

🕀 SHIMADZU

Shimadzu Atomic Absorption Spectrophotometer AA-7000 Series

Instruction Manual

Read this manual thoroughly before you use the product. Keep this manual for future reference. This page is intentionally left blank.

Introduction

Read this Instruction Manual thoroughly before using the product.

Thank you for purchasing this product. This manual describes the operation, hardware validation, 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. Original version is approved in English.

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, re-installation (after the product is moved), or repair 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.
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Safety Instructions

- To ensure safe operation of the instrument, read these Safety Instructions carefully before use.
- Observe all of the WARNINGS and CAUTIONS described in this section. They are extremely important for safety.
- In this manual, warnings and cautions 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.	

Application Precautions

WARNING This instrument is an atomic absorption spectrophotometer. Use this instrument ONLY for the intended purpose. Using this instrument for any other purpose could cause accidents.

Installation Site Precautions

WARNING

- This product is not an explosion-proof product. It ignites and causes a fire. Provide fire extinguishers for use in case of fire.
- Be sure to provide a duct made of metal for the expulsion of combustible gas above the atomic absorption spectrophotometer.

If you use a duct made of plastic it will burn due to the heat of the flame.

The weight of the body of the product is 76 kg. Take the weight of the entire analysis system into account when installing the product.

Install this product on a dealer stand that can accill the unitable of the entire analysis system and

Install this product on a desk or stand that can easily bear the weight of the entire analysis system and is flat and stable.

If these conditions are not met, accidents in which the product topples over or falls may occur.

CAUTION

- Avoid installing the product in locations that are exposed to corrosive gases, organic solvents, halogenated substances, gases containing polysiloxanes, oil mist, or a lot of dirt and dust. This may make it impossible to maintain the product's performance or shorten the life of the instrument.
- **Do NOT use the instrument in an environment where dew may condense on it.** This may prevent its normal operation.

Installation Precautions

WARNING

- To ensure safe operation, contact your Shimadzu representative if product installation, adjustment, or re-installation (after the product is moved) is required.
- Take measures to prevent the instrument from falling in the event of an earthquake or other disaster.

Strong vibrations could cause the instrument to fall over, resulting in injury.

- The power supply voltage of the instrument is indicated on the label on the right side of the instrument. Connect the instrument only to a power supply of the voltage indicated; otherwise, fire or electric shock could result. Check that the power supply voltage is stable and that its current capacity is sufficient to operate all the components of the system. If not, the instrument will not operate at its rated performance.
- Ground the instrument.

Grounding is necessary to prevent electric shock in the event of an accident or electrical discharge, and important for ensuring stable operation.

- Do NOT allow the grounding conductor of the adaptor to set in or contact the power outlet. This could cause fire or electric shock.
- Connect the instrument to a power supply that complies with the capacity and use a power cord that complies with the capacity.
 Insufficient capacity could cause fire, electrical shock or malfunction.
- Do NOT place heavy objects on the power cord, and keep any hot items away. The cord could be damaged, resulting in fire, electrical shock or malfunction. If the cord becomes damaged, contact your Shimadzu representative immediately.
- Do NOT modify the cord in any way. Do NOT bend it excessively or pull on it. The cord could be damaged, resulting in fire, electrical shock or malfunction. If the cord becomes damaged, contact your Shimadzu representative immediately.
- Please insert the power cord in the power outlet at an easily accessible position. The power cord must be unplugged from the power outlet in case of emergency.

CAUTION

- When installing the instrument, be careful not to pinch your fingers between the system components, as this could result in injury.
- When opening the doors to the hollow cathode lamps, be careful not to pinch your fingers as this could result in injury.
- Ground the PC and the optional device with equal potential grounds. Otherwise communications between devices may be interrupted and equipment failures could occur.

Precautions on Work and Operation

WARNING

• This instrument should be used only by personnel who have undergone special education and training.

"Special education and training" indicates studies including the following contents:

- 1. Information relating to high-pressure gas and the handling of this unit
- 2. Information relating to high-pressure gas supply equipment

WARNING

- Do NOT use flammable sprays (hairspray, spray insecticides, etc.) near this product. The spray could ignite and cause fire.
- Be sure to ventilate the room while using the instrument. The acetylene gas used with the atomic absorption spectrophotometer is inflammable. It could ignite and cause fire.
- When measuring an inflammable sample, take care about the handling of naked flame. The material could ignite and cause fire.
 Be sure to ventilate the room while using the instrument.
- Use the rubber hose supplied as a standard accessory with the product to supply gas. Using any part other than this may cause accidents.
- At a frequency of once a month, implement the inspections described in 8.4 "Checking the Pilot Flame Unit (AA-7000F, AA-7000F/AAC)" and 8.5 "Checking for Gas Leaks (AA-7000F, AA-7000F/AAC)".

If you do not, safety may be compromised.

• When using a nitrous oxide-acetylene flame, be sure to use the high-temperature burner head (option).

CAUTION

- If you spill a liquid like water or an organic solvent on the instrument, wipe it off immediately. It could cause equipment failure.
- Be careful not to spill water to office equipment such as the PC as well as the instrument.
 It could cause equipment failure.

Important data could be lost as the result of an accident.

- Be sure to make a backup copy of the contents of the PC's hard disk. Important data could be lost as the result of an unforeseen accident.
- The standard analysis conditions incorporated in the software are not the optimum conditions. Optimize the analysis conditions for each element of each sample to be measured. The analysis conditions may change according to the characteristics of the sample, the temperature, humidity and other environmental conditions, replacement of maintenance parts and consumable parts, and so on.

Precautions on Handling Gas

For details on the type and purity of gas to be used with this product, and the gas supply pressure, see 10.6.3 "Gas Requirements".

WARNING

- **Do NOT use oxygen gas.** This could cause ignition or equipment failure.
- Install the gas cylinders in an airy location outdoors that is not exposed to direct sunlight. Feed the high pressure gas indoors using piping.
- Take care that gas cylinders become no hotter than 40 °C, and do NOT allow any flame within 2 meters of the gas cylinders.
- With regard to the use of flammable gases (e.g. acetylene) and gases that increase the susceptibility of substances to burn (e.g. nitrous oxide), smoking and the use of fire is prohibited within 5 meters of the equipment that is using these gases. Install a fire extinguisher to be prepared in the event of an accident.
- Secure gas cylinders with ropes or chains while they are standing up. Liquefied gas cylinders in particular (acetylene, nitrous oxide, etc.) must never be allowed to fall to a horizontal position.
- Use approved pressure regulators and connectors.
- Be sure to use oil-free pressure regulators. Also use ones that have no oil adhering to the inside of the pipes, etc., where high-pressure gas comes into contact.
- Before mounting a pressure regulator on a gas cylinder, be sure to remove dust and other material adhering to the cylinder's outlet. Dust remaining on the outlet may cause gas leakage.
- If the mounting screw for a cylinder's pressure regulator becomes damaged or thread-stripped, do NOT attempt to mount the pressure regulator by force but replace the cylinder with a new one. Mounting the regulator by force in this condition could cause gas leakage.
- Even if the main valve is stiff and difficult to open, do NOT strike the handle or main valve with a hammer or spanner. This could cause gas leakage from the pressure regulator or cylinder outlet, or failure of the pressure regulator.
- When using high pressure gas, make sure that there is sufficient ventilation. Carry out leak
 inspections on the gas piping and pressure regulators at least once a month.
 For details, see 8.5 "Checking for Gas Leaks (AA-7000F, AA-7000F/AAC)" and 8.6 "Checking for
 Leaks of Pressure Regulators (Optional)".
- Before opening a gas cylinder, check that the stop valve is closed. Turn the secondary pressure adjusting valve fully to the left (counterclockwise), then open the cylinder using the handle for that purpose.
- Close the cylinder's main valve immediately after you have finished using gas.
- Inspect the pressure gauge at least once every three months.

Precautions on handling acetylene gas

Acetylene is a dangerous gas that explodes easily. When handling it, observe the following points in addition to the "Precautions on Handling Gas" stated above.

Characteristics of acetylene gas

Acetylene is a gas characterized by a tendency to explode easily if it is handled incorrectly. It should be handled correctly based on an understanding of its characteristics.

- It is a colorless, odorless gas (there is the smell of the solvent).
- It has a wide range of combustion, and will burn when it is mixed with air in a ratio by volume of 2.5% or greater.
- Even when it is not mixed with air or oxygen, ignition energy can cause explosive decomposition.
- Contact with copper (including alloys containing 62% or more copper), silver (including alloys), gold (including alloys) or mercuric materials (including alloys) may trigger explosive decomposition.
- It is lighter than air and therefore tends to stay at higher levels.

Acetylene gas is fed in to fill a container (cylinder) that incorporates a porous substance impregnated with acetone, and supplied as dissolved acetylene.

WARNING

- When using acetylene gas, be sure to use a pressure regulator for acetylene gas. If you use any other kind of regulator, gas will leak and may ignite.
- Do NOT use pipes made of copper (including alloys containing 62% or more copper), silver (including alloys), gold (including alloys) or mercuric materials (including alloys) to convey dissolved acetylene gas.

This will lead to the formation of metal acetylides, which could undergo explosive decomposition if subjected to shock.

• When opening the main valve on a dissolved acetylene cylinder, make sure to turn the valve at least one turn but no more than one and a half turns from the fully closed state. If it is opened by less than one full turn, it will not be possible to supply sufficient acetylene gas when using a nitrous oxide-acetylene flame with the high-temperature burner head (option), causing flashback and failure of the instrument.

CAUTION

- Use dissolved acetylene cylinders that use acetone as the solvent. Solvents other than acetone can cause the electromagnetic valves used with the unit to fail.
- When opening the main valve on a dissolved acetylene cylinder, turn it one turn or more but one and a half full turn or less from the fully closed state.
 If it is opened more than one and a half turns, the acetone that fills the cylinder will flow out and may influence measurement.
- **Do NOT use a dissolved acetylene cylinder with a primary pressure of less than 0.5 MPa.** The acetone that fills the cylinder will flow out and may influence measurement. When the primary pressure of a gas cylinder has fallen to 0.5 MPa, exchange it for a new one.
- Do NOT use a cylinder with an acetylene pressure regulator at the secondary pressure of greater than 0.127 MPa.

The acetone that fills the cylinder will flow out and may influence measurement.

· Precautions on handling nitrous oxide gas

Nitrous oxide is a dangerous gas with anesthetic characteristics. It is also known as laughing gas. When handling it, observe the following points in addition to the "Precautions on Handling Gas" stated above.

Characteristics of nitrous oxide gas

Nitrous oxide gas should be handled correctly based on an understanding of its characteristics.

- It is a colorless gas but has a characteristic odor.
- It increases the susceptibility of substances to burn.
- It has anesthetic characteristics.
- It is heavier than air and therefore tends to stay on the floor.

WARNING

• Nitrous oxide gas has anesthetic characteristics. Be sure to ventilate the room while you are using the instrument.

CAUTION

• Open the cylinder's main valve fully. If it is not opened sufficiently, the flow rate of the gas may fluctuate during use and this may affect measurement. • Precautions on handling of the air compressor (optional)

WARNING

• Handle the air compressor correctly by referring to the instruction manual that is supplied with it.

Incorrect usage could lead to accidents. It could also lead to equipment failure.

CAUTION

• Provide a device like a drain separator part way along the piping from the air supply source for dehumidification.

If you use air with a lot of moisture in it, the instrument may fail.

- Before using the air compressor, check the operation by referring to its instruction manual. Using it without checking its operation first could cause equipment failure.
- When using an oil supply type compressor, check that the oil level always remains between the red lines of the oil level gauge.
- After use, always open the drain cocks and discharge the water and oil inside the tank and the transformer.
- When supplying air from an existing compressor or compressed air tubing, verify the following:
 - That air is supplied at a pressure of 0.35 to 0.4 MPa
 - That the pressure does not fluctuate
 - · That the air supplied does not contain water, oil, or dust

Measures for Preventing Static Electricity Accidents

WARNING

• Take thorough measures to prevent buildup of static electricity. Static electricity could result in fire or explosion.

CAUTION

- The best way to prevent static electricity accidents is simply to prevent the occurrence and accumulation of electro-static charges.
- It is important to combine multiple preventive measures.
- If large amounts of flammable solvents are collected in a large container, implement preventive measures 1 to 5 below.

Preventive measure 1:

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

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

Preventive measure 2:

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

Preventive measure 3:

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

- Wear anti-static clothing and shoes.
- Ground the human body with anti-static wrist straps. (For safety, the wrist strap should be connected to the ground using an intervening resistor of about 1 M Ω).
- Spread anti-static matting or the like on the floor, to make the floor conductive.
- Persons who have not taken anti-static precautions should touch some grounded metal component before coming near the waste liquid container, in order to ground the body and clothing.

Preventive measure 4:

Use the tubing included in standard accessories for the drain line.

- Ensure that there is no entry of air from the tubing connections.
- Air bubbles in the tubing may cause the electro-static charge to be multiplied by a factor of 20, 30 or more.

Preventive measure 5:

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

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

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

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

Precautions on Handling Chemicals and Samples

WARNING

- When handling chemicals, wear protective goggles and protective gloves. If chemicals get into the eyes, there is a risk of loss of sight. If any chemical does get into the eyes, wash it out immediately and consult a doctor.
- Be sure to obtain the Safety Data Sheet (SDS) of the chemicals to handle those chemicals properly by understanding their characteristics and handling information.
- When handling a sample that is toxic or where there is a risk of biological infection, wear protective gloves.
- Do NOT measure explosive samples.
 Not only may this cause equipment failure and damage, it will also not be possible to assure the safety of the users of the instrument.
- When copper, silver, gold or mercury mixes with acetylene inside the chamber, deposits called
 metal acetylides are formed. After performing flame measurement for samples that contains any
 of these metals as main component or at a high concentration, thoroughly clean the chamber,
 nebulizer, burner head, drain bottle, drain tube, and other liquid-wetted parts. Even if the product
 is cleaned regularly, metal acetylides that cannot be removed may accumulate in long-term use.
 Perform periodic inspections by field engineers to check the necessity of replacing the parts in
 the atomizer, and periodically replace the parts to prevent deterioration of the parts and
 accumulation of metal acetylides.

Metal acetylides can cause explosion due to a decomposition reaction. In addition, deposits may interfere with drain drainage and lead to flashback.

• Perchloric acid reacts with salts intensely. If you fail to clean salt deposits on the chamber or burner head frequently or fail to wash them off with the blank solution completely, flashback may occur.

Measures to prevent explosion of acetylides due to a decomposition reaction

Preventive measure 1

Perform periodic inspections by Shimadzu service personnel to check the necessity of replacing the parts in the atomizer, and periodically replace the parts.

Maintenance parts	Standard Replacement Interval	Examples of replacement intervals
Burner head	3 years ^{*1}	2 years ^{*2}
Burner socket	3 years ^{*1}	2 years ^{*2}
Nebulizer	2 years ^{*1}	1 year ^{*2}
O-ring SET	3 years ^{*1}	1 year ^{*2}
Chamber	6 years ^{*1}	2 years ^{*2}
Mixer	6 years ^{*1}	2 years ^{*2}
Drain tank	3 years ^{*1}	1 year ^{*2}
U-tube	3 years ^{*1}	1 year ^{*2}

Maintenance parts and replacement interval of the atomizer (reference examples)

- *1: The standard replacement interval is the replacement interval under the usage conditions specified by Shimadzu.
- *2: The replacement interval may become shorter depending on the frequency of measurements and liquid properties of the samples.

Preventive measure 2

In addition to regular daily inspections, perform the following cleaning procedures for liquid-wetted parts.

Parts to be cleaned and the method of cleaning

Parts to be cleaned	REFERENCE
Burner head	8.2 "Burner Maintenance (AA-7000F,
Burner socket	AA-7000F/AAC)"
Nebulizer (including the spray unit and disperser)	8.2.2 "Nebulizer Maintenance"
Chamber	8.2.3 "Chamber Maintenance"
Mixer	
Drain tank	
U-tube	

NOTE

Waste liquid generated during cleaning should be treated appropriately in the same manner as waste liquid for atomic absorption spectrophotometry.

Frequency of cleaning (reference example)

(When the silver concentration in the sample is 1 g/100 ml) Clean the applicable parts every 40 sample measurements. Clean them once a week even if the number of samples is less than it.

CAUTION

- Maintenance should be immediately carried out when 30 minutes have elapsed after extinguishing the flame and the combustion chamber cools.
- Change the frequency of cleaning according to the concentration of acetylide-forming elements in the sample.
- If you have any questions in analyzing samples based on copper, silver, gold, or mercury, contact your Shimadzu representative.

NOTE

- Shortening the pre-spray time and integration time is effective in reducing the amount of produced metal acetylides. Optimize the analytical conditions according to the characteristics of samples and environment.
- To prevent drying and overheating of produced metal acetylides, it is recommended to suck distilled water or blank liquid during ignition unless the sample is measured.

Precautions for Instrument Inspection, Maintenance, Adjustment and Care

WARNING

- Unplug the instrument before inspection, maintenance, or parts replacement. Otherwise, electrical shock or short-circuit accidents could occur.
- Never remove the main cover.

This may cause injury or malfunction of the instrument or hazardous radiation exposure. The main cover does not need to be removed for routine maintenance, inspection and adjustment. Have your Shimadzu representative perform any repairs requiring removal of the main cover.

• Do NOT replace fuses.

The fuses in the instrument must be replaced by your Shimadzu representative. Using any other fuse could cause a fire.

• If the power cord plug gets dusty, remove the plug from the power outlet and wipe away the dust with a dry cloth.

If dust is allowed to accumulate, fire could result.

- Check that there is no cracking of the rubber hose for gas supply or deterioration due to adhesion of chemicals, every time you use the instrument. If there is any cracking or deterioration, replace the hose with a new one. Note that cracking will cause gas leakage or fire.
- When replacing a part, use the part described in the instruction manual. If you use any other part, that part may be damaged, meaning that it cannot be used normally.
- Do NOT attempt to perform any measurement other than those described in the instruction manual.

CAUTION

- Do NOT leave this product wet with water, or wipe it over with alcohol or thinner based solvents. This will cause rusting or discoloration.
- Deal with waste liquids appropriately in accordance with the regulations and directions of the relevant authorities.

Danger of Repairs, Disassembly and Modification

CAUTION

• Do NOT modify or disassemble the product without permission. This can result in accidents due to electric shock or short circuits. It can also result in injuries and

equipment failure.

Precautions on Use

In order to use the atomic absorption spectrophotometer safely, observe the following warnings and precautions.

If you fail to observe them, the safety of the instrument may be compromised.

WARNING

• At ignition, do NOT put your face or hand inside the burner module (burner compartment). Do NOT look in from above the burner compartment or hold your hand over it.

The nitrous oxide-acetylene flame reaches a height of 40 cm above the top face of the instrument. To prevent accidents that might result from carelessness, always fit the chimney and close the flame shield (chimney door) before ignition.

Check that all parts are correctly fitted before ignition. If any part is out of place there will be a risk of flashback. See 1.4.3 "Burner".

- Check that there is water in the drain tank before ignition.
- Do NOT remove the nebulizer, drain tube or burner head during combustion.
- Do NOT touch the chimney or burner head during ignition. You could be burned.
- Do NOT touch the chimney or burner head for 30 minutes after the flame has been extinguished. You could be burned.
- Do NOT touch the deuterium lamp while it is hot. You could be burned.
- **Do NOT touch the tip of the cleaning wire for the nebulizer capillary tube.** The wire may stick into your finger.

CAUTION

- Do NOT use the flame for any purpose other than analysis.
- During combustion, do NOT hold anything over the flame.
- When using the atomic absorption spectrophotometer in the flame method, do NOT remove the chimney.

If you remove it, the heat of the flame will transfer inside the instrument and the reliability of the measurement may be lost.

- Only open the chimney door when necessary, and do NOT leave it open during combustion. If the door is left open, the nitrous oxide flame will shift backward and come into contact with the chimney, and this will greatly speed up the rate of deterioration of the chimney.
- Check the gas flow rate setting before ignition.
- Do NOT use the standard burner head (10 cm slot burner) with the key for the high temperature burner head (option) left in the key slot of the burner selection keyswitch (BURNER SELECT).
- Do NOT allow foreign material to get into the key slot of the burner selection keyswitch (BURNER SELECT).
- Do NOT allow foreign material to get into the lamp socket.
- Do NOT insert your fingers or hand into the holes of the lamp turret, igniter, flame monitor, etc.

Emergency Action

In an Emergency

Take the following actions in case of emergency or when an abnormality occurs in the atomic absorption spectrophotometer.

Inspect the equipment before using it again and contact your Shimadzu representative if necessary.

- Emergency Action
 - Turn OFF the power switch of the atomic absorption spectrophotometer. Even if measurement is in progress, the burner flame can be safely extinguished by turning the power switch OFF.
 - 2. Turn OFF all the power switches of accessories.
 - 3. Close the main valves of the gas supply pipings for acetylene, air and nitrous oxide.
 - 4. Close the main valve of the cooling water and argon supply piping.
 - 5. Shut OFF the power supply.
 - Turn OFF the switchboard when the power cable is fixed to the switchboard with screws.
 - Disconnect the power cable when the power cable is connected with the plug.

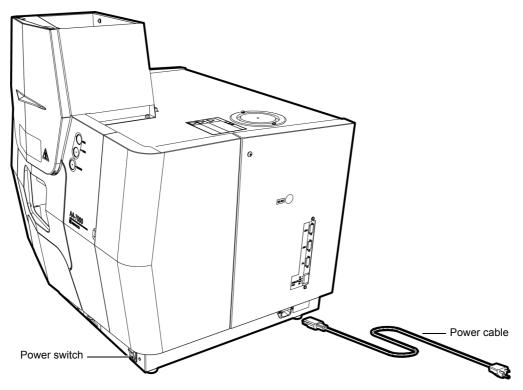


Fig. 1 Power Switch and Power Cable of Atomic Absorption Spectrophotometer

Measures in the Event of a Power Outage

When a power outage has occurred, take the following measures.

- 1. Stop the supply of acetylene gas promptly.
- 2. Turn OFF the power switches at the AA main unit and all options.
- 3. Open the windows and doors of the room in which the instrument is installed for ventilation.
- 4. Check that there is nothing that could ignite acetylene gas in the room.
- 5. After restoring the power supplies, check "Installation Precautions" and "Precautions on Work and Operation" and then start the instrument as normal.

If the instrument does not start up, request your Shimadzu representative to inspect it.

Measures in the Event of a Lightning Strike

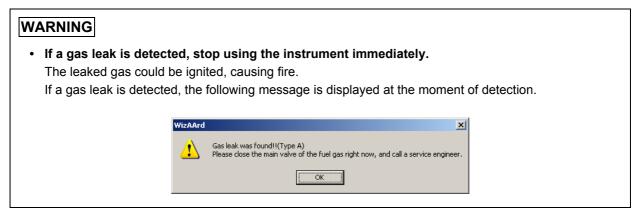
If a large fluctuation in the power supply voltage is experienced momentarily as the result, for example, of the electricity transmission system being struck by lightning, some of the functions are shut off in order to protect the instrument. As a result, the communication between AA main unit and PC is stopped. In this case, take the following measures.

- 1. Stop the supply of acetylene gas promptly.
- 2. Turn OFF the power switches at the AA main unit and all options.
- 3. Open the windows and doors of the room in which the instrument is installed for ventilation.
- 4. Check that there is nothing that could ignite acetylene gas in the room.
- 5. After restoring the power supplies, check "Installation Precautions" and "Precautions on Work and Operation" and then start the instrument as normal.

If the instrument does not start up, request your Shimadzu representative to inspect it.

Measures When Gas Leakage Is Detected

Execute [Start Leak Check] on the [Initialize] dialog box or select [Instrument] - [Gas Leak Check] from the menu, and a gas leak inspection lasting approximately 8 minutes will be performed automatically.



If a gas leak is detected, stop using the instrument immediately and take the following measures.

- 1. Stop the supply of acetylene gas promptly.
- 2. Turn OFF the power switches at the AA main unit and all options.
- 3. Open the windows and doors of the room in which the instrument is installed for ventilation.
- 4. Check that there is nothing that could ignite acetylene gas in the room.
- 5. Request your Shimadzu representative to inspect the instrument.

If no gas leak is detected as the result of the gas leakage inspection, the following message is displayed after the elapse of approximately 8 minutes, and from this point on it will be possible to ignite the flame.

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1	No gas leak is detected. Before you ignite, set the BURNER SELECT switch to Air-C2H2 and pushing the PURGE button, adjust Air flow rate between 13.5 and 17.5 L/min by the flowmeter knob. (In case optional flowmeter is attached.) After ignition, adjust gas supply pressure to maintain Fuel to 0.09MPa and Support to 0.35MPa during combustion.

NOTE

In the following cases the result of the gas leakage inspection is [NG] and it will not be possible to ignite the flame.

- · Gas was not being supplied at the start of the inspection.
- The inspection was stopped part way through.

Measures When Flashback Occurs

The AA-7000 series instruments are equipped with a safety device to prevent flashback, but flashback may still occur when using the flame method. If flashback occurs, stop using the instrument immediately and implement the measures in the event of an emergency.

WARNING

• When flashback occurs, the burner head can be thrown 10 cm upward.

NOTE

If the instrument detects flashback, ignition will become impossible.

Before using the instrument again, request your Shimadzu representative to inspect it.

Once the representative has inspected the instrument and confirmed that there is no problem with it, he will restore it to the condition in which ignition is possible.

Warning Labels on the Instrument

Types of Label	Corresponding Model	Details of the Warning	Position Where Affixed
Warning label Caution: HOT SURFACE	AA-7000F AA-7000F/AAC AA-7000G	Before replacing the deuterium lamp, wait at least 30 minutes after turning OFF the lamp.	(Part No.: 206-77429)
Warning label Caution: HOT SURFACE	AA-7000F AA-7000F/AAC	 During flame combustion, do NOT look into from above the chimney. Before starting maintenance of the burner compartment, wait at least 30 minutes after turning OFF the flame. Before starting ignition, be sure to set the chimney and front-panel. 	(Part No.: 206-77429)
Warning label Caution: HAZARDOUS VOLTAGE	AA-7000F AA-7000F/AAC AA-7000G	 Do NOT touch the lamp socket electrode while power is being supplied. To prevent electrical shocks, attach the accessory cap to the empty power socket. 	(Part No.: 206-77428)

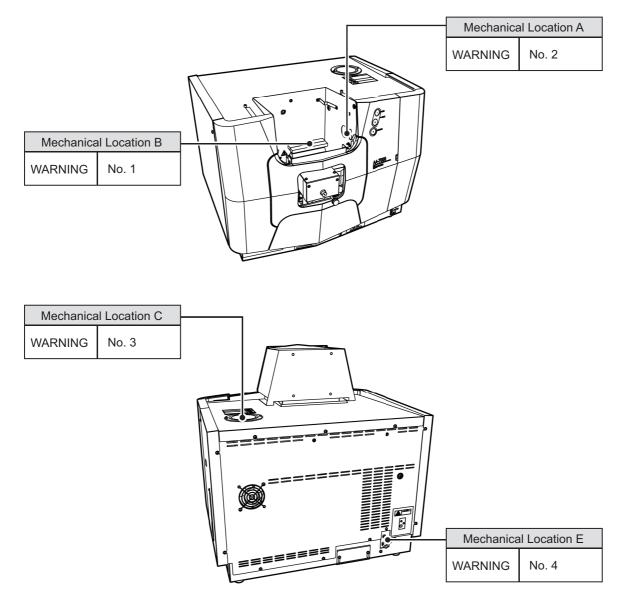
Types of Label	Corresponding Model	Details of the Warning	Position Where Affixed
Warning label Caution: HAZARDOUS VOLTAGE	AA-7000F AA-7000F/AAC	Before starting ignition, be sure to close the door of the chimney.	(Part No.: 206-77429)
Warning label Caution: FLAMMABLE GAS	AA-7000F AA-7000F/AAC	 During flame combustion, do NOT remove any parts of the burner compartment. After using a gas, be sure to close the main valve of the gas cylinder. 	(Part No.: 206-77429)
Warning label Caution: EXPLOSION POTENTIAL	AA-7000F AA-7000F/AAC	Do NOT use oxygen gas mixture.	(Part No.: 206-77429)

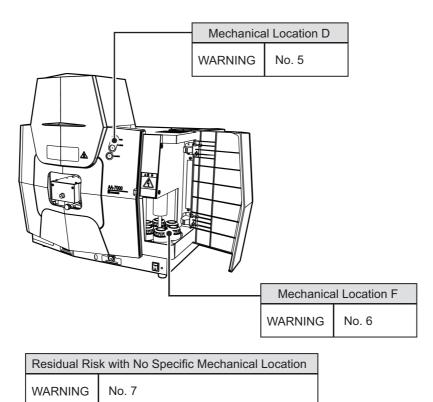
Residual Risk Information

A residual risk indicates a risk that could not be reduced or eliminated despite attempts to reduce it in appropriate manners in the process of design and manufacture. Check the risk locations in "Residual Risk Map", and take the relevant protective measures described in "List of Residual Risks".

Residual Risk Map

The "Mechanical Location" and "No." indicated below are in accordance with those in "List of Residual Risks". For details, see "List of Residual Risks".





List of Residual Risks

The "No." and "Mechanical Location" indicated below are in accordance with those in "Residual Risk Map". Be sure to check the actual "Mechanical Location" referring to "Residual Risk Map".

Furthermore, read through and understand the content in "Reference" to take appropriate protective measures.

Preparation

No.	Mechanical Location	Description	Protective Measure taken by machine user	-	-	
		WARNING Burns from heat of the flame Burns from heat of	Reference	"Precautions on Use"		
1	В		on the burner compartment,	Operation Category	Pre-operation check	
			wait at least 30 minutes after extinguishing the flame.	Required Qualification/ Education	Qualified person received training to use the instrument	
				Reference	8.4	
2	А	Electric shock from touching the	Do NOT touch the electrode while power is being	Operation Category	Pre-operation check	
		igniter discharger	supplied.	Required Qualification/ Education	Qualified person received training to use the instrument	
	Е			Reference	10.6.3	
4		Explosion or spreading of fire	e Do NOT use oxygen gas Car mixture. Re Quali	Operation Category	Installation	
		due to abnormal combustion of the flame		Required Qualification/ Education	Qualified person received training to use the instrument	
				Reference	4.5	
5	D	Explosion or spreading of fire	le Flame" before starting	Operation Category	Pre-operation check	
		due to flammable gas catching fire		Required Qualification/ Education	Qualified person received training to use the instrument	
			Do NOT touch the lamp	Reference	2.3	
6	F	Electric shock from touching the	socket electrode while power is being supplied.	Operation Category	Pre-operation check	
0		lamp	electrode attach the	To prevent electric shock, attach the accessory cap to the empty power socket.	Required Qualification/ Education	Qualified person received training to use the instrument

Opereation

No.	Mechanical Location	Description	Protective Measure taken by machine user	-	-
		WARNING Burns from heat of	During flame combustion, do NOT look into the burner	Reference Operation	"Precautions on Use" Running with the
		the flame	compartment from above the chimney.	Category	flame lit
1 B on the burner co wait at least 30 r extinguishing the Before starting is	Before starting maintenance on the burner compartment, wait at least 30 minutes after extinguishing the flame. Before starting ignition, attach the chimney and front panel.	Required Qualification/ Education	Qualified person received training to use the instrument		
				Reference	8.4
2	А	Electric shock from touching the	Before starting ignition, closeCatethe door of the chimney.RequQualifi	Operation Category	Igniting flame
		igniter discharger		Required Qualification/ Education	Qualified person received training to use the instrument
	D	WARNING Explosion or spreading of fire	Be sure to read 4.5 "Igniting and Extinguishing the Flame" before starting ignition.	Reference	4.5
5				Operation Category	Igniting flame
		due to flammable gas catching fire	During flame combustion, do NOT remove any parts of the burner compartment.	Required Qualification/ Education	Qualified person received training to use the instrument
			Do NOT touch the lamp	Reference	2.3
6	F	Electric shock from touching the	g the is being supplied.	Operation Category	Attaching a hollow cathode lamp
	electrode attach the accessory	attach the accessory cap to the empty power socket.	Required Qualification/ Education	Qualified person received training to use the instrument	
		Not specified Not	Reference	"Precautions on Handling Gas"	
7			close the main valve of the	Operation Category	Operation at end of work
				Required Qualification/ Education	Qualified person received training to use the instrument

Maintenance

No.	Mechanical Location	Description	Protective Measure taken by machine user	-	-
				Reference	"Precautions on Use"
1	В	Burns from heat of the flame	Before starting maintenance on the burner compartment, wait at least 30 minutes after extinguishing the flame.	Operation Category	Maintenance of the peripheral parts of the burner
				Required Qualification/ Education	Qualified person received training to use the instrument
				Reference	8.4
2	A	Electric shock from touching the igniter discharger	while power is being supplied. Required Qualification Education	•	Maintenance of the peripheral parts of the burner
				Required Qualification/ Education	Qualified person received training to use the instrument
			Do NOT touch the lamp	Reference	8.8
3	С	Burns from heat of the lamp	while power is being supplied. Turn off the power and change the lamp after	Operation Category	Changing D2 lamps
				Required Qualification/ Education	Qualified person received training to use the instrument
			Do NOT touch the lamp	Reference	2.3
6	F	Electric shock from touching the	socket electrode while power is being supplied.	Operation Category	Attaching a hollow cathode lamp
		lamp socket electrode	To prevent electric shock, attach the accessory cap to the empty power socket.	Required Qualification/ Education	Qualified person received training to use the instrument
	Not specified	WARNING Explosion or	After using gas, be sure to close the main valve of the	Reference	"Precautions on Handling Gas"
7		Not specified		Operation Category	Operation at end of work
		flammable gas	gas cylinder.	Required Qualification/ Education	Qualified person received training to use the instrument

Warranty and After-Sales Service

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.
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 - 1) Improper product handling
 - 2) Repairs or modifications performed by parties other than Shimadzu or Shimadzu designated companies
 - 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 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

If any problem occurs with this instrument, inspect it and take appropriate corrective action as described in this manual. If the problem persists, or symptoms not covered in this manual occur, contact your Shimadzu representative.

Replacement Parts Availability

Replacement parts for this instrument will be available for a period of seven (7) years after the discontinuation of the product. 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.

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- d. The invalidity or unenforceability of any provision of this Agreement shall not affect the validity or enforceability of any other provision.

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 the troubleshooting section of this manual.

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.

Maintenance, Inspections, and Adjustment

In order to maintain the instrument's performance and obtain accurate measurement data, daily inspection and periodic inspection/calibration are necessary.

- For daily maintenance, inspection, and replacement parts, see Chapter 8 "Maintenance" of this manual.
- Periodic inspection/calibration should be requested to your Shimadzu representative.
- Replacement cycles described for periodic replacement parts are rough estimate.

Replacement may be required earlier than the described replacement cycles depending on usage environment and frequency.

Handling Waste Liquid

WARNING

- Do NOT use a container made of glass as the waste liquid tank. In the event of a flashback there is a risk that glass fragments will be scattered.
- Select a waste liquid container made of material that is resistant to the chemicals used. If you use a container with no chemical resistance, waste liquid will leak and may cause burns or fire.

Waste liquid discharged during measurement or pretreatment should be handled in different ways in accordance with the solute and solvent contained in that waste liquid. The way of handling waste liquid is different depending upon the country or region.

Be sure to dispose of waste liquid according to the location of use.

When handling waste liquids, wear protective gloves and safety goggles.

Disposal Precautions

Disposal of the instrument

When scrapping the instrument, contact your Shimadzu representative.

If you dispose of it yourself, do so in accordance with the processing standards determined by law, separately from general industrial waste and household garbage.

Disposal of lamps

The raw materials used in hollow cathode lamps are metal (the lamp element), quartz glass and plastic. Some lamps also contain harmful metals (mercury, arsenic, berylium, selenium, etc.) or metals that ignite on contact with water (calcium, lithium, sodium). Carefully read the notes that accompany the hollow cathode lamp, and, if any of these metals is contained, ask an industrial waste disposer officially licensed for hazardous waste disposal.

When disposing of D_2 lamps, treat them as industrial waste. The raw materials used in D_2 (deuterium) lamps are metal (tungsten), quartz glass, ceramics and plastics.

Electromagnetic Compatibility

WARNING

• Graphite furnace atomizer GFA-7000A is a class A product, designed not for use in residential environment.

Graphite furnace atomizer GFA-7000A, an optional accessory for AA-7000 series, is a class A for electromagnetic interference (emission).

NOTE

When an electromagnetic disturbance occurs to the instruments being used close to this product, take the following measures:

- Take an appropriate distance among the instruments and this product in order to eliminate the disturbance.
- Supply power from a different power source.

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Chapter 1 Overview

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1.1

Overview of AA-7000 Series

The Atomic Absorption Spectrophotometer AA-7000 series incorporates two background correction functions, the D_2 method (Deuterium Lamp method) and the SR method (High-Speed Self-Reversal method), enabling selection of the appropriate method for the measurement sample.

- The AA-7000 series instruments support the following three measurement modes.
 Flame Continuous Method
 Flame Micro Sampling Method
 Furnace Method (Electrothermal Method)
- Instrument configurations

AA-7000F: Flame unit AA-7000F/AAC: Flame unit (can be extended for furnace analysis) AA-7000G: Furnace unit

The AA-7000F/AAC (and AA-7000F equipped with the optional AAC-7000 Auto Atomizer Charger) allows you to change the measurement mode while automatically setting the atomizer appropriate for the measurement mode on the optical axis. The instrument also enables quick and easy switching back and forth between flame measurement and furnace measurement. In addition, wide ranging measurement operations are available, from manual operation to automatic continuous measurement of multi-elements with the use of an autosampler. This enables selection of the appropriate combination to match the number of elements and samples to be analyzed as well as the skill of the operator.

The PC software controlling the AA-7000 series operates on Windows 10 Pro / Windows 7 Professional / Windows Vista Business / Windows XP Professional, and using the Wizard for parameter setting enables an operator, even a beginner with the atomic absorption spectrophotometer, to make measurement conditions easily. In addition, a hardware validation function is available as standard to allow you to check the performance of the AA-7000 series. This function is applicable to system suitability management for IQ/OQ or the like.

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

1.2.1 AA-7000F and AA-7000F/AAC Parts Information

No.		Parts	Q'ty	Part No.
1	Atom	ic Absorption Spectrophotometer Main Unit	1	
2	Acce	ssories	1	206-77534-xx
	-1	Cord Set	1	S071-60845-01
	-2	Hose ASSY (for Air)	1	S206-50389-41
	-3	Hose ASSY (for C ₂ H ₂)	1	S206-50389-42
	-4	Hose Clamp (16 mm)	2	S037-61019
	-5	Cleaning Wire	1	S201-79229-01
	-6	Sampling Tube (PTFE)	2	S204-05899-01
	-7	Sampling Tube (for Organic Solvent Sample)	1	S206-50772-91
	-8	Polyethylene Capillary, No.3	1 (0.3 m)	S200-31328-01
	-9	Polyethylene Tube, 8×1 (for Drain Tube)	1 (2.4 m)	S016-43201-02
	-10	Card (Packet of 10)	1	S206-52046-91
	-11	Drain ASSY	1	S206-77413-41
	-12	Grease, in Cup	1	S206-50442-91
	-13	Chimney	1	S206-77243-92
3	Softv	vare Package, WizAArd	1	S206-77525-93
	-1	CD, WizAArd	1	S206-77526-91
	-2	Cable, RS-232C 9P	1	S206-50325-91
	-3	Installation Procedure, WizAArd	1	S206-97230
	-4	Software Licence Agreement	1	223-00132
	-5	WizAArd Certificate of Compliance for ISO-9001 QA System	1	206-97259-xx
4	Instru	uction Manual	1	S206-97176
5	Inspe	ection Test Report	1	206-77551-02
6	State	ement of conformity (ISO-9001, English)	1	206-84934-66 (AA-7000F)
				206-84934-68 (AA-7000F/AAC)
7	Safe	ty Check-out Sheet (Safety Precautions)	1	206-97226
8	Warr	ing Label	1	037-72405-25
9	Seria	I Number Label	3	206-57770

1.2.2 AA-7000G Parts Information

No.		Parts	Q'ty	Part No.
1	Atom	ic Absorption Spectrophotometer Main Unit	1	
2	Acce	ssories	1	206-77535-xx
	-1	Cord Set	1	S071-60845-01
	-2	Furnace mounting board	1	S206-77704
3	Softw	vare Package, WizAArd	1	S206-77525-93
	-1	CD, WizAArd	1	S206-77526-91
	-2	Cable, RS-232C 9P	1	S206-50325-91
	-3	Installation Procedure, WizAArd	1	S206-97230
	-4	Software Licence Agreement	1	223-00132
	-5	WizAArd Certificate of Compliance for ISO-9001 QA System	1	206-97259-xx
4	Card	(Packet of 10)	1	S206-52046-91
5	Instru	uction Manual	1	S206-97176
6	Inspection Test Report 1 206-77551-02			
7	Certif	206-84934-67		
8	Safet	y Check-out Sheet (Safety Precautions)	1	206-97226
9	Seria	l Number Label	3	206-57770

NOTE

The parts with a part number starting with "S" are sold as maintenance parts or consumable parts. Those with a part number not starting with "S" are not sold individually.

1.3.1 Installation of AA-7000 Series

The instrument will be installed and adjusted by your Shimadzu representative. Attend and check these operations.

In order to use this product safely, contact your Shimadzu representative for details on reinstallation after moving the instrument.

1.3.2 Installation of PC Software

For installation, refer to the WizAArd Installation Manual (206-97230).

Name and Function of Each Part

1.4.1 Operation Switches/Connectors

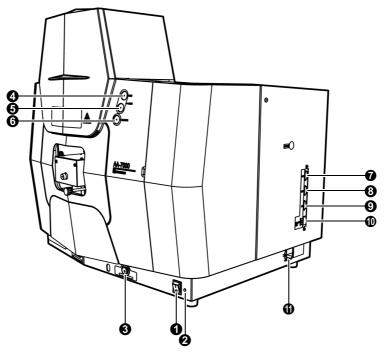


Fig. 1.1 AA-7000 Series Main Unit

No.	Name	Function
0	POWER switch	Turns ON the instrument. Press the " " side of the switch to turn ON; press the "()" side to turn OFF.
0	POWER indicator (Green light)	Lights when the instrument is tuned ON.
6	BURNER SELECT switch	This key switch is for preventing mistaken use of burner heads. When the high temperature burner head (optional) is to be used with the nitrous oxide-acetylene flame, a key attached to the burner head is used to change to the $N_2O-C_2H_2$ position. If not in this position, the nitrous oxide flame cannot be ignited.
4	PURGE button	When this button is pressed independently, the solenoid valve is opened to send support gas (air or N_2O). When this button and the IGNITE button are pressed simultaneously, flame is ignited.
6	IGNITE button	When this button is pressed independently, nothing happens. When this button and the PURGE button are pressed simultaneously, gas is fed to the burner and then flame is ignited with the pilot flame.
6	EXTINGUISH button	Pressing this button extinguish the flame.
Ø	GFA connector	The cable to GFA is connected.
8	ASC connector	The cable to ASC is connected.
9	PC connector	The cable to PC is connected.
0	ANALOG OUT	This is the analog voltage output terminal. Connect a pen recorder or other device here.
0	AC power supply (~) inlet	Connect the cable set to supply AC power here.

1.4.2 Burner Compartment

The AA-7000F and AA-7000G have adjusting knobs located on the front of the burner compartment for forward/ backward and up/down positioning of the atomizer (burner or graphite furnace). Use these knobs to move the atomizer.

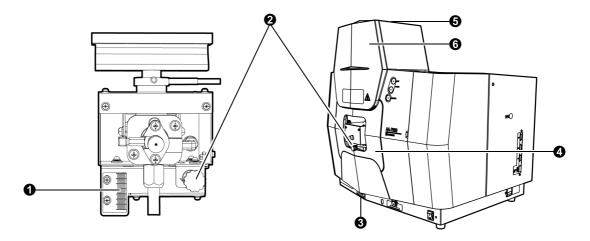


Fig. 1.2 Burner Compartment (AA-7000F and AA-7000G)

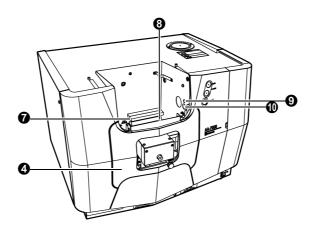


Fig. 1.3 AA-7000F/AAC Burner Compartment

No.	Name	Function
0	Atomizer position indicator scale	The scale indicates the height of the atomizer.
0	forward/backward adjusting knob	When the knob is turned clockwise, the atomizer moves forward. When the knob is turned counterclockwise, the atomizer moves backward. (1 mm per rotation)
8	upward/downward adjusting knob	When the knob is turned clockwise, the atomizer moves downward. When the knob is turned counterclockwise, the atomizer moves upward. (2 mm per rotation)
4	Front panel	The front panel serves to stabilize the combustion of the flame. Remove it when carrying out inspection and maintenance. For details, see 2.4 "Removing and Mounting the Front Panel".

No.	Name	Function
0	Chimney *	 Functions to stabilize the combustion of the flame. Remove the chimney when performing furnace analysis or carrying out inspection and maintenance. For details, see 2.5 "Removing and Mounting the Chimney (AA-7000F, AA-7000F/AAC)".
6	Flame shield (chimney door) [*]	The whole of the front of the chimney ③ slides upward. Open this door when maintenance of the atomizer is necessary.
0	Burner head *	The flame available for the burner head provided as standard is air- acetylene flame. Be sure to use the high temperature burner head (optional) when using a nitrous oxide-acetylene flame.
6	Burner head angle adjustment lever *	This allows the angle of burner slot to be adjusted relative to the light path. When measuring high-concentration samples, measurements of better linearity between concentration and absorbance can be obtained by angling the slot to decrease sensitivity.
0	Flame monitor *	An optical sensor is built in the instrument for monitoring the intensity of the light emitted by the flame. If the flame goes out and the light emission is lost, the solenoid valve in the gas control unit is closed by a signal from the sensor and the flow of raw gas is stopped. In addition, when switching between an air-acetylene flame and nitrous oxide- acetylene flame, an increase in the acetylene flow rate will be detected by an increase in the light emission intensity, and the air and nitrous oxide will be switched.
0	Pilot flame [*]	When the IGNITE and PURGE buttons located on the front of the AA-7000 series are pressed simultaneously, the pilot flame is ignited.

* Not featured on the AA-7000G.

NOTE

If using the AA-7000F/AAC or AA-7000F equipped with the AAC-7000 (optional), the atomizer (burner or graphite furnace) is driven by PC operation. Therefore, there are no positioning knobs for driving the burner module.

1.4.3 Burner

WARNING

In the event of a flashback, the mixer plays the role of absorbing the shock. Before igniting the flame, check that the mixer is correctly installed inside the chamber.

If it is damaged or deformed, it must be replaced.

If there is a flashback, it could cause a gas leak that will damage the chamber.

The AA-7000F and AA-7000F/AAC have a burner head with a 10 cm length burner slot (standard) mounted in the premixing atomizer chamber. This burner head can be used with air-acetylene flames. Please specially install a 5 cm high-temperature head (optional) when using a nitrous oxide-acetylene flame.

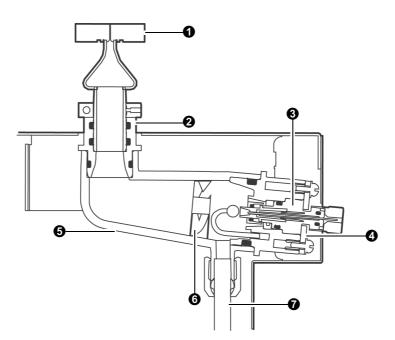


Fig. 1.4 Burner Module

No.	Name	Function
0	Burner head	The flame available for the burner head provided as standard is the air- acetylene flame. Be sure to use the high temperature burner head (optional) when using a nitrous oxide-acetylene flame.
0	Burner socket	Insert the burner head into this port. The burner head is fixed with the internal O-rings.
€	Nebulizer	This uses air negative pressure to suction the sample solution and changes it to a mist of fine particles.
4	Nebulizer retaining plate and fixing screw	Fixes the nebulizer to prevent from coming off.
0	Chamber	The particles of sprayed sample solution are mixed with the combustion gas in this chamber.
6	Mixer	Mixing of the particles and combustion gas is promoted. If a flashback should occur, the mixer also serves to absorb the shock.
0	U-tube (Drain discharge port)	Large particles are discharged from this port.

NOTE

When the GFA-7000A (optional) is installed on either the AA-7000F/AAC or AA-7000F equipped with the AAC-7000 (optional), adjustment of the burner head angle is no longer possible.

1.4.4 Hollow Cathode Lamp Turret

WARNING

- Do NOT touch the terminals of the lamp sockets while the power is on. Be sure to turn the lamp OFF before removal or fitting. If it is not OFF there will be a risk of electric shock.
- To prevent electric shock, fit the cap provided to empty sockets.

Six hollow cathode lamps (optional) can be installed in the Lamp Turret, and two of them can be on at one time. For routine analysis in which the analysis elements are known, it is convenient if all the lamps to be used in measurement (up to six) are installed in the turret and the lamp current values, lamp numbers (socket numbers) along with the other measurement parameters are stored beforehand in the instrument memory. This allows the required lamp to be automatically set in the optical path by just loading the stored parameters.

CAUTION

• To install the lamp, be sure to insert it until it reaches the bottom of the socket, and then secure it with the lamp securing ring.

If the lamp is not fully inserted, it is not properly positioned and this can cause a failure in lighting the lamp.

NOTE

Do NOT touch the window section of the lamp with bare hands.

Soiling such as finger grease will adhere to the window and diminish the strength of the lamp, affecting performance.

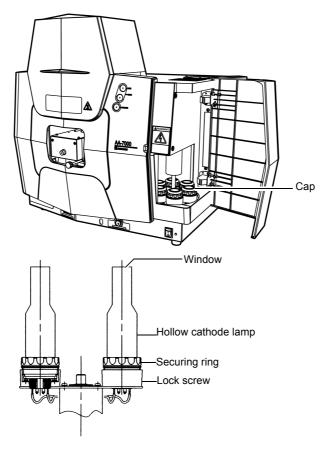


Fig. 1.5 Hollow Cathode Lamp Turret

1.4.5 Deuterium Lamp

The deuterium lamp is of the hot cathode type and is used at the wavelength range of 185 nm - 430 nm in background correction and measurement. The lamp is socketed in the instrument to allow easy replacement. If the lamp is replaced, adjustment may be required. For the replacement, see the section 8.8 "Replacing the Deuterium Lamp" on the Chapter 8 "Maintenance".

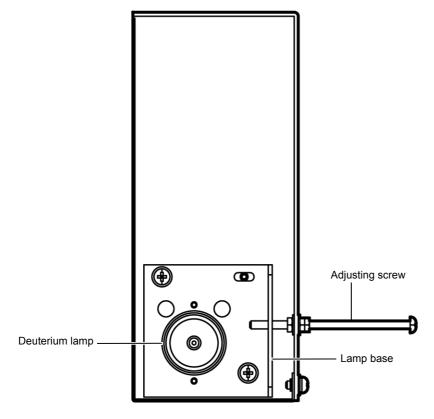


Fig. 1.6 Deuterium Lamp

1.4.6 Support Gas Flow Meter (When Option Equipped)

When the flow meter kit (optional) is attached to the AA-7000F or AA-7000F/AAC, it is possible to regulate the flow rate of support gas.

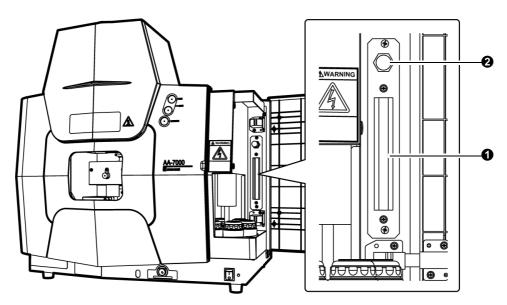


Fig. 1.7 Support Gas Flow Rate

No.	Name	Function
0	Support gas flow meter	This is a float type flow meter, and it displays the flow rate of the support gas (air or nitrous oxide) converted to atmospheric pressure. The gas flow rate is read at the center of the float. Use the scale on the left for air (indication: Air), and use the scale on the right for nitrous oxide (indication: N_2O).
0	Support gas flow rate adjusting knob	Turning this knob to the left increases the flow rate and turning it to the right decreases the flow rate. Normally, the setting for air is 15 L/min and the setting for nitrous oxide is 11 L/min.

NOTE

- The flow rate setting ranges for support gas are as follows.
 - Air: 13.5 to 17.5 L/min

Nitrous oxide: 10.0 to 12.5 L/min

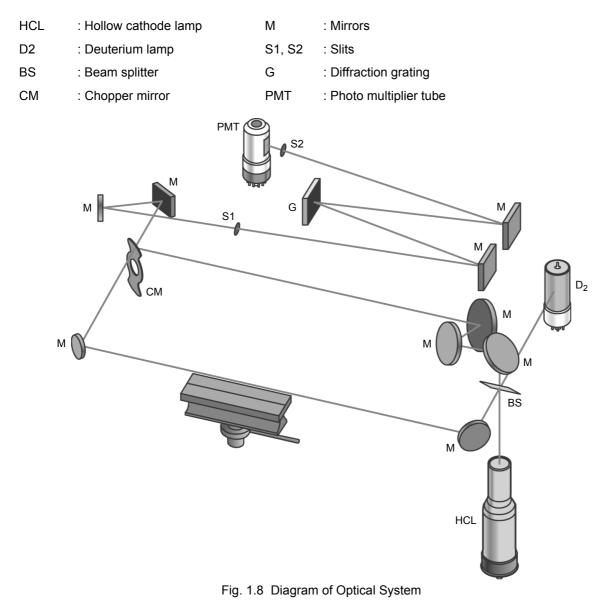
• AA-7000G cannot be equipped with the flow meter kit.

1.4.7 Optical System

The light emitted from the hollow cathode lamp and the deuterium lamp is divided into a sample beam and a reference beam by a beam splitter respectively. The light from the hollow cathode lamp and the deuterium lamp, which has been combined as the sample beam, is absorbed by atoms or backgrounds of coexistent substances while passing through the atomization section, and then enters the detector through the monochrometer. The reference beam passes through the space where the light is not absorbed by a sample and then enters the detector through the monochrometer.

The sample beam and reference are alternately detected as either of them is selected with the chopper mirror just before entering the monochrometer. The difference between the alternately received signals is obtained to allow reducing the baseline drift. Since the chopper mirror is used, no quantity of light is lost from the sample beam and reference beam.

The monochrometer is a high resolution Czerny-Turner type. The wavelength is selected by rotating a diffraction grating to separate the absorption spectrum of the element being analyzed from the other spectra. The direct drive mechanism used for wavelength selection is automated with the use of a motor. All of the optical elements are shielded from the outside air with the quartz window plates and are protected from dust and corrosive gases.



1.4.8 Photometric System

Fig. 1.9 shows the photometric system.

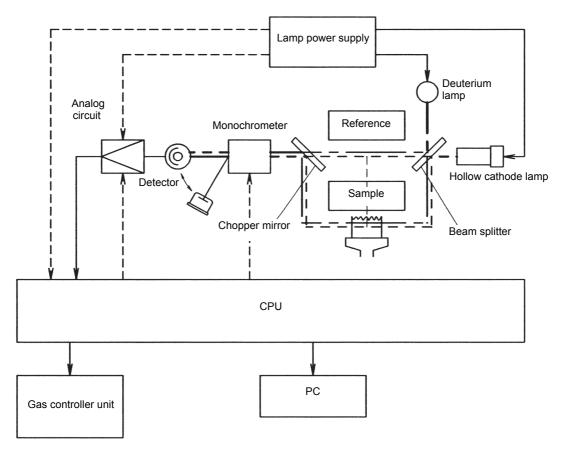


Fig. 1.9 Photometric System

The following four measurement modes can be selected in the AA-7000 series.

EMISSION mode	Used for flame emission analysis.
NON-BGC mode	Used for atomic absorption analysis that doesn't require background correction.
BGC-D2 mode	only available in a wavelength range between 185 and 430 nm.
BGC-SR mode	Available at any wavelength. Effective for compensating spectral interference resulting from nearby lines.

(1) EMISSION mode:

The spectrum produced by atomization of the analysis element in the flame is selected by the monochrometer, and its intensity is measured via photometric circuitry.

(2) NON-BGC mode:

The deuterium lamp is extinguished, and only the light from the hollow cathode lamp is passed through the atomizer. Then only the spectral line (analysis line) to be used for analysis is selected by the monochrometer. By measuring the light absorption at this time, it is possible to determine the concentrations of the elements being analyzed. The detector outputs a signal, which is the sum of a pulsed signal proportional to the analysis line intensity, and a direct current signal from the light emitted from the atomizer (flame or graphite tube).

(3) BGC-D2 mode:

Both light from the pulse-lighted hollow cathode lamp and that from the deuterium lamp that has been pulse-lighted at a different phase pass through the atomizer. The former light is subjected to both absorption by atoms of the element to be analyzed and background absorption attributable to coexisting substances, while the latter light is only subjected to the background absorption. The measurement of their difference in absorption allows correction for the background concomitant substances and an accurate measurement of the absorption by the element being analyzed. Characteristically, this mode offers higher photometric sensitivity than the BGC-SR mode.

(4) BGC-SR mode:

Light from a hollow cathode lamp generated by alternating low current and high current pulses is passed through the atomizer. The low-current-generated light is absorbed both by the element being analyzed and the concomitant substances as background absorption, while the high-current-generated light is absorbed only as background. The difference between them is calculated in order to correct for the background absorbance due to the concomitant substances and accurately determine the absorption by the analysis element. This mode is characterized by the elimination of the effects of nearby lines.

The hollow cathode lamp compatible with the SR method (self-reversal method) must be used.

Detector sensitivity adjustment

When the measurement mode is selected in the AA-7000 series, the detector sensitivity is automatically adjusted to the optimum condition. In the EMISSION mode, the emission signal is set to a fixed value; in the NON-BGC mode, the selected hollow cathode lamp signal is set; and in the BGC-D2 mode, the deuterium lamp signal is set, while in the BGC-SR mode, the high current hollow cathode lamp signal is set so that optimum detector sensitivity is obtained for each mode.

1.4.9 Analog Output

The AA-7000 series is equipped with analog output terminals as standard. You can use these analog output terminals to output signals to a pen recorder and other devices.

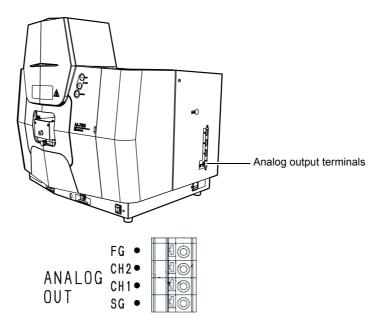


Fig. 1.10 Analog Output Terminals

Item	Specification
Number of channels	2 channels
Signals	 CH1: Atomic absorption signal output (NON-BGC, BGC-D2, BGC-SR) or energy signal output (EMISSION) CH2: Background signal output (BGC-D2, BGC-SR) * In the NON-BGC or EMISSION mode there is no output at this channel. FG: Ground (common) SG: Not used
Output range	5.0, 2.5, 1.25, 0.625 Abs./1 V (4-step switching for each) In the EMISSION mode, fixed at 1 V F.S
Full scale accuracy	±1% for each channel
Impedance	Less than 1 k Ω on each channel

For details on the output cables that are suited to these terminals (optional), see 10.4.8 "Other Parts" under 10.4 "Optional Parts".

1.4.10 [WizAArd] Launcher

[WizAArd] launcher is the menu option used to start the software for operation and management of the AA-7000 series.



Fig. 1.11 [WizAArd] Launcher

No.	Name	Function
0	[Administration]	This is the menu for carrying out system management. Using this menu, you can register, change and delete the user management policy and users, and view the system management history information. For details, see Chapter 5 "User Administration".
0	[Operation]	This is the menu for carrying out measuring operations. Click the AA- 7000 icon to start the WizAArd software. For details, see Chapter 3 "Software Operation Flow and Basic Operation" and Chapter 4 "Measurement Procedures".
8	[Validation]	This is the menu for validity verification (validation) of the hardware and software. For details, see Chapter 7 "Hardware Validation" and 5.5 "Performing Software Validation".

NOTE

[Agent Connection Setting] in the [Administration] menu becomes operational when WizAArd Agent Connection Kit (optional) is installed.

1.4.11 Safety Devices

The AA-7000 series incorporates the safety devices indicated below.

If a sensor detects an abnormality during flame combustion, the electromagnetic valve of the gas control unit closes and the flame is automatically extinguished.

Name	Function
Vibration sensor	Monitors the vibration imparted to the instrument.
Fan stop sensor	Monitors the operation status of the fan in the back of the instrument.
Drain level sensor*	Monitors the level in the drain tank.
Support gas pressure monitor*	Monitors the supply pressure of support gas (air or nitrous oxide) being supplied to the instrument.
Fuel gas pressure monitor*	Monitors the supply pressure of fuel gas (acetylene) being supplied to the instrument. During a gas leak inspection, it detects whether the pressure drop is within the permissible range to judge whether there is a gas leak.
Flame monitor	Monitors the luminous strength of the flame. For details, see 1.4.2 "Burner Compartment" No. 9 .
Flashback monitor device*	Monitors flashback occurrence. If flashback occurs, implement the "Measures When Flashback Occurs".

* Not featured on the AA-7000G.

NOTE

1. When a safety device is actuated, the following message is displayed after closing the error message window.

\bigcirc	A safety device was activated. Please make sure to ask a service engineer to conduct the safety check if a flashback occurred.
	If a flashback didn't occur, please remove causes of errors displayed previously, and ignite. The errors are recorded in the instrument log. Do you wish to show the instrument checklist before you ignite?
	Yes
	Tes [

2. The status of all the safety devices except the flame monitor can be checked in the [Gas Controller Status] dialog box.

For details, see 2.7 "Checking the Gas Controller Status (AA-7000F, AA-7000F/AAC)".

3. When a safety device is actuated, a message is displayed on the screen and the buzzer sounds. For details, see 9.3 "When the Buzzer Sounds".

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Chapter 2 Basic Operation

CONTENTS

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2.1.1 Activation

(1) Turn ON the PC to start Windows.

CAUTION

Never turn off the power to the PC or press the PC reset switch while Windows is running. This may cause Windows to fail to boot afterward.

(2) After the Windows desktop is displayed, set the power switch of AA-7000 main unit to the ON position ("|" position).

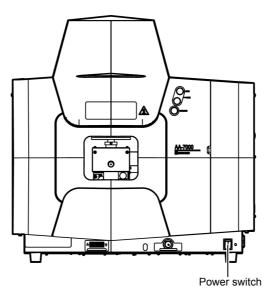
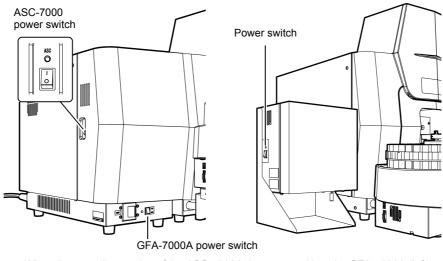


Fig. 2.1 Power Switch of AA-7000

NOTE

When the power to the instrument is turned on, the buzzer sounds once (pip). Following the buzzer, a selfdiagnosis session starts. When it is completed successfully, the buzzer sounds three times (pip, pip, pip). (3) When using the Autosampler ASC-7000 or Graphite Furnace Atomizer GFA-7000A, set the ASC-7000 power switch or the GFA-7000A power switch to the ON position (" | " position).



When the controller section of the ASC-7000 is incorporated into the GFA-7000A (left) When the ASC-7000 is mounted on a stand (right)

Fig. 2.2 Power Switches of GFA-7000A and ASC-7000

(4) Maintain the circuit protector on the GFA-7000A in the OFF position, and turn it ON when preparing for starting the measurement of the furnace.

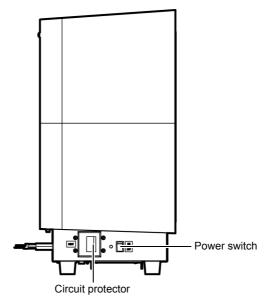
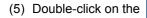


Fig. 2.3 Power Switch of GFA-7000A



(WizAArd) icon.

(6) Select [Operation] in the [WizAArd] launcher, and click the AA-7000 main unit icon.



Fig. 2.4 [WizAArd] Launcher

The WizAArd software for controlling the AA main unit is started up, and the [WizAArd Login] dialog box (Fig. 3.2) will appear at the center of the screen.

When you log into the wizard, the [Wizard Selection] dialog box (Fig. 3.3) appears.

After the WizAAd software is started up, simply follow the messages displayed on the screen to complete the settings necessary for measurement (wizard function).

2.1.2 Quitting

- (1) Confirm that all necessary WizAArd data has been saved.
- (2) Quit WizAArd with the [File] [Exit] command.
- (3) On quitting, shut off all main valves.

When using the GFA-7000A, shut off the main valve for the cooling water too.



Fig. 2.5 Closing the Main Valve

- (4) Quit Windows.
- (5) Verify that there is no remaining disk activity by checking the access indicator on the front panel of the PC, and turn off the PC.
- (6) Turn off the power switch at the AA main unit. The power indicator (green) should turn off.

NOTE

When the ASC-7000 and/or GFA-7000A are used, turn off their respective power switches.

CAUTION

Always turn OFF the GFA circuit protector after use.

If the circuit protector is left ON, the GFA may be damaged if a problem occurs with the power supply.

This is the procedure for connecting from WizAArd to the AA-7000 main unit and performing initialization.

- (1) Check that there is nothing that will obstruct the light path in the burner compartment in the AA-7000 main unit.
- (2) If the [WizAArd Selection] dialog box is displayed, click [Cancel].

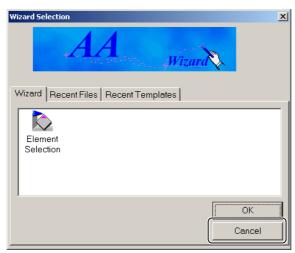


Fig. 2.6 [WizAArd Selection] Dialog Box

(3) Select [Instrument] - [Connect] from the WizAArd menu.

The initialization of the instrument starts.

For details, see the following sections:

- Flame continuous method: 3.1.6.1 "Initializing the Instrument"
- Flame micro sampling method: 3.2.6.1 "Initializing the Instrument"
- Furnace method: 3.3.6.1 "Initializing the Instrument"

NOTE

If as a result of initialization "O No Test (Not Connected)" is indicated for the ASC connection or GFA connection, check that the ASC-7000 or GFA-7000A power switch is ON. If the ASC connection or GFA connection is indicated as not connected despite the fact that the relevant power switch is ON, or if another item is flagged up as NG, stop using the instrument and contact your Shimadzu representative.

(4) When using the flame method, the [Instrument Check List for Flame Analysis] screen is displayed.

When using the furnace method, a screen on which you can select whether or not to implement the check list for the flame analysis is displayed.

For details, see the following sections:

- Flame continuous method: 3.1.6.2 "Instrument Check List for Flame Analysis"
- Flame micro sampling method: 3.2.6.2 "Instrument Check List for Flame Analysis"
- Furnace method: 3.3.6.2 "Bypass the Instrument Check List for Flame Analysis"



WARNING

- Do NOT touch the terminals of the lamp sockets while the power is on. Be sure to turn the lamp OFF before removal or fitting. If it is not OFF there will be a risk of electric shock.
- To prevent electric shock, fit the cap provided to empty sockets.

CAUTION

• To install the lamp, be sure to insert it until it reaches the bottom of the socket, and then secure it with the lamp securing ring.

If the lamp is not fully inserted, it is not properly positioned and this can cause a failure in lighting the lamp.

NOTE

Do NOT touch the window section of the lamp with bare hands.

Soiling such as finger grease will adhere to the window and diminish the strength of the lamp, affecting performance.

(1) Open the cover on the right side of the main unit.

Handle the grip to open or close the cover.

- (2) Remove the cap from the socket where the lamp is installed.
- (3) Remove the lamp securing ring from the lamp lock screw.
- (4) Insert the hollow cathode lamp into the lamp socket.
- (5) Pass the lamp through the lamp securing ring and then firmly tighten it to the lamp lock screw.

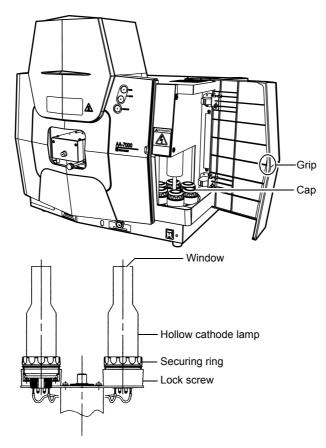


Fig. 2.7 Mounting a Hollow Cathode Lamp

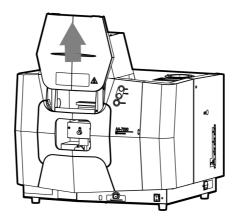
WARNING

• Wait at least 30 minutes after the flame has been extinguished before starting maintenance inside the burner compartment.

Otherwise you risk burns.

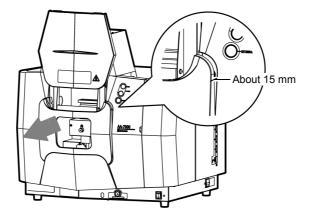
2.4.1 Removing the Front Panel

(1) Slide the door on the front face of the chimney upward.

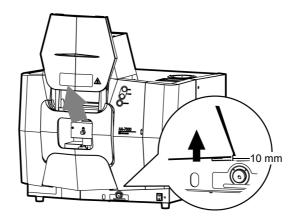


(2) While supporting the door that has been slid upward with your hand, pull the upper part of the front panel out toward you.

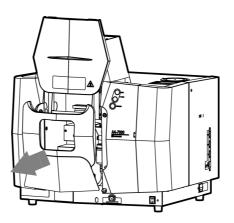
The front panel stops when it has tilted about 15 mm toward you.



(3) In this state, pull the front panel approximately 10 mm upward and toward you.

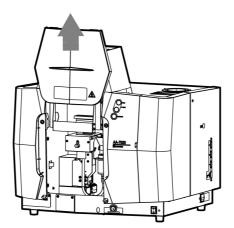


(4) Pull the front panel out toward you.

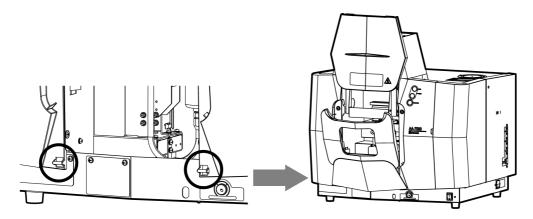


2.4.2 Mounting the Front Panel

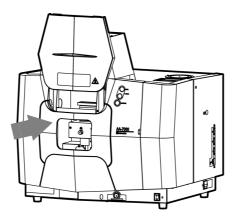
(1) Slide the door on the front face of the chimney upward.



(2) While supporting the door that has been slid upward with your hand, insert the lower part of the front panel into the two catches at the bottom of the front face of the instrument.



(3) Using the bottom of the front panel as the fulcrum, push in the top part of the panel.



2.5

Removing and Mounting the Chimney (AA-7000F, AA-7000F/AAC)

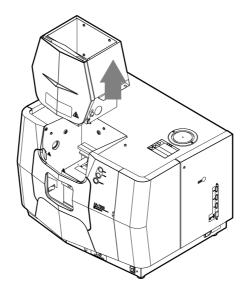
WARNING

• Wait at least 30 minutes after the flame has been extinguished before starting maintenance inside the burner compartment.

Otherwise you risk burns.

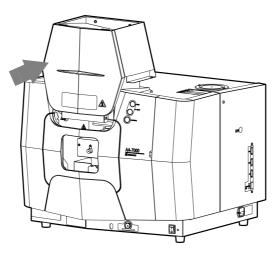
2.5.1 Removing the Chimney

(1) Remove the chimney by drawing it upward.

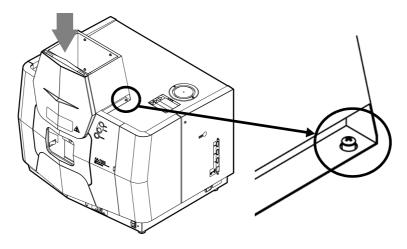


2.5.2 Mounting the Chimney

(1) Insert the chimney by working away from yourself and toward the burner compartment, and keeping the chimney a little raised so that it will not make contact with the front panel.



(2) Place the chimney on the body of the instrument so that the two pins will coincide with the two holes, as shown in the figure below.



Details of each of the screens in the software are given in the help information. This section explains how to refer to the help information.

NOTE

There is no help information for [Hardware Validation] and [Program Check] in the validation menu.

For explanations of each of the screens, see Chapter 7 "Hardware Validation" and 5.5 "Performing Software Validation".

2.6.1 Calling up the Help Screen

Using online help

"Online help" is a function that displays help information that corresponds to the currently displayed screen. To display the online help information, press the [F1] key on the keyboard.

Using the topic search

By using the topic search function, you can find the topic that you want to refer to. The method for displaying the topic search is explained below.

WizAArd software

Click [Search for Help on] in the [Help] menu.

ninistrato	or)
Window	Help
	Search for Help on
	About WizAArd

Fig. 2.8 Selecting [Search for Help on]

NOTE

To start up the WizAArd, select [Operation] from the [WizAArd] launcher, then click the AA-7000 icon.

System administration menu

Click (2) (the help button) on the [WizAArd] launcher.



Fig. 2.9 Help Button on the [WizAArd] Launcher

NOTE

To call up the [WizAArd] launcher, double-click the



(WizAArd) icon.

2.6.2 Explanation of the Help Screen

Index

(1) Use the [Contents] tab to display the item that you want to refer to.

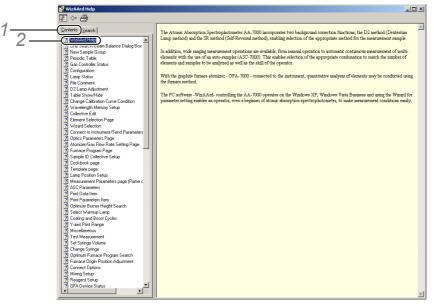


Fig. 2.10 Index of the Help Screen

- 1. Click the [Contents] tab.
- 2. Click the item that you want to refer to.

The explanation of the selected item will be displayed.

Search

(1) Use the [Search] tab to display the item that you want to refer to.

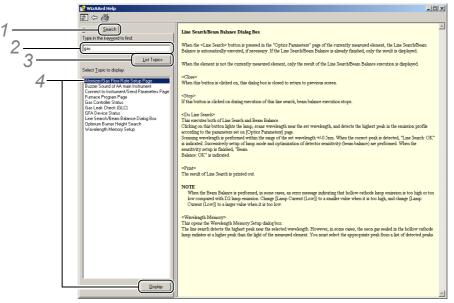


Fig. 2.11 Searching the Help Screen

- 1. Click the [Search] tab.
- 2. Enter the key word for the item you want to refer to.
- 3. Click [List Topics].
- 4. Either click the item that you want to refer to from among the displayed search results, or click [Display].

The explanation of the selected item will be displayed. The phrase that you searched for is highlighted in the text.

Key words

(1) Use the [Index] tab to display the item you want to refer to.

NOTE There is no [Index] screen in the WizAArd software.

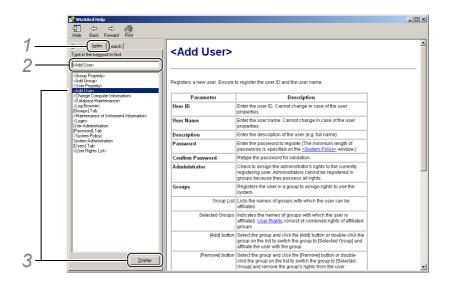


Fig. 2.12 Searching the Help Screen by Key Words

- 1. Click the [Index] tab.
- 2. Enter the key word for the item you want to refer to.
- Either click on the item that you want to refer to, or click [Display]. The explanation of the selected item will be displayed.

2.7

Checking the Gas Controller Status (AA-7000F, AA-7000F/AAC)

The [Gas Controller Status] dialog box displays the status of the gas controller safety devices and the inspection results.

WARNING

• If a gas leak is detected, stop using the instrument immediately.

If leaked gas is ignited it can cause a fire.

For details, see "Measures When Gas Leakage Is Detected".

Gas Leak Check	No gas l	eak is detected.
Drain Level	ОК	
Support Gas Pressure Monitor Level	LOW	
Fuel Gas Pressure Monitor Level	LOW	
Vibration Sensor	ОК	Reset
Fan Stop Sensor	ОК	
Burner Select Sensor Check	ОК	
Drain Level Sensor Check	ОК	Check expires 30 days later
Support Gas Pressure Monitor Check(Air)	ОК	
Support Gas Pressure Monitor Check(N2O)	ОК	Check expires 30 days later
Fuel Gas Pressure Monitor Check	ОК	Every Initialization
Flashback Monitor	Flashba	k is not detected.

Fig. 2.13 [Gas Controller Status]

NOTE

The AA-7000G has no [Gas Controller Status] dialog box.

No.	ltem	Function
0	[Gas Leak Check]	Indicates the status of gas leakage detection. Ignition is possible when [No gas leak is detected.] or [Checking (Ignite OK)] is displayed. If a gas leak is detected, stop using the instrument immediately. If no inspection has been implemented yet, carry out an inspection by selecting [Instrument] – [Gas Leak Check].
0	[Drain Level]	Indicates the status of the drain tank level. If the indication is [OK], ignition is possible. If the indication is [NG], ignition is not possible.
8	[Support Gas Pressure Monitor Level]	Indicates the status of the support gas pressure inside the gas controller.

No.	Item	Function
0	[Fuel Gas Pressure Monitor Level]	Indicates the status of the fuel gas pressure inside the gas controller.
6	[Vibration Sensor]	Indicates whether or not vibration has been detected. If the indication is [OK], ignition is possible. If the indication is [NG], ignition is not possible. Confirm safety in the vicinity and then press the [Reset] button.
6	[Fan Stop Sensor]	Indicates whether or not the fan in the body of the AA instrument has stopped. If the indication is [OK], ignition is possible. If the indication is [NG], ignition is not possible. Check whether or not the rotation of the fan has been impeded by a foreign body.
0	[Burner Select Sensor Check]	Indicates the inspection results when the instrument is initialized. If the indication is [OK], ignition is possible. If the indication is [NG] or [No Check], ignition is not possible. Reconnect to the instrument by selecting [Instrument] - [Connect], then implement an inspection from the [Initialize] screen.
8	[Drain Level Sensor Check]	Indicates the inspection results when the instrument is initialized. If the indication is [OK], ignition is possible. If the indication is [NG] or [No Check], ignition is not possible. Reconnect to the instrument by selecting [Instrument] – [Connect], then implement an inspection from the [Initialize] screen.
0	[Support Gas Pressure Monitor Check (Air)]	Indicates the inspection results when the instrument is initialized. If the indication is [OK], ignition is possible. If the indication is [NG] or [No Check], ignition is not possible. Reconnect to the instrument by selecting [Instrument] - [Connect], then implement an inspection from the [Initialize] screen.
•	[Support Gas Pressure Monitor Check (N ₂ O)]	Indicates the inspection results when the instrument is initialized. If the indication is $[OK]$, the $N_2O-C_2H_2$ flame can be used. If the indication is $[NG]$ or $[No Check]$, the $N_2O - C_2H_2$ flame cannot be used. To use the $N_2O - C_2H_2$ flame, reconnect to the instrument by selecting [Instrument] - [Connect], then implement an inspection from the [Initialize] screen.
0	[Fuel Gas Pressure Monitor Check]	Indicates the inspection results when the instrument is initialized. If the indication is [OK], ignition is possible. If the indication is [NG] or [No Check], ignition is not possible. Reconnect to the instrument by selecting [Instrument] - [Connect], then implement an inspection from the [Initialize] screen.
Ø	[Flashback Monitor]	Indicates the flashback detection status. When flashback has been detected, the instrument cannot be used. Request your Shimadzu representative to inspect the instrument.

NOTE

When any of the sensors of the gas controller detects an error, the buzzer sounds.

For details, see 9.3 "When the Buzzer Sounds".

2.8

Supplying Water to the Drain Tank (AA-7000F, AA-7000F/AAC)

WARNING

When handling chemicals, wear protective goggles and protective gloves.

If chemicals get into the eyes it can cause loss of sight. If any chemical does get into the eyes, wash it out immediately and have your eyes examined by a doctor.

(1) Insert the drain tube into the waste liquid container.

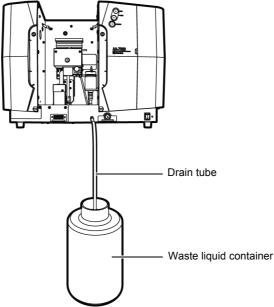


Fig. 2.14 Drain Tube and Waste Liquid Container

(2) Remove the front panel.

For details, see 2.4.1 "Removing the Front Panel".

(3) Remove the cap of the drain tank and take out the drain sensor.

NOTE

If the instrument has already been connected, taking the drain sensor out of the drain tank causes the buzzer to sound continuously - pip-pip...pip-pip... - and the message shown below is displayed. If this happens, click [OK] when you have finished the operation.



(4) Supply water to the drain tank until it is overflowing from the outlet.

NOTE

- A rinsing bottle (made of polythene) is useful for supplying water.
- If using a solvent other than water, ensure that it is a solvent to which the material of the rinsing bottle has chemical resistance.

(5) Mount the drain sensor and close the cap.

Chapter 3 Software Operation Flow and Basic Operation

This chapter describes the simple operating procedures using this wizard for the flame method, the flame micro sampling method and the furnace method. For a detailed explanation of the display items, menus and buttons on each screen, refer to the Help for the screen concerned.

CONTENTS

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3.2	Software Basic Operation (Flame Micro Sampling Method)	3-27
3.3	Software Basic Operation (Furnace Method)	3-54
3.4	Saving the Template	3-77
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3.6	Operating the MRT Work Sheet	3-90

Software Basic Operation (Flame Continuous Method)

3.1.1 Operation Flowchart (Flame Continuous Method)

This Wizard has a sequence for setting as shown below. You can proceed to the next step or return to the preceding step by using the [Next] or [Back] button, respectively.

(Starting up the AA Software)

 \downarrow

3.1

- 1. WizAArd Login
- 2. Wizard Selection
- 3. Select elements to be measured and edit the parameters.
- 4. Edit sample preparation parameters and QA/QC settings.
- 5. Connect to Instrument/Send Parameters
- 6. Optics Parameters
- 7. Atomizer/Gas Flow Rate Setup

 \downarrow

```
(Finish)
```

 \downarrow

(Start Measurement)

NOTE

When measuring multiple elements, you cannot set parameters for the elements other than the current measurement one on the "8. Optics Parameters" and "9. Atomizer/Gas Flow Rate Setup" pages. If you use the ASC to measure multiple elements automatically and you need to modify the parameters for other elements than the current measurement one, you can change these parameters by using the [Edit Parameters] button in the "3. Element Selection" page.

3.1.2 Logging in WizAArd

(1) Select [Operation] in the [WizAArd] launcher, and click the AA-7000 main unit icon.



Fig. 3.1 [WizAArd] Launcher

The [WizAArd Login] dialog box will appear at the center of the screen.

(2) Enter "Admin" to the Login ID box and no password to the Password box to log in for the first use.

NOTE

If a login ID and password are already specified, only the authorized user is permitted to use the WizAArd. Therefore, correctly enter the items to log in the WizAArd.



Fig. 3.2 [WizAArd Login] Dialog Box

The [WizAArd Selection] dialog box will appear at the center of the screen.

3.1.3 Wizard Selection

(1) If you are making a new parameter set, select the Element Selection icon on the [Wizard] sheet and click on [OK].

NOTE

For [Recent Files] sheet and [Recent Templates] sheet, you can open the recent files or templates quickly by selecting from the list.

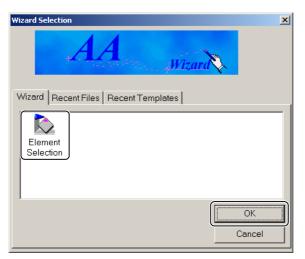


Fig. 3.3 [Wizard Selection] Dialog Box

The [Element Selection] page will appear.

3.1.4 Element Selection

The [Element Selection] page allows you to select the element to be measured, determine the measuring order, and edit the measurement parameters.

When the element to be measured and measurement method are selected, the standard parameters are displayed automatically.

(1) Click on the [Select Elements].

Element Selection	_					
	Select elements to be me	easured ar		arameters. At the E	Comment	Select Elements
	Lismone	Wain	DOCKSC #	Actio Em	Commerie	Edit Parameters
						Up
						Down
						Delete
						Meas. Element:
						
						,
						Connect
		< <u>B</u>	ack	<u>N</u> ext >	Finish	Cancel Help

Fig. 3.4 [Element Selection] Page

The [Load Parameters] will appear.

(2) In this page, select the element first.

NOTE

You can use one of the methods below to select the element.

- Enter the element symbol directly in the element field from the keyboard.
- Click on the [▼] button at the right of the element field, and select the element from the element symbol list shown in alphabetical order.
- Click on the [Periodic Table] button and select the element from the periodic table.

	Load Parameters			×
	Cookbook Template			
	Cu Periodic Table			
1—	Flame Continuous	Comment :	Flame	
-	C Flame Micro Sampling	Wavelength : Slit Width :	324.8 nm 0.7 nm	
	HVG MVU	Lamp Mode : Lamp Current Low(Peak) :	BGC-D2 8 mA	
	C Furnace	Burner Height : Burner Lateral : Burner Angle :	7.0 mm O pulse O degree	
2—	Normal Lamp	Flame Type : Fuel Gas :	Air-C2H2 1.8 L/min	
	O SR Lamp	Support Gas :	15.0 L/min	
3—	Using ASC			
				T
		4		
4—			OK)	Cancel Help

Fig. 3.5 [Cookbook] Page in [Load Parameters]

- 1. Next, select [Flame Continuous] for the measurement method by the radio button.
- 2. Select [Normal Lamp] when using a normal hollow cathode lamp ([SR Lamp] is selected only when the SR method is used as the background correction method).
- 3. When using the autosampler, click on [Using ASC] check box.
- 4. After finishing the settings, click on the [OK] button. If the message on the lamp setup appears, proceed to the section 3.1.4.1 "Lamp Setting Procedure".

WizAArd	×
(į)	None of the set lamps can be used for this setting. Are you going to make setting immediately?
	<u>Yes</u> <u>N</u> o

Fig. 3.6 Message Box

(3) To continue measuring multiple elements, temporarily return to the [Element Selection] page, click on [Select Elements], and then select the next element. Repeat the sequence of clicking on the [Select Elements] button, selecting an element and then clicking on the [OK] button, the number of times required.

NOTE

When you return to the [Element Selection] page after completing selecting elements, the selected elements are displayed in the order of selections. If there is any element you want to delete, click on the appropriate row to highlight it and then click on the [Delete] button. The [Meas. Element] field in the lower right part of the screen indicates the element to be measured first.

- (4) If you click on the [Edit Parameters] button, the parameters for the element on the highlighted row on the [Element Selection] page will be displayed. Those parameters may be modified as necessary. First proceed with the operations without using this function.
- (5) Click on [OK].
- (6) If you click on the [Next] button, the [Preparation Parameters] page will be displayed.

NOTE

When analyzing plural elements sequentially, the order on the [Element Selection] page becomes the measurement order. If you need to change the order, click on the element to highlight it and then click on [Up] or [Down] to move the row. If the [Meas. element] at the right lower of the page is different from the first row element, the measurement is started from the [Meas. Element] and the elements upper than it are not to be measured.

3.1.4.1 Lamp Setting Procedure

If a message about the lamp is displayed at step 4. in (2) above, set the lamp by following the procedure below.

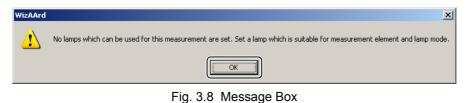
(1) Click on the [Yes] button.



Fig. 3.7 Message Box

The [Edit Parameters] page will appear with the message on the lamp setup displayed again.

(2) Click on the [OK] button.



The [Optics Parameters] sheet of the [Edit Parameters] page is displayed.

(3) Click on the [Lamp Pos. Setup] button.

dit Parameters				×
	/Gas Flow Rate Setup Jence Repeat Measu	Weight Correction Factors irement Conditions Measureme	Y-axis Print Range nt Parameters Calibra	Miscellaneous
	Cu Wavelength: (185.0 - 900.0 r Slit Width (nm): Lamp Mode: Socket #: If you click on th turn the lamp tur Lamp ID:	324.8 m) 0.7 BGC-D2 NONE Lamp Pos. Setup. Lamp Pos. Setup. Lamp Pos. Setup. Lamp Pos. Setup. Lamp Pos. Setup.		
	Lamp ON: Lamp Status:		- Search:	NONE
			Warmup Lamp	Line Search
			OK Ca	encel Help

Fig. 3.9 [Edit Parameters] Page

The [Lamp Position Setup] dialog box will be displayed.

(4) Enter [Element] (select the element symbol from the drop-down list) and [Lamp Type] (select the normal lamp or SR lamp from the drop-down list) of the lamp that has actually been allocated to each socket number. This allows you to select the lamp registered in [Lamp ID]. If any other element is to be measured, repeat these steps for convenience sake.

		Туре	Lamp ID	Judge	Life Time	Used Time	Unit		OK
Cu	u 🔽	Normal	Cu-1	ок	5000	0.0	mA*hrs	1	Cance
: [[]	None								
	None								<u>Print</u>
1	None								
1	None							1	
i 1	None								

Fig. 3.10 [Lamp Position Setup] Dialog Box

NOTE

When the [Lamp Position Setup] dialog box remains displayed, the lamp turret can be rotated to allow you to mount or replace the lamp.

- (5) Select the lamp to be used and then click on the [OK] button. You will return to the previous [Optics Parameters] sheet.
- (6) Enter [Socket Number] and click on the [OK] button.

3.1.5 Preparation Parameters

This page allows you to enter Calibration Curve Settings and Sample Group Settings. If you select multiple elements in the [Element Selection] page, multiple rows are displayed in this window.

(1) Click on the row including the desired element for settings. Click on the [Calibration Curve Setup] button to display the [Calibration Curve Setup] page.

For details, see 3.1.5.1 "Calibration Curve Setup".

Click on the [Sample Group Setup] button to display the [Sample Group Setup] page.

For details, see 3.1.5.2 "Sample Group Setup".

Preparation Parameters					×
		s, sample preparati	on parameters and QA	/QC settings. Calibration Curve Setup Sample <u>G</u> roup Setup	
			News	in a constant of the	
	_	< <u>B</u> ack	<u>N</u> ext > Fin	ish Cancel Help	

Fig. 3.11 [Preparation Parameters] Page

Now assume entering [Calibration Curve Setup] and [Sample Group Setup] under the standard parameters.

(2) Set the [Preparation Parameters] and click [Next].

3.1.5.1 Calibration Curve Setup

On this screen you can make the settings relating to the calibration curve. Upon setting the calibration curve and clicking [OK], you will be returned to the [Preparation Parameters] page.

		3	
	Calibration Curve Setup		×
0	Method of Standard Addition Order 1st Conc. ppm Unit Zero Intercept	Common Settings of Preparation Parameters Repeat Conditions	OK Cancel
Ğ—	QC Blank/QC Standard Setup		
0	Blank Preparation Parameters		
U	Auto Frequency		
0 —	Auto Frequency 20 0.0000		
	Measurement Sequence for Calibration Curve		
		Delete Line	
	Action Sample ID True Value		
0	STD 0.5000 STD 1.0000		
	STD 1.0000 STD 2.0000		

Fig. 3.12 [Calibration Curve Setup] Page

Since the calibration curve method is selected here, do not tick the [Method of Standard Addition] option. To use the standard addition method or the simple standard addition method, see the section 4.6 "Standard Addition Method and Simple Standard Addition Method".

② [Order] means the order of the calibration curve equation. When the calibration curve is linear, select "1st". If the calibration curve is likely to curve more or less, you may wish to select "2nd" or "3rd". Since this setting may be changed after viewing the actually measured values, select "1st" for now.

3 The [Zero Intercept] is used to force the calibration curve to pass through the origin. This setting may be changed later.

Select [Conc. Unit] of the prepared standard samples. Clicking on [▼] button to select it from the list.

The explanation proceeds forward without the QAQC setup. Therefore, do not click on the [QC Blank/QC Standard Setup] button. When the QAQC settings are necessary, refer to the Chapter 6 "QA/QC Setup".

G Clicking on the [Repeat Conditions] button displays the [Repeat measurement Conditions] page. The number of measurements for the same one sample is set here. The default value of the number of measurement repetitions is "1" in the case of flame continuous method. Click on the [OK] button to close the window with the default value remaining unchanged.

		Repea	t Conditions			
	Num. of Reps.	Max. Num. of Reps.	RSD Limit	SD Limit	Retry	OK Cancel
Blank	1	1	99.90	0.0000		
Standard	1	1	99.90	0.0000		
Sample	1	1	99.90	0.0000		
Reslope	1	1	99.90	0.0000		

Fig. 3.13 [Repeat Measurement Conditions] Dialog Box

In the [Blank Preparation Parameters], set up the automatic periodic blank measurement. The automatic periodic blank measurement is a function to create a measurement procedure on the MRT to eliminate the effect of baseline drift by inserting a blank measurement in a fixed interval. Use this function when there are many samples to be measured or when the baseline drifts.

- When periodic blank measurement is not performed: Do not tick the [Auto] field.
- When periodic blank measurement is performed:
- a. Tick the [Auto] field.
- b. Enter a value in the [Frequency] field to specify how many samples are measured between blank measurements.
- c. When using the ASC, the [Pos.] field is displayed if the [Using ASC] is ticked in the [Load Parameters] of [Element Selection] page. Enter the position of the blank sample on the turntable of the ASC.

③ Reslope is the sensitivity correction measurement. More specifically, a standard sample of a known concentration is measured during the measurement of unknown samples. Based on the measured absorbance, the slope of the calibration curve is corrected. Subsequently, the corrected new calibration curve will be used to calculate concentrations. If the [Auto] field is ticked in the [Reslope Preparation Parameters], reslope measurement is carried out at the concentration specified in [Conc.] by the specified frequency. The setup procedure is the same as in the [Blank Preparation Parameters].

In [Measurement Sequence for Calibration Curve], enter the number of standard samples and their concentrations. Enter the number of standard samples in the [No. of Lines] field and click on the [Update] button. A table with that number of rows will be created. In this table, the default values for the concentrations of standard samples are already displayed under the standard parameters, but can be changed with the values for the actually prepared standard samples. If the ASC is used, the [Pos.] field is displayed. Enter the positions of the turntable (1 to 60, R1 to R8).

3.1.5.2 Sample Group Setup

This window allows you to specify a sample group. If similar kinds of samples that are to be prepared with the same pretreatment are grouped, the effect of interference and the suitability of the pretreatment can be conveniently validated using the QA/AC function.

Upon making the sample group settings and clicking [OK], you will be returned to the [Preparation Parameters] page.

1		irrent Sample Gr lew Sample Grou : Setup	ıp		- We Volu Dilu	orrection Factors hight Factor: 1.00 ume Factor: 1.00 tion Factor: 1.00	00000	-Actual Conc.	Unit
Sam	Method SA C								
Spike	e 0.01		nce						
Spike			nce Pos.	WF	Add to MRT		No. of Samples	: ¹⁰ U	Jpdate
Spike Unki	nown/Spike Meas	surement Sequer	Pos.	1.000000	MRT			: 10 U	Ipdate
Spike Unki 1 2	nown/Spike Meas	surement Sequer	Pos. 1 2	1.000000 1.000000	MRT IZ		Samples		^j pdate
Spike Jnki 1 2 3	nown/Spike Meas Action UNK UNK UNK	surement Sequer	Pos. 1 2 3	1.000000 1.000000 1.000000	MRT V V		Samples	: 10 U	lpdate
Jnki 1 2 3 4	nown/Spike Meas Action UNK UNK UNK UNK	surement Sequer	Pos. 1 2 3 4	1.000000 1.000000 1.000000 1.000000	MRT V V V V V		Samples Colle	: ∫⊂ cti∨e Setup −	Jpdate
Spike	nown/Spike Meas Action UNK UNK UNK UNK UNK	surement Sequer	Pos. 1 2 3 4 5	1.000000 1.000000 1.000000 1.000000 1.000000	MRT IV IV IV IV IV IV		Samples Colle		Jpdate
Spike	nown/Spike Meas Action UNK UNK UNK UNK	surement Sequer	Pos. 1 2 3 4	1.000000 1.000000 1.000000 1.000000	MRT V V V V V V V V		Samples Colle	: ∫⊂ cti∨e Setup −	Jpdate
Spike Jnki 1 2 3 4 5 6 7	nown/Spike Meas Action UNK UNK UNK UNK UNK UNK	surement Sequer	Pos. 1 2 3 4 5 6	1.000000 1.000000 1.000000 1.000000 1.000000 1.000000	MRT		Samples Colle	ctive Setup	Ipdate
Spike Jnki 1 2	nown/Spike Meas Action UNK UNK UNK UNK UNK UNK UNK	surement Sequer	Pos. 1 2 3 4 5 6 7	1.000000 1.000000 1.000000 1.000000 1.000000 1.000000 1.000000	MRT V V V V V V V V		Samples Colle	ctive Setup	Jpdate

Fig. 3.14 [Sample Group Setup] Page

- 1 In this example, use the default value of "1" for [Sample Group Number] and proceed forward without selecting the [Update Current Sample Group Settings] option and the [New Sample Group] option.
- 2 The [QA/QC Setup] will be described in the Chapter 6 "QA/QC Setup".

Senter [Weight Correction Factors]. These factors are required to calculate the actual concentrations. Weight Factor [WF], Volume Factor [VF], Dilution Factor [DF], and Correction Factor [CF] are used for the following equation:

Actual concentration = Concentration × [VF] × [DF] × [CF]/[WF]

The actual concentration is calculated with the above equation. The function for automatically converting the units is not available. If conversion of any unit is required, make adjustment using Correction Factor [CF]. (See the example.) If the calculation of the actual concentration is not required, leave all the factors as "1".

To display the unit of the actual concentration, click the $[\mathbf{V}]$ button for [Actual Conc. Unit] and then select the unit from the drop-down list.

Example

Assume that 2 grams of sample is weighed, made up to 50 mL with a solution, and then diluted by a factor of 5 for measurement. To obtain the actual concentration from measured concentration, enter 2 (g) for Weight Factor [WF], 50 (mL) for Volume Factor [VF], 5 for Dilution Factor [DF], and 1 for Correction Factor [CF] as follows:

Actual concentration = Concentration × 50 (mL) × 5 (times) × 1/2 (grams)

In the same example, to obtain the actual concentration (%) from the concentration (ppm) by converting the unit, use 0.0001 for Correction Factor [CF] since 1 ppm is equal to 0.0001%, as follows: Actual concentration = Concentration \times 50 (mL) \times 5 (times) \times 0.0001/2 (grams)

Remarks

The unit of ppm indicates a concentration using the unit of 10^{-6} . In the atomic absorption analysis, both of μ g/g for solid samples and μ g/mL (mg/L) for liquid samples are, in practice, expressed in ppm.

- The [Unknown/Spike Preparation Parameters] allows you to enter the preparation parameters for unknown samples and spike samples. Spiking is one of the QA/QC techniques that are used to obtain the recovery rate by adding a solution of a known concentration to an unknown sample. In this example, proceed forward without entering a value (i.e., using the [S A Conc.] of 0.0000).
- In the [Unknown/Spike Measurement Sequence], enter the number of unknown samples and sample ID's. Enter the number of unknown samples in the [No. of Samples] field and click on the [Update] button. A table with that number of rows will be created. Sample ID can be entered one by one in the table, but can be entered at a time by clicking on the [Collective Setup] button. If the ASC is used, enter each turntable position (1 to 60) in the [Pos.] field.

NOTE

In the above ③, [Weight Correction Factors] has been entered. In general, [WF] varies depending upon each sample and can be entered in the [Unknown /Spike Measurement Sequence] table. Only the sample for which the [Add to MRT] field is ticked is inserted into the MRT worksheet on the main screen. The created [Unknown/Spike Measurement Sequence] table can be saved or loaded.

If you click on the [Collective Setup] button on the [Sample Group Setup] page of Fig. 3.14, the [Sample ID Collective Setup] dialog box will be displayed.

Sample ID Collective Setup

Clicking [Collective Setup] on the [Sample Group Setup] page displays the [Sample ID Collective Setup] dialog box.

Sample ID Collective Setup	×
– Number of Samples: 🔟 💻	ОК
	Cancel
Create Sample ID	
Sample ID Name Start No. Sample + 1	ASC Start Pos.
Pre-Digestion Spike (SPK) 20 C Post-Digestion Spike (PDS) 20 Duplicate (DUP)	
	Number of Samples: Create Sample ID Sample ID Sample Pre-Digestion Spike (SPK) Post-Digestion Spike (PDS)

Fig. 3.15 [Sample ID Collective Setup] Dialog Box

- 1 In the [Sample ID Collective Setup] dialog box, enter the number of unknown samples in the [Number of Samples] field.
- 2 To enter sample ID (sample name), tick the [Create Sample ID] field.

When you enter a name and starting number in the enabled [Sample ID] field, the same name will be given to all the samples with sequential numbers from the starting number given to them. If the ASC is used, specify the position of the 1st unknown sample in the [ASC Start Pos.] field. The 2nd and subsequent positions will be automatically entered in the table.

If [Pre-Digestion Spike (SPK)], [Post-Digestion Spike (PDS)], and [Duplicate (DUP)] are ticked, these measurements will be inserted in the analysis sequence for samples each in the number indicated on the right side field. Since these samples are used for QA/QC, proceed forward without ticking the above options in this example.

3.1.6 Connect to Instrument/Send Parameters

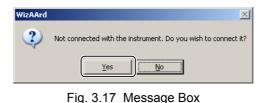
This section describes how to connect to the instrument and send the parameters. When the connection to the instrument is made, the instrument is initialized automatically.

(1) Check that the AA main unit and the related units are ON and click on the [Next] button.

Connect to Instrument/Ser	nd Parameters	×
	Power DN the instrument, and click on the [Connect/Send Parameters] button.	Connect/Send Parameters
	If the options are not recognized properly because the ASC/GFA are not powered on before initialization, click on the [Connect Options] button.	Connect Options
	44	Meas. Element: Cu:FlameCont
	< <u>B</u> ack Next >	Finish Cancel Help

Fig. 3.16 [Connect to Instrument/Send Parameters] Page

(2) Check that the chimney is correctly installed on the instrument and then click [Yes].



NOTE

If the chimney is not correctly installed on the instrument, the [Flame Monitor Check] may not be performed correctly at initialization, leading to an [NG] result.

The connection to the instrument will be started with the [Initialize] screen displayed and then the AA main unit will be initialized. After the initialization has been finished, the parameters for the element specified in [Meas. Element] are automatically sent to set up the instrument.

Alternatively, you may want to press the [Connect/Send Parameters] button in the [Connect to Instrument/Send Parameters] page to perform the same operation.

3.1.6.1 Initializing the Instrument

The results of initialization of the instrument are displayed on the [Initialize] screen. You can also check the versions of the AA-7000 main unit and the ASC and GFA ROMs on this screen.

CAUTION

• Expiration dates apply to inspections of safety devices.

Once an expiration date has passed the instrument can no longer be used. Even if the safety devices are within the expiration date for inspections, you are strongly recommended to implement an inspection at initialization.

• If the result of the inspection is that there is a fault in a safety device, a message describing the fault is displayed and the device cannot be used.

If a safety device is faulty, contact your Shimadzu representative.

initialize		
	1 A3000000000 .01 A3000000000	-0
GFA: GFA-7000 ∨1	.02 A3000000000	
ROM Check	C2H2 Valve Origin Search	•
S/N Check	Flame Monitor Check	-0
ASC Check		
GFA Check	Burner Select Sensor Check	
Slit Origin Search	Drain Sensor Check	
D2 Attenuator Origin	Support Gas Pressure Monitor Check(Air)	
🔵 Wavelength Origin Search	Support Gas Pressure Monitor Check(N2O)	
C Turret Origin Search	Fuel Gas Pressure Monitor Check	
Atomizer Up/Down	Start Leak Check	
Atomizer Fore/Back		
Testing 🔵 Succ	ess 🔶 Failure 🔿 No Test(Not Connected)	
	ОК	

Fig. 3.18 [Initialize] Screen (for AA-7000F/AAC)

No.	Name	Function
0	Instrument information	The models of the AA main unit, ASC and GFA, the ROM versions and the machine identification numbers are displayed here. If neither ASC nor GFA is connected, the ASC and GFA information is not displayed.
0	Automatic inspection points	These are inspections that the instrument performs automatically. If neither ASC nor GFA is connected, [Not Connected] () is shown for the [ASC Check] and [GFA Check] points.
0	Manual inspection points	Messages are displayed to prompt the successive performance of each of these inspection points. Carry out the inspections in accordance with the messages.
4	Start Leak Check	Starts an 8-minute automatic gas leakage inspection.

NOTE

The time required from the start of initialization to completion of the automatic inspection points is 4 to 5 minutes (it varies depending on whether or not options are installed).

(1) When initialization of the instrument starts, the instrument information is acquired and the automatic inspection points are implemented. On completion of the automatic inspection points, the [Gas Adjustment] screen is displayed. After setting the fuel gas and support gas, click [Close].

	Gas Adjustment
	Turn on the ventilation system. In case of flame analysis, open the main valves of fuel gas and support gas.
	1.Set the C2H2 supply pressure to 0.09MPa with purging C2H2.
0—	Purge C2H2 (5sec , till 5 times)
	2.Set the Air supply pressure to 0.35MPa with purging Air. In case optional flowmeter is installed, set the Air flow rate to 15.0L/min.
0	Purge Air (10sec)
	3.In case of using N2O-C2H2 flame, set the N2O supply pressure to 0.35MPa with purging N2O.
0 —	Purge N20 (10sec)
	Close

Fig. 3.19 [Gas Adjustment] Screen

No.	Name	Function
0	[Purge C2H2]	Performs the acetylene (C_2H_2) gas purge operation (5 seconds per purge, with a limit of 5 purges). During the purge operation, set the acetylene supply pressure to 0.09 MPa.
0	[Purge Air]	Performs the air purge operation (10 seconds per purge, with no limit on the number of purges). During the purge operation, set the air supply pressure to 0.35 MPa.
8	[Purge N2O]	Performs the nitrous oxide (N_2O) gas purge operation (10 seconds per purge, with no limit on the number of purges), when the high-temperature burner head (optional) is used. During the purge operation, set the nitrous oxide gas supply pressure to 0.35 MPa.

(2) Carry out the burner identification sensor inspection, drain sensor inspection, and support gas monitor inspection (air).

Clicking [Yes] starts the burner identification sensor inspection.

WizAArd
You can check the safety devices now. Burner Select Sensor Drain Sensor Support Gas Pressure Monitor(Air) Do you wish to check them? * Expires 26 days later * Igniting a flame without safety device check is unable when the check-free period expired or the previouse check(s) was(were) failed.
Fig. 3.20 Message Box
NOTE
 On clicking [No] when still within the expiration date, the following message is displayed without implementing an inspection. You are strongly recommended to perform inspections within the expiration date.
WizAArd Image: Constraint of the safety devices now. Burner Select Sensor Burner Select Sensor Drain Sensor Support Gas Pressure Monitor(Air) Do you wish to check them? * Expires 26 days later * Igniting a flame without safety device check is unable when the check-free period expired or the previouse check(s) was(were) failed.
 On clicking [No] when the expiration date has passed, the following message is displayed. If using the flame method, click [Recheck].
Initialize
The check was not executed. The flame can't be ignited, because it's beyond the safety device check-free period or the previouse check(s) was(were) failed.
OK : The initialization process is continued though the flame can't be ignited.
Recheck : The initialization process gets back to check process to enable the instrument to ignite the flame.
 If the following message is displayed after clicking [Yes], set the BURNER SELECT switch to the AIR- C₂H₂ position.
WizAArd Switch Burner Select Sensor to Air. OK
AIR-C ₂ H ₂ BURNER SELECT

(3) The drain sensor inspection starts. When the following message is displayed, remove the front panel.
 For details, see 2.4.1 "Removing the Front Panel".
 Take the sensor out of the drain tank, and lift it above the surface of the water.

 WizAArd

 Lift the Drain Sensor over the surface of water.

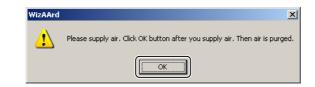
(4) Click [OK] in the [Lift the Drain Sensor over the surface of water.] dialog box.



(5) When the following message is displayed, mount the drain sensor in its original position. Mount the front panel and click [OK]. For details, see 2.4.2 "Mounting the Front Panel".

WizAArd	×
1	Move the Drain Sensor below the surface of the water.
	(K

(6) When the support gas pressure monitor inspection (Air) starts, the following message is displayed. Supply air and click [OK].



(7) When the support gas pressure monitor inspection (N₂O) starts, the following message is displayed.

WizAArd	<u>×</u>	1
!	You can check the safety device now. Support Gas Pressure Monitor(N2O) Do you wish to check It? * Expires 26 days later * Igniting N2O-C2H2 flame without safety device check is unable when the check-free period expired or the previouse check was failed.	
	<u>Yes</u> <u>N</u> o	

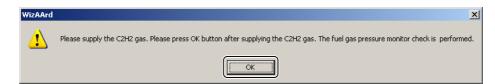
(8) Click [Yes]. The message shown below will be displayed. Supply N_2O and click [OK].

	WizAArd		×
	Please supply N2O. Clic	k OK button after you supply N2O. Then N2O is p	burged.
	<u> </u>		
NOTE			
 If the high-temper 	ature burner head (option	al) is not being used, click [No].
			ting an inspection. When the
••••	• •		I_2 flame is used, you are strongly
recommended to	perform inspections within	the expiration date.	
WizAArd			X
	device check is not performed. Igniting N2O-Ca the period.	2H2 flame is available during check-free period. Bi	ut, it is strongly recommended to check it
		()	
Even when the ex	piration date for the supp	ort gas pressure monitor ins	spection (N ₂ O) has passed,
		•	respection points, the Air- C_2H_2
flame can be used			

(9) When the fuel gas pressure monitor inspection starts, the following message is displayed.



(10) Click [Check it]. The following dialog box will be displayed. Supply C₂H₂ gas and click [OK].



NOTE			
If you click [Don't c	check it], the	following message is displayed.	
	Initialize		
	The check was The flame can'		
	OK :	The initialization process is continued though the flame can't be ignited.	
	Recheck:	The initialization process gets back to check process to enable the instrument to ignite the flame.	
		OK <u>B</u> echeck	

(11) On completion of the fuel gas pressure monitor inspection, a 8-minute automatic gas leakage inspection starts, and initialization is completed. On completion of initialization, click [OK]. The [Initialize] screen will close.

NOTE

The flame cannot be ignited during a gas leakage inspection.

3.1.6.2 Instrument Check List for Flame Analysis

- (1) After closing the [Initialize] screen, a message is displayed asking whether the flame measurement is performed. Click on the [Yes] button here. Perform the checks prior to the flame measurement in accordance with the [Instrument Check List for Flame Analysis].
- (2) Check all the items (1) through (8) and the emergency stop operation, and then tick them.

See also the introductory section "Emergency Action" and the section 4.5 "Igniting and Extinguishing the Flame".

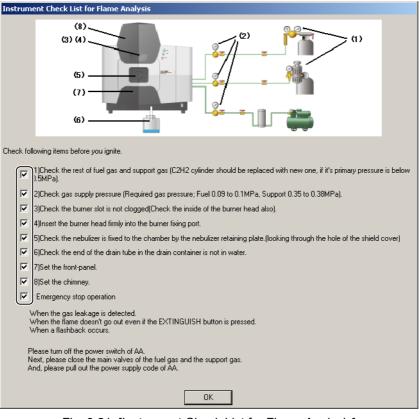


Fig. 3.21 [Instrument Check List for Flame Analysis]

(3) When all the items are checked, you can select [OK].

CAUTION

The checklist for starting the flame measurement assumes that the user checks the safety. When using flame, properly carry out the checks.

For details, see Chapter 8 "Maintenance".

3.1.7 **Optics Parameters**

The [Optics Parameters] page is used to set the parameters for the monochrometer and the lamps in the instrument. For the purpose of this example, proceed forward without changing the standard parameters (that were read out when the elements were selected). For changing each parameter, refer to the HELP information in the WizAArd software.

Optics Parameters					X
	📮 Cu				
	, Wavelength: (185.0 - 900.0 r Slit Width (nm): Lamp Mode: Socket #: If you click on th	324.8 m) 0.7 • BGC-D2 • 1 • Lamp Pos. Setup e Lamp Pos. Setup button, you can et manually and change the lamp. Cu-1 Cu-1 Line Search is necessary.	(0 - 600 mA,)	8 × 0 × • for EMISSION Line	
			Warmup Lamp	Line Search	
		< <u>B</u> ack <u>N</u> ext >	Finish	Cancel Help)

Fig. 3.22 [Optics Parameters] Page

NOTE

This page displays the wavelength, slit width, socket number, lamp current, lamp mode and so on. These parameters are set for only the element that will be firstly measured (has been specified in the [Meas. Element] field located in the lower right part of the [Element Select] page or the [Connect to Instrument/Send Parameters] page).

The measurement parameters for each element are loaded from the cookbook and automatically specified when the elements are selected.

Normally, you do not need to enter these measurement parameters. To modify them, however, you can enter a value for the wavelength and select a value for other conditions from the list pulled down by clicking on the $[\mathbf{V}]$ button. The lamp current value can be changed in units of 1 mA by clicking on the $[\mathbf{A}]$ or $[\mathbf{V}]$ buttons.

(1) Click on the [Next] button. A message will be displayed prompting you to do line search. Click on the [OK] button.

The [Line Search/Beam Balance] dialog box will be displayed and the process will be carried out automatically.

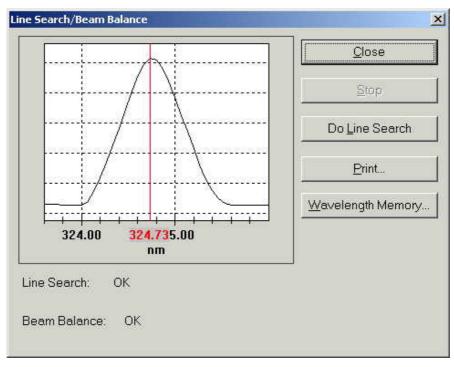


Fig. 3.23 [Line Search/Beam Balance] Dialog Box

- First the line search (wavelength matching) is carried out and then the beam balance (gain control for the detector) is performed. If only the beam balance is necessary, the line search is not performed.
- In the line search, the highest peak near the specified wavelength is detected. In some cases, however, the neon gas contained in the hollow cathode lamp radiates more intense light than the light from the element. In such a case, if the correctly analyzed line is stored by clicking on the [Wavelength Memory] button, its wavelength will be used for the subsequent line searches.
- (2) Upon completion of the process, click on the [Close] button to proceed forward to the [Atomizer/Gas Flow Rate Setup] page.

NOTE

When measuring multiple elements, you cannot set parameters for the elements other than the current measurement one on the [Optics Parameters] and [Atomizer/Gas Flow Rate Setup] pages. If you use the ASC to measure multiple elements automatically and you need to modify the parameters for other elements than the current measurement one, you can change these parameters by using the [Edit Parameters] button in the [Element Selection] page.

NOTE

SR lamp intensity is hard to stabilize in using NON-BGC mode or BGC-D2 mode in comparison with a normal lamp. Before performing the line search, wait for 15 to 20 minutes after turning on the lamp, and then start measurement.

3.1.8 Atomizer/Gas Flow Rate Setup

This page is used to specify the burner position and the flow rate of fuel gas and support gas. Select [Atomizer Position] or [Gas Flow Rate] using the radio button. The window display will be changed accordingly. Whichever parameter may be selected first.

(1) To set these parameters, ignite flame and allow a standard sample to be sucked.

Even if the ASC is used for the actual measurement, use this window to manually let the sample be sucked. Set the parameters so that the absorbance of the standard sample with a known concentration falls within the intended range of absorbance (normally the maximum absorbance is obtained).

Atomizer/Gas Flow Rate Setup		×					
Cu Cu	0.800						
Operation Object							
	0.700						
Measured Data: 0.0000							
Background Data: 0.0001	0.600						
Autozero(F3)	0.500						
Flame Type: Air-C2H2 💌							
Fuel Gas Flow Rate(L/min): 1.8	0.400						
Support Gas Flow Rate(L/min): 15.0	A b 0.300						
*When igniting a flame is not easy, please increase the fuel gas flow rate once and try to ignite. Please get the flow rate back to the value for the analysis after igniting.	0.200						
* In the case of N20-C2H2 flame, please make sure to set the gas flow rates so that the red feather of the flame gets more than 2 to 3 mm.	0.100						
Increase Fuel(F5) Flame Setup(F7)							
Decrease Fuel(F6) Gas Flow Auto(F8)	-0.100						
	-0.200						
< <u>B</u> ack Next> Finish Cancel Help							

Fig. 3.24 [Atomizer/Gas Flow Rate Setup] Page

When the [Atomizer Position] is selected

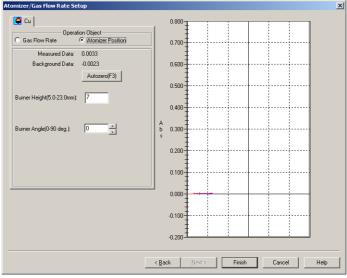


Fig. 3.25 [Atomizer Position] is Selected

- Leave the [Burner Angle] as "0" for usual use. Change it only when you want to decrease the sensitivity by changing the burner angle for high-concentration samples.
- For the [Burner Height], an appropriate value is indicated according to the element, so you need not change it for usual use. However, the optimum condition for the burner height may differ depending on the gas flow rate or the sample type. To change the value, enter a new value.

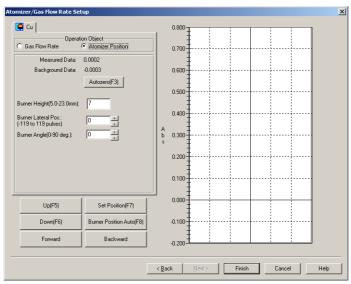


Fig. 3.26 [Atomizer Position] is Selected (with Auto Atomizer Changer)

- When the instrument is equipped with Auto Atomizer Changer (optional), clicking on the [Up] and [Down] buttons at the bottom of the page allows you to vertically change the burner position. As the burner position is vertically changed, the value in the [Burner Height] field is also changed accordingly.
- The [Burner Position Auto] button is used to obtain the optimum condition for the burner height through the measurement of the actual sample. For the steps to perform [Burner Position Auto], see the section 4.8.1 "Setting the Optimum Condition of Burner Height".

When the [Gas Flow Rate] is selected

- A type of flame, and flow rates of fuel gas and support gas can be specified.
- In the [Flame Type] field, a suitable flame type is indicated according to the element. To change
 the flame type, click on the [▼] button and select a desired flame type from the drop-down list.
 N₂O-C₂H₂ cannot be used with the standard burner head. It requires using the hightemperature burner head (optional).
- The [Increase Fuel] and [Decrease Fuel] buttons located at the lower of the page allow you to increase and decrease the flow rate of fuel gas. The current value is displayed in the [Fuel Gas Flow Rate] field.
- The [Support Gas Flow Rate] is fixed (at 15 L/min). It cannot be increased or decreased.

NOTE

If the flow meter kit (optional) has been attached, the flow rate of the support gas can be increased and decreased by using the flow rate adjusting control, and a setting can be recorded for [Support Gas Flow Rate].

- The [Gas Flow Auto] button is used to obtain the optimum condition for the flow rate of fuel gas through the measurement of the actual sample. For the steps to perform [Gas Flow Auto] see the section 4.8.2 "Setting the Optimum Condition of Fuel Gas Flow Rate".
- (2) After all the settings have been finished, click on the [Finish] button.

The wizard will be exited with the main screen displayed. To save the conditions specified here as a template, proceed to the section 3.4 "Saving the Template"; to start the measurement, proceed to the section Chapter 4 "Measurement Procedures".

3.2 Software Basic Operation (Flame Micro Sampling Method)

3.2.1 Operation Flowchart (Flame Micro Sampling Method)

This Wizard has a sequence for setting as shown below. You can proceed to the next step or return to the preceding step by using the [Next] or [Back] button, respectively.

(Starting up the AA Software)

 \downarrow

- 1. WizAArd Login
- 2. Wizard Selection
- 3. Select elements to be measured and edit the parameters.
- 4. Edit sample preparation parameters and QA/QC settings.
- 5. Connect to Instrument/Send Parameters
- 6. Optics Parameters
- 7. Atomizer/Gas Flow Rate Setup

 \downarrow

```
(Finish)
```

 \downarrow

(Start Measurement)

NOTE

When measuring multiple elements, you cannot set parameters for the elements other than the current measurement one on the "8. Optics Parameters" and "9. Atomizer/Gas Flow Rate Setup" pages. If you use the ASC to measure multiple elements automatically and you need to modify the parameters for other elements than the current measurement one, you can change these parameters by using the [Edit Parameters] button in the "3. Element Selection" page.

3.2.2 Logging in WizAArd

(1) Select [Operation] in the [WizAArd] launcher, and click the AA-7000 main unit icon.



Fig. 3.27 [WizAArd] Launcher

The [WizAArd Login] dialog box will appear at the center of the screen.

(2) Enter "Admin" to the Login ID box and no password to the Password box to log in for the first use.

NOTE

If a login ID and password are already specified, only the authorized user is permitted to use the WizAArd. Therefore, correctly enter the items to log in the WizAArd.



Fig. 3.28 [WizAArd Login] Dialog Box

The [WizAArd Selection] dialog box will appear at the center of the screen.

3.2.3 Wizard Selection

(1) If you are making a new parameter set, select the Element Selection icon on the [Wizard] screen and click on [OK].

NOTE

For the [Recent Files] screen and [Recent Templates] screen, you can open the recent files or templates quickly by selecting from the list.



Fig. 3.29 [Wizard Selection] Dialog Box

The [Element Selection] page will appear.

3.2.4 Element Selection

The [Element Selection] page allows you to select the element to be measured, determine the measuring order, and edit the measurement parameters.

When the element to be measured and measurement method are selected, the standard parameters are displayed automatically.

(1) Click on [Select Elements].

Element Selection							×
	Select elements to be me	asured ar	nd edit the pa	arameters.			
	Element	Wa		At the E	Comment	Select Elements	
						Edit Parameters	
						Up	
						Down	
						Delete	
						Meas. Element:	
						_	
						Connect	
					•		
			1	1		1	_
		< <u>B</u>	ack.	Next >	Finish	Cancel Help	

Fig. 3.30 [Element Selection] Page

The [Load Parameters] will appear.

(2) In this page, select the element first.

NOTE

You can use one of the methods below to select the element.

- · Enter the element symbol directly in the element field from the keyboard.
- Click on the [▼] button at the right of the element field, and select the element from the element symbol list shown in alphabetical order.
- Click on the [Periodic Table] button and select the element from the periodic table.

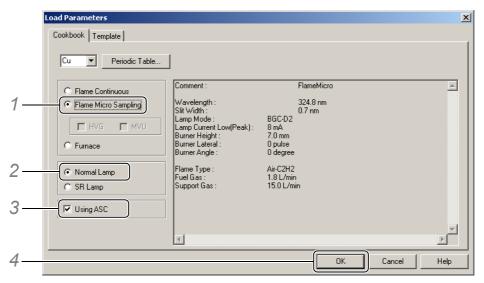


Fig. 3.31 [Cookbook] Page in [Load Parameters]

- 1. Next, select [Flame Micro Sampling] for the measurement method by the radio button.
- 2. Select [Normal Lamp] when using a normal hollow cathode lamp ([SR Lamp] is selected only when the SR method is used as the background correction method).
- 3. When using the autosampler, click on [Using ASC] check box.
- 4. After finishing the settings, click on the [OK] button. If the message on the lamp setup appears, proceed to the section 3.2.4.1 "Lamp Setting Procedure".

WizAArd			×
į	None of the set lamps of Are you going to make		
	Yes	No	

Fig. 3.32 Message Box

(3) To continue measuring multiple elements, temporarily return to the [Element Selection] page, click on [Select Elements], and then select the next element. Repeat the sequence of clicking on the [Select Elements] button, selecting an element and then clicking on the [OK] button, the number of times required.

NOTE

When you return to the [Element Selection] page after completing selecting elements, the selected elements are displayed in the order of selections. If there is any element you want to delete, click on the appropriate row to highlight it and then click on the [Delete] button. The [Meas. Element] field in the lower right part of the screen indicates the element to be measured first.

- (4) If you click on the [Edit Parameters] button, the parameters for the element on the highlighted row on the [Element Selection] page will be displayed. Those parameters may be modified as necessary. First proceed with the operations without using this function.
- (5) Click on [OK].
- (6) If you click on the [Next] button, the [Preparation Parameters] page will be displayed.

NOTE

When analyzing multiple elements sequentially, the order on the [Element Selection] page becomes the measurement order. If you need to change the order, click on the element to highlight it and then click on [Up] or [Down] to move the row. If the [Meas. element] at the right lower of the page is different from the first row element, the measurement is started from the [Meas. Element] and the elements upper than it are not to be measured.

3.2.4.1 Lamp Setting Procedure

If a message about the lamp is displayed at step 4. in (2) above, set the lamp by following the procedure below. (1) Click on the [Yes] button.



Fig. 3.33 Message Box

The [Edit Parameters] page will appear with the message on the lamp setup displayed again. (2) Click on the [OK] button.



Fig. 3.34 Message Box

The [Optics Parameters] sheet of the [Edit Parameters] page is displayed.

(3) Click on the [Lamp Pos. Setup] button.

Edit Parameters							X
Comment ASC Paramete Optics Parameters Seq		v Rate Setup surement Conditio	Weight Correction Factors ns Measurement Par			Miscellanec urve Paramete	
	Wavelength: (185.0 - 900.0 Slit Width (nm): Lamp Mode: Socket #:	324.8 nm) 0.7 BGC-D2 NONE	▼ Lamp Pos. Setup]	amp Current: Low(Peak): (0 - 40 mA) High(Peak):	8	× ×	
				(0 - 600 mA) SC Sample Pos. fr earch:	or EMISS	_	
			Warn	nup Lamp	Line S	earch	
				ОК	Cancel	Help	

Fig. 3.35 [Edit Parameters] Page

The [Lamp Position Setup] dialog box will be displayed.

(4) Enter [Element] (select the element symbol from the drop-down list) and [Lamp Type] (select the normal lamp or SR lamp from the drop-down list) of the lamp that has actually been allocated to each socket number. This allows you to select the lamp registered in [Lamp ID]. If any other element is to be measured, repeat these steps for convenience sake.

eck€t #Ele	ement	Lamp Type	Lamp ID	Judge	Life Time	Used Time	Unit		OK
Cu	ı 🔽	Normal	Cu-1	ок	5000	0.0	mA*hrs	1	Cano
<u> </u>	None								
1	None								<u>Prin</u>
١	None								
1	None]	
1	None							1	
1	None]	

Fig. 3.36 [Lamp Position Setup] Dialog Box

NOTE

When the [Lamp Position Setup] dialog box remains displayed, the lamp turret can be rotated to allow you to mount or replace the lamp.

(5) Select the lamp to be used and then click on the [OK] button. You will return to the previous [Optics Parameters] sheet.

(6) Enter [Socket Number] and click on the [OK] button.

3.2.5 Preparation Parameters

This page allows you to enter Calibration Curve Settings and Sample Group Settings. If you select multiple elements in the [Element Selection] page, multiple rows are displayed in this window.

(1) Click on the row including the desired element for settings. Click on the [Calibration Curve Setup] button to display the [Calibration Curve Setup] page.

For details, see 3.2.5.1 "Calibration Curve Setup".

Click on the [Sample Group Setup] button to display the [Sample Group Setup] page. For details, see 3.2.5.2 "Sample Group Setup".

Fig. 3.37 [Preparation Parameters] Page

Now assume entering [Calibration Curve Setup] and [Sample Group Setup] under the standard parameters.

(2) Set the [Preparation Parameters] and click [Next].

3.2.5.1 Calibration Curve Setup

On this screen you can make the settings relating to the calibration curve. Upon setting the calibration curve and clicking [OK], you will be returned to the [Preparation Parameters] page.

Orde		of Stan	dard Additio	n		Comm	on Settings	of Prepare	ation Para	meters		OK
orue	r 1s	at	Conc.	ppm	-		🗆 Mixing O	N	Mixin	a		Canc
			Unit U	Ibbu			Ĩ					Conc
÷ 20	ero Inte	ercept				Rep	eat Conditio	ns				
	Q	Blank	v/QC Standa	rd Setup)				Reage	ent	II —	
						U						ad
Blank	ı İ	Enned	Parameters	a 1	Diluent	Reagent 1	Reagent 2	Reagent	3 Tota	a		
	Auto	ency	Pos. (ul	_)	R1	Ř2	R3	R4	Volur			
		20	1 10)	0	0	0	0	10			
Resla	Auto	Frequ	Conc.	Pos.	VOL	Diluent P1	Reagent 1	Reagent P3				
	Auto	Frequ ency 20	Conc. 0.0000	Pos. 1	VOL (uL) 10	Diluent R1 0	Reagent 1 R2 0	Reagent R3 0	2 Reager R4 0		ne	
		ency 20		1	(uL) 10	R1	R2 0	R3 0	R4	Volur 10	ne	
Meas		ency 20 nt Seq	0.0000	1	(uL) 10	R1	R2 0 Auto [R3	R4	Volur	ne	0.00
Meas	ureme f Lines	ency 20 nt Seq	0.0000	1	(uL) 10 n Curve	R1 0 Delete Lir	R2 0 Auto I Reme	R3 0 Dilution & asuremer	R4	UNK. Samp	ne	 3 Т
Meas	ureme f Lines	ency 20 nt Seq	0.0000 uence for Ca	1 alibration e Ins True Va 0.500	(uL) 10 10 10 10 10 10 10 10 10 10	R1 0 Delete Lir Pos.	R2 0 Image: Provide state	R3 0 Dilution & easurement iluent F R1 0	R4 0 nt Reagent 1 R2 0	UNK. Samp Upper Limit Reagent 2 R3 0	ne le Conc. Reagent 3 R4 0	Vo
Meas	ureme f Lines	ency 20 nt Seq	0.0000 uence for Ca	1 alibration e Ins True Va	(uL) 10 Curve sert Line alue 10 10 10 10 10 10 10 10 10 10	R1 0 Delete Lir Pos.	R2 0 Auto [Reme VOL D (uL)	R3 0 Dilution & asuremer iluent F R1	R4 0 nt Reagent 1 R2	Volur 10 UNK. Samp Upper Limit Reagent 2 R3	ne le Conc. Reagent : R4	3 T Vo

Fig. 3.38 [Calibration Curve Setup] Page

- Since the calibration curve method is selected here, do not tick the [Method of Standard Addition] option. To use the standard addition method or the simple standard addition method, see the section 4.6 "Standard Addition Method and Simple Standard Addition Method".
- ② [Order] means the order of the calibration curve equation. When the calibration curve is linear, select "1st". If the calibration curve is likely to curve more or less, you may wish to select "2nd" or "3rd". Since this setting may be changed after viewing the actually measured values, select "1st" for now.
- 3 The [Zero Intercept] is used to force the calibration curve to pass through the origin. This setting may be changed later.
- ④ Select [Conc. Unit] of the prepared standard samples. Clicking on [▼] button to select it from the list.
- The explanation proceeds forward without the QAQC setup. Therefore, do not click on the [QC Blank/QC Standard Setup] button. When the QAQC settings are necessary, refer to the Chapter 6 "QA/QC Setup".
- [Common Settings for Preparation Parameters] allows you to enable/disable mixing and specify the mixing, the repeat conditions and the reagents.

• Tick the [Mixing ON] option and then click on the [Mixing] button. The [Mixing Setup] dialog box will be displayed.

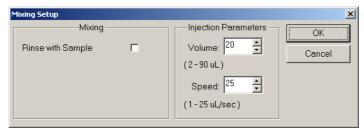


Fig. 3.39 [Mixing Setup] Dialog Box

In the [Mixing Setup] dialog box, set the [Volume] in the [Injection Parameters]. The default value is 20 (μ L), but 50 (μ L) is suitable for the flame micro sampling method. Leave the other settings on the [Mixing Setup] dialog box as they are as the default values.

• Clicking on the [Repeat Conditions] button displays the [Repeat measurement Conditions] dialog box. The number of measurements for the same one sample is set here. For the measurement by the flame micro sampling method, the default value of the number of measurement repetitions is "2" (the allowable maximum is "3"). As the default value is to be selected here, click on the [OK] button to close the dialog box.

		Repea	t Conditions			-
	Num. of Reps.	Max. Num. of Reps.	RSD Limit	SD Limit	Retry	OK Cance
Blank	2	3	7.00	0.00000	Г	
Standard	2	3	7.00	0.00000	Г	
Sample	2	3	7.00	0.00000	Г	
Reslope	2	3	7.00	0.00000	Г	

Fig. 3.40 [Repeat measurement Conditions] Dialog Box

NOTE

The number of repetition refers to the minimum number of measurement repetitions to be used in acquiring data. After completing this set number of measurement repetitions, the average value, relative standard deviation (RSD) and standard deviation (SD) are calculated. Then, the measurement repetitions will continue until (1) the RSD value limit is satisfied, (2) the standard deviation limit is satisfied, or (3) the maximum number of repetition is reached. Setup is possible up to 20 for each item.

• Click on the [Reagent] button. The [Reagent Setup] dialog box will be displayed. This dialog box allows you to enter [Reagent Name] and [Reagent Position] of 4 kinds of reagents.

- In the [Blank Preparation Parameters], set up the automatic periodic blank measurement. The automatic periodic blank measurement is a function to create a measurement procedure on the MRT to eliminate the effect of baseline drift by inserting a blank measurement in a fixed interval. Use this function when there are many samples to be measured or when the baseline drifts.
 - When periodic blank measurement is not performed: Do not tick the [Auto] field.
 - When periodic blank measurement is performed:
 - a. Tick the [Auto] field.
 - b. Enter a value in the [Frequency] field to specify how many samples are measured between blank measurements.
 - c. When using the ASC, the [Pos.] field is displayed if the [Using ASC] is ticked in the [Load Parameters] of [Element Selection] page. Enter the position of the blank sample on the turntable of the ASC.
- ③ Reslope is the sensitivity correction measurement. More specifically, a standard sample of a known concentration is measured during the measurement of unknown samples. Based on the measured absorbance, the slope of the calibration curve is corrected. Subsequently, the corrected new calibration curve will be used to calculate concentrations. If the [Auto] field is ticked in the [Reslope Preparation Parameters], reslope measurement is carried out at the concentration specified in [Conc.] by the specified frequency. The setup procedure is the same as in the [Blank Preparation Parameters].
- In [Measurement Sequence for Calibration Curve], enter the number of standard samples and their concentrations. Enter the number of standard samples in the [No. of Lines] field and click on the [Update] button. A table with that number of rows will be created. In this table, the default values for the concentrations of standard samples are already displayed under the standard parameters, but can be changed with the values for the actually prepared standard samples. If the ASC is used, the [Pos.] field is displayed. Enter the positions of the turntable (1 to 60, R1 to R8).

Mixing ON/OFF

When no samples are mixed using the ASC:

Do not tick [Mixing ON] or disable mixing. When mixing is disabled, the [Total Volume] in [Blank Preparation Parameters], [Reslope Preparation Parameters] and [Measurement Sequence for Calibration Curve] will be the amount injected to the micro sampling port. Enter the amount of the sample injected to the micro sampling port in the [Vol] field. (You cannot directly enter it in [Total Volume].) Leave all the [Diluent], [Reagent 1], [Reagent 2] and [Reagent 3] as zero. Then [Total Volume] will be the same value as [Vol]. When only sample is injected (all of the diluent and reagent parameters are set to zero), the instrument will automatically determine that no mixing is performed, even if the mixing is enabled.

When samples are mixed using the ASC:

Tick Mixing ON to enable mixing. Enter each value in the [Vol], [Diluent], [Reagent 1], [Reagent 2] and [Reagent 3] fields. The total of the values in those fields will be automatically calculated and the result will be included in [Total Volume]. Prepare the concentrate solution of a standard sample, dilute it with the mixing capability of the ASC, and then prepare standard samples of different concentrations for calibration curves. An example of this process is given below.

NOTE

The [Total Volume] must meet the following conditions:

• When mixing is not performed:

(Injection Vol.) = (Total Vol.) </= 90 μL

• When mixing is performed:

(Injection Vol.) = (Max. No. of Reps.) + 50 μ L </= (Total Vol.) </= 600 μ L

(In this regard, the injection volume is the value specified on the [Mixing Setup] dialog box; +50 μ L is the dead volume of the mixing port.)

Example

To prepare each 400 μ L of standard samples of 10, 20, and 30 ppb from the concentrate solution of a standard sample of 100 ppb, assume the concentrate solution of the standard sample as a "sample" and enter each value as follows. The total volume will be calculated automatically.

True Value	Pos	Standard Sample VOL	Diluent	Reagent 1	Reagent 2	Reagent 3	Total Volume
10	1	40	360	0	0	0	400
20	1	80	320	0	0	0	400
30	1	120	280	0	0	0	400

If the injection volume (entered on the [Mixing Setup] dialog box or the [ASC Parameters] page) is assumed as 50 μ L and the maximum number of repetition (entered in the [Repeat Measurement Conditions] dialog box) as 3, the calculation is as follows:

50 μL × 3 + 50 μL </= 400 μL </= 600 μL

This means that the total volume meets the above condition.

Save Calibration Curve Setup Information

The once created [Calibration Curve Setup] information may be stored in the preparation parameter file so that it can be reused to specifying the parameters for other elements. For the file type, specify "*.mix". The preparation parameter file contains the following parameters:

Parameters	Screens
(a) Measurement Type (calibration curve method / standard addition method)	[Calibration Curve Setup] dialog box
(b) Mixing ON/OFF	[Calibration Curve Setup] dialog box
(c) Preparation Parameters (Blank, Reslope, STD)	[Calibration Curve Setup] dialog box
(d) Reagent Name and Reagent Position	[Reagent Setup] dialog box

NOTE

No files in which (a) Measurement Type and (b) Mixing ON/OFF are different from the current settings can be loaded.

3.2.5.2 Sample Group Setup

This window allows you to specify a sample group. If similar kinds of samples that are to be prepared with the same pretreatment are grouped, the effect of interference and the suitability of the pretreatment can be conveniently validated using the QA/AC function.

Upon making the sample group settings and clicking [OK], you will be returned to the [Preparation Parameters] page.

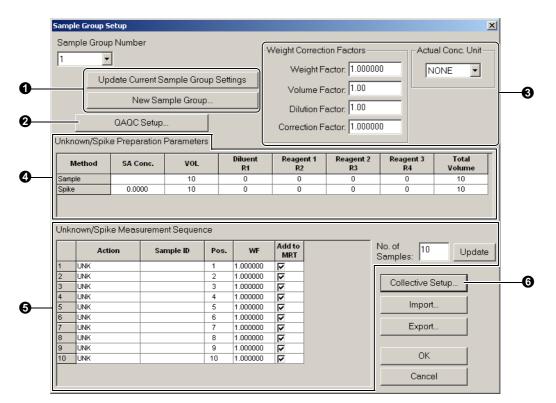


Fig. 3.41 [Sample Group Setup] Page

- In this example, use the default value of "1" for [Sample Group Number] and proceed forward without selecting the [Update Current Sample Group Settings] option and the [New Sample Group] option.
- 2 The [QA/QC Setup] will be described in the Chapter 6 "QA/QC Setup".
- Senter [Weight Correction Factors]. These factors are required to calculate the actual concentrations. Weight Factor [WF], Volume Factor [VF], Dilution Factor [DF], and Correction Factor [CF] are used for the following equation:

Actual concentration = Concentration × [VF] × [DF] × [CF]/[WF]

The actual concentration is calculated with the above equation. The function for automatically converting the units is not available. If conversion of any unit is required, make adjustment using Correction Factor [CF]. (See the example.) If the calculation of the actual concentration is not required, leave all the factors as "1".

To display the unit of the actual concentration, click the $[\mathbf{V}]$ button for [Actual Conc. Unit] and then select the unit from the drop-down list.

Example

Assume that 2 grams of sample is weighed, made up to 50 mL with a solution, and then diluted by a factor of 5 for measurement. To obtain the actual concentration from measured concentration, enter 2 (g) for Weight Factor [WF], 50 (mL) for Volume Factor [VF], 5 for Dilution Factor [DF], and 1 for Correction Factor [CF] as follows:

Actual concentration = Concentration × 50 (mL) × 5 (times) × 1/2 (grams)

In the same example, to obtain the actual concentration (%) from the concentration (ppm) by converting the unit, use 0.0001 for Correction Factor [CF] since 1 ppm is equal to 0.0001%, as follows: Actual concentration = Concentration \times 50 (mL) \times 5 (times) \times 0.0001/2 (grams)

Remarks

The unit of ppm indicates a concentration using the unit of 10-6. In the atomic absorption analysis, both of μ g/g for solid samples and μ g/mL (mg/L) for liquid samples are, in practice, expressed in ppm.

In the [Unknown/Spike Measurement Sequence], enter the number of unknown samples and sample ID's. Enter the number of unknown samples in the [No. of Samples] field and click on the [Update] button. A table with that number of rows will be created. Sample ID can be entered one by one in the table, but can be entered at a time by clicking on the [Collective Setup] button. If the ASC is used, enter each turntable position (1 to 60) in the [Pos.] field.

NOTE

In the above ③, [Weight Correction Factors] has been entered. In general, [WF] varies depending upon each sample and can be entered in the [Unknown /Spike Measurement Sequence] table. Only the sample for which the [Add to MRT] field is ticked is inserted into the MRT worksheet on the main screen. The created [Unknown/Spike Measurement Sequence] table can be saved or loaded.

If you click on the [Collective Setup] button on the [Sample Group Setup] page of Fig. 3.41, the [Sample ID Collective Setup] dialog box will be displayed.

The [Unknown/Spike Preparation Parameters] allows you to enter the preparation parameters for unknown samples and spike samples. Spiking is one of the QA/QC techniques that are used to obtain the recovery rate by adding a solution of a known concentration to an unknown sample. In this example, proceed forward without entering a value (i.e., using the [S A Conc.] of 0.0000).

Sample ID Collective Setup

Clicking [Collective Setup...] on the [Sample Group Setup] page displays the [Sample ID Collective Setup] dialog box.

	Sample ID Collective Setup	×
0	- Number of Samples: 🔟 🕂	ОК
•		Cancel
0	Create Sample ID	
0	Sample ID- Name Start No. Sample + 1 -	ASC Start Pos.
0—	Pre-Digestion Spike (SPK) 20 Post-Digestion Spike (PDS) 20 Duplicate (DUP)	

Fig. 3.42 [Sample ID Collective Setup] Dialog Box

- 1 In the [Sample ID Collective Setup] dialog box, enter the number of unknown samples in the [Number of Samples] field.
- 2 To enter sample ID (sample name), tick the [Create Sample ID] field.
- When you enter a name and starting number in the enabled [Sample ID] field, the same name will be given to all the samples with sequential numbers from the starting number given to them. If the ASC is used, specify the position of the 1st unknown sample in the [ASC Start Pos.] field. The 2nd and subsequent positions will be automatically entered in the table.
- If [Pre-Digestion Spike (SPK)], [Post-Digestion Spike (PDS)], and [Duplicate (DUP)] are ticked, these measurements will be inserted in the analysis sequence for samples each in the number indicated on the right side field. Since these samples are used for QA/QC, proceed forward without ticking the above options in this example.

3.2.6 Connect to Instrument/Send Parameters

This section describes how to connect to the instrument and send the parameters. When the connection to the instrument is made, the instrument is initialized automatically.

(1) Check that the AA main unit and the related units are ON and click on the [Next] button.

Connect to Instrument/Sen	d Parameters	X
	Power ON the instrument, and click on the [Connect/Send Parameters] button.	
	If the options are not recognized properly because the ASC/GFA are not powered on before initialization, click on the [Connect Options] button.	
	Meas. Element:	
		_
	<u>ABack</u> <u>Next</u> Finish Cancel Help	

Fig. 3.43 [Connect to Instrument/Send Parameters] Page

(2) Check that the chimney is correctly installed on the instrument and then click [Yes].

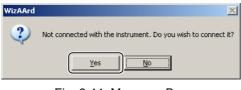


Fig. 3.44 Message Box

NOTE

If the chimney is not correctly installed on the instrument, the [Flame Monitor Check] may not be performed correctly at initialization, leading to an [NG] result.

The connection to the instrument will be started with the [Initialize] screen displayed and then the AA main unit will be initialized. After the initialization has been finished, the parameters for the element specified in [Meas. Element] are automatically sent to set up the instrument.

Alternatively, you may want to press the [Connect/Send Parameters] button in the [Connect to Instrument/ Send Parameters] page to perform the same operation.

3.2.6.1 Initializing the Instrument

The results of initialization of the instrument are displayed on the [Initialize] screen. You can also check the versions of the AA-7000 main unit and the ASC and GFA ROMs on this screen.

CAUTION

• Expiration dates apply to inspections of safety devices.

Once an expiration date has passed the instrument can no longer be used. Even if the safety devices are within the expiration date for inspections, you are strongly recommended to implement an inspection at initialization.

• If the result of the inspection is that there is a fault in a safety device, a message describing the fault is displayed and the device cannot be used.

If a safety device is faulty, contact your Shimadzu representative.

Initialize		
	1 A3000000000 .01 A3000000000	-0
GFA: GFA-7000 ∨1	.02 A3000000000	
ROM Check	C2H2 Valve Origin Search	•
S/N Check	Flame Monitor Check	-0
ASC Check		
GFA Check	Burner Select Sensor Check	
Slit Origin Search	Drain Sensor Check	
D2 Attenuator Origin	Support Gas Pressure Monitor Check(Air)	63
🔵 Wavelength Origin Search	Support Gas Pressure Monitor Check(N2O)	
🔵 Turret Origin Search	Fuel Gas Pressure Monitor Check	
Atomizer Up/Down	Start Leak Check	
Atomizer Fore/Back		
Testing 🔵 Succ	ess 🔶 Failure 🔿 No Test(Not Connected)	
	ОК	

Fig. 3.45 [Initialize] Screen (for AA-7000F/AAC)

No.	Name	Function
0	Instrument information	The models of the AA main unit, ASC and GFA, the ROM versions and the machine identification numbers are displayed here. If neither ASC nor GFA is connected, the ASC and GFA information is not displayed.
0	Automatic inspection points	These are inspections that the instrument performs automatically. If neither ASC nor GFA is connected, [Not Connected] () is shown for the [ASC Check] and [GFA Check] points.
0	Manual inspection points	Messages are displayed to prompt the successive performance of each of these inspection points. Carry out the inspections in accordance with the messages.
4	Start Leak Check	Starts an 8-minute automatic gas leakage inspection.

NOTE

The time required from the start of initialization to completion of the automatic inspection points is 4 to 5 minutes (it varies depending on whether or not options are installed).

(1) When initialization of the instrument starts, the instrument information is acquired and the automatic inspection points are implemented. On completion of the automatic inspection points, the [Gas Adjustment] screen is displayed. After setting the fuel gas and support gas, click [Close].

	Gas Adjustment						
	Turn on the ventilation system. In case of flame analysis, open the main valves of fuel gas and support gas.						
	1.Set the C2H2 supply pressure to 0.09MPa with purging C2H2.						
0—	Purge C2H2 (5sec , till 5 times)						
	2.Set the Air supply pressure to 0.35MPa with purging Air. In case optional flowmeter is installed, set the Air flow rate to 15.0L/min.						
0	Purge Air (10sec)						
	3.In case of using N2O-C2H2 flame, set the N2O supply pressure to 0.35MPa with purging N2O.						
0	Purge N20 (10sec)						
	Close						

Fig. 3.46	[Gas Adjustment] Screen
-----------	-------------------------

No.	Name	Function
0	[Purge C2H2]	Performs the acetylene (C_2H_2) gas purge operation (5 seconds per purge, with a limit of 5 purges). During the purge operation, set the acetylene supply pressure to 0.09 MPa.
0	[Purge Air]	Performs the air purge operation (10 seconds per purge, with no limit on the number of purges). During the purge operation, set the air supply pressure to 0.35 MPa.
8	[Purge N2O]	Performs the nitrous oxide (N_2O) gas purge operation (10 seconds per purge, with no limit on the number of purges), when the high-temperature burner head (optional) is used. During the purge operation, set the nitrous oxide gas supply pressure to 0.35 MPa.

(2) Carry out the burner identification sensor inspection, drain sensor inspection, and support gas monitor inspection (air).

Clicking [Yes] starts the burner identification sensor inspection.

WizAArd
You can check the safety devices now. Burner Select Sensor Drain Sensor Do you wish to check them? * Expires 26 days later * Igniting a flame without safety device check is unable when the check-free period expired or the previouse check(s) was(were) failed.
Fig. 3.47 Message Box
NOTE
 On clicking [No] when still within the expiration date, the following message is displayed without implementing an inspection. You are strongly recommended to perform inspections within the expiration date.
WizAArd X You can check the safety devices now. Burner Select Sensor Drain Sensor Support Gas Pressure Monitor(Air) Do you wish to check them? * Expires 26 days later * Igniting a flame without safety device check is unable when the check-free period expired or the previouse check(s) was(were) failed.
 On clicking [No] when the expiration date has passed, the following message is displayed. If using the flame method, click [Recheck].
Initialize
The check was not executed. The flame can't be ignited, because it's beyond the safety device check-free period or the previouse check(s) was(were) failed.
OK: The initialization process is continued though the flame can't be ignited.
Recheck : The initialization process gets back to check process to enable the instrument to ignite the flame.
 If the following message is displayed after clicking [Yes], set the BURNER SELECT switch to the AIR- C₂H₂ position.
WizAArd
Switch Burner Select Sensor to Air.
AIR-C ₂ H ₂
BURNER SELECT

(3) The drain sensor inspection starts. When the following message is displayed, remove the front panel.
 For details, see 2.4.1 "Removing the Front Panel".
 Take the sensor out of the drain tank, and lift it above the surface of the water.

WizAArd	X
1	Lift the Drain Sensor over the surface of water.
	OK

(4) Click [OK] in the [Lift the Drain Sensor over the surface of water.] dialog box.



(5) When the following message is displayed, mount the drain sensor in its original position. Mount the front panel and click [OK]. For details, see 2.4.2 "Mounting the Front Panel".

WizAArd	×
♪	Move the Drain Sensor below the surface of the water.
	ОК

(6) When the support gas pressure monitor inspection (Air) starts, the following message is displayed. Supply air and click [OK].



(7) When the support gas pressure monitor inspection (N₂O) starts, the following message is displayed.

You can check the safety device now. Support Gas Pressure Monitor(N2O) Do you wish to check it? * Expires 26 days later * Igniting N2O-C2H2 flame without safety device check is unable when the check-free period expired or the previouse check was failed.	WizAArd	×
<u>Yes</u> <u>N</u> o	1	Support Gas Pressure Monitor(N2O) Do you wish to check it? * Expires 26 days later * Igniting N2O-C2H2 flame without safety device check is unable when the check-free period expired or the previouse check was failed. Yes No

(8) Click [Yes]. The message shown below will be displayed. Supply N₂O and click [OK].



NOTE
 If the high-temperature burner head (optional) is not being used, click [No].
On clicking [No], the following message is displayed without implementing an inspection. When the high-temperature burner head (optional) is installed and the $N_2O - C_2H_2$ flame is used, you are strongly
recommended to perform inspections within the expiration date.
WizAArd
The safety device check is not performed. Igniting N2O-C2H2 flame is available during check-free period. But, it is strongly recommended to check it even during the period.
()
 Even when the expiration date for the support gas pressure monitor inspection (N₂O) has passed, provided the instrument is still within the expiration date for the other inspection points, the Air- C₂H₂ flame can be used.

(9) When the fuel gas pressure monitor inspection starts, the following message is displayed.

Fuel gas pressure monitor check	
The fuel gas pressure monitor is checke	ed here.
If this check is not executed, the flame ca	annot be ignited.
Check it	Don't check it

(10) Click [Check it]. The following dialog box will be displayed. Supply C_2H_2 gas and click [OK].



NOTE							
 If you click [Don't check it], the following message is displayed. 							
	Initialize						
	The check was The flame can'						
	OK :	The initialization process is continued though the flame can't be ignited.					
	Recheck:	The initialization process gets back to check process to enable the instrument to ignite the flame.					
		<u>QK</u> <u>Becheck</u>					

(11) On completion of the fuel gas pressure monitor inspection, a 8-minute automatic gas leakage inspection starts, and initialization is completed. On completion of initialization, click [OK]. The [Initialize] screen will close.

NOTE

The flame cannot be ignited during a gas leakage inspection.

3.2.6.2 Instrument Check List for Flame Analysis

- (1) After closing the [Initialize] screen, a message is displayed asking whether the flame measurement is performed. Click on the [Yes] button here. Perform the checks prior to the flame measurement in accordance with the [Instrument Check List for Flame Analysis].
- (2) Check all the items (1) through (8) and the emergency stop operation, and then tick them. See also the introductory section "Emergency Action" and the section 4.5 "Igniting and Extinguishing the Flame".

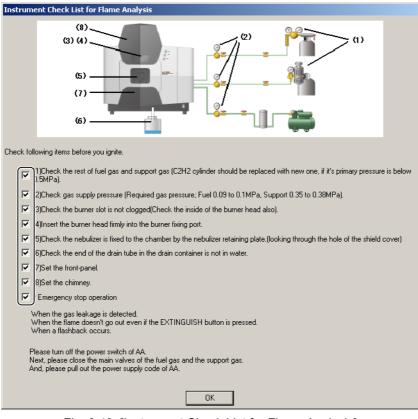


Fig. 3.48 [Instrument Check List for Flame Analysis]

(3) When all the items are checked, you can select [OK].

CAUTION

The checklist for starting the flame measurement assumes that the user checks the safety. When using flame, properly carry out the checks.

For details, see Chapter 8 "Maintenance".

3.2.7 **Optics Parameters**

The [Optics Parameters] page is used to set the parameters for the monochrometer and the lamps in the instrument. For the purpose of this example, proceed forward without changing the standard parameters (that were read out when the elements were selected). For changing each parameter, refer to the HELP information in the WizAArd software.

Optics Parameters					×
	📮 Cu				
	Wavelength: (185.0 - 900.0 i	324.8 1m)	Lamp Current:		
1.1.	Slit Width (nm):	0.7 💌	Low(Peak):	8 🔺	
	Lamp Mode:	BGC-D2	(0 - 40 mA)		
	Socket #:	1 Lamp Pos. Setup	High(Peak):		
		e Lamp Pos. Setup button, you can ret manually and change the lamp.	(0 - 600 mA)		
	Lamp ID:	Cu-1	ASC Sample Pos.	for EMISSION Line	
	Lamp ON:		Search:	hour -	
	Lamp Status:	Line Search is necessary.		NONE	
		_	Warmup Lamp	Line Search	
		< <u>B</u> ack <u>N</u> ext >	Finish	Cancel Help	

Fig. 3.49 [Optics Parameters] Page

NOTE

This page displays the wavelength, slit width, socket number, lamp current, lamp mode and so on. These parameters are set for only the element that will be firstly measured (has been specified in the [Meas. Element] field located in the lower right part of the [Element Select] page or the [Connect to Instrument/Send Parameters] page).

The measurement parameters for each element are loaded from the cookbook and automatically specified when the elements are selected.

Normally, you do not need to enter these measurement parameters. To modify them, however, you can enter a value for the wavelength and select a value for other conditions from the list pulled down by clicking on the $[\mathbf{V}]$ button. The lamp current value can be changed in units of 1 mA by clicking on the $[\mathbf{A}]$ or $[\mathbf{V}]$ buttons.

(1) Click on the [Next] button. A message will be displayed prompting you to do line search. Click on the [OK] button.

The [Line Search/Beam Balance] dialog box will be displayed and the process will be carried out automatically.

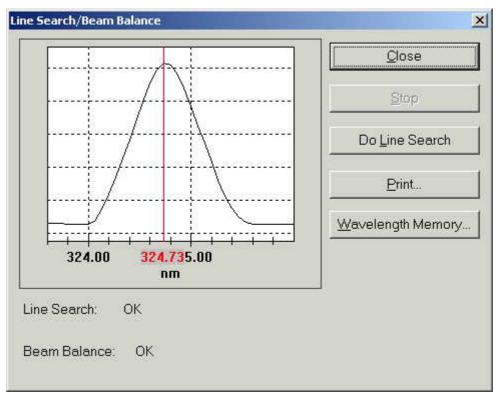


Fig. 3.50 [Line Search/Beam Balance] Dialog Box

- First the line search (wavelength matching) is carried out and then the beam balance (gain control for the detector) is performed. If only the beam balance is necessary, the line search is not performed.
- In the line search, the highest peak near the specified wavelength is detected. In some cases, however, the neon gas contained in the hollow cathode lamp radiates more intense light than the light from the element. In such a case, if the correctly analyzed line is stored by clicking on the [Wavelength Memory] button, its wavelength will be used for the subsequent line searches.
- (2) Upon completion of the process, click on the [Close] button to proceed forward to the [Atomizer/Gas Flow Rate Setup] page.

NOTE

When measuring multiple elements, you cannot set parameters for the elements other than the current measurement one on the [Optics Parameters] and [Atomizer/Gas Flow Rate Setup] pages. If you use the ASC to measure multiple elements automatically and you need to modify the parameters for other elements than the current measurement one, you can change these parameters by using the [Edit Parameters] button in the [Element Selection] page.

NOTE

SR lamp intensity is hard to stabilize in using NON-BGC mode or BGC-D2 mode in comparison with a normal lamp. Before performing the line search, wait for 15 to 20 minutes after turning on the lamp, and then start measurement.

3-50

3.2.8 Atomizer/Gas Flow Rate Setup

This page is used to specify the burner position and the flow rate of fuel gas and support gas. Select [Atomizer Position] or [Gas Flow Rate] using the radio button. The window display will be changed accordingly. Whichever parameter may be selected first.

(1) To set these parameters, ignite flame and allow a standard sample to be sucked.

Even if the ASC is used for the actual measurement, use this window to manually let the sample be sucked. Set the parameters so that the absorbance of the standard sample with a known concentration falls within the intended range of absorbance (normally the maximum absorbance is obtained).

Atomizer/Gas Flow Rate Setup		X
🖳 Cu		
Operation Object Gas Flow Rate Atomizer Position	0.700	
Measured Data: 0.0000 Background Data: 0.0001	0.600	
Autozero(F3) Flame Type: Air-C2H2	0.500	
Fuel Gas Flow Rate(L/min): [1.8	0.400	
Support Gas Flow Rate(L/min): 13.5 to 17.5	A b 0.300 s	
*When igniting a flame is not easy, please increase the fuel gas flow rate once and try to ignite. Please get the flow rate back to the value for the analysis after igniting.	0.200	
* In the case of N20-C2H2 flame, please make sure to set the gas flow rates so that the red feather of the flame gets more than 2 to 3 mm.	0.100	
Increase Fuel(F5) Flame Setup(F7)		
Decrease Fuel(F6) Gas Flow Auto(F8)	-0.100	
	-0.200 † : : : : : :	
	< Back Mext> Finish Cancel Help	

Fig. 3.51 [Atomizer/Gas Flow Rate Setup] Page

When the [Atomizer Position] is selected:

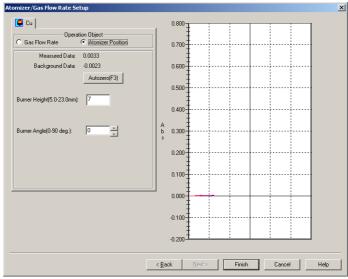


Fig. 3.52 [Atomizer Position] is Selected

- Leave the [Burner Angle] as "0" for usual use. Change it only when you want to decrease the sensitivity by changing the burner angle for high-concentration samples.
- For the [Burner Height], an appropriate value is indicated according to the element, so you need not change it for usual use. However, the optimum condition for the burner height may differ depending on the gas flow rate or the sample type. To change the value, enter a new value.

Operat	ion Object			-			1	
	 Atomizer Position 		0.700-		 			
Measured Data:	0.0002	1			1		1	
Background Data:	-0.0003		0.600-		 	+		
	Autozero(F3)							
Burner Height(5.0-23.0mm):	7		0.500-		 -			
Burner Lateral Pos.:			0.400-		 			
(-119 to 119 pulses)	0		-					
Burner Angle(0-90 deg.):	0	A b s	0.300-		 +	+		
			0.200-					
			0.200-					
			0.100-		 			
			-					
Up(F5)	Set Position(F7)		0.000-					
Down(F6)	Burner Position Auto(F8)		-0.100		 			
Forward	Backward		-0.200-					

Fig. 3.53 [Atomizer Position] is Selected (with Auto Atomizer Changer)

- When the instrument is equipped with Auto Atomizer Changer (optional), clicking on the [Up] and [Down] buttons at the bottom of the page allows you to vertically change the burner position. As the burner position is vertically changed, the value in the [Burner Height] field is also changed accordingly.
- The [Burner Position Auto] button is used to obtain the optimum condition for the burner height through the measurement of the actual sample. For the steps to perform [Burner Position Auto], see the section 4.8.1 "Setting the Optimum Condition of Burner Height".

When the [Gas Flow Rate] is selected

- A type of flame, and flow rates of fuel gas and support gas can be specified.
- In the [Flame Type] field, a suitable flame type is indicated according to the element. To change
 the flame type, click on the [▼] button and select a desired flame type from the drop-down list.
 N₂O-C₂H₂ cannot be used with the standard burner head. It requires using the hightemperature burner head (optional).
- The [Increase Fuel] and [Decrease Fuel] buttons located at the lower of the page allow you to increase and decrease the flow rate of fuel gas. The current value is displayed in the [Fuel Gas Flow Rate] field.
- The [Support Gas Flow Rate] is fixed (at 15 L/min). It cannot be increased or decreased.

NOTE

If the flow meter kit (optional) has been attached, the flow rate of the support gas can be increased and decreased by using the flow rate adjusting control, and a setting can be recorded for [Support Gas Flow Rate].

- The [Gas Flow Auto] button is used to obtain the optimum condition for the flow rate of fuel gas through the measurement of the actual sample. For the steps to perform [Gas Flow Auto] see the section 4.8.2 "Setting the Optimum Condition of Fuel Gas Flow Rate".
- (2) After all the settings have been finished, click on the [Finish] button.

The wizard will be exited with the main screen displayed. To save the conditions specified here as a template, proceed to the section 3.4 "Saving the Template"; to start the measurement, proceed to the section Chapter 4 "Measurement Procedures".

3.3

3.3.1 Operation Flowchart (Furnace Method)

This Wizard has a sequence for setting as shown below. You can proceed to the next step or return to the preceding step by using the [Next] or [Back] button, respectively.

(Starting up the AA Software)

- \downarrow
 - 1. WizAArd Login
 - 2. Wizard Selection
 - 3. Select elements to be measured and edit the parameters.
 - 4. Edit sample preparation parameters and QA/QC settings.
 - 5. Connect to Instrument/Send Parameters
 - 6. Optics Parameters
 - 7. Furnace Program

 \downarrow

(Finish)

 \downarrow

```
(Start Measurement)
```

NOTE

When measuring multiple elements, you cannot set parameters for the elements other than the current measurement one on the "8. Optics Parameters" and "9. Furnace Program" pages. If you use the ASC to measure multiple elements automatically and you need to modify the parameters for other elements than the current measurement one, you can change these parameters by using the [Edit Parameters] button in the "3. Element Selection" page.

3.3.2 Logging in WizAArd

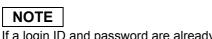
(1) Select [Operation] in the [WizAArd] launcher, and click the AA-7000 main unit icon.



Fig. 3.54 [WizAArd] Launcher

The [WizAArd Login] dialog box will appear at the center of the screen.

(2) Enter "Admin" to the Login ID box and no password to the Password box to log in for the first use.



If a login ID and password are already specified, only the authorized user is permitted to use the WizAArd. Therefore, correctly enter the items to log in the WizAArd.

WizAArd Login		
	WizAArd	
<u>L</u> ogin ID :	admin	ОК
Password :		Cancel

Fig. 3.55 [WizAArd Login] Dialog Box

The [WizAArd Selection] dialog box will appear at the center of the screen.

3.3.3 Wizard Selection

(1) If you are making a new parameter set, select the Element Selection icon on the [Wizard] screen and click on [OK].

NOTE

For the [Recent Files] screen and [Recent Templates] screen, you can open the recent files or templates quickly by selecting from the list.

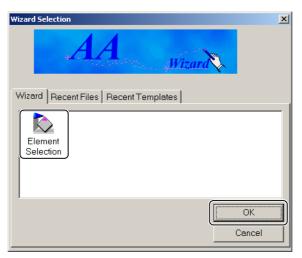


Fig. 3.56 [Wizard Selection] Dialog Box

The [Element Selection] page will appear.

3.3.4 Element Selection

The [Element Selection] page allows you to select the element to be measured, determine the measuring order, and edit the measurement parameters.

When the element to be measured and either the flame or furnace method for measurement are selected, the standard parameters are displayed automatically.

(1) Click on the [Select Elements].

Element Selection							×
	Select elements to be	measured an	d edit the pa	arameters.			
	Element	Wa	Socket #	At the E	Comment	Select Elements	
						Edit Parameters	
						Up	
						Down	
						Delete	
						Meas. Element:	
							
						,	
						Connect	
							_
		< <u>B</u> a	ick.	<u>N</u> ext >	Finish	Cancel Help	

Fig. 3.57 [Element Selection] Page

The [Load Parameters] will appear.

(2) In this page, select the element first.

NOTE

You can use one of the methods below to select the element.

- Enter the element symbol directly in the element field from the keyboard.
- Click on the [▼] button at the right of the element field, and select the element from the element symbol list shown in alphabetical order.
- Click on the [Periodic Table] button and select the element from the periodic table.

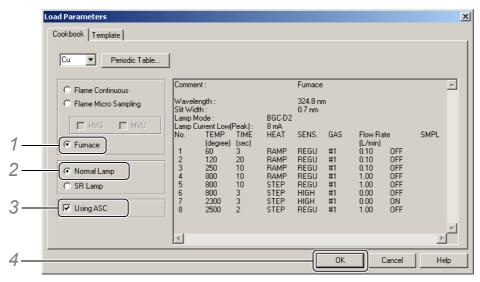


Fig. 3.58 [Cookbook] Page in [Load Parameters]

- 1. Next, select [Furnace] for the measurement method by the radio button.
- 2. Select [Normal Lamp] when using a normal hollow cathode lamp ([SR Lamp] is selected only when the SR method is used as the background correction method).
- 3. When using the autosampler, click on [Using ASC] check box.
- 4. After finishing the settings, click on the [OK] button. If the message on the lamp setup appears, proceed to the section 3.3.4.1 "Lamp Setting Procedure".

WizAArd	X
(į)	None of the set lamps can be used for this setting. Are you going to make setting immediately?
	<u>Yes</u> <u>N</u> o

Fig. 3.59 Message Box

(3) To continue measuring multiple elements, temporarily return to the [Element Selection] page, click on [Select Elements], and then select the next element. Repeat the sequence of clicking on the [Select Elements] button, selecting an element and then clicking on the [OK] button, the number of times required.

NOTE

When you return to the [Element Selection] page after completing selecting elements, the selected elements are displayed in the order of selections. If there is any element you want to delete, click on the appropriate row to highlight it and then click on the [Delete] button. The [Meas. Element] field in the lower right part of the screen indicates the element to be measured first.

- (4) If you click on the [Edit Parameters] button, the parameters for the element on the highlighted row on the [Element Selection] page will be displayed. Those parameters may be modified as necessary. First proceed with the operations without using this function.
- (5) Click on [OK].
- (6) If you click on the [Next] button, the [Preparation Parameters] page will be displayed.

NOTE

When analyzing multiple elements sequentially, the order on the [Element Selection] page becomes the measurement order. If you need to change the order, click on the element to highlight it and then click on [Up] or [Down] to move the row. If the [Meas. element] at the right lower of the page is different from the first row element, the measurement is started from the [Meas. Element] and the elements upper than it are not to be measured.

3.3.4.1 Lamp Setting Procedure

If a message about the lamp is displayed at step 4. in (2) above, set the lamp by following the procedure below.

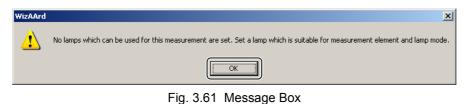
(1) Click on the [Yes] button.



Fig. 3.60 Message Box

The [Edit Parameters] page will appear with the message on the lamp setup displayed again.

(2) Click on the [OK] button.



The [Optics Parameters] sheet of the [Edit Parameters] page is displayed.

(3) Click on the [Lamp Pos. Setup] button.

Edit Parameters		×
Comment ASC Parameters Optics Parameters Sequence	Furnace Program Weight Correction Factors Repeat Measurement Conditions Measurement	Y-axis Print Range Miscellaneous Parameters Calibration Curve Parameters
	Cu Wavelength: 324.8 (185.0 - 900.0 nm) Silt Width (nm): 0.7 Lamp Mode: BGC-D2 Socket #: NONE Lamp Pos. Setup If you click on the Lamp Pos. Setup button, you can turn the lamp turret manually and change the lamp.	Lamp Current: Low(Peak): 8 * (0 - 40 mA) High(Peak): 0 * (0 - 600 mA.)
	Lamp ID: Lamp ON: Lamp Status:	ASC Sample Pos. for EMISSION Line Search:
		Varmup Lamp Line Search

Fig. 3.62 [Edit Parameters] Page

The [Lamp Position Setup] dialog box will be displayed.

(4) Enter [Element] (select the element symbol from the drop-down list) and [Lamp Type] (select the normal lamp or SR lamp from the drop-down list) of the lamp that has actually been allocated to each socket number. This allows you to select the lamp registered in [Lamp ID]. If any other element is to be measured, repeat these steps for convenience sake.

iock∉t #	Element	Lamp Type	Lamp ID	Judge	Life Time	Used Time	Unit	ОК
	Cu 🔽	Normal	Cu-1	ок	5000	0.0	mA*hrs	Cance
2	NUTE							
}	None							<u>P</u> rint
	None							
5	None							
5	None							

Fig. 3.63 [Lamp Position Setup] Dialog Box

NOTE

When the [Lamp Position Setup] dialog box remains displayed, the lamp turret can be rotated to allow you to mount or replace the lamp.

- (5) Select the lamp to be used and then click on the [OK] button. You will return to the previous [Optics Parameters] sheet.
- (6) Enter [Socket Number] and click on the [OK] button.

3.3.5 Preparation Parameters

This page allows you to enter Calibration Curve Settings and Sample Group Settings. If you select multiple elements in the [Element Selection] page, multiple rows are displayed in this window.

(1) Click on the row including the desired element for settings. Click on the [Calibration Curve Setup] button to display the [Calibration Curve Setup] page.

For details, see 3.3.5.1 "Calibration Curve Setup". Click on the [Sample Group Setup] button to display the [Sample Group Setup] page.

For details, see 3.3.5.2 "Sample Group Setup".

Preparation Parameters	×
Preparation Parameters Edit the calibration parameters, sample preparation parameters and QA/QC settings. Ele Method Or Ze Co Cu Calibration Curve Setup Sample Group Setup Sample Group Setup	X
< <u>₿</u> ack <u>N</u> ext > Finish Cancel Help	

Fig. 3.64 [Preparation Parameters] Page

Now assume entering [Calibration Curve Setup] and [Sample Group Setup] under the standard parameters.

(2) Set the [Preparation Parameters] and click [Next].

3.3.5.1 Calibration Curve Setup

On this screen you can make the settings relating to the calibration curve. Upon setting the calibration curve and clicking [OK], you will be returned to the [Preparation Parameters] page.

-Orde		of Stan	dard Additio	'n		Comm	ion Settings	of Prepa	ration Para	meters —		OK
	r [1s	t	Conc.	NO	NE 🔻		🗆 Mixing (DN	Mixin	g		Canc
	, ero Inte	ercept	- Unit			Bon	eat Conditio	- 	Coatir			
		p.					earconulu		Cuaur	ig		
	QC	Blank	v/QC Standa	rd Setu	р				Reage	ent		ad
Blank	Prena	ration	Parameters			<u> </u>						au
		Freque		L	Diluent R1	Reagent 1 R2	Reagent 2 R3	Reager R4	nt 3 Tota Volur			
		20	1 1	5	0	0	0	0	10			
		Frequ	on Paramet Conc.	ers Pos.	VOL	Diluent R1	Reagent 1					
	I	Erecul		1	VOL (uL) 10	Diluent R1 0	Reagent 1 R2 0	Reager R3 0	nt 2 Reage R4 0		me	
Meas	Auto	Freque ency 20	Conc. 0.0000	Pos.	(uL) 10 n Curve	R1 0	R2 0	Dilution 8	R4 0	UNK. Samp	ne	0.00
Meas	Auto	Freque ency 20	Conc. 0.0000	Pos.	(uL) 10	R1	R2 0	R3 0	R4 0	Volu 10	ne	0.00
Meas	Auto	Freque ency 20	Conc. 0.0000	Pos. 1 alibratio	(uL) 10 n Curve sert Line	R1 0	R2 0 Auto Rem	Dilution 8	R4 0	UNK. Samp	ne	ј 3 Т.
Meas	Auto	Frequeency 20	Conc. 0.0000 uence for Ca	Pos. 1 alibratio	(uL) 10 n Curve sert Line /alue	R1 0 Delete Li Pos.	R2 0 ne Auto Nem Rem VOL 1 10 10	R3 0 Dilution & easurem Diluent R1 0	R4 0 ent Reagent 1 R2 0	Volu 10 UNK. Samp Upper Limit Reagent 2 R3 0	ne ble Conc. t: Reagent R4 0	0.00 3 To Vol
Meas	Auto	Frequeency 20	Conc. 0.0000 uence for Ca	Pos. 1 IIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIII	(uL) 10 n Curve sert Line /alue 100 100	R1 0 Delete Li Pos.	R2 0 Auto Rem VOL 1 (uL)	R3 0 Dilution & easurem Diluent R1	R4 0 ent Reagent 1 R2	Volu 10 UNK. Samp Upper Limit Reagent 2 R3	ne ble Conc. t Reagent R4	3 T Voi

Fig. 3.65 [Calibration Curve Setup] Page

- Since the calibration curve method is selected here, do not tick the [Method of Standard Addition] option. To use the standard addition method or the simple standard addition method, see the section 4.6 "Standard Addition Method and Simple Standard Addition Method".
- ② [Order] means the order of the calibration curve equation. When the calibration curve is linear, select "1st". If the calibration curve is likely to curve more or less, you may wish to select "2nd" or "3rd". Since this setting may be changed after viewing the actually measured values, select "1st" for now.
- 3 The [Zero Intercept] is used to force the calibration curve to pass through the origin. This setting may be changed later.

Select [Conc. Unit] of the prepared standard samples. Clicking on [▼] button to select it from the list.

The explanation proceeds forward without the QAQC setup. Therefore, do not click on the [QC Blank/QC Standard Setup] button. When the QAQC settings are necessary, refer to the Chapter 6 "QA/QC Setup".

[Common Settings for Preparation Parameters] allows you to enable/disable mixing and specify the mixing, the repeat conditions, the coating/boost cycle and the reagents.

• Tick the [Mixing ON] option and then click on the [Mixing] button. The [Mixing Setup] dialog box will be displayed.

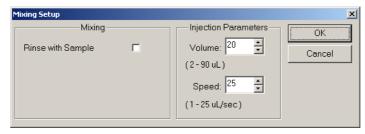


Fig. 3.66 [Mixing Setup] Dialog Box

In the [Mixing Setup] dialog box, set the [Volume] in the [Injection Parameters]. The default value is 20 (μ L). Leave the other settings on the [Mixing Setup] dialog box as they are as the default values.

Clicking on the [Repeat Conditions] button displays the [Repeat measurement Conditions] dialog box. The number of measurements for the same one sample is set here. For the measurement by the furnace method, the default value of the number of measurement repetitions is "2" (the allowable maximum is "3"). As the default value is to be selected here, click on the [OK] button to close the dialog box.

	Repeat Conditions										
	Num. of Reps.	Max. Num. of Reps.	RSD Limit	SD Limit	Retry	OK Cance					
Blank	2	3	7.00	0.00000	Г						
Standard	2	3	7.00	0.00000	Г						
Sample	2	3	7.00	0.00000	П						
Reslope	2	3	7.00	0.00000	Г						

Fig. 3.67 [Repeat Measurement Conditions] Dialog Box

NOTE

The number of repetition refers to the minimum number of measurement repetitions to be used in acquiring data. After completing this set number of measurement repetitions, the average value, relative standard deviation (RSD) and standard deviation (SD) are calculated. Then, the measurement repetitions will continue until (1) the RSD value limit is satisfied, (2) the standard deviation limit is satisfied, or (3) the maximum number of repetition is reached. Setup is possible up to 20 for each item.

- · Click on the [Coating] button. The [Coating and Boost Cycles] dialog box will be displayed.
 - Coating refers to injecting a specified reagent into the furnace and drying it prior to the sample injection (i.e., coating the tube surface). First, the checked mark reagent is injected in the furnace, and heating is executed up to the furnace program stage specified in [Last Coating Cycle]. After this, the sample is injected.

Furnace boost cycle refers to the repetitive cycle of sample injection into the furnace, and drying and ashing of the sample in order to raise the concentration of the target element in the sample. Repeating this boost cycle enables measurement of the sample whose concentration is lower than the quantification range by concentrating it and raising its absorbance to the quantitative range of the calibration curve.

In this case, use the default values as they are.

 Click on the [Reagent] button. The [Reagent Setup] dialog box will be displayed. This dialog box allows you to enter [Reagent Name] and [Reagent Position] of 4 kinds of reagents, and setup the intake order for the four reagents. In the [Blank Preparation Parameters], set up the automatic periodic blank measurement. The automatic periodic blank measurement is a function to create a measurement procedure on the MRT to eliminate the effect of baseline drift by inserting a blank measurement in a fixed interval.

- When periodic blank measurement is not performed: Do not tick the [Auto] field.
- When periodic blank measurement is performed:
- a. Tick the [Auto] field.
- b. Enter a value in the [Frequency] field to specify how many samples are measured between blank measurements.
- c. When using the ASC, the [Pos.] field is displayed if the [Using ASC] is ticked in the [Load Parameters] of [Element Selection] page. Enter the position of the blank sample on the turntable of the ASC.
- ③ Reslope is the sensitivity correction measurement. More specifically, a standard sample of a known concentration is measured during the measurement of unknown samples. Based on the measured absorbance, the slope of the calibration curve is corrected. Subsequently, the corrected new calibration curve will be used to calculate concentrations. If the [Auto] field is ticked in the [Reslope Preparation Parameters], reslope measurement is carried out at the concentration specified in [Conc.] by the specified frequency. The setup procedure is the same as in the [Blank Preparation Parameters].
- In [Measurement Sequence for Calibration Curve], enter the number of standard samples and their concentrations. Enter the number of standard samples in the [No. of Lines] field and click on the [Update] button. A table with that number of rows will be created. In this table, the default values for the concentrations of standard samples are already displayed under the standard parameters, but can be changed with the values for the actually prepared standard samples. If the ASC is used, the [Pos.] field is displayed. Enter the positions of the turntable (1 to 60, R1 to R8).

Mixing ON/OFF

When no samples are mixed using the ASC:

Do not tick [Mixing ON] or disable mixing. When mixing is disabled, the [Total Volume] in [Blank Preparation Parameters], [Reslope Preparation Parameters] and [Measurement Sequence for Calibration Curve] will be the amount injected to the graphite tube. Enter the amount of the sample injected to the graphite tube in the [Vol] field. (You cannot directly enter it in [Total Volume].) Leave all the [Diluent], [Reagent 1], [Reagent 2] and [Reagent 3] as zero. Then [Total Volume] will be the same value as [Vol]. Ensure that Total does not exceed 90 μ L. Normally, approximately 20 μ L would be appropriate. When only sample is injected (all of the diluent and reagent parameters are set to zero), the instrument will automatically determine that no mixing is performed, even if the mixing is enabled.

When samples are mixed using the ASC:

Tick Mixing ON to enable mixing. Enter each value in the [Vol], [Diluent], [Reagent 1], [Reagent 2] and [Reagent 3] fields. The total of the values in those fields will be automatically calculated and the result will be included in [Total Volume]. Prepare the concentrate solution of a standard sample, dilute it with the mixing capability of the ASC, and then prepare standard samples of different concentrations for calibration curves. An example of this process is given below.

NOTE

The [Total Volume] must meet the following conditions:

• When mixing is not performed:

(Injection Vol.) = (Total Vol.) </= 90 μL

• When mixing is performed:

(Injection Vol.) = (Max. No. of Reps.) × (No. of Boost cycles) + 50 μ L </= (Total Vol.) </= 600 μ L

(In this regard, the injection volume is the value specified on the [Mixing Setup] dialog box; +50 μ L is the dead volume of the mixing port.) If the boost cycle is not used, the No. of Boost cycles is "1".

Example

To prepare each 400 μ L of standard samples of 10, 20, and 30 ppb from the concentrate solution of a standard sample of 100 ppb, assume the concentrate solution of the standard sample as a "sample" and enter each value as follows. The total volume will be calculated automatically.

True Value	Pos	Standard Sample VOL	Diluent	Reagent 1	Reagent 2	Reagent 3	Total Volume
10	1	40	360	0	0	0	400
20	1	80	320	0	0	0	400
30	1	120	280	0	0	0	400

If the injection volume (entered on the [Mixing Setup] dialog box or the [ASC Parameters] page) is assumed as 20 μ L and the maximum number of repetition (entered in the [Repeat Measurement Conditions] dialog box) as 5, and the boost cycles as not performed ([Number of Boost Cycles] in the [Coating and Boost Cycles] dialog box is set to "1"), the calculation is as follows:

 $20 \ \mu\text{L} \times 5 \times 1 + 50 \ \mu\text{L} <= 400 \ \mu\text{L} <= 600 \ \mu\text{L}$

This means that the total volume meets the above condition.

Save Calibration Curve Setup Information

The once created [Calibration Curve Setup] information may be stored in the preparation parameter file so that it can be reused to specifying the parameters for other elements. For the file type, specify "*.mix". The preparation parameter file contains the following parameters:

Parameter	Screens
(a) Measurement Type (calibration curve method / standard addition method)	[Calibration Curve Setup] dialog box
(b) Mixing ON/OFF	[Calibration Curve Setup] dialog box
(c) Preparation Parameters (Blank, Reslope, STD)	[Calibration Curve Setup] dialog box
(d) Reagent Name and Reagent Position	[Reagent Setup] dialog box
(e) Reagent Intake Order	[Reagent Setup] dialog box
(f) Coating and Boost Cycles	[Coating and Boost Cycles] dialog box

NOTE

No files in which (a) Measurement Type and (b) Mixing ON/OFF are different from the current settings can be loaded.

3.3.5.2 Sample Group Setup

This window allows you to specify a sample group. If similar kinds of samples that are to be prepared with the same pretreatment are grouped, the effect of interference and the suitability of the pretreatment can be conveniently validated using the QA/AC function.

Upon making the sample group settings and clicking [OK], you will be returned to the [Preparation Parameters] page.

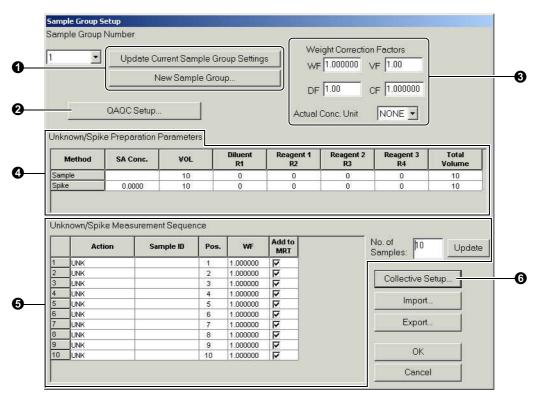


Fig. 3.68 [Sample Group Setup] Page

- In this example, use the default value of "1" for [Sample Group Number] and proceed forward without selecting the [Update Current Sample Group Settings] option and the [New Sample Group] option.
- 2 The [QA/QC Setup] will be described in the Chapter 6 "QA/QC Setup".

Senter [Weight Correction Factors]. These factors are required to calculate the actual concentrations. Weight Factor [WF], Volume Factor [VF], Dilution Factor [DF], and Correction Factor [CF] are used for the following equation:

Actual concentration = Concentration × [VF] × [DF] × [CF]/[WF]

The actual concentration is calculated with the above equation. The function for automatically converting the units is not available. If conversion of any unit is required, make adjustment using Correction Factor [CF]. (See the example.) If the calculation of the actual concentration is not required, leave all the factors as "1".

However, when the "Auto Dilution and Remeasurement" is performed, the dilution ratio used for the automatic dilution by the ASC is displayed in the [ASC DF] field (ASC dilution factor) on the MRT worksheet and the actual concentration is calculated as follows:

Actual concentration = Concentration × [VF] × [DF] × [ASC dilution factor] × [CF]/[WF]

Example

Assume that a reagent and diluent are added to 50 μ L of a sample into a total of 250 μ L. To obtain the actual concentration from the measured concentration, calculate it using 50 (μ L) for Weight Factor [WF], 250 (μ L) for Volume Factor [VF], and 1 for Correction Factor [CF] as follows:

Actual concentration = Concentration × (250 μ L) × 1 × 1/(50 μ L)

To obtain the actual concentration by converting the measured concentration (ppb) into the actual one (ppm) in the same example, use 0.001 (1 ppb = 0.001 ppm) for Correction Factor [CF] in the above equation as follows:

Actual concentration in ppm = Concentration in ppb × (250 μ L) × 1 × 0.001/(50 μ L)

- The [Unknown/Spike Preparation Parameters] allows you to enter the preparation parameters for unknown samples and spike samples. Spiking is one of the QA/QC techniques that are used to obtain the recovery rate by adding a solution of a known concentration to an unknown sample. In this example, proceed forward without entering a value (i.e., using the [S A Conc.] of 0.0000).
- In the [Unknown/Spike Measurement Sequence], enter the number of unknown samples and sample ID's. Enter the number of unknown samples in the [No. of Samples] field and click on the [Update] button. A table with that number of rows will be created. Sample ID can be entered one by one in the table, but can be entered at a time by clicking on the [Collective Setup] button. If the ASC is used, enter each turntable position (1 to 60) in the [Pos.] field.

NOTE

In the above ③, [Weight Correction Factors] has been entered. In general, [WF] varies depending upon each sample and can be entered in the [Unknown /Spike Measurement Sequence] table. Only the sample for which the [Add to MRT] field is ticked is inserted into the MRT worksheet on the main screen. The created [Unknown/Spike Measurement Sequence] table can be saved or loaded.

If you click on the [Collective Setup] button on the [Sample Group Setup] page of Fig. 3.68, the [Sample ID Collective Setup] dialog box will be displayed.

Sample ID Collective Setup

Clicking [Collective Setup...] on the [Sample Group Setup] page displays the [Sample ID Collective Setup] dialog box.

	Sample ID Collective Setup	×
0	— Number of Samples: 🔟 🐥	ОК
•		Cancel
0	Create Sample ID	
0	Sample ID- Name Start No. Sample + 1	ASC Start Pos.
0—	Pre-Digestion Spike (SPK) 20 Post-Digestion Spike (PDS) 20 Duplicate (DUP)	

Fig. 3.69 [Sample ID Collective Setup] Dialog Box

- 1 In the [Sample ID Collective Setup] dialog box, enter the number of unknown samples in the [Number of Samples] field.
- 2 To enter sample ID (sample name), tick the [Create Sample ID] field.
- When you enter a name and starting number in the enabled [Sample ID] field, the same name will be given to all the samples with sequential numbers from the starting number given to them. If the ASC is used, specify the position of the 1st unknown sample in the [ASC Start Pos.] field. The 2nd and subsequent positions will be automatically entered in the table.
- If [Pre-Digestion Spike (SPK)], [Post-Digestion Spike (PDS)], and [Duplicate (DUP)] are ticked, these measurements will be inserted in the analysis sequence for samples each in the number indicated on the right side field. Since these samples are used for QA/QC, proceed forward without ticking the above options in this example.

3.3.6 Connect to [Instrument/Send Parameters] Page

This section describes how to connect to the instrument and send the parameters. When the connection to the instrument is made, the instrument is initialized automatically.

(1) Check that the AA main unit and the related units are ON and click on the [Next] button.

Connect to Instrument/Ser	nd Parameters	X
	Power DN the instrument, and click on the [Connect/Send Parameters] button.	
	If the options are not recognized properly because the ASC/GFA are not powered on before initialization, click on the [Connect Options] button.	
	Meas. Element.	
	< <u>Back</u> Next > Finish Cancel Help	

Fig. 3.70 [Connect to Instrument/Send Parameters] Page

(2) Press the [Yes] button.



Fig. 3.71 Message Box

NOTE

- With the AA-7000F, check that the chimney has been correctly installed on the instrument, then click [Yes].
- If the chimney is not correctly installed on the instrument, the [Flame Monitor Check] may not be performed correctly at initialization, leading to an [NG] result.

The connection to the instrument will be started with the [Initialize] screen displayed and then the AA main unit will be initialized. After the initialization has been finished, the parameters for the element specified in [Meas. Element] are automatically sent to set up the instrument.

Alternatively, you may want to press the [Connect/Send Parameters] button in the [Connect to Instrument/Send Parameters] page to perform the same operation.

3.3.6.1 Initializing the Instrument

When the instrument is initialized, various items are automatically checked and the results are displayed as shown in the [Initialize] screen. The ROM versions of the AA main unit, ASC, and GFA can also be checked here.

a set to	
Initialize	
AA : AA-7000G v1.01 A3000000000	
ASC: ASC-7000 v1.01 A3000000000	
GFA: GFA-7000 v1.02 A3000000000	
ROM Check	
S/N Check	
ASC Check	
GFA Check	
Slit Origin Search	
D2 Attenuator Origin	
Turret Origin Search	
Testing 🔵 Success 🔶 Fa	illure ONo Test(Not Connected)
OK	
	ASC: ASC-7000 v1.01 A3000000000 GFA: GFA-7000 v1.02 A30000000000 ROM Check S/N Check ASC Check GFA Check GFA Check Slit Origin Search D2 Attenuator Origin Wavelength Origin Search Turret Origin Search Turret Origin Search

Fig. 3.72 [Initialize] Screen (AA-7000G)

No.	Name	Function
0	Instrument information	The models of the AA main unit, ASC and GFA, the ROM versions and the machine identification numbers are displayed here. If neither ASC nor GFA is connected, the ASC and GFA information is not displayed.
0	Automatic inspection points	These are inspections that the instrument performs automatically. If neither ASC nor GFA is connected, [Not Connected] () is shown for the [ASC Check] and [GFA Check] points.

For AA-7000F, see Fig. 3.18 "[Initialize] Screen (for AA-7000F/AAC)".

NOTE

- The time required from the start of initialization to completion of the automatic inspection points is 4 to 5 minutes (it varies depending on whether or not options are installed).
- For the Fuel Gas Pressure Monitor Check, Support Gas Pressure Monitor Check, or Drain Sensor Check item during the initialization, some messages may be displayed prompting you to check the safety devices. These checks must be periodically performed to check that the safety devices operate properly. When only the furnace measurement is performed, no check options are required. Click on the [No] button for all the options and then proceed with the initialization.
- (1) After all the items have been checked, a message may be displayed indicating that the fuel gas pressure or the drain tank water level is low. Click on the [OK] button here.
- (2) After the completion of the initialization, click on the [OK] button to close the [Initialize] screen.

3.3.6.2 Bypass the Instrument Check List for Flame Analysis

Since the furnace measurement is being setup and the flame measurement is not going to be carried out, click on the [No] button in the Message dialog box in Fig. 3.73.

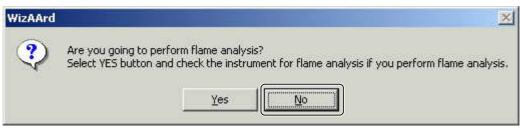


Fig. 3.73 Message Box

If you click on the [Yes] button when the flame measurement is not to be performed, tick all the check items in the Instrument Check List for Flame Analysis. This allows you to proceed forward to the next step.

3.3.7 **Optics Parameters**

The [Optics Parameters] page is used to set the parameters for the monochrometer and the lamps in the instrument. For the purpose of this example, proceed forward without changing the standard parameters (that were read out when the elements were selected). For changing each parameter, refer to the HELP information in the WizAArd software.

Optics Parameters					×
	📮 Cu				
	Wavelength: (185.0 - 900.0 r	324.8 m)	Lamp Current:		
1. July	Slit Width (nm): Lamp Mode:	0.7 V BGC-D2 V	Low(Peak): (0 - 40 mA)	8 🔺	
	Socket #:	Lamp Pos. Setup	High(Peak):		
		e Lamp Pos. Setup button, you can et manually and change the lamp.	(0 - 600 mA)		
	Lamp ID:	Cu-1	ASC Sample Pos Search:	. for EMISSION Line	
	Lamp ON: Lamp Status:	Line Search is necessary.]	NONE	
			Warmup Lamp	Line Search	
		< <u>B</u> ack <u>N</u> ext >	Finish	Cancel Help	

Fig. 3.74 [Optics Parameters] page

NOTE

This page displays the wavelength, slit width, socket number, lamp current, lamp mode and so on. These parameters are set for only the element that will be firstly measured (has been specified in the [Meas. Element] field located in the lower right part of the [Element Select] page or the [Connect to Instrument/Send Parameters] page).

The measurement parameters for each element are loaded from the cookbook and automatically specified when the elements are selected.

Normally, you do not need to enter these measurement parameters. To modify them, however, you can enter a value for the wavelength and select a value for other conditions from the list pulled down by clicking on the $[\mathbf{V}]$ button. The lamp current value can be changed in units of 1 mA by clicking on the $[\mathbf{A}]$ or $[\mathbf{V}]$ buttons.

(1) Click on the [Next] button. A message will be displayed prompting you to do line search. Click on the [OK] button.

The [Line Search/Beam Balance] dialog box will be displayed and the process will be carried out automatically.

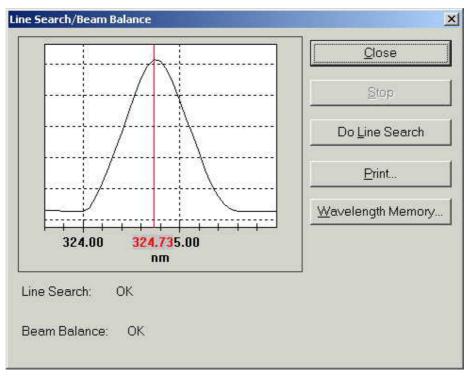


Fig. 3.75 [Line Search/Beam Balance] Dialog Box

- First the line search (wavelength matching) is carried out and then the beam balance (gain control for the detector) is performed. If only the beam balance is necessary, the line search is not performed.
- In the line search, the highest peak near the specified wavelength is detected. In some cases, however, the neon gas contained in the hollow cathode lamp radiates more intense light than the light from the element. In such a case, if the correctly analyzed line is stored by clicking on the [Wavelength Memory] button, its wavelength will be used for the subsequent line searches.
- (2) Upon completion of the process, click on the [Close] button to proceed forward to the [Atomizer/Gas Flow Rate Setup] page.

NOTE

When measuring multiple elements, you cannot set parameters for the elements other than the current measurement one on the [Optics Parameters] and [Furnace Program] pages. If you use the ASC to measure multiple elements automatically and you need to modify the parameters for other elements than the current measurement one, you can change these parameters by using the [Edit Parameters] button in the [Element Selection] page.

NOTE

SR lamp intensity is hard to stabilize in using NON-BGC mode or BGC-D2 mode in comparison with a normal lamp. Before performing the line search, wait for 15 to 20 minutes after turning on the lamp, and then start measurement.

3.3.8 Furnace Program

In the furnace measurement, the sensitivity can be changed or the effect of the background can be decreased if the settings for the furnace program are modified.

Stage#	Temp (°C)	Time (sec)	Heat Mode	Sensitivity	GAS Type	Flow Rate (L/min)
1	60	3	RAMP		#1	0.10
2	120	20	RAMP		#1	0.10
3	250	10	RAMP		#1	0.10
4	800	10	RAMP		#1	1.00
5	800	10	STEP		#1	1.00
6	800	3	STEP	V	#1	0.00
7	2300	3	STEP	V	#1	0.00
8	2500	2	STEP		#1	1.00

Example of Furnace Program

Sampling Stage No.: 7

The furnace program of "Example of Furnace Program" is explained as below.

- In this program, water is vaporized and the sample is "Dried" from stage #1 to #2, coexisting organic substances are "Ashed" from stage #3 to #6, and then the target element is "Atomized" at stage #7. The stage #8 is a "Cleaning" stage to eliminate the residue in the graphite tube.
- For the "Ashing" stage, change the [Temp.] and [Time] according to the contained coexisting substances (For example, if much amount of organic compounds are contained, set the [Temp.] higher and the [Time] longer).
- Set [Heat Mode] to "RAMP" (the temperature is increased gradually) during "Drying" stage and at the beginning of "Ashing" stage, and set it to "STEP" (heated directly to the set temperature) during "Ashing" and "Atomizing" stages.
- In the [Sensitivity] column, the "Atomizing" stage and its preceding stage are check marked to perform a high sensitivity measurement. At this time the inner gas is fixed to 0 (L/min).
- The [Sampling Stage No.] indicates a stage for data sampling. Usually, the atomizing stage is selected.
- The [Gas Type] is set to #1 for all the stages when a usual measurement is performed.

When changing/creating a furnace program, you can save it so that you can use the same program next time. For details, see 3.3.8.1 "Furnace Program Setup".

3.3.8.1 Furnace Program Setup

(1) In this page, set up the furnace program. The standard furnace program is displayed according to the selected element. In many cases, that program can be used as it is, but may be modified if necessary.

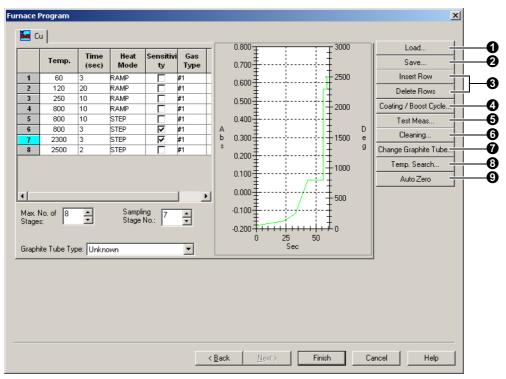


Fig. 3.76 [Furnace Program] Page

[Load]

To open the furnace program file, click on [Load]. The [Open] dialog box will appear. Then click on the file name to be opened and click on [Open].

2 [Save]

To save the furnace program, click on [Save] and enter the file name on the [Save As] dialog box. The extension for furnace program is ".fur" (For example, "copper.fur"). Then click on <Save>.

3 When editing a ready-made furnace program, use the [Insert Row] or [Delete Rows] buttons to insert a row or delete rows.

[Coating/Boost Cycle] button

Coating refers to injecting a specified reagent into the furnace and drying it prior to the sample injection (i.e., coating the tube surface). First, the checked mark reagent is injected in the furnace, and heating is executed up to the furnace program stage specified in [Last Coating Cycle]. After this, the sample is injected.

Furnace boost cycle refers to the repetitive cycle of sample injection into the furnace, and drying and ashing of the sample in order to raise the concentration of the target element in the sample. Repeating this boost cycle enables measurement of the sample whose concentration is lower than the quantification range by concentrating it and raising its absorbance to the quantitative range of the calibration curve.

G Clicking on the [Test Meas.] starts the test measurement allowing you to check to see whether the temperature program is appropriate. The peak profile of the measurement result will be displayed as a graph on the screen.

Clicking on the [Cleaning] button displays the [Cleaning] dialog box. If you click on the [Start] button, the graphite tube will be heated without a sample in accordance with the built-in heating program for cleaning. Also use the [Test Meas.] as necessary.

- Clicking on the [Change Graphite Tube] button displays the [Change Graphite Tube] dialog box.
- Clicking on the [Temp. Search] button displays the [Optimum Furnace Program Search] dialog box. This allows you to automatically carry out the operation of gradually changing the heating conditions and plotting the resulting data as a graph on the screen. Accordingly, the optimum atomization and ashing temperatures can be determined.
- Clicking on the [Auto Zero] button shifts the current displayed value to zero.
- (2) After completing all the settings, click on [Finish]. The Wizard finishes and the main window will appear.

If you want to use the parameters set with the above procedures from the next time, you can save them as a template. Then you can load it in the [Wizard Selection] dialog box.

- (1) Click on [File] in the menu bar and select [Save As].
- (2) Select "template (*.taa)" for [Save as type]. The extension of the file name displayed in [File name] is changed to ".taa".

Save As				? ×
Save jn: 🔂	WizAArd	•	🗢 🔁	-
) cookbook.t hyperlamp.				
l File <u>n</u> ame:	TEST.taa			<u>S</u> ave
Save as type:	Template (*.taa)		•	Cancel
				<u>Comment</u>

Fig. 3.77 [Save As] Dialog Box

Example

If "notitle.aa" is displayed in [File name] when the [Save As] dialog box is opened, selecting "template (*.taa)" changes the file name to "notitle.taa".

(3) Input the file name. Leave the extension as ".taa".

Example

Change the part "notitle" in the "notitle.taa" to a name you like.

(4) Press [Save] button. [Save As] dialog box is closed and the template file is saved.

Explanation of Main Window

When the Wizard is completed, the following main window appears.

- 1 Menu bar
- 2 Standard tool bar
- Measurement element tool bar (Current measurement element)
- Absorbance digital display
- **6** Real time graph (and temperature program graph)
- **6** Peak profile (latest four measurements and overlay display)
- Calibration curve tool bar (Selection of calibration curve and type)
- Calibration curve display
- Image: MRT work sheet
- Function buttons
- Status bar

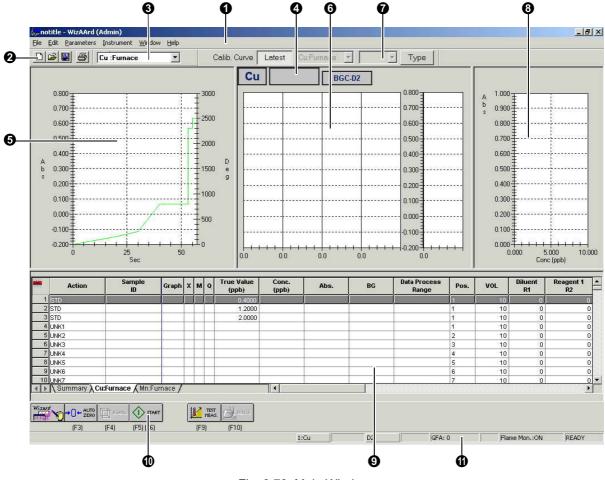


Fig. 3.78 Main Window

NOTE

While the [Properties] is opened from the right mouse button menu on the graph or MRT work sheet, the measurement is stopped. In this case, the measurement can be continued again when the [Properties] is closed.

3.5.1 Menu Bar

Place the mouse cursor onto the menu ([File], [Edit], [Parameters], etc.) and click on it. Then a drop-down menu appears. Select an item from the menu and click on it. For further details of each item, refer to the HELP information in the WizAArd software.

File	Edit	Parameters	Instrument	Window	Help
	2	8	Cu :Furnac	e	¥

Fig. 3.79 Menu Bar

3.5.2 Standard Tool Bar

The items frequently used are displayed in the tool bar. Quick selection is possible if you use this instead of selecting from the menu bar. Each tool function is described below.

New	D	: All the data and parameters are cancelled and parameters are set newly. (Same as [File]-[New] in the menu bar)		
Open	(J	: A file already created is opened. (Same as [File]-[Open] in the menu bar)		
Save As		: Current data and parameters are saved in the file. (Same as [File]-[Save As] in the menu bar)		
Print	6	: Data and parameters are printed. (Same as [File]-[Print Data/Parameters] in the menu bar)		
		😓 notitle - WizAArd (Admin)		
		File Edit Parameters Instrument Window Help		
		D 😂 📓 🥌 Cu :Furnace 💌		

Fig. 3.80 Standard Tool Bar

3.5.3 Measurement Element Tool Bar

The element currently measured (or to be measured) is indicated.

(1) If you want to change the element, click on the $[\mathbf{\nabla}]$ and select one from the drop-down list.

Then the page proceeds from [Optics Parameters] to [Atomizer/Gas Flow Rate Setup] (for flame method) or [Furnace Program] (for furnace method).

(2) Set the parameters if necessary and click on [Finish] (or [Cancel] to close the sheet without changes).

The MRT work sheet is also changed to that for selected element. This is the same action as results from selecting [Parameters] - [Measurement Preparation Wizard] from the menu.

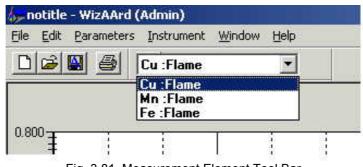
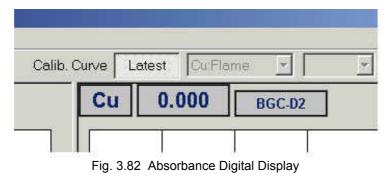


Fig. 3.81 Measurement Element Tool Bar

3.5.4 Absorbance Digital Display

When the instrument remains connected, the current absorbance value (or emission intensity in the EMISSION mode) is displayed in flame mode. The latest measured result is displayed in furnace mode. If not, nothing is displayed. When the instrument is in the BUSY mode, the value is highlighted.



3.5.5 Real Time Graph (and Furnace Program Graph)

The changing absorbance value (or emission intensity in EMISSION mode) under measurement is displayed in analog. You can check the peak shape and baseline conditions with this graph. In the case of furnace, "Furnace Program" is also displayed. The abscissa indicates time and the ordinate indicates absorbance (left scale) and temperature (right scale).

Right mouse button menu

If you want to change the scale or print the graph, place the cursor in the graph area and click on the right button of the mouse. Then the pop-up menu shown in Fig. 3.83 appears. Select an item and make settings.

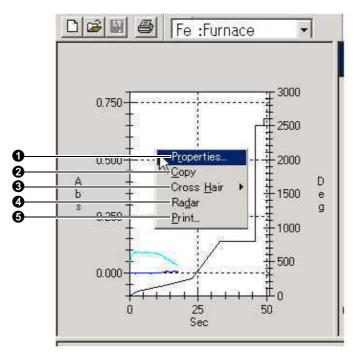


Fig. 3.83 Right Mouse Button Menu (Real Time Graph)

1 [Properties]

[Real Time Graph Scale] (Flame)

You can change the graph ordinate and abscissa scale. Enter the maximum value and minimum value in [Ordinate Max] and [Ordinate Min], respectively. Enter a numeric value in [Time Interval] (unit: second) for abscissa. Clicking on [Reset] sets them to the values specified in [Parameters]-[Default Parameters]-[Graph].

[Scale] (Furnace)

You can change the scales for [Absorbance], [Time] and [Temperature]. Enter the maximum value and minimum value in [Max] and [Min], respectively. Clicking on [Reset] sets the absorbance scale to the values specified in [Parameters]-[Default Parameters]-[Graph], and sets the time and temperature scales according to the furnace program.

[Color]

You can select colors of [Data Line], [BG Line], [Background] and [Grid] (also [Furnace Program] for furnace method). Click on the $\mathbf{\nabla}$ and select the color from the list.

[Grid]

You can select grids to be displayed from [Major & Minor Grid], [Major Grid] or [None]. You can also select the line type of [Major Grid Line] and [Minor Grid Line].

② [Copy]

Executing [Copy] copies the image data of currently displayed graph to the clipboard. Then start up the word processor or other application and move the cursor to the location where you want to paste the graph. Select [Paste] command in the word processor, etc., and the graph will be displayed at the cursor position.

3 [Cross Hair] (Furnace)

This is used to read coordinate values in the graph.

- Move the mouse cursor in the graph area first, then select [Cross Hair]-[Display] in the right button menu. When the mouse cursor is in the graph area, the coordinate values at the cross hair intersection position are displayed.
- · While the cross hair cursor is displayed, clicking on the right mouse button and selecting [Cross Hair]-[Lock] will fix the cross hair at that position. To free the cross hair, click on the right mouse button again and select [Cross Hair]-[Lock] (then the check mark of [Lock] will be deleted).
- · While the cross hair cursor is displayed, clicking on the right mouse button and selecting [Cross Hair]-[Display] (then the check mark of [Display] will be deleted) will erase the cross hair regardless of the [Lock] condition.

(Radar)

The display range is automatically set so as to display the entire graph selected by the mouse cursor.

6 [Print]

Selecting this opens the [Print] dialog box. Check the printer name, copies, etc. and click on [OK]. The real time waveform currently displayed will be printed.

3.5.6 Peak Profile (Latest Four Measurements and Overlay Display)

The latest four measurement signal profiles are displayed. Both standard samples and unknown samples are displayed here. The abscissa shows time and the ordinate shows absorbance value (emission intensity in EMISSION mode). The signal profile is displayed during the integral time in the case of flame continuous method, and during the pre-stage time and sampling time in the case of flame micro sampling method and furnace method. In the rightmost area, plural number of signal profiles can be overlapped and displayed. Specify their colors in the [Graph] on the MRT work sheet.

Right mouse button menu

If you want to change the scale or print the graph, place the cursor in the graph area and click on the right button of the mouse. Then the pop-up menu shown in Fig. 3.84 appears. Select an item and make settings.

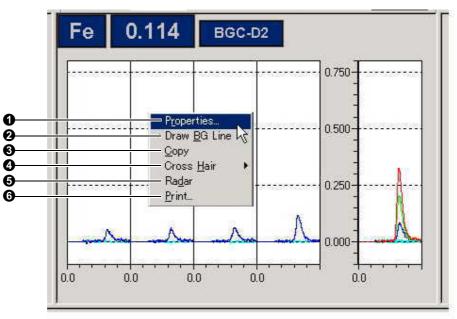


Fig. 3.84 Right Mouse Button Menu (Peak Profile)

1 [Properties]

[Scale]

You can set the maximum value [Max] and minimum value [Min] of the graph ordinate and abscissa scales. The scale setting is used commonly for the five graphs. Clicking on [Reset] sets the Y-axis to the value specified in [Parameters]-[Default Parameters]-[Graph].

[Color]

You can select colors of [Data Line], [BG Line], [Background] and [Grid]. Click on the ▼ and select the color from the list.

[Grid]

You can select grids to be displayed from [Major & Minor Grid], [Major Grid] or [None]. You can also select the line type of [Major Grid Line] and [Minor Grid Line].

2 [Draw BG Line]

Selecting this displays a profile of background signal. The display is deleted by selecting this menu again (The check mark of [Draw BG Line] is deleted).

(Copy)

Executing [Copy] copies the currently displayed graph to the clipboard. Then start up the word processor or other application and move the cursor to the location where you want to paste the graph. Select [Paste] command in the word processor, etc., and the graph will be displayed at the cursor position.

(Cross Hair)

This is used to read coordinate values in the graph.

• Move the mouse cursor in the graph area first, then select [Cross Hair]-[Display] from the right button menu. When the mouse cursor is in the graph area, the coordinate values at the cross hair intersection position are displayed.

The graph area is divided into five areas, and the coordinate values can be read only in each area.

- If you want to read an coordinate value in another graph, erase the cross hair cursor once by clicking the right mouse button and selecting [Cross Hair]-[Display] (then the check mark of [Display] will be deleted), then set the mouse cursor to another area and select Cursor]-[Display] again.
- While the cross hair cursor is displayed, clicking on the right mouse button and selecting [Cross Hair]-[Lock] will fix the cross hair at that position. To free the cross hair, click on the right mouse button again and select [Cross Hair]-[Lock] (then the check mark of [Lock] will be deleted).
- While the cross hair cursor is displayed, clicking on the right mouse button and selecting [Cross Hair]-[Display] (then the check mark of [Display] will be deleted) will erase the cross hair regardless of the [Lock] condition.

6 [Radar]

This automatically sets the display range so that the selected graph by the mouse cursor is displayed in an appropriate size. At the same time, other four data scales are also changed to the same scale.

6 [Print]

Move the mouse cursor to the graph area to be printed. Then the [Print] dialog box appears. Check the printer name, copies, etc. and click on <OK>. Only the selected peak profile will be printed.

3.5.7 Calibration Curve Display

After the standard samples are measured, the calibration curve is created and displayed.

Calibration Curve Display

If you want to check a calibration curve other than the currently displayed one, set the [Latest] button to OFF (the button is embossed) and enter the element name and calibration curve number (C#). The settings here are limited to display and recalculation is not executed.

Changing the Calibration Curve Conditions

If you want to change the calibration curve parameters after viewing the created calibration curve, use the [Type] button. Clicking on [Type] opens the [Change Calibration Curve Condition] dialog box where you can change order of calibration curve and permission of zero intercept.

Change Ca	ibration	Curve Condition		×
Element:	Cu	Flame	ОК	
<u>O</u> rder:	1st	•	Cancel	
∏ <u>Z</u> ero	Interce	pt		

Fig. 3.85 [Change Calibration Curve Condition] Dialog Box

Changing the condition and clicking on [OK] immediately recalculates the concentration of unknown samples based on the new calibration curve and renews the quantification result on the MRT work sheet. The recalculation is only applied to the unknown sample using the same C# as changed calibration curve. The result is invalid when the element is different, or when the C# is different even if the element is same. In the case of furnace method, whether the absorbance is obtained from the peak height or from the peak area is switched in this window.

NOTE

After the validation based on the QA/QC setup is executed, the calibration curve order, zero intercept and signal processing mode settings cannot be changed after the measurement. For details on QA/QC setup, refer to the Chapter 6 "QA/QC Setup".

Symbols in Calibration Curve Equation

When a calibration curve is created, calibration curve equation is displayed above the graph. Symbols in a calibration curve equation mean as follows:

Symbol	Meaning
Abs	Absorbance
E	Energy (Only for EMISSION mode)
Conc.	Concentration
r	Correlation Coefficient
e	When a coefficient is very small, this symbol is used to express it by using index. "AeB" means "A × 10 ^B "
٨	This symbol means power. "Conc^2" means the second power of concentration.

Example

Calibration curve equation <1> means equation <2>.

Abs = -9.5e-005Conc^2 + 0.011636Conc + 0	<1>
Abs = $-9.5 \times 10^{-5} \times \text{Conc}^2 + 0.011636 \times \text{Conc.} + 0$	<2>

Right mouse button menu

If you want to change the scale or print the graph, place the cursor in the graph area and click on the right button of the mouse. Then the pop-up menu shown in Fig. 3.86 appears. Select an item and make settings.

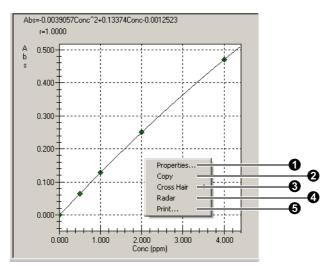


Fig. 3.86 Right Mouse Button Menu (Calibration Curve Display)

1 [Properties]

[Scale]

You can set the maximum value [Max] and minimum value [Min] of the graph ordinate and abscissa scales. Enter the values.

[Options]

You can set the graph color and data point shape. Clicking on each $[\Psi]$ of [Data Point], [Calibration Curve], [Background] and [Grid] displays the list from which you can select the color. The shape of [Data Point] can be selected from the list displayed by clicking on the $[\Psi]$.

[Grid]

You can select grids to be displayed from [Major & Miner Grids], [Major Grid] or [None]. You can also select the line type of [Major Grid Line] and [Minor Grid Line].

[Copy]

Executing [Copy] copies the currently displayed graph to the clipboard. Then start up the word processor or other application and move the cursor to the location where you want to paste the graph. Select [Paste] command in the word processor, etc., and the graph will be displayed at the cursor position.

3 [Cross Hair]

This is used to read coordinate values in the graph. Move the mouse cursor in the graph area first, then select [Cross Hair]-[Display] from the right button menu. When the mouse cursor is in the graph area, the coordinate values at the cross hair intersection position are displayed.

To erase the cross hair, select [Cross Hair]-[Display] (then the check mark of [Display] will be deleted).

While the cross hair cursor is displayed, clicking on the right mouse button and selecting [Cross Hair]-[Lock] will fix the cross hair at that position. To free the cross hair, click on the right mouse button again and select [Cross Hair]-[Lock] (then the check mark of [Lock] will be deleted).

While the cross hair cursor is displayed, clicking on the right mouse button and selecting [Cross Hair]-[Display] (then the check mark of [Display] will be deleted) will erase the cross hair regardless of the [Lock] condition.

4 [Radar]

This automatically sets the display range so that the entire graph can be displayed.

6 [Print]

Move the mouse cursor to the graph area to be printed. Then the [Print] dialog box appears. Check the printer name, copies, etc. and click on [OK]. Then the calibration curve currently displayed is printed out. This menu is not displayed when no calibration curve exists.

3.5.8 MRT Work Sheet

It is possible to create measurement sequence, execute measurement, display the result, and calculate the actual concentration in the MRT work sheet. If measurement of plural elements is required, the work sheet is prepared for each element and the work sheets can be changed over with the tab. In [Summary], the measurement result for sample can be displayed at one time. The tab title consists of the element name and comment. As the comment, only the first line is displayed when a line feed is included in the comment, and the first ten characters are displayed when the number of characters exceeds ten. The comment is inputted by selecting [Parameters]-[Edit Parameters]-[Comment] page from the menu bar.

For details on each item in MRT work sheet, refer to the section 3.6 "Operating the MRT Work Sheet".

NOTE

The currently measured element (or the element about to be measured) can be changed not by the tab at the left lower of the main window but by the measurement element tool bar.

3.5.9 Function Buttons

Functions of Wizard selection, auto zero execution, blank measurement, starting sample measurement, test measurement, ASC rinse are assigned to the buttons at the left below the MRT work sheet. These correspond to function keys on the keyboard as follows.

[AUTO ZERO] button	: [F3] key
[BLANK] button	: [F4] key
[START] button	: [F5], [F6] keys This switches to [STOP] button during measurement.
[TEST MEAS.] button	: [F9] key
[RINSE] button	: [F10] key

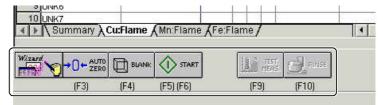


Fig. 3.87 Function Buttons

The relations between the current measurement mode and the available buttons are shown below. "Auto (using ASC)" means the status where the ASC is connected and the [Using ASC] check box is check marked in [Parameters]-[Edit Parameters]-[Sequence] page from the menu bar.

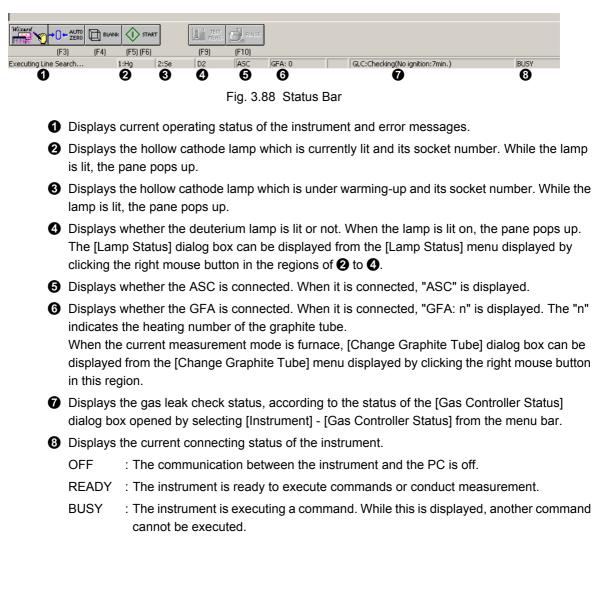
Operation	Flame Continuous Method Manual Flame Micro Sampling Method Manual	Flame Continuous Method Auto (using ASC)	Furnace Method Manual	Flame Micro Sampling Method Auto (using ASC) Furnace Method Auto (using ASC)	
[AUTO ZERO]	0	0	0	0	
[BLANK]	0	×	×	×	
[START]	0	0	0	0	
[TEST MEAS.]	×	×	0	0	
[RINSE]	×	0	×	0	

 \bigcirc = Available \times = Not available

- Clicking on [AUTO ZERO] button executes the Auto Zero while rinsing the nozzle in the case of flame continuous method auto (using ASC). In other cases, only the Auto Zero is executed.
- If you click on [Rinse] button, the nozzle is rinsed for the flame continuous method or the nozzle and mixing
 port are rinsed for the flame micro sampling method and the furnace method (the mixing port is only rinsed
 when the mixing ON option is selected.) The nozzle rinse time in flame continuous measurement is set in
 [Configuration] dialog box opened by selecting [Instrument]-[Configuration] from the menu bar. If the
 setting is "0", 10 seconds rinse is executed regardless the setting.
- Clicking on [STOP] stops the measurement. However, while acquiring the data, the measurement is stopped after the data acquisition is finished.

3.5.10 Status Bar

The current measurement mode, operating mode, operating status of instrument, and connecting status of peripheral equipments (ASC, GFA) are indicated here.



3.6

In the MRT (Measurement Result Table) work sheet, the functions of setting of measurement sequence, execution of measurement, result display, actual concentration calculation, and setting of ASC sample position are integrated. For example, if you create a calibration curve by measuring three standard samples and measure eight unknown samples, the measurement is proceeded in the following procedure.

Row 1 to 3 :Executes measurement of standard samples.

Row 4 to 11 :Executes measurement of unknown samples (SAMPLE001 to SAMPLE008).

As the measurement procedure, prepare the sample shown in the [Action] column and click on [START]. You can proceed the measurement in the order from top row to down. When a repeat measurement is set, rows for repetition are inserted at the time of measurement.

3.6.1 Fields of MRT Work Sheet

This section explains about the fields of MRT work sheet.

2	Action	Sample ID	Graph	x	м	Q	True Value (NONE)	Conc. (NONE)	Abs.	BG
- 1	STD						0.5000		l I	l
2	STD						1.0000		0	
3	STD						2.0000)(
4	UNK1)	
5	UNK2)(
6	UNK3)	
7	UNK4						0		(
8	UNK5								(
9	UNK6								(0
-10	UNK7								(
-11	UNK8						((
12	UNK9									
13	UNK10									

Fig. 3.89 MRT Work Sheet

· [Action]

100

Clicking on the cell in this field opens the drop-down list, showing the following indications. This [Action] field contains the measurement operations, operation using the ASC, QA/QC operation, etc.

For details, see 3.6.1.1 "Drop-down List of [Action]".

[Sample ID]

In normal operation, sample ID is entered in this field. Sample name can be entered only when [Action] field indicates measurement of standard sample or unknown sample (BLK, STD, UNK, LCS, SPK, RESLOPE, MSA, SMSA, MSA-RES, ICV, CCV, ICB, CCB, PB, PDS, DUP, and CRA). When the [Action] is CAL-CHK, the sample name cannot be entered.

If you previously enter Sample ID in the [Calibration Curve Setup] page or the [Sample Group Setup] page and then execute [Edit]-[Insert Calibration Curve] or [Insert Sample Group] from the menu bar, the data on rows of MRT worksheet can be given at a time. This eliminates the labor of entering data on each row.

NOTE

In the following six cases, the [Sample ID] has a special meaning.

- When [RINSE] is set in flame continuous measurement (using ASC), enter the rinsing time in second unit (0 to 600 seconds) in this [Sample ID].
- When [PAUSE] is set, enter the message to be displayed on the message dialog box in this [Sample ID].
- When [WAIT] is set, enter the wait time of second unit in this [Sample ID] (0 to 7200 seconds).
- When [COMMAND] is set, describe the command line in this [Sample ID].
- When [FILESAVE] is set, enter the file name together with path to save the data measured so far (e.g. C:\AA\WATER_CU.aa). Use ".aa" for extension.
- When [FILEEXPORT] is set, enter the file name together with path. Then the data on the MRT work sheet data of the currently measured element is saved in a text file (e.g. C:\AA\WATER_CU.txt). Use ".txt" for extension.
- [Graph]

When overlaying the peak profiles, click on the right part of the cell and select the color of data lines from the list. The overlaid profiles are displayed on the rightmost of peak profile area. On the other hand, the BG lines are overlaid by the same one color.

• [X] (Exclusion)

Double-clicking this excludes the data in the row. The exclusion can be deselected if you double-click on this button again.

• [M] (Modified)

Rewriting the sample ID and true value after measurement displays M mark (Modified). Once rewritten, this check mark is always displayed and the original data cannot be recovered.

• [Q]

A mark is displayed for each row to which the QA/QC check selected in the [QC Blank/QC Standard Setup] page or the [Sample Group QA/QC Setup] page has been applied.

Marked data cannot be excluded or modified.

• [True Value (and unit)]

The set concentration (and unit) of standard samples is indicated. The unit set in the [Calibration Curve Setup] Page is indicated in parentheses. This can be entered only when the [Action] is STD, LSC, RESLOPE, MSA, ICV, CCV, and CRA.

• [Conc. (and Unit)]

Displays the result of concentration obtained with measurement. As the [True Value] column heading, the unit specified in the [Calibration Curve Setup] page is automatically displayed.

• [Abs.]

Displays the measured absorbance. Energy intensity is displayed instead in the case of EMISSION mode. If a blank measurement is executed prior to this, the value is indicated after the measured blank value is subtracted from the actual measured value.

• [BG]

For measurement in BGC-D2 or BGC-SR mode, absorbance of background signal is displayed. This is not displayed in the lamp modes in which the background signal is not acquired.

When "Peak Height" is selected for signal processing mode in furnace measurement, this field indicates the background value at the time of acquisition of the absorbance signal peak height data.

• [Data Processing Range (sec)]

Specify the range in which data is used among the sampling data. The value that has been entered in the [Change Peak Data Processing Range] dialog box displayed by selecting [Edit]-[Change Peak Data Processing Range] from the menu bar is displayed.

• [Pos.]

Indicates the sample position in the turntable of the autosampler (ASC). For details, refer to the instruction manual provided with the ASC.

• [VOL] [Diluent] [Reagent 1] [Reagent 2] [Reagent 3] [Total Volume]

These are the amounts of sample and reagents that are required for mixing by the autosampler in the flame micro sampling method or the furnace measurement. The values entered in the tables of the standard preparation parameters in the [Calibration Curve Setup] page and unknown preparation parameters in the [Sample Group Setup] page are displayed here.

The header row of the [Diluent] [Reagent 1] [Reagent 2] [Reagent 3] displays the settings in [Diluent/ Reagent Name & Position] which are set in [Parameters]-[Edit Parameters]-[ASC Parameters] page from the menu bar.

• [WF] [VF] [DF] [ASC DF] [CF]

Each acronym means as follows.

[WF] [Weight Factor]

[VF] [Volume Factor]

[DF] [Dilution Factor]

[ASC DF] [ASC Dilution Factor]

[CF] [Correction Factor]

These are factors necessary for actual concentration calculation. The factors set in [Weight Correction Factors] in the [Sample Group Setup] page are displayed.

The [ASC DF] is displayed only in the case of furnace auto-measurement using ASC. The dilution factor of automatic dilution is displayed when the automatic dilution and remeasurement is performed.

• [Actual Conc.]

Indicates the result of actual concentration calculation. For the calculation, the factors of WF, VF, DF and CF are used.

NOTE

The actual concentration is calculated using the equation: Actual concentration = (concentration) \times VF \times DF \times ASC DF \times CF/WF. However, no functions are available for specifying the units of WF and VF. To convert the units, use CF.

ASC Dilution Factor is a value used to prevent the actual dilution ratio from changing when the software automatically modifies the mixing conditions due to the automatic dilution and remeasurement.

• [Actual Conc. Unit]

Enter the unit used for actual concentration of the sample. Clicking on the right part of the cell opens the drop-down list from which the unit can be selected. Note that changing this unit does not recalculate the actual concentration. It is necessary to change the factors used for calculation.

• [%RSD] [SD] [%R]

The [%RSD] and [SD] are calculated in repeat measurement.

The [%R] is calculated when "LCS", "SPK", "ICV", "CCV", "PDS" or "DUP" is measured.

For details on these calculation equations, refer to the Chapter 6 "QA/QC Setup".

• [C#]

Indicates calibration curve number. When plural calibration curves are created on the same sheet, number is put in the order of creation and indicated.

If entering a changed number, in the case of standard sample, a calibration curve is created by the standard samples of the same curve number and in the case of unknown sample, the concentration is calculated using the calibration curve of the entered number. This can be entered when the [Action] is STD, UNK, CAL-CHK, LCS, SPK, RESLOPE, MSA, SMSA, MSA-RES, ICV, CCV, ICB, CCB, PB, PDS, DUP or CRA.

This number is also used when selecting the calibration curve to be displayed (refer to the section 3.5.7 "Calibration Curve Display").

• [SG#]

Displays the sample group number.

• [Out of Control Remark]

A Note is displayed when QA/QC function judges that the acquired data is out of acceptance range.

• [Date]

Displays the date when the data was obtained by measurement.

• [Time]

Displays the time when the data was obtained by measurement. In the case of repeat measurement, the time displayed in the average row is the time of the first measurement.

• [User Name]

Displays the user name who acquired the data.

• [Device Name]

Displays the device name used to acquired the data. The device name comes from [Instrument]-[Configuration]-[Device Name].

3.6.1.1 Drop-down List of [Action]

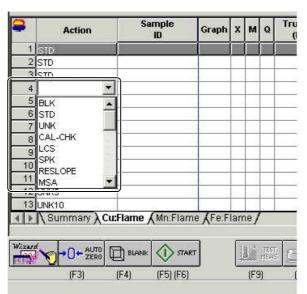


Fig. 3.90 Drop-down List of [Action]

BLK: Specifies the blank sample measurement. The blank measured value will be subtracted from the measured values of standard or unknown samples until the next blank measurement is executed.

STD: Specifies the standard sample measurement by calibration curve method.

- UNK: Specifies the unknown sample measurement by calibration curve method.
- CAL-CHK: Specifies the evaluation of calibration curve after the standard sample measurement (the correlation coefficient is checked). This is usually inserted to the row just after the last "STD". In the case of standard addition method or simple standard addition method, this setting is not necessary because this is checked in the "MSA-RES" row.
- LCS: Specifies the measurement and evaluation by LCS (Laboratory Control Sample/Standard substance).
- SPK: Specifies the measurement and evaluation by SPK (Pre-Digestion Spike/ addition and recovery check without pretreatment). This is usually inserted to the row just after the objective "UNK".
- RESLOPE: Specifies the measurement for sensitivity correction (Usually, the standard sample of highest concentration is used). After this, "A, B, C..." is added to C# on the MRT and a new calibration curve number (C#) is created.
- MSA: Specifies the measurement by standard addition method.
- SMSA: Specifies the unknown sample measurement by simple standard addition method.
- MSA-RES: Specified as the result of standard addition method. When the measurement of a set of standard addition samples is finished, the calibration curve is created and the result is indicated in this row at the same time.
- AUTOZERO: Shifts the current displayed value to zero. In the case of flame continuous method using the ASC, the Auto Zero can be executed while aspirating the rinse solution. The aspirating time (0 to 600 seconds) in second unit can be specified in [Sample ID] field.
- RINSE: In the case of flame micro sampling method or furnace method, the nozzle and mixing port are rinsed (mixing port is rinsed only when Mixing is ON). In the case of flame continuous method, the nozzle is rinsed. When the ASC is used, the rinse time (0 to 600 seconds) in second unit can be specified in [Sample ID] field.
- CLEANING: Specifies cleaning of the graphite tube.
- PAUSE: Specifies temporary stop until the [OK] is selected. Input a message to be displayed on the message dialog box in the [Sample ID] field.
- WAIT: Specifies a stop in a fixed time. Input waiting time in the unit of second in the [Sample ID] field (0~7200 seconds).
- COMMAND: Specifies execution of command line. Specify the command to be executed in the [Sample ID] field.
- FILESAVE: Specifies the file saving of the data collected so far. Specify the file name in the [Sample ID] field.

FILESpecifies the text file saving of the data collected so far on the MRT work sheet. Specify the
file name in the [Sample ID] field.

ICV, CCV, ICB, CCB, PB, PDS, DUP, and CRA also carry out the measurement and evaluation using each QC sample as LCS and SPK do. Normally, insert PDS and DUP next to the targeted UNK.

NOTE

- 1. Executing "FILESAVE" overwrites and saves the file even if the file of the same name exists.
- When the path name is not set at all in [FILESAVE] or [FILEEXPORT], a path name is created based on the setting [File]-[Auto Save]. When only the file name is set, the file is saved in the folder that was used in the last time for [Open] or [Save As] screen.
- 3. To enable the QA/QC actions, it is necessary to put a check mark in the check box of each QA/QC type in the [QC Blank/QC Standard Setup] page and [Sample Group QA/QC Setup] page.

3.6.2 Right Mouse Button Menu

Clicking on the right mouse button on the MRT work sheet opens the pop-up menu as below.

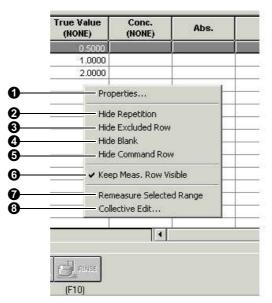


Fig. 3.91 Right Mouse Button Menu (MRT work sheet)

1 [Properties]

[Table Show/Hide]

You can select to show or hide each column on the MRT work sheet. Only check marked items are displayed. Click on the item you don't want to show, and erase the check mark. The show/hide selection for each column has no relation to the print items in the [File]-[Print Table Data]. To select the items to be printed in Table Print, select [File]-[Print Style] from the menu and select the items in [Table Show/Hide] page.

When the ASC is set not to be used in the [Parameters]-[Edit Parameters]-[Sequence], the items related to ASC such as [Pos.], [VOL], [Diluent], [Reagent 1], [Reagent 2], [Reagent 3], [Total Volume] and [ASC DF] are not shown or printed, even if these items are set to be shown or printed in the [Table Show/Hide] page.

						×
Tab	le Show/Hide Row	Style				
	- Measure	· · ·				
	I▼ BG	🔽 True 🛛	(alue	🔽 Dat <u>e</u>	🔽 User <u>N</u> a	ame
	✓ ≚(Exclude)	☑ C <u>o</u> nc		✓ <u>T</u> ime	🔽 Device	<u>N</u> ame
	M(Modify)	Actual	Conc	C#(Calibration	Curve Number)	
	🔽 Q(QC Lock)	🔽 Actual	Conc <u>U</u> nit	🔽 Select <u>G</u> raph	Color	
				🔽 <u>P</u> eak data pro	cessing range	
	-Weight Correction I	Factors	_QA/QC-		Autosampler-	
	Volume Eactor	r	✓ <u>%</u> RSI	D	✓ Position	
	Dilution Factor	r			🔽 Sam <u>p</u> le V	/olume
	ASC Dilution F	actor			🔽 Djluent	
	Correction Fac	tor	▼ % <u>B</u>		🔽 Reagent	1
	🔽 🔟 eight Factor				🔽 Reagent	2
					🔽 Reagent	3
			🔽 Out d	of Co <u>n</u> trol Remark	🔽 Total Voj	ume
			ОК	Cancel	Apply	Help
		L	OK		CPPV	

Fig. 3.92 Table Show/Hide (MRT Work Sheet)

[Row Style]

Font (character style, size, etc.), color and ruled line type can be set for each row group (row and column header, basic style, current row, measurement result row and excluded row).

Header of Row & Column		eview:	
Basic Style.,	7 I-	Header	
5 doi: 0 476		Basic	
Current Row	3	Current Result	
Measurement Result Row	4	Excluded	
measurement nesult now	5		
Measurement Excluded Row			
	J /		
	J /		

Fig. 3.93 Row Style (MRT Work Sheet)

2 [Hide Repetition]

When this is selected in the case of repeat measurement, only the row of average value is shown and the rows of repeating process are not shown. While this is selected, a check mark is indicated in the drop-down menu.

3 [Hide Excluded Row]

When this is selected, a row with a check mark in its [X] (excluded) field is not shown.

[Hide Blank]

When this is selected, a row of blank measurement is not shown.

(Hide Command Row]

When this is selected, a row whose [Action] field is "Command" is not shown.

6 [Keep Meas. Row Visible]

When this is selected, the MRT work sheet is automatically scrolled as the measurement proceeds so that the row under measurement may be always displayed.

[Remeasure Selected Range]

When excluding measured rows and remeasuring the sample, the operation is as follows.

- (1) First, select rows to be remeasured by using the mouse (Click the first row and drag to the last row while pressing the button). In the case of repeat measurement, the selected range must include the average row. A row (or sample) that is already excluded cannot be remeasured. If remeasuring the excluded row is necessary, double-click the [X] field to recover it once.
- (2) Click the right mouse button and select [Remeasure Selected Range] from the displayed menu.
- (3) The remeasurement rows are added below the last measured row on the MRT work sheet. The rows in the selected range are marked in the [X] fields and excluded.
- **8** [Collective Edit]

To change the factors and unit for actual concentration calculation together, which are already set on the MRT work sheet, follow the procedure described below.

- (1) Select the cell range to be changed by using the mouse (Click the first row to be changed and drag to the last row with pressing the button).
- (2) Click the right mouse button and select the [Collective Edit] from the displayed menu. Then the [Collective Edit] dialog box appears. Put a check mark to the check box of each item to be changed. Enter a new setup value to the item with the check mark.

ollective Edi		1	<u>× ×</u>			1
✓ Actual C ✓ Volume		NONE	•			1 1
Sample (uL)	Diluent R1	Reagent 1 R2	Reagent 2 R3	Reagent 3 R4		<u>.</u>
10	0	0	0	0		
12-52	agent1		1			OK
I He	eagent 2				_	Cancel
E Re	eagent 3					

Fig. 3.94 [Collective Edit] Dialog Box

(When using the flame continuous method, [Volume] and [Coating] are not displayed.) (When using the flame micro sampling method, [Coating] is not displayed.)

(3) When completed, click on [OK] to close the [Collective Edit] dialog box. Settings are changed and the actual concentration is recalculated simultaneously and the work sheet is renewed.

In rows already measured, however, [Volume] and [Coating] cannot be edited.

3.6.3 Right Mouse Button on Summary Table

Clicking on the right mouse button on the Summary table displays the pop-up menu where you can select Show/Hide of Absorbance, Concentration, Actual Concentration and Concentration Unit. This Show/Hide selection is also used for the "Print Table Data".

3.6.4 Inserting and Deleting the Measurement Row

Only the row that has not been measured can be inserted or deleted for measurement.

NOTE

Insertion and deletion of a row next to current measuring row cannot be done during measurement.

To insert the row

- (1) Click on the number of row position to be inserted (a new row is added above the row).
- (2) Select [Edit]-[Insert Row] from the menu bar.

To delete the row

- (1) Select the number of row (or the range of plural rows) to be deleted.
- (2) Select [Edit]-[Delete Rows] from the menu bar.

3.6.5 Active Cell Movement by Shortcut Key and Cell Selection

3.6.5.1 Moving an Active Cell

Clicking a cell with the mouse pointer displays a bold frame around the cell. The cell in this status is called an active cell. However, all the cells cannot be activated. Some cells can be activated but some cannot be. The active cell can be moved through the following key input.

Home

This key moves the active cell to the leftmost cell that can be active in the current row.

End

This key moves the active cell to the rightmost cell that can be active in the current row.

Ctrl+Home

These keys move the active cell to the leftmost cell that can be active in the first row unexecuted.

Ctrl+End

These keys move the active cell to the rightmost cell that can be active in the last measurement row.

Tab

This key moves the active cell to the right. After moving to the rightmost cell, it moves to the leftmost cell in the next row below. It skips a cell that cannot be activated and moves to the next cell.

Shift+Tab

These keys move the active cell to the left. After moving to the leftmost cell, it moves to the rightmost cell in the upper row. It skips a cell that cannot be activated and moves to the next cell.

3.6.5.2 Selecting Cells

Using the mouse can select contiguous plural cells as described below.

Cell range

Click the cell in the upper left corner of the region to be selected and drag to the cell in the lower right corner.

Column

When selecting a column, click the column header of the column to be selected. When selecting contiguous plural columns, drag along the column header.

Row

When selecting a row, click the row header of the row to be selected. When selecting contiguous plural rows, drag along the row header.

Whole table

Click the upper left cell of the header. Then all of the rows entered on the table are selected.

3.6.6 Copy and Paste

Numeric values and characters on the MRT worksheet and summary table can be copied to the clipboard. Also text data can be pasted from the clipboard only to the sample ID column in the MRT worksheet.

3.6.6.1 Copy

Select an area on the worksheet to be copied using the mouse and select [Edit]-[Copy] menu or press [Ctrl] + [C] key. Then the values in the selected area are copied to the clipboard. These copied values can be pasted as text data to the word processor or spreadsheet software.

NOTE

- The contents of column header and row header are not copied.
- · Graph column is never copied.
- Hidden column is not copied.
- Hidden row included in the selected area is always copied.

3.6.6.2 Paste

Text data can be pasted from the clipboard to the sample ID column.

Copy & Paste of Sample ID between MRT worksheets

 Select a cell area of sample ID as the copy source and copy it to the clipboard by [Edit]-[Copy] menu or [Ctrl] + [C] key.

If hidden rows are included in the selected area, the contents of the sample ID cells in the hidden rows are also copied. Therefore, set all the rows displayed and check the contents to be copied before executing the copy.

- (2) Click the top cell of the sample ID cell area in the paste destination. Note that paste is not applied to the rows already measured or rows under repeat measurement.
- (3) Select [Edit]-[Paste] menu or press [Ctrl] + [V] key in this status. Then the sample ID is overwritten on the cells from the top cell to down.

(4) When the number of sample ID in the copy source is over the number of rows in the paste destination, the excess are added as new rows.

NOTE

- If a value other than numeric value is pasted to the items for which should be set in the sample ID field (WAIT, AUTO ZERO or RINSE at the use of ASC), it is reset to "0". Numeric values out of the acceptable entry range are also ignored.
- If the paste is applied to the items for which nothing can be entered in sample ID field (CAL-CHK, etc.), it is ignored.
- Also, sample IDs created using word processor or spreadsheet software can be pasted to the MRT worksheet. When creating sample IDs using a word processor, insert a line feed between the sample IDs. Also, the sample ID must not contain the tab code.

3.6.7 Changing Column Width and Column Header Height of MRT Worksheet

The column width for an arbitrary column and the column header height can be changed in the MRT worksheet.

Procedure for setting the column width

- (1) Position the mouse cursor on the column borderline in the column header. The mouse shape changes to a two-direction arrow.
- (2) While the mouse cursor in the two-direction arrow status, press the left mouse button down and drag to the right or left. The cell width of the column header is changed.
- (3) Release the left mouse button at the appropriate position. The column width is set at the position.

Procedure for setting the column header height

- (1) Position the mouse cursor on the bottom line of the leftmost cell in the column header. The mouse shape changes to a two-direction arrow.
- (2) While the mouse cursor is in the two-direction arrow status, press the left mouse button down and drag upward or downward. The height of the leftmost cell is changed.
- (3) Release the left mouse button at the appropriate position. The column header height is set at the position.

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Chapter 4 Measurement Procedures

After completing all the settings, prepare the samples. Perform the measurement following the procedure indicated on the MRT work sheet. The current row is highlighted and its [Action] field shows the type of measurement. When using the ASC, set the samples in proper positions.

If no setting is made on the MRT work sheet, a message appears to indicate that no schedule exists. In this case, select [Parameters]-[Schedule Creation Wizard] from the menu and set the measurement procedures.

NOTE

If a menu or dialog box is kept opened during the measurement operation, the measurement operation may be interrupted. Be careful about it when checking the setup parameters during the operation.

CONTENTS

4.1	Measurement Operation (for Flame Method)	4-2
4.2	Measurement Operation (for Furnace Method)	4-5
4.3	Completing the Measurement	4-6
4.4	Saving and Printing the Data	4-7
4.5	Igniting and Extinguishing the Flame	4-10
4.6	Standard Addition Method and Simple Standard Addition Method	4-19
4.7	Conditions and Operation for Flame Emission Analysis	
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4.1

In the case of flame methods (Flame Continuous Method and Flame Micro Sampling Method), read 4.5 "Igniting and Extinguishing the Flame" thoroughly before starting the measurement. Confirm that the gases are supplied at correct pressures and the burner head is mounted properly. If the lamp warm-up time (unit: minute) is set, you may not start the measurement during the time after lighting on the lamp.

4.1.1 Manual Measurement

(1) Insert the sampling tube to the nebulizer and then ignite the flame.

Press the PURGE button and the IGNITE button on the front of the AA main unit to ignite the flame.

(2) Spray distilled water (or solvent).

Wait 30 seconds after igniting the flame and then put the sampling tube into the distilled water (or solvent) and start spraying. Spray for more than 5 minutes and then proceed to the next step.

(3) Auto Zero

Check that the absorbance signal has stabilized and then click [AUTOZERO] at the bottom of the Main window (or press the [F3] key).

- 1. Put the sampling tube into distilled water (or solvent) and spray it.
- 2. When the signal of real time graph is stabilized, click on [AUTO ZERO] (or [F3] key) at the bottom of the main window.

NOTE

If the "AUTO ZERO" line has already been set in the MRT worksheet, press [START] (or the [F5]/[F6] key).

- (4) Blank Measurement
 - 1. If necessary, spray the blank sample and click on [BLANK] at the bottom of the Main window (or press the [F4] key). The "BLANK" row is inserted on the MRT work sheet and the measurement result is displayed.

- When the "BLANK" row is prepared on the MRT worksheet beforehand, press [START] (or [F5]/[F6] key).
- If the standard or unknown samples are measured after executing the blank measurement, their
 measured values (absorbance or energy) are indicated after the measured blank value is subtracted
 from their actual measured values. The difference between this measured blank value and the actual
 measured standard or unknown sample value is displayed in [Abs] (or [Energy]) on the MRT until the
 next blank measurement is executed.
- (5) Standard Measurement
 - The standard samples are sprayed in the order of concentrations entered on the STD rows of the MRT worksheet. Click on [START] (or press the [F5] or [F6] key) located at the bottom of the main window.
 - 2. Carry out the measurement while checking that the sprayed sample corresponds to the sample on the current row of the window.
 - 3. If the repeat measurement is selected, clicking on [START] inserts a new row for repeat measurement according to the set repeat parameters. Repeat the measurement following the row indication.
- (6) Checking the Calibration Curve
 - 1. After measuring the standard samples, check the calibration curve displayed on the right upper of the window (Fig. 3.78 "Main Window" (3).

- 2. If the calibration curve graph display is too small, set the mouse cursor onto the border of the calibration curve graph to indicate an arrow with two directions. Drag it to specify the range ("drag" means moving the mouse while holding down the left mouse button).
- 3. If the calibration curve is correctly made, proceed to the measurement of unknown samples.
- 4. If you want to change the order or other parameters for calibration curve, click on [Type] above the calibration curve graph then make a change on the [Change Calibration Curve Condition] dialog box.
- (7) Unknown Sample Measurement
 - 1. Spray the unknown sample. Click on [START] at the bottom of the Main window (or press the [F5] or [F6] key).
 - 2. If the repeat measurement is selected, clicking on [START] inserts a new row for repeat measurement according to the set repeat parameters.
 - 3. Repeat the measurement following the row indication. Then continue to measure the unknown samples according to the specified order. Measure an unknown sample while checking that it accords with the current row on the window.

NOTE

If [START] key is pressed when all the rows on the MRT work sheet have been executed, a new "UNK" row (unknown sample row) is added and its measurement result is displayed.

When the "Auto Blank" is ticked in the [Calibration Curve Setup] page, a blank measurement is inserted in the specified interval. Spray the blank and measure it.

If you want to make the size of MRT work sheet larger, set the mouse cursor onto the upper border of the table to indicate an arrow with two directions. Press down the mouse left button and drag it to specify the range. More number of rows can be displayed. For details on changing sizes or using a scroll bar, refer to the Windows documentation.

(8) Finishing the Measurement

After finishing the measurement, put the sampling tube in distilled water and spray it for 30 seconds or more. Then take out the sampling tube, and press the EXTINGUISH button on the front of the instrument to extinguish the flame.

4.1.2 Using the ASC

NOTE

When using the ASC, put a check mark in the check box of [Using ASC] in the [Sequence] page of [Edit parameters] property sheet.

When using the ASC, verify that the position of each actual sample on the ASC turntable accords with the setting on the MRT work sheet.

(1) Set a "RINSE" row in the MRT work sheet.

Insert "RINSE" into first unexecuted row. Set the rinse time to more than 300 seconds (5 minutes) (maximum of 600 seconds).

(2) Igniting the Flame

Press the PURGE button and IGNITE button on the front of the instrument to ignite the flame.

(3) Starting the Measurement

Click on [START] at the bottom of the Main window (or press the [F5] or [F6] key). The automatic measurement is started with the ASC.

(4) Extinguishing the Flame

After finishing the measurement, press the EXTINGUISH button on the front of the AA main unit to extinguish the flame (A flame is automatically extinguished if the automatic flame extinction is selected).

(5) Checking the MRT work sheet

Check the measured results on the MRT work sheet. If a remeasurement is necessary, refer to the section 3.6 "Operating the MRT Work Sheet". When printing or saving the data, proceed to the section 4.4 "Saving and Printing the Data".

NOTE

When performing measurement in the flame method, you might find the absorbance value displayed as "-1". This tends to occur in case that there is strong emission of light from flame, such as $N_2O-C_2H_2$ flame or flame in spraying the sample such as Na, K, etc., although it depends on a target element, sample concentration and flame condition.

If such is the case, carry out countermeasures in order as indicated below.

Note that if "-1" absorbance value is no longer displayed after spraying the sample, start measurement without taking other countermeasures.

- In BGC-D2 mode, confirm whether the hollow cathode lamp beam and D2 lamp beam meat at the burner. If not, adjust D2 lamp position in accordance with 8.8.2 "Replacing Procedures of Deuterium Lamp".
- Perform the line search and beam balance while spraying the blank sample.
- Perform the line search and beam balance while spraying the standard sample with the highest concentration. Then, execute AUTO ZERO while spraying the blank sample.

If the concentration of the standard sample is too high, signal becomes noisy in measuring the low-concentration sample.

• Reduce fuel gas flow rate or change the burner angle, and perform the line search and beam balance. Note that this procedure results in deteriorating the sensitivity.

4.2.1 Preparation and Manual Measurement

- (1) Mount the graphite tube correctly.
- (2) Verify that the argon gas is supplied at the specified pressure, and flow the cooling water.
- (3) Verify that both the power switch and circuit protector located on the power supply unit of the GFA-7000A are turned ON.
- (4) For the manual measurement, temporarily click on the [START] button to calibrate the temperature before starting the measurement.

NOTE

Note that heating is performed when the temperature calibration is executed.

- (5) After the temperature calibration has been finished, the message "Inject a sample." should appear. Using a pipette or the like, inject the sample into the graphite tube.
- (6) After the sample has been injected, click on the [OK] button in the message dialog box. The measurement will be started.

4.2.2 Using the ASC

When using the ASC, verify that the position of each actual sample on the ASC turntable accords with the setting on the MRT work sheet.

(1) Checking the ASC Nozzle Position

Check that the nozzle of the ASC has been properly positioned. To position the nozzle of the ASC, select [Instrument] - [Furnace Nozzle Position] from the menu. For further details, refer to the HELP information in the WizAArd software.

(2) Starting the Measurement

Click on [START] at the bottom of the Main window (or press the F5 or F6 key). The automatic measurement is started with the ASC. Before starting the measurement, the temperature calibration is executed. Note that heating is performed when the temperature calibration is executed.

(3) Finishing the Measurement

When the last row of the MRT work sheet is finished, the instrument becomes in the waiting condition.

(4) Checking the MRT work sheet

Check the measured results on the MRT work sheet. If a remeasurement is necessary, refer to the section 3.6 "Operating the MRT Work Sheet". When printing or saving the data, proceed to the section 4.4 "Saving and Printing the Data".

Completing the Measurement

- (1) When the measurement is completed, verify that the flame is extinguished and tighten the main valve of the gas cylinder and compressor in the case of flame method. Close the main valves of the cooling water and gas in the case of furnace method.
- (2) Quit the software. For example, select [File]-[Exit] from the menu bar.

NOTE

The communication with the instrument is shut off first, and then the main window is closed. It takes approximately 30 seconds.

- (3) Turn off the power switch of AA main unit.
- (4) Turn off the power switches for peripheral equipments such as the ASC and GFA.

CAUTION

Always turn OFF the GFA circuit protector after use.

If the circuit protector is left ON, the GFA may be damaged if a problem occurs with the power supply.

4.4

4.4.1 Saving the Data

When all measurements are completed, save the data.

(1) Select [File]-[Save As] from the menu bar, and enter the file name in the [Save As] dialog box. File extension is limited to ".aa".

ave As			? ×
Savejn: 🔂	WizAArd	→ 🗢 Ē) (* 10-
File <u>n</u> ame:	TEST.aa		Save
	Data File(*.aa)		Cancel
	• * *	_	<u>C</u> omment

Fig. 4.1 [Save As] Dialog Box

NOTE

A measurement data whose repeat measurement has not finished is not saved in the file. If the data is necessary although the repeat measurement cannot be completed, print it out before ending the AA software.

4.4.2 Printing the Data

- (1) Select [File] [Print Data/Parameters] or [Print Table Data] from the menu bar. The [Select Schedule] dialog box will appear.
- (2) Click on the elements ("Summary Report" is also available for [Print Table Data]) to highlight it. Then click on [OK]. The [Print] dialog box will appear.

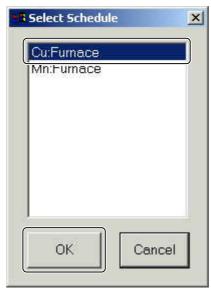


Fig. 4.2 [Select Schedule] Dialog Box

(3) After the print settings have been completed, click on [OK].

Printer —	HP LaserJet 4P	Properties
<u>N</u> ame:	•	
Status:	Ready	
Туре:	HP LaserJet 4P	
Where:	LPT1:	
Commen	t.	🥅 Print to file
Print rang	e	Copies
		Number of <u>c</u> opies: 1 🚊
C Page	s from to:	
C <u>S</u> elec	stian	

Fig. 4.3 [Print] Dialog Box

Printing will be executed.

[Print Data/Parameters]

The measurement parameters and measured data are printed.

Select Schedul	e 💌
Cu:Furnace	
Mn:Furnace	
1	
ок	Cancel
	Cancer

Fig. 4.4 [Select Schedule] Dialog Box

[Print Table Data]

The current MRT work sheet is printed.

(1) Select or change the items to be printed by selecting [File]-[Print Style]-[Table Show/Hide] page from the menu bar.

In the case of [Summary Report], the columns shown in the MRT work sheet on the window are printed.

Summan Cu:Furna Mn:Furna	ce	

Fig. 4.5 [Select Schedule] Dialog Box

4.5

4.5.1 Operation Locations

Locations associated with igniting and extinguishing of the flame are described here. Firstly, the IGNITE button, PURGE button, EXTINGUISH button and BURNER SELECT switch are located on the front side of the AA main unit.

4.5.2 Safety Precautions Prior to Ignition

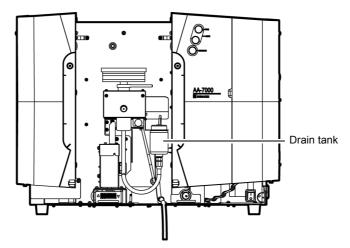
- (1) Switch ON the power for the fan of the room exhaust duct.
- (2) Verify that the gas to be used is appropriate and that the gas pressure is set correctly.

Gas type	Supply pi	ressure
Fuel gas	Acetylene (C ₂ H ₂)	0.09 MPa ± 0.01 MPa
Support gas	Air, Nitrous oxide (N ₂ O)	0.35 MPa ± 0.03 MPa

The above supply pressure must be kept during flame combustion.

(3) Confirm visually that the drain tank is filled with water.

When supplying water to the drain tank, refer to 2.8 "Supplying Water to the Drain Tank (AA-7000F, AA-7000F/AAC)".



If the front panel and chimney have been removed, mount them as they were originally.

For details, see 2.4 "Removing and Mounting the Front Panel" and 2.5 "Removing and Mounting the Chimney (AA-7000F, AA-7000F/AAC)".

- (4) Verify that there are no leaks in any of the gas lines (Refer to the section 8.5 "Checking for Gas Leaks (AA-7000F, AA-7000F/AAC)" on the Chapter 8 "Maintenance").
- (5) Verify that the flame and burner head to be used are appropriately matched.

Burner head	Slot length	Usable flame
Standard burner head	10 cm	Air-acetylene (C ₂ H ₂)
High temperature burner head (optional)	5 cm	Air-acetylene (C_2H_2) Nitrous oxide (N_2O) -acetylene (C_2H_2)

Table 4.2 Type of Burner Heads and Usable Flames

4.5.3 Igniting and Extinguishing Air-C₂H₂ Flame

(1) Specify the following values for the fuel gas flow rate. To set these rates, click on the [Gas Flow Rate] radio button in the [Atomizer/Gas Flow Rate Setup] page and use the [Increase Fuel] and [Decrease Fuel] buttons.

Gas type	Gas flow rate
Fuel gas (C ₂ H ₂)	2.0 (L/min)

NOTE

The flow rate of support gas is fixed (at 15 L/min).

If the flow meter kit (optional) is installed, set the flow rate of the support gas to the value indicated below.

Gas type	Gas flow rate
Support gas (Air)	15.0 (L/min)

(2) Press the two ignition buttons (PURGE and IGNITE) simultaneously.

- 1. Keep pressing the IGNITE button until the flame is completely ignited. If the flame does not ignite even if the buttons are pressed for ten seconds or more, the igniting operation is automatically stopped.
- 2. Retry after releasing the buttons for ten seconds or more.

NOTE

- If ignition fails, the AA software may display a message such as "Flame has been extinguished". If such a message is displayed, the AA instrument is locked so that ignition is not possible. To release this lock, close all of the error messages.
- If you cannot ignite the flame, see 9.1 "Failure to Ignite".
- (3) After the flame is ignited, confirm the settings of the fuel gas pressure and flow rate, and support gas pressure.

NOTE

If the flow meter kit (optional) is installed, you can also check the setting for the support gas flow rate.

(4) To extinguish the flame, press the EXTINGUISH button.

- After the flame has been extinguished a gas leakage inspection is automatically started, but the flame can be re-ignited by pressing the IGNITE and PURGE buttons.
- If the gas pressure decreases while the flame is burning, the flame is automatically extinguished. In that case, check the gas supply pressure before igniting the flame again.
- If a momentary AC supply power outage should occur, the flame is automatically extinguished.
- When the following error messages are displayed on the AA software, the AA main unit is locked so that the ignition cannot be done. To release this lock, solve the problems in the instrument settings then close all the error messages displayed on the software. While the error message is displayed, the ignition cannot be done.
 - a. Fuel gas pressure is too low.
 - b. Support gas pressure is too low.
 - c. Flame has been extinguished.
 - d. Drain tank water level is too low.
 - e. Momentary electric shutdown occurred in the gas controller.
 - f. Vibration was detected by the instrument.
- If the flame should not be extinguished even by pressing the EXTINGUISH button, do NOT panic and take action of following operation.
 - 1. Turn off the main unit POWER switch (on the right side of the main unit). This operation mechanically closes the solenoid valve of the gas controller, shutting off the gas supply safely.
 - 2. Close the gas main valve.
 - 3. Contact your service representative immediately. Do NOT operate the instrument until the repair by service personnel completes.

4.5.4 Igniting and Extinguishing N₂O-C₂H₂ Flame

- (1) Take off the standard burner head and mount the high temperature burner head supplied as an optional accessory.
- (2) Insert the burner recognizing key connected to the high temperature burner head with a wire into the hole of the BURNER SELECT switch, and turn it to the N₂O-C₂H₂ position.
- (3) Click on the [Gas Flow Rate] radio button in the [Atomizer/Gas Flow Rate Setup] page, select [N₂O-C₂H₂] in [Flame Type], and then specify the following values for the fuel gas flow rate. To set these rates, use the [Increase Fuel] and [Decrease Fuel] buttons.

Gas type	Gas flow rate
Fuel gas (C ₂ H ₂)	7.0 (L/min)

NOTE

The flow rate of support gas is fixed (at 11 L/min).

If the flow meter kit (optional) is installed, set the flow rate of the support gas to the value indicated below.

Gas type	Gas flow rate
Support gas (N ₂ O)	11.0 (L/min)

(4) Press the ignition buttons (PURGE and IGNITE) at the same time to ignite the flame.

First, the Air- C_2H_2 flame will be ignited.

Approx. 10 seconds after the flame ignition is achieved, the C_2H_2 flow rate is automatically increased 5.0 L/ min and the flame emission intensity is increased. Then the support gas is automatically switched from Air to N₂O, changing the Air-C₂H₂ flame to N₂O-C₂H₂ flame.

- 1. Keep pressing the IGNITE button until the flame is completely ignited. If the flame does not ignite even if the buttons are pressed for ten seconds or more, the igniting operation is automatically stopped.
- 2. Retry after releasing the buttons for ten seconds or more.

- If ignition fails, the AA software may display a message such as "Flame has been extinguished". If such a message is displayed, the AA instrument is locked so that ignition is not possible. To release this lock, close all of the error messages.
- Even if the [Flame Type] is set to N₂O-C₂H₂, the flame will not be switched unless the BURNER SELECT switch is set to N₂O-C₂H₂ with a key attached to the high temperature burner head. In this case, if the BURNER SELECT switch is changed from the Air-C₂H₂ to the N₂O-C₂H₂ position while the Air-C₂H₂ flame is combusting, the support gas is automatically switched from Air to N₂O, changing the Air-C₂H₂ flame to N₂O-C₂H₂ flame.
- If you cannot ignite the flame, see 9.1 "Failure to Ignite".

(5) After the flame is ignited, confirm the settings of the fuel gas pressure and flow rate, and support gas pressure.

NOTE

If the flow meter kit (option) is installed, you can also check the setting for the support gas flow rate.

(6) To extinguish the flame, press the EXTINGUISH button. The support gas is automatically switched from N₂O to Air, changing the N₂O-C₂H₂ flame to Air-C₂H₂ flame. Then the flame is extinguished.

- After the flame has been extinguished a gas leakage inspection is automatically started, but the flame can be re-ignited by pressing the IGNITE and PURGE buttons.
- If the gas pressure decreases while the flame is burning, the flame is automatically extinguished. In that case, check the gas supply pressure before igniting the flame again.
- If a momentary AC supply power outage should occur, the flame is automatically extinguished.
- When the following error messages are displayed on the AA software, the AA main unit is locked so that the ignition cannot be done. To release this lock, solve the problems in the instrument settings then close all the error messages displayed on the software. While the error message is displayed, the ignition cannot be done.
 - a. Fuel gas pressure is too low.
 - b. Support gas pressure is too low.
 - c. Flame has been extinguished.
 - d. Drain tank water level is too low.
 - e. Momentary electric shutdown occurred in the gas controller.
 - f. Vibration was detected by the instrument.
- If the flame should not be extinguished even by pressing the EXTINGUISH button, do NOT panic and take action of following operation.
 - 1. Turn off the main unit POWER switch (on the right side of the main unit). This operation mechanically closes the solenoid valve of the gas controller, shutting off the gas supply safely.
 - 2. Close the gas main valve.
 - Contact your service representative immediately. Do NOT operate the instrument until the repair by service personnel completes.

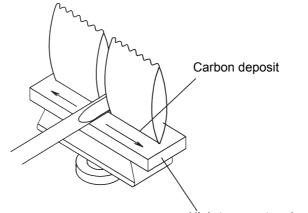
CAUTION

- 1. Always use high temperature burner head when using a nitrous oxide-acetylene flame. Using the standard burner head with this gas causes a danger of flashback.
- 2. When the acetylene flow rate is high, carbon is deposited around the burner slot. When this occurs, use the provided large screwdriver to scrap off the carbon deposit while taking care about the following points. Insert the screwdriver at the center on the burner slot while keeping the top portion of the screwdriver lengthwise. Don't close a large part of the burner slot by keeping the top portion of screwdriver horizontally.

While keeping the top portion of screwdriver lengthwise, slide it from the center to the outside (right or left side) and scrap off the carbon deposit gently. To prevent the flame from going out, don't slide the screwdriver from the outside to the center. Insert it at the center again and scrap off to the outside.

Be careful not to scratch the burner head and slot.

Remove the carbon deposit inside the burner slot using a piece of hard paper, after the flame is extinguished and the burner is completely cooled.

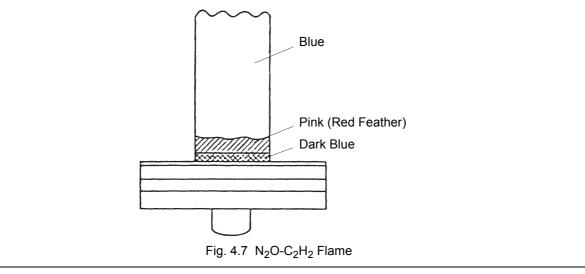


High-temperature burner head

Fig. 4.6 Removing Carbon Deposit in N₂O-C₂H₂ Flame

3. When using a nitrous oxide-acetylene, decreasing the acetylene or increasing the nitrous oxide will cause the pink portion of the flame (red feather) to gradually decrease. If this decreases to less than 2 mm, the flame will split, and if the acetylene flow is further reduced, a flashback may occur.

Therefore, control the gas flow rate so that the red feather does not become shorter than 2 mm. Under the standard conditions, the red feather is approximately 8 mm.



CAUTION

If flashback should occur, perform the checks in accordance with "Measures When Flashback Occurs" on the yellow pages.

4.5.5 Flame Conditions When Analyzing Organic Solvent Samples

WARNING

- Check that the parts of the atomizer are resistant to the chemicals used.
 - See 10.1.2 "Flame Specifications".

Since an organic solvent itself will burn in the flame, the flame will not be completely combusted using the normal flame conditions, as the result, the measurement accuracy will be decreased. To perform an accurate measurement, it is necessary to decrease the sample spray amount as well as decrease the acetylene gas flow rate, compared with the aqueous solution. The procedure for setting the flame conditions when using organic solvent is as follows.

- (1) Replace the sampling tube with the provided polyethylene capillary tube.
- (2) While solvent is being sprayed into the flame, decrease the acetylene gas flow rate so that the color of the flame is from blue or slightly red (in the case of air-acetylene flame).
- (3) While organic sample is being sprayed, try various acetylene flow rates and burner heights until the maximum S/N ratio (signal to noise ratio) is achieved.

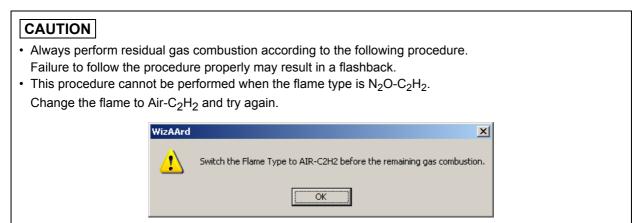
NOTE

Overly decreasing the acetylene flow may cause the flame to go out when spraying of the organic sample is stopped. In this situation, increase the acetylene gas flow rate in 0.1 L/min increments until the flame no longer goes out at the end of spraying the organic solvent sample, and then adjust the flame condition by increasing the support gas flow rate. To use flame of $N_2O-C_2H_2$, ensure that the pink portion of the flame (red feather) does not become shorter than 2 mm even if solvent spraying is stopped.

4.5.6 Residual Gas Combustion

Complete combustion of any acetylene gas in the gas piping between the gas main value and the instrument allows it to be discharged safely.

Although residual gas does not usually require complete combustion, a Shimadzu service representative may burn any residual gas before replacing the gas hose.



- (1) Set the correct supply pressure for the acetylene gas and air.
- (2) With flame measurement selected for the current measurement element in the AA software, select [Parameters] [Edit Parameters] from the menu bar and open the [Atomizer/Gas Flow Rate Setup] page.
- (3) Select [Gas Flow Rate] under [Operation Object] and select "Air-C₂H₂" for [Flame Type].
- (4) Click [OK] to close the page.
- (5) When using the high-temperature burner, set the burner selection keyswitch (BURNER SELECT) on the body of the AA instrument to the Air-C₂H₂ position.
- (6) Press the two ignition buttons (PURGE and IGNITE) simultaneously to ignite the Air- C_2H_2 flame.
- (7) Select [Instrument] [Remaining Gas Combustion] from the menu bar.
- (8) Check the displayed message and click [OK].

WizAArd	×
1	Please refer to the manual to operate the remaining gas combustion safely.

(9) If the following message is displayed, close the acetylene gas main value and click [OK]. The acetylene gas is fully discharged and the flame blows out.

WizAArd	×
1	Stop acetylene (fuel gas) supply.
	OK Cancel

NOTE

- Continue to supply support gas (air) without stopping it.
- To stop the residual gas combustion process, leave the main valve of the acetylene gas cylinder open and click [Cancel].

(10) Press the EXTINGUISH button when the following message is displayed.

Support gas (air) is flowing from the burner head after the flame blows out. Press the EXTINGUISH button to stop the support gas.



(11) Click [OK].

This completes the residual gas combustion procedure.

NOTE

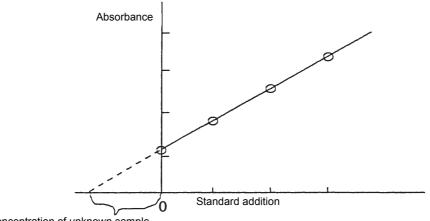
• To discharge the support gas, after extinction, close the support gas main value and press the PURGE button.

To discharge nitrous oxide gas, after extinction, set [Flame Type] to $N_2O-C_2H_2$ in the AA software, set the burner selection keyswitch (BURNER SELECT) on the body of the AA instrument to the N2O-C2H2 position, and then press the PURGE button.

Each purge is automatically stopped after a maximum of 10 seconds. If the gas is not completely ٠ discharged by a single purge, repeat the purge process several times.

Standard Addition Method and Simple Standard Addition Method

The standard addition method is used when there is interference by coexistent material (matrix) in the sample and its influence is given to the measured value. For the standard addition method, equal volumes of unknown sample solution are prepared, and a standard solution of different but known concentration is added to each of the unknown sample solutions. The absorbance is measured for each of these samples, and a calibration curve is created. The unknown sample concentration is obtained from the point at which the extended calibration curve intersects with the horizontal axis.

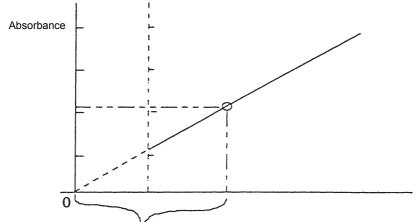


Concentration of unknown sample

4.6

Fig. 4.8 Standard Addition Method

When the coexistent material or matrix of plural unknown samples are similar, measure one of them by standard addition method, and you can use the slope of the same calibration curve to determine the concentrations of other unknown samples. This is called simple standard addition method.



Sample concentration by simple standard addition method

Fig. 4.9 Simple Standard Addition Method

NOTE

• "Auto Dilution and Remeasurement" is not available in the Standard Addition Method and Simple Standard Addition Method.

4.6.1 Setting the [Preparation Parameters] Page

The measurement procedure for the standard addition method can be set up using the wizard as in the case of the calibration curve method. Start the [Element Selection Wizard] or the [Schedule Creation Wizard] and complete the [Calibration Curve Setup] and [Sample Group Setup] in the [Preparation Parameters] page.

In the case of flame continuous method

- (1) When the [Preparation Parameters] page is displayed, first click on the row of a desired element to highlight it, and then click on the [Calibration Curve Setup] button. The [Calibration Curve Setup] page will be displayed.
- (2) Tick the Method of Standard Addition option.
 No settings are required for Order and Zero Intercept. The order automatically becomes 1st (linear equation) and the calibration curve does not pass through the origin.
- (3) Select a value for [Conc. Unit] from the list displayed by clicking on the $[\mathbf{V}]$ button.
- (4) Enter a value for [No. of Lines] under the [Measurement Sequence for Calibration Curve] section and click on [Update] button. The table will be displayed with that number of rows. Enter added concentrations for standard samples. Also enter [Sample ID] as necessary.

NOTE

When the ASC is used, the [Pos.] field is displayed, but does not accept input. Leave the field blank.

- (5) Click on the [OK] button in the [Calibration Curve Setup] page. The [Preparation Parameters] page will be displayed.
- (6) Click on the [Sample Group Setup] button. The [Sample Group Setup] page will be opened.
- (7) Enter values for [Weight Factor], [Volume Factor], [Dilution Factor] and [Correction Factor] in the [Weight Correction Factors] section.
- (8) Enter a value in [No. of Samples] under the [Unknown/Spike Measurement Sequence] section and click on [Update] button. The table will be displayed with the corresponding number of rows. Enter [Sample ID] and [WF] as necessary.

NOTE

When the ASC is used, the [Pos.] field is also displayed. In the standard addition method, there are multiple standard added samples for a single unknown sample. In this field, enter the position of the standard added sample of the smallest added concentration.

(9) After finishing the settings, click on the [OK] button. You will return to the [Preparation Parameters] page.

NOTE

• To measure multiple elements, similarly enter the settings for the other elements.

When the wizard is completed, the measurement procedure specified in the [Preparation Parameters] page is inserted into the MRT worksheet. Check that the measurement procedures for the calibration curves in the same number as that of unknown samples have been created and automatically given C# (calibration curve numbers).

• When the ASC is used, the same value is included in the [Pos.] field for the standard added samples for a single unknown sample. Manually change it to a correct value on the MRT worksheet.

NOTE

In the standard addition method, you may wish to have multiple sample groups in the [Sample Group Setup] page box, but no SG# (sample group number) can be given to the actual sample information.

In the case of flame micro sampling method and furnace method

(1) When the [Preparation Parameters] page is displayed, first click on the row of a desired element to highlight it, and then click on the [Calibration Curve Setup] button.

The [Calibration Curve Setup] page will be displayed.

- (2) Tick the Method of Standard Addition option. No settings are required for Order and Zero Intercept. The order automatically becomes 1st (linear equation) and the calibration curve does not pass through the origin.
- (3) Select a value for [Conc. Unit] from the list displayed by clicking on the [▼] button.
- (4) Enter a value for [No. of Lines] under the [Measurement Sequence for Calibration Curve] section and click on [Update] button. The table will be displayed with that number of rows. Enter added concentrations for standard samples. Also enter [Sample ID] as necessary.

NOTE

When the ASC is used, the [Pos.] field is displayed, but does not accept input. Leave the field blank.

- (5) To automatically add standard samples to mix them using the ASC, enter data in the [VOL], [Diluent], ..., [Reagent 3] fields. Specify each standard sample as [Reagent 3].
- (6) Click on the [OK] button in the [Calibration Curve Setup] page. The [Preparation Parameters] page will be displayed. Click on the [Sample Group Setup] button. The [Sample Group Setup] page will be opened.
- (7) Enter values for [Weight Factor], [Volume Factor], [Dilution Factor] and [Correction Factor] in the [Weight Correction Factors] section.

In the standard addition method, the settings for [Unknown/Spike Preparation Parameters] are irrelevant. Leave them the default values.

- (8) Enter a value in [No. of Samples] under the [Unknown/Spike Measurement Sequence] section and click on [Update] button. The table will be displayed with the corresponding number of rows. Enter [Sample ID] and [WF] as necessary. When the ASC is used, also enter a correct value in the [Pos.] field.
- (9) After finishing the settings, click on the [OK] button. You will return to the [Preparation Parameters] page.

NOTE

- To measure multiple elements, similarly enter the settings for the other elements.
- When the wizard is completed, the measurement procedure specified in the [Preparation Parameters] page is inserted into the MRT worksheet. Check that the measurement procedures for the calibration curves in the same number as that of unknown samples have been created and automatically given C# (calibration curve numbers).
- When standard added samples are previously supplied, i.e., when no standard samples are
 automatically mixed with the ASC, manually change the value in the [Pos.] field on the MRT worksheet
 as described in the above paragraph "In the case of flame continuous method".

NOTE

In the standard addition method, you may wish to have multiple sample groups in the [Sample Group Setup] page box, but no SG# (sample group number) can be given to the actual sample information.

In the case of simple standard addition method

In any of the flame continuous method, flame micro sampling method and furnace method, first set up the measurement procedure for the standard addition method.

- (1) Tick the Method of Standard Addition option in the [Calibration Curve Setup] page. Similarly enter the other settings as in the standard addition method.
- (2) Enter "1" in the [No. of Sample] field under the [Unknown/Spike Measurement Sequence] section in the [Sample Group Setup] page and then click on [Update] button. The table will be displayed with one row. Enter the same settings as in the standard addition method. When the wizard is completed, the measurement procedure for only one calibration curve is inserted into the MRT worksheet.
- (3) Add the unknown sample that is to be measured by the simple standard addition method to the MRT worksheet. Enter "SMSA" as [Action] on the row next to the last row.

NOTE

The C# (calibration curve number) that is automatically given here is increased by 1 from the C# of the previous calibration curve. However, manually change it to the C# of the previous calibration curve. Repeat the operations for "SMSA" settings by the number of unknown samples.

(4) After the settings for [Action] have been finished, enter data in the [Pos.], [Weight Factor], [Volume Factor], [Dilution Factor], [Correction Factor] etc. The input operation will become easier if you drag the cursor on the MRT worksheet to define a range, click on the right mouse button, and then use the [Collective Edit] function.

NOTE

In the simple standard addition method, no SG# (sample group number) can be given to the sample information.

Example

In this example, the ASC is used to add the standard samples of 0 ppb, 10 ppb, 20 ppb and 30 ppb to the unknown samples, which are being diluted 5 times. Standard sample of 100 ppb is prepared for addition and is regarded as the Reagent 3.

As other conditions, assume that [Mixing] is to be performed; [Injection Volume] is 20 μ L, and [No. of repetition] is 3 ([Max No. of repetition] is 5).

Conc	Sample	Diluent	Reagent 1	Reagent 2	Reagent 3	Total Volume
0	40	160	0	0	0	200
10	40	140	0	0	20	200
20	40	120	0	0	40	200
30	40	100	0	0	60	200

The [Sample] must be constant for all the concentrations. Here, assume it is 40 (μ L).

This [Total Vol] must satisfy the following condition.

[(Injection Volume) × (Max. No. of repetition) × (No. of Boost Cycles) + 50μ L] \leq [Total Volume] $\leq 600 (\mu$ L) In this equation, the [Injection Volume] means the [Volume] set in the [Mixing Setup] dialog box. The "50 μ L" indicates the dead volume of mixing port. (If the furnace boost cycle is not performed, No. of Boost Cycles is 1.)

Based on settings made up to now, the calculation is as follows: $[(20 \ \mu L) \times (5 \ times) \times (1 \ cycle) + 50 \ \mu L] \leq [200 \ \mu L] \leq 600 \ (\mu L)$ This indicates that the settings are correct.

Assume that there are unknown samples B and C, which have similar matrix components to that of unknown sample A in the above setup example. These concentrations are calculated using the calibration curve created by unknown sample A (simple standard addition method). In this case, since the [Sample] is 40 μ L and the [Total Vol] is 200 μ L, the [Unknown Sample] preparation parameters are set as below.

Sample	Diluent	Reagent 1	Reagent 2	Reagent 3	Total Volume
40	160	0	0	0	200

In the [Weight Correction Factors] section, enter 40 (μ L) in [Weight Factor] and 200 (μ L) in [Volume Factor]. Then the actual concentration before dilution can be calculated.

4.6.2 Measurement Procedures on MRT Work Sheet

The indication of [Action] on the MRT worksheet related to "SMSA (Simple Method of Standard Addition)" and "MSA (Method of Standard Addition)" is described below.

- MSA : Indicates standard addition method.
- MSA-RES : Indicates the concentration of unknown sample calculated from the result of measuring a set of standard addition samples.
- SMSA : Indicates simple standard addition method. Unknown sample is measured using a calibration curve created by another sample for standard addition method.

When samples A, B, and C are being measured by the standard addition method, three calibration curves will be created. When MSA is added to the MRT worksheet, C# (calibration curve numbers) are allocated automatically. Check that C# correctly correspond to the series of standard samples in the standard addition method.

Order	[Action]	Measurement	C#
1	MSA	(Measures Sample A with standard 0 ppb addition)	01
2	MSA	(Measures Sample A with standard 10 ppb addition)	01
3	MSA	(Measures Sample A with standard 20 ppb addition)	01
4	MSA	(Measures Sample A with standard 30 ppb addition)	01
5	MSA-RES	(Indicates concentration result of Sample A)	01
6	MSA	(Measures Sample B with standard 0 ppb addition)	02
7	MSA	(Measures Sample B with standard 10 ppb addition)	02
8	MSA	(Measures Sample B with standard 20 ppb addition)	02
9	MSA	(Measures Sample B with standard 30 ppb addition)	02
10	MSA-RES	(Indicates concentration result of Sample B)	02
11	MSA	(Measures Sample C with standard 0 ppb addition)	03
12	MSA	(Measures Sample C with standard 10 ppb addition)	03
13	MSA	(Measures Sample C with standard 20 ppb addition)	03
14	MSA	(Measures Sample C with standard 30 ppb addition)	03
15	MSA-RES	(Indicates concentration result of Sample C)	03

Table 4.3 Measurement sequence for standard addition method

Assume that there are unknown samples B and C that contain similar components to those of unknown sample A. To obtain their concentrations from the calibration created with unknown sample A, the following measurement sequence is used:

Order	[Action]	Measurement	C#
1	MSA	(Measures Sample A with standard 0 ppb addition)	01
2	MSA	(Measures Sample A with standard 10 ppb addition)	01
3	MSA	(Measures Sample A with standard 20 ppb addition)	01
4	MSA	(Measures Sample A with standard 30 ppb addition)	01
5	MSA-RES	(Indicates concentration result of Sample A)	01
6	SMSA	(Measures Unknown Sample B)	01
7	SMSA	(Measures Unknown Sample C)	01

4.7

Flame emission quantitative analysis is described here. As the case of atomic absorption analysis, the Wizard can be used for setup. However, the setup procedures in the [Optics Parameters] and [Atomizer/Gas Flow Rate Setup] pages are different in some points. Other operations are the same as those of the flame continuous method of atomic absorption analysis.

4.7.1 [Optics Parameters] Page

Optics Parameters		×	
	K		-6
	Socket #: 6 Lamp Pos. Setup If you click on the Lamp Pos. Setup button, you can turn the lamp turret manually and change the lamp. (0 - 600 mA.) Lamp ID: Emission Lamp ON:		-0
	Lamp Status: Line Search is necessary. Warmup Lamp Line Search		_(
	<u>≺B</u> ack <u>N</u> ext> Finish Cancel He	elp	

Fig. 4.10 [Optics Parameters] Page

1 [Wavelength]

For wavelength used for flame emission analysis, refer to Table 4.5. Enter the numeric value if the wavelength is different from that for atomic absorption analysis.

2 [Slit Width]

Normally, this value is 0.2 nm, but use 0.7 nm if there is a problem with baseline drifts.

3 [Lamp Mode]

Set the [Lamp Mode] to "EMISSION".

4 [Socket #]

Although a lamp is not used, it is necessary to set an arbitrary socket number as a dummy. (Procedure for dummy setup)

- 1. Press the [Lamp Pos. Setup] button to display the [Lamp Position Setup] dialog box.
- 2. For an arbitrary [Socket #], select " * " in the [Element] and "Normal" in the [Lamp Type].
- 3. Select [Emission] in [Lamp ID]. This lamp ID is a special ID of dummy lamp for flame emission analysis.
- 4. Press [OK] to close the [Lamp Position Setup] dialog box and return to the [Optics Parameters] page. Then select the socket # in which the dummy lamp for flame emission analysis was set.

When performing the line search using a lamp, set a proper lamp in [Lamp Position Setup] dialog box.

[Lamp Current]

Set the [Low] to "0" (zero). When the lamp mode is set to "EMISSION", the [High] is automatically set to "0" (zero) and cannot be entered.

([ASC Sample Pos. for EMISSION Line Search]

If the sample position of the highest concentration standard sample on the ASC turntable is set, automatic spray using the ASC is possible.

[Line Search]

In the case of flame emission analysis, a line search has to be performed when the flame has been ignited and the standard sample is being sprayed. So, the line search should not be performed at first. Proceed to the next [Atomizer/Gas Flow Rate Setup] page first and set the parameters so that ignition can be possible. After that, return to this page to perform the line search.

It is also possible to perform a line search using the hollow cathode lamp. In this case, light on the hollow cathode lamp to perform the line search. If the lamp is lit on in the EMISSION mode, the lamp is automatically turned off when the [START] key is pressed to start the measurement. Don't forget to turn off the lamp, however, when observing a signal in the [Atomizer/Gas Flow Rate Setup] page while aspirating the sample.

Element	Wave length (nm)
Са	422.7
Cs	852.1
Li	670.8
К	766.5, 769.9
Na	589.0, 589.5
Rb	780.0
Sr	460.7

Table 4.5 Analysis Line Wavelength Table for Flame Emission Analysis

4.7.2 [Atomizer/Gas Flow Rate Setup] Page

(1) Firstly, select the [Gas Flow Rate] for [Operation Object] by clicking on the radio button.

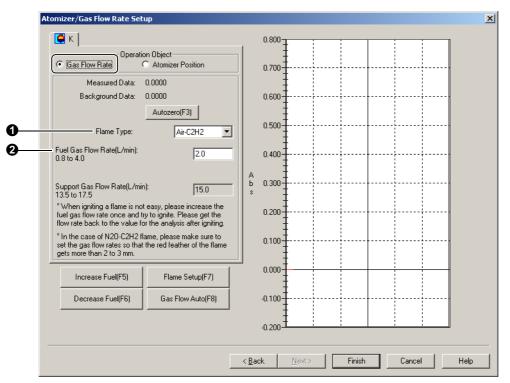


Fig. 4.11 [Atomizer/Gas Flow Rate Setup] Page (in the case of [Gas Flow Rate])

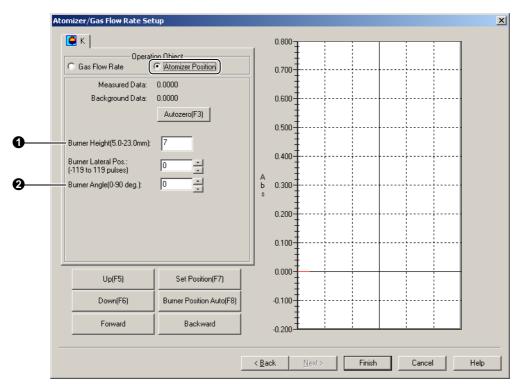
[Flame Type]

Since the flame used for atomic absorption analysis is indicated, click on the $[\Psi]$ and select the flame type used for flame emission analysis. As a flame type used for flame emission analysis, generally, the high temperature N₂O-C₂H₂ flame is good. However, in the cases of alkaline metals (Na, K, etc.), other flame (Air-C₂H₂) is also available for measurement of sufficiently minute quantities.

2 [Fuel Gas Flow Rate]

Since the flow rate for atomic absorption analysis is indicated by default, set the flow rate for flame emission analysis. It is necessary to check the optimum value for each element beforehand. In general, a low acetylene flow rate is best. In the case of flame emission analysis, when using the $N_2O-C_2H_2$ flame, adjust the flow rate so that the red feather (pink portion of flame) is approx. 2 to 3 mm in height.

To obtain the optimum gas flow rate, the function of [Gas Flow Auto] button is convenient. Use it after setting the ignition conditions and performing the line search.



(2) Select the [Atomizer Position] for [Operation Object] by clicking on the radio button.

Fig. 4.12 [Atomizer/Gas Flow Rate Setup] Page (in the case of [Atomizer Position])

1 [Burner Height]

Since the value for atomic absorption analysis is indicated by default, enter the optimum value for flame emission analysis. If the optimum value is not known, as a first step, set it to 7 mm for Air- C_2H_2 and 11 mm for N₂O- C_2H_2 . Then, after spraying the standard sample and performing the line search, obtain the optimum value again.

To obtain the optimum burner position, the function of [Burner Position Auto] button is convenient.

2 [Burner Angle]

After adjusting the angle of the burner head, you can record the reading on the scale.

Generally, the S/N ratio is better when the angle is set at 0 degree for optical axis (parallel) than at 90 degrees, but its linearity may become worse. When the concentration of analysis element is relatively high, the burner head is used at 90 degrees.

4.7.3 Line Search and Beam Balance

After completing the condition setup in the [Atomizer/Gas Flow Rate Setup] page, click on [Back] to return to the [Optics Parameters] page.

Read the section 4.5 "Igniting and Extinguishing the Flame" thoroughly and set the conditions so that a flame can be ignited.

- (1) Ignite the flame.
- (2) Start spraying the standard sample of the highest concentration. If the sample position of the highest concentration standard sample on ASC turntable is set to the [ASC Sample Pos. for EMISSION Line Search], the automatic spray using the ASC is possible.
- (3) While spraying the sample, click on [Line Search] button. Then the line search/beam balance is executed. Continue spraying the sample until the line search/beam balance operation is completely finished.

NOTE

When the ASC is used, the spray of the sample in the specified position on the ASC turntable is started before starting the line search/beam balance. The line search/beam balance is started when the pre-spray time set in the [Parameters]-[Edit Parameters]-[Measurement Parameters] is finished after starting the spray.

- (4) If the line search fails and the message stating insufficient energy is displayed, prepare and spray a standard sample of even higher concentration and try it again.
- (5) When the line search is completed, click on [Close] to return to the [Optics Parameters] page. Then click on [Next] button to proceed to the [Atomizer/Gas Flow Rate Setup] page again. While monitoring the real time graph on the [Atomizer/Gas Flow Rate Setup] page, spray distilled water and check that the signal becomes almost zero.

NOTE

If the signal does not become small, it is possible that the analysis line was not correctly detected. In that case, go back to the [Optics Parameters] page and perform the line search once again following the procedures (1) to (5).

4.7.4 Wavelength Shift

Wavelength shift is a function that relates to flame photometry test methods that comply with ISO 6353-1, Reagents for chemical analysis - Part 1 : General test method, when performing flame emission analysis of Ca, Sr, Na or K. This function is used to perform the background correction of the coexistent component in samples.

In order to perform wavelength shift, select [Instrument] - [Set Wavelength to Background WL] from the menu. When no line search has been conducted, only wavelength editing is valid.

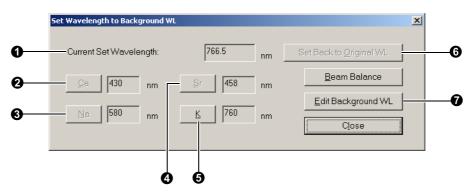


Fig. 4.13 [Set Wavelength to Background WL] Dialog Box

0	[Current Set Wavelength]	The currently set wavelength (nm) is indicated here. If the instrument settings have not been made, "" is displayed.
0	[Ca]	Shifts the wavelength for Ca to the wavelength shown to the right of this button (default: 430 nm).
8	[Na]	Shifts the wavelength for Na to the wavelength shown to the right of this button (default: 580 nm).
0	[Sr]	Shifts the wavelength for Sr to the wavelength shown to the right of this button (default: 458 nm).
6	[K]	Shifts the wavelength for K to the wavelength shown to the right of this button (default: 760 nm).
6	[Set Back to Original WL]	Returns the wavelength to the setting before the wavelength was shifted.
0	[Edit Background WL]	Displays the screen on which the wavelength to be shifted can be edited.

In the atomic absorption analysis, the [Fuel Gas Flow Rate] and [Burner Height] affect the absorption sensitivity. Although standard values are automatically set as default values by selecting an element, the optimum condition may differ depending on the sample characteristics and matrix components.

The "Optimum Burner Height Search" and "Optimum Gas Flow Rate Search" are the functions to search the optimum conditions by the measurement while changing the burner height or gas flow rate automatically. In this section, the operation procedures are described. For the details of each item in the window, refer to the HELP information in the WizAArd software.

In addition, read the section 4.5 "Igniting and Extinguishing the Flame" thoroughly and set the conditions for ignition.

4.8.1 Setting the Optimum Condition of Burner Height

- (1) Open the [Atomizer/Gas Flow Rate Setup] page in the Wizard.
- (2) Click on the radio button of [Atomizer Position] for operation object, and then the [Burner Position Auto] button is available. The optimum condition of burner height is obtained in the procedure described below.
- (3) Click on [Burner Position Auto]. Then the [Optimum Burner Height Search] dialog box appears.

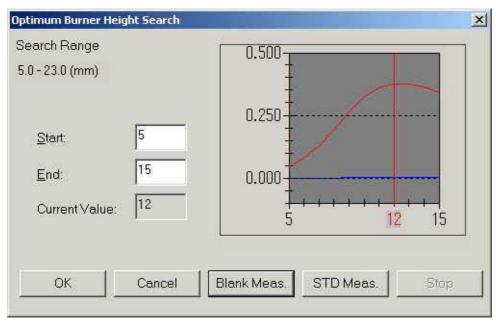


Fig. 4.14 [Optimum Burner Height Search] Dialog Box

- 1. Set the [Start] and [End] of the burner position search range. Although the available search range is within 5 to 23 (mm), you may set the range 5 to 10 (mm) for Air-C₂H₂ and 8 to 14 (mm) for N₂O-C₂H₂.
- 2. Press the PURGE button and IGNITE button on the front side of AA main unit to ignite a flame.
- 3. Start spraying the blank solution and click on [Blank Meas.] button. The measurement is performed while the burner height is changed by 0.5 mm step in the selected range. Continue to spray it until the measurement is completed.
- 4. Next, start spraying the standard (or sample) solution and click on [STD Meas.] button. In the same way as blank measurement, the measurement is performed while the burner height is changed. Continue to spray it until the measurement is completed.

The dialog box displays the graph which is created by subtracting the measurement result of <3> from the measurement result here. And the burner height with which the absorbance becomes the highest is also displayed in [Current Value].

5. If the result is acceptable, extinguish the flame and click on [OK]. The [Optimum Burner Height Search] dialog box is closed to return to the [Atomizer/Gas flow Rate Setup] page, and the obtained value is indicated and used as the [Burner Height]. If the [Cancel] is pressed, the burner height is returned to the original position.

4.8.2 Setting the Optimum Condition of Fuel Gas Flow Rate

- (1) Open the [Atomizer/Gas Flow Rate Setup] page in the Wizard.
- (2) Click on the radio button of [Gas Flow Rate] for operation object, and then the [Gas Flow Auto] button is available. The optimum condition of fuel gas flow rate is obtained in the procedure described below.
- (3) Click on [Gas Flow Auto]. Then the [Optimum Gas Flow Rate Search] dialog box appears.

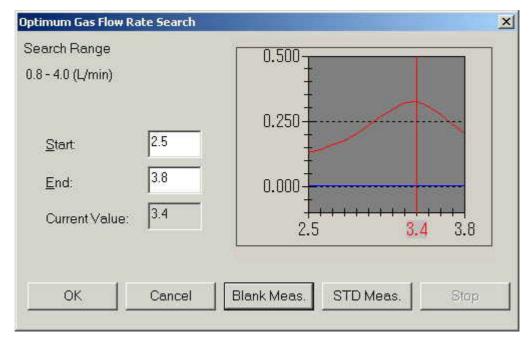


Fig. 4.15 [Optimum Gas Flow Rate Search] Dialog Box

- Set the [Start] and [End] of the gas flow search range. The range can be set within 0.8 to 4.0 L/min in the case of Air-C₂H₂ and within 5.8 to 9.0 L/min in the case of N₂O-C₂H₂. It is recommended to set the range within the standard condition ±0.5 L/min. The flow rate of support gas is fixed at the currently entered value. The optimal value for the flow rate of fuel gas must be obtained considering this fixed value.
- 2. Press the PURGE button and IGNITE button on the front side of AA main unit to ignite a flame.
- 3. Start spraying the blank solution and click on [Blank Meas.] button. The measurement is performed while the fuel gas flow rate is changed by 0.1 L/min step in the selected range. Continue to spray it until the measurement is completed.
- 4. Next, start spraying the standard (or sample) solution and click on [STD Meas.] button. In the same way as blank measurement, the measurement is performed while the fuel gas flow rate is changed. Continue to spray it until the measurement is completed.

The dialog box displays the graph which is created by subtracting the measurement result of <3> from the measurement result here. And the fuel gas flow rate with which the absorbance becomes the highest is also displayed in [Current Value].

5. If the result is acceptable, extinguish the flame and click on [OK]. The [Optimum Gas Flow Rate Search] dialog box is closed to return to the [Atomizer/Gas Flow Rate Setup] page, and the obtained value is indicated as the [Fuel Gas Flow Rate]. If the [Cancel] is pressed, the fuel gas flow rate is returned to the original position.

- If the [Fuel Gas Flow Rate] is changed, distribution of atoms in the flame changes, as the result, the optimum [Burner Height] also changes. On the other hand, changing the [Burner Height] also changes the optimum [Fuel Gas Flow Rate]. Note that the optimum condition changes if the other condition is changed.
- To search the optimum condition precisely, for example, select several levels of burner height and search the optimum gas flow rate at each level of burner height. Then select the best combination that shows highest sensitivity. (Or you can also select several levels of gas flow rate and search the burner height at each flow rate). Although some elements (such as Cu) are not affected so much by these conditions, some elements (such as Cr) are affected greatly.
- If these settings are not appropriate, not only the sensitivity decreases, but also poor reproducibility of data may be caused by sensitivity fluctuation. Utilize these functions for setting the optimum conditions when required.

Chapter 5 User Administration

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	System Administration Using the Security Support Functions Audit Trail Performing Software Validation Data Administration

5.1

This software is equipped with functions that meet the reliability requirements of GLP/GMP and FDA 21 CFR Part 11. The GxP support functions of this software provide effective and efficient help in achieving compliance with the rules and requirements of GLP/GMP and FDA 21 CFR Part 11.

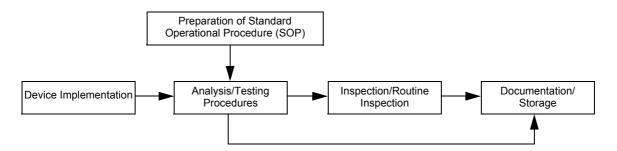
This chapter explains the basics of GLP/GMP and FDA 21 CFR Part 11, and gives an overview of GxP support functions provided in this software.

5.1.1 Overview of GxP Support Functions

5.1.1.1 What is GLP/GMP?

GLP (Good Laboratory Practice)/GMP (Good Manufacturing Practice) are guidelines/regulations to carry out analysis and testing practices properly. GLP is "a system to ensure the reliability of an analysis and test data", and GMP is "a set of standards for pharmaceutical production and its quality control".

Specifically, GLP/GMP assures the reliability of tests by ensuring the transparency of the test content to the outside. For that purpose, all testing practices are standardized in writing, and testing processes are recorded and saved. Also, a section is organized to conduct the systematic accuracy control and ensure the reliability of devices separately from the section that performs actual test, and this section inspects devices periodically.



The characteristic of this method is that an audit trail may be reviewed by managing and recording the entire process (validation, work flow, and data) until the results are obtained, in order to ensure the quality of the final results (data).

NOTE

- This software treats GLP and GMP together in the GxP support because, though they are different guidelines, there are many commonalities between the two in terms of methodology.
- For proper execution of a task, we recommend the usage of GLP/GMP methods, even in the analysis lab where GLP/GMP compliance is not necessarily required.

5.1.1.2 What is FDA 21 CFR Part 11?

It stands for the FDA (Food and Drug Administration) 21 Code of Federal Regulations Part 11, which states that the FDA approves an electronic record with an electronic signature as equivalent to a document record with a handwritten signature so far as that record conforms to this code.

The code is intended to speed up the FDA's process for approving new drugs. In the U.S., the electronic signature in the global/commercial trade is legislated and the legal status of the electronic signature is established. The above code may be regarded as a common regulation for handling computer-based documents as they are in the electronic format.

Since this trend is worldwide, electronic data output from a computerized analysis system conforming to the FDA 21 CFR Part 11 can be used throughout the world not only in the medical, pharmaceutical, and food categories, but also in other categories. The use of such electronic data provides effective means taking the place of long-term storage of paper-based data.

The FDA 21 CFR Part 11 mainly refers to the data security (function and access restrictions through security), data integrity (all-in-one file structure), audit trail, and login authentication, all of which are covered by the GxP support functions.

5.1.2 GxP Support Functions

WizAArd supports the conformance to the GLP/GMP and the FDA 21 CFR Part 11 through the following functions:

System Administration

- Establishment of user administration policy
- Establishment of system administration policy
- Registration of right groups
- Registration of users and permission of rights to users

Audit Trail

- · All-in-one data file structure
- · Audit trail log in file
- Operation log and system
 administration log

Data Administration

- · Data management and search based on sample information
- · Functions associated with CLASS-Agent/LabSolutions (Option)

The use of the GxP support functions available in the WizAArd is optional, but will powerfully support and save your GLP/GMP-related work that tends to become troublesome.

NOTE

For electronic signatures for reviews and approvals of electronic records or for long-term storage of such records, the combination with the optionally available CLASS-Agent/LabSolutions conforms to the FDA 21 CFR Part 11. Though this software is equipped also with the electronic signature function, this function only is insufficient for the FDA 21 CFR Part 11.

Work flow Management

- Login authentication and screen lockup
- Operational restrictions by specifying rights

Validation

- Hardware validation
- · Software validation
- · Software alteration check

5.2.1 Reviewing System Administration

This software includes a "System Administration" function. Users can use this function to limit the use of functions and programs to specific individuals to protect programs and data.

By utilizing this function, your laboratory can conform with GLP/GMP such as for analysis environment and analysis data reliability and management. This section explains the overall concept of the system administration function.

5.2.1.1 What is System Administration?

The system administration function will administer the following items:

(1) User

When this software is installed, an "Admin" user with complete access (i.e., system administration right) is registered. When you use the system administration function for the first time, use the "Admin" setting to register one person who uses this software as a "User".

User ID:	(Input when log in the system.)
User Name:	(Identify the user.)
Description:	
Pass <u>w</u> ord:	
Confirm Password:	
Administrator	
Groups Instruments	
<u>G</u> roup List	Selected Groups
H/W Administrator Method Developer	Add >>
Operator	
	<< Remove

Fig. 5.1 [Add User] Dialog Box

Enter the following settings to register a user:

No.	Parameter	Description
0	[User ID] [User Name]	Enter a user ID for login or a user name for display. Since the same name/ID can be used only one time in this software, you can enter your full name or detailed information in the [Description] field and an abbreviated name or alias in the [User Name] field.
0	[Description]	Enter a comment for the user ID/user name.
8	[Password]	Enter a password to be registered, if used.
4	[Administrator]	Put a check mark to provide the system administration right to the currently registered user. The administrator has complete access and cannot be registered in a group.
6	[Group]	Select groups where the user belongs. The user can use all functions that his/ her group has the rights to use.

No.	Parameter	Description
6	[Instruments]	Select the analytical instrument that the user can use.

To use functions that require their usage rights, the user needs to enter his/her registered ID and password in the [Login] window displayed when each program starts up.

If an ID or password entered in the [Login] window is different from the ones registered, the program will not start up.

(2) Rights (Group)

The [Rights] setting can limit the functions that can be used, using the system administration function.

The following groups with the rights shown have been registered. Registering a user to a group that has the necessary rights enables the user to have the same usage rights.

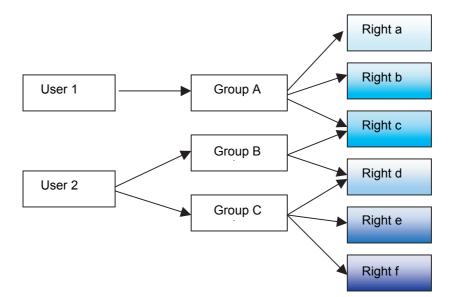


Fig. 5.2 Relation between User, Group and Right

Default rights

Right	Group with H/W Administrator rights	Group with Method Developer rights	Group with Operator rights
Edit Configuration	~		
Run Measurement		\checkmark	\checkmark
Edit Measurement Table		\checkmark	\checkmark
Edit Measurement Parameters		\checkmark	
Edit Data Processing Method		\checkmark	
Edit Acquisition Display Method		\checkmark	\checkmark
Edit Print Format		\checkmark	
Put Electronic Signature		\checkmark	\checkmark
Maintenance	✓		
QAQC Settings		\checkmark	
Instrument Manipulation	✓	\checkmark	\checkmark
Exit during Screen Lock	\checkmark		
Invalidate Electronic Signature			
Trial measurement	✓	\checkmark	\checkmark

NOTE

- 1. A user can belong to more than one group. In this case, that user will have all the rights of whichever groups he/she belongs.
- 2. The "System administrator" will have complete rights.
- 3. A new group with the desired rights can also be created.

For more information about creating new rights groups, refer to 5.2.3.2 "Registering (Changing/Deleting) Rights Groups".

5.2.1.2 System Administration Application

When performing system administration, select the level of administration you require from the following:

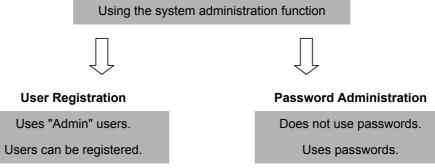


Fig. 5.3 System Administration Application

(1) Performing User Registration

Use the User Registration feature to clarify who the users are and specify which Analytical Instruments and functions each user can use.

Uses "Admin" users.

Installing this software registers an "Admin" user with complete rights.

If the System Administration function is not needed, any person can use the "Admin" User ID when the software is installed (no password and no [Normal Login] mode).

Users can be registered.

If the user registration feature is needed, the Analytical Instrument and functions allowable by each **user** can be limited (i.e.,usage rights can be specified). Also, complete rights can be given to all registered users as system administrators, to use their usage rights in the same way as being an "Admin" user.

(2) Performing Password Administration

A password is used to limit the software to particular users.

Does not use passwords.

Entering only a correct User ID in the [Login] window and leaving the [Password] field blank will enable the program to start.

Uses passwords.

If the registered password is not entered correctly in the [Password] field in the [Login] window, the program will not start.

5.2.1.3 System Administration Flow

System administration will be performed in the following flow:

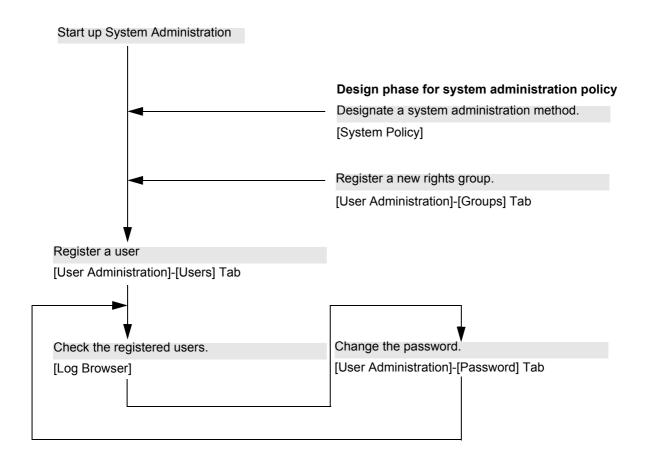


Fig. 5.4 System Administration Flow

5.2.1.4 Designing a System Administration Policy

(1) Designing a security level

Using the [System Policy] window (5.2.3.1 "Setting User Administration Policies"), which will be described later, set the security levels for user administration, etc.

On the [System Policy], the security level basically* becomes higher as the number of checked setting items increases.

The numeric settings are regarded appropriate if each parameter falls within the practical range* when a sufficiently short life span of password (approximately one several thousandth or less) is set for the mean decryption time for a password, which is obtained from the following equation:

* It is important to specify such Minimum Password Length and Maximum Password Age as you can remember without writing them down, such a Lockout Duration value as would not cause great inconveniences on your tasks if you should become unable to log in the system, and such a Login Attempt Limit value as provides allowance for you to correct it if necessary.

Lockout Duration × Number of Characters Used (Password Minimum Length)

Mean decryption time (min) =

Login Attempt Limit \times 2

NOTE

If the user ID is properly managed, it requires decryption resulting in a higher security level. For the above calculation, however, no user ID is taken into consideration assuming that character strings such as employee numbers, which may be found externally, are used.

The following example calculated b	w the above procedure shows one of the recommended set of values.
The following example calculated b	y the above procedure shows one of the recommended set of values:

Item	Recommended Value	Remarks
Number of Characters Used	36*	
Minimum Password Length	6	
Maximum Password Age	180 [days]	(3 to 4 month)
Lockout Duration	10 [minutes]	(15 to 30 minutes)
Login Attempt Limit	5 [Times]	(3Times)

In the case, the mean decryption time (converted into years) is 4,000 years or more providing an allowance of at least 8,000 times the above life span value of password (0.5 year). If you need an allowance of 10,000 times or more, enter stricter values than those specified in parentheses, which may be regarded as falling in the practical ranges.

If any of the above values is less strict, the other values will relatively become stricter for practical use. Therefore, Minimum Password Length = 6 and Maximum Password Age = 180 days are general settings for similar types of systems.

* Assuming that only alphanumeric characters are used. This case is not (Upper/Lower) case-sensitive for difficulty to force random mixed-case-strings.

NOTE

The WizAArd has more powerful user administration functions than Windows in that:

- Even if a user is deleted, it is only hidden, but its entry is retained. This prevents any other user with the same name from being registered. Therefore, the traceability of data that was acquired by the deleted user can be kept intact.
- The user ID is managed in combination with the corresponding user name and only used at login. For the subsequent display, the user name is used.
- (2) Designing right groups

On the [User Administration] - [Groups] tabbed page, which will be described later, group the rights in accordance with 5.2.3.2 "Registering (Changing/Deleting) Rights Groups".

For this grouping, it is important that the rights for the actual analytical tasks match with the settings for the right groups. Unless the unit set of rights to be included in each right group is appropriate, excessive rights may be given when a user is registered. This may make it difficult to assure that the final data has been obtained within the defined range of rights by the proper procedure.

5.2.2 Using the System Administration Tools to Set Available Items

When using any functions related to system administration, call up the System Administration Tools.

(1) When the [WizAArd] launcher has not started yet, double-click the

(WizAArd) icon on the desktop.

44

(2) Click on [Administration] tab from the [WizAArd] launcher. Log in with a User ID that has system administration right.



Fig. 5.5 [WizAArd] Launcher

NOTE

The settings entered [System Policy] and [User Administration] will become effective from the next time the program window is started (logged in).

Functions available in the "System Administration" tools

	Function	System Administration Right		Remarks	
		Yes	No		
[Sys	tem Policy]				
	Set general system administration parameters.	~			
[Use	r Administration]				
	Create a new rights group.	\checkmark			
	Change group registration information.	~		After changing settings, new settings become effective from the next time the program is started.	
	Register a new user.	\checkmark			
	Change user registration information.	~		Cannot delete a user currently logged in User Administration nor the Admin user registered at software installation. And can not change their rights, too.	
	Change the password.	~	~	Regular users can change only his/her password.	
[Log	Browser]				
	Display event logs.	\checkmark	\checkmark		
	Maintain event logs.	\checkmark			

5.2.3 Starting the System Operation

5.2.3.1 Setting User Administration Policies

To use the system administration function in this software, set the policies like login method and password limitations.

- (1) When the [WizAArd] launcher has not started yet, double-click the
- (2) Click on [System Policy] on the [WizAArd] launcher.



(WizAArd) icon on the desktop.

Administration Operation Validation User Administration

Fig. 5.6 [WizAArd] Launcher

Log in with a User ID that has system administration right.

	🗿 System Policy		
0	Setting		
	User Administration	,	
~			
8	Minimum Password Length:	0 🗦	D
0	Maximum <u>P</u> assword Age:	0 🕂 days	Permanent if set to 0 days.
4	Passwords must meet compl	e <u>x</u> ity requirements	
	Lockout Settings (for PC)		
6	Login <u>A</u> ttempt Limit:	0 🕂	Unlimited if set to 0.
•	Lockout Duration:	15 📑 min	
	Lockout Settings (for User)		
•		0 📫	Unlimited if set to 0.
6	Login Attempt Limit:		Orimniked in set to o.
	Lockout D <u>u</u> ration:	15 🕂 min	
0	# of Levels for Duplicate	0 🗧	No duplicate password
	Password Checking		checking if set to 0.

Fig. 5.7 Left half of [System Policy] Dialog Box

0	[Level] allows you to select the preset policy settings by clicking on the [Setting] button.		ct the preset policy settings by clicking on the [Setting] button.		
	Level		Description		
	Level 1	Low	No restriction is required for system operation.		
	Level 2		No restriction is required, but the system is operated by multiple users.		
	Level 3		The system must comply with GLP/ GMP regulation.		
	21 CFR P11	High	The system must comply with the FDA21 CFR Part 11.		
0	[Minimum Passv Length]	vord	Minimum number of password characters, used for user registration, and user password changes.		
8	[Maximum Pass Age]	word	If a program is started after number of password effective days, window appears to change password.		
4	[Password must meet complexity requirement]				
6	[Lockout Setting	s [for PC]	11		
	[Login Attempt L	imit]	If login failures exceed the specified number of times, all the users on the PC or network will become unable to log into the PC or network for the specified period of time.		
	[Lockout Duration]		Allows you to set the login lockout duration in the unit of PC.		
6	[Lockout Setting	s [for Use	er]]		
	[Login Attempt L	imit]	Allows you to set the upper limit of the number of login attempts in the unit of user.		
	[Lockout Duratio	n]	Allows you to set the login lockout duration in the unit of user.		
0	[# of levels for Duplicate Password Checking]				
			NOTE The setting is "0" or "10". When "10" is set, the setting in the configuration settings of the Shimadzu user authentication tool is reflected.		

To set the system administration policies other than user administration, use the right half of the [System Policy] window.

		×
0 0-	Audit Trail Apply audit trail function when creating file Reguire input of reason in recording autit trail Log	
0 —	Clear log when quitting WizAArd Report Output data <u>s</u> tatus in header	
4 —	File Management Prohibit overwriting of other <u>d</u> ata files Prohibit overwriting of other te <u>m</u> plate files Prohibit disposing of u <u>n</u> saved measured data on the memory	
	OK Cancel Help	

Fig. 5.8 Right half of [System Policy] Dialog Box

0	[Audit Trail]				
	[Apply audit trail function when creating file]	If this check mark is checked, audit trails are recorded automatically since a new file is created.			
	[Require input of reason in recording audit trail]	If this check mark is checked, the screen prompting to enter the reason for operation is displayed every time an audit trail is recorded automatically.			
0	[Log]				
	[Clear log when quitting WizAArd]	Clear "Log for System Administration" and "Instrument Log" when quitting WizAArd. (Same behavior to clicking "Clear Log" on Log Browser.)			
3	[Report]				
	[Output data status in header]	If this check mark is checked, "Status: Temporary" is printed at the header of the printed matter when printing is executed while data is not saved. The user can check for mismatch between the printed information and the information registered in the CLASS-Agent/LabSolutions.			

0	[File Management]	
	[Prohibit overwriting of other data files]	If this check mark is checked, if there is a file having the same name in the saving folder at the time of saving, overwriting the existing file is disabled.
	[Prohibit overwriting of other template files]	If this check mark is checked, if there is a file having the same name in the saving folder at the time of saving, overwriting the existing file is disabled.
	[Prohibit disposing of unsaved measured data on the memory]	If this check mark is checked, if there is unsaved measured data, shutdown of the WizAArd, creation of new files, reading of files and deletion of elements are disabled.

NOTE

- 1. The settings on the [System Policy] window will become valid at the next login.
- 2. The above setting to protect against overwriting files is not applicable when you open a file and modify it and then save. The setting is applied to prohibit overwriting other existing files if autosave function works during data acquisition or if the file is saved using [Save As] function.
- 3. "unsaved measured data" means the data that is acquired from the instrument and is not saved to a file yet. If you just open a data file and modify it and then the modified file is not saved yet, data in the file is not "unsaved measured data".
- 4. If file editing itself is the problem, use User Administration tool to specify whether or not to authorize each user with the right to perform edit and postrun analysis, or enable the audit trail for data files and record the modification log when saving the file.

5.2.3.2 Registering (Changing/Deleting) Rights Groups

When this software is installed, a number of standard rights groups are set up such as H/W Administrator, Method Developer, Operator. This section explains how to register (change/delete) a rights group.

(1) When the [WizAArd] launcher has not started yet, double-click the



(WizAArd) icon on the desktop.

(2) Click on [User Administration] icon from the [WizAArd] launcher.



Fig. 5.9 [WizAArd] Launcher

Log in with a User ID that has system administration right.

(3) Click on the [Group] tab from the [User Administration] property sheet.

Password Users Groups	
Group Name Description Image: H/W Ad Instrument Configuration, Control and Adju Image: Method Method Development. Image: Operator Data Acquisition and Data Analysis.	
Add <u>Remove</u> Properties	
OK Cancel Help	

Fig. 5.10 [User Administration]- [Groups] Tab

0	[Add]	Click [Add] to display [Add Group] window. Settings are same as [Group Properties] as follows.
0	[Remove]	Click [Remove] and a highlighted group name in group list is deleted.

(4) Click on [Properties] in the [User Administration] property sheet to set the group property.

Group Property

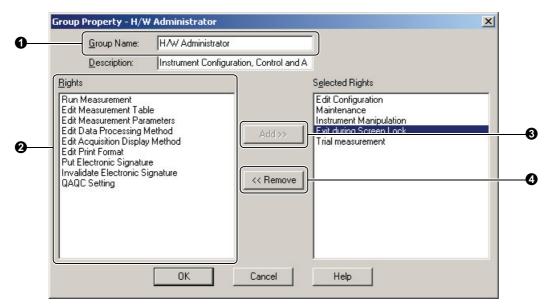


Fig. 5.11 [Group Property]

0	[Group Name]	Enter a group name.
0	[Right]	Select a desired item.
0	[Add]	Click [Add] to move the item from [Rights] to [Selected Rights]. Those rights are given to the group.
4	[Remove]	Click [Remove] to move a highlighted item from [Selected Rights] to [Rights].

NOTE

To specify a system administrator, use the [Add User] or [User Property] window.

5.2.4 Registering (Changing/Deleting) Users

Users can be registered not only to limit who can use this software, but also to understand each user's program usage conditions.

(WizAArd) icon on the desktop.

(1) When the [WizAArd] launcher has not started yet, double-click the

(2) Click on [User Administration] icon from the [WizAArd] launcher.



Fig. 5.12 [WizAArd] Launcher

Log in with a User ID that has system administration right.

(3) Click on [Users] Tab from the [User Administration] property sheet.

[User Administration]-[Users] Tab

	😵 User Administration	×
	Password Users Groups	
	User Name Description	
	System Administrator	
	Raro Shimadzu H/W Administrator	
	La Musashi Miyamoto Method Developer	
D	Add	
a	Groups Instrument	
	(All Group)	
	Close Cancel Help	

Fig. 5.13 [User Administration]-[Users] Tab

0	[Add]	Click [Add] to display [Add User] window. Settings are same as [User Property] as follows.
0	[Remove]	Clicking on the [Remove] button hides the user highlighted in the users list as deleted user.

User Property

(×
User ID:	musash	1		(Input v	vhen log in the system.)	
User Name:	Musash	ii Miyamot	D	(Identify) the user.)	
Description:	H/W A	dministrato	10			_
		*****			1	
–Password:		<u> </u>				
-Confirm Pas	sword:	******				
- Administra	ator					
- Administre	100					
o 1.						
- Groups Ins	truments <mark>1</mark>					1
Groups Ins	truments <mark> </mark>			Selecte	d Groups	
Group List Method Dev			Add >>		d Groups .dministrator	
Group List			Add >>			
Group List Method Dev		_	Add >> << Remove			
Group List Method Dev						
Group List Method Dev						
Group List						

Fig. 5.14 [Add User] Dialog Box

0	[User ID] [User Name]	Cannot skip User ID/user name entry.
0	[Password]	Click here to use password.
8	[Confirm Password]	Enter password twice for confirmation. Password length must be longer than that specified on [System Policy]'s [Minimum Password Length:]. Input will display with '*' marks.
4	[Administrator]	If checked, system administrator settings will be used. Regardless of [Selected Groups] settings, user will have all rights.
0	[Groups]	Select desired item from [Group List] and move to [Selected Groups:] using [Add>>]. User will have group's rights.
6	[Instruments]	Select desired item from [Selected Instrument items:] and move to [Instrument Item List:] using [< <remove]. analytical="" can't="" instrument.<="" th="" that="" use="" user=""></remove].>

NOTE

If you try to register a user with the same user ID/user name as that of a previously deleted user through the [Add User] window, a dialog box will be opened prompting you to confirm whether or not to restore the previous user.



Fig. 5.15 Message for Confirmation

5.2.5 Short Time after System Operation Has Been Started

After the system has been used for a while, recheck to see whether the parameters set in 5.2.3 "Starting the System Operation" (or the default settings used by registering a user) have met your purpose.

If the password validity is set, you will need to change the password after the expiry has come.

5.2.5.1 Viewing the History of System Administration

The system administration is traceable since changes in the system administration settings and operations performed on the System Administration window, such as password changes and user registrations are recorded as event logs.

- (1) When the [WizAArd] launcher has not started yet, double-click the
- e 🙀 (WizA

(WizAArd) icon on the desktop.

(2) Click on [Log Browser] Icon from the [WizAArd] launcher.



Fig. 5.16 [WizAArd] Launcher

Log Browser

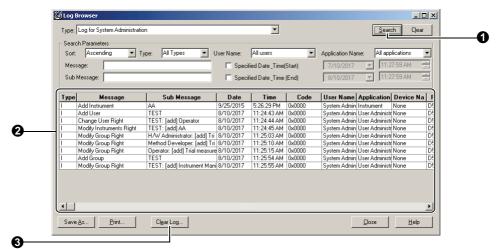


Fig. 5.17 [Log Browser]

(WizAArd) icon on the desktop.

0	[Search]	Enter search parameters and click [Search] button to pickup the records of Log.
0	Event log	Event log data can be converted to text file as input to commercial spreadsheet software such as MS-Excel.
8	[Clear Log]	If log data is not necessary, it can be deleted. The operation to save the log as a text file is required before it is deleted.

5.2.5.2 Changing Passwords

You can change your own password at any time, whether you are a system administrator or a regular user. Also, if a password expiration date is set with the system administration settings, any attempt to start a program after this date will bring up a dialog box prompting to change the password after the [Login] window appears. At this time, use the procedure below and change the password.

2

- (1) When the [WizAArd] launcher has not started yet, double-click the
- (2) Click on [User Administration] Icon from the [WizAArd] launcher.



Fig. 5.18 [WizAArd] Launcher

(3) Click on [Password] Tab from the [User Administration] property sheet. [User Administration] - [Password] Tab

	🛷 User Administration		×
	Password Users Group	s	
	<u>U</u> ser Name:	System Administrator	
0	Old Password:		
໑	New Password:		
9	Confirm New Password:		
	Clo	se Cancel	Help

Fig. 5.19 [User Administration] - [Password] Tab

0	[Old Password]	Enter current password for security.
0	[New Password] [Confirm New Password]	Enter new password twice for confirmation. Password length must be longer than that specified on [System Policy]'s [Minimum Password Length:]. Input will display with '*' marks.

NOTE

If a user logs in who does not have system administration right, [User Administration] window displays only [Password] tab.

	User Administration	×
	Password	
	User Name: Musashi Miyamoto	
	Old Password:	
	New Password:	
	Confirm New Password:	
-	OK Cancel Help	-
	OK Cancel Help	
	Fig. 5.20 [User Administration] - [Password] Tab	

NOTE

A user with the system administration right can perform initial settings/resettings of passwords all user (except Admin) in the [Users] tab's [User Property] window.

For more information about of the initial settings/resettings of user's passwords, refer to 5.2.4 "Registering (Changing/Deleting) Users".

5.3

The use of the security support functions facilitates controlling the workflow and improves the reliability of the obtained data.

5.3.1 Login Authentication

The system administration function uses login ID to clarify a user, and password confirmation to secure the data and system when he/she uses a program. This is called the login procedure.

In particular, the FDA 21 CFR Part 11 clearly positions login authentication as an essential element of electronic signature. Unless the requirements are satisfied, no electronic data can replace data on paper.

The concept of login ID and the function of password are in a mutually supplemental relation. Both must be valid to meet the requirements of electronic record and electronic signature and to manage work flow of individual users.

WizAArd Login		
	WizAArd	
Login ID :		ОК
<u>P</u> assword :		Cancel

Fig. 5.21 [WizAArd Login] Dialog Box

- The combination of two components (login ID and password) serves as an electronic signature.
- The login ID is only used at login and subsequently the user name is used for display.
- The entered password is displayed using asterisks (*). The system policy settings allow you to specify the minimum length and life span for a password.

NOTE

Employment of two distinct components in an electronic signature such as combination of a login ID and password is mandatory for compliant systems; however, it is meaningful only when accompanied by proper user management. Below are examples of some common practices that must be avoided.

- Multiple individuals use the same login ID (i.e. "Admin") for login. This practice is dangerous as it inhibits the ability to identify who handled the data, and to prevent unauthorized access to the system.
- Registers multiple login IDs with no password. Users can use any login IDs easily and there is no record who actually performed the operation.

For more information, refer to 5.2.3.1 "Setting User Administration Policies", 5.2.4 "Registering (Changing/ Deleting) Users".

5.3.2 Current User Display

One of the GLP/GMP requirements is to display the current user name in each window.

When logging in using a correct user ID using the system administration function, the user name will be displayed in each window's title area.



Fig. 5.22 Title Bar

5.3.3 Restricting the Operations according to User Rights

In order to restrict a task so as to be performed in accordance with the standardized procedure or to certify that the task has been done under that restriction, the system administration functions allow you to restrict the operable functions.

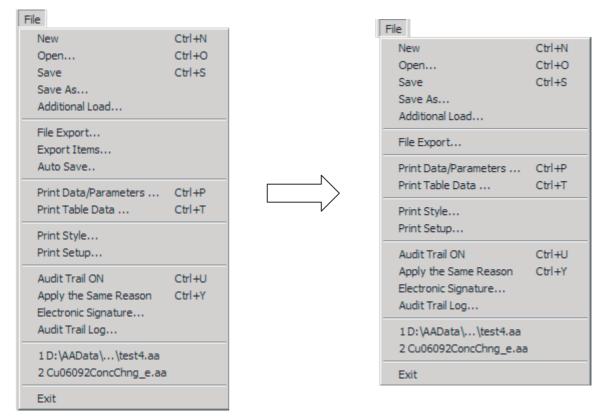


Fig. 5.23 Restricting the Operations according to User Rights

Left menu All the operations can be performed under the system administration right.

Right menu If the operator level right is selected, analytical tasks can only be done through a prepared method.

NOTE

- 1. The GLP/GMP assures the quality of the acquired data by showing the fact that the section for securing the reliability has been separated from that for actually carrying out the inspection and analysis and by certifying that the tasks are performed in accordance with the standardized procedures.
- 2. This function is also effective for preventing wrong operations and decreasing the time required for mastering the operations by hiding the unnecessary functions.

For more information, refer to 5.2.3.2 "Registering (Changing/Deleting) Rights Groups", 5.2.4 "Registering (Changing/Deleting) Users".

5.3.4 Protecting an Analytical Task by Locking the Screen

The locking function is available to protect the data from any accident through operations by another operator or any malicious operation when you temporarily leave the workstation during the automatic analysis or for any other reason.

(1) Click on [Window]-[Lock].

It is locked as Task Bar icon for a moment.



Fig. 5.24 Locking function

NOTE

If you attempt to open the screen, the [User Authentication] dialog box will pop up. The system will accept no operation while this dialog box is open.

User Authentio	cation		
<u>U</u> ser ID :		OK	
Password :		Cancel	
		Help	
Fig.	5.25 [User Authentication] Dialog	j Box	

Audit Trail

1	Data History	us	•	ansitions contained in the measurement data itself, and rity of the data. This history is displayed from the
2	System History		is is the history of tra zAArd supports the t	ansitions indicating changes on the workstation. The following logs.
		1	Log administration log	History of log administration. Only the Log Browser outputs this log. This log cannot be deleted.
		2	System administration log	 History of changes in the system administration tool. The following contents are displayed: User administration information change log System policy change log Instrument maintenance information change log Database maintenance change log
		3	User authentication tool log	Stores the user administration log information on the user authentication tool side. The log is recorded by the user authentication tool, and a copy is created locally when the Log Browser is started up. (There is information overlapping the user administration change log in the system administration log.)
		4	Instrument log	 Instrument log of WizAArd Login/logout log for each application History of changes in the WizAArd environmental settings Lamp setting change log Print format change log Instrument Configuration change log Text file export item change log Automatic save setting change log GFA heating times change log Electronic signature reason change log

5.4.1 Referencing a Record in a Data File

Using [File]-[Audit Trail Log], like sample information, related files, and so on that has been registered during data acquisition can be referred.

	User	Schedule	Item	Previous	After	F.▲
6/30/2003 6:59 3	Syst	Ca:0123	Row10 Insert Rows		UNK	
6/30/2003 6:59 3	Syst	Ca:0123	Row9 Insert Rows		UNK	
6/30/2003 6:59 3	Syst	Ca:0123	Row8 Insert Rows		CAL-CHK	
6/30/2003 6:59 3	Syst	Ca:0123	Row7 Sample ID		sample 0001	
6/30/2003 6:59 3	Syst	Ca:0123	Row7 Insert Rows		STD	
6/30/2003 6:57 3	Syst	Ca:0123	Calibration Curve	2	1	
6/30/2003 6:57 3	Syst	Ca:0123	Using ASC	No	Yes	
6/30/2003 6:57 3	Syst	Ca:0123	Socket#	1	NONE	
6/30/2003 6:57 3	Syst	Ca:0123	Blank Repeat	7-7-12.60	7-7-12.60-0.1	
	0		Carrier Data Dia	17 A	Vensien 404	۰
						_

Fig. 5.26 [Audit trail log] Dialog Box

5.4.2 Viewing the History of System Changes

For viewing the history of system changes, use the Log Browser explained in 5.2.5.1 "Viewing the History of System Administration". Operate as described below.

7

(WizAArd) icon on the desktop.

- (1) When the [WizAArd] launcher has not started yet, double-click the
- (2) Click on [Log Browser] from the [WizAArd] launcher.



Fig. 5.27 [WizAArd] Launcher

(3) Select [Type].

Sort	Ascending 💌 T	ype: 🛛 All Types 💌 L	Jser Name:	All users	-	Application Na	me: All app	lications	۲
Mess	age:		🗌 Specif	ied Date _Time(Start)	7/10/2017	▼ 11:2	7:59 AM	A
Subl	Message:		🗖 Specif	ied Date _Time	(End)	8/10/2017	▼ 11:2	7:59 AM	A
	,					·			
Гуре	Message	Sub Message	Date	Time	Code	User Name	Application	Device Na	Ţ
	Add Instrument	AA	9/25/2015	5:26:29 PM	0x0000	System Admin	Instrument	None	
	Add User	TEST	8/10/2017	11:24:43 AM	0x0000	System Admin	User Administr	None	Τ
	Change User Right	TEST: [add] Operator	8/10/2017	11:24:44 AM	0x0000	System Admin	User Administr	None	Т
	Modify Instruments Right	TEST: [add] AA	8/10/2017	11:24:45 AM	0x0000	System Admin	User Administr	None	Τ
	Modify Group Right	H/W Administrator: [add] Tri	8/10/2017	11:25:03 AM	0x0000	System Admin	User Administr	None	
	Modify Group Right	Method Developer: [add] Tri	8/10/2017	11:25:10 AM	0x0000	System Admin	User Administr	None	
	Modify Group Right	Operator: [add] Trial measure	8/10/2017		0x0000	System Admin	User Administr	None	
	Add Group	TEST	8/10/2017	11:25:54 AM	0x0000	System Admin	User Administr	None	
	Modify Group Right	TEST: [add] Instrument Mani	8/10/2017	11:25:55 AM	0x0000	System Admin	User Administr	None	

Fig. 5.28 Log Browser

The list of logs will be displayed.

NOTE

If the optional Agent Connection Kit and the CLASS-Public Agent/LabSolutions Multi-Data Register have been installed, instrument logs and system administration logs unique to WizAArd can be registered to CLASS-Agent/LabSolutions for management.

5.5

Performing Software Validation

5.5.1 Performing Program Checks

The WizAArd provides the "Program Alteration Check" function, which is used to see whether the program related files for this software remains unchanged.

This function allows you to find the identity of the software (eliminate any wrong program component and immediately find infection with PC virus after installation).



Fig. 5.29 [WizAArd] Launcher

(1) Specify a program used in the program alteration check, then click on [Check].

😫 Check the program	×
Program List: User Authentication Tool Ver.1.08 WizAArd Ver.5.00	
System Administrator Select from Program List and Click [Check] buttor	n.

Fig. 5.30 Check the Program

(2) Click on the [Execute] button.



Fig. 5.31 Check the Program Files

Program check result details are viewed via the Result Viewer.

After program check is complete, check results appear.

🖳 Notepad			
Eile			
** Check the Program Files **			A
Operator : System Administrator Program Name : User Authentication Tool Version : 1.08 Check Date : 2010-02-19 15:27:41			
Filename			File.
<win.sys>SMZATST.exe <win.sys>SmzAtst.ocx</win.sys></win.sys>			Pas Pas
Total : Pass			
			▼
System Administrator	2/19/2010	3:27 PM	

Fig. 5.32 Notepad

NOTE

To get report of Program Check Result, Click on the [Print] button.

5.5.2 Performing the Function Check

Check whether or not the installed program is operating normally.

NOTE

Load the check data file from the WizAArd Setup CD-ROM every time software validation is performed to prevent it from being overwritten in previous function checks or operations. The data is saved in the "Sample" folder in the WizAArd setup disk.

(1) Use the WizAArd function check data file "AAIQOQFlame.aa".

When the file "AAIQOQFlame.aa" is loaded correctly, a calibration curve is displayed as shown below.

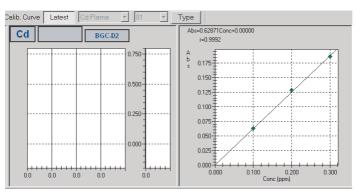


Fig. 5.33 Calibration curve displayed in WizAArd main window

(2) Change the calibration curve type, and confirm that the quantitative result is calculated again correctly. Check the quantitative result using the concentration of "UNK1-AV" in the sample table.

Linear equation (pass through the origin)

Calibration curve equation	Abs=0.62871Conc+0.0000
Correlation Coefficient	r=0.9992
Concentration of UNK1-AV	0.0956

Quadratic equation (pass through the origin)

Calibration curve equation	Abs=-0.098947Conc^2+0.65416Conc+0.0000
Correlation coefficient	r=0.9996
Concentration of UNK1-AV	0.0932

5.6.1 Reviewing the Long-Term Storage of Data

The previous sections describe the functions to support the management of working data. This section explains important points in the long-term storage of data.

5.6.1.1 Managing the Print Images

5.6

Among the existing techniques, printing out a report on paper and then storing it is the most excellent one for the long-term storage of data. This is still true today, but the disadvantage of this method lies in a problem with storage space and costs.

As a solution, print images are photographed on microfilm in some cases. At present, however, an approach for recording reports in an electronic document format that will be supported for a long period of time is adopted. Since some electronic document formats are accepted for formal documents, the electronic recording of documents has become popular.

The WizAArd provides the capability of outputting reports in the PDF (Portable Document Format) among the above electronic document formats when it is used with Adobe Acrobat, a tool for the creation, management, and edit of PDF documents.

It is also available to select "Adobe PDF" as the printer driver in printing in WizAArd to create PDF documents.

5.6.1.2 Storing a Data File for a Long Period of Time

In addition to print images, the WizAArd's data files(*.aa) those that can be analyzed (machine readable data) may be stored for a long period of time.

(1) Storing a file using an archiver product such as CLASS-Agent

WizAArd offers powerful link* with our data management software with built-in archive function such as CLASS-Agent and LabSolutions. Such software including CLASS-Agent and LabSolutions is compliant with FDA 21 CFR Part 11. Some of the commercially available electronic document management software for PDF files or the like provide effective capabilities for document management.

- * This function is optional. WizAArd has a function to create a print image file in the PDF format while saving data and transfer it to CLASS-Agent and LabSolutions.
- (2) Storing a file on CD-R or the like

The WizAArd has the data file format of the All-in-One File Structure. In addition, within the range of normal operations, related files such as method files are managed so that they are included in a single file.

Upon completion of a project, a set of related files on a folder basis should be stored on a non-rewritable media such as CD-R.

NOTE

CLASS-Agent, LabSolutions and some of other archiver products described above also have a function to store files on CD-R.

5.6.1.3 Devising a Data Storage Method

To store both printed image data and machine readable data, the relation between print images and the data files used to acquire them must be maintained.

Using CLASS-Agent and LabSolutions is the most recommendable methods to manage data. CLASS-Agent and LabSolutions can properly manage WizAArd data files and PDF files of print images.

NOTE

Managing PDF file with CLASS-Agent and LabSolutions can be turned ON/OFF.

5.6.1.4 Using a Print Image

If a report image of an analysis result report is saved as a PDF file, the character information and image information on the report can be utilized on other software.

5.7

Other System Management Tools

5.7.1 Performing the Maintenance of System Management Database

The [Database Maintenance] window allows you to perform maintenance of the database, such as compacting or repairing the database and assigning the database file.

5.7.1.1 Using the Database Maintenance Tool

The [Database Maintenance] window appears when:

- 1. Shut down all the WizAArd programs.
- 2. Double-click on the WizAArdMntDB.exe program in the folder where WizAArd is installed. [Login] window will appear.

WizAArd Login	WizAArd	
Login ID : Password :		OK Cancel

Fig. 5.34 [Login Dialog] Box

3. When the user having the system administration right logs in the system, the [Database Maintenance] window will be displayed.

-Database File • System Administration:	C:\Program Files\Shimadzu\WizAArd\System\WizA	Close
O Log:	C:\Program Files\Shimadzu\WizAArd\Log	Help
		Network

Fig. 5.35 Database Maintenance

0	[Repair Database]	Clicking on this button duplicates the database and then reconstructs the fragmented database into a more compact one or attempts to repair the database having a fault.
0	[Change Database]	Clicking on this button displays the Select File window. If a file in a new reference source is selected, that management database will be referenced starting with the next activation.
6	[Network]	After you have selected referencing the common management database on the LAN, checking this option will cause the system administration function to operate in the network mode starting with the next activation.

NOTE

If the network mode is selected, the system administration information (user rights, etc.) entered from one point on the LAN can be shared by all the WizAArd that uses the common system administration database.

5.7.2 Managing the System on the Network

Using the Shimadzu user authentication tool and the maintenance of instrument information tool allows you to share the system administration information on the network.

5.7.2.1 Using Shimadzu User Authentication Tool on the Network

You can share the user information (User ID, User Name, Password, etc.) and the user administration policy with other software that use Shimadzu user authentication tool when it is used on the network.

Refer to Using Shimadzu User Authentication Tool Instruction Manual to know how to setup to use it on the network.

NOTE

- MSDE (Microsoft Data Engine) is required to use Shimadzu user authentication tool on the network. MSDE installer is attached in CLASS-Agent setup disk, Office2000/XP setup disk. You can download it from Microsoft web site.
- If you use MSDE installer of Office2000/XP or one downloaded from web site, and do NOT use one of CLASS-Agent setup disk, please note the followings. If MSDE2000 is installed by default options, it is Windows authentication mode and it requires password. It is also prohibited from using it on the network by default. Thus, add the following options to MSDE2000 install command for Shimadzu user authentication tool to use it.

(Setup file name) BLANKSAPWD=1 SECURITYMODE=SQL DISABLENETWORKPROTOCOLS=0

(Example) Execute following command in [Run] menu. c:\sql2kdesk.exe BLANKSAPWD=1 SECURITYMODE=SQL DISABLENETWORKPROTOCOLS=0

 MSDE has different programs for each language. If you use CLASS-Agent English version, install MSDE English version. If you install other language version of MSDE, CLASS-Agent does not work.

5.7.2.2 Using the Maintenance of Instrument Information Tool

CAUTION

If you share system the administration database file on the network, please note the followings.

- 1. Setup to use Shimadzu user authentication tool on the network.
- 2. If you edit PC information, device name, user information, etc., exit all other WizAArd programs that share the administration database file on the network.

The [Maintenance of Instrument Information] window is opened when the following steps are performed:

- (1) Shut down all the WizAArd programs.
- (2) Using the [Database Maintenance] tool, select the network mode for all the WizAArd software that shares the system administration information.

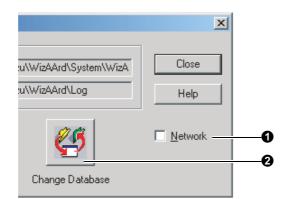


Fig. 5.36 Database Maintenance

0	Check this option to select the network mode for the system administration function.
0	Select referencing the system administration database files that are shared on the network.

- (3) Disable the [Database Maintenance] tool.
- (4) Unlike the standalone mode, multiple PCs are managed on the [System Administration Database]. Using the following [Maintenance of Instrument Information] tool, therefore, associate the current PCs with PC information on the [System Administration Database] (or "Add Computer" information if not available). Double-click on [WizAArdMntPC.exe] in the folder where WizAArd is installed. [Login] dialog box comes up.

(5) Log in with a User ID that has system administration right. The [Add/Specify Computer] dialog box of the [Maintenance of Instrument Information] will appear.

NOTE

If the Add/Specify Computer option is already selected and if the current PCs are associated with PC information, then this window is skipped.

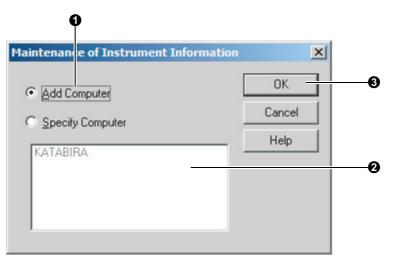


Fig. 5.37 Add/Specify Computer Dialog Box

0	Add the computer as the one that is managed (identified) on the system administration database in the network mode.
0	For the computer that is already registered on the system administration database (e.g., the first computer or a computer that was once added, but has the software reinstalled for any reason), select it from the list and then specify it so that it is associated with the displayed existing computer information.
€	After finishing the setting, click on the [OK] button.

	🅌 Maintenance of	Instrument Inform	ation (System 🗙
	Computer	PCID	Close
0	KATABIRA	▼ 1	Help
	Instrument AA	1D	
0			
	Deļete		
	Information about C	omputer	
	Computer Name:	KATABIRA	
	Description:		
	PCID:	1	Change

[Maintenance of Instrument Information] main window comes up after finishing the above setting.

Fig. 5.38 Maintenance of Instrument Information

0	Displays the list of computers that are centrally managed on the network
0	Analytical Instruments, which are linked with the computer above selected, is shown here. The entry of the analysis system selected from the [Instrument] list may be deleted,e.g., when the actual system is abolished.

(6) At this point, the network settings for the system administration are complete. After checking that there is any registration in the [Computer] pull-down list, click on the [Close] button to close the [Maintenance of Instrument Information] window.

Chapter 6 QA/QC Setup

CONTENTS

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6.2	QA/QC Check Items and Their Settings	6-3

QA/QC as described here is an abbreviation for Quality Assurance/Quality Control included in the analysis method that the U.S. Environmental Protection Agency requires for inorganic analysis of water and soil (USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis).

The Statement of Work for Inorganic Analysis describes the specific methods of demonstrating that the quality of the actually collected data is assured, as well as the pretreatment method and analysis conditions that should be observed by the analyst.

It is not obligatory to perform the QA/QC checks for any document other than the reports to the EPA. However, the concepts and approaches of the QA/QC checks seem effective in many cases. The WizAArd software allows you to automatically perform these checks.

This chapter describes the requirements of the QA/QC checks and the corresponding settings of the WizAArd software.

6.1.1 Overview of QA/QC

This section describes the specific operations for the QA/QC checks. A number of abbreviations will be used, for which you may refer to the terminology at the end of this chapter.

The flow of measurement using the atomic absorption spectrophotometer consists of pretreatment of the sample, creation of a calibration using the instrument, and measurement of the pretreated sample.

The QA/QC check items are classified into those used to validate the created calibration curve and those used to verify the effects of the pretreatment and liquid properties.

- (1) In validating the calibration curve, the calibration curve is evaluated using the correlation coefficient and a standard solution other than the one used for creating the calibration curve is measured. These checks are classified into ICV and ICB that are carried out immediately after a calibration curve has been created, and CCV and CCB that are used to check the sensitivity of the instrument that changes with time.
- (2) For the effects of the pretreatment and liquid properties, conduct the spike test. The spike test is an addition/recovery test, which includes SPK (to comprehensively verify the sublimation during processing and the effect of liquid properties) in which a known amount of a standard solution is added before the pretreatment and PDS in which a known amount of a standard solution is added after the pretreatment. This test also includes duplication in which the same pretreatment is separately applied to the same samples to verify the reproducibility of the pretreatment and PB in which a blank for the pretreatment is verified. If samples are common, they may be handled as a group. When one sample from that group is verified, the entire group is also verified.

In EPA, a reference value is established for each check item. There are different instructions depending on whether the established value is met or not met. More specific reference values and instructions will be mentioned in the description of each item.

6.2

This section specifically describes the QA/QC items in the order conforming to the USEPA's standard.

Order

- 1. Calibration of instrument (creation of calibration curve)
- 2. ICV and ICB
- 3. CCV and CCB
- 4. CRA: CRDL for AA
- 5. PB: Preparation blank
- 6. LCS: Laboratory control sample analysis
- 7. S: Spike test
- 8. D: Duplicate sample analysis
- 9. IDL: Instrument detection limit determination

The reference value for each item must be met. If it is met, you unconditionally go forward to the next measurement. If it is not met, you may select processing.

"Mark & Continue"	: A comment stating the reference value is not met is displayed in Out-of-Control Note and the measurement continues.
"Pause"	: The measurement stops when the reference value is not met (a message is displayed in the case of the manual measurement).
[Retry]	 If ticked, only the samples that did not meet the reference value will be re- measured only once under the same conditions. Either "Mark & Continue" or "Pause" is selected in accordance with the 2nd result.

6.2.1 Calibrating the Instrument (Creating a Calibration Curve)

The instrument must be calibrated daily or once or more frequently within 24 hours and the standard solution that is used for measurement must be prepared just when analysis is made.

A blank sample and standard solutions of at least 3 concentrations must be measured. One of them must have CRDL (Contract required detection limit). The kind and concentration of acid used must be same as those used for samples.

The difference between the concentrations of the standard solution that has been re-determined based on the created calibration curve (TV: self-determined true value) and the prepared concentration must be within 5%.

(1) In the WizAArd software, click on the [Calibration Curve Setup] button in the [Preparation Parameters] page of the element selection wizard.

The [Calibration Curve Setup] window is displayed.

(2) Click on the [QC Blank/QC Standard Setup] button.

Calibration Curve Setu	ıp								×	
Method of Stand	ard Additior	1		ommon Setti	ngs of Prep	aration Para	meters		ОК	
Order 1st 💌 Conc. NONE 💌				Mixing ON Mixing			g	Cancel		
Zero Intercept				Repeat Con	ditions	Coati	ng			
QC Blank/	'QC Standar	d Setup				Reag	ent	Load	Save	
Blank Preparation F	arameters									
Auto Frequ ency	Pos. (uL		nt Reage R2							
20	1 10	0	0	0	0	10				
Reslope Preparatio	Conc.	rs Pos. VOL (uL)			R	3 R4		ne		
Measurement Sequ	ence for Cal	-			uto Dilution temeasurer		UNK. Samp Upper Limit	le Conc.	0.0000	
Action	Sample ID	True Value	Pos.	VOL (uL)	Diluent R1	Reagent 1 R2	Reagent 2 R3	Reagent 3 R4	Total Volume	
STD		0.5000	1	10	0	0	0	0	10	
STD		1.0000	1	10	0	0	0	0	10	
			· · · · · · · · · · · · · · · · · · ·							

Fig. 6.1 [Calibration Curve Setup] page

The [QC Black/QC Standard Setup] window is displayed.

C	QA/QC Type	Accepte	ance Crite	ria f	Retry	Out of Control Act	ion
-	CORR		>= 0.99	5	г [Mark & Continue	ue
-	%TV	95	%- 10	5 %	– [Mark & Continue	
	Set %T	✓ Application	Lower Li	mit 🔽 🛛	.0000 <	STD true value	
Г	ICV	90	%- 11	0 %	г [Mark & Continue	
Г	ICB	,	<= CRD	L	г [Mark & Continue	
Г	CCV	90	%- 11	0 %	п [Mark & Continue	
Г	ССВ		<= CRD	L	п [Mark & Continue	
	CCB, CC	√ Frequency	per 2	0 s	ample(s), o	r 120 r	ninute(
Pre	paration Par	ameters	tie ee				
	Method	True Value	Pos.	VOL	Diluent R1	Reagent 1 R2	121212-000
	ICV/CCV	0.0000	1	10	0	0	
	ICB/CCB		1	10	0	0	

Fig. 6.2 [QC Blank/QC Standard Setup] dialog box

To perform the QA/QC checks, tick desired QA/QC Type as shown in the figure.

0	[Acceptance Criteria]	[Acceptance Criteria] may be set freely, but the default value that is first displayed is the USEPA's recommended condition.
0	[Retry]	[Retry] is used to perform re-measurement if the measurement result does not meet the acceptance criteria. If this option is not ticked, the next action is taken directly. If ticked, the measurement is performed only once if the measurement result does not meet the acceptance criteria and then the next action is determined by the 2nd result.
0	[Out of Control Action]	[Out of Control Action] allows you to select either "Mark & Continue" or "Pause". Select "Mark & Continue", if you want to continue the measurement regardless of the result of the QA/QC check. Select "Pause" to review the preparation of the sample and other factors when the result shows a failure in meeting the reference value.

3	[CORR] (Correlation coefficient = r)	This coefficient shows how much the measured value is close to the calibration curve obtained by the least square method. As the coefficient is closer to 1, the result becomes better. r is calculated with the following equation. r = (N Σ xi yi- Σ xi Σ yi)/{N Σ xi ² -(Σ xi) ² } ^{1/2} {N Σ yi ² -(Σ yi) ² } ^{1/2} To evaluate the correlation coefficient, tick this check box and select "CAL-CHK" in the [Action] field of the MRT worksheet. NOTE "CAL-CHK" must be selected only for the calibration curve method. This setting is not required for the simple standard addition method or the standard addition method.
0	[%TV] (Self-determined true value)	The calibration curve obtained by measuring a set of standard samples is used to evaluate how much the result obtained by re-determining the concentration of the standard sample at different points is correct relative to the prepared concentration. The difference from the prepared concentration must be within 5% (USEPA's recommended condition). To enter the %TV applicable lower limit, tick the [Set %TV Application Lower Limit] check box and enter a value. The %TV applicable lower limits of only 23 elements are recommended by the USEPA as shown in the Table below, but the measurement method differs depending upon the element. (Flame) indicates that this element is used for measurement by the flame continuous method. As, Se, and Sb are used for the hydride generation method, Hg for the reduction and vaporization method, and others for the furnace method.

Table 6.1 List of CRDLs

Analyte	CRDL (ug/L)
AI	200
Sb (*)	60
As (*)	10
Ва	200
Ве	5
Cd	5
Ca (Flame)	5000
Cr	10
Со	50
Cu	25
Fe	100
Pb	3

Analyte	CRDL (ug/L)
Mg (Flame)	5000
Mn	15
Hg (*)	0.2
Ni	40
K (Flame)	5000
Se (*)	5
Ag	10
Na (Flame)	5000
Thallium	10
V	50
Zn	20

IDL/CRDL may also be specified in the window displayed by selecting [Parameters]-[IDL/CRDL Setup] from the menu bar.

6.2.2 ICV and ICB

QA/QC Type		ance Crite		Retry		t of Control Act	
CORR		>= 0.995	ñ.	Г	[Ma	rk & Continue	
□ %TV	95	% - 10	5 %	Г	Ма	rk & Continue	
Set%T	V Application	Lower Lir	nit 🔽 🛛	0000	< ST	D true value	
−r ICV	90	% - 110) %	Г	Ма	rk & Continue	
-⊽ ICB		<= CRDL	-	Γ	Ма	rk & Continue	
	90	%- 111	%	Г	Ma	rk & Continue	
Г ССВ		<= CRDI	-	Г	Ma	rk.& Continue	
CCB, CC ⁴	√ Frequency	per 20) s	ample(s), or	120 r	ninute(s
Preparation Par	ameters						
Method	True Value	Pos.	VOL	Dilu	10525	Reagent 1 R2	Reag R
ICV/CCV	0.5000	1	10	(0	
ICB/CCB		1	10	0)	0	(

Fig. 6.3 Settings for ICV and ICB

0	[ICV] (Initial Calibration Verification)	Perform this verification immediately after a calibration has been created. The solution for use in verification is different from the standard solution used to create the calibration curve and must have a different concentration from the one used for creating the calibration curve, within the concentration range for calibration curves. The EPA's acceptance criteria require that the measured value fall within a range between 90% and 110% of the prepared concentration (between 80% and 120% for HG). If the result exceeds this range, the measurement is stopped.
0	[ICB] (Initial Calibration Blank)	Perform this measurement immediately after ICV. It is intended to check that the blank value decreases properly. The EPA's acceptance criteria requires that the analysis should be stopped if CRDL is exceeded and that the re-measurement should be performed after the problem has been solved.

6.2.3 CCV and CCB

07	A/QCType	Accepta	ance Crite	ria F	Retry	Out of Control Act	ion
Γ	CORR		>= 0.99	5	Г	Mark & Continue	
	%TV	95	%- 10	5 %	Г	Mark & Continue	
	Set %T	/ Application	Lower Li	nit 🔽 🛛	0000	< STD true value	
V	ICV	90	% - 11	0 %	Г	Mark & Continue	
₹	ICB		<= CRD	_		Mark & Continue	
-⊽-	CCV	90	%- 11	0. %		Mark & Continue	
-	ССВ		<= CRD		Г	Mark & Continue	
	CCB, CC\	/Frequency	per 2) s	ample(s),	or 120 n	ninute(s)
Pre	paration Par	ameters					
	Method	True Value	Pos.	VOL	Dilue R1	nt Reagent 1 R2	Reagen R3
	ICV/CCV	0.5000	1	10	0	0	0
			2	10	0	0	0

Fig. 6.4 Settings for CCV and CCB

0	[CCV] (Continuing Calibration Verification)	Perform this verification at certain intervals of sample measurement (either 10 samples or 2 hours, whichever shorter) in order to verify the accuracy of the calibration curve during analysis. This standard solution has a concentration that is different from that of the standard solution used for ICV and is nearly in the middle of the range of calibration curves. The acceptance criteria require that the measured value fall within a range between 90% and 110% of the prepared concentration (a range between 80% and 120% for Hg). If the result exceeds this range, the measurement is stopped.
0	[CCB] (Continuing Calibration Blank)	Perform blank measurement immediately after CCV. The EPA's acceptance criteria requires that the analysis should be stopped if CRDL is exceeded and that the re-measurement be performed either for 10 previous samples or for all the samples up to the former ICB and CCB after the problem has been solved.

6.2.4 CRA (CRDL for AA)

In order to verify the linearity in the neighborhood of the CRDL concentration, perform measurement at either CRDL or IDL, whichever higher concentration. There is no applicable reference value. Merely enter the ratio (%R) of the actually measured concentration to the prepared concentration.

The CRA setting should be entered on the MRT worksheet.

(1) Click on the [Action] cell in the row to which CRA is applied, to display the drop-down list.

	Action	Sample ID	Graph	x	м	Q
1	STD	8				5
2	STD					
3	STD			-		
4	JUNK1			-	_	
5	UNK2					
6	UNK3					
7	ныка					
8						
9	ICV 🔳					
10	ccv 🚽					
11	ICB					
12	ССВ					
13	PB					
	PDS DUP	-				
	CRA 🔽	-				
		IJ				

Fig. 6.5 CRA setting

(2) Select CRA from the list to establish it. After establishing the CRA, enter a value in the [True Value] field.

6.2.5 PB (Preparation Blank)

PB is a solution that contains no sample and has been prepared under the same reagent and conditions used for the pretreatment. It is used to check for a reagent and contamination during processing. PB is classified as follows by comparing it with CRDL:

- If the absolute value for the blank concentration is lower than CRDL, no correction is required for the measurement result of the sample.
- If the blank concentration is higher than CRDL, the minimum concentration of the sample must be 10 times larger than the blank value. All the samples of which concentration is less than 10 times the blank concentration and higher than CRDL must be re-treated and reanalyzed. For the concentrations of samples, no blank values are subtracted.
- If the blank concentration is less than negative CRDL, all the samples of which concentration is less than 10 times CRDL must be re-treated and reanalyzed.
- (1) Click on the [Sample Group Setup] button in the [Preparation Parameters] page of the element selection wizard, and then the [Sample Group Setup] window shown in Fig. 6.6 is displayed.
- (2) Click on the [QAQC Setup] button.

	etup							
ample Group	Number							
					. We	ight Correctio	n Factors	
•	Upda	ate Current San	nple Gro	up Setting			VF 1.00	
	1	New Samp	nle Grou	n	VVr	-11.000000	VF 11.00	
	-	11011 0 0.111		-		1.00	CF 1.000000	
(D,	1.000000	.	
	QAQCS	etup			Actual	Conc. Unit	NONE -	
L								
nknown/Spik	e Prepara	ation Paramete	rs					
Method	SA Con	nc. VOL	1	Diluent	Reagent 1	Reagent 2	Reagent 3	Total
Sample				R1	R2	R3	R4	Volume
sampie Spike	0.000	0 10			0	10 10		
					ti (a)	1 319	d	10021
		rement Sequer Sample ID	nce Pos.	WF	Add to		No. of ho	
Jnknown/Spik		rement Sequer Sample ID	Pos.		MRT		No. of 10 Samples:	Upc
Inknown/Spik		 	Pos.	1.000000	MRT		Samples:	
Jnknown/Spik Acti UNK 2 UNK		 	Pos. 1 2	1.000000 1.000000	MRT IV		00000000000000000000000000000000000000	Upc
Jnknown/Spik Acti		 	Pos.	1.000000	MRT		Samples: ¹⁰ Collective S	etup
Jnknown/Spik Acti UNK UNK		 	Pos. 1 2 3	1.000000 1.000000 1.000000	MRT IV IV IV		Samples:	etup
Jnknown/Spik Acti UNK UNK UNK UNK		 	Pos. 1 2 3 4	1.000000 1.000000 1.000000 1.000000	MRT		Samples: Collective S Import.	etup
Jnknown/Spik Acti UNK 2 UNK 3 UNK 4 UNK 5 UNK 5 UNK 7 UNK		 	Pos. 1 2 3 4 5	1.000000 1.000000 1.000000 1.000000 1.000000	MRT V V V V V V V		Samples: ¹⁰ Collective S	etup
Jnknown/Spik		 	Pos. 1 2 3 4 5 6	1.000000 1.000000 1.000000 1.000000 1.000000 1.000000 1.000000	MRT 17 17 17 17 17 17 17 17 17 17		Samples: Collective S Import.	etup
Jnknown/Spik		 	Pos. 1 2 3 4 5 6 7 8 9	1.00000 1.00000 1.00000 1.00000 1.00000 1.00000 1.00000 1.00000 1.00000 1.00000	MRT IV IV		Samples: ¹⁰ Collective S Import. Export.	etup
Jnknown/Spik		 	Pos. 1 2 3 4 5 6 7 8	1.000000 1.000000 1.000000 1.000000 1.000000 1.000000 1.000000 1.000000	MRT 17 17 17 17 17 17 17 17 17 17		Samples: Collective S Import.	etup
Jnknown/Spik		 	Pos. 1 2 3 4 5 6 7 8 9	1.00000 1.00000 1.00000 1.00000 1.00000 1.00000 1.00000 1.00000 1.00000 1.00000	MRT IV		Samples: ¹⁰ Collective S Import. Export.	etup

Fig. 6.6 [Sample Group Setup] Page

The [Sample Group QA/QC Setup] dialog box is displayed.

(3) In the window shown in Fig. 6.7, tick the [Preparation Blank (PB)] check box. If the ASC is used, enter the preparation parameters such as position and sample volume in the preparation parameters table located at the bottom of the window.

Q.	A/QC Type		Acceptar	nce Criteria	Retry	U	ut of Control A	cuon
LC LC	S		80	%-120	% □	Mar	k & Continue	*
	eparation Bla	nk(PB)		<= CRDL	Γ	Mar	k & Continue	•
Pre-C	igestion Spik	e(SPK)	85	%- 115	% □	Mar	k & Continue	•
Post-I	Digestion Spi	ke(PDS)	85	%- 115	% □	Mar	k & Continue	-
				00	% □	Mar	k & Continue	
Dupli	cate(DUP)			< = 20	/0	1.40	K & Continue	
repare	cate(DUP) ition Parametr True Value	ers Pos.	VOL	<= 20 Diluent R1	Reage R2	nt 1	Reagent 2 R3	Reagent 3 R4
^o repare Metho	ition Paramet		VOL 10 10	Diluent	Reage	nt 1	Reagent 2	Reagent 3

Fig. 6.7 [Sample Group QA/QC Setup] dialog box

PB must be lower than CRDL, but if CRDL is exceeded, mark the measurement result in accordance with actions (2) and (3).

NOTE

Like LCS, PB is automatically inserted into the MRT worksheet after the wizard has been completed, so far as the corresponding check mark is ticked in the [Sample Group QA/QC Setup] dialog box. To add PB to the already prepared MRT worksheet, [Action] may be specified in any row of the MRT as shown in Fig. 6.8. If the ASC is used, enter the preparation parameters such as position and sample volume.

*	Action	Sample ID	Graph	x	м	Q	
1	STD						I
2	STD						T
3	STD		-				T
4	UNK1						T
5	UNK2						T
6	UNK3						T
7	ныга						T
8	PB 💌						T
9	ICV 🔺						T
10	ccv —						T
11	ICB						t
12	CCB						t
13	PB						t
	PDS						t
	DUP CRA 🔻	-					t
_		y		-		-	t

6.2.6 LCS (Laboratory Control Samples)

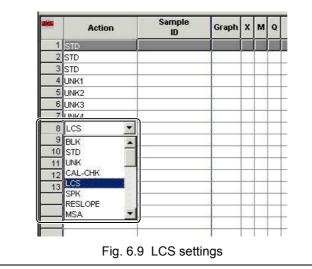
LCS means analyzing a standard substance that has similar liquid properties to those of the actual sample. Primarily, the EPA's LCS is used to analyze such a substance by the same pretreatment and measuring method as those for the actual sample. If it is not available, ICV is used.

When %R, the actually measured value divided by the certificated value is calculated, the reference value must fall within a range between 80% and 120% (exceptions: Ag and Sb). If this range is exceeded, the analysis must be retried starting with the pretreatment.

(1) For the LCS settings, tick the [LCS] check box in the [Sample Group QA/QC Setup] dialog box shown in Fig. 6.7. Enter the known concentration in the preparation parameters table at the bottom of the window. If the ASC is used, also enter the preparation parameters such as position and sample volume.

NOTE

Like PB, LCS is automatically inserted into the MRT worksheet after the wizard has been completed, so far as the corresponding check mark is ticked in the [Sample Group QA/QC Setup] dialog box. To add LCS to the already prepared MRT worksheet, [Action] may be specified in any row of the MRT as shown in Fig. 6.9 and enter the known concentration to the [True Value] field. If the ASC is used, enter the preparation parameters such as position and sample volume.



6.2.7 S (Spike: Spike Test)

S means an addition and recovery test. The EPA classified it into two kinds, SPK where a standard solution is added before the pretreatment and PDS where a standard solution is added after the pretreatment.

Spike Sample Analysis (S) = SPK

This analysis is carried out to obtain information on the effect of matrix on digestion/distillation and measurement.

To perform the digestion/distillation operation, apply spike in advance of adding other reagents.

Apply spike for each group of reagents and each concentration level.

The acceptance criteria require that the spike recovery fall within a range between 75% and 125%. If this range is exceeded, "N" is marked. However, if the sample concentration is more than 4 times the spike concentration, "N" is not required.

PDS

In case of the flame AA (and ICP), if the pre-digestion/pre-distillation spike ratio does not fall within a range between 75% and 125% and if the sample concentration does not exceed 4 times the spike concentration, then carry out post-digestion/post-distillation spike (except for Ag). The added concentration must be 2 times the original sample concentration or 2 times CRDL, whichever higher (PDS is not required for Hg).

Equation for recovery ratio

%Recovery= $\frac{SSR-SR}{SA}$ x100

SSR: Measured value (concentration) of spiked sample

SR: Measured value (concentration) of original (unspiked) sample

SA: Spiked (added) concentration

NOTE

When the same sample as the spiked one is duplicated, use the analysis result of the original sample rather than the mean value for the duplicated one.

The SPK and PDS settings can be entered in the [Sample ID Collective Setup] dialog box displayed by clicking on the [Collective Setup] button in the [Sample Group Setup] Page in Fig. 6.6.

cel
Pos.

Fig. 6.10 [Sample ID Collective Setup] dialog box

- (1) Tick the [Pre-Digestion Spike (SPK)] and [Post-Digestion Spike (PDS)] check boxes and enter a measurement interval (the default value is 20) in the respective right fields. This measurement interval must be smaller than the number of samples.
- (2) Click on the [OK] button to return to the [Sample Group Setup] page. Now [Pre-Digestion Spike (SPK)] and [Post-Digestion Spike (PDS)] are automatically entered in the [Unknown/Spike Measurement Sequence] table of the current sample group number.

To add them to the prepared MRT worksheet, specify "SPK" or "PDS" action in any row of the MRT worksheet and then enter the added concentration as shown in Fig. 6.11. If the ASC is used, enter the preparation parameters such as position and sample volume.

NOTE

The [Unknown/Spike Measurement Sequence] table in the [Sample Group Setup] page shown in Fig. 6.6 also allows you to enter "SPK" and "PDS" actions by performing similar operations.

SPK and PDS must be compared with directly previous UNK. Therefore, specify the preparation parameters accordingly. (Fig. 6.11 shows that SPK will be performed for UNK3.)

	Action	Sample ID	Graph	x	м	Q
1	STD					
2	STD					
3	STD					
4	UNK1					
5	UNK2					
6	UNK3					
7	ныка					
8	SPK 💌					
9	UNK					
10	CAL-CHK 🔤					
11	LCS					
12	SPK					
13	RESLOPE					
	MSA SMSA					
	MSA-RES					
	MORANES					

6.2.8 D (Duplicate Sample Analysis)

Separately apply the pretreatment to the same samples, measure them, and then compare the results with each other. Verify the reproducibility including the pretreatment. Use the following equation to obtain RPD.

RPD=
$$\frac{|S-D|}{(S+D)/2}$$
 x100

RPD=Relative Percent Difference

S=First Sample Value (original)

D=Second Sample Value (duplicate)

If the concentrations of both (S and D) are more than 5 times CRDL, the reference value for RPD is 20% maximum. If either or both of S and D do not reach 5 times CRDL, use the reference value for \pm CRDL (i.e., enter the absolute value for CRDL as the reference value for RPD).

- (1) For the duplication settings, tick the [Duplicate] check box in [Sample ID Collective Setup] dialog box shown in Fig. 6.12 and then enter a measurement interval (the default value is 20) in the right field, as in the case of Spike. This measurement interval must be smaller than the number of samples.
- (2) Click on the [OK] button to return to the [Sample Group Setup] page. Now Duplicate is automatically entered in the [Unknown/Spike Measurement Sequence] table of the current sample group number.

To add it to the prepared MRT worksheet, specify "DUP" action in any row of the MRT worksheet. If the ASC is used, enter the preparation parameters such as position and sample volume.

NOTE

The [Unknown/Spike Measurement Sequence] table in the [Sample Group Setup] page shown in Fig. 6.6 also allows you to enter "DUP" action by performing similar operations.

DUP must be compared with directly previous UNK. Therefore, specify the preparation parameters accordingly.

	Action	Sample ID	Graph	x	м	Q	
1	STD					U.	Q
2	STD						Γ
3	STD						T
4	UNK1						Γ
5	UNK2						T
6	UNK3						T
7	li ikila						T
8							t
9	ICV 🔺	1					Γ
10	ccv 👘	1	1.0				Γ
11	ICB						Γ
12	CCB						Ī
13	PB						t
	PDS						t
	DUP CRA						t
		IJ		-	-		t

Fig. 6.12 Duplicate settings

6.2.9 IDL (Instrument Detection Limit Determination)

IDL is determined as follows.

Repeatedly measure a sample solution of a concentration that is 3 to 5 times higher than IDL, which is announced by the instrument manufacturer, 7 times daily. Obtain the concentration that is 3 times the standard deviation resulting from such measurement runs performed for inconsecutive 3 days (e.g., Monday, Wednesday, and Friday). The repeated measurement runs must be performed by the normal measuring method including rinsing for each measurement run.

This software allows you to automatically evaluate the measurement results of the above items, stop measurement, or mark and continue the measurement. Notice, however, that some of the acceptance criteria do not accurately conform to the EPA's QA/QC reference (e.g., the ratio between the sample concentration and spike concentration at the Spike test and that between the measured concentration and CRDL at the Duplicate test).

Abbreviation	Description
CRDL	Contract Required Detection limit The lower limit of detection required for measurement conforming to EPA (see Table 6.1).
CRA	CRDL standard for AA CRDL standard for atomic adsorption spectrophotometry
LCS	Laboratory Control Samples Standard sample of which composition and concentration are known
РВ	Preparation Blank Standard sample for analysis use containing water and reagents used in analysis process
SPK	Spike Spike of adding a standard solution before digestion
D	Duplicate The second sample that has been pretreated like the original sample to determine the accuracy of the measuring method
ICV	Initial Calibration Verification The first evaluation of the accuracy of a calibration curve immediately after the creation of the calibration curve
CCV	Continuing Calibration Verification Evaluation of sensitivity changes during measurement
ICB	Initial Calibration Blank Calibration blank measurement that is performed immediately after ICV
ССВ	Continuing Calibration Blank Calibration blank measurement that is performed immediately after CCV. At least one reagent blank must be prepared for each sample group or each sample processing batch.
IDL	Instrument Detection Limit Detection limit of the instrument

6.2.10 Abbreviation of QA/QC

ICVS	Initial Calibration Verification Solution Solution for ICV
S	Spike sample analysis SPK and PDS
TV	True Value Self-determination true value
PDS	Post Digestion Spike Spike of adding a standard solution after digestion
RPD	Relative Percent Difference Relative deviation percentage
EPA USEPA	United States Environmental Protection Agency The U.S. Environmental Protection Agency
QA/QC	Quality Assurance/Quality Control
r	Correlation coefficient

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Chapter 7 Hardware Validation

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The hardware validation is intended to check the basic performance of the AA-7000 series, evaluate the results, and then record the validation results together with the date and time.

To ensure the reliability of data obtained from the analytical instrument, it is important to check the performance of analytical instruments periodically, record the data, and take measures such as a correction work based on the check result.

The hardware validation is based on the performance check procedures carried out in our factory, therefore, you can execute the performance check work properly and more easily by utilizing this software.

The necessary tools and samples are shown in Table 7.1 below. These tools and samples are necessary when the standard parameters of hardware validation are used for the performance check.

NOTE

Be sure to prepare the hollow cathode lamp (Hg) that is necessary for the wavelength accuracy check in any cases.

Name	How to get or Shimadzu P/N	Note			
Common tools for flame system/	Common tools for flame system/ furnace system				
Hollow cathode lamp (Hg)					
Hollow cathode lamp (Se) S200-38422-46					
Tools and samples for flame sys	tem				
Hollow cathode lamp (Cu)	S200-38422-08				
Cu 2ppm Standard sample	Purchase a commercially available one.	Dilute the 1000 ppm standard solution for atomic absorption spectrophotometry.			
Tools and sample for furnace sy	stem				
Hollow cathode lamp (Mn)	S200-38422-13				
Pyrolytic coated graphite tube	S206-50588				
Mn 1ppb Standard sample	Purchase a commercially available one.	Dilute the 1000 ppm standard solution for atomic absorption spectrophotometry.			

Table 7.1 Items required for hardware validation

7.3

The test items in the hardware validation and their outlines are introduced.

For the default tolerance limit for each test item, see 10.1.4 "Performance Specifications".

7.3.1 Wavelength Accuracy

The spectral lines emitted from the mercury hollow cathode lamp are measured and the accuracy is checked from the difference between the reading of each spectral line and the resonance wavelength.

253.65 365.01	435.84	546.08	585.25(Ne)	640.22(Ne)	724.52(Ne)
---------------	--------	--------	------------	------------	------------

Note: (Ne) shows the Neon line.

Necessary tool

Hollow cathode lamp (Hg)

7.3.2 Baseline Drift

Light ON the hollow cathode lamp (Cu) and record variation of signal at the analysis wavelength of Cu around 0 Abs absorbance for a given time. Measure the amount of change of baseline to determine the baseline drift.

Necessary tool

Hollow cathode lamp (Cu)

7.3.3 Noise Level

(1) NON-BGC noise level

The signal changes at first resonance line (196.0 nm) of the hollow cathode lamp (Se) are recorded in the neighborhood of 0 Abs of absorbance for given time. The amplitude of absorbance for this duration is taken as the NON-GBC noise level.

(2) BGC-D2 noise level

The changes in signal difference (BGC-D2 signal) at the wavelength of Se (196.0 nm) of the hollow cathode lamp (Se) and the D2 lamp are recorded in the neighborhood of 0 Abs of absorbance for given time. The amplitude of absorbance for this duration is taken as the BGC-D2 noise level.

Necessary tool

Hollow cathode lamp (Se)

NOTE

Acetylene gas is purged from within the instrument after the automatic gas leakage inspection is completed successfully. This operation is performed to guarantee the reliability of the instrument. Although trace amounts of purged acetylene gas are not dangerous, a minute amount of the purged gas may remain on the optical axis for 30 minutes after the gas leakage inspection is complete. If a noise level inspection is performed during this period, the noise level will deteriorate due to the effects of absorption caused by the gas.

This problem can be avoided by performing inspections in the order of wavelength accuracy, baseline drift, and noise level according to the standard procedure.

7.3.4 Absorbance and Repeatability (In the Case of Flame Analysis)

The absorbance of the Cu standard solution is measured with the air-acetylene flame and the average absorbance of 5-time repeated measurements is treated as the absorbance. The coefficient of variation (RSD) of these 5-time absorbance values is calculated and this is treated as the repeatability. Confidence coefficient is 95% for the repeatability.

 Necessary tool and reagents Hollow cathode lamp (Cu) Cu 2 ppm standard solution Distilled water

7.3.5 Detection Limit (In the Case of Flame Analysis)

Use the air-acetylene flame to measure the absorbance of the sample used to verify the detection limit (blank solution or standard solution with a concentration that is 3 to 5 times higher than the concentration of the expected detection limit). Repeat this measurement the specified number of times to calculate the standard deviation, and take this value as "s". Take as "A" the mean value for the absorbance of the standard solution of Cu 2.0 ppm, which was obtained in 7.3.4 "Absorbance and Repeatability (In the Case of Flame Analysis)". Use the following equation to obtain the Cu concentration for which absorbance is equivalent to 3 times higher than the standard deviation of the measured values for the sample used to verify the detection limit. Take the obtained value as the detection limit.

Detection limit = $(2.0 \times 3 \times s)/A$

· Necessary tool and reagents

Hollow cathode lamp (Cu)

Sample used to verify detection limit (blank solution or standard solution with a concentration that is 3 to 5 times higher than the concentration of the expected detection limit) Distilled water

7.3.6 Stability (In the Case of Flame Analysis)

Use the air-acetylene flame to continuously record the absorbance of the Cu standard solution (for integration time of 30 seconds) and measure the maximum amplitude of absorbance values. Take the ratio of the maximum amplitude (w) to the measured value (B) for the recorded absorbance as the fluctuations of the reading.

Stability = w/B

 Necessary tool and reagents Hollow cathode lamp (Cu) Cu 2 ppm standard solution Distilled water

7.3.7 Absorbance and Repeatability (In the Case of Furnace Analysis)

The pyrolytic coated tube is set in the graphite furnace and the absorbance is measured when 20 μ L of the Mn 1 ppb standard solution is injected. The average absorbance of 5-time repeated measurement is treated as the absorbance. The coefficient of variation (RSD) of these 5-time absorbance values is also calculated and this is treated as the repeatability.

- Necessary tools and reagents
 - Hollow cathode lamp (Mn)
 - Mn 1 ppb standard solution
 - Pyrolytic coated graphite tube
 - Pipette (For 20 μ L injection. Not necessary if you have the autosampler ASC-7000) Distilled water

7.3.8 Detection Limit (In the Case of Furnace Analysis)

Set the pyrolytic coated graphite tube in the graphite furnace and measure the absorbance of the sample used to verify the detection limit (blank solution or standard solution with a concentration that is higher than 3 to 5 times than the concentration of the expected detection limit). Repeat this measurement the specified number of times to calculate the standard deviation, and take this value as "s". Take as "A" the mean value for the absorbance of the standard solution Mn 1.0 ppb, which was obtained in 7.3.7 "Absorbance and Repeatability (In the Case of Furnace Analysis)".

Use the following equation to obtain the Mn concentration for which absorbance is equivalent to 3 times higher than the standard deviation of the measured values for the sample used to verify the detection limit. Take the obtained value as the detection limit.

Detection limit = $(1.0 \times 3 \times s)/A$

· Necessary tools and reagents

Hollow cathode lamp (Mn)

Sample used to verifying detection limit (blank solution or standard solution with a concentration that is 3 to 5 times higher than the concentration of the expected detection limit)

Pyrolytic coated graphite tube

Pipette (capacity: 20 $\mu\text{L};$ not required if you have the autosampler ASC-7000) Distilled water

7.4

Basic Operation Procedures

In this section, the basic operation procedures are described. When changing the parameters, refer to the section 7.5 "Parameter Change".

7.4.1 Preparation

7.4.1.1 Preparation of Hollow Cathode Lamp

First, set the hollow cathode lamps to the AA main unit referring to Table 7.3 below.

Hollow cathode lamp	Socket No.	Note
Hg	1	Used for accuracy of wavelength
Se	2	Used for noise level
Cu	3	Used for baseline drift and flame analysis
Mn	4	Used for furnace analysis

Table 7.3 Hollow cathode lamp positions

7.4.1.2 Sample Preparation

(1) In the case of flame analysis

When using the autosampler, set the turntable for flame analysis to the ASC and place the samples as the table below.

Table 1	7.4	Test	of	flame	analysis
---------	-----	------	----	-------	----------

Sample	Turntable position
Distilled water	R0
Blank solution	R1
Cu 2.0 ppm Standard solution	R2
Sample used to verify detection limit:	R3

In the case of manual measurement, prepare the above samples in the measuring flasks or beakers.

(2) In the case of furnace analysis

In the case of furnace analysis, set the turntable for furnace analysis to the ASC and place the samples as the table below.

Table 7.5 Test of furnace analysis

Sample	Turntable position
Blank solution	R1
Mn 1.0 ppb Standard solution	R2
Sample used to verify detection limit:	R3

In the case of manual measurement, prepare the above samples in the measuring flasks or beakers and prepare a pipette for sample injection (with which 20 μ L injection is possible).

Set a pyrolytic coated graphite tube in the graphite furnace.

7.4.2 Selecting the Test Items

(1) After the preparation for the instruction has been finished, select [Programs] - [WizAArd] - [Hardware Validation] from the Start menu to start the software.

NOTE

Alternatively, you may want to double-click on the AA hardware validation icon on the Desktop.

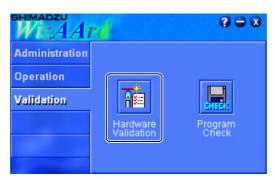


Fig. 7.1 [AA Hardware Validation Login] dialog box

(2) After the software has been started, the following login dialog box is opened allowing you to log in.

AA Hardware Va	lidation Software Login	
	WizAArd	
Login ID		OK
Password		Cancel

Fig. 7.2 [AA Hardware Validation Login] dialog box

After the login has been completed, the window shown in Fig. 7.3 is displayed.

🏫 AAYAL - AA Hardware Validation Software (System Administrator)	
<u>File Tests Configuration Perform Graph Window Help</u>	
Test Item:	100.00
WL Accuracy 253.65nm	1
WL Accuracy 365.01nm	50.00 +
WL Accuracy 435.84nm WL Accuracy 546.08nm	‡
WL Accuracy 546.06nm WL Accuracy 585.25nm	25.00
WL Accuracy 640.22nm	25.00
WL Accuracy 724.52nm	±
Baseline Drift	0.00 + + + + + + + + + + + + + + + + + +
NON-BGC Noise Level	0.00 50.00 100.00
BGC-D2 Noise Level	
Absorbance/Rep.Accur.(Flame)	-
Limit SD Testing 1 (Flame)	
Limit SD Testing 2(Flame)	
Limit Result(Flame)	
Stability(Flame) Absorbance/Rep.Accur.(Furnace)	
Limit SD Testing 1(Furnace)	
Limit SD Testing 2(Furnace)	
Limit Result(Furnace)	
[],	

Fig. 7.3 [AA Hardware Validation] window

The test items that can be validated through the hardware validation are roughly classified as follows:

a. Common test items involving no sample measurement

Wavelength accuracy NON-BGC and BGC-D2 noise levels Baseline drift

b. Flame method test items involving sample measurement

Absorbance/Repeatability Detection limit Stability

 c. Furnace method test items involving sample measurement Absorbance/Repeatability Detection limit

The items in group (a) are automatically proceed, but those in groups (b) and (c) proceed semi-automatically or interactively.

No flame is ignited for the items in group (a).

If the ASC is used for the items in groups (b) and (c), the procedure for nozzle position adjustment is performed before the tests.

The procedures to select the test items are described as follows.

(1) Select [Tests]-[Modify Batch] in the menu bar. Or click on the 🖹 in the tool bar.

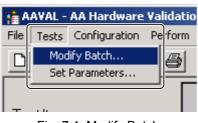


Fig. 7.4 Modify Batch

The dialog box for [Modify Batch] is displayed. The inspection items indicated in the "Item to Perform" will be executed.

NOTE

Since all the test items are displayed in the right-side column "Item to Perform" on the screen when sent from the factory, delete unnecessary items for execution from this table.

(2) Click the item, which is not to be executed.

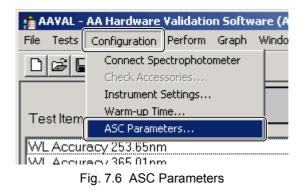
Modify Batch	×
Selectable Item	Add> Add> WL Accuracy 253.65nm WL Accuracy 253.65nm WL Accuracy 355.01nm WL Accuracy 358.84nm WL Accuracy 546.08nm WL Accuracy 585.25nm WL Accuracy 540.22nm WL Accuracy 724.52nm Baseline Drift NON-BGC Noise Level BGC-D2 Noise Level BGC-D2 Noise Level Limit SD Testing 1(Flame) Limit SD Testing 2(Flame) Limit SD Testing 1(Flame) Stability(Flame) Absorbance/Rep.Accur.(Furnace) Limit SD Testing 1(Furnace) Limit SD Testing 1(Furnace) Limit Result(Furnace) Limit Result(Furnace)
	DK Cancel

Fig. 7.5 [Modify Batch] dialog box

- (3) Press the [Delete] button at the center on the screen with the mouse. The selected items are moved to the left column [Selectable item].
- (4) When adding an item from the [Selectable item] to the [Item to Perform], select the item to add with the mouse and press the [Add] button at the center of the screen with the mouse.
- (5) With the above procedures, select the items for execution and press the [OK] button with the mouse. The [Modify Batch] dialog box will be closed.

7.4.3 Setting the ASC Parameters

(1) When using the autosampler ASC to execute the tests for absorbance, repeatability and detection limit, select the [Configuration]-[ASC Parameters] in the menu bar to open the [ASC Parameters] dialog box.



(2) Click on the check box to make the ASC connection operative.

ASC Parameters			×
Use ASC in Flame Mea	surement		
Absorbance/ <u>R</u> epeat Accuracy:	Blank R1 🔻	Sample R2 💌	
Detection Limit:		R2 💌	
<u>S</u> tability:	R1 💌	R2 🔻	
Use ASC in Furnace Me	asurement)	
	Blank	Sample	
Absorbance/Repeat Accuracy:	R1 💌	R2 💌	
Detection Limit:		R2 💌	
ОК	Cano	el	

Fig. 7.7 [ASC Parameters] dialog box

NOTE

- Without this setting, the ASC cannot be operated even if the ASC is connected to the AA main unit.
- If the ASC is used, match the position settings for [Blank] and [Sample] with the position of the ASC turntable for the sample under measurement. In 7.4.1.2 "Sample Preparation", the position of the turntable for the sample used to verify the detection limit is specified as "R3". Therefore, also specify "R3" for the sample used to verify the lower detection limit in the [ASC Parameters] dialog box.

Refer to 7.4.1.2 "Sample Preparation".

7.4.4 Setting the Warm-up Time of Lamps and Stabilization Time for Flame

Set the warm-up lighting time of the hollow cathode lamp and D_2 lamp when starting the performance check. If the lamps are not stable, they may cause a drift and noises.

It is recommended to prepare the warm-up time longer than approx. 10 to 15 minutes in the case of hollow cathode lamp, and the warm-up time for approx. 5 to 10 minutes in the case of D_2 lamp.

Also, in the case of tests for Absorbance, repeatability, detection limit and stability with the flame analysis, the waiting time for starting the data acquisition after the flame ignition can be set. Since the temperature of the burner head is not constant right after the flame ignition, the flame temperature is not stable and it may cause a sensitivity drift or instability. It is recommended to prepare the stabilization time at least longer than 1 minute after the flame ignition.

(1) Select [Configuration]-[Warm-up Time] in the menu bar.

The [Lamp Warm-up Parameters] dialog box is displayed.

Lamp Warm-up Parameters	×			
HCL Lamp(min) D2 Lamp(min) Flame(min)	10 5 10			
The lamp warm-up settings are not available for the inspection of Wavelength Accuracy.				
OK Car	ncel			

Fig. 7.8 [Lamp Warm-up Parameters] dialog box

(2) Input the stabilization time for each item and press the [OK] button.

The waiting condition continues during the input time, which works as the warm-up time in the case of lamp and as the measurement waiting time (burner head preheating time) in the case of flame.

7.4.5 Starting the Test

Connecting the communication between AA main unit and PC. The AA main unit must be previously turned on. When using the ASC and GFA, switch on the power and complete the initialization beforehand.

NOTE

If another WizAArd software for controlling AA main unit is already connected for communication, firstly finish the connection with the WizAArd software, then connect this validation program.

(1) Select [Configuration] - [Connect Spectrophotometer] in the menu bar.



Fig. 7.9 Configuration menu

When the connection is started, the AA main unit is initialized. During initialization of the instrument, carry out inspection of the safety devices.

For AA-7000F and AA-7000F/AAC, see 3.1.6.1 "Initializing the Instrument".

For AA-7000G, see 3.2.6.1 "Initializing the Instrument".

(2) When all the items on the initialization screen are indicated in green, click on the [OK] button.

nitialize			
	AA : ASC: GFA:	AA-7000 ∨1.01 A300000000 ASC-7000 ∨1.01 A30000000 GFA-7000 ∨1.02 A30000000	000
\bigcirc	ROM Check		C2H2 Valve Origin Search
\bigcirc	S/N Check		Flame Monitor Check
\bigcirc	ASC Check		
\bigcirc	GFA Check		Burner Select Sensor Check
\bigcirc	Slit Origin Se	arch	🔵 Drain Sensor Check
\bigcirc	D2 Attenuato	r Origin	Support Gas Pressure Monitor Check(Air)
\bigcirc	Wavelength	Origin Search	
\bigcirc	Turret Origin	Search	Fuel Gas Pressure Monitor Check
\bigcirc	Atomizer Up,	/Down	🔵 Start Gas Leak Check
\bigcirc	Atomizer For	e/Back	
	Testing	Success	► Failure No Test(Not Connected)

Fig. 7.10 [Initialize Instrument] screen

The main screen appears again. Before starting the tests, check that the printer is connected to the PC correctly, the power is turned on and the printer paper is set.

(3) Click on the 🔄 in the tool bar as shown in the right figure, or select [Perform]-[Start] in the menu bar.



The dialog box for inputting the room temperature opens.

(4) After inputting temperature, press the [Start]. Then the test starts. The items in the dialog box (Fig. 7.10) will be printed on the check result, which is output after the test.

Start Inspection					
AA: AA-7000 v1.01 A3000000000					
ASC: ASC-7000 v1.01 A3000000000					
GFA: GFA-7000 v1.02 A3000000000					
Date: Monday, February 22, 2010					
Temp. of Room: 25 degrees C	Start				
Analyst Name: System Administrator	Cancel				

Fig. 7.12 Inputting the room temperature

The validation proceeds from the top of the test items. The result for each item is indicated on the right of each item in the test item column as [OK] or [NG].

The following indications are displayed according to the result and conditions.

- [OK] : As the check result, the tolerance limits is satisfied.
- [NG] : As the check result, the tolerance limits is not satisfied.
- [ERROR] : The test could not be completed normally due to an error.

[ABORT] : The measurement was stopped during the test.

Blank : The test is not performed yet.

The tolerance limits and measurement value, result of OK or NG, and message showing the condition of test are indicated on the lower right part of the screen.

Whenever one test item is completed, the measurement data is output to the printer.

📬 AAVAL - AA Hardware Validation Software (System Administrator)	
Eile Tests Configuration Perform Graph Window Help	
DFN BY 5	
Test Item:	E 0.50
WL Accuracy 253.65nm OK WL Accuracy 365.01nm OK WL Accuracy 435.84nm Performing WL Accuracy 546.08nm WL Accuracy 546.08nm WL Accuracy 585.25nm WL Accuracy 540.02nm WL Accuracy 724.52nm Baseline Drift NON-BGC Noise Level BGC-D2 Noise Level BGC-D2 Noise Level BGC-D2 Noise Level Limit SD Testing 1(Flame) Limit SD Testing 2(Flame) Limit SD Testing 2(Flame) Limit SD Testing 1(Flame) Limit SD Testing 1(Flame) Limit SD Testing 2(Furnace) Limit SD Testing 2(Furnace) Limit Result(Flame)	0.25 0.00
) Executing Line Search	ASC GFA BUSY
	proc part poor

Fig. 7.13 Screen during test

7.4.6 Items with Sample Measurement

Sample measurements are necessary for the items such as absorbance, repeatability, detection limit and stability for flame analysis and absorbance, repeatability and detection limit for furnace analysis. They are set in the following order through the dialog.

Before starting these test items, prepare the necessary samples beforehand. When using the autosampler, set the prepared samples to the specified positions on the turntable (Refer to 7.4.1.2 "Sample Preparation").

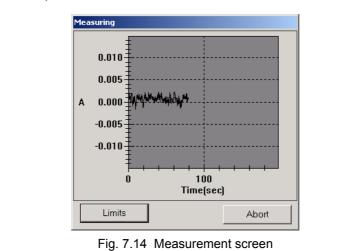
- In the case of flame analysis
 - 1. Positioning of ASC sampling nozzle (when using the ASC)
 - 2. Flame ignition
 - 3. Sample suction
- · In the case of furnace analysis
 - 1. Positioning of ASC sampling nozzle (when using the ASC)
 - 2. Sample injection

7.4.7 Stop the Test

(1) When stopping all the tests, select [Perform]-[Stop] in the menu bar or click on 💓 in the tool bar.

NOTE

In the case of test items except the wavelength accuracy, the [Abort] button is on the measurement screen as shown in Fig. 7.14 below. Clicking on this button stops the current measurement. In the case of furnace measurement, inspection stops after the current measurement finishes.



7.4.8 Print the Check Result

When completing the entire test, the check result can be output as the summary report.

(1) When the selected test items are finished, select [File]-[Print Summary Report] in the menu bar or click on in the tool bar. The tolerance limits, check results and OK / NG judgment can be output together as the result report (Refer to Fig. 7.15).

Only when all the results satisfy the tolerance limits, the total result judgment is "Passed".

A Hardware Validation Report	Wednesda	y, February 10, 2	010 5:11:14 I	PM(+090
AA:	AA-7000 v1.01 A30000000000			
ASC:	ASC-7000 v1.01 A3000000000			
GFA:	GFA-7000 v1.02 A3000000000			
Date:	Wednesday, February 10, 2010	3:22:56 PM(+090	00)	
Temp. of Room:	25degrees C			
Analyst Name:	System Administrator			
Test Item	Contents	Tolerance:	Value	Result
Wavelength Accuracy	Hg	+/-0.30nm		
	Slit0.2nm			
	253.65nm		253.72nm	Passed
	365.01nm		364.98nm	Passed
	435.84nm		435.78nm	Passed
	546.08nm		546.10nm	Passed
	585.25nm		585.29nm	Passed
	640.22nm		640.20nm	Passed
	724.52nm		724.42nm	Passed
Baseline Drift	Cu	0.0050Abs	0.0024Abs	Passed
	Slit0.2nm			
	324.8nm			
NON-BGC Noise Level	Se	0.0100Abs	0.0038Abs	Passed
	Slit0.7nm			
	196.0nm			
BGC-D2 Noise Level	Se	0.0150Abs	0.0069Abs	Passed
	Slit0.7nm			
	196.0nm			
Absorbance(Flame)	Cu2.0ppm5times AVE	0.2300Abs <=		Not Ye
Rep. Accuracy(Flame)	Cu2.0ppm5times CV	2.00%		Not Ye
3 Sigma Limit(Flame)	Sample	0.01ppm		
	Repeat Times	7time[s]		
	SD	0.00000		
	SD	0.00000		
	SD	0.000000		
	Average	0.000000		
	Line: Absorbance Result Used			
		0.00600ppm	0.00000ppr	nNot Ye
Stability(Flame)	Cu2.0ppm 30sec	6.0%		Not Ye

Certified by:

C:/Program Files\Shimadzu\WizAArd\Optics.vld

Page 1

Fig. 7.15 Summary Report

7.4.9 Saving and Loading the Data

7.4.9.1 Saving the Data

The test data can be saved as a validation file.

- (1) Select [File]-[Save As] in the menu bar. Then the dialog box [Save As] opens.
 - Input the file name and click on the [OK]. Be sure to use "*.vld" for the extension of file name. The test result is saved together with the test item and its parameters.

7.4.9.2 Loading the Data

The saved file can be called by [File]-[Open].

(1) The dialog box [Open] opens. Select the file to be called and click on [OK]. When the file is read in, the data of each test item can be checked on the screen and the data can be output to the printer.

7.5 Parameter Change

The test parameters can be changed except when the test item is the wavelength accuracy.

7.5.1 Entering the Parameter Change Screen

(1) Select [Tests]-[Set Parameters] in the menu bar to change the test parameters.

The A	AVAL -	AA Hardware	Validatio
File	Tests	Configuration	Perform
Modify Batch			
	Set Parameters		

Fig. 7.16 Parameter change execution

The dialog box [Set Parameters] as shown in Fig. 7.17 opens.

(2) Select the test item whose parameter is to be changed from the dialog box [Set Parameters] and doubleclick on it, or move the cursor to the item and press the [Edit]. The dialog box for setting the parameters opens.

Set	Set Parameters 🛛 🔀		
]	[est ltem:		
	WL Accuracy 253.65nm		
	WL Accuracy 365.01nm		
	WL Accuracy 435.84nm		
	WL Accuracy 546.08nm		
	WL Accuracy 585.25nm		
	WL Accuracy 640.22nm		
	WL Accuracy 724.52nm		
	Baseline Drift NON-BGC Noise Level		
	RGC-D2 Noise Level		
	Absorbance/Rep.Accur.(Flame)		
	Limit SD Testing 1(Flame)		
	Limit SD Testing 2(Flame)		
	Limit Result(Flame)		
	Stability(Flame)		
	Absorbance/Rep.Accur.(Furnace)		
	Limit SD Testing 1(Furnace)		
1	Limit SD Testing 2(Furnace)		
<u> </u>	Limit Result(Furnace)		
1			
	Close <u>E</u> dit		

Fig. 7.17 Setting the test parameters

NOTE

When the measured data exists, the parameter cannot be changed. In this case execute [File]-[New] in the menu bar to clear all the data.

7.5.2 Setting the Test Parameters

The test parameters can be set for each test item except the wavelength accuracy.

When the test item whose parameter is to be changed is selected in the above procedures, the dialog box for setting the test parameter according to each item opens (refer to the example in Fig. 7.18).

Noise Level Test Par	ameters			×
<u>W</u> avelength:	196.0	nm	ОК	
<u>T</u> olerance:	0.0150	Abs.		
			Cancel	
E <u>l</u> ement:	Se			
Socke <u>t</u> #:	2			
L <u>a</u> mp Current Low	c 23	mA		
Sl <u>i</u> t Width:	0.7 💌	nm		
<u>R</u> esponse:	1 💌	[

Fig. 7.18 Noise Level Test Parameters

NOTE

- 1. When the element name of the hollow cathode lamp is input, the typical set wavelength and lamp current are set. When changing the set wavelength and lamp current, change them after inputting the element name.
- 2. When changing the lamp socket No., don't use the same socket No. for different hollow cathode lamps.
- 3. Also, since the mercury (Hg) hollow cathode lamp is usually set to the socket No. 1, select the No. from No.2 to 6. in the case of the test items except the wavelength accuracy test.
- 4. "Detection limit" is calculated with the result from "Absorbance/ Repeatability". Set the same element for these two inspections. When they have the different element set, "Detection limit" will not be calculated properly.

The test parameters, which can be set for each item, are as follows.

Test Item	Parameter
NON-BGC noise level BGC-D2 noise level Baseline drift	 Wavelength setting Lamp (element) Socket number and lamp current Slit width Response Acceptance criteria

Test Item	Parameter
Test items involving flame measurement (Absorbance, repeatability and stability)	 Wavelength setting Lamp (element) Socket number and lamp current Slit width Lamp mode Pre-spray time and integration time Response Burner height Flow rate of fuel gas Flow rate of support gas Concentration of sample used for measurement Acceptance criteria
Test items involving flame measurement (Detection limit)	 Wavelength setting Lamp (element) Socket number and lamp current Slit width Lamp mode Pre-spray time and integration time Response Burner height Flow rate of fuel gas Flow rate of support gas Concentration of sample used for measursement Acceptance criteria Calculation method Repetition times
Test items involving furnace measurement (Absorbance and repeatability)	 Wavelength setting Lamp (element) Socket number and lamp current Slit width Lamp mode Signal processing Injection volume and injection speed GFA tube type GFA heating temperature program Concentration of sample used for measurement Acceptance criteria
Test items involving furnace measurement (Detection limit)	 Wavelength setting Lamp (element) Socket number and lamp current Slit width Lamp mode Signal processing Injection volume and injection speed GFA tube type GFA heating temperature program Concentration of sample used for measurement Acceptance criteria Calculation method Repetition times

7.5.3 Setting the Scale for Graph Display/Output of Each Data

For each test item except the wavelength accuracy, the graph ordinate scale for graph display/output of the test data can be set.

- (1) Select [Graph]-[Set Y-Axis in Each Test], then the dialog box "Set Y Axis Limits" as shown in Fig. 7.19 opens.
- (2) Double-click on the test item to be changed with mouse or move the cursor to click on the [Change].

Set Y Axis Limits	×
<u>T</u> est Item:	
NON-BGC Noise Level BGC-D2 Noise Level Baseline Drift Absorbance/Rep.Accur.(Flame) Limit SD Testing 1(Flame) Limit SD Testing 2(Flame) Limit Result(Flame) Stability(Flame) Absorbance/Rep.Accur.(Furnace) Limit SD Testing 1(Furnace) Limit SD Testing 2(Furnace) Limit Result(Furnace)	
Close Change	

Fig. 7.19 Set Y Axis Limits

The dialog box to input the upper and lower limits of the ordinate scale opens. Input the values so that the range for display/output may be in accordance with the tolerance limits.

Set Y Axis Limits		×
Absorbance/Rep	o.Accur.(Flame)	
<u>M</u> ax: Min:	0.8000	
OK	Cancel	

Fig. 7.20 Input Scale

7.5.4 Saving/Loading the Parameters

There are two methods to save the conditions for test item after changing the parameters. One is to save them as an individual validation file, and the other is to save them as a validation file, which is read in automatically when the validation program is started.

The contents saved as the information specific to the file are shown as follows.

- a. Test item to be executed
- b. Test parameters for each test item
- c. Ordinate scale for graph display/output
- d. Test result

NOTE

The parameters which can be set in the [Configuration] in the menu bar (preheating time set, ASC setting) are not saved as the parameters specific to the file. When the program is started, these parameters are set to the same condition that is set at the previous time.

7.5.4.1 When Saved as the Individual Validation File

(1) After setting the test items and test parameters, select [File]-[Save As] in the menu bar.

Then the dialog box [Save As] opens.

(2) Input the file name (The file name extension must be *.vld) and click on [OK] to save the test items and their parameters.

7.5.4.2 Loading the Individual Validation File

- (1) The saved file can be loaded by the [File]-[Open]. Then the dialog box [Open] opens.
- (2) Select the validation file to be loaded and click on [OK].

NOTE

It is convenient to use different files according to the test content differences. For example, you can save a validation file used for monthly performance check and a validation file for semiannual performance check separately.

7.5.4.3 When Saved as the Auto Load Validation File

NOTE

Since there is a default (initial condition just after installation) validation file "autoload.vld" as read-only, rename "autoload.vld" to "autoload.org" before this operation.

(1) After setting the test items and test parameters, select [File]-[Save As] in the menu bar.

Then the dialog box [Save As] opens.

(2) Select the folder where the validation program execution file "aaval.exe" exists (usually ":\Program Files\Shimadzu\WizAArd") first, then input the file name "autoload.vld" and click on [Save].

Save As		<u>? ×</u>
Save in: ն	WizAArd 💽 🖨 🛗 🖽 -	
Export Log Manual System Work autoload.vl	d	
File <u>n</u> ame:	autoload.vld	
Save as <u>t</u> ype:	Validation File(*.vld)	<u>الا</u>

Fig. 7.21 Autoload file setup

From the next time, the newly set "autoload.val" will be automatically read in when the validation software is started up.

NOTE

When setting the start-up validation file back to the default, delete the "autoload.vld" file, and then rename "autoload.org" to "autoload.vld".

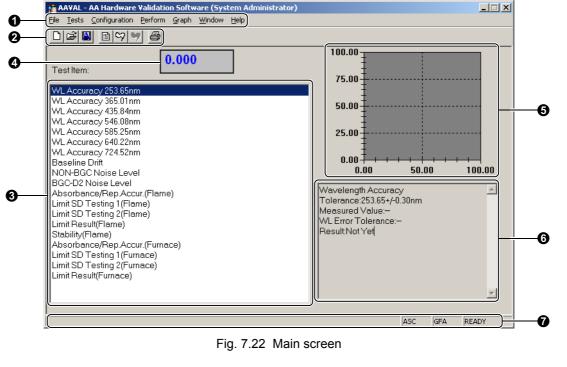
Functions of Operation Menu

In this chapter, the items displayed on the screen and their functions are described.

7.6.1 Main Screen

Starting the program will display the following screen.

The items on this screen are described here.



- 1 This is called menu bar, which shows a basic menu. Set the mouse cursor onto the function indication (File etc.) and click on the left button of the mouse to select.
- 2 This is called tool bar, which used to shortcut selecting the same functions as the commands selected from the Menu bar.

The each tool function is shown as below.

D	This is used to create a validation file.
È	This is used to read out a validation file.
	This is used to save a validation file.
	This is used to select test items.
5	This is used to execute the test.
4	This is used to stop the test.
4	This is used to print out the test result.

- 3 This indicates the available test items. The test is executed according to the order indicated here. After executing the test, the result OK / NG is indicated. The data of the finished test items can be checked by selecting the item with double-clicking.
- The current absorbance value is indicated at the time of measurement.
- **G** The measured data profile is displayed here.
- The detail information of the test result is indicated here. The tolerance limits and measured values, the result of OK / NG judgment and the message showing other test conditions are indicated.
- This is called status bar, which indicates the current instrument working conditions, messages and connections of the peripheral equipment (ASC, GFA).

7.6.2 File

Selecting [File] in the menu bar will show the screen as Fig. 7.23. The functions related to the validation files are collected.

The validation file is a file with extension ".vld" and contains the test items, measurement parameters and test result data.

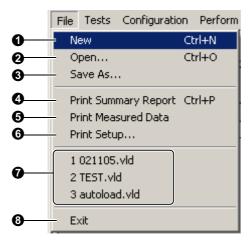


Fig. 7.23 File menu

0	[New]	This is used to create a new inspection file.
		If the inspection file named "autoload.vld" has been created in the same folder (normally,:\Program Files\Shimadzu\WizAArd) where the "aaval.exe", execution file of this program, exists, the "autoload.vld" is read in. After executing this, all the results of tests executed before are deleted from the memory.
0	[Open]	This is used to read in a validation file existing on the disk.
€	[Save As]	This is used to save the current validation file with a new name.
4	[Print Summary Report]	The test result is output to the printer.
6	[Print Measured Data]	All the measured data of each test item are output to the printer.
6	[Print Setup]	Settings related to the printer are set up. Selecting this function will open the dialog box [Print Setup]. See 7.6.2.1 "[Print Setup] Dialog Box".

0	[File History]	Maximum four validation files, which are lately read in, can be displayed. Click on the displayed file name. Then the validation file can be read in.
8	[Exit]	This is selected to close this program. If the test data is not saved, the warning message verifying that you wish to close this program is indicated.

7.6.2.1 [Print Setup] Dialog Box

	Print Setup			<u>? ×</u>	
	Printer —				
0	<u>N</u> ame:	HP LaserJet 4P	•	Properties	-0
	Status: Type: Where:	Ready HP LaserJet 4P LPT1:			
	Comment:				
0 —	Paper		- Orientatio		-4
	Si <u>z</u> e: <u>S</u> ource:	Automatically Select	A	 Portrait Landscape 	
	Net <u>w</u> ork		(OK		

Fig. 7.24 Print Setup

0	[Name]	Normally, the printer set here is used. Set the printer name, which agrees with the printer you use.	
0	[Properties]	The printing conditions are set here.	
8	[Paper]	The paper size and paper feed method are selected.	
4	[Orientation]	The portrait / landscape print direction is selected.	

7.6.3 Tests

Under this menu, the functions related to the setup of the test items to be executed are collected.

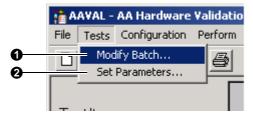


Fig. 7.25 Test menu

0	[Modify Batch]	This is used to select the test items to be executed.
0	[Set Parameters]	This is used to change the test parameters for each test item. However, the parameters for the wavelength accuracy items cannot be changed. The parameters cannot be changed if the test data exists.

7.6.4 Configuration

Under this menu, the functions to set up the conditions for executing this software are collected.





Fig. 7.26 Configuration menu

0	[Connect Spectrophotometer]	The communication between the AA main unit and the PC are set ON/ OFF. Start the instrument before setting the communication ON. For details, see 2.1 "Power ON/OFF". Selecting this function after starting the instrument sets the communication ON and indicates the check mark on the head of the menu. When setting OFF the communication, select this function again.
0	[Check Accessories]	The communication between the AA main unit and the peripheral equipment (ASC, GFA) is set ON.
0	[Instrument Settings]	This is used to set up the serial port of the PC used for connection with the AA main unit.
0	[Warm-up Time]	The lamp warm-up time and the waiting time after flame ignition during the tests are set.
0	[ASC Parameters]	This is used to select whether the ASC is used for the test or not and to set the sample position when using the ASC.

7.6.5 Perform

This is the menu to start or stop the menu.

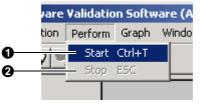


Fig. 7.27 Perform menu

0	[Start]	This is used to start the test. Before starting, it is necessary that the connection for communication between the PC and the AA main unit is completed.
0	[Stop]	This is used to stop the test under execution halfway.

7.6.6 Graph

This is the menu to set up the graph scale when the result data of each test is displayed or output.

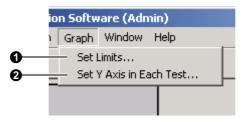


Fig. 7.28 Graph menu

0	[Set Limits]	The result of the finished test item is displayed on the main screen as the profile graph. This is used when changing the graph ordinate/ abscissa scale for the data currently displayed on the main screen. The scale values are valid only right after they are set. If another item graph is displayed once, the scale returns to the initial condition.
0	[Set Y Axis in Each Test]	This is used to set up the initial scale values of the ordinate when the graph of each test result is displayed or output. They can be set for each test item except the wavelength accuracy. The scale values set here are saved together with the test parameters as the initial ordinate values for the graph display and printer output when the file is saved (Refer to the section 7.5.3 "Setting the Scale for Graph Display/Output of Each Data").

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Chapter 8 Maintenance

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In order to keep the instrument in good condition, carry out routine inspections and periodic inspections.

8.1.1 Routine Inspections

The routine inspection that must always be carried out before using the instrument is described here.

WARNING

• The items with \bigcirc marked in the "Safety" column are the inspection points that relate to safety. Failure to complete these inspection points may result in gas leaks or flashback.

8.1.1.1 Common to All AA-7000 Series Models

Safety	Inspection Point	Details	Remarks
	Unit surface	Wipe off soiling on the surface of the instrument with a soft dry cloth. If the soiling is severe, wipe it off with a cloth that has been soaked in a diluted neutral detergent and well wrung out. Do not use organic solvents for cleaning since they can damage plastics and painted surfaces.	

8.1.1.2 AA-7000F, AA-7000F/AAC

Points to check before ignition

Safety	Inspection Point	Details	Remarks
0	Burner head	If there is clogging in the burner slot, wash it free.	See 8.2.1 "Burner Head Maintenance".
0	Nebulizer	If the nebulizer fixing screws are loose, tighten them up.	See Fig. 8.2.
0	Drain tank	Check the water level in the drain tank. If it has dropped, supply water.	See 2.8 "Supplying Water to the Drain Tank (AA-7000F, AA-7000F/AAC)".
0	Waste liquid container	Check that the end of the drain tube is not submerged. If waste liquid has accumulated, dispose of it.	

Points to check during combustion

Safety	Inspection Point	Details	Remarks
	Nebulizer	If the capillary tube is clogged, clean it. If the polythene tube that covers the capillary tube is broken, replace it.	See 8.2.2 "Nebulizer Maintenance".

8.1.2 Monthly Inspection

The inspection that must be carried out at least once a month even if the instrument is not being used is described here.

WARNING

• The items with \bigcirc marked in the "Safety" column are the inspection points that relate to safety. Failure to complete these inspection points may result in gas leaks or flashback.

8.1.2.1 Common to All AA-7000 Series Models

Safety	Inspection Point	Details	Remarks
	Window plate of the atomizer	Wipe off soiling from it with a soft cloth moistened with alcohol.	

8.1.2.2 AA-7000F, AA-7000F/AAC

Points to check before ignition

Safety	Inspection Point	Details	Remarks
0	Burner head	If the burner slot is clogged or has been widened, replace it with a new one.	Use the card provided as an accessory.
0	Pilot flame section	If any soot has settled on the electrode, remove it.	See 8.4 "Checking the Pilot Flame Unit (AA-7000F, AA- 7000F/AAC)".
0	Purge button	Check that the assist gas flows for a few seconds after the button has been pressed and released.	
0	O-rings	Check that there is no damage or deformation.	See 8.2.3.2 "Replacing O- rings".
0	Drain tube	Check that there is no damage or deformation.	See 8.7 "Replacing the Drain Tube (AA-7000F, AA-7000F/ AAC)".
0	Piping tube	Replace the tube if there is any damage on the surface.	
0	Rubber hose	Replace the tube is there is any cracking on the surface.	

Safety	Inspection Point	Details	Remarks
0	Flame monitor	When the flow rate of the fuel gas has been set to 0.8 L/min, check that the flame is extinguished and the gas stops automatically. To specify the flow rate of fuel gas, see 3.1.8 "Atomizer/Gas Flow Rate Setup".	If the gas does not stop automatically there has been a failure. Implement the "In an Emergency" and contact your Shimadzu representative.

Points to check during combustion

8.1.2.3 Equipment and Options

Safety	Inspection Point	Details	Remarks
0	Gas piping equipment	Check that there are no leaks in the piping and joints.	See 8.5 "Checking for Gas Leaks (AA-7000F, AA-7000F/ AAC)".
0	Pressure regulator	Check that there is nothing abnormal in the operation of the pressure gauge and check that there is no gas leak in any part.	See 8.5 "Checking for Gas Leaks (AA-7000F, AA-7000F/ AAC)" and 8.6 "Checking for Leaks of Pressure Regulators (Optional)".

When foreign objects such as dirt and contaminants adhere to the burner head, nebulizer or chamber interior, the indicated value becomes unreliable and absorbance sensitivity is decreased. It is recommended that these burner components be periodically cleaned.

WARNING

- Wear protective goggles and protective gloves to maintain the burner head.
 If chemicals get into the eyes, there is a risk of loss of sight.
 For details, see "Precautions on Handling Chemicals and Samples" in the introductory section.
- Do NOT remove parts inside the burner compartment during flame combustion. Otherwise there will be a risk of gas leakage or flashback.
- Check that an O-ring is fitted, and check that the O-ring is not damaged or deformed. If no O-ring is fitted, or if the O-ring is damaged or deformed, it could cause an accident. To replace the O-ring, contact your Shimadzu representative.

The O-rings are at the positions indicated below.

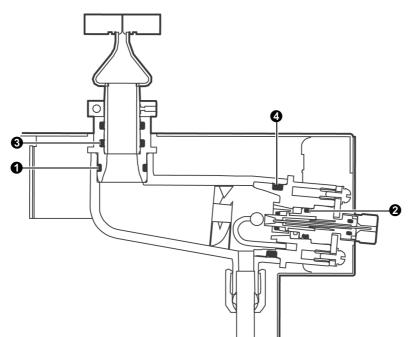
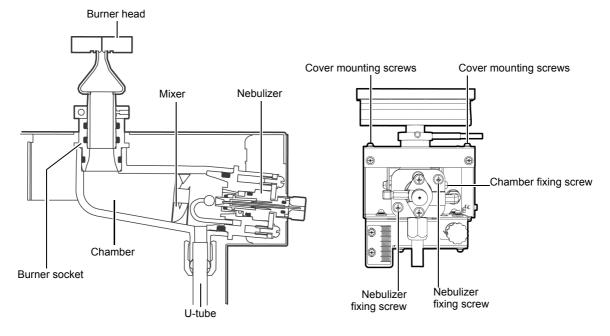


Fig. 8.1 Positions of O-rings

No.	Position	Туре	Number
0	Burner socket (chamber side)	P22	1
0	Between nebulizer and disperser	P12	1
0	Burner socket (burner head side)	AS568A-116	2
4	Between nebulizer and chamber	P32	1



The construction of the burner is as shown below. For the function of each part, see 1.4.3 "Burner".

Fig. 8.2 Burner Unit

NOTE

- Do NOT use metal brushes to maintain the burner head.
 - This will scratch the burner head and shorten its replacemental interval.

8.2.1 Burner Head Maintenance

WARNING

- Immediately after the flame has been extinguished the burner head is hot, so leave it for at least 30 minutes to cool before removing it.
 - If you touch the burner head immediately after the flame has been extinguished, you risk being burned.

If the slot of burner head is clogged by carbide or salts etc., the reduction flame will have fine irregularities when the degree of blockage is small. When the slot is clogged more heavily, the flame splits as the left figure in Fig. 8.3 (The figure is exaggerated to illustrate). Before the flame becomes the state indicated in the figure, extinguish the flame and then clean the inner wall of the slot by lightly rubbing it with the burner slot cleaning/ burner height check card.

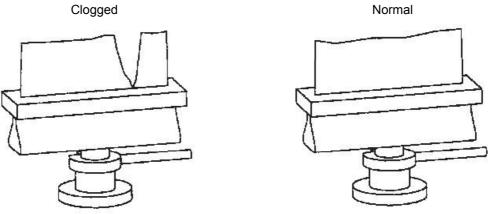


Fig. 8.3 Flame Symptoms due to Clogged Slot

NOTE

• When measuring samples with high concentrations of coexistent components, such as salts etc, these may adhere to the slot inner wall or inside of burner head. In some cases, orange colored flame may flicker when the burner is ignited again. In such cases, spray distilled water until flickering stops.

If flickering does not easily disappear, after extinguishing the flame and cooling down the burner head for 30 minutes or more, remove the burner head from the chamber; wash the inside with distilled water. If it is extremely dirty, soak it in acid or an appropriate detergent overnight then brush the inner wall of the slot with the burner slot cleaning/burner height check card.

• After washing, mount the burner head and adjust the burner position.

For details, see the section 8.3.1 "Burner Positioning Adjustment".

8.2.2 Nebulizer Maintenance

WARNING

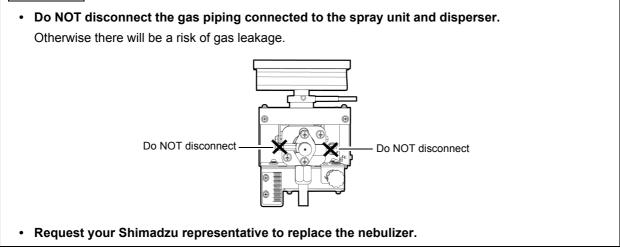


Fig. 8.4 shows the construction of the nebulizer. The nebulizer comprises the spray unit and the disperser.

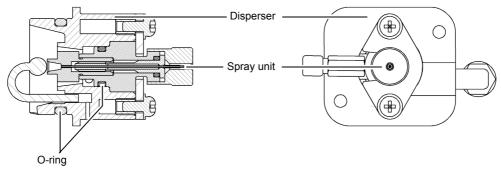


Fig. 8.4 Detail of Nebulizer Section

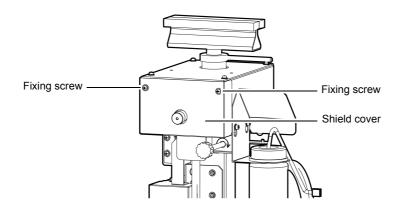
8.2.2.1 Removing and Mounting the Sampling Tube

Before carrying out maintenance on the nebulizer, remove the sampling tube that is fitted to the spray unit.

After carrying out maintenance on the nebulizer, mount the sampling tube in its original position.

8.2.2.2 Removing and Mounting the Shield Cover

Before carrying out maintenance on the nebulizer, remove the shield cover. Loosen the two fixing screws and remove the shield cover. After carrying out maintenance on the nebulizer, mount the shield cover in its original position.



8.2.2.3 Cleaning the Capillary

CAUTION

• The capillary is a thin and easily broken part. Take care not to apply too much force during this operation.

(1) While the flame is extinguished, insert the cleaning wire provided as an accessory into the capillary.

- (2) Clean the inner wall of the capillary by repeatedly passing the cleaning wire in and out of it.
- (3) Withdraw the cleaning wire, ignite the flame and spray distilled water.
- (4) Install a sampling tube and check absorption sensitivity and stability using, for example, the standard sample.

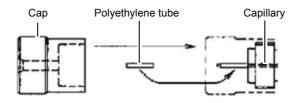
NOTE

If no improvement is seen after performing steps (1) to (4) above, inspect and replace the polyethylene tube.

8.2.2.4 Inspecting and Replacing the Polyethylene Tube

CAUTION

- If the polyethylene tube at the capillary of the spray unit has deteriorated or has come off, sample suction or mounting and holding of the sampling tube will not be possible.
- Do NOT remove the capillary from the spray unit. This could affect performance.
- (1) While the flame is extinguished, take out the sampling tube.
- (2) Turn the cap to the left and remove it.
- (3) Inspect the polyethylene tube fitted to the capillary and, if it has deteriorated, remove it.



(4) Cut the accessory polyethylene tube to a length of approximately 6 to 7 mm and slide it carefully onto the capillary while taking care not to bend the capillary.

NOTE

If it is difficult to slide the polyethylene tube on, increase the internal diameter at one of its ends by using a pointed implement such as a toothpick.

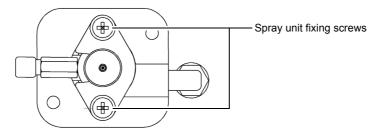
- (5) Once the polyethylene tube has been slid on, refit the cap as it was originally.
- (6) Install the sampling tube.
- (7) Ignite the flame and spray distilled water.
- (8) Check absorption sensitivity and stability using, for example, the standard sample.

NOTE

If no improvement is seen after performing steps (1) to (8) above, inspect and clean the tip of the spray unit.

8.2.2.5 Inspecting and Cleaning the Tip of the Spray Unit

(1) While the flame is extinguished, remove the two spray unit fixing screws.



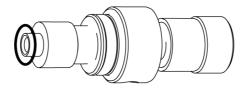
(2) Pull the spray unit toward you without disconnecting the gas piping.



Check whether there is any scratching or deformation of the O-ring.

For the position of the O-ring, see Fig. 8.4 "Detail of Nebulizer Section".

(3) Check the end of the spray unit and, if there is any contamination adhering to it, clean it off, for example with a plastic brush.



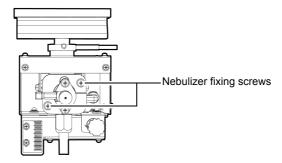
- (4) Mount the spray unit as it was originally.
- (5) While the flame is extinguished, insert the cleaning wire into the capillary and clean the inner wall of the capillary.
- (6) Withdraw the cleaning wire and install a sampling tube.
- (7) Ignite the flame and spray distilled water.
- (8) Check absorption sensitivity and stability using, for example, the standard sample.

NOTE

If no improvement is seen after performing steps (1) to (8) above, clean the disperser.

8.2.2.6 Cleaning the Disperser

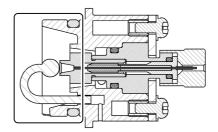
(1) While the flame is extinguished, remove the two nebulizer fixing screws.



(2) Pull the body of the nebulizer toward you without disconnecting the gas piping.

NOTE

Check whether there is any scratching or deformation of the O-ring. For the position of the O-ring, see Fig. 8.4 "Detail of Nebulizer Section". (3) Check the disperser and, if there is any contamination adhering to it, clean it off, for example with a plastic brush.



- (4) Mount the body of the nebulizer as it was originally.
- (5) While the flame is extinguished, insert the cleaning wire into the capillary and clean the inner wall of the capillary.
- (6) Withdraw the cleaning wire and install a sampling tube.
- (7) Ignite the flame and spray distilled water.
- (8) Check absorption sensitivity and stability using, for example, the standard sample.

NOTE

If no improvement is seen after performing steps (1) to (8) above, ask your Shimadzu representative to replace the nebulizer.

8.2.3 Chamber Maintenance

The procedure for cleaning the chamber components is explained below.

8.2.3.1 Cleaning the Chamber

WARNING

- Do NOT disconnect the piping that is connected to the nebulizer. This could cause a gas leak.
- Entrust the replacement of the nebulizer to your Shimadzu representative.

CAUTION

Do NOT use metal brushes.

This will scratch the chamber and shorten its replacement interval.

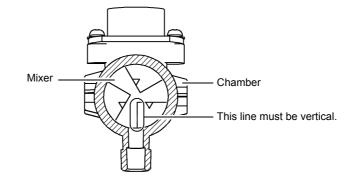
- On assembly after cleaning take care about the direction in which the mixer is mounted. The mixer has a slightly tapered shape, so insert it in the direction in which its taper agrees with the taper of the inside wall of the chamber. If it is not inserted in the correct direction, performance will be affected.
- (1) Remove the burner head (refer to 8.2.1 "Burner Head Maintenance").
- (2) Remove the sampling tube that is connected to the nebulizer.

- (3) Remove the 2 nebulizer fixing screws, and pull the nebulizer toward you.
- (4) Take the mixer out from the chamber.
- (5) Wash the inside of the chamber and the mixer with distilled water.

NOTE

If there is heavy soiling, remove it with a brush for plastics.

(6) Mount the mixer in the chamber as shown below.



8.2.3.2 Replacing O-rings

WARNING

- The O-rings are parts for periodic replacement. If an O-ring is damaged or deformed it must be replaced. Failure to do so could cause accidents.
- Select O-rings that are compatible with the solvent used. If an organic solvent is using while leaving standard O-rings fitted, the O-rings will be eroded by the solvent and this could cause accidents.

Request your Shimadzu representative to replace the O-rings.

NOTE

The O-rings, which have to be replaced as maintenance parts, are provided in a set.

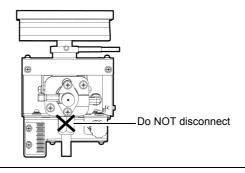
Туре	Part No.	Material
Standard part	S206-77620-91	Fluoro rubber
Resistant part (optional)	S206-77620-93	Silicon rubber
Highly resistant part (optional)	S206-77620-92	Perfluoroelastomer

For details on the chemicals that can be used with each type of O-ring, see 10.1.2 "Flame Specifications".

8.2.3.3 Cleaning the Drain Tank and U-Tube

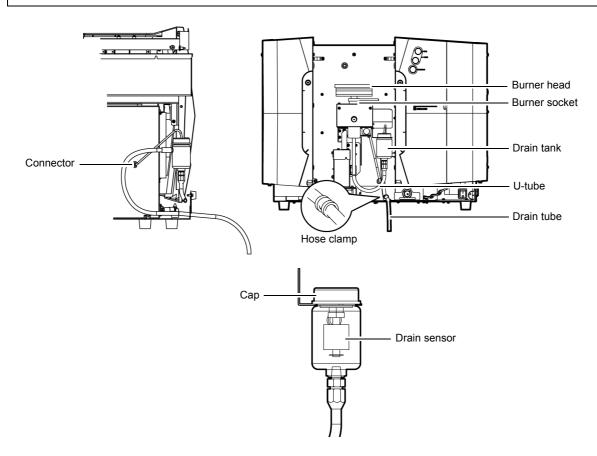
WARNING

- Wear protective goggles and protective gloves to maintain the parts.
 If chemicals get into the eyes, there is a risk of loss of sight.
 For details, see "Precautions on Handling Chemicals and Samples" in the Introductory section.
- Do NOT remove the joint between the chamber and the U-tube. This could cause a gas leak.

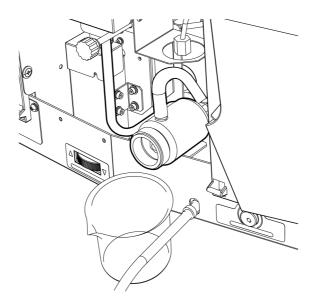


CAUTION

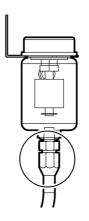
- If you spill any waste liquid on the instrument, wipe it up immediately.
- It could cause rusting or instrument failure.



- (1) In order to deal with the waste liquid in the drain tank and the U-tube, prepare a container with chemical resistance to the waste liquid (such as a beaker).
- (2) Remove the burner head.
- (3) Remove the drain sensor's connector.
- (4) Open the cap on the drain tank and take out the drain sensor.
- (5) So as not to break the U-tube, tilt the drain tank forward gently while draining all of the waste liquid in the drain tank into the waste liquid container.



- (6) Remove the drain tube and hose clamp.
- (7) Remove the joint between the drain tank and U-tube, and take out the drain tank.



(8) Clean the drain tank using distilled water.

NOTE

If there is heavy soiling, remove it with a plastic brush.

- (9) Pour in distilled water through the burner socket and fill the U-tube.
- (10) While pressing a clean rag against the U-tube's outlet, drain the liquid from the U-tube by bending it gently so that it will not break.

(11) Repeat the operations in steps (9) and (10) to clean the U-tube.

NOTE

If there is heavy soiling, remove it with a plastic brush.

(12) When the cleaning work is finished, return all the parts to their original situations.

NOTE

If you spill any waste liquid, wipe it up with a clean rag.

Atomizer Positioning Adjustment

8.3.1 Burner Positioning Adjustment

8.3.1.1 AA-7000F

The following is the procedure for adjusting the forward/backward position of the AA-7000F's burner to achieve the position with the highest sensitivity.

- (1) Select an element that you usually use in the flame continuous method.
- (2) In the [Optics Parameters] page, set the lamp mode to [NON-BGC] and turn on the lamp to perform the line search.
- (3) Check that the burner head is mounted securely, then adjust the burner to the 10 mm position by using the burner up/down position adjusting knobs. Set the burner angle to 0°.
- (4) Set the burner height checking card provided as an accessory at the center of the burner head so that the scale on it faces the lamp.

Adjust the forward/backward position of the burner with the burner forward/backward adjusting knobs so that the light beam shines directly above the burner slot.

- (5) Move the burner height checking card to the right end of the burner head and, while turning the burner head little by little by using the burner head angle adjusting lever, ensure that the light beam shines directly above the burner slot.
- (6) Set the burner height checking card at the center of the burner head again and check that the light beam is directly above the burner slot.

The burner is now set at the most sensitive position in the forward/backward direction. You should adjust the burner height in accordance with the measurement conditions.

8.3.1.2 AA-7000F/AAC

In the case of flame method, the burner is set so that the burner head slot is positioned directly beneath the optical path. However, when the burner is dismantled for cleaning etc., the burner position must readjusted. The procedure is described below.

- (1) Select an element, which you usually use in flame continuous method.
- (2) In the [Optics Parameters] page, set the lamp mode to [NON-BGC] and light on the lamp to perform the line search.
- (3) Select [Instrument]-[Maintenance]-[Burner Origin Position Adjustment] from the menu. Then [Burner Origin Position Adjustment] dialog box opens.
- (4) Verify that the burner head is securely mounted. Then click on [Up] and [Down] buttons to set the burner height to about 10 mm. In addition, set the burner angle to 0°.
- (5) Set the burner height checking card provided as an accessory at the center of the burner head so that the scale on it faces the lamp.
- (6) Using the [Forward] and [Backward] buttons, adjust the forward/backward position of the burner so that the light beam shines directly over the burner slot.
- (7) Move the burner height checking card to the right end of the burner head without changing the direction in which it is facing and, while turning the burner head little by little by using the burner head angle adjusting lever, ensure that the light beam shines directly above the burner slot.
- (8) Set the burner height checking card at the center of the burner head again and check that the light beam is directly above the burner slot.
- (9) Click on the [Up] and [Down] buttons to set the burner height to 10 mm. Set the check card on the burner. Adjust the burner height so that the beam spot on the paper is semicircular as shown in Fig. 8.6.

(10) Click on [Origin Memory].

The current burner position is memorized as "Forward/Backward = 0" and "Up/down = 10".

After the above procedure, the burner position is adjusted.

The burner height should be adjusted according to the measurement conditions.

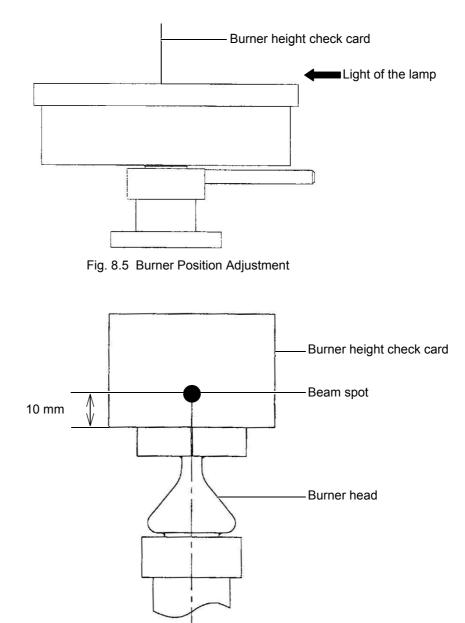


Fig. 8.6 Burner Height Adjustment

8.3.2 Furnace Position Adjustment

The procedure for adjusting the position of the furnace is given below. Position adjustment should be carried out while leaving the graphite furnace installed.

8.3.2.1 AA-7000G

- (1) Select an element that you usually use in the furnace method.
- (2) On the [Optics Parameters] page, set the lamp mode to [NON-BGC]. Press [Cancel] in response to the message "Line Search/Beam Balance is necessary" in this procedure.
- (3) From the Menu, select [Instrument]-[Maintenance]-[Furnace Origin Position Adjustment]. This opens the [Furnace Origin Position Adjustment] dialog box.
- (4) Click on [Line Search] to open [Line Search/Beam Balance] and execute Line Search/Beam Balance.
- (5) After both Line Search and Beam Balance have been completed successfully, click on the [Close] button.
- (6) Using the manual control knob, adjust the horizontal and vertical positions to minimize the measured value in the [Furnace Origin Position Adjustment] dialog box.

NOTE

Immediately after the lamp has been on the baseline may not be stable. If this is the case, wait until the baseline has stabilized and then carry out position adjustment.

The furnace position has now been adjusted.

8.3.2.2 AA-7000F/AAC

- (1) Select an element that you usually use in the furnace method.
- (2) In the [Optics Parameters] page, set the lamp mode to [NON-BGC].
 Press [Cancel] in response to the message [Line Search/Beam Balance is necessary] in this procedure.
- (3) From the Menu, select [Instrument]-[Maintenance]-[Furnace Origin Position Adjustment]. This opens the [Furnace Origin Position Adjustment] dialog box.
- (4) Now tick the [Move the furnace down to the lower position at the time of Line Search] check box and then click [Line Search].

After the furnace has been retracted from the optical axis, the [Line Search/Beam Balance] dialog box is displayed and a line search is performed.

- (5) After both Line Search and Beam Balance have been completed successfully, click on the [Close] button. This returns you to the [Furnace Origin Position Adjustment] dialog box and moves the furnace into the optical axis.
- (6) Using the [Forward], [Backward], [Up] and [Down] buttons, adjust the horizontal and vertical positions to minimize the measured value.

NOTE

Immediately after the lamp has been on the baseline may not be stable. If this is the case, wait until the baseline has stabilized and then carry out position adjustment.

(7) After adjusting the position, click on [Origin Memory]. The current furnace position is memorized as "Forward/Backward=0" and "Up/Down=0".

The furnace position has now been adjusted.

With extended use of the instrument, soot may accumulate on the electrode in the pilot flame unit. When soot builds up in this area, it may obstruct the pilot flame, making it difficult to ignite the burner. In the worst case, the pilot flame may remain lit inside the instrument, causing a fire hazard.

8.4.1 Inspection Procedure

- (1) Verify that the instrument is turned off.
- (2) Remove the chimney.
- (3) Peer into the pilot flame unit and examine the electrode.

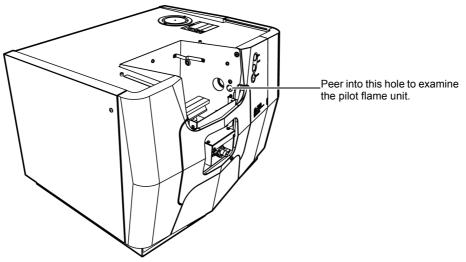
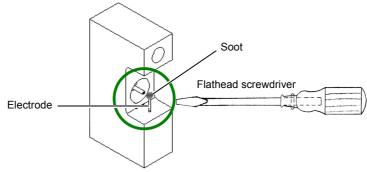
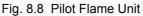


Fig. 8.7 Position of Pilot Flame Unit

(4) If soot has accumulated on the electrode, scrape it off with a slender, rigid object, such as the tip of a flathead screwdriver. Not much force is needed to remove the soot.





(5) Reattach the chimney.

A constant check is conducted for gas leakage from the fuel gas tube inside the gas control unit. This section describes the method for inspecting for leakage from the gas piping (customer's equipment), the gas hose (standard accessory), and the gas piping that includes the gas control section inside the instrument.

WARNING

• If a gas leak occurs, stop using the instrument immediately and request your Shimadzu representative to inspect the instrument.

Continuing to use the instrument without taking action could lead to an accident.

Carry out an inspection at least once a month by following the procedure below. Note that the gas leakage inspection described here is based on the recommended piping example in 10.6.3 (3) "Gas piping". For details, see 10.6.3 (3) "Gas piping".

- (1) Turn the power switch of the AA-7000 OFF. Also start the compressor.
- (2) Open the main valves on the gas cylinder and compressor, and all the stop cocks.
- (3) Verify that the gas pressure is set correctly.

Gas type		Supply pressure
Fuel gas	Acetylene (C ₂ H ₂)	0.09 MPa ± 0.01 MPa
Support gas	Air, Nitrousoxide (N ₂ O)	0.35 MPa ± 0.03 MPa

- (4) Wait for at least 5 seconds, then close the main valves.
- (5) Read the indications of the pressure gauge between the main valve and the instrument in each case.
- (6) Read the indication of each pressure gauge after an interval of 30 minutes.
 - If the drop in pressure over 30 minutes at the fuel gas side is greater than 0.01 MPa, or if the drop in pressure at the assist gas side is greater than 0.02 MPa, it is judged that there is a gas leak.

NOTE

- If it is judged that there is a gas leak, apply soapy water or leak detection fluid (optional) at each connection between the gas cylinder or compressor and the gas control unit and burner to check the location of the leak.
- If a gas leak has occurred at a connection, make the connection again. If it is judged that there is a gas leak inside the gas control unit, stop using the instrument and contact your Shimadzu representative.

Checking for Leaks of Pressure Regulators (Optional)

WARNING

- 1. If a mistake is made in the piping, it may cause flashback on ignition.
- 2. Always set the gas supply pressures at the specified values.
- 3. Never use a broken regulator. This may result in gas leaks.
- 4. If nitrous oxide is used, use the dedicated pressure regulator.
- Wipe any dust adhering to the outlet of the cylinder.
- Refer to Fig. 8.9 and Fig. 8.10 to install these pressure regulators on the cylinders. If the screw for mounting the regulator on the cylinder appears ready to break, replace the cylinder without attempting to mount the regulator.
- Open the cylinder main valve gently after closing the stopcock and turning the secondary pressure control handle sufficiently counterclockwise.
- Turn the secondary pressure control handle clockwise and set the secondary pressure to the following values:

Acetylene 0.09 MPa Nitrous oxide 0.35 MPa

• Apply soapy water or leak detection fluid (optional) to each of the connecting joints to check for leaks. Due care should be taken to detect and eliminate any leaking at the cylinder cock.

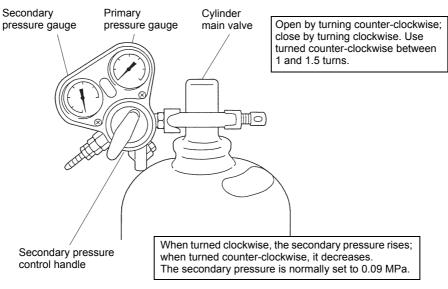


Fig. 8.9 Pressure Regulator for Acetylene

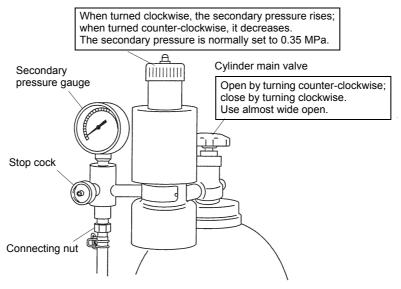


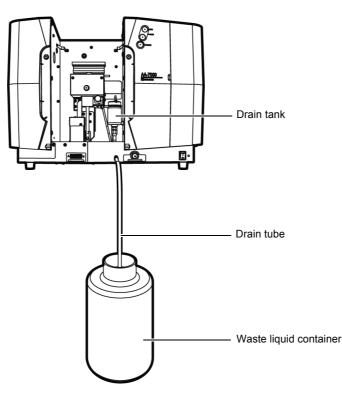
Fig. 8.10 Pressure Regulator for Nitrous Oxide

When the drain tube becomes old it may rupture, allowing drain to leak out. Replace the drain tube by following the procedure below.

- (1) Remove the old polythene tube connected to the outlet of the drain tank.
- (2) Connect the new polythene tube.
- (3) Prepare a container to receive the drain from the instrument.
- (4) Cut the polythene tube to the appropriate length.

Make sure that the end of the tube is not submerged in the waste liquid in the container.

(5) Supply water a little at a time through the burner socket until water overflows from the drain tank. If the water level is too low the liquid level sensor actuates and ignition will not be possible.



CAUTION

• Be sure to lower the drain tube as smoothly as possible. Also ensure that the end of the tube is not submerged in the waste liquid in the container.

If the waste liquid cannot flow smoothly the level of noise will be increased and this may have an adverse affect on repeatability.

Replacing the Deuterium Lamp

8.8.1 Specifications of Deuterium Lamp

Table 8.1 Specifications of Deuterium Lamp

Part No	S062-65055-05
Туре	L6380
Average Life	500 hours

8.8.2 Replacing Procedures of Deuterium Lamp

WARNING

- Wait at least 30 minutes after turning the D_2 lamp off before replacing the lamp.

Otherwise you could be burned.

CAUTION

- 1. Handle the new lamp using gloves so as not to leave fingerprints on the light beam window. Otherwise, the window is stained with the fingerprints when it is hot, and as the result the light transmission will be reduced. If contaminated, clean it with alcohol or the like.
- 2. When detaching or attaching the cover of the deuterium lamp house, be very careful not to hit the protruding upper edge of the deuterium lamp against the cover rear side. It may cause a vacuum leak of the bulb.

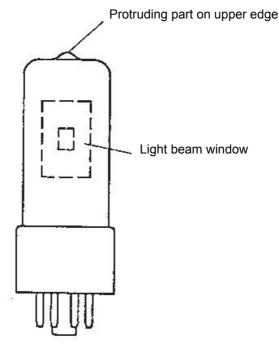


Fig. 8.11 Deuterium Lamp

8.8.2.1 Replacing Deuterium Lamp

- (1) Turn the connection between the PC and the instrument OFF.
- (2) Click [Instrument] [Lamp History] on the WizAArd screen.

🊧 п	otitle	- WizAArd (System Administrator)		
File	Edit	Parameters	Instrument Window Help		
	2	🛛 🖉 🛛	Connect Option Connect	ib. Curve	Latest
			Configuration Lamp Position Setup	ļļ	-0.001
	0.80	Ē	Lamp History		
	0.70	0	Set Wavelength to Background WL	·	
	0.60	0	Maintenance Lamp Status		
	0.50	0	Change Graphite Tube	····	
	0.40	0	Gas Controller Status Gas Leak Check	····	
b s	0.30	0	Remaining Gas Combustion Execute Line Search		
	0.20	0	Cleaning Rinse Nozzle		
	0.10	0	Flame Cont. Nozzle Position Flame Micro, Nozzle Position		
	0.00	0	Furnace Nozzle Position	ΗI	
	-0.10	۰ ‡ ۰۰۰۰۰			
	-0.20	0 <u>‡</u> Inte	rval 600 sec		

The [Lamp History] dialog box will be displayed.

(3) If there is no check mark in the [Lock] column for the deuterium lamp (D2), enter a check mark and then click the number "1" at the left hand end to highlight the relevant row.

amp	np History													
	Lamp ID	Element	Lamp Type	Life Time	Used Time	Unit	Judge	Comment	Lock	-[OK			
1	D2		D2	500	1.0	hrs	ОК							
2	Ag-1	Ag	Normai	3000	0.0	mentrs	UN			' II	Cancel			
3	Al-1	AI	Normal	5000	0.0	mA*hrs	OK							
4	As-1	As	Normal	3000	0.0	mA*hrs	OK				<u>C</u> lear			
5	Au-1	Au	Normal	5000	0.0	mA*hrs	OK			1				
6	B-1	В	Normal	5000	0.0	mA*hrs	OK				Delete			
7	Ba-1	Ba	Normal	5000	0.0	mA*hrs	ОК				<u>D</u> 0/010			
8	Be-1	Be	Normal	5000	0.0	mA*hrs	OK				Dia			
9	Bi-1	Bi	Normal	5000	0.0	mA*hrs	ОК				<u>P</u> rint			
10	Ca-1	Ca	Normal	5000	0.0	m&*brs	OK			-				

(4) Click [Clear].

Lamp	History									×
	Lamp ID	Element	Lamp Type	Life Time	Used Time	Unit	Judge	Comment	Lock 📥	ОК
1	D2		D2	500	1.0	hrs	ок			l
2	Ag-1	Ag	Normal	5000	0.0	mA*hrs	OK			Cancel
3	AI-1	AI	Normal	5000	0.0	mA*hrs	OK			
4	As-1	As	Normal	3000	0.0	mA*hrs	OK			<u>C</u> lear
5	Au-1	Au	Normal	5000	0.0	mA*hrs	OK			
6	B-1	В	Normal	5000	0.0	mA*hrs	OK			Delete
7	Ba-1	Ba	Normal	5000	0.0	mA*hrs	OK			
8	Be-1	Be	Normal	5000	0.0	mA*hrs	OK			
9	Bi-1	Bi	Normal	5000	0.0	mA*hrs	OK			<u>P</u> rint
10	Ca.1	Ca	Normal	5000	0.0	m≜*hr≈	OK .			

(5) When the message "The operator is about to set lamp usage time to zero. Are you sure?" is displayed, click [Yes].



(6) Check that the [Used Time] of the changed lamp is [0], then click [OK] on the [Lamp History] dialog box.

Lamp	History				_	_				×
	Lamp ID	Element	Lamp Type	Life Time	Used Time	Unit	Judge	Comment	Lock	ОК
1	D2		D2	500	0.0	Irs	ок			
2	Ag-1	Ag	Normal	5000	0.0	mA*hrs	OK			Cancel
3	Al-1	AI	Normal	5000	0.0	mA*hrs	OK			
4	As-1	As	Normal	3000	0.0	mA*hrs	OK			<u>C</u> lear
5	Au-1	Au	Normal	5000	0.0	mA*hrs	OK			
6	B-1	B	Normal	5000	0.0	mA*hrs	OK			Delete
7	Ba-1	Ba	Normal	5000	0.0	mA*hrs	OK			
8	Be-1	Be	Normal	5000	0.0	mA*hrs	OK			Drint
9	Bi-1	Bi	Normal	5000	0.0	mA*hrs	OK			<u>Print</u>
10	Ca-1	Ca	Normal	5000	0.0	m&*hrs	OK			

(7) As shown below, remove the two screws to detach the lamp cover. When the cover is removed, the deuterium lamp will appear as shown in Fig. 8.11.

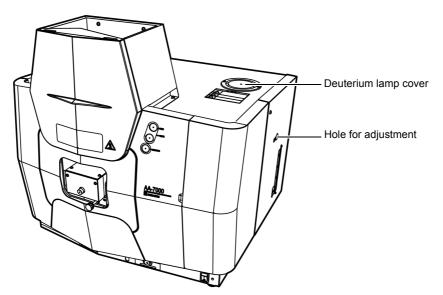
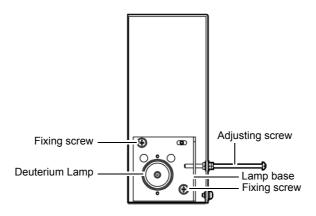


Fig. 8.12 Deuterium Lamp Cover

- (8) Pull the deuterium lamp slowly upward and out of its socket.
- (9) Insert a new deuterium lamp into the socket. Check that the plastic part of the electrode makes tight contact with that of the lamp socket.

8.8.2.2 Adjusting Position of Deuterium Lamp

(1) Loosen the 2 fixing screws.



- (2) Turn on the power of the instrument.
- (3) Start up the PC software and select [Instrument]-[Connect] from the menu to make the connection for communication with the instrument.
- (4) Select an element that you usually use, and measurement methods (flame continuous method, flame micro sampling method and furnace method).
- (5) Select [Instrument]-[Maintenance]-[D2 Lamp Position] from the menu. The message "D2 lamp position adjustment procedure is going to start. Please adjust D2 lamp position roughly first." is displayed.
- (6) Set the deuterium lamp to almost center with the adjusting screw.
- (7) Click on [OK] and close the lamp cover.
- (8) Next, the deuterium lamp intensity in absorbance is displayed in [D2 Lamp Adjustment] dialog box. Adjust the position using the adjusting knob so that the indicated value may become minimum.

NOTE

Immediately after the lamp has been on, the baseline may not be stable. If this is the case, wait until the baseline has stabilized and then carry out position adjustment.

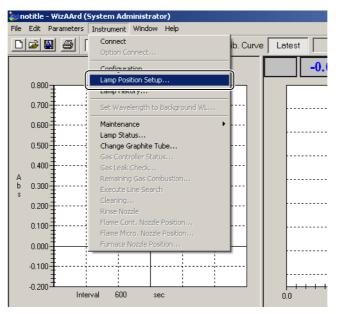
(9) Tighten the 2 deuterium lamp fixing screws, and click on [OK] to close the [D2 Lamp Adjustment] dialog box.

NOTE

After replacing the deuterium lamp, exit the PC software and turn off the power to the instrument.

8.9

- (1) Turn the connection between the PC and unit OFF.
- (2) Click [Instrument] [Lamp Position Setup] on the WizAArd screen.



The [Lamp Position Setup] dialog box will be displayed.

(3) Change the setting for [Element] of the [Socket #] to be changed to [None].

Socket #	Element	Lamp Type	Lamp ID	Judge	Life Time	Used Time	Unit		OK
1	Zn	Normal	Zn-1	ок	5000	0.0	mA*hrs		Cano
2	Cd	Normal	Cd-1	OK	5000	0.0	mA*hrs	1	
3	Pb	Normal	Pb-1	OK	5000	0.0	mA*hrs	1	Print
4	.0.0	Normal	As-1	OK	3000	0.0	mA*hrs	1	
5	Se 🔻	Normal	Se-1	over	5000	5028.0	mA*hrs	1	
5	None 🔺]	
	Ag — Al								
	As								
	Au B Ba								
	Be								

(4) Click [OK].

	fime Time	Tim	Judge	Lamp ID	Lamp Type	Element	Socket #
Zn-1 OK 5000 0.0 mA*hrs C	0 0.0 mA	5000	OK	Zn-1	Normal	Zn	
Cd-1 OK 5000 0.0 mA*hrs	0.0 m.A ³	5000	OK	Cd-1	Normal	Cd	2
Pb-1 OK 5000 0.0 mA*hrs	0.0 m.A ³	5000	OK	Pb-1	Normal	Pb	3
As-1 OK 3000 0.0 mA*hrs	0.0 m.A ³	3000	OK	As-1	Normal	As	ł
						None 💌	5
						None	3
							5

🎉 n File	otitle - WizAArd Edit Parameters	(System Administrator) Instrument Window Help	
	2	Connect ib. Curve	e Latest
		Configuration	-0.
	0.800	Lamp History	
	0.700	Set Wavelength to Background WL	
	0.600	Maintenance	
	0.500	Lamp Status Change Graphite Tube	
	0.400	Gas Controller Status Gas Leak Check	
A b	0.300	Remaining Gas Combustion Execute Line Search	
s	0.200	Cleaning	
	0.100	Rinse Nozzle Flame Cont. Nozzle Position Flame Micro. Nozzle Position	
	0.000	Furnace Nozzle Position	
	-0.100		
	-0.200	erval 600 sec	0.0

(5) Click [Instrument] - [Lamp History] on the WizAArd screen.

The [Lamp History] dialog box will be displayed.

(6) If there is no check mark in the [Lock] column for the lamp to be replaced, enter a check mark and then click the number at the left hand end to highlight the relevant row.

Lamp	History									×
	Lamp ID	Element	Lamp Type	Life Time	Used Time	Unit	Judge	Comment	Lock	ОК
49	Sc.1	Sc	Normal	5000	0.0	m&*hrs	OK			_
50	Se-1	Se	Normal	5000	5028.0	mA*hrs	over			Cancel
151	31-1	31	Normai	5000	0.0	mAmis	On			
52	Sm-1	Sm	Normal	5000	0.0	mA*hrs	OK			<u>C</u> lear
53	Sn-1	Sn	Normal	5000	0.0	mA*hrs	OK			
54	Sr-1	Sr	Normal	5000	0.0	mA*hrs	OK			Delete
55	Ta-1	Ta	Normal	5000	0.0	mA*hrs	OK			
56	Tb-1	Tb	Normal	5000	0.0	mA*hrs	ок			Dia I
57	Te-1	Te	Normal	5000	0.0	mA*hrs	OK			<u>P</u> rint
58	Ti-1	Ti	Normal	5000	0.0	mA*hrs	OK			

(7) Click [Clear].

.amp	mp History													
	Lamp ID	Element	Lamp Type	Life Time	Used Time	Unit	Judge	Comment	Lock		OK			
49	Sc-1	Sc	Normal	5000	0.0	mA*hrs	ок							
50	Se-1	Se	Normal	5000	5028.0	mA*hrs	over			_	Cancel			
51	Si-1	Si	Normal	5000	0.0	mA*hrs	ок			Ć				
52	Sm-1	Sm	Normal	5000	0.0	mA*hrs	ок				<u>C</u> lear			
53	Sn-1	Sn	Normal	5000	0.0	mA*hrs	ок			U				
54	Sr-1	Sr	Normal	5000	0.0	mA*hrs	ок				Delete			
55	Ta-1	Ta	Normal	5000	0.0	mA*hrs	OK				<u>D</u> 01010			
56	Tb-1	Tb	Normal	5000	0.0	mA*hrs	ок				Duint			
57	Te-1	Те	Normal	5000	0.0	mA*hrs	ок				<u>P</u> rint			
58	Ti-1	Ti	Normal	5000	0.0	mA*hrs	ок							

(8) When the message "The operator is about to set lamp usage time to zero. Are you sure?" is displayed, click [Yes].

WizAArd	×
2	The operator is about to set lamp usage time to zero. Are you sure?
	Yes No

(9) Check that the [Used Time] of the changed lamp is "0", then click [OK] on the [Lamp History] dialog box.

Lamp	History				_	_					×
	Lamp ID	Element	Lamp Type	Life Time	Used Time	Unit	Judge	Comment	Lock	▲ [ОК
49	Sc-1	Sc	Normal	5000	0.0	nA*hrs	ок				
50	Se-1	Se	Normal	5000	0.0	nA*hrs	ОК				Cancel
51	Si-1	Si	Normal	5000	0.0	nA*hrs	ок				
52	Sm-1	Sm	Normal	5000	0.0	mA*hrs	OK				<u>C</u> lear
53	Sn-1	Sn	Normal	5000	0.0	mA*hrs	OK				
54	Sr-1	Sr	Normal	5000	0.0	mA*hrs	OK				Delete
55	Ta-1	Ta	Normal	5000	0.0	mA*hrs	OK				<u></u>
56	Tb-1	Tb	Normal	5000	0.0	mA*hrs	OK				Print
57	Te-1	Te	Normal	5000	0.0	mA*hrs	OK				<u>-</u> mit
58	Ti-1	Ti	Normal	5000	0.0	mA*hrs	OK				

(10) Click [Instrument] - [Lamp Position Setup] on the WizAArd screen.

🪧 n	otitle	- WizAArd (System Adr	ninistrat	or)			
File	Edit	Parameters	Instrument	Window	Help			
	2	86	Connect Option Co	nnect			ib. Curve	Latest
			Configure	tion				-0.
	0.80	0	Lamp Pos	ition Setup)			
	0.00	÷ ‡	camp hist	0ry			í I I	
	0.70	0₫	Set Wave	length to I	Background '	WL	····	
	0.60	۰ ‡	Maintena	nce		•		
		Ŧ	Lamp Stal					
	0.50	0	-	raphite Tu roller Statu			····	
	0.40	o <u>‡</u>	Gas Conu Gas Leak		15		····	
A		.‡		g Gas Com	bustion			
b s	0.30	۰ <u>۲</u>		ine Search			····	
	0.20	₀₫	Cleaning, Rinse Noz				····	
	0.10	. Ŧ			Position			
	0.10	Ŧ	Flame Mic	ro, Nozzle	Position			
	0.00	0	Furnace I	lozzle Pos	tion		H	
	-0.10	<u>م</u>						
		Ŧ						
	-0.20	0_ <u>t </u>	rval 600	se	:			
		inte		36				0.0

The [Lamp Position Setup] dialog box will be displayed.

(11) Return the lamp for which [None] was set at step (3) to its original setting.

Socket #	Element	Lamp Type	Lamp ID	Judge	Life Time	Used Time	Unit		OK
1	Zn	Normal	Zn-1	ок	5000	0.0	mA*hrs	-	Cano
2	Cd	Normal	Cd-1	OK	5000	0.0	mA*hrs		
3	Pb	Normal	Pb-1	OK	5000	0.0	mA*hrs		Print
1	<u>Ac</u>	Normal	As-1	OK	3000	0.0	mA*hrs		
5	None 🔻								
6	Se 🔺								
	Sm Sm Sr Sr Ta Tb Tc Te	,							

(12) Click [OK].

Socket #	Element	Lamp Туре	Lamp ID	Judge	Life Time	Used Time	Unit		ОК
1	Zn	Normal	Zn-1	ок	5000	0.0	mA*hrs	1	Cance
2	Cd	Normal	Cd-1	ОК	5000	0.0	mA*hrs		
3	Pb	Normal	Pb-1	OK	5000	0.0	mA*hrs		Print
4	As	Normal	As-1	ок	3000	0.0	mA*hrs		
5	Se 🔻	Normal	Se-1	OK	5000	0.0	mA*hrs		
3	None								

Exit the [Lamp Position Setup] dialog box.

(13) Fit the lamp in accordance with 2.3 "Mounting a Hollow Cathode Lamp (Optional)".

The parts for periodic replacement are indicated below. All parts for periodic replacement are consumable parts.

The service life of consumable parts changes depending on the working environment and measurement samples. The standard replacement interval indicates the period for periodically replacing parts and does not indicate the guaranteed service life of parts. Instrument downtime can be reduced by replacing parts at the standard replacement interval.

8.10.1 Parts Relating to Safety of Product

NOTE

Parts listed below relate to the safety of the product. Be sure to replace the parts periodically.

Part Name	Part No.	Remarks	Standard Replacement Interval
O-ring SET	S206-77620-91	For burner unit (Fluorine-containing rubber)	Depends on the chemical used ^{*1}
Burner head ASSY	S206-50370-94	Titanium, 10 cm	3 years *2
Drain ASSY	S206-77413-41	Overflow-type drainage trap	Depends on the chemical used ^{*1}
Hose, 8×2B-BK	S016-02101-04	For support gas (Air), Black Specify the necessary length. (Standard length: 5 m)	3 years
Hose, 8×2B-R	S016-02101-03	For fuel gas, Red Specify the necessary length. (Standard length: 5 m)	3 years
Hose, 8×2B-G	S016-02101-01	For support gas (N ₂ O), Green Specify the necessary length. (Standard length: 5 m)	3 years

*1: For details, see 10.1.2 "Flame Specifications".

*2: Number of days of actual usage is 600 days.

Part Name	Part No.	Remarks	Standard Replacement Interval
Lamp, D ₂ L6380	S062-65055-05	Deuterium lamp	1 year
Nebulizer	S206-77760-41	With impact bead	2 years
Burner socket	S206-77297		3 years
PE tube, 8×1	S016-43201-02	Drain tube, connects to drain bottle Specify the necessary length. (Standard length: 2.4 m)	3 years
Chamber ASSY	S206-77295-01	Engineering plastic (PP)	6 years
Mixer	S206-50486		6 years

8.10.2 Parts Relating to Performance of Product

8.11

Part Name	Part No.	Remarks
Cleaning wire	S201-79229-01	For cleaning the capillary
Sampling tube	S204-05899-01	PTFE tube for sample uptake
Sampling tube ASSY	S206-50772-91	Polyethylene tube for sample uptake
Wire bands, for 16 mm	S037-61019	Hose band
Polyethylene capillary No.3	S200-31328-01	For sealing the capillary Specify the necessary length (Standard length: 30 cm)
Card	S206-52046-91	For cleaning the burner/for checking the burner height (Set of 10)
Fixing screw	S206-51843	For retaining hollow cathode lamp
Window panel	S206-25346-91	
PCB ASSY, AA-MX CPU	S206-77135-42	
PCB ASSY, AA-MX POWER	S206-77140-42	
Communication cable	S206-50325-91	For connection to the PC
Power cable (for 100 V, 120 V)	S071-60821-08	For Japan and North America
Power cable (for 220 V)	S071-60827-05	For China
Power cable (for 230 V)	S071-60825-51	For Europe
WizAArd CD-ROM	S206-77526-91	
Instruction Manual	S206-97176	
"Safety Precautions" Check-out Sheet	S206-97226	

Chapter 9 Troubleshooting

CONTENTS

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9.1

When it is not possible to ignite the flame of the AA-7000F or AA-7000F/AAC, take the following corrective action in accordance with the symptoms as follows.

Symptom	Cause / Details	Checking Method	Corrective Action
Nothing happens in response to the ignition operation. No error message is displayed.	Either communications between the instrument and the PC are OFF or the instrument's power is OFF.	Check the power switch and indicator at the bottom right of the front of the instrument.	Turn the power switch ON. Select [Instrument] - [Connect] from the WizAArd menu to complete initialization. See 2.1.1 "Activation".
	Gas leakage detection is being executed.	The status on the [Gas Controller Status] dialog box is [Checking]. The remaining time for gas leakage detection is indicated by the status bar display.	Wait until the gas leakage inspection is completed. See 2.7 "Checking the Gas Controller Status (AA-7000F, AA-7000F/ AAC)".
	The gas leakage inspection has produced an [NG] result.	The indication on the [Gas Controller Status] dialog box is [Gas leak was found!!]. [NG] is indicated for the gas leakage inspection in the status bar display.	Stop using the instrument and contact your Shimadzu representative. See 2.7 "Checking the Gas Controller Status (AA-7000F, AA-7000F/ AAC)".
The pilot flame establishes, but the burner goes out immediately after igniting. Only the following error message is displayed: [Fuel gas pressure is too low].	The supply pressure of the fuel gas is actually low.	Ignition will be possible if the supply pressure of the fuel gas is increased.	Check if the supply pressure of the fuel gas is 0.09 MPa. It is necessary that this set pressure is maintained while the gas is flowing.
The pilot flame establishes but it is difficult to get the burner to ignite. When ignition fails, the message "Flame has been extinguished." is	The ratio of fuel gas in the mixture is too low and the flame blows out.	Ignition will be possible if the flow rate of fuel gas is increased.	Increase the flow rate of the fuel gas, ignite the flame, then decrease the flow rate later. See 3.1.8 "Atomizer/ Gas Flow Rate Setup".
displayed.	The pilot flame is too small to reach the burner.	The flame is actually too small.	Adjust the pilot flame.
The pilot flame fails to establish. Gas is flowing.	The igniter has failed.	The igniter does not discharge.	Repair the igniter.
When ignition fails, the message "Flame has been extinguished." is displayed.	The electromagnetic valve for the pilot has failed.	The igniter does discharge but no pilot flame is generated.	Repair the electromagnetic valve for the pilot.

This section describes the types of trouble that could occur while using an AA-7000 series instrument, and the corrective actions to take against them.

When any kind of trouble occurs, first see Chapter 8 "Maintenance" and Chapter 9 "Troubleshooting". If this does not resolve the trouble, contact your Shimadzu representative.

9.2.1 Common to All AA-7000 Series Instruments

Symptoms	Cause / Details	Corrective Action
The indicator does not light when the power switch is turned ON.	The power plug or power cable is disconnected.	Plug in the power plug and power cable.
A wavelength origin error occurs at initialization.	There is something blocking the light path in the atomizer.	If there is something blocking the light path, remove it.
	The position of the deuterium lamp is not adjusted appropriately.	Adjust the position of the deuterium lamp. See 8.8.2.2 "Adjusting Position of Deuterium Lamp".
	The lit time of the deuterium lamp has exceeded the cumulative life time of the lamp.	Replace the deuterium lamp. See 8.8 "Replacing the Deuterium Lamp".
The ASC-7000 or GFA- 7000A is not connected at	The power switch of the ASC-7000 or GFA-7000A is OFF.	Turn the power switch ON. See 2.1.1 "Activation".
initialization.	The ASC-7000 or GFA-7000A communications cable is disconnected.	Connect the communications cable. See 1.4.1 "Operation Switches/ Connectors".
The signal-to-noise ratio is bad (there is a lot of baseline noise).	Either the hollow cathode lamp for the target element has not been installed, or it has been installed in a socket that differs from the analysis conditions.	Correctly install the hollow cathode lamp for the target element. See 1.4.4 "Hollow Cathode Lamp Turret" and 8.9 "Method for Replacing the Hollow Cathode Lamp".
	The window plates at left and right of the burner compartment are soiled.	Wipe off the soiling with a soft cloth moistened with alcohol.
	The slit width setting is not appropriate.	Change the slit width to suit the target element by referring to the standard analysis conditions. See 10.2 "Measurement Conditions Table for Flame Atomic Absorption Analysis". For furnace analysis, refer to 6.2 "Standard Analysis Conditions" in GFA-7000A Instruction Manual.
	The lit time of the deuterium lamp has exceeded the cumulative life time of the lamp.	Replace the deuterium lamp. See 8.8 "Replacing the Deuterium Lamp".
	The deuterium lamp is not lit.	

Symptoms	Cause / Details	Corrective Action
The signal-to-noise ratio is bad (there is a lot of baseline noise).	The lit time of the hollow cathode lamp has exceeded the cumulative life time of the lamp.	Replace the hollow cathode lamp. See 8.9 "Method for Replacing the Hollow Cathode Lamp".
	The hollow cathode lamp does not light.	
The line search / beam balancing failed.	There is something blocking the light path in the atomizer.	If there is something blocking the light path, remove it.
	Either the hollow cathode lamp for the target element has not been installed, or it has been installed in a socket that differs from the analysis conditions.	Correctly install the hollow cathode lamp for the target element. See 1.4.4 "Hollow Cathode Lamp Turret" and 8.9 "Method for Replacing the Hollow Cathode Lamp".
	The window plates at left and right of the burner compartment are soiled.	Wipe off the soiling with a soft cloth moistened with alcohol.
	The slit width setting is not appropriate.	Change the slit width to suit the target element by referring to the standard analysis conditions. See 10.2 "Measurement Conditions Table for Flame Atomic Absorption Analysis". For furnace analysis, refer to 6.2 "Standard Analysis Conditions" in GFA-7000A Instruction Manual.
	The lamp current value setting is not appropriate.	Set the lamp current value that is appropriate for the target element by referring to the standard analysis conditions. See 10.2 "Measurement Conditions Table for Flame Atomic Absorption Analysis". For furnace analysis, refer to 6.2 "Standard Analysis Conditions" in GFA-7000A Instruction Manual.
	The lamp mode has been set to BGC- D2 for an element that does not have an analysis line in the wavelength range for which background compensation using the deuterium lamp is possible (185 to 430 nm).	Set the lamp mode to NON-BGC or BGC-SR (a hollow cathode tube compatible with the SR method is required).
	The wavelength memory setting is not appropriate.	Clear the wavelength memory setting.
	The lit time of the hollow cathode lamp has exceeded the cumulative life time of the lamp.	Replace the deuterium lamp. See 8.8 "Replacing the Deuterium Lamp".
	The deuterium lamp is not lit.	

Symptoms	Cause / Details	Corrective Action
The line search / beam balancing failed.	The lit time of the hollow cathode lamp has exceeded the cumulative life time of the lamp.	Replace the hollow cathode lamp. See 8.9 "Method for Replacing the Hollow Cathode Lamp".
	The hollow cathode lamp does not light.	
The absorbance is –1 (minus one).	The light paths of the hollow cathode lamp and the deuterium lamp do not coincide. (When the BGC-D2 mode is set)	Adjust the position of the deuterium lamp. See 8.8.2.2 "Adjusting Position of Deuterium Lamp".
Spike noise is generated.	The hollow cathode lamp does not light stably.	After performing a line search, leave the lamp for about 15 minutes until it warms up. Perform a line search once again after warm-up.

9.2.2 AA-7000F, AA-7000F/AAC

Symptoms	Cause / Details	Corrective Action
The absorbance or energy value has fallen very considerably, or it does not move from zero.	Either the hollow cathode lamp for the target element has not been installed, or it has been installed in a socket that differs from the analysis conditions.	Correctly install the hollow cathode lamp for the target element. See 1.4.4 "Hollow Cathode Lamp Turret".
	The [AUTO ZERO] button has been pressed by mistake during measurement.	Press the [AUTO ZERO] button while spraying the solvent instead of the sample. Then resume measurement.
	The lit time of the deuterium lamp has exceeded the cumulative life time of the lamp.	Replace the deuterium lamp. See 8.8 "Replacing the Deuterium Lamp".
	The lit time of the hollow cathode lamp has exceeded the cumulative life time of the lamp.	Replace the hollow cathode lamp. See 8.9 "Method for Replacing the Hollow Cathode Lamp".
	The slot of the burner head is not on the optical axis.	Adjust the position of the atomizer in the forward/backward direction or the up/down direction to adjust the positional relationship between the burner slot and the optical axis. See 3.1.8 "Atomizer/Gas Flow Rate Setup".
	Deposits or foreign objects have become attached to the capillary tube in the nebulizer section.	Clean the capillary tube using a cleaning wire. See 8.2.2.3 "Cleaning the Capillary".
	The nebulizer has deteriorated.	Replace the nebulizer (ask your Shimadzu representative).
	The sampling tube has deteriorated.	Replace the sampling tube. See 8.2.2.1 "Removing and Mounting the Sampling Tube".
	The polyethene tube covering the capillary tube in the nebulizer section has broken.	Replace the polyethene tube. See 8.2.2.4 "Inspecting and Replacing the Polyethylene Tube".
The flame is not stable.	Deposits have become attached to the slot in the burner head.	Wash the burner head. See 8.2.1 "Burner Head Maintenance".
	The volume of gas remaining in the dissolved acetylene cylinder is low.	Replace the cylinder with a new one.

Symptoms	Cause / Details	Corrective Action
When using the high- temperature burner head (option), the instrument will not change from an air-	N ₂ O gas is not being supplied.	Open the main valve on the N_2O gas cylinder and supply N_2O gas at the stipulated pressure. See 10.6.3 "Gas Requirements".
acetylene (Air- C_2H_2) flame to a nitrous oxide- acetylene (N ₂ O- C_2H_2) flame.	The flame type is set to Air- C_2H_2 .	Set the flame type to $N_2O-C_2H_2$ on the gas condition setting screen.
The absorbance is –1 (minus one).	Light radiation derived from the flame or sample is exerting an influence.	Carry out a line search while spraying the blank solution.
		Carry out a line search while spraying the standard sample at the maximum concentration in the measurement range, and then perform auto zeroing while spraying the blank solution.
		Adjust the C_2H_2 flow rate and the burner angle.

9.3

The events that cause the buzzer to sound while using an AA-7000 series unit, and the action to take in each case, are described below.

NOTE

When the power to the instrument is turned ON, the buzzer sounds briefly once: "pip". After that, self diagnosis is performed and if an abnormality is found the buzzer sounds three times: "pip pip pip".

9.3.1 When the Buzzer Sounds Continuously (beep, beep)

A gas leak has been detected.



• Follow the "Measures When Gas Leakage Is Detected".

WARNING

• When a gas leak has been detected, stop using the instrument immediately. Leaked gas could be ignited, causing a fire.

NOTE

If no gas leak is detected as the result of a gas leakage inspection, the message shown below is displayed. Click [OK] and you will be able to ignite the flame.

WizAArd	<u>×</u>
<u>.</u>	No gas leak is detected. Before you ignite, set the BURNER SELECT switch to Air-C2H2 and pushing the PURGE button, adjust Air flow rate between 13.5 and 17.5 L/min by the flowmeter knob. (In case optional flowmeter is attached.) After ignition, adjust gas supply pressure to maintain Fuel to 0.09MPa and Support to 0.35MPa during combustion.
	СССК

9.3.2 When the Buzzer Sounds Continuously (pippip, pippip)

The liquid level in the drain tank is too low.

WizAArd	X
8	Drain tank water level is too low.(E 33345)
	()

• If this error occurs even when the drain tank is topped up with water, stop using the instrument and contact your Shimadzu representative.

The ventilation fan inside the AA main unit has stopped.



- Check if there is anything obstructing the fan.
- If the fan is stopped, or if an error occurs even though the fan is operating, stop using the instrument and contact your Shimadzu representative.

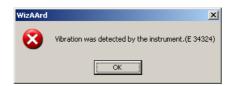
9.3.3 When the Buzzer Sounds Four Times (beep, beep, beep, beep)

A flashback has occurred.

WizAArd		×
8	Flashback has occurred!! Close the Fuel eas supply valve immediately, and contact your Shimadzu service representative (E 34325)	
	C OK	

• Follow the "Measures When Flashback Occurs" in the Introductory section.

The instrument has detected vibration.



• Follow the "Emergency Action" included in the introductory section.

• After confirming safety in the vicinity, display the [Gas Controller Status] dialog box and click the [Reset] button.

Gas Leak Check	Checking	(No ignition:5min. to go)	
Drain Level	ОК		
Support Gas Pressure Monitor Level	LOW		
Fuel Gas Pressure Monitor Level	LOW		
Vibration Sensor	NG	Reset	
Fan Stop Sensor	ок		
Burner Select Sensor Check	ОК		
Drain Level Sensor Check	ок	Check expires 30 days later	
Support Gas Pressure Monitor Check(Air)	ОК		
Support Gas Pressure Monitor Check(N2O)	ОК	Check expires 30 days later	
Fuel Gas Pressure Monitor Check	ОК	Every Initialization	
Flashback Monitor	Flashbac	Flashback is not detected.	
	[

The fuel gas pressure is too low.



- If this error occurs, it is necessary to conduct a gas leak check before ignite again. The gas leak check will start automatically.
- If this error occurs even though fuel gas is being supplied to the instrument, stop using the instrument and contact your Shimadzu representative.

The support gas pressure is too low.

WizAArd	X	
8	Support gas pressure is too low.(E 33343)	
	()	

• If this error occurs even though assist gas is being supplied to the instrument and the flame ignites, stop using the instrument and contact your Shimadzu representative.

The flame has gone out.



• If this error occurs even though there is nothing obstructing the flame monitor, stop using the instrument and contact your Shimadzu representative.

The environs of the flame monitor are too bright.

WizAArd	×
8	It is too bright around the Flame Monitor.(E 34318)
	ОК

- Lessen the amount of external light and illumination entering the burner compartment to make the inside of the chamber dark.
- If this error occurs even though the inside of the burner compartment has been made dark, stop using the instrument and contact your Shimadzu representative.

Error Messages

Please note that error messages not listed in the table may be displayed.

If the remedial measure does not solve the problem or the error message is not listed in the table, please contact your Shimadzu representative.

NOTE

Before contacting your Shimadzu representative, record the following items.

- 1. Content of error message
- 2. Status in which the error message is displayed (indication screen, performed operation, measurement file, etc.)
- 3. Instrument information displayed at the upper in the initialization screen (model names of AA/ASC/GFA, ROM version and serial No.)
- 4. AA software version No. ([Help]-[About Wizard] menu)

Error Message	Cause/Details	Remedial Measure
Number		-
x,x,x,This lamp ID is already in use. Please select another ID.		 Select [None] for the Element field to clear the overlapped lamp in [Lamp Position Setup] dialog box. When using plural lamps of the same element and type simultaneously, register each of the lamps in [Instrument]-[Lamp History].
Α		
ACK has not been accepted from ASC.	Communication protocol with ASC was not completed. (:A)	Verify the ASC power supply and the cable connection between ASC and AA main unit, then connect the ASC in [Instrument]-[Option Connect] menu.
ACK has not been accepted from GFA.	Communication protocol with GFA was not completed. (:A)	Verify the GFA power supply and the cable connection between GFA and AA main unit, then connect the GFA in [Instrument]-[Option Connect] menu.
Flashback has occurred!! Close the fuel gas supply valve immediately, and contact your Shimadzu service representative.		Stop using the instrument and contact your Shimadzu representative.
Analytical Line is not found.	 Line Search has failed. The following causes are considerable. (1) The lamp is not properly mounted to the lamp turret. (2) The wavelength shift has occurred in the monochrometer. 	 Verify that the lamp is correctly mounted to the socket and the lamp turret position. Perform the wavelength correction in [Instrument]-[Maintenance]- [Wavelength Adjustment].
Another error No. was issued from GFA.	The ROM program of the AA main unit has a problem.	Record the instrument information displayed on the initialization screen and the status in which this error occurs. Then contact your Shimadzu representative.

Error Message	Cause/Details	Remedial Measure
Another error occurred while recovering ASC error.	The ROM program of the AA main unit has a problem.	Record the instrument information displayed on the initial screen and the status in which this error occurs. Then contact your Shimadzu representative.
ASC arm motor slipped.		 Verify that the ASC arm movement is not impeded. Switch on the ASC again and connect the ASC in [Instrument]- [Option Connect] menu.
ASC arm rotation position cannot be initialized.	 The origin detector didn't detect the origin correctly. The arm rotation drive motor does not operate. 	 (1), (2) Switch on the ASC main unit again. If the error occurs again, record the status in which this error message appears and contact your Shimadzu representative.
ASC arm vertical position cannot be initialized.	 The origin detector didn't detect the origin correctly. The vertical arm drive motor does not operate. 	 (1), (2) Switch on the ASC main unit again. If the error occurs again, record the status in which this error message appears and contact your Shimadzu representative.
ASC cannot be detected.	Connection with ASC was interrupted.	Verify the ASC power supply and the cable connection between ASC and AA main unit, then connect the ASC in [Instrument]-[Option Connect] menu.
ASC Error occurred during action. Press Cancel to close this dialog box. Resolve the ASC problem then retry.		Remove the cause of error and switch on the ASC power again. Then connect the ASC in [Instrument]-[Option Connect] menu and try this operation again.
ASC I/F port is used for another device.	Communication with ASC is unstable.	Verify the ASC power supply and the cable connection between ASC and AA main unit, then connect the ASC in [Instrument]-[Option Connect] menu.
ASC is disconnected, or ASC is set not to be used in sequence parameter settings.	The ASC is not connected or the setup for using ASC is not made, although [ASC Sample Pos. for EMISSION Line Search] for Emission mode is selected in the [Optics Parameters] page to execute the Line Search/Beam Balance in Emission mode.	 Switch on the ASC and use [Instrument]-[Option Connect] menu to connect the ASC. To make a setting to use the ASC, select [Using ASC] check box in the [Sequence] page.
ASC Syringe 1 cannot be initialized.	 The origin detector didn't detect the origin correctly. The syringe drive motor does not operate. 	(1), (2) Switch on the ASC main unit again.If the error occurs again, record the status in which this error message appears and contact your Shimadzu representative.

Error Message	Cause/Details	Remedial Measure
ASC table cannot be initialized.	 (1) The origin detector didn't detect the origin correctly. (2) The turntable drive motor does not operate. 	 (1), (2) Switch on the ASC main unit again. If the error occurs again, record the status in which this error message appears and contact your Shimadzu representative.
ASC table motor slipped.		 Verify that rotation of the ASC table is not impeded. Switch on the ASC again and connect the ASC in [Instrument]-[Option Connect] menu.
ASC Time out.	The ASC does not response on the way of the communication. The communication cable with ASC has a problem.	Verify the ASC power supply and the cable connection between ASC and AA main unit, then connect the ASC in [Instrument]-[Option Connect] menu.
В		
Background signal is too large to execute Beam Balance.	 Beam Balance failed because the light intensity of BG measurement is too greater than the light intensity of hollow cathode lamp. The following causes are considerable. (1) The light intensity of the hollow cathode lamp becomes too smaller than the light intensity of BG measurement as the result of change in the Optics Parameters, etc. (2) The Line Search failed to catch the correct analytical line. (3) The light intensity becomes smaller because of the exhaustion of hollow cathode lamp. 	 (1) Increase the hollow cathode lamp current (Low) (pay attention to the max. current). When the lamp mode is BGC-D2, narrow the slit width. (2) Set [Wavelength] correctly in [Optics Parameters] page. If the wavelength shift has occurred in the monochrometer, perform the wavelength correction in [Instrument]-[Maintenance]-[Wavelength Adjustment]. (3) Change the hollow cathode lamp.

Error Message	Cause/Details	Remedial Measure
Background signal is too small to execute Beam Balance.	 Beam Balance failed because the light intensity of hollow cathode lamp is too greater than the light intensity of BG measurement. The following causes are considerable. (1) The light intensity of hollow cathode lamp becomes too greater than the light intensity of BG measurement as the result of change in the Optics Parameters, etc. (2) The Line Search failed to catch the correct analytical line. (3) The light intensity becomes greater because of the exhaustion of hollow cathode lamp. (4) When the lamp mode is BGC-D2, the light intensity of BG measurement diminishes due to exhaustion of the D2 lamp. (5) When the lamp mode is BGC-D2, 	 (1) Decrease the hollow cathode lamp current (Low). When the lamp mode is BGC-D2, widen the slit width (pay attention to the neighboring line). (2) Set [Wavelength] correctly in [Optics Parameters] page. Narrow the slit width so as not to catch the neighboring line. If the wavelength shift has occurred in the monochrometer, perform the wavelength correction in [Instrument]-[Maintenance]- [Wavelength Adjustment]. (3) Change the hollow cathode lamp. (4) Change the D2 lamp position in
BG signal is too large.	 the optical axis of the D2 lamp is shifted. Beam Balance failed because the light intensity of BG measurement is too greater than the light intensity of hollow cathode lamp. The following causes are considerable. (1) The light intensity of the hollow cathode lamp becomes too smaller than the light intensity of BG measurement as the result of change in the Optics Parameters, etc. (2) The Line Search failed to catch the correct analytical line. (3) The light intensity becomes smaller because of the exhaustion of hollow cathode lamp. 	 [Instrument]-[Maintenance]-[D2 Lamp Position]. (1) Increase the hollow cathode lamp current (Low) (pay attention to the max. current). When the lamp mode is BGC-D2, narrow the slit width. (2) Set [Wavelength] correctly in [Optics Parameters] page. If the wavelength shift has occurred in the monochrometer, perform the wavelength correction in [Instrument]-[Maintenance]-[Wavelength Adjustment]. (3) Change the hollow cathode lamp.

Error Message	Cause/Details	Remedial Measure
BG signal is too small.	Beam Balance failed because the light intensity of hollow cathode lamp is too greater than the light intensity of BG measurement. The following causes are considerable.	 (1) Decrease the hollow cathode lamp current (Low). When the lamp mode is BGC-D2,
	 The lonowing causes are considerable. The light intensity of hollow cathode lamp becomes too greater than the light intensity of BG measurement as the result of change in the Optics Parameters, etc. The Line Search failed to catch the correct analytical line. [When the lamp mode is BGC-D2] The light intensity of BG measurement becomes smaller because of the exhaustion of D2 lamp. The optical axis of D2 lamp is shifted. The light intensity becomes greater because of the exhaustion of hollow cathode lamp. 	 widen the slit width (pay attention to the neighboring line). (2) Set [Wavelength] correctly in [Optics Parameters] page. Narrow the slit width so as not to catch the neighboring line. If the wavelength shift has occurred in the monochrometer, perform the wavelength correction in [Instrument]-[Maintenance]- [Wavelength Adjustment]. (3) Change the hollow cathode lamp. (4) Change the D2 lamp. (5) Adjust the D2 lamp position in [Instrument]-[Maintenance]-[D2
Blank cannot be inserted	Inserting a blank measurement row	Lamp Position]. Execute the blank measurement after
here.	between the repeat measurement rows was tried.	the repeat measurements are finished.
Buffer Overflow.	Amount of the acquired data is larger than the communication buffer of AA software.	Switch on the PC power and the instrument power again. If the error occurs again, check the following items.
		 Set all of the PC BIOS setup and the electric power saving function of control panel to OFF.
		 Don't start up other application software simultaneously.
C		
Calibration Curve cannot be created.	The calibration curve order does not match with the number of standard samples.	When the Zero Intercept is not selected, set the calibration curve order below the number of standard samples. When the Zero Intercept is selected, set the calibration curve order below the number of standard samples or the same.
Calibration Curve does not exist!	Standard samples (STD or MSA) have not been measured.	Set up STD, in the case of calibration curve method, and MSA, in the case of standard addition method or simple standard addition method, on the MRT work sheet and measure them.

Error Message	Cause/Details	Remedial Measure
Calibration Curve is not valid!	The calibration curve order does not match with the number of standard samples.	When the Zero Intercept is not selected, set the calibration curve order below the number of standard samples. When the Zero Intercept is selected, set the calibration curve order below the number of standard samples or the same.
Cannot load file. Measurement Type (Calibration/SMSA) is different.		Change the current setting of [Measurement Type] to other type in [Edit Preparation Parameters] dialog box then read out the target file.
Cannot load file. Mixing ON/ OFF status is different.		Change the current setting of [Mixing ON] check box to the opposite one in [Edit Preparation Parameters] dialog box then read out the target file.
Can't find drive X:	The setup disk of AA software is broken (at the time of installation).	Try to recover the setup disk using the scan disk of Windows.
Can't heat furnace during the GFA transformer cooling period. Please wait for transformer to cool then retry the operation.	Heating was tried before the GFA transformer had been cooled sufficiently.	Wait until the GFA transformer is cooled sufficiently, and then try the operation again.
Can't set temperature program when the GFA heats furnace.	Sending the command to change the furnace program was tried during heating.	Set the furnace program after the heating is finished.
Communication Time out.	The communication between AA software and AA main unit has a problem.	 Switch on the PC power and the instrument power again. If the error occurs again, check the following items. Set all of the PC BIOS setup and the electric power saving function of control panel to OFF. Fix all the communication cables (AA-PC, AA-ASC, AA-GFA) with screws to avoid an insufficient
		contact and ground level mismatch of communication port.

Error Message	Cause/Details	Remedial Measure
Communication was aborted from the spectrophotometer side.	The communication between AA software and AA main unit has a problem.	Switch on the PC power and the instrument power again. If the error occurs again, check the following items.
		Set all of the PC BIOS setup and the electric power saving function of control panel to OFF.
		 Fix all the communication cables (AA-PC, AA-ASC, AA-GFA) with screws to avoid an insufficient contact and ground level mismatch of communication port.
Could not stop Chopper Mirror at the position where it does not cut off the light beam. We recommend you to retry it.		Display the drop-down list of measurable elements from the Toolbar and then click on the element under measurement.
Current measurement schedule cannot be selected.	The lamp of current measurement element cannot be selected as warm- up lamp.	Select [Lamp ON] check box in the [Optics Parameters] page to light on the lamp of current measurement element.
D		
D2 lamp energy is too low	 (1) The light intensity becomes smaller as the result of D2 lamp exhaustion. (2) The extinct exits of D2 lamp is 	 Change the D2 lamp. Adjust the D2 lamp position in [Instrument]-[Maintenance]-[D2
	(2) The optical axis of D2 lamp is shifted.	Lamp Position].
D2 lamp energy is too low	Beam balance failed. The following causes are considerable.	(1) Change the D2 lamp.(2) Adjust the D2 lamp position in
	 The light intensity becomes smaller as the result of D2 lamp exhaustion. 	[Instrument]-[Maintenance]-[D2 Lamp Position].
	(2) The optical axis of D2 lamp is shifted.	
Drain tank water level is too low.	A flashback may be caused by an ignition without sufficient water in drain tank. Therefore, be sure to fill the drain tank with water before igniting a flame.	Fill water via the breather at the top of drain tank little by little until the water overflows from the drain tank to waste container.
E		
Emission vial position has not been set in the following measurement.	Although the ASC is to be used to execute Line Search/Beam Balance in Emission mode in the setting, [ASC Sample Pos. for EMISSION Line Search] in [Optics Parameters] page is set to [NONE].	Set the position of sample to be sprayed at the time of Line Search/ Beam Balance in [ASC Sample Pos. for EMISSION Line Search]. Entry for [ASC Sample Pos. for EMISSION Line Search] is available when the [Lamp Mode] is set to "EMISSION" and [Lamp Current (Low)] to "0".

Error Message	Cause/Details	Remedial Measure
Error: Pressure Too Low!	In the case of Ar gas pressure The Ar gas pressure supplied for GFA is too low.	In the case of Ar gas pressure Open the stopcock of Ar gas and adjust the pressure regulator so that the supply pressure satisfies the specification.
Expired Lamp is selected. Reestablish the setting?	The lamp selected in [Lamp Position Setup] dialog box has a relation "(Life Time) < (Used Time)".	 Although the [Used Time] is one of the signs to change the lamp, a lamp used for longer than the life time can be used if Line Search/ Beam Balance is performed successfully and if the noise level required for the analysis is satisfied.
		 Select [Instrument]-[Configuration] menu and clear the check box of [Show lamp used time over operating life message] not to display this message.
F		
Failed to Add New Schedule.	When the number of Element Schedules on the MRT work sheet exceeds 20, the excess schedules cannot be read out on the MRT.	Delete unnecessary Element Schedules in [Element Selection] page. Also, clear the check box of unnecessary element in [Element Selection] list when reading out parameters in the [Template] page of [Load Parameters] dialog box.
Failed to detect burner moving motor (fore/back) origin.	 The origin detector couldn't detect the origin correctly. The burner fore/back movement motor does not operate properly. 	 (1), (2) Switch on the AA main unit again. If the error occurs again, record the status in which this error message appears and contact your Shimadzu representative.
Failed to detect burner moving motor (up/down) origin.	 The origin detector couldn't detect the origin correctly. The burner up/down movement motor does not operate properly. 	(1), (2) Switch on the AA main unit again.If the error occurs again, record the status in which this error message appears and contact your Shimadzu representative.
Failed to detect lamp turret motor origin.	 The origin detector couldn't detect the origin correctly. The lamp turret motor does not operate properly. 	 (1), (2) Switch on the AA main unit again. If the error occurs again, record the status in which this error message appears and contact your Shimadzu representative.
Failed to detect slit motor origin.	 The origin detector didn't detect the origin correctly. The slit drive motor does not operate. 	(1), (2) Switch on the AA main unit again.If the error occurs again, record the status in which this error message appears and contact your Shimadzu representative.

Error Message	Cause/Details	Remedial Measure
Failed to detect wavelength motor origin.	 The origin detector couldn't detect the origin correctly. The wavelength drive motor does not operate properly. 	
Failed to extinguish the flame.	EXTINGUISH button of the instrument was not pressed while executing the remaining gas combustion.	Press the EXTINGUISH button following the instruction displayed while executing the residual gas combustion.
Failed to load Cookbook	The "cookbook.ref" file in the folder in which the AA software is installed cannot be read in. The version of "cookbook.ref" does not match the execution file, or the file is broken.	Delete the "cookbook.ref" file and install it again.
Failed to load document.	Measurement file or template file cannot be read out in [Wizard Selection] dialog box.	 Check the following points. The file tried to load actually exists. The file name contains no period except the period just before the extension (for example, a file named "Cu.Ag.aa" cannot read out).
Failed to open the serial port.	The PC serial port cannot be used.	 Check the following points. [COM port] in [Instrument]- [Configuration] is properly set. Other application software (communication software, FAX software, etc.) is not used. The resource of the communication port does not compete with other device.
Failed to send parameters to the instrument. Wait until the instrument becomes READY then retry it.		Close this message and wait until [READY] is displayed at the right lower of the status bar. Then perform the target operation again.
Failed to send the furnace program for GFA cleaning.	The GFA instrument type selected in the AA software is wrong.	Set the correct [GFA] in [Instrument]- [Configuration] menu then start up the AA software again.
Failed to send the temperature coefficient.	The GFA instrument type selected in the AA software is wrong.	Set the correct [GFA] in [Instrument]- [Configuration] menu then start up the AA software again.
Failed to send the temperature stabilization time.	The GFA instrument type selected in the AA software is wrong.	Set the correct [GFA] in [Instrument]- [Configuration] menu then start up the AA software again.
Failed to set Flame Monitor Safety Device to OFF.	Flame monitor couldn't be set OFF while executing the residual gas combustion.	Stop using the instrument, switch on the power again and connect it with the PC again. If the item "Flame Monitor ON" becomes NG, contact your Shimadzu representative.

Error Message	Cause/Details	Remedial Measure
Failed to turn OFF the gas pressure monitor.	The gas pressure monitor couldn't be set OFF while executing the residual gas combustion.	Stop using the instrument. Then switch on the power again and reconnect it with the PC. If NG is displayed for the item "Gas Pressure Monitor ON" on the initialization screen, contact your Shimadzu representative.
Failed to turn ON the gas pressure monitor.	The gas pressure monitor couldn't be set ON when the residual gas combustion was finished or cancelled.	Stop using the instrument. Then switch on the power again and reconnect it with the PC. If NG is displayed for the item "Gas Pressure Monitor ON" on the initialization screen, contact your Shimadzu representative.
Flame has been extinguished.	The flame monitor detected that the flame is extinguished although the EXTINGUISH button is not pressed.	 Verify that nothing obstructs the flame monitor. Remove the cause(s) of the flame extinguishment.
Flame Monitor Safety Device was not set.	Flame monitor couldn't be set ON while executing the residual gas combustion.	Stop using the instrument, switch on the power again and connect it with the PC again. If the item "Flame Monitor ON" becomes NG, contact your Shimadzu representative.
Fuel gas pressure is too low.	The supply pressure of fuel gas (C_2H_2) or support gas (Air or N ₂ O) has decreased.	Open the stopcock and check the supply pressure of fuel/support gas by the meter of pressure regulator. If the required pressure is not satisfied, change the fuel/support gas cylinder, check/adjust the air compressor operation or perform other appropriate treatment.
Furnace is not sufficiently cooled. Check that cooling water is supplied properly.	Heating was tried before the graphite furnace is cooled sufficiently. The temperature of the cooling water is outside the acceptable range or the flow of the cooling water is not sufficient.	 Wait until the graphite tube is cooled sufficiently, and then try the operation again. Verify that the cooling water temperature and pressure are within the specifications of the instrument and that the cooling tubes are not pressed or bent.
Furnace program does not exist.	Original furnace program for the optimum furnace program search has not been set up.	Return to [Furnace Program] page and set up a furnace program.
G		
GFA cannot be detected. GFA gas pressure is too	Connection with GFA was interrupted.	Verify the GFA power supply and the cable connection between GFA and AA main unit, then connect the GFA in [Instrument]-[Option Connect] menu. Open the Ar gas stopcock and verify
low.		that the supply pressure satisfies the specification.

Error Message	Cause/Details	Remedial Measure
GFA heating is OFF. 1.Turn on the power switch (HEAT) of GFA. 2.Make sure that a graphite tube is set and the graphite furnace is fastened. GFA I/F port is used for other device.	If this error occurs again after executing the measures following the message, the contact between the graphite cap or holder and the graphite tube may be improper. Communication with GFA is unstable.	Open the graphite furnace and rotate the graphite tube forward and back while pressing the end of the graphite tube to the graphite cap or holder. Through this procedure, the contact with the graphite tube will be recovered. Verify the GFA power supply and the cable connection between GFA and AA main unit, then connect the GFA in
GFA is NOT initialized. Please perform option connect. If you see this message during option connect, check GFA instrument type in [Instrument] -[Configuration].	 Although the initialization command of GFA had not been sent, other command was sent. (1) The power supply of GFA has once failed then recovered. (2) The initialization command was not sent because the GFA instrument type selected in the AA software was wrong. 	 [Instrument]-[Option Connect] menu. (1) Verify the GFA power supply and the cable connections between GFA and AA main unit, then connect the GFA in [Instrument]- [Option Connect] menu. (2) Set the correct [GFA] in [Instrument]-[Configuration] menu then start up the AA software again.
GFA returns invalid Temperature Coefficient! - (Value of Invalid Temperature Coefficient)-	The temperature calibration couldn't be performed correctly.	Switch on the GFA again. If the error occurs again, record the number indicated in this message and the status in which this error message appears. Then contact your Shimadzu representative.
GFA serial interface cannot be opened.	The ROM program of the AA main unit has a problem.	Record the instrument information displayed on the initialization screen and the status in which this error occurs. Then contact your Shimadzu representative.
GFA Temperature Calibration Error.	The graphite tube may have been worn out. Or the optical temperature sensor has a problem.	Change the graphite tube for a good one. If the error occurs again, contact your Shimadzu representative.
GFA Time out.	The GFA does not response on the way of the communication. The communication cable with GFA has a problem.	Verify the GFA power supply and the cable connection between GFA and AA main unit, then connect the GFA in [Instrument]-[Option Connect] menu.

Error Message	Cause/Details	Remedial Measure
н		
HC Lamp Energy is insufficient.	 Sufficient light intensity of hollow cathode lamp could not be obtained during the wavelength adjustment. The following causes are considerable. (1) The optical axis has a problem. (2) The light intensity of the hollow cathode lamp becomes too small as the result of change in the Optics Parameters, etc. (3) The Line Search failed to catch the correct analytical line. (4) The light intensity becomes smaller because of the exhaustion of hollow cathode lamp. 	 (1) Verify that the atomizer or any other thing does not obstruct the optical axis. Verify that the lamp is correctly mounted to the socket and the lamp turret position. Rotate the hollow cathode lamp and adjust the optical axis. (2) Increase the hollow cathode lamp current (Low) (pay attention to the max. current). Widen the slit width (pay attention to neighboring lines). (3) Set [Wavelength] correctly in [Optics Parameters] page. (4) Change the hollow cathode lamp.

Error Message	Cause/Details	Remedial Measure
Incorrect interface handshake with GFA.	The ROM program of the AA main unit has a problem.	Record the instrument information displayed on the initialization screen and the status in which this error occurs. Then contact your Shimadzu representative.
Instrument failed to execute the command. Please restart the instrument and software and retry Leak Check.	Gas leak check start command was not executed properly.	Switch on the PC power and instrument power again. If the error occurs again, contact your Shimadzu representative.
Instrument is not ready!	 A command was tried to send when the communication between the instrument and PC is OFF. While the instrument is executing the command, the next command was tried to send. 	 Set the communication with the instrument to ON from [Instrument]-[Connect] menu. Close this message box and wait until the command currently executed in the instrument is
		finished. Perform the target operation again after "READY" is displayed at the right lower of the status bar.
Instrument is still busy.	The operation that cannot be executed during BUSY status of instrument was tried.	Close this message and wait until [READY] is displayed at the right lower of the status bar. Then perform the target operation again.
Invalid ASC Command.	A command that is not supported by the ASC was sent from the AA software.	Verify that the AA software supports the your ASC.
Invalid ASC Parameter.	Parameters that are not supported by the ASC were sent from the AA software.	Verify that the AA software supports the your ASC.
Invalid error No. has been sent from ASC.	The ROM program of the AA main unit has a problem.	Record the instrument information displayed on the initialization screen and the status in which this error occurs. Then contact your Shimadzu representative.
Invalid error No. was sent from GFA.	The ROM program of the AA main unit has a problem.	Record the instrument information displayed on the initialization screen and the status in which this error occurs. Then contact your Shimadzu representative.
Invalid GFA Command	A command that is not supported by the GFA was sent from the AA software.	Verify that the [GFA] is correctly selected in the [Instrument]- [Configuration].
Invalid GFA Parameters	Parameters that are not supported by the GFA were sent from the AA software.	Verify that the [GFA] is correctly selected in the [Instrument]- [Configuration].

Error Message	Cause/Details	Remedial Measure
It is too bright around the Flame Monitor.	Darken the inside of the burner compartment by turning down the light (external light or illumination) entering the burner compartment.	If this does not work out: Completely cover the top of the burner compartment with a plate having a light blocking effect. If the error is still generated, the flame monitor is suspected to malfunction. Contact your Shimadzu representative.
J,K		1
L		
Lamp energy is too low to execute Beam Balance.	 Beam Balance failed because the light intensity of hollow cathode lamp is too small. The following causes are considerable. (1) The light intensity of the hollow cathode lamp becomes too small as the result of change in the Optics Parameters, etc. (2) The Line Search failed to catch the correct analytical line. (3) The light intensity becomes smaller because of the exhaustion of hollow cathode lamp. 	 (1) Increase the hollow cathode lamp current (Low) (pay attention to the max. current). Widen the slit width (pay attention to neighboring lines). (2) Set [Wavelength] correctly in [Optics Parameters] page. If the wavelength shift has occurred in the monochrometer, perform the wavelength correction in [Instrument]-[Maintenance]-[Wavelength Adjustment]. (3) Change the hollow cathode lamp.
Lamp History data file (lamphist.ref) cannot be found.	 (1) "lamphist.ref" file does not exist in the folder in which the AA software is installed. (2) Execution file (wizaa.exe) was copied in the folder other than the installation folder and was executed. 	 (1) Check whether the name "lamphist.ref" was changed or not and whether it has been moved to other folder or not. If so, set it back to original condition. If "lamphist.ref" file does not exist in the install folder, the AA software creates a new file. (2) Be sure to execute "wizaa.exe" from the install folder.
Lamp ID is not valid. Register the proper lamp in Lamp History dialog box, then set the lamp turret position.	In [Lamp Position Setup] dialog box, select a lamp to be used from the lamps registered in [Lamp History] dialog box. Therefore, register the lamp in [Lamp History] dialog box when using a lamp that is not registered.	Select [Instrument]-[Lamp History] menu and register the lamp to be used.

Error Message	Cause/Details	Remedial Measure
Lamp Socket # is not selected in this schedule.	[Socket #] in the [Optics Parameters] page of the element for warm-up is set [NONE].	Set up the Socket # in the [Optics Parameters] page of the element for warm-up. When the Socket # you wish to set is not displayed in the list, set up the lamp mounted to the Socket # in [Lamp Pos. Setup]. (If the Lamp ID of the lamp you wish to set is not displayed in the [Lamp Position Setup] dialog box, register the lamp in [Instrument]-[Lamp History]).
Lamp Socket # is wrong. This schedule cannot be selected for warmup lamp.	[Socket #] of the element for warm-up in the [Optics Parameters] page is set [NONE].	Set up the Socket # in the [Optics Parameters] page. When the Socket # you wish to set is not displayed in the list, set up the lamp mounted to the Socket # in [Lamp Pos. Setup]. (If the Lamp ID of the lamp you wish to set is not displayed in the [Lamp Position Setup] dialog box, register the lamp in [Instrument]-[Lamp History].)
Lamp socket# is not specified! Please set socket# for the following elements.		In the [Optics Parameters] page of the element named in the message, set the socket number. If the aimed socket number is not displayed on the list, set the lamp mounted to the aimed socket number in [Lamp Pos. Setup]. (Furthermore, if the lamp of aimed element and type is not displayed on the list in [Lamp Position Setup] dialog box, register the lamp in [Instrument]- [Lamp History].)
Lamp Used Time exceeded the Life Time	In [Lamp History] dialog box, the lamp to be used has a relation "(Life Time) < (Used Time)".	 Although the [Used Time] is one of the sign to change the lamp, a lamp used for longer than the life time can be used if Line Search/ Beam Balance is performed successfully and if the noise level required for the analysis is satisfied. Select [Instrument]-[Configuration] menu and clear the checkbox of [Show lamp used time over operating life message] not to display this message.

Error Message	Cause/Details	Remedial Measure
Μ		
Memory Lock Failure	The memory reserved for communication by AA software couldn't be locked.	 Switch on the PC power and the instrument power again. If the error occurs again, check the following items. Set all of the PC BIOS setup and the electric power saving function of control panel to OFF. Don't start up other application software simultaneously.
Mixing is OFF and Injection volume is more than 100uL. Are you sure?	If the injection volume is too large, the sample overflows from the graphite tube.	Select an appropriate injection volume after checking that the sample does not overflow from the graphite tube.
Momentary electric shutdown occurred in the gas controller. Forcibly support gas type is changed to Air and flame monitor is set to ON.	Momentary power failure of the AC power supply was detected.	Switch on the AA main unit again. If the error occurs again, record the status in which this error occurs. Then contact your Shimadzu representative.
Ν		
No lamps which can be used for this measurement are set. Set a lamp which is suitable for measurement element and lamp mode.	[Socket #] in the [Optics Parameters] page is set [NONE].	Set up the Socket # in the [Optics Parameters] page. When the Socket # you wish to set is not displayed in the list, set up the lamp mounted to the Socket # in [Lamp Pos. Setup]. (If the Lamp ID of the lamp you wish to set is not displayed in the [Lamp Position Setup] dialog box, register the lamp in [Instrument]-[Lamp History].)
No. of Mixing Cycles is Invalid.	The setting of Mixing ON is not properly saved in the measurement parameters.	Set up [Mixing ON] in [ASC Parameters] page again.
0		
Other program is using lamphist.ref file. Lamp data will not be saved.	 "lamphist.ref" file is a data file for lamp history. The following causes are considerable when this message is displayed. (1) The second AA software has been started up. (2) The file attribute of "lamphist.ref" is read-only. 	 "lamphist.ref" can be renewed from the AA software firstly started up but cannot be renewed from the AA software secondly started up. Therefore, use the AA software firstly started up to perform measurement. There is no problem to use the AA software secondly started up to edit parameters. Turn the read-only attribute of "lamphist.ref" file in AA software folder OFF.

Error Message	Cause/Details	Remedial Measure
Other program is using lampplace. ref file. Lamp data will not be saved.	 "lampplace.ref" file is a data file for lamp position setup. The following causes are considerable when this message is displayed. (1) The second AA software has been started up. (2) The file attribute of "lampplace.ref" is read-only. 	(1) "lampplace.ref" can be renewed from the AA software firstly started up but cannot be renewed from the AA software secondly started up. Therefore, use the AA software firstly started up to perform measurement. There is no problem to use the AA software secondly started up to edit parameters.
		(2) Turn the read-only attribute of "lampplace.ref" file in AA software folder OFF.
Р		
Periodic Blank Preparation Parameters is Invalid.		If an invalid value that is not usually entered has been entered in the Blank Samples table of the [Edit Preparation Parameters] dialog box, enter a correct value.
Please connect ASC.	 Although the autosampler is necessary for the operation, the ASC is not connected. Although the ASC is used in the measurement conditions, the ASC is not connected. 	 Verify the ASC power supply and the cable connection between ASC and AA main unit, then connect the ASC in [Instrument]- [Option Connect] menu. When the ASC is necessary for the measurement, connect the ASC in the procedure as above. When the ASC is not necessary, clear the [Using ASC] check box in the [Parameters]-[Edit Parameters]-[Sequence].
Please connect GFA serial interface.	The GFA power is set OFF or the communication cable between GFA and AA main unit is disconnected.	Verify the GFA power supply and the cable connection between GFA and AA main unit, then connect the GFA in [Instrument]-[Option Connect] menu. Fix the communication cable between GFA and AA main unit with the screws.
Please connect GFA.	The GFA is not connected although the GFA is necessary for the operation or measurement.	Verify the GFA power supply and the cable connection between GFA and AA main unit, then connect the GFA in [Instrument]-[Option Connect] menu.
Please connect the ASC serial interface.	The ASC power is set OFF or the communication cable between ASC and AA main unit is disconnected.	Verify the ASC power supply and the cable connection between ASC and AA main unit, then connect the ASC in [Instrument]-[Option Connect] menu. Fix the communication cable between ASC and AA with the screws.
Please select the Flame Type.	Flame type is not selected properly in [Atomizer/Gas Flow Rate Setup] page.	Select a correct flame type in [Atomizer/Gas Flow Rate Setup] page.

Error Message	Cause/Details	Remedial Measure
Please set the socket # combining even/odd in turn.	The lamps cannot be lit on simultaneously when their socket # combination is even/even or odd/odd. Therefore, when measuring many elements, it is recommended to set up the even/odd of the socket # in turn to	 Change the order of measurements in the [Element Selection] page so that the socket number combination may be even/odd in turn. When you wish not to change the
	perform efficient warm-up.	order of measurements or when changing the measurement order cannot make the even/odd combination, change the lamp mounting position by [Lamp Pos. Setup] in [Optics Parameters] page.
Please set the total time of Furnace Program less than 300 (sec).		Change the furnace program so that the total time of the furnace program may be within 300 seconds.
Please set the total time	Since the data sampling is started	[When the sampling stage is in the first
before the sampling stage more than 3 (sec).	before executing the sampling is started the furnace program, a stage of 3 seconds or longer is always necessary prior to the sampling stage.	row] Insert a stage of 3 seconds or longer before the sampling stage. [When the sampling stage is in the second row or after]
		Change the program so that the total time of the stages before the sampling stage may be 3 seconds or longer.
Please specify the lamp socket#.	Line Search/Beam Balance couldn't be executed because [Socket #] in the [Optics Parameters] page is set [NONE].	Set up the Socket # in [Optics Parameters] page of the current measurement element. When the Socket # you wish to set is not displayed in the list, set up the lamp mounted to the Socket # in [Lamp Pos. Setup]. (If the Lamp ID of the lamp you wish to set is not displayed in the [Lamp Position Setup] dialog box, register the lamp in [Instrument]-[Lamp History].)
Q,R		
S	1	T
Sample Preparation Parameters is Invalid.		If an invalid value that is not usually entered has been entered, enter a correct value in the Unknown Samples table in the [Edit Preparation Parameters] dialog box.
Schedule does not exist!	In the measurement using the ASC, Element schedule has not been selected, or the measurement procedure has not been set up although the element schedule exists.	Select the measurement element and set up the measurement procedure in [Action] fields on the MRT work sheet.

Error Message	Cause/Details	Remedial Measure
Serial interface cannot be opened.		The ROM program of the AA main unit has a problem. Record the instrument information displayed on the initialization screen and the status in which this error occurs. Then contact your Shimadzu representative.
Serial Port Error.	The PC serial port has a problem.	 Switch on the PC power and the instrument power again. If the error occurs again, check the following items. Set all of the PC BIOS setup and the electric power saving function of control panel to OFF.
		 Fix all the communication cables (AA-PC, AA-ASC, AA-GFA) with screws to avoid an insufficient contact and ground level mismatch of communication port.
		 To avoid overrun of PC serial port, lower the receive buffer of FIFO on the control panel.
Set the flame measurement as a current measurement.	The tried operation cannot be executed unless the flame measurement mode is set in the instrument.	After changing the measurement mode of the instrument to flame, execute the target operation. Perform the following operation to change the measurement mode of the instrument to flame. [When the flame measurement exists on the MRT] Select the flame measurement from the measurement element tool bar. [When the flame measurement does not exist on the MRT] Select [Parameters]-[Element Selection Wizard] and add a flame measurement by [Select Elements] in the [Element Selection] page. Then select the measurement from the [Meas. Element] drop-down list as current measurement element and click on [Finish].

Error Message	Cause/Details	Remedial Measure
Set the furnace	The tried operation cannot be executed	After changing the measurement mode
measurement as a current measurement.	unless the furnace measurement mode is set in the instrument.	of the instrument to furnace, execute the target operation. Perform the following operation to change the
		measurement mode of the instrument to furnace.
		[When the furnace measurement exists on the MRT]
		Select the furnace measurement from the measurement element tool bar. [When the furnace measurement does not exist on the MRT]
		Select [Parameters]-[Element Selection Wizard] and add a furnace measurement by <select elements=""> in</select>
		the [Element Selection] page. Then select the measurement from the
		[Meas. Element] drop-down list as current measurement element and click on [Finish].
Spectrophotometer has not been turned ON or cable is disconnected.		Switch on the PC power and the instrument power again. If the error occurs again, check the following items.
		 Verify that the communication cables between AA main unit and PC are connected properly and fixed with screws.
		• Set all of the PC BIOS setup and the electric power saving function of control panel to OFF.
Standard Preparation Parameters is Invalid.		If an invalid value that is not usually entered has been entered in the STD Samples table of the [Edit Preparation Parameters] dialog box, enter a correct value.
Support gas pressure is too low.	The supply pressure of support gas (Air or N ₂ O) has decreased.	Open the stopcock and check the supply pressure of support gas by the meter of the pressure regulator. If the required pressure is not satisfied, change the support gas cylinder, check/adjust the air compressor operation or perform other appropriate treatment.
Switch the Flame Type to AIR- C_2H_2 before the remaining gas combustion.	When the support gas is N ₂ O, executing the residual gas combustion may cause a flashback.	Set [Flame Type] to $[AIR-C_2H_2]$ in [Atomizer/Gas Flow Rate Setup] page and switch [BURNER SELECT] (burner selection key switch) of the AA main unit to $[AIR-C_2H_2]$ side. Then execute the residual gas combustion.

Error Message	Cause/Details	Remedial Measure
Т		
Termination has not been accepted from ASC.	Communication protocol with ASC was not completed. (:T)	Verify the ASC power supply and the cable connection between ASC and AA main unit, then connect the ASC in [Instrument]-[Option Connect] menu.
Termination has not been accepted from GFA.	Communication protocol with GFA was not completed. (:T)	Verify the GFA power supply and the cable connection between GFA and AA main unit, then connect the GFA in [Instrument]-[Option Connect] menu.
The fan of the AA main unit has been stopped.	There is an impediment in the fan.The instrument has a fault.	 Check if there is any impediment in the fan and, if there is one, remove it.
		 If the fan has stopped, or if an error has occurred even though the fan is rotating, stop using the instrument and contact your Shimadzu representative.
The following lamps exist in the Lamp Position Setup Dialog Box, but do not exist in the Lamp History Dialog Box. Please update the Lamp History Dialog Box or delete lamps from the Lamp Position Setup Dialog Box. Socket #x,x,x,	The data mismatching between the lamp history and the lamp position setup was detected at the time of starting up the AA software.	Click on [Yes] and delete the data from the lamp position setup. Register the necessary lamps in [Instrument]-[Lamp History]. Next, make a setting of the lamp mounted to each socket in [Instrument]-[Lamp Position Setup].
The GFA instrument type is not selected correctly. Please select proper GFA instrument type and restart this software.	The GFA instrument type selected in the AA software is wrong.	Set the correct [GFA] in [Instrument]- [Configuration] menu then start up the AA software again.
The instrument fail to adjust the gas flow rate to the optimal position.	The gas flow rate setup command has failed.	When an error occurs to the fuel gas flow rate setup in [Atomizer/Gas Flow Rate Setup] page, stop using the instrument and contact your Shimadzu representative.
The instrument fail to move the burner to the optimal position.	The burner movement command has failed.	Remove the obstacles that disturb the burner movement then switch on the instrument power again.
Vibration was detected by the instrument.		Implement the "Emergency Action".
The selected area cannot be cleared as the information is stored in the Lamp Placement Dialog Box.		In [Instrument]-[Lamp Position Setup] menu, select [NONE] for [Element] field of the lamp ID you wish to clear, and click on [OK]. Then clear the target lamp in [Lamp History] dialog box.

Error Message	Cause/Details	Remedial Measure
The selected area cannot be deleted as the information is stored in the Lamp Placement Dialog Box.		In [Instrument]-[Lamp Position Setup] menu, select [NONE] for [Element] field of the lamp ID you wish to delete, and click on [OK]. Then delete the target lamp in [Lamp History] dialog box.
The selected area contains the D2 lamp. D2 lamp row cannot be deleted.	Deleting the first row for D2 lamp in [Lamp History] table was tried.	The first row for D2 lamp is fixed and cannot be deleted.
This file is not to be loaded. Software doesn't support this file.	The extension of file is wrong.	Load a file that has the extension indicated in [Files of type] in [Open] dialog box.
This operation cannot be executed during action or measurement.	The operation tried cannot be executed when the instrument is BUSY status or while the measurement is executed.	Close this message and wait until [READY] is displayed at the right lower of the status bar. Then perform the target operation again.
This operation cannot be executed while the hollow cathode lamp is lit ON. Please retry it after you turn off the lamp.		Turn off the lamp in [Optics Parameters] page of the current measurement element. Use [Lamp ON] check box in the case of measurement lamp and [Warmup Lamp] button in the case of warm-up lamp. Then, execute the aimed operation.
Time out occurred during the GFA transformer is cooling down.	The GFA is in waiting status for cooling transformer and cannot recover.	Switch on the GFA and PC again. If the error occurs again, record the status in which this error occurs. Then contact your Shimadzu representative.
Timer cannot be created. After closing other applications, try it again.	The number of timers currently used exceeds the limit of the Windows system.	Start up the PC again. Also, do not start other applications.
Too many schedules. Failed to add all schedules.	When executing [File]-[Additional Load], the excess schedules cannot be read out on the MRT work sheet if the number of Element Schedules on the MRT work sheet exceeds 20.	When the Element schedule you wish to add is located in the latter part of the original file of adding, start up another AA software and load the original file for adding and move the order of target Element schedule. Or take out only the element schedule and save it as different file name to create an original file for adding.
U		
Unexpected file format.	 The extension of file is wrong. The file name contains a period except the period just before the extension (for example, a file named "Cu.Ag.aa" cannot be read out). 	Load a file that has the extension indicated in [Files of type] in [Open] dialog box.

Error Message	Cause/Details	Remedial Measure
Unknown Status Code: / (Error Code)	An error that is not defined in the AA software inside has occurred.	Switch on the PC power and the instrument power again. If the error occurs again, record the number indicated in the error message and the status in which the errors occurred. Then contact your Shimadzu representative.
Usage time has exceed lamp life time.	When registering the lamp in [Lamp History] dialog box, the input values has a relation "(Life Time) < (Used Time)".	It is possible to input the values as "(Life Time) < (Used Time)". If the values are not wrong, continue the input.
V		
Valid Injection position is not set. Use <move upward="">/ <move downward=""> button to move the nozzle to proper position.</move></move>	The injection position is set higher than the destination nozzle position in [ASC Furnace Nozzle Position Adjustment] dialog box, and as the result, [Move Near to Tube] cannot be executed.	Set the injection position properly then [Move near to Tube] can be used.
W		
Wizard cannot be executed during measurement or when the instrument is BUSY.		Close this message and wait until [READY] is displayed at the right lower of the status bar. Then perform the target operation again.
X,Y,Z	1	1

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Chapter 10 Technical Data

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The specifications of the AA-7000 series are as follows. The AA-7000 series does not conform to IEC60601.

10.1.1 Basic Specifications

Measurement System	Measurement Wavelength Range	185.0 to 900.0 nm		
	Monochrometer	Aberration-corrected Czerny-Turner mount		
	Bandwidth	0.2, 0.7,1.3, 2.0 L nm (4-step automatic switching)		
	Detector	Photo multiplier		
	Photometric Method	Flame: Optical double beam Furnace: High throughput single beam		
	Background Correction	High-speed self reversal method (BGC-SR) (185 to 900 mm) Deuterium lamp method (BGC-D2) (185 to 430 mm)		
	Number of Lamp Sockets	6 lamp sockets, 2 lamps simultaneously lit (1 for warming up)		
	Lamp Mode	EMISSION, NON-BGC, BGC-SR, BGC-D2		

Data Processing	Software Environment	Microsoft Windows 10 Pro (32 bit/64 bit) / Windows 7 Professional (32 bit/64 bit) / Windows Vista Business (32 bit) / Windows XP Professional (32 bit)		
	Parameter Setting	Wizard Method		
	Measurement Mode	Flame continuous method, Flame micro sampling method, Furnace method		
	Concentration Conversion Mode	 Calibration Curve (selectable among 1st, 2nd, 3rd order function) Standard addition method/Simple standard 		
		addition method (1st order function)		
	Repeat Measurement	 Max. 20 repetitions Indication of Average, Standard deviation (SD) and Relative Standard Deviation (RSD) 		
		 Exclusion of deviant value based on SD value and RSD value 		
	Baseline Correction	Automatic correction of base line drift using offset correction with peak height and peak area mode		
	Peak data processing range	Peak data processing range can be changed with peak height and peak area mode.		
	Sensitivity Correction	Automatic calibration curve correction function by sensitivity observation		
	Analog output	2 channels (atomic absorption / energy signal, background signal) Output range: 5.0, 2.5, 1.25, 0.625 Abs. / V (4-step switching for each) In the EMISSION mode, fixed at 1 V F.S.		
	Table Data Processing Functions	Actual concentration calculation using Weight factor, Dilution factor, Volume factor and Correction factor.		
	Loading Parameters	Template function		
	Procedure/Result Display	MRT Work Sheet (MRT: Measurement Result Table)		
	Result Print	Summary Report		
	QA/QC	Correlation factor, %RSD, ICV/ICB, CCV/CCB, PB, LCS, SPK, PDS, DUP Pause or Mark&Continue is selected by these QA/QC checks.		
	Re-measurement	Retry/Not retry can be selected. Automatic dilution re-measurement of unknown sample by the autosampler (Flame micro sampling method, Furnace method)		
	Electronic records and electric signatures	 User administration by login ID/password Limitation of assigned rights with user level Recording of log Audit trail Electronic signatures 		

Power Requirements	AC100, 120, 220, 230 V selectable, 230 VA, 50/60 Hz Additional power for PC is necessary.
Short-circuit rating	50 A
Dimensions, Weight	 AA-7000F: W700 × D588 × H714 mm, 73 kg AA-7000F/AAC: W700 × D588 × H714 mm, 76 kg AA-7000G: W700 × D580 × H538 mm, 66 kg (not including chimney and protrusions)
Ambient Temperature and Humidity Range	10 °C to 35 °C, 20% to 80% (less than 70% if temperature is over 30 °C)

10.1.2 Flame Specifications

* Throughout this manual, polytetrafluorethylene is abbreviated PTFE.

Burner Unit	Туре	Air-cooled pre-mix type
	Burner Head	10 cm slot made of Titanium (5 cm slot made of Titanium for $\rm N_2O-C_2H_2$ flame is optional item.)
	Nebulizer	 Pt-Ir capillary PTFE orifice Impact bead made of Ceramic (Fluoric acid is usable.)
	Chamber	Engineering Plastic
	Positioning	 AA-7000F Manual Adjustment for back-for and up-down positions AA-7000F/AAC Automatic switching between burner and furnace by motor drive Automatic search for optimum burner height
	Angle adjustment	 0 to 90 degrees When the automatic atomizer changer (optional) and GFA-7000A are both provided, angle adjustment is not possible.
Gas Controller	Flame Type	Air- C_2H_2 , N_2O - C_2H_2 (not support Hydrogen flame)
	Flow Rate Control	 Automatic flow rate setting for fuel gas (0.1 L/min increments) Manual flow rate setting for support gas (when equipped with optional instruments) Automatic searching for optimum gas flow rate
	Safety Features	 Automatic gas leakage inspection Air/N₂O auto switching with C₂H₂ flow rate monitor (optical sensor) Flame monitor Prevents usage of incorrect burner head Gas pressure monitor Drain water level monitor Automatic extinction by detection of momentary power failure Automatic extinguishing by the vibration sensor Detection of stoppage of the in-unit fan

10.1.2.1 Chemicals That can be Used

WARNING

- Check that the parts of the atomizer are resistant to the chemicals to be used. Using such chemicals will cause accidents.
- Replace each part at the specified intervals.

The chemical resistance and usage conditions of the materials used in the atomizer of the AA-7000F and AA-7000F/AAC are as follows.

Before making a measurement, check that there is resistance to the chemicals to be used by referring to the table below.

	Drain	Drain	Tube		O-rings		Chamber	
Chemical Name	Tank (Polyethylene)	Standard	Options	Standard	Options (Silicon rubber)	Options (Kalrez)	Nebulizer	Conditions of Use
Water *1	Ô	Ô	Ô	O	Ô	Ô	O	
Ethanol	0	0	0	0	0	0	0	
Hydrochloric acid	0	0	0	0	0	0	0	
Nitric acid	0	0	0	0	0	0	0	
Sulfuric acid	0	0	0	0	0	0	0	
Hydrofluoric acid	0	0	0	0	×	0	0	
Perchloric acid	0	0	0	0	×	0	0	
Methanol	0	0	0	\bigtriangleup	0	0	0	Change the O-rings to the
Sodium hydroxide	0	0	0	×	×	0	0	optional products.
Gasoline	Δ	\triangle	0	0	×	0	0	Change the drain tank once
Petroleum	Δ	Δ	0	0	Δ	0	0	every month. Change the standard drain tube once every month, or replace it with the optional product.
Acetic acid	Δ	\triangle	0	×	0	0	0	Change the O-rings to the
МІВК	\triangle	\triangle	0	×	\triangle	0	0	optional products. Change the drain tank once
Butyl acetate	\bigtriangleup	\triangle	0	×	\triangle	0	0	every month. Change the
Acetone	\bigtriangleup	Δ	0	×	Δ	0	0	standard drain tube once every month, or replace it with the optional product.

The chemical resistance stated is that when the chemical is at room temperature.

*1: Acid concentration of 0.5% or lower

 \odot : Can be used, replacement interval of 3 years

 \bigcirc : Can be used, replacement interval of 1 year

 \triangle : Can be used, replacement interval of 1 month

 $\times\colon \$ Use prohibited

CAUTION

Chemicals that are not covered in the table above cannot be used.

NOTE

When a mixture of two or more types of chemical is used, abide by the stricter conditions.

10.1.3 Furnace Specifications

Heating Temperature Range	Ambient~3000 °C
Heating Control Method	Drying: Current control method (with automatic temperature calibration function) Ashing/Atomizing: Optical temperature control method
Heating Parameter settings	 Max. 20 stages Heating mode: RAMP/STEP Inner gas type: 2-line switching High sensitivity mode setting Furnace concentration boost cycle: Max 20 times Optimum furnace program search function Inner gas flow rate: 0~1.50 L/min
Safety Features	 Cooling water flow rate monitor Gas pressure monitor Over current protection (double check by breaker and optical sensor) Furnace block cooling check Furnace block over heat protection
Positioning	 AA-7000G Manual Adjustment for back-for and up-down positions AA-7000F/AAC Automatic switching between the burner and furnace by motor drive

10.1.4 Performance Specifications

Item	AA-7000 Series				
Specifications common for flame and furnace					
Wavelength accuracy	≤± 0.30 nm				
NON-BGC noise level	≤ 0.0100 Abs				
BGC-D2 noise level	≤ 0.0150 Abs				
Baseline drift	≤ 0.0050 Abs / 30 min				
Specifications only for flame analysis					
Absorbance	≥ 0.2300 Abs				
Repeatability	\leq 2.00 % (No. of repetition 5 times, confidence coefficient 95%)				
Detection limit	≤ 0.00600 ppm				
Stability	≤ 6.0%				
Specifications only for furnace analysis					
Absorbance	≥ 0.15 Abs				
Repeatability	\leq 2.5 % (No. of repetition 5 times, confidence coefficient 95%)				
Detection limit	≤ 0.03000 ppb				

NOTE

The performance is premised on the following conditions.

- At least 30 minutes has elapsed since the power to the instrument that includes the option was turned ON.
- Each of the lamps used have been lit for at least 10 minutes in preparation.
- The cumulative operation time of each of the lamps is within the guaranteed service life.

10.1.5 Software Operation Minimum Requirements

To operate the AA-7000 series for measurement, WizAArd, the software for controlling the AA-7000 series and a PC system are required.

The following must be purchased separately.

 Comprising the AA-7000 series control software package (software package, WizAArd), CD-ROM (WizAArd), PC-AA connecting cable and so on. No PC system is included. Separately prepare a PC and monitor that meet the following specifications.

	Specifications
CPU	[In the case of 64 bit OS] Intel Core 2 Duo E7500 (2.93 GHz) or faster [In the case of 32 bit OS] Intel Celeron 420 (1.50 GHz) or faster
RAM	[In the case of Windows 10, Windows 7 and Windows Vista] 2 GB or larger [In the case of Windows XP] 1 GB or larger
Screen resolution	XGA (1024 × 768 dots) or higher
Storage device	1 CD-ROM drive (for installing the program)
	(With at least 60MB free disk space for AA software)
I/O port	1 serial port for AA
Peripherals	Monitor
	Keyboard
	Mouse or other pointing device
	Printer

Table 10.	1 PC	Specifications
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10.1.5.1 Operating System

To operate the AA-7000 series software on the PC, Windows 10 Pro (32 bit/64 bit), Windows 7 Professional (32 bit/64 bit), Windows Vista Business (32 bit), or Windows XP Professional (32 bit) is required.

NOTE

The AA-7000 series software does not support "switching users".

10.2

Measurement Conditions Table for Flame Atomic Absorption Analysis

NOTE

- Most of these measurement conditions are included in the software as standard analysis conditions, but the optimal measurement conditions vary depending upon the properties of the sample. (A single element each is assumed as a standard condition. Therefore, if there are multiple conditions, one of them is included in the software.)
- 2. <HVG-1> represents the Hydride Vapor Generator (optional).

Element	Wave length (nm)	L233 (mA)	L2433 (mA)	Slit (nm)	Flame Type	Flow (L/min)	Burner. H (mm)
Ag	328.1	10	10/400	0.7	Air-C ₂ H ₂	2.2	7
Al	309.3	10	10/600	0.7	$N_2O-C_2H_2$	6.6	11
As (H)	193.7	12	12/500	0.7	Air-C ₂ H ₂	2.0	<hvg-1></hvg-1>
Au	242.8	10	10/400	0.7	Air-C ₂ H ₂	1.8	7
В	249.7	16	10/500	0.2	N ₂ O-C ₂ H ₂	7.5	11
Ва	553.5	16	12/600	0.2	N ₂ O-C ₂ H ₂	7.0	11
Ве	234.9	16	10/600	0.7	N ₂ O-C ₂ H ₂	6.6	11
Bi (H)	223.1	10	10/300	0.7	Air-C ₂ H ₂	2.0	<hvg-1></hvg-1>
Bi	223.1	10	10/300	0.7	Air-C ₂ H ₂	2.2	7
Ca (1)	422.7	10	10/600	0.7	Air-C ₂ H ₂	2.0	7
Ca (2)	422.7	10	10/600	0.7	N ₂ O-C ₂ H ₂	6.5	11
Cd	228.8	8	8/100	0.7	Air-C ₂ H ₂	1.8	7
Со	240.7	12	12/400	0.2	Air-C ₂ H ₂	1.6	7
Cr	357.9	10	10/600	0.7	Air-C ₂ H ₂	2.8	9
Cs	852.1	16		0.7	Air-C ₂ H ₂	2.0	7
Cu	324.8	8	10/500	0.7	Air-C ₂ H ₂	1.8	7
Dy	421.2	14	15/600	0.2	N ₂ O-C ₂ H ₂	7.0	11
Er	400.8	14	15/500	0.7	N ₂ O-C ₂ H ₂	7.0	11
Eu	459.4	14	10/600	0.7	N ₂ O-C ₂ H ₂	7.0	11
Fe	248.3	12	12/400	0.2	Air-C ₂ H ₂	2.2	9
Ga	287.4	4	4/400	0.2	Air-C ₂ H ₂	2.2	7
Gd	368.4	12		0.2	N ₂ O-C ₂ H ₂	7.0	11
Ge	265.2	18	20/500	0.2	N ₂ O-C ₂ H ₂	7.5	11
Hf	307.3	24	20/600	0.2	N ₂ O-C ₂ H ₂	7.0	11
Hg	253.7	4		0.7	By the cold vapor mercury technique		
Но	410.4	14	10/600	0.2	N ₂ O-C ₂ H ₂	7.0	11
In	303.9	6		0.7	Air-C ₂ H ₂	2.0	7
Ir	208.8	20		0.2	Air-C ₂ H ₂	2.2	7
К	766.5	10	8/600	0.7	Air-C ₂ H ₂	2.0	7
La	550.1	18	18/600	0.7	$N_2O-C_2H_2$	7.5	11
Li	670.8	8	8/500	0.7	Air-C ₂ H ₂	1.8	7
Lu	360.0	14		0.7	$N_2O-C_2H_2$	7.0	11

Element	Wave length (nm)	L233 (mA)	L2433 (mA)	Slit (nm)	Flame Type	Flow (L/min)	Burner. H (mm)
Mg	285.2	8	8/500	0.7	Air-C ₂ H ₂	1.8	7
Mn	279.5	10	10/600	0.2	Air-C ₂ H ₂	2.0	7
Мо	313.3	10	10/500	0.7	N ₂ O-C ₂ H ₂	7.2	11
Na	589.0	12	8/600	0.2	Air-C ₂ H ₂	1.8	7
Nb	334.9	24		0.2	$N_2O-C_2H_2$	7.0	11
Ni	232.0	12	10/400	0.2	Air-C ₂ H ₂	1.6	7
Os	290.9	14		0.2	N ₂ O-C ₂ H ₂	7.0	11
Pb (1)	217.0	12	8/300	0.7	Air-C ₂ H ₂	2.0	7
Pb (2)	283.3	10	8/300	0.7	Air-C ₂ H ₂	2.0	7
Pd	247.6	10	10/300	0.7	Air-C ₂ H ₂	1.8	7
Pr	495.1	14		0.7	N ₂ O-C ₂ H ₂	7.0	11
Pt	265.9	14	10/300	0.7	Air-C ₂ H ₂	1.8	7
Rb	780.0	14		0.2	Air-C ₂ H ₂	1.8	7
Re	346.0	20		0.2	$N_2O-C_2H_2$	7.0	11
Rh	343.5	12		0.7	Air-C ₂ H ₂	1.6	7
Ru	349.9	20	20/600	0.2	Air-C ₂ H ₂	2.4	7
Sb (H)	217.6	13	15/500	0.7	Air-C ₂ H ₂	2.0	<hvg-1></hvg-1>
Sb	217.6	13	15/500	0.7	Air-C ₂ H ₂	2.0	7
Sc	391.2	10		0.2	N ₂ O-C ₂ H ₂	7.0	11
Se (H)	196.0	23	15/300	0.7	Air-C ₂ H ₂	2.0	<hvg-1></hvg-1>
Se	196.0	23	15/300	0.7	Ar-H2	[3.7]	15
Si	251.6	15	10/500	0.7	$N_2O-C_2H_2$	7.5	11
Sm	429.7	14	15/600	0.2	N ₂ O-C ₂ H ₂	7.0	11
Sn (H)	286.3	10	20/500	0.7	Air-C ₂ H ₂	2.0	<hvg-1></hvg-1>
Sn (1)	224.6	10	20/500	0.7	Air-C ₂ H ₂	3.0	9
Sn (2)	286.3	10	20/500	0.7	Air-C ₂ H ₂	3.0	9
Sn (3)	224.6	10	20/500	0.7	N ₂ O-C ₂ H ₂	6.8	11
Sn (4)	286.3	10	20/500	0.7	N ₂ O-C ₂ H ₂	6.8	11
Sr	460.7	8	6/500	0.7	Air-C ₂ H ₂	1.8	7
Та	271.5	18		0.2	$N_2O-C_2H_2$	7.0	11
Tb	432.6	10		0.2	$N_2O-C_2H_2$	7.0	11
Te (H)	214.3	14	15/400	0.2	Air-C ₂ H ₂	2.0	<hvg-1></hvg-1>
Те	214.3	14	15/400	0.2	Air-C ₂ H ₂	1.8	7
Ti	364.3	12	10/600	0.7	N ₂ O-C ₂ H ₂	6.8	11
TI	276.8	6		0.7	Air-C ₂ H ₂	1.8	7
V	318.4	10	10/600	0.7	N ₂ O-C ₂ H ₂	7.0	11
W	255.1	24		0.2	N ₂ O-C ₂ H ₂	7.7	11
Y	410.2	14	10/600	0.7	N ₂ O-C ₂ H ₂	7.5	11
Yb	398.8	10	5/200	0.7	N ₂ O-C ₂ H ₂	7.5	11
Zn	213.9	8	10/300	0.7	Air-C ₂ H ₂	2.0	7
Zr	360.1	18		0.2	$N_2O-C_2H_2$	7.5	11

10.3 Analysis Line Wavelength Table for Flame Emission Analysis

Element	Wave length (nm)
Са	422.7
Cs	852.1
Li	670.8
К	766.5, 769.9
Na	589.0, 589.5
Rb	780.0
Sr	460.7

The optional parts available for this instrument are listed below.

10.4.1 For Flame Analysis

Part Name	Part No.	Remarks
High temperature burner head	S206-77530-91	Titanium, $N_2O-C_2H_2$ 5 cm slot for flame
Flow meter kit	S206-77617-41	Float type flow meter for assist gas
Sample stage	S206-77655-91	External dimensions: 250 wide \times 130 deep \times 170 high (mm) Container loading compartment: 220 wide \times 95 deep (mm) Container position: Selectable from 5 stages
Air compressor	S208-91753-92	AC 100 V for both 50 Hz/60 Hz With mist separator
Silent-type air compressor	S208-91750-36	AC 220 V/230 V for both 50 Hz/60 Hz With mist separator
Mist separator kit	S206-52458-41	Required at the time of using a different air compressor described above.
Precision gas pressure regulator YR- 71	S040-72020-01	For acetylene gas
Precision gas pressure regulator MAF-85S	S040-72019-11	For nitrous oxide gas
Microsampling kit	S206-77540-91	This is a kit that makes it possible to use the flame micro sampling method. An ASC-7000 or ASK-7000 unit (or the ASC stand kit) is required separately.
O-ring set (Silicon rubber)	S206-77620-93	For details, see 10.1.2.1 "Chemicals That can be Used".
O-ring set (Kalrez)	S206-77620-92	Set of O-rings for use with organic solvents For details, see 10.1.2.1 "Chemicals That can be Used".
Polyethylene container	S038-00525-01	Waste liquid container, 10 L For details, see 10.1.2.1 "Chemicals That can be Used"
Waste liquid hook	S206-77565-91	Clamps the drain tube to the waste liquid container.
Gas leak detection fluid	S670-11514	Used for gas leak test

10.4.2 For Furnace Analysis

Part Name	Part No.	Remarks
Graphite Furnace Atomizer GFA-7000A	S206-77777-44/45	
Graphite Furnace Atomizer Camera GFA-TV	S206-52950-91	To display images of the inside of the graphite furnace on a PC display GFA-TV Viewer software (CD-ROM) attached
High-density graphite tube	S206-50587-12	1 set
	S206-50587-85	10 set
Pyrolytic coating graphite tube	S206-50588-11	1 set
	S206-50588-84	10 set
Platform tube	S206-50887-02	
Cooling water circulating unit CA-1115A-1	S044-01813-01	Setting temperature range: –20 to +30°C AC 100V for both 50/60Hz
Cooler connection kit	S206-84373-91	Required when CA-1114A-1 is used
Tube ASSY for cooling water	S206-51028-91	Used when the cooling water is drawn from tap water
Regulator ASSY	S206-86147-91	Pressure reducing valve for cooling water, used when the cooling water is drawn from tap water
Precision gas pressure regulator JET-SCB-STB*	S040-72027-01	For argon gas

10.4.3 Autosampler

Part Name	Part No.	Remarks
Autosampler ASC-7000	S206-77600-42/48	
ASC stand kit	S206-77650-41	Necessary for installation of ASC-7000 when executing only flame analysis.
Extended unit for furnace analysis ASK-7000	S206-77550-41	For both flame and furnace analyses
Nozzle ASSY, HVG	S206-67563	Required when using ASC-6100F with HVG-1

10.4.4 Dual Atomizer System

Part Name	Part No.	Remarks
Automatic atomizer changer AAC-7000	S206-77701-41	Required when performing furnace analysis by adding a GFA-7000A to an AA-7000F When performing flame analysis on an AA-7000F, enables automation of burner position setting This part is built into the AA-7000F/AAC

10.4.5 Hollow Cathode Lamps (Single Element Lamps)

HAMAMATSU PHOTONICS Co.LTD L233 Series

NOTE

This lamp cannot be used for the high-speed self reversal method.

Element Symbol	Element Name	Part No.	Product Name
Ag	Silver	S200-38422-02	L233-47NB
AI	Aluminum	S200-38422-01	L233-13NB
As	Arsenic	S200-38422-42	L233-33NQ
Au	Gold	S200-38422-25	L233-79NQ
В	Boron	S200-38422-39	L233-5NQ
Ва	Barium	S200-38422-03	L233-56NB
Ве	Beryllium	S200-38422-04	L233-4NQ
Bi	Bismuth	S200-38422-43	L233-83NQ
Са	Calcium	S200-38422-05	L233-20NU
Cd	Cadmium	S200-38422-06	L233-48NQ
Со	Cobalt	S200-38422-09	L233-27NU
Cr	Chromium	S200-38422-07	L233-24NB
Cs	Cesium	S200-38422-27	L233-55NB
Cu	Copper	S200-38422-08	L233-29NB
Dy	Dysprosium	S200-38422-60	L233-66NB
Er	Erbium	S200-38422-61	L233-68NB
Eu	Europium	S200-38422-62	L233-63NB
Fe	Iron	S200-38422-10	L233-26NU
Ga	Gallium	S200-38422-40	L233-31NU
Gd	Gadolinium	S200-38422-63	L233-64NB
Ge	Germanium	S200-38422-11	L233-32NU
Hf	Hafnium	S200-38422-64	L233-72NU
Hg	Mercury	S200-38422-28	L233-80NU
Но	Holmium	S200-38422-65	L233-67NB
In	Indium	S200-38422-48	L233-49NB
lr	Iridium	S200-38422-66	L233-77NQ
К	Potassium	S200-38422-22	L233-19NB
La	Lanthanum	S200-38422-29	L233-57NB
Li	Lithium	S200-38422-30	L233-3NB
Lu	Lutetium	S200-38422-67	L233-71NB
a i	Iridium Potassium Lanthanum Lithium	S200-38422-66 S200-38422-22 S200-38422-29 S200-38422-30	L233-77NQ L233-19NB L233-57NB L233-3NB

Element Symbol	Element Name	Part No.	Product Name
Mg	Magnesium	S200-38422-12	L233-12NU
Mn	Manganese	S200-38422-13	L233-25NU
Мо	Molybdenum	S200-38422-31	L233-42NB
Na	Sodium	S200-38422-14	L233-11NB
Nb	Niobium	S200-38422-32	L233-41NB
Nd	Neodymium	S200-38422-68	L233-60NB
Ni	Nickel	S200-38422-15	L233-28NQ
Os	Osmium	S200-38422-69	L233-76NU
Pb	Lead	S200-38422-21	L233-82NQ
Pd	Palladium	S200-38422-41	L233-46NQ
Pr	Praseodymium	S200-38422-70	L233-59NB
Pt	Platinum	S200-38422-20	L233-78NU
Rb	Rubidium	S200-38422-33	L233-37NB
Re	Rhenium	S200-38422-44	L233-75NB
Rh	Rhodium	S200-38422-49	L233-45NB
Ru	Ruthenium	S200-38422-45	L233-44NB
Sb	Antimon	S200-38422-24	L233-51NQ
Sc	Scandium	S200-38422-71	L233-21NB
Se	Selenium	S200-38422-46	L233-34NQ
Si	Silicon	S200-38422-16	L233-14NU
Sm	Samarium	S200-38422-72	L233-62NB
Sn	Tin	S200-38422-18	L233-50NQ
Sr	Strontium	S200-38422-34	L233-38NB
Та	Tantalum	S200-38422-35	L233-73NU
Tb	Terbium	S200-38422-73	L233-65NB
Те	Tellurium	S200-38422-47	L233-52NQ
Ti	Titanium	S200-38422-17	L233-22NB
TI	Thallium	S200-38422-74	L233-81NU
Tm	Thulium	S200-38422-75	L233-69NB
V	Vanadium	S200-38422-19	L233-23NB
W	Tungsten	S200-38422-36	L233-74NU
Y	Yttrium	S200-38422-76	L233-39NB
Yb	Ytterbium	S200-38422-77	L233-70NB
Zn	Zinc	S200-38422-23	L233-30NQ
Zr	Zirconium	S200-38422-37	L233-40NB

Products from Heraeus

Element Symbol	Element Name	Part No.	Product Name
Na	Sodium (for 330.3 nm)	S208-94048-01	3QNYNA

10.4.6 Hollow Cathode Lamps (Single Element Lamps, Usable for SR Method)

• HAMAMATSU PHOTONICS Co.LTD L2433 Series

Element Symbol	Element Name	Part No.	Product Name
Ag	Silver	S200-38456-01	L2433-47NB
AI	Aluminum	S200-38456-02	L2433-13NB
As	Arsenic	S200-38456-03	L2433-33NQ
Au	Gold	S200-38456-04	L2433-79NQ
В	Boron	S200-38456-05	L2433-5NQ
Ва	Barium	S200-38456-06	L2433-56NB
Be	Beryllium	S200-38456-07	L2433-4NQ
Ві	Bismuth	S200-38456-08	L2433-83NQ
Са	Calcium	S200-38456-09	L2433-20NU
Cd	Cadmium	S200-38456-10	L2433-48NQ
Со	Cobalt	S200-38456-11	L2433-27NU
Cr	Chromium	S200-38456-12	L2433-24NB
Cu	Copper	S200-38456-14	L2433-29NB
Dy	Dysprosium	S200-38456-15	L2433-66NB
Er	Erbium	S200-38456-16	L2433-68NB
Eu	Europium	S200-38456-17	L2433-63NB
Fe	Iron	S200-38456-18	L2433-26NQ
Ga	Gallium	S200-38456-19	L2433-31NU
Ge	Germanium	S200-38456-21	L2433-32NU
Hf	Hafnium	S200-38456-22	L2433-72NU
Но	Holmium	S200-38456-24	L2433-67NB
К	Potassium	S200-38456-26	L2433-19NB
La	Lanthanum	S200-38456-27	L2433-57NB
Li	Lithium	S200-38456-28	L2433-3NB
Mg	Magnesium	S200-38456-30	L2433-12NU
Mn	Manganese	S200-38456-31	L2433-25NU
Мо	Molybdenum	S200-38456-32	L2433-42NB
Na	Sodium	S200-38456-33	L2433-11NB
Ni	Nickel	S200-38456-35	L2433-28NQ
Pb	Lead	S200-38456-37	L2433-82NQ
Pd	Palladium	S200-38456-38	L2433-46NQ
Pt	Platinum	S200-38456-40	L2433-78NU

Element Symbol	Element Name	Part No.	Product Name
Ru	Ruthenium	S200-38456-43	L2433-44NB
Sb	Antimony	S200-38456-44	L2433-51NQ
Se	Selenium	S200-38456-46	L2433-34NQ
Si	Silicon	S200-38456-47	L2433-14NQ
Sm	Samarium	S200-38456-48	L2433-62NB
Sn	Tin	S200-38456-49	L2433-50NQ
Sr	Strontium	S200-38456-50	L2433-38NB
Те	Tellurium	S200-38456-53	L2433-52NQ
Ті	Titanium	S200-38456-54	L2433-22NB
V	Vanadium	S200-38456-56	L2433-23NB
Y	Yttrium	S200-38456-58	L2433-39NB
Yb	Ytterbium	S200-38456-59	L2433-70NB
Zn	Zinc	S200-38456-60	L2433-30NQ

10.4.7 Hollow Cathode Lamp (Multi-Element Lamps)

• HAMAMATSU PHOTONICS Co. LTD L733 Series

NOTE

This lamp cannot be used for the high-speed self reversal method.

Element	Part No.	Product Name
Ca, Mg	S200-38422-51	L733-202NU
Fe, Ni	S200-38422-53	L733-204NQ
Na, K	S200-38422-50	L733-201NB
Si, Al	S200-38422-52	L733-203NU
Si, Ba	S200-38422-54	L733-205NU

10.4.8 Other Parts

Element	Part No.	Product Name
Mercury vaporizer unit MVU-1A	S206-58780-58	
Gas flow cell	S201-98687	For MVU
Cell holder for gas flow cell	S206-77703-91	For MVU
Hydride vaporizer unit HVG-1	S206-17143-××	
Analog output cable	S206-77707-41	Used when making analog outputs, e.g. to a pen recorder One required per channel

The instrument components are positioned as shown in Fig. 10.1. In addition to this, the installation site for PC and printer is necessary.

WARNING

• Take the weight of the entire measuring system into account when installing the product.

Install this product on a desk or stand that can easily bear the weight of the entire measuring system and is flat and stable. If these conditions are not met, accidents in which the product topples over or falls may occur.

CAUTION

• Make sure that it is not exposed to strong outdoor daylight.

The AA-7000 series are equipped with a safety system using an optical sensor. Exposure to strong outdoor daylight may interfere with normal operation of the flame extinction safety system for monitoring flame combustion (Flame Monitor). When using incandescent lamp or other heat light sources near the main unit, select the installation site where the luminous intensity by the light source only is below 400 Lx. If not available, don't allow the direct light from the light source to come into the burner compartment.

• If the temperature changes by 1 °C, the absorbance may change by 0.010 Abs maximum.

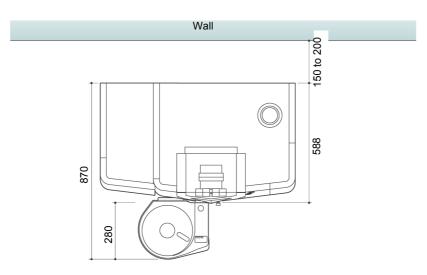
To carry out such long-hour measurement as may be affected by temperature changes, execute AUTO ZERO during the measurement if necessary.

In the case of manual flame measurement (not using ASC), you can execute AUTO ZERO at any time by clicking on [AUTO ZERO] button on the left lower of the main window. In the case of automatic flame measurement (using ASC), if set "AUTO ZERO" or "RINSE" in [Action] field on the MRT work sheet, AUTO ZERO can be executed during the nozzle rinsing. In the case of furnace measurement, setting is not necessary because the instrument automatically execute AUTO ZERO just before measurement.

- The installation site should be:
 - a. Sufficiently distant from devices that generate strong magnetic and electrical fields and high frequency
 - b. Sufficiently ventilated
 - c. Free from vibration
 - d. Free from excessive dust and moisture and should not be exposed to corrosive gases (POLLUTION DEGREE 2)
 - e. The luminous intensity of installation site for this instrument should be below 1300 Lx in the case of fluorescent lamp, below 800 Lx in the case of indirect outdoor daylight, and below 400 Lx in the case of heat light source like incandescent lamp.
 - f. Ambient temperature range: 10 to 35 °C
 Humidity: 20 to 80% (but less than 70% if temperature is over 30 °C)
 - g. Ground is available.
 - h. In an altitude up to 2000 m
 - i. In an indoor location
- The installation platform (table or counter top) must be capable of supporting the combined weights of the components listed below and a PC system. One whose specifications state that it is for use with atomic absorption spectrophotometers is recommended.

AA-7000 series	76 kg
GFA-7000A	47 kg
ASC-7000+ASK-7000	17 kg

- Since gas and water pipes cables are connected at the back of the AA main unit, there must be a space of about 15 to 20 cm from the back of the AA main unit to a wall.
- There must be more than 30 cm of space from the right side of the AA main unit so that the HCL cover on the right side of the AA-7000 series can be opened/closed.



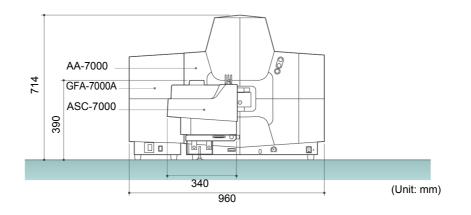


Fig. 10.1 Instrument Placement

10.6 Equipment Requirements

10.6.1 Power Requirements

The following table gives the power requirements for each of the components (OVERVOLTAGE CATEGORY II).

Table 10.2 Power Requirements

Voltage	Either one of 100, 120, 220, or 230 V
Allowable voltage range	Within ± 10% (Free from sudden voltage change)
Power capacity	230 VA
Source frequency	50/60 Hz
Connections	3P grounded outlet, cable length: approx. 2.4 m

NOTE

The power requirement for a PC system is not shown in the table.

10.6.2 Cooling Water Supply Requirement (GFA Only)

Use of the GFA-7000A requires a cooling water supply system to cool the graphite furnace. A method in which city water is used and a method in which the optional cooling water circulation unit is used are available.

The points to note when supplying the cooling water from the city water are given below.

- A water outlet with sink must be located within 7 m of the instrument.
- A flow rate of 0.6 to 1.5 L/min is required. Use a water pressure of 0.08 to 0.15 MPa. If this flow rate is not met, the safety device is actuated to stop operation.

If the water pressure exceeds 0.17 MPa, use the specially provided pressure reducing valve (regulator assembly, P/N 206-86147-91).

Maintain the water temperature in the range between 10 °C and 30 °C. If the water temperature is less
than 10 °C or more than 30 °C, use a cooling water circulating unit (optional) rather than piped water. If the
water temperature is greatly different from the room temperature, condensation may occur in the graphite
furnace. If the water temperature drifts out of this range, the safety device is actuated to stop operation.

CAUTION

To be prepared in case there is water leakage, the water outlet must be equipped with a sink.

If there is no sink, the equipment may be caused to fail by outflowing water. There is also the possibility of material damage at the installation site.

NOTE

When using a cooling water circulating unit (optional), be sure to use a cooler connecting kit.

10.6.3 Gas Requirements

(1) Gas Specifications

Be sure that gases are supplied for flame analysis according to the specifications given in Table 10.3.

Type of gas	Supply pressure (MPa)	Max. Consumption (L/min)	Purity
Air	0.35 ± 0.03	17.5	Free from oil, moisture and dust
Nitrous oxide	0.35 ± 0.03	12.5	Purity \geq 98%, moisture \leq 1%
Acetylene	0.09 ± 0.01	4.0 (Air-C ₂ H ₂ flame) 9.0 (N ₂ O-C ₂ H ₂ flame)	Purity ≥ 98%

Table 10.3 Gas Specifications

WARNING

Do NOT use oxygen gas.

Otherwise, fire or malfunction could result.

CAUTION

• Take care about fluctuations in gas pressure during analysis operations.

If the gas supply pressure changes during the analysis, resultant changes in flame combustion will adversely affect measurement reproducibility. The supply pressure shown in Table 10.3 must be maintained during flame combustion.

(2) Placement of gas cylinders

Select a location for the cylinders which satisfies the following conditions:

WARNING

• Place the cylinders outdoors.

Otherwise, fire or accidents could result.

- a. Not exposed to heat sources such as direct sunlight, furnaces, and heaters: Always keep the cylinders at a temperature below 40 °C
- b. Away from spark sources such as switchboards, ground wires, and high voltage power sources
- c. Away from flammable materials such as oil, gasoline, and organic solvents
- d. Sufficiently ventilated
- e. If outdoors, not exposed to wind and rain
- (3) Gas piping

If the cylinders are placed outdoors, piping must be performed separately within 5 m of the instrument. In this case, be sure follow the precautionary measures listed below.

- a. Use stainless steel pipes for the piping. Do not use of pipes that contain over 62% copper for acetylene piping.
- Ensure that the pipe diameters are not too small to supply gas at the pressures indicated in Table 10.3. A pipe diameter of at least 7 mm will be sufficient.

- c. Place a mist separator in the air piping system at the location indicated in Fig. 10.2. If a sufficiently dry air supply source is used, a drain separator is unnecessary.
- d. Install the stopcock and pressure regulator at a point within 5 meters of the instrument, in the analysis room.
- e. Provide a hose nipple with an outside diameter of 8.4 to 9.5 mm so that the provided gas supply hose (inside diameter 7.9 mm) can be connected to the end of the gas piping.

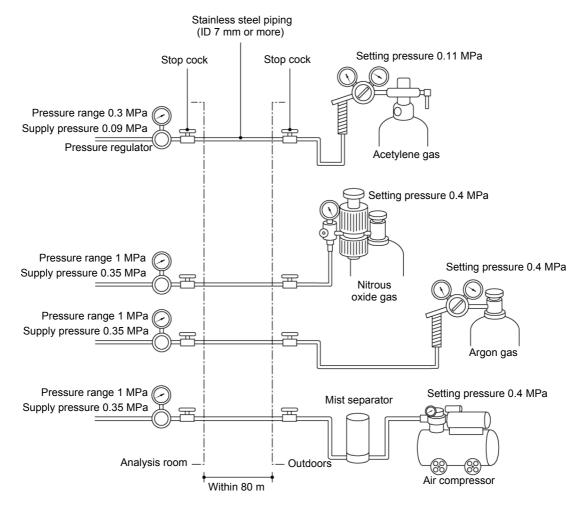


Fig. 10.2 Recommended Piping Setups for AA-7000 Series

CAUTION

- (1) Install the gas cylinders in the airy outdoors where they are not exposed to direct sunlight.
- (2) Take care that gas cylinders become no hotter than 40 °C, and do NOT allow any flame within 2 meters of the gas cylinders.
- (3) When using high pressure gas, make sure that there is sufficient ventilation. At the start of work inspection, check for gas leakage with soapy water or by other means. With regard to the use of flammable gases (e.g. acetylene) and gases that increase the susceptibility of substances to burn (e.g. nitrous oxide) in particular, smoking and the use of fire is prohibited within 5 meters of the equipment that is using these gases. Install a fire extinguisher to be prepared in the event of an accident.
- (4) Secure gas cylinders in a vertical position so that they cannot drop or fall over. Always keep liquefied gas cylinders (acetylene, nitrous oxide, etc.) in a vertical position and do NOT allow them to fall to a horizontal position.
- (5) Be sure to use oil-free pressure reducing valves. Also use ones that have no oil adhering to the inside of the pipes, etc., where high-pressure gas comes into contact.
- (6) Use approved pressure regulators and connectors. For details, contact your Shimadzu representative.
- (7) When installing the pressure regulator on a cylinder, be sure to wipe the dust off the outlet of the cylinder. Dust at the outlet of the cylinder could cause gas leakage.

10.6.4 Ventilation System

Place a ventilation duct with a hood above the atomizer.

WARNING

Be sure to provide a duct made of metal for the expulsion of combustible gas above the atomic absorption spectrophotometer.

If you use a duct made of plastic it will burn due to the heat of the flame.

A ventilator with an airflow rate of 600 to 1200 m³/h is appropriate for flame analysis, while one with an airflow rate of 10 to 180 m³/h is appropriate for furnace analysis. Too much suction force will affect measurement. Placement of a damper in the duct system is ideal for setting the optimum airflow.

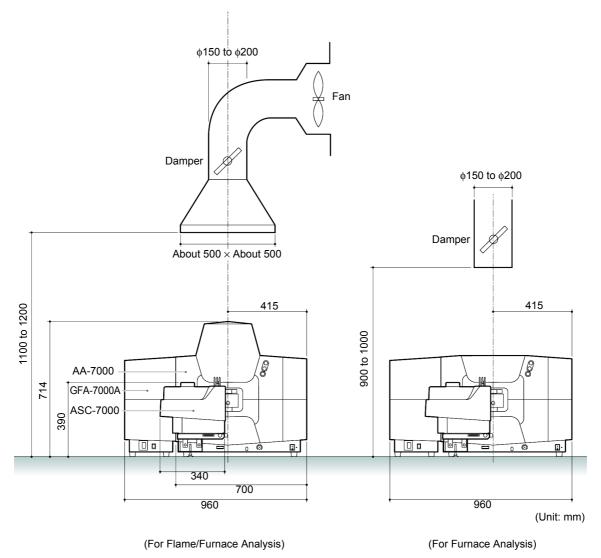


Fig. 10.3 Typical Ventilation System

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