

Analysis of polymers/additives in three types of cable materials

Part 4 Thermal desorption (TD)-GC/MS and Pyrolysis (Py)-GC/MS of Pellet B

[Background] In the previous note (PYA3-032E), the evolved gas analysis of Pellets B and C was described. In this note, qualitative analysis of Pellet B for polymer and additives is carried out by thermal desorption (TD)-GC/MS and pyrolysis (Py)-GC/MS. Also, to determine an additive, Irgafos 168, in Pellet B, TD-GC/MS was conducted.

[Experimental] Pellet B 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. First, the sample was introduced into the pyrolyzer furnace for thermal desorption (TD), and the volatiles components generated were temporarily cryo-trapped at the head of the separation column, followed by GC/MS analysis. The sample cup was then returned to the standby position and the pyrolysis (Py)-GC/MS of the residue was performed by introducing the sample cup into the furnace heated at 600 °C to obtain a pyrogram.

[Results] From the EGA thermogram (Fig. 1), the TD and Py temperatures were determined, and from the TD chromatogram (Fig. 2a), an antioxidant tris(2,4-di-tert-butylphenyl)phosphite (commercial name Irgafos 168, etc.) was detected. Irgafos 168 was determined to be 218 ppm by the standard addition method. In addition, a small amount of acetic acid was detected, which may be ascribed to the scission of the side chains of the polymer. Also, the pyrogram (Fig. 2b) shows a pattern characteristic to polyethylene, indicating that the polymer is ethylene vinyl acetate (EVA).

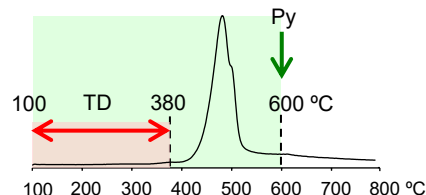


Fig. 1 EGA thermogram of Pellet B (Taken from the technical note PYA3-032E)

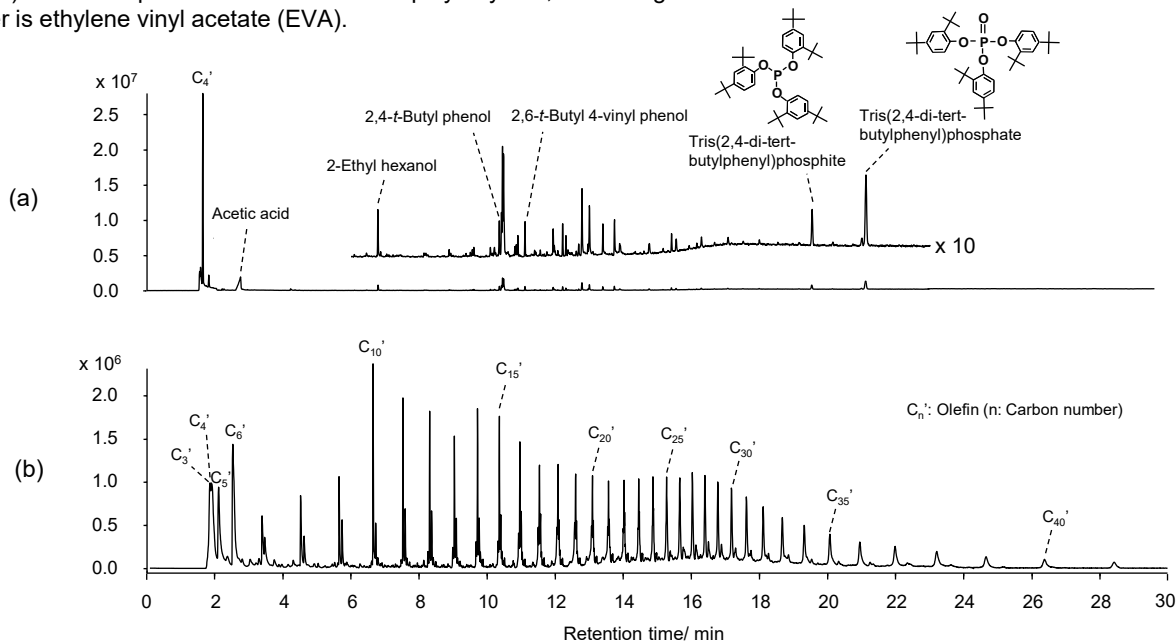


Fig. 2 TD chromatogram (a) and pyrogram (b) of Pellet B

(a) TD temp.: 100 – 380 °C (20 °C/min, 1 min hold). (b) Py temp.: 600 °C
 GC injector temp.: 300 °C, GC oven temp.: 40 (2 min hold) - 320 °C (20 °C/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 % dimethylsiloxane), L=30 m, i.d.=0.25 mm, df=0.25 μm
 MS scan range: m/z 29 – 1000, MS scan rate: 3 scans/s, Sample amount: (a) 1 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 (Part 2), PYA3-032E (Part 3), PYA1-125E (Part 5)

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