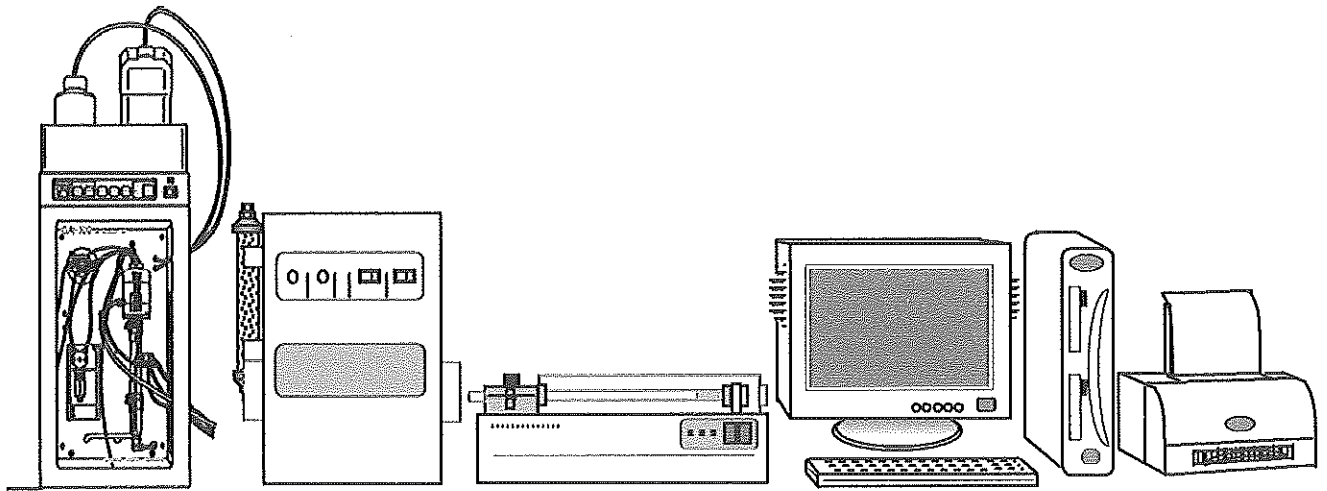


Control No. : ZAQQME-02

**Digest Manual**  
**For**  
**Automatic Quick Furnace**  
**MODEL AQF-100**



**MITSUBISHI CHEMICAL ANALYTECH**

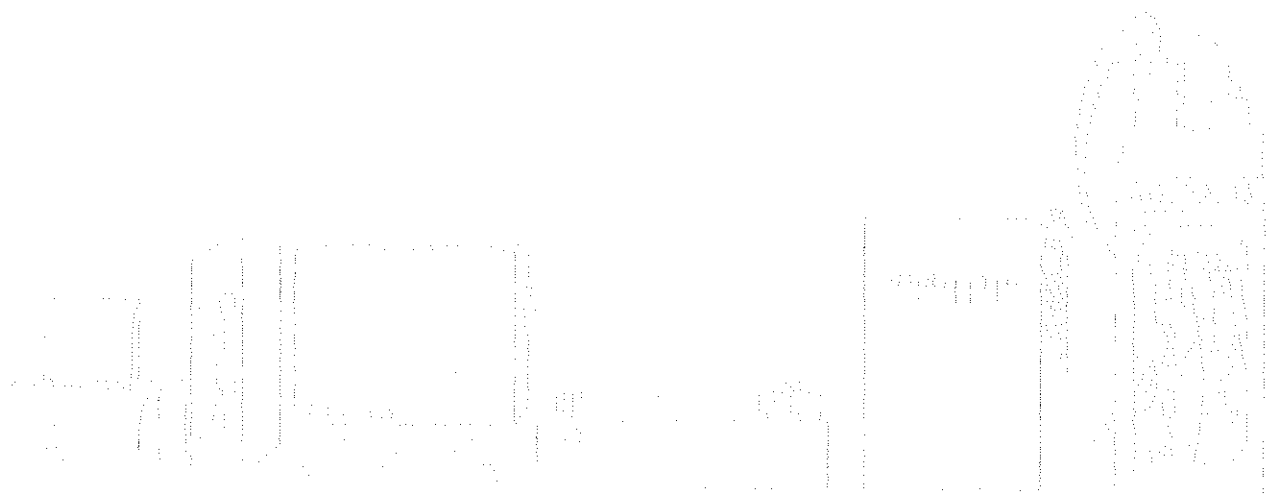
The following are the principal rivers of India, with their courses and basins.

INDIAN RIVERS

AND

THEIR COURSES AND BASINS

BY J. H. COLEMAN



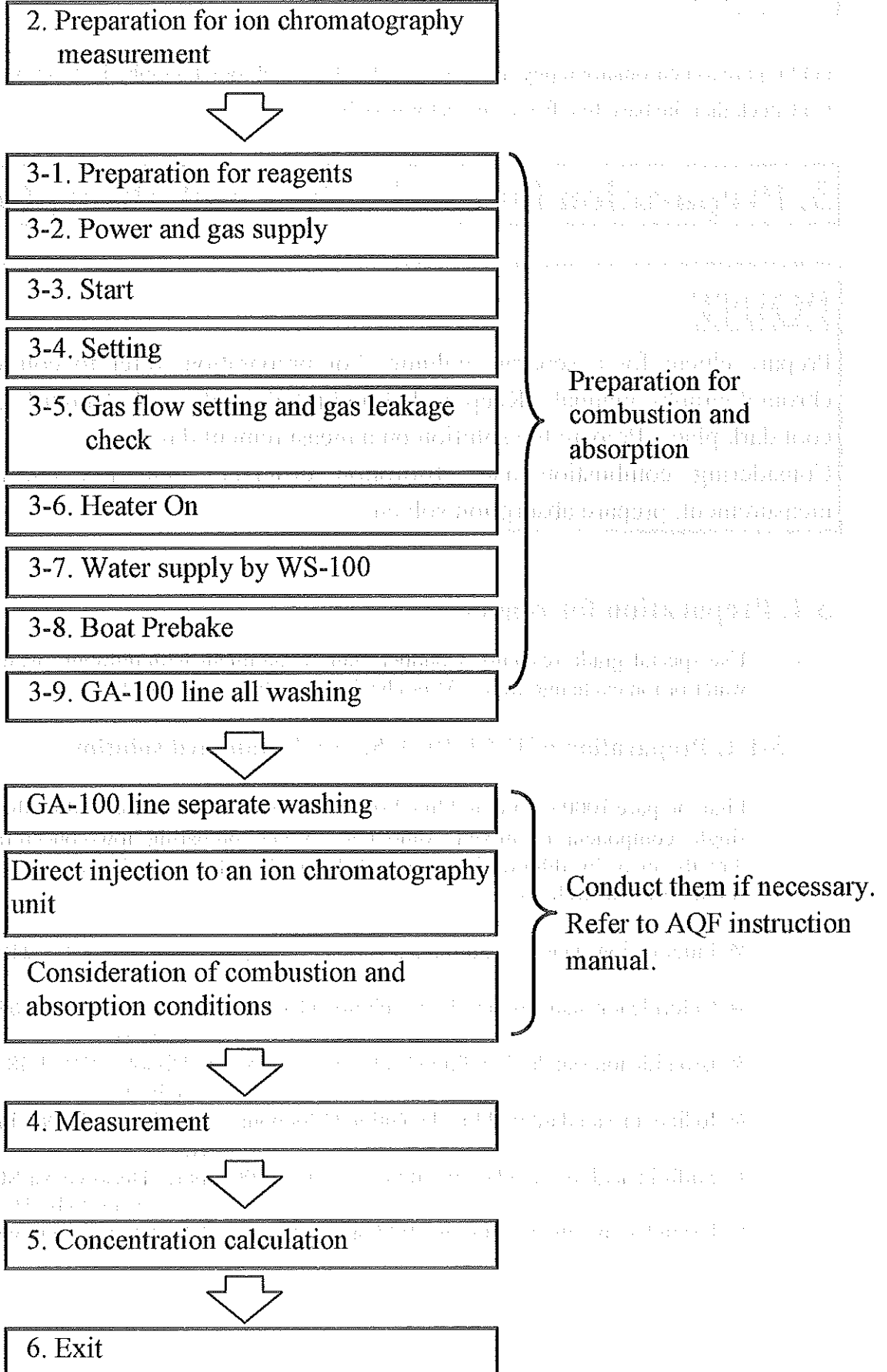
## Table of contents

1. Measurement flow .....	1
2. Preparation for ion chromatography measurement.....	2
3. Preparation for combustion and absorption .....	2
3-1. Preparation for reagents.....	2
3-1-1. Preparation of F, Cl, Br, I, S, and P standard solution.....	2
3-1-2. Preparation for absorption solvent.....	3
3-2. Power and gas supply .....	3
3-3. Start .....	3
3-4. Setting.....	3
3-4-1. System Setup.....	3
3-4-2. Analysis Parameters.....	4
3-4-3. Settings.....	4
3-5. Gas flow setting and gas leakage check .....	5
3-5-1. Gas flow setting .....	5
3-5-2. Gas leakage check.....	5
3-6. Heater On.....	6
3-7. Water supply by WS-100 .....	6
3-8. Boat Prebake.....	7
3-9. GA-100 line all washing.....	8
4. Measurement .....	9
4-1. Method edit.....	9
4-2. Connection of a pyrolysis tube outlet.....	12
4-3. Combustion.....	12
4-4. Method edit during measurement .....	14
5. Concentration calculation .....	15
5-1. Standard sample combustion method .....	15
5-2. Ion chromatography calibration method.....	17
6. Exit .....	18



# 1. Measurement flow

This section describes combustion and absorption and ion chromatography.



## 2. Preparation for ion chromatography measurement

- (1) Prepare ion chromatography measurement by the ion chromatography instruction manual.
- (2) Check that the base line from a detector is stable.

## 3. Preparation for combustion and absorption

### POINT

Prepare eluent for a separate column. For preparation, refer to column and ion chromatography manuals. Keep sealed undiluted solution of standard solution in a cool dark place. Prepare the solution on a measurement day.

Considering combustion and absorption efficiency and ion chromatography measurement, prepare absorption solvent.

### 3-1. Preparation for reagents

Use special grade reagents. Conduct blank experiment with ultrapure water or distilled water or ion exchange water. After checking use fitness, use water.

#### 3-1-1. Preparation of F, Cl, Br, I, S, and P standard solution

First, prepare 1000  $\mu$ g/ml undiluted solution of standard solution. Dilute the solution into single component or mixed component. When preparing low-concentration sample directly, error by dilution is generated. Examples of preparation of standard undiluted solutions are as follows.

- Fluoride ion standard undiluted solution (1000ppm) : Dissolve NaF 2.210g in water to be 1L.
- Chloride ion standard undiluted solution (1000ppm) : Dissolve NaCl 1.648g in water to be 1L.
- Bromide ion standard undiluted solution (1000ppm) : Dissolve KBr 1.489g in water to be 1L.
- Iodide ion standard undiluted solution (1000ppm) : Dissolve KI 1.308g in water to be 1L.
- Sulfuric acid ion standard undiluted solution (1000ppm) : Dissolve Na<sub>2</sub>SO<sub>4</sub> 1.479g in water to be 1L.
- P standard undiluted solution (1000ppm) : Dissolve KH<sub>2</sub>PO<sub>4</sub> 4.394g in water to be 1L.

### 3-1-2. Preparation for absorption solvent

Considering combustion and absorption efficiency and ion chromatography measurement, prepare absorption solvent. By adding absorption solution to eluant composition, water dip influence can be reduced.

- Absorption solvent for low-concentration samples (P 0.5ppm H<sub>2</sub>O<sub>2</sub> 30ppm)  
Pour water into 250 μl of 1000ppm P and 50 μl of 30% H<sub>2</sub>O<sub>2</sub> to be 500ml.
- Absorption solvent for high-concentration samples (P 25ppm H<sub>2</sub>O<sub>2</sub> 900ppm)  
Pour water into 25ml of 1000ppm P and 3ml of 30% H<sub>2</sub>O<sub>2</sub> to be 1000ml.

## 3-2. Power and gas supply

- (1) Open main valves of O<sub>2</sub> gas and Ar gas cylinders.
- (2) Adjust the second pressures of a cylinder and a STOP valves to  $0.4 \pm 0.1$ MPa with a reducing valve.
- (3) Supply power to the outlet on the table.

## 3-3. Start

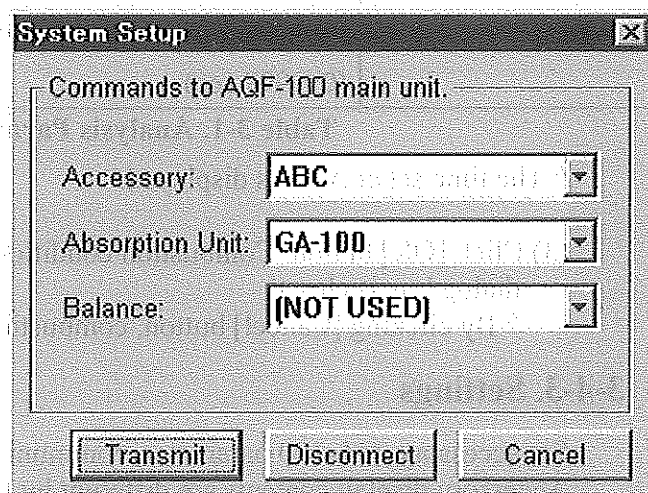
- (1) Turn on the following power switches.
  - AQF-100 power switch and heater switch (Front panel)  
The power switch of an automatic boat controller (ABC) is turned on automatically.
  - Cooler switch of ABC (Rear panel)
  - GA-100 power switch (Front panel)
  - Option power switch  
Turn on WS-100 power switch before measurement.
- (2) Turn on the power switches of a personal computer, a monitor, and a printer.
- (3) Start AQF-100 system program.

## 3-4. Setting

### 3-4-1. System Setup

Select an accessory, an absorption unit, and a balance in "System Setup" dialog box and start the communication to the main unit.

- (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) When a balance is connected, Click ▼ of “Balance” to select a balance maker.


Marker: A&D, Sartorius, Mettler, and Shimadzu

\* By connecting a balance and setting the maker at this dialog box when weighing sample with a balance, sampling weight can be transferred into “Sample Size” in a method file. When a balance is not connected, select “NOT USED”.

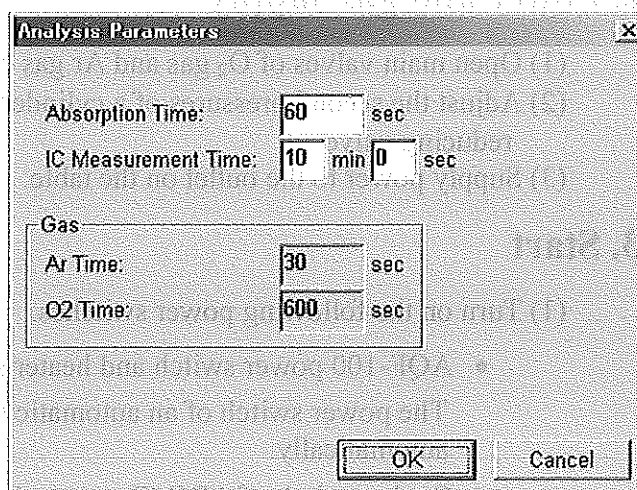
(5) Click [Transmit] button.

“ABC Home Moving” is displayed. It indicates the time for turning ABC to initial setting. “System Setup” contents are saved and “System Setup” dialog box is closed. A/QF communication to the system program starts.

### 3-4-2. Analysis Parameters

(1) Click  or click “System” and “Analysis Parameters”. “Analysis Parameters” dialog box is displayed.

(2) Set each item. (Refer to Table 3-1. Analysis Parameters setting item.)



Item	Setting contents
Absorption Time	Time of combustion gas absorption after ABC boat returns to the home position (0~9999 sec)
IC Measurement Time	Measure previously time from ion chromatography measurement start time to data processing end and input it. (0~9999 sec)
Ar Time*	Time for flowing argon gas into Ar/ O <sub>2</sub> line (inner pyrolysis tube) (0~999 sec)
O <sub>2</sub> Time*	Time for flowing oxygen gas into Ar/ O <sub>2</sub> line (inner pyrolysis tube) (0~999 sec)

Table 3-1. Analysis Parameters setting item

\* The time set at ABC is displayed.

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

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

### 3-4-3. Settings

Set accessory, GA-100 parameter, communication, and preference. Accessory display contents are different by the selection at “System Setup”. Refer to each instruction manual for details.

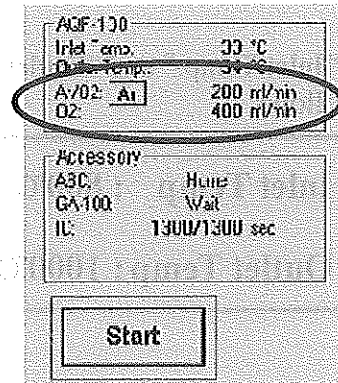


### 3-5. Gas flow setting and gas leakage check

Change gas at the system program side, adjust AQF-100 gas flow, and check gas leakage.

#### 3-5-1. Gas flow setting


- (1) Gas flow is displayed in AQF-100 frame of the main window.
- (2) Turn a gas flow adjustment knob of AQF-100 front to adjust gas flow.  
Adjust Ar/O<sub>2</sub> flow with "Ar".  
Adjust O<sub>2</sub> flow with "O<sub>2</sub>".  
Total argon flow of WS-100 and AQF-100 frame flows into a pyrolysis tube.



#### 3-5-2. Gas leakage check

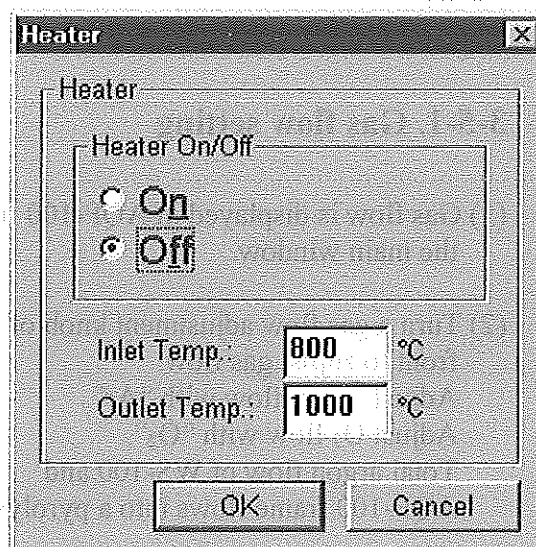
- (1) Replace a ball joint (with branch tubes) of a pyrolysis tube outlet with one of a flow meter (option) for gas leakage check.
- (2) Check the flow meter value is within  $600 \pm 60$  ml/min of O<sub>2</sub> scale. (Ar 200ml/min, O<sub>2</sub> 400ml/min)
- (3) If the value is under the set flow, 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.

### 3-6. Heater On

(1) Click  or click "System" and "Heater". "Heater" dialog box is displayed.

(2) Set "Inlet Temp." and "Outlet Temp." as follows.

**Inlet Temp : 800°C**  
**Outlet Temp : 1000°C**



(3) Click "On".

(4) Click [OK] button.

Temperature starts to rise.

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

### 3-7. Water supply by WS-100

Argon gas including ultrapure water is poured into a pyrolysis tube with WS-100.


(1) Turn a flow adjustment knob to set argon gas flow to 150ml.  
Total argon gas flow of WS-100 and AQF-100 frame pours into a pyrolysis tube.

(2) After temperature reaches a setting value, turn on the power switch.

(3) Rotate WATER of a water supply dial.

For XS-100 series, set the dial to "4~6". For CIC-100 series, set the dial to "1".

### 3-8. Boat Prebake

(1) Click  or click “Run” and “Boat Prebake” dialog box is displayed.

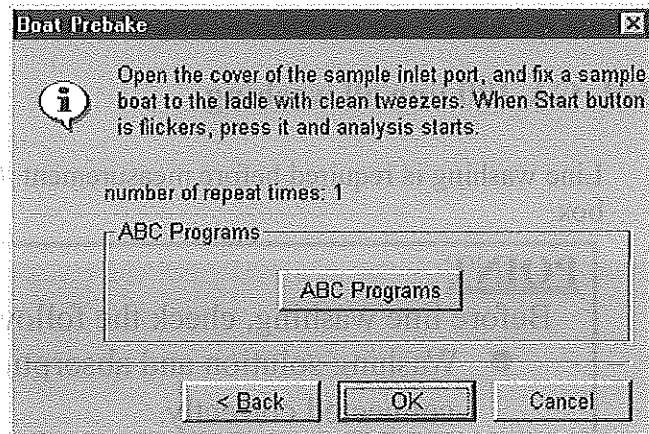
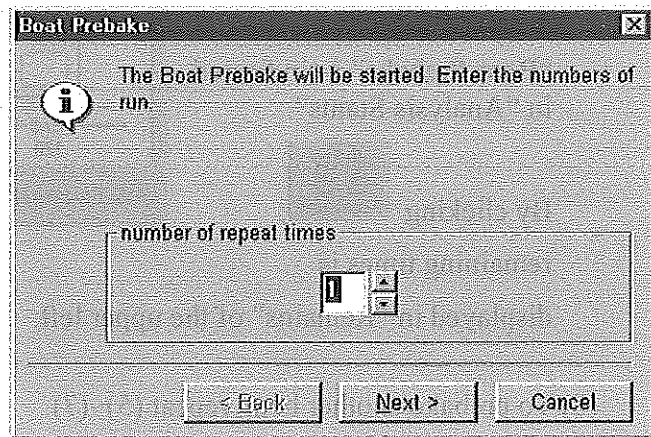
(2) Input “number of repeat times”. (Usually 2~5 times )

(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.

\* When handling sample boats, use tweezers to prevent contamination. For more than 2 sample boats prebaking, sample boats should be kept in a glass petri dish until measurement.


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

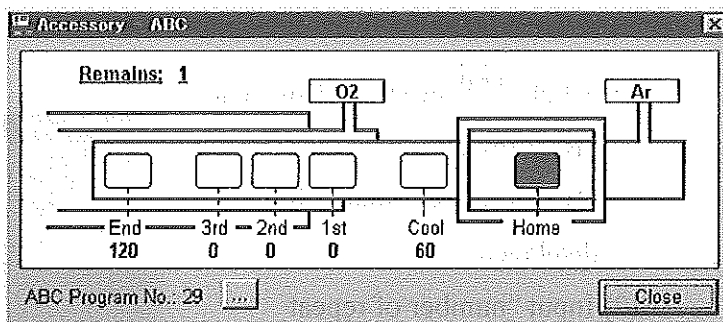
(7) 1 prebaking requires about

4~5 minutes and setting times are repeated automatically.

“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.

### 3-9. GA-100 line all washing

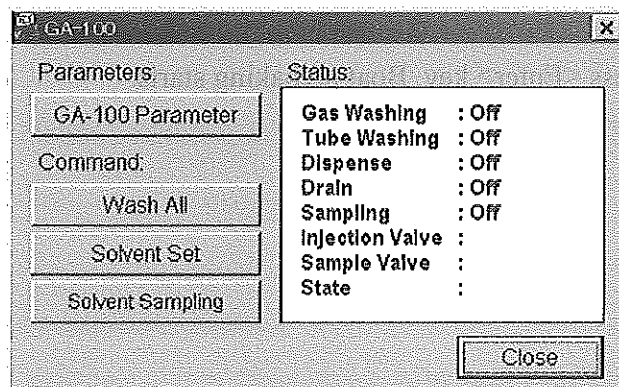
Line washing is required before measurement. Click [Wash All] button of a GA-100 dialog box.

#### **POINT**

Before line washing, check the following points.

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

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




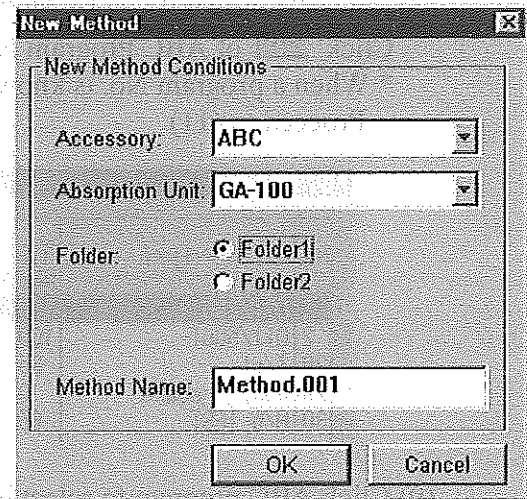
(2) Click [Wash All] button. Each GA-100 gas line is washed automatically.

# 4. Measurement

## 4-1. Method edit

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

- (1) Click  or click “File” and “New Method”. “New Method” dialog box is displayed.
- (2) Check the contents of “Accessory” set at 3-4-1. System Setup. To change them, click ▼ of the right to select “Accessory”. For standard composition, select “ABC”.
- (3) Check “Absorption Unit” set at 3-4-1. System Setup. To change it, click ▼ of the right to select “Absorption Unit”. For standard composition, select it from “GA-100” or “NOT USED”.
- (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)
- (6) Click [OK] button.  
The method edit dialog box is displayed.



- (7) Set 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 4-1. Sample measurement input item

When measuring liquid samples

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

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

Proceed to (11).

No.	Program	ABC Parameter							Analysis			
		1st Pos. Time	2nd Pos. Time	3rd Pos. Time	End Time	Cool Time	Boat Speed	Ar Time	O2 Time			
1	Oil/20ul	100	0	120	30	130	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

(9) Click ABC program to select it.

(10) Click [OK] button. The method edit dialog box is displayed. The selected Program No. is displayed in "ABC Program No."

Absorption Unit: GA-100	ABC			
ABC Program No.: 1	[...]			
Sample ID: Oil	[v]			
Sample Size: 15.40	mg [v] Balance			
[Add]	[Accumulate] [Insert]			
[Delete All]	[Delete]			
Tube	Sample ID	Sample Size	Sample Size Unit	ABC
1	Oil	15.40	mg	1

(11) Click [Add] button by combustion times. The same setting contents are added into the below list by clicked times.

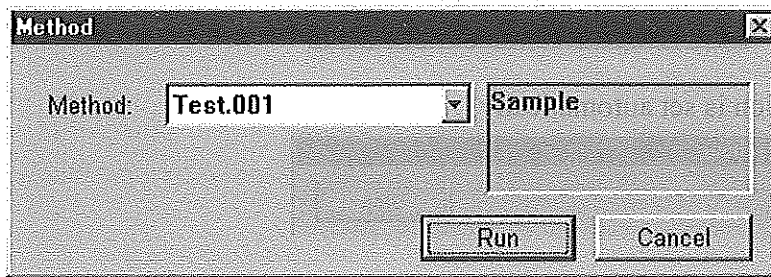
Tube	Sample ID	Sample Size	Sample Size Unit	ABC
1	Oil	15.40	mg	1
2	Oil	15.70	mg	1
3	Pellet	20.20	mg	2
4	Pellet	19.80	mg	2

(12) Like other sample settings, add them into the list by (7)~ (11).

- (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

- (14) To start measurement when method edit is completed, click [Run Method] button. "Method" dialog box is displayed.



- (15) Click [Run] button.

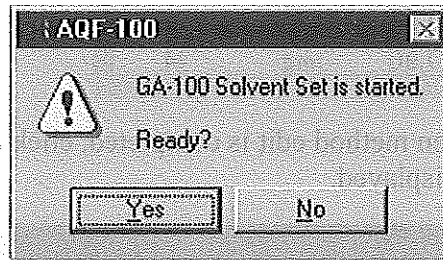


## 4-2. Connection of a pyrolysis tube outlet

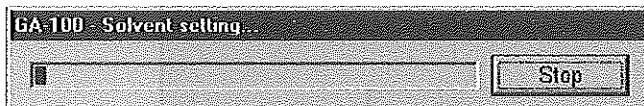
Set a ball joint (with branch tubes) to the outlet of a pyrolysis tube and fix it to the pyrolysis tube with a clamp for a ball joint.

## 4-3. Combustion

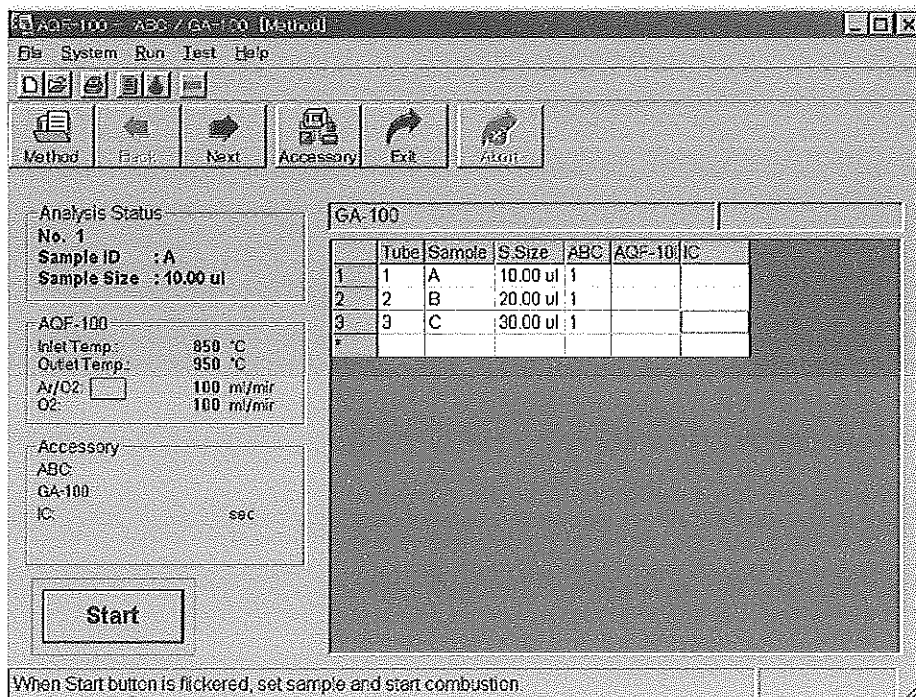
- (1) Set absorption solvent to an absorption tube. When the following message is displayed, click [Yes] button. To set absorption solvent after a while, click [No] button.  
To run combustion again, click "Run" and "GA-100 Start". The following window is displayed.



- (2) Absorption solution is set automatically.



- (3) Measurement contents set at methods are displayed in "Analysis Status" from No.1.  
Check this indication and prepare samples.



- (4) Check that [Start] button is flickering and set a sample.



For liquid samples

- ① Set a prebaked sample boat to the sample introduction box.  
(After the second time, use the previous boat.)
- ② Take a sample into a microsyringe. Inject the sample into the sample boat from the injection port.

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

- ① Place a prebaked boat on a balance and weigh solid samples.
- ② Input sample weight into "Sample Size".
- ③ Put a sample boat into the 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.

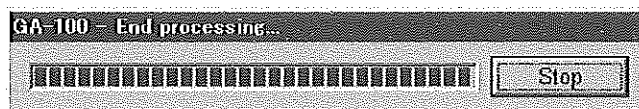
(6) The following contents are displayed in "AQF-100" and "IC" of the list in [Progress] frame.

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	

(7) For measurement on and after No.2, repeat (3)~(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  or click "Run", "Operation", and "Exit Run". "GA-100-End processing" is displayed.




End washing is run automatically.

(10) Run "Print Method" and all of "Print" in "File" if necessary.

## 4-4. Method edit during measurement

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

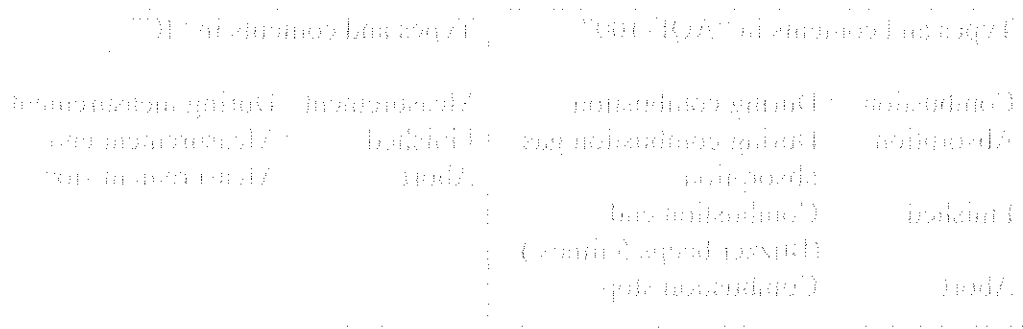


- (1) Click  or click “Run”, “Operation”, and “Method”.

The method edit dialog box is displayed.

- (2) Add or delete methods like method edit.

- (3) Click [Run Method] button. The main window returns.



## 5. Concentration calculation

To calculate the concentration of components in samples with AQF-100, the following two methods are available.

### <Standard sample combustion method>

Combust and absorb standard samples and samples. Gain concentration from peak area of each component or internal standard components.

1. Result can be gained by correcting the recovery from combustion to absorption.
2. When using ASC-150L, all standard solutions and unknown samples can be measured automatically.
3. AQF-100 recovery can't be checked.

### <Ion chromatography calibration method>

By injecting directly the standard solution of components and internal standard components, prepare ion chromatography calibration. Combust and absorb samples or gain component concentration from calibration.

1. By combusting standard samples, recovery can be checked.
2. It is unnecessary to combust standard solution. (when liquid samples are difficult to combust with a solid sampler)
3. It is necessary to prepare ion chromatography calibration manually.

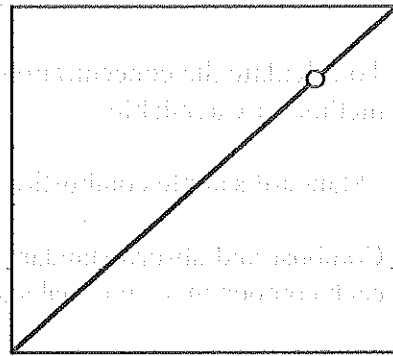
### 5-1. Standard sample combustion method

- (1) Combust prepared standard solution with organic solvent and measure the solution by ion chromatography.
- (2) Gain the area of components and internal standard components from ion chromatography peak.
- (3) Calculate the area ratio of components and internal standard components ( $A_{std}/I_{std}$ ).
- (4) Gain the relation between component volume and area ratio. (calibration)
- (5) Measure unknown samples and gain the area ratio of components and internal standard components ( $A_{samp}/I_{samp}$ ).
- (6) Gain component volume from calibration.
- (7) Calculate component concentration ( $\mu\text{ g/ml}$  or  $\mu\text{ g/g}$ ) in samples by the following method.

<One point calibration>

One example of one point calibration curve

$$\text{Concentration} = M_{\text{std}} \times \frac{A_{\text{samp}}/I_{\text{samp}}}{A_{\text{std}}/I_{\text{std}}} \times \frac{1}{SS}$$



Component volume

Volume of components in combusted standard solution:  $M_{\text{std}}$  (ng)

Area of components at standard solution measurement:  $A_{\text{std}}$

Area of internal standard components at standard solution measurement:  $I_{\text{std}}$

Unknown sample volume:  $SS$  ( $\mu$ l or mg)

Area of components at unknown sample measurement:  $A_{\text{samp}}$

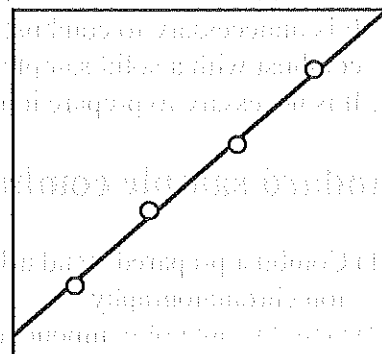
Area of internal standard components at unknown sample measurement:  $I_{\text{samp}}$

<Plural points calibration>

One example of plural points calibration curve

Prepare calibration from  $A_{\text{std}}/I_{\text{std}}$  and  $M_{\text{std}}$  of each calibration solution.

Gain component volume ( $\mu$ g) in unknown samples from  $A_{\text{samp}}/I_{\text{samp}}$  of unknown samples.



Component concentration

$$\text{Concentration} = \text{Value gained from calibration} \times \frac{1}{SS}$$

## 5-2. Ion chromatography calibration method

- (1) Inject directly standard solution of changed components and internal standard components and measure the solution by ion chromatography.
- (2) Gain the area of components and internal standard components.
- (3) Measure unknown samples and gain ion chromatography peak area.
- (4) Gain each component in absorption solvent from calibration.
- (5) Calculate component concentration in samples by the following method.

$$\text{Concentration} = C_c \times A \times \frac{C_o}{C_i} \times \frac{1}{SS}$$

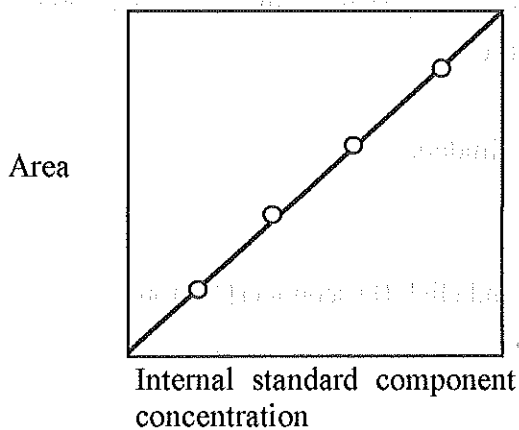
$C_c$  : Component concentration

$A$  : Absorption solvent volume

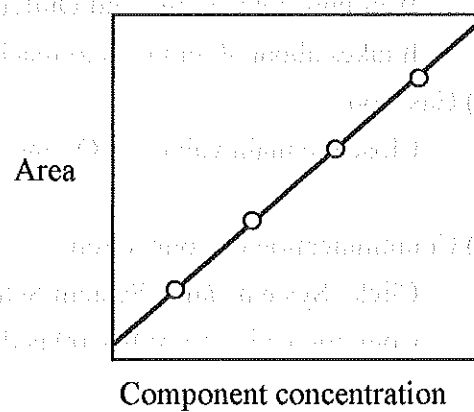
$C_o$  : Original internal standard component concentration

$C_i$  : Internal standard component measurement concentration

One example of internal standard component calibration



One example of component calibration






## 6. Exit

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

(1) WS-100 stop

Turn off the power switch.

(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).

(3) 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.

It takes about 30 minutes to reach 500°C.

(4) Gas stop

Close the main valves of O<sub>2</sub> and Ar cylinders.

(5) Communication disconnection

Click "System" and "System Setup" and click [Disconnect] button.

Communication to AQF-100 is disconnected.

(6) Unit stop

Turn off the following power switches.

- ① GA-100 power switch
- ② Cooling unit power switch of ABC rear
- ③ Option power switch

(7) System program exit

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

(8) Personal computer exit

- ① Click [Start] button of a taskbar.
- ② Click “Shut Down”. Windows exit page is indicated. Shut down Windows.
- ③ Check “Shut down the computer?” is selected. Click [Yes] button.  
The computer power is off.
- ④ Turn off the power switches of a printer and a monitor.

(9) AQF-100 stop

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

(1)  $\int_{-\infty}^{\infty} f(x) \delta(x-a) dx = f(a)$   
 (2)  $\int_{-\infty}^{\infty} f(x) \delta(x) dx = f(0)$   
 (3)  $\int_{-\infty}^{\infty} f(x) \delta(x-a) dx = f(a)$   
 (4)  $\int_{-\infty}^{\infty} f(x) \delta(x) dx = f(0)$   
 (5)  $\int_{-\infty}^{\infty} f(x) \delta(x-a) dx = f(a)$   
 (6)  $\int_{-\infty}^{\infty} f(x) \delta(x) dx = f(0)$   
 (7)  $\int_{-\infty}^{\infty} f(x) \delta(x-a) dx = f(a)$   
 (8)  $\int_{-\infty}^{\infty} f(x) \delta(x) dx = f(0)$   
 (9)  $\int_{-\infty}^{\infty} f(x) \delta(x-a) dx = f(a)$   
 (10)  $\int_{-\infty}^{\infty} f(x) \delta(x) dx = f(0)$

(11)  $\int_{-\infty}^{\infty} f(x) \delta(x-a) dx = f(a)$   
 (12)  $\int_{-\infty}^{\infty} f(x) \delta(x) dx = f(0)$   
 (13)  $\int_{-\infty}^{\infty} f(x) \delta(x-a) dx = f(a)$   
 (14)  $\int_{-\infty}^{\infty} f(x) \delta(x) dx = f(0)$   
 (15)  $\int_{-\infty}^{\infty} f(x) \delta(x-a) dx = f(a)$   
 (16)  $\int_{-\infty}^{\infty} f(x) \delta(x) dx = f(0)$   
 (17)  $\int_{-\infty}^{\infty} f(x) \delta(x-a) dx = f(a)$   
 (18)  $\int_{-\infty}^{\infty} f(x) \delta(x) dx = f(0)$   
 (19)  $\int_{-\infty}^{\infty} f(x) \delta(x-a) dx = f(a)$   
 (20)  $\int_{-\infty}^{\infty} f(x) \delta(x) dx = f(0)$