

# Accelerated Solvent Extraction — GC-MS Analysis and Detection of Polycyclic Aromatic Hydrocarbons in Soil

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## Key Words

Accelerated solvent extraction, Gas Chromatography - Mass Spectrometry (GC-MS), soil, Polycyclic Aromatic Hydrocarbons, PAHs

## Introduction

Accelerated solvent extraction is an automated extraction technique that significantly streamlines sample preparation. A commonly used solvent is pumped into an extraction cell containing the sample, which is then brought to an elevated temperature and pressure. Minutes later, the extract is transferred from the heated cell to a standard collection vial for cleanup or analysis. The entire extraction process is fully automated and performed in minutes for fast and easy extraction with low solvent consumption.

Previously, the extraction of polycyclic aromatic hydrocarbon (PAH) compounds from environmental materials including soils, sludge, and other solid wastes typically required large amounts of solvents. Soxhlet, for example, can use 250 to 500 mL of solvent for most environmental samples. Recent and anticipated changes in environmental regulations will cause severe restrictions on the amount of solvent usage in laboratories worldwide. Accelerated solvent extraction was developed to meet the new requirements for reducing solvent usage in the preparation of solid waste samples.

Accelerated solvent extraction provides a more convenient, faster, and less solvent intensive method than previously available for the extraction of PAHs from solid wastes. PAH recoveries by accelerated solvent extraction are equivalent to other more solvent intense methods such as Soxhlet or sonication. Accelerated solvent extraction also avoids the problem of multiple washing procedures associated with sonication. Accelerated solvent extraction



extracts from a 10 g sample of a typical soil in about 12 min with a total solvent consumption of approximately 15 mL. The procedures described in this application note meet the requirements for the extraction of PAHs from solid waste as described in U.S. EPA Method 3545A. This method is applicable to solid wastes including soils, sludges, and sediments.

This Application Note updates the work that was done in Thermo Scientific Application Note 313 (AN 313) for the extraction of PAHs from environmental samples. A Thermo Scientific™ Dionex™ ASE™ 350 Accelerated Solvent Extractor system was used for the extraction and Thermo Scientific™ ISQ™ Single Quadrupole GC-MS system to analyze 16 PAHs from soil.

**Accelerated Solvent Extraction Conditions**

Solvent:	Methylene chloride/acetone (1:1 v/v)
Temperature:	100 °C
Static Extraction Time	5 min
Number of Static Cycles:	2
Purge Volume:	60%
Purge Time:	90 sec
Extraction Cell Size:	34 mL stainless steel
Filters:	Cellulose (30 mm)
Total Extraction Time per Sample:	20 min
Total Solvent Volume per Sample:	40 mL
Sample Size:	10 g

The extracted liquid was concentrated on a nitrogen evaporator until nearly dry. The extract was reconstituted with 0.5 mL of acetone and passed through a 0.45 µm filter membrane prior to GC-MS analysis.

**Conditions for Gas Chromatography - Mass Spectrometry**

Chromatography column: Thermo TR-5, 60.0 m × 0.25 mm × 0.25 µm

Programmed temperature increases: 60 °C (maintained for 2 min); 25 °C/min up to 160 °C (maintain for 1 min), then an increase of 6 °C/min up to 300 °C (maintain for 8 min).

For splitless mode, the SSL injector filling inlet temperature was 300 °C with a splitless time of 1 min. The carrier gas was high-purity helium (99.999%) with a column flow rate of 1 mL/min. The mass spectrometry transferline temperature was 320 °C and the ion source temperature was 280 °C

EI ionization was accomplished with an electronic energy of 70 eV with selected ion mode (SIM) scanning. See Table 1 for the quantitative and qualitative ions for each compound.

Table 1. Retention times, quantitative ions, and qualitative ions for the 16 PAHs.

Number	Name of Compound	Retention Time (min)	Quantitative Ions (m/z)	Qualitative Ions (m/z)
1	Naphthalene	6.05	128	127,129
2	Acenaphthylene	8.49	152	151,153
3	Acenaphthene	8.83	153	152,154
4	Fluorene	10.12	166	165,167
5	Phenanthrene	13.06	178	176,179
6	Anthracene	13.24	178	176,179
7	Fluoranthene	17.35	202	200,203
8	Pyrene	18.20	202	200,203
9	Benzo(a)anthracene	22.95	228	226,229
10	Chrysene	23.09	228	226,229
11	Benzo(a)fluoranthene	27.97	252	250,253
12	Benzo(k)fluoranthene	27.07	252	150,156
13	Benz(a)pyrene	28.13	252	250,253
14	Indeno(1,2,3-cd)pyrene	31.79	276	274,277
15	Dibenzo(a,h)anthracene	31.86	278	276,279
16	Benzo(g,h,i)perylene	32.78	276	138,277

### Methodology

Standard solutions were prepared for 16 types of polycyclic aromatic carbons, with the respective concentrations: 20.0, 50.0, 100.0, 200.0 and 500.0 µg/L. The method outlined above was used to carry out sample analysis and investigate the linearity of each component within a concentration range of 20.0–500.0 µg/L.

A signal-to-noise ratio of three was used to calculate the detection limit of each component. The results showed good linear relationships for the 16 types of PAHs, with detection limits between 0.10 and 3.90 µg/L. Instrument detection limits were very good (see Table 2 for results). The chromatogram for a 200 ng/mL standard sample is shown in Figure 1.

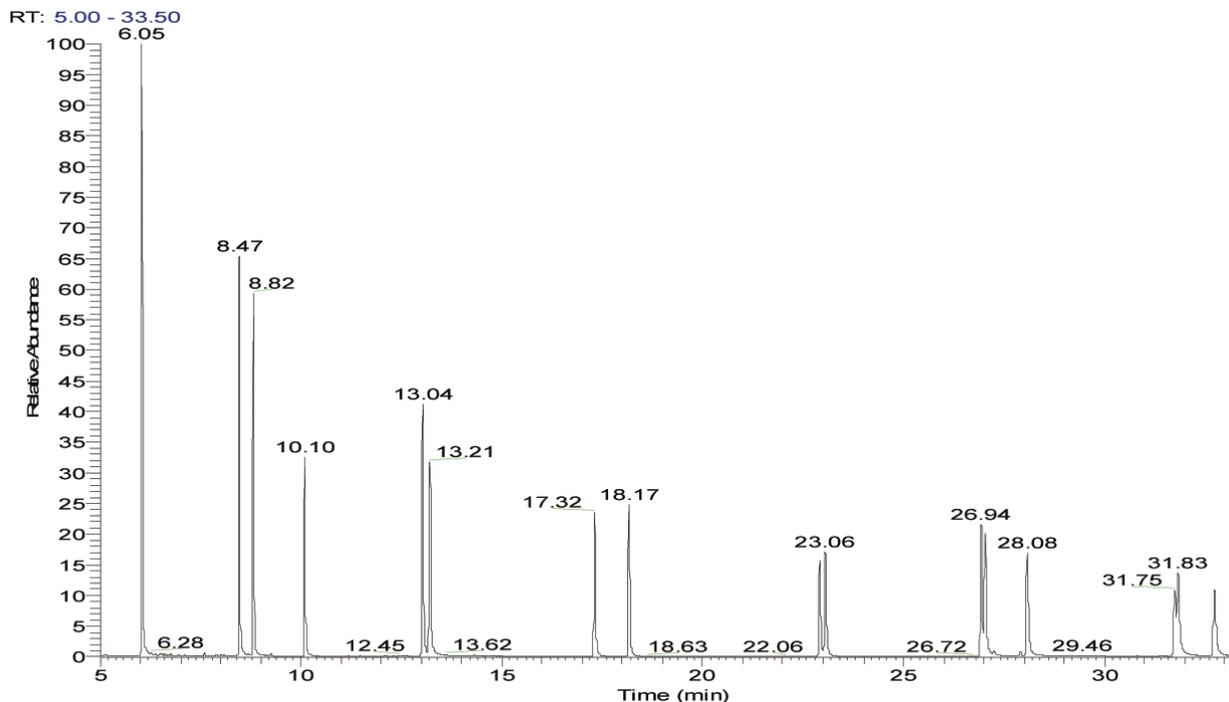


Figure 1. Total Ion Chromatogram for 200 ng/mL PAH Standard.

Table 2. Linear equation, linear range (ng/g), linear coefficient and detection limit (ng/g) for 16 PAH compounds.

Analyte	Linear Equation	Linear Range (ng/g)	Liner Coefficient	Detection Limits (ng/g)
Naphthalene	$Y=5178.3+9135.7X$	20–500	99.74	0.10
Acenaphylene	$Y=18240.7+50535.2X$	20–500	99.87	0.12
Acenaphthene	$Y=18950.6+36413.5X$	20–500	99.99	0.14
Fluorene	$Y=-21178.1+33985.8X$	20–500	99.97	0.14
Phenanthrene	$Y=-39858.5+40100.4X$	20–500	99.86	0.19
Anthracene	$Y=-43418+37336.1X$	20–500	99.7	0.28
Fluoranthene	$Y=-56630.4+31183.5X$	20–500	99.6	0.29
Pyrene	$Y=-60689.1+34303.8X$	20–500	99.55	0.28
Benzo(a)anthracene	$Y=-57184.5+21435.1X$	20–500	99.37	0.40
Chrysene	$Y=-53097.4+28211.1X$	20–500	99.9	0.30
Benzo(a)fluoranthene	$Y=-83317.2+31775.1X$	20–500	99.88	0.79
Benzo(k)fluoranthene	$Y=-34068.3+33667.3X$	20–500	99.9	1.01
Benz(a)pyrene	$Y=-45101.5+29060.5X$	20–500	99.62	1.50
Indeno(1,2,3-cd)pyrene	$Y=-38746.8+34967.2X$	20–500	99.92	3.70
Dibenzo(a,h)anthracene	$Y=-52161.9+28385X$	20–500	99.97	3.30
Benzo(g,h,i)perylene	$Y=-8250.1+29841.6X$	20–500	99.81	3.90

## Experimental Section

### Instruments and Equipment

- Dionex ASE 350 Accelerated Solvent Extractor system
- Thermo Scientific™ TRACE™ GC Ultra Gas Chromatograph
- Thermo Scientific™ ISQ™ Single Quadrupole GC-MS
- Thermo Scientific™ TriPlus™ RSH Autosampler
- Reacti-Therm III Nitrogen Evaporator

### Reagents

The 16 standard PAHs were purchased from Shanghai ANPEL Scientific Instrument Co., Ltd., the silica and cellulose filter membranes were purchased from Thermo Fisher Scientific and reagents such as n-hexane and acetone were purchased from the Sinopharm Group.

### Sample Testing and Spike Recovery

This method was used to carry out analysis and detection in soil samples from different areas. The test results show that this method can accurately detect the content of each component, and that none of the components produced interference peaks (Figure 2). At the same time, a spike test was carried out on a random soil sample (spike concentrations: 50 and 200 µg/L) to investigate the spike recovery in the 16 types of PAHs. The test results show that spike recovery rates for each component are between 70% and 120%, and are in compliance with the requirements for everyday analysis and detection. At the same time, the same sample was injected five times, and an instrument RSD of 0.7–5.3% was obtained, in compliance with detection requirements.

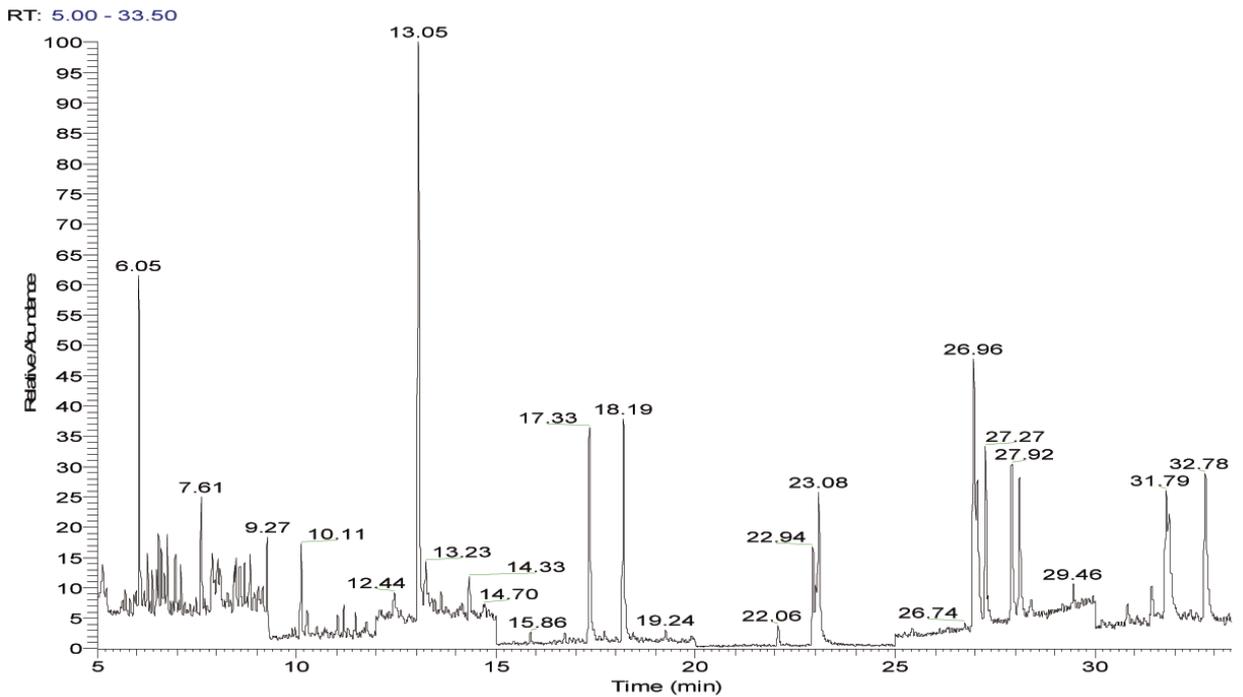


Figure 2. Sample ion chromatogram of 16 PAH compounds.

Table 3: Spike recovery rate test.

Analyte	% Recovery (50 ng/g)	% Recovery (200 ng/g)	%RSD
Naphthalene	113.8	92.6	1.2
Acenaphylene	97.2	106.7	1.7
Acenaphthene	103.1	100.5	2.1
Fluorene	115.6	89.5	3.6
Phenanthrene	112.1	100.2	2.2
Anthracene	98.3	100.7	0.7
Fluoranthene	86.7	91.6	3.5
Pyrene	115.3	88.7	3.1
Benzo(a)anthracene	110.1	95.0	1.8
Chrysene	109.5	93.4	4.2
Benzo(a)fluoranthene	103.4	85.0	0.9
Benzo(k)fluoranthene	101.2	95.8	3.0
Benz(a)pyrene	116.2	102.1	1.9
Indeno(1,2,3-cd)pyrene	97.0	101.1	3.3
Dibenzo(a,h)anthracene	110.7	106.5	4.8
Benzo(g,h,i)perylene	112.3	99.0	5.3

## Summary

When using accelerated solvent extraction to extract PAHs from soil, the spike recovery rate for the 16 PAHs is between 86.7% and 116.2%, showing that accelerated solvent extraction is suitable for extracting PAHs from soil. Pre-processing a sample using accelerated solvent extraction takes only 20 min and requires only 40 mL of solution. Accelerated solvent extraction is simple, fast, and highly efficient and reduces the amount of solution required.

The new-generation ISQ Single Quadrupole GC-MS system possesses high contamination resistance and high sensitivity. Using the ISQ Single Quadrupole GC-MS system to detect the 16 kinds of PAH is simple, highly efficient, fast and accurate, with high sensitivity and good usability. The spike recovery rate, accuracy and sensitivity all satisfy the quality requirements for the organic analysis of soil samples.

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