

Analysis of Brominated Flame Retardants by GC/MS

In July of 2006, the RoHS (Restriction on Hazardous Substances) directive will come into effect in the EU (European Union), specifying that certain hazardous substance shall not present in electrical and electronic equipment. These substances include lead, mercury, cadmium, hexavalent chromium and some brominated flame retardants (polybrominated biphenyls : PBBs and polybrominated diphenyl ethers ; PBDEs). Introduced here is an example of analysis of brominated flame retardants in plastic using GC/MS.

■ Analysis in EI Scan Mode

Fig. 1 shows total ion chromatogram (TIC) of PBDEs. Fig. 2 shows mass spectra of Penta-, Octa- and Deca-BDEs, substances to be restricted. These compounds produce typical strong fragment peaks of M-2Br.

Table 1 Analytical Conditions

GCMS-QP2010

-GC-	
Column	: UA1(HT) 15m × 0.25mm I.D. df=0.1μm
Col.Temp.	: 80°C(2min)-20°C/min-360°C(2min)
Inj. Temp.	: 300°C
He	: 150kPa(1.5min)/Splitless
	: 49.4cm/sec, Constant Linear Velocity Mode
Inj. Volume	: 1μL
-MS-	
I.F.Temp.	: 350°C
I.S.Temp	: 250°C
Scan Range	: 50-1000/0.6sec
PBDEs	: 5ppm in iso-Octane

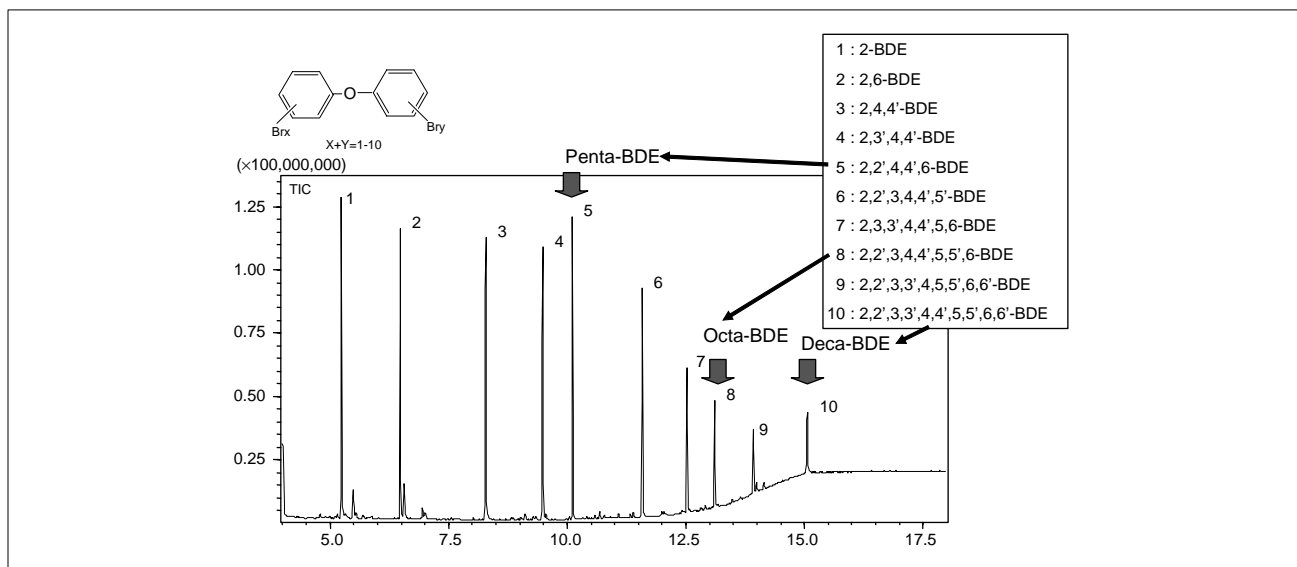


Fig. 1 TIC of Standard Sample (5ng)

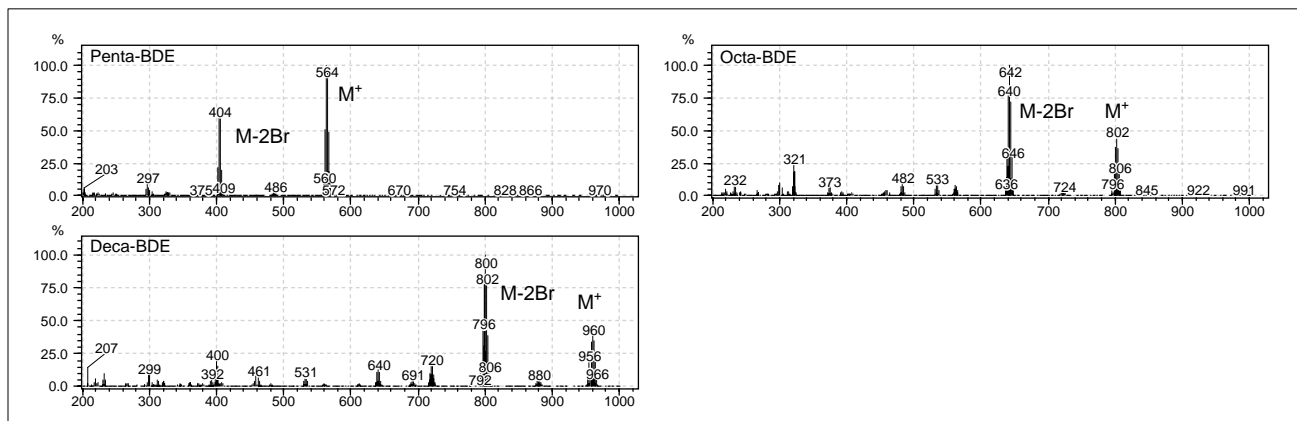


Fig. 2 Mass Spectrum of PBDEs

■ Analysis of PBDEs in Polystyrene Sample (1)

A polystyrene (PS) plastic sample containing 5% of Deca-BDE was dissolved in THF for analysis. The result is shown in Fig. 3. Nona-BDE is included as an impurity in the Deca-BDE.

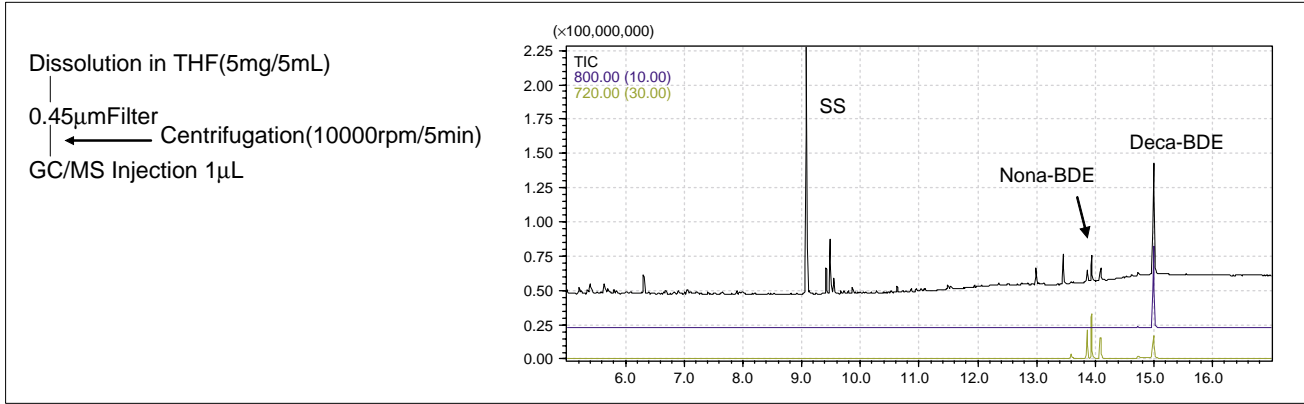


Fig.3 Mass Chromatogram of PS Extract

■ Analysis of PBDEs in Polystyrene Sample (2)

Fig. 4 shows the result of analysis of PBDEs that were not identified by FTIR. They were not detected in Scan mode, but clearly detected in SIM mode.

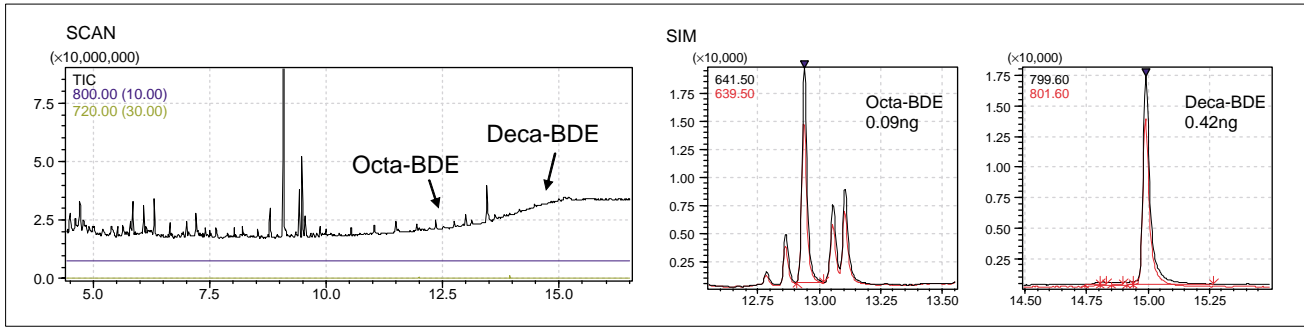


Fig. 4 Mass Chromatogram and SIM Chromatograms of PS Extract

■ Deca-BDE Calibration Curve

Figs. 5 and 6 show the SIM data and calibration curve for the Deca-BDE 799.3 (M-2Br) ion.

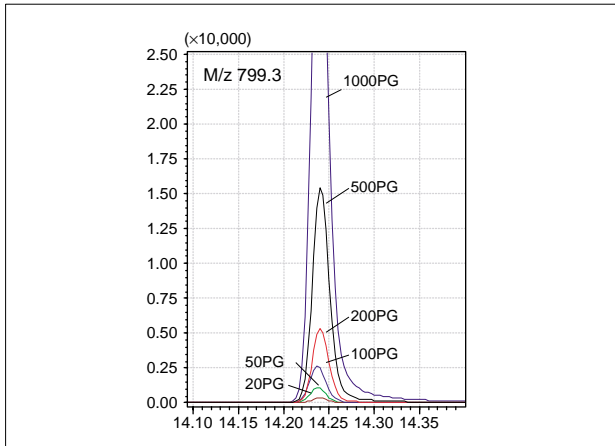


Fig. 5 SIM Chromatogram of Deca-BDE

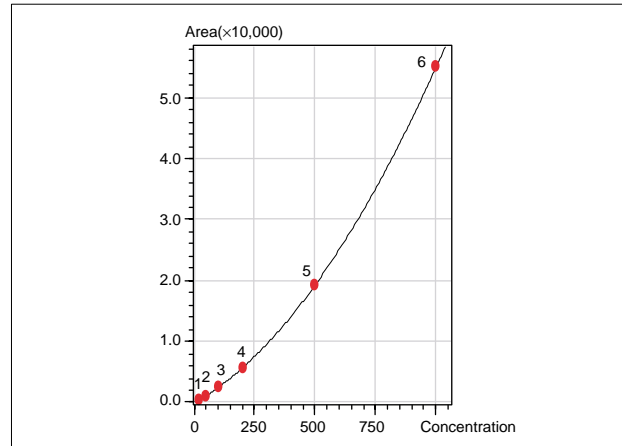


Fig. 6 Deca-BDE Calibration Curve (20 - 1000pg)