Quantitative Analysis of Brominated Flame Retardant by Thermal Desorption-GC Technique

[Background] Brominated flame retardants are widely used and contained in electric and electronic devices, and are to be controlled under the Restriction of Hazardous Substances (RoHS directive). If the quantitative analysis of them is done by thermal desorption-GC technique (TD-GC), a various measurement parameters need to be optimized. Fig. 1 shows the construction of the TD-GC system. In this report, the temperature optimization for Py-GC interface (ITF) and GC injection port is described.

[Experimental] The Double-Shot Pyrolyzer® which is temperature programmable microfurnace pyrolyzer was directly attached to the split/splitless injection port of a GC equipped with a FID detector. The sample solution was prepared by adding ca. 5% (based on PS) of DeBDE into a THF solution containing 10 µg/µL of polystyrene (PS), then an aliquot (5µL) of the sample solution was placed in a sample cup and was allowed to dry prior to analysis. The thermal desorption temperature employed was 100~350ºC (20ºC/min), which was determined based on the EGA results.

[Results] Fig. 2 shows the peak intensity change of DeBDE contained in PS obtained by TD-GC technique as a function of the temperature of the Py-GC ITF and the GC injection port between 250 and 400ºC. It was found that between 300 and 370ºC, the peak intensity stayed constant, however, at temperatures below 300ºC and above 400ºC, the intensity decreased. At temperatures below 300ºC, DeBDE might have been absorbed in the flow path, and at temperatures above 400ºC DeBDE might have been thermally decomposed. Fig. 3 shows the reproducibility of the peak intensity plotted against the temperatures of the Py-GC ITF and the GC injection port. At temperatures between 300 and 370ºC, good RSD values of ca. 2% were obtained, while below 300 and above 400ºC, the reproducibilities were deteriorated. Based on these results, the optimum temperature for Py-GC ITF and GC injection port was found to be 320ºC, at which temperature DeBDE absorption and thermal decomposition can be avoided.

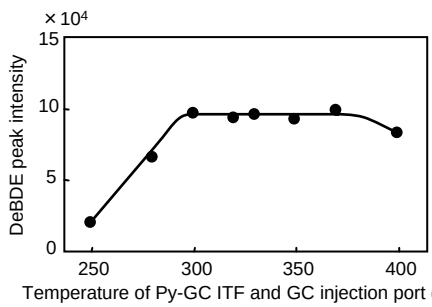

Figure 2 DeBDE peak area vs. Py-GC ITF and GC injection port temperatures

Furnace temp/: 100~350ºC (20ºC/min), sample size: 50µg (4.8% DeBDE contained)
Separation column: UA-PBDE (polydimethylsiloxane, length 15m, id 0.25mm, film thickness 0.05µm), Column flow rate: 1mL/min, split ratio: 1/50, detector: FID (360ºC)

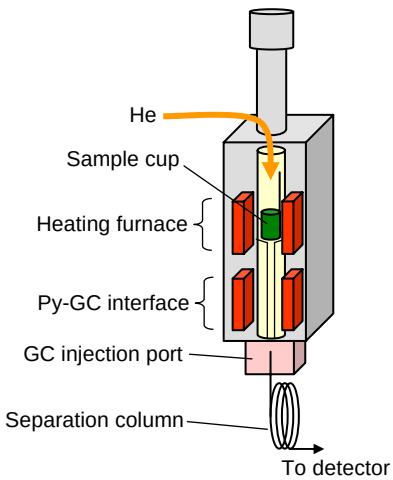

Figure 1 Construction of sample introduction section of Py-GC system

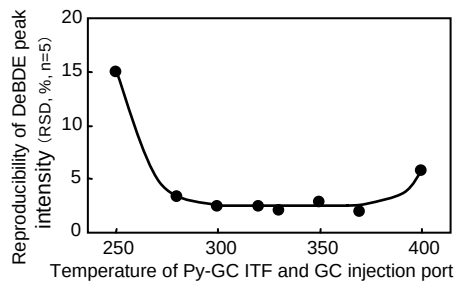

Figure 3 Reproducibility of DeBDE peak area vs. Py-GC ITF and GC injection temperatures
(analysis conditions are the same as above)

Contents excerpted from A. Hosaka, C. Watanabe, S. Tsuge, *Anal. Sci.*, 2005, 21, 1145

Keyword : Brominated flame retardant, RoHS directive, thermal desorption, decabromodiphenyl ether

Applications : Electrical and electronics industry, environmental analysis, general polymer analysis

Related technical notes : 1) PYA1-052E, 2) UAT-006E

Please direct your inquiries via our web site at :

http://www.frontier-lab.com/

R&D and manufactured by :
Frontier Laboratories Ltd.
1-8-14 Saikon, Koriyama
Fukushima-ken 963-8862 JAPAN
Phone: (81)24-935-5100 Fax: (81)24-935-5102

Dealer