

Agilent 55B AA Spectrometer

User's Guide



Notices

Manual Part Number

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Safety Notices

CAUTION

A **CAUTION** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

WARNING

A **WARNING** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a **WARNING** notice until the indicated conditions are fully understood and met.

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Safety Practices and Hazards

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Your Agilent 55B AA instrument and accessories have been carefully designed so when used properly, you have an accurate, fast, flexible and safe analytical system.

If the equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired.

Operation of an atomic absorption spectrometer can involve the use of compressed gases, flames, and hazardous materials including corrosive fluids and flammable liquids. Unskilled, improper, or careless use of this instrument can create explosion hazards, fire hazards, or other hazards which can cause death, serious injury to personnel, or severe damage to equipment and property.

Information on safety practices is provided with your instrument and operation manuals and referenced in your Agilent accessory manuals. Before using your instrument or accessories, thoroughly read these safety practices.

Observe relevant safety practices at all times.

Documentation

This manual covers the setup and operation of the basic Agilent AA system only. Operating instructions for the Sample Introduction Pump System (SIPS) and other AA accessories are given in the manuals accompanying those accessories.

NOTE

If you are controlling the instrument with an external computer and SpectrAA software, refer to the documentation and Help provided with the SpectrAA software.

Conventions

The following conventions have been used throughout this manual:

Single quotes (' ') indicate menu options and field names (e.g., select the 'Cookbook' option).

Bold text indicates the Keypad keys on your Agilent AA instrument (e.g., press the **Read** key).

ALL CAPITALS indicates text you must type in from the keyboard (attached to an external PC).

Other messages

Other messages appear in the manual where appropriate, and detail advice, information specific to the topic, or helpful tips.

NOTE

A 'Note' message is used to give advice or information.

Verifying safe state

The following general safety precautions must be observed during all phases of operation, maintenance and service of this instrument.

After maintenance or service procedures verify the instrument is returned to a safe state for the user. This includes running performance checks to verify the instrument safety systems are functioning correctly. Check the general condition

of the instrument during operation for wear or signs of corrosion that are likely to inhibit function or safety.

Failure to comply with these precautions or with specific warnings elsewhere in this manual violates safety standards of design, manufacture, and intended use of the instrument. Agilent Technologies assumes no liability for the customer's failure to comply with these requirements.

Electrical hazards

The instrument and accessories contain electrical circuits, devices and components operating at dangerous voltages. Contact with these items can cause death, serious injury, or painful electric shock.

Panels and covers that are retained by fasteners which require the use of a tool for removal may be opened only by Agilent trained and certified personnel. Consult the manuals or product labels supplied with your PC, monitor, printer/plotter, water-cooling system and/or vacuum pump to determine which parts are operator accessible.

Application of the wrong supply voltage, connection of the instrument to an incorrectly wired supply outlet, or lack of proper electrical grounding can create a fire hazard or a potentially serious shock hazard and could seriously damage the instrument. Any attached ancillary equipment could also be seriously damaged.

Always use a 3-wire outlet with ground connection which is adequately rated for the load. The installation must comply with local, State, and Federal safety regulations.

Do not connect the instrument to the mains power supply until you have made sure that the operating voltage is correctly set for the mains power supply in the specific outlet in your laboratory to which the equipment will be connected.

Heat, vapors and fumes

The heat, vapors and fumes generated can be hazardous, toxic, or otherwise injurious to personnel.

Safety Practices and Hazards

Heat, vapors and fumes must be extracted from the instrument by means of an exhaust system.

- The instrument must be vented into a self-contained arrangement of collector hood, ducting and exhaust fan.
- The system must be vented to the outside air, never within the building.
- Position the system outlet so that the exhaust cannot re-enter the building through any door, window, air conditioning inlet, or other ventilator.
- Construct the system in accordance with the appropriate codes and regulations for ventilation.

The exhaust system must be capable of an exhaust ventilation rate of at least 6 cubic meters per minute (200 scfm).

- Locate the exhaust fan at least 3 meters (10 feet) away from the flame and as close to the outlet as possible.
- The motor must be mounted away from the hot gases, and plastic parts must not be used as they will melt.
- Fit a back-draft damper to the outlet end of the system.
- Equip the exhaust fan power supply with a pilot light situated immediately adjacent to the instrument to indicate whether the exhaust fan is on or off.
- ALWAYS switch the exhaust fan on BEFORE lighting the flame.

Use fireproof ducting which is in accordance with your local fire prevention regulations.

- Locate the ducting away from fire alarms, sprinkler heads and other heat-sensitive devices.
- Do not make solder joints in the ducting—the hot exhaust in the duct may melt the joint.

Regularly check the system by smoke test to ensure that the exhaust system is functioning correctly.

When operating the atomic absorption spectrometer, ALWAYS have the chimney in place to ensure correct ventilation.

Compressed gas cylinder storage and handling

All compressed gases (other than air) can create a hazard if they leak into the atmosphere.

WARNING**Explosion Hazard, Fire Hazard, Noxious Gas**

Even small leaks in gas supply systems can be dangerous.



A leak can create an explosion hazard, a fire hazard, or can result in an oxygen-deficient atmosphere.



Such hazards can cause death, serious injury, asphyxiation, anesthetic effects, and serious damage to equipment and property.

Cylinder requirements:

- Stored and handled strictly in accordance with local safety codes and regulations.
- Used and stored only in a vertical position.
- Secured to an immovable structure or a properly constructed cylinder stand.
- Stored in an adequately ventilated area to prevent toxic or explosive accumulations.
- Moved only on a properly constructed trolley.
- Never locate gas cylinders near a source of ignition or in a position that is subject to direct heat.
- Keep cylinders cool. This rule applies to every cylinder of compressed gas. Cylinders have pressure relief devices that will release the contents of the cylinder if the temperature exceeds 52 °C (125 °F).
- Ensure that all cylinders are clearly labeled so that there can be no doubt about the contents. If the cylinder label is not legible, do not use the cylinder—return it to your supplier.
- Always ensure that you have the right cylinder before connecting the cylinder to the instrument.

Safety Practices and Hazards

- Use only approved regulators and hose connectors.
- Never attempt to refill a cylinder.

For cylinder connections, left-hand thread fittings are used for fuel; right-hand thread fittings are used for support gases.

When your analytical program is complete, or at the end of the working day, always ensure that all gas supplies are turned off at the cylinder.

If air is supplied from a compressor, all moisture must be extracted from the air before it is supplied to the gas control module. Moisture can affect the internal tubing and components of the gas control system and create a potentially hazardous situation.

Gas hoses and connections

Use only approved regulators, connectors and fittings. If in any doubt, consult your local gas supplier or your Agilent representative.

Ensure that all gas connectors and hoses are correctly assembled.

Arrange gas hoses so that they will not be damaged, stepped on, or have things dropped on them.

Never use frayed or damaged hoses. Frayed or damaged hoses must be replaced immediately. This should be done by a trained Agilent representative.

Perform leak tests at all joints and seals every day before the instrument is used. Test for leaks with a brush and soapy water or a proprietary leak-detecting solution. NEVER use a naked flame when testing for leaks.

Ultraviolet radiation

Hazardous ultraviolet radiation is emitted by flames, hollow cathode lamps and deuterium lamps. This radiation can cause serious damage to human eyes and skin.

- Always wear safety glasses conforming to an approved standard, and certified or otherwise warranted to protect the eyes from ultraviolet radiation.

- Never look directly at the light emitted by a hollow cathode lamp.
- When using a flame, always operate your spectrometer with the flame shield closed, and the sample compartment front panel and chimney in place.

Warning symbols

The following is a list of symbols that appear in conjunction with warnings in this manual and on the spectrometer. The hazard they describe is also shown. The beginning of the warning text is noted by a warning icon:

WARNING

A triangular symbol indicates a warning. The meanings of the symbols that may appear alongside warnings in the documentation or on the instrument itself are as follows:



Corrosive liquid



Electrical shock



Eye hazard



Explosion hazard



Fire hazard



*Heavy weight
(danger to feet)*



*Heavy weight
(danger to hands)*



Hot surface



Moving parts



Noxious gas



Sharp object



Toxic hazard

Read all warnings and cautions carefully and observe them always.







Safety Practices and Hazards

The following symbol may be used on warning labels attached to the instrument. When you see this symbol, refer to the relevant operation or service manual for the correct procedure referred to by that warning label.



The following symbols appear on the instrument

Table 1. Instrument symbols

I	Mains power on
0	Mains power off
	Fuse
	Single phase alternating current
	Flame off
	Flame on
	Indicates lamp present.
	High leakage current – ensure proper grounding

Flame operation

Flammable solvents

Unskilled, improper, or careless use of flammable solvents in or near an atomic absorption spectrometer can create explosion hazards and fire hazards. These can result in death, or severe personal injury or burns.

Always remember that the combination of a flame and flammable solvents can present a serious hazard. All relevant safety practices governing the use of flammable solvents must be strictly followed.

To reduce the possibility of fire or explosion:

- When initially selecting an organic solvent, choose a solvent having the highest flash point consistent with your analytical requirements.
- Never use a solvent having a specific gravity lower than 0.75.
- Never leave uncovered containers of flammable solvent standing near the burner. When aspirating such solvents, always use a covered container and feed the capillary tubing through a 2 mm diameter hole in the cover. Always use the smallest volume of solvent consistent with your analytical requirements.
- Always use solvent-resistant tubing such as nitrile rubber for the drainage system and the vapor vent. Lead the drainage tube to a suitable wide-necked waste vessel (as described in the next paragraph).

The standard plastic laboratory tubing supplied with your instrument is not suitable for draining organic solvents or venting organic vapors.

If organic or toxic solutions are being used in the spray chamber, vent tubing must be connected to the vapor vent on the liquid trap and led to an active exhaust system. Do not lead the vapor tube to the waste vessel.

If you are not using toxic or hazardous liquids in the spray chamber, leave the vapor vent uncovered.

- Use small, wide-necked waste vessels and empty them frequently—do not accumulate large volumes of flammable solvent.

Do not use glass waste vessels—use vessels made of a material that will not shatter in the event of a flashback. See 'Flashbacks' on Page 27 for more information. Metal vessels will corrode and are difficult to determine the level of liquid in them.

Safety Practices and Hazards

Ensure that your waste vessel is below the instrument and located in an open, well-ventilated position where you can see it. Never locate the vessel in a confined space. When your analytical program has been completed, or at the end of the working day, always empty the waste vessel.

- When your analytical program has been completed, or at the end of the working day, always empty and clean the liquid trap.
- Do not mix nitric or perchloric acid residues with organic solvent residues.
- Keep the burner slot, spray chamber and liquid trap clean.
- Always use the internal igniter to light the flame as its operation indicates that all the safety interlocks are satisfied. Do not attempt to bypass these safety interlocks.

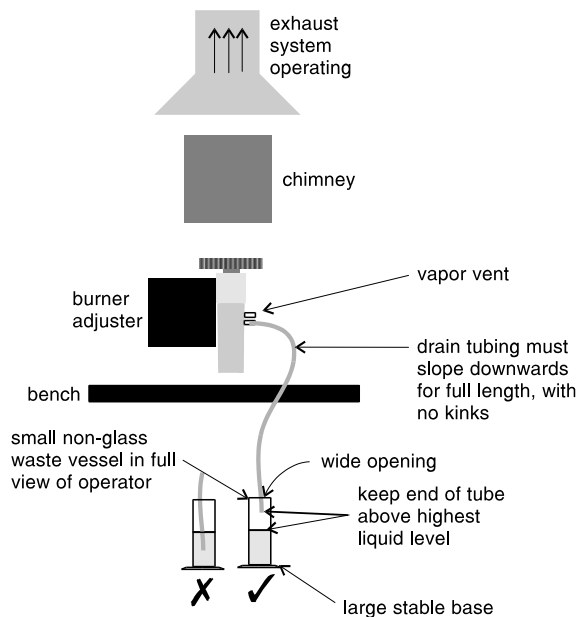


Figure 1. How drain and vent should be set up

Compressed gases and cylinders for flame operation

CAUTION

This spectrometer is to be used only with air, nitrous oxide, and acetylene for flame operation.

WARNING**Explosion Hazard**

Never use oxygen or oxygen-enriched air as the oxidant because this will result in an explosion.

NEVER use any gas except acetylene as the fuel gas.

WARNING**Explosion Hazard, Fire Hazard**

Unskilled, improper, or careless use of acetylene can create explosion hazards and fire hazards which can result in death, severe personal injury or burns.

Acetylene

Unskilled, improper, or careless use of acetylene can create explosion hazards and fire hazards that can result in death, severe personal injury, or burns.

Adhere to your local regulations governing the use of acetylene.

Use acetylene at pressures lower than 105 kPa (15 psig). At pressures above this level, acetylene can explode spontaneously. Your Agilent AA is designed to operate at fuel supply pressures between 65 and 100 kPa (9.5–14.5 psig). Refer to the 'Specifications' section or the rear of the instrument for the exact range and recommended pressure.

Do not use any tubing or connector that reacts chemically with acetylene. Never pass acetylene through copper tubing, or brass tubing or fittings containing more than 65% copper, since this may provoke an explosion. Never bring acetylene into direct contact with copper, silver, liquid mercury, gaseous chlorine, or grease, as an explosion could result.

Use only acetylene that is packed in acetone. Some gas suppliers offer acetylene packed in material other than acetone. While these alternatives may overcome some of the disadvantages of acetone, they may also introduce the more serious problem of corrosion in the gas control module and must not be used with Agilent atomic absorption spectrometers.

Safety Practices and Hazards

Acetone in the spectrometer can damage seals, O-rings and hoses, degrade analytical performance and precipitate flashbacks. Acetone may be carried over from the cylinder and into the spectrometer if either of the following conditions occur:

- Pressure in the acetylene cylinder falls below 700 kPa (100 psig).
- Consumption is greater than 1/7 of the cylinder contents per hour.

Minimize the amount of acetone that is carried over with the acetylene by:

- Replacing cylinders when their contents pressure drops below 700 kPa (100 psi)
- Ensuring that the rate of acetylene drawn off from each cylinder is not excessive.
- If high rates of consumption are observed, then connect two or more cylinders in parallel to a manifold. This reduces the rate at which acetylene is drawn from each cylinder.

To reduce the possibility of fire or explosion:

- Test the supply 'plumbing' regularly for leaks with a brush and soapy water or a proprietary leak-detecting solution (never use a naked flame when testing for leaks)
- Keep the valve outlet free from dust and debris. Before assembling regulators and fittings ensure there are no particles of dirt in the cylinder valve outlet. As regulations covering preparation and handling of gas cylinders vary from country to country, refer to local regulations to ensure compliance before connecting to your instrument.

Use 'Instrument Grade' acetylene that is at least 99.5% pure.

Turn off fuel gas at the cylinder when you have completed your flame analysis.

Nitrous oxide

The decompression of high-pressure N₂O gas at the regulator can cause excessive cooling and eventual freezing of the regulator. To prevent regulator malfunction and possible flashback, the gas should be heated with an in-line or wrap-around heater.

Burners

Improper or careless use of burners can create explosion hazards and fire hazards which can cause death, serious injury to personnel and damage to equipment and property.

WARNING

Hot Surface



The burner will remain hot for some time after the flame has been turned off. Always use protective gloves to handle hot burners.

Burners are clearly identified by the fuel/oxidant combination for which they are intended. Always fit the correct burner. Never attempt to use an air-acetylene burner for nitrous oxide-acetylene, as this will cause a flashback.

If the width of the burner slot exceeds 0.47 mm (0.0185 in) for a nitrous-oxide/acetylene burner or 0.54 mm (0.021 in) for an air/acetylene burner, the burner cannot be refurbished and must be replaced with a new one.

To ensure safe operation of burners:

- Use only acetylene as the fuel gas.
- Use only air or nitrous oxide as oxidant. Never attempt to use oxygen or oxygen-enriched air, as this will cause a flashback.
- Burner interlocks are incorporated to minimize the possibility of using the wrong burner. Never interfere with or attempt to bypass any interlock fitted to this instrument.
- To minimize the rate of burner blockage, the burner slot must be cleaned and polished as described in the Burner section of the Maintenance [Burner_maintenance](#) chapter—Page 113.
- Never allow burners to become blocked. Progressive burner blockage can increase the static pressure in the liquid trap to the point at which the liquid seal is breached. This can cause a flashback and create an explosion hazard or a fire hazard.
- Never allow carbon to build up on the slot, as glowing particles can dislodge and fall through the slot, thus causing a flashback.
- Always turn the flame off before attempting to clean the burner slot. Never clean the slot of a burner while a flame is running.
- Never leave a flame unattended.
- Never disassemble or modify a burner. *Never* use a damaged burner.

Safety Practices and Hazards

Nebulizer

Incorrect assembly and fitting of nebulizers to an atomic absorption spectrometer can create explosion hazards and fire hazards which can cause serious injury to personnel and damage to equipment and property.

Ensure that the nebulizer is correctly assembled and correctly fitted to the spray chamber before lighting the flame. Nebulizers should be correctly adjusted before lighting the flame.

Never remove a nebulizer while the flame is on, and do not use a mechanical device (e.g., a wire) to clean the capillary of a nebulizer while a flame is operating. ALWAYS extinguish the flame before removing the nebulizer from the nebulizer block.

Regularly test all connections for leaks. Rectify all leaks before lighting the flame.

Liquid trap

WARNING

Explosion Hazard, Fire Hazard, Noxious Gas



Improper use of the liquid trap can create explosion hazards, fire hazards, and toxic vapor hazards which can result in death or serious personal injury.



Never use a solution or solvent having a specific gravity lower than 0.75, otherwise the liquid seal can be breached. This can create a flashback and create an explosion hazard or a fire hazard.



The liquid trap interlock is incorporated to minimize the possibility of attempting to operate the instrument with an empty trap. Never interfere with this interlock. Never attempt to bypass this interlock.

Always fill the liquid trap with the same solvent that is being used for your samples.

The trap is designed to provide a liquid seal under all normal conditions with solutions having a specific gravity greater than 0.75.

A drainage tube must be connected to the drain outlet (the lower nipple) on the liquid trap and led to a suitable waste vessel. The free end of the tube must remain above the liquid in the waste vessel. Do not use glass waste vessels—use vessels made of a material that will not shatter in the event of a flashback.

A venting tube should be connected to the vapor vent (the upper nipple) on the liquid trap when you are analyzing organic or toxic liquids. This tubing should be led out from the sample compartment, parallel to the drain tubing, and **MUST** slope downwards to enable any liquid overflow to drain out and prevent the tubing from becoming blocked. Do **NOT** lead the vapor tube to the waste vessel. If necessary, an active exhaust system should be used to draw away toxic vapors. If you are not analyzing solutions of a toxic nature, leave the vapor outlet uncovered.

Heat hazards

An open flame, burners and other hot surfaces can present heat hazards that can result in severe burns.

When operating a flame system, always operate your spectrometer with the flame shield closed, the chimney in place, and the sample compartment front panel in place.

Chimney

Before you touch the instrument chimney, turn the flame off and allow the chimney time to cool.

Burner and flame compartment

Keep your hands out of the sample compartment while a flame is burning.

WARNING



Hot Surface

The burner and flame compartment become extremely hot during instrument operation and remain hot for some time after the instrument has been switched off.

Allow the system time to cool before attempting to access the burner or sample compartment components.

Always use protective gloves when removing a burner from the instrument.

Perchloric acid

WARNING

Explosion Hazard, Fire Hazard



Aspiration of perchloric acid and perchlorates into a nitrous oxide-acetylene flame can create an explosion hazard which can result in death or serious injury, including temporary or permanent impairment of hearing.

Do not use perchloric acid unless it is absolutely essential for sample preparation. If perchloric acid must be used, it may be possible to reduce the risk of explosion by taking the following measures:

- Use an air-acetylene flame instead of a nitrous oxide-acetylene flame.
- Reduce the concentration of perchloric acid and metal in all analytical solutions to the lowest practicable level. The concentration of perchloric acid should be reduced in the digestion stage and further reduced by extending the fuming stage.
- Aspirate all solutions for the shortest practicable period.
- Aspirate distilled water between samples. Minimize the aspiration of air.
- Use separate spray chamber/liquid trap and drain assemblies for perchloric acid analyses and organic solvent analyses to prevent perchloric acid from mixing with organic solvent residues.

NOTE

When solvent extractions of perchloric solutions are performed, some of the acid may dissolve in the organic solvent that is subsequently aspirated. Also, if the organic solution is aspirated while floating on the surface of the acid, do not allow the capillary tube to drop below the organic layer and suck up aqueous perchloric acid.

When using perchloric acid, wear approved ear protectors and approved safety glasses and ensure that all instrument safety covers are in position.

Flashbacks

A flashback is an explosion of the gas mixture in the spray chamber, which can occur for several reasons.

Agilent AA spectrometers have several safety features in place to prevent flashbacks, and flashbacks are very rare in circumstances where instruments are properly maintained.

In the rare event that a flashback does occur, the Agilent AA instrument safety features are designed to safely relieve the pressure and minimize damage. In addition to reading the list below, refer to the Maintenance section of this User's Guide (page 89).

Analysis over many years has shown that in most cases, flashbacks are associated with one or more of the following points. If you experience a flashback, check this list to see if any of the points are relevant, and take steps to remedy the situation.

- 1** Keep the burner clean. Deposits must not be allowed to build up in or on the burner slot because they can partially block it. Blockages can cause the pressure to build up in the spray chamber and breach the seal provided by the liquid trap. Alternatively, glowing particles can fall down through the slot into the spray chamber and ignite the combustible gas mixture inside.

Be especially careful of very small deposits in the ends of the burner slot because they disrupt the laminar gas flow and allow the flame to burn back into the spraychamber at "Flame Off" when the gas flow is reduced.

The use of a hard object to brush off glowing carbon particles during flame operation is not recommended because of the increased risk of knocking one of the particles down the slot.

When using an organic solvent, a reduced uptake rate should be used to restrict the amount of liquid fuel that is fed to the flame. (See page 81 of the Operations section of this manual for further information)

- 2** The width of the burner slot must not exceed the maximum design specifications:
 - Mark 7 0.46 mm (0.0181 in) for N₂O/Acetylene burner
 - Mark 7 0.54 mm (0.021 in) for Air/Acetylene burner

Safety Practices and Hazards

Even a small increase in width can greatly increase the possibility of a flashback occurring.

The burner slot must be regularly cleaned according to the instructions included in the 'Maintenance' chapter of this manual.

- 3** Ensure that the spray chamber and liquid trap are kept clean.
If dirty solutions are being analyzed (for example, engine oils), ensure that the spray chamber, liquid trap, float, and drain tube are regularly cleaned and flushed with a suitable solvent so that sludge does not build up in the parts.
- 4** Ensure that the correct O-rings are used on the burner, nebulizer block and nebulizer, and that they remain undamaged.
Damage to O-rings in the spray chamber can result in the leakage of gas that can be ignited by the flame and in turn set fire to the spray chamber.
Damage to O-rings in the nebulizer can allow leakage of the oxidant that can reduce the total flow of gas through the burner slot and so increase the possibility of a flashback occurring. Any cracked or deformed O-rings must be replaced immediately.
Ensure O-rings are correctly seated in the spray chamber, burner, and fuel and oxidant outlet nipples prior to reassembly. (See the Maintenance and Troubleshooting sections on Pages 89 and 131.)
- 5** The liquid trap must be filled with the same solution as the matrix used for the standards and samples.
- 6** The drain tube must be attached to the lower nipple of the liquid trap. The tubing must slope downwards all the way to the drain vessel so that the waste liquid drains smoothly.
The end of the drain tube must not be allowed to drop below the level of the liquid in the vessel. (Conversely, the level of liquid must not be allowed to rise sufficiently to cover the end of the tube.)

When using organic or toxic liquids in the spray chamber, a vent tube must be attached to the upper vent nipple of the liquid trap. It must slope downwards (running parallel to the drain tube) to prevent it becoming blocked should any liquid drain out, and be vented to an active exhaust system.
All the above points must be observed because a sudden surge of waste liquid can affect the pressure in the spray chamber and result in a flashback.
- 7** Since N_2O is stored in the cylinder under pressure as a liquid, when it expands through the regulator it can cool the regulator sufficiently to form ice on the outside and prevent it from operating correctly.
Prevent freezing by using a heater on the N_2O regulator on the supply cylinder. Contact the supplier of the regulator for a suitable heater.

- 8** As free acetylene is unstable at elevated pressure, it has to be stored in the cylinder by dissolving it in acetone.
If the gas is withdrawn too quickly, or the cylinder pressure is allowed to drop below 700 kPa, acetone may be drawn off in sufficient quantities to affect analytical performance, damage seals, O-rings and hoses, or even cause a flashback. Observe the recommendations concerning the use of acetylene.
- 9** Where possible, do not perform digests with perchloric acid. As this acid is known for forming unstable salts, operators using this acid should ensure that the minimum amount is allowed to reach the spectrometer, and that the burner, spray chamber and liquid trap are thoroughly cleaned after each analysis to ensure that unstable salts are not allowed to build up. Failure to do this can result in unpredictable flashbacks.
- 10** Aspirating solutions (especially alkaline/ammoniacal ones) that contain high concentrations of Ag and Cu can lead to the formation of acetylides that can spontaneously decompose and cause a flashback.

What to do in the Event of a Flashback

- Turn off the gas supply at the regulator.
- Turn off power to the instrument.
- Ensure the general area around the instrument safe by cleaning up spills in the sample introduction area or on the instrument
- Burners involved in a flashback event should not be re-used.
- Contact and report the incident to your local Agilent service representative before commencing operation of the instrument.

Resuming Operation after a Flashback

A burner that has experienced a flashback should be considered damaged and destroyed. Once a flashback has occurred there is no guarantee that the slot of the burner has not become distorted and/or enlarged to the point that it is out of production specifications.

- Inspect the sample introduction components including the spray chamber and nebulizer. Clean or replace as necessary.
 - Check the new burner condition and O-ring for damage.
 - Check the O-rings within the spraychamber, especially the pressure relief bung for damage.
 - Check the nebulizer for damage.
 - Check the optical windows on either side of the flame area for damage.

Safety Practices and Hazards

Review the relevant Safety section in this manual or the section above for detailed information on how to prevent flashbacks.

For applications that create high particulate deposits on the burner, it is recommended that additional burner cleaning/maintenance be performed. Contact Agilent for assistance if your application requires additional cleaning procedures.

NOTE

For applications that create high particulate deposits on the burner, it is recommended that additional burner cleaning\maintenance be performed.

For assistance with the flashback prevention or for additional cleaning procedures, contact your local Agilent representative.

2

Introduction

Site preparation requirements	31
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The Agilent 55B AA (double beam) flame atomic absorption spectrometer combines minimal user setup and fast sample throughput with unparalleled ease of use. The instrument is controlled via a built-in keyboard and display and can be upgraded by purchasing a PC and Agilent SpectrAA software (for more information, contact your local Agilent office).

Agilent 55B AA features include:

- Automatic monochromator, slit width, and gas selection.
- Two fixed hollow cathode lamp positions with automatic lamp selection.
- Universal Mark 7 spray chamber/nebulizer with Mark 7 burner.
- Deuterium background correction and wide range PMT.

Site preparation requirements

Prior to receiving your instrument, you will have been provided with an Agilent AA Systems Site Preparation Guide which describes the environmental and operating requirements of the Agilent AA system.

You must prepare your laboratory according to these instructions before the Agilent AA can be installed. You should keep the Site Preparation Guide for future reference. If you have misplaced your copy, you can obtain a replacement from your local Agilent office.

Specifications

Your Agilent AA instrument is designed for indoor use only. See the Agilent AA Spectrometer Site Prep Guide for specifications.

Introduction

Environmental specifications

For optimum analytical performance it is recommended that the ambient temperature of the laboratory be between 20-25 °C (68-77 °F) and be held constant to within ± 2 °C (± 3.6 °F) throughout the entire working day.

Other connections

Rear

IEEE 488

RS-232C 9-way male D-range type

Accessory 9-way female D-range type

UltrAA-lamp #1 & 2

Burndy circular 6-way, optional

WARNING



Shock Hazard

To maintain safety, only UltrAA accessories should be used at this connection.

Front (lamp compartment)

Deuterium lamp: Molex 3-way connection, behind lamp panel.



Figure 2. Deuterium lamp cover panel

WARNING



Shock Hazard

To maintain safety, only the deuterium lamp assembly should be used at this connection.

Hollow cathode lamps

2 lamp positions, automatically selected.

WARNING**Shock Hazard**

To maintain safety, only the hollow cathode-lamps should be used at this connection.

Fuses

T2.5 A H250 V, 5 x 20 mm (100-120 & 220-240 VAC)

Fuse information on the rear of the instrument is the most up to date.

NOTE

For safety reasons, any other internal fuse or circuit breaker is not operator accessible and should be replaced only by Agilent trained and certified personnel.

Gas supplies

Comply with the gas supply requirements on the rear of the instrument, these are the most current specifications.

Table 2. Gas supply requirements

	C₂H₂ (Acetylene)	Air	N₂O (Nitrous Oxide)	Air purge
	Instrument grade >99.0% pure	Clean, dry, free of oil	Instrument grade >99.5% pure	Must be clean & dry. Air filter to be used.
Allowed range	65-100 kPa (9.5-14.5 psi)	245-455 kPa (35-65 psi)	245-455 kPa (35-65 psi)	245-455 kPa (35-65 psi)
Recommended	75 kPa (11 psi)	350 kPa (50 psi)	350 kPa (50 psi)	
Normal flow rate (L/min)	0-10	13.5-20	11-16	10

Other gas connections

Sample compartment: Push-on Air/N₂O connector for burner
Push-on C₂H₂ connector for burner

Introduction

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3

Overview

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Instrument overview	36

Instructions for the customer-installable system components are outlined below. Prior to setting up your system, ensure all the requirements detailed in the AA Systems Site Preparation Guide are satisfied.

Installation checklist

Before operating the equipment, complete the following tasks:

- Connect the system components
- Connect the equipment to the power supply, and check the setting of the two voltage selection switches (see Power, Page 37)
- Install the hardware components
 - Lamps
 - Nebulizer
 - Spray chamber
 - Burner
 - Sample compartment front panel
 - Flame shield
 - Chimney
- Provide a suitable waste vessel

Overview

Instrument overview

Use the following labeled picture as a guide when installing the various components of your Agilent AA system.



Figure 3. Agilent 55B AA flame atomic absorption spectrometer – front view

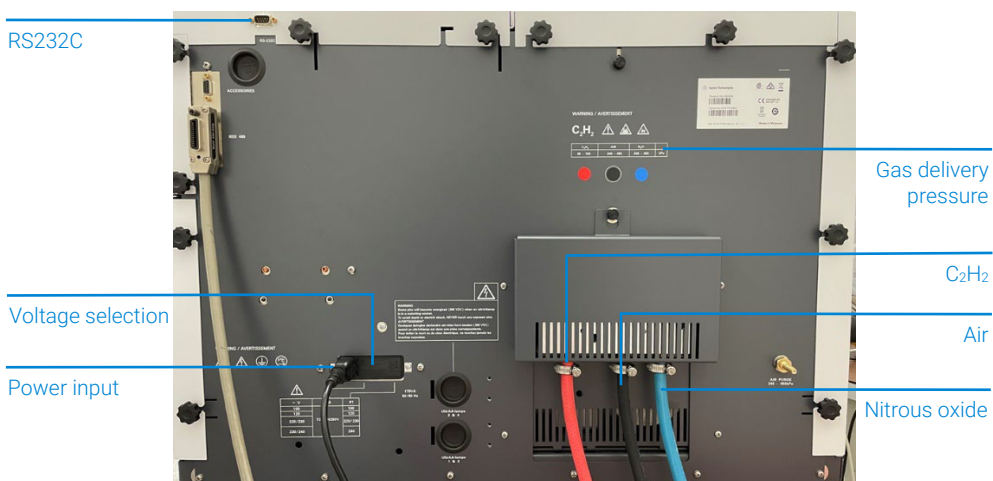


Figure 4. Agilent 55B AA flame atomic absorption spectrometer – rear view

Your Agilent AA instrument is supplied with a country kit that includes a set of gas hose fittings and a mains cable which suits the common utility standard of the local region.

NOTE

The country kit must be ordered with the Agilent AA instrument.

Power

Power requirements are detailed in the 'Electrical Power Supplies' section of the Agilent AA Site Preparation Guide. Ensure you comply with these electrical requirements before connecting the Agilent 55B AA system.

Before connecting the instrument to the power supply, ensure:

- the spectrometer is turned OFF.
- the voltage selector switch on the instrument rear panel is set to the correct mains power supply voltage.
 - refer to the table on the rear panel.
 - The voltage selector is set by the engineer when the instrument is first installed.

Plug the mains power cord into the back of the instrument and the free end of the power cord into the mains power supply.

Gas hoses

Three rubber hoses are permanently attached to the instrument. Each hose is two meters long and is color-coded for air (black), nitrous oxide (blue) and acetylene (red). Each hose is fitted with female fittings suitable for USA standard regulators.

NOTE

For gas hose connection details, refer to the Agilent AA Site Preparation Guide.

Printer

If you are using a printer, it should be connected to the instrument via a RS-232 cable in the RS-232C 9-pin port at the upper left corner of the rear of the instrument - see Figure 4, page 36. If your printer is not a serial printer, you must use a serial to parallel converter.

Overview

Accessories

Accessories, such as SIPS or computer and monitor, might require separate connection instructions. Refer to the relevant the manual for details.

Do not position the equipment so that it is difficult to operate the disconnecting device.

Moving your instrument

WARNING



Heavy Weight

The instrument weighs over 50 kg (110 lb). Do not attempt to lift the instrument alone. Always use two or more people when lifting or carrying the instrument into position.

Lamp compartment



Figure 5. Lamp compartment

The lamp compartment contains positions for two hollow cathode lamps.

The D₂ lamp module is located within the lamp compartment, on the lower left side and is interlocked to prevent inadvertent exposure to hazardous UV radiation. Power to the lamp is removed when the deuterium lamp panel is removed.

Hollow cathode lamps

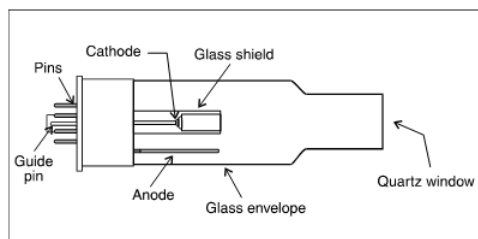


Figure 7. Hollow cathode lamp

You can use the following types of lamps:

- Single element
- Multi-element

Instructions on how to install and remove hollow cathode lamps can be found in the Maintenance section, Page 119.

Deuterium (D₂) lamp

You will need to replace the D₂ lamp approximately every 1000 hours of operation. For instructions on how to change a D₂ lamp, refer to the Maintenance chapter - Page 121.

Sample compartment



Figure 6. Sample compartment

Overview

The major components in the Sample Compartment includes:

- Nebulizer block assembly
- Nebulizer
- Spray chamber
- Burner
- Sample compartment front panel
- Flame shield

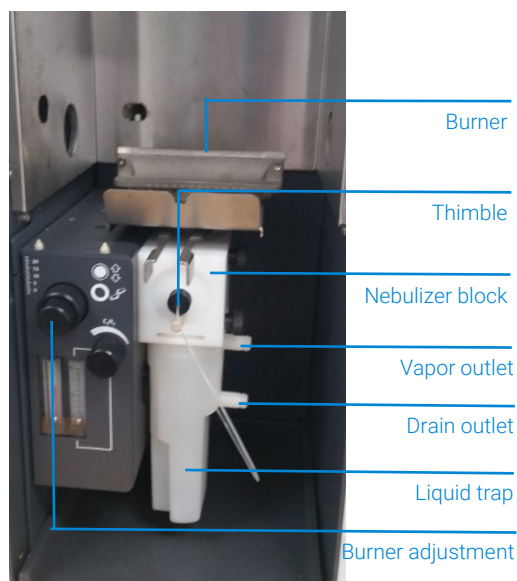


Figure 7. Sample compartment with front panel and flame shield removed

Nebulizer block assembly

The nebulizer block connects the following components:

- Nebulizer
- Impact bead
- Liquid trap
- Drain tube
- Spray chamber

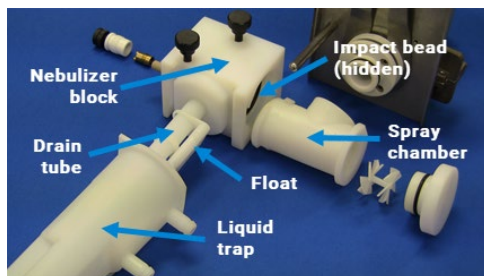


Figure 8. Nebulizer block assembly (for products with serial numbers 0110xxxx and later)

Nebulizer

The nebulizer uses a capillary tube, venturi, and impact bead to convert the sample solution into a spray.

NOTE

When you receive your instrument, the nebulizer will be assembled and installed in the sample compartment. For instructions on how to remove, disassemble, and reassemble the nebulizer refer to 'Nebulizer block assembly' - Page 91.

Ensure that the nebulizer is correctly assembled in the nebulizer block with the metal capillary protruding through the locking thimble (see 'Reassemble the nebulizer', Page 95).

- Attach the spray chamber to the nebulizer block and install the complete assembly in the sample compartment as described in 'To assemble the spray chamber' - Page 106.)

Impact bead

The impact bead is located within the nebulizer block and is aligned so that the bead is centered directly in front of the capillary tube outlet. Large droplets from the capillary hits the glass impact bead and forms a fine mist of the sample solution.

Instructions on replacing the Impact bead are in the Maintenance section, see Page 98.

Liquid trap

The liquid trap enables excess solution to be drained from the nebulizer assembly. The trap is designed to provide a gas seal under all normal gas flows when using solutions having a specific gravity greater than 0.75.

Overview

Connect a length of tubing to the lower drain outlet and into a suitable waste vessel. The free end of the tube must always remain above the level of liquid in the waste vessel. Use vessels made of an inert material that will not corrode or shatter in the event of a flashback. Do not use waste vessels made of glass or metal.

If you are using organic or toxic liquids in the spray chamber, a length of tubing must be connected to the vapor vent (upper nipple) to vent toxic vapors to an active exhaust system. The tubing should run parallel to the drain tubing and must slope downwards to prevent it becoming blocked should liquid overflow and drain from the vapor vent.

CAUTION

Do not lead the vapor vent tubing to the waste vessel.

If you are not using hazardous solutions in the spray chamber, leave the vapor vent free.

Drain tube

The drain tube is a plastic tube which screws into the bottom of the nebulizer block to drain waste solution into the liquid trap. It should be inspected regularly to ensure it is clean and free from blockages.

Float

For products with serial numbers 0110xxxx and later, the float is captive to the drain tube. When the float and tube are present, and the trap is filled to the correct liquid level (Page 66), a magnet in the float triggers an interlock which enables the burner to ignite.

For instructions on how to set up the nebulizer to suit your analytical requirements, refer to Nebulizer setup- see Page 79.

Spray chamber

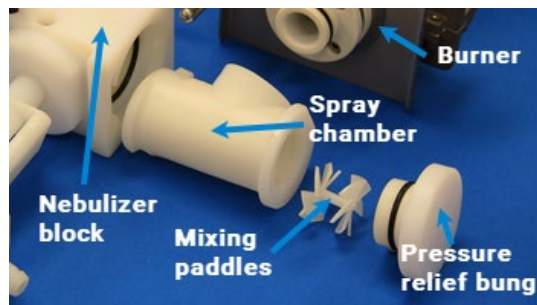


Figure 9. Nebulizer/spray chamber components

The spray chamber attaches to the rear of the nebulizer block via a bayonet ('twist and lock') mount and connects with the burner. Combustible gases and sample solution are mixed in the spray chamber before passing through the burner slot for consumption in the flame.

The spray chamber contains the following components:

- mixing paddles
- pressure relief bung (also called the spray chamber bung).

Mixing paddles

The mixing paddles fit into the entry of the spray chamber. They remove the larger aerosol droplets which can cause photometric noise and burner blockage.

The front of the mixing paddles has a boss at the center of the blades.

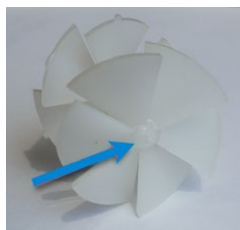


Figure 10. Front of mixing paddles showing the boss

Three of the blades at the back of the mixing paddles have small retaining lugs on their edges. These lugs hold the paddles in position within the spray chamber.

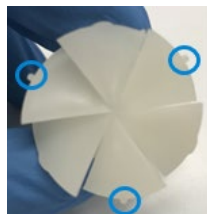


Figure 11. Back of the mixing paddles

When installing the mixing paddles, the front end of the paddle is inserted into the front of the spray chamber – the end further away from the nebulizer.

Overview

Pressure relief bung

A pressure relief bung is located at the rear of the spray chamber. In the event of a flashback occurring, this bung is ejected to relieve excess pressure. For safety, the bung points away from the operator.

The burner/spray chamber system incorporates an interlock design with two functions:

- Prevent ignition if the pressure relief bung is not correctly fitted.
- Shut down the flame if the bung is ejected because of a flashback.

Instructions to assemble the spray chamber are in the Maintenance section - Page 106.

Burner

There are two types of burner available for use with your Agilent AA system: an air-acetylene burner or a nitrous oxide-acetylene burner.

Both burners include an interlock key designed to prevent ignition if either a burner is not fitted, or a burner is fitted which is not suitable for the selected flame type. A handle enables you to rotate the burner so that it aligns with the optical path. The burner can also be adjusted vertically and horizontally by rotating the adjustment knobs found on the front face of the burner adjuster.

The burners are identified by their labels and dimensions of the burner slot:

- Air-acetylene burner: 10 cm slot length.
- Nitrous oxide-acetylene burner: 6 cm slot length.
- Instructions on how to clean and install burners are in the Maintenance section - see Page 91.

CAUTION

Only Mark VI A or Mark 7 burners should be used with this system. Previous versions should not be installed.

WARNING

Explosion Hazard, Fire Hazard, Hot Surface.

Improper or careless use of burners can create explosion hazards and fire hazards which can cause death, serious injury to personnel, and damage to equipment and property. Whenever you handle a burner, remember that it may be very hot.



Always use protective gloves to handle hot burners.

Always fit the correct burner.



Never attempt to use an air-acetylene burner with a nitrous oxide-acetylene flame, this will cause a flashback.

Never interfere with or attempt to bypass burner interlocks.

Never attempt to dismantle or modify a burner.

Sample compartment front panel

The sample compartment front panel is designed to reduce heat and radiation hazards. Always fit this panel before using your instrument.

To fit the sample compartment front panel:

- 1 Position the panel in the sample compartment, orientated as shown in Figure 12 below.
- 2 Align the panel so that the two small holes in the cover fit over the two lugs on top of the burner adjuster.



Figure 12. Lower Sample Compartment Front Panel over the two lugs on top of the burner adjuster.

- 3 Rest the panel on the burner adjuster.

Flame shield

The flame shield hinges onto the front of the sample compartment and is designed to reduce heat and radiation hazards from the flame. An interlock prevents you from igniting the flame unless the flame shield is closed. Always fit

Overview

the flame shield before using your instrument, and leave it closed unless you need access to the burner.

WARNING



Eye Hazard

Some flames emit hazardous levels of UV radiation, which can cause cataracts and skin cancer. Always keep the flame shield, sample compartment front panel, and chimney in place when the flame is operating.

To fit the flame shield:

- 1 Hook the flame shield onto the bar at the top of the sample compartment.
- 2 Lower the flame shield until it rests on the stainless-steel bosses on either side of the sample compartment.

To remove the flame shield, lift it up and unhook it from the front of the sample compartment.

NOTE

A distinct click sound can be heard from the micro-switches within the instrument when the flame shield is fitted correctly into place.

WARNING



Hot Surface

The chimney and flame shield may become very hot during operation and cause severe burns if touched. Always wear protective gloves when handling a hot chimney.

Chimney

The chimney sits on top of the sample compartment and is designed to protect the operator from the heat and UV radiation emitted by the flame. It is a double-wall construction to reduce the surface temperature of the outside wall. It also ensures that the exhaust system is effective in extracting vapors and fumes.

NOTE

The chimney should be approximately 100mm (4in) from the extraction hood of your exhaust system. This distance should be increased when operating a nitrous-oxide flame due to the higher flame height.

To fit the chimney:

- 1 Hold the chimney over the sample compartment, with the warning information facing towards the front of the instrument.
- 2 Lower the chimney onto the sample compartment.

4

Interface

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Display pages	56

This chapter provides an overview of the Agilent 55B AA user interface. Read through this chapter while exploring the interface.



Figure 13. Front panel interface

Front panel overview

The front panel of the Agilent 55B AA consists of the following:

- Liquid Crystal Display—LCD
- Keypad
- Speaker, to provide audible prompts or warnings
- Memory for storage of up to 30 customized methods

Interface

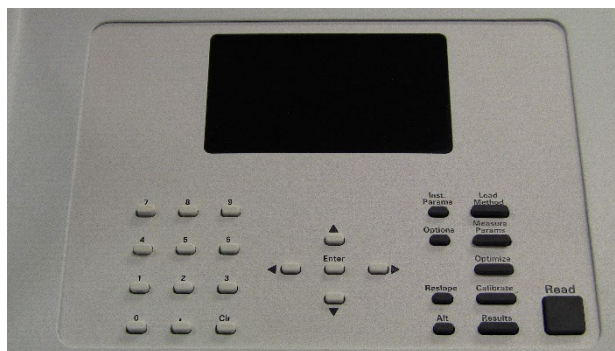


Figure 14. Agilent 55B AA front panel

Display screen



Figure 15. Display screen

The display is divided into four areas: status area, signal bar, page area and message line, each of which is discussed below.

Status area

The status area displays the method details and solution results. Method details include settings relating to the method currently loaded, such as method number and element. Solution results include:

- The mean value in absorbance (Abs), or transmittance (T) if the method is uncalibrated, or
- Concentration if calibrated and
- Precision in %RSD or %Pr, depending on the measurement mode.

Method details are displayed in normal font. Results are shown in enlarged font.

Signal bar

The signal bar appears:

- at the top of the display screen where required.
- spans the entire width of the screen, and
- graphically displays signal values, depending on the type of reading that the instrument is performing.

If you are optimizing:

- A hollow cathode lamp or D₂ lamp, the signal bar will display the lamp emission.
- The flame signal, the signal bar will display the atomic absorption or emission.

The signal bar is only active when the Optimize, Calibrate, or Results page is selected. At other times the bar is blank.

Page area

The page area displays the name of the current page and page-specific information such as input fields, text, or graphics. Refer to Display pages on Page 56 for details on the different pages in the system.

Message line

The message line displays information/warning/error messages. This information may be the permissible range of the current parameter, or a warning or error message. Refer to Troubleshooting – Page 131 for a detailed description of possible error messages, and possible solutions to these errors.










The Message line also informs you of the calibration status of the method. In the message line:

- “Cal” indicates the method has been calibrated.
- “Rslp” indicates the calibration has been resloped.
- No text (“ ”) indicates that the method has not been calibrated.

Icons appear in the message line, to provide you with instrument status information. Some of these icons are animated, to indicate that the instrument is active.







Interface

Table 3. Instrument icons and their meanings

Icon	Meaning
	Instrument busy - please wait
	Monochromator slewing
	Instrument peaking
	Slit changing
	D ₂ lamp warming up
	Instrument finding zero order
	No burner fitted
	Air/acetylene burner and ignition sequence
	N ₂ O/acetylene burner (showing narrower slit) and ignition sequence

Gas type and oxidant pressure status icons are as follows:

Table 4. Gas type and oxidant pressure icons and their meaning

Icon	Meaning
	Air-acetylene, oxidant pressure present
	Air-acetylene, no oxidant pressure
	Air only, oxidant pressure present
	Air only, no oxidant pressure
	Nitrous oxide-acetylene, oxidant pressure present
	Nitrous oxide-acetylene, no oxidant pressure

NOTE

Gas type icons displayed in Table 3 are only displayed during the initiation sequence.

Keypad

The keypad keys are divided into five groups:

- Page keys
- Numeric keys
- Cursor keys
- Alt key
- Miscellaneous keys

Page keys



Figure 16. Page keys

There are seven page keys, each of which take you to a specific page in the system. These keys are:

- Load Method,
- Instrument Parameters,
- Options,
- Optimization,
- Measurement Parameters,
- Calibrate, and
- Results.

For a description of the function of each page, refer to 'Display pages' on Page 56.

The **Calibrate** key is also used, in conjunction with the **Alt** key, to view the calibration graph. There are other diagnostic pages accessed via key combinations. For details, refer to Alt key- Page 52.

Interface

Numeric Keys



Figure 17. Numeric keys

The numeric keypad (0-9 and decimal point) is used to enter field values. A Clear button ('Clr') is also included in this group to delete any values entered.

Cursor keys



Figure 18. Cursor keys

There are four cursor keys: 'Up', 'Down', 'Left', and 'Right'. They are used to move around the display. The 'Enter' key is used to update the parameter setting with the new value.

NOTE

If the cursor is moved out of a field without 'Enter' being pressed, the field's contents returns to its previous value.

'Enter' is also used on the 'Optimization' page to perform a Rescale command.

Alt key



Figure 19. Alt key

'Alt' is used in conjunction with other keys to modify their meaning. Pressing 'Alt' alone has no effect.

The table below lists the shortcuts available via the keypad.

Table 5. Keypad shortcuts available

Key sequence	Action	Available
ALT+Calibrate	Display calibration graph	Anywhere
ALT+Results	Display error log	Anywhere
ALT+Up	Increase LCD contrast	Anywhere
ALT+Down	Decrease LCD contrast	Anywhere
ALT+Clear	Flame shutoff (for N ₂ O)	Anywhere
ALT+Enter	Print method (if Serial==PRINTER)	Anywhere
ALT+.	Save system information to EEPROM	Anywhere
ALT+50	General diagnostic page	Anywhere
ALT+51	EEPROM data diagnostic	Anywhere
ALT+52	Motor diagnostic page	Anywhere
ALT+53	RS232 loopback test	Anywhere
ALT+54	SIPS arm pressure page	Anywhere
ALT+59	Keyboard diagnostic	Anywhere
ALT+Calibrate	Exit keyboard diagnostics	Keyboard diagnostic page
ALT+Results	Exit keyboard diagnostics	Keyboard diagnostic page
ALT+Left	Menu scroll text	Any menu
ALT+Right	Menu scroll text	Any menu
ALT+Read	Instrument zero	Optimization, Results or Calibrate page
Read	Read next standard	Calibrate page
Reslope	Perform reslope	Calibrate page
ALT+Reslope	Drop reslope standard	Calibrate page
Read	Take next reading	Results page
Reslope	Perform reslope	Results page
ALT+Reslope	Drop reslope standard	Results page
Right	Prime SIPS pump page	Results page
Up	Increment next sample	Results page
Down	Decrement next sample	Results page
Clear	Clear result window, start new page if Serial=PRINTER	Results page

Interface

Miscellaneous keys



Figure 20. Miscellaneous keys

The 'Read' key is used to start an analysis (see 'Calibrate the method' - Page 84 and 'Sample measurement – Page 86).

The **Reslope** key is used to select the reslope standard, and to perform a reslope operation (see 'Reslope the calibration' - Page 85).

Navigating the interface

Changing pages

Press the required 'Page' Key. Changes made on a given page are only downloaded and executed when another page is selected.

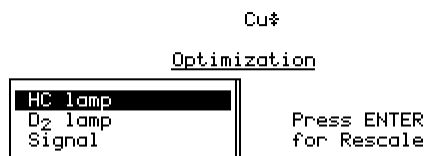
The exception to this is when changing back and forth between the Load Method page and the Instrument Parameters page. The parameters are not downloaded at this time, allowing you to load a method and edit the instrument parameters simultaneously. Open another page for the Load Method and Instrument Parameters page changes to be enacted.

Selecting menus

Command menus are single-level menus appearing in the left of the screen. Use the 'Up' and 'Down' keys to move from one item to the next, and, if necessary, press 'Enter' to select the desired option.

In the example below, to rescale the signal:

- 1 Press the **Down** key twice.
- 2 Press **Enter**.



Cal

Figure 21. An example of a Command menu

A Parameter menu has a left list box that contains items which access other sub-menus. Press the 'Right arrow' key on the required menu option to access the available sub-menu.

In the example below, to change the Instrument mode to Emission:

- 1 Press **Right arrow**.
- 2 Press **Down**.
- 3 Exit the Instrument Parameters page for the changes to be incorporated.

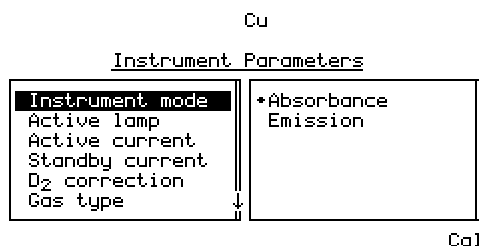


Figure 22. An example of a Parameter menu

NOTE

An arrow appearing in the frame around a menu indicates there are additional options available. Use the **Up/Down arrow** keys to scroll through the list, or the **Left/Right arrow** keys to move from one menu to the next.

Interface

Moving between fields

The cursor shows where the current focus is. When the cursor is on an entry field, the entire field is highlighted. To move between entry fields, use the 'Up/Down arrow' keys.

```
          Cu#
      Measurement Parameters
Next sample      [  1 ]
Batch number     [          ]
Pre-read delay   [ 0.0 ] secs
Read time        [ 3.0 ] secs
Replicates       [  3 ]

Range is 0.2 to 60.0          Cal
```

Figure 23. The Measurement Parameters page, with the focus on the 'Read time' field

As data is entered (via the Numeric keys) it appears at the end of the field. If 'Clr' is pressed the entire field value is deleted. When 'Enter' is pressed, the cursor moves to the next field (providing the value is valid). If the value entered is invalid, the cursor remains on the entry field and the value is not accepted.

Display pages

The system consists of seven pages, each accessed by pressing its corresponding key. The pages are as follows:

Load Method page

The Load Method page is used to load either a user method or a cookbook method. You should follow the prompts appearing on this page to load the type of method you want. For more information, refer to Method development. - Page 67.

Instrument Parameters page

The Instrument Parameters page is used to set the instrument parameters and the save and delete methods.

NOTE

The element can only be selected by loading an appropriate method (user or cookbook).

This page includes the following instrument-specific fields:

Instrument mode

Defines the mode you want to use, 'Absorbance' or 'Emission' - default is Absorbance.

Active lamp

Defines which lamp position to use - (default is position 1).

Active current

Defines the current for the active lamp - default setting is the recommended current for the lamp of the selected element.

Standby current

Defines the current for the standby lamp - default is 0.0.

D₂ correction

Defines whether D₂ correction is to be used or not - default is 'No'.

Gas type

Defines the flame type to be used for the analysis - default is the recommended gas type for the selected element.

Wavelength

Defines the wavelength to be used for the current element - default is the first listed wavelength.

NOTE

The listed wavelengths are element and instrument-mode specific.

Other λ

Defines the wavelength to be used when 'Other' is selected in the Wavelength field. Range is 180.0–900.0 nm.

Slit

Defines the spectral bandwidth (SBW) for the current wavelength - default is the recommended slit width for the current wavelength.

Interface

Save method

Saves the current method and completed calibration to memory.

NOTE

Any conditions being used on the Measurement Parameters page and Options page should also be saved using this function.

Delete method

Deletes a method from memory.

Measurement parameters page

This page is used to set the measurement parameters to be used.

NOTE

The Measurement mode is located on the Options page (see Page 59). This parameter must be set prior to setting the remaining measurement parameters.

Batch no. & Next sample

Identifies the next sample to be measured. This has no effect on the reading.

Pre-read delay

Defines the time allowed for the sample to reach the light path and for the signal to stabilize.

Read time

Defines the time for which the signal is measured.

Replicates

Defines the number of readings per solution. This option is only available in Integration mode.

Precision

Defines the % precision for the Precision Optimized Measurement Time (PROMT) measurement mode. Not available when SIPS is enabled.

Options page

In addition to selecting the Measurement mode, this page is used to enable options such as SIPS use and serial output and select the language of the interface.

Measurement mode

Determines how the signal will be measured. The following modes are available:

Integration

The signal is averaged over the selected 'Read time' for the set number of replicates.

PROMT (PRrecision Optimized Measurement Time)

The signal is averaged until the desired precision (%RSD) is achieved or until the end of the 'Read time'.

NOTE

When SIPS is being used in a PROMPT, set this value to 0.3%.

Integrate repeat

Replicates are read continuously without being initiated by the 'Read' key. Pressing the 'Read' key takes the most recent replicate as the solution (sample or standard) result.

This mode should NOT be used when the SIPS accessory is being used.

SIPS

Displays the following sub-menu:

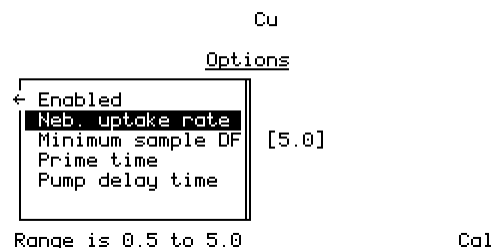


Figure 24. The SIPS sub-menu of options

Interface

Enabled

Enables SIPS use - default is 'No'.

Neb. uptake rate

Controls the maximum pump speed. Although expressed in mL/min, the actual flow rate may be less than this, depending on tube condition, solution viscosity etc. Ensuring the nebulizer uptake rate exceeds the number entered guarantees correct pump operation (Beware subsequent nebulizer blockages, which may invalidate this condition).

Minimum sample DF

Defines the minimal sample dilution factor to be used. Primarily intended for where the majority of samples are suspected over-range. This will prevent unnecessary dilution factor changes, thus speeding up sample throughput.

Prime time

Defines the SIPS pre-read delay, which is the time required to pump solutions introduced with SIPS through the pump tubing from the solution vessel to the T-piece.

Pump delay time

Defines the time taken for the sample absorbance to reach equilibrium after a change in pump speed and for the signal to stabilize. Further information on how to use SIPS is outlined in the Operation section – see Page 65.

Serial port

Displays the following sub-menu:

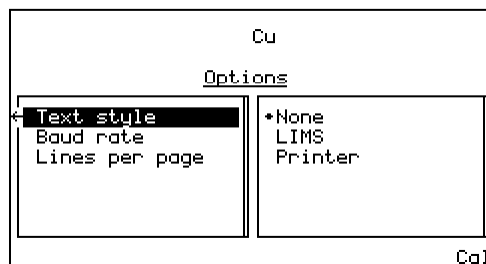


Figure 25. The Serial port sub-menu of options

Text style

Enables LIMS or printer output - default is none. All output requires the 'Baud rate' to be set correctly. 'LIMS' output has comma-delimited fields. 'Printer' output has space-delimited fields.

See Print the results - Page 87 for details on output options.

Baud rate

Specify the baud rate to be used for the serial port. Default is 9600.

Lines per page

Used when 'Printer' is selected as the text style- default is 66 lines per page. Each page has a header. Form feeds are used to separate pages.

Language

Select the language of the interface - default is English. Refer to 'Select the language of the interface' - Page 67 for instructions on how to change the language of the interface.

Optimization page

The Optimization page is used to set up:

- the hollow cathode lamps
- the D₂ lamp, and
- the signal.

In Absorbance mode there are up to three options present on the page:

- HC lamp (default position 1)
- D₂ lamp and
- Signal.

In Emission mode, only 'Signal' is available, because during emission optimization, the system uses the highest standard sample to carry out the wavelength peaking procedure (i.e., no lamps are used).

The signal bar at the top of the page facilitates optimization of the lamps, burner position, and impact bead position to obtain the strongest signal.

Refer to 'Optimization' on page 72 for instructions on fine tuning the system.

Interface

Calibration Parameters page

Use this page to define the standards used for the calibration, to measure the standards, and to select the reslope.

Standard conc. 0

This field is set to zero, and is not editable, but enables the blank to be measured.

Standards 1...5

Defines the concentrations of the calibration standards to be used. The value must be greater than zero and have four significant figures, with up to 3 decimal places. No two standards may have the same value, and they must be entered in increasing order. Entering a value of zero will delete the current and subsequent standards.

The reslope standard is set by selecting the desired standard and pressing the **Reslope** key. The standard selected will be marked with an asterisk beside it to indicate it is the Reslope standard. See 'Reslope the calibration' on Page 85 for more information.

NOTE

If SIPS is enabled, the cursor is restricted to the last entry field, which is the concentration of the bulk standard. Standards 1-4 are calculated from the diluted bulk standard. The reslope standard is fixed at 3.

Results page

The Results page displays the solution results in tabular format, and displays sample name, concentration, %RSD or precision, mean solution reading, dilution factor and the last replicate value.

Cu

Sample	Conc	Results		DF	Reps
		%Pr	Mean		

Cal

Figure 26. The Results page

Table 6. Interpreting the Results page

Column	Entry	Comment
Sample	'nnn' for a sample 'Std-n' for a standard, and 'Reslope' for the reslope standard	
Conc	The concentration values of the solutions measured The text UNDER or OVER if the solution is under-range or over- range or UNCAL if the method is uncalibrated	
%Pr	<ul style="list-style-type: none"> • %RSD in Integration mode • %Pr in PROMT mode and • Remains blank in Integrate Repeat mode (with the heading %Pr). 	
Mean	The mean of the cumulated readings for each solution.	
DF (dilution factor)	The dilution of the solution Range available is 1.000...200.0.	Only includes values if SIPS is enabled
Reps (last replicate value)	The value of each replicate as it is calculated	Only used for Integration mode Each value is used to calculate the mean result, but the display only shows the last value.

The contents of the Results page are cleared if:

- A new method is loaded,
- The Measurement mode is changed or
- The Instrument mode is changed

Interface

This page is intentionally left blank.

5

Operation

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This chapter provides instructions for running your Agilent 55B AA instrument, including:

- How to develop methods.
- Optimize the system.
- Perform measurements and
- Print the results.

It is assumed that the appropriate hardware has been correctly installed and that you are familiar with the interface.

Pre-analysis checklist

To prepare for analysis:

- 1** Check that all the required hardware (including SIPS and printer if necessary) is correctly installed.
- 2** Turn on the instrument and any peripheral accessories.
- 3** Ensure the exhaust system is working by testing the extraction system with lab tissue paper.

Operation

- 4 Check the gas supplies to make sure you have enough gas to complete your analysis. Ensure that acetone is not drawn into the instrument by checking that the acetylene cylinder pressure is above 700 kPa (about 100 psi) and that the rate of acetylene drawn from each cylinder is not greater than 1/7 of the cylinder contents per hour.
- 5 Set the delivery pressure of the gases to the following:

Table 7. Gas delivery pressures – Refer to the panel on the rear of the instrument

Gas	Recommended		Permissible	
	kPa	PSI	kPa	PSI
Acetylene	75	11	65-100	9.5-14.5
Air	350	50	245-455	35-65
Nitrous oxide	350	50	245-455	35-65

- 6 Inspect the gas hoses for damage. Hoses showing any signs of damage or deterioration must be replaced immediately. This should be done by a trained Agilent representative.
 - Turn on the gas supplies to your instrument and test all hoses and connections for leaks. If a leak is found, turn off the gas supplies immediately and rectify the problem.
- 7 Fill the liquid trap with the solvent being used until the float just starts to move.
 - If the liquid trap has not been filled to the correct level, or if the drain tube has been left out, an interlock in the float prevents the ignition of the burner. The interlock will also shut down the flame if the liquid level falls below the required minimum during operation.
- 8 Develop or load a method - see 'Method development' on Page 67.
- 9 Optimize the system - see 'Optimization' on Page 72.
- 10 Set the nebulizer uptake rate - see 'Nebulizer setup' on Page 79.
- 11 Calibrate the system - see 'Calibrate the method' on Page 84.
- 12 Proceed with your analysis.

Start the system



Figure 27. Power switch

The power switch is shown above. Switch this to 'I' to start the system. The green indicator light to the right of the switch should come on.

When you switch on your instrument, the startup screen will appear in the display area while the instrument performs its initialization tests. When the initialization setup is complete the Load Method page is displayed.

For instructions on how to start any accessories, refer to the manuals supplied with the accessories.

Method development

A method stores the parameters to be used for analysis, such as instrument, measurement and calibration settings. You must have a method loaded in order to run samples. There are two types of methods— 'user' methods where settings are defined by the user and 'cookbook' methods.

Select the language of the interface

If the required language is displayed, skip this step.

Operation

To change the language of the interface:

- 1 Press the **Options** key. If the Options page displays the SIPS sub-menu, press the **Left arrow** key to return to the main menu.
- 2 Move the cursor to 'Language' and press the **Right arrow** key.
- 3 Move the cursor to the desired language and press **Enter**.

NOTE

Keyboard overlays are also available for supported languages.

Load a method

You can:

- Load a 'cookbook' method.
- Customize your own 'user' method, or
- Load an existing 'user' method.

To load a cookbook method:

- 1 Press the **Load Method** button.
- 2 Select 'Cookbook'.
- 3 Select the appropriate element group.
- 4 Select the required element from the list.
- 5 Press **Enter**.

The cookbook parameters will load:

- Element
- Method number
- Instrument mode
- Active lamp current
- Gas type
- Wavelength and
- Slit width

In addition, all previous standard concentration values will be deleted.

Enter concentration values before starting the analysis.

To load/modify an existing user method:

- 1 Press the **Load Method** button.
- 2 Select 'User' methods and press **Enter**.
- 3 Select the method you wish to use.
- 4 Modify the method as required.

To customize a method:

- 1 Load the cookbook or user method for the required element and modify the parameter settings as necessary. The following sections include instructions on setting the various parameters.
- 2 Save the method on the Instrument Parameters page.

Save/Delete methods

Methods are saved on the Instrument Parameters page. There are 30 slots available for customized methods. Slots that are occupied are displayed on the Load Method page. A method can be saved into any slot, even if the slot is already occupied by an existing method. The existing method will be overwritten by the new method (without a request for confirmation).

To save a method:

- 1 Press the **Instrument Parameters** button.
- 2 Move the highlight to the 'Save Method' option and press **Enter**.
- 3 Select an empty position and press **Enter**.

The method will be saved in the selected slot, with the slot number and method element displayed in the list.

To delete a method:

- 1 Press the **Instrument Parameters** button.
- 2 Move the highlight to the 'Delete Method' option and press **Enter**.
- 3 Move the highlight to the method in the list to be deleted and press **Enter**. The method will be deleted, leaving an empty slot in the list.

Operation

Set the instrument parameters

To set the Instrument parameters:

Select the Instrument Parameters page and specify:

- 'Instrument mode' (Absorbance or Emission)
- 'Active lamp' (position 1 or 2, position 1 is the default)
- 'Active current' for the active lamp
- 'Standby current' for the standby lamp
- 'D₂ correction' – 'On' or 'Off'
- 'Gas type'
- 'Wavelength'
- Spectral bandwidth (the width of the 'Slit').

Set the measurement parameters

All measurement parameters are set on the Measurement Parameters page, with the exception of the Measurement mode and the use of SIPS, which are set on the Options page.

To set the measurement parameters:

- 1 Select the Options page and specify:
 - The 'Measurement mode'.
 - Whether 'SIPS' is enabled or not. If you choose to enable SIPS, you will also need to set the SIPS parameters.
- 2 Select the Measurement Parameters page and specify:
 - 'Next sample' to be measured
 - 'Batch no'
 - 'Read time'
 - Number of 'Replicates' (Integration mode only)
 - 'Precision' (PROMT mode only)
 - 'Pre-read delay'

Define the calibration standards

Calibration standards are entered on the 'Calibration Parameters' page. Up to five standards may be defined. Calibration standards must be unique, and they must be entered in increasing order.

Cal Zero is not editable and is included solely to allow for measurement of the blank. The field value is always zero.

NOTE

If SIPS is enabled the only editable field on the Calibration Parameters page is Conc5, which represents the concentration of the bulk standard. Conc1 to 4 are calculated from the bulk standard, and the Reslope standard is set at 3. For more information about Reslope the calibration see Page 85.

To enter the calibration information:

- 1** Press the **Calibrate** button.
The Calibration Parameters page appears.
- 2** Move the highlight to the 'Conc1' field and enter the concentration of standard
The number of decimal places entered will set the number of decimal places used for the remaining standard values. Even when changing the number of decimal places, all concentrations must still be valid (not over range or identical).
- 3** Enter the remaining standard concentrations. The values must be unique and increasing.
- 4** Set the reslope standard to be used by moving the cursor to the appropriate standard and pressing the **Reslope** key. An asterisk indicates the reslope standard.

Optimization

This section describes how to optimize the various components of the Agilent 55B AA system, including the lamp, burner and flame signal.

Align lamps

Hollow cathode lamps

Install the lamp in the position designated in the method. The position number of the lamp is located on the Instrument Parameters page. If you are developing a new method, install the lamp and note the position. Ensure this position is defined in the method for that element.

Align a hollow cathode lamp if:

- you have just installed the lamp.
- you need the best possible signal to noise ratio.
- you are using a non-Agilent lamp.
- you are using a position previously used for a non-Agilent lamp.

NOTE

Lamps are not used in Emission mode. Models that use this mode do not require lamp alignment. Signal optimization is performed while aspirating the top/bulk standard to carry out the wavelength peaking procedure.

To align a hollow cathode lamp:

- 1 Load a Method.
- 2 Ensure the required lamp is installed in the correct position. This is defined on the Instrument Parameters page.
- 3 Turn on the lamp and allow approximately 15 minutes for the lamp to warm up. If the lamp is not glowing, review the method and set the 'Active lamp' and 'Active current' on the Instrument Parameters page.
The Active lamp number can be found on the lamp holder and the Active current is printed on the lamp.
- 4 Press the **Optimize** button and ensure nothing is in the optical path. The 'HC lamp' option is selected automatically upon entering the page.
- 5 Maximize the signal:

Watch the lamp signal bar on the display as you slowly turn one of the lamp adjustment knobs.



Figure 28. Lamp adjustment knobs

If the signal decreases, turn the knob in the other direction.



Figure 29. Lamp signal bar

- 6 If the HC lamp signal is too small or too large:
 - check that you have the correct lamp for the current method.
 - If the correct lamp is installed, press **Enter** to Rescale. This will bring the signal back into range for the display.
- 7 Repeat the previous step with the other adjustment knob.

D₂ lamp

The D₂ lamp only needs aligning after a new D₂ lamp has been installed.

To align a D₂ lamp:

- 1 On the 'Optimize' page, select the 'D₂ lamp' option.
- 2 Maximize the signal.
 - a Watch the lamp signal bar on the display as you slowly turn one of the D₂ lamp adjustment knobs located on the front of the D₂ lamp compartment.

Operation



Figure 30. D₂ lamp adjustment knobs

- b** If the signal decreases, turn the knob in the other direction.
- c** If the D₂ lamp signal is too small:
 - Check that the D₂ lamp is installed correctly. (refer to Page 121).
 - If the problem persists, press **Enter** to Rescale. This will bring the signal back into range for display.
 - A signal that becomes too large should also be rescaled.
- 3** Repeat the previous step with the second D₂ lamp adjustment knob.

Align the burner

- 1** Allow approximately 10-15 minutes for the hollow cathode lamp to warm up and ensure nothing is in the optical path.
- 2** Rotate the burner by squeezing the prongs of the rotation handle, until the slot is parallel to the light path.



Figure 31. Squeeze prongs of rotation handle to rotate burner

- 3 Locate the light path using an Agilent supplied burner cleaning and alignment card.

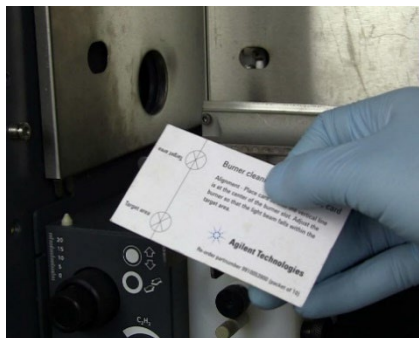


Figure 32. Agilent Burner Cleaning and Alignment Card

NOTE

Additional burner cleaning and alignment strips are available from Agilent.

- 4 Place the alignment card halfway along the burner slot. Position the card with the vertical line perpendicular to the slot.

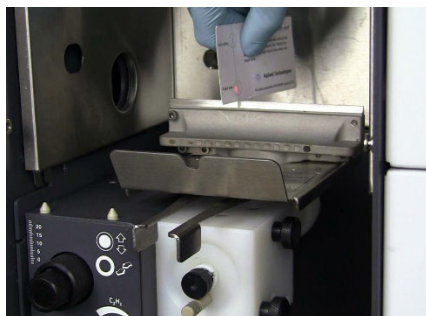


Figure 33. Card with vertical line perpendicular to the slot

- 5 Rotate the burner adjustment knobs (Figure 34 below) to position the burner horizontally and vertically with the light beam in the target area.

Operation



Figure 34. Burner adjustment knobs (left). Rotating the knob (right).

- 6 Check that the slot is parallel and centered on the light path by placing the card at each end of the burner slot.

The light path should fall in the target area along the length of the slot. Readjust if necessary.

You can now light the flame and optimize the signal.

Light the flame

Prior to lighting the flame, you must ensure that:

- The working area is completely clear of all hazardous materials including corrosive liquids and flammable solvents.
- The liquid trap is filled with an appropriate solvent.
 - Fill the liquid trap with the solvent being used until the float just starts to move.
 - If the liquid trap has not been filled to the correct level, or if the drain tube has been left out, an interlock in the float prevents the ignition of the burner. The interlock will also shut down the flame if the liquid level falls below the required minimum during operation.
- The drain tube is positioned sloping downwards and is free of kinks or sharp bends so that the waste liquid can drain freely into the vessel. The end of the tube must always be above the expected level of liquid in the vessel.
- Dependent on the solvent used, ensure the ventilation tube is attached and is free of kinks and bends. The end of the tube must be positioned sloping downwards and be free of kinks or sharp bends so that the waste liquid can

drain freely into a separate overflow vessel. The end of the tube must be above the expected level of liquid in the vessel.

- The correct burner is fitted.
- The flame shield, sample compartment front panel, and chimney have been fitted.
- The pressure relief bung O ring has been lubricated with distilled water and the bung has been installed correctly.
- The exhaust system has been checked and the gas supplies are sufficient for the analysis. Refer Page 33.

To light the flame:

- 1 Press and hold the 'Flame on' button. until the flame ignites.



Figure 35. Flame on button

If the ignition sequence times out before the flame ignites, release the button, wait about five seconds, and restart the sequence.

CAUTION

If an error is displayed, ensure all safety interlocks have been satisfied before continuing. Do not attempt to bypass any safety interlock.

WARNING



Explosion Hazard

Operation of a flame with an incomplete safety system can result in a violent explosion that can cause death, injury, hearing loss or property damage.

To prevent unsafe operation, ignition of the flame is prevented unless all safety interlocks are satisfied. If the igniter fails to operate, and all safety interlocks are satisfied, call an Agilent trained and certified engineer to find and fix the fault.

Never use an external igniter to light the flame as this will bypass the inherent self-checking of the safety system.

- 2 Check the gas regulator settings and readjust if necessary.
- 3 When the flame has stabilized, optimize the flame conditions.
- 4 Aspirate 50 mL of the solution. If not a solution, aspirate 50 mL of the appropriate solvent.

Optimize the flame signal

To optimize the flame signal:

- 1 Press the **Optimize** key and select the 'Signal' option.
- 2 Aspirate the blank and press the **Alt** and **Read** keys together to perform an Instrument Zero.
- 3 Aspirate a standard solution that will give an absorbance of at least 0.2. If you are in emission mode, aspirate the bulk standard or the highest concentration standard.

Watch the signal bar and adjust the burner height using the outer knob on the burner adjuster to obtain the maximum absorbance.

Ensure the burner is below the light path.

Alternate between aspirating the blank and the top standard, making a note of the net absorbance value. When this value stops increasing the burner height is correct.

The burner height controls the sensitivity and will also influence atomization interferences.

- 4 Carefully move the burner horizontally by turning the inner adjustment knob on the burner adjuster. Watch the signal bar to obtain maximum absorbance. In

general, once optimized for maximum sensitivity, this position can be used for all analyses.

- 5 Adjust the impact bead position by gradually turning the impact bead adjustment screw first clockwise, then counter-clockwise to find the maximum absorbance.



Figure 36. Impact bead adjustment screw

TIP

You can find the optimum bead position for most applications by aspirating a 5 mg/L solution of copper.

The system is now optimized. You can now take readings, measure your calibration standards, or perform a reslope as described in the following sections.

Nebulizer setup

This section assumes you have correctly assembled the nebulizer and installed the nebulizer/spray chamber assembly in the sample compartment.

The nebulizer is fully adjustable, allowing you to change the position of the internal capillary to vary the uptake rate from 0 to 8 mL/min, compensating for the fueling effect of organic solvents.

The setting of the thimble determines how close the impact bead is to the outlet of the capillary tube. This enables the optimal nebulizer setting based on the characteristics of your samples.

Operation



Figure 37. Thimble location. The locking ring is between the thimble and the nebulizer block

High vacuum setting

For samples with relatively low levels of dissolved solids, optimum performance is achieved with the nebulizer in high vacuum mode. Here the capillary is set to give the maximum pressure drop (vacuum) at the capillary tip. This setting uses narrow-bore plastic capillary tubing (0.38 mm ID).

A nebulizer set up in this way will show minimum variation in uptake rate with variation in liquid level in the sample tubes. The nebulizer may, however, be more susceptible to clogging by salts deposited from the sample.

To set the nebulizer in high vacuum mode:

- 1 Take one length of the narrow-bore (0.38 mm ID) plastic capillary tubing supplied with your nebulizer.
- 2 Fit the flared end of this tubing to the nebulizer capillary - trying to fit the other end will damage the tubing.
- 3 Set up the instrument to measure absorbance using an easily atomized element (such as copper) in an air-acetylene flame.
Since the uptake rate will be low, the initial sensitivity will be less than normal.
- 4 Set the instrument in continuous read mode.
- 5 Zero the instrument by pressing the **Alt** and **Read** keys together.
- 6 Loosen the locking ring and turn the ring and thimble fully clockwise.
- 7 Observe the absorbance while nebulizing a suitable standard solution.
- 8 Slowly and carefully turn the thimble counter-clockwise until the absorbance reaches a maximum.
- 9 Hold the thimble so it cannot turn, then turn the locking ring until it is firmly against the spray chamber body, locking the thimble in place.

The nebulizer is now ready for use. If you have just installed the nebulizer, you should carry out the Performance checks- see Page 83.

Low vacuum setting

In low vacuum mode (the high solids setting) the capillary position is adjusted close to the end of the venturi throat. The greater clearance around the capillary tip reduces clogging caused by solutions with a high solids content. When using this setting, the pressure drop (vacuum) at the capillary tip is relatively low. Consequently, you must use wide bore (0.64 mm ID) plastic sampling capillary tube to achieve the uptake rates required for maximum sensitivity.

As the uptake rate will be sensitive to the hydrostatic head of the liquid in the sample container, care is required to minimize variations in liquid levels in the sample and standard containers.

To set the nebulizer to low vacuum mode:

- 1 Fit the wide bore (0.64 mm ID) tubing to the capillary.
- 2 Loosen the locking ring and turn the thimble (and ring) fully clockwise.

The nebulizer is now ready for use.

NOTE

At this setting, the uptake rate will be sensitive to the hydrostatic head of the liquid in the sample container, thus care is required to minimize variations in liquid levels in the sample and standard containers.

Set zero uptake rate – organic solvents

Before adjusting the nebulizer for use with organic solvents, set zero uptake rate using distilled water as the solvent:

- 1 Fit the wide bore (0.64 mm ID) tubing to the capillary.
- 2 Turn the locking ring anti-clockwise until it is up against the thimble.
- 3 Turn the thimble clockwise as far as it will go.
- 4 Aspirating distilled water, turn the thimble counter-clockwise until bubbles emerge from the end of the capillary tubing.
- 5 Turn the thimble clockwise to the point where bubbling ceases.

This is the zero-uptake point (no solution is being aspirated through the nebulizer).

Set the uptake rate for organic solvents

The fueling effect of organic solvents require careful adjustment of the nebulizer uptake rate for all analyses that use organic solutions.

Operation

Set the uptake rate as follows, using an air-acetylene flame and the selected solvent:

- 1 Light the flame and adjust the acetylene flow to give a very lean flame.
- 2 Set the nebulizer for zero uptake as described in the previous section.
- 3 Slowly rotate the thimble clockwise until the flame is beginning to turn fuel-rich. Some yellow may be seen.
- 4 Tighten the locking ring against the nebulizer block to prevent movement of the thimble during normal operation.
- 5 Measure and record the uptake rate.

This should be between 3 and 6.5 mL/min.

The nebulizer is now ready for use. If you have just installed the nebulizer, you should carry out the performance checks described in the next section.

Flame types

The appearance of the flame changes dependent on the fuel/oxidant mix. Typical flames are shown below:

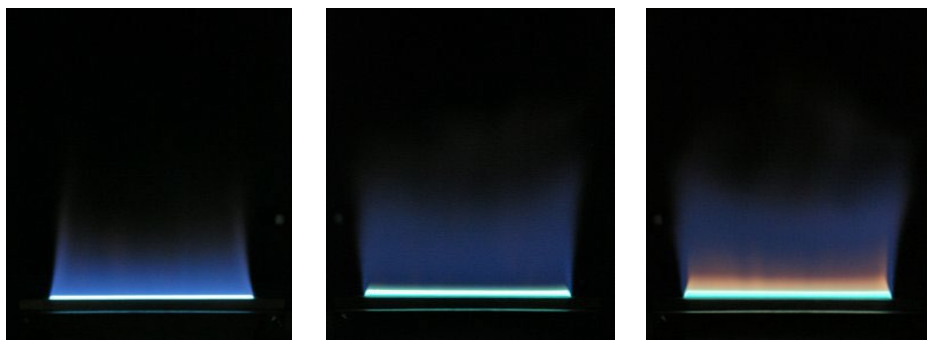


Figure 38. Air/Acetylene flames. Left to right – Lean, Stoichiometric, Rich

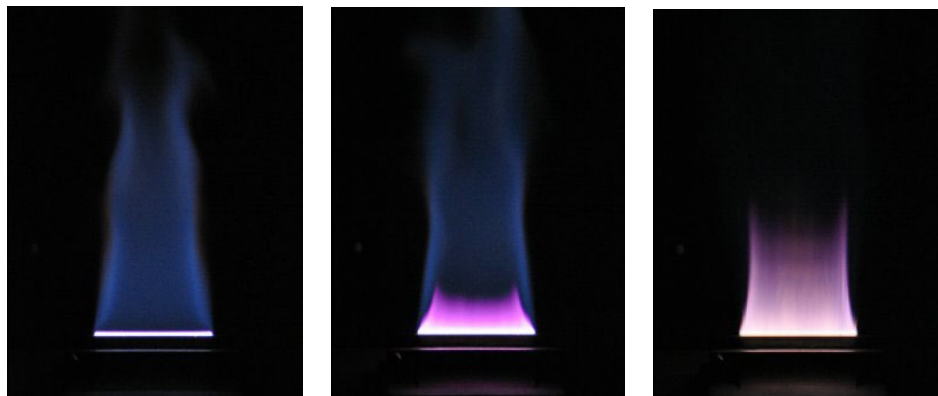


Figure 39. Nitrous oxide/Acetylene flames. Left to right – Lean, Stoichiometric, Rich

Performance checks

Check the performance of your nebulizer:

- At installation.
- Weekly.
- When using a new type of solution.
- After the nebulizer has been pulled apart and re-assembled.

To check the performance of your nebulizer:

Ensure the nebulizer is correctly installed and set up. Set the nebulizer to high vacuum mode as per 'High vacuum setting on Page 80, or if you are using organics, set the nebulizer to maximum uptake rate (thimble fully clockwise).

- 1** Prepare an aqueous solution of copper with a concentration of 5 mg/mL.
- 2** Set the instrument up to measure the absorbance of copper in an air-acetylene flame.
- 3** With the system fully optimized as described in 'Optimization' on Page 72, aspirate distilled water from a 10 mL calibrated cylinder for a carefully timed period and calculate the uptake rate. This should be between 3 and 6.5 mL/min for a High Vacuum setting.
- 4** Zero the instrument (press the **Alt** and **Read** keys together).
- 5** Aspirate a solution of 5 mg/mL copper and note the absorbance reading.

Agilent guarantee an absorbance of 0.75 or better using the Mark 7 flame atomization system under fully optimized conditions.

Calibrate the method

All calibrations use the New Rational algorithm, which provides a quadratic least squares line of best fit in the Abs/Conc vs. Abs domain:

$$A/C = a + b \cdot A + c \cdot A^2$$

(where A = Absorbance; C = concentration; and a,b,c are curve coefficients).

Calibration standards are measured on the Calibration Parameters page. You can measure the standards manually or use SIPS to prepare and present the standards automatically from one bulk standard.

NOTE

Remeasuring Cal Zero adjusts the signals of all standards.

If you receive an error due to calibration failure, note the error number and refer to Troubleshooting (see page 131) for a description of the problem.

Measure standards manually

To measure the calibration standards manually:

- 1 Define the standards to be used for the calibration.
- 2 Select the Cal Blank by moving the focus to the 'Conc0' field, aspirate the blank and press the **Read** key.
- 3 Select 'Conc1', aspirate the first standard and press the **Read** key.
- 4 Repeat Step 3 for the remaining standards.

Measure standards using SIPS 10

Automatic calibration from a single standard can be performed using the SIPS 10.

To calibrate a method using the SIPS 10:

- 1 Set up the SIPS 10 as described in the SIPS manual.
- 2 Develop or modify a method.
- 3 Enable SIPS on the Options page.
- 4 In the SIPS sub-menu, set the required parameters.
- 5 Press the **Calibrate** button to display the 'Calibration Parameters page'.

- 6 Enter the concentration of the bulk standard in the Conc5 field. (When SIPS is enabled, the cursor is restricted to the Conc5 entry field.) The first four standards will be calculated automatically, with the Reslope standard fixed at Conc3.
- 7 Ensure the diluent tubing is connected to the Mariotte vessel.
- 8 Ensure the pump tubing is placed in the bulk standard and press **Read**. Standards 0-5 are read in order. SIPS stops when the calibration is complete.

Display the calibration graph

To view the calibration graph, you must be on the 'Calibration Parameters page', at least one standard must be defined and measured, and the calibration must be valid (i.e., the fit is good).

The calibration graph is viewed by pressing the **Calibrate + Alt** key. Use the 'Arrow' keys to move the cross hairs on the graph. The numbers displayed indicate the current signal and concentration at the location of the cross hairs.

Reslope the calibration

Reslopes are a quick way of adjusting the slope of the current calibration.

The nominated standard (indicated on the Calibration Parameters page by an asterisk) is remeasured, a concentration calculated for the Reslope standard (C_r) and compared to the previous known concentration for that standard (C_s).

All subsequent sample results are multiplied by the Reslope correction factor, C_s/C_r .

To perform a reslope:

- 1 On the Calibration Parameters page, move the cursor to the standard you want to use as the Reslope standard.
- 2 Press the **Reslope** key to mark the concentration to be used for the Reslope standard.

NOTE

If you are using SIPS the Reslope standard defaults to Conc3.

- 3 Aspirate the selected standard (if you are using SIPS the standard will be prepared automatically).
- 4 On the Results page, press the **Reslope** key.

Operation

The system will:

- Measure the standard selected in the method.
- Mark this measurement as the Reslope standard, and
- Update the existing calibration.

NOTE

Cal Zero is not remeasured when a Reslope is performed.

Sample measurement

Sample measurements are displayed on the Results page. You can measure samples manually or use SIPS to facilitate online dilution of over range samples.

Measure samples manually

To measure samples manually:

- 1 Set the Set the measurement parameters as described on Page 70.
- 2 Press the **Results** key.
- 3 Aspirate the appropriate sample (the sample highlighted on the 'Results' page) and press the **Read** key.
- 4 Repeat the previous step for the remaining samples.

Measure samples with SIPS

You can use SIPS 10 to perform automatic dilution of over range samples.

If the sample is over range, SIPS automatically dilutes the sample being tested and measurement continues. The dilution factor used is given on the Results page and the final dilution-corrected concentration is reported.

To measure samples using SIPS 10:

- 1 Set up the SIPS 10 as described in the SIPS manual.
Select SIPS on the Options page and set the required parameters - see Page 59.
- 2 Set the remaining Instrument Parameters as described in Page 56.

- 3 Press the **Results** key.
- 4 Ensure the diluent tubing is connected to the Mariotte vessel.
- 5 Place the pump tubing in the appropriate sample and press the **Read** key.

Results

The solution results are displayed on the Results page - see Page 62.

Print the results

You can print the solution results to an external printer, using the RS-232 9-pin cable to connect the instrument to your PC or printer.

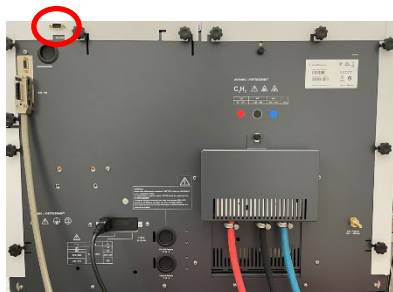


Figure 40. RS-232 9-pin socket located on the rear of the instrument

If the printer is not a serial printer, use a serial to parallel converter.

To print the results:

- 1 Go to **Options** page > **Printer** > **Text**.
- 2 Set the required 'Baud rate' and 'Lines per page'.
- 3 Ensure the instrument is connected to the printer.
- 4 Press the **Read** key to measure the current solution.
As each solution is measured, the result will be sent to the printer.

Operation

Output to LIMS system

Results can be sent to a LIMS system using the RS-232 output on the rear of the Instrument.

To output to a LIMS system:

- 1 Connect a RS-232 cable from the rear of the instrument to the RS-232 input on the LIMS, for example the COM1 connector on the rear of a PC.
- 2 Set the baud rate of the connection to that of the controlling system. This is done at the Serial Port sub menu of the Options menu.

Each time the READ key is pressed data is output, the layout is as follows:

Calibration Standard ID, Concentration, Background Absorbance, Mean Absorbance, Replicates Absorbances.

Batch Number, Sample Number, Concentration, Background Absorbance, Mean Absorbance, Replicates Absorbances.

Shut down the system

Prior to shutting down the system at the end of the working day you should aspirate pure solvent for 10 minutes.

To shut down the system:

- 1 Shut down any accessories according to their manuals.
- 2 Turn off the instrument.
- 3 Shut off all gas supplies at the regulators and gas cylinders.
- 4 Empty the waste vessel.
- 5 Turn off the exhaust fan.
- 6 Remove and clean any hardware according to the instructions in the Maintenance Chapter – see Page 90.

6 Maintenance

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This chapter describes how to maintain your Agilent 55B AA system.

You should keep a record in your Instrument Log Book of all maintenance carried out.

CAUTION Before you remove any hardware, you must shut down the instrument.

Maintenance schedule - general

Daily

- Check the exhaust system has positive extraction.
- Check the gas supplies.
- Check all hoses and connections for damage, leaks and wear.
- Clean the instrument.
- Clean the burner.
- Empty the drain/waste vessels.
- Spray chamber and liquid trap – detailed below.

Maintenance

Weekly

- Check for O-ring deterioration.
- Clean the lamp and sample compartment windows.
- Check the air filter, spray chamber and liquid trap.

Yearly

- Arrange for Agilent trained and certified personnel to perform preventative maintenance.

Maintenance schedule – nebulizer/spray chamber

Daily

- Clean the nebulizer/spray chamber assembly thoroughly at the end of every working day by aspirating at least 50 mL of the appropriate solvent through the system.
- Test all connections for leaks with a brush and soap solution or a proprietary leak-detecting solution. Never use a naked flame when testing for leaks. Rectify all leaks before lighting the flame.
- Test the nebulizer for leaks. If any leakage is detected, replace the internal O-rings.

Monthly

- Remove and check the nebulizer, re-install the nebulizer (outlined in the following section) and measure the uptake rate to verify the nebulizer performance checks– see Page 83.

Cleaning

General

Clean the instrument surfaces:

- 1 When you are finished, switch off the instrument and allow it to cool.

- 2 Remove any hardware present in the sample compartment (refer to the appropriate instructions if necessary).
- 3 Using a cloth dampened with water, clean the sample compartment, instrument cover, flame shield and chimney.

CAUTION

Do NOT use an abrasive cleaner.

If necessary, use a mild detergent to clean the cover.

Windows

Clean the windows:

- 1 Clean the sample compartment window and the lamp compartment windows with a gentle stream of filtered air or inert gas, or a brush similar to those used to clean camera lenses.
- 2 If the windows are dirty, wipe them using an optical tissue held in plastic tweezers and dampened with a 50% ethanol/water solution.

Filters

If using an air compressor to supply the air, check the filters in the air supply line and clean if necessary.

Nebulizer

This section describes how to remove, disassemble, clean and reassemble your nebulizer.

Remove the nebulizer block

To remove the nebulizer:

- 1 Turn the flame off.
- 2 Allow the burner to cool.
- 3 Remove the burner.
- 4 Remove the length of capillary tubing from the nebulizer.

Maintenance

- 5 Pour about 500 mL of water through the top of the spray chamber to remove toxic or corrosive waste out of the spray chamber drain tube and liquid trap.

WARNING



Corrosive Liquid, Toxic Hazard.

Contact with corrosive or toxic substances can result in death, or severe personal injury or burns.

Always wear appropriate safety equipment and dispose of waste liquids according to your local laws and regulations.

-
- 6 Detach the length of tubing from the liquid trap drain outlet. If fitted, detach the length of tubing from the liquid trap vapor outlet.
 - 7 Unscrew both knurled retaining screws on the side of the nebulizer block.
 - 8 Pull the complete nebulizer/spray chamber assembly sideways until it is clear of the mounting and remove the assembly from the sample compartment.
 - 9 Remove the liquid trap:
 - Hold the nebulizer/spray chamber assembly firmly in one hand.
 - Twist the liquid trap in a counter-clockwise direction to unlock the bayonet mount.
 - 10 Hold the nebulizer/spray chamber assembly upside down, unscrew the plastic drain tube from the bottom of the nebulizer block.
 - 11 Separate the nebulizer block and spray chamber.
 - Hold the nebulizer block firmly in one hand and twisting the spray chamber in a counter-clockwise direction to unlock the bayonet mount.
 - 12 Disassemble the nebulizer as described in the following section.

Disassemble the nebulizer

The nebulizer is comprised of multiple components.

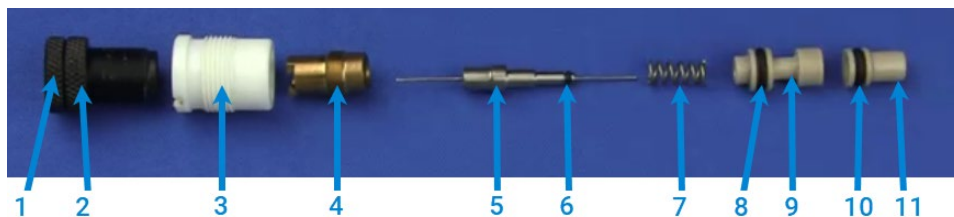


Figure 41. Nebulizer components:

1. Thimble 2. Locking ring 3. Ertalyte Bush Bearing (white plastic insert) 4. Clamp bush guide 5. Capillary assembly 6. O-ring, 1/32 ID x 3/32 OD x 1/32" 7. Spring 8. O-ring, 3/16 ID x 5/16 OD x 1/16" 9. Capillary guide 10. O-ring, 3/16 ID x 5/16 OD x 1/16 11. Venturi

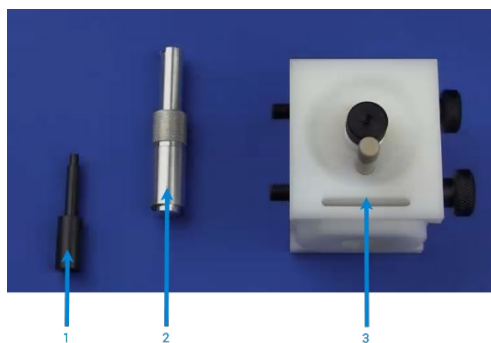


Figure 42. Nebulizer block and specialized tools

1. Venturi extraction tool 2. Nebulizer tool 3. Nebulizer block – front face

To disassemble the nebulizer:

- 1 Hold the nebulizer block front face up.
- 2 Unscrew the thimble in a counter clockwise direction and remove the thimble and locking ring.

Use the large end of the nebulizer tool to unscrew the white plastic insert from the nebulizer body.

NOTE

If the white plastic insert has stripped threads. Remove the old insert as described above and insert a new white plastic insert.

- 3 Use the narrow end of the nebulizer tool to unscrew the clamp bush guide from the white plastic insert.
- 4 Remove the capillary assembly and the spring.
- 5 Turn the nebulizer block over to the back face.

Maintenance

This is where you access the venturi.

NOTE

Before you remove the venturi, the impact bead must be removed or moved away from the venturi. To do this, slacken the clamping screw that secures the impact bead in the adjuster block, and move the impact bead to the side (see Page 99).

- 6 Insert the small end of the venturi extraction tool into the venturi.



Figure 43. Insert the extraction tool into the venturi

- 7 Push down on the extraction tool and both the venturi and capillary guide are removed.
- 8 Carefully inspect all components for wear and corrosion. Replace all faulty items. Instructions on how to clean a blocked nebulizer follows.

Clean a blocked nebulizer

Check the nebulizer body, capillary and venturi for corrosion. Nebulizer issues can be minimized by aspirating 50-500 mL of distilled water at the end of each working day.

- 1 Turn the flame and instrument off.
- 2 Remove the plastic capillary tubing from the nebulizer.
- 3 Remove and disassemble the nebulizer, check the components.
- 4 Place the nebulizer components in an ultrasonic cleaner containing 0.5% liquid detergent solution for 5 to 10 minutes.
- 5 If the ultrasonic bath fails to clear the blockage, pass a burr-free nebulizer cleaning wire (supplied with the nebulizer) carefully through the capillary, then repeat the ultrasonic cleaning procedure.
- 6 Rinse the nebulizer components in distilled water, then allow to dry.

- 7 Check for a blockage at the junction between the plastic capillary tubing and the capillary assembly. If a blockage is evident, either cut off the clogged section of plastic capillary or fit a new piece. Ensure the plastic capillary tubing fits tightly on the nebulizer capillary.

Cut off only the required amount of the plastic tubing that is required to remove the blockage.

NOTE

After cutting off the clogged section, it may be necessary to 'flare' the end of the capillary tube before it can be fitted to the nebulizer. You can do this with a heated needle.

Reassemble the nebulizer

WARNING

Fire Hazard, Explosion Hazard.



Incorrect assembly and/or fitting of the nebulizer can create explosion hazards and fire hazards which can cause serious injury to personnel and damage to equipment and property.

NOTE

When you fit a new capillary assembly, you should also fit a new capillary guide and spring.

When you fit a new venturi, replace all O-rings. Always replace O-rings as a set. If you are using organics, ensure you fit the specific organic O-ring kit.

To reassemble the components of the nebulizer:

- 1 Hold the nebulizer block front end up.
- 2 Place the venturi in the nebulizer body, thin end first.

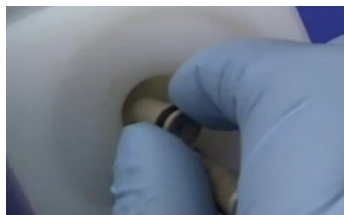


Figure 44. Insert Venturi into Nebulizer Block

Maintenance

- 3 Place the capillary guide into the nebulizer body, with the O-ring end up.



Figure 45. Insert Capillary Guide into the Nebulizer Block

- 4 Place the large end of the venturi extraction tool over the capillary guide.
- 5 Push down on the capillary guide and venturi.
The venturi should protrude from the opposite end of the nebulizer block.



Figure 46. Venturi extending into the back of the Nebulizer Block

- 6 Insert the O-ring end of the capillary assembly into the spring.



Figure 47. Insert spring over the capillary assembly
1. Spring 2. O-ring 3. Capillary assembly

- 7 Insert the capillary assembly and spring into the capillary guide.

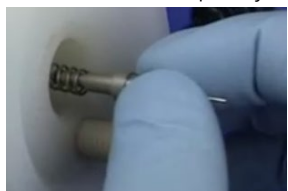


Figure 48. Capillary assembly and spring being inserted into the capillary guide.

- 8 Screw the white plastic insert into the nebulizer block using the large end of the Nebulizer tool.

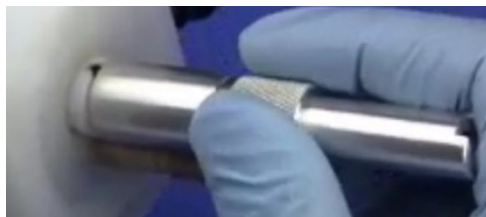


Figure 49. Screw the white plastic insert into the Nebulizer block

NOTE

Do not force the white plastic insert. It may be necessary to screw counter-clockwise first for lead-in thread to be located.

- 9 Place the clamp bush guide, thread end first, in the nebulizer body.



Figure 50. Insert the clamp bush guide into the Nebulizer block.

- 10 Screw the clamp bush guide into the white plastic insert using the small end of the Nebulizer tool. Do not over-tighten.
- 11 Check the spring is operational by pushing the capillary assembly with a screw driver. The assembly should move in and out freely.



Figure 51. Check spring function

Maintenance

12 Thread the thimble and locking ring over the capillary.

13 Screw the thimble onto the nebulizer body.

Instructions on how to adjust the Nebulizer are detailed in 'Nebulizer setup' on Page 79.

Impact bead

NOTE

Glass impact beads are most common and are suitable for most solvents. An impact bead made from PTFE is available for use with Hydrofluoric Acid. The instructions are valid for both types of impact bead.

The spherical end of the impact bead is positioned over the venturi throat of nebulizer. The impact bead helps to separate the larger droplets from the sample aerosol. Its position and adjustment have significant effect on the sensitivity and noise performance of the instrument.

This section describes how to remove and replace the impact bead.

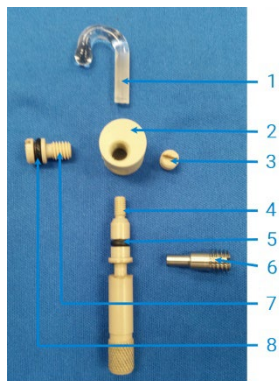


Figure 52. Impact bead components

1: Impact bead

2: Impact bead adjustment block

3: Impact bead locking screw

4: Impact bead adjustment screw

5: O-ring

6: Trapping screw

7: Clamping screw

8: O-ring 3/16 ID

Check the impact bead for erosion or other damage. If the bead is damaged or broken, fit a new one.

Remove the impact bead

- 1 Remove the nebulizer block from the sample compartment.
- 2 Isolate the nebulizer block by removing the nebulizer block, the liquid trap, drain tube and spray chamber -see Page 91.
- 3 Loosen the Impact bead locking screw approximately two turns.

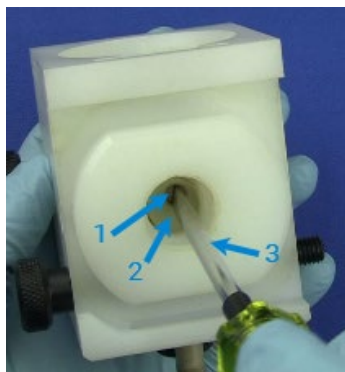


Figure 53. Loosen the Impact locking screw - bottom of the nebulizer block

1. Impact bead locking screw 2. Impact bead adjustment block 3. Flat-blade screw driver

- 4 Using a slight twisting motion, remove the impact bead from the nebulizer block.

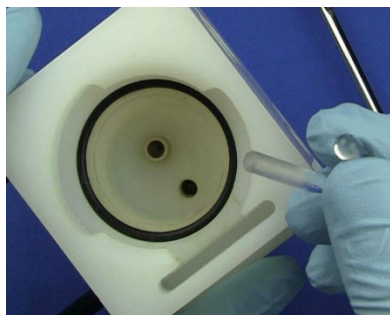


Figure 54. Glass Impact Bead removed from the back of the Nebulizer Block

NOTE

You should only perform the remainder of this procedure if the bead is broken and trapped in the adjuster block.

- 5 Loosen the Trapping screw 3-4 turns.

Maintenance

The Trapping screw retains the Impact bead adjustment screw in the nebulizer block.

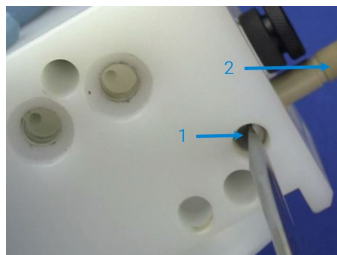


Figure 55. Loosen the trapping screw - gas inlet side of nebulizer block
1. Trapping screw 2. Impact bead adjustment screw

- 6 Unscrew the Impact bead adjustment screw and remove it from the nebulizer block. Some effort may be required to pull past the O-ring.

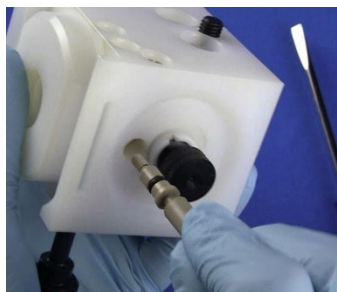


Figure 56. Remove the impact bead adjustment screw – front of the nebulizer

- 7 Loosen the Clamping screw to remove the Impact bead adjustment block.

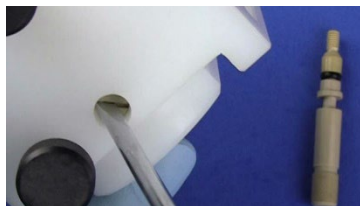


Figure 57. Loosen the clamping screw – side of nebulizer.

- 8 Remove the Impact bead adjustment block from the nebulizer block - it should drop out once the Clamping screw is sufficiently loosened.

WARNING**Eye Hazard**

This procedure may dislodge small fragments of glass. To protect the eyes from flying pieces of glass, protective eye wear should always be worn when performing this procedure.

- 9 Carefully push the broken portion of the Impact bead out from the Impact bead adjustment block.

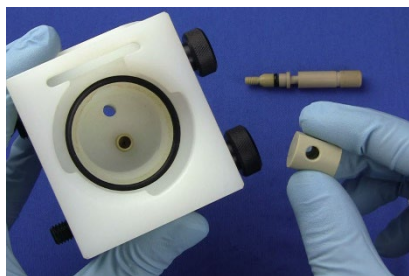


Figure 58. Impact adjustment block removed from the nebulizer block

Replace the impact bead

- 1 Insert the Impact bead adjustment block into the bottom of the nebulizer block.

Ensure the hole in the Impact bead adjustment block is aligned with the opening in the nebulizer block. This enables the assembly of the Impact bead adjustment screw.

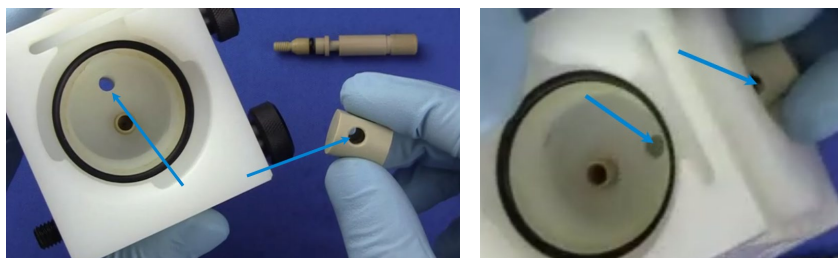


Figure 59. Align holes to enable assembly with the Impact bead adjustment screw

- 2 Replace the Impact bead adjustment screw by gently pushing past the O-ring.

Maintenance



Figure 60. Insert the impact bead adjustment screw

- 3 Turn the Impact bead adjustment screw until it engages with Impact bead adjustment block.
- 4 Tighten the Trapping screw clockwise as far as possible. Do not over-tighten.



Figure 61. Tighten the trapping screw – gas inlet side of the nebulizer

- 5 Loosen the Trapping screw one turn.
This enables free movement of the Impact bead adjustment screw.
- 6 Tighten the Clamping screw clockwise until it gently touches the Impact bead adjustment block. Do not over-tighten.



Figure 62. Tighten the clamping screw – side of nebulizer

- 7 Loosen the Clamping screw one-eighth of a turn.
This restricts the horizontal movement of the Impact bead adjustment block.



Figure 63. Clamp screw touching the impact bead adjustment block

Ensure free movement of the Impact bead adjustment block which should move without forcing the Impact bead adjustment screw.



Figure 64. Range of movement of the impact bead adjustment block

- 8** Turn the Impact bead adjustment screw fully clockwise. (Right hand image in Figure 64 above).

From this position the Impact bead adjustment block can only be adjusted away from the venturi.

Before you install a new impact bead or replace the nebulizer:

- Always inspect the impact bead for pitting, cracks or breaks.
The efficiency of the impact bead can be degraded by surface cracks, pitting and the accumulation of solid material. The reduction in bead efficiency can cause lower absorbance readings and noisy signals.
 - Ensure the Impact bead adjustment screw operates properly.
- 9** If necessary, remove the old impact bead as described in the previous section.

NOTE

The impact bead adjuster block must be positioned as far as possible to the edge closest to the impact bead adjuster. Refer to the right-hand image in Figure 64 above.

This will avoid breaking the impact bead after installation.

Maintenance

- 10 Carefully insert the tail of the Impact bead through the nebulizer block hole into the Impact bead adjustment block.

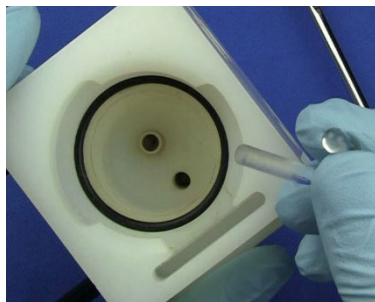


Figure 65. Insert tail of impact bead into the nebulizer block.

- 11 Push the impact bead in as far as possible so that the bead is in the throat of the venturi.
- 12 Center the impact bead in the venturi throat so that there is a small even gap of approximately 0.5 mm between the impact bead and the venturi throat.



Figure 66. Even gap of around 0.5mm between the impact bead and the venturi throat

- 13 Carefully tighten the Impact bead locking screw.
Check that the impact bead will not rotate under gentle pressure.
- 14 Turn the impact bead adjustment screw counter-clockwise until the block appears midway in the cutout in the nebulizer block.

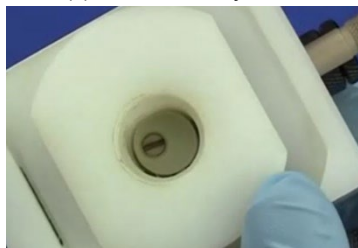


Figure 67. Centered impact adjustment block

- 15** Re-assemble the nebulizer and spray chamber components and install the complete assembly in the sample compartment - see Chapter 3 – Page 40.

The location of the Impact bead is close to the maximum sensitivity position for easily atomized elements.

To confirm, aspirate a 5 ppm copper standard and monitor the absorbance. Gradually turn the Impact bead adjustment screw first clockwise, then anticlockwise until the maximum signal is achieved.

Improve the signal-to-noise ratio by reducing the sensitivity setting. To do this, turn the impact bead adjuster clockwise until the signal is about 40% of the maximum signal.

Spray chamber

The spray chamber should be cleaned as outlined in Table 8.

Table 8. Spray chamber cleaning schedule

Test material	Frequency	Cleaning method
Organic or hazardous	After each analysis	Disassemble and clean.
Inorganic, non-hazardous or aqueous solutions	End of each day	Aspirate 0.1% detergent solution for 10 minutes
Inorganic, non-hazardous or aqueous solutions	Every 1-4 weeks depending on use	Disassemble and clean

At the end of every working day by aspirating at least 50 mL of the appropriate solvent through the system.

Disassemble the spray chamber

To disassemble the spray chamber:

- 1 Remove the nebulizer block/spray chamber assembly from the sample compartment.
- 2 Separate the spray chamber from the nebulizer block – see Page 91.
- 3 Twist and pull the pressure relief bung from the rear of the spray chamber. Inspect the O-ring and replace if cracked or deformed.
- 4 Insert a blunt, inert object inserted through the front of the spray chamber.
- 5 Push on the mixing paddles boss to remove the part.

Maintenance

To clean the spray chamber:

NOTE

Use a high vacuum grease to sparingly lubricate O-rings, immediately wiping off any excess with a clean cloth.

Do not use hydrocarbon or silicon grease.

O-ring replacement kits are available from Agilent (refer to the Spare Parts chapter for ordering information).

-
- 1 Remove the nebulizer/spray chamber assembly from the sample compartment. Disassemble the spray chamber and liquid trap.
 - 2 Flush the spray chamber components with an appropriate solvent and then wash them thoroughly in laboratory detergent and warm water.
 - 3 Use a bottlebrush to clean the inside of the spray chamber.
 - 4 If necessary, clean the mixing paddles in an ultrasonic bath.
 - 5 Rinse all parts thoroughly with distilled water and dry thoroughly.
 - 6 Inspect all O-rings and replace any that are cracked or deformed. Replacement is easier if the O-rings are lubricated with distilled water.
 - 7 Re-assemble the spray chamber according to the instructions below.

To assemble the spray chamber

NOTE

Touching or otherwise contaminating any part of the mixing paddles will degrade the performance of the spray chamber. For cleaning instructions, see 'To clean the spray chamber' – Page 106.

-
- 1 Position the front (boss end) of the mixing paddles in the opening at the rear (bung end) of the spray chamber, being careful not to contaminate them.
 - 2 Align the mixing paddles so that the edges of a pair of blades are vertical when installed.

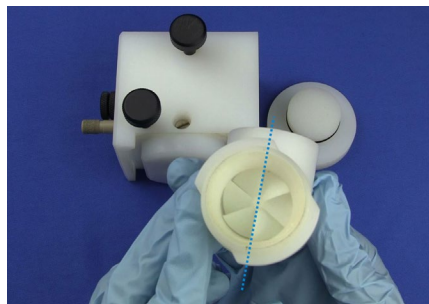


Figure 68. Mixing paddles with opening at the bottom of the spray chamber

NOTE

Liquid can condense on horizontal surfaces and results in random %RSD “spikes”. Vertical blade alignment ensures the other paddles blades are also not horizontal, minimizing the resultant noise.

- 3** Slide the paddles into the spray chamber until the rear paddle is about to enter the spray chamber.
- 4** Check that the paddles are still positioned with the vertical blade edges and then carefully push the back paddle into the spray chamber using a blunt, inert and clean object (for example, a plastic stirring rod) until the front paddle is against the shoulder at the front of the spray chamber.
- 5** Lubricate the O-ring of the pressure relief bung with distilled water.
- 6** Insert the pressure relief bung into the rear of the spray chamber.

With a gentle twisting motion, firmly press the pressure relief bung as far as it will go into the spray chamber.

Assemble the spray chamber and nebulizer block:

Ensure the parts in both the spray chamber and nebulizer block have been properly installed and aligned. The requirements are outlined in the previous two sections.

- 1 Place the O-ring in the recess at the front of the nebulizer block as shown below.

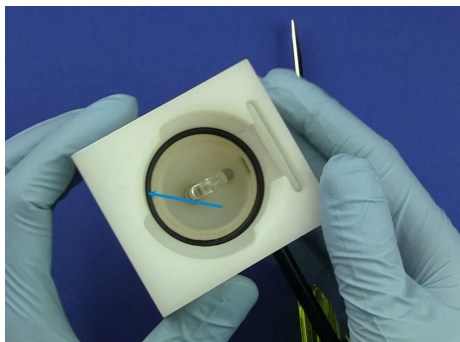


Figure 69. O-ring in the recess of the nebulizer block

- 2 Holding the nebulizer block in one hand, insert the front end (bayonet mount) of the spray chamber into the rear end of the nebulizer block. Twist the spray chamber $\frac{1}{4}$ turn clockwise to lock the bayonet mount.

Attach the drain tube/float/liquid trap to the nebulizer block

This applies to products with serial numbers 0110xxxx and later.

- 1 Hold the nebulizer block/spray chamber assembly upside down so that the float falls clear of the hand and screw the new drain tube into the nebulizer block - see Figure 72 on page 109.

WARNING



Explosion Hazard.

Failure to install the drain tube will cause violent and loud explosions to occur in both the liquid trap and drain vessel when the flame is lit. The explosion may be severe enough to cause death, personal injury or damage to the instrument or laboratory.



Figure 70. Screw the drain tube into the nebulizer block

- 2 Lower the drain tube into the trap, ensuring that the float drops into the space between the two ribs that are opposite the two vent nipples – see Figure 71 below.

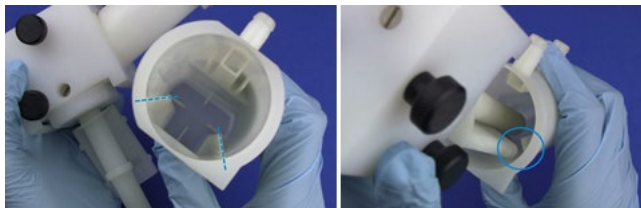


Figure 71. Lower the drain tube and float into the trap

- 3 Push the trap home and then twist it 1/4 turn clockwise to lock it into position - see Figure 74 below.

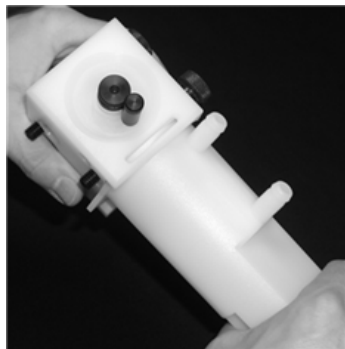


Figure 72. Lock the trap into position

Maintenance

WARNING

Explosion Hazard



Failure to fill the liquid trap will cause violent and loud explosions to occur in both the liquid trap and drain vessel when the flame is lit. The explosion may be severe enough to cause death, personal injury or damage to the instrument or laboratory.

NEVER attempt to light a flame unless the liquid trap is filled with solvent so that the float just starts to move.



Figure 73. Complete nebulizer/spray chamber assembly

The complete spray chamber/nebulizer assembly (refer Figure 73 above) is ready to be installed in the sample compartment of the instrument.

Installing the spray chamber/nebulizer assembly

CAUTION

Always check the assembly before installing the spray chamber in the sample compartment. Especially check the correct positioning of the mixing.

To install the spray chamber/nebulizer assembly:

- 1 Place the spray chamber/nebulizer assembly in position in the sample compartment so that the fuel and oxidant outlet nipples on the burner adjuster. (see Figure 74 below) are engaged in the fuel and oxidant ports in the side of the nebulizer block.



Figure 74. Fuel and oxidant outlet nipples

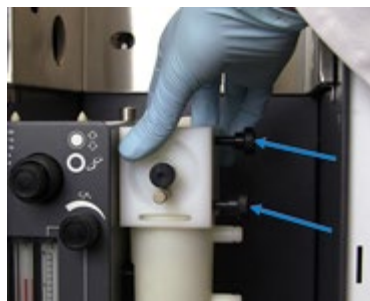


Figure 75. Retaining screws

- 2 Push the assembly firmly sideways. Tighten the knurled retaining screws alternately, one turn each at a time, so that the assembly is pulled up evenly into place (see Figure 75 above). Tighten both screws firmly with the fingers only.
- 3 Place a suitable wide-necked waste vessel in a convenient position at floor level.
 - The waste vessel must be made of a non-glass material which will not shatter in the event of a flashback or fire.
 - Locate your waste vessel in an open well-ventilated area where you can see it.
 - Never locate the waste vessel in a confined space.
 - Use small stable vessels and empty them frequently.

WARNING**Fire Hazard.**

Do not accumulate large volumes of flammable liquid.

Dispose of flammable liquids in accordance with local regulations.

- 4 Attach a convenient length of 9 mm ID tubing to the lower outlet of the liquid trap.

Maintenance

- 5 Lead the free end of this tubing to your waste vessel.
 - Ensure the tubing is relatively straight and free of kinks, sharp bends or upward slopes so waste liquid can drain freely into the waste vessel.
 - The free end of the tubing must remain above the level of the liquid in the vessel.

WARNING



Fire Hazard, Explosion Hazard.

Failure to connect the drain tubing correctly can cause a flashback or an explosion in the waste vessel.



Ensure that the tubing is configured as described in the previous step.

Use vessels made of a material that will not shatter in the event of a flashback.

Do not use glass vessels.

- 6 If you are using materials that produce hazardous or flammable vapors, attach a convenient length of 9 mm ID tubing to the upper outlet of the liquid trap.
- 7 Lead the free end of the tubing out from the sample compartment, parallel to the drain tubing.
 - The tubing must slope downwards for its entire length to prevent it becoming blocked should liquid drain from the vapor vent.
 - Lead the tube to an active exhaust system.
 - Do not lead the vapor tubing to the waste vessel.

WARNING



Fire Hazard, Explosion Hazard.

Failure to connect the vapor vent tubing correctly can cause a flashback or explosion. Ensure that the tubing is configured as described in the previous step.



Burners

Burners should be cleaned after each day of use, particularly if the solutions being analyzed have a high solids content or a rich nitrous oxide-acetylene flame has been used.

If they are not cleaned regularly, hard deposits that will be difficult to remove can build up. The use of organic solvents with a nitrous oxide-acetylene flame increases the possibility of carbon build up.

Inspect the O-ring on the burner, replace immediately if damaged or worn.

TIP

It is impossible to determine with the naked eye if the inside of a burner is clean. Even a low power inspection lens will fail to show low levels of contamination. The best way is to use a binocular microscope which enables you to look down inside the burner slot.

WARNING**Explosion Hazard.**

The burner is correctly assembled, sealed and pressure tested during manufacture. To prevent gas leaks and flashbacks, the burner must NEVER be disassembled, modified or mistreated.

ALWAYS use the correct procedure to clean the burner slot, since the use of a burner with an oversize slot can cause a flashback.

Failure to follow correct procedures can cause death, injury or property damage.

To install a burner:

- 1 Check the O-ring on the burner. It must be supple and free from nicks or cracks to ensure that the burner makes a gas tight seal with the spray chamber. Replace a damaged or worn O-ring immediately.
- 2 Position the burner in the sample compartment, with the slot along the optical path and the plate with the warning towards the front of the instrument.

Maintenance

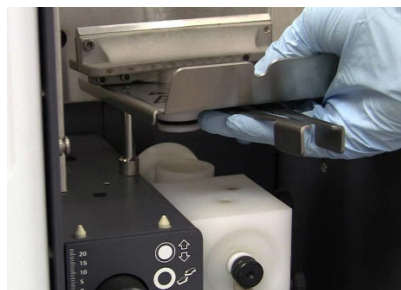
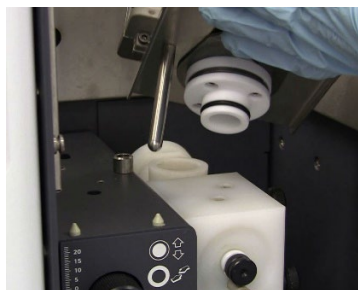


Figure 76. Alignment pin and burner neck O-
Figure 77. Lower burner into position ring

- 3 Position the alignment pin (see Figure 76 above) above the guide in the burner adjuster.
- 4 Lower the burner so that the interlock key enters the guide in the burner adjuster and the burner neck is inserted into the top of the spray chamber. See Figure 77 above.
- 5 With a slight twisting motion, push the burner down as far as it will go.
 - Do not touch the burner slot.
- 6 Ensure the neck and O-ring are fully inserted into the spray chamber.
- 7 Position the burner in the sample compartment with the slot in the burner aligned along the optical path, (Figure 78 below) and the plate with the warning towards the front of the instrument.
- 8 Squeeze the prongs of the rotation handle together to move the handle left or right to rotate the burner for alignment (Figure 79 below).

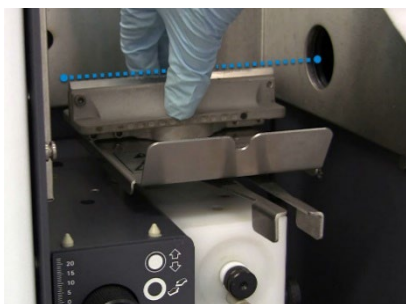


Figure 78. Push the burner down, avoid touching the burner slot, align the slot along the optical path.



Figure 79. Squeeze Rotation Handle to rotate the burner

After installation, Align the burner -see Page 74.

WARNING**Fire Hazard, Explosion Hazard.**

Leaking gas mixtures may explode or ignite and cause injury and damage.



Do not damage the O-ring when fitting the burner.

Clean the burner

WARNING**Hot Surface, Chemical Hazard.**

The burner and flame compartment become extremely hot during instrument operation. Allow the burner and flame compartment to cool for at least twenty-five minutes before attempting to remove the burner.



Nitric and hydrochloric acids are very corrosive and can cause severe burns when they contact the skin. Preparation of acid soak solutions and any soaking should be done under an extraction fume hood. It is essential that appropriate protective clothing be worn at all times when handling acids. If acid contacts the skin, wash off with copious amounts of water and seek medical attention immediately.

To clean the burner:

- 1 Extinguish the flame.
- 2 Allow surfaces around the flame compartment to cool.
- 3 Remove the flame shield and sample compartment front panel from the instrument.
- 4 To remove the burner, gently twist the burner assembly as you lift it straight up out of the spray chamber.
- 5 Using an Agilent burner cleaning and alignment card, gently scrape off any excessive carbon build up in the burner slot.

Maintenance

NOTE

Soaking the burner in nitric acid and using an ultrasonic bath effectively removes soft chemical deposits inside the slot. The removal of hard carbon deposits requires more significant physical effort.

- 6 Prepare a bath of 5% v/v nitric acid solution in a container that is designed to hold an inverted burner.
- 7 Submerge only the slotted part of the burner in acid.

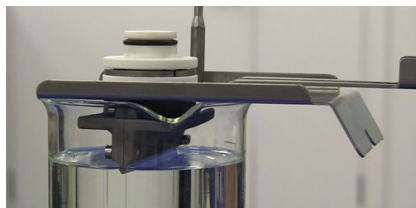


Figure 80. Burner slot soaking in acid

- 8 Leave to stand overnight to soften the deposits.
- 9 Rinse with distilled water and place the inverted burner in an ultrasonic bath containing dilute laboratory detergent for approximately 10 minutes.
- 10 Rinse with distilled water and allow to dry.

TIP

To speed up drying, dry the burner with a paper or lint-free towel, and then use compressed oil-free air or inert gas.

NOTE

In the following the steps, ensure the card does not tear or disintegrate. This may clog the burner slot causing uneven atomization at the flame, affecting sensitivity. Use several cleaning cards if necessary.

CAUTION

Never slide the card sideways within the burner slot.

- 11 Insert a clean Agilent burner cleaning and alignment card into the burner slot.
- 12 Use a dropper to pour a small amount of metal polishing fluid on each side of the burner cleaning card just above the burner slot.

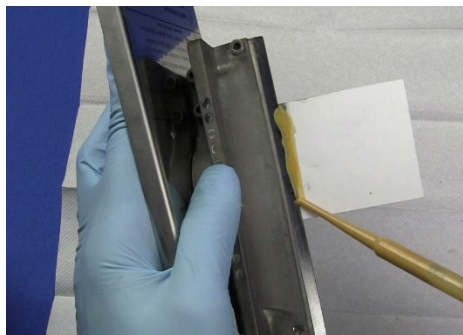


Figure 81. Apply polish to the burner cleaning card

- 13** Slide the card in and out of the slot (perpendicular to the top) to remove any deposits that may be present.
 - a** Ensure the ends of the slot are also cleaned.



Figure 82. Slide the burner cleaning card in and out of the slot

NOTE

If the deposits are baked on hard, their removal may require some effort, and several cards may be required.

CAUTION

Under no circumstances should a metal strip or implement be used on or in the burner slot as it will scratch the surface and promote the rapid buildup of carbon or other solids.

- 14** Place some metal polishing liquid on the card and polish the **outer** surfaces along the top of the burner slot.

Maintenance



Figure 83. Polish the OUTER surface of the burner slot

- 15 Rinse the burner with distilled water.
- 16 Wash and rinse the burner with a diluted 10% v/v solution of an alkali detergent to remove any traces of the metal polishing liquid.
- 17 Dry the burner. Use compressed air to fasten the drying process.

NOTE

The top of the burner slot should be clean and shiny to indicate that all deposits have been cleaned off.

- 18 If necessary, repeat some or all the previous steps until the burner slot is clean.

NOTE

For applications with high deposits, additional burner cleaning procedures are recommended. Contact your Agilent customer support representative for recommendations.

Table 9. Maximum allowable burner slot width

	N ₂ O	Air
Mark VIA	0.47 mm (0.0185 in.)	
Mark 7	0.46 mm (0.0181 in.)	0.54 mm (0.021 in.)

Burners cannot be refurbished and must be replaced when the maximum slot widths are exceeded.

Gas supplies

Change gas cylinders according to the instructions supplied by the manufacturer. Refer also to the Safety and Hazards chapter at the start of this manual, see Page 15.

When any gas cylinder is changed, inspect all gas hoses. Hoses showing any signs of damage or deterioration must be replaced immediately. This should be done by a trained Agilent representative.

Test all connections for leaks using a commercial leak testing solution, a mild detergent, or an electronic leak detector.

When changing gas cylinders:

- 1 Check all gas supply pipes and hoses for leaks.
- 2 Test the operation of all regulators.
- 3 Ensure that shut-off valves are functioning correctly.

Lamps

CAUTION

Care must be taken when handling lamps. Always hold the lamp by its base. Do not touch the quartz window at the end of the lamp. Touching the glass envelope will reduce the lamps' efficiency.

To install a hollow cathode lamp:

- 1 Open the lamp compartment door.
- 2 Ensure that the required lamp position is turned off.

This is controlled from the Instrument Parameters page of the LCD display. To turn the lamp off, set the 'Active current' field to zero (for more information, see 'Instrument Parameters page', on Page 56).

Maintenance

- 3 Hold the lamp by its base and align the lamp base so that the ridge on the guide pin (see Figure 84 below) is matched with the notch in the socket (see Figure 85 below).

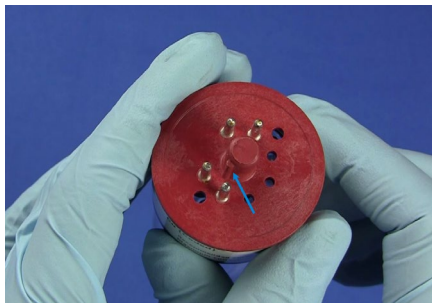


Figure 84. Lamp base guide pin ridge



Figure 85. Lamp socket – notch highlighted

- 4 Press and hold the white button located at the underside of the socket (see Figure 86 on page Figure 86).
- 5 Slowly release the button as you push the lamp fully into position.

CAUTION

Hold the lamp by its base. Do not touch the quartz window at the end of the lamp.

Removing hollow cathode lamps

To remove a hollow cathode lamp:

- 1 Open the lamp compartment door.
- 2 Ensure that the lamp is turned off.

This is controlled from the Instrument Parameters page of the LCD display. Set the 'Active current' field to zero (for more information, see 'Instrument Parameters page', Page 56).

WARNING

Hot Surface.



The lamp will become very hot during operation and cause severe burns if touched. Allow time for the lamp to cool.

Always wear protective gloves when handling a hot lamp.

- 3 Hold the lamp by its base.

- 4 Press and hold the white button on the underside of the socket to release the lamp.

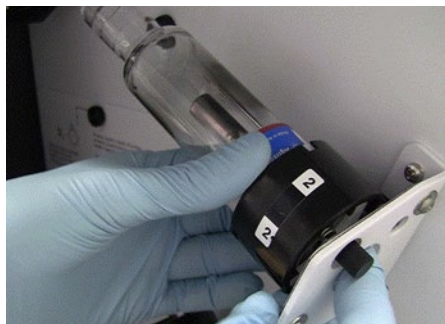


Figure 86. Hollow cathode lamp mount

- 5 Gently remove the lamp from the socket and release the white button.

D₂ lamp

The D₂ lamp will need changing approximately every 1000 operating hours. If you need to replace D₂ lamps more frequently you should contact Agilent trained and certified personnel for advice.

WARNING



Hot Surface.

The D₂ lamp and its cover become very hot during operation.

To prevent skin burns, allow the assembly to cool down before removing.

CAUTION

Do not handle the D₂ lamp or hollow cathode lamp optical surfaces with bare hands.

To change a D₂ lamp:

- 1 Turn the instrument off and unplug the instrument from the power source.
- 2 Allow time for the D₂ lamp and housing to cool.
- 3 Remove the hollow cathode lamp in position 1 if fitted.
- 4 Undo the thumbscrew at the top of the D₂ lamp compartment.
- 5 Lift the lamp assembly up and out from the lamp compartment using the two lamp alignment screws.

Maintenance

- 6 Disconnect the white plug at the end of the D₂ lamp lead and remove the D₂ lamp assembly from the instrument.
- 7 Loosen the Philips head screw in the lamp holder assembly (1 in Figure 87) and slide out the lamp.
- 8 Slide a new D₂ lamp through the clamp until the notch in the backing plate (2) is aligned with the lamp aperture (3) shown in Figure 87.

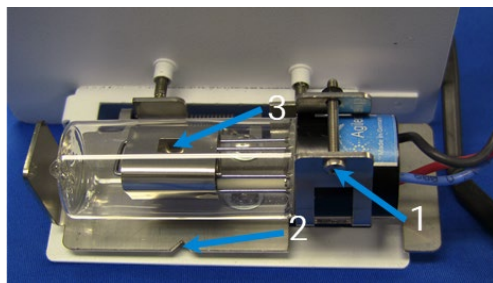


Figure 87. D2 lamp alignment (top view)

- 9 Rotate the lamp so that the face with the aperture (4) is parallel with the top edge of the bracket (5) – see Figure 88 below.

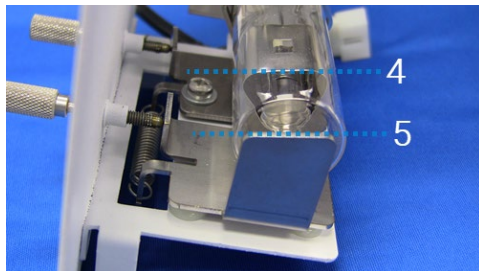


Figure 88. D2 lamp alignment (end view)

- 10 Tighten the Philips head screw to secure the lamp in the holder.
- 11 Reconnect the D₂ lamp plug.
- 12 Replace the lamp assembly in the lamp compartment.
- 13 Replace the thumbscrew at the top of the D₂ lamp compartment.

NOTE

After installation, a new D₂ lamp must be aligned. Refer to Optimization for instructions on how to align lamps – see Page 72.

Fuses

The spectrometer contains two operator accessible fuses located at the back of the instrument. To replace a fuse, you must disconnect the instrument from the power supply and replace the blown fuse with the type and rating indicated in the Specifications section – Page 31.

The fuses have a code marked on the cap (e.g., T 2.5A H250V). This refers to the fuse characteristic ('T'= time lag, 'F'= fast acting), the current rating (in amperes), the breaking capacity ('H'= heavy, 'L'= low) and the voltage rating (in volts). This code must correspond to the code appearing next to the fuse-holders.

WARNING

Shock Hazard, Fire Hazard, Hot Surface.



To prevent reduced safety protection or unwanted fusing, **ALWAYS** ensure that the code on the fuse cap matches the printed information shown adjacent to the fuse-holders.

To check a fuse:

- 1 Disconnect the instrument from the mains power supply.
- 2 Remove the fuse holder, located to the right of the mains input connection as shown in Figure 89 below.
- 3 Place the tip of a flat head screw driver into the slot (1).

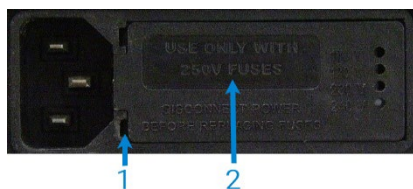


Figure 89. Fuse cover
1. Removal slot 2. Fuse holder

Maintenance

- 4 Gently lever the fuse holder (2) from the instrument.
- 5 Check that the fuses are the correct type and are not damaged. Replace if necessary.
- 6 Replace the fuse holder in the instrument and reconnect the instrument to the mains power supply.

NOTE

If a fuse blows repeatedly, it may indicate other problems with the instrument. Contact your local Agilent sales office and arrange a service call.

7

Spare Parts

Sample lists	125
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Covers/doors	128
Fuses	128
Miscellaneous	129

This chapter contains information for your Agilent 55B AA spare parts. Unless otherwise specified, only Agilent supplied parts should be used. See the Agilent website for part number and ordering information at www.agilent.com.

Sample lists

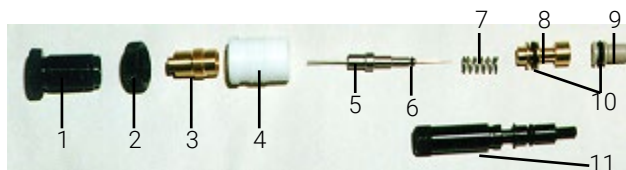


Figure 90. Nebulizer components

- 1 Thimble, capillary adjuster
- 2 Locking ring
- 3 Clamp bush guide
- 4 Ertalyte bush bearing
- 5 Capillary assembly
- 6 O-ring 1/32 in. ID x 3/32 in. OD x 1/32 in. nitrile
- 7 Spring
- 8 Capillary guide
- 9 Venturi
- 10 O-ring 3/16 in. ID x 5/16 in. OD x 1/16 in. nitrile
O-ring 3/16 in. ID x 5/16 in. OD x 1/16 in. organics
- 11 Impact bead adjuster screw

Spare Parts

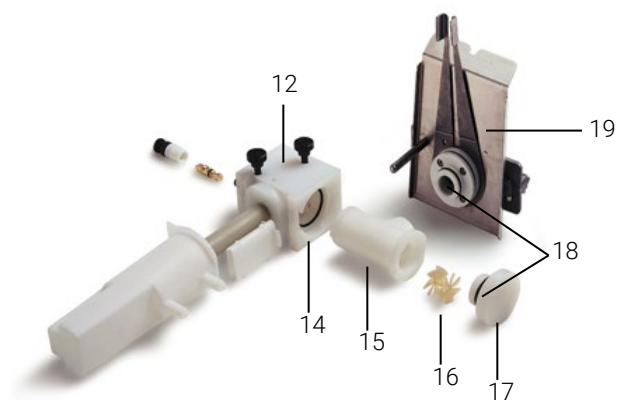


Figure 91. Spray chamber/nebulizer assembly with Mark 7 burner. (Older style drain tube, float and liquid trap shown. See following picture for new drain tube/float/liquid trap.)

- 12 Kit nebulizer block Mark 7
- 13 Kit, liquid trap system—integrated (comprising float, liquid trap and drain tube)
- 14 O-ring, nitrile
O-ring, organics
- 15 Spray chamber, molding fluorinated
- 16 Mixing paddles
- 17 Pressure relief bung
- 18 O-ring, pressure relief bung, nitrile
O-ring, pressure relief bung, organics
- 19 Air/acetylene burner, Mark 7
N₂O/acetylene burner, Mark 7

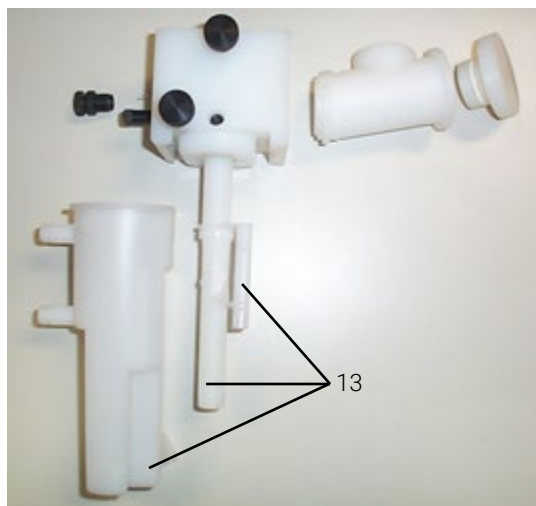


Figure 92. Spray chamber/nebulizer assembly showing new drain tube/float/liquid trap

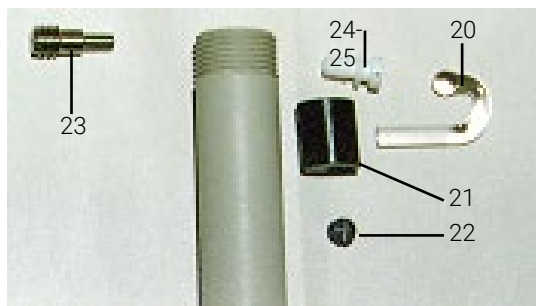


Figure 93. Various components from the nebulizer block

- 20 Impact bead – glass or PTFE (packet of 5)
- 21 Clamp bead adjuster
- 22 Impact bead locking screw (PEEK)
- 23 Trapping screw (stainless steel)
- 24 Plug grey PEEK
- 25 O-ring 3/16 in. ID

Spare Parts

Other

Universal Mark 7 spray chamber assembly complete. Includes nebulizer block and spray chamber, spray chamber bung and liquid trap.

Aqueous o-ring kit

Organics o-ring kit

Capillary tubing, standard

Capillary tubing, Hi-vac

Nebulizer tool—capillary extraction

Nebulizer tool—venture extraction

Burner cleaning and alignment strip (packet of 100)

Nebulizer cleaning wire

Covers/doors

Flame shield

Sample compartment front panel (lower)

Flame shield window only

Fuses

T2.5 A H 250 V

Spare Parts

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8 Troubleshooting/Errors

Common problems	131
Error messages	134

Common problems

The following troubleshooting chart provides solutions to some common problems you may encounter during routine AA analysis.

Please note that this list is not exhaustive.

Table 10. Common faults

Fault	Check
1 Oxidant pressure low	a) Check gas supply pressure.
2 Burner fault	a) Check the burner type is correct for the selected flame type and it is correctly installed.
3 Liquid trap fault	a) Check liquid trap is full and float is free to move. b) Check the operation of the liquid trap interlock in the burner adjuster.
4 Spray chamber bung fault	a) Check pressure relief bung is correctly installed in the spray chamber. b) Check operation of pressure relief bung interlock.
5 Lack of control of gas flows	a) Check as for 1a). b) Check for blockages in gas supply lines.
6 High oxidant flow	a) Check O-rings in nebulizer block. b) Check nebulizer is correctly assembled and adjusted. c) Check for leaks.
7 Flame sensed with no flame	a) Too much light in the sample compartment. Ensure chimney and flame shields are fitted. Turn off nearby lights. b) Flame sensor faulty.

Troubleshooting/Errors

8	Igniter flame too short	a) Low acetylene pressure, check supply. b) Check that igniter solenoid operates correctly. c) Check for blockage or carbon in igniter capillary.
9	Igniter will not light	a) Check that there is a spark to light the tongue of the flame. If not, call Agilent trained and certified personnel. b) Check operation of Ignite push-button. c) Check as for fault 8.
10	Flame will not light	a) Oxidant flow may be too high - see fault 5. There may not be an error message. b) Check as for faults 1, 2, 3, 8. c) Check operation of Ignite push-button. d) If "flame sensed" error message displayed, check as for fault 7. e) Check flame shield interlock.
11	Poor light throughput	a) Check lamp alignment. b) Clean lamp windows.
12	High gain readings	a) Check PMT supply. b) Change HC lamp. c) Check as for fault 11.
13	Low sensitivity with flame	a) Check nebulizer uptake rate. b) Check nebulizer capillary is not blocked. c) Check impact bead alignment. d) Check for chemical interference. e) Check fuel to oxidant ratio is correct. f) Check burner is clean and clear of deposits. g) Check solutions have not expired. h) Check wavelength selection and calibration.

<p>14 Unstable signals with flame</p>	<p>a) Check burner alignment. b) Check flame shield and front panel installed in sample compartment. c) Check purity of gas supplies. No moisture or oil in the air supply. d) Check nebulizer, burner, spray chamber and paddles are clean. e) Check without mixing paddles installed. No change in signal stability indicates the issue is occurring before the spray chamber. f) Check nebulizer is correctly set up and correct plastic capillary (wide or narrow bore) is used. g) Check the impact bead adjustment. h) Check that the acetylene bottle pressure is >700 kPa and that acetone has not been allowed to enter the instrument.</p>
<p>15 When using SIPS, the high standards curve excessively</p>	<p>a) Check nebulizer uptake rate is faster than fastest SIPS pump rate.</p>
<p>16 No peak found</p>	<p>a) Check as for fault 11. b) Check correct wavelength selected. c) For flame emission, check sufficient concentration of solution and the high standard is used to optimize. d) Check that the correct hollow cathode lamp is being used.</p>
<p>17 D₂ lamp energy low</p>	<p>a) Check as for fault 11. b) Reduce hollow cathode lamp current. c) Increase slit width. d) Change D₂ lamp.</p>
<p>18 D₂ lamp energy high</p>	<p>a) Check hollow cathode lamp energy. b) Reduce slit width.</p>
<p>19 Signal not increasing and excessive curvature</p>	<p>a) Check standard solutions (particularly high standard). b) Select a different wavelength. c) Reduce lamp current. d) Reduce slit width. e) Operate within the working range (0.1 to 0.8 Abs).</p>

Error messages

This section lists all of the errors messages, and includes some possible reasons for the occurrence.

Error messages appear on the bottom line of the display and remain there for five seconds. They consist of a single letter severity code, followed by a four-digit number and a short message.

For example:

W1234 Check HC lamp selection.

The severity codes are listed below.

Table 11. Severity Codes

Code	Meaning	Explanation
I	Information	This type of message provides information only, such as prompting the user to perform some action.
W	Warning.	Provides information about a system difficulty, or potential difficulty, which the user should be aware of.
E	Error	A message relating to a condition that will terminate the current operation. The system will continue to run, but the operation will have failed.

0804 LIMS port error

A communication error or problem has been identified with the LIMS (Laboratory Information Management System) via the serial RS-232C port.

2851 SIPS comms error

There has been a communication problem with the SIPS sample introduction pump unit. Check all cables to this unit are connected securely.

3800 EEPROM storage error

A problem has been identified with the User Method storage device. The method parameters have failed to store or were retrieved with error and so ignored.

5004 Signals not increasing

The calibration has failed. The signal readings of the calibration standard solutions are not increasing in the expected order. The instrument cannot calibrate using this sequence of standards.

Suggested actions:

Move the cursor to the last standard measured and press Enter to clear the field and remove the invalid concentration from the calibration. If more than one standard has been measured the calibration should now be valid, indicated by the symbol **Cal** in the bottom right of the display screen area.

Alternatively, remeasure the previous standard (ensuring you are using the correct standard) by returning the cursor to the appropriate field and press **Read**.

5005 Slope test failure

The calibration has failed. The slope of the calibration curve changes too much between successive standard signal readings.

5006 Calibration fit failure

The calibration has failed. A calibration curve could not be fitted to the measured standards. Effective calibration is indicated by the symbol **Cal** in the bottom right of the display screen area.

5008 Reslope signal out of range

Reslope calibration has failed. The reslope standard signal reading differed from the original value by more than 25%).

6000 Check HC lamp selection

This message is for information only. It is intended as a prompt for the operator to ensure that the lamp selection lever is in the position consistent with both the selected method element and lamp current.

9159 EEPROM checksum zero w/

The instrument will take several minutes to start up but will have no effect on operation. If the error persists, a service call may be required.

9160 EEPROM checksum mono correction

The monochromator wavelength may not be accurate. Because of this the instrument may fail to find a particular Hollow Cathode lamp Resonance Line, resulting in **No Peak Detected** errors. If the error persists, a service call may be required.

9307 RBC/Mains frequency below 48Hz.

The instrument detected a power mains supply fault: Low mains frequency.

Possible causes include:

- Power supply failure.
- Faulty RBC (Rotating Beam Combiner) on double beam instruments only.
- Faulty Mains Supply to the instrument.

If the error persists, a service call may be required.

9308 RBC/Mains frequency

The instrument detected a power mains supply fault: Mains frequency out of range.

Possible causes include:

- Power supply failure.
- Faulty RBC (Rotating Beam Combiner) on double beam instruments only.
- Faulty Mains Supply to the instrument.

If the error persists, a service call may be required.

9309 RBC/Mains frequency above 62Hz

The instrument detected a power mains supply fault: High mains frequency.

Possible causes include:

- Power supply failure.
- Faulty RBC (Rotating Beam Combiner) on double beam instruments only.
- Faulty Mains Supply to the instrument.

If the error persists, a service call may be required.

9310 Optical RBC frequency

Instrument fault. The instrument detected a problem with the RBC. A service call may be required.

Optical sensor or associated circuitry may be faulty.

9311 Instrument fault +12V PSU

Instrument Fault. The instrument detected a problem with the +12 volt supply. A service call may be required.

9312 Instrument fault -12V PSU

Instrument Fault. The instrument detected a problem with the -12 volt supply. A service call may be required.

9313 Instrument fault 5V PSU

Instrument Fault. The instrument detected a problem with the 5 volt supply. A service call may be required.

9316 Wavelength out of range

The Instrument is reporting that the current method has specified a wavelength that is out of range for the Photomultiplier Tube (PMT).

Possible causes include:

- A method wavelength suitable for a Wide Range PMT was specified but the instrument is currently fitted with a Short Range PMT. Use a valid wavelength.

Currently available PMTs:

- Wide range: up to 900 nm.

9317 No peak: low HC lamp energy

Low hollow cathode lamp energy.

The instrument Gain had been raised to its highest value but the hollow cathode lamp signal was too low.

Possible causes include:

- Hollow cathode lamp misaligned.

Troubleshooting/Errors

- The light path is obstructed in the sample compartment.
- Faulty hollow cathode lamp.
- Faulty PMT or related circuits.

9318 No peak: high HC lamp energy

High hollow cathode lamp energy.

The instrument Gain had been set to its lowest value but the hollow cathode lamp signal was too high.

Possible causes include:

- Hollow cathode lamp misaligned.
- Faulty hollow cathode lamp.
- Faulty PMT or related circuits.

9319 No peak: low BC lamp energy

Low Background Corrector (D₂) lamp energy.

Possible causes include:

- D₂ lamp misaligned.
- The light path has been obstructed.
- Faulty D₂ lamp or circuits.

9320 No peak: high BC lamp energy

High Background Corrector (D₂) lamp energy.

Possible causes include:

- D₂ lamp misaligned.
- Faulty D₂ lamp.
- Faulty PMT or related circuits.

9321 No peak: low HC lamp energy

Low hollow cathode lamp energy. Monochromator reference wavelength not found.

The instrument Gain had been raised to its highest value but the hollow cathode lamp signal was too low.

Possible causes include:

- Hollow cathode lamp misaligned.
- The light path is obstructed.
- Faulty hollow cathode lamp.

Suggested action:

Check that the optical light path is not obstructed in the sample compartment. Exit then re-enter the Optimize page/window. This will cause the instrument to retry the peaking.

9322 No peak: high HC lamp energy

High hollow cathode lamp energy. Monochromator reference wavelength not found. The instrument Gain had been set to its lowest value but the hollow cathode lamp signal was too high.

Possible causes include:

- Hollow cathode lamp misaligned.
- Faulty hollow cathode lamp.
- Faulty EHT circuit.

9323 Low emission: no peak

The instrument failed to peak at the selected wavelength. Low Emission energy at the selected wavelength. The instrument Gain had been raised to its highest value but the signal was too low.

Possible causes include:

- Flame not lit, or correctly aligned.
- Wrong wavelength selected.
- The standard solution currently being aspirated has low concentration.
- *Emission Setup* has not been performed.
- Faulty PMT or related circuits.

9324 High emission: no peak

The instrument failed to peak at the selected emission wavelength. The instrument Gain had been set to its lowest value but the signal was too high.

Possible causes include:

Troubleshooting/Errors

- *Emission Setup* has not been performed.
- Wrong wavelength selected.
- Faulty PMT or related circuit.
- The standard solution currently being aspirated has high concentration.

9329 No peak detected

The instrument failed to peak at the selected wavelength. The instrument Gain had been raised to its highest value, but the hollow cathode lamp signal was too low. Incorrect or noisy readings will result if the instrument is not optimized again.

Possible causes include:

- Wrong hollow cathode lamp for this element.
- Hollow cathode lamp misaligned, or the light path is blocked.
- Faulty hollow cathode lamp.
- The lamp current has been set to zero or too low.
- The lamp position in the method is incorrect.

Suggested action:

Check that the optical light path is not obstructed in the sample compartment. Exit then re-enter the Optimize page/window. This will cause the instrument to retry the peaking.

9330 No peak detected

The instrument failed to peak at the selected wavelength. Another peak may be near this wavelength. Incorrect or noisy readings will result if the instrument is not optimized again.

Possible causes include:

- Wrong hollow cathode lamp.
- Wrong wavelength or method selected.
- Slit width (SBW) selection incorrect.
- The lamp position in the method is incorrect.

A wavelength correction fault. The error **9621 Instrument Fault: Wl correction** occurred prior to this error.

Suggested action:

Check that the optical light path is not obstructed in the sample compartment. Correct any of the possible causes listed above. Exit, then re-enter the Optimize page/window. This will cause the instrument to retry the peaking.

9337 Instrument fault 310V PSU

Instrument Fault. The instrument detected a problem with the 310 volt supply on the control board. A service call may be required.

9339 Mono resetting. Please wait

This message is for information only. It can only occur when the instrument is just switched on. If this message appears then the instrument will take several minutes to find its wavelength reference. The instrument will function correctly after the delay. If this message continues to occur and the startup delay is a problem, then a service call will be required.

9415 Instrument fault: lamp code

The instrument could not recognize the lamp code for one of the installed hollow cathode lamps.

Possible causes include:

- One of the installed hollow cathode lamps is faulty.
- A electronic fault in the instrument's lamp recognition circuitry.

9422 Instrument fault: lamp current

The instrument could not detect any lamp current for the selected hollow cathode lamp.

Possible causes include:

- No hollow cathode lamp is installed in the selected lamp position.
- Faulty hollow cathode lamp.
- Faulty lamp supply circuits.

9514 Instrument signal saturation

The instrument signal reached saturation on one or more signal values. Either the signal went below -0.3 Abs or went too high (above 130%) in a Flame Emission method.

Suggested action:

Re-optimize the instrument (or, for a Flame Emission method, redo *Emission Setup*).

9517 Low HC lamp energy

Low hollow cathode lamp energy.

The instrument Gain had been raised to its highest value but the hollow cathode lamp signal was too low.

Possible causes include:

- Hollow cathode lamp misaligned.
- The light path is obstructed.
- Faulty hollow cathode lamp.
- The lamp position in the method is incorrect.

9518 High HC lamp energy

High hollow cathode lamp energy.

The instrument Gain had been set to its lowest value but the hollow cathode lamp signal was too high.

Possible causes include:

- Hollow cathode lamp misaligned.
- Faulty hollow cathode lamp.
- Faulty PMT or circuits.
- The lamp was in the wrong position when the instrument was optimized.

9524 Low BC lamp energy

Low Background Corrector (D₂) lamp energy.

Possible causes include:

- D₂ lamp misaligned.
- The wavelength selected is inappropriate for D₂ background correction.
- The light path has been obstructed in the sample compartment.
- Faulty D₂ lamp.

9525 High BC lamp energy

High Background Corrector (D₂) lamp energy.

Possible causes include:

- D₂ lamp misaligned.
- The hollow cathode lamp energy is too low.
- Faulty D₂ lamp.
- Faulty EHT circuit.

9527 Instrument fault: EHT failed

Instrument Fault. The instrument detected a problem with the EHT supply. Restart the instrument. If the problem persists a service call may be required.

9528 Background lamp failed

Instrument Fault. The instrument detected a problem with the Background Corrector (D₂) lamp. No lamp current has been detected and the D₂ lamp has been presumed faulty. Change the D₂ lamp as described in Chapter 6. If the problem persists a service call may be required.

9529 Background lamp interlock

Instrument Fault. The instrument detected a problem with the Background Corrector (D₂) lamp. The safety interlock switch suggests that the D₂ lamp is not safely installed in the instrument. The lamp will not light in this position. Remove and re-install the D₂ lamp bracket as described in Chapter 6. If the problem persists a service call may be required.

9530 Instrument fault: signal diagnostics

Instrument Fault. The instrument detected a problem with the signal electronics on the control board. Restart the instrument. If the problem persists a service call will be required.

9531 No lamp current detected

The instrument could not detect any lamp current for the selected Hollow Cathode lamp and has aborted the peaking. Rectify the problem and optimize the instrument.

Possible causes include:

- No hollow cathode lamp in the selected lamp position.
- Faulty hollow cathode lamp.

9602 Instrument fault: mono datum

Instrument Fault. The instrument detected a problem with the monochromator. Restart the instrument. If the problem persists a service call will be required.

9911 No burner fitted

An attempt was made to ignite the flame without any burner installed in the sample compartment.

9912 N₂O burner not fitted

Wrong burner fitted. Change to N₂O-acetylene burner.

Possible causes include:

Wrong burner fitted. The burner fitted is an air-acetylene type, but an N₂O-acetylene flame is requested.

Burner detection circuitry failure.

9914 No gas control unit

There is no flame control unit fitted to this instrument. An attempt was made to load a flame method.

9915 Flame shield open

The flame shield was detected open or removed when the gas-box attempted to ignite the flame.

9916 Fault: gas pressure sensor

The oxidant pressure in the reservoir is detected when the Ignite button is pressed but the gas control is shutdown. This is either a control board electronics or pressure sensor fault.

9917 No oxidant gas pressure

The oxidant pressure in the reservoir failed to reach the required operating pressure during the ignition sequence or has dropped unexpectedly while a flame was operating. This error may also be returned while running in Air Only mode.

Possible causes include:

- Air or N₂O gas cylinders empty or turned off.
- Faulty pressure sensor.
- Faulty gas supply regulation.

9920 Fault: flame detected

When the Ignite button was pressed the flame detector sensed that a flame is present prior to the ignition sequence. For safety reasons this error will prevent the flame from being lit. Possible causes include:

- Excessive light in the sample compartment.
- A faulty flame sensor.
- The flame override service switch is in the Flame Sense position.

9921 Flame out detected

After the flame ignition, the flame had been detected by the flame sensor but it is now no longer detected. This is unexpected as no Flame Off command has been given. Possible causes include:

- The acetylene gas flow may be too low to sustain a flame.
- The oxidant flow may be too high to sustain a flame.
- The detector may be faulty.

9922 Flame shutdown: gas type

This is an internal system error. If a flame was present this error will cause the flame to be shut down for safety reasons.

9923 Flame ignition timeout

The flame failed to ignite within 7 seconds during an ignition sequence. Wait at least 4 seconds for the gases to disperse, then try again. Possible causes include:

- The acetylene gas cylinder is empty or turned off.
- The fuel flow is too low (only on the MANUAL gas box).
- The igniter failed.

9934 Flame shutdown: host offline

The Flame has been extinguished due to an internal fault or power failure.

9937 Flame pressure relief bung

The pressure relief bung is detected as not fitted either while a flame is attempting to ignite or while a flame is active. The pressure relief bung is located at the rear of the nebulizer assembly in the sample compartment.

9938 Liquid trap not ready

The liquid trap connected to the bottom of the nebulizer assembly has insufficient liquid. Possible causes include:

- Insufficient liquid in the trap.
- The float in the liquid trap has jammed or been inserted incorrectly.
- Faulty sensor.

9xxx GPIB fault

This is an internal instrument fault. If a SIPS unit is attached, check that the cable is correctly installed. Restart the instrument. If the error persists contact your Agilent field service engineer.

9xxx Instrument error

This is an internal instrument error and should not occur.

Record the actual error number (e.g. 9903) Restart the instrument. If the error persists, contact Agilent trained and certified personnel.

9xxx SpectrAA error

This is an internal instrument error and should not occur.

Record the actual error number (e.g. 9903). Restart the instrument. If the error persists, contact Agilent trained and certified personnel.

In This Book

The manual describes the following:

- Introduction
- Site Requirements and Specifications
- Software and Hardware Installation
- Operation of Hardware and Software
- Maintenance Procedures
- Instrument Cleaning
- Spare Parts
- Troubleshooting

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