

Comparing glass and deactivated stainless steel sample cups - Part 1 Thermal desorption analysis of decabromodiphenylether (DeBDE)

[Background] The Multi-Shot Pyrolyzer (EGA/PY-3030D) is based on a vertical micro-furnace. The sample is placed in a cup which is dropped into the furnace. Such a design ensures that each sample experiences exactly the same thermal profile which is essential when high quality data is a necessity. The “standard” sample cup (Eco-cup) is made of deactivated stainless steel (SS). Therefore, it is difficult to visually examine the sample or residues residing in the SS cup. To address this problem, sample cups made of glass (Eco-cup G) have been developed so that the inside of the sample cup can be visually inspected. In this note, the performance of Eco-cup G and Eco-cup are compared using thermal desorption analysis (EGA-MS and TD-GC/MS) of DeBDE, a metal-susceptible brominated flame retardant.

[Experimental] Polystyrene (PS) which contained 317 ppm of DeBDE (AIST approved standard) was dissolved in dichloromethane/xylene (9:1). 20 µL of the solution (25 µg/µL) was placed in a sample cup and the solvent was evaporated. A GC/MS system with a Multi-Shot Pyrolyzer installed on the injection port of the GC was used for the analysis.

[Results] EGA thermogram of PS containing DeBDE is shown in Fig 1 inset and extracted ion chromatograms (EICs) for m/z 799, characteristic to DeBDE,¹⁾ are shown in Fig. 1. It is easily seen that the two EIC profiles obtained using Eco-cup G and Eco-cup are very similar. Typical TD-GC/MS results obtained from the thermal zone ranging from 200 to 340°C are shown in Fig. 2. Using the extracted ion profile of $m/z=799$, the peak at 12 min is identified as DeBDE. It should be noted that other peaks related to the debromination of DeBDE are not recognized. The reproducibility ($n=3$) of the DeBDE peak area is high and the relative standard deviation (RSD) values are 3.8% and 5.1% for Eco-cup G and Eco-cup, respectively (Table 1). These results show that both Eco-cup and Eco-cup G give comparable results and that both types of sample cups can be used for the determination of DeBDE.

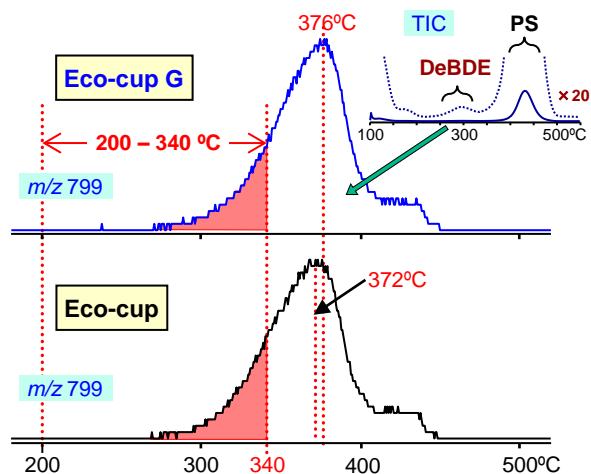


Fig. 1 EGA thermograms of DeBDE (317 ppm) in PS
Deactivated EGA tube ($L=2.5$ m, i.d.=0.15 mm)

Table 1 Peak area for m/z 799 and reproducibility in RSD ($n=3$)

	Peak area	RSD (%)
Eco-cup G	4,992	3.8
Eco-cup	5,517	5.1

1) T. Yuzawa, et al., *Anal. Sci.*, 24 (2008) 953-955

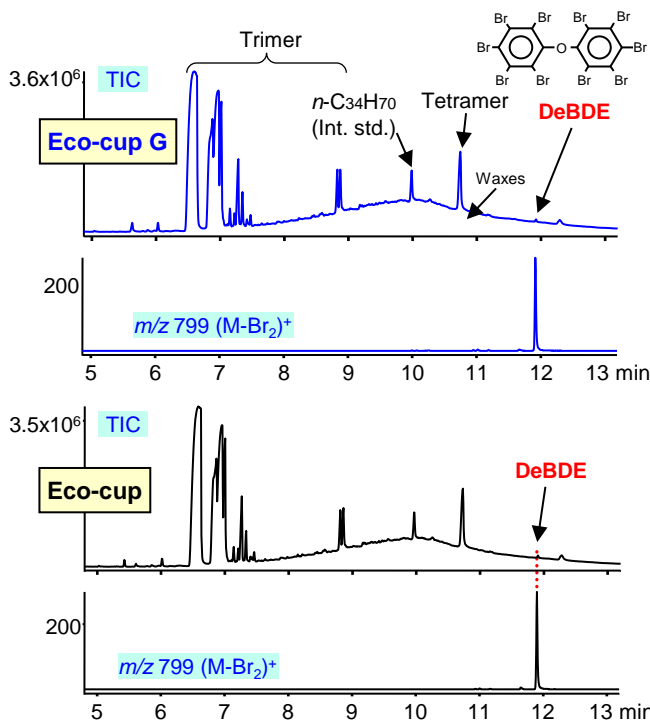


Fig. 2 Thermal desorption chromatogram of DeBDE in PS

Column: UA-PBDE ($L=15$ m, i.d.=0.25 mm, d.f.=0.05 µm)
TD: 200 - 340°C (20 °C/min), GC: 80 - 320°C (20 °C/min)

Keywords : Glass sample cup, Transparence, Decabromodiphenylether, DeBDE, Polystyrene, Thermal desorption analysis

Products used : Multi-functional pyrolyzer, UA-DeBDE, Vent-free GC/MS adapter, Eco-cup G

Applications : General polymer analysis, Thermal desorption analysis

Related technical notes : PYA1-071E, PYA1-072E, PYA1-073E, PYA1-075E, PYA1-076E, PYA1-084E

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