

Development of a new Ultra ALLOY® metal capillary column for analysis of brominated flame retardants

[Background] The RoHS Directive, which restricts specific toxic chemical substances used in electric and electronic devices, became effective in 2006 for EU countries. Brominated flame retardants were specifically addressed in the Directive; consequently a number of analytical methods have been studied. The analysis of brominated flame retardants can be performed using GC; however, the analysis of decabromodiphenylether (DeBDE) is chromatographically challenging, because it will debrominate if it comes in contact with reactive sites at high temperatures during the elution of DeBDE from the column. Of course, debromination will result in the deterioration of the analytical accuracy. Frontier Laboratories has developed a new Ultra ALLOY® metal capillary column: UA-PBDE, for the analysis of brominated flame retardants. UA-PBDE is very inert and has an ultra thin stationary phase film.

[Experimental] Employing a proprietary deactivation process, a stainless steel UA-PBDE column (liquid phase: dimethylpolysiloxane, L=15 m, i.d.=0.25 mm, df=0.05 µm) was prepared. The chromatograms of a 1,000 ppm solution of DeBDE obtained using the UA-PBDE and a conventional standard separation column (UA-1, dimethylpolysiloxane, L=15 m, i.d.=0.25 mm, df=0.1 µm) were compared to evaluate the performance.

[Results] As shown in Fig. 1, an asymmetric peak was observed on the UA-1 column. The ‘fronting’ can be attributed to the decomposition of DeBDE. On the other hand, no decomposition was observed when the new UA-PBDE column was used. The UA-PBDE stationary phase film thickness is only 0.05 µm which lowers the DeBDE elution temperature to 340°C. By lowering the elution temperature, the possibility of debromination is further decreased. Furthermore, the bleeding at 340°C is almost five times smaller than that of columns with 0.1 µm film.

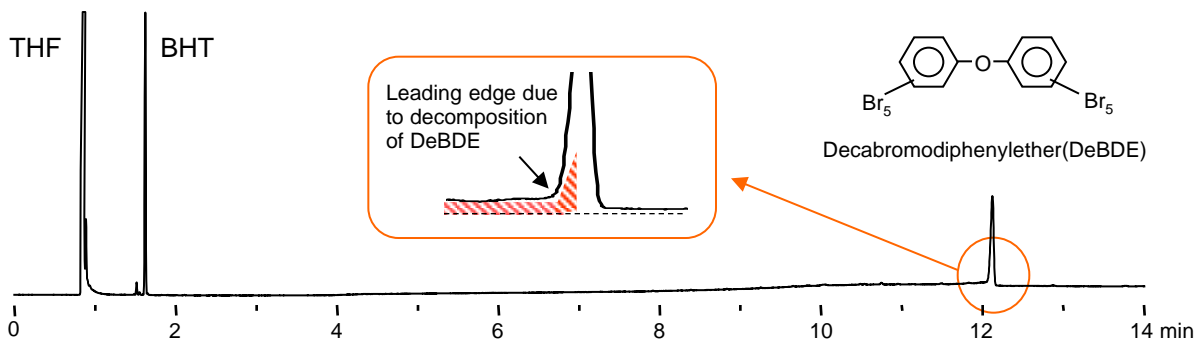


Fig. 1. Chromatogram of DeBDE obtained using UA-1 (dimethylpolysiloxane, L=15 m, i.d.=0.25 mm, df=0.1 µm)

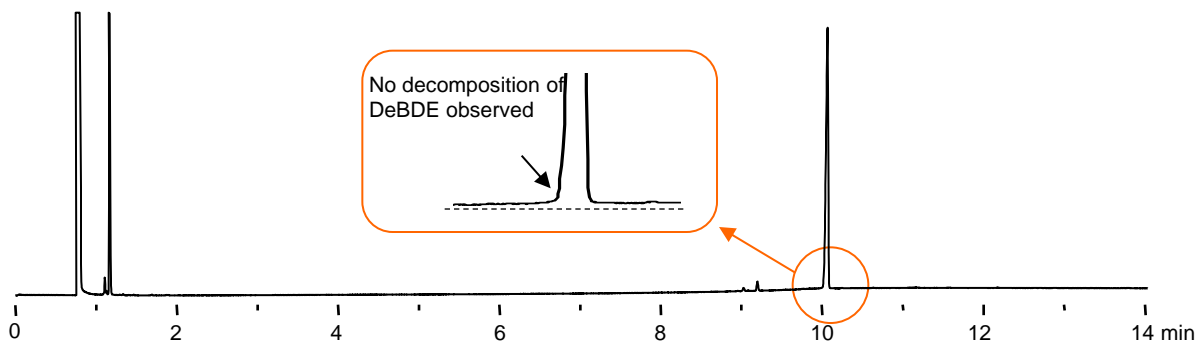


Fig. 2. Chromatogram of DeBDE obtained using UA-PBDE (dimethylpolysiloxane, L=15 m, i.d.=0.25 mm, df=0.05 µm)

Injector temp.: 300°C, column inlet pressure: 70 kPa, split ratio: 1/50,
 GC oven temp.: 150 – 340°C (20 °C/min)
 Sample: 1 µL of 1000 ppm DeBDE (decabromodiphenylether), detector: FID, detector temp.: 340°C

Keywords : DeBDE, PBDE, In-column decomposition, RoHS

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

Applications : Brominated flame retardant

Related technical notes :

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