

Simple quantitative analysis of brominated flame retardant in polyester film pressure sensitive electrical insulating tape by thermal desorption GC/MS

[Background] The quantitative analysis of brominated flame retardants (BFRs) in consumer products such as electrical insulating adhesive tape, historically utilizes Soxhlet extraction to isolate and concentrate the compounds of interest. Soxhlet extraction is cumbersome, time consuming and often contributes to a reduction in data quality. Thermal desorption (TD)-GC/MS has been used as a simple technique for determining brominated flame retardants (PBDEs).^{1,2)} This note describes the quantitative analysis of BFRs in two types of polyester pressure sensitive electrical insulating tapes by TD-GC/MS.

[Experimental] Two polyester film pressure sensitive tapes (Permacelel P286 and ECRW507A) were characterized using Evolved Gas Analysis (EGA-MS). Small discs (0.5 mm in diameter) weighing around 70 µg) were obtained using a Harris Micro Puncher and placed in a sample cup for analysis. The desorption thermal zone of DeBDE was obtained from the EGA thermogram. The quantitation of DeBDE was based on a TD-GC/MS calibration curve prepared from DeBDE.

[Results] The EGA thermogram of polyester pressure sensitive tape (P286) is shown in Fig. 1(a). From the extracted ion chromatogram - m/z:799 (which is a characteristic ion of DeBDE), it can be determined that DeBDE evolves between 200°C and 350°C with a maximum at 320°C. Note that the thermal decomposition of the polymer starts at 200°C and ends at 450°C. Given these results, 200–380°C was selected as the thermal desorption temperature range for DeBDE. This maximizes the desorption of the DeBDE while minimizing the thermal decomposition of the polymer. The chromatogram of the sample obtained by TD-GC/MS is shown in Fig. 2 (a). The peak for DeBDE is observed at 13 min, and the DeBDE content of the adhesive tape was found to be 7.9% (n=3, RSD=2.4%). The EGA thermogram of the second polyester tape (ECRW507A) is shown in Fig. 1(b). While there was no peak observed in the extracted ion chromatogram: m/z:799, 1,2-bis(pentabromophenyl) ethane (BPBPE), which is a brominated flame retardant, was observed at 14 min as a broad unsymmetrical peak due to the sample capacity overloading. As demonstrated here, TD-GC/MS is a very powerful tool which greatly simplifies the analysis of brominated flame retardants in pressure sensitive tapes.

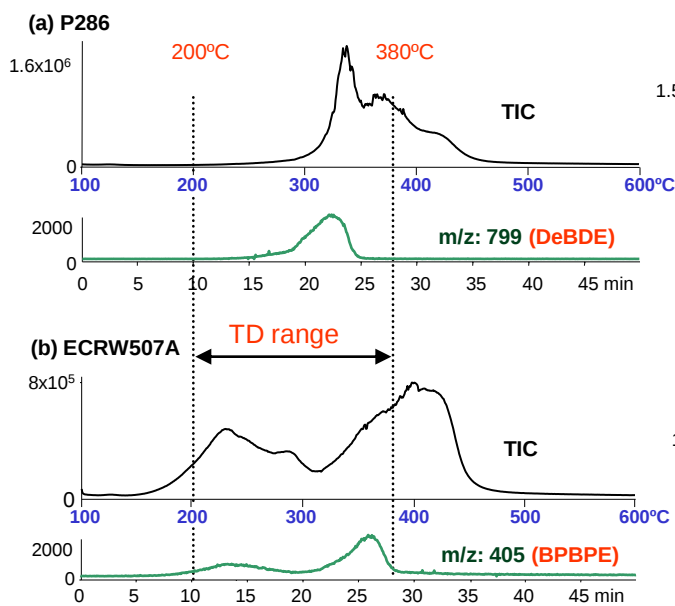


Fig. 1. EGA thermograms of (a) tape P286 and (b) tape ECRW507A

Py furnace temp.: 100-600°C (10°C/min), GC oven: 300°C

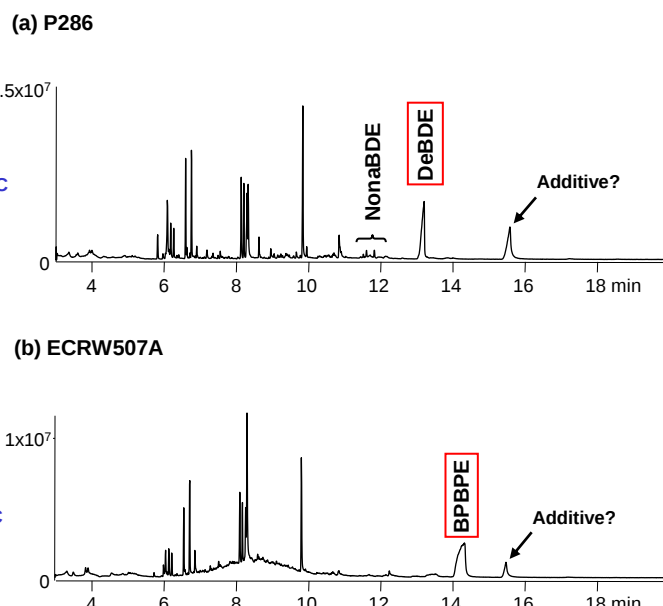


Fig. 2. Chromatograms of (a) tape P286 and (b) tape ECRW507A

Py furnace temp.: 200-380°C (5°C/min), GC oven: 80-300°C (20°C/min, 6 min hold)
Separation column: Ultra ALLOY-PBDE, L=15 m, id.=0.25 mm, df=0.05 µm

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

3) Yuzawa et al., 2008, 13th Poly. Anal. & Char

Keywords : Brominated flame retardant, DeBDE, BPBPE, thermal desorption, pressure sensitive adhesive tape

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

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

Related technical notes : PYA1-051E, PYA1-052E, PYA1-071E, UAT-006E

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