

Analysis of polyvinyl alcohol contaminated by a small amount of polymer using heart-cut (HC) EGA-GC/MS

[Background] When a contaminated polyvinyl alcohol (PVA) sample was analyzed using pyrolysis-GC/MS, peaks associated with the contaminant could not be isolated because of the interference generated by the ionization of the PVA base polymer. To reduce the impact of the interference, heart-cut/evolved gas analysis (HC/EGA)-GC/MS was used. Only the portion of the sample eluting over a pre-selected temperature range was transferred to the column and subsequently analyzed by GC/MS. This eliminated a significant contribution from the interference.

[Experimental] A Multi-Shot Pyrolyzer (EGA/PY-3030D) was interfaced directly to the split injector of the GC. A Selective Sampler (SS-1010E)¹ was attached directly to the exit of the splitter. Two samples were analyzed using EGA-MS: (1) a PVA sample and (2) a white-turbid PVA sample which was thought to be contaminated by some unknown polymer. The two EGA thermograms had similar profiles (Fig. 1).

[Results] Chromatograms of the 460-540°C heart-cut zone for both samples are shown in Fig. 2. There are no discernible differences in the total ion current (TIC) chromatograms of the two samples; however, in the extracted ion chromatogram (EIC) of the ion at m/z 57, the white-turbid PVA sample shows peaks associated with C₁₆-C₃₃ saturated hydrocarbons. Such peaks are not observed in the EIC of PVA. These results suggest that the white-turbid PVA sample is contaminated with a polymer or polymers possessing a saturated hydrocarbon structure.

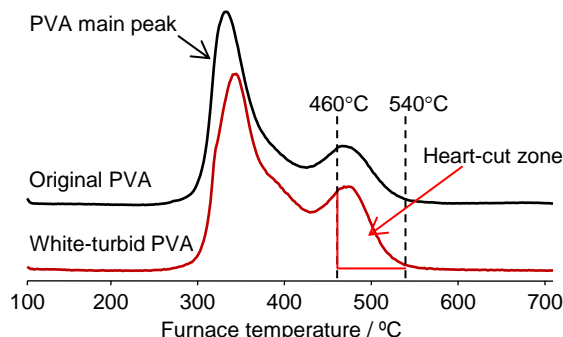


Fig. 1 EGA thermograms of PVA and white-turbid PVA
 Py furnace temp.: 100 – 700°C (20 °C/min), split ratio: 1/50,
 GC oven temp.: 300°C, column flow rate: 1 mL/min (He), EGA tube:
 UADTM-2.5N, L=2.5 m, i.d.=0.15 mm, **Sample amount.: 0.2 mg**

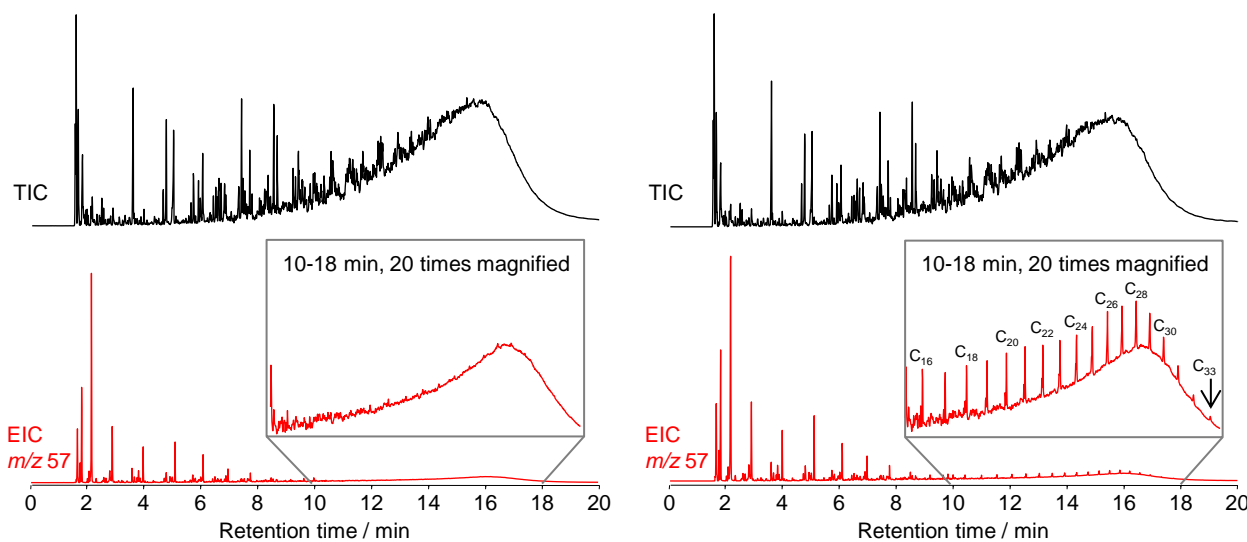


Fig. 2 Chromatograms of PVA samples (heart-cut zone: 460-540°C, left: original PVA, right: white-turbid PVA)

Py furnace temp.: 460 – 540°C (20 °C/min), split ratio: 1/20, GC oven temp.: 40 (2 min hold) – 320°C (20 °C/min, 10 min hold)
 Separation column: UA+5 (5% diphenyl 95% dimethylpolysiloxane), L=30 m, i.d.=0.25 mm, df=0.25 µm, column flow rate: 1 mL/min (He),
Sample amount.: 11.7 mg,
 Heart-cut zone cryo-trapped by MicroJet Cryo-Trap (MJT-1035E) and Selective Sampler (SS-1010E).

¹) See [catalog](#) or [operation manual](#) for details.

Keywords : Impurities, Hear-cut analysis, HC/EGA-GC/MS, Polyvinyl alcohol

Products used : Multi-functional Pyrolyzer, Selective Sampler, MicroJet Cryo-Trap, UA+5, Vent-free GC/MS adapter

Applications : Polymer analysis, Quality assurance

Related technical notes : [PYA1-070E](#), [PYA3-022E](#)

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