Improvement in peak separation of low-boiling components in polyethylene pyrograms using Flow-Through Cups

[Background] In GC analysis, trace amounts of samples or analytes usually lead to decreased peak intensities. In such cases, reducing the carrier gas flow rate and lowering the split ratio at the GC inlet are common approaches to increase the amount of sample introduced into the detector. However, in pyrolysis (Py-) GC measurements, these approaches are likely to promote secondary reactions during pyrolysis, and peak broadening due to the stagnation of pyrolyzates within the sample cup becomes non-negligible. On the other hand, using a “flow-through cup”, which has a single hole at the bottom of the sample cup, is expected to suppress significant stagnation of the pyrolyzates (Fig. 1). In this note, the effect of the cup structure on the peak width and peak separation of the pyrolyzates of polyethylene (PE) was examined.

[Experimental] A Py-GC/MS system with a Multi-Shot Pyrolyzer (EGA/PY-3030D) directly interfaced to the GC inlet was used. Approximately 300 µg of PE was placed in either an Eco-Cup LF or a flow-through cup LHF. Prior to sample loading, quartz wool was placed at the bottom of the flow-through cup to prevent the sample from falling through the cup. The furnace temperature was set to 800 ºC, and pyrograms of PE were measured at total carrier gas flow rates ranging from 7 to 100 mL/min.

[Results] The pyrograms of PE obtained at a total flow rate of 15 mL/min using two types of sample cups are shown in Fig. 2. With the Eco-Cup LF, the low-boiling components show broader peaks due to stagnation of the carrier gas containing the pyrolyzates, whereas narrower peaks with better separation are obtained with the flow-through cup LHF . These results indicate that the flow-through cup LHF is effective when the total flow rate is low. However, at higher flow rates (50-100 mL/min), no difference in the half-width of 1-octene (C8’) peak was observed between the two cups (Fig.3).

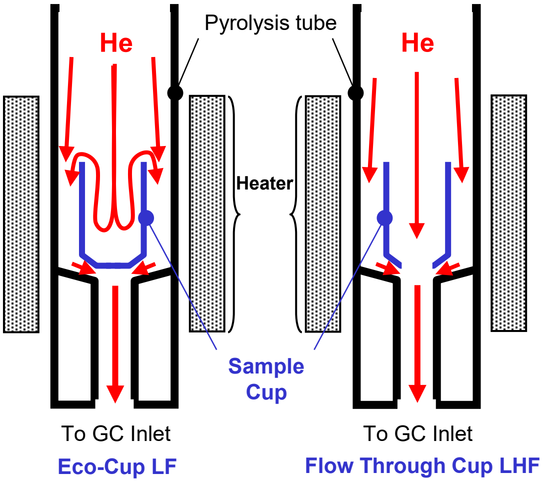

Fig. 1 Design of the flow-through sample cup and the carrier-gas flow

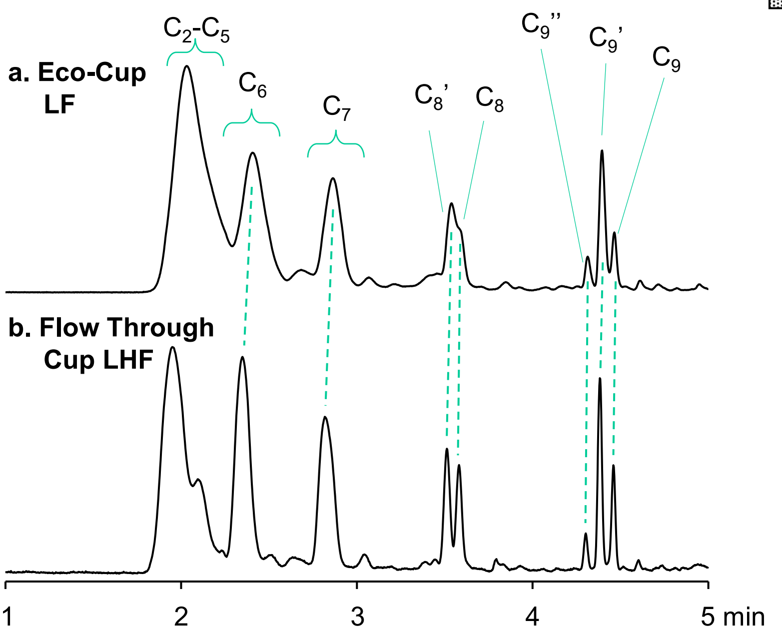

Fig. 2 Pyrograms of PE (Low-boiling components only)

Pyrolysis temp.: 800ºC, GC inlet temp.: 300 ºC, column: UA5-30M-0.25F, (*L* = 30 m, i.d.= 0.25 mm, df = 0.25 µm), GC oven temp.: 40-300 ºC (20 ºC/min), column flow: 1.0 mL/min (He), total flow: 15 mL/min, Sample amount: ca. 300 µg

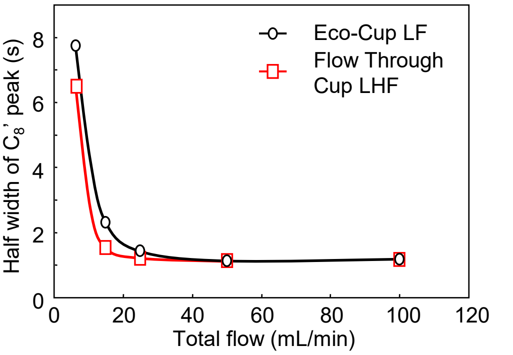

Fig. 3 Relationship between the total flow during pyrolysis and the half-width of the C8′ peak in the pyrogram of PE (EIC: *m/z* 112)

Ref.) A. Hosaka *et al.*, *J. Anal. Appl. Pyrolysis* **78** (2007), 452-455.

Keywords : Polyethylene, low boiling-point compounds, improving peak resolution

Products used : Multi-functional Pyrolyzer, Vent-free GC/MS adapter, Eco-Cup LF, Eco-Cup LHF (Flow Through)

Applications : Polymer analysis

Related technical notes : PYT-029E

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