



Determination of Polychlorinated Biphenyls (PCBs) in Soil

■ Introduction

This study references the method "HJ 743-2015 Determination of Polychlorinated Biphenyls in Soil and Sediment by Gas Chromatography/Mass Spectrometry" to establish a method for detecting PCBs in sediments using the Fotector Plus High-Throughput Fully Automated Solid-Phase Extraction Instrument combined with GC-MS. In an acetone-n-hexane (1:1) system, the extraction is performed using the HPFE High-Throughput Pressurized Fluid Extractor. The extract is then concentrated to 1 mL using the MPE Vacuum Parallel Concentrator. Following this, the sample is purified using 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 EVA 80 High-Throughput Fully Automated Parallel Concentrator before being analyzed by GC-MS.

Instruments	Raykol Fotector Plus Automated Solid Phase Extraction System
	Raykol HPFE 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: Silica column (RayCure Silica, 1g/6mL, RC-204-16845)
	Solid Phase Extraction Column:Florisil Column (RayCure Florisil, 1g/6 mL, RC-204-16945)
	Solid-phase extraction column: Graphitized Carbon Black column (RayCure GCB, 1g/6 mL, RC-204-16945)
Reagents	n-Hexane, Acetone; Anhydrous sodium sulfate (analytical grade)

■ Sample Preparation

1 Extraction

Weigh 10 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. Place the six extracted simultaneously samples into the HPFE. Perform two extraction cycles, collect the extracts, and remove water using anhydrous sodium sulfate.



HPFE
Pressurized Fluid Extraction System



MPE Plus
Automated Vacuum Evaporation System

2 Pre-concentration

Concentrate the sample to 1 mL, then add 10 mL of n-hexane and continue concentrating to 1 mL.



Fotector Plus
Automated Solid Phase Extraction System

3 Purification

Solid-phase extraction column: Florisil column (RayCure, 1g/6mL)

Activation: n-Hexane

Clean sample bottle: Acetone: n-Hexane (1:9)

Elution: Acetone: n-Hexane (1:9)



Auto EVA 80
Automated Evaporation System

4 Concentration

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

Gas Chromatography-Mass Spectrometry Conditions

Column	HP-5MS 60 m × 0.25 mm × 0.25 µm
Injection Port Temperature	280°C
Column Flow Rate	1 mL/min
Injection Volume	1 µL
Initial Temperature	110°C, hold for 2 min
Temperature Ramp	6°C/min to 290°C, hold for 3 min
Split Outlet Purge	0.75 min, 60 mL/min
Carrier Gas Saving	2 min, 20 mL/min
MSD Transfer Line Auxiliary Heating	280°C
Ion Source Temperature	230°C
Quadrupole Temperature	150°C
Mode	SIM
EM Voltage	1654
Solvent Delay	4.5 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 77.67% to 106.01%, with an RSD of less than 7.2% (n=4), meeting the standard requirements for recovery rate and complying with the allowable deviation requirements of the HJ 743-2015 Determination of Polychlorinated Biphenyls in Soil and Sediment by Gas Chromatography/Mass Spectrometry. The recovery rates and RSD are shown in Table 4:

Table 1: Recovery Rates and Relative Standard Deviations (RSD) for Each Component (n=4)

Compound	Retention Time (min)	Spiked Level (µg/kg)	Average Recovery Rate (%)	RSD
2-Chlorobiphenyl	16.314	20	77.67	4.7
2,3-Dichlorobiphenyl	20.251	20	86.27	3.2
2,4,5-Trichlorobiphenyl	22.837	20	88.82	6.5
2,2',4,4'-Tetrachlorobiphenyl	24.726	20	93.41	3.3
2,2',3,4,6-Pentachlorobiphenyl	26.762	20	93.80	7.2
2,2',4,4',5,6-Hexachlorobiphenyl	28.380	20	101.43	5.3
2,2',3,3',4,4',6-Heptachlorobiphenyl	32.249	20	103.15	1.2
2,2',3,3',4,5,6,6'-Octachlorobiphenyl	32.134	20	106.01	3.9

Summary

This experiment demonstrates that the Raykol Fotector Plus Automated Solid Phase Extraction System effectively reproduces the method outlined in "HJ 743-2015 Determination of Polychlorinated Biphenyls in Soil and Sediment by Gas Chromatography/Mass Spectrometry." The recovery rates and RSDs meet the allowable deviation requirements of HJ 743-2015 and comply with the quality control requirements for analytical results specified in HJ 743-2015.

The Raykol HPFE series Pressurized Fluid Extraction System is an essential device for soil extraction, capable of simultaneously extracting 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 Vacuum Parallel Concentrator can concentrate 16 large-volume samples or 36 small-volume samples within half an hour, greatly improving the efficiency of sample preparation. The Fotector Plus High-Throughput Fully Automated Solid-Phase Extraction Instrument can handle six samples simultaneously and continuously process up to 60 samples, automating various basic commands from activation, sample loading, washing to elution. This significantly enhances the precision and reliability of the instrument, ensuring high recovery rates.

The Raykol Auto EVA 80 features nitrogen blowdown needles that automatically lower with the liquid level, maintaining the optimal concentration distance throughout the process. This enables rapid and parallel concentration, with a fully automated workflow that truly frees laboratory personnel from manual labor.



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