Analysis of Brominated Flame Retardant in a Waste Plastics by Thermal Desorption-GC/MS Technique

[Background] In the analysis of brominated flame retardants, to be controlled under the Restriction of Hazardous Substances (RoHS directive), GC analysis is generally conducted after solvent extraction, although, it involves cumbersome operations. Therefore, simpler analytical technique needed to be developed. Here, the analysis of decabromodiphenyl ether (DeBDE), a most commonly used brominated flame retardant is achieved by thermal desorption (TD)-GC/MS technique.

[Experimental] A TD-GC system in which a Double-Shot Pyrolyzer® was directly attached to the split/splitless injection port of a GC was used. A polystyrene (PS) based waste plastic containing brominated flame retardants was used as a sample, and an aliquot (5 µL) of THF solution (10 µg/µL) was placed in a sample cup for analysis. The temperature of the PY-GC interface and the GC injection port was set to 320ºC, at which temperature no absorption or thermal decomposition of DeBDE has been reported.[1] A metal capillary column specifically designed for brominated flame retardants [2] [Ultra ALLOY-PBDE] with highly deactivated inner wall was used.

[Result] Fig. 1 shows evolved gas analysis (EGA) curves of waste plastic, which was obtained in order to find optimum thermal desorption conditions. The major peak between 400 and 500ºC proved to be derived from thermal decomposition of the base polymer PS. Also a small peak observed between 250 and 350ºC showed m/z 799 and m/z 959 (molecular ion) on the average mass spectrum, hence it was considered to be due to thermally desorbed DeBDE. From this result, the optimal thermal desorption temperature for DeBDE was determined to be 200~400ºC (20 ºC/min). With this thermal desorption condition, the quantitative analysis of DeBDE in the waste plastic was performed by the TD-GC/MS technique. Fig 2. shows a chromatogram obtained, giving well-resolved peaks without interferences from coexisting species. By this method, it was confirmed that the waste plastic contained 7.1 wt% of DeBDE, with a good reproducibility (RSD=3.5%).

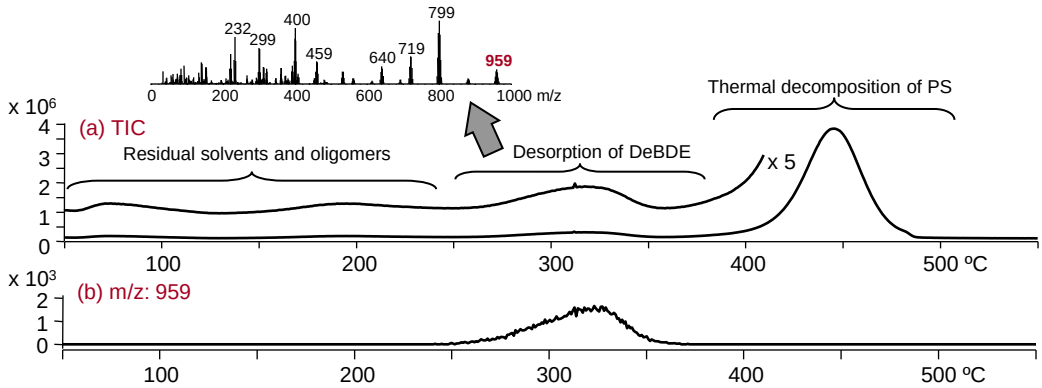

Figure 1 EGA curve of waste plastic

Pyrolysis furnace temp.: 50-550ºC (20ºC/min), GC oven temp.: 300ºC,column flow rate: 1 mL/min, split ratio: 1/50, GC/MS ITF temp.: 320ºC
MS ion source temp.: 250ºC, scan range: 29-1000 (m/z), scan rate: 0.2 scan/sec, sample size: 50µg

	Peak area of DeBDE ($x\ 10^6$, m/z: 799)
1	17.05
2	18.15
3	18.78
4	19.07
5	18.30
Average	18.27
RSD (n=5)	3.5 %

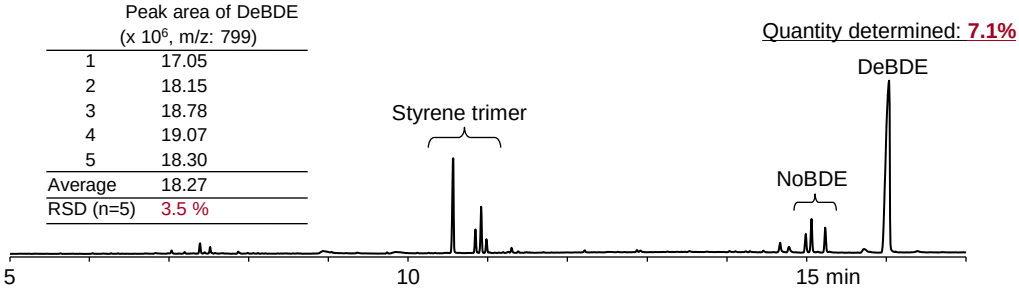

Figure 2 Chromatogram of waste plastic obtained by TD-GC/MS

Pyrolysis furnace temp.: 200-400ºC (20ºC/min), Separation column: UA-PBDE (polydimethylsiloxane, length 15m, id 0.25mm, film thickness 0.05 µm), Sample size: 50 µg
Column flow rate: 1mL/min, Split ratio: 1/50, GC/MS ITF temp.: 320ºC, MS ion source temp.: 230ºC, Scan range: 29-1000 (m/z), Scan rate: 3 scans/sec

Keyword : Brominated flame retardant, RoHS directive, thermal desorption, decabromodiphenyl ether

Applications : Electric and electronics industry, environmental analysis, general polymer analysis

Related technical notes : 1) PYA1-051E, 2) UAT-006E

Contents excerpted from A. Hosaka, C. Watanabe, S. Tsuge, *Anal. Sci.*, 2005, 21, 1145

R&D and manufactured by :
Frontier Laboratories Ltd.
1-8-14 Saikon, Koriyama
Fukushima-ken 963-8862 JAPAN
Phone: (81)24-935-5100 Fax: (81)24-935-5102
www.frontier-lab.com

SHIMADZU
Excellence in Science

® : Registered trademark of Frontier Laboratories Ltd.