

# Agilent 5100 ICP-OES

## **User's Guide**



## Notices

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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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## 1. Safety Practices and Hazards

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## General

Operation of an Agilent 5100 Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES) involves the use of compressed gases, high voltage radio frequency energy and hazardous materials including corrosive fluids and flammable liquids. Careless, improper or unskilled use of this spectrometer or chemicals used with it can cause death or serious injury to personnel, and/or severe damage to equipment and property. Only trained personnel should use this instrument.

The spectrometer incorporates interlocks and covers that are designed to prevent inadvertent contact with any potential hazards. If the instrument is used in any manner not specified by Agilent, this protection provided by the equipment may be impaired. It is good practice to develop safe working habits that do not depend upon the correct operation of the interlocks for safe operation. It is essential that no interlock or cover is bypassed, damaged or removed. The safety practices described below are provided to help the user 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.

## Plasma

The plasma is extremely hot (about 10,000  $^{\circ}$ C) and radiates dangerous levels of radio frequency (RF) and ultraviolet (UV) energy. The work coil operates at 1,500 V RMS and about 27 MHz. Exposure to the RF and UV energy can cause severe skin damage and cataracts of the eyes, while close contact with the operating plasma can result in severe heat burns to the skin, and an electrical discharge that can jump a considerable distance and may cause death, severe electric shock or sub-surface skin burns.

The plasma must *never* be operated unless:

- the plasma compartment door is closed, with the door handle fully closed; and
- the space above the chimney and air intake is clear of objects.

The shielding around the torch compartment is designed to reduce UV, visible and RF radiation to safe levels while still permitting easy access to, as well as installation and viewing of, the torch. The spectrometer has an interlock system that is designed to extinguish the plasma if the mains supply fails, the handle on the torch compartment door is opened, or the torch loading handle is open. *Do not* attempt to bypass the interlock system.

Before opening the torch compartment door, *always* extinguish the plasma by pressing SHIFT + F5 on the keyboard or by clicking the 'Plasma Off' icon on the ICP Expert software toolbar.

The torch and its surroundings remain hot for up to five minutes after the plasma is extinguished. Touching this area before it has cooled sufficiently may result in burns. Allow the torch and torch compartment to cool before carrying out any work in this area, or wear heat-resistant gloves. The plasma system has been carefully designed to operate safely and effectively when using torches and related components that conform to Agilent's design criteria. Use of non-approved components in the plasma compartment may render the system inoperative and/or hazardous. It may also invalidate the warranty on the instrument. Use only torches and related components supplied or authorized by Agilent.

## **Heat, Vapors and Fumes**

Heat, ozone, vapors and fumes generated by the plasma can be hazardous, and must be extracted from the instrument by means of an exhaust system. Ensure that an exhaust system of the appropriate type is fitted (as specified in the Site Preparation Guide). The system must be vented to the outside air in accordance with local regulations and never within the building. Regularly check the exhaust system by smoke test to ensure that the exhaust system is functioning correctly. The exhaust system must always be switched on *before* igniting the plasma.

## **Compressed Gas Hazards**

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 or oxygen) can result in an oxygen-deficient atmosphere, which can cause asphyxiation. The area in which cylinders are stored and the area surrounding the instrument must be adequately ventilated to prevent such gas accumulations.

Gas 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 and secured to an immovable structure or a properly constructed cylinder stand. Move cylinders only by securing them to a properly constructed trolley.

Use only approved regulator and hose connectors (refer to the gas supplier's instructions). Keep gas cylinders cool and properly labeled. (All cylinders are fitted with a pressure relief device that will rupture and empty the cylinder if the internal pressure is raised above the safe limit by excessive temperatures.) Ensure that you have the correct gas before connecting it to the instrument. The primary gas used with the spectrometer is argon, which is the conductive gas for the plasma. Argon or nitrogen can be used as the polychromator purge gas. Other gases may be required for future options and accessories. Use only 'instrument grade' gases with your spectrometer.

If using cryogenic gases (for example, liquid argon), prevent severe burns by wearing suitable protective clothing and gloves.

## **Electrical Hazards**

The spectrometer system and some 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 or covers which are retained by screws on the spectrometer and accessories may be opened *only* by Agilent-trained, Agilent-qualified or Agilent-approved field service engineers (unless specified otherwise). Consult the manuals or product labels supplied with your personal computer (PC), monitor, printer and water-cooling system to determine which parts of those systems are operator-accessible.

Connecting the Agilent 5100 to a power source that is not equipped with a protective earth contact creates a shock hazard for the operator and can damage the instrument. Likewise, interrupting the protective conductor inside or outside the Agilent 5100 or defeating the power cord ground creates a shock hazard for the operator and can damage the instrument.

## **Other Precautions**

Use of the spectrometer system and accessories may involve materials, solvents and solutions which are flammable, corrosive, toxic or otherwise hazardous. Careless, improper or unskilled use of such materials, solvents and solutions can create explosion hazards, chemical burn hazards, fire hazards, toxicity and other hazards that can result in death, serious personal injury or damage to equipment. Apply all necessary precautions including use of lab coats, safety goggles and other appropriate forms of personal protection. All wastes should be disposed of in accordance with local regulatory requirements. Operation of an ICP-OES involves analysis of solutions that have been prepared in or digested with acids, or in some cases, samples that have been prepared in organic solvents.

The acid concentration in the sample that is measured varies, depending upon the digestion steps and acid types used. Instrument users should be aware of the hazards associated with use of the acids used for sample preparation and apply all necessary precautions including use of lab coats, safety goggles and other appropriate forms of personal protection. The acid wastes should be disposed of in accordance with local regulatory requirements.

The type, volatility and concentration of the organic solvents used in the sample that is measured varies, depending upon the selected solvent and the sample preparation involved. Instrument users should be aware of the hazards associated with use of the organic solvents for sample preparation, and apply all necessary precautions including ensuring adequate ventilation during use, and use of lab coats, safety goggles, gloves and other appropriate forms of personal protection. The organic wastes should be disposed of in accordance with local regulatory requirements.

Air flow to the air intake port of the spectrometer and accessories must be unobstructed. Do not block the ventilation grills on the spectrometer and accessories. Consult the manuals supplied with your PC, monitor, printer and water-cooling system for their specific ventilation requirements.

Great care should be taken when working with glass or quartz parts to prevent breakage and cuts. This is especially important when inserting the nebulizer into the spraychamber, or removing and replacing pieces of broken torch.

The spectrometer weighs approximately 106 kg (234 lb). To avoid injury to personnel or damage to the instrument or property, always use suitable mechanical lifting device to move the instrument.

Use only Agilent-supplied or approved spares with your instrument. Only trained operators should use the instrument.

## 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:



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.



Indication of correct orientation of gas filter flow direction

## **CE Compliance**

Your Agilent 5100 ICP-OES instrument has been designed to comply with the requirements of the Electromagnetic Compatibility (EMC) Directive and the Machinery Directive (MD) 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 Industrial, Scientific and Medical (ISM) equipment in which there is intentionally generated and/or used conductively coupled radio- frequency energy which 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 which 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.
- 2 Move the device away from the radio or television.
- **3** Plug the device into a different electrical outlet, so that the device and the radio or television are on separate electrical circuits.
- 4 Make sure that all peripheral devices are also certified.
- **5** Make sure that appropriate cables are used to connect the device to peripheral equipment.
- **6** 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.

#### **South Korean Class A EMC declaration**

A 급 기기 (업무용 방송통신기자재)

This equipment is Class A suitable for professional use and is for use in electromagnetic environments outside of the home.

이 기기는 업무용 (A 급 ) 전자파적합기기로서 판 매자 또는 사용자는 이 점을 주

의하시기 바라 며, 가정외의 지역에서 사용하는 것을 목적으 로 합니다. **Safety Practices and Hazards** 

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## **2.** Introduction

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## **Site Preparation Requirements**

Prior to receiving your instrument you will have been provided with an Agilent 5100 ICP Optical Emission Spectrometer Site Preparation Guide, which describes the environmental and operating requirements of the ICP-OES system. You must prepare your laboratory according to these instructions before the ICP-OES can be installed. You should keep the Site Preparation Guide for future reference for example, if you plan to move your instrument. If you have misplaced your copy, you can obtain a free replacement from your local Agilent office.

Position the equipment for easy access to the disconnecting switch on the left side of the instrument.

## **User Documentation**

You have been provided with the following documentation to help you set up and operate your Agilent ICP-OES system:

- This User's Guide, with safety practices and hazards information, instructions for finding information about installing and maintaining the components of the ICP-OES and a brief operation overview.
- An extensive Help system containing context-sensitive Help, stepby-step instructions for frequently performed operations and instructions for using any accessories you ordered.
- A Familiarization DVD that includes information on how to set up methods and run instrument tests, some hardware basics and maintenance videos.

#### Conventions

The following conventions have been used throughout the documentation:

- Menu items, menu options and field names (for example, click **Copy** from the **Edit** menu) have been typed in bold. Bold is also used to signify the buttons appearing throughout the software (e.g., click **OK**).
- ALL CAPITALS indicate keyboard commands (e.g., press the F2 key) and text you must type in from the keyboard (e.g., type SETUP at the prompt).

#### Notes and tips

A Note is used to give advice or information.

A Tip is used to give practical hints to help you achieve the best possible performance from your ICP-OES.

## **Specifications**

The Agilent 5100 ICP-OES instrument is suitable for indoor use *only* and is classified suitable under Equipment class I category.

#### Installation category

The installation category is based on IEC61010:II. The installation category implies the regulation for impulse withstand voltage. It is also called the 'Over voltage category'. 'II' applies to electrical equipment with detachable power cords.

#### **Pollution level**

The pollution level is based on IEC61010:2. Pollution level describes the degree to which a solid, liquid or gas that deteriorates dielectric strength is adhering. '2' applies to a normal indoor atmosphere, where only non-conductive pollution occurs.

#### **Temperature control**

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

#### **Environmental conditions**

See the Agilent 5100 ICP-OES Site Preparation Guide for specifications.

## **Electrical Power Supplies**

For electrical specifications, refer to your Agilent 5100 ICP-OES Site Preparation Guide.

All power supplies should be single phase AC, 3 wire system (active, neutral, ground or two active and ground) and should be terminated at an appropriate connection receptacle that is within reach of the system power cable. Use of power boards or extension cables is *not* recommended.

The installation of electrical power supplies must comply with the rules and/or regulations imposed by the local authorities responsible for the use of electrical energy in the workplace.

Avoid using power supplies from a source that may be subject to electrical interference from other services (such as large electric motors, elevators, welders and air conditioning units).

#### **Circuit breaker**

#### NOTE

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

The mains power switch contains a 20 A circuit breaker, which is reset when the power switch is cycled.

If necessary, replace the power cord only with a cord equivalent to the one specified in the site preparation guide.

## **Other Connections**

IEEE 802.3, Ethernet LAN cable

## **Personal Computer Requirements**

The recommended and minimum PC specifications can be found in the Agilent 5100 ICP-OES Site Preparation Guide.

Locate the PC keyboard and mouse for ergonomically correct access.

## **Gas Supplies**

The installation of compressed or liquid gas supplies must comply with the rules and/or regulations imposed by the local authorities responsible for such use in the workplace. Liquid or gaseous argon and nitrogen may be used with Agilent ICP-OES spectrometer systems. Agilent recommends the use of liquid gases, which are purer, more convenient and cheaper per unit volume.

The main gas supply requirement is argon for supply to the plasma, nebulizer and optics interface purge. Gas is also required to purge the polychromator assembly, and this may be either argon or nitrogen. A separate gas line to the polychromator connects internally to the argon supply unless the optional nitrogen purge kit is fitted. Gas supply regulator pressure setting may need to be adjusted to ensure the pressure is in the permissible pressure range when delivering the gas flow demanded during operation.

#### Table 1. Gas requirements

| Argon                 | Nitrogen  |
|-----------------------|---|
| 99.996%               | 99.996%   |
| <5 ppm                | <5 ppm  |
| <20 ppm               | -   |
| <4 ppm                | <4 ppm  |
| 500-600 kPa (73 to 88 | 3 psi) gauge  |
| 550 kPa (80 psi) gaug | ge regulated  |
|                       | Argon<br>99.996%<br><5 ppm<br><20 ppm<br><4 ppm<br>500-600 kPa (73 to 84<br>550 kPa (80 psi) gaug |

\*when supplying required gas flows

#### Table 2. Typical flow rates for the Agilent 5100 ICP-OES instruments

|  | Argon (with argon purge gas) | Nitrogen (as purge gas)                                  |
|--|------------------------------|--|
| Instrument idle state                              | 0.70 L/min                   | Nitrogen flow 0.8 L/min                                  |
| Operational range<br>(minimum-maximum, plasma on)  | 9.1–31.8 L/min               | Argon flow 8.4–28.1 L/min<br>Nitrogen flow 0.8–4.4 L/min |
| Typical Flows                                      |                              |  |
| Measuring wavelengths<br>> 189 nm (poly boost off) | 14.65–20.65 L/min            | Argon flow 13.95–19.95 L/min<br>Nitrogen flow 0.8 L/min  |
| Measuring wavelengths<br>< 189 nm (poly boost on)  | 19.25–25.25 L/min            | Argon flow 15.55–21.55 L/min<br>Nitrogen flow 4.4 L/min  |

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The spectrometer is supplied with three PVDF gas supply hose assemblies, 3 m (9.8 ft) in length. Gas supply adapters are included with each instrument to connect the instrument to regulated gas supplies.

The user (or other authorized personnel) must carry out appropriate leak tests necessary to ensure safety on the gas and liquid connections that the operator is directed to assemble during installation, normal use or maintenance.

#### Storage cylinder instructions

Cylinders containing gas under pressure should be firmly secured to a rigid structure, and the storage area must be adequately ventilated.

Never locate gas cylinders near a source of ignition, or in a position that is subject to direct heat. Gas storage cylinders often incorporate a pressure release device, which will discharge the gas at a predetermined temperature, usually around 52 °C (125 °F).

If gases are to be plumbed from a remote storage area to the instrument site, ensure that the local outlets are fitted with stop valves, pressure gauges and suitable regulators, which are easily accessible to the instrument operator.

#### **Cryogenic liquids**

**Extreme Cold Hazard** 

Cryogenic liquid gases are stored under pressure at very low temperatures in Portable Liquid Cylinders (PLCs).

## WARNING



Contact with the super-cold liquid, gas or pipe surfaces can cause severe skin damage. The PLCs should be located in a shielded position, and all piping should be routed or covered to prevent skin contact.

For high gas flow rates and/or low ambient temperatures, it may be necessary to obtain sufficient gas pressure by passing the liquid through an external evaporator rather than use the internal pressure building facility of the PLCs. Liquid argon and liquid nitrogen may *not* be stored for extended periods and often have special storage requirements. Contact your local authorities and cryogenic gas supplier for more detailed information on storage requirements and boil-off rates for local types of PLCs.

## **Exhaust system**

The plasma operates at extremely high temperatures. The ICP-OES instrument exhaust fumes can be noxious or corrosive.

The ICP-OES instrument exhaust must be directly connected to an exhaust system. The exhaust system must include an exhaust fan, be ducted to an external vent and provide a minimum flow of  $2.5 \text{ m}^3/\text{min}$  (88 ft<sup>3</sup>/min) at 2.4 m/s (7.7 ft/s) and a maximum flow of  $6.0\text{m}^3/\text{min}$  (212 ft<sup>3</sup>/min) at 5.7 m/s (18.6 ft/s).

The exhaust system installation must comply with any rules and/or regulations that may be imposed by the local authorities responsible for control of facilities and fixtures in the workplace.

The exhaust fan should be located at least 2 meters (6 ft, 6 in) away from the top of the instrument chimney. The fan control switch and running indicator lamp should be located in a position where the instrument operator can view the indicator and access the control switch.





#### **Hot Surface**

The external vent may become hot during ICP-OES instrument operation and remain hot for some time after the instrument has been switched off. Allow the external vent to cool for at least five minutes before attempting to remove the exhaust hose. Use heat-resistant gloves.

## **ICP-OES** instrument cooling air supply

The ICP-OES instrument requires *clean*, *dry*, *non-corrosive air for cooling purposes*. This is supplied to the instrument through an air supply vent located at the top, left of the instrument. The vent has a dust filter, to filter out particulate matter.

The air supply is used to cool the internal mechanical and electronic components of the instrument. Several of these assemblies contain parts prone to corrosion. The introduction of cooling air contaminated with high levels of acid vapors or other corrosive substances may cause damage to the instrument.

Due to the corrosive nature of some analytical work, it is recommended that in applications demanding high usage of corrosive materials, an external cooling air supply system is provided. It is *required* that the cooling air be supplied from an environmentallycontrolled area that is away from the instrument exhaust and any other area where corrosive materials are stored or used. Do not duct humid, warm air into an instrument in a cooled laboratory environment.

The cooling air system with flue, fan, ducting and supply cowl, must provide positive air pressure at the instrument inlet of 4 m<sup>3</sup>/min (141 ft<sup>3</sup>/min) when using the External Inlet Duct Adaptor Kit (G8010-68002). The ducting should be corrosion-resistant and fire-proof.

## Water Cooling System

Agilent ICP-OES instruments require a source of cooling water. Refer to the Site Preparation Guide for compatible water cooling systems.

**NOTE** The operation manual, mounting and assembly hardware for the cooling system are included in the water cooler packaging. Care should be exercised to locate all of these articles before the pack is discarded.

# **NOTE** Pressure regulation is recommended for supplies where the cooling water pressure may be subject to fluctuations. Pressure regulation is necessary for supplies that may exceed the maximum permissible pressure of 400 kPa (58 psi).

The instrument is equipped with a water flow sensor, which will stop operation of the plasma if the cooling water flow through the instrument drops below 1.7 L/min (0.45 gpm). A second water flow sensor will stop the camera Peltier cooling assembly if the water flow through the instrument drops below 0.2 L/min (0.05 gpm).

CAUTION Always ensure the water cooling system is on before igniting the plasma.

## **Drain Vessel**

The Agilent ICP-OES system needs a drain vessel for disposal of excess fluids and vapors from the spraychamber or autosampler. Suitable tubing is supplied with the spectrometer for use with inorganic solvents. When using organic solvents, different drain tubing that is suitable for the solvent in use will be required.

A chemically inert container, not glass or of a narrow-necked style, to hold a minimum of 2 liters (4 pints) of waste must be provided by the user. It should be located underneath the sample compartment (or on the right side of the instrument), where it is protected by the bench and in full view of the operator. Introduction

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## 3. Installation

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The Agilent 5100 ICP-OES must be installed by an Agilent-trained, Agilent-qualified or Agilent-authorized field service engineer.

You should have completed and returned the form in the Site Preparation Guide stating that you have prepared the laboratory in accordance with the requirements detailed in that manual. An Agilent representative will then arrange a suitable installation date with you.

Details for unpacking the instrument and what to do in case it has been damaged in transit are also outlined in the Site Preparation Guide.



## Agilent 5100 ICP-OES Instrument Overview

## Figure 1. Front and side of the ICP-OES instrument

| 1. Exhaust   | 8. Torch loader handle                      | 15. Torch compartment handle                  |
|--|---|---|
| 2. Air inlet filter  | 9. Spraychamber                             | 16. Water assembly                            |
| 3. Cone, radial and axial pre-<br>optics windows (not shown) | 10. Nebulizer                               | 17. Optics purge filter for argon or nitrogen |
| 4. Snout   | 11. Peristaltic pump                        | 18. Gas supply assembly                       |
| 5. Induction coil  | 12. Mains power button and cable connection | 19. Drain for liquid overflow                 |
| 6. Torch   | 13. Front panel power button                |   |
| 7. Nebulizer and make up gas connections                     | 14. LED instrument status<br>indicator      |   |

All connections of services to the ICP-OES instrument are made on the right side of the instrument, except for the mains power which is on the left side of the instrument. Remove the cover on the right side by hand to gain access to the Ethernet, accessory, water and gas connections. The Power on/off button is located on the front lower left side of the instrument and the mains power switch is located on the left back side of the instrument.



Figure 2. Input and output connections on the side of the ICP-OES instrument

|   | ltem                                | Description   |
|---|-------------------------------------|---|
| 1 | Accessory and LAN cable connections | Agilent accessory connections and Ethernet cable for PC to instrument communication |
| 2 | Argon gas inlet                     | Argon gas inlet, standard   |
| 3 | Option gas inlet                    | Option gas (80% Ar/20% $O_2$ mix) inlet, optional                                   |
|   |                                     | Nitrogen gas inlet, optional (not shown)  |
| 4 | Optics purge filter                 | Argon or nitrogen gas filter  |
| 5 | Water outlet                        | Connect to water chiller  |
| 6 | Water filter                        | Coarse particulate water filter   |
| 7 | Water inlet                         | Connect to water chiller  |

## **Instrument Status LED Color Coding**

The instrument status indicator light on the front top right of the Agilent 5100 ICP-OES displays different colors to represent the status of the instrument:

- A green light indicates the following:
  - The instrument and software are connected and are ready to attempt a plasma ignition sequence, or
  - The instrument and software are connected and the plasma ignition sequence is underway, or
  - The instrument and software are connected, the plasma is lit and running and it is possible to run analysis of samples.
- A yellow blinking light indicates the instrument and software are not connected (instrument is off-line).
- A yellow light indicates that the instrument and software are connected, but the instrument is not ready to begin a plasma ignition sequence due to a tripped door or torch handle interlock, low gas pressure, low water flow or low cooling air flow. User intervention is required to resolve the problem.
- A red light indicates an instrument firmware error causing a halt to analysis or restricting normal instrument operation. User intervention is required to resolve the problem.

## Front Power On/Off Switch

The LED in the front on/off switch indicates the basic instrument on/off state.

- **Off** indicates that the instrument is completely unpowered when the mains power switch on the left side of the instrument is also off.
- **Green flash once every 10 seconds** indicates most of the instrument is unpowered, with the exception of the front panel Power on/off button switch sensing circuit inside the mains power module. Press the front panel Power on/off switch at the front to fully power up the instrument.

- **Green blinking slow (1Hz).** The mains power module is initializing and running through power up self-check, or is shutting off the instrument.
- **Green blinking fast (4Hz).** Mains power module failed its initialization power-up self-check, instrument is unusable. The mains power supply is not adequate or the internal mains module needs to be replaced.
- Green constantly on indicates the instrument has been switched on.

## **ICP Expert Software**

The Agilent-trained, Agilent-qualified or Agilent-authorized field service engineer will install the ICP Expert software for you during the installation process. However, you may need to install the software yourself at some later stage, for example if you change the PC. Instructions are provided.

#### There are several installation scenarios:

- Standalone or network Flat File for Microsoft Windows 7
- Standalone or network Database for 21 CFR Part 11 installations for Microsoft Windows 7
- Upgrading Standalone or Network Database ICP Expert installations for Microsoft Windows 7

#### Installation includes:

- Installing the ICP Expert software
- Installing the ICP Expert Help
- Installing the LAN cable
- Setting the instrument IP address
- Installing and configuring SDA for 21 CFR Part 11 compliance
- Installing and configuring SCM for 21 CFR Part 11 compliance
- Completing a dark current scan and wavelength calibration

#### For instructions on how to install your ICP Expert software, refer to the documentation provided with your software:

- ICP Expert Windows 7 64-bit (SP1) Software Installation Instructions
- ICP Expert Software Installation Instructions for 21 CFR Part 11 Environments for Windows 7 64-bit (SP1) only

Once you have installed the appropriate software, connect the Ethernet cable, set the instrument IP address if necessary, and then perform the Detector Calibration and Wavelength Calibration.

## **Connecting ICP Expert software to the ICP-OES**

#### To connect the software and instrument:

- 1 Start the ICP Expert software by double-clicking on the desktop icon or from Start > All Programs > Agilent > ICP Expert > ICP Expert.
- 2 Click Instrument.
- 3 Click Connect.
- **4** Enter the IP address of the instrument or select an existing instrument.
- 5 Click Connect.
- 6 Click Close.

## Performing a Detector and Wavelength Calibration

#### To perform a dark current scan and wavelength calibration:

- **1** Turn on the external exhaust.
- 2 Start the ICP Expert software and open the Instrument Set-up Window.
- **3** Connect the software to the instrument, if needed.
  - **a** Click **Instrument** in the ICP Expert toolbar.
  - **b** Click **Connect**.
  - **c** Select the instrument from the list and the click **Connect**.

- 4 Ensure a standard glass concentric nebulizer, a double pass spray chamber and the appropriate plasma torch (for radial or dual view) are installed. Use manual sampling. The recommended peristaltic pump tubing is White/White for the pump and Blue/Blue for the drain.
- **5** Ignite the plasma.
- **6** Check that the Polychromator Boost purge is on.
  - **a** On the 'Instrument' window of the ICP Expert software, click the **Status** tab and check that under the Polychromator section Boost is selected.
  - b If the instrument was turned on from an idle state, the wavelength calibration in Steps 7-10 can be performed immediately. If the instrument was turned on after more than a few hours of being unpowered the polychromator can take several hours to stabilize the temperature and to purge.
- 7 Aspirate a blank solution and then select the **Calibration** tab.
- 8 Click Calibrate in the 'Detector' section.

**NOTE** Once the detector calibration is completed, the date and time of the last successful calibration will be displayed, confirming the dark current measurement has been completed.

- **9** Aspirate the wavelength calibration solution and then select the **Calibration** tab.
- **10** Click **Calibrate** in the 'Instrument' section to perform a wavelength calibration.
- **NOTE** Once completed, the date and time of the last successful calibration will be displayed, a pass or fail mark will appear along with the wavelength error indicating whether the wavelength calibration is complete.

#### Saving and viewing the calibration data

The calibration data is stored in the Logs. To view the data, export the Logs.

- From the main ICP Expert window, click File > Logs > Export Logs.
- **2** Save the zipped Log file.
- **3** Open the .zip file to view the Log files.

#### NOTE

The default save location is Users'Username'DocumentsAgilentICP ExpertExported Results on the drive on which the ICP Expert software is installed.

## **Hardware Components Replacement**

Your ICP-OES should be ready to operate after the Agilent field service engineer has installed it. However, you may need to set up items such as the peristaltic pump, nebulizer, torch or spraychamber. For instructions on how to do this, please refer to the 5100 ICP-OES Familiarization DVD or the 'How to' section in the ICP Expert Help.

#### 5100 ICP-OES torches

Many of the 5100 ICP-OES torches are listed below, each with unique properties to suit a wide range of applications. Additional torches may be available. See the Agilent website for more information.

| Parameter              |                  | Dual View Torches              |                          | Dual view, large<br>diameter injector |
|------------------------|------------------|--------------------------------|--------------------------|---------------------------------------|
|                        | Dual view torch  | Dual view torch<br>demountable | HF dual view torch       | High salt dual view<br>torch          |
| Construction           | One piece        | Two piece<br>demountable       | Two piece<br>demountable | Two piece<br>demountable              |
| Injector diameter (mm) | 1.8              | 1.8                            | 1.8                      | 2.4                                   |
| Injector material      | Quartz           | Quartz                         | Ceramic                  | Quartz                                |
| Outer tube length      | Medium with slot | Medium with slot               | Medium with slot         | Medium with slot                      |
| Outer tube material    | Quartz           | Quartz                         | Quartz                   | Quartz                                |
| Intermediate tube      | Quartz tulip     | Quartz tulip                   | Quartz tulip             | Quartz tulip                          |

| Parameter              | Organics                                     | Radial View Torches |                                |                          | Volatile Organics<br>Torch        |
|------------------------|--|---------------------|--------------------------------|--------------------------|-----------------------------------|
|                        | Semi-volatile<br>organics dual<br>view torch | Radial torch        | Radial<br>demountable<br>torch | HF radial torch          | Radial volatile<br>organics torch |
| Construction           | Two piece<br>demountable                     | One piece           | Two piece<br>demountable       | Two piece<br>demountable | Two piece<br>demountable          |
| Injector diameter (mm) | 1.4  | 1.4                 | 1.4                            | 1.8                      | 0.8                               |
| Injector material      | Quartz                                       | Quartz              | Quartz                         | Ceramic                  | Quartz                            |
| Outer tube length      | Medium with slot                             | Short (no slot)     | Short (no slot)                | Short (no slot)          | Short (no slot)                   |
| Outer tube material    | Quartz                                       | Quartz              | Quartz                         | Quartz                   | Quartz                            |
| Intermediate tube      | Quartz tulip                                 | Quartz tulip        | Quartz tulip                   | Quartz tulip             | Quartz tulip                      |

## **Torch Assembly and Disassembly**

The demountable torch models allow the outer/intermediate tube set to be separated from the base/injector for easier cleaning.



#### **Hot Surface**

The torch and torch compartment become warm during instrument operation and remain so for some time after the plasma has been switched off. Allow the plasma compartment to cool for at least five minutes before accessing the plasma compartment. Allow the torch to cool for two minutes before touching the outer tube or attempting to dismantle the torch.

## WARNING



#### **Chemical Hazard**

Nitric and hydrochloric acids are very corrosive and can cause severe burns when they come into contact with the skin. It is essential that appropriate protective clothing be worn at all times when handling these acids. If acid contacts the skin, wash off with copious amounts of water and seek medical attention immediately.

## CAUTION

To prevent damage to the torch, always take care when handling or storing it. To prevent damage to the instrument, do not use the torch if it is damaged.



**Figure 3.** Demountable torch where: 1. Torch, 2. Torch securing ring and 3. Torch body

#### **Torch assembly**

#### To assemble the torch:

1 Position the flat side of the torch securing ring so that it faces the top of the torch.



Figure 4. Flat side of torch securing ring

2 Slide the torch securing ring approximately 1/3 the way up the torch.



Figure 5. Torch securing ring on torch

**3** <u>Slide the torch into the torch body.</u>



Figure 6. Torch inserted into torch body

**4** Position the torch so that the groove on the torch body aligns with the left side of the hole in the torch.



Figure 7. Torch alignment

#### Installation

**5** Press the torch fully into the torch body and then press down on the torch securing ring.



Figure 8. Torch inserted into torch body

## **Torch disassembly**

#### To disassemble a two piece demountable torch:

You may need a flat head screwdriver to loosen the securing ring if you cannot gently slide the torch out of the torch body by hand.

- 1 Insert the screwdriver into the slot between the torch securing ring and the torch body.
- **2** Gently twist the screwdriver to separate the torch securing ring from the torch body.



Figure 9. Separating the torch securing ring from the torch body

**3** Repeat Steps 1 and 2 on the slot on the opposite side.

**4** Gently slide the torch out of the torch body.



Figure 10. Removing the torch from the torch body

**5** Slide the torch securing ring off the torch.



See Page 56 for the torch cleaning procedure.

## Accessories

The following accessories may be available for use with your ICP-OES instrument:

- SPS 3 Sample Preparation System
- SVS 2 and SVS 2+ Switching Valve Systems
- 5 channel peristaltic pump
- Air inlet dust filter
- External inlet duct adapter

#### Installation

- Vapor generation accessory (VGA)
- Multimode sample introduction system (MSIS)
- Argon humidifier

For safety and installation information, please see the accessory documentation or the ICP Expert Help.

#### SPS 3

For safety information and to prepare the Sample Preparation System (SPS 3) for installation, please see the instructions that came with the accessory.

The SPS 3 is compatible with a wide range of commercially available low-cost autoclavable sample racks.

Sample contamination from airborne particles is eliminated and corrosive or toxic fumes are removed during sampling with the optional environmental enclosure.

#### SVS 2 and SVS 2+

For safety information and to prepare the accessory for installation, please see the instructions that came with the accessory.

The SVS 2 and SVS 2+ increase sample throughput and decrease turnaround time and operating costs. The switching valves are positioned between the nebulizer and the peristaltic pump of the spectrometer. Samples are quickly loaded into the sample loop, ready for immediate analysis by the ICP-OES, greatly reducing sample uptake delays. Pre-emptive rinsing of the sample line means analysis times are reduced. The switching valve also features an internal Tpiece within the valve, reducing dead volume and providing online addition of internal standard and ionization buffer solutions. A bubble injector automatically injects bubbles after the sample is loaded into the loop, isolating the sample from the rinse solution. This reduces the volume of sample required for measurement as tailing (or dilution) effects are minimized.

#### **5-channel Peristaltic Pump**

The five channel peristaltic pump can be used for additional solution introduction and is required when using the MSIS accessory.

#### **External Inlet Duct Adapter**

The External Inlet Duct Adapter provides an attachment for ducting air into the air intake port, for use in labs with harsher environments.

#### Air inlet dust filter

The air inlet dust filter provides fine dust filtration of the air drawn into the air intake port.

#### Vapor Generation Accessory (VGA)

The VGA 77P is a continuous flow vapor generation accessory for Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) instruments that determines Hg and the hydride-forming elements at parts per billion (ppb) levels. The entire plumbing assembly is integrated into a separate module and can be simply exchanged when switching elements.

#### Multimode Sample Introduction System (MSIS)

For safety information and to prepare the MSIS for installation, please see the instructions that came with this accessory and the ICP Expert Help.

The MSIS is used with the ICP-OES instrument to provide simultaneous vapor generation of several hydride forming elements, enabling determination with low ppb detection limits.

The MSIS consists of a nebulizer and modified glass cyclonic spray chamber that has two vertical conical tubes in the center of the chamber. This allows for reductant and sample to mix quickly and thoroughly in the chamber using thin film hydride technology to form the hydrides. The MSIS can be operated in three modes: hydride only, simultaneous hydride and conventional nebulization or conventional nebulization only.

#### **Argon Humidifier**

The Argon Humidifier is commonly used when running aqueous samples with high dissolved solids or high dissolved salt content. When using the accessory, the nebulizer gas flow is passed through the humidifier to increase the water vapor in the gas. This has been found to be beneficial by reducing the build-up of salt and other dissolved solids in the sample introduction system. By reducing blockages, the Argon Humidifier helps to ensure uninterrupted, maintenance-free operation.



## 4. Operation

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This chapter provides a quick guide to getting the instrument set up and running samples.

You will find step-by-step instructions for common operations in the ICP Expert Help. To access this information:

- 1 Select the Windows Start button and choose Programs > Agilent > ICP Expert > ICP Expert Help.
- **2** When the ICP Expert Help appears, click **How to** to view the available step-by-step instructions.

## **Analysis Checklist**

You need to complete the following steps in turn to measure a sample/s. You will find information on each step in this chapter.

- Turn on the PC, instrument and software
- Connect the software to the instrument
- Prepare for analysis
- Perform a dark current scan and wavelength calibration

#### **Operation**

- Create/open a worksheet
- Develop a method
- Run samples
- Print a report

## **Turning On the Instrument and Software**

Before starting the system, carefully read the Safety practices and hazards section at the front of this manual and ensure that the laboratory is set up according to the details specified in the Site Preparation Guide.

# Turning on the instrument for the first time (or from long term shutdown)

#### To turn on the instrument for the first time or from shutdown:

- 1 Check that the exhaust and intake lines are secured to the ICP-OES instrument, and that the air filter is not blocked.
- 2 Turn on the laboratory exhaust system.
- **3** Ensure the gas and water lines are connected to the ICP-OES instrument
- **4** Switch on the water cooler.
- **5** Turn on the gas supply.
- **6** Ensure that the gas and water supplies are turned on and set to the correct pressures, and that the water cooler is set to the correct temperature.
- 7 Check that the torch is clean and in good condition, and installed with the torch handle fully closed.
- 8 Check that all tubing on the spraychamber, nebulizer and peristaltic pump is installed and correctly connected.
- **9** Check that the plasma compartment door is fully closed.
- **10** Check that the Ethernet LAN cable is connected to the computer or Local Area Network.
- **11** Switch on the computer, monitor and printer.

- **12** Plug the ICP-OES cable into the wall socket and set the mains power switch on the left side of the instrument to 'On'.
- **13** Press the Power on/off switch on the front of the instrument. The LED will display green when it is on. The 5100 ICP-OES is now in the idle state, which continually purges and thermostatically controls the polychromator.

# **NOTE** Both the mains power switch on the left side of the instrument and front panel power switch must be on for the instrument to work.

14 To start the ICP Expert software, click Start > All Programs > Agilent > ICP Expert > ICP Expert or double-click the ICP Expert desktop icon. The Main Index window will appear.

#### Running the instrument from an idle state

During an idle state (both power switches are 'On'), the polychromator temperature is regulated and polychromator gas purge is running to ensure the instrument is ready for analysis within 20 minutes after lighting the plasma.

#### To begin using the instrument from an idle state:

- **1** Turn on the laboratory exhaust system.
- **2** Check that the torch is clean and in good condition, and installed with the torch handle fully closed.
- **3** Check that all tubing on the spraychamber, nebulizer and peristaltic pump is correctly connected.
- 4 Check that the torch compartment door is fully closed.
- **5** Switch on the monitor and printer (if they are off).
- **6** Switch on the water cooler (if it is off).
- 7 If you have accessories fitted, switch them on.

## **Preparing for Analysis**

#### To prepare for analysis:

- Click the **Plasma** button in the ICP Expert software. Alternatively, press F5 or choose **Plasma on** from the arrow under the **Plasma** button.
- **NOTE** The plasma ignition sequence will take up to 60 seconds to complete. If the plasma fails to ignite, refer to the Troubleshooting section in the Help for further information.

#### NOTE

For optimum performance and stability, for the ICP-OES instrument a warm up time of 20 minutes is recommended after igniting the plasma.

If using wavelengths below 189 nm: purging the polychromator can take up to several hours. The polychromator thermal stabilization can take up to several hours from long term shutdown.

- 2 Ensure that the peristaltic pump is correctly set up (refer to the Peristaltic Pump section of the 5100 ICP-OES Familiarization DVD or the ICP Expert Help). If you have not already done so, adjust the pressure bars on the peristaltic pump for even sample flow and drainage.
- **3** Place the sample tubing from the peristaltic pump into the rinse solution and the drain tubing into the drainage vessel.
- 4 Click the **Pump** button in the ICP Expert software and choose **Normal (15 rpm)** from the arrow under the **Pump** button. The pump will be initialized and the solution will begin aspirating.

## Performing a Detector and Wavelength Calibration

See Page 32 for instructions.

## **Creating/Opening a Worksheet**

#### Creating a new worksheet

To create a new worksheet, click **New** from the Start page or the File menu.

A list of recently used files will be presented when creating a new worksheet from a template; otherwise you may Browse for more files. The 'New From Template' dialog box will be displayed in this instance.

#### **Opening an existing worksheet**

#### To open an existing worksheet:

- 1 Click **Open** from the Start page or from the File menu.
- **2** A list of recently used files will be displayed. Otherwise, you may **Browse** for more files. The '**Open**' dialog box will be displayed in this instance.

#### Creating a new worksheet from a template

To create a new worksheet from a template click **New From** on the Start page or **New From Template** from the File menu.

A list of recently used files will be presented, otherwise you may **Browse** for more files. The New From Template dialog box will be displayed in this instance.

The Worksheet window will appear with the new worksheet loaded.

## **Developing a Method**

#### To develop a method:

- **1** Open a new worksheet or one from a template.
- **2** On the 'Elements' page, select the element(s) from the 'Element' drop-down box or type the element name or symbol and then perform one of the following:
  - Click to add the primary wavelength for the selected element.

#### **Operation**

- Highlight the wavelength you wish to use from the list of available wavelengths displayed. Click **Add.**
- **NOTE** Alternatively, press CTRL and select the element from the periodic table to add the primary wavelength for the selected element to your method.

The element will appear in the table with the selected wavelength and default settings selected.

- **3** Check that there are no known interferents or other analytical lines close to the selected analytical line. Their relative intensity will govern how close the lines can be to each other. For example, if your matrix contains an element that is not of analytical interest, but is a potential interferent and has a line close to one of your analytical lines, the concentration of that element in your matrix will determine whether you need to choose another analytical line.
- 4 Make any required adjustments to each element including selecting a different wavelength, entering additional information into the 'Label' column, and selecting the type (choose from analyte, internal standard or interferent).
- 5 Enable QC and/or IEC if these features are going to be used. (Rate QC is only available in ICP Expert Pro software.)
- **6** Click **Conditions** to modify both common settings for the run and settings for each element. Up to four different measurement conditions can be used.
- 7 Click **QC** to enter the method detection limits, select the QC tests to be used and which error actions should be performed if an error occurs.
- 8 Click **IEC** to enter the concentration of the elements in your analyte and interferent standards.
- **9** Click **Standards** to enter the concentration of the elements in your standards and select whether to use other options such as Standard Additions or MultiCal. In addition, also select whether to use the blank in calibrations and whether to enable reslope.
- **10** Click **Sequence** to specify the number of samples, insert QC tests, select the solution type, edit the sample labels and end of run actions.

- **11** Click **FACT** to manually create FACT models or to set the method up to automatically create them during analysis.
- **12** If you are using an autosampler, click the 'Autosampler' tab to select the racks and probe depth (if needed). Depending on the autosampler selected, options may vary.
- **NOTE** See the ICP Expert Help for a more detailed description of setting up a method.

## **Running Samples**

#### To run samples:

- 1 Set up the autosampler with all solutions, if required.
- 2 Click the **Analysis** tab and do the following:
  - **a** Ensure your samples are selected. This will be indicated by a check next to the Rack:Tube column. To select all solutions, select the checkbox next to the Rack:Tube title.



**b** Click the **Run** icon in the toolbar (or press the SHIFT+F8 keys) to begin the analysis, and follow the prompts.

**NOTE** For further information about running an analysis, refer to the ICP Expert Help.

## **Printing a Report**

#### To print a report:

- 1 Click **Report** on the toolbar or **File > Report**.
- **2** Choose whether you want to print or preview the report or save the report as a PDF file.

#### Operation

#### TIP

Previewing the report allows you to ensure that you have included all of the data you require.

- **3** Select a report template and then click **Open**.
- 4 Click the **Print** button to generate a report as specified. A message will appear briefly indicating the progress status of the report being generated.
- 5 The Print dialog box will then be opened. You can select to print all of the report, or a range of pages. Reports are printed to your default printer, unless you specify otherwise. You can specify your printer options in the Print Setup dialog box, accessible from the File menu.

## **Instrument Idle State**

In between routine use, it is recommended the instrument be left in the idle state, which is when the instrument is fully powered but the plasma off. In this state the polychromator thermostating system and purge systems remain operational, and the internal air cooling system runs at reduced capacity. If the ICP-OES is not going to be in use for a prolonged period of time, fully shutdown the instrument (which turns off all purging as well as the polychromator thermostating system).

#### Setting the instrument to idle

#### To put the instrument into an idle state:

- **1** Rinse the spraychamber by aspirating water for a few minutes.
- **NOTE** When running organic samples it is recommended that the spraychamber be cleaned and dried thoroughly between analyses.
  - Extinguish the plasma by clicking the Plasma Off icon, pressing SHIFT + F5 on the keyboard or choosing Plasma Off from the Analyze menu. The peristaltic pump stops automatically when the plasma is extinguished.

- 3 To save on argon costs, you can turn the polychromator boost off overnight, by deselecting **Polychromator > Boost** on the Status tab on **Instrument window**. This is not recommended if you are routinely analyzing lines below 190 nm, as the instrument will take some time to stabilize when the polychromator boost is turned on again.
- **4** To increase the pump tubing lifetime, loosen the peristaltic pump tubes by releasing the pressure bars and lift the tubes out of the grooves. To do this:
  - **a** Push up the pressure bar screws. This releases them from the pressure bar (refer to Figure 11).
  - **b** Allow the pressure bar to swing downwards.
  - **c** Lift the tubing out of the grooves.



**Figure 11.** Peristaltic pump with pressure bar screws pushed up and pressure bar freely down, allowing tubing to be loosened

- **5** Switch off the water cooler.
- **6** Close the worksheet by clicking 'Close' from the 'File' menu but leave the ICP Expert software running. You may switch off the printer, monitor and any accessories if desired.

Ensure that the Powersave option on your PC is disabled (this will prevent the shutdown of your hard disk). If this option is not disabled, you could lose data during an unexpected shutdown.

#### **Operation**



Noxious Fumes Inhalation danger. The exhaust system MUST remain on if the gas supplies are on.

#### Turning off the instrument for long term shutdown

To shut down your instrument completely:

#### CAUTION

The ICP-OES gas control unit supplies uninterrupted gas purge to the polychromator assembly during the run and idle states only to minimize the ingress of moisture. It is recommended that both the instrument and gas supplies be left on at all times except during long periods of non-use.

- **1** Follow Steps 1-6 of the idle state procedure.
- 2 Switch off any accessories (where applicable), and then shut down the argon gas supply at the cylinder.
- **3** Switch off the Power at the front lower left of the instrument.
- **4** Switch off the mains power switch on the left side of the instrument. This will turn off the complete instrument as well as the polychromator thermostating system.
- **5** Turn off the laboratory exhaust system.
- **6** Exit the ICP Expert software, if it is no longer required, by choosing **Exit** from the **File** menu. Switch off the printer and monitor.

Restarting the instrument to be ready for analysis again will take several hours due to the gas purge and polychromator thermal stabilization time.

**NOTE** If the instrument is not going to be in use for a period of time the torch, cone, snout and torch compartment should be cleaned of any deposits, dirt or residue. Check the cleanliness of the removable axial window when cone is removed.



## 5. Maintenance and Troubleshooting

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This chapter includes the Agilent 5100 ICP-OES maintenance requirements that may be carried out by an operator. Any maintenance procedures not specifically mentioned in this chapter should be carried out only by Agilent-trained, Agilent-qualified or Agilent-authorized field service engineers.





#### Eye Hazard

Danger to eyes. The plasma is an intense light source. Direct viewing of the light source will cause eye damage. Operators and other unauthorized personnel must NEVER remove the main covers or disable the safety interlock system.



#### **Shock Hazard**

This instrument contains electrical circuits, devices and components operating at dangerous voltages. Contact with these circuits, devices and components can result in death, cause serious injury, or painful electrical shock.





**RF Hazard and Hot surfaces** 

The plasma radiates dangerous levels of radio frequency (RF) energy. Exposure to the RF energy can cause severe skin damage and cataracts of the eyes, while close contact with the operating plasma can result in severe heat burns to the skin, and an electrical discharge which can jump a considerable distance and may cause death, severe electric shock or subsurface skin burns.

#### NOTE

This section refers to maintenance procedures for the ICP-OES instrument. You should refer to your PC and printer manuals for their maintenance procedures, and to the ICP Expert Help for the maintenance procedures for any accessories you ordered.

## **Routine**

The following parts of the ICP-OES, consumables and accessories require routine maintenance. Maintenance instructions are included in the 5100 ICP-OES Familiarization DVD and the ICP Expert Help. To access these instructions, click the Maintenance tab on the DVD or click **Start > All Programs > Agilent > ICP Expert > ICP Expert Help**. Click the **Maintenance** link.

#### Hourly

□ Check and, if necessary, empty the drain vessel.

#### Daily

- Check the water level in the Argon Humidifier before every use (if applicable).
- □ Clean the surface of your ICP-OES (spills should be cleaned up immediately).
- □ Inspect the pump tubing and replace if it is flat or has lost its elasticity. Unclip the pump tubes when the pump is not in use.

#### Weekly

- $\hfill\square$  Clean the torch.
- $\hfill\square$  Clean the cone
- $\Box$  Clean the snout.
- □ Clean the spraychamber.
- □ Clean the nebulizer.

#### Monthly

- □ Inspect the removable axial and radial pre-optics windows for cleanliness. Clean or replace as necessary.
- **Clean** the cooling air intake filter on top of your instrument.
- Inspect the state of the induction coil. Some discoloration is expected, however, extensive discoloration may indicate servicing is required. Contact your local Agilent office or representative if maintenance is required. See the following section for additional information.
- □ Remove and clean the water filter on the right side of the instrument.
- □ Check the water level in the water cooler (refer to the manual supplied with the water cooler for details).
- □ Check/clean the heat exchanger (radiator) on the cooling system to remove any build-up of dust and dirt.
- Periodically, drain the coolant from the cooling system and then refill/ treat with an appropriate algaecide (as recommended by the manufacturer).
- Derform a wavelength calibration.
- Inspect the external gas supply system for leaks including the tubing connected to the instrument. Replace any damaged, leaking or worn components.

## Cleaning

Any spills in the sample compartment should be wiped up immediately.

The user (or other authorized personnel) must perform the appropriate decontamination procedure if hazardous material is spilled on or inside the ICP-OES.

The exterior surfaces of the ICP-OES should be kept clean. All cleaning should be done with a soft cloth. If necessary, this cloth can be dampened with water or a mild detergent. Do not use organic solvents or abrasive cleaning agents.

Before using any cleaning agent, procedure or decontamination method except those specified by Agilent, the user (or other authorized personnel) should check with your local authorized Agilent field service engineer or representative to confirm that the proposed method will not damage the equipment.

## **Torch Cleaning**

#### Acid cleaning of the torch

Hot Surface and Chemical Hazard





The torch and torch compartment become hot during instrument operation and remain hot for some time after the instrument has been switched off. Allow the torch and torch compartment to cool for at least five minutes before attempting to remove the torch. Use heat-resistant gloves. Nitric and hydrochloric acids are very corrosive and can cause severe burns when they come into contact with the skin. It is essential that appropriate protective clothing be worn at all times when handling these acids. If acid contacts the skin, wash off with copious amounts of water and seek medical attention immediately.

See Page 35 for demountable torch disassembly instructions.

#### **Maintenance and Troubleshooting**



**Figure 12.** Torch components where 1. Ball joint, 2. Gas ports, 3. Torch securing ring (demountable torch only), and 4. Torch outer tube

#### To clean the torch:

- 1 Soak the quartz parts of the torch in 50% aqua regia (1 part water to 1 part aqua regia) for at least 1 hour. To make aqua regia combine 1:3 concentrated nitric acid: hydrochloric acid. The length of time required for the cleaning procedure will depend upon the extent of the contamination. Do not leave the torch in the acid for longer than 8 hours.
- IMPORTANTFor one-piece torches: use a clean, wide diameter, open-top beaker or a similar<br/>container to hold the torch inverted while soaking in acid.For two-piece demountable torches:Use a large enough beaker or similar<br/>container to fully submerge the torch quartz tube.<br/>Use clean/particle free acid to perform the soak.
  - **2 One-piece torches:** Place the torch in the vessel so that the acid covers the quartz to just below the plastic base. See Figures 13A and 14. To remove build up from the lower part of the injector, pipette some of the acid through the ball joint of the injector. See Figure 12.

**Two-piece demountable torches:** The quartz outer tube set can be fully immersed in acid. See Figures 13B, 13C and 14. The injector can be inverted and dipped in the acid to just before the plastic base.

#### **Maintenance and Troubleshooting**

## CAUTION

Avoid acid contact with the seal where the quartz meets the plastic base. Damage to the seals and torch body may occur.

3 Keep the one piece torch inverted through all the cleaning and rinsing steps unless otherwise directed.



**Figure 13.** A. Bonded torch in acid and B. demountable torch body with injector in the acid, C. Torch outer tube in acid. A and B placed in an open vessel with acid level just below the plastic base.



**Figure 14.** Close up of torch placed in vessel, highlighting acid level relative to the plastic base

#### **Rinsing the torch**

#### To rinse bonded torches:

- 1 Hold the torch with the ball joint connector at the top.
- 2 Thoroughly flush the inside and outside of the torch with deionized water (18 M $\Omega$ .cm) using a wash bottle to direct the water stream. See Figure 14A and B.
- 3 Invert the torch (see Figure 14C) so that the quartz tubes are at the top and the ball joint connector is at the bottom. Direct rinse water through the quartz tubes so that the water flows out of the gas entry ports and ball joint connector for at least 30 s.



**Figure 15.** A. Rinsing the torch upper gas supply port. B Close-up shows rinsing through the upper gas supply port. C. Torch shown inverted.

#### To rinse demountable torches:

1 Thoroughly flush the inside and outside of the torch quartz components, including through the gas holes, with deionized water (18 M $\Omega$ .cm) using a wash bottle to direct the water stream.

#### Drying the torch

Oven drying is not recommended. It is not as effective at removing moisture as using compressed air or nitrogen.

#### To dry bonded torches:

1 Hold the torch inverted (with the ball joint connector at the top). See Figure 15A.

- **2** Blow clean compressed air or nitrogen through the three gas supply ports (two on the base and through the ball joint connector) to remove moisture.
- **3** Ensure that all moisture is removed before refitting the torch into the instrument.



**Figure 16.** A. Drying the torch upper gas supply port. B Close-up shows drying through the lower gas supply port. C. Close-up shows drying through ball joint.

#### To dry demountable torches:

- 1 Blow clean compressed air or nitrogen through the quartz tube, injector, torch body and the three gas holes (see Figure 16) to remove moisture.
- **2** Ensure that all moisture is removed before reassembling the torch.

#### Additional checks after cleaning

#### Perform the following checks after cleaning:

- 1 Inspect the torch for damage such as loose fitting of the quartz tubes in the plastic base, holes or significant cracks. If any damage is found, replace the torch immediately.
- **2** Check for carry-over after refitting the torch to the instrument to determine if the cleaning procedure has been sufficient. If carry-over is found, repeat the cleaning process.
- **3** Replace the torch when the outer surface of the quartz outer tube is rough to the touch (which indicates signs of wear), or if there are any cracks visible.

## NOTE

Long term exposure to acid during cleaning may lead to discoloration of the plastic base. This change is cosmetic only and should not impact performance if the torch is clean and the results of the other torch checks are satisfactory.

Store the torch in the original box or a plastic bag when not in use.

## Troubleshooting

For troubleshooting information, please see the ICP Expert Help:

- 1 Click Start > All Programs > Agilent > ICP Expert > ICP Expert Help.
- **2** When the ICP Expert Help appears, click **Troubleshooting** to view instructions on how to troubleshoot.

## **Spare Parts**

For spare parts and consumables ordering information, refer to the Agilent Technologies website:

www.agilent.com

To replace the items listed below, you must use Agilentmanufactured parts, which can be ordered online from the Agilent website or through your local sales representative.

The following is a list of recommended spares to keep on hand to minimize downtime during maintenance and repairs:

- Torch
- Air inlet filter (basic)
- Air inlet filter (dust filter)
- Axial pre-optic window
- Radial pre-optic window
- Spray chamber
- Nebulizer
- Peristaltic pump tubing
- Drain tubing

## **Technical Support**

For technical support contact information, refer to the Agilent Technologies website for details:

www.agilent.com

#### www.agilent.com

#### In This Book

The manual describes the following:

- Safety Practices and Hazards
- Introduction
- Installation
- Operation
- Maintenance and Troubleshooting

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