

# Development of an On-line micro Reaction Sampler

**[Background]** Reactive pyrolysis GC/MS has been used as a powerful tool for composition analysis of condensation polymers such as polyesters. The reaction with a reagent is generally carried out in an open system under near atmospheric pressure; however, polymers such as polyamides do not undergo thermally assisted hydrolysis and methylation (THM) reaction at atmospheric pressure. Alternatively, the method can be performed at elevated temperatures under high pressures in a sealed vessel. Because of safety concerns, the reaction is generally performed off-line.

We have developed an On-line micro Reaction Sampler for use with the Multi-Shot Pyrolyzer. A minute amount of sample and a reagent are sealed in a glass capsule. The reactions occur at high temperatures under high pressures. Reaction products are analyzed using GC/MS. In this technical note, the Reaction Sampler is briefly described. The reactive pyrolysis of nylon 6,6 in a closed system and an open system served to illustrate the effect of working at elevated pressures.

**[Experimental]** A diagram of the micro Reaction Sampler (part number PY1-1050) is shown in Fig. 1. Nylon 6,6 (40 µg) and tetra-methyl ammonium hydroxide (TMAH - 10 µL of 25 wt% methanol solution) were placed in a glass capsule (od. 2.5 mm, L=30-35 mm, vol.=76 µL). The capsule was flame-sealed and attached to the Reaction Sampler. The sampler was then attached to a Multi-Shot Pyrolyzer (EGA/PY-3030D). Pushing the knob on the top of the sampler down, lowers the capsule into the pyrolyzer's furnace (300°C). The capsule was heated for one hour. At this time, the capsule was crushed and the reaction products flushed into the GC column by the carrier gas. Reactive pyrolysis in open system was performed in a standard manner using the EGA/PY-3030D Pyrolyzer.

**[Results]** The chromatogram of nylon 6,6 obtained by reactive pyrolysis in open system is shown in Fig. 2a). Peaks for the reaction products were very small because the reaction does not occur at low pressure. The chromatogram of nylon 6,6 obtained by reactive pyrolysis in the closed system using the micro Reaction Sampler is shown in Fig. 2b). The monomers of nylon 6,6 were present as methylated products.

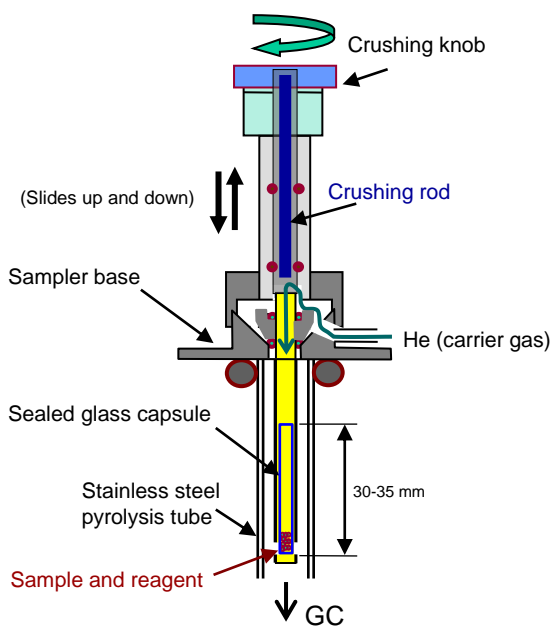


Fig. 1 Diagram of On-line micro Reaction Sampler.\*

\* Patent pending

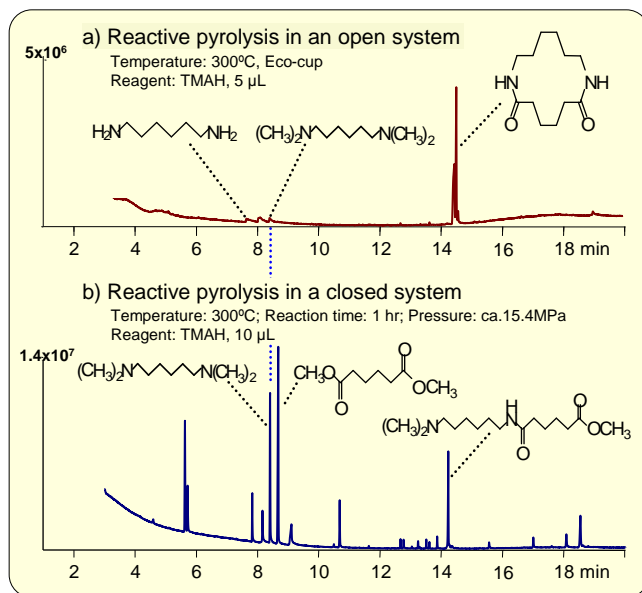


Fig. 2 Comparison of open and closed systems using reactive pyrolysis for nylon 6,6.

**Keywords :** On-line micro Reaction Sampler, Reactive pyrolysis, TMAH, Condensation polymers, Nylon 6,6

**Products used :** Multi-functional pyrolyzer, On-line Micro Reaction Sampler, Vent-free GC/MS adapter

**Applications :** General polymer analysis, composition analysis, Biomass

**Related technical notes :** PYA2-030E

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