

A Fast Method for Identification and Semi-Quantitation of Soil Contaminants Using SPME and the PolarisQ GC-MS

Gail Harkey, Thermo Electron Corporation, Schaumburg, IL

Overview

This project compares concentrations of contaminants from soil extracts using 3 different methods in gas chromatography-mass spectrometry (GC-MS). Soil toxicity levels will be compared by running methods using Solid Phase MicroExtraction (SPME) fibers, EPA Method 8270, and Microtox Solid Phase Tests (SPTs).

Introduction

Remedial investigations of sites historically contaminated with petroleum hydrocarbons typically use measurements of contaminants (e.g., EPA Method 8270) to determine risk. Soil extraction and analytical methods can be lengthy and costly. The objective of this study was to compare relative concentrations of soil contaminants by two methods: EPA Method 8270, and SPME fibers coupled to GC-MS. The research compared toxicity of the soils to determine whether relative contaminant concentrations would correlate with EC50 values obtained from Microtox SPTs.

Methods

Three historically contaminated soils were extracted and analyzed using EPA Method 8270. Relative concentrations of contaminants were also obtained by immersing SPME fibers into the soil samples and analyzing them with GC-MS. The relative contaminant levels in the soils were then compared with the EC50 values obtained with Microtox SPTs.

Soils were extracted and analyzed following EPA Method 8270 protocol and concentrations of 16 polycyclic aromatic hydrocarbons (PAHs) were reported.

100 μm polydimethylsiloxane SPME fibers were directly immersed into soil samples for 20 min at room temperature, then placed into the split/splitless injector of a Thermo Electron TRACE gas chromatograph and analyzed as follows:

TRACE GC

Column:	Rtx®-5MS, 0.25 mm x 30 m; 0.25 μm film thickness
Oven:	50°C, 4.0 min
Ramp:	20°C/min to 290°C, hold 10 min
SPME fiber desorption time:	5 min
Injector Temp:	200°C
Mode:	Split
Split Ratio:	10:1
Constant Flow:	1.0 mL/min He

POLARISQ ION TRAP MASS SPECTROMETER

Source Temp:	200°C
Ionization Mode:	Electron Ionization
Scan Type:	Full Scan, 30-600 m/z

Microtox SPTs followed the manufacturer's protocol. Soil extracts were combined with Microtox solid phase diluent in a 1:5 (w/v) ratio and mixed for 10 min prior to routine SPT testing.

Results

PAH concentrations between Method 8270 (Table 1) and SPME fiber extractions were relatively similar, except for chrysene and dibenzo(a,h)anthracene, which were not detected with SPME/GC-MS (Figure 1). Additional contaminants were reported in the SPME/GC-MS analysis where Soils 2 and 3 contained large quantities of methyl naphthalenes (Table 2). A large amount of lower molecular weight naphthalenes was found in Soils 2 and 3, with Soil 2 containing proportionately more methyl naphthalenes and other lower molecular weight compounds than the other soils (Table 2, Figure 2). Toxicity was greatest for Soil 2, which contained the lowest amount of total contaminants (Table 3).

Key Words

- PolarisQ Ion Trap
- EPA Method 8270
- PAH Analysis
- Soil extraction
- SPME
- SPT

Mean Concentration (ppm)

COMPOUND	SOIL 1	SOIL 2	SOIL 3
Naphthalene	388	3	253
Acenaphthylene	99	3	299
Acenaphthene	418	3	36
Fluorene	195	10	488
Phenanthrene	1373	35	916
Anthracene	143	18	569
Fluoranthene	4117	87	1308
Pyrene	3637	110	894
Benzo(a)anthracene	529	8	297
Chrysene	678	7	349
Benzo(b)fluoranthene	698	6	169
Benzo(k)fluoranthene	298	7	84
Benzo(a)pyrene	734	9	186
Dibenzo(a,h)anthracene	425	7	50
Benzo(ghi)perylene	689	10	25
Indeno(1,2,3-cd)pyrene	804	11	51
Total PAHs	15292	355	3494

Table 1: Average concentrations of PAHs in soils

Area abundance (x 10,000)

COMPOUND	SOIL 1	SOIL 2	SOIL 3
2-methyl naphthalene	5	23	51
Benzocycloheptatriene	4	20	63
Biphenyl	14	3	1
Dimethyl naphthylene	3	62	102
Trimethyl naphthylene	0	10	10
Tetradecane	0	10	0
3-methyl anthracene	4	1	20
Methyl phenanthrene	6	2	26
2-phenyl naphthalene	32	0	3
2-methyl fluoranthene	18	0	11
1-methyl pyrene	8	0	8
Dimethyl pyrene	2	0	1
Triphenylene	254	0	12
Perylene	119	0	3

Table 2: Additional contaminants reported with SPME fiber extracts

SOIL ID	EC50 (MG/ML EXTRACTANT + 1 SD OF TRIPLICATE SAMPLES)
1	2.099 (0.98)
2	0.435 (0.13)
3	3.125 (0.14)

Table 3: EC50 values obtained with Microtox Solid Phase Tests

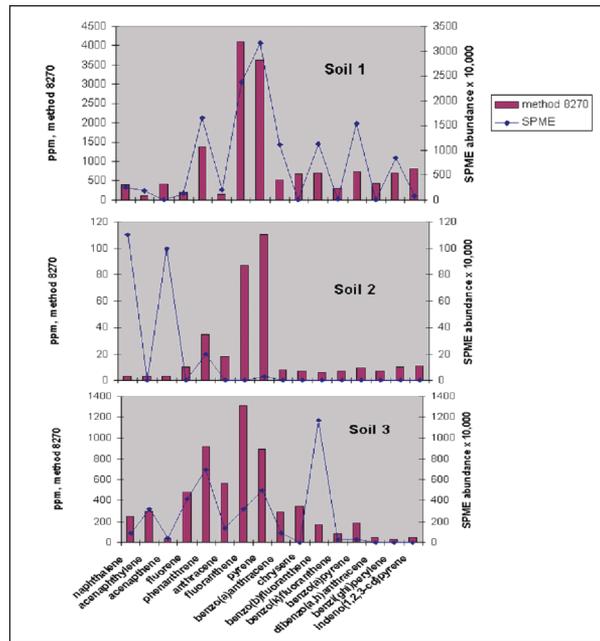


Figure 1: Relative amounts of PAHs in Method 8270 and SPME extracts

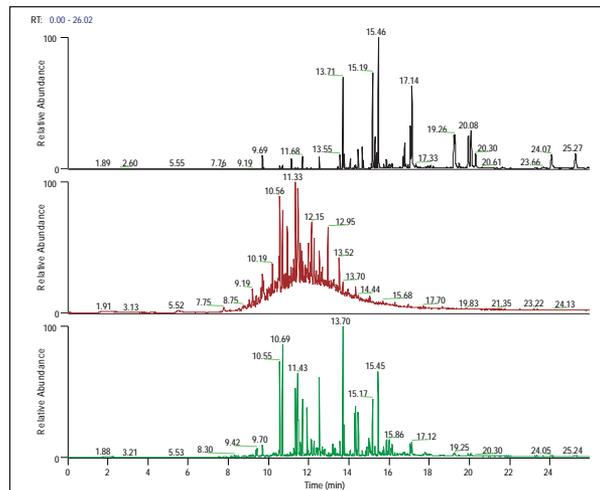


Figure 2: TICs of Soils Sampled with SPME

Conclusions

SPME/GC-MS was used to demonstrate a fast and semi-quantitative method to determine relative soil contaminant concentrations. This screening method tracked most PAHs relative to the established EPA method of quantitation. However, toxicity was greatest in the soil possessing the lowest contaminant concentration. These results show that routine measurements of volatile and semi-volatile compounds may not be an adequate way to establish risk. The results show that a rapid and easy screening method such as SPME/GC-MS can be used to identify and estimate relative concentrations of historically contaminated soils.

Acknowledgements

Thanks to B. Paterek and V. Trbovic of the Gas Technology Institute in Des Plaines, IL for providing soil samples and Method 8270 results.

In addition to these offices, Thermo Electron Corporation maintains a network of representative organizations throughout the world.

- Australia**
+61 2 9898 1244
- Austria**
+43 1 333 50340
- Belgium**
+32 2 482 30 30
- Canada**
+1 800 532 4752
- China**
+86 10 5850 3588
- France**
+33 1 60 92 48 00
- Germany**
+49 6103 4080
- Italy**
+39 02 950 591
- Japan**
+81 45 453 9100
- Netherlands**
+31 76 587 98 88
- Nordic**
+46 8 556 468 00
- South Africa**
+27 11 570 1840
- Spain**
+34 91 657 4930
- Switzerland**
+41 61 48784 00
- UK**
+44 1442 233555
- USA**
+1 800 532 4752

www.thermo.com

ISO 9001
DNV-CERT-00203-94-A0

Thermo Electron Limited, Milan, Italy
is ISO Certified.

©2005 Thermo Electron Corporation. All rights reserved. All other trademarks are the property of Thermo Electron Corporation and its subsidiaries.

Specifications, terms and pricing are subject to change. Not all products are available in all countries. Please consult your local sales representative for details.

AN10099_E 01/05C

Thermo
ELECTRON CORPORATION