

NEW

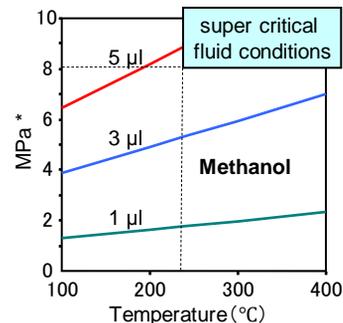
Leading the way in material characterization

“On-line micro reaction sampler” (P/N: PY1-1050)

The on-line micro reaction sampler is used in conjunction with the Multi-Shot pyrolyzer EGA/PY-3030D. Milligram quantities of the sample and reagent are placed in a glass capsule (id. 2 mm, length 25-30 mm) which is then flame-sealed. The sealed capsule is placed in the deactivated, stainless steel reaction chamber and attached to the sampler. The reaction chamber is lowered into the furnace. After the high pressure, high temperature reaction, the capsule is shattered and the reaction products are introduced on-line into a GC separation column.

The figure on the right shows the calculated relationship between the pressure and temperature inside of the glass capsule, when 1, 3 and 5 μL of methanol are added to the glass capsule (volume: 60 μL). Methanol is a supercritical fluid above the critical temperature and critical pressure ($T = 239^\circ\text{C}$, $P = 8.1 \text{ MPa}$). A supercritical fluid has unique properties which will affect the chemical reactions occurring at elevated temperatures and pressures. Often they are quite different from those obtained under standard conditions.

Temperature and pressure isothermals obtained when different volumes of methanol are used.



*MPa = 10atm

Typical applications of the micro reaction sampler:

Example 1: Polyamide (e.g. nylon) is one of the polymers that is difficult to analyze using ordinary reactive pyrolysis GC or Py-GC. However, performing the analysis at elevated pressures significantly enhances the organic alkali hydrolysis and derivatization process.

Example 2: Methyl esterification of vegetable oils and animal fats which are the common sources of biodiesel fuel can be studied using the micro reaction sampler.

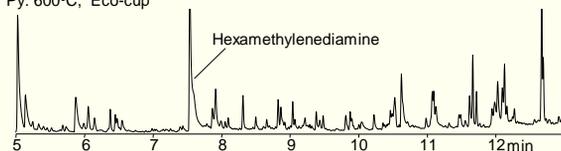
Reactive pyrolysis of nylon 6.6

The figure below shows the chromatograms of nylon 6.6 obtained by 1) flash pyrolysis, 2) conventional reactive pyrolysis, and 3) reactive pyrolysis at elevated pressure. Only the chromatogram obtained at elevated pressure has large peaks for diamine and adipic acid ester which are indicative of the nylon 6.6 structure.

1) Flash pyrolysis

Py: 600°C, Eco-cup

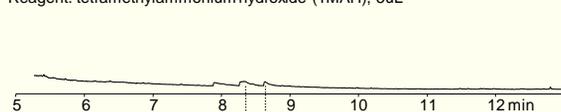
GC: 40→320°C(20°C/min),
Column: UA5-30M-0.25F



2) Conventional reactive pyrolysis

Py: 300°C, Eco-cup

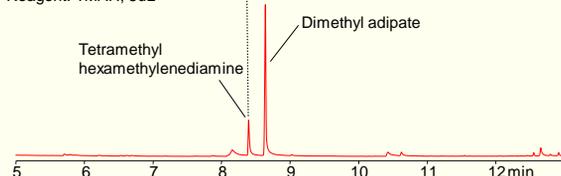
Reagent: tetramethylammonium hydroxide (TMAH), 5 μL



3) Reactive pyrolysis using the micro reaction sampler

Py: 300°C, reaction time: 1hr

Reagent: TMAH, 5 μL



Reactive pyrolysis of fat (butter)

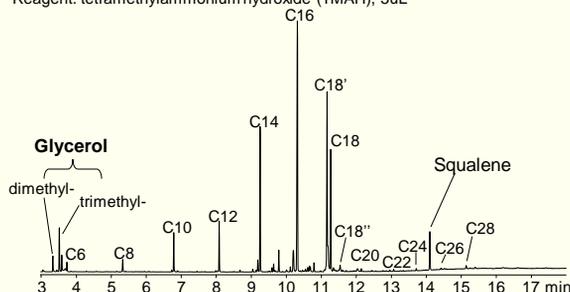
The top chromatogram was obtained using conventional reactive pyrolysis (350°C). The bottom chromatogram was obtained using the micro reaction sampler. While the ratios of the methyl esters of the aliphatic acids are comparable, the ratios of methylated glycerol are quite different.

1) Reactive pyrolysis in open system

Py: 350°C, Eco-cup

Reagent: tetramethylammonium hydroxide (TMAH), 5 μL

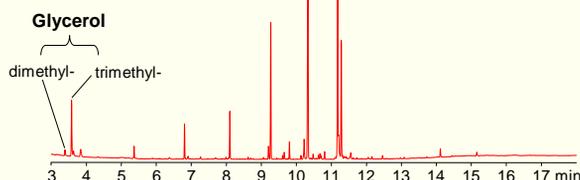
GC: 40→320°C (20°C/min),
Column: UA5-30M-0.25F



2) Reactive pyrolysis using the micro reaction sampler

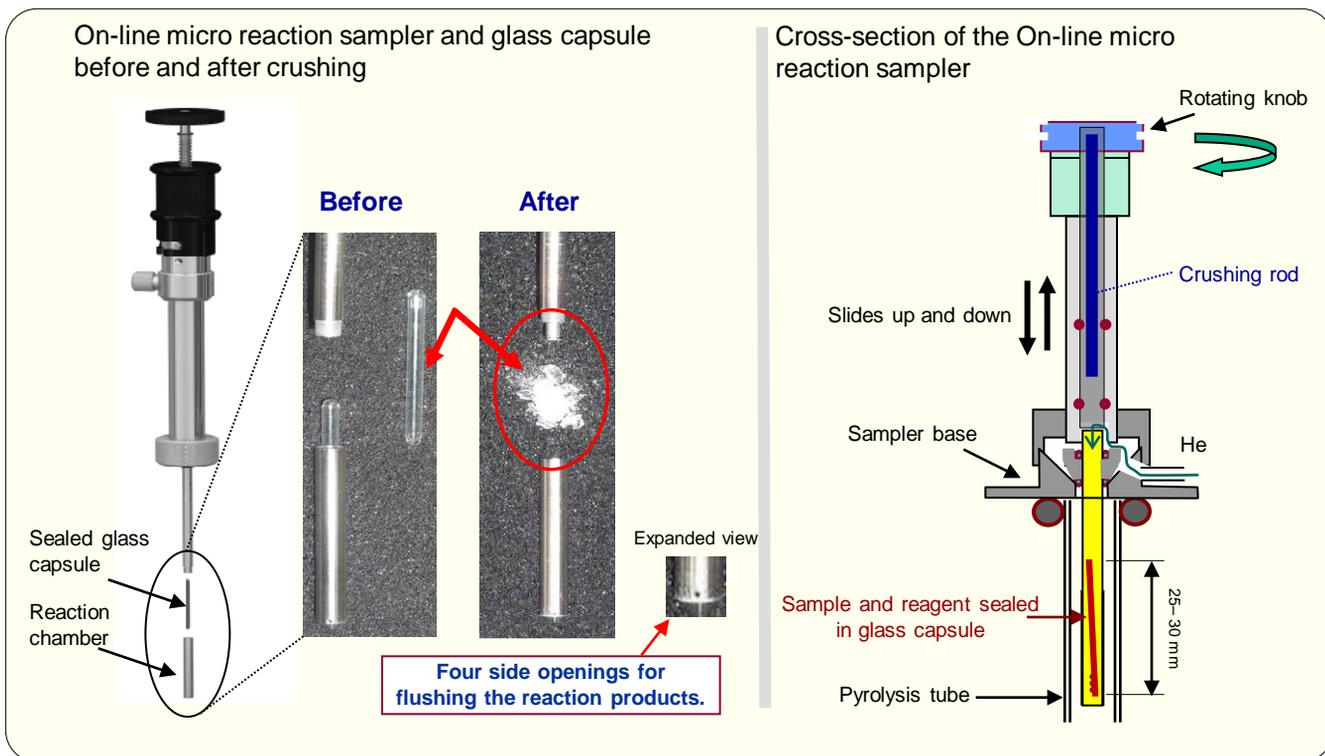
Py: 100°C, reaction time: 1hr

Reagent: TMAH, 5 μL



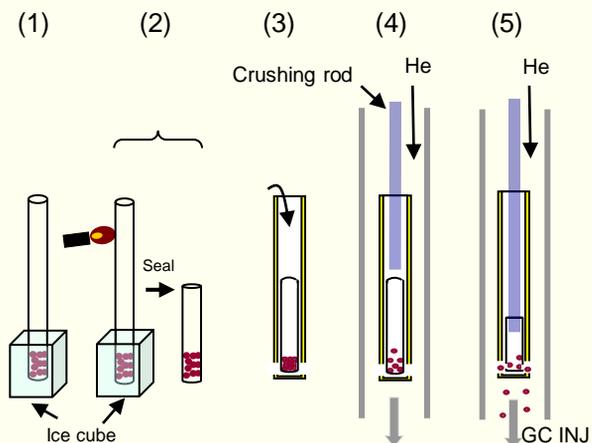
Operating principal

The operating sequence described earlier is shown below:



Using on-line micro reaction sampler:

- (1) Place sample and reagent in a glass capsule.
- (2) Drill a 2.6 mm (bit provided) hole in an ice cube and insert the glass capsule. This cools the sample and reagent. Flame-seal the end of the capsule using a small torch.
- (3) Place the sealed capsule in the reaction chamber and attach it to the sampler.
- (4) Attach the sampler to the pyrolyzer, purge the air from the sampler. When the furnace and GC are READY, PUSH the sample into the furnace. The sample is allowed to react at a given temperature for a given period of time.
- (5) After the reaction is completed, rotate the top knob of the sampler to lower the crushing rod. When the capsule chatters, the reaction products are flushed onto the GC column.



Contents in package

• On-line micro reaction sampler		1 pc
• Standard accessories		
Glass capsule	(PY1-5110)	1 set (50 pcs)
2.6 mm drill bit	(PY1-7115)	1 pc
Sampler stand	(UV1-3802)	1 pc
Reaction chamber (spare)	(PY1-5311)	1 pc



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