclotophos
24	Bitertanol	64	Diethofencarb
25	Boscalid	65	Difenoconazole
26	Bromacil	66	Diflubenzuron
27	Bromuconazole	67	Dimefuron
28	Bupirimate	68	Dimethametryn
29	Buprofezin	69	Dimethenamid
30	Butachlor	70	Dimethoate
31	Butafenacil_M+NH4	71	Dimethomorph
32	Butocarboxim Sulfoxide	72	Dimoxystrobin
33	Butoxycarboxim	73	Dinotefuran
34	Carbaryl	74	Dithiopyr
35	Carbendazim	75	Diuron
36	Carbetamide	76	Dodemorph
37	Carbofuran	77	Epoxiconazole
38	Carbofuran, 3OH-	78	Esprocarb
39	Carfentrazone-ethyl	79	Etaconazol
40	Carpropamid	80	Ethiofencarb sulfone

81	Ethiofencarb Sulfoxide	121	Haloxifop
82	Ethiprole	122	Haloxifop-methyl
83	Ethirimol	123	Heptenophos
84	Ethofumesate	124	Hexaconazole
85	Etoxazole	125	Hexazinone
86	Etrimfos	126	Hexythiazox
87	Fenamidone	127	Imazalil
88	Fenamiphos	128	Imazaquin
89	Fenarimol	129	Imazethapyr
90	Fenazaquin	130	Imidacloprid
91	Fenbuconazole	131	Indoxacarb
92	Fenhexamid	132	Iprovalicarb
93	Fenobucarb	133	Isocarbophos
94	Fenoxanil	134	Isoprocab
95	Fenoxycarb	135	Isoprothiolane
96	Fensulfothion	136	Isoproturon
97	Fenthion	137	Isoxaben
98	Fenthion-sulfoxide	138	Isoxadifen-ethyl
99	Fenuron	139	Kresoxim-methyl
100	Flazasulfuron	140	Lenacil
101	Florasulam	141	Mandipropamid
102	Fluazifop	142	Mefenacet
103	Flufenacet	143	Mepronil
104	Flumetsulam	144	Metamitron
105	Fluometuron	145	Metazachlor
106	Fluopicolide	146	Metconazole
107	Fluopyram	147	Methabenzthiazuron
108	Fluoxastrobin	148	Methiocarb
109	Fluquinconazole	149	Methiocarb-sulfone
110	Flurochloridone	150	Methiocarb-sulfoxide
111	Flusilazole	151	Methoprotryne
112	Flutriafol	152	Methoxyfenozide
113	Forchlorfenuron	153	Metobromuron
114	Formetanate	154	Metolachlor
115	Formothion	155	Metolcarb
116	Fosthiazate	156	Metosulam
117	Fuberidazole	157	Metoxuron
118	Furathiocarb	158	Metrafenone
119	Griseofulvin	159	Metsulfuron-methyl
120	Halofenozide	160	Mevinphos

161	Mexacarbate	201	Quizalofop P
162	Monocrotophos	202	Quizalofop-ethyl
163	Monolinuron	203	Rimsulfuron
164	Napropamide	204	Rotenone
165	Neburon	205	Schradan
166	Nicosulfuron	206	Simeconazole
167	Nuarimol	207	Simetryn
168	Ofurace	208	Spinosad A
169	Omethoate	209	Spinosad D
170	Oxadixyl	210	Spiromesifen
171	Oxamyl + NH4	211	Spirotetramat
172	Paclobutrazol	212	Spiroxamine
173	Penconazole	213	Sulfotep
174	Pencycuron	214	Tebuconazole
175	Phenmedipham	215	Tebufenozide
176	Phenthoate	216	Tebufenpyrad
177	Phoxim	217	Tebuthiuron
178	Picoxystrobin	218	Teflubenzuron
179	Piperonyl-butoxide	219	Terbumeton
180	Piperophos	220	Terbuthylazine
181	Pirimicarb	221	Terbutryn
182	Pirimiphos-methyl	222	Tetraconazole
183	Primisulfuron-methyl	223	Tetramethrin
184	Prochloraz	224	Thiabendazole
185	Profenofos	225	Thiacloprid
186	Promecarb	226	Thiamethoxam
187	Prometon	227	Thidiazuron
188	Prometryn	228	Thiobencarb
189	Propamocarb	229	Tolfenpyrad
190	Propazine	230	Tralkoxydim
191	Propetamphos	231	Triadimefon
192	Propiconazole	232	Triadimenol
193	Propoxur	233	Triazophos
194	Propyzamide	234	Trichlorfon
195	Prosulfocarb	235	Tricyclazole
196	Pymetrozine	236	Tridemorph
197	Pyraclostrobin	237	Trietazine
198	Pyrimethanil	238	Trifloxystrobin
199	Pyroxulam	239	Triflumizole
200	Quinoxyfen	240	Vamidothion
		241	Zoxamide

Appendix 1b: Examined pesticides by HPLC-MS technique in negative measurement mode

242	2,4-D
243	Bentazone
244	Bromoxynil
245	DNOC
246	Fluazinam
247	Flubendiamide
248	Hexaflumuron
249	loxynil
250	MCPA
251	Tepraloxydim

Appendix 1c: Examined veterinary drugs by HPLC-MS technique in positive measurement mode

		18	Flubendazole
1	2-NP-AOZ	19	Fludrocortisone acetate
2	Albendazole	20	Flumequine
3	Aminophenazone	21	Flumethasone
4	Beclomethasone dipropionate	22	Flunixin
5	Chlortetracycline 1	23	Furaltadone
6	Cinoxacin	24	Furazolidone
7	Ciprofloxacin	25	Hydrocortisone
8	Clarithromycin	26	Iprnidazole
9	Danofloxacin	27	Josamycin
10	Dexamethasone	28	Ketoprofen
11	Diclofenac	29	Lomefloxacin
12	Difloxacin	30	Maduramicin
13	Dimetridazole	31	Marbofloxacin
14	Enoxacin	32	Mebendazole
15	Enrofloxacin	33	Meloxicam
16	Erythromycin	34	Methylprednisolone
17	Etodolac	35	Metronidazole

Appendix 1c: Examined veterinary drugs by HPLC-MS technique in positive measurement mode

36	Mometasone furoate	54	Salinomycin
37	Monensin	55	Sarafloxacin
38	Nalidixic acid	56	Spiramycin
39	Naproxen	57	Sulfabenzamide
40	Narasin	58	Sulfacetamide
41	Nitrofurantoin	59	Sulfachloropyridazine
42	Ofloxacin	60	Sulfadiazine
43	Oleandomycin	61	Sulfamerazine
44	Orbifloxacin	62	Sulfamethizole
45	Oxibendazole	63	Sulfamethoxazol
46	Oxolinic acid	64	Sulfaphenazole
47	Oxytetracycline	65	Sulfapyridine
48	Paracetamol	66	Sulfaquinoxaline
49	Phenylbutazone	67	Sulfathiazole
50	Pipemidic acid	68	Sulfisoxazole
51	Prednicarbate	69	Thiabendazole
52	Prednisolone	70	Tilmicosin
53	Ronidazole	71	Tinidazole

Appendix 1c: Examined veterinary drugs by HPLC-MS technique in positive measurement mode

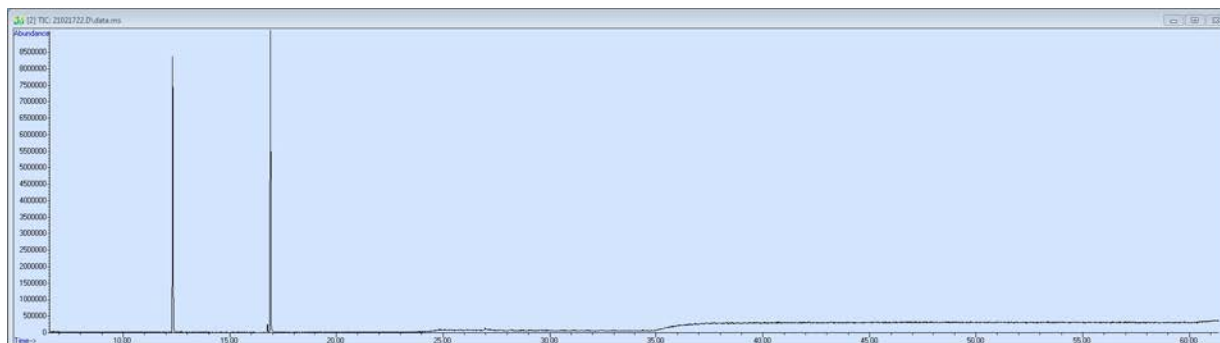
72	Tolfenamic acid
73	Triamcinolone
74	Triamcinolone acetonide
75	Triclabendazole
76	Tylosin

Appendix 1d: Examined veterinary drugs by HPLC-MS technique in negative measurement mode

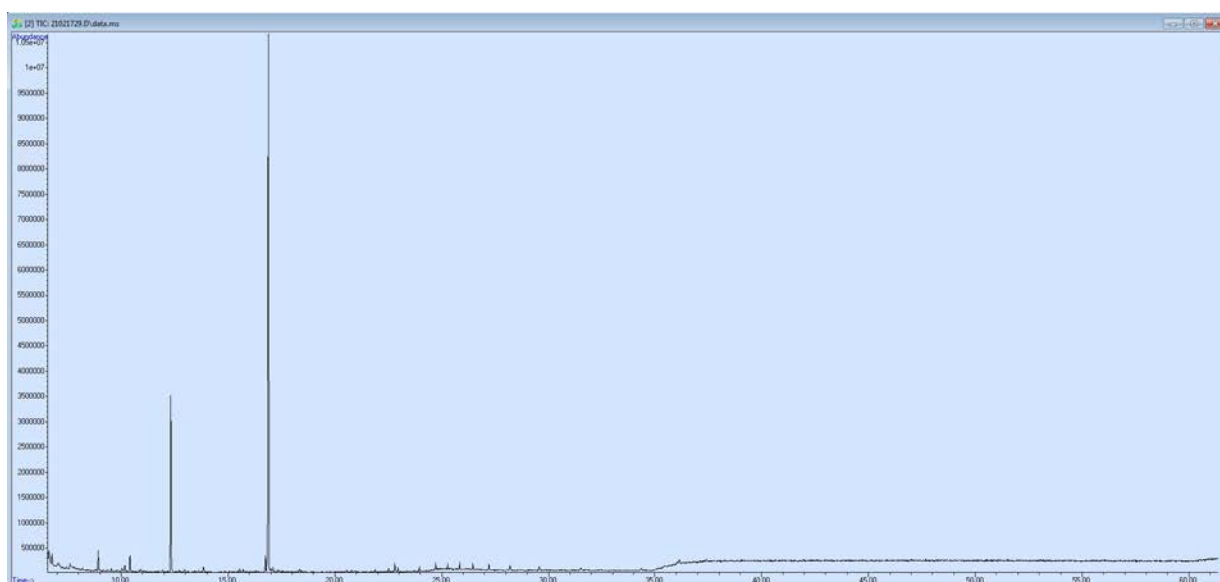
77	Acetylsalicylic acid
78	Carprofen
79	Sulfanitran

Appendix 2: detailed results for qualitative screening of semi-volatile organic compounds

Internal QC standards (deuterated and brominated naphthalene) only



Process/extraction blank



NGP 20001 (004, Old farm 1, centre)

Number of compounds isolated: 12

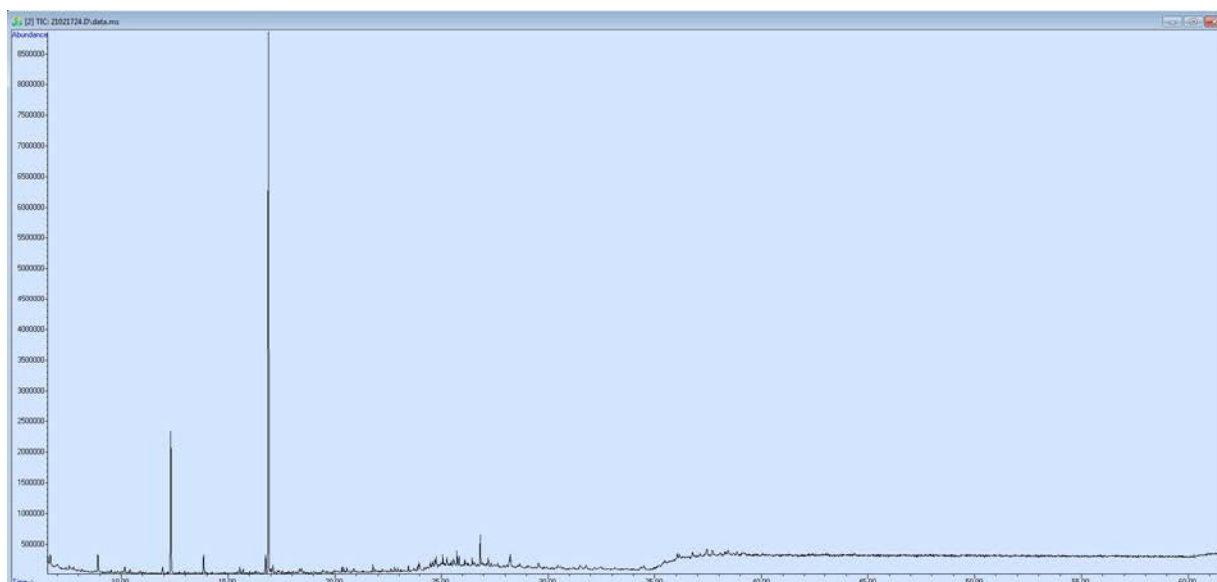
Compounds identified to better than 90%:

1. In total ion chromatogram (TIC)

None

2. In selective ion monitoring mode (SIM)

CAS#	Name
000086-73-7	9H-Fluorene
000120-12-7	Anthracene
000056-55-3	Benzo[a]anthracene
000050-32-8	Benzo[a]pyrene
000207-08-9	Benzo[k]fluoranthene
000205-99-2	Benzo[b]fluoranthene
000191-24-2	Benzo[ghi]perylene
000218-01-9	Chrysene
000206-44-0	Fluoranthene
000193-39-5	Indeno[1,2,3-cd]pyrene
000085-01-8	Phenanthrene
000129-00-0	Pyrene



NGP 20002 (005, Old farm 2, 10m N)

Number of compounds isolated: 13

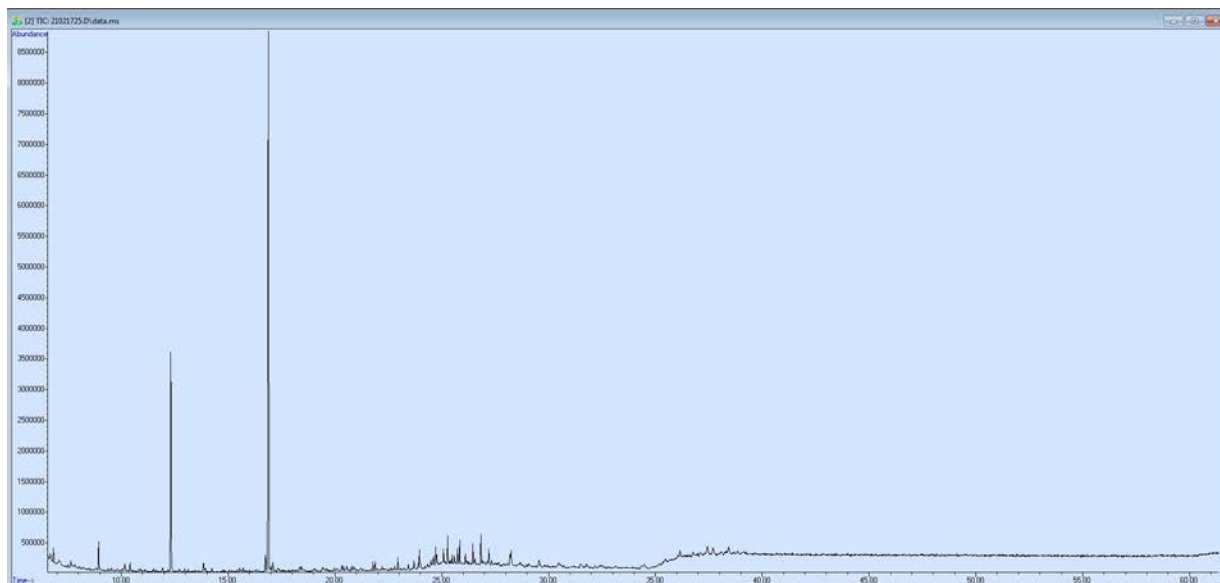
Compounds identified to better than 90%:

1. In total ion chromatogram (TIC)

CAS#	Name
000206-44-0	Fluoranthene
000129-00-0	Pyrene

2. In selective ion monitoring mode (SIM)

CAS#	Name
000086-73-7	9H-Fluorene
000120-12-7	Anthracene
000056-55-3	Benz[a]anthracene
000050-32-8	Benzo[a]pyrene
000207-08-9	Benzo[k]fluoranthene
000205-99-2	Benzo[b]fluoranthene
000191-24-2	Benzo[ghi]perylene
000218-01-9	Chrysene
000193-39-5	Indeno[1,2,3-cd]pyrene
000085-01-8	Phenanthrene



NGP 20003 (007, Control 1, centre)

Number of compounds isolated: 17

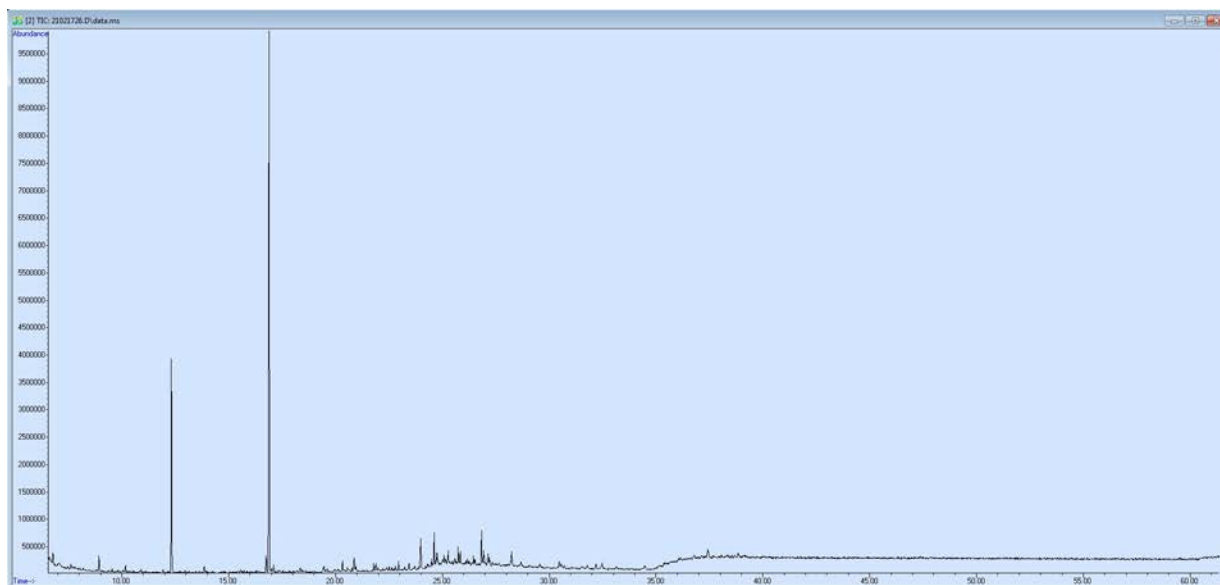
Compounds identified to better than 90%:

1. In total ion chromatogram (TIC)

CAS#	Name
000120-12-7	Anthracene
000191-24-2	Benzo[ghi]perylene
000193-39-5	Indeno[1,2,3-cd]pyrene
000056-55-3	Benzo[a]anthracene
000218-01-9	Chrysene
000206-44-0	Fluoranthene
000129-00-0	Pyrene
000050-32-8	Benzo[a]pyrene
000207-08-9	Benzo[k]fluoranthene
000205-99-2	Benzo[b]fluoranthene
000085-01-8	Phenanthrene
000000-00-0	Linear & branched alkanes, 2 compounds

2. In selective ion monitoring mode (SIM)

CAS#	Name
000086-73-7	9H-Fluorene



NGP 20004 (019, Old farm 2, 10m N)

Number of compounds isolated: 12

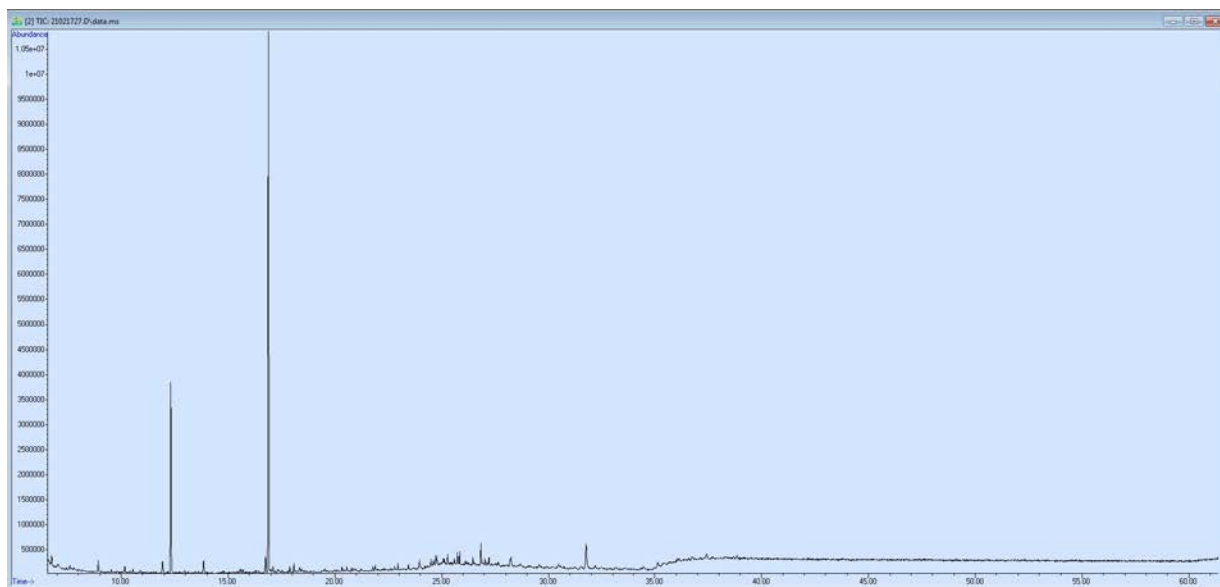
Compounds identified to better than 90%:

1. In total ion chromatogram (TIC)

CAS#	Name
000050-32-8	Benzo[a]pyrene
000207-08-9	Benzo[k]fluoranthene
000205-99-2	Benzo[b]fluoranthene
000218-01-9	Chrysene
000206-44-0	Fluoranthene
000129-00-0	Pyrene

2. In selective ion monitoring mode (SIM)

CAS#	Name
000086-73-7	9H-Fluorene
000120-12-7	Anthracene
000056-55-3	Benz[a]anthracene
000191-24-2	Benzo[ghi]perylene
000193-39-5	Indeno[1,2,3-cd]pyrene
000085-01-8	Phenanthrene



NGP 20005 (021, Old farm 2, centre)

Number of compounds isolated: 16

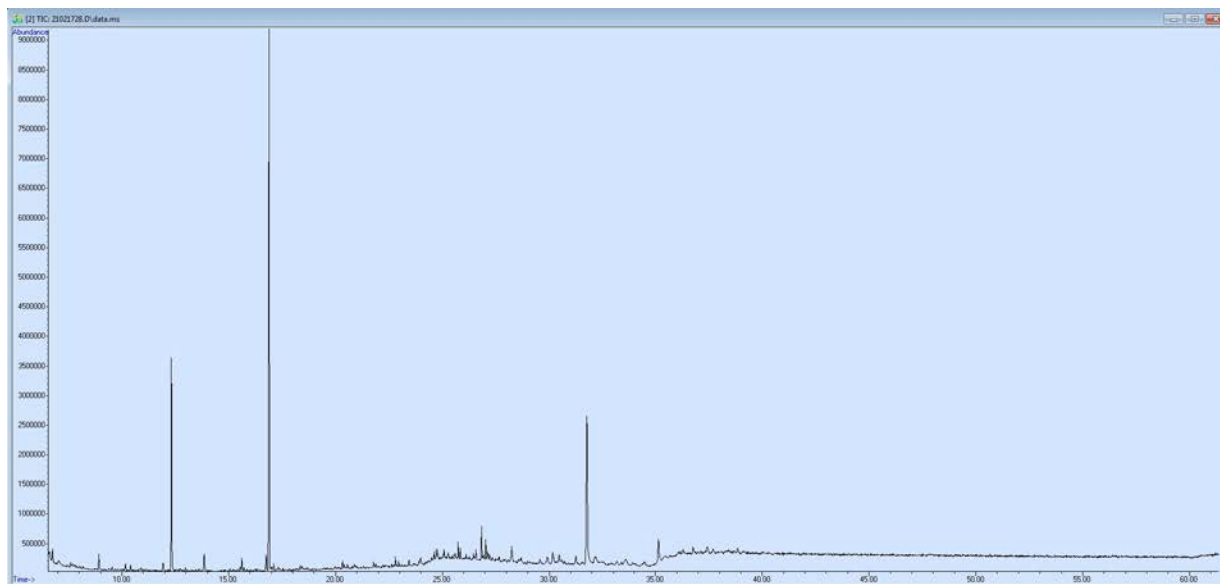
Compounds identified to better than 90%:

1. In total ion chromatogram (TIC)

CAS#	Name
000050-32-8	Benzo[a]pyrene
000218-01-9	Chrysene
000206-44-0	Fluoranthene
000129-00-0	Pyrene
000000-00-0	Linear & branched alkanes, 2 compounds
000085-01-8	Phenanthrene
000191-24-2	Benzo[ghi]perylene
000193-39-5	Indeno[1,2,3-cd]pyrene
007704-34-9	Sulfur

2. In selective ion monitoring mode (SIM)

CAS#	Name
000086-73-7	9H-Fluorene
000120-12-7	Anthracene
000207-08-9	Benzo[k]fluoranthene
000205-99-2	Benzo[b]fluoranthene
000056-55-3	Benz[a]anthracene



NGP 20006 (038, Old farm 3, centre)

Number of compounds isolated: 15

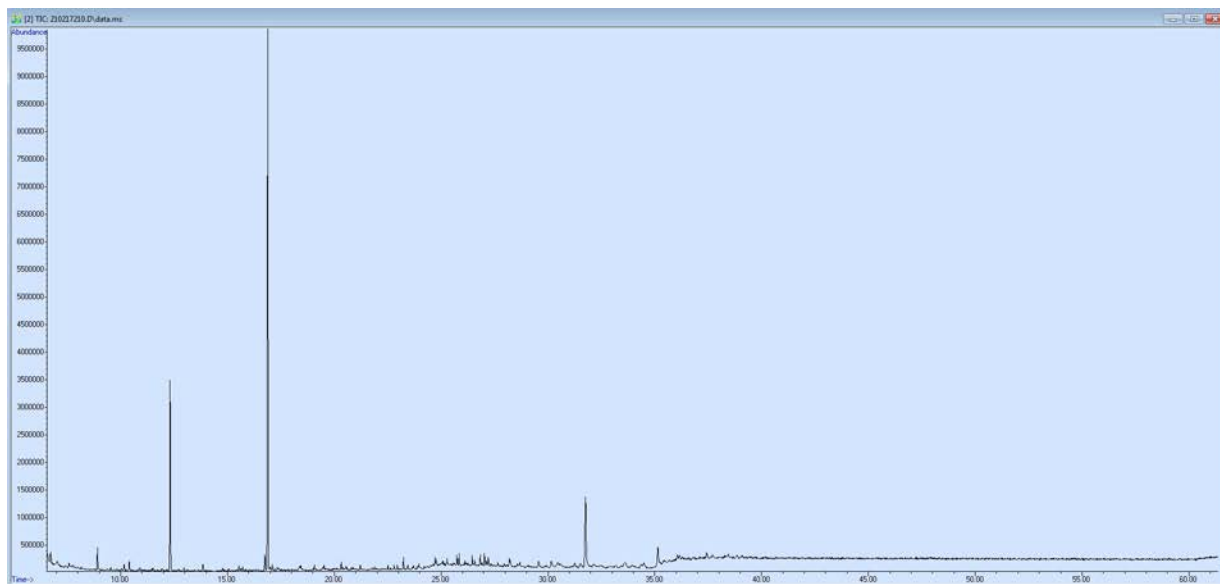
Compounds identified to better than 90%:

1. In total ion chromatogram (TIC)

CAS#	Name
000120-72-9	1H-Indole
010075-50-0	1H- Indole, 5-bromo-
000205-99-2	Benzo[b]fluoranthene
000050-32-8	Benzo[a]pyrene

2. In selective ion monitoring mode (SIM)

CAS#	Name
000086-73-7	9H-Fluorene
000120-12-7	Anthracene
000056-55-3	Benz[a]anthracene
000207-08-9	Benzo[k]fluoranthene
000191-24-2	Benzo[ghi]perylene
000218-01-9	Chrysene
000206-44-0	Fluoranthene
000193-39-5	Indeno[1,2,3-cd]pyrene
000085-01-8	Phenanthrene
000129-00-0	Pyrene



NGP 20007 (040, Old farm 3, 10m N)

Number of compounds isolated: 19

Compounds identified to better than 90%:

1. In total ion chromatogram (TIC)

CAS#	Name
000120-72-9	1H-Indole
010075-50-0	1H- Indole, 5-bromo-
000000-00-0	Linear & branched alkanes, 2 compounds
000085-01-8	Phenanthrene

2. In selective ion monitoring mode (SIM)

CAS#	Name
000086-73-7	9H-Fluorene
000120-12-7	Anthracene
000056-55-3	Benz[a]anthracene
000050-32-8	Benzo[a]pyrene
000207-08-9	Benzo[k]fluoranthene
000205-99-2	Benzo[b]fluoranthene
000191-24-2	Benzo[ghi]perylene
000218-01-9	Chrysene
000206-44-0	Fluoranthene
000193-39-5	Indeno[1,2,3-cd]pyrene
000129-00-0	Pyrene

