Improvement of Temperature Profile at Py-GC Interface by Heat Sink Adaptor and Reduction of Memory Effect for Low and Reactive Volatile Components

[Background] The split/splitless injection port of the today's GCs is designed to keep the vicinity of the septum at relatively lower temperatures so that gas bleedings from the septum is minimized. A Py-GC system consisting of such a GC and a pyrolyzer has a temperature trough at the interface between the GC and pyrolyzer, resulting in trapping of higher boiling pyrolyzates. This phenomenon gives rise to reduced reproducibility and memory effects in repeated runs, impairing the basic performance of the Py-GC system. Therefore when building a Py-GC system, this point needs to be taken into account and appropriate measures be implemented. In a Double-Shot Pyrolyzer system having this implemented, a heat sink adaptor is placed at the Py/GC interface to reduce the temperature trough. The effectiveness of the improved design is described here.

[Experimental] A thermocouple (outer diameter 1mm) was inserted either from the top of the pyrolyzer or from the bottom of the GC injection port to determine the temperature profile inside the system with and without a heat sink adaptor. Also, in both cases, pyrograms of polyurethane were obtained and the memory effects were compared. Upon pyrolysis, polyurethanes give diphenylmethane diisocyanate (MDI), a very reactive and high boiling species.

[Results] As seen by the temperature profile inside the system shown in Fig. 1, the temperature profile of the pyrolyzer section was not influenced by the heat sink adaptor, but at the interface a significant difference was observed. The temperature of the septum rubber at the GC injection port was 170ºC, a large temperature trough, in the system without a heat sink adaptor. On the other hand, in the system with a heat sink adaptor it was 210ºC, or 40ºC higher, because the septum rubber is heated by the Py/GC interface block through the heat sink adaptor, resulting in a smaller temperature trough. Fig. 2 shows how the heat sink adaptor reduces the memory effect in a pyrolysis of polyurethane giving MDI. It was clearly shown that by inserting the heat sink adaptor, the ghost peak intensity of MDI was able to be reduced to 0.8% from 4.3% in blank runs.

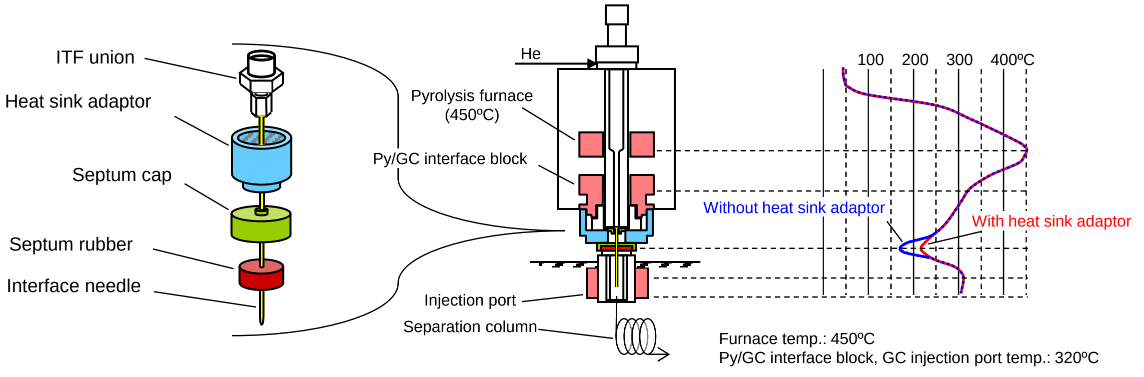

Fig. 1 Py-GC system with heat sink adaptor, improving temperature profile

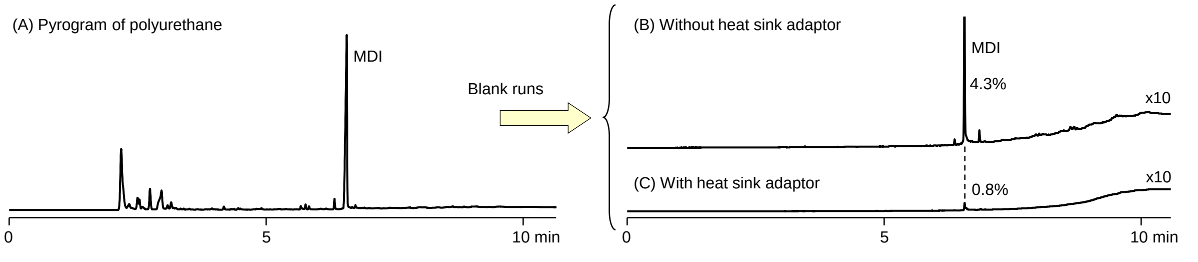

Fig. 2 Pyrogram of polyurethane and memory effects after blank runs

Pyrolysis furnace temp. : 550ºC, Flow rate : 1ml/min, Split ratio : ca.1/50, Separation column : Ultra ALLOY5+ (5% diphenyl 95% dimethylpolysiloxane, length 15m, id 0.25 mm, film thickness 0.25µm)
GC oven temp.: 70-350ºC (30ºC/min), Injection port temp.:320ºC, Sample size : ca 300 µg

Keywords : Heat sink adaptor, memory effect, polyurethane, MDI

Products used : Multi-functional pyrolyzer, UA-5

Applications : General polymer analysis

Related technical notes : PYT-026E

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