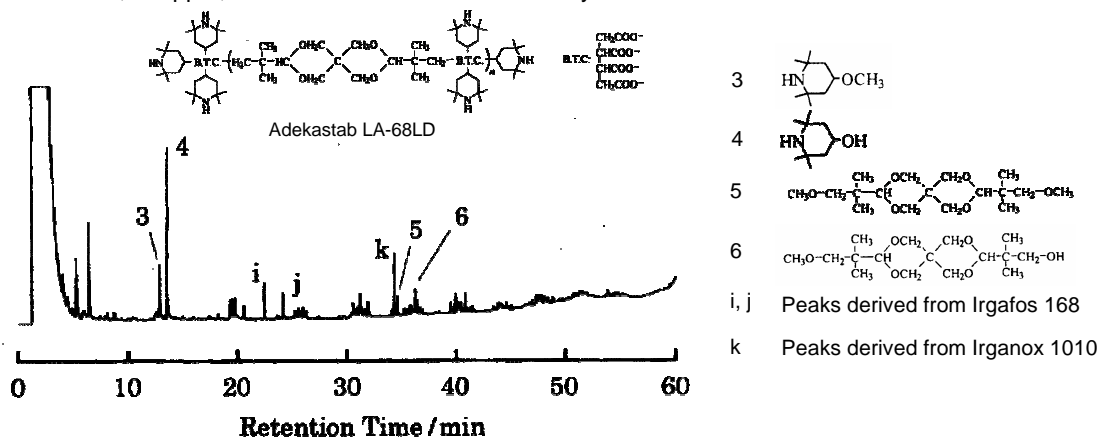


## Direct Determination of a Polymeric Hindered Amine Light Stabilizer in Polypropylene by Reactive Thermal Desorption-GC

**[Background]** Thermal desorption (TD)-GC has been successfully applied to direct analyses of various additives occluded in polymeric materials. Recently, a new TD-GC technique assisted by on-line chemical reaction, “reactive thermal desorption (RTD)-GC”, was reported to be a highly sensitive method to analyze polymeric additives including ester bondings, and/or free carboxylic and hydroxyl groups in their molecular structures. This note describes the direct determination of small amounts of polymeric hindered amine light stabilizers (HALSs) added to polypropylene (PP) by means of RTD-GC in the presence of tetramethylammonium hydroxide (TMAH).

**[Experimental]** Adekastab LA-68LD [MW ≈ 1900] was used as a polymeric HALS sample. PP composites containing the polymeric HALS along with antioxidants, Irganox 1010 and Irgafos 168, were prepared by blending PP with the additives by a kneader at 180–190°C in 5 min. About 30 μg of the cryo-milled PP composite sample was subjected to RTD-GC at 300°C in the presence of 2 μl of 25wt% TMAH methanol solution. The column temperature was set at 50°C, and heated to 300°C at a rate of 5 °C / min.

**[Results]** Figure 1 shows a typical chromatogram of the PP composite sample containing 10,000 ppm of HALS obtained by RTD-GC in the presence of TMAH at 300 °C. In this chromatogram, characteristic products reflecting the piperidine (peaks 3 and 4) and spiro moieties (peaks 5 and 6) in the original HALS molecules, formed through the partial methylation of the associated hydroxyl groups, were clearly observed without interference from the pyrolyzates of the PP backbones. Based on the intensities of peak 3-6, the direct determination of small amount of the polymeric HALS components, added to PP at concentration between 1,000 and 50,000 ppm, was carried out with a satisfactory relative standard deviation of ca. 5%.



**Figure 1.** Typical chromatogram of PP composite sample obtained by RTD-GC at 300°C.

Desorption temp. : 300°C, GC oven temp. : 50°C-(5 °C/min)-300°C

Separation column : HP Ultra1 (100%dimethylpolysiloxane), Length 25 m, 0.2 mm i.d., Film thickness 0.33 μm

Carrier gas flow : 50 ml/min, Column flow : 1.0 ml/min

\*Contents excerpted from K. Kimura, T. Yoshioka, Y. Taguchi, Y. Ishida, H. Ohtani, S. Tsuge, *Analyst* **2000**, 125, 465-468.

Keyword : Polymeric hindered amine light stabilizer, Polypropylene, Reactive thermal desorption-GC, TMAH

Applications : Additive analysis

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