# CDD-10Avp/10Asp CONDUCTIVITY DETECTOR INSTRUCTION MANUAL

Read the instruction manual thoroughly before you use this unit. Save this instruction manual for future reference.

### SHIMADZU CORPORATION

ANALYTICAL INSTRUMENTS DIVISION

## Introduction

#### Read this manual thoroughly before using the instrument.

Thank you for purchasing this product. This manual describes: the installation, operation and hardware validation procedures, precautions for use, and details on accessories and options. Read the manual throughly before operating the unit, and operate the unit in accordance with the manual's instructions. Keep this manual for future reference.

- **IMPORTANT:** Do not operate this unit before fully understanding the contents of this manual.
  - Give this manual to the next user in the event that the unit is borrowed or sold.
  - If this manual or the unit's warning labels become lost or damaged, promptly obtain replacements from your Shimadzu representative.
  - To ensure safe operation, read the manual's Safety Instructions before operating the unit, and follow the procedures described in the manual. Otherwise, unit safety may be compromised.

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The CDD-10Asp and the optional parts for the CDD-10Avp cannot be used in some countries due to a patent restriction. Contact your Shimadzu representative for more information.

# Warranty and post-sales service

#### Warranty

#### 1. Validity

Please consult your Shimadzu representative for information about the extent of the warranty.

#### 2. Terms

The manufacturer will provide free replacement parts for, or repair free of charge, any instrument that fails during the warranty period, if the cause can be attributed to a defect in manufacturing.

#### 3. Items not covered by the warranty

The warranty does not cover malfunctions that result from:

- a) misuse;
- b) repairs or modifications made by any company other than the manufacturer or an approved company;
- c) external factors;
- d) operation under severe conditions, such as: environments with high temperature, high humidity, corrosive gas, vibration, etc.;
- e) fire, earthquake or other forces of nature;
- f) moving or transporting the unit after its initial installation. The warranty does not cover replacement of consumable items or parts that can be regarded as consumable. (For example, the service life of an LCD display panel depends upon actual operating conditions.)

# Post-sales service

If any problem occurs with this unit, inspect it and take appropriate corrective action as described in "9. Troubleshooting." If the problem persists, or symptoms not covered by the Troubleshooting section occur, contact your Shimadzu representative.

# Replacement parts availability

Replacement parts for this unit will be available for a period of ten (10) years after the discontinuation of the product. Thereafter, such parts may cease to be available. Note, however, that the availability of parts of the unit not manufactured by Shimadzu shall be determined by the relevant manufacturers.

# Hardware validation

The unit and the systems should be checked periodically to ensure that they function normally, or the analytical data may not be reliable. To this end, it is necessary to carry out periodic hardware validation and keep records of the validation. There are two types of hardware validation - component validation and system validation. The purpose of component validation is to check that the individual components of the system function normally, while that of system validation is to check that the system as a whole (the components in combination) functions normally.

Before shipment from the factory, this unit was inspected carefully. Detailed information on the hardware validation can be found in "8. Hardware Validation."

#### Service contract

A service contract is available where a qualified Shimadzu-approved service person performs periodic inspection of the components and the system, and provides reports of results. Details of the contract can be obtained from your Shimadzu representative.

# CONTENTS

#### Introduction

Chapte	Safety Instructions 1	- 1
1.1	ecautions for Safe Operation	- 2
1.2		- 7
1.3	**************************************	- 10
1.4		- 11
Chapte	Overview and Features 2	1
2.1	verview and Features	2
Chapte	Component Description 3	
3.1	ont	- 2
3.2	ack	3 - 3
3.3	eil (CDD-10Avp)	- 4
Chapte	Accessories and Options4	- 1
4.1	ccessories4	- 2
4.2	ptional Parts	1- 3
Chapte	5 Installation Procedures 5	5 - 1
5.1	stallation site5	5 - 2
5.1	Suitable sites, and site preparation	5 - 2
5.1	Required installation space	5 - 3
5.2	stallation	5 - 4
5.2	Installation	5 - 4
5.2	Stacking brackets	5 - 4
5.3	onnecting the Power Supply5	5 - 6
5.3	Connection to power outlet	5 - 7
5.4	lumbing Preparation5	5 - 9
5.4	Tubing and connection types	5 - 9
5.4	Cutting the tubing	5 - 9
5.4	Connecting tubing	5 - 9
5.4	Precautions when using PEEK	5 - 10
5.4		5 - 12

5.5 E		
E E 1	Plumbing	
	Waste container preparation	
5.5.2		
5.5.3		
	Installation of Manual Injector and Column	
	Flow Line Plumbing	
5.7.1	<i>y</i>	
5.7.2		
5.7.3	3	
5.8 V	Wiring	
5.8.1	Connectors	
5.8.2	Connecting the flow cell cables	5 - 2
5.8.3	Connecting the fiber optic cable	5 - 2
5.8.4	Connection to system controller	5 - 2
5.8.5	Connection to Chromatopac	5 - 2
5.8.6	Connection to strip-chart recorder	5 - 3
5.9	Cleaning the flow lines	5 - 3
-	6 Operation	
-	Precautions	
6.1 I	•	6 -
6.1 I	Precautions	6 - 6 -
6.1 II 6.2 I 6.2.1	Precautions	6 - 6 - 6 -
6.1 II 6.2 I 6.2.1 6.2.2	Precautions	6 - 6 - 6 -
6.1 H 6.2 I 6.2.1 6.2.2 6.3 T	Precautions Display Panel and Keypad Display panel Keypad	6 - 6 - 6 - 6 -
6.1 H 6.2 I 6.2.1 6.2.2 6.3 7 6.4 I	Precautions	6 - 6 - 6 - 6 -
6.1 H 6.2 I 6.2.1 6.2.2 6.3 7 6.4 I	Precautions Display Panel and Keypad Display panel Keypad Turning Power ON/OFF Display Panel Types of screens	6 - 6 - 6 - 6 -
6.1 H 6.2 I 6.2.1 6.2.2 6.3 T 6.4 I 6.4.1 6.4.2	Precautions Display Panel and Keypad Display panel Keypad Turning Power ON/OFF Display Panel Types of screens	6 - 6 - 6 - 6 - 6 - 1
6.1 H 6.2 I 6.2.1 6.2.2 6.3 7 6.4 I 6.4.1 6.4.2 6.4.3	Precautions Display Panel and Keypad Display panel Keypad Furning Power ON/OFF Display Panel Types of screens Setting screens	6 - 6 - 6 - 6 - 6 - 1
6.1 H 6.2 I 6.2.1 6.2.2 6.3 7 6.4 I 6.4.1 6.4.2 6.4.3	Precautions Display Panel and Keypad Display panel Keypad Turning Power ON/OFF Display Panel Types of screens Setting screens VP function screens	6 - 6 - 6 - 6 - 6 - 1 6 - 1
6.1 H 6.2 I 6.2.1 6.2.2 6.3 7 6.4 I 6.4.1 6.4.2 6.4.3	Precautions Display Panel and Keypad Display panel Keypad  Furning Power ON/OFF Display Panel Types of screens Setting screens VP function screens Sasic Settings Setting cell temperature	6 - 6 - 6 - 6 - 1 6 - 1
6.1 H 6.2 I 6.2.1 6.2.2 6.3 7 6.4 I 6.4.1 6.4.2 6.4.3 6.5 I 6.5.1	Precautions  Display Panel and Keypad  Display panel  Keypad  Turning Power ON/OFF  Display Panel  Types of screens  Setting screens  VP function screens  Sasic Settings  Setting cell temperature  Setting GAIN	6 - 6 - 6 - 6 - 1 6 - 1 6 - 1
6.1 H 6.2 I 6.2.1 6.2.2 6.3 7 6.4 I 6.4.1 6.4.2 6.4.3 6.5 I 6.5.1	Precautions Display Panel and Keypad Display panel Keypad Furning Power ON/OFF Display Panel Types of screens Setting screens VP function screens Sasic Settings Setting cell temperature Setting GAIN Setting RANGE	6 - 6 - 6 - 6 - 1 6 - 1 6 - 1 6 - 1
6.1 H 6.2 I 6.2.1 6.2.2 6.3 7 6.4 I 6.4.1 6.4.2 6.4.3 6.5 I 6.5.1 6.5.2 6.5.3	Precautions Display Panel and Keypad Display panel Keypad  Furning Power ON/OFF Display Panel Types of screens Setting screens VP function screens Basic Settings Settings Setting cell temperature Setting GAIN Setting RANGE Setting polarity	6- 6- 6- 6- 6- 6-1 6-1 6-1 6-1
6.1 H 6.2 I 6.2.1 6.2.2 6.3 7 6.4 I 6.4.1 6.4.2 6.4.3 6.5 I 6.5.1 6.5.2 6.5.3 6.5.4 6.5.5	Precautions Display Panel and Keypad Display panel Keypad Turning Power ON/OFF Display Panel Types of screens Setting screens VP function screens Sasic Settings Setting cell temperature Setting GAIN Setting RANGE Setting polarity Zeroing a strip-chart recorder	6 - 6 - 6 - 6 - 1 6 - 1 6 - 1 6 - 1 6 - 1
6.1 H 6.2 I 6.2.1 6.2.2 6.3 T 6.4 I 6.4.1 6.4.2 6.4.3 6.5 I 6.5.1 6.5.2 6.5.3 6.5.4 6.5.5	Precautions Display Panel and Keypad Display panel Keypad  Furning Power ON/OFF Display Panel Types of screens Setting screens VP function screens Basic Settings Settings Setting cell temperature Setting GAIN Setting RANGE Setting polarity	6- 6- 6- 6- 6- 6- 1 6-1 6-1 6-1 6-1 6-1
6.1 H 6.2 I 6.2.1 6.2.2 6.3 T 6.4 I 6.4.1 6.4.2 6.4.3 6.5 I 6.5.1 6.5.2 6.5.3 6.5.4 6.5.5	Precautions Display Panel and Keypad Display panel Keypad Turning Power ON/OFF Display Panel Types of screens Setting screens VP function screens Basic Settings Setting cell temperature Setting GAIN Setting RANGE Setting polarity Zeroing a strip-chart recorder Setting RESPONSE Control by SCL-10Asp	6- 6- 6- 6- 6- 6- 1 6-1 6-1 6-1 6-1 6-2 6-2

6.7 Operation of the non-suppressor system	6 - 28
6.7.1 Start of analysis	6 - 28
6.7.2 Anion analysis method	6 - 28
6.7.3 Cation analysis method	6 - 29
6.8 Operation of the suppressor system	6 - 30
6.8.1 Starting the analysis	6 - 30
6.8.2 Anion analysis method	6 - 32
6.8.3 Cation analysis method	6 - 33
6.8.4 Setting the suppressor control parameter (reference)	6 - 33
6.9 Creating and Executing Time Programs	6 - 34
6.9.1 Program commands	6 - 34
6.9.2 Basics of Time Programming	6 - 34
6.9.3 Time program writing flow chart	6 - 36
6.9.4 Creating a typical Time Program	6 - 37
6.9.5 Editing an existing Time Program (viewing, deleting and inserting steps)	6 - 38
6.9.6 Loop Command	6 - 38
6.9.7 Executing a Time Program	6 - 39
6.10 Auxiliary Functions	6 - 40
6.10.1 Summary of auxiliary functions	6 - 40
6.10.2 Auxiliary function descriptions	6 - 41
6.11 VP Functions	6 - 46
6.11.1 Summary of VP functions	6 - 47
6.11.2 Using VP functions	6 - 49
6.11.3 Setting VP series functions	6 - 49
6.12 Cleaning	6 - 61
6.12.1 Cleaning the exterior	6 - 61
Chapter 7 Control by External Devices	7 - 1
7.1 External Control Connections	
7.1.1 Wiring Information	7 - 2
7.1.2 Connecting the event cable	7 - 3
Chapter 8 Hardware Validation	8′- 1
8.1 Hardware Validation Overview	
8.1.1 Types of validation	
8.1.2 Types of hardware validation	8 7

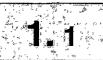
8.2 Hardware Validation Implementation	8 -	4
8.2.1 At installation	8 -	4
8.2.2 Daily inspection	8 -	4
8.2.3 Periodic validation	8 -	5
8.3 Validation Precautions	8 -	6
8.3.1 Environment	8 -	6
8.3.2 Installation site	8 -	6
8.4 Validation Equipment	8 -	7
8.4.1 Component validation equipment	8 -	7
8.4.2 System validation equipment	8 -	7
8.5 Component Validation Procedure	8 -	8
8.5.1 Conductivity span check	8 -	8
8.5.2 Drift/noise check	8 -	9
8.6 System Validation	8 -	14
8.6.1 Validation of non-suppressor system	8 -	14
8.6.2 Validation of suppressor system	8 -	18
Chapter 9 Troubleshooting	9 -	1
9.1 Symptoms, Causes and Corrective Action	9 -	2
9.2 Error Messages	9 -	5
Chapter 10 Maintenance	10 -	1
10.1 Periodic Inspection and Servicing	10 -	2
10.1.1 Preparation for inspection/maintenance	10 -	2
10.1.2 Maintenance operations and intervals	10 -	2
10.1.3 Post-inspection/maintenance leak check	10 -	2
10.2 Flow Cell Cleaning	10 -	3
10.3 Fuse Replacement	10 -	4
Chapter 11 Miscellaneous	11 -	1
11.1 Specifications		
11.2 Replacement Parts		
11.2.1 Replacement Parts	11 -	3



# **Chapter 1 Safety Instructions**

#### CONTENTS

1.1	Precautions for Safe Operation	1 -	-	2
1.2	Static Electricity Precautions	1 -	-	7
1.3	Precautions for Mobile Phase Selection and Use	1	- 1	0
1.4	Warning Labels	1	- ]	1



### **Precautions for Safe Operation**

- In order to operate the unit properly, read these Safety Instructions carefully before use.
- · Be sure to observe all the warnings and precautions described in this section. They are extremely important for safety.
- · In this manual, warnings and cautions are indicated using the following conventions:

### **⚠ WARNING**

Signifies danger of death, or of serious injury, if the instructions given are not followed.

### 

Signifies danger of minor injury, or of damage to objects, if the instructions given are not followed.

- \* "Minor injury:" Injury not requiring hospitalization.
- \* "Damage to objects:" Damage to the unit itself and to other nearby objects.

NOTE

Emphasizes essential information.

In this manual, the symbols described below are used as indicated:

- 1 indicates power ON
- 0 indicates power OFF
- indicates AC (Alternative Current) voltage
- indicates Earth (ground) TERMINAL
- indicates fuse

#### 1. Application precautions



### **⚠ WARNING**

This unit is a conductivity detector for use with a ion chromatography system. Operate this unit ONLY for the purpose for which it is intended. Using this unit for any other purpose could cause accidents.

#### 2. Installation site precautions

### **⚠ WARNING**

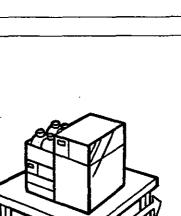
- The solvents used in ion chromatography are flammable and toxic. The room where the equipment is installed should be well-ventilated. Otherwise, solvent vapors could cause poisoning, or ignite and cause a fire.
- Ion chromatography uses the flammable organic solvents. Use of open flame in the vicinity of this unit must be strictly prohibited. Do not install in the same room with equipment that emits or could potentially emit sparks, since sparks could result in fire. Provide fire extinguishers for use in case of fire.



Provide protective equipment near the unit.
 If solvent gets into the eyes, or on the skin, it must be flushed away immediately.
 Provide equipment, such as eye wash stations and safety showers, as close to the unit as possible.

### **⚠** CAUTION

- The lab table or other surface on which this unit is installed should be level, stable, sufficiently strong to support the unit's weight, and have a depth of at least 60 cm. Otherwise, the unit could tip over or fall off the surface.
- Avoid installing the unit where there is corrosive gas or excessive dust. The unit performance could be affected, and its service life shortened.



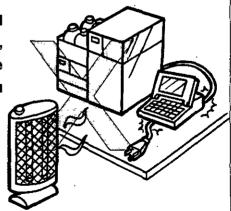
#### 3. Installation precautions

### **MARNING**

- Take measures to prevent the unit falling in the event of an earthquake or other disaster. Strong vibrations could cause the unit to fall over, resulting in injury.
- The power supply voltages and power consumption of this unit are listed below. The power supply voltage of the unit is indicated by the voltage selector on its back, and on the label affixed to its right side. Connect the unit only to a power supply of the voltage indicated. Otherwise, fire or electric shock could result. Check that the power supply voltage is stable and its current capacity sufficient. If not, the unit will not operate at its rated performance levels. Also ensure that the power supply has sufficient current capacity required to operate all the components of the system.

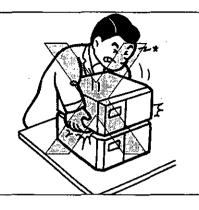
Part	No.	Power supply	Power consumption	Eroguenev
CDD-10Avp	CDD-10Asp	voltage		Frequency
228-41300-31	460-08101-31	AC100V	250VA	50-60Hz
228-41300-32	460-08101-32	AC110V-120V	250VA	50-60Hz
228-41300-38	460-08101-38	AC220-230V/240V	250VA	50-60Hz

- Ground the unit. Grounding is necessary to prevent electric shock in the event of an accident or electrical leakage, and important for ensuring stable operation. (See "5.3.1 Connection to power outlet" on p. 5-7)
- Do not place heavy objects on the power cord, and keep any hot items away. It could be damaged, resulting in fire, electric shock or malfunction. If the cord becomes damaged, contact your Shimadzu representative immediately.
- Do not modify the cord in any way. Also, do not bend it excessively or pull on it. It could be damaged, resulting in fire or electric shock.
   If the cord becomes damaged, contact your Shimadzu representative.



### **⚠** CAUTION

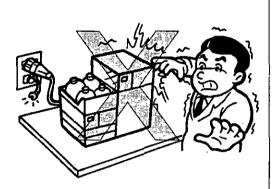
• When installing the unit, be careful not to pinch your fingers between the unit and the unit beneath it.



### 4. Operation precautions

### **MWARNING**

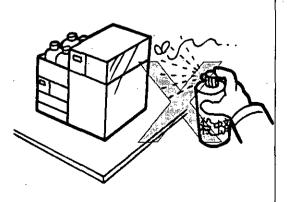
 Take thorough measures to prevent buildup of static electricity. (See "1.2 Static Electricity Precautions" on p. 1-7). Static buildup could result in fires or explosions.



Always wear goggles when handling solvents. If solvent gets into the eyes, blindness could result. Should solvents get into the eyes, flush immediately with large amounts of water and get medical attention.



- If a helium degasser is used, pressure is exerted on the reservoir bottles and may cause cracks in them. NEVER use a cracked bottle. It could break and cause injury.
- Do not use flammable sprays (hair sprays, insecticide sprays, etc.) near this unit. They could ignite and cause a fire.



5. Precautions for inspection, maintenance, adjustment and care of the unit

### **MARNING**

- Unplug the unit before performing inspection, maintenance, or parts replacement. Otherwise, electric shock or short-circuit accidents could occur.
- Never remove the main cover. This may cause injury or malfunction of the equipment. The main cover does not need to be removed for routine maintenance, inspection and adjustment. Have your Shimadzu representative perform any repairs requiring removal of the main cover.



- Replace fuses only with fuses of proper type and capacity. Any other fuses could cause a fire.
- If the power cord plug gets dusty, remove the plug from the power outlet and wipe away the dust with a dry cloth. If dust is allowed to accumulate, fire could result.
- Replacement parts must be of the specifications given in "4.1 Accessories" or "11.2 Replacement Parts." If any other parts are used, they could become damaged and cause a malfunction.
- If any water gets onto the unit, wipe it away immediately to prevent rust. Never use alcohol or thinner solvents for cleaning the unit. They could cause discoloration.

### Static Electricity Precautions

Liquid chromatography (LC) uses flammable organic solvent(s) as the mobile phase. Additionally, LC systems are often used in situations where large amounts of flammable substances are in use, so that once an accident occurs, it can produce large-scale damage. Operators must be constantly on guard against accidents involving fire or explosion.

The major cause of these accidents is static electricity. Devising preventive measures for static can be difficult, because the symptoms preceding an accident vary and can be hard to detect, and because such accidents tend to occur only as a result of several coincidences occurring simultaneously. Recommended methods for preventing static electricity accidents are provided below, along with descriptions of the sequences of events that lead up to them. Take thorough safety measures based on this information.

#### 1. Causes of static electricity accidents (typical example)

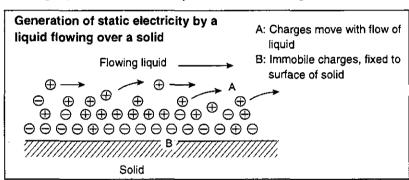
Static electricity accidents are generally caused by this sequence of events:

Electro-static charges and accumulation of static electricity

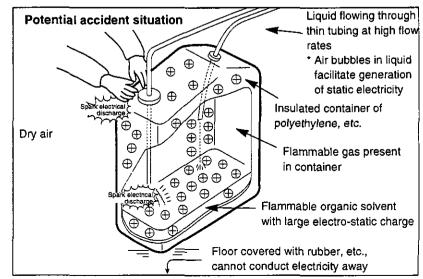
Release of energy through electrical discharge

Ignition of flammable substances

When liquid is passed through thin tubing at high flow rates (as in liquid chromatography), the electro-static charges of the flowing matter generate static electricity, as shown in the figure below.



If electro-statically charged liquid is allowed to accumulate in an electrically insulated container, the charge will gradually increase, and can eventually reach several thousand volts. If this happens and an electrical conductor is brought within a certain distance of the container, electrical discharge will occur, releasing thermal energy which will ignite any flammable gas of sufficient density in the vicinity.



tions .

### 2. Measures for preventing static electricity accidents

The best way to prevent static electricity accidents is simply to prevent the occurrence and accumulation of electro-static charges.

#### NOTE

- It is important to combine multiple preventive measures.
- If large amounts of flammable solvents are collected in a large container, implement preventive measures 1, 2, and 3 below.

#### Preventive measure 1

Use a metal container for the waste liquid, and ground the container. This will ensure that the electrical charges of the container and liquid pass to ground. Accessories for this measure:

1) Grounding wire with clip

Part No. 228-21353-91

2) 18 liter metal container

Part No. 038-00044

3) 4 liter metal container

Part No. 038-00043-01

#### NOTE

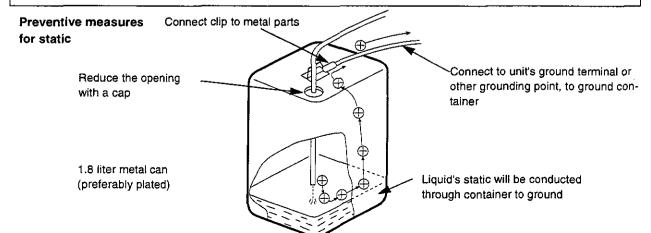
- Be sure to ground the metal waste container properly. If the grounding wire is not properly attached or connected to ground, static electricity can build up in the metal container.
- Some metal containers have surfaces that are laminated or oxidized, and therefore do not conduct
  electricity. After grounding the metal container, use a tester to make sure that electricity is conduct
  ed to ground.
- If the liquid to be drained into the waste liquid container is virtually non-conductive (10<sup>-10</sup> S/m or less), it will be necessary to add properly conductive (and therefore safe) liquid to the tank. (This conductive liquid may be added beforehand.)

#### Preventive measure 2

Cover the spaces between the tubing and the sides of the inlet and outlet openings of the waste container (with caps or the like). This will prevent any sparks generated outside the container from getting inside.

#### Accessories for this measure:

Caps for 18 liter or 4 liter containers (set of 3 caps covering 3 mm diameter openings)
Part No. 228-21354-91



#### Preventive measure 3

Keep electro-statically charged objects, including the human body, away from the waste liquid container. To prevent electro-static charging of the human body, take the following precautions:

- · Wear anti-static clothing and shoes.
- Ground the human body with anti-static wrist straps. (For safety, the wrist strap should be connected to ground using an intervening resistor of about 1MΩ.)
- · Spread anti-static matting or the like on the floor, to make the floor conductive.

#### NOTE

 Persons who have not taken anti-static precautions should touch some grounded metal component before coming near the waste liquid container, in order to ground the body and clothing.

#### Preventive measure 4

Use tubing with an inner diameter of at least 2 mm for a drain line with high flow rates.

#### NOTE

• Air bubbles in liquid can multiply the electro-static charge by a factor of 20, 30 or more. Periodically check the tubing connections for leaks.

#### Preventive measure 5

If it is not possible to use a conductive waste liquid container, take the following precautions:

 Ensure that the end of the inflow tube is always submerged inside the container. Also, place some type or grounded metal object (wire connected to the unit, etc.) in the liquid.

#### NOTE

The above precautions will be ineffective for liquid of low conductivity (less than 10<sup>-10</sup> S/m). For such liquid:

- Use as small a container as possible, to minimize damage in the event of fire.
- Ambient humidity exceeding 65% will prevent static. Keep the room at a proper humidity.

#### For reference

Anti-static equipment (anti-static clothing, shoes and matting) and charge measurement equipment (potentiometer) are sold by specialty manufacturers.

#### **Precatuions for Mobile Phase Selection and Use**

### **!** CAUTION

 If PEEK resin parts are used in the plumbing, do not use the following mobile phases: concentrated sulfuric acid, concentrated nitric acid, dichloroacetic acid, acetone, tetrahydrofuran (THF), dichloromethane, chloroform, dimethyl sulfoxide (DMSO).

These mobile phases weaken the PEEK resin, which could result in cracked plumbing and mobile phase leaks.

Note: Briefly using a weak solution of no more than 0.5% acetone in water (e.g. in order to check gradient performance) will present no problems.

#### NOTE

- Use only HPLC grade or comparable mobile phase, and filter it before use to remove particulates and foreign matter (using a filter of 0.45μm mesh or finer).
- Halogen ions can corrode the stainless steel material (SUS316) used in the plumbing, so avoid, as
  much as possible, mobile phase which contains halogen ions such as KCl, NaCl and NH<sub>4</sub>Cl or
  mobile phase that generates halogen ions in certain reactions. If such mobile phases must be used,
  clean all flow lines thoroughly with distilled water immediately after analysis.
- Always degas the mobile phase. Otherwise, air bubbles tend to form in it during mixing of solvents
  or when a change in temperature or pressure occurs. Air bubbles can cause pump malfunctions and
  detector signal noise.

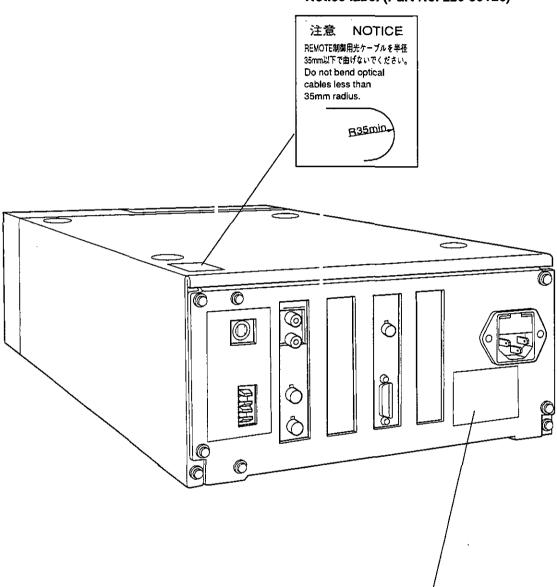


### **Warning Labels**

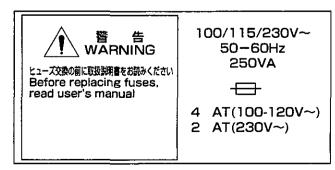
To help users operate the unit safety, warning labels are affixed where special attention is required. Should any of these labels peel off or become damaged, obtain replacements from Shimadzu.



#### Notice label (Part No. 228-35126)



Warning label (Part No. 460-08085)



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# **Chapter 2 Overview and Features**

CONTENTS	2.1	Overview and Features	2 -	· 2
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#### Overview

The Shimadzu CDD-10Avp and CDD-10Asp are high performance conductivity detectors for HPLC. These highly sensitive detectors can be used for a wide variety of applications, especially ion chromatography.

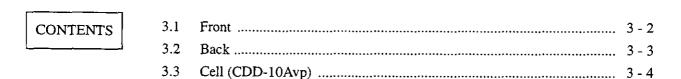
The system configuration is flexible, depending on the application. The CDD-10Avp consists of a main unit and a flow cell, to be installed in the column oven. It is primarily for non-suppressor ion chromatography analysis, but can also be used for other applications, such as post-column derivatization systems.

The CDD-10Asp detector is dedicated for the Shimadzu suppressor ion chromatograph system, and features both high sensitivity and suppressor control capabilities. The suppressor drastically reduces background noise, enabling the detector to exhibit the high sensitivity required in many application fields. (The SCL-10Asp, CTO-10ACsp and other accessories are required when using the CDD-10Asp as a suppressor system.) The CDD-10Asp is one of the composed unit of HIC-10Asuper (suppressor package). The CDD-10Asp is not sold separately.

#### **Features**

- Low noise and wide dynamic range
  With multiple gain settings, the CDD-10Avp/10Asp can measure within a range of 10nScm<sup>-1</sup> to
  100,000μScm<sup>-1</sup> full scale, enabling both high sensitivity detection and analysis of high concentration samples.
- Dual channel detection
   With the dual channel option, the simultaneous detection of cations and anions is possible with a single detector, both for non-suppressor and suppressor systems.
- Suppressor control
   A compact and reliable suppressor system is possible with the CDD-10Asp, which has full suppressor control capabilities for two channels.
- VP functions
   The VP functions provide quick solutions for setting up hardware validation and maintenance procedures, which are essential for GLP/GMP.
- Flexible system configuration
   The CDD-10Avp can also be upgraded for use in a suppressor system by installing the suppressor option.





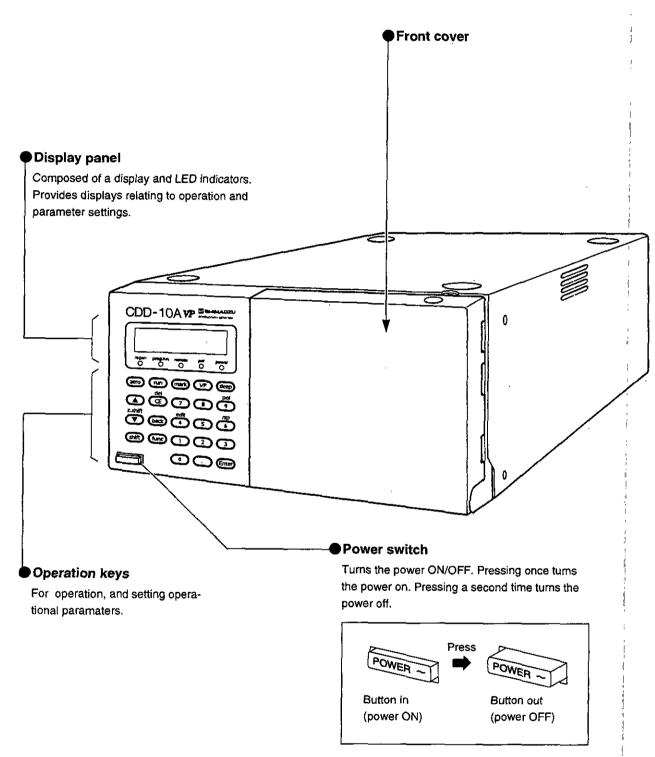


Fig. 3.1

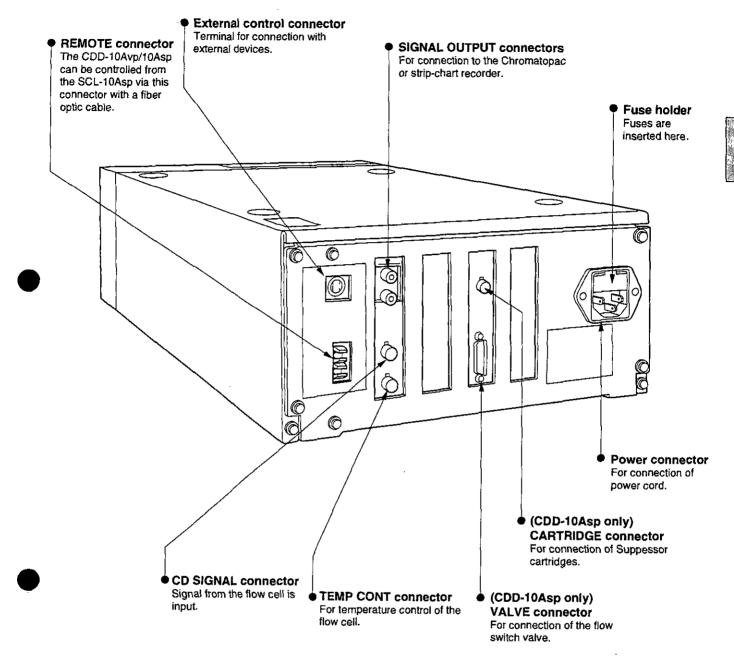


Fig. 3.2

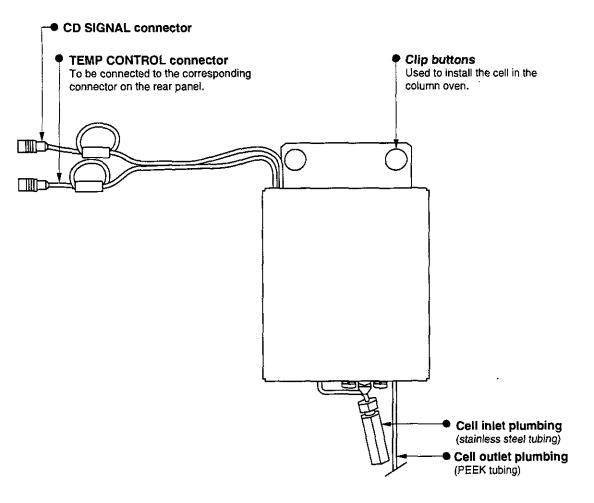


Fig. 3.3

The cell is intended to be used inside the column oven for the double temperature control configuration. Placed in a stabilized oven temperature, the heater inside the cell controls its temperature more precisely.

# **Chapter 4 Accessories and Options**

CONTENTS	4.1	Accessories	4 - 2
	4.2	Optional Parts	4 - 3

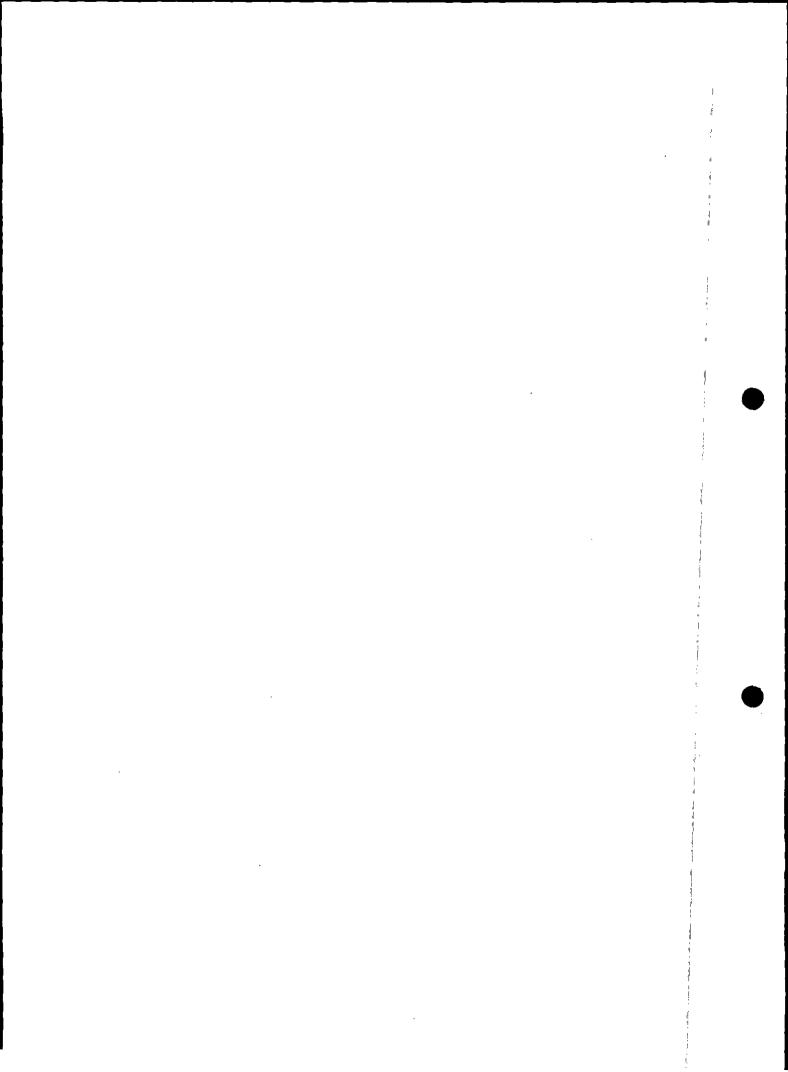
### **Accessories**

This unit consists of the standard parts listed below. Check the parts against this list after unpacking.

Part name	Part No.	Q'ty	Notes
Main body		I	
Power cord (for 100V, 120V)	071-60814-01	1	Not supplied with 230V spec.
Power cord (for 230V)	071-60814-06	1	Not supplied with 100V, 120V spec.
Fiber optic cable	070-92025-51	1	
Drain tubing	228-18495-06	1	2m, Not supplied with CDD-10Asp.
Event cable	228-35048-91	1	
Signal cable	228-35047-92	1	
Terminals	071-03511-01	12	
Label calibration	460-08181	1	Parameter values of
			factory adjustment
Instruction manual	460-08123	1	

For installation of this unit, please order Inert LC plumbing kit (P/N 228-33285-91) as well.

Part Name	Part No.	Usage
2 channel option for non-	228-41302-91	This option equips the CDD-10Avp/
suppressor system	(For all voltages)	10Asp with 2 channel processing
		capability in non-suppressor mode. A
		CDD-10Avp flow cell and the PC-
	}	2CD 2Ch PCB are included.
		·
li .		NOTE
li .		For dual flow line analyses, additional
		accessories are required, such as a
		pump and flow line valve. Ask your
		Shimadzu representative for details.
2 channel option for	228-39881-31 (100V)	This option upgrades the CDD-10Asp
suppressor system	228-39881-32 (120V)	to a dual channel suppressor system. A
	228-39881-38 (230V)	suppressor cell unit, a valve unit for
		cartridge switching, and the PC-4CD
		suppressor Ch2 control PCB are
		included.
		}
		NOTE
		For dual flow line analyses, additional
		accessories are required, such as a
		pump and flow line valve. Ask your
		Shimadzu representative for details.
Suppressor Option	228-41304-31 (100V)	This CDD-10Avp can be upgraded to
	228-41304-32 (120V)	a suppressor system with the PC-3D
	228-41304-38 (230V)	suppressor Ch1 control PCB. The
		Suppressor Option also includes a sup-
		pressor cell unit, a valve unit for car-
		tridge switching and other parts to
		modify the CTO-10ACvp.
		NOTE
		NOTE
		Along with this option, the SCL- 10Asp system controller is required
		for suppressor control. Also, the LC-
		10ADsp should be used as the pump
		for the suppressor system. For details,
		ask your Shimadzu representative.
<u></u>	<u> </u>	ask your simmaded representative.



# **Chapter 5 Installation Procedures**

#### CONTENTS

5.1	Installation site	. 5 -	- 2
5.2	Installation	. 5 -	- 4
5.3	Connecting the Power Supply	. 5 -	- 6
5.4	Plumbing Preparation	. 5 -	. 9
5.5	Plumbing	. 5 -	- 13
5.6	Installation of Manual Injector and Column	. 5 -	. 19
5.7	Flow Line Plumbing	. 5 -	- 20
5.8	Wiring	. 5 -	- 24
5.9	Cleaning the flow lines	. 5 -	- 31

#### 5.1.1 Suitable sites, and site preparation

The site where the unit is installed is of great importance for ensuring safe use.

Install the unit in a suitable location that satisfies the following conditions.

### **MARNING**

#### 1. Ample ventilation

The solvents used with the HPLC system are often flammable and toxic. Therefore, the room where the unit is installed must be well-ventilated.

#### 2. No fire sources used near the unit

The solvents used with the HPLC are often flammable. Therefore, the use of open flame where the unit is installed must be strictly prohibited. Also, do not install in the same room with equipment that emits or could potentially emit sparks.

#### 3. Fire extinguishers permanently available

Have fire extinguishers permanently available in case of fire.

#### 4. Protective equipment provided near the unit

If solvent gets into the eyes, or onto the skin, it must be flushed away immediately. Provide equipment, such as eye wash stations and safety showers, as close to the unit as possible.

### **⚠** CAUTION

#### 1. Avoid dust or corrosive gas

To ensure a long service life for the unit and preserve its performance levels, avoid installing it in places subject to large amounts of dust or corrosive gas.

#### 2. Keep away from equipment generating strong magnetic fields

Do not install the unit in places subject to strong magnetic fields, or it might not operate normally. If the power supply line is subject to high electrical noise, install a surge protector.

#### 3. To preserve the unit's performance levels, select an installation site where:

- room temperature is between 4 and 35°C, with minimal temperature variation in the course of a day
- air currents from heating or air conditioning equipment are not directed on the unit
- sunlight does not shine directly on the unit
- there is no vibration
- humidity stays within 45 85%.

### 5.1.2 Required installation space

### **A** CAUTION

Install this unit on a lab table or similar surface that can support its weight and has ample surface area.

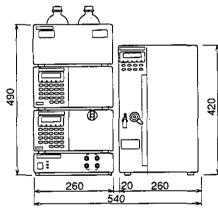
This unit is designed to be used on a lab table or similar working surface. The surface that it is installed on must be firm and level, with a depth of at least 60 cm. Also, there should be a clearance of at least 10 cm between the rear of the table/surface and the wall behind it.

### **ACAUTION**

Do not stack any module which may leak on the CDD-10Avp/10Asp main unit. Unlike other LC-VP modules, the CDD-10Avp/10Asp does not have any leakage plumbing.

Typical system configurations and required installation clearances are shown in the figures below.

CDD-10Avp
 Manual injector system



 CDD-10Avp/10Asp Autosampler system

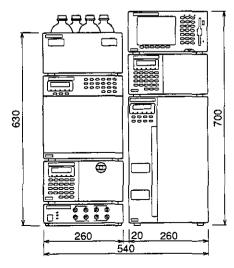
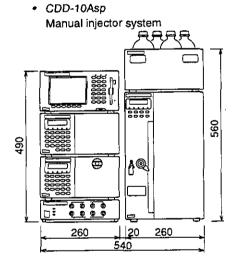


Fig. 5.1



# 5.2

#### Installation

#### 5.2.1 Installation

The CDD-10Avp/10Asp is designed for stacking on other Shimadzu HPLC components. Refer to the preceding section before installing the unit.

NOTE

To ensure optimal detection, the unit should be installed close to the column. (The unit is typically installed on top of the column oven.)



Fig. 5.2

### **ACAUTION**

When a CDD-10Avp/10Asp is stacked on top of another module, the clearance between the two is only 5 mm. Take care not to pinch your fingers.

#### 5.2.2 Stacking brackets

The use of stacking brackets (available commercially) is recommended. These brackets limit the possibility of knocking a module off the lab table or other working surface.

Various brands of stacking brackets are available.

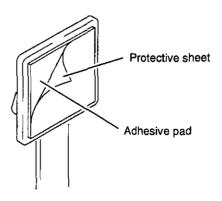
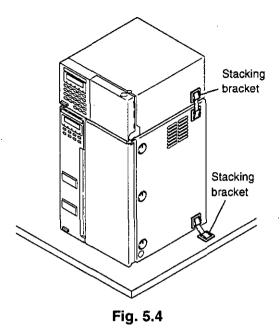


Fig. 5.3



- 1. Peel off the protective sheets from the bracket's adhesive pads.
- 2. Affix one of the bracket's adhesive pads to the unit, and the other to the module above.

### **A** CAUTION

To fasten the unit firmly in place, attach stacking brackets to both its right and left sides.

The table below lists the power supply voltage and frequency for the CDD-10Avp and CDD-10Asp, along with their power requirements.

Part No.		Power	Power	
CDD-10Avp	CDD-10Asp	supply voltage	require- ment	Frequency
228-41300-31	460-08101-31	AC100V	250VA	50/60Hz
228-41300-32	460-08101-32	AC110-120V		
228-41300-38	460-08101-38	AC220-230V/240V		

### **MARNING**

The power supply voltage is indicated on the warning label on the back of the unit. Be sure to connect the unit to a power supply of the voltage indicated. Use of any other voltage could result in fire, electric shock or malfunction.

Make sure that the power supply to which the unit is connected has sufficient current capacity available. Insufficient current capacity could cause outages or voltage drops, affecting not only this unit but also all other equipment connected to the same supply.

#### Warning label (Part No. 460-08085)

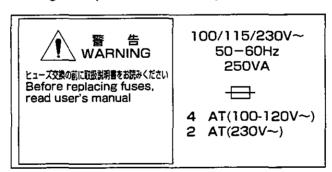


Fig. 5.5

#### 5.3.1 Connection to power outlet

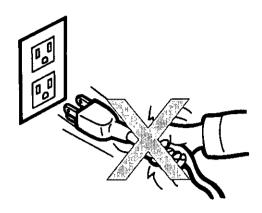


Fig. 5.6

## **⚠ WARNING**

Handle the power cord with care, and observe the following precautions. Failure to do so could result in damage to the cord, fire, electric shock or malfunction.

- Do not place heavy objects on the cord.
- Keep hot items away from the cord.
- Do not modify the cord in any way.
- Do not bend the cord excessively or pull on it.
- To unplug the unit, pull on the plug itself, Not on the cord.

If the cord becomes damaged, have it replaced with a new one immediately.

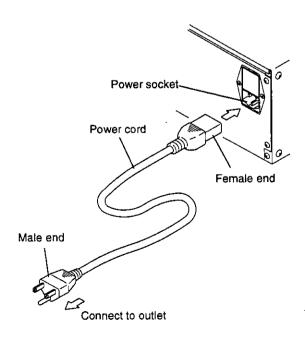


Fig. 5.7



Before connecting the power plug to the outlet, make sure that the unit's power switch is set to OFF.

- 1. Insert the female end of the power cord into the power socket at the back of the unit.
- 2. Insert the male end of the power cord into the power supply outlet.

Many different types of tubing and connectors are used for plumbing the unit at installation. Before the plumbing is connected, tubing must be cut and connectors attached. Instructions and precautions for these preparations are provided below.

#### 5.4.1 Tubing and connection types

The tubing and connectors used for the plumbing are made of resin. The types are listed below. **Resin** 

- FEP tubing, Teflon tubing, Tefzel tubing, PEEK tubing, Tygon tubing, etc.
- PEEK Male nuts
- PEEK ferrules
- Teflon ferrules

### 5.4.2 Cutting the tubing

The tubing supplied must be cut to the lengths necessary for installation.

#### **Cutting resin tubing**

Cut the resin tubing with a razor knife. Be sure to make a straight cut.

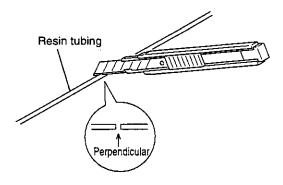


Fig. 5.8

### 5.4.3 Connecting tubing

1. Slide a male nut and a ferrule on the tubing to be connected.

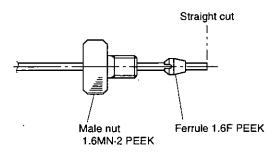


Fig. 5.9

Insert the end of the tubing, with the ferrule on it, into the appropriate opening. Then tighten the male nut. The ferrule will be secured on the tubing.

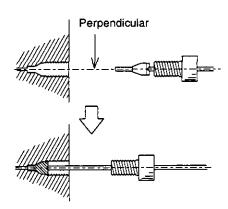


Fig. 5.10

## **⚠** CAUTION

- Insert the tubing completely into the opening, until it butts against the end of the opening. Otherwise, dead volume will be generated and may cause chromatographic peak broadening.
- Do not overtighten the male nut, or the threads will become damaged.

#### 5.4.4 Precautions when using PEEK

PEEK resin has a high mechanical strength and is a material suitable for HPLC. However it has some restrictions on usage, and it is important to observe the following precautions for safe use.

Solvents prohibited for use with PEEK

The following solvents cause stress cracks on PEEK tubing and significantly deteriorate its mechanical strength. Never use these solvents with PEEK tubing;

Concentrated sulfuric acid, concentrated nitric acid, dichloroacetic acid, acetone\*, tetrahydrofuran (THF), dichloromethane, chloroform, dimethylsulfo oxide (DMSO)

\*Note: Briefly using a weak solution of no more than 0.5% acetone in water (e.g., for checking gradient performance) will present no problems.

#### (2) PEEK tubing

The pressure that PEEK tubing can withstand varies depending on the inner diameter, manufacturer and liquid to be used as the mobile phase. When plumbing, be sure to use PEEK tubing of 0.25 mm I.D. or tubing provided by Shimadzu. Shimadzu is not responsible for any problems caused by the use of non-genuine parts.

PEEK tubing of three different inner diameters is provided for inert HPLC plumbing: 0.25 mm, 0.5 mm and 0.75 mm.

The maximum operating pressure of the CDD-10Avp/10Asp cell is 2.9MPa (30 kgf/cm<sup>2</sup>). Do not apply higher pressure to the cell. Also, observe maximum pressures for other modules and columns.

Although organic solvents, such as acetonitrile, are not used in ion chromatography, note that the PEEK tubing pressure calculations were made with organic solvents. For example, if acetonitrile were continuously used with PEEK tubing with pressures exceeding the ranges shown below, the tubing could burst. Do not use PEEK tubing when exceeding these pressure conditions.

0.25 mm I.D. ... 19.6 MPa (200 kgf/cm<sup>2</sup>) or less

0.5 mm I.D. ... 14.7 MPa (150 kgf/cm<sup>2</sup>) or less

0.75 mm I.D. ... 9.8 MPa (100 kgf/cm<sup>2</sup>) or less

(ii) Bending PEEK tubing too far weakens it. Do not bend the tubing such that its radius is less than 10mm. Do not force the PEEK tubing to bend.

PEEK tubing used for instrumentation is heat-formed. When replacing this tubing, use the correct tubing.

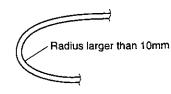


Fig. 5.11

- (iii) Scratches on the surface of PEEK tubing can weaken it. Be careful not to scratch the tubing when cutting it.
- (iv) For safety in the event the wrong solvent is used, cover the PEEK tubing with FEP tubing. Cut the FEP tubing 35-40 mm shorter than the PEEK tubing and slide it over the PEEK tubing. Both ends of the PEEK tubing are exposed for the connections to the nuts.

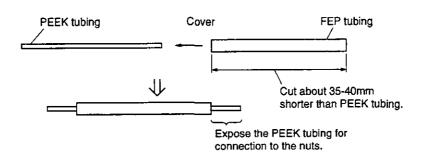


Fig. 5.12

#### 5.4.5 Removing protective plugs

The unit inlets and outlets are fitted with protective plugs (bushings, blank plugs, caps and similar items) to keep out dirt and dust during shipment.

If connections are not made, do not remove the protective plugs. Otherwise, dirt and dust could enter the system and cause clogging.

Keep the plugs, and replace them if the unit will not be used for a long time.

NOTE

For resin plugs:

Remove and replace the plugs by hand.

## **A CAUTION**

- Before connecting the plumbing, turn off the power supply to all the system components, and unplug them.
- For plumbing, use Inert LC Plumbing Kit (P/N 228-33285-91).
- Connect only the tubing described in the instructions.
   Failure to heed the above could result in injury or equipment failure.

The plumbing necessary for this unit is as follows:

- Cell inlet plumbing ....... Tubing that carries mobile phase from the column outlet to the unit.
- Cell outlet plumbing ...... Tubing that drains away mobile phase after analysis.
- Leakage plumbing ....... If leakage occurs in any of the system devices, it is directed to the lowest unit in the stack, and from there out of the system.

#### 5.5.1 Waste container preparation

Before connecting the plumbing, prepare glass or metal waste containers to receive the mobile phase drained after analysis.

## **MARNING**

Do not use cracked or damaged bottles. They could break.

## **⚠** CAUTION

A mobile phase with a low dielectric constant, such as hexane, requires special precautions. Its insulating properties can cause static electricity to collect in the waste liquid container. When using such a mobile phase, use a metal waste container and ground the container.

## **CAUTION**

The waste container must be positioned lower than the unit (for example, on the floor). If it is positioned higher than the unit, liquid will not be drained, and will leak from the connections.

#### 5.5.2 Plumbing (CDD-10Avp)

The CDD-10Avp flow cell should be installed inside the column oven (CTO-10Avp/10ACvp). The following figure shows how to connect the flow lines for a non-suppressor system with a CDD-10Avp. Use Inert LC plumbing kit (P/N 228-33285-91) etc. for connectors and tubing.

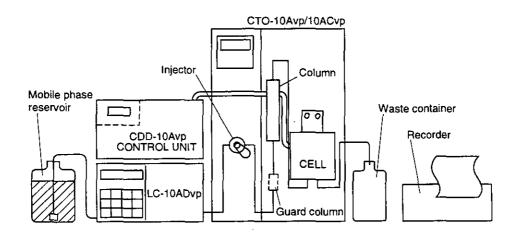
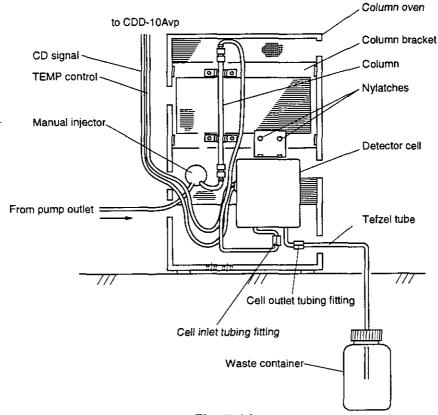


Fig. 5.13 System Connection Diagram

NOTE

If a manual injector is already present in the oven, the detector cell must be installed on the lower right side of the oven interior, as shown in the figure below.



1. Install the cell inside the oven. Insert the Nylatch clips into the appropriate holes on the column bracket, and lock the clips as shown.

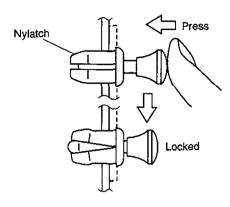
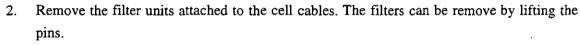


Fig. 5.15



- 3. Route the two cell cables through the middle outlet on the left side of the oven to the exterior of the oven.
- 4. Re-attach the filters to the cable, approximately 2 cm from the connector.
- 5. Cut PEEK tubing to an appropriate length and connect the column outlet to the detector cell inlet tubing.
- 6. Connect the Tefzel tubing to the cell outlet tubing using Male connector, PEEK. Route this Tefzel tubing through one of the oven outlets, and place it in the waste container.

NOTE

To ensure a smooth flow of liquid, verify that the tubing end points down into the waste container.

## 5.5.3 Plumbing (CDD-10Asp)

In a CDD-10Asp suppressor system, the cell and suppressors are installed inside the CTO-10ACsp oven.

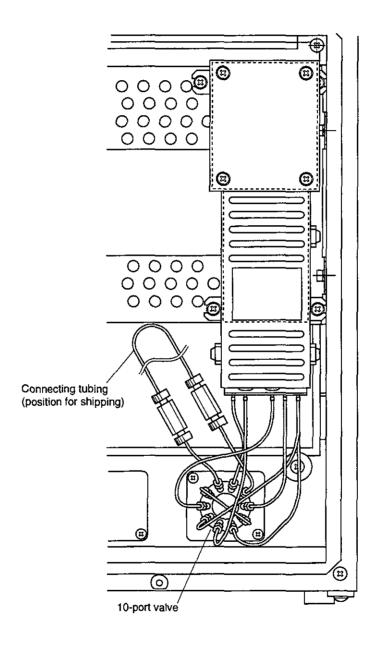


Fig. 5.16

To connect unit tubing, refer to the flow line diagram shown below.

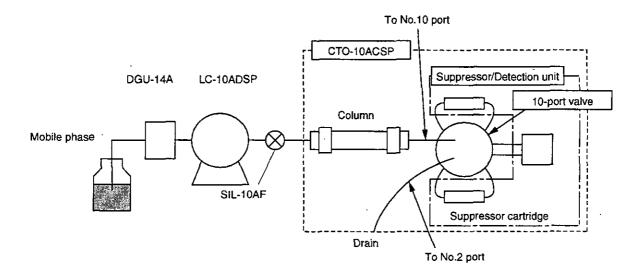


Fig. 5.17

The suppressor/detection unit and the 10-port valve are installed, and all tubing connections are made, inside the column oven. Remove the PEEK tubing that connects port 10 and the 10-port valve. Use this tubing to connect the units. Plumbing for a system with an autosampler (SIL-10AF) or manual injector is shown below.

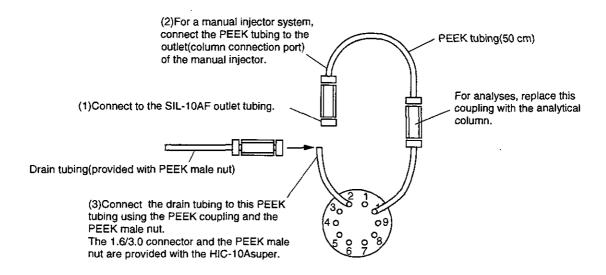


Fig. 5.18

**NOTE** Do not install the analysis column or the suppressor cartridge until the flow lines have been completely flushed.

- For an autosampler system: Connect the autosampler (SIL-10AF) inlet tubing to the pump outlet (LC-10ADsp). Next, remove (1) shown in Fig. 5.18 and connect it to the autosampler outlet tubing.
- For a manual injector system: Connect the pump (LC-10ADsp) to the manual injector inlet pump port with PEEK tubing (0.3 mm i.d.: provided with the HIC-10Asuper.) Next, connect the PEEK tubing shown in (2) of Fig. 5.18 to the manual injector outlet (port where the column is connected).
- Connect the drain tubing to the PEEK tubing located on Port 2 of the 10-port valve. Use the 1. 1.6/3.0 connector and the PEEK male nut for making the connection. (The connector and male nut are provided with the HIC-10Asuper.)
- Connect other tubing, such as FEP tubing, according to the instruction manuals provided with the other units.

#### NOTE

If particles, such as small metal shards, remain inside the tubing when it is connected, the smaller tubing within other units, such as the autosampler, may become clogged. Before connecting the tubing, flush thoroughly with water from the pump to remove any particles.

Tighten PEEK nuts by hand. Do not overtighten the PEEK nut and ferrule when installing the tubing on the 10 port valve. Tighten gradually with the supplied wrench, 30° at a time, only when solvent leaks occur.

Installation Procedures

Injector type	Part No.	Features
Non-metallic manual injector type 9725	228-32650-91	Has parts which contact liquid made of non-metallic materials.  Maximum use temperature: 60°C
Non-metallic manual injector type 9725i	228-32650-93	Same as type 9725, but with a position sensing switch. Can send signals synchronized with injection of samples to system controller or Chromatopac.

Install the manual injector and column as shown in the figures below. For detailed installation procedures, see the Instruction Manual for the pump or the column oven.

• Installed on the pump:

• Installed in the column oven:

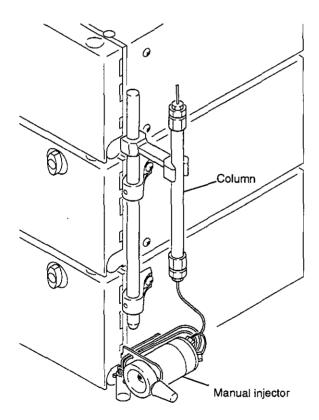


Fig. 19

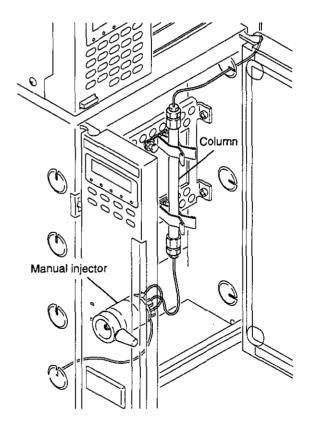


Fig. 20



## Flow Line Plumbing

### 5.7.1 Manual injector plumbing

NOTE

To connect ports 1 to 6 of the manual injector, use the male nuts and ferrules supplied as standard accessories of the manual injector.

- 1. Screw the sample loop male nuts into ports 1 and 4 of the manual injector.
- 2. Slide a male nut and ferrule on one end of each of the two waste liquid tubing sections. Then insert the tubing ends, with their ferrules, into ports 5 and 6 of the manual injector, and tighten the nuts.
- 3. Unscrew and remove the vial cap.
- 4. Route the other ends of the waste liquid tubing through the tubing outlet and into the vial.
- Back of manual injector

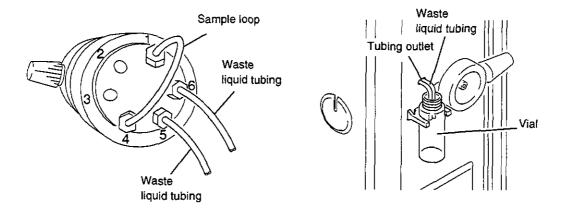


Fig. 5.21

Fig. 5.22

NOTE

To prevent liquid from flowing out due to the siphon effect, position the ends of the waste liquid tubing level with the needle port.

NOTE

The waste liquid tubing should be straight, and perpendicular to the left door. If the tubing curves outward, it could lodge against the side of the unit and prevent closing of the left door.

NOTE

Use PEEK nuts and ferrules for tubing connections with resin parts. Metal nuts and ferrules can damage resin tubing.

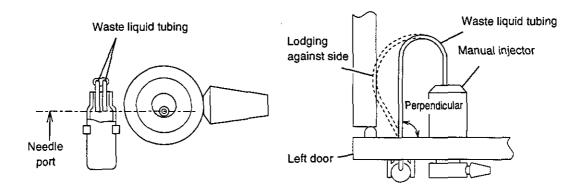


Fig. 5.23

Fig. 5.24

### 5.7.2 Plumbing between pump and manual injector

- 1. Cut the  $\emptyset$  1.6  $\times$  025 PEEK tubing to a length appropriate for connecting the pump outlet and port 2 of the manual injector.
- 2. Slide a male nut and ferrule onto both ends of the PEEK tubing.
  - Pump outlet end: 1.6 MN-2 PEEK male nut and 1.6F PEEK ferrule.
  - Manual injector end: Male nut and ferrule supplied as standard accessories of the manual injector.
- 3. Insert the ends of the SUS tubing into the pump outlet and port 2 of the manual injector, and tighten the male nuts.

NOTE

The tubing should have a little extra length. Otherwise, it will not bend easily, and may prevent closing of the door.

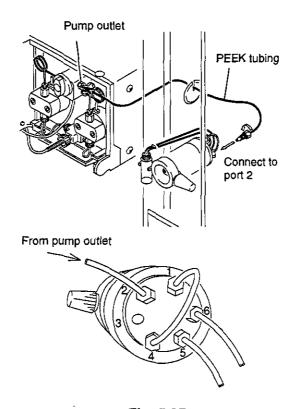


Fig. 5.25

### 5.7.3 Plumbing between manual injector and column

1. Cut the Ø 1.6 × Ø 0.25 PEEK tubing to a length appropriate for connecting port 3 of the manual injector to the column inlet.

NOTE

The tubing should have a little extra length. Otherwise, it may pull on the column when the left door is opened.

- 2. Slide a male nut and ferrule onto both ends of the PEEK tubing
  - Manual injector end: Male nut and ferrule supplied as standard accessories of the manual injector.
  - Column inlet end: PEEK male nut and PEEK ferrule.

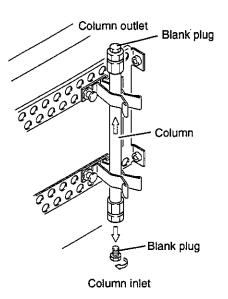


Fig. 5.26

- 3. Unscrew and remove the blank plug from the column inlet.
- 4. Insert the ends of the PEEK tubing into port 2 of the manual injector and the column inlet, and tighten the male nuts.

NOTE

If the tubing has no extra length, unscrew and remove the male nut from the column inlet before opening the left door.

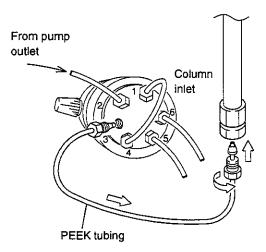


Fig. 5.27

## **⚠** CAUTION

- Before wiring the unit, turn off the power to all the system components and unplug them.
- Use only the cables specified.
- Connect only the cables that the instructions describe.

Failure to heed the above could result in fire, electric shock or malfunction.

#### 5.8.1 Connectors

- REMOTE connector...... For connection to the system controller
- SIGNAL OUTPUT connector

Pin No. 1 (Chromatopac output connector) ...... For connection to Chromatopac

Pin No. 2 (recorder output connector)...... For connection to recorder

connection instructions see "7.1 External

Control Connections" on p. 7-2.

The connectors required depend on the system configuration.

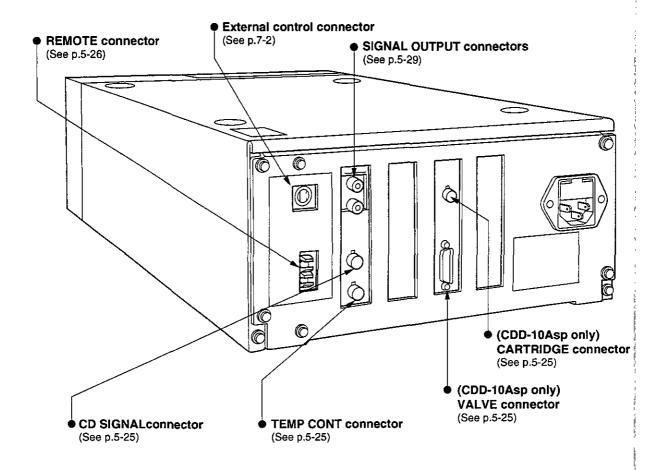


Fig. 5.28

### 5.8.2 Connecting the flow cell cables

## **A CAUTION**

When connecting and disconnecting the flow cell cables, turn the power off to avoide damage to

#### 1. CDD-10Avp/10Asp

Connect the cable labeled CD SIGNAL to the corresponding terminal on the back of the detector. Similarly, connect the TEMP CONTROL cable.

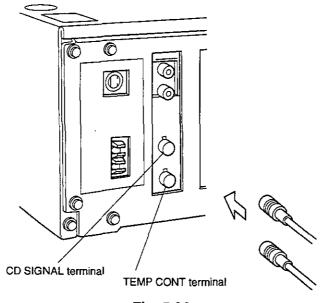


Fig. 5.29

#### 2. CDD-10Asp

Connect the CELL cable from the CTO-10ACsp to the CARTRIDGE terminal on the back of the CDD-10Asp.

#### 3. CDD-10Asp

Using the dedicated cable (Cable Valve Assy) supplied with the HIC-10Asuper, connect the VALVE of the CTO-10ACsp to the corresponding terminal on the back of the CDD-10Asp.

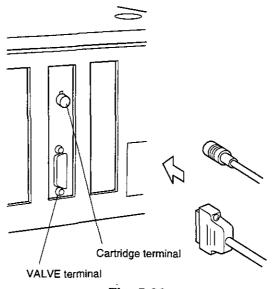


Fig. 5.30

#### 5.8.3 Connecting the fiber optic cable

The fiber optic cable supplied with this unit is a two-way assembly for both transmission and reception of signals, and is connected to the REMOTE terminals.

Instructions and precautions for connecting the fiber optic cable are provided below.

1. Before connection, remove the cap from the connection channel to be used.

#### NOTE

The caps on the REMOTE connectors prevent dirt or dust from getting into the connector.

If a REMOTE connector is not used, leave the cap on it to prevent dirt or dust from interfering with communication.

When a cap is removed, keep it in a safe place for future use.

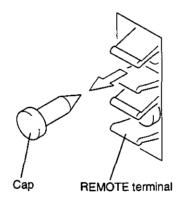


Fig. 5.31

2. Insert the fiber optic cable plug into the REMOTE connector until it clicks into place.

#### NOTE

- Make sure there is no dirt or dust on the plug. Dirt or dust on the plug will get inside
  the REMOTE terminal.
- Be careful not to insert the plug across two different channels.
   Failure to follow the precautions above could result in malfunctions or communication problems.

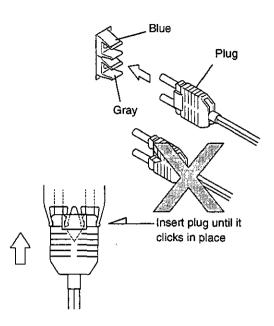


Fig. 5.32

#### NOTE

- Do not bend the fiber optic cable less than 35 mm in radius.
- When inserting and removing the plug, grip the plug itself, not the cable.
- Do not bend the cable where it joins the plug.
   Failure to follow the precautions could result in damage to the plug or a broken wire in the cable.

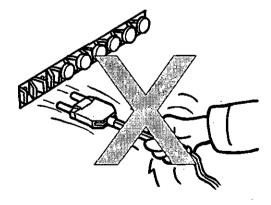


Fig. 5.33

#### 5.8.4 Connection to system controller

1. Referring to "3. Connecting the fiber optic cable," connect the detector REMOTE terminal to the system controller REMOTE terminal with the fiber optic cable.

NOTE

Channels 3-8 of the system controller REMOTE terminal are typically used for this purpose.

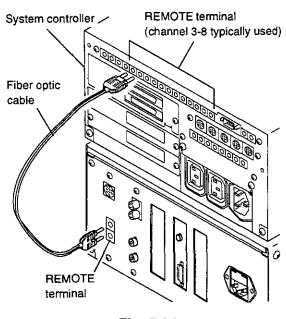
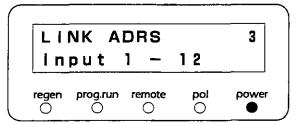
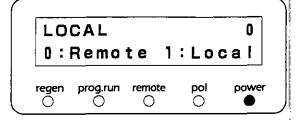


Fig. 5.34

- 2. Insert the power plug into the outlet, and turn the power switch on.
- 3. Set the LINK ADDRESS and LOCAL parameters. (See "6.10 Auxiliary Functions" on p. 6-40)
  - LINK ADRS: Enter the number of the system controller REMOTE channel.
  - LOCAL: Enter "0" (for remote mode).





NOTE

In case of suppressor system, set PUMP (A~C) and DET.A (CDD-10Avp/sp) to SEL state on the System Configuration screen of the system controller. (SEL/DEL state is toggled by f1 key. See "6.5 System Control settings" of SCL-10Avp in struction manual.) When the power switch of a pump is turned off in DEL state, suppressor cartridges may be damaged (broken).

When the power switch of the CDD-10Avp/sp is turned off in DEL state, full regeneration procedure will be required because of the saturation of the cartridge.

#### 5.8.5 Connection to Chromatopac

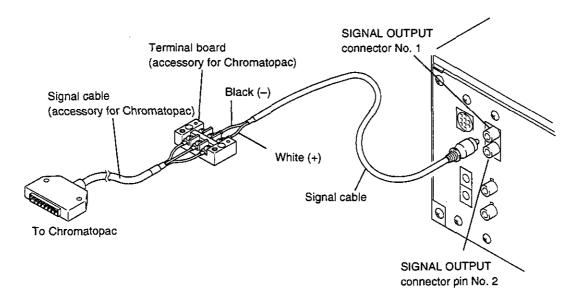
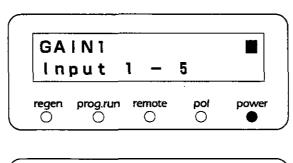


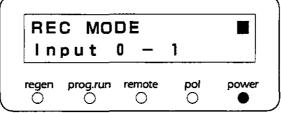
Fig. 5.35

1. Connect the signal cable (supplied) to pin No. 1 of the unit's SIGNAL OUTPUT terminals. Then connect the unit to the Chromatopac as shown in the figure above.

**NOTE**To record channel 2 data, connect an additional signal cable to terminal pin No. 2 (for recorder output).

- 2. Insert the power plug into the outlet, and turn the power switch on.
- 3. Set the GAIN1 and REC MODE parameter (if the 2 channel option is used). (See "6.10 Auxiliary functions," p.6-42.)





### 5.8.6 Connection to strip-chart recorder

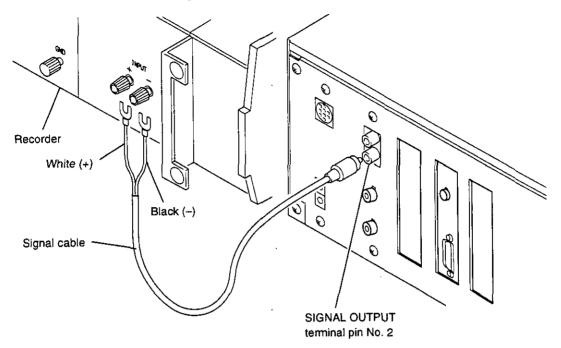
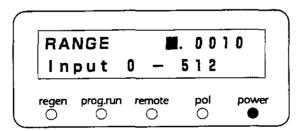


Fig. 5.36

- 1. Connect the signal cable (supplied) to pin No. 2 of the detector SIGNAL OUTUT terminal.
- 2. Connect the other end of the signal cable to the strip-chart recorder input terminals.
- 3. Insert the power plug into the outlet, and turn the power switch on.
- 4. Set the strip-chart recorder range. (see "6.5.3 Settings RANGE" on p. 6-18.)



Similarly, clean the flow lines of systems which have been inactive for over a month to remove possible contamination.

If the column and the suppressor cartridges have already been removed, skip to step 3 below.

- 1. Remove the column. Connect the column inlet tubing directly to the column outlet tubing using the PEEK male connector or coupling C.
- CDD-10Asp only: Slide the suppressor/detection unit cover up and pull it towards you to remove it. Remove the suppressor cartridges from the suppressor/detection unit and connect the inlet to the outlet tubing using the special dummy coupling (dummy coupling, P/N: 228-36573). (Use the dummy coupling that was shipped in the unit in the place of the suppressor cartridge.)
- 3. Turn ON the pump and deliver ethanol at 1.0 mL/min for 10 minutes for cleaning. All cleaning solvents described in this section are delivered at flow rate of 1.0 mL/min.
- 4. Flush with deionized water for 5 minutes, then deliver 1N nitric acid solution for 10 minutes.
- 5. Flush with deionized water for 5 minutes, then 0.1% ethylendiaminetetracetic acid disodium salt (EDTA-2Na) solution for 30 minutes.
- 6. Finally, deliver deionized water for 30 minutes.

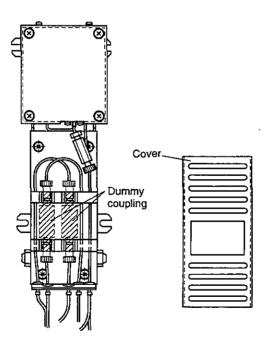
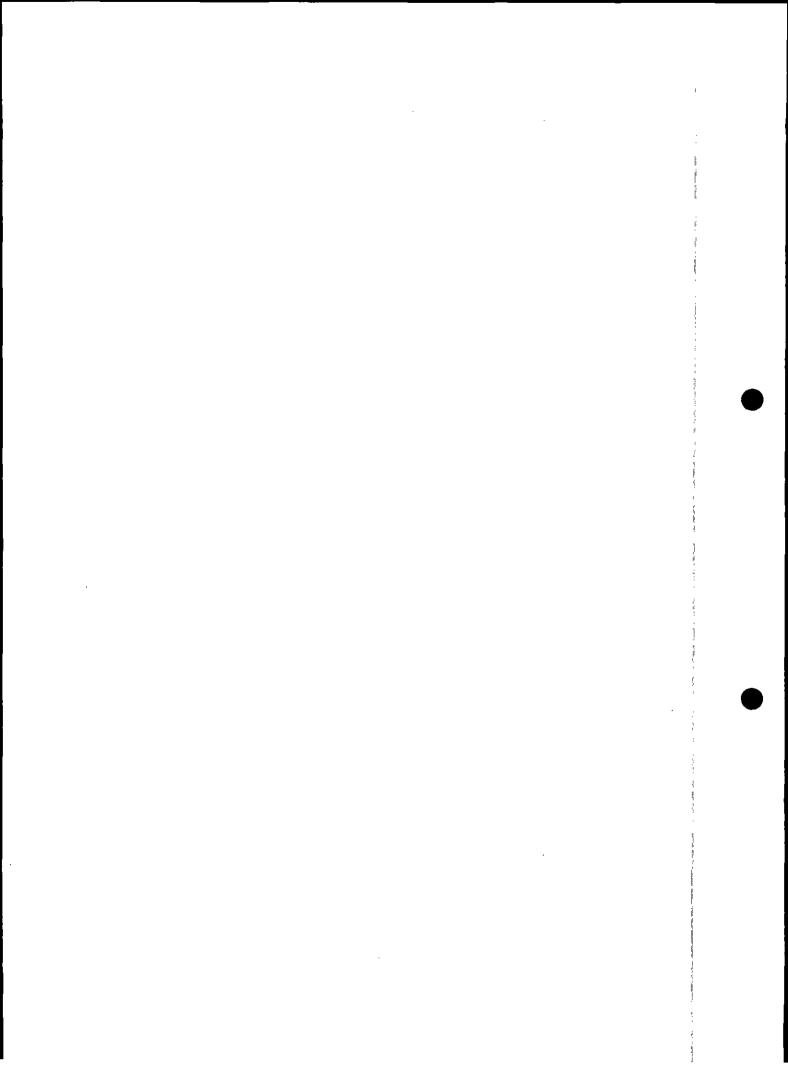


Fig. 5.37



# **Chapter 6 Operation**



6.1	Precautions 6 - 2
6.2	Display Panel and Keypad
6.3	Turning Power ON/OFF
6.4	Display Panel6 - 9
6.5	Basic Settings 6 - 15
6.6	Control by SCL-10Avp/10Asp
6.7	Operation of the non-suppressor system 6 - 28
6.8	Operation of the suppressor system
6.9	Creating and Executing Time Programs
6.10	Auxiliary Functions
6.11	VP Functions
6.12	Cleaning 6 - 61

NOTE To prevent clogging of the flow cell:

> Dirty, clogged flow cells are the most frequent cause of detector problems. After analyzing a highly concentrated sample, flush the flow cell thoroughly, using plenty of mobile phase. When a buffer solution is used as the mobile phase, clean the flow cell with water after completing analysis. Buffer solutions crystallize upon evaporation, and can clog the flow cell and tubing.

## **Display Panel and Keypad**

This unit is operated by the keypad. The display screen provides accompanying displays. The unit can be operated by a system controller instead (see "7. Control by External Devices" for details).

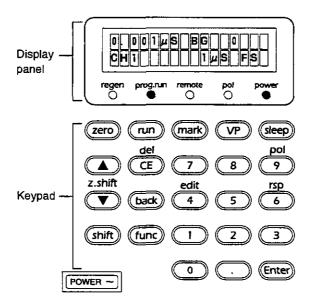


Fig. 6.1

#### 6.2.1 Display panel

The display panel consists of a display screen and LED indicators. Names and functions of the screen sections and the indicators are given below.

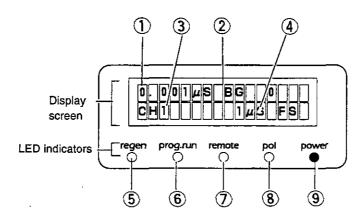


Fig. 6.2

No.	Section/indicator	Function				
1	Conductivity	Displays measurement conductivity (uS stands for µScm <sup>-1</sup> )				
2	Background	Displays background (in µScm <sup>-1</sup> )				
3	Channel	Displays "CH1," indicating CH-1 signal measurement.				
4	range	Displays full scale value ("uS FS" stands for µScm <sup>-1</sup> FS) of sig-				
		nal output to the recorder terminals. Can also display full scale				
		value (uS/V stands for µScm <sup>-1</sup> /V) of signal output to integrator				
		terminals by means of VP function.				
(5)	regen	For the CDD-10Asp, or when the suppressor option is used,				
		this LED indicates that a voltage is being applied to suppressor				
		cartridges.				
6	prog. run	Time program indicator.				
		On when time program is being executed.				
7	remote	Remote control mode indicator.				
		On when unit is controlled by system controller.				
8	pol	Polarity indicator.				
İ		On in reverse polarity output mode.				
9	power	Power indicator.				
		Green when power is on.				
		Orange in sleep mode.				

## 6.2.2 Keypad

The 23 keys of the keypad on the front of the unit are used to operate and set parameters.

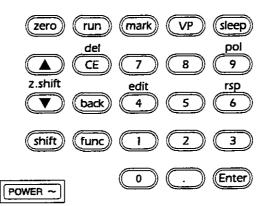


Fig. 6.3

#### <Function keys>

Key	Name	Function		
	Auto zero key	Adjusts recorder zero position, returning baseline to zero		
(zero)		position set with  and  keys. When the 2ch		
		option is used, auto zero is performed for both channels.		
	Zero shift keys	Shifts the recorder zero position. Pressing ( shifts it		
Z.Shift		up; pressing v shifts it down.		
run	Run key	Starts and stops time programs.		
(mark)	Marker key	Draws a mark on recorder chart paper.		
mark)		Has no effect on integrator output.		
\$	VP key	Switches from initial screen to VP mode.		
Clama	Sleep key	Turns off display screen.		
меер		Has no effect on operation.		

#### <Shift-function keys>

To execute shift functions, press the shift key, then the desired func key.

When the shift key is pressed, "Shift pressed" appears on the display screen. Press any key to clear.

Key	Name	Function			
edit (shift) • 4	Edit key	Activates time program edit mode (from initial screen)			
shift • 9	Polarity key	Switches the polarity of recorder output. The [pol] indicator illuminates for (-) polarity.			
shift • 6	Response key	Switches to the response setting mode.			

### <Edit keys>

Key	Name	Function					
0 - 9	Numeric keys	Enter numbers with these keys.					
Enter	Enter key	Validates entries.					
Œ	Clear key	<ul> <li>Initializes the screen.</li> <li>Cancels values input since  was last pressed.</li> <li>Clears error messages and cancels alarms (but does not resolve the source of the error).</li> </ul>					
shift • CE	Delete key	Deletes individual lines of a time program on the display screen (when time program is being written).					
func Function ke		<ul> <li>Scrolls forward through auxiliary functions. Press repeatedly to reach desired parameters.</li> <li>In time program editing, scrolls through list of time-programmable functions.</li> </ul>					
(back)	Back key	<ul> <li>Scrolls backward through auxiliary functions. Press repeatedly to reach desired parameters.</li> <li>In time program editing, scrolls back through list of time-programmable functions.</li> </ul>					

#### Power switch

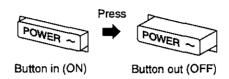


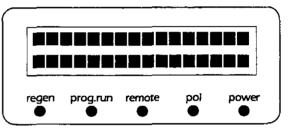
Fig. 6.4

- When the power is turned ON, the following sequence of events occurs:
  - Power ON (1)



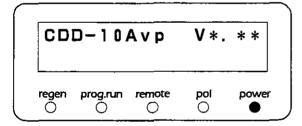
version.)

- All the dots in the display matrix and all the (2) indicator lamps illuminate.
- The unit's memory is automatically checked, (3) and the version number of the control program is momentarily displayed. ("\*. \*\*" in the example screens below represents the ROM

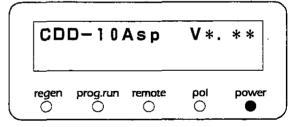




CCD-10Avp



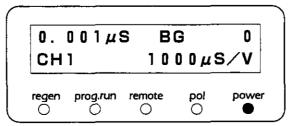
CCD-10Asp





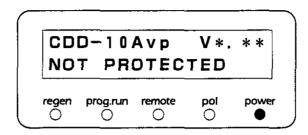
If no error is detected, the initial screen appears, and operation can begin. For the CDD-10Asp, or when the suppressor option is used, the rotary valves are initialized and checked at this point.

Initial screen



• If an alarm sounds and a "NOT PROTECTED" message is displayed on the screen:

Press the key to clear the alarm. When this message is displayed, the time program and certain other parameters will be initialized (replaced with default values).



• If any other error message is displayed: Refer to "9.2 Error Messages" on p. 9-5.

## 6.4.1 Types of screens

The screen can be switched from the initial screen to the various screens described below by operating the keys.

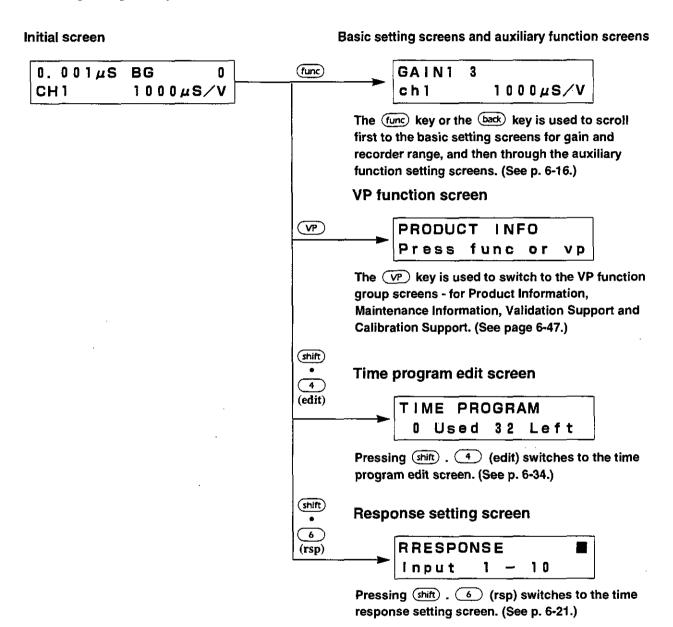


Fig. 6.5

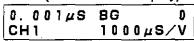
#### 6.4.2 Setting screens

Soon after the power is turned on, the initial screen appears (showing the conductivity, background, and conductivity full scale). To switch to the parameter setting screens, press the key. The display will switch to the first of the screens listed below (screen (1)). Then press repeatedly to scroll through the screens in the sequence given. (Use back to scroll back.)

\* Press (CE) to return to the initial screen.

For details of each setting, refer to "6.10.2 Auxiliary function descriptions"

Initial screen (Ch. 1 monitor display)



(1) Ch. 2 monitor display

••	-	•••		••	<u></u>	<u>~,_</u>				
	0.	0	0	1	μS	В	G		0	
	СН	2	_			1	0	0	0 μS/V	
						- 7				

\*\*This screen appears only when the PC-2CD (2-Channel Option PCB) is installed.

(2) Suppressor Ch. 1 monitor display

SUP1 L:USE R:REG 10.0V 100ohm

% This screen appears only when the PC-3CD (Suppressor Ch. 1 PCB) is installed.

(3) Suppressor Ch. 2 monitor

SUP2 L:USE R:REG 10,0V T00ohm

\*\*This screen appears only when the PC-4CD (Suppressor Ch. 2 PCB) is installed.

(4) Gain setting

(5) Range setting

(6) Cell temperature settting

(7) Recorder mode setting

(8) Cell temperature display

CELL1_T	 000

(9) EVENT terminal setting

EVENT 0 0, 1, 2 or 12

(10) EVENT terminal function setting

(11) Program elapsed time display

MON			0
0:0	ff <u>1</u>	: On	

(12) Keypad disabling

(13) Remote control address setting

(14) Local/remote control setting

(15) Switching suppressor cartridges (Ch. 1)

% This screen appears only when the PC-3CD (Suppressor Ch. 1 PCB) is installed.

(16) Switching suppressor cartridges (ch. 2)

\*\*This screen appears only when the PC-4CD (Suppressor Ch. 2 PCB) is installed.

(17) Hardware configuration

To initial screen

### 6.4.3 VP function screens

VP functions are divided into 4 groups - Product Information, Maintenance Information, Validation Support and Calibration Support.

Pressing the VP key when the initial screen is displayed accesses the Product Information Group screen. Scroll through to the other Groups by pressing VP repeatedly. Functions within the Groups are selected using the func and back keys.

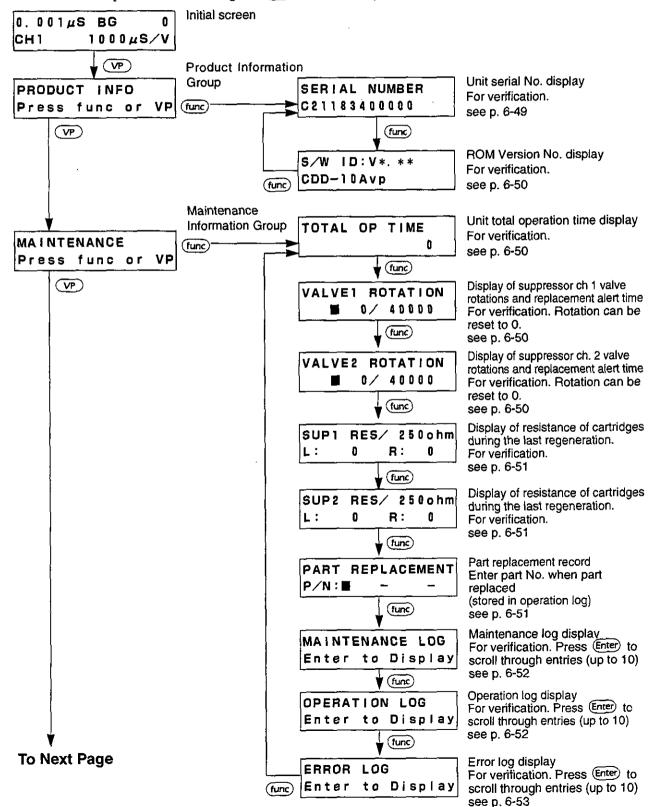




Fig. 6.7

Password input. For setting

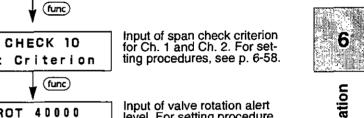
If password input was incorrect, subsequent screens cannot be displayed.

procedure, see P. 6-56.

Zero calibration for Ch. 1.

Press Enter to execute.

see P. 6-56.



Span calibration for Ch. 1. SPAN CALIB CHI Press (Enter) to execute. Enter to Calib See p. 6-57. (func) Zero calibration for Ch. 2. ZERO CALIB CH2 Press Enter to execute. Enter to Calib See p. 6-58. (func) Span calibration for Ch. 2. SPAN CALIB CH2 Press Enter to execute. Enter to Calib See p. 6-57. SPAN CHECK 10 Input Criterion VLV ROT level. For setting procedure, see p. 6-58. Input Alert Lv (func) Input of alert level for Ch. 1 SUP1 RES/ 250ohm suppressor Ch. 2 resistance. Input Alert Lv For setting procedure, see p. 6-59. (func) input of alert level for suppres-SUP2 RES/ 250ohm sor Ch. 2 resistance. For setinput Alert Lv ting procesure, see p. 6-59. (func) Selection of conductivity full RNG DISP MODE 0 scale display. For setting pro-0:REC 1:ITG cedure, see p. 6-59. (func) Parameter initialization. See INITIALIZE PARAM p. 6-59. Enter to Init (func) Changing of password. For CHANGE PASSWORD procedure, see p. 6-59. Enter to change (func) Span adjustment value of Ch. S1 16384 1 conductivity signal. For set-Ch. 1 Span ting procedure, see p. 6-60.

(func)

To Next Page

Calibration Support

Input PASSWORD

ZERO CALIB CHI

Enter to Calib

(func)

(func)

Group

(func)

CALIBRATION

(VP)

Press func or VP

To Product Information Group

6 - 13

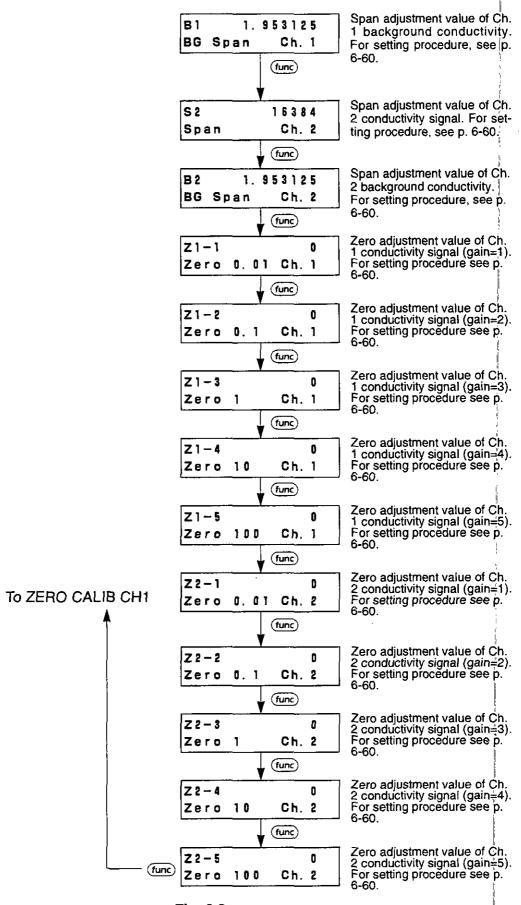


Fig. 6.8

To start an analysis with the CDD-10Avp, the cell temperature and measurement range (gain) should be set, followed by the recording range setting for the integrator (or recorder). These basic operations can be performed from the SCL-10Asp system controller along with settings for the pump and other system components.

For the CDD-10Asp, the settings are mainly performed from the SCL-10Asp system controller for complete suppressor system control.

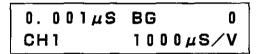
Refer to "6.6 Control by SCL-10Asp" and subsequent sections for details on system controller operation.

### 6.5.1 Setting cell temperature

The cell temperature of the CDD-10Avp should be set 3°C higher than the oven temperature. Follow the procedure below to set the cell temperature.

Example: To change the cell temperature from 35°C to 43°C, assuming the oven temperature is 40°C.

- 1. Press CE to display the initial screen.
- Press func several times until [CELL TEMP] appears.
  - \* The cell temperature parameter field blinks, prompting the user to enter a value for temperature.
- 3. Input 4, 3 and press Enter. The setting is changed and temperature control immediately starts with the new value.



CELL TEMP 85 25 - 55 C 0:off

CELL TEMP 43 1 25 - 55 C 0: off

Operation

### 6.5.2 Setting GAIN

To provide a wide dynamic range, the detector uses five measurement ranges of different sensitivities. The measurement range can be selected by the GAIN1 parameter. (Parameter GAIN2 for Ch2 appears in the menu with the 2-channel option.) The signal processing range of the CDD-10Avp/10Asp changes according to GAIN1/GAIN2 as shown in the table below, which also shows the signal amplitude from the Signal output connector, the required sensitivity, and signal range of analysis.

GAIN1/GAIN2 setting*	Approximate maximum conductivity that can be measured	Signal amplitude of Signal output connector1/ Signal output connector2**
1 (0.01μScm <sup>-1</sup> )	6μScm⁻¹	10µScm <sup>-1</sup> /V
2 (0.1µScm <sup>-1</sup> )	60µScm <sup>-1</sup>	100µScm <sup>-1</sup> /V
3 (1µScm <sup>-1</sup> )	600μScm <sup>-1</sup>	1,000μScm <sup>-1</sup> /V
4 (10μScm <sup>-1</sup> )	6,000μScm <sup>-1</sup>	10,000µScm <sup>-1</sup> /V
5 (100μScm <sup>-1</sup> )	60,000µScm <sup>-1</sup>	100,000μScm <sup>-1</sup> /V

- \* The value in parentheses indicates the most sensitive recording full scale that is available for each gain setting.
- \*\* The signal amplitude of Signal output connector 2 varies according to the RANGE setting.

  This table shows values when the RANGE is set to 10 for the use of the Chromatopac.

### NOTE

A setting of 1 (0.01μScm<sup>-1</sup>) for GAIN1/GAIN2 cannot be used when the background is higher than 100μScm<sup>-1</sup>.

The unit checks the background level when the auto zero function is executed and generates a warning message if it exceeds 100µScm<sup>-1</sup> with a GAIN1/GAIN2 setting of 1. The measurement of peak area and height would not be accurate under these conditions.

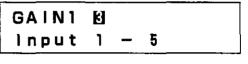
Once a measurement range has been selected for GAIN1/GAIN2, further adjustment of the plot full scale can be performed by setting the integrator's attenuation parameter (ATTEN for Chromatopac), or by setting the RANGE parameter when a strip-chart recorder is used.

A Shimadzu Chromatopac is typically connected to the Signal output connector 1 (integrator output) and this signal is not affected by the RANGE parameter. Setting the RANGE is required when the Signal output connector 2 is used. (See "6.5.3. Setting RANGE".)

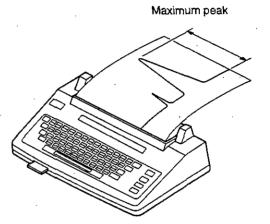
The procedure for setting the GAIN1 parameter is shown below.

Example: To change GAIN1 from 3 to 1.

- 1. Press **CE** to display the initial screen.
- Press func several times until [GAIN1] appears.
- Input i and press Enter. The setting is changed.
  - The Chromatopac full scale produced by various combinations of GAIN1/GAIN2 and ATTEN are shown in the table below. Once a GAIN setting has been selected, the plot full scale can be adjusted by changing [ATTEN]. It is recommended to adjust [ATTEN] so that the maximum peak height of the chromatogram reaches approximately 80% of full scale.



GAIN1 1**■** Input 1 — 5





### GAIN1/GAIN2\* and ATTEN settings vs. plot full scale [µScm<sup>-1</sup>] on the Chromatopac

ATTEN	· · · · · · · · · · · · · · · · · · ·	•	GAIN setting	 J	
setting	1	2	3	4	5
0	0.01	0.1	1	10	100
1	0.02	0.2	2	20	200
2	0.04	0.4	4	40	400
3	0.08	0.8	8	80	800
4	0.16	1.6	16	160	1,600
5	0.32	3.2	32	320	3,200
6	0.64	6.4	64	640	6,400
7	1.28	12.8	128	1,280	12,800
8	2.56	25.6	256	2,560	25,600
9	5.12	51.2	512	5,120	51,200
10	10.24	102.4	1,024	10,240	102,400

\* When a Chromatopac is connected to Signal output connector 2, RANGE should be set to 10 to match the output to the table.

### 6.5.3 Setting RANGE

When a strip-chart recorder is used, it should be connected to the Signal output connector 2 (Recorder output). This connector can also be used for the Chromatopac when two-channel recording is necessary. The signal amplitude of this connector is determined by GAIN1/GAIN2 and RANGE settings.

According to the factory setting, the Signal output connector 2 outputs channel 1 signals. Another parameter, REC MODE, can be set to 1 to assign channel 2 signals to this connector. Refer to <<REC MODE>> on page 6-42 for details.

GAIN setting	Signal amplitude of Signal output connector 2 for RANGE of 1-512
1 (0.01µScm <sup>-1</sup> )	0.01-5.12µScm <sup>-1</sup> /10mV
2 (0.1µScm <sup>-1</sup> )	0.1-51.2µScm <sup>-1</sup> /10mV
3 (1µScm <sup>-1</sup> )	1-512μScm <sup>-1</sup> /10mV
4 (10µScm <sup>-1</sup> )	10-5,120µScm <sup>-1</sup> /10mV
5 (100μScm <sup>-1</sup> )	100-51,200µScm <sup>-1</sup> /10mV

### To use a strip-chart recorder:

- Connect the recorder to the Signal output connector 2.
- Parameter RANGE can be set to 1-512, and the plot full scale is determined as follows for a 10mV strip-chart recorder:

Plot full scale 
$$[\mu Scm^{-1}] = GAIN \times RANGE$$

where GAIN is the value in parentheses in the table on the right, and RANGE is a value between 1 and 512. For example, if GAIN1 is set to 1 (0.01µScm<sup>-1</sup>) and RANGE is 10, the plot full scale is:

 $0.01 \ [\mu \text{Scm}^{-1}] \times 10 = 0.1 \ [\mu \text{Scm}^{-1}]$ 

• The recorder range should be set to approximatery 120% of the expected maximum peak. This will produce a chromatogram with maximum peaks near 80% of full scale.

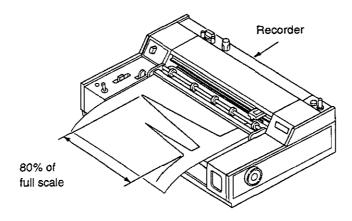


Fig. 6.9

### When the Chromatopac is connected to Signal output connector2:

- Connect the recorder to Signal output connector 2.
- Set RANGE to 10. This will provide the output of this connector with the same amplitude as Signal output connector 1 for the same GAIN setting. Refer to the table in the previous section for the plot full scale on the Chromatopac.

GAIN setting	Signal amplitude of Signal output connector 2 for RANGE of 10
1 (0.01µScm <sup>-1</sup> )	10μScm <sup>-1</sup> /V
2 (0.1µScm <sup>-1</sup> )	100μScm <sup>-1</sup> /V
3 (1µScm <sup>-1</sup> )	1,000μScm <sup>-1</sup> /V
4 (10µScm <sup>-1</sup> )	10,000μScm <sup>-1</sup> /V
5 (100µScm <sup>-1</sup> )	100,000μScm <sup>-1</sup> /V

The procedure for setting the range of the recorder terminal is shown below.

Example: To change GAIN1 from 3 to 1.

- 1. Press (CE) to display the initial screen.
- 2. Press (func) several times until [RANGE] appears on the display.
- Input 1 and press Enter. The setting is changed.

RANGE		6	3		
Input	1	_	5 1	2	

RANGE 512 Input

To return to the initial screen, press **CE**.

#### 6.5.4 Setting polarity

When the conductivity of the target compounds is less than that of the mobile phase, the compound peaks appear as negative in the chromatogram. When this occurs, reverse the polarity of the detector output signal (some data processors can only handle positive peaks).

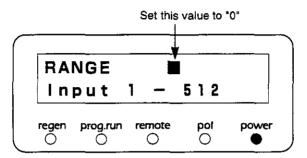
The polarity of channel 1 can be set by pressing the [pol] key (shift) + (9) from the initial screen (Ch. 1 monitor display). The polarity of Ch. 1 toggles from positive to reversed each time the [pol] key is pressed. The pol LED indicates the current Ch. 1 polarity.

The polarity of Channel 2 is set in the same way from the Ch. 2 monitor display screen. The pol LED indicates the Ch. 2 polarity setting only when the Ch. 2 monitor screen is displayed.

### 6.5.5 Zeroing a strip-chart recorder

Before beginning analysis, adjust the zero position of the recorder as follows:

- 1. Press (CE) to display the initial screen.
- 2. Press func several times until [RANGE (AUFS)] (the measurement range setting) is displayed.



3. Press O.

This will short-circuit the detector's output to the recorder.

- 4. Using the recorder's pen position adjusting knob, move the pen to the desired 0 or baseline level..
- 5. Reset the detector's measuring range to a range appropriate for the analysis. (See p. 6-18.)

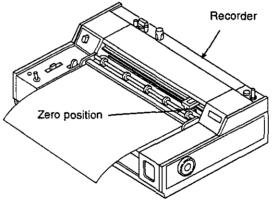


Fig. 6.10

6. Press zero.

The pen will return to a position close to the selected baseline.

7. Move the pen to the exact desired baseline by pressing or , and start the analysis.

Pressing the (zero) key will restore the baseline that was set in this procedure.

#### 6.5.6 **Setting RESPONSE**

This detector uses a digital noise filter to improve the signal-to-noise (S/N) ratio. Noise increases as the filter's response speed is raised, and decreases as it is lowered.

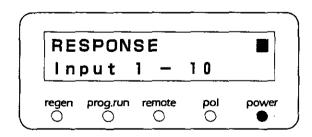
Ten filter response speeds (RESPONSE values) are available for the RESPONSE parameter. RESPONSE values and the corresponding time constants for an analog filter are shown in the table below.

RESPONSE value	Corresponding time constant of analog filter	Minimum peak width at half-height
1	0.05 sec (FAST)	0.2 sec
2	0.1 sec	0.4 sec
3	0.5 sec (STD)	2.2 sec
4	1.0 sec	4.8 sec
5	1.5 sec (SLOW)	7.2 sec
6	2.0 sec	9 sec
7	3.0 sec	13 sec
8	6.0 sec	26 sec
9	8.0 sec	36 sec
10	10.0 sec	45 sec

Expressions in parentheses indicate corresponding response settings of the CDD-6A.

#### To set RESPONSE:

- 1. Press (CE) to display the initial screen.
- Press shift), 6 (rsp). The RESPONSE value is now active, and can be changed. 2.
- 3. Input the RESPONSE value using numeric keys. The setting range is given in the table above.



- 4. Press (Enter).
- 5. Press (E) to return to the initial screen.

Fig. 6.11

NOTE

As RESPONSE increases, data processor responsiveness and peak height decrease. The smaller a peak's width at half-height, the greater the decrease in the peak's height.

It is recommended that RESPONSE be set such that, for a given half-height width, the peak height drops no more than 10%. The graph on the right shows the relationship among response time, peak half-height width and peak height reduction. Use it to determine appropriate RESPONSE values.

Note that RESPONSE has no effect on peak area. Peak area does not change even when a low RESPONSE value broadens the peak.

When controlling the detector from the SCL-10Asp, set the LINK ADRS parameter to the remote connector number on the rear panel of the SCL-10Asp to which the detector is connected. Also ensure that the LOCAL parameter is set to 0, which disables local control. To switch back to local control, set LOCAL to 1.

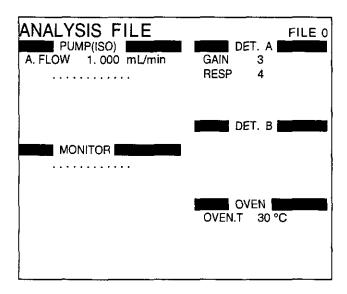
Most of the basic parameters can be set on the SCL-10Asp, including time program setting. However, note that some parameters are not available on the SCL-10Asp and should be set on the detector (these include RANGE, REC MODE, etc.)

This section describes general SCL-10Asp parameter setting. For details on these operations, refer to the SCL-10Asp user's manual. Also see "6.7 Operation of non-suppressor system" and "6.8 Operation of suppressor system."

#### 6.6.1 **Basic parameters**

The analysis file screen displays parameters for the various modules connected to the controller. The detector's parameters are shown in DET.A (DET.B) parameter groups. In this screen, some of the detector parameters are shown for setting and some for display. Parameters which can be selected often display normal default values. Each parameter can be set by moving the cursor to the value, inputting the value, and pressing Enter.

For detailed settings, use the Detector control screen. Move the cursor to any of the detector parameters with the cursor keys, and press the [param] function key (f5). If the [param] function key is not shown, press func until [param] appears in the [f5] position.



Operation

### 6.6.2 Det control screen

In the Det (detector) control screen, all the parameters available on the SCL-10Asp are displayed. The parameter set shown depends on the options installed in the detector.

When the suppressor options are installed, the status of suppressor cartridges are shown in the bottom right of the screen. See "6.8 Operation of the suppressor system" for details.

The table below shows the parameters used in this screen.

ANALYS *** Det. A	S FILE CDD-10Asp	***	FILE 0
RESP	4		
TYPE	312		
MPCNC PUMP (FLOW)	14meq 1 1. 000mL/min		ı
CELL. T (OVEN)	43°C 40°C	CH 1 L R USE RDY	CH 2 L R
INJDLY S. INTVL REGENTM	7min 22min 8. 8min		

### SCL-10Asp parameters for the CDD-10Avp/10Asp

<u> </u>			
7	Detector parameters (Ch. 1)		
GAIN	Gain for Ch. 1		
RESP	Response	Common setting for both channels.	
POL	Polarity for Ch. 1	Set 1 for positive, -1 for negative.	
		Only for non-suppressor system.	
CELL.T	Cell temperature (°C)	Common setting both channels.	
(OVEN)	Oven temperature setting is dis-	Display only; setting should be done in	
	played for reference	the parent screen.	
Detector param	neters (Ch. 2)		
GAIN2	Gain for Ch. 2		
POL2	Polarity for Ch. 2	Set 1 for positive, -1 for negative	
		Only for non-suppressor system.	
Suppressor co	ntrol (Ch. 1)		
TYPE	Cartridge type for Ch. 1 (3 digit).	The first digit shows type of analysis	
		(3: anion, 2: cation). The last two digits	
		show the cartridge capacity in [meq].	
		EX) 312 for anion analysis.	
		206 for cation analysis.	
MPCNC	Mobile phase concentration for		
	Ch. 1 (meq).		
PUMP	Specify the pump to be used.	1, 2 and 3 correspond to Pump A, B	
		and C, respectively.	
(FLOW)	Flow rate setting is displayed.	Display only; setting should be done in	
		the parent screen.	
INJDLY	Time between cartridge switching	Common setting for both channels.	
	and sample injection (min).		
S.INTVL	Cartridge switching interval (min).	Common setting for both channels.	
REGENTM	Regeneration time (min).	Common setting for both channels.	
Suppressor co	ntrol (Ch. 2)		
TYPE2	Cartridge type for Ch. 2 (3 digit).	The first digit shows type of analysis	
		(3: anion, 2: cation). The last two digits	
Ì		show the cartridge capacity in [meq].	
MPCNC2	Mobile phase concentration for		
	Ch. 2 (meq).		
PUMP2	Specify the pump to be used.	1, 2 and 3 correspond to Pump A, B	
•		and C, respectively.	
(FLOW)	Flow rate setting for the pump is	Display only; setting should be done in	
	displayed.	the parent screen.	

NOTE

- REC MODE (recorder mode) and RANGE cannot be set on the SCL-10Asp. When Signal output connector 2 is used for an integrator or recorder, set those parameters on the CDD-10Avp/10Asp.
- Similarly, other parameters not shown in the table should be set from the detector.

### Function keys in the Det control screen

The following are function keys on the Det control screen. Five functions are shown at the bottom of the screen. They can be operated by pressing the corresponding function key ([f1] to [f5]). If a function key is not found, press the function key to show the next set of five function keys.

#### <<SWITCH>>

### (For suppressor systems)



This function manually switches the left and right suppressor cartridges to interrupt the current process and advance to the next analysis. The cartridge in the analysis flow line is switched to the regeneration flow line, while the other cartridge currently in the regeneration flow line is switched to the analysis flow line.

When using this function, ensure that the cartridge that will be switched to the analysis flow line has been sufficiently regenerated. If cartridges are switched during regeneration, gases generated in the regenerated cartridge flows into the detector cell, which may cause unstable baseline.

#### <<A.FULL1>> for Detector A

#### (For suppressor systems)



Press this function key to perform FULL REGENERATION of channel 1 cartridges.

#### <<A.FULL2>> for Detector A

#### (For suppressor systems)



This function key can be used to change the cartridge switching interval during the course of an analysis. Each time this key is pressed, the interval is increased by 5 minutes, up to the maximum of plus 20 minutes. If the key is pressed further, then the interval is reduced by 5 minutes. The remaining time display is updated by this operation.

#### <<EXTEND>>

### (For suppressor systems)



This function key can be used to change the cartridge switching interval during the course of an analysis. Each time this key is pressed, the interval is increased by 5 minutes, up to the maximum of plus 20 minutes. The remaining time display is updated by this operation. When the interval is increased by the maximum of 20 minutes, the remaining cartridge capacity

\*

B.ZERO

A.MARK

B.MARK



### <<SEL/DEL>>

Select parameters to be shown in the Analysis file screen. Move the cursor to a parameter from the screen; repeat the same operation.

will be 40% of the total capacity under the standard analysis conditions.

<<A.ZERO>> for Detector A

<<B.ZERO>> for Detector B

Executes an auto zero for the designated detector.

<<A.MARK>> for Detector A

<<B.MARK>> for Detector B

Makes a vertical mark on the strip-chart recorder.



Returns to the Analysis file screen.

# 6

### 6.7.1 Start of analysis

The following two sections show the standard anion and cation analysis methods. Make all the necessary parameter settings and start operation of the pump and column oven by pressing [act] on the SCL-10Asp.

If a manual injector is used, set a STOP command at the necessary time in the system controller time program. When an autosampler is used, specify sampling conditions and run time settings.

Monitor the detector baseline on the recorder or integrator; usually the baseline is stable about two hours after starting temperature control. When the baseline is stable, press the run key to start analysis.

### 6.7.2 Anion analysis method

The following shows standard conditions for anion analysis.

<Separation>

Column

: Shim-pack IC-A1

Mobile phase

: 2.5 mM phthalic acid and 2.4 mM Tris (hydroxymethyl) aminomethane

(pH4.0)

Flow rate

: 1.5 mL/min

Column temperature

: 40°C

Column

: Shim-pack IC-A3

Mobile phase

: 8 mM p-hydroxybenzoic acid, 3.2 mM Bis-Tris, and 50 mM boric acid

Flow rate

: 1.2 mL/min

Column temperature

: 40°C

<Detector: CDD-10Avp>

Gain

: 2 (0.1µScm<sup>-1</sup>)

Response

: 4(1s)

Polarity

: +

Cell temperature

: 43°C

## 6.7.3 Cation analysis method

The following shows standard conditions for cation analysis.

<Separation>

Column : Shim-pack IC-C1

Mobile phase : 3 mM oxalic acid

Flow rate : 1.5 mL/min

Column temperature : 40°C

Column : Shim-pack IC-C3

Mobile phase : 2.5 mM oxalic acid

Flow rate : 1.2 mL/min

Column temperature : 40°C

<Detection: CDD-10Avp>

Gain : 3 (1µScm<sup>-1</sup>)

Response : 4(1 s)

Polarity : -

Cell temperature : 43°C

### 6.8.1 Starting the analysis

The following two sections show the standard anion and cation analysis methods. Make all the necessary parameter settings and start operation of the pump and column oven by pressing [act] on the SCL-10Asp. At the same time, the suppressor control begins, and the regeneration cycle starts at the set interval.

Set the autosampler and run time parameters. If a manual injector is used, set a STOP command at necessary time in the system controller time program.

Monitor the detector baseline on the recorder or integrator; usually the baseline is stable about one hour after starting temperature control. After the baseline is stable, press the run key to start analysis.

NOTE

The analysis sequence and timing described assume the use of on SIL-10AF autosam-

### Status display for suppressor systems

When the suppressor options are installed, the system status is shown in the bottom right of the screen. The status is shown for the left (L) and right (R) cartridge of each channel (CH 1 and CH2) as follows:

USE The cartridge is in the analysis flow line; it is being used or is ready to use for analysis.

RDY The cartridge is in the regeneration flow line; regeneration is completed and the cartridge is ready for analysis.

RGN The cartridge is being regenerated.

FUL Full regeneration is under way.

OFF Regeneration is suspended by opening the oven door.

--- Suppressor option is not installed.

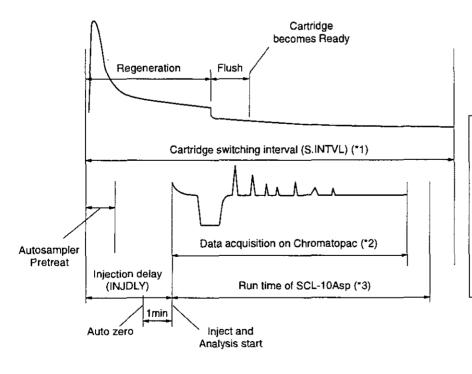
When the [act] key is pressed and an analysis is started, the remaining time is displayed for S.INTVL (cartridge switch interval) and REGEN (regeneration time) /FLUSH.

Also, when full regeneration is being conducted, the status and remaining time is shown.

CH 1 L R USE RDY	CH 2 L R
FULL REGENEI	0. 00

### Analysis period and cartridge switching interval

The following figure shows the relationship of the parameters which determine the timing of cartridge switching for suppressor systems



- (\*1) When there is no analysis, the suppressor cartridges are switched at the end of S. INTVL. During the analysis, the suppressor cartridges are switched at the end of run time of SCL-10Asp. (Cartridge are switched before the end of S. INTVL.)
- (\*2) STOP.TM on Chromatopac
- (\*3) RUN.T or TIME setting of STOP command on SCL-10Asp

Fig. 6.12

#### **Full Regeneration**

The Full Regeneration function can be used to completely regenerate both (left and right) of the suppressor cartridges. When the anion suppressor cartridge is fully regenerated, the resin is in the hydrogen form (H<sup>+</sup>) and a generated cation suppressor cartridge is in the hydroxide form (OH).

In the full regeneration sequence, the regeneration process is carried out three times with the cartridges switched. The duration of each regeneration process is automatically calculated based on flow rate, cartridge capacity and mobile phase concentration parameters, with an upper limit of 30 minutes.

### 6.8.2 Anion analysis method

The following shows standard conditions for anion analysis.

<Separation>

Mobile phase : 12 mM sodium hydrogen carbonate / 0.6 mM sodium carbonate

Flow rate : 1.0 mL/min

Temperature : 30°C

Column : Shim-pack IC-SA2 (250 mmL × 4.0 mm i.d.)

Shim-pack IC-SA2 (G) (10 mmL  $\times$  4.6 mm i.d.)

Injection volume : 50 μL

<Detection: CDD-10Asp>

Gain :  $2 (0.1 \mu \text{Scm}^{-1})$ 

Response : 4(1 s)

Polarity :+

<Suppressor control>

TYPE (TYPE2) : 312

MPCNC (MPCNC2) : 14 meq

INJDLY : 4.5 min

S.INTVL : 21 min

REGENTM : 6.5 min

Data acquisition time : 16 min

### 6.8.3 Cation analysis method

The following shows standard conditions for anion analysis.

<Separation>

Mobile phase

: 7 mM methane sulfonic acid

Flow rate

: 1.0 mL/min

Temperature

: 30°C

Column

: Shim-pack IC-SC1 (150 mmL × 4.6 mm i.d.) with Shim-pack IC-

SC1 (G) (10 mmL  $\times$  4.6 mm i.d.)

Injection volume

 $: 50 \, \mu L$ 

<Detection: CDD-10Asp>

Gain

 $: 2 (0.1 \mu \text{Scm}^{-1})$ 

Response

: 4 (1 s)

Polarity

:+

<Suppressor control>

TYPE (TYPE2)

: 206

MPCNC (MPCNC2)

: 7 meq

INJDLY

: 4.5 min

S.INTVL

: 21 min

REGENTM

: 6.5 min

Data acquisition time

: 16 min

### 6.8.4 Setting the suppressor control parameter (reference)

The SCL-10Asp checks that the control parameters are acceptable for the cartridge type.

A Time Program is used to program certain functions of the unit automatically during an analysis.

Time Programs are retained in memory when the power is turned off.

### 6.9.1 Program commands

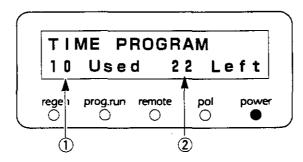
The commands that can be used in Time Programs are listed below.

Function	Description	Value range	Remarks
GAIN1	Gain for channel 1	1-5	
GAIN2	Gain for channel 2		
ZERO	Executes detector autozero	None	
MARK	Marks recorder output	None	
RANG	Sets full scale output range of recorder terminals	1-512	A range of 0 short-cir- cuits the recorder ter- minals, preventing sig- nal output.
RESP	Sets detector time response	1-10	
EVNT	Sets EVENT outputs ON/OFF	0, 1, 2, 12	
POL1 POL2	Sets polarity of detector output for channel 1 and channel 2	0: Positive polarity 1: Reversed polarity	
LOOP	Repeats preceding time program steps the specified number of times	0-255 0: Repeats program endlessly	
STOP	Stops time program	None	

### 6.9.2 Basics of Time Programming

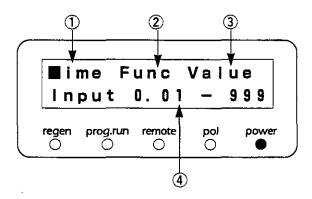
To write or edit a time program, enter the EDIT mode as described below.

- 1. Press (E) to display the initial screen.
- 2. Press shift + 4 (edit).



- ① Number of steps already set
- ② Number of steps remaining

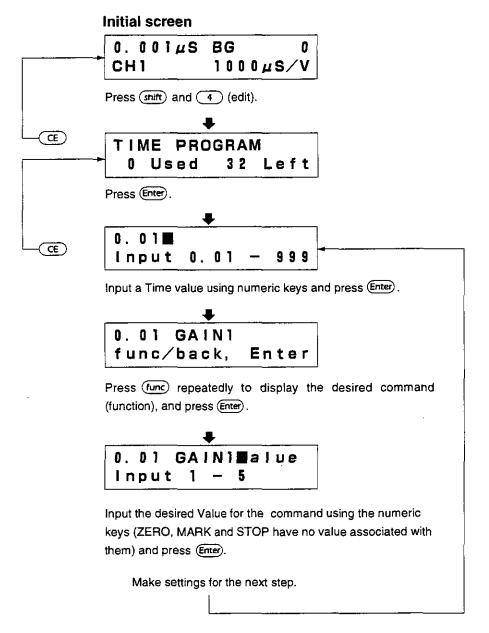
3. Press Enter .The display shows the three parameters that may be set for each program step:



- ① Elapsed time from program start (minutes)
- 2 Command
- 3 Set value
- 4 Possible range for each parameter (0.01-999.99 mins shown here for time)
  - \* The blinking TIME field indicates that a new step can be entered. Input the desired value and press Enter).
- 4. Press CE to return to the initial screen when the time program is complete.

### 6.9.3 Time program writing flow chart

The flow chart shows the steps involved in writing time programs.



To move back one step at a time, press the back key.

Fig. 6.13

### 6.9.4 Creating a typical Time Program

The following example shows the steps involved in creating a Time Program which will execute an auto zero five minutes into the analysis.

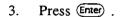
- 1. Press CE to return to the initial screen.
- 0. 0 0 1 μS BG 0
  CH1 1 0 0 0 μS / V

  regen prog.run remote pol power

- 2. Press (shift), (4) (edit).
- TIME PROGRAM

  Used 32 Left

  regen prog.run remote pol power



- 4. Input the time (in minutes) for the first step. Press 5 and Enter .
- ime Func Value
  Input 0.01 999

  regen prog.run remote pol power

ZERO

remote

 $\circ$ 

pol

0

power

power

- Select the ZERO command by pressing func until ZERO is displayed.
- 6. Press Enter .
- No value is necessary for this command. A step has been completed.
- 8. To input the final step, which will stop the program at 10 minutes, first input the TIME and press Enter.
- Press the func key until STOP is displayed.
- 10. Press Enter .

1 0. 0 STOP

prog.run

5.00

regen

Ō

- 11. Press (E) to exit the Time Program mode and return to the initial screen.
- NOTE
- When setting multiple steps, it is not necessary to input them in order. The unit sequences the steps automatically.
- Always end the program with a STOP command, unless the program is to run indefinitely and will be stopped manually.
- When selecting commands, press back to display the previous command.

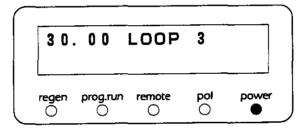
# 6.9.5 Editing an existing Time Program (viewing, deleting and inserting steps)

- 1. Press shift, edit to enter Time Program Editing mode.
- 2. Press Enter repeatedly to scroll through the programmed steps.
- 3. To delete a step: Press (enter) to display it, then press (snift) and (CE). The step is deleted and the step that follows is displayed. If no steps follow, the summary information is displayed.
- 4. To insert a step: With a step displayed and the TIME value blinking, input a new TIME and press Enter. Complete the step using the previous instructions.
- 5. Press CE once to display the summary information. Press CE again to exit Time Program Editing mode and return to the initial screen.

### 6.9.6 Loop Command

The Loop Command allows the steps of a Time Program to be repeated.

This feature could be used for repeated autozero, or to control external devices.



The example shown below will run steps 1 and 2 three times, generating on Event pulse output for six seconds, with each cycle taking 30 minutes. At the end of the third cycle, the Time Program will stop.

	TIME	FUNC	VALUE
1	15.00	EVNT	1
2	15.10	EVNT	0
3	30.00	LOOP	3

NOTE

- If any of the set parameters are changed while the time program is running, the new parameter takes effect.
- Up to 255 loops may be specified as a value for the LOOP command. Setting the LOOP value to 0 causes the loop to repeat endlessly.
- Steps which follow a LOOP command are ignored.

#### **Executing a Time Program** 6.9.7

A Time Program may be started or stopped manually or remotely.

To manually start/stop a Time Program:

- Press (run). The [program] LED illuminates and the program starts. 1.
- Press run again to stop the Time Program. A program may also be stopped by a STOP 2. command included in the program.

To remotely start/stop a Time Program:

- Short the START and COM terminals (e.g. by rotating an injection valve) to start the Time Program. The [program] LED illuminates and the program starts.
- Short the STOP and COM terminals (e.g. by rotating an injection valve) to stop the Time 2. Program. A program may also be stopped by a STOP command included in the program.

Each time the detector receives a remote Time Program START signal, the Time Program will be reset and begin again.

NOTE

In order to remotely control the Time Program, a switch from a suitable device such as a manual injector or autosampler must be connected to the START and COM terminals (for starting) and the STOP and COM terminals (for stopping) or the External Control Connector located on the back of the detector (see Section 7). This switch must provide a contact closure.

In addition to the initial display screen, the display monitors and controls many additional functions. These functions are accessed by pressing the two key repeatedly. As the key is pressed, the active field of the display will step first through the labeled functions, and then access the auxiliary functions listed in the following table. Return to the initial screen at any time by pressing the auxiliary functions are explained in this section.

### 6.10.1 Summary of auxiliary functions

Туре	Command	Function
4	0.001μS BG 0	Displays conductivity and background of Ch. 2
	CH2 1000µS/V	*Appears only when the 2 channel option is installed.
4	SUP1 L: USE R: REG	Displays the status of suppressor channel 1.
ĺ	10.0V 100ohm	*Appears only when the PC-3CD (Suppressor Ch. 1
		PCB) is installed.
4	SUP2 L: USE R: REG	Displays the status of suppressor channel 2.
]	10.0V 100ohm	*Appears only when the PC-4CD (Suppressor Ch. 2
		PCB) is installed.
1	GAIN1	Sets gain of channel 1.
1	GAIN2	Sets gain of channel 2.
}		*Appears only when the 2 channel option is installed.
1	RANGE	Sets signal full scale for signal output connector 2.
1	CELL TEMP	Sets cell temperature.
1	REC MODE	Sets output mode of signal output connector 2.
-4	CELL1_T	Displays actual cell temperature for channel 1.
4	CELL2_T	Displays actual cell temperature for channel 2.
•		*Appears only when the 2 channel option is installed.
1	EVENT	Sets output signal status of external event connector.
1	EXT-S	Sets function of external event connector.
1	MONIT-TIME	Controls display of elapsed time when executing a time
		program.
3	KEY CLOSE	Locks key input.
1	LINK ADRS	Sets address when controlled by SCL-10Asp.
1	LOCAL	Selects local control or control by SCL-10Asp.
1	VLV1	Switches the suppressor valve and cartridges of Ch. 1
1	VLV2	Switches the suppressor valve and cartridges of Ch. 2
1	H/W CONFIG	Enables/Disables suppressor interfaces.

The "Type" column in the above table indicates the key operation required for the function

Type 1: Input a value using numeric keys, and press Enter . The value input is set.

Type 3: Press Enter to execute the function.

Type 4: Function provides display only. No key operation.

### 6.10.2 Auxiliary function descriptions

### <<Ch. 2 monitor display>>

Displays channel 2 conductivity measurement and background. The bottom line shows the signal output full scale in uS ( $\mu$ Scm<sup>-1</sup>) or in uS/V ( $\mu$ Scm<sup>-1</sup>), which can be selected by one of the VP functions.

\* This screen appears only when the PC-2CD (2-Channel Option PCB) is installed.

0.001µS BG 0 CH2 1000µS

### << Suppressor Ch. 1 monitor display>>

Displays the status of suppressor channel 1. The top line shows the status of the left (L) and right (R) cartridges as follows:

USE: Being used for analysis.

REG: Currently being regenerated.

RDY: Regeneration completed and ready for analysis.

The bottom line shows the actual voltage and resistance across the cartridge being regenerated. The voltage becomes negative for anion analysis, and pasitive for cation analysis.

\* This screen appears only when the PC-3CD (Suppressor Ch. 1 PCB) is installed.

SUP1 L:USE R:REG 10.0V 1000hm

### << Suppressor Ch. 2 monitor display>>

Displays the status of suppressor channel 2. The top line shows the status of the left (L) and right (R) cartridges as follows:

USE: Being used for analysis.

REG: Currently being regenerated.

RDY: Regeneration completed and ready for analysis.

The bottom line shows the actual voltage and resistance across the cartridge being regenerated. The voltage becomes negative for anion analysis, and pasitive for cation analysis.

\* This screen appears only when the PC-4CD (Suppressor Ch. 2 PCB) is installed.

SUP2 L:USE R:REG 10.0V 100ohm

#### <<GAIN>>

Sets the gain of channel 1 (GAIN1). Also sets gain of channel 2 when the 2ch option is installed (GAIN2).

When GAIN2 is displayed, entering a value moves the cursor to the other gain parameter. The can also be moved by pressing Enter). Refer to "6.5.2 Setting GAIN" for details.

> GAINI GAIN2 3 Input

#### <<RANGE>>

Sets the signal output full scale for 10 mV recorder (signal output connector 2). The output signal becomes 0 when 0 is input for RANGE. This is useful for adjusting the zero point of the recorder. Refer to "6.5.3 Setting RANGE" for details.

> RANGE 0 — 5 1 2 Input

#### <<CELL TEMP>>

Sets the cell temperature. Normally, set the cell temperature 3°C higher than the oven temperature.

This temperature setting applies to both cells when the 2Ch. option is installed.

35 CELL TEMP 25 5 5 0: of f

NOTE

Stability (degree of fluctuation) in the detector cell temperature is essential for high sen! sitivity and precision in the measurement of conductivity. For this reason, the cell temperature display of the unit is made so that temperature fluctuation as small as 1/1000°C can be monitored. The circuit is optimized for this purpose and temperature shown on the display may have some deviation from the set temperature, which is not abnormal.

#### << REC MODE>>

Sets output mode of Signal output connector 2. Input a value and press Enter.

REC MODE 0 Input

Set value	Output mode
0	Output the Ch. 1 conductivity signal to Signal output connector 2
1	When the 2ch Option is installed, outputs the Ch. 2 conductivity signal to Signal output connector 2.

### <<CELL1\_T, CELL2\_T>>

The actual cell temperature of channel 1 is displayed (CELL\_T). When the 2Ch option is installed, its actual cell temperature is also displayed (CELL2\_T).

### <<EVENT>>

Relay output signals of the EVENT terminals on the rear panel can be set with this parameter. Input a value and press Enter.

ON = contact closure OFF = contact open

ΕV	EN	JT			0
0,	1,	2	ОГ	12	

Set value	EVENT1	EVENT2
0	OFF	OFF
1	ON	OFF
2	OFF	ON
12	ON	ОИ

### <<EXT-\$>>

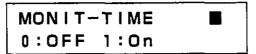
Sets specialized control of EVENT1 and EVENT2 relays. Input a value, then press Enter .

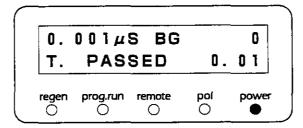
Set value	Control mode
0	EVENT outputs are controlled by the EVENT value.
1	EVENT1 operates as a time program start signal.
	When the detector time program starts, EVENT1
	closes for about 0.6s. Useful for starting a data
	processor or other eternal deveice
2	EVENT2 operates as an error output signal. When
	the detector generates an error, EVENT2 closes until
	error is reset. EVENT2 opens when error is reset by
	pressing [CE] on the CDD-10Avp/10Asp or on the
	SCL-10Asp.
3	EVENT1 operates as a time program start signal and
	EVENT2 operates as an error output signal.

### <<MONIT-TIME>>

Displays the elapsed time of the event time program.

Input a value, then press Enter .





Set value	Function
0	Cancels display of elapsed time
1	Enables display of elapsed time

When this function is enabled, the display appears as shown.

#### <<KEY CLOSE>>

Disables the keypad. To disable, press Enter . To reenable the keypad, press and hold E, and press shift .

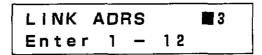
> KEY CLOSE to Close Enter

#### <<LINK ADRS>>

Sets the SCL-10Asp System Controller address (fiber optic channel number) to which the CDD-10Avp/10Asp is connected.

Input a value and then press Enter .

See the SCL-10Asp manual for guidance in assigning channel numbers.



#### <<LQCAL>>

Selects local control or control by SCL-10Asp. Input a value, then press Enter.



### <<VLV1>>

Switches the suppressor valve and cartridges of Ch. 1. This function is similar to [A.SW1] on the SCL-10Asp, but VLV1 can also be used when the suppressor system is not running (when the [act] key of the SCL-10Asp has not been pressed).

Set value	Output mode
0	The left suppressor is connected to the regeneration flow
	line, and the right suppressor to the analysis flow line.
1	The right suppressor is connected to the regeneration
	flow line, and the left suppressor to the analysis flow
	line.

#### <<VLV2>>

Switches the suppressor valve and cartridges of Ch 2. This function is similar to [A.SW2] of the SCL-10Asp, but VLV2 can be used also when the suppressor system is not running (when the [act] key of the SCL-10Asp has not been pressed).

Set value	Output mode
0	The left suppressor is connected to the regeneration flow
	line, and the right suppressor to the analysis flow line.
1	The right suppressor is connected to the regeneration
	flow line, and the left suppressor to the analysis flow
	line.

### <<H/W CONFIG>>

Enables/disables suppressor interfaces that are installed in the CDD-10Avp/10Asp. If suppressor options are installed for two channels, this setting can be made to each of the channels independently.

When the interface has been installed, the CDD-10Asp, or the CDD-10Avp with suppressor options, recognizes the interface and works only in the suppressor mode.

Set this function when non-suppressor operation is desired by connecting CDD-10Avp type cells, without removing suppressor interfaces.

Set value	Operation
0	All the installed suppressor interfaces
	operate.
1	Disables Ch. 1 suppressor interface, if
	installed, so that Ch. 1 can operate in
	non-suppressor mode.
2	Disables Ch. 2 suppressor interface, if
ł	installed, so that Ch. 2 can operate in
	non-suppressor mode.
3	Disables all the installed suppressor
	interfaces. Both channels can operate in
j	non-suppressor mode.

## 6.11.1 Summary of VP functions

The unit's VP functions are listed in the table below. They are accessed by repeatedly pressing the func key. As the key is pressed, the display will sequentially access the functions in order.

\*Items are shown only when corresponding hardware is installed.

### **Product Information Group**

Command	Function
SERIAL NUMBER	Displays unit's serial number
S/W ID: V*.**	Displays unit name and ROM version

### **Maintenance Information Group**

Command	Function
TOTAL OP TIME	Displays unit's total cumulative operating time
VALVE1 ROTATION	Displays Valve 1 rotations and replacement alert level*
VALVE2 ROTATION	Displays Valve 2 rotations and replacement alert level*
SUP1 RES	Displays resistance of cartridges for Ch 1.*
SUP2 RES	Displays resistance of cartridges for Ch 2.*
PART REPLACEMENT	For inputting parts replacement records
MAINTENANCE LOG	Displays maintenance log
OPERATION LOG	Displays operation log
ERROR LOG	Displays error log

### **Validation Support Group**

Command	Function	
DATE YY-MM-DD	Displays/sets the date	-
TIME HH:MM:SS	Displays/sets the time	
MEMORY CHECK	Runs auto checks on memory	
CH1 SPAN CHECK	Span check for Ch. 1.	
CH2 SPAN CHECK	Span check for Ch. 2.	

## **Calibration support group**

Operation	Function		
Input PASSWORD	For input of password.		
ZERO CALIB CH1	Zero calibration for Ch. 1.		
SPAN CALIB CH1	Span calibration for Ch. 1.		
ZERO CALIB CH2	Zero calibration for Ch. 2.		
SPAN CALIB CH2	Span calibration for Ch. 2 *		
SPAN CHECK	Setting criterion for Ch. 1 and Ch. 2 span check.		
VLV ROT	Setting alert level for valve rotation. *		
SUP1 RES	Setting alert level for cartridge resistance of Ch. 1. *		
SUP2 RES	Setting alert level for cartridge resistance of Ch. 2.		
RNG DISP MODE	Setting the conductivity full scale shown in the initial screen.		
INITIALIZE PARAM	For initializing parameters.		
CHANGE PASSWORD	For changing password.		
H/W CONFIG	Enables/disables installed suppressor options.		
S1	Span adjustment value of Ch. 1 conductivity signal.		
B1	Span adjustment value of Ch. 1 background conductivity.		
S2	Span adjustment value of Ch. 2 conductivity signal.		
B2	Span adjustment value of Ch. 2 background conductivity. *		
Z1-1	Zero adjustment value of Ch. 1 conductivity signal. (gain = 1)		
Z1-2	Zero adjustment value of Ch. 1 conductivity signal. (gain = 2)		
Z1-3	Zero adjustment value of Ch. 1 conductivity signal. (gain = 3)		
Z1-4	Zero adjustment value of Ch. 1 conductivity signal. (gain = 4)		
Z1-5	Zero adjustment value of Ch. 1 conductivity signal. (gain = 5)		
Z2-1	Zero adjustment value of Ch. 2 conductivity signal. (gain = 1) *		
Z2-2	Zero adjustment value of Ch. 2 conductivity signal. (gain = 2) *		
Z2-3	Zero adjustment value of Ch. 2 conductivity signal. (gain = 3) *		
Z2-4	Zero adjustment value of Ch. 2 conductivity signal. (gain = 4) *		
Z2-5	Zero adjustment value of Ch. 2 conductivity signal. (gain = 5) *		

<sup>\*</sup> If the password was not entered correctly, the items from ZERO CALIB CH1 onwards in the Calibration Support Group cannot be accessed (pressing func will not scroll to them).

## 6.11.2 Using VP functions

- 1. Press (CE) to return to the initial screen.
- 2. Use the VP key to select the desired Group.
- Press the (func) key until the desired function is displayed. (To scroll back by one function, press (back).)
- Follow the relevant instructions for the function selected. 4.
- To change to a different VP Function Group, press VP to return to the group title screen, 5. then press (VP) to select the desired group. Then use (func) (and (back) if needed) to display the desired function.
- To return to the initial screen, press (CE). 6.

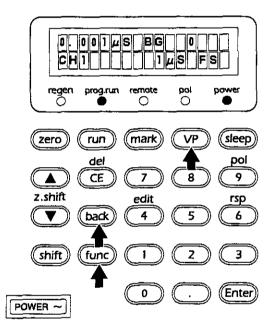


Fig. 6.14

## 6.11.3 Setting VP series functions

### **Product Information Group**

Functions in this Group provide information about the unit.

PRODUCT INFO **VP** Press func or

### <<SERIAL NUMBER>>

Displays the unit serial number.

SERIAL NUMBER C21183400000

# eration

### <<S/W ID>>

Displays the unit name and ROM version.

S/W ID: V\*. \*\* CDD-10Avp

### **Maintenance Information Group**

Functions in this Group provide maintenance-related information.

MAINTENANCE Press func or VP

### <<TOTAL OP TIME>>

Displays the unit's total cumulative operating time.

The example on the right shows an operating time of 123 hours.

TOTAL OP TIME 123

### << VALVE 1 ROTATION>>

Display the total rotations of Ch 1. valve and replacement alert level.

When the valve rotor and stator are replaced, access this item and press o, Enter to reset the rotations to zero.

The example below shows 12 valve rotations and a replacement alert level of 40000 times.

VALVE1 ROTATION

12 / 40000

### <<VALVE 2 ROTATION>>

Displays the total rotations of Ch 2. valve and replacement alert level.

When the valve rotor and stator are replaced, access this item and press o, Enter to reset the rotations to zero.

The example below shows 3 valve rotations and a replacement alert level of 40000 times.

VALVE2 ROTATION

3 / 40000

### <<SUP1 RES>>

Displays resistance of suppressor cartridges for Ch. 1 and replacement alert level. This resistance is measured at the end of the regeneration process.

Usually, resistance during the last regeneration is shown.

SUP1 RES/ 250 ohm L: 0 R: 0

### <<SUP2 RES>>

Displays resistance of suppressor cartridges for Ch. 2 and replacement alert level. This resistance is measured at the end of the regeneration process.

Usually, resistance during the last regeneration is shown.

SUP2 RES/ 250 ohm L: 0 R: 0

### << PART REPLACEMENT>>

For inputting the part No. when a part is replaced. The number is stored in the maintenance log.

• This input is generally performed by a service representative.

PART REPLACEMENT
P/N: = - -

### <<MAINTENANCE LOG>>

Displays the maintenance log, which contains records (part Nos. and replacement dates) of the most recent parts replacements (up to 10).

Pressing Enter repeatedly scrolls through entries Log 1 to Log 10 in sequence.

In the example on the right, the Log 1 entry indicates that part No. 012-34567-89 was replaced on May 12, 2000.

If fewer than 10 part replacements have been logged, the screen on the right appears after the last entry. Press CE to return to the function title screen.

### <<OPERATION LOG>>

Displays the operation log, which contains the most recent password settings and parameter initializations (up to 10).

Pressing Enter repeatedly scrolls through entries Log 1 to Log 10 in sequence.

In the example on the right, the Log 1 entry indicates that a password setting was made on May 12, 2000.

If fewer than 10 password settings have been logged, the screen on the right appears after the last entry. Press CE to return to the function title screen.

MAINTENANCE LOG Enter to Display



00 - 05 - 12LOG 1 P/N:012-34567-89

No more Logs

OPERATION LOG to Display Enter



00 - 05 - 12LOG 1 **PASSWORD** CHANGE

Nο more Logs

### <<ERROR LOG>>

Displays the error log, which contains the most recent errors (up to 10) and their occurrence dates.

Pressing Enter repeatedly scrolls through entries Log 1 to Log 10 in sequence.

In the example on the right, the Log I entry indicates that a leak was detected on May 12, 2000.

If fewer than 10 errors have been logged, the screen on the right appears after the last entry. Press CE to return to the function title screen.

### ERROR LQG to Display Enter

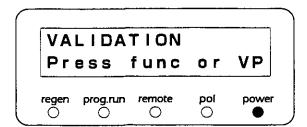


00 - 05 - 12LOG ERR VALVE1 HOME

No more Logs

### Validation Support Group

This Group is for verifying that the unit is operating correctly.

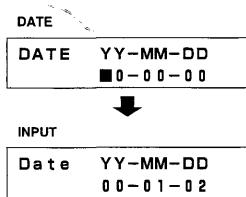


### <<DATE>>

This displays the current date, or allows it to be set. (Only for local control; for control by a system controller, the date is transmitted during link-up, and cannot be changed.)

### Example: Setting January 2, 2000.

- Use numeric keys to first set the year, then the month, then the day. For the year, input the year of the decade only. For each item, be sure to input 2 digits, (i.e. input a zero in the tenths column if necessary).
- 2. When the setting is complete, press Enter .



### <<TIME>>

This displays the current time, or allows it to be set. (Only for local control; for control by a system controller, the time is transmitted during link-up, and cannot be changed.)

### Example: Setting 5:30:55 p.m.

- 1. Use numeric keys to first set the hours, then the minutes, then the seconds. The display uses a 24-hour clock. For each item, be sure to input 2 digits, (i.e. input a zero in the tenths column if necessary).
- 2. When the setting is complete, press Enter .

### TIME

TIME HH: MM: SS



### **INPUT**

TIME HH:MM:SS 17:30:55

### <<MEMORY>>

- Press Enter . The memory check is executed.
   The result of the check is displayed when it is completed.
- 2. Press CE to erase the resulting display.

MEMORY CHECK ■ Enter to Check



MEMORY CHECK RAM OK / ROM OK

## <<CH1 SPAN CHECK>>

### <<CH2 SPAN CHECK>>

Before executing this function, set the cell temperature to 25°C and wait until the temperature has stabilized.

> SPAN CHECK CHI to Start Enter

- Prepare a 10 mM KCl (Potassium chloride) conductivity standard solution, which has a conductivity of 1413µScm<sup>-1</sup>.
- The cell should be filled with the KCl solution. This can be done by filling and sealing the cell using or a syringe.

NOTE

When sealing the cell with the liquid within, do not allow the liquid to come out of the cell. Also, ensure that no bubbles are present in the cell.

2. Press Enter.

Fill the cell with the KCI solution and wait until the baseline has stabilized.

Fill 10mM KCI And Enter

Once the baseline is stable, press Enter to start measurement. The measurement takes 3. approximately 6 seconds.

> Measuring... Criterion: xx. x%

The result is displayed.

Enter to store the result in memory. If CE is pressed, the result is not memorized.

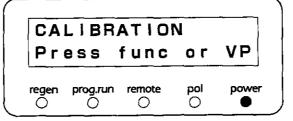
1.0 CD % GOOD Enter to Memory

### **Calibration Support Group**

This group is for performing calibration of the unit.

NOTE

The unit is adjusted before leaving the factory. Normally, these values should not be changed.



### <<input PASSWORD>>

The password should be set by the system manager.

Passwords must consist of five digits.

Input PASSWORD
\*\*\*\*\*

The password set at delivery is [00000].

If the password is incorrect, subsequent functions cannot be accessed.

### <<ZERO CALIB CH1>>

Zero (offset) calibration is done for Ch. 1. This should be executed when the instrument has warmed up for more than 10 minutes after turning it on.

1. Turn the power off and disconnect the CD SIGNAL cable from the rear panel terminal.

Turn the power on and wait for 10 minutes for warming up.

ZERO CALIB CH1 Enter to Calib

2. Press Enter. Zero calibration is made for each of the gain settings automatically. It takes approximately 30 seconds to complete calibration.

ADJUSTING GO. 01

### ZERO-CAL CHI

NOTE

When the calibration is complete, parameters have been automatically set as follows.

Press (Enter) to exit this menu. Reconnect the CD SIGNAL cable to the back panel.

 $GAIN1 = 5 (100 \mu Scm^{-1})$ 

RESP = 4 (1s)

### <<SPAN CALIB CH1>>

Conductivity span is calibrated for Ch. 1. This should be executed when the instrument has warmed up for more than 10 minutes after turning it on, and is usually performed after execut-~ (20) C ing ZERO CALIB.

Before span calibration

- Set the cell temperature to 25°C (set the oven temperature to 25°C) and wait until the temperature has stabilized.
- Prepare a 10 mM KCl (Potassium chloride) conductivity standard solution, which has a conductivity of 1413µScm<sup>-1</sup>.
- Fill the cell with the KCl solution. This can be done by filling and sealing the cell using a syringe. 1 min wait

SPAN CALIB CHI to Calib Enter

NOTE

When sealing the cell with the liquid within, do not allow the liquid to come out of the cell. Also, ensure that no bubbles are present in the cell.

2. Press Enter to start calibration.

ADJUSTING SPAN

3. Calibration is completed in approximately 20 seconds. Press Enter to exit this menu.

SPAN-CAL CHI OK

NOTE

When the calibration is complete, parameters have been automatically set as follows:

 $GAIN1 = 5 (100 \mu Scm^{-1})$ 

RESP = 4 (1s)

### << ZERO CALIB CH2>>

Zero (offset) calibration is done for Ch. 2. Follow the procedure described in ZERO CALIB CH1.

ZERO CALIB CH2 Enter to Calib

### <<SPAN CALIB CH2>>

Conductivity span is calibrated for Ch. 2. Follow the procedure described in ZERO CALIB CH1.

SPAN CALIB CH2 Enter to Calib

### <<SPAN CHECK>>

SPAN CHECK 10
Input Criterion

The criterion (%) for span check is set here. The setting is commonly used for Ch. 1 and Ch. 2.

The default setting is 10 (%)

### <<VLV ROT>>

The alert level for the valve rotations is set here. Each turn of the valve from one position to the other is counted as 1 rotation. The value set here applies to both of the suppressor channels in the dual channel configuration.

The default setting is 40,000.

VLV ROT 40000 Input Alert Lv

<<SUP1 RES>>

<<SUP2 RES>>

The alert levels for suppressor cartridge resistance for Ch. 1 and Ch. 2, respectively, can be set in ohm. These values are compared with resistances at the end of the regeneration process.

The default setting is 250.

SUP1 RES/ 250ohm Input Alert Lv

SUP2 RES/ 250ohm input Alert Lv

### << RNG DISP MODE>>

Specify whether the conductivity full scale displayed on the initial screen is the recorder full scale (uSFS) or integrator full scale (uS/V).

Press o or 1, then Enter.

The default setting is ①.

- For display of uSFS full scale, set .
- For display of uS/V full scale, set .

RNG DISP MODE 0:REC 1:ITG

### <<INITIALIZE PARAM>>

Initializes GAIN1 and other parameters, and deletes the Time Program. (This operation is recorded in the operation log.)

Press Enter to execute.

INITIALIZE PARAM Enter to Init

### << CHANGE PASSWORD>>

- Press Enter .
- 2. Use numeric keys to enter the new password (the password must consist of five digits), and press Enter .

- 3. Repeat step 2.
- 4. It the new password is valid, [CHANGED] appears. (This operation is recorded in the operation log.)

CHANGE PASSWORD Enter to Change

NOTE

Do not forget or share the password.

These are calibration parameters for each channel, which are automatically set when span or zero calibration is executed (with SPAN CALIB CH1, etc.). Usually these parameters are used only for reference.

However, if the cell unit is temporarily replaced with another cell and then reinstalled, the cell calibration parameters are required to maintain calibrated conditions.

S1 and B1 (S2 and B2 for Ch. 2), which must be previously recorded, are used to restore the calibration parameters. Zero adjustment values (Z1-5) are not required when reinstalling the cell.

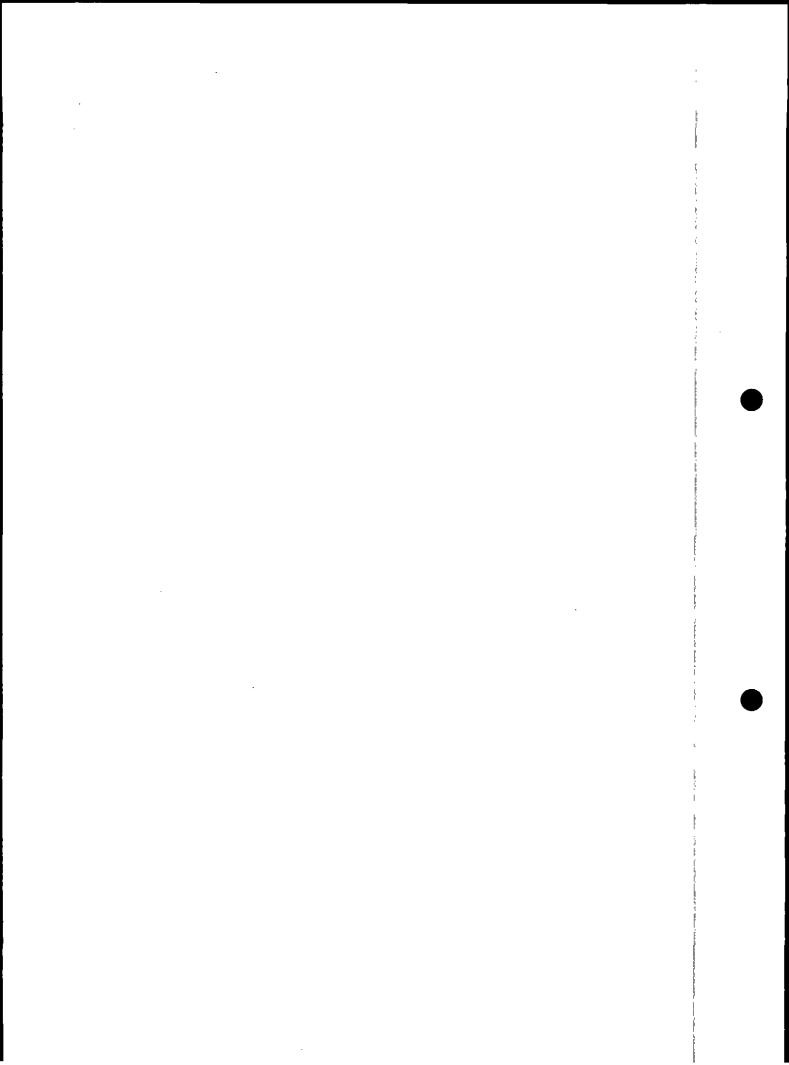
Note that the span values from another instrument should not be used.

## 6.12.1 Cleaning the exterior

- If the covers, front panel or other exterior surfaces of the unit become dirty, wipe them clean with a soft dry cloth or paper towel.
- If surfaces become very dirty, follow the procedure below to clean them.
- 1. Soak a cloth in diluted neutral detergent, wring it out very thoroughly, and use it to wipe away the dirt.
- 2. Soak another cloth in water, wring it out thoroughly, and use it to wipe away all traces of detergent. Then wipe away any remaining moisture with a dry cloth.

## **ACAUTION**

Be sure to wipe surfaces dry after cleaning, and never use alcohol of thinner solvents for cleaning. Otherwise, rust or discoloration could result.



## **Chapter 7 Control by External Devices**



**CONTENTS** 



## **External Control Connections**

The External Control Connector is connected to the event output device or other external device with the event cable supplied.

## **A** CAUTION

- Before connecting the cable, turn off the power to the unit and unplug it.
- Use only the cable specified.
- Follow these instructions.

Failure to heed the above could result in fire, electric shock or malfunction.

## 7.1.1 Wiring Information

Event cable (9-wire)

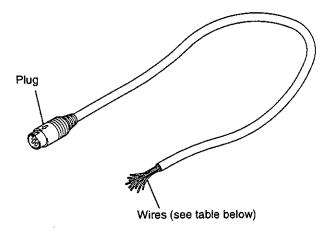


Fig. 7.1

Pin No.	Wire color	Signal	Function	Notes
1	Brown	EVENT1	Event 1/2 output terminals. Internal	Contact rating:
2	Red	(output)	relay contacts which are turned ON/	30VDC/0.1A
3	Orange	EVENT2	OFF according to a time program or	
4	Yellow	(output)	the EVENT parameter value.	
5	Green	NC	Not used	
6	Blue			
7	Violet	PRG.START	START input terminal. START	START/STOP sig-
ŀ		(input)	signal starts the unit's time pro-	nals are imple-
			gram. If signal is received while	mented by shorting
			program is running, program is	the respective
			restarted from time "0"	input terminal with
8	Gray	PRG.START	STOP input terminal. STOP signal	the COMMON ter-
		(input)	stops the unit's time program.	minal. Duration of
9	Black	COMMON	COMMON terminal for input sig-	shorting, tc, should
			nals.	be such that:
1				0.5 sec <tc<10 sec.<="" td=""></tc<10>

## 7.1.2 Connecting the event cable

1. Insert the event cable plug into the external control connector.

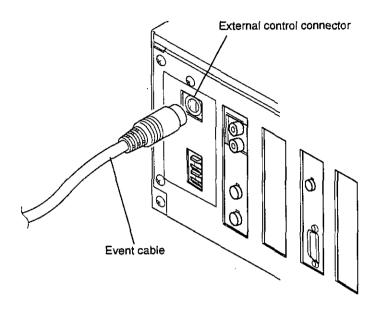


Fig. 7.2

2. Referring to the Signal column on the previous page, connect the external device leads to the appropriate event cable wires using the connector supplied.

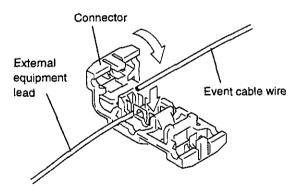


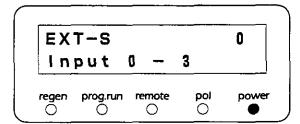
Fig. 7.3

NOTE

If the EVENT1 and/or EVENT2 signal is used, set the EVENT and EXT-S parameters. (See pages 6-43.)

EVENT 1
0, 1, 2 or 12

regen prog.run remote pol power



## **Chapter 8 Hardware Validation**

## CONTENTS

8.1	Hardware Validation Overview	8 -	- 2
8.2	Hardware Validation Implementation	8 -	- 4
8.3	Validation Precautions	8 -	- 6
8.4	Validation Equipment	8 -	- 7
8.5	Component Validation Procedure	8 -	- 8
8.6	System Validation	8 -	- 14

# 8.1

### **Hardware Validation Overview**

## 8.1.1 Types of validation

High perfomance liquid chromatography (HPLC) systems are used to perform demanding analyses including, in some cases, Good Laboratory Practice (GLP). The following three major types of validation are conducted for HPLC systems.

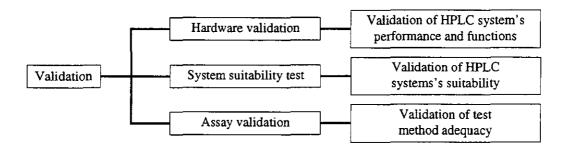


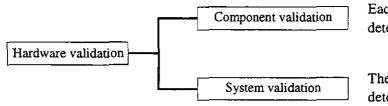
Fig. 8.1

Hardware validation consists of examining the HPLC system to verify that it is operating normally. This validation is performed when the HPLC system is installed, and as part of periodic inspections.

This section focuses on hardware validation.

## 8.1.2 Types of hardware validation

An LC-10Avp module is part of a system of LC components. Two types of hardware validation are performed:



Each system component is checked to determine whether it functions normally.

The system as a whole is checked to determine whether it functions normally.

Fig. 8.2

- At installation, total hardware validation is performed component validation first, followed by system validation.
- Thereafter, system validation is generally performed first, and component validation is only
  implemented if system validation indicates some abnormality. If the system is functioning
  normally, it can be assumed that its components are functioning components. If problems
  exist, one or more of the components may not be functioning properly. Once identified,
  such components must be repaired.

- For a Shimadzu LC-10Avp system, the following measures are implemented to conform to GLP requirements:
  - 1. A certificate of compliance is supplied with each system component.
  - 2. To enable the HPLC system user to perform validation, the validation procedure and guidelines for setting validation criteria are provided on pages 8-4 to 8-21 of this Instruction Manual.

Inspection and validation of the HPLC system are performed within the time frames listed below.

### 8.2.1 At installation

Hardware validation is performed.

Component validation is performed first, followed by system validation. If the HPLC system satisfies all the hardware validation criteria, it is considered to be functioning normally.

### Component validation

This verifes that the individual system components are functioning normally.

### System validation

This checks the performance of the LC system as a whole.

### 8.2.2 Daily inspection

For most LC systems, periodic system validation suffices, and there is no need for daily inspection.

For GLP systems, however, assay validation (validation of analytical methods) and system suitability tests are required in addition to hardware validation, in order to ensure high levels of analysis data reliability. The suitability tests must be run daily.

### **Assay validation**

This validates the analytical methods. It evaluates the following aspects of chromatography: precision, accuracy, detection limits, quantitation limits, specificity, quantitation range, linearity, and ruggedness.

### System suitability test

- After assay validation, the LC system must be validated daily and the results recorded, to
  ensure that the system functions as initially intended.
  - Before each analysis, a standard is run to verify that there are no abnormalities in the LC system.
- If the system suitability test indicates an abnormality, then system validation must be performed to determine whether the problem lies with the LC system or is due to some problem in the analytical method.
  - If system validation finds an abnormality in the LC system, component validation must be performed to identify the malfunctioning component(s), and appropriate corrective action taken.

### 8.2.3 Periodic validation

Hardware validation must be performed every 6-12 months.

System validation should be performed first; component validation is not necessary unless the system validation indicates an abnormality.

### System validation

This checks the performance of the LC system as a whole. Records of this validation must be kept. Other remarks are the same as in 8.2.1 above.

### Component validation

This verifies that the individual system components are functioning normally. Consumable items can be replaced during component validation. Periodic component validation usually calls for maintenance.

NOTE

Maintenance information and results of hardware validation must be recorded and kept for future reference.



## 8.3

## **Validation Precautions**

### 8.3.1 Environment

Abrupt changes in ambient temperature, or heat or air conditioning directed on the instrument could affect data reproducibility. Therefore, the equipment should be installed in a room with minimal  $(< 2^{\circ}C)$  temperature variation and no air currents.

### 8.3.2 Installation site

The installation site is very important for ensuring correct validation. The site should satisfy the following conditions:

## **MARNING**

· Provide ample ventilation, with no fire sources in vicinity

When flammable or toxic solvents are used as the mobile phase, the room must be properly ventilated. When flammable solvents are used, open flame or other fire sources must be strictly prohibited.

## **ACAUTION**

· Avoid dust or corrosive gas

Avoid installing the instrument in places subject to excessive dust or corrosive gas. Otherwise, service life and performance levels may by affected.

Keep away from strong magnetic fields

Do not install the instrument near equipment that generates strong magnetic fields. If the power supply line is subject to high electrical noise, use a commercially-available power surge protector.

• Provide adequate installation surface and space

This instrument is designed to be used on a lab table or similar working surface. The surface that it is installed on must be firm and level, with a depth of at least 60 cm. If components are installed side by side, there should be a clearance of at least 20 mm between them.

Regulate room temperature and humidity

The room temperature should be between 4 and 35°C, with minimal temperature variations throughout the course of a day. Humidity should be kept within 45-85%.

• Position instrument properly in the room

Install the instrument where sunlight, heat or air conditioners are not directed on it, and away from vibration.

## 8.4.1 Component validation equipment

item	Part No.	Remarks	
10 mM-KCl (potassium chloride)	228-41216	Conductivity standard solution of	
aq. solution		1413µScm <sup>-1</sup>	
Syringe	046-00001		
Syringe adapter	228-15672-91		
PEEK tubing Assy	460-08174-91	For drift/noise check	
Data processor	-	Chromatopac or CLASS Software series	
Measuring ruler or calipers	-		

### For non-suppressor system

Item	Part No.	Remarks
2.5 mM phthalic acid and 2.4 mM		For drift/noise check
tris (hydroxymethyl)	-	
aminomethane		

### For suppressor system

Item	Part No.	Remarks
14 mM sodium hydrogen carbon-	-	For drift/noise check
ate		

## 8.4.2 System validation equipment

For non-suppressor system

ltem	Part No.	Remarks
Shim-pack IC-A3	228-31076-91	Anion analysis column
8.0 mM p-Hydroxybenzoic acid		Anion analysis mobile phase
and 3.2 mM Bis-Tris and 50 mM	_	
boric acid		
Anion standard sample	-	F <sup>-</sup> , Cl <sup>-</sup> lppm each

### For suppressor system

Item	Part No.	Remarks
Shim-pack IC-SA2	228-38983-91	Anion analysis column
12 mM sodium hydrogen carbon-	-	Anion análysis mobile phase
ate and 0.6 mM sodium carbonate		
Anion standard sample		F <sup>-</sup> , Cl <sup>-</sup> 1ppm each

### 8.5.1 Conductivity span check

### What the check does:

For this check, the accuracy of the instrument's conductivity span is verified.

This check uses the SPAN CHECK function of the detector, which measures and records the deviation of the actual conductivity from the reference.

While the check procedure shown here is for channel 1, channel 2 can be checked in the same way.

### Items required for the check

10mM-KCl (potassis	m chloride)	aq.	Conductivity standard solution of 1413µScm <sup>-1</sup>
solution (P/N 228-41)	16)		(25°C)

### **Detector preparation**

Set the cell temperature to 25°C and the oven temperature to 22°C. Connect a ~0.5mm i.d. tube to the inlet of the cell and route this tube to the outside of the oven. Connect a syringe to the other end of the tube. Wait until the temperature has stabilized.

### Check procedure

Press (VP) until [VALIDATION] is shown on the display.

VALIDATION Press func or **VP** 

Press (func) until [CH1 SPAN CHECK] is displayed.

CHI SPAN CHECK to Start Enter

Press Enter and fill the cell with the KCl solution with a syringe. Be careful not to allow bubbles in the cell. Also ensure that the solution remains stationary in the cell.

> 10mM KCI Fill And Enter

- 4. Ensure the baseline is stabilized. The baseline can be best monitored at Gain = 4 and ATTEN = 8 with a Chromatopac, or at Gain = 4 and range = 200 with a 10mV strip chart recorder.
- Press Enter to start the measurement. The measurement takes approximately 6 seconds. 5.

Measuring... Criterion: 10.0%

The result is displayed. Press Enter to store the result in memory. Otherwise press CE 6. to disregard the result.

> 1.0% GOOD CD + Enter to memory

Acceptance criterion: [CD xx.x% GOOD] must appear on the screen.

### Completing the conductivity accuracy check

This test automatically sets the gain to 4 (10µScm<sup>-1</sup>) and response to 4 (1s). Return these parameters to the original values after the test.

NOTE

When the CTO-10ACvp is used and the ambient temperature is higher than 32°C, the over temperature cannot be set at 22°C. In this case, take the alternative way shown below for span check.

- 1. Set the over temperature and cell temperature appropriately for the ambient temperature. EX) Oven temperature: 40°C and cell temperature: 43°C
- 2. Set GAIN and other parameters according to the standard span check method.
- 3. Perform autozero after filling and sealing the detector cell with water.
- Fill and seal the detector cell with the 10mM KCl solution. Read the conductivity display when it is stabilized.
- 5. Calculate conductivity K(T) [ $\mu$ Scm<sup>-1</sup>] for the cell temperature of T [°C] using the following equation. This value is used as the criterion for the span check.

$$K(T) = 1413 \times (1+0.02 \times (T-25))$$

6. Calculate the difference between the criterion and the actual conductivity.

**Acceptance Criterion**: The deviation from the criterion must be  $\pm 15\%$  or less.

### 8.5.2 Drift/noise check

### What the check does:

It verifies that drift and noise do not exceed the specified tolerance levels.

### Items required for the check

For non-suppressor system:

2.5 mM phthalic acid and 2.4 mM tris (hydroxymethyl) amino methane

For suppressor system:

14 mM sodium hydrogen carbonate

PEEK tubing (0.75 mm ID  $\times$  2m + 0.13 mm ID  $\times$  2 m)

Data processor

Measuring ruler or calipers

### Preparation

- 1. Connect the detector to the data processor. Be sure to use the signal cables supplied with the detector.
- 2. For a non-suppressor system, connect the outlet of the pump and the inlet tubing of the cell with the PEEK tubing. For a suppressor system, connect the pump outlet and port 10 of the 10-port valve.

### Check procedure (For non-suppressor system)

- 1. Use a mobile phase of phthalic acid and tris aq. solution. Flush the flow lines of the pump with the mobile phase using the PURGE function.
- 2. Close the drain valve and start the pump to deliver the mobile phase at 1.5 mL/min.
- 3. Set the oven temperature to 40°C and the cell temperature to 43°C. Start temperature control of the oven.

4. Set GAIN to 2 (0.1μScm<sup>-1</sup>).

5. Set Response to 4 (1s).

6. Set RANGE to 100 if a strip-chart recorder is used. Recording will be made at 1μScm<sup>-1</sup> full scale. Set a similar recording range if a data processor is used. For the Chromatopac, ATTEN = 7 will give 1.28μScm<sup>-1</sup> full scale.

- 7. Monitoring the baseline plot, wait until the baseline has stabilized.
- 8. Set RANGE to 4 for 400nScm<sup>-1</sup> full scale plotting if a strip-chart recorder is used, or set a similar recording range for the data processor (for the Chromatopac, set ATTEN to 2).

9. Plot the baseline for fifteen minutes. Divide the plot data by 30s to get 30 segments, and measure the maximum width of noise for each segment. (Drift should be excluded.) Record the average of those values as the noise level.

Also measure the maximum baseline fluctuation in fifteen minutes, and multiply this value by 4 to get the drift in  $\mu$ Scm<sup>-1</sup>/h.

### Plotting data and taking readings

<When the Chromatopac is used>

Press the [PLOT] key to begin plotting. Use the following to measure the recording.

• C-R7Aplus and C-R8A	1/10 of the chart full scale is 14 mm.
• C-R7A	1/10 of the chart full scale is 15 mm.
• C-R4A	1/10 of the chart full scale is 16 mm.
• C-R5A/C-R6A	1/10 of the chart full scale is 13.5 mm.

<When CLASS software is used>

Press the [Start analysis] button to begin plotting. When analysis ends, print a report and read the noise and drift values from the report.

### Parameter settings for validation

Flow rate:

For validation analysis, the detector parameters should be set to the following values.

GAIN:	2 (0.1µScm <sup>-1</sup> )
RESPONSE:	4 (1s)
RANGE:	100 (for recorder; to check stabilization)
	4 (for recorder; to measure noise/drift)
ATTEN:	7 (for Chromatopac; to check stabilization)
	2 (for Chromatopac; to measure noise/drift)
CELL TEMP:	43 (CDD-10Avp)
Other parameters:	

Acceptance criteria (factory inspection criteria):

Oven temperature: 40°C (CDD-10Avp)

1.5 mL/min

Noise level must not exceed 4nScm<sup>-1</sup>. Drift must not exceed 75nScm<sup>-1</sup>/2hr.



### Check procedure (For suppressor system)

- Use a mobile phase of 14 mM sodium hydrogen carbonate. Flush the flow lines of the pump with the mobile phase using the PURGE function.
- Close the drain valve and start the pump to deliver the mobile phase at 1 mL/min. 2.
- Set the oven temperature to 30°C and the cell temperature to 33°C. For the CDD-10Asp 3. (suppressor system), set the oven temperature to 30°C. Start temperature control of the oven.

**TEMP** 3 3 CELL 2 5 55 0: of f

Set GAIN to 1 (0.01µScm<sup>-1</sup>).

GAIN1 1 Input

Set Response to 4 (1s). 5.

> RESPONSE Input 10

Set RANGE to 100 if a strip-chart recorder is used. Recording will be made at 1µScm<sup>-1</sup> full scale. Set a similar recording range if a data processor is used. For the Chromatopac, ATTEN = 7 will give 1.28µScm<sup>-1</sup> full scale.

> RANGE 100 Input 512

- Press [act] on the SCL-10Asp to start suppressor operation, and wait until the system stabilizes. Usually it takes four suppressor cycles for the system to stabilize.
- Set RANGE to 10 for 100nScm<sup>-1</sup> full scale plotting if a strip-chart recorder is used, or set 8. a similar recording range for the data processor (for the Chromatopac, set ATTEN to 4 for 160nScm<sup>-1</sup> full scale).

RANGE 10 Input 5 1 2 9. Plot the baseline for one cycle of suppressor operation, and check the baseline between 10 and 22 minutes after the cartridge switching. Select any segment of five minutes from the part where baseline is relatively flat. Divide the plot data by 30s to get 10 segments, and measure the maximum width of noise for each segment. (Drift should be excluded.) Record the average of those values as the noise level.

Also measure the maximum baseline fluctuation in twelve minutes between 10 to 22 minutes after the cartridge switching, and record this valve.

### Plotting data and taking readings

<When the Chromatopac is used>

Press the [PLOT] key to begin plotting. Use the following to measure the recording.

C-R7Aplus and C-R8A	1/10 of the chart full scale is 14 mm.
• C-R7A	1/10 of the chart full scale is 15 mm.
• C-R4A	1/10 of the chart full scale is 16 mm.
• C-R5A/C-R6A	1/10 of the chart full scale is 13.5 mm.

<sup>&</sup>lt;When CLASS software is used>

Press the [Start analysis] button to begin plotting. When analysis ends, print a report and read the noise and drift values from the report.

### Parameter settings for validation

For validation analysis, the detector parameters should be set to the following values.

	GAIN:	1 (0.01µScm <sup>-1</sup> )
	RESPONSE:	4 (1s)
	RANGE:	100 (for recorder; to check stabilization)
		10 (for recorder; to measure noise/drift)
	ATTEN:	7 (for Chromatopac; to check stabilization)
İ		4 (for Chromatopac; to measure noise/drift)
	CELL TEMP:	33 (CDD-10Avp)
	TYPE:	312
	MPCNC:	14
	INJDLY:	7
	S.INTVL:	22
	REGENTM:	8.8
	Other parameters:	
	Flow rate:	1 mL/min
	Oven temperature:	30°C

Acceptance criteria (factory inspection criteria):

Noise level must not exceed 0.7nScm<sup>-1</sup>.

Drift must not exceed 48nScm<sup>-1</sup>/12min.



- If a problem occurs during operation, system validation is performed to determine whether the cause lies with the LC system or with the analytical method.
- The system validation procedure described in this section is the standard procedure for determining whether the LC system itself is functioning normally, and constitutes the basis of LC system inspection.
- System suitability tests must be run to verify that the system is performing in accordance with
  the analytical conditions set by the user. If suitability checks indicate an abnormality, the system
  validation described below should be performed before proceeding any further.
  - If the LC system passes the system validation, it can be assumed that the LC system is normal and that the problem lies with the particular analytical method/conditions being used.
  - If the LC system does not pass the system validation, it may be assumed that there is an abnormality in the system, and component validation must be performed to identify the malfunctioning component(s).
- System validation is performed at installation, and at periodic intervals thereafter.

## 8.6.1 Validation of non-suppressor system What the validation consists of:

The system being validated is used to perform an analysis, and the retention time, peak area and peak height information is obtained for each peak. The data is then examined to check for reproducibility. Reproducible data validates the system.

The system should consist of the following components: pump, injector, column oven, detector and data processor.

## **Equipment required for validation**

Column	Shim-pack IC-A3 (Part No. 228-31076-91).
Mobile phase	8 mM p-hydroxybenzoic acid and 3.2 mM bis-tris and
	50 mM boric acid
Anion standard sample*	F <sup>-</sup> , Cl <sup>-</sup> 1ppm each

<sup>\*</sup>Use commercially available standard samples after dilution.

## Checking and preparing the LC system

- 1. Check all the connections in the LC system. For details, refer to individual instruction manuals. If a Chromatopac is used, it should be connected to the detector accessory signal cable with the connector supplied with the Chromatopac, and the signal cable should then be connected to the detector integrator terminals.
- 2. Check the LC system plumbing.

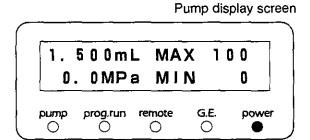
  In particular, ensure that the tubing between (a) the autosampler plumbing outlet and the column inlet, and (b) the column outlet and the detector inlet, has an I.D. of no more than 0.3 mm, and is no more than 30 cm in length. This will keep the volume of liquid that is not in the column as low as possible.



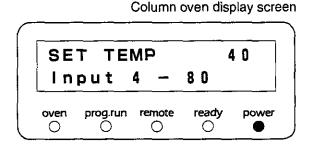
- 3. Clean the system flow lines as required. Refer to "5.9 Cleaning the flow lines" for the procedure.
- 4. When cleaning is finished, pour mobile phase into the reservoir, and reconnect the column.

### Validation procedure

1. On the pump display screen, set the pumping flow rate to 1.5 mL/min.



2. On the column oven display screen, set the column oven temperature to 40°C.



3. On the CDD-10Avp, set the cell temperature to 43°C.



- 4. Press pump on the pump keypad, and oven on the column oven keypad. Pumping and temperature regulation will begin.
  - At this point, verify that liquid flows through the detector outlet tubing and that there are no leaks from any connections.
- 5. Set the detector parameters. For the settings, see "Parameter settings for validation" on the next page. (See the relevant section of this manual for setting procedures.)
- 6. Set the data processor parameters. For the settings, see "Parameter settings for validation" on the next page. (See the data processor instruction manual for setting procedures.)
- 7. Monitor the baseline.

When the baseline has stabilized, press the detector vero key, then inject 50µL of mobile phase twice, and verify that no peaks are observed at the second injection.



- 8. Inject 50µL of the test sample, and analyze it. Repeat this operation at least five times. For a manual injector, inject an amount of test sample equal to 5 times the loop capacity.
- 9. From the peak data obtained from five successive analyses, derive the coefficients of variation (C.V.'s) for: retention time, peak area and peak height of F and Cl.

$$RSD(C.V.) = (SD/X) \times 100$$

$$SD = \sqrt{\frac{\sum\limits_{i=1}^{n}(Xi - \overline{X})^{2}}{n-1}}$$

$$X = (X1 + X2 \cdot \cdot \cdot Xn - 1 + Xn)/n$$

n : Number of analyses

 $X1 \cdot \cdot Xn$ : retention time (or areas/heights) of various peaks

X: Average

SD: Standard deviation C.V: Coefficient of variation

Fig. 8.3

#### Parameter settings for non-suppressor system validation

The parameters to be set for the various devices when validation analysis of an isocratic system is performed are given below.

• Pump	Flow rate:	1.5 mL/min	
	P.MAX:	20.0 MPa	
Column oven	Oven tempera	ture: 40°C	
• Time program	15.00 STOP		
Autosampler	(For SIL-10A	/10ADvp Series:)	
	SAMPLING S	SYRINGE SPEED:	$2\mu L/\text{sec}$ (SIL-10AF (10AP))
	SAMPLING S	SYRINGE SPEED:	15μL/sec (SIL-10ADvp)
	EXCESS VOI	LUME:	50μL (SIL-10AF (10AP))
• CDD-10Avp	GAIN:	3 (1μScm <sup>-1</sup> )	
	RESPONSE:	4 (1.0s)	
	POLARITY:	+	
	CELL TEMP:	: 43 (°C)	
Data processor	WIDTH:	5	
	DRIFT:	0	
	T.DBL:	0	
	ATTEN:	10	
	SLOPE:	1200	
	MIN.AREA:	10000	
	STOP.TM:	7	
	SPEED:	5	

#### Acceptance criteria

The C.V.'s obtained must satisfy the following criteria:

Retention time C.V. must not exceed 0.5%

Peak area C.V. must not exceed 1.0%

Peak height C.V. must not exceed 1.0%

#### 8.6.2 Validation of suppressor system

What the validation consists of:

The system being validated is used to perform an analysis, and the retention time, peak area and peak height information is obtained to each peak. Then the data is examined to check for reproducibility. Reproducible data validates the system.

The system should consist of the following components: pump, autosampler, column oven, detector and data processor.

#### **Equipment required for validation**

Column	Shim-pack IC-SA2 (Part No. 228-38983-91).
Mobile phase	12 mM sodium hydrogen carbonate
	0.6 mM sodium carbonate
Suppressor cartridge	For anion analysis (Part No.228-40612-91)
Anion standard sample	F-, Cl- 1ppm each

#### Validation procedure

- 1. In the Analysis File screen of the SCL-10Asp, set A.FLOW (pump flow rate) to 1 mL/min, and OVEN.T (oven temperature) to 30°C.
- 2. In the same screen, move the cursor to the DET.A area. Press func, and the [PARAM] function key appears at [f5].

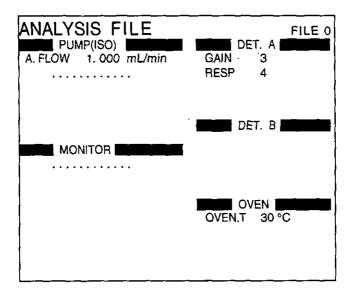


Fig. 8.4

- 3. The Detector Control screen for the CDD-10Avp/10Asp appears. Set the parameters as shown in "Parameter settings for validation," p. 8-21.
- 4. Press [act] on the SCL-10Asp to start pump delivery and oven temperature control. Also, the suppressor control is started.

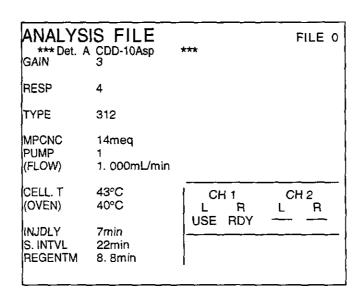


Fig. 8.5



- Set the data processor parameters. For the settings, see "parameter settings for validation", p. 8-21.
- Monitor the baseline. When the baseline has stabilized, press the [A.ZERO] function key 6. on the SCL-10Asp or the detector's zero key. Then inject 50µL of mobile phase twice, and verify that no peaks are observed the second time.
- Inject 50µL of the test sample and perform an analysis run. Repeat the injection at least 7. five times.

For a manual injector, inject an amount of test sample equal to 5 times the loop capacity.

From the peak data obtained from five successive analyses, derive the coefficients of variation (C.V.'s) for retention time, peak area and peak height of the chlorine ion.

 $RSD(C.V.) = (SD/X) \times 100$ 

$$SD = \sqrt{\frac{\sum\limits_{i=1}^{n}(Xi - \overline{X})^{2}}{n-1}}$$

 $X = (X1 + X2 \cdot \cdot \cdot \cdot Xn - 1 + Xn)/n$ 

n: Number of analyses

X1 · · Xn: retention time (or areas/heights) of various peaks

X : Average

SD: Standard deviation C.V.: Coefficient of variation

Fig. 8.6

#### Parameter settings for suppressor system validation

The parameters to be set for the relevant components for suppressor system validation are shown below.

• Pump	A.FLOW:	1.0 mL/min	
	P.MAX:	10 MPa	
<ul> <li>Column oven</li> </ul>	OVEN.T:	30°C	
<ul> <li>Time program</li> </ul>	(TIME: FUN:	VALUE)	
	16.5:	STOP	
<ul> <li>Autosampler</li> </ul>	(For SIL-10A	/SIL-10Dvp Series:	r)
	SAMPLING	SYRINGE SPEED:	2μL/s (SIL-10AF (10AP))
	SAMPLING S	SYRINGE SPEED:	15μL/s (SIL-10ADvp)
	EXCESS VO	LUME:	50μL (SIL-10AF (10AP))
• CDD-10Asp	GAIN:	3 (1µScm <sup>-1</sup> )	
	RESP:	4 (1 s)	
	CELL TEMP	: 33 (°C)	
	TYPE:	312	'
	MPCNC	14 meq	
	INJDLY:	4.5 min	
	S.INTVL:	21 min	
	REGENTM:	6.5 min	•
<ul> <li>Data processor</li> </ul>	WIDTH:	5	
	DRIFT:	0	
	T.DBL:	0	
	ATTEN:	10	
	SLOPE:	100	
	MIN.AREA:	100000	
	STOP.TM:	16 min	
	SPEED:	5 mm/min	

#### Acceptance criteria

The C.V. values obtained must satisfy the following criteria:

Retention time C.V. must not exceed 0.5%

Peak area C.V. must not exceed 1.0%

Peak Height C.V. must not exceed 1.0%



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# **Chapter 9 Troubleshooting**

CONTENTS	9.1	Symptoms, Causes and Corrective Action	9 - 2
	9.2	Error Messages	9 - 5



# 9.1

### **Symptoms, Causes and Corrective Action**

There are many possible reasons for problems, therefore, only most common are described here. The tables below describe major symptoms that are likely to occur, their probable causes, and corrective action. Detailed information and procedures can be found on the pages indicated. If the corrective action fails to rectify the problem, or if other problems occur, contact your Shimadzu representative.

Symptom or malfunction	Probable cause	Correc	ctive action	Page
Power does	1. Power plug disconnected.	Connect plug	;-	5-7
not come on when power	Broken wire in the power cord	Replace with same type.	• Replace with a new cord of the	
switch is turned ON	3. Power supply does not conform to unit's power supply specification	• Use a power to unit's portion.	• Use a power supply that conforms to unit's power supply specifica-	
	4. Fuse blown	Replace fuse		10-4
[OVER] is dis- played for con- ductivity value	Recorder pen is far below the original baseline	Press zero		6-5
Recorder base-	1. RANGE set to 0	Set appropria	te value for RANGE.	6-18
line does not change	2. The cell is not connected properly.	Check the c cables.	onnection of the cell	5-25
	<ol> <li>Recorder pen is far below the original baseline.</li> <li>([OVER] will be displayed for the absorbance value.)</li> </ol>	Press (zero). The pen will return to the baseline.		6-5
Peak area is	The peak is so large that the	Select a GAIN	value of lower sensi-	
not propor-	signal is saturated in the	tivity. The cond	uctivity signal is satu-	
tional to the	detector.	rated beyond the	e following ranges.	
concentration.		Gain	Signal	
	•	1 (0.01µScm <sup>-1</sup> )	about 6μScm <sup>-1</sup>	
		2 (0.1µScm <sup>-1</sup> )	about 60µScm <sup>-1</sup>	
		3 (1µScm <sup>-1</sup> )	about 600µScm <sup>-1</sup>	6-16
j		4 (10μScm <sup>-1</sup> )	about 6,000µScm <sup>-1</sup>	]
<u> </u>		5 (100μScm <sup>-1</sup> )	about 60,000µScm <sup>-1</sup>	
	Conductivity of eluent (background) is exceeding 100µScm <sup>-1</sup> when GAIN is	Select a GAIN tivity.	value of lower sensi-	
	set to 1 (0.01µScm <sup>-1</sup> ).			
[OVER +] or	Conductivity of eluent	The auto zero ad	justment range is as fol-	
[OVER -] is	(background) is too high.		opriate GAIN setting.	
shown on the		Gain	Auto zero range	
display and		I (0.01μScm <sup>-1</sup> )	about 100µScm <sup>-1</sup>	]
auto zero has		2 (0.1µScm <sup>-1</sup> )	about 4,000µScm <sup>-1</sup>	6-16
no effect.		3 (1μScm <sup>-1</sup> )	about 4,000µScm <sup>-1</sup>	
		4 (10μScm <sup>-1</sup> )	about 9,000µScm <sup>-1</sup>	
1		5 (100μScm <sup>-1</sup> )	about 90,000µScm <sup>-1</sup>	

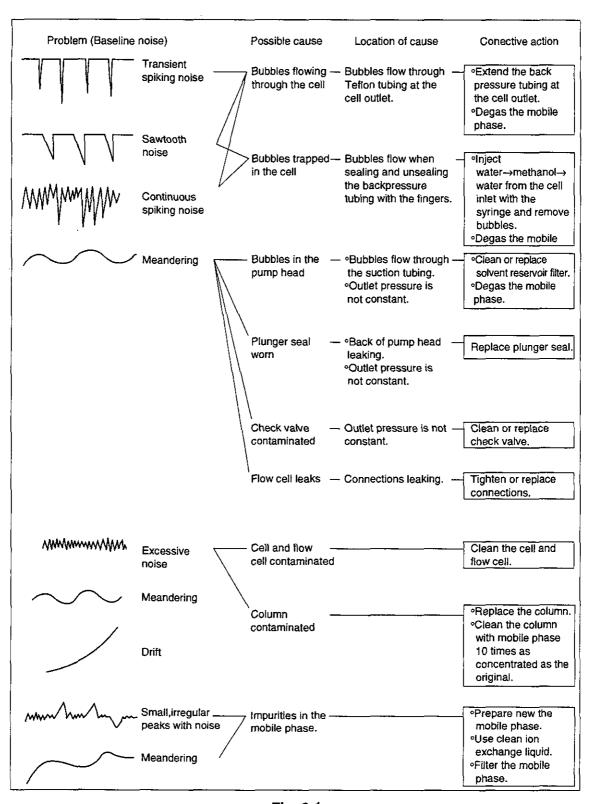


Fig. 9.1

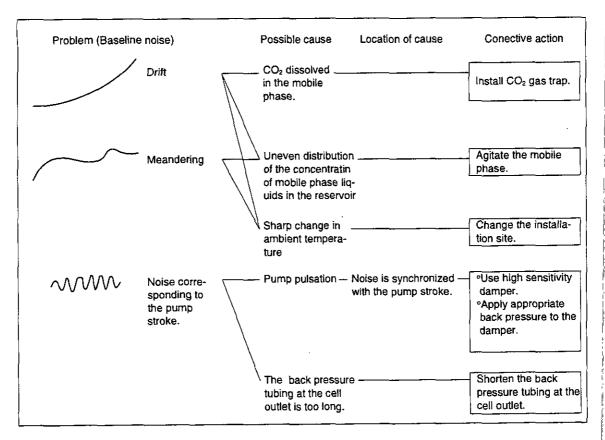


Fig. 9.2

This unit has several diagnostic functions. Upon detection of an abnormality, an alarm sounds and an error message appears on the display.

The error messages are listed below, along with possible causes and corrective actions.

NOTE

Each message falls into one of the following three categories. The category is shown in

the "Type" column.

Fatal:

The unit stops operation.

Press CE to clear the error message.

Alarm:

The unit continues operating.

Press (E) to clear the error message.

Warning:

The unit continues operating.

Error message disappears after a few seconds.

Error	message				
RC	OM FA	ILUR	E		
reger	n prog.run	remote	pol	power	,

**Troubleshooting** 

Error message	Type		Cause and action
ROM FAILURE (ROM error)	Fatal	Action:	ROM error (electronic failure). Turn power OFF and contact your Shimadzu representative.
RAM FAILURE (RAM error)	Fatal	Action:	RAM error (electronic failure). Turn power OFF and contact your Shimadzurepresentative.
ERR VALVE1 HOME  Suppressor Ch. 1 valve home position error.  (For CDD-10Asp only.)	Alarm		The valve is not operating properly due to one of the following reasons.  (1) The valve is stuck due to dirt or precipitates.  (2) The VALVE cable is not connected properly.
Suppressor Ch. 2 valve home position error.  (For CDD-10Asp only.)		Action:	<ul> <li>(3) Problem in the valve motor or drive circuit</li> <li>(1) Disassemble the valve and clean or replace the rotor and stator.</li> <li>(2) Check the connection of the cables.  If those are not the cause, contact your Shimadzu representative.</li> </ul>
ERR SUP1 HI VOLT Suppressor Ch. 1 high voltage error (For CDD-10Asp only.)	Alarm	Cause:	The actual current passing through the suppressor cartridge did not reach the preservalue one minute after the start of regeneration
ERR SUP2 HI VOLT Suppressor Ch. 2 high voltage error		Action:	tion. The suppressor cartridge may have become degraded. Replace the cartridge.
(For CDD-10Asp only.)  ERR BG1 TOO HIGH  ERR BG2 TOO HIGH	Alarm	Cause:	GAIN1/GAIN2 was set to 1 and the background level exceeded 100μScm <sup>-1</sup> whe autozero was performed. GAIN1/GAIN should be 2 or larger if background is morthan 100μScm <sup>-1</sup> .
NOT PROTECTED	Alarm	Cause:	Set GAIN1/GAIN2 to 2 or higher.  The memory contents were lost due to abnormal line voltage or other reasons.  If this error is displayed each time the power cycled, the back-up battery may have been consumed. Turn the power off and contains

# Maintenance

# Chapter 10 Maintenance

CONTENTS	10.1	Periodic Inspection and Servicing	10 -	2
	10.2	Flow Cell Cleaning	10 -	3
	10.3	Fuse Replacement	10 -	4

To ensure long-term, safe and trouble-free operation, this unit must be periodically inspected and serviced. If a service and inspection contract was purchased, a Shimadzu service representative can perform this periodic inspection and servicing. Contact your Shimadzu representative for details.

## **!** CAUTION

- Replacement parts must be of the specifications given in "4.1 Accessories" or "11.2 Replacement Parts." If any other parts are used, they could cause injury or malfunction.
- Never remove the unit's main cover. Doing so could result in injury or malfunction. Contact your Shimadzu representative for any repairs requiring removal of the main cover.
- Unless the instructions tell you otherwise, always turn off the power and disconnect the power cable plug from the outlet before performing inspection or maintenance. Otherwise, fire, electric shock or malfunctions could occur.

#### 10.1.1 Preparation for inspection/maintenance

- Replace the mobile phase in the flow lines with water.
- Wipe away any dirt from the front panel and the main cover.
- Wipe away any dirt from the keypad with a paper towel or a soft cloth moistened with water.

#### 10.1.2 Maintenance operations and intervals

Operation	1 year	2year	3 year	6 year	Remarks	Page
Cell cleaning	0					10-3
Fuse replacement			0			10-4

10

#### 10.1.3 Post-inspection/maintenance leak check

After inspection/maintenance, pump liquid through the flow lines, and check for any leaks. If any leaks are found, refer to Section 9.1 and take appropriate action.

Since contaminated electrodes in the cell do not allow for stable analysis, regular cell cleaning is recommended. Also, when the cell is used after a long storage period, or exhibits baseline noise, cleaning of the cell is required.

To clean the cell, follow the procedure described in "5.9 Cleaning the flow lines," so that the cell is cleaned along with the entire analytical flow lines.

## **MARNING**

- Before replacing fuses, turn the unit's power switch off and remove its power plug from the outlet.
- For replacement, only use fuses of the correct type and rating.

Failure to heed the above could result in fire, electric shock or short circuits.

The correct rating of the fuses is:

#### For 100V AC/120V AC unit

Part	Туре	Part No.
250V 4AT (5 × 20)	Maintenance part	072-02004-22

#### For 230V AC unit

Part	Туре	Part No.
250V 2AT (5 × 20)	Maintenance part	072-02004-19

Use a flat head screwdriver to pry off the fuse holder.

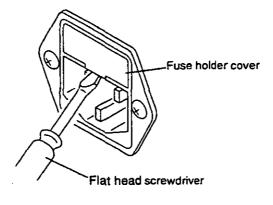


Fig. 10.1

- Pull out the fuse holder, and remove the fuses from it.
- Insert new fuses into the fuse holder. 3.

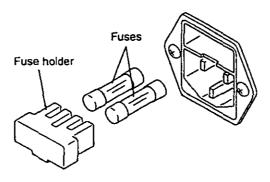


Fig. 10.2

4. Push the fuse holder into its socket so that it clicks in to place.

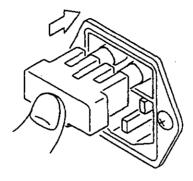


Fig. 10.3

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# Chapter 11 Miscellaneous

CONTENTS	11.1	Specifications	11	- 2
	11.2	Replacement Parts	11	- :

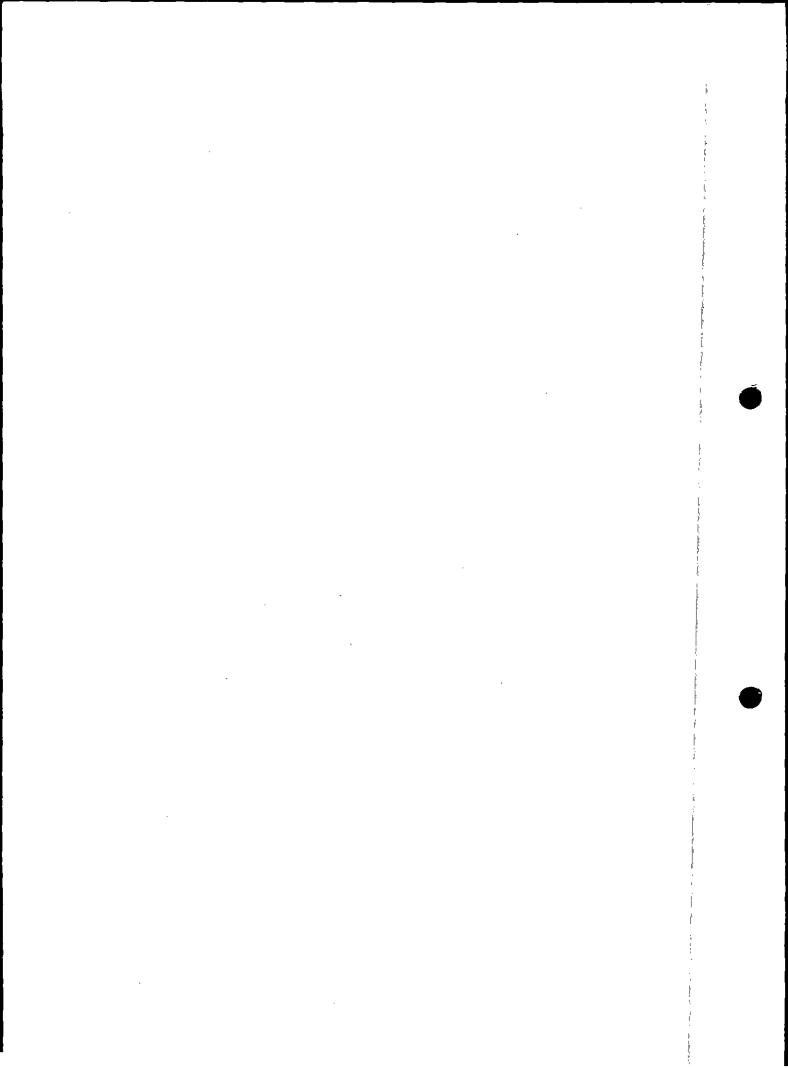


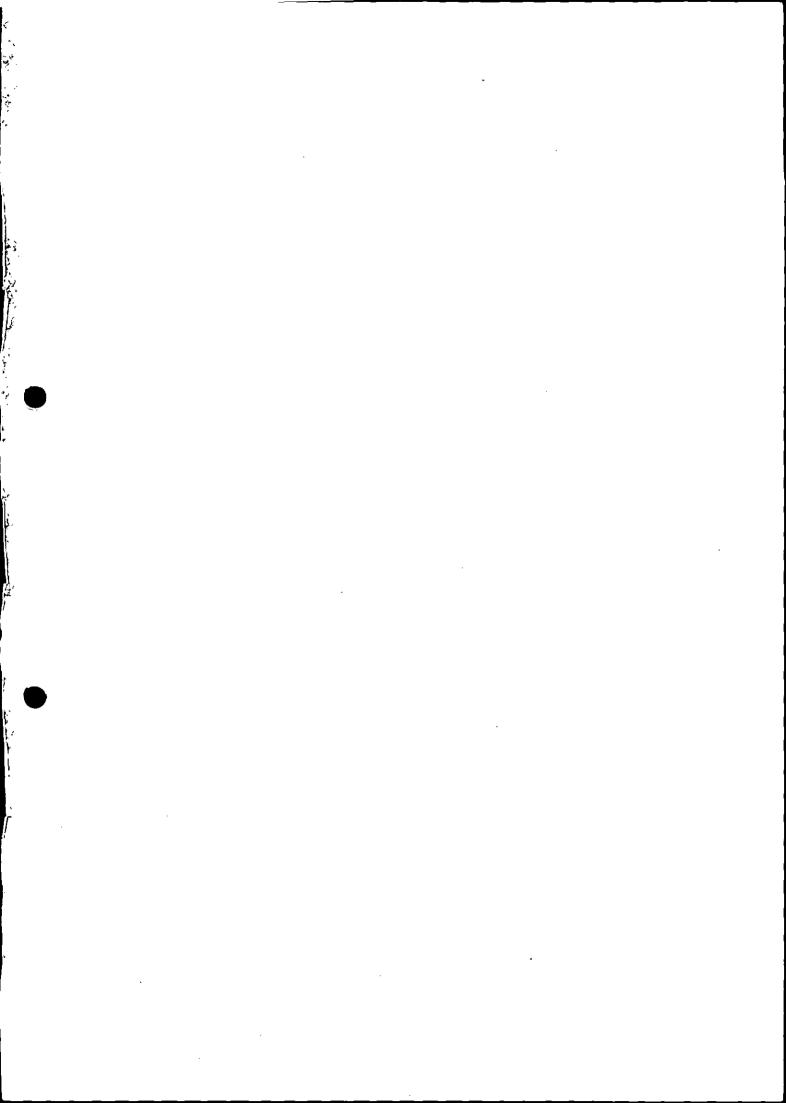
# **Specifications**

ltem	Specifications				
Noise level	0.7nScm <sup>-1</sup>				
(suppressor system)	Background: 40µScm <sup>-1</sup> , cell temperature: 33°C				
Conditions	Flow rate: 1 mL/min, response: 1s				
(non-suppressor system)	4nScm <sup>-1</sup>				
Conditions	Background: 285µScm <sup>-1</sup> , cell temperature: 43°C				
	Flow rate: 1.5 mL/min, response: 1s				
Drift	48 nScm <sup>-1</sup> /12min				
(suppressor system)	Background: 40µScm <sup>-1</sup> , cell temperature: 33°C				
Conditions	Flow rate: 1 mL/min, response: 1s				
(non-suppressor system)	25nScm <sup>-1</sup> /hr With no change in temperature.				
Conditions	Background: 285µScm <sup>-1</sup> , cell temperature: 43°C				
	Flow rate: 1.5 mL/min, response: 1s				
Temperature Coefficient	25nScm <sup>-1</sup> /°C				
Conditions	Background: 285µScm <sup>-1</sup> , cell temperature: 43°C				
	Flow rate: 1.5 mL/min, response: 1s				
Cell	Cell volume: 0.25µL				
	Cell constant: 25 cm <sup>-1</sup>				
	Solvent-contacting materials: PEEK, Teflon, and SUS316				
	Max. operating pressure: 2.9 MPa (30kgf/cm²)				
Response	Selectable in 10 steps corresponding to time constant:				
*	0.05s, 0.1s, 0.5s, 1.0s, 1.5s, 2.0s, 3.0s, 6.0s, 8.0s, 10.0s				
Zero adjustment	Auto zero function and baseline shift function				
Output Connectors	REMOTE connector (for connection with the system controller)				
-	EVENT connector				
	Output: EVENT1, EVENT2				
}	Input: Program start, program stop				
	Signal output connector 1 (Integrator output)				
	Signal output connector 2 (10 mV recorder output)				
Operating temperature	4-35°C				
range	ļ		ļ		
Dimensions	260W × 140H × 420D				
Weight	6.0 kg				
Power supply	CDD-10Avp				
}	Part No.	Power	supply		
	228-41300-31	100VAC ±10%	250VA 50-60Hz		
	228-41300-32	120VAC ±10%	250VA 50-60Hz		
Į	228-41300-38	230VAC ±10%	250VA 50-60Hz		
}	CDD-10Asp				
	Part No.	Power	supply		
	460-08101-31	100VAC ±10%	250VA 50-60Hz		
{	460-08101-32	120VAC ±10%	250VA 50-60Hz		
	460-08101-33	230VAC ±10%	250VA 50-60Hz		

## 11.2.1 Replacement Parts

Part	Part No.	Remarks	
Cell Semi Assy	460-08135-92	Plastic cell block with PEEK inlet/outlet tubing	
PEEK tubing, S	460-08131	Outlet PEEK tubing (heat-formed)	
Fuse, 4A	072-02004-22	For 100V/120V	
Fuse, 2A	072-02004-19	For 230V	
PCB Assy, CDD10A CPU	460-08102-91	With ROM	
PCB Assy, 10Avp CPU	228-34259-91	Without ROM	
PCB Assy, PC-1CD	460-08057-91	Main PCB (Ch. 1)	
PCB Assy, PC-3CD	460-08087-91	Suppressor PCB (Ch. 1)	
PCB Panel Assy	228-39061-92		
Switch Assy, CDD-10A	460-08146-91	Power switch	
Lithium Battery	074-73307-01		
Key-top Det	228-39180-02		
Terminal for EVENT cable	228-35307-91	10 pcs.	
Interconnect PCB Assy	228-40453-91		
Power supply LDC-30F-2	074-80678-11	For Ch. 1 and Ch. 2	
Power supply GLM65-48	460-08177	For suppressor	
Temperature control block	228-17656-92		
Assy			







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