Analysis of polymers/additives in three types of cable materials

Part 1 Evolved gas analysis (EGA)-MS of Pellet A

[Background] Due to the recent enforcement of the revised RoHS Directive, analytical methods for polymers and additives contained in electrical cables have gained much interest. In this note, the evolved gas analysis (EGA)-MS of Pellet A, a raw material for cables used in home electric appliances, is described.

[Experimental] Pellet A was pulverized by cryo-milling and used for the analysis. A GC/MS system with a Multi-Shot Pyrolyzer directly interfaced to the GC injector was used. A deactivated metal tube connected the GC injector to the MS detector. About 0.2 mg sample was put in a sample cup (Eco-Cup) and introduced into the pyrolyzer furnace for EGA-MS analysis.

[Results] EGA thermogram of Pellet A is divided into 5 zones (Fig. 1). For Zone A-1, based on the library search on its averaged mass spectrum using F-Search, the large peak at around 250 ºC is ascribed to dioctyl terephthalate (DOTP), a plasticizer. It was also supported by the library search on its characteristic ions. DOTP was probably compounded as an additive, and its amount is suggested to be high, due to the high intensity of the peak. Similarly, from the averaged mass spectrum of the Zone A-2, the peak is ascribed to palmitic acid (characteristic ion: *m/z* 256) and stearic acid (characteristic ion: *m/z* 284). The peaks in Zones A-3 and A-4 are originated from the decomposition of polyvinyl chloride (PVC), from the observation of HCl (*m/z* 36) and naphthalene (*m/z* 128) in the averaged mass spectra. The averaged mass spectrum of Zone A-5 suggests the emission of carbon dioxide (*m/z* 44) in this zone, probably due to the release of carbon dioxide from calcium carbonate used as a filler by heating. For more detailed analysis, chromatographic analysis using the double-shot method (combination of thermal desorption and flash pyrolysis) will be described in another note (PYA1-123E).

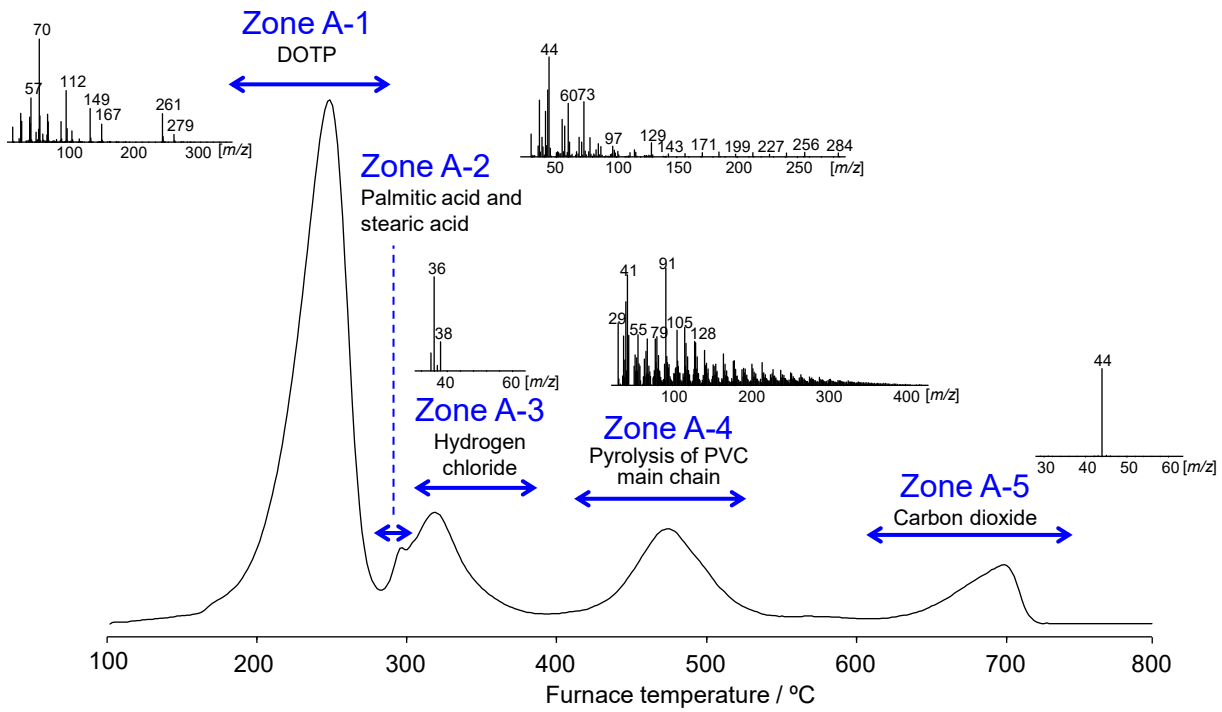

Fig. 1 EGA thermogram and averaged mass spectrum of each zone

Furnace temp.: 100 - 800ºC (20 ºC/min), EGA tube: UADTM-2.5N (*L*=2.5 m, id.=0.15 mm), Column flow rate: 1.0 mL/min (He), Split ratio: 1/50
GC oven temp.: 300ºC, MS scan range: *m/z* 29 - 1000, MS scan rate: 0.2 scan/s, Sample amount: 0.2 mg

Keywords : Cable, Pellet, EGA-MS, Evolved gas analysis

Products used : Multi-functional pyrolyzer, Auto-Shot Sampler, UADTM-2.5N, 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 : PYA1-123E (Part 2), PYA3-032E (Part 3), PYA1-124E (Part 4), PYA1-125E (Part 5)

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