Ver.1.29

MULTI-SHOT PYROLYZER
MODEL EGA/PY-3030D
OPERATION MANUAL

FRONTIER LABORATORIES LTD.

www.frontier-lab.com

BEFORE USING THIS PYROLYZER

1. Thank you for purchasing Frontier Laboratories' Multi-Shot pyrolyzer. Read through this operation manual carefully before attempting any operation, particularly if you are a first-time user. This manual is designed to assist you with your daily operations.
2. This manual contains categorized descriptions of the installation, operation, maintenance, trouble-shooting, etc of the Multi-Shot pyrolyzer. We recommend that you read this manual along with the operation manual of the gas chromatograph you are using.
3. Consumable parts are described in Appendix-1 and 2. Parts can be purchased from your local sales representative office.

Physical appearance and specifications are subject to modifications without notice.

PRODUCT WARRANTY

Frontier Laboratories Ltd. warrants this product against defects or failures in accordance with the warranty terms and conditions stipulated in a separate sheet. The product warranty can also be downloaded from our website.

ABOUT PRODUCT SUPPORT PERIOD

We will stock consumable and maintenance parts and will accept inspection and repair orders seven years from the date of product sales termination. However, in the event where electronic parts supplied from parts manufacturers are discontinued; thus our stock level becomes too low, we may not be able to provide support even within seven years from the date of product sales termination,.

FOR YOUR SAFETY

To use this product safely and properly, be sure to read the safety precautions and warnings before attempting to operate. If this product is used in a manner not instructed in this manual, the protective functions of this product may not be activated. Frontier Laboratories Ltd. will not be responsible for losses incurred due to the neglect of these precautions and warnings.

Warning symbol and signal words

The following warnings and precautions labels are affixed to this product and this manual. When these precautions are displayed, it indicates that misoperation may cause severe injury to your body and the products

In this manual, the safety instructions are ranked as warning, caution, and caution hot.

WARNING

This hazard signs indicates that incorrect handling may cause hazardous conditions, resulting in death or severe personal injury.

CAUTION

This sign indicates that incorrect handling may cause hazardous conditions resulting in minor or moderate personal injury and physical damage.

CAUTION HOT

It indicates that incorrect handling may cause severe personal burn.

Other important handling information is placed in a frame like this.

WARNING

Risk of electrical shock. Do not remove the temperature controller housing cover, unless you are a service engineer certified by Frontier Laboratories Ltd..

WARNING

If dust is deposited on the power code plug or the cable connectors, clean them thoroughly. Be sure to unplug the power code. Do not use water or organic solvent. Use a dry cloth or brush. There is a risk of fire, if dusty connectors are used.

WARNING

This product uses compressed air or nitrogen for cooling the pyrolyzer furnace. Ensure that you have a good ventilation if nitrogen gas is used. There is a risk of suffocation, if it is used in a confined area with poor ventilation.

CAUTION

When flammable solvents such as benzene, toluene, or acetone are used for your sample, work in a well-ventilated area such as in a hood and area where no open flame is used nearby to avoid catching fire.

CAUTION

The interface needle of the pyrolyzer has a very sharp point. When replacing the interface needle, be sure to wear protective gears such as gloves, safety goggles, etc.

CAUTION

The pyrolyzer uses a quartz pyrolysis tube. If this tube is broken, broken pieces have very sharp edges. When replacing the tube, be sure to wear protective gears such as gloves, safety goggles, etc.

CAUTION HOT

There is a risk of severe burn. Always ensure that the protective housing cover is placed properly on the pyrolyzer. When removing the cover from the pyrolyzer, make sure that the furnace temperature is 100°C or below.

IMPORTANT NOTE WHEN OPERATING PYROLYZER OVER 800°C

Operation of the pyrolyzer with the furnace temperature over 800°C for a long period time will shorten the useful lifetime of the furnace heater. Operation of the pyrolyzer over 800°C should be 20 minutes or less. Also, when the pyrolyzer is at standby, keep the furnace temperature below 200°C. This will extend the useful time of the furnace heater.

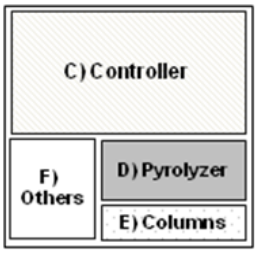
CONTENTS IN THE PACKAGE

EGA/PY-3030D Packing contents check list

This product is including following items. We recommend that you first examine the contents of the package you have received. If the contents differ from the package list below, or some items are missing, contact your dealer immediately.

※ View from the top

(top layer)



(bottom layer)

A) contents: ☐

A1)

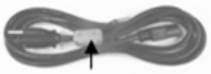
CD-ROM ☐
(Control software, Operation manual (PDF))

Performance Guarantee ☐

Catalog ☐

B) contents: ☐

B1)
AC Power Cable (1ea) ☐
100V (UL) (Re-order: PY1-7001, 1ea) ☐
or
200V (VDE) (Re-order: PY1-7002, 1ea) ☐
or
200V for China (CCC) (Re-order: PY1-7024, 1ea) ☐



Noise filter for AC Power Cable (1ea)
(Re-order: PY1-6034, 1ea)

B2) ☐

110V extension cable (1ea)
(220 V model, PY-3030D-220, only)
(Re-order: PY1-7105)

B3) ☐

Double-Shot Sampler Stand (1ea)
(Re-order: PY1-2022)

B4) ☐

Furnace purge gas restrictor B (1ea)
(Re-order: PY1-7618)

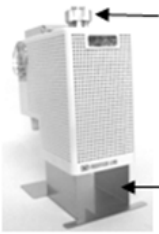
C) contents: ☐

C1)
Temperature Controller (1ea)

100V-120VAC ☐
or
200V-240VAC ☐

D) contents: ☐

D1) Pyrolyzer (1unit) ☐



Liquid Injection Sampler (1ea)
(Re-order: PY1-1036, 1ea)

Storage Stand 30A (1ea)
(Re-order: PY1-3631, 1ea)

100V-120VAC ☐
or
200V-240VAC ☐

D2) Heater sensor cable (1ea) ☐

100V-120VAC ☐
or
200V-240VAC ☐

E) contents: ☐

E1)

System Check Column (1ea)
(Re-order: UA5-30M-0.25F)

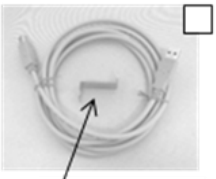
EGA Capillary Tube (1ea)
(Re-order: UADTM-2.5N)

F) contents: ☐

F1)

Cooling Gas Connecting Tube
(for cooling gas, 1.5 m, with one-touch connector 1/8 Swagelok female, 1ea.
Re-order: PY1-7801, 1ea)

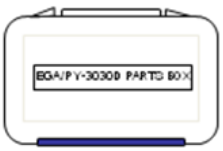
Cooling Gas Tube (with restrictor)
(for cooling gas, 1.5m, 1ea.
Re-order: PY1-7613, 1ea)



USB Cable
(2m, A-type male, B-type male, 1ea.
Re-order: PY1-7805, 1ea)

USB cable fixing bracket
(Re-order: PY1-3751, 1ea)

F2) ☐



EGA/PY-3030D PARTS BOX

EGA/PY-3030D PARTS BOX

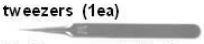
☐ **Eco-Stick SF (10ea, 1set)**
(Re-order : PY1-ES10F, 50ea, 1set)

☐ **Eco-Stick DF (10ea, 1set)**
(Re-order : PY1-ES20F、50ea, 1set)

☐ **Quartz Pyrolysis Tube A30 (2ea)**
(Re-order : PY1-3018A,1ea)

☐ **3030 Tool Set B (1set)**
(Re-order: PY1-K031, 1set)

tweezers (1ea)



10-12mm wrench (1ea)

hexagon screwdriver(1ea)

2.0mm

☐ **Spare Socket Button Head Cap Screws with Flange (5ea)**
(M3x6 ,for Storage Stand 30A)

☐ **Septum**
(for liquid sampler, 5ea, 1set)
(Re-order : PY1-2025, 20ea, 1set)

☐ **Graphite Vespel Ferrule**
(3ea, 1set)
(Re-order : PY1-7911, 5ea, 1set)

☐ **Eco-Stand AL (5ea)**
(Re-order: PY1-EH10AL, 5ea, 1set)

☐ **Double-Shot Sampler (1ea)**
(Re-order: PY1-1020, 1ea)

☐ **Spare cap screws (5ea)**
(M2.5x4 ,for sampler base and ITF cover)

☐ **Eco-cup SF (20ea, 1set)**
(Re-order : PY1-EC50F, 100ea, 1set)

☐ **Standard Sample**
(Polystyrene 2.5mg thin film, Me-stearate 5wt%, 1ea)
(Re-order: PY1-4908, 1ea)

Add 0.5 ml dichloromethane, benzene or toluene to dissolve polystyrene before use.

☐ **Level Vial (1ea)**
(Re-order: PY1-7131, 1ea)

☐ **O-ring P-6W (10ea, 1set)**
(Re-order:PY1-2017, 20ea, 1set)

☐ **ITF Union N set (A + B, 1set)**
(Re-order: PY1-3515, 1set)

ITF Union N (A without B, 1ea)
(Re-order : PY1-3513 , 1ea)

ITF Needle N (B, 2ea)
(Re-order : PY1-1274 , 3ea)

☐ **Eco Pick-up F (5ea)**
(Re-order: PY1-EP55F, 5ea)

☐ **Eco-cup LF (20ea, 1set)**
(Re-order : PY1-EC80F, 100ea, 1set)

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CHAPTER 1 ABOUT MULTI-SHOT PYROLYER

1.1 BACKGROUND OF DEVELOPMENT

Pyrolysis gas chromatography (Py-GC) can be used to characterize most materials including insoluble materials and complex materials at trace levels often without any pretreatment of the samples. Unique information about the sample, which is unobtainable by other analytical techniques, makes Py-GC techniques essential in virtually every type of laboratory. Historically, data obtained using Py-GC has suffered from poor reproducibility and poor recoveries of reactive compounds.

Frontier Laboratories has overcome these shortcomings by designing a system based on a technique developed by Emeritus Professor Shin Tsuge of Nagoya University. Professor Tsuge's technique utilizes a small cup. Sample is placed in the cup which then free-falls into a micro furnace. The sample temperature goes from ambient to the pyrolysis (i.e., furnace) temperature in several tens of milliseconds. This system features precise temperature control and minimal condensation of pyrolyzates in the system. In 1992, Frontier Laboratories commercialized the Double-Shot pyrolyzer.

The Double-Shot pyrolyzer added a completely new dimension to polymer analysis. For the first time the volatile compounds and pyrolyzates originating from a polymer could be analyzed separately.

The Double-Shot pyrolyzer features a temperature programmable furnace which can be used to perform Evolved Gas Analysis (EGA). With this technique, volatiles and pyrolyzate gasses from a polymeric sample evolve continuously as the sample is heated. A plot of sample temperature *vs.* detector response is termed as an EGA thermogram. EGA is similar to thermal gravimetric analysis (TGA). EGA is currently used in many research laboratories for the detailed determination of the thermal and chemical behavior of polymers, and also in production facilities as a quality control method.

Frontier Laboratories also offers a number of Ultra ALLOY® metal capillary columns. They provide contamination resistance about four times greater and thermal stability 50ºC higher than columns made using fused silica tubing. They are ideally suited for the analysis of pyrolyzates ranging from C_1 to over C_{100}.

Our research and development team has devoted years of basic research to improve the quality of the Double-Shot pyrolyzer and has developed and commercialized a variety of peripheral devices. These have greatly expanded the application areas of the Double-Shot pyrolyzer.

Over the past 20 years, a variety of high performance polymeric materials have appeared in the market which has generated a demand for a quantitative technique to characterize these materials. In particular, the thermal decomposition behaviors of the materials at high temperatures are of considerable interest. The sheer number of new formulations and additive packages has increased

the need for greater laboratory productivity.

In response to these needs, Frontier Laboratories has completely redesigned the pyrolyzer furnace using the expertise acquired in the development of the Double-Shot pyrolyzer PY-2020iD. Four years of research has led to the development of the Multi-Shot pyrolyzer EGA/PY-3030D. The Multi-Shot pyrolyzer EGA/PY-3030D is based on a proprietary high temperature resistant ceramic heater with very low heat capacity. The ceramic heater along with a re-engineering of the sampler base and the injection port interface has yielded a sophisticated GC inlet that has the versatility to analyze virtually any sample, produces amazing precision, and increases laboratory productivity by over 60%. The EGA/PY-3030D appears to offer the analyst the best of all worlds.

* Double-Shot pyrolyzer: patent no. 2742492

* Multi-Shot pyrolyzer, Double-Shot pyrolyzer, Ultra ALLOY®: The registered trademarks of Frontier Laboratories Ltd.

1.2 FEATURES OF EGA/PY-3030D

The features of the EGA/PY-3030D pyrolyzer are summarized below:

1. Operational temperature range: ambient +10ºC to 1,050ºC.
2. A low mass, high temperature ceramic heater rapidly heats and cools
3. Performance guaranteed in both the EGA and Single-Shot modes.
4. All wetted surfaces deactivated
5. Needle interface is heated using a specially designed adapter. This ensures thermo homogeneity and greatly improves the analysis of high boiling compounds
6. Various types of sample analysis (four analytical methods and five samplers)

Choose an analytical method and a sampler based on your analytical purposes and sample matrix.

The standard operating software is used for:

- Single-Shot GC
- Double-Shot GC
- Evolved gas analysis (EGA-MS)
- Heart-Cut EGA-GC/MS

Five samplers

- Double-Shot sampler (standard)
- Liquid sampler (standard)
- Online-micro reaction sampler (optional)
- Micro thermal desorption sampler (optional)
- Online micro UV sampler (optional)

1.3 PERIPHERAL DEVICES

The analytical capability of the EGA/PY-3030D can be expanded by adding peripheral devices such as the Selective Sampler, Microjet Cryo-trap, etc. In addition, Frontier Laboratories has developed MS search software which facilitates the rapid and in-depth characterization of polymeric materials. In support of the search engine, Frontier offers a host of polymer/additives libraries. Table 1 lists the various peripheral devices designed to be used with the EGA/PY-3030D pyrolyzer.

Table 1 Analytical methods and peripheral devices available with the EGA/PY-3030D

		Peripheral devices					
		Auto-shot sampler	Micro UV irradiator	F-Search *1	Carrier gas selector	Selective sampler	MicroJet Cryo-Trap
Analytical methods	Evolved gas analysis (EGA-MS)	√.		a, d			
	Single-Shot analysis	√		b, c, d		√	√
	Double-Shot analysis	√		b, c, d			req.
	Heart-Cut EGA analysis	√		b, c, d		Req.	Req.
Other analytical methods	Thermal desorption analysis	√		d		√.	√
	Analysis in air atmosphere (Except EGA)	√		c	Req.	Req.	Req.
	UV/PY-GC/MS analysis	n/r	Req.	c, d	*2	√	√

Req.: required, √= compatible, n/r: not required

*1 recommended library: (a) EGA polymer library. (b) Pyrogram polymer library, (c) Pyrolyzates library, (d) Additive library

*2 Micro UV irradiator standard package contains a device having equivalent function as Carrier gas selector.

1.4 SCHEMATIC OF MULTI-SHOT PYROLYZER FURNACE

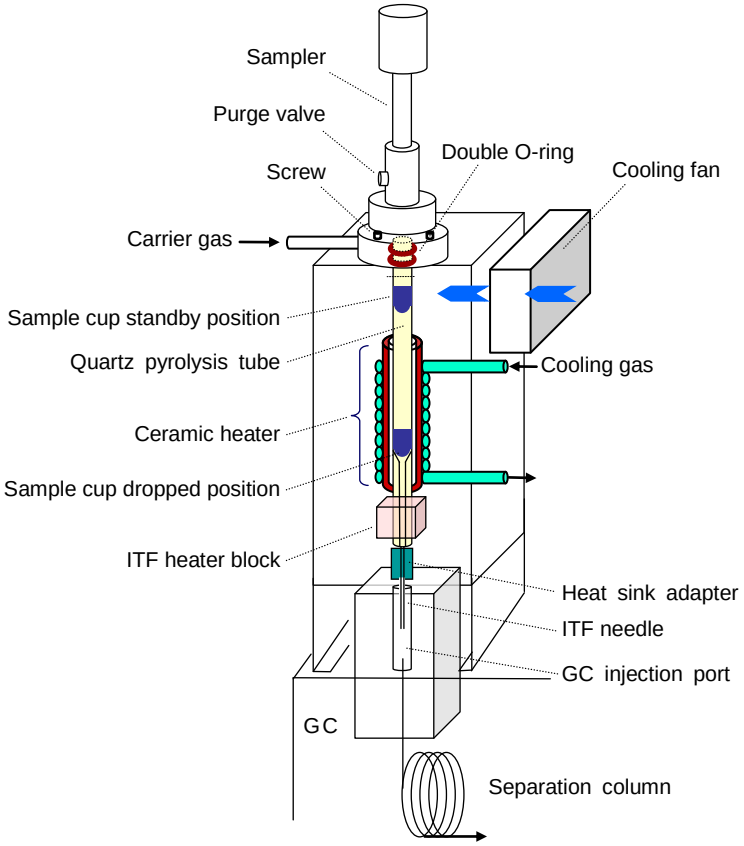


Fig. 1.1 Schematic of Multi-Shot Pyrolyzer

CHAPTER 2 SPECIFICATIONS

The system consists of the pyrolyzer furnace, which also serves as thermal desorption unit, and temperature controller.

	Multi-Shot pyrolyzer EGA/PY-3030D
1. Performance guaranteed (detector: MS)	See CHAPTER 8 of this manual for analytical conditions.
1. Reproducibility of pyrograms 2. Reproducibility of EGA thermograms	Flash pyrolysis of PS (25 µg) at 550°C (Eco cup LF used), the coefficient of variation (RSD) of the peak area ratio of styrene trimer (SSS) and methyl stearate (Me-Ste): ≤2% (Eco cup LF, single cup used) The coefficient of variation (RSD) of the peak top temperature of PS: ≤0.3%
2. Furnace and sampling unit	(Vertical micro furnace)
● **Pyrolyzer furnace** Sample introduction Pyrolysis tube material Temperature control range and stability Cooling system Cooling time (pressure: 500kPa)	Cylindrical ceramic heater Push-button to free-fall system with manual up/down-movement Quartz Room temperature + 10°C up to 1,050°C (1°C step) / ±0.1°C Forced air cooling by nitrogen or compressed air Within 10 min (800°C down to 50°C when ITF in auto mode)
● **Interface (ITF)** Temperature/Stability ITF needle	Cartridge heater 40 up to 450°C (1°C step) / ±0.1°C Needle with side opening (inner surface deactivated by gradient multi-layer treatment)
● **Sampler**	
Double-Shot sampler	For Double-Shot and Single-Shot analyses
Liquid sampler	For introduction of liquid samples (regular 10 µL micro syringe can be used)
● **Sample cup**	
Eco cup SF (small)	Maximum capacity: 50 µL (deactivated stainless steel)
Eco cup LF (large)	Maximum capacity: 80 µL (deactivated stainless steel)
3. Temperature controller	PC control (PC not included)
● **Functions**	SGL/DBL, Direct EGA, Heart-Cut EGA, ITF temp, Method File, Temp Monitor, Maintenance
	Other functions: control of Selective Sampler, MicroJet Cryo-Trap, temperature calibration, and Carrier Gas Selector
● **Temperature control range**	
Pyrolyzer furnace	30 up to 1,050°C (1°C step)
Ramp rate	600°C/min max (1°C step)
Hold time	0 up to 999.9 min (0.1 min step)
Pyrolysis time	0 up to 999.9 min (0.1 min step)
Interface	30 up to 450°C (1°C step) Auto/Manual control
● **Overheat protection temperature**	PY: 1100°C, ITF: 500°C, Sample cup standby position: 100°C
● **Communication**	USB 2.0

*: Prolonged operation of the pyrolyzer at a furnace temperature higher than 800°C will shorten the furnace heater life. When operating at 800°C or higher, keep the operation time less than 20 minutes. If it is operated at 800°C or higher for a prolonged period time, contact us at cs@frontier-lab.com. When the pyrolyzer is in a standby state, we recommend that the furnace temperature should be kept below 200°C. This will extend the furnace heater life.

	Multi-Shot pyrolyzer EGA/PY-3030D
4. Standard accessories	
Quartz pyrolysis tube	2 pcs
Ultra ALLOY capillary column	5% diphenyl 95% dimethylpolysiloxane, L=30 m (i.d.=0.25mm), df=0.25 µm 1 pc
Capillary tube for evolved gas analysis	(UADTM, deactivated tube, L=2.5 m, i.d.=0.15 mm) 1 pc
ITF needle N	2 pcs (with one ITF union)
Eco cup	Eco cup (50 µL) 20 pcs, Eco cup LF (80 µL) 20 pcs
Eco stick	Eco stick SF 10 pcs, Eco stick DF 10 pcs
Sampler	Double-Shot sampler 1 pc, Double-Shot sampler stand 1 pc, Liquid sampler 1 pc, septum for liquid sampler 5 pcs
Graphite Vespel ferrule	3 pcs
Performance evaluation polystyrene standard sample	Polystyrene 2.5mg film, Me-stearate 5wt% , in 2ml glass bottle. Add 0.5ml dichloromethane, benzene or toluene to dissolve polystyrene before use.
Miscellaneous	Control software CD (Windows 10, 8.1, 8, 7, Vista, XP compatible）1 pc, Power cable (1.5 m) 1 pc, Vinyl tube for cooling gas (1.5 m) 2 pcs, USB cable (2 m) 1 pc, Valve control cable adapter 1 pc, Tool kit 1 set, Level vial 1 pc, Sample for performance check (polystyrene solution containing methyl stearate, 5 µg/µL) 1 pc (0.5 ml), Eco pickup F 5 pcs, Eco stand AL 5 pcs, Operation manual (pdf)
5. Miscellaneous	
Power	AC100/120V (50/60 Hz) 400W (MAX) or AC200/240V (50/60 Hz) 400W (MAX)
Dimension Pyrolyzer unit Temperature controller	 76(W) x 143(D) x 150(H) mm/1.6kg (less cables, excluding protuberances) 120(W) x 310(D) x 310(H) mm/5.4kg or 120(W) x 310(D) x 310(H) mm/ 7.6kg
Recommended GC and GC/MS	Agilent: Agilent 7890 GC, Agilent 5975 GC/MS Jeol: JMS-Q1000GC Mk II Perkin Elmer: Clarus GC, GC/MS Shimadzu: GCMS-QP2010, GC-2010 Thermo Fisher: Trace GC, Trace ISQ SCION / Bruker: SCION GC Others: Contact us for details
6. Options Peripheral device	• AS-1020E: Auto sampler for Multi-Shot pyrolyzer (automated analysis of max 48 samples) • CGS-1050Ex: Carrier gas selector (switches between air and He, use with SS-1010E recommended) • MJT-1035E: MicroJet Cryo-Trap (completely traps compounds of C_4 or greater by cooling the head of a column with liquid nitrogen jet in GC oven) • PY-1110E: F-Search system (evolved gases from polymer/pyrogram-MS library, additive library, pyrolyzates library) • PY1-1050: On-line micro reaction sampler (introduces compounds generated at high temperatures/pressures into separation column) • PY1-1060: Micro thermal desorption sampler (allows for the thermal desorption analysis of volatile gases) • SS-1010E: Selective Sampler (selectively introduces any temperature zone on EGA thermogram into a separation column with the use of gas pressure difference) • UV-1047Xe: Micro UV irradiator (comes with 1 pc of On-line micro UV sampler) • PY-7105E: Thermometer (used to measure the Multi-Shot pyrolyzer furnace temperature)
Consumables	• Vent-free GC/MS adapter • PY-K303D: Consumables set (estimated annual use for Multi-Shot pyrolyzer) • PY-K313D:Consumables set (estimated annual use for Multi-Shot pyrolyzer with AS-1020E) • Ultra ALLOY Metal capillary columns (id=0.25, 0.53 mm, L=5~60 m, variety of chemically bonded stationary liquid phases)

Other requirements and utilities

1) Gas chromatograph (with a split/splitless injection port): some limitations in installation depending on GC model.
2) Multi-shot pyrolyzer: pyrolysis furnace cooling gas (nitrogen or compressed air, pressure: 400-600kPa) supplied via o.d. 1/8 in or 3 mm pipe (cupper or stainless steel), readily accessible from the temperature controller of pyrolyzer (operating flow rate: 7 L/min)
3) Power outlet: AC100/120V or AC200/240V, 400W (MAX) with 3P socket
4) Control PC: A PC running Windows 10, 8.1, 8, 7, Vista, or XP operating system with a USB port. If the recommended GC or GC/MS is used, the PC can be shared with GC/MS control software programs.

About cooling time of pyrolyzer furnace

Fig. 2.1 shows the relationship between pyrolyzer furnace temperature and cooling time with varied cooling air pressures. The pressures (P) here are ones measured at the outlet of nylon tube (PY1-7801) that feeds air to the pyrolyzer.

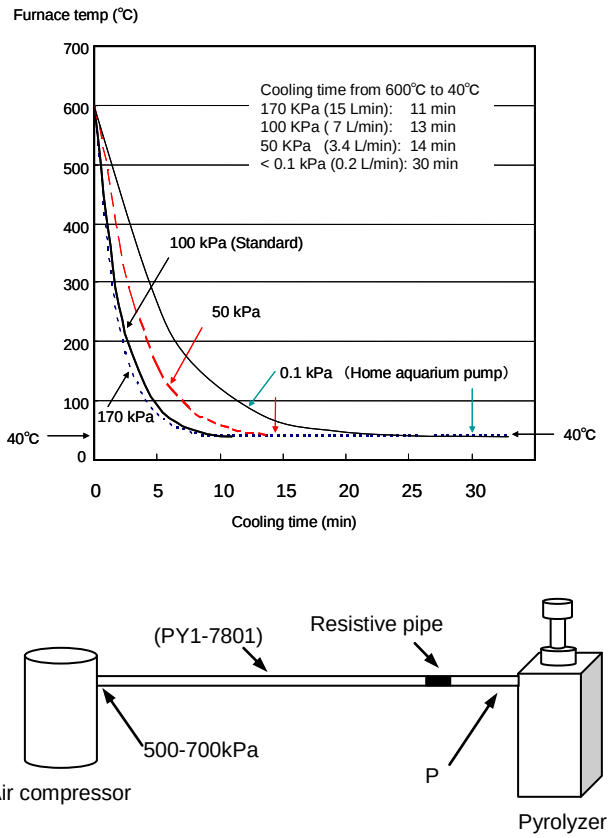


Fig. 2.1 Relationship between pyrolyzer furnace temperature and cooling time when varying cooling air pressure (ambient temperature: 20°C)

The relationship between air flow rate and air pressure is shown in Fig. 2.2.

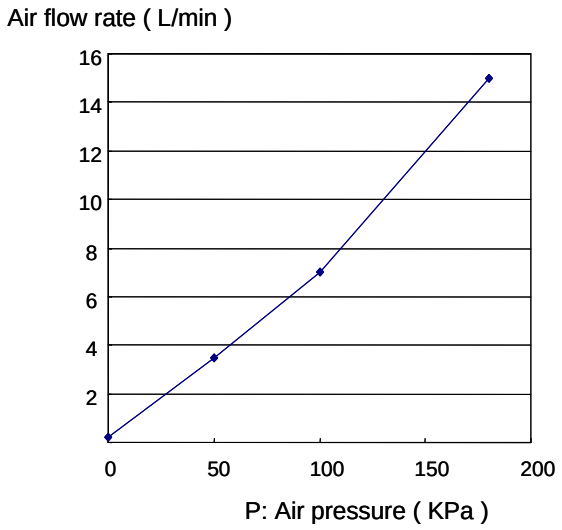


Fig. 2.2 Relationship between air flow rate and air pressure

CHAPTER 3 INSTALLATION

In this chapter, the overview of the installation process is described. For detailed installation instructions, consult the installation manual that comes with the installation kit for your GC model.

CAUTION

1. For your safety, wear protective goggles to protect your eyes during operation.
2. Before installation to your GC, TURN OFF the injection port temperature and wait until it drops below 100°C, or you are running at the risk of burns.
3. While installing the pyrolyzer to your GC. Be sure to TURN OFF the power of the temperature controller. If the pyrolyzer is powered, it quickly becomes very hot.

3.1 PREPARING INSTALLATION

3.1.1 Before installation

Ensure that you have the following items before installing the pyrolyzer. These are not included in the package.

No.	Item	Requirement	Note
1	Installation kit	For your specific GC model	Not included in this package (purchased separately)
2	Power	AC100/120V or AC200/240V, 400W (MAX) Plug with a ground pin	Supplied power cable is 2 m long.
3	Control PC	OS: Windows 10, 8.1, 8, 7, Vista, XP One USB port CD ROM drive	PC that controls GC or GC/MS can be shared. Supplied USB cable is 2 m long.
4	Cooling gas[1)]	Compressed air or nitrogen gas Pressure: 500~800 kPa Consumption: about 7L/min (at 600 kPa)	1/8 in female Swagelok outlet must be accessible within 1.5 m from the temperature controller. Alternatively, 1/8 in or 3 mm pipe can be used for connection with supplied nylon tube having flow restrictor.
5	Vent-free GC/MS adapter[2)]	Part No. : MS402180 MS402190 MS402195 (Depends on GC/MS model)	*Optional Permits switching of separation columns while maintaining vacuum of MS.
6	Installation space	Space for temperature controller Width: 20 cm Depth: 35 cm or more	The length of cable connecting the pyrolyzer to temperature controller is 2 m.

1) Air used for FID can be branched and used; however, high sensitivity FID work may be affected by this gas sharing.
2) The use of this adapter greatly saves time when switching columns and or an EGA tube and is a must-have item for productive analytical work.

3.1.2 Pumping down MS (for saving installation time)

It is recommended that the GC/MS be shut down during the installation of the pyrolyzer; however, to save time, the installation may be performed while the MS is pumping down.

- If you have a Vent-free GC/MS adapter, install it first, pump down the MS and start the pyrolyzer installation.
- If a Vent-free GC/MS adapter is not used, connect the inlet of the supplied Ultra ALLOY column to the GC injection port. Then after allowing the He carrier gas to flow through the column for 5 min, connect the column outlet to the MS and start pumping down the MS. After ten minutes, disconnect the column from the GC injection port and plug the column inlet with a septum, continue the MS pump down.

A separation column for the pyrolyzer operational verification (Ultra ALLOY, 5% diphenyl 95% dimethylpolysiloxane, L=30 m, id.=0.25 mm, df=0.25 µm) is supplied with the pyrolyzer as standard.

3.2 REPIPING CARRIER GAS LINE

The installation of the pyrolyzer onto a GC requires redirecting the carrier gas line. Fig. 3.1 shows the carrier gas flow paths before and after the installation of pyrolyzer. Refer to this figure throughout the installation steps.

Disconnect the carrier gas line that runs through the carrier gas controller to the GC injector port, and reconnect it to the pyrolyzer. Plug the carrier gas line at the injection port with a cap nut. Depending on your GC model, the carrier gas line may need to be cut.

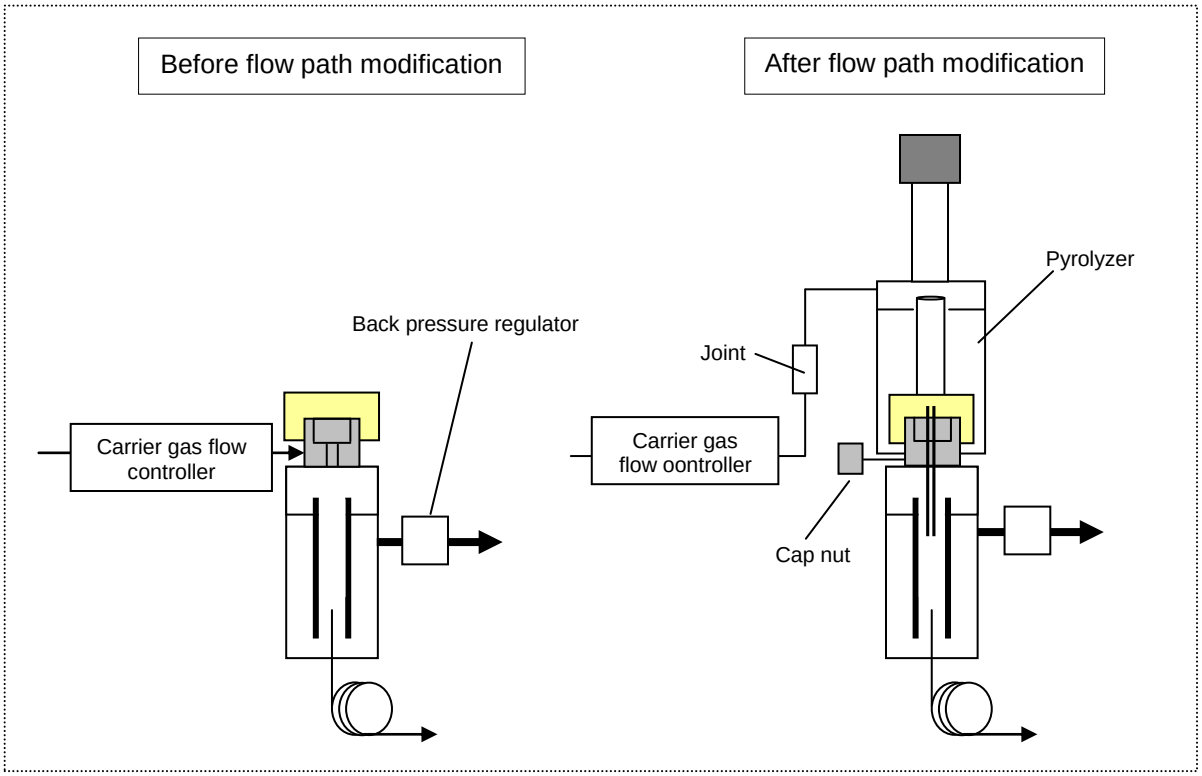


Fig. 3.1 Carrier gas flow paths before and after installation of pyrolyzer

3.3 SETTING UP GC INJECTION PORT

3.3.1 Replacing septum nut of GC injection port

The GC specific installation kit includes a septum nut for the GC injection port, a needle guide, and heat sink that is attached to the pyrolyzer.

The septum attached to your GC is replaced with the one in the kit. The heat sink is secured to the pyrolyzer interface. The bottom end of the heat sink is designed to fit into the septum nut. These parts need to be properly installed in order to uniformly transfer heat to the septum. This minimizes low temperature valleys in the flow path that sample passes through.

3.3.2 Replacing septum

It is recommended that the septum contained in the installation kit be used. This septum has a thin coating on the surface in order to minimize adhesion to metal surfaces at high temperatures. Then finger-tighten the septum cap at the GC injector. The coating facilitates the removal of the septum when it is replaced.

3.3.3 About Injection port liner

Any injector liner recommended by your GC manufacturer can be used. When using the pyrolyzer, the sample is vaporized in the pyrolyzer furnace, therefore, there is no impact with regards to mixing efficiency and the associated discrimination that occurs in conventional split injection ports.

3.4 SETTING UP PYROLYZER FURNACE

3.4.1 Installing quartz pyrolysis tube to pyrolyzer

Be sure to use the supplied quartz pyrolysis tube (part No. PY1-3018A). The tubes (PY1-2018A) used for the preceding models such as PY-2020iD/iS cannot be used for this pyrolyzer because they are different lengths.

3.4.2 Attaching interface union and needle

Ensure that you use the supplied interface union and needle. The ones for PY-2020iD/iS may be attached, but are not suited for this pyrolyzer.

The supplied graphite Vespel ferrule (part No. PY1-7911) that attaches to the quartz pyrolysis tube should be used. 100% graphite ferrules (PY1-7921) may be used, but with lower sealing capability and replacement can be troublesome.

3.4.3 Mounting pyrolyzer on GC

Insert the ITF needle at the bottom of the pyrolyzer to the GC injector by carefully lowering the pyrolyzer while holding it in the upright position until the heat sink hits the septum nut. At this point, make sure that the heat sink makes a good contact with the septum nut. Also, while lowering the pyrolyzer, engage the screws on the both sides of the pyrolyzer to the slots of the pyrolyzer stand which seats on the GC, then secure it by tightening the screws with the supplied hexagonal screwdriver (2 mm). Use a level to ensure that the pyrolyzer is level.

3.5 CONNECTING TEMPERATURE CONTROLLER

3.5.1 Connecting power cable to outlet

Use only a power outlet with a ground terminal and correct voltage specified. Required power can be found near the power cable connector on the rear panel of the temperature controller.

Also, ensure that the power requirement for the pyrolyzer is met by checking the pyrolyzer rear panel.

3.5.2 Connecting control PC

Connect the temperature controller to your PC using the supplied USB cable (2 m long). The control software for the pyrolyzer is independent from GC or GC/MS control software, and it does not exchange data with other instrument.

3.5.3 Hooking up remote signal cable

Use the remote signal cable to connect the temperature controller to your GC. Through this cable, the temperature controller receives “Ready” signal from the GC, and sends “GC START” signal back to the GC.

3.6 CONNECTING COOLING GAS LINE

3.6.1 About cooling gas

Nitrogen or compressed air is normally used as a cooling gas. Since the gas passes through high temperature surfaces, it should not contain oil or moisture. The purity of the gas does not directly influence analytical results.

A low capacity aquarium air pump for home fish tank can be also used. Automatic control is available through the external power outlet (EXT PUMP, 100V 3W) located at the rear of the temperature controller.

WARNING

This product uses compressed air or nitrogen for cooling the pyrolyzer furnace. Ensure that you have a good ventilation if nitrogen gas is used. There is a risk of suffocation, if it is used in a confined area with poor ventilation.

3.6.2 Connecting temperature controller and pyrolyzer

Use the supplied vinyl tubing to connect the gas outlet to the temperature controller then the temperature controller to the pyrolyzer.

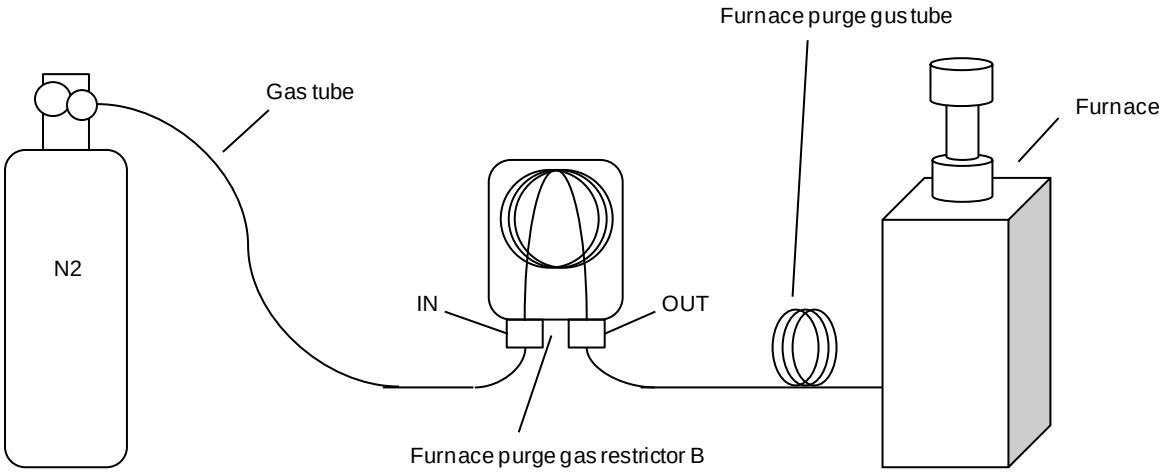
The cooling gas is automatically controlled and is consistent with the pyrolyzer temperature settings and with the temperature program.

3.7 Connecting furnace purge gas tube

By streaming an inert gas such as N2 through the furnace purge gas tube (see below), oxygen in the furnace is purged and temperature sensor and other parts in the furnace is protected from oxidative degradation. In particular, when the furnace is operated at 800ºC or higher, be sure to connect the tube to an inert gas source.

3.7.1 Connecting N_2 gas tube

A inert gas line such as N_2, and connect it to the furnace purge gas tube via the purge gas restrictor B supplied in the package. In the case where the supply pressure is 500 kPa, there is approximately 100mL/min of gas flow. If air is used, there is no effect of preventing oxidative degradation.



WARNING Do not connect flammable gas line such as hydrogen gas. There is a risk of explosion.

CAUTION Do not position the warning label of the furnace purge gas tube over a high temperature GC oven. The label may be unreadable by thermal degradation.

3.8 INSTALLING PERIPHERAL DEVICES

See also the instruction in the operation manual that comes with the peripheral device. All of peripherals of Multi-Shot Pyrolyzer require a 100-115VAC power supply. The power outlets marked “100 / 115VAC OUT, 100W” on the rear panel of the temperature controller can be used to power each peripheral. When more than two outlets are required, use “110V extension cable” supplied in the package.

CAUTION The total of power cumsumption for the outlets must be less than 100W. Otherwise the breaker turns OFF.

3.8.1 Connecting Auto-shot sampler (AS-1020E)

Connect the RS-232C cable (for control of AS-1020E) to the RS-232C port located at the rear of the temperature controller. Do not directly connect it to your PC. The Auto-shot sampler is indirectly connected to PC via the temperature controller.

3.8.2 Connecting other peripheral devices

Selective sampler (SS-1010E), MicroJet Cryo-Trap (MJT-1030Ex), and Carrier gas selector (CGS-1050Ex) can be also controlled through the temperature controller. The valve control cable that comes with the individual device is used to connect to the VALVE connector of the temperature controller. The supplied valve control cable adapter is used for the connection.

3.9 INSTALLING CONTROL SOFTWARE

3.9.1 Installing USB serial communication device driver

Before installing the control software, the USB serial communication driver needs to be installed.

1. Make sure that the USB cable is properly connected. The PC screen will display a “Found New Hardware” window when the temperature controller is turned ON.
2. Place the CD-ROM (EGA/PY-3030D control software) in the CD drive. (in the example below, CD drive is assigned to D: drive.)
3. A dialog box asks whether you want to start a setup program. Do not proceed, and just cancel and terminate the dialog.
4. Now, as shown in Fig. 3.4, specify a folder and install the USB-serial driver.

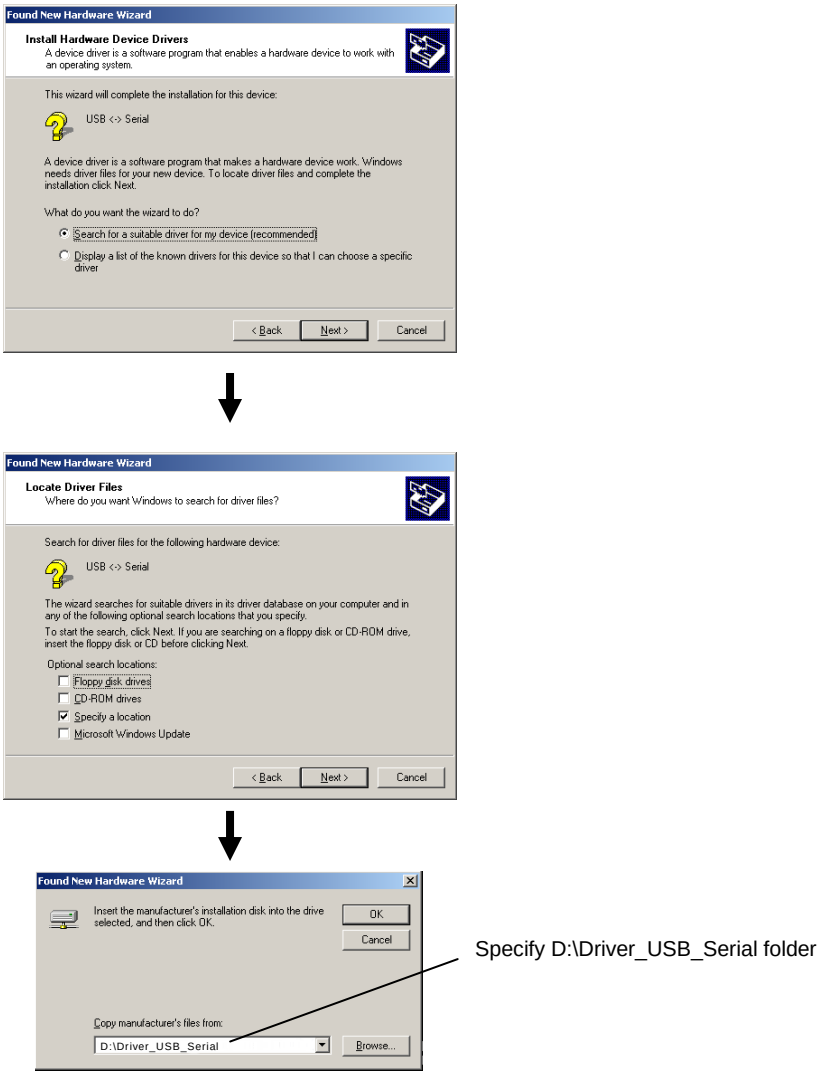


Fig. 3.4 Installing USB serial communication driver

3.9.2 Installing Control software

A password is required to install the control software. It is obtained through the Frontier Labs web page. For details about how to obtain the password, read the description contained on the CD-ROM software package.

1. Insert the “EGA/PY-3030D control software” CD-ROM into the CD drive of your PC.

2. A setup program will start. Proceed according to the instructions on the screen.

3.9.3 Setting COM port

Once the control software is opened, and if the dialog box shown in Fig. 3.5 is displayed, the USB serial port needs to be specified.

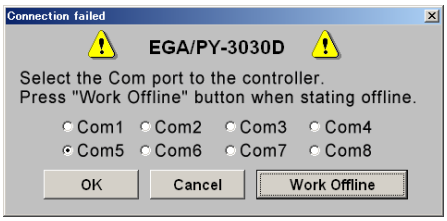


Fig. 3.5 Selecting a COM port

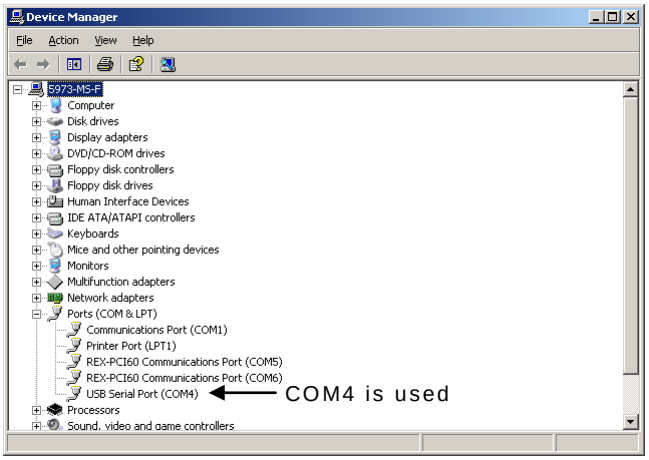


Fig. 3.6 Checking COM port from Device manager

1. Go to the Windows Control Panel, select “System”. Move to the “Hardware” tab, and select the “Device manager” button. Open “Port (COM and LPT)” as shown in Fig. 3.6 to obtain the COM port number used for the “USB serial port”.
2. Select the COM port number in the dialog box shown in Fig. 3.5. Select the OK button will start the control software.
3. If the port assigned is not in the range between 1 and 8, the port number needs to be reassigned. Right-click on “USB serial port” in Fig. 3.6 and select “Property”, go to “Port setting” tab, select the “Details” button. As shown in Fig. 3.7, select a port number between 1 and 8 that is not already assigned to others - see Fig. 3.6. If a used port number is selected, a warning dialog will pop up. Click on the OK button. Then follow Step 2. above.

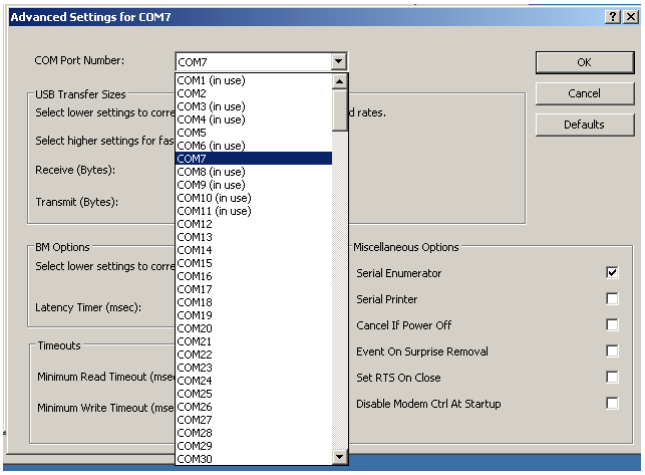


Fig. 3.7 Resetting COM port number

3.10 FRONT, REAR, SIDES, AND TOP VIEWS

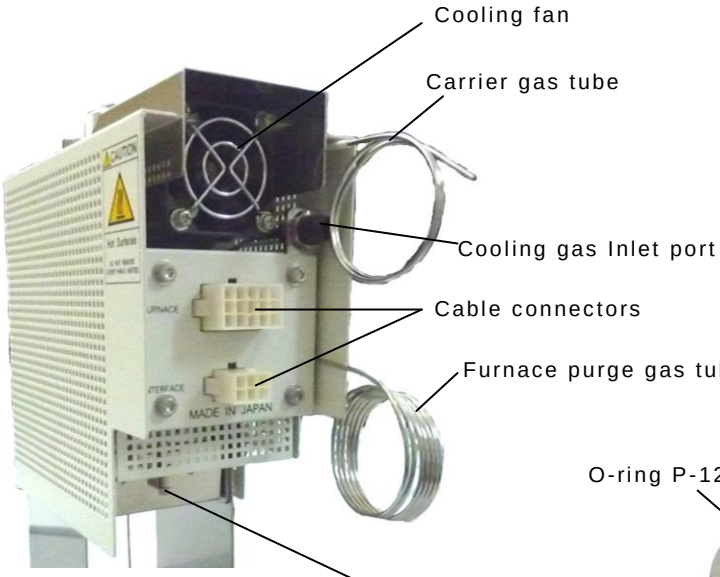
3.10.1 Pyrolyzer

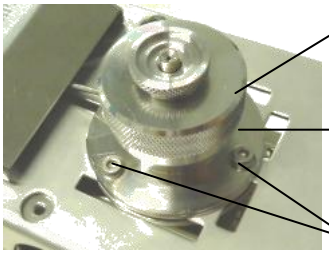
Views from different angles of the pyrolyzer are shown in Fig 3.8.

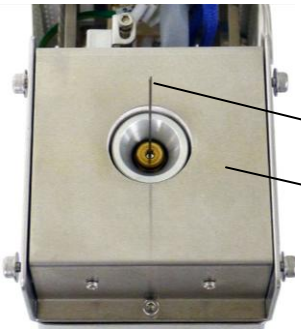
Side view

Front view

Rear view







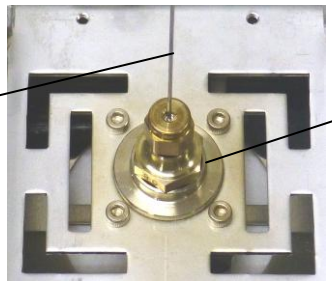


Fig. 3.8 Views from different angles of pyrolyzer

3.10.2 Temperature controller

The front and back panels of Temperature controller are shown below in Fig. 3.9.

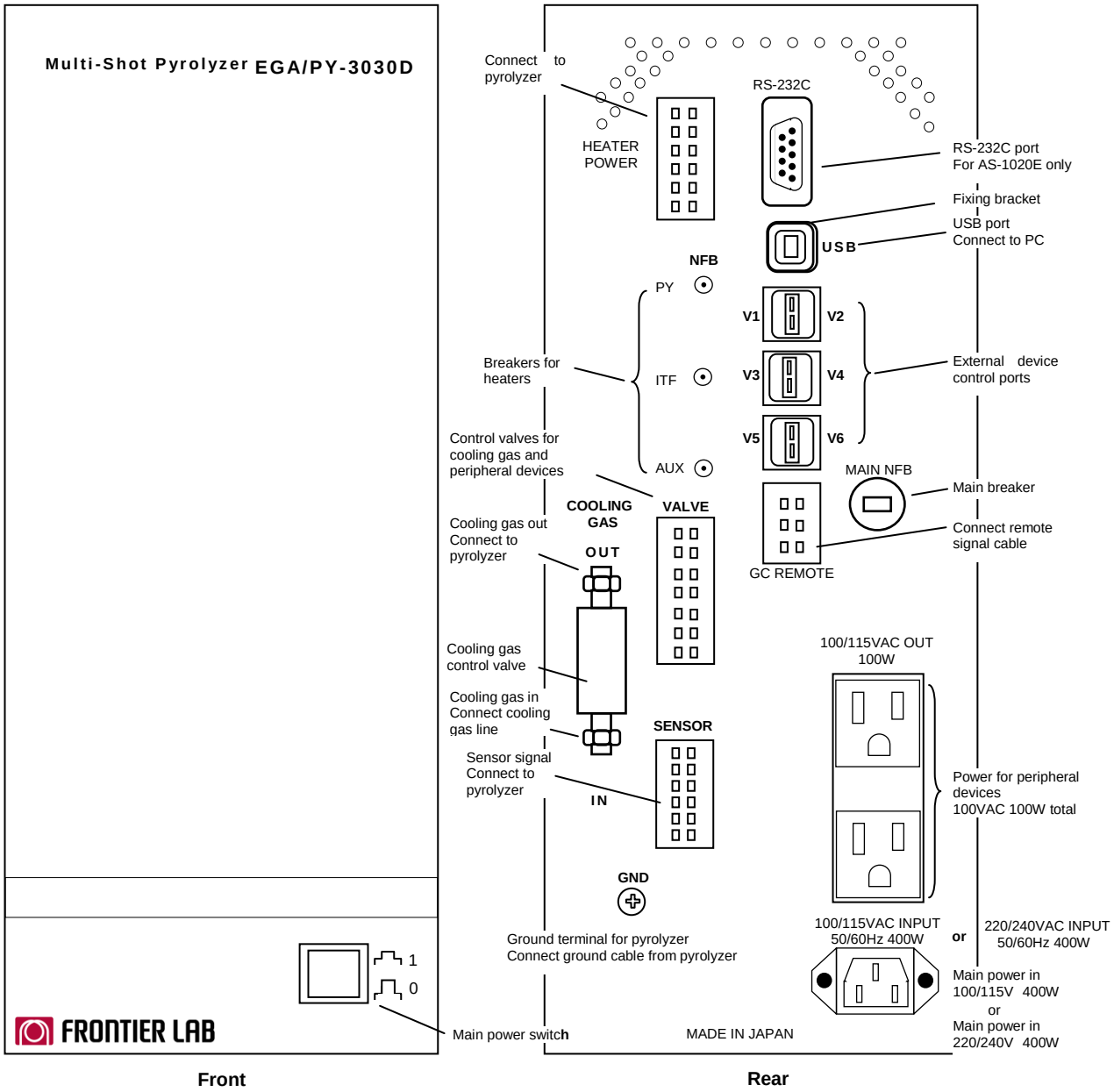


Fig. 3.9 Front and rear views of Temperature controller

CHAPTER 4 USING CONTROL SOFTWARE

4.1 START UP AND SCREEN LAYOUT

4.1.1 Starting program

From the **Start** menu, select [Programs] - [EGA/PY-3030D Control]. The start-up screen shown in Fig. 4.1 is displayed, followed by the set-up screen shown in Fig. 4.2

Fig. 4.1 Start-up screen for the EGA/PY-3030D Control software

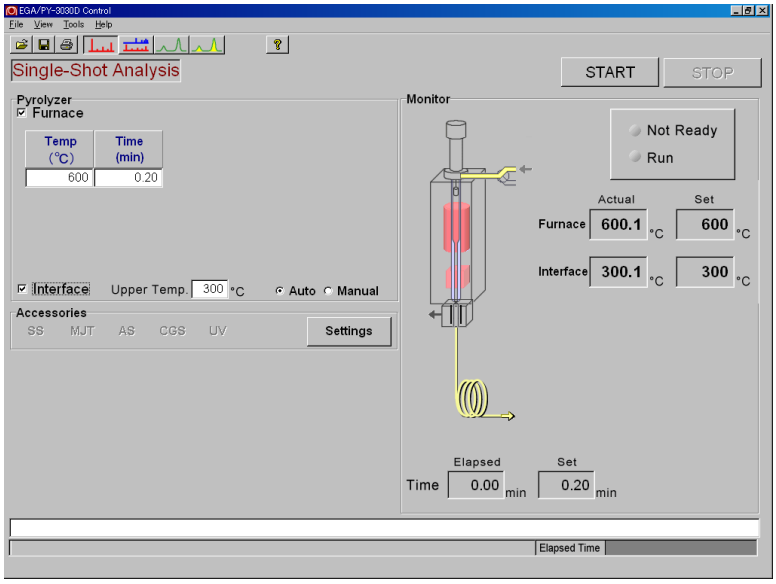


Fig. 4.2 Set-up screen for Single-Shot analysis

4.1.2 Screen layout

Fig. 4.3 shows the Double-shot screen of the operating software.

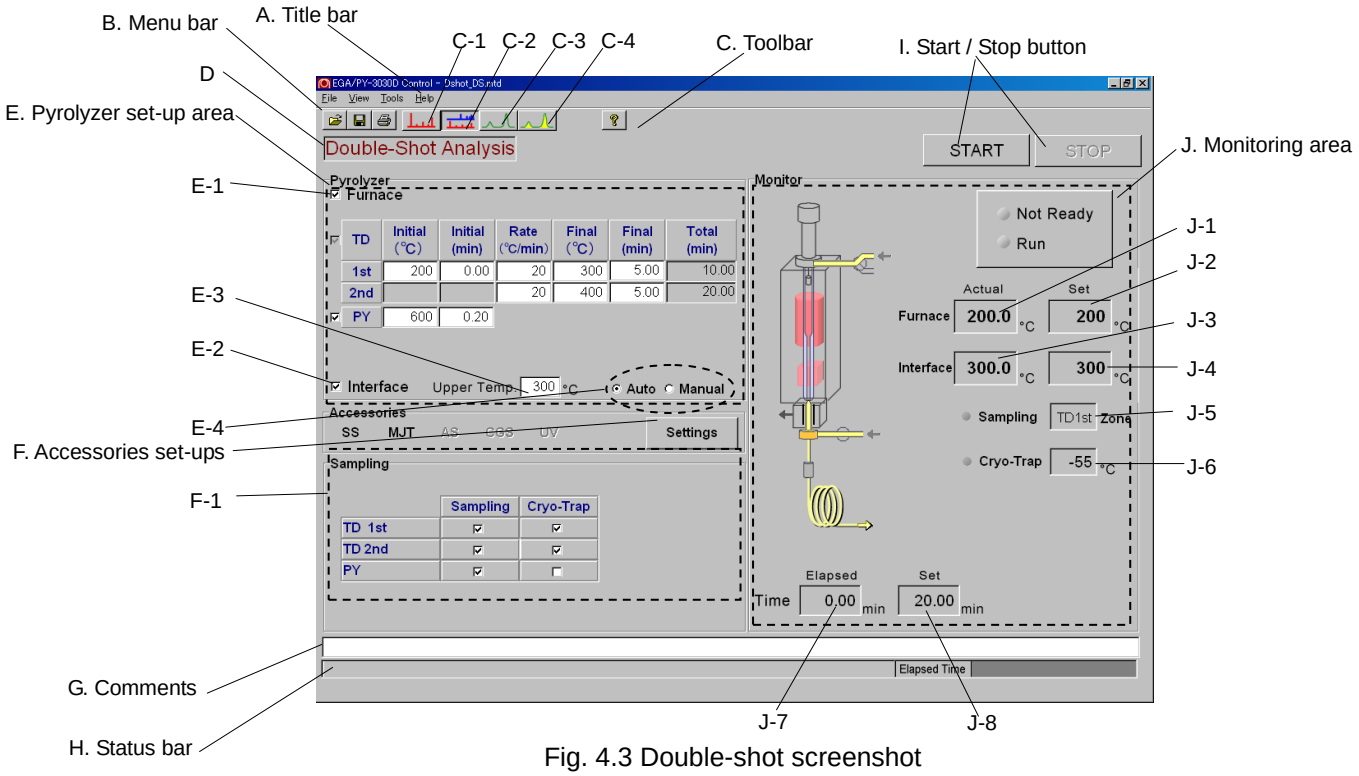


Fig. 4.3 Double-shot screenshot

A. Title bar: Shows a method currently used.

B. Menu bar: Provides access to options and submenus.

C. Toolbar: Shows icons for functions.

C-1: Selects Single-Shot Analysis.

C-2: Selects Double-Shot Analysis.

C-3: Selects Direct EGA Analysis.

C-4: Selects Heart-Cut EGA Analysis.

D: Shows analysis mode currently selected.

E. Pyrolyzer set-ups: Set parameters for pyrolyzer. This area depends on analysis mode selected.

E-1: Turns ON/OFF the pyrolyzer furnace heater. Checking the box turns the heater ON.

E-2: Turns ON/OFF the interface heater. Checking the box turns the heater ON.

E-3: Sets interface temperature.

E-4: Select a mode for interface control. If “Manual” is selected, the temperature is controlled at the one set using E-3. If “AUTO” is selected, the interface temperature is maintained 100°C above the pyrolyzer furnace temperature; however, the temperature set by E-3 is the upper limit temperature.

F. Accessories set-up: Sets parameters for accessories such as Selective Sampler, MicroJet Cryo-Trap, etc. The actual set-ups are dependent upon the analysis mode selected.

F-1: Sets operating parameters for the accessories to be used.

G. Comments: Comments can be added for each analysis mode.

H. Status bar: Displays the progress of the program.

I. Start/Stop button: Starts or stops the program.

J. Monitoring area: Shows operating condition of each device connected.

J-1: Shows the current temperature of the pyrolyzer furnace.

J-2: Shows the set temperature of pyrolyzer furnace or final temperature set in the program.

J-3: Shows the current temperature of the interface.

J-4: Shows the set temperature of the interface.

J-5: Shows the temperature zone currently being sampled.

J-6: Show the trap temperature of MicroJet Cryo-Trap.

J-7: Show the elapsed time since the start of program.

J-8: Shows the time required to complete the program.

4.2 BEFORE STARTING AN ANALYSIS

Before starting an analysis the system configuration (e.g., Selective Sampler, MicroJet Cryo-Trap, etc.) must be entered into the system. Set the interface temperature and its control mode according to the procedure described below.

4.2.1 System Configuration

4.2.1.1 Selecting a device or devices

(1) Click on “Settings” button (F. in Fig. 4.3) will display the screen shown in Fig. 4.4.

(2) “Check” the small checkbox to indicate that the device will be used.

(3) If air is used as an atmosphere gas, refer to section 4.2.1.3 of this manual.

(4) Click “OK” to end.

4.2.1.2 Abbreviations for accessory devices

CGS: Carrier Gas Selector

SS: Selective Sampler

MJT: MicroJet Cryo-Trap

AS: Auto-Shot Sampler

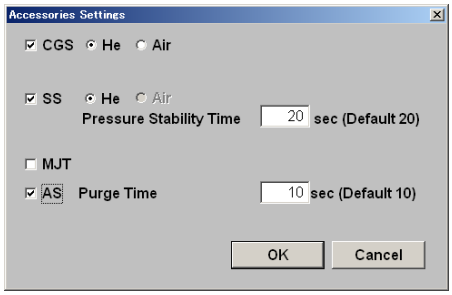


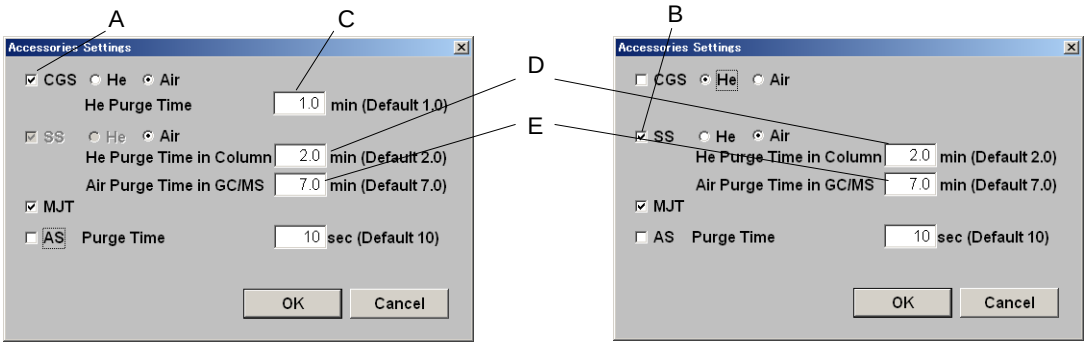
Fig. 4.4 System configuration

4.2.1.3 Using air as atmosphere gas

In this section, analysis in an air atmosphere is described. In this analysis, the sample is first heated in air; the carrier gas is then switched to helium in order to purge air from the column and detector. If the analysis is automated using the AS1-1020E Auto-shot sampler, the MicroJet Cryo-Trap (MJT-1030Ex) and Selective Sampler (SS-1010E) are required. In addition, the Carrier Gas Selector (CGS-1050Ex) is highly recommended. The procedure for performing an automated sequence of analysis in air is described below. Manual operation requires in-depth professional knowledge and extensive experiences. Contact us if assistance is needed (cs@frontier-lab.com).

(1) "Check" the checkboxes for MicroJet cryo-Trap and Selective Sampler - see Fig. 4.5.

(2) If using an automated Carrier Gas Selector, select "Air" as shown in Fig. 4.5(a). If a Carrier Gas Selector is not used or if manual control is used, select "Air" as shown in Fig. 4.5(b).



(a) Automatic control of Carrier gas selector. (b) Automatic control of Carrier gas selector not in use.

Fig. 4.5 Accessory device set-ups when analysis is being done in an air atmosphere.

(3) "He Purge Time" (C) in Fig. 4.5(a) is the time it takes for the Carrier Gas Selector to completely purge the helium in the Pyrolyzer with air. Normally the default value (1.0 min) is used.

(4) "He Purge Time in Column" (D) is the time it takes to completely replace the helium in the column with air. Normally 2.0 min is used.

(5) "Air Purge Time in GC/MS" (E) is the time it takes to completely purge the air in the column and detector with helium after the sample has completed the heating process. In particular, if a mass spectrometer is used as a detector, air in the mass spectrometer must be completely purged before turning on the filament. If this is the case, use a default value of 7.0 min, although this time is somewhat dependents on the MS model.

CAUTION

When performing a pyrolysis in air, air is used as a carrier gas. Be sure to allow a long purging time to completely replace air with inert gas such as helium, before raising the oven temperature.

4.2.2 Set-up the interface temperature

The interface temperature can be set to either the “Auto” or “Manual” mode. In the “Auto” mode, the interface temperature is automatically controlled based upon the pyrolyzer furnace temperature; on the other hand, in the “Manual” mode, it is always controlled at the selected temperature. These modes are entered - see E-4 of Fig. 4.3. Normally, the “Auto” mode is used. If the “Manual” mode is selected, the interface temperature is controlled at a constant temperature - see E-3 of Fig. 4.3. In the “Auto” mode, the temperature is maintained 100°C above the pyrolyzer furnace temperature; the interface temperature set in E-3 is the upper limit temperature. For example, the relationship between the Pyrolyzer furnace and interface temperatures is shown in Fig. 4.6. In this example, the “Auto” mode is selected using E-4 and the upper temperature is set to 300°C using E-3; the Pyrolyzer furnace is heated from 50°C to 500°C. The interface initial temperature is automatically set to 150°C, 100°C above the Pyrolyzer furnace temperature, and the temperature increases to 300°C - the upper temperature limit (E-3).

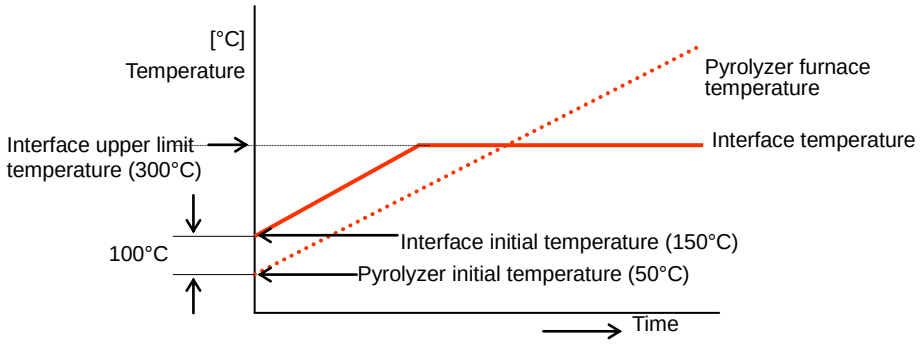


Fig. 4.6 Auto control of the interface temperature

4.3 SET-UP ANALYTICAL MODES AND OPERATION

Four analytical modes: Single-Shot Analysis, Double-Shot Analysis, Direct EGA Analysis, Heart-Cut EGA Analysis are available.

4.3.1 Set-up for Single-Shot Analysis and operation procedure

From the menu bar, select “Tool” – “Analytical Modes” – “Single-Shot Analysis”, or click on the “Single-Shot Analysis” icon (A) on the toolbar. The set-up screen shown in Fig. 4.7 will be displayed.

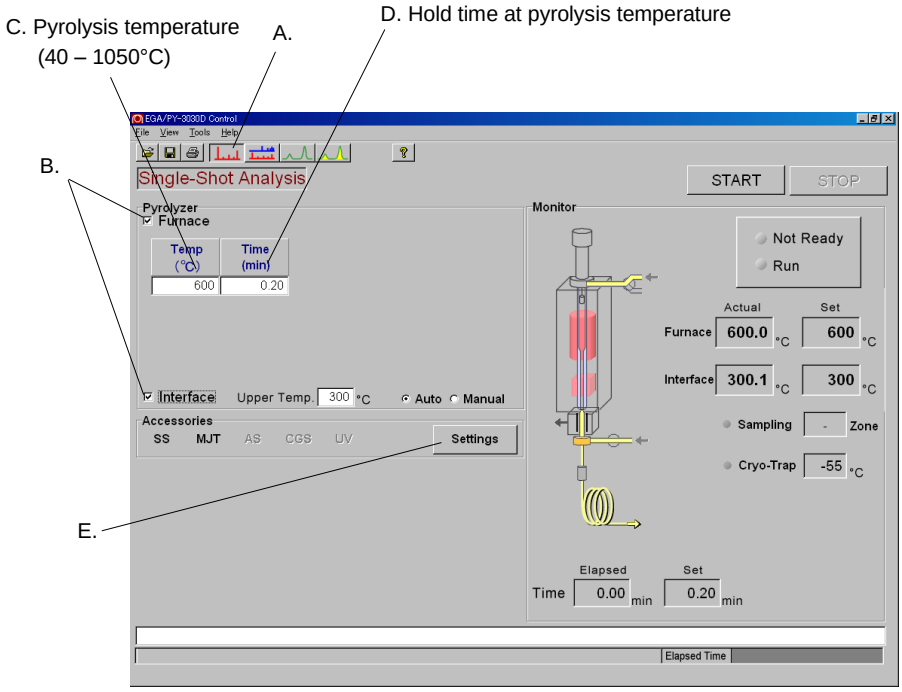


Fig. 4.7 Set-up screen for Single-Shot Analysis

(1) “Check” the Furnace and Interface checkboxes (B) to turn the heaters ON.

(2) Type in a furnace temperature in C, and allow it to stabilize.

(3) Enter a desired time in D. if the Selective Sampler is used, the sample vapors formed during the time defined in D after the “Start” button is clicked in (7), are introduced into the separation column.

(4) In the case where accessory devices such as Selective Sampler and MicroJet Cryo-Trap are automatically controlled, click on the “Settings” button (E) to display the accessory devices set-up screen. Then check the checkboxes for the needed devices.

(5) Place sample in a sample cup and attach the sample cup to the sampler. Then attach the sampler to the Pyrolyzer. Wait 2 minutes in order to purge the dead space at the sampler – furnace coupling.

A Double-Shot sampler can be used as a Single-Shot sampler. Just slide down the sampler head and secure it at Single-Shot position.

(6) Press “START” button.

(7) The dialog box shown in Fig. 4.8 indicates when the GC, Pyrolyzer, Interface, Selective Sampler and MicroJet Cryo-Trap are all READY. Manually press the sample drop button on the top of the sampler. This releases the cup which drops into the furnace. Immediately click on the “Start” button of the dialog box or hit the “ENTER” key on the PC keyboard.

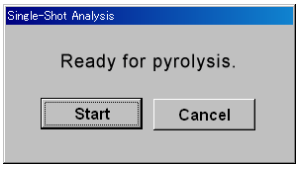


Fig. 4.8 Start dialog box for Single-Shot Analysis

4.3.2 Set-up for Double-Shot Analysis and operation procedures

From the menu bar, select “Tools” – “Analytical Modes” – “Double-Shot Analysis” or click on “Double-Shot Analysis” icon (A) on the toolbar. The set-up screen shown in Fig. 4.9 is displayed. In this analysis mode, thermal desorption (TD) and pyrolysis (PY) can be performed sequentially.

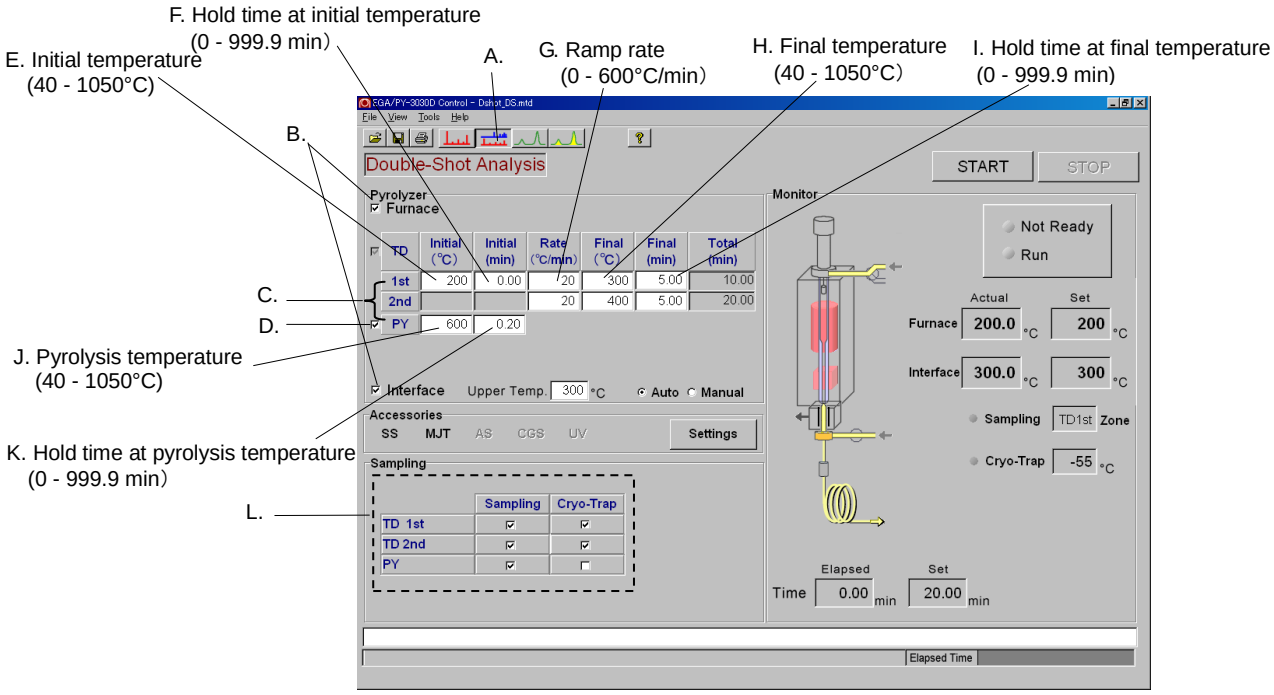


Fig. 4.9 Set-up screen for Double-Shot Analysis

(1) Check the Furnace and Interface checkboxes (B) to turn on heaters.

(2) Fill in parameters for thermal desorption in E through I. If a single step thermal desorption is to be done, enter “0” in the second row in the “Rate” column.

(3) If pyrolysis is to be done following the thermal desorption, check the checkbox in D, and enter parameters for thermal desorption in J through K.

(4) If auto sampling is to be done using the Selective Sampler, and/or if auto cryo-trapping is to be done using the MicroJet Cryo-Trap, check the checkbox for each step desired in L.

(5) Place a sample in a sample cup and attach it to the Pyrolyzer. *When using a Double-Shot sampler the dead space is purged by loosening the air purge nut on the sampler as shown on page 1-5. Tighten it after two minutes. During this period, the GC may generate warning sounds if the carrier gas is controlled by EPC, but if it is within 5 min, the GC will NOT power down.*

(6) Click “START” button.

(7) When the GC, pyrolyzer furnace, interface, Selective Sampler, and MicroJet Cryo-Trap are all READY, the dialog box shown in Fig. 4.10 will be displayed.

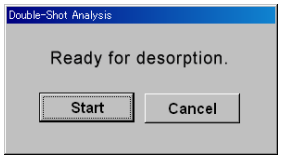


Fig. 4.10 Dialog box showing the start of the desorption program in Double-Shot Analysis

(8) Manually push the Double-Shot sampler down which positions the sample cup in the furnace. Immediately, click on the “Start” button of the dialog box or hit “ENTER” key on the PC keyboard. *This will start the thermal desorption sequence.*

(9) When the thermal desorption program is complete, the dialog box shown in Fig. 4.11 will be displayed. Pull up the Double-Shot sampler; this positions the sample cup above the furnace at near ambient temperature. When the column inlet pressure has stabilized, click “Yes” button. *This will start the GC and the pyrolyzer furnace temperature will be heated to the pyrolysis temperature set (J) in Fig. 4.9.*

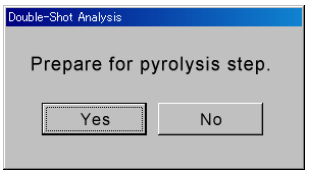


Fig. 4.11 Dialog box showing the end of desorption program in Double-Shot analysis

(10) Thermally desorbed components are then analyzed by GC. When the analysis completes, the GC oven will cool down to the initial temperature.

(11) When the GC sends a READY signal, the dialog box shown in Fig. 4.12 will be displayed. Manually press the sample cup drop button at the top of the sampler. After the sample cup drops into the furnace, immediately click the “Start” button on the dialog box or hit “ENTER” key on the PC keyboard. *This will start the GC.*

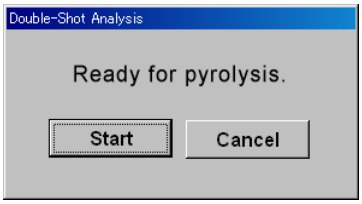


Fig. 4.12 Pyrolysis start dialog box in Double-Shot analysis

This completes the Double-Shot operational sequence. Fig. 4.13 below shows the schematic diagram for a series of operational events as described above.

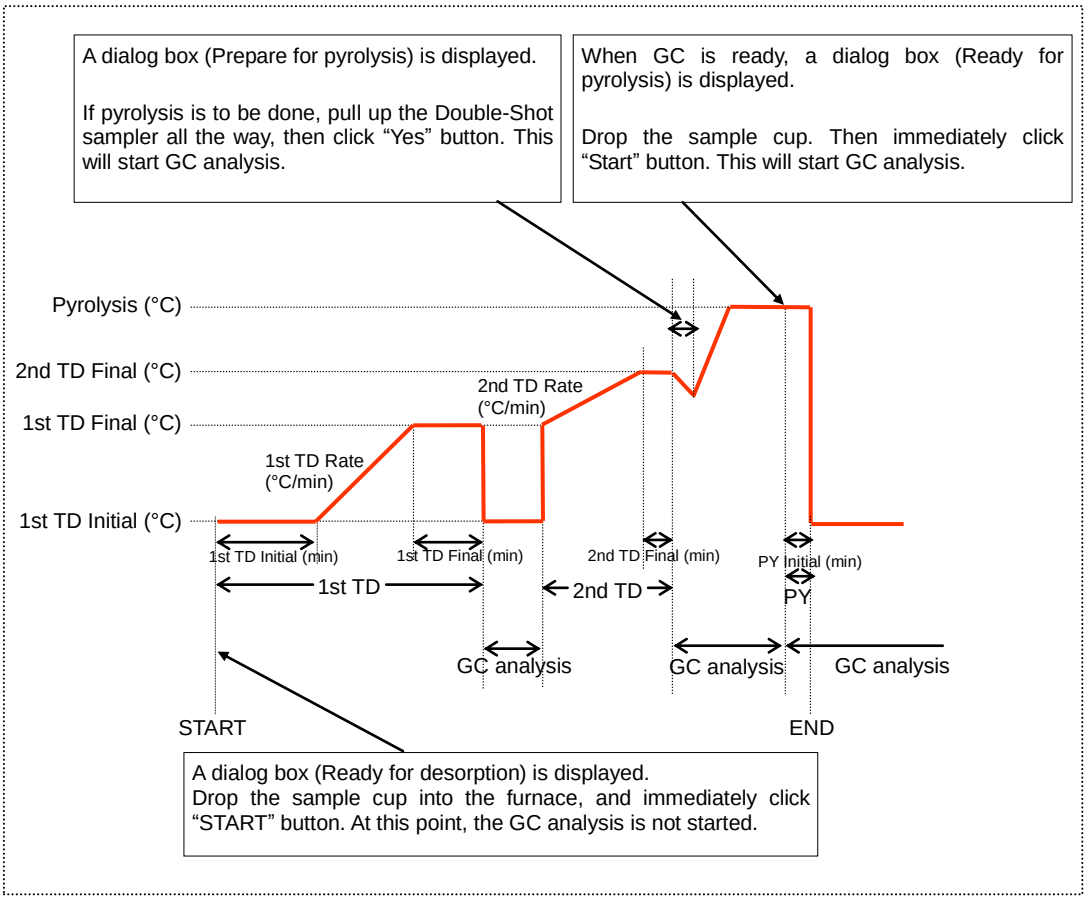


Fig. 4.13 Sequence program in Double-Shot Analysis

4.3.3 Set-up for Direct EGA Analysis and operation procedure

In this analysis mode, an EGA capillary tube (part No.: UADTM-2.5N), is used to connect the GC injector to the detector. Also, the GC oven temperature is set between 250 and 300°C (isothermal)

From the menu bar, select “Tools” – “Analytical Modes” - “Direct EGA Analysis”, or click on the icon for Direct EGA Analysis (A) on the toolbar. The set-up screen shown in Fig. 4.14 is displayed.

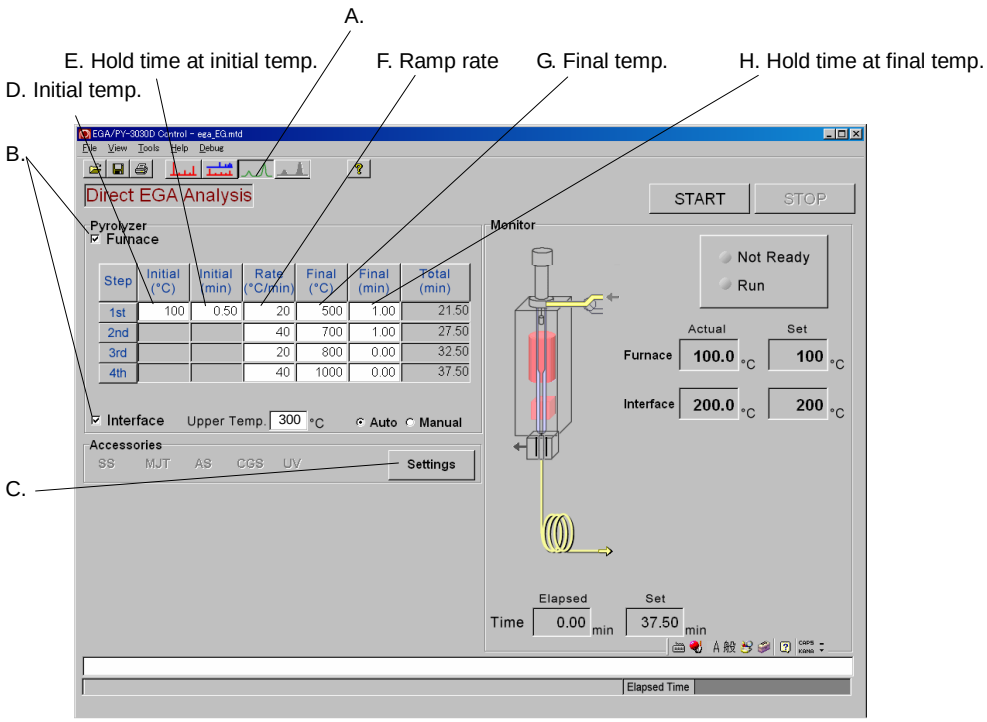


Fig. 4.14 Set-up screen of Direct EGA Analysis

(1) Check the checkboxes for Furnace and Interface (B) to turn on the heaters.

(2) Enter parameters for Step 1 in D through H.

(3) Type in parameters for Step 2. Entering “0” for Rate (°C/min) in subsequent rows will stop executions of the steps beyond. Fig. 4.15 illustrates a schematic diagram for a 4-step heating sequence. In direct EGA analysis, a one-step sequence (step 1 only) is normally used.

(4) When the sequence is automated using the Selective Sampler, click on “Settings” (C) to open a dialog box and check the checkbox for SS.

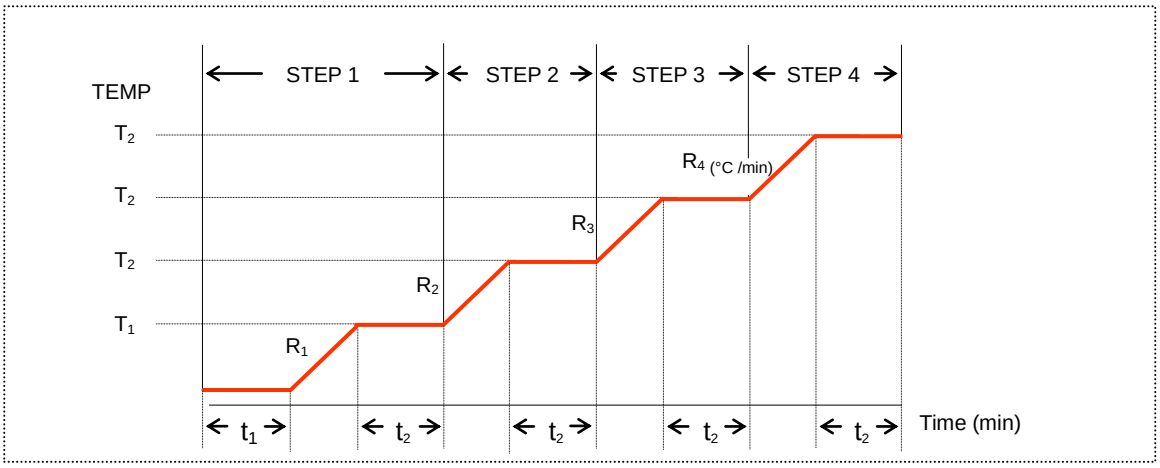


Fig. 4.15 EGA Multi-step heating program

(5) Place a sample in a sample cup and attach it to the sampler. Attach the sampler to the pyrolyzer and wait two minutes in order to purge the air in the dead space before proceeding.

(6) Click “START” button.

(7) When the GC, pyrolyzer furnace, interface, and Selective Sampler are all READY, the dialog box shown in Fig. 4.16 will be displayed. Immediately drop the sample cup into the furnace; click the “Start” button in the dialog box or hit “ENTER” key on the PC keyboard.

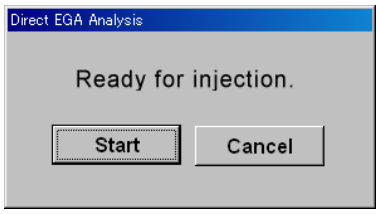


Fig. 4.16 Dialog box showing start of Direct EGA Analysis

4.3.4 Set-up for Heart-Cut EGA Analysis and operation procedure

Heart-cut EGA analysis requires a Selective Sampler. If low boiling components below C_{15} are to be analyzed, a cryo trap is required. Frontier Labs MicroJet Cryo-Trap is recommended.

Heart-Cut EGA analysis is based on evolved gas curve (EGA thermogram) obtained by Direct EGA Analysis. The temperature zone (elution time range) to be analyzed is based upon the EGA thermogram. Each EGA thermal zone is automatically introduced into the GC column utilizing a Selective Sampler. For example, six EGA thermal zones are observed in the EGA thermogram (100 to 600°C (20°C/min) of a ceramic composite material – see Fig. 4.17.

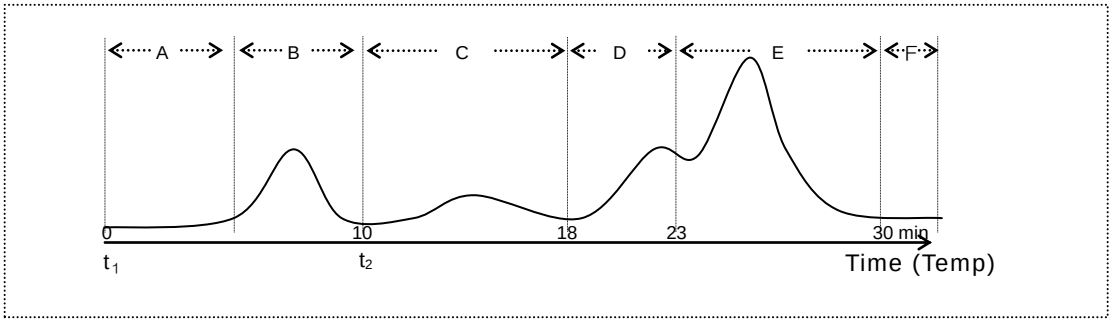


Fig. 4.17 EGA thermogram of a ceramic composite

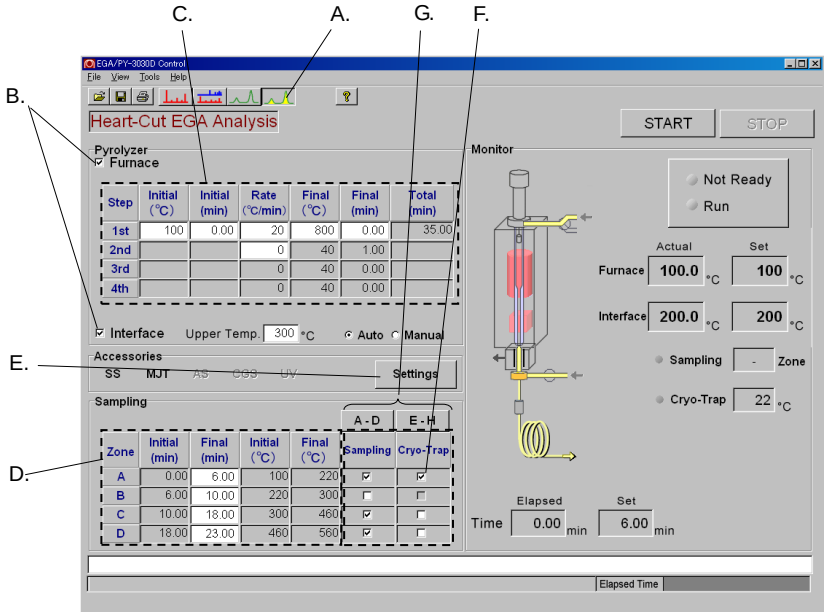


Fig. 4.18 Set-up screen for Heart-Cut EGA Analysis

From the menu bar, select “Tools” – “Analytical Modes” – “Hear-Cut EGA Analysis” or click on the icon for “Heart-Cut EGA Analysis (A) on the toolbar. The set-up screen shown in Fig. 4.18 is displayed.

(1) Check the checkboxes for Furnace and Interface (B) to turn on heaters.

(2) Type in the parameters for a heating program in the table marked by C, in the same manner as described for Direct EGA analysis.

(3) Determine the end time of each thermal zone using the EGA curve in Fig. 4.17. Enter these times (min) in the table marked as D. When using a mass spectrometer as a detector, the elution temperatures of the target components can be easily determined by using extracted ion chromatograms of the target compounds. *The number entered in Final (min) of each zone is automatically entered as the Initial (min) of the next zone. The Initial time of Zone A starts from 0.0 min (fixed). Temperatures of the Initial (°C) and Final (°C) are automatically calculated and displayed.*

(4) The Selective Sampler and MicroJet Cryo-Trap are automatically controlled by the software. “Click” on the “Settings” button (E) to display the accessory devices set-up screen. Then check the checkboxes for desired devices.

(5) Whether a zone is analyzed or vented and whether or not auto cryo trapping is used are all set through using the checkboxes for desired functions (F).

(6) The button marked by G allows you to switch zone displays between A through D and E through H.

(7) Place a sample in a sample cup and attach it to the sampler; attach the sampler to the pyrolyzer. Wait 2 minutes to purge air in the dead space.

(8) Click “START” button.

(8) When the GC, furnace, interface, Selective Sampler, and MicroJet Cryo-Trap are ready, the dialog box in Fig. 4.19 will be displayed. Push the Double-Shot sampler down to position the sample cup in the furnace. Click the “Start” button or hit the “ENTER” key on the PC keyboard.

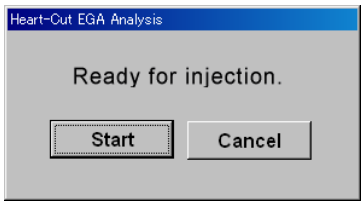


Fig. 4.19 Starting dialog box for Heart-Cut EGA Analysis

This completes the operational sequence for a Heart-Cut EGA analysis. Fig. 4.20 below shows the schematic diagram for a series of operational events as described above.

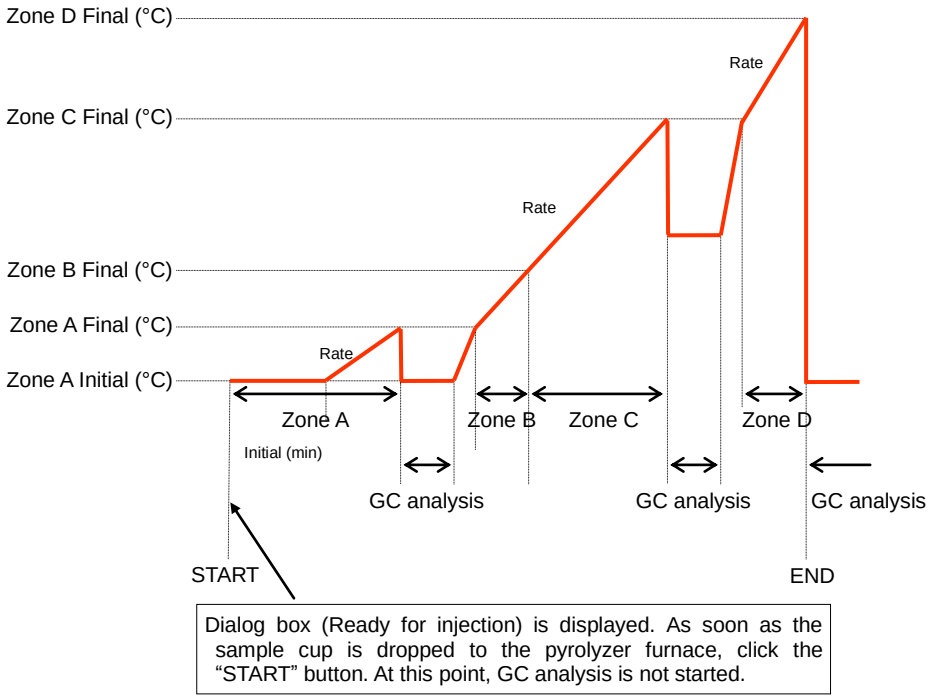


Fig. 4.20 Sequence diagram of Heart-Cut EGA Analysis
(In this example, Zones A through D are sampled, but Zone B is not analyzed.)

4.4 OTHER FUNCTIONS

4.4.1 Saving/loading analysis set-ups

The set-up methods can be saved using a filename. When all the set-ups are completed, choose “File” – “Save As”, and type in a filename to save the set-ups in a file. As shown in Table 4.1, a suffix is added automatically to the filename. The suffix depends on the analytical mode. For example, in the case of the Single-Shot analysis, if the filename is specified as Test.mtd, the actual filename saved will be Test_SS.mtd. To overwrite a file with the current file, select “File” – “Save”.

Table. 4.1 Suffix added to filename

Analytical mode	Suffix added to filename
Single-Shot	SS
Double-Shot	DS
Direct EGA	EG
Heart-Cut EGA	HC

4.4.2 Temperature calibration of the pyrolyzer furnace and other set-ups

From the menu bar, choose “Tools” – “Maintenance”. A dialog box shown in Fig. 4.21 is displayed. Type in “3030” for password, and press the “OK” button. The set-up screen shown in Fig. 4.22 will be displayed.

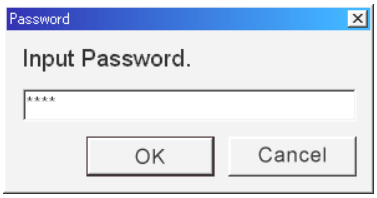


Fig. 4.21 Entering password

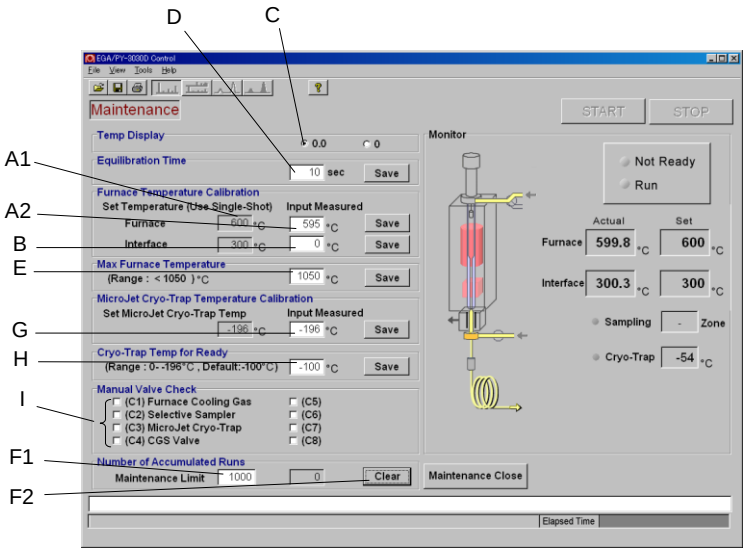


Fig. 4.22 Screen shot of temperature calibration of pyrolyzer and other set-ups

4.4.2.1 Temperature calibration of pyrolyzer furnace

The temperature calibration of the pyrolyzer furnace is done by entering the actual temperature measured by other means. Because the pyrolyzer furnace temperature greatly influences analytical results, you should familiarize yourself with this operation and verify the calibration on a periodic basis.

Ensure that the pyrolyzer furnace temperature is stabilized at the set temperature. The set temperature is shown in A1, but this is automatically entered with the temperature defined in the Single-Shot analysis mode.

(1) Enter the actual temperature measured in A2, using a reliable thermometer of your choice.

(2) Click the “Save” button to finish the temperature calibration.

(3) Make sure that the temperature displayed matches the actual temperature measured as Fig.4.23. If not, repeat the operations (1) through (3) described above.

(4) The interface temperature calibration can be done in a similar manner by entering the actual value measured in B.

CAUTION

When performing a temperature calibration, follow the instructions in the manual to measure the correct temperature. If wrong values are entered, the control of temperature is not properly done.

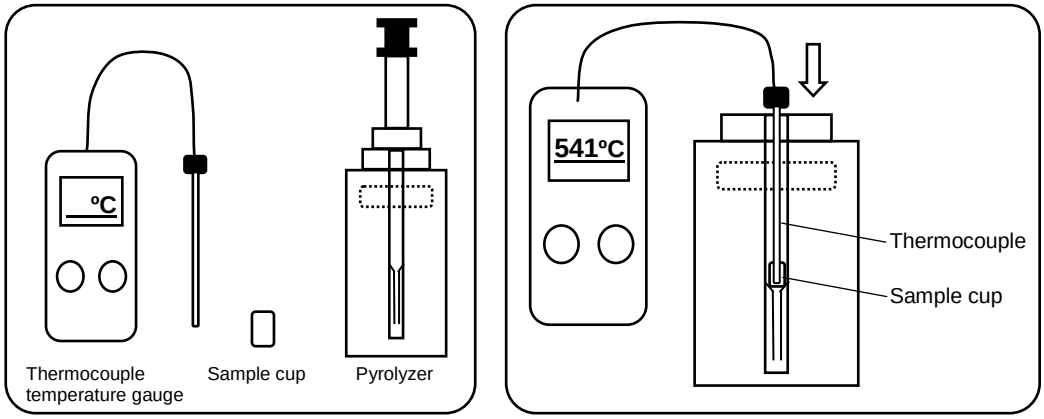


Fig. 4.23 Measurement of actual temperature using thermocouple

4.4.2.2 Other set-ups for Pyrolyzer furnace

C: The temperature display can be switched between the first decimal place and integer.

D: The equilibration time between the time for the furnace and interface temperatures to reach the set points and the time before starting the next operation can be changed.

E: Maximum input temperature for all analytical modes

F1: Sets the upper limit for the number of analytical runs. A message urging you to perform maintenance of the instrument will be displayed when the total accumulated runs reach the number set here. After performing the necessary maintenance (septum, liner, needle, etc.) is finished, click on the “Clear” button (F2).

4.4.2.3 Checking valves operations

Valves which are controlled by the pyrolyzer (EM valves for Pyrolyzer cooling air, Selective Sampler, MicroJet Cryo-Trap, and Carrier Gas Selector) can be functionally tested (i.e., opened and closed). This is done by checking the checkboxes in Manual valve check (I). Closing the Maintenance screen will bring all valve operations back to normal.

4.4.2.4 Set-up MicroJet Cryo-Trap

G: Allows calibration of temperature sensor.

H: Sets the cryo trap temperature. Keeping the temperature equal or lower than -170°C for 90 seconds will activate the operations of the MicroJet Cryo-Trap.

4.4.3 Printing set-ups

Set-ups saved in the currently loaded file can be printed. Go to the menu bar, select “File” – “Print”.

4.4.4 Set-up the Py Conditioning Table

From the menu bar, choose “Tools” – “Py Conditioning Table”. The Py conditioning table shown in Fig. 4.24 is displayed. This allows you to perform the conditioning of the pyrolyzer furnace. Enter desired date and time, pyrolyzer furnace temperature, and interface temperature, and click the “Run” button to activate the timer. In the example shown in Fig. 4.24, at 21:00 on March 10, 2011 the pyrolyzer furnace temperature will be set to 700°C and the interface temperature will be 300°C. At 0:00 on March 11, 2011 the pyrolyzer furnace temperature will be set to 100°C and the interface will be set to 200°C. Note that while this is running, no other methods can be changed.

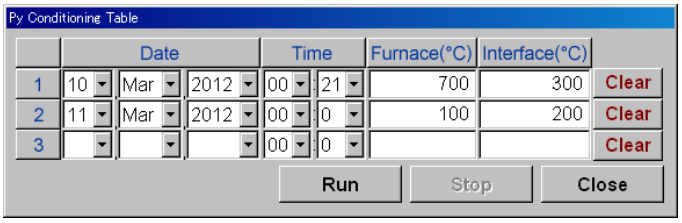


Fig. 4.24 Set-up the Py Conditioning Table

CHAPTER 5 FOUR ANALYSIS METHODS AND SAMPLE PREPARATION

Four analysis methods (Single-Shot, Double-Shot, evolved gas analysis, and Heart-Cut EGA analysis) are described. Refer to Chapter 4 for details about settings and operations. Sampling techniques are also described here.

5.1 SINGLE-SHOT ANALYSIS

This method is also called flash pyrolysis. The sample is placed in a sample cup which is then attached to the sampler. The sampler is attached to the Multi-Shot pyrolyzer. Pyrolysis occurs when the cup is released and “free falls” into the pyrolyzer furnace which is at the pre-selected pyrolysis temperature. The gases generated in the furnace are swept into the splitter by the carrier gas. The sample vapors are split; a portion going to the analytical column and a portion exiting via the split vent. A typical instrument configuration for a Py-GC/MS system is shown in Fig. 5.1.

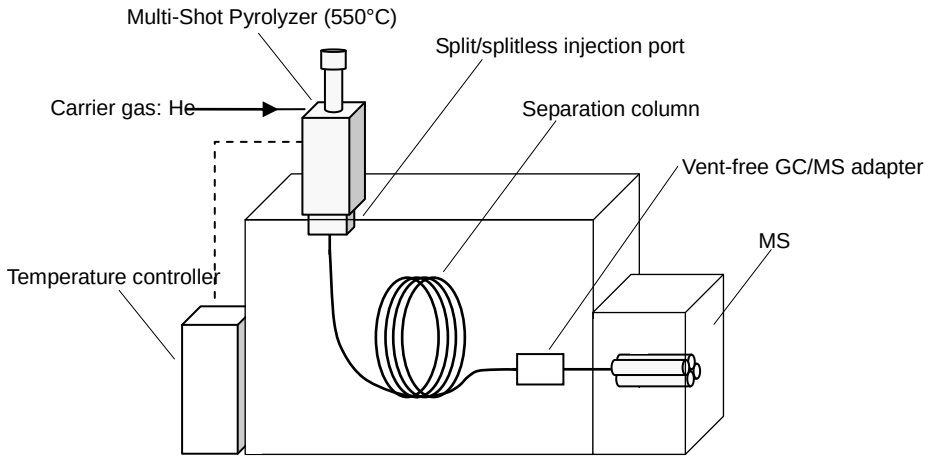


Fig. 5.1 Typical instrument configuration for Py-GC/MS

An example of a Single-Shot analysis using the polystyrene solution is described below. The test sample vial contains 2.5 mg of polystyrene and approx. 5% (wt/wt) of methyl stearate. Adding 0.5 mL of dichloromethane, benzene, or toluene makes a test solution (5 µg/µL). The sample used here is a dichloromethane solution of polystyrene (5 µg/µl) with methyl stearate.

STEP1 Install the 30 m capillary column provided with the 3030D pyrolyzer.

STEP2 Set the pyrolysis and GC conditions (see Fig. 5.3)

(1) Set the interface temperature (ITF TEMP) to “Auto” mode.

(2) Set the pyrolysis temperature in “Single-Shot Analysis” screen.

STEP3 Prepare the sample

(1) Place 5 µL of the sample in an Eco cup SF and evaporate the solvent.

(2) Attach a stick (Eco stick SF) to the sample cup and fix the assembly to the sampler. Position the slider of the sampler in the down position. Attach the sampler to the pyrolyzer and wait for 1 min. (Fig. 5.2B) so that the residual air can be purged.

STEP4 Click the "START" button when the temperatures and pressures are stabilized.

When the "Ready for injection" dialog box is displayed, press the sample drop button located at the top of the sampler. This releases the cup and it drops into the furnace (Fig. 5.2B). Immediately click the "Start" button. GC analysis will start.

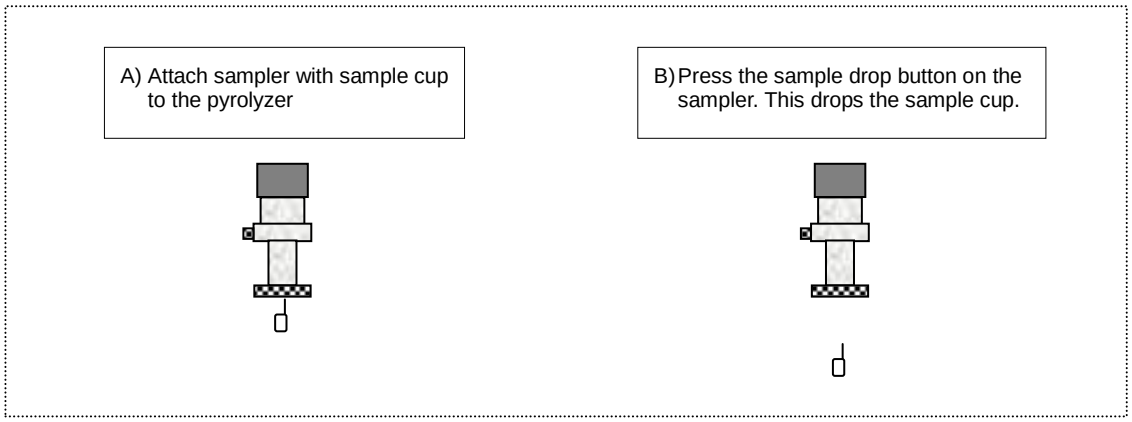


Fig. 5.2 Sample introduction procedure in Single-Shot analysis

The pyrogram in Fig. 5.3 has peaks for the polystyrene pyrolyzates: monomer, dimmer, trimer, and methyl stearate (Fig. 5.3).

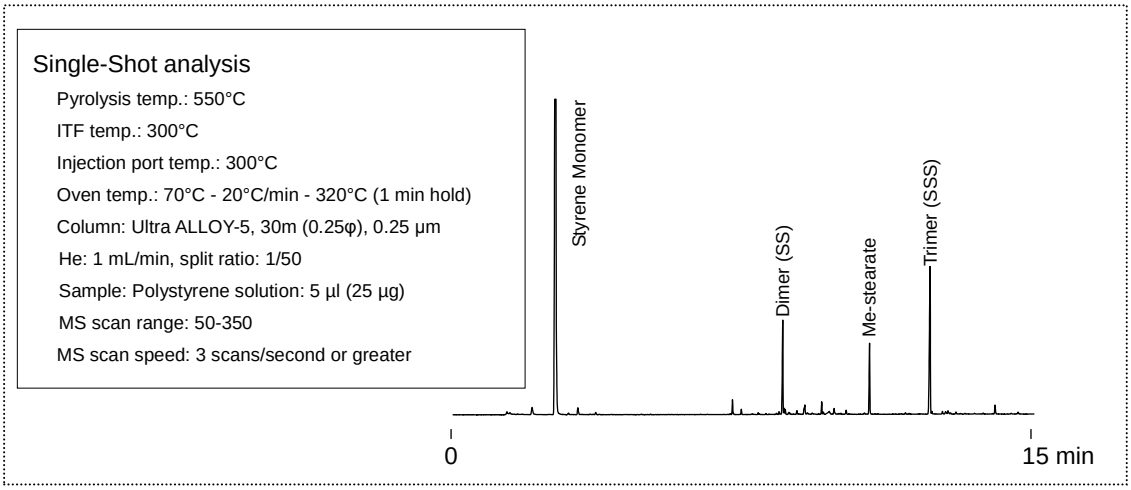


Fig. 5.3 Pyrogram of polystyrene obtained by Single-Shot analysis

5.2 EVOLVED GAS ANALYSIS (EGA)

When a polymeric sample is heated, volatile components contained in the polymer are released and ultimately, the polymer is degraded. The result is a plot of sample temperature vs. detector response. Such a plot has been termed a 'thermogram'. The thermogram provides an indication of the sample complexity. Using a combination of extracted ion chromatograms and average mass spectra, the thermal region containing compounds of interest can be determined

A typical instrument configuration for EGA is shown in Fig. 5.4. The pyrolyzer and a detector are connected via a deactivated EGA tube (L=2.5 m, id.= 0.15 mm). The GC oven is normally held at 300°C. When the sample is heated, the thermally desorbed components and gases arising from the decomposition of the polymer sample are released and detected as shown in Fig. 5.6.

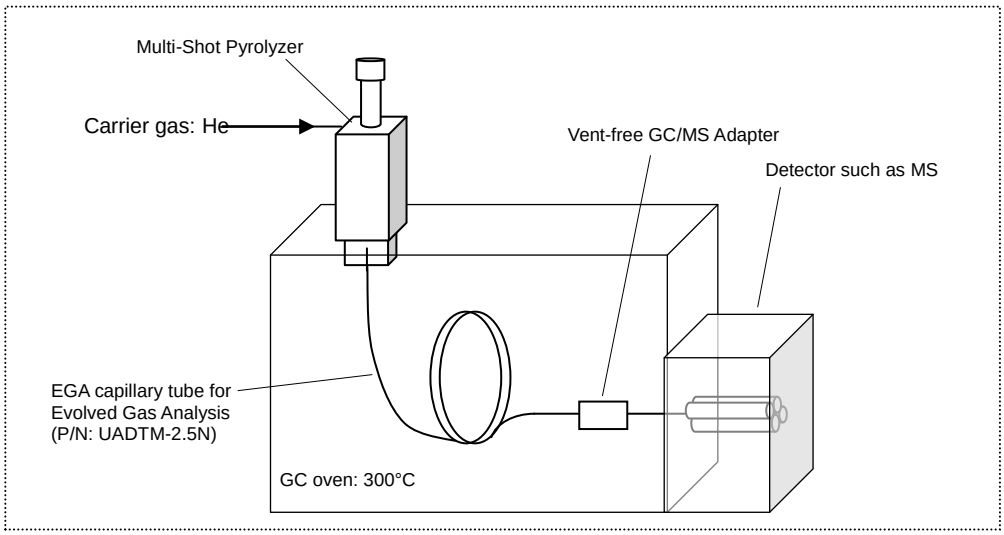


Fig.5.4 Typical instrument configuration for Evolved Gas Analysis

The standard polystyrene solution is used to demonstrate EGA. This sample is a dichloromethane solution of polystyrene (5 µg/µl) with methyl stearate added. An analysis example is described below. From the EGA thermogram obtained, the thermal desorption and pyrolysis conditions can be determined.

STEP1 Installing EGA capillary tube for evolved gas analysis

Attach the supplied EGA capillary tube (id.=0.15 mm, L=2.5 m) for evolved gas analysis.

STEP2 Setting analytical conditions for pyrolyzer and GC (see Fig. 5.6)

(1) Set the interface temperature (ITF TEMP) to "Auto" mode.

(2) Set the furnace temperature in the "Direct EGA Analysis" screen.

STEP3 Loading sample

(1) Place 5 µL of the sample into an Eco cup SF and evaporate the solvent

(2) Attach a stick (Eco stick SF) to the sample cup and fix it to the sampler. Push the slider of the sampler down and attach the sampler to the pyrolyzer. Wait for 1 min (Fig. 5.5A).

STEP4 Click the "START" button when temperatures and pressures are stabilized.

When the "Ready for injection" dialog box is displayed, press the sample drop button located at the top of the sampler. This drops the sample cup into the furnace (Fig. 5.5B). Immediately click the "Start" button. The GC analysis will start

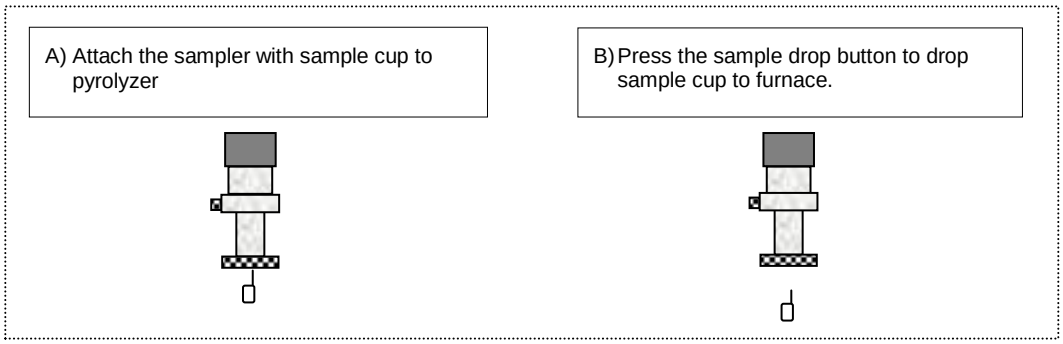


Fig. 5.5 Sample introduction procedure in EGA analysis

The analysis result is shown in Fig. 5.6. From this EGA thermogram, the following can be observed.

(1) Volatile components elute at temperatures between 50°C and 200°C.

(2) The polymer decomposition starts about 350°C, and ends at 500°C.

Therefore, the EGA results indicate that the volatiles can be thermally desorbed between 50°C to 200°C, while the pyrolysis temperature for the polymer is 550°C. *Our experiences indicate that optimal pyrolysis temperature should be set 50°C higher than the end of the EGA signal. This helps ensure good reproducibility.*

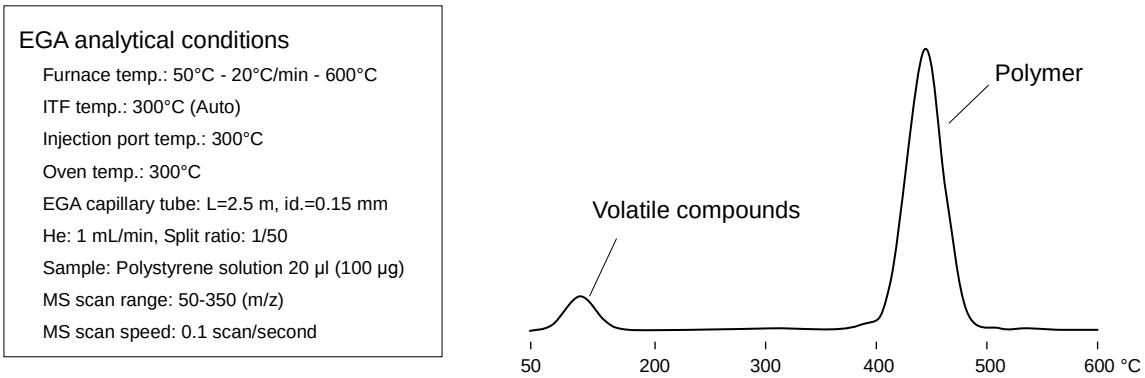


Fig. 5.6 EGA Thermogram

5.3 DOUBLE-SHOT ANALYSIS

This is a two-step analysis in which volatiles are analyzed using thermal desorption technique, and the residual polymeric fraction is then pyrolyzed. A typical instrument configuration for the Py-GC/MS is shown in Fig. 5.1. The sample is the supplied polystyrene solution. The analytical conditions for thermal desorption and flash pyrolysis are determined using the EGA thermogram shown in Fig. 5.6.

STEP1 Install the separation column (contained in the package)

STEP2 Set the analytical conditions for the pyrolyzer and GC (see Fig. 5.8)

(1) Set the interface temperature (ITF TEMP) to “Auto” mode.

(2) Set the furnace temperature in “Double-Shot Analysis” screen.

STEP3 Loading sample

(1) Place 5 µL of the sample in an Eco cup LF and evaporate the solvent

(2) Attach a Double-Shot stick (Eco stick DF) to the sample cup and fix it to the sampler (Fig. 5.7A). Pull the slider of the sampler to the up position and attach the sampler to the pyrolyzer (Fig. 5.7B). Loosen the air purge nut located on the side of the sampler and leave it open for 2 min. Tighten the air purge nut before proceeding.

STEP4 Click the “START” button when the temperatures and pressures are stabilized.

When the “Ready for injection” dialog box is displayed, push the sampler slider all the way down. This positions the sample cup in the furnace. Click the “Start” button (Fig. 5.7C). The thermal desorption program will start. Pull the sampler slider to the up position. When the TD GC analysis finishes, the “Prepare for pyrolysis step” dialog box is displayed. Click “Yes” button to start the GC analysis.

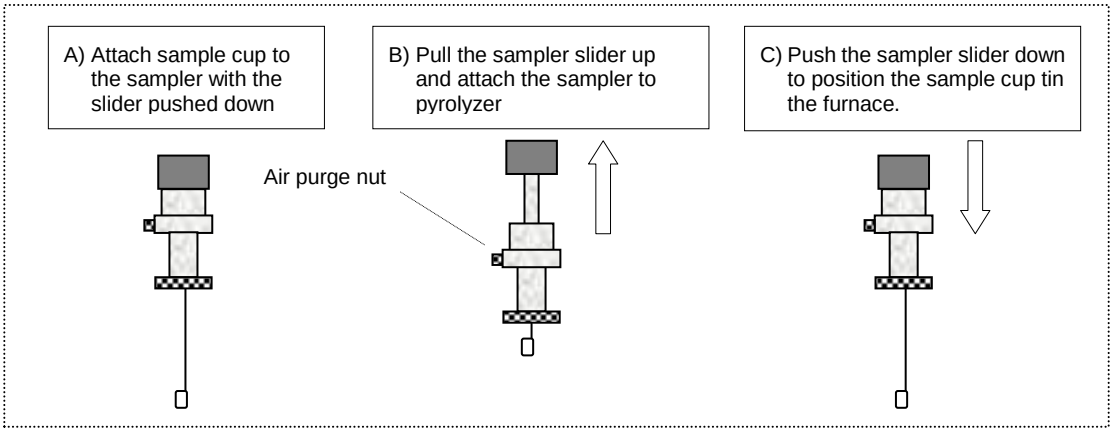


Fig. 5.7 Sample introduction procedure for Double-Shot analysis

STEP5 Flash pyrolysis

When the GC analysis of the thermally desorbed compounds is completed and the “Ready for pyrolysis” dialog box is displayed, press the sample drop button located at the top end of the sampler to drops the sample cup to the furnace. Now, immediately click “Start” button to start the GC analysis.

Fig. 5.8 A-1) and Fig. 5.8 A-2) shows typical analysis results obtained by the Double-Shot method, i.e., results of thermal desorption and flash pyrolysis.

For comparison, the analytical result for the same sample obtained by the Single-Shot method is shown in Fig. 5.8B. The pyrogram contains peaks of both A-1 and A-1 combined

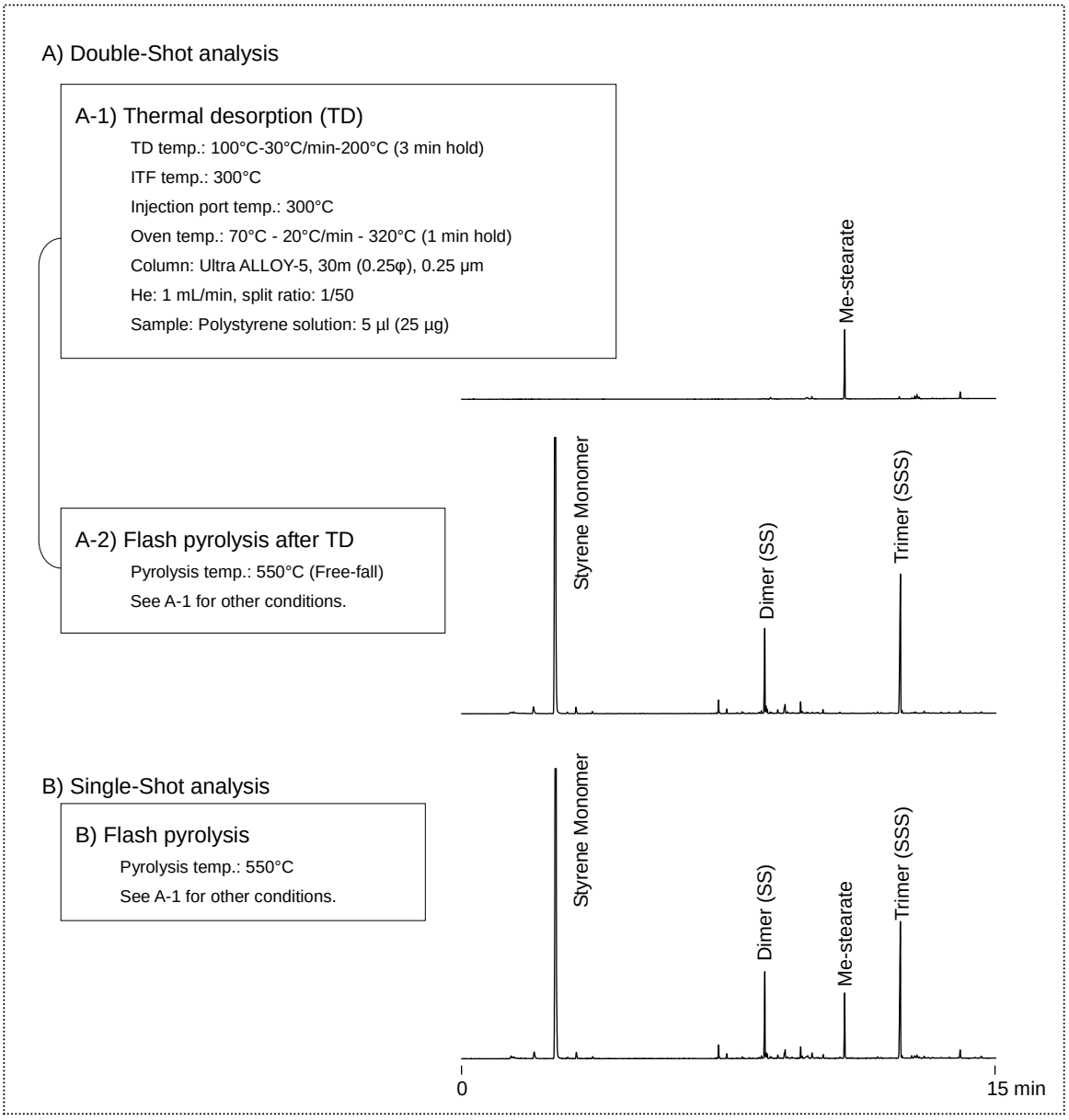


Fig. 5.8 Comparison of results obtained between Double-Shot and Single-Shot methods

5.4 HEART-CUT EGA ANALYSIS

This is a technique in which gases eluted from any temperature zone of the EGA thermogram is selectively introduced to the separation column for analysis (GC/MS). This analysis requires a Selective Sampler (optional). Also for analysis of low boiling evolved gases with C_{15} or less requires a cryo trap. Frontier Laboratories MicroJet Cryo-Trap is highly recommended. The setup for a typical Heart-Cut EGA analysis is shown in Fig. 5.9. By using the Selective Sampler and MicroJet Cryo-Trap, a maximum of eight temperature zones can be automatically analyzed by GC/MS.

Here, ceramic composite material is analyzed. Based on its EGA thermogram (Fig.5.10), four temperature zones are determined and the evolved gases from each temperature zone are analyzed by GC/MS.

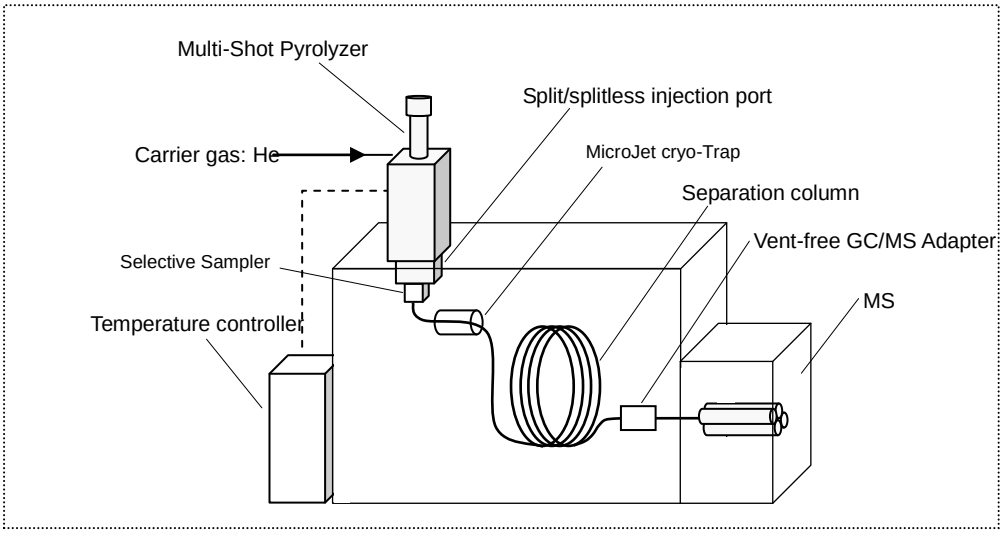


Fig. 5.9 Instrument setup for a typical Heart-Cut EGA analysis

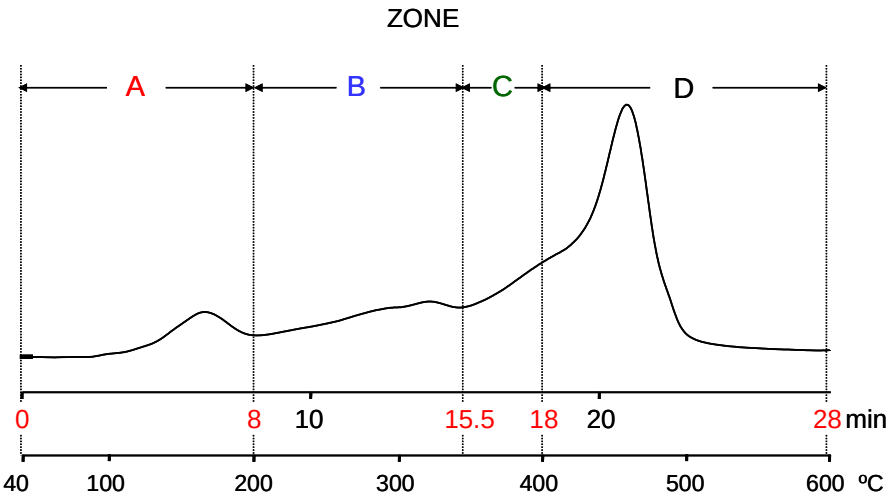


Fig. 5.10 EGA thermogram of a ceramic composite

STEP1 Instrument set up

Attach Selective Sampler and separation column.

STEP2 Setting analytical conditions for pyrolyzer and GC (see Fig. 5.11)

(1) Set ITF TEMP to “Auto” mode.

(2) Set the furnace temperature and others in “Heart-Cut EGA Analysis” screen.

STEP3 Loading sample

(1) Weigh out sample and place it in an Eco cup SF.

(2) Attach a stick for Double-Shot (Eco stick DF) to the sample cup and fix it to the sampler (Fig. 5.7A). While the slider of the sampler pulled up all the way, attach the sampler to the pyrolyzer (Fig. 5.7B). Then loosen the air purge nut located on the side of the sampler and keep it loosened up for a few minutes then tighten it back to close

STEP4 Click “START” button when temperatures and pressures are stabilized.

When “Ready for injection” dialog is displayed, slide down the sampler slider to introduce the sample cup into the furnace, then click “Start” button. The controlled heating program will start. When the program for each temperature zone is completed, GC analysis will start.

The chromatograms of evolved gases from each temperature zone obtained by GC/MS are shown in Fig. 5.11. As shown here, detailed analysis of evolved gases can be accomplished by analyzing each temperature zone of EGA thermogram.

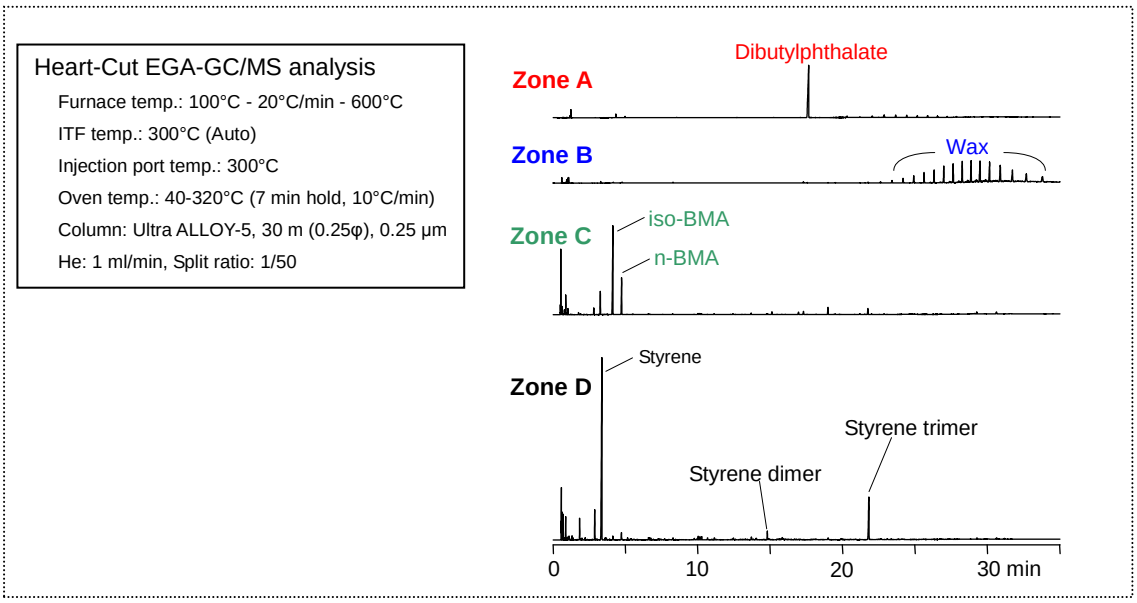


Fig. 5.11 Chromatograms of four zones

5.5 SAMPLE PREPARATIONS

In the Single-Shot analysis (flash pyrolysis) using Py-GC/MS, a sample form, shape and condition affect results as well as the reproducibility. If the sample is soluble in a solvent, it is recommended to dissolve it in the solvent, place in a sample cup, and evaporate the solvent to form a film on the surface of the sample cup. If the sample is insoluble in solvents, it is recommended to pulverize it into a fine powder. The amount of sample needed is usually 0.1 mg or less. Thermal desorption and EGA analysis may require more depending on target compounds.

Granular or chunk polymer samples are not uniform in sample shapes, and have low thermal conductivities, resulting in a poor reproducibility of pyrograms. If this is the case, you may need to use a small amount of sample or to make a thin film or powder.

There are four methods to make a film or powder out of solid samples. Out of these methods A1), A2) and A3) are easiest; however, A1) and A2) are recommended in the point of view of obtainment of good reproducibility.

CAUTION

When preparing samples, organic solvents and knife or blade with sharp edge are used. For your safety, wear protective gloves and safety goggles.

A1) Preparing a thin film

This method involves dissolving a sample in a solvent, followed by placing a small amount of it in a sample cup then evaporating the solvent. Because this method provides a uniform thin film, the reproducibility of results is excellent; nonetheless, it does not work for non-soluble samples such as thermosetting polymers.

A2) Using pulverizing tool (Polymer Prepper from Frontier Laboratories Ltd.)

Using a Polymer Prepper (a nickel coated special file), fine powers with the particle size of 0.1 mm can be prepared easily and rapidly. Fine and coarse surfaces are available in a single tool as your choice. After use, applying the standard special cleaning tape on the surface can clean the surface completely for repeated use. This method is not for elastic materials such as rubber. For elastic materials, use either method A1) or A4).

A3) Using a cutter knife

Using a sharp knife or surgical knife, a chunk of sample material is sliced into thin flakes. It is difficult to obtain good uniform slices, thus giving a poor reproducibility.

A4) Freeze-grinding using liquid nitrogen and metal ball

This method involves pulverization of a liquid-nitrogen frozen material using a metal crushing ball. Note that the pulverized sample must be retrieved only after the pulverizing chamber is at room temperature in order to avoid moisture condensation. Also, caution must be exercised that the pulverized sample may be contaminated with a small amount of metal powders, which may cause reduce data quality.

CAUTION

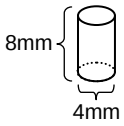
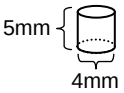
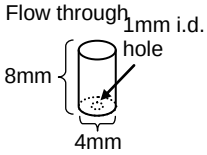
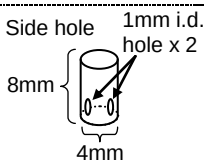
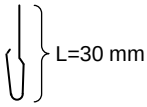
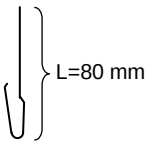
Care should be exercised when handling liquid nitrogen. There is a risk of frostbite and suffocation when a large amount of liquid nitrogen is spilled out. For your safety, wear protective gear such as leather gloves, safety goggles, etc. when using a cryogenic mill, read the manual thoroughly before attempting to use it.

5.6 SELECTION OF SAMPLE CUPS AND STICKS

The types of Eco-cups and Eco-sticks are selected from Table 5.1 depending on your analytical purpose and the technique that you use.

Eco-cups and Eco-sticks are made of stainless steel; the entire surface has been deactivated by coating with fused silica on the surface. The wall thickness is 0.1 mm for all.

Table. 5.1 Selection of sample cups and sticks

Type / Appearance	Name	Part Number	Volume (μL)	Compatibility with AS-1020E	Description
Eco-cups					
Regular L 	Eco-cup LF	PY1-EC80F (100 pcs)	80	Yes	For all applications except for Micro UV Irradiator (UV/Py-GC/MS). Optimum pyrolysis temperature is 30ºC lower than Eco-cup SF for most polymeric materials due to large surface area. This is useful for samples with relatively large volumes.
Regular S 	Eco-cup SF	PY1-EC50F (100 pcs)	50	No	For all applications except for Micro UV Irradiator (UV/Py-GC/MS). This cup provides slightly better pyrogram reproducibility in flash pyrolysis-GC mode than Eco-cup LF.
Flow through 	Eco-cup LHF	PY1-EC80HF (20 pcs)	80	No	This cup provides better peak shape resolution of volatile compounds (C3-10) than regular cups. And it prevents secondary reactions under a low carrier gas flow, e,g. 10mL/min.
Side hole 	Eco-cup UV	PY1-EC80UV	80	Yes	Used for analysis with Micro UV Irradiator (UV/Py-GC/MS)
Eco-sticks					
Short 	Eco-Stick SF	PY1-ES10F (50 pcs)	---	---	Used for manual Single-Shot, EGA or Heart-Cut EGA analysis.
Long 	Eco-Stick DF	PY1-ES20F (50 pcs)	---	---	Used for manual Double-Shot analysis.

CHAPTER 6 MAINTENANCE

A pyrolysis GC system is significantly different from a standalone GC in that each component of the system must work in harmony with the other components in the system. The reliability of the analytical results depends on the condition of each component; therefore, regular maintenance is extremely important.

There is a risk of severe burn. Before starting maintenance work, make sure that the pyrolyzer furnace and interface temperatures are below 50°C.

6.1 ROUTINE MAINTENANCE

The three components which need attention are the sample cups, the quartz pyrolysis tube, and the interface needle.

On the support page of the Frontier Lab's web site, several maintenance videos are available for your viewing. These how-to videos will help you perform maintenance procedures. Please visit our web site at www.frontier-lab.com.

6.1.1 Cleaning sample cups (Eco-cup)

6.1.1.1 About reusing an Eco-cup F

(1) Depending on the types of samples and analytical requirements, sample cups may be reused.

(2) As shown in Fig. 6.1, to reuse a sample cup, red-heat the sample cup for 2 seconds using a small torch or bake it in a muffle furnace at 600°C for one hour. Depending on the nature of the samples, polymeric residues (carbon) and inorganic residues may remain in the cup. If this is the case, physically remove all the residues using a cotton swab and the like, then clean it using volatile solvents such as acetone and dichloromethane and dry it.

(3) Tests conducted by Frontier Laboratories with polyethylene and polystyrene show that reproducible pyrograms (n=5) are obtained when the cups are cleaned as described above.

(4) The decrease of deactivation of the sample cup after use depends on the type of samples analyzed, the amount of sample analyzed, the exposure time at elevated temperature and heating time at high temperatures when cleaning. *The longer it is heated at high temperatures, the greater the loss of inertness.*

(5) When reusing the cups, analysts must be able to judge whether the cups are acceptable for their analytical purposes. In particular, when analyzing polar compounds or those susceptible to thermal decomposition such as brominated flame retardants (PBDE etc.), extra care is required when reusing sample cups. Analytical precision is strongly dependent upon overall system inertness and thermal homogeneity.

(6) Sample cups can be cleaned with a solvent. The procedure is described in section 6.1.1.3.

6.1.1.2 Cleaning by red-heating

This is very simple and works well in most cases.

(1) Remove all the residues left in the cup using a cotton swab. When using a pair of tweezers, do not scratch the inner surface of the sample cup.

(2) As shown in Fig. 6.1, using a small torch heat the cup for 1 or 2 seconds until it glows slightly red. If heated longer, it will degrade the overall inertness of the surface. This will shorten the life of the cup.

(3) When the cup cools (approx. 20 sec) set it in a sample cup holder. Be careful *not to burn your fingers.*

Fig. 6.1 Red-heating with a small torch

CAUTION

When using a flame, make sure there is no flammable materials around and work only in a well-ventilated area. Wear safety goggles for your safety.

6.1.1.3 Solvent cleaning

When analyzing samples containing or forming polar compounds, sample cups should not be cleaned using heat.

(1) Remove sample residues remained in the sample cups using a cotton swab. If a pair of tweezers is used, do not to scratch the inner surface of the sample cup.

(2) Place sample cups to be cleaned in a beaker containing a solvent. This is to dissolve or swell residual polymer. Verify that the solvent used will dissolve or swell the polymer sample.

(3) Apply ultrasonication for one minute.

(4) Remove the solvent from the sample cups, and clean the inner wall of the cup with a cotton swab and the like.

(5) Place the cups in a beaker containing the solvent and swirl them for a short period of time.

(6) Replace the solvent and repeat (3) through (5) two to three times. Dry the cups in an oven before use.

CAUTION

When handling solvents, ensure there is no ignition source around and work only in a well-ventilated area. Wear safety goggles for your safety.

6.1.2 Replacing and cleaning quartz pyrolysis tube

CAUTION

The pyrolyzer is equipped with a very sharp interface needle. For your safety, work only in a stable place and wear protective gears such as safety goggles.

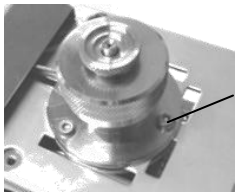
6.1.2.1 Replacing the quartz pyrolysis tube

The Quartz pyrolysis tube may be contaminated by the deposition of inorganics and tars. It is recommended that the tube be cleaned or replaced after a series of analyses. While the number of analysis that can be performed is clearly a function of the sample, the analytical requirements and the furnace temperature, Frontier Laboratories recommends that the liner be inspected after 200 injections or whenever there is a loss of analytical precision. The instruction for replacing the contaminated pyrolysis tube with a new one is provided below.

CAUTION

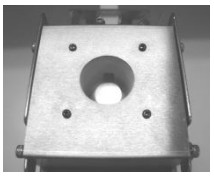
1. Work only after the pyrolyzer furnace is fully cooled down.
2. Broken quartz pyrolysis tube has very sharp edges. Wear safety goggles and gloves for your safety.

(1) Loosen the three securing screws of the sampler base. The O-rings do not usually require replacement, but look for contamination or cuts.

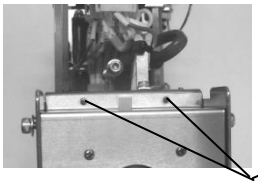


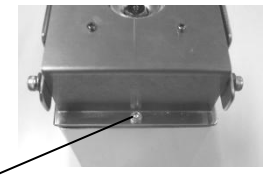
Securing screws (3 pcs)

(2) Remove the ITF cover of the pyrolyzer furnace.



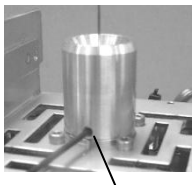
ITF protection cover





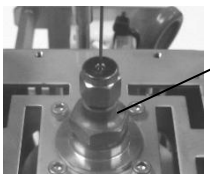
Securing screws

(3) Remove the heat sink adapter.

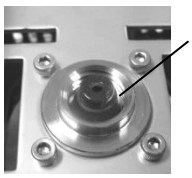


Securing screws (2 pcs)

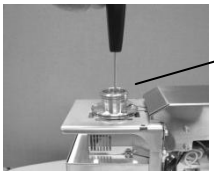
(4) Remove ITF union N using a 12 mm wrench. The ITF union and pyrolysis tube may stick together. When this happens, heat the ITF union with a torch to detach the pyrolysis tube from interface needle. If the ITF union is separated from the pyrolysis tube, remove the liquid sampler located at the top of the pyrolyzer. Then using the supplied hexagonal screw driver (2 mm), push the pyrolysis tube down and remove it.



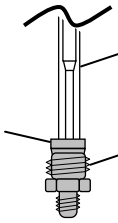
ITF Union N
(use 12 mm wrench)



Graphite Vespel ferrule



Push the pyrolysis tube down using hexagonal screw driver (2.5 mm).

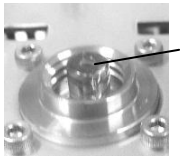


Quartz pyrolysis tube

Graphite Vespel ferrule

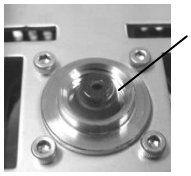
ITF union

(5) Insert a new or clean quartz pyrolysis tube through the bottom of the pyrolyzer furnace. Ensure that the tube end with larger inner diameter goes into the furnace first. While gently rotating the tube, push the bottom of the tube toward the sampler base until the bottom end of the tube sticks out by 1-2 mm from the bottom surface of the pyrolyzer.

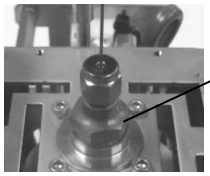


Quartz pyrolysis tube

(6) Attach a new graphite Vespel ferrule to the bottom of the pyrolysis tube which is sticking out from the pyrolyzer at the bottom. Make sure that the large outer diameter end goes in first. Finger - tighten the ITF union.

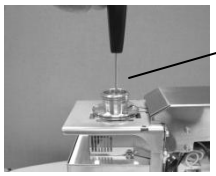


Graphite Vespel ferrule
(Lager outer diameter end goes in first.)



ITF Union
(finger-tighten it)

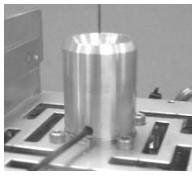
(7) Gently insert the supplied hexagonal screw driver from the top of the pyrolyzer and push the pyrolysis tube down until the end of the quartz tube hits the ITF union. Failure to do so will create a dead space and lead to broadening of peaks on pyrogram.



Push the quartz pyrolysis tube down by inserting hexagonal screw driver through the tube. As shown, the end of the driver is hitting the bottom end of the tube where sample cup situates

(8) Tighten the ITF union slowly about 1/4 turn using a 12 mm wrench. The amount to be tightened depends on the tolerance of the pyrolysis tube and graphite Vespel ferrule.

(9) Attach the heat sink adapter. Secure the adapter so that there is no large gap between the adapter and interface block.



(10) Put the ITF cover back in place. Install a new septum in the injection port, and then install the pyrolyzer on top of the GC. Gently tighten the septum cap at the GC injection port.

(11) Perform a leak test.

- Plug the outlet of the separation column or EGA tube.
- Turn on the carrier gas. The column inlet pressure goes up and should be the same as the carrier gas supply pressure, if there is no leakage. If leaking, refer to Chapter 7.3 “Typical troubleshooting procedure”.

CAUTION

When joining the quartz pyrolysis tube and ITF union, graphite the Vespel ferrule must be firmly tightened, particularly when using a new graphite Vespel ferrule with which a leakage can occur when cooled down. If this is the case, tighten the union further by 15 to 30 degree turn. If the joint is properly tightened, when the interface union is removed, the union, graphite Vespel ferrule and quartz pyrolysis tube should all be stuck together and hard to pull them apart. When the joint is tightened this much, there should be no leakage even when the temperature is lowered.

6.1.2.2 Solvent cleaning

When a large amount of material is pyrolyzed, the upper part of the tube (A in Fig. 6.2) may be contaminated with high boiling compounds. After cooling the furnace to room temperature, the contamination is removed by rubbing the inner wall of the tube with a cotton swab dampened with a solvent as shown in the figure below. If this *is-situ* method fails to achieve the desired results, the quartz pyrolysis tube must be removed from the pyrolyzer, and cleaned, following the instruction given in section 6.1.2.3

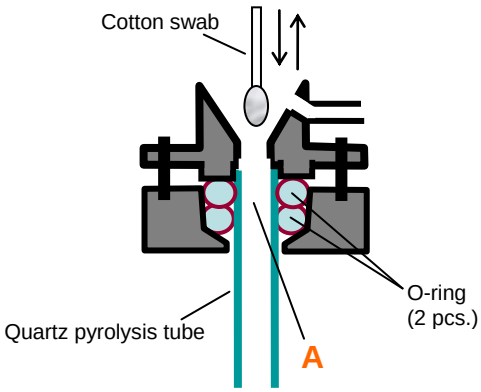


Fig. 6.2 In situ cleaning of the pyrolysis tube

6.1.2.3 Flame cleaning with a torch

CAUTION When using a flame, make sure there is no flammable materials around and work only in a well-ventilated area. Wear safety goggles for your safety.

For your safety, this operation must be done in a designated place. Because the very hot quartz pyrolysis tube needs to be handled, heat-resistant gloves and safety goggles must be worn while working.

(1) As shown in Fig. 6.3, red-heat the quartz pyrolysis tube. This can be done most effectively while flowing air through the tube.

(2) Cool down the heated pyrolysis tube in a secured place. Allow 15 min to cool the tube to room temperature since you cannot tell whether it is still hot by just looking at it.

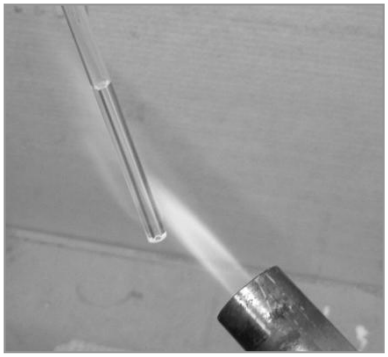


Fig. 6.3 Heating with a torch

6.1.3 Interface needle (ITF needle N)

CAUTION

The end of the interface needle is very sharp. When handling the needle, wear protective gears such as safety goggles for your safety.

6.1.3.1 Clogging by sample contamination

Needle contamination consists of mostly tars and stained tars. The most effective way to clean a contaminated needle is to sonicate it in an appropriate solvent. If this does not show improvements, replace it with a new one. Interface temperature (ITF TEMP) of 250-320°C is recommended for daily operations.

6.1.3.2 Clogging by septum debris

If the septum nut at the GC injection port is over-tightened, the side opening of the needle may be clogged by septum debris when the needle is inserting through the septum.

(1) Heat the sharp end of the needle using a small torch to burn out the debris. This is most effective when an inert gas is flowing through the needle

(2) Plug the sharp end of the needle with a septum, and pressurize the needle using an inert gas. Remove the septum; the sudden depressurization may push the debris to the opening. If so, remove the debris using a pair of tweezers.

6.1.3.3 Connecting union (ITF union N)

Metal ferrule is attached to the ITF needle. Connect the needle to ITF union N using the needle nut. Use a wrench to tighten the needle nut.

When the temperature is lowered, the nut may become loosen. Even in this situation, the metal ferrule is stuck to the union and no gas leakage occurs; however, when performing maintenance, tighten further by 1/8 to 1/4 turn.

6.1.4 GC injection port

6.1.4.1 Septa

When the pyrolyzer is installed on a GC, the injection port is in close contact with the heat sink adapter; therefore, the temperature of the septum is at least 50°C higher than its normal operating temperature. This increases the silicon rubber septum's susceptibility to thermal degradation. Our experiences show that high quality septa will last about one month at injection port temperature of 300°C. However, once the septum is removed from the injector port, a new septum must be used. The use of the supplied pretreated septa (see consumables list for details) will facilitate the removal of the septum.

If the septum is stuck to the metal surface of the injector, cool the temperature down to 100°C or lower and apply hexane to the septum to swell it for easy removal.

6.1.4.2 Insert liner

High boiling compounds from the pyrolyzates travel to the GC injection port via the pyrolyzer. If the injection port temperature is low, such compounds tend to deposit on the interface needle and on the glass insert liner. If this occurs, set the injector temperature to 350°C and allow it to bake for two to three hours with the carrier gas on; remove the column during this bake-out. Inspect the injection port inner wall every one to two weeks.

6.1.4.3 Column

Column degradation depends primarily on the analytical conditions and the nature of the pyrolyzates. Tars and reactive gases such as HCl from pyrolysis can degrade part of or the entire column. Generally, tars are often impossible to remove completely by rinsing the column with a solvent because tars are baked and bonded tightly to the inner wall of the column at high temperatures. If the degradation occurs due to strong acidic gasses such as HCl or HBr, the column cannot be restored to its original performance. If the sample is expected to generate these acidic gases, put some glass wool or fiber coated with potassium hydroxide (a few percents) in the insert liner to protect the column.

6.1.4.4 Absorption trap tube

If an absorption trap set is installed in the split line of the GC injector, replace the trap tube (PY1-2216).

6.2 REGULAR MAINTENANCE

In addition to routine maintenance, regular maintenance every three to four months is recommended. This will ensure trouble-free operation.

6.2.1 Pyrolyzer furnace

(1) Cooling fan

Check to see if the cooling fan starts spinning as soon as the temperature controller is on. If the cooling fan does not start immediately, the fan needs to be replaced. The cooling fan maintains the the sample temperature near room temperature when the system is at stand by. If the temperature of the sample when in the standby position exceeds 100°C, a protection function is activated and the heater power is turned off.

(2) Liquid sampler

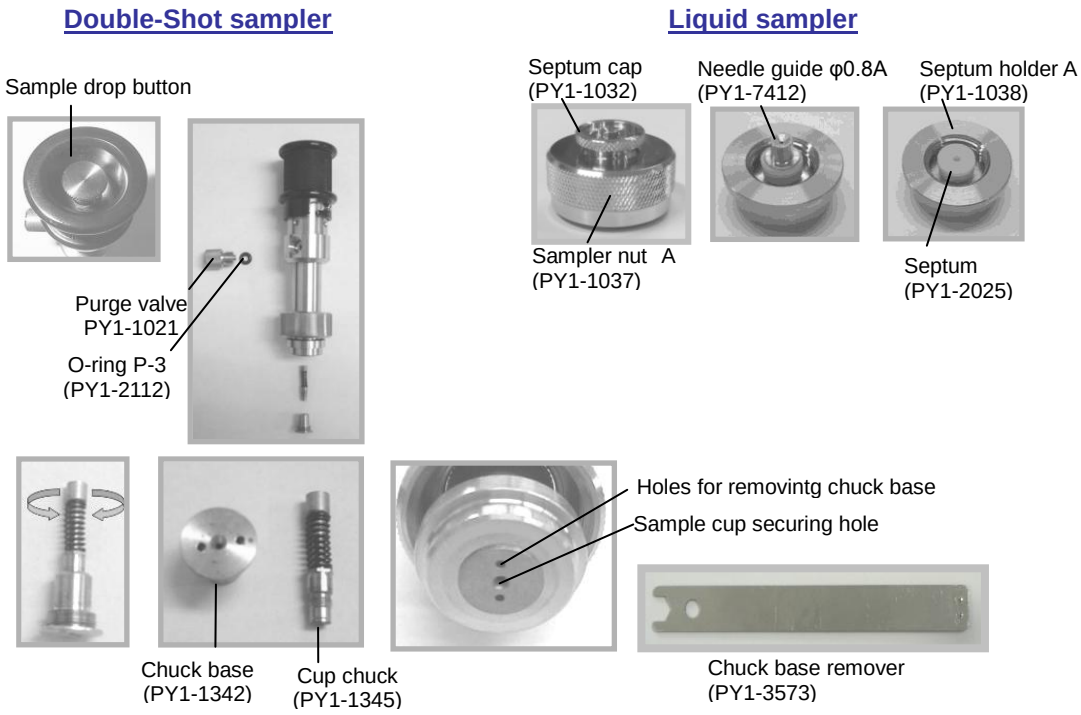
Replace the septum (P/N:PY1-2025). There is no need to perform maintenance work; however, the septum should be regularly replaced to avoid unexpected troubles

(3) Double-Shot sampler

a) Make sure that the slider moves smoothly. If there is difficulty, slide it up and down several times until it moves smoothly; if this fails to smooth out the movement it may be necessary to disassemble it prior to cleaning. After cleaning, a lubricant must be applied. Contact us if you prefer it to be re-conditioned at Frontier Labs (charged).

b) Check the O-ring for air purge valve for any damage. The damage can lead to gas leakage.

c) The mounting surface of the sampler is a mirror finish. Contamination or scratches on the surface can result in gas leakages. When storing the sampler, the surface should be protected.



d) The sample cup chuck is a consumable part. Verify that it securely holds the Eco-Stick and that pressing the release button releases the sample cup. If there is any problem, the sample cup chuck (PY1-1345) needs to be replaced with a new one. This is customer serviceable, contact your nearest sales office or Frontier Laboratories.

(4) Sampler base

Contamination or damage of the O-rings (P-12, P/N: PY1-7811) at the sampler base may result in gas leakage. Clean them by rubbing them with a cotton swab dampened with methanol. Avoid using other solvents such as acetone, dichloromethane, or etc.

6.2.2 GC injection port

Upon extended use, the injection port split outlet pipe may be clogged by tars. If this occurs, remove it from the GC and clean it by flushing a solvent through it, and then heat it by a torch while running an inert gas through it. The pipe needs to be checked every three to four months of use.

There is a risk of burn injury. Before starting work, make sure that the injector temperature is below 50°C.

When cleaning with volatile organic solvents, ensure there is no ignition source around. When using a burner flame, work in a well-ventilated area free from flammable organic solvents.

6.2.3 Temperature controller

Before removing cables, turn off the power switch and unplug the power code of the temperature controller.

(1) Make sure that connectors for heater power of pyrolyzer, temperature sensor, and temperature controller have good electrical contact.

(2) Make sure the temperature controller is properly grounded.

6.2.4 When not in use for a long period of time

If the pyrolyzer is not going to be used for a long period of time, remove the pyrolyzer from GC and store in a nylon bag so that the cartridge heater used in the interface is protected from moisture.

6.2.5 List of recommended replacement parts

Pats listed below are consumable parts that are recommended to replace during maintenance work. The replacement interval raries depending on the usage.

Device	Part name	Part No.	Replacement cycle	Note
Pyrolyzer (Furnace)	Quartz pyrolysis tube A30	PY1-3018A	As needed	• Replacement interval depends on type of samples, amount used, and number of runs. • Can be heat-cleaned with torch flame.
	Graphite Vespel ferrule	PY1-7911	As needed	• Replace when pyrolysis tube M30 is replaced.
	Needle set N	PY1-1274	As needed	• Replacement recommended when pyrolysis tube M30 is replaced. • Over tightening injector septum may clog needle opening with septum debris.
	ITF union N	PY1-3513	1 year	• Standard accessory for EGA/PY-3030D
	ITF union ASN	PY1-3533	1 year	• Used when Auto-Shot sampler is used.
	O-ring P-6W	PY1-2017	As needed	• For sealing pyrolysis tube M30, 2 pcs used
	Eco pickup	PY1-EP55F	1 year	• Replacement not needed when Auto-Shot Sampler is used all the time.
	Septa for liquid sampler, 20 pcs	PY1-2025	1 year	• Inspection may only be needed if liquid sample injection with micro syringe is not done.
	O-ring P12	PY1-7811	1 year	• For sampler base.
	Inlet/outlet septum	PY1-2028	6 months	• Used for carrier gas tube joints
Double-Shot sampler	Cup chuck	PY1-1345	1 year	• Worn-out chuck does not hold eco stick firmly. • Special tool required.
	Overhaul		2 years	• The product needs to be returned to F-Lab.
	O-ring P-3	PY1-2112	6 months	
GC	Absorption trap tube	PY1-2216	3~6 months	
	O-ring P-5	PY1-7814	3~6 months	• Replace when trap tube is replaced. 4 pcs used.
	Septum	Agilent PY1-7301	As needed	• Replace if leakage detected when pyrolyzer interface or GC injector cools down. • Replace when pyrolyzer is removed from GC injector.
		Shimadzu PY1-7304		
		Others		
	Other parts that GC manufacturers recommend			

6.2.6 Periodic maintenance and inspection

We recommend that the pyrolyzer be overhauled and inspected every two years and that hard-to-replace parts such as heater and thermal insulator be replaced for your stable and safety operation of the product.

CHAPTER 7 TROUBLESHOOTING

Symptoms, causes, and their solutions are summarized below. If your problem persists after implementing the suggested solutions, or if you experience problems not addressed here, contact your nearest sales office or contact Frontier Laboratories directly: www.frontier-lab.com.

7.1 PYROLYZER FURNACE

SYMPTOM	CAUSE	SOLUTION/CHECK POINTS
No peaks	Gas leak	See section 7.3 Check joints between column and GC injection port. Replace the septum
	No signal from GC detector	Consult your GC operation manual.
	ITF needle is clogged up	See section 7.3 (2) Inspect the ITF needle.. If it is clogged, replace the ITF needle.
Broad peaks	Degraded column	Replace column.
	Abnormal diffusion from pyrolyzer furnace up to column inlet	Raise split ratio.
	Pyrolysis temperature too low	Increase pyrolysis temperature by 100ºC.
Peak tailing	Degraded column	Replace column
	Abnormal diffusion from pyrolyzer furnace up to column inlet	Increase split vent flow.
	Pyrolysis temperature too low	Increase pyrolysis temperature by 100ºC.
Low recovery of high boiling compounds Peak tailing of high boiling compounds	ITF and injector temperature too low	Increase temperature. Eg.: from 200ºC to 300ºC Do not set GC injector temperature over 350ºC unless absolutely necessary.
	GC/MS ITF temperature too low	Increase the temperature.
Abnormal ghost peaks appear in the high temperature region	Not all pyrolyzates have eluted from the column	Use Selective Sampler to prevent high boiling compounds from getting into column. Use high temperature resistant column and bake column after each run.
	Septum debris in insert liner	Clean insert liner.
	Contaminated pyrolysis tube	See section 6.1.2 for details.
Quartz pyrolysis tube and needle union are stuck together	Phenomenon due to thermal shrinkage of graphite Vespel ferrule	Heat ferrule with torch flame to soften and expand the ferrule.

SYMPTOM	CAUSE	SOLUTION/CHECK POINTS
Polar compounds not eluting from the system	Degraded column	Replace column or check again using Ultra ALLOY column (UA5) to see how this changes.
	Tars deposited in the ITF needle	Clean or replace it. Increase ITF temperature.
	Poor deactivation of pyrolysis tube	Inorganic compounds may have deposited. Clean or replace it.
Poor reproducibility of pyrograms	Gas leak	See section 7.3 for details.
	Improper pyrolysis temperature	Set temperature to proper pyrolysis temperature. (Set furnace 50ºC higher than end temperature observed in EGA thermogram.)
	Sample amount or shape not uniform	Try to use same amount of sample and uniform sample shape.
	Depending on samples, good reproducibility may not be obtained. If the problem persists, check the system with polystyrene standard.	
Furnace and ITF temperatures do not reach set temperatures	Heaters not turned on	Turn heater on.
	Power breaker open	Re-set the main breaker on the back of the controller.
	Broken heater	Repair needed
	Temperature controller malfunctioning	Repair needed
No cooling gas	Cooling gas not supplied	Check cooling gas supply valve and pressure.
	Malfunctioning of EM valve in the rear of controller	Repair needed
	Cooling in the range of 800 to 1050 ºC	If furnace temperature is 800ºC or higher, the cooling gas does not flow; the furnace is allowed to cool itself. Once the furnace is below 800 ºC, the cooling gas is turned ON.
	Cooling gas tube is plugged.	Connect the cooling gas directly to the cooling gas inlet port of the furnace to confirm the tube is open. Repair needed if the gas does not flow.
Septum residues remain in the septum area when replacing septum	Adhesion caused by the thermal decomposition of rubber	Remove the residues using a solvent such as hexane. Use Frontier Labs' surface treated septum to reduce the adhesion of the septum.
Increase baseline at high temperature region under programmed heating (high background)	Bleeding due to deterioration of column stationary phase	Perform solvent cleaning. This may help reduce bleeding. Run 1 mL of acetone through the column, then connect it to GC and turn carrier gas ON. Set the oven temperature to 40ºC and hold for 30 min then raise the temperature at 10ºC /min until dry. If column is not Frontier Labs' Ultra ALLOY column, contact column manufacturer.

Quartz pyrolysis tube is broken	Tube cracks in lengthwise direction. The sampler base is fixed with screws while the quartz pyrolysis tube is not inserted to the dead end of the furnace.	Fixed the upper part in the state that a quartz pyrolysis tube was not inserted in properly to the depths and might tighten up ITF union. To see an instruction manual, please confirm whether a quartz pyrolysis tube is attached definitely.
	Tube cracks laterally. Tube cracks at the welded portion. Misaligned blocks (interface, furnace and sampler base).	Alignment blocks are required. Ask for repair.

7.2 TEMPERATURE CONTROLLER

SYMPTOM	CAUSE	SOLUTION/CHECK POINTS
Furnace will not heat	Heater setting is OFF.	Set it to ON (check the furnace box in the configuration software)
	Breaker is off.	Re-set the main breaker on the rear panel of the controller.
Beep sound	Sensor cable is not connected or contact failure	Ensure cable from pyrolyzer is properly connected to connector labeled POWER, SENSOR at rear of controller.
	Sensor failure	Repair needed.
Cooling fan not running	It is blocked by a foreign object	Make sure it is not blocked by foreign object or gas tubing.
	Fan failure	Repair needed.
	Fan power not supplied	Ensure fan power cable is properly connected. If it is, repair is needed.
Abnormal temperature	Sensor contact failure	Check to see if sensor cable from pyrolyzer is properly connected.
	Error in temperature calibration	Redo calibration.
	Sensor circuit failure	Repair needed.
Temperature unstable at low temperatures (eg. 100ºC or below)	ITF temperature may be too high	Set ITF temperature control to Auto. (In Auto mode, it is controlled to 100 ºC higher temperature than pyrolyzer furnace.)

7.3 TYPICAL TROUBLESHOOTING PROCEDURE

The two most prevalent problems encountered when using the pyrolyzer are gas leaks (at the needle interface) and a clogged ITF needle. Here are some examples of the symptoms associated with these two problems:

- Background peaks in the MS spectrum have a high abundance of m/z 28 and 32.
- Poor reproducibility of pyrograms.
- Insufficient column head pressure (flow).

  The total flow setting for the carrier gas may be too low. Set it at 50 mL/min or more.
- In the constant flow mode, the carrier gas pressure plateaus while the column is being temperature programmed.

  The carrier gas supply pressure may be too low. Set it to the proper pressure by consulting your GC manual.

Troubleshooting for gas leaks is described. If symptoms mentioned above are observed, follow the procedure below to troubleshoot.

CAUTION The work is done with an ITF needle N whose end is very sharp. For your safety, wear protective gears such as safety goggles.

(1) **Checking gas leakage of pyrolyzer**

Perform the leak test below.

(1)-1 Plug the outlet of the separation column or EGA tube using a septum.

(1)-2 Plug the outlets of the split and purge vents using cap nuts.

(1)-3 When the carrier gas is turned on, the column head pressure increases and should be the same as the supply pressure. If not, there is a leakage. Use a He leak detector to locate the problem.

The causes of gas leakage originating from the pyrolyzer are provided here along with the solutions:

- O-ring (Fig. 7.1a) that seals the upper end of pyrolysis tube has become loose. ➔ Further tighten the three hexagonal screws or replace the O-ring (P/N: PY1-2012)
- The graphite Vespel ferrule or graphite ferrule at ITF union has become loose or degraded (Fig. 7.1b) ➔ Further tighten (work gently to avoid breaking the tube) or replace the ferrule (P/N: PY1-7911 graphite Vespel ferrule, PY1-7921 graphite)
- The metal ferrule on the ITF needle (Fig. 7.1b) has become loose or degraded. ➔ Tighten it further or replace the ITF needle (the metal ferrule is fixed to the needle, needle set P/N: PY1-1174).
- Liquid sampler (Fig. 7.1c) has become loosen, the O-ring of the sampler base has degraded or foreign objects present. ➔ Tighten it further (normally finger-tightening is adequate). Clean the O-ring and the mirror-finished part of the sampler using a cotton swab dampened with methanol, or replace O-ring (P-6 P/N: PY1-2012)

If the remedies mentioned above do not work, the gas leak may be due to a damaged quartz pyrolysis tube.

➔ Remove the tube from the pyrolyzer and inspect it for damage; replace it with a new one if damage is observed (quartz pyrolysis tube, P/N: PY1-3018M)

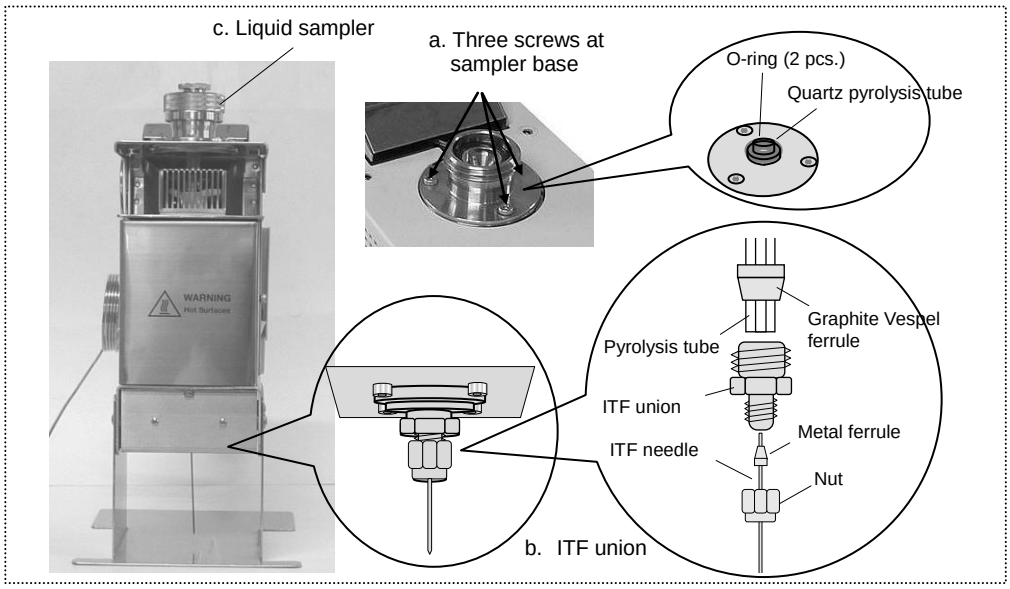


Fig. 7.1 Checking gas leakage

(2) **Checking clogging of ITF needle**

If no gas leakage is found, check to see if the ITF needle may be clogged. Set the carrier gas total flow to 100 mL/min and the column flow rate to 1 mL/min, and make sure that the readings are equal to the set values. If the actual flows are equal to the set values and the problem persists, the ITF needle opening may be clogged with septum or sample residues. If this is the case, the symptoms shown below will be observed. After addressing the needle clogging, loosen the septum cap nut a bit, and then repeat the leak check.

- When the sampler is removed, the injector pressure drops very slowly or does not drop. *It should drop to zero in 1 to 2 seconds.*
- The carrier gas total flow is unstable and eventually goes to zero.
- When the sampler is removed from the pyrolyzer a gas releasing sound is heard.

If your problem persists contact your nearest sales office or Frontier Laboratories: www.frontier-lab.com .

7.4 LIST OF ERROR CODES FROM CONTROL SOFTWARE

ERRO NO./DISPLAY	CAUSE	SOLUTION/CHECK POINTS
1. Heater	Heater not powered	Check to see if the cable from the pyrolyzer is properly connected to the POWER connector located on the rear of controller.
2. Sensor	Abnormal input from sensor	[Furnace or Interface] Make sure the cable from the pyrolyzer is properly connected to the SENSOR connector located on the rear of the controller. [Cryo-Trap] Ensure that the temperature sensor connector is short-circuited when MicroJet Cryo-Trap is not used.
3. Overheating	Overheated.	Make sure the cable from the pyrolyzer is properly connected to the SENSOR connector located on rear of the controller. If this does not resolve the problem, it may be heater failure (Furnace or Interface). Or it may be a cooling fan failure (Sample cup stand by position). Contact your nearest sales office.
4. COM Port	USB cable contact failure	Re-seat the USB cable at the PC
11. Connection	Connection failure to Auto-Shot Sampler	(1) Ensure that the cable from Auto-Shot Sampler is properly connected to RS-232C connector on the rear of the controller. (2) Make sure the Auto-Shot Sampler is powered on.
12. Cup chute	Cup chute not attached to the Auto-Shot Sampler.	Properly attach cup chute to Auto-Shot Sampler.
13. Sample cup retrieval	Sample cups not properly retrieved.	(1) Make sure supply pressure of gas reservoir is 300 kPa – 500 kPa. (2) Ensure that the valve control cable from the gas reservoir is properly connected to the rear of the Auto-Shot Sampler. (3) Make sure sample cups are not deformed.
21.Cryo-trap Temperature	MJT sensor not properly cooled.	(1) While the thermal exchange coil is at room temperature, make sure the pressure is 250 – 300 kPa when Cooling is turned ON. (2) Make sure there is ample amount of liquid nitrogen.

CHAPTER 8 GUARANTEE OF BASIC PERFORMANCE (REPRODUCIBILITY)

In a GC pyrolysis system, the path from the pyrolyzer to the detector via the separation column is very important. Every element in the system (i.e., the pyrolyzer, interface, GC injection port, separation column, detector crossover and detector) plays an important role in the reproducibility of the data.

CAUTION

When using solvents, wear protective gears such as safety goggles and work only in a well-ventilated area free of ignition sources.

CAUTION

The ends of separation column and eco stick are very sharp. When handling these items, wear safety goggles for your safety.

CAUTION HOT

There is a risk of burn injury.
Since the pyrolyzer furnace can be very hot, ensure that the protective housing cover is properly placed on the pyrolyzer.

Reproducibility (expressed as the %RSD of three sequential analyses) of the polystyrene EGA thermogram and the polystyrene pyrogram are guaranteed to meet the following specifications:

Evolved Gas Analysis: Frontier Laboratories guarantees that the reproducibility of the apex of the polystyrene peak is <0.3%RSD.

Pyrolysis: Frontier Laboratories guarantees that the reproducibility of the peak area ratio between methyl stearate, added as internal standard, and the styrene trimer (SSS) is <2% RSD.

The analytical conditions and typical chromatograms are shown in Figures 8.1 and 8.2.

Analytical conditions

ITF tube	: EGA tube (deactivated metal capillary, L=2.5 m, id.=0.15 mm, no stationary phase)
Column flow	: 1.0 ml/min
Split ratio	: 1/100
Oven temp.	: 300°C (isothermal)
Injector temp.	: 300°C
Py furnace temp.	: 100 - 550°C (20°C/min)
Py-GC ITF temp.	: 300°C (AUTO mode)
Sample	: Polystyrene (contains 5 wt% methyl stearate)
Sample wt.	: 125 µg (5µl of 25 µg/µl dichloromethane solution)
Sample cup	: Eco-Cup LF
Detector	: MS or FID
GC/MS ITF temp.	: 300°C
Scan range	: 29 - 350 (m/z)
Scan speed	: 0.1 scan/sec

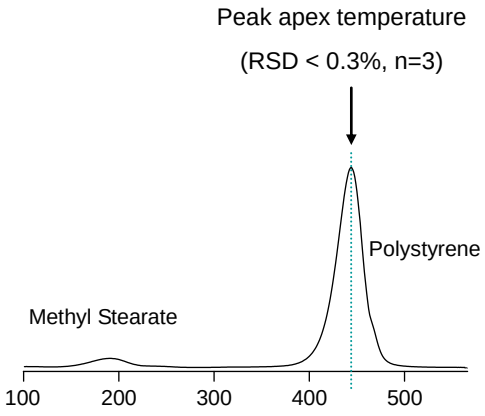
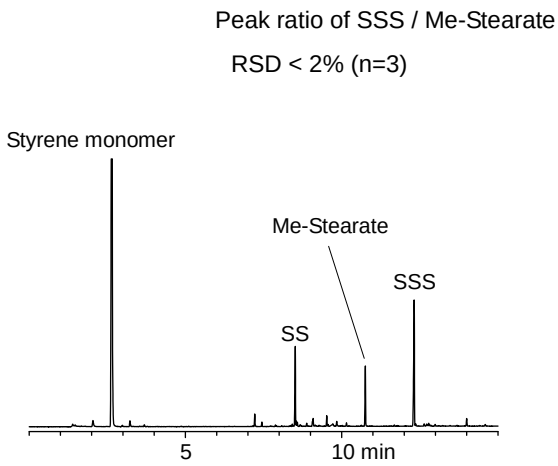


Fig. 8.1 Test conditions and a typical EGA thermogram

Analytical conditions

Separation column	: Ultra ALLOY^{+}-5 (5% phenyl 95% dimehtylpolysiloxane L=30m, id.=0.25mm, df=0.25 µm)
Column flow	: 1.0ml/min
Split ratio	: 1/100
Oven temp.	: 70 - 320°C (20°C/min, 1 min hold)
Injector temp.	: 300°C
Py furnace temp.	: 550°C
Py-GC ITF temp.	: 300°C
Sample	: Polystyrene (contains 5 wt% methyl stearate)
Sample wt.	: 25 µg (5µl of 5µg/µl dichloromethane solution)
Sample cup	: Eco-Cup LF (single cup used)
Detector	: MS (200V lower than that of Autotune)
GC/MS ITF temp.	: 300°C
Scan range	: 29 – 350 (m/z)
Scan speed	: 3 scans/sec or greater



Mass spectra of maijor peaks

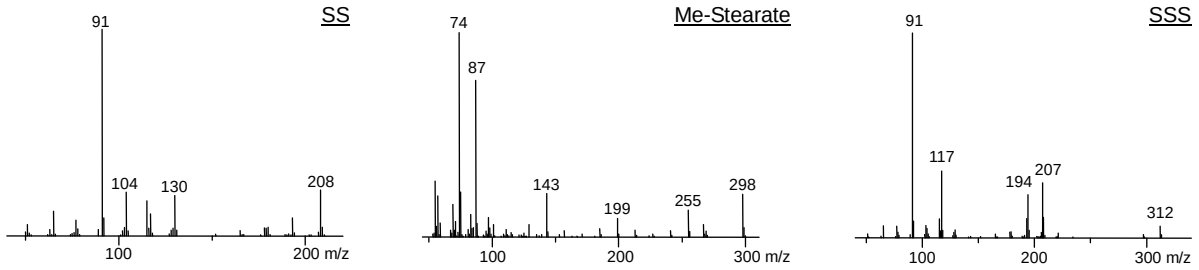


Fig. 8.2 Test conditions and a typical check-out sample pyrogram

CONSUMABLE PARTS LIST

(Please visit our web site for photographs of each consumable part.)

As of October 1, 2016

	Part name	Part No.	Unit	Description
1. Sample cup and related parts (All sample cups treated by special deactivation process)				
○	Eco cup SF	PY1-EC50F	1 set	50 µl, id. 3.8 mm, deactivated stainless steel, contains 100 pcs
○	Eco cup LF	PY1-EC80F	1 set	80 µl, id. 3.8 mm, deactivated stainless steel, contains 100 pcs
	Eco cup G	PY1-EC50G	1 set	50 µl, id. 3.0 mm, glass, no quartz coating, contains 100 pcs
	Eco cup GQ	PY1-EC50GQ	1 set	50 µl, id. 3.0 mm, quartz coated glass, contains 30 pcs
	Flow through Eco cup SHF	PY1-EC50HF	1 set	50 µl, id. 3.8 mm, deactivated stainless steel, hole at bottom, 20 pcs
	Flow through Eco cup LHF	PY1-EC80HF	1 set	80 µl, id. 3.8 mm, deactivated stainless steel, hole at bottom, 20 pcs
○	Eco stick SF	PY1-ES10F	1 set	For Single-Shot, contains 50 pcs
○	Eco stick DF	PY1-ES20F	1 set	For Double-Shot, contains 50 pcs
	Eco stick GS	PY1-ES10G	1 set	For Single-Shot with Eco cup G/GQ, contains 50 pcs
	Eco stick GD	PY1-ES20G	1 set	For Double-Shot with Eco cup G/GQ, contains 50 pcs
M	Eco Pickup F	PY1-EP55F	1 set	Stainless steel Eco cups retriever, contains 5 pcs
	Eco Pickup GF	PY1-EP55GF	1 set	Eco cup G/GQ retriever, contains 5 pcs
	Eco Stand AL	PY1-EH10AL	1 set	Eco-cup stand (for S and L), used when sampling, 5 pcs
	Performance evaluation polystyrene standard sample	PY1-4908	1 set	Polystyrene 2.5mg film, Me-stearate 5wt.% , 2ml glass bottle.
	Fine quartz wool	PY1-5111	1 pkg	2 g, covering the sample in the eco-cup or other universal use.
2. Pyrolyzer furnace				
○	Quartz pyrolysis tube A30	PY1-3018A	1 pc	Quartz pyrolysis tube for EGA/PY-3030D
	Needle Guide φ0.8A	PY1-7412	1 pc	For liquid sampler, φ0.8mm, 1 ea
M	Septum	PY1-2025	1 pkg	Silicon rubber for liquid sampler (pale yellow), contains 20 pcs
M	O-ring (P-6W)	PY1-2017	1 pkg	For sealing quartz pyrolysis tube, standard for EGA/PY-3030D, white silicon rubber, contains 20 pcs
M	O-ring (P-12)	PY1-7811	1 pkg	For sampler base, black, 10pcs
3. Connection parts				
M	ITF union N	PY1-3513	1 pc	EGA/PY-3030D standard part. Deactivated.
	ITF Union N Set	PY1-3515	1 set	Includes ITF union N(1ea), ITF needle(2ea) and Needle nut(1ea), 1 set
M	ITF union ASN	PY1-3533	1 pcs	For Auto-Shot Sampler and EGA/PY-3030D, Deactivated.
	Heat sink adapter kit 3030SA	PY1-3551	1 set	For EGA/PY-3030D. Used for Agilent7890/6890 and Shimadzu GC-2010 /17A, includes 2 securing screws.
	Heat sink adapter kit 3030PE	PY1-3552	1 set	For EGA/PY-3030D. For PE Auto system/Clarus GC, includes 2 securing screws.
	Needle nut	PY1-3512	1 pkg	Interface needle securing nut. Contains 3 pcs.
○	Needle set N	PY1-1274	1 pkg	Deactivated needle, contains 3pcs (with 2 side openings)
○	Graphite Vespel ferrule	PY1-7911	1 pkg	6mm, for PY1-3513, contains 5 pcs.
M	Inlet outlet packing	PY1-2028	1 pkg	For tube connections, silicon rubber (pale yellow) with hole, contains 20 pcs.
4. GC related parts				
○	Septum	PY1-7301	1 pkg	for Agilent GC, Thermo TRACE 1300/1310GC injection port, surface treated, 5 ea
○	Septum (for Shimadzu)	PY1-7304	1 pkg	For injection port of Shimadzu GC, for pyrolysis only, contains 5 pcs, surface specially treated.
○	Glass insert (with wool)	PY1-3337	1 pc	For injection port of Agilent 6890 and 7890, surface deactivated, with deactivated quartz wool.
	Quartz insert	PY1-3361	1 pc	For Agilent 6890 and 7890, highly deactivated.
	Graphite O-ring (for Agilent)	PY1-2202		Graphite O-ring for Agilent GC liners, 3 ea
	Adsorption trap set 17SV	PY1-2227	1 pc	For Shimadzu GC-17A/14A and GC-2010 split vent
M	Adsorption trap tube	PY1-2216	1 pc	Activated carbon filled trap tube only, used for all models, contains 3 pc.
	GC/MS connector kit	PY1-2210	1 pc	Metal capillary column connection kit for magnetic field MS.
	Vent-free GC/MS Adapter-50	MS402180	1 set	Recommended for Agilent, JEOL, Shimadzu GC/MS,VF tube A-50 (50cm, 2 set) with, VF holder A (1ea), Column nut (2 ea), VF metal ferrule D (10 ea), Plug nut A (1ea), 1 set
	Vent-free GC/MS Adapter-70	MS402190	1 set	Recommended for Thermo GC/MS, VF tube A-70 (70 cm, 2 set), Column nut (2 ea), VF holder A(1 ea), VF metal ferrule D(10 ea), Plug nut A (1 ea), 1 set
	Vent-free GC/MSAdapter-70PE	MS402195	1 set	Recommended for PerkinElmer Clarus series,VF tube A-70PE (70cm, 2 set), VF holder A(1 ea), Column nut (2 ea), VF metal ferrule D (10 ea), Plug nut A (1 ea), 1 set
	VF Tube A-50	MS402159	1 pc	VF tube A-50, includes each one of Vent-free tube 0.15mm id / 50cm, VF metal ferrule D and VF nut B. For Agilent, JEOL, Shimadzu GC/MS
	VF Tube A-70	MS402191	1 pc	VF tube A-70, includes each 1 pcs of Vent-free tube 0.15mm id / 70cm, graphite vespel ferrule and 1/16 nut (SS), 1 set. For Thermo Fisher GC/MS

VF Tube A-70PE	MS402196	1 pc	VF tube A-70, includes each one of Vent-free tube 0.15mm id / 70cm, VF metal ferrule D and VF nut B, For PerkinElmer Clarus series

Part name	Part No.	Unit	Description
4. GC related parts			
VF Nut B	MS402286	3 pc	VF MS nut B, 3 ea
○ VF Metal Ferrule D	MS402167	20 pc	VF metal ferrule D, for column connection, 20 ea
○ Column Nut	MS604234	1 pc	Column nut, 1 ea
EGA Capillary Tube	UADTM-2.5N	1 pc	Deactivated metal capillary tube for evolved gas analysis (Ultra ALLOY 2.5 m, id. 0.25 mm, od. 0.47 mm, with bobbin).
5. Double-Shot / Single-Shot Sampler related parts			
Sample cup Chuck	PY1-1345	1 pc	Sample cup chuck for Single and Double-Shot samplers, 1 ea
Chuck Base	PY1-1342	1 pc	Chuck base for Single and Double-Shot samplers, 1 ea
Chuck Base Fixture	PY1-3573	1 pc	Chuck base fixture
O-ring (P-3)	PY1-2112	1 pkg	For purge valve of Double-Shot sampler, 20 ea
6. Magic Chemisorber (solid phase extraction element)			
Magic Chemisorber S500	PY1-MC01S	1 set	100% methyl silicon, df=500 µm, length 6 mm, contains 10 pcs.
Magic Chemisorber S500 kit	PY1-MC01S-K	1 set	Eco-stick DF 5 pcs, Magic Chemisorber S500, 3 pcs, Flow through Eco-cup LHF, 5 pcs.
Magic Chemisorber L500	PY1-MC01L	1 set	100% methyl silicon, df=500 µm, length 30 mm, contains 5 pcs.
Magic Chemisorber L500 kit	PY1-MC01L-K	1 set	Eco-stick DF 5 pcs, Magic Chemisorber L500, 3 pcs, Protection tube for Magic Chemisorber L500, 2 pcs.
Magic Chemisorber L100	PY1-MC02L	1 set	100% methyl silicon, df=100 µm, length 30 mm, contains 5 pcs.
Magic Chemisorber L100 kit	PY1-MC02L-K	1 set	Eco-stick DF 5 pcs, Magic Chemisorber L100, contains 3 pcs. Protection tube for Magic Chemisorber L, contains 2 pcs.
Magic Chemisorber SL kit	PY1-MC01SL-K	1 set	Magic Chemisorber S500, L500, L100, each 2 pcs.
Magic Chemisorber protection tube	PY1-MCP01	1 set	Protection tube for Magic Chemisorber L, contains 2 pcs.
7. Harris Micro Puncher (tool used to easily punch out discs from a thin sheet of sample)			
Micro-puncher 075S	FMP-0.75S	1 set	Disc size 0.75 mmΦ, green. Contains 3 pcs., cutting mat L 1pc
Micro-puncher 125S	FMP-1.25S	1 set	Disc size 1.25 mmΦ, red, contains 3 pcs. , cutting mat L 1pc
Micro-puncher 200S	FMP-2.00S	1 set	Disc size 2.00 mmΦ, black, contains 3 pcs. , cutting mat L 1pc
Micro-puncher 03 Kit	FMP-03KIT	1 set	includes each 3 pcs of Micro-puncher 0.75 / 1.25 / 2.0 mmΦ and 2 sheets of cutting mats L 65x75mm/green, 1 set
Micro-puncher 050S	HMP-0.50S	1 set	Disc size 0.50 mmΦ, blue, contains 3 pcs. , cutting mat L 1pc
Micro-puncher 300S	HMP-3.00S	1 set	Disc size 3.00 mmΦ, navy,, contains 3 pcs. , cutting mat L 1pc
Micro-puncher 400S	HMP-4.00S	1 set	Disc size 4.00 mmΦ, brown,, contains 3 pcs. , cutting mat L 1pc
Micro-puncher 500S	HMP-5.00S	1 set	Disc size 5.00 mmΦ, orange, contains 3 pcs. , cutting mat L1pc
Micro-puncher 04 Kit	HMP-04KIT	1 set	includes each 3 pcs of Micro-puncher 3.0 / 4.0 / 5.0 Φmm and 2 sheets of cutting mats L 65x75mm/green, 1 set
	FCM-6577	1 set	
8. Consumables set (one year's worth)			
Consumables set 3030D (for EGA/PY-3030D)	PY-K303D	1 set	Eco-cup SF, Eco-cup LF, Eco-stick SF, Eco-stick DF, Eco stand, quartz pyrolysis tube A30, needle set, 6 mm graphite Vespel ferrule, Eco-pickup F, Septum (for liquid sampler)
Consumables set 3030S (for PY-3030S)	PY-K303S	1 set	Eco-cup SF, Eco-cup LF, Eco-stick SF, Eco stand, quartz pyrolysis tube A30, needle set, 6 mm graphite Vespel ferrule, Eco-pickup F, Septum (for liquid sampler)
9. Miscellaneous			
Polymer Prepper	PY1-7510	1 set	Tool to pulverize polymer samples for Py-GC、FT-IR、XRD measurement, contains 3 pcs, with cleaning tape (2 m)
Cleaning Tape	PY1-7515	1 set	Cleaning tape for Polymer Prepper (2 m), set of 3.

Part name marked with ○ is required for daily maintenance.

Part name marked with M is recommended replacement at periodic maintenance (biannually or annually).

PYROLYZER MAJOR CONSUMABLE PARTS

Major consumable parts are visually illustrated with part names (part numbers, quantities).

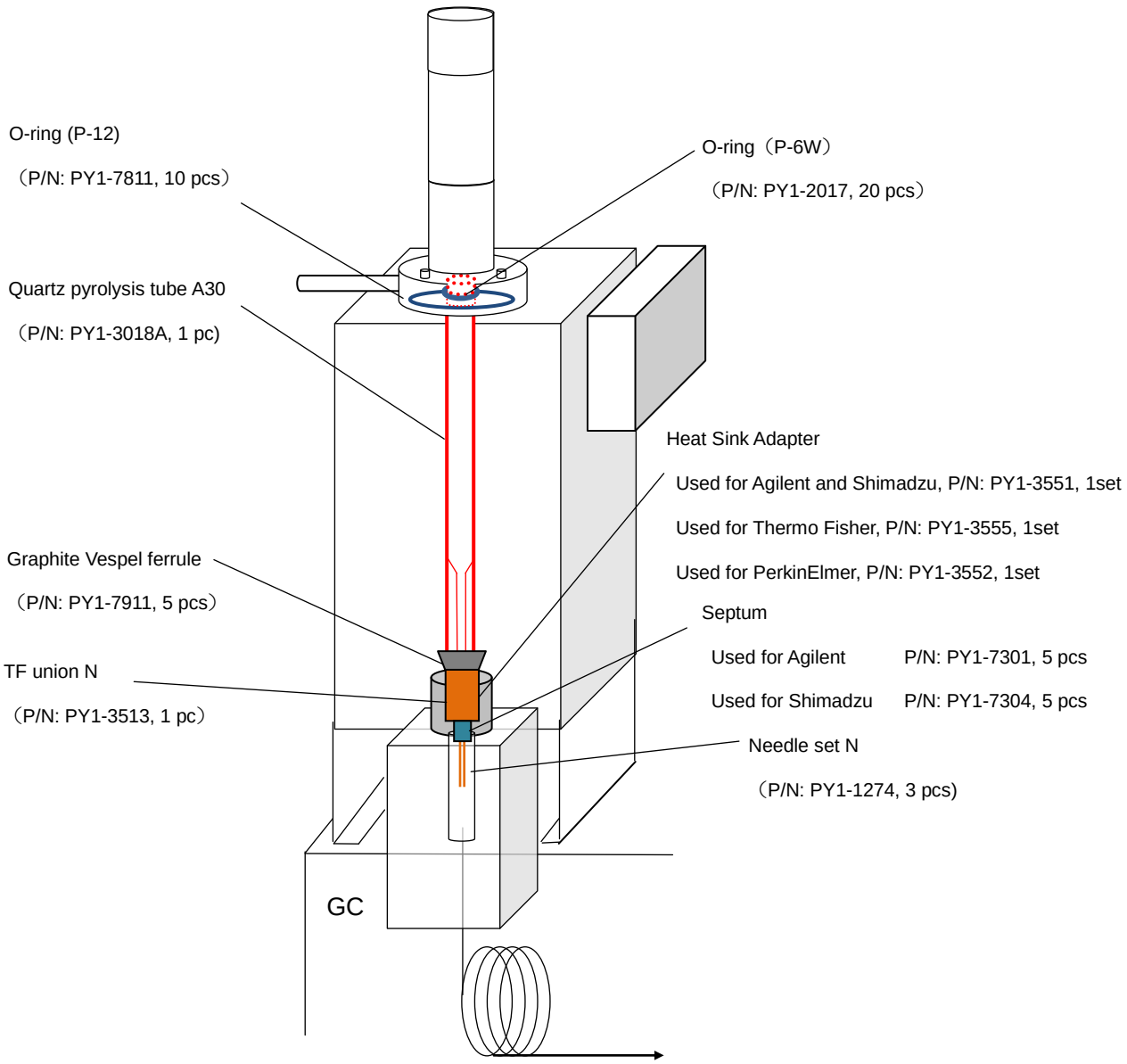


Fig. A.1 PYROLYZER MAJOR CONSUMABLE PARTS

LIST OF OPTIONAL PRODUCTS

A variety of optional products are available for you to better use Frontier Laboratories Multi-Shot or Single-Shot pyrolyzers. Contact your nearest sales office or Frontier Laboratories for details.

Product No.	Product name	Description
PY-1010E	Selective Sampler (SS-1010E)	The device introduces gases eluted from pyrolyzer in any temperature zone into a separation column, utilizing gas pressure difference at the column inlet. When used in combination with Multi-Shot pyrolyzer, maximum of eight temperature zones can be automatically analyzed.
PY-1020E	Auto-Shot Sampler (AS-1020E)	Auto sampler for Multi-Shot pyrolyzer, 48 samples max.
PY-1035E	MicroJet Cryo-Trap （MJT-1035E）	With the basic performance of the MicroJet Cryo-Trap (MJT-1030Ex), this model works collaboratively with a variety of sample introduction devices such as purge & trap, headspace sampler, pyrolyzer, etc. and can be controlled independently
PY-1047Xe	Micro UV Irradiator Xe (UV-1047Xe)	The device allows rapid analysis of photo, thermal, and oxidative degradation of polymers. This powerful optional device for Multi-Shot pyrolyzer helps design and develop new materials by evaluating the weatherability.
PY-1050Ex	Carrier Gas Selector (CGS-1050Ex)	The device allows easy switching of atmosphere gases (e.g. Helium to air) when thermal decomposition or reaction in active gases such as in air is studied.
PY-1110E-131	F-Search Ver.3.4 All-in-one (Polymer evolved gas/Pyrogram MS library, Additives library, pyrolyzates library)	This software program allows library search of EGA thermograms and pyrograms of polymers/additives obtained using MS as a detector. Compatible to GC/MS formats of Agilent, JEOL, and Shimadzu and to AIA format. The package includes two polymer libraries containing data for 700 polymers, library containing data for pyrolyzates of 165 polymers, and library containing data for 358 additives and their pyrolyzates
PY-1111E-131	F-Search Library Search software Ver.3.4	Library search software program, required for library search.
PY-1112E-131	EGA-MS 14B Library	Evolved gas MS library contains thermograms and average MS spectra of 700 polymers.
PY-1113E-131	PyGC-MS14B Library	Py-GC/MS library contains pyrograms and integration-summation MS spectra of 700 polymers.
PY-1114E-131	ADD-MS13B Library	Library containing additives used in polymers contains pyrograms of 358 additives and about 3,400 MS spectra of additives and their pyrolyzates.
PY-1115E-131	Pyrolyzate-MS13B Library	Library contains about 3,200 MS spectra of pyrolyzates of 165 polymers.
PY-K303D	Consumable part set 3030D	The set contains one year's worth of consumable parts used for Multi-Shot pyrolyzer.
PY-K313D	Consumable part set 3030AS	This is for EGA/PY-3030D and contains one year's worth of consumable parts used for Auto-Shot Sampler.
PY1-2210	GC/MS connector kit	This kit enables Ultra ALLOY metal capillary column to be used with a magnetic field MS and connection to fused silica capillary column. This is not needed for Q-pole MS.
MS402180	Vent-free GC/MS adapter kit-50 (Agilent, JEOL, Shimadzu)	The adapter allows switching between separation column and EGA tube without opening MS detector to air. A great time saving.
MS402190	Vent-free GC/MS adapter kit-70 (Thermo Fischer MS)	
	Ultra ALLOY metal capillary column	In comparison to fuse silica capillary columns, UA metal capillary columns have greater mechanical strength, heat resistance, and contamination resistance.