

Thermal desorption GC/MS of brominated flame retardant

- Optimization study of PY/GC interface and GC injector temperatures for EGA/PY-3030D -

[Background] With regard to the analysis of brominated flame retardant (eg. PBDE) using thermal desorption GC/MS, extended studies on the optimization of analytical conditions have been reported.^{1,2)} However, such studies have been done using the previous model Double-Shot Pyrolyzer (PY-2020iD). The Multi-Shot Pyrolyzer (EGA/PY-3030D) has been significantly improved in terms of the pyrolyzer-GC injector interface design; therefore, it was considered that temperatures of the pyrolyzer-GC injector interface (PY/GC-ITF) and of the GC injector could be lowered without deterioration of performance. Lowered temperatures help not only reduce the thermal decomposition of target compounds in the flow path, but also extend the life of the injector septum.

[Experimental] A certified polystyrene standard (obtained from AIST, Japan) containing 317 ppm of decabromodiphenylether (DeBDE) was used for analysis, and the previously recommended conditions (PY ITF: 340°C, GC injector: 320°C) for PY-2020iD was compared, in terms of reproducibility of repeated runs of DeBDE samples, with new conditions for EGA/PY-3030D (PY ITF: 300°C, GC injector 300°C).

[Results] A chromatogram obtained by thermal desorption GC/MS is shown in Fig. 1. The peak for the characteristic ion of DeBDE (m/z 799) is clearly observed in the extracted ion chromatogram. The peak area counts obtained by eight repeated runs for both methods are shown in Table 1. As clearly seen, similar results were obtained for both methods with good reproducibilities of less than 5 % RSD. This indicates that when using EGA/PY-3030D, PY ITF and GC injector temperatures can be as low as 300°C, lower than those recommended for PY-2020iD.

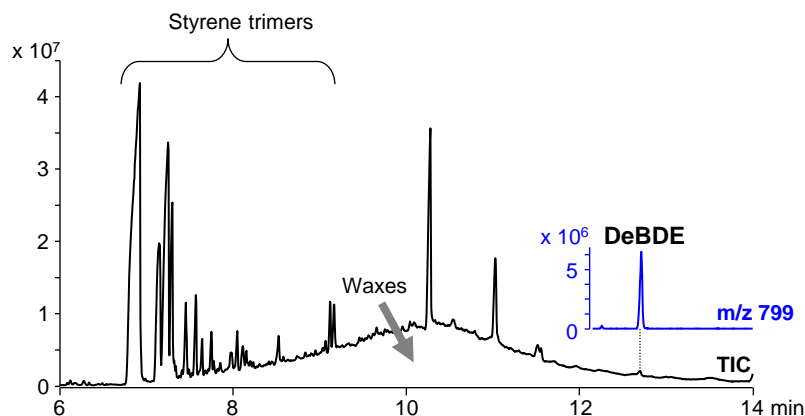


Table 1 Comparison of peak areas and reproducibilities of DeBDE

n	OLD*1	NEW*2
1	105222	105295
2	103226	110675
3	107656	104612
4	101563	106757
5	101622	112106
6	108631	105124
7	110670	119439
8	114883	112451
Average	109557	106684
RSD (%)	4.68	4.39

Fig. 1 Chromatogram of certified polystyrene standard obtained by thermal desorption GC/MS

Pyrolyzer furnace temp.: 200-300 (20 °C/min) – 340°C (5 °C/min, 1 min hold)
 GC oven temp.: 80-300°C (20 °C/min, 3 min hold)
 Separation column: Ultra ALLOY-PBDE (dimethylpolysiloxane, L=15 m, id=0.25 mm, df=0.05 μm)
 Carrier gas: He, 1 ml/min, split ratio: 1/20, sample amount: 500 μg

*1 Old: PY ITF 340°C, GC injector 320°C
 *2 New: PY ITF 300°C, GC injector 300°C

1) A. Hosaka, et al., *Anal. Sci.*, 2005, 21, 1145; 2) T. Yuzawa, et al., *Anal. Sci.*, 2008, 24, 953

Keywords : Brominated flame retardants, Decabromodiphenylether, RoHS directive, TD-GC/MS, PY/GC-ITF temp., GC injector temp.

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

Applications : Electrical / Electronic industry, Environmental analysis, General polymer analysis

Related technical notes : PYA1-071E, PYA1-072E

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