RayKol

Knowledge Optimized Laboratory

Determination of Polycyclic Aromatic Hydrocarbons in Soil

RayKol Group Corp., Ltd.

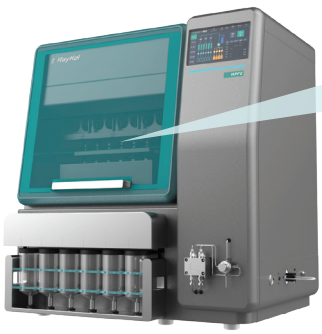
Introduction

This study refers to the method "HJ 805-2016 Determination of Polycyclic Aromatic Hydrocarbons in Soil and Sediment by Gas Chromatography-Mass Spectrometry" and the technical regulations in soil detailed surveys to establish a method for detecting polycyclic aromatic hydrocarbon residues in soil sediments using the Fotector Plus high-throughput fully automated solid-phase extraction instrument combined with GC-MS. After extraction with the HPFE high-throughput pressurized fluid extractor, the extract is concentrated and solvent-exchanged to 1 mL of n-hexane using the MPE fully automated vacuum parallel concentrator. The extract is then purified with the Fotector Plus high-throughput fully automated solid-phase extraction instrument, which automatically completes the steps of SPE column activation, sample loading, washing and collection. The collected solution is further concentrated, solvent-exchanged, and volume-adjusted using the Auto EVA 80 high-throughput fully automated parallel concentrator before being analyzed by gas chromatography-mass spectrometry.

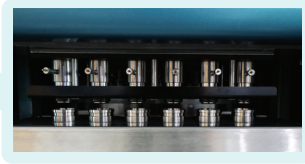
Instruments	Raykol Fotector Plus Automated Solid Phase Extraction System
	Raykol HPFE series Pressurized Fluid Extraction System
	Raykol MPE Plus Automated Vacumn Evaporation System
	Raykol Auto EVA 80 Automated Evaporation System
	GC-MS, Agilent 7890 A/5975C Gas Chromatography-Mass Spectrometer
Consumables	Solid Phase Extraction Column(RayCure Silica, 1g/6mL, RC-204-16845)
	Gas Chromatography Column: HP-5MS (30 m × 0.25 mm × 0.25 μm)
Reagents	n-Hexane, Acetone, Dichloromethane; Anhydrous sodium sulfate (analytical grade)

Sample Preparation

Extraction



HPFE series
Pressurized Fluid Extraction System



- Weigh 20 g of finely ground and sieved environmental soil sample, add an appropriate amount of diatomaceous earth, and mix uniformly. Load the mixture into a 34 mL extraction cell. Prepare six extraction cells in the same manner.

- The six simultaneously extracted samples are placed in the HPFE

  The extraction solvent is a mixture of dichloromethane and acetone (1:1 volume ratio). Set the system pressure to 10 MPa, the extraction temperature to 100°C, the preheating temperature to 100°C, the static extraction time to 5 minutes, the extraction purge time to 1 minute, the rinse volume to 60% of the extraction cell volume, and the rinse time to 20 seconds.

- Perform two extraction cycles, collect the extracts, and remove water using anhydrous sodium sulfate.

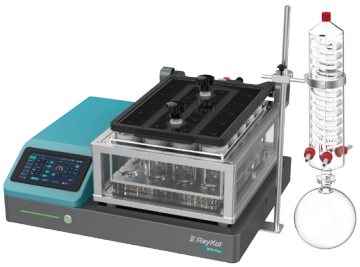
Pre-concentration

Concentrate the sample to 1 mL.Add 10 mL of n-hexane and continue concentrating until the volume is 1 mL.

Purification

Concentration

Use nitrogen blowdown with n-hexane for solvent exchange for GC-MS analysis.



MPE Plus
Automated Vacumn Evaporation System

Fotector Plus
Automated Solid Phase Extraction System

Auto EVA 80
Automated Evaporation System

Solid-Phase Extraction Purification Conditions

Automated SPE Instrument	Fotector Plus High-Throughput Fully Automated SPE Instrument
SPE Column	Silica Column (RayCure, 1g/6mL)
Activation	Acetone, n-Hexane
Sample Bottle Cleaning	n-Hexane
Elution	Acetone: n-Hexane (1:9)

GC-MS Conditions

Injection Port Temperature	260°C
Column Flow Rate	1 mL/min
Injection Volume	1 µL
Column Temperature	60°C for 1 min, then ramp up to 200°C at 10°C/min, hold for 2 min, then ramp up to 300°C at 5°C/min, hold for 2 min
Auxiliary Heating Temperature	280°C
Ion Source Temperature	230°C
Quadrupole Temperature	150°C
Mode	Full Scan
Solvent Delay	6 min

Results and Discussion

After sample pretreatment and extraction, purification was performed using the Fotector Plus. The overall recovery rate was calculated using a matrix spiked calibration curve. The overall average recovery rate ranged from 75.34% to 120.00%, with an RSD of less than 13% (n=4), meeting the standard requirements for recovery rate and complying with the allowable deviation requirements of the Technical Regulations for the Detection of Polycyclic Aromatic Hydrocarbons in Soil Surveys. The recovery rates and RSD are shown in Table 1:

Table 1 Recovery Rates and Relative Standard Deviations (RSD) for 16 PAHs (n=4)

Compound	Spiked Level (μg/kg)	Average Recovery Rate (%)	RSD (n=4)	Spiked Level (μg/kg)	Average Recovery Rate (%)	RSD (n=4)
Naphthalene	20	76.80	3.83	200	71.17	7.94
Acenaphthylene	20	75.87	7.12	200	71.88	2.88
Acenaphthene	20	72.34	7.47	200	80.45	0.46
Fluorene	20	85.15	7.47	200	79.77	5.75
Phenanthrene	20	99.91	4.45	200	104.09	1.41
Anthracene	20	87.70	7.00	200	91.65	4.34
Fluoranthene	20	90.95	4.72	200	104.16	6.50
Pyrene	20	89.05	12.74	200	94.32	1.05
Chrysene	20	100.77	8.39	200	115.39	11.20
Benz[a]anthracene	20	113.09	11.89	200	103.70	8.32
Benzo[b]fluoranthene	20	93.44	3.57	200	114.77	1.66
Benzo[k]fluoranthene	20	98.51	3.94	200	101.26	1.34
Benzo[a]pyrene	20	66.77	4.26	200	74.86	0.80
Dibenzo[a,h]anthracene	20	106.01	2.68	200	78.00	8.84
Benzo[ghi]perylene	20	88.07	4.73	200	76.00	1.17
Indeno[1,2,3-cd]pyrene	20	100.01	6.59	200	119.00	6.14

Summary

This method utilizes the Raykol HPFE series Pressurized Fluid Extraction Systemas an essential device for soil extraction. It can simultaneously extract six samples within 30 minutes. Considering an 8-hour workday, the daily throughput can reach up to 96 samples. The instrument is simple to operate, controlled via a touchscreen with one-click operation, making it accessible for new laboratory staff to use immediately. The extractor can utilize four different solvents, which can be mixed in various proportions without manual preparation.

The Raykol MPE Plus Automated Vacumn Evaporation System can concentrate 16 large-volume samples or 36 small-volume samples within half an hour, greatly improving the efficiency of sample preparation and truly freeing laboratory personnel from manual labor.

The Raykol Fotector Plus Automated Solid Phase Extraction System can handle six samples simultaneously and continuously process up to 60 samples. It automates various basic commands from activation, sample loading, washing to elution, significantly enhancing the precision and reliability of the instrument and ensuring high recovery rates.

The Raykol Auto EVA 80 Automated Evaporation System can automatically perform rapid parallel concentration of small-volume samples.

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