iCAP PRO Series ICP-OES

Operating Manual

BRE0016949 Revision C August 2020

Thermo Fisher SCIENTIFIC

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Legal Notices

Original Operating Instructions

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Contents

Technical Data of iCAP PRO Series ICP-OES Systems

The table summarizes the most important technical data of the iCAP PRO Series ICP-OES systems. See the respective chapters of the manual for details and additional instrument properties.

Table 2-1. Technical data of iCAP PRO Series ICP-OES systems (Sheet 1 of 2)

Parameter	Specification	Value	
Instrument Dimensions			
	Length × width × height	690 mm × 615 mm × 933 mm	
	Weight	80 kg	
Power Requirements			
Instrument	Nominal voltage	200–240 V AC, 50–60 Hz, single phase	
	Power	apparent power: 2.694 kVA effective power: 2.605 kW	
	Wire	2-pole, 3-wire grounding	
	Fuse[a]	16 A (Europe) 20 A (North America, Japan)	
Accessories		Standard mains plug 200–240 V AC, 50–60 Hz, single phase for each accessory	
Operating Environment			
General	Altitude of installation site	≤ 3000 m above sea level	
	Laboratory atmosphere	non-condensing and non-corrosive	
Temperature	Laboratory temperature	15–35 °C	
	Optimum operation temperature	19–25 °C	
	Temperature fluctuations	≤ 2.5 °C/h	
Humidity	Laboratory humidity	20–80% (< 30 °C) 20–60% (≥ 30 °C)	
Exhaust	Optimum exhaust velocity	approximately 5.5 m/s	
	Exhaust volume flow rate	180–220 m^3/h	
Heat		**Dissipation into lab**	**Absorption**
	Instrument	<650 W	
	Recirculation chiller	<1900 W	<900 W
	Extraction vent		<2000 W
Cooling Water Requirements			
Cooling water[b]	Required minimum flow	> 2 L/min	
	Temperature	5 °C below ambient; 10–30 °C	
	pH	6–8	
	Recommended conductivity	< 1000 µS/cm	
	Solid residual	< 50 µm particle size	

Table 2-1. Technical data of iCAP PRO Series ICP-OES systems (Sheet 2 of 2)

Cooling system/Chiller[c]	Maximum pressure	0.6 MPa (6 bar)
	Operating pressure	0.2–0.6 MPa (2–6 bar)
	Cooling capacity	≥ 750 W
Gas Supply		
	Regulators	2-stage regulators on cylinders stainless steel diaphragm recommended
	Shut-off valves	Ball valves recommended (no needle valves)
Plasma Gas[d]		
Argon	Purity	≥ 99.995%
	Maximum water content	< 10 ppm
	Maximum oxygen content	< 10 ppm
	Maximum supply rate	25 L/min
	Operating pressure	0.55–0.6 MPa (5.5–6 bar)
	Pressure range of regulator valve (wall)	0–1 MPa (0–10 bar)
Additional Gas (optional)[e]		
Air, oxygen, argon	Purity	≥ 99.99%
	Maximum water content	< 100 ppm
	Maximum supply rate	1 L/min
	Operating pressure	0.2–0.6 MPa (2–6 bar)
	Pressure range of regulator valve (wall)	0–0.6 MPa (0–6 bar)
Purge Gas		
Argon[d]	Purity	≥ 99.995%
	Maximum water content	< 10 ppm
	Maximum oxygen content	< 10 ppm
	Maximum supply rate	10.2 L/min
	Operating pressure	0.55–0.6 MPa (5.5–6 bar)
	Pressure range of regulator valve (wall)	0–1 MPa (0–10 bar)
Nitrogen[d]	Purity	≥ 99.995%
	Maximum water content	< 10 ppm
	Maximum oxygen content	< 10 ppm
	Maximum supply rate	10.2 L/min
	Operating pressure	0.55–0.6 MPa (5.5–6 bar)
	Pressure range of regulator valve (wall)	0–1 MPa (0–10 bar)

[a] dedicated wall outlet

[b] pipe supply outer diameter is 12 mm.

[c] The ThermoFlex chillers have a water reservoir with a volume of 7.2 L.

[d] Gas tubings to the instrument connect with 6 mm OD push fittings.

[e] Gas tubings to the instrument connect with 4 mm OD push fittings.

1 Using this Manual

Welcome to the Thermo Scientific™ iCAP PRO Series ICP-OES system. The iCAP PRO Series ICP-OES is a member of inductively coupled plasma-optical emission spectrometers (ICP-OES).

Contents

About this Manual

This *iCAP PRO Series ICP-OES Operating Manual* contains precautionary statements that can prevent personal injury, instrument damage, and loss of data if they are properly followed. It also describes the modes of operation and principle hardware components of your iCAP PRO Series ICP-OES instrument. In addition, this manual provides step-by-step instructions for cleaning and maintaining your instrument.

This *iCAP PRO Series ICP-OES Operating Manual* is intended for all personnel that need a thorough understanding of the instrument (to perform maintenance or troubleshooting, for example).

Designed, manufactured and tested in an ISO9001 certified facility, this instrument has been shipped to you from our manufacturing facility in a safe condition. This instrument must be used as described in this manual. Any use of this instrument in a manner other than described here may result in instrument damage and/or operator injury.

Read this manual carefully before using the instrument and keep it for future reference.

Typographical Conventions

This section describes typographical conventions that have been established for Thermo Fisher Scientific manuals.

Signal Words

Make sure that you follow the precautionary statements presented in this manual. The special notices appear different from the main flow of text:

NOTICE Points out possible material damage, data loss, impaired data quality and other important information in connection with the instrument.

Tip Highlights helpful information that can make a task easier.

Viewpoint Orientation

The expressions *left* and *right* used in this manual always refer to the viewpoint of a person that is facing the front side of the instrument.

Data Input

Throughout this manual, the following conventions indicate data input and output via the computer:

- Messages displayed on the screen are represented by capitalizing the initial letter of each word and by italicizing each word.
- Input that you enter by keyboard is identified by quotation marks: single quotes for single characters, double quotes for strings.
- For brevity, expressions such as "choose **File > Directories**" are used rather than "pull down the File menu and choose Directories."
- Any command enclosed in angle brackets < > represents a single keystroke. For example, "press **<F1>**" means press the key labeled *F1*.
- Any command that requires pressing two or more keys simultaneously is shown with a plus sign connecting the keys. For example, "press **<Shift>** + **<F1>**" means press and hold the <Shift> key and then press the <F1> key.
- Any button that you click on the screen is represented in bold face letters. For example, "click **Close**."

Topic Headings

These headings are used to show the organization of topics in a chapter:

Chapter Name

Second Level Topics

Third Level Topics

Fourth Level Topics

Reference Documentation

This *iCAP PRO Series ICP-OES Operating Manual* represents the Original Operating Instructions. In addition to this manual, Thermo Fisher Scientific provides other documents for the iCAP PRO Series ICP-OES that are not part of the Original Operating Instructions.

Reference documentation for the iCAP PRO Series ICP-OES includes the following:

- *iCAP PRO Series ICP-OES Pre-Installation Requirements Guide*

  This guide contains information on the required environmental conditions in the intended location for the instrument.

- *iCAP PRO Series ICP-OES Consumables and Parts Catalog*

  This document lists the part numbers of key consumables and spare part items for the instrument.

- *PC Specifications for Qtegra ISDS Software*

  This document lists the minimum hardware and software requirements of the instrument control PC to ensure the optimum operation of the Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software.

- *Qtegra ISDS Software for iCAP PRO ICP-OES Installation Guide*

  This manual provides reference information about the Qtegra ISDS Software installation process.

- *iCAP PRO Series ICP-OES Software Manual*

  This manual describes the features of the Qtegra ISDS Software.

- *Qtegra ISDS Software Data Processing Algorithms for ICP-OES*

  This software manual provides additional information about the algorithms used for Qtegra evaluations.

You can access PDF files of the documents listed above and of this manual from the data system computer or other electronic storage devices. The Qtegra ISDS Help page provides help with running your instrument, including software guides. A printed version of this *iCAP PRO Series ICP-OES Operating Manual* is shipped with the instrument.

Refer also to the user documentation provided by the manufacturers of third party components, for example:

- Autosamplers
- External chillers

- Data system computer and monitor
- Material Safety Data Sheets (MSDSs)

Contacting Us

There are several ways to contact Thermo Fisher Scientific. In addition to using the browser or email application on your computer, you can scan the QR Code with your smartphone or tablet.

Contact	Link / Remarks	QR Code
Brochures and Ordering Information	www.thermofisher.com/icp-oes	
Service Contact	For technical support related to your instrument or software, visit the **Services & Support** tab at www.thermofisher.com or visit www.unitylabservices.com to find the customer care telephone line or email address for your geographical region.	
EU REACH Statement Health and Safety Form	www.thermoscientific.com/Technicaldocumentation	
Technical Documentation SharePoint	❖ **To get user manuals for your product** 1. With the serial number (S/N) of your instrument, request access on our customer SharePoint as a customer at www.thermoscientific.com/Technicaldocumentation 2. For the first login, you have to create an account. Follow the instructions given on screen. Accept the invitation within six days and log in with your created Microsoft™ password. 3. Download current revisions of user manuals and other customer-oriented documents for your product. Translations into other languages may be available there as well.	
Customer Feedback	❖ **To suggest changes to this manual** You are encouraged to report errors or omissions in the manual. Send an email to the Technical Documentation at documentation.bremen@thermofisher.com. The PDF versions of our manuals allow adding comments with Adobe Acrobat Reader or other freely available PDF reader programs.	

Training

Thermo Fisher Scientific offers worldwide training on instruments and software. Experience has shown that maximum results can be obtained from a scientific instrument if the instrument operator receives an adequate training.

We recommend that the key operator attends training at Thermo Fisher Scientific Bremen (Germany), at your site, or at one of the local Thermo Fisher Scientific offices. For information on training courses and enrollment, contact your local Thermo Fisher Scientific office.

2

System Accessories

In addition to the iCAP PRO Series ICP-OES, some of the system accessories are essential whereas others are optional. This chapter lists the standard components of your system and optional components.

Contents

Essential Accessories

The following accessories are essential for operating your iCAP PRO Series ICP-OES. They can be ordered from Thermo Fisher Scientific, or an equivalent can be supplied by the user:

- Recirculating chiller unit (TF900 Turbine Pump Chiller, or equivalent)
- Data station

Chapter 5, “Installation” contains the required specifications for these accessories.

Optional Accessories

The following accessories are optional:

- Teledyne CETAC™ ASX-100 Autosampler[1]
- Teledyne CETAC ASX-112FR Autosampler[1]
- Teledyne CETAC ASX-1400 Autosampler[1]
- Teledyne CETAC ASX-7400 Autosampler[1]
- Teledyne CETAC ASX-260 Autosampler[1]
- Teledyne CETAC ASX-280 Autosampler[1]
- Teledyne CETAC ASX-520 Autosampler[1]
- Teledyne CETAC ASX-560 Autosampler[1]
- Teledyne CETAC XLR-8 Autosampler[1]
- Teledyne CETAC XLR-860 Autosampler[1]
- Teledyne CETAC SDX_{HPLD} High Performance Liquid Dilution System
- ESI SC-14DX Autosampler
- ESI SC-4DX Autosampler
- ESI SC-4Q Autosampler
- ESI prep*FAST*™ Auto-dilution System
- CETAC U-5000AT+ Ultrasonic Nebulizer
- IsoMist™ XR Programmable Temperature Spray Chamber[2]

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[1] requires dedicated RS232 or USB port on Data Station

[2] requires dedicated USB port on Data Station

- Hydride Generation Accessory
- Argon Humidifier
- High Solids Sample Introduction Kit
- HF Acid Sample Introduction Kit
- Organics Sample Introduction Kit
- Volatile Organics Sample Introduction Kit
- Sheath Gas Adaptor

The above list is subject to change. Contact Thermo Fisher Scientific or a local representative for a list of currently supported accessories. Request site requirement guides for any purchased accessories.

Functional Description

This chapter provides an overview of the functional elements of the iCAP PRO Series ICP-OES.

Contents

Introduction

The Thermo Scientific iCAP PRO Series ICP-OES are a range of inductively coupled plasma-optical emission spectrometers (ICP-OES), which use an echelle optical design and a Charge Injection Device (CID) solid-state detector to measure trace elemental concentrations in a wide range of samples. See Figure 3-1.

Figure 3-1. iCAP PRO Series ICP-OES

Typical samples that are analyzed by ICP-OES are liquids. They are pumped through a nebulizer to produce a fine spray. The large droplets are removed by a spray chamber and the small droplets then pass through to the plasma.

The solvent is evaporated and the residual sample decomposes to atoms and ions. These are excited by the electrical Radio Frequency (RF) generated plasma, which is at a temperature of approximately 9000 K. When they decay to a lower energy state, they emit a set of wavelengths of light that is unique for each element. The intensity of this light is measured and corresponds to the concentration of the element in the original sample.

The iCAP PRO Series ICP-OES consists of several major components:

- Plasma torch and sample introduction parts
- Radio frequency power generator
- Echelle polychromator optical system
- CID detector with thermoelectric cooling
- Interlocks

The control of the spectrometer is provided by the PC-based Thermo Scientific™ Qtegra Intelligent Scientific Data Solution™ (ISDS) Software. To avoid loss of analytical performance and compromising safety, use only Thermo Scientific specified parts.

The following sections describe the specification details.

Instrument Overview

This section provides an overview of the functional elements of the iCAP PRO Series ICP-OES.

Instrument Front Side

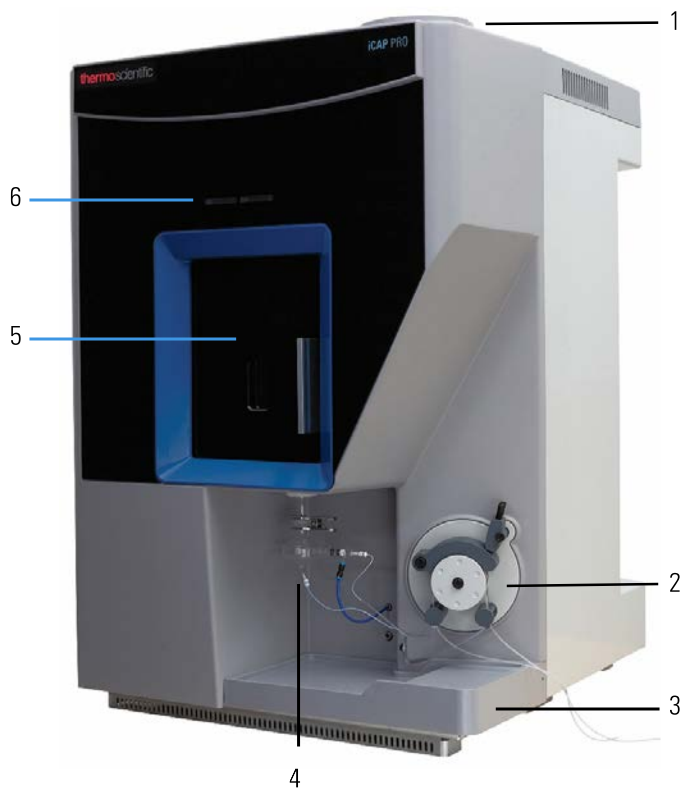


Figure 3-2. Instrument front side

No.	Description	No.	Description
1.	Exhaust port	2.	Peristaltic pump
3.	Drip tray	4.	Sample introduction
5.	Door	6.	System status LEDs

An exhaust port is located at the top of the instrument. See pos. 1 in Figure 3-2.

The drip tray protects the instrument from minor spillage from the sample introduction system. See pos. 3.

A sliding door gives access to the torch box. See pos. 5. The window in the door allows viewing the plasma if it is ignited.

Two LEDs indicate the statuses of the instrument (left) and the active analysis (right). See pos. 6.

Table 3-1. Available statuses of System LEDs

LED Color	Information
Instrument Status	
Green	Plasma is on and instrument is ready. For maximum five minutes, the system checks the temperature but does not indicate a Detector Temperature interlock to allow for reaching the required temperature. If the temperature is not reached after that time, the plasma is switched off, Get Ready fails, and the LED is flashing red.
Green flashing	Instrument is starting up, warming up, or performing Get Ready.
Blue	Instrument is in standby.
Blue flashing	Plasma if off and glassware is cooling down.
Red flashing	Hardware interlock is active.
Analysis Status	
Blue	LabBook acquisition is being initialized.
Blue flashing	LabBook is running.
Green[a]	LabBook is successfully completed.
Red[a]	LabBook has finished with error.
Off	No LabBook was run since the instrument startup.

[a] The instrument keeps this LED status during standby.

Tip If an interlock causes an instrument error, the Qtegra ISDS Software shows a message below the source settings panel that describes the issue and gives a possible solution.

Instrument Rear Side

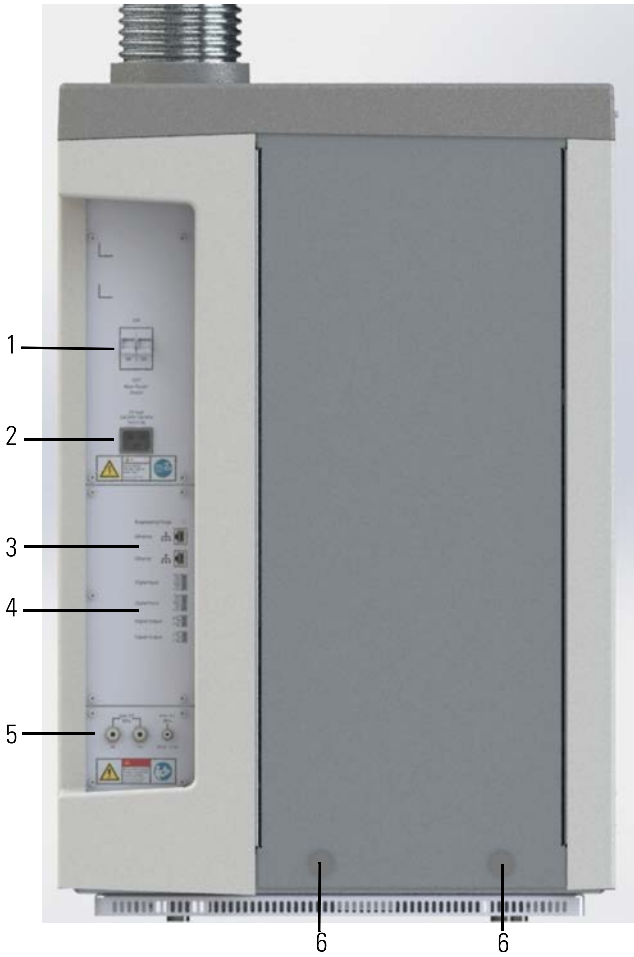


Figure 3-3. Instrument rear side

No.	Description	No.	Description
1.	Power switch	2.	Power connector
3.	Ethernet ports	4.	Digital input/output
5.	Gas ports	6.	Spacers

The instrument is powered with an on/off switch on the left part of the rear side. See pos. 1 in Figure 3-3.

The instrument is equipped with a standard IEC 60320 C20 socket for the power cord. See pos. 2.

An Ethernet port for the communication of the spectrometer with the data station and a second Ethernet port for the communication with peripheral instruments are located below the power socket. See pos. 3.

The iCAP PRO ICP-OES provides two digital input ports and two digital output ports for the communication with external devices. See pos. 4.

The external gas lines connect to the three ports (Ar, N_2, additional gas) at the bottom of the main connections panel at the rear side of the instrument. See pos. 5.

Two spacers provide sufficient clearance for the gas and water lines and electrical connections, as well as for the connections to the computer and peripheral devices between the instrument and the wall. See pos. 6.

Left Instrument Side

Figure 3-4. Instrument left side

The hoses (blue for inlet and black for outlet) for the cooling water connect to push fit connectors on the left side of the instrument. See Figure 3-4.

Right Instrument Side

Figure 3-5. Instrument right side

No.	Description	No.	Description
1.	Exhaust port	2.	Peristaltic pump
3.	Drip tray		

Instrument Specification

Table 3-2 gives an overview of the characteristic properties of the available configurations of the iCAP PRO Series ICP-OES. The following sections provide detailed information.

Table 3-2. Specifications for iCAP PRO Series ICP-OES

Parameter	iCAP PRO	iCAP PRO X	iCAP PRO XP	iCAP PRO XPS
Dimensions (l × w × h)	690 × 615 × 933 mm			
Weight	80 kg			
Peristaltic pump	3 Channel, standard peristaltic pump pump speed: 0 rpm or 45 rpm		4 Channel, standard peristaltic pump pump speed: 0–125 rpm	
Bubble sensor	✓			
Plasma gas	Fixed, 12.5 L/min; 0 L/min only when plasma off		8–20 L/min; 0 L/min only when plasma off	
Auxiliary gas	Fixed: aqueous 0.5 L/min, organic 1.5 L/min; 0 L/min only when plasma off		0.4–2 L/min; 0 L/min only when plasma off	
Nebulizer gas	Fixed: 0 L/min or 0.6 L/min	0–1.2 L/min		
Additional gas	—		0–0.25 L/min	
Plasma viewing	Duo or Radial			
RF source	27.12 MHz solid state			
Power output	1150 W or 1350 W	750, 1150, 1350 W	Duo: 750–1400 W Radial: 750–1600 W	
Ceramic torch	—		Radial only	
Spectral bandpass	7 pm at 200 nm			
Wavelength range	167–852 nm			
Measuring time	3–4 min	2–3 min	1–2 min	0.5–1 min

Optical System

The dispersive elements in the optic system are the echelle grating and the prism. The orientation of the prism is such that the light is dispersed at right angles to the direction of light dispersal by the grating. This combined dispersal generates a two dimensional spectrum (“echellogram”) consisting of a wavelength and order separation. The CID detector is cooled to -45 °C to increase sensitivity and dynamic range.

Optical System Specifications

Table 3-3. Optical system specifications

Specification	Comment
Type	High energy echelle cross dispersion optical system with “side by side” optical arrangement of prism and grating
Spectrometer optical path	The entire spectrometer and fore optics are purged with either argon or nitrogen. A normal running flow of 2 L/min is used. A purged environment is maintained with a standby flow of 0.2 L/min when the plasma is extinguished.
Spectral bandpass	7 pm at 200 nm
Wavelength coverage	The iCAP PRO Series ICP-OES has a wavelength range from 167 nm to 852 nm.

Detector Specifications

Table 3-4. Detector specifications

Specification	Comment
Type	High performance solid state Charge Injection Device (CID) camera system The CID is an enhanced charge-transfer-device, which delivers high contrast/low noise imaging and quantification of all wavelengths in the analytical range without blooming.
Pixel size	12 × 12 µm
Detector cooling	Peltier cooling device, maintains the detector at a constant temperature of -45 °C. Interlocks for purge gas and cooling water prevent possible damage if the services fail.

Detector Mode

Random Access Integration (RAI): user-selected analytical wavelengths are simultaneously integrated in a manner whereby the signal to noise ratio is optimized while the photo-generated charge level is maintained in the linear range of the CID.

This is accomplished by utilizing the unique nondestructive readout (NDRO) capability only available with a CID. NDRO allows for the measurement of the signal level on any pixel at any point in the exposure. In this manner, the readout frequency is varied from pixel to pixel based on the real time observation of the emission intensity.

The major advantage of the CID's pixel-to-pixel-based real time observation is that the optimum signal-to-noise ratio is achieved for any wavelength anywhere on the detector while maintaining wide dynamic range for all signals.

Plasma Viewing Specifications

Table 3-5. Plasma viewing specifications

Specification	Comment
Dedicated radial plasma	The plasma is viewed directly in a radial mode using high efficiency mirrors. The entrance optics are housed in a purged enclosure that provides corrosion resistance and enhanced performance in the UV region of the spectrum. To maximize performance, the operator can select the viewing height.
Duo view plasma	The plasma may be viewed axially for applications requiring low detection limits or radially to minimize matrix effects. Radial signals are measured by an auxiliary optical path that collects light from an aperture in the side of the torch and images it on the entrance optics. The orientation of the plasma view can be set in the method and is completely automatic. Available viewing options are axial and radial, and can be selected for individual wavelengths by the operator.

Plasma Source Specification

Table 3-6. Plasma source specifications

Specification	Comment
Type	Inductively coupled argon plasma using the internationally approved Industrial Scientific and Medical (ISM) radio frequency bands. Solid state RF generator with power efficiency above 78%.
Nominal frequency	27.12 MHz
Operation	Plasma ignition and operation are fully automated and software controlled. Directly coupled autotune with swing frequency impedance control. Power regulation better than 0.1%.

Sample Introduction Specifications

Table 3-7. Sample introduction specifications

Specification	Comment
Nebulizer	Glass concentric fitted as standard **Optional**: Aerosalt (high solids concentric), V-groove, Mira Mist (HF resistant), Ultrasonic nebulizer
Spray chamber	Glass cyclonic type fitted as standard **Optional**: IsoMist™ XR temperature controlled spray chamber, baffled cyclonic spray chamber, HF-resistant cyclonic spray chamber
Sample pump	High precision, 12 roller, 3-/4-channel standard peristaltic pump with fixed/adjustable pump speed depending on the instrument configuration

Table 3-7. Sample introduction specifications, continued

Specification	Comment
Torch	Semi-demountable quartz torch fitted with • either a 1.5 mm center tube (Radial instruments) or • 2 mm center tube (Duo instruments) The torch design includes a quick release, prealigned mounting block, which does not require tools for removal. The mount incorporates automatic torch gas connections making removal and replacement a very simple operation.
Center tubes	Optional 1.0, 1.5, and 2.0 mm quartz center tubes; 2.0 mm HF resistant center tubes
Gas control	All gases are MFC-controlled. Flows are either fixed or tunable, depending on the instrument configuration.

Sample Introduction System

When a sample is aspirated, a proportion of the aerosol generated by the nebulizer is passed to the plasma through the torch assembly. The aerosol generates a load on the plasma causing a change in the plasma conditions. The RF power, other plasma conditions, and the sample introduction system should be optimized for the particular sample and solvent type being introduced to compensate for these changes.

Tip Make sure that you are using the appropriate tubing, spray chamber, nebulizer, center tube, etc. for your application. It is also important that your sample introduction system is assembled and maintained properly.

Center Tube Options

One of the options to change the characteristics of the sample reaching the plasma is to use a different center tube in the torch. Table 3-8 and Figure 3-6 show the available options.

Table 3-8. Torch center tube options

Option	Comment
1.0 mm quartz (double red ring)	Used for organic solvent analysis, with Radial and Duo spectrometers. This is to reduce the amount of sample reaching the plasma because larger center tubes result in too much sample reaching the plasma and the plasma might go out.
1.5 mm quartz for aqueous solutions (single red ring)	Standard on Radial configurations
2.0 mm quartz for high dissolved solids solutions (single blue ring)	Standard on Duo configurations
2.0 mm ceramic for HF solutions	Used for specific sample types (for example, hydrofluoric acid digests)

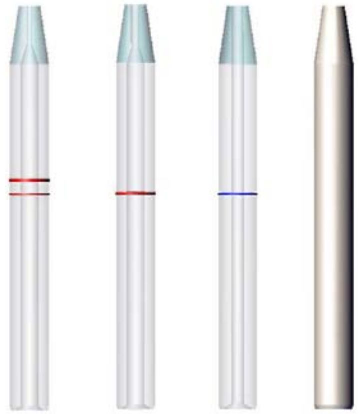


Figure 3-6. Torch center tube options

Nebulizer Options

Various nebulizer options are available for the iCAP PRO Series ICP-OES. The use of each type is application- and method-specific.

Control of the nebulizer pressure or flow is achieved either through the control software or by a manual adjustment. The control is specific to the model of the iCAP PRO Series ICP-OES.

Pump Tubing Options

The iCAP PRO Series ICP-OES requires pump tubing with two clamping points (bridges). See Table 7-2 on page 7-11 for the available tubing material.

When the appropriate peristaltic pump tubing has been selected and configured, it is necessary to optimize the speed of the pump. This ensures optimum sample delivery to the nebulizer and drainage of the spray chamber during instrument operation.

Typically, the peristaltic pump is operated at a speed of approximately 50 rpm when using an iCAP PRO Series ICP-OES. If you intend to analyze samples containing high concentrations of dissolved solids or volatile organic constituents, it may be necessary to reduce the pump speed to between 35–45 rpm.

Using a reduced peristaltic pump speed reduces the sample loading in the plasma. It may therefore contribute to an improved plasma processing efficiency and a resultant analytical performance.

NOTICE Operating the peristaltic pump at low speeds (typically below 35 rpm) might lead to pulsing of the analytical signal. This might compromise the associated internal precision of the measurements.

Internal Standards Mixing Kit

An internal standard is a reference element that can be used to correct for changes in signal intensity caused by external factors. By definition, it should not occur naturally in the sample, but it is added to compensate for sampling differences. It must behave the same as other elements requiring analysis in the sample.

The use of internal standards is not required for all types of analysis, but it is typically employed when the sample loading of the plasma might vary. This is often caused by differing physical properties of the sample (for example, viscosity, dissolved solids, surface tension, or volatility).

Rather than adding an internal standard manually to all the solutions to be analyzed, it can be added automatically on-line with the Internal Standards Mixing Kit. See Figure 3-7.

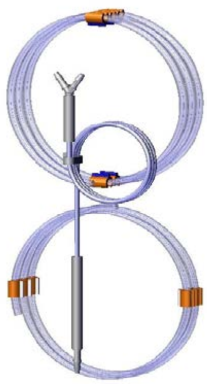


Figure 3-7. Internal Standards Mixing Kit

To minimize the sample dilution, the tubing for the internal standard solution has a smaller bore than that of the sample solution. To ensure the thorough mixing of the internal standard with the sample, a mixing loop is provided after the Y-piece before connecting to the nebulizer. The pump tubing for the Internal Standards Mixing Kit should be installed in a similar manner to the sample tube as described in Chapter 5, “Installation.”

Organic Solvent Analysis

When an organic solvent rather than an aqueous solvent is used, the lower boiling point of the solvent leads to more sample and matrix loading of the plasma. This might impair the analysis, or even extinguish the plasma.

⚠ Organic solvents are a fire hazard and no buildup is permitted in the sample introduction system or its vicinity. Therefore, a specific sample introduction system should be used for organic samples.

Light Organic Samples

The organic sample spray chamber has a baffle tube inside. This reduces the sample aerosol density transported to the plasma. An organic center tube is also used. See Figure 3-8.

Figure 3-8. Baffled cyclonic spray chamber

Heavy Organic Samples

In addition to the sample introduction options for light organic samples, a quartz V-groove nebulizer is essential for heavy organic samples. The V-groove nebulizer should be configured in the cyclonic spray chamber with the gas connection pointing vertically downwards. The orientation of the nebulizer is essential to enable successful sample aspiration. See Figure 3-9.

Figure 3-9. V-groove nebulizer for heavy organic sample analysis

Tip This design of nebulizer does not freely aspirate. Flow rates must be carefully controlled by adjusting the pump speed as described in "Pump Tubing Options" on page 3-15.

Volatile Organic Samples

The analysis of organic samples that exhibit a high vapor pressure may require a cooled spray chamber to control the sample loading of the plasma. An efficient and flexible approach to ensure control over the spray chamber temperature is to use a programmable temperature controlled device such as a Peltier unit.

The IsoMist™ XR programmable temperature control device from Glass Expansion can be used effectively with the iCAP PRO Series ICP-OES for applications with volatile organic solvents. See Figure 3-10.

Figure 3-10. IsoMist™ XR - temperature controlled spray chamber

High Dissolved Solid Samples

A standard nebulizer may be used in a wide range of applications. However, samples containing levels of dissolved solids greater than 2–3% m/v might cause the standard nebulizer and the torch center tube to block. To prevent the nebulizer from blocking, these sample introduction accessories are available:

- Argon Humidifier
- Aerosalt Nebulizer
- Parallel Path Nebulizer

To prevent the torch center tube from blocking, a sheath gas adaptor can be used. See page A-11 for details.

Argon Humidifier Accessory

The ESI *pergo* Argon Humidifier is designed to be used when analyzing high concentrations of dissolved solids. The humidifier uses Nafion™ tubing that selectively permeates water vapor through its membrane, humidifying the argon gas used for the nebulizer. The water vapor prevents salt from forming deposits in the nebulizer and allows uninterrupted analysis and operation with no maintenance for extended periods of time. See Figure 3-11.

Figure 3-11. *pergo* argon nebulizer gas humidifier (source: ESI)

Humidified argon permits using a standard nebulizer for samples with a dissolved solid content of up to approximately 5% m/v. The ESI *pergo* is connected in line with the argon nebulizer gas. For information on installation and maintenance of the ESI *pergo* Argon Humidifer, refer to the instructions of the supplier.

Aerosalt Nebulizer

Samples containing dissolved solids above approximately 5% m/v require an Aerosalt nebulizer. A high solids center tube and an argon humidifier should also be used.

Parallel Path Nebulizer

Above 15% m/v dissolved solids in a sample require a parallel path nebulizer (V-groove nebulizer or Mira Mist nebulizer, for example). A high solids center tube and a sheath gas adaptor (see page A-11) should also be used.

HF Samples

For certain applications, hydrofluoric acid (HF) has to be used to dissolve a sample. HF reacts with and dissolves the standard sample introduction glassware of an iCAP PRO Series ICP-OES. Most of the glassware has to be replaced with optional HF resistant components. Replacing the components follows the same procedure as detailed for the standard sample introduction parts. These components replace the standard equipment (see Figure 3-12):

- Ceramic center tube
- HF resistant nebulizer
- HF resistant spray chamber with spray chamber adapter

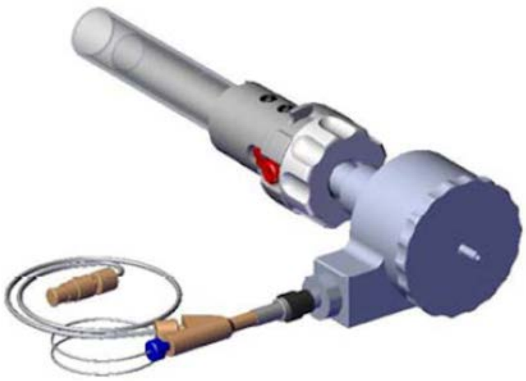


Figure 3-12. HF sample introduction configuration

4

Safety

This chapter provides information about machine safety. For your own safety, the safety of others and to prevent damage to the instrument, it is important that this chapter is carefully read and understood before installing, operating or coming into contact with the instrument and its accessories.

⚠ To comply with safety and warranty requirements, the instrument and accessories described in this manual are designed to be used by properly trained personnel only.

Any installation, adjustment and repair of this equipment must be carried out only by a certified Thermo Fisher Scientific service representative who is aware of the hazards involved.

To protect our operating personnel, we ask you to adhere to special precautions when you send back parts to the factory for exchange or repair. See “Returning Parts” on page 8-11.

Contents

Safety Symbols and Signal Words in this Manual

Notices concerning the safety of the personnel who operate the iCAP PRO Series ICP-OES appear different from the main flow of text. Safety notices include the following:

⚠ Always be aware of what to do with, and the effect of, safety information.

⚠ CAUTION — Points out a hazardous situation that can lead to minor or medium injury if it is not avoided.

⚠ WARNING — Points out a hazardous situation that can lead to severe injury or death if it is not avoided.

⚠ DANGER — Points out a hazardous situation that will lead to severe injury or death if it is not avoided.

Observing this Manual

Keep this manual always near the instrument to have it available for quick reference.

⚠ Before you operate your iCAP PRO Series ICP-OES system, read and understand all the safety information described in this manual.

Be sure to read and comply with all precautions described in this manual.

System configurations and specifications in this manual supersede all previous information received by the purchaser.

Safety Symbols on the Instrument

Table 4-1 lists and explains the safety labels on the instrument and their respective positions. See the indicated safety notices to prevent harm to the operator and to protect the instrument against damage. If they are present, read and obey the instructions on the labels.

The safety labels in the torch box become visible after opening the door at the front side of the instrument.

Table 4-1. Safety labels on the instrument

Label	Description	Position
	**Electric Shock Hazard.** Hazardous electric voltage capable of causing personal injury is used in the instrument. To make sure that the instrument is free from all electric current, always disconnect the power cord of the instrument before you try any type of maintenance.	Rear side of instrument, see Figure 5-13 on page 5-18.
	**Read and fully understand operator's manual before you use this machine.** Failure to follow operating instructions could result in death or serious injury.	Rear side of instrument, see Figure 5-14 on page 5-22.
	**Heavy Object. Can cause back injury during transportation.** See manual for instructions.	Top side of instrument, see "Moving the Instrument" on page 5-3.

Name Plate

To correctly identify the instrument when you contact Thermo Fisher Scientific, always have the information from the name plate (also called rating plate) available. The rating plate is attached to the left side of the rear panel. It contains the serial number, which is important in any type of communication with Thermo Fisher Scientific. See Figure 4-1 and Figure 4-2 as examples. Especially, the serial number is needed to access the SharePoint of the Bremen Technical Documentation group. See "Contacting Us" on page 1-6.

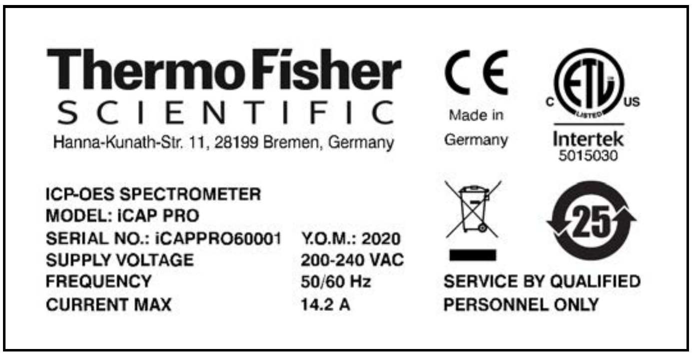


Figure 4-1. Name plate (example)

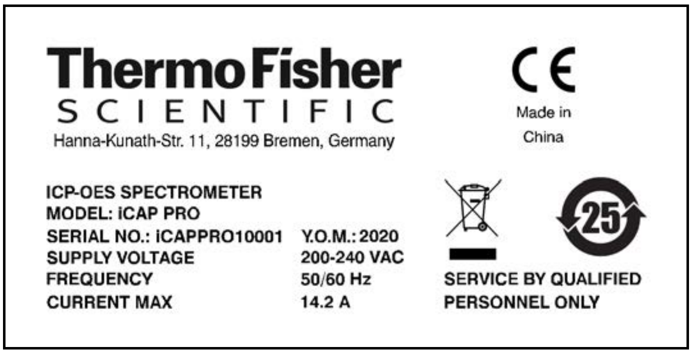


Figure 4-2. Name plate (example)

Intended Use

The iCAP PRO Series ICP-OES are a range of inductively coupled argon plasma optical emission spectrometers (ICP-OES). They use an echelle optical design and a Charge Injection Device (CID) solid-state detector to measure trace elemental concentrations in a wide range of samples.

⚠ Follow the following guidelines when you operate your iCAP PRO Series ICP-OES system:

- The instrument is designed to be placed on a bench in the laboratory. It is not designed for the use outdoors.
- The instrument is designed for laboratory research use only. It is not designed for use in diagnostic or medical therapeutic procedures.

If the iCAP PRO Series ICP-OES system is used in a manner not specified by Thermo Fisher Scientific, the protection provided by the instrument could be impaired. Thermo Fisher Scientific assumes no responsibility and will not be liable for instrument damage and/or operator injury.

Notice on the Susceptibility to Electromagnetic Transmissions

Your instrument is designed to work in a controlled electromagnetic environment. It has been tested in accordance to DIN EN 61326-1 (emission and susceptibility).

Qualification of the Personnel

The primary audience for this manual consists of analytical chemists and laboratory technicians. To use this manual effectively, you should have a basic knowledge of chemistry, a basic knowledge of electronic sampling equipment, at least a beginning level of computer experience, and working knowledge of the analytical instrument used with the sample introduction system.

⚠ Only certified Thermo Fisher Scientific service representatives are allowed to install the iCAP PRO Series ICP-OES system. Personnel that install or operate the iCAP PRO Series ICP-OES system must have the following qualifications:

- **Electrical Connections**
  The electrical installation must be carried out by qualified and skilled personnel (electrician) according to the appropriate regulations (for example, cable cross-sections, fuses, earth grounding connection). Refer to the *iCAP PRO Series ICP-OES Pre-Installation Requirements Guide* for the specifications.

- **Installation**
  Only employees of Thermo Fisher Scientific or personnel who act on behalf of Thermo Fisher Scientific are allowed to install iCAP PRO Series ICP-OES systems.

- **General Operation**
  The iCAP PRO Series ICP-OES system is designed to be operated by qualified laboratory personnel. Before they start, all users must be instructed about the hazards presented by the instrument and the chemicals applied. The users must be advised to read and obey the relevant Material Safety Data Sheets (MSDSs).

- **Decommissioning**
  Only qualified employees of Thermo Fisher Scientific or qualified personnel who act on behalf of Thermo Fisher Scientific are allowed to decommission the instrument.

- **Disposal**
  It is your responsibility to dispose of contaminated parts complying with legal regulations.

  Only qualified employees of Thermo Fisher Scientific or qualified personnel who act on behalf of Thermo Fisher Scientific are allowed to dispose of the instrument.

Electric Safety Precautions

⚠ **WARNING**

High Voltage. High voltages capable of causing an electric shock are used in the instrument.

Observe the following safety precautions when you operate or perform service on your instrument.

- The instrument is properly grounded in accordance with regulations when shipped. You do not need to make any changes to the electrical connections or to the chassis of the instrument to ensure safe operation.
- Do not run the system without the housing on. Permanent damage can occur. When you leave the system, make sure that all protective covers and doors are properly connected and closed, and that heated areas are separated and marked to protect unqualified personnel. Do not rig or override any safety switches or safety functions. Risk of electric shock, burn hazard or damage to your system can occur.
- Do not turn on the instrument if you suspect that any kind of electrical damage has incurred. Instead, disconnect the power cord of the instrument and contact a Thermo Fisher Scientific field service engineer for a product evaluation. Do not try to use the instrument until it has been evaluated. Electrical damage might have occurred if the system shows visible signs of damage or has been transported under severe stress.
- Do not place any objects upon the instrument—especially not containers with liquids—unless it is requested by the user documentation. Leaking liquids might get into contact with electronic components and cause a short circuit.
- If liquid is spilled on or adjacent to the instrument, immediately isolate the instrument and the accessories from the electrical supply by turning off the power remote to the instrumentation.

Impaired Safety Protection

When safety protection has been impaired, the instrument and the accessories must be made inoperative and secured against any unintended operation. The matter should then be referred to the local Thermo Fisher Scientific service organization. Safety protection is likely to be impaired, if the instrument fails to operate normally or shows visible damage. If the equipment is used in a manner that is not specified by the manufacturer, the safety protection provided by the equipment might be impaired.

In Case of Emergency

⚠ The power plug must be accessible easily and at all times to quickly separate the instrument from the mains in case of emergency.

⚠ CAUTION

UV Radiation. Risk of severe bodily harm. UV radiation might lead to severe eye injury or blindness. Do not look into the plasma if this is unexpectedly possible.

UV radiation is released when the spray chamber is disassembled. Do not open the door nor disassemble the spray chamber when the plasma is still on.

Use only the correct Thermo Scientific part for the spray chamber adapter. Do not bypass the safety interlocks. Shut down the system as described on page 6-14. Do not use the door to shut down the system.

❖ To shut down the system in case of emergency

1. At the rear panel of the spectrometer, put the power switch to the **Off** position. See Figure 4-3.

   All power to the spectrometer is shut off.

Figure 4-3. Power switch of iCAP PRO Series ICP-OES

2. Switch off the computer, the recirculating chiller, and other present accessories (an autosampler, for example) with their respective On/Off switches.

⚠ WARNING

Electric Current. Electric shock hazard. Capacitors inside the instrument might still be charged for some time even if the instrument is turned off.

Residual Hazards

Users of the iCAP PRO Series ICP-OES system must pay attention to the following residual hazards.

⚠ **WARNING**

Toxic Gases. Risk of poisoning. Toxic gases are released that might lead to severe bodily harm when the plasma exhaust is not connected to an exhaust system or the spray chamber is disassembled.

Do not operate the instrument without an effective fume extraction system attached to the torch box chimney. Do not disassemble the spray chamber when the plasma is still on.

⚠ **WARNING**

Suffocation Hazard. Nitrogen and argon gas might cause suffocation if they accumulate in the laboratory. Make sure that the laboratory is well ventilated. If the extraction is turned off, sufficient air must be supplied to prevent the concentration of these gases from reaching a harmful level.

⚠ **CAUTION**

Sharp Edges. Risk of cuts. Follow appropriate care and safety procedures to avoid breaking any glassware and causing injury to the operator. Handle any broken glassware with appropriate care and wear protective gloves.

⚠ **CAUTION**

Pinch Point Hazard. Risk of injuries. The peristaltic pump rollers can pull in hair, clothing, ties and other loose objects. Stay clear from the peristaltic pump during operation.

⚠ **CAUTION**

Hot Surface. Touching hot parts of the system might cause severe burns. Let any hot components cool to room temperature (wait at least 10 minutes) before you remove them from the torch box.

⚠ **CAUTION**

UV Radiation. Risk of severe bodily harm. UV radiation might lead to severe eye injury or blindness. Do not look into the plasma if this is unexpectedly possible.

UV radiation is released when the spray chamber is disassembled. Do not open the door nor disassemble the spray chamber when the plasma is still on.

Use only the correct Thermo Scientific part for the spray chamber adapter. Do not bypass the safety interlocks. Shut down the system as described on page 6-14. Do not use the door to shut down the system.

⚠CAUTION

Hazardous Chemicals. Samples and solvents might contain toxic, carcinogenic, mutagenic, or corrosive/irritant chemicals. Avoid exposure to potentially harmful materials.

Always wear protective clothing, gloves, and safety glasses when you handle solvents or samples.

Contain waste streams and use proper ventilation. Refer to your supplier's Material Safety Data Sheet (MSDS) for proper handling of a particular compound.

NOTICE

To ensure safety and proper cooling, always operate the instrument with its covers in place. This is also necessary to comply with product safety and electromagnetic interference regulations. No user serviceable parts are inside the instrument.

Personal Protective Equipment

Appropriate safety clothing must be worn at all times while operating the instrument, particularly when handling hazardous material. This manual can only give general suggestions for personal protective equipment (PPE), which protects the wearer from hazardous substances.

Refer to the Material Safety Data Sheets (MSDSs) of the chemicals handled in your laboratory for advice on specific hazards or additional equipment.

Obey local laws and regulations regarding PPE for radioactive samples.

Eye Protection

The type of eye protection required depends on the hazard. For most situations, safety glasses with side shields are adequate. Where there is a risk of splashing chemicals, goggles are required.

Protective Clothing

When the possibility of chemical contamination exists, protective clothing that resists physical and chemical hazards should be worn over street clothes. Lab coats are appropriate for minor chemical splashes and solids contamination, whereas plastic or rubber aprons are best for protection from corrosive or irritating liquids.

Gloves

For handling chemical compounds and organic solvents, Thermo Fisher Scientific recommends white nitrile clean room gloves from Fisher Scientific or Unity Lab Services.

For handling hot objects, gloves made of heat-resistant materials (for example, leather) should be available.

Safety Boots and Shoes

Safety boots and shoes, which are penetration-resistant and feature protective toecaps, can protect employees from falling objects, vehicles and other heavy loads (for example, when moving the instrument).

5

Installation

This chapter describes the conditions for an operating environment that ensures continued high performance of your iCAP PRO Series ICP-OES system. Before installation, make sure that the proposed area is compatible with the conditions specified. Carry out a comprehensive risk assessment that is specific to the handling of solvents, samples and sample preparation.

To be sure that your laboratory is ready for the installation of the iCAP PRO Series ICP-OES, you must meet all requirements specified in the *iCAP PRO Series ICP-OES Pre-Installation Requirements Guide*. This guide also provides comprehensive information to assist in planning and preparing your lab site.

NOTICE It is your responsibility as the customer to provide a suitable location, a source of power of acceptable quality, a suitable operating environment, the proper quality of consumables and a proper exhaust system.

⚠ To comply with safety and warranty requirements, the iCAP PRO Series ICP-OES, accessories and associated equipment must be installed by a certified Thermo Fisher Scientific service representative.

The installation of all services must comply with the appropriate rules and regulations required by the local authorities responsible for those services in the workplace.
A Thermo Fisher Scientific field service engineer is not responsible for the fitting or compliance of the facilities or services.

Contents

Placing the System

This section provides information that helps you with positioning the system in the laboratory.

Spectrometer Dimensions

Table 5-1. Dimensions and weight of iCAP PRO Series ICP-OES

Length	Width	Height	Weight
690 mm	615 mm	933 mm	80 kg

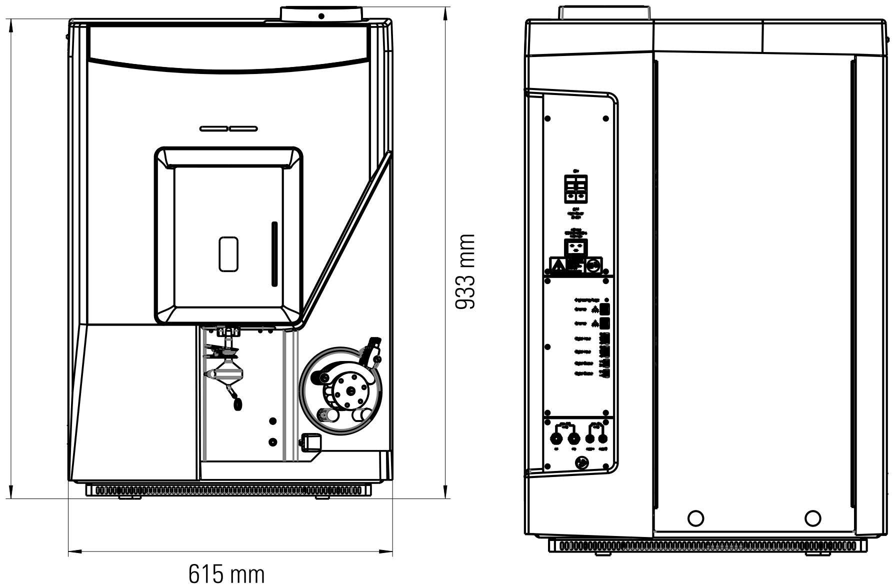


Figure 5-1. Dimensions of iCAP PRO ICP-OES - front view (left) and rear view (right)

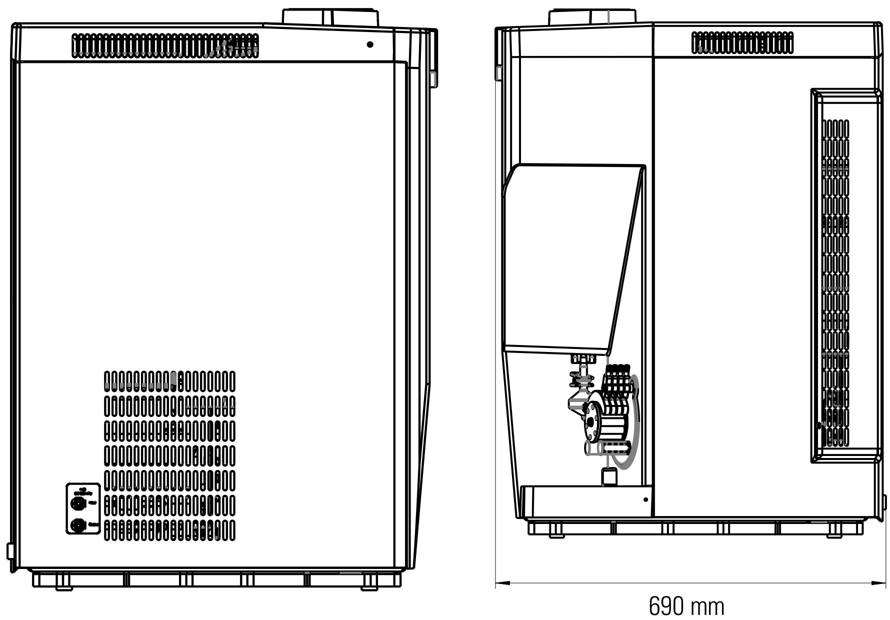


Figure 5-2. Dimensions of iCAP PRO ICP-OES - left view (left) and right view (right)

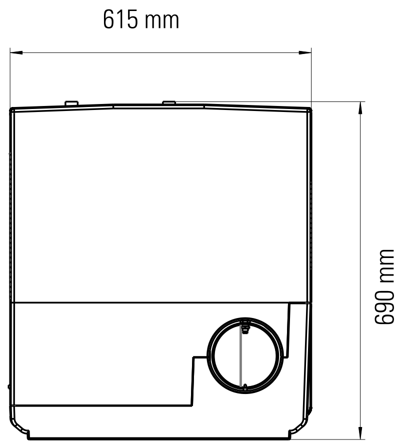


Figure 5-3. Dimensions of iCAP PRO ICP-OES - top view

The floor of your laboratory must be able to accommodate the weight of the instrument including all components and any options that are added to the system.

Make sure that you have access to the left and right side of the installed instrument.

Moving the Instrument

All items should be transported to the installation site in their original packaging and the right way up. A means of transport (a pallet jack, for example) will be required for in-house transportation of the instrument in its shipping container and additional components.

The iCAP PRO Series instrument is equipped with four shell-type handles within its base plate: two long handles on the right side and two on the left side of the base plate allow two persons to lift the unpacked instrument safely. The arrows in Figure 5-4 point to the shell-type handles in bottom view.

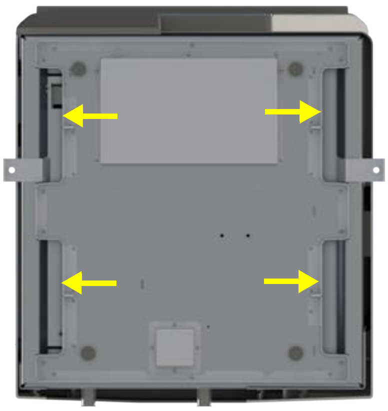


Figure 5-4. Position of shell-type handles - bottom view

⚠ CAUTION

Heavy Load. Because of its weight of 80 kg, handling the instrument alone might cause muscle strain and back injury. Lifting and moving the iCAP PRO Series instrument requires the effort of at least two persons to keep the individual load within acceptable limits (maximum 40 kg for men or 15 kg for women for a duration of five seconds).

The carriers must be trained in how to carry loads properly (for example, by rising from the knees with a straight back) to move the instrument onto the workbench.

⚠ CAUTION

Top Heavy Load. The instrument might tip over while being moved in the laboratory and cause bodily injury or instrument damage. The instrument has a tendency to tilt forward. When lifting the instrument, support the top of the instrument and keep the tilt angle below 5°. Do not exceed 10°.

Figure 5-5 shows the center of gravity of the instrument in front view (left) and in top view (right).

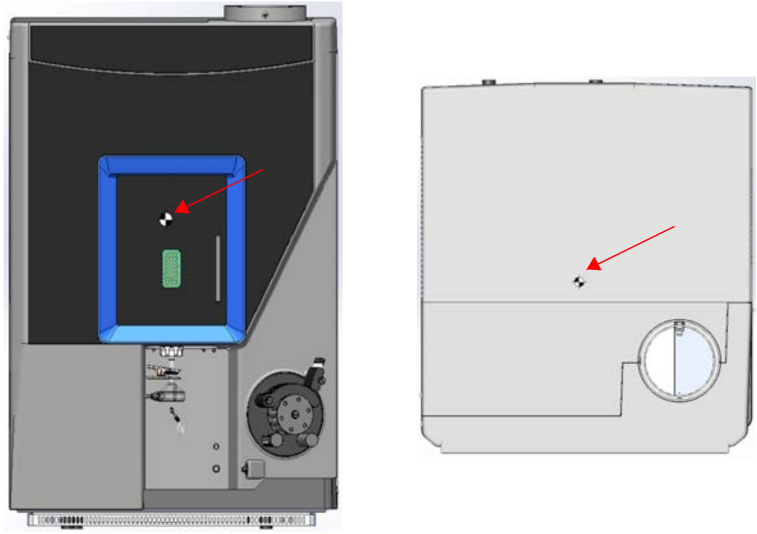


Figure 5-5. Center of gravity in front view and top view

Workbench for the Instrument

The iCAP PRO Series instrument is designed to be placed on a bench with its rear panel against a wall. The bench surface should ideally be 80–85 cm above the floor.

The workbench for the iCAP PRO Series system must be capable of supporting the weight of the spectrometer (80 kg) plus the weight of any peripherals (autosampler, for example) and stand in a secure and level position.

> ⚠ Use only workbenches that are able to carry the weight of the iCAP PRO Series instrument and provide sufficient stability. The workbench must be dry and clean.

To allow shutting off the instrument in an emergency, free access must be possible at any time to the power connections at the left rear side of the instrument. See Figure 5-6.

Two spacers at the rear side of the iCAP PRO Series instrument guarantee sufficient clearance for the gas and water lines and electrical connections, as well as for the connections to the computer and peripheral devices. Owing to these spacers, the instrument can be pushed all the way towards the wall until it makes contact. See Figure 5-6.

Figure 5-6. iCAP PRO Series instrument in rear side view

No.	Description	No.	Description
1.	Power switch	2.	Power connector
3.	Spacers		

Floor Space for the System

Figure 5-7 shows an example of the typical required floor space for the system (instrument with autosampler, monitor and keyboard). An autosampler is not shown for simplicity.

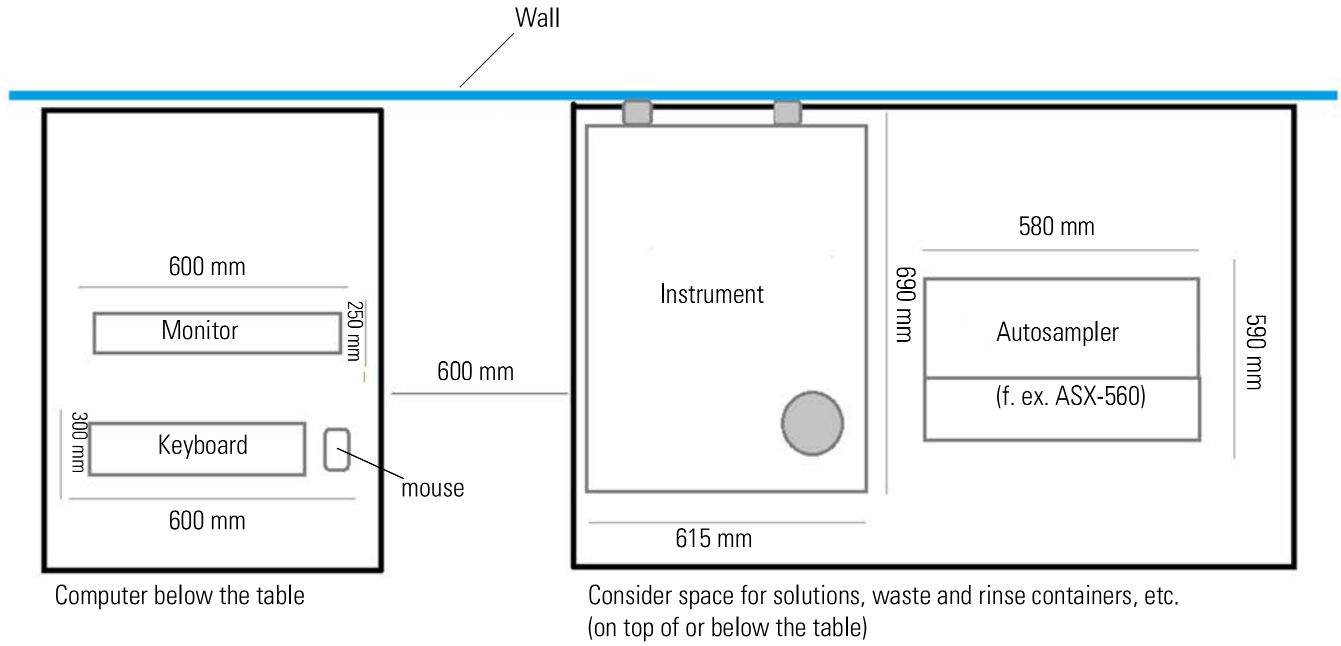


Figure 5-7. Typical floor space requirement for iCAP PRO Series instrument, autosampler and data station

Placing the Data System

For the data system, Thermo Fisher Scientific recommends using one workbench with minimum dimensions of 1.00 m × 1.20 m. The workbench must be capable of supporting the weight of the data system and a printer, if applicable. A printer is not shipped with the instrument. Use an Ethernet cable to connect the iCAP PRO Series instrument to the computer. See Figure 5-8.

Table 5-2 lists space requirements and weights of the typical data system hardware components. The actual values depend upon your equipment. Figure 5-9 shows typical data system hardware.

Table 5-2. Typical data system space and load requirements

Module	Height	Width	Length	Weight
Monitor	36 cm	41 cm	18 cm	6 kg
Minitower computer	48 cm	18 cm	43 cm	14 kg

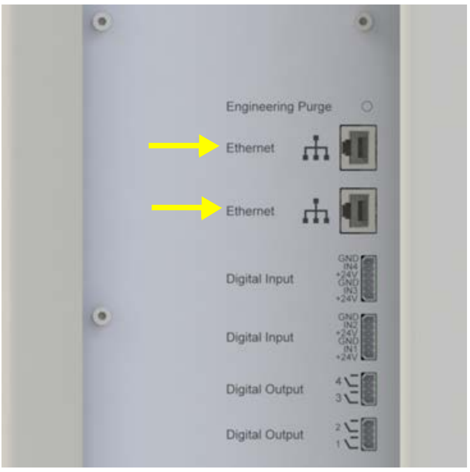


Figure 5-8. Connections for data system

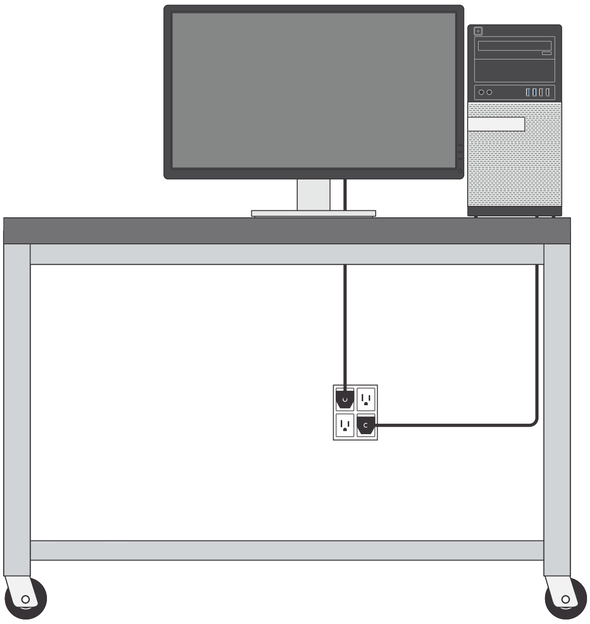


Figure 5-9. Typical data system hardware

Laboratory Conditions

The choice of an operating site for the instrument is influenced by:

- local considerations, such as ease of access and availability of electrical power.
- the dimensions and weights of the spectrometer and its accessories.
- the availability of water and gas supplies, as well as the need for a suitable ventilation system to dispose of the exhaust gases.

To ensure optimum analytical performance, reliability and longevity of your iCAP PRO Series ICP-OES, observe a number of basic laboratory considerations that are listed in Table 5-3.

Table 5-3. Laboratory considerations

Consideration	Why is this aspect important?
General environmental conditions of the laboratory	The environmental conditions of the laboratory should be maintained in compliance with Good Laboratory Practice (GLP) to ensure a clean and safe work environment for laboratory personnel.
Appropriate placement of the instrument with availability of a clean air supply	The iCAP PRO Series ICP-OES, as with all ICP-OES, draws air through the unit as a source of cooling. If the laboratory air contains a high content of particulates, exhibits high humidity and/or is contaminated with corrosive gases, this could lead to premature corrosion, component failure and aging of the instrumentation.
Appropriate placement of sample introduction accessories when working with corrosive or volatile liquids	To ensure a sufficient air clearance, liquid autosamplers should be installed around the base of the iCAP PRO Series ICP-OES. Thermo Fisher Scientific also recommends using a cover and/or appropriate extraction in association with this accessory when working with corrosive or volatile liquids.
Placement of sample preparation accessories if working with acids or volatile liquids	The sample preparation accessories should be installed and operated in an area that is completely separated from the instrument laboratory when performing operations with corrosive or volatile matrices.
Chemical storage and spillage control	Thermo Fisher Scientific recommends keeping the handling of chemicals and reagents in the instrument laboratory to a minimum. Clean up any chemical or reagent spillages immediately to reduce contamination of the laboratory air. All chemicals and reagents should be handled and stored externally to the instrument laboratory and in accordance with the appropriate MSDS.
Using flammable liquids	If you use flammable liquids, be aware of the basic standards that apply to safe handling and storage of such materials. Thermo Fisher Scientific recommends putting in place procedures to prevent accidents and to protect people from the hazards of flammable substances.

Temperature

The spectrometer is designed to operate at the temperatures specified in Table 5-4.

Table 5-4. Temperature requirements for iCAP PRO Series

Specification	Value
Laboratory temperature	15–35 °C
Optimum operation temperature	19–25 °C
Temperature fluctuations	≤ 2.5 °C/h

As the laboratory temperature increases, system reliability decreases. All electronics components generate heat while operating.

NOTICE Do not put the system where sudden changes in temperature or air drafts can occur, for example near a door or a window. Take care with the location of items such as air conditioning vents and heating vents. Avoid direct sunlight, proximity to heating or cooling sources and air drafts.

Exhaust System

Consider the following guidelines for ventilation and exhaust.

WARNING **Suffocation Hazard**. Nitrogen and argon gas might cause suffocation if they accumulate in the laboratory. Make sure that the laboratory is well ventilated. If the extraction is turned off, sufficient air must be supplied to prevent the concentration of these gases from reaching a harmful level.

⚠ Correct extraction is critical to instrument performance and safety. Do not operate the spectrometer without an effective fume extraction system attached to the torch box chimney. It should include appropriate filtering of hazardous toxic fumes.

NOTICE This instrumentation is designed for operation in clean air conditions. The laboratory must be free of all contaminants that could have a degrading effect on the instrument components. Dust, acid and organic vapors must be excluded from the work area. The warranty does not cover operation in substandard conditions.

The exhaust system is needed to remove plasma gases that may contain ozone and other noxious substances as well as heat generated by the electronics of the instrument. Hot fumes, which might be corrosive and toxic, are discharged from the instrument chimney during operation.

NOTICE

Flexible hoses must be connected to the instrument and plasma exhaust while operating the instrument. Thermo Fisher Scientific will install your system only if an adequate exhaust system is present and functioning. Exhaust gas venting must comply with all local environmental codes.

NOTICE

The exhaust system for the iCAP PRO Series instrument must not be affected by external weather conditions or other recipients potentially connected to the same system.

The exhaust system must not let the fumes or the fumes condensation return toward the instrument. It must be tested for leakage before connecting it to the instrument.

Other peripheral devices might need additional exhaust connections. Refer to the manuals of the peripheral devices.

An exhaust port is situated at the top of the instrument. See arrows in Figure 5-10.

Figure 5-10. Exhaust port of the iCAP PRO Series instrument

The exhaust port at the iCAP PRO Series instruments has an outer diameter of 135 mm. The exhaust lines will be connected via flexible hoses with an inner diameter of 120 mm. The exhaust hose should be equipped with a throttle valve.

The exhaust flow must be set to the values specified in Table 5-5, measured in the center of the extraction tube.

Table 5-5. Specifications for the exhaust

Parameter	Value
Outer diameter of exhaust port	135 mm
Inner diameter of exhaust hose	120 mm
Exhaust flow[a]	180–220 m^3/h
Optimum exhaust velocity	5.5 m/s

[a] for an inner diameter of exhaust port of 120 mm

The extraction tube has an inlet diameter of 125 mm. A 2 m flexible tube is supplied with the instrument.

Tip The extraction requirements are not the same as for previous Thermo Scientific ICP spectrometers. An iCAP PRO Series ICP-OES requires an extraction of 3.5–4.3 m/s.

The extraction flow must be adjustable to correctly set the flow and achieve the required extraction. This can be achieved with a butterfly valve or an adjustable gate.

The extraction fan specification varies depending on the laboratory layout, as well as on the lengths, diameters and type of the tubing required to reach the instrument. Consult a qualified extraction specialist to ensure that the correct flow is achieved at the instrument.

Additionally, consider a separate extraction if significant numbers of volatile or acidic samples are left in the proximity of the instrumentation. If an autosampler is used with volatile or acidic samples, a separately vented enclosure should be used. This should include appropriate filtering of hazardous toxic fumes.

Heat Generation and Dissipation

The heat generation and the heat dissipation of the system depend on the equipment employed. However, the iCAP PRO Series system must always be operated with an active plasma and instrument exhaust. For a description of the exhaust system, see page 5-9.

For estimated values of the average heat dissipation of the spectrometer and other heat sources during analysis, see Table 5-6.

Table 5-6. Approximate heat generation of a typical instrument

Module	Heat generation [W]
iCAP PRO Series system	650[a]
Recirculating chiller	1900[a]
Monitor	25
Computer	75
Total	**2650**

[a] maximum value

The values listed in Table 5-6 are based on a laboratory temperature of 23 °C and a cooling water temperature of 20 °C. A higher room temperature and/or a lower cooling water temperature increase the heat generation.

Recirculating Chiller

NOTICE Water-cooled chillers minimize the heat dissipation into the laboratory environment. However, a constant source of running water must be available for this optional chiller.

A recirculating chiller is required to remove waste heat from the spectrometer. The chiller must be able to supply a minimum flow rate of 2 L/min with a back pressure of about 2 bar (0.2 MPa) and a minimum cooling capacity of 750 W at a room temperature of 25 °C and at your altitude. The chiller must provide 50 µm particulate filtration.

A suitable chiller (Thermo Scientific ThermoFlex™ 900) can be supplied by your local Thermo Fisher Scientific organization. For instruments used at an altitude above 1500 m, the ThermoFlex 1400 is recommended.

The water used in the chiller must be distilled or deionized water. The water temperature should be set to 5 °C below ambient temperature, and must be kept between 15 and 30 °C. The temperature variation must be less than 0.2 °C per hour. See page 5-20 for more information about the cooling water.

The airflow into and from the chiller must not be blocked or diverted by placing the chiller in an enclosed space.

The water hoses (blue for inlet and black for outlet) are shipped with the instrument. Each hose has a length of 3 m, an outer diameter of 12 mm, and an inner diameter of 9 mm. They connect to push fit connectors on the left side of the instrument. See Figure 5-11.

Tip If the distance between the water chiller and the spectrometer is larger than 3 m, insulated tubing must be used.

NOTICE To ensure safety and proper cooling, always operate the instrument with its covers in place. This is also necessary to comply with product safety and electromagnetic interference regulations.

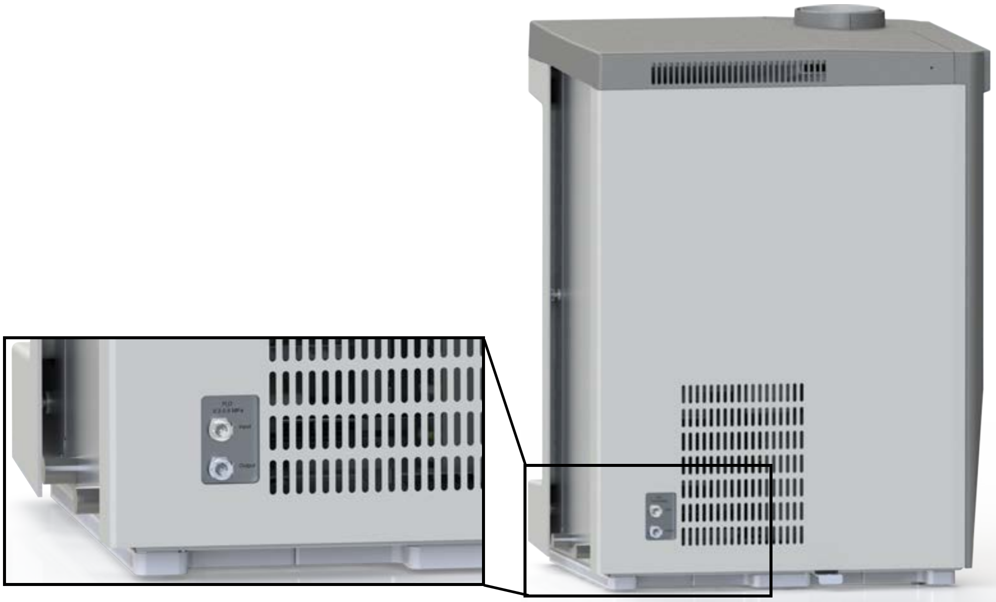


Figure 5-11. Cooling water connections

Air Conditioning

Any expenditures for air conditioning are more than offset by good sample throughput and reduced repair costs.

The air conditioning must be capable of maintaining a constant temperature as specified at “Temperature” on page 5-9 in the immediate vicinity of the system without producing excessive draft.

Thermo Fisher Scientific recommends installing an air conditioner if the specified limits are exceeded due to unfavorable climatic conditions. For reliable operation of the iCAP PRO Series instrument, a fluctuation of the room temperature of less than 2.5 °C/h is required.

NOTICE

Do not locate the instrument under an air duct, near windows, or near heating or cooling sources. Temperature fluctuations of 1 °C or more over a ten minutes period can affect performance.

Humidity

The relative humidity of the operating environment must be between 20 and 80% (< 30 °C) or 20 and 60% (≥ 30 °C), with non-condensing and non-corrosive atmosphere.

Thermo Fisher Scientific recommends equipping your laboratory with a temperature and humidity monitor to ensure that your laboratory is always within the required temperature and humidity specifications.

NOTICE

Operating an iCAP PRO Series instrument at very low humidity might cause accumulation and discharge of static electricity, which can shorten the life of electronics components.

Operating the system at high humidity might cause condensation, oxidation, and short circuits.

Locate sample preparation activities and corrosive materials in a separate room to avoid problems due to corrosive fumes.

Altitude

The iCAP PRO Series instruments are designed for indoor use at an altitude of up to 3000 m above sea level. For altitudes above 3000 m, contact Thermo Fisher Scientific.

Vibration

Floors must be free of vibration caused, for example, by equipment in adjoining locations. Propagation of vibrations and their influence on complex instrumentations are difficult to predict. Contact your local Thermo Fisher Scientific representative if you have questions or concerns about your laboratory.

Airborne Noise Emission

The A-weighted emission sound pressure level created by the iCAP PRO Series spectrometer does not exceed 70 dB(A). Therefore, wearing ear protection is not necessary.

Radio Frequencies

iCAP PRO Series instruments are designed to work in a controlled electromagnetic environment. The iCAP PRO Series system withstands electric fields of up to 1–3 V/m within a frequency range of 80 MHz to 2.7 GHz without any influence on operation. See Table 5-7.

Table 5-7. Immunity to electrical fields from 80 MHz to 2.7 GHz

Frequency range	Test level
80 MHz–1.0 GHz	3 V/m
1.4–2.0 GHz	3 V/m
2.0–2.7 GHz	1 V/m

Magnetic Fields

The instrument site must be free of interfering magnetic fields. The maximum acceptable field amplitude (AC) for any frequency is 3 A/m (3.8 µT).

Tip Sources of disturbing fields are, for example, other analytical instruments such as NMR systems or Zeeman AAS, train, tram, subway, high power cables crossing the ceiling, large electric motors (elevators), radio stations nearby.

Liquids

Avoid the possibility of liquid ingress into the top of the spectrometer. The location must ensure that it is not possible to store samples or other liquids directly above the instrumentation. Do not store organic or volatile solvents near the instrument, not even for a short time.

Solvent Waste

The analysis of a sample by ICP-OES usually involves the production of a fine mist from a liquid sample. Waste is produced that could be harmful, corrosive and toxic, or an organic solvent.

Waste solvents occurring during operation should be collected in an appropriate waste container that is solvent-proof, shatter-proof and vented away from the instrument. Compatible container materials ensure that waste does not react with them or corrode them.

This waste container must be provided by the customer. During installation, the waste container must be inert against 0.2% nitric acid.

Make sure that the waste container does not constitute a spill or trip hazard. It may be necessary to neutralize the waste to prevent any toxin formation.

Make sure that the pump, sample, waste tubing and vessels are labeled with correct safety symbols to protect personnel that are near the equipment, or that are using or maintaining it.

NOTICE Thermo Fisher Scientific recommends placing the waste containers in some type of secondary containment, such as a plastic bin, in case of a spill or overflow. To prevent the laboratory from being accidentally contaminated by solvent waste, protect the waste container against overturning.

The waste solution level must be kept below the level of the pump and spray chamber to prevent back siphoning with associated contamination and leakage risks.

The volume of waste should be kept to a minimum, following a risk assessment in accordance with local legal and Health and Safety guidelines. Consider the mixing of chemicals and chemical or physical effects (for example, expansion and heat production).

Inspect and empty the waste regularly. Appropriate facilities should be provided for the disposal of any waste following local legal and Health and safety guidelines for disposal. Do not store large volumes of chemicals (including samples) near the instrument or the operators.

The drip tray, which is part of the cover set of the spectrometer, is only meant to protect the instrument from minor spillage from the sample introduction system. See Figure 5-12.

Figure 5-12. Drip tray

Line Power

The performance and longevity of your system can be affected by the quality of line power delivered to the system. To ensure that your instrument performs optimally and is not damaged by line power fluctuations, verify that you comply with all power quality requirements listed in this manual.

NOTICE It is your responsibility as the customer to provide a source of power of acceptable quality for the operation of your system.

Electrical Power Requirements

The iCAP PRO Series instruments are designed to operate at a nominal voltage of 200–240 V, 50/60 Hz. For details, see Table 5-8.

Table 5-8. Basic power requirements

Specification	Value
Nominal Voltage	200–240 V AC, 50–60 Hz AC, single phase
Working Voltage	180–264 V AC
Power	apparent power: 2.694 kVA effective power: 2.605 kW
Wire	2-pole, 3-wire grounding
Fuse	Thermo Fisher Scientific recommends fusing the wall receptacle with 16 A (Europe) and 20 A (North America, Japan).

NOTICE **Customers in North America and Japan** Systems installed in areas with low line voltage (208 V in North America, 200 V in Japan), experience voltage sags during high use periods that might place the line voltage below the operating parameters described in this section. In this case, you must protect your instrument by using a suitable power conditioner or uninterruptible power supply (UPS).

NOTICE When using a boost transformer or power conditioning device with higher output than input voltage, the input voltage of the instrument must not exceed 264 V, even when line voltage fluctuations of ±10% occur at the input of the transformer or power conditioning device.

The iCAP PRO Series instruments must have a separate “clean” line leading to a main fuse to guarantee disturbance-free operation.

Power Connections

The electrical wall outlet for the main power of the iCAP PRO Series instruments should be located at the wall near the intended location of the instrument, ideally at the right side of the instrument.

When looking at the rear panel of the instrument, the power connector is located at the left side. Figure 5-13 shows the power connection of the iCAP PRO Series instrument.

Figure 5-13. Power connection at rear panel of iCAP PRO Series instrument

The instrument is equipped with a standard IEC 60320 C20 socket and will be shipped with a power cable suitable for your country. Depending on your area, different types of plugs and cables may be required. Refer to the *iCAP PRO Series ICP-OES Pre-Installation Requirements Guide* for a list of the available power cables.

Tip For more information on the correct cable, contact your certified Thermo Fisher Scientific service representative.

The mains cord supplied with your Country Kit has a length of 2.5 m. The electrical outlet must have an effective earth/ground connection. This protection must not be negated by the use of an extension cable without a protective earth conductor.

Additional standard mains sockets are required for the Data Station PC and for an autosampler, chiller, printer, or any other additional accessory.

Auxiliary Wall Outlets

Additional single-phase outlets are needed for additional parts such as computer and monitor. Thermo Fisher Scientific recommends at least five spare outlets in the near vicinity of the left side of the system and five close to the workbench space within the your laboratory.

The components of the data system (computer, monitor) require wall outlets at a nominal voltage of 200–240 V AC, 50/60 Hz. For more information, refer to the respective manufacturer's manual.

Quality of Power

The iCAP PRO Series instruments comply with the requirements listed in Table 5-9.

Table 5-9. Immunity compliance of iCAP PRO

Description	Requirement
Immunity to electrical fast transient/burst	EN 61326-1, EN 61000-4-4
Immunity to electrical slow transient/surge	EN 61326-1, EN 61000-4-5
Immunity to conducted RF voltage	EN 61326-1, EN 61000-4-6
Immunity to voltage dips, short interruptions and voltage variation	EN 61326-1, EN 61000-4-11

The instrument is tested in accordance to DIN EN 61326-1 (emission and susceptibility).

Avoid brownouts (drop in voltage) and spikes (increase in voltage) over short timescales. If doubts about the quality of the power supplied exist, use an uninterruptible power supply (UPS) and a voltage stabilizer.

Consumables

Your instrument requires gases and solvents that must meet defined purity specifications. The Thermo Fisher Scientific representative might also require certain solvents for the installation verification of your system.

NOTICE It is your responsibility as the customer to provide correct gas and solvent supplies for the operation of your system.

Cooling Water

For operating the iCAP PRO Series instruments, water cooling of the camera and the RF generator is required. The water used for the coolant setup should be free of suspended matter to avoid clogging of the cooling circuit. An in-line filter is supplied with the instrument to guarantee consistent water quality.

The use of additives to prevent build-up of biological growth in the water cooling system is not required per factory specification for the system. If required by local regulations, it is not prohibited as long as the recommended conductivity, pH and any other specifications (see Table 5-10) are met.

NOTICE Make sure that the cooling water specifications listed in Table 5-10 are met when you connect the instrument, regardless of connecting the instrument to an in-house water supply or to an external recirculating chiller.

If the water temperature is below the range specified in Table 5-10, condensation of atmospheric water might occur.

Table 5-10. Cooling water specifications

Specification	Value
Temperature	5 °C below ambient (10–30 °C)
Recommended conductivity	< 1000 µS/cm
pH	6–8
Solid residual	< 50 µm particle size
Pressure	0.2–0.6 MPa (2–6 bar)
Cooling capacity	≥ 750 W

The ThermoFlex™ 900 recirculating chiller is a suitable external chiller for the iCAP PRO Series instruments with sufficient cooling capacity.[1] See page 5-12 for more information about the chiller.

[1] For instruments operated at an altitude above 1500 m, the ThermoFlex 1400 is recommended. Refer to the *iCAP PRO Series ICP-OES Pre-Installation Requirements Guide*.

Gas Supply

WARNING **Suffocation Hazard**. Nitrogen and argon gas might cause suffocation if they accumulate in the laboratory. Even in Standby mode of the instrument, the purge gases are still released into the laboratory. Make sure that the laboratory is well ventilated. If the extraction is turned off, sufficient air must be supplied to prevent the concentration of these gases from reaching a harmful level.

NOTICE Make sure that the laboratory gas lines do not contaminate the gases used for the iCAP PRO Series ICP-OES.

Tip The gas requirements are not the same as for previous Thermo Scientific ICP spectrometers, for example additional gas for iCAP 6000 Series ICP-OES or iCAP 7000 Plus Series ICP-OES.

The iCAP PRO Series instrument requires argon as the **plasma gas** supply for the inductively coupled plasma. Air will be optional when an additional gas is required depending on the type of analysis. The purging of the optical path is guaranteed by the **purge gas**, which can be argon or nitrogen.

It is essential that the gases be delivered with the necessary pressure and purity. See the specifications given on page 5-23. In case of questions, contact your certified Thermo Fisher Scientific service representative.

Gas Consumption

The gas consumption of the iCAP PRO Series ICP-OES varies with plasma conditions and purge operating parameters. The gas consumption in Standby mode is approximately 0.4 L/min of purge gas. With argon being used as plasma and purge gas, the instrument consumes a minimum of 11.4 L/min of argon (maximum 26.4 L/min) in operation with a running plasma. Table 5-11 shows the approximate gas consumption for plasma gas and purge gas at different conditions.

Table 5-11. Typical gas consumption of iCAP PRO Series ICP-OES

Operating mode	Plasma gas [L/min][a]	Purge gas [L/min]	Total
Standby	0	0.4	0.4
		2.4 (extended UV performance)	2.4
Plasma	10 (minimum)	1.4	11.4
	23 (maximum)	3.4 (extended UV performance)	26.4

[a] method-dependent

Gas Supply Lines

The external gas lines to the instrument connect to the bottom of the main connections panel of the instrument at the rear side. The gas connections are shown in Figure 5-14.

Figure 5-14. Gas connections

The external gas lines for the plasma gas and purge gas are shipped with the instrument at a length of 3 m. See Table 5-12 for other dimensions.

Table 5-12. Dimensions of gas supply lines

Gas lines for	Outer diameter	Inner diameter
Argon	6 mm	4 mm
Purge gas	6 mm	4 mm
Additional gas	4 mm	2.5 mm

The connection to the laboratory gas supply should be within 1 m of the instrument. A regulator should be close to the connection. The instrument is supplied with particulate gas filters. These must be correctly fitted and maintained.

Tip If you wish to use argon for both plasma gas and purge gas, use the Y-piece that is shipped with the instrument.

Gas Specifications

Argon is used as plasma gas or purge gas. The argon supply to the iCAP PRO Series instrument must be stabilized. The gas pressure regulator must be qualified for the purity specification of the plasma gas.

To ensure that the argon supply quality to the instrument is not contaminated by the gas lines, refrigerator grade copper or stainless steel must be used. The pipe work must be cleaned by a suitably qualified contractor before use.

If liquid argon is not used, Thermo Fisher Scientific recommends using banks of cylinders with switch over valves so that the gas supplies can be used continuously.

Table 5-13. Argon gas requirements

Specification	Value
Purity	≥ 99.995%
Maximum water content	< 10 ppm
Maximum oxygen content	< 10 ppm
Operating pressure	0.55–0.6 MPa (5.5–6 bar)

Nitrogen may be used as optional purge gas.

Table 5-14. Nitrogen gas requirements

Specification	Value
Purity	≥ 99.995%[a]
Maximum water content	< 10 ppm
Maximum oxygen content	< 10 ppm
Operating pressure	0.55–0.6 MPa (5.5–6 bar)

[a] For optimum performance, Thermo Fisher Scientific recommends a purity of 99.998%.

Air, oxygen or argon can be used as additional gas. Figure 5-14 on page 5-22 shows the connection for the additional gas supply on the right side. See Table 5-15 for the requirements.

Table 5-15. Additional gas requirements

Specification	Value
Purity	≥ 99.99%
Maximum water content	< 100 ppm
Operating pressure	0.2–0.6 MPa (2–6 bar)

NOTICE Make sure that the compressed air is clean, dry and oil-free. Otherwise, damage to the mass flow controller (MFC) results.

If the additional gas MFC is fitted to the iCAP PRO XP or iCAP PRO XPS spectrometer, compressed air, oxygen or argon is required.

NOTICE

The usage of the additional gas is defined on the Dashboard instrument Home Page of the Qtegra ISDS Software. Depending on whether **Auxiliary Gas** (for organics analysis) or **Front Outlet** (for analysis with the sheath gas adaptor) is selected, the correct gas line has to be attached to the Add Gas inlet at the rear side of the instrument. When this parameter on the Qtegra Dashboard is changed, the gas line has to be exchanged at the rear side of the instrument as well.

Permitted Materials

The iCAP PRO Series ICP-OES system is designed to be operated with the materials listed in Table 5-16.

Table 5-16. Materials and their use

Material	Used for
Plasma Gas	
Argon	Used to maintain the plasma and to purge the optical system.
Purge Gas	
Nitrogen	Optional to purge the optical path of the light.
Additional Gases	
Argon	Used as a sheath gas in high salt applications, in combination with the sheath gas adaptor.
Air, oxygen	Used for organic matrices as additional gas to oxidize excessive carbon in the plasma.
Sample matrices	
Water based solutions with mixed acid content	Preparation of calibration solutions, samples. Maximum permitted content: HNO_3 < 10%, HCl < 5%, H_2SO_4 < 10%, HF < 1% (5% when inert sample introduction system is used).
Organic solvents (like ethanol, isopropanol)	Applications that require the analysis of organic solvents require the addition of air of a specified quality. Running organic matrices typically requires non-standard sample introduction systems.

Cleaning Agents

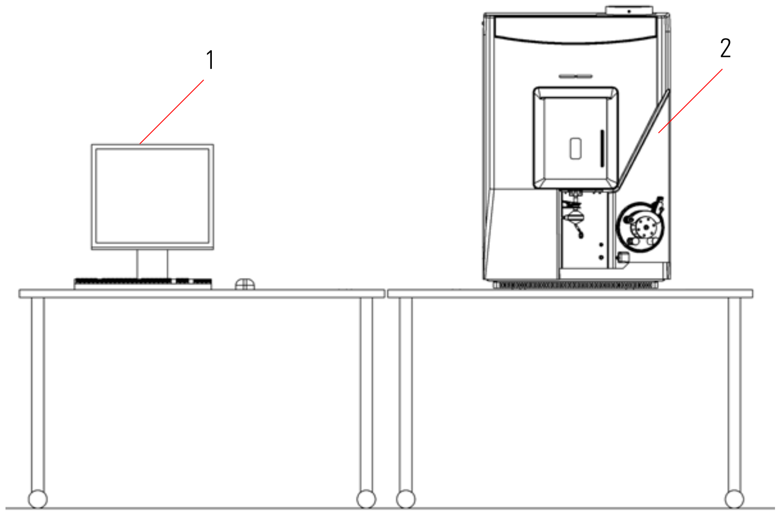
Thermo Fisher Scientific recommends having the following cleaning agents available:

- A diluted solvent like isopropanol in water (in accordance with your local safety practices)
- Distilled water

NOTICE The iCAP PRO Series ICP-OES covers can be damaged by solvents (ethanol, for example) and concentrated acids.

Communications Interface

An Ethernet connection is used between the instrument and a computer for Data Station communication. Therefore, if the Data Station computer is connected to a local area network, an additional Ethernet port is required for connection to the instrument. See Figure 5-15.



Labeled Components: 1=Data Station computer, 2=instrument

Figure 5-15. iCAP PRO Series ICP-OES diagram

Spectrometer Communication Requirements

Any firewalls installed on the computer must be disabled for the instrument Ethernet adapter, or alternatively configured to allow the appropriate instrument communications socket traffic. The Ethernet adapter is dedicated to the instrument and will not be connected to any other networks. Therefore, there is no security risk associated with allowing communication by the instrument Ethernet adapter.

The IP address of the instrument Ethernet adapter is by default configured by the Thermo Fisher Scientific field service engineer:

Instrument IP address:	192.168.1.100
Subnet mask:	255.255.254.0

Tip The instrument Ethernet port must be configured to allow it to communicate with the instrument.

If a router is used for the Internet connection on the PC, the router cannot use the same subnet as the iCAP PRO Series ICP-OES. For example, the IP address of the router can be set to 192.168.2

Data Station Requirements

The data station requirements are usually met by a PC that meets the minimum specifications listed in the document *PC Specifications for Qtegra ISDS Software*. A suitable computer is available from your local Thermo Fisher Scientific organization.

⚠ The Thermo Fisher Scientific field service engineer is not responsible for the network and customer-specific setup of the computer.

6

System Operation

This chapter describes the checks of the iCAP PRO Series ICP-OES that you should perform every day to ensure the correct operation of your system.

Contents

Preparing the System for Use

The iCAP PRO Series ICP-OES is designed to be constantly powered up and the optical system continuously purged. The instrument is powered with an on/off switch on the left part of the rear side. See Figure 6-1.

Figure 6-1. Main power switch of iCAP PRO Series ICP-OES

Two LEDs above the door at the front side of the instrument indicate the statuses of the instrument (left) and the active analysis (right.) See Table 3-1 on page 3-5 for further information.

Instrument Optimization

The iCAP PRO Series ICP-OES requires optimization dependent on the samples being analyzed and the method requirements.

It is important that the method development verifies the data produced by the method. It is also important that a suitable quality control regime is established that verifies the continuing validity of data.

Training courses are available through a local Thermo Scientific Sales Office. See page 1-7.

Method Optimization

All the parameters listed in Table 6-1 affect the data obtained and should be optimized. Usually, a default setting yields data that are satisfactory (apart from Duo spectrometers, where radial/axial views have to be selected manually), but may not be optimal for the analysis requirements.

Table 6-1. Parameters affecting data

Parameter	Comment
Nebulizer gas flow	Changes the nebulization performance and emission height on a radial instrument
Auxiliary gas flow	0.5 L/min works on most samples. 1 L/min may be required for high TDS, and 1.5–2 L/min for organic samples.
Coolant gas flow	12 L/min works with most samples. 14 L/min for organics if a higher power is selected
Additional Gas Supply	For organics samples to burn off the excess carbon Normally set to about 25 mL/min of air For high salt samples to avoid blockage of the center tube due to crystallization Normally set to about 200 mL/min of air or argon
Radial Instrument Plasma viewing height	Used to select optimum viewing height for different emission zones in the plasma. Optimum viewing heights are 11 mm for a Radial instrument and 10 mm for a Duo instrument.
Radial/Axial View	On a Duo instrument, you have a choice of axial and radial views. You have to select the view that you want to use manually. As a general rule, select axial view for all low wavelengths and select radial view for all high wavelengths.
RF power	1150 W works with most samples. You may want to select higher power for organics and High TDS samples.
Pump speed	45 to 50 rpm for most samples As low as 30 rpm for organics to reduce the plasma loading
Sample Chemistry	All instrument parameters are separate to the development of the chemical requirements of the method, for example variation in sample ionization, solvent volatility and viscosity effects.

Adjusting the Pump Tubing Tension

To ensure long life of the pump tubing and correct operation, the pump tubing tension needs to be adjusted. The pump tubing tension can be adjusted with the plasma running and the pump stopped.

❖ **To adjust the pump tubing tension**

1. Lock the sample pump tubing and clamp into position.
2. Release the tension adjustment and let the nebulizer freely aspirate.
3. Tighten the tension adjustment until the flow just stops. Then tighten by a quarter to half of a turn.
4. Turn on the pump and, if necessary, tighten the tension until a smooth flow is produced.

NOTICE Do not overtighten the pump clamps, because this results in excessive wear and tear of the pump tubing and requires replacement tubing at more frequent intervals.

Assembling the Standard Sample Introduction Glassware

⚠CAUTION Follow appropriate care and safety procedures to avoid breaking any glassware and causing injury to the operator. Handle any broken glassware with care.

NOTICE Always wear gloves when you handle glass or ceramic torches. Handprints reduce the lifetime of the torch and might cause the torch not to ignite.

Assembling the Duo Torch

❖ **To assemble and install the Duo Torch assembly**

1. Check the O-rings in the metal torch mount (three internal and two external). Replace them if any wear or damage is visible.

2. Push the quartz body of the torch fully into the metal torch mount. The marked circle on the quartz torch should be aligned with the notch on the torch holder assembly. The associated line marking on the torch should be aligned with the edge of the torch holder assembly.

3. Make sure that the four O-rings in the center tube holder are not damaged. Fully insert the center tube into the plastic center tube holder.

   Tip The tip of the center tube holder discolors with use. This discoloration is normal and does not affect the performance of the torch holder assembly.

   To replace or exchange the center tube, adding a drop of DI water will help loosening it from the O-ring inside the holder.

4. Insert the center tube assembly into the metal torch holder.

5. Screw the center tube holder assembly clockwise into the metal torch holder until the O-ring is compressed. Do not overtighten, because this would reduce the lifetime of the O-ring seal.

   When fitted, the center tube should be 1–3 mm lower than the intermediate tube.

6. Insert the torch holder into the torch box. Turn the metal torch holder clockwise until the red orientation lock self locates in the torch box casting.

   Make sure that both slots, that of the duo torch and that of the radial view cone, are aligned correctly. If this is not the case, align the torch inside the torch holder accordingly.

NOTICE

If the two slots of the duo torch and the radial view cone are not aligned correctly, the performance of the instrument might be compromised.

7. Insert the white plastic tubing connector (black ring) and wide bore tubing (0.79 mm ID) into the spray chamber drain tube. The drain and spray chamber should be positioned so that no pulsing occurs during the liquid removal. See Figure 6-2.

Figure 6-2. Spray chamber drain

8. Liquid should be delivered to the nebulizer using an identical plastic tubing connector, but with narrow bore tubing (0.50 mm ID) relative to the spray chamber drain tubing.

   Push the white plastic tubing connector with the attached narrow bore sample tubing into the rear of the nebulizer as far as possible without exerting undue pressure.

   Inspect the O-rings in the spray chamber. Replace them, if any wear or damage is visible.

9. Using a twisting motion, insert the nebulizer into the spray chamber so that the collar is a tight fit. See Figure 6-3. The collar will set the insertion depth and aid reproducibility of results.

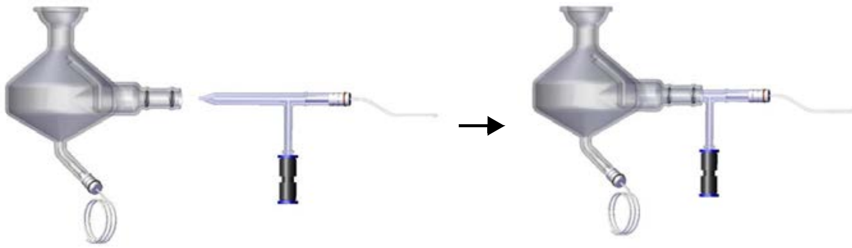


Figure 6-3. Nebulizer spray chamber assembly[a]

[a] Here without collar, which would sit between the vertical glass tube of the nebulizer and the horizontal entrance tube of the spray chamber.

10. Attach the spray chamber adapter to the spray chamber with the fitting clamp provided. The adapter provided with the instrument is

specially designed to prevent the UV radiation from escaping from the torch box.

11. Insert the spray chamber adapter fitting into the torch assembly holder as far as it will go. Connect the nebulizer gas supply to the push-fit fitting.

Proceed with "Preparing the Ignition of the Plasma" on page 6-8.

Assembling the Radial Torch

❖ **To assemble and install the Radial Torch assembly**

1. Check the O-rings in the metal torch mount (three internal and two external). Replace them if any wear or damage is visible.

2. Push the quartz body of the torch fully into the metal torch mount. The marked circle on the quartz torch should be aligned with the notch on the torch holder assembly. The associated line marking on the torch should be aligned with the edge of the torch holder assembly.

⚠ The alignment of the torch marking with the torch holder assembly is essential to ensure that the radial view hole and gas holes are correctly aligned.

3. Insert the spray chamber adapter (ground glass joint) into the rear of the center tube holder.

4. Insert the center tube fully into the center tube holder.

Tip The tip of the center tube holder discolors with use. This discoloration is normal and does not affect the performance of the torch holder assembly.

To replace or exchange the center tube, adding a drop of DI water will help loosening it from the O-ring inside the holder.

5. Insert the center tube assembly into the torch mount assembly.

   Screw the center tube holder clockwise into the base of the torch holder assembly until the O-ring is compressed. Do not overtighten, because this would reduce the lifetime of the O-ring seal.

6. Push the torch assembly up into the torch box and twist clockwise until the red orientation lock locates in the torch box casting.

7. Insert the white plastic tubing connector and wide bore tubing (0.79 mm ID) into the spray chamber drain tube.

   Liquid should be delivered to the nebulizer using an identical plastic tubing connector, but with narrow bore tubing (0.50 mm ID) relative to the spray chamber drain tubing.

8. Push the white plastic tubing connector with the attached narrow bore sample tubing into the rear of the nebulizer as far as possible without exerting undue pressure. See Figure 6-4.

9. Inspect the O-rings in the spray chamber. Replace them if any wear or damage is visible.

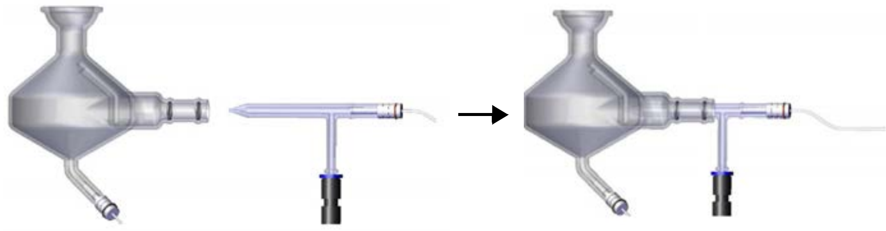


Figure 6-4. Cyclone spray chamber

10. With a twisting motion, insert the nebulizer into the spray chamber so that the collar is tight fit. The collar will set the insertion depth and aid reproducibility of results.

11. Connect the spray chamber and the nebulizer assembly to the torch assembly and lock it into place in the instrument with the stainless steel clamp.

Proceed with "Preparing the Ignition of the Plasma."

Preparing the Ignition of the Plasma

⚠ CAUTION

It is extremely important that the correct Thermo Scientific part is used for the spray chamber adapter. Operators could be exposed to dangerous UV radiation and radio frequency radiation if alternate parts are used for the spray chamber adapter. In addition, system interlocks on the torch holder and elsewhere are there for safety reasons and must not be bypassed.

❖ **To prepare the ignition of the plasma**

1. Close the door of the torch box before you ignite the plasma. This will be confirmed by the door interlock indicator in the Qtegra ISDS Software.

2. After assembling the sample introduction system and before igniting the plasma, check the following items to assure correct assembly:

   - The torch is fully rotated and locked into place.
   - The center tube holder is fully rotated and locked into the torch.
   - The spray chamber adapter is fully pushed into the torch body.
   - The spray chamber is tightly clamped to the spray chamber adapter.

   Problems in any of these areas might cause air leaks or disruption of the gas flows making the plasma difficult to ignite and might cause damage to the torch.

To feed the sample capillary tubing, proceed with "Connecting the Pump Tubing" on page 6-9.

Connecting the Pump Tubing

Figure 6-5 shows the peristaltic pump and 2-stop tubing of the iCAP PRO Series ICP-OES. The sample pump tubing has white and orange stops, and the drain tubing has white and white stops.

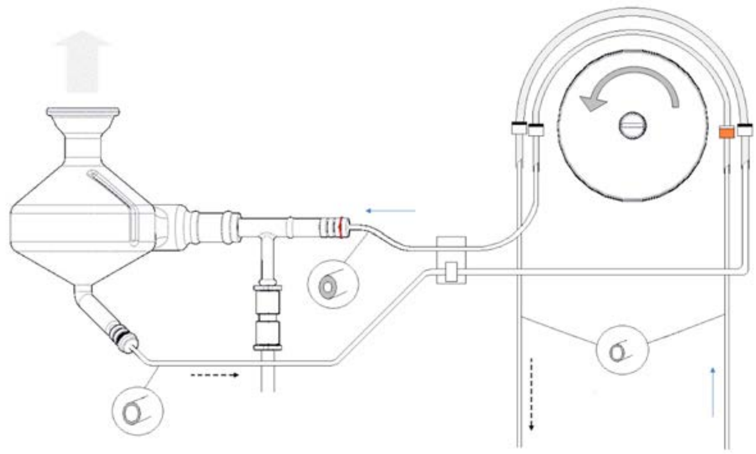


Figure 6-5. Sample capillary tubing scheme

For all instrumentation, the assembly procedure is similar.

❖ **To connect the pump tubing**

1. Feed the sample capillary tubing from the rear of the nebulizer towards the pump. Make sure that there are no twists or bends in the nebulizer and drain PTFE tubing that might prevent the sample flow.

2. Pass the drain capillary tubing through the drain sensor and towards the pump.

3. Insert the sample tubing and the drain PTFE tubing into their respective peristaltic pump tubes. Gripping the PTFE tube with fine sandpaper is recommended.

NOTICE Connect the drain tubing correctly to the peristaltic pump to account for the counterclockwise flow.

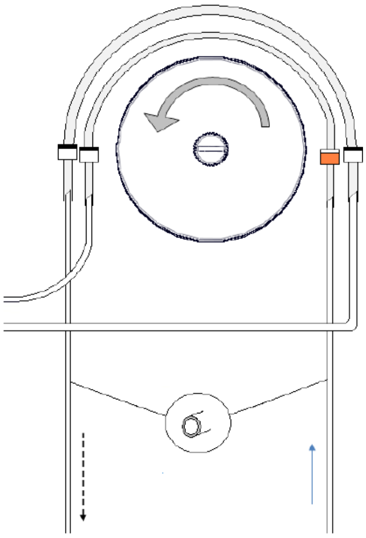


Figure 6-6. Pump tubing

4. Release the tension fingers and arms of the peristaltic pump.

   Put the sample and drain pump tubing over the pump rollers, locking the colored stops on the pump tubing into the left and right clamps. Remember to allow for the correct direction of flow.

5. Clap the tension arms onto the peristaltic pump tubing. Make sure that the peristaltic pump tubing is aligned along the center of the tension arm. Push the tension fingers back in place.

   For instructions to set the pump tubing tension, see page 6-4.

Inspect the pump tubing before each analysis. Replace it, if there are indications of wear.

Use additional lengths of capillary tubing to allow the connection to the input of the sample pump tubing to the sample and the output of the drain pump tubing to a waste container.

Example Standard Operating Procedures

The following method setup procedure is an example standard operating procedure and should be enough to set up a basic analysis. By no means does it cover all possible parameters used in the Qtegra ISDS Software. Read the *iCAP PRO Series ICP-OES Software Manual* for instructions for a more advanced use of the system.

Preparing the System

If the gas supplies have been switched off, the optical system should be purged for at least two hours before you switch on the water chiller and run samples. Purging is necessary to prevent damage to the camera, which is cooled to -45 °C. Even if the purge gas has been off for some time, signals in the low wavelength range may only be acceptable after several hours. It takes at least five hours of normal purge to measure aluminum at 167 nm with the specified stability and sensitivity.

❖ **To prepare the system**

1. Turn on the argon plasma gas. Set the pressure to 5.5 bar (0.55 MPa) on the gauge near the instrument.

   For normal use, leave the purge gas constantly on to protect the CID detector and to maintain high performance in the low wavelength range. Under trickle purge, this is a very small gas flow. See Table 5-11 on page 5-21.

2. Turn on the air supply for the additional gas supply if used.

3. Switch on the power to the iCAP PRO Series ICP-OES.

   For normal use, leave the power constantly on. If the instrument power has been switched off, wait at least for two hours after restoring power to let the instrument thermally stabilize.

4. Switch on the water chiller.

NOTICE Do not switch on the water chiller until the instrument has been purging with gas for at least two hours. Doing so can cause serious damage to the CID detector.

5. Push the tension arms onto the rollers of the peristaltic pump using the required number of the total four (three) tension fingers. The sample requires one channel, the drain another one. Further channels are optional (for example, for the internal standard addition).

NOTICE To avoid unnecessary wear of material, tighten only the tension fingers on the tension arms that are used.

6. Make sure that the drain tube is placed in an open neck vessel.
7. Place the sample tube in a blank solution.

8. Switch on the computer.
9. Click the Qtegra ISDS icon on the computer desktop.

Igniting the Plasma

❖ **To ignite the plasma**

1. Make sure that the door has been physically closed.

2. Click the Qtegra ISDS icon on the computer desktop.
3. Make sure that the interlock indicators are all green and take appropriate action if any of the following are red:
   - Torch Holder
   - Door Lock
   - Water Leak Detector
   - Board Temperature
   - Exhaust Pressure Interlock
   - Optical Heater Interlock
   - Water Flow
   - Camera Temperature
   - Plasma Gas Pressure
   - Purge Gas Pressure

   If an interlock causes an instrument error, the Qtegra ISDS Software shows a message below the source settings panel that describes the issue and gives a possible solution.

4. When ready, click the **Get Ready** icon on the Dashboard and select the desired options.

The Get Ready window opens showing several options:

Warm-Up Time	Is usually set to 15 minutes to enable the system to stabilize before spectrometer optimization.
Nebulizer Gas Autotune	If required, select this check box and choose the plasma view (All, Axial or Radial) to be optimized. An optimized nebulizer gas flow guarantees maximum intensities (determined with the Zn (213) peak).
Performance Check	Runs the factory-recommended performance test using defined sample introduction and standards.
Use Manual Sampling	If an autosampler is configured, select this to enable manual sampling.

Tip On installation, the Fore Optics Alignment must be completed first. See page 8-10.

5. Click **OK**.

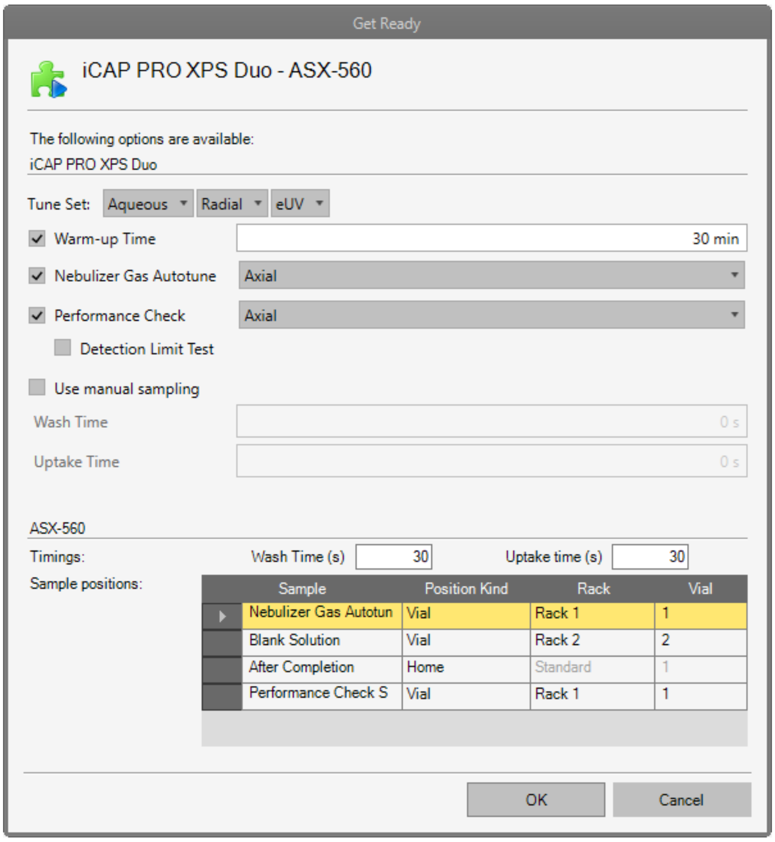


Figure 6-7. Get Ready window

The plasma is switched on, and selected procedures are performed. During these procedures, the current step in the Get Ready process and waiting times are shown on the Dashboard.

Tip To let the plasma stabilize, leave the plasma on with a blank solution running for about 15 minutes before you perform an analysis.

Shutting Down the System

❖ **To shut down the system**

1. Aspirate a blank sample for five minutes to ensure that the sample introduction components have been rinsed of sample.

2. To remove the blank sample, aspirate deionized or distilled water for a further minute.

   When organic solvent based samples are being analyzed, the final rinse should be the pure solvent. Air should be aspirated for one minute to remove organic solvent from the sample lines and to remove organic vapors.

3. If it is not already open, click **Qtegra ISDS** to open the program.

4. On the Dashboard, click the green **Get Ready** icon to open the toolbar.

5. Click **Shut Down**. See Figure 6-8.

Figure 6-8. System shutdown

The plasma is switched off and the iCAP PRO Series ICP-OES shuts down. The state of the instrument changes to **NotReady**.

NOTICE

Do not open the torch box or remove any of the torch components (torch holder, center tube holder) for some time after the plasma has been extinguished. After extinguishing the plasma, coolant gases are still running through the torch. These gas streams should not be interrupted to avoid damage to the components.

6. Wait two minutes before you switch off the water chiller and the gas supplies.

Follow the instructions in the manufacturer's manual for instructions about switching off the chiller.

NOTICE

Switching off the chiller by removing the power can often cause breakages.

7. Wait five minutes before you switch off the power to the instrument.

8. Release the tension on the sample pump tension arms to preserve the life of the pump tubing.

7

Troubleshooting

This chapter provides information about resolving common problems with plasma ignition, problems with plasma going out unexpectedly, and problems with analytical results.

Contents

Problems with the Plasma Ignition

If you have trouble with plasma ignition, try to isolate the source of the problem.

NOTICE Do not try to ignite the plasma with the spray chamber and connector removed and leaving the end of the center tube holder open.

❖ **To identify causes for plasma ignition problems**

1. First, take a note of any error messages displayed on screen, and then in the journal. These messages may suggest a cause of the problem, such as gas supplies, extraction, etc.

2. Make sure that your instrument is operating within the required parameters for gas, extraction, chiller, etc. as described in Chapter 5, "Installation."

3. If you are using a ceramic torch or any other hardware that is not part of the standard aqueous kit supplied with the instrument, remove it and use the standard glass torch, holder, center tube, etc.

4. If you are using an argon humidifier, remove it and try to ignite.

   If the plasma ignites now, there might be a leak in the argon humidifier tubing. To help with ignition when using an argon humidifier, try turning on the nebulizer gas manually for 10 seconds before ignition.

5. Also check for other leaking tubings or damaged O-rings. Replace them if necessary.

Argon Gas

To form a plasma, the argon gas must be of high quality (99.995%). If you have changed gas supplies recently, this could be the problem.

⚠ When you carry out the procedure, make sure that the end of the torch is completely sealed to avoid exposure to harmful UV radiation.

❖ **To sort problems with argon gas supply**

1. Make sure that your argon plasma gas supply pressure is set to 5.5 bar (0.55 MPa) with a regulator close to the instrument.

   Make sure that the pressure is stable and does not fall during ignition.

2. Examine your gas filter(s) on the argon gas input, if fitted. If the filters appear dirty, replace them and check the quality of your argon gas supply.

3. If the plasma still fails to ignite, see “Ignition Spark” on page 7-3 and “RF Power to the Coil from the RF Generator” on page 7-3.

Ignition Spark

❖ **To check the ignition spark**

1. Listen for the ignition spark. When the plasma ignites, you can hear the spark quite easily.

2. If it is very quiet, a gas leak may exist. Check for the gas leak as described in “Argon Gas” on page 7-2.

RF Power to the Coil from the RF Generator

The plasma is formed by seeding ions in a stream of argon that is flowing through RF fields and magnetic fields in the region of the induction coil.

❖ **To check the load coil**

1. Look inside the torch box and make sure that the load coil has not been knocked out of position by the torch.

2. Make sure that the chiller is operating correctly and that there is enough water in it.

3. Examine the water filters on the input to the instrument, if fitted.

4. Look for deposits on the torch. Replace the torch, if necessary.

Problems with Plasma going out unexpectedly

Several causes might result in the plasma going out unexpectedly.

❖ **To find causes for plasma going out unexpectedly**

1. Try to ignite the plasma again if it goes out in the middle of a run or more than one minute after ignition.

   If the plasma ignites correctly, the problem was probably caused by the sample introduction or by a transient interlock.

2. Look for droplets in your spray chamber and clean the glassware, if necessary.

   Large droplets of sample that enter the plasma can put it out.

3. Check for leaks in your gas supply and the sample introduction system.

   Air introduced into the plasma can cause it to go out.

4. Make sure that the pre-installation parameters are met.

   A momentary change in the gas pressure or in the extraction rate can cause the plasma to go out.

Resolving Problems with the Analytical Results

Your sample introduction system must be set up correctly and be clean. The glassware must be properly assembled.

Tip Thermo Fisher Scientific recommends having several spares for each part available, because blockages, sample contamination and breakages might happen at critical moments during an analysis.

❖ **To check the condition of the sample introduction components**

1. Check the condition of the pump tubing. The tube must be uniform and show no signs of damage.
2. Feel for flats where the rollers have been. If flats exist, change the tubing.
3. Make sure that the tubing is on the pump correctly, with no pinching. Set the pump turning (50 rpm) in the instrument control panel before engaging the clamps to align the tubing. Then stop the pump and engage the clamps.
4. Make sure that there are no air bubbles in the nebulizer and that the connector is pushed all the way in.
5. If there is an air bubble in the nebulizer at the end of the white connector, pull the connector out. Re-insert the connector while the pump is still pumping water, getting rid of the bubble.
6. Make sure that there are no droplets on the end of the nebulizer.
7. If there is a droplet on the tip of the nebulizer, push the nebulizer fully in (with no plastic ring on its neck) and flush the system for a couple of minutes with a 10% Decon™ 90 solution, or similar diluted detergent/surfactant.

   Aspirate the Decon solution with the plasma off. Afterwards, run deionized water and pull the nebulizer out so that the side arm is approximately 8 mm from the end of the neck of the spray chamber.
8. Make sure that there are no droplets condensed on the walls of the spray chamber or in the spray chamber connector to the torch.

   No droplets, even small ones, should appear on the walls of the spray chamber.
9. If there are droplets on the surfaces, flush the system with a 10% Decon 90 solution for 30 seconds. Aspirate the Decon solution with the plasma off.
10. Make sure that the nebulizer is inserted consistently into the spray chamber. Generally, the tip of the nebulizer should be positioned level with the circumference of the spray chamber.

11. Check for leaks in the uptake tubing by looking for air bubbles in the tubing connecting to the pump.

    If there are bubbles, examine the junction between the uptake tube and the tubing connection to the pump. This is the most likely place for a leak.

12. Make sure that the spray chamber drains correctly and uniformly.

13. If the draining is poor, check the pressure of the pump clamp for the drain tube.

    Otherwise, flush the system with a 10% Decon™ 90 solution for a couple of minutes. Aspirate the Decon solution with the plasma off.

14. Make sure that your torch is fitted properly. This is especially important with an iCAP PRO Series ICP-OES Duo to ensure that Radial view is aligned well.

❖ To make sure that your sample introduction system is clean

1. After use, follow the instrument shutdown procedures. See "Shutting Down the System" on page 6-14.

2. Clean the components that are contaminated with sample residues. Wear suitable protective clothing, glasses and gloves.

❖ To make sure that your glassware is properly assembled

1. Insert the torch into the torch holder.

2. Insert the center tube into the center tube holder.

3. Assemble the nebulizer and the spray chamber.

4. Fit the torch and the center tube into the instrument.

5. Attach the spray chamber with the adapter and clamp to the instrument.

Resolving Specific Analytical Problems

This section provides troubleshooting for problems with precision, accuracy, and detections limits. See also "Suggested Maintenance in Case of Poor Precision and Detection Limits" on page 7-11.

Poor Precision

❖ **To resolve problems with poor precision**

1. Run a quick test to determine whether the poor analytical results are matrix-related.
2. Put a 10 ppm solution of a few common elements such as Cu, Mn, Ba, Cd, Zn and Fe together and make a method using the primary wavelength and default conditions.
3. After standardization, the standard should read back with a precision of typically 0.2 to 0.5% (with a 10 second integration time) on the instrument.

   If the precision obtained is substantially greater than this, try a fore optics alignment. See page 8-10. Go on to troubleshoot further, if the problem persists.
4. If the instrument meets these specifications, the sample matrix itself is suspect.
5. Try modifying plasma parameters such as the power to improve the precision.

Poor precision generally relates to problems in the sample introduction system:

- First, make sure that the nebulizer flow is set correctly by aspirating a 1000 ppm yttrium solution (sodium also works if no yttrium is available).
- Make sure that the center orange "bullet" is even with or slightly above the top of the coil. If not, adjust the nebulizer gas flow up or down until the "bullet" looks correct.
- At this point, the nebulizer back pressure should be approximately 2 bar (0.2 MPa) for aqueous solutions (when using the standard glass concentric nebulizer). If the pressure is substantially higher, the nebulizer orifice is generally to blame and should be cleaned. See also the pressure readback in the software.
- Pooling and dripping in the spray chamber can also cause many precision problems. You may perceive this using the yttrium test described above.

- If the yttrium "bullet" is bouncing up and down inside the plasma, it is usually indicative of dripping. In a glass or Teflon™ chamber, the chamber should be wetted properly: there should be no water droplets building up on the walls of the spray chamber.

  This is usually caused by an oily film and can be easily cured by aspirating a 1–5% Decon™ 90 solution for about 30 seconds. See also "Cleaning the Spray Chamber" on page 8-6.

- Other causes of poor precision can be in the expendable parts such as nebulizer and torch/center tube. Spares should always be available and these should be substituted one by one to observe the result. If the nebulizer is the cause, examine the inside of the orifice by removing the gas fitting. Then, with a magnifying glass, look for any small obstruction. Also, examine the capillary for obstructions.

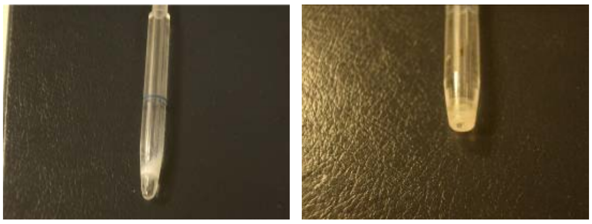


Figure 7-1. Examples of obstructions in the center tube

Teflon Capillary Tubing

The Teflon™ capillary tubing, see Figure 7-2, should be free of kinks and scissor or pinch cut ends. For best results, razor cut at 45° for the drain. Also, make sure that the sample peristaltic pump tubing condition and tubing pressure are properly adjusted.

Replace the tubing if it is used or collapsed. The pump tubing typically lasts only a couple of days. Introduce an air bubble into the sample uptake tube and watch its migration through the tubing. It should be smooth and consistent.

Figure 7-2. Teflon capillary tubing

Worn rollers, bent pump head shaft or bad roller bearings can cause inconsistent pump action. Any such damaged pumps should be replaced.

The peristaltic pump may also be used for the pumped drain spray chamber systems and the Internal Standards Mixing Kit.

Peristaltic Sample Pump

⚠CAUTION **Pinch Point Hazard.** Risk of injuries. Keep your hands clear from the peristaltic pump during operation.

Tip When you use a concentric or crossflow-free aspirating type nebulizer, adjust the pump tubing pressure with the plasma torch ignited and the pump stopped until free aspiration stops.

❖ **To adjust the tubing pressure**

1. With the plasma ignited, stop the peristaltic pump and dip the uptake capillary (which is normally connected to a sipper probe) into deionized water.

2. With the nebulizer gas switched on, gradually reduce the tubing pressure by releasing the screw on the tension finger until the water freely aspirates through the pump tubing. You can briefly remove the capillary or sipper probe from the rinse for a short period to introduce a small air bubble.

3. Tighten the tension adjustment until the flow stops. Then tighten by one more turn. Turn on the pump and, if necessary, tighten the tension until a smooth flow is produced.

4. With the pump turned on, adjust the drain pressure to let small bubbles flow in the drain tube. Any pulsing of the liquid in the spray chamber can affect the precision of the results.

   Argon and/or air leaks can cause many problems including poor precision.

Poor Accuracy/Feedback

❖ **To test for poor accuracy**

1. Accuracy in this manual means reproducing the standard value that was once standardized. Proceed by making a standard as in “Poor Precision” on page 7-7 (10 ppm Cu, Mn, Cd, Zn, Fe) and using this as the test solution. Remember that for this test accuracy is not defined as the ability to read a 1 or 100 ppm standard after standardizing on the 10 ppm.

Most of the time, this problem is operator-related. As far as we are concerned at this point, we have only one standard to test with. If this simple standard does not repeat back for all elements, examine the pump tubing first. Replace it if it is used or collapsed.

> **Tip** Depending on the frequency of usage, the pump tubing typically lasts between weeks to only a couple of days. Wear of tubing is indicated by an irreversible flattening of the tubing part that is pressed down onto the rollers of the peristaltic pump.

2. Check the method to see if a flush pump speed is used for the wash/uptake period. If it is, make the flush pump rate the same as the analysis rate and try it again. Inaccuracy can sometimes be traced to the inability of the pump tubing to recover its shape after being stretched.

3. Check the wash time for adequate rinse time. A 30 second wash time is adequate in most cases, but not if a slow pump rate is used or if a very long piece of tubing is used (as with the autosampler probe).

Poor Detection Limits

This problem can also be related to the poor precision problem discussed in "Poor Precision" on page 7-7 and is usually solved by approaching it as such.

However, if the loss of intensities is especially pronounced at lower wavelengths, it might be due to a dirty window mounted in the Purged Optical Path.

UV burn or a dirty mirror is characterized by a long term decline (six months or longer) of intensities.

Suggested Maintenance in Case of Poor Precision and Detection Limits

Maintenance refers to a series of periodic activities that should be performed on a periodic basis to optimize the short term and long term performance of the system. This section describes activities that the user of the instrument should perform.

Table 7-1. Typical maintenance schedule

Procedure	Frequency	Reference
Replace the pump tubing	weekly	See page 7-11
Clean the nebulizer	weekly	See page 7-12
Clean the plasma torch	weekly	See page 8-3

Replacing the Pump Tubing

Table 7-2 lists the tubing material that is suitable for a specific solvent type.

Table 7-2. Tubing materials

Type	Solvent Types
Tygon™	Aqueous solutions, strong acids and highly polar organic solvents (methanol and ethanol, for example)
Solvent Flex	Solvents of low polarity (for example alkanes, aromatics and halogenated hydrocarbons such as gasoline, kerosene, toluene, xylene, chloroform and carbon tetrachloride)

A pump tubing in poor condition is either flattened, hard or discolored.

Squashed tubing is usually caused by leaving the tubing pressure on the tubing when it is not used. To minimize squashing of the tubing, release the tubing pressure when the pump is not used, even for short periods.

Hardened and discolored tubing is caused by chemical reactions with the sample. While these phenomena cannot be avoided, they can be minimized by frequently flushing the tubing with deionized water.

Preventing the Blockage of the Nebulizer

The most common problem with the nebulizer is the blockage of the tip by the deposition of particulate matter. This section provides a series of suggestions to minimize blockage.

In most instances, blockage in the nebulizer is caused by either particulate matter (from the sample) or chemical deposits.

It normally occurs in the nozzle where the flow passages are extremely small and constriction is greatest in the annular gas channel between the tip of the capillary and the taper of the nozzle.

Tip Filter the sample. The sample capillary is more tolerant of particulate matter than the gas annulus. For high sample uptake nebulizers, the capillary frequently transports visibly turbid suspensions.

Thermo Fisher Scientific suggests that you filter or centrifuge the sample if the solids are not of analytical importance. Particulates and colloids of a polar nature such as silica, peptides, polyvalent metal hydroxides and others tend to build up on the (polar) glass and impede the fluid flow. In some instances, you can prevent deposition by adjusting the pH of the suspension away from its isoelectric point.

Tip It is very important that you rinse the nebulizer before you turn off the gas. It is advisable to rinse the nebulizer periodically throughout the sequence (depending on the chemistry of your samples).

- Solids might be deposited in the nozzle as sample solvent evaporates, further constricting the flow passages and reducing the signal. Rinsing minimizes or eliminates these deposits.
- Gas flow through most nebulizer models creates a venturi suction at the capillary tip, which can be used to draw rinse liquid through the capillary.
- After the testing of any salt solution, promptly rinse the system with a chemically compatible rinse consisting only of volatiles (this is especially necessary in flow injection analysis systems).

- A low-pH (acidic) sample should be followed by a low-pH rinse, a high-pH sample by a high-pH rinse, and an organic sample by an appropriate solvent.

  The final rinse should use deionized water and/or isopropanol.

Let the nebulizer dry before you turn off the gas. Make sure that the liquid feed is disconnected or arranged so that siphoning into the nebulizer cannot occur while the gas is off.

NOTICE Do not use ultrasonic cleaning to remove particulate matter, because sympathetic vibrations might be set up in the capillary causing it to bounce against the inside of the nozzle and chip. Also, do not use any wire to clean the capillary of the nebulizer. As a result, the performance of the nebulizer might decrease.

Removing Solids from the Nebulizer

If solids inside the nebulizer are interfering with the performance of the system, the following steps usually remove them and provide normal operation.

❖ **To rinse the nebulizer**

1. Introduce a rinsing agent into the shell, either from the gas input or the nozzle. A squeeze bottle works well in both cases. Fill all areas that were previously exposed to corrosive solutions.
2. Attach pressurized gas to the side-arm to expel the liquid.
3. Inject more rinse solution into the liquid input while the gas is flowing and let the venturi suction draw it through the capillary.
4. The final rinse should use isopropanol to accelerate the drying process.
5. Repeat the treatment, if you think it is necessary.
6. After the rinse is complete, dry the nebulizer completely.

Removing Particles from the Nebulizer

These operations are ranked in order of increasing aggressiveness. We recommend starting with the gentlest procedure and continuing with more aggressive procedures as required.

❖ **To remove particles**

1. Tap the liquid input line of the nebulizer gently against a wooden surface (or a surface of comparable hardness) to shake the particle loose. This helps the particle to move in the direction of increasing

inner diameter. Repeat the tapping as necessary to work the particle toward the appropriate exit orifice. Avoid extremely harsh tapping.

2. Gently tap or flick the shell soundly with your fingernail a few times. If this fails to dislodge the particle, close off the liquid and gas input tubes with your fingertips.

3. Force isopropanol backwards through the nozzle in a try to float the particle out through the larger gas and liquid input tubes. Use a squeeze bottle or plastic dropper with a tip that forms a good seal over the nebulizer nozzle. After the particle has been removed, remove the alcohol through the input tubes using compressed gas, or drain it onto lint-free tissue.

Removing Solid Deposits in the Sample Capillary

This step assumes that a passage still exists through the contaminating material (the tip is not entirely clogged).

❖ **To remove solid deposits**

1. Try to deduce the chemical nature of the deposit from the type of samples that are being analyzed. Select the solvent most likely to dissolve it.

2. Inject the solvent into the nozzle with a plastic dropper or squeeze bottle until the affected area is filled.

3. Expel the solvent with compressed gas.

4. Refill and expel the solvent repeatedly.

5. Examine the nebulizer under magnification. If the material is gone, rinse the nebulizer with isopropanol and dry it thoroughly.

6. Immerse the nozzle in a rinse solution. Warm the solution for stubborn deposits.

7. First rinse with pure solvent, then with isopropanol. Dry it thoroughly.

Removing Organic Matter

⚠CAUTION

Corrosive Acids. When you use the corrosive acids required to perform the cleaning procedures described here, take these precautions:

- Use a fume hood for the acid cleaning.
- Wear protective clothing.
- Use acid resistant laboratory gloves and glasses.
- Make yourself aware of all safety procedures in case of spillage or exposure to skin or eyes before you start the cleaning.

❖ **To remove organic matter**

1. Immerse the nozzle of your nebulizer in a hot cleaning solution of either chromic acid or sulphuric acid at 100 °C.
2. Let the solution rinse into the passages of the nebulizer until the affected area is filled.
3. Expel and replace the solution at intervals until the deposit is gone or until the chromium reduction (green color) ceases.
4. Rinse the nebulizer thoroughly with water, then with isopropanol and dry it completely.

Opening a Plugged Capillary (Fusible Solids, for Example Waxes)

Use this procedure only when no passage remains through the deposit.

❖ **To open a plugged capillary**

1. Carefully heat the nebulizer in the region of the capillary obstruction. Simultaneously (or intermittently) apply gentle gas pressure to the sample input tube.

NOTICE

Avoid overheating residues that might produce insoluble pyrolysis products.

2. Stop the treatment when you have opened a passage through the blockage.

8

Maintenance

This chapter describes maintenance procedures that the user must perform to ensure optimum performance of the system. iCAP PRO Series ICP-OES are designed for minimum maintenance. Therefore, routine user maintenance of the iCAP PRO Series ICP-OES is mainly concerned with keeping the instrument clean.

NOTICE Examine the sample introduction components regularly for contamination and wear. Failure to maintain the sample introduction system can result in erroneous results, poor precision, poor detection limits and blockages.

Before using any cleaning or decontamination methods except those specified by the manufacturer, make sure with the manufacturer that the proposed method does not cause damage to the equipment.

Contents

Maintenance and Cleaning Intervals

Routine and infrequent maintenance procedures to be performed by the user are listed in Table 8-1.

Table 8-1. Maintenance and Cleaning Intervals

Procedure	Interval	See page
Examine the tubing for clogging or damage.	daily	7-11
Examine the extraction tubing for damage.	daily	7-11
Examine the peristaltic pump and the tubing for wear.	daily	7-11
Clean the water filters.	quarterly	8-9
Leak test the gas lines.	once per year	
Leak test the water lines.	once per year	
Test the swim switch.	once per year	
Examine the extraction sensor.	once per year	
Examine the torch and door interlocks.	once per year	
Examine the rollers of the peristaltic pump.	once per year	
Examine the peristaltic bumpers.	once per year	
Clean the inner torch box	If the instruments shows a loss of performance.	8-4
Clean the spray chamber.	If the instruments shows a loss of performance.	8-6
Examine the torch.	If the instruments shows a loss of performance.	8-3
Clean the POP Window.	If the instruments shows a loss of performance.	8-6
Check the optical components.	If the instruments shows a loss of performance.	
Replace the molecular sieve.	If the detector shows signs of damage.	
Perform the chiller maintenance.	Refer to the manufacturer's maintenance instructions.	8-9

Cleaning the Instrument

It is the user's responsibility to carry out appropriate cleaning and decontamination of the equipment if hazardous material is spilled on or inside the equipment, sample introduction and accessories.

Cleaning the Covers

Clean up any spillage on the external covers or within the sample introduction areas immediately using appropriate safety precautions. Prolonged contact with solvents and acids could result in permanent damage.

Remove stains and marks on the covers with a soft cloth moistened with a mild detergent solution. Do not use any solvent-based cleaners.

NOTICE The iCAP PRO Series ICP-OES covers can be damaged by solvents (ethanol, for example) and concentrated acids.

Cleaning and Decontaminating the Sample Introduction System

NOTICE Failure to maintain the sample introduction system can result in erroneous results, poor precision, poor detection limits and blockages.

After use, follow the instrument shutdown procedures described on page 6-14. Clean components contaminated with sample residues.

Thermo Fisher Scientific recommends having several spares for each part available if blockages, sample contamination and breakages happen at critical moments during analysis.

Wear suitable protective clothing, glasses and gloves. See "Personal Protective Equipment" on page 4-9.

Cleaning the Torch

⚠CAUTION **Hot Parts.** Risk of burns. Let any hot components cool for at least 10 minutes before you remove them from the torch box.

⚠CAUTION **Sharp Edges.** Risk of cuts. Follow appropriate care and safety procedures to avoid breaking any glassware and causing injury to the operator. Handle any broken glassware with appropriate care and wear protective gloves.

❖ To clean the torch

1. Shut down the system as described on page 6-14.
2. Inspect the O-rings in the metal torch mount (three internal and two external). Replace them, if any wear or damage is visible.
3. Soak the torch in a dilute analytical-grade surfactant for five minutes to remove salt deposits.
4. To remove metallic deposits from the tip, separate the torch quartz section, immerse the tip of the torch in acid. A mixture of nitric and hydrochloric acid similar to aqua regia is suitable.
5. Rinse the torch with deionized water. Place it in a drying oven at 95 °C until it is dry. Rinsing with a volatile, zero residue, organic solvent (propanol is suitable) aids drying.

❖ To clean the torch of carbon deposits

NOTICE

Before you carry out this procedure, make sure that the torch is completely dry. If any liquid, even traces, resides in microfissures in the torch, then the torch may crack due to quick liquid expansion during the heating process.

1. Place the torch in a muffle furnace and heat it to 750 °C.
2. Open the door of the furnace to admit air for a few seconds.
3. Close the door. Let the temperature return to 750 °C.
4. Repeat step 1 to step 3 two or three times until the carbon is burned off.
5. Switch off the muffle furnace. Let it cool without opening the door. This takes several hours.

The furnace cools sufficiently slow to prevent stress in the quartz. Thermo Fisher Scientific recommends that at least two torches are rotated, so that you do not have to stop working while you are waiting for the torch to be cleaned.

Cleaning the Inner Torch Box

If high matrices like brines are analyzed over prolonged periods of time, deposits can form on the inside of the inner torch box. This inner torch box can be removed and cleaned.

Tip Two inner torch boxes can be used in turns, so that you do not have to stop working on the ICP-OES while the inner torch box is cleaned.

❖ To clean the inner torch box

1. Shut down the system as described on page 6-14.

⚠CAUTION

Hot Parts. Risk of Burns. Let any hot components cool for at least ten minutes before you remove them from the torch box.

2. Open the torch box door and remove the torch from the torch box.
3. Remove the axial POP cone from the torch box (if applicable).
4. Loosen the three screws on the rim of the inner torch box facing to you and take out the inner torch box. Do not loose the screws inside the torch box.

Figure 8-1. Screws to inner torch box

5. You can now also remove and clean the radial POP cone, if necessary.
6. Soak the torch box in a dilute analytical-grade surfactant for five minutes to remove salt deposits.
7. Rinse the torch box with deionized water. Place it in a drying oven at 95 °C until it is dry. Rinsing with a volatile, zero residue, organic solvent (propanol is suitable) aids drying.
8. Install back the inner torch box by reversing the steps 5 to 1.

Cleaning the Spray Chamber

If the spray chamber becomes greasy and droplets form on the inside (see Figure 8-2, left), then soak the spray chamber in a dilute analytical-grade surfactant for five minutes.

If the spray chamber becomes dirty or deposits form inside it, then soak the spray chamber in cold acid for two hours. A mixture of nitric acid and hydrochloric acid is normally suitable.

After cleaning, rinse the spray chamber in deionized water.

Figure 8-2. Spray chamber: dirty (left) - clean (right)

Cleaning the Nebulizer

NOTICE

A nebulizer should be cleaned with great care. Cleaning should only involve the soaking of the nebulizer in an appropriate cleaning solution or a gentle backflushing of cleaning solution through the tip of the nebulizer.

Do not clean a nebulizer by inserting any items into the capillary or by using an ultrasonic bath.

Rinse the nebulizer with deionized water or the organic solvent at the end of each day, or aspirate a cleaning solution through it. If the nebulizer is blocked, then use an Eluo™ Nebulizer cleaner or a similar tool to get rid of the blockage. See "Preventing the Blockage of the Nebulizer" on page 7-12 for details.

Cleaning the Purged Optical Path Window

⚠CAUTION

Hot Surface. Touching hot parts of the system might cause severe burns. Before you clean the Purged Optical Path (POP) window, turn off the plasma and wait for 10 minutes to let any hot areas cool to room temperature.

⚠CAUTION

UV Radiation. Risk of severe bodily harm. UV radiation might lead to severe eye injury or blindness. Do not look into the plasma if this is unexpectedly possible.

UV radiation is released when the spray chamber is disassembled. Do not open the access door or disassemble the spray chamber when the plasma is still on.

Use only the correct Thermo Scientific part for the spray chamber adapter. Do not bypass the safety interlocks. Shut down the system as described on page 6-14. Do not use the door to shut down the system.

❖ To clean the POP window

1. Shut down the system as described at page 6-14.
2. Open the door of the torch box.
3. Open the small access door on top of the torch box. See Figure 8-3.

Figure 8-3. Access to torch box

4. Withdraw the POP window holder. See Figure 8-4.

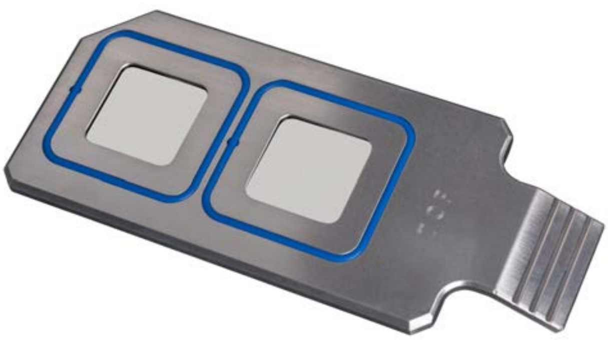


Figure 8-4. POP window holder

5. Clean the POP window using a lint-free cloth and clean water.
6. Repeat the cleaning procedure with methanol.
7. When the POP window is dry, re-insert it into the fore optic assembly.
8. If further cleaning is necessary, then remove the quartz window from the POP window holder. Remove the O-rings. Soak the holder in cold acid for two hours. A mixture of nitric acid and hydrochloric acid similar to aqua regia is suitable.
9. Rinse the POP window holder in deionized water.
10. Rinse the POP window holder with a volatile, zero residue, organic solvent (propanol is suitable) to aid drying.
11. Insert the POP window holder back.
12. Close the small access door on top of the torch box.
13. Close the door of the torch box.
14. Start up the instrument as described in Chapter 6, "System Operation."

NOTICE

If the POP window holder is not inserted correctly or the window inside the holder is missing, then fumes and heat of the plasma can cause damage to the fore optics optical components.

Moreover, wrong insertion of the POP window holder or a missing POP window results in an unpurged polychromator, leading to a loss in performance.

Preventive Maintenance

Although minimum user maintenance is required, periodic checks of performance are required by many laboratories. This is particularly important for customers subject to external standards and regulations (for example, ISO 9000, EPA, or NAMAS). Details of these options are available from a local Thermo Scientific office.

All electrical supplies, gas supplies and extraction must be checked to make sure that local health and safety guidelines and regulations are complied with. Examine the gas and cooling water for leaks at regular intervals.

Maintenance of the Water Chiller

It is critical to the performance of your instrument that the cooling fluid used for your iCAP PRO Series ICP-OES is made up correctly as specified on page 5-20. Replace the cooling fluid with new fluid periodically depending on the usage of your instrument. Also make sure that air filters and water filters are kept clean. Refer to the manufacturer's documentation of the chiller for details.

NOTICE Failure to maintain your chiller with the appropriate cooling fluid might cause internal damage to your instrument.

If an in-line water filter is fitted between your instrument and your chiller, then check it for cleanliness to prevent loss of instrument performance.

❖ **If the filter appears dirty**

1. Depending on the filter type, you may try to clean it. Otherwise, replace it.
2. Flush your water system.
3. Replace it with correctly made up cooling fluid.

Maintenance of the Gas Filters

If gas filters are fitted to your purge and plasma gas inlets, then check them for cleanliness to prevent loss of instrument performance. If the filters appear dirty, then replace them and check the quality of your gas supplies.

Fore Optics Alignment

For maximum sensitivity and optimum results, the alignment of the plasma image is critical. When a torch component has been replaced in the instrument, or if maintenance has been done to the fore optics, then carry out a fore optics alignment procedure.

❖ **To align the fore optics**

1. Click the Qtegra ISDS icon on the computer desktop.
2. On the Dashboard from the Auto Tuning tab, select **Fore Optics Alignment** to start the procedure.
3. Aspirate the so-called “Loaded Blank” solution as supplied with the iCAP PRO Series ICP-OES or any other aqueous 2 ppm Zn solution.
4. Observe the transport of the sample until it has entered the plasma.
5. Click **OK** to start the procedure.
6. Aspirate until the process is finished. The result of the test is displayed on the next wizard page as well as in the Autotune Report.

Thermo Fisher Scientific Service

This section contains information that concerns maintenance work that must be performed by Thermo Fisher Scientific personnel.

It is the user's responsibility to carry out appropriate cleaning and decontamination of the equipment before a service visit. The safety documentation must be made available to the Thermo Fisher Scientific field service engineer.

Returning Parts

⚠**CAUTION**

Hazardous Chemicals. Hazardous material might contaminate certain parts of your system during analysis. To protect our operating personnel, we ask you to adhere to special precautions when you send back parts to the factory for exchange or repair. If hazardous materials have contaminated instrument parts, then Thermo Fisher Scientific can only accept these parts for repair when they have been properly decontaminated.

Materials that might be toxic due to their structure and the applied concentration or which are reported in publications to be toxic are regarded as hazardous. Materials that will generate synergetic hazardous effects in combination with other present materials are also considered hazardous.

Parts contaminated by radioisotopes must not be returned to Thermo Fisher Scientific—neither under warranty nor within the exchange part program. If you are not sure whether parts of the system are possibly contaminated by hazardous material, then make sure that the Thermo Fisher Scientific field service engineer is informed before the engineer starts working on the system.

Your signature on the Health and Safety Form confirms that the returned parts have been decontaminated and that they are free of hazardous materials. This form is available on page B-5. Instead of copying or printing this page, request a copy from the Thermo Fisher Scientific field service engineer.

Services to be Performed by Thermo Fisher Scientific Service Only

The annual preventive maintenance must be performed by a Thermo Fisher Scientific field service engineer only.

A

Optional Accessories

This appendix describes frequently used accessories that are not part of the standard iCAP PRO Series ICP-OES installation.

Refer to the *iCAP PRO Series ICP-OES Consumables and Parts Catalog* for information about the available accessories. Request site requirement guides for any purchased accessories.

Contents

Autosampler

The autosampler can be configured to suit only one application, or several applications. Volume, number and type of sample influence the setup of the autosampler and the spectrometer.

To comply with safety and warranty requirements, the iCAP PRO Series ICP-OES, accessories and associated equipment must be installed by a certified Thermo Fisher Scientific service representative. For details, refer to the installation and operation documentation of your particular model of autosampler that is supplied with your iCAP PRO Series ICP-OES.

❖ **To set up the tubing to the autosampler**

For analysis with an autosampler, attach the capillary tubing that is attached to the end of the autosampler probe to the end of the sample pump tubing on the iCAP PRO Series ICP-OES. To minimize the sample volume required, minimize the length of the capillary tubing. It should allow free movement over the whole sample area of the autosampler.

Ultrasonic Nebulizer

The use of an ultrasonic nebulizer (USN) in conjunction with an ICP-OES has long been accepted as a simple and cost effective way to increase sensitivity and to decrease detection limits.

The CETAC™ U5000AT+ Ultrasonic Nebulizer converts more of the liquid sample into a usable aerosol, with an efficiency of 10 to 15%. See Figure A-1. The use of the CETAC U5000AT+ Ultrasonic Nebulizer is a simple and efficient way to gain an average 12-fold improvement in sensitivity over a full range of elements.

Figure A-1. CETAC U5000AT+ Ultrasonic Nebulizer

Tip To comply with safety and warranty requirements, the iCAP PRO Series ICP-OES, accessories and associated equipment must be installed by a certified Thermo Fisher Scientific service representative.

❖ **To set up the tubing to the ultrasonic nebulizer**

1. Put the ultrasonic nebulizer on the bench to the right of the iCAP PRO Series ICP-OES.
2. Remove the cyclone spray chamber, concentric nebulizer and associated pump tubing, if fitted.
3. Connect the sample pump tubing to the sampler capillary tubing of the ultrasonic nebulizer.
4. Use the 4 mm argon tubing supplied with the accessory to connect the nebulizer gas outlet of the iCAP PRO Series ICP-OES with the inlet on the rear panel of the ultrasonic nebulizer.
5. Using the adapter provided with the accessory, connect the sample outlet from the ultrasonic nebulizer to the plasma torch. Push the adapter firmly into the base of the torch until it “bottoms out” to prevent leaks.
6. Place the three drain tubes from the outlet of the pump on the rear panel into a suitable waste container.

Hydride Generation

A simple solution for increasing the detection capability of the hydride forming elements is delivered by hydride generation. The following options are available for confident detection of these elements at sub ppb concentration:

- The Basic Hydride Kit enables both non and hydride forming elements to be determined simultaneously.
- The integrated hydride generation accessory enables the maximum improvement in detection of the hydride forming elements.

Basic Hydride Kit

The Basic Hydride Kit consists of two pump tubes linked to a Y-piece and mixing loop. Acidified sample solution and sodium borohydride solution are mixed in the loop to generate the hydride gases. The mixture is passed to the spray chamber where separation of liquid and hydride gases takes place.

Setup

To ensure a thorough mixing of the sodium borohydride solution with the sample, a mixing loop is provided after the Y-piece before connecting it to the nebulizer. See Figure A-2.

Install the pump tubing for the Basic Hydride Kit in a manner similar to the sample tube and the correct tension set.

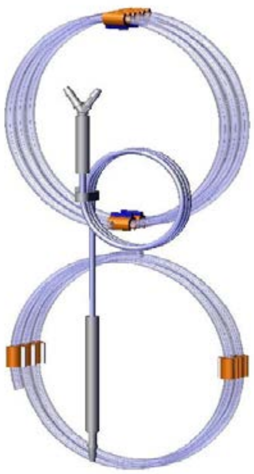


Figure A-2. Basic Hydride Kit with mixing loop

All standard and sample solutions must be acidified to 10% hydrochloric acid before analysis. A solution of 0.5% sodium borohydride prepared in 0.5% sodium hydroxide solution is used as the reducing agent.

❖ To set up the Basic Hydride Kit

1. Place the orange/blue pump tubing in the sodium borohydride solution. The orange/white pump tubing is used for the sample and standard solutions.

   For best plasma stability, it is recommended that sodium borohydride and an acidified wash solution are pumped continuously between measurements.

2. Ignite the plasma. Set the operating parameters listed in Table A-1 using the plasma control dialog.

Table A-1. Operating parameters for on-line hydride generation

Parameter	Setting
RF power	1350 W
Auxiliary gas	0.5 L/min
Nebulizer gas typical operating range	about 0.5 L/min
Pump speed	30 rpm

3. Aspirate a solution of 50 µg/L arsenic solution and check the signal in the subarray window.

   Compare the net signal intensity to the intensity of the background signal.

4. Vary the nebulizer flow over the range 0.3–0.6 L/min while you check the signal-to-background ratio until a maximum is found.

   This gives a good compromise set of operating conditions.

If better sensitivity is required for a particular element, then use that element for optimization.

Integrated Hydride Generation Accessory

The integrated hydride generation accessory (see Figure A-3) requires the use of a four channel sample pump. It consists of an integrated acrylic reaction cell and a gas/liquid separator for the generation and the separation of the hydride gases. It is connected directly to the plasma torch after the removal of the spray chamber.

Figure A-3. Integrated hydride generation accessory

❖ **Assembly of the integrated hydride generation accessory**

- The unit is supplied with all pump tubing connected.
- The user must attach the pump tubing to the peristaltic pump on the ICP-OES and set up the Gas Liquid Separator.
- The unit should sit comfortably in the kitchen area of the ICP-OES to the left of the peristaltic pump. The lengths of Tygon™ peristaltic pump tubing supplied are sufficiently long so that the acid blank (orange/yellow) and reductant (black) pump tubing go directly into their respective solution containers.
- The sample and drain Tygon peristaltic tubing are supplied connected to the Ultem™ sample probe and the 3 mm ID PTFE drain tubing, respectively.
- Prepare the gas liquid separator as described at page A-7 before use.

Gas Liquid Separator (GLS)

When it is assembled, the reaction zone of the GLS (see Figure A-4) contains 4 mm glass beads. They minimize the dead volume of the zone and ensure proper mixing of the carrier gas and liquid reagents. It also contains a semi-permeable Teflon™ membrane to prevent moisture and salts from being carried over into the tube leading to the ICP torch. These parts are supplied with the unit and must be fitted before the accessory is used.

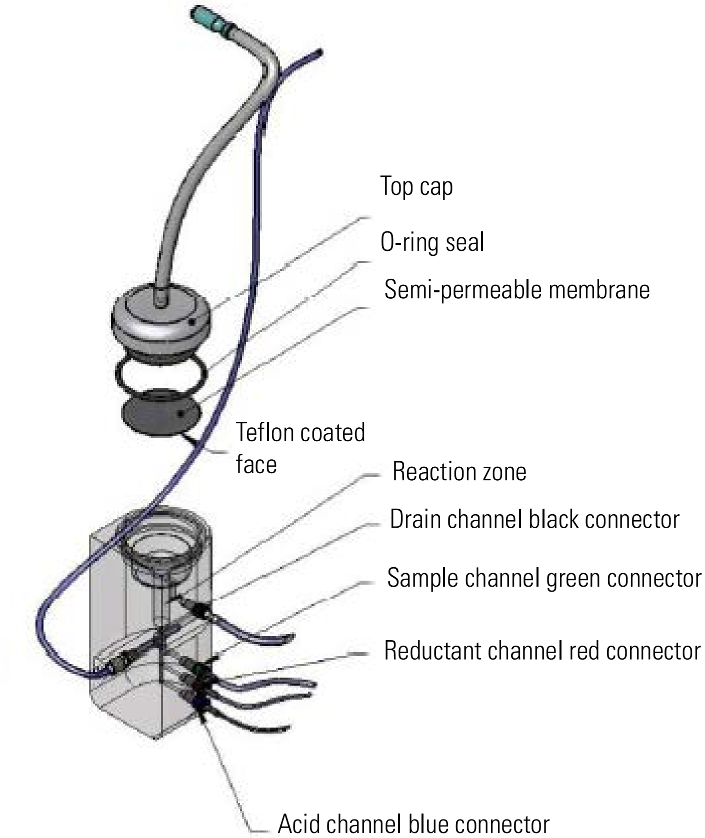


Figure A-4. Schematic of Gas Liquid Separator

❖ **To prepare the Gas Liquid Separator**

1. Unscrew the cap of the GLS. See Figure A-4.
2. Add a sufficient quantity of the supplied 4 mm glass beads to fill the reaction zone.

   There should be no beads on the floor of the expansion volume (at the top of the reaction zone column).

   Prevent the glass beads from falling into the drain where they might cause blockage.
3. Orientate one of the 47 mm Teflon membranes supplied so that the shiny side is to the top. Place it in position.
4. Carefully re-fit the O-ring seal and the cap. Make sure that the position of the membrane is not disturbed.

Pump Tubing

The unit is supplied with four types of pump tubing, which fit onto the standard pump and the mini four channel peristaltic pump. The colors of the pump tubing bridges correspond to the color-coded connectors on the GLS unit according to Table A-2.

Table A-2. Color coded connectors at GLS unit

Connector color	Transport function	Pump tubing
Black	Drain	black/white
Green Yellow	Sample	green/green
Red	Reductant	black/black
Blue	Acid	orange/yellow

The peristaltic pump of the ICP-OES operates in an counterclockwise direction. Place the GLS to the left of the pump in the kitchen area.

All of the pump tubing (with the exception of the drain) attach with the inlet on the right side of the pump. That is, from the sample/reductant/acid blank solutions across the pump and into the GLS.

The drain is the only tubing that needs to be connected in the opposite direction. That is, the tubing is passed under the pump so that the drain outlet is on the left hand side.

Drain

The black (drain) channel must be fitted to pump tubing with black/white bridges, that is a single piece of pump tubing will have a black bridge fitted at one end and white bridge fitted at the other end.

This tubing has an ID of 3.17 mm and an OD of 4.85 mm. It runs from the black connector of the GLS across the peristaltic pump and to a drain.

Connect a suitable length of the 1.6 mm OD tubing, using the barbed connector supplied, from the end of the black/white pump tubing (at the peristaltic pump) to a suitable low level drain or wide-necked plastic container.

Tip The drain tubing is connected opposite to the other pump tubings, to allow draining from the GLS.

Sample Uptake

The green/yellow (sample) channel must be fitted to pump tubing with green bridges.

This tubing has an ID of 1.85 mm and an OD of 3.53 mm. It goes from the sample across the pump to the GLS.

Reductant

The red (reductant) channel must be fitted to pump tubing with black bridges.

This tubing has an ID of 0.76 mm and an OD of 2.43 mm. It goes from the reductant across the pump to the GLS.

The PEEK™ barb is fitted with an additional piece of 1.6 mm ID Tygon™ tubing for easy connection to the peristaltic pump tubing.

Acid

The blue (acid) channel must be fitted to pump tubing with yellow/orange bridges.

This tubing has an ID of 0.5 mm and an OD of 2.33 mm. It goes from the acid across the pump to the GLS.

The PEEK barb is fitted with an additional piece of 1.6 mm ID Tygon tubing, for easy connection to the peristaltic pump tubing.

❖ **To fit the pump tubing**

1. Release the tension fingers to free the pump tension arms.
2. Feed the pump tubing around the pump rollers.
3. Stretch the pump tubing slightly. Fit the bridges under the bridge retaining pillars.
4. Push the ends of the tubing over the appropriate push on connectors on the GLS connection panel.
5. Move the tension arm back over the rollers. Confirm that the tubing is properly located beneath it.
6. Return the tension finger to the normal position. Adjust the pressure screw to release the pressure on the tubing.

❖ **To install the gas supply**

1. Connect a supply of argon (normally from the nebulizer supply on the front of the sample introduction area of the ICP-OES) to the supplied 4 mm OD rigid black tubing to the gas inlet at the rear of the GLS.
2. Regulate the flow between 0.3–0.6 L/min.

❖ To optimize the unit

1. Ignite the plasma. Set the operating parameters listed in Table A-3 using the plasma control dialog.

Table A-3. Operating parameters for integrated hydride generation

Parameter	Value
RF power	1350 W
Coolant gas	14 L/min
Auxiliary gas	0.5 L/min
Nebulizer gas	0.3–0.6 L/min
Pump speed	30 rpm

2. Aspirate a solution of 50 µg/L arsenic solution. Check the signal in the subarray window.

3. Compare the net signal intensity to the intensity of the background signal.

4. Vary the nebulizer flow over the range 0.3–0.6 L/min while checking the signal-to-background ratio until a maximum is found.

   This gives a good compromise set of operating conditions.

   If better sensitivity is required for a particular element, then use that element for optimization.

For each hydride group element, there is an optimum nebulizer gas flow rate that yields the maximum sensitivity for that element. For mercury, the analytical sensitivity increases as the nebulizer gas rate decreases.

Low nebulizer gas rates require longer flush times (to let the hydride travel to the plasma), and, except for mercury, offer no benefits.

Higher nebulizer gas flows allow the use of shorter flush times, at the expense of some analytical sensitivity. They can be used if you want to complete your analysis as quickly as possible.

The sample pump speed also exerts an influence on the sensitivity: as the pump speed is increased, the analytical sensitivity increases, but so does the consumption of samples and reagents.

The flush time required decreases at higher pump speeds. Reducing the pump speed reduces the reagent consumption at the expense of the analytical sensitivity and increased flush time.

The default pump speed is 30 rpm. Good results can be obtained with pump speeds up to 45 rpm.

Sheath Gas Adaptor

To increase long-term analytical stability for solutions containing more than 15% m/v dissolved solids, a sheath gas adaptor should be used. A sheath gas envelopes the sample aerosol tangentially. It prevents contact with the injector tube and reduces the deposition of the sample on the injector. The sheath gas is introduced between the spray chamber and the torch using the sheath gas adaptor, which must be supplied with a constant gas flow of argon via a mass flow controller.

The additional gas mass flow controller can be used for the addition of the sheath gas.

NOTICE Make sure that the correct gas line (argon) for sheath gas is attached to the Add Gas inlet at the rear side of the instrument (see Figure 5-14 on page 5-22) and that **Front Outlet** is selected on the Dashboard of the instrument Home Page of the Qtegra ISDS Software.

Installation of the Sheath Gas Adaptor

Required Material

Part	P/N
Sheath gas adaptor	BRE0011651
L-Bow push fit 6 mm	BRE0013208
Tubing, 6 mm OD 4 mm ID, soft polyolefin white	BRE0013210

❖ **To install the sheath gas adaptor**

1. Remove the spray chamber and the spray chamber adaptor from the injector holder.

Figure A-5. Spray chamber and the spray chamber adaptor

2. Insert the sheath gas adaptor into the injector holder.
3. Connect the L-bow push fit 6 mm onto the sheath gas adaptor .
4. Connect the white 6 mm OD tubing onto the free side of the L-bow push fit connector.

Figure A-6. Sheath gas adaptor inserted and connected

5. Insert the loose end of the tubing into the 6 mm push fit connector in the front of the instrument below the nebulizer gas connection.

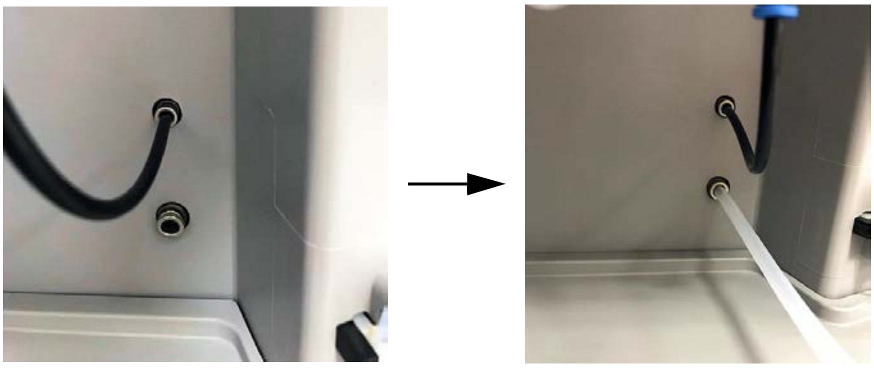


Figure A-7. Connecting the sheath gas tubing to the instrument

6. Reattach the spray chamber to the sheath gas adaptor.

Figure A-8. Spray chamber and sheath gas adaptor

Legal Documents

Contents

WEEE Compliance

This product is required to comply with the European Union's Waste Electrical & Electronic Equipment (WEEE) Directive 2012/19/EU. It is marked with the following symbol:

Thermo Fisher Scientific is registered with B2B Compliance (B2Bcompliance.org.uk) in the UK and with the European Recycling Platform (ERP-recycling.org) in all other countries of the European Union and in Norway.

If this product is located in Europe and you want to participate in the Thermo Fisher Scientific Business-to-Business (B2B) Recycling Program, send an email request to weee.recycle@thermofisher.com with the following information:

- WEEE product class
- Name of the manufacturer or distributor (where you purchased the product)
- Number of product pieces, and the estimated total weight and volume
- Pick-up address and contact person (include contact information)
- Appropriate pick-up time
- Declaration of decontamination, stating that all hazardous fluids or material have been removed from the product

This recycling program is not for biological hazard products or for products that have been medically contaminated. You must treat these types of products as biohazard waste and dispose of them in accordance with your local regulations.

EU REACH Statement

The European Commission promulgated legislation that covers the registration, evaluation, authorization and restriction of chemicals within the European Union community under (EC) No 1907/2006. This regulation is commonly known as REACH. Thermo Fisher

Scientific is committed to meeting all compliance obligations under REACH. As per Article 33 of the Regulation, this product may include items which contain more than 0.1% by weight of some SVHC Candidate Substance. Some electronic parts and copper alloys can contain lead.

ETL Listing Mark

Conforms to UL Std. UL 61010-1:2012 Ed.3 +R:21Nov2018
Conforms to UL Std. UL 61010-2-010:2015 Ed.3
Conforms to UL Std. UL 61010-2-061:2015 Ed.3
Cert. to CSA Std. C22.2#61010-1:2012 Ed.3 + U1;U2;A1
Cert. to CSA Std. C22.2#61010-2-010:2015 Ed.3
Cert. to CSA Std. C22.2#61010-2-061 Issued: 2015/01/11

Declaration of Conformity

-Original-

EU-Konformitätserklärung
EU Declaration of Conformity

CE

Thermo Fisher Scientific (Bremen) GmbH
Hanna-Kunath-Str. 11
28199 Bremen, Germany

Wir erklären hiermit, dass die folgenden Produkte
We hereby declare that the following products

Bezeichnung: *Designation:*	**Optische Emissionsspektrometer** *Optical Emission Spectrometer*
Modell: *Model:*	**Thermo Scientific iCAP PRO Serie** *Thermo Scientific iCAP PRO Series* *(iCAP PRO, iCAP PRO X, iCAP PRO XP, iCAP PRO XPS)*

alle einschlägigen Anforderungen der folgenden Richtlinien erfüllen:
fulfill all the relevant requirements of the following directives:

Niederspannungsrichtlinie *Low Voltage Directive*	**2014/35/EU** *2014/35/EU*
Richtlinie über elektromagnetische Verträglichkeit *Electromagnetic Compatibility Directive*	**2014/30/EU** *2014/30/EU*
RoHS-Richtlinie *RoHS Directive*	**2011/65/EU** *2011/65/EU*

Die folgenden einschlägigen harmonisierten Normen wurden zugrunde gelegt:
The following relevant harmonized standards were used:

EN 61010-1:2010 *EN 61010-1:2010*	**EN 61326-1:2013** *EN 61326-1:2013*

Für die Zusammenstellung der technischen Unterlagen ist bevollmächtigt:
Person authorized to compile the technical file:

Jörg Behrens (Director Operations)
Thermo Fisher Scientific (Bremen) GmbH

Jörg Behrens	Bremen, 2019-09-03
Unterschrift *Signature*	**Datum** *Date*

ThermoFisher SCIENTIFIC

Health and Safety Form

This Decontamination Declaration Form must be completed for all materials returned to Thermo Fisher Scientific. It should be sent to the destination by e-mail, with approval from an authorized person. A signed hardcopy should be attached to the outside of the package with shipping paperwork and a further copy should be placed inside the packaging. The receiving Thermo Fisher Scientific office can help with this form and supply a return number, shipping address and e-mail address. This form can be used to request warranty. Use the text "not used" to indicate a field not being used. Where a Thermo Scientific part number is not known, add the supplier name (as for the examples below).

1. General information

Thermo Fisher Scientific contact name for delivery ________________

Thermo Fisher Scientific receiving site ________________

Customer ________________ Instrument type ________________

Address ________________ Instrument SN ________________

________________ Order number ________________

Phone ________________ Return number ________________

E-Mail ________________ Medical Device ☐ Research Use Only ☐

SAP Service Notification ________________

Part Number	Quantity	Material Description	Error Description / Reason for Return	Return Part Serial No

2. Condition of the material or instrument

Has the material or instrumentation been removed from the shipping packaging or in contact with

- pump fluids
- service fluids
- samples
- standard solutions
- other chemicals
- hazardous materials

Tick the applicable check box.

☐ Yes → go to section 3

☐ No → go to section 5

3. Contamination

Use the check boxes to state any contaminants the material/instrumentation been exposed to. Contaminated materials must not be shipped to Thermo Fisher Scientific. If any exposure boxes are ticked, select 'Yes', if none, select 'No'.

	☐ toxic		☐ flammable		☐ serious health hazard		☐ corrosive		☐ oxidizing
	☐ hazardous to environment		☐ explosive		☐ gas under pressure		☐ other harmful substances		
	☐ biological contaminated		☐ radioactive contaminated						

☐ Yes → go to section 4

☐ No → go to section 5

4. Description of process substances and/or compounds

Which substances have been in contact with the material or instrumentation? (trade name and/or chemical term of service fluids and substances; properties of substances or compounds according to the Material Safety Data Sheet; e.g. toxic, flammable, corrosive, radioactive)

	Part Number	Trade Name	Chemical / Substance Name / Properties
a)			
b)			
c)			
d)			
e)			
f)			

5. Legally binding declaration

Has the material/instrument undergone a decontamination process? ☐ Yes → go to section 6 ☐ No

Is the material/instrument safe to handle for Thermo Fisher Scientific and third-party personnel? ☐ Yes ☐ No

Components, materials and/or instruments that have been contaminated to a harmful level by whatever substances and/or compounds as stated in sections 3. and 4. above will not be accepted without written evidence of proper decontamination.
I hereby declare that the instrument has undergone successfully all required decontamination procedures and is safe to handle for Thermo Fisher Scientific and/or third-party service personnel or suppliers such as Pfeiffer Vacuum, Leybold Vacuum, Edwards Vacuum products, or others.
I confirm that all information, which is supplied on this form, is accurate, complete and sufficient to judge any contamination level. I acknowledge and agree that I will be liable for any personal injury or any other damage, which might result from a false, inaccurate or incomplete statement and that I will indemnify and defend Thermo Fisher Scientific and/or any other concerned third party for and against any liabilities, claims, losses, and/or damages of all kinds arising out of and/or caused by such false, inaccurate or incomplete statements.
Thermo Fisher Scientific reserves the right not to process refunds or returns where the declared or observed use or previous contamination of the product/material has by Thermo Fisher Scientific judgement impacted its integrity.

6. Detailed description of the decontamination process used

Part Number	Serial Number	Describe the decontamination process

Return Number	Name of authorized person (block letters)	Date	Signature	Company stamp

Glossary

This section lists and defines terms used in this manual. It also includes acronyms, metric prefixes, symbols, and abbreviations.

A B C D E F G H I J K L M N O P Q R S T U V W X Y Z

A

ABS Acrylonitrile butadiene styrene.

AC abbr. for alternating current, for example, an electric current that reverses its direction at regularly recurring intervals.

Acid Resistant Sample Inlet resistant inlet with a special nebulizer chamber and torch.

ADC abbr. for analog-to-digital converter; a device that converts data from analog to digital form.

AL/VI abbr. for Aluminum/Viton™; material used for gaskets.

analog mode the detection mode "Analog" can be used for high signals between 5×10^4 to 5×10^9 cps. The electrical current measured is converted into the intensity information, which is stored in the data file.

aux gas auxiliary gas (argon), serves to generate the plasma.

B

BEC abbr. for Background Equivalent Concentration (normally in ppt); n=10, depends on the concentration in the blank.

$$BEC = \frac{(blank\ intensities) \times (concentration\ of\ standard)}{(intensity\ standard - average\ intensity\ blank)}$$

BLK abbr. for a blank (analyte).

C

°C degrees Celsius.

CE European conformity. Mandatory European marking for certain product groups to indicate conformity with essential health and safety requirements set out in European Directives.

CID Charge Injection Device.

cool gas serves to prevent the glass torch from melting.

counting mode the detection mode "Counting" is a digital measurement and counts electron pulses. It is very sensitive and can be used for the detection of low signals. During acquisition, the number of occurrences is used to generate the intensity information (in counts per seconds) that is stored in the data file. The operating range of the counting mode is between 0 and ~5×10^6 cps.

D

DAC abbr. for digital-to-analog converter; a device that converts data from digital to analog form.

DC abbr. for direct current, for example, an electric current flowing in one direction only.

DDS abbr. for direct digital synthesizer.

DSP abbr. for digital signal processor.

E

eV abbr. for electron volt; the energy gained by an electron which accelerates through a potential difference of one volt.

F

f femto (10^{-15}).

°F degrees Fahrenheit.

FTP file transfer protocol.

G

G Gauss; giga (10^9).

GC gas chromatograph; gas chromatography.

GC/MS gas chromatograph / mass spectrometer.

GLP Good Laboratory Practice.

GLS Gas Liquid Separator.

GND electrical ground.

GUI graphical user interface.

H

h hour.

h height.

HPLC high-performance liquid chromatograph.

HR abbr. for High Resolution.

HV high voltage.

Hz hertz (cycles per second).

I

ICIS™ Interactive Chemical Information System.

ICP-OES Inductively Coupled Plasma Optical Emission Spectrometer.

ID inside diameter.

in. inch.

internal standards are used in ICP-MS analyses to compensate for drift effects in response or sensitivity caused by various processes in sample introduction or ion extraction.

I/O input/output.

IP internet protocol.

IRIS Isotope Ratio Infrared Spectrometer.

ISDS Intelligent Scientific Data Solution.

ISM Industrial Scientific and Medical.

ISO abbr. for International Organization for Standardization.

L

LAN local area network.

lb pound.

LC liquid chromatograph; liquid chromatography.

LED light-emitting diode.

linear regression type: linear regression analyses.

LOD abbr. for Limit of Detection (normally in ppt); n = 10, depends on the stability of the blank measurement.

$$LOD = \frac{(3 \times stdev\ of\ BLK\ intensities) \times (concentration\ of\ STD)}{(intensity\ STD - average\ intensity\ BLK)}$$

LR bbr. for Low Resolution.

M

MEK methylethylketone.

MFC mass flow controller.

MSDS Material Safety Data Sheet.

N

NDRO nondestructive readout.

O

OD outside diameter.

P

PCB printed circuit board.

PCL abbr. for Process Control Language.

PEEK Polyether ether ketone.

P/N part number.

POP window Purged Optical Path window.

ppb abbr. for parts per billion. A unit of measure expressed as parts per billion. Equivalent to 1×10^{-9}. Similar to µg/L or micrograms per liter.

PPE personal protective equipment.

PPS polyphenylene sulphide.

ppt abbr. for parts per trillion. A concentration unit of chemical constituents in solution, the weight of solute per unit volume of solvent.

psig pounds per square inch, gauge.

PTFE Polytetrafluorethylene.

R

RAI Random Access Integration.

RAM random access memory.

regression types are used in the creation of calibration curves during a sequence of analyses: the software offers four types: linear, thru zero, weighted, and square fit.

RF radio frequency.

RoHS Restriction of Hazardous Substances. EU directive on the restriction of the use of certain hazardous substances in electrical and electronic equipment.

S

s second.

square fit regression type: the fit is performed with a second order (quadratic) function.

STD abbr. for standard solution (analyte).

T

TCP/IP transmission control protocol/Internet protocol.

U

u symbol for atomic mass unit.

UPS uninterruptible power supply.

UPW Ultra Pure Water.

USN ultrasonic nebulizer.

V

V AC volts alternating current.

V DC volts direct current.

W

weighted regression type: linear regression weighted by the reciprocal of the standard deviation (1/standard deviation).

WEEE European Union Waste Electrical and Electronic Equipment Directive. Provides guidelines for disposal of electronic waste.

Index

U

V

W

Y