

Empirical Determination of Proper Pyrolysis Temperature

In pyrolysis gas chromatography (Py-GC), the pyrolysis temperature of a given sample among one of the most important factors influencing analytical results. In order to obtain a good reproducibility, finding a proper pyrolysis temperature is important. Using polystyrene (PS) as a sample, effects of pyrolysis temperature to the reproducibility of their pyrograms is described. The pyrogram of PS (Fig.1) comprises styrene monomer (S), styrene dimer (SS) and styrene trimer (SSS). Fig. 2 shows a plot of the observed relative standard deviation, RSD (%) for the formation ratio (SSS/S) against pyrolysis temperature. RSD curve shows the minimal at 550°C, and rises as the pyrolysis temperature above 550°C.

Pyrolysis temperature giving an excellent reproducibility can be empirically determined to be about 50°C above the finishing temperature of the EGA curve obtained by the evolved gas analysis technique¹ (EGA).

The optimum pyrolysis temperature for a given polymer sample changes depending on its thermal characteristics such as thermal stability, depolymerizability and etc. depending on compounds to be analyzed².

- 1) Double-Shot Pyrolyzer® Technical Note, PYT-004
- 2) Basic Pyrolysis Gas Chromatography of Polymers and Database, P9, S.Tsuge and H. Ohtani

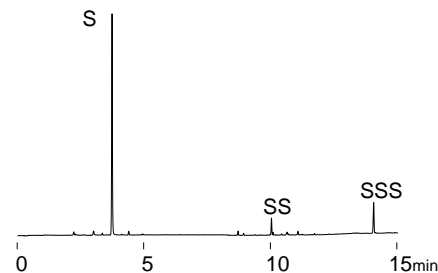


Fig. 1. Pyrogram of PS

Sample : 30µg, Pyrolysis temp.: 550°C, Split ratio : 1/50
 Column : 5% diphenylpolysiloxane, Length 30m, Id 0.25mm
 Film thickness 0.25µm, (Ultra ALLOY®-5, Frontier Labs)
 GC oven temp.: 70°C~320°C (20°C/min), Detector : FID

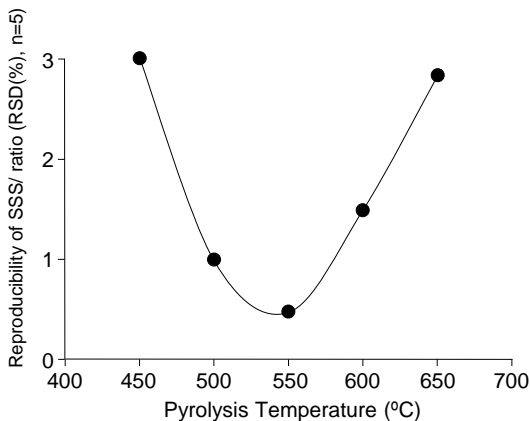


Fig. 2. Plot of Reproducibility of SSS/S (%) vs. Pyrolysis Temperature

Sample cup SS (P/N : PY1-F003, Single-Shot (5µL), platinum)

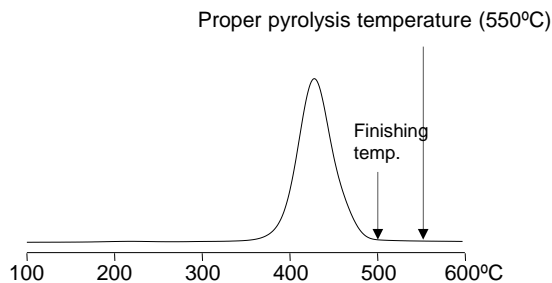


Fig. 3. EGA Curve of PS

Pyrolysis temp.: 100°C~6000°C (20°C/min), Carrier gas : He 50kPa, Split ratio : ca. 1/50
 EGA capillary tube : Id 0.15mm, Length 2.5m (UADTM-2.5N)
 GC oven temp.: 300°C, Injection. port temp.: 320°C, Sample : 30µg, Detector : FID

Keywords : Proper Pyrolysis Temperature, Polystyrene, Reproducibility, Evolved Gas Analysis

Products used : Multi-functional pyrolyzer, UA-1, UADTM-2.0N

Applications : General Polymer Analysis

Related technical notes :

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