

Screening Analysis of Brominated Flame Retardants with ATR Accessory (Part 2) -RoHS Directive-

The former Application News (No. A358) "Screening Analysis of Brominated Flame Retardants with ATR Accessory (Part 1) -RoHS Directive-" introduced a method for evaluating the presence of percent-order levels of decabromodiphenylether (DBDPE or deca-

BDE) in polystyrene.

This Application News presents spectra and evaluation results on plastics containing additives or octabromodiphenylether (octa-BDE).

■ Influence of additives

Calcium carbonate (CaCO₃) is a common additive to plastics. This additive reduces costs, and such plastics are widely used. For this work, polystyrene samples prepared with the addition of 40wt% CaCO₃ were used to confirm whether the presence of brominated flame retardants can be evaluated. Fig. 1 shows the spectrum of polystyrene blended with 40wt% CaCO₃ as an additive.

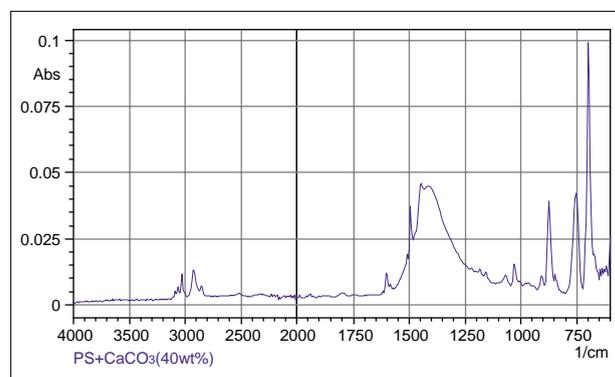


Fig.1 Spectrum of Polystyrene Blended with 40wt% CaCO₃ as an Additive

Fig. 1 exhibits a very large peak near 1400cm⁻¹. We prepared another sample that contained 5wt% deca-BDE as a brominated flame retardant and took its spectrum. Fig. 2 shows this spectrum superimposed over the spectrum in Fig. 1. The red line is the spectrum for the plastic contained with deca-BDE, while the blue line is for the not contained plastic (same as Fig. 1). The inserted diagram is an enlargement of the range 1750 to 1050cm⁻¹.

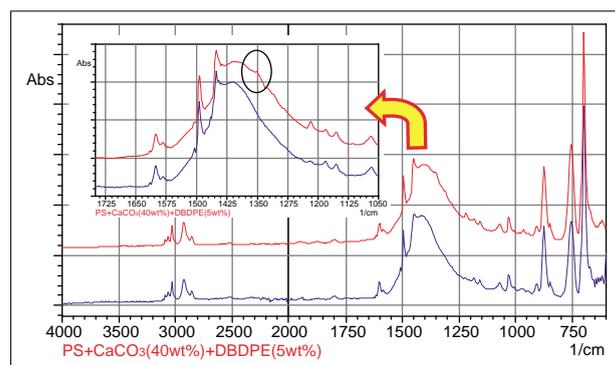


Fig.2 Spectra of PS/CaCO₃ that deca-BDE was contained and not contained

The inserted diagram in Fig. 2 exhibits a very small peak near 1350cm⁻¹ in the deca-BDE (red) spectrum. Fig. 3 shows the results of the evaluation of this spectrum by the brominated flame retardant-evaluation macro program. (deca-BDE is labeled PBDE in Fig. 3. The customer can freely change this notation.)

This result indicates that the presence of a brominated flame retardant can be evaluated, despite the additive. It also shows that the brominated flame retardant-evaluation macro program permits evaluation even for very small peaks, as in this example.

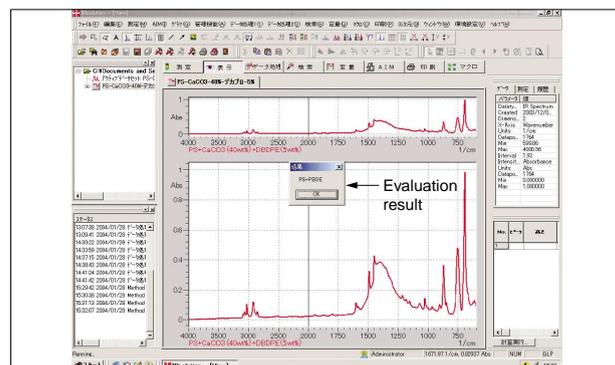


Fig.3 Evaluation by Macro Program of PS/CaCO₃ Doped with deca-BDE

■ ABS that contains octabromodiphenylether

We introduced the addition of decabromodiphenylether (deca-BDE) to polystyrene as a fire retardant in the Application News "Screening Analysis of Brominated Flame Retardants with ATR Accessory (Part 1) -RoHS Directive-".

This time, we introduce an ABS that contained octabromodiphenylether (octa-BDE), as a different polybromodiphenylether (PBDE). Firstly, we will discuss refer to the octa-BDE.

Commercial octa-BDE is generally in a mixture with penta-, hexa-, hepta-, nona-, and deca-BDE. Table 1 shows the average component ratio.

Octa-BDE was often added to ABS or PS as a fire retardant, but the demand for it finally dropped to zero in 2000. However, the introduction of the RoHS regulations brings a requirement to check samples containing octa-BDE. Consequently, we measured samples that contain octa-BDE. To check these samples, gas chromatograph-mass spectrometer (GCMS-QP2010) measurements were conducted initially and these spectra are shown in Fig. 4.

Fig. 4 confirms the addition of octa-BDE, as the content of hepta-BDE is highest, which matches the component ratio in Table 1. Fig. 5 shows the spectrum for the sample measured by FTIR. Fig. 5 also shows the spectra for ABS that contained deca-BDE and for ABS alone.

Fig. 5 reveals peaks caused by the flame retardant at approximately the same positions for octa-BDE and deca-BDE. Fig. 6 shows the results of evaluation of these spectra by the macro program.

Fig. 6 confirms that the "brominated flame retardant-evaluation macro program" can also be applied to the samples containing octa-BDE. However, the software cannot distinguish between octa-BDE and deca-BDE. For distinguish them, the usage of the GC-MS is recommended.

Table 1 Component ratio of octabromodiphenylether

penta-BDE	0.5% max.
hexa-BDE	12% max.
hepta-BDE	45% max.
octa-BDE	33% max.
nona-BDE	10% max.
deca-BDE	0.7% max.

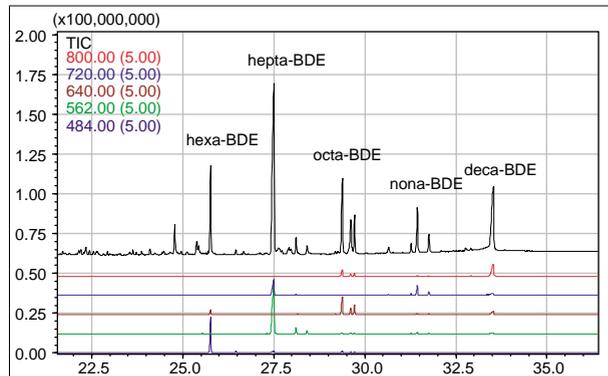


Fig.4 Mass Spectra of ABS Doped with octa-BDE

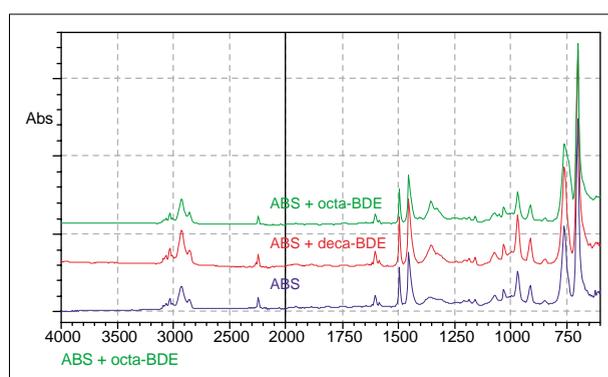


Fig.5 Spectra of ABS Doped with octa- and deca-BDE

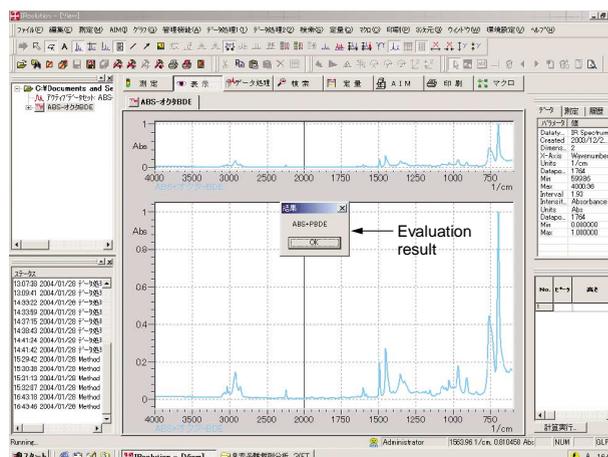


Fig.6 Evaluation by Macro Program of ABS Doped with octa-BDE