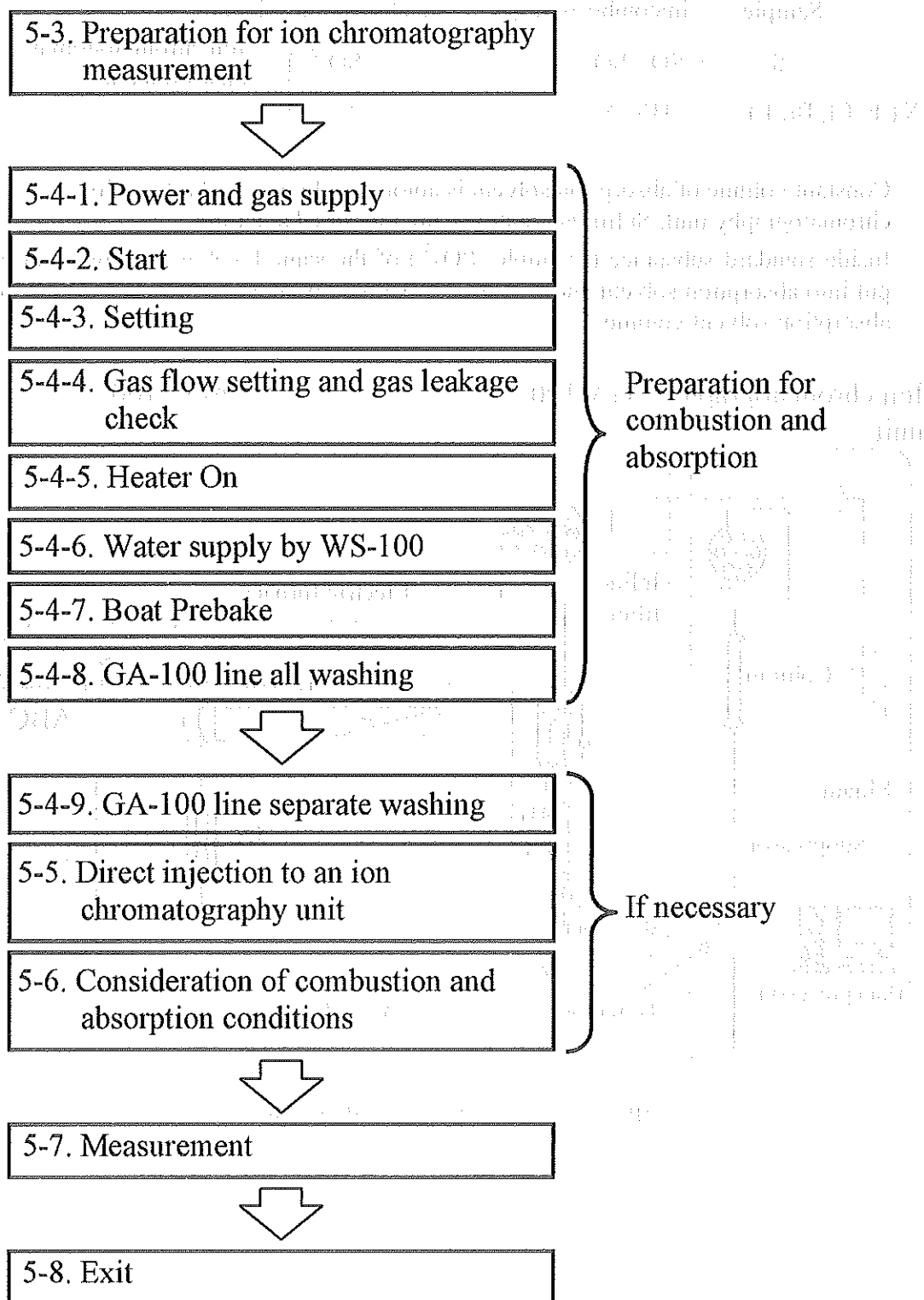


Section 5: Measurement

This section describes the details of combustion and absorption and ion chromatography. For ion chromatography contents, refer to the unit instruction manual.

5-1. Operation Flow

Basic flow of combustion and absorption and ion chromatography is as follows.

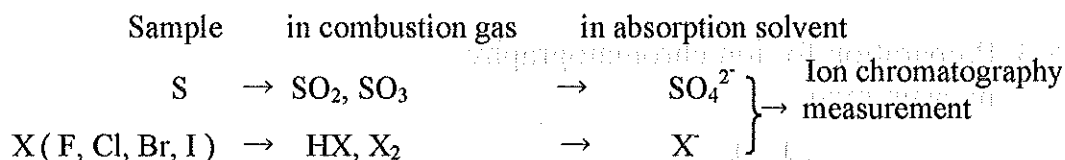


5-2. Combustion and Absorption Flow

5-2-1. Combustion and absorption principle

Sample is injected into a pyrolysis tube where Ar gas flows and temperature is high (800~1100°C).

After sulfur and halogen compound in sample are pyrolyzed, they are combusted and oxidized by O₂ gas as follows and collected to absorption solvent.



Constant volume of absorption solvent is automatically injected into an ion chromatography unit. Sulfur and halogen are measured at a time.

Inside standard substance (Example: PO₄³⁻) of the same level as measurement element is put into absorption solvent and used to correct measurement values by the gain and loss of absorption solvent volume.

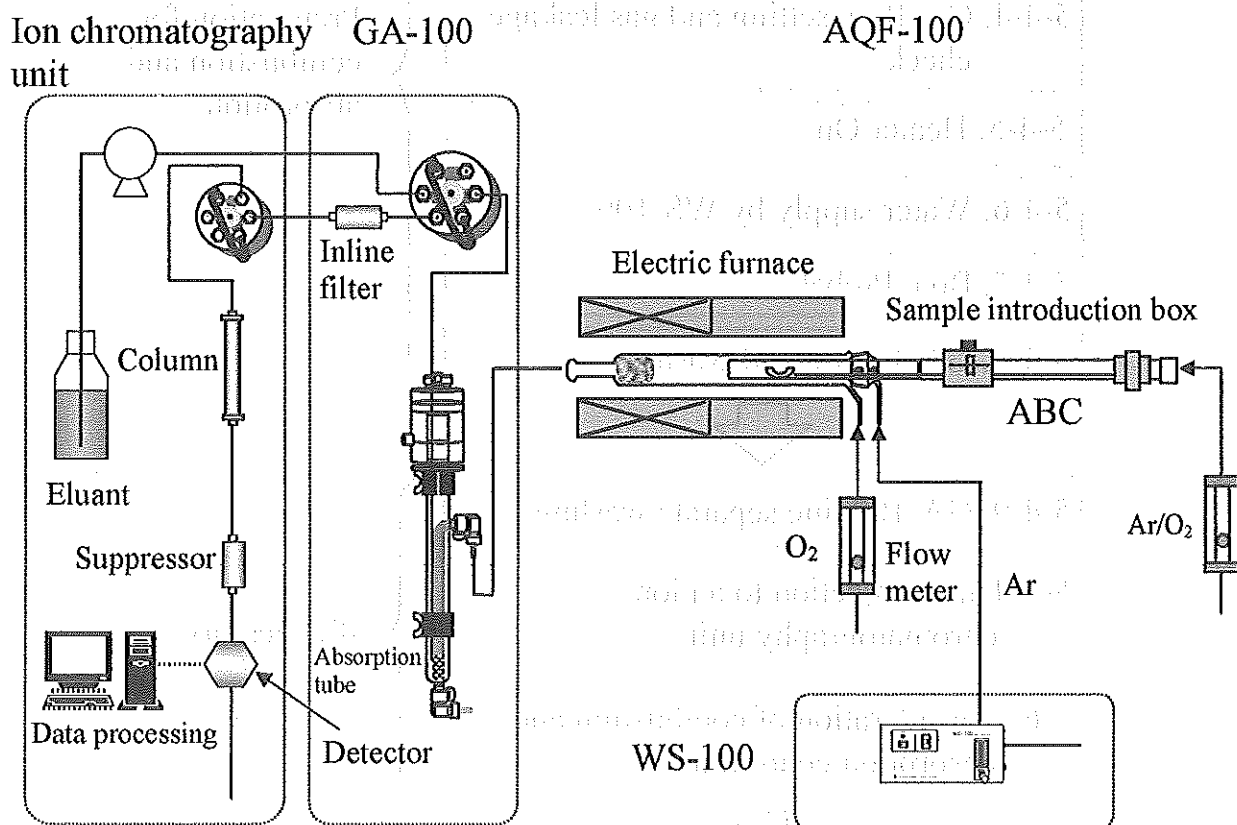


Illustration 5-1. System schematic

5-2-2. Samples combustion and absorption examples

To gain optimum conditions in combustion and absorption process, understand the following points.

1. Sample quality
2. Sample volume
3. Pyrolysis tube temperature
4. Combustion position and time
5. Oxygen/Argon gas flow
6. Absorption solvent quality
7. Absorption solvent concentration and volume

Table 5-1 shows ABC combustion condition data. Consider combustion and absorption conditions.

	1st		2nd		3rd		End	Cool	Boat	Ar	O ₂
	Pos mm	Time sec	Pos mm	Time sec	Pos mm	Time sec	Time sec		Speed mm/s	Time sec	
Coal	0	0	0	0	0	0	360	30	10	0	300
Organic/Et 30 μ l	95	90	120	30	150	30	120	30	10	0	300
Toluene 30 μ l	100	120	110	30	180	0	120	30	10	0	300
Gasoline 50 μ l	95	120	110	30	180	0	60	30	10	0	300
Heavy oil 30 μ l	110	30	130	120	150	60	60	30	10	0	300
Gelatin (solid 10mg)	130	180	160	90	180	30	180	30	10	0	300
Kerosene 50 μ l	100	30	120	120	180	0	60	30	10	0	300
Waste oil 30 μ l	100	120	150	30	180	30	60	30	10	0	300

Table 5-1. Sample combustion and absorption condition examples

Common conditions

Heater temperature : Inlet Temp : 800°C Outlet Temp : 1000°C

Argon /Oxygen flow : Ar/O₂ : 200ml/min O₂ : 400ml/min

WS-100 flow rate : Ar: 150ml/min

5-2-3. Analysis schedule

For efficient ion chromatography measurement, combust and absorb the next sample during measurement. After absorption, absorption solvent sampling starts without waiting time.

The following chart shows two types of time of ion chromatography measurement.

The first is the case where ion chromatography measurement is longer than total time of absorption solvent setting, combustion and absorption, and absorption time.

The second is the case where ion chromatography measurement is shorter than total time of absorption solvent setting, combustion and absorption, and absorption time.

① Absorption solvent set : Time for injecting absorption solvent into an absorption tube
(Refer to 4-5-2-2. "Absorption solvent set" flow.)

② Combustion and absorption : Time required for combustion and absorption

③ ABC H.C.T. : ABC HOME COOL TIME

(Boat cooling time at ABC home position)

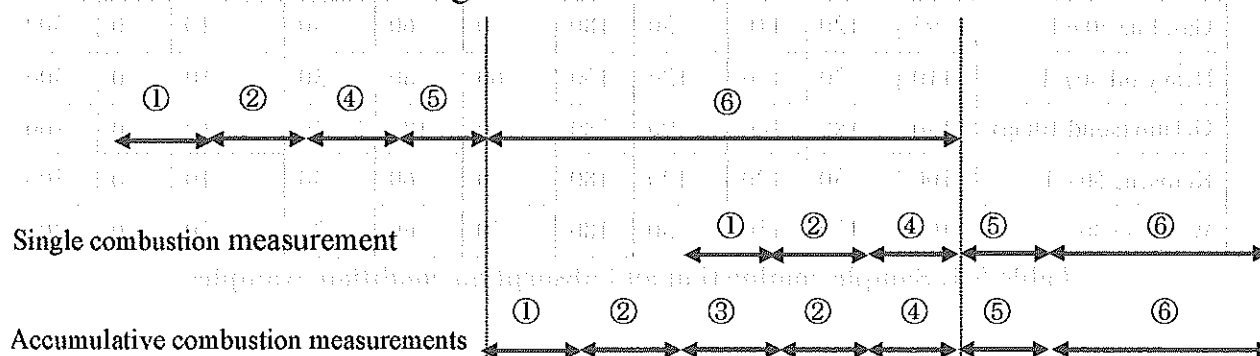
④ Absorption time : Combustion gas absorption time after a sample boat returns to ABC home position

⑤ Absorption solvent sampling : Absorption solvent sampling time

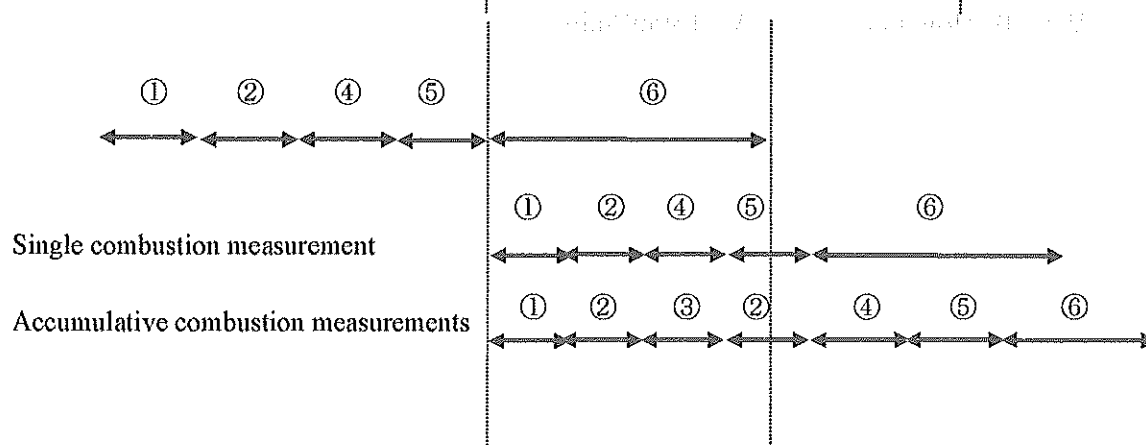
(Refer to 4-5-2-3. "Absorption solvent sampling" flow.)

⑥ Ion chromatography measurement : Time from ion chromatography measurement to measurement and data processing ends and the reception

Measurement time is long.



Measurement time is short



5-3. Preparation for ion chromatography measurement

- (1) Prepare for ion chromatography measurement by the instruction manual.
- (2) Check that a detector base line is stable.
- (3) The start signal from GA-100 is waited.

5-4. Preparation for combustion and absorption

5-4-1. Power and gas supply

- (1) Open main valves of O₂ gas and Ar gas cylinders.
- (2) Adjust the second pressures of a cylinder and a STOP valve to 0.4 ± 0.1 MPa with a reducing valve.
- (3) Supply power to the outlet on the table.

5-4-2. Start

- (1) Turn on the following power switches.
 - ① AQF-100 power switch and heater switch (Front panel)
The power switch of an automatic boat controller (ABC) is turned on automatically.
 - ② Cooling the unit power switch of ABC rear
 - ③ Cooler switch of ABC front
 - ④ GA-100 power switch (Front panel)
- (2) When using ASC-150L, turn on the power switch.
- (3) When using ASC-120S, turn on the power switch.
- (4) Turn on the power switches of a personal computer, a monitor, and a printer.
- (5) Start AQF-100 system program. (Refer to 4-1-1. Start.)

POINT

When measuring fluorine, use Water Supplier Model WS-100. Absorption rate is better with WS-100.

5-4-3. Setting

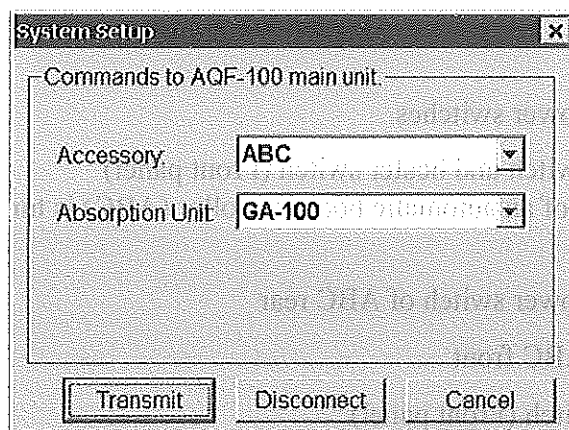
5-4-3-1. System Setup

Select an accessory and GA-100 in “System Setup” dialog box and start the communication to the main unit.

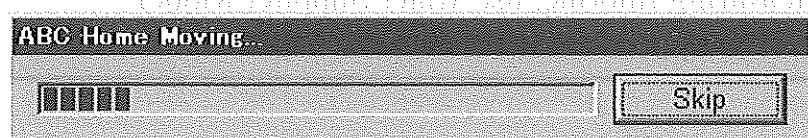
POINT

Before measurement, run “System Setup” if necessary.
After “System Setup”, AQF communication to the system program starts. “Analysis Parameters”, “Heater”, and “Boat Prebake” are effective and their operations can be run.

- (1) Press <F5> key or click “System” and “System Setup”.
“System Setup” dialog box is displayed.




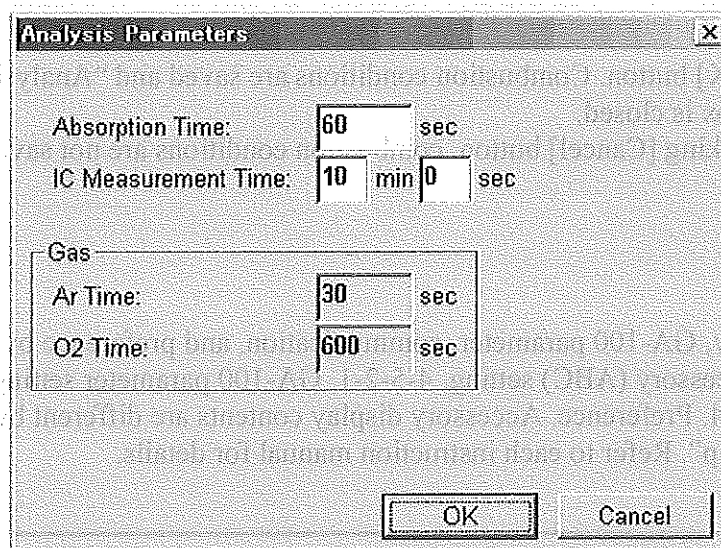
- (2) Click ▼ of “Accessory” to select it. For standard composition, select “ABC”.
- (3) Select “Absorption Unit”. For standard composition, select “GA-100”.
- (4) Click [Transmit] button.



“ABC Home Moving” is displayed. It indicates the time of initializing ABC.
“System Setup” contents are saved and “System Setup” dialog box is closed.
AQF communication to the system program starts.

5-4-3-2. Analysis Parameters

- (1) Click  or click “System” and “Analysis Parameters”.
“Analysis Parameters” dialog box is displayed.
- (2) Set each item. (Refer to Table 5-2. Analysis Parameters setting item.)



Analysis Parameters

Absorption Time: 60 sec

IC Measurement Time: 10 min 0 sec

Gas

Ar Time: 30 sec

O2 Time: 600 sec

OK Cancel

Item	Setting contents
Absorption Time	Time of combustion gas absorption after ABC boat return to the home position (0~9999 sec)
IC Measurement Time	Measure previously time from ion chromatography measurement start time to data processing and input it. (99min, 0~59sec)
Ar Time	Time for flowing argon gas into Ar/ O ₂ line (an inner pyrolysis tube) (0~999 sec) * Set it by ABC program.
O2 Time	Time for flowing oxygen gas into Ar/ O ₂ line (an inner pyrolysis tube) (0~999 sec) * Set it by ABC program.

Table 5-2. Analysis Parameters setting item

POINT

If ion chromatography measurement time is too short, next measurement can't be run.

If setting time is too long, ion chromatography unit waiting is long and total time is long.

(3) Click [OK] button. Combustion conditions are saved and "Analysis Parameters" dialog box is closed.

* By clicking [Cancel] button, combustion conditions are not saved.

5-4-3-3. Settings

Set accessory, GA-100 parameter, communication, and preference by referring to 4-5-1. Accessory (ABC) setting, 4-5-2-1. GA-100 parameter setting, 4-5-3. Computer I/F, and 4-5-4. Preference. Accessory display contents are different by the selection at "System Setup". Refer to each instruction manual for details.

POINT

Even when each setting is not changed, check the contents.

5-4-4. Gas flow setting and gas leakage check

Change gas at the personal computer side, adjust AQF-100 gas flow, and check gas leakage.

CAUTION

After turning on the power switch, more than 30 minutes is required to stabilize AQF-100 flow sensor.

The flow can be checked in AQF-100 frame of the main window.

After sufficient time, check gas leakage.

5-4-4-1. Gas flow setting

- (1) Gas flow is displayed in AQF-100 frame of the main window.

The screenshot shows the AQF-100 main window with the following data:

AQF-100	
Inlet Temp.:	33 °C
Outlet Temp.:	34 °C
Ar/O ₂ :	200 ml/min
O ₂ :	400 ml/min

ACCESSORY	
ABC:	Home
GA-100:	
IC:	sec

Start

System Setup is completed.

- (2) Turn the knob of AQF-100 front and adjust gas flow. Adjust Ar flow with Ar. Adjust O₂ flow with O₂. Total argon flow of WS-100 and the main window AQF-100 frame flows to a pyrolysis tube.

	ml/min
Ar/ O ₂	200
O ₂	400

Gas flow setting example

5-4-4-2. Gas leakage check

CAUTION

Check gas leakage when the heater switch is off. By checking gas leakage when the switch is on, you can get burned.

When setting a gas flow meter for gas leakage check, a pyrolysis tube already can be hot. Take care not to get yourself.

CAUTION

Do not clog the outlet of a pyrolysis tube. The pyrolysis tube breaks and you can get hurt.

CAUTION

After turning on the power switch, more than 30 minutes is required to stabilize AQF-100 flow sensor.

The flow can be checked in AQF-100 frame of the main window.

After full time, check gas leakage.

CAUTION

If flow is under the set value, gas can leak. Stop the unit use immediately and check gas leakage.

- (1) Replace a ball joint (with branch tubes) of a pyrolysis tube outlet with one of a flow meter for gas leakage check.
- (2) Check that the flow meter value is within 750 ± 75 ml/min of O₂ scale.
(When Ar is 350 ml/min (including WS-100 flow 150 ml/min) and O₂ is 400 ml/min, total flow is 750 ml/min.)
- (3) If the value is under the set flow, refer to Illustration 5-2 and check gas leakage.
- (4) Recover gas leakage points and run (2) again.
- (5) Change the ball joint of the flow meter for gas leakage check with one with branch tubes.

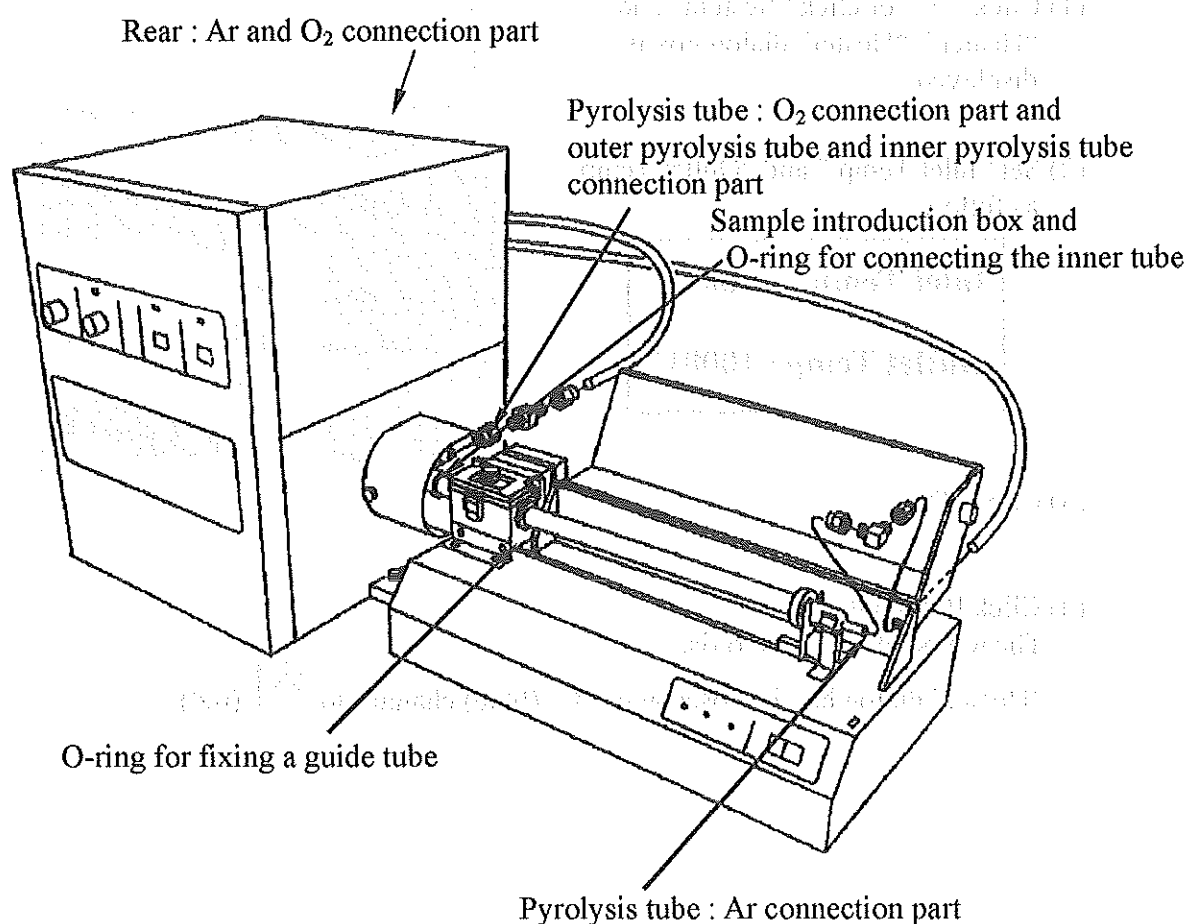



Illustration 5-2. Gas leakage check position

5-4-5. Heater On

POINT

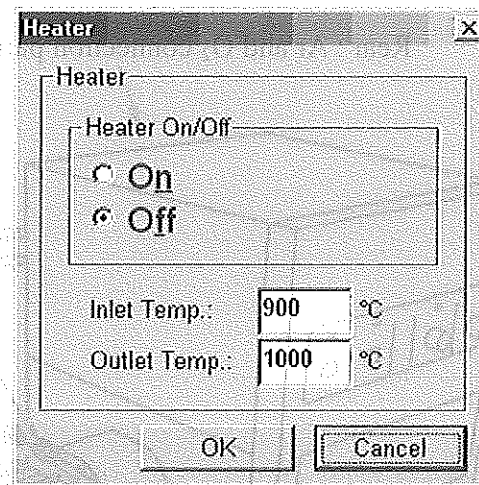
Turn on AQF-100 heater switch and click “On” and [OK] button in “Heater” dialog box. Without the setting in “Heater” dialog box, the temperature does not start to rise.

Before the temperature reaches the set temperature and is stable, more than 60 minutes is required.

- (1) Click  or click “System” and “Heater”. “Heater” dialog box is displayed.

- (2) Set “Inlet Temp.” and “Outlet Temp.” as follows.

Inlet Temp : 900°C
Outlet Temp : 1000°C



- (3) Click “On”.

- (4) Click [OK] button.

The temperature starts to rise.

“Heater” dialog box is closed and  (blue) changes to  (red).

5-4-6. Water supply by WS-100

Argon gas including ultrapure water is supplied to a pyrolysis tube with WS-100.

POINT

By supplying argon gas including ultrapure water, component recovery is up. When contamination is in WS-100 line, operate it fully with ultrapure water and set it to a pyrolysis tube.

By using WS-100, water is supplied to an absorption tube between absorption solvent set and sampling and the solvent volume increases.

Consider increasing amount and set GA-100 parameter absorption solvent volume.

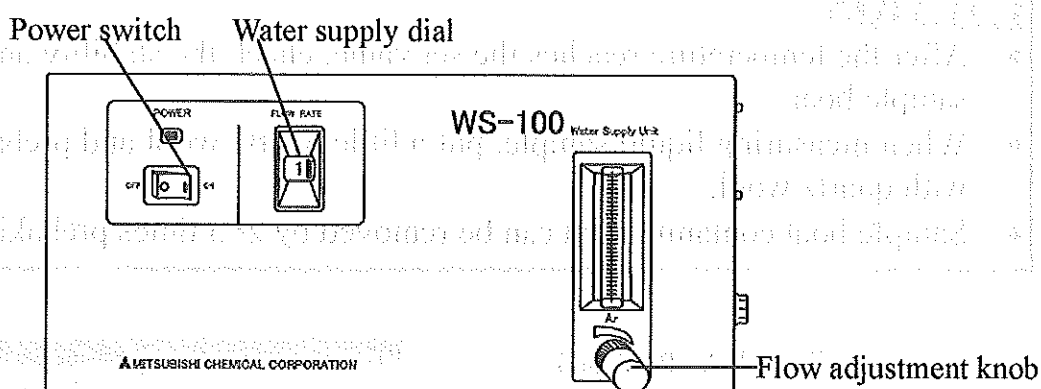


Illustration 5-3. WS-100 front panel

- (1) Turn a flow adjustment knob to set argon gas flow to 150ml.
Total argon gas flow of WS-100 and the main window AQF-100 frame goes into a pyrolysis tube.
- (2) Adjust "FLOW RATE" of a water supply dial. For high concentration analysis, set the dial to "4". For low concentration analysis, set the dial to "1".
* The indication of water supply volume is a dial gauge $\times 0.05$ (ml/min).
Water supply volume is $\pm 30\%$ of setting values.
- (3) After the temperature reaches the preset value, the dialog box is displayed.
Turn on WS-100 power switch and click [OK] button. Water supply starts.

CAUTION

When the temperature does not reach the preset value, water is not supplied even by turning on WS-100 power switch.

5-4-7. Boat Prebake

Prebake a boat to remove the contamination.

Before prebaking, remove a clamp for a ball joint and a ball joint (with branch tubes) from a pyrolysis tube outlet.

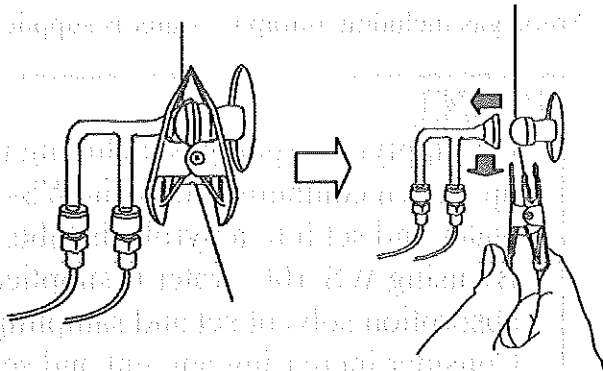

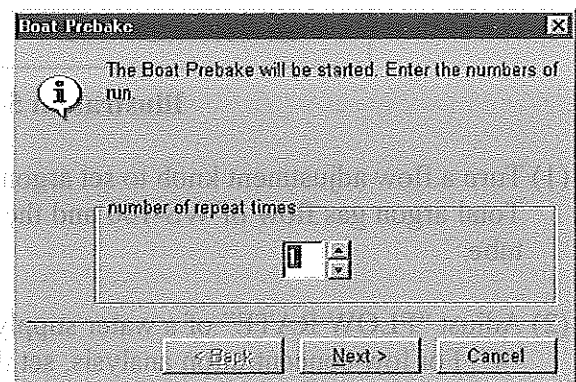


Illustration 5-4. Pyrolysis tube outlet removal

CAUTION

- After the temperature reaches the set value, check the stability and prebake a sample boat.
- When measuring liquid sample, put a little quartz wool and prebake the boat with quartz wool.
- Sample boat contamination can be removed by 2~5 times prebaking.

- (1) Click  or click "Run" and "Boat Prebake".
"Boat Prebake" dialog box is displayed.



- (2) Input "number of repeat times". (Usually 2~5 times)

Setting of a sample boat

- Open the cover of a sample introduction box.
- Put a sample boat into the sample introduction box and set it to a ladle.
For liquid samples, put 0.02~0.03g quartz wool without running over from the boat and flatten it fully.

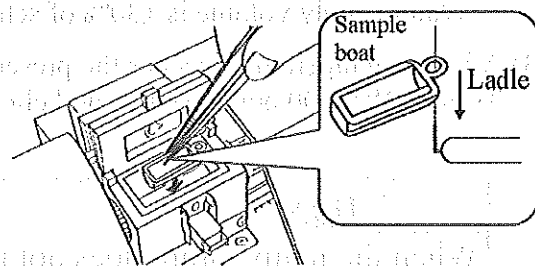


Illustration 5-5. Sample boat setting

- Close the cover of the sample introduction box and lock it.

- (3) Click [Next] button.
The following dialog box is displayed.
* By clicking [ABC Programs] button, ABC program can be checked.

(4) Click [OK] button. "Boat Prebake" dialog box is closed.

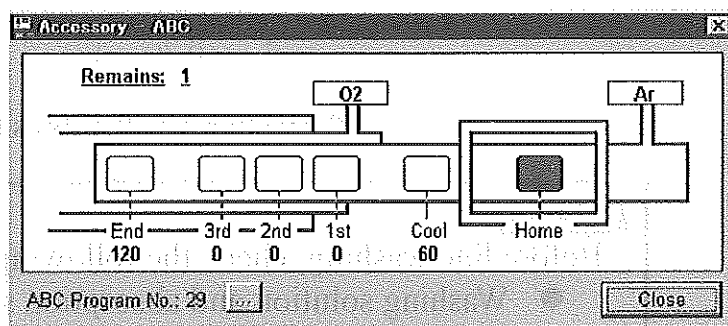
(5) When [Start] button flickers, set a sample boat to ABC sample introduction box.


POINT

When handling a sample boat, use tweezers to prevent contamination. For more than 2 sample boats prebaking, keep sample boats in a glass petri dish until measurement.

(6) Click [Start] or press <Enter> key. Boat prebaking starts.


(7) 1 prebaking requires about 4~5 minutes and prebaking is repeated automatically by the predetermined number. "Boat Prebake" is indicated in "Analysis Status".



By clicking ,

remaining times is displayed in "Remains" of the upper left.

(8) To prebake another boat, repeat (5)~(7).

(9) To end boat prebaking, click . Boat prebaking ends.

5-4-8. GA-100 line all washing

Line washing is required before measurement. Click [Wash All] button of GA-100 dialog box. Refer to 4-5-2-5, “Wash All” flow. Set a ball joint with branch tubes before “Wash All” and fix it to a pyrolysis tube with a clamp for a ball joint.

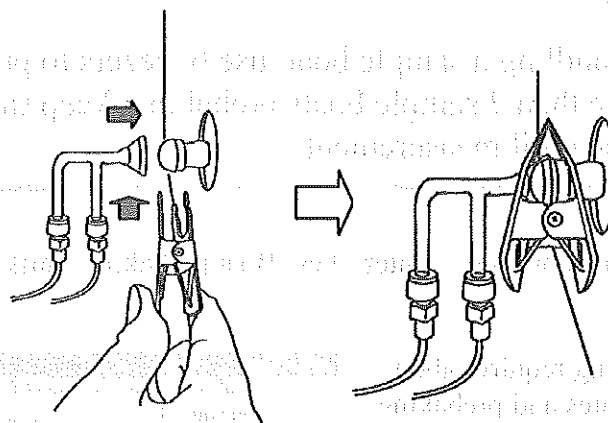


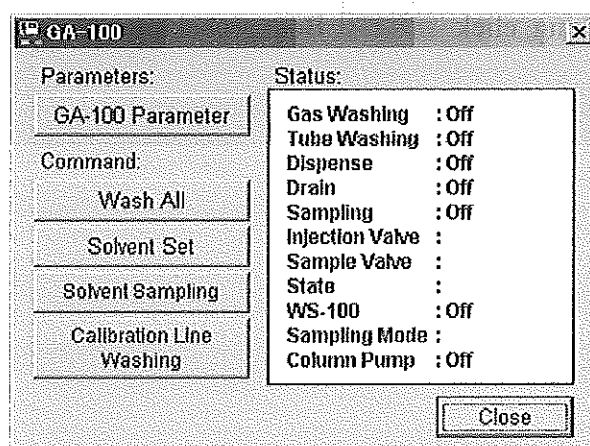
Illustration 5-6. Pyrolysis tube outlet connection

POINT

Before line washing, check the following points.

- Washing solution and absorption solvent are set and lines are connected correctly.
- A ball joint with branch tubes is connected.
- ABC sample introduction box cover is closed.
- Pour washing solution by pressing <Absorption Tube> key and check that gas flows into the solution.

(1) Click “System” and “GA-100”. “GA-100” dialog box is displayed.



(2) Click [Wash All] button. Each gas line is washed automatically.

5-4-9. GA-100 line separate washing

Line washing is required in the following cases. When washing is not required, skip it.

- ① In the first use of the unit, When the line inside is empty, Out of use for long time
- ② When absorption solvent is prepared again
- ③ When lines are contaminated

Refer to Illustration 1-8. GA-100 absorption part line, Illustration 1-9. GA-100 operation panel, Table 1-9. GA-100 operation panel names and functions.

POINT

Before line washing, check the following points.

- Washing solution and absorption solvent are set and lines are connected correctly.
- A ball joint with branch tubes is connected.
- ABC sample introduction box cover is closed.
- Pour washing solution by pressing <Absorption Tube> key and check that gas flows into the solution.

5-4-9-1. Gas line washing

- (1) Check gas flow and press <Gas Line> key for one second to pour washing solution.
- (2) Continue to press <Drain> key to drain washing solution.
- (3) Repeat (1) and (2) several times and wash gas lines.

5-4-9-2. Absorption tube washing

- (1) Continue to press <Absorption Tube> key and fill washing solution into an absorption tube by overflow. Pour it again for about 5 seconds.
- (2) Continue to press <Drain> key to drain washing solution.
- (3) Repeat (1) and (2) several times. Wash the absorption tube.

5-4-9-3. Absorption solvent tube washing

- (1) Press <Dispense> key and inject the specified volume of absorption solvent into an absorption tube.
- (2) Continue to press <Drain> key to drain absorption solvent.
- (3) Repeat (1) and (2) 2~3 times and wash the absorption solvent tube.

5-4-9-4. Standard solution tube washing

To wash a standard solution tube, dip the tube in ultrapure water and click "System" and "GA-100" of AQF system program menu and [Calibration Line Washing] button.
Refer to 4-5-2-5. Standard solution sampling line washing flow.

5-5. Direct injection to an ion chromatography unit

Without combustion and absorption, standard solution can be injected directly from GA-100 to an ion chromatography unit. By comparing the case of direct injection of standard solution with the case of injection from absorption solvent by combustion and absorption, the recovery can be obtained. When direct injection is not required, skip this item. Refer to Illustration 1-9. GA-100 operation panel and Table 1-9. GA-100 operation panel names and functions.

- (1) Put a standard solution injection tube into standard solution.
Press <Calibration> key.
- (2) Left lamp of <Calibration> key is on and standard solution is injected while the lamp is on.

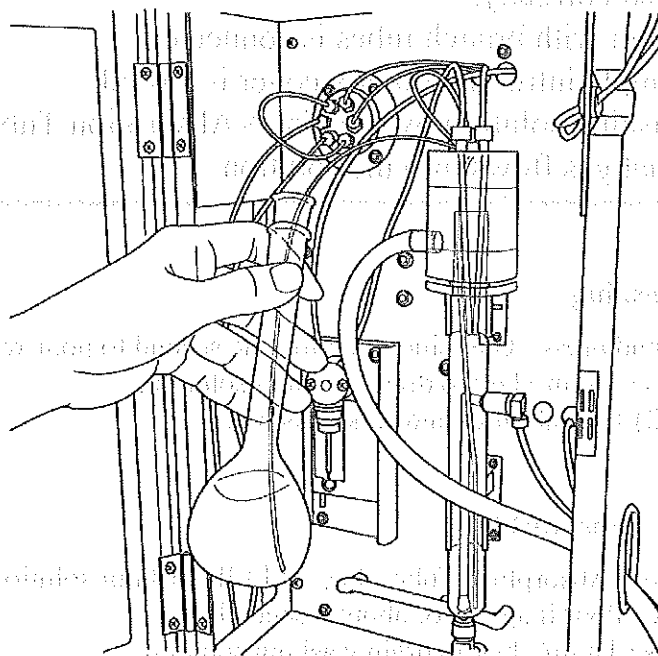


Illustration 5-7. Direct injection to an ion chromatography unit

POINT

When the unit is not used, immerse the tip of a standard solution injection tube in ultrapure water to prevent the tube contamination.

5-6. Consideration of combustion and absorption conditions

When combustion and absorption conditions are known, skip this section.

POINT

Remove the clamp for a ball joint of a pyrolysis tube outlet and remove the pyrolysis tube outlet and the ball joint (with branch tubes).

Incomplete combustion when a ball joint is connected contaminates the pyrolysis tube and gas lines.

5-6-1. Removal of a pyrolysis tube outlet

Remove a clamp for a ball joint and remove a ball joint (with branch tubes) from a pyrolysis tube outlet. Illustration 5-8 shows pyrolysis tube outlet removal.

CAUTION

Do not touch high-temperature part. The temperature of an electric furnace is usually 900~1000°C. Therefore, both ends of a pyrolysis tube, the pyrolysis side, and a thermal insulator are hot. Do not touch them with naked hands.

CAUTION

For samples generating harmful gas, prepare connection lines from a pyrolysis tube outlet and discharge the gas by local exhaust.

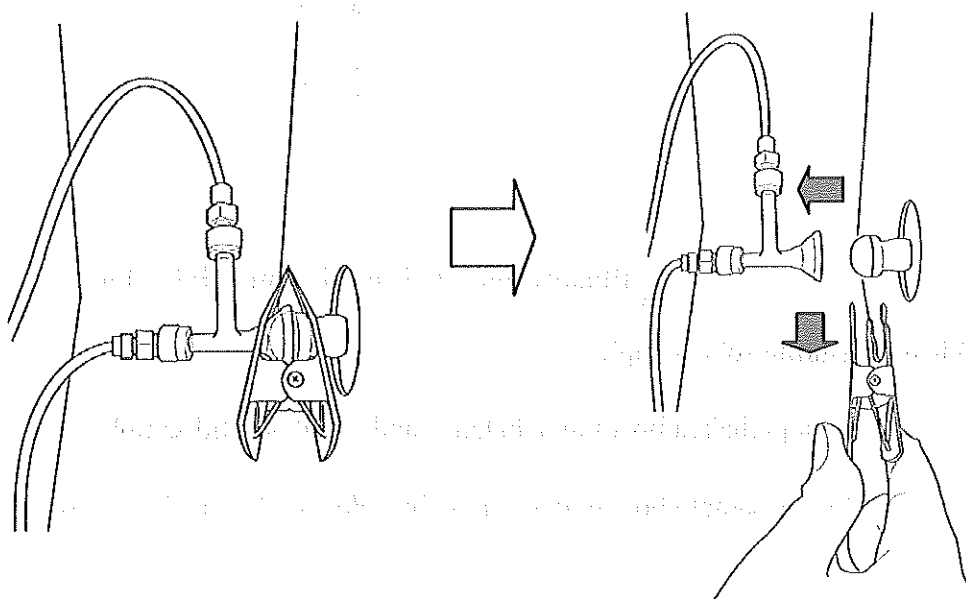


Illustration 5-8. Pyrolysis tube outlet removal

5-6-2. Sample injection

Weigh solid samples before 5-6-3. Combustion by ABC manual operation.

For solid samples and liquid samples, when the message of sample boat setting is displayed at (2) of 5-6-3. Combustion by ABC manual operation, inject sample.

CAUTION

Sample volume should be under $100\ \mu\text{l}$ or 100mg .

Some samples can cause incomplete combustion.

In that case, reduce the sample volume and consider combustion conditions. Incomplete combustion when a ball joint is connected causes the contamination of a pyrolysis tube and a PTFE tube with "WASH. G".

When measuring liquid samples

- ① Set the boat which is prebaked with $0.02\sim 0.03\text{g}$ quartz wool to a sample introduction box. (After the second time, use the previous boat.)
- ② Take a sample into a microsyringe. Inject it into the sample boat.
(Refer to Illustration 5-9 Liquid sample injection.)

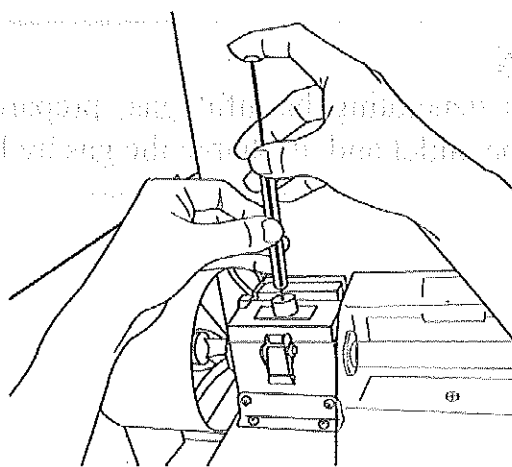


Illustration 5-9. Liquid sample injection

When measuring solid samples

- ① Put a prebaked boat into a balance and weigh a solid sample.
- ② Put the sample boat into a sample introduction box and set it to a ladle.
- ③ Close a sample introduction box cover and lock it.

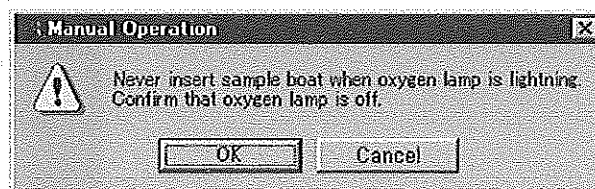
5-6-3. Combustion by ABC manual operation

Operate ABC manually and check appropriate combustion conditions for samples.

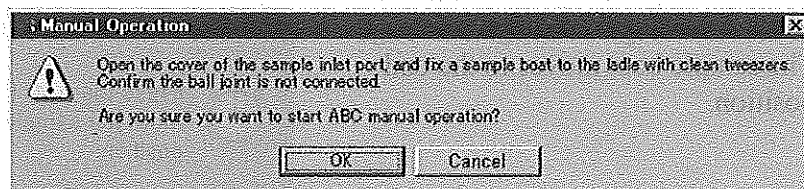
POINT

- Combustion conditions are different by sample quality and volume. By setting it so that the combustion should be peak at the second stop position, the gain of optimum conditions is easier. Optimum conditions are the recovery maximum value by complete combustion.
- Remove a clamp for a ball joint of a pyrolysis tube outlet and check that the pyrolysis tube outlet and a ball joint (with branch tubes) are removed. Incomplete combustion when a ball joint is connected contaminates a pyrolysis tube and gas lines.

- (1) Click "Run" and "ABC Manual". "ABC Programs" dialog box and "Manual Operation" dialog box for caution are displayed.



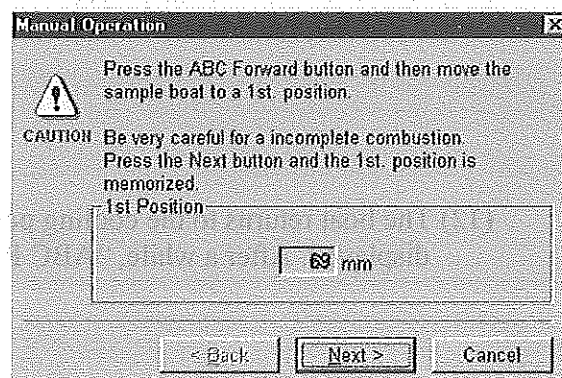
- (2) Click [OK] button. "Manual Operation" dialog box is displayed to check accessory manual operation start.



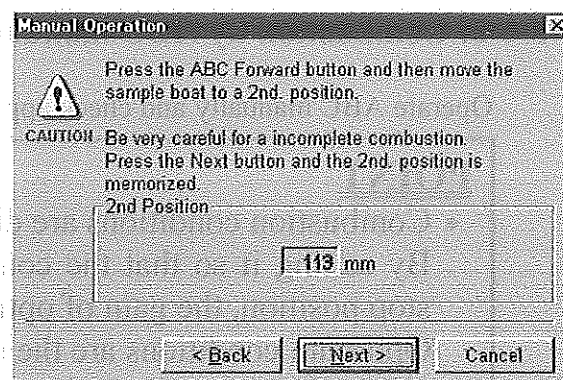
- (3) Set sample in a sample boat by referring to 5-6-2. Sample injection. Click [OK] button. ABC AUTO lamp is off and "Manual Operation" dialog box is displayed to check the first stop position of a sample boat.

- (4) Adjust the first stop position of the sample boat with <FWD> or <REV> button of ABC. The set first stop position is displayed in the dialog box.

- (5) When the first stop position and time are determined, click [NEXT] button. "Manual Operation" dialog box is displayed to check the second stop position of the sample boat.

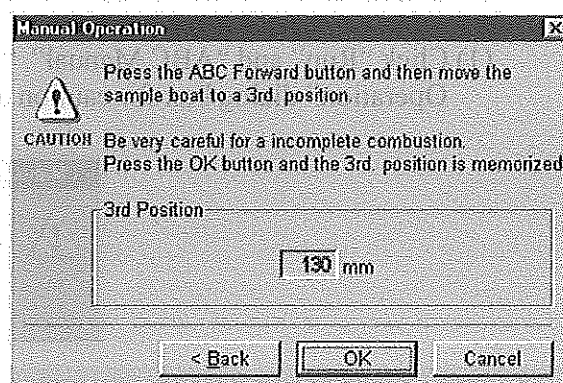


- (6) Adjust the second stop position of the sample boat with <FWD> or <REV> button of ABC. The set second stop position is displayed in the dialog box.



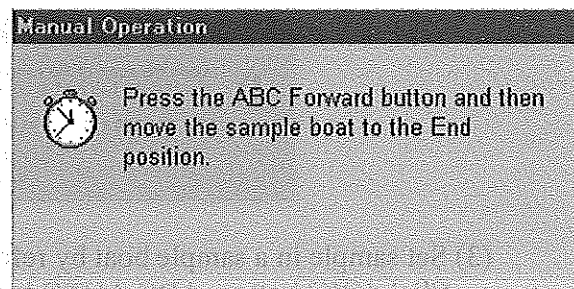
- (7) When the second stop position and time are determined, click [NEXT] button. "Manual Operation" dialog box is displayed to check the third stop position of the sample boat.

- (8) Adjust the third stop position of the sample boat with <FWD> or <REV> button of ABC. The set third stop position is displayed in the dialog box.

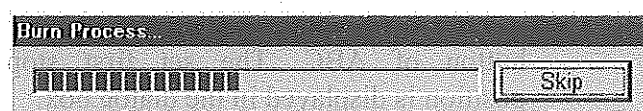


- (9) When the third stop position and time are determined, click [OK] button.

- (10) Continue to press <FWD> button of ABC according to the right message. The sample boat moves to the end position.



- (11) "Burns Process" is displayed.



- (12) The boat returns to the cooling position after combustion. "O2 Off Process Waiting" is displayed. After a while, "ABC Programs" dialog box returns.

- (13) The result of manual operation is inputted into "Edit" frame as program name of "Manual".

Program name can be inputted directly and changed.

Input Program No. (1~28) which is not in the list and press <Enter> key. [Add] button is effective.

The screenshot shows the 'ABC Programs' window. It contains a table of programs and an 'Edit' form below it.

No.	Program	ABC Parameter								Analysis		
		1st Pos. Time	2nd Pos. Time	3rd Pos. Time	End Time	Cool Time	Boil. Secs	Ar Time	O2 Time			
1	Oil/20ul	100	0	120	30	150	0	40	20	10	0	40
2	Oil/50ul	100	0	120	60	180	0	40	20	10	0	40
20	Test	85	5	110	5	125	5	100	5	20	30	600
29	Boat Prebake	0	0	0	0	0	0	120	60	20	0	120
30	H/W TEST	65	5	135	5	145	5	5	5	50	0	60

Below the table is an 'Edit' form with the following fields and buttons:

- Program Name: Manual
- 1st Pos. Time: 69
- 2nd Pos. Time: 30
- 3rd Pos. Time: 113
- End Time: 30
- Cool Time: 136
- Boil. Secs: 30
- Ar Time: 120
- O2 Time: 120
- Analysis Time: 10
- Analysis Time: 30
- Buttons: Manual Operation, Add, Delete, OK, Cancel
- Status: No.: 1-28 ABC Max Position: 269mm

- (14) When transferring manual operation result into ABC program, click [Add] button. The result is added to the list as new ABC program.

- (15) Click [OK] button. The changed contents of ABC program list are saved and the main window returns.

* Click [Cancel] button to return to the main window without saving changed contents.

* Click [Manual Operation] button to run accessory manual operation again.

By repeating the consideration of combustion conditions by necessary times, obtain optimum conditions. Run (4)~(15) again.

- (16) After the consideration of combustion conditions by ABC manual operation, set a clamp for a ball joint of a pyrolysis tube outlet, connect gas lines, and prepare combustion and absorption ion chromatography measurement. When connecting a pyrolysis tube outlet, refer to 5-7-4. Connection of a pyrolysis tube outlet.

POINT

After the consideration, set a ball joint (with branch tubes) to a pyrolysis tube outlet with a clamp so that combustion gas should be absorbed into absorption solvent. Consider combustion and absorption conditions by total operation including ion chromatography measurement.

5-7. Measurement

5-7-1. Measurement flow

POINT

When blank measurement values influence sample measurement values, correct blank values.

Prepare calibration curve including combustion and absorption like sample. By comparing A with B, recovery can be obtained.

A : Calibration curve prepared by combustion and absorption of standard solution and the direct injection into an ion chromatography unit

B : Calibration curve prepared by direct injection of standard solution into an ion chromatography unit

When recovery is known, calibration curve directly injected into an ion chromatography unit is available.

5-7-3. Method edit



5-7-4. Connection of a pyrolysis tube outlet

If necessary



5-7-5. Combustion



5-7-6. Method edit during measurement

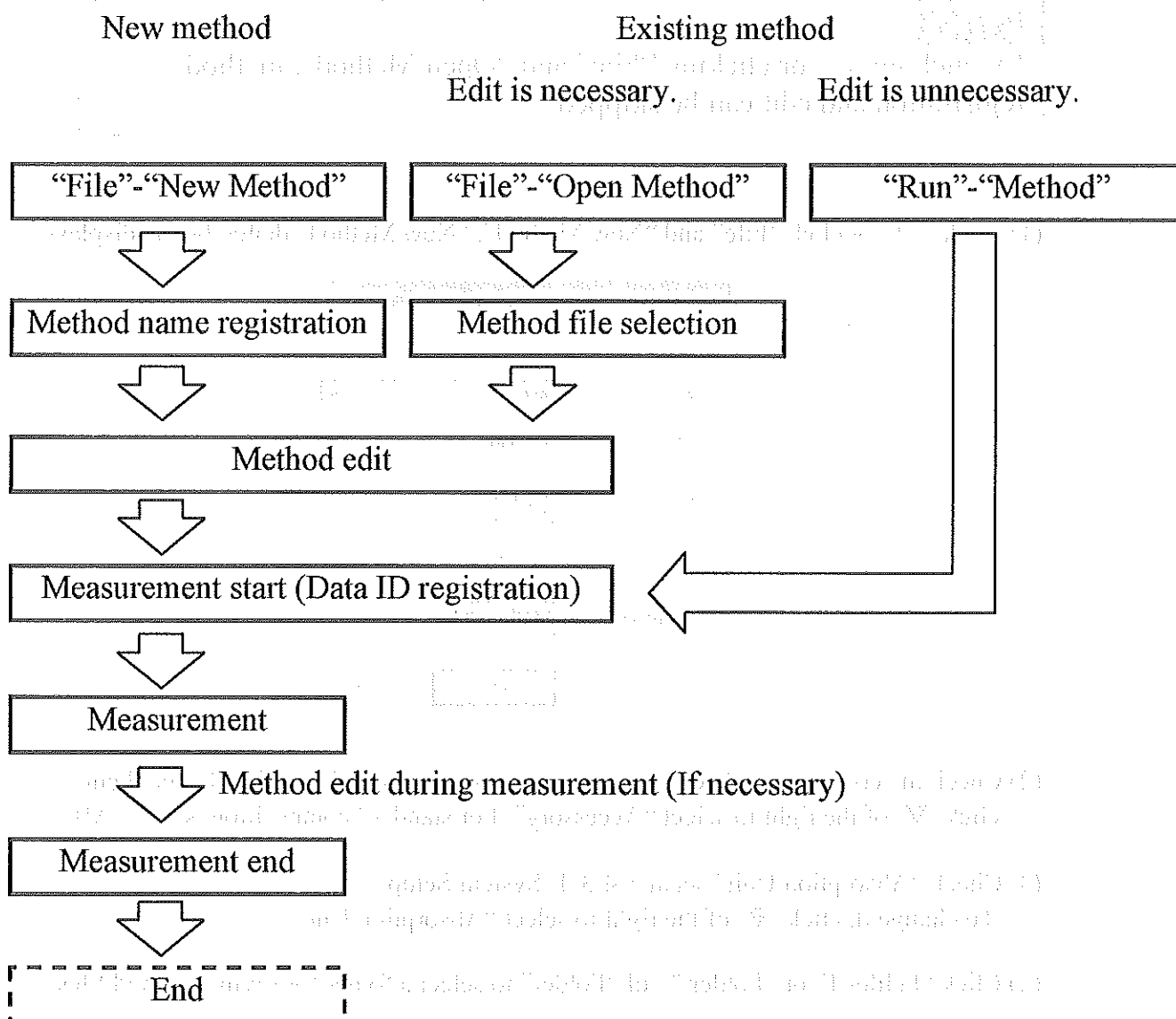
If necessary



5-8. Exit

5-7-2. Method setting


Basic method setting is as follows.



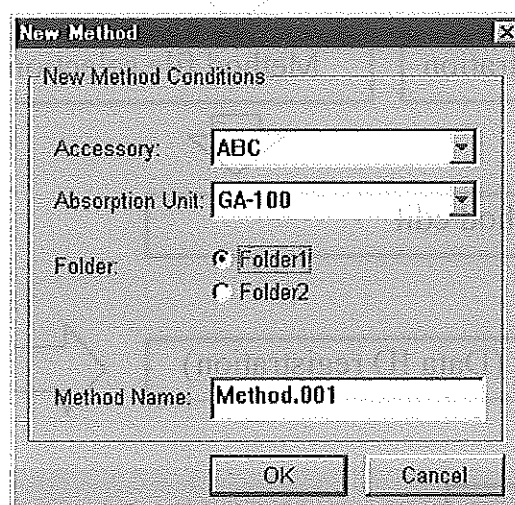
5-7-3. Method edit

Edit a measurement method. The procedure for preparing and editing a new method is described here.

POINT

By clicking  or clicking “File” and “Open Method”, method registration and edit can be skipped.

- (1) Click  or click “File” and “New Method”. “New Method” dialog box is displayed.



- (2) Check the contents of “Accessory” set at 5-4-3-1. System Setup. To change them, click ▼ of the right to select “Accessory”. For standard composition, select “ABC”.
- (3) Check “Absorption Unit” set at 5-4-3-1. System Setup.
To change it, click ▼ of the right to select “Absorption Unit”.
- (4) Click “Folder 1” or “Folder 2” of “Folder” to select a folder for saving method files.
- (5) Input a method name into “Method Name”. (Up to 20 characters)
* By clicking “System”, “Preference”, and “Measurement” tab, setting “Optional Method Naming Rule”, and clicking [OK] button, the set name is displayed in “Method Name” of “New Method” dialog box.

(6) Click [OK] button. Method edit dialog box is displayed.

When “ASC-150L+ABC” is selected, the above dialog box is displayed.

The dialog box has ASC-150L frame with “Btl No.”, “150L”, and “ABC”, but, for standard composition (when selecting ABC as an accessory), “Btl No.” and “150L” are not displayed.

(7) Set the following contents.

Item	Contents	Input range
Sample ID	Sample name	Input it necessarily.
Sample Size	Sample volume	0.01~9999.99
Sample Size Unit	Sample volume unit	Select it from μl , mg, ml, or g.
ABC Program No.	ABC Program No.	1~28

Table 5-3. Sample measurement input item

CAUTION

Sample volume should be under $100\ \mu\text{l}$ or 100mg.

Some samples can cause incomplete combustion.

In that case, reduce sample and consider combustion conditions.

Incomplete combustion when a ball joint is connected contaminates a pyrolysis tube and a PTFE tube with “WASH. G”.


When measuring liquid samples

Input sample volume into "Sample Size" and click ▼ of the right to set the unit to " μ l".

When measuring solid samples

Put a sample into a sample boat and weigh it. Input sample weight into "Sample Size" and click ▼ of the right to set the unit to "mg".

(8) Set ABC Program No. by sample conditions and volume.

Click  of ABC Program No. right. "ABC Programs" dialog box is displayed.

* When program No. is known, input it directly into "ABC Program No."

Proceed to (11).

ABC Programs												
No.	Program	ABC Parameter								Analysis		
		1st Pos. Time	2nd Pos. Time	3rd Pos. Time	End Time	Cool Time	Boil Speed	Ar Time	O2 Time			
1	Oil/20ul	100	0	120	30	180	0	40	20	10	0	40
2	Oil/50ul	100	0	120	60	180	0	40	20	10	0	40
20	Test	85	5	110	5	125	5	100	5	20	30	600
29	Boat Prebake	0	0	0	0	0	120	60	20		0	120
30	H/W TEST	65	5	135	5	145	5	5	5	50	0	60

(9) Click ABC program to select it.

For preparing a new ABC program, refer to 4-5-1. Accessory (ABC) setting for the details.

- ① Click [Edit] button.
- ② Input a new number (1~28) into "No.".
- ③ Input "Program" and "ABC Parameter". Click [Add] button.
- ④ Click an added Program No.

- (10) Click [OK] button. The method edit dialog box is displayed. Selected Program No. is displayed in "ABC Program No."
- (11) Click [Add] button by combustion times. The same setting contents are added into the below list by clicked times.

	Tube	Sample ID	Sample Size	Sample Size Unit	ABC
▶	1	oil	15.40	mg	1

- (12) Like other sample settings, add them into the list by (7)~ (11).
 * For each setting addition and deletion, refer to 4-4-3-1. Edit flow.

	Tube	Sample ID	Sample Size	Sample Size Unit	ABC
	1	Oil	15.40	mg	1
	2	Oil	15.70	mg	1
	3	Pellet	20.20	mg	2
▶	4	Pellet	19.80	mg	2

- (13) By clicking [Accumulate] button, combusted sample gas is absorbed into the same tube as the cursor position number. After the last combusted sample gas is absorbed into the same number tube, absorption solvent is injected into an ion chromatography unit.

	Tube	Sample ID	Sample Size	Sample Size Unit	ABC
	1	Oil	15.40	mg	1
	2	Oil	15.70	mg	1
	3	Pellet	20.20	mg	2
	4	Pellet	19.80	mg	2
	5	Resin	18.00	mg	4
	5	Resin	17.60	mg	4
▶	5	Resin	17.90	mg	4

POINT

[Accumulate] button is useful when combusting and absorbing many samples.

5-7-4. Connection of a pyrolysis tube outlet

Set a ball joint (with branch tubes) to the outlet of a pyrolysis tube and fix it to the pyrolysis tube with a clamp for a ball joint.

CAUTION

Do not touch high-temperature part. The temperature of an electric furnace is usually 900~1000°C. Therefore, both ends of a pyrolysis tube, the pyrolysis side, and a thermal insulator are hot. Do not touch them with naked hands.

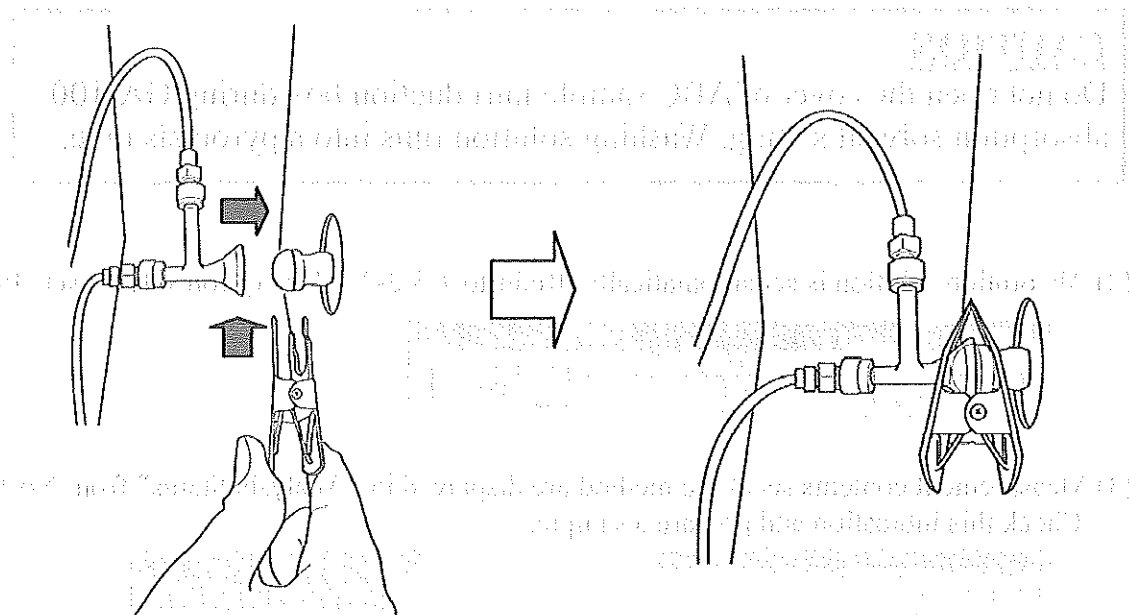
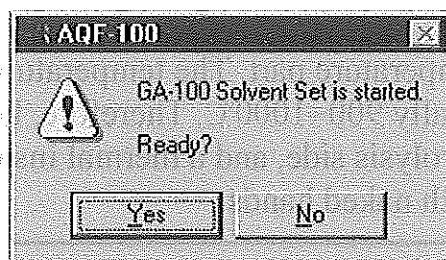


Illustration 5-10. Connection of a pyrolysis tube outlet

5-7-5. Combustion

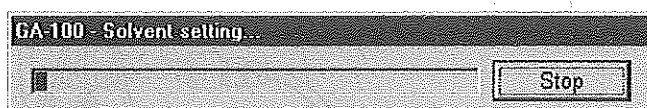
- (1) Set absorption solvent to an absorption tube. When the following message is displayed, click [Yes] button. To set absorption solvent after a while, click [No] button. To run combustion again, click "Run" and "GA-100 Start". The following window is displayed.



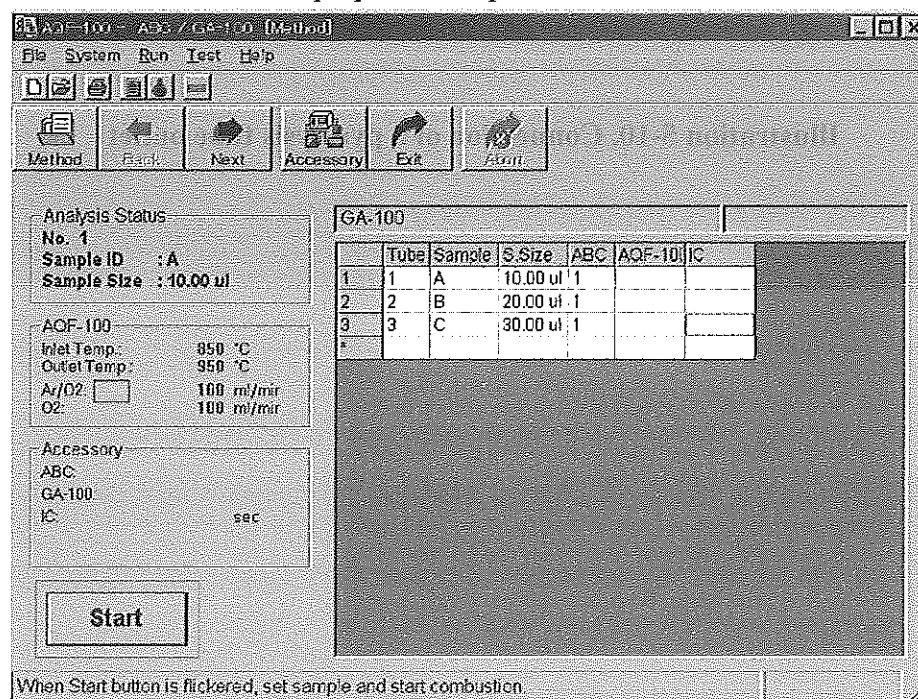
CAUTION

Do not open the cover of ABC sample introduction box during GA-100 absorption solvent setting. Washing solution runs into a pyrolysis tube.

- (2) Absorption solution is set automatically. Refer to 4-5-2-2. "Absorption solvent set" flow.



- (3) Measurement contents set at the method are displayed in "Analysis Status" from No.1. Check this indication and prepare a sample.



- (4) Check that [Start] button flickers and set the sample.

For liquid samples

- ① Set a prebaked sample boat to a sample introduction box.
(After the second time, use the previous boat.)
- ② Take a sample into a microsyringe. Inject it into the sample boat.
(Refer to Illustration 5-9. Liquid sample injection.)

For solid samples (Run ① and ② at method edit.)

- ① Set a prebaked boat on a balance and weigh a sample.
- ② Input the sample weight into "Sample Size". (Refer to 5-7-3. Method edit.)
- ③ Put the sample boat into a sample introduction box and set it to a ladle.
- ④ Close the cover of the sample introduction box and lock it.

(5) After sample setting, click [Start] button or <Enter> key.


Combustion starts by ABC program contents.

(6) The following contents are displayed in "AQF-100" and "IC" of the list in [Progress] frame.

Types and contents in "AQF-100"	Types and contents in "IC"
Combustion : During combustion	Measurement : During measurement
Absorption : During combustion gas absorption	Finished : Measurement end
Finished : Combustion end (Buzzer beeps 5 times.)	Abort : Measurement stop
Abort : Combustion stop	

POINT



By clicking , ABC conditions can be displayed.
Red part is the position of a sample boat.

POINT

When incomplete combustion occurs, turn off GA-100 power switch before the absorption solvent is sampled.
When the solvent runs into the separation column of an ion chromatography unit, the column is contaminated and separation power can be bad.

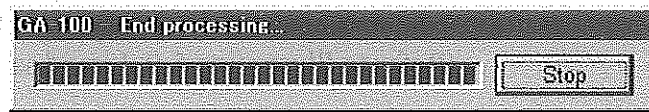
(7) For measurement on and after No.2, repeat (1)~(6).

When ASC-150L or ASC-120S is used, subsequent measurement is run automatically.

(8) After all measurement, "Finished" is displayed in "AQF-100" and "IC" frames.



- (9) After method measurement, click **Exit** or click “Run”, “Operation”, and “Exit Run”. “GA-100-End processing” is displayed.



End washing is run automatically. Refer to 4-5-2-6. End Wash flow.

- (10) Print methods if necessary. Refer to 4-6-2. Print type.

When troubles occur during combustion.

CAUTION

When troubles occur during combustion, click [Abort] button and suspend combustion. Each unit operates as follows.

GA-100 : Absorption solvent sampling stops.


AQF-100 : Heater is off.

ABC : A sample boat stops at the cool position and returns to the home position.

Take measures against troubles and check that the unit is not wrong.

Combustion can't be continued. Therefore, run combustion again by the same method.


Measurement stop

By clicking  button during method measurement, the current sample measurement is stopped.

Measurement suspension (displayed when ASC-150L or ASC-120S is used)

Click "Run", "Operation", and "Abort". The automatic start of the next sample measurement is suspended after the current sample measurement.

Remeasurement


During method measurement, by clicking  or clicking "Run", "Operation", and "Back" before measurement, the previous measurement can be run again. But the previous result is overwritten and canceled.

Measurement skip

During method measurement, by clicking  or clicking "Run", "Operation", and "Next" before measurement, the next measurement can be run.

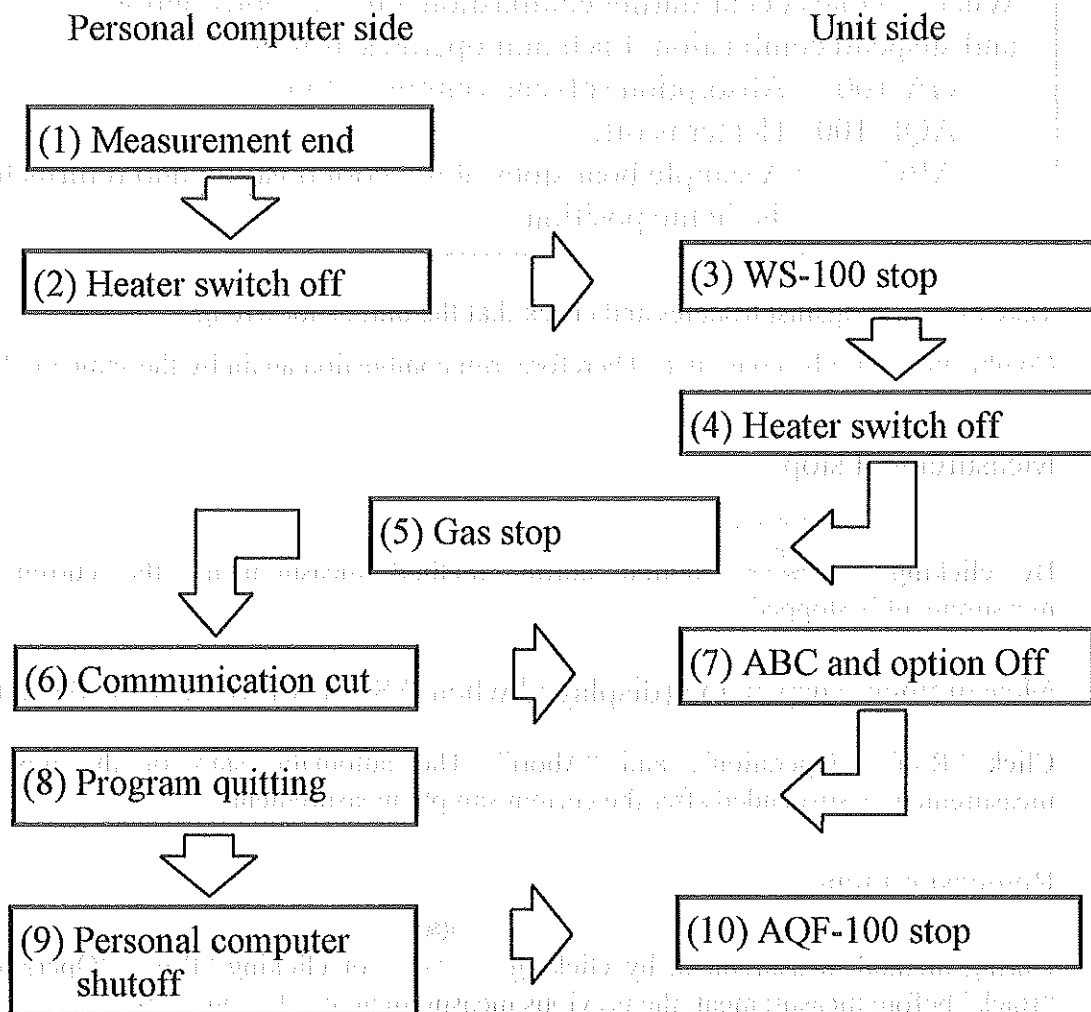
5-7-6. Method edit during measurement

By editing method during measurement, measurement can be added and deleted.

- (1) Click  or click "Run", "Operation", and "Method". Method edit dialog box is displayed.
- (2) Add or delete a method like method edit.
- (3) Click [Run Method] button. The main window returns.

5-8. Exit

After measurement, stop the unit as follows and exit system program.






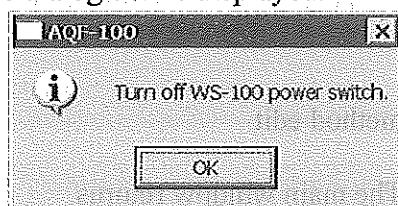
- (1) After method measurement, click  or click “Run”, “Operation”, and “Exit Run” to end measurement.

* Measurement can be ended halfway.

End Wash is run automatically. Print a method if necessary.

- (2) System program heater switch off

- ① Click  or click “System” and “Heater”. “Heater” dialog box is displayed.
- ② Click “Off”.
- ③ Click [OK] button. “Heater” dialog box is closed and  (red) changed to  (blue). The dialog box is displayed.



- ④ Click [OK] button. Water supply stops automatically.

(3) WS-100 stop

Turn off the power switch.

* If WS-100 power switch is on when the heater switch is off, the pyrolysis tube is filled with water.

(4) AQF-100 heater switch off

Turn off the front heater switch.

Wait until Inlet Temp. and Outlet Temp. of a pyrolysis tube are under 500°C.

POINT

By closing gas valves before the electric furnace cools, moisture enter the mass flow sensor and the unit breaks.
After it cools, proceed to the next procedure.

(5) Gas stop

Close the main valves of O₂ and Ar cylinders.

(6) Communication disconnection

Click "System", and "System Setup", and [Disconnect] button.
Communication to AQF-100 is disconnected.

(7) Unit stop

Turn off the following power switches.

- ① GA-100 power switch
- ② Cooler switch of ABC front
- ③ Option power switch (if connected)

(8) System program quitting

Click "×" of upper right or click "File" and "Exit". AQF-100 system program ends.

(9) Personal computer shutoff

- ① Click [Start] button of a taskbar.
- ② Check that "Shut down the computer?" is selected. Click [Yes] button.
The computer power is off.
- ③ Turn off the power switches of a printer and a monitor.

(10) AQF-100 stop

CAUTION

To cool an electric furnace with a cooling fan, stop AQF-100 after the temperature of a pyrolysis tube is under 500°C.

Turn off AQF-100 front power switch. ABC power switch is off automatically.

Section 6: Troubleshooting

Countermeasures against hardware or software troubles when using AQF-100 and system program are described in this section.

CAUTION

Do not take AQF-100 cover except our servicemen.
An electric shock and a fire can be caused.

No power of AQF-100

Point	Countermeasure
Is the main power switch of the rear of AQF-100 ON?	Turn on the power switch.
Is the power connector in the rear of AQF-100 disconnected?	Connect it firmly.
Is the power plug put in the outlet?	Connect it firmly.
Is the power supplied to the outlet?	Connect another electric appliance to the outlet. When it operates normally, AQF-100 can be broken. Contact local distributors.
Is AQF-100 power fuse cut?	(1) Take off the fuse holder of the rear panel with a screwdriver. (2) Check the conduction with a tester. (3) Without conduction, change it for new one.

No power of GA-100

Point	Countermeasure
Is the main power switch of the rear of GA-100 ON?	Turn on the power switch.
Is the power connector in the rear of GA-100 disconnected?	Connect it firmly.
Is the power plug put in the outlet?	Connect it firmly.
Is the power supplied to the outlet?	Connect another electric appliance to the outlet. When it operates normally, GA-100 can be broken. Contact local distributors.
Is GA-100 fuse switch lowered?	(1) Check the cause and take measures against it. (2) Raise the switch.


No gas flow

Point	Countermeasure
Is the gas cylinder valve open?	Open the gas cylinder valve.
Is the quantity of gas in the cylinder sufficient?	If the gas quantity is insufficient, change the cylinder for new one.
Is the room gas supply valve open?	Open it.
Is the line disconnected?	Connect the line.
Is the line clogged?	Change the clogged line.



ABC does not operate.

Point	Countermeasure
Is an error message is displayed?	Check "System Setup" by the following procedure. (1) When the main window of AQF-100 system program is displayed, press <F5> key. "System Setup" dialog box is displayed. (2) Click ▼ of "Accessory" to select "ABC". (3) Click [Transmit] button.
Is the cable to AQF-100 disconnected?	Insert firmly a signal connector of ABC rear. Insert firmly ABC connector of AQF-100 rear.

The temperature of an electric furnace doesn't rise.

Point	Countermeasure
Is an error message indicated in a personal computer display?	Operate the system program according to the error message.
Is the heater switch on the front of AQF-100 ON?	Turn on the heater switch.
Is the heater switch of system program ON?	Turn on heater switch by the following procedure. (1) Click  or "System" and "Heater". (2) Check the heater temperature setting. (3) To change the set contents, input the value directly. (4) Click "On". (5) Click [OK] button.
Is the circuit protector operating?	When the circuit protector button on the rear of AQF-100 stands out, the circuit protector is operating. The heater circuit can be wrong. Contact local distributors.

Peak of ion chromatography is not obtained.

Point	Countermeasure
Is the temperature of an electric furnace up?	<p>Check the following points by AQF-100 system program.</p> <p>(1) Is  (Heater off) changed to  (Heater ON)? When the button does not change, turn on the heater switch. Refer to 5-4-5. Heater On.</p> <p>(2) Are Inlet Temp. and Outlet Temp. indicated? When they are not indicated, check the measurement condition.</p> <p>(3) Is the measurement condition correct? When the condition is not correct, set the appropriate condition. Refer to 5-4-3-2. Analysis Parameters. Without the recovery, contact our distributors.</p> <p>(4) Is sample concentration is low? Increase sampling volume or run accumulative measurement.</p>
Is a clip for a ball joint disconnected?	Fix a ball joint to a pyrolysis tube outlet with the clip.
Is the line to an ion chromatography unit disconnected?	Connect the line firmly.
Is constituent concentration in sample too low?	Add sample or combust it repeatedly.

Dispersion of measurement values

Point	Countermeasure
Are the pressure and flow of oxygen and argon inappropriate?	<p>Set gas pressure as follows.</p> <p>(1) Set the secondary pressure of a cylinder or a stop valve to 0.4 ± 0.1 MPa with the reducing valve.</p> <p>(2) Set gas flow to the specified value by the knob.</p>
Is the temperature setting of the electric furnace in combustion part inappropriate?	Set properly the temperature of the electric furnace in combustion part. Refer to 5-4-5. Heater On.
Does the gas leak from the connection part of a pyrolysis tube?	Check the gas leakage. Refer to 5-4-4-2. Gas leakage check.
Is the pyrolysis tube contaminated?	<p>Clean the contaminated pyrolysis tube as follows.</p> <ul style="list-style-type: none"> Soak the whole of a pyrolysis tube in washing solution. Supersonic cleaning with washing solution, etc. <p>In either case, wash the pyrolysis tube with pure water and dry it.</p>
Is the quartz wool of a pyrolysis tube deteriorated?	Change the quartz wool in the pyrolysis tube. Refer to 3-4-1. Filling of quartz wool for the details.

Leakage buzzer sounds.

Point	Countermeasure
Does solution leak?	Check leakage points and take measures. Refer to 8-3. Taking out of GA-100 inside case.

GA-100 stops.

Point	Countermeasure
Does solution flow to a drain tube?	<p>“Gas Line” When the pump stops</p> <ol style="list-style-type: none"> 1. Remove the PTFE tube set to the branch tube (near the ground part) of the ball joint connected to a pyrolysis tube outlet. 2. Set an evacuation syringe of a WS-100 attachment to the tip of the removed PTFE tube. 3. Press Gas Line key while absorbing washing water with the syringe. <p>“Absorption Tube” When the pump stops</p> <ol style="list-style-type: none"> 1. Remove the PTFE tube (not connected to an absorption solution injection pump). 2. Set an evacuation syringe of a WS-100 attachment to the tip of the removed PTFE tube. 3. Press Absorption Tube key while absorbing washing water with the syringe. <p>“Drain” When the pump stops</p> <ol style="list-style-type: none"> 1. Remove the PTFE tube with “ABS DRAIN” tag in a drain tank. 2. Set an evacuation syringe of a WS-100 attachment to the tip of the removed PTFE tube. 3. Press Absorption Tube key to pour washing water into the absorption tube. 4. Press Drain key while absorbing washing water with the syringe. <p>“Sampling” When the pump stops</p> <ol style="list-style-type: none"> 1. Remove the PTFE tube with “SMP DRAIN” tag in a drain tank. 2. Set an evacuation syringe of a WS-100 attachment to the tip of the removed PTFE tube. 3. Press Absorption Tube key to pour washing water into the absorption tube. 4. Press Sampling key while absorbing washing water with the syringe.

Absorption solution and standard solution are not absorbed and a chromatography peak is not displayed.

Point	Countermeasure
Does solution flow to a drain tube?	<p>When absorption solution is not absorbed</p> <ol style="list-style-type: none"> 1. Remove the ball joint connected to a pyrolysis tube outlet. 2. Press Absorption Tube key to pour washing water into the absorption up to the branch tube height. 3. Remove the PTFE tube with “SMP DRAIN” tag in a drain tank. 4. Set an evacuation syringe of a WS-100 attachment to the tip of the removed PTFE tube. 5. Press Sampling key while absorbing washing water with the syringe. 6. Check that the PTFE tube is filled with washing solution.

Section 6: Troubleshooting

Section 7: Error Messages

This section describes warnings and error messages displayed in a monitor when troubles occur.

CAUTION

Danger and Warning displayed in a personal computer monitor show the danger of a serious accident. When this message is displayed, take measures immediately.

ERROR No.	Error messages	Status	Countermeasures
010	WATER LEAKAGE ERROR	Warning	Check leak parts and handle them.
061	THERMOSTAT ON ERROR	Warning	Turn off AQF-100 power switch immediately. Contact our distributors.
062	OVER TEMP. ERROR	System Down	Turn off AQF-100 power switch immediately. Heater errors occurred and system down was run. Check the cause of heater temperature errors.
063	OVER TEMP. 1 ERROR		
064	OVER TEMP. 2 ERROR		
065	UNDER TEMP. 1 ERROR		
066	UNDER TEMP. 2 ERROR		
077	COMMUNICATION ERROR	System Down	Check the cable connecting AQF-100 to a personal computer and restart the system. When error occurs even after the restart, contact our distributors.
082	Ar/O2 GAS FLOW ERROR	System Down	Dander of explosion Keep off the unit more than 5m. Gas flow errors occurred and an option was reset. Therefore system down was run. After the option returns to the home position and 30 minutes passes, check the cause of gas flow decrease.
086	O2 GAS FLOW ERROR		
101	ABC CABLE CONNECTION ERROR	Warning	The cable between AQF and ABC is not connected. Connect the cable and restart the system.
105	COVER OPEN ERROR	Warning	Close ABC safety cover and restart the system.

Table 7-1. Error messages

Section 7: Error Messages

Section 8: Maintenance and Inspection

8-1. Unit Inspection

8-1-1. Daily inspection

CAUTION

Check the unit every day before the use. If you fail to check it, it doesn't operate properly and a serious accident can occur.

No.	Item	Contents
1	Gas leakage check	Check the leakage of O ₂ and Ar gases with a flow meter for gas leakage check.
2	Septum change	Change the septum every day at measurement start.
3	Ball joint contamination check	Check that the ball joint is free from contamination such as soot.
4	Inline filter contamination check	A inline filter is contaminated by incomplete combustion.
5	PTFE tube contamination check	A PTFE tube is contaminated by incomplete combustion.
6	Microsyringe contamination and needlepoint bend checks	Check that a syringe plunger is not contaminated and the needlepoint is not bent.
7	Sample boat contamination check	Check that a sample boat is not contaminated or devitrified.
8	Absorption part contamination check	Check the contamination of absorption part of sample gas.
9	Absorption tube change	Change an absorption tube by sample concentration.

Table 8-1. Items of daily inspection

1. Gas leakage check

Check no leakage of supply gas (O₂ and Ar). Refer to 5-4-4-2. Gas leakage check.

(1) Connect the ball joint of a flow meter for gas leak check to the outlet of a pyrolysis tube.

(2) Check that the set gas flow and the indicated value of the flow meter agree.

2. Septum change

Replace the septum with new one before measurement every day.

Refer to 3-6-2. Setting of a septum and a septum holder.

3. Ball joint contamination check

Check that a ball joint is not contaminated with soot by incomplete combustion.
When the ball joint is contaminated, wash it as follows.

- (1) Wipe the ball joint with absorbent cotton immersed in toluene.
- (2) Prepare washing water by mixing acetone and distilled water in the ratio of 1 : 10.
- (3) Immerse the ball joint in washing water.
- (4) Wash it with mild detergent for 5 minutes.
- (5) Replace washing solution with distilled water.
- (6) Wash it with mild detergent for 5 minutes like (4).
- (7) After the washing, dry it fully.

4. Inline filter contamination check

By long-term use and sample incomplete combustion, an inline filter is contaminated.
Column pressure of an ion chromatography unit is up and peak form is wrong.
Replace it with new one.

5. PTFE tube contamination check

By long-term use and sample incomplete combustion, PTFE tubes are contaminated.
Replace them with new ones.

6. Check of microsyringe contamination and needlepoint bend

In the following cases, change a microsyringe.

- A microsyringe plunger doesn't move smoothly.
- PTFE part of the plunger tip is thin and the plunger is loose.
- The microsyringe needlepoint is bent.

7. Sample boat contamination check

When sample boat contamination and devitrification are heavy, replace it with new one.

8. Absorption part contamination check

Absorption part is damaged by washing with a supersonic washing machine or a brush. Therefore wash absorption part as follows.

- (1) Prepare cleanser to a beaker. For the preparation and the use, follow the contents described in a cleanser package.
- (2) Remove an absorption tube and immerse respective parts in a beaker not to break them. After the immersion, wash them with ultrapure water. Dry and keep them. If absorption part contamination is difficult to remove by incomplete combustion, wash only an absorption tube with "organic solvent".

CAUTION

Do not flow directly organic solvent such as toluene and acetone into absorption parts. GA-100 inside valve can break.

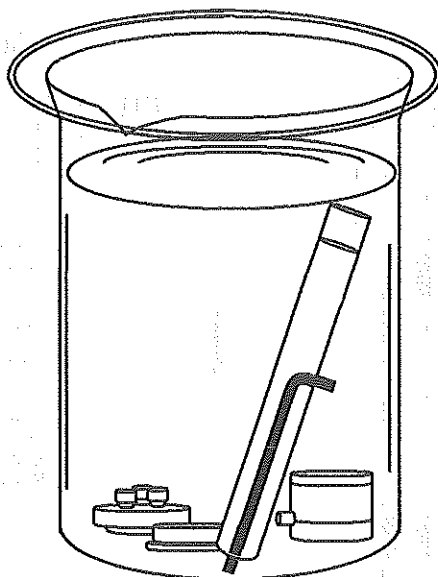


Illustration 8-1. Absorption part washing example

9. Absorption tube change

Illustration 8-2-1. and Illustration 8-2-2. indicate GA-100 absorption part change.

- (1) Remove a vinyl tube from an overflow tank.
- (2) Remove $\phi 6/3$ gas inlet connectors (2 pcs) of PTFE tubes of an absorption tube combustion gas inlet and for drain.
- (3) Loosen 2 thumbscrews and remove the absorption part.
- (4) Remove the overflow tank cap, the overflow tank, and an absorption tube.
- (5) Put an O-ring into the overflow tank. Screw in an O-ring holder lightly to fix it.

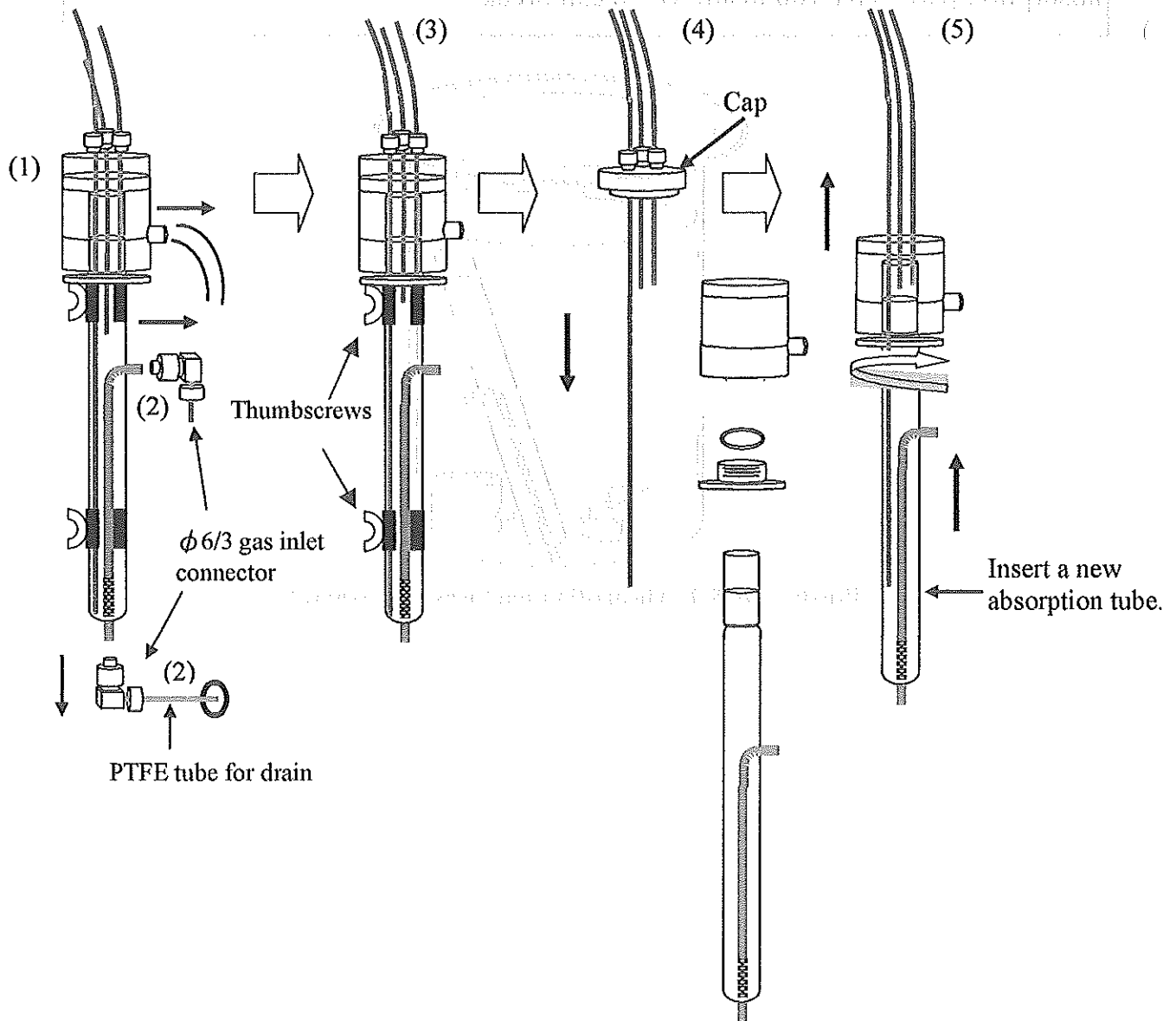


Illustration 8-2-1. GA-100 absorption part change

- (6) Put the overflow tank cap to which tubes (3 pcs) are set into the tank so that each tube should be put into the absorption tube.
- (7) Insert a new absorption tube into GA-100 holder and fix it with thumbscrews.
Adjust the tip length of PEEK tube (green) to touch the bottom of the absorption tube.
- (8) Connect $\phi 6/3$ gas inlet connectors (2 pcs) removed at (2).
- (9) Connect the vinyl tube to the overflow tank from GA-100 right side tube outlet.

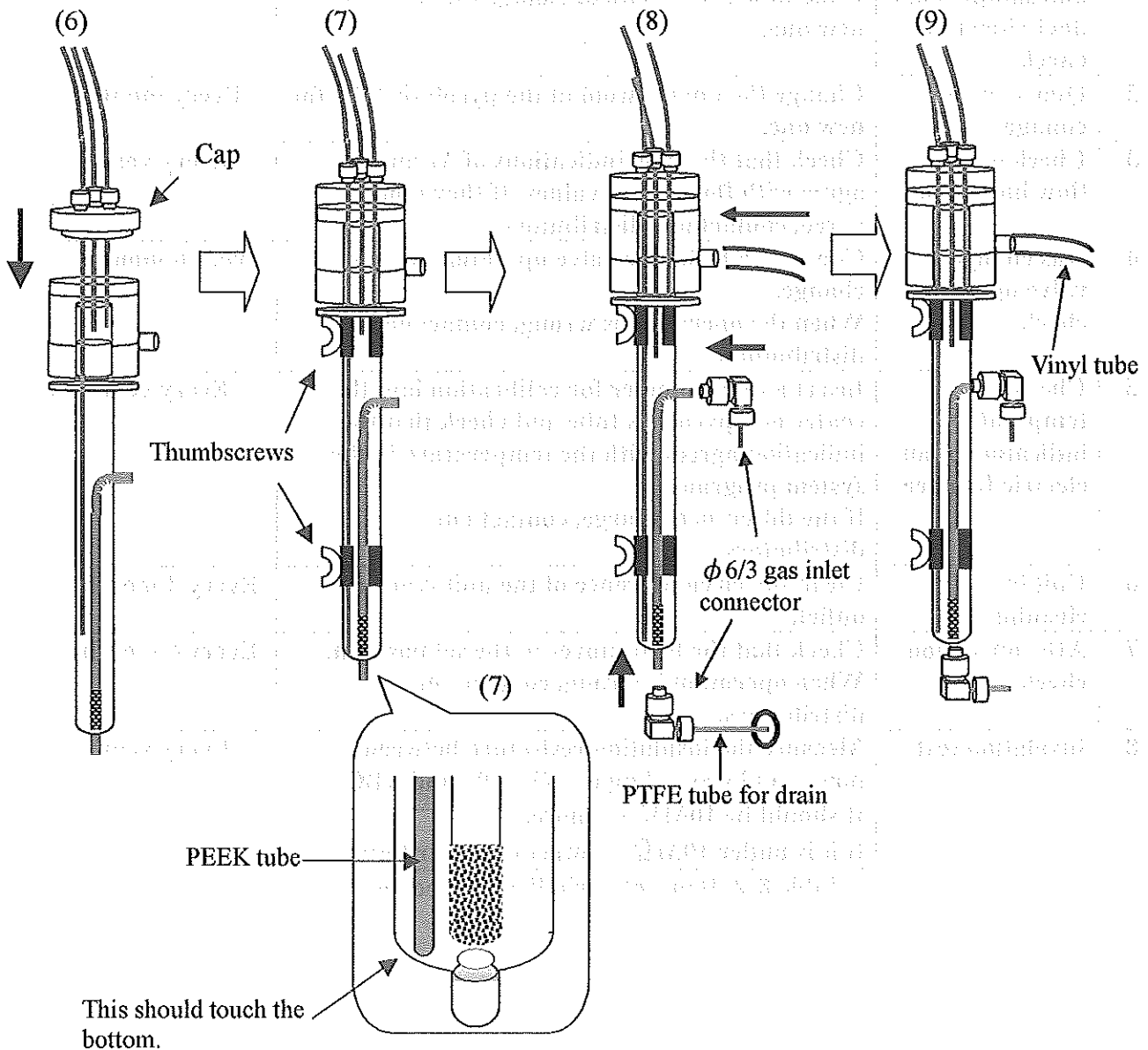


Illustration 8-2-2. GA-100 absorption part change

8-1-2. Periodical maintenance**CAUTION**

Maintain the unit necessarily by regular frequency. If you fail to inspect the unit regularly, the unit doesn't perform properly and serious accidents can be caused.

No.	Item	Contents	Frequency
1	Pyrolysis tube and sample boat devitrification check	Check discoloring (whitening). If the tube is devitrified, change the tube for new one.	Every month
2	Quartz wool change	Change the quartz wool in the pyrolysis tube for new one.	Every month
3	Check of gas flow indication	Check that the flow indications of Ar and O ₂ agree with flow meter values. If they don't agree, contact our distributors.	Every year
4	Gas change valve operation check	Check Ar/O ₂ change valve operation by gas change. When the operation is wrong, contact our distributors.	Every 6 months
5	Check of temperature indication of an electric furnace	Insert a thermometer for calibration into the center of a pyrolysis tube and check that the indication agrees with the temperature in the system program. If the difference is large, contact our distributors.	Every year
6	Unit fan cleaning	Clean the circumference of the unit rear fan outlet.	Every 3 months
7	ABC operation check	Check that the boat moves to the set position. When operation is wrong, contact our distributors.	Every 6 months
8	Insulation test	Measure the insulation resistance between power and grounding of AQF-100 and ABC. It should be 10M Ω or more. If it is under 10M Ω , contact our distributors.	Every year

Table 8-2. Items of periodical inspection

1. Pyrolysis tube and sample boat devitrification check

CAUTION

Pyrolysis tube and sample boat devitrification deteriorate mechanical strength and causes the breakage. White discoloration of a pyrolysis tube is devitrification. When devitrification is heavy and fine cracks are in devitrified parts, mechanical strength is low. Change the tube immediately.

Pyrolysis tube inspection and change

Inspection timing: Every month or after measurement of samples containing much alkali

(1) Check mechanical strength of a pyrolysis tube by checking the devitrified part.
* When fine cracks are in white devitrified parts, mechanical strength is low.

(2) When a pyrolysis tube is devitrified and mechanical strength is low, change the pyrolysis tube immediately. Refer to 3-4. Preparation for pyrolysis tubes.

Sample boat inspection and change

(1) Salt in samples adheres to the sample boat and the boat is whitened. After measuring samples containing much salts and metals, check devitrification.

(2) When devitrification is heavy, replace it with new one.

2. Change of quartz wool in pyrolysis tubes

Salts and metals in samples are accumulated on the quartz wool in the pyrolysis tube.

Inspection timing: Every month or after measurement of sample containing much salts and metals

When the quartz wool is harder than original one or powdery, change it immediately.

Read 3-4-1. Filling of quartz wool.

3. Gas flow indication check

Inspection timing: Every year

(1) Connect a calibrated flow meter to GAS-OUT O₂ and GAS-OUT Ar.

(2) After System Setup, check the difference between Ar and O₂ flow displayed in AQF-100 frame and indicated the value of the flow meter.

Ar : 200ml/min

O₂ : 400ml/min

They should be within $\pm 10\%$.

4. Ar/O₂ gas change valve operation check

Check Ar/O₂ gas change valve operation by the following procedure.

Inspection timing: Every 6 months

CAUTION

Before this operation, turn off WS-100 power switch and check that there are no waterdrops in an inner pyrolysis tube.


If waterdrops remain, water vapor goes into the flow sensors of the unit inside and the flow sensors can break.

- (1) After System Setup, set Ar and O₂ as follows.
Ar: 200ml/min
O₂: 400ml/min
- (2) Stop oxygen gas supply. (If a stop valve is connected, close it.)
- (3) After 10 minutes or more, check that O₂ flow indication is 10ml/min or less.
(It can be checked sooner by loosening the middle of an oxygen gas line and discharging oxygen gas.)
- (4) Click "System" and "Ar/O₂ Gas" of the menu.
Click [Ar/O₂] button to change Ar to O₂.
- (5) After several minutes, check that Ar/O₂ flow indication is 10ml/min or less.
If it is not 10ml/min or less, the change valve can be broken.
- (6) Click [Close] button of "Ar/O₂ Gas" window to change O₂ to Ar.
- (7) Supply oxygen gas and stop argon gas supply.
- (8) After more than 15 minutes, check that Ar/O₂ flow indication is 10ml/min or less.
(It can be checked sooner by loosening the middle of an argon gas line and discharging argon gas.)
If it is not 10ml/min or less, the change valve can be broken.
- (9) Supply argon gas.

5. Check of temperature indication of an electric furnace

Inspection timing: Every year

- (1) Insert a calibrated thermometer (R-type thermocouple) into the center of a pyrolysis tube.

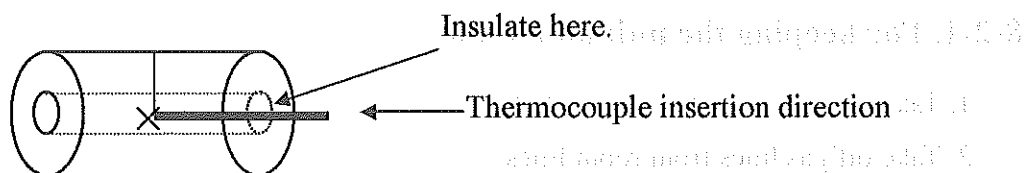
- (2) Click  or click "System" and "Heater" in the menu to set the temperature as follows.

Inlet Temp. : 900°C
Outlet Temp. : 900°C

- (3) Check the difference between the heater temperature displayed in AQF frame and thermometer one. The temperature in the center of the pyrolysis tube should be $900 \pm 50^\circ\text{C}$.

Take care of the following points.

- Insert the thermometer into the center in the depth direction of the electric furnace and in the radius direction of the pyrolysis tube.



- Insulate a thermometer.
- Use a calibrated thermocouple and an indicator for a thermometer.

6. Unit fan cleaning

Clean the circumference of the unit rear fan with an electric vacuum cleaner.

By using the unit when dust adheres to the circumference, the following phenomena occur.

- Heater cooling time is longer.
- After heater temperature rise, the sensor of temperature control functions and the heater switch is off automatically.

Inspection timing: Every 3 months (For dusty places, increase cleaning times.)

Section 8: Maintenance and Inspection

7. ABC operation check

Check that the boat moves to the set position.

Check it by measuring boat movement distance with a ruler.

Inspection timing: Every 6 months

8. Insulation test

Measure the insulation resistance between a power terminal and a protection terminal.

Inspection timing: Every year

Measure the insulation resistance between power terminals of AQF-100, GA-100 and, ABC and a protection ground terminal.

It should be 10M Ω or more at insulation resistance tester (DC 500V).

8-2. Keeping of System

8-2-1. For keeping the unit on a table

1. Take off power cables from outlets.
2. Take off gas lines from room lines.
3. Take off the cable connecting AQF-100 to a personal computer.
4. Put a cover on the whole of the unit.

8-2-2. For keeping the unit in a package

1. Take off power cables from outlets and the unit.
2. Take off gas lines from room lines and the unit.
3. Take off the cable connecting AQF-100 to a personal computer from the unit.
4. Take off ABC from AQF-100 and take off a ladle, a boat, and a guide tube.
5. Pull out a pyrolysis tube from AQF-100.
6. Put AQF-100 and parts in the package as before.
7. No direct sunlight
8. At low temperature and low humidity, no vibration
9. No strong electromagnetic field
10. No corrosive gas
11. No fire
12. Keep the package horizontally.

8-3. Taking out of GA-100 inside case

- (1) Turn off the power switch of GA-100. Remove the following lines.
(Refer to Illustration 8-3 and Table 8-3.)

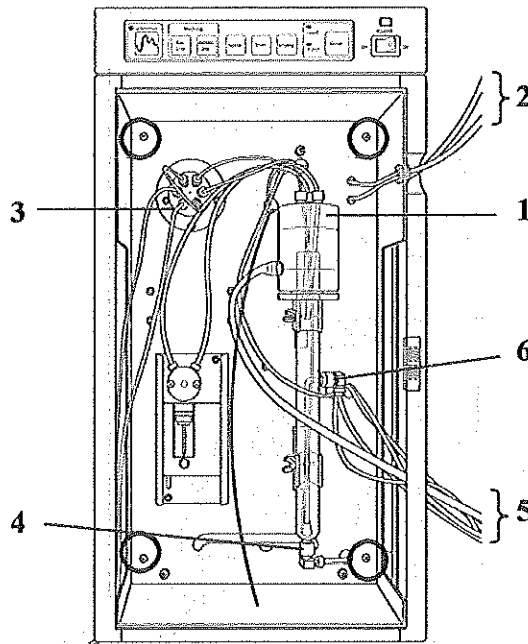


Illustration 8-3. GA-100 lines removal points

No.	Removal items
1	Overflow tank cap (with each line)
2	Absorption solvent and washing solution lines (2pcs) from each container
3	Connectors (2pcs) of an ion chromatography unit
4	Drain line connector
5	Drain lines (2pcs) and a vinyl tube
6	Connector from a pyrolysis tube

Table 8-3. GA-100 lines removal points

- (2) Remove 4 screws. Pull a handle and put out the inside case slowly.
(See Illustration 8-3.)

(3) The below illustration shows valve and pump positions. Table 8-4 shows the details.

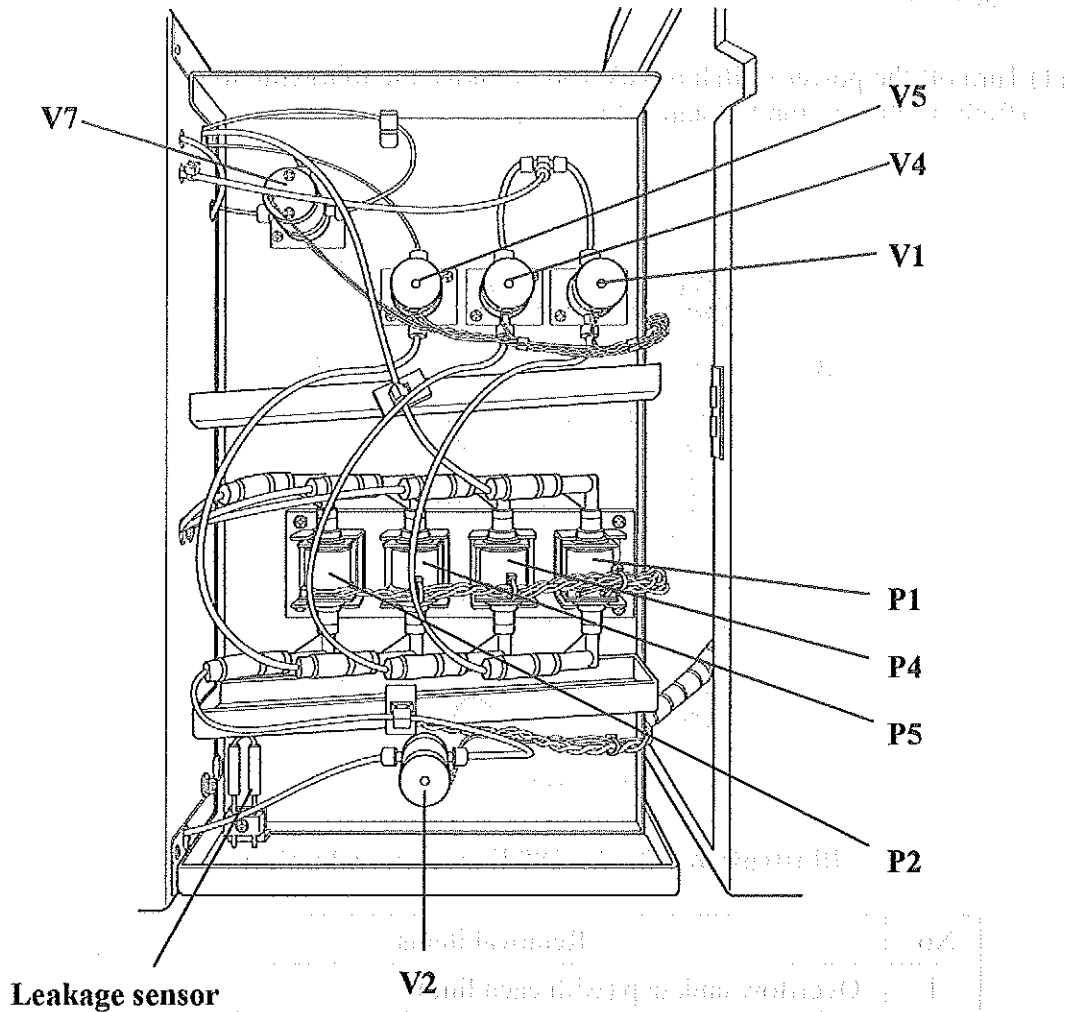


Illustration 8-4. GA-100 line control part and leakage sensor

Name	Mark	Function
Pump 1	P1	For washing an absorption tube
Pump 2	P2	For draining absorption solvent of an absorption tube
Pump 4	P4	For washing a gas line
Pump 5	P5	For sampling absorption solvent of an absorption tube and standard solution from an injection line
Valve 1	V1	Stop valve for washing an absorption tube
Valve 2	V2	Stop valve for draining absorption solvent of an absorption tube
Valve 4	V4	Stop valve for washing a gas line
Valve 5	V5	Stop valve for sampling absorption solvent and standard solution
Valve 7	V7	For changing the line from an absorption tube and a standard solution injection line

Table 8-4. Valve and pump functions

(4) Return the inside case and connect lines again.

Section 9: Specifications

Standard specifications

Measurement sample	Solid samples, nonaqueous liquid samples
Measurement substance	S, F, Cl, Br, I
Analysis method	Oxidative decomposition and gas absorption operation
Sample injection method	Automatic injection by a sample boat (Use ABC.)
Furnace temperature	Max. 1100°C (It can be set separately at sample injection part and combustion part.)
Sample volume	Solid samples : 1 ~ 100mg, liquid samples : 5 ~ 100μl
Combustion time	3 ~ 10 minutes every sample
Absorption part	Absorption tube : PYREX 20ml Sample injector : Ceramics sample injector Dispenser : Syringe pump, 5ml gastight syringe Drain pump : Diaphragm pump Line : Fluororesin tube, PEEK tube
Balance input	RS-232C
Gas	Oxygen gas (Purity : more than 99.7 %, 0.4 ± 0.1 MPa) Argon gas (Purity : more than 99.98%, 0.4 ± 0.1 MPa) Standard use amount is as follows. Argon gas : 200ml/min Oxygen gas : 400ml/min Supply gas pressure fluctuation : under $\pm 2\%$ in one day
Power	AQF-100 : AC 100V/115V/230V/240V, 50/60Hz, 1000VA GA-100 : AC 100V/115V/230V/240V, 50/60Hz, 30VA ABC : AC 100V/115V/230V/240V, 50/60Hz, 22VA WS-100 : AC 100V/115V/230V/240V, 50/60Hz, 20VA

Dimension and weight	AQF-100 : Approx. 560(W) × 400(D) × 435(H) mm (except a projection), Approx. 17kg GA-100 : Approx. 250(W) × 425(D) × 560(H) mm, Approx. 23kg ABC : Approx. 440(W) × 250(D) × 180(H) mm, Approx. 11kg WS-100 : Approx. 280(W) × 115(D) × 140(H) mm, (except a projection), Approx. 3kg
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Section 10: Consumables

10-1. AQF-100 Consumables

No.	Description	Part Number	Quantity	Remarks
1	Outer pyrolysis tube for TSV	TS8QPG	1 pc	
2	Inner pyrolysis tube (with a branch tube) for AQF-100	AQ1QPN	1 pc	
3	Spring for a pyrolysis tube	SX1QSP	4 pcs/set	
4	Quartz wool 10g	TNQWL	1 pc	
5	Gas purification filter (HYDRO-PURGE II)	TS6GPR	1 pc	
6	Clip, P18	TX017	2 pcs/set	
7	Fuse 3.15A	FU31MS	2 pcs/set	For 100/115V
8	Fuse 2A	FU02MS	2 pcs/set	For 230/240V
9	φ6/4 L-type joint	GA164E	1 pc	Elbow
10	Thermal fuse 93°C	FU93TR	1 pc	
11	φ4/2 Tube 10m	TX3RPP	1 pc	
12	φ4/2 Tube 2m	TNFT42	1 pc	
13	Insert	TNIS4	5 pcs	
14	Ferrule	TNFR4	5 pcs	

Table 10-1. AQF-100 consumables

10-2. GA-100 Consumables

No.	Description	Part Number	Quantity	Remarks
1	Absorption tube 10ml	GA1ABT	1 pc	
2	Absorption tube 20ml	GA1AB2	1 pc	
3	Overflow tank (with an O-ring)	GA1OFT	1 pc	
4	O-ring (for a P-18 overflow tank)	TNP18	5 pcs/set	
5	Ball joint with branch tubes (Quartz)	GA1BJQ	1 pc	
6	Syringe 5ml (Cavro)	GA1SB5	1 pc	
7	Nut	GA1NFY	5 pcs/set	Yellow, 1/8", for a selector valve, with a ferrule
8	PEEK tube	GA1PTB	1 pc	Inside diameter 0.25mm, blue 3m, φ 1/16" × 0.25

No.	Description	Part Number	Quantity	Remarks
9	φ6/3 Joint (30-6RU3-S)	TS3JLJ	1 pc	Straight
10	φ6/3 Joint (30-6RUE3-S)	GA163E	1 pc	Elbow
11	PTFE tube φ 3/2 1m	TN5FTS	1 pc	
12	Inline filter (with a filter)	GA1ILF	1 pc	
13	Filter for an inline filter	GA1IFP	5 pcs/set	
14	Nut (hexagonal)	GA1NHF	10 pcs/set	For a sample injector /an inline filter
15	Nut (with a ferrule) φ 3	GA1N03	5 pcs/set	For an overflow tank
16	Nut (#9734), for φ 1/16" (with a ferrule #9706)	GA1N16S	5 pcs/set	For an overflow tank

Table 10-2. GA-100 consumables

10-3. ABC Consumables

No.	Description	Part Number	Quantity	Remarks
1	Guide tube	TX3RTG	1 pc	
2	Ladle common use for ABC and ASC	TX3SCR	1 pc	
3	Magnet	TX2RTM	1 pc	
4	Sample boat (Quartz)	TX2SBT	5 pcs/set	
5	Septum holder	TX3SED	10 pcs	
6	Septum for a sample injection port	TN5SIS	100 pcs/set	
7	Packing for a sample inlet port	TXPKG	1 pc	
8	Glass plate for a sample inlet port	TX3BSI	1 pc	
9	Packing for a sample injection port	TN10SP	2 sets	
10	O-ring, P16	TX020	5 pcs/set	
11	O-ring holder	TX0103	1 pc	
12	Fuse 2A	FU02MS	2 pcs/set	
13	φ6/4 L-type joint (30-6RUE4-S)	GA164E	1 pc	
14	Ceramic sample boat	SXSMBS	100 pcs/bag	For inorganic measurement

Table 10-3. ABC consumables

10-4. WS-100 Consumables

No.	Description	Part Number	Quantity	Remarks
1	ϕ 6/3 Connector (30-6RUE3-S)	GA163E	1 pc	Connect it to the branch tube of an inner pyrolysis tube.
2	ϕ 3/2 PTFE tube 1m	TN5FTS	1 pc	
3	PTFE tube (blue) for Ar ϕ 4/2 3m	TN5FTA	1 pc	
4	Fuse 2A ϕ 5.2/20 midget-type slow-blow	FU02MS	2 pcs/set	

Table 10-4. WS-100 parts**10-5. Maintenance Consumables**

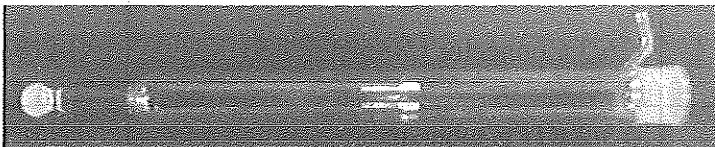
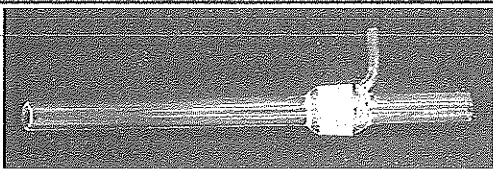

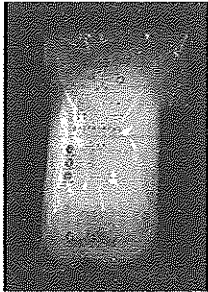
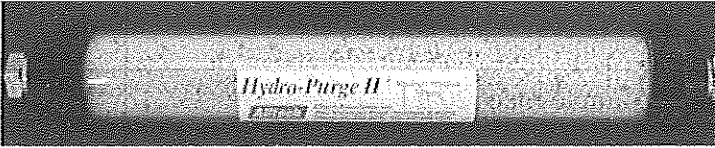

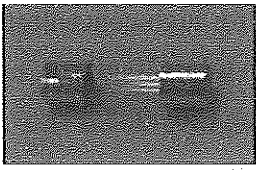
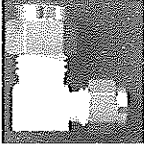
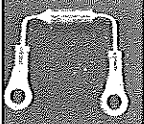
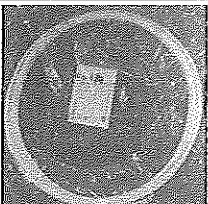
No.	Description	Part Number	Quantity	Remarks
1	Flow meter for gas leak check (1L/min)	TX3FFF	1 pc	
2	Quartz wool poker	TX2RTL	1 pc	

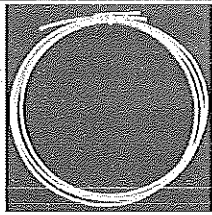

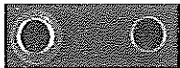
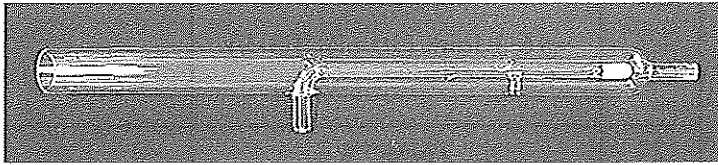
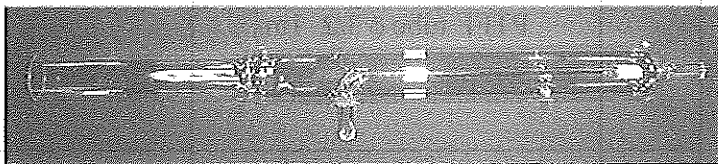
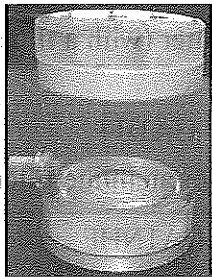

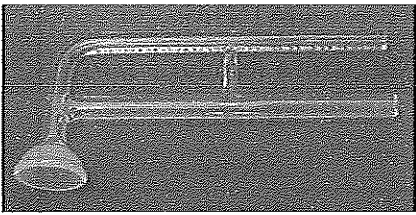
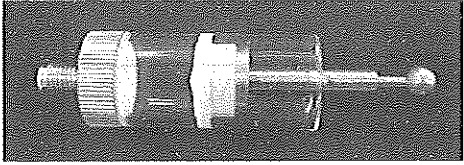

Table 10-5. Maintenance consumables**10-6. Option Consumables**

No.	Description	Part Number	Quantity	Remarks
1	Sample loop 5 μ l	GA1L05	1 pc	For a sample injector
2	Sample loop 20 μ l	GA1L20	1 pc	For a sample injector
3	Sample loop 50 μ l	GA1L50	1 pc	For a sample injector
4	Sample loop 100 μ l	GA1L11	1 pc	For a sample injector
5	Sample loop 200 μ l	GA1L21	1 pc	For a sample injector
6	Attachments for high concentration Absorption tube (20ml) Sample loop (20 μ l, 5 μ l) Trap column	AQF1PK	1 set	

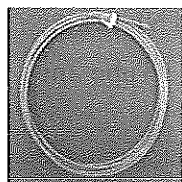
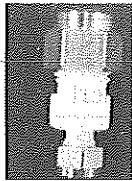
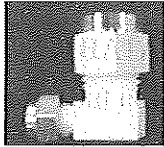
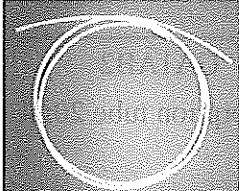
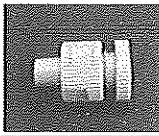
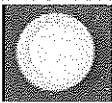
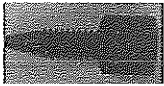

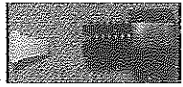
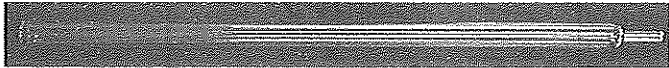
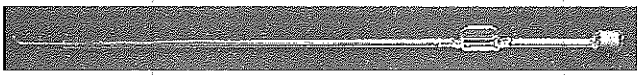
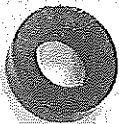
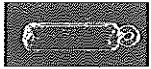

Table 10-6. Option consumables

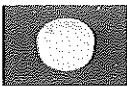
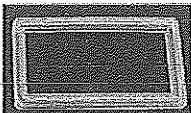
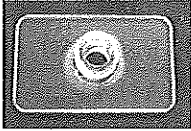


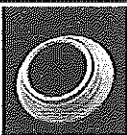
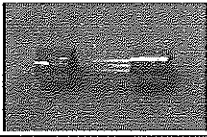

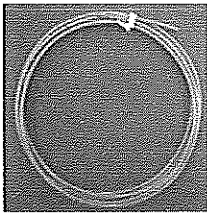
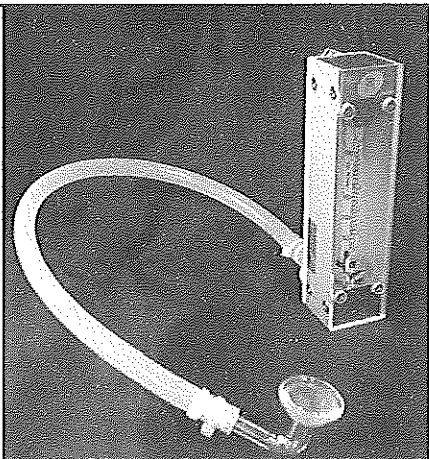
10-7. Parts Pictures

No.	Part numbers	Pictures
1	TS8QPG Outer pyrolysis tube for TSV	
2	AQ1QPN Inner pyrolysis tube (with a branch tube) for AQF-100	
3	SX1QSP Spring for a pyrolysis tube	
4	TNQWL Quartz wool 10g	
5	TS6GPR Gas purification filter (HYDRO-PURGE II)	
6	TX017 Clip, P18	
7	FU31MS Fuse 3.15A FU02MS Fuse 2A	
8	GA164E $\phi 6/4$ L-type joint	
9	FU93TR Thermal fuse 93°C	
10	TX3RPP $\phi 4/2$ Tube 10m	

No.	Part numbers	Pictures
11	TNFT42 φ4/2 Tube 2m	
12	TNIS4 Insert	
13	TNFR4 Ferrule	
14	GA1ABT Absorption tube 10ml	
15	GA1AB2 Absorption tube 20ml	
16	GA1OFT Overflow tank (with a O-ring)	
17	TNP18 O-ring (For P-18 overflow tank)	
18	GA1BJQ Ball joint with branch tubes (Quartz)	
19	GA1SB5 Syringe 5ml (Cavro)	
20	GA1NFY Nut yellow 1/8"(for a selector valve) with a ferrule	

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No.	Part numbers	Pictures
21	GA1PTB PEEK tube inside diameter 0.25mm blue 3m ϕ 1/16"/0.25	
22	TS3JLJ ϕ 6/3 Joint (30-6RU3-S)	
23	GA163E ϕ 6/3 Joint (30-6RUE3-S)	
24	TN5FTS PTFE tube ϕ 3/2 1m	
25	GA1ILF Inline filter (with a filter)	
26	GA1IFP Filter for an inline filter	
27	GA1NHF Nut (hexagonal)	
28	GA1N03 Nut (with a ferrule) ϕ 3	
29	GA1N16S Nut (with a ferrule) ϕ 1/16"	
30	TX3RTG Guide tube	
31	TX3SCR Ladle	
32	TX2RTM Magnet	
33	TX2SBT Sample boat (Quartz)	
34	TX3SED Septum holder	

No.	Part numbers	Pictures
35	TN5SIS Septum for a sample injection port	
36	TXPKG Packing for a sample inlet port	
37	TX3BSI Glass plate for a sample inlet port	
38	TN10SP Packing for a sample injection port	
39	TX020 O-ring, P16	
40	TX0103 O-ring holder	
41	FU02MS Fuse 2A	
42	SXSMBS Ceramic sample boat	
43	TN5FTA PTFE tube (blue) for Ar ϕ 4/2 3m	
44	TX3FFF Flow meter for gas leak check (1L/min)	

Section 10: Consumables

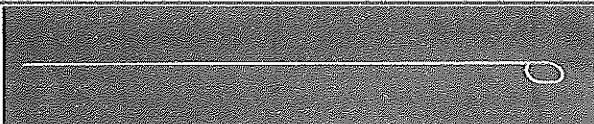
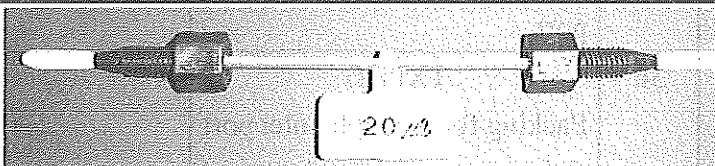
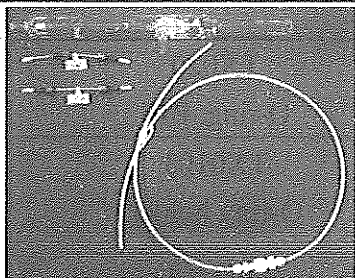
No.	Part numbers	Pictures
45	TX2RTL Quartz wool poker	
46	GA1L05 Sample loop 5 μ l	 20 μ l sample loop
47	GA1L20 Sample loop 20 μ l	
48	GA1L50 Sample loop 50 μ l	
49	GA1L11 Sample loop 100 μ l	
50	GA1L21 Sample loop 200 μ l	
51	AQF1PK Attachments for high concentration Absorption tube (20ml) Sample loop (20 μ l) Trap column	

Table 10-7. AQF-100 parts pictures