4) A glass bottle is the most appropriate as a container. When plastic container is used, care must be taken because TOC composition may eluded from the container.

To minimize contamination by sample container and measuring parts or the one accompanying sampling and measuring operations, it is preferable to use a large amount of sample. Sample of 100 mℓ at least, or 200 to 300 mℓ, if possible, should be used for measurement.

5) When pretreatment for IC removal is to be performed, use sample of approximately 50~100mℓ and leave space on the upper part of the container so as to avoid an overflow of sample in sparging treatment.

6) To isolate sample from external contamination source, seal the container with a paraffinic film or the like after inserting the sampling tube into the container.

7) High sensitivity measurement should be performed when blank of the equipment is small and stabilized. For blank, refer to the next paragraph.

[Ultra pure water measurement example]

An example of measurement of ultra pure water is shown below.

1) Since the TOC (TC-IC) measuring method tends to bring about larger measured value error than the NPOC measuring method due to the following reasons, measurement is taken of NPOC.

   (1) A considerable portion of TC is occupied by IC in many cases, so that errors of TC and IC measured values are added, bringing about large influence on TOC value.

   (2) IC value easily changes due to absorption of carbon dioxide in atmosphere during handling or measurement of a sample. Especially, change of IC value largely affects TOC value, if it occurs during TC measurement-to-IC measurement interval.

   (3) Two kinds of calibration curves of TC and IC are necessary. Unless measurement is taken with the standard solutions prepared with considerable care, TOC value is influenced largely.

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In other words, it is required to pay special attention to the above items, when use of TOC (TC-IC) measuring method is intended.

2) Concerning the calibration curves, those with 2-point calibration curves moved to the origin are used. For instance, by creating a 2-point calibration curve of 0 and 400ppb and moving them to the origin, correction can be made of TC contained in pure water used for preparation of TC standard solutions of 0 and 400ppb. Be sure to prepare TC standard solutions of 0 and 400ppb simultaneously using a same pure water. For example, prepare two 250ml measuring flasks, rinse them using pure water (with TC concentration as low as possible) sufficiently, pour a specified amount of TC standard stock solution (1ml of 100ppm TC standard stock solution in the case of preparation of 400ppb standard solution) precisely, and add pure water up to the marked lines on two measuring flasks. Execute these operations in time as short as possible with care not to allow contamination from the outside.

In the case of use of water purifying apparatus, do not use pure water flowing out first due to high TC concentration. Also, take pure water into two measuring flasks alternately in several times so as to use pure water TC concentration as same as possible. When lastly adding pure water up to the marked lines of the measuring flasks, avoid use of pure water left in a washing bottle for a long time (TC concentration of such water is considerably high), but use one newly sampled.

3) Before measurement of the standard solution, repeat measurement at least five times of pure water with 2~3 droplets of 1N hydrochloric acid added per 100ml on the conditions (range, injection volume and sparge time) same as those of measurement of the sample, and make sure that the peak is stabilized. This operation is required due to the following reasons. Especially, peak of high sensitivity measurement tends to appear rather high after

start of measurement, then lowers gradually and finally stabilizes. Therefore, it is likely that the calibration curve is created using data obtained in state with the peak not stabilized, if its creation is conducted immediately after start of measurement. Select any one of a calibration curve number on 「CALIBRATION/CONDITIONS」 screen, set "400ppb (or 0.4ppm)" for concentration of 1st standard solution, "5~10 times" for number of injections and "10 minutes" for sparge time. Set acidified pure water, start measurement, and check if the peak area is stabilized. Make additional injection, as necessary. (This measurement is not for the purpose of creation of a calibration curve but for check of stabilization of peak on the conditions same as those of measurement of the sample.)

4) Put the screen back to 「CALIBRATION/CONDITIONS」 screen, select the calibration curve number same as used for the above, and make setting of "400ppb" and "0ppb" for concentrations of 1st standard solution and 2nd standard solution respectively.
Now, start measurement for creation of the calibration curve.
There are two calibration curve creating methods.

(1) After measurement of the standard solution kept in 250ml measuring flask, create 2-point calibration curve. Then, move it to the origin before use. 4~5-time repetitive measurement is available with little influence due to absorption of carbon dioxide in atmosphere, if it is performed promptly with an end of a sampling tube set on bottom of the measuring flask. This method is applicable when IC in pure water used for preparation of the standard solution (i.e. used as 0ppb standard solution) is less than several 10ppb (about 50ppb or low as standard).

(2) Put the standard solution (about 60ml) into a clean sample vial, add 2~3 droplets of 1N hydrochloric acid, set the sparge time to "10 minutes", and then start

measurement. Use the created 2-point calibration curve after moving it to the origin.

As a sample vial used for sparging of a large amount of a sample, a 200~250mm high glass vial with outside diameter of 24~25mm is considered proper. Vials in the proper size can be found in commercial items such as test tube, stopped test tube and color comparison tube.

If a vial with larger outside diameter is used, gas-liquid contact efficiency lowers during sparging. Therefore, it is required to further prolong sparge time.

Remove the sample pan of the instrument, and extend the sampling tube and the sparging tube, when use of the sample vial described above and 250ml measuring flask is intended. Height is disagreeable. For details, see Paragraph 5.1.14. The closer an end of the sparging tube is to the bottom of the sample vial, the better the sparging efficiency is. Sample vial such as test tube, etc. cannot stand for itself. Place them on a test tube stand or the like.

3) Measure NPOC of a sample using the calibration curve created above. Measure the sample by the method same as that for measurement of the standard solution described in (2) above. If measured value lowers gradually in repetitive measurement of a same sample, sparge time may be short. Prolong it.

Necessary sparge time varies with shape of the sample vial, amount of sample, sparge gas flow rate and amount of IC. Set the proper sparge time based on respective conditions.

6.5 TC Blank Checking

Generally, TOC analyzer produces a peak even if it measures carbon-free water by TC channel. This peak is called blank or system blank. Sizes of blank peaks differ depending on configuration of equipment, catalysts and other various factors. TOC-5000 is designed to produce as small a blank peak as possible especially in measurement with high sensitivity catalyst. Measurement of extremely small

amount of TOC (several ten ppb) of such sample as ultra pure water should be performed when blank is sufficiently small and stabilized. TOC-5000 performs blank checking, using blank checking water produced inside the equipment.

The blank checking procedure is as follows:

1) Be sure to use high sensitivity catalyst and 2,500 μℓ syringe for this program. A range is automatically set at ×1.

2) Move the cursor to TC BLANK CHECK on 「MAINTENANCE」 screen and depress ENTER. INJECT VOLUME: **** μℓ is displayed. Then, set injection volume, and depress ENTER.

3) Put pure water on the sample pan, and insert the sampling tube with sampling needle into it. Inject the water into TC combustion tube. Collect condensate from TC combustion tube for use as blank checking water.

4) Depress START/STOP key. TC blank checking program is run automatically. While equipment is operated to prepare for blank checking, PREPARING is displayed on the screen together with the remaining time of the preparing motion. As the first step of preparing operation, blank checking ultra pure water trap is washed in the following procedure: Ultra pure water is sucked and discharged by 2 mℓ three times from trap to make it empty. Then, 2 mℓ of pure water is sucked from bottle on sample pan and injected into TC injection port, and two minutes later 2 mℓ of water accumulated in the trap is sucked and discharged. This operation is repeated twice.

5) 2 mℓ of pure water is injected from bottle on sample pan into TC injection port twice, and then 2 mℓ of water accumulated in trap is sucked and injected into TC injection port three times. Injection is performed at three minutes' intervals. Operation for preparation is thus completed.

6) Blank checking water in the purified water trap is injected by the injection volume set in advance into TC combustion tube, and peak area measuring operation is performed five times.

7) Repeat steps 5) and 6) ten times.

8) When the last time (the tenth time) of this repetition is over, the mean value (MN), standard deviation (SD), coefficient of vari-

ation (CV) of measurement value (peak area value) of this time are displayed. Then, the screen is halted in this state. The size and stability of blank can be judged from these values. If blank checking is to be continued, depress ‖RESTART‖ (F1) key to start this program again. If ‖FINAL‖ (F3) key is depressed during running of blank checking, blank checking ends with the prevailing time taken as the last time.

The result of blank checking is displayed in term of peak area value. Convert it to concentration if necessary, with reference to the peak area value obtained in measurement of standard solution.

Due to contamination of the sample passage in the equipment and the syringe pump inside wall, or extraneous matter on catalyst, blank may not be minimized unless blank checking is repeated several times.

Blank peak is seldom produced in IC measurement channel. This can be confirmed easily by measuring in IC measurement circuit the water acidified and sparged to make the IC content zero; however, such confirmation is not necessary in normal cases. Therefore, a special program like TC blank checking is not provided for IC measurement channel.

6.6 Pretreatment for removal of IC (sparging)

If TOC in a sample containing a large amount of IC than that of TOC is obtained as difference between TC measured value and IC measured value, error of both measurements is added so that error of TOC measured value is large. For example, samples of natural environmental water such as river water, lake water, seawater, underground water, etc., and tap water apply. In such a case, the NPOC method is preferable; apply pretreatment to the sample for removal of IC content in advance, and then conduct measurement of TOC using the TC circuit.

Here, IC (Inorganic Carbon) refers to, strictly speaking, dissolved CO_2 and carbon in form of carbonate in water. Apply small amount of hydrochloric acid to sample to reduce hydrogen ion concentration to pH 3 or lower. Then following reaction occurs so that all CO_2 is separated from carbonate.

$$Me_2CO_3 + 2HC\ell \rightarrow CO_2 + 2MeC\ell + H_2O$$

$$MeHCO_3 + HC\ell \rightarrow CO_2 + MeC\ell + H_2O$$
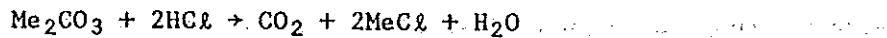
Separated CO_2 and dissolved CO_2 are eliminated from sample by passing nitrogen or CO_2-free air.

Pretreatment process should not be applied to sample containing volatile organic substance which may volatilize during pretreatment. Be sure that sample does not contain such substance before performing pretreatment.

This equipment performs the automatical sparging. Its procedure is described as follows:

1) Move the cursor to item SPARGE GAS FLOW on 「MAINTENANCE」 screen, and depress ENTER key. At the same time when FLOW is displayed, solenoid valve for sparging is opened. Turn the pressure controller for sparging gas to the right, and adjust flow rate to 150 ∿ 200 mℓ/min. Depress ENTER again at this item after setting the flow rate. The solenoid valve is closed.

2) A sparging tube can be attached to the connector of a sampling tube with sampling needle. It is recommended to widen a little the angle of the L-shaped bent portion of the sparging tube to allow the sampling needle to pass through the tube smoothly. It is preferable that the sampling needle alone is projected from the end of the sparging tube when attached. Cut the sampling tube or sparging tube to the appropriate length to effect such status.

3) To remove IC, add a small amount of hydrochloric acid to sample for concentration adjustment to pH 3 or less. 2N-hydrochloric acid (standard accessory) can be used for this acidification. (For this acidification, avoid using phosphoric acid and sulfuric acid; phosphoric acid used for IC reagent in particular would contaminate catalyst.) Put this sample on the sample pan and put sampling tube with sparging tube into it.

4) Set the sparging time (in minutes) at item SPARGING TIME on 「SAMPLE MEASUREMENT/CONDITIONS」 screen. The sparging time differs depending on shapes of sample container, sample volume and sparging gas flow rate since it is affected by efficiency of air/liquid contact. It is recommended to test the required sparging time

under the usual measurement conditions in advance.

5) Now, the equipment has been set for automatic sparging treatment.

When measurement is started, sparging treatment is performed for the
set time, and then the operation turns to be on normal measurement
program. When repetitive measurement is performed, gas is turned
off only during sample sucking.

By the above setting for automatic sparging, sparging is performed
automatically followed by measurement of NPOC. Sparging can be per-
formed independently by setting the equipment as follows:

1) Open front door. Teflon gas tube is connected to the side of the
   connector to which sparging tube is connected. Disconnect this
   Teflon tube together with black rubber tube and take them out
   through the hole in the front side of equipment side panel. Con-
   nect sparging tube with the black rubber tube.

2) Move cursor to SPARGE GAS FLOW on 「MAINTENANCE」 screen and de-
   press ENTER . Then, solenoid valve for sparge gas opens, allow-
   ing sparge gas to flow.
   Adjust the flow rate with sparge gas pressure controller at a
   specified value (normally at 150 mℓ/min.).

With the above setting, since sparging can be performed independ-
ently, it is possible to measure sparged sample at TC measurement
mode while sparging next sample, which improves the efficiency of
measurement. However, it must be noted that sample cannot be meas-
ured (or sucked for measurement) as it is being sparged.

6.7 Measurement of Suspended Solids (SS) – Containing Sample

Since a thin needle (about 0.18 mm I.D.) is used for sample intro-
duction for measurement by this equipment, there is certain restric-
tion in measuring organic SS-containing sample. SS that does not
pass through the sampling needle cannot be measured. Supposing it
can pass through the needle, if it is not uniformly distributed in
the sample in the microliter syringe, measurement with poor repeat-
ability results. One of the possible measures to be taken for such
sample is to crush SS by homogenizer so as to effect uniform dis-
tribution. Homogenizer has two types: high speed agitation type

like a mixer and ultrasonic type. The latter is more powerful. Every type of SS cannot be treated for homogenization. Sludgy SS is relatively easy to homogenize, whereas fibrous SS is difficult. In general, when sample contains sedimented foreign substance, it is recommended to use the supernatant for measurement.

Only DOC (dissolved TOC) can be measured after passing sample through filter. Care should be taken in selecting filter. Do not select a filter from which organic substance may be eluted into sample or whose filter paper fiber may enter the sample.

Avoid measuring sample with sampling needle disconnected; SS and other foreign substance may clog sample injection needle. If all the holes in the sampling needle or the sample injection needle are clogged, plunger-driving motor runs abnormally during suction or injection operation of the syringe pump. Then, "ERROR!! SYRINGE PUMP" is displayed and the equipment is turned off automatically. In such a case, clean clogged needle with supplied cleaning wire and turn on the power switch again.

6.8 Handling of Samples Containing Acids, Alkalis or Salts

Sample containing acid, alkali or salts must be measured with care, considering its influence on the measurement result and on the service lives of the combustion tube and catalyst as well as its possibility of corroding measurement cell and other parts. In measuring such a sample, be sure to observe the following precautions. Please understand that the sample can shorten the life of the measurement cell even if the following precautions are observed. When base line often drifts to the plus side so that optical zero adjustment of the NDIR must be carried out frequently, the measurement cell interior is suspected to have been corroded to a considerable extent. In such a case, stop measurement and take necessary measures, taking care of acids, alkalis, and salts contained in the sample.

The concentrations of these components which influence measurement vary depending on the kind of the components as well as on the TOC content (that is, range and injection volume), although they are normally higher than the order of several 1,000 ppm. Therefore, it seems most essential to dilute the sample as much as possible to lower the concentrations of these components.

When there is possibility of considerable volume of acid and its reaction product remaining in the TC combustion tube, IC reactor and flow path, use the RESIDUE REMOVAL function on the "MAINTENANCE" screen.

6.8.1 Chloric acids and salines

When a sample containing chloric (or bromic) acids and salines is injected into the TC combustion tube, chlorine (Cl_2) is generated. Hydrochloric acid, hypochlorite and such salines as ammonium chloride, calcium chloride and magnesium chloride that are easily decomposed by heat can generate much chlorine.

They are removed by halogen scrubber.

Beside chlorine, hydrogen chloride and other gaseous or misty components are also generated from the sample, but they are eliminated with the IC solution in the IC reaction vessel. If these components are contained in very high concentrations, however, they may not be removed completely. Therefore, dilute the sample to drop the concentrations of salines lower than 3% and acids lower than 1%.

6.8.2 Acids and salts of non-chloric type

When a sample containing other inorganic acids such as sulfuric acid and nitric acid or their salts is measured, it generates interfering components and corrosive components with less interfering or corroding property than those generated by a sample containing chloric acid. Halogen scrubber provides virtually no effect on these components. Therefore, dilute the sample moderately as much as possible. Acids and salts themselves generate components interfering with NDIR detector, possibly causing blank peaks. It is necessary, therefore, to prepare blank test solution containing acids and salts in the same concentrations as those in the sample by using high purity reagent and measure the test solution to check whether or not it produces a blank peak. If it produces a blank peak, check for its reproducibility and examine if it is possible to make measurement with the blank peak subtracted. The influence on the TOC measurement by a blank peak is, in most cases, a positive error. Take care of the blank peak because it may grow larger as the measurement is repeated. The blank peak can be corrected by the two-point calibra-

tion curve method using a blank solution containing blank component alone and a standard solution to which blank component has been added.

TOC in an acid sample can be also measured in the following method. Taking sulfuric acid as an example, neutralize the sulfuric acid with potassium hydroxide or sodium hydroxide to generate thermally stable potassium sulfate or sadium sulfate. At this time, do not neutralize the sulfuric acid completely but make it a weak acid of pH 2 to 3. Sparge the acidified sample for IC removal and measure TOC directly. Neutralization is not effective for nitric acid because every salt of nitric acid is thermally decomposed.

In neutralization, care must be taken so that TOC components in alkali used for neutralization do not enter the acid sample. Normally, acid sample of pH not higher than 3 hardly contains IC (whatever kind of acid it may be). If it should be contained, it can be removed easily by sparging because, at pH not higher than 3, more than 99.9% of carbonate ions or hydrogencarbonate ions as IC components become non-ionization carbonic acid (H_2CO_3), which is easily exhausted as CO_2 in the atmosphere.

6.8.3 Alkali sample

Alkali sample, if measured as it is, deteriorates the sensitivity and reproducibility in a short period and damages the catalyst and combustion tube. Therefore, be sure to adjust it to pH 2 to 3 by hydrochloric acid and pretreat it for IC removal. The sample neutralized by chloric acid generates chloride, which must be treated according to the procedure described in Para. 6.8.1.

6.8.4 Accumulation of salt

As salt-containing sample is measured frequently, salt is accumulated in the combustion tube, increasing resistance against carrier gas flow. When this deteriorates measurement reproducibility, maintenance of the combustion tube interior is necessary. (Refer to Para. 7.3.1 and 7.3.2.)

7. MAINTENANCE

Inspection and maintenance are essential for assuring normal operation
of equipment and accurate and stable measurement.

Inspection and maintenance frequencies and the number of required
consumables vary, depending upon service time of equipment, number
of samples that have been analyzed, quality of samples (particularly
salt concentration) and sample injection volume. Here, it is as-
sumed that service time is eight hours/day and five days/week and
that about 25 samples with salt content not higher than 500 ppm are
measured each day. These are approximate figures intended for refer-
ence in determining replacement time.

7.1 Inspection

Prior to starting measurement, check the following items.

7.1.1 Inspection of printer chart paper

Red mark on chart paper indicates that paper has been used nearly
to the end. Therefore, when red mark appears, replace chart paper
following the procedure shown in Para. 5.1.7. Be sure to use chart
paper (thermosensitive type) specified by Shimadzu.

The entire length of chart paper is 40 m. Assuming 2.5 m of paper
is used daily, one paper roll lasts about 14 days.

7.1.2 Inspection of dehumidifier drain pot water level

Check that drain pot water level is within about 10 mm below over-
flow pipe provided on the drain pot side wall. If the level is
lower, add purified (deionized) water.

Also check that carrier gas does not leak from the drain tube in
drain pot while allowing carrier gas to flow at 150 ml/min. When
If the carrier gas leaks in spite of normal water level in the
drain container, clogging is possible of the membrane filter or
halogen scrubber.

7.1.3 Inspection of humidifier water level

Occasionally check that humidifier water level is between two mark
lines. The volume of humidifier between the upper and lower lines
is about 25 ml which accommodates water for about three-week service.
When water level drops below the lower line, add purified water through
supply port.

7.1.4 Inspection of IC reaction vessel

1) Check that three-way cock lever of IC reaction vessel is positioned as shown in Fig. 1.2.

2) Check that IC reaction vessel water level is within about 10 mm below or above the drain pipe on the vessel side wall. When the water level is lower, run REGENERATION OF IC SOLUTION program on the 「MAINTENANCE」 screen. (See Para. 5.1.10.)

7.2 Regeneration

When measurement sensitivity or repeatability has dropped, it is necessary to regenerate TC catalyst or IC SOLUTION.
This regeneration can be conducted automatically on the 「MAINTENANCE」 screen.

7.2.1 Regeneration of TC catalyst

As samples are measured, various inorganic substances contained in the samples will remain in form of salts or oxides on TC catalyst or high sensitivity TC catalyst surface. Accumulation of such residues can cause measurement sensitivity or repeatability to deteriorate. (Normally, standard solution is measured to check for the measuring performance.) The performance deterioration is obvious particularly after measuring alkaline sample or sample with high IC content. The performance may be restored by injecting diluted hydrochloric acid on the catalyst.

When standard solution or zero water is measured repeatedly with new catalyst or with catalyst which has long been out of service, obtained measurement tends to be higher than usual in the beginning and drop gradually. Injection of diluted hydrochloric acid is also effective when this tendency is prominent.

Catalyst is regenerated in the following procedure on the 「MAINTENANCE」 screen. (The procedure is common for both TC catalyst and high sensitivity TC catalyst.)

1) Replace inner cap of 2N-hydrochloric acid bottle (standard accessory) with supplied holed inner cap and put the bottle on sample pan.

2) Move cursor to REGENERATION OF TC CATALYST and depress ENTER key. The 「REGENERATION OF TC CATALYST」 screen (Fig. 7.1) appears.

3) Following the message, insert sampling tube through the holed inner cap into 2N-hydrochloric acid bottle, and depress START/STOP key.

4) The screen changes as shown right in Fig. 7.1, and the TC catalyst regeneration program is executed automatically. Processing time varies with size of the micro syringe, but it is about 5 minutes and 30 seconds or 7 minutes as longest. Rest time is displayed on the screen.

5) Regeneration can be interrupted any time by depressing ESCAPE (F5) key.
   If designed performance is not restored by regeneration process, replace catalyst according to Para. 7.3.1 or 7.3.2.

For regeneration of TC catalyst and high sensitivity TC catalyst, acids other than diluted hydrochloric acid should not be used. Particularly, avoid using phosphoric acid used for IC reagent because it would generate abnormally high peaks or lead to fluctuated peak values; replacement of catalyst would be necessary to restore normal peaks.

7.2.2 Regeneration of IC solution

As sample is measured repeatedly, IC solution in IC reaction vessel reduces in its acidity gradually through reaction with IC components in sample and dilution by sample. Finally, it cannot react sufficiently with IC components. (Peak will linger and peak area will become smaller than that in normal state.)
When lingering of peak is obvious and it is necessary to add IC reagent, T mark appears on the right of peak area value. If AUTO REGENERATION OF IC SOLUTION has been set ON on the ⌈GENERAL CONDITIONS⌋ screen, regeneration is performed automatically. (Refer to Para. 6.1.5.)
If the above program has been set OFF, the message of ⌈GO TO "MAINTENANCE" DISPLAY (#8) THEN REGENERATE IC SOLUTION⌋ appears on the screen. Go to the ⌈MAINTENANCE⌋ screen and perform REGENERATION OF IC SOLUTION.

7.2.3 Regeneration of TC catalyst and IC solution

TC catalyst and IC solution can be regenerated automatically in succession. Move cursor to ⌈REGENERATION OF TC CATALYST AND IC SOLUTION⌋ and depress ENTER key. Regeneration program can be started by

the same operation as for "Regeneration of TC Catalyst" of Para.
7.2.1. This program takes about 8 minutes.

REGENERATION OF TC CATALYST	REGENERATION OF TC CATALYST
PUT SAMPLE TUBE IN ca. 2N-HCl SOLUTION, THEN PRESS [START]	[REGENERATING] WAIT 4'20" UNTIL FINISHED PRESS [ESCAPE] TO STOP
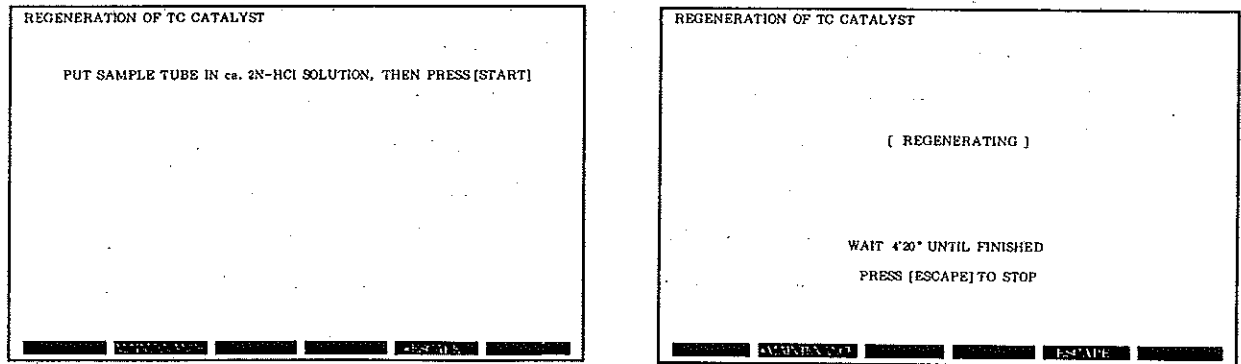
Message is displayed.   Regeneration is under way.

Fig. 7.1 "REGENERATION OF TC CATALYST" Screen

7.2.4 SAMPLE VOLUME COUNT (TC)

Position the cursor at this item and press [ENTER] and set a value for PRESET VOLUME.

The sample volume injected into the TC combustion tube will be totalled at "SAMPLE MEASUREMENT". The accumulated total value will be displayed at the CURRENT item. When the accumulated total value exceeds the preset value, the message "TOTAL INJ VOL EXCEEDS PRESET VOL" is displayed in the DATA PROCESSING, SAMPLE screen, but measurement can be continued.

This function is used to indicate the need to replace the liquid absorbent in the scrubber for the ESU-1.

It is also use to know the need to replace TC catalyst or other consumable by setting the PRESET VOLUME to a value corresponding to the service life of the consumable part.

The user must accumulate data on the sample injection volume to service life ratio prior to using this function. The ratio is dependant on matrix, measurement conditions, and other factors that are typically unique to the individual lab or application.

7.2.5 RESIDUE REMOVAL

This function is used to eliminate the hydrochloric acid and/or chlorine gas remaining in the flow path components after sample containing concentrated hydrochloric acid measurement when using the ESU-1.

When it is intended to remove residue such as reaction product resulting from measurement of samples containing much acid or salt, this program can be used, even if ESU-1 is not used. It is, however, difficult for this program to remove solid such as salt deposited in the TC combustion tube.

Place the sample sipping tube in a vessel of clean water. Position the cursor on RESIDUE REMOVAL and press [ENTER]. The flow lines to the TC and IC injection ports and the microsyringe are washed. Residual chlorine in the TC combustion chamber is flushed out by the water injections. The solution in the IC Reaction Vessel is replaced with the supply of IC Reaction Liquid. This procedure requires about 6 minutes.

About 20-40ml of IC Reaction Liquid is needed for each program execution. Check that there is sufficient volume in the IC Reagent Reservoir. If IC measurement is not performed on any samples, IC Reaction Liquid may be replaced with pure water.

7.3 Maintenance

7.3.1 Washing or replacement of TC catalyst

If measurement sensitivity or repeatability cannot be recovered by the regeneration operation of Para. 7.2, catalyst must be washed or replaced.

When large volume of salt has accumulated in TC catalyst through measurement of sample with high salt content, wash catalyst in tap water to remove accumulating or sticking salt. After neutralizing alkali with five-time-diluted hydrochloric acid, rise catalyst in pure water (or deionized water) and dry at a temperature not higher than 700°C. TC catalyst can be recovered several times by this treatment. (If reproducibility cannot be restored by this treatment, renew TC catalyst.)

When temperature is raised with recovered catalyst set in the TOC-5000, the equipment may generate large amount of hot steam for a while in the beginning. Leave combustion tube outlet open until steam does not come out any more.

When the performance is not recovered by washing, renew platinum catalyst (alumina spherical support) and quartz wool. For this purpose, use platinum catalyst (P/N 017-42801-01) and quartz wool (P/N 603-00557) of TC catalyst set. Each platinum catalyst bottle contains one replacement batch of catalyst, and each bag of quartz wool contains five replacement batches. Clean platinum nets (2 sheets)

(P/N 630-00105-01) and combustion tube inside wall in (1 + 2) hydro-
chloric acid (prepared by diluting one part of hydrochloric acid with
two parts of water), rinse them in water and then dry for recycled
use. Fill combustion tube with catalyst following the procedure
shown in Para. 5.1.4.

Danger
  When dismounting or replacing TC combustion tube, wait until TC
  furnace temperature has dropped sufficiently so that you may not
  get burnt. (Turn ON or OFF TC furnace on the [GENERAL CONDITION]
  screen.) If it is inevitable to handle TC combustion tube at a
  raised temperature, take appropriate protective measure such as
  wearing heat-resistant gloves. If TC injection block is exposed
  to radiation heat of TC furnace center (high temperature part),
  the block may be deformed. Therefore, when combustion tube is
  disconnected, be sure to dismount injection block as well.

7.3.2 Replacement of high sensitivity TC catalyst
  Unlike TC catalyst, high sensitivity TC catalyst cannot be recovered
  by washing. Regeneration is necessary. If measuring sensitivity
  and repeatability cannot be restored by regeneration treatment, re-
  new high sensitivity TC catalyst and treat platinum nets and combus-
  tion tube with hydrochloric acid for recycled use. Thus, high sen-
  sitivity TC catalyst (P/N 630-00996) of high sensitivity TC catalyst
  set is required for maintenance, it is recommended to keep it as
  spare part in stock. Use one bottle of catalyst for each renewal.
  For the filling procedure, see Para. 5.1.5.

7.3.3 Supply of IC reagent
  Check the amount of IC reagent in IC reagent container occasionally.
  If it has been consumed to a certain level, supply IC reagent.

7.3.4 Washing or replacement of TC combustion tube

TC combustion tube can be used until it is broken or cracked to have
gas leakage. As it is used, the tube will become white, losing
transparency because of reaction with salts in samples. Such com-
bustion tube can be used as long as it does not leak gas. When
washing or replacing catalyst, also wash the combustion tube inte-
rior in (1+2) hydrochloric acid solution, rinse in water and dry.

7.3.5 Replacement of high purity air (in cylinder)

Carrier gas high purity air in a 47 ℓ cylinder lasts about three
months. Do not use cylinder gas to the last. Replace it when cyl-
inder pressure drops to several kg/cm^2. Before connecting a new
cylinder, clean connection port so that dust does not enter equip-
ment after connection of cylinder.
It is recommended to keep at least one cylinder as spare in stock.

7.3.6 Replacement of CO$_2$ absorber

Replace CO$_2$ absorber (P/N 630-00999) installed behind equipment
annually.

7.3.7 Replacement of halogen scrubber

The color of the absorbent in the halogen scrubber turns locally as it absorbes
chlorine. The white type absorbent turns blue (or green), and the brown type ab-
sorbent turns blackish color. This colored area will expand toward the outlet side
as absorbed chlorine is accumulated. Renew halogen scrubber (replacement halogen
scrubber: P/N 630-00992) before the front end of the colored area reaches the
point illustrated below. Base line may fluctuate immediately after replacement of
halogen scrubber. In such a case, leave equipment in operational state for a while
until base line stabilizes. Normally, it will stabilize within an hour. Color of
halogen scrubber may turn pale brown eventually in case of the white type
halogen scrubber, which does not provide any problem. When replacing halogen
scrubber, renew scrubber container as well. Replacement of internal absorbent
alone is not permitted. Dispose of used halogen scrubber as hazardous waste.
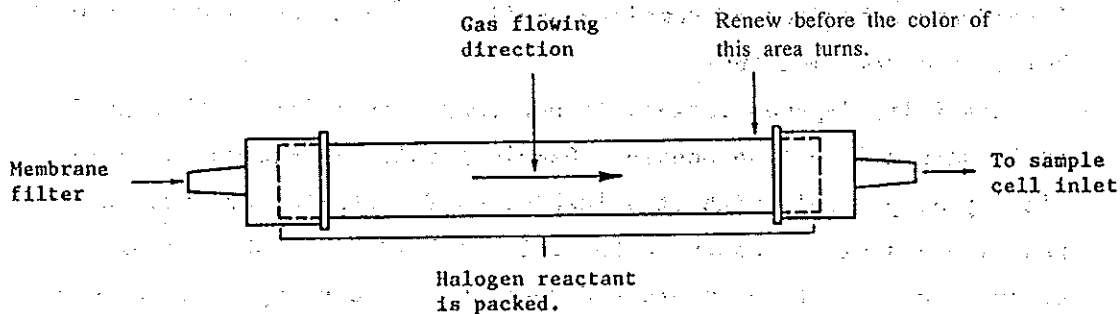
Fig. 7.2 Replacement of Halogen Scrubber

7.3.8 Replacement of syringe pump plunger tip

Plunger tip (made of fluorocarbon polymers) will be worn through service, generating gap between the tip and inner surface of syringe barrel (glass cylinder) and thus causing leakage. If there is leakage, bubbles are formed around plunger tip when sample is sucked, or sample leaks from lower part of barrel when sample is injected. In such a case, replace plunger tip with supplied new one. Old plunger tip can be disconnected from plunger end by pulling by force. Put new plunger tip on a desk or the like flat base with connection hole side faced up, and insert plunger end into this hole perpendicularly from above.

Be careful not to give scratch or any damage to the plunger tip surface; tip with flaw would cause leakage. Be sure to use plunger tip designated by us. Otherwise, malfunction or performance deterioration could occur.

7.3.9 Washing or replacement of sample injection needle
        and sampling needle

Sampling needle serves as a protection filter which prevents SS and other foreign substance from being sucked. If all the holes formed on the side on the needle have been clogged, plunger-driving motor runs abnormally during suction stroke of syringe pump. Then, the message "ERROR!! SYRINGE PUMP" appears and equipment stops automatically. (The same phenomenon occurs during the injection stroke of syringe pump if sample injection needle has been clogged.)

In such a case, remove foreign substance with supplied cleaning wire and turn on the power switch again to resume measurement. Holes are

formed on either end of sampling needle. Insert the needle end with
holes into sampling tube.

Repeatability depends largely on how sample is injected through TC
or IC sample injection needle. Sample should be injected as ver-
tically as possible without splashing on TC catalyst or into IC
reaction vessel, as shown in the left of Fig. 7.3. When sample
splashes or is jetted in wrong directions, remove injection needle
from slider and pass supplied cleaning wire through the needle.
Any foreign substance in needle end must be eliminated by washing.
Sample may be jetted sideway, if not splashing. If such sideway
jetting cannot be corrected by passing cleaning wire through the
needle, bend the needle end a little to allow sample to be jetted in
normal direction. If this method is not effective, renew injection
needle.

Ensure that needle end does not come out of the slider bottom. The
needle end coming out of the slider bottom would damage the O-ring
for sealing when sliding, causing gas leakage.

Besides, the injection needle end shall protrude into the concave
area of the slider bottom by 1~2 mm. If not, water droplets are left
in the concave area after injection of a sample. They may stay
therein to cause a sample to inject poorly.
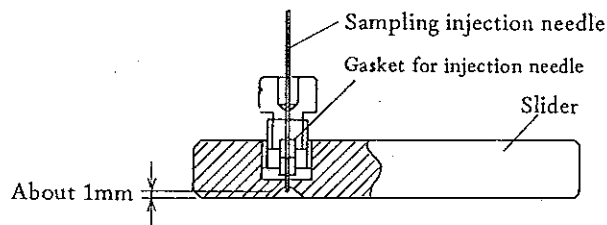
Fig. 7.3   Location of sample injection needle end

Sample injection state in TC injection port can be observed directly
through TC combustion tube with injection needle set in the slider.
It can be also observed as water is injected with injection needle
dismounted from slider but with tube from syringe pump connected
with the needle, in the MECHANICAL CHECK mode on the 「MAINTENANCE」
screen.

Through repetitive connection and disconnection of injection needle
to and from the tube, the tube inner diameter increases, possibly
causing sample to leak during injection. In such a case, cut away
5 to 10 mm tube end portion (on the needle connection side) prior
to connection with injection needle. Since tube becomes shorter

by the cut length, check that the remaining tube is not too short for smooth slider movement. If the tube is too short, cut flare pipe supplied as standard accessory (P/N 638-41269: 1.5 mm O.D. × 0.5 mm I.D. Teflon tube with a flared end) to an appropriate length for use.
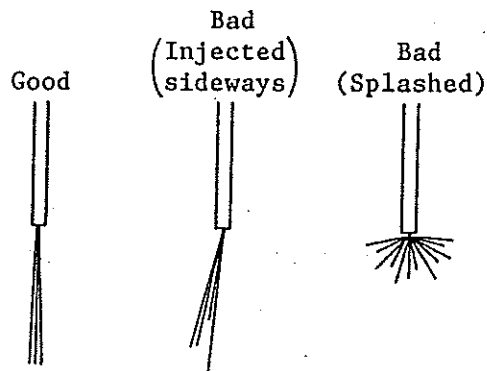
Fig. 7.4 Sample Injecting State from Sample Injection Needle

7.3.10 Replacement of ferrules in TC combustion tube outlet connector

Teflon-made front and rear ferrules in Swagelok fitting at the leg portion of TC combustion tube will be deformed or damaged through service, possibly causing gas leakage. Renew deformed or damaged ferrules. For replacement part, keep sleeve set 6F-T (P/N 035-62994-03) in stock.

7.3.11 Replacement of silicon tube in IC drain pinch valve

Silicon tube in pinch valve for IC drain may be deformed through service; it remains flat when valve is opened. With deformed tube, IC drain cannot be discharged. If sample is injected with this state, sample will overflow and enter dehumidifier, causing IC solution to flow out.

Therefore, renew silicon tube when it has been deformed. At the same time, add IC reagent in IC reaction vessel.

When replacing the tube, set pinch valve (IC-D) ON in the MECHANICAL CHECK mode on the 「MAINTENANCE」 screen.

Renewal of the silicon tube is not necessary if tube portion set in pinch valve is changed or tube is connected inversely.

7.3.12 Replacement of O-ring in slidable sample injection block

Two different kinds of O-rings are placed one over the other between slider and injecting block. When gas leaks from this area, replace both O-rings. The upper white O-ring is O-ring, Teflon P10 (P/N 036-11408) and the Black O-ring is O-ring, 4DP10A (P/N 036-11209). It is also

necessary to replace Teflon-coated O-ring, AS568A-116 (P/N 630-01566), in the
joint between injecting block and TC combustion tube, and O-ring, 4DP22 (P/N
036-11222) in the joint between injecting block and IC reaction vessel if there is
gas leakage.

In replacing O-rings, be careful not to damage O-ring itself and
its mounting surface.

7.4 Adjustment

7.4.1 Zero adjustment of optical system of infrared gas analyzer (NDIR)

To ensure constantly accurate peak area measurement irrespective of
drifting base line, base line signal is always subject to auto
zero correction excepting the peak-measuring period. If base line
exceeds the auto zero correction range by some reason, NG is dis-
played for the base line position (one of conditions for the equip-
ment to be ready for measurement) on the 「MONITOR」 screen, and
READY lamp on keyboard goes off, indicating that measurement is
not possible (WAITING state). In such a case, perform zero adjust-
ment of NDIR optical system in the following sequence:

1) Go to the 「MONITOR」 screen. Base line before auto zero correc-
   tion is shown on this screen.

2) An adjust screw (minus screw) is located in either of two adjust-
   ment holes labeled 「OPTICAL ZERO」 at the back of the equipment
   upper cover. Turn the adjust screw for adjusting base line to
   about 0% while watching the base line on the 「MONITOR」 screen.
   Base line is shifted to the plus side by turning the screw in
   the arrow direction.

3) Since indication on the display is slow in response, rotate the
   screw slowly little by little. If RANGE setting is not ×30, it
   is recommended to change the setting to ×30 on the 「MAINTENANCE」
   screen prior to zero adjustment.

In a very rare case which seldom occurs under normal measurement
condition, rotation of adjust screw in the arrow direction may cause
base line to move to the minus side if such rotation is conducted
after base line made abnormally large amount of shift to the minus
side. In such a case, continue rotating adjust screw until the base
line signal goes to the minus limit and returns to the plus side.

When zero adjustment of optical system is required frequently (or
particularly when base line tends to make drift to the plus side),
it is suspected that the sample cell inner surface of NDIR may be
soiled or corroded. In such a case, replace halogen scrubber or
take care of the acid or salt concentration of sample to be measured.
If measurement is continued without any of measures, cell replace-
ment may become necessary.

8. TROUBLESHOOTING

8.1 Error Messages

Several kinds of error messages appear on the screen, when improper setting, misoperation and instrument fault occur. Descriptions thereof are given below. Date and time of occurrence of a trouble is printed out, if it cannot be recovered unless power is resupplied. (Ex: STOP 06(JUN)-22-1990 18:20)

- GENERAL CONDITION MISMATCH TC CAT (or SYR SIZE, UNIT, NO OF WASHES, INJ VOL) GO TO "CALIBRATION CURVE FILE LIST" DISPLAY (#4), AND [CLEAR FILE]

  When stored calibration curve is read on the 「CALIBRATION/CONDITIONS」 screen or when calibration curve No. is input on the 「SAMPLE MEASUREMENT/CONDITIONS」 screen, if the general measurement condition of the calibration curve does not conform to the current setting of kind of TC catalyst (TC CAT), microliter syringe size (SYR SIZE), unit (UNIT) or number of washings (NO OF WASHES) of general measurement condition, this message appears, and no further measuring operation is conducted. (On the 「SAMPLE MEASUREMENT/CONDITIONS」 screen, the settings that must be in conformance are kind of TC catalyst, microliter syringe size and injection volume.) Set calibration curve whose general measurement condition coincides with the currently set general condition.

- CONCENTRATION IS TOO HIGH

  This message appears when concentration standard solution setting on the 「CALIBRATION/CONDITIONS」 screen is too high. Measurement can be continued but generated peak may exceed the full scale. Decrease the concentration setting or, if microliter syringe used is 500 µℓ to 2,500 µℓ in capacity, replace it with smaller one.

- GENERAL CONDITION MISMATCH

  1) Assuming NO UNIT has been designated for the general measurement condition, if FREE calibration curve No. is used and NEXT key is depressed without setting range or injection volume on the 「CALIBRATION/CONDITIONS」 screen, this message appears; when NO UNIT has been designated, automatic measuring condition setting function does not work even if a value is set for the standard solution concentration. Set range and injection volume or, unless there is special reason for using the NO UNIT

mode, designate unit for the general measurement condition.

2) This message appears if, after a calibration curve No. is designated on 「SAMPLE MEASUREMENT/CONDITIONS」 screen, it is cleared on 「CAL CURVE FILE LIST」 screen and ‖NEXT‖ key is depressed on 「SAMPLE MEASUREMENT/CONDITIONS」 screen with the cleared calibration curve No. left unerased.

When you return to 「SAMPLE MEASUREMENT/CONDITIONS」 screen, delete the calibration curve No. which has been cleared (or made FREE) on 「CAL CURVE FILE LIST」 screen.

• | SPARGE GAS FLOW IS SET ON "MAINTENANCE" DISPLAY (#8) |

This message appears when a value other than zero is set for SPARGE TIME on the 「CALIBRATION/CONDITIONS」 screen followed by setting SPARGE GAS to FLOW state on the 「MAINTENANCE」 screen and returning to the 「CALIBRATION/CONDITIONS」 screen. This message indicates that since SPARGE GAS has been set to FLOW state, solenoid valve for sparge gas is left open so that SPARGE TIME cannot be set. To erase this message, set SPARGE TIME to zero or cancel FLOW setting of SPARGE GAS on the 「MAINTENANCE」 screen.

• | UNIT MISMATCH |

Assuming two or more calibration curve Nos. are set on the 「SAMPLE MEASUREMENT/CONDITIONS」 screen (that is, automatic optimum calibration curve selecting function is used), if each calibration curve uses unit of different group, this message appears. In that case, calibration curve Nos. cannot be entered by | ENTER | key. mg/ℓ and µg/ℓ are of the same group, and ppm and ppb are of the same group.

Set calibration curves with units of the same group.

• | MAXIMUM CONCENTRATION IS THE SAME AS OTHER |

This message appears when setting of calibration curve numbers of same maximum concentration is attempted for "CAL CURVE#" on the 「SAMPLE MEASUREMENT/CONDITIONS」 screen during setting of two or more calibration curve numbers. Set calibration curve numbers of different maximum concentrations.

• | SET SYRINGE SIZE ON "GENERAL CONDITIONS" DISPLAY (#3) |

When equipment has been started with ALL RESET, if | ENTER | is depressed to execute zero point detection of syringe pump without

setting the size of microliter syringe on 「GENERAL CONDITIONS」
screen, this message appears.

Set microliter syringe size on 「GENERAL CONDITIONS」 screen in
advance.

- | GO TO "MONITOR" DISPLAY (#6) TO SEE READY STATE |

After SAMPLING (before INJECTING) on 「DATA PROCESSING」 screen,
if any conditions for READY state are inadequate (some conditions
are NG), equipment is made into WAITING state and this message
appears.

Go to 「MONITOR」 screen and check READY conditions. If BASE LINE
FLUCTUATION or BASE LINE NOISE is NG, it will, in most cases, be-
come OK after a while. If BASE LINE POSITION is NG, pinch with
finger or bend the tube leading to CO_2 absorber in the rear of
equipment to block gas flow, and check that carrier gas is bub-
bling out of the drain tube end inserted in the drain pot.
(With this checking, it can be confirmed that there is no signi-
ficant gas leakage in the flow line to the measuring cell of NDIR.)
Then, adjust zero point, referring to Para. 7.4.1. When tempera-
ture of TC furnace or dehumidifier is inadequate, please contact
our local branch office or agent.

- | FIND SYRINGE ZERO POINT ON "MAINTENANCE" DISPLAY (#8) |

This message appears when zero point detection of syringe pump
is necessary.

Execute zero point detection on 「MAINTENANCE」 screen, ensuring that
the size of microliter syringe attached on the equipment is iden-
tical with the setting on 「GENERAL CONDITIONS」 screen.

- | SET DATE & TIME ON "MAINTENANCE" DISPLAY (#8) |

This message appears when date and time (year, month, day, hour
and minute) have not been set.

Set these data on 「MAINTENANCE」 screen.

- | GO TO "MAINTENANCE" DISPLAY (#8) THEN REGENERATE IC SOLUTION |

When AUTO REGENERATION OF IC SOLUTION has been set at OFF on
「GENERAL CONDITIONS」 screen, if T mark appears at a peak for IC
measurement, this message appears.

This indicates that acid content in IC solution is too low. Go
to 「MAINTENANCE」 screen and perform REGENERATION OF IC SOLUTION
(IC reagent is added in IC solution).

- ERROR!! ZERO POINT DEFECT, SO CHECK SYR SIZE (Reversed display)

  If syringe pump zero point detecting operation has been repeated specified times and zero point cannot be detected, this message appears. This trouble occurs when microliter syringe has not been mounted or when syringe size setting on the display is different from that of syringe actually set in equipment.

  This message will not appear when 500 µℓ is set for 250 µℓ or vice versa or 1,000 µℓ is set for 2,500 µℓ or vice versa.

  Set the same syringe size on the display as that of microliter syringe actually mounted on syringe pump.

- TURN MAIN SWITCH OFF
  THEN TURN IT ON AGAIN.

  This message appears when instantaneous power failure occurs. Resupply power for recovery.

- ERROR!! TC FURNACE OVER HEAT (Reversed display)

  This message appears when TC furnace is overheated above 780°C. Simultaneously with this message, power supply to TC furnace is automatically shut off. Once this message is displayed, it will not be erased even when TC furnace temperature has dropped. Particular measure must be taken. Contact our local agent or office.

- ERROR!! THERMOCOUPLE DISCONNECTED (Reversed display)

  This message appears when TC furnace temperature-detecting thermocouple wire is broken. Power is not supplied to TC furnace when the thermocouple wire is broken.

  Thermocouple must be replaced. Contact our local agent or office.

- ERROR!! THERMOCOUPLE SHORTED (Reversed display)

  This message appears when TC furnace temperature does not rise to 100°C in 10 minutes after setting TC furnace to ON on "GENERAL CONDITIONS" screen.

  This indicates short of thrmocouple, disconnection of power supply for TC furnace or ather etc.

  Contact our local agent or office.

- `RAM ERROR, ROM ERROR` (Reversed display)

  This message appears when error is detected by RAM and ROM checking. If the same message appears when equipment has been started with ALL RESET, please contact our local office of agent.

- `ERROR!! SYRINGE PUMP`

  When syringe pump operation is abnormal due to foreign substance clogging sampling needle or injection needle or other reason, this message appears. Turn off POWER switch, eliminate foreign substance from needle, and turn on POWER switch again. If syringe pump operation has been restored by the above procedure, perform automatic zero point detection for syringe pump on the 「MAINTENANCE」 screen prior to resuming measurement.

- `ERROR!! SYRINGE H.P. DEFECT`

  This message appears when syringe pump home position (H.P.) cannot be detected. Particular measure must be taken. Contact our local agent or office.

- `ERROR!! SELECT VALVE DEFECT`

  This message appears when position detection for syringe pump four-port valve is abnormal. Particular measure must be taken. Contact our local agent or office.

8.2 Troubleshooting

This paragraph describes troubles that can be located relatively easily by users. When cause cannot be identified by procedures shown below or when replacement of parts is required, contact our local office or agent.

1. Error message appears.   → See Para. 8.1.

2. READY lamp does not light. → Go to the 「MONITOR」 screen and identify condition setting which makes equipment out of READY state.

3 Base line remains exceeding auto zero correction range. (NO GOOD base line position)

```
              ┌─────────────┐
              │  Is car-    │
              │ rier gas flow-│      no    ┌──────────────────────┐
              │ ing normally to├──────────→│ Carrier gas is leaking.│
              │  measuring   │            ├──────────────────────┤
              │    cell?     │            │ Eliminate leakage.    │
              └──────┬──────┘            └──────────────────────┘
                  yes│
              ┌──────┴──────┐
              │    Is       │
              │ dehumidifier│      no
              │ temperature is├──────────┐
              │within specified│         │
              │   range?    │           │
              └──────┬──────┘           │
                  yes│                  │
              ┌──────┴──────┐    ┌───────┴──────────────┐
              │Deviated zero point│  │Inadequate temperature│
              │of NDIR      │    │setting for dehumidifier│
              ├─────────────┤    ├──────────────────────┤
              │Adjust zero point.│ │Contact our office for│
              │(See Para. 7.4.1.)│ │after-service.        │
              └─────────────┘    └──────────────────────┘
```
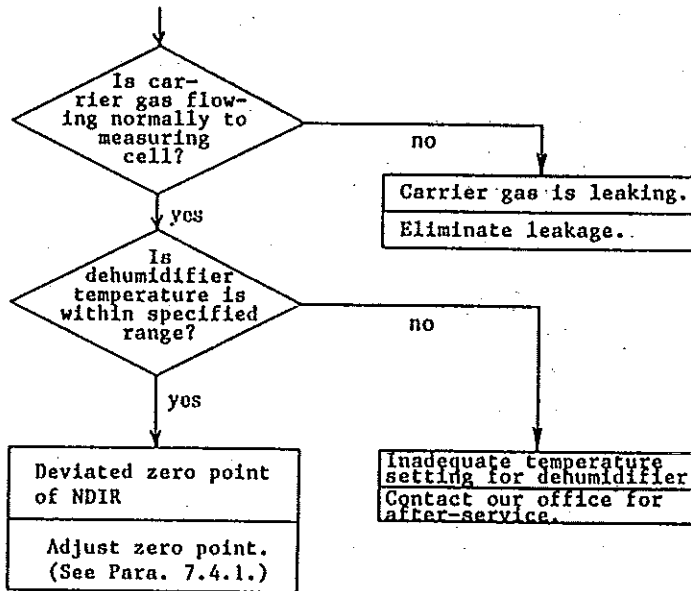
(Note)

If zero point of NDIR cannot be adjusted, defective optical component (such as soiled inner surface of sample cell) is suspected.

4 Base line is not stable.

```
              ┌─────────────┐
              │    Is       │
              │ carrier gas │      no    ┌──────────────────────┐
              │flow normal with-├────────→│ Carrier gas is leaking.│
              │  out leak-  │            ├──────────────────────┤
              │    age?     │            │ Eliminate leakage.    │
              └──────┬──────┘            └──────────────────────┘
                     │
              ┌──────┴──────┐
              │  Was TC     │      no    ┌──────────────────────┐
              │  catalyst   ├────────────→│Disturbing substance  │
              │  replaced?  │            │accumulates.          │
              └──────┬──────┘            ├──────────────────────┤
                  yes│                   │Replace TC catalyst.   │
                     │                   │(See Para. 7.3.1.)     │
              ┌──────┴──────┐            └──────────────────────┘
              │  Is de-     │      no
              │ humidifier  ├──────────┐
              │operating nor-│         │
              │   mally?    │          │
              └──────┬──────┘          │
                  yes│         ┌───────┴──────────────┐
                     │         │Defective dehumidifier│
              ┌──────┴──────┐  ├──────────────────────┤
              │     Is      │  │Contact our office for│
              │ equipment   │  │after-service.        │
              │free from dis-│ │(See Para. 7.2.2.)    │
              │turbance such as vi-│ no └──────────────────────┘
              │bration, abnormal│
              │ambient temperature├──────┐
              │and abnormal │          │
              │supply volt- │          │
              │   age?      │   ┌───────┴──────────────┐
              └──────┬──────┘   │There is disturbance. │
                  yes│          ├──────────────────────┤
                     │          │Eliminate disturbance.│
              ┌──────┴──────┐   └──────────────────────┘
              │Defective NDIR│
              ├─────────────┤
              │Contact our office for│
              │after-service.│
              └─────────────┘
```

5 Standard solution provides poor repeatability.

```
        ┌─────────────────┐
        │ Is H printed out? │─── yes ──────────┐
        └─────────────────┘                     │
                 │ no                            ▼
                 │                    ┌──────────────────────────┐
                 │                    │ Injection volume exceeds │
                 │                    │ measuring range.         │
                 │                    ├──────────────────────────┤
                 │                    │ Adjust injection volume to│
                 │                    │ be within measuring range.│
                 │                    └──────────────────────────┘
                 ▼
        ┌─────────────────┐
        │ Is T printed out? │─── yes ──────────┐
        └─────────────────┘                     │
                 │ no                            ▼
                 │                    ┌──────────────────────────┐
                 │                    │ Insufficient carrier gas │
                 │                    │ flow rate                │
                 │                    ├──────────────────────────┤
                 │                    │ Adjust carrier gas flow  │
                 │                    │ rate properly.           │
                 │                    └──────────────────────────┘
                 ▼
        ┌─────────────────┐
        │ Is standard     │─── no ───────────┐
        │ solution normal? │                  │
        └─────────────────┘                   ▼
                 │ yes                ┌──────────────────────────┐
                 │                    │ Defective standard solu- │
                 │                    │ tion                     │
                 │                    ├──────────────────────────┤
                 │                    │ Prepare standard solution│
                 │                    │ again.                   │
                 │                    └──────────────────────────┘
                 ▼
        ┌─────────────────┐
        │ Is sample in-   │
        │ jected normally │─── no ───────────┐
        │ through needle? │                  │
        └─────────────────┘                   ▼
                 │ no                 ┌──────────────────────────┐
                 │                    │ Defective injection      │
                 │                    ├──────────────────────────┤
                 │                    │ Wash or replace injection│
                 │                    │ needle. (See Para. 7.3.9.)│
                 │                    └──────────────────────────┘
                 ▼
        ┌─────────────────┐
        │ Are there bubbles│─── yes ──────────┐
        │ in syringe?      │                  │
        └─────────────────┘                   ▼
                 │ no                 ┌──────────────────────────┐
                 │                    │ Injection volume is not  │
                 │                    │ accurate.                │
                 │                    ├──────────────────────────┤
                 │                    │ Eliminate bubbles.       │
                 │                    └──────────────────────────┘
                 ▼
        ┌─────────────────┐
        │ Does carrier    │─── yes ──────────┐
        │ gas leak from   │                  │
        │ drain pot?      │                  ▼
        └─────────────────┘        ┌──────────────────────────┐
                 │ no              │ Water level is low.      │
                 │                 ├──────────────────────────┤
                 │                 │ Add water to raise water │
                 │                 │ level.                   │
                 │                 └──────────────────────────┘
                 ▼
        ┌─────────────────┐
        │ Is poor         │
        │ repeatability   │─── IC ───────────┐
        │ for TC channel or│                 │
        │ IC channel?     │                  ▼
        └─────────────────┘        ┌──────────────────────────┐
                 │ TC              │ Deteriorated IC solution │
                 │                 ├──────────────────────────┤
                 │                 │ Regenerate IC solution   │
                 │                 │ (See Para. 7.2.2.)       │
                 │                 └──────────────────────────┘
                 ▼
        ┌─────────────────┐
        │ Was TC catalyst │─── no ───────────┐
        │ regenerated?    │                  │
        └─────────────────┘                   ▼
                 │ yes                ┌──────────────────────────┐
                 ▼                    │ TC catalyst regeneration │
 ┌──────────────────────────┐        │ is insufficient.         │
 │ Deteriorated TC catalyst │        ├──────────────────────────┤
 ├──────────────────────────┤        │ Regenerate TC catalyst.  │
 │ Replace TC catalyst. (See│        │ (See Para. 7.2.1.)       │
 │ Para. 7.3.1 and 7.3.2.)  │        └──────────────────────────┘
 └──────────────────────────┘
```

(Note)
H is printed out when peak exceeds full scale.

(Note)
T is printed out when peak has not been ter- minated at the end of measurement.

(Note)
Bubble forming at the same time as sample in- jection is not abnormal. Carrier gas leakage at any other time is abnor- mal.
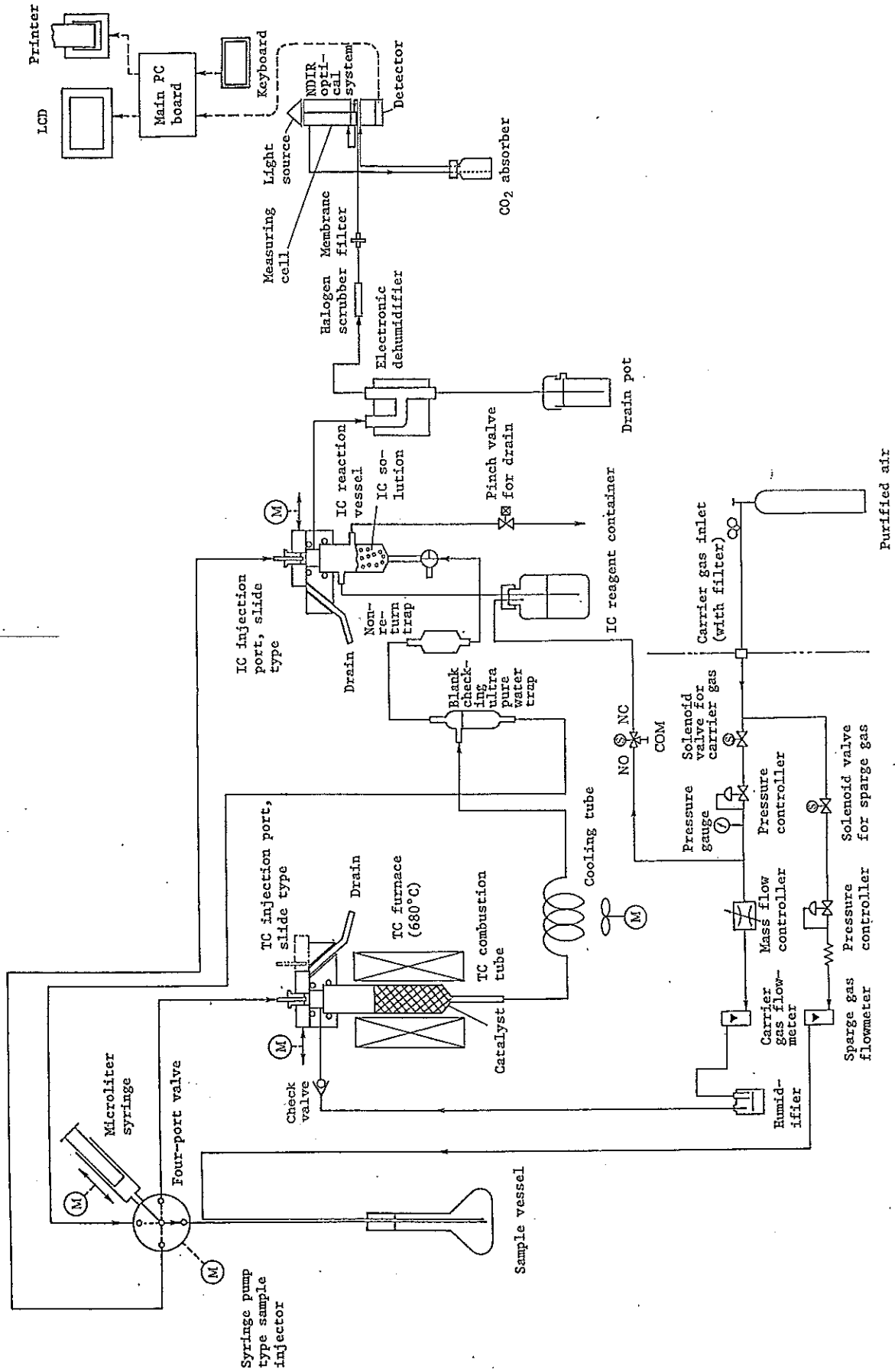
When creation of all calibration curves is completed.
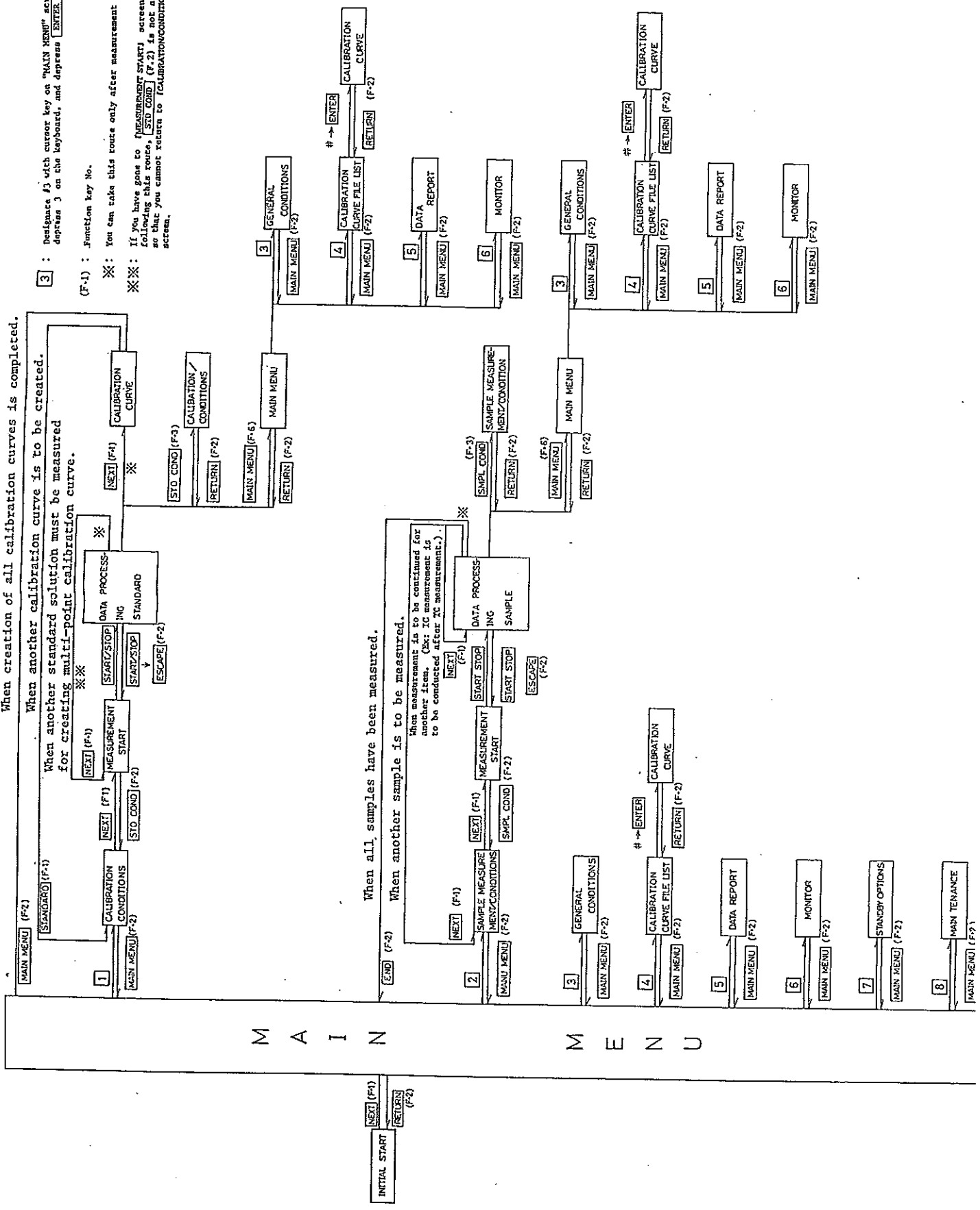
When another calibration curve is to be created.

When another standard solution must be measured for creating multi-point calibration curve.

When all samples have been measured.

When another sample is to be measured.

MAIN MENU

INITIAL START — NEXT (F1) / RETURN (F2)

1 MAIN MENU (F2) → CALIBRATION CONDITIONS → STD COND (F2) / NEXT (F1) → MEASUREMENT START → STANDARD (F1) → NEXT (F1) → DATA PROCESSING STANDARD → START/STOP → START/STOP → ESCAPE (F2) → CALIBRATION CURVE → NEXT (F1) → STO COND (F3) → CALIBRATION/CONDITIONS → RETURN (F2) → MAIN MENU (F6) / RETURN

2 MAIN MENU (F2) → SAMPLE MEASUREMENT/CONDITIONS → SMPL COND (F2) / NEXT (F1) → MEASUREMENT START → NEXT (F1) → DATA PROCESSING SAMPLE → START STOP → START STOP → ESCAPE (F2) → SAMPLE MEASUREMENT/CONDITION → SMPL COND (F3) → RETURN (F2) → MAIN MENU (F6) / RETURN → END (F2)

When measurement is to be continued for another item. (※※: IC measurement is to be conducted after TC measurement.)

3 GENERAL CONDITIONS → MAIN MENU (F2)

4 CALIBRATION CURVE FILE LIST → MAIN MENU (F2) → # → ENTER → CALIBRATION CURVE → RETURN (F2)

5 DATA REPORT → MAIN MENU (F2)

6 MONITOR → MAIN MENU (F2)

7 STANDBY OPTIONS → MAIN MENU (F2)

8 MAINTENANCE → MAIN MENU (F2)

3 GENERAL CONDITIONS → MAIN MENU (F2)

4 CALIBRATION CURVE FILE LIST → MAIN MENU (F2) → # → ENTER → CALIBRATION CURVE → RETURN (F2)

5 DATA REPORT → MAIN MENU (F2)

6 MONITOR → MAIN MENU (F2)

3 GENERAL CONDITIONS → MAIN MENU (F2)

4 CALIBRATION CURVE FILE LIST → MAIN MENU (F2) → # → ENTER → CALIBRATION CURVE → RETURN (F2)

5 DATA REPORT → MAIN MENU (F2)

6 MONITOR → MAIN MENU (F2)

3 : Designate #3 with cursor key on "MAIN MENU" screen or depress 3 on the keyboard, and depress ENTER.

(F·1) : Function key No.

※ : You can take this route only after measurement is completed.

※※ : If you have gone to [MEASUREMENT START] screen, following this route, STD COND (F.2) is not accepted so that you cannot return to [CALIBRATION/CONDITIONS] screen.