

Analysis of brominated flame retardants in a waste plastic using thermal desorption (TD)-GC/MS - Part 2 Determination of TBBPA

[Background] In the recycling process of polymers, it is important to monitor the levels of flame retardants that are restricted by RoHS directive (2011/65/EU). As reported in a previous note (PYA1-095E), the TD-GC/MS analysis of a waste plastic foam revealed the existence of tetrabromobisphenol A (TBBPA, Fig. 1), a bromine-containing flame retardant. This note describes the quantitation of TBBPA in the waste plastic foam.

[Experimental] A pyrolysis-GC/MS system with a Multi-Shot Pyrolyzer (EGA/PY-3030D) directly interfaced to the GC injector was used. The standard addition method was used to determine the concentration of TBBPA in the waste plastic foam. Approximately 25 mg of the sample was dissolved in 1 mL of dichloromethane/xylene (90/10), and 20 µL of this solution was put in a sample cup, followed by solvent evaporation to form a thin film (ca. 0.5 mg) on the inner-surface of the sample cup. About 0.01 mg of TBBPA was dissolved in 1 mL methanol, which was used as a standard sample solution. 10 and 20 µL of this solution were separately added to the sample cups containing the thin sample film. After evaporating methanol, each sample spiked with TBBPA was analyzed by TD-GC/MS to make a calibration curve.

[Results] Fig. 2 shows the TD chromatograms of the waste plastic foam measured by the scan mode. As expected, peaks of styrene oligomers (S, SS, and SSS) are observed, while TBBPA is observed at 18.8 min and its existence is also confirmed by extracted ion chromatograms (m/z 529 and 544). Because the peak intensity of TBBPA is very weak in the TIC chromatogram, determination of TBBPA was done by the selected ion monitoring (SIM) mode and the three SIM TD chromatograms are shown in Fig. 3. From the calibration curve made in the SIM mode where abundances of ions (m/z 529 and 544) are counted, concentration of TBBPA was determined as 88 ppm. The relative standard deviation was 2.4% ($n=3$). As shown above, TD-GC/MS can be successfully used to determine the concentration of brominated fire retardants contained in waste plastic construction foam materials.

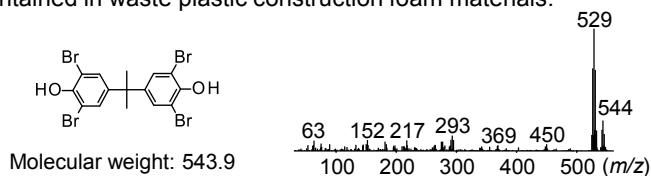


Fig. 1 Structure of TBBPA and its mass spectrum

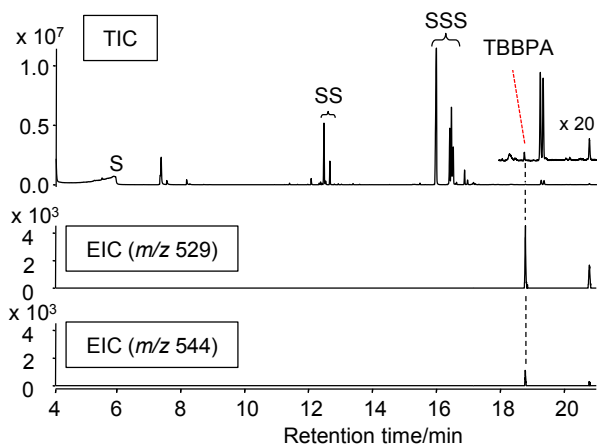


Fig. 2 TD chromatograms of sample obtained in the scan mode

Py furnace temp.: 200 – 300 °C (20 °C/min) – 340 °C (1 min hold, 5 °C/min),
 Separation column: UA⁺-5 (5 % diphenyl 95 % dimethylpolysiloxane, L=30 m, i.d.=0.25 mm, df=0.25 µm), Column flow rate: 1 mL/min (He), Split ratio: 1/20, GC oven temp.: 40 (2 min) – 320 °C (10 min hold, 20 °C/min), Sample amount: ca. 0.5 mg, Scan range: m/z 29 – 600, Scan rate: 5 scans/s. S, SS, and SSS represent monomer, dimer, and trimer of styrene, respectively.

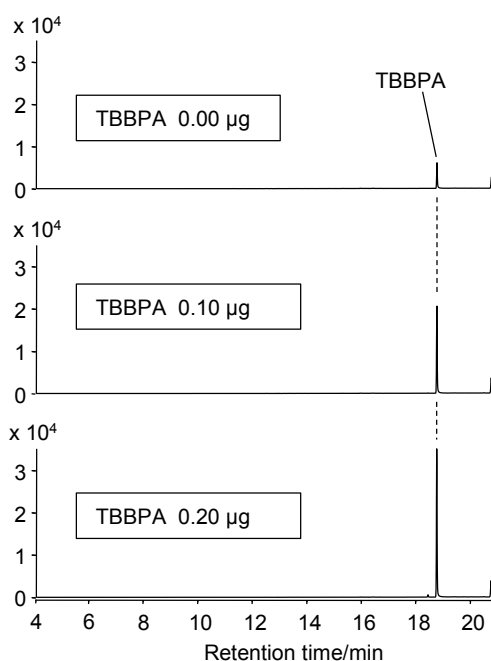


Fig. 3 TD chromatograms of sample obtained in the SIM mode (Standard addition method)

SIM ion; m/z 529 + 544, Dwell time: 50 msec, the other experimental conditions are the same as those listed in Fig. 2.

Keywords : Recycled polymer, Thermal desorption-GC/MS, Polystyrene, Brominated flame retardant, Standard addition

Products used : Multi-functional pyrolyzer, Auto-Shot Sampler, MicroJet Cryo-Trap, Vent-free GC/MS adapter, UA⁺-5, Eco-Cup LF

Applications : General polymer analysis, Quality assurance

Related technical notes : [PYA1-095E](#), [PYA1-052E](#)

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