Determination of Semi-Volatile Organic Compounds in Soil

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Introduction

This solution refers to the method "HJ 834-2017 Determination of Semi-Volatile Organic Compounds in Soil and Sediment by Gas Chromatography-Mass Spectrometry". The extraction is performed using the HPFE series high-throughput pressurized fluid extractor in an acetone + dichloromethane (1+1) environment. The extract is then concentrated to 1 mL using the MPE series high-throughput vacuum parallel concentrator, followed by the addition of 5 mL of n-hexane for solute transfer. The sample is then purified in the Fotector Plus high-throughput fully automatic solid-phase extraction instrument, which automatically completes the SPE column activation, sample loading, elution, and collection steps. The collected liquid is further concentrated under nitrogen, undergoes solvent exchange and final volume adjustment, and is then analyzed by GC-MS.

Instruments	Raykol AP 300 Automated Liquid Handling Station
	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	Anhydrous Sodium Sulfate (Analytical Grade); Solid Phase Extraction Columns;
	Florisil Columns (RayCure Florisil, 1g/6 ml, RC-204-16945)
	Gas Chromatography Column: HP-5MS (60 m × 0.25 mm × 0.25 µm)
Reagents	n-Hexane, Acetone; Dichloromethane

Sample Preparation

Preparation of Standard Solutions

The AP 300 Fully Automated Liquid Sample Handling Workstation is used to automatically prepare standard solutions of semi-volatile organic compounds and surrogates with concentration gradients of 50 µg/L, 100 µg/L, 200 µg/L, 500 µg/L and 1000 µg/L, or 1 µg/mL, 2 µg/mL, 5 µg/mL, 10 µg/mL and 20 µg/mL. Internal standard solutions are added to these standard solutions to achieve a concentration of 200 µ g/L (or 20 µg/mL) in the calibration curve.

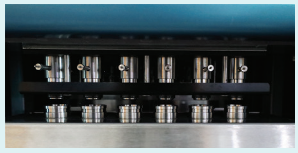
AP 300
Automated Liquid Handling Station

Extraction

- Weigh 20g of fresh environmental soil sample, add an appropriate amount of diatomaceous earth, and mix uniformly. Grind the mixture until it has a free-flowing sand-like texture. Load the mixture into a 34 mL extraction cell. Prepare six extraction cells in the same manner.

- Place the prepared extraction cells into the HPFE system

> 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.



HPFE series
Pressurized Fluid Extraction System

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

Concentration

Place the collection tubes into the MPE series high-throughput vacuum parallel concentrator. Set the concentration temperature to 40°C. The final solvent exchange should be done with n-hexane, and concentrate the sample to a volume of approximately 2 mL.

MPE Plus
Automated Vacumn Evaporation System

Purification

Cleaning	Clean the sample channel by washing the loading needle with a mixture of n-hexane and acetone (9:1).
Activation	Use Florisil columns (1g/6mL) for purification. Activate the columns with 5 mL of n-hexane and acetone (9:1) and 5 mL of n-hexane.
Loading	Load the sample at a flow rate of 0.5 mL/min, and collect the solution.
Washing	Rinse the sample bottle with n-hexane and collect the effluent.
Elution	Elute the column with 10 mL of n-hexane and acetone (9:1) at a flow rate of 1 mL/min.
Concentration	Concentrate the collected solution to 0.5 mL using the Auto EVA 80 high-throughput fully automated parallel concentrator. Add 20 μL of 10 μg/mL internal standard solution and use n-hexane to adjust the volume to 1 mL. The sample is then ready for GC-MS analysis.

Fotector Plus
Automated Solid Phase Extraction System

Auto EVA 80
Automated Evaporation System

Gas Chromatography-Mass Spectrometer Conditions

Column	30 m × 0.25 mm × 0.25 μm
Injection Port Temperature	280°C
Column Flow Rate	1 mL/min
Injection Volume	1 μL
Column Temperature	Start at 120°C for 2 min, then ramp up to 180°C at 12°C/min, hold for 5 min, then ramp up to 240°C at 7°C/min, and finally ramp up to 280°C at 5°C/min, hold for 2 min
MSD Transfer Line Auxiliary Heating	280°C
Ion Source Temperature	230°C
Quadrupole Temperature	150°C
Mode	SIM (Selected Ion Monitoring)
Scan Range	45 amu to 450 amu
Solvent Delay	5 min

Results and Discussion

Add 100 μL, 300 μL and 500 μL of 1 ppm (or 20 μL, 50 μL, and 100 μL of 100 ppm) semi-volatile organic compound standard solution to the soil samples, then perform the experiment. Add 20 μL (10 μg/mL) of internal standard solution, and finally use n-hexane to adjust the volume to 1 mL to determine the spiked recovery rate.

Table 1 Spiked Sample Recovery Rates

No.	Name	Recovery Rates (%)		No.	Name	Recovery Rates (%)	
		Avg.	RSD(%)			Avg.	RSD(%)
1	N-Nitrosodimethylamine	65	3	35	2,6-Dinitrotoluene	74	3
2	2-Fluorophenol (Substitute)	83	1	36	3-Nitroaniline	79	5
3	Phenol-d6 (Substitute)	91	2	37	2,4-Dinitrophenol	71	6
4	Phenol	87	1	38	Acenaphthene	76	3
5	Bis(2-chloroethyl) ether	93	3	39	Dibenzofuran	86	2
6	1,3-Dichlorobenzene	55	2	40	4-Nitrophenol	81	3
7	1,4-Dichlorobenzene	64	4	41	2,4-Dinitrotoluene	87	4
8	1,2-Dichlorobenzene	52	3	42	Fluorene	91	7
9	o-Cresol	82	2	43	Diethyl phthalate	110	5
10	Bis(2-chloroisopropyl) ether	79	3	44	4-Chlorophenyl phenyl ether	86	3
11	Hexachloroethane	66	3	45	4-Nitroaniline	78	6
12	N-Nitrosodi-n-propylamine	74	2	46	2,4-Dinitro-6-methylphenol	80	4
13	p-Cresol	85	2	47	Azobenzene	95	3
14	Nitrobenzene-d5 (Substitute)	81	4	48	2,4,6-Tribromophenol (TS)	90	2
15	Nitrobenzene	75	5	49	4-Bromodiphenyl ether	88	3
16	Isophorone	83	3	50	Hexachlorobenzene	84	2
17	2-Nitrophenol	65	4	51	Pentachlorophenol	76	4
18	2,4-Dimethylphenol	72	5	52	Phenanthrene	95	6
19	Bis(2-chloroethoxy)methane	83	3	53	Anthracene	91	8
20	2,4-Dichlorophenol	79	5	54	Carbazole	88	6
21	1,2,4-Trichlorobenzene	67	7	55	Di-n-butyl phthalate	105	4
22	Naphthalene	75	5	56	Fluoranthene	90	3
23	4-Chloroaniline	84	2	57	Pyrene	88	2
24	Hexachlorobutadiene	67	3	58	4,4'-Dibromodiphenyl ether	86	2
25	4-Chloro-3-methylphenol	78	4	59	Benzyl butyl phthalate	100	5
26	2-Methylnaphthalene	77	4	60	Benzo[a]anthracene	93	3
27	Hexachlorocyclopentadiene	70	3	61	Chrysene	88	5
28	2,4,6-Trichlorophenol	76	5	62	Di(2-ethylhexyl) phthalate	98	3
29	2,4,5-Trichlorophenol	81	4	63	Di-n-octyl phthalate	112	4
30	2-Fluorobiphenyl (Substitute)	85	2	64	Benzo[b]fluoranthene	90	3
31	2-Chloronaphthalene	79	3	65	Benzo[k]fluoranthene	93	5
32	2-Nitroaniline	75	3	66	Benzo[a]pyrene	95	3
33	Acenaphthylene	69	5	67	Indeno[1,2,3-cd]pyrene	92	2
34	Dimethyl phthalate	97	6	68	Dibenz[a,h]anthracene	90	4

Summary

The Raykol AP 300 Automated Liquid Handling Station enables the fully automated preparation of standard solutions, assisting laboratory personnel in easily preparing mixed standard calibration curves.

The Raykol HPFE series Pressurized Fluid Extraction System is an essential device for soil extraction, capable of extracting six samples simultaneously 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 Fotector Plus Automated Solid Phase Extraction System can handle six samples simultaneously and can continuously process up to 60 samples automatically. 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 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.

A series of automated sample preparation solutions significantly enhance work efficiency and free laboratory personnel from manual labor.

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