

Simple quantitative analysis of a brominated flame retardant in polystyrene by thermal desorption GC/MS < study using an AIST RoHS certified standard >

[Background] In the quantitative analysis of brominated flame retardants which are regulated by RoHS directive for specific toxic materials used in electric and electronic devices, care must be taken to consider the thermally labile nature of brominated flame retardants. In this note, in order to verify the effectiveness of the simple qualitative analysis by TD-GC/MS, the content of a brominated flame retardant was determined using an RoHS certified standard material.

[Experimental] An AIST RoHS certified polystyrene (PS) standard containing 317 ppm of decabromodiphenylether (DeBDE) and PS samples containing 200-1000 ppm of DeBDE were analyzed using TD-GC/MS. For each point on the calibration curve, 20 µL of each PS solution (25 µg/µL, xylene/dichloromethane 10:90) were placed in a sample cup and the solvent allowed to evaporate prior to the analysis.

[Results] The EGA thermogram of the certified standard and the extracted ion ($m/z=799$) chromatogram of DeBDE are shown in Fig. 1. DeBDE evolves from the sample between 250 and 400°C; however, in order to minimize system contamination from the PS pyrolyzates, 340°C was selected as the upper desorption temperature for DeBDE. Fig 2 shows the total ion chromatogram (TIC) of the AIST standard. The quantitation of DeBDE was based on the peak area of the DeBDE peak in the TIC. The concentration of the certified material was determined to be 347 ppm ($n=5$, 6.1 %RSD) which is within 10% of the actual value.

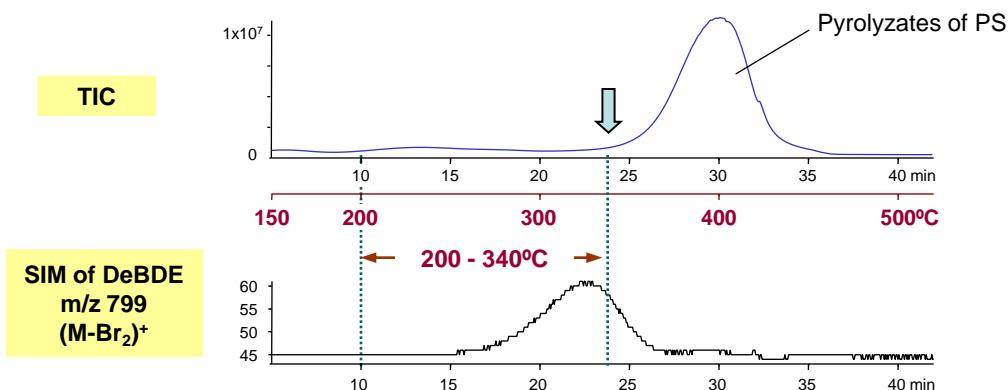


Fig. 1. EGA thermogram of an RoHS certified PS standard containing DeBDE and SIM of DeBDE (m/z 799)

Pyrolyzer furnace: 100-600 (10°C/min), GC oven: 300°C, Carrier gas flow rate: 1 mL/min, He, split ratio: 1/20, sample: 500 µg

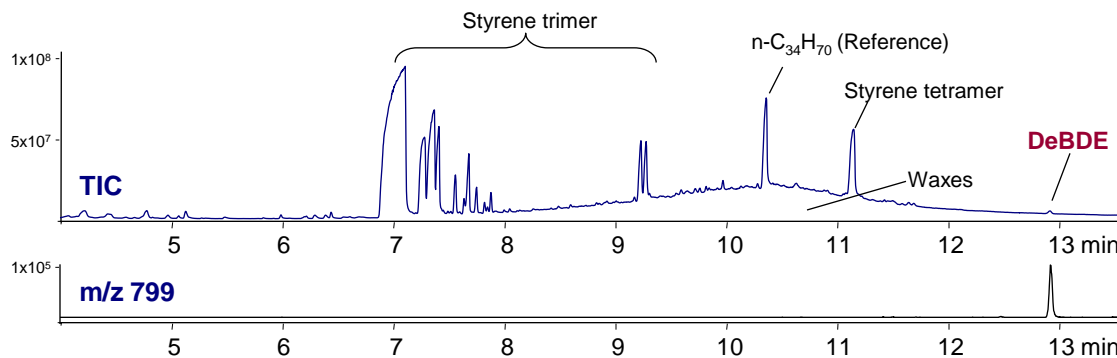


Fig. 2. Chromatogram of a certified material obtained by TD-GC/MS

Thermal desorption temp: 200 – 300 (40°C/min) - 340°C (5°C/min, 1 min), GC oven temp: 80 - 300°C (20°C/min), separation column: Ultra ALLOY-PBDE (dimethylpolysiloxane, L=15 m, i.d.=0.25 mm, df=0.05 µm), column flow rate: 1 mL/min, He, split ratio: 1/20, sample: 500 µg

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Keywords : Brominated flame retardant, RoHS directive, Thermal desorption, Decabromodiphenylether

Products used : Multi-functional pyrolyzer, Vent-free GC/MS adapter, UA-PBDE

Applications : Electric and electronic industries, environmental analysis, general polymer analysis

Related technical notes : PYA1-051E, PYA1-052E, UAT-006E

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