

## 2) High sensitivity TC catalyst set (P/N 638-92070-1)

This catalyst is designed especially for high sensitivity TC measurement and accepts up to 2,000  $\mu\text{l}$  of sample injection. It provides high oxidation performance even with large injection volume, and produces negligibly small blank peaks.

Use of this catalyst is restricted when measuring sample containing 5 ppm or more IC. IC detection efficiency in TC measurement drops as IC concentration increases. However, this catalyst has no such restriction for TOC measurement. Its oxidation rate is almost 100% for even highly concentrated TOC. Accordingly, if TOC is measured after pretreatment as described in Para. 6.6 this catalyst can be used for any measurement range because sample contains no IC.

This catalyst ensures highly reproducible measurement of several ppm order of TOC content in river, lake or seawater, underground water or tap water. Since IC content is much larger than TOC content in this type of sample, measurement error tends to be large in the case of measurement of TOC (TC-IC). Thus, take measurement of TOC after removal of IC content. (NPOC)

### 6.1.2 Microliter syringe size (SYRINGE SIZE or SYR SIZE)

There are two sizes of microliter syringe for use in the syringe pump: 250  $\mu\text{l}$  and 2,500  $\mu\text{l}$ . 500  $\mu\text{l}$  and 5,000  $\mu\text{l}$  are also available as optional accessories.

Appropriate size should be selected depending on injection volume range. The injection volume range and concentration range for each microliter syringe are shown in Fig. 6.1.

The values of the concentration range are approximate. Especially for the low concentrations, if very small peak signal is allowed, much lower concentration is also acceptable. On the contrary, if more accurate and reliable measurement value is necessary, use the value twice or three times as much as the low concentration.

Normally, measurement of more than several hundred ppb is most frequent. In that case, every measurement can be performed, if 250  $\mu\text{l}$  microliter syringe is set. As the microliter syringe size is smaller, a plunger moves faster. Accordingly, the measuring time becomes

shorter. 2,500  $\mu\text{l}$  microliter syringe is used when measurement of not more than 100 ppb is made much of. In this case, to avoid errors produced by contamination, different microliter syringe from the one used for sample of relatively high concentration should be used. Injection volume can be set in step of 1  $\mu\text{l}$  on the screen, although actual injection volume changes in step of about 3  $\mu\text{l}$  with a 2,500  $\mu\text{l}$  syringe and 1 to 2  $\mu\text{l}$  with a 1,000  $\mu\text{l}$  syringe.

Table 6.1 Microliter Syringe and Concentration Range

Size	Part No.	Part No. of Plunger Tip	Injection Volume	TC Concentration Range	IC Concentration Range
250 $\mu\text{l}$	630-02591-04	638-60734-05	4 ~ 250 $\mu\text{l}$	100ppb ~ 4000ppm	100ppb ~ 5000ppm
500 $\mu\text{l}$	630-02591-05	638-60734-06	10 ~ 500 $\mu\text{l}$	50ppb ~ 1600ppm	50ppb ~ 2000ppm
1000 $\mu\text{l}$	630-02591-06	638-60734-07	20 ~ 950 $\mu\text{l}$	26ppb ~ 800ppm	26ppb ~ 1000ppm
2500 $\mu\text{l}$	630-02591-07	638-60734-08	50 ~ 2000 $\mu\text{l}$	320ppm max.	400ppm max.

6.1.3 The number of times to wash microliter syringe (NUMBER OF WASHES or NO OF WASHES)

Set the number of times to wash the microliter syringe. (The microliter syringe is washed by sucking standard solution or sample and discharging it.) Unless the microliter syringe is washed enough, the residue of the sample measured before produces a harmful effect (carry-over). On the other hand, if more number of times to wash the microliter syringe is set than necessary, it causes measuring time to prolong and the life of the plunger tip to shorten.

The number of times to wash the microliter syringe is set in the range from 0 (zero) to 9. The recommended times to wash each microliter syringe are as follows:

	Initial volume for washing
Microliter syringe of 250 $\mu\text{l}$ ——— 4 times	(250 $\mu\text{l}$ )
Microliter syringe of 500 $\mu\text{l}$ ——— 3 times	(500 $\mu\text{l}$ )
Microliter syringe of 1,000 $\mu\text{l}$ ——— 2 times	(950 $\mu\text{l}$ )
Microliter syringe of 2,500 $\mu\text{l}$ ——— 2 times	(2200 $\mu\text{l}$ )

These values are displayed on the right of this item on the screen as NORMAL in parentheses.

If the microliter syringe is washed the above times, the effect of the carry-over is within 1%.

Carry-over may be greater consideration when sample whose concentration is quite different from the former one is measured. In such a case, one more time of washing than the above number is enough. In the repetitive measurement, it is washed only one time on and after each measurement, irrespective of the number of times set. When replicate measurements are performed on the same sample aliquot, the washing volume between injections may be selected from among 1=Initial VOLUME, 2=100 $\mu$ l, 3=50 $\mu$ l and 4=0 $\mu$ l. This is useful when a smaller consumption of sample is desired. Confirm that there is no influence on the measurement value when using this function by performing repeat injections with the same sample to determine if each of the measurement values is stable. Generally, setting 0 (zero) time is not used. With this setting, sample of 2% of full stroke of microliter syringe is sucked and discharged only one time.

#### 6.1.4 Unit of concentration (UNIT OF CONC or UNIT)

Unit of concentration is selected among ppm, ppb, mg/l, and  $\mu$ g/l. ppm and ppb, and mg/l and  $\mu$ g/l are treated as the unit of different systems. Therefore, either unit system should be used all the time. If NO UNIT is selected, there is no unit of concentration.

#### 6.1.5 Automatic regeneration of IC solution

IC solution in IC reaction vessel, which has been acidified with IC reagent, converts IC components (carbonic and hydrogencarbonic ions) in sample into carbon dioxide. However, acid concentration in the IC solution gradually decreases due to reaction with IC components and dilution by the injected sample. If the acid concentration becomes too low, the IC components cannot be fully converted into carbon dioxide. (Since the sample injected in TC combustion tube is also finally drained into the IC reaction vessel, it will reduce the IC solution acid concentration in the same manner as the sample injected in IC reactor.)

Setting automatic regeneration of IC solution (AUTO REGENERATION OF IC SOLUTION) ON keeps the IC solution acid concentration at an appropriate level as follows:

- 1) After power supply, automatic regeneration of IC solution always takes place (IC reagent is added to IC solution) immediately before initial IC measurement. If the equipment is turned ON again within 24 hours after automatic regeneration, the following 2) occurs.
- 2) While power is kept ON, the quantities of samples injected (for TC, IC, and NPOC measurement and TC blank checking) and the number of injections are individually counted after automatic regeneration of IC solution. After either of the values exceeds the preset level, regeneration of IC solution is started automatically without delay just before initial IC measurement.
- 3) If peak ending in IC measurement is obviously delayed (T mark appears at the right of peak area value), automatic regeneration of IC solution takes place.

#### 6.1.6 Manner of printout

There are 3 modes in the manner of printout; automatic printout with numeral data only (DATA ONLY), automatic printout with numeral data and peaks (DATA & PEAKPLOT) and no automatic printout (OFF).

In the automatic printout modes, the data displayed on [DATA PROCESSING STANDARD] and [DATA PROCESSING SAMPLE] alone are printed out automatically in the termination of these screen before going to the next screen; automatic printout mode is selected for these screens because you cannot return to these screens once you have left them after termination.

Normally, either the automatic printout with numeral data only or no automatic printout is selected, and the necessary screen alone is printed out using the function keys on the screen.

The printout of peaks is necessary only in the special case such as troubleshooting or the like. Normally, it is not used because it takes a long printing time and needs many charts.

## 6.2 Measuring Procedure

Measuring operation is performed in the following procedure. For the functions on each screen and the details of the operation, refer to the explanation in Para. 6.3.

70-85 psi

- ① Supply carrier gas to TOC-5000 by pressure of 5-6 kg/cm<sup>2</sup>G from a carrier gas supply source (a high purity air cylinder).

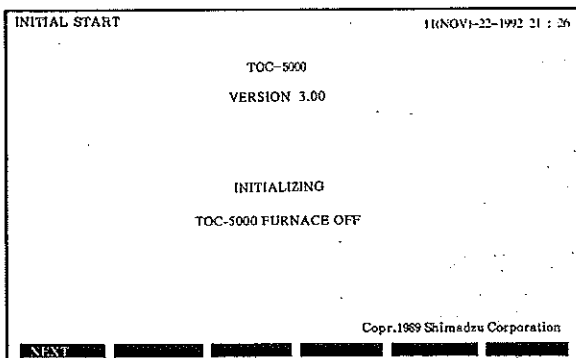
"For normal measurement"

"For measurement with ALL RESET START"  
For the first starting up after the installation or for starting up again after an accident

- ② Turn on the power switch on the right.

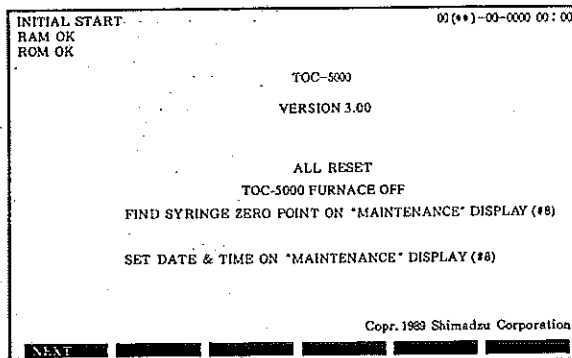
- ②-1 Turn on the power switch on the right, depressing [ENTER] key until "ALL RESET" is displayed on the screen.

③



Depress [NEXT] key, and go to [MAIN MENU] screen.

③-1



Depress [NEXT] key, and go to [MAIN MENU] screen.

- ④ Confirm that the pressure of carrier gas is 4-5 kg/cm<sup>2</sup>G and flow rate is 150 ml/min. Adjust them if necessary. Open the front door, and adjust the carrier gas pressure controller and the mass flow controller.

⑤

MAIN MENU		< GENERAL CONDITIONS >	
1	1 CALIBRATION	TC CATALYST	: NORMAL SENS
2	2 SAMPLE MEASUREMENT	SYR SIZE	: 250 $\mu$ l
3	3 GENERAL CONDITIONS	NO OF WASHES	: 4
4	4 CALIBRATION CURVE FILE LIST	UNIT	: ppm
5	5 DATA REPORT	AUTO RANGE	: ON
6	6 MONITOR	AUTO PRINT	: OFF
7	7 STANDBY OPTIONS	FURNACE	: OFF
8	8 MAINTENANCE	INJ SPEED	: STD
		ESU	: NOT USED
		CELL LENGTH	: STD

MOVE CURSOR TO ITEM OR INPUT ITEM: THEN PRESS [ENTER]

[RETURN]

⑤-1

MAIN MENU		< GENERAL CONDITIONS >	
1	1 CALIBRATION	TC CATALYST	: NORMAL SENS
2	2 SAMPLE MEASUREMENT	SYR SIZE	: *****
3	3 GENERAL CONDITIONS	NO OF WASHES	: *
4	4 CALIBRATION CURVE FILE LIST	UNIT	: ppm
5	5 DATA REPORT	AUTO RANGE	: ON
6	6 MONITOR	AUTO PRINT	: OFF
7	7 STANDBY OPTIONS	FURNACE	: OFF
8	8 MAINTENANCE	INJ SPEED	: STD
		ESU	: NOT USED
		CELL LENGTH	: STD

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

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

MOVE CURSOR TO ITEM OR INPUT ITEM: THEN PRESS [ENTER]

[RETURN]

Go to [GENERAL CONDITIONS] screen by either moving the cursor to the item number (#) or inputting the item directly, and depressing [ENTER].

⑤-2

GENERAL CONDITIONS	
TC CATALYST(TC CAT)	: [ ] (1=NORMAL SENS 2=HIGH SENS)
SYRINGE SIZE, $\mu$ l(SYR SIZE)	: 1 (1=250 2=500 3=1000 4=2500)
NUMBER OF WASHES	: 4 (0-9, NORMAL 4)
UNIT OF CONC(UNIT)	: 1 (1=ppm 2=ppb 3=mg/l 4= $\mu$ g/l 5=NO UNIT)
AUTO RANGING AND INJ VOL	: 1 (1=ON 2=OFF)
AUTO REGENERATION OF IC SOLUTION	: 1 (1=ON 2=OFF)
AUTO PRINTOUT	: 3 (1=DATA ONLY 2=DATA & PEAK PLOT 3=OFF)
FURNACE ON/OFF	: 2 (1=TOC 2=OFF)
BUZZER	: 1 (1=USED 2=NOT USED)
INJECTION SPEED	: 1 (1=STD 2=0.5mm 3=0.8mm)
ESU(OPTION)	: 2 (1=USED 2=NOT USED)
BUBBLE REMOVAL	: 2 (1=ON 2=OFF)
SYRINGE WASH, $\mu$ l	: 1 (1=STD 2=100 $\mu$ l 3=50 $\mu$ l 4=0 $\mu$ l)
CELL LENGTH	: 1 (1=STD 2=W-LONG 3=W-SHORT)
TOC OR SSM	: 1 (1=TOC 2=SSM)
PRINTER DEVICE	: 2 (1=EXTERNAL 2=INTERNAL)
PAGE LENGTH	: * (1=11in 2=12in 3=8.5in 4=70/8in(A4))
CALIBRATION CURVE FORM	: 1 (1=POLYGON 2=LEAST SQUARES)

[MAIN MENU] [PRINT]

Set "FURNACE" to "TOC". Check if other items have correct settings. If necessary, conduct setting again.

Then, call the [MAIN MENU] screen, and move the cursor to [#1 CALIBRATION] or input the item number (#) "1" and press [ENTER] key to bring about appearance of the CALIBRATION screen.

Press [numeric] and then [ENTER] keys to set conditions on respective items. Then, go to #8 [MAINTENANCE] screen via [MAIN MENU] screen.

(Note 1) Be sure to set TC catalyst (TC CAT) and the syringe size (SYR SIZE) mounted.

(Note 2) Be sure to heat up the TC furnace in state with the carrier gas flown.

MAINTENANCE FOR OPERATION <input type="checkbox"/> DATE & TIME ZERO POINT DETECTION OF SAMPLE SYRINGE PUMP REGENERATION OF TC CATALYST REGENERATION OF IC SOLUTION REGENERATION OF TC CATALYST & IC SOLUTION SPARGE GAS FLOW TC BLANK CHECK SAMPLE VOLUME COUNT/TC RESIDUE REMOVAL  FOR SERVICE KEYBOARD CHECK PRINTER CHECK MECHANICAL CHECK READY STATE SENS RS-232C I/F CHECK RANGE SET	
MAIN MENU	

Set date and hour (DATE & TIME SET), and carry out the ZERO point detection of the syringe pump (ZERO POINT DETECTION OF SAMPLE SYRINGE PUMP). Then, go to [MAIN MENU] screen and designate [CALIBRATION].

CALIBRATION / CONDITIONS		< GENERAL CONDITIONS >	
CAL CURVE F# : 6(SEE CAL FILE STATUS) TYPE : 1 (1-TC 2-IC) 1st STD CONC : 10.0ppm 2nd STD CONC : *****ppm 3rd STD CONC : *****ppm 4th STD CONC : *****ppm RANGE : x5 (x1 x5 x30) INJ VOL : 20µl (3-100) NO OF INJECTS : 1 (1-10) MAX NO OF INJ : 1 (1-10) SD : CV : SPARGE TIME : 0min(0-20)	TC CATALYST: NORMAL SENS SYR SIZE : 250µl NO OF WASHES: 4 UNIT : ppm AUTO RANGE : ON AUTO IC REGEN : ON AUTO PRINT : OFF FURNACE : TOC INJ SPEED : STD ESU : NOT USED	< CALIBRATION FILE STATUS > #1: PROTECT #10: FREE #2: PROTECT #11: FREE #3: PROTECT #12: FREE #4: PROTECT #13: FREE #5: STORED #14: FREE #6: FREE #15: FREE #7: FREE #16: FREE #8: FREE #17: FREE #9: FREE #18: FREE	
NEXT MAIN MENU PRINT			

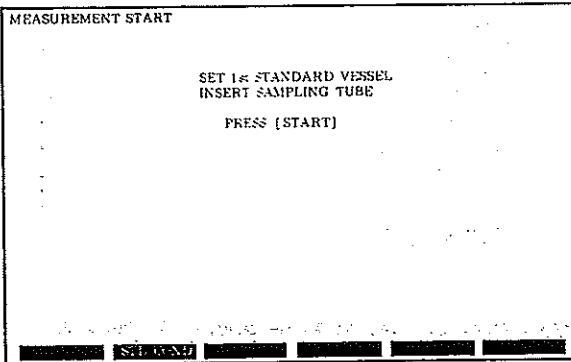
Set each condition, and go to [MEASUREMENT START] screen by [NEXT] (F1) key.

(Note)

Either go to the next screen after confirming that READY lamp flickers, or go to the next screen first and wait until it flickers there. (READY lamp normally starts flickering in 40-50 minutes after the power switch is on.)

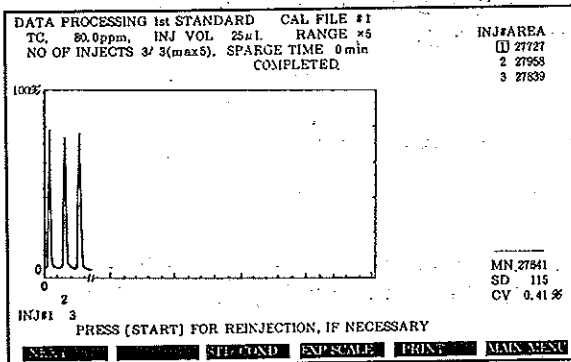
If READY lamp does not flicker, go to [MONITOR] screen, check the cause, and take an appropriate measure if necessary.

⑦



Put the first standard solution on the sample pan, insert the sampling tube with sampling needle into it, and then depress **START/STOP** key.

⑧



The standard solution is automatically measured under the conditions set on **CALIBRATION/CONDITIONS** screen. The progress of the measurement, a peak, and a peak area are then displayed. When the sign of completion (COMPLETED) is displayed, depress **NEXT** (F1) key.

When the additional measurement is to be performed after the completion of this measurement, depress **START/STOP** key.

(Note) Depress **PRINT** (F5) key to print out if necessary.

When two or more kinds of standard solution have been set on **CALIBRATION/CONDITIONS** screen (or for creating multipoint calibration curve), return to ⑦ **MEASUREMENT START** screen by **NEXT** (F1) key. Since the setting the second (2nd), third (3rd) or forth (4th) standard sample is instructed. The measurement is performed following this designation. When the measurement of every standard sample has been completed, go to **CALIBRATION CURVE** screen by **NEXT** (F1) key.



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**CALIBRATION CURVE**  
 CAL CURVE #1  
 TC 1-POINT  
 FILE NOT PROTECTED

SHIFT TO ORIGIN: [2] (1=YES, 2=NO)  
 PROTECT : 2 (1=YES, 2=NO)  
 CAL CURVE FORM : 1 (1=POLY, 2=LIST SQ)

**[CONDITIONS]**  
 TC CATALYST : NORMAL SENS  
 INJ VOL/SYR SIZE : 25/250 μl  
 RANGE : \*5  
 SPARGE TIME : 0 min  
 NO OF WASHES : 4  
 DATE : 06(JUN)-22-1989

**[DATA]**  
 STD CONC AREA #INJ SD CV  
 80.0ppm 27841 3 115 0.41%

STANDARD MAIN MENU VOID PRINT

Designate shifting of calibration curve to the zero point (SHIFT TO ORIGIN), the PROTECT processing to store calibration curve data (PROTECT), and the form of the calibration curve for three or four points calibration curve; polygonal or linear regression (CAL CURVE FORM), if necessary.

If another calibration curve is to be created after that (for example, if IC calibration curve is to be created following TC calibration curve), depress **[STANDARD]** (F1) key and return to **6** **[CALIBRATION/CONDITIONS]** screen.

When the sample is to be measured after that, go to **#2** **[SAMPLE MEASUREMENT]** screen via **[MAIN MENU]** screen.

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SAMPLE MEASUREMENT / CONDITIONS < CALIBRATION CURVE FILE LIST >

FILE	TYP	HIGH CONC	INJ/SYR	RG	CAT
1	TC	80.0 ppm	25/250		5 NORM
2	TC	400.0 ppm	30/250		30 NORM
3	TC	20.0 ppm	20/250		1 NORM
4	TC	240.0 ppm	8/250		5 NORM
5	TC	240.0 ppm	8/250		5 NORMNEW

SAMPLE# : 1

1st CAL CURVE# 1 \*\* \*\*  
 2nd CAL CURVE# 2 \*\* \*\*  
 3rd CAL CURVE# 3 \*\* \*\*

RANGE(\*1 \*5 \*30) 5 0 0  
 INJ VOL μl 25 50 50  
 SPARGE TIME min --- --- 1  
 NO OF WASHES 4 1 1  
 DIL FACTOR 1 1 1  
 NO OF INJECTS 3 1 1  
 MAX NO OF INJ 4 1 1  
 SD 100  
 CV % 1.0

NEXT MAIN MENU PRINT

Set each condition, and go to **[MEASUREMENT START]** screen by **[NEXT]** (F1) key.

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**MEASUREMENT START**

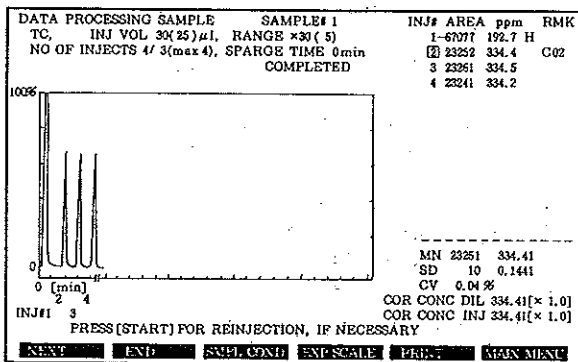
CYCLE MODE = [2] (1=ON 2=OFF)  
 NON-STOP MODE=2 (1=ON 2=OFF)

SET SAMPLE VESSEL  
 INSERT SAMPLING TUBE

PRESS [START]

SAMPL COND

Put the sample on the sample pan and insert the sampling tube with sampling needle into it, then depress **[START/STOP]** key.



The sample is measured under the conditions set on [SAMPLE MEASUREMENT/CONDITIONS] screen, and the progress of the measurement, a peak, and the result of the measurement are displayed. When the sign of completion (COMPLETED) is displayed;

- 1) If the measuring items or other samples to be measured remain, depress [NEXT] (F1) key. When measuring items remain, the present screen starts again, and the measurement for these items is performed. When measuring items do not remain, the screen returns to ⑩ [SAMPLE MEASUREMENT/CONDITION], and the number of the next sample is displayed. So, proceed to the measurement of the next sample.
- 2) When the measurement of every sample has been completed, depress [END] (F2) key to go to #5 [DATA REPORT] screen via [MAIN MENU] screen.

DATA REPORT PAGE1 07(JUL)-06-1989 13: 47

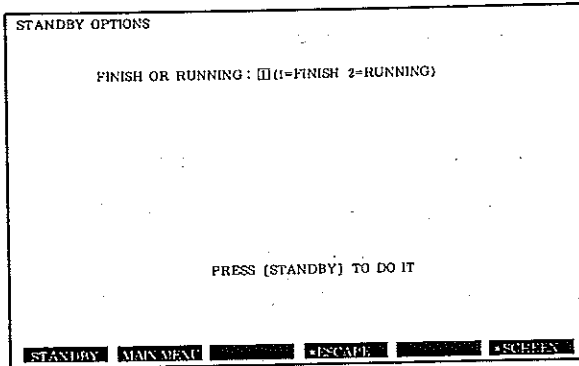
SPL#	TC	RMK	IC	RMK	TOC
1	14.23 ppm	1****	8.820 ppm	2****	5.412 ppm
2	11.13 ppm	1****	8.114 ppm	2RV* I	3.022 ppm
3	12.10 ppm	1****	9.457 ppm	2****	2.647 ppm
4	78.42 ppm	3****	42.51 ppm	4****	35.91 ppm
5	98.25 ppm	5****	41.32 ppm	6****	67.02 ppm
6	50.32 ppm	5****	28.14 ppm	6****	24.17 ppm
7	111.1 ppm	5RV*	55.53 ppm	6****	55.58 ppm

MAIN MENU NEXT PAGE BACK PAGE PRINT DATA CLEAR

As the all result of every measurement of sample is displayed, print it out by [PRINT] (F5) key.

As the display of the function key is changed to [DATA CLEAR] (F5) and [NO CLEAR] (F2) when [DATA CLEAR] (F6) key is depressed, depress [DATA CLEAR] key to delete the data displayed.

When operation of the equipment is to be terminated, go to #7 [STANDBY OPTIONS] screen via [MAIN MENU] screen.

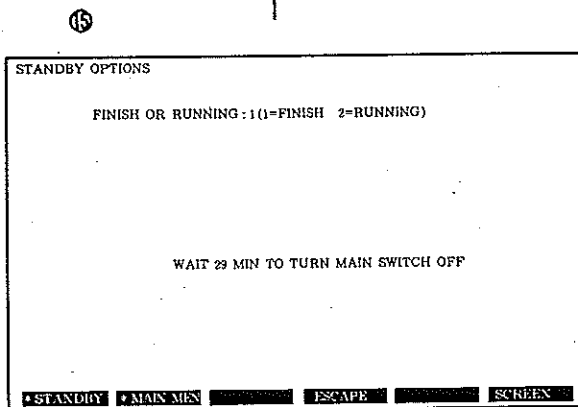


To stop the equipment, depress [1] + [ENTER], and then [STANDBY] (F1) key. In order to leave the equipment in the running state until the next use, depress [2] + [ENTER] key and proceed to the next step.

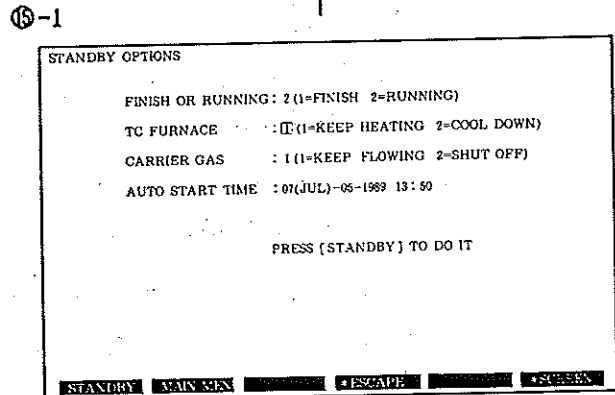
After measuring sample which contains large amount (about 1,000 ppm or more) of acid or salt, if equipment is left with sample remaining in sample injection needle, the needle may be corroded by sample or clogged with precipitated salt. To prevent this, execute TC and IC WASHING with sampling tube end put in water, using MECHANICAL CHECK function on [MAINTENANCE] screen. (See Fig. 5.4.)

"When terminating (FINISH)"

"When running (RUNNING)"



When the indication of the remaining time count before the power switch may be turned off becomes 0 (zero), turn off the power switch and the carrier gas supply.



Choose to use or to turn off the TC furnace and carrier gas during running, set the starting date and time for the next operation, and then depress [STANDBY] (F1) key. To ensure longer service of LCD, depress [SCREEN] (F6) key to halt LCD. LCD starts again if any of keys is depressed.

### 6.3 Screen Display, Operation by Keys and Function

In this paragraph, LCD screen and operation by keys are explained. Firstly, general items which are common in every explanation and operation are described.

1) If [MAIN MENU] screen is displayed, transition to another screen is performed by designating an item displayed as MENU. On other screens, it is performed by function keys displayed on the bottom of the screen. Keys from  to  on the keyboard correspond to the function keys on the screen and are depressed to execute them.

2) In the explanations, names of the keys and titles of the screen are expressed as follows:

The name of a key on the keyboard is put in :

Ex.

The name of a function key displayed on the screen is put in :

Ex.

The title of a screen is put in [ ]:

Ex. [MAIN MENU]

Message displayed on the screen is put in [ ]:

Ex. [PRESS [START] FOR REINJECTION]

When keys are operated successively, they are expressed, for example, as  + .

3) All key names in the messages on the screen are put in [ ].

The item number of MAIN MENU is denoted as (#3), for instance.

4) When an asterisk is attached with a function key on the screen, its function is invalid.

5) Note that positions and intervals of characters, numeric values and symbols and values of measuring conditions shown on drawings/diagrams in this manual may be different from those displayed actually on the screen.

# 【INITIAL START】

## 1. Contents of the display

When operation is started normally:

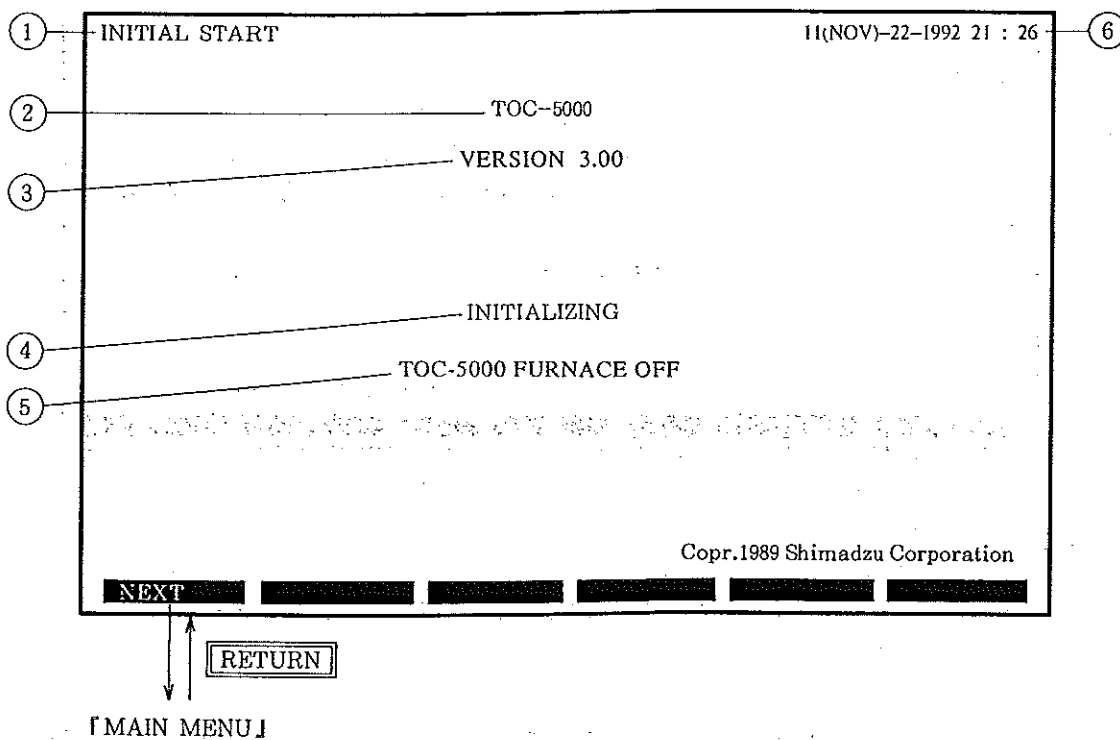


Fig. 6.1 【INITIAL START】 Screen (Normal Start)

When operation is started with ALL RESET:

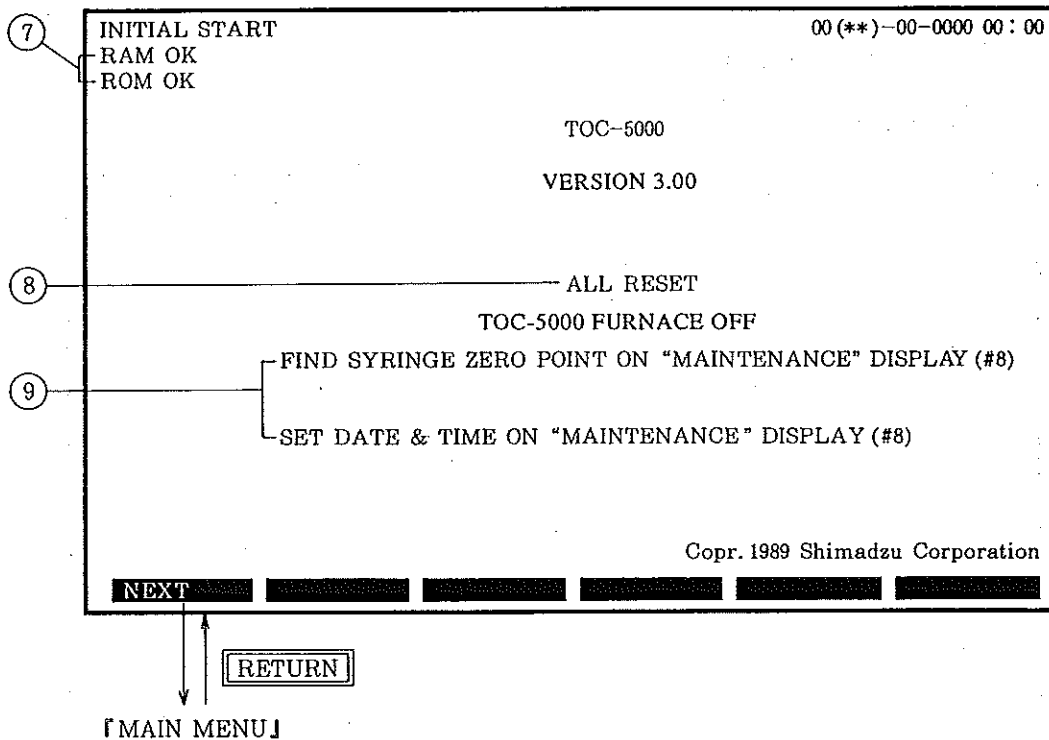


Fig. 6.2. INITIAL START Screen (ALL RESET start)

- ① The title of the screen is displayed on this position. In this case, the title is INITIAL START.
- ② The model name of this equipment is displayed.
- ③ The version number of ROM is displayed.
- ④ INITIALIZING: This indicates that check is under way if mechanically operating components such as the syringe pump, slide type sample injection port and solenoid valves are in respective home positions or if their transfer to home positions and detection of the syringe pump zero point are complete.  
INITIALIZED: This indicates completion of check operation described above.
- ⑤ TOC-5000 FURNACE OFF: This indicates that the TC furnace is in OFF position.  
TOC-5000 WARMING UP: This indicates that the TC furnace, electric dehumidifier and the NDIR are in warm-up before it is put in READY state.

TOC-5000 READY: This indicates that the temperature of TC furnace and electric dehumidifier, and base line position, fluctuation and noise are now ready for the measurement.

- ⑥ Date and hour are displayed. When the operation starts with ALL RESET, 0 (zero) is displayed.
- ⑦ The results of RAM and ROM checking are displayed. If an error is found, either RAM ERROR or ROM ERROR is displayed, and other messages are disappeared.

This message appears only immediately after power is supplied.

- ⑧ ALL RESET: If the power switch is turned on while **ENTER** is being pressed, the operation starts with ALL RESET and message ALL RESET is displayed. Or, if there is something abnormal about the memory content of RAM when the power switch is on, the operation starts with ALL RESET state automatically.

⑨ Messages

[FIND SYRINGE ZERO POINT ON "MAINTENANCE" DISPLAY(#8)]

Go to #8 MAINTENANCE, and execute the ZERO POINT DETECTION OF THE MICROLITER SYRINGE.

[SET DATE&TIME ON "MAINTENANCE" DISPLAY(#8)]

Go to #8 MAINTENANCE, set the date and hour.

2. Function

- 1) It is the first screen to be displayed when the power switch is turned on. The model name, version number of RAM, the results of RAM and ROM checking are displayed.
- 2) Initialization of mechanical parts (INITIALIZE), warming up of the equipment (WARMING UP), and confirming of the possible condition for the measurement (READY) are performed and displayed.
- 3) Messages of the necessary operation (instructions) to go to the next step are displayed.
- 4) The date and time of last cutoff of supply of power and those of supply of power are printed out when power is supplied again.

3. Operation

Purpose	Operation	Function
Go to the next step.	<b>NEXT</b>	Go to [MAIN MENU]. If started with ALL RESET, be sure to carry out the operation instructed by the messages first.

# [MAIN MENU]

## 1. Contents of the display

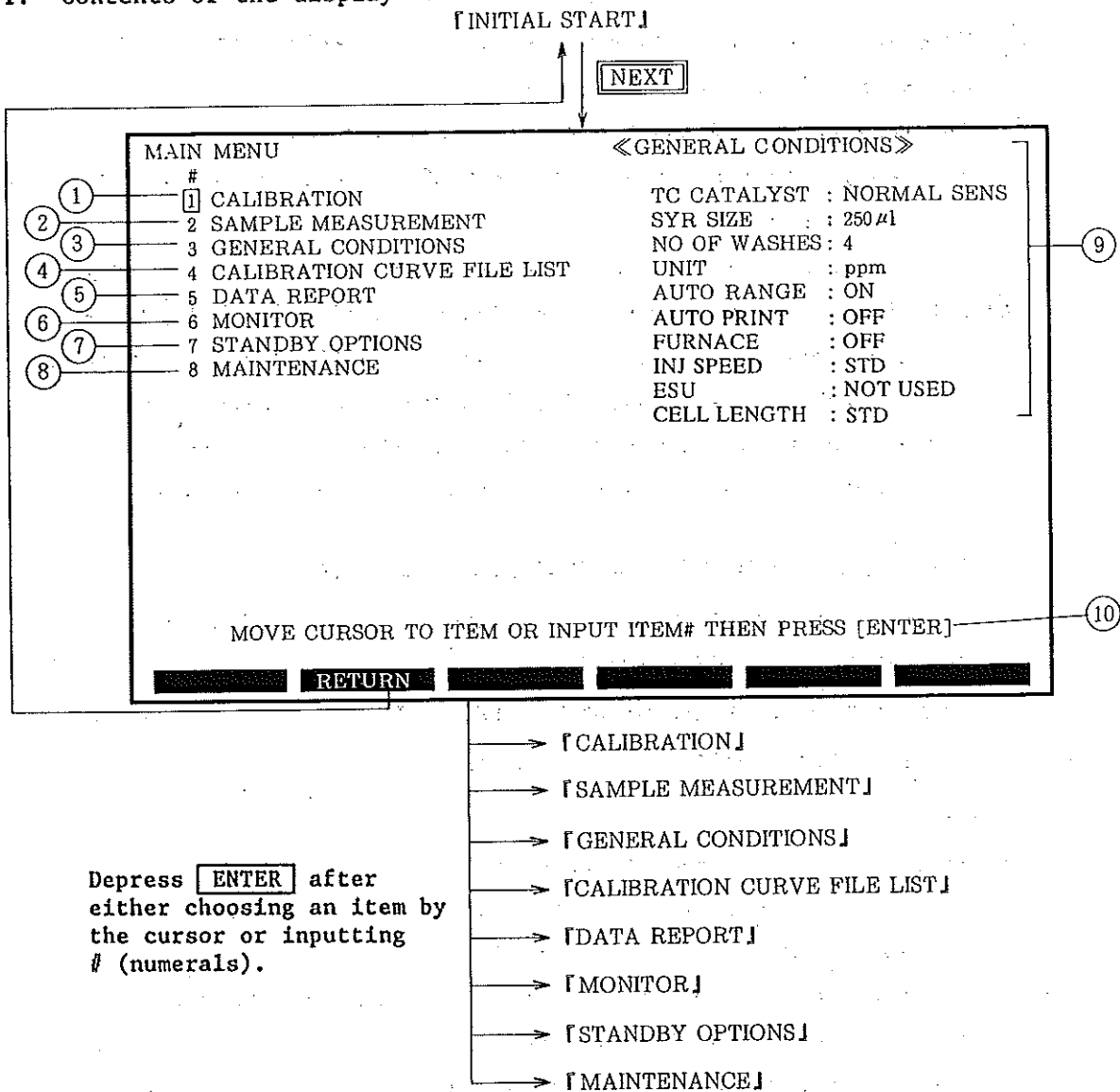


Fig. 6.3 [MAIN MENU]

- ① #1 CALIBRATION: To create a calibration curve
- ② #2 SAMPLE MEASUREMENT: To measure a sample
- ③ #3 GENERAL CONDITIONS: To set the general items for measurement
- ④ #4 CALIBRATION CURVE FILE LIST: To list up calibration curves stored
- ⑤ #5 DATA REPORT: To display the total results of measurement



- ⑥ #6 MONITOR: To display analog signals of NDIR, temperature of TC furnace and electronic dehumidifier
- ⑦ #7 STANDBY OPTIONS: To designate either termination or run in stopping the use of this equipment
- ⑧ #8 MAINTENANCE: Various kinds of functions and operations for the maintenance and service of the equipment are provided.
- ⑨ The general conditions for analysis set on 『GENERAL CONDITIONS』 are displayed. Confirm that they are set appropriately.
- ⑩ Messages (Instructions for the next operation are displayed.)  
『MOVE CURSOR TO ITEM OR INPUT ITEM# THEN PRESS [ENTER] 』

Either move the cursor to the item to choose or input the item number # (numerals) by key, and depress .

『FIND SYRINGE ZERO POINT ON "MAINTENANCE" DISPLAY(#8)』

『SET DATE&TIME ON "MAINTENANCE" DISPLAY(#8)』

Go to #8 MAINTENANCE and set zero point detection of the micro-liter syringe and date and hour. (This is displayed when the operation is started with ALL RESET.)

## 2. Function

Practically, every operation is started from this screen. When a series of work is completed and other work is started, or when other screen is referred to on the way of a work, go to the screen via this screen.

## 3. Operation

When the operation is started normally:

Purpose	Operation	Function
To choose an item	Depress <input type="button" value="ENTER"/> after either choosing an item or inputting # (numerals).	To go to the screen of the item chosen
To go to 『INITIAL START』	Depress <input type="button" value="RETURN"/> .	To return to 『INITIAL START』

When the operation is started with ALL RESET:

Purpose	Operation	Function
To go to 『GENERAL CONDITIONS』	Depress <b>ENTER</b> after either moving the cursor to #3 or inputting <b>3</b> .	The screen proceeds to 『GENERAL CONDITIONS』. Return to 『MAIN MENU』 after setting the condition of each item.
To go to 『MAINTENANCE』	Depress <b>ENTER</b> after either moving the cursor to #8 or inputting <b>8</b> .	The screen proceeds to 『MAINTENANCE』. Carry out zero point detection of the microliter syringe at ZERO POINT DETECTION OF SAMPLE SYRINGE PUMP after setting year, month, day, hour and minute at DATE & TIME SET.
To choose an item	Depress <b>ENTER</b> after either choosing an item by cursor or inputting # (numerals).	The screen proceeds to the item chosen.
To go to 『INITIAL START』	Depress <b>RETURN</b> .	The screen returns to 『INITIAL START』.

#### 4. Notice

- 1) The items of #1 CALIBRATION and #2 SAMPLE MEASUREMENT cannot be designated when operation is started with ALL RESET, because # is not displayed for these items; these operations can be executed only after the microliter syringe size is designated at GENERAL CONDITIONS and its zero point detection is completed.
- 2) Be sure to make the settings for the kind of TC catalyst and size of the microliter syringe correspond with those actually attached in the equipment. If they are different from the actual ones, not only accurate measurement cannot be carried out but TC combustion tube and parts around it may be damaged.

# 【CALIBRATION/CONDITIONS】

## 1. The contents of the display

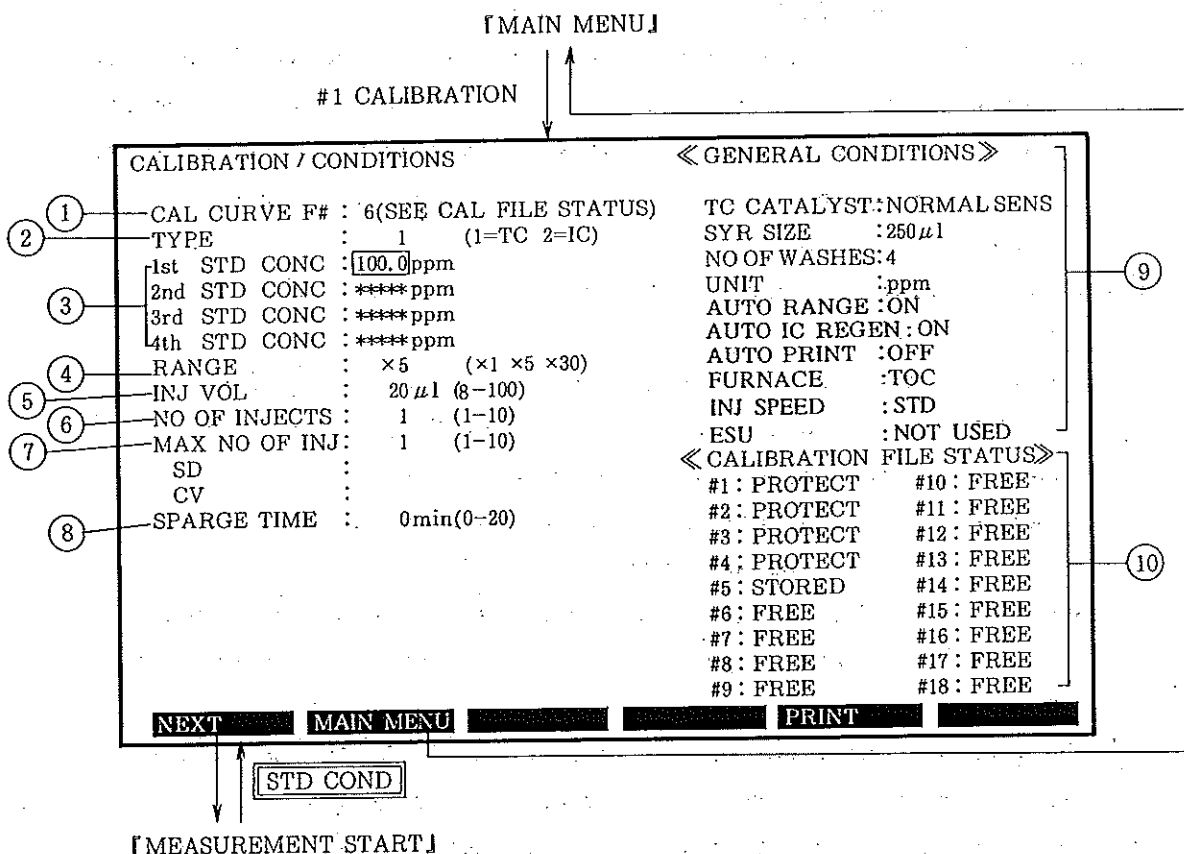


Fig. 6.4 【CALIBRATION/CONDITIONS】

- ① CAL CURVE F#: The number of CALIBRATION CURVE FILE. Up to 18 calibration curves can be stored. When they are stored and read, the number is given for their identification.
- ② TYPE: Designate TC or IC. TC calibration curve should be used for NPOC measurement.
- ③ STD CONC: Concentration of standard solution is to be set. Up to four kinds of standard solution can be set. The maximum concentration to be set is shown in Table 6.1, in accordance with a size of the microliter syringe to be used (or, the minimum injection volume).

- ④ RANGE: The range of NDIR is to be set. The optimum combination of range and sample injection volume is determined according to the maximum concentration of the standard solution set at ③ STD CONC, and displayed automatically.
- ⑤ INJ VOL: The injection volume is to be set. Numeral(s) in parentheses on the right denotes possible injection volume to be set. The injection volume is determined according to the size of the microliter syringe and the kind of TC catalyst to be set at GENERAL CONDITIONS.
- ⑥ NO OF INJECTS: The number of times of repetitive measurement is to be set.
- ⑦ MAX NO OF INJ: The same number as the one set at ⑥ NO OF INJECTS is set automatically. When the "automatic measurement to replace abnormal value" function is used, larger number than NO OF INJECTS must be set, and appropriate values must be set for the subsequent SD and CV.
- ⑧ SPARGE TIME: The sparging time is to be set when the sparging treatment by carrier gas is performed to standard solution in TC measurement.
- ⑨ The general conditions of analyses set at [GENERAL CONDITIONS] are displayed. Confirm that they are set properly.
- ⑩ The condition of calibration curve storage file is displayed. FREE denotes that the file is empty, STORED denotes that curves are stored and PROTECT denotes the stored curves are protected against writing.
- ⑪ Messages
- [GENERAL CONDITIONS MISMATCH TC CAT (or SYR SIZE, UNIT, NO OF WASHES) GO TO "CALIBRATION CURVE FILE LIST" DISPLAY (#4), AND [CLEAR FILE]]
- The general conditions do not correspond with the settings.
- [CONCENTRATION IS TOO HIGH]
- Concentration of standard solution is too high.

## 2. Function

- 1) Measurement conditions to create a calibration curve of standard solution are set by the instrument or the operator.
- 2) The general conditions of analyses set at GENERAL CONDITIONS and the condition of calibration curve storage file are displayed.

### 3. Operation

Purpose	Operation	Function
To set conditions	Depress <b>[ENTER]</b> after inputting numeral(s) for each item.	To set conditions for every item. The optimum range and injection volume are set according to the maximum concentration of standard solution: They are changeable. Use the calibration number indicated FREE. The "automatic measurement to replace abnormal value" function becomes available by setting larger number to MAX NO OF INJ than to NO OF INJ. → Automatic Setting of Measuring Conditions (Refer to Para. 3.13.) → Automatic Measurement to Replace Abnormal Value (Refer to Para. 3.11).
To print out	Depress <b>[PRINT]</b> .	The contents of CALIBRATION/CONDITIONS alone are printed out.
To go to the next step	Depress <b>[NEXT]</b> .	The screen goes to <b>[MEASUREMENT START]</b> .
To go to <b>[MAIN MENU]</b>	Depress <b>[MAIN MENU]</b> .	The screen returns to <b>[MAIN MENU]</b> .

### 4. Notice

- 1) Be sure to depress **[ENTER]** key after inputting numerals to each item. When the cursor is moved to other item without this key operation, the old value is set for the item.
- 2) When FLOW is designated for the item of SPARGE GAS FLOW on **[MAINTENANCE]** screen, 0 (zero) only can be set to SPARGE TIME (the sparging time). If calibration curves for which SPARGE TIME has been set are read out, they cannot be used.

【MEASUREMENT START】

1. Contents of the display

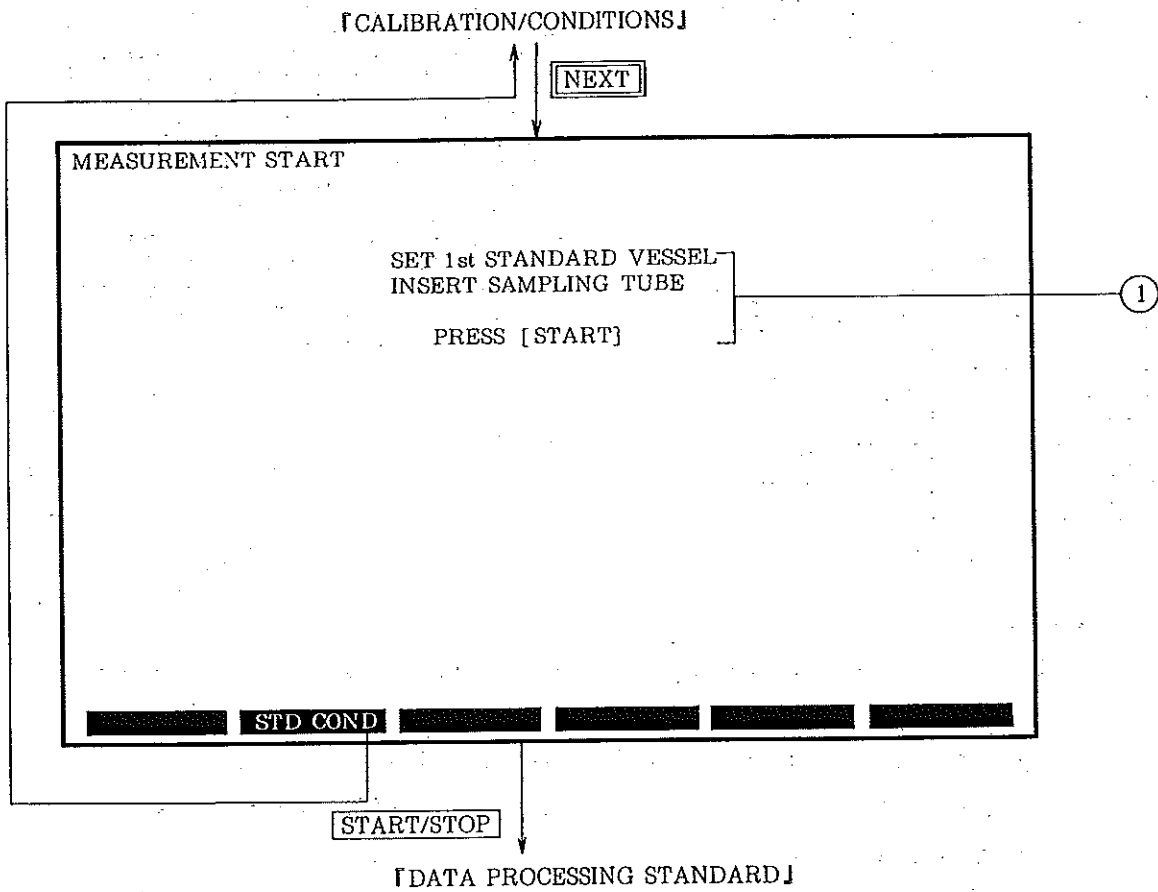


Fig. 6.5 【MEASUREMENT START】 Screen (at CALIBRATION)

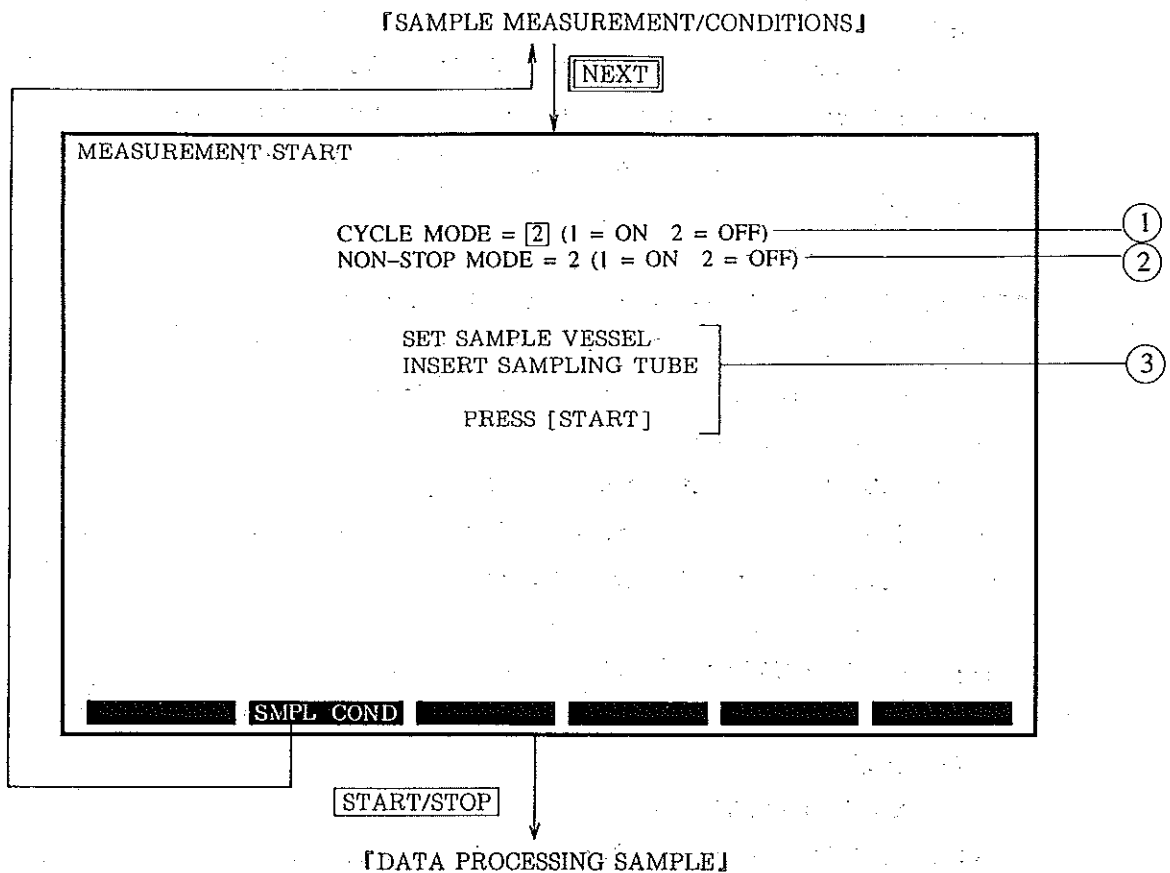


Fig. 6.6 『MEASUREMENT START』 Screen (at SAMPLE MEASUREMENT)

- ① Continuous injection of a sample is performed by setting the CYCLE MODE to ON until the [ESCAPE] is executed to exit the cycling measurement mode. This function may be used to reduce "blank peaks" or continuous sample measurement.
- ② NON-STOP MODE: In the case of multiple measuring items preset for 『SAMPLE MEASUREMENT/CONDITIONS』, measurement is sequentially taken beginning with the item displayed at extreme left, when NON-STOP MODE is put to "ON". If "OFF", job stops on 『DATA PROCESSING SAMPLE』 screen after end of measurement of each item. Press **NEXT** key to start measurement of the succeeding item. The NON-STOP MODE is mainly used for calculation of TOC contents by means of continuous measurement of TC and IC.

③ Messages

「SET 1st STANDARD VESSEL. INSERT SAMPLING TUBE. PRESS (START) 」

Put the first standard solution on the sample pan, and insert the sampling tube with sampling needle to it, then depress

**START/STOP**.

(For CALIBRATION)

「SET SAMPLE VESSEL. INSERT SAMPLING TUBE. PRESS (START) 」

Put sample on the sample pan, and insert the sampling tube with sampling needle to it, then depress **START/STOP**.

(For SAMPLE MEASUREMENT)

「STANDARD CONC OF PREVIOUS DISPLAY IS NOT YET SET」

Concentration of the standard solution is not set on the previous screen (「CALIBRATION/CONDITIONS」).

(For CALIBRATION)

「RANGE VALUE OF PREVIOUS DISPLAY IS NOT YET SET」

Range is not set on the previous screen (「SAMPLE MEASUREMENT/CONDITIONS」).

(For SAMPLE MEASUREMENT)

「FIND SYRINGE ZERO POINT ON "MAINTENANCE" DISPLAY(#8)」

Carry out automatic zero point detection of the microliter syringe on 「MAINTENANCE」 screen.

2. Function

This is the screen for starting either standard solution measurement (CALIBRATION) or sample measurement (SAMPLE MEASUREMENT). Necessary instructions for the operation to start measurement are displayed.



### 3. Operation

Purpose	Operation	Function
Setting of CYCLE MODE	[1] → [ENTER]	Measurement continues until [ESCAPE] is pressed on [DATA PROCESSING SAMPLE] screen.
To set NON-STOP MODE	[1] → [ENTER]	If multiple measuring items are set, measurement of each item is made sequentially. This mode is used for calculation of TOC content by means of continuous measurement of TC and IC.
To start measurement	[CALIBRATION] After setting standard solution, press [START/STOP].	Measurement of the standard solution starts.
	[SAMPLE MEASUREMENT] After setting sample, press [START/STOP].	Measurement of a sample starts.
To return to [CALIBRATION/CONDITION] screen	[Screen of [MEASUREMENT START] using 1st standard solution] [STD COND]	Goes back to [CALIBRATION/CONDITIONS] screen.
	[Screen of [MEASUREMENT START] using 2nd-4th standard solution] [1st START] ↓	Goes back to [MEASUREMENT START] (for 1st standard solution) screen.
	[STD COND] (1st standard solution [MEASUREMENT START] screen).	Goes back to [CALIBRATION/CONDITIONS] screen.
	[SAMPLE MEASUREMENT] [SMPL COND]	Goes back to [SAMPLE MEASUREMENT/CONDITIONS] screen.

### 4. Notice

- 1) Attach the gas sparging tube when sparging treatment is carried out with SPARGE TIME set.
- 2) When plural types are set on the "SAMPLE MEASUREMENT/CONDITIONS" screen, NON-STOP MODE also turns to ON automatically, if CYCLE MODE is put to ON.

# 『DATA PROCESSING STANDARD』

## 1. Contents of the display

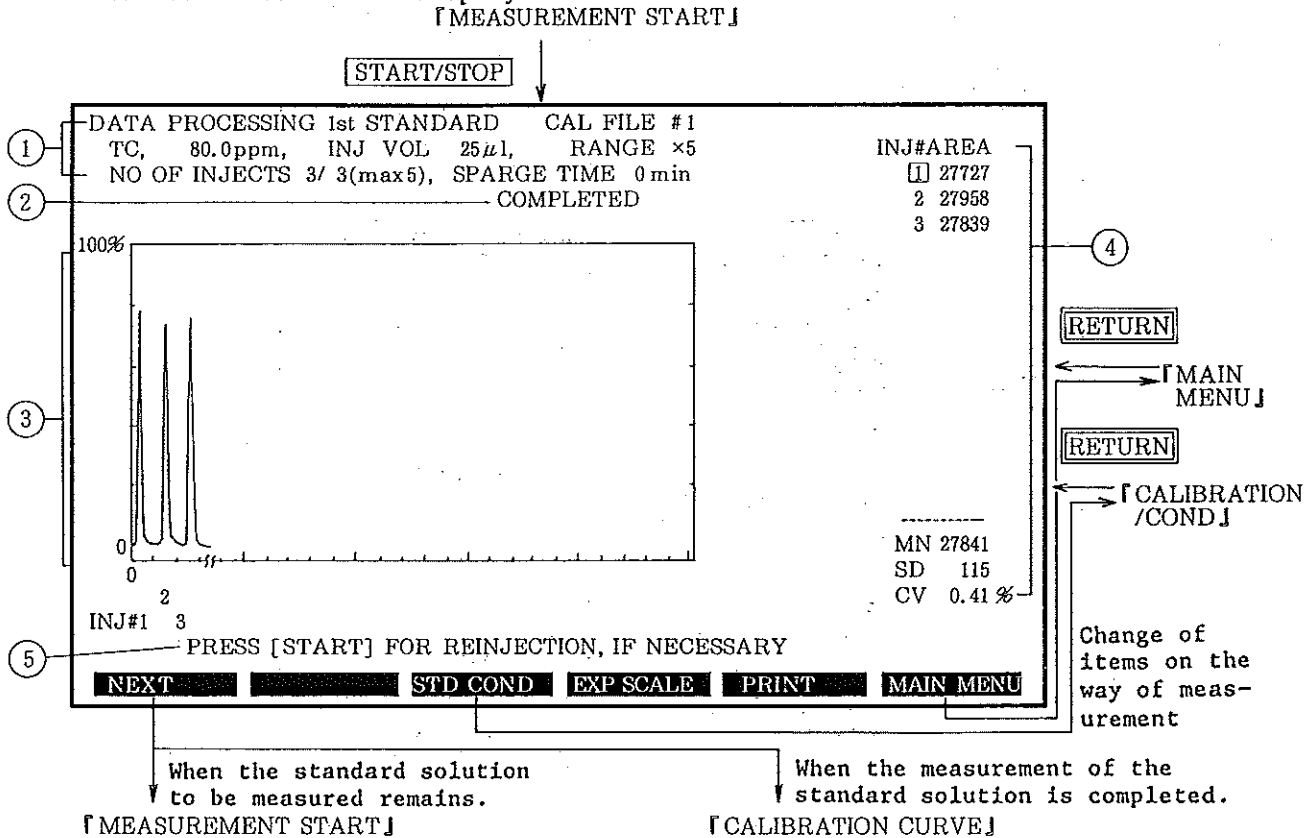


Fig. 6.7 『DATA PROCESSING STANDARD』 Screen

- ① The outline of the measurement conditions set at 『CALIBRATION/CONDITIONS』 is displayed. NO OF INJECTS (the repeating number) denotes the number of times of measurement finished.
- ② The progress of the measurement program is displayed as follows:
  - SPARGING XX min to end: When SPARGE TIME is set, the remaining time is displayed during sparging treatment.
  - WASHING : Washing of the microliter syringe by sample
  - SAMPLING : Measuring off sample
  - INJECTING: Injection of sample
  - MEASURING: Detection of a peak and measurement of the area

- WAITING: 1) Standby due to unsatisfied measurement conditions  
(READY STATE SENS)
- 2) Displayed when **START/STOP** key is depressed during SPARGING, WASHING, OR SAMPLING

COMPLETED: The set times of measurement is completed.

MEASURING/WASHING: Washing motion for the next injection starts overlapping peak processing.

- ③ A peak is displayed. The time display along the transversal axis (in minutes) stops when the set times of measurement is completed. For additional injection (REINJECTION), time is not displayed.
- ④ Injection No. (INJ#) and peak area (AREA) as well as mean value (MN), standard deviation (SD), and coefficient of variation (CV) calculated with peak area data obtained at that time are displayed.
- ⑤ Message  
[PRESS (START) FOR REINJECTION, IF NECESSARY]  
Depress **START/STOP** if additional measurement is necessary.

## 2. Function

The progress of the measurement of standard solution, a peak, the outline of the measuring conditions and the result of the measurement (peak area) are displayed.

## 3. Operation

Purpose	Operation	Function
Delete/retrieve measured value	Designate INJ# by cursor, and depress <b>ENTER</b> .	"-" displayed at deleted INJ#, and MN, SD, and CV values are recalculated/ "-" removed at INJ#, value is retrieved and all values are recalculated.
Additional measurement	Depress <b>START/STOP</b> after COMPLETED is displayed.	The additional measurement is performed, and the measurement value is displayed with the sign of +.
To change conditions during measurement	Go to [CALIBRATION /CONDITIONS] by depressing <b>[STD COND]</b> , change conditions, and then return to this screen by <b>[RETURN]</b> .	The repeating number (NO OF INJECTS), the maximum repeating number (MAX NO OF INJ), SD, and CV can be changed. Concentration of standard solution which has not been set yet can be set; however, the lower concentration than ones already set only can be set.

Purpose	Operation	Function
To halt or terminate the measurement halfway	<p><b>START/STOP</b></p> <p>(The function keys change as shown in Fig. 6.8.)</p> <ul style="list-style-type: none"> <li>→ <b>ESCAPE</b></li> <li>→ <b>CONTINUE</b></li> <li>→ <b>PEAK STOP</b></li> </ul>	<p>WAITING is displayed, and the equipment is in the standby state. During WASHING or SAMPLING, the equipment is put in the standby state after the microliter syringe goes to either the zero position or a measuring position. During MEASURING, peak processing at this time is executed.</p>
		<p>The screen returns to 『MEASUREMENT START』, and measurement values displayed on this screen are lost. (The operation returns to re-start.)</p>
		<p>The standby state is cancelled, and measuring work is continued.</p>
		<p>It can be used only when MEASURING is displayed. At this time, measurement of a peak area is completed and the result is displayed.</p>
To magnify the display of a peak	<p><b>EXP SCALE</b></p> <p>↑</p> <p><b>STD SCALE</b></p>	<p>The height of the peak display is magnified four times (0~25% F.S).</p> <p>The magnified display changes back to the original size (0~100% F.S).</p>
Print-out	<p><b>PRINT</b> (The function keys change as shown in Fig. 6.9.)</p> <ul style="list-style-type: none"> <li>→ <b>ALL PRINT</b></li> <li>→ <b>DATA PRINT</b></li> <li>→ <b>PEAK PRINT</b></li> <li>→ <b>NO PRINT</b></li> </ul>	<p>All display on the screen is printed out.</p> <p>All display except the peak display is printed out.</p> <p>The peak display alone is displayed.</p> <p>Print-out is halted.</p>
To go to 『MAIN MENU』	<b>MAIN MENU</b>	<p>It is possible to go to #3 to #6 screen via 『MAIN MENU』, but the set items are not changeable on these screens. Be sure to return to 『DATA PROCESSING STANDARD』 screen to go to the next step.</p>
To go to the next step	Depress <b>NEXT</b> after <b>COMPLETED</b> is displayed.	<p>The screen goes to the next screen, 『CALIBRATION CURVE』, when the measurement of every standard solution set at 『CALIBRATION/CONDITIONS』 is completed. When standard solution to be measured remains, the screen returns to 『MEASUREMENT START』.</p>

4. Notice

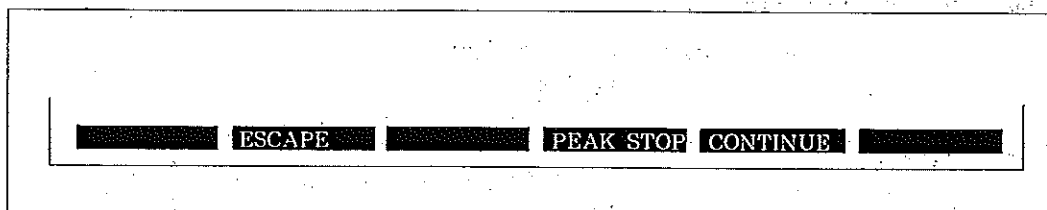


Fig. 6.8 The Display of the Function Keys in Halting the Operation on the Way of Measurement

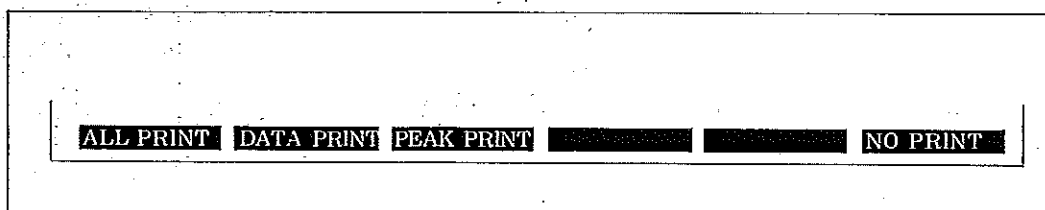


Fig. 6.9 The Display of the Function Keys in Designating Printout

- 1) It must be noted that you cannot return to this screen if you have proceeded to the next step by depressing **NEXT**.
- 2) If large amount of sample (300  $\mu$ l or more) is injected for TC or NPOC measurement, asterisk \* is displayed at **ESCAPE** and **PEAK STOP** for about 1.5 minutes following the sample injection, indicating that these function keys do not work.
- 3) Execute the following operation, when suspension is intended of measurement using 2nd-4th standard solution for multiple-point calibration curve.

Press **START/STOP** key.



MEASUREMENT START for 2nd-4th standard solution



Press **1st STD** (F5) key.

MEASUREMENT START for 1st standard solution



Press **STD COND** (F2) key.

CALIBRATION/CONDITIONS

Note that all values measured for the multiple-point calibration curve are deleted.

# [CALIBRATION CURVE]

## 1. Contents of the display

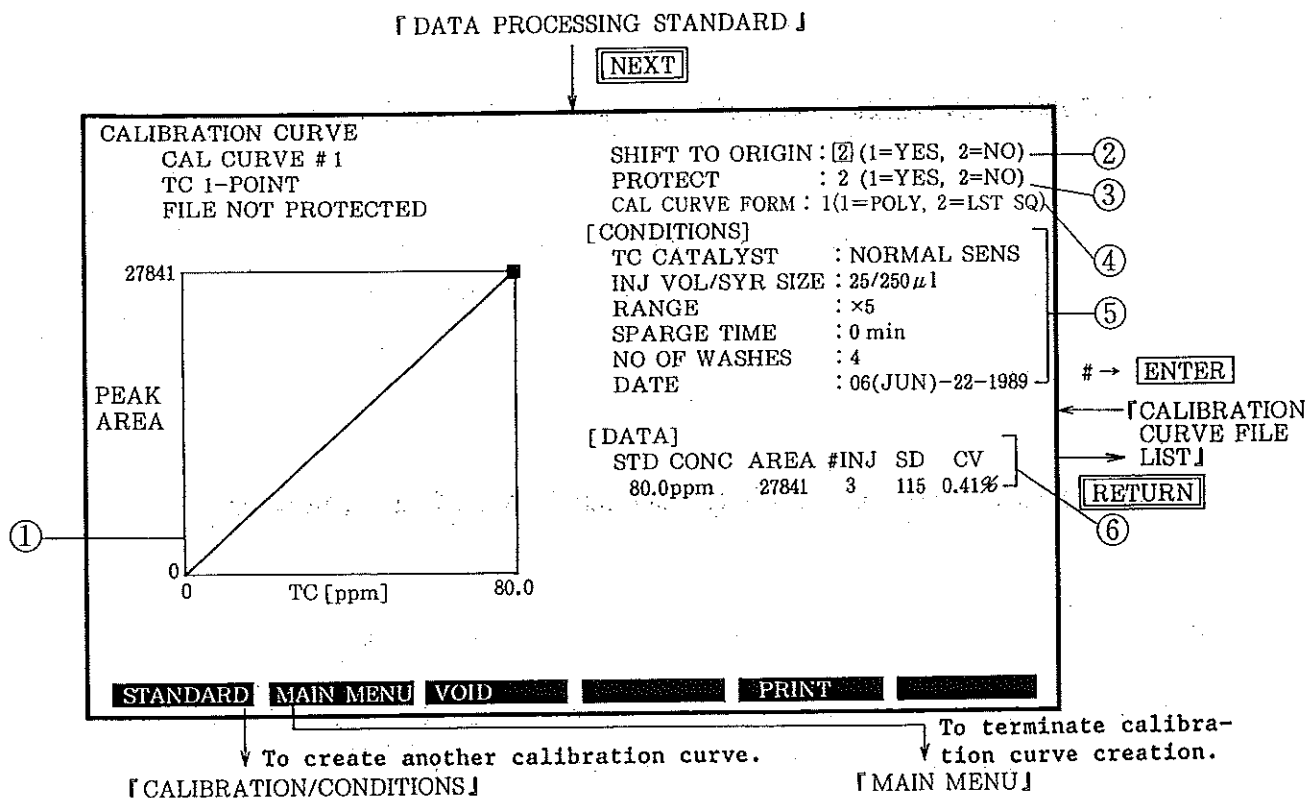


Fig. 6.10 [CALIBRATION CURVE]

- ① The display of a calibration curve.
- ② If YES is designated, a calibration curve moves vertically to pass the origin.
- ③ If YES is designated, PROTECT processing is performed to store calibration curve data, and the left display changes to FILE PROTECTED.
- ④ For the three or four points calibration curve, the polygonal form (1=POLY) or the linear regression form (2=LST SQ) can be created.
- ⑤ Main measurement conditions including GENERAL CONDITIONS are displayed.
- ⑥ The measurement result of each standard solution (the mean value, SD, and CV) is displayed.
- ⑦ Message  
"FAILED CALIBRATION CAL CURVE WAS NOT MADE"  
If measurement values of standard solutions produce calibration curve of reverse slope, calibration curve is not made. Check if standard solutions are switched one another, or erroneously prepared.