

Agilent 240/280 Series AA (including Zeeman)

User's Guide



Notices

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Agilent Technologies Australia [M] Pty Ltd

679 Springvale Road

Mulgrave, Victoria 3170, Australia

www.agilent.com

Errata statement

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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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General

Your Agilent AA instrument and accessories have been carefully designed so that 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 that 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 also referenced in your Agilent accessory manuals. Thoroughly read these safety practices before using your instrument or accessories.

Observe all relevant safety practices always.

Safety Practices and Hazards

The safety practices described below are provided to help you operate the instrument safely. Read each safety topic thoroughly before attempting to operate the instrument and *always* operate the spectrometer in accordance with these safety practices. Only trained operators should use this instrument.

Verifying safe state

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

To ensure continued safety of the 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 instruments 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 circuits, devices and components 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 must only be opened by Agilent field service engineers. Consult the manuals or product labels supplied with your PC, monitor, printer/plotter, water-cooling system, and vacuum pump (where required) 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. This could seriously damage the instrument and any attached ancillary equipment.

Always use a 3-wire outlet with ground connection that is adequately rated for the load. The installation must comply with local and national safety regulations. Use only an Agilent supplied power cord. Replace the power cord only with a cord equivalent to the one specified in the AA Site Preparation Guide.

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 is connected.

WARNING



Electrical shock hazard To avoid electric shock this equipment must be disconnected from the mains supply before servicing.

Heat, vapors and fumes

Heat, vapors and fumes generated by flame, furnace and vapor generation methods can be hazardous to personnel.

Heat, vapors and fumes must be extracted from the instrument by 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. Locate the system outlet such that the exhaust cannot re-enter the building through any door, window, air conditioning inlet, or other ventilator. Construct the system in accordance with local codes and regulations for ventilation.

The exhaust system must provide an exhaust ventilation rate of at least 6 cubic meters per minute (200 cfm). 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 an indicator close to the instrument to indicate whether the exhaust fan is on or off. *Always* switch on the exhaust fan *before* lighting the flame.

Use fireproof ducting that is in accordance with your local fire prevention regulations. Locate the ducting away from fire alarms, sprinkler heads, and other

Safety Practices and Hazards

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 working.

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

Compressed gases and cylinders

All compressed gases (other than air) can create a hazard if they leak into the atmosphere. Even small leaks in gas supply systems can be dangerous. Any leak (except that of air) can create an explosion hazard, a fire hazard, or result in an oxygen–deficient atmosphere. Such hazards can cause death, serious injury, asphyxiation, anesthetic effects, and serious damage to equipment and property.

Cylinders must be stored and handled strictly in accordance with local safety codes and regulations. Cylinders must be used and stored only in a vertical position. Secure all cylinders to an immovable structure or a properly constructed cylinder stand. The area in which cylinders are stored must be adequately ventilated to prevent toxic or explosive accumulations. Move cylinders 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 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.

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 components of the gas control system and create a potentially hazardous situation.

Use only approved regulators and hose connectors.

Never attempt to refill cylinders.

Remember that 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 cylinders.

Gas hoses and connections

Even small leaks in gas supply systems can be dangerous. Any 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.

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.

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

Flames, hollow cathode lamps, and deuterium lamps emit hazardous ultraviolet radiation. 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.

Other

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

NOTE

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

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. A warning icon indicates the beginning of the warning text:

WARNING

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



Corrosive liquids









Toxic material





Heavy weight (danger to feet)







Heavy weight (danger to hands)



Noxious gases







Hot surface



Sharp object

٦

Read all warnings and cautions carefully and observe them always.

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 for your information.

I	Mains power on
0	Mains power off
ф	Fuse
\sim	Single phase alternating current
	Flame off
No	Flame on
-	Indicates lamp present
	Disconnect all power plugs.
	Caution, disconnect all supplies, risk of electric shock

Color coding

The various indicator lights appearing on Agilent instruments and associated accessories are color coded to represent the status of the instrument or accessory.

Safety Practices and Hazards

- A green light indicates that the instrument is in normal/standby mode.
- An orange light indicates that a potential hazard is present.
- A blue light indicates that operator intervention is required.
- A red light warns of danger or an emergency.

CE compliance

Your Agilent AA instrument has been designed to comply with the requirements of the Electromagnetic Compatibility (EMC) Directive and the Low Voltage (electrical safety) Directive (commonly referred to as the LVD) of the European Union. Agilent has confirmed that each product complies with the relevant Directives by testing a prototype against the prescribed EN (European Norm) standards.

Proof that a product complies with these directives is indicated by:

- The CE Marking appearing on the rear of the product, and
- The documentation package that accompanies the product containing a copy of the Declaration of Conformity. The Declaration of Conformity is the legal declaration by Agilent that the product complies with the directives listed above, and shows the EN standards to which the product was tested to demonstrate compliance.

Electromagnetic compatibility

EN55011/CISPR11

Group 1 ISM equipment: group 1 contains all ISM equipment in which there is intentionally generated and/or used conductively coupled radio- frequency energy that is necessary for the internal functioning of the equipment itself.

Class A equipment is equipment suitable for use in all establishments other than domestic and those directly connected to a low voltage power supply network that supplies buildings used for domestic purposes.

This device complies with the requirements of CISPR11, Group 1, Class A as radiation professional equipment. Therefore, there may be potential difficulties in

ensuring electromagnetic compatibility in other environments, due to conducted as well as radiated disturbances.

Operation is subject to the following two conditions:

- 1 This device may not cause harmful interference.
- 2 This device must accept any interference received, including interference that may cause undesired operation.

If this equipment does cause harmful interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try one or more of the following measures:

- **1** Relocate the radio or antenna.
- **3** Move the device away from the radio or television.
- **4** Plug the device into a different electrical outlet, so that the device and the radio or television are on separate electrical circuits.
- 5 Make sure that all peripheral devices are also certified.
- **6** Make sure that appropriate cables are used to connect the device to peripheral equipment.

Consult your equipment dealer, Agilent Technologies, or an experienced technician for assistance.

Changes or modifications not expressly approved by Agilent Technologies could void the user's authority to operate the equipment.

ICES/NMB-001

This ISM device complies with Canadian ICES-001.

Cet appareil ISM est conforme à la norme NMB-001 du Canada.

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. This can result in death, or severe personal injury or burns.

Safety Practices and Hazards

Remember always 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, run parallel to the drain tubing, and led to an active exhaust system. Do NOT lead the vapor tubing 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. Metal vessels will corrode and it is difficult to determine the level of liquid in them. 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 and rinse the waste vessel.
- When your analytical program has been completed, or at the end of the working day, always empty, clean, and refill 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 the flame will not operate unless all the safety interlocks are satisfied. Do not attempt to bypass these safety interlocks.



Figure 1. System setup diagram

Compressed gases and cylinders for flame operation

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

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

Never use any gas except acetylene as the fuel gas.

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.

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.

Safety Practices and Hazards

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.

If the pressure in the acetylene cylinder is allowed to fall below 700 kPa (100 psig), or the consumption is greater than 1/7 of the cylinder contents per hour, acetone may be carried over from the cylinder and into the spectrometer. Acetone in the spectrometer can damage seals, O-rings and hoses, degrade analytical performance and precipitate flashbacks.

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)
- 'Crack' the cylinder before use by gently opening the valve to check for any drops or spray of acetone. Any cylinder showing acetone should be returned to the supplier for replacement.

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.

Refer also to your local regulations governing the use of acetylene.

Nitrous oxide

The decompression of high-pressure N_2O 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 that can cause death, serious injury to personnel and damage to equipment and property.

Whenever you handle burners, remember that the burner may be very hot. Always use protective gloves to handle burners. Note the burner will remain hot for some time after the flame has been turned off.

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 causes a flashback.

If the width of the burner slot exceeds 0.47 mm (0.0185 in) for a nitrousoxide/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 causes 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 'Maintenance' section.
- 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, causing a flashback.
- Always turn off the flame 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 that 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 from the spray chamber while the flame is on, and do not use a mechanical device (for example, a wire) to clean the capillary of a nebulizer while a flame is operating. Always extinguish the flame before removing the nebulizer from the spray chamber.

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

Liquid trap

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

The liquid trap interlock is incorporated to minimize the possibility of attempting to operate the instrument with an empty trap or with the drain tube missing. 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. 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.

A length of tubing must be connected to the drain outlet on the trap and led to a suitable waste vessel. The free end of the tubing 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 length of tubing 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 tubing 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, and the sample compartment front panel in place. Keep your hands out of the sample compartment while a flame is burning.

When operating a flame system make sure that the chimney is in place with the cutaway skirt (if present) to the back. Before you touch the instrument chimney, turn off the flame and allow the chimney to cool. 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 burner and flame compartment to cool before attempting to access the burner or sample compartment components.

When you change burners, remember that the burner may be very hot. Always use protective gloves when removing a burner from the instrument.

Perchloric acid

Aspiration of perchloric acid and perchlorates into a nitrous oxide-acetylene flame can create an explosion hazard that 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.

Safety Practices and Hazards

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. For more details, refer to the SpectrAA software Help.

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 and the SpectrAA software Help for recommended maintenance procedures to prevent flashbacks.

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.

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.

- 2 The width of the burner slot must not exceed the maximum design specification [Mark VIA: 0.47 mm (0.0185 in) for N₂O; Mark 7 0.46 mm (0.0181 in) for N₂O; or 0.54 mm (0.021 in) for air]. 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.
- 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.
- **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 N₂O 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.

Safety Practices and Hazards

- 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

- Immediately press the red Flame-off button to turn off gas flows to the instrument.
- Turn off the gas supply at the regulator.
- Turn off power to the instrument.
- Inspect the sample introduction components including the burner, spray chamber and nebulizer. Clean or replace as necessary.
 - Check the 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.
- Clean up any spills in the sample introduction area or on the instrument.

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.

Furnace and Zeeman operation

Gases

The graphite tube atomizer gas supply system is designed for use with inert gases and air.

Never use pure hydrogen with the graphite tube atomizer since this could result in leakage and potentially explosive accumulation of hydrogen. You may, however, use a proprietary, prepackaged mixture of 95% argon (or nitrogen) and 5% hydrogen. *Never* attempt to create your own mixture of hydrogen and an inert gas for use with the GTA system.

Heat hazards

A hot furnace atomizer can present heat hazards that can result in severe burns to personnel. Never put your hands in the sample compartment while you are operating your furnace.

Allow the furnace atomizer to cool before removing it from the sample compartment.

Vapors and fumes

Never place your head over the graphite tube atomizer while it is operating. This could cause inhalation of hazardous or toxic fumes, or your skin and eyes could be injured by corrosive vapors or fumes.

The chimney or the optional exhaust accessory must be in place for furnace operation to ensure that toxic vapors and heat are exhausted.

UV radiation

When viewing the sample during the drying stage, use only a rear coated mirror (as supplied) or the Tube-CAM option. *Never* look directly at the furnace during either the ash or atomize stages.

Magnetic field (Zeeman only)

The magnet produces a variable magnetic field of up to 0.8 Tesla peak at mains frequency in the workhead during the read stage.

To avoid interference with heart pacemakers or magnetic storage media, keep them at least 300 mm from the magnet.

Safety Practices and Hazards

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The Agilent 200 series AA spectrometers are controlled by Agilent's innovative AA worksheet software. The Agilent series AA includes the following instruments:

This documentation is appropriate for the 240/240FS AA, 240Z AA, 280 AA, and 280Z AA instruments.

The software is based on a spreadsheet that mimics an analyst's workbook and combines flame, furnace, Zeeman, and vapor operation in one integrated package.

The SpectrAA Base, PRO and CFR software versions run under Windows 10 64-bit operating systems, and provides the following:

- Innovative, easy to use interface
- Dedicated function keys for critical functions
- Extensive Help including audio and video demonstrations of how to set up your instrument
- "Tool Tips" state the allowed range for each field
- Comprehensive error system
- The capacity to simultaneously operate one flame and one furnace (deuterium or Zeeman) spectrometer simultaneously from one computer (requires SpectrAA PRO version software)
- Features to assist users to achieve compliance with US FDA 21 CFR Part 11 requirements (optional CFR version only)

Introduction

Instrument Overview

Flame instruments front view



Where:

1. Flame shield	7. Flame-on button
2. Burner adjustment handles	8. Instrument status LED
3. Burner height adjustment	9. Power switch
4. Nebulizer and flow adjustment	10. Spray chamber and liquid trap assembly
	(behind the grey cover)
5. Impact bead adjustment	11. Lamp compartment (Eight lamps are shown
6. Flame-off button	here. A four lamp compartment is also available.)

Flame instruments back view



Where:

1. Accessories serial port, such as the GTA	7. C ₂ H ₂ gas line
Viewing/Fume Extraction accessory	
2. USB camera cable port	8. Air gas line
3. IEEE 488 cable port	9. N ₂ O gas line
4. Power cable	10. Air purge port
5. Fuse and power setting housing	11. SIPS pump option port
6. UltrAA lamp option ports	

Introduction

Zeeman instruments front view



Where:

1. GTA 120 Graphite Tube Atomizer	7. Zeeman AA instrument status LED and power switch
2. Zeeman workhead	8. PSD 120 waste port
3. PSD 120 sample tray	9. PSD 120 rinse tube
4. PSD 120 Programmable Sample Dispenser	10. PSD 120 probe
5. Rinse solution	11. Lamp compartment (Eight lamps are
6. GTA 120 instrument status LED and power	shown here. A four lamp compartment
switch	is also available.)

See the User's Guides supplied with the GTA 120 and PSD 120 for more information about those accessories.

Zeeman instrument back view



Where:

8. GTA 120 power cable
9. GTA 120 fuses
10. Water in
11. Water out
12. Alternate gas inlet, typically nitrogen
or air
13. Normal gas inlet, typically argon
14. GTA 120 IEEE 488 cable port

Introduction

Furnace instruments front view



Where:

1. GTA 120 Graphite Tube Atomizer	8. Flame-on button
2. Workhead	9. Instrument status LED
3. PSD 120 sample tray	10. AA instrument power switch
4. PSD 120 Programmable Sample	11. PSD 120 waste port
Dispenser	
5. Rinse solution	12. PSD 120 rinse tube
6. GTA 120 instrument status LED and	13. PSD 120 probe
power switch	
7. Flame-off button	14. Lamp compartment (Four lamps are
	shown here. An eight lamp compartment
	is also available.)

See the User's Guides supplied with the GTA 120 and PSD 120 for more information about those accessories.



Furnace instruments back view

Where:

1. Accessories serial port, such as the GTA	10. SIPS pump option port
Viewing/Fume Extraction Accessory	
2. USB camera cable port	11. Air purge port
3. AA instrument IEEE 488 cable port	12. GTA 120 power cable
4. AA instrument power cable	13. GTA 120 fuses
5. AA instrument fuse and power setting housing	14. Water in
6. UltrAA lamp option ports	15. Water out
7. C ₂ H ₂ gas line	16. Alternate gas inlet, typically nitrogen or air
8. Air gas line	17. Normal gas inlet, typically argon
9. N ₂ O gas line	18. GTA 120 IEEE 488 cable port

Installation requirements

Before you receive your instrument, you are given an Agilent AA Site Preparation Guide; which describes the environmental and operating requirements of the Agilent AA system. Prepare your laboratory according to those instructions before the Agilent AA can be installed. Keep the Site Preparation Guide for future reference. If you have misplaced your copy, you can obtain a replacement from your local Agilent office or the website at www.agilent.com.

Introduction

Agilent AA documentation

This manual covers the installation of the Agilent SpectrAA software for 240/280 series AA instruments. Instructions for installing, operating, and maintaining the instruments and components are included in the SpectrAA Help (see Page 51).

Operating instructions for the Sample Introduction Pump System (SIPS) and other AA accessories are given in the manuals accompanying the accessories or in the SpectrAA Help

Conventions

The following conventions have been used throughout this manual:

- Single quotes ('') indicate a selection that you can make from several choices, such as radio buttons, checkboxes, and software items.
- ALL CAPITALS indicates text that you must type in from the keyboard (for example, type SETUP at the prompt).

Electrical Connections

Rear of instrument:

- IEEE 488
- Accessory, 9-way female D-range type
- Accessory, MCA, 6-way DIN type (AA280FS and AA280Z only)
- USB port, optional
- UltrAA lamp connections: Burndy circular 6-way, optional (actual number depends on the model and option selected)

WARNING Shock Hazard



High voltage- To maintain safety, only the UltrAA lamp power supplies should be used at these connections.

Some pins will become energized (300 VDC) when an UltrAA lamp is in a matching socket. To avoid death or electric shock, NEVER touch any exposed pins.

Certaines broches du culot des lampes sont sous tension (300 VCC) à la mise en place d'une UltrAA lampe. Pour éviter l'électrocution ou la mort, ne jamais toucher les broches sous tension.

Front of instrument:

• Zeeman workhead CPC 14 way connection behind the left cover (Zeeman only).





To prevent connector damage, switch OFF the instrument before inserting the plug and *always* turn the locknut fully clockwise to the detent position. To maintain safety, only the Zeeman workhead connector should be used at this connection.

Lamp bay:

• Deuterium lamp: Molex 3-way connection, in lamp compartment (behind lamp panel in lamp compartment on 240/280 series AA instruments).

WARNING Shock Hazard

To maintain safety, only the deuterium lamp should be used at these connections.



• Hollow cathode lamps:

- Four lamp capacity on the 240 series AA
- Eight lamp capacity on the 280 series AA



Shock Hazard

High voltage – To maintain safety, only hollow cathode lamps should be used at these connections.

Fuses

CAUTION

For continued protection against fire hazard replace fuse only with same type and rating.

Introduction

Non-Zeeman

240 series AA

T2.5 A H250 V, IEC 127 sheet 5, 5 x 20 mm (100–120 & 220–240 VAC) **280FS AA** T4 A H250 V, IEC 127 sheet 5, 5 x 20 mm (100–120 & 220–240 VAC) **Zeeman**

240Z/280Z AA

15 A long delayed-action circuit breaker with a thermal cutout. T3.15 A H 250 V, IEC 127 sheet 5, 5 x 20 mm (208–240 VAC) T3.15 mA L 250 V, IEC 127 sheet 2, 5 x 20 mm (208–240 VAC) T1 A L 250 V, IEC 127 sheet 2, 5 x 20 mm (208–240 VAC) T6-3 AL 250 V, IEC 60127-2 sheet 3, 5 x 20 T800 mA L 250 V, IEC 127 sheet 2, 5 x 20 mm (208–240 VAC)

NOTE

For safety reasons, any other internal fuse or circuit breaker is not operator accessible and should be replaced only by Agilent field service engineers.

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

Gas supplies

	C ₂ H ₂	Air	N ₂ O	Air purge
	Instrument grade	Must be clean and	Instrument grade	
	>99.5% pure	dry. (Air filter to be	>99.5% pure	
		used if required)		
Allowed range	65–100 kPa	245–455 kPa	245–455 kPa	245–455 kPa
	(9.5–14.5psi)	(35–65 psi)	(35–65 psi)	(35–65 psi)
Recommended	75 kPa (11 psi)	350 kPa (50 psi)	350 kPa (50 psi)	
Typical flow rate	0-10 (L/min)	13.5-20 (L/min)	11–16 (L/min)	10 (L/min)
Connection	1.8 m (6 ft) length	1.8 m (6 ft) length	1.8 m (6 ft) length	Barb to take
	of 6.3 mm (1/4 in)	of 6.3 mm (1/4 in)	of 6.3 mm (1/4 in)	6.3 mm (1/4 in)
	ID red hose with	ID black hose with	ID blue hose with	hose
	9/16 in-18UNF L/H	9/16 in-18UNF	3/4 in-16UNF	
	thread fitting#	fitting#	fitting#	
# .				

[#]Adaptors are available

Other gas connections

Sample compartment:	Push-on Air/N ₂ O connector for burner
	Push-on C_2H_2 connector for burner

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This chapter describes how to prepare your personal computer (PC) and printer for use with the Agilent AA system and install the SpectrAA software on your PC.

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

PC requirements

If you are supplying your own PC for use with an Agilent AA instrument, the recommended configurations of that PC are listed in the AA Site Preparation Guide.

Installation procedure overview

There are four software installation scenarios:

- SpectrAA Base
- SpectrAA PRO (or upgrade from Base to PRO)
- SpectrAA Base to PRO upgrade
- SpectrAA CFR

For installation instructions, refer to the appropriate instructions supplied with your software:

- SpectrAA Software installation instructions
- SpectrAA Software Installation Instructions for 21 CFR Part 11 Environments

GPIB communications

A USB-GPIB converter must be installed in your computer to interface the computer and the Agilent AA. See below for information on installing the USB-GPIB converter.

NOTE Although the Agilent field service engineer will install the GPIB communications device for you during the installation process, you may need to configure the driver yourself at some later stage, for example if you change the PC.

Installing the USB-GPIB converter

To install a National Instruments USB-GPIB converter:

- NOTE Ensure that the SpectrAA software has been installed.
 - **1** Turn off the computer.
 - **2** Connect one end of the converter to the instrument and the other end to the USB connection on the back of the computer.
 - **3** Turn on the computer.

Connecting and configuring an Agilent AA spectrometer DUO system

Setting up for DUO operation and connecting the IEEE cables

NOTE This function is not available in the SpectrAA Base version. Install SpectrAA Base and PRO or CFR software and the USB-GPIB converter first NOTE before performing this procedure. To set up and connect an AA Duo system: 1 Open SpectrAA. 2 On the 'Index' page, click the instrument link on the left side of the page. The 'SpectrAA Instrument Configuration' dialog box opens. Ensure 'Duo system' is not ticked and that an instrument is selected in the 3 'Instrument #1' tab. 4 Restart SpectrAA to save the configuration changes. 5 Connect both instruments to the PC using IEEE cables as shown in the following diagram. Do not turn on the instruments. Or other Instrument #1 Instrument #2 optional Flame PC Zeeman GTA accessory Instrument Instrument Figure 2. Example of a Flame and Zeeman instrument connected with a GTA 6 Return to the 'Index' page of SpectrAA and reopen the 'SpectrAA Instrument Configuration' dialog box. Select the Duo System checkbox. 7 8 Select the appropriate models in the drop-down box for Instruments #1 and #2. If you are running a Duo system that includes an Agilent 55B AA instrument, the 55B NOTE AA MUST be entered as Instrument #1 in the SpectrAA Instrument Configuration dialog box.

9 Click **OK** to close the dialog box.

- **10** Restart SpectrAA to save the configuration settings.
- **11** Go to the next section 'Configuring the IEEE addresses for Agilent AA-DUO operation'.

Configuring the IEEE addresses for Agilent AA-DUO operation

If you are running an Agilent AA-DUO system, you must reconfigure one of the instruments so they have different IEEE addresses. Agilent 55B AA instruments should have a default IEEE address of 1. Agilent 240FS AA, 240Z AA, 280FS AA and 280Z AA instruments should have a default IEEE address of 9. Because 55B AA's must be Instrument #1 in the configuration dialog, Instrument #2 will always have a default IEEE of 9. It is important not to change the IEEE address of a 55B AA as it may cause conflicts with the inbuilt computer.

- 1 Close SpectrAA if it is open.
- 2 Navigate to C:\Program Files (x86)\Agilent\sp100\Run and then double-click DUAL.EXE. Click **OK** to start the program.
- 3 A window appears asking you to turn on the instrument you wish to reconfigure. You must select 'Instrument #2' in the configuration. Switch on Instrument #2 and then click OK. Do not turn on the Instrument #1. A command window is displayed. The process may take about a minute. During this time, you will hear the instrument resetting. Once the process is complete, a message appears in the command window saying 'Addr changed from 9 to 8. Press any key to continue...' If this message does not appear, continue the procedure below. If it does appear, you have completed the setup.
- 4 In the search field type **cmd.exe**. Select cmd.exe from the list that appears.



- **5** Turn on Instrument #1 and Instrument #2, if not on.
- 6 Choose one of the following options:
 - For a Duo system with a 55B AA: Type: CD C:\Program Files (x86)\Agilent\SP100\Run\ and then press Enter.

Type: setaddr.exe -q1:8 and then press Enter.

• For a Duo system without a 55B AA: Type: CD C:\Program Files (x86)\Agilent\SP100\Run\ and then press

Enter.

Type: setaddr.exe -q9:8 and then press Enter.

The process may take about a minute after which a dialog will appear saying that both addresses (1 and 8 or 8 and 9) are 'found'.

7 Restart the computer, turn on both AA instruments, and then start the Agilent SpectrAA software.

The instruments are now ready to be used as a DUO system. Refer to the Help for further information on dual instrument operation.

- NOTEDuo mode will only work if the instrument selected as 'Instrument #1' in the
SpectrAA Instrument Configuration dialog box retains its default IEEE address (that
is, 1 for 55B or 9 for 240FS, 240Z, 280FS or 280Z.
- NOTE During dual instrument operation, do not turn any connected instrument ON or OFF while readings are being taken.
- NOTE You can only operate in Duo mode with one of each accessory type connected. That is, one GTA and one PSD and one SPS 3/SPS 4.
 - For the Base version of SpectrAA software, the check box will show one instrument.
- NOTE For more information about changing the DUO system configuration or to revert back to single instrument use, see the Setup Agilent AA Duo operation instructions in the How to section in the SpectrAA Help.

Configuring the SPS 4 autosampler

To configure the SPS 4 autosampler:

- 1 Shut down the SpectrAA software if it is open.
- 2 Follow the instructions provided in the SPS 4 Autosampler User's Guide to set up the hardware.
- **3** Plug one end of the USB cable into the USB port on the autosampler and the other end into a USB port on the computer.
- **4** Navigate to C:\Program Files (x86)\Agilent\SP100\Run.
- **5** Double-click the IHelper.EXE application.
- 6 Click the Accessories tab.

- 7 Select Agilent SPS 4 Autosampler in the Flame Auto Sampler section if needed.
- 8 Click OK to save the changes.
- **9** Continue to 'Installing the SpectrAA Pro software' if applicable or skip to 'GPIB Communications' on Page 38.

Determining the SPS 4 COM port

When running the Calibration Wizard, you must select the SPS 4 COM port to start communication between the SPS 4 and the PC. The following procedure explains how to determine the COM port.

For information on how to use the Calibration Wizard, see the SPS 4 Autosampler User's Guide or the SpectrAA Help > Accessories >SPS 4 section.

To determine the COM port:

- 1 Right-click on Start and then select Device Manager.
- 2 Expand 'Ports'.
- **3** Find the 'SPS4 USB Interface' and note the assigned COM port (COM5 in the example shown below).



Figure 3. Example of SPS 4 USB interface COM port

Configuring the software to use manual sampling

If you are NOT using any flame autosamplers, set the Sampler Type in the **Accessories** tab of 'SpectrAA Instrument Configuration' dialog box to the SPS 5 Autosampler. The SPS 5 Autosampler uses IEEE communications instead of RS-232 communications. By specifying the flame autosampler type as the SPS 5 Autosampler (even though no autosampler is used), the software will not capture the RS-232 port during start-up or shut down of the SpectrAA software. This will minimize delays during start-up, operation, and shut down of the software.

To define the SPS 5 flame autosampler:

- Activate the IHelper.EXE application (from the installation drive: \Program Files (x86)\Agilent\SP100\Run directory); OR click the model number displayed at the Main Index page of the SpectrAA software. This displays the 'SpectrAA Instrument Configuration' dialog box.
- 2 From the 'SpectrAA Instrument Configuration' dialog box, click the Accessories tab.
- 3 Select Varian SPS5 Autosampler in the Flame Auto Sampler section.
- **4** To complete the installation and configuration, shut down and restart the SpectrAA software, as prompted.

USB to RS-232 interfaces

There are several USB to RS-232 adapters available for use on computers that are not fitted with or not equipped with sufficient RS-232 interfaces. Depending on the instrument configuration, operation of all accessories may require the use of two RS-232 interfaces:

- An RS-232 interface is required for operation with the SPS 3 autosampler
- Another RS-232 interface is required for software control of the ETC 60 Electrothermal Temperature Controller.
- NOTE Other modem and LIMS system may be also connected to the available RS-232 COM ports.

Where there are not enough RS-232 interfaces to support all the devices that use these COM ports, an acceptable alternative is to use a USB to RS-232 adapter.

To use a USB to RS-232 adapter:

- 1 Install the USB-RS-232 device according to the manufacturer's specification.
- 2 Right-click on **Start** and then select **Device Manager**. Locate the COM USB Com port and open **Properties**.
- **3** Select the **Port Settings** tab and open the **Advanced** settings.
- 4 Set the COM PORT number to 1 to 4 using an available number from the dropdown list. If the numbers are already used, you have to set one of the other Com ports to a higher number and then set the one you are going to use to the vacancy created.

SpectrAA software only accepts values from 1 to 4.

- **5** Close all dialog boxes.
- 6 Shut down and restart the computer. You may need to repeat this process if the port was not enabled the first time due to it being used by another device.

Starting the SpectrAA software

To start the SpectrAA software:

- 1 Double-click the SpectrAA icon on the desktop.
- 2 The first time the SpectrAA software is opened a Software Registration dialog appears. Click **Next**.

NOTE Ensure that the user of the SpectrAA software completes the software registration. For further information, refer to the Software Registration Help. 3 Complete all the fields on the 'Customer Details' page. Click Next. NOTE The Product Key is found on the cover of the Agilent SpectrAA software CD case that was delivered with the instrument.

- 4 Complete all the fields on the 'Product Details' page. Click Next.
 - **5** Complete all the fields on the 'Work Environment Details' page.
- 6 Click Register.
- **7** A dialog appears stating 'Your Agilent Software Registration has been successful'.

NOTE If your computer is not connected to the Internet, refer to the Software Registration Help for further information.

8 The application will now open, and you can collect your data.

SpectrAA Release Notes and Software Status Bulletin

Refer to the SpectrAAReleaseNotes.pdf file in the installation directory and the Software Status Bulletin document for further details. The file includes latest release information and important notes including the detail of any known problems and suggested work-arounds.

Installing the drivers for the Tube-CAM Furnace Camera option

For instructions on how to install the Tube CAM drivers, see the "Tube-CAM Furnace Video Option Software Installation Instructions" that came with your accessory.

Printer setup

The printer must be a make and model supported by Microsoft Windows. To install your printer, follow the instructions provided with the printer. For more information, refer to your printer documentation or Microsoft Windows Help.

Troubleshooting

This section contains solutions to some common problems you may encounter during the installation of the SpectrAA software.

The SpectrAA software is not communicating with the instrument.

- Ensure that the computer has been restarted after installing the software and USB-GPIB converter.
- Check the cables and connections.
- Check that the instrument and any associated accessories are turned on.
- Check that you are not running the Simulator software. (Select Help about system information.)

Service installation module

The installation test software is accessed directly from the Administration window. As shown below, this control is *only* activated when the software has

been installed. This software must be installed from the Service folder of the BASE CD.

To do this:

- 1 Insert the SpectrAA Base CD in the computer CD-ROM drive.
- **2** Use Windows Explorer to open the Service folder 'D:\service', where 'D' is the CD-ROM driver letter.
- **3** Locate and run the setup.exe file to install the necessary components and enable the VALIDATE button in the Administration window.

Installation and commissioning tests can be selected and run from the 'Installation' test window that is displayed. This allows easy testing and provides a report of the tests conducted covering power-on self-tests, safety interlocks, and wavelength performance checks.

4 Software Overview

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The Agilent AA system software (SpectrAA) is a powerful interface that provides high levels of spectrometer control and is easy to use. This chapter provides a brief overview of the SpectrAA software to help you familiarize yourself with its various windows. A more detailed description and instructions for use are included in the extensive Help. The optional SpectrAA CFR version software offers you the tools to help you achieve compliance with the US FDA 21 CFR Part 11 requirements. More information on the CFR compliance features of the software is detailed in the Help.

Starting the SpectrAA software

To start the SpectrAA software double-click on the desktop SpectrAA icon (created during the installation of the software).

SpectrAA windows

Index page

When you start the SpectrAA software, the first screen to appear is the introductory screen.

The Index page contains four buttons: Worksheet, Reports, Administration, and Exit.

Software Overview

The function of each button is as follows:

Worksheet	Opens the Worksheet window (see Page 48)
Reports	Opens the Reports window (see Page 49)
Administration	Opens the Administration window (see Page 50)
Exit	Shuts down the SpectrAA software.

Worksheet window

The Worksheet window allows you to develop and edit methods, sequences and labels, and initiate sample analysis.

The worksheet is the fundamental file type for SpectrAA, containing one or more methods, sample labels, sequence information, and analytical results. You can load an existing worksheet or develop a new one.

The Worksheet window consists of four tabbed pages: Filing, Develop, Labels, and Analysis. To access a particular page, click on the appropriate page tab appearing under the menu bar.

The Filing page

Use the Filing page to perform basic worksheet operations such as opening, saving, and closing worksheets, creating a template from a worksheet, and loading worksheets. You can also view information about the worksheet currently open. If you have the CFR version of the software, the Filing page enables you to apply electronic signatures (approval) to the worksheet.

The Develop page

Use the Develop page to add, delete, review and modify methods, modify sequence parameters, change the order of methods and copy methods to the Method Library.

The Labels page

Use the Labels page to set up solution labels, weights, volumes, and dilution factors and to configure SPS or PSD samplers.

The Analysis page

Use the Analysis page to control the AA and initiate analysis. Data is presented in the form of a spreadsheet, with one row per sample. The sample labels are displayed in the left-hand column with concentration results for each element across the remaining columns.

The SpectrAA software enables you to have up to a maximum of 10 worksheets open at the same time. The Worksheet tab at the bottom of the Analysis page enables you to switch between worksheets.

Worksheet menus

When you are	viewing the worksheet, the following menu items are available:
File	This menu allows you to perform filing tasks such as opening, saving and renaming files.
Edit	The Edit menu allows you to copy and paste information, Edit Replicates and Go To certain parts of a worksheet.
View	The View menu enables you to nominate what is displayed on the Analysis page.
Instrument	This menu enables you to perform many Instrument functions such as Optimization, Reslope, and Zero.
Options	The Options menu enables you to alter the displays on the Analysis page.
Window	This menu enables you to quickly move around the software.
Help	This menu enables you to access the Help.

NOTE

Not all menu items are available on all pages of the Worksheet window. If an item or menu is grayed, it is unavailable on that page.

Menu items can be accessed by clicking the desired item with the mouse, or pressing Alt and the active (underlined) letter in the menu name (for example, Alt+E to access the "Edit" menu). This displays a menu list. The options in the menu list can be accessed in the same manner.

Reports window

The Reports window allows you to generate a report for the current worksheet or any of the worksheets saved in the system.

The Reports window consists of four tabbed pages: Worksheet, Select, Settings, and Report. To select a particular page, click on the appropriate page tab appearing under the menu bar.

Worksheet page

Use the Worksheet page to select the worksheet results to include in the report. You can use the "Filter" and "Search" buttons to refine your search.

Select page

Use the Select page to choose methods and solutions to include in the report.

Software Overview

Settings page

Use the Settings page to specify the report style and content.

Report page

Use the Report page to view and print the report, write it to a text file, or export it to a PRN file.

Administration window

The Administration window allows you to: migrate worksheets into the system (such as old worksheets on CD-ROM, etc.); delete worksheets and library methods; and activate and modify password protection of the system. The Administration window also allows you to configure your system.

Working with the SpectrAA software

Menu bar

The Worksheet window contains a menu bar directly beneath the window title bar. This bar displays several menu items. For more information on these menus, see Page 49.

NOTE

When menu items or options appear grayed, they are unavailable for selection.

Toolbar

The Toolbar appears on the Index page and the Worksheet window and provides easy shortcuts to many common functions and other windows.

Dialog boxes

In some cases, selecting a menu option or pressing a button activates a dialog box. This is a box that contains several different input fields relevant to that operation.

You can move the focus from field to field in a dialog box either by clicking each entry item with the mouse or by pressing the **Tab** and arrow keys to move the cursor from field to field. After entering/changing any values in a dialog box, press "OK" to accept the changes and close the dialog.

Help

The SpectrAA software contains extensive Help, which serves as your primary source of information on how to use the software and the instrument. The Help consists of contextual help and multimedia help. The Help also contains an extensive 'How To...' section. This section provides many set by set instructions on how to perform common procedures.

The contextual help is accessed from any window, page, or dialog box in the SpectrAA software by pressing F1 (the Help function key), the 'Help' button (where available), or by selecting 'On-line Help' from the Help menu at the top of any page in the Worksheet window and provides help specific to that screen.

The multimedia help contains hardware-related information and other details to help you set up, operate and maintain your Agilent AA instrument.

For example, the Help includes:

- Installation procedures for the components of the instrument that are customer-installable
- Maintenance procedures for the parts of the spectrometer that are customerserviceable
- Checklists to help you ensure that you have correctly prepared your system for analysis.

Searching for help

You can quickly get help on a specific topic using keywords and the Search facility.

To search for information on a particular subject:

- **1** Open the Help (if it is not already open).
- 2 Select the **Search** tab at the top of the SpectrAA Help window.
- **3** Enter the word(s) you want to search for in the field provided and select **List Topics**.
- **4** A list of all the Help topics associated with the keyword(s) displays in the second list.
- **5** Select the desired topic and click **Display**.

Software Overview

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5 Getting Started

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This chapter describes how to set up the Agilent AA system for analysis and get you started using the instrument.

You should have already:

- Prepared your PC and installed the SpectrAA software described in Chapter 3.
- Become familiar with the SpectrAA user interface as described in Chapter 4.

Instrument setup

Use the following checklist to ensure that you set up your system correctly. You must:

- Connect the components of the system to one another (see 'Connections' on Page 53).
- Connect the equipment to the power supply and check the setting of the two voltage taps where fitted (see 'Power' on Page 54).
- Install the hardware components as per your analytical requirements. Refer to the Help for details.

Connections

Your Agilent AA instrument is supplied with a set of gas hose fittings and a mains cable that suits the common utility standard in the local region. A Country Kit must be ordered with the Agilent AA instrument.

Power

The mains power connection is located at the rear of the instrument. Refer to the next section for instructions on connecting the instrument to the mains power supply.

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Gas hoses

Three rubber hoses are permanently attached to the instrument. Each hose is 1.8 meters long and is color-coded for air (black), nitrous oxide (blue), and acetylene (red). Each is fitted with female fittings suitable for USA standard regulators. Adaptors are supplied for other areas.

NOTE Gas hose connections are not detailed in this section as they are described in your Agilent AA Site Preparation Guide.

Accessories

For details on connecting accessories such as SIPS or UltrAA lamps, refer to the manuals accompanying the accessories.

Power

Requirements

Power requirements are detailed in your Agilent AA Site Preparation Guide. Check the power requirements and read through the section 'Electrical power supplies' in the Site Preparation Guide before connecting the Agilent AA system to the power supply.

Consult the manuals supplied with your printer and PC (if required) for their power requirements.

Connection

Before connecting the instrument to the power supply, ensure that both the spectrometer and the mains power supply are turned off. Check that the two voltage selector switches on the instrument rear panel (if present) are set to the correct mains power supply voltage—refer to the table on the rear panel. The Agilent field service engineer sets the voltage selectors when the instrument is first installed.

To connect the instrument to the power supply, plug the mains power cord into the back of the instrument and the free end of the power cord into the mains power supply, and then switch on the mains power supply.

Moving your instrument



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.

Starting the system

The instrument power switch is a rocker type, located on the front of the instrument on the left-hand side. Switch the power switch to 'l' to start the system. The green indicator light will come on.

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

If you have not already done so, start the SpectrAA software as described on Page 47.

Pre-analysis checklist

The general preparation procedure is as follows:

- 1 Install all the required hardware, including any associated accessories such as SIPS, according to the instructions in the Help (see Page 51) and any accessory manuals.
- 2 Turn on the instrument and any peripheral accessories as described in the previous section.
- **3** Hold a thin single-ply tissue up to the mouth of the extraction hood to ensure that the exhaust system is working. The tissue should be drawn towards the hood.
- 4 Check the gas supplies to make sure that you have enough gas to complete your analysis. Also, check that the acetylene cylinder pressure is above 700 kPa (about 100 psi) to ensure that acetone is not drawn into the instrument.

Getting Started

5 Set the delivery pressure of the gases to the following:

	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, replacing any damaged hoses. Turn on the gas supplies to your instrument and test all hoses and connections for leaks. If a leak is found, repair it immediately.
- **7** Load a worksheet and develop the method(s) as per your analytical requirements.
- **8** Optimize the system.
- 9 Start an Autorun.

NOTE Detailed procedural help for Steps 7 to 9 are provided in the Help. See Page 51 for details of how to find information in the Help.

6 Maintenance and Troubleshooting

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The following is a list of maintenance procedures that must be performed to keep your instrument running safely and with optimum performance. See the 'Maintenance' section in the SpectrAA Help for replacing and cleaning procedures.

To access the Help click **Start > All Apps > SpectrAA > SpectrAA Help** or, with the SpectrAA software, open press F1 on your keyboard.

Do not perform any procedures not listed in this manual or in the SpectrAA Help. Contact your Agilent representative if further information is needed.

The procedures listed below may involve hazardous chemicals and fumes, hot surfaces, and handling fragile lamps and glass. Always follow the correct procedure listed in the SpectrAA Help, adhere to all safety and chemical disposal regulations, and wear the appropriate safety equipment.

WARNING Corrosive liquid



The contents of the liquid trap may be toxic or corrosive. Dispose of the contents in accordance with approved procedures and always wear the appropriate safety equipment.





Some components within the spectrometer may remain hot after turning off the instrument. Before performing any maintenance procedures always allow the components to cool.

Hot surfaces



Sharp objects Great care should be taken when working with glass or quartz parts to prevent breakage and cuts.

Schedule

Daily

- Check that the exhaust system has positive extraction
- Check the gas supplies
- Check all hoses and connections for damage, leaks and wear
- Empty the drain vessel
- Clean the instrument
- Clean the burner (Flame systems only)
- Rinse out the spray chamber and liquid trap (Flame systems only)
- Check graphite components including the shroud, electrode, and tube (Furnace/Zeeman systems only)
- Clean the electrodes (Furnace/Zeeman systems only)
- Replace the tube if necessary (Furnace/Zeeman systems only)

Weekly

- Check for o-ring deterioration:
 - Three o-rings inside the burner, spray chamber, and the bung (see Figure 4)
 - Two o-rings that seal the gas inlets to the spray chamber (see Figure 5)

NOTE Always ensure the sealing surfaces are clean and free of debris before re-inserting the component.

Maintenance and Troubleshooting



Figure 4. Disassembled spray chamber and burner where:

- 1. O-ring on burner assembly
- 2. O-ring inside the spray chamber
- 3. O-ring inside the bung



Figure 5. Spray chamber removed from gas inlet where: 1. O-rings (x2) that seal the gas inlets to the spray chamber

- Clean the lamp and sample compartment windows
- Check the air compressor filter
- Check the water levels in recirculating supplies (Furnace/Zeeman systems only)

Yearly

Arrange for an Agilent service engineer to perform preventative maintenance.

Cleaning

Instrument surfaces

Clean contamination immediately using the approved laboratory procedures.

If there is contamination inside the instrument immediately turn off the instrument and call your Agilent representative.

- 1 Switch off the instrument and allow it to cool.
- 2 Remove any hardware present in the sample compartment (refer to the appropriate instructions in this User's Guide and the SpectrAA Help if necessary).
- **3** Using a cloth dampened with water, clean the sample compartment, instrument cover, and chimney. On Flame systems, also clean the front panel

Maintenance and Troubleshooting

and flame shield. If necessary, use a mild detergent to clean the cover. Do NOT use an abrasive cleaner. When analyzing biological samples, wipe the instrument with chlorhexidine in 95% ethanol.

Windows

Clean the sample compartment windows and the lamp compartment window with a gentle stream of filtered air, or a puffer brush, like those used to clean camera lenses.

If the windows are dirty, wipe them using an optical tissue held between tweezers and dampened with a 50% ethanol/water solution.

Filters

Check the filter in the air compressor supply line and clean if necessary (instructions are included with the filter.)

Gas cylinders

Change gas cylinders according to the instructions supplied by the manufacturer.

When changing gas cylinders:

- Inspect all gas supply pipes and hoses. Replace any hose showing signs of damage or deterioration.
- Test all connections for leaks using a commercial leak testing solution, a mild dish washing soap solution, or an electronic leak detector.
- Test the operation of all regulators.
- Ensure that shut off valves are correctly functioning.

Preventing flashbacks

Instrument and consumable maintenance is important to prevent flashbacks or unexpected flame events. See the Safety section on Page 22 for detailed information on how to prevent flashbacks and Page 24 for what to do in the event of a flashback.

Spare parts

See the Agilent website for ordering information.

Use only Agilent-supplied or approved spare parts with your instrument.

Maintenance and Troubleshooting

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In this guide

- The guide describes the following:
- Safety practices and hazards
- Introduction
- Installation
- Software overview
- Getting started
- Maintenance and troubleshooting

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