

GC-MS

APPLICATION NOTES

Determination of 16 polycyclic aromatic hydrocarbons in soil by gas chromatography-mass spectrometry



GC-MS



Introduction

An analytical method for the simultaneous detection of 16 PAHs in soil by gas chromatography-mass spectrometry (GC-MS) was established with reference to the environmental standard HJ 805-2016. An Equity-5 (30 m × 0.25 mm × 0.25 μm) quartz capillary column was used to separate the components, and the mass spectrometer was used for the detection, characterization by comparing the retention time and standard mass spectra with the standard substances and their characteristic ions, and quantification by external standard method.

Experimental

Main equipment and reagents

Instrument	Aludra gas chromatograph mass spectrometer
Chromatographic Column	Equity-5 (30 m × 0.25 mm × 0.25 μm) capillary column
Extraction Device	Pressurized fluid extractor
Purification Device	Gel permeation chromatograph
Concentration Device	Nitrogen blow concentrator or rotary evaporation concentrator
Laboratory Equipment	Analytical balance, pipette gun and its supporting tip, microsyringe, etc.
Carrier Gas	High purity helium
Reagent	16 kinds of PAH mixed standard (1000 μg/mL), acetone, hexane (chromatographic pure)

Preparation of standard solutions

Pipette 10 μL, 25 μL, 50 μL, 100 μL 1000 μg/mL PAHs in a 5 mL volumetric flask, acetone: n-hexane (1:1) to the scale, shaking, that is, the concentration of 2 μg/mL, 5 μg/mL, 10 μg/mL, 20 μg/mL PAHs standard series solution; take 1 μL on the GC-MS analysis.

10 μg/mL, 20 μg/mL; take 1 μL to be analyzed by GC-MS.

Gas chromatograph conditions

Column	Equity-5 (30 m × 0.25 mm × 0.25 μm) quartz capillary column
Injection	Non-split injection
Injection Volume	1 μL
Injection Port	280°C
Flow Mode	Constant flow mode
Column Flow	1 mL/min
Carrier Gas	High purity helium
Purge Flow	3 mL/min
Column Temperature	60°C held for 1 minute, with 20°C/min rate to 200°C, held for 4 minutes, then 10°C/min to 300°C held for 10 minutes. Keep 4 minutes, then increase to 300°C with 10°C/min and keep 10 minutes

Mass spectrometer conditions

Ion Source	El Source
Ion Source Temperature	230°C
Electron Energy	70 eV
Scanning Mode	Full scan qualitatively, selective ion scan quantitatively
Scanning Range	45 u – 450 u
Interface Temperature	290°C
Solvent Delay	5.5 minutes
Electron Multiplier High Voltage	1000 V
Acquisition Time	5.7 minutes to 32 minutes

NOTE: The analytical conditions were optimized on the basis of HJ 805-2016 instrumental conditions, with better naphthalene peak shape and relatively higher peak intensity.

Results

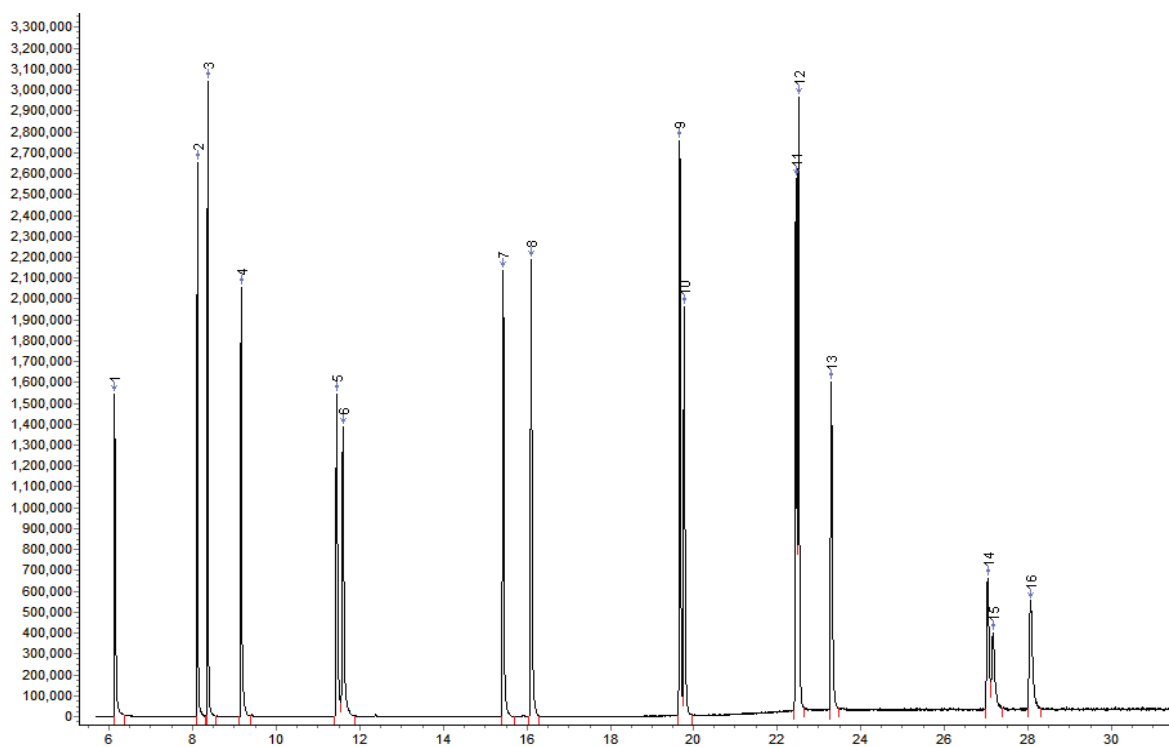


Figure 1: Full-scan spectra of 16 PAHs mixed standards

No.	Description	CAS No.	Retention Time (min)	Qualitative Ions	Quantitative Ions
1	Naphthalene	91-20-3	6.14	128,127	128
2	Acenaphthylene	208-96-8	8.11	152,151,153	152
3	Acenaphthene	83-32-9	8.36	153,154,152	154
4	Fluorene	86-73-7	9.15	165,166,167	166
5	Phenanthrene	85-01-8	11.43	178,176	178
6	Anthracene	120-12-7	11.57	178,176	178
7	Fluoranthene	206-44-0	15.41	202,200,101	202
8	Pyrene	129-00-0	16.08	202,200,101	202
9	Benz[a]anthracene	56-55-3	19.64	228,226,114	228
10	Chrysene	218-01-9	19.75	228,226,114	228
11	Benzo[b]fluoranthene	205-99-2	22.42	252,250,126	252
12	Benzo[k]fluoranthene	207-08-9	22.48	252,250,126	252
13	Benzo[a]pyrene	50-32-8	23.26	252,250,126	252
14	Indeno[1,2,3-cd]pyrene	193-39-5	26.98	276,274,138	276
15	Dibenz[a,h]anthracene	53-70-3	27.10	278,276	278
16	Benzo[g,h,i]perylene	191-24-2	28.00	276,274	276

Table 1: Information on 16 PAHs

No.	Description	Retention Time (min)	Curvilinear Equation	Correlation Coefficient
1	Naphthalene C ₁₀ H ₈	6.14	$Y = 267427.06087 X + 22184.043478$	0.99973
2	Acenaphthylene	8.11	$Y = 278716.832676 X - 69340.458076$	0.99960
3	Acenaphthene	8.36	$Y = 180532.313043 X + 2706.652174$	0.99981
4	Fluorene C ₁₃ H ₁₀	9.15	$Y = 198746.608696 X - 29034.565217$	0.99992
5	Phenanthrene C ₁₄ H ₁₀	11.43	$Y = 284807.872037 X - 25384.200311$	0.99971
6	Anthracene	11.57	$Y = 314603.031028 X - 120799.444995$	0.99899
7	Fluoranthene	15.41	$Y = 385384.054348 X - 80303.282609$	0.99986
8	Malva sylvestris	16.08	$Y = 381411.391304 X - 74339.434783$	0.99990
9	Benz[a]anthracene	19.64	$Y = 448638.367285 X - 222265.340384$	0.99950
10	Chrysene	19.75	$Y = 429776.926736 X - 90574.163349$	0.99890
11	Benzo[b]fluoranthene	22.42	$Y = 493858.326063 X - 515021.103383$	0.99933
12	Benzo[k]fluoranthene	22.48	$Y = 502623.026998 X - 309183.124285$	0.99919
13	Benzo[a]pyrene	23.26	$Y = 351742.9 X - 442719.000000$	0.99922
14	Indeno[1,2,3-cd]pyrene	26.98	$Y = 26220.21436 X - 58156.588655$	0.99648
15	Dibenz[a,h]anthracene	27.10	$Y = 201633.291304 X - 440711.434783$	0.99922
16	Benzo[g,h,i]zone	28.00	$Y = 246713.919565 X - 420668.021739$	0.99896

Table 2: Linear equations and linear correlation coefficients for 16 PAHs

No.	Description	Retention Time (min)	Soil Samples (mg/kg)
1	Naphthalene	6.14	ND
2	Acenaphthylene	8.11	ND
3	Acenaphthene	8.36	ND
4	Fluorene	9.15	ND
5	Phenanthrene	11.43	ND
6	Anthracene	11.57	ND
7	Fluoranthene	15.41	ND
8	Malva sylvestris	16.08	ND
9	Benz[a]anthracene	19.64	ND
10	Chrysene	19.75	ND
11	Benzo[b]fluoranthene	22.42	ND
12	Benzo[k]fluoranthene	22.48	ND
13	Benzo[a]pyrene	23.26	ND
14	Indeno[1,2,3-cd]pyrene	26.98	ND
15	Dibenz[a,h]anthracene	27.10	ND
16	Benzo[g,h,i]zone	28.00	ND

Table 3: Sample test results

Note: ND indicates not detected

Conclusion

In this paper, an analytical method for the simultaneous detection of 16 PAHs in soil by gas chromatography-mass spectrometry (GC-MS) was established with reference to the environmental standard HJ 805-2016; and the instrumental analytical conditions were optimized on the basis of the standard method, which led to sharper naphthalene peaks, higher peak intensities and better repeatability. The components were separated on an Equity-5 (30 m × 0.25 mm × 0.25 μm) quartz capillary column, detected by mass spectrometer and quantified by external standard method. From the experimental results, the linear relationship was good, the precision was good, and the standard requirements were fully met.