4. CONSTRUCTION

The front door of the equipment can be opened. LCD, keyboard and sample pan are built in the front door.

The front door need not be opened during measurement. At the time of maintenance and other services, the equipment interior can be accessed easily if front door is opened. Top cover is removed easily. Side panels and bottom panel can be also dismounted by removing screws so that the equipment is very easy to inspect or repair.

Side panels and bottom panel are expected to be opened only by our service staff for repair. If they must be opened by the user, be sure to turn off power switch beforehand.

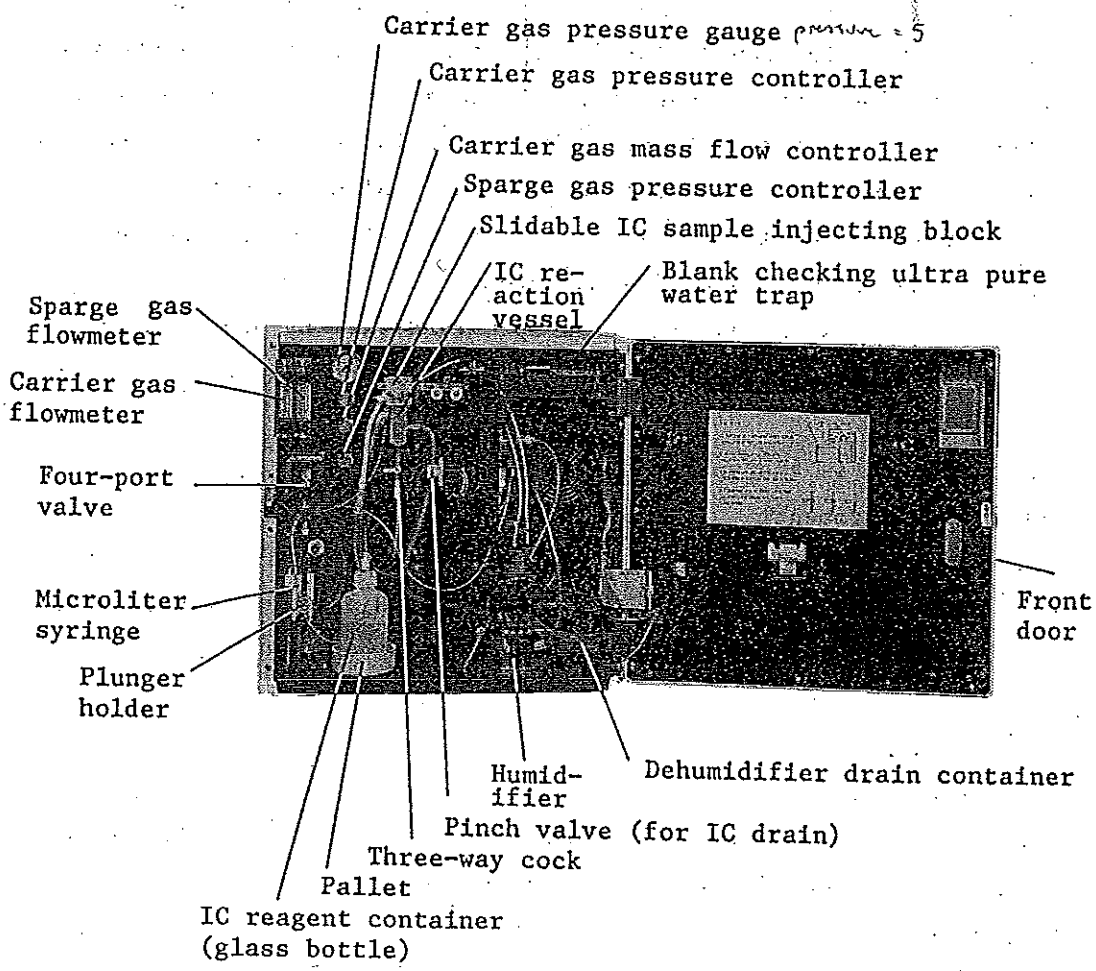


Fig. 4.1 Front View of the TOC-5000 Interior
(Viewed with the Front Door Open)

4.1 Gas Control Section

This section opens and closes the valves of carrier gas and sparge gas (sparging gas for NPOC Measurement) and controls their flow rate.

High purity air supplied through the gas inlet is divided into two flows and used in these gases. A filter is built in the gas inlet connection port.

Supply of carrier gas and that of sparge gas are controlled by respective solenoid valves. When equipment power is turned off, the solenoid valves are closed automatically so that gas supply is interrupted.

The carrier gas pressure is controlled to be constant by pressure controller and pressure gauge before the flow rate is adjusted to a specified value (150 mℓ/min.) by mass flow controller and flowmeter. Mass flow controller is designed to operate to maintain a constant flow rate irrespective of change in the outlet pressure. Therefore, if the resistance in TC combustion tube changes, the carrier flow rate is maintained constant. Since peak area varies in inverse proportion to carrier gas flow rate, do not change carrier gas flow rate during measurement.

Sparge gas flow is adjusted to a specified rate by pressure controller, capillary connected to the pressure regulator outlet and flow meter.

Minor change in the sparge gas flow rate does not give influence on the measurement.

Carrier gas is moistened as it passes over the water in humidifier. Accurate measurement is ensured by the humidifier particularly in high sensitivity measurement.

4.2 TC Combustion Section

TC furnace temperature is controlled at 680°C by CPU. When equipment is operated with power supply of other than 100 V, voltage supplied to the electric furnace heater is controlled accordingly. When TC furnace temperature rises to about 780°C, heater current is cut off automatically by overheat prevention circuit. Once actuated, this circuit is kept operating and it remains ON even after temperature drops, unless power switch is turned off. This

circuit is designed to continue lowering temperature even when temperature-detecting C·A thermostat (sheath type) has broken.
TC furnace is a power saving type, made of high performance adiabatic material molded in one integral body for improved heat insulation (maximum rating: about 330 W). It incorporated KANTHAL heater inside. Due to the small watt density, the heater has longer life. However, if heater wire is broken, renew the entire electric furnace.
Set TC combustion tube made of transparent quartz glass in the center of TC furnace.
TC furnace temperature is displayed on the LCD screen (monitor screen). The temperature is fixed at 680°C before shipment and this setting cannot be changed by the user.

4.3 IC Reaction Section

Plastic IC reaction vessel is equipped on its bottom with a plate having numerous pores for passing carrier gas. Water acidified with IC reagent is put in the vessel. Drain pipe is connected to the side of the IC reaction vessel so that excess water is discharged through pinch valve and this pipe before sample is injected.
Drain generated in TC combustion tube flows together with carrier gas into the IC reaction vessel. When sample containing acids or salts composed of mineral acid components (such as magnesium chloride and ammonium sulfide) is injected into TC combustion tube, drain is made acidic by generated acid components. The acidic drain is used as IC solution for IC reaction.

4.4 Section for Treating Gas Generated through Combustion and Reaction

This section is composed of dehumidifier, dust-eliminating membrane filter and halogen scrubber for eliminating halogen gas.
Dehumidifier cools carrier gas flowing from IC reaction vessel down to about 1°C to maintain the moisture content of the gas low and constant.

Halogen scrubber absorbs and eliminates chloric gas generated by chloric acids and salts contained in sample, thus protecting sample cell inner surface. Since halogen scrubber turns blue as it absorbs chlorine, it is easy to know its replacement time.

4.5 Infrared Gas Analyzer (NDIR)

While monatomic molecules such as N_2, O_2 and H_2 do not absorb infrared radiation, polyatomic molecules such as CO_2 and CH_4 absorb infrared radiation of different wavelengths depending upon the bonding condition and kind of the atoms constituting each molecule. For instance, N_2 and O_2 in the carrier gas used in the TOC-5000 do not absorb infrared radiation but CO_2 in the gas does at 4.3 μ.

The amount of rays absorbed is proportional to the density of the gas according to the Lambert-Beer's Law. Therefore, the gas density can be determined by measuring the amount of rays absorbed. The analyzer measures absorption using the non-dispersive method. That is, infrared radiation is not spectrally dispersed. Instead, intermittent two parallel optical beams are measured by a selective detector E. Sample gas is led to two sample cells connected in series, M_1 and M_2, located in the measurement beam path. Reference cell M_3 in the second beam path contains nitrogen gas which does not absorb infrared radiation. The short sample cell M_2 is used as an independent sample cell when the optional solid sample module SSM-5000 is used. Radiation emitted by light source element S_1 is divided into two beams which pass through the sample cell and the reference cell, respectively, into the detector. The detector is filled with sample gas component (CO_2) to a specified concentration and divided into two chambers by a metal diaphragm. The incident radiation is absorbed selectively only in the specific absorption bands of the CO_2 gas in the detector. The absorbed energy is instantaneously transformed to thermal energy through molecular collision.

Due to the selectivity of the detector, absorbed energy variation depending on the CO_2 concentration in the sample cell alone causes temperature or pressure difference between the two detector chambers. Metal diaphragm is deformed by the pressure difference, thus changing the capacitance of a capacitor made of diaphragm E_1 and opposing electrode E_2. Since two beams are interrupted simul-

taneously at a specified frequency by a rotating sector S_3, pressure and capacitance variation is also periodic. The diaphragm capacitor is connected through high-value resistor R to DC power source G. Capacitance fluctuation results in periodic charging and discharging current which in turn generates AC voltage in mV range through the high-value resistor. The voltage is amplified, rectified, changed in the range and applied to A/D converter circuit.

The optical system of NDIR consists of light source S, measuring cell M, detector E and preamplifier connected to each other by screws in one unit which is fixed on an optical base (plate) and mounted in the upper part at the back of the equipment.

Rubber basket is provided in the joints between light source and measuring cell and between measuring cell and detector to ensure airtightness. The space between light source and measuring cell is connected via piping with the space between measuring cell and detector. Purge gas inlet fitting is provided in the latter space so as to introduce purge gas from the exterior into the space. Carrier gas discharged from sample cell is purified in CO_2 absorber and flows through this fitting into the space as purge gas. After purging each space, purge gas is exhausted to the exterior from through-hole for the rotating sector-driving shaft of the light source. Purging is necessary to prevent NDIR indication to fluctuate with CO_2 concentration variation which would occur if these spaces were exposed to atmosphere.

Measuring cell is composed of sample cell (at the back of equipment) and reference cell. The cell inside walls are gold-plated. If foreign substance enters the cell, remove it by passing clean air or N_2 gas through the cell. When it is necessary to wash the cell interior for some reason, wash it in clean ethanol or methanol gently with care, discharge the cleaning solution from the cell and dry it thoroughly in air.

Detector contains two light-receiving chambers each having a window on the side facing to the measuring cell. Optical zero adjuster E_3 is provided in front of either of the windows so as to balance the light amount between two light paths. The adjuster E_3 is inserted in the light path with larger light amount and can be adjusted from outside the equipment with a minus driver through the adjustment hole in the top panel. Rotation direction for adjustment is

indicated by arrow on the label on the adjustment hole. Rotation along arrow decreases light amount in sample cell (or increases light amount in reference cell), causing analog signal from NDIR to increase toward plus side.

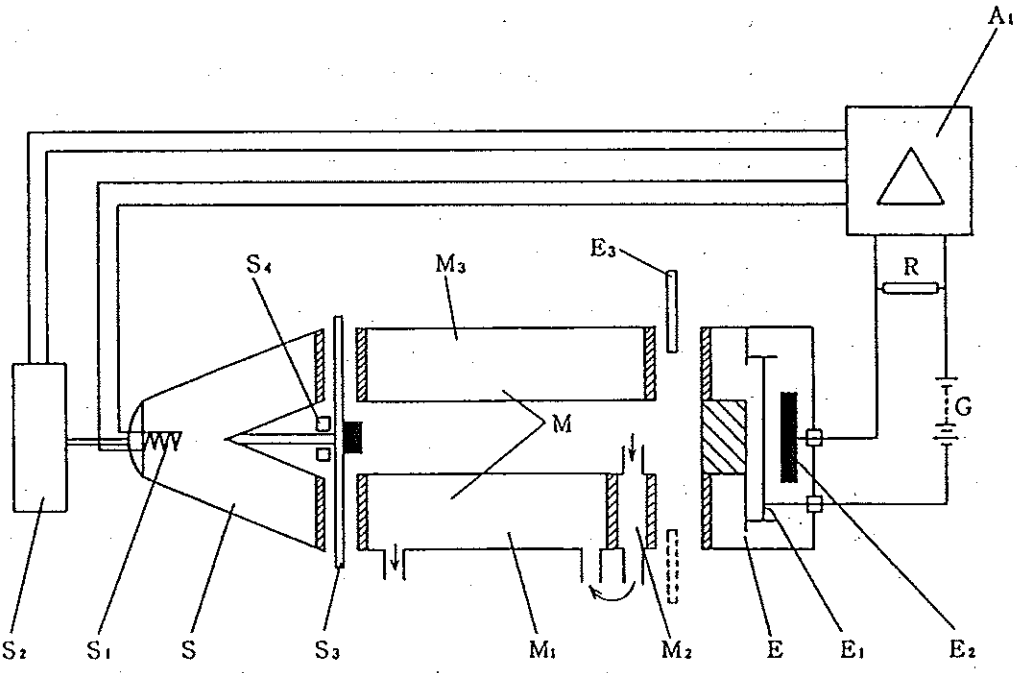
Shielding cable from preamplifier is connected via a connector to mother PC board.

Absolutely do not connect or disconnect the cable with power ON.

4.6 Electrical Section

Electrical section is located at the back of equipment. It consists of power transformer, switching power sources (two), main PC board and power supply PC board, etc.

Power switch is equipped with thermal breaker so that it is turned off automatically by overcurrent. Equipment can be powered by turning on the switch again. If the switch is turned off frequently, locate the cause.



E	Detector	M_1	Sample cell (long cell)	S_1	Light source element
E_1	Diaphragm	M_2	Sample cell (short cell)	S_2	Motor
E_2	Opposing electrode	M_3	Reference cell	S_3	Rotating sector
E_3	Optical zero adjuster	R	High-value resistor	S_4	Bearing
G	DC power source	S	Light source	A_1	Main amplifier
M	Measuring cell				

Fig. 4.2 Non-dispersive Infrared Gas Analyzer

4.7 Sample Injection Section

This section is composed of sample injector and TC and IC sample injection ports, slide type. The sample injector is of syringe pump type combining motor-driven four-port valve with microliter syringe whose plunger is operated by pulse motor. The common port of four-port valve is connected at all time with syringe. The common port is controlled by CPU to be connected selectively with sampling port for sample sucking into syringe, TC injection port for sample injection into TC combustion tube, IC injection port for sample injection into IC reaction vessel, or auxiliary (AUX) port for sucking blank checking pure water. Plunger stroke is controlled by steps of about 0.4% of full stroke. Piston movement speed of plunger is controlled to be most suitable for syringe size. Normally, microliter syringe of 250 μℓ capacity is mounted. It can be replaced easily with 500 μℓ, 1,000 μℓ or 2,500 μℓ type. (500 μℓ and 1,000 μℓ syringes are optional accessories.)

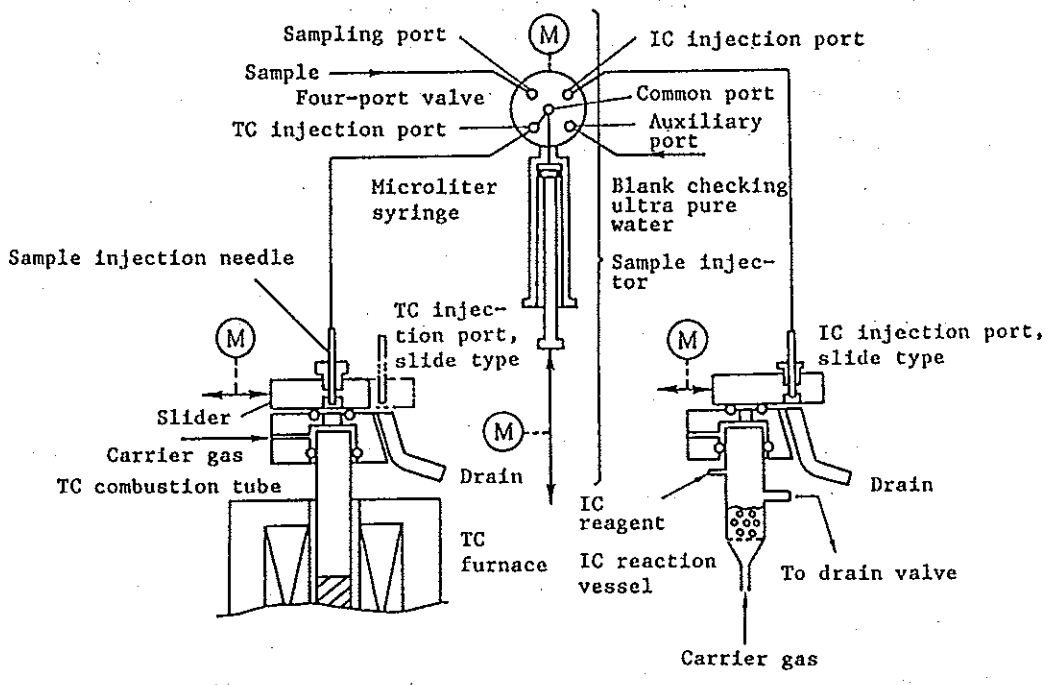


Fig. 4.3 Construction of Sample Injection Section

In the slidable sample injecting block, fluorocarbon polymers-made slider moves between the upper opening of TC combustion tube (or IC reaction vessel) and drain port, actuated by motor. Sample injection needle is built in the slider.

Specific operation for sample injection into TC combustion tube will be explained below.

1) Slider moves so that sample injection needle is positioned at drain port.
2) Common port of valve comes in connection with sampling port, and syringe plunger lowers by a full stroke to such sample into syringe.
3) After common port comes in connection with TC injection port, syringe plunger is pushed up to zero point so that sample in syringe is discharged into drain port.
4) The process of 2) and 3) above is repeated the number of times preset on the LCD screen so that sample contained in the flow line from the sampling needle to sample injection needle (including microliter syringe) is replaced with new sample. The above process is hereinafter called "syringe washing".
5) With common port connected with sampling port, specified volume of sample is sucked.
6) Sample injection needle moves to the center of the upper opening of TC combustion tube.
7) With common port connected with TC injection port, plunger is pushed up to zero point to inject sample into TC combustion tube.

The volume of sample injected into TC combustion tube by the above operation depends on the plunger stroke for sample injection. Sample actually injected into the tube contains the portion which has been contained in Teflon tubing between sample injection needle and four-port valve.

In both syringe cleaning process and sample injecting process, plunger retracts a little after it is pushed up to zero point to inject sample through injection needle. This retraction helps prevent drops of sample from remaining on the needle end after injection. The retracting amount is corrected appropriately at

each time of sample measuring-off and injection so that actual injection volume always conforms to the setting on the LCD screen. The sampling needle set in the sampling tube end has ten small holes on its side so as to serve as a protection filter which prevents SS in the sample from being sucked into the sampling tube and from clogging the injection needle.

If sample contains large amount of SS, the sampling needle is readily clogged. Therefore, use sample which contains as small quantity of SS as possible. If sampling needle is clogged, remove the foreign matter with supplied cleaning wire.

The sample flow line from sampling needle to injection needle is all made of fluorocarbon polymers or hard glass excepting the needle which is stainless steel.

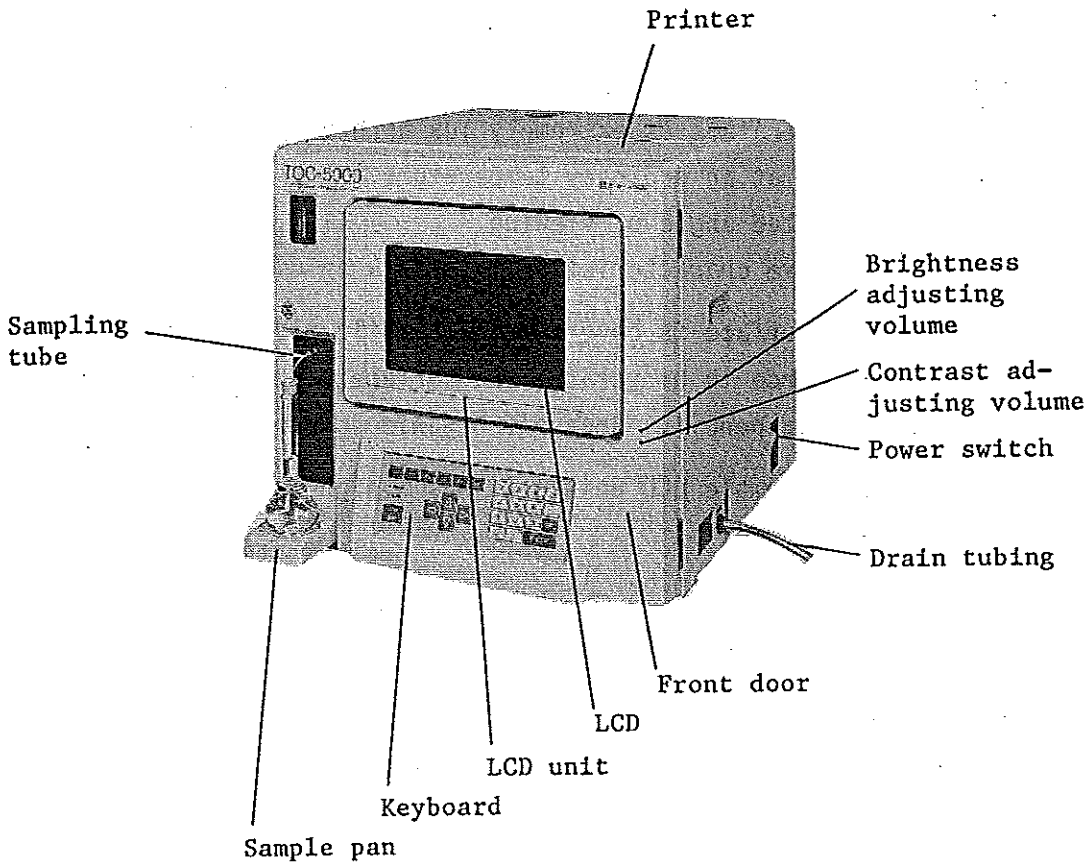


Fig. 4.4 Front View of TOC-5000

4.8 LCD Unit

The TOC-5000 employs a high contrast, 640 × 400 dots large LCD with backlight. The unit can be rotated vertically at about 10°C to allow adjustment of the viewing angle. Adjust the angle gently. Contrast adjusting volume (lower) and brightness adjusting volume (upper) are provided to the right of LCD unit on the front panel. They have been adjusted appropriately before shipment. Make adjustment if necessary.

Contrast adjusting volume (lower):

For adjusting picture contrast

Brightness adjusting volume (upper):

For adjusting brightness of the LCD backlight. Backlight life becomes shorter as brightness is increased. Therefore, it is advisable not to adjust for unnecessarily high brightness.

1. LCD may deteriorate in display performance or response when ambient temperature is extremely low or high. (It resumes its normal state at an ordinary temperature.)
   LCD has limited angle of viewing.
   Keep the above characteristics in mind when using the unit.
2. LCD screen surface is covered with plastic plate. Do not apply excessive force to or scratch the surface with sharp-pointed end such as a pencil. Keep off organic solvent.
3. Do not disassemble LCD unit. It has a high tension part inside and is dangerous.

4.9 Printer

The TOC-5000 uses a thermosensitive serial dot printer which prints out measured data and peaks on 112 mm wide thermosensitive chart paper.

4.10 Keyboard

Operation of the equipment is very easy. It is operated conversationally through LCD screen, using only a small number of keys. Function of each key is shown below.

Key-top Indication	Name	Function
START/STOP	Start/stop key	Starts or stops measurement.
F1 ~ F6	Function key	Allocated for different functions for each screen. The functions are displayed at the bottom of each screen.
↑ ↓ ← →	Cursor key	Shifts cursor to desired position to select an item, input a character or execute a particular function.
0 ~ 9	Ten key	Inputs numeric values.
ENTER	Enter key	Enters input numeric values or selected condition.
CE	Clear entry key	Corrects erroneous inputs. This key does not work for inputs already entered by ENTER key.
FEED	Feed key	Feeds printer chart paper. Chart paper is kept being fed as long as this key is depressed.

5. PREPARATION

5.1 Preparation for Measurement

Some parts are supplied as disassembled from the main body, for protection against possible damage during transportation. Mount such parts in the main body.

5.1.1 Water supply to dehumidifier drain pot

1) Supply transparent plastic-made dehumidifier drain pot with pure water (or deionized water) to the level of drain pipe on the drain pot side wall.

2) Cap the drain pot and fix it in a specified position (See Fig. 4.1.) with fixing band.

5.1.2 Mounting of humidifier

Fill humidifier with distilled water to the upper line through port on the side wall. For high sensitivity measurement, supply water with as low TC content as possible.

Connect longer glass tube to the reducing union of Teflon tubing with L mark, and shorter glass tube to the reducing union of Teflon tubing with S mark. Then, set humidifier to fixing band. In this state, carrier gas can be humidified adequately.

5.1.3 Setting of CO_2 absorber

1) Cut the ends of two pipes on the cap of CO_2 absorber, with cutter edge.

2) Two flexible tubes extend from the upper part of the equipment rear panel. Connect the one with L mark to the L-labeled pipe, and the one with S mark to the S-labeled pipe of the CO_2 absorber. Then, place CO_2 absorber on the equipment side, taking care so that flexible tubes are not bend at an acute angle.

5.1.4 Filling of TC combustion tube with TC catalyst

There are two kinds of catalyst: TC catalyst set (P/N 638-92069-01) and high sensitivity TC catalyst set (P/N 638-92070-01). Select suitable type according to intended measurement with reference to Para. 6.1.1.

Fill TC combustion tube with TC catalyst as shown in Fig. 5.1. One full bottle of TC catalyst is required for each batch of filling. When filling the tube with catalyst, be sure that the catalyst is not soiled and is free from foreign substance; soil or foreign substance in catalyst causes high blank value or abnormal measurement. A new catalyst tends to cause largish blank peaks in the beginning which turn smaller gradually with increase in the number of times of injection of water or samples and finally stabilizes in the size (peak area). Accordingly, execution of calibration or sample measurement in state of large blank peaks causes change of the measured values with change of the blank peak area, bringing about an error. To make the blank peak small and stable, inject distilled water or ion exhanged water at least for 4~5 hours (about 100 times) in the CYCLE MODE (refer to the Section "MEASUREMENT START"). Normally, distilled or ion exchanged water is injected on the conditions of the range X1 and the injection volume of 100 μl, and the peak areas during the while are printed out for judgement whether blank peaks are nearly stabilized. A proper size of blank peaks depends on the quality of water used (concentration of TC contained there in as impurity) or the TC concentration range when a sample is measured actually. When measurement is intended of the concentration of about 100 ppm or higher, blank peaks corresponding to 1 ppm bring about little effect on measured values. For measurement of the concentration of about 10 ppm or so, however, blank peaks are required to be corresponding to 0.5 ppm max. and stable. Generation of blank peaks owing to use of TC catalysts cannot be avoided in consideration of their characteristics. When measurement under condition of smaller blank peaks is intended, use of high sensitivity catalysts is recommended.

5.1.5 Filling of TC combustion tube with high sensitivity TC catalyst

Fill TC combustion tube with high sensitivity TC catalyst set (P/N 638-92070-01) as shown in Fig. 5.1. Two cylindrical containers of catalyst are required for one batch of filling. Bring one open end of catalyst container close to the opening of TC combustion tube and push catalyst with supplied catalyst filling stick from the other open end of container into combustion tube.
Whenever the TC combustion tube has been filled with new TC catalyst or high sensitivity TC catalyst, execute REGENERATION OF TC CATALYST once or twice on 『MAINTENANCE』 screen.

5.1.6 Connection of TC combustion tube

1) Disconnect mounting plate with slidable TC sample injecting block from fixed plate and insert TC combustion tube into the center hole (about 22 mϕ) of TC furnace with leg tube down.

2) Connect leg tube projecting from TC furnace bottom with spiral stainless tube whose inner surface is glass-coated.
Insert the leg tube end in swage lock fitting, and tighten box nut lightly to allow leg tube to be movable vertically. With this state, lift up TC combustion tube so that leg tube end is 2 to 3 mm apart from the contact face (marked with asterisk in Fig. 5.2) of swage lock fitting, and tighten box nut firmly with fingers. If TC combustion tube cannot be moved upward, sufficient sealing has been obtained. Avoid using tool (such as spanner) to tighten box nut. If tool must be used to ensure complete tightening, retighten only lightly with tool. Since TC combustion tube is made of quartz glass, tightening to excessive torque, wrenching or careless handling will damage the tube. Do not tighten box nut with leg tube end on the contact face of swage lock fitting; the end may be broken.
Never use metallic or other front and rear ferrules than those (Teflon-made) supplied with swage lock fitting.
Protect inner and outer surfaces of TC combustion tube lower end, inner surface of swage lock fitting, ferrules and other gas contact surfaces from grease, oils and other organic substances, particularly when high sensitivity measurement is conducted.

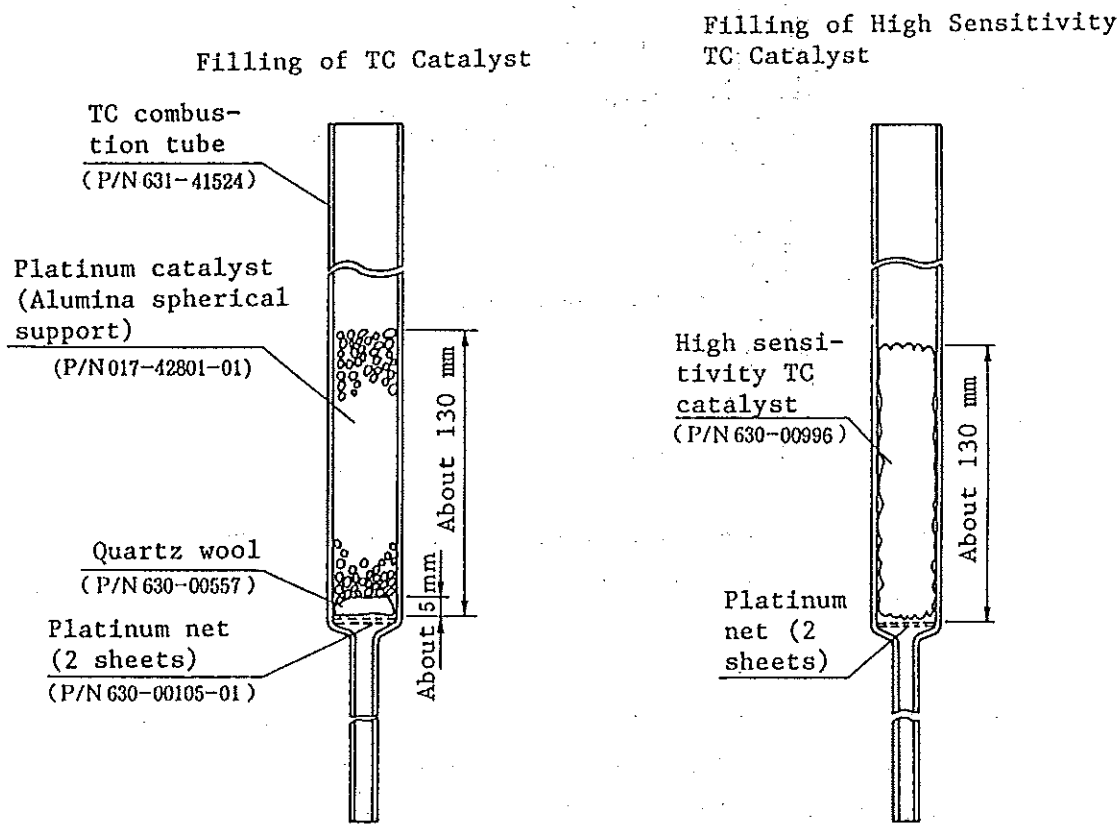


Fig. 5.1 Filling of TC Combustion Tube with Catalyst

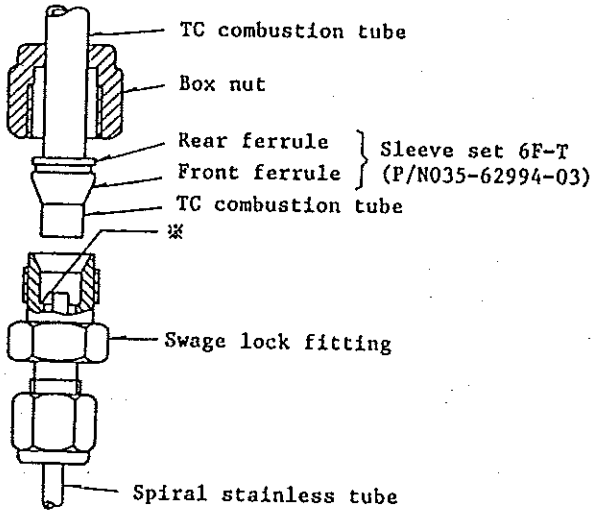


Fig. 5.2 Connection between TC Combustion Tube Lower End and Swage Lock Fitting

3) Apply thin coat of supplied high vacuum silicon grease on the outer surface of TC combustion tube upper end (which is to be in contact with O-ring), and insert the coated upper tube end portion into the bottom hole of slidable TC sample injecting block. For this coating, use as little silicon grease as possible, taking great care so that grease does not enter TC combustion tube. Screw mounting plate with slidable TC sample injecting block to the fixed plate, ensuring that slider and slider-driving gear are engaged with each other as shown in Fig. 5.3. Take care so that vertical force is not applied on slider; otherwise, airtightness between slider and O-ring in sample injecting block would be lost.

4) Finally, check that the clearance between spiral stainless tube lower end and equipment bottom plate is within about 1 mm (No clearance is optimum). When the clearance is too large, loosen the joint (the box nut on the lower part of the swage lock fitting) between spiral stainless tube and swage lock fitting, lower the stainless tube (or decrease the insertion amount of the stainless tube in the swage lock fitting) to minimize or eliminate the clearance, and tighten the box nut firmly. If large volume

of sample is injected with large clearance here, pressure increase due to rapid evaporation of the sample in TC combustion tube may cause disconnection between TC combustion tube and slidable TC sample injecting block.

5.1.7 Setting of chart paper on printer

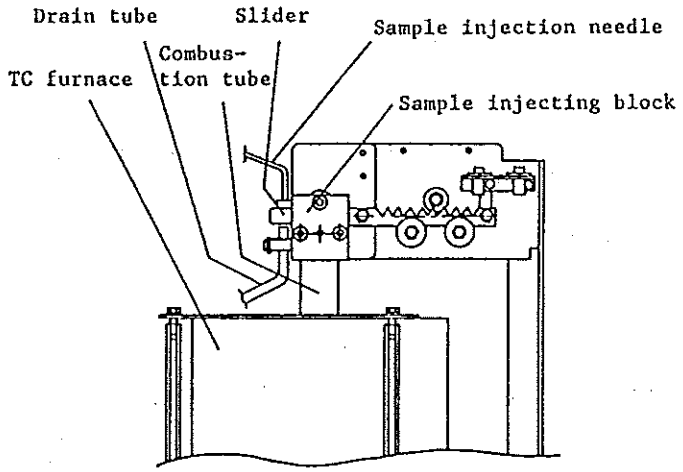
1) Open printer cover and place a chart paper roll having a shaft in the center hole on chart holder.

2) Fold the front end of paper obliquely and insert it into paper insertion slit of printer. Turn on power switch and depress [FEED] key to send the paper into printer until the folded front end comes out of cutter section.

3) Correct the paper direction.
Do not actuate printer without chart paper set on the printer; printer head would be damaged.

5.1.8 Mounting of microliter syringe on syringe pump

Mount microliter syringe of appropriate size on syringe pump. 250 µℓ and 2,500 µℓ microliter syringes are supplied as standard accessories. Normally, 250 µℓ syringe is used.
Refer to Para. 6.1.2 in selecting microliter syringe. Some of the operation for mounting the syringe is to be conducted through LCD screen. For detail, refer to the paragraph indicated in parentheses.



TC furnace block (viewed from right side)

Fig. 5.3 Connection of TC Combustion Tube Upper End

Mount microliter syringe in the following procedure:

1) For normal measurement, use supplied microliter syringe directly. For high sensitivity measurement, immerse syringe barrel (with plunger separated from glass cylinder) for 20 to 30 minutes in cleaning detergent for physical and chemical glass appliances, rinse thoroughly in running water and finally in ultra pure water prior to mounting.

2) Lower plunger holder of syringe pump. If power switch of equipment has not been turned on, the holder can be moved manually.

3) Put plunger of microliter syringe through plunger head-fixing screw and mount the plunger in barrel of microliter syringe.

4) Suck pure water into microliter syringe, taking care so that bubbles are not formed near plunger tip. Screw the microliter syringe manually in the threaded hole in the lower face of four-port valve and fix it.
   Then, pull plunger head downward and secure it with fixing screw in the recess of plunger holder so that plunger will not move. At this time, there is no problem if air enters the upper part of microliter syringe.
   In fixing threaded parts, be careful not to tighten the screws to excessive torque with tool.

5) Turn on power switch while depressing [ENTER] key, and start the equipment in the ALL RESET mode. [ENTER] key must be kept depressed until ALL RESET is displayed on the screen.

6) Set microliter syringe size (SYRINGE SIZE) on 『GENERAL CONDITIONS』 screen.

7) Conduct automatic zero point detection of syringe pump (ZERO POINT DETECTION OF SAMPLE SYRINGE PUMP ...) on 『MAINTENANCE』 screen. Also set date at the same time. This completes mounting of microliter syringe.

If it is necessary to replace microliter syringe with power switch ON, use MECHANICAL CHECK function on 『MAINTENANCE』 screen. Lower plunger holder of syringe pump by key operation and replace syringe. Specific procedure for this replacement is as follows:

1) Bring cursor to MECHANICAL CHECK on 『MAINTENANCE』 screen and depress [ENTER] key. Then the screen will change as shown in Fig. 5.4.
On this screen, syringe pump plunger, four-port valve, TC and IC slidable sample injecting blocks and various solenoid valves can be operated with keyboard. Operation effected by each key is shown in Fig. 5.4.

2) Depress [DOWN] key to lower syringe pump plunger. When this key is depressed, plunger lowers for full stroke and stops automatically. Downward movement of plunger can be stopped at any time by depressing DOWN key again. ([UP] key is used in the same way for upward movement of plunger.)

3) Remove plunger head fixing screw. Push up plunger by about 20 to 30 mm and dismount microliter syringe barrel from the threaded hole of the four-port valve.

4) Mount new microliter syringe, referring to the steps 3) and 4) of the syringe mounting procedure.

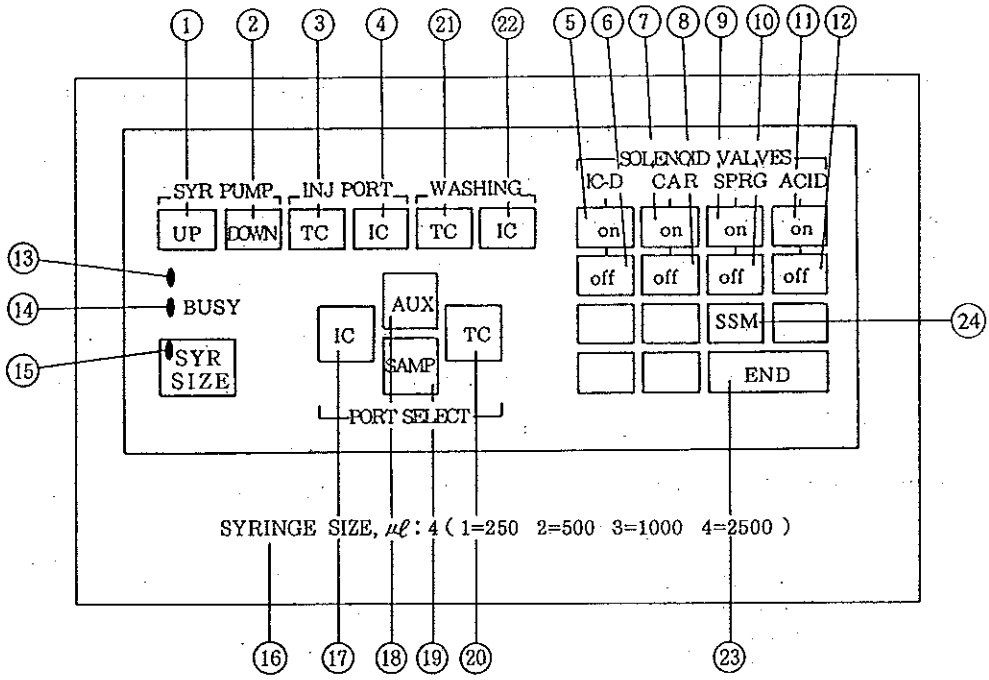
5) Depress [SYR SIZE] key.
Microliter syringe size can be set on this screen. Input the size of microliter syringe which has been mounted, and depress [ENTER] key. Then the screen returns to the one shown in Fig. 5.4.

6) Put sampling tube in water-containing vessel, and move plunger up and down one or two times by operating [UP] and [DOWN] keys to check for smooth movement. Then depress [END] key ([ENTER] key) to go out of this screen and return to 『MAINTENANCE』 screen.

7) Move cursor to ZERO POINT DETECTION OF SAMPLE SYRINGE PUMP and depress [ENTER] key to execute this program.

8) Take care of the following points in operation for MECHANICAL CHECK. (These points must be also kept in mind in operations other than replacement of microliter syringe.)
- When certain operation is conducted by depressing a key, BUSY lamp will remain lit until the operation is completed. Therefore, do not perform next operation until the light goes out.

· TC or IC slidable sample injecting block (INJ PORT) may not be moved by one key depression. It may be necessary to depress the key again after a while to move it. Therefore, after depressing the key, be sure to check that the block is moving.



① UP : Moves microliter syringe plunger upward.

② DOWN: Moves microliter syringe plunger downward.

③ TC : Moves TC slider with sample injection needle.

④ IC : Moves IC slider with sample injection needle.

⑤ on : Opens drain discharging pinch valve of IC reaction vessel.

⑥ off : Closes drain discharging pinch valve of IC reaction vessel.

⑦ on : Opens solenoid valve for carrier gas.

⑧ off : Closes solenoid valve for carrier gas.

⑨ on : Opens solenoid valve for sparge gas.

⑩ off : Closes solenoid valve for sparge gas.

⑪ on : Changes solenoid valve position for IC reagent (acid) feed gas to feed side.

⑫ off : Changes solenoid valve position for IC reagent (acid) feed gas to stop side.

⑬ Lamp with no indication : Power lamp

⑭ BUSY : Indicates that a program is being run.

⑮ SYR SIZE : Sets microliter syringe size.

⑯ Displays the size of microliter syringe set at present.

⑰ IC : Connects microliter syringe with IC injection port.

⑱ AUX : Connects microliter syringe with AUX port.

⑲ SAMP : Connects microliter syringe with sampling tube.

⑳ TC : Connects microliter syringe with TC injection port.

㉑ TC : Washes microliter syringe twice in the discharge port of TC injecting block.

㉒ IC : Washes microliter syringe twice in the discharge port of IC injecting block.

㉓ END : Ends the current screen.

㉔ SSM : Functions when the optional solid sample module SSM-5000 is connected.

Fig. 5.4 MECHANICAL CHECK Screen

5.1.9 Setting of IC reagent

1) Put a pallet on the bottom at the center front behind the front door of equipment. There are two projections on the back side of the pallet. Insert these projections in the two holes in the bottom plate to prevent the pallet from moving.

2) Add approx. 120 ∿ 130 mℓ of IC reagent (250 mℓ in white plastic bottle) to IC reagent container (125 mℓ glass bottle) provided.

3) Apply thin coat of supplied silicon grease on O-ring of adapter for IC reagent container; connect adapter to IC reagent container inlet and secure cap. Check that black plastic tube (IC reagent carrier gas tube) and slim plastic tube leading to branch tube on the left surface of IC reaction vessel are connected to adapter. Also check that slim plastic tube tip is located at the bottom of IC reagent container. Through this tube, IC reagent is sent to IC reaction vessel.
Make sure that IC reagent container and adapter connecting section or adapter and tube connecting section is free from any gas leakage; otherwise, IC reagent cannot be fully sent to IC reaction vessel, resulting in inaccurate IC measurement. Check for gas leakage as described in the following paragraph.

4) Place adapter-mounted IC reagent container in pallet.

5.1.10 Preparation of IC reaction vessel

1) Dismount IC reaction vessel, and position three-way cock lever as shown in the center of Fig. 1.2. (Three-way cock is located in the lower part of the vessel.)

2) Fill the vessel with pure water (or deionized water) to the level of the drain pipe on the right side wall of the vessel.

3) Apply very thin coat of silicon grease on the outer surface of IC reaction vessel upper portion, fit this coated portion in slidable IC sample injection block, and fix it.

4) Position three-way cock lever as shown in the left of Fig. 1.2.

5) Run the program for regenerating IC solution (REGENERATION OF IC SOLUTION) on 『MAINTENANCE』 screen. (Move cursor to the appropriate item, depress [ENTER] key to enter the program and depress [START/STOP] key.)

6) When the program starts, drain valve for IC reaction vessel opens for 3 to 20 seconds (depending on the microliter syringe used). After the valve is closed, two pulses of pressurized air is supplied to IC reagent container so that IC reagent is sent to IC reaction vessel. At this time, check that IC reagent is flowing through the tube to IC reaction vessel. If IC reagent does not flow into the vessel, stopping in the middle of the tube, start REGENERATION OF IC SOLUTION program again after the program ends. (IC solution refers to water acidified by IC reagent in the IC reaction vessel.) When IC reagent does not rise in the tube at all or when it rises to some extent and drops, gas leakage is suspected at a joint of IC reagent container. In such a case, locate and eliminate leakage.

5.1.11 Affixing of function key label for LCD

To facilitate operators to know the function of each function key (F1 through F6) on each screen, labels indicating the functions are supplied by us. Affix these labels below LCD unit at positions corresponding to the appropriate function keys on the screen.

5.1.12 Preparation of standard solution

1) Preparation of TC (TOC) standard solution

Dissolve accurately measured 2.125 g of reagent grade potassium hydrogen phthalate in zero water in 1 ℓ measuring flask, and add zero water to the marked line. The obtained solution contains 1,000 mg/ℓ or carbon (1,000 mg C/ℓ) or equivalent to 1,000 ppm C. Store the solution as standard stock solution. The concentration of the standard stock solution need not always be 1,000 ppm C. It may be, for example, 2,000 ppm C.
Dilute standard stock solution in appropriate amount of zero water to obtain standard solution of required concentration.
The TOC-5000 controls the combustion furnace temperature at 680°C. Therefore, even if the TOC concentration is the same, organic substance which is hard to vaporize tends to produce

lower peak than the one which is easy to vaporize. This tendency increases with injection volume. (However, since the peak area is the same, measurement result is not affected by this tendency.) Potassium hydrogen phthalate is an example which provides this tendency. Suppose range and injection volume have been set so that the peak height generated with potassium hydrogen phthalate as standard solution is close to the full scale on the screen at the time of calibration. If sample, whose TOC component can generate sharp and high peaks because of the low boiling point, is measured with this state, it may generate higher peaks than the full scale although the TOC content is lower than that in the potassium hydrogen phthalate standard solution.
Standard solution prepared from organic substance of relatively low boiling point may be used to prevent the above inconvenience. However, certain organic substances of low boiling point or low water solubility are inadequate for use in preparing standard solution; it is difficult to prepare standard solution from some organic substances, and TOC concentration tends to change in standard solution prepared from some organic substances. It is necessary to select appropriate organic substances.
As an example, process of preparing standard solution using n-propyl alcohol (1-propanol) will be described below.
Put precisely measured 1.67 g or 2.07 mℓ of reagent grade (99.5% or higher) n-propyl alcohol in 1 ℓ measuring flask and add water to the mark line to make 1 ℓ of solution. Stir it completely to obtain 1,000 mg C/ℓ = 1,000 ppm C standard stock solution.
When the "automatic setting of measuring condition" function (See Para. 3.14.) is used, measuring condition setting provides for TOC components which generate sharp, high peaks. Therefore, potassium hydrogen phthalate may be used to prepare standard solution.

2) Preparation of IC standard solution

Dissolve precisely measured 3.50 g of reagent grade sodium hydrogen carbonate and 4.41 g of sodium carbonate (which was heated 1 hour at 285°C and cooled in sulfate desiccator) in zero water in 1 ℓ measuring flask, and add zero water to marked line. The obtained standard solution contains 1,000

mg C/ℓ of carbon or equivalent to 1,000 ppm C. The subsequent procedure is the same as that for TC standard solution.

3) Zero water

Zero water refers to water which is used as standard solution of TC or IC zero concentration or used for preparing standard solution as mentioned above.

Ideally, zero water should be carbon (TC)-free water. Actually, it is almost impossible to obtain completely carbon-free water. Even water distilled repeatedly and ultra pure water obtained by advanced membrane technology contain about 0.1 ppm (100 ppb) of carbon when measured immediately after manufacturing. Carbon content in distilled water or ultra pure water will increase further while it is stored. CO_2 in the atmosphere (normally 300 to 500 ppm, but much more higher when measured in a room with high population or where combustion apparatus is used.) dissolves in water to become inorganic carbon. The dissolving CO_2 amount is about 0.2 ppm depending on the CO_2 concentration in the atmosphere which contacts water and on the water temperature. (See Table 3.1.) Commercially available distilled water in plastic or glass bottles sometimes contains considerably large amount (nearly 1 ppm) of TC.

Required purity of zero water varies depending on the measuring range. For example, commercially available purified water may be used as zero water for measuring several 100 ppm sample. Select suitable quality of zero calibration water according to the intended measuring range.

5.1.13 Storage of standard solution

Concentration of standard solution changes easily in a short period particularly when it is low.

For this reason, it is recommended to store standard stock solution of high concentration (say, 1,000 ppm C) in airtight containers in a dark, cool place, and dilute it each time for use. Glass bottle is suitable for storing container.

Storing period of TC standard solution is limited. As a standard, 1,000 ppm C of standard stock solution keeps about two months,

and diluted 100 ppm C of standard solution about a week (if stored airtight in a refrigerator).

Airtight storage is important particularly for IC standard solution whose concentration is easy to change due to absorption of CO_2 contained in the atmosphere.

When measurement reproducibility has deteriorated or sensitivity has changed, prepare new standard solution. Any turbidity or foreign matters in standard solution is an indication of possible deterioration. New standard solution must be prepared again.

5.1.14 Sample pan

Normally, measurement is conducted by placing the sample vessel on the sample pan installed on the front door. If a tall sample vessel (e.g. a measuring flask with a capacity of 250 ml or more) should be used, however, it is difficult to insert the sampling tube fully into the sample vessel placed on the sample pan. To counter this problem, measurement may be conducted by removing the sample pan along with supporting metal fittings and placing the sample vessel on the floor. In such a case, use the attached flare pipe as sampling tube, after cutting it to an appropriate length, as the supplied sampling tube is not sufficiently long. Be sure to attach the sampling needle to the tip of the tube.

As the supplied sparging tube is not sufficiently long for such use either, use as sparging tube the Teflon tube (inside diameter: 2 mm, outside diameter: 3 mm, length: 500 mm), supplied for the protection of the sampling tube, after cutting it to an appropriate length.

6. MEASUREMENT

6.1 General Conditions for Analysis

Conditions which are common in each measurement and seldom changed in every measurement is called general conditions for analysis. They are set on 『GENERAL CONDITIONS』 screen separately from other conditions.

Items of general conditions for analysis which especially need to be explained are described below. For the contents of this screen display and the manner of operation, refer to 『GENERAL CONDITIONS』 (P. 98). For AUTO RANGING AND INJ VOL, refer to Para. 3.14 "Automatic Change of Range and Injection Volume".

Be sure to set the same kind of catalyst and microliter syringe size, as mounted in this equipment since the equipment operation, data processing and display manner are determined by these settings. Otherwise, satisfactory performance cannot be expected and, in the worst case, some parts may be damaged.

6.1.1 TC catalyst

TC catalyst set (NORMAL) and high sensitivity TC catalyst set (HIGH SENS) are provided. Appropriate set should be used depending on the measuring range.

1) TC catalyst set (P/N 638-92069-01)

When this catalyst is used, the maximum sample injection volume is limited to 100 μℓ. If the limit is exceeded, the rapid increase of internal pressure may be caused, damaging the piping system and so on. It may cause TC combustion tube to be damaged and piping to be disconnected.

Although measurement of the order less than 1 ppm is possible, blank peak is generated in such measurement. Therefore, in view of measurement limit and reliability, for measurement of the order less than several ppm, it is recommended to use high sensitivity TC catalyst set. This set is recommended to be used for measurement of higher concentration.

There is no other restriction in using it. It is usually used under the conditions other than the high sensitivity measurement because it is less expensive than the high sensitivity TC catalyst set.