Analysis of polymers/additives in three types of cable materials

Part 2 Double-shot analysis of Pellet A

[Background] In the previous note (PYA3-031E), the evolved gas analysis of the gases generated from Pellet A, a raw material of cables, was described. In this note, the qualitative analysis of polymer and additives in the pellet was conducted on the same Pellet A as the one used in the previous note by using the double-shot method. Further, quantitative analysis of the additives was performed by thermal desorption (TD)-GC/MS.

[Experimental] About 0.1 mg Pellet A powdered by cryo-milling was 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. Thermally desorbed gases and pyrolyzates were separated by UA$^+$-5 metal capillary column. First, the sample was introduced into the pyrolyzer furnace for thermal desorption, and the volatile components generated were temporarily cryo-trapped at the head of the separation column, followed by GC/MS analysis. Then, the sample cup containing nonvolatile residue was returned to the standby position and the pyrolysis (Py)-GC/MS of the residue was performed by introducing it into the furnace preheated at 600 ºC to obtain a pyrogram.

[Results] From the EGA thermogram (Fig. 1), the TD and Py temperatures used in the double-shot analysis were determined. In the TD chromatogram (Fig. 2a), a non-phthalate plasticizer, dioctyl terephthalate (DOTP), was detected. The amount of DOTP was determined as 12.6 % by the standard addition method. In the pyrogram (Fig. 2b), pyrolyzates characteristic to polyvinyl chloride (PVC), such as HCl, indene, and naphthalene, were detected, indicating that Pellet A is mainly composed of PVC. In addition, a small amount of acetic acid, which is probably formed from the cleavage of the side chain of a polymer, was detected, suggesting that the polymer in the Pellet A is present as a blend of polyvinyl chloride and polyvinyl acetate, or a copolymer of polyvinyl chloride-vinyl acetate (P(VC-VAc)). Palmitic acid and stearic acid, used as lubricants, were also detected.

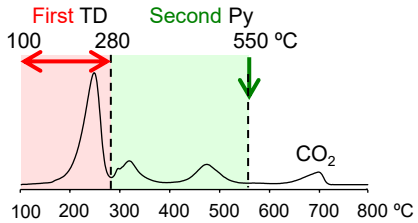

Fig. 1 EGA thermogram of Pellet A
(Taken from technical note PYA3-031E)

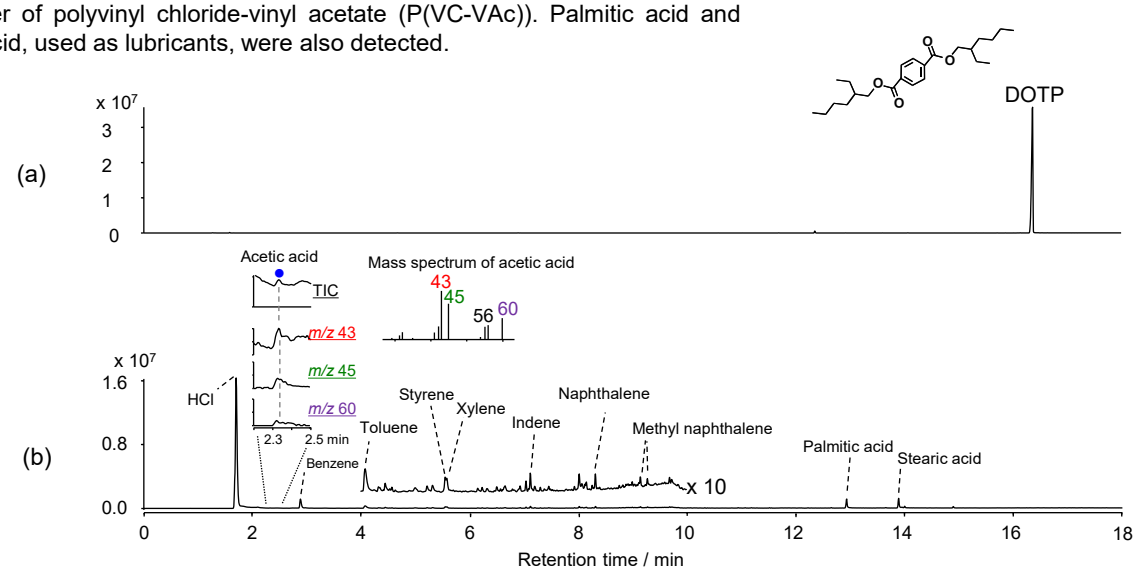

Fig. 2 TD chromatogram (a) and pyrogram (b) of Pellet A

(a) TD temp.: 100 – 280 ºC (20 ºC/min, 1 min hold), (b) Py temp.: 550 ºC
GC injector temp.: 300 ºC, GC oven temp.: 40 (2 min hold) - 320 ºC (20 ºC/min, 2 min hold), Split ratio: (a) 1/100, (b) 1/20
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: ca. 3 scan/s, Sample amount: ca. 0.1 mg

Keywords : Cable, Pellet, EGA-MS, Double-shot analysis

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, Additive analysis, Quality assurance, Electronics, Materials analysis

Related technical notes : PYA3-031E (Part 1), PYA3-032E (Part 3), PYA1-124E (Part 4), PYA1-125E (Part 5)

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