

GC-MS

APPLICATION NOTES

Solution GC-MS determination of polybrominated biphenyls and polybrominated diphenyl ethers in plastic plug samples preamble



GC-MS

Introduction

PBBs and PBDEs are brominated flame retardants (BFRs), which are commonly used industrial chemicals in printed circuit boards, plastics, coatings, wires and cables, and resin-based electronic components. Bioaccumulation, persistence and toxicity are the main environmental characteristics of PBBs and PBDEs, which tend to accumulate in living organisms as well as in human fat, and can cause acute or chronic toxicity to organs such as the brain, liver and kidneys, as well as to the nervous system, endocrine system and reproductive system of human beings, as well as affecting brain development, behaviour and learning ability.



Figure 1: Typical plastics for analysis

Polybrominated diphenyl ethers (PBDEs) and polybrominated biphenyls (PBBs), which are heavily used flame retardants in plastics, are harmful to the environment and human health. Determination of PBDEs and PBBs in plastics is important for method development. In this paper, an analytical method for the determination of mono- to decabromo PBBs and PBDEs in plastics was established by an Aludra gas chromatograph after optimizing the detection conditions according to international standards. The method has a wide linear range, a good linear relationship, a good method reproducibility and a high degree of accuracy, and it can be used as a reference for the related personnel.



Figure 2: Aludra gas chromatography mass spectrometer

Instrumentation

Aludra gas chromatography mass spectrometer.

Instrument conditions

Gas chromatograph conditions

Column	DB-5MS (15 m × 0.25 mm × 0.1 µm) capillary column
Mode	Constant Flow Mode
Carrier Gas	High Purity Helium
Injection Mode	Non-shunt Injection
Inlet Volume	1 µL
Inlet Port	300°C

Mass spectrometer conditions

Ion Source	EI Source
Electron Energy	70 eV
Interface temperature	320°C
Ion Source Temperature	250°C
Scanning Mode	Full Scan
Electron Multiplier High Voltage	1400

Sample pre-treatment

The sample was cut into small pieces with a paper cutter, weighed accurately with an analytical balance to take 0.1 g of the sample in a 40 mL brown sample bottle, and accurately pipetted 10 mL of toluene into the sample bottle, screwed the cap on tightly, and placed in an ultrasonic instrument to ultrasonic treatment for 60 minutes, and then placed in a refrigerator for overnight immersion, and then placed in a vortex mixer to vortex mix for 1 minute, and then ultrasonic extraction for 60 minutes. The extract was then placed in a vortex mixer for 1 minute, and then ultrasonicated for 60 minutes. At the end of ultrasonication, the extract was placed in a vortex mixer for 60 minutes, and then allowed to stand until room temperature. 1 mL of the extract was pipetted accurately into a 10 mL brown volumetric flask, and then the extract was calibrated with iso-octane and shaken well to prepare for the extraction; 1 µL of the supernatant was extracted and analyzed by GC-MS.

Experimental results

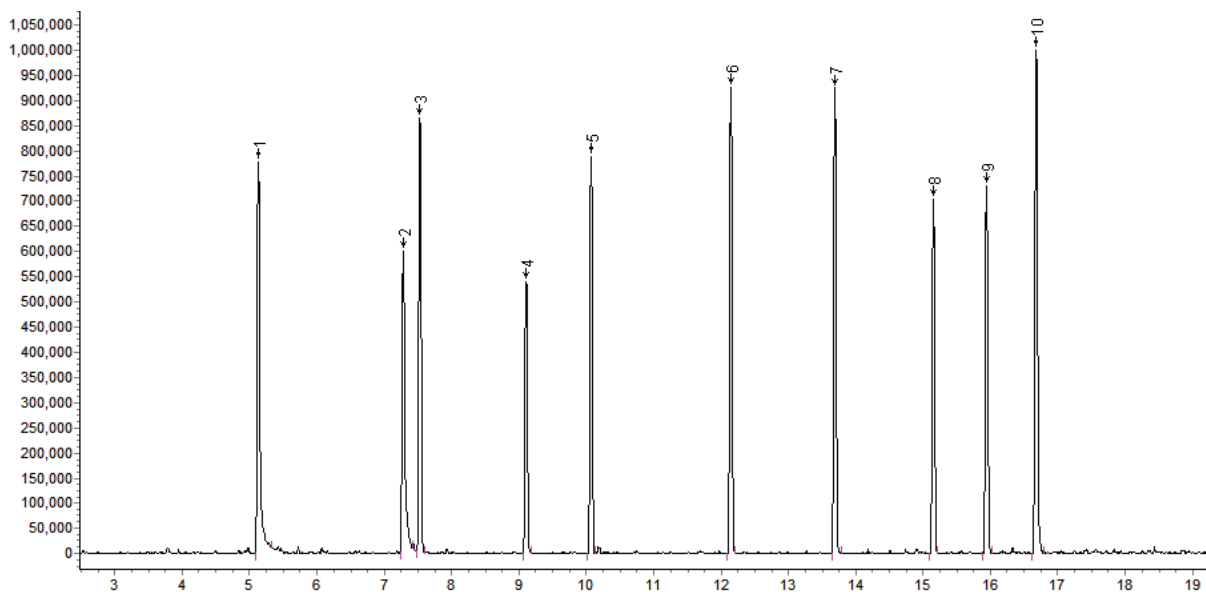


Figure 3: Total ion flow chromatogram of polybrominated biphenyls (PBBs)

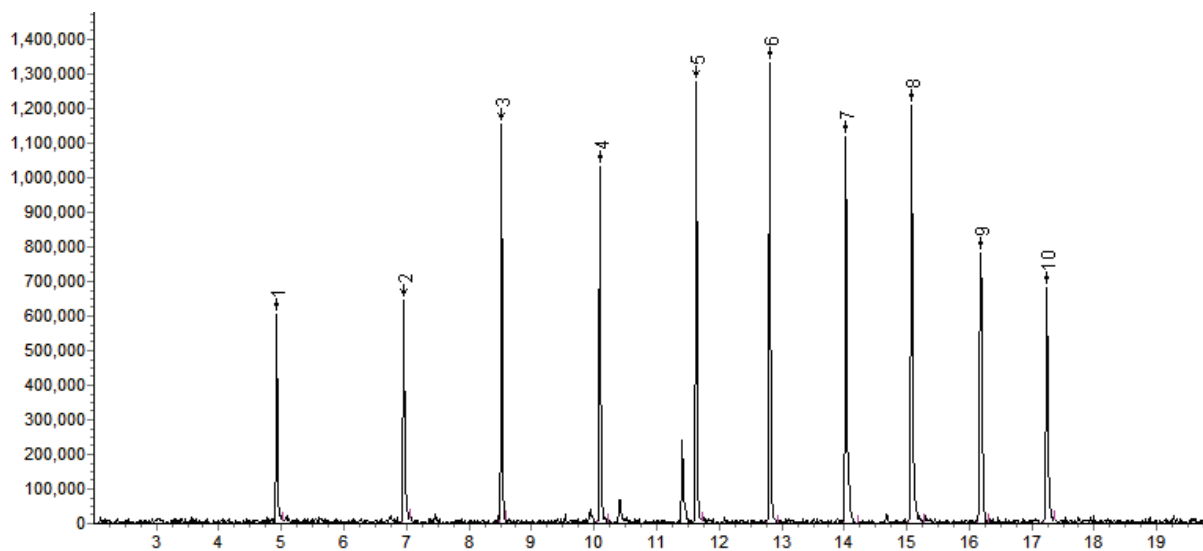


Figure 4: Total ion flow chromatogram of PBDEs

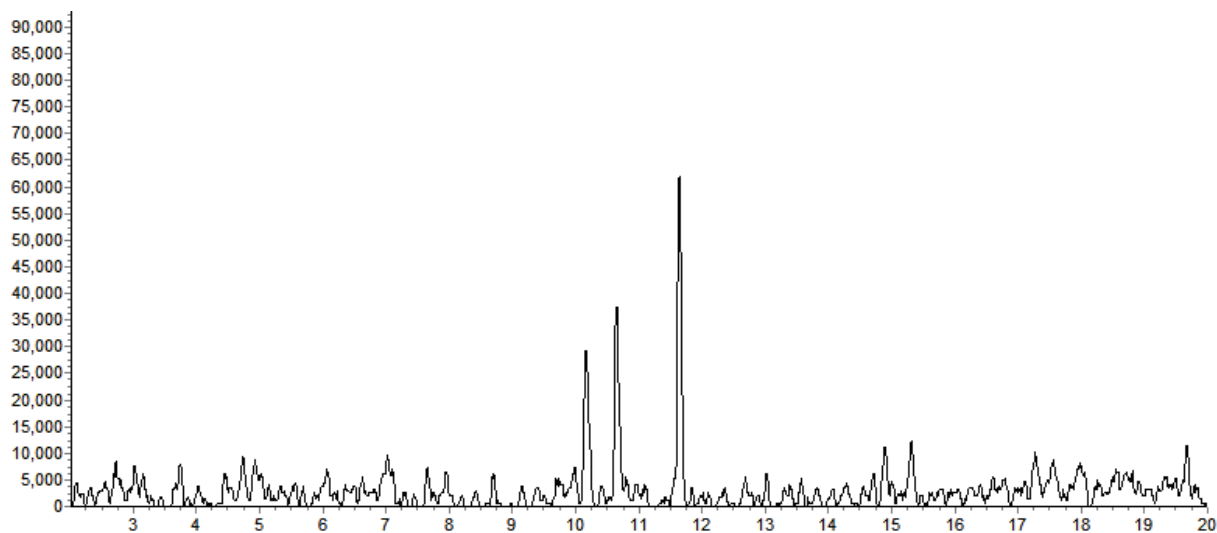


Figure 5: Total ion flow chromatogram of the sample

Sample measurement results

Item	Description	CAS.NO	Sample Content mg/kg
1	4- bromobiphenyl	92-66-0	ND
2	4,4'-Dibromobiphenyl	92-86-4	ND
3	2,2',5- tribromobiphenyl	59080-34-1	ND
4	2,2',5,5'- tetrabromobiphenyl	59080-37-4	ND
5	2,2',4,5',6- pentabromobiphenyl	59080-39-6	ND
6	2,2',4,4',5,5'- hexabromobiphenyl	59080-40-9	ND
7	2,2',3,4,4',5,5'- heptabromobiphenyl	67733-52-2	ND
8	2,2',3,3',4,4',5,5'- octabromobiphenyl	67889-00-3	ND
9	2,2',3,3',4,4',5,5',6- nonabromobiphenyl	69278-62-2	ND
10	decabromobiphenyl	13654-09-6	ND

Table 1: Test results for PBBs in samples

Note: ND indicates not detected

Item	Description	CAS.NO	Sample Content mg/kg
1	3-Bromodiphenyl ether	6876-00-2	ND
2	3,4'-Dibromodiphenyl ether	83694-71-7	ND
3	2,4,4'-Tribromodiphenyl ether	41318-75-6	ND
4	2,3',4,4'-Tetrabromodiphenyl ether	189084-61-5	ND
5	2,2',3,4,4'-Pentabromodiphenyl ether	182346-21-0	ND
6	2,2',3,4,4',5'-Hexabromodiphenyl ether	182677-30-1	ND
7	2,3,3',4,4',5,6-Heptabromodiphenyl ether	189084-68-2	ND
8	2,3,3',4,4',5,5',6-Octabromodiphenyl ether	446255-56-7	ND
9	2,2',3,3',4,4',5,5',6-Nonabromodiphenyl ether	63387-28-0	ND
10	Decabromodiphenyl ether	1163-19-5	256

Table 2: Test results for PBDEs in samples

Note: ND indicates not detected

Conclusion

A method for the determination of polybrominated biphenyls (PBBs) and polybrominated diphenyl ethers (PBDEs) was established by using an Aludra gas chromatograph, which has good separation, wide linear range, high sensitivity and accuracy, and can be used as a reference for the personnel of quality control and testing industries in the related industries.