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228-10492A

CDD-6A
CONDUCTIVITY DETECTOR
INSTRUCTION MANUAL

SHIMADZU CORPORATION

ANALYTICAL INSTRUMENTS DIVISION

KYOTO, JAPAN

CAUTION

We thank you for having purchased our CDD-6A Conductivity Detector. You are requested to pay special attention to the following instructions so that operation under optimum conditions over a long period of time will be ensured.

(1) Flow cell tubing connection

> The Shimadzu LC tubing fittings, for the flow cell as well as other connections, have been modified except for the fittings applied to both ends of a column. Before connecting the column to the detector flow cell tubing, be sure to read the supplied "New Connector Instruction Manual" carefully.

(2) Installation site

To maintain equipment service life and reliability, the installation site should be free of vibration, sources of electrical noise, corrosive gases and dust.

In addition to the above, the equipment should not be exposed to extreme temperature changes, strong air currents or direct sunlight, since these conditions may adversely affect equipment performance, especially optical stability when it is used at high sensitivity.

(3) Power source

To ensure safety, use a three-hole outlet as the power source: the power cord of this equipment is of the 3-wire type including a grounding conductor.

When the use of a two-hole outlet is unavoidable, be sure to attach the supplied 2P adaptor to the plug at the tip of cord and to provide adequate grounding by using the grounding terminal at the rear of the equipment.

(4) Precautions to prevent clogging of the flow cell:

Dirty or clogged flow cells are the most frequent causes of trouble in the detector. After analyzing a highly viscous sample, flush it thoroughly from the flow cell, using a large amount of mobile phase.

When this instrument is used in ion chromatography, a buffer solution is usually employed as the mobile phase.

Before suspending the analysis for one day or more, clean the flow cell with distilled water to prevent clogging the cell with the recrystallized buffer solution.

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1. Parts Check List

After unpacking, check the main body and attachments against the following list.

CDD-6A Main Body (228-16750-91 ~ 95)

	Description	Parts No.	Quantity
1	CDD-6A Controller	228-16730-{91 / 95}	1
2	CDD-6A Detection cell	228-17200-91	1

The last two digits 91 ~ 95 of the controller parts No. are classified as follows depending on the power requirements.

P/N 228-16730-91	100V AC ± 10V	1.5A	50, 60Hz
P/N 228-16730-92	115V AC ± 10V	1.5A	50, 60Hz
P/N 228-16730-93	220V AC ± 20V	1.0A	50, 60Hz
P/N 228-16730-94	240V AC ± 20V	1.0A	50, 60Hz
P/N 228-16730-95	230V AC ± 20V	1.0A	50, 60Hz

Standard Attachments

	Description	Parts No.	Quantity
1	Signal cord	201-47223	1
2	Preheat coil	228-17659-91	1
3	Pipe cover 4ϕ X 2ϕ X 2m	016-43205	1
4	Ferrule 1.6F	228-16000	8
5	Male nut 1.6MN	228-16001	8
6	Female nut 1.6FN	228-16002	2
7	Coupling union 1.6C	228-16004-03	4
8	Union 1.6U	228-00034	1
9	SUS 316 pipe 1.6ϕ X 0.3ϕ X 50cm	670-10006-02	1
10	Syringe lock 10, with needle	046-00003	1
11	Syringe adaptor	228-15672-91	1
12	Former connector ASSY	228-16063-91	1
13	Clamp	228-20486	1
14	Screw, M4 X 8	020-46547	1
15	New connector instruction manual	228-10432	1
16	Connector caution label	228-16065-02	1
17	Power cord for 100V or Power cord for 200V	071-60814-01 071-60814-02	1
18	Grounding adaptor (for 100V only)	071-60803-01	1
19	CDD-6A Instruction manual	228-10492	1
20	Ethylenediaminetetraacetic Acid, Disodium Salt (25g)	670-10480-03	1
21	HIC-6A Instruction manual	228-10490	1
22	Grounding adaptor ASSY	228-17246-91	1
23	Grommet 12 X 19	072-60012	2

2. Power Connection

(1) This instrument has the following power requirements.

P/N 228-16750-91	100V AC ± 10V	1A	50, 60Hz
P/N 228-16750-92	115V AC ± 10V	1A	50, 60Hz
P/N 228-16750-93	220V AC ± 20V	0.5A	50, 60Hz
P/N 228-16750-94	240V AC ± 20V	0.5A	50. 60Hz
P/N 228-16750-95	230V AC ± 20V	0.5A	50, 60Hz

Ensure that the voltage value indicated at the rear of the instrument conforms to the voltage of the power outlet. As unstable supply voltage cannot guarantee the desired performance, be sure to operate the instrument with stable voltage in the range indicated.

(2) Grounding

The power cord of this instrument contains three wires including the grounding conductor. Plug it into a three-hole outlet for proper grounding. When a two-hole outlet is used, attach a 2P adaptor to the plug at the cord tip, and be sure to ground the earth terminal at the rear of the instrument first before plugging in.

(3) After checking that the "POWER" switch is OFF, insert the supplied power cord into the power connector at the rear of the instrument.

3. Specifications

Cell volume:	0.25μℓ
Cell constant:	$25cm^{-1}$
Materials in contact with liquid:	SUS 316, PTFE, PCTFE
Cell pressure tolerance:	$30kg/cm^2$

Measuring sensitivity:	0.1 ~ 5120μScm^{-1} FS
	Determined by [gain setting] X [range setting]
Gain:	0.1, 1, 10μScm^{-1} 3-step selection
Range:	1, 2, 4, 8, 16, 32, 64, 128, 256, 512 10-step selection
Zero adjustment:	Auto zero by pushbutton switch
Zero adjustment range:	4000μScm^{-1} (with the gain set of 0.1 or 1μScm^{-1})
	9000μScm^{-1} (with the gain set of 10μScm^{-1})
Zero adjustment accuracy:	± 10/N% (N: Range)
Baseline positioning:	By FINE ZERO key switch (effective for RECORDER output terminals only)

Fine zero adjustment range:	Beyond the recorder full scale
Response:	FAST, STD, SLOW 3-step selection Corresponding time constants are 0.05, 0.75 and 1.5 sec.
Noise:	$0.004\mu Scm^{-1}$ max. With the response at STD when 2.5mM phthalic acid + 2.4mM Tris (hydroxymethyl) aminomethane solution at pH 4.0 (conductivity: approx. $250\mu Scm^{-1}$) is fed at the rate of 1.5mℓ/min. into the Shim-Pack IC-A1 Column to be used in the HIC-6A Ion Chromatograph.
Drift:	$0.025\mu Scm^{-1}$/h max. (Under the same conditions as above and at constant ambient temperature.)
Drift by ambient temperature change:	$0.025\mu Scm^{-1}$/°C max. (Under the same conditions as noise measurement)
Recorder:	1mV, 10mV
Integrator terminal output:	The signals of 10, 1 & $0.1mV/\mu Scm^{-1}$ are output according to the gain settings of 0.1, 1 & $10\mu Scm^{-1}$.
Optimum operating temperature range:	10°C ~ 35°C

Size:	Controller	25 (W) X 16 (H) X 40 (D) cm
	Cell	10 (W) X 19 (H) X 4 (D) cm
Weight:	Controller: 7kg	Cell: 0.8kg

Optimum operating temperature range:	10°C ~ 35°C

4. Measuring Mechanism

4.1 Cell Unit

Conductivity K of a solution is generally expressed as follows, by measuring the current I flowing when voltage E is applied across two opposite electrodes:

$$K_{[S]} = \frac{I\,[A]}{E\,[V]}$$

K is expressed in a unit of [S] (siemens) or (mho) that is the reciprocal of [Ω] (ohm).

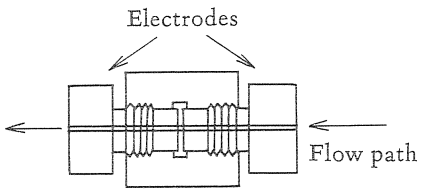
Conductivity is proportional to the area of electrodes (A) and inversely proportional to the distance between electrodes (L).

$$\text{Thus,}\quad K_{[S]} = \frac{A\,[cm^2]}{L\,[cm]} \cdot k$$

Since conductivity of the solution changes depending on the shape of the cell used in measurement, it generally refers to the conductivity measured at k, that is, A= 1 and L = 1, in the equation equation above. (More accurately, k is called specific conductivity.)

$$k = \frac{L\,[cm]}{A\,[cm^2]} \cdot K_{[S]} \qquad \text{Unit of } k = [Scm^{-1}]$$

This instrument is designed to detect this specific conductivity converting it to an output voltage. As the target solution is very low in conductivity, [μScm^{-1}] is used as the unit of measure.



Proportional constant L/A [cm^{-1}] in the equation above is determined by the cell shape, and is called the cell constant.

In this instrument, the cell is structured as shown in the figure above, and the cell constant is approx. 25cm^{-1}.

4.2 Signal Processing System

Fig. 4.1 illustrates the block diagram of the signal processing system. This instrument is designed to detect conductivity in the cell, i.e., the output voltage is proportional to the current flowing in the cell.

To measure the current flowing in the cell, an AC voltage of 1KHz is applied to the cell because DC voltage polarizes the electrode environs and does not allows accurate measurement of the conductivity of the solution.

The current flowing in the cell is amplified by A2, and fed to the balanced demodulator circuit in the subsequent stages as the voltage input. This input will be saturated if the cell is

high in conductivity. In non-suppressor type ion chromatography, high conductivity of the eluent itself (background) will cause amplifier saturation, disabling accurate measurement of conductivity.

To avoid this, the instrument is designed to produce AC $\sin\omega t$ with the phase opposite to AC $-\sin\omega t$ at A1, apply it to resistor array Sr and add the resulting current to the one flowing from the cell, thereby suppressing the background. With the "GAIN" key or "ZERO" key pressed, the microprocessor reads the output of amplifier A2 via comparator (1), selects the desired reference resistor via select switches K1 to K8 so as to minimize the output value, and changes the conductivity of synthesized resistor Sr. The output voltage Vi (t) of A2 is expressed:

$$Vi\ (t) = Rf \cdot (Ss - Sr)\ \sin\omega t$$

Where, Rf: amplification factor determined by the gain

Ss: conductivity of the cell

Sr: conductivity of reference resistor

Then, the resulting voltage Vi (t) is integrated with the cell applied signal $\sin\omega t$ by the balanced demodulator circuit in the subsequent stage, and the converted to Vo (t):

$$Vo\ (t) = Vi\ (t) \cdot Vr\ (t) = \frac{Rf\ (Ss - Sr)}{2} \cdot (1 - \cos 2\omega t)$$

The DC component in Vo (t) alone is extracted as Vf (t) by the smoothing circuit:

$$Vf\ (t) = \frac{1}{2} Rf\ (Ss - Sr)$$

The value (Ss - Sr) is reduced to some inadequate degrees by the background suppressor circuit; the background is nearly completely suppressed by the zero adjustment circuit made up of D/A converter and A4. This function is achieved by the feedback control, in which microprocessor changes the output to D/A converter so as to minimize output of A4 while observing the output of A4 via comparator (2). In this way, the relative value of conductivity of the cell with that of the eluent is available at the output of A4. In other words, the output of A4 is 0 when the eluent alone is present in the cell. When the sample ion flows into the cell, the difference in conductivity between the sample ion and the eluent appears at the output of A4. This value is read by the microprocessor via A/D converter and displayed on the digital meter on the panel.

In anion analysis, equivalent conductivity of the sample ion is generally higher than that of the eluent; the sample peak appears in a positive direction. In cation analysis, equivalent conductivity of the eluent becomes higher than that of the sample so that the peak generally appears in a negative direction. In this case, the output of A4 is inverted by A5 to give the apparent peak in a positive direction. This selection can be done by the POLARITY key on the front panel. This output is drived by the resistor and fed to AUX terminal as the integrator output. This is also fed to the recorder terminal through the range selection.

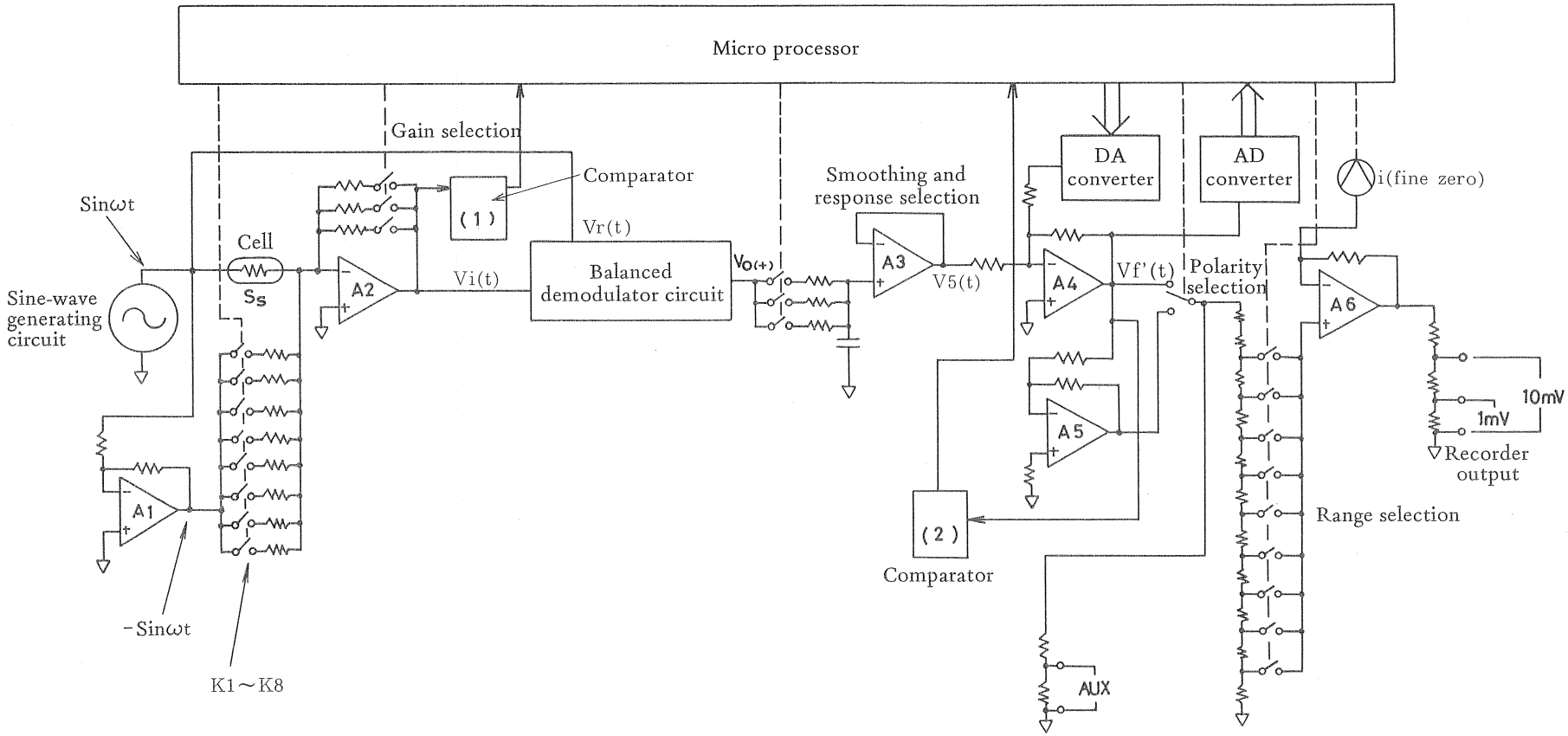
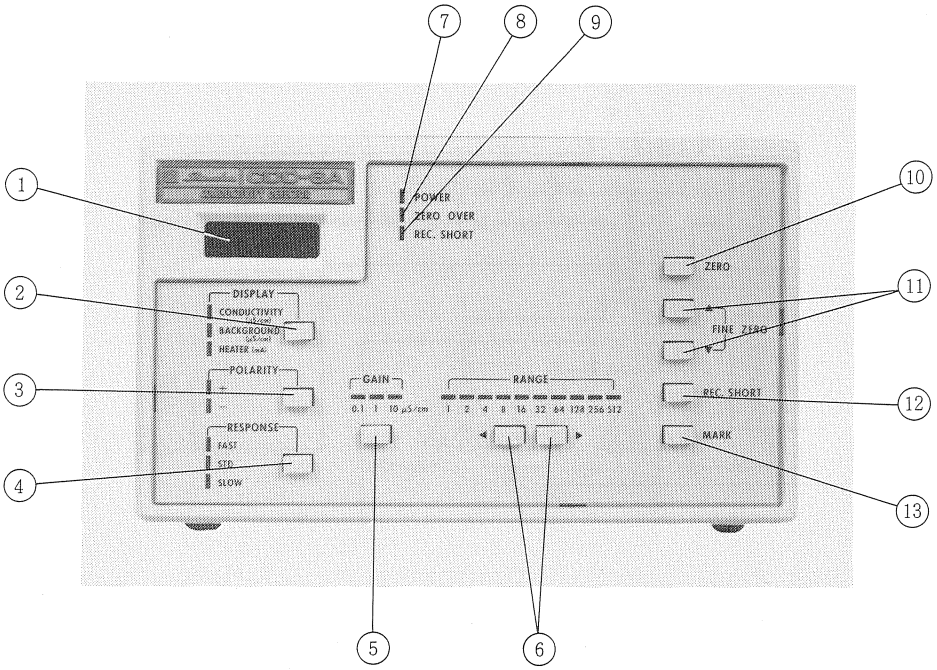


Fig. 4.1 Block Diagram of CDD-6A Signal Processing System

5. Parts Name and Function

5.1 Front View

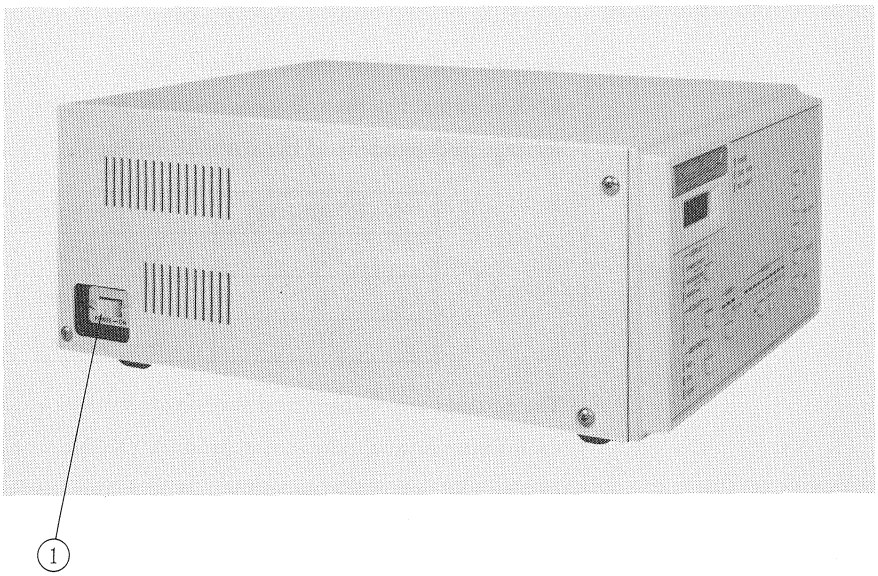


Reference No.	Display or Description	Function
①	Display	Displays conductivity of the sample peak, conductivity of the mobile phase (background) and the current values of the heat exchange heater. The display figures ranges from -0999 to +9999. When conductivity of the solution changes sharply in exchange of the mobile phase, the display blinks. In this case, press the "ZERO" switch; blinking stops to display the correct value. This is not a failure, so requires no special measures in exchange of the mobile phase.
②	Display select switch	Lights either of the following three LEDs and displays the selected content on the digital meter.
	CONDUCTIVITY (μS/cm)	Displays conductivity of the sample peak, or more accurately, the ratio of the change in conductivity before and after the auto zero.
	BACKGROUND (μS/cm)	Displays conductivity of the entire solution flowing in the flow cell. When the sample is not present in the cell, conductivity of the mobile phase is displayed.
	HEATER (mA)	Displays the current values of the heat exchange heater in the cell; approx. 200mA at the start of temperature control, and 30 ~ 60mA when the temperature reaches the maximum.

<table>
<tr><th>Reference No.</th><th>Display or Description</th><th>Function</th></tr>
<tr><td>③</td><td>Polarity select switch</td><td>Selects polarity of the voltage output for the recorder and integrator. Generally, (+) in anion analysis and (−) in cation analysis. Polarity is displayed by LED at the left of the switch.</td></tr>
<tr><td>④</td><td>Response select switch</td><td>Selects output response for the recorder and AUX signal. Time constants FAST, STD and SLOW are 0.05, 0.5 and 1.5 seconds respectively. LED at the left of the switch lights according to the response settings.</td></tr>
<tr><td>⑤
⑥</td><td>GAIN select switch
RANGE select switch</td><td>The measuring sensitivity of the recorder full scale is determined by [GAIN] × [RANGE] (μScm^{-1}).
For instance, at GAIN = 1 and RANGE = 4, the sensitivity of the recorder full scale becomes $4\mu Scm^{-1}$.
AUX terminal has the following outputs according to the GAIN settings (independent of the RANGE settings).
<table>
<tr><th>GAIN (μS/cm)</th><th>Output voltage</th></tr>
<tr><td>0.1</td><td>$10mV/\mu Scm^{-1}$</td></tr>
<tr><td>1</td><td>$1mV/\mu Scm^{-1}$</td></tr>
<tr><td>10</td><td>$0.1mV/\mu Scm^{-1}$</td></tr>
</table>
The settings are changed and the selected values are LED-displayed each time these switches are depressed. RANGE select switch is available in two modes ◀ and ▶ ; ◀ decreases the RANGE settings, and ▶ increases the settings. GAIN select switch changes the settings in the sequence of ↳ 0.1 → 1 → 10┘ each time it is depressed. Each mode selection actuates the auto zero function thus being delayed by a few seconds.</td></tr>
<tr><td>⑦</td><td>POWER pilot lamp</td><td>Lights when POWER is turned ON.</td></tr>
<tr><td>⑧</td><td>ZERO OVER pilot lamp</td><td>Lights when zero auto adjustment exceeds the specified range.</td></tr>
<tr><td>⑨</td><td>REC. SHORT pilot lamp</td><td>Lights when REC. SHORT switch is depressed to short the recorder signal output.</td></tr>
<tr><td>⑩
⑪</td><td>ZERO
FINE ZERO</td><td>Auto zero switch used to trigger the auto zero.
Shifts the baseline to the desired position on the recorder chart paper. This function is only effective for the recorder output and not available for AUX output.
▲ switch moves the baseline in a (+) direction, and ▼ switch moves it in a (−) direction while each is being depressed.</td></tr>
</table>

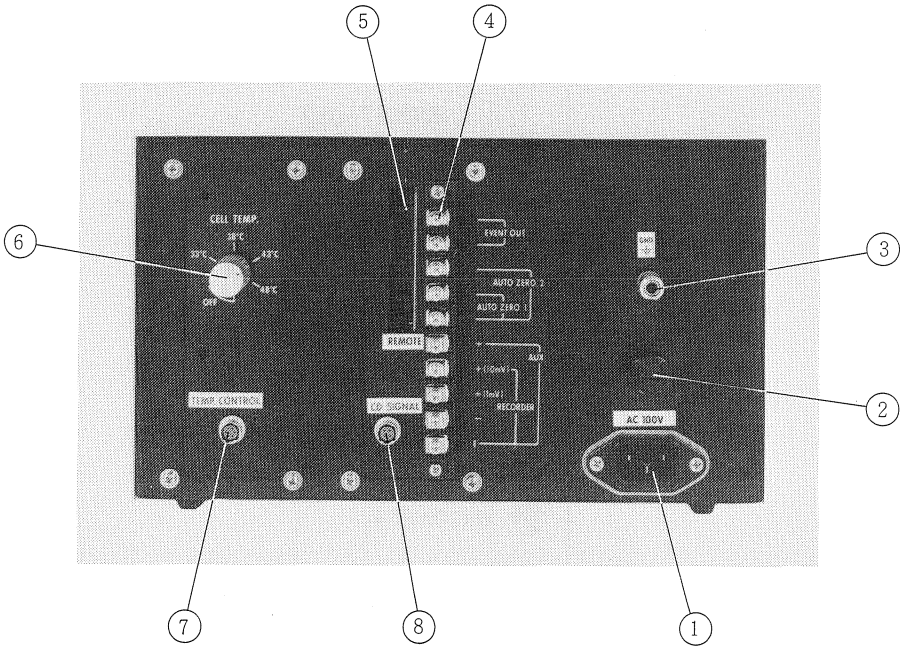
Reference No.	Display or Description	Function
⑫	REC. SHORT	With this depressed once, the recorder signal is shorted and REC. SHORT pilot lamp lights. With this depressed again, the switch mode is released. Keep it released during analysis.
⑬	MARK	Marks the baseline in a (−) direction. (Recorder signal output terminal only)

5.2 Left Side View



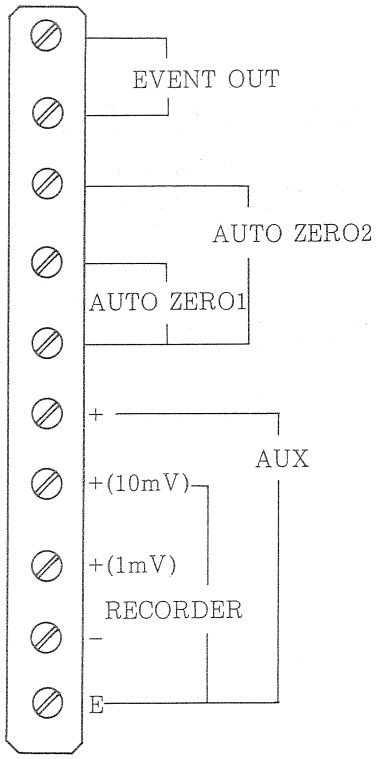
Reference No.	Display or Description	Function
①	Power switch	Power switch of this instrument. Setting this to the front panel side turns the power on. Setting this to the rear panel side turns the power off.

5.3 Rear View



Reference No.	Display or Description	Function
①	Power outlet	AC power cord with AC power input terminal is connected here.
②	Fuse holder	1.5A fuse (for the instrument with a rating of 100V AC or 115V AC) 1A fuse (for the instrument with a rating of 220V AC or 240V AC)
③	Earth (grounding) terminal	When a two-hole power outlet is used, be sure to ground this terminal first before plugging in.
④	Signal I/O terminals	Refer to 5.4.
⑤	Remote control connector	Used to interface this instrument with the Shimadzu system controller SCL-6A. This allows the setting of auto zero, polarity and gain, and their time programming from the system controller.
⑥	Temperature setting knob	Sets the heat exchanger temperature in the detection cell to 33, 38, 43 or 48°C. The detection cell is contained in the column oven. Be sure to set the oven temperature 3°C lower than the cell temperature. Set this switch to OFF when the cell temperature is not controlled.
⑦	TEMP CONTROL connector	Attached to the corresponding connector from the detection cell.
⑧	CD SIGNAL connector	Attached to the corresponding connector from the detection cell.

5.4 Signal I/O Terminals at Rear of Instrument



EVENT OUT	Connected across these terminals is a reed relay (50V, 0.1A) whose contact closes (for about 1 sec.) when the "MARK" switch on the front panel is depressed, or when AUTO ZERO-2 terminals are shorted. These terminals can be used to start external devices when a sample is injected.
AUTO ZERO-1	These terminals are used to trigger the auto zero from external devices. The auto zero is triggered by a momentary short (0.1 sec. or more) across these terminals via an external relay contact.
AUTO ZERO-2	A momentary short (0.1 sec. or more) across these terminals via an external relay contact triggers the auto zero, puts an event mark on the baseline, and closes the reed relay contact connected across EVENT OUT terminals.
AUX output terminal	The signal output terminal for the data processor. The (+) terminal of the signal cord is connected to this terminal. The (-) terminal is connected to RECORDER (-) terminal and earth terminal to E terminal.

This terminal has the output voltage corresponding to the "GAIN" setting. For instance, when the gain is set at $10\mu Scm^{-1}$, a peak of $5\mu Scm^{-1}$ is output to this terminal as the voltage change of 0.5mV. The Shimadzu C-R3A Chromatopac data processor has a plotter full scale of 1mV. Therefore the peak is half that of full scale when the attenuation is set at 0.

The following table shows the relation between the gain settings and AUX terminal output voltages.

Gain setting	AUX output voltage
$10\mu S/cm$	$0.1mV/\mu Scm^{-1}$
$1\mu S/cm$	$1mV/\mu Scm^{-1}$
$0.1\mu S/cm$	$10mV/\mu Scm^{-1}$

Note that if the output voltage exceeds 600mV, the signal will become saturated. At the gain setting of $1\mu Scm^{-1}$, for instance, the output signal will be saturated if a peak exceeds $600\mu Scm^{-1}$.

RECORDER terminal

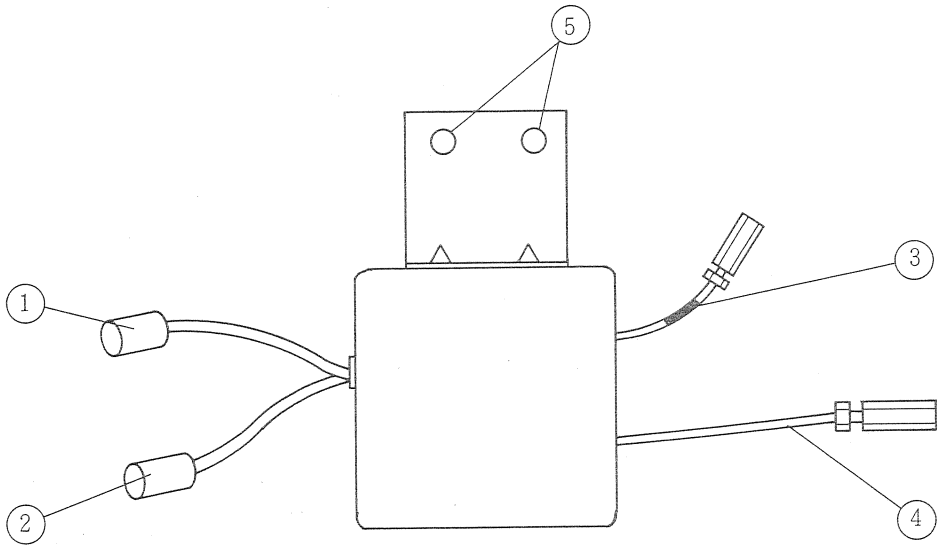
The signal output terminal for the recorder.

The (+) terminal of the signal cord is connected to the 10mV or 1mV terminal according to the full scale voltage of the recorder to be used. The (-) terminal is connected to the corresponding (-) terminal and earth terminal to E terminal.

When connecting this terminal to the data processors, the following should be noted; the output signal will be saturated if a peak is 600 time (or more) the gain setting.

The 1mV terminal output is equal to the AUX terminal output at RANGE setting of 1. In this instance, the voltage 10 times that of the 1mV terminal is output to the 10mV terminal.

5.5 Detection Cell



Reference No.	Description	Function
①	Connector (CD SIGNAL)	Attached to the corresponding connector at the rear of the controller.
②	Connector (TEMP CONTROL)	
③	Cell input pipe	Covered with blue tube.
④	Cell output pipe	
⑤	Clip buttons	Used to mount the cell in the Shimadzu CTO-6AS Column Oven. [Mounting] Pull this buttons forward until each clicks. Insert the protrusion at the rear into the CTO-6AS column mounting plate hole, then push it in until the latch clicks. [Removal] Pull the buttons forward until each clicks. Then, gently withdraw the cell assembly forward.

6. Installation, Operation and Instruction after Analysis

6.1 Installation and Operation

Installation and operation follow the procedures below. This detector is basically combined with the Shimadzu CTO-6AS Column Oven and preheater, so explanation is given on the basis of that combination.

(1) Installation and connection of flow cell tubing

(2) Flow cell cleaning

(3) Mobile phase liquid feed

(4) Column connection

(5) Startup and inspection

(6) Baseline recording and standard sample analysis

Each item will be hereafter described in some detail.

(1) Installation and connection of flow cell tubing

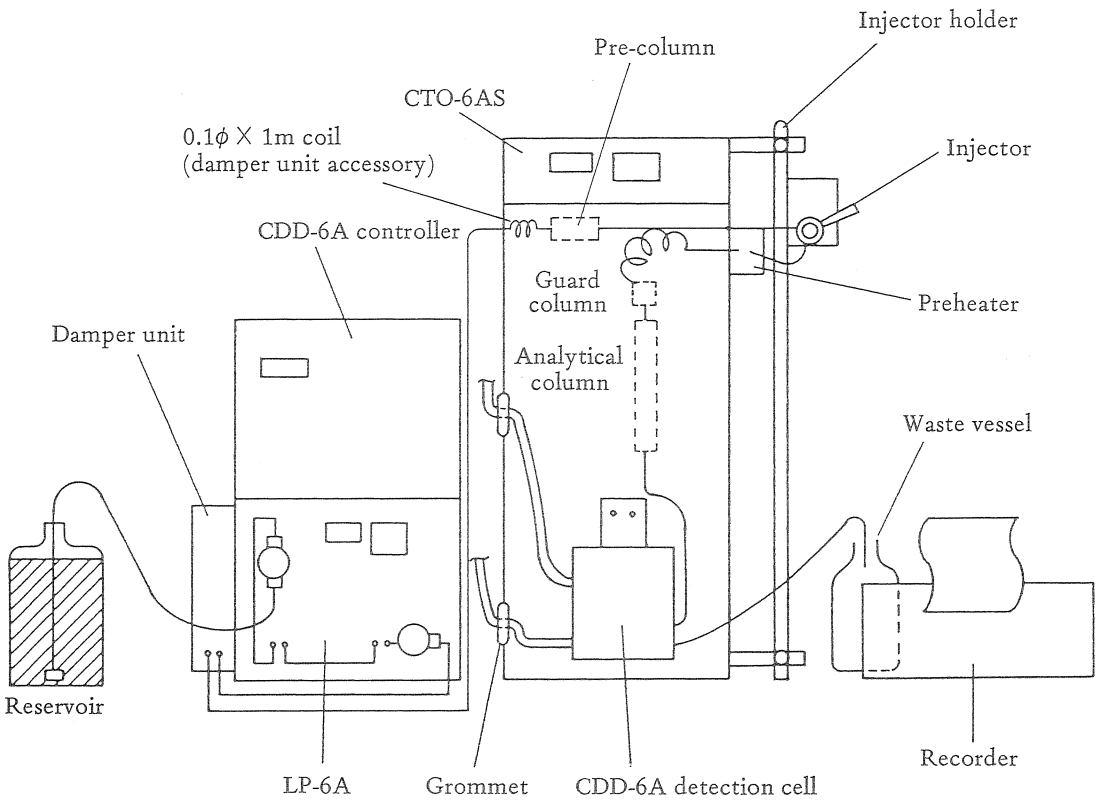


Fig. 1 HIC-6A Connection Diagram

The flow cell tubing is connected as shown in Fig. 1. Before connecting the flow cell tubing, be sure to read the attached "New Connector Instruction Manual" together with "HIC-6A Instruction Manual" carefully. Do not connect the column here; connect the detection cell directly with the preheat coil in the supplied white pipe cover.

There are two SUS pipes (0.1mm I.D.) included with the damper unit accessory. Installed the 2m length pipe in the damper unit and the 1m length pipe in the column oven.

Cover the pipe from the outlet of the damper unit to the coil in the CTO-6AS with the accessory white pipe cover.

Connect two cables from the detection cell to the rear of the CDD-6A controller through the upper and lower holes at the left side of the column oven (CTO-6AS). In this instance, attach grommets to the cable holes. Fix the detection cell to the column mounting plate with the clip buttons so that it is located at the center of the CTO-6AS base.

Connect the drain pipe from the detection cell to the back pressure pipe through a hole at the right bottom of the CTO-6AS, then introduce it into the drain vessel.

If the backpressure pipe is inadvertently touched or subjected to vibration noise may appear on the baseline. Secure the pipe to the side of the CTO-6AS oven with the clamp provided. (See Fig. 2.)

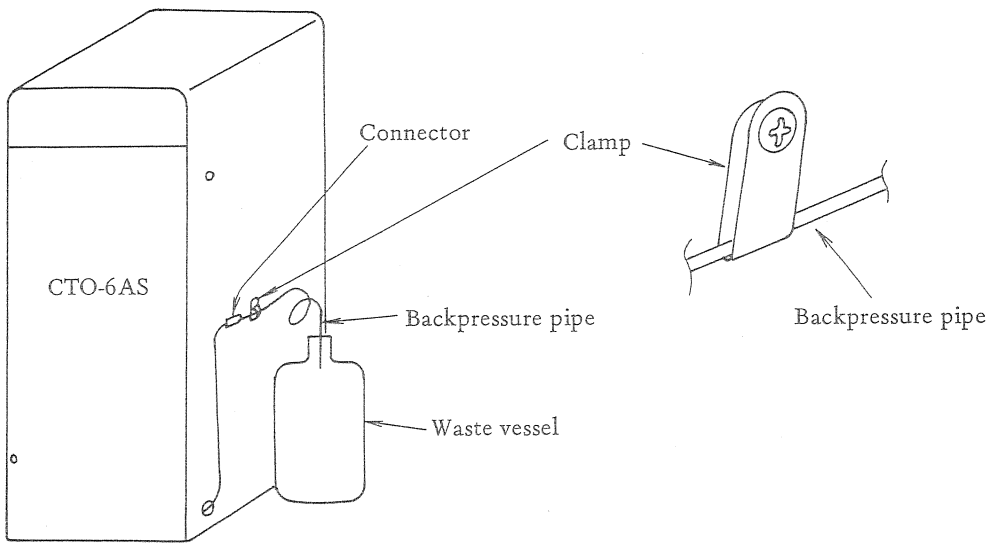


Fig. 2

When the data processor is used, connect the (+) terminal to AUX terminal. When the recorder is used, connect the (+) terminal to the 1mV terminal.

(2) Flow cell cleaning

The liquid feed unit and flow cell parts have been already cleaned in production, but are likely to be contaminated during storage or transportation. Clean the flow cell system to maintain it stability during analysis.

A nitric acid solution and isopropyl alcohol are used for cleaning the cell. Do not mix them at any time, including in the waste solutions, as they generate low flash point substances when mixed resulting in spontaneous combustion at room temp.

Prepare the following four solutions:

(1)	Distilled water (or water for HPLC)	1ℓ
(2)	Isoprophyl alcohol	100mℓ
(3)	1N nitric acid solution	100mℓ
(4)	0.1% EDTA·2Na (ethylenediamine-tetraacetic acid disodium salt) solution	200mℓ

Prepare 0.1% EDTA·2Na solution using EDTA·2Na supplied with this detector. Prepare 1N nitric acid solution by diluting commercial 1N nitric acid or conc. nitric acid.

Operate the liquid feed unit and feed the solutions at a rate of 1mℓ/min. in the following order. Set the upper limit pressure to 250kg/cm. For detailed operation, refer to the "Instruction Manual for Liquid Pump Unit".

1. Distilled water ..... 10 min. ..... Check the tubing for liquid leaks.
2. Isopropyl alcohol ..... 60 min.
3. Distilled water ..... 30 min.
4. 1N nitric acid solution ..... 10 min. } Dispose of separately from (2) and (3) above.
5. Distilled water ..... 30 min. }
6. 0.1% EDTA.2Na ..... 120 min.
7. Distilled water ..... 30 min.

(3) Mobile phase liquid feed

After cleaning the flow cell, feed the mobile liquid at 1.5mℓ/min. for 20 minutes to replace the flow cell with the mobile phase.

Note: As the mobile liquid is relatively quick in developing microorganisms, do not use the same solution for more than three days.

(4) Column connection

Stop the liquid pump unit, connect the column across the preheat coil and detection cell, then feed the mobile phase at the analytical rate.

(5) Startup and inspection

1) Detection cell, column oven and preheater temperature setting

Temperature combinations	Column oven and preheater temperatures	Detection cell temperature
No. 1	30°C	33°C
No. 2	35°C	38°C
No. 3	40°C	43°C
No. 4	45°C	48°C

Select the desired detection cell and column oven temperatures from the combinations No. 1 ~ 4 as above, depending on the ambient temperature. (It is recommended to select the conbination No. 3 (40°C ~ 43°C) at the ambient temperature of 10°C ~ 30°C, the combination No. 4 above 30°C.)

The Shimadzu preheater is automatically set to the same temperature as the CTO-6AS.

2) To start the temperature control, turn ON the POWER switch of the CDD-6A, and the POWER switch and OVEN switch of the CTO-6AS column oven.

After POWER is ON, the CDD-6A automatically selects the following settings and starts the temperature control according to the temperature settings. Therefore after POWER is ON, no further operations are required unless the initial settings are changed. Initial settings GAIN: 10, RANGE: 1, POLARITY: +, RESPONSE: STD, DISPLAY: CONDUCTIVITY.

(6) Baseline recording and standard sample analysis

The baseline is stabilized about 2 hours after starting temperature control. Then, record the baseline, inject the standard sample, and examine the separation.

The relation between baseline noise and mobile phase in Shimadzu Shim-pack columns is as represented below.

Analysis	Column	Mobile phase	Conductivity of mobile phase [μScm^{-1}]	Baseline noise [μScm^{-1}]
Anion analysis	Shim-pack IC-A1	2.5mM phthalic acid 2.4mM Tris (hydroxymethyl) aminomethane (pH 4.0)	about 250	0.004
		1mM p-hydroxybenzonic acid 1.1mM diethylethanolamine (pH 7.9)	about 110	0.003
	Shim-pack IC-A2	0.75mM potassium hydrogen phthalate (ph 4.2)	about 130	0.002
Monovalent cation analysis	Shim-pack IC-C1	5mM nitric acid	about 2500	0.06
Divalent cation analysis		4mM tartaric acid 2mM ethylenediamine	about 800	0.01

(Conductivity of the mobile phase and the baseline noise are measured at an oven temperature of 40°C and a detection cell temperature of 43°C.)

The separation of standard samples is measured at the following sensitivity. (The amount of samples injected is 20$\mu\ell$.)

	Measuring sensitivity
Anion analysis	Gain: 1μScm^{-1}, Range: 4
Monovalant cation analysis	Gain: 10μScm^{-1}, Range: 4
Divalent cation analysis	Gain: 1μScm^{-1}, Range: 4

The ions in the standard samples and the concentration are as shown below. All ions need not be mixed together. Select the required ions.

Anion standard sample

* PO_4^{3-}	30ppm
$C\ell^-$	10ppm
NO_2^-	15ppm
Br^-	10ppm
NO_3^-	30ppm
SO_4^{2-}	40ppm
* (F^-	5ppm)

* In analysis at pH of 4.0, F^- and PO_4^{3-} ($H_2PO_4^-$) cannot be separated as their peaks appear at the same points.

Monovalent cation standard solution

Li^+	5ppm
Na^+	10ppm
NH_4^+	10ppm
K^+	20ppm
Rb^+	30ppm
Cs^+	40ppm

Divalent cation standard solution

Mg^{2+}	10ppm
Ca^{2+}	10ppm
Sr^{2+}	20ppm
Ba^{2+}	30ppm

Note: Use sodium salts and potassium salts as the anion standard samples, and chloride and nitrate as the cation standard samples. Other salts are likely to precipitate depending on the ion combinations.

6.2 Instructions after Analysis

(1) Instructions in stopping liquid pump and temperature control units (column oven, preheater and cell)

If the liquid pump is stopped while the flow cell is at high temperature, the buffer mobile phase may crystalize, clogging the flow cell. Therefore after analysis, continue the liquid pump for about 20 ~ 30 minutes after the column oven and detector POWER switches are turned OFF.

When the CTO-6AS Column Oven is used, open the oven door first after operation. Then, the fan keeps rotating to accelerate cooling the oven. After the oven is cooled down, turn the oven POWER switch OFF.

(2) Instruction in suspending analysis for one day or more

To protect the flow cell and column, reduce the column temperature and stop the liquid pump. Remove the column from the flow cell, then stopper the column ends.

Next, short the flow cell across the column, and replace the flow paths of the liquid pump and flow cell with the distilled water. The amount of distilled water is as much as 100mℓ.

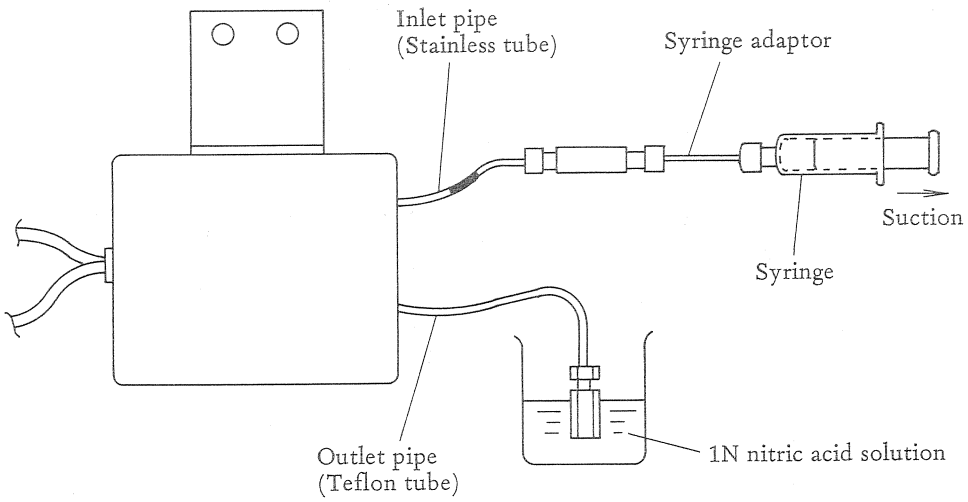
The column can be sealed and stored filled with the mobile phase. If the column is not used for one month or more, it is recommended that the storage liquid contain an antimicrobial agent (e.g. 0.01% sodium azide).

7. Maintenance

7.1 Cell Cleaning

Contaminated electrodes in the cell do not allow stable analysis. When the cell is used after long storage, or exhibits baseline noise, clean it in the following procedures.

(1) Draw 1N nitric acid solution into the syringe, and feed a 5mℓ portion into the cell as shown below.



As the syringe cannot draw a total amount of 5mℓ once, draw 1 ~ 2mℓ portions several times. Discard the nitric acid solution into the drain beaker each time it is drawn into the syringe.

(2) Washing

Place distilled water in the beaker, and feed a 10mℓ portion in the same manner as above.

(3) Connect the cell inlet pipe to the liquid feed pump outlet, and feed the liquid at a rate of 5mℓ/min for about 10 minutes. The liquid to be fed is either the mobile phase used in the ion-exchange chromatography or distilled water.

(4) Separate the cell from the pump and restore the flow cell.

7.2 Liquid Leakage

The cell is designed to withstand pressures up to 30kgf/cm^2, with freedom from liquid leakage in the normal conditions. If leakage occurs due to clogging in the outlet flow line, the baseline will become unstable, and the liquid leaks from the small cover at the bottom of the cell. Should this occur, turn the POWER switch OFF, and contact our branch office or agent.

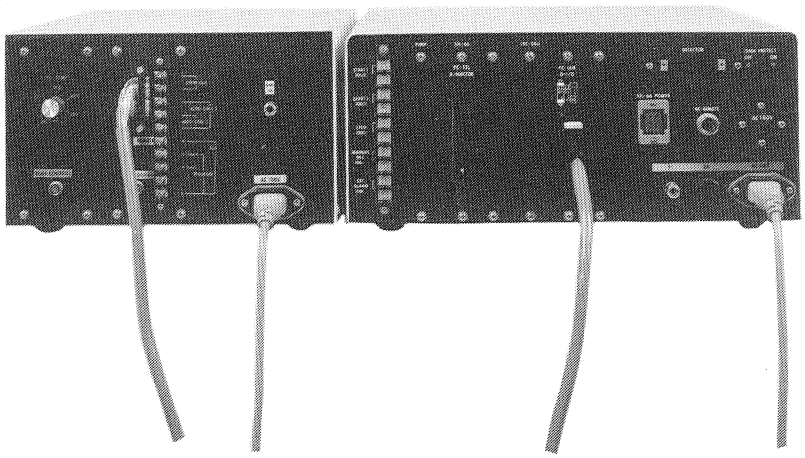
8. Interface with SCL-6A

When this instrument is interfaced with the SCL-6A System Controller, polarity, gain and auto zero can be controlled from the SCL-6A.

The following components are required for interface.

1. Digital I/O (PC-16) P/N 221-21193-91
2. PC-16N remote cable P/N 228-17459-91

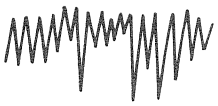
For operation of the SCL-6A, refer to the attached "SCL-6A Instruction Manual".

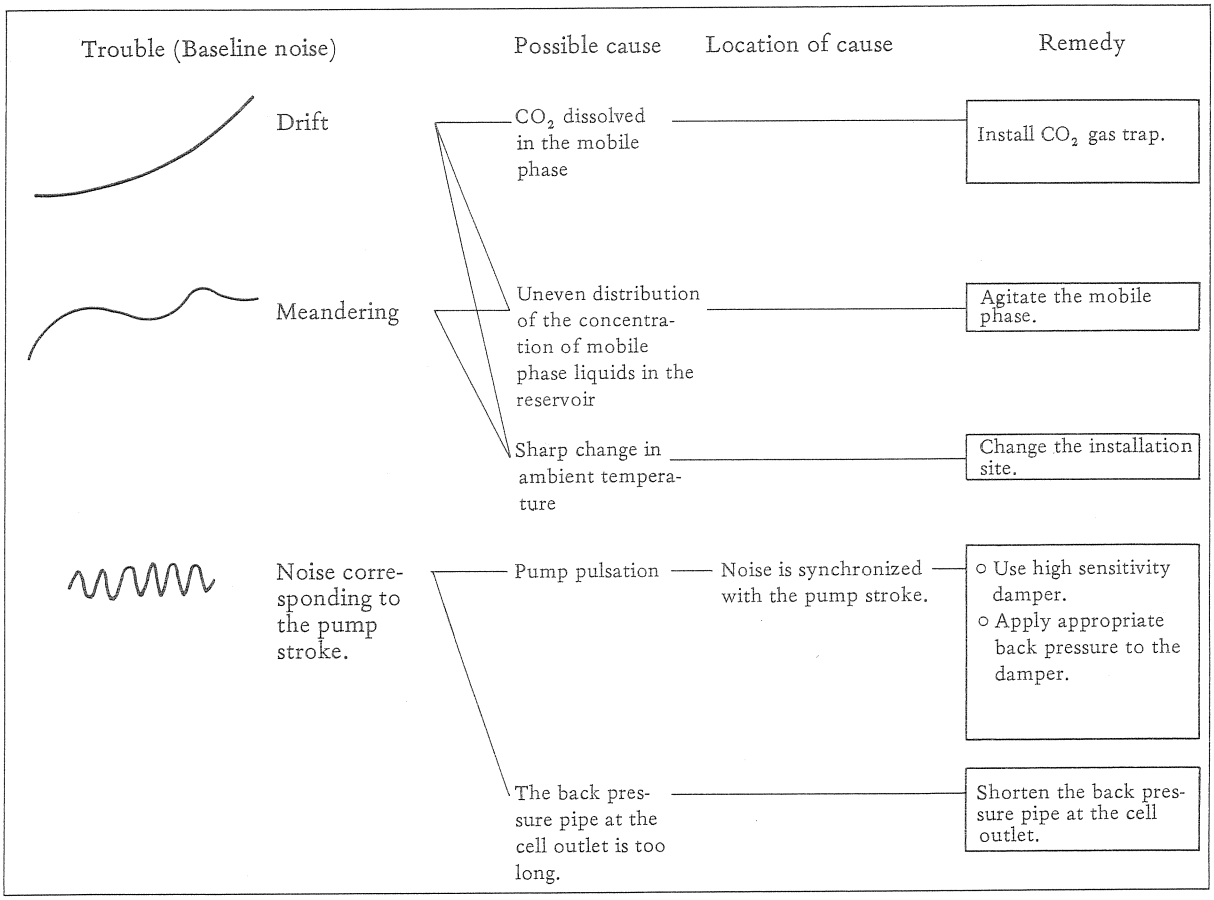


Rear View of CDD-6A Rear View of SCL-6A

9. Troubleshooting

If trouble occurs, locate the cause and troubleshoot as follows.

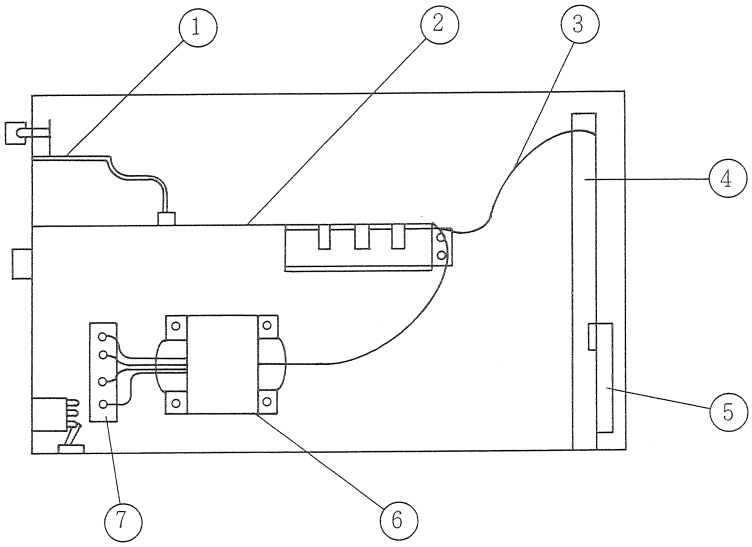
Trouble (Baseline noise)	Possible cause	Location of cause	Remedy
 Transient spiking noise	Bubbles flowing through the cell	Bubbles flow through Teflon pipe at the cell outlet.	○ Extend the back pressure pipe at the cell outlet. ○ Degas the mobile phase.
 Sawtooth noise	Bubbles trapped in the cell	Bubbles flow when sealing and unsealing the backpressure pipe by fingers.	○ Feed water → methanol → water from the cell inlet with the syringe and remove bubbles. ○ Degas the mobile phase.
 Continuous spiking noise			
 Meandering	Bubbles in the pump head	○ Bubbles flow through the suction pipe. ○ Outlet pressure is not constant.	○ Clean or replace suction filter. ○ Degas the mobile phase.
	Plunger seal worn	○ Liquid leaks from the rear of pump head. ○ Outlet pressure is not constant.	Replace plunger seal.
	Check valve contaminated	Outlet pressure is not constant.	Clean or replace check valve.
	Liquid leakage from the flow cell	Liquid leaks from the joints.	Tighten or replace joints.
 Excessive noise	Cell and flow cell contaminated		Clean the cell and flow cell.
 Meandering	Column contaminated		○ Replace the column. ○ Clean the column by feeding the mobile phase 10 times as concentrated as the original.
 Drift			
 Small, irregular peaks with noise	Inpurities in the mobile phase.		○ Reprepare the mobile phase. ○ Use clean ion exchange liquid. ○ Filter the mobile phase.
 Meandering			



Trouble	Possible cause	Remedy
Data processor (Chromatopac) area value does not increase with concentration.	Peak is so large that the signal is saturated. The signal is saturated beyond the following ranges depending on the gain settings. 0.1μScm^{-1}: about 60μScm^{-1} 1μScm^{-1}: about 600μScm^{-1} 10μScm^{-1}: about 6000μScm^{-1}	Set the front panel gain to lower sensitivity.
"ZERO OVER" lamp lights and zero adjustment is ineffective.	Conductivity of eluent is too high. Circuit malfunction	The auto zero adjustment range of this instrument is up to 4000μScm^{-1} at the gain setting of 0.1 or 1μScm^{-1}, and to approx. 9000μScm^{-1} at 10μScm^{-1}. When the eluent ranges from 4000μScm^{-1} to 9000μScm^{-1}, set the gain to to 10μScm^{-1}.

10. Inner Structure

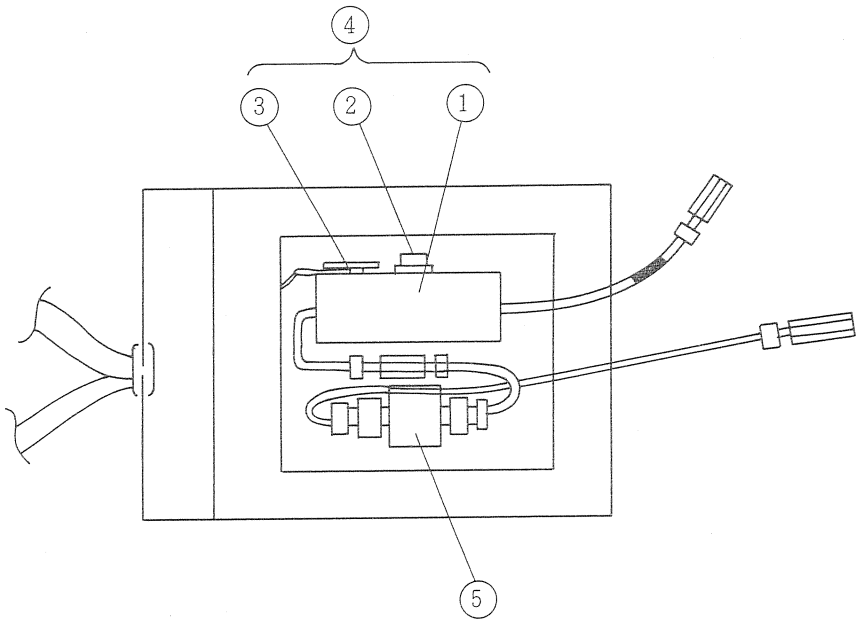
10.1 Inner Structure of Controller



Reference No.	Description	Function	Parts No.
①	PB-4CD ASSY	Contains the cell temperature control circuit.	228-17230-91
②	PB-1CD ASSY	Contains the signal processing circuit.	228-16400-91
③	Flat cable ASSY	Used to connect PB-1–PB-2–PB-3.	228-17236-91
④	Key board ASSY	Contains PC boards for the front panel operation switches.	228-17601-91
⑤	PB-3CD ASSY	Contains the digital meter control circuit.	228-16404-91
⑥	Power transformer		228-17604-91 (for 100V) 228-17604-92 (for 220V)
⑦	Terminal board	Selects AC voltage to 100V or 115V, or to 220V or 240V.	071-30054

10.2 Inner Structure of Cell

Apart from emergency, if trouble occurs in the cell, contact our branch office, sales office or agent.



Turn POWER switch OFF, remove the cell outer cover, and take out the sponge sheet. Remove the inner case by loosening four mounting screws. Then, you can see the cell interior as shown above. Parts No. and names are as follows.

Reference No.	Description	Parts No.	Remark
(1)	Heat exchanger ASSY	228-17100-96	
(2)	TR, 2SC234G-O	060-27234-12	
(3)	Thermistor, 103AT2	061-74328	
(4)	Temperature control block ASSY	228-17656-91	Contains (1), (2) & (3) and cable.
(5)	Cell semi ASSY	228-17653-91	Contains the temperature block relay Teflon pipe and outlet Teflon pipe.

11. Parts List

Detection Cell

No.	Description	Parts No.	Remark
1	Cell semi ASSY	228 - 17653 - 91	Refer to 10.2.
2	Heat exchanger ASSY	228 - 17100 - 91	
3	TR, 2SD234G-O	060 - 27234 - 12	
4	Thermistor 103AT2	061 - 74328	
5	Temperature control block	228 - 17656 - 91	

Electrical System

No.	Description	Parts No.	Remark	
1	SW, paddle, M-20022N-2-W	064 - 14086 - 01	POWER switch	
2	Filter, FN322-3/01	075 - 00056	Power line filter	
3	Fuse, F7161 1.5A	072 - 01024 - 01	For 100V and 115V	
	Fuse, ULCS-61M 1.0A	072 - 01660 - 13	For 220V ~ 240V	
4	ZNR, ERZ-C10DK201	061 - 80703 - 12	For 100V and 115V	Surge absorber
	ZNR, ERZ-C10DK431	061 - 80703 - 17	For 220V ~ 240V	
5	Transformer ASSY	228 - 17604 - 91	For 100V and 115V	Power transformer
	Transformer ASSY	228 - 17604 - 92	For 220V ~ 240V	
6	Flat cable ASSY	228 - 17236 - 91		
7	PC board PB-1CD ASSY	228 - 16400 - 91	Complete PC board for signal processing	
8	ic, TMP8085AP	075 - 33852	CPU, M28	PB-1 components
9	〃, TC5092AP	075 - 33330	ADC, M29	
10	〃, M5L8225 AP-5	075 - 30305 - 01	P·I/O, M30	
11	〃, M5L8155P	075 - 30304 - 01	M34	
12	〃, AD DAC80D-CBI-V	075 - 29196 - 06	M20	
13	〃, DAC0808 LCN	075 - 29066 - 03	M6, M36	
14	〃, AD7542JN	075 - 23641 - 01	M39	
15	〃, AD630JN	075 - 23670 - 01	M27	
16	〃, TL7705P	075 - 21217 - 02	M17	
17	〃, AD OP-07CN	075 - 23652 - 02		
18	〃, TL-072 CP	075 - 21208		
19	〃, M54563P	075 - 30995	M23	
20	〃, MC74HC373N	075 - 22203 - 73	M33, M35	
21	〃, MC74HC27N	075 - 22200 - 27	M22, M24	

No.	Description	Parts No.	Remark
22	ic, UPC14305H	075 - 31131	Q2
23	〃, UPC14315H	075 - 31134	Q3
24	〃, MC7915C-T	075 - 22026	Q4
25	PROM, CDD-6A ASSY	228 - 17597 - 91	ROM1
26	PROM, CDD-6A ASSY	228 - 17597 - 92	ROM2
27	ic, HI-201-05	075 - 27809 - 01	M1～M3, M12
28	Diode, 3BZ61	060 - 15017	D1, D2
29	〃 , 1BZ61	060 - 15011	D4～D6, D13
30	〃 , 2B4DM	060 - 13812	D3
31	〃 , 05AZ5.1x	060 - 14319 - 23	D7, D8
32	〃 , 05AZ5.6x	060 - 14319 - 26	D9, D10
33	〃 , 1SZ47	060 - 14074 - 04	D11
34	Transistor, 2SA1012-O	060 - 21012 - 01	Q1
35	〃 , 2SA1015-O	060 - 21015 - 01	Q6
36	〃 , 2SC1815-O	060 - 25815 - 01	Q5, Q7
37	Reed relay, PG1C-05D	065 - 63061 - 17	RE1
38	〃 , RRF51A05D	065 - 63147 - 01	RE2～RE12
39	PC board, PB-4CD ASSY	228 - 17230 - 91	Complete PC board for cell temperature control
40	ic, AD-0P-07CN	070 - 23652 - 02	A1～A3 (PB-4 components)
41	SW, RP7×1-1-6AG60°	064 - 73443	Temperature setting switch (PB-4 components)
42	Key board ASSY	228 - 17601 - 91	
43	PB-2CD ASSY	228 - 16403 - 91	PC board for key board (Key board ASSY components)
44	SW, FES-360-001	064 - 48044	Key switch (Key board ASSY components)
45	Key top, N860-0268×155	221 - 24297 - 03	Key top (Key board ASSY components)
46	LED, TLG-205-5	061 - 78907 - 02	Light-emitting diode for display (Key board ASSY components)
47	PB-3CD ASSY	228 - 16406 - 91	PC board for digital panel meter control
48	Indicator, SL-1170	061 - 78564 - 31	7-segment light-emitting diode
49	LED, TLG-205	061 - 78067 - 02	Light-emitting diode (green)
50	LED, TLR-205	061 - 78067 - 01	Light-emitting diode (red)
51	ic, MC74HC373N	075 - 22203 - 73	M3, M4
52	ic, TD62003P	075 - 33999 - 03	M5

New Tubing Connector Type for use in the Shimadzu High Performance Liquid Chromatograph

1. Introduction

The LC-6A modular HPLC system as well as the subsequent product series will employ a new type of tubing connector. The new connectors are incompatible with the former Shimadzu connectors. Prior to installation of tubing, carefully read through this manual to ensure proper usage of the connectors.

A tag describing usage of the new connector is supplied as a standard accessory of each module. On installation, place the tag where it will be visible, preferably the front panel of a module.

> CAUTION
>
> The new connector type used in the equipment is not compatible with our former connectors. Never use the former type of fittings to connect tubing to the equipment. Also avoid using any parts (such as front ferrule 1.6FF or bushing 1.6B) of the conventional connectors.

[Connector for Shimadzu-made and Du Pont-made columns]

1. The former connector type is used in Shimadzu-made columns.

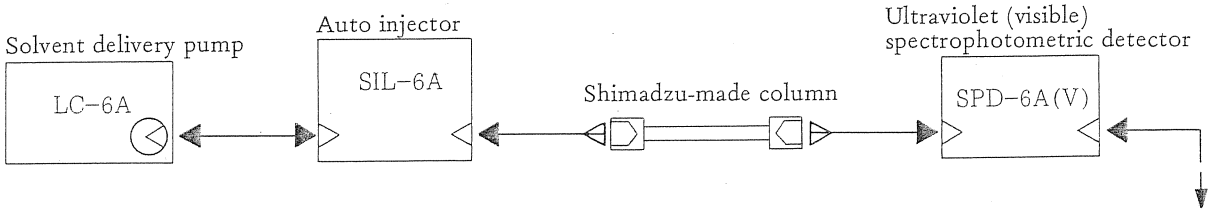
   For connection of the column, use the former connectors which are supplied as a standard accessory of the LC-6A solvent delivery pump.

2. New connector type can be used for the Du Pont-made columns (such as the Zorbax series) when they are employed for our products.

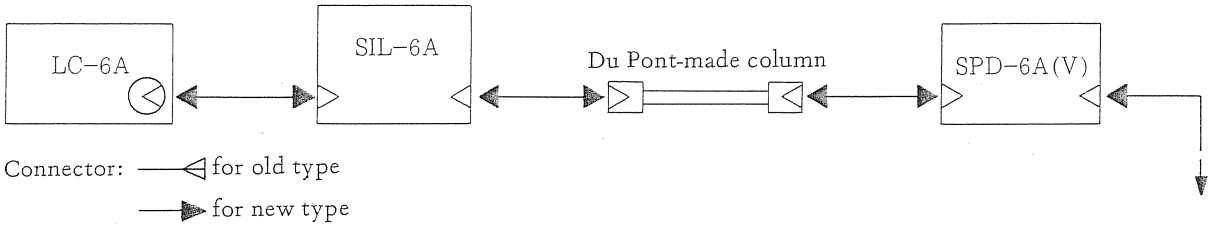
2. Connection of LC System

The following are examples of connection of LC-6A series

① When a Shimadzu-made column is used:



② When a Du Pont-made column is used:



3. How to Mount Connectors

(1) New connector type

There are two kinds of connection ports. Mount the appropriate fitting in the following manner:

A. Female connection port

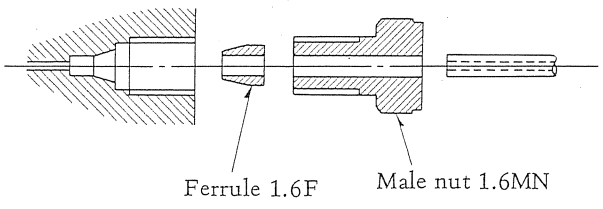


Fig. 1

B. Male connection port

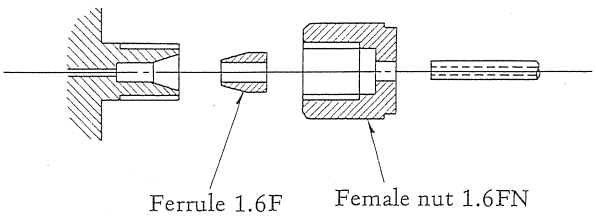


Fig. 2

(2) Former connector type

When Shimadzu-made columns or similar hardware utilizing the former type connectors are employed, use connector parts of the former type (dual ferrule in reverse configuration). Refer to the illustrations below for proper mounting of both types of connectors.

A. Female connection port

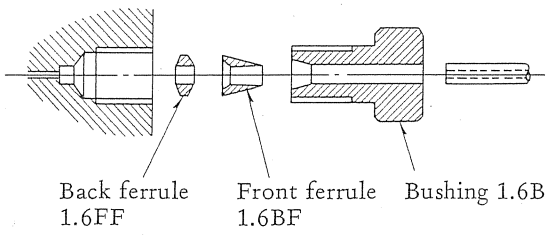


Fig. 3

B. Male connection port

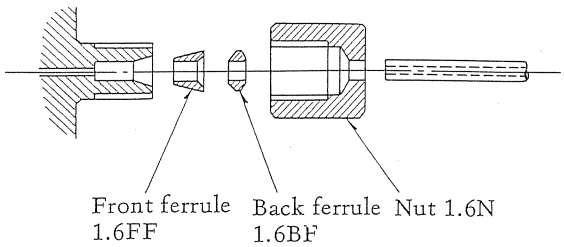
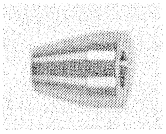
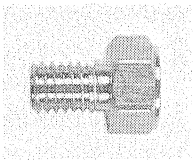
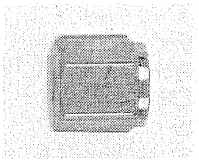
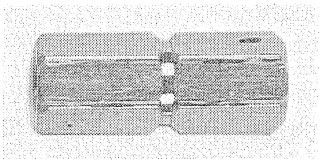
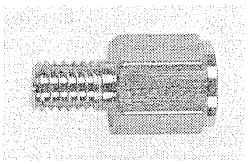
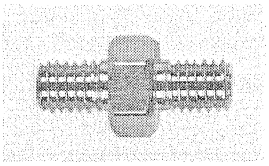
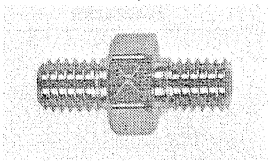
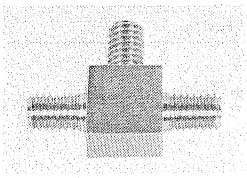
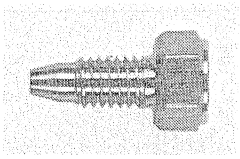
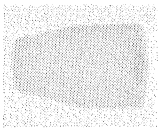
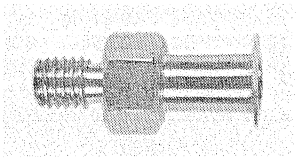


Fig. 4

4. Parts for New Connectors

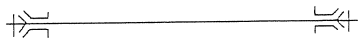
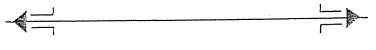
The table below lists major parts of the new connector. These are not compatible with parts of the conventional connector, though union 1.6U, union 1.6U(X) and male tee 1.6MT are the same as the former ones.

Part Name	Part No.	Part Name	Part No.
① Ferrule 1.6F 	228-16000	② Male nut 1.6MN 	228-16001
③ Female nut 1.6FN 	228-16002	④ Coupling 1.6C 	228-16004-03
⑤ Adaptor 1.6A 	228-16005-03	⑥ Union 1.6U 	228-00034
⑦ Union 1.6U(X) (Zero dead volume) 	228-01356	⑧ Male tee 1.6MT 	228-00126
⑨ Plug 1.6P 	228-16006	⑩ Teflon ferrule 1.6F-T 	228-16007
⑪ Needle adaptor (M) 	228-00379-01		

The following parts of the new connector are always kept in our inventory stock and are available if ordered from our branch office.

Part Name	Part No.	Remarks
Ferrule 1.6F	228-16000-84	3 pcs in a set
Male nut 1.6MN	228-16001	1 pcs in a set
Female nut 1.6FN	228-16002	1 pcs in a set
Coupling 1.6C	228-16004-03	1 pc
Adaptor 1.6A	228-16005-03	1 pc
Union 1.6U	228-00034	1 pc
Union 1.6U(X)	228-01356	1 pc
Male tee 1.6MT	228-00126	1 pc
Plug 1.6P	228-16006	1 pc
Teflon ferrule 1.6F-T	228-16007-84	5 pcs in a set
Coupling 1.6C ASSY	228-16051-91	Coupling 1.6C 1 pc Ferrule 1.6F 2 pcs } in a set Male nut 1.6MN 2 pcs
Adaptor 1.6A ASSY	228-16052-91	Adaptor 1.6A 1 pc Ferrule 1.6F 2 pcs Male nut 1.6MN 1 pc } in a set Female nut 1.6FN 1 pc
Union 1.6U (new type) ASSY	228-16053-91	Union 1.6U 1 pc Ferrule 1.6F 2 pcs } in a set Female nut 1.6FN 2 pcs
Union 1.6U(X) (new type) ASSY	228-16054-91	Union 1.6U(X) 1 pc (Zero dead volume) Ferrule 1.6F 2 pcs } in a set Female nut 1.6FN 2 pcs
Male tee 1.6MT (new type) ASSY	228-16055-91	Male tee 1.6MT 1 pc Ferrule 1.6F 3 pcs } in a set Female nut 1.6FN 3 pcs

Tubing kits (including 25cm long tubing) are also available if ordered.

Part Name	Part No.	Remarks
Tubing kit A ASSY (For former part to former part connection)	228-16056-91	 Tubing 1.6φ OD X 0.3φ ID X 25cm 1 pc Bushing 1.6B 2 pcs Front ferrule 1.6FF 2 pcs } in a set Back ferrule 1.6BF 2 pcs
Tubing kit B ASSY (For former part to new part connection)	228-16057-91	 Tubing 1.6φ ODX0.3φ IDX25cm 1 pc Bushing 1.6B 1 pc Front ferrule 1.6FF 1 pc Back ferrule 1.6BF 1 pc } in a set Male nut 1.6MN 1 pc Ferrule 1.6F 1 pc
Tubing kit C ASSY (For new part to new part connection)	228-16058-91	 Tubing 1.6φ ODX0.3φ IDX25cm 1 pc Male nut 1.6MN 2 pcs } in a set Ferrule 1.6F 2 pcs

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