Application Note

No. 030TR0186-E

Simple Method for Quantitative Analysis of Brominated Flame Retardants with Far-Infrared Spectrometers (RoHS Directive)

Introduction

The simple methods for analysis of Brominated Flame Retardants in the mid-infrared region and in far-infrared region were reported in the FTIR application data No.030TR0185-E, and also it was suggested that the quantitative analysis in the mid-infrared region would be possible to some extent. This time, quantitative analysis results by the calibration curve in far-infrared region were compared with the results in the mid-infrared region in order to confirm the usefulness of analysis by FTIR spectroscopy. Based on such calibration curve, PBDE (Decabromodiphenylether) of electric and electronic part plastics were quantitatively analyzed.

Experimental

As the samples, electric and electronic part plastics were broken into pieces of a few millimeters and formed it into a film of 1 mm in thickness, 10 mm in size with a hot press (heated and pressed). The ATR method was used for the measurement in the mid-infrared region and the transmittance method was applied for the far-infrared region. The full vacuum type of model FT/IR-6000 series (for far-infrared) was used for the measurement. Both the measurements in the mid-infrared region and the one in far-infrared region are possible only by changing the beam splitter and detector. In the far-infrared region, it is possible to perform even the measurement of colour plastics including a large amount of inorganic compounds and also black plastics which are normally difficult with IR spectrometers and Raman spectrometers. The calibration curve was generated by utilizing the second derivative of absorption peak, 355 cm^{-1} of spectra of polystyrene containing PBDE. In this case the correction due to film thickness was implemented.

Results and Discussions

Fig. 1 shows the far-infrared spectra of polystyrene(Black) containing 0%, 15% and 30% of Brominated Flame Retardants. There is no absorption peaks of polystyrene in far-infrared region. The peaks of the antimony oxide added to improve the flame resistance are indicated by the red arrow. Since the presence of flame retardants can be confirmed clearly by the absorption peaks, it is very easy to analyze. In addition, it is possible to distinguish whether the sample contains the regulated Br compounds or the non-regulated ones.

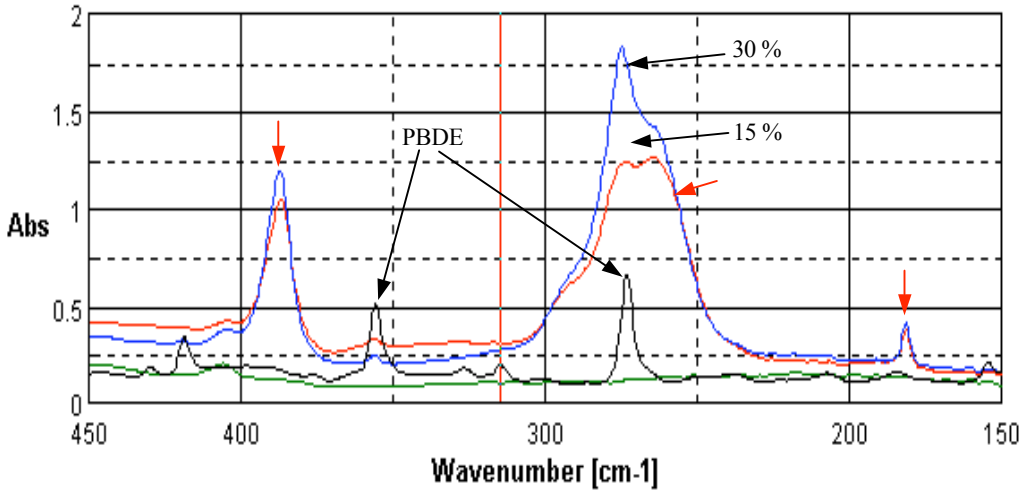

Fig. 1 Far-infrared spectra of polystyrene containing PBDE

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Fig. 2 is an example of the calibration curve in the far-infrared region. It is generated by one-peak method with the thickness correction.

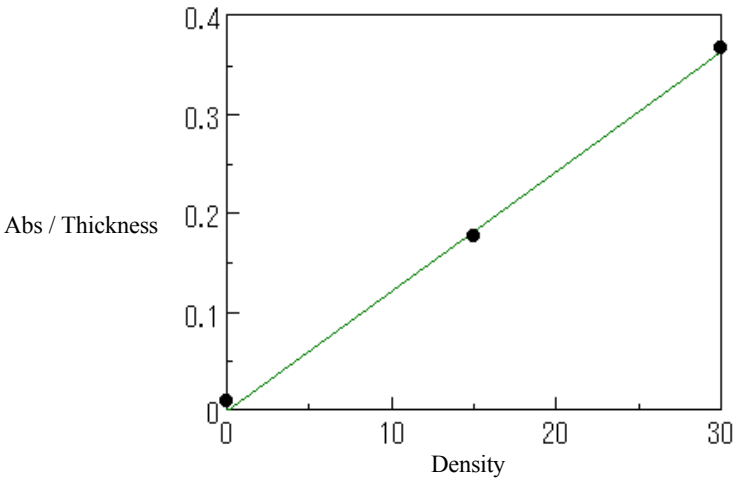

Fig. 2 Calibration curve by far-infrared spectra

Fig. 3 is another example of spectra of electric and electronic parts made of polystyrene obtained by the same method. Even if A and B look similar as the black plastics containing antimony oxide, B has three peaks due to PBDE, while A shows only one peak at the same three wavenumbers. A can be considered as decomposition product or isomer. C is transparent brown polystyrene and contained neither PBDE nor antimony oxide.

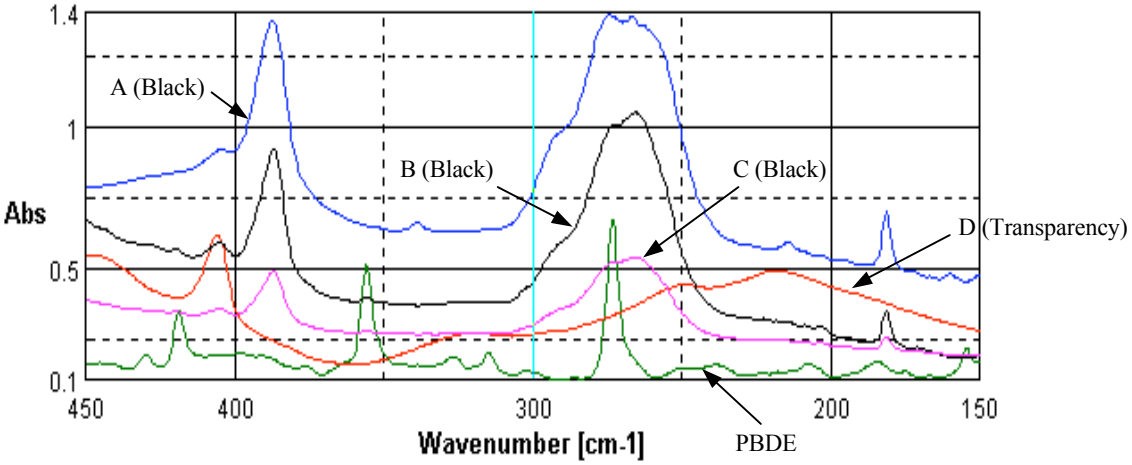

Fig. 3 Far-infrared spectra of electric and electronic parts made of polystyrene