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iCAP 7000 Plus Series ICP-0ES

Operating Manual

BRE0004150 Revision C January 2018



iCAP 7000 Plus Series ICP-OES

Operating Manual

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Original Operating Instructions

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Technical Data for iCAP 7000 Plus Series ICP-0ES Systems

The table below summarizes the most important technical specifications of the iCAP 7000 Plus Series ICP-OES systems, in relation to the installation. See the respective chapters of the manual for details and additional information.

Spectrometer Depth × width × height Neight Neigh	Parameter	Specification		Value
Power Requirements	Instrument Properties			
Spectrometer Input Power Power Prise a 2.88 kVA 2.88 kVA 3.2 A Cooling Water Requirements Temperature fluctuation of Pressure and the pressure of t	Spectrometer	7		
Power Fuse* 32 A	Power Requireme	ents		
Chiller Supply rate Temperature 5 L/min Temperature 15 to 30 °C, 5 °C below ambient temperature Max. temperature fluctuation Pressure 0.6 MPa (6 bar, 90 psi) Cooling capability 750 W minimum Flow rate > 5 L/min Water Quality Quality distilled or deionized water Solid residual Solid residual < 50 μm particle size Corrosion inhibitor 2.5% v/v Fargon Purity 99.995% or better, < 10 ppm H ₂ O, < 10 ppm O ₂ Maximum supply rate 25 L/min Pressure 0.55 MPa (5.5 bar, 80 psi), at maximum 0.6 MPa (6 bar, 90 psi) Nitrogen Purity 99.995% or better, < 10 ppm H ₂ O, < 10 ppm O ₂ (optional) Maximum supply rate 15 L/min (perssure 0.55 MPa (5.5 bar, 80 psi) 15 L/min Compressed Air Requirements (iCAP 7400 ICP-0ES fitted with an additional gas mass flow controller, or the iCAP 7600 ICP-0ES) Compresser 100 mL/min 100 mL/min Purity 99.99% or better, < 100 ppm H ₂ O Supply rate 0.2 MPa (2 bar, 30 psi) Operating Environment 100 m	Spectrometer	Input	Power	2.88 kVA
Temperature	Cooling Water Re	quirements		
Max. temperature fluctuation Co.2 °C/h Pressure O.6 MPa (6 bar, 90 psi) Cooling capability 750 W minimum Flow rate > 5 L/min Water Quality distilled or deionized water Solid residual <50 µm particle size Corrosion inhibitor 2.5% v/v Gas Requirements Argon Purity 99.995% or better, <10 ppm H ₂ O, <10 ppm O ₂ Maximum supply rate 25 L/min Pressure 0.55 MPa (5.5 bar, 80 psi), at maximum 0.6 MPa (6 bar, 90 psi) Nitrogen Purity 99.995% or better, <10 ppm H ₂ O, <10 ppm O ₂ (optional) Maximum supply rate 15 L/min Pressure 0.55 MPa (5.5 bar, 80 psi) Compressed Air Requirements (iCAP 7400 ICP-0ES fitted with an additional gas mass flow controller, or the iCAP 7600 ICP-0ES Purity 99.99% or better, <100 ppm H ₂ O Supply rate 100 mL/min Pressure 0.2 MPa (2 bar, 30 psi) Operating Enviroment Laboratory Temperature 15-35 °C Max. temperature fluctuation 2 °C/hour Humidity 20-80% (15-30 °C), 20-60% (30-35 °C); non-condensing and non-corrosive atmosphere Max. altitude 3000 m above sea level Extraction Tube diameter 125 mm	Chiller	Supply ra	ite	> 5 L/min
Pressure		Temperat	ture	15 to 30 °C, 5 °C below ambient temperature
Cooling capability Flow rate 750 W minimum Flow rate Water Quality Solid residual Corrosion inhibitor distilled or deionized water Solid residual Corrosion inhibitor 2.5% v/v Gas Requirements Argon Purity Maximum supply rate Pressure 99.995% or better, < 10 ppm H₂O, < 10 ppm O₂		Max. tem	perature fluctuation	< 0.2 °C/h
Flow rate S L/min		Pressure		0.6 MPa (6 bar, 90 psi)
WaterQuality Solid residual Corrosion inhibitor< 50 μm particle size 2.5% v/vGas RequirementsArgonPurity Maximum supply rate Pressure99.995% or better, < 10 ppm H₂O, < 10 ppm O₂ 25 L/min PressureNitrogenPurity Pressure99.995% or better, < 10 ppm H₂O, < 10 ppm O₂		Cooling	capability	750 W minimum
Solid residual Corrosion inhibitor Solid residual Corrosion inhibitor Z5% v/v Gas Requirements Argon Purity Pressure Purity Purity Pressure Purity Purity Purity Purity Purity Purity Purity Pressure Purity Purity Pressure Purity Pressure Purity Purity Pressure Purity		Flow rate	:	> 5 L/min
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Gas Requirements Argon Purity 99.995% or better, < 10 ppm H ₂ O, < 10 ppm O ₂ Maximum supply rate 25 L/min Pressure 0.55 MPa (5.5 bar, 80 psi), at maximum 0.6 MPa (6 bar, 90 psi) Nitrogen Purity 99.995% or better, < 10 ppm H ₂ O, < 10 ppm O ₂ (optional) Maximum supply rate 15 L/min Pressure 0.55 MPa (5.5 bar, 80 psi) Compressed Air Requirements (iCAP 7400 ICP-0ES fitted with an additional gas mass flow controller, or the iCAP 7600 ICP-0ES) Purity 99.99% or better, < 100 ppm H ₂ O Supply rate 100 mL/min Pressure 0.2 MPa (2 bar, 30 psi) Operating Environment Laboratory Temperature 15-35 °C Max. temperature fluctuation 2 °C/hour Humidity 20-80% (15-30 °C), 20-60% (30-35 °C); non-condensing and non-corrosive atmosphere Max. altitude 3000 m above sea level Extraction Tube diameter		Solid resi	dual	< 50 μm particle size
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Nitrogen Purity 99.995% or better, < 10 ppm H ₂ O, < 10 ppm O ₂ (optional) Maximum supply rate 15 L/min Pressure 0.55 MPa (5.5 bar, 80 psi) Compressed Air Requirements (iCAP 7400 ICP-0ES fitted with an additional gas mass flow controller, or the iCAP 7600 ICP-0ES) Purity 99.99% or better, < 100 ppm H ₂ O Supply rate 100 mL/min Pressure 0.2 MPa (2 bar, 30 psi) Operating Environment Laboratory Temperature 15–35 °C Max. temperature fluctuation 2 °C/hour Humidity 20–80% (15–30 °C), 20–60% (30–35 °C); non-condensing and non-corrosive atmosphere Max. altitude 3000 m above sea level Extraction Tube diameter 125 mm		Maximur	n supply rate	25 L/min
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Max. altitude 3000 m above sea level Extraction Tube diameter 125 mm		Flumidity		· ·
Extraction Tube diameter 125 mm		May alti	tude	•
	Zatiuction			

^a dedicated wall outlet

Technical Data for iCAP 7000 Plus Series ICP-0ES Systems

Chapter 1 Using this Manual

Contents

- About this Manual on page 1-1
- Typographical Conventions on page 1-2
- Reference Documentation on page 1-4
- Contacting Us on page 1-5

About this Manual

This *iCAP 7000 Plus Series ICP-OES Operating Manual* contains precautionary statements that can prevent personal injury, instrument damage, and loss of data if properly followed. It also describes the modes of operation and principle hardware components of your Thermo Scientific™ iCAP™ 7000 Series ICP-OES. In addition, this manual provides step-by-step instructions for cleaning and maintaining your instrument.

This *iCAP 7000 Plus 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). Read this manual carefully before using the instrument and keep it for future reference.

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 might result in instrument damage and/or operator injury.

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 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

The following headings are used to show the organization of topics within a chapter:

Chapter 1 Chapter Name

Second Level Topics

Third Level Topics

Fourth Level Topics

Reference Documentation

This *iCAP 7000 Plus Series ICP-OES Operating Manual* represents the Original Operating Instructions. In addition to this manual, Thermo Fisher Scientific provides other documents for the iCAP 7000 Plus Series ICP-OES that are not part of the Original Operating Instructions. Reference documentation for the iCAP 7000 Plus Series ICP-OES includes the following:

- iCAP 7000 Plus Series ICP-OES Pre-Installation Requirements Guide
- iCAP 7000 Plus Series ICP-OES Quick Start Guide
- iCAP 7000 Series ICP-OES Accessories Guide
- Qtegra for iCAP 7000 Series ICP-OES Software Manual

The Qtegra *iCAP 7000 Plus Series ICP-OES Online Help* page provides help with running your instrument, including software guides. See Figure 1-1.

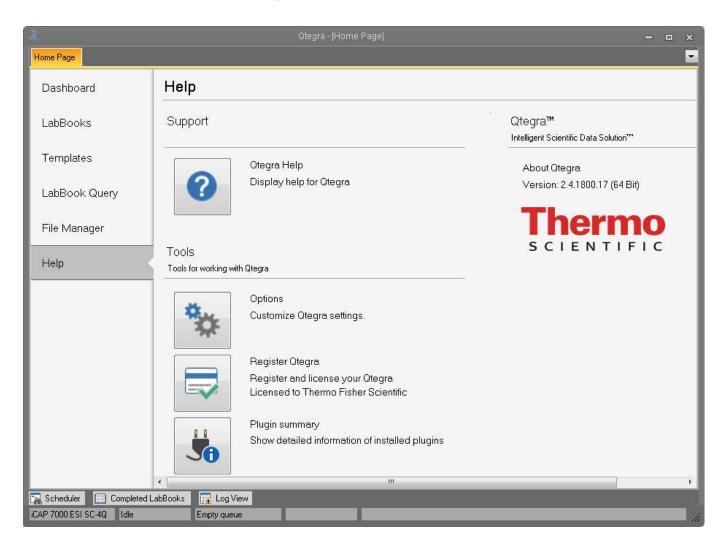


Figure 1-1. Otegra ISDS Software Help topics

Contacting Us

There are several ways to contact Thermo Fisher Scientific.

Assistance

To get brochures and ordering information

Web site www.thermofisher.com/ICP-OES

To contact Customer Service

Web site www.unitylabservices.com

❖ To get user manuals for your product

Customer www.thermoscientific.com/Technicaldocumentation SharePoint

1. With the serial number (S/N) of your instrument, request access on our customer SharePoint as a **customer**.

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.

2. Download current revisions of user manuals and other customer-oriented documents for your product. Translations into other languages may be available there as well.

Suggestions to the Manual

❖ To suggest changes to this manual

• Send your comments to:

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

• Send an e-mail message to the Technical Editor at

documentation.bremen@thermofisher.com

You are encouraged to report errors or omissions in the text or index. Thank you.

Using this Manual Contacting Us

Chapter 2 System Accessories

In addition to the iCAP 7000 Plus Series ICP-OES, some of the system accessories are required whereas others are optional.

Contents

- Required Accessories on page 2-1
- Optional Accessories on page 2-1

Required Accessories

The following accessories are mandatory. 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:

- CETAC[™] ASX-560 Autosampler¹
- CETAC ASX-280 Autosampler¹
- CETAC XLR-8 Autosampler¹
- CETAC ASX 1400 Autosampler (stirring)¹
- CETAC U-5000AT+ Ultrasonic Nebulizer
- IsoMist[™] XR Programmable Temperature Spray Chamber²
- Hydride Generation Accessory

¹ requires dedicated RS232 or USB port on Data Station.

² requires dedicated USB port on Data Station.

System Accessories

Optional Accessories

- Argon Humidifier
- High Solids Sample Introduction kit
- HF Acid Sample Introduction kit
- Organics Sample Introduction kit
- Volatile Organics Sample Introduction kit
- Sheath Gas Adaptor
- Radial POP Interface

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.

Chapter 3 Functional Description

Contents

- Introduction on page 3-2
- Instrument Specification on page 3-4
- Sample Introduction System on page 3-11
- Internal Standards Kit on page 3-13
- Organic Solvent Analysis on page 3-14
- High Dissolved Solid Samples on page 3-16
- HF Samples on page 3-18

Introduction

The Thermo Scientific iCAP 7000 Plus Series ICP-OES is 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.



Figure 3-1. iCAP 7000 Plus 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 7000 Plus 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. Table 3-1 lists the available configurations for iCAP 7000 Plus Series ICP-OES.

Table 3-1. System configurations

Model	Description
iCAP 7200 ICP-OES Radial	Mass flow controlled nebulizer gas and radial plasma view optics
iCAP 7200 ICP-OES Duo	Pressure controlled gas box, axial and radial plasma view optics
iCAP 7400 ICP-OES Radial	Full mass flow controlled gas box and radial plasma view optics
iCAP 7400 ICP-OES Duo	Full mass flow controlled gas box and axial and radial plasma view optics
iCAP 7600 ICP-OES Radial	Full mass flow controlled gas box, radial plasma view optics, advanced sample introduction, and enhanced software
iCAP 7600 ICP-OES Duo	Full mass flow controlled gas box, axial and radial plasma view optics, advanced sample introduction, and enhanced software

The following sections describe the specification details.

Instrument Specification

Table 3-2 gives an overview of the characteristic properties of the various instruments of the iCAP 7000 Plus Series ICP-OES. The following sections provide detailed information.

Table 3-2. Instrument specification

Parameter	iCAP 7200 ICP-OES	ICAP 7400 ICP-OES	ICAP 7600 ICP-OES
Dimensions (d×w×h)		750×840×590 mm	
Weight		85 kg	
Peristaltic pump	3 channel, standard peristaltic pump Duo: pump speed: 0 or 45 rpm Radial: pump speed: 0–125 rpm	4 channel, standard peristaltic pump pump speed: 0–125 rpm	4 channel, mini peristaltic pump pump speed: 0–125 rpm
Integrated sampling valve	—	_	Standard: Sprint Valve aqueous (4 mL loop) Optional: Sprint Valve organic (3 mL loop)
ESI prepFAST Auto-dilution system	_	Optional	_
Teledyne CETAC SDX Auto-dilution system	_	Optional	Optional
Standard sample		Concentric glass nebulizer	
introduction kit		Glass cyclonic spray chamber Semi-demountable EMT torch (ceramic D-Torch on 7400/7600 ICP-OES Radial) Duo: 2.0 mm bore quartz center tube Radial: 1.5 mm bore quartz center tube	
Plasma gas	Fixed, 12 L/min		
Auxiliary gas	Fixed, 12 L/min Mass flow control, 0–20 L/min Fixed, with flows of 0, 0.5, 1.0, and 1.5 L/min Mass flow control, 0–2 L/min		
Nebulizer gas	Duo: Pressure control from 0 to 0.4 MPa Mass flow control, 0–1.5 L/min Radial: Mass flow control, 0–1.5 L/min		crol, 0–1.5 L/min
Additional gas	——————————————————————————————————————	Optional	Mass flow control, 0–100 mL/min
Plasma Viewing		Duo or Radial	iviass now control, 0=100 mE/mm
RF source		27.12 MHz solid state	
Power output	Duo: 1150 W or 1300 W		0–1350 W
rower output	Radial: 750–1500 W	Radial: 750–1500 W in steps of 25 W	Radial: 750–1600 W in steps of 25 W
Spectrometer	Simultaneous echelle type 52.91 groves/mm ruled grating 383 mm effective focal length 9.5° UV fused silica cross dispersion prism		
Spectral bandpass		7 pm at 200 nm	
Wavelength range		166–847 nm	
Detector	High performance solid state CID86 chip		
	Normal mode (precision)	Normal mode Speed mode Fullframe imaging	Normal mode Speed mode Sprint mode Fullframe imaging
Productivity features	_	Intelligent Uptake and Rinse Speed acquisition mode	Sprint Valve Intelligent Uptake and Rinse Speed acquisition mode Sprint acquisition mode

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.

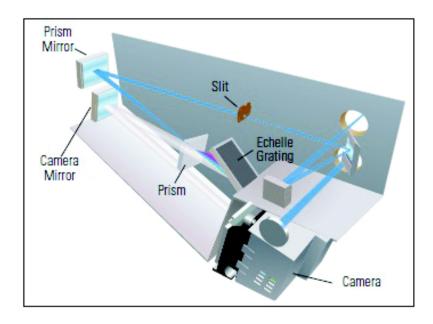


Figure 3-2. Light path

Optical System Specifications

Type

High energy echelle cross dispersion optical system with "side by side" optical arrangement of prism and grating. Unique, all spherical mirror design for very high image quality, improved optical resolution and very low stray light performance.

Focal Length

Effective focal length 383 mm

Imaging

1:1

Spectrometer Optical Path

The entire spectrometer and fore optics are purged with either argon or nitrogen. A normal running flow of 3 L/min is used with a high flow of 5 L/min for optimum performance at wavelengths below 200 nm. A purged environment is maintained with a standby flow of 1 L/min when the plasma is extinguished.

Grating

52.91 grooves/mm, 63.5° blaze angle manufactured using exclusive high resolution interferometric ruling technology.

Cross Dispersion

9.5° prism. Ultra pure ultraviolet fused silica.

Spectral Bandpass

7 pm at 200 nm, 3.5 pm per pixel.

Wavelength Coverage

iCAP 7000 Plus Series ICP-OES have a wavelength range from 166 nm to 847 nm.

Detector Specifications

Type

RACID86 Charge Injection Device (CID). High performance solid state CID camera system. The CID is an enhanced charge transfer device delivering high contrast/low noise imaging and quantification of all wavelengths in the analytical range without blooming.

Pixel Size

 $27 \times 27 \mu m$

Detector Cooling

High efficiency triple stage thermoelectric cooling device maintains the detector at a constant –45 °C. Interlocks are provided for purge gas and cooling water to prevent possible damage should a failure of the services occur.

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 optimum signal to noise is achieved for any wavelength anywhere on the detector while maintaining wide dynamic range for all signals.

Plasma Viewing Specifications

Dedicated Radial Plasma

The plasma is viewed directly in a radial mode using high efficiency magnesium fluoride coated 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 viewing height can either be selected by the operator or automatically optimized using a software routine.

Duo View Plasma

The plasma may be viewed axially for applications requiring the lowest 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 all axial, all radial, or on a wavelength by wavelength basis selected by the operator.

Plasma Source Specification

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 PC-controlled. Directly coupled autotune with swing frequency impedance control. Power regulation better than 0.1%.

Power Output

Table 3-3. Power output

Instrument	Comment
iCAP 7200 ICP-OES Duo	Power is pre-optimized at 1150 W or 1300 W
iCAP 7200 ICP-OES Radial	750–1500 W computer controlled in steps of 25 W
iCAP 7400 ICP-OES Duo	The instrument is restricted to a maximum of 1350 W
iCAP 7400 ICP-OES Radial	750–1500 W computer controlled in steps of 25 W
iCAP 7600 ICP-OES Duo	The instrument is restricted to a maximum of 1350 W
iCAP 7600 ICP-OES Radial	750–1600 W computer controlled in steps of 25 W

Sample Introduction Specifications

Nebulizer

Glass concentric fitted as standard

Optional: Aerosalt (high solids concentric), V-groove, Mira Mist (HF resistant), Ultrasonic nebulizer

Spray Chamber

Glass cyclonic type with separate radial or axial connection adapter fitted as standard

Optional: IsoMist[™] XR temperature controlled spray chamber, baffled cyclonic spray chamber, HF resistant cyclonic spray chamber

Sample Pump

Table 3-4.Sample pump

Instrument	Comment
iCAP 7200 ICP-OES Duo	High precision 12 roller,3 channel standard pump.Fixed pump speed of 45 rpm
iCAP 7200 ICP-OES Radial	 High precision 12 roller, 3 channel standard pump. Pump speed adjustable from 0–125 rpm.
iCAP 7400 ICP-OES	 High precision 4 channel standard pump with speed adjustable from 0–125 rpm. Pump tubing is available for both aqueous samples and samples containing organic solvents.
iCAP 7600 ICP-OES	 High precision 4 channel mini pump with speed adjustable from 0–125 rpm. Pump tubing is available for both aqueous samples and samples containing organic solvents.

Integrated Sprint Valve

The iCAP 7600 ICP-OES incorporates an integrated sample loop, delivering the sample to the plasma in the most efficient method to drive increased productivity.

ESI PrepFAST II

Optional on iCAP 7400 ICP-OES for intelligent dilution only in combination with a SC-DX ESI Autosampler.

Teledyne CETAC SDX

Optional on iCAP 7400 ICP-OES and iCAP 7600 ICP-OES in combination with an ASX-560 Autosampler.

Torch

Semi-demountable quartz torch fitted with either a 1.5 mm center tube (Radial instruments) or 2 mm center tube (Duo instruments).

On Radial instruments the fully demountable ceramic D-Torch is delivered as standard.

Functional Description

Instrument Specification

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

Table 3-5. Gas control

Instrument	Comment
iCAP 7200 ICP-OES Duo	• The nebulizer gas flow is manually controlled from 0 to 0.4 MPa.
	• The auxiliary gas is controlled with precision restrictors with flows of 0, 0.5, 1.0, and 1.5 L/min.
	• The coolant flow is fixed at 12 L/min.
iCAP 7200 ICP-OES Radial	• The nebulizer gas flow is controlled with a mass flow controller (MFC) from 0–1.5 L/min.
	 The auxiliary gas is controlled with precision restrictors with flows of 0, 0.5, 1.0, and 1.5 L/min.
	• The coolant flow is fixed at 12 L/min.
iCAP 7400 ICP-OES	• An MFC with flows of 0–1.5 L/min in 0.1 L/min increments controls the nebulizer gas.
	• The auxiliary gas is computer controlled through an MFC with options from 0–2 L/min in 0.1 L/min increments.
	• The coolant flow is computer controlled through a MFC from 0–20 L/min in 1 L/min increments.
	Air as additional gas is optional.
iCAP 7600 ICP-OES	• An MFC with flows of 0–1.5 L/min in 0.1 L/min increments controls the nebulizer gas.
	• The auxiliary gas is computer controlled through a MFC with options from 0–2 L/min in 0.1 L/min increments.
	• The coolant flow is computer controlled through a MFC from 0–20 L/min in 1 L/min increments.
	Air as additional gas is standard.

Operating Noise Level

The maximum normal operating noise level of the iCAP 7000 Plus Series ICP-OES has been recorded at 62 dB(A) at one meter. Therefore, wearing ear protection is not necessary.

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. Ensure that you are using 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 alter the characteristics of the sample reaching the plasma is to use a different center tube in the torch. Table 3-6 and Figure 3-3 show the available options.

Table 3-6. 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)

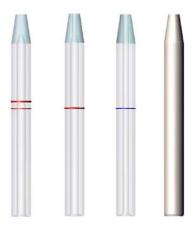


Figure 3-3. Torch center tube options

Nebulizer Options

Various nebulizer options are available for the iCAP 7000 Plus Series ICP-OES. The use of each type is application- and method-specific. This is discussed below.

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 7000 Plus Series ICP-OES.

Pump Tubing Options

The iCAP 7000 Plus Series ICP-OES requires pump tubing with either two or three clamping points (bridges). The iCAP 7200 ICP-OES and the iCAP 7400 ICP-OES pump tubing has two bridges whereas the iCAP 7600 ICP-OES pump tubing has three bridges. See Table 7-2 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 7000 Plus 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 sample loading in the plasma and may therefore contribute to improved plasma processing efficiency and resultant analytical performance.



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 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.



Figure 3-4. Internal Standards mixing kit

The use of internal standards is not required for all types of analysis, but it is typically employed where fluctuations in sample loading of the plasma may 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 using the Internal Standards Kit.

To minimize sample dilution, the tubing for the internal standard solution has a smaller bore than that of the sample solution. To ensure thorough mixing of the internal standard with the sample, a mixing loop

is provided after the Y-piece before connecting to the nebulizer. Pump tubing for the Internal Standards Kit should be installed in a similar manner to the sample tube as described at "Installation" on page 5-1.

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 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 3-5.



Figure 3-5. 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. See Figure 3-6. The orientation of the nebulizer is essential to enable successful sample aspiration.



Figure 3-6. 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 at "Pump Tubing Options" on page 3-12.

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.

Figure 3-7 shows the IsoMist[™] XR programmable temperature control device from Glass Expansion, which can be used effectively with iCAP 7000 Plus Series ICP-OES for applications with volatile organic solvents.



Figure 3-7. 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 may cause the standard nebulizer to block.

NOTICE

Cleaning a nebulizer is very difficult and should be done with great care. Cleaning should only involve the soaking of the nebulizer in an appropriate cleaning solution. Do not clean a nebulizer by inserting any items into the capillary or by using an ultrasonic bath.

To prevent the nebulizer from blocking, a variety of sample introduction accessories are available. These include the following:

- Argon Humidifier
- Aerosalt Nebulizer
- Parallel Path Nebulizer
- Sheath Gas Adaptor

Argon Humidifier Accessory

The ESI *pergo* Argon Humidifier, see Figure 3-8, 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.



Figure 3-8. *pergo* argon nebulizer gas humidifier and *pergo* system diagram (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 (e.g. V-groove nebulizer or Mira Mist nebulizer). A high solids center tube and an argon humidifier should also be used.

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, preventing contact with the injector tube, reducing the deposition of the sample on the injector. The sheath gas is introduced between the spray chamber and the torch (see Figure 3-9) using the sheath gas adaptor which must be supplied with a constant gas flow of argon via a mass flow controller.



Figure 3-9. Sheath gas introduced between spray chamber and torch

The additional gas mass flow controller can be used for the addition of the sheath gas (standard on the iCAP 7600 ICP-OES and optional on the iCAP 7400 ICP-OES).

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 supplied with an iCAP 7000 Plus 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.

The components that replace the standard equipment are the following ones (see Figure 3-10):

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

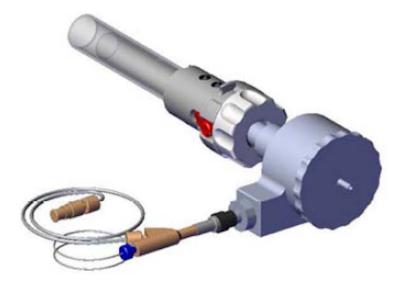


Figure 3-10. HF sample introduction configuration

Chapter 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.

Contents

- Safety Symbols and Signal Words in this Manual on page 4-2
- Safety Symbols on the Instrument on page 4-2
- Intended Use on page 4-4
- Electric Safety Precautions on page 4-6
- In Case of Emergency on page 4-7
- Residual Hazards on page 4-8

Safety Symbols and Signal Words in this Manual

Notices concerning the safety of the personnel operating the iCAP 7000 Plus 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.



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



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



Points out a hazardous situation that leads to severe injury or death if it is not avoided.

Observing this Manual

Ensure permanent access to this document for quick reference. System configurations and specifications in this manual supersede all previous information received by the purchaser.



Before you operate your iCAP 7000 Plus Series ICP-OES, read and understand all the safety information in this manual.

Safety Symbols on the Instrument

Table 4-1 lists and explains the warning labels in the torch compartment. They become visible after opening the door at the front side of the instrument.

Table 4-1. Warning labels

<u>^</u>

Symbol

Statement

This label reminds you to read the manual before using the instrument.



This label indicates the presence of hot parts in the instrument. Let all parts cool before you remove them from the torch compartment.

Table 4-1. Warning labels, continued

Symbol

Statement



This label indicates the presence of electric current in the instrument. Do not open the instrument or remove covers. No user serviceable parts are inside.



This label indicates the presence of radio frequency radiation in the instrument. Radio frequency radiation is generated to produce plasma. This radiation is present within the torch compartment. Do not try to override or defeat the interlock system.



This label indicates the presence of UV radiation in the instrument. This radiation is released when the spray chamber is disassembled. UV radiation might lead to severe eye injury or blindness. Protect eyes and skin from exposure to UV light. Do not disassemble the spray chamber when the plasma is still on.

Rating Plate

To correctly identify the instrument when you contact Thermo Fisher Scientific, always have the information from the rating plate available. The rating plate is attached to the top of the torch compartment. It becomes visible after you open the door at the front side of the instrument. See Figure 4-1 as an example. It contains the serial number, which is important in any type of communication with Thermo Fisher Scientific.



ICP-OES SPECTROMETER Serial No: IC72RA0000

MODEL: iCAP 7200 Radial

YEAR OF MANUFACTURE 2017

SUPPLY VOLTAGE 200-240 V~ FREQUENCY 50/60 Hz

POWER MAX 2880 VA SERVICE BY QUALIFIED PERSONNEL ONLY

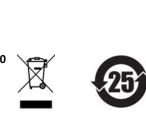


Figure 4-1. Rating plate (example)

Intended Use

The iCAP 7000 Plus Series ICP-OES is a range of inductively coupled argon 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.



Observe the following usage guidelines when you operate your iCAP 7000 Plus Series ICP-OES:

- 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 7000 Plus Series ICP-OES 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. Do not use radio frequency transmitters, such as mobile phones, in close proximity to the instrument.

Intended User

The primary audience for this manual consists of analytical chemists and lab 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 7000 Plus Series ICP-OES system. Personnel that install or operate iCAP 7000 Plus Series ICP-OES 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 7000 Plus Series ICP-OES Pre-Installation Requirements Guide* for the specifications.

General Operation

The iCAP 7000 Plus Series ICP-OES is designed to be operated by qualified laboratory personnel. Before starting, all users must be instructed about the hazards presented by the instrument and the chemicals applied. The users must be advised to read the relevant Material Safety Data Sheets (MSDSs).

Permitted Materials

The iCAP 7000 Plus Series ICP-OES system is designed to be operated with the materials listed in Table 4-2.

Table 4-2. Materials and their use

Material	Used for
Gases	
Ar	Used to maintain the plasma of the iCAP 7000 Plus Series ICP-OES. Optionally used for purging the optical path
$\overline{N_2}$	Optionally used for purging the optical path
Compressed air	Optionally used as additional gas
Sample matrices	
Water based solutions with mixed acid content	Preparation of calibration solutions, samples
Organic solvents like ethanol, isopropanol	Applications that require the analysis of organic solvents

Electric Safety Precautions



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 it has incurred any kind of electrical damage. Instead, disconnect the power cord of the spectrometer 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

To shut down the system in case of emergency

ACAUTION

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 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 at "Shutting Down the System" on page 6-32. Do not use the door to shut down the system.

1. Turn the power switch at the rear left side of the spectrometer to the Off position. See Figure 4-2.

All power to the spectrometer is shut off.



Figure 4-2. iCAP 7000 Plus Series ICP-OES power switch

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



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 7000 Plus Series ICP-OES must pay attention to the following residual hazards.

∴ WARNING

Toxic Gases. Risk of intoxication. 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 spectrometer without an effective fume extraction system attached to the torch compartment 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. If the extraction is turned off, sufficient air must be supplied to prevent the concentration of these gases reaching a harmful level. Ensure that the laboratory is well ventilated.

ACAUTION

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. Risk of burns. Let any hot components cool for at least 10 minutes before you remove them from the torch compartment.

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 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 at "Shutting Down the System" on page 6-32. Do not use the door to shut down the system.

ACAUTION

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

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.

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, while 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

Residual Hazards

Chapter 5 Installation

This chapter describes the conditions for an operating environment that ensures continued high performance of your iCAP 7000 Plus Series ICP-OES system.

To be sure that your laboratory is ready for installation of the iCAP 7000 Plus Series ICP-OES, you have to meet all requirements specified in the *iCAP 7000 Plus Series ICP-OES Pre-Installation Requirements Guide*. This guide also provides comprehensive information to assist in planning and preparing your lab site.



To comply with safety and warranty requirements, the iCAP 7000 Plus Series ICP-OES, accessories and associated equipment must be installed by a certified Thermo Fisher Scientific service representative.

Contents

- Safety Guidelines for Installation on page 5-2
- Spectrometer Dimensions on page 5-3
- Installation Requirements on page 5-4
- Communications Interface on page 5-12
- Installation Checklist on page 5-13

Safety Guidelines for Installation

When installing the iCAP 7000 Plus Series ICP-OES system, pay attention to the following general safety guidelines.

⚠ WARNING

Toxic Gases. Risk of intoxication. 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 spectrometer without an effective fume extraction system attached to the torch compartment 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. If the extraction is turned off, sufficient air must be supplied to prevent the concentration of these gases reaching a harmful level. Ensure that the laboratory is well ventilated.

ACAUTION

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.

ACAUTION

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 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 at "Shutting Down the System" on page 6-32. Do not use the door to shut down the system.

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. Risk of burns. Let any hot components cool for at least 10 minutes before you remove them from the torch compartment.

Spectrometer Dimensions

Table 5-1. Dimensions and weight of iCAP 7000 Plus Series ICP-OES

Width	Depth	Height	Weight
840 mm	750 mm	590 mm	85 kg
(33 ¹ / ₁₆ inch)	(29½ inch)	(23¼ inch)	(187 lb)

Figure 5-1 shows a schematic of the iCAP 7000 Plus Series ICP-OES in front view, whereas Figure 5-2 shows it in top view.

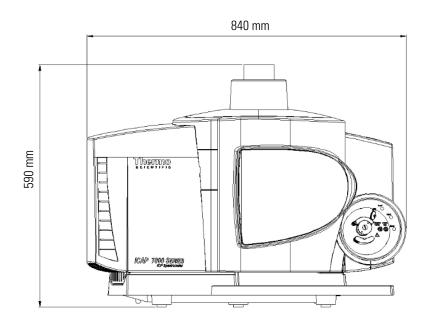


Figure 5-1. iCAP 7000 Plus Series ICP-OES in front view

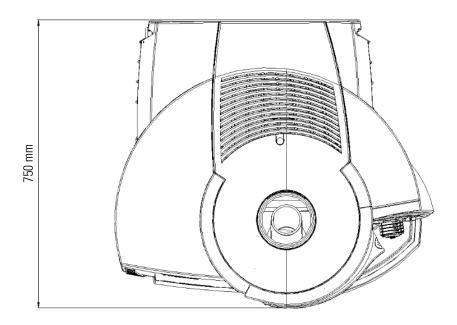


Figure 5-2. iCAP 7000 Plus Series ICP-OES in top view

Installation Requirements

This section describes the environment and the resources required at the installation site of the iCAP 7000 Plus Series ICP-OES and associated equipment. Safety requirements for the installation are detailed as well.



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.

The choice of an operating site for the instrument is influenced by local considerations such as ease of access and availability of electrical power.

Before Installation

Before installation, make sure that the proposed area is compatible with the conditions specified. The laboratory must offer a dry even temperature and dust-free conditions, with no possibility of generating condensation. Locate sample preparation activities and corrosive materials in a separate room to avoid problems due to corrosive fumes.

Carry out a comprehensive risk assessment that is specific to the handling of solvents, samples and sample preparation.

Take particular consideration to avoid direct sunlight, proximity to heat sources, air drafts and vibration. Do not locate the system where sudden changes in temperature can occur, for example near a door or window. Take care with the location of items such as air conditioning vents and heating vents.

Location Requirements

The choice of site is influenced by the dimensions and weights of the spectrometer and its accessories. Other factors are the environment and the availability of electricity, water and gas supplies, as well as the need for a suitable ventilation system to dispose of the exhaust gases. All of these factors are covered in the following sections.

The instruments are designed for use on a normal laboratory bench. Ideally, the instrument is placed on a movable bench with 0.5 m of access behind the instrument. The mounting surface must be level and the instruments must not be placed on any type of cushioning as this could block ventilation. A bench length of approximately 3 m is required for spectrometer, PC and autosampler. A chiller is usually located under the same bench.

The mounting arrangements must be capable of supporting the weight of the spectrometer and its accessories. Make sure that the working surface is sufficiently rigid to prevent vibration as this might affect the optical alignment of the spectrometer and the accessories.

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, even for a short time, near the instrument.

Do not position the equipment in such a way that it is difficult to operate the extraction, electrical supply, cooling water, purge gas and plasma gas controls.

Environmental Requirements

The atmospheric temperature requirement is 15 to 35 °C (60 to 95 °F). The temperature should not change by more than 2 °C per hour.

Atmospheric humidity should be 20 to 80% relative humidity for an ambient temperature between 15 and 30 °C and 20 to 60% relative humidity for an ambient temperature between 30 and 35 °C. Atmospheric conditions must be non-condensing. The instrument room should be at a positive pressure with respect to rooms with a corrosive atmosphere.

The recommended atmospheric pressure is between 0.7 bar to 1.06 bar—approximately 400 m (1300 ft) below sea level and 3000 m (10000 ft) above sea level.



Suffocation Hazard. If the extraction is turned off, sufficient air must be supplied to prevent the concentration of argon reaching a harmful level.

To ensure optimum analytical performance, reliability and longevity of your iCAP 7000 Plus Series ICP-OES, observe a number of basic laboratory considerations that are listed in Table 5-2.

Table 5-2. 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 7000 Plus 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.

Table 5-2. Laboratory considerations, continued

Consideration	Why is this aspect important?
Appropriate placement of sample introduction accessories when working with corrosive or volatile liquids	Liquid autosamplers should be installed to ensure a sufficient air clearance around the base of the iCAP 7000 Plus 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	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.
Handling of flammable liquids	If handling 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.

Electrical Requirements

The spectrometer requires an electrical supply at 230 $\pm 10\%$ V AC, 4 kVA, 50/60 Hz. The spectrometer is supplied with a 2.5 m mains cord.

For customers in the USA and Canada, the mains cord supplied terminates in a NEMA L6-20P Twist&Lock plug. Coding is black (X - AC high), white (Y - AC low), green (G - earth/ground). See Figure 5-3.

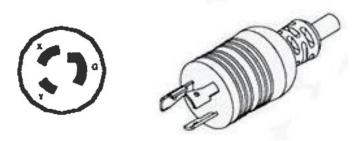


Figure 5-3. NEMA L6-20P plug for USA and Canada

For customers in the rest of the world, the mains lead is shipped with no terminating plug, but with wires ready for a suitable 20-32 A plug. Coding is blue (neutral), brown (live), green/yellow (earth/ground).

Connection to the laboratory supply must be with an appropriately rated plug conforming to local electrical guidelines and local requirements. It must be possible to electrically isolate the instrumentation.

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

Each 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.

Power Quality

The power supplied to iCAP 7000 Plus Series ICP-OES must be stable. The input (line) voltage must be 230 V AC. The input frequency must be between 47 and 63 Hz. 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.

Gas Requirements



Suffocation Hazard. Nitrogen and argon gas might cause suffocation if they accumulate in the laboratory. Make sure that the laboratory is well ventilated.

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

Argon

The spectrometer requires argon at 5.5 bar (80 psi). Argon must have a minimum purity of 99.995%, with less than 10 ppm water and less than 10 ppm oxygen, used for operation and optical path purge. The maximum flow requirement could be up to 25 L/min during installation.

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.



Do not exceed a pressure of 6 bar (87 psi).

Nitrogen

For purging the optical path in the spectrometer, the same argon supply can be used. Alternatively, nitrogen at 5.5 bar (80 psi) with a minimum purity of 99.995% with less than 10 ppm water and less than 10 ppm oxygen can be used instead of argon. The maximum flow requirement is 15 L/min during installation. For optimum performance, Thermo Fisher Scientific recommends a purity of 99.998% nitrogen.

Compressed Air

For the additional gas MFC fitted to the iCAP 7600 ICP-OES, clean dry oil-free air is required at a pressure of 2 bar (30 psi) and a flow of approximately 100 mL/min. Thermo Fisher Scientific recommends a quality of 99.99% with less than 100 ppm water.



Make sure that the compressed air is dry and oil-free. Otherwise, damage to the MFC results.

Gas Supply Lines

The instrument is supplied with three 1.6 m lengths of plastic tubing (6 mm OD Polyolefin for the purge and the plasma gas, and 4 mm for the additional gas).

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.



Make sure that the laboratory gas lines do not contaminate the gases used for the iCAP 7000 Plus Series ICP-OES.

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.

Gas Consumption

The gas consumption of an iCAP 7000 Plus Series ICP-OES varies with plasma conditions and purge operating parameters. Table 5-3 shows the approximate gas consumption for plasma gas and purge gas at different conditions.

Table 5-3. Typical gas consumption of iCAP 7000 Plus Series ICP-OES

Instrument	Plasma gas	Purge gas option		on
	(cool + aux + neb) min - max	Trickle 1.2 L/min	Normal 3.2 L/min	High 5.2 L/min
iCAP 7200 ICP-OES	13–16 L/min	x	-	-
iCAP 7400 ICP-OES	10-23.5 L/min	X	X	X
iCAP 7600 ICP-OES	10-23.5 L/min	X	x	X

Recirculating 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 5 L/min with a back pressure of about 6 bar (90 psi) and a minimum cooling capacity of 750 W at a room temperature of 25 °C and at your altitude. The chiller must provide 50 μ m particulate filtration. A suitable chiller (Thermo Scientific ThermoFlex 900) can be supplied from your local Thermo Fisher Scientific organization. For instruments used at an altitude above 1500 m (5000 ft), the ThermoFlex 1400 is recommended.

The water temperature should be set to 5 °C below ambient temperature, and must be set between 15 °C and 30 °C. Temperature variation must be less than 0.2 °C per hour.

The water used in the chiller must be distilled or deionized water containing 2.5% by volume of the correct inhibitor. The correct inhibitor is included with a new instrument and a maintenance kit, which can be ordered from your local Thermo Fisher Scientific organization.

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

The spectrometer is supplied with plastic connecting tubing of 12 mm OD. Connection to the water chiller should be within 3 m of the instrument, or insulated tubing must be used.

Waste Storage

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.

An appropriate container is required 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.



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

Ensure that pump, sample, waste tubing and vessels are labeled with appropriate safety symbols to protect anyone in the proximity of, using or maintaining the equipment.

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 operators.

Spectrometer Fume Extraction

This instrumentation is designed for operation under 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 will be void if the equipment is operated under sub-standard conditions.



Correct extraction is critical to instrument performance and safety. Do not operate the spectrometer without an effective fume extraction system attached to the torch compartment chimney.

Hot fumes, which might be corrosive and toxic, are discharged from the instrument chimney during operation. To ensure a safe working environment and safe removal of waste combustion products, an effective extraction system must be installed. It should include appropriate filtering of hazardous toxic fumes.

The extraction must be set as shown in Figure 5-4, measured in the center of the extraction tube at the spectrometer end, while disconnected from the instrument. The extraction tube has an inlet diameter of 125 mm (5 inch). A 2 m (6.5 ft) flexible tube is supplied with the instrument.

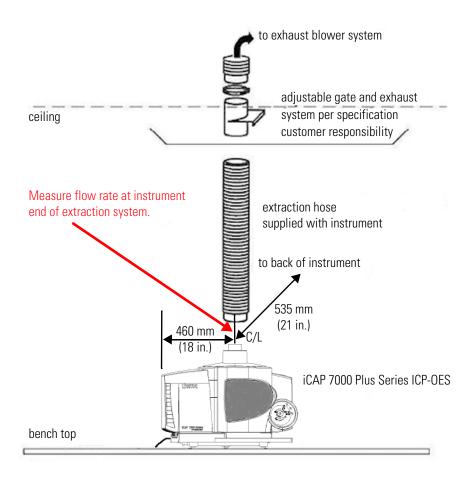


Figure 5-4. iCAP 7000 Plus Series ICP-OES extraction system

Tip Extraction requirements are not the same as for previous Thermo Scientific ICP spectrometers. An iCAP 7000 Plus Series ICP-OES Duo requires an extraction of 10 m/s (33 ft/s; 22.4 mph). An iCAP 7000 Plus Series ICP-OES Radial requires an extraction of 5 m/s (16 ft/s; 11.2 mph).

The extraction flow must be adjustable to enable the Thermo Fisher Scientific field service engineer to correctly set the flow and achieve the required extraction. This can be achieved with a butterfly valve or an adjustable gate as shown in Figure 5-4. A flow of about 14 m/s before the supplied flexible tube should be sufficient.

The extraction system must not be affected by external weather conditions or other uses the system may be used for. The extraction system must not let the fumes or the fumes condensation return toward the instrument.

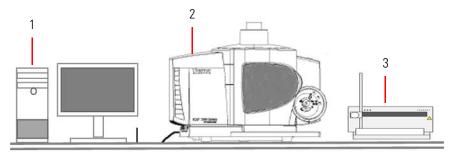
Extraction fan specification varies depending on laboratory layout, as well as on lengths, diameters and type of tubing required to reach the instrument. Consult a qualified extraction specialist to ensure that the correct flow is achieved at the instrument.

Sample Fume Extraction

Additional, separate extraction should be considered 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.

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 5-5.



Labeled Components: 1=Data Station computer, 2=instrument, 3=autosampler

Figure 5-5. iCAP 7000 Plus 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. The instrument IP address is 90.0.0.50.

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

Data Station Requirements

The Data Station requirements are normally met by a PC that meets the minimum specifications listed in Table 5-4. A suitable computer is available from your local Thermo Fisher Scientific organization.

Table 5-4. Data Station requirements

System	Requirements
Hardware	 Dual core 2.4 GHz with 4 GB RAM 500 GB of free hard disk space DVD ROM drive for software installation Dedicated Ethernet port for spectrometer communication 2 × USB 2.0 Monitor 21", 16:9, display resolution 1920×1080
	 Some accessories require a dedicated RS232 port. A second Ethernet port for local networking, if required Internet connection (recommended) A printer port may be required.
Software	 Microsoft™ Windows™ 7 Professional 32 bit (Service Pack 1) Microsoft Office (recommended)



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

Installation Checklist

Table 5-5. Overview of site requirements

Property	Value
Location	
Table dimensions for the spectrometer	900 × 600 mm
Table dimensions for the autosampler (right side of the spectrometer)	600 × 550 mm
Table dimensions for keyboard and mouse (left side of the spectrometer)	600 × 30 mm
Electrical supply	
Plug 200–240 V AC, 20–32 A, single phase 4000 W, 50/60 Hz	required
Standard mains plug 200–240 V AC for each accessory	required

 Table 5-5.
 Overview of site requirements, continued

Property	Value
Argon gas	
Regulator valve in the wall 0–10 bar (0–150 psi)	required
Gas union dimension at the exit of the regulator valve	push fitting for plastic 6 mm OD
Maximum distance between instrument and regulator valve	1 m
Purity of argon (gas or liquid)	99.995% < 10 ppm O ₂ ; < 10 ppm H ₂ O
Pressure required at the input of the spectrometer	5.5 bar (80 psi) Do not exceed 6 bar.
Optic nitrogen purge (optional)	
Regulator valve in wall 0–10 bar (0–150 psi)	required
Gas union dimension at the exit of the regulator valve	push fitting for plastic 6 mm OD
Maximum distance between the instrument and the regulator valve	1 m
Purity of nitrogen (gas or liquid)	> 99.995% < 10 ppm O ₂ ; < 10 ppm H ₂ O
Pressure required at the input of the spectrometer	3.5 bar (50 psi)
Additional gas - compressed air (iCAP 7600 ICP-OES onl	y)
Regulator valve in the wall 0–4 bar (0–60 psi)	required
Gas union dimension at the exit of the regulator valve	push fitting for plastic 4 mm OD
Maximum distance between the instrument and the regulator valve	1 m
Purity of bottled air or dried compressed air	99.99% < 100 ppm H ₂ O
Pressure required at the input of the spectrometer	2 bar (30 psi)
Autosampler rinse and waste	
Minimum 1 L container for the rinse supply station and waste	2 needed
Chiller	
Maximum distance from the spectrometer	2 m
Pipe supply outer diameter dimension	12 mm

 Table 5-5.
 Overview of site requirements, continued

Property	Value
Cooling capability	750 W minimum
Flow rate (all chillers)	> 5 L/min
Pressure at 5 L/min (ThermoFlex 900 chiller only)	5.5 bar (80 psi)
Corrosion inhibitor	required

Installation

Installation Checklist

Chapter 6 System Operation

Contents

- Safety Guidelines for Operation on page 6-2
- Preparing the System for Use on page 6-3
- Instrument Optimization on page 6-4
- Standard Sample Introduction Glassware Assembly on page 6-5
- Example Standard Operating Procedures on page 6-19
- Reporting Results on page 6-27
- Exporting LabBooks on page 6-28
- Auto Peak Adjust on page 6-30
- Shutting Down the System on page 6-32

Safety Guidelines for Operation

When operating the iCAP 7000 Plus Series ICP-OES system, pay attention to the following general safety guidelines.

∴ WARNING

Toxic Gases. Risk of intoxication. Toxic gases are released that might lead to severe bodily harm when the spray chamber is disassembled. Do not operate the spectrometer without an effective fume extraction system attached to the torch compartment 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. If the extraction is turned off, sufficient air must be supplied to prevent the concentration of these gases reaching a harmful level. Ensure that the laboratory is well ventilated.

ACAUTION

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

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 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 at "Shutting Down the System" on page 6-32. Do not use the door to shut down the system.

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.

ACAUTION

Hot Surface. Risk of burns. Let any hot components cool for at least 10 minutes before you remove them from the torch compartment.

Preparing the System for Use

The iCAP 7000 Plus 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 at the rear of the left side. See Figure 6-1.



Figure 6-1. iCAP 7000 Plus Series ICP-OES power switch

LED Indicators

A row of LEDs, which indicate the status of the instrument is located on the rear right hand side of the instrument.



Figure 6-2. LED indicators

When the chiller has been turned on and has reached its set temperature, the LEDs 2–7 should be on and the LEDs 1 and 8 should be flashing. LED 9 indicates engineer fast purge has been selected and should be turned off when analyzing samples.

Instrument Optimization

The iCAP 7000 Plus 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 "Assistance" on page 1-5.

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 is 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
Radial Instrument Plasma viewing height	Used to select optimum viewing height for different emission zones in the plasma
Radial/Axial View	On a Duo instrument, you have a choice of axial and radial views. You have to select the view 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

Table 6-1. Parameters affecting data, continued

Parameter	Comment
Organics check box	To enable Torch Alignment to be performed with organic standards
	See "Torch Alignment" on page 8-9.
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.

Setting the Pump Tension

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

❖ To adjust the pump 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.



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.

Standard Sample Introduction Glassware Assembly

CAUTION

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



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

Duo Torch Assembly



Figure 6-3. Duo Torch assembly



Figure 6-4. Duo Torch assembly - metal torch mount

❖ 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. See Figure 6-5.

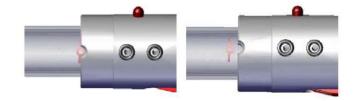


Figure 6-5. Torch assembly - torch body

3. Check that the four O-rings in the center tube holder are not damaged. Fully insert the center tube into the plastic center tube holder. See Figure 6-6.



Figure 6-6. Torch assembly - center tube

- **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.
- 4. Insert the center tube assembly into the metal torch holder. See Figure 6-7.

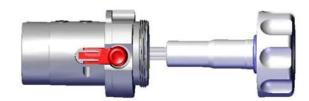


Figure 6-7. Torch assembly - center tube insertion

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

When fitted, the center tube should be 1–3 mm lower than the intermediate tube. See Figure 6-8.

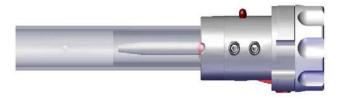


Figure 6-8. Center tube inserted into torch assembly

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. See Figure 6-9.

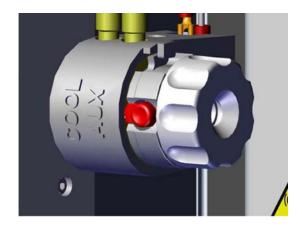


Figure 6-9. Insertion of the torch assembly into the box

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 6-10.



Figure 6-10. 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.

The collar will set the insertion depth and aid reproducibility of results.

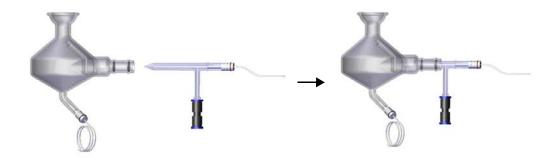


Figure 6-11. Nebulizer spray chamber assembly^a

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 UV radiation from escaping from the torch box. See Figure 6-12.

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



Figure 6-12. Spray chamber/adapter assembly

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. See Figure 6-13.

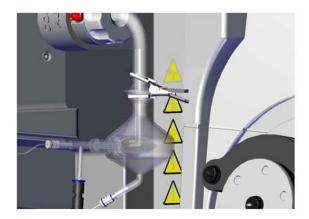


Figure 6-13. Spray chamber fitted

- 12. After assembling the sample introduction system and before igniting the plasma, check the following to assure correct assembly:
 - The torch is fully rotated and locked in 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.



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 feed the sample capillary tubing, proceed with "Connection of Pump Tubing" on page 6-14.

Radial Torch Assembly



Figure 6-14. Radial torch assembly

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. See Figure 6-15.



Figure 6-15. Radial torch assembly - metal torch mount

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.



Figure 6-16. Radial torch 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. See Figure 6-17.



Figure 6-17. Torch assembly - spray chamber adapter

4. Insert the center tube fully into the center tube holder. See Figure 6-18.



Figure 6-18. Center tube holder assembly

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.

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 reduces the lifetime of the O-ring seal.



Figure 6-19. Torch assembly - center tube to torch mount

6. Push the torch assembly up into the torch box and twist clockwise until the red orientation lock locates in the torch box casting. See Figure 6-20.

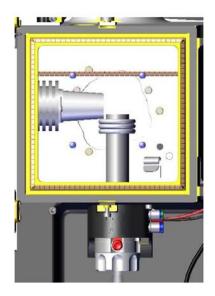


Figure 6-20. Torch installation in the torch box

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

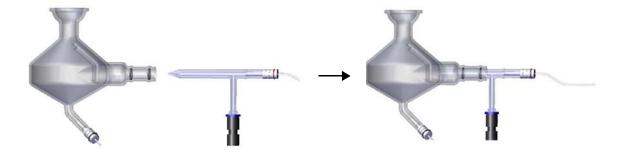


Figure 6-21. Cyclone spray chamber

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 spray chamber and nebulizer assembly to the torch assembly and lock into place in the instrument using the stainless steel clamp.

The torch compartment door must be closed before ignition of plasma. This will be confirmed by the torch box door interlock.



Figure 6-22. Torch/spray chamber assembly

After the assembly of the sample introduction system and before ignition of the plasma, check the following to ensure correct assembly:

- The torch is fully rotated and locked in place.
- The center tube holder is fully rotated and locked into the torch.
- The spray chamber connector 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.



It is extremely important that the safety interlocks are not disabled, This could result in exposure to dangerous radio and UV frequency radiation.

To feed the sample capillary tubing, proceed with "Connection of Pump Tubing" on page 6-14.

Connection of Pump Tubing

Figure 6-23 shows the peristaltic pump and 2-stop tubing that is typical of the iCAP 7000 Plus Series ICP-OES, except for the iCAP 7600 ICP-OES. Here, the sample pump tubing has white and orange stops, and the drain tubing has white and white stops.

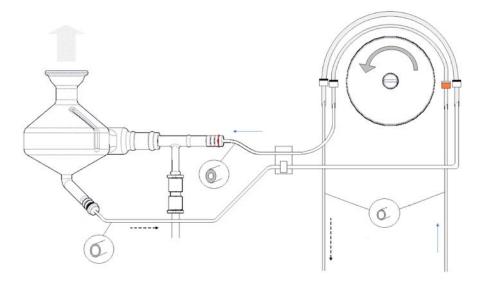


Figure 6-23. Sample capillary tubing scheme

The iCAP 7600 ICP-OES has a mini pump and 3-stop tubing (providing two sections for reduced running costs).

The sample pump tubing has white, white and white stops. The drain pump tubing has yellow, blue and yellow stops.

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 through the upper holder in the cover and towards the pump.
 - Ensure 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 lower holder in the cover and towards the pump.

The lower holder contains a drain sensor detecting bubbles produced when the spray chamber is draining normally. The plasma and the pump are switched off after 2 minutes if no bubbles are detected.

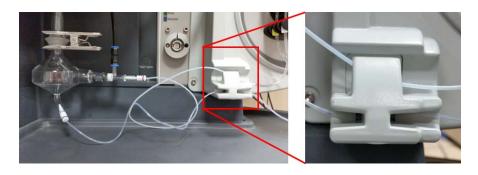


Figure 6-24. Tubing holder with drain sensor

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

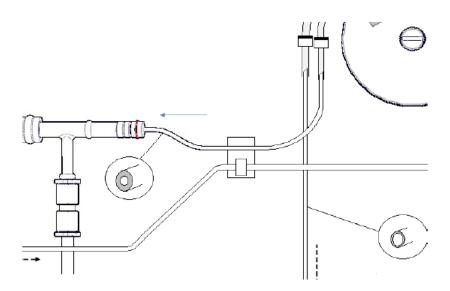


Figure 6-25. Sample capillary tubing

NOTICE

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

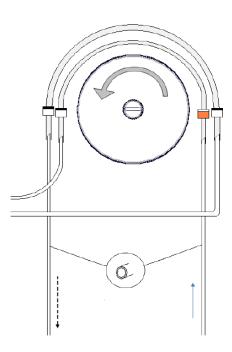


Figure 6-26. Pump tubing^a

- $^{\rm a}$ Here showing the two bridge tubing for the large peristaltic pump on the iCAP 7200 ICP-OES and the iCAP 7400 ICP-OES
- 4. Release the pump tubing clamps. Locate the sample and drain pump tubing over the pump rollers, locking the lugs on the pump tubing into the left and right clamps.
- 5. Connect the sample pump tubing to the sample capillary tubing and the drain pump tubing to the drain capillary tubing. Remember to allow for the direction of flow.

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

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

To set the pump tension, see "Setting the Pump Tension" on page 6-5.

Tip The internal dimensions of the drain pump tubing for the mini pump (iCAP 7600 ICP-OES) are different to the iCAP 7200 ICP-OES and iCAP 7400 ICP-OES with the large pump. This requires a larger external diameter connection tube from the spray chamber for the iCAP 7600 ICP-OES.

Tubings of both diameters are part of the sample introduction kits supplied with a spectrometer. The wider tube is available as P/N 8423 120 52671.

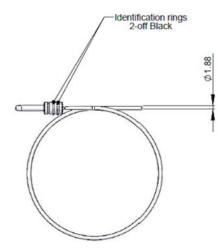


Figure 6-27. Tubing diameter

Radial View Interfaces on a Duo Instrument

The radial view on the Duo instrument allows the user to collect light from a cross-section of the plasma. This view is less sensitive, however it increases the linear range and has less matrix interferences. Two types of interfaces between plasma and fore-optics, with different properties, can be used.

Radial Glass Window

For a Duo instrument, check that the radial (bucket shaped) glass window is in place and clean. The radial view periscope can be rotated to gain access (take the torch out before inspection). When assembling the torch and the radial glass window, ensure that the window is located against the torch as shown in Figure 6-28.

If the radial glass window is removed from the periscope, ensure that sufficient time is left to stabilize the purge after replacing the window.

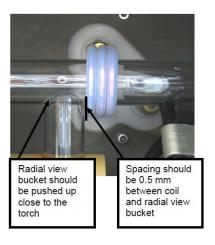


Figure 6-28. Radial glass window

Radial POP Interface

The radial POP (purged optical pathway) interface, Figure 6-30, is an accessory that allows for reduced user maintenance, improved UV detection limits and long-term stability of high matrix samples. It must be adjusted such that the radial observation hole in the torch matches the bore of the interface. For the adjustment of the radial POP interface, an alignment tool is available.

❖ To align the radial POP interface

- 1. Remove the EMT quartz torch from the torch box.
- 2. Fit the radial POP interface into the radial view periscope.
- 3. Insert the EMT quartz torch into the torch box.
- 4. Remove the center tube holder from the torch.
- 5. Introduce the alignment tool for radial POP interface, Figure 6-29, into the torch tubes.



Figure 6-29. Alignment tool for radial POP interface

6. Use the conical tip of the tool to align bore and hole.

Make sure that the radial POP interface, Figure 6-30, is as close as possible to the hole of the torch.



Figure 6-30. Radial POP interface



First time installation of the radial POP interface is done by a certified Thermo Fisher Scientific field service engineer because it may be necessary to perform a fore-optics alignment. Later maintenance of the radial POP interface can be done by the user.

Example Standard Operating Procedures

The following method setup procedure, which by no means covers all possible parameters used in Qtegra ISDS Software, is an example standard operating procedure and should be enough to set up a basic analysis. Read the *Qtegra for iCAP 7000 Series ICP-OES Software Manual* for 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 turning on the water chiller and running 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 5 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 0.6 MPa (87 psi) on the gauge near the instrument.

NOTICE

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 5-3.

- 2. Turn on the air supply for the Additional gas supply (iCAP 7400 ICP-OES and iCAP 7600 ICP-OES) if used.
- 3. Switch on power to the iCAP 7000 Plus Series ICP-OES.

NOTICE

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 turn on the water chiller until the instrument has been purging with gas for at least two hours. Doing so can seriously damage the CID detector.

5. Push the platens on to rollers of the peristaltic pump using the required number of the total four (three) pressure screws. The sample requires one channel, the drain another one. Further channels are optional (for example, for the internal standard addition).



To avoid unnecessary wear of material, tighten only the pressure screws on platens 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 icon on the computer desktop.

Lighting the Plasma

To light the plasma

Detector water flow

- 1. Ensure that the torch box door has been physically closed.
- 2. Click the Qtegra icon on the computer desktop.
- Check that the interlocks are all green and take appropriate action if any are red:

Interlo	Interlocks		
	Torch Compartment		
	Plasma Gas Pressure		
	Purge Gas Pressure		
	Detector Water Flow		
	Drain Flow		
	Exhaust Flow		
	Detector Temperature		
	Optics Temperature		

Torch Compartment Interlock	If red, this indicates the torch box door is open or the torch holder is not inserted correctly. The plasma will not light.
Plasma gas pressure	Should be green if the plasma gas input pressure is 5.5 bar.
	If it turns red during the ignition sequence, this indicates problems with the external gas supply to the spectrometer.
	If red, the plasma will not light.
D	C1 111

If red, the plasma will not light.

Purge gas pressure

Should be green if the input pressure for the plasma gas is 5.5 bar.

If it turns red during the ignition

sequence, this indicates problems with the external gas supply to the spectrometer.

If red, the plasma will not light.

Should be green if the correct water flow is flowing for the camera to cool and the RF to light the plasma.

If red, the plasma will not light.

If the LED flickers even slightly, the plasma will go out. This indicates a problem with the chiller.

Drain Flow sensor If it is red, this indicates that the

> spectrometer has not seen an air bubble in the drain sensor for two minutes, it will

turn the plasma off.

To reset the drain sensor, turn on the

pump to 45 rpm.

Exhaust flow This interlock checks that the exhaust is of

sufficient flow to ensure safe removal of

heat and combustion gases.

In a 20 second period, the extraction needs to be low for 5 seconds for the interlock to

occur.

This interlock indicates whether the Detector temperature

> camera has cooled to -45 °C and is ready to measure samples. (Notes: RED = too hot, Green = -45 °C, Blue = too cold.)

When the chiller is turned on, the camera

takes 5 minutes to cool to -45 °C.

Optics temperature This indicates that the optical tank has

reached the operating temperature.

From cold, it could take 2 hours to reach 38 °C and an additional hour to fully

stabilize.



4. When ready, click the "red ringed" **Get Ready** icon on the Dashboard page.

The Get Ready window opens with several options:

Move to rinse when Can be selected when using an autosampler, if the probe has not been used for a while. idle

Warm Up Is usually set to 15 minutes to enable the

system to stabilize before spectrometer

optimization.

Spectrometer Is usually selected to perform an automatic minor correction of the location of the Optimization

emission spectrum on the detector.

Nebulizer Is usually selected to improve performance

Optimization tests. Run Performance Checks Runs the factory-recommended performance test using defined sample introduction and

Standards.

On installation, the Torch Alignment and

Autopeak must be completed first.

Use Manual Sampling

If an autosampler is configured, select this to

enable manual sampling.

5. Click OK.

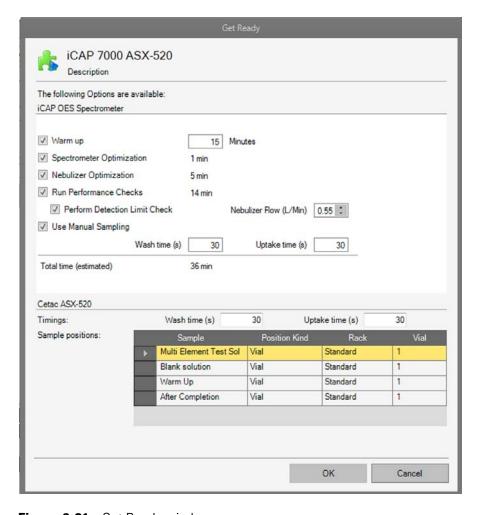


Figure 6-31. Get Ready window

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

Tip To let the plasma stabilize, leave the plasma on with a blank solution running for about 10 minutes before carrying out an analysis.

Setting Up Analyses

To set up an analysis



- 1. Click the Qtegra icon on the computer desktop.
- 2. On the Home Page, click LabBooks.

The LabBook page of Qtegra opens.

LabBooks can be created from blank templates, existing templates or from existing LabBooks.

3. To create a new LabBook, enter a name for it, select a location, and select an evaluation (for example, eQuant). See Figure 6-32.

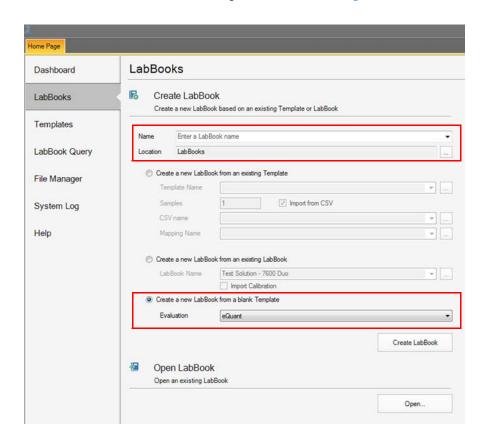


Figure 6-32. Creating a new LabBook window

- 4. Click **Create LabBook**. A new tab opens for the new LabBook.
- 5. When you create a LabBook from a blank template, you need to specify method parameters. Method parameters might differ, depending on the Evaluation selected for the Template or LabBook in Qtegra.

The availability of each parameter is controlled by the type of Evaluation defined for the Template or LabBook. Refer to the *Qtegra for iCAP 7000 Series ICP-OES Software Manual* for more information.

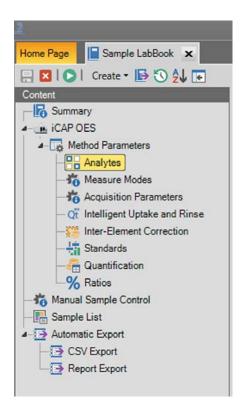


Figure 6-33. Method Parameters of the LabBook

- 6. Specify the settings for either manual sample control or an autosampler:
 - Use **Manual Sample Control** to disable the autosampler and define the Uptake time and the Wash time. See Figure 6-34.

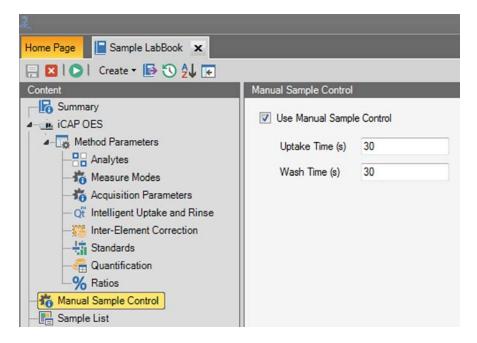


Figure 6-34. Manual Sample Control page

 Select the appropriate autosampler. Specify Wash Time and Uptake Time in the autosampler settings. Rack type selection changes may also be made here. See Figure 6-35.

Tip When using the iCAP 7600 ICP-OES Sprint Valve, the Wash Time may be set to 0 because this function is tasked by the Sprint Valve timings. Uptake Time can also be reduced to about 20 seconds because using the valve provides quicker sampling.

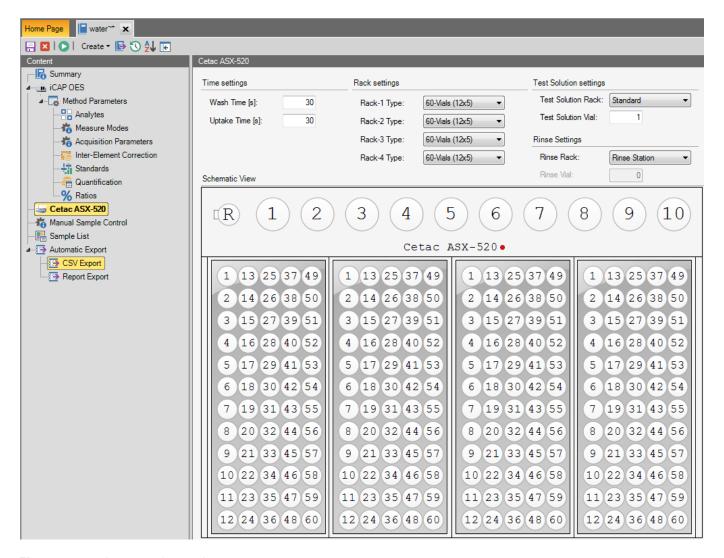


Figure 6-35. Autosampler settings

7. Use the Sample List to build a sequence and specify the samples to be analyzed. Add lines individually for BLANK and each of the calibration standards and QC checks. Use the label identifier and sample type drop down list as required.

If using an autosampler, identify the rack and vial for each sample.

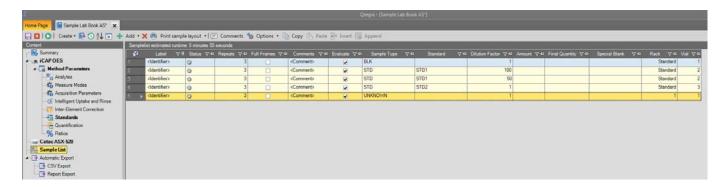


Figure 6-36. Sample List page



8. On the toolbar of your LabBook page, click this icon to save your LabBook.

Running the Analysis

To run an analysis



In the toolbar of your LabBook, click this icon to schedule the LabBook for execution. The LabBook is added to the Scheduler.

If the Automatic check box has been selected for Start Queue in the Options settings of the Scheduler, the measurement is started immediately.

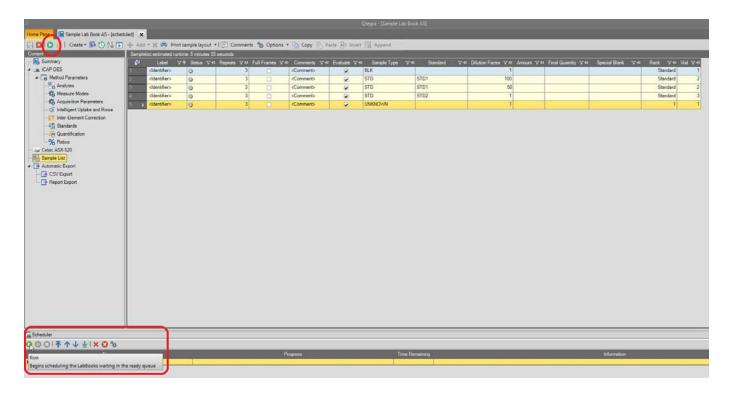


Figure 6-37. Running a LabBook

The completed LabBook is automatically deleted from the Scheduler and added to Completed LabBooks.

Reporting Results

Reporting can be found in the Reports tab of acquired LabBooks. Reports are global and not saved per LabBook. All report styles are available for any LabBooks that are opened subsequently (where they can also be modified).

To change general settings, click the **Edit Report** button and change the required parameters: Report Settings such as Image (for example, a company logo can be used) or Page (for example, paper format, Portrait or Landscape, Font size) and Content (such as Table information, page breaks and Grouping). See Figure 6-38.

Save Report applies settings to the current report style. The reported LabBook can also be printed or saved in a different file.

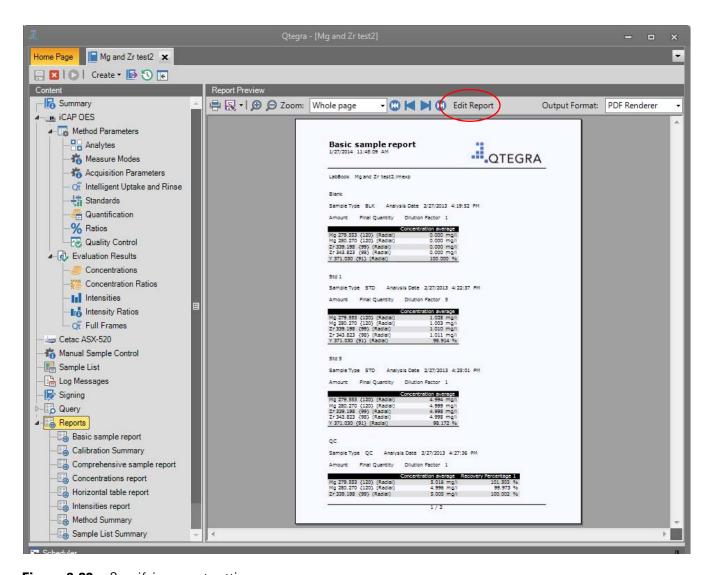


Figure 6-38. Specifying report settings

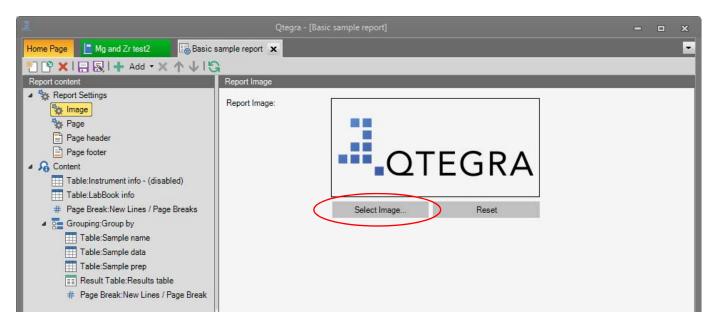


Figure 6-39. Selecting an image for the report

Exporting LabBooks

This can be used to obtain help by e-mail (problem reporting).

❖ To export a LabBook

1. From the Home Page, go to File Manager, right-click a LabBook and select **Export** from the menu. See Figure 6-40.

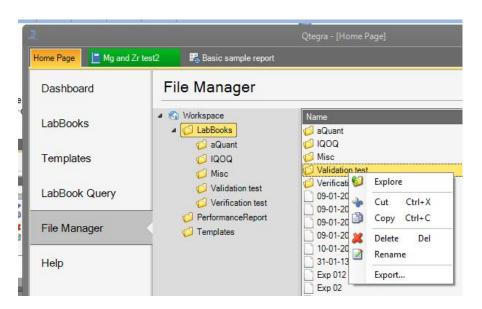


Figure 6-40. Exporting a LabBook

2. Select the location to save the data. Click **OK**. See Figure 6-41.

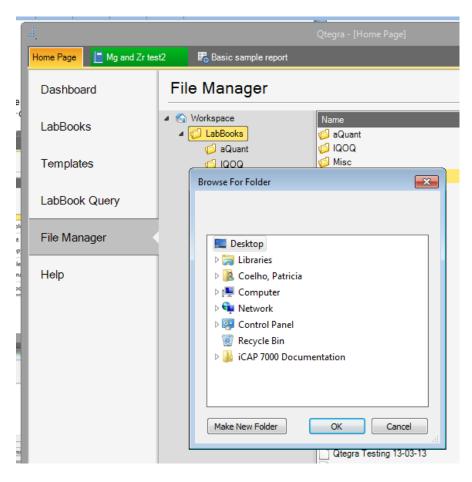


Figure 6-41. Selecting the export folder

Other data (such as instrument logs) can also be exported as a .csv file. This is particularly useful when requesting Service Engineer help. See Figure 6-42.

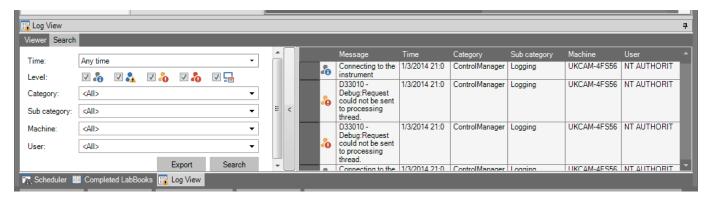


Figure 6-42. Exporting a log

Auto Peak Adjust

For optimum performance, it is important that the analyte wavelengths are correctly aligned in the center of the sub array measurement window. The iCAP 7000 Plus Series ICP-OES automatically checks a reference wavelength each time the plasma is ignited to maintain wavelength accuracy.

AutoPeak only needs to be run when a new wavelength is used for analysis. It may also need to be run if the instrument has been switched off for an extended period of time.

❖ To carry out an AutoPeak

1. Let the plasma stabilize for 10 minutes.



- 2. In **Methods Parameters > Acquisition Parameters** of your LabBook, click the **AutoPeak** icon.
- 3. In the Select analytes dialog box, select the check boxes for the analytes you want to perform an AutoPeak for. Click **OK** to confirm your selection and to close the dialog box.
- 4. Aspirate the High Concentration Standard Solution, that is the method development solution containing all the elements to be auto peaked in sufficient concentration.
- 5. Ensure that you leave enough time for the sample to enter the plasma.
- 6. Click **OK**. See Figure 6-43.

This procedure sets the default positions for the selected wavelengths until the next Auto Peak adjustment takes place. If not all elements to be auto peaked are in the same solution, multiple standards may be used.

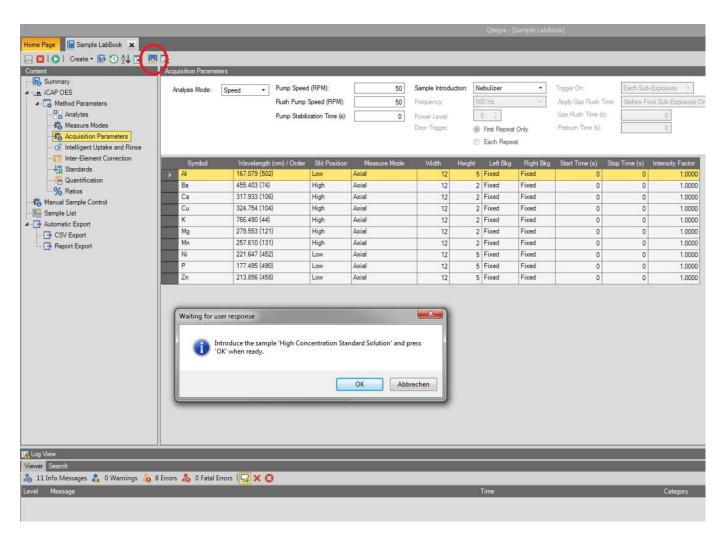


Figure 6-43. AutoPeak procedure

The result of the test is displayed at the bottom of the page.

If the test is unsuccessful or only partially successful, a message in the Log View tab is displayed stating the reason of the failure. This is usually due to the solution not being aspirated for long enough before the test began, or a problem with the solution. See Figure 6-44.

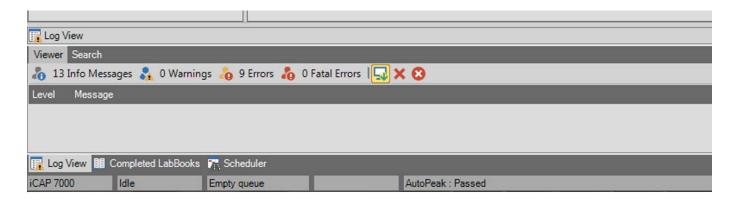


Figure 6-44. AutoPeak procedure result

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** to open the program.
- 4. On the Dashboard page, click the "green ringed" **Get Ready** icon to open the toolbar.
- 5. Click **Shut Down**. See Figure 6-45.



Figure 6-45. System shutdown

The optical components move to a parked position after about 30 seconds. The iCAP 7000 Plus Series ICP-OES shuts down and the plasma is switched off.

The state of the Configuration changes to **NotReady**.

6. When using the Sprint Valve on the iCAP 7600 ICP-OES, it has to be emptied at the end of a run.

Go to the manual Sprint Valve control section. See "Sprint Valve" on page A-2.

7. Verify both Sample and Rinse probes are in air.

Confirm or set the Sprint Valve to **Load**.

Turn on the vacuum pump for several seconds to evacuate the Sample Loop and the Valve.

When the Sample Loop and Valve are emptied, turn **Off**.

8. After switching off the plasma, wait two minutes before switching off the water chiller and the gas supplies. Follow the instructions in the manufacturer's manual for switching off the chiller.

NOTICE

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

- 9. After the plasma has been switched off, wait five minutes before switching off the power to the instrument.
- 10. Release the tension on the sample pump platens to preserve the life of the pump tubing.

System OperationShutting Down the System

Chapter 7 Troubleshooting

This chapter offers solutions for typical problems with plasma ignition, plasma going out unexpectedly and problems with analytical results.

Contents

- Safety Guidelines for Troubleshooting on page 7-2
- Problems with Plasma Ignition on page 7-3
- Resolving Problems with Analytical Results on page 7-5
- Resolving Specific Analytical Problems on page 7-7
- Suggested Maintenance in Case of Poor Precision and Detection Limits on page 7-12

Safety Guidelines for Troubleshooting

When troubleshooting the iCAP 7000 Plus Series ICP-OES system, pay attention to the following general safety guidelines.

∴ WARNING

Toxic Gases. Risk of intoxication. Toxic gases are released that might lead to severe bodily harm when the spray chamber is disassembled. Do not operate the spectrometer without an effective fume extraction system attached to the torch compartment 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. If the extraction is turned off, sufficient air must be supplied to prevent the concentration of these gases reaching a harmful level. Ensure that the laboratory is well ventilated.

ACAUTION

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

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

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 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 at "Shutting Down the System" on page 6-32. Do not use the door to shut down the system.

ACAUTION

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.



Hot Surface. Risk of burns. Let any hot components cool for at least 10 minutes before you remove them from the torch compartment.

Problems with 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. Ensure 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 lights 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.

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, ensure 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 with a regulator close to the instrument.

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

- 2. Check your gas filter(s) on the argon gas input, if fitted. If they are dirty, clean or replace it/them.
- 3. If the plasma still fails to light, see "Ignition Spark" on page 7-4 and "RF Power to the Coil from the RF Generator" on page 7-4.

Ignition Spark

During the ignition sequence, there is first some gas purging, and then the instrument uses a spark to ignite the plasma.

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 at "Argon Gas" on page 7-3.
- 3. Look inside the torch box to check the position of the ignitor relative to the torch.

If it is more than a few millimeters from the torch, ignition might be affected.

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 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. Check that the chiller is operating correctly and that there is enough water in it.

5% of the recommended inhibitor solution must be added to the water.

- 3. Check the water filters on 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, if necessary, clean the glassware.
 - Large droplets of sample entering the plasma can put it out.
- 3. Check for leaks in your gas supply and sample introduction system.
 - Air introduced into the plasma can cause it to go out.
- 4. Ensure 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.
- 5. Check if any automatic updates or security/firewall activities might have occurred at the time of the issue. Check the log messages dialog for more information.
 - It is possible that the computer problems cause an interruption of the operation of your instrument.

Resolving Problems with 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 analysis.

❖ To check sample introduction

- 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. It is advisable to change the pump tubing just before starting the soak tests.

- 3. Check 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. Check that there are no air bubbles in the nebulizer and that the connector is pushed fully home.
- 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. Check 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 with the plasma running. 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. Check 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 for a couple of minutes or so with a 10% Decon 90 solution. Aspirate the Decon with the plasma running.
- 10. Ensure 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 as shown below.
- 11. Check for leaks in the uptake tubing by checking for air bubbles in the tubing connecting to the pump.
 - If there are bubbles, check the junction between the uptake tube and the tubing connection to the pump, which is the most likely place for a leak.
- 12. Check 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 for a couple of minutes with a 10% Decon[™] 90 solution. Aspirate the Decon with the plasma running.

14. Make sure that your torch is fitted properly. This is especially important with a iCAP 7000 Plus Series ICP-OES Duo to ensure that Radial view is good.

❖ 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 6-32.
- 2. Clean 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 torch holder.
- 3. Assemble the nebulizer and the spray chamber.
- 4. Fit the torch and the center tube into the instrument.
- 5. Add the spray chamber 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 7-12.

Poor Precision

- Run a quick test to determine whether the poor analytical results are matrix-related.
- 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.
- After standardization, the standard should read back, on the iCAP 7000 Plus Series ICP-OES, for example, with a precision of typically 0.2 to 0.5% (with a 10 second integration time).
 - If the precision obtained is substantially greater than this, try torch alignment. See "Torch Alignment" on page 8-9. Go on to troubleshoot further, if the problem persists.
- If the instrument meets these specifications, the sample matrix itself is suspect.
- Possibly, modifying plasma parameters such as power help the situation.

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

- First, ensure that the nebulizer pressure or flow is set correctly by aspirating a 1000 ppm yttrium solution (sodium also works if no yttrium is available).
- Ensure that the center orange "bullet" is even with or slightly above the top of the Radial torch. If not, adjust the nebulizer pressure up or down until the "bullet" looks correct.
- At this point, the nebulizer pressure should be approximately 0.15 MPa (25 psi) for aqueous solutions. If the pressure is substantially higher, the nebulizer orifice is generally to blame and should be cleaned.
- 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.





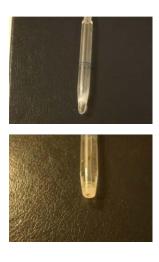


dirty spray chamber



clean spray chamber

• 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, check the inside of the orifice, by removing the gas fitting. Then, with a magnifying glass, look for any small obstruction. Also check the capillary for obstructions.



Teflon Capillary Tubing

The Teflon capillary tubing, Figure 7-1, should be free of kinks and scissor or pinch cut ends. For best results, razor cut at 45° for the drain. Also check the sample peristaltic pump tubing condition and tubing pressure for proper adjustment.

Replace the tubing if it is used or collapsed. 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 7-1. 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 kits.

Peristaltic Sample Pump



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

Tip When using 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.



Figure 7-2. Peristaltic sample pump

❖ 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 pushing in the tension adjustment lever 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.
 - Finally, argon/air leaks can cause many problems including poor precision.
- 5. Check the gas fittings on the lines coming from the bulkhead to the torch and nebulizer with a suitable soapy liquid such as Snoop™. Leaks at these joints are usually caused by rough tubing and can be stopped by cutting off about 1/2 inch of the tubing and reinserting it. Replace the tubing with 1/4 inch Teflon™ if the tubing becomes too short.

Poor Accuracy/Feedback

Accuracy in this manual means reproducing the standard value that was once standardized. Proceed by making a standard as in the previous section (10 ppm Cu, Mn, Cd, Zn, Fe) and using this as the test solution. Remember that we are not defining accuracy for this test 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, check the pump tubing first. Replace it if it is used or collapsed.

Tip Depending on the frequency of usage, 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.

- 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.
- 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 at "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 7-12
Clean the nebulizer	weekly	See page 7-13 and following
Clean the plasma torch	weekly	See page 8-3
Clean the Sprint Valve	weekly	See page A-16

Replacing the Pump Tubing

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

Table 7-2. Tubing materials

Туре	Solvent Types
Tygon [™]	Aqueous solutions, strong acids and highly polar organic solvents (methanol and ethanol, for example)
Viton [™]	Solvents of low polarity (for example alkanes, aromatics and halogenated hydrocarbons such as gasoline, kerosene, toluene, xylene, chloroform and carbon tetrachloride)
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 it with deionized water.

Preventing 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 turning the gas off. 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 isopropyl alcohol.

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.



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, performance of the nebulizer can decline.

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 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 venturi suction draw it through the capillary.
- 4. The final rinse should use isopropyl alcohol 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

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 isopropyl alcohol 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 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 isopropyl alcohol and dry it thoroughly.
- 6. Immerse the nozzle in a rinse solution. Warm the solution for stubborn deposits. Follow with a rinse of pure solvent, then isopropyl alcohol and dry it thoroughly.

Removing Organic Matter



Corrosive Acids. When you handle the corrosive acids required to perform the cleaning procedures described here, take the following 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 commencing 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 isopropyl alcohol 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 at the sample input tube.



Avoid overheating residues that might produce insoluble pyrolysis products.

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

Chapter 8 Maintenance

iCAP 7000 Plus Series ICP-OES are designed for minimum maintenance. Therefore, routine user maintenance of the iCAP 7000 Plus Series ICP-OES is mainly concerned with keeping the instrument clean. However, it is critical to check 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, check with the manufacturer that the proposed method does not cause damage to the equipment.

Contents

- Safety Guidelines for Maintenance on page 8-2
- Cleaning the Instrument on page 8-3
- Preventive Maintenance on page 8-8
- Torch Alignment on page 8-9
- Thermo Fisher Scientific Service on page 8-10

Safety Guidelines for Maintenance

When performing maintenance on the iCAP 7000 Plus Series ICP-OES system, pay attention to the following general safety guidelines.

ACAUTION

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

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

ACAUTION

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.

ACAUTION

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 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 at "Shutting Down the System" on page 6-32. Do not use the door to shut down the system.

ACAUTION

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.



Do not open the instrument or remove covers. No user serviceable parts are inside.

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.

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 7000 Plus Series ICP-OES covers are made of ABS plastic which can be damaged by solvents and concentrated acids.

Cleaning and Decontaminating the Sample Introduction System

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 at "Shutting Down the System" on page 6-32. Clean components contaminated with sample residues.

Thermo Fisher Scientific recommends that several spares for each part are 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

ACAUTION

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

ACAUTION

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 at "To shut down the system" on page 6-32.
- 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

- 1. Place the torch in a muffle furnace and heat it to 750 °C.
- 2. Open the door 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.
- 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 Spray Chamber

If the spray chamber becomes greasy and droplets form on the inside (see Figure 8-1, left), soak the spray chamber in a dilute analytical-grade surfactant for five minutes.

If the spray chamber becomes dirty or deposits form inside it, 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 8-1. Spray chamber: dirty (left) - clean (right)

Cleaning the Nebulizer

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, use an Eluo™ or similar to get rid of the blockage. See "Preventing Blockage of the Nebulizer" on page 7-13 for details.

NOTICE

Do not put the concentric nebulizer in an ultrasonic bath or heat it in an oven.

Cleaning the Purged Optical Path Window

Before you clean the Purged Optical Path (POP) window, turn off the plasma and let any hot areas cool for 30 minutes.

❖ To clean the POP window

- 1. Shut down the system as described at "To shut down the system" on page 6-32.
- 2. Open the small access door next to the sample compartment door. See Figure 8-2.



Figure 8-2. Access to POP window

3. Withdraw the POP window holder, Figure 8-3.



Figure 8-3. POP window holder

- 4. Clean the POP window using a lint-free cloth and clean water.
- 5. Repeat the cleaning procedure with methanol.
- 6. When the POP window is dry, re-insert it into the fore optic assembly.



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 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 at "Shutting Down the System" on page 6-32. Do not use the door to shut down the system.



As all mirrors in the optical system are coated, do not touch the mirror below the radial view POP window in the Duo configuration.

- 7. If further cleaning is necessary, remove the quartz window from the POP window holder. Soak in cold acid for two hours. A mixture of nitric acid and hydrochloric acid similar to aqua regia is suitable.
- 8. Rinse in deionized water.
- 9. Rinse with a volatile, zero residue, organic solvent (propanol is suitable) to aid drying.

Cleaning the Radial View Interfaces

If you are using a Duo instrument, the interfaces for the radial view can get dirty and have an impact on the measured signal.

When using the radial glass window for radial view, the cleaning procedure is as follows:

❖ To clean the radial glass window

- 1. Shut down the system as described at "To shut down the system" on page 6-32.
- 2. Remove the torch from the torch box.
- 3. Remove the glass bucket from the radial view periscope.

Tip An O-ring holds the bucket in place.

- 4. Clean the bucket using a lint-free cloth and clean water.
- 5. Repeat step 4 with methanol.
- When the radial glass window is dry, re-insert it into the holder.
 Figure 6-28 shows how the radial glass window should be fitted.
- 7. Re-insert the torch.

When using the radial POP interface for the radial view, the cleaning procedure is as follows:

❖ To clean the radial POP interface

- 1. Shut down the system as described at "To shut down the system" on page 6-32.
- 2. Remove the torch from the torch box.
- 3. Remove the radial POP interface from the radial view periscope.

Tip An O-ring holds the bucket in place.

4. Remove the green O-ring from the bottom of the interface by using the pointed tip of the alignment tool. See Figure 8-4.



Figure 8-4. Removing green O-ring

- 5. Clean the circular glass window using a lint-free cloth and clean water.
- 6. Repeat step 4 with methanol.

7. When the circular glass window is dry, re-insert it into the bottom of the radial view interface.

Insert the green O-ring into the groove and push it in tightly with the alignment tool. See Figure 8-5.



Figure 8-5. Inserting green O-ring

Figure 6-30 shows how the radial POP interface should be fitted.

8. Re-insert the torch and align the radial POP interface as described at "To align the radial POP interface" on page 6-18.

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 ensure that local health and safety guidelines and regulations are complied with. Check the gas and cooling water for leaks at regular intervals.

Water Chiller

It is critical to the performance of your instrument that the cooling fluid used for your iCAP 7000 Plus Series ICP-OES is made up correctly as specified in the *iCAP 7000 ICP-OES Pre-Installation Requirements Guide*. An effective maintenance plan includes replacing the cooling fluid with new fluid periodically depending on the usage of your instrument, and also to ensure that any air filters and water filters are kept clean. Refer to the manufacturer's documentation of the chiller for details.



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

Water Filter

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

If the filter appears dirty

- 1. Replace it.
- 2. Flush your water system.
- 3. Replace it with correctly made up cooling fluid.

Gas Filters

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

Torch Alignment

For maximum sensitivity and optimum results, the alignment of the plasma image is critical. When a torch has been removed or replaced in the instrument, or if the torch body or center tube has been replaced, a torch alignment procedure should be carried out.

To align the torch



- 1. Click the Qtegra icon on the computer desktop.
- 2. On the Dashboard, click **Torch Alignment** to start the procedure.
- 3. Aspirate the so-called "Loaded Blank" solution as supplied with the iCAP 7000 Plus Series ICP-OES or any other aqueous 2 ppm Zn solution.

Optionally, use an organic solvent 2 ppm Zn solution, which applies slightly different plasma settings (higher auxiliary gas flow, slightly decreased nebulizer gas flow, slightly decreased pump speed). In this case, select the **Organic Matrix** check box next to the Torch Alignment button.

- 4. Leave enough time for the sample to enter the plasma.
- 5. Click OK.

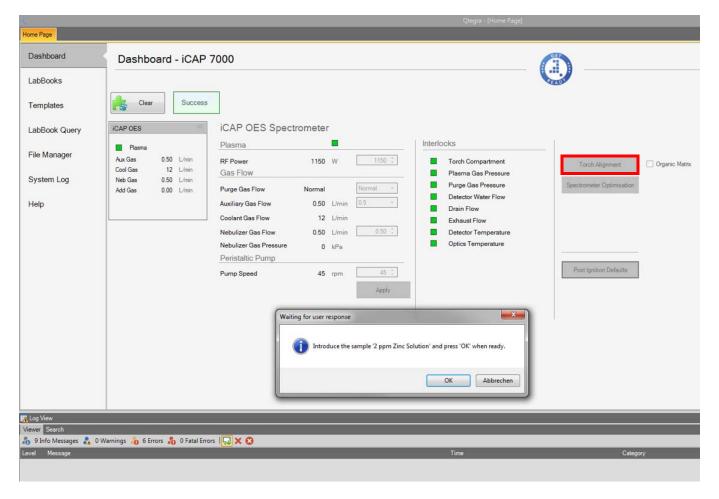


Figure 8-6. Torch alignment procedure

6. Aspirate until the process is finished.

The result of the test is displayed at the bottom of the page as well as in the journal.

Thermo Fisher Scientific Service

This section contains information concerning 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 or performing maintenance. Safety documentation and the decontamination certificate must be made available to the Thermo Fisher Scientific field service engineer.

Safety Advice for Possible Contamination



Hazardous Chemicals. Hazardous material might contaminate certain parts of your system during analysis. To protect our employees, we ask you to adhere to special precautions when you return parts for exchange or repair.

If hazardous materials have contaminated instrument parts, Thermo Fisher Scientific can only accept these parts for repair if they have been properly decontaminated.

Materials that due to their structure and the applied concentration might be toxic 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 unsure whether parts of the system are possibly contaminated by hazardous material, make sure that the Thermo Fisher Scientific field service engineer is informed before the engineer starts working on the system.

Returning Parts

To protect our employees, we ask you for some special precautions when you send parts back to the factory for exchange or repair.

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 C-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.

Maintenance

Thermo Fisher Scientific Service

Appendix A Advanced Hardware

This appendix describes the components that are integrated parts of the iCAP 7600 ICP-OES and optional accessories for the other spectrometers of the iCAP 7000 Plus Series ICP-OES.

Contents

- Sprint Valve on page A-2
- Additional Gas Accessory on page A-24

Sprint Valve

This section describes the procedures for using the Sprint Valve rapid sample introduction system.



Explosion Hazard. Do not operate the Sprint Valve in an explosive atmosphere.

Supported Autosamplers

The integrated Sprint Valve rapid sample introduction system works with many autosamplers including the following:

- CETAC[™] ASX-280 (old model: ASX-260)
- CETAC ASX-560 (old model ASX-520)
- CETAC ASX-520HS
- CETAC ASX-520/520HS with XLR-8 extended rack
- CETAC ASX-1400
- CETAC ASX-1600

Functional Description

The 6-port/7-port injection valve is constructed of polyphenylene sulphide (PPS), an inert and dimensionally stable material. The tubing is all inert PTFE (waste tubing materials may vary by application). Components in the sample flow path are inert, non-metallic materials. They can withstand repeated exposure to the following substances:

- Predominantly aqueous solutions of strong acids (less than 40%).
- Common organic solvents such as acetone, alcohols, ethyl acetate, methylethylketone (MEK), petroleum oils and derived fuels, tetrachloroethylene, toluene, and xylene.
- When using a 7-port valve, port 7 can be used to add an internal standard online.



Prolonged or repeated exposure to temperatures above 135 °C and to the following substances can cause failure of the flow path components:

- Solutions of concentrated acids (greater than 40%)
- Partially halogenated hydrocarbons or extremely aggressive organic solvents (chloroform, methylene dichloride, 1,1,2-trichloroethane).

Plumbing Connections

NOTICE

Establish the plumbing connections without using tools. Using tools such as screwdrivers or pliers to perform installation tasks might result in a damaged or unusable instrument. Do not tighten fittings with anything other than your fingers.

Set up the Sprint Valve as described at "Connecting the Tubing" on page A-5. The plumbing configuration is crucial to the proper operation of the Sprint Valve system and must be followed. See Figures A-1 to A-4.

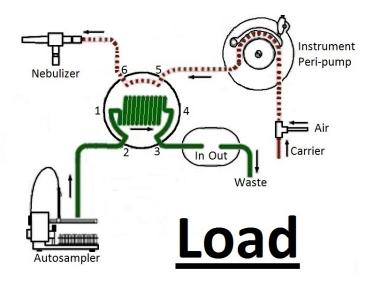


Figure A-1. Standard configuration for loading sample into sample loop

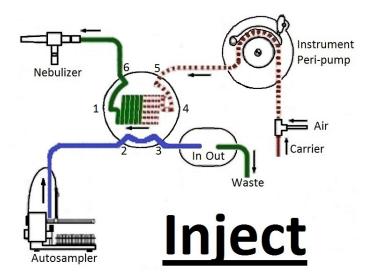


Figure A-2. Standard configuration for injection of sample from sample loop

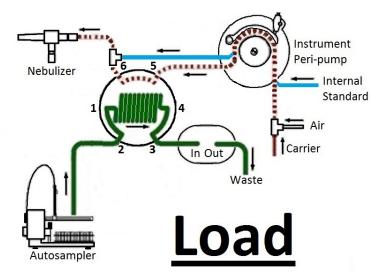


Figure A-3. Standard configuration for loading sample into sample loop with additional internal standard

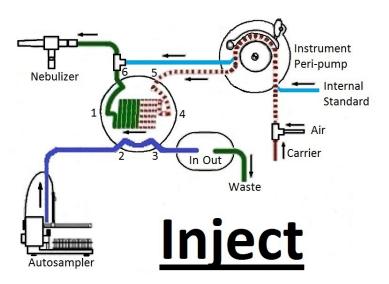


Figure A-4. Standard configuration for injection of sample from sample loop with additional internal standard

Connecting the Tubing

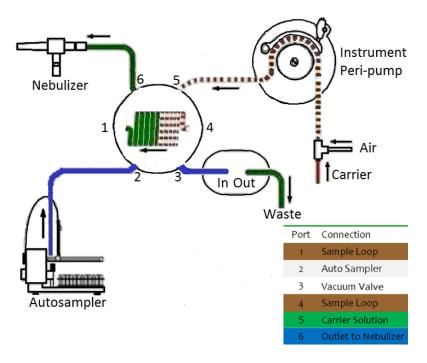


Figure A-5. Sprint Valve tubing connections with 6-port valve

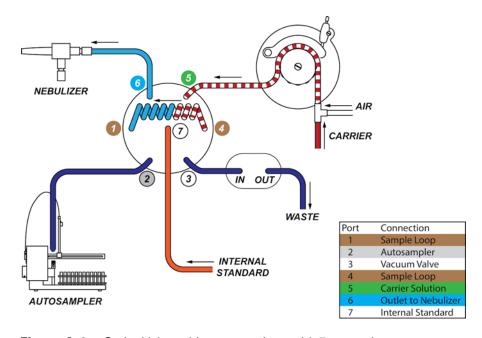


Figure A-6. Sprint Valve tubing connections with 7-port valve

❖ To connect the tubing

1. Fit the drain fitting to the spray chamber (black ringed). Insert the fittings capillary tube into the yellow/blue peristaltic pump tubing and attach it to the pump so that the waste runs counterclockwise across the pump.



Cut a length of the supplied drain tubing so that it can reach between the pump tubing and the waste container.

2. Insert the tubing with the green valve fitting and the carrier uptake tube (bubbler) into the white/white peristaltic pump tubing.



3. Attach the pump tubing onto the peristaltic pump so that the bubbler runs counterclockwise across the pump.



4. Screw the green valve fitting into port 5 on the valve head.



5. Take the white valve fitting tube and attach one end to port 3 on the valve head.



6. Screw the other end of the fitting tube into the vacuum valve connector of the iCAP 7000 Plus Series ICP-OES.



7. Attach the vacuum pump waste tubing to the waste out connector of the iCAP 7000 Plus Series ICP-OES.

Insert the other end into the waste container.



8. Screw the autosampler probe valve fitting into port 2 of the valve head.



9. Attach the sample loop into ports 1 and 4 of the valve head (this is the same as the loop provided in the Field Service Test Kit).



10. Screw the fitted nebulizer tube into port 6.



11. Attach the other end on the nebulizer line and the nebulizer gas fitting onto the nebulizer.



12. In case that a 7-port valve is used, screw the yellow internal standard fitting into port 7 in the center of the Sprint Valve.

Attach the other end of the internal standard tube to the green-orange internal standard pump tubing.

Attach the pump tubing onto the peristaltic pump so that the internal standard solution runs counterclockwise across the pump.



Operational Settings

The operational settings of the iCAP 7000 Plus Series ICP-OES Sprint Valve are either controlled

- by a separate software package (Sprint Valve Configurator), which is installed along with your installation of Qtegra ISDS Software or
- from within a plugin in the Qtegra ISDS Software (software version 2.7 or higher)

Both options let the user upload and save the operational settings of the Sprint Valve in the firmware of the instrument.

Setup with the External Sprint Valve Configurator

To operate the Sprint Valve with the external software package, the cables between Sprint Valve, autosampler and PC need to be connected as shown in Figure A-7.

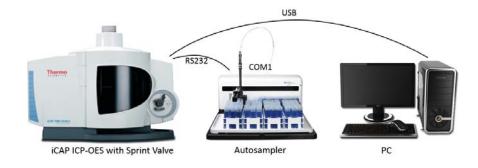


Figure A-7. Cable connections with external Sprint Valve Configurator

The Sprint Valve Configurator is not required to operate the valve on a day-to-day basis. It is rather used to enable or disable, to set up and change the valve settings, and to provide manual control of the valve.

When the software is loading, it looks like Figure A-8, with most of the options not being available.

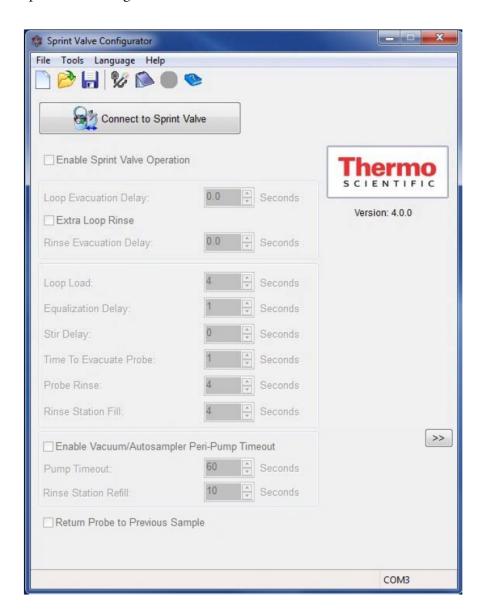


Figure A-8. Sprint Valve Configurator

- To define the Sprint Valve settings with the external Sprint Valve Configurator
- 1. Click Connect to Sprint Valve.

The parameters of the Sprint Valve Configurator window become available. See Figure A-9.



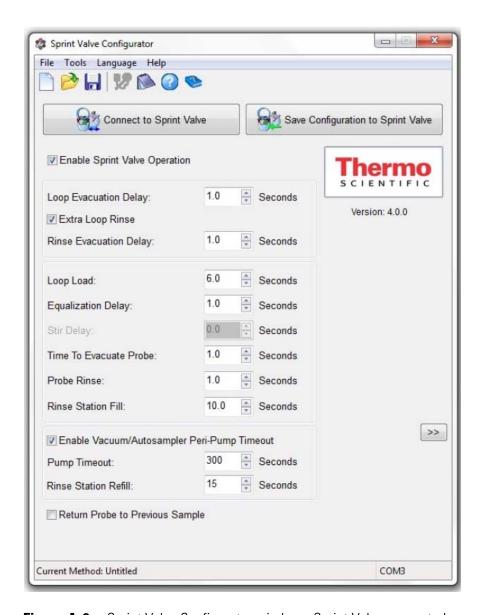


Figure A-9. Sprint Valve Configurator window - Sprint Valve connected

- 2. Select and modify the required options. See also Table A-1.
- 3. Click **Save Configuration to Sprint Valve** to upload these settings into the firmware of the valve.

The new settings are saved.

Setup with the Sprint Valve Plugin in Otegra ISDS Software

To operate the Sprint Valve with the plugin in Qtegra ISDS Software, the cables between Sprint Valve, autosampler and PC need to be connected as shown in Figure A-10.



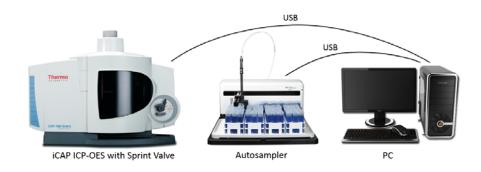


Figure A-10. Cable connections with Otegra ISDS Software plugin

The configuration of the Sprint Valve is performed in the Qtegra Configurator in the autosampler plugin. This configuration is not required to be changed to operate the Sprint Valve on a day-to-day basis. It is rather used to enable or disable the Sprint Valve and to set up and change the valve settings. See Figure A-11 and Figure A-12.

To define the Sprint Valve settings with the Sprint Valve plugin

1. Enable Sprint Valve in the autosampler plugin in the configuration. See Figure A-11.

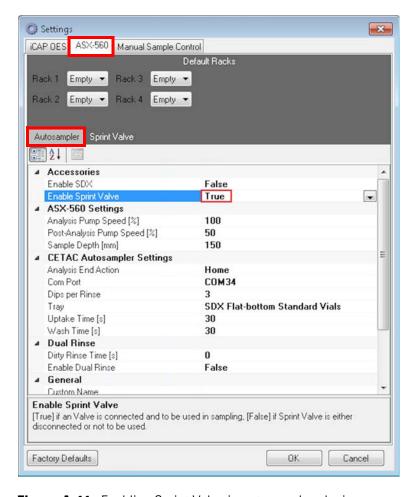
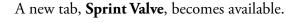


Figure A-11. Enabling Sprint Valve in autosampler plugin

Thermo Scientific



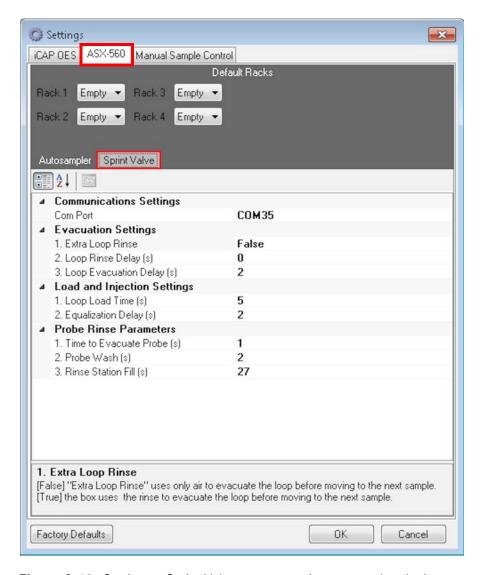


Figure A-12. Setting up Sprint Valve parameters in autosampler plugin

- 2. Click the **Sprint Valve** tab.
- 3. Select and modify the required settings. See Figure A-12 and Table A-1.

When the instrument configuration is saved, the settings are uploaded to the Sprint Valve.

Description of Timings

Table A-1 lists the available Sprint Valve timing parameters.

Table A-1. Sprint Valve settings

Description
Enables/disables the Sprint Valve control, allowing the autosampler to be used without the valve
Time spent evacuating the loop before the sample is loaded
Enables rinse solution to be pumped through the loop
Time taken to perform extra loop rinse
Time for vacuum pump to load sample loop
Time to let the sample settle in the loop before the valve is switched
When using the ASX 1400 stirring autosampler, time required to stir the sample
Time to drain probe before rinse
Rinse time using the autosampler rinse during analysis
Time for autosampler peri pump to continue after rinse uptake to refill rinse station
Enables/disables the pump timeout after safety feature
Maximum time allowed between pump operations before the analysis is aborted
Time taken to refill the rinse station after a pump timeout
Activates option to have the autosampler probe to wait over the sample being analyzed after rinse operations are completed
This can reduce analysis time be several seconds.

^a Setting only available in external Sprint Valve Configurator

Figure A-13 shows a time diagram of the Sprint Valve operation. The Qtegra uptake time is a total of the following:

- Loop Evacuation Delay
- Rinse Evacuation Delay/Loop Rinse Delay
- Loop Load
- Equalization Delay
- Time to get the sample from loop to plasma
- Stabilization time (~5 seconds)

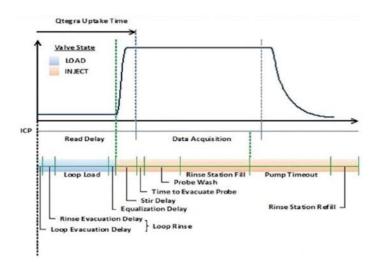


Figure A-13. Time diagram of Sprint Valve operation

Manual Control Features

Manual control of the Sprint Valve is necessary during maintenance of the Sprint Valve or at the end of analysis to clean and dry the system.

The manual control features of the Sprint Valve can be accessed either through the external Sprint Valve Configurator or through the Sprint Valve plugin in Qtegra ISDS Software.

External Sprint Valve Configurator



To access the manual control features in the external Sprint Valve Configurator, click this button. The window expands to show the manual control features. See Figure A-14.



Figure A-14. Manual control features in external Sprint Valve Configurator

Sprint Valve Plugin in Otegra ISDS Software

The manual control features of the Sprint Valve, using the plugin in Qtegra ISDS Software, are accessible through the Autosampler tab on the Qtegra Dashboard. See Figure A-15.

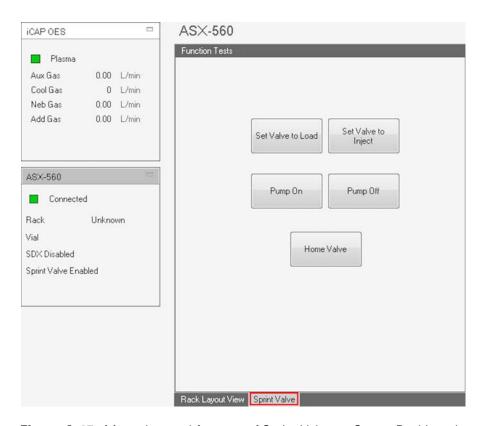


Figure A-15. Manual control features of Sprint Valve on Otegra Dashboard

Table A-2 lists the manual control features.

Table A-2. Manual control features

Feature	Description
Home AutoSampler ^a	Sends the autosampler to the home position (above the rinse)
Autosampler Pump Control On/Off ^a	Turns autosampler rinse pump on and off
Valve Control Load/Inject	Switches the valve between load and inject
Home Valve	Rapidly switches the valve between load and inject mode several times.
	"Homing the valve" needs to be performed each time the valve is removed and re-attached.
Vacuum Pump Control	Switches the iCAP 7600 ICP-OES vacuum pump on and off

Table A-2. Manual control features, continued

Feature	Description
Manual Cycle at Rinse ^b	Sets the manual cycle to be performed with rinse solution
Manual Cycle ^b	Gets the valve/autosampler to perform a complete cycle

When using the Sprint Valve plugin, these features are controlled through the autosampler control on the Qtegra Dashboard.

Manual Sampling Using the Sprint Valve

Manual sampling uses the Inject position **5** with a standard or non-aerating sample probe. To perform manual sampling, for example for the torch alignment or AutoPeak features, remove the bubbler from the white peristaltic pump tubing and replace it with a standard sampling probe. See Figure A-16.

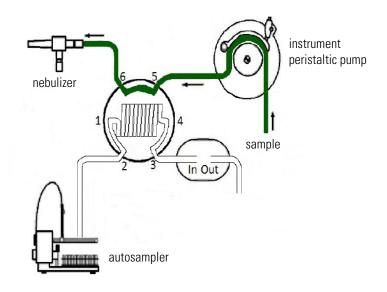


Figure A-16. Manual sampling

Routine Maintenance

- Routine maintenance of the Sprint Valve rapid sample introduction system consists of daily and weekly cleaning of specific components.
- Routine maintenance also includes checking the Sprint Valve components for leaks or other damage.
- Additional periodic maintenance tasks may be required, including replacement of peristaltic pump tubing, rinse tubing, and sample probe.

^b Only available in the external Sprint Valve Configurator

Regular Inspection of Components

It is important to verify that all system components are in good working order and are undamaged before operation.

Visually inspect these components:

- Valve/pump module
 - 6-port/7-port valve
 - Vacuum pump ports
- Peristaltic pump on the autosampler
- Tubing

Pay special attention to all tubing to ensure that no kinks exist, because this condition impairs proper performance of the Sprint Valve system by reducing flow rates.

Check that the tubing is not rubbing against moving parts.

Cables

If you detect a leak or other damage to any Sprint Valve Rapid Sample Introduction System component, replace it.

Checking for Leaks

The tubing has a finite lifespan, and wears out under normal use. Standard maintenance procedures require that you periodically check for leaks.

To check the tubing for leaks

- Visually inspect all tubing and valves for leaks or signs of deterioration.
- Visually inspect the surfaces below all tubing for signs of liquid.

If you detect a leak or other damage to any component, replace it.

❖ To replace the tubing

Remove and replace all tubing as necessary. Remove/replace tubing at barb fittings and at compression type fittings without causing damage to those fittings to which they connect.

For more information on how to install the rinse tubing, see "Plumbing Connections" on page A-3.

Cleaning the System

Cleaning the Sprint Valve Rapid Sample Introduction System is a primary maintenance task. Failure to do so regularly causes increased wear and reduces the life of the system.

Clean the Sprint Valve Rapid Sample Introduction System both daily and weekly to protect the instrument, prevent damage and extend its life. It is especially important to clean up spills, which might infiltrate the instrument case/cabinet whereby damage to components might result. It may also be necessary to chemically neutralize spills. The following sections explain daily and weekly cleaning procedures.

Daily External Cleaning

The Sprint Valve is often operated in environments where spills and exposure to vapors are common. Good maintenance requires that you clean the system daily.

Cleaning the 6-Port/7-Port Valve

It is necessary to periodically disassemble the 6-port/7-port valve and clean the inside to prolong the life of the valve. Cleaning must be performed in a clean area to prevent contamination of the valve.

Tip Thermo Fisher Scientific recommends cleaning the 6-port/7-port valve every 20,000 cycles, or approximately every 1–2 weeks. However, the frequency of cleaning varies depending on the application. The higher the concentration of dissolved solids, the more frequent cleaning is required.

Clean the valve head regularly as part of the routine maintenance of the sample introduction components (spray chamber, nebulizer, etc.).

Tools

The following tools are required:

- 9/64 inch hex wrench
- 3/32 inch hex wrench

❖ To clean the valve

- 1. Dry out the loop as follows:
 - a. Send the probe to the Home position above the rinse station.
 - b. In the manual control of the Sprint Valve, set the valve to **Load**.
 - c. Turn on the vacuum pump until the loop is dry.
 - d. Turn off the vacuum pump.

2. Remove all connections from the 6-port/7-port valve.

NOTICE

Be careful with the nebulizer connection.

3. Use the 9/64 inch hex wrench to loosen the metal collar. See Figure A-17.

The screws do not need to be completely removed, just loosened enough to pull off the 6-port/7-port valve.



Figure A-17. Loosening metal collar on Sprint Valve

4. Use the 3/32 inch hex wrench to loosen the three top screws. Remove the stator, ring spacer, and the rotor. See Figure A-18.





Figure A-18. Disassembled Sprint Valve showing spacer, rotor and stator

- 5. Inspect the sample channels in the rotor and stator for scratches and debris.
- 6. Clean rotor and stator as required:
 - Option A Sonication
 - i. Sonicate in acid, kerosene, any solvent that works for about 30 minutes.
 - ii. Inspect rotor and stator for scratches or blockage.
 - Option B Cotton swab + solvent
 Inspect rotor and stator for scratches or blockage.

- Option C Use compressed air or argon to clean the surfaces and ports.
- 7. Reassemble the 6-port/7-port valve. The parts only fit together one way.
- 8. Tighten the three 3/32 inch hex screws incrementally in a circle (as if changing a tire). Do not overtighten.

Re-Homing the 6-Port/7-Port Valve

To reinsert the Sprint Valve

1. Before you reinsert the valve into the valve/pump module, use the manual Sprint Valve control to switch the valve position.

The vacuum pump module makes a long tone noise.

When using the external Sprint Valve Configurator, you may see the dialog box that shows an Error 1206 – valve switching error. See Figure A-19.

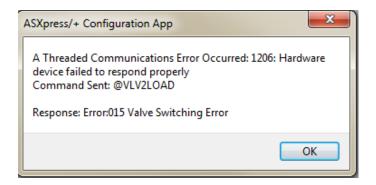


Figure A-19. Error 1206 – valve switching error

- 2. Reinsert the 6-port/7-port valve into the vacuum pump module.
- 3. Tighten the collar. The valve should not move when it is switched.
- 4. Toggle the valve between load and inject positions several times. The vacuum pump module makes a chirping noise when properly homed.

Replacing or Reorienting the 6-Port/7-Port Valve

The 6-port/7-port valve assembly has a finite lifespan that depends upon the conditions and sample media to which it is exposed. Exposure to higher sample solids levels reduces the lifespan of the valve.

❖ To determine whether the 6-port/7-port valve requires replacement

Inspect the unit for these conditions:

- Valve dripping or leaking from the overflow hole behind port 4 at the bottom of the valve body
- With no other apparent problems, air is present in the lines (indicating a leak or poor seal).

The valve can also be reoriented so that the nebulizer port is as close as possible to the nebulizer.

Tip Any time the 6-port/7-port valve body is removed from its actuator, the valve requires retraining (re-initialization).

❖ To replace or re-orientate the 6-port/7-port valve

1. Remove all of the tube connectors to the 6-port/7-port valve. See Figure A-20.



Figure A-20. Removing the tube connectors

2. Using the provided 9/64 inch hex wrench, loosen the hex screw on the locking collar, which secures the base of the valve to the instrument. See Figure A-21.



Figure A-21. Loosening the hex screw

3. Firmly but carefully remove the valve head.

4. Insert the new valve, or reinsert the existing valve, at the desired angle. See Figure A-22.



Figure A-22. Inserting the valve

- 5. Rotate it so that the nebulizer port is as close as possible to the nebulizer.
- 6. Push in the valve to make sure that it is completely seated. There should be no gap between the valve and the collar. See Figure A-23.



Figure A-23. Checking the valve seat

7. Use the hex key to tighten the locking collar. See Figure A-24.



Figure A-24. Tightening the locking collar

8. In the manual Sprint Valve control, click **Home Valve**. See Figure A-25.

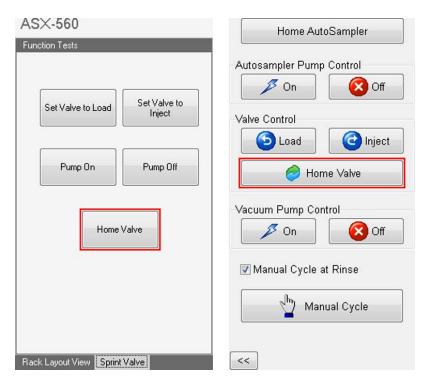


Figure A-25. Home Valve in Sprint Valve plugin (left) and in external Sprint Valve Configurator (right)

9. Reconnect the tubing. See Figure A-26.



Figure A-26. Reconnecting the tubing

10. When using the external Sprint Valve Configurator, check for any leaks using the **Manual Cycle** function. See Figure A-27.

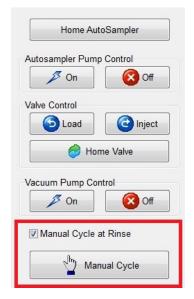


Figure A-27. Manual Cycle

Additional Gas Accessory

The additional gas accessory is fitted as standard in the iCAP 7600 ICP-OES and optional in the iCAP 7400 ICP-OES. The additional gas is added to the plasma to modify the conditions. For example, the additional gas (clean, dry, oil-free air) is used to prevent the building of carbon on the torch when running organic samples.

For best performance, it is recommended that the addition is made to the auxiliary gas in preference to the nebulizer gas (this is the standard configuration). See Figure A-28.



Figure A-28. Additional gas accessory aux gas connections

Installing the Additional Gas Accessory

To install the additional gas accessory

- 1. Connect the "unequal" T-piece provided to the additional gas connection fitting using the short length of the 6 mm tubing provided.
- 2. Connect the nebulizer gas fitting to the T-piece with a second piece of 6 mm tubing.
- 3. Connect the nebulizer tubing to the remaining port on the T-piece.



Follow appropriate precautions that depend on the gas used. These precautions must comply with local and national safety requirements and guidelines.

For safety reasons, 100% (pure) oxygen is not approved to be used as additional gas in the iCAP 7000 Plus Series ICP-OES. For the additional gas MFC, clean dry oil-free air is required.

Thermo Scientific

Advanced Hardware

Additional Gas Accessory

Appendix B Optional Accessories

This appendix describes frequently used accessories that are not part of the standard iCAP 7000 Plus Series ICP-OES installation.

Refer to the *iCAP 7000 Series ICP-OES Accessories Guide* for information about the available accessories. Request site requirement guides for any purchased accessories.

Contents

- Autosampler on page B-2
- Ultrasonic Nebulizer on page B-3
- Hydride Generation on page B-4

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 7000 Plus 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 7000 Plus Series ICP-OES.

Autosampler Setup

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 7000 Plus 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. See Figure B-1.

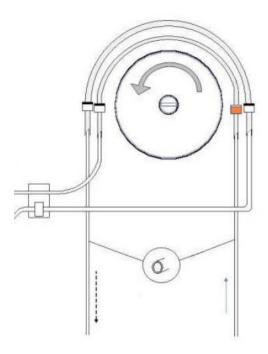


Figure B-1. Schematic of autosampler tubing setup

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%.

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 B-2. CETAC U5000AT+ Ultrasonic Nebulizer

Tip To comply with safety and warranty requirements, the iCAP 7000 Plus Series ICP-OES, accessories and associated equipment must be installed by a certified Thermo Fisher Scientific service representative.

To set up the ultrasonic nebulizer

- 1. Put the ultrasonic nebulizer on the bench to the right of the iCAP 7000 Plus 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 7000 Plus Series ICP-OES with the inlet on the rear panel of the ultrasonic nebulizer.

Optional Accessories

Hydride Generation

- 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.

Tip Because two different peristaltic pumps (standard and mini) are used with the iCAP 7000 Plus Series ICP-OES, tubing sizes are adjusted to fit the purpose. Tubing sizes for the standard pump (mini pump in brackets) are indicated.

On-line Hydride Generation Kit

The on-line hydride generation kit is typically used with the iCAP 7200 ICP-OES. It 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 internal standard with the sample, a mixing loop is provided after the Y-piece before connecting to the nebulizer. Install pump tubing for the Internal Standards Kit in a manner similar to the sample tube and the correct tension set.

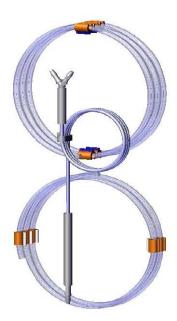


Figure B-3. On-line hydride generation kit with mixing loop

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 hydride generation kit

1. Place the orange/blue (mini: orange/green) pump tubing in the sodium borohydride solution. The orange/white (mini: white/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 B-1 using the plasma control dialog.

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

Parameter	Setting
RF power	1300 W
Auxiliary gas	0.5 L/min
Nebulizer gas typical operating range	0.19-0.29 MPa (~0.5 L/min)
Pump speed	45 rpm

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

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

4. With the iCAP 7200 ICP-OES Duo, vary the nebulizer pressure over the range 0.15–0.20 MPa 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, use that element for optimization.

Integrated Hydride Generation Kit

The integrated hydride generation accessory (see Figure B-4) requires the use of a four channel sample pump and may only be used with the iCAP 7400 ICP-OES or the iCAP 7600 ICP-OES. It consists of an integrated acrylic reaction cell and gas/liquid separator for generation and separation of the hydride gases. It is connected directly to the plasma torch after removal of the spray chamber.



Figure B-4. Integrated hydride generation kit

Setup

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 3 mm ID PTFE drain tubing, respectively.
- Prepare the gas liquid separator as described at "To prepare the Gas Liquid Separator" on page B-8 before use.

Gas Liquid Separator (GLS)

When it is assembled, the reaction zone of the GLS (see Figure B-5) 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.

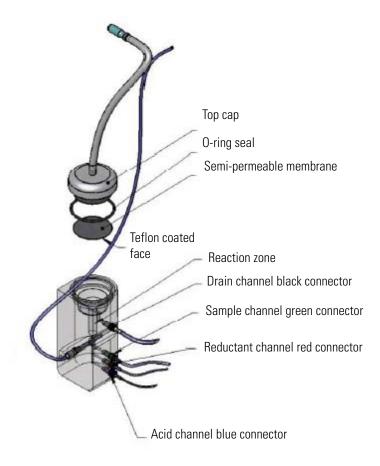


Figure B-5. Schematic of Gas Liquid Separator

To prepare the Gas Liquid Separator

- 1. Unscrew the cap of the GLS. See Figure B-5.
- 2. Add a sufficient quantity of the 4 mm glass beads supplied 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 B-2.

Table B-2. Color coded connectors at GLS unit

Connector color	Transport function	Standard pump tubing	Mini pump tubing
Black	Drain	black/white	purple/white
Green Yellow	Sample	green/green	green/green
Red	Reductant	black/black	black/black
Blue	Acid	orange/yellow	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 hand 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 (mini: PURPLE/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 (mini: 2.794 mm) and an OD of 4.85 mm (mini: 4.47 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 (mini: purple/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 plungers to free the pump platens.
- 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 platen arm hack over the rollers. Confirm that the tubing is properly located beneath it.
- 6. Return the plunger 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 0.4–0.6 L/min.

❖ To optimize the unit

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

Table B-3. Operating parameters for integrated hydride generation

Parameter	Settings
RF power	1300 W
Coolant gas	14 L/min
Auxiliary gas	0.5 L/min
Nebulizer gas	0.5 L/min
Pump speed	45 rpm

- 2. Aspirate a solution of 50 μ g/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 0.4–0.6 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, this element should be used 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.

Optional AccessoriesHydride Generation

Appendix C Legal Documents

Contents

- WEEE Compliance on page C-2
- Declaration of Conformity on page C-3
- Health and Safety Form on page C-5

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

RoHS

For additional information about the Restriction on Hazardous Substances (RoHS) Directive for the European Union, search for RoHS on the Thermo Fisher Scientific European language websites.

Declaration of Conformity

-Original-

EG-Konformitätserklärung EC Declaration of Conformity





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

Designation.

Thermo Scientific iCAP 7000 Serie

Model: Model:

Thermo Scientific iCAP 7000 Series

alle einschlägigen Anforderungen der folgenden Richtlinien erfüllen:

fulfill all the relevant requirements of the following directives:

Niederspannungsrichtlinie

2014/35/EU

Low Voltage Directive

2014/35/EU

Richtlinie über elektromagnetische

2014/30/EU

Verträglichkeit

2014/30/EU

Electromagnetic Compatibility Directive

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 61326-1:2013

EN 61010-1:2010

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

Unterschrift Signature Bremen, 2017-05-17

Datum

Date

Legal Documents

Declaration of Conformity



Health and Safety Form

This Decontamination Declaration Form must be completed for <u>all</u> materials returned to Thermo Fisher Scientific, it should be sent to the destination by e-mail after printing and with an authorization signature. A hard copy 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. 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).

Customer			Instrument t	type		
Address			Instrument S	Instrument SN		
·			Order numb	ner		
Phone			Return num			
E-Mail						
Part Number	Quantity	Material Description	Error Description / Reason for	r Return	Return Part Serial #	
e.g. Thermo Scientific PN	Quarterty	Wideena Bescription	Error Beseription, reason to	· recuiri	Necami are seriar ii	
e.g. Pfeiffer						
e.g. Leybold						
e.g. Edwards						
Has the mater shipping packate pump - service - sample - standate - other	ial or instrum ging or in co fluids, e fluids,		☐ Yes → g	olicable check b o to section 3 o to section 5	oox.	
Has the mater shipping packate pump service sample standate other of or haz	ial or instruminging or in confluids, effuids, effuids, es, and solutions, chemicals, erdous materials.	nentation been removed ntact with rials?	☐ Yes → g	o to section 3 to section 5	ive & contaminated	
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substances

explosive



Health and Safety Form

4. Description of Process Substances and/or Compound

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 material safety data sheet; e.g. toxic, flammable, corrosive, radioactive)

flammable, corrosive, radioactive)								
	Part Number		Serial Number	Trade	Name		Chemical / Substance	e Name / Properties
a)								
b)								
c)								
d)								
e)								
f)								
<u>5. l</u>	Legally bind	ling dec	laration_					
Has	the materia	l/instrun	nent undergone a deco	ntamination	n process?	Y	'es → go to section	6 No
	he material/i d party pers		nt safe to handle for Th	ermo Fishe	r Scientific and	I Y	'es	No
ack inco aga sta The cor	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							
				1				
Par	t Number		Serial Number	Describe ti	ne decontamina	tion process		
Ret	urn 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.

В

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 $\sim 5 \times 10^6$ 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.

Ε

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.

Н

h hour.

h height.

HPLC high-performance liquid chromatograph.

HR abbr. for High Resolution.

HV high voltage.

Hz hertz (cycles per second).

ı

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.

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

LR abbr. for Low Resolution.

M

MEK methylethylketone.

MFC mass flow controller.

MSDS Material Safety Data Sheet.

N

NDRO nondestructive readout.

0

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 μ g/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).

Τ

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

VAC volts alternating current.

VDC 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.

Glossary: WEEE-WEEE

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