

Ver.S2.03

DOUBLE-SHOT PYROLYZER
MODEL PY-2020iD
OPERATION MANUAL



Safety Information

The PY-2020iD has been designed and tested in accordance with EN directives

Whenever the safety protection of the PY-2020iD has been compromised, disconnect the unit from all power sources and secure the unit against unintended operation.

Safety Symbols

This manual contains safety information that should be followed by the user to insure safe operation.

WARNING

A WARNING CALLS ATTENTION TO A CONDITION OR POSSIBLE SITUATION THAT COULD CAUSE INJURY TO THE USER

CAUTION

A Caution calls attention to a condition or possible situation that could damage or destroy the product or the user's work

BEFORE USING THIS PYROLYZER

1. Thank you for purchasing the pyrolyzer from Frontier Laboratories Ltd. Read through this operation manual carefully before attempting any operation, especially if you are a first-time user. This manual is designed to assist you with your daily operations.

DESCRIPTION OF ESPECIALLY IMPORTANT NOTES AND SAFETY PRECAUTIONS ARE FRAMED LIKE THIS.

2. This manual contains categorized descriptions of general notes on pyrolyzer, operations, maintenance, trouble-shooting, and others. We highly recommend that you read this manual along with the operation manual of the gas chromatography you are using.
3. Replacement parts are described in Appendix-1~2. Parts can be obtained from your dealer or directly from Frontier Laboratories Ltd.

Physical appearance and specifications are subject to modifications for product improvements.

PRODUCT WARRANTY

Frontier Laboratories Ltd. warrants the product you purchased in the following terms and conditions.

1. Warranty period 1 year from the date of complete installation.
2. Warranty service During the warranty period, if the product fails due to the defects in material or workmanship, Frontier Laboratories Ltd. will repair the product free of charge.
3. Items not warranted Even during the warranty period, the warranty does not cover failures due to the items listed below, and the product will not be repaired free of charge.
 - 1) Improper installation.
 - 2) Misuse and abuse.
 - 3) Dislocation or shipment after initial installation.
 - 4) Disassembling or alterations.
 - 5) Fire or natural hazards such as earthquakes.
 - 6) Replacement parts and parts whose warranty is limited.

WARNING

FOR YOUR SAFETY

- 1) Wear safety glasses during the installation, troubleshooting and operation.
- 2) Although the pyrolyzer is fully covered by meshed cover for your safety, the temperature of the cover can be 50~80°C when it is in operation. Do not touch the pyrolyzer with bare hands.
- 3) In the event where inspection or removal of the pyrolyzer is required, make sure it fully cools down and unplug the power cable.

CHECKING CONTENTS IN THE PACKAGE

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.

LIST OF CONTENTS

Item	Part No. (P/N)	Qty.	Check	
A. Pyrolyzer		Refer to Replacement parts list (Appendix-1)	1set	
A1	Pyrolyzer	-----	1	
A2*	Double-Shot Sampler	PY1-1020	1	
A3	Liquid Injection Sampler (attached to the pyrolyzer)	PY1-1030	1	
A4	Double-Shot Sampler Stand	PY1-2022	1	
A5	Storage Stand	PY1-3610	1	
A6*	Sample Cup (Disposable Eco-cup SF)	PY1-EC50F	20cups	
A7*	Sample Cup (Disposable Eco-cup LF)	PY1-EC80F	20cups	
A8*	Eco-stick SF	PY1-ES10F	10pcs	
A9*	Eco-stick DF	PY1-ES20F	10pcs	
A10*	Sample Cup Stand (Eco stand)	PY1-EH10	2	
A11*	Eco-cup Picker (Eco pick-up)	PY1-EP50	1	
A12*	Quartz Pyrolysis Tube	PY1-2018	3	
A13*	Graphite-Vespel Ferrule for ITF (6mm id) (5 pcs/pk)	PY1-7911	1	
A14*	Interface Needle Assembly (w/2 spare needles)	PY1-3510	1	
A15*	Interface Needle set (3 pcs/pk)	PY1-1174	1	
A16*	Pretreated Septum (5 pcs/pk)	PY1-7302	3	
A17*	Heat sink adaptor (screw 2ea,hexagon wrench 1.5mm)	PY1-3570	1	
A18*	Level vial	PY1-7131	1	
A19*	Septum For liquid sampler	PY-2025	5	
B. Temperature Controller				
B1	Temperature Controller	-----	1	
B2	AC Power Cable	PY1-7003	1	
B3	Nylon Tube (for cooling air, 1.5m)	PY1-7801	2	
C. Accessories				
C1	System Check Column (5% Methyl silicon, 30m (0.25mm ϕ) 0.25 μ m)	UA5-30M-0.25F	1	
C2*	Standard Sample (polystyrene solution 6 μ g/ μ l contains methyl ester, 0.5ml)	PY1-4905	1	
C3*	Tool Kit (tweezers, wrench, hexagon screwdriver, O-ring (SM-6))	PY1-K020	1	
C4	Deactivated Metal Capillary Tube for EGA Ultra ALLOY [®] 2.5m (0.15m id)	UADTM-2.5N	1	
C5	Operation Manual (this manual)	PY1-7015	1	
C6	Communication Cable (RS-232C, D-sub 9 pin female)	PY1-7811	1	
C7	Control Software (CD ROM)	PY1-5801	1	

* Indicates that the item is included in the accessory container.

OPTIONS (see Chapter 8 for other replacement parts)

- 1) Sample Cups
(Platinum sample cups for Single-Shot Small (SS) and Large (SL)
for Double-Shot Small (DS) and Large (DL) are available)

- 2) Auto sampler stand for HP5890A (specify one from the list below)
 - a. Auto sampler stand (Agilent Part No.: 18597-60255, newest type)
 - b. Auto sampler stand (for older type than a. above)
 - c. Mounter integrated with 100-sample tray

- 3) Mounting Kit for Shimadzu GC (available for GC-17/18/2010/14)

- 4) Selective Sampler (SS-1010E)

- 5) Carrier Gas Selector (CGS-1050E)

- 6) F-Search Ver.2 (Polymer EGA and Pyrogram-MS Library, ADD-MS Library)
(PY-1082E)

- 7) MicroJet Cryo-Trap (MJT-1030E)

- 8) Auto-Shot Sampler (AS-1020E)

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CHAPTER 1 ABOUT DOUBLE-SHOT PYROLYZER

1.1 BACKGROUND OF DEVELOPMENT

Pyrolysis Gas Chromatography (Py-GC) allows analysis of almost all sorts of materials including insoluble materials and complex materials at trace levels without any pretreatment of samples, and information otherwise unobtainable by other techniques can be obtained; therefore, Py-GC technique is becoming more important technique in the area of polymer characterization. However, pyrograms obtained by this technique have problems with reproducibility and difficulties in eluting polar and high boiling species.

We at Frontier Laboratories have addressed these problems and developed a highly improved pyrolyzer based on a technique in which a small cup loaded with a sample is dropped into a small sized vertical furnace by gravitational free-fall at room temperature, affording a pyrolysis time of less than several tens of milliseconds. This system is designed to provide precise temperature control and minimal condensation of pyrolyzates in the system with unparalleled reproducibility. In 1992 we commercialized Double-Shot Pyrolyzer (PY-2010D).

This double-shot pyrolyzer offered completely new dimension in polymer analysis in which volatile components and fragments originated from polymer pyrolysis can be separately analyzed. This feature was not available in conventional pyrolyzers. To accomplish this, volatile components are first analyzed at 100 ~ 300°C at a programmed rate or at a constant temperature, this is followed by instantly heating the basic polymer to 500 ~ 800°C by dropping the sample into the pyrolysis furnace with a free-fall mechanism. This double-shot pyrolyzer offers two techniques on a single system. We commercialized conventional instant pyrolyzer with the free-fall mechanism as Single-Shot Pyrolyzer.

In 1993 utilizing the Double-Shot Pyrolyzer with highly precise temperature controlled programming, we proposed the Evolved Gas Analysis (EGA). With this new technique, volatiles and pyrolyzate gases from a polymer sample evolved continuously at programmed heating process are introduced directly into an analysis system such as GC or mass spectrometer (MS). Because evolved gas curves similar to Thermal Gravimetric analysis (TG) can easily be obtained with this technique, EGA is currently widely used in research laboratories for detailed analysis of thermal and chemical behavior of polymers, and also in factories as a quality control method.

Only for the past 6 years, 350 units of the Double-Shot Pyrolyzers have been installed both domestically and internationally, and in comparison with other methods such as filament and Curie point methods, this device has been proven to be of high reproducibility as well as high reliability.

Ultra ALLOY[®] metal capillary columns we have developed show contamination resistance about 4 times greater and 50°C higher thermal resistance in Py-GC technique than with fused silica (FS) capillary columns and are extremely effective in analyses of pyrolyzates ranging from C1 to over C100.

Double-shot Pyrolyzer: patent no. 2742492

Ultra ALLOY[®]: the registered trademark of Frontier Laboratories Ltd.

Our research and development staff has devoted 15 years in the basic research to improve the quality of the Double-Shot Pyrolyzer and have developed the following new products.

a. Deactivated Sample Cups and Sample Picker - deactivated stainless steel cups with 100% retrieving rate at one try (with detachable handle)

b. Disposable Eco-cup – By achieving deactivation of the stainless steel cups with lower costs, this new cup enabled us to offer lower cost and a single-round usage. Herewith, there is no need to clean the sample cup to do the next analysis. Moreover, there is no need to concern undesirable influences as well as carryover of the previous samples.

c. Carrier Gas Selector (CGS-1050E) - switchable between two carrier gases

d. Purgeable Sampler - sampler with purging mechanism

e. GC/MS Adapter (MS604230) - inexpensive adapter with extremely low leakage at 350°C

f. F-Search Ver.2 (PY-1082E) - Polymer EGA and Pyrogram-MS Library, Additive-MS Library

g. Selective Sampler (SS-1010E) - device that selectively introduces components of any temperature range of EGA curve to the separation column

As these peripheral devices have been developed, the double-shot pyrolyzer has created a wide spectrum of applications. Our research and development team have continued further efforts in improving the system and have developed the next generation Double-Shot Pyrolyzer. The new greatly improved system incorporates more thermal analysis techniques allowing more detailed analysis of polymer components, precise temperature control from near room temperature as initial temperature, and capability to control various peripheral devices for mass spectrometer.

1.2 FEATURES OF PY-2020iD

Double-shot pyrolyzer PY-2020iD is the next generation pyrolyzer with much improved pyrolysis furnace and its temperature control over previous model PY-2010D. This manual describes details about this new pyrolyzer. The following summarizes the features of the pyrolyzer.

1. Two important basic performances are guaranteed.

Inactivity of flow path (judged by peak shapes of polar species)

Relative intensities of polystyrene pyrolyzates at 550°C: $14 \pm 1.5\%$

Their fluctuations: within 2% (with Eco-cup SF)

2. Four analytical techniques available in one system.

Single-shot technique

Double-shot technique

Evolved Gas Analysis Technique

Heart-Cut EGA Technique

3. New algorithm that automatically controls the interface furnace temperature makes it possible to program initial temperature of the pyrolysis furnace starting from 40°C.

4. Any segment of evolved gas analysis curve can be automatically sampled.

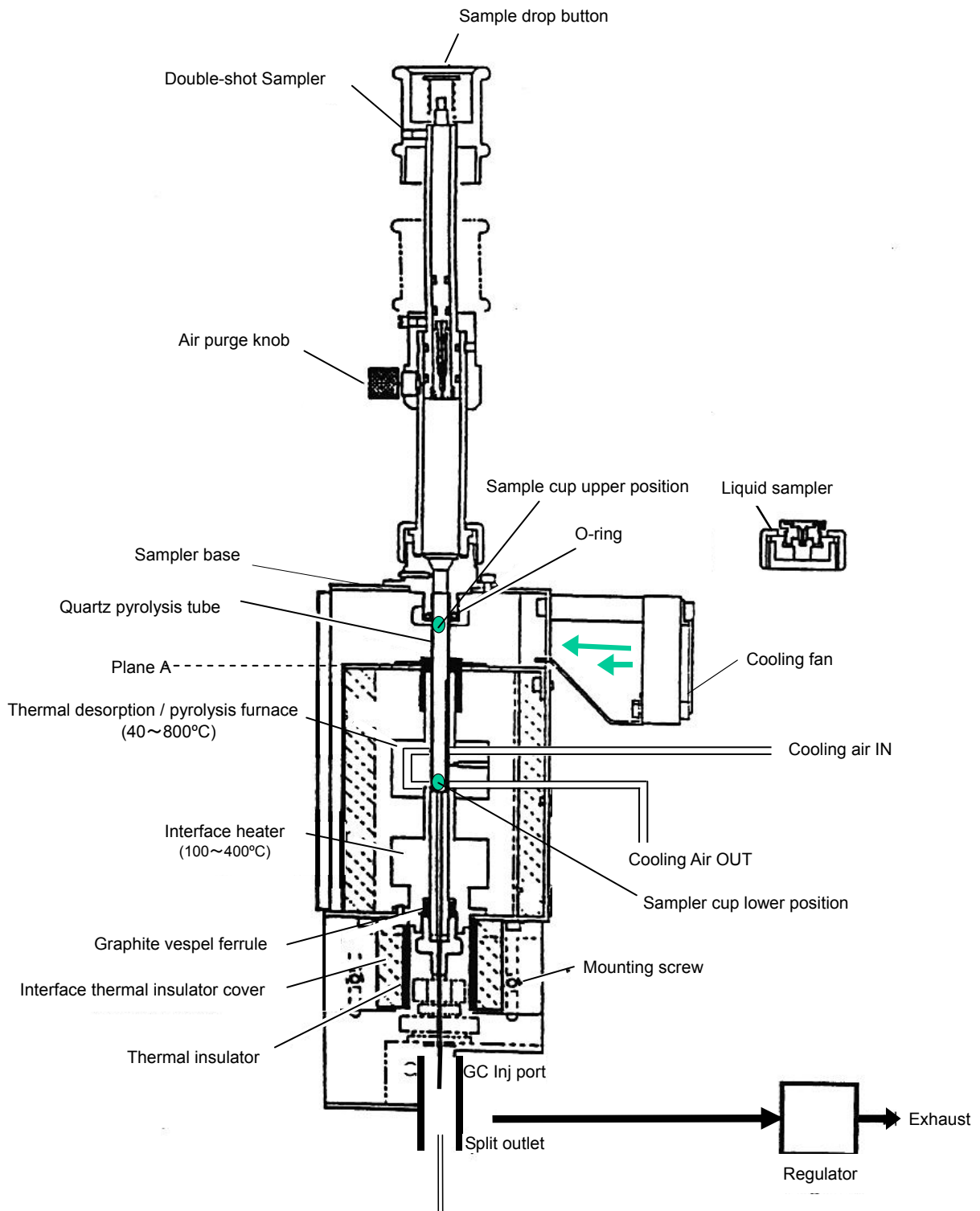
5. PC control software offers easy operation and easy temperature calibration.

Table 1 shows optional peripheral devices that can be attached to Double-Shot Pyrolyzer.

Table1 Analytical Techniques Available for PY-2020iD and With Optional Devices

Optional Device Analytical Technique	GC capillary column	Deactivated capillary tube	Carrier gas selector	Selective sampler	MicroJet Cryo-trap	F-Search	Auto-Shot Sampler
Flash pyrolysis (single-shot)	Best	NA	Best	Good	Best	Best	Best
Multistage pyrolysis (double-shot)	Best	NA	Best	Good	Best	Best	Best
Evolved gas analysis (EGA)	Best	Best	Best	Best	Best	Good	Best

STRUCTURE OF DOUBLE-SHOT PYROLYZER



CHAPTER 2 SPECIFICATIONS

(The system consists of pyrolysis furnace with interface, sampler, temperature controller, standard accessories, and options)

MODEL	Double-Shot Pyrolyzer PY-2020iD (Patent No:2742492)	Single-Shot Pyrolyzer PY-2020iS
1. PERFORMANCE	<i>Guaranteed performance</i>	<i>Guaranteed performance</i>
1. Reproducibility of pyrogram 2. Full deactivation and minimum dead space of Py/GC flow path up to the column outlet	1. Polystyrene Monomer(S) and trimer(SSS) ratio: SS/S(%):14±1%, RSD: less than 2% for Trimer vs Monomer peaks of S pyrogram at 550°C 2. No peak tailing with polar test compounds	Same as left Same as left
2. HEATING & SAMPLING	<i>Vertical type of micro-furnace (Double cartridge heating system)</i>	
• PY furnace (patent pending) Sample introduction Pyrolysis tubing Temp. control range & stab. Cooling time	<i>Heat & Cooling by compressed gas</i> Free fall push button system Quartz 40~800°C (1°C step), ±1°C for each set temp 30 min (600 to 50°C, Auto ITF mode)	<i>Heating only</i> Same as left Same as left 100~800°C (same as left) N.A.
• Interface (ITF) ITF needle Heater Temp Control range Temp stability	Deactivated needle by multi-layer treatment Cartridge heater RT - 400°C (1°C step) ±1°C for each set temp	Same as left
• Sampler	DBL/SGL-Shot : (Sample cup moves up/down for thermal desorption from sample holding position at room temp and/or drops down to the furnace for flush pyrolysis by gravity)	Single-Shot (Sample cup drops down to the furnace for flush pyrolysis by gravity)
• Sample Cup (patent pending) Disposable Eco-cup SF	All deactivated stainless steel cup & stick Small:50 ul (deactivated stainless-steel)	Same as left
Disposable Eco-cup LF	large:80 ul (deactivated stainless-steel)	
• Liquid Sampler	Liquid sample can inject with a regular micro-syringe	Same as left
3. TEMP CONTROLLER	<i>PC control (Install exclusive software in PC for GC or GC/MS)</i>	
• Functional Key	SGL/DBL, EGA multi-temp prog, Automatic EGA Sampling, Interface temp, Monitor, File, Function, Date/Time	SGL/DBL, ITF temp and others
• TEMP Control	Furnace ITF 30~800°C (1°C step) Temp prog. Capability 30~400°C (1°C step) Auto/Manual mode	30~800°C (1°C step) Isothermal 30~400°C (1°C) Manual mode
• Furnace Temp Prog. Prog. Rate Holding time Pyrolysis time	4 steps of multi temp programming 1~100°C /min (1°C /min step) 1~999.9 min (0.1 min step) 1~999.9 min (0.1 min step)	N.A.
• Over heating Protection	PY:820°C, ITF:420°C	Same as left
• External Communication	RS232C (standard for PY-2020 iD)	Same as left
4. Standard Accessories	(one year consumable kit)	
• Pyrolysis tube (Quartz)	2 pcs	Same as left
• Ultra ALLOY Cap. Col	UA-5 (5% diphenyl), 30m (0.25mm id) 0.25um	Same as left
• ITF tube for EGA analysis	2.5 m (0.15 mm id) (Deactivated metal tube)	N.A.
• Standard sample	1 ml (polystyrene 6ug/ul, additives: fatty acid esters)	1 ml (polystyrene, 30 ug/ml)
• Tool, sample cup etc.	1 set	Same as left
5. Miscellaneous		
• Power requirement	AC110V ±10% (50/60 Hz) 4A	Same as left
• Size (W x D x H)/ (kg) Pyrolyzer Temp. Controller	76 x125 x 260 mm / 2kg 160 x 280 x 360 mm / 5kg	Same as left
• Recommended GC	Agilent 7890, 6890GC, Agilent5975 GC/MS, Shimadzu GC-2010, QP2010, QP5050 and others	Same as left

6. Options	<ul style="list-style-type: none"> • Auto-Shot Sampler (AS-1020E) • F-Search Ver.2 (Polymer EGA and Pyrogram-MS Library, Additive-MS Library) (PY-1082E) • Carrier Gas Selector (CGS-1050E, use of air, He, and Selective Sampler is recommended) • Selective Sampler (SS-1010E, any segment of the curve in EGA is selectively sampled.) • GC/MS Adapter (MS604230, max temp : 350°C) • Ultra ALLOY® Metal Capillary column (0.25, 0.5, 1.2mm id; length : 5~60mm, wide selections of chemically bonded stationary phases available)
<p>USER PREPARATIONS</p> <ol style="list-style-type: none"> 1. Gas chromatograph (with split/splitless injection port): Some restrictions exist depending on models. 2. Nitrogen gas or compressed air source (for cooling PY furnace, pressure: 400~600kPa) with 1/8" od or 3mm od tube (copper or stainless steel) immediately accessible from Temperature Controller. (flow rate: 7L/min) 3. PC requirements: OS Windows 98, NT(service pack 5.0 or later), 2000, XP English, Vista, required disc space : 10MB, one RS-232C port (Auto-Shot Sampler requires another RS-232C port) 	

CHAPTER 3 INSTALLATION

3.1 Before Installing Pyrolyzer

Installation of Pyrolyzer (PY) onto Shimadzu GC-2010, as an example, is described here. For installation on the other GCs, see the instruction manual that comes with Pyrolyzer Installation Kit.

1. PC controlled Double-Shot Pyrolyzer (PY-2020iD) requires N₂ source or compressed air for cooling. Make a cooling gas tube with o.d. 3mm or 1/8" available in the vicinity of the temperature controller. The end of the tube is cut off and is connected to the temperature controller with a 1/8" ferrule and a nut.
2. Power source of AC 100V (4A or greater) (a 3P power outlet with a grounding wire required)
3. Allow 20cm wide space at either side of GC or GC/MS. If other optional devices are connected to the Pyrolyzer, reserve space for them.
4. If your GC has multiple injection ports installed, they interfere with Pyrolyzer, because the injection ports are situated close together. If this is the case, Pyrolyzer is connected to the one in the back.
5. If Auto Sampler for liquid samples (AOC) is installed, remove it. AOC cannot be used simultaneously with Pyrolyzer.
6. To check the performance of Pyrolyzer, Ultra ALLOY capillary column (5% phenyl silicone, length 30m, id 0.25mm, film thickness 0.25µm), supplied as a standard accessory, is used. If the performance check is to be conducted with GC/MS, it is recommended, prior to installation of Pyrolyzer, to follow the steps below to prepare for MS analysis in order to save time for installation.

<<Preparing for MS analysis>>

Allow the MS open to air. Attach the column for performance check to the injection port and run He carrier gas through it for 5min. Connect the column outlet to the MS. Then remove the column from the injection port, and immediately stopper the column inlet by sticking it into a septum and start pumping down vacuum of MS. After installation of Pyrolyzer is complete, remove the septum and cut off 1cm of the column end and reconnect it to the injection port. (We recommend that this should be done as quickly as possible.)

WARNING

1. The control cable from the pyrolysis furnace (PY) must be connected to the temperature controller AFTER installation of Pyrolyzer onto GC is completed. (This is to avoid heating of pyrolysis furnace during the installation of Pyrolyzer.)
2. Before starting installation, set the GC injection port temperature to OFF and make sure it is fully cooled down, to avoid the risk of burns.
3. The tip of ITF needle is sharp. Wear a pair of safety goggles and execute cautions when handling.

3.2 Overview of Modification of Carrier Gas Flow Path

Fig. 3.1 shows carrier gas flow schemes before and after the installation of Pyrolyzer. Refer to these schemes at each step of installation.

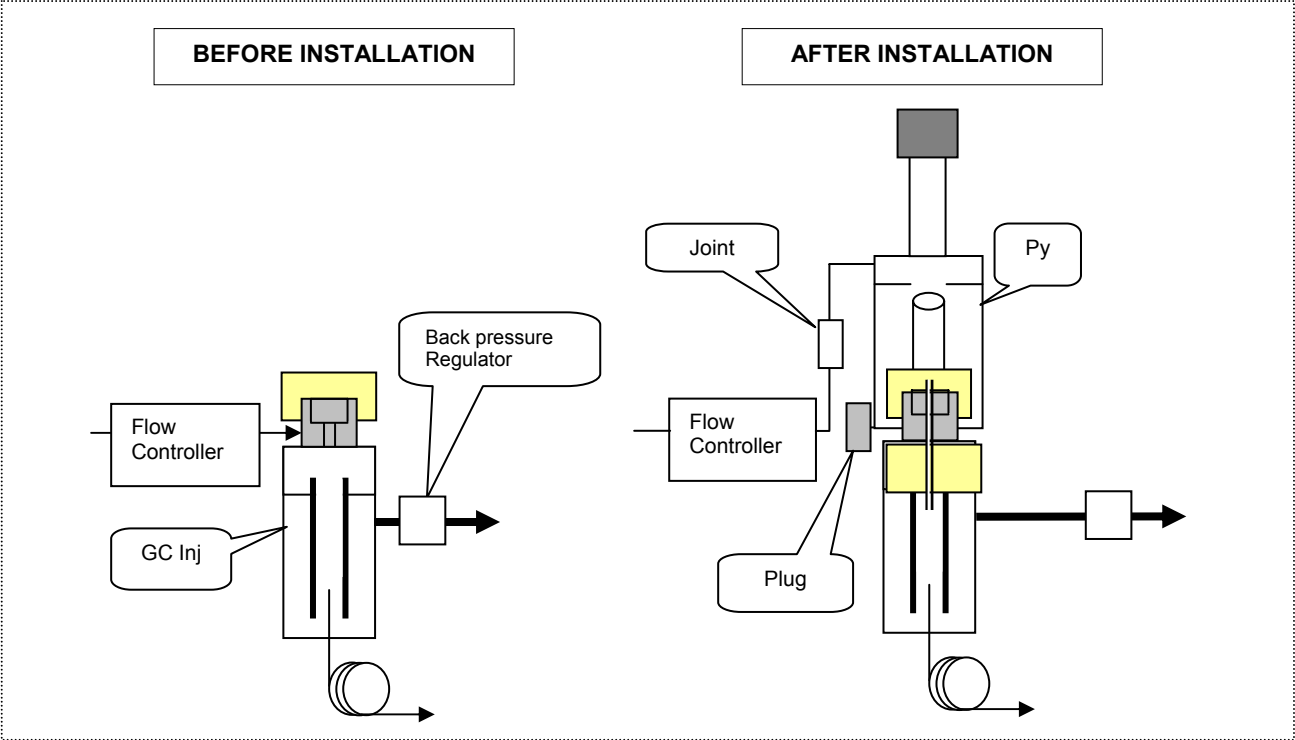


Fig. 3.1 Flow Scheme Before and After Pyrolyzer Installation

3.3 About Installation Kit

For installation of Pyrolyzer onto your GC, an optional installation kit specially designed for your GC is required.

In this chapter, procedures for installation onto Shimadzu GC-2010 are described as an example. See the instruction manual that comes with the Installation Kit for installation onto GC-17 or other GCs.

3.3.1 Contents of Shimadzu GC-2010 Installation Kit (P/N: PY-S2010)

The installation kit contains the following parts A~F (Fig. 3.2).

- A. Carrier gas tube set (for connecting a carrier gas source to Pyrolyzer)
- B. GC-2010 mounting stand (for mounting Pyrolyzer on the GC injection port)
- C. Septum cap set (when Pyrolyzer is used, use the septum cap set contained in the kit)
- D. Adsorption trap set (this is connected to the split vent outlet)
- E. Remote signal cable (for connecting GC Ready and GC Start signals)
- F. Type G socket set (when Pyrolyzer is used, this is used as a cap nut for the carrier gas line)
- G. Instruction manual

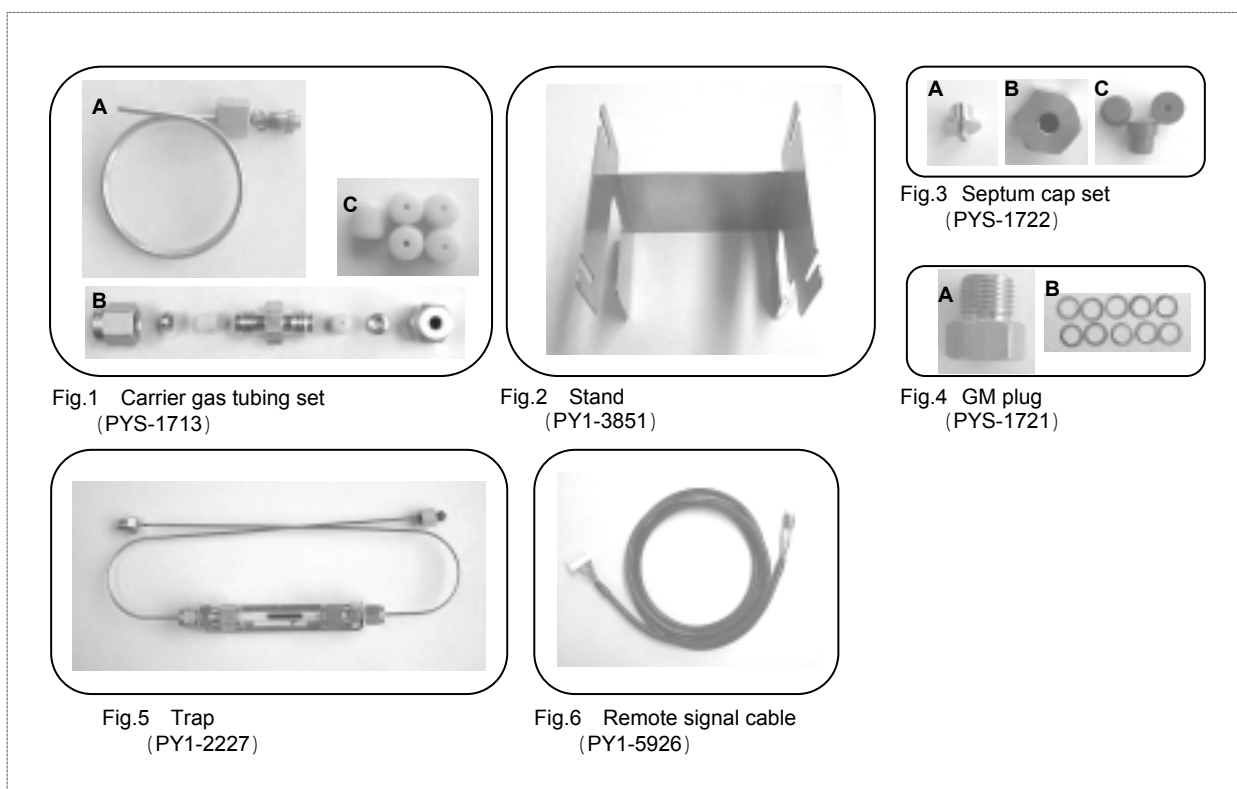


Fig. 3.2 Contents of Installation Kit for Shimadzu GC-2010

3.4 Installation Procedure

STEP 1 Installing adsorption trap (Figs. 3.3 and 3.4)

Disconnect split vent tube and connect the adsorption trap.

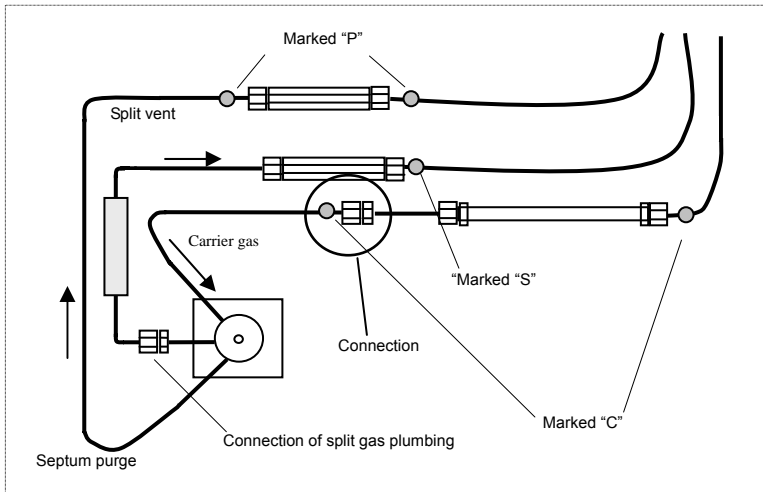


Fig.3.3 Before modification of carrier gas plumbing

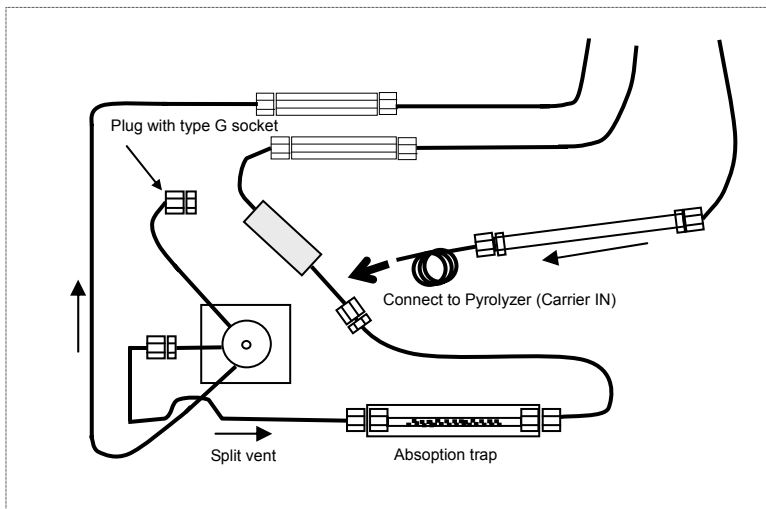


Fig.3.4 Modification of carrier gas plumbing and the absorption trap installation

STEP 2 Modifying carrier gas flow path (Figs. 3.3 and 3.4)

1. Disconnect the carrier gas line at the connectors enclosed in circle in Fig. 3.3 (Indicated by "C") and attach Type G plug (cap nut) to the tube from the injection port, using 3 pieces of aluminum packing together (F (b) of Fig. 3.2).
2. Connect the stainless steel pipe contained in the carrier gas tube kit (F (a) in Fig. 3.2) to the carrier gas line just disconnected. Connect this stainless steel pipe to the carrier gas inlet of Pyrolyzer using a 1/8" union and a rubber stopper with a hole.

STEP 3 Replacing septum caps (Fig. 3.5)

Replace the septum cap, needle guide, and septum of GC-2010 with contents of the septum cap set in the kit.

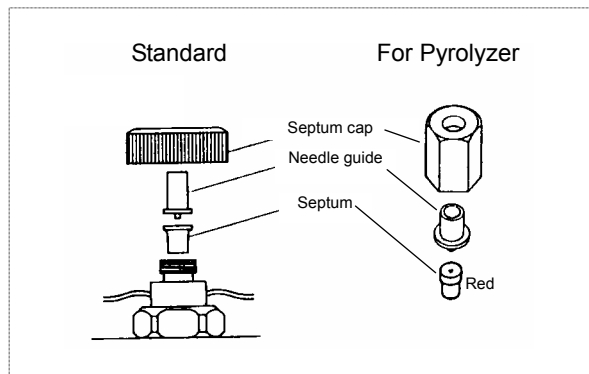


Fig.3.5 Replace septum caps

STEP 4 Attaching Mounting Stand (Fig. 3.6)

Secure the mounting stand onto the injection port using two screws that fix the injection port.

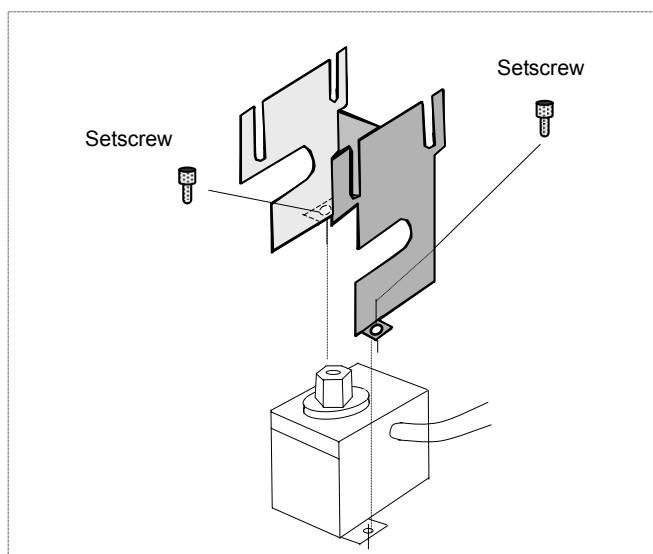


Fig. 3.6 Fixing mounting stand

STEP 5 Removing the stainless steel protective tube in the Pyrolyzer (Fig. 3.7)

The stainless steel protective tube that was used for transportation is removed.

1. Unpack Pyrolyzer from the package and remove the protective mesh cover.
2. Remove storage stand from the Pyrolyzer by loosening four screws located on both sides at the bottom of the Pyrolyzer.
3. Remove the ITF heating block.
4. Loosen the nut that secures the protective tube at the bottom of the Pyrolyzer; then loosen the quartz tube securing nut located at the top. The tube can now be pulled out downward.

<<Do not completely remove the quartz tube securing nut at the top. Just loosen it instead. This will prevent the O-ring from coming off and make insertion of the quartz tube easier. >>

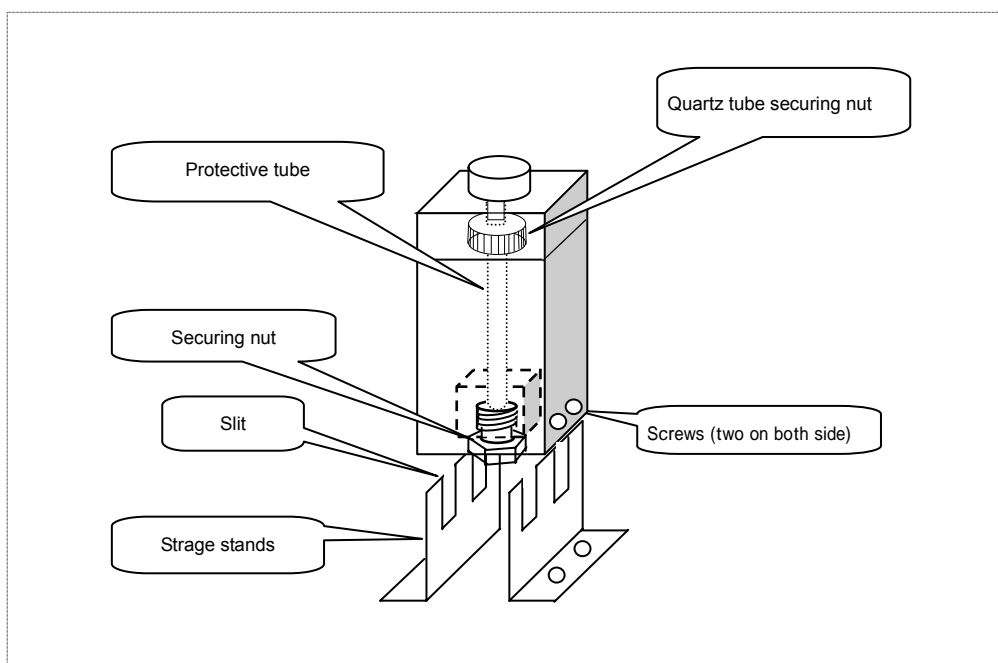


Fig.3.7 Removing protective tube

STEP 6 Attaching quartz tube and ITF needle to Pyrolyzer (Figs. 3.8, 3.9 and 3.10)

A mastery of this procedure is recommended because this is required in daily maintenance routine.

1. Attach ITF needle to the ITF union using a 1/16" nut. After hand tightening, it must be further tighten by 1/8 turn using a wrench. (Fig. 3.8)
2. Insert the quartz pyrolysis tube from the bottom of the pyrolyzer. Start inserting the tube end with larger inner diameter while holding the bottom end and gently turning the tube, until it hits the sampler stand nut at the top of the Pyrolyzer (Figs. 3.8 and 3.9).
3. Hand tighten the quartz pyrolysis tube securing nut at the top. At this point, 1~2mm of the bottom end of the quartz tube should be sticking out from the bottom of the Pyrolyzer (Fig. 3.10).
4. Insert a graphite Vesper ferrule at the end of the quartz tube and hand tightens the ITF union (Fig. 3.8).
5. From the top of the pyrolyzer gently push down the pyrolysis tube with the tip of a supplied wrench (12mm) and continue pushing the quartz tube down until it hits the ITF union (Fig. 3.9). The presence of dead space between the bottom of the pyrolysis tube and ITF union results in peak broadening on the chromatograms.
6. Using the supplied wrench tighten the ITF union by a 1/4 turn with a wrench.

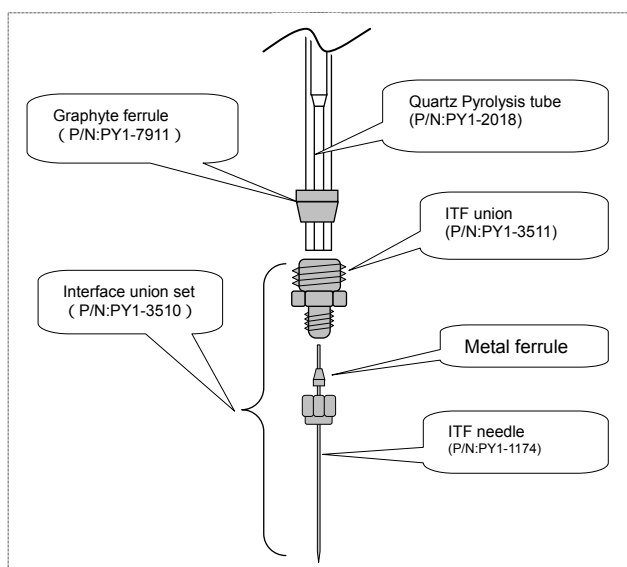


Fig.3.8 Quartz pyrolysis tube and ITF needle

The maximum use temperature of Vesper ferrule mentioned above is 320°C. For higher temperatures use a graphite ferrule (P/N: PY1-7921) in the same manner.

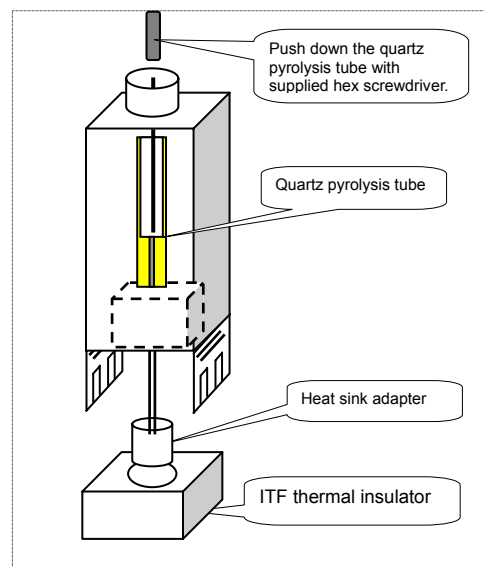


Fig3.9 Install quartz pyrolysis tube

WARNING

The ITF needle at the bottom of the Pyrolyzer is sharp and may cause injury. When the Pyrolyzer is not in use, it should be stored with the storage stand attached.

STEP 7 Gas leakage check (Fig. 3.10)

Gas leakage check is performed before installing Pyrolyzer onto GC.

There are four plausible locations in the Pyrolyzer where gas leakage can occur (Fig. 3.10).

- a) O-ring at the joint of the quartz pyrolysis tube and the sampler stand.
- b) O-ring at the top of Pyrolyzer.
- c) Ferrule at the bottom of Pyrolyzer.
- d) Metal ferrule at the ITF needle.

Pressurized Gas Leakage Tests:

1. Connect the carrier gas tube to the Pyrolyzer.
2. Drive the ITF needle into a septum to plug the needle opening, and then apply a gas pressure.
3. Perform tests of gas leakage using a leakage detector at the locations, a)~d) listed above.

If there is no gas leakage, gas releasing sound will be heard when the septum is removed from the needle. *If the quartz pyrolysis tube is broken, the leakage detector may not be able to detect the leakage.*

NOTE: *Snoop* may be used as a gas leakage detector, but it can cause the insulation failure of the furnace heater. Therefore, use it with a special care not to allow the liquid to get into the furnace heater.

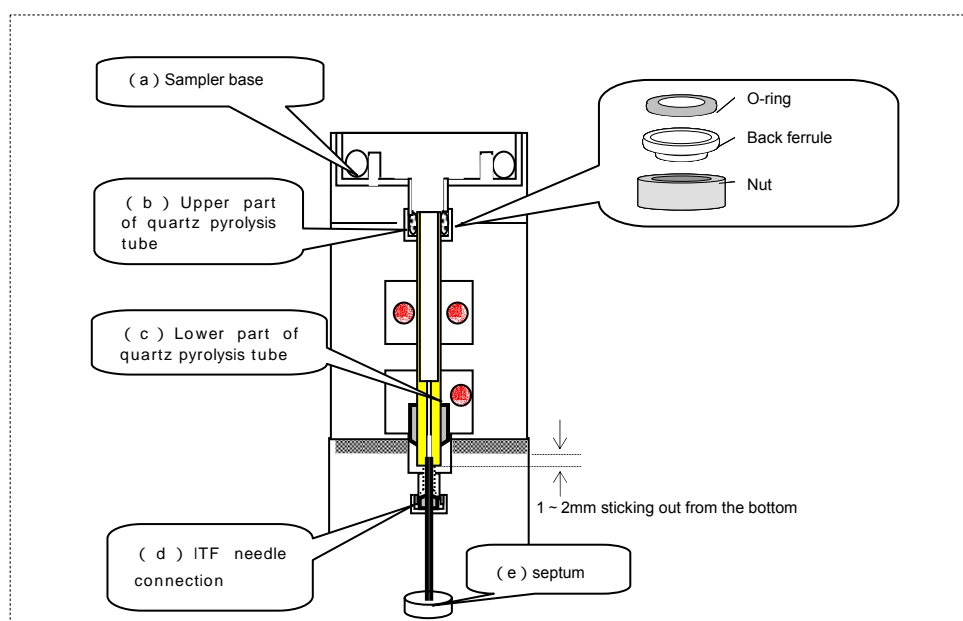


Fig.3.10 Possible locations of gas leakage

STEP 8 Heat sink adapter installation (Fig. 3.11)

Insert the heat sink adapter on to ITF block as shown in Fig.3.11 and fix it with the supplied 1.5mm hex-driver. The slit should face to backward of the furnace for easy accessing to both of two fixing screws. When interface union accommodate to Auto-shot sampler is used, the slit of the heat sink adapter can lead the 1.6mm tube from the union. The clearance between ITF block and the heat sink adapter should be minimized to provide the best performance.

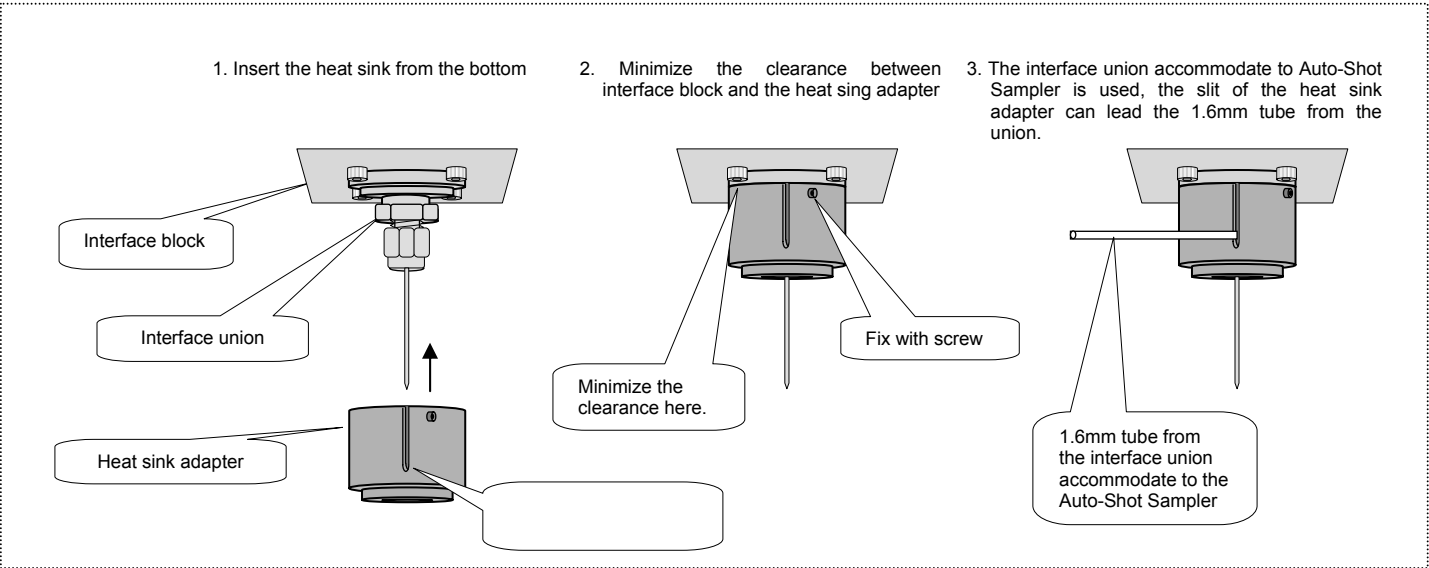


Fig. 3.11 Heat sink adpter installation

STEP 9 Attaching ITF thermal insulation cover (Fig. 3.12)

Once gas leakage tests are finished, put the ITF thermal insulation cover back in place. Then attach the ITF thermal insulation cover and secure it with screws.

STEP 10 Mounting Pyrolyzer (Fig. 3.13)

- (1) Allow the ITF needle to pierce straight into the GC injection port by lowering the Pyrolyzer. At this point, bottom four screws on both sides are engaged into the slots of the mounting stand seated on the GC (Fig. 3.12). Then tighten all four screws using the supplied Allen wrench, making sure that the Pyrolyzer stands in the upright position with keeping the sampler base in a horizontal position (recommended to use the level vial contained in the part box).
- (2) Run the carrier gas and check the column pressure.

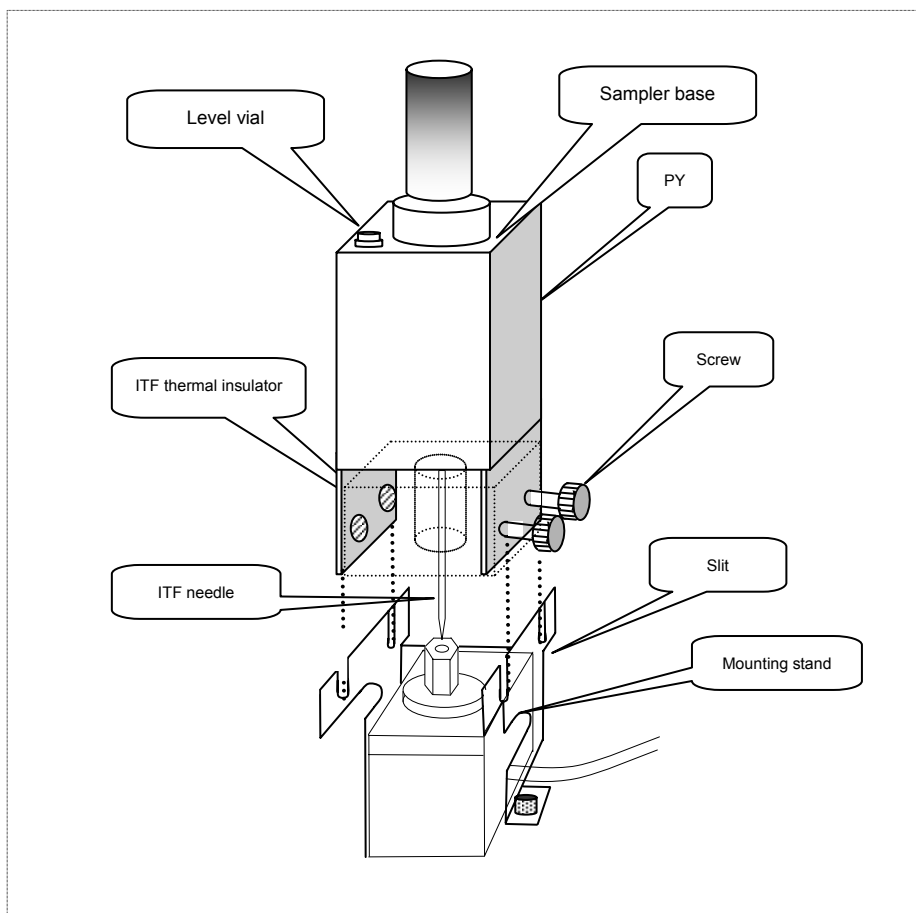


Fig.3.12 Mounting pyrolyzer

STEP 11 Connecting control cables (Figs. 3.13 and 3.14)

Connect Pyrolyzer control cables to the connectors labeled as “POWER” and “SENSOR” located on the back panel of the Temperature Controller. Also connect ground wires to ground terminals.

STEP 12 Connecting cooling gas line to Pyrolyzer (Figs. 3.13 and 3.14)

Connect the Nylon tube from Electro Magnetic (EM) valve located on the back panel of the Temperature Controller (AIR OUT) to the cooling gas inlet tube (COOLING GAS) from the Pyrolyzer.

STEP 13 Connecting remote signal cable (Figs. 3.13 and 3.14)

1. Connect the connectors labeled “GC START IN” and “GC READY” of the supplied remote signal cable to the connectors labeled “START IN” and “READY” of GC-2010, respectively.
2. Connect the other end of the cable to the connector labeled “GC REMOTE” at the rear panel of the Temperature Controller.

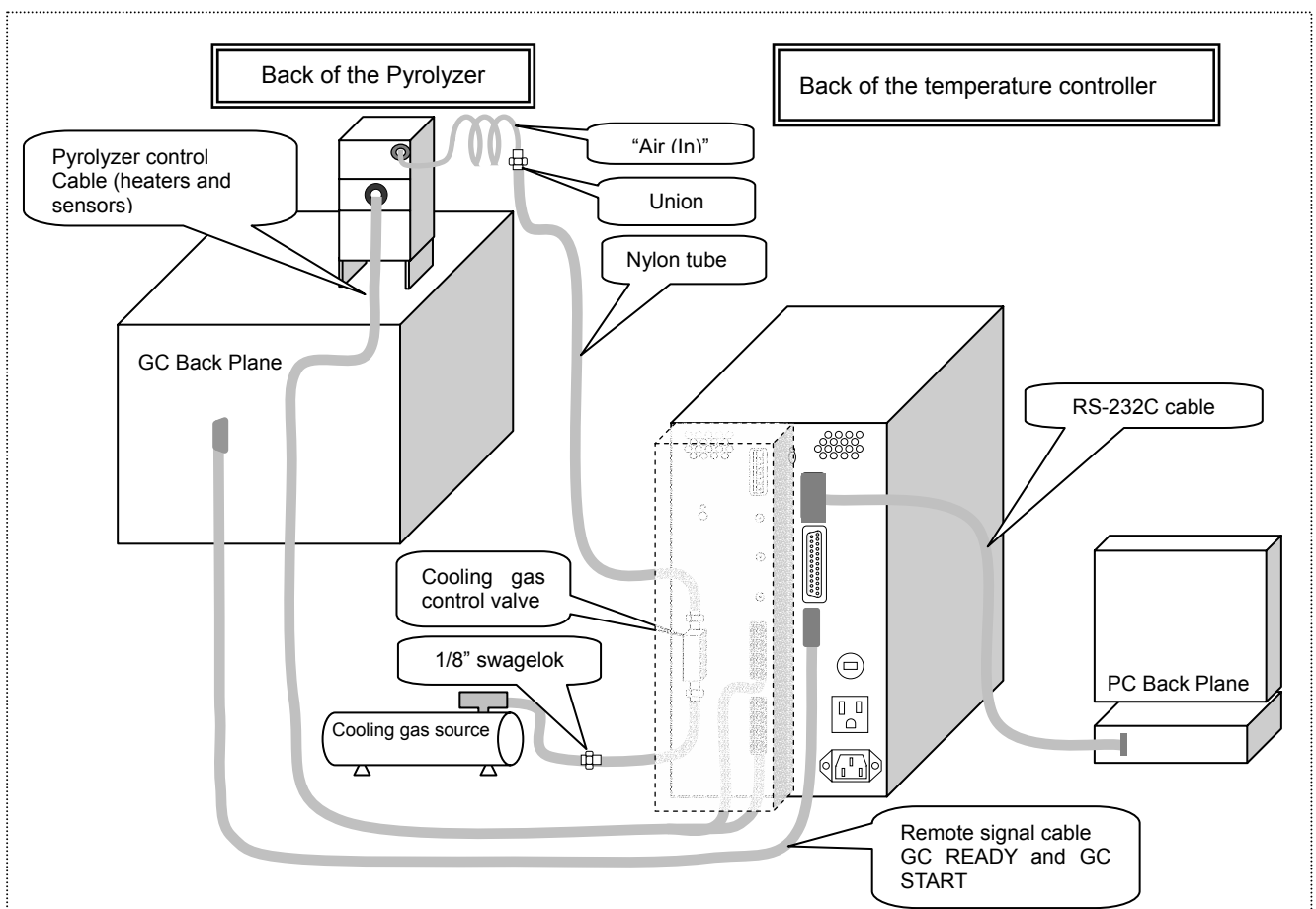


Fig. 3.13 Connection Scheme of Double-Shot Pyrolyzer PY-2020iD, Temperature Controller, and GC

STEP 14 Reversing polarity of GC READY signal

Hit [FUNC] key of GC-2010 and select "6. Setups". Then select "9. Other Setups" to display "Other Setup Screen". Choose "Close" at "Ready Signal Polarity".

NOTE

When GC-2010 is used with other peripheral devices such as Auto Sampler, Head Space Sampler, etc., the polarity of READY signal must be changed so as to comply with the specific device used. See the instruction manual for details.

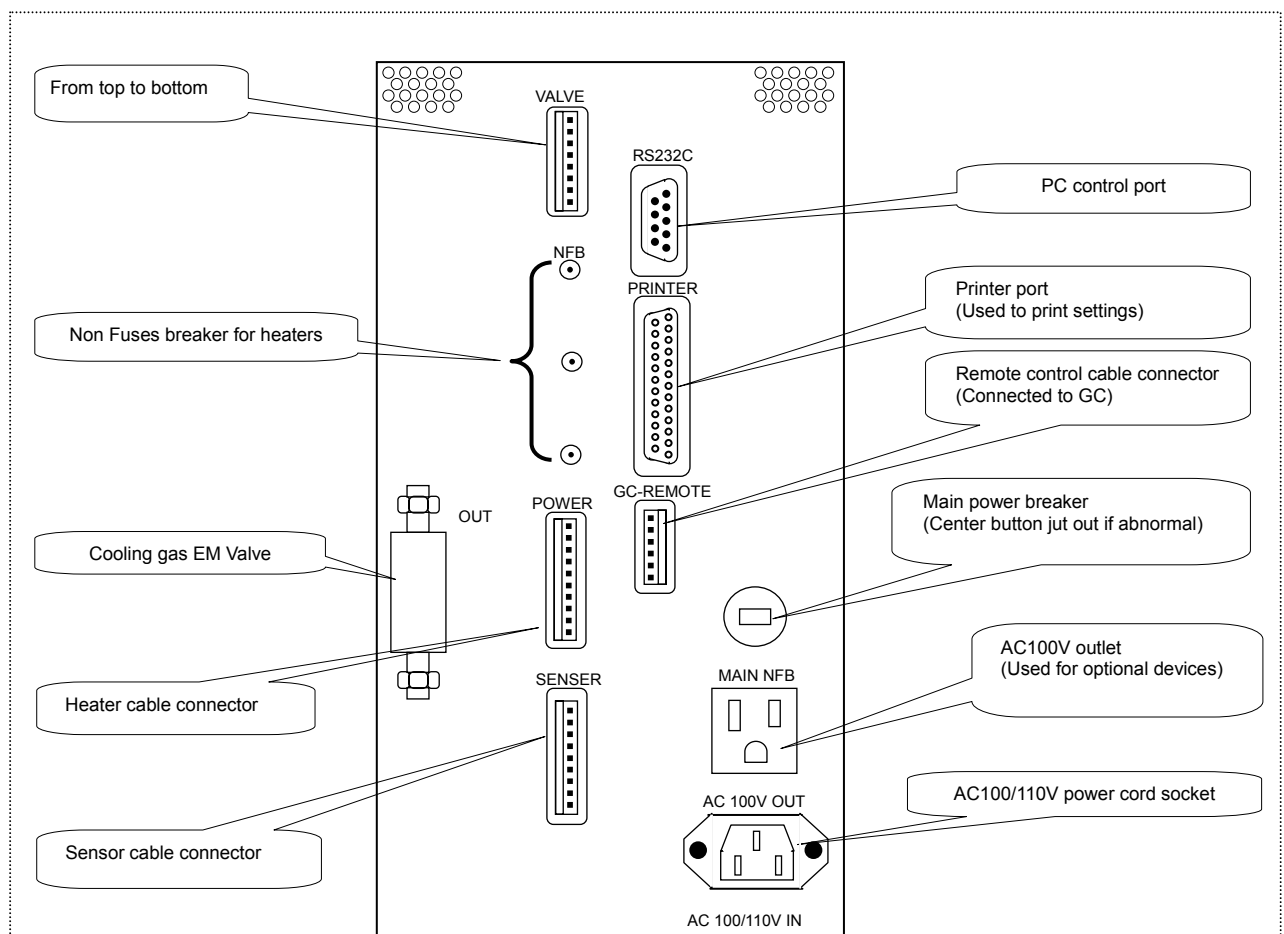


Fig. 3.14 Back Panel of Temperature Controller

STEP 15 Connecting PC to Temperature Controller

1. Using an RS232C cable, connect a serial port of PC (COM1~COM8) to "PC control port" located at the back of Temperature Controller.
2. If Auto-shot Sampler (AS-1020E) is used, connect a serial port of PC (COM1~COM8) to a port located at the rear of the Auto-shot Sampler with an RS232C cable.

STEP 16 Installation of Software

1. Insert "PY-2020iD / iS control software" CD ROM to the CD drive of your PC.
2. Setup program automatically starts and follow the instructions. Find the password at the back of the CD-ROM jacket.

STEP 17 Setting COM Port

If used in Windows NT environment, follow the procedures below to set the COM port.

1. Select "Start" > "Settings" > "Control Panel" > "Ports" from the Windows task bar. A port selection screen will be displayed.
2. Select a port to use and click "Settings" button to show the setting screen for the port selected.
3. Click "Advanced" button to display a screen for advanced settings.
4. Uncheck "FIFO" check box, then click "OK" button.
5. Restart Windows NT.

CHAPTER 4 USING PY-2020iD CONTROL SOFTWARE

4.1 STARTING PY-2020iD AND SCREEN LAYOUT

4.1.1 Starting Up

From the **Start** menu, Select “Program” > “PY-2020iD Control”. After a splash screen (Fig. 4.1) is displayed for a short time, the main screen (Fig. 4.2) is displayed.



Fig. 4.1 Splash Screen of PY-2020iD

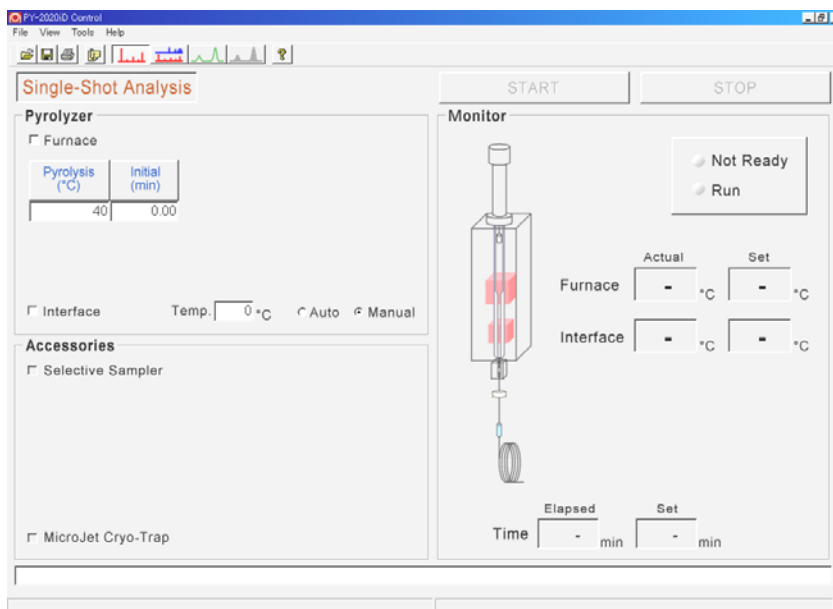


Fig. 4.2 Setting Screen of Single-Shot

4.1.2 Main Screen

Fig. 4.3 shows the main screen of the PY-2020id control software.

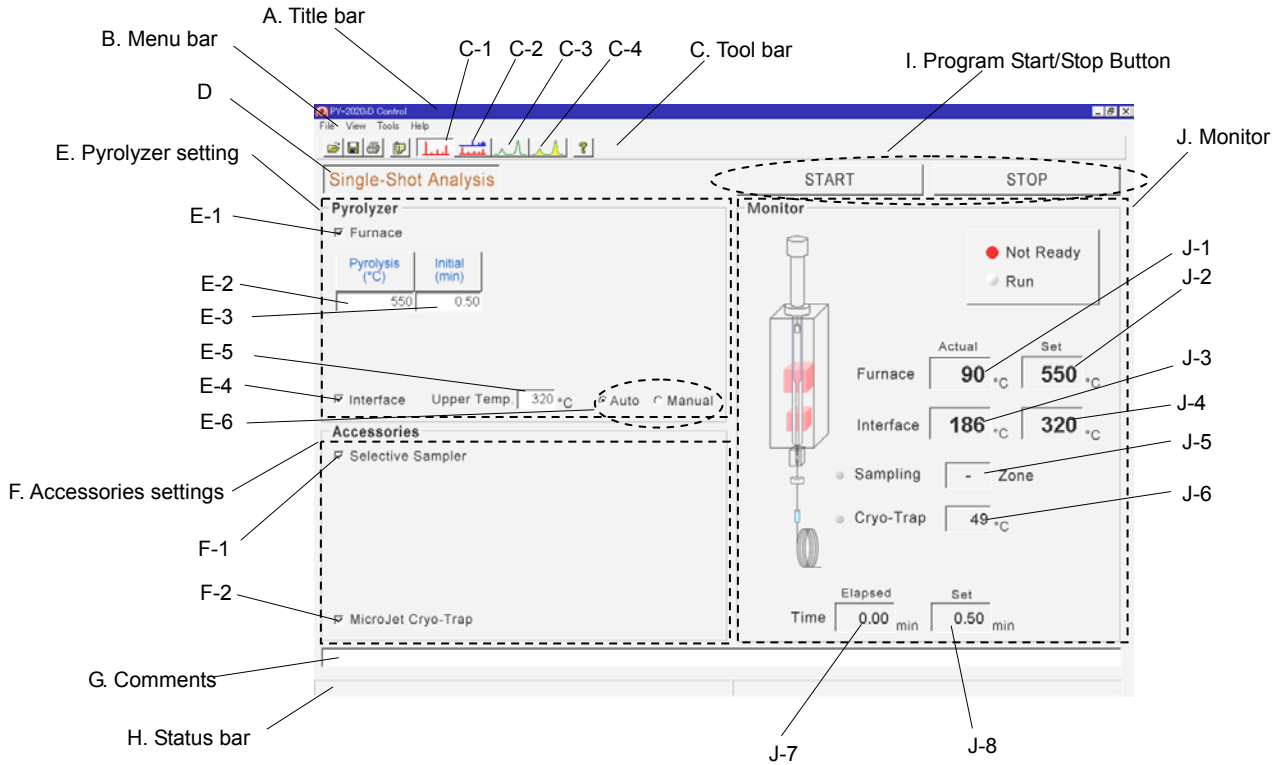


Fig. 4.3 Main Screen

- A. Title bar: Shows method currently used.
- B. Menu bar: Access to options and submenus.
- C. Tool bar: 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 setting: Sets parameters for Pyrolyzer. This area depends on analysis mode selected.
- E-1: Turns On/Off of Pyrolyzer heater. Checking the box turns heater on.
- E-2: Sets Pyrolyzer temperatures.
- E-3: Sets time for automatic control of Selective Sampler or MicroJet Cryo-Trap. Upon starting this program, Selective Sampler performs sampling during the period set here. MicroJet Cryo-Trap operates during the time defined here.
- E-4: Turns On/Off of interface heater. Checking the box turns heater on.
- E-5: Sets interface temperature.
- E-6: Selects a mode of interface control. If "Manual" is selected, temperature is controlled to the temperature defined in E-5. If "AUTO" is selected, it is controlled to 100°C above the Pyrolyzer furnace temperature, however, the temperature set in E-5 is the upper limit temperature.
- F. Accessories setting: Sets parameters for Selective Sampler and MicroJet Cryo-Trap. Appearance of this area depends on analysis mode selected.
- F-1: Select a control mode of Selective Sampler. Check this box, if automatic control is desired. Uncheck it for manual mode.
- F-2: Select a control mode of MicroJet Cryo-Trap. Check this box, if automatic control is desired. Uncheck it for manual mode.
- G. Comments: Used to write down notes for each analysis mode.
- H. Status bar: Shows the progress of the program.
- I. Start/Stop button Used to start or stop the program.

- J. Monitor: Shows operating conditions of each device connected.
- J-1: Shows current temperature of Pyrolyzer.
- J-2: Shows set temperature of Pyrolyzer or final temperature of the heating program.
- J-3: Shows current temperature of the interface.
- J-4: Shows set temperature of interface.
- J-5: Shows the region currently being sampled in Heart-Cut EGA Analysis.
- J-6: Shows trap temperature of Micro jet CryoTrap.
- J-7: Shows the elapsed time of Pyrolyzer being run.
- J-8: Shows time required for Pyrolyzer to complete the operation.

4.2 BEFORE STARTING ANALYSIS

Before starting analysis, set interface temperature and its control mode; and tell the system what Pyrolyzer's accessories (Selective Sampler and/or MicroJet Cryo-Trap) are used according to the procedure shown below.

4.2.1 Setting Pyrolyzer's Accessories

4.2.1.1 Selecting accessory devices used.

1. Select "Tools" > "Instrument Set up". A screen shown in Fig. 4.4 is displayed.
2. If any of the accessory devices is used, check the box to indicate the device used.
3. Select a carrier gas (atmosphere gas) used to heat the sample as shown in Fig. 4.4 A. Helium is normally used. If air is used, refer to section 4.2.1.2 of this manual.
4. Click "Use as Default Value" to make the current settings default.
5. Click "OK" to end.

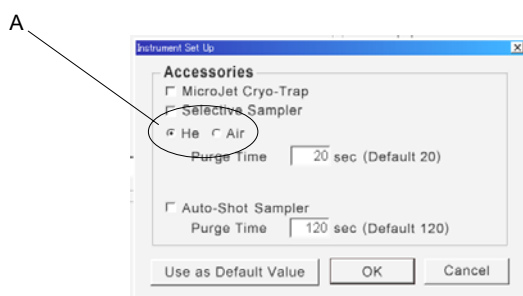
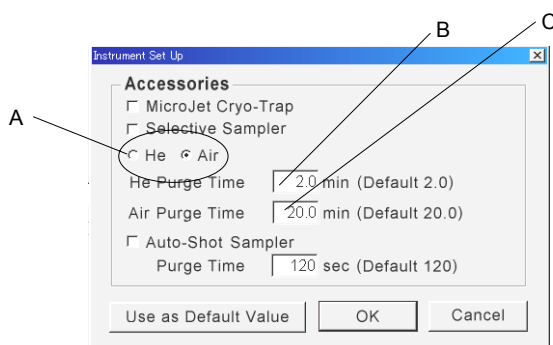


Fig. 4.4 Setting Accessory Devices

4.2.1.2 Using Air as atmosphere gas

In this analysis, the sample is first heated in air, then the carrier gas is switched to helium to purge the column and detector. If automated analysis is desired, MicroJet Cryo-Trap (MJT-1030E), Selective Sampler (SS-1010E) and Carrier Gas Switcher (CGS-1050E) are required. Shown below describes the operation using these devices. Manual operation requires in-depth professional knowledge and extensive experiences.

1. As shown in Fig. 4.5, check the boxes for MicroJet Cryo-Trap and Selective Sampler.
2. Choose Air as shown in Fig. 4.5 A



**Fig. 4.5 Setting Selective Sampler
(Air Used as Atmosphere Gas)**

3. “He Purge Time” in Fig 4.5 B is the time it takes to replace all the all the Helium with Air. Normally default value (2min) is used.
4. “Air Purge Time” in Fig 4.5 C is the time it takes to replace all the Air in column and detector with Helium when heating of a sample has been completed. In particular, if a mass spectrometer is used as a detector, all the air in the mass spectrometer must be replaced before turning on the filament. If this is the case, use default value of 20min.

4.2.2 Setting Interface Temperature

Auto mode and Manual mode are available for setting interface temperature. In Auto mode, the interface temperature is automatically controlled according to the temperature of Pyrolyzer furnace, on the other hand, in Manual mode, it is controlled at a set temperature. Modes are selected in E-6 of Fig. 4.3. Usually it should be set to Auto mode. If Manual mode is selected, the temperature is controlled to a constant temperature determined by the value set in E-5 of Fig. 4.3. In Auto mode, it is controlled 100°C above the temperature of Pyrolyzer furnace, but the interface temperature defined in E-5 is the upper limit temperature. As an example, relation between temperature of Pyrolyzer furnace and interface is shown in Fig. 4.6 where Auto mode is selected in E-6 and the temperature is set to 300°C in E-5; and the Pyrolyzer furnace is heated from 50°C to 500°C. The interface initial temperature is automatically set to 150°C, and the temperature rises up to the upper limit temperature of 300°C, 100°C above the Pyrolyzer furnace temperature.

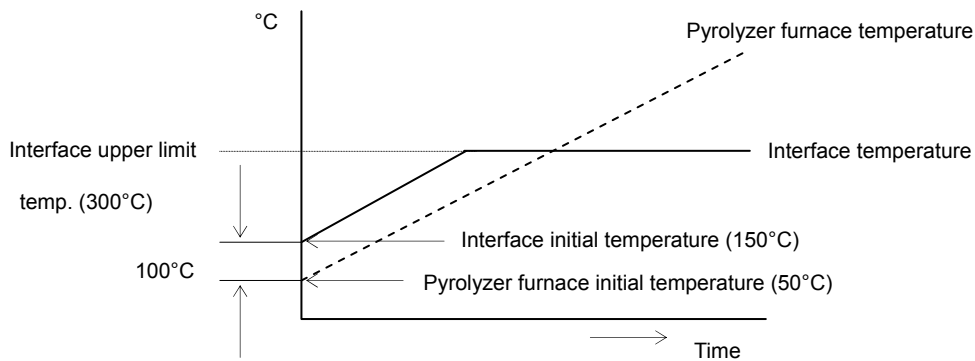


Fig. 4.6 Interface Temperature in Auto Mode

4.3 SETTING ANALYTICAL MODES AND OPERATIONS

4.3.1 Settings for Single-Shot Analysis and Operation Example

Choose “Tools” > “Analytical Modes” > “Single-Shot Analysis” or click the tool bar icon of Single-Shot Analysis (A). A screen shown in Fig. 4.7 is displayed.

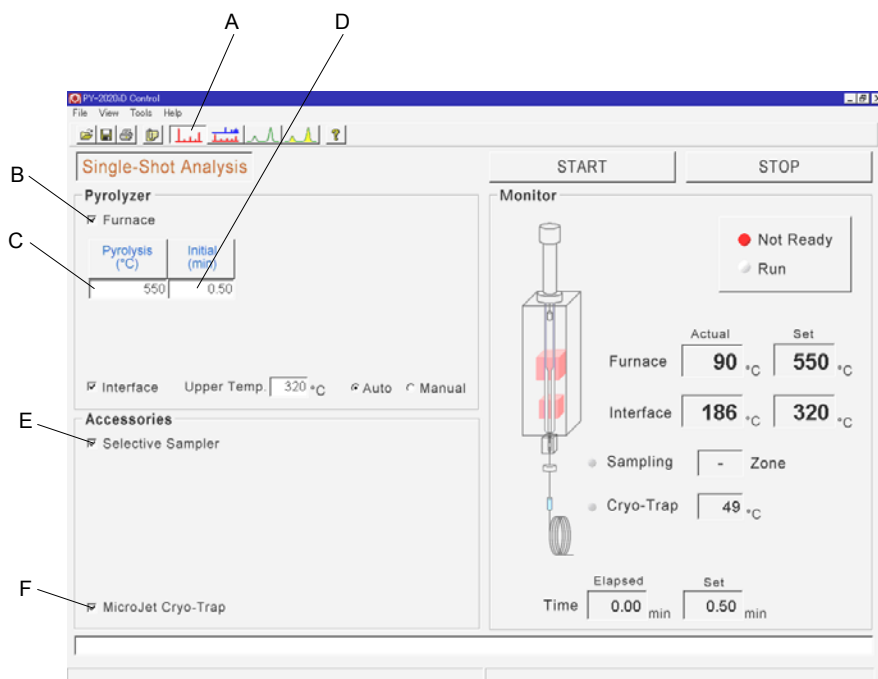


Fig. 4.7 Setting Screen of Single-Shot Analysis

1. Check the Furnace check box (B) to turn on Pyrolyzer heater.
2. Type in Pyrolyzer furnace temperature in C, and allow it to stabilize.
3. Enter a desired time in D. Once the program starts, the Selective Sampler stops sampling after the time defined in D has past. Then MicroJet Cryo-Trap stops cooling and GC ready signal is issued. (Actual pyrolysis time is within several milliseconds.)
4. To auto control the Selective Sampler and/or MicroJet Cryo-Trap, check boxes in E and/or F.
5. Place a sample in a sample cup and attach it to the Pyrolyzer. Once the sample cup is in place, in order to purge the dead space, allow 2min before proceeding to the next step. [If a double-shot sampler is used in Single-Shot mode, hold the top of the sampler and lower it until it hits the single-shot position.
6. Click "START" button.
7. A dialog box shown in Fig. 4.8 is displayed indicating that GC, Pyrolyzer, Interface, Selective Sampler, and MicroJet Cryo-Trap are all ready. Manually press the sample drop button of the Pyrolyzer to drop the sample cup, then immediately click "START" button of the dialog box or hit "ENTER" of the keyboard.

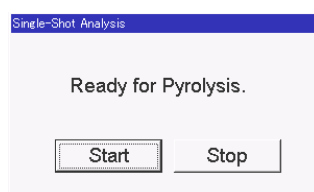


Fig. 4.8 Dialog Box Showing Start of Single-Shot Analysis

4.3.2 Setting Double-Shot Analysis Mode and Operation

Choose “Tools” > “Analytical Modes” > “Double-Shot Analysis” or click the tool bar icon for Double-Shot Analysis (A). A screen shown in Fig. 4.9 is displayed.

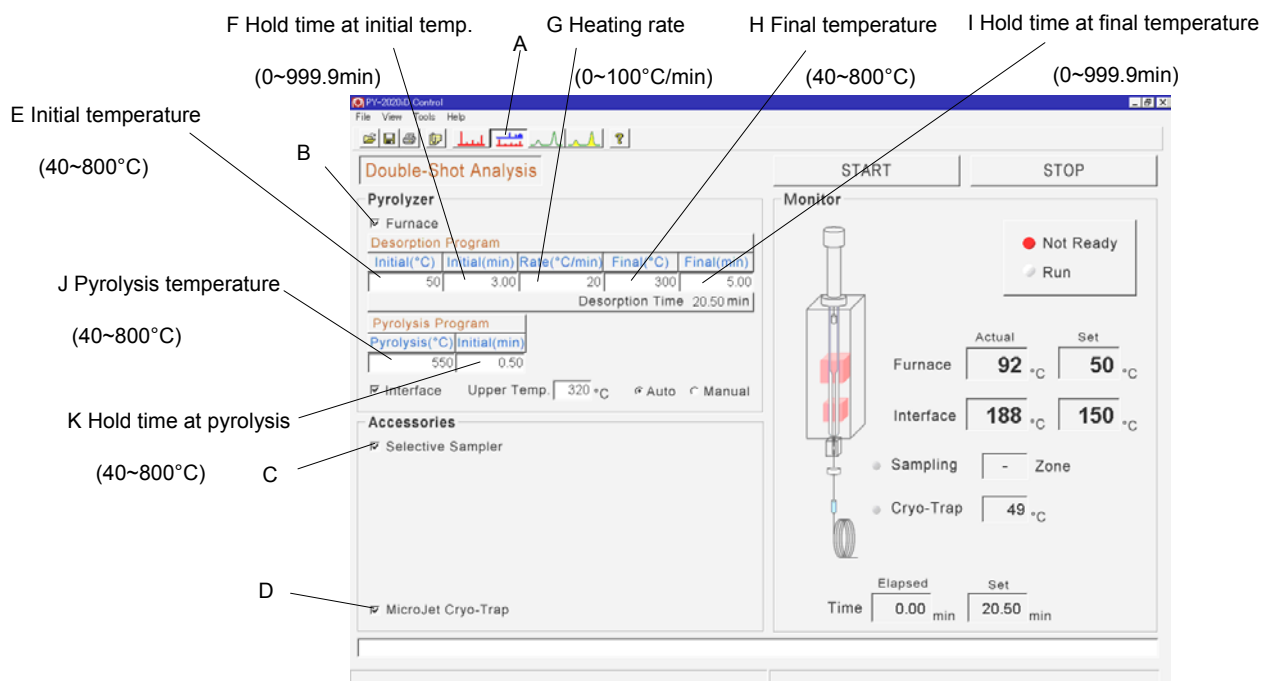


Fig. 4.9 Setting Screen of Double-Shot Analysis

1. Check the Furnace check box (B) to turn on Pyrolyzer heater.
2. To auto control the Selective Sampler and MicroJet Cryo-Trap, check boxes in C and D.
3. Enter parameters in E through K.
4. Place a sample in a sample cup and attach it to the Pyrolyzer. [In Double-Shot technique (combination of thermal desorption and pyrolysis), to purge the dead space, loosen the purge knob shown in page 1-4 then tighten it back after 3 minutes. During this period, GC may generate warning sounds if the carrier gas is controlled by EPC, but if it is within 5 min, GC will stay turned on (Agilent6890, Shimadzu GC-17A.)
5. Click “START” button.

6. A dialog box shown in Fig. 4.10 will be displayed, if GC, Pyrolyzer furnace, interface, Selective Sampler, and MicroJet Cryo-Trap are ready.

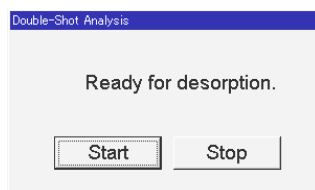


Fig. 4.10 Dialog Box Showing Start of Desorption Program in Double-Shot Analysis

7. Manually lower the double-shot sampler located at the top of the Pyrolyzer to introduce the sample cup to the Pyrolyzer furnace. Immediately following this, click “START” button of the dialog box or hit “ENTER” key on the keyboard. [Thermal desorption sequence will be started.]
8. When thermal desorption program is completed, a dialog box shown in Fig.4.11 will be displayed. Pull up the double-shot sampler slider so that the sample cup is positioned just underneath the sampler. After the column inlet pressure has been stabilized, click “YES” button. [GC will start and the pyrolyzer furnace temperature will rise up to the temperature set in I of Fig. 4.9.]

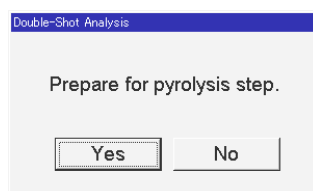


Fig. 4.11 Dialog Box Showing End of Desorption in Double-Shot Analysis

9. Thermally desorbed components are analyzed by GC. When analysis is completed, GC oven will be cooled down to the initial temperature.
10. A dialog box shown in Fig. 4.12 will be displayed when GC oven temperature cools down to the initial temperature and GC issues a ready signal. Immediately after manually pressing the sample drop button of the Pyrolyzer, click “START” button of the dialog box or hit “ENTER” of the keyboard.

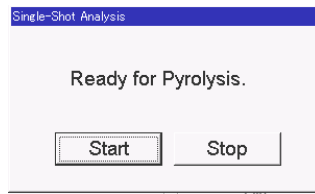


Fig. 4.12 Starting Pyrolysis in Double-Shot Analysis

Fig. 4.13 shows schematic diagram of a series of operational events described above.

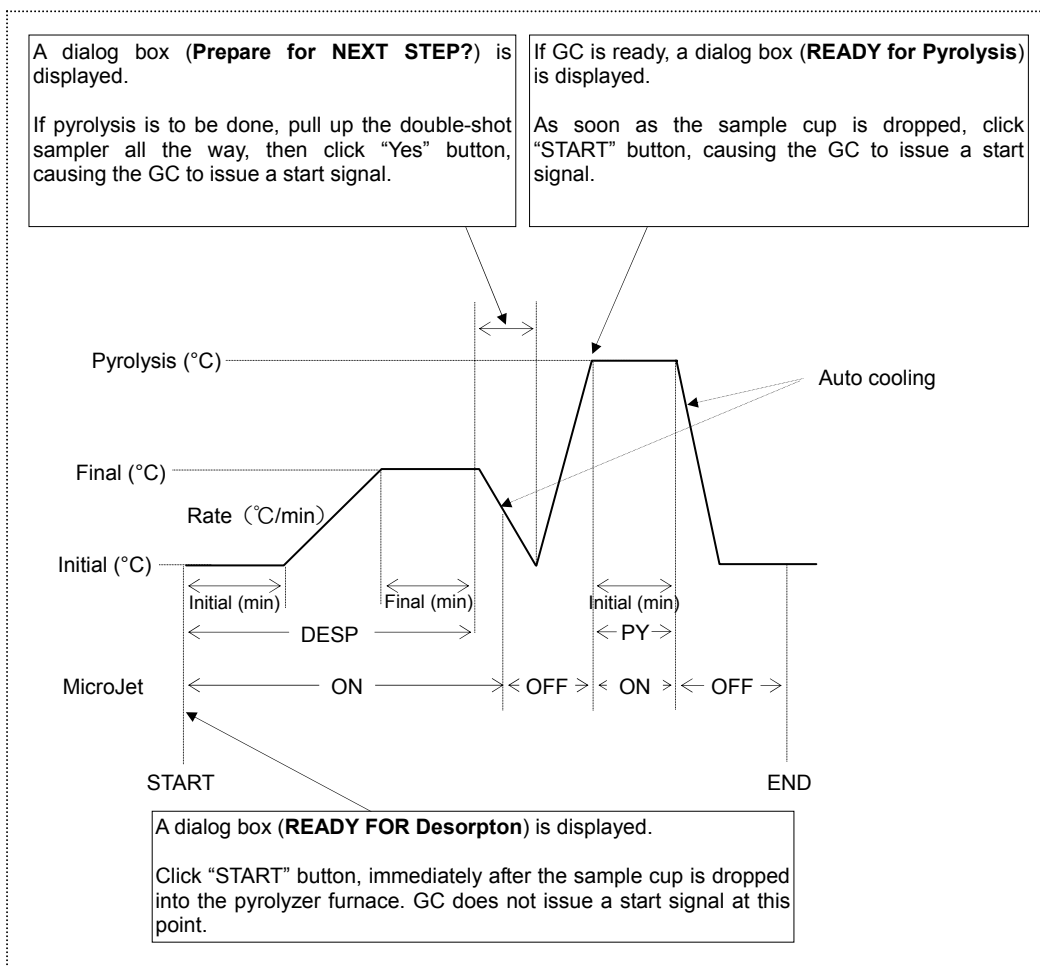


Fig. 4.13 Sequence Program in Double-Shot Analysis

4.3.3 Setting for Direct EGA Analysis and Analytical Procedures

This analytical mode is used for Evolved Gas Analysis as well as for normal multi-step thermal analysis. If “Heart Cut EGA Analysis”, described in the next section, is performed, an EGA curve of a sample is obtained in advance in this analytical mode. Using the Selective Sampler (optional), you can sample out any of the temperature zones of the resultant EGA curve and analyze it by GC all in automated fashion. Before analysis, install an EGA capillary tube (part No.: UADTM-2.5N) into your GC, in place of a separation column, and set the GC oven temperature to 250~300°C.

1. Choose “Tools” > “Analytical Modes” > “Direct EGA Analysis”, or click the tool bar icon for Direct EGA Analysis (A). A screen shown in Fig. 4.14 is displayed.

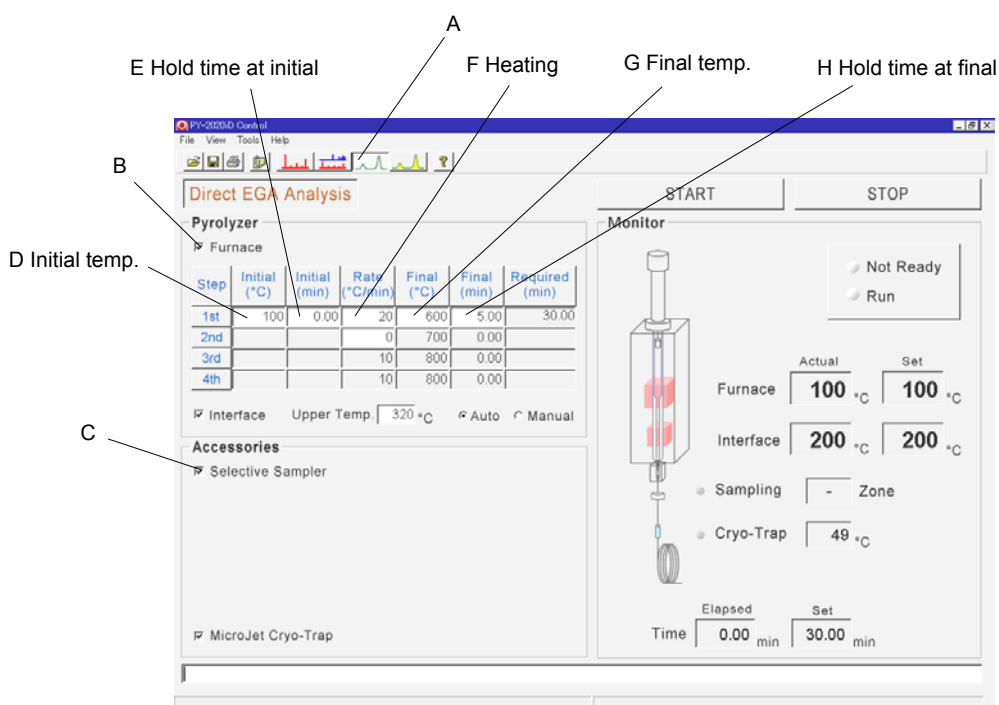


Fig. 4.14 Setting Screen of Direct EGA Analysis

2. Check the box of Furnace to turn on the Pyrolyzer heater.
3. Check the box of Selective Sampler (C), if automatic control is desired.
4. Enter parameters for Step 1 of heating program in D through H.

5. Enter parameters for Step 2 of heating program. Entering "0" for Rate (°C/min) in subsequent spaces will prohibit executions of the steps and beyond. Fig. 4.15 illustrates an example of 4-Step heating program.

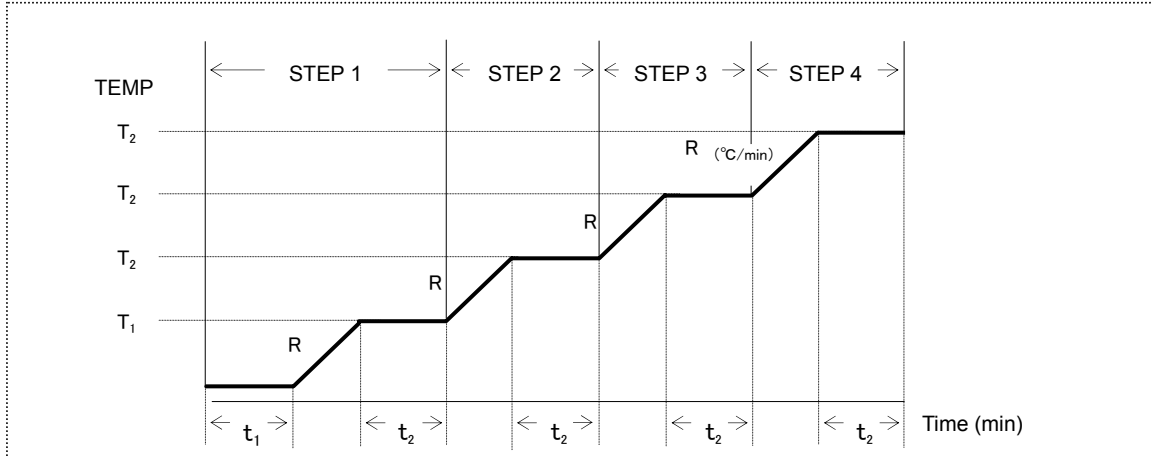


Fig. 4.15 EGA Multi-Step Program

6. Place a sample in a sample cup and attach the cup onto the Pyrolyzer. Wait 2 minutes before proceeding further in order to purge the dead space.
7. Click "START" button.
8. A dialog box shown in Fig. 4.16 will be displayed when GC, Pyrolyzer, interface and Selective Sampler are all ready. Immediately after dropping the sample cup into the Pyrolyzer furnace, click "START" button of the dialog box or hit "ENTER" key of the keyboard.

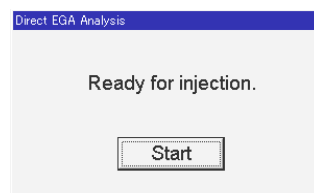


Fig. 4.16 Dialog Box Showing Start of Direct EGA Analysis

4.3.4 Setting for Heart-Cut EGA Analysis Mode and Procedures

A Selective Sampler (optional) is required for this analysis mode. If low boiling components below C₁₅ are to be analyzed, a cooling trap is also required. Frontier Lab's MicroJet Cryo-Trap is recommended.

In Heart-Cut EGA Analysis, based on evolved gas curve (EGA curve) obtained by Direct EGA Analysis, whether each zone range (elution time) can be sampled or not is determined, and sampling zones are automatically introduced into a GC for analysis using a Selective Sampler. An analysis example in which EGA curve of a ceramic composite material obtained by heating it from 100 to 600°C (20°C/min) is shown in Fig. 4.17.

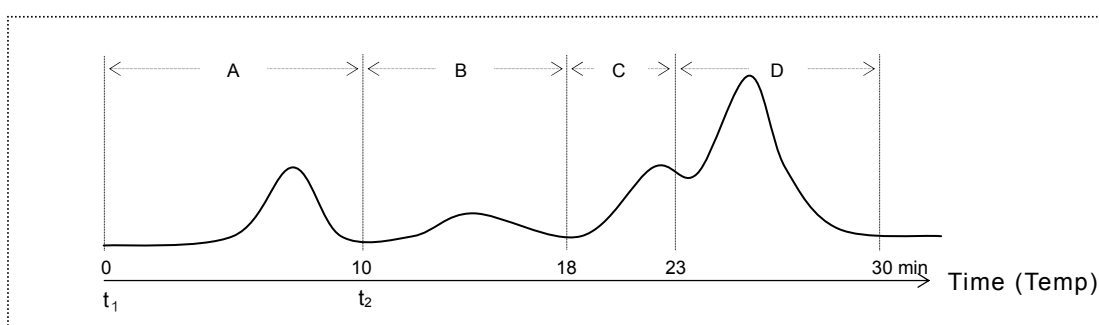


Fig. 4.17 EGA Curve of Ceramic Composite Material

1. Choose "Tools" > "Analytical Modes" > "Heart-Cut EGA Analysis" or click the Heart-Cut EGA Analysis icon in the tool bar. A screen shown in Fig. 4.18 will be displayed.
2. Check the box of Furnace (B) to turn on the heater of Pyrolyzer.
3. Check the box of Selective Sampler (C) and Micro Jet CryoTrap (D), if auto control is desired.
4. Type in parameters for heating program in the table marked by E similarly in the manner of Direct EGA Analysis.
5. Determine the end time of each zone from the EGA curve in Fig. 4.16, and type in the number in F. (Using an MS as a detector and looking at the mass chromatogram, elution temperatures of target components are easier to determine.) [Final (min) of each zone is automatically entered in Initial (min) of the previous zone. Initial of Zone A starts from 0.0min (fixed). Initial (°C) and Final (°C) are automatically calculated and displayed.]

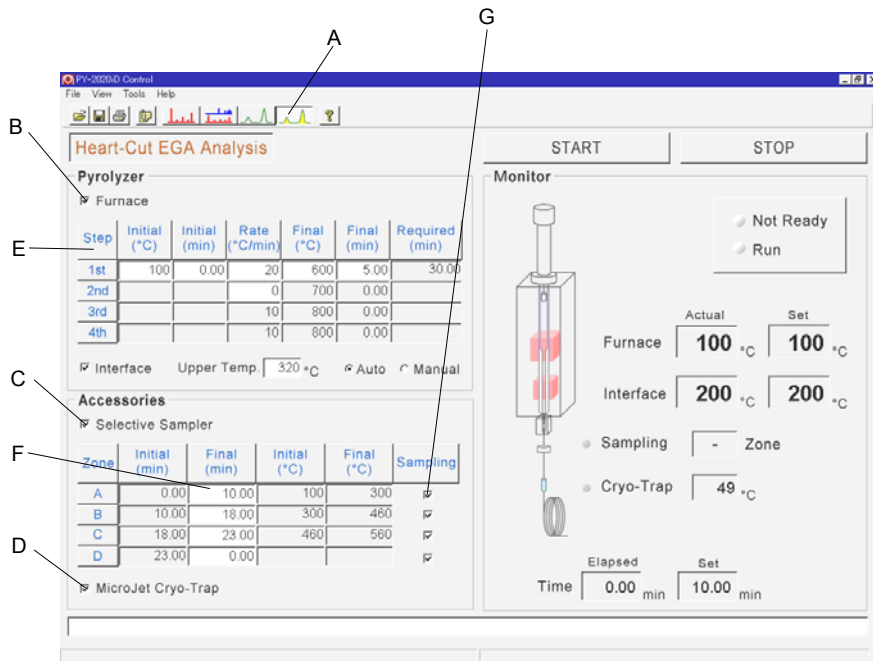


Fig. 4.18 Setting Heart-Cut EGA Analysis Mode

6. If Zone A is to be sampled, check the box of G.
7. To sample Zone B through D, check the corresponding boxes as in the case of Zone A.
8. Place a sample in a sample cup and install it onto the Pyrolyzer. Wait 2 minutes before proceeding further to purge the dead space at the connection.
9. Click "START" button.
10. A dialog box shown in Fig. 4.19 will be displayed if GC, Pyrolyzer, interface and Selective Sampler are all ready. Manually press down the double shot sampler located at the top of the Pyrolyzer to introduce the sample cup into the pyrolyzer furnace. Immediately after this, click "START" button in the dialog box or hit "ENTER" key of the keyboard.

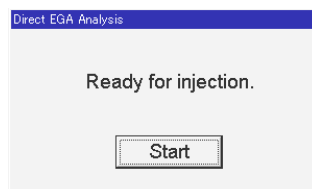


Fig. 4.19 Dialog Box Showing Start of Heart-Cut EGA Analysis

4.4 OTHER FUNCTIONS

4.4.1 Saving and Loading Analysis Parameters

Analysis parameters can be saved with a filename. When all the settings are completed, choose “File” > “Save As” and type in a filename to save the settings in a file. To overwrite a file with the current file, select “File” > “Save”.

4.4.2 Testing Valves and Calibration of Pyrolyzer Furnace Temperature

Choose “Tools” > “Maintenance”. Fig. 4.20 will be displayed asking for a password. Type in “2020” and click “OK”. A screen shown in Fig. 4.21 will be displayed.

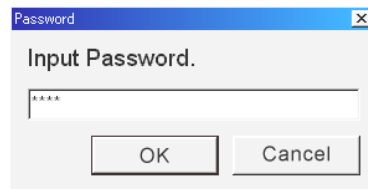


Fig. 4.20 Entering a Password

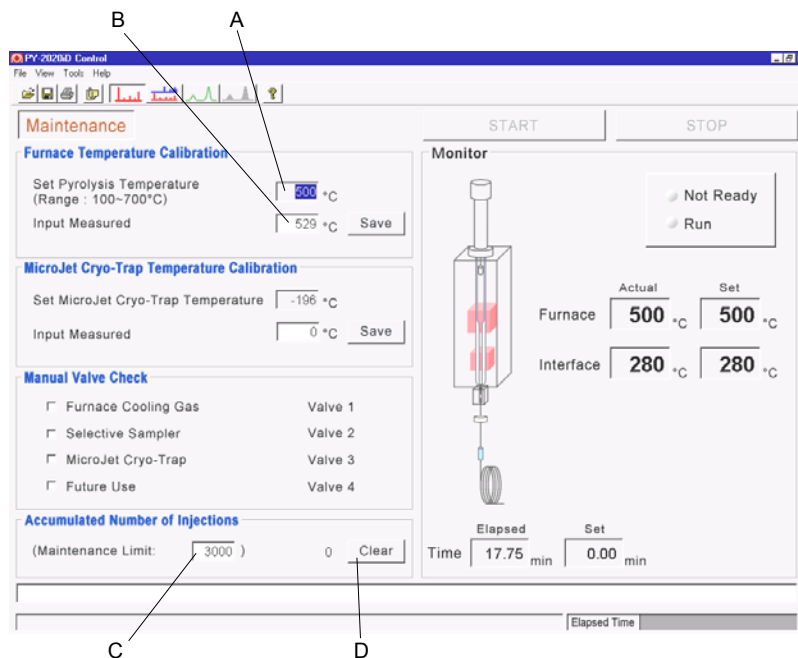


Fig. 4.21 Calibration of Pyrolyzer Furnace Temperature and Tests of Valves

4.4.2.1 Calibration of Pyrolyzer Furnace Temperature

Calibration of Pyrolyzer furnace temperature can be done by entering a temperature actually determined by other thermometer. Because the Pyrolyzer furnace temperature greatly influences analytical results, you should familiarize yourself with the operations prior to proceeding further.

1. Make sure that the pyrolyzer furnace temperature is well stabilized at the set temperature. The set temperature is shown in A, but this is automatically entered with a temperature defined in Single-Shot Analysis. Calibration must be done at temperatures between 100 and 700°C.
2. Type in actually determined temperature in B, using a thermometer of your choice.
3. Click "SAVE" to complete the calibration.
4. Make sure that the temperature displayed matches the actual temperature reading. If not, repeat the operations (1) through (3) described above.

4.4.2.2 Manual Tests of Valves

Operations of valves can be tested by manually turning ON and OFF each valve (EM valves of cooling air for pyrolyzer, Selective Sampler, and MicroJet Cryo-Trap). Closing the screen shown below will bring each valve back to normal operation.

4.4.2.3 Setting Upper Limit of Accumulated Number of Runs

Enter a value for the upper limit of accumulated number of runs (C in Fig. 4.2.1). When the accumulated number of runs reaches this value, a message advising you to perform maintenance of the system is displayed. If so, maintenance should immediately be performed. After it is finished, click Clear button (D in Fig. 4.21) to reset it. The default value is 3000.

4.4.3 Printing Settings

Settings in the currently loaded file can be printed. Choose "File" > "Print". An example of print out is shown in Fig. 4.22.

Analytical Method Information			
Method File Name	:	Default.py	
Created	:	7, Apr, 1999 17:35:25	
Modified	:	24, Sep, 1999 10:15:04	
<i>Heater Control</i>		<i>Direct EGA Analysis</i>	
Furnace	:	1st	
Interface	:	Initial Temperature (°C)	: 40
Interface Auto Control	:	Initial Time (min)	: 1.00
Interface Temperature (°C)	: 320	Rate (°C/min)	: 10
		Final Temperature (°C)	: 250
		Final Time (min)	: 2.00
<i>Accessories</i>		<i>2nd</i>	
MicroJet Cryo-Trap	:	Rate (°C/min)	: 20
Selective Sampler	:	Final Temperature (°C)	: 600
		Final Time (min)	: 2.00
		<i>3rd</i>	
		Rate (°C/min)	: 0.00
		Final Temperature (°C)	: -
		Final Time (min)	: -
<i>Accessories Auto Control</i>		<i>4th</i>	
MicroJet Cryo-Trap	:	Rate (°C/min)	: -
Selective Sampler	:	Final Temperature (°C)	: -
		Final Time (min)	: -
Monitoring Display Interval (sec)	:	1	
Keyboard Lock	:	ON	
<i>Single Shot Analysis</i>		<i>Heart Cut EGA GC Analysis</i>	
Pyrolysis Temperature (°C)	:	Zone A (min, °C)	: 0.00-3.00, 40-40
Initial Time (min)	: 0.20	Zone B (min, °C)	: 3.00-11.00, 40-260
		Zone C (min, °C)	: 11.00-18.00, 260-280
		Zone D (min, °C)	: 18.00-55.50, 280-700
		Method Information : This Method is Default.	
<i>Double Shot Analysis</i>			
<i>Desorption Program</i>			
Initial Temperature (°C)	:	40	
Initial Time (min)	:	0.50	
Rate (°C/min)	:	20	
Final Temperature (°C)	:	250	
Final Time (min)	:	2.00	
<i>Pyrolysis Program</i>			
Pyrolysis Temperature (°C)	:	600	
Initial Time (min)	:	0.20	
Date:	_____		
Sign:	_____		

Fig. 4.22 Printing Settings

4.4.4 Setting Communication Speed

This is provided to help resolve problems involved in the communication between your PC and temperature controller of the Pyrolyzer. To change communication speed between PC and the temperature controller, choose “Tools” > “Options”. Fig. 4.23 is displayed. Select a time (sec) and click “OK”. 2sec is normally used.

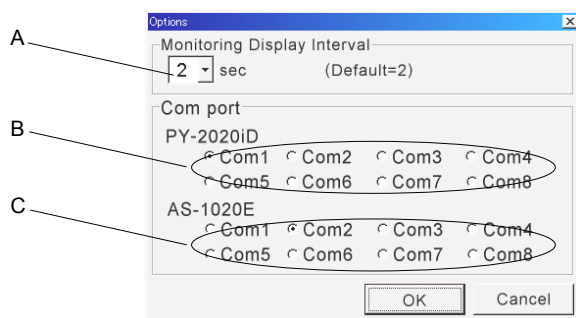


Fig. 4.23 Setting Communication

4.4.5 Setting Timer for Pyrolysis Furnace Temperature by Py Conditioning Table

Select [Tools] – [Py Conditioning Table]. Timer setting table is displayed (Fig. 4.24). Enter desired date and time, pyrolysis furnace temperature, and interface temperature. Hit the Run button to activate the timer. In an example in Fig.4.24, at 6:00am of June 1, 2003, pyrolysis furnace temperature will be 700°C and interface temperature will be 320°C. On the same day at 7:00am, the pyrolysis temperature will be 40°C and interface temperature will be 140°C.

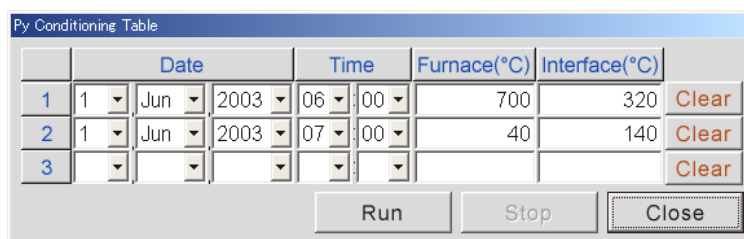


Fig. 4.24 Setting Timer in Py Conditioning

CHAPTER 5 MAINTENANCE

A pyrolysis GC system is largely different from a GC alone system in that each component of the system is closely working with others and together forming a complete integrated system. The reliability of analysis results highly depend on the condition of each component device, therefore, daily and regular maintenance work is extremely important after installation.

5.1 DAILY MAINTENANCE

In daily operation, maintenance work required is cleaning of sample cups, quartz pyrolysis tube and interface needle. Consult the GC manual for the maintenance of your GC.

5.1.1 Pyrolysis Furnace

1. SAMPLE CUPS

Disposable Eco-cup is highly recommended to use only once. Thereby there will be no need to pester with residues of previous sample. If you want to reuse them, physically remove all the residues in the cup with tweezers first. Then wipe the inner surface of the sample cup with cotton swab, soaked with solvent. In addition to that, stir the sample cups with just enough solvent to cover them. The cups should be well-dried before use.

2. QUARTZ PYROLYSIS TUBES

The degree of contaminations of the quartz tubes depends on how they are used. Especially when a large amount of materials are pyrolyzed, the upper part of the tubing (A in Fig. 5.1) is contaminated with high boiling species. After cooling the furnace to room temperature the contaminations are removed by rubbing the inner wall of the tube with a cotton swab dampened with a solvent (Fig. 5.1). For a complete cleaning of the quartz tube, the tube must be removed from the assembly. Note1) Tough contaminations must be physically removed first, then cleaned by red-heating the tube with a burner while blowing air through the tube.

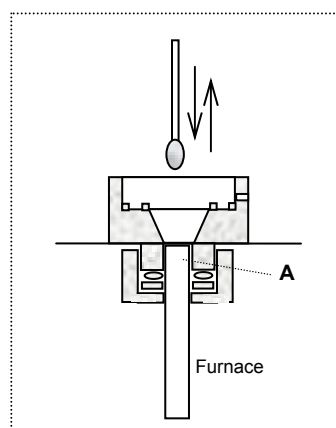


Fig. 5.1 Contamination and Cleaning

3. INTERFACE NEEDLE

Contaminations are mostly tars and stained tars. Contaminated needles are sonicated in a solvent to clean the inside. If this does not show improvements, replace them with new ones. Interface temperature (ITF TEMP) of 280 ~ 320°C is recommended for daily operations.

CAUTION

When removing the quartz pyrolysis tube, it may be difficult to remove it as the interface union and quartz tube are stuck together. If this is the case, heating the union with a burner will help these come off. Vespel ferrule cannot be used.

5.1.2 GC Injection Port

1. SEPTA

When the pyrolyzer is installed onto a GC, the injection port is thermally protected by the interface cover; therefore, the temperature of the septum is higher by at least 50°C than in GC alone operation, and this makes the silicon rubber septum susceptible to thermal degradation. Our experiences show that high quality septa will last about one month at injection port temperature of 300°C. The use of supplied pretreated septum is recommended to allow facile removal of the septum at the next replacement (prevent the septum to stuck).

2. INSERTS

High boiling species from the pyrolyzates travel to the GC injection port via the pyrolyzer. If the injection port temperature is low, such species tend to deposit on the interface needle and on the glass insert. If this is the case, set the port temperature to 350°C and allow it to bake for 2 ~ 3 hours with carrier gas on, but without the column installed. In the normal use check the insert inner wall every one to two weeks to see if there is contamination.

3. COLUMNS

The degradation of columns depends mainly on use conditions (analysis conditions) as well as the nature of pyrolyzates. Tars, reactive gases such as HCl from pyrolysis can degrade part of or sometimes the entire column. Generally tars are often too tough 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 is made by strong acid gases such as HCl or HBr, the column cannot be restored to its original performance. If the sample is considered to generate these acid gases, put glass wool or filler coated with potassium hydroxide in the glass insert to protect the column from exposing to the acid gases.

5.2 REGULAR MAINTENANCE

In addition to daily maintenance work, regular maintenance in every three to four months is recommended. This will ensure trouble-free operations.

5.2.1 Pyrolysis Furnace

1. COOLING FAN

Check to see if the cooling fan starts spinning as soon as the temperature controller is powered on. If the cooling fan does not start immediately, the fan needs to be replaced. The cooling fan does an important role to keep the sample temperature near room temperature when the system is at stand by.

2. LIQUID SAMPLER

Replace the septum. There is no need to perform maintenance work, unless the sample is used daily, however, the septum should be replaced regularly to avoid unexpected troubles.

3. DOUBLE-SHOT SAMPLER

- a) Make sure that the slider moves smoothly. If there is difficulty, move it up and down several times until it moves smoothly, or disassemble it to clean.
- b) Check the O-ring of air purge valve for any damage. This can lead to gas leakage.
- c) The mounting surface of the sampler is mirror finished. Contamination or scratches on the surface can result in gas leakages. When storing the sampler, the surface should be protected from scratches.
- d) Check to see if the cup chuck can securely hold the sample cup, and if pressing the release button can drop the sample cup. If it fails, factory repair may be required. Consult your local agent.

4. SAMPLER BASE (sampler mounter)

Contamination of an O-ring at the sampler base is the cause of gas leakage. Clean it by rubbing with a cotton swab dampened with methanol. Avoid using other solvents such as acetone, methylene chloride, etc.

5.2.2 Gas Chromatograph

1. INJECTION PORT

Upon extended use, the injection port split pipe tends to be clogged by tars. If this is the case, remove it from the GC and clean it by flushing with a solvent through it, then heat it by a burner while running a carrier gas through it. The pipe needs to be checked every three to four months of use.

2. ABSORPTION TRAP

If the acrylic wool in the trap tube at the outlet of the split/splitless injection port gets colored due to deposition of tars, replace it with a new trap tube loaded with activated carbon (Trap set, P/N: PY1-2216), or fill it with new activated carbon (60/80 mesh) and acrylic wool. Do not use glass wool for this purpose, as chips from the glass wool travel to valves and can lead to malfunction of the valves.

5.2.3 Temperature Controller

1. Make sure that connectors for heater power of pyrolyzer, temperature sensor, and temperature controller all have a good electrical contact.
2. Make sure that temperature controller is properly grounded.

5.2.4 When Pyrolyzer is Not Used for a Long Time

If the pyrolyzer is not used for extended period of time, the pyrolyzer removed from GC should be stored in a nylon bag, so that the cartridge heaters are protected from moisture.

CHAPTER 6 TROUBLESHOOTING

Symptom, causes, and their solutions are summarized below.

6.1 ABNORMAL PYROGRAM (CHROMATOGRAM)

SYMPTOM	CAUSE	SOLUTION/CHECK POINTS
1. NO Peaks	1.1 Gas leakage	See Operation Manual 6.3 for details. Check GC injection port, detector, and column connection.
	1.2 No signal from GC detector	Consult GC manual for solution.
2. Broadened peaks	2.1 Deteriorated column	Replace with new one and perform column performance test using standard.
	2.2 Abnormal diffusion	Increase split-vent flow rate.
	2.3 Pyrolysis temperature too low	Increase pyrolysis temperature.
3. Tailing peaks	3.1 Deteriorated column	Replace with new one.
	3.2 Abnormal diffusion	Increase split-vent flow rate.
	3.3 Pyrolysis temperature too low	Increase pyrolysis temperature. Recommended for cross-linked polymers (eg.550 to 650°C)
4. High boiling species not eluting out. High boiling species smaller than expected Broadened high boiling species peaks	4.1 Interface or injection port temperature is too low	Increase temperature (200 to 320°C) (Max septum temperature depends on GC used, but do not go beyond 320°C except when absolutely required.)
5. Ghost peaks at high temperatures	5.1 Residues from the previous runs still remain in the column	Use Selective Sampler to limit high boiling components to get into the column. Use column with higher temperature resistance and bake the column every time after use.
	5.2 Broken pieces of septum in the insert	Clean the insert.
	5.3 Contamination in pyrolysis tube	See Operation Manual 5.1.1for details.
6. Polar species not eluting out	6.1 Deteriorated column	Replace the column or check column performance using UA-5 column that is used in installation.
	6.2 Tars in interface needle	Clean or replace the needle. Increase the use temperature.
	6.3 Activated quartz tube due to deposition of inorganics.	Clean or replace the tube.

SYMPTOM	CAUSE	SOLUTION/CHECK POINTS
7. Pyrogramas are not reproducible	7.1 Gas leakage	See Operation Manual 6.3.
	7.2 Improper pyrolysis temperature	Set it to optimal temperature. (Reproducibility depends on samples. Check the system with standard sample and UA-5 column)
8. Septum residues remain when replacing septum	8.1 Over heated septum	Surface treated septum is recommended
9. Raised baseline at high temperatures in program operation (high background)	9.1 Liquid phase of column oxidized by air	For Ultra ALLOY [®] columns (UA-1,UA-5,PY), clean the column with 2ml of methanol. Then bake the column in program mode <60°C (5min.) ~ 10°C /min ~ 400°C (60min.) >. Do not connect the column end to the detector. For other columns, check with your dealers.
10. Split peaks or abnormal peaks. Specially at about C ₂₀	10.1 Sample temperature from pyrolyzer is too high and sample cannot be trapped at the column head uniformly	Fill the liner used in the GC injection port with filler coated with 5% silicon gum. Bake it thoroughly before use. Do not connect the column to the injection port.

6.2 PYROLYSIS FURNACE

SYMPTOM	CAUSE	SOLUTION/CHECK POINTS	
1. Cannot obtain column head pressure.	1.1 No carrier gas	Check carrier gas source valve and pressure. Check total flow rate of carrier gas and assignment of the injection port in use.	
	1.2 Gas leakage	Absence of a column on injection port or gas leakage at the connection of column	Install a column. Check the column seal.
		Gas leakage at the septum	Replace the septum with a new one. Gas leakage may occur at the septum when cooled the Interface of the Pyrolyzer and/or GC injection port after the run. Interface and injection port temperatures are recommended to keep high at your operation temperature (PY temp. should not higher than 600°C) during the system is not in use.
		Absence or looseness of the plug cap on the carrier gas inlet pipe of the GC injection port GC	Carrier gas inlet pipe of the GC injection port should be plugged with a supplied cap. (Refer Operation Manual page 3-3) The cap is not required when the carrier gas-switching valve is installed.
		Absence of sampler	Sampler should be installed on the sampler base of the pyrolyzer.
		Looseness of the sampler or poor seal by foreign objects on O-ring.	Clean the O-ring and/or contact surface of the sampler with a cotton swab dampened with methanol
		Looseness of the seal at the upper part of the quartz pyrolysis tube	O-ring and metal washer should be used to seal this portion. The metal washer is essential to prevent the O-ring to be twisted. Hand tightening will seal this portion sufficiently.
		Looseness of the interface union or worn out graphite vesper ferrule	Securely tighten the union with a supplied wrench. Or replace a graphite vesper ferrule.
		Looseness of the interface needle or worn out metal ferrule	Securely tighten the needle nut. Or replace the interface needle.
		Looseness of the SS adapter (When the SS-1010E is installed)	Securely tighten the SS adapter (attached onto the outlet of the GC injection port). Or replace aluminum washer (See SS-1010E Operation Manual 3-2).
1.3 Clogged interface needle	Replace the interface needle. Clogged interface needle may cause the following symptoms <ul style="list-style-type: none"> ■ Takes longer time to recover the column head pressure when install the sampler. ■ Column head pressure will go down slowly when the PY furnace is open to atmosphere (Normally, pressure dawns instantly) ■ The screw nut of the sampler is tighter than usual to loose it. And gas will blow out when loose the screw nut. 		
2. Column head pressure will not follow the column temperature program when constant column flow mode is used.	2.1 Gas leakage	See 1.2 mentioned above.	
	2.2 Total gas flow rate is not high enough to keep required column head pressure	Set the total carrier gas flow rate more than 50ml/min. When the gas saver is used, minimum total flow rate of carrier gas should add 10 ml/min to the value instructed in GC manual. Because the pyrolyzer always purges the dead volume of the sampler.	

SYMPTOM	CAUSE	SOLUTION/CHECK POINTS
3. Too tight to loose the screw nut of the sampler. And will blow out gas when loose the screw nut	3.1 Interface needle is clogged and the inner pressure became high	Replace the interface needle. See 1.3 mentioned above.
4. Interface needle is clogged frequently	4.1 Interface and/or GC injection port temperature(s) is (are) too low	Increase temperature. Max interface temperature is 320°C when graphite vesper ferrule is in use. Max septum temperature depends on GC in use. But do not set it beyond 350°C except when absolutely required.
	4.2 Cooling point exists between the outlet of the quartz pyrolysis tube and the GC injection port because of improper Pyrolyzer installation	Interface needle is completely covered by cover and not visible from outside when the Pyrolyzer is installed properly.
5. Quartz pyrolysis tube will be broken easily when the interface union is tightened.	5.1 Over- tightens the interface union	Excess force may results in breakage of the quartz pyrolysis tube, so work slowly. Hand-tighten the interface union first, and then rotate it by 60° ~ 90° with a supplied wrench.
	5.2 Some extra forces have shifted the furnace alignment.	Extra force should not be needed to insert the quartz pyrolysis tube into the furnace. If you feel difficulties in inserting the tube into the furnace, alignment of the heated blocks at the factory may be required.
6. Stuck quartz pyrolysis tube and interface union	6.1 Stuck because of high temperatures	Heat with a burner and try to take them apart. (Vesper ferrule is cannot be reused.)
7. Column head pressure will dorp as the PY temp becomes high (e.g. Over 600°C) and will be recovered when the temperature becomes low.	7.1 Some extra forces have shifted the furnace alignment.	The total alignment may be required at the factory.
8. High background by air (MS is used)	8.1 Gas leakage.	See 1.2 mentioned above.
	8.2 Fewer purge time after installing the sampler onto the furnace to start the run.	Install the double shot sampler on to the furnace and wait to reach the column pressure at the set point. Loose the purge nut of the sampler to drop the pressure to atmosphere. Repeat this two times and close the purge nut. Then wait more than 2 min. before start the run.
	8.3 Graphite (100%) ferrule is used at the interface union. This ferrule will cause very small leakage and be unavoidable.	The graphite vesper ferrule (P/N:PY1-7911) is recommended (ITF temp. should be set to below 320°C).
9. Heated block(s) is(are) not heated	9.1 Heater (s) concerned is (are) set to OFF	Set it ON (See CHAPTER 4 in Operation Manual)
	9.2 Defective heater(s)	Repair at the factory is required.
10. Slow heating speed (PY)	10.1 One of the double heaters is defective	Repair at the factory is required.

Causes of most troubles while using our pyrolyzer are gas leakage and clogged interface needle and showing the symptoms such as,

- **Bad reproducibility of pyrogram.**
- **Insufficient column head pressure.**
<This symptom sometimes caused by less total flow rate of carrier gas. >
- **Lack of controlling carrier gas flow rate of GC under constant flow mode.**
<This symptom sometimes caused by less gas source pressure. Verify appropriate gas source pressure>

6.3 TYPICAL TROUBLE SHOOTING PROCEDURE

WARNING

Wear safety glasses to protect your eyes while performing the trouble shooting. Use care in handling the interface needle to prevent puncture wounds.

1. **Set the total carrier gas flow rate to 50ml/min. and the column head pressure to 40 ~ 60kPa(0.25mm id., 30m, MS is in use as the detector)**
2. **Remove pyrolyzer from the GC injection port and connect carrier gas tube to GC injection port. Then make sure that the column head pressure reaches to the set pressure value.**

If the column head pressure does not reach to the set pressure value, following causes are possible.

- a Worn out or loose ferrule of column seal. Securely tighten the column nut or replace the ferrule.
- b If the SS adapter is installed, adapter is loose or metal seal is worn out. Securely tighten the adapter or replace the seal. (refer to SS-1010E operation Manual 3-2)
- c The worn out septum. (Replace the septum)
- d Gas leakage at the connection of absorption trap in split vent gas line (if installed). (Securely tighten the nut and replace the seals if needed)
- e Gas leakage at the septum may occur when cooled down the injection port and/or interface (PY) temperature.

3. Install the pyrolyzer onto the GC injection port. Then make sure that the column head pressure reaches to the set pressure value. Use a liquid sampler for the trouble shooting.

CAUTION

Because of the double-shot sampler has gas purging mechanism, it is possible to make a wrong judgement about gas leakage

If the column head pressure does not reach to the set pressure value, perform the gas leakage check by following the procedure described below.

- ① Plug the carrier gas inlet tube to the GC injection port with a supplied cap. (refer to CHAPTER 3 in this manual).
- ② Before installing pyrolyzer on to the GC injection port, connect carrier gas tube to pyrolyzer. While flowing gas through pyrolyzer, plug the end of the interface needle with a septum to increase inner gas pressure. Wait 30 seconds then remove the septum. If you hear the sound of blowing out the gas from the interface needle, there's enough gas flow to obtain column head pressure required. If you can not hear the sound, gas leakage may exist. (For more precise way to check out the gas leakage, a leak detector can be used.)

When the following parts are losing or worn out, gas leakage often occur;

- O-ring, for sealing upper end of the quartz pyrolysis tube (Fig. 6.1 b).
Enough seal can be formed by finger tightening. If needed, replace O-ring (P/N: PY1-2012). Metal washer is also used here, which prevent gas leakage by twisting an O-ring.
- Graphite vesper ferrule, sealing quartz pyrolysis tube and interface union (Fig. 6.1 c).
Securely tighten the interface union with the supplied wrench and if needed, replace the graphite vesper ferrule (P/N: PY1-7911).

CAUTION

Excess force may result in breakage of the quartz pyrolysis tube. So work slowly.

- Metal ferrule of the interface needle (Fig. 6.1 c).
Securely tighten the interface needle nut. If it does not work, interface needle assembly (P/N : PY1-1174) may need to replace.
- Sampler or foreign objects on sampler base (Fig. 6.1 d).
Securely tighten the sampler or wipe out foreign objects on the sampler base or the contact surface of the sampler by cotton swab dampened with methanol. If needed, replace a O-ring (P/N:PY1-7812).

If nothing works, check breakage of the quartz pyrolysis tube (P/N: PY1-2018)

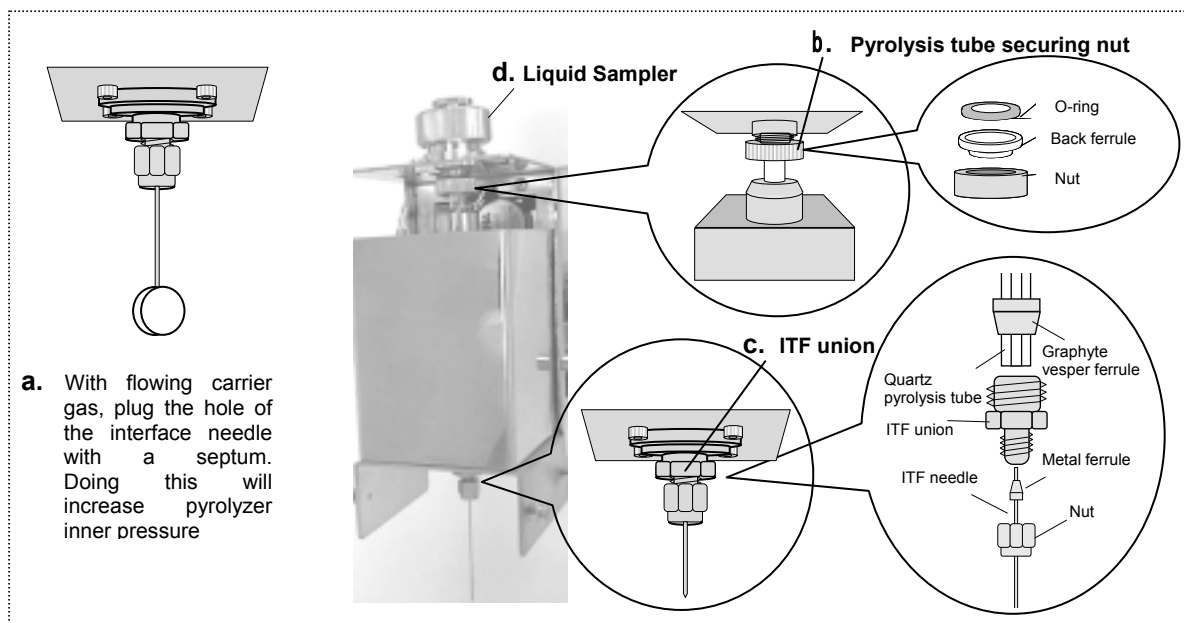


Fig. 6.1 Where to check gas leakage

4. If gas leakage can not be observed in step 3, check if the interface needle might be clogged.

Clogged interface needle may show the following symptoms;

- When removing sampler, column head pressure drops slowly.
- Takes longer time to recover the column pressure when attach the sampler.
- The screw nut of the sampler is tighter than usual and may hear the sound of blowing out the gas when loosen it.

When observed the symptoms mentioned above, measure the carrier gas flow rate at the inlet of pyrolyzer and the out let of the interface needle. If the flow rate at the needle end is less than that of inlet of the furnace, interface needle might be clogged.

6. 4 TEMPERATURE CONTROLLER

SYMPTOM	CAUSE	SOLUTION/CHECK POINTS
1. Connection failed Error appears	1.1 No power	Check power source. Turn power ON.
	1.2 RS-232C cable is not connected.	Check the RS-232C cable is properly connected between PC and temperature controller.
	1.3 RS-232C cable is connected to the different port.	Check to see if the RS-232C cable is connected to that of assigned by the control software. Try to assign to another port numbers.
2. Heated block(s) is(are) not heated	2.1 Heater concerned is set to OFF	Set it ON
	2.2 Circuit breaker (NFB on the back plane) activated	Press NFB button. If repeated tries do not work, repair at the factory is required.
	2.3 Defective heater(s)	Repair at the factory is required.
3. Temp. controller beeps	3.1 Sensor cable is not connected, or loose connection	Check to see if the cable from pyrolyzer is connected to POWER SENSOR
	3.2 Defective sensor	Repair of the furnace at the factory is required.
4. Cooling fan is not spinning	4.1 Fan blocked with foreign objects	Check to see if foreign object such as gas pipe blocks the fan.
	4.2 Defective fan	Replace the fan. Consult to your local our agent
	4.3 No power for fan	Check to see if the fan's power cable is properly connected. Reconnect it securely. If this does not fix the problem, Consult to your local our agent.

SYMPTOM	CAUSE	SOLUTION/CHECK POINTS
5. Abnormal temperature shown	5.1 Poor connection of sensor connector.	Check to see if the sensor cable form the Pyrolyzer is properly
	5.2 Error in calibration	Perform the temperature calibration.
	5.3 Defective sensor circuit	Short the concerned sensor signal input terminal by a short plug or other equivalent. Then look at its indicated temperature. If the temperature indicates far away from the room temperature, repair at the factory is required.
6. Bad temperature control at low temps (e.g. 100°C)	6.1 ITF TEMP too high	Set ITF TEMP to auto mode. ITF temperature will be set to low automatically as the PY temp. is set to low.
	6.2 No N ₂ (OR air) cooling gas pressure	Check N ₂ (or air) source valve and pressure. (400 ~ 600kPa is recommended for the source pressure)
7. Abnormal date and time display	7.1 Memory back-up battery is discharged below the level of keeping the data in the memory chip.	<p>Recharge the battery for at least 10 hours by keeping power on. Then set date and time.</p> <p>Check to see if the calibration factor is alive or not. If the calibration factor turned to zero input the calibration factor that is labeled on the bottom of the controller. Or perform temperature calibration along with the procedure mentioned in this manual (CHAPTER 4).</p> <p>If the data are turned to the default value again, it is necessary to replace the battery. Consult to your local our agent.</p>
8. When starts the temp program, GC status-monitoring step is skipped.	8.1 Remote signal cable is not connected to the temperature controller.	Connect the remote signal cable.

CHAPTER 7 BASIC PERFORMANCE TESTS

In a GC pyrolysis system the path from the pyrolyzer to the detector via the separation column is very important, and the pyrolyzer, interface, GC injection port, separation column, and detector each plays an important role. Therefore, the performance of the system as a whole is checked according to the instructions described below. When in operation, if problems are experienced, using UA-5 column (30m, 0.25mm id., 0.25 μ m, 5%phenylsilicon), the basic performance of the system can be rechecked and the problems can be identified in a shortest possible time.

7.1 TESTING BASIC PERFORMANCE AS SINGLE-SHOT PYROLYZER

7.1.1 Testing GC Performance (with pyrolyzer disconnected)

Following the instruction of the GC manual install the supplied UA-5 column into GC. Inject 1 μ l of Test mixture sample into GC using a normal GC micro syringe. Fig. 7.1 shows an example of chromatogram.

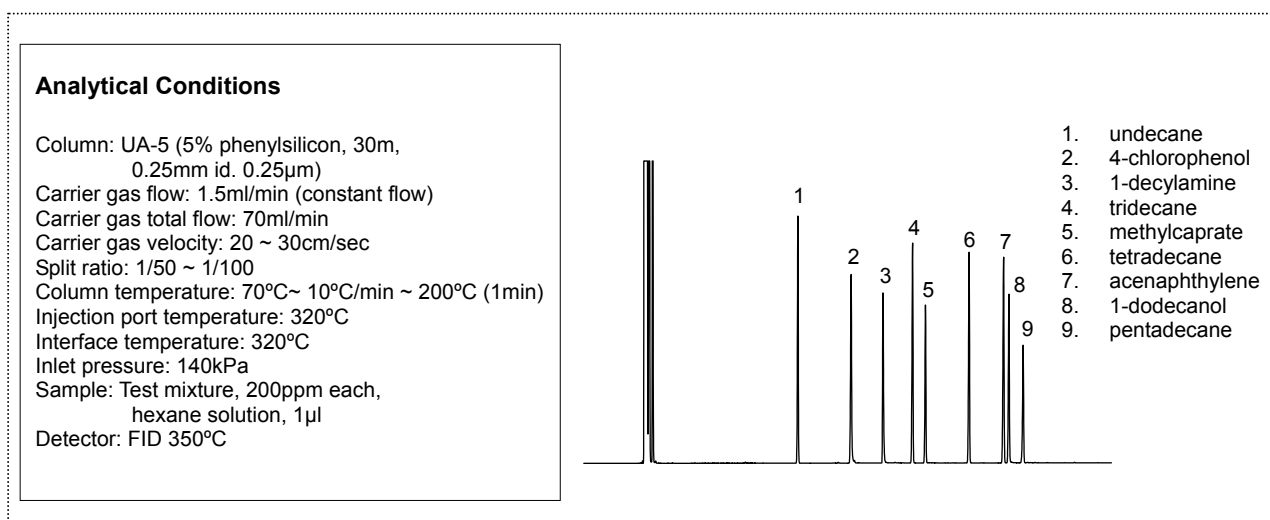


Fig. 7.1 GC System Performance Test

7.1.2 Testing Inertness of Flow Path and Resolution with Pyrolyzer Connected

(Equivalent to Data 1 in Inspection Report)

Attach the pyrolyzer onto GC (see Chapter 3 for instruction). Then install the liquid sampler onto the pyrolyzer, and inject 1 μ l of the sample (Test Mix-1) used in 7.1.1. Except for the solvent peak which is a little broader, all the other peaks should be the same as in Fig. 7.1.

If peak tailings are observed for polar compounds (especially peak No. 2, 3, and 8), pyrolysis tube or interface needle may be contaminated, and need to be cleaned or replaced with new one. Refer to CHAPTER 5 for cleaning procedure.

7.1.3 Testing Basic Performance with Pyrolyzer Connected

(Reproducibly of pyrograms)

This is the final performance test under the conditions of pyrolysis. There are two methods available: 1) Using standard sample that is used for checking columns; 2) Using a polymer.

1) Using standard sample that is used for checking columns

Test the performance according to the instruction described in 7.1.2.

2) Using polystyrene for system checking (Equivalent to Data 2 in Inspection Report)

The basic performance of pyrolyzer can be tested by a pyrogram of polystyrene (Fig. 7.2). Pyrolyzates from this polymer includes monomer, dimer, and trimer, and the ratio of these has been reported to be ca. 100/8/13 at pyrolysis temperature of 550°C. Frontier Laboratories guarantees that using FID as the detector, the relative area of the trimer vs. monomer be $14 \pm 1.5\%$, and its fluctuation be within 2% (Eco-cup SF 50 μ l).

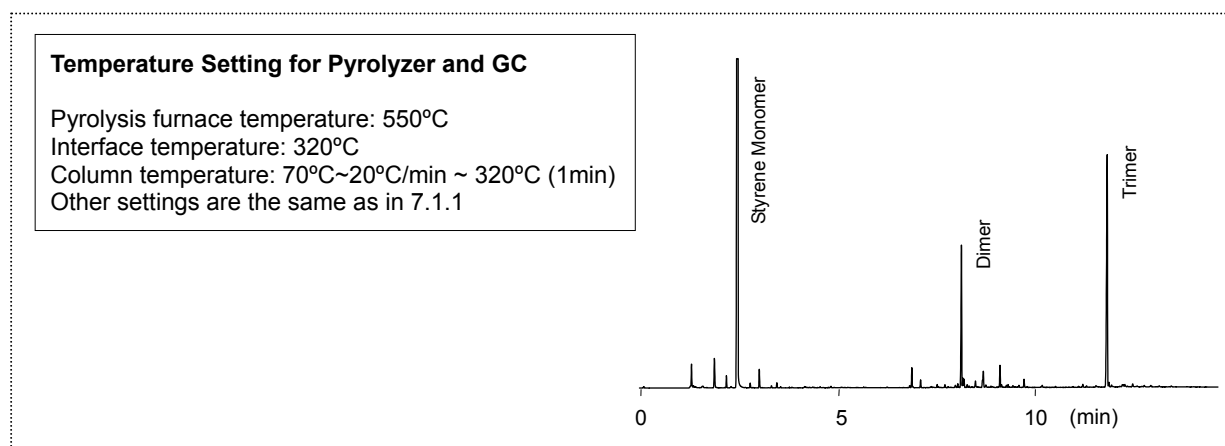


Fig. 7.2 Pyrogram of Polystyrene

7.2 EXAMPLES OF EVOLVED GAS ANALYSIS AND DOUBLE-SHOT TECHNIQUE

By continuously heating up a sample, following volatile components contained in the sample, evolved gases are generated from a polymer material as result of degradation of the polymer. The thermal properties of a sample can be determined by monitoring the evolved gases directly by a detector (FID or MS). From the evolved gas curve obtained, thermal desorption temperature for a volatile component as well as pyrolysis temperature of the polymer can be obtained.

Supplied polystyrene solution for double-shot is used as a sample for the test. This sample solution is composed of polystyrene (6 μ g/ μ l) in dichloromethane and ca. 5% (wt/wt against polystyrene) each of methylpalmitate and methylstearate. Described below is an analysis example with FID detector used as a detector. Based on the results obtained here, thermal desorption and pyrolysis temperatures are determined for the double-shot technique.

7.2.1 Evolved Gas Analysis Tests (See Fig.7.4.1, Operating Procedure P.4-13, 4-14)

STEP1 Attaching EGA tube (0.15mm id, 2.5m, UADTM-2.5N) for evolved gas analysis

In place of separation column, install supplied EGA tube for evolved gas analysis into GC.

STEP2 Setting up pyrolyzer and GC (see Fig.7.4.2)

- 1) Set ITF TEMP to AUTO mode and set the temperature to 320 for the upper limit column
- 2) Select EGA TEMP PRG and enter pyrolysis furnace temperature program described in Fig. 7.4.2.

STEP3 Loading sample

- 1) Take 5 μ l of sample in Disposable Eco-cup SF and allow the sample to dry.
- 2) Attach a double-shot stick (Eco-stick DF) to the cup and set it to the sampler as shown in Fig. 7.3A. With the upper slider pulled up (Fig. 7.3B) place the sampler onto the pyrolyzer. Then open the purge knob, wait 3 minutes, and then close it.

STEP4 When temperatures are stable, start the EGA temperature program

When READY FOR INJECTION is displayed, push down the sampler to load the sample into the pyrolysis furnace (Fig. 7.3C). Then start EGA temperature program.

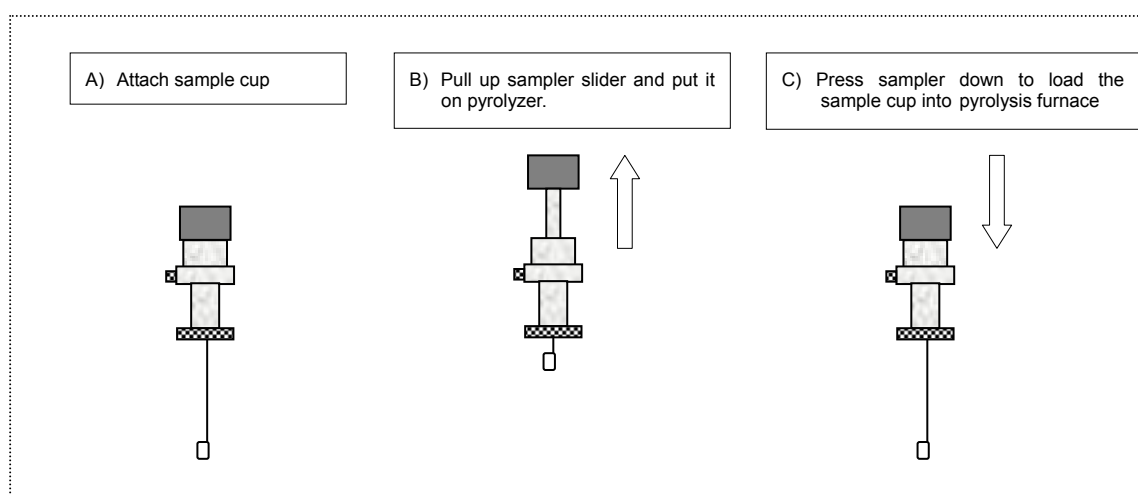


Fig. 7.3 Sample Loading Procedure in Double-shot Technique

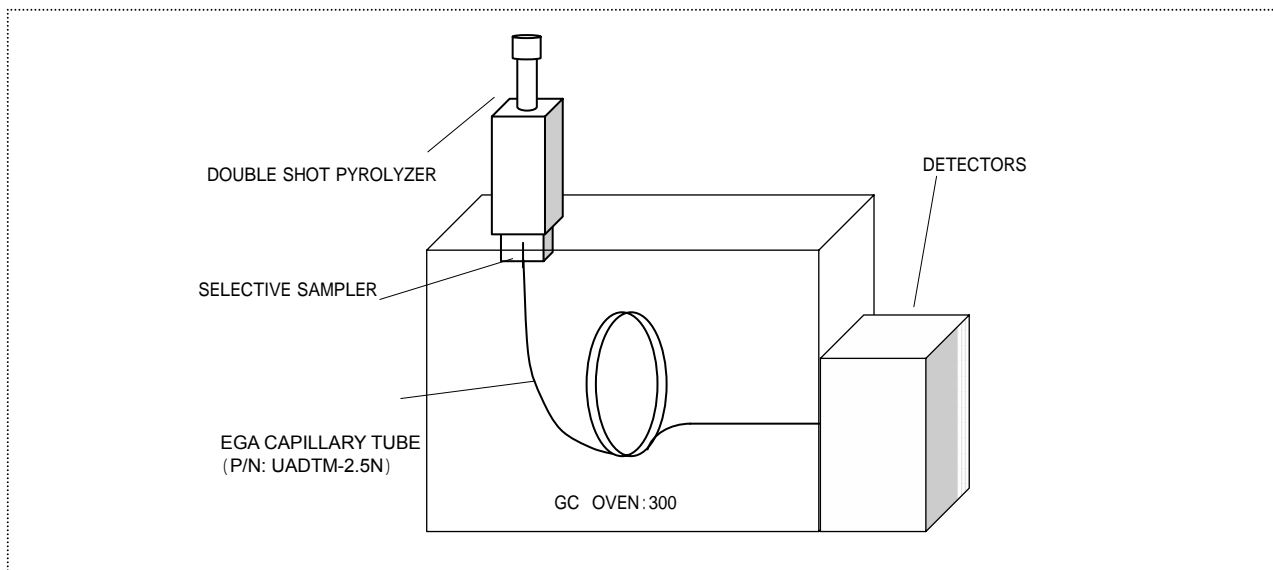


Fig. 7.4.1 Evolved Gas Analysis Construction

The result should look like Fig. 7.4.2 From this evolved gas curve the following can be drawn.

- 1) Volatile components are observed from 50°C to 200°C.
- 2) Pyrolysis of polymer starts around 380°C and ends around 500°C

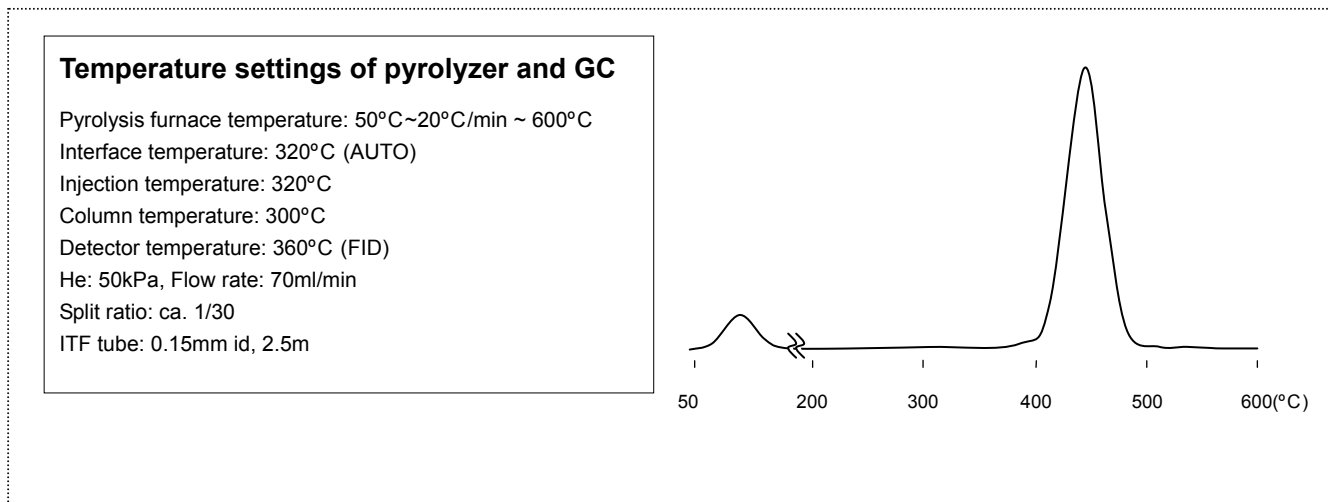


Fig. 7.4.2 Evolved Gas Curve

Here, volatile components are observed from 50°C ~ 200°C in thermal desorption technique, hence, preferred pyrolysis temperature for the polymer should be 550°C. (Our experiences show that the pyrolysis temperature that give the best reproducibility is the temperature that completes pyrolysis plus 50°C.)

7.2.2 Analysis with Double-Shot Technique

From the EGA curve shown in Fig. 7.4.2, the parameters for thermal desorption and free-fall flush pyrolysis are obtained. The results are shown in Fig. 7.5 A-1 and A-2.

Fig. 7.5B shows the result obtained by single-shot technique using the same sample. (It is apparent this pyrogram contains both A-1 and A-2.)

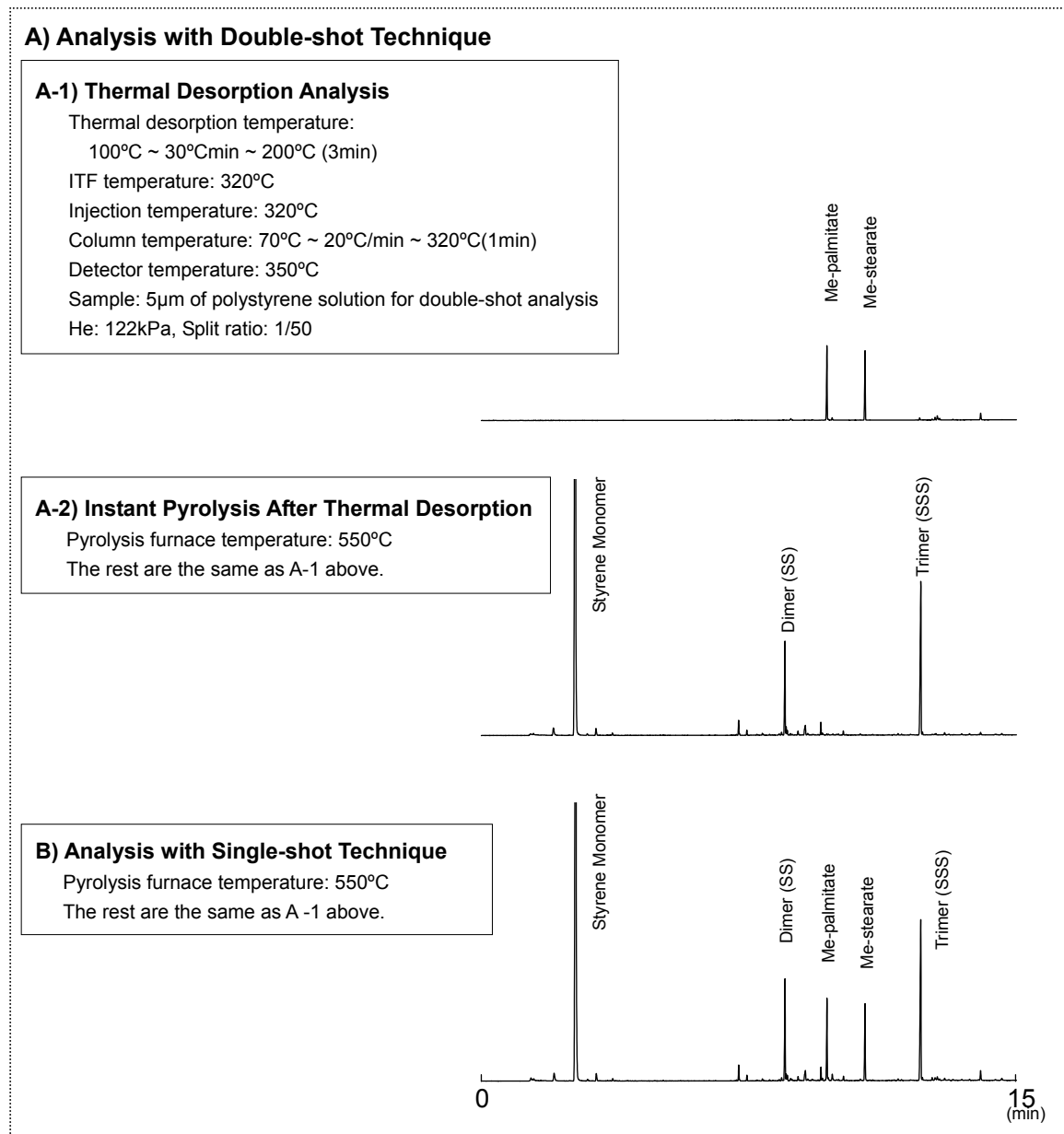


Fig. 7.5 Comparison of Double-shot and Single-shot Analyses



DOUBLE-SHOT PYROLYZER INSPECTION REPORT

Date: _____

Inspected by: _____ Approved by: _____

The results of performance inspection of the Double-Shot pyrolyzer is reported here.
 Because data were generated with a GC at Frontier Laboratories Ltd. with the pyrolyzer that you received, the reproducibility of chromatogram is not warranted.

ANALYTICAL CONDITIONS:

Pyrolyzer : Double Shot Pyrolyzer (PY-2020iD) Serial No.: _____

Integrator: : Chrom Perfect

Column : UA-5 (5% phenylsilicon), 30m(0.25mm id), 0.25 μ mCarrier Gas : He (Typical pressure FID:122kPa, MS:60kPa),
Column Flow: 1.5ml/min(constant flow), Total Flow: 70ml/min

Detector : FID

PY-GC Interface Temp: 320°C Injection Temp: 320°C FID Temp: 350°C

For other conditions, see Chapter 7. TESTING BASIC PERFORMANCE of the Operation Manual.

		Deactivation Test GC + PY	Pyrolysis Reproducibility Test GC + PY
Factory Verification	Sample	Test Mix-1	Peak areas of monomer (S) and trimer (SSS) of polystyrene are used.
	Inject. volume	1 μ l	5 μ l (drying required)
	Column temp.	70°C~10°C/min~200°C(1 min hold)	70°C~20°C/min~320°C (1 min hold)
	PY temp.	400°C	550°C (actual: _____ °C) [INPUT PY: _____ °C]
	Data attached	DATA 1	DATA 2
	Judging criteria and actual results	No abnormality of peak shapes	1. SSS/S = _____ % (n=3) (Criterion : 14 \pm 1.5 %) 2. Reproducibility _____ % (n=3, Eco-cup S used) (Criterion : < 2%, Eco-cup SF)
	Peak shape	<input type="checkbox"/> Pass	<input type="checkbox"/> Pass
Customer Verification	Inspection at installation	----- -----	Peak shape (n=1) <input type="checkbox"/> Pass

Dealer's name : _____

Installer's name : _____ Date: _____

Customer signature : _____ Date: _____

LIST OF REPLACEMENT PARTS FOR PY-2020iD & PY-2020iS

Part Name	Part No.	Qty.	Description
1 Sample cups and related parts (all cups are deactivated by proprietary technology)			
# Disposable Eco-cup SF	PY1-EC50F	1bag	50 µl, stainless steel, containing 100 pcs
# Disposable Eco-cup LF	PY1-EC80F	1bag	80 µl, stainless steel, containing 100 pcs
Flow Through Eco-cup SHF	PY1-EC50HF	1bag	50 µl, flow through type, disposable, containing 20 pcs
Flow Through Eco-cup LHF	PY1-EC80HF	1bag	80 µl, flow through type, disposable, containing 20 pcs
# Eco-stick SF	PY1-ES10F	1bag	30mm, for single-shot py, contains 50 pcs.
# Eco-stick DF	PY1-ES20F	1bag	80mm, for double-shot py, contains 50 pcs.
Eco-pick-up	PY1-EP50	1pcs	Eco-cup retriever
# Eco-stand	PY1-EH10	1pcs	Eco-cup stand (for S and L), for sampling.
2 Pyrolysis furnace parts			
# Quartz pyrolysis tube	PY1-2018	1pcs	6mm o.d. (4.7mm i.d.)
Needle guide	PY1-2014	1pcs	For Liquid sampler
Septum	PY1-2025	1bag	For Liquid sampler, pale yellow, contains 10pcs
3 Connecting parts			
Interface union assembly	PY1-3510	1set	A set of union, interface needles (2pcs), & nut
Interface union	PY1-3511	1pcs	For graphite Vespel ferrule
Heat-sink adapter set	PY1-3570	1set	screw 2ea, 1.5mm hexagonal wrench 1ea
Needle Nut	PY1-3512	1bag	Needle nut for interface needle, 3pcs
# Needle set	PY1-1174	1set	deactivated tube, 3pcs
# Graphite Vespel ferrule	PY1-7911	1bag	6mm i.d., for PY1-3511, contains 5pcs (Air Leak Free)
Graphite ferrule	PY1-7921	1bag	6mm i.d., for PY1-3511, 100% graphite, 5pcs
Inlet/outlet septum	PY1-2028	1bag	For connecting tubes, pale yellow, with hole, 10pcs
4 GC related parts			
Septum (11mm in diameter)	PY1-7301	1bag	For Agilent 6890 (coated releasing reagent & conditioned, 5pcs)
Septum	PY1-7302	1bag	For Shimadzu GCs (coated releasing reagent & conditioned, 5pcs)
Glass insert (with wool)	PY1-3337	1pcs	For Agilent 6890, deactivated, with deactivated wool
Absorption trap set 5890SV	PY1-2212	1set	For HP5890 split vent
Absorption trap set 17SV	PY1-2227	1set	For Shimadzu GCs
Absorption trap tube	PY1-2216	1set	Trap tubes only, packed with activated charcoal, 3pcs, for PY1-2212
GC/MS connector kit	PY1-2210	1set	Ultra ALLOY cap. column connection kit for sector MS
GC/MS adapter	MS604230	1set	HP5989A,HP597x (for 0.2 to 0.32mm id columns)
GC/MS adapter (HP5989B)	MS604240	1set	HP5989B (for 0.2 to 0.32mm i.d. columns)
Vent-free GC/MS adaptor	MS402140	1set	Vent free tube 1ea, vent free, Vent free holder 1ea, Holder mounting bracket 1ea, Column nut 1ea, GC/MS adaptor 1set, 1.5mm hexagonal wrench 1ea
EGA tube	UADTM-2.5N	1pcs	0.15mm id, 2.5m, with cage, 1 pcs

Part Name	Part No.	Qty.	Description
5 Replacement parts set (A supply for about a year)			
Replacement parts set (Double)	PY-K202D	1 set	Disposable Eco-cup SF(2), Disposable Eco-cup LF(1), Eco-stick SF(1,2*), Eco-stick DF(1, 0*),Eco-stand (2),Quartz pyrolysis tube(3),Needle set (3), graphite Vespel ferrule (1set), (* for PY-K2020S)
Replacement parts set (Single)	PY-K202S	1 set	
6 Ultra ALLOY Metal Capillary Column (Available: other liquid phases, length and film thickness, See column brochure)			
5% diphenyl	UA5-30M-0.25F	1pcs	General purpose, 30m (0.25mm id) 0.25 µm film (max temp.: 380°C)
5% diphenyl	UA5-30M-0.5F	1pcs	High volatiles, 30m (0.25mm id) 0.5 µm film (max temp.: 360°C)
5% diphenyl	UA5-30M-0.1F	1pcs	Low volatiles, 30m (0.25mm id) 0.1 µm film (max temp.: 380°C)
UA-CW	UA-CW-30M-0.25F	1pcs	Polar pyrolyzates, 30m (0.25mm id) 0.25 µm film (max temp.: 260°C)
7 Magic Chemisorber			
Magic Chemisorber S	PY1-MC01S	1set	Magic Chemisorber S, 100% dimethylpolysiloxane, 6mm, 10pcs
Magic Chemisorber S Kit	PY1-MC01S-K	1set	Magic Chemisorber S 3pcs, Eco stick DF 5pcs, MC holder S (Flow Through Eco-cup LHF) 5pcs
Magic Chemisorber L500	PY1-MC01L	1set	100% dimethyl polysiloxane, 500 µm 30mm, 5pcs
Magic Chemisorber L500 Kit	PY1-MC01L-K	1set	Magic Chemisorber L500 3pcs, Eco stick DF 5pcs, MC holder L 2pcs
Magic Chemisorber L100	PY1-MC02L	1set	100% dimethylpolysiloxane, 100µm,30mm, 5pcs
Magic Chemisorber L100 Kit	PY1-MC02L-K	1set	Magic Chemisorber L100 3pcs, Eco stick DF 5pcs, MC holder L 2pcs
MC holder L	PY1-MCP01	1set	Protective tube for Magic Chemisorber L500, 3pcs
8 Harris Micro-Puncher			
Harris Micro-Puncher 050	HMP-0.50D	1set	0.50mm id, 10pcs, Blue
Harris Micro-Puncher 075	HMP-0.75D	1set	0.75mm id, 10pcs, Green
Harris Micro-Puncher 125	HMP-1.25D	1set	1.25mm id, 10pcs, Red
Harris Micro-Puncher 200	HMP-2.00D	1set	2.00mm id, 10pcs, Black
Harris Cutting Mat	HCM-6575	1pkg	65 × 75mm, 5 sheets/pkg Green
Harris • 01Kit	HMP-01kit	1set	Each 3pcs of HMP-0.50D, 0.75D, 1.25D, 2.00D and 2 sheets of Harris Cutting Mats
9 Others			
Polymer Prepper	PY1-7510	1set	Polymer Prepper with two different grades of files, for Pulverizing samples, 3pcs, with 1roll of cleaning tape(2m)
Cleaning Tape	PY1-7515	1set	Cleaning Tape for cleaning deposited powder sample on the files of Polymer Prepper for repeated use, 3rolls(2m each)

<> Checked (#) items are replacement parts required in daily maintenance work.

<> Price and specifications are subjected to change.

