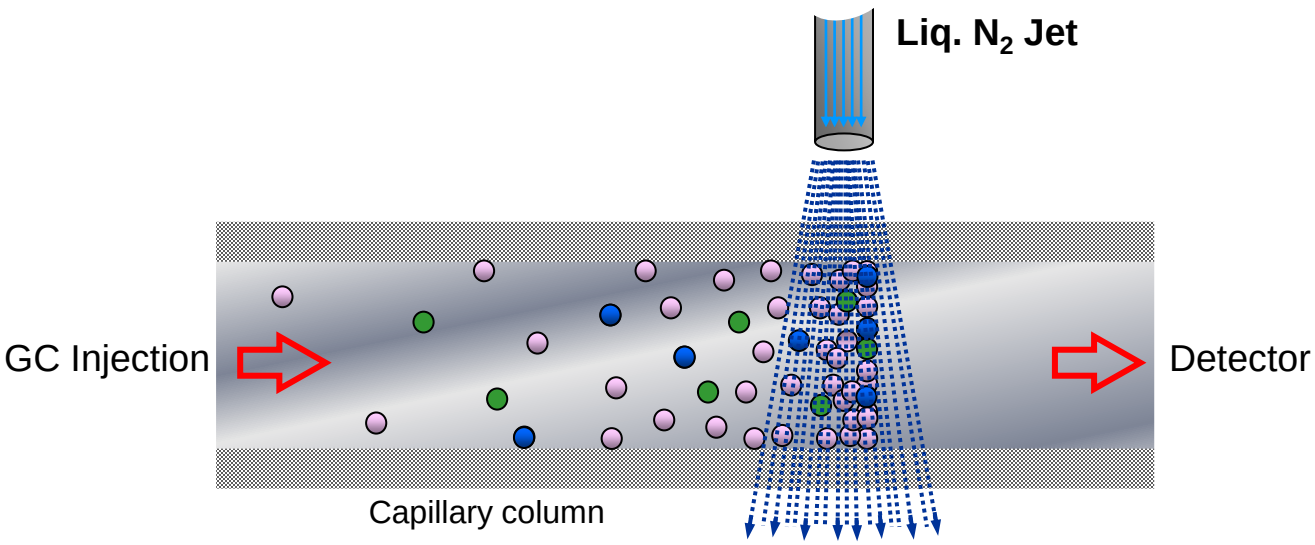
FRONTIER LAB

A cryo-trapping temperature at -196 ˚C in GC
Compact and energy saving design with safety features

MicroJet Cryo-Trap
MJT-2030E



MJT-2030E flow controller

Background of development

The thermal desorption analysis and headspace analysis require a considerably long time for sample introduction. High-resolution separation analysis of low boiling point compounds can be easily achieved by cryo-trapping the compounds with a narrow bandwidth, followed by rapid thermal desorption. For this purpose, the MicroJet Cryo-Trap (MJT-1030Ex), a cryo-trapping system for pyrolysis GC analysis, has been supplied to the market over ten years. The new model, the MicroJet Cryo-Trap (MJT-2030E), has the same fundamental performance as the previous model, yet with much improved ease of use.

Three features

1) Energy efficient rapid cooling and instant thermal desorption

A stable cooling temperature of -196 ºC is achieved within 2 minutes from the start of the liquid nitrogen jet flow. After the cooling is terminated, the trapped volatiles are rapidly thermally desorbed by the heat of the GC oven at a rate of 800 ºC/min. The total liquid nitrogen consumption is less than 1/3 of that of competitors' products, thus achieving low energy consumption.

* In the trapping method of low volatiles using adsorbents such as Tenax, an external heater is used for thermal desorption. On the other hand, this method achieves separation of low boiling point compounds by rapid thermal desorption using the heat of the GC oven to prevent abnormal separation of peaks.

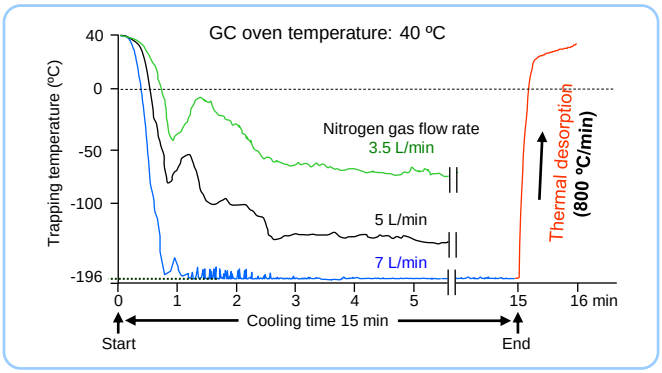


Fig. 1 Cooling rate, temperature stability and thermal desorption

2) Variable cooling temperatures

As shown in Fig.1, by adjusting the nitrogen flow rate, the desired cooling temperature can be controlled.

Trapping low boiling compounds (CO_2, ethane, etc.)

In the operation principle shown in Fig. 4, while a liquid nitrogen jet was blown against the head of a separation column, 5 µL of lighter gas was injected and cryo-trapped at the head of the column for 5 minutes. Then the jet flow was terminated for analysis as shown in Fig. 2. It is seen that low boiling point compounds such as carbon dioxide and ethane are trapped.

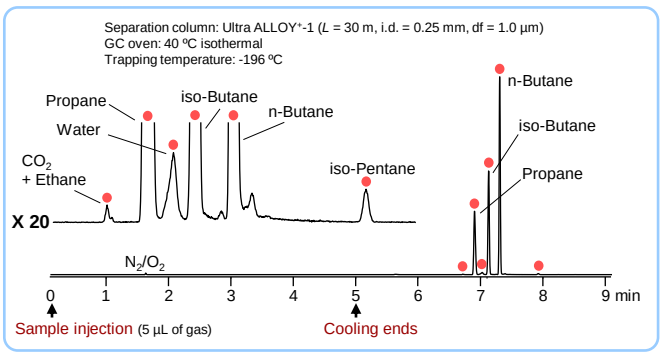


Fig. 2 Trapping performance using lighter gas

3) Automatic heart-cut analysis of gases evolved from a ceramic composite by Multi-Shot Pyrolyzer (EGA/PY-3030D)

An example of automated analysis of temperature zones A to D of the EGA thermogram obtained by continuous heating of a ceramic composite is shown in Fig. 3. In zone A, DBP, a plasticizer, was detected, in zone B, thermal desorption components of Wax were mainly detected, and in zones C and D, pyrolyzates of polybutyl methacrylate and polystyrene were detected, respectively.

The use of this system allows you to automate the manual analysis of zones A through D, which takes about four hours, including the heating of each zone and GC analysis.

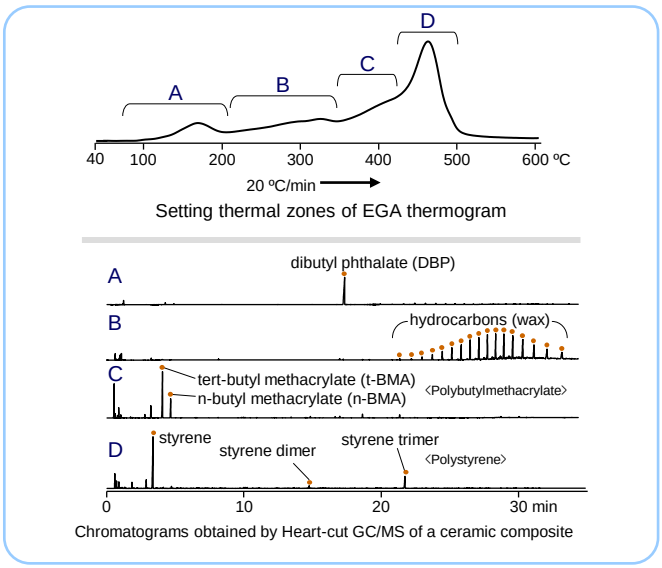


Fig. 3 EGA thermogram of a ceramic composite and automatic analysis of each temperature zone

Operation principle and flow diagram (US patent US6,190,613B1)

In this system, nitrogen gas is liquefied by flowing about 3 to 7 L/min through a heat exchange coil immersed in liquid nitrogen and is blown as a jet flow against the head of the separation column in the GC oven to cool the local area where the analytes are trapped. Then the jet flow is stopped instantly by closing the solenoid valve, and at the same time the circulating heated air in the oven rapidly heats the cooled section of the column at approx. 800 ºC/min for thermal desorption. In addition, the MicroJet tube prevents moisture in the air from icing on the separation column, making it possible to use it in a humid environment.

Also, the end of the liquid nitrogen feed tube has been modified and its U-shaped end will facilitate the column installation.

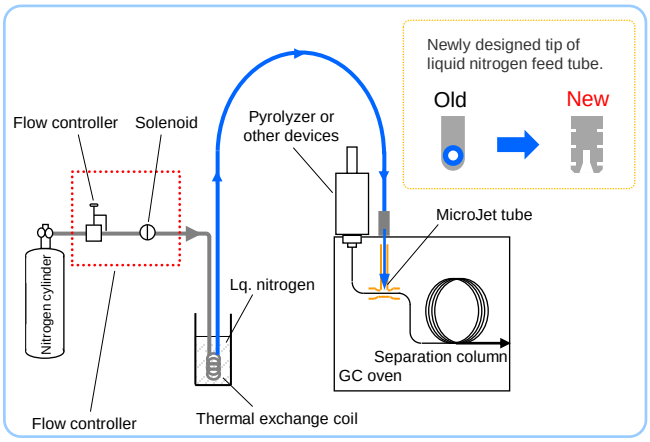


Fig.4 Flow diagram of MJT-2030E

Cryo-trapping at the head of separation column and thermal desorption

By using the anti-icing baffles, nitrogen gas is discharged from both sides of the MicroJet tube to prevent water in the air from icing on the cooling section of the separation column. If icing occurs, it may cause abnormal phenomena such as peak cracking.

Further, by adjusting the nitrogen gas flow rate, the separation column can be cooled to any desired temperature (see Table 1).

Also, the MJT-2030E has a digital display for the nitrogen pressure gauge value, making it easier to adjust the nitrogen gas flow rate.

Table 1. Nitrogen consumption and trapping performance

Approx. cooling temp. (ºC)	-190	-150	-50
N_2 gas flow rate (L/min)	7	5*	3.5
Liq. N_2 consumption (mL/min)	20	15*	10
Compounds that can be trapped	>C4	>C6	>C11

* 1/3 or less than that of competitors.

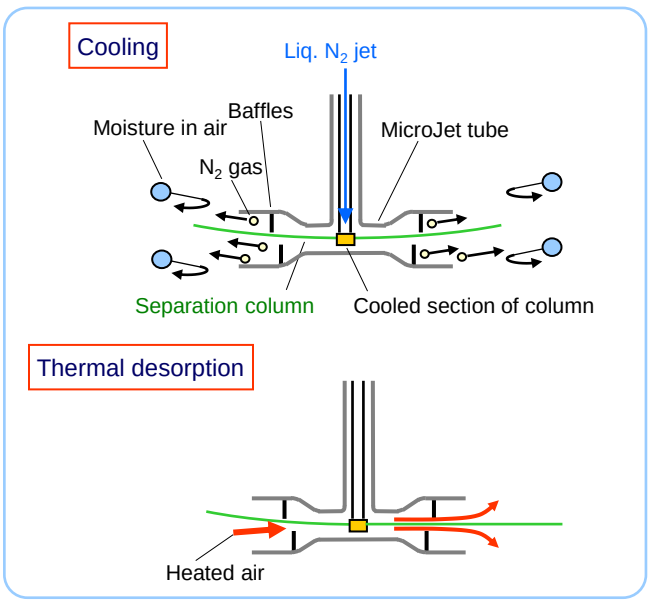
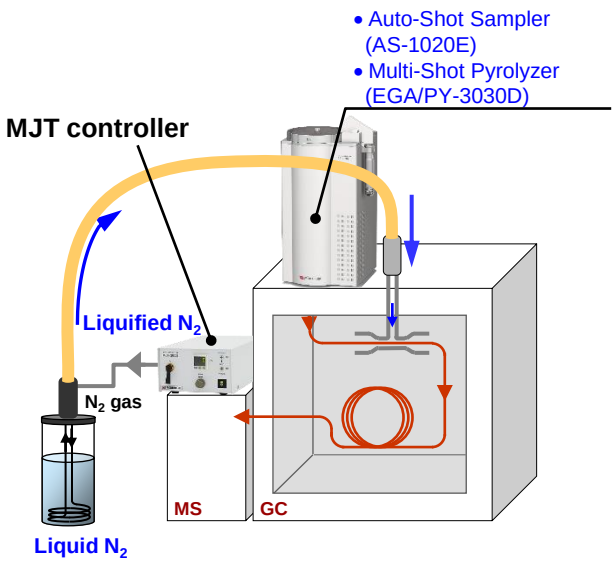
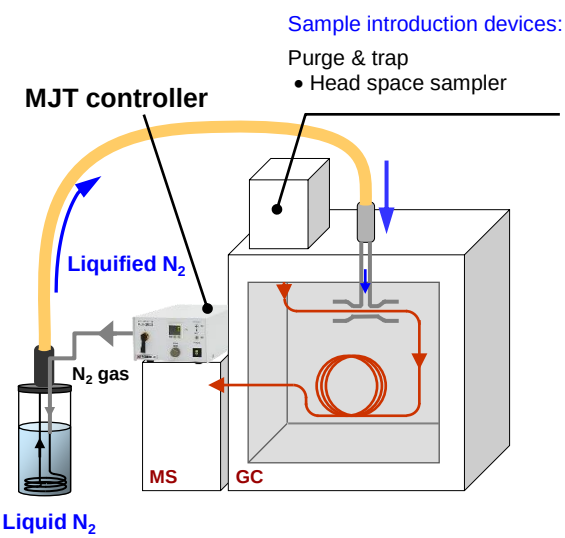


Fig. 5 Gas flow scheme inside MicroJet tube

MJT coupled with a variety of sample introduction devices



Combination with pyrolyzer and GC/MS



Combination with sample introduction devices and GC/MS

Analysis examples using MicroJet Cryo-Trap

Headspace analysis of red wine

A headspace analysis of red wine using MicroJet Cryo-Trap is shown in Fig. 6. A one mL sample from the headspace gas of a red wine bottle was injected directly into the GC injector. The volatiles were cryo-trapped at the head of a separation column before starting GC/MS analysis. Typical compounds found in wine are observed.

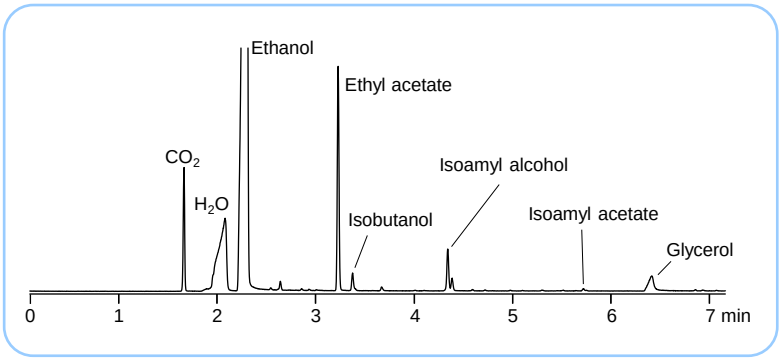


Fig. 6 Headspace analysis of red wine

Analytical conditions

- Cryo-trapping temperature: -196 ºC
- GC oven: 40 – 280 ºC (20 ºC/min, 5 min hold)
- Separation column: UA⁺-1 (*L*=30 m, i.d.=0.25 mm, df=1.0 µm)
- Carrier gas: He, 1 mL/min, split ratio: 1/50

Analysis of biomass plastic bags

An analysis example of a biomass plastic bag is shown in Fig. 7. The unprinted part of the plastic bag was cut out and placed in a sample cup for thermal desorption, and the volatile components generated were cryo-trapped using MicroJet Cryo-Trap. The chromatogram was obtained by GC/MS analysis of the cryo-trapped components. Various additives are detected.

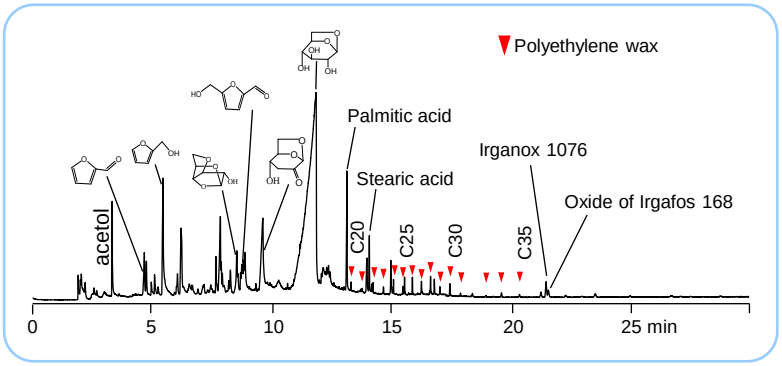


Fig. 7 Analysis of biomass plastic bag

Analytical conditions

- Cryo-trapping temperature: -196 ºC
- GC oven: 40 – 320 ºC (20 ºC/min)
- Separation column: UA⁺-5 (*L*=30 m, i.d.=0.25 mm, df=1.0 µm)
- Carrier gas: He, 1 mL/min, split ratio: 1/10

Specifications

MicroJet Cryo-Trap (MJT-2030E)	
Lowest cooling temp.	Approx. -196ºC (N_2: 7 L/min; GC oven temp.: 40 ºC)
Automatic control / temperature monitoring	EGA/PY-3030D, Rx-3050TR, and Rx-3050SR
Supported separation column	Metal capillary separation column (id. 0.25 mm or smaller), Fused silica (FS) capillary separation column* (id. 0.53 mm or smaller)
Power requirement	100 - 240 VAC, 40 VA (support for a wide range of power voltage)
Supported GC model **	Agilent, Thermo Fisher, Shimadzu, Perkin Elmer, and Scion
Unit and accessory	Flow controller, MicroJet unit, liquid nitrogen container (2 L)
Utility requirements	Liquid nitrogen (minimum volume 2 L): liquid nitrogen consumption at -150 ºC approx. 15 mL/min Nitrogen cylinder (secondary pressure: up to 600 kPa): nitrogen gas consumption approx. 7 L/min
Option	Thermal exchange coil and lid for Chart MVE Lab 30 Dewar (30 L)***

* FS column may break, if it is rapidly cooled or heated.
** May not be installable, if another device is on the top of your GC oven.
*** Customer can purchase a 30 L Dewar from a local supplier.