
Instruction Manual

Pi500 Process Ion Chromatograph CM500 Clean Room Gas Monitor Operation Guide

IM 12Y4H1-E

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1. WARRANTY

1.1 Scope of Warranty

The warranty period and scope of warranty for this system are as follows:

Warranty period

One (1) year after delivery

Scope of warranty

If, during the warranty period, any defects in materials or workmanship cause system trouble and are acknowledged by the manufacturer, the system will be repaired at no cost to the user. However, repair costs are excluded from warranty and will be charged to the user for any of the following:

Exclusions

- 1 Trouble due to inappropriate handling or operation. Extra care is particularly required when handling the columns.
- 2 Trouble resulting from handling, operation, or storage of the system that is contrary to the required design, installation, or operating conditions.
- 3 Trouble due to normal or natural consumable parts or materials. Or liquid supply failures due to pump plunger seal wear, filter clogging, or check valve clogging.
- 4 Normal or natural consumable parts and materials and parts whose normal product life is limited. The following parts, for example, are excluded from warranty:
 - a) Parts brought into contact with the liquids, such as: plunger seals, other seals, and tubing parts
 - b) Consumable parts such as: ferrules, bolts and nuts, filters, columns, syringes, and spare parts
- 5 Trouble due to modification or repair by the user or any third party other than those authorized by the manufacturer
- 6 Trouble caused by using parts other than those authorized by the manufacturer
- 7 Trouble caused by factors not directly related to the system, including: fire, flood, earthquake, and other factors beyond human control
- 8 Other trouble not attributable to the manufacturer

1.2 Definitions of Notes, Cautions, and Warnings

Notes, cautions, and warnings appear throughout this guide and should be read carefully to avoid potential hazards and system damage. These notes, cautions, and warnings are defined below.



NOTE

: Gives suggestions/hints or cautionary information on operations.



CAUTION

: Indicates any situation or practice which, if not detected and/or strictly remedied, may result in bodily injury or system damage.



WARNING

: Indicates any situation or practice which, if not detected and/or strictly remedied, may result in serious bodily injury or death.

1.3 Other Cautionary Information



NOTE

The contents of this manual and those of the programs within the system are protected under the relevant copyright law. They may not be reproduced or applied to other purposes, without prior written consent from the publisher.

The contents of this manual and the programs within the system are subject to change at any time without prior written notice.

The screen displays shown in this manual may differ from the actual displays.

The manufacturer takes no responsibility for trouble arising from the use of this manual or from the operation of the system.

2. HANDLING PRECAUTIONS

This section outlines the handling precautions required for the system. Potential hazards of the more dangerous chemicals used are also explained.

2.1 Handling the System



WARNING

The system contains electrically sophisticated circuits and high-voltage sections. Mere removal of the rear or top covers while the power is on may cause electric shock.

If it is absolutely necessary to remove the cover or other sections for replacement purposes, etc., turn off the power and unplug the power cord beforehand.

2.2 Handling of Chemicals

Many of the chemicals used in the system are dangerous and are classified as toxic substances. The typically used chemicals are listed below. Pay careful attention to ventilation, liquid waste disposal, as well as to the prevention of leakage, et cetera.

| Chemical | Hazards |
|------------------------|---|
| Nitric acid | Oxidizing, harmful, and potentially corrosive. A toxic substance categorized as a Class-VI Dangerous Substance |
| Sulfuric acid | Harmful, and potentially corrosive |
| Toxic sodium hydroxide | Oxidizing, harmful, and potentially corrosive. A toxic substance categorized as a Class-VI Dangerous Substance |
| Ethylene diamine | Irritating, harmful, and potentially corrosive. #2 petroleum categorized as a Class-IV Dangerous Substance |
| Sodium nitrite | Toxic |
| Sodium nitrate | Class-I dangerous substance |

3. ACCESSORIES CHECK

3.1 Unpacking

Check the packages for obvious defects. If there are any damaged or missing parts, please contact your Yokogawa sales representative.

3.2 Standard Accessories List

| Accessory | Qty. | Remarks |
|-------------------------|------|---------|
| Liquid waste reservoir | 1 | 20L |
| Eluant reserve-tank | 1 | 10L |
| Pure water reserve-tank | 1 | 10L |
| Instruction manual | 1 | |

4. SYSTEM COMPONENTS AND THEIR FUNCTIONS

4.1 Pi500 Components

| No. | Part name | Function/Description | See Figure: |
|-----|------------------------------|--|-------------|
| 1 | Power switch | | 4.1 |
| 2 | Analyzer run/stop switch | Starts or stops the analyzer. | |
| 3 | Alarm reset switch | Resets alarms (except for analysis data alarms). | |
| 4 | LED status indicators | Indicate the normal operating or alarm status of the system. | |
| 5 | System check solenoid valve | | 4.2 |
| 6 | Degassing unit | Degassing unit for eluant (EL) | |
| 7 | Concentration valve | | |
| 8 | Oven | Contains a separation column and a detector. | |
| 9 | Sample supply pumps | | |
| 10 | Sample inlet pump | Admits a sample into the sample supply pumps. | |
| 11 | Line filter | | |
| 12 | Eluant (EL) inlet valve | Admits EL into the EL pumps. | |
| 13 | System check valve | | |
| 14 | Priming pump | | |
| 15 | Eluant (EL) pumps | | |
| 16 | Oven door | Column outlet port | 4.3 |
| 17 | Mounting screw for oven door | | |
| 18 | Input/output terminal plate | See Fig. 4.8. | 4.4 |
| 19 | DCS communication port | | |
| 20 | PC communication port | For connection to Workstation | |
| 21 | Power connector | 100 V AC at 50/60 Hz | |
| 22 | Power switch | | |

4.2 CM500 Components

| No. | Part name | Function/Description | See Figure: |
|-----|----------------------------------|---|-------------|
| 23 | Power switch | | 4.5 |
| 24 | Alarm/reset switch | Resets the analysis data alarm. | |
| 25 | Emergency stop switch | Stops the system in emergencies. | |
| 26 | Pi500 | | 4.6 |
| 27 | Pure water circulation unit | | |
| 28 | Drain tank | | |
| 29 | Pure water tank | | |
| 30 | Flowmeter 1 | Provided only for stream selection. | |
| 31 | Flow controller | Controls the flow rate of the sample gas supplied. | |
| 32 | Flowmeter 2 | | |
| 33 | Scrubber unit | Absorbs the sample gas into pure water. | |
| 34 | Hand-operated valve | Changes the sample to a standard liquid, or vice versa. | |
| 35 | Solenoid valve | | |
| 36 | Overflow tank | Supplies the scrubber unit with pure water. | |
| 37 | Eluant (EL) tank | | 4.7 |
| 38 | Sample inlet ports | | |
| 39 | Fuses | | |
| 40 | Input/output terminal plate | See Fig. 4.9. | |
| 41 | Power switch | | |
| 42 | Power terminal | | |
| 43 | Electric leakage circuit breaker | | |

4.3 Input/Output Terminals

The input/output terminals have covers. Remove the covers before wiring. Route external signal lines through the opening in the lower section of terminal covers.

(A) Pi500 terminal functions

See Fig. 4.8 for the following terminals:

D/I terminals

These are input-contact terminals. These terminals accept only voltage-free contacts.

The system's operational status, pattern status, and so on can be specified via the D/I terminals.

D/O terminals

These are output-contact terminals. These terminals accept only voltage-free contacts.

COMON is separated for each signal terminal. Connect terminals for use as common terminals.

Alarm, timing, operational status, and measurement pattern status signals can be output via the D/O terminal.

PC COMMUNICATION PORT (D-SUB nine-pin connector)

This RS-232C terminal is provided for data exchange with a personal computer.

DCS COMMUNICATION PORT (D-SUB nine-pin connector)

This RS-232C terminal is provided for data exchange with a supervisory computer.

(B) CM500 terminal functions

See Fig. 4.9 for the following terminals:

Alarm Buzzer terminals

These terminals are for sounding an alarm buzzer. A voltage of +24 V DC is applied to these terminals.

These alarm buzzer terminals are not usually mounted.

Alarm Light terminals

These terminals are for activating an alarm light. A voltage of +24 VDC is applied to the terminals.

These alarm light terminals are not usually mounted.

Retry Measurement terminals

These voltage-free contact output terminals are activated if abnormal measurement results are obtained and measurement repeated for the same stream.

Alarm terminals

These voltage-free contact output terminals are activated if an alarm of a high level occurs.

Measurement Stream Bit terminals

These voltage-free contact output terminals represent measurement streams in bits.

| Measurement stream | Bit0 | Bit1 | Bit2 | Bit3 | Bit4 |
|--------------------|------|------|------|------|------|
| 1 | 1 | 0 | 0 | 0 | 0 |
| 2 | 0 | 1 | 0 | 0 | 0 |
| 3 | 1 | 1 | 0 | 0 | 0 |
| 4 | 0 | 0 | 1 | 0 | 0 |
| 5 | 1 | 0 | 1 | 0 | 0 |
| 6 | 0 | 1 | 1 | 0 | 0 |
| 7 | 1 | 1 | 1 | 0 | 0 |
| 8 | 0 | 0 | 0 | 1 | 0 |
| 9 | 1 | 0 | 0 | 1 | 0 |
| 10 | 0 | 1 | 0 | 1 | 0 |
| 11 | 1 | 1 | 0 | 1 | 0 |
| 12 | 0 | 0 | 1 | 1 | 0 |
| 13 | 1 | 0 | 1 | 1 | 0 |
| 14 | 0 | 1 | 1 | 1 | 0 |
| 15 | 1 | 1 | 1 | 1 | 0 |
| 16 | 0 | 0 | 0 | 0 | 1 |

Note: Logical status '1' indicates that the bit is on.

Logical status '0' indicates that the bit is off.

Stream Alarm terminals

These terminals generate a voltage-free contact output signals for alarms occurring in each measurement stream.

(C) Contact specifications

Contact input

The input status depends on the resistance between contacts A and B, as follows:

If the resistance is 200 ohms or less, input is on.

If the resistance is 100 kilo-ohms or more, input is off.

If the resistance is between 200 ohms and 100 kilo-ohms, normal operation is not guaranteed.

Contact output

- Except for the Alarm Buzzer and Alarm Light terminals

Each contact can operate under either a normally open or normally closed status.

Always limit the contact voltage and current to 30 V DC and 0.5 A, respectively.

- For the Alarm Buzzer and Alarm Light terminals

A voltage of +24 V DC will be applied if an alarm occurs. No circuit protection is provided.

(D) CM500 internal power outlet

The power outlet inside the CM500 is permits a maximum of 100 V AC at 2 A. Use this outlet to connect a personal computer for controlling the CM500.

The outlet can also be used for maintenance services, but amperage must not exceed 2 A.

4. SYSTEM COMPONENTS AND THEIR FUNCTIONS

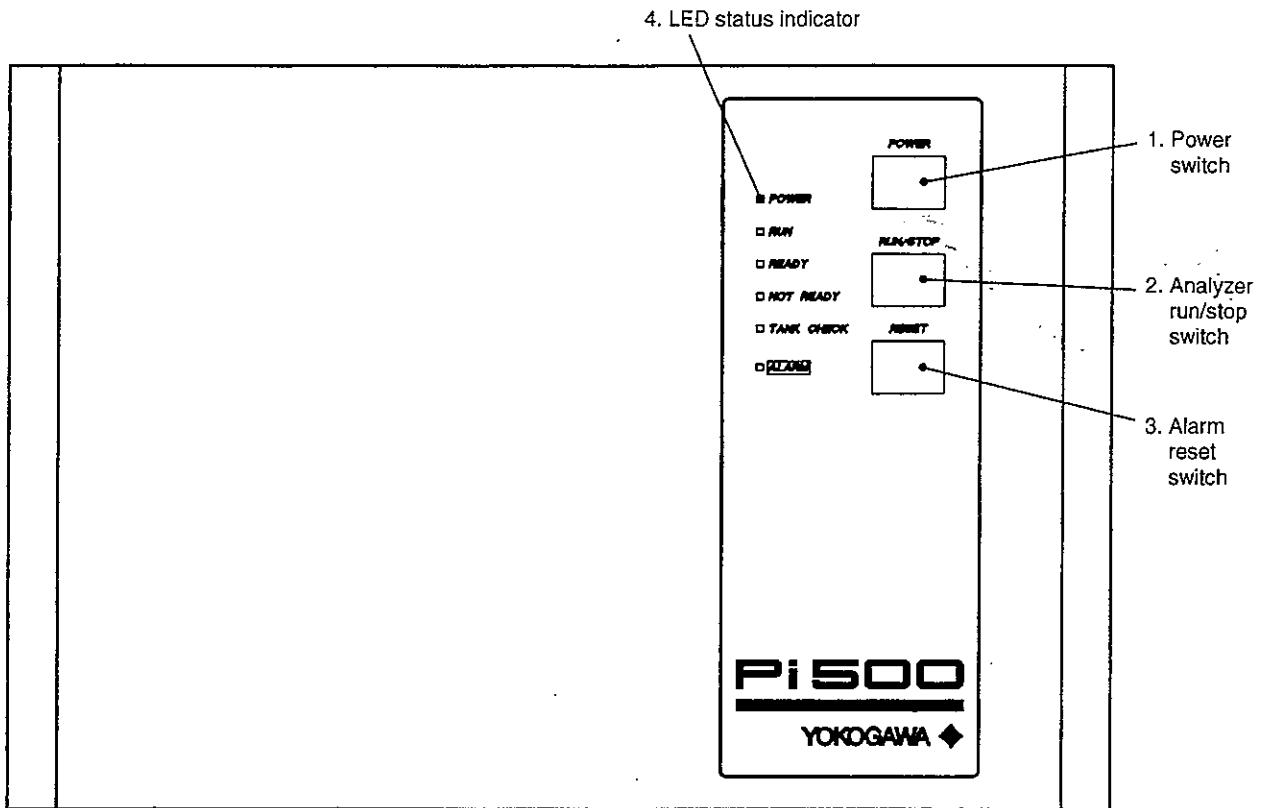


Figure 4.1 Pi500 Front View (with door closed)

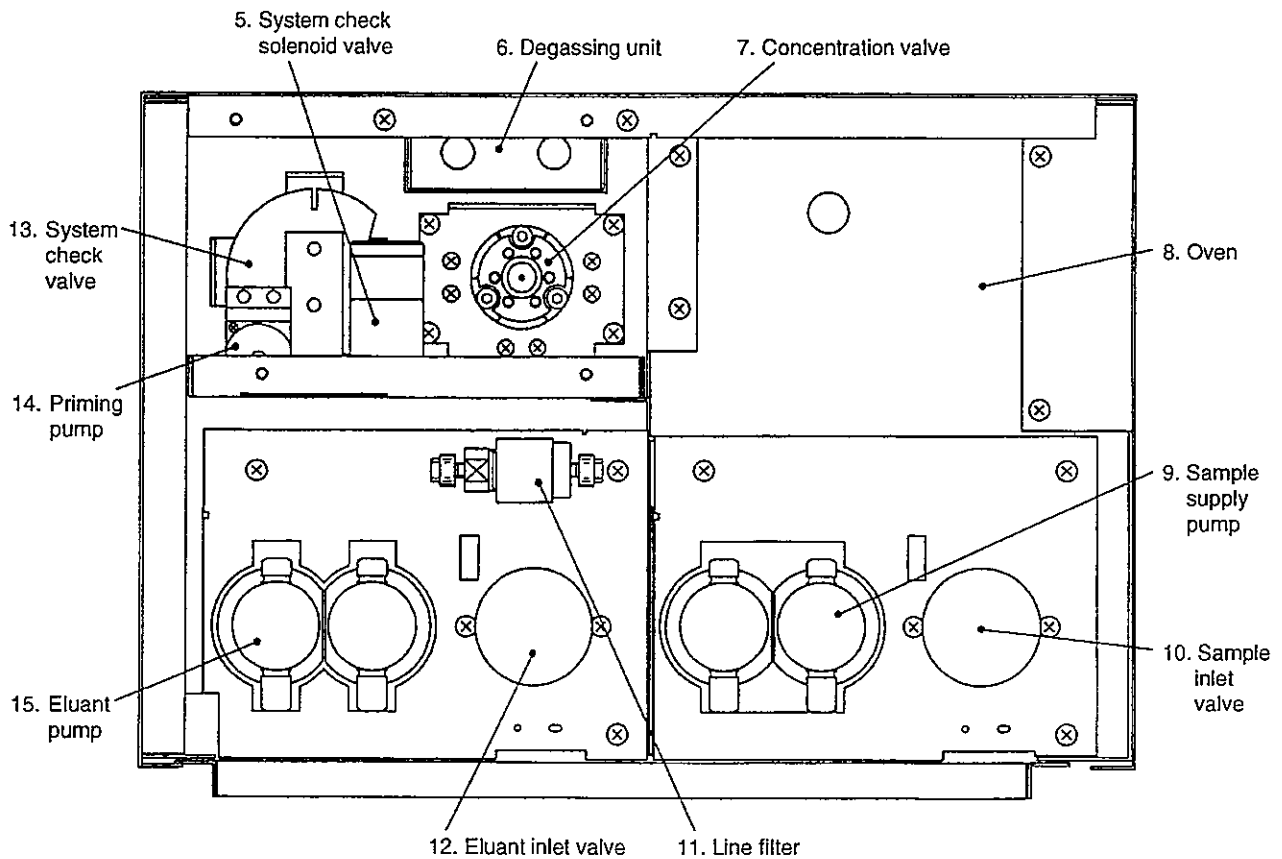


Figure 4.2 Pi500 Front View (with door open)

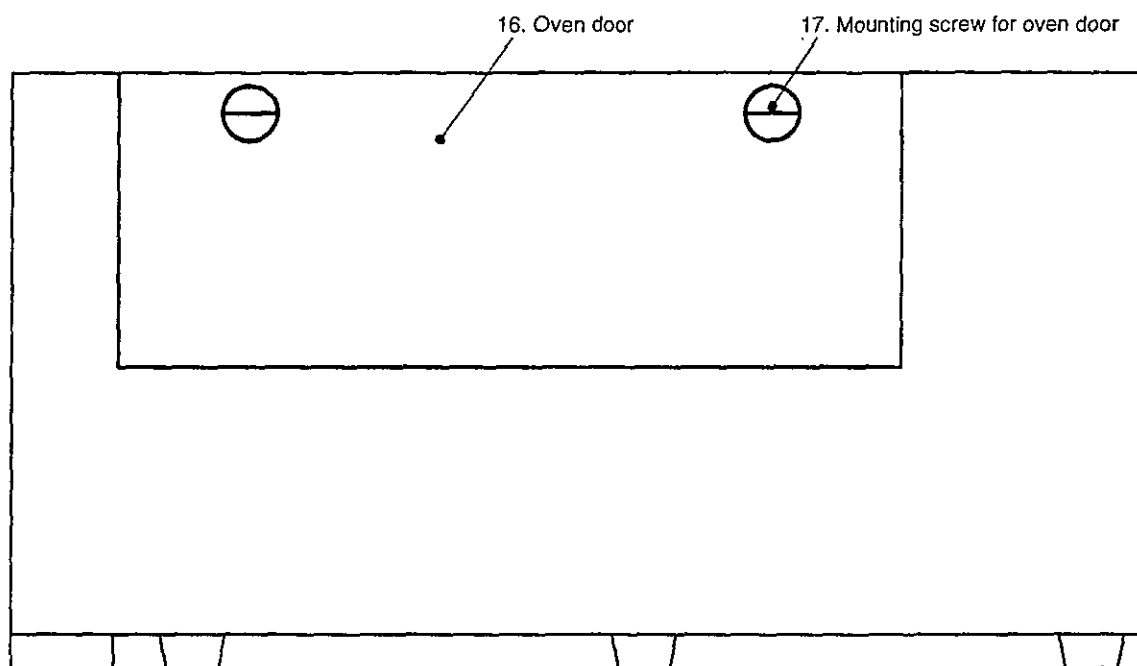


Figure 4.3 Pi500 Side View

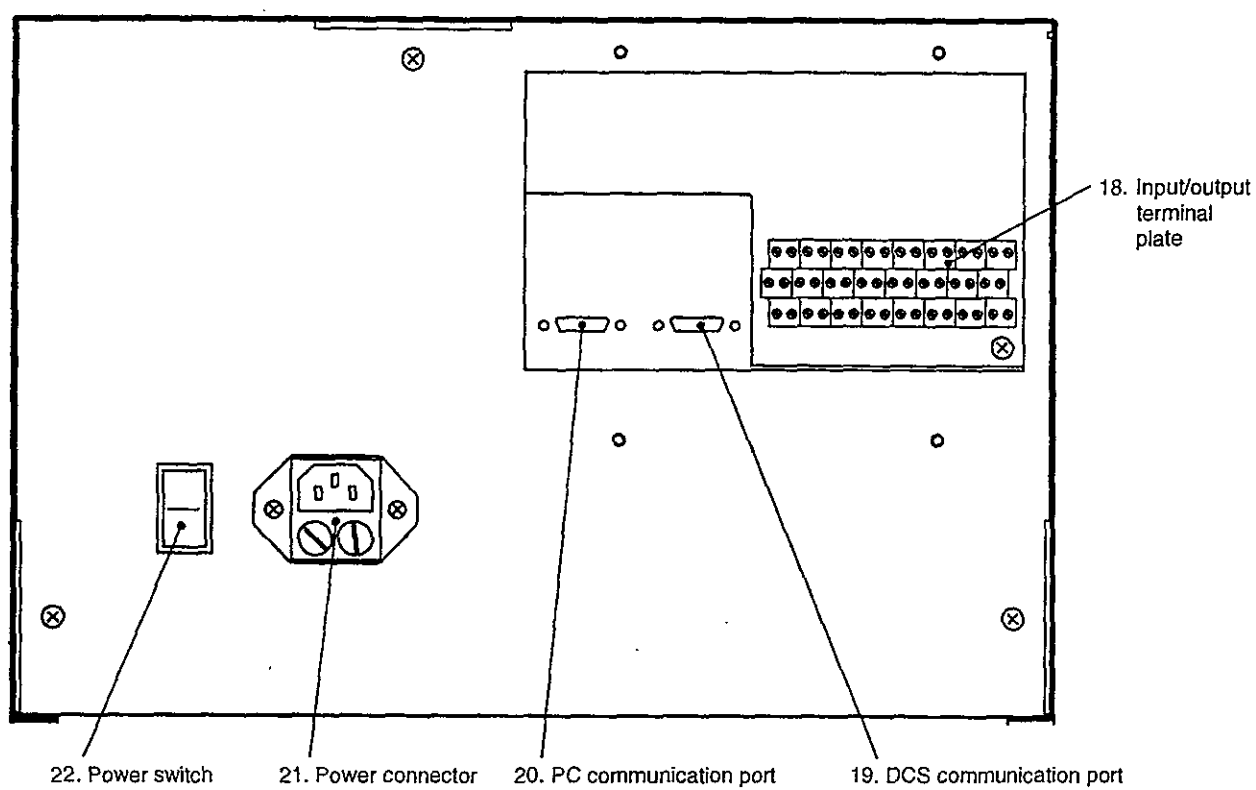


Figure 4.4 Pi500 Rear View

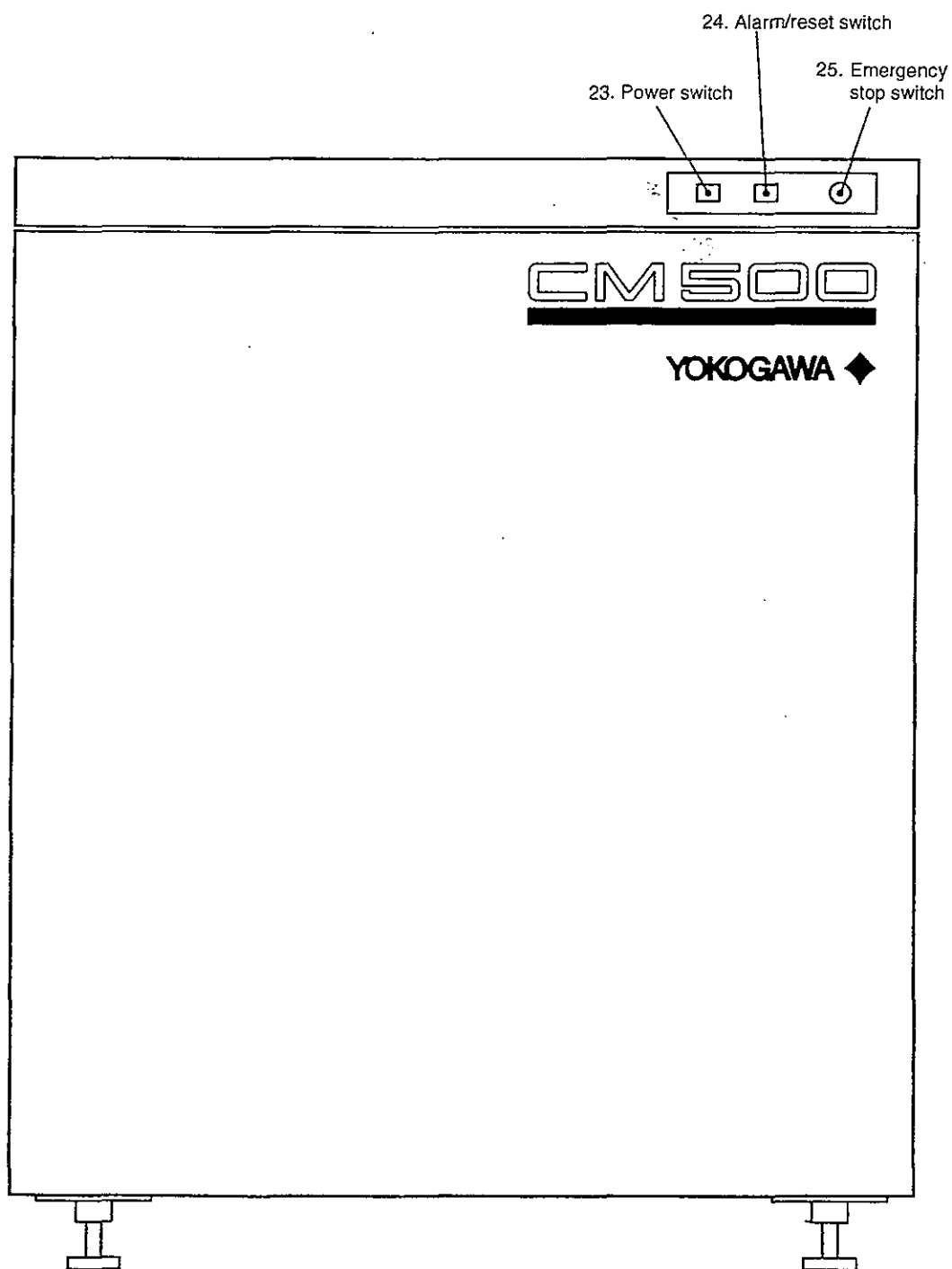


Figure 4.5 CM500 Front View (with door closed)

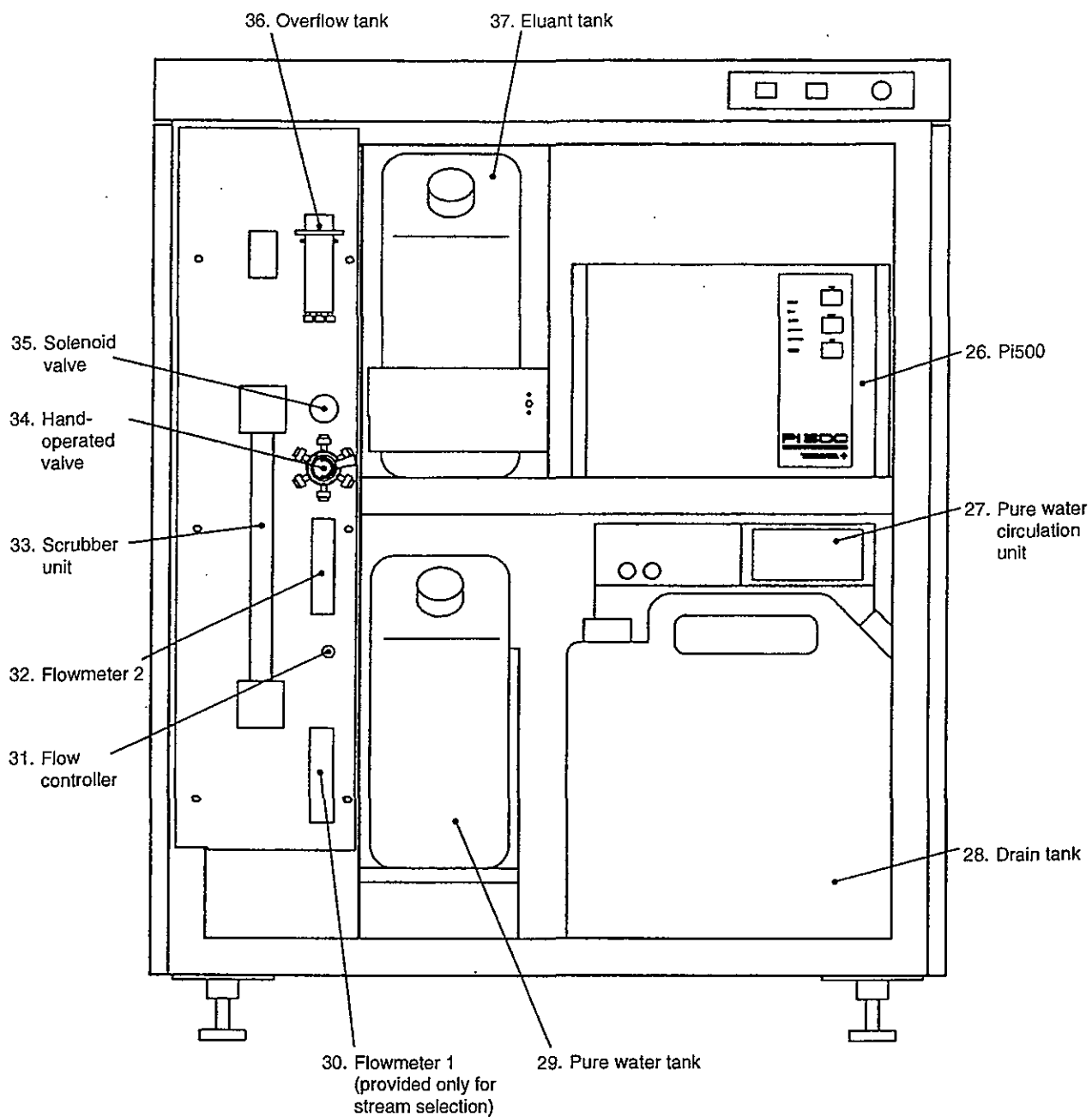


Figure 4.6 CM500 Front View (with door open)

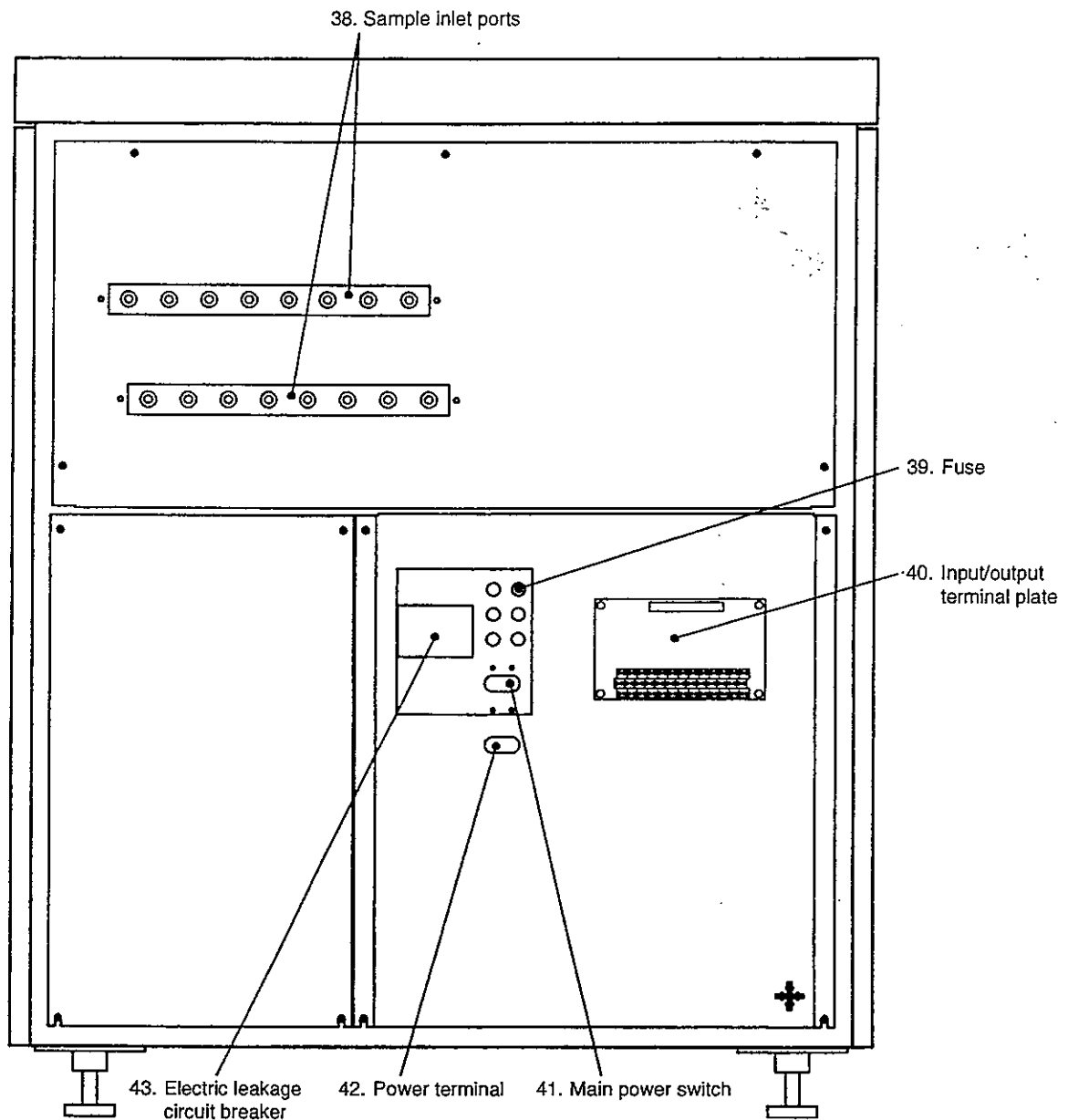


Figure 4.7 CM500 Rear View (with protecting cover open)

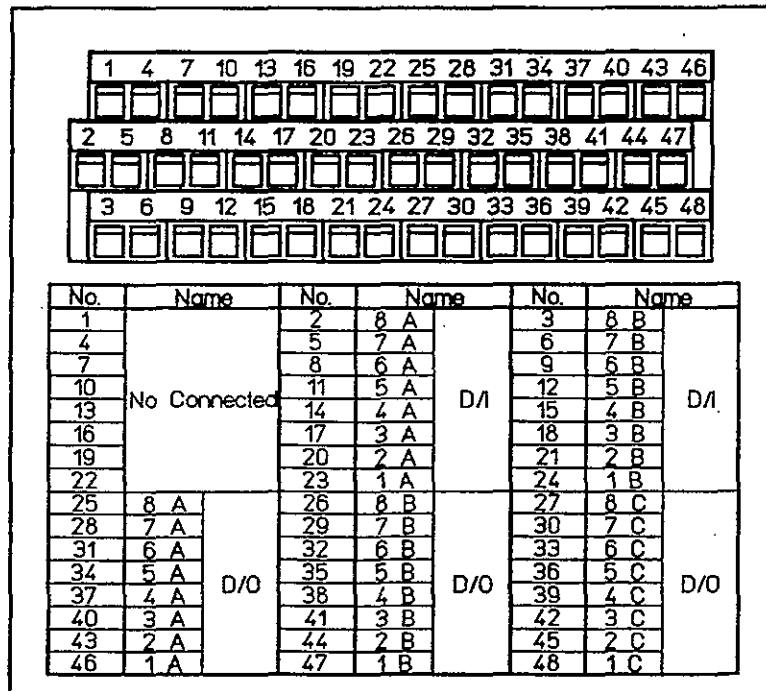


Figure 4.8 PI500 Input/Output Terminal Plate

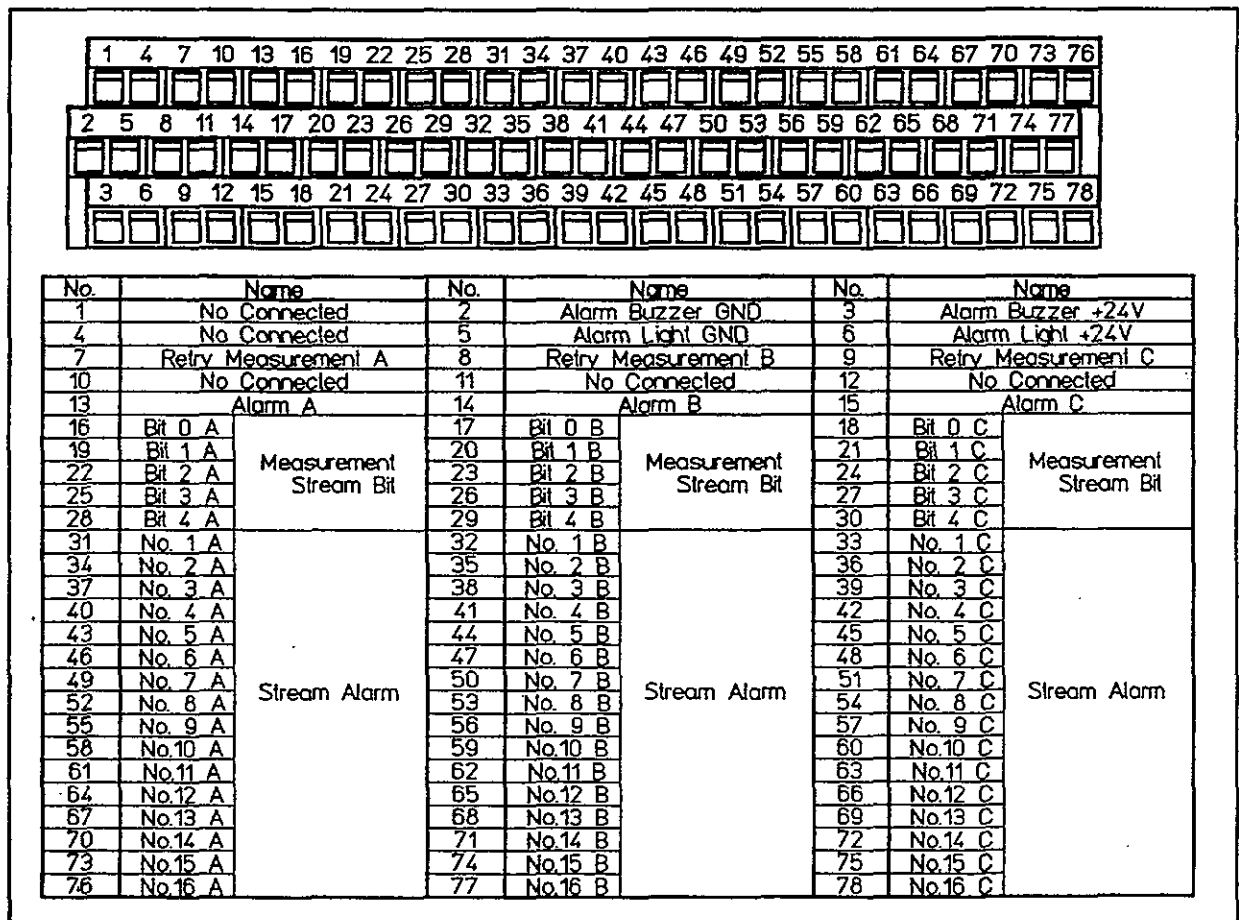


Figure 4.9 CM500 Input/Output Terminal Plate

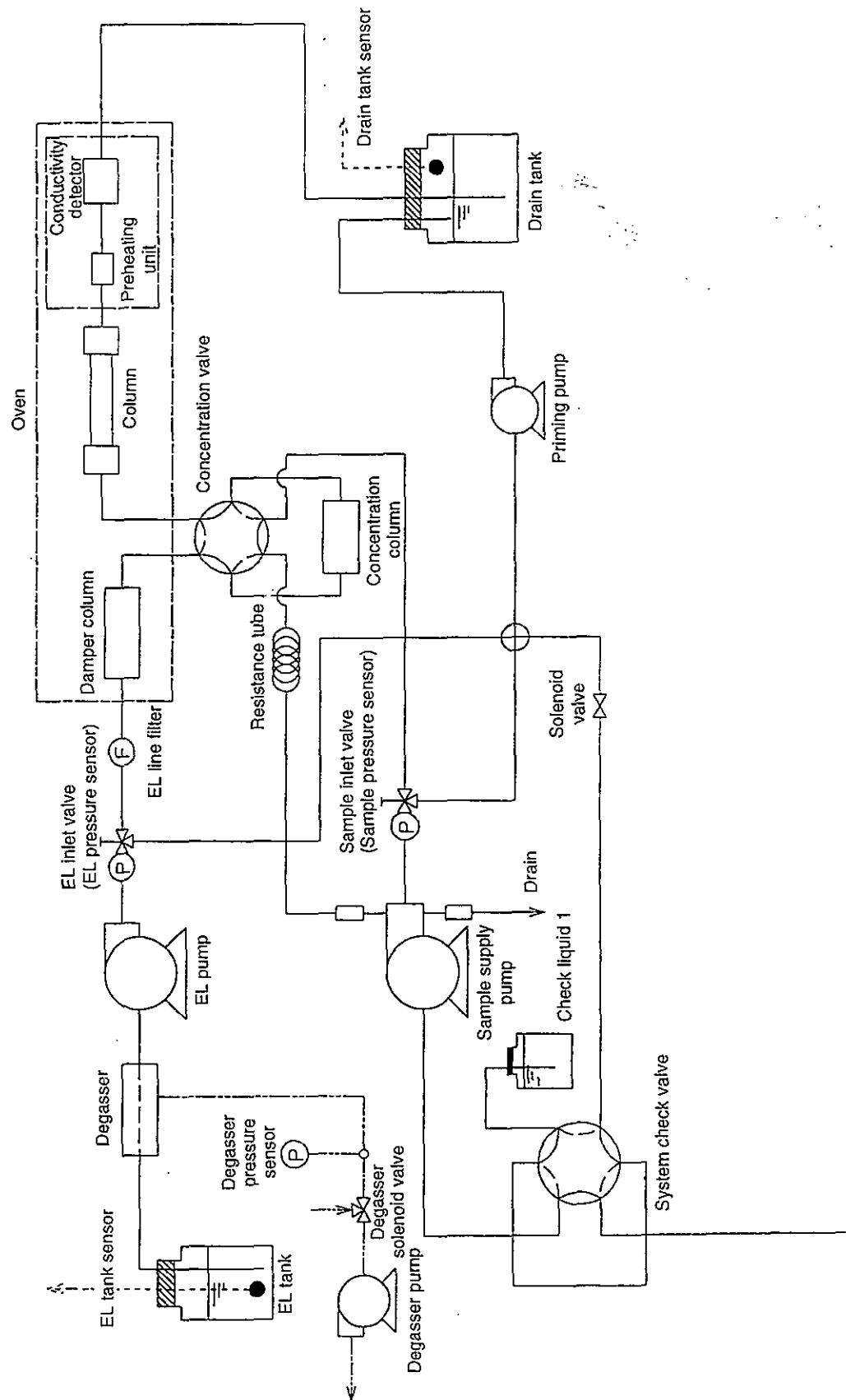


Figure 4.10 Pi500 Flow Diagram

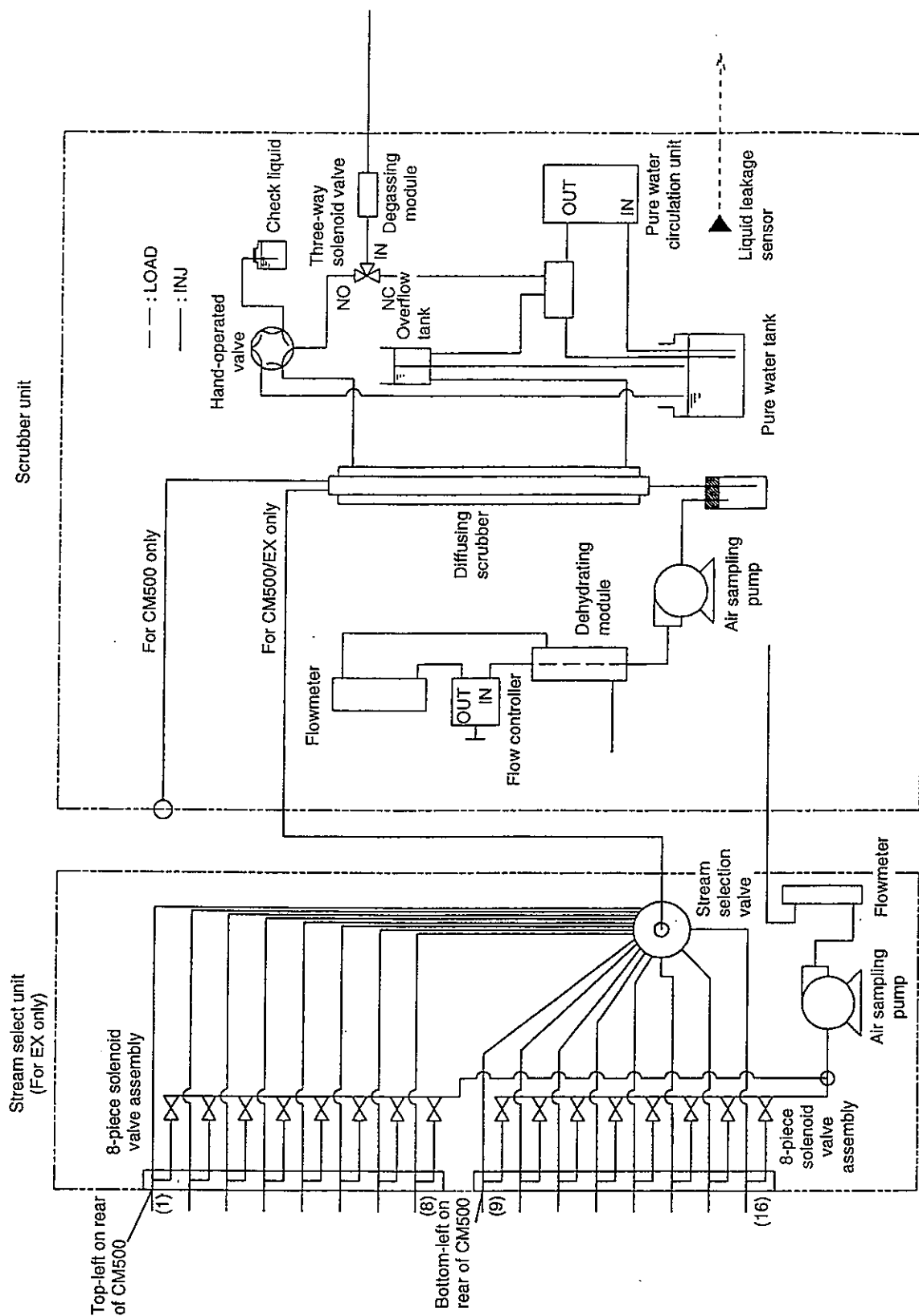


Figure 4.11 CM500 Flow Diagram

5. INSTALLATION

It is assumed that startup (unpacking, accessories checks, system installation, adjustment, and function checks) of the Pi500 and the CM500 is performed by our engineers. This section describes the precautions to be taken if, for some reason, the user is to install the system.

5.1 Installation Conditions

(A) Environment

For safe operation and to achieve highest performance of the analyzer, install the analyzer at a location that satisfies the following environmental conditions:

- Ambient temperature must be between 5 and 35°C throughout the year, and must not change significantly during measurement.
- Relative humidity must be between 15 and 95%, and no condensation must occur.
- No fires in close proximity.
- Must provide adequate ventilation.
- Must be free from direct sunlight.
- Must be free from such strong vibration or shocks that one can feel bodily.
- No potentially corrosive gases.

(B) Power supply

| | |
|-----------|---|
| Voltage | 90 to 110 V AC |
| Frequency | 47 to 63 Hz |
| Capacity | 250 VA (for the Pi500 only) or 12 kVA (for the CM500) |
| Grounding | Provide electrical grounding of Class 3 or higher (100 ohms or less) using the grounding terminal located on the rear panel of the CM500. |

(C) Installation location

Install the analyzer at a location that is level, capable of withstanding its weight and dimensions, as well as satisfying the conditions listed above.

| | | |
|------------|-------------|---|
| Weight | Pi500 | 25 kgf |
| | CM500 | 120 kgf |
| Dimensions | Pi500 | 340 mm wide × 245 mm high × 480 mm deep |
| | CM500 | 860 mm wide × 1,060 mm high × 730 mm deep |

(D) Pure water used

Highly pure water is required for eluant and standard solution conditioning. Use deionized water whose conductivity does not exceed 1 $\mu\text{S}/\text{cm}$ at 25°C and from which particles have been removed using filters of 0.5 μm or less.

5.2 Assembling the System

(1) Installation

When moving the Pi500, hold the analyzer by the side and lower edge.



CAUTION

Do not hold the analyzer by the bottom of the front door.

The analyzer weighs 23 kgf. Be careful not to drop it!

(2) Mounting the columns

The separation column and concentration column are usually shipped in mounted form. If, for some reason, they are shipped in unmounted form, connect them according to the fluid line diagrams given in Figs. 4.10 and 4.11. See Chapter 9 for connection.

(3) Connecting liquid lines

Connect the eluant line to the corresponding column.

(4) Connecting the liquid waste tank

Connect the liquid waste tank to a position lower than the bottom of the analyzer body.

5.3 Tubing Precautions

- (1) The tubing to the main column can withstand pressures of up to 15 MPa. However, since the flow rates and pressures allowed for the column suppressor are limited, the respective handling notes should be adhered to.
- (2) The system is checked prior to shipping, to ensure that all fluid lines are free from leakage under standard analyzing conditions for mounted columns and other required analysis components. Under non- standard analyzing conditions, make sure that each section is free from leakage before performing the analysis. If leakage is found, retighten the bolts and nuts on the tube connections. Vibration during transport may cause loose connections. If leakage from a connection is found during startup, check that the connector itself is normal and that the tubing is connected correctly to the extreme end, before retightening the connection screws.
- (3) Sections of the Pi500 and CM500 that are to be brought into contact with liquids are made of resin. Since temperature changes often result in significant deformation of these resin parts, liquid may leak from a connection when the oven temperature is increased. Thus, after changing the oven temperature, make sure that no liquid is leaking from any sections before performing the analysis. If leakage is found, retighten the screws on the corresponding connections.

(4) Damper

The damper minimizes changes in pump pressure while at the same preheating the liquid. Strictly observe the following notes during handling:

- 1 Provide tubing, only after checking that the damper is filled with the required liquid. If liquid leakage or other events allow air to enter the damper, disconnect the connector from the downstream side of the damper, tilt this section of the damper upward, and remove the air before tubing the line.
- 2 The capacity of the damper is about 4.2 ml. Drain out the liquid completely before replacing it with another liquid. Liquid replacement can be performed faster by draining out the liquid after disconnecting the connectors located in front and at rear of the damper. This also involves removing the air completely from the damper before tubing the line.

5.4 Connecting the Columns

(1) Column connecting methods

See Section 9.6, "Replacing the Columns," for column connecting methods.

(2) General notes on column replacement

The columns are initially charged with the same type of eluant as that which was used during inspection by the manufacturer. Before connecting the columns, replace the eluant using the procedure explained in item (3) below.

(3) General notes on eluant replacement (Changing the chemical composition of the eluant)

First, disconnect the column to be replaced and close both ends of the column with a cap. Pure water and two beakers are also required. When the composition of the eluant is not to be changed, perform the priming operations only and supply the liquid taking care not to allow air bubbles to enter the column.

Eluant replacement procedure

- 1 Remove the tube set from the eluant tank and lightly wash the solvent filter (located at the end of the tube set) in a beaker of pure water.
- 2 Replace the pure water contained in the beaker, then remount the end of the tube set, and prime the pump. See Section 6.3, "Priming Operations," for the priming procedure.
- 3 Close the inlet valve and feed pure water for about 30 minutes at a flow rate of 0.2 ml/min. Then, completely flush the fluid line leading to the front of the column, with the pure water. Collect the liquid waste using the other beaker.
- 4 Fill the beaker with a suitable amount of eluant and repeat steps 1 to 3 above.
- 5 Connect the column and supply the eluant at a flow rate of 0.2 ml/min. This completes the preparation for analysis.

5.5 General Notes on Sample Injection

The notes to be observed when injecting samples vary slightly from column to column. Refer to the operating manual of the column to be used. General notes are listed below.

- 1 Before using a sample, filter it using a filter of 0.45 μm or less.
- 2 Extract the sample if it does not mix with the eluant.
- 3 If the pH value of the sample differs significantly from that of the eluant, perform pH adjustments using neutralization or dilution type methods.

6. BASIC OPERATIONS

6.1 Before Turning the Power On

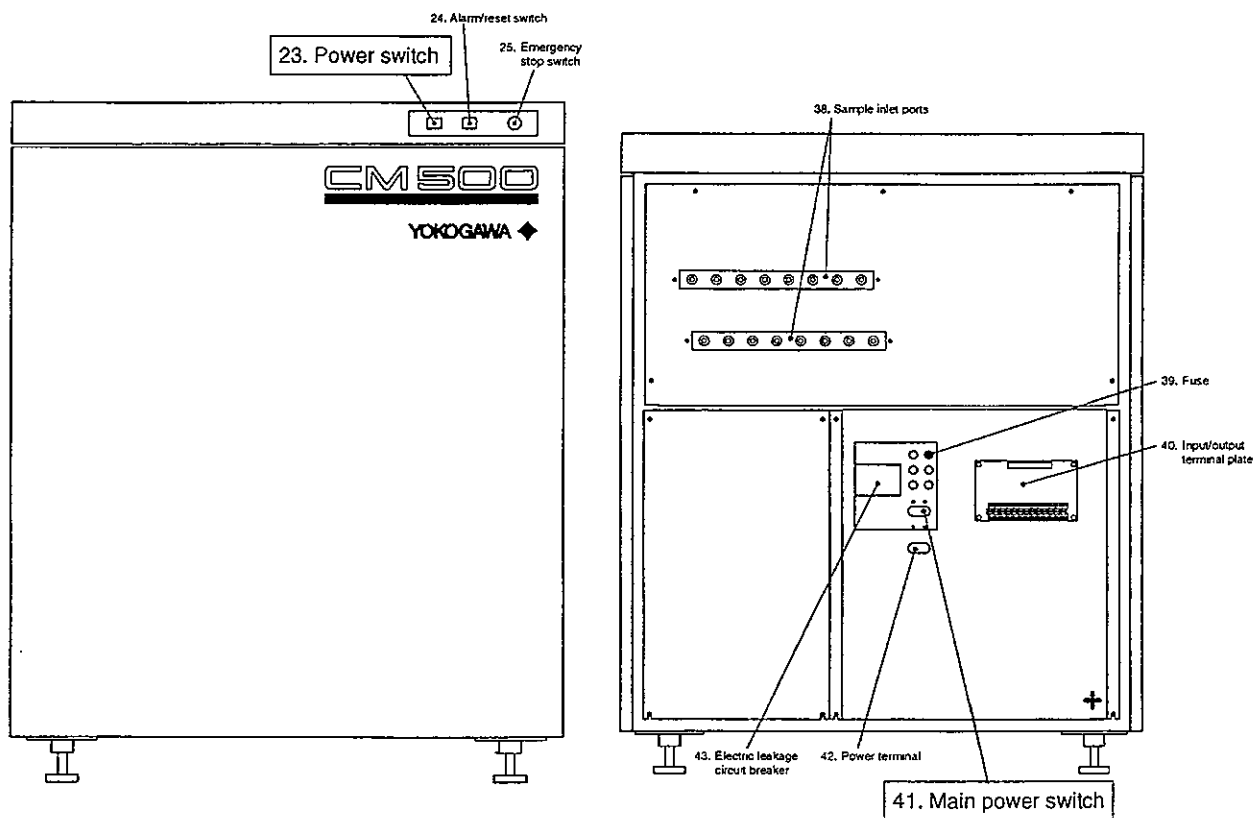
Before turning on the power switch, perform the following operations:

- (1) Check that the required eluant is charged and that the liquid line is tubed correctly.
- (2) Check that pure water is charged and that the water line is tubed correctly.
- (3) Empty the liquid waste tank.

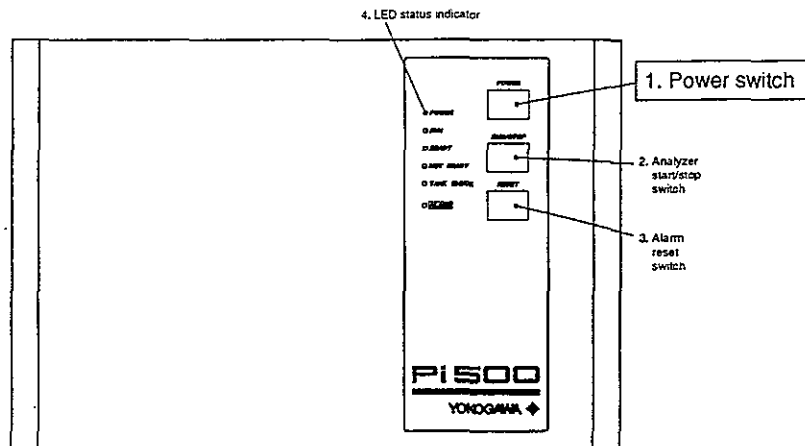
6.2 Turning Power On

Proceed as follows to turn power on:

- (1) Connect the power cable to an outlet having a grounding terminal.
- (2) Turn on the main power switch located on the rear panel of the CM500.
- (3) Turn on the power switch located on the front panel of the CM500. When power is turned on, self-checking will begin and the CM500 will be initialized.



(4) Turn on the Pi500 power switch or leave on if it is already turned on.



(5) Starting up the Pi500 Workstation

Turn the personal computer power on and then after starting Windows 95, click the mouse button on the "Start" menu, "Programs," "Pi500 WorkStation," and "PIWS," in this order. For further details, refer to Section 2.1, Running and Quitting Workstation, of the Pi500 Workstation Operation Guide.

6.3 Priming Operations

The eluant pumps and the sample supply pumps require priming. Both these pumps use a plunger to pump the respective fluids and are not effective if air bubbles are contained. It is therefore, necessary to perform priming operations such as filling the pumps with fluids and removing air bubbles from the pumps, beforehand. Prime the pumps as follows:

- (1) Rotate the eluant (EL) inlet valve approx. 90 degrees counterclockwise. When the pump switch turns on, ELINT of the Workstation will change from OFF (blue) to ON (yellow). After leaving this state intact for about 2 to 3 minutes, close the EL inlet valve.
- (2) Rotate the sample (SM) inlet valve approx. 90 degrees counterclockwise. When the pump switch turns on, SMINT of the Workstation will change from OFF (blue) to ON (yellow). After leaving this state intact for about 2 to 3 minutes, close the SM inlet valve.

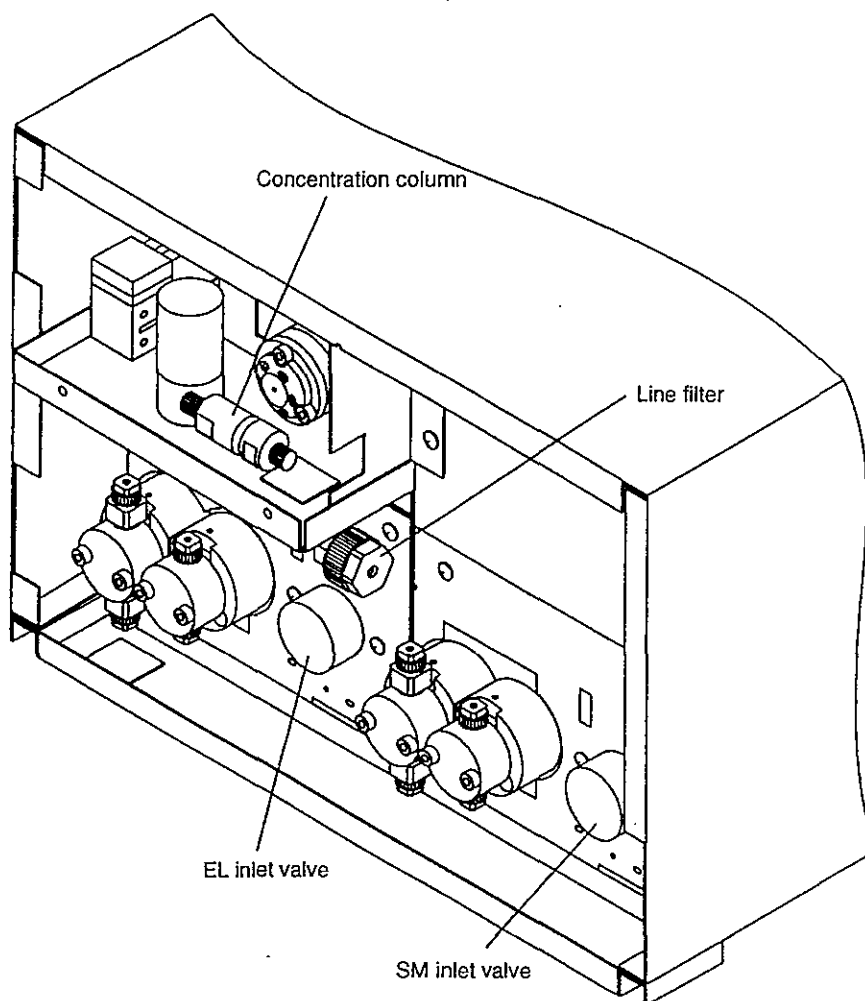
**NOTE**

Slowly rotate the inlet valves approx. 90 degrees. The pumps will switch off if the valves are rotated too far.

The pumps are switched on when the inlet valves are activated, and switched off when priming is complete. To restart the pumps, change the mode to manual operation and switch the pumps back on.

Air bubbles inside the pumps cause the supply of fluids to be irregular. Priming is therefore required to remove the air bubbles. However, if the inlet valves are opened abruptly, the resulting pressure shock will adversely affect the separation column.

Before opening the inlet valves, stop the pumps and make sure that the internal pressures are below 0.5 MPa.



6.4 Standby Mode

Change the Workstation mode from “Not Ready” to “Standby,” and then press the standby mode button. The EL and SM pumps will start operating. When the system is ready for measurement, standby mode processing will begin and the color of the corresponding icon will change to black. The standby mode’s waiting status (when the icon is not black), means that the Workstation is still waiting to enter the standby mode.



Standby mode Processing



Waiting for Standby mode

Conditions for entering the standby mode are listed below. All of these conditions must be satisfied before the Workstation can enter the standby mode, that is, before it can advance to the next mode, Run. If the data display area is red (in the table below), the corresponding item does not satisfy the required condition.

| Conditions for the Workstation to enter the standby mode | Data display area |
|--|-------------------------------|
| The oven temperature must be between 39 and 41 °C. | Oven temperature display |
| The EL pump pressure must be within its upper and lower limits, and the pump itself must be on. | EL pump pressure display |
| The SM pump pressure must be within its upper and lower limits, and the pump itself must be on. | SM pump pressure display |
| The absolute conductivity of the EL must be within the required limits. | Absolute conductivity display |
| The difference between the maximum and minimum heights of the base line must not exceed 100 nS/cm in any 20 second period. | Conductivity display |

Note: The values in the above conditions are the original settings. These settings can be modified by operating the control panel.

6.5 Selecting an Operation Pattern

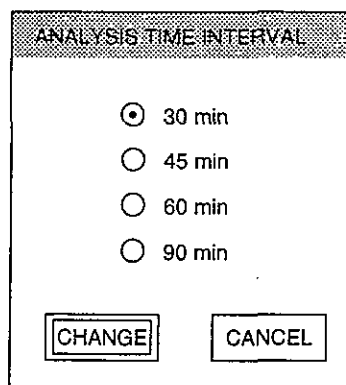
Four operation patterns are available. See Section 7.1, Operation Patterns, for the list of original settings.

This section explains initial operation pattern selection.

Select operation pattern 1. Only level B or C users can change the selected pattern. No other users can change the pattern unless their user levels are changed. For further details, refer to Section 2.4, “Changing the Operation Pattern,” of the Pi500 Workstation Operation Guide.

6.6 Selecting an Operating Time Interval

Operating time intervals can be selected while the Workstation is in the standby mode. Pressing corresponding operation buttons in the Analyzer Operation window, displays operating time intervals of 30 min, 45 min, 60 min, and 90 min. Select an operating time interval from these initial settings.



ANALYSIS TIME INTERVAL

☒ 30 min
☐ 45 min
☐ 60 min
☐ 90 min

CHANGE CANCEL



NOTE

The selected operating time interval is common to all tasks. For example, if the operating time interval is 90 min, one analytical operation takes 90 min, even after calibration.

When the operating time interval is extended, the operating time of the scrubber will increase correspondingly. To analyze a low concentration sample after sampling a highly concentrated one, use the longest possible analysis time interval.

Set the interval to at least 45 min to perform measurements under normal conditions (excluding calibration, system checks, and pure water supply).

6.7 Measurement

Measurement can be started at any time after the Workstation has entered the standby mode and the Analyzer Operation window has changed to blue. When the RUN button is pressed in this state, automatic operation is executed.

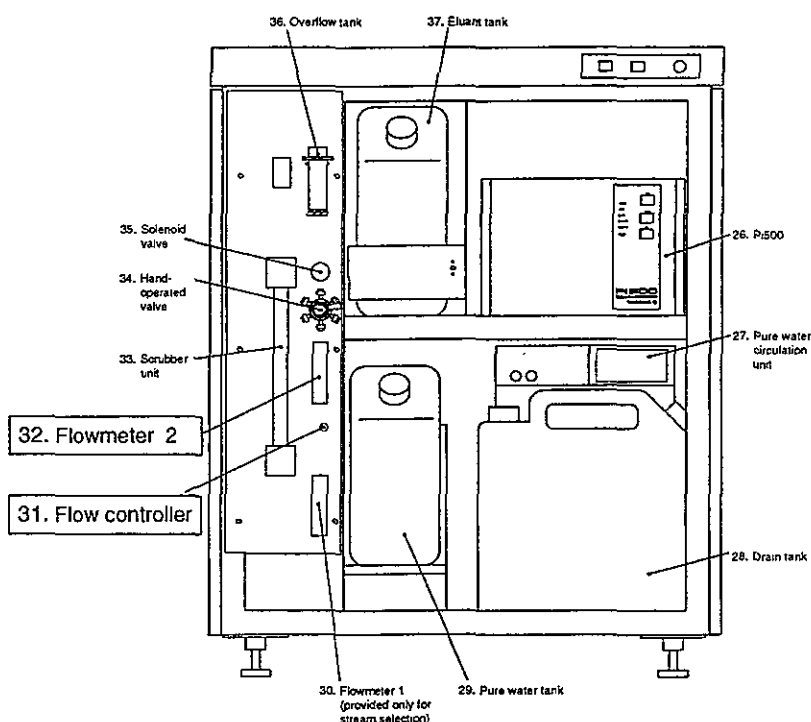
6.8 Checking and Adjusting the Flow Rate of the Gas

When automatic operation starts, the selection valve will be activated and the gas suction pump will also start operating. Check flowmeter 2 on the front panel of the CM500 to ensure that the gas flow rate is 1 l/min. A flow controller for adjusting the flow rate is located under the flowmeter. Rotate the knob of the flow controller and set it to "1L."



CAUTION

Significant changes in the flow rate indicate that there is a problem with the gas suction port. After checking for loose tube connectors, please contact your Yokogawa service representative.



6.9 Terminating Measurement

To terminate measurement, press the STANDBY button. The current measurement cycle will then end and the Workstation will return to the standby mode.

6.10 Stopping Analysis Urgently

To stop analysis urgently, press the STOP command button. The current measurement cycle will then end and the Workstation will return to the standby mode.



NOTE

It takes about 15 minutes to completely drain the sample once it has entered the concentration column (that is, once the display of CONv has changed to yellow). Therefore, wait for at least 15 minutes after a sample has been supplied before starting to analyze it.

6.11 Terminating Operation

Terminate operations as follows:

- (1) Press the "Not Ready" button in the standby mode. The EL and SM pumps, the oven, and so on, will all turn off.
- (2) Terminate the Workstation.
- (3) Turn off the power switch located on the front panel of the CM500.

6.12 Measuring Data of the Same Stream Line

Data of only one fluid line can be measured continuously. The procedure is as follows:

- (1) Select the standby mode.
- (2) Select operation pattern T.

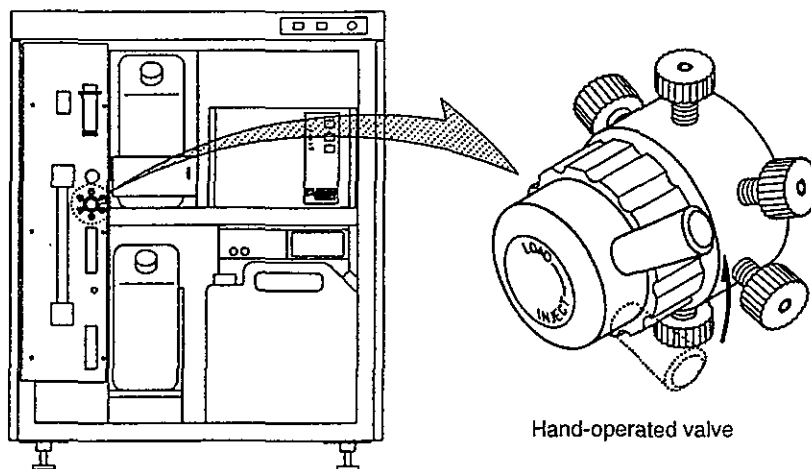


- (3) Select a task number. For example, if continuous analysis is to be performed for stream 3 only, select task number 3.
- (4) Select an operating time interval of 45 min or more.
- (5) Press the RUN button to start the measuring operations.

6.13 Creating Calibration Curves

Proceed as follows to create calibration curves:

- (1) Select the standby mode.
- (2) Prepare about 1 liter of a standard solution of 10-ug/l ammonium in a beaker and place on the CM500.
- (3) Insert the standard solution line (above the hand-operated valve of the CM500) into the standard solution of ammonium.
- (4) Turn the hand-operated valve to the upper (LOAD) position.



- (5) Prime the SM pump for 1 to 2 minutes.
- (6) Reselect the standby mode by pressing the **STANDBY** button once again.
- (7) Change the pattern number to 2, and set this pattern as an interruption pattern.
- (8) Set the analysis interval time to 30 minutes.
- (9) When the standby mode is set, press the **RUN** button to initiate operation.

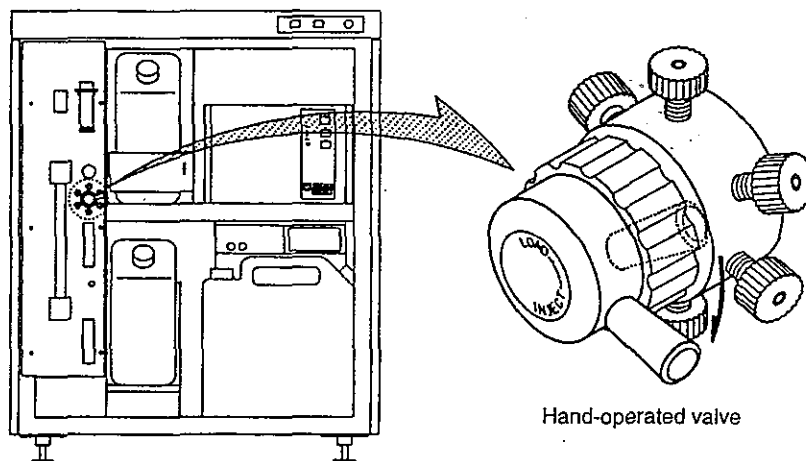


CAUTION

In the calibration curve creating pattern, the current standard solution is analyzed using the previous calibration curve, the new calibration curve is created, and finally, the data check results are analyzed using the new calibration curve. After this series of analysis, operation automatically enters the standby mode and if pattern 2 is set as the main pattern, not an interruption pattern, analysis of the data check results will be repeated.

If the prepared 10 ug/l of standard solution is left as it is, external ammonium will increase the concentration of the internal ammonia. Thus, use the standard as soon as possible after preparation.

- (10) Press the stop button.
- (11) When operation returns to the standby mode, return the hand-operated valve to its original (Inject) position to prepare for the next analytical cycle.



6.14 System Check Function

If the prepared 10 ug/l of standard solution is left as it is, external ammonium will increase the concentration of the internal ammonia. Preparation of a standard must therefore be repeated each time. The system check scheme is provided to avoid repeating preparation of standards that consume time and labor. This scheme uses a highly concentrated standard of the order of ug/l for checking whether the system is operating normally.

The checking procedure is as follows:

- (1) Select the standby mode.
- (2) Set the pattern number to 3, and set this pattern as an interruption pattern.
- (3) Set the analysis interval time to 30 minutes.
- (4) Press the RUN button to execute the system check function.



NOTE

About 3 ml of check liquid is consumed during one system check operation. Since the check liquid is minimal, take note of the residual quantity.

6.15 Pure Water Check Function

Use this function to analyze the pure water used in the scrubber. The procedure is as follows:

- (1) Select the standby mode.
- (2) Set the pattern number to 4, and set this pattern as the main pattern.
- (3) Set the analysis interval time to 30 minutes.
- (4) Press the RUN button to start the pure water analysis.
- (5) Repeat measurement a number of times, and when the data stabilizes, terminate the measuring cycle.



NOTE

When checking the pure water, make judgments based on the results of a series of analysis, not the results of one analysis.

6.16 Routine Check

The system consumes pure water at a rate of 2 ml/min, that is, almost 3 liters a day. If the system is to be continuously operated except for on Saturdays and Sundays, fill up the pure water tank at the end of Fridays. It takes about 21 hours for an alarm to occur after the message warning of a shortage of pure water.

The system consumes eluant (EL) at a rate of 0.2 ml/min, that is, almost 300 milliliters a day. The EL tank has a capacity of 10 liters to enable about one month of operation. Operation can be continued for at least 3 days after the message warning of a shortage of EL. However, replace the EL as soon as possible after this warning message is displayed.

The pure water and the EL accumulate in the liquid waste tank at a rate of 2.2 ml/min, that is, about 3.2 liters a day. If the system is to be continuously operated except for on Saturdays and Sundays, empty the liquid waste tank at the end of Fridays. It takes about 19 hours for an alarm to occur after the message warning of a full tank.

7. OPERATION PATTERNS, TASKS, AND SEQUENCES

Before running the Workstation, a pattern number must be set. An operation pattern consists of the integrated program data for one analytical cycle. Each part of the analytical cycle is referred to as a task. Thus, operation patterns specify the order of task execution. Each task includes a sequence number for specifying the valve selection time or the auto-zeroing time.

7.1 Operation Patterns

The initial settings of operation patterns are shown below.

(1) If an automatic stream selection unit is not provided

| Operation pattern number | Description | Task numbers |
|--------------------------|--|--------------|
| 1 | Analyzing a sample gas | 1 |
| 2 | Analyzing a standard solution and creating a calibration curve | 3, 2, 3 |
| 3 | Analyzing the check liquid and checking the system | 4 |
| 4 | Analyzing the water used in the scrubber | 5 |

(2) If an automatic stream selection unit is provided

| Operation pattern number | Description | Task numbers |
|--------------------------|--|--------------------------------|
| 1 | Performing sequential analyses on stream numbers 1 to 16 | 1, 2, 3, 4, 5, — 14, 15, 16 |
| 2 | Analyzing a standard solution and creating a calibration curve | 18, 17, 18 |
| 3 | Analyzing the check liquid and checking the system | 19 |
| 4 | Analyzing the water used for the scrubber | 20 |

7.2 Tasks

The initial settings of tasks are shown below.

(1) If an automatic stream selection unit is not provided

| Task number | Description | Sequence number |
|-------------|--|-----------------|
| 1 | Analyzing a sample gas | 1 |
| 2 | Analyzing a standard solution and creating a calibration curve | 2 |
| 3 | Analyzing a standard solution and checking data | 2 |
| 4 | Analyzing the check liquid and checking the system | 3 |
| 5 | Analyzing the pure water | 4 |

(2) If an automatic stream selection unit is provided

| Task number | Description | Sequence number |
|-------------|--|-----------------|
| 1 | Performing analyses on stream number 1 | 1 |
| 2 | Performing analyses on stream number 2 | 1 |
| 3 | Performing analyses on stream number 3 | 1 |
| 4 | Performing analyses on stream number 4 | 1 |
| 5 | Performing analyses on stream number 5 | 1 |
| 6 | Performing analyses on stream number 6 | 1 |
| 7 | Performing analyses on stream number 7 | 1 |
| 8 | Performing analyses on stream number 8 | 1 |
| 9 | Performing analyses on stream number 9 | 1 |
| 10 | Performing analyses on stream number 10 | 1 |
| 11 | Performing analyses on stream number 11 | 1 |
| 12 | Performing analyses on stream number 12 | 1 |
| 13 | Performing analyses on stream number 13 | 1 |
| 14 | Performing analyses on stream number 14 | 1 |
| 15 | Performing analyses on stream number 15 | 1 |
| 16 | Performing analyses on stream number 16 | 1 |
| 17 | Analyzing a standard solution and creating a calibration curve | 2 |
| 18 | Analyzing a standard solution and checking data | 2 |
| 19 | Analyzing the pure water | 3 |
| 20 | Analyzing the pure water | 4 |

7.3 Sequences

The initial settings of sequences are shown below.

The sequence data is independent of whether an automatic stream selection unit is provided.

| Sequence number | 1 | 2 | 3 | 4 |
|-------------------------|-------|------|-----|------|
| Analysis time | 900 | 900 | 900 | 900 |
| Warm-up time | 1200 | 900 | 100 | 900 |
| Allowed Stopping time | 880 | 880 | 880 | 880 |
| Allowed Calc time | 870 | 870 | 870 | 870 |
| Auto-zeroing | 6 | 6 | 6 | 6 |
| Concentration valve on | -420 | -420 | -20 | -420 |
| Concentration valve off | 0 | 0 | 0 | 0 |
| Check valve on | *** | *** | -50 | *** |
| Check valve off | *** | *** | -10 | *** |
| Standard valve on | -440 | -895 | 100 | 890 |
| Standard valve off | -1160 | *** | -95 | -899 |
| Suction valve on | -1160 | *** | *** | *** |
| Suction valve off | *** | -895 | -95 | -899 |



NOTE

If the system has an automatic stream selection unit, turn on the suction pump at least 30 minutes after warm-up has begun. During this time, the route of the sample gas is automatically switched and the pump cannot be activated.

8. ALARM DISPLAY

This section explains the display of alarms.

8.1 Level 1 Alarms

If a level 1 alarm occurs, operation will change from the "Run" mode to the "Not Ready" mode and the pumps and other components will stop operating. After resetting the alarm, restart the pure water circulation unit in the manual operation mode and then press the standby button again. If the liquid leaks from the oven or the oven itself becomes abnormal, stop the oven at once and then restart it in the manual operation mode.



NOTE

If a level 1 alarm occurs, the supply of liquid from the pure water circulation unit to the scrubber will stop. If, under this state, the standby button is pressed again, the pure water circulation unit and the SM pump will restart simultaneously. Since the SM pump restarts before the liquid from the circulation unit arrives at the pump, air bubbles will enter. To avoid this, it is necessary to change the mode to manual operation turn the ultra-pure water circulation unit back on, and wait for about 30 seconds before priming the SM pump and pressing the standby button to restart.

| Level-I alarm | Status |
|---|--|
| System alarm | CPU error |
| DRAM error | CPU error |
| Exceptional interrupt error | CPU error |
| Path error | CPU error |
| Device initialize error | CPU error |
| Buffer initialize error | CPU error |
| Buffer full error | CPU error |
| EL pump pressure upper-limit error | The preset upper-limit has been exceeded. |
| EL pump pressure lower-limit error | The preset lower-limit has been overstepped. |
| EL pump original position error | Although 1,780 pulses have occurred, the pump has not yet returned to its home position. |
| EL pump rotation error | Although 160 pulses have occurred after returning to the home position, the pump still remains at its home position. |
| SM pump pressure upper-limit error | The preset upper-limit has been exceeded. |
| SM pump pressure lower-limit error | The preset lower-limit has been overstepped. |
| SM pump home position error | Although 1,780 pulses have occurred, the pump has not yet returned to its home position. |
| SM pump rotation error | Although 160 pulses have occurred after returning to the home position, the pump still remains at its home position. |
| Suppressor error | An error signal has been received from the suppressor module. |
| Liquid leakage other than the oven | Liquid leakage other than the oven has been detected. |
| Liquid leakage inside the oven | Liquid leakage inside the oven has been detected. |
| Liquid leakage outside the oven | Liquid leakage outside the oven has been detected. |
| Scrubber liquid leakage | Scrubber liquid leakage has been detected. |
| Full liquid waste tank | The liquid waste tank has become full. |
| EL level alarm | The EL level has become abnormal. |
| Internal oven temperature is too high | The temperature of the inside oven has increased above 55°C. |
| Internal oven temperature is too low | The temperature of the inside oven has decreased below 5°C. |
| External oven temperature is too high | The temperature of the outside oven has increased above 55°C. |
| External oven temperature is too low | The temperature of the outside oven has decreased below 5°C. |
| Conductivity meter CD temperature is too high | The temperature of conductivity meter CD has increased above 55°C. |
| Conductivity meter CD temperature is too low | The temperature of conductivity meter CD has decreased below 5°C. |
| Temperature zero point alarm | The zero point of temperature has become abnormal. |
| Temperature span point alarm | The span point of temperature has become abnormal. |
| Concentration valve error | The home position of the concentration valve has not been reached. |
| Concentration valve sensor error | The concentration valve sensor has become abnormal. |
| System check valve error | The home position of the system check valve has not been reached. |
| System check valve sensor error | The system check valve sensor has become abnormal. |
| Degasser pressure alarm | The preset degasser pressure has not been reached. |
| Stream selection valve home error | The home position of the stream selection valve has not been reached. |
| Stream selection valve sensor error | The stream selection valve sensor has become abnormal. |
| Water lower-limit error | The pure water level has decreased. |



NOTE

Pump upper or lower limit errors remain on the display until the pump has been powered back on, after which they will be cleared.

8.2 Level 2 Alarms

If level 2 alarms occur, warnings will be issued. These warnings will be maintained until the particular status is reset.

| Level-2 alarm | Status |
|-------------------------------------|--|
| Full liquid waste tank alarm | The liquid waste tank has become full. If this alarm remains uncleared for 19 hours (approx.), it will change to a level 1 alarm and the pumps and other components will stop. |
| EL level alarm | The EL level has decreased. If this alarm remains uncleared for 125 hours (approx.), it will change to a level 1 alarm and the pumps and other components will stop. |
| Water level alarm | The pure water level has decreased. If this alarm remains uncleared for 21 hours (approx.), it will change to a level 1 alarm and the pumps and other components will stop. |
| Communication port initialize error | System communication has failed. |
| Concentration alarm | The ammonium concentration has overstepped its upper or lower limit. |

Concentration alarms continue until the next measurement shows that the fluid line has returned to normal. To reset these alarms, press the concentration alarm reset button on the front panel of the CM500.

8.3 Level 3 Alarms

If level 3 alarms occur, only messages will be displayed. These alarms relate mainly to peak recognition software.

| Level-3 alarm | Status |
|------------------------------------|--|
| System error message | Software-related system error |
| Mode change error | Not possible to change the operation mode. |
| Buffer busy | Software-related system error |
| Invalid peak | Peak overflow |
| Gate skipped | Peak-specified gate error |
| Peak tracking error | Not possible to track the peak. |
| Too many peaks | The total number of peaks exceeds 255. |
| Overflow | The concentration value is too large. |
| Reference peak invalid | Calibration data setting error |
| Total compensation error | Error in calculation of the peak total |
| Out of calibration range | Calibration error |
| Calibration repeat error | Calibration error |
| Calibration coefficient error | Calibration error |
| Lab mode analysis parameter error | Analysis parameter error |
| Chromatogram data error | System error |
| Saved chromatogram data error | Invalid data |
| Saved chromatogram error | The source chromatogram for calculating any differences in saved chromatogram data does not exist. |
| Peak processing information error | Peak processing has failed. |
| Peak detection failure | Peak detection has failed. |
| Message output destination error | No message output destinations exist. |
| Message code selection error | The selected message code number is incorrect. |
| Data send error | Communication error |
| Data receive error | Communication error |
| Measuring signal range overstepped | The measuring signal range is too wide. |

9. MAINTENANCE

This section describes maintenance operations on the system components and storage of the fluids required for analysis.

9.1 Adding Fluids

Check and, if necessary, add eluant (EL) and pure water at fixed periods. If the EL tank and water tank both contain 10 liters, discharging the EL and water at flow rates of 0.2 ml/min and 2.0 ml/min respectively, will make the EL run short after about 830 hours of operation, and the water, after about 83 hours.

Firmly cap respective fluid lines to avoid deterioration of the fluids and entry of dust and dirt.

9.2 Checking for Alarms

Check that there are no occurring alarms such as a temperature or pressure alarm.

9.3 Checking the Pressures

Keep a record of pressures. If they become abnormal, check for clogging of the damper filter or tubing connections.

9.4 Checking the Base Conductivity

For standard cation analysis, the base conductivity of 1 mM HNO_3 ranges from 400 to 500 $\mu\text{S}/\text{cm}$. Check for deviations from this normal conductivity range. Be careful that the difference in conductivity is not due to the particular oven temperature.

9.5 Routine Maintenance of Pumps

9.5.1 Routine check

To ensure normal operation of the Pi500 internal pumps over long periods, the pumps require appropriate periodic maintenance and replacement of parts. The performance of plunger seals, check valves, tubing parts, etc. decrease with time, and unusual wear on these components causes fluid leakage and abnormal pump pulsations. Replace worn-out components as soon as possible.

| Check item | Checking method and corrective action required |
|--------------------------|--|
| Leakage from fluid lines | Closely observe the tube connections, and if leakage is found, retighten the nuts and bolts on the connections or replace the seal(s). |
| Pressure check | Make it a routine daily practice to investigate abnormal changes in pressure. If possible, the system operating log should be filled in with the column status, analysis parameters, and pressure changes. Increases in pump pulsations should also be logged. |

9.5.2 Flushing the EL line

Some specific types of eluants (ELs) corrode the system. High concentration salt buffer solutions, in particular, may have their salt precipitated and damage the EL line. Therefore, wash the inside of the EL line using a flushing agent after operation.

9.5.3 Flushing the pump head

After using a salt solvent, feed water from the hole provided on the pump head (see Fig. 9.1) and wash the rear side of the plunger seal. Use a suitable injector, such as a syringe, to feed water. Use tissue paper to absorb the waste water flowing out from the bottom hole.

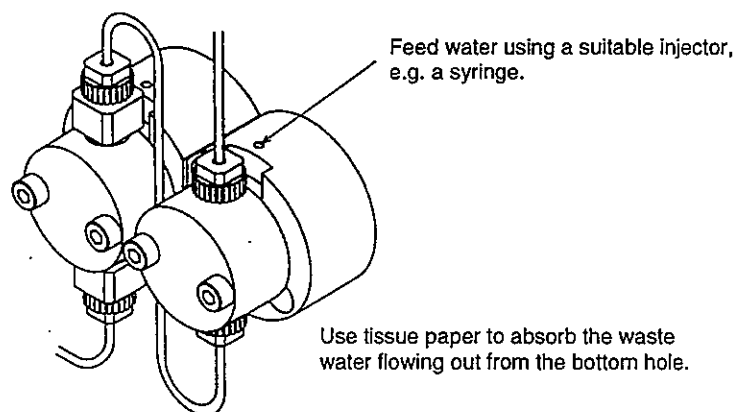


Figure 9.1 Flushing the pump head

9.5.4 Replacing the plunger seals

Replace both plunger seals using the following procedure:

- (1) Stop the pump.
Wait until pressure has decreased.
Turn power off.
- (2) Disconnect the tubing from the pump head.
- (3) Remove the four setscrews from the pump head. See Fig. 9.2.

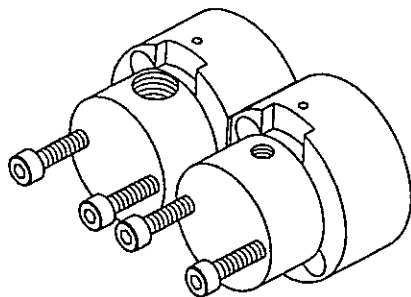


Figure 9.2 Removing the head setscrews

- (4) Gently remove the pump head keeping it horizontal. See Fig. 9.3.

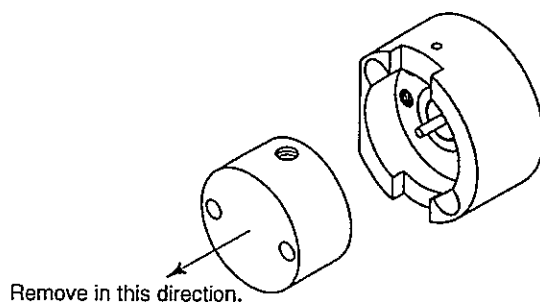


Figure 9.3 Removing the pump head

- (5) Remove the plunger seal from the pump head. See Fig. 9.4.
If the plunger seal still remains on the pump head, the seal can be easily removed by slightly moving the pump and then moving the plunger forward as shown in Fig. 9.4.

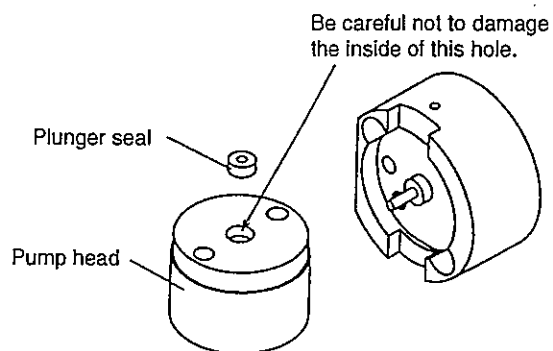


Figure 9.4 Removing the plunger seal

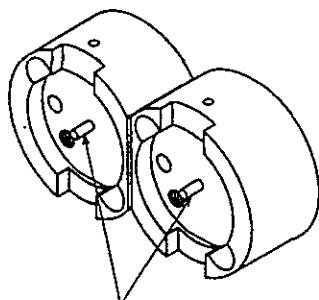
- (6) Clean the plunger seal insertion hole completely with pure water. If the hole is dirty, perform ultrasound cleaning or remove the dirt taking care not to damage the inside of the hole.



NOTE

Very dirty pump head interiors suggest that the check valves are also dirty. If this is the case, remove the check valves from the pump heads and clean the valves.

- (7) Move the pump slowly and completely remove any dirt from the plunger using a wiper or gauze dampened with clean water or organic solvent. See Fig. 9-5.



Remove any dirt from both plungers.

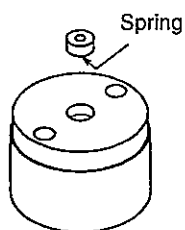
Figure 9.5 Cleaning the plungers

- (8) Mount new plunger seals on the pump head.



NOTE

Orient the spring backward and insert the seal straight into the hole in the pump head. See Fig. 9.6.



Insert the seal straightly.

Figure 9.6 Mounting a new plunger seal

- (9) After performing checks 1, 2, and 3 shown in Fig. 9.7, remount the pump head horizontally.

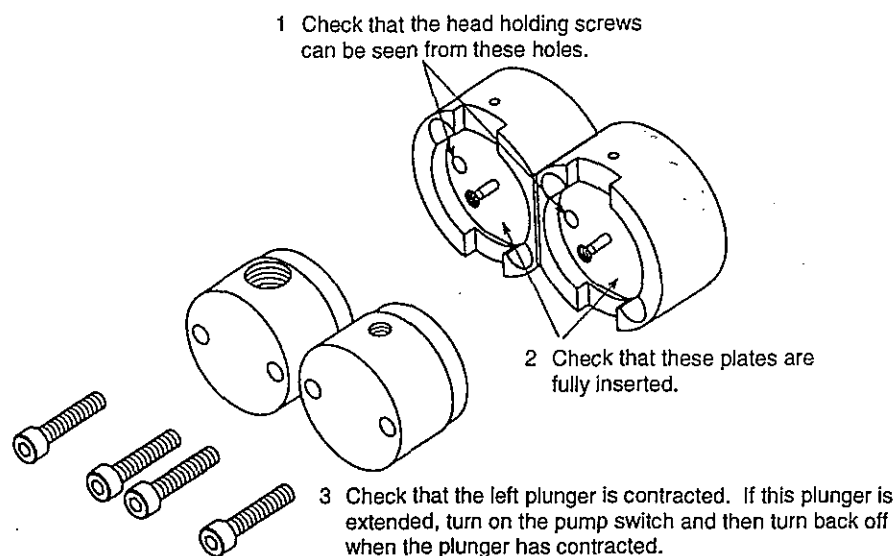


Figure 9.7 Remounting the pump heads

- (10) Fix the pump heads as follows (see Fig. 9.7):
- (10-1) Use your thumb to hold down the center of the pump head firmly.
 - (10-1) Hand-tighten the four screws lightly.
 - (10-3) Retighten the screws one at a time using an Allen wrench, and ensure each is tightened securely.



NOTE

Perform steps (10-2) and (10-3) above with the center of the pump head held down.

- (11) Remount the tubing parts and the check valves are in their original positions. See Fig. 9.8.

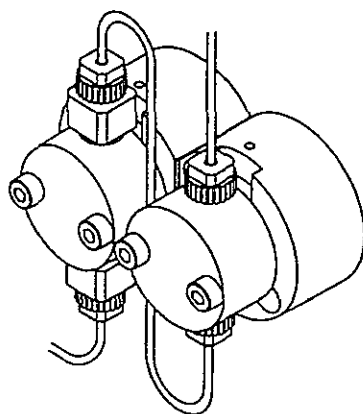


Figure 9.8 Remounting the tubing parts and the check valves

- (12) The pump pulsations immediately after the plungers have been replaced will be significant until the new seals are acclimatized. Since the plungers cannot be used under this state, the pumps need run-in operation. Run in the pumps as follows:
- (a) Mount a resistance tube or an old column, instead of the analytical column. The waste from the mounted resistance tube or old column can be drained directly.

- (b) Supply the pumps with the same EL or pure water that is be used for the pumps.
- (c) Start the pumps after priming. Adjust the pressure to a value higher than that which is to be actually used as the final operating pressure. Run in the pumps at a pressure of at least 50 kgf/cm² to ensure acclimatization of the seals. Terminate the run-in operation when the pressure stabilizes with a fluctuation spread of about 5 kgf/cm².

9.5.5 Cleaning the check valves

Disconnect the tubing connected to the check valves as shown in Fig. 9.9. Remove the check valves using a wrench and/or other tools, and then proceed to the procedure explained after Fig. 9.9.

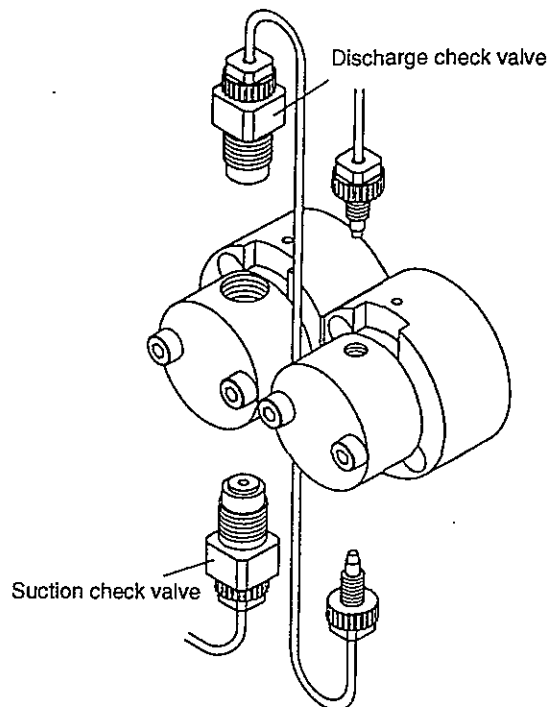


Figure 9.9 Removing the tubing and the check valves

- (1) Put pure water and one check valve into a beaker, and clean the valve using an ultrasound cleaning machine. Do this for both check valves.



NOTE

Clean the check valves one at a time to prevent one from contaminating the other.

- (2) After cleaning, remount the check valves as follows:
Hand-tighten the valves as much as possible. Next, tighten both by between 45 to 90 degrees using a wrench. The tightening operation can be stopped when more strength is required to retighten the valves within this range.
- (3) After the check valves have been remounted, reconnect the tubing. Immobilize the check valves by locking them securely with wrenches, and tightening the bolts and nuts on the tubing.
- (4) After retubing, prime the pumps and supply liquids. If the liquids leak from the check valves, retighten the valves a little.

- (5) If it is not easy to prime the pumps whose check valves have been cleaned, replace the check valve with a new one.

9.6 Replacing the Columns

9.6.1 Replacing the separation column

Replace the separation column using the following procedure: Note: skip to step (2) below if the Pi500 is used alone.

- (1) Remove the two screws located on the bottom of the right-hand side plate (viewed from the CM500). Slide the side plate backward about 10 cm and remove so as to expose the oven door of the Pi500. See Fig. 9.10.



CAUTION

Remove the side plate taking care not to drop it. Dropping the plate may result in bodily injury.

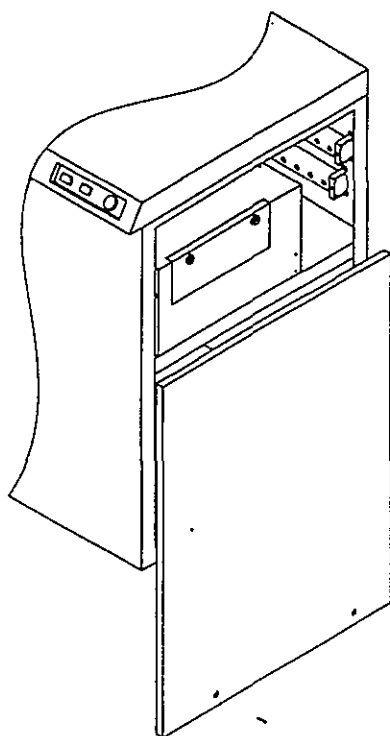


Figure 9.10 Removing the side plate of the CM500

- (2) Remove the oven door, and remove the front plate of the column oven. See Fig. 9.11.

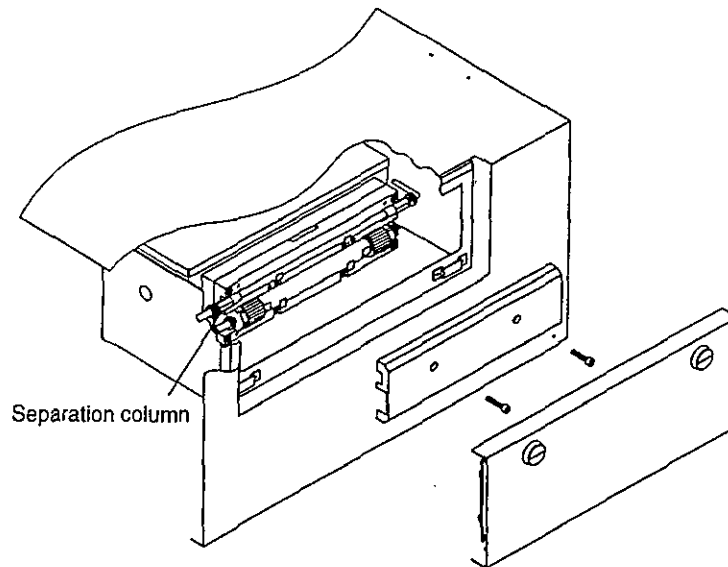


Figure 9.11 Removing the oven door and the front plate

- (3) Remove the separation column from the column holder and disconnect the tubing connected to the holder. Cap the removed column.
- (4) After ensuring the liquid is filled up to the tubing located immediately in front of the column, connect the column and tubing and fix them to the holder.
- (5) Remount the front plate, the door, and the side plate, in the reverse order required for removal, that is, follow step (2) first and then step (1).

9.6.2 Replacing the concentration column

Proceed as follows to replace the concentration column:

- (1) Open the front door of the Pi500, and remove the front panel located above the EL pumps. See Fig. 9.12.

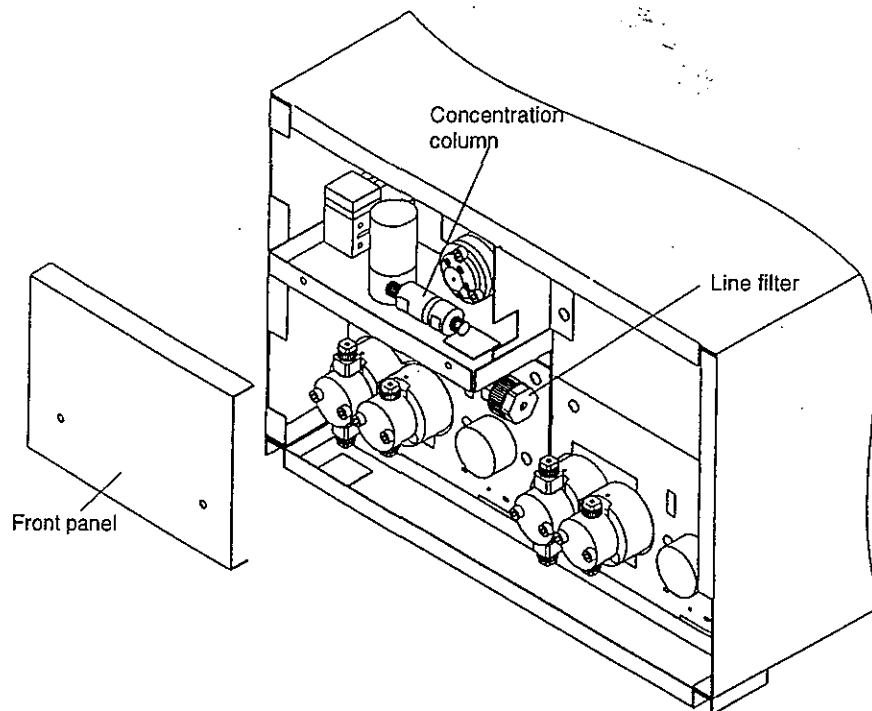


Figure 9.12 Removing the front panel

- (2) Disconnect the tubing from the concentration column, and remove the column. Cap the removed column.
- (3) After ensuring the liquid is filled up to the tubing located immediately in front of the column, connect the column and tubing. Connect so that the direction of flow from the column is the same as the flow from the EL pumps.
- (4) Remount the front panel and close the front door.

9.7 Replacing the Line Filter

The line filter replacement procedure is as follows:

- (1) Disconnect the tubing from the line filter, and slide the filter horizontally to remove it from the holder.
- (2) Open the body of the line filter using a wrench, and remove the filter inside. See Fig. 9.13.
- (3) Mount a new filter and close the body using a wrench.
- (4) Reconnect the tubing and fix it to the holder. The line filter must have its thin (hole) section facing upstream and its thick section facing downstream so that the direction of flow of the EL and that of the filter are matched.

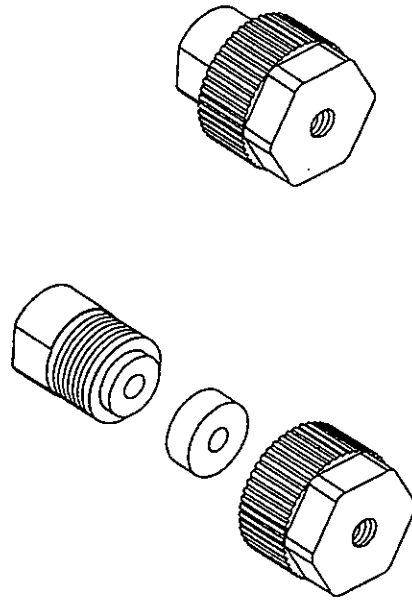


Figure 9.13 Disassembling the line filter