

GC-MS

APPLICATION NOTES

Determination of organochlorine pesticides in soil by gas chromatography-mass spectrometry



GC-MS



Introduction

An analytical method for the simultaneous detection of 23 organochlorine pesticides in soil by gas chromatography-mass spectrometry (GC-MS) was established with reference to the environmental standard HJ 835-2017. 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 with the retention time of the standard substance and the standard mass spectrogram and its 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.
Reagents	n-hexane, acetone (chromatographic pure), 23 kinds of organochlorine pesticides mixed standard

Preparation of standard solutions

Pipette 10 μL, 25 μL, 50 μL, 100 μL, 250 μL 1000 μg/mL organochlorine mixed standard solution in a 5 mL volumetric flask, with n-hexane: acetone (1:1), shaking well, that is, respectively, 2, 5, 10, 20, 50 μg/mL organochlorine pesticide standard use solution.

Instrument conditions

Gas chromatograph conditions

Column	Equity-5 (30 m × 0.25 mm × 0.25 μm) quartz capillary column
Carrier Gas	High purity helium
Flow Mode	Constant flow mode
Column Flow Rate	1 mL/min
Injection	Non-split injection
Inlet	250°C
Purge Flow Rate	3 mL/min
Column Box	120°C to keep 1 minute, 12°C/min to 180°C to keep 5 minutes, then 7°C/min to 240°C to keep 1 minute, then 1°C/min to 250°C to keep 2 minutes, then 10°C/min to 280°C to keep 1 minute.

Mass spectrometer conditions

Ion Source	El Source
Electron Energy	70 eV
Ion Source Temperature	230°C
Interface Temperature	270°C
Solvent Peak Time	6 minutes
Scanning Mode	Full scan
Scanning Mass Number Range	45 u – 450 u
Electron Multiplier High Voltage	1050 V, external standard method of quantification

Sample spectra

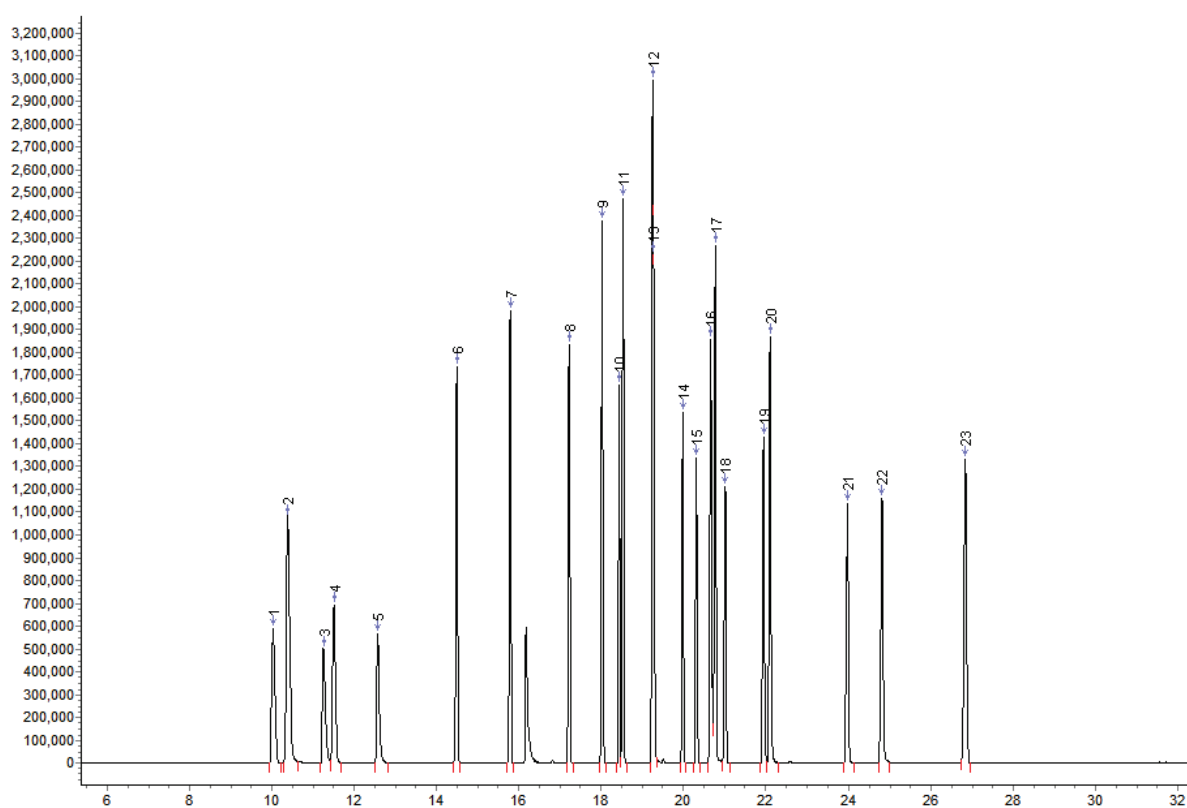


Figure 1: Organochlorine pesticide mixtures

No.	Description	CAS No.	Retention Time (min)	Quantitative Ion	Auxiliary Qualitative Ion
1	Alpha-hexahydroxy	319-84-6	10.05	219	181, 183
2	Hexachlorobenzene (HCB)	118-74-1	10.40	284	286, 282
3	Beta-hexahydroxylin	319-85-7	11.28	219	181, 183
4	Gamma-hexahydroxy	58-89-9	11.51	219	181, 183
5	Hexachlorocyclohexane-hexahydroxy	319-86-8	12.59	219	181, 183
6	Heptachlor	76-44-8	14.49	272	100, 274
7	Aldrin	309-00-2	15.80	263	66, 293
8	Heptachlor epoxide	1024-57-3	17.22	353	355, 351
9	Alpha-chlordane	5103-71-9	18.03	373	375, 377
10	α -Endosulfan	959-98-8	18.44	241	195, 339
11	Gamma-chlordane	5103-74-2	18.53	373	375, 377
12	p,p'-DDE	72-55-9	19.25	246	248, 318
13	Dieldrin	60-57-1	19.29	79	263, 279
14	Endrin	72-20-8	19.99	263	317
15	β -Endosulfan	33213-65-9	20.32	241	339, 337
16	p,p'-DDD	72-54-8	20.66	235	237, 165
17	o,p'-DDT	789-02-6	20.78	235	237, 165
18	Endrin aldehyde	7421-93-4	21.02	67	250, 345
19	Endosulfan sulfate	1031-07-8	21.94	272	387, 422
20	p,p'-DDT	50-29-3	22.10	235	237, 165
21	Endrinone	53494-70-5	23.98	317	67, 345
22	Mono-methoxytrichloroethane	72-43-5	24.82	227	228, 274
23	Mirex	2385-85-5	26.85	272	274, 270

Table 1: Information on 23 organochlorine pesticides

Standard curves

Pipette gun accurately pipette a certain amount of organochlorine mixed standard solution in a volumetric flask, n-hexane: acetone (1:1), shaking well, prepared into the concentration of 2, 5, 10, 20, 50 $\mu\text{g/mL}$ of organochlorine pesticide standard solution, respectively, take 1 μL on the GC-MS analysis, the concentration of the cross-coordinate, the corresponding peak area of the vertical coordinate to plot the standard curve.

No.	Description	Retention Time	Curvilinear Equation	Correlation Coefficient	RSD %
1	Alpha-hexahydroxy	10.05	$Y = 50455.71 X - 65624.33$	0.99977	2.5
2	Hexachlorobenzene (HCB)	10.40	$Y = 163878.3 X - 5723.22$	0.99916	2.89
3	Beta-hexahydroxylin	11.28	$Y = 36161.84 X - 53410.28$	0.99985	4.59
4	Gamma-hexahydro	11.51	$Y = 40754.17 X - 29328.24$	0.99923	3.97
5	Hexachlorocyclohexane-hexahydroxy	12.59	$Y = 37610.94 X - 71680.94$	0.99969	3.14
6	Heptachlor	14.49	$Y = 70013.42 X - 93570.51$	0.99950	3.45
7	Aldrin	15.80	$Y = 50735.59 X - 13486.93$	0.99983	2.04
8	Heptachlor epoxide	17.22	$Y = 50694.72 X - 43774.29$	0.99892	2.48
9	Alpha-chlordane	18.03	$Y = 86384.01 X - 6929.69$	0.99986	1.59
10	Alpha-endosulfan	18.44	$Y = 16138.85 X - 21269.62$	0.99927	4.63
11	Gamma-chlordane	18.53	$Y = 75381.44 X + 10071.36$	0.99963	2.38
12	p,p'-DDE	19.25	$Y = 141475.2 X - 80309.55$	0.99887	5.60
13	Dieldrin	19.29	$Y = 51743.79 X - 43146.33$	0.99899	6.72
14	Endrin	19.99	$Y = 15888.51 X - 22695.41$	0.99964	5.24
15	β -Endosulfan	20.32	$Y = 13499.08 X - 22153.99$	0.99985	4.48
16	p,p'-DDD	20.66	$Y = 190746.8 X - 420785.80$	0.99921	3.92
17	o,p'-DDT	20.78	$Y = 188747.1 X - 110064.80$	0.99992	3.73
18	Endrin aldehyde	21.02	$Y = 29233.3 X - 64061.69$	0.99961	5.16
19	Endosulfan sulfate	21.94	$Y = 39842.33 X - 77216.22$	0.99879	3.48
20	p,p'-DDT	22.10	$Y = 198635.8 X - 383066.40$	0.99944	3.70
21	Endrinone	23.98	$Y = 31344.47 X - 58880.45$	0.99978	4.52
22	Mono-methoxytrichloroethane	24.82	$Y = 305209.9 X - 971954.60$	0.99757	3.40
23	Mirex	26.85	$Y = 118641.4 X - 53491.42$	0.99992	3.34

Table 2: Linear equations and linear correlation coefficients for 23 organochlorine pesticides

Results and discussion

In this paper, an analytical method for the simultaneous detection of 23 organochlorine pesticides in soil by gas chromatography-mass spectrometry (GC-MS) was established with reference to the environmental standard HJ 835-2017. Equity-5 (30 m \times 0.25 mm \times 0.25 μ m) capillary column was used to separate the components, and the mass spectrometer was used for the detection, with good linearity and reproducibility, which can meet the requirements of the standard detection.