211-78001E
Jun. 2018

SHIMADZU OPTICAL EMISSION SPECTROMETER

PDA-7000

INSTRUCTION MANUAL

Read the instruction manual thoroughly before you use the product.
Keep this instruction manual for future reference.

* Refer to the following document for detailed operation of software.

Data Processing Software PDA for Windows Operatioin Manual
P/N 211-54796

Data Processing Software PDA for Windows Installation Manual
P/N 211-78114

SHIMADZU CORPORATION
ANALYTICAL & MEASURING INSTRUMENTS DIVISION

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Introduction

Read this Instruction Manual thoroughly before using the product.

Thank you for purchasing Shimadzu optical emission spectrometer PDA-7000.

PDA-7000 is a PDA photoelectric-photometric optical emission spectrometer which simultaneously and quickly carries out analyses of multi-elements in metal with a high precision. The photoelectric-photometric emission spectrometer, which is adopted in a wide range of fields, is an essential instrument for quality control.

This manual describes the installation, operation, usage cautions, accessories and options for this product. Read this manual thoroughly before using the product and operate the product in accordance with the instructions in this manual.

Also, keep this manual for future reference.

IMPORTANT

- If the user or usage location changes, ensure that this Instruction Manual is always kept together with the product.
- If this manual or a product warning label is lost or damaged, immediately contact your Shimadzu representative to request a replacement.
- To ensure safe operation, read all Safety Instructions before using the product.
- To ensure safe operation, contact your Shimadzu representative if product installation, adjustment, or re-installation (after the product is moved) is required.

Notice

- Information in this manual is subject to change without notice and does not represent a commitment on the part of the vendor.
- Any errors or omissions which may have occurred in this manual despite the utmost care taken in its production will be corrected as soon as possible, although not necessarily immediately after detection.
- All rights are reserved, including those to reproduce this manual or parts thereof in any form without permission in writing from Shimadzu Corporation.
- Microsoft and Windows are registered trademarks of Microsoft Corporation in the United States and/or other countries. Other company names and product names mentioned in this manual are trademarks or registered trademarks of their respective companies.
  The TM and ® symbols are omitted in this manual.

Indications Used in This Manual

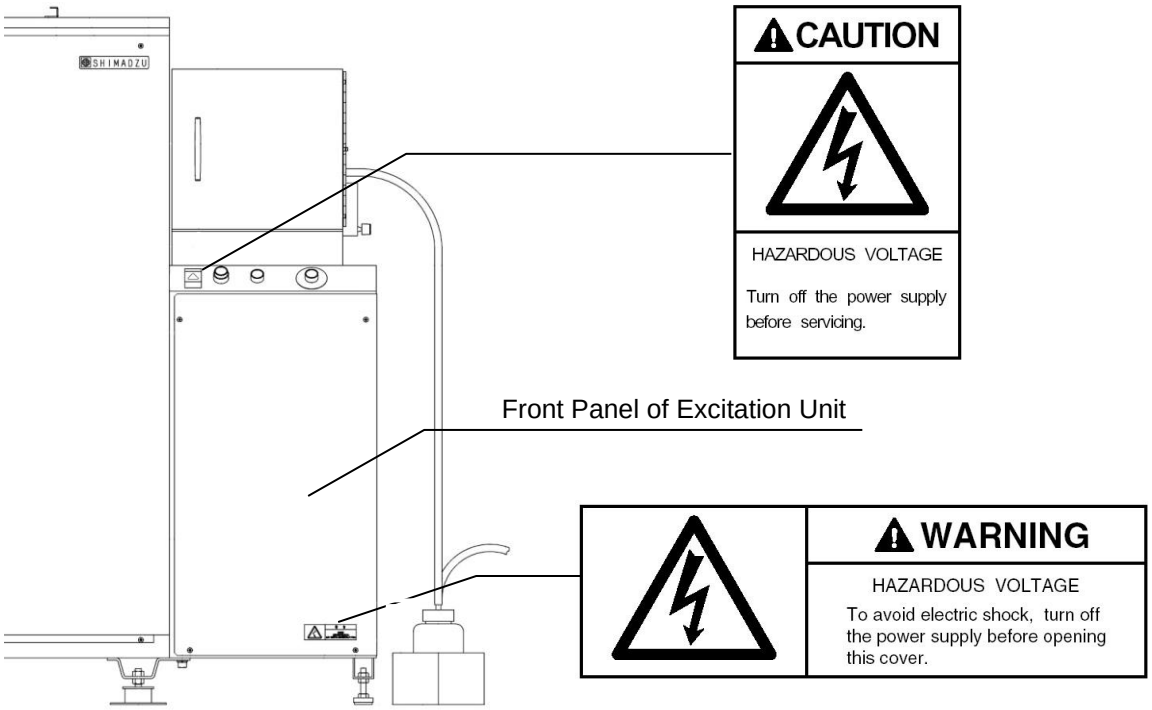
Dangers, Warnings, Cautions, and Notes are indicated using the following conventions:

Note

Dangers, Warnings, Cautions, and Notes are indicated using the following conventions:

Warning	**Indicates a potentially hazardous situation which, if not avoided, could result in serious injury or possibly death.**
Caution	**Indicates a potentially hazardous situation which, if not avoided, may result in minor to moderate injury, or equipment damage.**
Note	**Emphasizes additional information that is provided to ensure the proper use of this product.**

Warning Labels



Warning

Electric Shock

Before removing the excitation unit front panel, be sure to turn OFF the power of the excitation source unit. Never remove other panels.

Safety Instructions

To ensure safe product operation, read these important safety instructions carefully before use and follow all DANGER, WARNING and CAUTION instructions given in this section.

Installation Site

Warning

Do not operate PDA-7000 under explosive and/or corrosive atmosphere.

Note

When installing PDA-7000 near apparatus generating a ferromagnetic field such as an electric arc furnace, contact your Shimadzu representative.

Installation

To ensure safe operation, contact your Shimadzu representative if product installation, adjustment, or re-installation (after the product is moved) is required.

Operation

PDA-7000 generates high voltage based on the measurement principles. If it is correctly used, no problem will occur. If not, it may cause an accident and damage. For safety operation and dangerous accident prevention, strictly observe the following precautions.

1. Precautions for operator safety and for handling the instrument

Warning

PDA-7000 may cause radio disturbance during discharge. Personnel with pacemakers should be especially careful during discharge.

Caution

Electric Shock

Never start the discharge without correctly setting and holding the sample on the spark stand.

Caution

- **Thoroughly read the instruction manual to strictly observe it. If you have any question, contact your Shimadzu representative.**
- **Keep this instruction manual near the instrument for quick reference of the operation procedures and safety precautions anytime necessary.**

Note

The contents of the hard disk of the personal computer may be lost as the result of an unforeseen accident. Always create a backup to protect critical data from such accidents.

2. Restriction of operators

Warning

The inside of the instrument should only be repaired by personnel specially trained by Shimadzu Corporation since it is dangerous. Do not disassemble or modify the instrument without permission as it may compromise the safety of the instrument.

Caution

Use of the instrument is allowed only to personnel who have understood the safety precautions well and have been well-trained for the operating procedure.
It is a must to establish a system by which no one other than the operator above is allowed to use it.

In an emergency

Warning

- **<u>In case of leakage of argon gas</u>**
  If it is left, lack of oxygen may occur. Immediately ventilate the working area and check for gas piping.

- **<u>In case of abnormal temperature raise of the spectrometer unit</u>**
  The room temperature should be kept within the range of 10 - 28°C. If it exceeds 30°C, the temperature of the spectrometer unit may raise abnormally.
  If the temperature of the spectrometer unit exceeds 42°C, the buzzer at the temperature alarm will give a warning sound. In that case, turn OFF the AIRCON switch on the switch panel and make sure the room temperature is within the range of 10 - 28°C. Then, wait until the temperature inside the spectrometer goes down to the room temperature, and turn ON the AIRCON switch again.
  If the buzzer gives a warning sound even after that, turn OFF the AIRCON switch and contact your Shimadzu representative.

- **<u>Safety measure for other emergency occurrences</u>**
  If an unpredictable happening like an earthquake, fire, etc. occurs, first press the emergency stop switch, and then turn OFF all the power of the distribution panel. After this, each staff must carefully take an appropriate action to maintain his/her safety.

During a power outage

In case of a power failure, follow the procedure described below.

1. Turn off the MAIN breaker on the switch panel.
2. Turn off the AIRCON, CONSOLE, and SOURCE switches on the switch panel.
3. Turn off the power switch of the vacuum pump.
4. When the power is back, turn on the MAIN breaker on the switch panel.
5. Turn on the AIRCON, CONSOLE, and SOURCE switches on the switch panel.
6. Turn on the power of the vacuum pump.

Handling High-pressure Gas

PDA-7000 requires the use of high-pressure argon gas.

Note that the following points regarding the handling of high-pressure gases in Japan:

1) By law, permission is required to use cylinders containing more than 300m^3.
2) When using gas of a pressure that exceeds 980.665kPa (10kgf / cm^2) inside the equipment at room temperature, the equipment is regarded as high-pressure gas equipment and, by law, notification of use must be submitted.

Refer to your government laws and regulations related to high pressure gas control, liquefied petroleum gas control, general high pressure gas control, and fire defense.

Also, follow the precautions and instructions below for the safe use of high-pressure gas.

Warning

- **Install the gas cylinder in a well-ventilated outdoor location that is not exposed to direct sunlight and convey the gas indoors via a pipe. Regarding liquefied gases in particular, this setup is required by law in Japan.**
- **Ensure that the temperature of the gas cylinder never exceeds 40°C. Also, ensure that there are no open flames within 2m of the gas cylinder.**
- **Ensure that the location where high-pressure gas is used is well ventilated and, as part of initial inspection, check for gas leaks with soapy water.**
- **Prevent the cylinder from falling or toppling over by securing it with rope or chain. Be sure not to let liquefied-gas cylinders topple over into a horizontal position.**
- **After using the gas, close the cylinder's main valve immediately.**
- **Inspect the functional capability of the pressure gauge at least once every three months.**

Note

Do not use the argon gas cylinder until it is empty. Otherwise, the inside of the cylinder will be contaminated.
For the exchange procedures of the cylinder, refer to “5.13 Replacing argon gas cylinder”

Warranty

Shimadzu provides the following warranty for this product.

1. Period:

Please contact your Shimadzu representative for information about the period of this warranty.

2. Description:

If a product/part failure occurs for reasons attributable to Shimadzu during the warranty period, Shimadzu will repair or replace the product/part free of charge. However, in the case of products which are usually available on the market only for a short time, such as personal computers and their peripherals/parts, Shimadzu may not be able to provide identical replacement products.

3. Limitation of Liability:

(1) In no event will Shimadzu be liable for any lost revenue, profit or data, or for special, indirect, consequential, incidental or punitive damages, however caused regardless of the theory of liability, arising out of or related to the used of or inability to use the product, even if Shimadzu has been advised of the possibility of such damage.

(2) In no event will Shimadzu's liability to you, whether in contract, tort (including negligence), or otherwise, exceed the amount you paid for the product.

4. Exceptions:

Failures caused by the following are excluded from the warranty, even if they occur during the warranty period.

(1) Improper product handling

(2) Repairs or modifications performed by parties other than Shimadzu or Shimadzu designated companies

(3) Product use in combination with hardware or software other than that designated by Shimadzu

(4) Computer viruses leading to device failures and damage to data and software, including the product's basic software

(5) Power failures, including power outages and sudden voltage drops, leading to device failures and damage to data and software, including the product's basic software

(6) Turning OFF the product without following the proper shutdown procedure leading to device failures and damage to data and software, including the product's basic software

(7) Reasons unrelated to the product itself

(8) Product use in harsh environments, such as those subject to high temperatures or humidity levels, corrosive gases, or strong vibrations

(9) Fires, earthquakes, or any other act of nature, contamination by radioactive or hazardous substances, or any other force majeure event, including wars, riots, and crimes

(10) Product movement or transportation after installation

(11) Consumable items
Recording media such as floppy disks and CD-ROMs are considered consumable items.

* If there is a document such as a warranty provided with the product, or there is a separate contract agreed upon that includes warranty conditions, the provisions of those documents shall apply.

After-sales Service and Availability of Replacement Parts

After-Sales Service

If any problem occurs with this product, perform an inspection and take appropriate corrective action as described in this manual's troubleshooting section. If the problem persists, or the symptoms are not covered in the troubleshooting section, contact your Shimadzu representative.

Replacement Parts Availability

Replacement parts for this product will be available for a period of seven (7) years after the product is discontinued. Thereafter, such parts may cease to be available.
Note, however, that the availability of parts not manufactured by Shimadzu shall be determined by the relevant manufacturers.

If Shimadzu receives notice of the discontinuation of units or parts, the necessary quantity for the above period is immediately calculated and secured. However, such units or parts may cease to be available within seven years after the discontinuation of the product, depending on individual manufacturer conditions and on changes in the quantity required.

Note

When contacting your Shimadzu representative, please provide the following information.

(1) Product model
(2) Year/Month/Date of purchase
(3) Version No. (for software)
(4) Detailed explanation of the problem

Disposal Precautions

Dispose of this product using a qualified industrial waste management company, in compliance with the applicable laws in the country where it is used.

Contents

Definition of Terminology

Emission analysis	Quantitative analysis method using the emission spectrometer by measuring the intensity of emission of atomic spectral line obtained by vaporized excitation by means of discharge of the analytical object element contained in a sample.
Photoelectric photometric method	Spectrum intensity measurement method using the photomultiplier
Electrode	Same as “Counter Electrode”. (tungsten electrode) Strictly, the sample is also an electrode, because the discharge is generated in the gap between the counter electrode and the sample. But, in this instruction manual, it means counter electrode unless specially noted.
Pre-burn time	Time set as non-measurement time from discharge start until emission intensity is stabilized
Integral time	Measurement time in case of measurement of emission intensity by a specified-time integration
Argon gas	Gas to be carried around the electrode in order to stabilize discharge and remove influence due to sample history or absorption by oxygen in air
Analysis sample	Sample prepared for measurement
Standard sample	Sample for creating a working curve of which quantitative element content was precisely determined by the chemical analysis method
Set up sample	Sample to be used for calibration of the working curve for a specified time or at every measurement of a specified number of samples

Parts List

Checking of parts

PDA-7000 is provided with the following parts. After unpacking, check that there are no missing parts and no damage is given to each of them.

1 Emission spectrometer, main unit 1 set

*2 Computer 1 set

*3 Monitor 1 set

*4 Printer 1 set

*5 Computer rack 1 set

* Depending upon the instrument specification, items 2 through 5 may not be included.

6 Instruction Manual

No.	P/N	Part name	Q'ty
1	211-78001	PDA-7000 Instruction Manual	1 pc
2	211-54796	Data Processing software PDA for Windows Operation Manual	1 pc
3	211-78114	Data Processing software PDA for Windows Installation Manual	1 pc

7 Standard accessories

No.	P/N	Part name	Q'ty
1	211-74363-02	Gap gauge 4mm	1 pc
2	202-41917	Electrode extraction tool	1 pc
3	211-74362	Electrode D6 x 100	1 pc
4	036-11204	O ring 4D P6	10 pcs
5	211-74663-91	Electrode brush with holder	1 pc
6	017-30159-03	Rotary pump oil MR200 1L	1 can
7	239-07003-08	Molecular sieves 13 x 4 x 8M	1 bottle
8	038-03594-02	Plastic case with lid R20	1 pc
9	201-24629	Lens rotation fitting	1 pc
10	211-51930	Argon gas bubbler	1 pc
11	776-01449	Control gap gauge	1 pc
12	017-30813-01	Silicon grease HIVAC-G	1 pc
13	016-43505	Polypropylene tube 44-PP white	5 m
14	016-31330	Vinyl hose 9x2.5 CL 2MT	5 m
15	035-65503	Half union C1N1/4 x PT1/4	1 pc
16	072-01122	Fuse F-7165 10A 250V	1 pc
17	072-01214-52	Fuse FGB-01 20A 250V	2 pcs
18	062-62104	Lamp H0857	1 pc

Consumables and maintenance parts

No.	P/N	Part name	Replacement interval	Recommended number of parts
1	211-74362	Electrode D6 x 100	*1 5000 emissions	4 pcs
2	036-11204	O-ring 4D P6	*2 1 month	10 pcs
3	042-60424-23	Fan 4710PS-20T-B30-B00	*3 2 - 3 years	3 pcs
4	042-60474-01	Fan 4715MS-10T-B50-B00	2 - 3 years	3 pcs
5	065-80826-06	Relay MY4N AC200V	*4 1 year	1 pc
6	065-60648-11	Relay LY2N DC24V	*4 1 year	5 pcs
7	065-60848-02	Relay LY4N DC24V	*4 1 year	3 pcs
8	211-76668	Dust cup	When damaged	1 pc
9	211-74359-03	*5 Sample plate (hole diameter 12mm)	When damaged	1 pc
10	211-74439	Electrode holder assembly	When damaged	1 pc
11	211-51031	Sample holder assembly	*6 When damaged	1 pc
12	211-74965	*7 Brush	When damaged	1 pc
13	017-30159-03	Rotary pump oil MR-200 1L	6 months	1 can
14	239-07003-08	Molecular sieves 13 x 4 x 8M	1 year	1 bottle
15	017-30813-01	Silicon grease HIVAC-G		1 can

*1 5000 emissions is a typical value. Electrode can be used until its length reduces to 65 mm. (in the case used with automatic electrode cleaning unit, 82mm)

*2 Replacement interval varies depending on the frequency of analysis. 1 month is a typical value.

*3 There are three No.3 Fans used in PDA-7000.
The one used in excitation source unit has to be replaced once every two years.
The other two attached on the right side panel have to be replaced once every three years.

*4 Relays have to be replaced on an annual inspection.

*5 Turn sample plate upside down after every spark stand cleanup.

*6 The lifetime of the sample holder varies depending on how it is used.
For example, if it is used with the flexure of the plate spring around 10mm, the expected lifetime is about 8000-times use. In the case of around 5mm, it is about 24000-times use.

*7 The spare head for brush 211-74663-91.

Repair parts

No.	P/N	Part name	Notes
1	211-52482-91	Cable 15C	Spark stand – Sample holder
2	080-68536-01	Flowmeter F900	Spark stand argon gas purge line
3	211-75818	Flow meter、RK1650	
4	211-74517	Valve V1 Assy	
5	211-74517-01	Valve V2 Assy	
6	040-21333-57	Valve、2400L-B-N1-1/4	
7	016-31330	Vinyl hose 9x2.5 CL (2MT)	Spark stand argon gas exhaust line
8	211-51930	Argon gas bubbler	
9	036-19003-33	O-ring 1A S42	Used in spark stand
10	036-12001	O-ring 1A G25	
11	036-10341-12	O-ring R-LEP12	
12	064-60774-02	Switch PB ABW110-G	START switch
13	064-60776-09	Switch PB ABW201-R	STOP switch
14	066-81601	Switch DPRI-01	Proximity sensor switch Used for stand door and excita-tion unit panel
*1 15	202-46611-01	Lens assembly (Q1)	Condenser lens (quartz)
	205-01583-01	Lens assembly (MgF_2)	Condenser lens (MgF_2)
16	211-74649-91	Ignition coil assembly	
17	211-50467-01	Control gap electrode (Φ2mm)	
18	042-60424-23	Fan 4710PS-20T-B30-B00	
19	042-60934-13	Filter set 9450-M	(Filter set used for fan above)
20	211-74756-91	FET assembly	
21	060-40917-12	Thyristor PK25GB-80	Excitation unit
22	072-06035-11	Fuse 600CF-5	
23	072-01673-13	Fuse 3AG-312.500	
24	065-80826-06	Relay MY4N AC200V	
25	065-60648-11	Relay LY2N DC24V	
26	065-60848-02	Relay LY4N DC24V	
27	211-50828-92	Excitation unit control board HPSG CONT PCB	
28	211-50765-01	Cable 15A	Excitation unit – sample (–)
29	211-50765-02	Cable 15B	Excitation unit – electrode (+)
30	211-50797	Read-out unit power board DC PCB	Read-out unit
31	211-50785	A/D converter board AD PCB	
32	211-50794-92	Control circuit board CHECK PCB	
33	211-50791	Integrator board ITG PCB	
34	211-50788	Negative HV power board HV PCB	
35	211-72137-93	CPU cont. unit	SCSI control box
36	062-62104	Lamp H0857	Fatigue lamp
37	211-66527-91	Pirani tube filament	

38	080-80202-04	Temperature controller E5CN-Q2T-W	Switch panel
39	066-19180-02	Timer H3Y-2 AC200V 3M	
40	072-01122	Fuse F-7165 10A 250V	
41	072-01412-52	Fuse FGB01 20A 250V	
42	042-60474-01	Fan 4715MS-10T-B50-B00	Air conditioning unit
43	078-29503-02	Heater PTWHC3AF800Y140D00	
44	211-50411	Exhaust solenoid valve assembly	Spectrometer exhaust line
45	042-00126-33	Vacuum pump GDH-162-200KF	
46	018-31550-09	Super sun spring hose (1.7MT)	

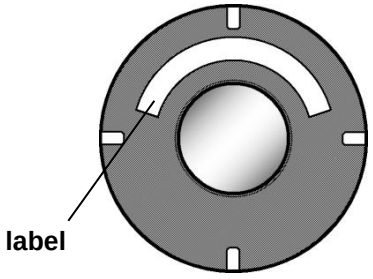
*1 There are two types of condenser lenses, and which one to use depends on the specification of the instrument. See below for more details.

How to distinguish similar parts

1. Condenser lens

There are two types of condenser lenses.

Part No. is shown on the label put on the lens assembly, as described on the figure right.



(1) 202-46611-01 Lens assembly (Q1)

Standard type lens.

(2) 205-01583-01 Lens assembly (MgF_2)

Used for the analysis of elements that emit vacuum-UV lines, such as oxygen or nitrogen.

Chapter 1

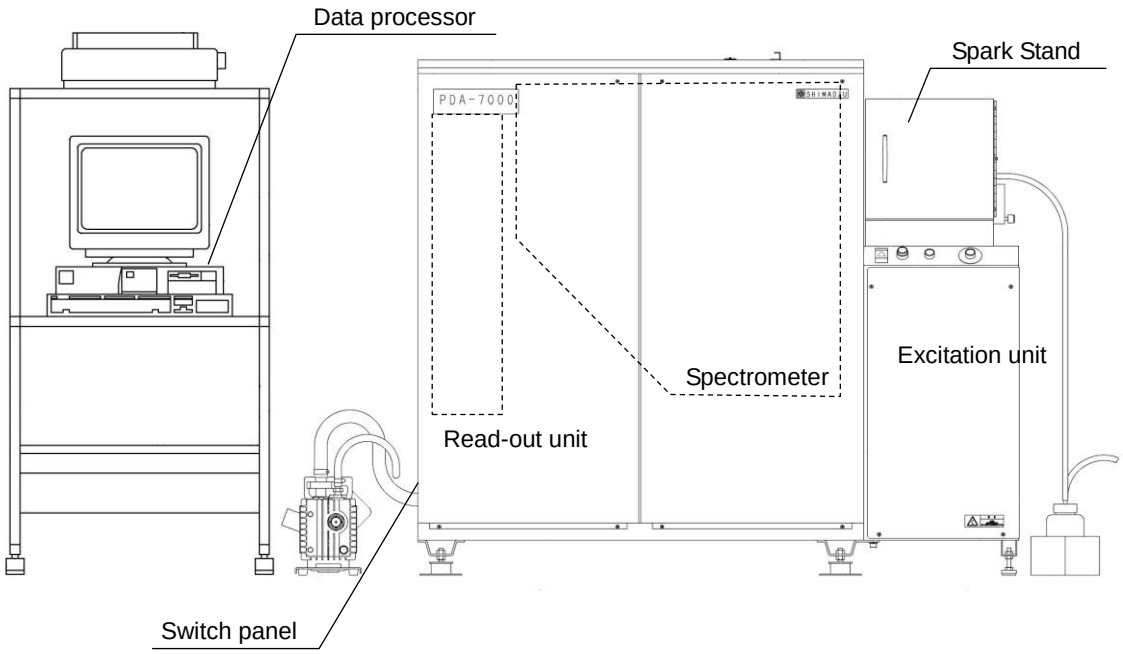
Configuration and Function of the Instrument

1 Configuration and Function of the Instrument

The emission analysis is a method to carry out a quantitative analysis of elements contained in a sample, by generating the optical emission by giving the sample electric and thermal energy, and then measuring the intensity of the bright-line spectra peculiar to those elements using the spectrometer.

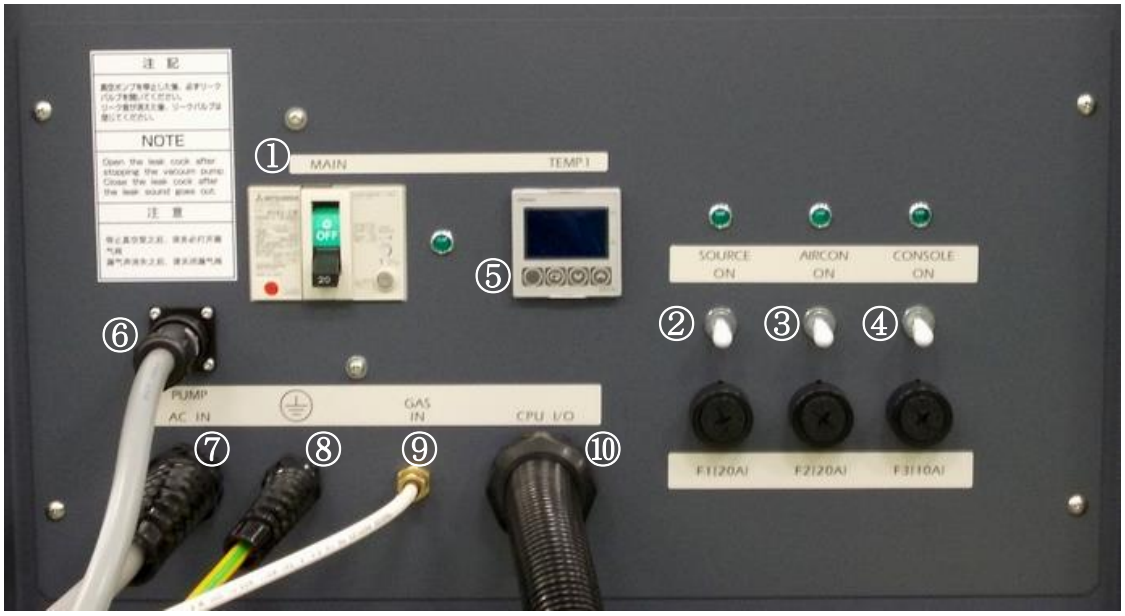
The quantitative analysis for emission analysis is carried out by utilizing a proportional relation existing within a certain range, between the content of an element and the intensity of its spectral line.

The configuration of the instrument is shown in the figure below.



Configuration of PDA-7000

1.1 Switch panel



Switch panel

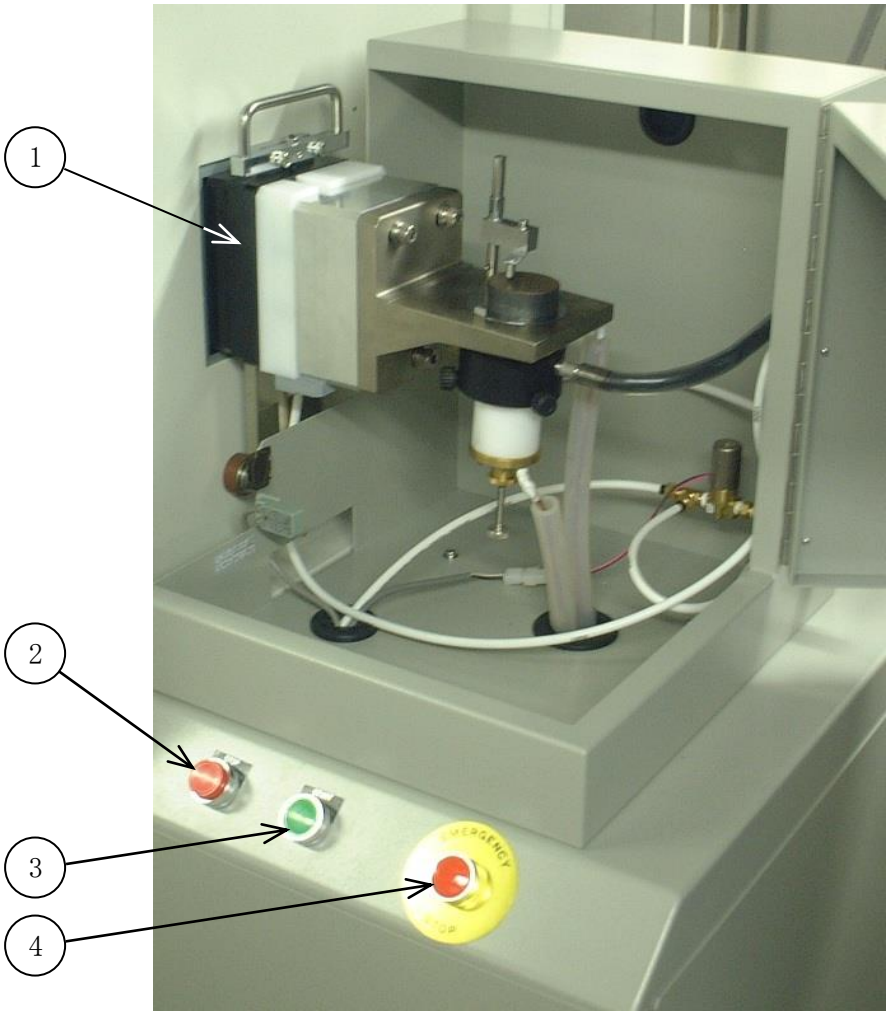
① MAIN	Main power switch	Supplies power to the entire instrument.
② SOURCE	Excitation unit power switch	Supplies power to the excitation unit.
③ AIRCON	Temperature controller power switch	Supplies power to the temperature control system.
④ CONSOLE	Read-out unit power switch	Supplies power to the read-out and data processor system.
⑤ TEMP	Temperature control display	Displays temperature of the spectrometer system and set temperature.
⑥ PUMP	Power supply for pump	Supplies power to the vacuum pump.
⑦ AC IN	Main power	Accepts the power cable.
⑧ ⏚	Grounding terminal	Accepts the grounding cable.
⑨ GAS IN	Gas flow entrance	Accepts the gas tube.
⑩ CPU I/O	Data processor system	Accepts the cable for data processor system (bellows type tube)

Note

- **Never touch any switch other than the power switch.**
- **Never change the set temperature of the spectrophotometer system; this may cause deterioration of analysis precision.**
  The set temperature is 40°C.
- **Before turning off the main power, be sure to turn off the SOURCE, AIRCON and CONSOLE switches.**
- **If the temperature of the spectrophotometer system exceeds 42°C, the buzzer on the temperature display will give a sound. (Regarding the corrective action, refer to "Chapter 7 Troubleshooting")**

1.2 Spark stand

The excitation unit consists of a spark stand which generates spark discharge between a metal sample and electrode and an excitation unit which controls high voltage for discharge.



Spark stand

① Condenser lens
(inside the flange, so not directly visible)
② Stop switch
③ Start switch
④ Emergency stop switch

The spark stand door is provided with a safety switch. The safety switch prevents emission from being started by mistake at the time of maintenance and inspection of the spark stand. Opening the spark stand door activates the safety circuit, cutting off the emission start signal.

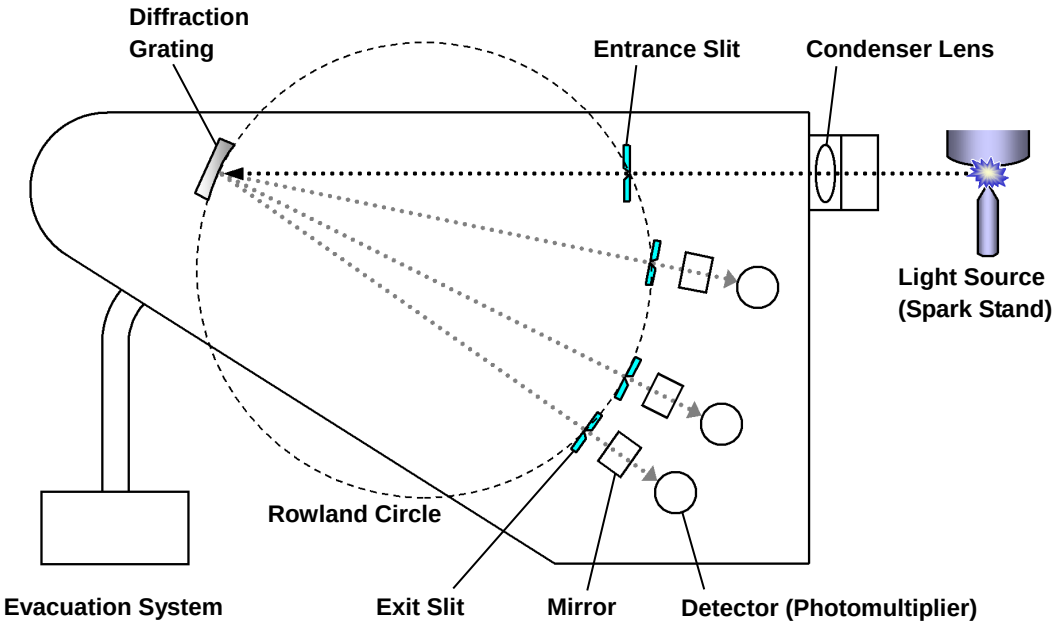
Warning

Electric Shock

Never carry out analysis or maintenance service with the safety switch disabled.

1.3 Spectrometer

The configuration of the spectrometer system is shown in the figure below.



Configuration of the spectrometer

The light coming out of the light source goes through the condenser lens and the entrance slit, then diffracted by the diffraction grating and focused on the Rowland circle based on each wavelength. Only the needed lines out of these spectra pass through the exit slits, and enter the photomultipliers through the mirrors.

The entrance slit can be moved along the Rowland circle by the scanning dial, so that the spectral line could be aligned correctly on the exit slit.

(1) Condenser lens

The condenser lens leads the light generated by the light source to the spectrometer. At the same time, it serves as a partition between the vacuum in the spectrometer and the outside air.

The condenser lens is made of fused quartz which allows the ultraviolet radiation to easily pass through and is designed so that the light at 200 nm would be focused on the diffraction grating surface.

(2) Temperature control circuit

To prevent the optical system deviation due to a temperature change, the spectrometer is kept within a temperature range of 40±1°C by the temperature control circuit.

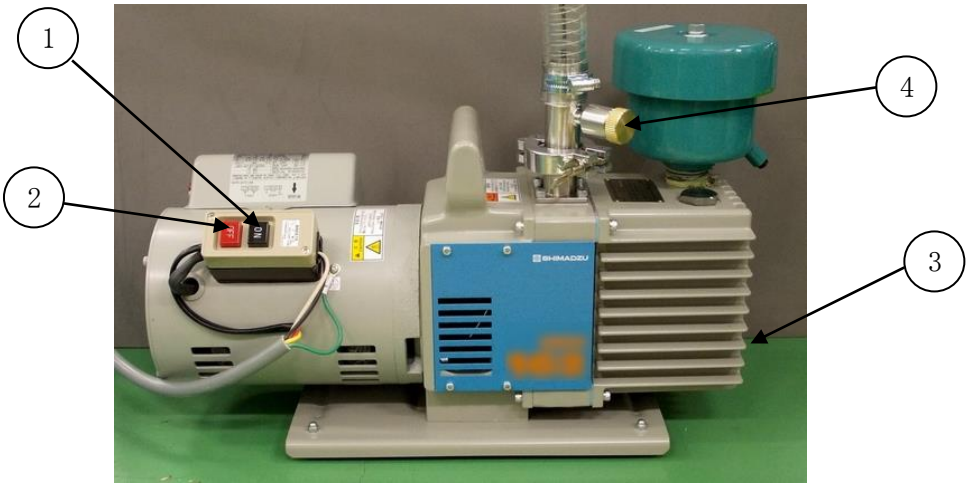
(3) Lamp

The lamp is used as a monitor light source at the time of the read-out unit inspection.

(4) Evacuation system

The light at the wavelength of 200 nm or less which is called the vacuum ultraviolet area is absorbed by oxygen in the air.

Therefore, the spectrometer inside is kept at the pressure of 2Pa or less by the evacuation system.



Vacuum pump

① Power On switch	Starts the vacuum pump.
② Power Off switch	Stops the vacuum pump.
③ Oil gauge	Indicates the oil level.
④ Pump Leak Valve	Used to prevent the lowering of the pump oil level when the pump is idle.

(The oil mist separator on the photo above is an option)

Caution

- **Check that the oil level of the vacuum pump is within the red circle of the oil gauge.**
- **When the temperature is low such as in winter, make sure that the room temperature is high enough before turning ON the power of the pump.**

Note

When the vacuum pump is OFF, confirm that the vacuum status inside the spectrometer is "OK" on Instrument Check screen of the data processing software at least once a day.
If it is "Air", turn ON the vacuum pump and wait until the vacuum status becomes "OK", and then continue to run the vacuum pump at least three more hours.

1.4 Read-out unit

The read-out unit converts the intensity of the dispersed light into the current, and measures it.

On an emission analysis, the intensity of the light emitted from a sample generates large fluctuation at a short time. Therefore, the method of integrating the output current for a specified time to measure the average current value is adopted.

(1) Photo detector (PMT)

Converts the intensity of the light dispersed by the spectrometer unit into the current.

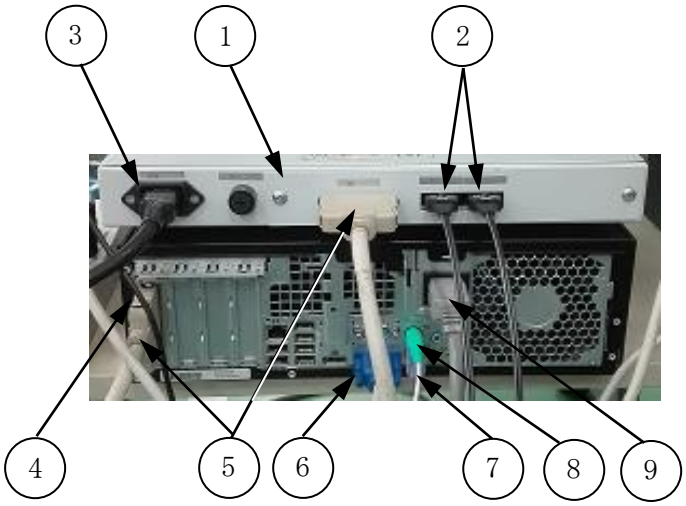
(2) Photo detector sensitivity controller (Attenuator)

Changes high voltage supplied to the photo detector, to control the sensitivity of it. A high voltage generator and an attenuator are provided at each channel.

(3) Integrator

Integrates the output current from the detector.

1.5 Data processor system



Data processor system (rear side)

① CPU Cont. UNIT
② Communications connectors
③ Power cable for CPU Cont. UNIT
④ SCSI card
⑤ SCSI cable
⑥ Display cable
⑦ Keyboard cable
⑧ Mouse cable
⑨ Power cable for PC

Printer

① Power switch (on the lower part of the left side face)
② Switch to enable printing

Note

- **Do not print when the switch to enable printing is turned OFF. Attempting to print when the printer is in a state where it is unable to print (switch to enable printing is OFF, or there is no printing paper, etc.) causes the print data to accumulate in the printer buffer, resulting in abnormal printing when print operation is restarted. When this type of situation occurs, quickly resolve the problem, and restart the printer. If the problem cannot be solved, use the analyzer with the printer turned off.**

- **Do not use paper feed knob on the side of the printer while the printer is ON, as that may cause a failure.**

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Chapter 2 Specification

2 Specification

2.1 Spectrometer

• Mounting	Paschen-Runge mounting of concave grating	
• Focal length	600 mm	
• Wavelength range	121 to 589 nm	
• Evacuation system	Vacuum pump	Rotary vacuum pump
• Temperature control	Set temperature	40°C
	Control precision	Set temperature ±1°C
	(Under room temperature of 10 - 28°C)	
• Number of photomultipliers	64 maximum	

2.2 Excitation unit

(1) Spark stand

• Gas type	Argon gas	
• Gas consumption	During analysis	Approximately 3L (n=2)
	Standby	1L / min.
	Sleep	0.1L / min.

(2) Excitatiion source unit

• Main voltage	500V / 300V
• Discharge frequency	40Hz - 500Hz
	(Three types of setting available)
• Discharge conditions	5 conditions
• Voltage precision	Main voltage ±1%
	(Input voltage fluctuation should be within ±10%)
• Electrode reconditioning	> Auto reconditioning by discharge polarity reverse at the end of each analysis
	> Manual reconditioning

2.3 Read-out unit

• Number of channels	64 maximum
• Measurement method	> PDA measurement
	> Digital integration
• Integrator	Single pulse integrator
	Miller integration (With sample hold)
• High voltage supply	-1100V maximum

2.4 Data processor system

(1) Hardware

Specifications required for the data processing PC

• CPU	Core i3 3.00GHz or more
• Memory	2GB or more
• Storage devices	> HDD with 100MB or more free space
	> CD-R Drive x 1
• Operating system	Windows 10 64bit
• Extension slot	PCI Express bus x 1
• Monitor resolution	1280 x 1024 pixels

Specifications required for SCSI

• Interface board	ADAPTEC SCSI-Card ASC-29320LPE
• Cable	High pitch 50-pin - 68-pin, plug-plug type

(2) Software

• Number of elements	64 elements maximum (Including internal standard elements)
• Number of channels	64 channels maximum (Including internal standard element channels)
• Number of PDA channels	64 channels maximum (including internal standard element channels)
• PDA processing	> Number of samplings 2000 pulse maximum / sequence > Intensity distribution measurement
• Analytical group	> Number of analytical groups registered Free (depends on hard disk capacity) > Analytical conditions, working curves and master curve coefficients can be shared.
• Number of internal standard elements	2 maximum for each channel
• Recalibration	1point / 2 points recalibration
• Working curve calculation	> 1st to 3rd order formulas > Channel skip > Number of standard samples 2000 samples maximum / analytical group > Number of working curves 4 working curves maximum / channel
• Matrix correction	16 elements maximum / channel
• Standard samples	2000 samples maximum / base element
• Other corrections	> 100% correction > Master curve correction > Special calculation
• Analysis types	> Content analysis > 4-times analysis > Global analysis > Round analysis > Intensity analysis for recalibration > Intensity analysis for working curve > Raw / Corrected intensity analysis > Attenuator adjustment (with / without internal standard correction)

- Analysis functions
  - > Number of analyses 21 maximum
  - > Measurement sequence 3 maximum
  - > Cancel of analysis results
  - > Immediate calculation
    (Average value calculation before the end of multi-time analysis)
  - > Recycle calculation
  - > Supplementary analysis
  - > Abnormal burn monitoring
  - > Frequency distribution and pulse-intensity profile data
- Process control
  - > Standard control
  - > R-control
- Analysis result output settings
  - > Display and print-out orders
  - > Number of digits
  - > Display and print-out items (Ave./R/SD/CV)
  - > Display and print-out layouts and fonts
- Result transmission
  - > Serial single-host transmission
  - > Serial multi-host transmission (option)
    5 hosts maximum
  - > Serial multi-printer transmission (option)
    5 printers maximum
  - > LAN (Ethernet) transmission
- Result storage settings
  - > Items below can be stored
    - Each time analysis result and Average
    - Intensity value
    - Round analysis result
  - > Number of results stored
    Depends only on the HDD capacity
- Alloy identification
  - > Number of standards groups
    Depends only on the HDD capacity
  - > Number standards
    1000 maximum / group

• Report	> Number of results processed at one time 15000 maximum > $\overline{X}$-R control chart > Histogram > Daily report > Multiple data transmission > Data conversion to CSV format > Database support (Microsoft Access)
• Maintenance	> Instrument check - Internal pressure of spectrometer - Temperature of spectrometer > Maintenance Management - Spark stand cleaning - Electrode change - Slit Adjustment - Pump oil change - Cleaning of the Igniter > Waste discharge > Manual scanning > Lamp test > File maintenance
• Security	Password protection of analysis information

Chapter 3

Starting and Stopping the Instrument

3 Starting and Stopping the Instrument

3.1 Inspection before using the instrument

Caution

- **Check that the room temperature is within the range of 10 - 28°C.**
- **Check that the power cable is correctly connected to the control panel and the switch on the distribution board which supplies power to the instrument is ON.**
- **Check that the grounding cable is correctly connected to the switch panel.**
- **Check that the vacuum pump is working correctly.**
- **Check that the oil level of the vacuum pump is within the red circle of the oil gauge while the pump is running.**
- **Check that the preset temperature of the air-conditioning system is set at 40°C.**
- **Check that the electrode and the sample are correctly set on the spark stand.**

3.2 Starting the instrument (Daily use)

The routine procedure to start the instrument is described below.

1 Turn on the **SOURCE** switch on the switch panel.
2 Confirm that the temperature control indication on the switch panel is 40°C.
3 Confirm that the vacuum pump is operating.
4 Check the water level in the argon gas bubbler.
(See "4.1 Argon gas bubbler".)
5 Adjust the argon gas flow rate to 1L / min. using the BYPASS valve.
(See "4.2 Checking gas flow rate".)
6 Turn on the printer.
7 Turn on the monitor.
8 Carry out recalibration.
(See "4.7 Recalibration".)
9 Carry out master curve recalibration when necessary.
(See "4.8 Master curve recalibration".)
10 Carry out check analysis using the sample for checking.
(See "4.9 Check analysis".)

3.3 Starting the instrument after it has not been used for a long time

The procedure to start up the instrument after it has not been used for a long period of time is described below.

1 Turn on the **MAIN** switch on the switch panel, and then turn on the **AIRCON**, **CONSOLE**, and **SOURCE** switches.
2 Turn on the printer.
3 Turn on the monitor.
4 Turn on the data processor system. The window to enter a password is displayed. Enter the password.
5 Check the water level of the argon gas bubbler.
(See "4.1 Argon gas bubbler".)

6 Adjust the argon gas flow rate during standby to 1L / min and the flow rate during analysis to 10L / min.
(See "4.2 Checking gas flow rate".)

7 Confirm that the pump leak valve is closed, and then turn on the vacuum pump.

Note

When the vacuum pump is OFF, confirm that the vacuum status inside the spectrometer is "OK" on Instrument Check screen of the data processing software at least once a day.
If it is "Air", turn ON the vacuum pump and wait until the vacuum status becomes "OK", and then continue to run the vacuum pump at least three more hours.

8 Wait for four to five hours until the spectrometer temperature stabilizes.
9 When the inner pressure of the spectrometer becomes OK on **Instrument Check** screen on the data processor system, turn on the photo detector.
(See "4.3 Turning ON / OFF photo detector".)

10 Carry out waste discharge.
(See "5.10 Waste discharge".)

11 Carry out recalibration.
(See "4.7 Recalibration".)

12 Carry out master curve recalibration when necessary.
(See "4.8 Master curve recalibration".)

13 Carry out check analysis using the sample for checking.
(See "4.9 Check analysis".)

3.4 Shutting down the instrument (Daily use)

The routine procedure to shut down the instrument is described below.

1 Turn off the **SOURCE** switch on the switch panel. Keep the **MAIN**, **AIRCON**, and **CONSOLE** switches ON.
2 Place an analysis sample for blank analysis on the sample plate of the spark stand and prevent air or debris from entering into the emission chamber.
3 Adjust the BYPASS valve to reduce the argon gas flow rate.
(See "4.2 Checking gas flow rate".)
4 Turn off the printer.
5 Turn off the monitor.

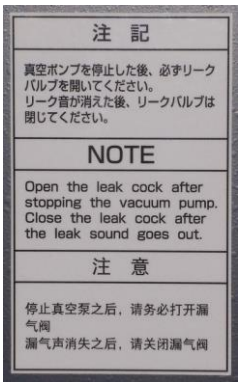
Note
When analysis is not performed for a long time, for example at night, display a window other than the analysis window on the data processing software and then turn off the monitor.

3.5 Shutting down the instrument for a long time

The procedure to shut down the instrument for a long period of time is described below.

1 Turn off the photo detector on **Instrument Check** screen on the data processor system.
(See "4.3 Turning ON / OFF photo detector".)

2 Turn off the vacuum pump, and then open the pump leak valve slowly to allow the air into the suction hose to prevent the contamination of the hose by the pump oil.
After the sound of the air flowing into the hose disappears, close the pump leak valve.



Pump Leak Valve

3 Close the BYPASS valve.
See "4.2 Checking gas flow rate".)

Caution
Do not over-tighten the BYPASS valve because it may break.

4 Turn off the printer.
5 Turn off the data processor system.
6 Turn off the monitor.
7 Turn off the **AIRCON**, **CONSOLE**, and **SOURCE** switches on the switch panel.
8 Turn off the **MAIN** switch on the switch panel.

9 Place an analysis sample for blank analysis on the sample plate of the spark stand and prevent air or debris from entering into the emission chamber.

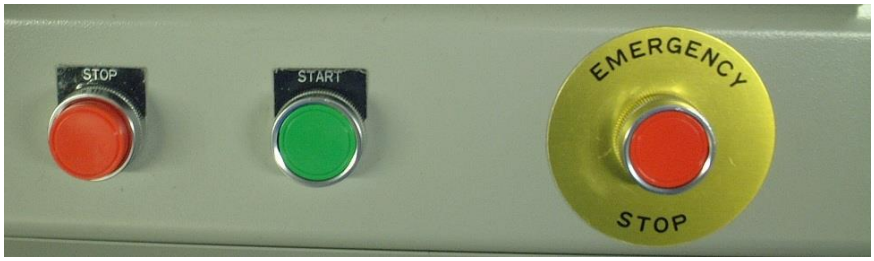
10 Clean the spark stand and exchange the electrode.
(See "5.3 Maintaining the spark stand".)

3.6 Emergency stop and recovering

Caution
If any problem should occur on the instrument, stop the instrument by emergency stop and immediately remove the cause of the problem.

(1) Emergency stop

To stop the instrument in an emergency, press the emergency stop switch. It will cut off power supply to the entire instrument.



Emergency stop switch

Caution
If the vacuum pump cannot be turned on immediately after the emergency stop, open the pump leak valve following step (2) of "3.5 Shutting down the instrument for longtime".

(2)Recovering from emergency stop

1 Turn OFF the **AIRCON**, **CONSOLE**, **SOURCE** and then **MAIN** switches on the switch panel.
2 Turn on the **MAIN** switch on the switch panel, and then turn on the **AIRCON**, **CONSOLE** and **SOURCE** switches.
3 Turn on the PC. Then start up PDA-Win following the usual procedures.

Chapter 4 Operation

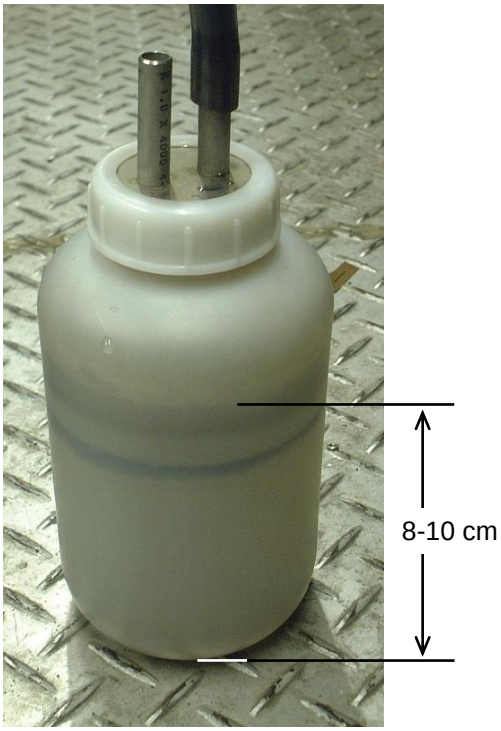
4 Operation

4.1 Argon gas bubbler

An argon gas bubbler is installed on the argon gas exhaust line. The water in the argon gas bubbler works as a resistance to suppress the pressure fluctuation in the spark stand.

Keep the water level in the argon gas bubbler at 8 to 10cm. Replace the water if it is heavily contaminated.

After adding or changing water in the argon gas bubbler, recalibration is necessary. For details, see the table shown in "5.2 Maintenance Flow" and follow the specified procedure.



Gas Bubbler

Caution
Do not wrap flammable cloth around the outlet of the argon gas bubbler.

Note

- **When the water level in the bubbler lowers, the analytical value varies.**
- **Connect a vinyl hose or something similar to the outlet of the bubbler to discharge the exhaust air from the bubbler out of the room.**
- **Fine powder of evaporated samples (iron, aluminum, copper, etc.) is accumulated in the bubbler.**
- **Evaporated samples accumulated in the vinyl hose that connects the spark stand and bubbler may cause analytical value fluctuations. Blow air into the vinyl hose or replace it with a new one regularly.**
- **Confirm that the spark stand outlet pipe and vinyl hose are firmly connected. Loose connection may result in analytical value fluctuations.**

4.2 Checking gas flow rate

(1) When the instrument is in operation

Confirm that argon gas is flowing at the following rate: 10L / min during analysis (discharge) and 1L / min during the standby, by a flow meter.

To adjust the flow rate during analysis, use screens such as **Manual Scanning**, on which continuous discharge can be made.

Note

- **Read the gas flow rate at the center of the ball.**
- **When the argon gas supply pressure, which is set to 140kPa to 200kPa (1.4kg / cm^2 to 2.0kg / cm^2), changes, the argon gas flow rate changes accordingly. In this case, set the flow rate to an appropriate value again.**
- **Check the argon gas flow rate on daily basis. If the residual pressure of argon gas cylinder lowers, the argon gas flow rate changes accordingly, and may cause analytical value fluctuations.**

(2) When the instrument is not used or at nighttime

When analysis is not carried out for a long time or at nighttime, argon gas flow rate can be reduced to 100mL / min - 200mL / min by adjusting the valve for no use instrument or nighttime.

When source SW is turned off, argon gas flow rate can be set to this flow rate.

The BYPASS valve can also be closed with the main cock of the argon gas cylinder open.

Return the argon gas flow rate during standby to the original value prior to starting analysis.

Caution

Do not over-tighten the BYPASS valve because it may break.

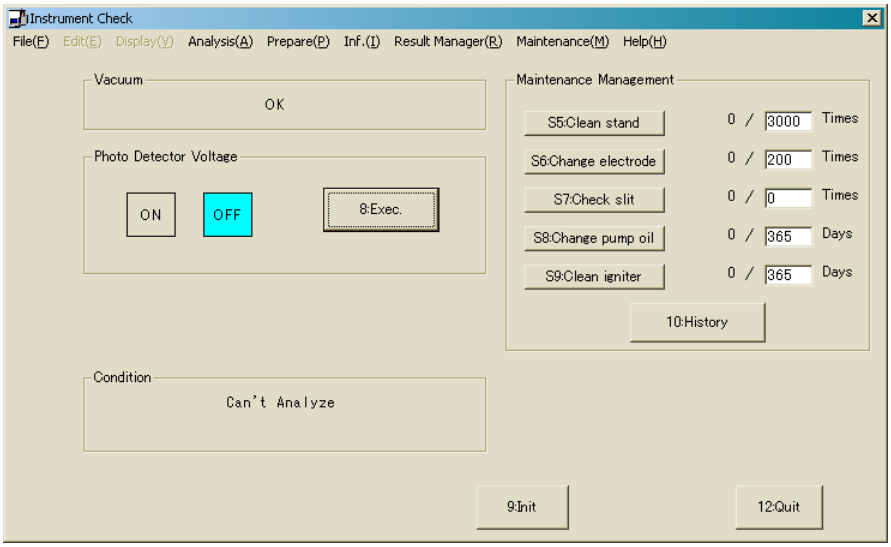
Note

If nitrogen is contained in an analytical element, reducing the gas flow rate during standby may influence analytical values.

4.3 Turning ON / OFF photo detector

The light emitted from the spark stand can be detected by the photo detector (photomultiplier) by turning the detector ON. Follow the procedures below to turn ON / OFF the detector.

1 Select **Instrument Check** on **Maintenance** menu.
Instrument Check window appears.



Screen 4.1 Instrument check

2 Check the photo detector status by the indicator in **Photo Detector Voltage** area.
3 Confirm that the vacuum status displayed in **Vacuum** area is **OK**.
4 Press **8:Exec.** to turn On or OFF the photo detector.

Caution
Prior to turning on the photo detector, be sure to confirm that Vacuum Status is OK.

4.4 Sample pretreatment

Analytical samples need to be ground or polished before analysis. If samples are not ground or polished properly, correct analysis results cannot be obtained.

The following shows the grinding/polishing methods according to the type of metals.

Steel, nickel, cobalt, titanium, and their alloyed metals
: Belt sander (grain size #60 to 80)

Cast iron : Belt sander (grain size #40)

Nonferrous metals
(aluminum, copper, zinc, lead, tin, magnesium, and their alloyed metals
: Lathe

Caution

Regularly clean the inside of the belt sander and the dust collector filter, and also replace the filter. Dust accumulated in the dust collector may become a fire hazard.
There is a high risk of fire when aluminum or magnesium samples are used.

Note

- **Use the belt sander and turning tool of the lathe only for sample pretreatment.**
  If they are used for polishing or grinding other materials, remaining impurities may stick to samples, resulting in analytical value fluctuations.
  If they are used for polishing or grinding tools, remaining oils may stick to samples, resulting in analytical value fluctuations.

- **After polishing or grinding, store recalibration samples in a desiccator or something similar in order to prevent oxidation of the surface to be analyzed.**

Note

When using a belt sander for sample pretreatment, follow the instructions below.

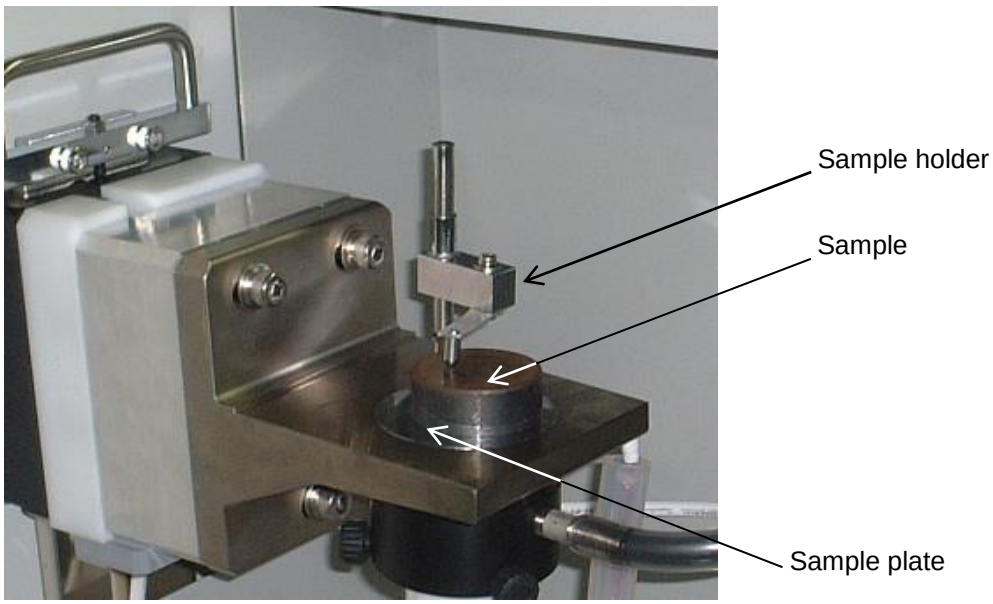
- **Use the belt sander dry.**
- **Regularly replace the belt sander. If the belt is worn, samples cannot be polished properly, which results in lower analytical accuracy.**
- **Regularly replace the backing plate of the belt sander.**
  If an old backing plate is continuously used, flatness of the polished sample surface becomes poor, which may result in argon gas leakage and unstable discharge.

Note

When using a lathe for sample pretreatment, follow the instructions below.

- **Regularly clean the lathe.**
- **Regularly grind or replace the turning tool of the lathe. If the tool is worn, flatness of the ground sample surface becomes poor.**
- **When a lubricant is necessary for grinding samples, use ethanol. Do not use grinding oil for machining or methanol.**
  Using machine oil may cause abnormal discharge.
- **If machine oil or other oil sticks to a sample, clean it with ethanol. Do not use methanol.**

4.5 Setting the sample



Spark stand

Note

<u>Be sure to tightly cover the hole of the sample plate with the sample.</u>
A clearance between the sample plate and sample allows the air to flow into the stand, causing abnormal discharge. In the same manner, rough sample surface or flaws on the sample surface may cause the air to flow into the stand.

<u>Be sure to tightly hold the sample using the sample holder.</u>
Tightly hold the sample over the hole of the sample plate with the sample holder. Insufficient holding creates a clearance between the sample and the sample plate; this may cause the air to flow into the stand.

<u>Never make the discharge toward an already created emission trace.</u>
The discharge to an emission trace again will cause analytical problems.

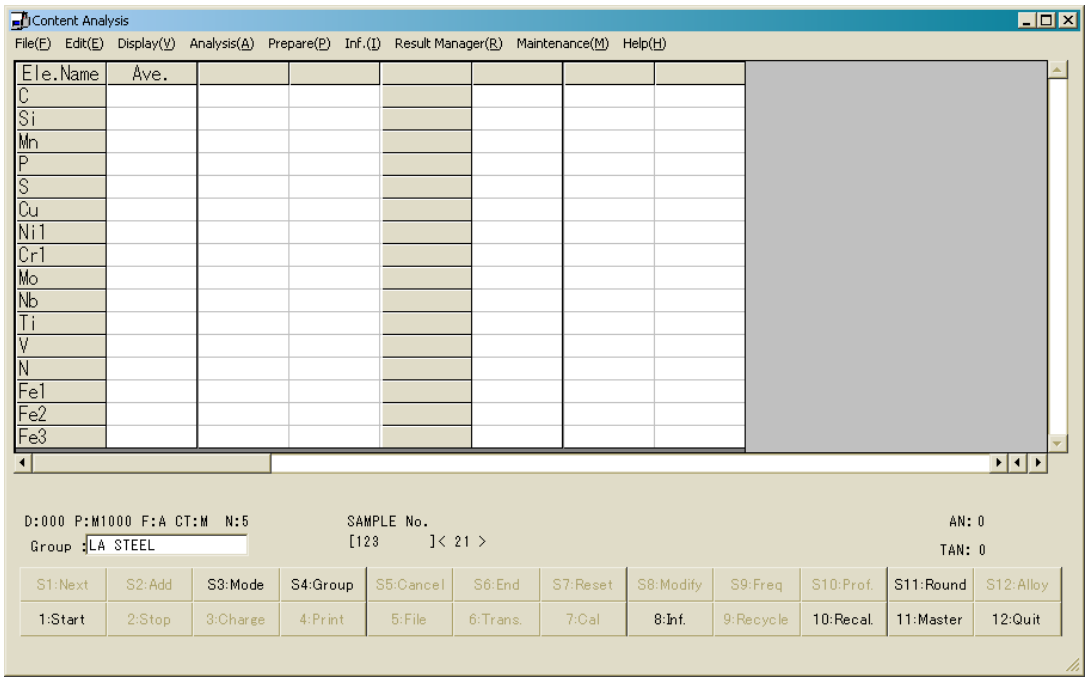
<u>Analyze the circumferential part of the sample.</u>
Generally, circumferential area is more uniform than the sample center, allowing stable analysis.

4.6 Content analysis

The contents of each element in the sample can be measured on this screen.

1 Select **Analysis** - **Cont.** on the menu bar.

2 Input the name of the analytical group in the **Group** textbox or select it by pressing **S4:Group** key.



Screen 4.2 Content analysis

3 Set the sample on the spark stand and hold it by the sample holder.

4 Start the discharge by pressing **1:Start** key or the start button on the spark stand.

5 Take out the sample, and change the position of the sample to be analyzed. Then, repeat procedures 3 and 4 specified times.

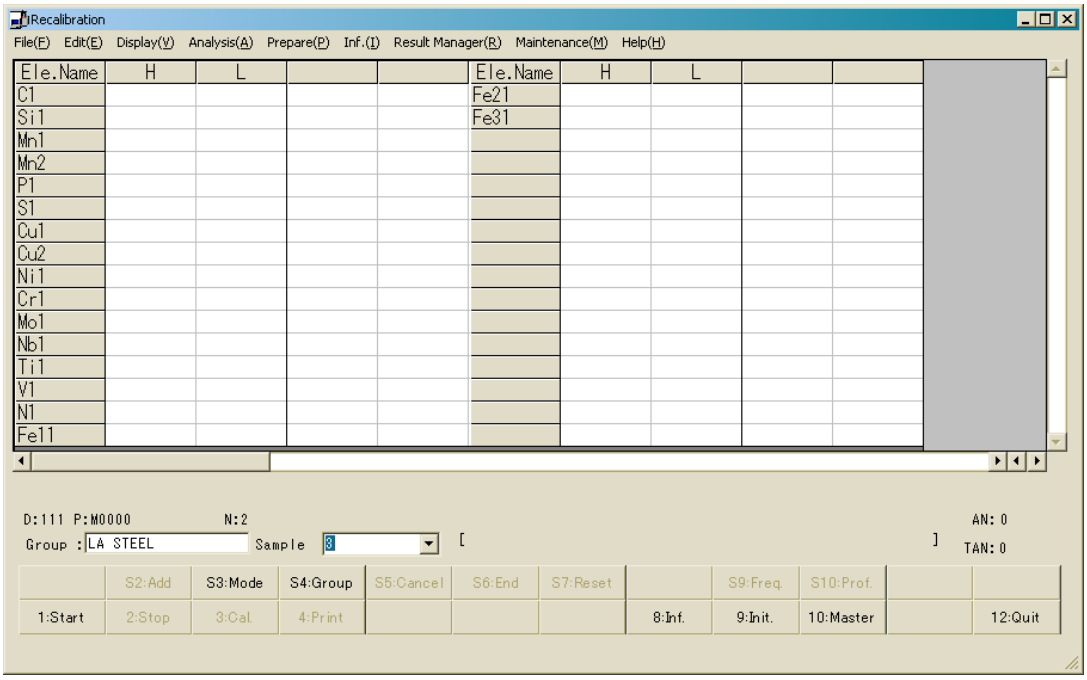
4.7 Recalibration

To calculate the recalibration coefficient, carry out the recalibration analysis.

1 Select **Analysis** - **Recalibration** on the menu bar.

2 Input the name of the analytical group in the **Group** textbox or select it by pressing **S4:Group** key.

3 Select the name of the recalibration sample to be analyzed in the **Sample** combo box.



Screen 4.3 Recalibration

4 Set the sample on the spark stand and hold it by the sample holder.

5 Start the discharge by pressing **1:Start** key or the start button on the spark stand.

6 Take out the sample, and change the position of the sample to be analyzed.
Then, repeat procedures 3 and 4 specified times.

7 Repeat procedures 3 to 6 until the analyses of all the recalibration samples are completed.

8 Press **3:Cal.** Key.
The recalibration coefficients (α, β and k values) are calculated and they are automatically stored in the recalibration information.
In the case of one point recalibration, coefficients α and β are not initialized. In the case of two-point recalibration, k is initialized to 1.0.

Note

After recalibration analysis, check the intensity of the internal standard element and also recalibration coefficients α, β and k to make sure that they have not changed considerably from the values before recalibration.

Note

After recalibration, carry out check analysis using the sample for checking (see page 4-14).
When master curve correction is used, carry out master curve correction (see page 4-13) after recalibration and then carry out check analysis using the sample for checking.

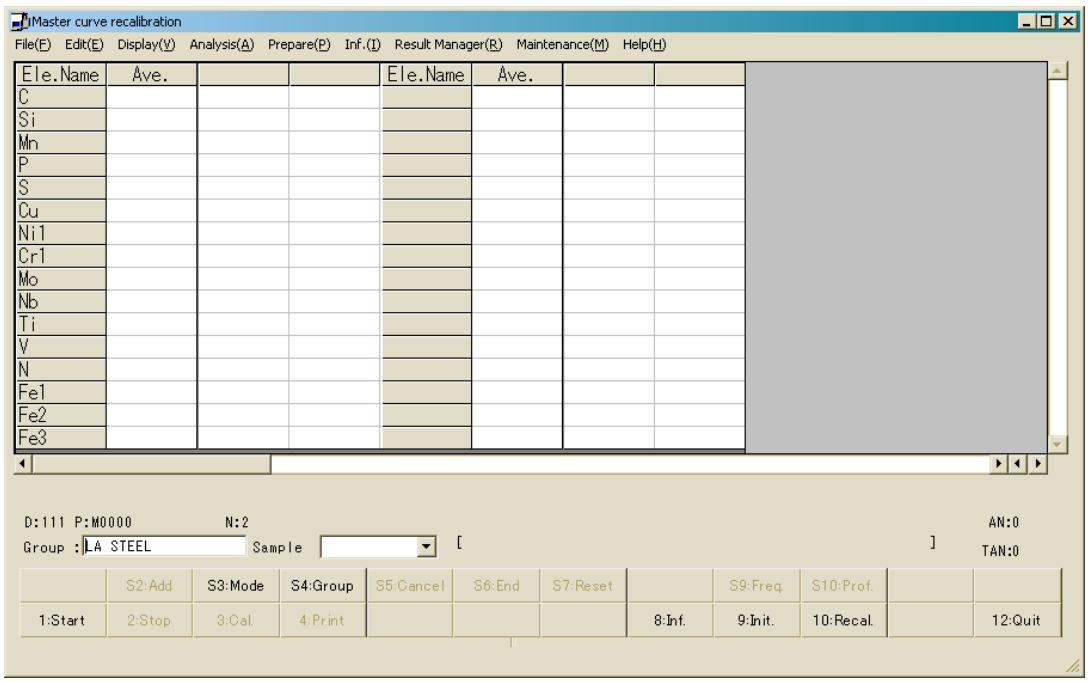
4.8 Master curve recalibration

To calculate the master curve correction coefficient, carry out the master curve recalibration analysis.

1 Select **Analysis** -**Master Curve** on the menu bar.

2 Input the name of the analytical group in the **Group** textbox or select it by pressing **S4:Group** key.

3 Select the name of the master curve sample to be analyzed in the **Sample** combo box.



Screen 4.4 Master curve recalibration analysis

4 Set the sample on the spark stand and hold it by the sample holder.

5 Start the discharge by pressing **1:Start** key or the start button on the spark stand.

6 Take out the sample, and change the position of the sample to be analyzed.
Then, repeat procedures 3 and 4 specified times.

7 Repeat procedures 3 to 6 until the analyses of all the master curve samples are completed.

8 Press **3:Cal.** Key.
The master curve coefficients (AC and MC values) are calculated and they are automatically stored in the master curve information.

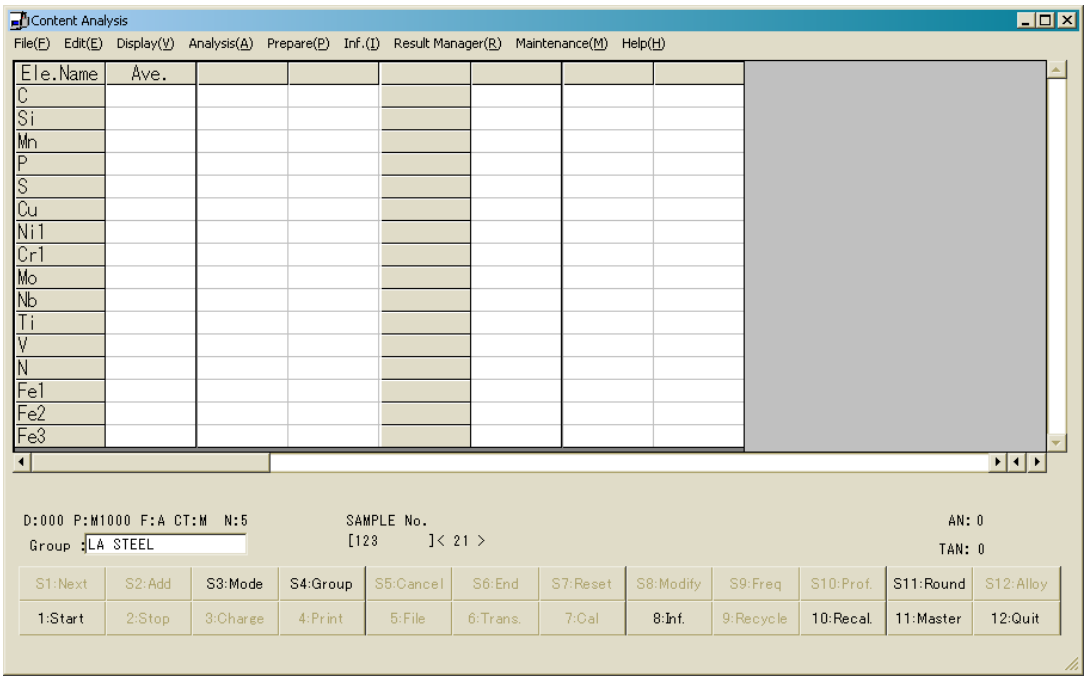
Note
After master curve correction, carry out check analysis using the sample for checking (below).

4.9 Check analysis

Confirm whether the content percentages of the elements are correctly measured, using a sample with known composition.

1 Select **Analysis** - **Cont.** on the menu bar.

2 Input the name of the analytical group in the **Group** textbox or select it by pressing **S4:Group** key.



Screen 4.5 Content analysis

3 Set the sample on the spark stand and hold it by the sample holder.

4 Start the discharge by pressing **1:Start** key or the start button on the spark stand.

5 Take out the sample, and change the position of the sample to be analyzed. Then, repeat procedures 3 and 4 specified times.

6 Confirm that the correct content percentages are obtained.

Chapter 5 Maintenance

5 Maintenance

To use the instrument correctly and safely, provide maintenance and inspection regularly.

5.1 Maintenance and inspection items

Items that need to be maintained or inspected are listed below.

For the flow of maintenance service, see "5.2 Maintenance Flow". For detailed procedure of each maintenance service, see the applicable section described in the “Reference” column in the table below.

(1) Periodical Maintenance

Maintenance Item	Cycle	Reference
Spark stand and electrode	Using reverse discharge: Every 200 to 300 discharges	5.3 Maintaining the spark stand
	Using manual brushing: Every 3,000 discharges	
Electrode O-ring	Monthly	5.4 Exchanging the electrode O-ring
Condenser lens	When MgF_2 lens is used Every 10,000 discharges	5.5 Cleaning the condenser lens
	When quartz lens is used Every 100,000 discharges	
Control gap	Every 100,000 discharges	5.6 Adjusting the control gap
Pump oil	Every 6 months	5.7 Replacing pump oil

(2) Other Maintenance

Maintenance Item	Cycle	Reference
Entrance slit	When necessary	5.8 Checking the entrance slit
Read-out unit	When necessary	5.9 Lamp test
Recalibration sample replacement	When recalibration sample runs out	5.12 Replacing recalibration samples
Argon gas replacement	When argon gas runs out	5.13 Replacing argon gas
Software data	Monthly	5.11 File maintenance

(3) Work required after maintenance service

Work	Reference
Waste discharge	5.10 Waste discharge
Recalibration	4.8 Recalibration
Master curve correction (only when necessary)	4.9 Master curve correction
Check analysis using the Sample for checking	4.10 Check analysis

(4) Maintenance service by Shimadzu service engineer

Maintenance Item	Cycle	Reference
Periodic inspection on the instrument	Annually	5.14 Periodic inspection
Temperature control fan heater	Annually	5.15 Cleaning the temperature control fan heater

5.2 Maintenance Flow

After maintenance service, works described in “(3) Work required after maintenance” in "5.1 Maintenance and inspection items" may be necessary. The table below shows the work necessary after each maintenance service.

Maintenance Item	Waste discharge	Recalibration	Master curve correction	Check Analysis
Changing / adding argon gas bubbler water	► **X**	► **O**	► **Ø**	► **O**
Spark stand maintenance	► **O**	► **O**	► **Ø**	► **O**
Exchanging electrode O-ring	► **O**	► **O**	► **Ø**	► **O**
Cleaning the condenser lens	► **O**	► **O**	► **Ø**	► **O**
Control gap adjustment	► **X**	► **O**	► **Ø**	► **O**
Exchanging pump oil	► **X**	► **O**	► **Ø**	► **O**
Entrance slit adjustment	► **O**	► **O**	► **Ø**	► **O**
Lamp test	**X**	**X**	**X**	**X**
Recalibration sample replacement	► **X**	► **O**	► **Ø**	► **O**
Argon gas replacement	► **O**	► **O**	► **Ø**	► **O**
File maintenance	**X**	**X**	**X**	**X**
Bad result value in check analysis	► **X**	► **O**	► **Ø**	► **O**

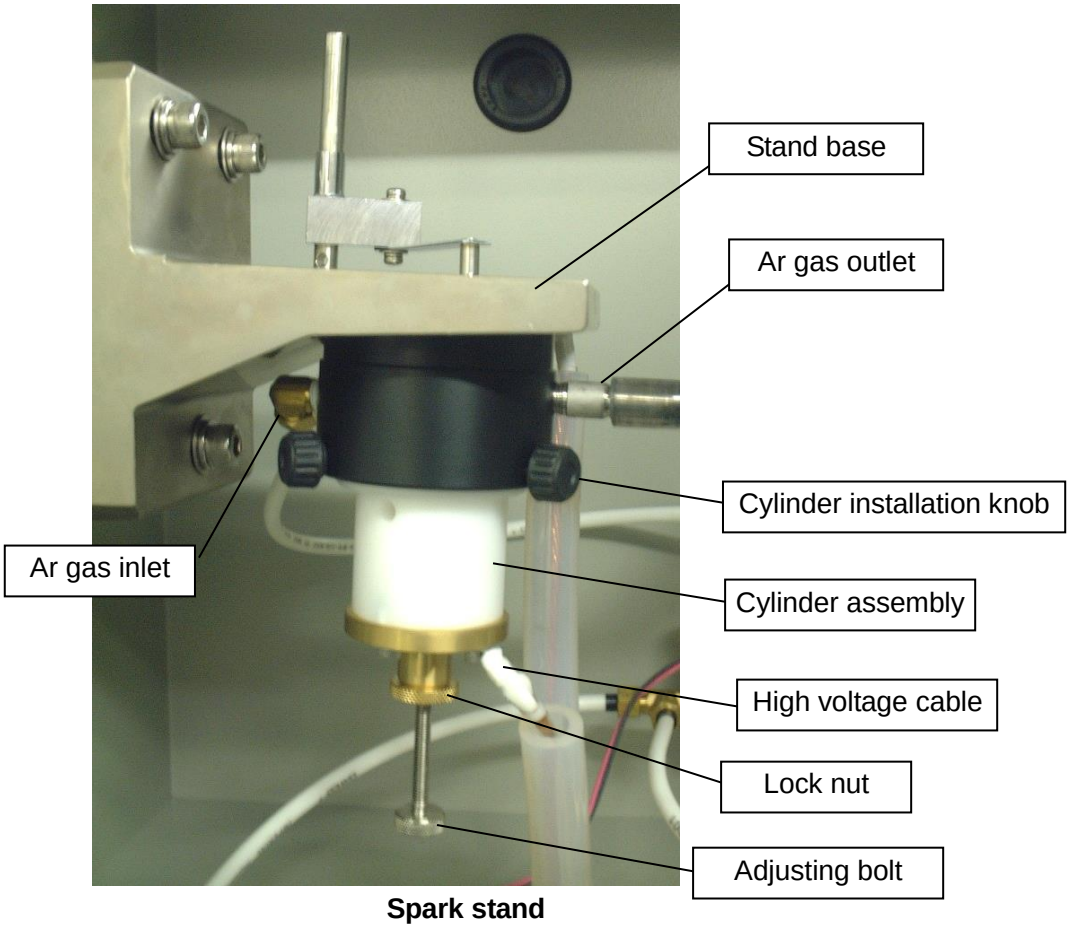
O : Always perform **Ø** : Perform if necessary **X** : Not necessary

Note

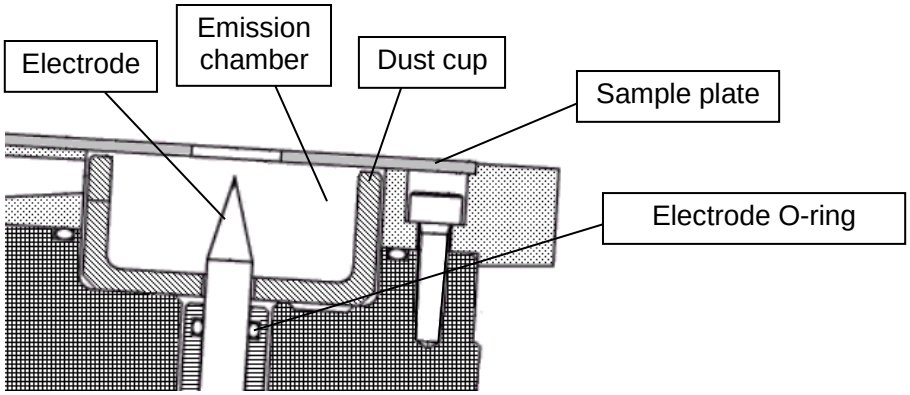
After each maintenance service, be sure to carry out waste discharge, recalibration, master curve correction, and check analysis using the sample for checking according to the table above.

5.3 Maintaining the spark stand

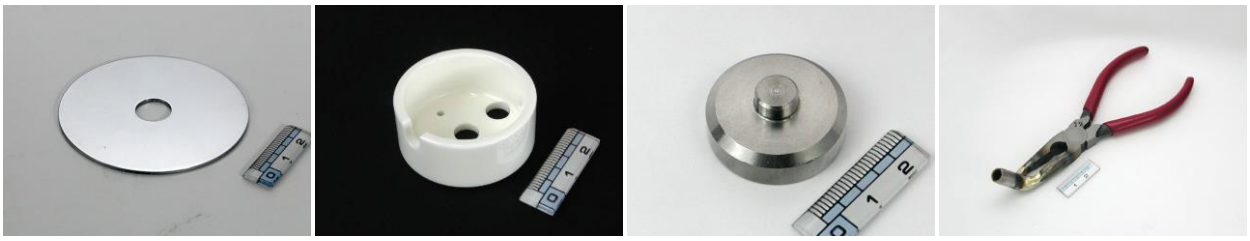
After analysis using the optical emission spectrometer, evaporated sample accumulates inside the spark stand. Evaporated sample sticking to the tip of the electrode changes the analysis gap distance and in turn fluctuates analytical values. To prevent this and keep appropriate conditions for correct analysis, perform maintenance service on the spark stand regularly.



Spark stand



Cross section through the spark stand



(From left to right)Sample plate, Dust cup, Gap gauge and Electrode removal tool

(1) Maintenance cycle

To remove evaporated sample from the tip of the electrode, either of the two methods listed below is usually used. The maintenance cycle for the spark stand depends on the method selected. (The cycle is described in the table about periodical maintenance in page 5-2.)

<Cleaning by reverse discharge>

In this method, discharge is performed with the polarity of the sample and electrode opposite to automatically remove evaporant.

If the reverse discharge time is too short, evaporant may not be removed completely. On the contrary, if the reverse discharge time is too long, the tip of the electrode may wear.

Maintenance cycle: Every 200 to 300 discharges

<Manual brushing>

In this method, the tip of the electrode is cleaned with the accessory brush to remove evaporant. The tip of the electrode must be cleaned every time analysis is finished to prevent excessive evaporant accumulation.

Maintenance cycle: Every 3,000 discharges

Performing either of the above shortens the electrode. The electrode can be used until its total length becomes 65mm. (A new electrode is 100mm long.)

Note

- **To recondition the electrode, select either reverse discharge or using the brush. Do not perform both on one electrode.**
- **When cleaning the tip of the electrode using the brush, remove the evaporant from the entire circumference of the electrode. Brush the electrode from the lower section (blackish area) to the top.**
- **Although the maintenance cycle for reconditioning the electrode using the brush is set to every 3,000 discharges, carry out check analysis using the sample for checking when necessary, even within the maintenance cycle.**

(2) Maintenance procedure

1 Turn off the SOURCE switch on the switch panel for safety.

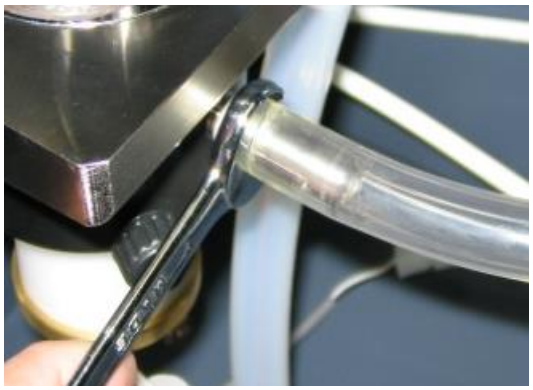
2 Open the BYPASS needle valve on the back of the stand cover and feed 5L/min to 10L/min of argon gas.

Warning

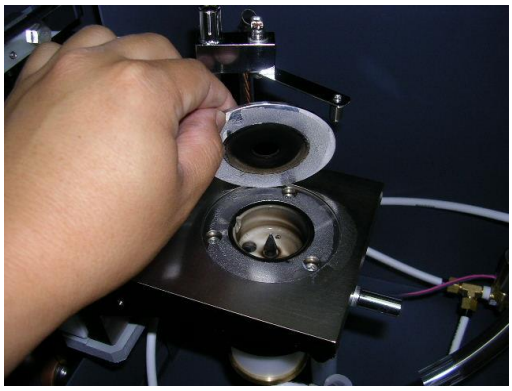
Electric Shock

Never carry out maintenance operations on the spark stand unless the SOURCE switch on the switch panel is turned OFF.

3 Press out the end of the vinyl hose connected to the argon gas exhaust-outlet on the spark stand using a size 11 or 12 hexagonal wrench and remove it.

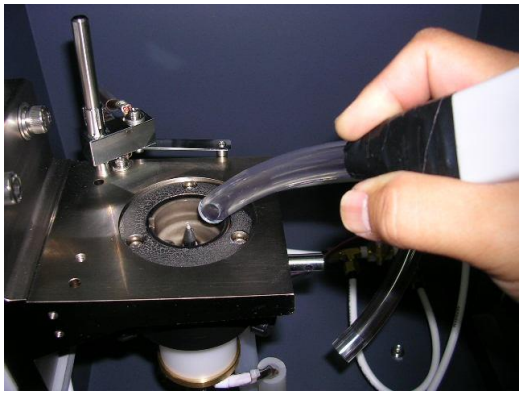


4 Detach the sample plate from the stand base.



5 Wipe off the grease on the stand base surface with gauze.

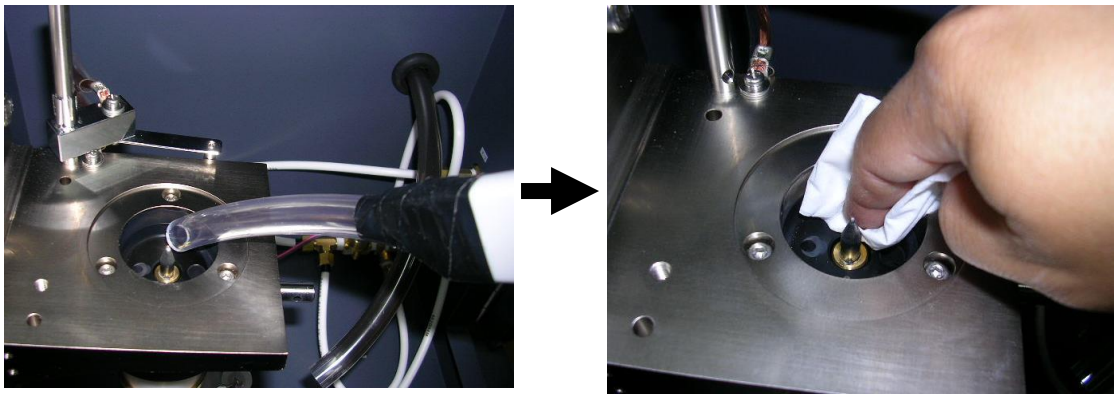
6 Remove evaporant in the dust cup using a vacuum cleaner.



Caution

Before using the vacuum cleaner to clean the emission chamber, be sure to remove the vinyl hose.
If the vacuum cleaner is used without removing the hose, water in the gas bubbler may rise into the stand, and thus could damage the instrument.

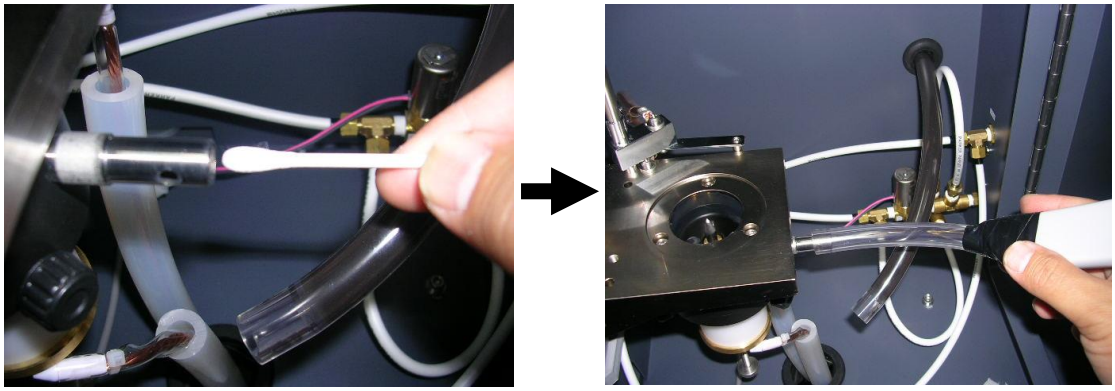
7 Detach the dust cup, remove evaporant under the cup using a vacuum cleaner and dry-wipe the area.



Note

The dust cup is made of ceramics, so dropping it or hitting it against hard objects can damage it.
Also, if a damaged dust cup is used it can cause the analysis results to be abnormal, so under no circumstances should a damaged dust cup be used.

8 Clean the argon gas exhaust outlet with a cotton swab or gauze and then a vacuum cleaner.

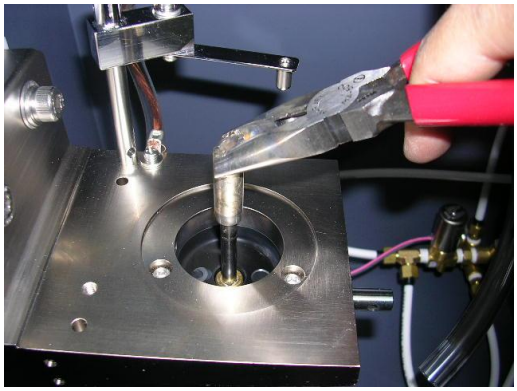


9 Blow off the evaporant in the vinyl hose outside of the room and clean the inside of the hose. If the hose has hardened, replace it with a new one. After cleaning, attach the vinyl hose. Apply a small amount of grease on the outer surface of the exhaust pipe and insert the hose.

Note

- **Improper or loose fitting of the exhaust outlet pipe and vinyl hose may result in analytical value fluctuations. Cut the tip of the hose and use an appropriate part for connection or replace the hose with a new one.**
- **If the exhaust hose is bent, gas cannot flow properly, leading to analytical value fluctuations. Make sure that the exhaust hose is not bent.**

10 Pull out the tungsten electrode straight up using the electrode removal tool and remove it.

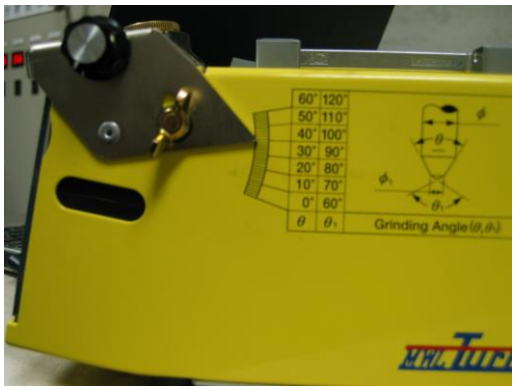


11 Wipe off the grease on the electrode.

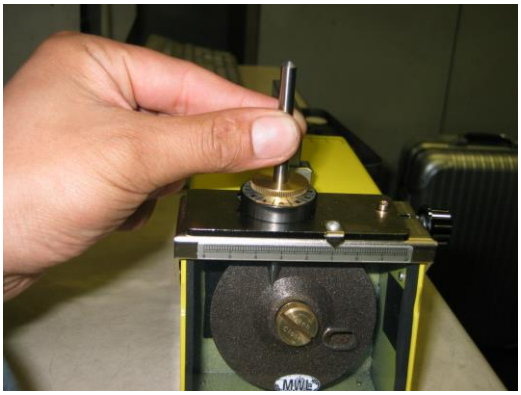
Caution

- **Prior to pulling out the electrode, be sure to clean the emission chamber following steps 1 through 9. Attaching or detaching the electrode when the emission chamber is dirty may damage the electrode O-ring.**
- **Oil has been applied to the electrode removal tool to prevent rust generation. Wipe off the oil before use.**

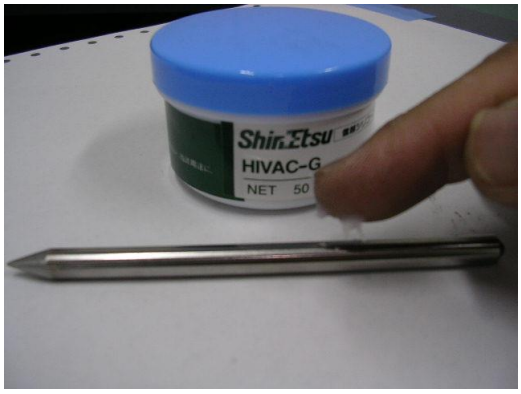
12 Adjust the sharpening angle of the electrode sharpener according to the electrode currently used.



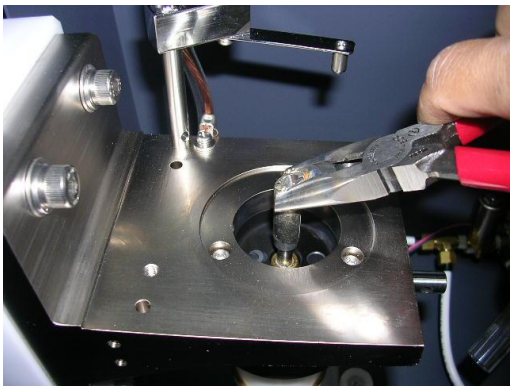
13 Sharpen the tip while slowly turning the electrode.



14 After sharpening the electrode, apply a small amount of grease (just enough to prevent damage on the O-ring when inserting the electrode as described in step 15) on the lower part of the electrode.



15 Using the electrode removal tool, insert the electrode straight down until its bottom contacts the electrode holder.



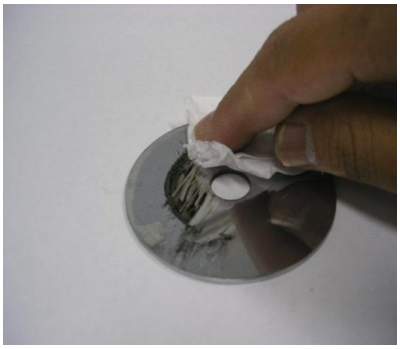
Note

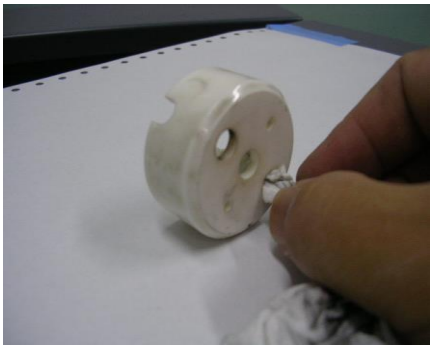
Electrode tip contamination may cause analytical value fluctuations. Handle the electrode with care. Touching the tip with your hand may cause contamination.

16 Clean the sample plate and dust cup.

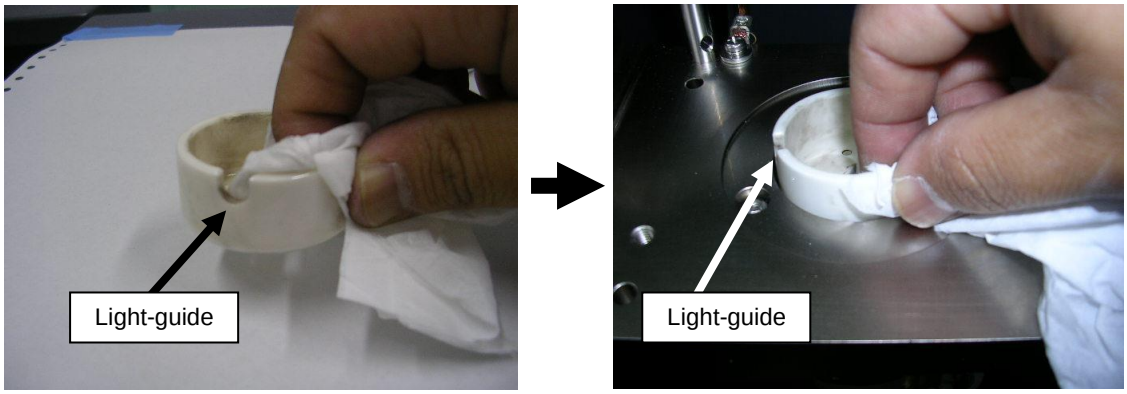
Wipe off dirt on the sample plate using a clean cloth or something similar. Ethanol can be used if necessary. Make sure that the sample plate is completely dry before attaching it.

Wipe off dirt on the dust cup with a dry cloth or something similar.





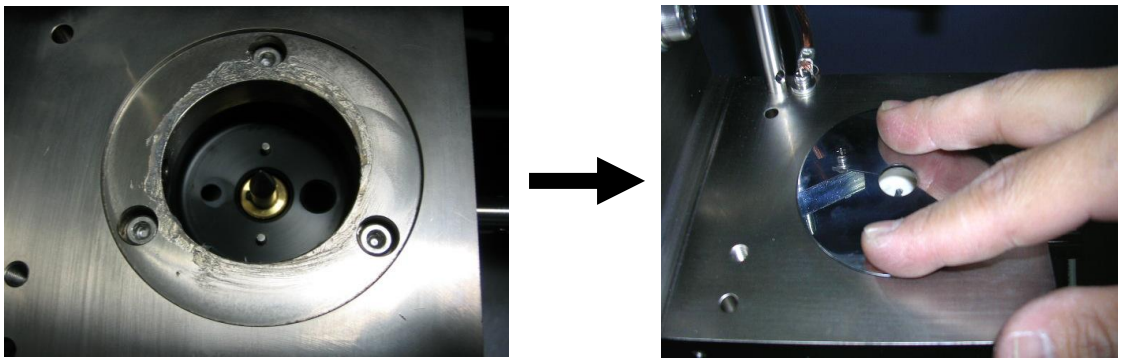
17 Wipe off fingerprints on the dust cup and place the cup onto the stand using care not to directly touching the cup. Place it so that the light-guide (concave part) is on the spectrometer side.



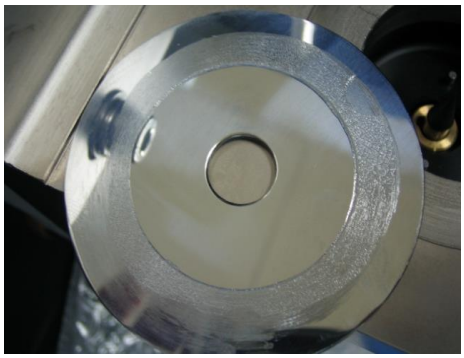
Note

If the dust cup is mounted in the incorrect direction, spectral light cannot go into the spectrometer, inhibiting analysis.

18 Apply a small amount of silicone grease to the inside of the three-point set screw inside the stand and then, turn the sample plate while pressing it down so that the grease settles on the junction surface.



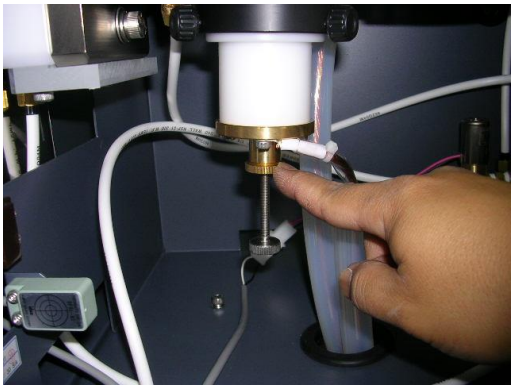
19 Detach the sample plate and confirm that the grease covers the entire joint surfaces of both stand and sample plate. Confirm that the grease is not on areas around the sample plate or the light-guide hole of the stand.



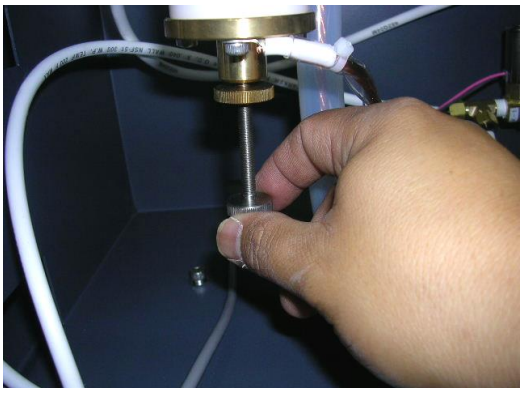
Note

- **The sample plate is a consumable part. Replace it with a new one when its surface is scratched or damaged. Reverse the top and bottom faces of the sample plate every time the stand is cleaned.**
- **Be careful with the amount of silicone grease applied to the joint surfaces of the sample plate and stand. If there is too little, it causes argon gas to leak. If there is too much, it causes abnormal discharge.**
- **Improper or loose fitting of the sample plate and stand may result in lower analytical accuracy.**

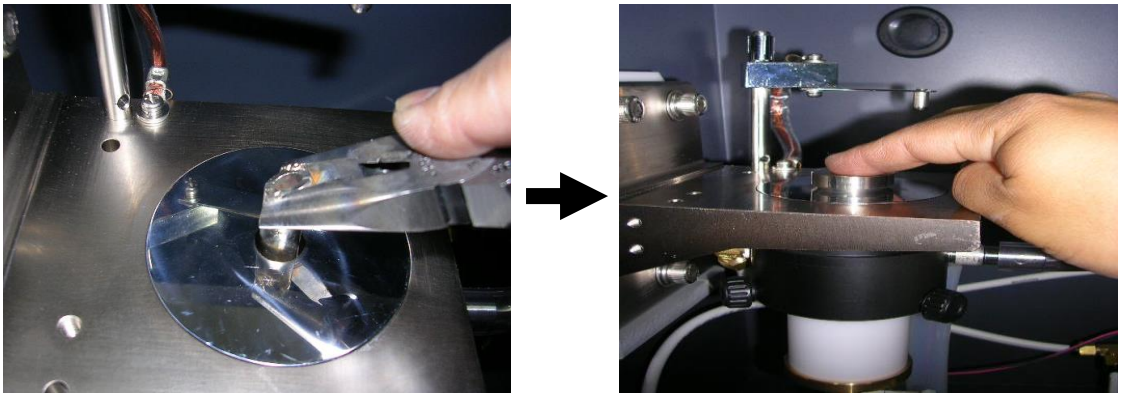
20 Loosen the lock nut of the electrode holder.



21 Loosen the adjusting bolt.



22 Insert the electrode into the holder using the electrode removal tool and press in the electrode using the gap gauge.

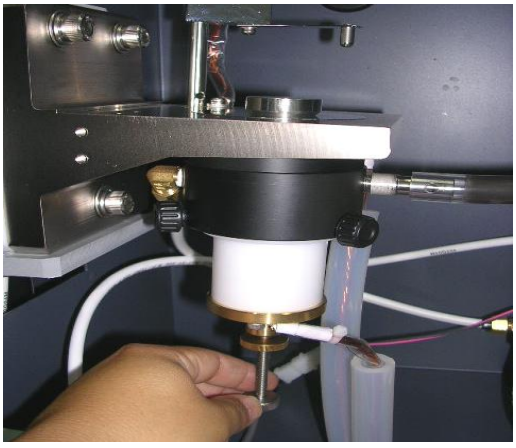


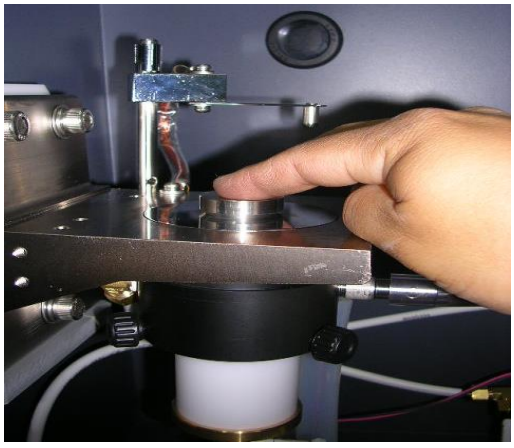
Note

Oil has been applied to the gap gauge to prevent rust generation.
Be sure to wipe the oil off before use.

23 With the gap gauge placed on the spark stand, tighten the adjusting bolt gradually until the tip of the electrode contacts the gap gauge.

24 Press down the gap gauge and confirm that it is not lifted by the electrode.

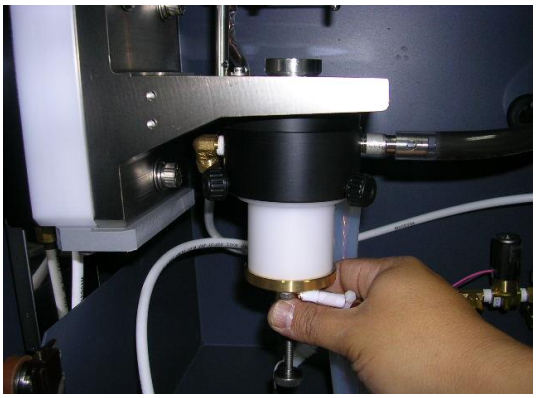




Note

Be sure to adjust the gap in a direction that the electrode moves upward.

25 Tighten the lock nut and fix the electrode position.



26 Place an analytical sample on the spark stand and tighten the BYPASS needle valve to return the argon gas flow rate to the original BYPASS rate.

27 Turn on the SOURCE switch on the switch panel and turn on the power of the excitation unit.

28 In order to even out the tip of the electrode that has been sharpened, recondition the tip using discharge by following the procedure below. With this setting, discharge lasts for approximately two minutes.

(i) Place a sample on the spark stand, select **Preparation** - **Waste Discharge** in the menu to open the **Waste Discharge** screen, and set the condition below on **Condition 2** tab.

No. of emission	**1** time	
Purge time	**3** sec.	
Discharge condition	**SEQ1**	**Cleaning**
	SEQ2	**Cleaning**
	SEQ3	**Cleaning**
	cleaning	**Cleaning**
Discharge time	**SEQ1**	**9999**
	SEQ2	**9999**
	SEQ3	**9999**
	cleaning	**9999**

(ii) Start the discharge by pressing **1:Start** and wait until it is finished.

29 After reconditioning of the tip of the electrode, the total length of the electrode becomes approximately 0.6mm shorter.
Adjust the position of the electrode again following steps 1, 2, 20 to 27.

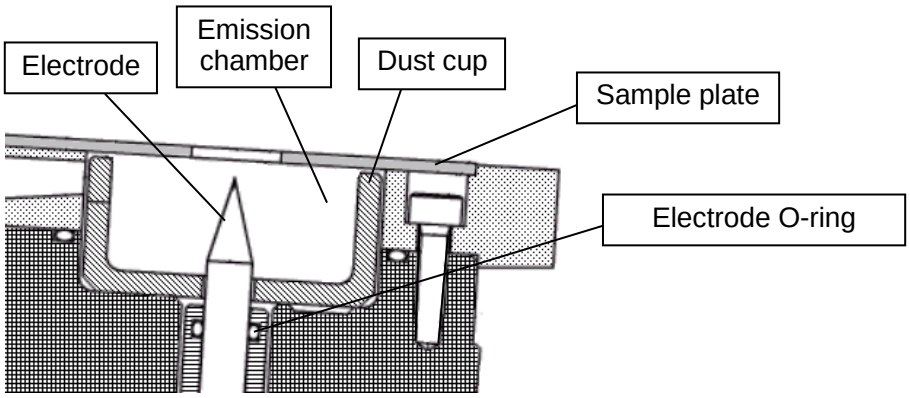
Note

Be sure to readjust the analysis gap after reconditioning the tip of the electrode.

5.4 Exchanging the electrode O-ring

Electrode O-ring deteriorates due to the temperature rise caused by discharge and also by repeated removal and insertion. Although the deterioration level differs according to the usage frequency, replace it with a new one monthly.

1 The electrode should be replaced when it is pulled out. Pull out the electrode following steps 1 through 10 described in "5.3 Maintaining the spark stand".
2 Detach the O-ring using a toothpick or plastic screwdriver. (Do not use a metal screwdriver for watches because it may damage the sealing surface of the O-ring.)
3 Apply a small amount of silicone grease to the new O-ring before attaching it.
4 Fix the electrode following steps 11 through 29 described in "5.3 Maintaining the spark stand". In step 15, insert the electrode while rotating it so that the O-ring can fit on the groove properly.



Cross section through the spark stand

5.5 Cleaning the condenser lens

The condenser lens gradually becomes contaminated due to ultraviolet light. Clean the condenser lens following the procedure below. If you do not provide cleaning at a proper interval, contaminant deposited inside will cause errors in analysis results.

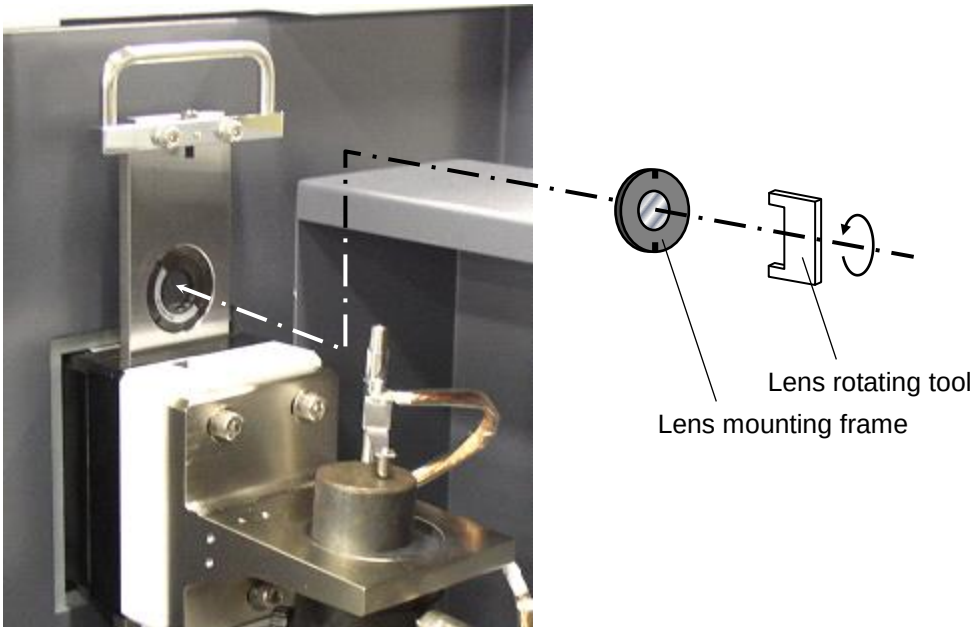
1 Disconnect the vinyl hose for exhausting argon gas from the spark stand following step 3 described in "5.3 Maintaining the spark stand".

Caution

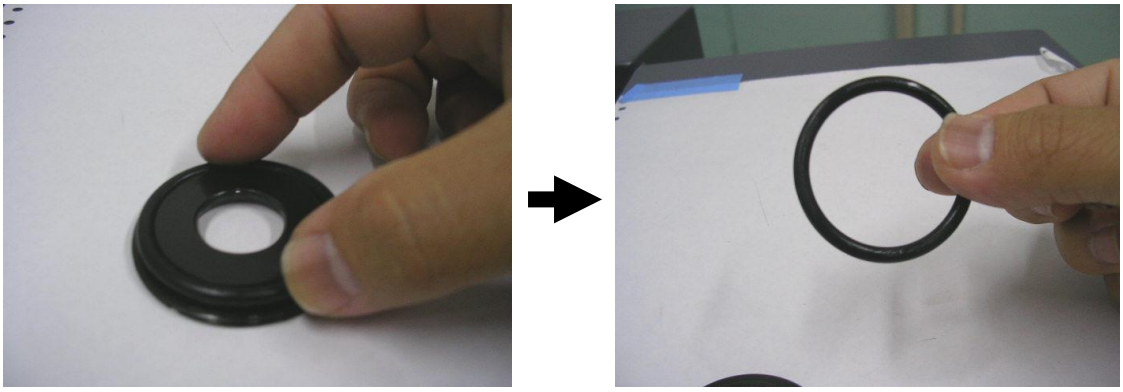
Be sure to disconnect the vinyl hose prior to cleaning the condenser lens, or evaporant in the piping or water in the bubbler may flow back and enter the stand, damaging the instrument.

2 Pull the handle to pull up the condenser lens unit.

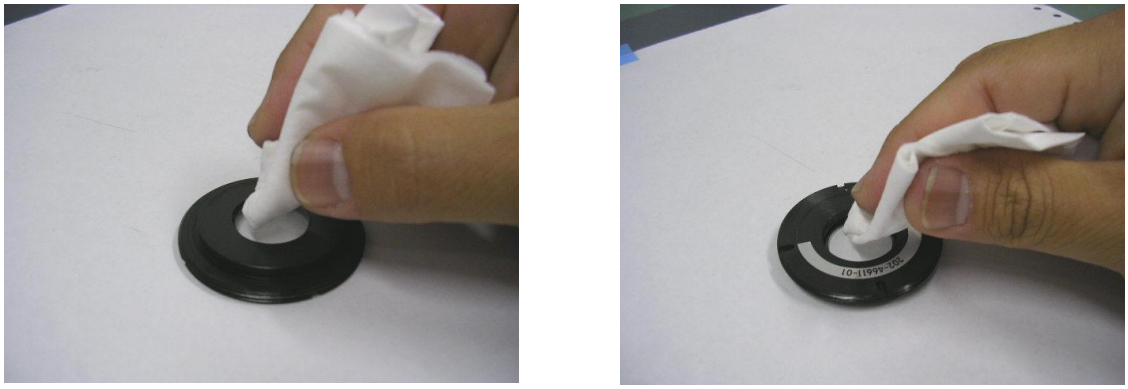
3 Remove the lens mounting frame by rotating it with a lens rotating tool.



4 Place the condenser on a clean paper and detach the O-ring (1A P34).



5 Apply ethanol on gauze and clean both sides of the lens using care not to rub the joint part of the holder and lens.



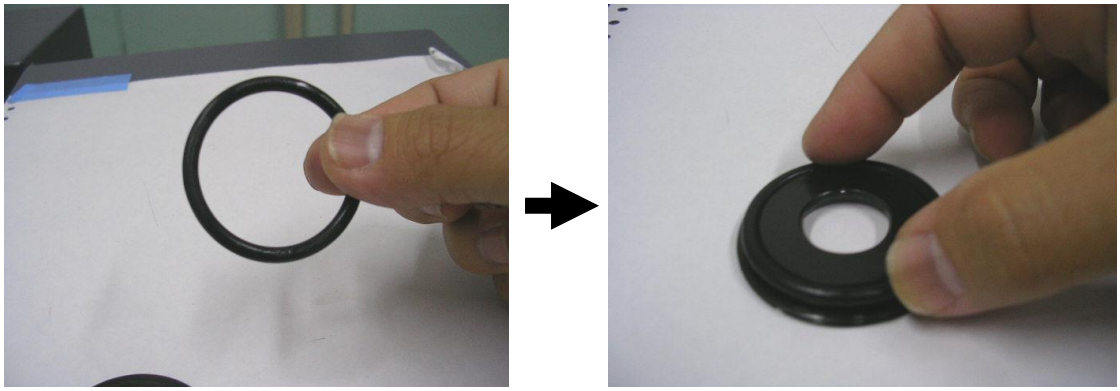
6 In case of heavy contamination, rub the lens with gauze with toothpaste. Then, remove the toothpaste with gauze soaked in ethanol.

Caution
Do not soak the lens in organic solvents such as benzene or paint thinner for a long time or polish the bonding part at the lens end. **It may deteriorate the bonding part and cause poor vacuum condition.**

7 Wipe the lens with dry gauze.

8 Confirm that the lens surfaces are free from any cloudiness. (Be sure to clean both sides of the lens.)

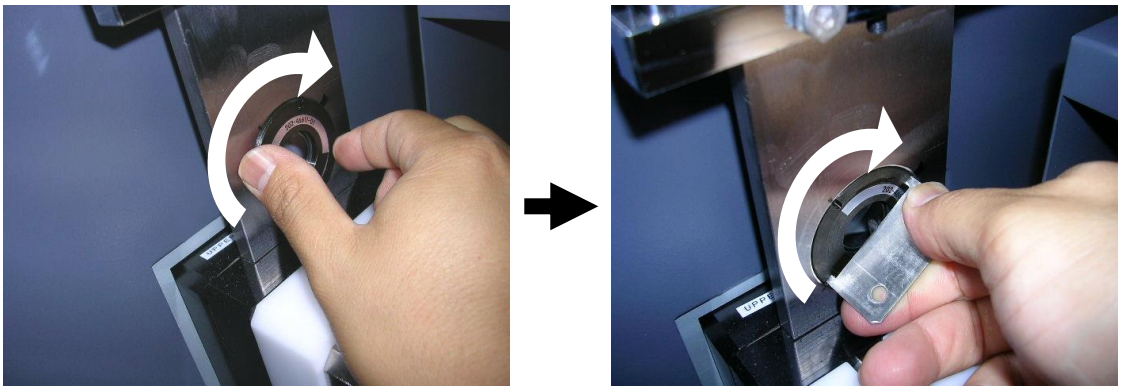
9 Apply a small amount of silicone grease to the O-ring and place it on the lens holder.



Note

Be sure that silicone grease does not stick to the condenser lens, since it is difficult to remove. Be sure your hands are not dirty when cleaning the lens.

10 Return the lens-mounting frame to its original position using the lens rotating tool.



11 Press in the handle of the condenser lens unit to place the lens at its original position.

Caution

Do not press in the condenser lens unit without the condenser lens or without the O-ring (1A P34).
It may break the vacuum inside the spectrometer and contaminated water in the spark stand or bubbler may enter the spectrometer. As a result, the optical units may be contaminated, causing permanent damage to the instrument.

12 Connect the vinyl hose for exhausting argon gas for the spark stand to the stand following step 9 described in "5.3 Maintaining the spark stand".

5.6 Adjusting the control gap

Carry out maintenance for the control gap following the procedure described below.

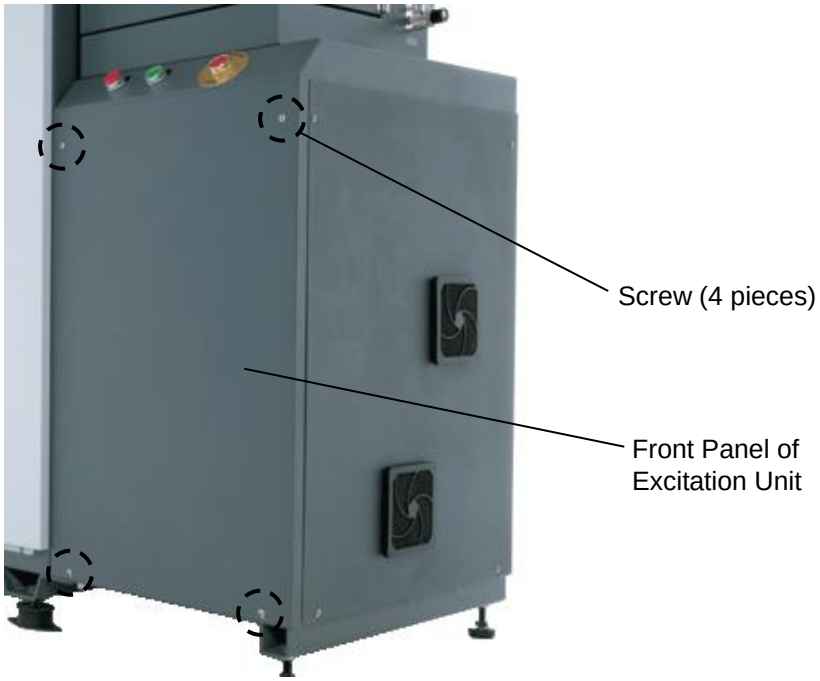
1 Turn OFF the SOURCE switch on the switch panel for safety.

Warning

Electric Shock

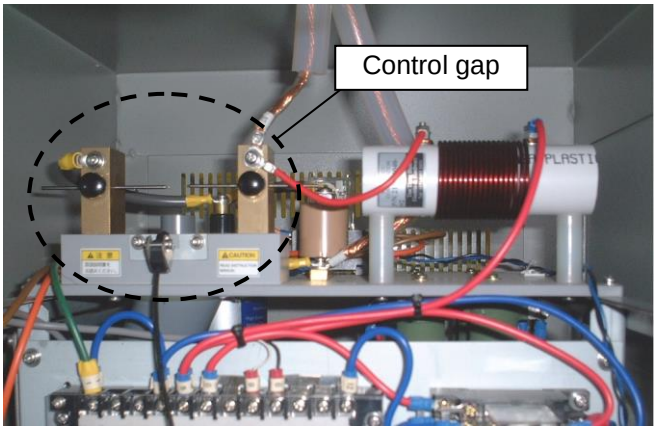
Never carry out maintenance operations unless the SOURCE switch on the switch panel is turned OFF.

2 Detach the front panel of the excitation unit.

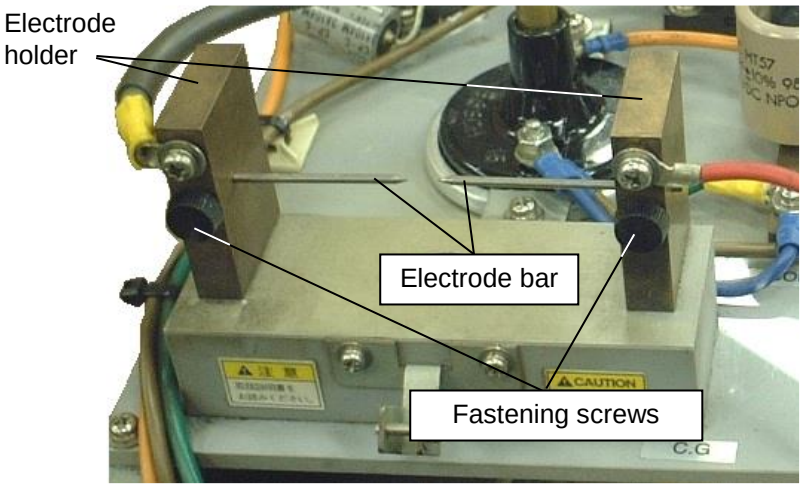


Exterior View of Excitation Unit

3 Loosen the fastening screws on the electrode holder of the control gap in the upper part of the excitation unit to remove the electrode.

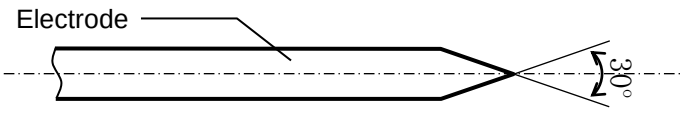


Upper Part of Excitation Unit



Control Gap

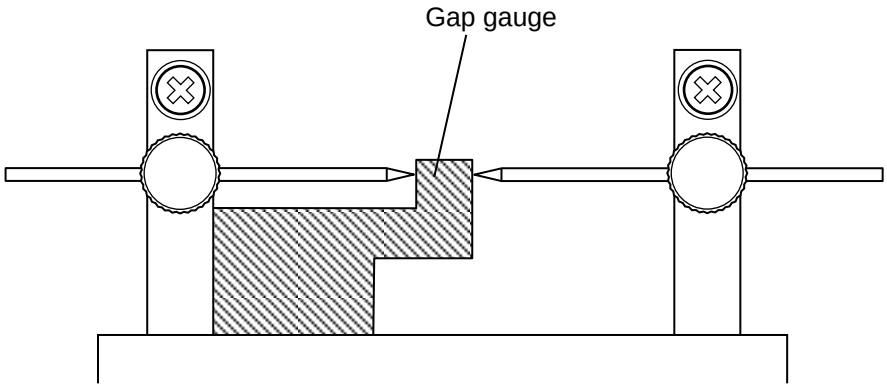
The used electrode should be polished with sandpaper (#240) or machined in a lathe. (It is also possible to use a special electrode sharpener.)



Electrode of Control Gap

4 Place a gap gauge between the electrode holders so that the left side of the gauge would contact the electrode holder.

5 Insert a new electrode (or reconditioned one) into the electrode holder so that the tip would contact the gap gauge. Then, tighten the fastening screw.



How to Adjust Control Gap

6 Take off the gap gauge.

7 Mount the front panel of the excitation unit.

8 Turn ON the SOURCE switch on the switch panel.

Caution

- **If the gap gauge is used upside down, control gap becomes too large, generating abnormal voltage. It may cause dielectric breakdown on the igniter or damage the igniter coil.**
  Be sure to use the gap gauge in the correct direction. (Control gap should be 7mm)

- **Do not make discharge with the gap gauge on.**

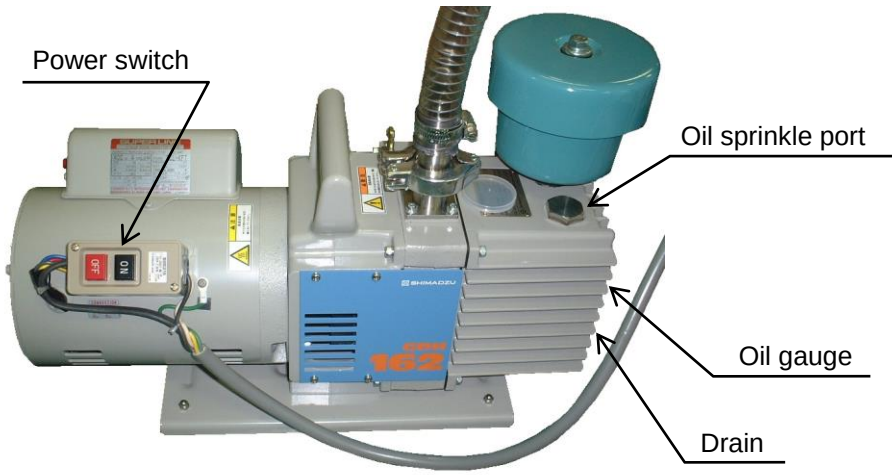
5.7 Replacing pump oil

Replace the vacuum pump oil following the procedure described below.

1 Turn OFF the power switch of the vacuum pump.

2 Open the pump leak valve, allow the air into the suction hose.(Refer to 3.5)

3 Loosen the drain cock to drain the oil. While doing this, rotate the vacuum pump for a short time (repeat a few seconds of ON and OFF several times) to prevent contaminated oil from staying in the pump.



4 After draining the oil, close the drain cock.

5 Open the oil filler port and fill new oil. While doing this, rotate the vacuum pump for a short time, as described above, to let the new oil go over entire inside of the pump. Fill the new oil until the oil surface reaches within the red circle at the center of the oil gauge.

6 Close the oil filler port.

7 Turn ON the power switch of the vacuum pump.

Caution

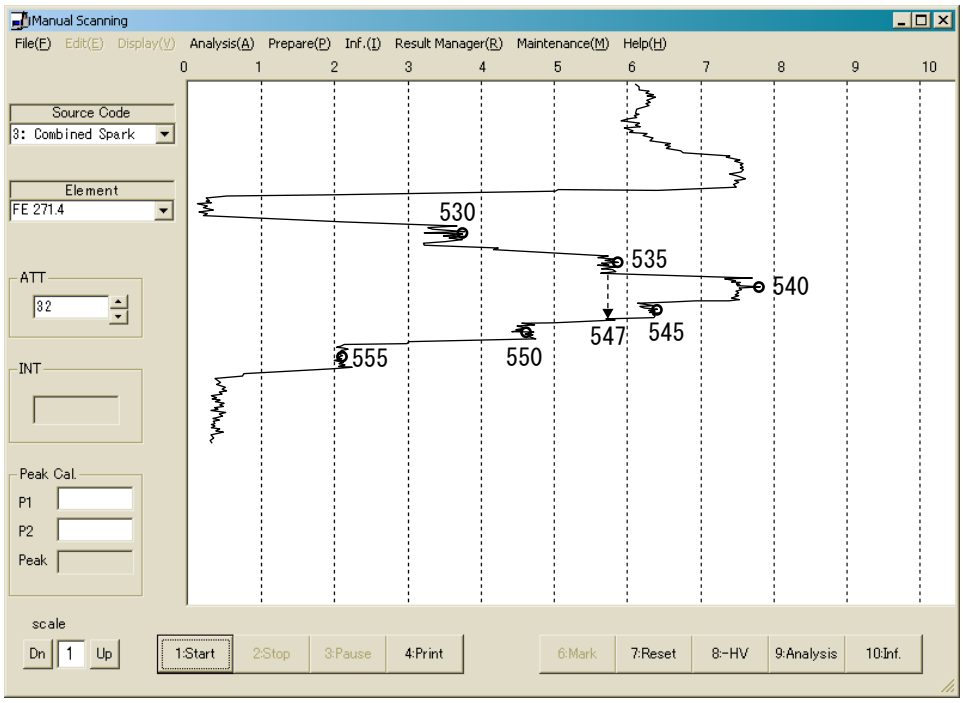
- **Do not rotate the vacuum pump longer than 2 minutes during the oil replacement. It may damage the exhaust valve.**
- **Check that the oil level of the vacuum pump is within the red circle of the oil gauge while the pump is running.**
- **When the temperature is low such as in winter, make sure that the room temperature is high enough before turning ON the power of the pump.**

5.8 Manual scanning

The position of the peak of each spectrum may shift during the long term of operation, due to the changes of the environment or the instrument itself, such as the drift of the temperature inside the spectrometer.

On **Manual Scanning** screen, the peak position can be adjusted at the right position by moving the entrance slit using the scanning dial.

1 Select **Prepare** - **Manual Scanning** on the menu bar.



Screen 5.1 Manual scanning

2 Set the sample on the spark stand and hold it by the sample holder.

3 Start the discharge by pressing **1:Start** or the start button on the spark stand. The discharge begins, and the intensity profile starts being displayed.

4 Change the sensitivity value using arrow keys or by directly inputting numerical values, and adjust the intensity to about "8" on the horizontal axis.

5 Unlock and turn the scanning dial in minus direction (counter-clockwise) to remove backlash (see the explanation on the next page), and adjust the dial to a round number.

6 Wait about 1 second until the intensity becomes stable.

7 Turn the dial in plus direction (clockwise) by 5 graduations, press **6:Mark** when the intensity becomes stable to put “O” mark on the profile. Take notes of the dial graduation.

8 Repeat procedure 7 until the intensity matches the background intensity.

9 Press **2: Stop** to stop the discharge.

10 Calculate the number to set the scanning dial at, from the profile curve on the screen.

For example, the calculation in the case of the profile on the previous page is as follows.

$$\text{Dial Number} = \frac{535 + 547}{2}$$

$$= 541$$

535 : The dial number right before the intensity hits the maximum.

547 : The estimated dial number at the intersection of the profile curve and a perpendicular line from the point above. (in this case 535)

11 Set and lock the scanning dial at the number calculated above.

Be sure to remove the backlash by the way described below.

Removing backlash

When turning the dial largely, first turn it to a value 20-30 lower than the target value. Then turn the dial clockwise slowly to adjust at the intended position.

Note

- **For elements with profile curve peaks which cannot be scanned every 5 graduations, such as internal standard elements Al 237.2nm, Al 266.0nm, and Pb 322.0nm, calculation error may occur and accurate peak values may not be obtained. In this case, create profiles using a sample with content of 0.5 to 1% at Cu 327.4nm of the analysis line, and set the scanning dial to the peak value calculated with the analysis line.**
- **When the calculated dial number is deviated more than ±2scales from the original position, retry manual scanning.**

Caution

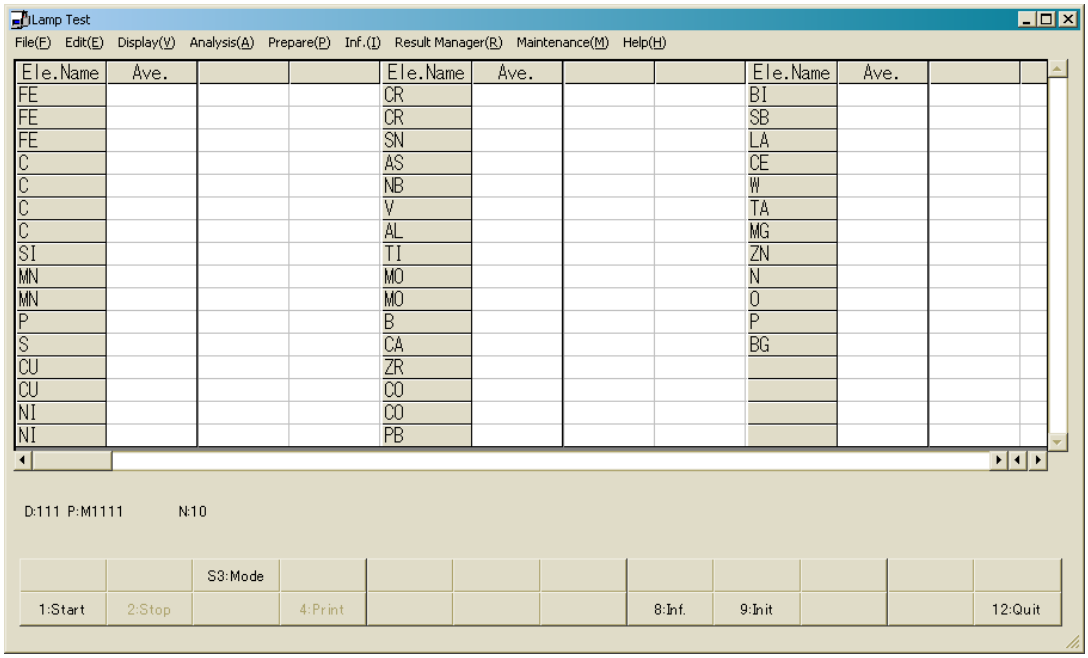
Do not turn the scanning dial before unlocking .
It may damage the scanning dial.

5.9 Lamp test

To check if the read-out unit works correctly, perform Lamp Test. You can get the stability of the entire read-out unit, including the photomultiplier (photo detector), by Lamp Test, based on the reproducibility of the measured intensity.

The following describes the procedure of Lamp Test. Confirm that CV value of every element is equal to or less than 0.1 after the test.

1 Select **Maintenance** - **Lamp Test** on the menu bar.



Screen 5.2 Lamp Test

2 Start the measurement by pressing **1:Start** or the start button on the spark stand.

3 Measurements of specified times (10 times by default) are automatically carried out and the result is calculated

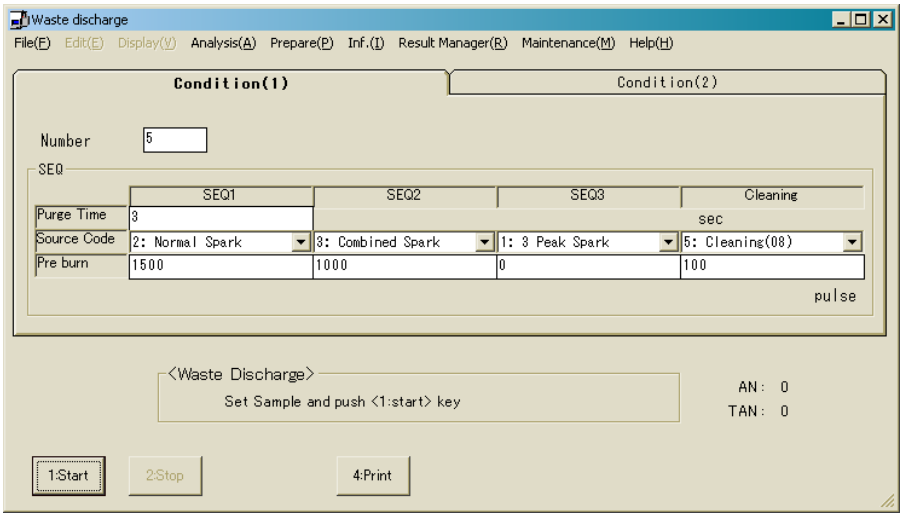
Confirm that CV value for every element is 0.1 or less, except for elements for which gas-type or higher-order-type photomultiplier is used.

Note
Execute lamp test at least 30minutes after Lamp Test window is opened.

5.10 Waste discharge

After the maintenance of the instrument, carry out waste discharge to stabilize emission.

1 Select **Prepare** - **Waste Discharge** on the menu bar.



Screen 5.3 Waste discharge

2 Set the sample on the spark stand and hold it by the sample holder.

3 Start the discharge by pressing **1:Start** or the start button on the spark stand.

4 After repeating the discharge specified times, **Content Analysis** screen is displayed automatically.

Note

Below are the recommended conditions for waste discharge.

No. of emissions: 5 times
(The number can be set between 5 and 20 according to the discharge conditions. Increase the number for poor discharge conditions.)
Purge time: 3 sec.
Discharge condition: SEQ1 Normal Spark
SEQ2 Combined Spark
Discharge time: SEQ1 1,500 pulses
SEQ2 1,000 pulses
Cleaning: Cleaning → 100 pulses

5.11 File utility

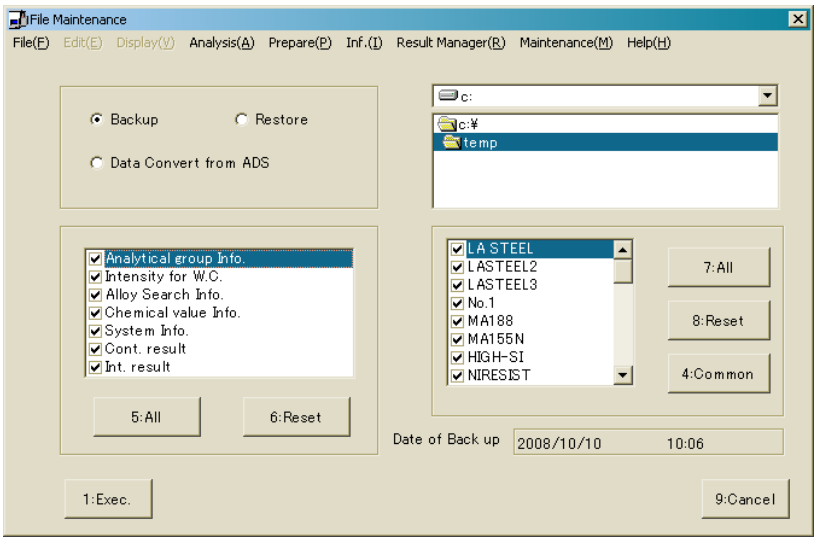
Important data stored in the computer may be lost due to an unexpected accident. If this happens, only the data processing software PDA-Win can be recovered with the installation CD, which is provided with the instrument. Information files (analysis information and other information) and data report files (analysis results) that were made or changed after the shipment from the factory cannot be recovered.

To prevent the loss of important data, it is strongly recommended to backup information and data report files onto removable media regularly, using **File Maintenance** window.

(1) Saving (Backup)

The procedure of how to backup analysis information files is described here. Data report files also can be backed up by the same procedures.

1 Select **Maintenance** - **File Maintenance** on the menu bar.



Screen 5.4 File Maintenance (Backup is selected)

2 Select **Backup** in the upper left area on the screen.

3 Select the backup target items in the lower left area. Press **5:All** if you would like to backup all the target items.

4 Specify the drive and the directory in which data backup files will be stored in the upper right area.

5 Press **1:Exec.** to start the backup process. Follow the indication message displayed on the screen.

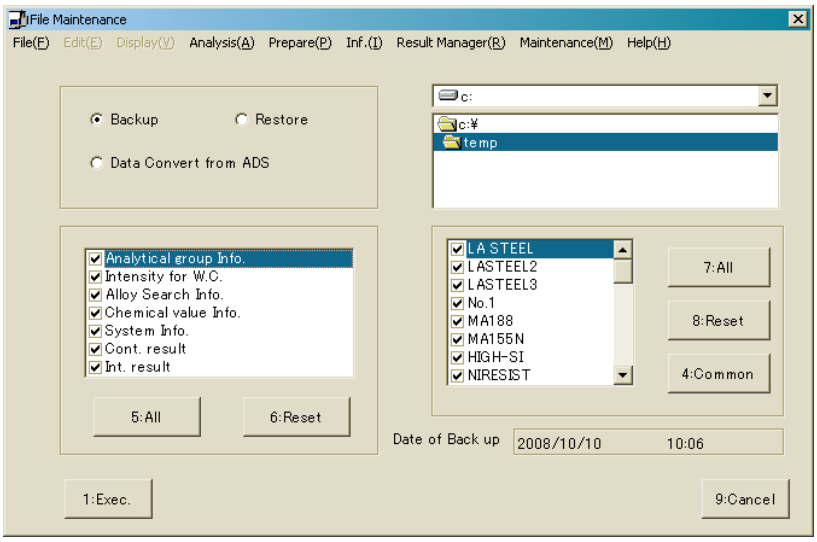
Note

- **Usually, the backup process is completed within one minute.**
- **Put a label indicating the contents on the backup media.**

(2) Loading (Restoration)

The procedure of how to restore the backup data is described here..

1 Select **Maintenance** - **File Maintenance** on the menu bar.



Screen 5.5 File Maintenance (Restore is selected)

2 Select **Restore** in the upper left area on the screen.

3 Select the restoration target items in the lower left area. Press **5:All** if you would like to backup all the target items.

4 Specify the drive and the directory in which data backup files are stored, in the upper right area. All the backup files of restoration target selected above should exist there.

5 Press **1:Exec.** to start the restoration process. Follow the indication message displayed on the screen.

5.12 Replacing recalibration samples

When the recalibration sample gets deteriorated and analysis cannot be performed, replace it with a new one following the procedure described below.

1 Select **Inf.** - **Analytical Inf.** and open **Recalibration Information** screen. Then, print out the recalibration information of the analytical group with which recalibration samples will be replaced.

2 Carry out the below.
   (1) Carry out maintenance of the spark stand. (“5.3 Maintaining the spark stand”)
   (2) Clean the condenser lens. (“5.5 Cleaning the condenser lens”)
   (3) Adjust the control gap. (“5.6 Adjusting the control gap”)
   (4) Replenish the argon gas bubbler with water. (“4.1 Argon gas bubbler”)
   (5) Adjust the entrance slit. (“5.8 Manual scanning”)
   (6) Carry out waste discharge (“5.10 Waste discharge”) 30 times.

3 Carry out 2-point recalibration analysis using the recalibration sample before replacement on **Recalibration** window.

4 If the components of the new recalibration sample are the same as those of the sample currently used, carry out intensity reference value analysis for the recalibration on **Int. for target** window. The reference value is automatically registered.

5 If the components of the new recalibration sample are different from those of the sample currently used or the former sample is replaced with a different type of recalibration sample, analyze the new recalibration sample on **Drift Corrected Intensity Analysis** window.

   Then, register the name of the new standard sample and the intensity value after the drift correction obtained through the analysis above in **Sample Name** and **Target** sections respectively **Recalibration Information** window.

6 Carry out recalibration using the new recalibration sample and check recalibration coefficients α, β and k. (“4.7 Recalibration”)

   If necessary, carry out master curve correction as well. (“4.8 Master curve recalibration”)

7 Carry out check analysis using the check sample and confirm that the analytical values are normal (“4.9 Check analysis”)

8 After checking the analytical values, back up the analysis information. (“5.11 File utility”)

9 Store all the old recalibration samples that can be no longer used.
Do not discard them.

10 Save data of analytical values and screen copies obtained through recalibration sample replacement by the procedure above.

5.13 Replacing argon gas cylinder

If air enters the piping during argon gas replacement, the argon gas purity in the piping lowers due to contamination. In this condition, it takes longer for analytical values to stabilize. Use the procedure described below to replace the argon gas.

(For numbers (1) through (3) in the procedure, see the reference piping diagram on the next page.)

1 Close the main valve (2) on the piping of cylinder No.1 to be replaced.

2 Replace cylinder No.1 with a new one and open the valve of the pressure regulator (1).

3 Open and close the purge valve (3) two or three times to release contaminated gas from the piping.

4 Open the main valve (2).

Note

- **When nitrogen is contained in the element to be analyzed, pay extra attention while changing argon gas.**
- **When changing a gas cylinder, use care not to allow dust or debris to enter the piping.**

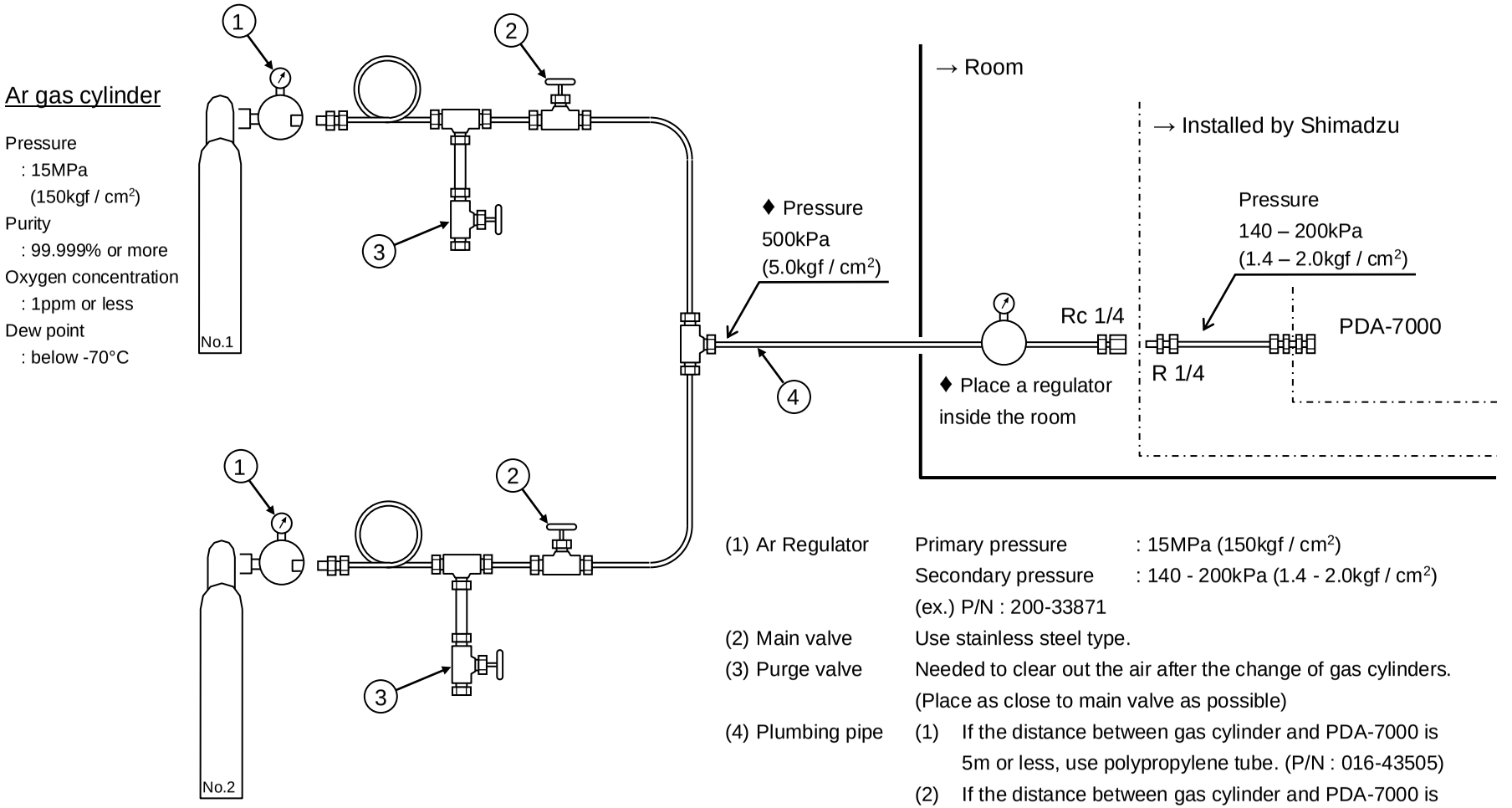
5.14 Periodic inspection

To use the instrument safely and correctly, the periodic inspection is needed.

For the details of the periodic inspection, contact your Shimadzu service representative.

5.15 Cleaning the temperature control fan heater

The temperature control fan heater needs cleaning depending on the conditions of the use. Ask a Shimadzu service engineer to clean it at least once a year.



(1) Ar Regulator
Primary pressure : 15MPa (150kgf / cm^2)
Secondary pressure : 140 - 200kPa (1.4 - 2.0kgf / cm^2)
(ex.) P/N : 200-33871

(2) Main valve
Use stainless steel type.

(3) Purge valve
Needed to clear out the air after the change of gas cylinders.
(Place as close to main valve as possible)

(4) Plumbing pipe
(1) If the distance between gas cylinder and PDA-7000 is 5m or less, use polypropylene tube. (P/N : 016-43505)
(2) If the distance between gas cylinder and PDA-7000 is 5 - 40m, set the secondary pressure to 500kPa (5kgf / cm^2) and place a regulator inside analysis room to set the pressure at PDA-7000 gas inlet 140 - 200kPa (1.4 - 2.0kgf / cm^2), as described with ♦ in the figure.
> Use stainless steel plumbing pipes for high purity gases.
> For the details of the plumbing such as the thickness of the pipe, consult a plumber company.

Cylinder replacement procedure

(1) Close main valve (2) connected to the cylinder to be replaced.
(2) Replace the Ar gas cylinder and open the valve of regulator (1) .
(3) Open and then close purge valve (3) several times, to clear out the air and contamination.
(4) Open main valve (2) .
Take special care if nitrogen is to be measured.

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Chapter 6 Installation

6 Installation

Before installing PDA-7000, carefully prepare the location of the installation and the configuration of the peripheral equipment by referring the following guidelines.

6.1 Temperature and humidity

- The installation site should be air-conditioned and meet the following conditions.

  Temperature 10 - 28°C (variations within ±5°C / hour)
  Humidity 15% - 70%RH (no condensation)

- Instruments should not be exposed to direct sunlight.
- PDA-7000 system generates about 1,100kcal / hour of heat.

  To estimate the total heat release, the heat generated by operators and the room light also should be considered.

6.2 Vibration

- The installation site should meet the following conditions.

  Acceleration less than 0.2G
  Amplitude less than 80μm
  Velocity less than 0.8cm / sec

6.3 Dust

PDA-7000 uses high voltages and tiny electrical currents. Therefore, dust can cause it to malfunction. The installation site should be as free as possible of dust.

To prevent dust, implement dust-prevention measures in the doors and windows of the room where the system is installed.

6.4 Grounding

- If the grounding is shared with other electrical or electronic devices, the spark discharge noise may cause them to malfunction. Therefore, provide an independent grounding.
- The grounding condition should meet the following requirement.

  Grounding resistance less than 30Ω, independent grounding
  (if possible, less than 10Ω, independent grounding is highly recommended)

- If the distance between the instrument and ground point is too far, it will increase the chance of generating the noise. Therefore, keep the length of the grounding wire as short as possible. Also, the grounding wire should be as thick as 8mm^2 (AWG#8) or more. (14mm^2 (AWG#6) or more is recommended.)

6.5 Power supply

- The power supply should meet the following requirement.

Voltage	single phase 200V/220V/230V±10% or 240V±5%
Frequency	50Hz / 60Hz
Power	4kVA

- If the power source is shared with other electrical or electronic devices, the spark discharge noise may cause them to malfunction. Therefore, if possible, provide an independent power supply.
- Put a power switchboard between the power source and the instrument so that the power can be turned off as needed.

6.6 Argon gas

- The argon gas should meet the following requirement.

Purity	99.999% or more
Oxygen concentration	1ppm or less
Dew point	below -70°C

- 140 - 200kPa (1.4 - 2.0kgf / cm^2) of pressure is required at PDA-7000 gas inlet.
  If the argon gas is provided from a gas cylinder, a pressure regulator is needed.
  (Ex.) Ar gas regulator P/N : 200-33871
  Secondary pressure: 150kPa(1.5kgf / cm^2)
- Plumbing pipes for high purity gases should be used. When the argon gas cylinder is placed outdoors or 5m or more away from the instrument, use stainless steel pipes.
- Attach a purge valve to the argon gas piping to clear out the air after the change of gas cylinders.
- To prevent loss of pressure, thicker plumbing pipes are recommended.
- For the details of the plumbing, consult a plumber company.

6.7 Exhaust equipment

- Exhaust the oil mist from the vacuum pump to outdoors, or use oil mist separator.

(Ex.)	Oil mist separator	P/N 040-82714-31
	Socket adapter FCMB1	P/N 035-42121 (needed to attach the oil mist separator to the vacuum pump)

- Exhaust the argon gas emitted from the outlet of gas bubbler to outdoors using exhaust systems.

• If the sample is lead, tin or zinc-based, connect the gas outlet on the back of spark stand case to exhaust system, and exhaust the air inside spark stand case to outdoors.

6.8 Peripheral equipment

• Table
• Shelf
• Desiccator
• Thermometer and Hygrometer
• Belt sander / Lathe
• Sample pretreatment system

(Ex.)	Belt sander FS-3N	P/N 085-50206-11	50Hz type
	Belt sander FS-3N	P/N 085-50206-12	60Hz type
	Bench lathe L-1000	P/N 085-50102-01	

• Electrode grinder

(Ex.)	Electrode grinder MT-11M	P/N 085-50802-01	AC100V type
	Grinding wheel CBN75#100	P/N 085-50802-51	

• Electrode grinder (for control gap electrode ø2mm)

(Ex.)	Electrode grinder MT-10M	P/N 085-50801-01	AC100V type
	Grinding wheel CBN60#170	P/N 085-50801-11	

6.9 Other environmental requirement

• The installation site should be free of corrosive or explosive elements.
• Avoid use near equipment that generates powerful magnetic fields such as an electric furnace.
If the installation site is near such equipment, contact your Shimadzu service representative.

6.10 Moving and Disposing of the product

Contact your Shimadzu service representative before moving or disposing PDA-7000.

Chapter 7 Troubleshooting

7 Troubleshooting

7.1 Basic items to be checked

Sometimes, problems are caused by a simple cause. If a problem happens, check the followings first.

Are the power of every unit ON?

Referring to "4.2 Checking gas flow rate", check if the power supply of the spectrometer main unit and the vacuum pump are turned on. If they are not powered even by the correct operation, check the distribution panel which supplies the power to the instrument.

Is the photo detector ON?

If you carry out an analysis with the photo detector OFF, the error message will appear. Turn ON the photo detector on **Instrument Check** screen.

Is the internal pressure of the spectrometer ok?

Check if the internal pressure of the spectrometer reaches the sufficient level on **Instrument Check** screen.

Is the temperature of the spectrometer ok?

Check if the temperature display on the indicator on the switch panel stands within the range of 40±1°C and the set temperature is 40°C.

Is the spark stand door correctly closed?

If the spark stand door is not correctly closed, the safety circuit works, stopping the discharge. Check that the spark stand door is correctly closed.

Is the sample correctly set with the sample holder?

If the sample is not correctly set, the air will flow into the spark stand. This may cause an abnormal discharge. Check if the sample is correctly held by the sample holder.

Are the purity and the flow rate of the argon gas correct?

Argon gas to be used must conform to the following requirements.

Purity	:	99.999% or more
Flow rate	:	10L / min. during analysis (discharge)
		1L / min. during standby
Oxygen	:	1ppm or less
Dew point	:	below -70°C

7.2 Troubleshooting

When all the basic check items listed on the previous page are OK, take a corrective action on the list below. If the same problem still remains after that, or a problem other than the ones described below happens, contact Shimadzu's service engineer.

Problem	Corrective action
The Vacuum does not become OK.	It is suspected that the mounting of the O-ring around the condenser lens is not correct, or the pump oil of the vacuum pump is contaminated, resulting in the deterioration of the exhaust ability. Referring to "5.7 Replacing pump oil" and "5.5 Cleaning the condenser lens", carry out the maintenance and inspection of the vacuum pump and condenser lens.
Temperature of the spectrometer is unstable, or does not reach the set temperature.	It is suspected that the room temperature is not normal, or the fan which supplies the hot air has a problem. Check that the room temperature is within 10 - 28°C and reset the AIRCON switch on the switch panel.
Analysis accuracy is poor.	It is suspected that the argon gas leaks during analysis or contaminant is deposited on the electrode. Check if the sample is set correctly, and carry out an analysis again. If the analysis accuracy is not recovered even after that, refer to "5.3 Maintaining the spark stand" to "5.6 Adjusting the control gap" to carry out maintenance.
The buzzer on the temperature indicator sounds. (It sounds when the temperature inside the spectrometer exceeds 42°C.)	It is suspected that the room temperature is not normal, or the temperature controller has a problem. Check that the room temperature is within 10 - 28°C. Turn OFF the AIRCON switch on the switch panel. Wait until the temperature reading goes down to the room temperature, and turn ON the AIRCON switch again.

7.3 Installing the software

Unexpected troubles or accidental miss-operation may destroy the data processing system stored in the computer. In such a case, recover it using the software installer, which consists of one CD-ROM, provided with PDA-7000.

Foer the recovery procedure, refer to the installation manual below.

P/N	Part name
211-78114	Data Processing software PDA for Windows Installation Manual

By the procedure above, the data processing software will be recovered. However, at this point, the analysis information and results have been initialized, and analysis information and results made or modified after the shipment from the factory have been deleted. Referring to "5.11 File utility", restore the analysis information and data report files.

To prevent the loss of important data, it is recommended to backup information and data report files onto removable media regularly.