FRONTIER LAB

Rapid Catalyst Screening Reactors

Rapid screening of catalysts

Analysis of a variety of sample types

Multi-modes of operation

Tandem μ-Reactor
Rx-3050TR

Single μ-Reactor
Rx-3050SR

Overview

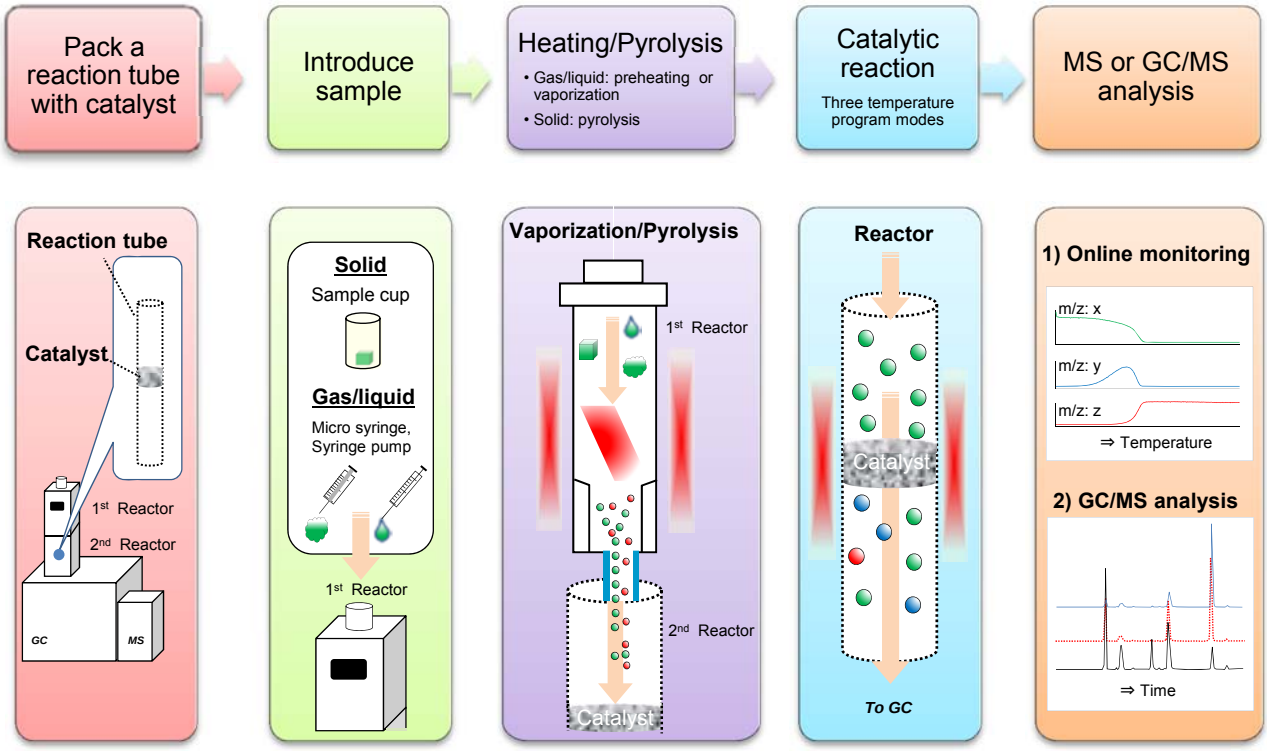
Two types of rapid catalyst screening reactors (Tandem μ-Reactor and Single μ-Reactor) have been developed to facilitate the rapid characterization of catalysts. These μ-Reactors are easily interfaced to a gas chromatography-mass spectroscopy (GC/MS) system, allowing real-time monitoring of the chemical species generated by vapor phase contact reaction with a catalyst. The catalyst is packed into a quick-change catalyst reaction tube prior to testing.

Two accessories, the Selective Sampler and the MicroJet Cryo-Trap, enable the products formed in up to eight thermal zones, to be automatically analyzed. Both reactors are designed to heat and cool rapidly which increase the number of catalysts that can be characterized in a given period of time. Three different reaction gases can be connected to the system so that each catalyst can be evaluated under a variety of reaction conditions.

Rapid catalyst evaluation using a mass spectrometer (MS) as a detector

In the Tandem μ-Reactor, two reactors (upper and lower), are individually temperature-controlled. The upper reactor (1st Reactor) is used to preheat a gas, vaporize a liquid, or thermally decompose organic solids to generate gases. The catalyst reaction tube is packed with a catalyst and placed in the lower reactor (2nd Reactor).

Volatiles released from the heated or thermally decomposed sample stream flow into the catalyst reaction tube in the lower reactor via carrier gas flow, where they react with the catalyst. Then the products formed flow into the GC for analysis. The catalyst is evaluated by noting what compounds are found and their relative distribution using MS detection.



Rapid catalyst evaluation system

Features of the Rapid Catalyst Screening Reactors

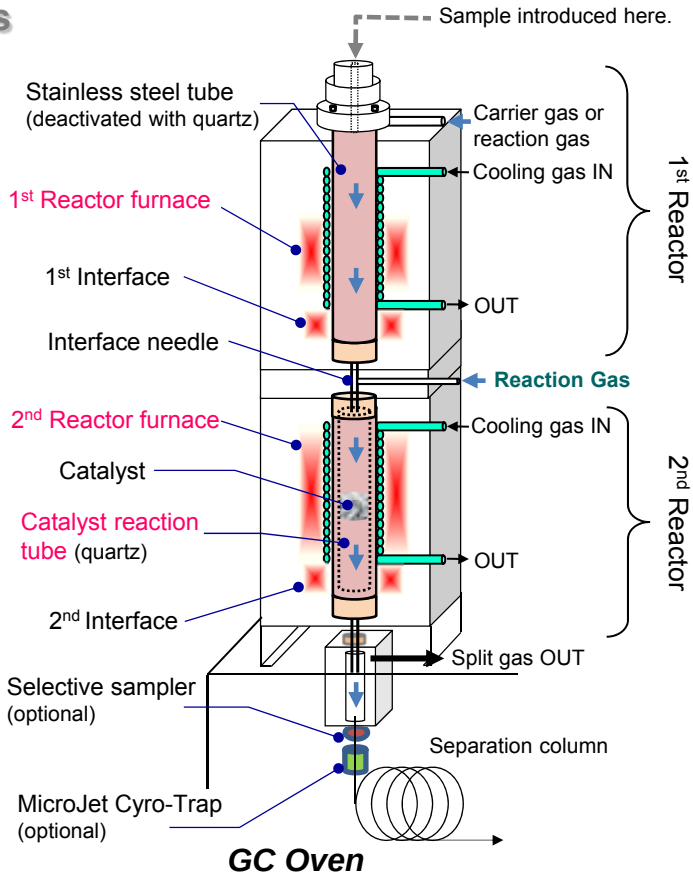
1. Internal design of the reactors

Tandem µ-Reactor

Tandem µ-Reactor consists of two reactors connected in series. The 1st Reactor is used to preheat a gaseous sample, vaporize a liquid sample, or pyrolyze a solid sample. The 1st Reactor operates at a constant temperature. The volatiles exit the 1st Reactor and flow to the 2nd Reactor. A dedicated reaction temperature controller controls the heating and cooling of the reactor. It also has a switching valve and flow controller so that one of three reaction gases can be introduced during the testing.

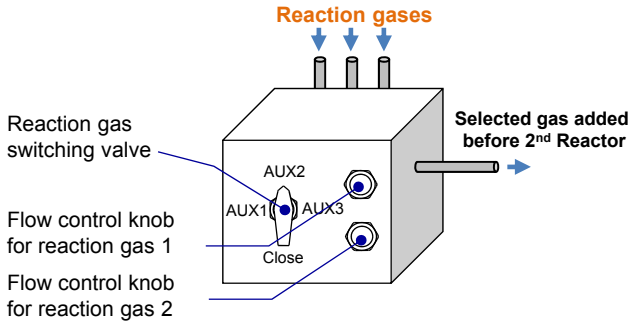
The 2nd Reactor has a quick-change catalyst reaction tube. The temperature of the reactor can be programmed through a variety of heating modes such as isothermal, linear, and stepwise temperature programs.

Parameters such as reactor temperatures, interface (ITF) temperatures, time, and settings for other accessory devices are set through the operating software on the PC. Method set points and real-time actual temperatures and times are also displayed.



Flow control of reaction gas

Three different gases can be connected to the reactor controller. The flow rate of each gas is controlled by a separate mass flow controller. The reaction gas switching valve is used to select the reaction gas that flows to the reactor. (Note: the flow control knob for reaction gas 3 is located on the rear of the Tandem 1st Reactor controller.)



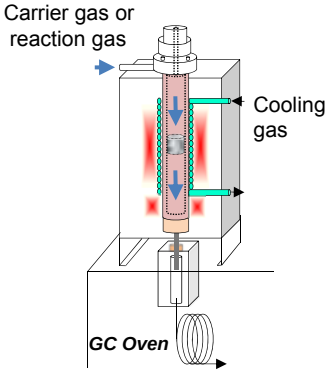
Tandem 1st Reactor controller

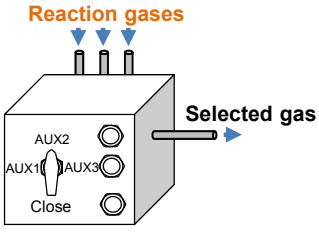
Single µ-Reactor

The flow control system for the Single µ-Reactor is identical to that of the Tandem µ-reactor. The controller has three adjustable gas flow valves and a stream selection valve.

Heating and analysis modes of the reactor are also the same as those of Tandem µ-reactor. Isothermal, linear, and stepwise temperature programs are available through the software.

Only liquids or gases can be introduced to the Single µ-Reactor. Solids cannot be analyzed with the Single µ-Reactor.

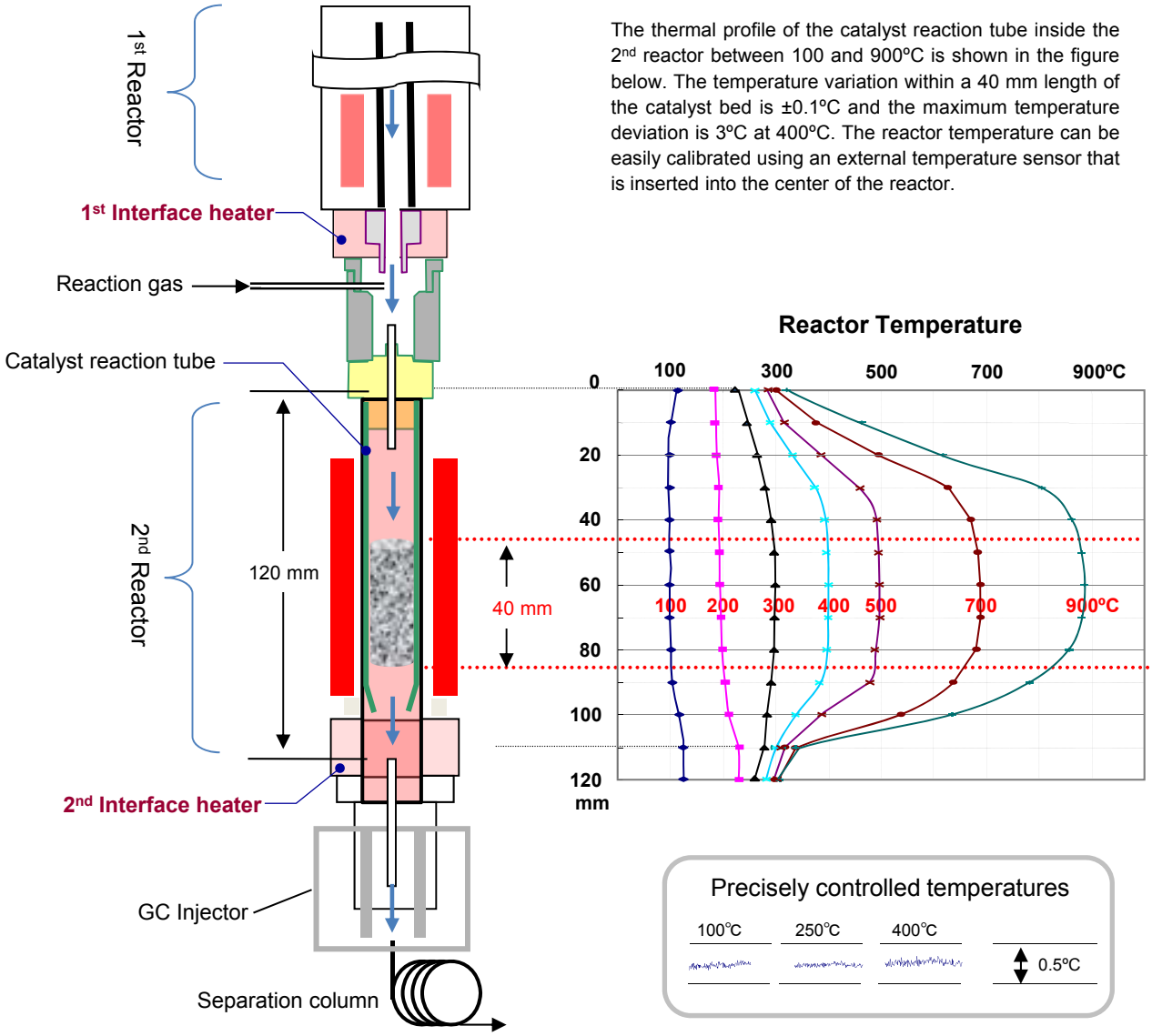




Reactor flow controller

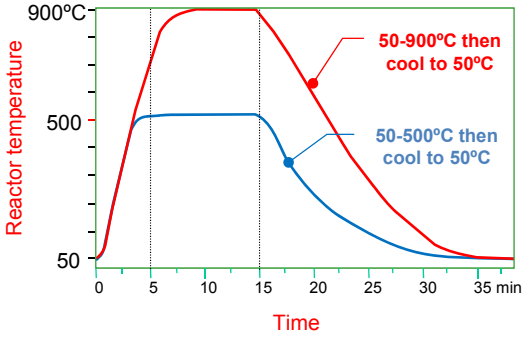
Features of the Rapid Catalyst Screening Reactor

2. Highly precise temperature control for minimal reactor temperature fluctuations



The thermal profile of the catalyst reaction tube inside the 2nd reactor between 100 and 900ºC is shown in the figure below. The temperature variation within a 40 mm length of the catalyst bed is ±0.1ºC and the maximum temperature deviation is 3ºC at 400ºC. The reactor temperature can be easily calibrated using an external temperature sensor that is inserted into the center of the reactor.

3. Rapid heating and cooling



The temperature profile shown on the left is obtained by rapid heating the reactor from 50 to 500ºC (blue trace) and also from 50 to 900ºC (red trace). After a 10 minute hold at the final temperature, the furnace was cooled. Note that it took about 5 minutes to cool from 900ºC to 500ºC. The time required to cool down to 50ºC is about 15-20 minutes depending on whether the reactor was set to 500ºC or 900ºC. This rapid heating and cooling feature makes catalyst reaction tube change-over simple, quick and easy after the each catalyst has been evaluated.

4. Exchanging the catalyst reaction tube

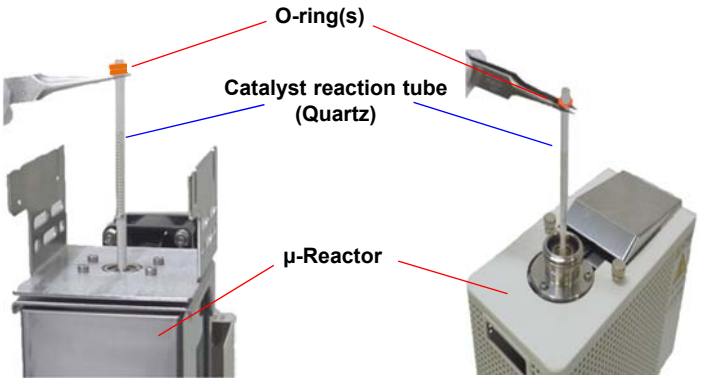
Quick-change catalyst reaction tubes

For the Tandem µ-Reactor, remove the 1st Reactor. The reaction tube (located in the 2nd Reactor) can then be easily replaced by lifting the tube from the top of the reactor.

When using the Single µ-Reactor, simply unscrew the liquid sampler, and lift the reaction tube as shown in the figure on the far right.

Liquid Sampler
(syringe injection)

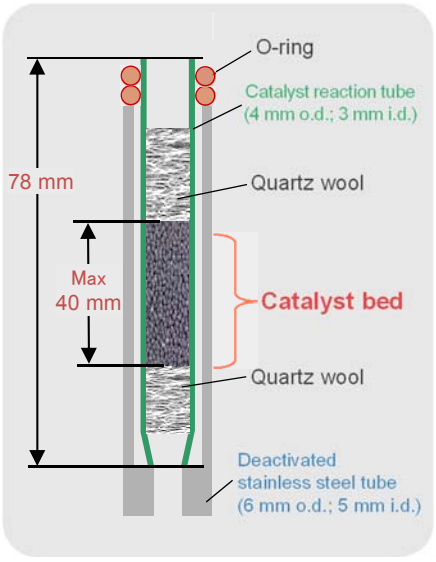
Tandem µ-Reactor **Single µ-Reactor**



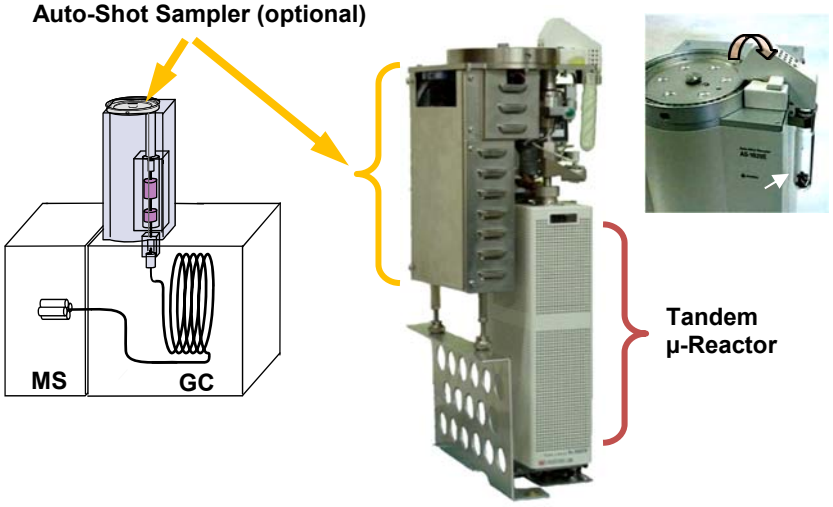
Packing the catalyst reaction tube

The catalyst reaction tube (4 mm o.d.; 3 mm i.d.) is packed with a catalyst. Small amounts of quartz wool are placed both at the bottom and top of the catalyst bed to keep it in position. The figure on the right shows the packed catalyst reaction tube inserted into the deactivated stainless steel tube.

The useable vertical length of the catalyst bed is 40 mm. Catalysts with the particle size of 20 to 60 mesh are used. Normally the catalyst reaction tubes are used, however if a larger volume of catalyst is required for testing, the deactivated stainless steel reaction tube can also be packed.



Auto-Shot Sampler (optional)



The analysis of solid samples can be automated using the Auto-Shot Sampler. The figure on the left (center) shows the Auto-Shot Sampler, with the housing cover removed, installed on the Tandem µ-Reactor.

The Auto-Shot Sampler carousel holds up to 48 sample cups (Eco-cups). After analysis the sample cups are ejected into the glass receptacle (indicated by the white arrow).

Control software

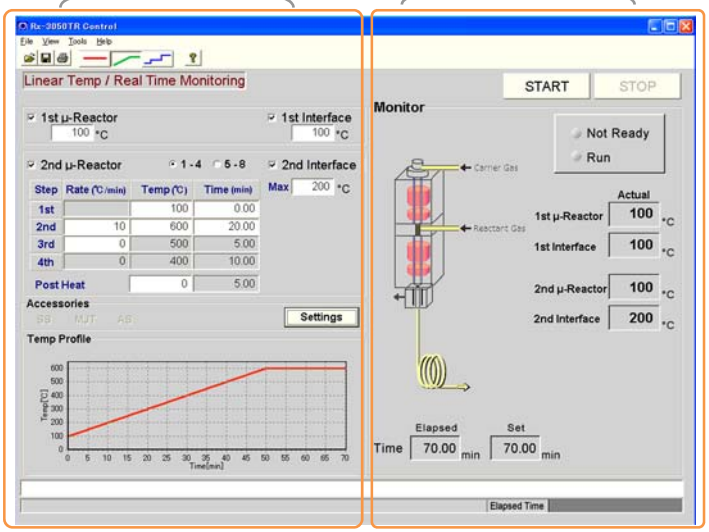
Setting analytical conditions

The left side of the control software screen is used to set up the temperatures, rates and times for the reactors, interfaces and accessory devices such as the MicroJet Cryo-Trap (MJT) and Selective Sampler (SS).

The right side of the control software screen displays operation status such as set points, elapsed time and actual temperatures real-time.

Note the colored graphical icons at the top of these screens. Simply clicking on these toggles the screen view for each of three temperature analysis modes.

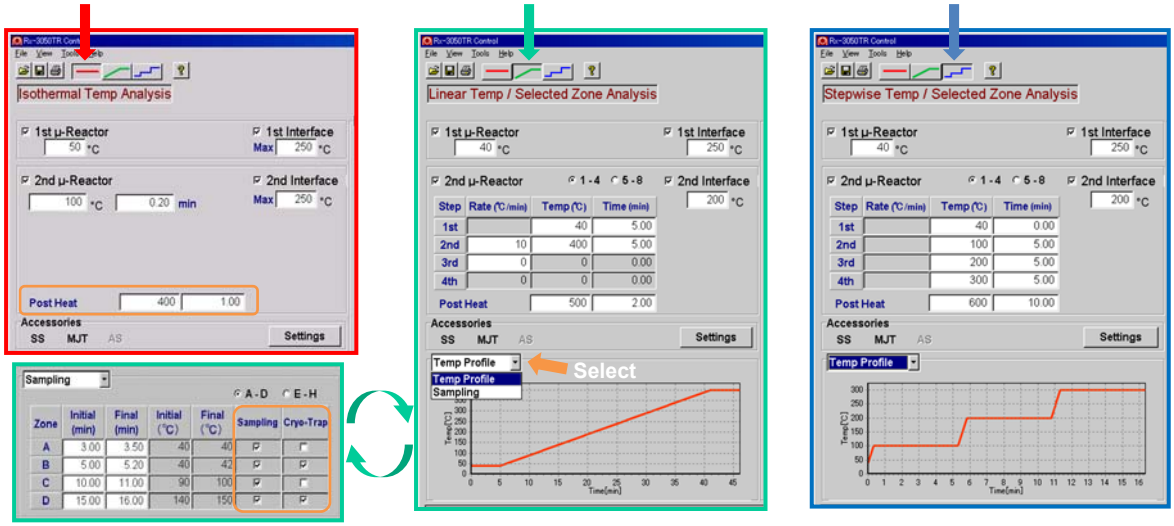
Reactor temp. settings　　Operation status



Three analysis modes

The Tandem and Single μ-Reactors can be used in three different modes. These modes differ in how the reactor temperature is controlled. Isothermal mode **1** has a "Post Heat" function to thermally desorb reaction products in the micro-pores of various types of catalysts. In modes **2** and **3**, the furnace temperature can be programmed in either a linear or stepwise progression, up to a maximum of 8 steps. When used with both the optional Selective Sampler and MicroJet Cryo-Trap, a maximum of 8 temperature zones can be automatically and individually analyzed.

1. Isothermal temp. analysis　　2. Linear temp. analysis　　3. Stepwise temp. analysis



By checking (v) the appropriate “Sampling” and “Cryo-Trap” boxes, the volatiles in the selected zones flow into the separation column. The Cryo-Trap is activated if desired.

Switching analysis modes

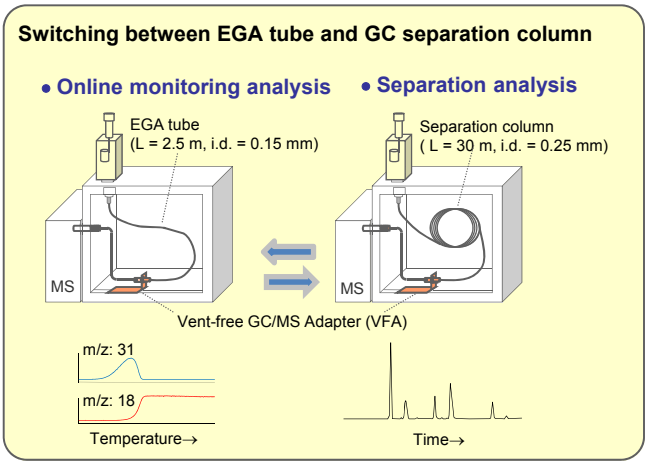
To switch from real-time monitoring, which uses an EGA tube, to GC/MS analysis using a GC separation column is a simple process:

(1) Switch to the desired analysis mode in the control software,

(2) Replace the EGA tube with the GC separation column.

The Vent-free GC/MS Adapter (VFA), included in the standard kit, facilitates switching a GC separation column and an EGA tube without venting the MS system. This switchover takes about 10 minutes.

Switching between EGA tube and GC separation column

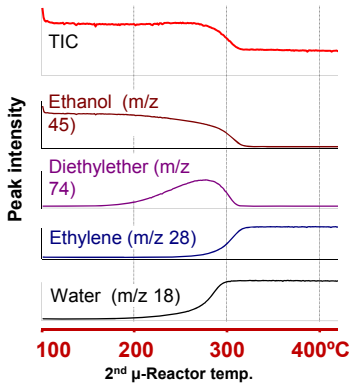


Application 1: Catalytic conversion of ethanol to ethylene

Online – MS analysis

" Linear temp. mode "

1st μ-Reactor: 100ºC,
2nd μ-Reactor: 100-400ºC (20 ºC/min)
Catalyst: H-ZSM-5

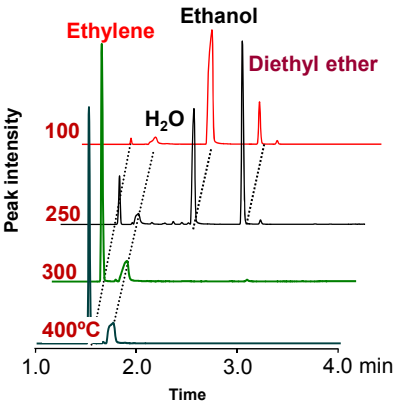


Catalytic reaction products were monitored as the reaction temperature was raised at a constant rate. The amount of ethanol sharply dropped when the temperature reached 280ºC, while the amount of diethyl ether increased. Also, the formation of ethylene and water is observed.

Separation analysis

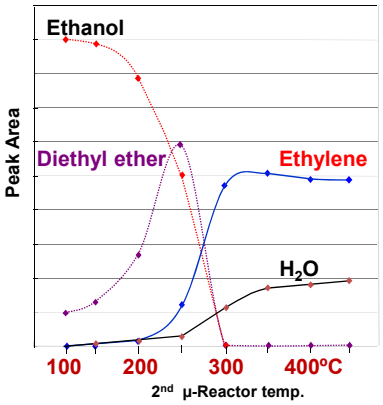
" Stepwise temp. mode "

1st μ-Reactor: 100ºC,
2nd μ-Reactor: 100, 250, 300 & 400ºC
Catalyst: H-ZSM-5



Based on the online-MS analysis results, the volatiles released from each temperature zone were introduced to a separation column and analyzed. As the reactor temperature was raised, ethylene and water were formed while the amount of ethanol formed dramatically decreased.

Reaction temp. vs. Peak area



These are the plots of the peak areas obtained from the chromatogram on the left as the reactor temperature incrases. It clearly shows how the function of the ZSM-5 catalyst various with the temperature of the reactor.

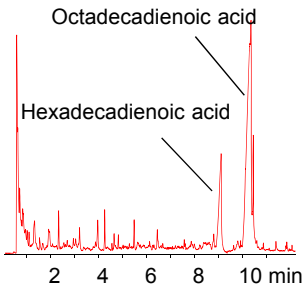
Application 2: Conversion of Jatropha "press cake" to bio-based chemicals

Courtesy: Dr. Murata of AIST Japan

1st μ-Reactor & 2nd μ-Reactor: 550ºC, Catalyst: Zeolite

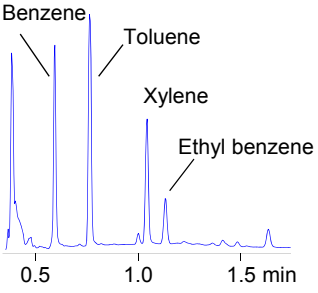
Without Catalyst

The constituents obtained by flash pyrolysis of a finely powdered Jatropha press cake were primarily two fatty acids (C16 and C18) which have two double bonds in their structure.



With Catalyst

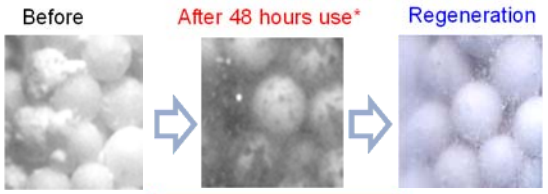
When the 2nd μ-Reactor contains a packed bed of a Zeolite catalyst, Jatropha press cake is converted to monocyclic aromatics such as benzene, toluene, xylene and ethyl benzene.



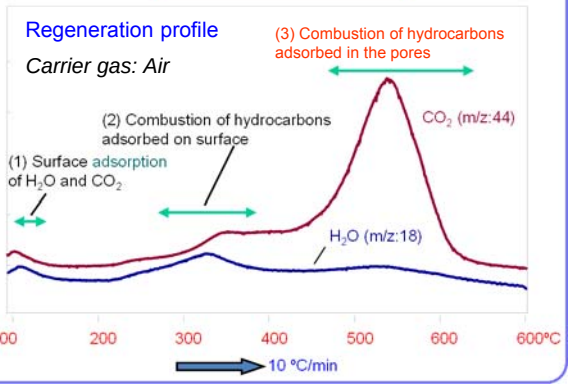
Application 3: Study of catalyst regeneration

The reactor is heated from 100 to 600ºC in a linear mode in an air atmosphere.

Catalyst: 20 % H-ZSM-5, (SiO_2/Al_2O_3 = 150) on Al_2O_3 (20/30 mesh)

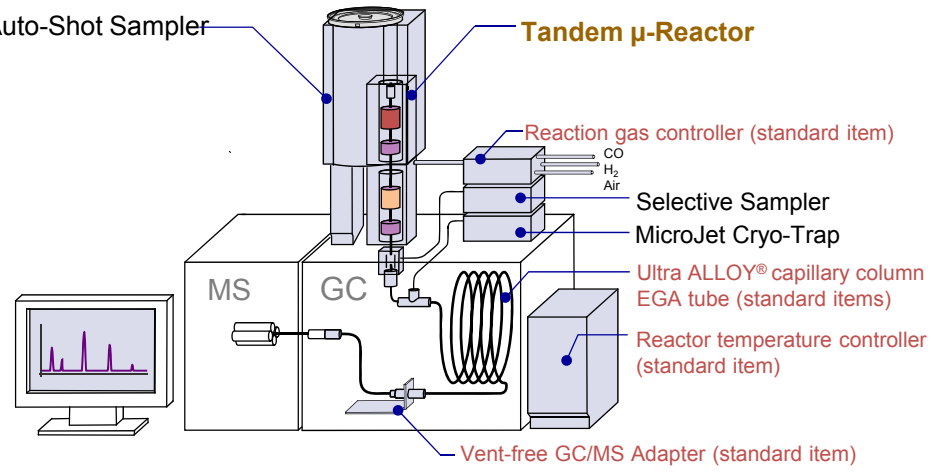


*Sample: 3 % ethanol in He
Flow rate: 50 mL/min
2nd Reactor temp.: 550ºC



Specifications

		Tandem μ-Reactor Rx-3050TR	Single μ-Reactor Rx-3050SR
1st Reactor			-
Temperature control range		40 to 900ºC (1ºC steps); cooling gas	-
Heater		Cylindrical ceramic heater (400 W)	-
Flow path material		Stainless steel (surface deactivated with a bonded silica thin film).	Same as left specification
Interface (ITF) temperature range		40 to 400ºC (1ºC step, constant temp. control)	-
Reaction gas control		Three gas lines manual valve switching, Gas flow rate control (Max 200 ml/min, 1 MPa)	Same as left specification
2nd Reactor			**Single μ-Reactor**
Temperature control range		40 to 900ºC (1ºC steps); cooling gas	Same as left specification
Heater		Cartridge heater	Same as left specification
Reaction tube		Catalyst reaction tube (quartz) : 3 mm i.d.; 4 mm o.d.; Length: 78 mm	Same as left specification
Interface (ITF) temperature		40 to 400ºC (1ºC steps; constant temp. control)	Same as left specification
Control software			
System requirement		PC (2 USB ports and a CD drive), Compatible OS (Microsoft Windows 8.1, 8, 7, Vista, XP)	Same as left specification
Others			
Analytical modes	Temperature control	Isothermal, Linear, and Stepwise Temperature Analyses	Same as left specification
	Sampling	Selectively introduces a maximum of 8 zones from an EGA thermogram onto a GC separation column. Requires Selective Sampler and MicroJet Cryo-Trap	Same as left specification
Pre-requirements		1. GC/MS with Split/Splitless injector 2. Compressed gas for cooling (air or nitrogen)	Same as left specification
Power requirement		100 – 120 VAC or 200 – 240 VAC, 50/60 Hz, Max 800 W	100 – 120 VAC or 200 – 240 VAC, 50/60 Hz, Max 450 W
Dimension (W x D x H) / (kg) 1st Reactor 2nd Reactor Temperature control unit Reaction gas control unit		 76 x 125 x 260 mm / 1.6 kg 76 x 125 x 90 mm / 1.7 kg 120 x 310 x 310 mm / 4.0 kg 160 x 150 x 280 mm / 6.1 kg	 - 76 x 125 x 260 mm / 1.6 kg 120 x 310 x 310 mm / 4.0 kg 160 x 150 x 280 mm / 6.1 kg
Standard items		Vent-free GC/MS Adapter, EGA tube for online analysis, Ultra ALLOY® capillary column, Catalytic tube (Packed with ZSM-5), Reaction gas controller, Reaction temp. controller etc.	



Rapid Screening Reactor（RSR-GC/MS）System

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