Analysis of polymers/additives in three types of cable materials

Part 5 Thermal desorption (TD)-GC/MS and pyrolysis (Py)-GC/MS of Pellet C

[Background] In the previous note (PYA3-032E), the gases evolved from Pellet B, a raw material of cables for home electric appliances, were described. In this note, the qualitative analysis is described for polymer and additive components of Pellet C using TD-GC/MS and Py-GC/MS. Also, determination of an additive in Pellet C is described using TD-GC/MS.

[Experimental] Pellet C was pulverized by cryo-milling and used for the analysis. A Multi-Shot Pyrolyzer directly interfaced to the GC injector of a GC/MS system equipped with a MicroJet Cryo-Trap was used. A UA$^+$-5 column was used as a separation column and connected between the GC injector and the quadrupole MS detector. In TD-GC/MS, the sample was introduced into the pyrolyzer furnace, and the volatile components generated were temporarily coldcryo-trapped at the head of the separation column, followed by GC/MS analysis. In Py-GC/MS, the sample was dropped into the furnace heated at 600 ºC for flash pyrolysis.

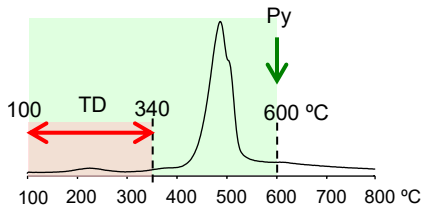

Fig. 1 EGA thermogram of Pellet C
(Excerpted from technical note PYA3-032E)

[Results] TD and Py temperatures were determined from the EGA thermogram (Fig. 1). From the TD chromatogram (Fig. 2a), pyrolyzates of a light stabilizer 2-(2'-hydroxy-3'-tert-butyl-5'-methylphenyl)-5-chlorobenzotriazole (commercial name Sumisorb 300, etc.) and an anti-aging agent 3,3'-thiodipropionic acid didodecyl (commercial name Nocrac 400, etc.), and saturated hydrocarbons derived from wax were detected. The concentration of the light stabilizer was found to be 2418 ppm as determined by the standard addition method. A small amount of acetic acid was also detected, which may be ascribed to the scission of the side chains of the polymer. The pyrogram (Fig. 2b) shows a pattern characteristic to polyethylene, indicating that the polymer is ethylene vinyl acetate (EVA).

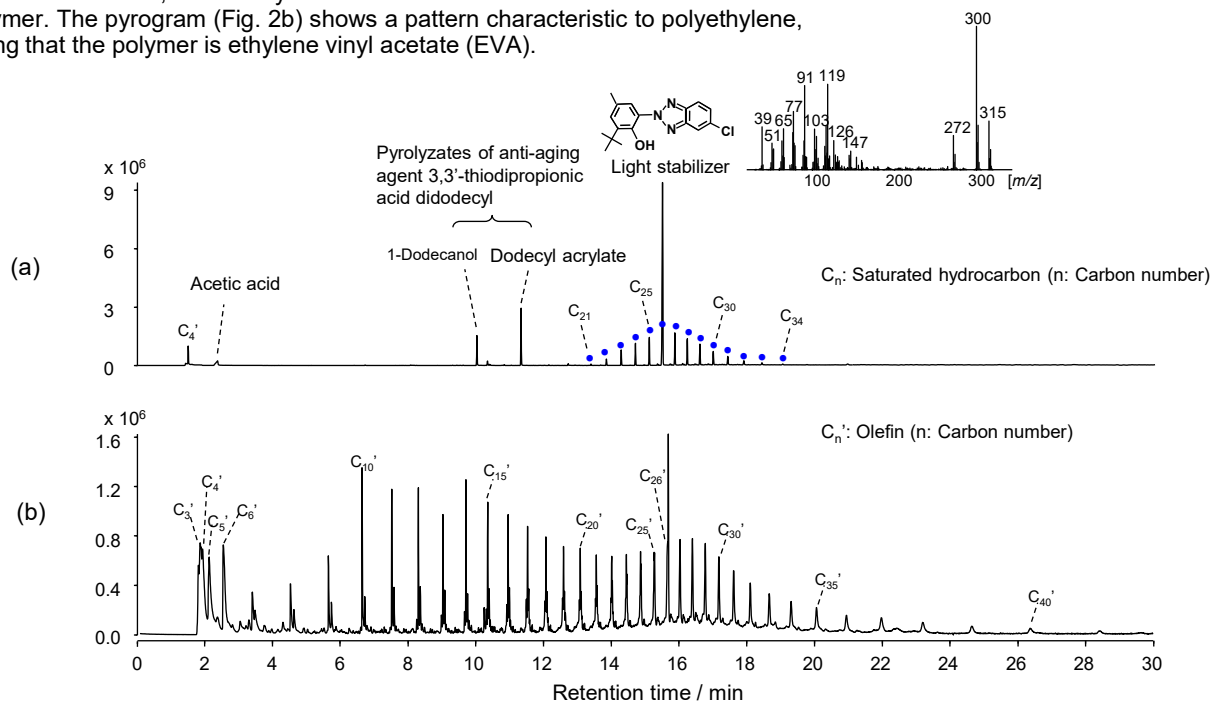

Fig. 2 TD chromatogram (a) and pyrogram (b) of Pellet C

(a) TD temp.: 100 – 340 ºC (20 ºC/min, 1 min hold), (b) Py temp.: 600 ºC
GC Inj. temp.: 300 ºC, GC oven temp.: 40 (2 min hold) - 320 ºC (20 ºC/min, 14 min hold), Split ratio: (a) 1/10, (b) 1/100
Column flow rate: 1.0 mL/min (He), Separation column: UA$^+$-5 (5 % diphenyl 95 % dimethylpolysiloxane), *L*=30 m, i.d.=0.25 mm, df=0.25 µm
MS scan range: *m/z* 29 – 1000, MS scan rate: 3 scan/s, Sample amount: (a) 0.5 mg, (b) 0.2 mg

Keywords : Cable, Pellet, EGA-MS, Thermal desorption-GC/MS, Pyrolysis-GC/MS

Products used : Multi-functional pyrolyzer, Auto-Shot Sampler, MicroJet Cryo-Trap, UA$^+$-5, Eco-Cup LF. Phthalate free quartz wool, F-Search, Vent-free GC/MS adapter

Applications : General polymer analysis, additives analysis, Quality assurance, Electronics, Materials analysis

Related technical notes : PYA3-031E (Part 1), PYA1-123E (Part2), PYA3-032E (Part 3), PYA1-124E (Part 4)

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Frontier Laboratories Ltd.
Phone: (81)24-935-5100 Fax: (81)24-935-5102
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