Effects of measurement conditions on peak temperature in Evolved Gas Analysis (EGA)-MS Part 1: Sample amount

[**Background**] Evolved gas analysis (EGA)-MS is an effective analytical tool to obtain information on the thermal desorption temperature, pyrolysis behavior of polymers, and the chemical species of the gases evolved from a sample. In this report, the relationship between the sample amount of polystyrene (PS) and the peak temperature of the EGA curve in EGA-MS was examined.

[**Experimental**] A GC/MS system with a Multi-Shot Pyrolyzer directly interfaced to the GC inlet was used. A deactivated metal tube (UADTM-2.5N) and a Vent-free GC/MS adapter were used to connect the GC inlet to the MS detector. Five powder PS samples from 0.1 mg to 5.0 mg were weighed into an Eco-Cup and introduced into the furnace, and EGA-MS measurements of the samples were performed from 100 to 700 ºC at a temperature ramp rate of 20 ºC/min.

[**Results**] The intensity-normalized EGA curves of PS are shown in Fig. 1. In each EGA curve, a single peak with a peak temperature of 440 - 450 ºC was observed. The peak temperature plotted against the sample amount is shown in Fig. 2. As the sample amount increases, the peak temperature shifts from 441.2 ºC (sample amount: 0.136 mg) to 446.9 ºC (sample amount: 4.916 mg), observing a nearly linear correlation between sample amount and peak temperature. However, as shown in Fig. 1, the shape of the EGA curve is distorted when the sample amount exceeds 1 mg. The larger sample amount is directly related to the larger sample volume, and the distorted EGA curve will be ascribed to the unfavorable thermal conduction for samples with larger volumes. The depolymerization reaction of PS is endothermic, and PS has low thermal conductivity (<0.2 W/m·K). Accordingly, as the sample volume increases, it becomes more difficult for heat to be transferred to the inside of the sample. Therefore, to obtain data on the peak temperature in EGA-MS and investigate thermal desorption/pyrolysis behavior, it is crucial to measure a sample with a minimal amount and ensure uniform sample volume across all samples.

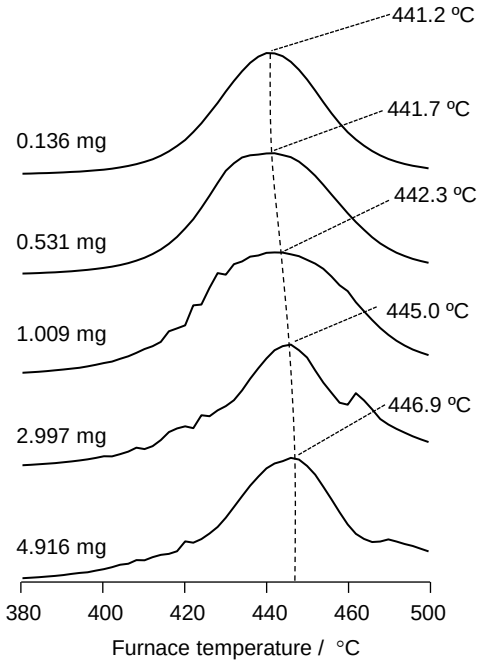

Fig. 1 EGA curves of PS with varied sample amounts.

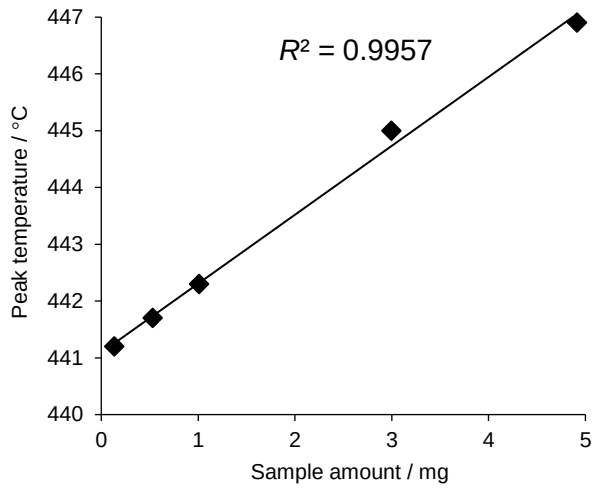

Fig. 2 Relationship between peak temperature and sample amount in EGA curves of PS.

Furnace temp.: 100 - 700 ºC (20 ºC/min), EGA tube: UADTM-2.5N (*L*=2.5 m, i.d.=0.15 mm), Flow rate: 1 mL/min (He), Split ratio: 1/50, GC oven temp.: 300 ºC, MS scan range: *m/z* 29 - 550, MS scan rate: approx. 0.2 scan/s, Sample amount: 0.1 - 5.0 mg.

Reference: A. Shiono *et al.*, *Polym. Test.*, 42 (2015) 54-61.

Keywords : EGA-MS, Thermal analysis, Thermal decomposition behavior, Polystyrene

Products used : Multi-Shot Pyrolyzer, Auto-Shot Sampler, UADTM-2.5N, Eco-Cup LF, Vent-free GC/MS adapter

Applications : General polymer analysis, Material analysis

Related technical notes : PYA3-046E (Part 2)

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