

Application Data Sheet

No.47

GC-MS

Gas Chromatograph - Mass Spectrometer

Analysis of Brominated Flame Retardants and Phthalate Esters In Polymers Under the Same Conditions Using a Pyrolysis GC-MS System (1) – PBBs and PBDEs –

In recent years, an analysis method is required to determine not only polybrominated biphenyls (PBBs) and polybrominated diphenyl ethers (PBDEs), which are regulated under the RoHS Directive, but also phthalate esters and other brominated flame retardants not governed by the directive (such as tetrabromobisphenol A, hexabromocyclododecane, and bis(pentabromophenyl)ethane). The method was developed to analyze those compounds under the same analytical conditions using EGA/PY-3030D Multi-Shot Pyrolyzer and GCMS-QP2020 Ultra systems. This Application Data Sheet shows the results from analyzing the brominated flame retardants PBB and PBDE using the developed method.

Analytical Conditions

Polyethylene and polypropylene samples containing brominated flame retardants (ERM®-EC590 and ERM®-EC591, respectively) were used as evaluating samples. 0.5 mg of shavings from each sample was measured. FASST (Fast Automated Scan/SIM Type), which is capable of simultaneous Scan and SIM measurements, was used as the measurement mode. Table 1 shows the analysis conditions and Fig. 1 shows the SIM measurement program.

Table 1: Analytical Conditions

Pyrolysis Instrument	:EGA/PY-3030D Multi-Shot Pyrolyzer		
GC-MS	:GCMS-QP2010 Ultra		
Column	:Ultra ALLOY-PBDE [15 m length, 0.25 mm I.D., df = 0.05 µm]		
[Pyrolyzer]			
Pyrolysis Furnace Temp.	:200 °C → (20 °C/min) → 300 °C → (5 °C/min) → 340 °C (1 min)		
Interface Temp.	:Manual (300 °C)		
[GC]		[MS]	
Injection Temp.	: 320 °C	Interface Temp.	:320 °C
Column Oven Temp.	:80 °C → (20 °C/min) →300 °C (5 min)	Ion Source Temp.	:230 °C
Injection Mode	:Split	Solvent Cut Time	:0.5 min
Carrier Gas	:Helium	Tuning Mode	:Normal
Flow Control Mode	:Constant linear velocity (52.1 cm/sec)	Measurement Mode	:FASST (simultaneous Scan/SIM measurements)
Purge Flow Rate	:3.0 mL/min	Scan Mass Range	:m/z 50 - 1000
Split Ratio	:50	Scan Event Time	:0.15 sec
		Scan Speed	:10,000 u/sec
		SIM Monitoring m/z:	See Fig. 2.
		SIM Event Time	:0.3 sec
		SIM Micro-Scan Width	:0.5 u

1 min	Group 1 (No. of m/z channels: 21)	10 min	Group 2 (No. of m/z channels: 11)	16 min
	Tetra-BDE (<i>m/z</i> 325.9, 483.7) Penta-BDE (<i>m/z</i> 403.8, 563.6) Hexa-BDE (<i>m/z</i> 483.7, 641.5) Hepta-BDE (<i>m/z</i> 563.6, 721.4) Tetrabromobisphenol A [TBBPA] (<i>m/z</i> 528.7, 543.7) Hexabromocyclododecane [HBCDD] (<i>m/z</i> 319.1, 560.6) Diisobutyl phthalate [DIBP] (<i>m/z</i> 149.0, 205.1, 223.1) Di- <i>n</i> -butyl phthalate [DIBP] (<i>m/z</i> 149.0, 205.1, 223.1) Benzylbutyl phthalate [BBP] (<i>m/z</i> 91.0, 149.0, 206.1) Bis(2-ethylhexyl) phthalate [DEHP] (<i>m/z</i> 149.0, 167.0, 279.1) Di- <i>n</i> -octyl phthalate [DOP] (<i>m/z</i> 149.0, 261.1, 279.1) Di-isononyl phthalate [DINP] (<i>m/z</i> 149.0, 167.0, 293.1) Di-isodecyl phthalate [DIDP] (<i>m/z</i> 149.0, 167.0, 307.1)		Hexa-BDE (<i>m/z</i> 483.7, 641.5) Hepta-BDE (<i>m/z</i> 563.6, 721.4) Octa-BDE (<i>m/z</i> 641.5, 801.3) Nona-BDE (<i>m/z</i> 719.4, 721.4) Deca-BDE (<i>m/z</i> 799.3, 801.3) Deca-BB (<i>m/z</i> 941.3, 943.3) Bis(pentabromophenyl)ethane (<i>m/z</i> 484.5, 969.2)	

Fig. 1: SIM Measurement Program

Results

The total ion current chromatogram and SIM mass chromatogram for the polyethylene containing BDEs (ERM®-EC590) are shown in Fig. 2. The total ion current chromatogram and SIM mass chromatogram for the polypropylene containing BDEs and BB (ERM®-EC591) are shown in Fig. 3.

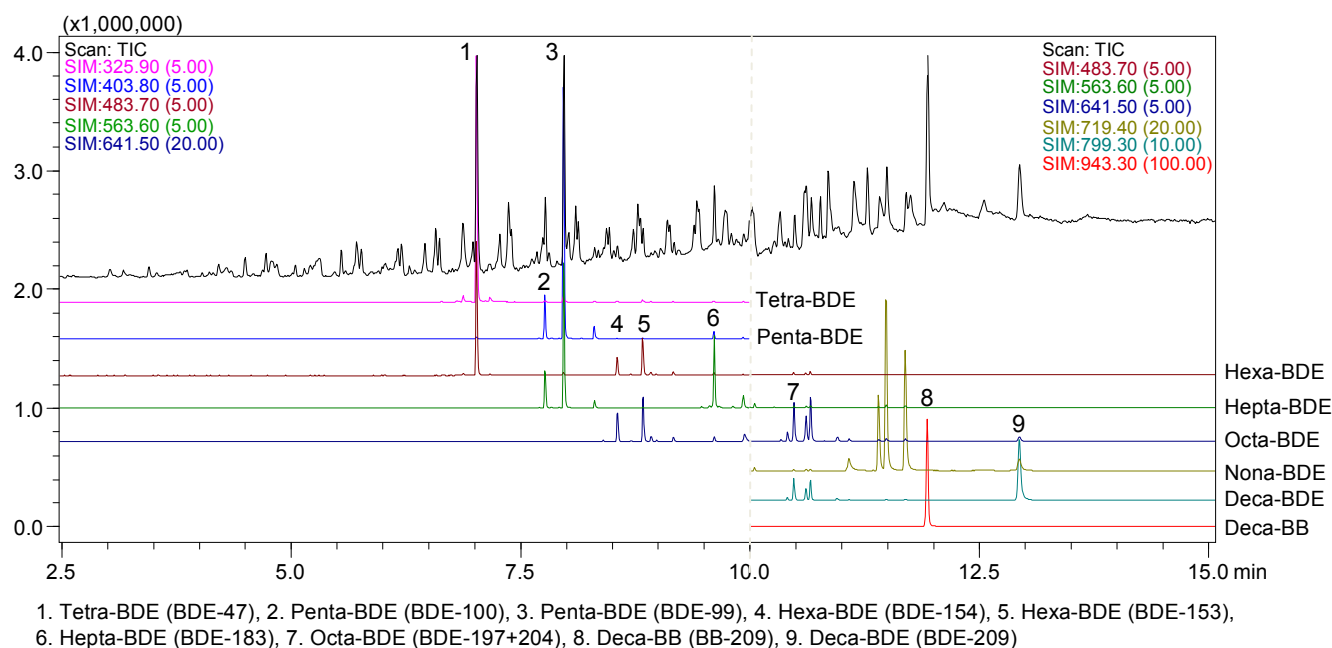


Fig. 2: Total Ion Current Chromatogram of Polyethylene Containing BDEs (ERM®-EC590)

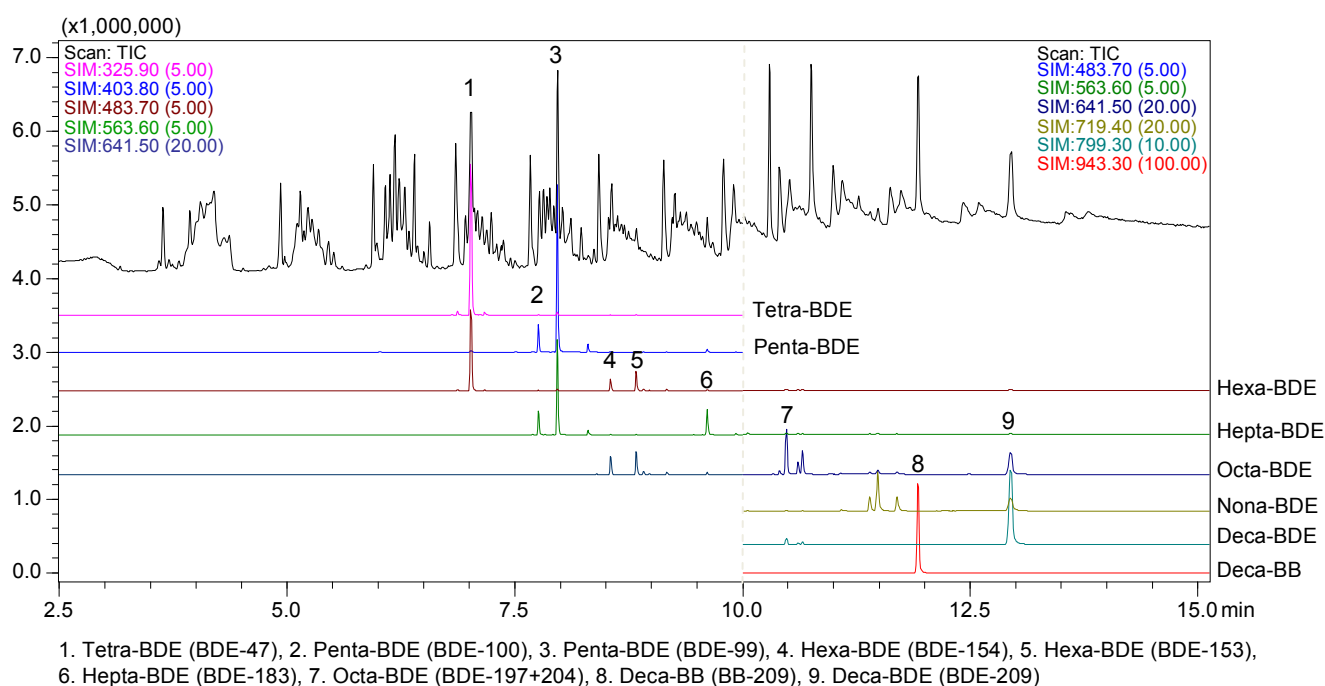


Fig. 3: Total Ion Current Chromatogram of Polypropylene Containing BDEs and BB (ERM®-EC591)