

“On-line micro reaction sampler”

The on-line micro reaction sampler is a device that allows a small amount of a sample and reagent to react under semi-supercritical or supercritical fluid conditions in a small glass capsule (id 2 mm, length 30-35 mm), and is fitted to a Multi-Functional Pyrolyzer, then reaction products formed are analyzed online directly by GC.

Features

• Reactions under high pressure and high temperature in a closed system

Polyamides that can hardly be analyzed by normal reactive pyrolysis GC or Py-GC can be analyzed by organic alkali derivatization under high pressures and high temperatures.

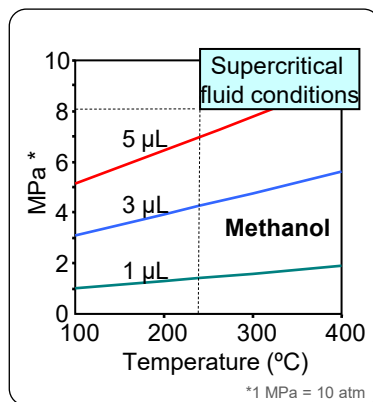
• Online GC analysis of reaction products

After reaction in a sealed glass capsule, the products can be analyzed online directly by introducing them to a GC separation column without working up the reaction mixture.

• High pressure reactions using a small amount of sample and reagent

Only a few milligrams of sample and reagent are required for the online micro reaction sampler; therefore, it is safe to handle.

The figure on the right shows the calculated relationship between the pressure and temperature inside of a glass capsule, when 1, 3 and 5 μL of methanol are added to the glass capsule (volume: 76 μL). Methanol becomes 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 and often gives quite different results from those of normal conditions.



Temperature and pressure isothermals with varied volumes of methanol

Typical applications of the micro reaction sampler

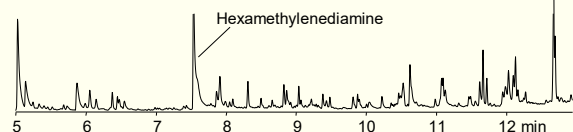
Reactive pyrolysis of nylon 6.6

Polyamide (e.g. nylon) is one of the polymers that are 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.

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 3) has large peaks which are indicative of the nylon 6.6 structure.

1) Flash pyrolysis GC: 40→320°C (20 °C/min)
Py: 600°C, Eco-Cup Column: UA5-30M-0.25F

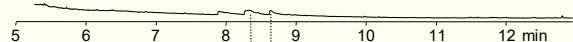
Py: 600°C, Eco-Cup



2) Conventional reactive pyrolysis

Py: 300°C, Eco-cup

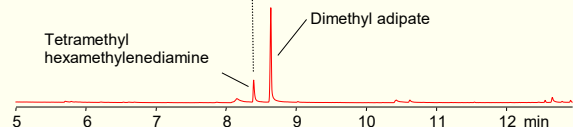
Reagent: tetramethylammonium hydroxide (TMAH), 5 μL



3) Reactive pyrolysis using the micro reaction sampler

Py: 300°C, reaction time: 1 hr

Reagent: TMAH, 5 μL



Reactive pyrolysis of fat (butter)

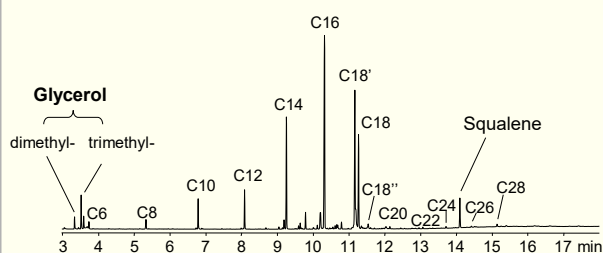
Methyl esterification of vegetable oils and animal fats which are the common sources of biodiesel fuel can be studied using the micro reaction sampler.

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 GC: 40→320°C (20 °C/min)

Py: 350°C, Eco-Cup

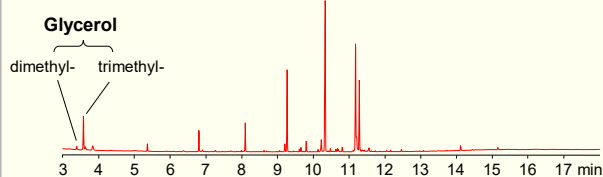
Reagent: tetramethylammonium hydroxide (TMAH), 5 μL



2) Reactive pyrolysis using the micro reaction sampler

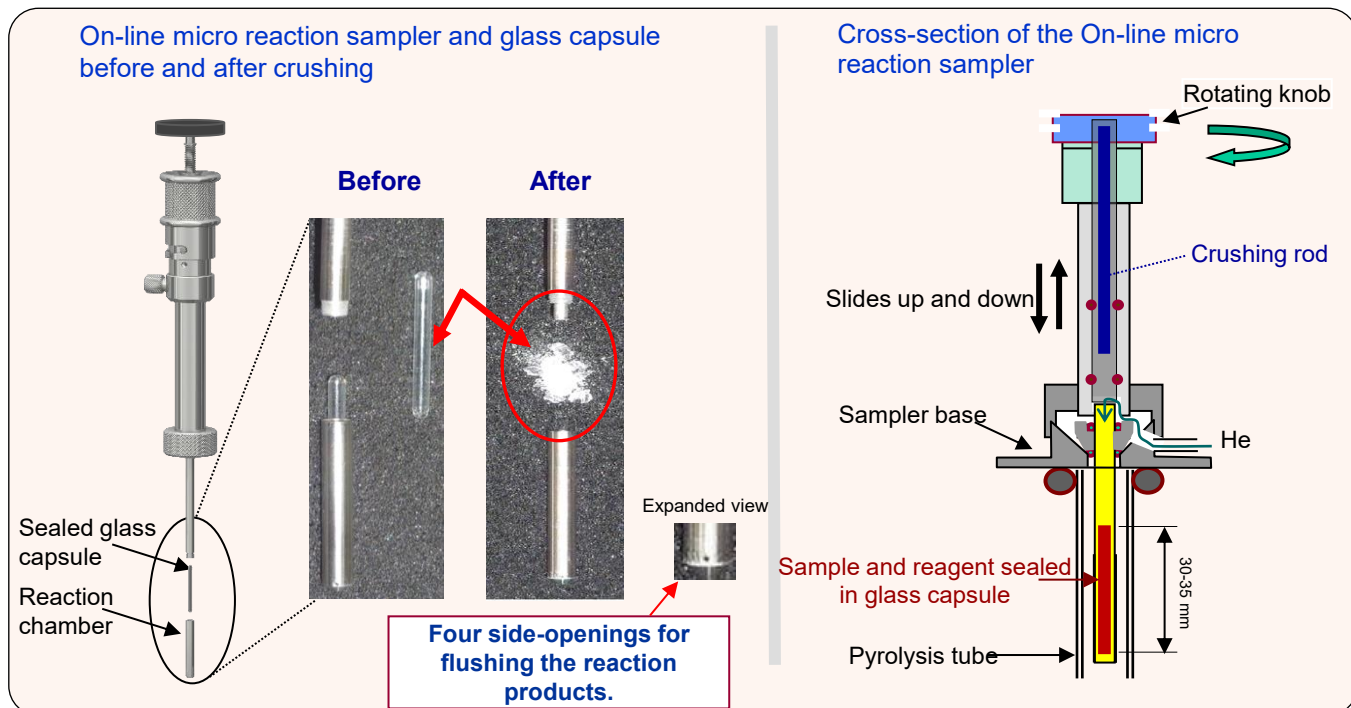
Py: 100°C, reaction time: 1 hr

Reagent: TMAH, 5 μL



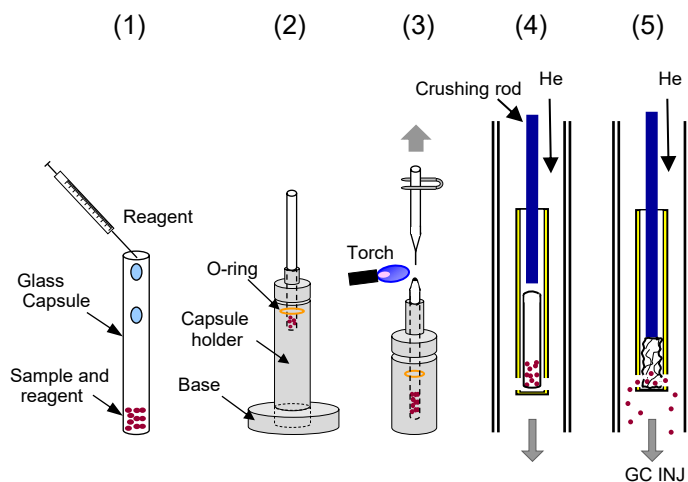
Operating principal

The operating sequence described earlier is shown below:



Using on-line micro reaction sampler

- (1) Place sample and reagent in a glass capsule.
- (2) Insert the glass capsule into the capsule holder until it hits the bottom of the hole. The capsule holder and the base must be chilled in a freezer in advance.
- (3) Flame-seal the top end of the capsule using a small torch as in the figure on the right.
- (4) Place the sealed capsule in the reaction chamber and attach it to the sampler. Attach the sampler to the Multi-Functional pyrolyzer, purge the air from the sampler. When the furnace and GC are READY, push the sampler down into the furnace. The sample is allowed to react at a given temperature for a given period of time. Alternatively, batch treatment can be done outside of the sampler such as in an oven.
- (5) After the reaction is completed, rotate the top knob to lower the crushing rod. When the capsule shatters, the reaction products are flushed onto the GC column.



Specifications

Compatible pyrolyzer : EGA/PY-3030D, PY-3030S
 Glass capsule : od. 2.5 mm, length 25~34 mm
 Max use temperature : 400°C
 Max reagent volume : 10 µL

Contents in package	Qty	P/N
• On-line Micro Reaction Sampler	1 pc	PY1-1050
• Standard accessories		
• Glass capsule sealing assembly (incl. O-ring S3)	1 pc	PY1-5315
• Glass capsule B	3 pc	PY1-5319
• Sampler stand	1 set (40 pcs)	PY1-5113
• Reaction chamber (spare)	1 pc	UV1-3802
• O-ring S4 (spare)1	1 pc	PY1-5311
• SUS pyrolysis tube 3030	5 pcs	UV1-7701
	2 pc	PY1-3321

* On-line Micro Reaction Sampler (P/N: PY1-1050) contains all the items listed above. The quantities of each parts may differ from the package when ordering individually.