

Improvement of Temperature Profile at Py-GC Interface and Yield Enhancement of High Boiling Components by Heat Sink Adaptor

Background] The temperature “trough” at the Py-GC interface gives rise to a reduced reproducibility of pyrograms and memory effects in repeated runs. The Double-Shot® Pyrolyzer is designed to reduce the temperature “trough” through inserting a heat sink adaptor(PYT-025E). Here, the effect on the yield of high boiling components of pyrolysates is described.

[Experimental] A thermocouple 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. In both cases, pyrograms were obtained by vaporizing polywax 655, which consists of linear hydrocarbons of C₂₀~C₉₀, and the yields of high boiling components were compared.

[Results] As seen by the temperature profile inside the system shown in Fig. 1, the temperature profile at the center of the pyrolyzer was not influenced at all by the heat sink adaptor, but a significant difference was observed at the interface. The temperature of the septum at the GC injection port was 170°C, creating 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 210°C, or 40°C 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 the effect of the heat sink adaptor on the yield of high boiling components. In the n-C₂₀~C₃₀ region, the peak intensities were almost the same for both cases, while in the n-C₃₀~C₈₀ region, the yield was much improved with the introduction of the heat sink adaptor. The yield of n-C₄₀ component was greatly improved to near 100% from 25%.

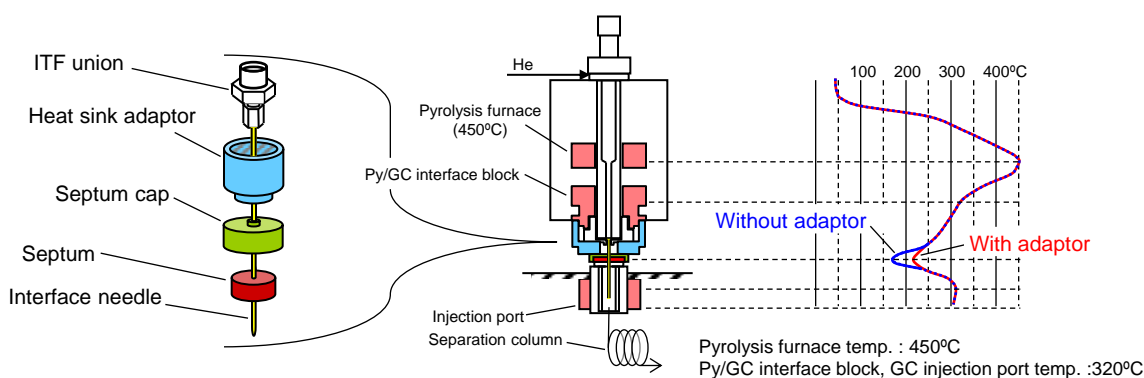


Figure 1. Py-GC system with a heat sink adaptor install and improvement of temperature profile

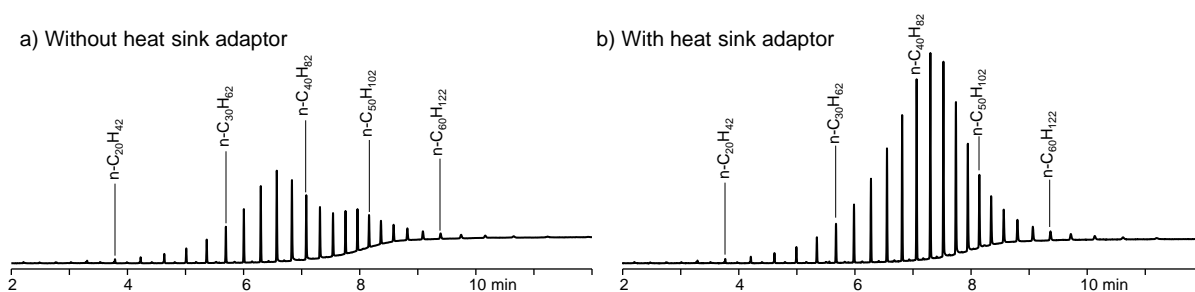


Figure 2. Chromatograms of mixture of hydrocarbons (n-C₂₀~n-C₉₀)

Pyrolysis furnace temp.: 450°C, carrier gas flow rate : 1ml/min, split ratio : 1/50
 Separation column : Ultra ALLOY-5(MS/HT) (5%-diphenyl-95%-dimethylpolysiloxane, length 15m, id 0.25mm, film thickness : 0.1µm)
 GC oven temp.: 100~ 420°C (40°C/min), Injection port temp.: 320°C, Py-GC interface temp.: 320°C, Sample size : ca.10µg

Keywords : Heat sink adaptor, memory effect, high boiling components, hydrocarbons

Products used : Multi-functional pyrolyzer, UA-5 (MS/HT)

Applications : General polymer analysis, Polywax 655

Related technical notes : PYT-025E

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