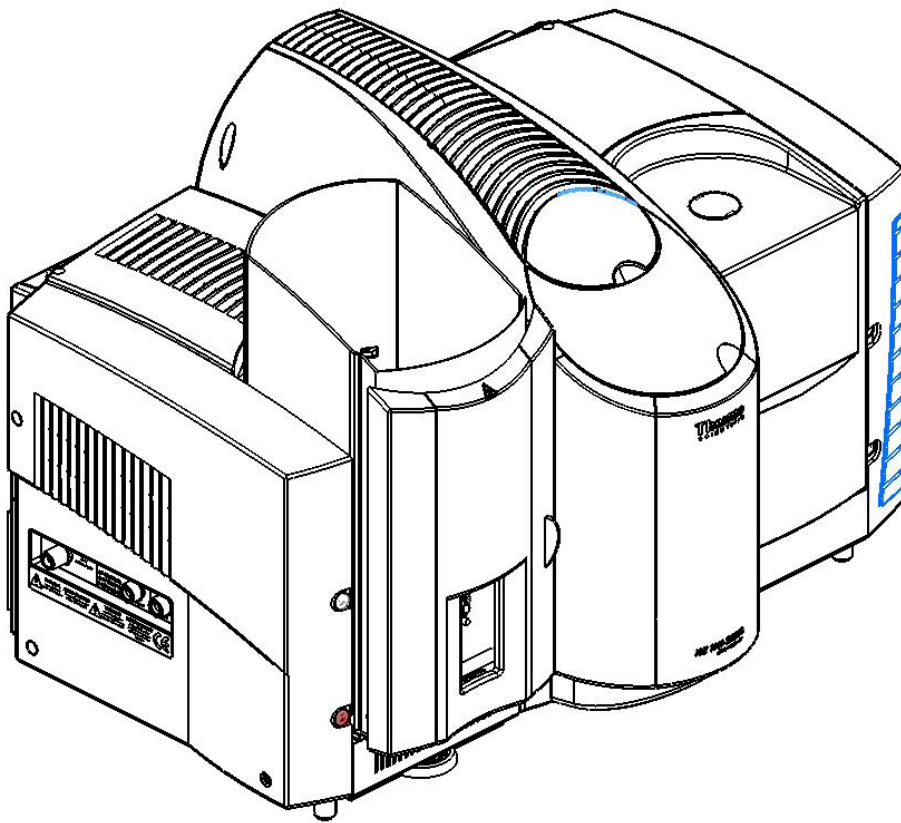


# iCE 3000 Series AA Spectrometers

Operators Manuals

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Version 2.0



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# **Chapter 1 Introduction**

## **The Operators Manuals**

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## The Operators Manual

It describes:

- the safety hazards involved in working with the spectrometer and its accessories, and the means by which such hazards can be minimised.
- the spectrometer.
- the flame atomisation system.
- the flame accessories.
- the furnace atomisation system.
- the furnace accessories.
- the vapour generation accessories.

## The Data Station Software Manual

The Data Station Software Manual is supplied with the SOLAAR Series Data Station Software.

It describes:

- the software installation procedure.
- the functions and features of the Data Station software, including the Online Help System.
- a series of familiarisation experiments to get you up and running quickly with your new instrument.

## Accessory Installation Manuals

Several of the the major accessories are supplied with printed manuals. In general, these describe the use of the accessories with older or obsolete ranges of spectrometers. This Hardware Manual contains the information required to use these accessories with the iCE 3000 Series instruments, which supersedes that provided in the Accessory Manuals.

Some of major accessories require installation; the Accessory Manuals contain the installation instructions to set up the accessories so that they can be successfully used with your Atomic Absorption spectrometer.

## The Data Station On-line Help System

The Data Station software contains a comprehensive Online Help system.

It describes:

- features and functions of the software, as overviews.
- every parameter, function and menu item with context sensitive help.
- step by step procedures for common tasks.
- detailed trouble shooting procedures for identifying problems encountered with your analysis.

## The Data Station Software Wizards

The Data Station Software Wizards guide you through several common procedures in a step by step fashion. The Wizards use the default parameters provided, and allow you to change them as necessary to suit your work.

Wizards are available to help you with:

- setting up a Method.
- running an Analysis.

- adjusting and optimising your instrument and analysis parameters
- viewing the Results of an analysis.
- printing a Report of an analysis.

## **The Methods Manual**

The Methods Manual is an optional document that you can order with your instrument.

It describes

- the theory of atomic absorption spectrometry.
- the main features of a practical AA spectrometer.
- common standard and sample preparation methods.
- the purpose and design of common accessories.

## **The Applications Information**

The SOLAAR Software Installation CD-ROM contains an Applications Library to help you get the best from your AA instrument.

To install the Applications Library:

1. Insert the Software Installation CD-ROM in the CD drive of a PC running Windows.
2. The CD should start automatically, and display the main introduction screen.
  - if it does not start automatically, run the application `autoplay.exe` located in the root directory of the CD. Refer to the Windows Online Help system if you do not know how to do this.
3. Click on the Support Documentation option on the introduction screen, then follow the on-screen instructions.

The Applications Library includes:

- Methods, providing descriptions of various simple and complex analyses.
- Performance Sheets, providing details of the analytical performance of the iCE 3000 Series instruments
- Technical Articles, providing in-depth discussion of the Atomic Absorption technique and instrumentation.

### Technical Support

Contact details for Thermo Fisher Scientific organisations and agents world wide can be found on :

<http://www.thermoscientific.com>

**Contacting Us** There are several ways to contact Thermo Fisher Scientific

**Assistance:** For new product updates, technical and application support, and ordering information visit us on the web:  
<http://www.thermoscientific.com/ice>

**Technical Support** Contact details for Thermo Fisher Scientific organisations and agents world wide can be found on :  
<http://www.thermoscientific.com>

**Training** Training courses are available through a local Thermo Scientific sales office. Contact details can be found on <http://www.thermoscientific.com>

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## Chapter 2 **Safety in Atomic Absorption Spectrometry**

Read this Chapter carefully before installing and operating the instrument and its accessories.

The safety standards contained in this manual comply with the requirements of the Health and Safety at Work Act 1974.

Lesen sie diese seiten besonders sorgfältig vor der installation und dem gebrauch des gerätes.

Die Sicherheitsstandards, die in diesem Handbuch enthalten werden, halten sich an die Bedingungen von der «Health and Safety at Work Act 1974» als herausgegeben durch die Gesundheit und die Sicherheitsexekutive.

Lire très attentivement ce chapitre avant d'installer et d'utiliser l'instrument et ses accessoires.

Les normes de sécurités contenues dans ce manuel sont conformes aux recommandations du: «Health and Safety at Work Act 1974».

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**Introduction** The instrument and accessories described in this manual are designed to be used by properly trained personnel only.

Adjustment, maintenance and repair of equipment, which involves the removal of covers, must only be carried out by qualified Service Engineers who are aware of the hazards involved.

**Safety Precautions** For the correct and safe use of the instrument and its accessories, it is essential that the operating and service personnel follow generally accepted safety procedures in addition to the specific precautions specified in this manual. Specific warning and caution statements and symbols are included in the relevant sections of this manual.

A safety warning triangle means that when operating or exchanging parts, the correct operating steps, as described in the user documentation, must be adhered to. Non-observance can cause accidents, injuries and/or damage to the instrument and accessories.

Warning and caution statements and symbols are marked on the apparatus where appropriate. The symbols are described in the table below:



**Yellow/Black or Red/White General Warning Symbol** This symbol indicates imminent or potential hazard. The operator must refer to an explanation in the User Documentation before proceeding. ▲



**White/Black Molded Protective earth (ground) terminal** ▲



**Yellow/Black Hot Surface Warning Symbol** The “burn hazard” warning symbol indicates a hot surface. These surfaces may cause burns if touched.



**Yellow/Black Electrical Shock Warning Symbol** An electrical shock will, could or may occur: Hazardous voltages present. Handle by insulation only. Do not touch terminal points. ▲

### Other Warning Symbols

Other warning symbols may be used on accessories and should be adhered to. ▲

The covers of the instrument and accessories should only be removed by qualified, trained Thermo Fisher Scientific Service personnel.

All spare parts and consumable items used must be approved by Thermo Scientific.

Some of the chemicals used in Atomic Absorption spectrometry are corrosive and/or flammable, and samples may be radioactive, toxic or potentially infective.

Normal laboratory procedures and regulations for handling such materials should be followed. A complete risk assessment should be completed for all operations and analysis.

## Electrical Safety

All mains powered equipment is designed for operation with a fully earthed mains supply. The mains earth connection to the equipment must be connected, otherwise safety may be impaired.

Where reference is made to electrical safety, the following points apply:

- Follow the National Regulations for the country of use when fitting mains plugs. A qualified electrician should be consulted.
- If liquid is spilled on or adjacent to the instrument, immediately isolate the instrument and accessories from the mains supply.

## Equipment Cleaning and Decontamination

It is the user's responsibility to carry out appropriate cleaning and decontamination of the equipment if hazardous material is spilt on or inside the equipment.

- Cleaning and decontamination procedures are specified in the relevant sections of this manual.
- Before using any other procedures, users should check with the manufacturers that the proposed method will not damage the equipment.

## Impaired Safety Protection

Whenever Safety Protection has been impaired, the instrument and accessories must be made inoperative and secured against any unintended operation. The matter should then be referred to the Servicing authority.

- 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 not specified by the manufacturer, the Safety Protection provided by the equipment may be impaired.

## WEEE compliance

This product is required to comply with the European Union's Waste Electrical & Electronic Equipment (WEEE) Directive 2002/96/EC. It is marked with this symbol:



Thermo Fisher Scientific has contracted with one or more recycling/ disposal companies in each EU Member State, and this product should be disposed of or recycled through them. Further information on Thermo Fisher Scientific's compliance with these Directives, the recyclers in your country, and information on Thermo Fisher Scientific products which may assist the detection of substances subject to the RoHS Directive are available at [www.thermoscientific.com](http://www.thermoscientific.com).



**Einführung** Die hier beschriebenen Geräte erfordern gründlich ausgebildetes Bedienungspersonal.

Spezielle Justierungen, Wartungen und Reparaturen am geöffneten Gerät dürfen nur von autorisierten Personen durchgeführt werden.

## Allgemeine richtlinien zur sicheren handhabung des Gerätes

Für die korrekte und sichere Handhabung des Gerätes und des entsprechenden Zubehörs ist es unbedingt erforderlich, dass das Bedienungs- und Service-Personal den in der Bedienungsanleitung angegebenen richtlinien Folge leistet.

Ein Warnungsdreieck bedeutet, daß es überaus wichtig ist, die Verfahren zu verfolgen, die im Benutzerhandbuch bei der Benutzung oder Austausch der Stücke beschrieben wurden. Die Nicht-Beobachtung dieser Regeln kann Unfälle und/oder Schäden am Instrument und Zubehör verursachen.

Spezielle Vorsichts- und Warnungshinweise finden Sie der Bedienungsanleitung besonders vermerkt. Die Symbole sind im Tisch unten beschrieben:



### Gelb/Schwarz oder Rot/Weiss Allgemeines Warnungssymbol

Dieses Symbol gibt ein unmittelbar bevorstehendes Risiko oder eine potentielle Gefahr an. Der Wirtschaftsbeteiligte muß sich auf eine Erklärung in der Benutzerdokumentation beziehen, bevor er andauert ▲



### Schwarz/Weiss Masse/schutzkontakt ▲



**Gelb/Schwarz Heißes Oberflächenc Warnsymbol** Das Warnsymbol "der Brandgefahr" zeigt eine heiße Oberfläche an. Diese Oberflächen können Brände verursachen. ▲



**Gelb/Schwarz Elektrisches Symbol der Schockwarnung** Ein Wille des elektrischen Schlages, konnte oder kann auftreten: Gefährliche hochspannung. Nur an der isolierung berühren. Niemals kontakte anfassen. ▲

### Andere Warnungssymbole

Andere Warnsymbole, die möglicherweise auf Zusatzgeräten verwendet werden und sollten an gehaftet werden. ▲

Die Decken vom Instrument und die Zubehörteile sollten nur von einem Thermo Fisher Scientific Dienstingenieur herausgenommen werden.

Alle Ersatzteile und verbrauchbaren benutzten Einzelteile müssen Thermo Scientific genehmigt werden.

Einige der in der Spektroskopie zur Anwendung kommenden Chemikalien sind corrosive, leicht entzündbar, radioaktiv, infektiös oder toxisch.

Daher muß dafür Sorge getraden werden, dass die normalen Laborrichtlinien zur Handhabung dieser Chemikalien zur Anwendung kommen.

Eine komplette Risikobeurteilung sollte für alle Betriebe und Analyse abgeschlossen werden.

## **Elektrische Sicherheit**

Vom Netz gespeiste Geräte sind so entwickelt, dass eine Masseverbindung vorhanden sein muss. Diese Masseverbindung sollte vorhanden sein, da sonst die elektrische Sicherheit beeinflusst werden könnte.

## **Reinigung und Dekontamination der Geräte**

Im Hinblick auf die elektrische Sicherheit müssen die folgenden Punkte beachtet werden:

- Die elektrische Installation muß den jeweiligen Bestimmungen des Landes durch qualifiziertes Fachpersonal erfolgen.
- Verschüttete Flüssigkeiten: Das Gerät und/oder Zubehör sofort ausschalten.
- Es liegt in der direkten Verantwortung des ANWENDERS und nicht des Geräteherstellers, eine Dekontamination des Gerätes durchzuführen, falls sich toxische Substanzen im oder dem System befinden.
- Reinigungs- und Dekontaminationsarbeiten sind in der Bedienungsanleitung beschrieben.
- Bevor Sie eine Reinigungs- bzw. Dekontaminierungsmethode einsetzen, die nicht vom Hersteller empfohlen wird (vgl. Wartungshinweise dieser Bedienungsanleitung), sollten Sie zusammen mit dem Hersteller sicherstellen, dass diese Vorgehensweise das System nicht beschädigt.

## **Beeinträchtigung der Sicherheitseinstellungen.**

Immer dann, wenn eine Beeinträchtigung der Sicherheit vorliegt, muß dafür Sorge getragen werden, daß keine weitere unbefugte Bedienung des Gerätes oder des Zubehörs erfolgen kann und der autorisierte Service-Ingenieur informiert wird.

- Eine Sicherheitsbeeinträchtigung liegt z.B. dann vor, wenn nicht mehr die erwarteten Ergebnisse oder eine sichtbare Beschädigung vorliegen.
- Wenn das Gerät nicht gemäss Spezifikationen des Herstellers eingesetzt wird, könnte die Sicherheit beeinträchtigt werden.

**WEEE Konformität:** Dieses Produkt muss die EU Waste Electrical & Electronic Equipment (WEEE) Richtlinie 2002/96/EC erfüllen. Das Produkt ist durch folgendes Symbol gekennzeichnet:



Thermo Fisher Scientific hat Vereinbarungen getroffen mit Verwertungs-/ Entsorgungsanlagen in allen EU-Mitgliederstaaten und dieses Produkt muss durch diese Firmen wiederverwertet oder entsorgt werden. Mehr Informationen über die Einhaltung dieser Anweisungen durch Thermo Fisher, die Verwerter und Hinweise die Ihnen nützlich sein können, die Thermo Fisher Produkte zu identifizieren, die unter diese RoHS Anweisung fallen, finden Sie unter [www.thermoscientific.com](http://www.thermoscientific.com).

## Introduction

L'instrument et les accessoires décrits dans ce manuel sont conçus pour être utilisés uniquement par un personnel proprement qualifié.

Le réglage, la maintenance et les réparations de l'équipement, impliquant qu'un capot soit retiré, doivent être seulement réalisés par des ingénieurs de maintenance qualifiés qui connaissent les risques encourus.

## Précautions de Sécurité

Pour un usage correct et sûr de l'instrument et de ses accessoires, il est essentiel que les utilisateurs et le personnel de maintenance suivent les procédures de sécurité généralement acceptées en plus des précautions spécifiques indiquées dans ce manuel.

Les indications et les symboles spécifiques de mis en garde et de précaution sont incluses dans les sections appropriées de ce manuel.

Les indications et les symboles de mis en garde et de précaution sont marqués sur l'instrument où cela est approprié. Ces symboles sont décrits dans le tableau suivant :

Un triangle d'avertissement signifie qu'il est impératif de suivre les procédures décrites dans le manuel d'utilisateur lors de l'utilisation ou d'échange des pièces. La non-observance de ces règles peut causer des accidents et/ou des dommages à l'instrument et les accessoires.



### **Jaune/Noir ou Rouge/Blanc Symbole général d'avertissement**

Ce symbole indique un risque imminent ou un danger potentiel. L'opérateur doit se référer à une explication dans la documentation d'utilisateur avant de continuer. ▲



### **Blanc/Noir Terminaison de mise à la terre (masse) ▲**



**Jaune/Noir Symbole d'avertissement de surface brûlante** Ce symbole "de risque de brûlure" indique une surface chaude. Ces surfaces peuvent causer des brûlures si touchées. ▲



**Jaune/Noir Symbole d'avertissement de choc électrique** Un choc électrique peut ou pourrait se produire : Présence de tensions dangereuses. Manipuler uniquement par l'isolation. Ne pas toucher les extrémités. ▲

### **Autres Symboles d'avertissement**

D'autres symboles d'avertissement peuvent être utilisés sur les accessoires et doivent être respectés. ▲

Les panneaux de l'instrument et des accessoires doivent être seulement retirés par un ingénieur de maintenance proprement qualifié de Thermo Fisher Scientific.

Toutes les pièces de rechange et les articles consommables utilisés doivent être approuvés par Thermo Scientific.

Certains produits chimiques utilisés en spectrométrie sont corrosifs et/ou inflammables, et les échantillons peuvent être radioactifs, toxiques ou potentiellement contagieux. Les procédures et règlements normaux de laboratoire pour la manipulation de tels matériels doivent être observés.

Une évaluation complète des risques doit être accomplie pour toutes les opérations et analyses.

## **Sécurité Electrique**

Tous les équipements alimentés par le courant électrique sont conçus pour fonctionner avec une alimentation électrique avec prise de terre. L'équipement doit être raccordé à la terre, dans le cas contraire, la sécurité peut être compromise.

Lorsqu'il est fait référence à la sécurité électrique, les points suivants s'appliquent :

- Suivre les règlements nationaux du pays d'utilisation lors de l'installation électrique. Un électricien qualifié doit être consulté.
- Si du liquide est renversé sur ou à proximité de l'instrument, isoler immédiatement l'instrument et les accessoires de l'alimentation électrique.

## **Nettoyage et Décontamination de l'Equipement**

Il est de la responsabilité de l'utilisateur de procéder au nettoyage et à la décontamination appropriés de l'équipement si des matériaux dangereux ont été répandus sur, ou dans, l'équipement.

- Les procédures de nettoyage et de décontamination sont spécifiées dans les sections appropriées de ce manuel.
- Avant d'utiliser toutes autres procédures, les utilisateurs doivent vérifier avec le constructeur que la méthode proposée ne peut pas endommager l'équipement.

## **Altération de la Protection de Sécurité**

Si jamais les Protections de Sécurité ont été altérées, l'instrument et les accessoires doivent être rendus inopérants et protégés contre toute utilisation involontaire. La cause du problème doit alors être communiquée au Service Après Vente.

- Les Protections de Sécurité sont susceptibles d'être altérées si l'instrument ne fonctionne normalement, ou montre des dommages évidents.
- Les Protections de Sécurité peuvent être compromises si l'utilisation de l'équipement n'est pas conforme aux spécifications du fabricant.

**Conformité DEEE:** Ce produit doit être conforme à la directive européenne (2002/96/EC) des Déchets d'Équipements Électriques et Électroniques (DEEE). Il est marqué par le symbole suivant:

Thermo Fisher Scientific s'est associé avec une ou plusieurs compagnies de recyclage dans chaque état membre de l'union européenne et ce produit devrait être collecté ou recyclé par celles-ci. Davantage d'informations sur la conformité de Thermo Fisher Scientific à ces directives, les recycleurs dans votre pays et les informations sur les produits Thermo Scientific qui peuvent aider la détection des substances sujettes à la directive RoHS sont disponibles sur [www.thermoscientific.com](http://www.thermoscientific.com).

**Regulatory Compliance** The iCE 3000 Series Spectrometers and accessories are CE marked, indicating compliance with the following European Directives:

2004/108/EC Electromagnetic Compatibility Directive (EMC Directive)

2006/95/EC Electrical Equipment designed for use within certain voltage limits (Low Voltage Directive).

For details of these standards refer to the regulatory notice provided for the iCE 3000 Series spectrometers and accessories, which is reproduced here.

**Übereinstimmung mit Regularien** Alle Spektrometer der iCE 3000-Serie tragen das CE Zeichen und entsprechen damit den europäischen Regelwerken.

2004/108/EC Elektromagnetische Kompatibilität (EMC)

2006/95/EC Elektrische Geräte, die für den Einsatz innerhalb bestimmter Spannungsgrenzen konzipiert sind (Regularien zur Minimalspannung)

Weitere Angaben über die oben genannten Standards finden Sie auf der nächsten Seite im Auszug aus den Regelwerken für die Atomabsorptionsspektrometer der M-Serie.

**Conformité Normative** Tous les Spectromètres Série iCE 3000 et accessoires sont marqués CE, indiquant leur conformité avec les Directives Européennes suivantes:

2004/108/EC Directive de Compatibilité Electromagnétique (Directive EMC).

2006/95/EC Equipement Electrique conçu pour une utilisation avec des limites de tension fixées (Directive Basse Tension).

Pour plus de détails sur ces normes, se référer à la notice de conformité fournie avec les spectromètres Série iCE 3000, reproduite sur la page suivante.

## EC DECLARATION OF CONFORMITY

No. : UAA110001

The undersigned, representing the following manufacturer

<b>manufacturer or authorised representative:</b>	Thermo Electron Manufacturing Ltd
<b>address :</b>	SOLAAR House, 19 Mercers Row, Cambridge CB5 8BZ, UK

herewith declares that the product

<b>product identification :</b>	iCE 3000 Series Atomic Absorption Spectrometer Systems (3300, 3400 and 3500)
---------------------------------	---

is in conformity with the provisions of the following EC directive(s)

reference no	title
2006/95/EC	Low Voltage Directive
2004/108/EC	EMC Directive

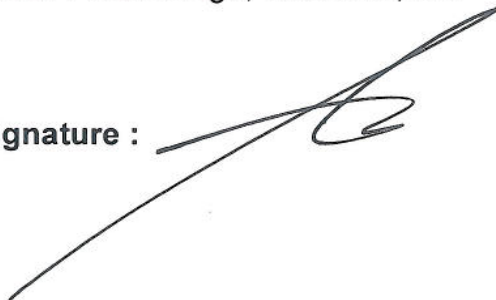
and that the standard(s) and/or technical specifications referenced overleaf have been applied.

Last two digits of the year in which the CE marking was affixed : 08

Place : Cambridge, CB5 8BZ, UK

Date : 14<sup>th</sup> January 2011

Signature :



Name : Dr Neil C. Bird

Function : R&D Director



## EC DECLARATION OF CONFORMITY

No. : UAA110001

**References of standards and/or technical specifications applied for this declaration of conformity, or parts thereof :**

**- harmonized standards :**

number	title	result
<b>Safety:</b>		
BS EN 61010-1:2001	Safety Requirements for Electrical Equipment for Measurement, Control and Laboratory Use. General Requirements.	Pass
IEC 61010-2-061:2003	Safety Requirements for Electrical Equipment for Measurement Control and Laboratory Use. Particular Requirements for Laboratory Atomic Spectrometers with Thermal Atomization and Ionization	Pass
<b>EMC:</b>		
<b>Conducted &amp; Radiated Emissions</b>		
EN61326-1:2006	Electrical Equipment for Measurement, Control and Laboratory Use. EMC Requirements. General Requirements.	Pass
EN61000-3-3	Flicker	Pass
EN61010-3-2	Harmonic Currents	Pass
<i>Methods:</i>		
CISPR11	Conducted Emissions	Pass
CISPR11	Radiated Emissions	Pass
<b>Immunity</b>		
EN61326-1:2006	Electrical Equipment for Measurement, Control and Laboratory Use. EMC Requirements. General Requirements.	Pass
<i>Methods:</i>		
IEC61000-4-3	Radiated Field Immunity	Pass
IEC61000-4-6	Conducted RF Immunity	Pass
IEC61000-4-4	Electrical Fast Transients	Pass
IEC61000-4-2	Electrostatic Discharge	Pass
IEC61000-4-5	Surges	Pass
IEC61000-4-11	Voltage Dips & Interruptions	Pass

**- other standards and/or technical specifications :**

<b>Conducted &amp; Radiated Emissions</b>		
CFR47:2008 (Part 15, Subpart B)		
<i>Methods:</i>		
ANSI C63.4:2003	Conducted Emissions	Pass
ANSI C63.4:2003	Radiated Emissions	Pass

**- other technical solutions, the details of which are included in the technical documentation or the technical construction file :**

**Other references or information required by the applicable EC directive(s):**

The documentation relating to this declaration is on file.

**Notices:**

1. About the System: Use only with Thermo Fisher Scientific approved computer and accessories
  2. About Shielded Cables: Use only shielded cables supplied by Thermo Fisher Scientific when connecting this instrument to the computer and other accessories
  3. About System Installation: Installation of the system to be performed in accordance with instructions contained in the appropriate Thermo Fisher Scientific Pre-Installation Manual.
- Compliance with the above notices is necessary to ensure that the appropriate radio frequency emissions will be maintained within the limits of the specifications referred to in this declaration.



## Hollow Cathode Lamp Hazards

**Introduction** Hollow cathode lamps present several hazards, which you should be aware of before you use them.

**Risk of Implosion** Hollow cathode lamps are filled with inert gas at pressures significantly below normal atmospheric pressure.

- If the envelope is scratched or damaged, the lamp could implode.
- Inspect your lamps regularly, and discard any that have scratches or damage to the envelope.

**High Voltage** Hollow cathode lamps are powered by high voltage power supplies.

- Ensure that the power to the lamp is turned off before you remove a lamp from the spectrometer.
- Confirm that the socket is not powered before you install a lamp.
- If the lamp becomes damaged or broken while it is fitted to the spectrometer, switch off the spectrometer and disconnect it from the mains supply before attempting to remove the lamp.

**Ultra-violet emission** Some hollow cathode lamps emit ultra-violet radiation.

- Refer to the table to identify these lamps.
- Avoid exposing your eyes or your skin to radiation from these lamps.

**Hazardous contents** The content of certain hollow cathode lamps may present health hazards if the lamp envelope is broken.

Refer to the table to identify these lamps. Refer to your local regulations for safe procedures for handling and disposing of the hazardous material.

Lamp	UV Hazard	Cathode material hazard
Aluminium		
Antimony	●	Toxic by inhalation, skin contact and ingestion. Irritant.
Arsenic	●	Toxic by inhalation and ingestion.
Barium		Toxic. Flammable on contact with water.
Beryllium	●	Toxic. Serious health damage from prolonged exposure by inhalation. Irritant to eyes, skin and respiratory system.
Bismuth	●	Harmful by ingestion.
Boron	●	Harmful by ingestion and contact with open wounds.

Lamp	UV Hazard	Cathode material hazard
Cadmium	•	Toxic.
Caesium		Highly toxic (contains lead). Flammable.
Calcium	•	Contact with water releases flammable gas. Harmful to eyes and skin by contact.
Chromium		May cause sensitisation by skin contact.
Cobalt	•	May cause sensitisation by skin contact.
Copper	•	Toxic by ingestion.
Dysprosium		Harmful by ingestion.
Erbium		Harmful by ingestion.
Europium		Contact with water releases flammable gas.
Gadolinium	•	Harmful by ingestion.
Gallium	•	May cause sensitisation by skin contact.
Germanium	•	
Gold	•	
Hafnium	•	Toxic. Mild irritant.
Holmium		Harmful by ingestion.
Indium		
Iridium	•	
Iron	•	
Lanthanum		Contact with water releases flammable gas.
Lead	•	Highly toxic.
Lithium		
Lutetium		Harmful by ingestion.
Magnesium	•	Contact with water releases flammable gas. Spontaneously flammable in air.
Manganese		Toxic. May cause sensitisation by contact.
Mercury	•	Toxic by inhalation. Cumulative. Prolonged exposure causes serious health damage.
Molybdenum		
Neodymium		Harmful by ingestion.
Nickel	•	May cause sensitisation by skin contact.
Niobium		
Osmium		Highly toxic.
Palladium	•	
Phosphorus	•	
Platinum		
Potassium	•	Spontaneously flammable in air. Reacts violently with water to release flammable gases. Causes burns on contact with skin.
Praseodymium		
Rhenium		
Rhodium		
Rubidium		Highly toxic. Flammable.
Ruthenium		
Samarium		

Lamp	UV Hazard	Cathode material hazard
Scandium		Harmful by ingestion. Irritant.
Selenium	●	Toxic by inhalation and ingestion. Cumulative. Prolonged exposure causes health damage.
Silicon	●	
Silver		
Sodium		Spontaneously flammable in air. Reacts violently with water to release flammable gas. Causes burns on contact with skin. Highly toxic (contains lead).
Strontium		Contact with water releases flammable gas.
Tantalum		
Tellurium	●	Highly toxic.
Terbium		Harmful by ingestion.
Thallium	●	Highly toxic by inhalation and ingestion. Cumulative. Prolonged exposure causes health damage.
Thorium		Radioactive. Irritant.
Thulium		
Tin	●	
Titanium		
Tungsten	●	
Uranium		Radioactive. Highly toxic by inhalation and ingestion. Cumulative. Prolonged exposure causes health damage.
Vanadium		May cause sensitisation by skin contact.
Ytterbium		Harmful by ingestion.
Yttrium		
Zinc	●	Toxic by ingestion.
Zirconium		

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# Chapter 3 Spectrometer Operation

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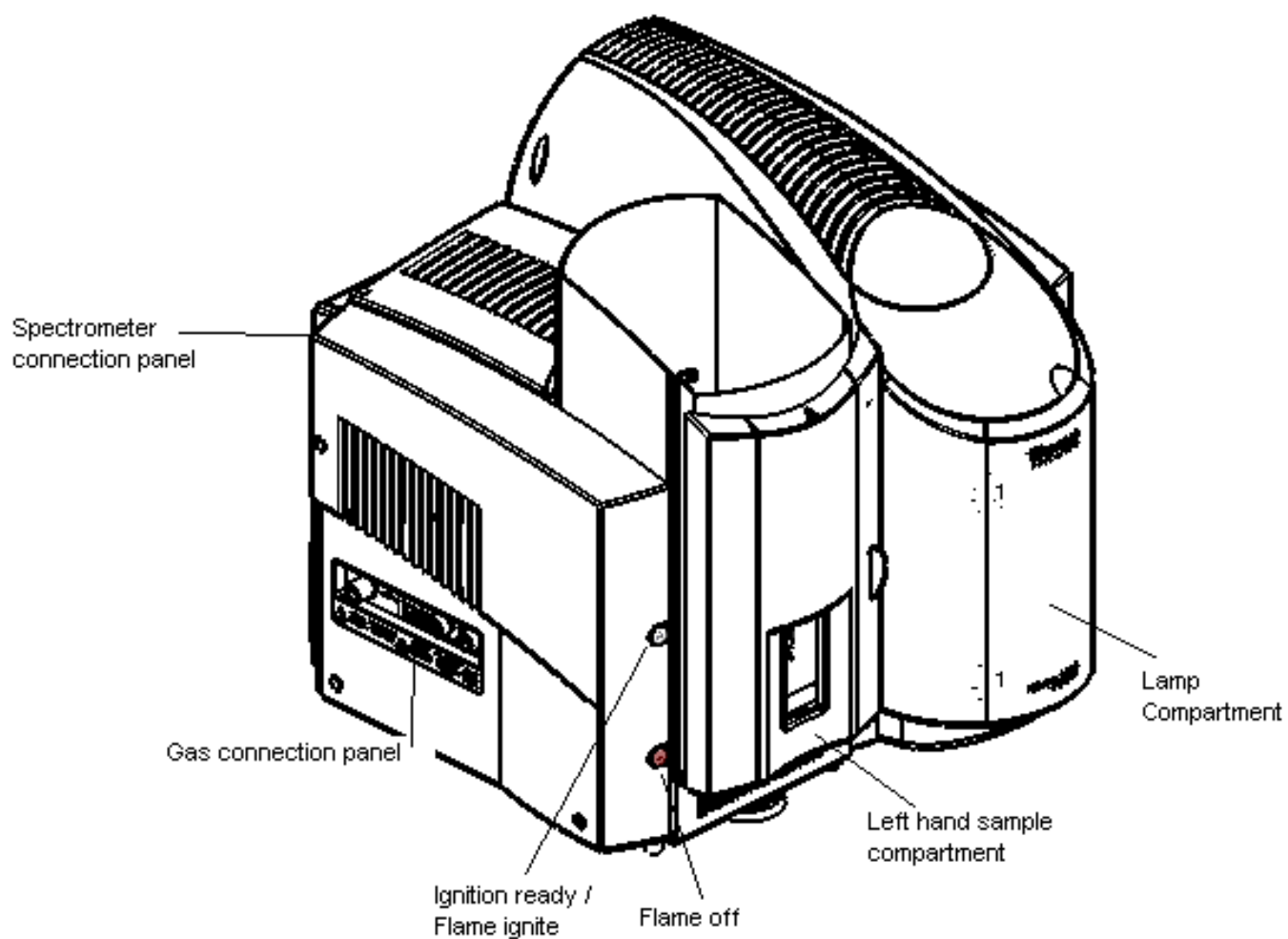
## Spectrometer Installation

**Pre-Installation** The laboratory facilities necessary to safely install and use your iCE 3000 Series Spectrometer are described in detail in the Pre-Installation manual. Confirm that these facilities are available and operating correctly before arranging for your spectrometer to be installed.

**Installation** Your spectrometer must be installed and tested by a trained Service Engineer.

**Data Station Installation** Refer to the Software Manual.

**Data Station Printer Installation** You may connect a suitable printer to the Data Station. Any printer that is supported by your Data Station operating system may be used; printer installation procedures are described in the Software Manual.



**Fig 3 - 1** ICE 3000 Series Spectrometer - General View



## Installing Hollow Cathode Lamps

**Introduction** Hollow Cathode Lamps (HCLs) are high intensity, stable light sources that emit the element specific spectral lines required for Atomic Absorption spectrometry.

You can install up to 6 Hollow Cathode Lamps in your iCE 3000 Series spectrometer.

**Safety** Refer to the section of this manual describing the hazards associated with hollow cathode lamps, and ensure that you understand the hazards involved and the precautions necessary before you install a lamp.

- Lamp types**
- Coded or uncoded lamps supplied by Thermo Fisher Scientific are recommended.
  - Most 37 mm diameter uncoded lamps supplied by third party manufacturers may be used.
  - Most 37 mm diameter coded lamps supplied by third party manufacturers may also be used, but the spectrometer will not recognise the coding, and will treat such lamps as uncoded.
    - Thermo Fisher does not guarantee that the instrument will work, nor that the published specification will be met when using such third party lamps.
  - 50 mm lamps cannot be fitted in the carousel.

**WARNING:** Certain older types of HCL are fitted with 4 base pins. Do not use these lamps with your iCE 3000 Series spectrometer; they could cause severe damage to the instrument. ▲

- Lamp installation**
1. Turn on the system and start the Data Station software.
  2. Open the lamp carousel door.
  3. Use the Lamp commands in the system software to rotate the carousel to bring an empty lamp position to the front, and confirm from the software Lamp Status display that the power to the lamp position is OFF.
  4. Orientate the lamp correctly, then install it by pushing it firmly into the socket, so that it is held in place by the Lamp Clip.
  5. Confirm that the software lamp status displays are updated correctly.

## Lamp removal

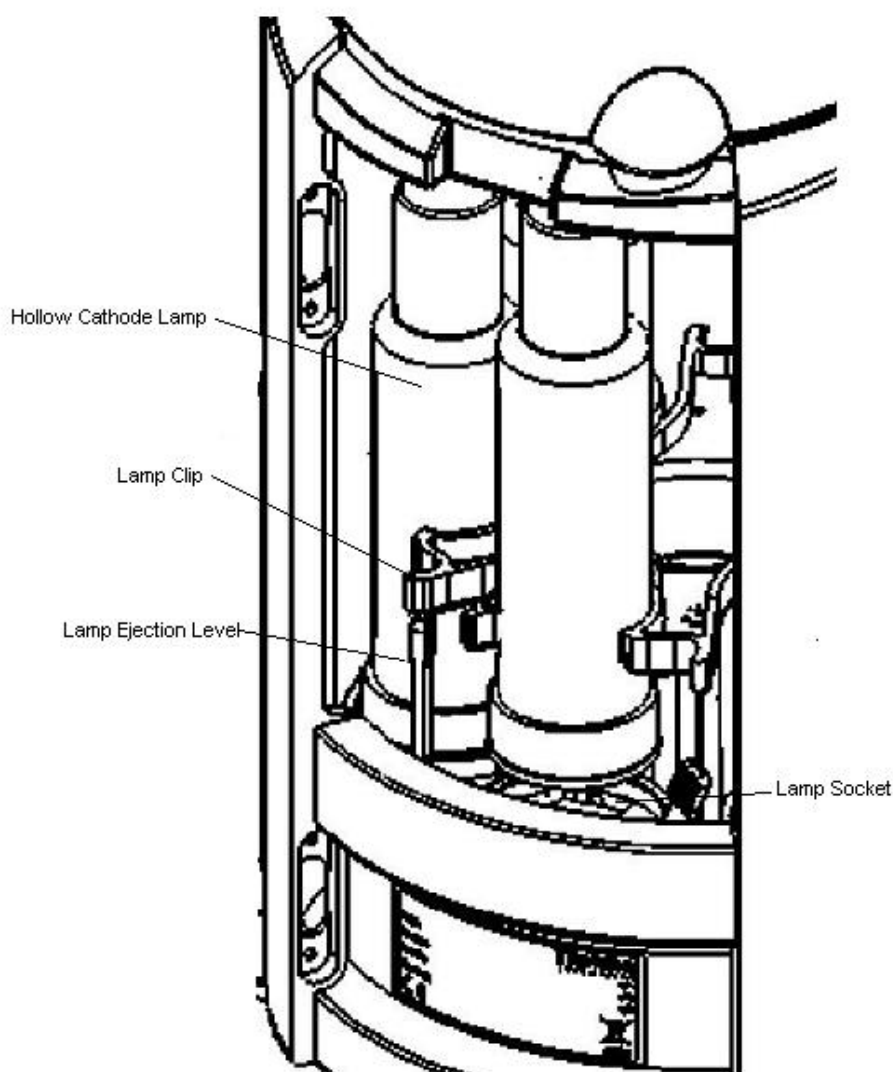
1. Turn on the system and start the Data Station software if required.
2. Open the lamp carousel door, and use the software commands to rotate the carousel to bring the lamp to the front.

**NOTE:** If, and only if, the spectrometer is not turned on, you can carefully rotate the carousel by hand. ▲

3. Confirm from the software lamp status display that the power to the lamp is OFF.
4. Press the lamp ejection lever downward to eject lamp. Release lamp from clip and remove.

## Lamp alignment

The iCE 3000 Series spectrometers provide automatic lamp alignment facilities, with manual overrides if required. You can access these facilities from the Lamp page of the Data Station (Edit, Lamps...).



**Fig 3 - 2** Lamp Compartment - General view

# The Right Hand Sample Compartment Universal Accessory Mount

**Introduction** This is a Universal Mount for fitting several accessories in the Right Hand Sample Compartment of the iCE 3400 / 3500 systems.

- If a Graphite Furnace on a Tilt Mount is fitted in the RH Sample Compartment, it can be tilted forward to allow the Universal Mount to be fitted.
- If you want to use a Graphite Furnace and other accessories on the Universal Mount in the Right Hand Sample Compartment, we strongly recommend that you use the Tilt Mount - if a Graphite Furnace on a Fixed Mount is fitted, the Furnace and the mount must be removed before the Universal Mount can be fitted.

**Accessories** Please refer to the individual accessory pages for details of the procedures for attaching the accessories to the Universal Mount.

**Installation** Fit the accessory to the Universal Mount before installing the assembly in the Right Hand Sample Compartment.

## To fit the Universal Mount and Accessory

1. Tilt the Graphite Furnace forward on the Tilt Mount or remove the Graphite Furnace on its Fixed Mount.
2. Refer to the figure and place the Universal Mount assembly in the Right Hand Sample Compartment, locating the Adjustment Screws on the Sample Compartment Mounting Pads.
3. Use the supplied tool to push the Spring Retainer into the Location Plate, then give it one quarter turn to lock the assembly into position.

Reverse this procedure to remove the assembly.

**Alignment** Accessories fitted to the Universal Mount normally require alignment with the Spectrometer optical system. However, once the alignment has been set up and locked, the Universal Mount and accessory assembly can be removed and replaced without disturbing the alignment.

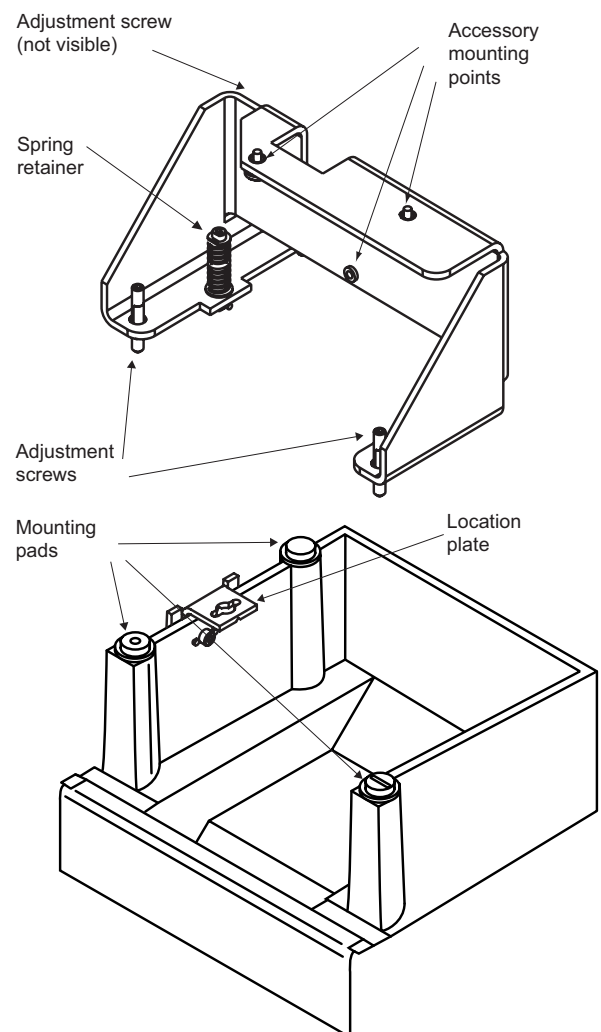
## To align an accessory fitted to the Universal Mount

1. Before fitting the Universal Mount and accessory assembly, you must:
  - ensure that the right hand Sample Compartment optical path is clear of obstruction.

## Spectrometer Operation

### The Right Hand Sample Compartment Universal Accessory Mount

- fit a suitable hollow cathode lamp.
  - use the system control software to set up the optical system in the RH Sample Compartment. Do not select any form of background correction.
  - use the system control software to display the live absorbance signal from the RH Sample Compartment.
2. Fit the Universal Mount and accessory assembly.
  3. If necessary, fit a suitable absorption cell in the accessory.
  4. Use a piece of white card to locate the optical beam in the Sample Compartment, and adjust the position of the assembly using the Adjustment Screws until the light beam passes through the accessory.
    - You will find that the long handled, ball ended Allen key tool supplied will be helpful when adjusting the rear Adjustment Screw.
  5. Make fine adjustments to the position of the assembly to obtain a minimum in the displayed absorbance signal.



**Fig 3 - 3.** The Right hand sample compartment with Universal Mount

# The Left Hand Sample Compartment Universal Accessory Mount

**Introduction** This is a Universal Mount for fitting several accessories in the Left Hand Sample Compartment of the iCE 3300 / 3500 systems.

**Accessories** Please refer to the individual accessory pages for details of the procedures for attaching the accessories to the Universal Mount.

**Installation** The Accessory mount is fitted in the LH sample compartment. You should first refer to the relevant section of this manual, and fit the required accessory to the Universal Mount.

## **To fit the LH Compartment Universal Mount**

1. If necessary, use the appropriate Software commands to move the Burner to the Parked position.
2. If necessary, remove the Burner Head as described in the flame section of this manual.
3. Orientate the Mount correctly, then engage the support brackets with the Accessory Support Bar at the front of the Sample Compartment.
4. Tilt the Mount towards the rear of the Sample Compartment, until the Rear Adjustment Screw rests securely against the rear wall.

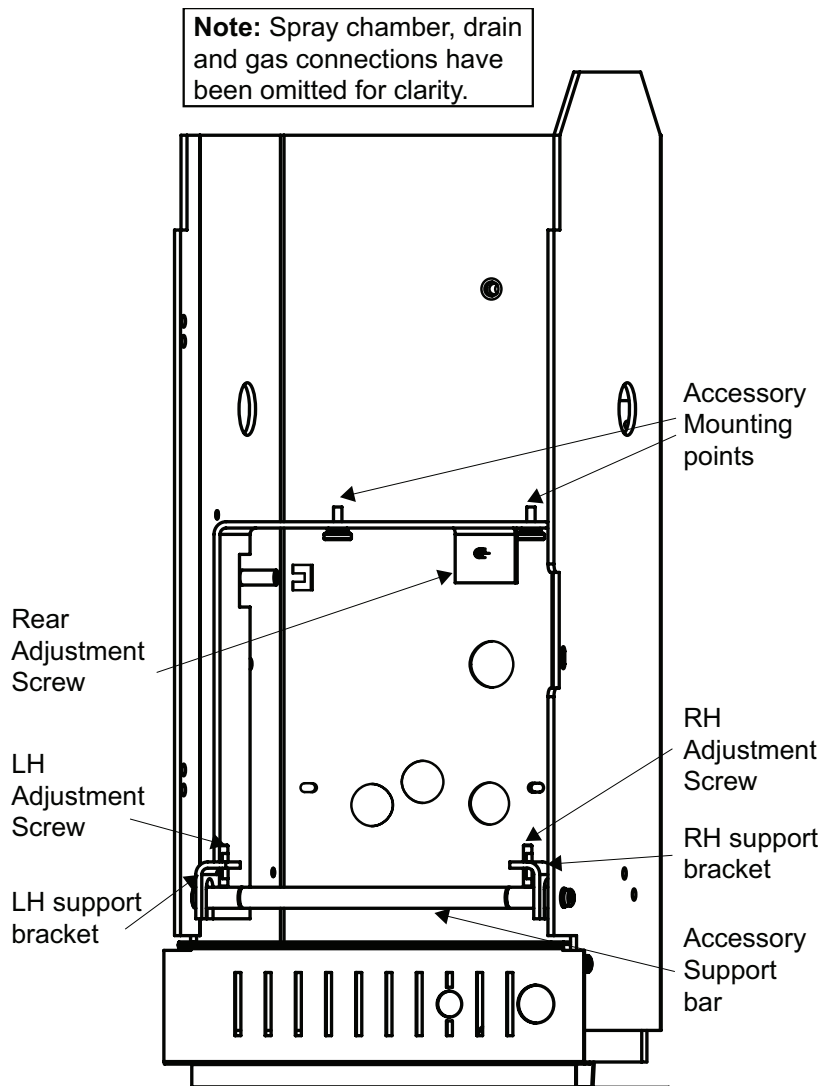
Reverse this procedure to remove the assembly.

**Alignment** Accessories fitted to the Accessory Mount should be aligned with the optical system of the Spectrometer before the accessory is used. When the alignment has been completed, it will be possible to remove and replace the Mount and accessory without re-alignment.

## **To align an accessory fitted to the Universal Mount**

1. Before fitting the Accessory Mount and accessory assembly, you must:
  - ensure that the LH Sample Compartment optical path is clear of obstruction.
  - fit a suitable hollow cathode lamp.
  - use the appropriate Software commands to set up the optical system in the LH Sample Compartment. Do not select any form of background correction.
  - use the Software to display the live absorbance signal from the LH Sample Compartment.
2. Fit the Accessory Mount and accessory assembly into the operating position.

3. If necessary, fit a suitable absorption cell in the accessory.
4. Use a piece of white card to locate the optical beam in the Sample Compartment, and adjust the position of the assembly using the Adjustment Screws until the light beam passes through the accessory.
  - You will need the 3 mm ball ended Allen key supplied to adjust the screws.
5. Make fine adjustments to the position of the assembly to obtain a minimum in the displayed absorbance signal.



**Fig 3 - 4.** Left hand sample compartment of a flame atomizer with universal mount

**NOTE:** Spray chamber, drain and gas connections have been omitted for clarity. ▲

## The iSQ Module

### Introduction

This module allows Intelligent Spectrometer Qualification (iSQ) tests to be run by the SOLAAR Software, to confirm that your spectrometer is operating correctly. The iSQ Module provides a set of certified traceable Neutral Density and Polaroid Filters in a motorised filter mount that are used to automatically measure the performance of your spectrometer.

The iSQ package consists of :

- the iSQ Module
- a calcium/magnesium Data-coded Hollow Cathode Lamp
- support and accessory brackets
- a RH Accessory Mount or Universal Accessory Mount
- an in-line power supply unit
- a serial data lead

### Safety

Observe electrical safety precautions as described in the safety section of this manual.

### Assembly

**To prepare the module for use:**

1. Use the two supplied screws to fasten the iSQ module to the support bracket, as shown in the **Fig 3.5 / 3.6** overleaf.
2. Attach the assembly to the relevant Mount using the accessory mounting points.
3. Connect one end of the 9-way data lead to the data socket on the iSQ module and tighten the locking screws.
4. Fit the round plug from the power supply in the power socket on the iSQ module. Connect the mains lead to the power supply.

### Installation

**To install the iSQ module in the spectrometer refer to the relevant sections of this manual, then:**

1. If a graphite furnace is fitted to the system do the following:
  - a. **iCE 3300 system**  
Remove the GFS33 Furnace and Autosampler Assembly
  - b. **iCE 3400 / 3500 system**  
If the furnace head is fitted on a tilt mount, tilt the furnace head forward out of the sample compartment.  
  
If the furnace head is fitted on a fixed mount, remove the furnace and fixed mount from the sample compartment.

2. Fit the Accessory Mount and iSQ Module assembly in the relevant compartment.
3. Connect the free end of the data lead to a free accessory port on the spectrometer connection panel.
4. Connect the mains lead to a suitable power outlet.
5. Switch on the spectrometer and then the iSQ module power supply. Check that the green LED on the power supply is illuminated.
  - During a normal start, the red LED 3 on the iSQ module flashes and LED's 1 and 2 are extinguished.
  - Other LED indications are :

LED 1 lit	Flash checksum test failed
LED 2 lit	RAM test failed
LED 3 lit continuously	Fatal error has occurred

## **Alignment and Operation**

The iSQ Wizard in the SOLAAR Data Station software provides facilities for aligning the iSQ module. When the iSQ Module has been correctly installed, you can carry out the alignment and the suite of iSQ Test procedures by running the iSQ Wizard. Please refer to the SOLAAR On-line Help system and SOLAAR Software manual to learn how to run a SOLAAR Wizard.

Ensure an optical set up operation has been successfully carried out prior to iSQ module installation.

### **To align the iSQ module and carry out the iSQ tests:**

1. Turn on the spectrometer, and turn on the power to the iSQ module.
2. Open the shutter of the iSQ module.
3. Start the SOLAAR Data Station software.
4. Start the iSQ Wizard, and follow the on-screen instructions until you reach the 'Install and Align Lamps' page.
5. Install the Ca/Mg lamp supplied with the iSQ module, and use the commands on the Wizard to perform an optical setup operation.
6. Refer to the RH Accessory Mount section of this manual, and carry out the alignment procedure described.
  - if the assembly is a long way out of alignment, it may be necessary to remove the iSQ module and Universal mount assembly to allow the initial optical setup operation to be completed successfully. Once the optical setup operation has been completed successfully, re-fit the assembly and proceed with the alignment procedure.
7. Continue to follow the Wizard instructions to complete the iSQ Tests sequence.



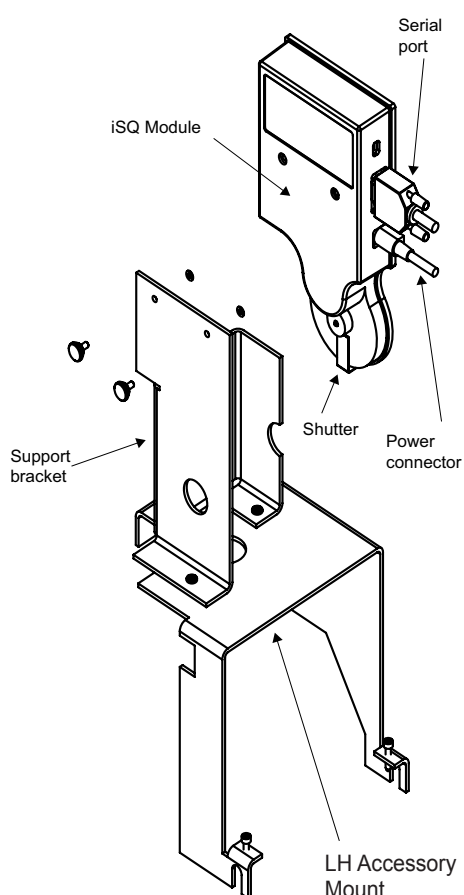
## Maintenance

It is strongly recommended that the Hollow Cathode Lamps supplied with the iSQ Module should be used for iSQ validation purposes only and not for routine analysis. The iSQ Test software automatically monitors and records lamp usage in mA.hours, provided that the lamp serial numbers are registered in the Lamp database.

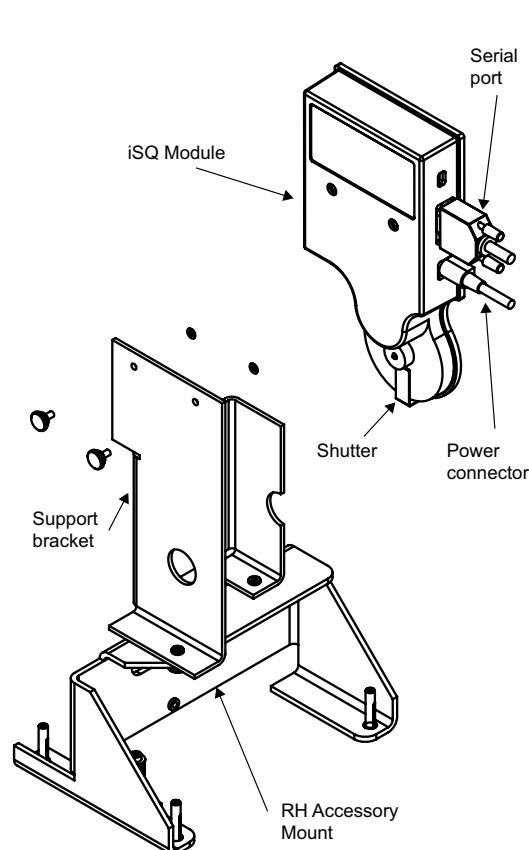
Thermo Fisher Scientific Hollow Cathode Lamps are guaranteed to emit spectra for a period of 24 months from date of dispatch or 5000 milliampere hours usage, whichever ever occurs first. If either of these limits are exceeded, a new Hollow Cathode Lamp should be obtained.

**To ensure that the accessory operates correctly at all times, the following guidelines must be followed:**

1. The iSQ module is designed to ensure reliable operation and must only be serviced by Thermo Fisher Scientific. If the module needs repair, it must be returned to Thermo Fisher Scientific, via your local sales office.
2. The iSQ module is a high precision optical instrument, and must be handled and stored carefully. When the module is not in use, the shutter must be placed over the measurement aperture in order to prevent dust falling on the filters. The module should be stored in its original packing material to prevent damage to the delicate operating parts.



**Fig 3 - 5.** iSQ Module Assembly iCE 3300 System



**Fig 3 - 6.** iSQ Module Assembly iCE 3400 / 3500 Systems

# Spectrometer Maintenance

## Introduction

To ensure that your instrument operates safely and reliably, you should regularly carry out routine maintenance.

Routine maintenance is mainly concerned with keeping the instrument clean.

## Instrument Cleaning

**WARNING:** The iCE 3000 Series instrument covers are made of ABS plastic material, which can be damaged by strong solvents and concentrated acids.▲

- Any spillage on the external covers or within the kitchen areas should be cleaned up immediately, using appropriate safety protection if necessary.
- Stains and marks on the covers should be removed with a soft cloth moistened with dilute detergent solution. Do NOT use any type of solvent based cleaners.

## Spectrometer Status Indicators

When the system starts without error a pattern will move across the LEDs for a second, and stop with the Standby LED flashing rapidly (~5Hz) and all other LEDs off. Any other pattern denotes a failure as shown in the table below. An X denotes that the LED can be in either state.

Standby	FC3	FC2	FC1	Halt	
On*	Off	Off	Off	Off	OK
x	Off	Off	On	Off	Boot Checksum Failure
x	Off	On	Off	Off	Ram Failure
x	Off	On	On	Off	Flash Checksum Failure
x	On	Off	Off	Off	Timer Failure
x	On	Off	On	Off	RS232C Crystal Failure
x	x	x	x	On	Processor Halt

\* When connected to a data station the status LED will instead flash slowly, once a second, otherwise it will flash rapidly, 5 times a second.

- The flash checksum failure may be corrected by downloading the spectrometer software, as described in the Software Manual. The other errors and the display of any code not shown in the table will require service attention.

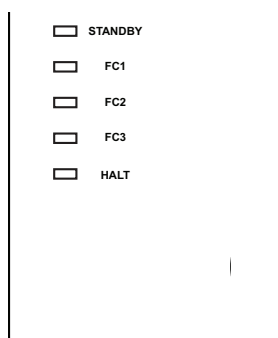


Fig 3 - 7. Connection panel and Status Indicators

## **Deuterium Lamp iCE 3300**

The Deuterium Lamp (D2) is a consumable part and will eventually reach the end of its useful life. You should suspect this if:

- The lamp fails to strike
- Your zero absorbance baseline drifts when Quadline Background Correction is selected.
- You get repeated warnings that the intensity of the Deuterium Lamp is low.

If your Deuterium Lamp has reached the end of its life, you should contact your local service engineer. On the iCE 3300, the deuterium lamp must be replaced by a service engineer.

## **Deuterium Lamp Alignment iCE 3400 / 3500**

The Deuterium (D2) lamp is located under the Deuterium Lamp Cover, on the top of the Spectrometer, behind the Lamp compartment.

The D2 Lamp should not normally require routine alignment. However, if you have replaced the lamp, or if you suspect that the Quadline Background Correction accuracy is poor, you should align the lamp.

### **To align the Deuterium Lamp:**

1. Install a suitable hollow cathode lamp.
2. Ensure that Quadline background correction is selected, and perform an Optical Setup.
  - Choose an element whose principal line is between 350 - 200 nm. spectrometer. The D2 Energy Bar Graph should be live.
3. Use the system Software to display the optical status of the spectrometer. The D2 Energy Bar Graph should be live.
4. Open the Deuterium Lamp cover. Refer to the figure and locate the Deuterium Lamp adjustment controls.
5. Adjust the D2 Lamp controls to obtain the maximum D2 Energy shown on the bar graph.
  - You can make the final adjustment to give a maximum displayed absorbance value.
  - If the D2 Energy Bar Graph goes off scale, use the Auto Zero command to bring it back to the centre of the scale.
6. Perform a final Optical Setup, and confirm that the Optical Setup is completed correctly, and that no errors or warnings are displayed.

### **Deuterium Lamp Replacement**

The Deuterium Lamp is a consumable item and will eventually reach the end of its useful life. You should suspect this if:

- The lamp fails to strike
- Your zero absorbance baseline drifts when Quadline Background Correction is selected.
- You get repeated warnings that the intensity of the Deuterium Lamp is low.

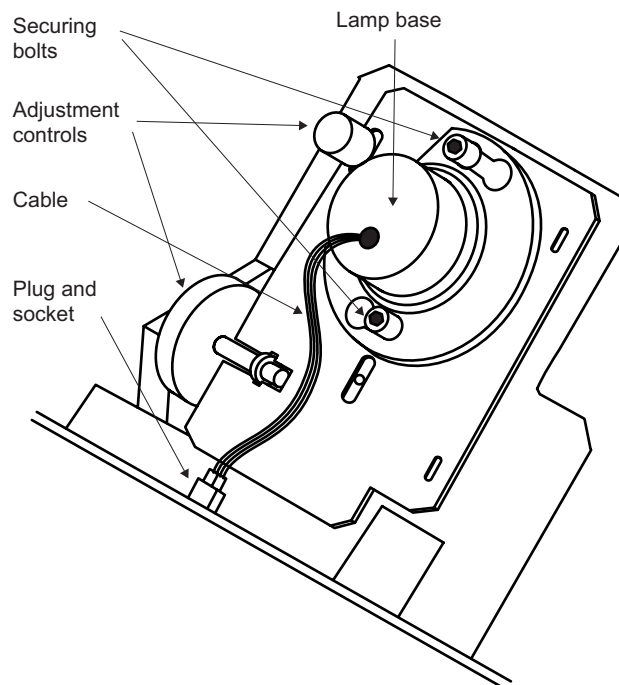
On the iCE 3400 and 3500, if your Deuterium Lamp has reached the end of its life, you can replace it yourself.

**To replace your Deuterium Lamp:**

1. Ensure that the Spectrometer is disconnected from the mains electricity supply, and has been switched off for at least 15 minutes, to allow the lamp to cool.
2. Open the Deuterium Lamp Cover.
3. Refer to the figure, and locate the Lamp.
4. Disconnect the Lamp Cable.
5. Slacken the Lamp Securing Bolts with a suitable Allen key.
6. Rotate the Lamp slightly anti-clockwise, then withdraw it from the mount.
7. Remove the new Lamp from its packaging.

**WARNING:** Be very careful not to touch the quartz envelope of the lamp. ▲

8. Fit the new Lamp into the mount, ensuring that the flat on the base is to your left.
9. Rotate it slightly clockwise, then tighten the Lamp Securing Bolts.
10. Connect the Lamp Cable to the socket.
11. Align the new Lamp as described above.



**Fig 3 - 8.** Deuterium Lamp Assembly iCE 3400 / 3500

**Spares and Consumables**

Deuterium Lamp

9423 420 30004

## Spectrometer fuse replacement

The iCE 3000 Series spectrometers have twin mains fuses in the power supply module. If the instrument does not appear to turn on when the mains switch is pressed, check these fuses.

### To replace the fuse:

1. Locate the combined power inlet connector and fuse holder on the power supply unit.
2. Disconnect the spectrometer from the mains and wait 60 seconds for components to discharge.
3. Pull out the fuse holder tray, and remove the two fuses.
4. Fit new fuses, type F5A HRC 250V, and push the fuse holder tray back into place.
5. Re-connect the mains supply.

**WARNING:** If the new fuse fails immediately, there is a serious fault in the power supply. Isolate the instrument until it can be rectified by a Service Engineer. ▲

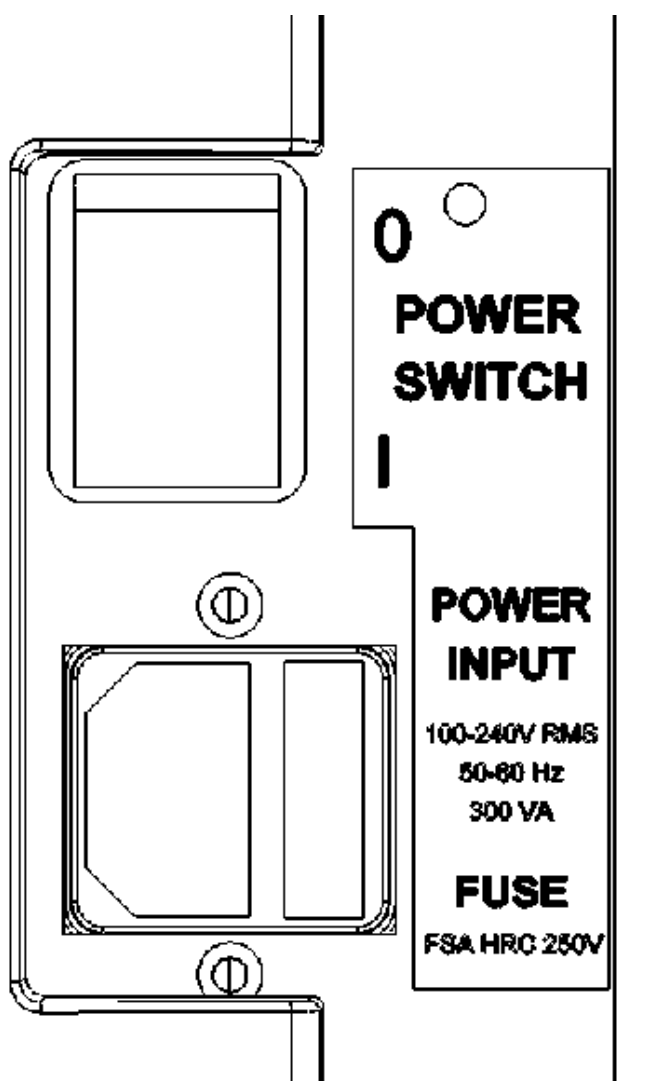


Fig 3 - 9. Mains Inlet and Fuse (upper rear right corner of spectrometer)

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## Chapter 4 Flame Operation



**CAUTION:** Flame AAS involves the use of large quantities of inflammable, explosive and toxic gases, and high temperature flames. To minimise the hazards involved, you must read and understand the contents of this section before using the equipment. ▲



**CAUTION:** A flame AAS instrument must never be left unattended while the flame is burning. ▲

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## Flame Safety

- Flames** Nitrous oxide supported flames emit intense radiation which can damage your eyes if viewed directly.
- Nitrous oxide supported flames burn at a high temperature, and emit large amounts of heat, which can cause serious injury.
- Nitrous oxide/acetylene flames can deposit carbon along the edges of the burner slot, which can build up and partially block the slot, causing a serious hazard.
- Carbon build up can be reduced by ensuring that the burner is clean, and by warming the burner up with a fuel lean nitrous oxide flame for at least 10 minutes before use.
  - If carbon deposits appear, the flame must be extinguished immediately and the deposits removed.

- Flashbacks** In the unlikely event of a flashback, a small explosion occurs in the spray chamber, rupturing the over-pressure disc at the rear of the spray chamber. If a flashback occurs disconnect all gas and power lines and contact your local service engineer. A list of Thermo Fisher Scientific organisations and agents can be found on <http://www.thermoscientific.com>. A Flashback Report form must be completed and returned to the address on the form.
- The normal operating sound level of the spectrometer will not exceed 85 dBA. In the event of a flashback the sound level will not exceed 140 dBA.

- Perchloric acid** Aspiration of solutions of perchloric acid and metal perchlorates into a nitrous oxide supported flame can increase the risk of explosion or flashback; consequently we do not recommend the use of perchloric acid in sample preparation for nitrous oxide supported flame analyses. If the use of perchloric acid is essential, you can minimise the hazards if you:
- ensure that the burner is kept clean, and do not allow deposits to accumulate around the slot.
  - reduce the perchlorate ion concentration as much as possible before aspirating the solution.
  - aspirate the solution for the minimum time necessary.
  - flush the spray chamber thoroughly with deionised water between measurements.
  - never allow the spray chamber to run dry.
  - never allow perchlorate solutions to come into contact with organic

- solvents. If organic solvents have previously been aspirated, ensure that all traces have been removed from the spray chamber before aspirating perchlorate solutions.
- ensure that the acetylene cylinder is changed when the cylinder pressure drops to 7 bar (100 psi), to prevent acetone carryover.
- thoroughly flush the spray chamber, and empty and refill the drain trap with clean water at the end of the analysis.

## Silver, gold and copper samples

Certain elements, notably Ag, Au and Cu can form unstable acetylides, increasing the risk of explosion or flashback.

To minimise these hazards when aspirating solutions containing these elements, you should:

- ensure that the burner is kept clean, and do not allow deposits to accumulate around the slot.
- reduce the metal concentrations as much as possible before aspirating the solutions.
- flush the spray chamber thoroughly with deionised water between measurements.
- never allow the spray chamber to run dry.
- thoroughly flush the spray chamber, and empty and refill the drain trap with clean water at the end of the analysis.

## Organic solvents

The use of organic solvents in flame AAS is an inherently hazardous procedure. To protect yourself, we recommend that you should carry out a comprehensive Risk Assessment before performing the analysis. As a minimum, you should:

- establish that the solvent you propose to use has suitable characteristics.
  - Avoid halogenated solvents as these can create toxic fumes,
  - Avoid solvents with low flash points as they have a potential explosive risk,
  - Avoid solvents with low specific gravity (generally lower than 0.75) as they can breach the seals within the nebulizer and sample chamber
  - Avoid unsaturated hydrocarbons, such as xylene and toluene, as these have poor burning characteristics
- As a minimum, confirm that the hazards associated with the flashpoint, combustion products, volatility and toxicity of the solvents are acceptable.
- use the minimum possible quantity of solvent in the vicinity of the spectrometer.
- ensure that any aqueous solution in the drain trap is removed and replaced with organic solvent before starting the analysis.
- ensure that the organic solvent is removed from the drain trap and replaced with clean water as soon as the analysis is finished.

If you wish to use organic solvents on a regular basis, we recommend that you have the Solvent Resistant Flame Kit (part number 9423 420 31051) fitted to your instrument.

**WARNING:** Many common laboratory solvents are unsuitable for use in flame AAS. Halogenated solvents (chloroform, Freons) produce large quantities of toxic gases when burned; solvents with low flash points (ethers, light petroleum spirit) present too great a flammability hazard to be safely used, and unsaturated aromatic hydrocarbons (toluene, xylene) do not have suitable burning characteristics. ▲

## Gases

Always follow the Safety Guidelines provided by the Gas Supplier, such as the 'Gas Data and Safety Sheet' issued by the British Oxygen Company for specific gases.

Ensure that all gas lines, fittings, etc., are free from oil or grease contamination.

Check regularly for leaks in all gas lines and fittings.

Fit gas cylinders with appropriate pressure reducing regulators. We strongly recommend that acetylene gas supplies should also be fitted with flash back arrestors.

### Acetylene

- is highly inflammable and forms an explosive mixture with air.
- has a strong garlic-like odour caused by inherently present impurities.
- if supplied or used above 0.62 bar (9 psi) in the United Kingdom, the Health and Safety Executive of H.M. Factory Inspectorate must be informed.
- forms explosive compounds with copper and silver metal. The following materials must not be used for parts that will come into contact with the gas:
  - pure copper metal
  - copper alloys with a copper content >70%
  - copper alloys with a copper content <70%, if used as filters or sieves
  - silver and silver alloys, except where used as brazing materials. Silver alloys used as a braze must contain <43% of silver and <21% of copper. The width of the braze gap exposed to the acetylene gas must be <0.3mm.
- is supplied in cylinders dissolved in acetone
  - as the cylinder pressure falls, acetone carryover increases. The cylinder should be replaced when the internal pressure drops to below 6.9 bar (100 psi).

**WARNING:** Some specialist grades of acetylene may be supplied in solvents other than acetone. The iCE 3000 Series gas control system has not been designed to withstand carryover of these solvents, which may cause severe damage to the instrument. ▲

## Nitrous oxide

- is an anaesthetic, and will cause drowsiness, unconsciousness and eventual death if inhaled.
- is a powerful supporter of combustion, and allows many materials to ignite and burn more readily than they would in air.

## Flame Sample Compartment Door

The door to the Flame Sample Compartment is an important part of the Safety features of your instrument. The door must be closed when lighting a flame, and during normal operation.

**WARNING:** The door must always be closed when a nitrous oxide supported flame is burning. ▲

You can open the door to make adjustments to the burner position while an air supported flame is burning.

## Fume Extraction

All flames produce large quantities of heat and toxic combustion products. These must be removed by a suitable fume extraction system. Specifications of a suitable extraction system are provided in the Pre-Installation Manual.

**WARNING:** With the flame running (and for some minutes after it is turned off) the metal fume extraction chimney may reach elevated temperatures. Take sensible precautions to avoid touching it. ▲

## Drain

- The position and routing of the internal spectrometer drain tube and liquid trap are critical to the safe operation of the flame system. They must not be modified in any way.
- The spray chamber drain contains a ball valve sealing device, which must not be modified, removed or re-sited.
- The drain must discharge to a low level waste system capable of handling acidic solutions, or a suitable, wide necked plastic container. Do not use a glass or a narrow necked plastic waste container.
- The drain extension tube must provide a free-flowing outlet from the instrument drain, without kinks or obstructions. The lower end must always be above the liquid level in the waste container.
- Empty and clean the waste container regularly. If you have used an organic solvent, do not allow the solvent to accumulate in the waste container, as vapor may cause a fire hazard.

## Setting up the Flame System

### Introduction

The Flame System consists of:

- the fuel and oxidant gas supplies and connectors.
- the Spectrometer automatic gas control system.
- the Nebuliser and Impact Bead.
- the Spray Chamber.
- the Spray Chamber Drain assembly.

### Safety

Read and understand the Flame Safety sections in this manual.

Before using the flame system, refer to the Flame System Maintenance section below:

- inspect and, if necessary, replace the Over-Pressure Disc at the rear of the Spray Chamber. (A small dentist's mirror is provided to help you make this inspection).
- inspect and, if necessary, clean the Burner.
- inspect and, if necessary, clean the Spray Chamber and Nebuliser.
- confirm that the fume extraction system is switched on and is operating correctly.
- check that the Spray Chamber Drain is clear, and that it discharges freely into a suitable receptacle.
- set all gases to the correct inlet pressures.
- perform a Gas Leak Test.

### Sample Uptake capillary tube

- For normal use, fit a 250 mm length of 0.5 mm ID sample uptake tubing to the nebuliser.
- Use a longer length, and/or 0.4 mm ID tube to reduce the uptake rate and/or the analytical sensitivity.

### Gas Supply inlet pressures

Set the gas supply regulators to deliver the gases at the spectrometer inlet ports at the following pressures:

### Support gases

- Compressed air 2.00-2.07 bar (29-30 psi)
- Nitrous oxide 2.70-2.75 bar (39-40 psi)

**WARNING:** Never attempt to use oxygen as a support gas. ▲

### Fuel gases

- Acetylene 0.57-0.62 bar (8.5-9 psi)

**WARNING:** Never allow the acetylene pressure to exceed 9 psi. ▲

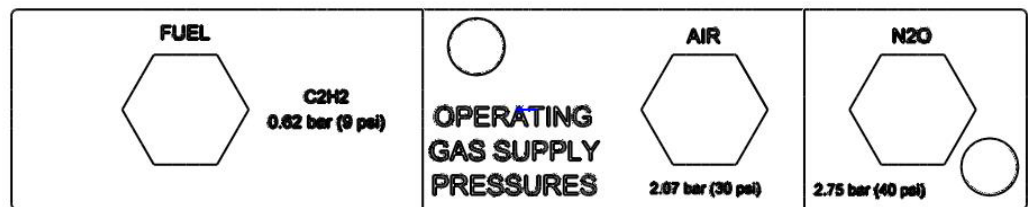


Fig 4 - 1. Spectrometer gas connection panel

**WARNING:** the gas pressures written on the gas box are maximum pressures. ▲

### Gas connections

Three Gas Inlet connections are provided on the Spectrometer, and are shown in the figure.

**WARNING:** Before disconnecting any gases from the Spectrometer, ensure that the gas supply is shut off at source, and the gas lines are vented. ▲

**WARNING:** After connecting the gas supplies to the Spectrometer, always carry out a Gas Leak Test, and rectify any leaks before using the instrument. Gas leak tests should be carried out at regular intervals thereafter ▲

### Air/acetylene flame

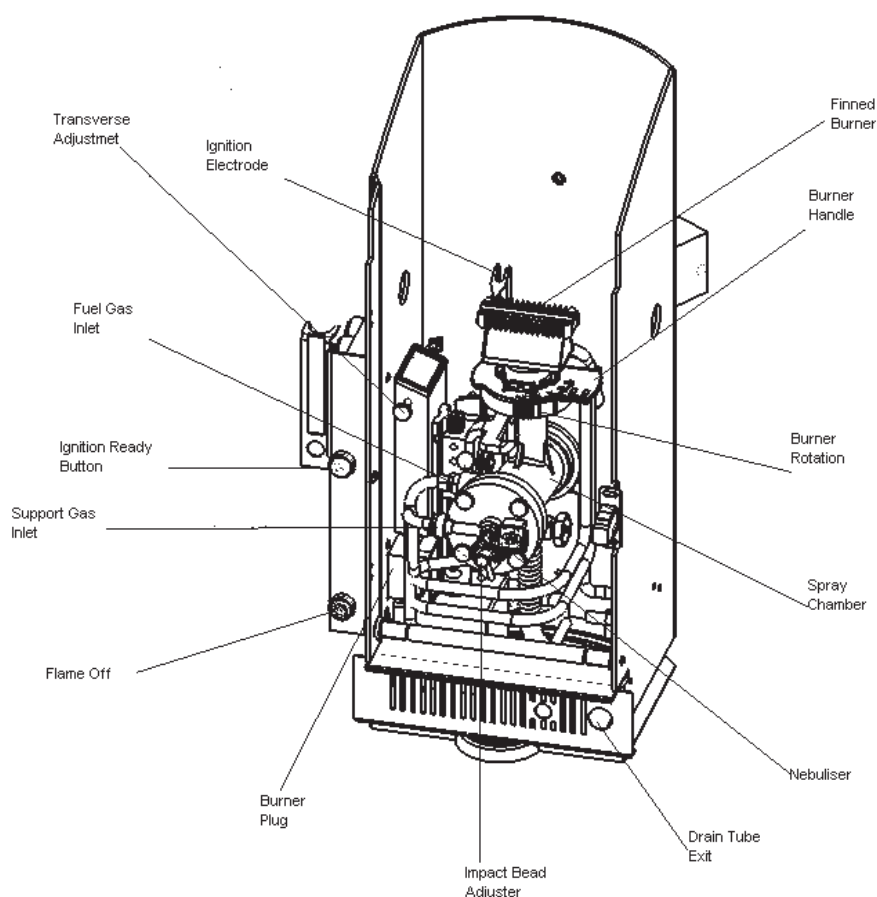
- connect the AIR supply to the connector marked AIR, using the CLEAR hose supplied.
- connect the ACETYLENE supply to the connector marked FUEL, using the RED hose supplied.

### Nitrous oxide/acetylene flame

- connect the AIR supply to the connector marked AIR, using the CLEAR hose supplied.
- connect the ACETYLENE supply to the connector marked FUEL, using the RED hose supplied.
- connect the NITROUS OXIDE supply to the connector marked N<sub>2</sub>O, using the BLUE hose supplied.

**Burners** Two types of burner are available for your iCE 3000 Series spectrometer:

- 50 mm slot Universal Finned Titanium Burner
  - suitable for general purpose use with all flame types
- 100 mm slot Titanium Burner
  - suitable for air/acetylene flames only. Sensitivity for elements measured with this flame will be improved compared to the Universal Finned Titanium Burner.



**Fig 4 - 2.** Burner Compartment - general view

**WARNING:** Inspect burner handle prior to use and do not use if locking mechanism is damaged. ▲

**To install a Burner**

Refer to the figure and:

1. Open the Left Hand Sample Compartment door.
2. Orientate the Burner so that the Ignition Electrode is to the rear of the Burner.
3. Fit the Burner to the Spray Chamber stem, and push firmly home.
  - take care to avoid damaging the O-ring seals.
4. Fit the Burner Plug to the Burner Socket located on the lower left panel of the Sample Compartment.

### To remove a Burner

1. Ensure that the flame has been extinguished and that the Burner has cooled sufficiently for it to be safely handled.
2. Disconnect the Burner Plug from the Burner Socket.
3. Lift the Burner from the Spray Chamber stem using the heat resistant handle.

**WARNING:** Do not separate the burner from the plastic base when it is fitted to the spray chamber. ▲

## Organic Solvents

To measure samples in an organic solvent, you must first refer to the Flame Safety pages. If you are certain that the solvent is safe to use for Flame Atomic Absorption measurement, before lighting the flame, you must:

1. Empty any aqueous liquids from the drain.
2. Refill the drain with clean solvent.

Refer to the relevant instructions in the Flame Maintenance section below to learn how to do this.

If you are using methyl isobutyl ketone (MIBK) or similar solvents, you must prevent the solvent from coming into direct contact with the top of the spray chamber. Place a long stemmed funnel into the spray chamber neck, and pour the solvent through this until it flows freely from the drain.

You will normally use the Auxiliary Oxidant feature and adjust the fuel gas flow rate to obtain the correct flame chemistry for your analysis. These commands are on the Flame page of the system software. If your instrument is fitted with Variable Flow Auxiliary Oxidant kit, refer to the section below to learn how to adjust the flame chemistry.

When you have completed your analysis, empty the residual organic solvent from the drain, and replace it with clean water.

**WARNING:** Do not leave the drain full of organic solvent when you are not using the instrument. The solvent vapours can cause damage if they accumulate. ▲

## Lighting the flame

Before attempting to light the flame, confirm that:

- The gas supplies are correctly connected, and leakfree, and the gas supply pressures are correct.
- The Spray Chamber is clean, and the Drain Trap is filled with clean water or solvent.
- The Spray Chamber drains freely to waste.
- The Burner is clean and correctly fitted.



- The Sample Compartment Door is securely closed.
- The fume extraction system is turned on and working correctly.

When the gas pressures are correct, and the burner is correctly fitted, the Ignition Ready light beside the Sample Compartment Door will flash.

#### To light the flame:

1. Confirm that the Ignition Ready light is flashing.
2. Press and hold the Ignition Ready/Flame On switch until the default flame is established.
  - the default flame is a stoichiometric air/fuel flame.
  - if a flame is not established within 30 seconds, the automatic flame ignition system will stop. You will then have to wait approximately 30 seconds before you can try again.

**WARNING:** If you cannot ignite a flame after two attempts, it is likely that there is a problem with the burner, ignition system, or gas supplies. Refer to the Flame Maintenance section below and identify and rectify any problems before making another attempt to light the flame. ▲

**WARNING:** High voltages are present between the Ignition Electrode and the body of the burner during the ignition sequence. Do not touch either of these components during the ignition sequence. ▲

3. Use the system software to adjust the fuel flow rate and oxidant gas type as required.

#### To extinguish the flame:

1. Aspirate clean water or solvent to remove all traces of sample solution from the Spray Chamber and Drain.
2. Press the Flame Off button.
  - the oxidant gas will change to air, if necessary.
  - the fuel flow will increase to give a fuel rich flame.
  - the flame will then be shut off.

## Warming up the burner

It is important that you allow the burner to warm up properly before you start to take measurements, to ensure that your results are stable, and to minimise carbon deposition when using a nitrous oxide supported acetylene flame.

#### To warm up the burner for use with an air acetylene flame:

1. Set up a suitable analysis with the system software, ensuring that you select the Air Acetylene Flame Type, with a Fuel Gas flow rate of between 0.8 and 1.2l/min.
2. Light the flame, and confirm that it is burning correctly.
3. Aspirate an appropriate solution, such as deionised water.

4. Use the system software to execute a Flame Setup command.
5. Allow the flame to burn for ten minutes before starting your analysis.

**To warm up the burner for use with a nitrous oxide acetylene flame:**

1. Warm up the burner with an air acetylene flame, as described above.
2. Change the Flame Type to Nitrous Oxide Acetylene, and set the Fuel Gas flow rate to 3.6 - 3.8 l/min.
3. Use the system software to execute a Flame Setup command.
4. Confirm that the oxidant gas changeover takes place correctly, and that the nitrous oxide acetylene flame is burning correctly.
5. Allow the flame to burn for ten minutes before starting your analysis.

## **Burner Alignment**

Burner alignment is achieved by aspirating a suitable sample solution, and adjusting the Burner position to obtain the maximum absorbance signal.

**To align the Burner:**

1. Set up a suitable analysis with the system software, install a hollow cathode lamp, and perform an Optical Setup. Ensure that the live absorbance signal is displayed.
2. Light the flame, and allow a few minutes for it to stabilise.
3. Aspirate deionised water and, if necessary, autozero the absorbance signal.
4. Open the Flame Sample Compartment door, and aspirate a suitable test solution that gives a signal between 0.1 and 0.8 absorbance units.
5. Use the Transverse Adjustment control to adjust the transverse position of the Burner to maximise the signal from the test solution.
6. Adjust the angular position of the Burner by rotating the whole burner on the Spray Chamber stem to maximise the absorbance of the test signal.

**WARNING:** Do not attempt to adjust the Burner with a nitrous oxide supported flame burning. When rotating the Burner with a flame burning, hold it only by the heat resistant handle. ▲

7. Close and secure the flame sample compartment door.
8. Use the Burner Height controls in the system software to adjust the height of the Burner to maximise the test signal.
  - The SOLAAR Data Station software provides an automatic Burner Height optimisation function to optimise this adjustment for you.

**WARNING:** Do not attempt to touch the burner support or height adjustment mechanism when the height adjustment mechanism is operating. ▲

## Impact Bead alignment

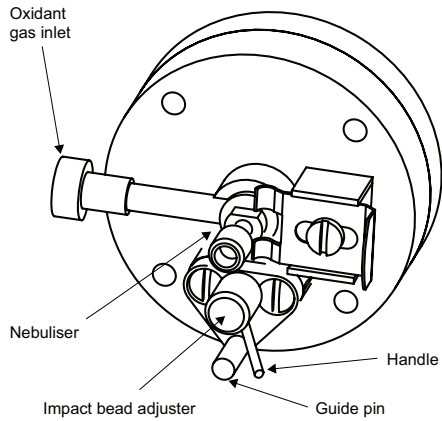
The impact bead is aligned by aspirating a suitable sample solution, and adjusting the Impact Bead position to obtain the maximum absorbance signal.

### To align the Impact Bead:

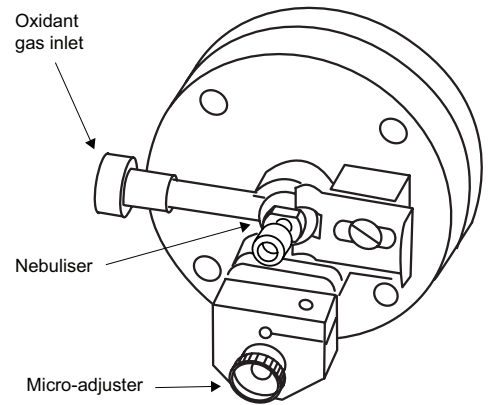
1. Set up a suitable analysis with the system software, install a hollow cathode lamp, and perform an Optical Setup. Ensure that the live absorbance signal is displayed.
2. Light the flame, and allow a few minutes for it to stabilise.
3. Align the Burner, as described above.
4. Aspirate deionised water and, if necessary, autozero the absorbance signal.
5. Aspirate a suitable test solution that gives a signal between 0.1 and 0.8 absorbance units.
6. Use the Impact Bead control to adjust the position of the Impact Bead to maximise the signal from the test solution.
  - Flame capable iCE 3000 Series Spectrometers may be fitted with either a Micro-adjustable Impact Bead control or a Standard Impact Bead control. Refer to the figure to identify the type of adjustment required.
  - Adjust the Standard Impact Bead control by pulling or pushing it into and out of the Spray Chamber cap. Ensure that the handle remains in contact with the guide pin at all times.
  - Adjust the Micro-adjustable Impact Bead control by rotating the Micro-adjuster.

**WARNING:** Do not attempt to adjust the Impact Bead with a nitrous oxide supported flame burning. ▲

**WARNING:** Do not attempt to adjust the Impact Bead position too far towards the Nebuliser as it may damage either the Nebuliser or the Impact Bead surface. ▲



**Fig 4 - 3. Standard Impact Bead Adjuster**



**Micro-adjustable Impact Bead**

**Venting the Gas System**

Venting the Gas System releases the gas pressure in the lines to the spectrometer. The Gas System should be vented at the end of the working day, or when the instrument will not be used again immediately.

**To vent the Gas System:**

1. Turn off the flame.
2. Shut off the gas supplies at source.
3. Press and hold the Flame Off button.
  - After about 10 seconds, you will hear a click and the gases will be vented through the Burner.
4. When the pressure gauges on the fuel and nitrous oxide gas lines have fallen to zero, release the Flame Off button.

**Flame System Spares and Consumables**

Universal Finned Titanium Burner (5 cm)	9423 520 31011
Air/acetylene Ti Burner (10 cm)	9423 420 31021
0.4 mm sample uptake tube	9423 390 05411
0.5 mm sample uptake tube	9423 390 05421
Acetylene gas pressure regulator	9423 352 43881
Nitrous oxide gas pressure regulator	9423 354 03111
Acetylene flash back arrestor	9423 393 46061

## Flame System Maintenance

**Introduction** Flame System maintenance must be performed at regular intervals to ensure safe and reliable operation. Flame AAS involves high temperatures and corrosive solutions, therefore system maintenance is essential.

**Safety** Turn off all gas supplies and disconnect the spectrometer and accessories from the mains before carrying out maintenance work.

## Burner



**CAUTION:** Do not attempt to clean the burner while a flame is burning, and ensure that the burner has cooled before handling it. ▲

### To clean the external surfaces of the Burner

1. Clean and polish the top surfaces using a mild abrasive soap cleaner.
2. Clean the burner slot with the Burner Cleaning Tool supplied or a piece of stiff card. Do NOT use an abrasive material inside the slot.
3. Wash the Burner with detergent solution and rinse with deionised water. Dry the Burner carefully before using it again.



**CAUTION:** Do not attempt to dismantle the burner while it's still plugged to the spectrometer. ▲

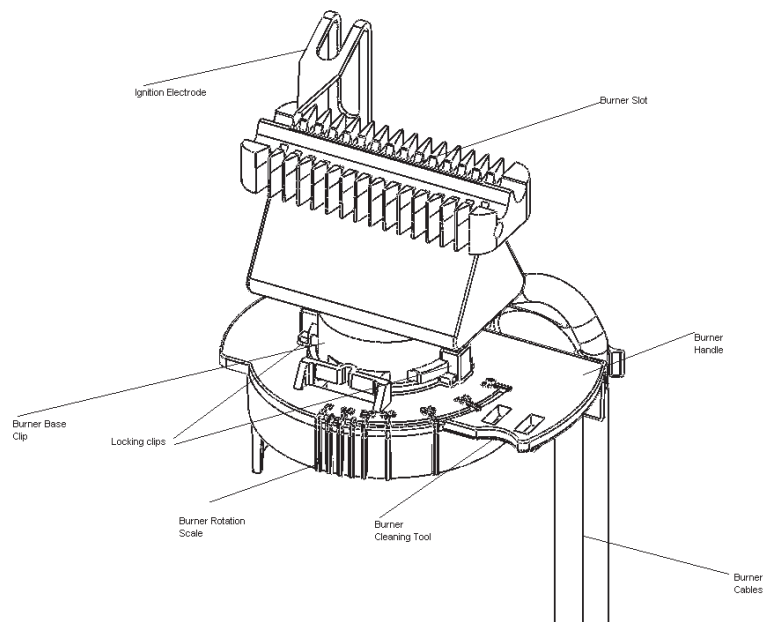
### To dismantle the Burner

1. While holding the base firmly, twist the burner body anti-clockwise to release it from the burner retention clip and securing lugs.
2. Lift the Burner Head away from the Burner Base to separate the two parts.

### To clean the internal surfaces of the Burner

1. Use an ultrasonic bath filled with deionised water, dilute detergent solution or 5% v/v solution of nitric acid to clean the internal surfaces of the Burner Head. Dry the burner thoroughly before use.

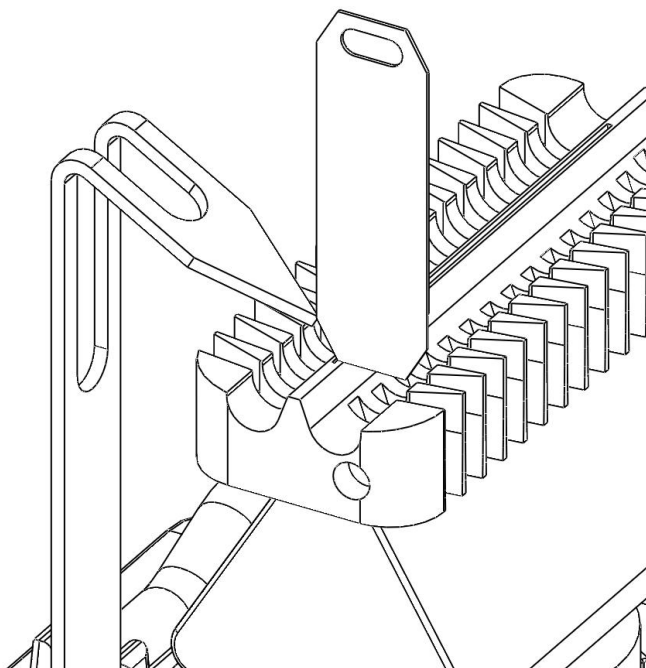
**WARNING:** Do NOT put the plastic Burner Base and Ignition Electrode assembly into the ultrasonic bath. ▲



**Fig 4 - 4.** Universal finned titanium burner head

### To re-assemble the Burner

1. Orientate the Burner Base so that it is upright, with the Burner Handle to the right.
2. Orientate the Burner Head so that the Ignition Well is on the left hand side to the rear.
3. Rotate the burner head about 45° anti-clockwise and push it firmly into the base.
4. Rotate the burner head clockwise in the base so that the securing lugs engage, and the burner retention clip snaps into place.



**Fig 4 - 5.** Ignition electrode adjustment

**WARNING:** The Burner must always be fitted to the correct Burner Base, as supplied with the burner. ▲

### To align the Ignition Electrode

1. Refer to the figure, and place the Cleaning Tool/Setting Jig as shown, aligning the bottom corner of the chamfer with the edge of the Ignition Well.
2. Gently bend the Electrode until the tip touches the top corner of the chamfer on the Setting Jig, as shown above.

### Spray Chamber

To clean the Spray Chamber

1. Aspirate a solution of dilute (1% v/v) hydrochloric acid, or a solution of laboratory detergent, whichever is most appropriate. Aspirate the solution for 5 minutes, then aspirate deionised water.
2. For more thorough cleaning, dismantle the Spray Chamber, clean the individual components with laboratory detergent, rinse thoroughly with deionised water, dry and re-assemble.

### To remove and dismantle the Spray Chamber

1. Use the system software commands to raise the Burner until the Burner Height is less than 10 mm.
2. Remove the Burner.
3. Empty the Spray Chamber Drain.
4. Disconnect the gas lines to the Spray Chamber and disconnect the Drain Tube.
5. Release the Burner Mount Clamp Screw by at least 3 full turns, then rotate the Spray Chamber downwards around the axis of the bayonet mount to release it and the Clamp Ring/Mounting Bracket from the bayonet mount, and remove the assembly.
6. Unscrew the 4 thumbscrews, and remove the Spray Chamber Front Cap.
7. Unscrew the 3 Rear Clamp screws, and remove the Clamp Ring or Clamp Ring/Mounting Bracket and the Over-Pressure Disc.
8. Push the Baffle Assembly out of the Spray Chamber body towards the front.
9. Undo the Fuel Gas Coupling on the Spray Chamber body, and remove the Gauze Disc assembly.



**CAUTION:** Do not attempt to dismantle the cap assembly while the spray chamber is still in the flame compartment. ▲.

### To dismantle the Cap Assembly

1. Slacken the Impact Bead Clamp Adjusting Screw, and withdraw the Bead from the Alumina Disc side of the Cap.
2. Remove the Seal and the Alumina Disc.
3. Undo the securing screws, and remove the Bead Adjustment Assembly.
4. Slacken the Nebuliser Retaining Clip screw, withdraw the Clip, and then remove the Nebuliser and O-ring.



**CAUTION:** Never re-assemble the spray chamber with damaged parts.▲.

### To re-assemble the Cap Assembly

1. Fit the O-ring to the Nebuliser body, re-fit the Nebuliser to the Cap, and replace the Clip and tighten the screw.
2. Lightly lubricate the Impact Bead stem with silicone grease, pass it through the Alumina Disc, and fit one O-ring over the stem.

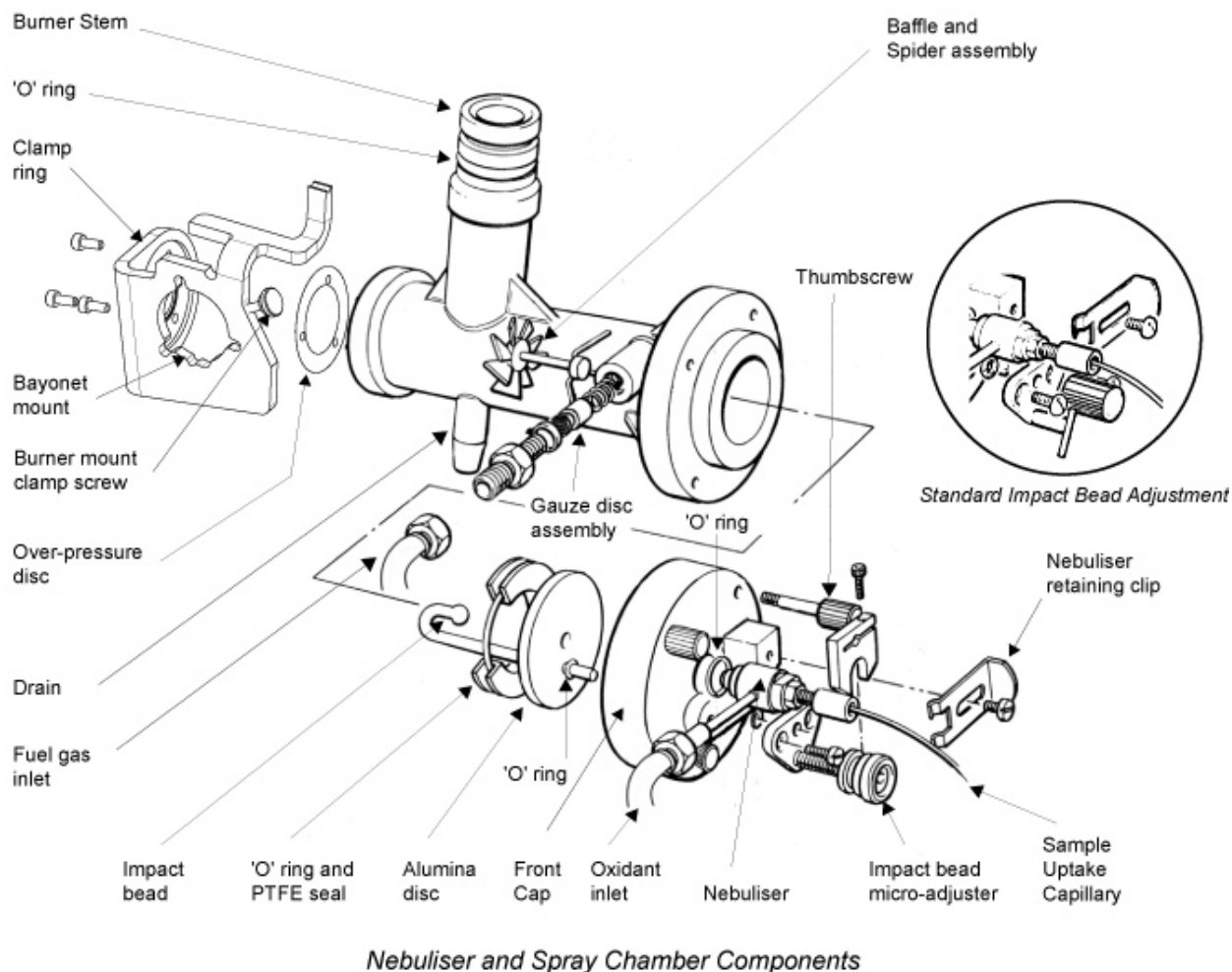


**CAUTION:** Take great care not to contaminate the Bead itself or the Alumina Disc with grease ▲.

3. Fit the Alumina Disc to the Cap, passing the Impact Bead stem through the hole in the Cap.
4. Fit the second O-ring to the stem, slide the Bead Adjustment assembly on to the stem, and secure it to the Cap.
5. Set the Bead Adjuster to the fully out position.



6. Position the Impact Bead so that the Bead is centrally located in the Nebuliser Nose Cone, and is as close as possible to the Nose Cone.
7. Tighten the Bead Clamp screw.
8. Refit the Alumina Disc and Seal.



**Fig 4 - 6.** Nebuliser and Spray Chamber Components

**To re-assemble the spray chamber**

1. Inspect, clean and replace any damaged components.
2. Push the Baffle Assembly into the front of the Spray Chamber body, with the Baffle to the rear, and a short Baffle Blade at the bottom. Push the assembly tightly into the Spray Chamber body taper.
3. Re-assemble and re-fit the Gauze Disc assembly and the Fuel Inlet Coupling.
4. Using a new Over-Pressure Disc, refit the Disc and Clamp Ring.
5. Re-fit the Cap Assembly, and tighten the thumbscrews.
6. Re-fit the Spray Chamber to the Spectrometer Mount, and re-connect the Gas Lines, Drain and Burner. Perform a Leak Test, and rectify any leaks before lighting a flame.

## **Nebuliser** To clean the Nebuliser

1. Extinguish the flame.
2. Remove the Nebuliser Uptake Capillary.
3. Push the Nebuliser Cleaning Probe through the Nebuliser Capillary to remove any blockages.

**WARNING:** Do not use any other tool or wire to clean the nebuliser. ▲

4. Replace the Nebuliser Uptake Tube, light an air/acetylene flame and check the Nebuliser uptake rate.

### **To measure the Nebuliser uptake rate**

1. Fill a 10 ml measuring cylinder with deionised water.
2. Fit a standard length (250 mm) of 0.5 mm ID Nebuliser Capillary.
3. Light an air/acetylene flame.
4. Aspirate the water from the measuring cylinder and note the volume aspirated in 1 minute.

The normal Nebuliser uptake rate under these conditions should be > 6 mL/min.

Periodically check the external Drain Tube, and replace it if it is cracked or damaged.

- If the Drain becomes blocked, it can be cleaned by pouring dilute hydrochloric acid (5% v/v) or laboratory detergent down the Spray Chamber Stem, and leaving it to soak. When the blockage has dissolved, empty the Drain, and refill it with clean water.

## **Drain** To empty and refill the Drain.

1. Turn off the fuel and nitrous oxide gas supplies at source, but leave the air supply turned on.
2. Remove the Burner.
3. Ensure that a suitable container is positioned to receive the contents of the Drain.
4. Block the top of the Spray Chamber with a suitable bung.
5. Vent the Gas System by holding down the Flame Off button for at least 30 seconds. This will cause the contents of the Drain to be blown out to waste.
6. Refill the Drain by pouring clean water or solvent into the top of the Spray Chamber Stem until it flows freely from the Drain.

## **Gas Supply hoses**

- Inspect the Gas Supply hoses regularly, and replace any that are cracked or damaged.
- We recommend that all Gas Supply hoses are replaced every three years.
- Run the Gas Leak Test at least once every six months, and whenever the instrument is moved, ensure no leaks are found.

**Gas Leak Test** The software commands necessary to run the Gas Leak test are included in the SOLAAR OQ Tests Data Station application.

The Data Station OQ Tests software presents the Gas Leak Tests as a Wizard, with full on-screen instructions.

<b>Spares and Consumables</b>	Flame compartment User Spares Kit	9423 450 30001
	Nebuliser	9423 390 05481
	Nebuliser Probe	9423 390 05441
	Spray Chamber Renovation Kit	9423 390 05461
	Replacement 'O' ring Kit	9423 390 05151

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## Air Compressor

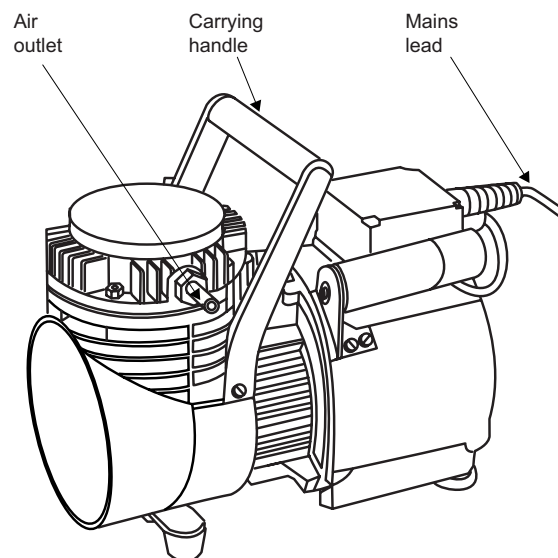
- Introduction** This is an accessory which provides a flow of clean, dry, compressed air for use as the oxidant for air supported flames.
- A Filter/Regulator unit is included to remove particles and moisture, and ensure that a stable air pressure to the Spectrometer is maintained.
  - Performance is reduced at high altitude.
- Safety**
- Observe electrical safety precautions.
  - Use the accessory in a well ventilated position.
  - Ensure that the accessory is not close to a heat source.
    - This could result in poor removal of water vapour from the compressed air, causing flame instability.
  - Do not use the accessory as a pump for combustible liquids or vapours.
  - This accessory is an oil-free pump, and does NOT require lubrication. Do **NOT** allow hydrocarbons to come into contact with the diaphragm.
  - Do **NOT** operate the accessory without the fan guards in place.

## Installation

1. Confirm that your Compressor is suitable for the mains supply in your laboratory.  
9423 393 34225                      220V/50Hz  $\pm$  10%  
9423 393 34226                      220V/60Hz  $\pm$  10%  
9423 393 34115                      110V/50Hz  $\pm$  10%  
9423 393 34116                      110V/60Hz  $\pm$  10%
2. Fit a suitable plug to the Mains Cable.
3. Place the Compressor in a well ventilated position close to the Spectrometer.
4. Use the black Connecting Hose to connect the Compressor Outlet to the Filter/Regulator inlet.
5. Position the hose so that it is:
  - free from bends
  - away from heat sources
  - not possible for condensation to run back into the compressor.
6. Connect the clear air hose from the outlet of the Filter/Regulator unit to the Spectrometer Air inlet connector.
7. The Compressor is now ready to use.



**CAUTION:** Do not shorten pipe



**Fig 4 - 7.** Air Compressor

## Operation

1. Ensure that the Drain Plug at the base of the Filter/Regulator unit is closed, and that the system is leak free.
2. Switch on the Compressor.
3. Set the Filter/Regulator to 2.1bar (30 psi).
4. Allow the unit to run for 30 minutes to achieve normal operating temperature, then check and reset the pressure if necessary.
5. Switch on the Spectrometer, and carry out your analysis.
6. After use, switch off the Compressor and vent the Gas Control system.



**CAUTION:** Once the Compressor has been switched off, do not switch it on again until the line pressure has dropped to below 0.14 bar (2 psi), otherwise you may damage the Compressor Motor. ▲

## Maintenance

### Compressor

- Inspect and clean external surfaces regularly.

### Filter/Regulator unit

- Inspect the Filter Trap regularly, and drain out any liquid collected.
- Clean the Filter Bowl by rinsing with paraffin if necessary. Do not use any other solvent.
- If any liquid has collected in the hose, drain it into a suitable container.
- Inspect rubber components regularly for deterioration and damage.

## Spares and Consumables

Filter/Regulator Assembly

4013 229 31741

Compressor Service Kit

9423 393 34005

(User documentation for this kit is included in the manual supplied with your compressor.)

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# The ASX-520 and ASX-260 Flame Autosamplers

**Introduction** The ASX-520 is a high capacity Autosampler, with integral wash facilities, capable of handling up to 360 samples. The ASX-260 is a medium capacity Autosampler capable of handling up to 180 samples.

They are compatible with

- Normal flame operation.
- Flame dilution with the ID100 Autodiluter Accessory.
- Vapour operation with the VP100 Accessory.

**Safety**

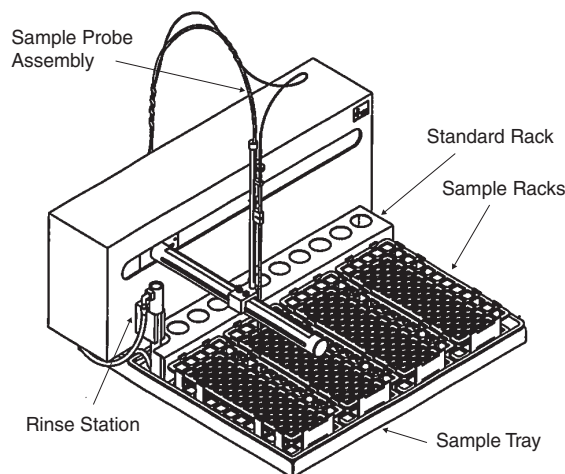
- Observe electrical safety precautions.
- Read the Safety Section of the User documentation supplied with the unit.

## Installation Assembly

1. Unpack and assemble the unit as detailed in the Autosampler Operators Manual supplied with the unit.
  - The Autosampler Operators Manual may be provided in electronic form on a CD included with the parts sent with the Autosampler.
2. Position the Autosampler on the Accessory Trolley, or on the bench in front of the appropriate Sample Compartment.
3. Mount the Sample Probe Assembly, install the Sample Probe and set the Z axis travel as described in the Autosampler Manual.
  - Two types of uptake tube are supplied - one with an internal diameter of 0.5 mm, and one with an internal diameter of 0.8 mm. The 0.5 mm ID uptake tube is suitable for general purpose use, but will cause some loss of analytical sensitivity, particularly when longer lengths are used. In these circumstances, use of the 0.8 mm ID uptake tube will restore the sensitivity.
4. Connect the Rinse Station as described. Either configuration may be used.

### System interconnections

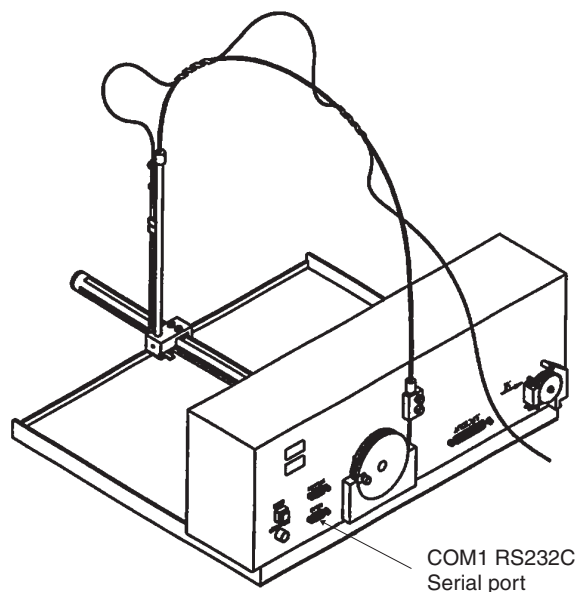
1. Connect the 9-9 way RS232C Interface Cable supplied between one of the Accessory connections on the Spectrometer, and the COM 1 RS232C socket on the rear of the unit.
2. Connect a suitable mains cable to the in-line mains power supply, and connect the power supply output to the Power connection on the unit.



**Fig 4 - 8. Cetac ASX-520 Sample Changer - General View**

### Capillary tubing connections

1. Select the appropriate Sample Transfer Tube interface.
2. Connect the Sample Transfer Tube to the Autosampler.
3. Connect the free end of the tube as follows:
  - Normal Flame operation. Cut back the tube to the minimum convenient length, and connect the free end to the Nebuliser.
  - Other Accessories. Refer to the appropriate accessory page.



**Fig 4 - 9.** Cetac ASX-520 - Rear View

**Note:** If you choose to use the 0.8 mm ID uptake tube supplied, you will find a short length of 0.5 mm ID tube attached to the free end of the tube. This is necessary to allow the larger tube to fit securely on to the nebuliser capillary. Remove the section of 0.5 mm ID tube before cutting the main tube to length, then refit it after cutting. ▲

**Sample Racks**

1. Assemble the Sample Racks required and place them in the Sample Tray. All the Sample Racks must be of the same type.
2. Place the appropriate Sample Tubes into the Racks:
3. Place the Standard Tubes into the rear Standards Rack.

Sample positions in rack	Tube diameter
21	30mm
24	25mm
40	20mm
60	16mm
90	13mm

**Operation**

1. Switch on and set up the Spectrometer and accessories.
2. Switch on the Autosampler, and wait for it to initialise.
3. Use the Data Station software facilities to define the type and number of Sample Racks, and the number of solutions required.
4. Refer to the Autosampler Loading Guide facility in the software, and place the solutions in the correct positions in the Racks.
5. The system is now ready for you to start the analysis.

**Spares and Consumables**

**Maintenance**

- Ensure that the Autosampler is switched off and isolated from the mains supply before carrying out any maintenance.

## Flame Operation

### The ASX-520 and ASX-260 Flame Autosamplers - Spares and Consumables

- Wipe the exterior surfaces with a soft cloth dipped in soapy water to remove any marks.
- If a spillage occurs, isolate the Autosampler from the mains supply immediately, and wipe up the spillage with a dry cloth.
- The ASX-520 and ASX-260 Autosamplers do NOT require routine lubrication.
- The Autosampler must never be picked up by either the vertical or horizontal arms, but only by supporting the base of the device.

21 position Rack	9423 470 03901
24 position Rack	9423 470 03911
40 position Rack	9423 470 03921
60 position Rack	9423 470 03931
90 position Rack	9423 470 03941
Sample Probe (0.5mm)	9423 470 03991
Sample Probe (0.8mm)	9423 470 04182
Tray Sub-assembly with Standard Rack	9423 470 03951
Rinse Station	9423 470 03961
Drain Pump Tubing/Connector Kit	9423 470 03971
Standard Tubes	
(50 ml polypropylene, 500)	9423 470 04151
50 ml Polypropylene Sample Tubes (for 21 position rack, 500)	9423 470 04101
30 ml Polypropylene Sample Tubes (for 24 position rack, 500)	9423 470 04111
18 ml Nalgene Sample Tubes (for 40 position rack, 100)	9423 470 04121
14 ml Polypropylene Sample Tubes (for 60 position rack, 1000)	9423 470 04131
8 ml Polypropylene Sample Tubes (for 90 position rack, 1000)	9423 470 04141

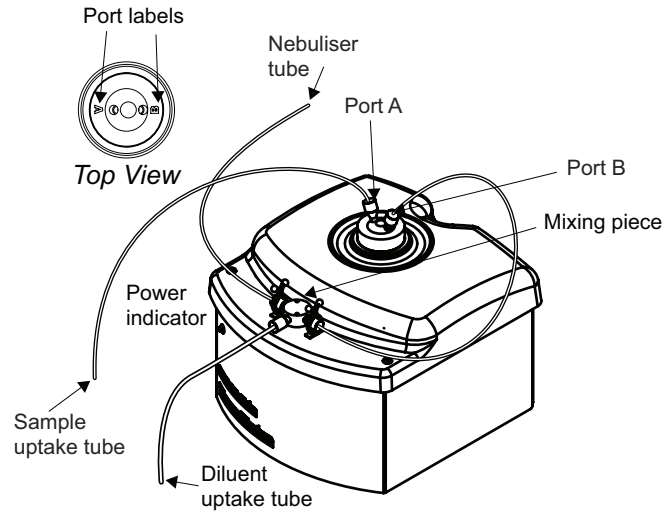
## The ID100 Autodilutor Accessory

**Introduction** This is an accessory that will provide automatic sample dilution and standard preparation for flame AAS.

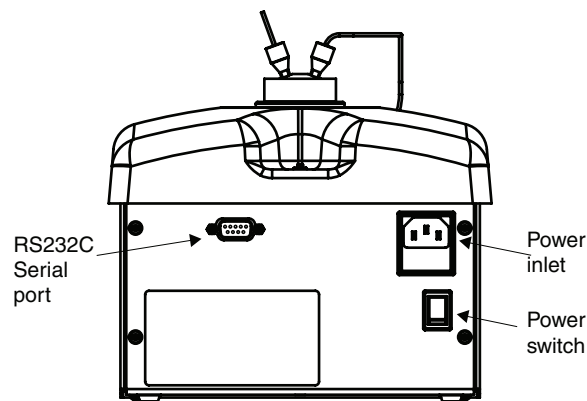
- It can be used manually or with any of the Flame Autosamplers.
- It is not compatible with the VP100 Vapour Generation accessory.

## Installation

Before installing your Autodilutor, refer to the figure and familiarise yourself with the parts shown.



**Fig 4 - 10.** ID100 Autodilutor - General View



**Fig 4 - 11.** ID100 Autodilutor - Rear View

### To install your Autodilutor:

1. Place the accessory in a convenient position in front of the spectrometer Flame Compartment.
2. Connect a suitable power lead between the power inlet socket and a power outlet.
3. Connect an RS232C cable between the RS232C port on the accessory and an Accessory port on the Spectrometer Connection Panel.
4. Locate the 1.5 mm OD, 0.5 mm ID polythene nebuliser tubing, and connect one end to the mixing piece using one of the white finger nut and ferrule connectors in the plumbing kit. Cut the tube to length, and fit the free end to the Nebuliser.
5. Locate the 1.5 mm OD, 0.8 mm ID PTFE tubing, and fit a suitable length between the 'B' port of the Autodilutor and port 2 of the mixing piece. Use a white finger nut and ferrule connector at the mixing piece, and a grey 10/32 miniature fitting at the Autodilutor end.

6. Locate the 3.2 mm OD wide bore PTFE tubing supplied, and fit it to the remaining free port of the mixing piece, using the red finger nut and ferrule connector. Trim the tube to a suitable length and place the free end in the diluent container.

### **Manual Sampling**

Fit a suitable length of 0.8 mm ID PTFE tubing to Port A of the Autodilutor using a grey miniature 10/32 connector.

### **Cetac Autosampler**

Either fit a spare grey miniature 10/32 connector to the free end of the Cetac Sample Probe tube, and fit that to Port A of the Autodilutor, or fit a 10/32 connector to a short piece of 0.8 mm ID PTFE tubing, fit that to Port A of the Autodilutor, then attach the Cetac Sample Probe tubing to this with a short piece of the thick walled silicone rubber tube supplied with the autosampler.

### **Connectors**

Miniature 10/32 compression fittings are used for the Autodilutor fluid connections.

#### **To fit a miniature 10/32 connector:**

1. Use a sharp blade to trim the end of the 1.6 mm OD tubing to a shallow taper.
2. Slide the nut over the tubing.
3. Slide the ferrule on to the tubing, until part of the taper has passed through the ferrule.
4. Grip the tapered end of the tubing, and pull it through the ferrule, so that the full width of the tubing passes through the ferrule.
5. Trim the tapered end from the tubing, leaving 0.5 - 1 mm of the tubing visible at the end of the ferrule.
6. Bring the nut up to the ferrule, and fit the assembly into the port. Tighten the nut until the assembly is secure.

## **Operation**

1. Prepare a sufficient quantity of diluent solution. Place the Diluent Uptake tube in this solution.
  - The diluent solution should be similar in composition to the sample solution matrix.
2. Turn on and set up the Spectrometer and lamps in the normal way, and light the flame.
  - You should then see that the Diluent solution is drawn up through the Mixing Piece into the Nebuliser.
3. Use the appropriate Wizards to optimise the Burner and Nebuliser.
4. Otherwise, disconnect the Nebuliser Uptake Tubing between the Autodilutor Mixing Piece and the Nebuliser at the Nebuliser end, and replace it with a standard piece of Nebuliser Tubing, then optimise the Burner position and Impact Bead as usual.

5. Replace the tube connecting the ID100 to the Nebuliser.
6. Set the Pre-Fill volume on the Sampling tab of the View.Options dialogue.
  - A Pre-Fill volume of 1200 - 1500 µl will be suitable for use with manual sampling with the ASX260 autosamplers. Up to 2000 µl will be needed for the larger ASX-520 autosampler.
  - If you notice memory effects when measuring solutions with widely different concentrations, increase the Pre-Fill volume.
7. Set up the analysis parameters in the normal way.
8. When working at high dilutions, small bubbles in the sample can disturb the operation of the ID100, resulting in poor measurement precision and incorrect results. You should take precautions to remove as much dissolved gas from the sample as possible, but it is not practicable to completely prevent the formation of such bubbles. We therefore recommend that you use the RSD Test facility in the SOLAAR Data Station software to identify and automatically repeat any measurements with poor (>5% RSD) precision.
8. Set the Nebuliser Delay flame parameter to a value of 10 seconds.
  - If the measured signal drifts during the measurement, increase the Nebuliser Delay Parameter.
9. Set the Wash Time parameter on the Sampling parameters page to 30 seconds, and set the Wash Frequency to Wash between Samples.
  - If you are analysing samples that are similar to each other in concentration, you may find that it is not necessary to wash the Autodilutor between samples.
10. Load the samples into the Autosampler, if required, then start the analysis in the normal way.
  - During the analysis, ensure that the liquid level in the Diluent Container does not fall below the levels of the uptake tube.
11. If you are working manually, carefully follow the prompts displayed. Particularly, always confirm that the Sample Uptake tube connected to Port A of the Autodilutor is placed in the correct solution before acknowledging the prompt.
12. When the analysis has finished, replace the sample and diluent with clean deionised water, and perform 2-5 wash cycles, to ensure that all sample and diluent is flushed from the diluter. Do NOT leave any sample or diluent in the diluter. If you do not intend to use the Autodilutor again immediately, remove the Sample and Diluent Uptake tubes from the liquid containers, and perform another wash cycle. This will remove all the fluid from inside the Autodilutor.

**WARNING:** If the outlet of the Diluter should become blocked, a small amount of liquid may be ejected from the Leak Port. If this happens, stop the Diluter immediately. Investigate and rectify the cause of the problem before using the accessory again. ▲



**WARNING:** Particulate material present in the samples can cause serious damage to the ID100. Ensure that your samples do NOT contain any particulate material before using them with the ID100. ▲

**WARNING:** Prolonged exposure to mineral acids at concentrations above 10% can cause degradation of the material used in the ID100 high precision piston pump. Avoid the use of samples containing high concentrations of mineral acid if possible; otherwise, ensure that you wash the ID100 with clean water between each measurement and at the end of the analysis. ▲

### **Maintenance**

- Wipe up any leaks or spillages as soon as they occur, and do not allow liquid to accumulate in the drip tray.
- Periodically, check for visible signs of leakage from the connectors, and replace any that are faulty.

### **Spares and Consumables**

ID100 Autodilutor Consumables Kit

9423 450 03201

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## The Slotted Tube Atom Trap (STAT)



**CAUTION:** Never attempt to use nitrous oxide supported flames with this accessory. ▲

### Introduction

- This is an accessory that enhances the flame sensitivity for certain elements by 2 - 5 times.
- It consists of a Slotted Tube held in the flame by a simple Holder.

### Safety

- The temperature of the Slotted Tube will be in excess of 1000° C during normal operation - ensure that the Tube and its Holder have cooled for at least 15 minutes before handling them.
- Use the STAT only with the 50 mm Universal Burner.



**CAUTION:** Never attempt to light or extinguish a flame with the STAT in the operational position. ▲

## Installation

The STAT Tube is held in the flame by the STAT Holder, which clips to the Burner.

### To install the STAT Holder:

1. Remove the Burner from the Spectrometer.
2. Orientate the burner with the Burner Handle to the right and the Ignition Electrode to the left.
3. Unclip the Strap Clip, to allow the Securing Clips to spread open.
4. Gently push the STAT Holder assembly on to the Burner until the Securing Clips click into the Locating holes on the Burner.
5. Clip the Strap Clip together again to hold the assembly in place.
6. Refit the Burner to the Spectrometer.

### To fit a STAT Tube to the STAT Holder:

1. Orientate the STAT Tube so that the longer slot is at the bottom.
2. Clip the STAT Tube into the STAT Holder.

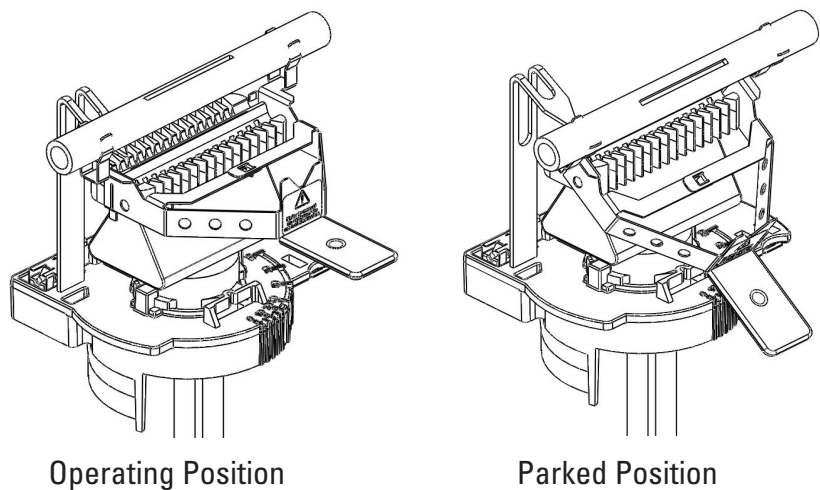


Fig 4 - 12. STAT Tube and Holder

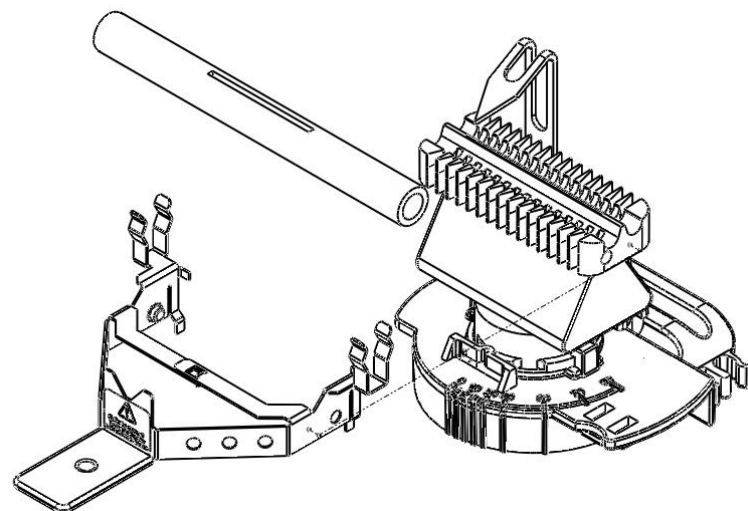


Fig 4 - 13. STAT Assembly

- Alignment**
1. With the Burner and STAT Holder fitted to the spectrometer, fit a STAT Tube to the Holder, then move the Holder to the Parked position
  2. Install a suitable hollow cathode lamp, and perform an optical set up.
  3. Move the Holder to the Operating position.
  4. Use a piece of white card at the right hand side of the Sample Compartment to locate the light beam.
  5. Adjust the Burner position until the light beam passes along the axis of the STAT Tube.
  6. Move the STAT Tube away from the Burner slot.

- Operation**
1. Ensure that the STAT Tube is not over the Burner.
  2. Set up your method as required. The STAT does not require any additional parameters to be set.
  3. Light the flame, and aspirate deionised water.
  4. When the flame is established, move the STAT Tube into its operating position over the Burner.
  5. Allow the STAT Tube to stabilise for 5 minutes, then start your analysis in the normal way.
  6. When the analysis has finished, move the STAT Tube out of the flame, then extinguish the flame.

- Maintenance**
- Remove the the STAT Tube from the Holder after using it. The Holder can remain on the Burner, where it will not interfere with normal operation.
  - If necessary internal deposits can be removed by rinsing with water or dilute acid.
  - If the flame is extinguished with the STAT Tube in the Operating position, it will become coated with soot. This will burn off when the tube is next used, or it can be removed by wiping the Tube with a dry cloth.

**Spares and Consumables**    Slotted Silica STAT tubes (5)    9423 393 35021

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# The Variable Flow Auxiliary Oxidant Accessory

## Introduction

- An auxiliary supply of Oxidant gas to the air/acetylene flame is necessary to maintain the optimum flame chemistry when using non-aqueous solvents.
- The standard configuration of the flame instruments provides a fixed Auxiliary Oxidant flow that is suitable for use with common solvents such as white spirit and methyl isobutyl ketone (MIBK).
- This accessory provides a variable flow of Auxiliary Oxidant to allow the flame chemistry to be optimised for other less common solvents.
- If you want to use your instrument to analyse this type of sample on a regular basis, we recommend that your instrument should also be fitted with the Solvent Resistant Flame Kit (part number 9423 420 31051).

## Safety

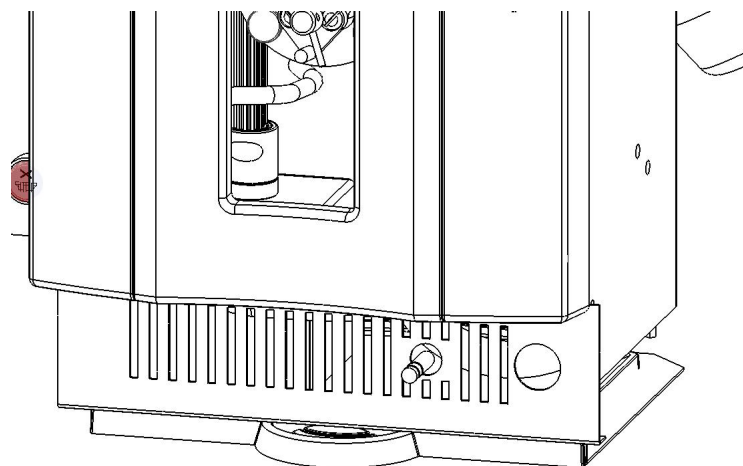
The use of organic solvents with flame AA spectrometry is inherently hazardous. Ensure that you read and fully understand the hazards and precautions described in the Flame Safety section of this manual.

**Installation** The accessory must be installed by a trained Service Engineer.

**Operation**

1. Set up the instrument in the normal way.
  - It may be beneficial to use a longer and/or narrower length of nebuliser uptake tube to reduce the aspiration rate of the sample, although this will also reduce the chemical sensitivity.
2. Ensure that the Auxiliary Oxidant parameter on the Flame page of the Method is selected.
  - You will normally also set the Fuel Flow rate parameter to its minimum value.
3. Confirm that the Variable Flow Auxiliary Oxidant Needle Valve is closed, and light the flame.
4. Use the Flame Setup command in the system software to set your chosen flame parameters.
5. Aspirate a typical sample solution, and slowly open the Needle Valve until the yellow colour disappears from the flame.
6. Stop aspirating the sample, and confirm that the flame remains stable.
  - If the flame lifts off the burner, adjust the needle valve to slightly decrease the auxiliary oxidant flow rate and/or increase the fuel flow rate to the flame.
7. Use the system software to display the analytical signal, and make fine adjustments to the auxiliary oxidant flow rate, the fuel gas flow rate, the burner height and the impact bead position, to obtain the best signal from your sample.

**Maintenance** Check the accessory for wear and damage at regular intervals.



**Fig 4 - 14.** Flame Compartment Valance with Variable Flow Auxiliary Oxidant fitted

**Spares and Consumables**

0.5 mm ID nebuliser uptake tubing (standard)	9423 390 05421
0.4 mm ID nebuliser uptake tubing (reduced uptake rate)	9423 390 05411



# Solvent Resistant Flame Kit

**Introduction** This manual describes the use of the components included in the Solvent Resistant Flame Kit (part number 9423 420 31051).

This Kit has been designed for the Thermo Fisher Scientific Flame Atomic Absorption Spectrometers. It provides replacements for certain parts in the standard flame atomisation system that can be susceptible to damage if subject to long term exposure to certain organic solvents.

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**Installation** The Solvent Resistant Flame Kit will be installed at our factory, if you specified it on your original spectrometer order.

Alternatively it can be installed as a field upgrade by a Field Service Engineer who has been properly trained and authorized to do this work by Thermo Fisher Scientific.

Do **NOT** attempt to install the Solvent Resistant Flame kit yourself.

**Components**  
**Spray Chamber** The Kit includes a modified Spray Chamber. The top of the spray chamber stem has been modified with a Teflon sleeve. This prevents the top of the stem from softening when ketone solvents such as methyl isobutyl ketone, di-isobutyl ketone and methyl ethyl ketone are run for long periods with a high temperature nitrous oxide supported flame.

**WARNING:** The teflon sleeve should not be removed from the stem of the spray chamber. ▲

The drain tube connection of the Spray Chamber has also been modified to incorporate an 'O' ring seal. This prevents leaks that can occur with certain low viscosity solvents such as white spirit.

**Drain Tube** The Kit includes an extended length of the convoluted PTFE Drain that extends down to the drain vessel, and does not require a joint between the PTFE tube and the standard polythene drain tube. This prevents leakage of certain low viscosity solvents at the joint, and prevents degradation of the standard polythene drain tube.

**'O' rings** The Kit includes a set of 'O' rings made of the perfluoroelastomer 'Kalrez' which are resistant to attack by certain solvents that cause the standard Viton 'O' rings to swell and degrade. Three of the standard Viton 'O' rings are replaced with Kalrez 'O' rings. These are located at the spray chamber neck, the spray chamber drain, and the nebuliser-to-spray chamber seal.

**0.4 mm ID Sample Uptake Tube** It is often desirable to reduce the sample uptake rate when measuring samples in organic solvents, as this can make it easier to optimize the flame chemistry. This can be conveniently achieved by replacing the standard 0.5 mm ID sample uptake tubing with the 0.4 mm ID tubing supplied in the Kit.

**Flame Measurements in organic solvents Safety** Before using your instrument, refer to the Safety sections of the main Operators Manual that are concerned with safe use of the spectrometer, and safe use of the flame atomisation system.

If you wish to use organic solvents on a regular basis, we recommend that you have the Solvent Resistant Flame Kit (part number 9423 420 31051) fitted to your instrument.

**Risk Assessment** The use of organic solvents in flame AAS is an inherently hazardous procedure. As a minimum, you should:

- use the minimum possible quantity of solvent in the vicinity of the spectrometer.
- ensure that any aqueous solution in the drain trap is removed and replaced with organic solvent before starting the analysis.
- ensure that the organic solvent is removed from the drain trap, and the vicinity of the instrument, as soon as the analysis is finished.
- ensure that the drain vessel is resistant to the solvent being used, and is suitably vented to prevent build-up of vapour inside the vessel. Empty the drain vessel frequently, to minimize the quantity of solvent in the vicinity of the spectrometer.
- ensure that the extraction system is operating properly, to minimize the risk of solvent vapours accumulating in the vicinity of the spectrometer.
- ensure that the nebuliser, spray chamber and burner are clean and free from leaks
- ensure that the gas supplies are set to the correct operating pressures, as listed in the main Operators Manuals, that the pressure regulators are operating properly and that the gas connections are free from leaks.

**WARNING:** Many common laboratory solvents are unsuitable for use with flame AAS. Halogenated solvents (chloroform, Freons) produce large quantities of toxic gases when burned; solvents with low flash points (ethers, light petroleum spirit) present too great a flammability hazard to be safely used, and unsaturated aromatic hydrocarbons (toluene, xylene) do not have suitable burning characteristics. ▲

To minimise the hazards to personnel and equipment, we recommend that you should carry out a comprehensive risk reduction assessment before performing the analysis. This should, at a minimum, include measures to reduce the hazards associated with:

- the risk of accidental spillage of solvent in and around the instrument.
- the risk of accidental ignition of solvent.
- the risk of a flash-back occurring in the spray chamber.

**Drain Vessel** Waste solvent draining from the instrument will accumulate in the drain vessel. To minimise the hazards associated with this, you should ensure that:

- the drain vessel is constructed of a material that is resistant to the solvent used.
- the drain vessel is located in a secure position such that it cannot be accidentally over-turned or its contents spilt.
- the capacity of the drain vessel is no greater than 500ml, to

prevent the accumulation of large volumes of waste solvent. It must be emptied frequently.

- the neck of the drain vessel is wide relative to the body of the vessel, so that waste solvent can enter freely and vapours can disperse easily. Alternatively, vapours can be removed from the vessel with a suitable venting system, but it is still important that vessel has a large opening, and is not sealed in any way.

### Auxiliary Oxidant

When an organic solvent is sprayed into a flame, the solvent combusts and so increases the effective fuel supply to the flame. This causes a change in the flame chemistry that can result in reduced sensitivity and increased interferences for certain elements. This problem affects only air/acetylene flames; with nitrous oxide supported flames, the oxidant is sufficiently powerful that compensation for the additional fuel effect of the solvent can be achieved with the normal fuel flow rate adjustments available.

With an air supported flame, an additional supply of oxidant is normally required to obtain the optimum flame chemistry. The flame capable iCE 3000 series spectrometers fitted with fully automatic gas handling systems, provide a facility known as the Auxiliary Oxidant that provides this additional oxidant supply. This can be selected on the Flame Parameters page of the SOLAAR Data Station.

The standard Auxiliary Oxidant feature supplies a fixed flow rate of additional oxidant that will be suitable for flame measurements with the solvents commonly used in AAS, such as methyl isobutyl ketone and white spirit.

The Variable Flow Oxidant Kit Auxiliary (part number 9423 393 46111) is an optional accessory that provides facilities for adjusting the flow rate of the additional oxidant, so permitting a wider range of less common solvents to be used.

### Flame Atomiser Spray Chamber Drain

Unless the solvent that you are planning to use is freely miscible with water, you must remove any aqueous solutions from the spray chamber before you introduce the solvent. You must then re-fill the spray chamber with clean solvent. These procedures are described in the main spectrometer Operators Manual.

We strongly recommend that you do **NOT** leave solvent in the spray chamber when you are not using the instrument, as solvent vapor can accumulate in and around the spectrometer, creating a fire hazard and potentially damaging plastic and other components.

Therefore, you should remove any solvent from the spray chamber drain when you have finished your analysis. If you intend to use the solvent again when you next use the instrument, you can leave the spray chamber drain dry. If you intend to use aqueous samples next re-fill the spray chamber drain with clean water.

### **Burner warm-up**

It is always important to ensure that your burner head has been properly warmed up before you start to take measurements. Not only will this give you the most stable and accurate results, but it will also minimise carbon deposition on and around the burner slot if you are using a nitrous oxide supported flame.

We recommend the following procedure for warming up your burner when using an organic solvent:

1. Perform the normal flame safety checks described in the Operators Manual, and ensure that any additional checks and actions associated with your risk reduction assessment have been properly performed.
2. Fill the spray chamber drain with clean solvent as described above.
3. Set the flame type to Air Acetylene, with a fuel flow rate of 1.2 l/min.
4. Ignite the flame, and perform a flame setup. Do NOT aspirate solvent into the nebuliser at this time.
5. Allow the flame to burn for at least ten minutes.
6. If you intend to use an air supported flame, proceed with the optimisation of the flame described below
7. If you intend to use a nitrous oxide supported flame, set the flame type to Nitrous Oxide Acetylene, with a fuel flow rate of 4.2 l/min. Perform a flame setup, and, when the flame has stabilised, reduce the fuel flow rate to 3.8-3.8 l/min. This should result in a lean flame, with a small red zone no more than 1-2 mm high. Do **NOT** aspirate solvent into the nebuliser at this time.

**WARNING:** If the red zone at the base of flame disappears entirely, immediately extinguish the flame and check the nitrous oxide supply pressure - it should be no higher than 40 psi (2.7 bar). ▲

8. Allow the flame to burn for at least ten minutes, then proceed with the optimisation of the flame described below.

### **Flame optimisation Initial optimisation for air supported flames**

When the burner has been properly warmed up, set the Auxiliary Oxidant option on the Flame Parameters page of the SOLAAR software, and perform a flame setup. The appearance of the flame will change as the oxidant flow is increased.

Aspirate clean solvent into the flame. If the flame colour changes to yellow, indicating a fuel rich flame, reduce the Fuel flow rate until a clear flame is obtained. If you reach the minimum fuel flow rate (0.8 l/min) before the flame becomes clear, you should reduce the uptake of the solvent by changing to a narrower bore and/or longer length of nebuliser uptake tubing.

When you have established the conditions that give you a clean flame, stop aspirating the solvent, and confirm that the flame does not lift off

the burner. It may be necessary to make further minor adjustments to the fuel gas flow rate and sample uptake rate to obtain a clean flame that is stable when no solvent is being aspirated.

### **The Variable Flow Auxiliary Oxidant Kit**

If you have the Variable Flow Auxiliary Oxidant accessory fitted to your spectrometer, you should optimise the flame according to the procedure below.

Having warmed up the burner, confirm the Auxiliary Oxidant flow control needle valve is fully closed, then turn on the Auxiliary Oxidant facility on the Flame Parameters page as described above. When you execute a flame setup command, the appearance of the flame should not change.

Aspirate the clean solvent, then slowly and carefully open the Auxiliary Oxidant flow control needle valve until the flame becomes clear. If you open the needle valve too far, you may find that the flame lifts off the burner head and may go out. If this happens, close the valve, re-light the flame, and open the valve a lesser amount. You can also adjust the fuel flow rate, and the nebuliser uptake rate, as described above, to obtain a clean flame while aspirating your solvent, that is also stable when solvent is not aspirated.

### **Initial optimisation for nitrous oxide supported flames**

Auxiliary oxidant is not required with nitrous oxide supported flames, and the facility cannot be selected in the software when the Flame Type is Nitrous Oxide/Acetylene.

When the burner has been properly warmed, aspirate the clean solvent into the flame. If the bright white colour of free carbon appears in the flame, reduce the fuel flow rate and/or reduce the sample uptake rate as described above to obtain a clean flame.

### **Final optimisation for all flames**

When you have obtained settings that give you a clean flame while aspirating clean solvent, you can then perform the final optimisation to obtain the best signals from your samples. You can use the Wizards and automatic flame optimisation tools provided in the SOLAAR Data Station software, or the normal manual procedures, to perform the final optimisation of fuel gas flow and burner height while monitoring the absorbance signal from your sample. If it is necessary to optimise the transverse position and rotational positions of the burner, take extra care when opening the sample compartment door, as there will be inflammable solvents in the vicinity of the exposed flame.

**Do not attempt any optimisations that require you to open the sample compartment door while a nitrous oxide flame is burning.**

The transverse and rotational burner positions can both be set while measuring an element that can be atomised in an air supported flame, and they will then also be in the correct positions for measurements in a nitrous oxide supported flame.

**Note:** The On-Line Cookbook and other documentation that we provide list typical performance data, such as characteristic

concentration, that you should be able to obtain from your instrument. **This data is always measured with aqueous samples, and is not applicable when performing measurements in non-aqueous solvents.**

**Impact bead adjustment** The impact bead has been designed to provide the optimum aerosol particle size when used with aqueous solvents. When working with organic solvents, where the density, viscosity and volatility are quite different from water, the effect of the impact bead cannot be predicted. You should investigate the effect of the impact bead position on the signal from your sample and may well find that the optimum signal will be obtained with the impact bead moved out of the aerosol path altogether.

**Other Accessories** If you plan to use an autosampler fitted with a wash facility, ensure that you fill the wash reservoir with clean solvent.

If you plan to use the ID100 Autodilutor, ensure that you use clean solvent as the diluent.

**Maintenance**

- Check the various parts for wear and damage at regular intervals, and replace any damaged parts immediately.

<b>Spares and Consumables</b>	0.5 mm ID nebuliser uptake tubing (standard)	9423 390 05421
	0.4 mm ID nebuliser uptake tubing (reduced uptake rate)	9423 390 05411
	Solvent Resistant Drain Tubing	9423 390 05491
	Solvent Resistant O-ring Kit	9423 390 05151



## Chapter 5 Furnace Operation

**Introduction** Graphite Furnace AAS does not involve the use of large quantities of inflammable, explosive or toxic gases, and so is relatively free from hazard. To minimise the hazards involved, read and understand the contents of this section before using the system.

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## Furnace Safety

### The GFS33/35 and GFS35Z Furnace Heads

- The intense light emitted by a heated Graphite Cuvette can harm your eyes. Although you are protected from direct glare in normal operation, light escapes from the Injection Port and the Furnace Head windows. To avoid this hazard, do not operate the Furnace at high temperatures unless it is in the normal operating position in the Sample Compartment.
- The Furnace Head is water cooled, and all components will return to ambient temperature within about a minute from the end of the Furnace cycle. Nevertheless, ensure that the heating cycle has ended, and that all components have cooled before handling or opening the Furnace Head.
- The Graphite Furnace system is not capable of detecting the type of Cuvette you have fitted; you must select the correct type on the Furnace parameters software page. If you select the wrong type of Cuvette, incorrect heating regimes will be applied, which may damage the Furnace Head and/or the Cuvette.
- If a Cuvette fails in use, it may break up into small pieces inside the Furnace Head. Remove any debris that could cause a short circuit and consequent damage to the head, and clean the Temperature Control System Window and Lens before fitting a new Cuvette.

### The GFS35Z Zeeman Furnace Head

- The magnet fitted to the GFS35Z Zeeman Furnace Head produces a variable magnetic field up to 0.85 Tesla at mains frequency during the atomisation and autozero phases. This can affect other electronic systems in the vicinity.



**CAUTION:** If you use a heart pacemaker, do not stand closer than one metre from the magnet when the system is operating. ▲

- Keep all magnetic material and magnetic storage media at least 300 mm from the magnet when it is operating.
- The Magnet and Head assembly is heavy; take care if you have to lift or move the assembly, and ensure that it is securely attached to the Spectrometer before use.
- When tilting the GFS35Z on the Tilt Mount, take care to avoid trapping your fingers in the moving mechanism.

### Fume Extraction

- Hot, potentially toxic and corrosive fumes derived from the sample are vented above the Furnace Head, and must be removed by a suitable fume extraction system.
- The Furnace should only be fired when the furnace head is securely attached to the spectrometer and not tilted and there is suitable extraction.

- Gases**
- Follow the safety guidelines provided by Gas Suppliers, such as the 'Gas Data and Safety Sheet' issued by the British Oxygen Company for specific gases at all times.
  - All gas lines, fittings and regulators must be free from oil or grease.
  - Gas cylinders must be fitted with suitable pressure reducing regulators.

## Furnace Measurements in organic solvents

Before using your instrument, refer to the Safety sections of the main Operators Manual that are concerned with safe use of the spectrometer, and safe use of the Furnace atomisation system.

- Safety**
- If you wish to use organic solvents on a regular basis, we recommend that you use PTFE sample cups (9423 393 80051) and PTFE sampling capillary (94230393 82261).

### Risk Assessment

The use of organic solvents in furnace AAS is an inherently hazardous procedure. As a minimum, you should:

- unless the solvent that you are planning to use is freely miscible with water, you must remove any aqueous solutions from the autosampler before you introduce the solvent.
- use the minimum possible quantity of solvent in the vicinity of the spectrometer.
- ensure that any aqueous solution in the wash and waste bottle is removed and replaced with organic solvent before starting the analysis.
- ensure that the autosampler has been purged 5 times.
- ensure that the organic solvent is removed from the wash and waste bottle, and the vicinity of the instrument, as soon as the analysis is finished.
- empty the waste bottle frequently, to minimize the quantity of solvent in the vicinity of the spectrometer.
- ensure that the extraction system is operating properly, to minimize the risk of solvent vapors accumulating in the vicinity of the spectrometer.
- ensure that the autosampler is free from leaks
- ensure that the gas supplies are set to the correct operating pressures, as listed in the main Operators Manuals, that the pressure regulators are operating properly and that the gas connections are free from leaks.

**WARNING:** Many common laboratory solvents are unsuitable for use in Graphite Furnace. Halogenated solvents (chloroform, Freons) produce toxic gases; solvents with low flash points (ethers, light petroleum spirit) present too great a flammability hazard to be safely used, and unsaturated aromatic hydrocarbons (toluene, xylene) are unsuitable. ▲

## The GFS35 and GFS35Z Graphite Furnace Systems

- Introduction**
- The GFS35 and GFS35Z Graphite Furnace systems are major accessories that are fitted in the right hand Sample Compartment of the iCE 3400 / 3500 Series spectrometers.
  - The GFS35 and GFS35Z use an electrically heated graphite cuvette, enhancing the analytical sensitivity compared to flame atomization by 100-1000 times, and reduce the sample volume required to a few microlitres.
  - The GFS35Z Zeeman Graphite Furnace system can be used on an iCE 3400 and 3500 spectrometer to provide graphite furnace analyses with Zeeman background correction capability.

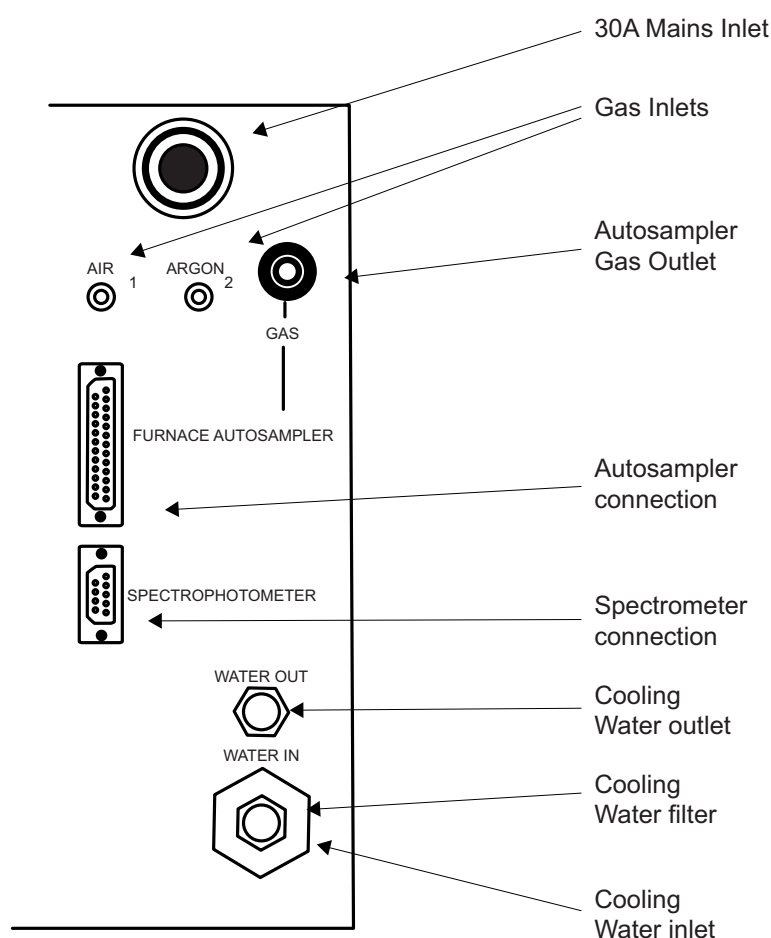
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## Services

- Inert Gas**
- This protects the hot cuvette from atmospheric oxygen, and flushes sample vapours from the cuvette interior.
  - Argon is recommended; nitrogen can be used with some loss of performance for some elements.
  - Connect the inert gas supply to the inlet port labelled ARGON 2 at the rear of the Furnace Power Supply unit.
  - The inert gas supply must be regulated to  $1.03 \pm 0.14$  bar ( $15 \pm 2$  psi).

- Alternate Gas**
- An alternate type of gas can be passed through the cuvette to modify the sample behaviour.
  - Connect the alternate gas supply to the inlet port labelled AIR 1 at the rear of the Furnace Power Supply unit.
  - The alternate gas supply must be regulated to  $1.03 \pm 0.14$  bar ( $15 \pm 2$  psi).

- Fume Extraction**
- Smoke and fumes from the sample decomposition must be extracted away from the vicinity of the instrument.
  - Refer to the Pre-Installation Manual for the specifications of a suitable extraction system.
  - Ensure that the extraction system is switched on and is operating correctly before using the furnace.



**Fig 5 - 1. Furnace Power Supply Connections Panel**

### Cooling Water

- A supply of reasonably clean (e.g. drinking) water, at a temperature of less than 30 °C and a pressure of 1.4 to 6.9 bar (20 - 100 psi), capable of providing a minimum flowrate of 0.7 l/min is required.
- Do not allow the pressure to exceed 6.9 bar (100 psi).
- Connect the cooling water inlet and outlet hoses to the water inlet and outlet connections on the Furnace Power Supply unit.

### Recirculators

- The Furnace can be cooled by a temperature controlled recirculator/chiller unit instead of mains water.
- The specifications of a suitable unit are given in the Pre-Installation Manual.
- Set the recirculating water temperature to about 5 °C above ambient temperature, providing that this is less than 30 °C.

### Installation

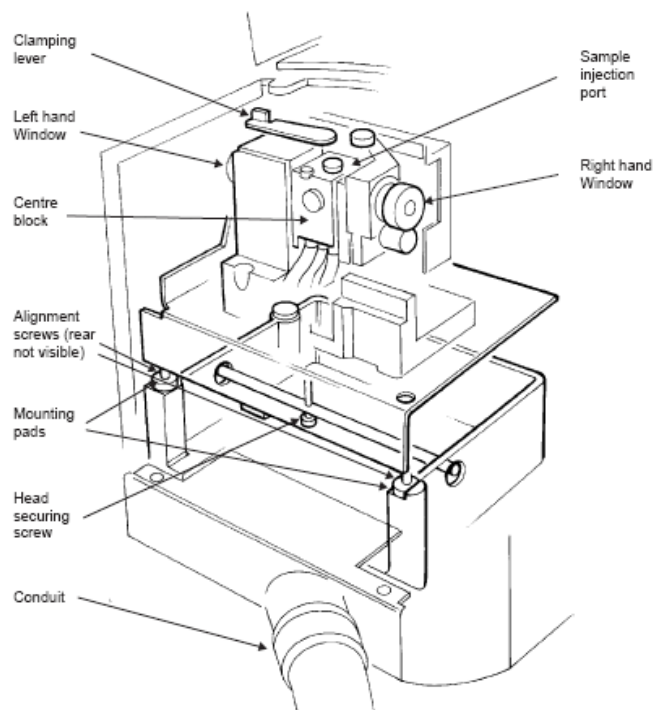
The GFS33 / GFS35 and GFS35Z Furnace Systems must be installed in your spectrometer by a trained Service Engineer.

### Furnace Head Mounts

- The GFS35 Furnace can be supplied with either a Fixed Head Mount, or a Tilting Head Mount.
- The GFS35Z Furnace is always supplied with a Tilting Head Mount.
- The Tilting Head Mount allows the furnace head to be tilted forward for maintenance and cleaning, and to allow other accessories to be fitted.

### Fitting and removing the GFS35 on the Fixed Mount

It may occasionally be necessary to remove the GFS35 from the Fixed Mount to gain better access to the head for cleaning and maintenance, or to fit an accessory in the right hand sample compartment.



**Fig 5 - 2.** GFS35 Furnace Head on a Fixed Mount



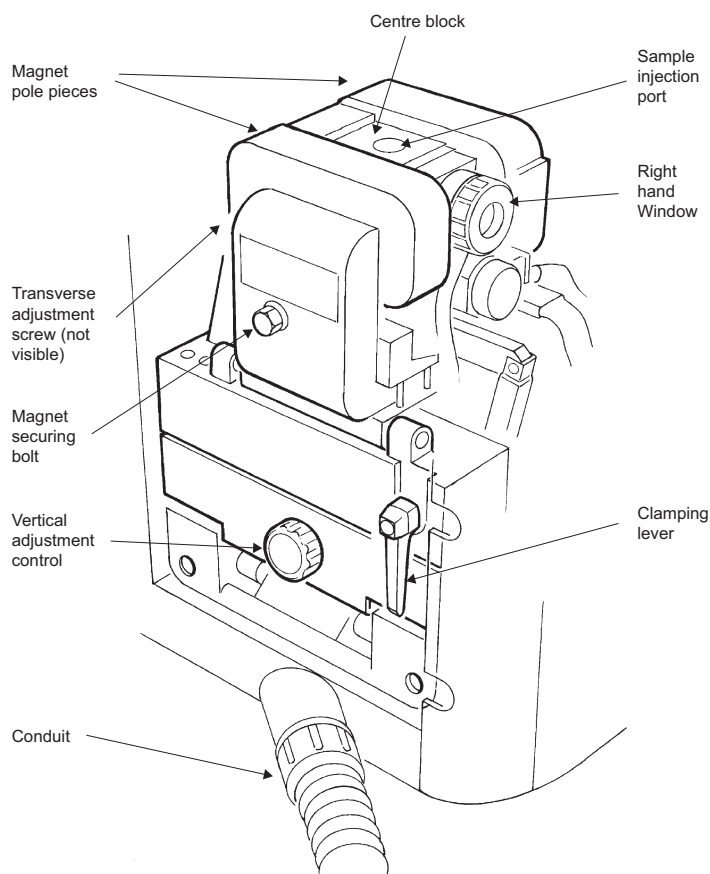
**To remove the GFS35 Furnace Head on the Fixed Mount**

1. Refer to the figure, and locate the Head Securing screw.
2. Remove the Head Securing screw and the 2 Valance screws above the Furnace Conduit.
3. Carefully lift the Head and the Mount, together with the Conduit and Valance, and place the assembly safely on the bench in front of the Spectrometer.

To refit the the head, reverse the procedure.

**Electrical Connections**

- Connect the 9 pin serial port on the Furnace Power Supply unit to the Accessory port on the Spectrometer Connection Panel, using a standard RS232C cable.
- Connect the Furnace Power Supply mains lead to a suitable 30A mains outlet.
  - Note that the GFS35Z unit requires a second mains power connection to supply the Zeeman magnet drive board.



**Fig 5 - 3. GFS35Z Furnace Head on a Tilt Mount**

**Furnace Head Optical Alignment**

The Furnace Head will have been aligned with the Spectrometer optical system when it was installed, and both types of mounting arrangements have been designed so that the alignment is not lost when the Furnace Head is moved. Furnace Head alignment should, therefore, seldom be required.

### To align the Furnace Head

1. If the Tilt Mount is fitted, tilt the Head out of the Sample Compartment. It will be helpful to remove the Furnace Head windows, although this is not essential.
2. Install a suitable hollow cathode lamp, and set up a Furnace Method. Set up the optical system in the normal way.
  - Ensure that you do **NOT** select any form of background correction.
3. Set up the system software to display the live absorbance signal from the right hand Sample Compartment, and confirm that the display shows zero. Use the auto-zero command if necessary.
4. Return the Furnace Head to its normal operating position.
5. Refer to the figure, and use the positioning controls to adjust the position of the Furnace Head to obtain a minimum in the displayed absorbance signal.
  - If you have removed the Furnace Head Windows, you should be able to position the Furnace Head so that it does not absorb any radiation i.e. the displayed absorbance will remain at zero.
  - With the Windows in place, you should be able to obtain a minimum absorbance value of below 0.1 absorbance units when the Furnace Head is correctly aligned.

### Cuvette Fitting

- Handle new Cuvettes carefully to avoid contamination.
- The GFS33, GFS35 and GFS35Z Furnace Heads will accept all types of Cuvette supplied by Thermo Fisher Scientific for use in these Heads. Do not attempt to use cuvettes from any other source, nor those not specifically designed for these Furnace Heads.

### To fit a Cuvette

1. Refer to the respective figures, and unclamp the Furnace Head by turning the Clamping Lever.
2. Remove the old Cuvette.
  - Note that if the Cuvette has failed in use, there may be soot and debris inside the Furnace Head. Remove any debris, and clean the interior of the Head as described in the Maintenance section below before fitting the new Cuvette.
3. Take the new Cuvette, and side orientate it so that the Injection Hole is pointing upwards.
4. Position the Cuvette fully in the Furnace Head, and carefully close the Clamping Lever.
5. Check that the Cuvette Injection Hole is positioned correctly. If necessary, unclamp the Furnace Head and rotate the Cuvette slightly. Use the Cuvette Tool shown in the figure to make the Injection Hole central in the Furnace Head Injection Port.
6. Zero the Cuvette Life Counter, and ensure that you set the correct Cuvette Type on the Furnace Parameters page of the System Software before attempting to use the Cuvette.

<b>Spares and Consumables</b>	Normal Electrographite Cuvettes (10)	9423 393 95031
	Pyrolytically Coated Electrographite Cuvettes (10)	9423 393 95071
	Extended Lifetime Cuvettes (ELCs) (10)	9423 393 95041
	Extended Lifetime Cuvettes (ELCs) (20)	9423 393 95051
	Omega Platform Extended Lifetime Cuvettes (10)	9423 490 20101

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## The GFS35 / 35Z System Maintenance

- Introduction**
- Furnace System maintenance must be performed regularly to ensure safe and reliable operation.
  - Contamination is the major cause of problems with the Graphite Furnace System. It is essential to maintain scrupulous cleanliness of the Furnace Head, Furnace Autosampler and all sample handling apparatus.

**Cleaning** **Furnace Head**

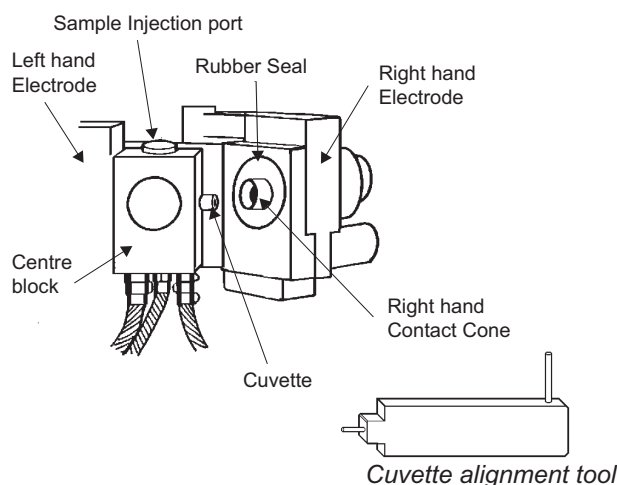
- At least once a week, open the Furnace Head, remove the cuvette, and inspect all graphite components. Use a cotton bud to remove any sample deposits or debris. An air jet can also be used to blow out any dust.
- Inspect all other Head components. Clean exterior surfaces with tissue or soft cloth moistened with dilute detergent solution.

**Furnace Head Windows**

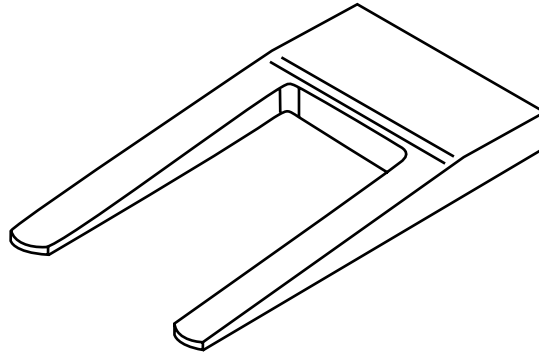
If the Furnace Head Windows become dirty, they will reduce the optical energy passing through the cuvette, and so increase the baseline noise.

**To remove and clean the Furnace Head Windows**

1. If the Furnace Head is fitted on the Tilting Mount, tilt the Head forward, out of the Spectrometer Sample Compartment. If the Furnace Head is mounted on the Fixed Mount, remove the Head from the Sample Compartment, and place it in a secure, accessible position in front of the spectrometer.
2. Take the Furnace Window Removal Tool supplied, and push it firmly into the gap between the Window Holder and the furnace body. Use the Tool to lever the Window Holder from its mounting, then remove each Window Holder by pulling it from the body of the Furnace.
3. Remove any debris or deposits from the Window Holder gas ways.

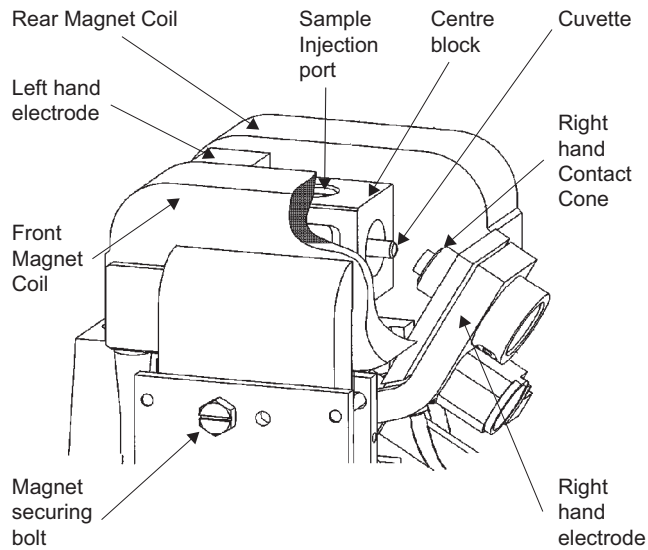


**Fig 5 - 4. GFS35 Furnace Head - interior view**



**Fig 5 - 5.** Furnace Window Removal Tool

4. Clean the Windows with a moist cotton bud.
  - The Windows can be removed from the Window Holders by pulling out the O-ring seal. Take care to avoid damaging the O-ring seals if you do this.
5. Refit the Window Holders, taking care not to damage the O-ring seals.
  - Note that the Window Holder O-ring seals should not be lubricated.



**Fig 5 - 6.** GFS35Z Furnace Head - interior view

### Optical Temperature Feedback System

Thermal radiation emitted by the hot Cuvette is used to control the Cuvette temperature when the Temperature Control command is selected. If the Temperature Control Window or Lens are dirty, temperature accuracy, analytical precision and cuvette lifetime will be degraded.

If a Cuvette has failed in use, it is likely that soot and debris will have been deposited on the Temperature Control Window. The Window should be cleaned before fitting a new Cuvette.

### **To clean the Temperature Feedback System**

1. Remove the Furnace Head Centre Block, to expose the Temperature Control Window in the base of the Centre Block, and the Lens in the body of the Furnace.
2. Clean the Lens with a cotton bud.
3. Unscrew the Window Holder from the rear of the Centre Block.
  - Take care that the Window does not fall out.
4. Inspect and clean the Window.
5. Replace the Window and Window Holder.
6. Refit the Centre Block.

### **To remove the GFS 33/ 35 Centre Block**

1. Open the Furnace Head, and remove the Cuvette.
2. Pull the Centre Block gently to the right, to free it from the locating spigots.
3. Gently pull the hoses and connections out from the body of the Furnace, until you can obtain good access to the rear of the Centre Block.
4. Reverse the procedure to refit the Centre Block.
  - When feeding the hoses and connection back into the Furnace body, ensure that none are trapped or kinked, or otherwise damaged.

### **To remove the GFS35Z Centre Block**

1. Open the Furnace Head, and remove the cuvette.
2. Tilt the Head back into its normal position in the Sample Compartment, and remove the Magnet Securing Bolt with the spanner provided.
3. Carefully tilt the Head forward.
  - Note that the front Magnet Pole Piece will now open.
4. Lift the rear Magnet Pole Piece, and secure it with the catch provided.
5. Remove the Centre Block by pulling it gently to the right.
6. Gently pull the hoses and connections out from the body of the Furnace, until you can obtain good access to the rear of the Centre Block.
7. Reverse the procedure to refit the Centre Block.
  - When feeding the hoses and connection back into the Furnace body, ensure that none are trapped or kinked, or otherwise damaged.
  - When refitting the Magnet Securing Bolt, take care not to over tighten it - finger tight plus half a turn is sufficient.

## Cuvette Contacts

After typically 6 months of use, the Cuvette Contact Cones may become worn, causing poor contact with the Cuvette, and unreproducible Cuvette temperatures.

- Inspect the Cones regularly.
  - A shallow groove in the contact area is normal, but if the contact areas show signs of pitting or burning, the Cones must be replaced.
- The Contact Cones must be replaced in pairs.
- The Contact Cones cannot be re-used once they have been removed from the Head.

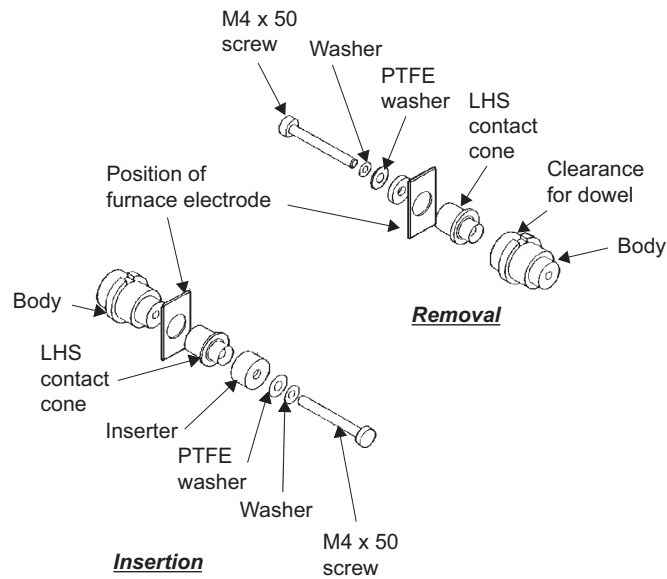
### To remove used Contact Cones

1. If the Furnace Head is on the Tilt Mount, tilt it forwards out of the Sample Compartment. If it is on the Fixed Mount, remove the Head and position it securely in front of the spectrometer.
2. Remove the Sample Injection Port Sleeve, and both Furnace Head Window Holders.
3. In the case of the GFS35Z, open the magnet.
4. Remove the Centre Block.
5. Remove the rubber Seal from the right hand Electrode
6. Referring to the relevant figure, assemble the Cone Replacement Tool, and insert it into one of the Electrodes.
  - Ensure that the body of the Tool is locked into position against the dowel stop, to prevent rotation.
7. Gently tighten the screw with the hex wrench provided, until the Cone is drawn out of the Electrode.
8. Repeat the procedure for the other Electrode.

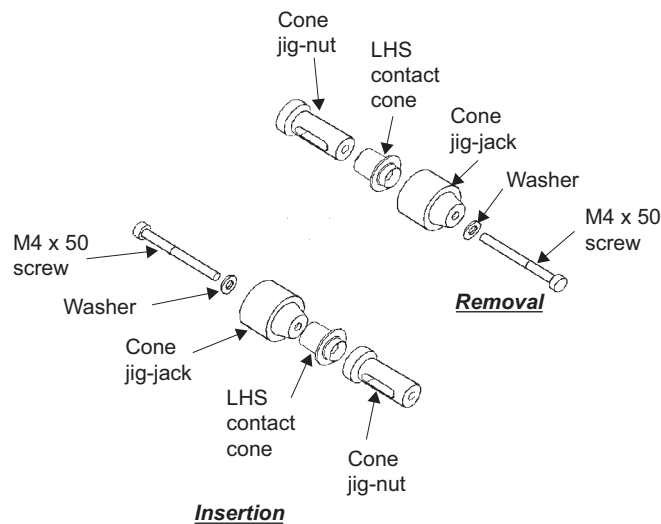
### To fit new Contact Cones

1. Referring to the relevant figure, assemble the new Cone on the Cone Replacement Tool.
2. Insert the assembly into the Electrode.
3. Fit, then gently tighten the screw until the Cone is fully home.
  - Take care not to over tighten the screw, which may damage the Cone.
4. Repeat the procedure for the other Electrode.
5. Refit the Rubber Seal in the right hand Electrode.
6. Refit the Centre Block, and close the GFS35Z magnet.
7. Refit the Window Holders and Sample Injection Port sleeve.
8. Fit a new Cuvette, and replace the Furnace Head in its normal position in the Spectrometer Sample Compartment.
9. Check the optical alignment of the Head before using it again.





**Fig 5 - 7. GFS 33 / 35 Contact Cone Replacement**



**Fig 5 - 8. GFS35Z Contact Cone Replacement**

## Leak Testing

Leaks may occur in the gas handling system or the water cooling system. Inspect your Furnace System regularly for leaks in both these areas.

### Gas System

1. Set the inert gas supply pressure to 1.03 bar (15 psi).
2. Close the cylinder valve to isolate the gas lines and equipment.
3. Monitor the line pressure shown by the cylinder gauge for 10 minutes.
4. If the pressure falls by more than 0.07 bar (1 psi), check each connection by brushing dilute soap solution on it. Bubbles will indicate the presence of a leak.
  - Ensure that you wipe off the soap solution at the end of the test, so that it does not cause corrosion of the fittings.
5. Rectify any leaks found.

**Water Cooling System**

- Water leaks usually occur at connections, and are often self-evident. Rectify them by tightening or re-making the connection.

**Internal Leaks**

- If you suspect a gas or water leak to be present inside the Furnace Power Supply, Conduit or Furnace Head, turn off all services to the equipment, and do not use it again until the leak has been located and rectified by a trained Service Engineer.

**Fuses**

The Furnace Power Supply contains some internal fuses. These must only be replaced by a trained Service Engineer. The GFS35Z Power Supply contains, in addition, two user replaceable fuses. These are located adjacent to the Zeeman magnet mains inlet, on the rear left hand panel of the Power Supply module. If these fuses fail, they can be replaced with suitable replacements, type 8A(T). If the replacement fuses fail, it is likely that a serious fault is present, and you should call a Service Engineer to diagnose and rectify the fault.

**Spares and Consumables**

Furnace Head Window (1)	9423 393 68561
Standard Furnace Contact Cone set	9423 393 95011
Standard Furnace Contact Cone Replacement Tool	9423 393 95101
Standard Furnace User spares Kit	9423 450 20002
Zeeman Furnace Contact Cone set	9423 393 95161
Zeeman Furnace Contact Cone Replacement Tool	9423 393 95181
Zeeman Furnace User spares Kit	9423 450 20003

## The GFS33 Graphite Furnace System

- Introduction**
- The GFS33 Graphite Furnace system is major accessories that can be fitted in the Sample Compartment of the iCE 3300 Series spectrometers.
  - It utilises an electrically heated graphite cuvette, enhancing the analytical sensitivity by 100-1000 times compared to flame atomization and reducing the sample volume required to a few microlitres.
  - The GFS33 accessory includes an integrated Furnace Autosampler, to automatically inject the sample solutions into the Graphite Furnace.

**Installation** Your GFS33 Furnace System must initially be installed by a trained Service Engineer, who will make all the necessary connections to the services and align the instrument ready for use. This information is included on the Maintenance pages of this section and will be useful if you have to move the system to another location, or otherwise re-install it.

### GFS33 Fitting and Removal

You will have to remove the GFS33 assembly if you want to use the flame or vapor atomisation systems on your spectrometer. When not fitted in the Sample Compartment of the spectrometer, the GFS33 should be parked between the LHS of the spectrometer and the Furnace Power Supply.

#### To remove the GFS33:

1. Identify the Securing Screw at the top rear of the mount.
2. Unscrew the Securing Screw to release the assembly from the rear wall of the Sample Compartment,
  - Take care not to disturb the setting of the Transverse Alignment nut.
3. Raise the whole assembly until it is free of the Accessory Support Bar.
4. Move the assembly towards you and to the left until it is clear of the spectrometer, then park it between the spectrometer and Furnace Power Supply.
5. Refer to the appropriate section of this manual, and if applicable, fit the flame or vapor atomisation system that you want to use.
6. If appropriate, fit the Sample Compartment Door.

#### To fit the GFS33:

1. If applicable, use the system software to move the Burner to the Parked position.
2. If applicable, refer to the appropriate section of this manual, and remove the Burner or Vapor atomisation accessory.
3. If applicable, remove the Sample Compartment Door, by lifting it off its hinges.
4. Move the GFS33 assembly towards you and to the right, then lift it slightly so that you can position it on the Accessory Support Bar.

5. Secure the GFS33 in the Sample Compartment with the Securing Screw on the rear of the mounting assembly.
  - Take care not to disturb the setting of the Transverse Alignment nut.
6. The GFS33 is now ready for use.

## Operation - Graphite Furnace

The GFS33 Furnace Head and Autosampler will have been aligned with each other and with the Spectrometer optical system at installation, and the mounting arrangements have been designed so that the alignment is not lost when the assembly is removed from and replaced in the Sample Compartment.

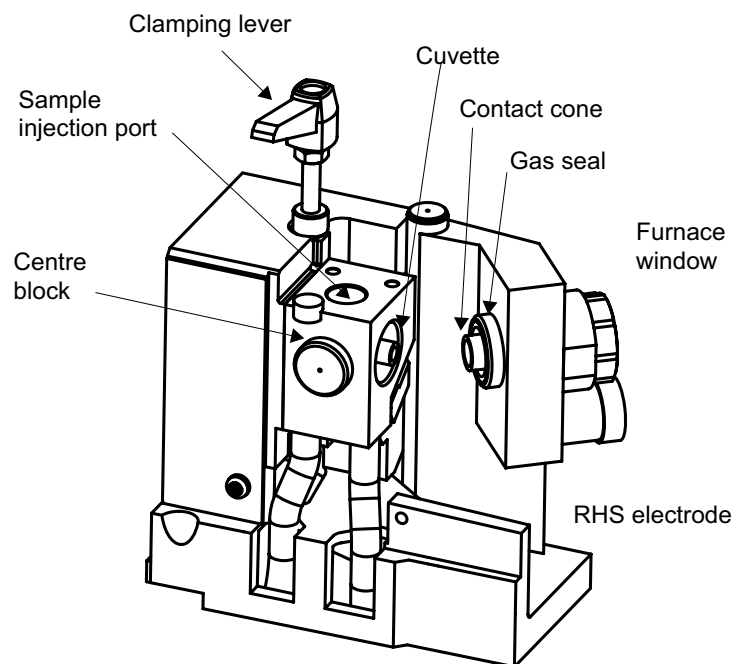
### Graphite Cuvettes

The GFS33 will accept all types of Cuvette supplied by Thermo Fisher Scientific for use in this Furnace Head.

- Do not attempt to use cuvettes from any other source, nor those not specifically designed for this Furnace Head.
- Handle new Cuvettes carefully to avoid contamination.

### To fit a Cuvette

1. Refer to the figure, and unclamp the Furnace Head by turning the Clamping Lever.
2. Remove the old Cuvette.
  - Note that if the Cuvette has failed in use, there may be soot and debris inside the Furnace Head. Remove any debris, and clean the interior of the Head as described in the Maintenance section below before fitting the new Cuvette.
3. Take the new Cuvette, and orientate it so that the Injection Hole is pointing upwards.



**Fig. 5 - 9.** GFS33 Graphite Furnace Head

3. Position the Cuvette fully in the Furnace Head, and carefully close the Head with the Clamping Lever.
4. Check that the Cuvette Injection Hole is positioned correctly. If necessary, unclamp the Furnace Head and rotate the Cuvette slightly. Use the Cuvette Tool to make the Injection Hole central in the Furnace Head Injection Port.
5. Zero the Cuvette Life Counter, and ensure that you set the correct Cuvette Type on the Furnace Parameters page of the System Software before attempting to use the Cuvette.

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## The GFS33 System Maintenance

### Introduction

- Furnace System maintenance must be performed regularly to ensure safe and reliable operation.
- Contamination is the major cause of problems with the Graphite Furnace System. It is essential to maintain scrupulous cleanliness of the Furnace Head, Furnace Autosampler and all sample handling apparatus.

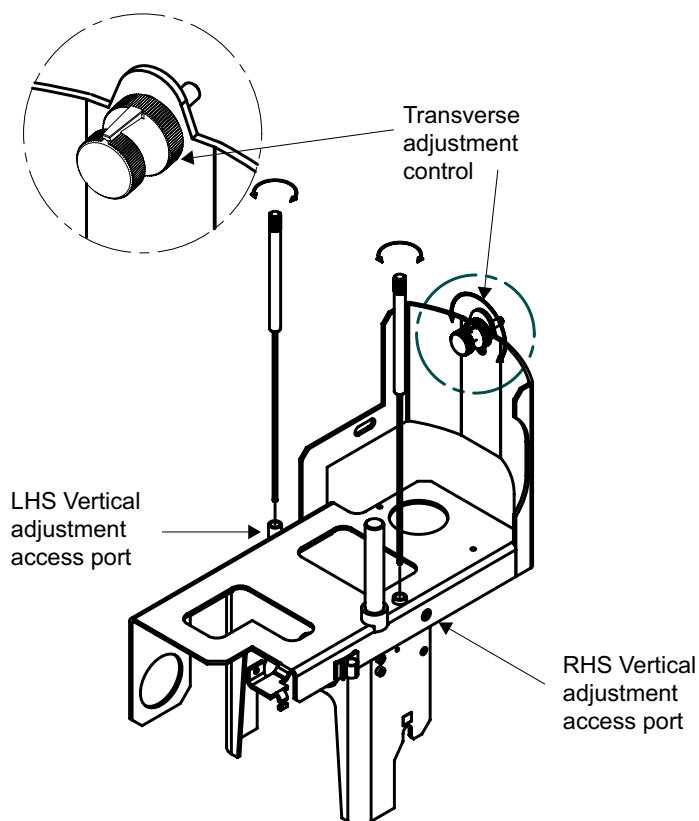
### Installation

Your GFS33 Furnace System must initially be installed by a trained Service Engineer, who will make all the necessary connections to the services and align the instrument ready for use. Installation and alignment procedures included in this section to help you if you have to move the system to another location, or otherwise re-install it.

**Alignment** Routine alignment of the GFS33 should seldom be required. However, it is good practice to check the alignment if a component has been replaced, and as part of the routine maintenance procedures for your instrument.

### To align the Furnace Head

1. Remove the GFS33 from the Sample Compartment. It will be helpful to remove the Furnace Head Windows, although this is not essential.
  - The Windows are held in place on an 'O' ring seal. Remove each Window by grasping the Window Mount, and twisting and pulling until the Window Mount is free.
2. Install a suitable hollow cathode lamp, and set up a Furnace Method. Set up the optical system in the normal way.
  - Ensure that you do NOT select any form of background correction.



**Fig. 5 - 10.** GFS33 - Furnace Adjustment Controls

3. Set up the system software to display the live absorbance signal and confirm that the display shows zero. Use the auto-zero command if necessary.
4. Replace the GFS33 in its normal operating position.
5. Refer to the figure, and use the Transverse and Vertical position adjustment controls to adjust the position of the Furnace Head so that the light beam passes through the cuvette. Then make final

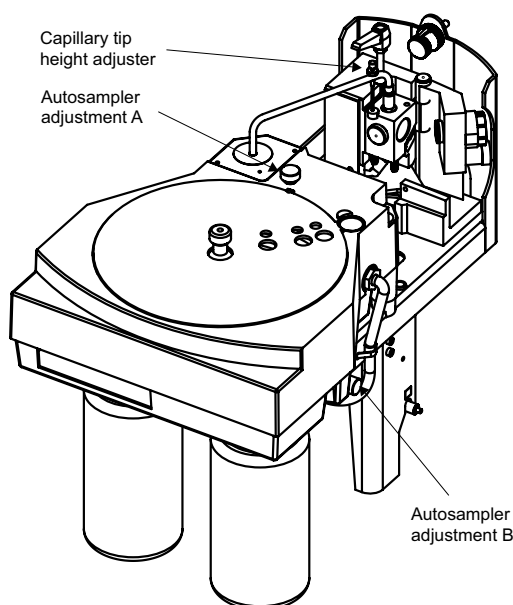


adjustments of each of the controls to obtain a minimum in the displayed absorbance signal.

- You will need the long 8 mm ball ended Allen key supplied with your Furnace System to adjust the Vertical position controls. Insert the tip of the tool in the Access Port, and push it gently downwards until it engages with the head of the adjuster screw.
- If you have removed the Furnace Head Windows, you should be able to position the Furnace Head so that it does not absorb any radiation i.e. the displayed absorbance will remain at zero.
- With the Windows in place, you should be able to obtain a minimum absorbance value of below 0.1 absorbance units when the Furnace Head is correctly aligned.

### To align the Autosampler Capillary Tip

1. Ensure that the Graphite Furnace is correctly aligned before aligning the Capillary Tip.
2. Refer to the figure, and locate the Capillary Tip Adjustment controls A and B.
3. Use the Align Capillary Tip command in the System Software to move the Autosampler Arm over the Cuvette. Do not attempt to move the Arm horizontally by hand - this may damage the mechanism.
4. Adjust the position of the Tip until it enters the Cuvette cleanly, without touching the sides of the Injection Hole.
  - Control A will move the tip diagonally across the Sample Compartment from the rear left to the front right.
  - Control B will move the tip diagonally across the Sample Compartment from the rear right to the front left.
5. Use a dentists mirror or the GFTV image to view the Capillary Tip inside the Cuvette.
6. Refer to the figure, loosen the Adjustment Lock Nut, then use the Height Adjustment Control to set the depth of the Capillary Tip



**Fig 5 - 11.** GFS33 - Autosampler adjustments

so that the Tip is approximately 1 mm clear of the bottom of the Cuvette.

- You may have to optimise the Capillary Depth Setting further, by observing a sample injection.
7. Lock the Adjustment Screw with the Adjustment Lock Nut, then use the Park Capillary Tip command to return the Autosampler Arm to its normal parked position.
  8. Align the probe automatically by selecting the “Align Probe” command in the SOLAAR software. You may need to make fine adjustments to the position of the capillary tip. Repeat if necessary.

## **Maintenance - Graphite Furnace**

Refer to the **GFS35 / 35Z system maintenance section** on page 5 - 13

## The GFS Autosampler

- Introduction**
- This is an Autosampler designed for use with the GFS33 / 35 and GFS35Z Graphite Furnace systems.
  - It provides fully automated sample injection facilities.
  - It also provides facilities for automated matrix modification, and standard and sample solution preparation.

## Installation **To fit the GFS Autosampler (GFS 35 and GFS 35Z)**

1. Fit and align the GFS35 or GFS35Z Graphite Furnace System. Ensure that the Furnace Head is in the normal operating position.
2. Lift the GFS Autosampler, and insert the supporting arms into the holes on the right of the Sample Compartment.
3. Push the Autosampler body home until the arms secure into place.

## **To remove the GFS Autosampler (GFS 35 and GFS 35Z)**

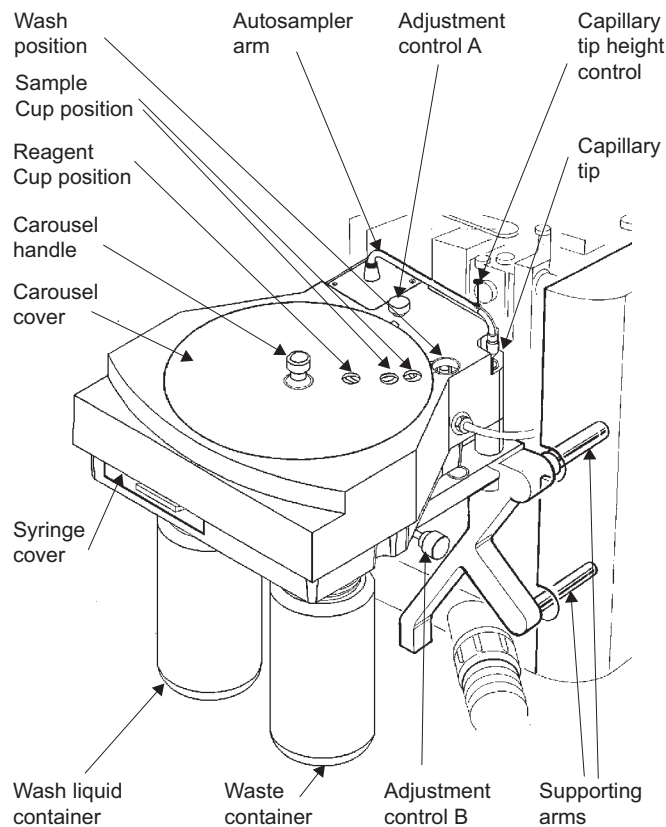
1. Ensure both the Wash and Waste containers are fitted to the Autosampler.
2. Grasp the body of the Autosampler firmly, and pull it towards you, until the supporting arms come free from the Spectrometer.
3. Place the Autosampler upright on the bench.

## **Gas Connection**

The Autosampler Wash facility is pressurised by an inert gas supply routed through the Furnace Power Supply unit.

## **To connect the Autosampler Gas supply**

1. Locate the Autosampler Gas Inlet tube.
2. Fit the quick release connector on the free end of the tube to the Autosampler Gas Port on the Furnace Power Supply connection panel.



**Fig 5 - 12.** GFS Autosampler

### Electrical Connection

The Autosampler power and control signals are generated by the Furnace Power Supply unit.

#### To connect the Autosampler to the Furnace Power Supply

1. Locate the Autosampler Connection cable.
2. Fit the free end of the cable to the Autosampler socket on the Furnace Power Supply connection panel.

### Drain Assembly

The GFS Autosampler is fitted with a Waste container to collect the used Wash liquid. This container can be replaced by a permanent connection to a suitable drain, if required.

#### To connect a permanent Drain

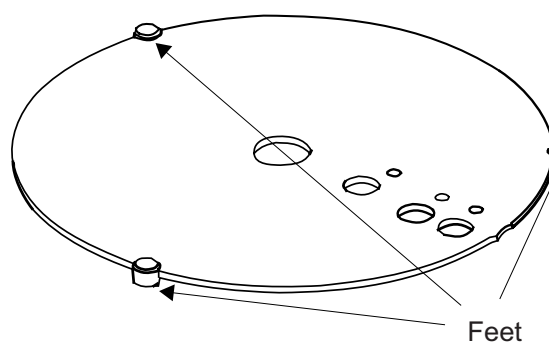
1. Remove the Waste container.
2. Locate the waste tube that discharges into the Waste container.
3. Use the connector and spare length of tygon tubing supplied to extend the waste tube as required.
  - Note that the liquid must be able to run freely down the drain tube, which must not contain kinks or liquid traps.

### Carousel Cover

The Carousel Cover protects your sample solution from airborne dust, and prevents contamination. It will also reduce evaporation of the sample solutions. In some ambient conditions, condensation can form on the lower surface of the cover, which can also contaminate the samples. You can fit small feet to the Carousel Cover to reduce the condensation on the under surface, although this will increase the sample evaporation rate. We recommend that you only fit these feet, if you have a problem with condensation.

#### To fit the Carousel Cover Elevation Feet

1. Locate the three Feet supplied.
2. Refer to the figure and fit the Feet to the Carousel Cover.



**Fig 5 - 13.** GFS Autosampler Cover

## Operation Wash Liquid

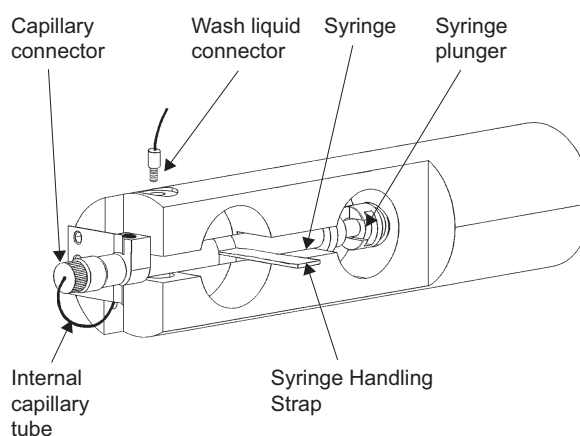
A small quantity of the Wash Liquid is pumped through the internal plumbing of the unit into the Wash Reservoir after each injection. Choose the composition of the Wash Liquid to efficiently remove traces of the sample solutions from the capillary. Dilute nitric acid (approximately 0.1% v/v) will be suitable for many aqueous sample types.

Wash Liquid contamination can be a serious problem, affecting the accuracy and precision of your results. Check that the Wash Liquid is acceptable, by analysing a sample of the liquid before use.

## Syringe Purge

A command is provided in the System Software to purge the Syringe to remove bubbles and contamination before use. You should use this command whenever the Wash Reservoir is refilled.

## Sample Cups



**Fig 5 - 14.** GFS Autosampler Syringe Assembly

Three types of Sample Cup are available for use with the GFS Autosampler. These are:

Polypropylene Cups for general use. These will contain approximately 2 ml of liquid, and are normally not re-used.

Fluoroplastic Cups for ultra-trace applications. These also contain approximately 2 ml, and can be subjected to rigorous cleaning procedures when necessary.

Reduced Volume Cups for analyses where the available sample volume is limited. These are polypropylene, and contain approximately 1.5 ml. The bottom of the cup is 'V' shaped, allowing sample volumes down to 100  $\mu$ l to be sampled reliably.

Reagent Cups are provided for use in the six larger Reagent Positions on the Autosampler Carousel. These are polypropylene, and hold approximately 25 ml.

Cup Reducing Rings are available, which allow a normal Sample Cup to be used in a Reagent Position.

### **Cleaning Autosampler Cups**

All Autosampler Cups should be cleaned by soaking in 5-10% v/v nitric acid and rinsing with clean water before use. More rigorous cleaning procedures may be necessary for ultra-trace applications.

### **Capillary Tip Alignment**

The Autosampler Capillary Tip must be aligned with the Cuvette Injection Hole before use, and the Capillary Tip Depth Adjustment set correctly so that the sample is injected properly on to the floor of the Cuvette.

#### **To align the Capillary Tip**

1. Ensure that the Graphite Furnace is correctly aligned before aligning the Capillary Tip.
2. Refer to the figure, and locate the Capillary Tip Adjustment controls A and B.
3. Use the Align Capillary Tip command in the System Software to move the Autosampler Arm over the Cuvette. Do not attempt to move the Arm horizontally by hand - this may damage the mechanism.
4. Adjust the position of the Tip until it enters the Cuvette cleanly, without touching the sides of the Injection Hole.
  - Control A will move the tip diagonally across the Sample Compartment from the rear left to the front right.
  - Control B will move the tip diagonally across the Sample Compartment from the rear right to the front left.
5. Use a dentists mirror or the GFTV image to view the Capillary Tip inside the Cuvette.
6. Refer to the figure, loosen the Adjustment Lock Nut, then use the Height Adjustment Control C to set the depth of the Capillary Tip so that the Tip is approximately 1 mm clear of the bottom of the Cuvette.
  - You may have to optimise the Capillary Depth Setting further, by observing a sample injection.
7. Lock the Adjustment Screw with the Adjustment Lock Nut, then use the Park Capillary Tip command to return the Autosampler Arm to its normal parked position.
8. Align the probe automatically by selecting the “Align Probe” command in the SOLAAR software. You may need to make fine adjustments to the position of the capillary tip. Repeat if necessary.

## **Maintenance Syringe**

If persistent bubbles or leaks occur, it may be necessary to remove the Syringe and inspect it for blockage or damage.

#### **To remove the syringe**

1. Ensure that the Autosampler Arm is in the parked position, by using the Park Autosampler command in the System Software.
2. Locate the Syringe Compartment on the Autosampler front panel.
3. Remove the Syringe from its mount by carefully pulling the Syringe

Handling Strap. Take great care not to damage the Syringe or Plunger during this operation.

4. Unscrew the capillary tube connections and remove the Syringe.

Reverse this procedure to refit the Syringe. Take particular care not to trap any of the capillary tubing between the Syringe or Connector bodies and the Autosampler.

### Internal Capillary tubing

You can replace the capillary tube between the Syringe and Capillary Tip if it should become blocked or damaged, although this is unlikely to happen in normal use.

### To replace the Internal Capillary Tubing

1. Remove the Capillary Tip and Locking Cap.
2. Pull the exposed Capillary Tubing gently from the arm, and remove the PTFE cone.
3. Remove the Syringe, and disconnect the Capillary Tubing connector.
4. Gently pull the old Capillary Tubing out from the Autosampler.
5. Feed the new Capillary Tubing into the Autosampler, until the end emerges from the Autosampler Arm.
6. Trim the free end of the Tubing to an acute angle with a sharp scalpel blade, and feed it through a new PTFE cone.
7. Pull the tubing through the cone, and trim it flush with the flat face of the cone.
  - Take great care not to damage the flat sealing face of the cone.
8. Push the cone back into the Arm, and refit the Capillary Tip.
9. Attach the new Capillary Tubing to the Syringe, and refit the Syringe.
10. Thoroughly purge the system before use, to remove any bubbles and contamination.

### Capillary Tip

The Autosampler Capillary tip can be replaced if it becomes bent or contaminated.

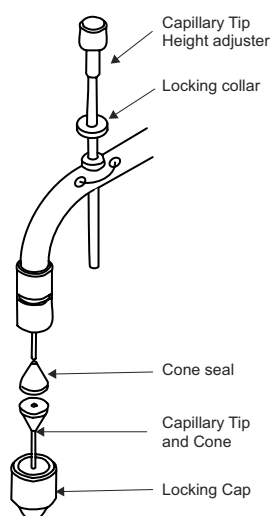


Fig. 5 - 15. Capillary Tip detail



**To replace the Autosampler Capillary Tip**

1. Refer to the figure, and gently lift the Autosampler Arm. Do not attempt to move the arm horizontally, as this may damage the arm drive mechanism.
2. Carefully unscrew the Locking Cap, and remove the old Capillary Tip.
3. Fit a new Capillary Tip into the Locking Cap, and refit the Cap to the Arm.

**Spares and Consumables**

Polypropylene Sample Cups (1000)	9423 393 80031
Fluoroplastic Sample Cups (20)	9423 393 80051
Reduced Volume Sample Cups (20)	9423 393 80061
Reagent Cups (50)	9423 393 80021
Cup Reducing Ring	9423 393 80071
Capillary Tips (10)	9423 393 81261
Internal Capillary Tubing	9423 393 82261
Spare Carousel and Tray	9423 393 83261
Furnace Auto-sampler User Spares Kit	9423 450 20004
Floor Standing 5L Waste Vessel	9423 390 05471

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# Chapter 6 Vapor Operation

- Introduction**
- The gaseous products of the hydride reaction used in Vapor AAS are combustible and may be toxic. Ensure that your fume extraction system is working correctly before using the technique, and that all gas and liquid connections are secure and free from leaks.

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## Vapor Safety

### Reagents

- Sample solutions and reagents used in Vapor AAS are corrosive and may be toxic. Ensure that you take all necessary precautions when handling these materials.
- Reagents are contained in the Reagent Containers supported on the rear of the unit. If it is necessary to move the unit, ensure that the Reagent Containers are empty, or are removed to prevent spillages, before moving the unit.
- The sodium borohydride reagent used is unstable, and will gradually evolve hydrogen on standing. Ensure that any containers used for this reagent are suitably vented, and do not store the reagent for any length of time.

### Flame Heated Atomisation Cell

- Before using a Flame Heated Atomisation Cell, refer to the Flame Safety Page of this manual, and ensure that you fully understand the hazards involved in using the flame.
- Never attempt to use a nitrous oxide supported flame to heat the Atomisation Cell.
- Flame Heated Atomisation Cells can reach temperatures in excess of 1100 °C during use. Do not handle the cell or the cell holder until they have cooled for at least 15 minutes after the flame has been extinguished.
- Ensure that the Atomisation Cell is in the parked position away from the flame before attempting to ignite or extinguish the flame.

### Electrically Heated Atomisation Cell

- The Electrically Heated Atomisation Cell can reach temperatures in excess of 1100 °C during use. Do not open the furnace or handle the cell for at least 15 minutes after the power to the furnace has been switched off.



## The VP100 Continuous Flow Vapor Accessory

### Introduction

- The VP100 is an accessory for measuring hydride-forming elements (and mercury) with better analytical sensitivity than can typically be obtained with flame or furnace atomisation.
- The analyte elements in the sample solution are reduced to volatile hydrides using sodium borohydride, and are carried in a carrier gas stream to a heated Measurement Cell for measurement.
- The Measurement Cell is normally heated by an air supported acetylene flame. The EC90 Electrically Heated Atomisation Cell accessory can also be used.
- The VP100 can be used to measure mercury, as well as the hydride group elements. As it is not necessary to use heat for mercury measurements, the VP100 includes a Mercury Absorption Cell, which can be used in place of the normal 'T' cell. The Mercury Cell will improve the sensitivity of the system for mercury measurements by a factor of about 1.3x relative to the standard 'T' cell.

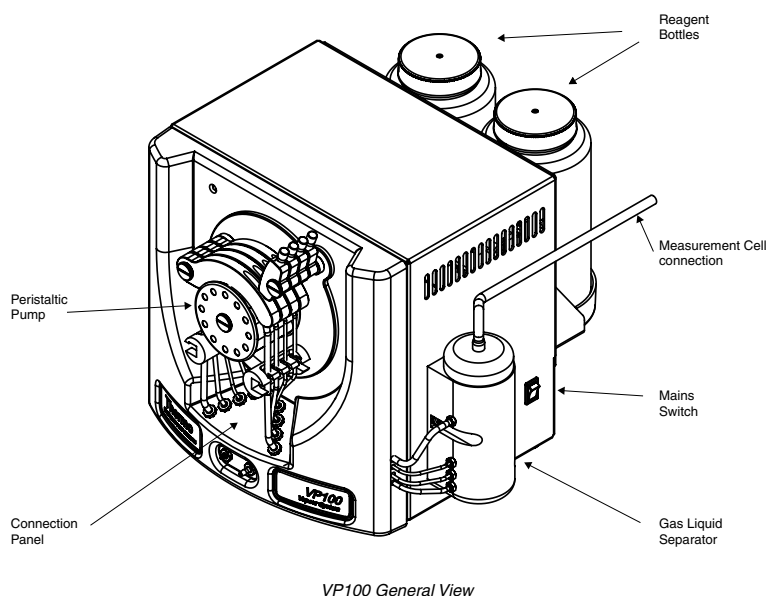


Fig 6 - 1. VP100 General View

### Power

- Connect the unit to a suitable mains supply via the mains cable supplied.

### Control Signals

- Connect the Serial Cable supplied between the RS232C connection on the rear of the unit and a free Accessory port on the Spectrometer Connections panel.

### Gas Supply

- Using the black nylon tubing supplied, connect a supply of argon or nitrogen, regulated to a pressure of 0.34 bar (5 psi), to the gas inlet at the rear of the unit.

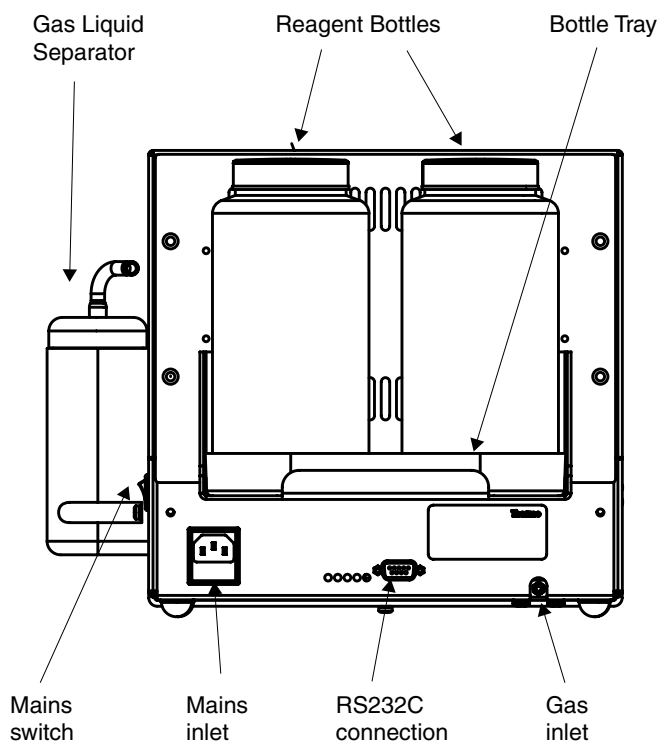


Fig 6 - 2. VP100 Rear View

### Drain

- Connect a suitable length of the 3.2 mm ID Tygon drain tubing supplied, from the Drain Outlet on the front of the unit, identified with a BLACK connector, to a suitable low level drain or wide necked plastic container.

**WARNING:** Do not mix the waste from Vapor Analysis with waste from other analysis. ▲

### Main Unit

- Support the main unit to one side or in front of the Spectrometer, adjacent to the Sample Compartment that you will be using.
- Ensure that you can access the main power switch on the RHS of the unit.
- Best results will be obtained when the distance between the Gas Liquid Separator and the Measurement Cell is kept as short as possible.

### Sample Uptake

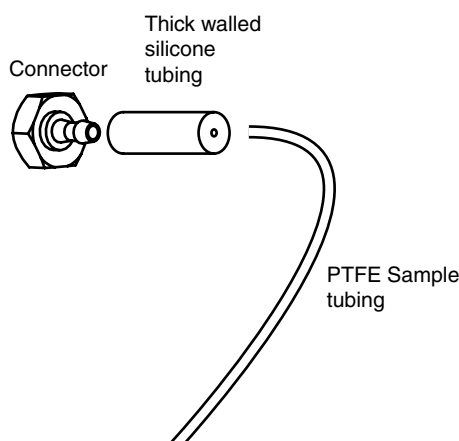
The Sample Uptake tubing must be attached to the GREEN Sample Uptake connector on the front panel of the VP100 using a short piece of the thick walled silicone tube supplied.



- **Manual sampling.** Take a suitable length of the PTFE Sample Uptake tubing supplied, and connect it to the VP100 Sample Uptake connector.
- **Autosampler.** Connect the tube from the autosampler sample probe to the VP100 Sample Uptake Connector.
  - If you are using a CETAC autosampler, we recommend that you use the larger 0.8 mm ID sample probe and uptake tube supplied with the autosampler.

**To connect the Sample Uptake tube:**

1. Locate the thick walled silicone tubing supplied, and cut off a piece 15-20 mm long.
2. Grip the sample uptake tube firmly and push it into one end of the silicone tubing, until 10 mm is inside the silicone tubing.
  - Use a small piece of abrasive paper to grasp the sample uptake tube more firmly.
  - Use a drop of water to lubricate the tubing, if necessary.
3. Push the free end of the silicone tubing over the GREEN push-on connector on the VP100 front panel.



**Fig 6 - 3.** Sample Uptake tubing connection

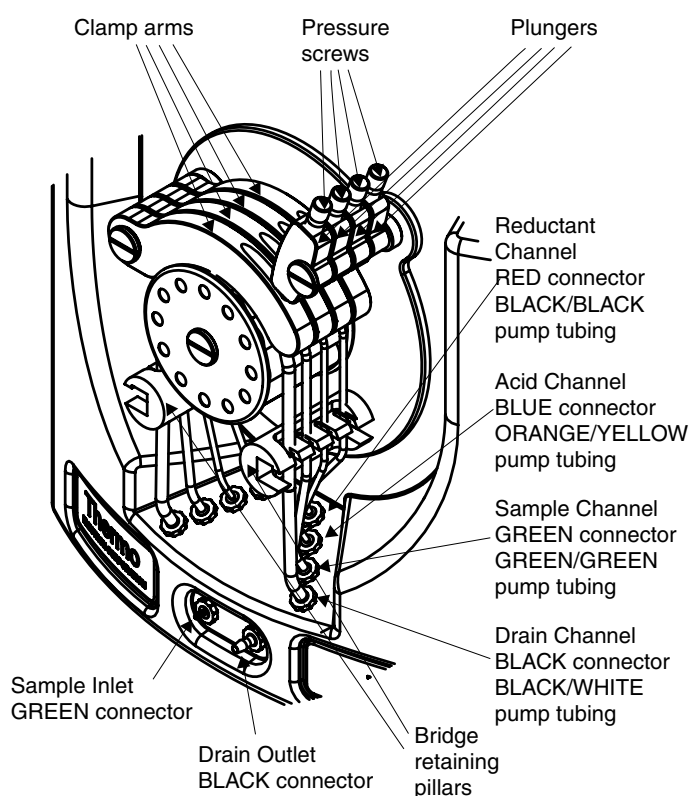
**Pump Tubing**

The VP100 is fitted with a four channel Peristaltic Pump. The channels are identified by the colour coded connectors on the pump tube connection panel of the unit, as follows:

BLACK channel	Drain
GREEN channel	Sample
RED channel	Reductant reagent
BLUE channel	Acid reagent

- The BLACK (Drain) channel must be fitted with pump tubing with BLACK/WHITE bridges i.e. a single piece of pump tubing will have a black bridge fitted at one end, and white bridge fitted at the other end. This tubing has an ID of 3.17 mm and an OD of 4.85 mm.

- The GREEN (sample) channel must be fitted with pump tubing with two GREEN bridges. This tubing has an ID of 1.85 mm and an OD of 3.53 mm.
- The RED (Reductant) channel must be fitted with pump tubing with two BLACK bridges. This tubing has an ID of 0.76 mm and an OD of 2.43 mm.
- The BLUE (Acid) channel must be fitted with pump tubing with YELLOW/ORANGE bridges. This tubing has an ID of 0.5 mm and an OD of 2.33 mm.

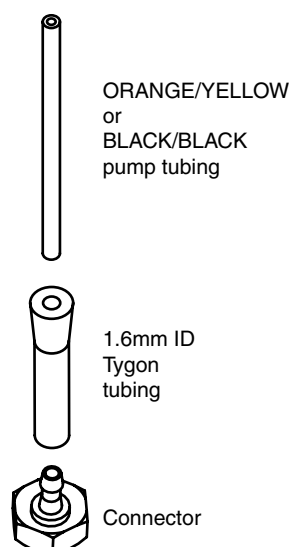


**Fig 6 - 4. VP100 Connection Panel and Peristaltic Pump**

The internal diameters of the pump tubing with the BLACK bridges, and the pump tubing with the YELLOW/ORANGE bridges are too small to allow the pump tubing to be attached directly to the connectors on the VP100 connection panel. You must fit a short length of the 1.6 mm ID Tygon tubing supplied over the ends of the pump tubing, then fit that to the VP100 connectors.

**To attach 1.6 mm ID Tygon tubing to the pump tubing:**

1. Take a piece of 1.6 mm ID Tygon tubing, and expand one end.
  - A disposable pipette tip is ideal for this, but any other object with a tapering point can be used.
2. Take the pump tubing, and insert 3 - 5 mm inside the expanded Tygon tubing.
  - Lubricate the tubes with a drop of water, if necessary
  - You can use a small piece of abrasive paper to grasp the tube securely.



**Fig 6 - 5.** Pump tubing connection

3. Trim back the Tygon tubing to 15 mm or so.
4. Repeat for the other end of the pump tubing.

#### **To fit the pump tubing**

1. You will find that it is most convenient to fit the pump tubes from the rear of the connection panel towards the front, in the following order:
  1. Reductant channel (RED connectors, pump tubing with BLACK bridges).
  2. Acid channel (BLUE connectors, pump tubing with YELLOW/ORANGE bridges).
  3. Sample channel (GREEN connectors, pump tubing with GREEN bridges).
  4. Drain channel (BLACK connectors, pump tubing with BLACK/WHITE bridges).
2. Release the plungers to free the clamp arms, then move the arms anti-clockwise to reveal the pump rollers.
3. Take the pump tubing, and feed it around the pump rollers.
4. Stretch the pump tubing slightly, and fit the bridges under the bridge retaining pillars.
5. Push the ends of the tubing over the appropriate push on connectors on the VP100 connection panel.
6. Move the clamp arm back over the rollers, and confirm that the tubing is properly located beneath it.
7. Return the plunger to the normal position, and adjust the pressure screw to release the pressure on the tubing.

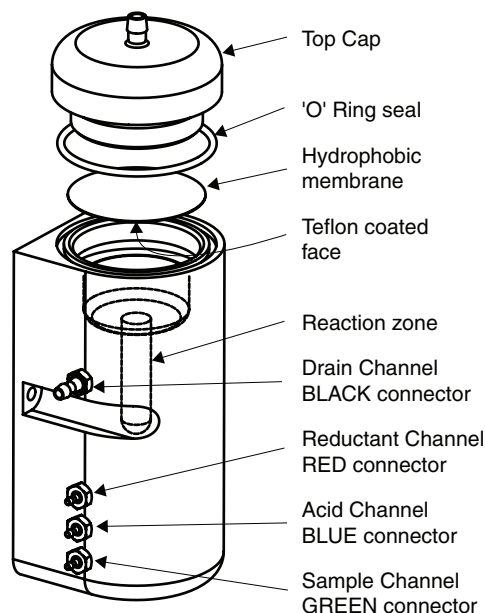
#### **To adjust the peristaltic pump tube pressure:**

1. Turn on the Spectrometer, Data Station and VP100. Turn on the inert gas supply to the VP100.
2. Place the Reagent Uptake tubes and the Sample Uptake tube in a container of clean water.
3. Ensure that the Drain tube discharges to a suitable low level drain or drain vessel.

1. Ensure that the clamp arms are correctly positioned, and that the plunger arm pressure screws have been fully released.
2. Use the system software to set up a Vapor Method using the VP100, and set an appropriate value for the Carrier Gas flow rate. Set the Pump Speed to 30 RPM.
3. Use the commands on the Action menu in the system software to turn on the VP100.
4. Tighten the pressure screws in each plunger arm in turn until each channel starts to pump.
  - Do not overtighten the pressure screw - this will shorten the life of the pump tubing and may damage the pump mechanism.
  - The flow rate in the Drain channel is significantly greater than the total flow rate of the three other channels. In normal operation there will be bubbles present from the evolved reactant gases in the pumped fluid in the Drain channel.
  - The flow rates in the Acid and Reductant channels are low, and so it will take a short time before the fluid reaches the pump. Do not attempt to set the final pressure on the tube until the fluid has filled the pump tubing.

### Gas Liquid Separator

The Reaction Zone of the Gas Liquid Separator contains 4 mm glass beads, which minimise the dead volume of the zone and ensure proper mixing of the carrier gas and liquid reagents. It also contains an hydrophobic Teflon membrane to prevent moisture and salts from being carried over into the Measurement Cell. These parts are supplied with the VP100, and must be fitted before the accessory is used.



**Fig 6 - 6.** VP100 Gas Liquid Separator

### To prepare the Gas Liquid Separator

1. Refer to the figure, and unscrew the Top Cap of the Gas Liquid Separator.

1. 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.
  - Take care to prevent the glass beads from falling into the Drain, where they may cause blockage.
3. Take one of the 47 mm Teflon membranes supplied. Orientate it so that the Teflon covered face is on the underside, and place it in position.
4. Carefully re-fit the 'O' Ring seal and Top Cap, ensuring that the position of the membrane is not disturbed.

### Flame Heated Measurement Cell

The Flame Heated Measurement Cell ('T' Cell) is supported over the Universal Burner by the 'T' Cell Holder.

**Note:** The STAT Holder and 'T' Cell Holder are the same device. If you have already fitted a STAT holder to your Burner, this will also support the 'T' Cell.

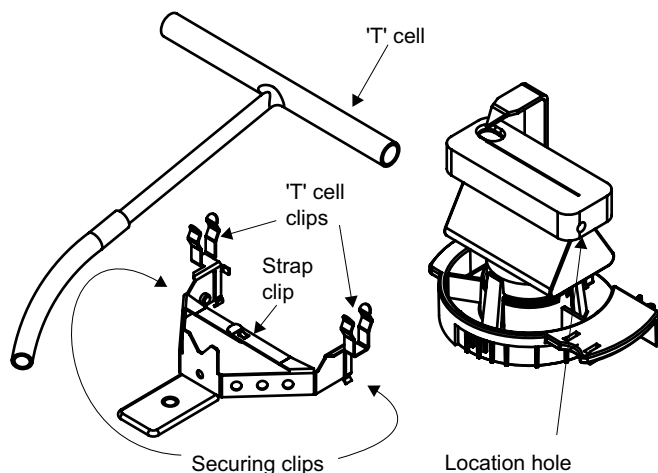


Fig 6 - 7. 'T' Cell Holder Assembly

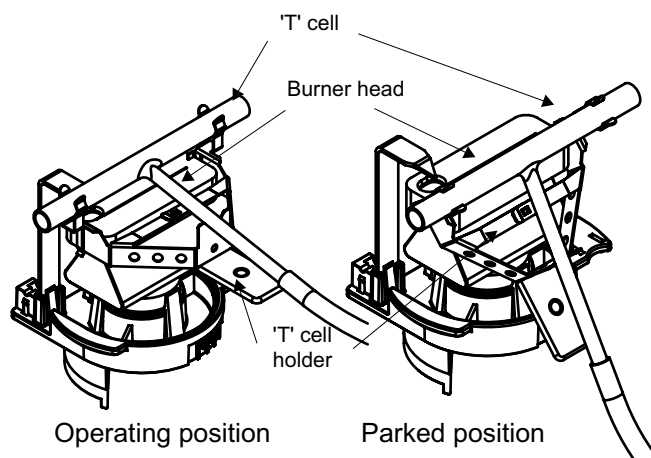


Fig 6 - 8. 'T' Cell and Holder

### **To fit the Cell Holder to the burner:**

1. Remove the Burner from the Spectrometer.
2. Orientate the Burner with the Burner Handle to the right and the Ignition Electrode to the left.
3. Unclip the Strap Clip on the 'T' Cell Holder, and spread the Securing Clips slightly.
4. Gently push the 'T' Cell Holder assembly on to the Burner until the Securing Clips click into the Locating holes on the Burner.
5. Re-clip the Strap Clip together, to secure the Holder to the Burner.
6. Refit the Burner to the Spectrometer.
7. Clip the 'T' Cell into the 'T' Cell Holder Clips.

### **'T' Cell Alignment**

1. Fit the Burner, Cell Holder and 'T' Cell assembly to the Spray Chamber Stem.
2. Move the Cell Holder so that the 'T' Cell is in the Parked position, away from the Burner slot.
3. Install a suitable hollow cathode lamp, and perform an optical set up.
4. Move the Cell Holder so that the 'T' Cell is in the Operating position over the Burner slot.
5. Use a piece of white card at the right hand side of the Sample Compartment to locate the light beam.
6. Adjust the Burner position until the light beam passes along the axis of the 'T' Cell.
7. Move the 'T' Cell away from the Burner slot.

### **Electrically Heated Atomisation Cell**

The EC90 Electrically Heated Atomisation Cell can be fitted to either the L.H. or R.H. Sample Compartment of the Spectrometer, and must be mounted on the appropriate Universal Mount. Instructions for fitting and aligning the Cell are provided in the EC90 section of this manual.

### **Mercury Absorption Cell**

You can measure mercury with the VP100 using the standard 'T' cell, although it will not be necessary to heat the cell. However, you will obtain improved sensitivity and precision by using the Mercury Absorption Cell supplied with the VP100.

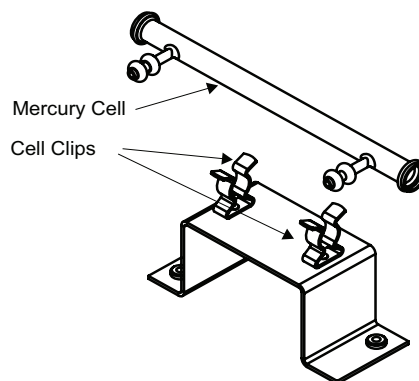
The Mercury Absorption Cell can be fitted in either Sample Compartment of the Spectrometer, and should be mounted on the appropriate Universal Mount.

- It is possible to fit the Mercury Absorption Cell to the standard 'T' cell holder. This is not recommended, as it is then possible to light a flame, which will destroy the Mercury cell.

### **To fit the Mercury Absorption Cell to the Universal Accessory Mounts:**

1. Locate the Mercury Cell Holder shown, and fit it to the required Universal Mount.

2. Refer to the Universal Mount pages in the Spectrometer section of this manual, and fit the assembly in appropriate Sample Compartment.
3. Fit the Absorption Cell in the Cell Clips of the Cell Mount.
4. Refer to the Universal Mount pages in the Spectrometer section of this manual, and align the assembly in the Sample Compartment.



**Fig 6 - 9.** Mercury Absorption Cell and Cell Holder

#### **Absorption Cell connection**

The 'T' cell or Mercury Absorption cell must be connected to the Exit port at the top of the Gas Liquid Separator of the VP100.

#### **To connect the Absorption cell:**

1. Cut a suitable length of the 8 mm OD Tygon tubing provided.
  - Use the shortest possible length of tubing.
  - If you are using the LH (Flame) sample compartment, you can feed the tubing through the aperture in the sample compartment door, so that you can make your measurements with the door closed.
2. Connect the tubing between the Top Cap connection on the Gas Liquid Separator and the inlet of the 'T' Cell or Mercury Cell.
  - You can loosely plug the other arm of the Mercury Cell with a small piece of cotton wool, to prevent the ingress of dust. This will also increase the sensitivity, but will also increase the washout time.

#### **'T' Cell Conditioning**

A new 'T' cell may require conditioning before you obtain stable measurements.

#### **To condition a new 'T' cell**

- Run a high concentration standard (typically 1-10 mg/L) for at least 5 complete measurement cycles, followed by a blank to wash out the high concentration standard. The signal may not return to baseline. Wait for the signal to stabilize and then perform an autozero.

## Reagents

The optimum composition of the reagents used depends upon the element being measured and the composition of the sample. The reagent compositions described below will be suitable for the analysis of simple aqueous solutions.

### Acid Blank Reagent

This reagent is suitable for all type of measurement.

- 50 % v/v (6M) hydrochloric acid solution.

### Borohydride Reductant Reagent

This Reagent is suitable for all hydride elements, and for mercury.

- 0.5 % m/v sodium tetrahydroborate (sodium borohydride,  $\text{NaBH}_4$ ), stabilised in 0.5 % m/v sodium hydroxide ( $\text{NaOH}$ ).
- The Reagent should be filtered through a coarse filter paper immediately after preparation.
- The Reagent slowly evolves hydrogen gas, and so must be stored in a vented container.
- The Reagent will remain usable for 2-3 days if stored in a refrigerator at 4 °C, but it is recommended to prepare it daily, immediately before use.

### Stannous Chloride Reductant reagent.

This reagent is suitable for mercury analyses only.

- 0.1 - 10 % m/v of stannous chloride ( $\text{SnCl}_2$ ) in 1 - 10 % v/v hydrochloric acid.
- If you have pre-reduced your sample with hydroxylamine hydrochloride, or if your sample contains little or no oxidising residues, you can use the lower concentrations of stannous chloride.
- We strongly recommend the use of Low Mercury grades of stannous chloride, which are available from most major chemical suppliers.
- Dissolve the stannous chloride in the concentrated hydrochloric acid, with gentle heating. The stannous chloride should dissolve to form a clear solution. Cool, and make to volume with deionised water. the final solution should be clear. A milky suspension indicates hydrolysis of the stannous chloride and should not be used.

## Parameter Optimisation

The VP100 has three parameters that control its operation:

- The Carrier Gas Flow rate.
- The Pump Speed.
- The Measurement Delay time.

Default values are provided for these parameters, can altered in the System software, on the Vapor page of the Method.

### VP100 Carrier Gas Flow rate

For each hydride group element, there is an optimum Carrier Gas Flow rate that will give the maximum sensitivity for that element. For mercury, the analytical sensitivity increases as the Carrier Gas Flow rate decreases. Low Carrier Gas Flow rates require longer Measurement Delays. Higher Carrier Gas Flow rates allow the use



of shorter Measurement Delays, at the expense of some analytical sensitivity, and can be used if you want to complete your analysis as quickly as possible. Typical default values are in the range 100 - 200 mL/min. It is unlikely that flow rates higher than 300 mL/min will be useful.

### **VP100 Pump Speed**

As the VP100 Pump Speed is increased, the analytical sensitivity increases, but so does the consumption of samples and reagents. The Measurement Delay required will be decreased at higher pump speeds. Reducing the pump speed reduces the reagent consumption at the expense of the analytical sensitivity and increased Measurement Delay time. The default pump speed is 30 RPM, and good results can be obtained with pump speeds up to 45 RPM.

### **VP100 Measurement Delay**

A certain amount of time is required to allow the sample solution to reach the Gas Liquid Separator and for the reduction reaction to stabilise, so that a stable analytical signal can be measured. This is the Measurement Delay time. The optimum Measurement Delay is determined by the Carrier Gas Flow rate and the Pump Speed, but will typically be 40 - 60 seconds.

### **To optimise the VP100 parameters**

You will need:

- Freshly prepared Reagents.
  - A blank solution.
  - A test sample that should give a signal of between 0.1 and 0.4 A.
1. Fill the VP100 Reagent Bottles, and place the Reagent Uptake tubes into the correct containers. Place the Sample Uptake tube into a container of clean water.
  2. Install a suitable hollow cathode lamp, and set up or load a suitable Method.
  3. If you are using Flame Heating, move the 'T' cell to the Parked position, light the flame, then move the 'T' cell to the Operating position. If you are using Electrical Heating, turn on the EC90, set the furnace temperature, and wait until the furnace temperature stabilises.
  4. Perform an Optical Setup operation, and open the Spectrometer Status Window to display the live absorbance signal.
  5. Open the Vapor Status Window.
  6. Use the commands on the VP100 submenu on the Actions menu to start the VP100.
    - The Vapor Status Window will update to show the actual pump speed and carrier gas flow rate.
  7. Wait until the Reagents have filled the Gas Liquid Separator Mixing Zone. If necessary, perform an Autozero to reset the signal display to 0.000 A.
  8. Place the Sample Uptake tube in your test sample. After 30 - 60

- seconds, the absorbance signal should rise and stabilise.
9. Use the commands on the VP100 submenu to adjust the Carrier Gas Flow rate and the Pump Speed to obtain the optimum signal for your analysis.
    - As you change the Carrier Gas Flow rate and Pump Speed, the Vapor Status display will be updated. The parameter values in the Method will also automatically be updated to reflect the changes that you make.
  10. When you have obtained the optimum signal for your analysis, place the Uptake tube in the Blank solution, and wait until the displayed signal returns to the baseline.
  11. Place the Uptake tube into the test solution, and measure the time required for the signal to stabilise.
    - You can use the Running Signal display facility of the Spectrometer Status Window to help you with this. Refer to the On Line Help system provided with the Data Station software to learn how to access and use the Running Signal display.
    - If you are using an autosampler, you may need to add a few seconds to the Measurement Delay that you have measured, to allow for the longer length of Uptake tube used with the Autosampler.
  12. The time period that you have measured is the Measurement Delay time that you should set in the Measurement Delay parameter field on the Vapor page of the Method.

## **Sample Measurement**

When you are satisfied that you have successfully set up the VP100, and have optimised the measurement parameters, you can use the system to measure your samples.

### **Sample Preparation**

Successful analyses using the Hydride Generation technique depend critically on the sample preparation procedure used. It is not possible in this manual to provide details for all types of samples, but the following factors should be considered:

1. It is normal practice to acidify samples for trace metal analysis, and many types of sample require acid digestion to bring them into a suitable form for analysis. The hydride generation technique is most successful when the samples are presented in a hydrochloric acid solution, and every effort should be made to design a sample preparation procedure that ends with the final solution in hydrochloric acid.
2. The acid concentration in the samples affects the size of the analytical signal. Samples, blanks and standards should therefore all contain the same concentration of acid. A final acid concentration of 10 % v/v (1.2M) will be suitable for many analyses.
3. The sensitivity of the Hydride Generation technique depends on the oxidation state of the analyte element, and the lower

oxidations states (As(III), Sb(III), Bi(III) SeI(V), and Te(IV)) are more sensitive. It is therefore normal practice to pre-reduce the analytes before analysis.

- As and Sb can be pre-reduced with a potassium iodide/ascorbic acid mixture.
  - Se and Te can be pre-reduced by boiling in 6M hydrochloric acid for ten minutes.
  - The Bi(V) oxidation state is unstable, and all bismuth solutions contain Bi(III). Bismuth analyses therefore do not normally require pre-reduction.
4. Many transition metals (such as copper and nickel) interfere with the reduction reaction. Refer to the SOLAAR On-line Cookbook for further information.
  5. Some samples may contain mercury in the form of organo-mercury compounds. The Stannous Chloride reductant reagent will only reduce ionic mercury, and the degree to which the Borohydride reductant reacts with organo-mercury compounds varies with the type of compound. It is therefore normal practice to use an oxidative digestion for samples for mercury analyses, to convert the organo-mercury compounds to ionic mercury.

#### **To measure samples with the VP100 Vapor Kit**

1. Install and align all parts of the VP100 Vapor Kit.
2. Prepare your samples, standards and blank solutions.
3. Prepare the Reagents required and place them in the Reagent bottles.
4. Turn on the Spectrometer, Data Station and VP100. Install suitable hollow cathode lamps in the Spectrometer.
5. Turn on the gas supplies to the Spectrometer and VP100, and confirm that the VP100 Drain discharges into a suitable container.
6. Set up a suitable Method. Turn on the hollow cathode lamp, and/or perform an Optical Setup, and allow the lamp to warm up.
7. If you are using a Flame Heated cell, ignite the flame and move the 'T' cell into the Operating position. If you are using the Electrically Heated cell, turn on the EC90 and wait until the furnace temperature has stabilised.
8. Optimise the Method parameters if necessary.
9. If you are using an Autosampler, refer to the Autosampler Loading Guide, and place your samples and standards into the autosampler racks.
10. Start the VP100, and allow it to run until the Reagents reach the Gas Liquid Separator, and are pumped away through the Drain channel.
11. Start your analysis by clicking the Analyse button, or selecting the Analyse command on the Actions menu in the usual way.
  - Note that the VP100 does not monitor the amount of Reagents in the Reagent bottles, nor detect if the Reagents are being pumped into the Gas Liquid Separator. Especially when working with an autosampler, you must periodically check that the Reagent bottles have not been emptied.

- While the analysis is running, you can use the Pause button to halt analysis, if, for example, it is necessary to re-fill the Reagent bottles. You can then use the VP100 Run and Stop commands to ensure that the Reagents have reached the Gas Liquid Separator before using the Continue button to complete the analysis.

## To shut down the VP100

When your analysis has finished, you must shut down the VP100 properly if you do not intend to use it again immediately.

1. Move the 'T' cell to the Parked position, and extinguish the flame, or turn off the power to the EC90 furnace.
2. Place the Reagent and Sample Uptake tubes in clean water, and run the VP100 until all the samples and reagents have been flushed from the tubes, and clean water is being discharged from the Drain.
3. Remove the Uptake tubes from the water, but allow the VP100 to run for another few minutes to remove the liquid from the tubes.
4. Stop the VP100, and flip open the Plunger arm pressure screws to release the pressure on the pump tubes.
5. Empty and rinse the Reagent bottles. Wipe up any liquid that may have been spilled on or around the VP100.
6. Turn off the inert gas supply to the VP100, and turn off the power to the accessory.
7. When all the parts have cooled to room temperature, remove the 'T' cell and EC90 furnace, if necessary.
  - If you are using Flame Heating, the 'T' cell Holder can be left attached to the Burner in the Parked position, where it will allow the Burner to be used normally.

## Consumables

'T' Cells (2)	9423 390 60101
VP100 Pump Tubing set	9423 460 10011

## VP100 Maintenance

### Introduction

- Routine maintenance mainly concerns keeping the apparatus clean. If it is necessary to clean the casework, use a mild, water based detergent. Do NOT use any form of solvent based cleaner, as it may damage the plastic front cover.
- Some of the Reagents used are corrosive. To reduce potential corrosion and hazards:
  - All spills must be wiped up immediately.
  - All leaks must be rectified immediately.
- Problems can be caused by blockages formed from dried Reagents and Sample solutions. To minimise these problems:
  - Remove all Reagents from the instrument when it is not being used.
  - Flush all liquid paths with clean water at the end of your analysis.

### Peristaltic pump

- After extended use, the pump tubing will become worn and must be replaced.
- You should inspect the tubing at regular intervals by removing it from the pump, and examining the part of the tube that is in contact with the pump rollers. If the tubing appears to be stretched, or does not appear to have a circular cross section, it should be replaced.

**NOTE:** The tubing in the Acid channel will gradually become opaque. This does not affect normal operation, and is not an indication that the tubing requires replacement. ▲

- After you have replaced the pump tubing, or if you suspect that there is a blockage, you should check the flow rates of the Reductant, Acid and Sample channels.

### To check the flow rates in the Acid, Reductant and Sample channels

1. Place the Uptake tubes in a container of clean water.
2. Start the VP100 at a Pump Speed of 30 RPM, and allow it to run until the water has filled the Gas Liquid Separator, and is being discharged from the Drain.
3. Take a 10 mL measuring cylinder, and fill it to the 10 mL mark with clean water.
4. Stop the VP100. Place the Uptake tube of the channel that you want to test into the measuring cylinder.
5. Start the VP100, and measure the volume of liquid drawn from the measuring cylinder in 1 minute.

The flow rates should be:

<b>Acid channel</b>	0.7 mL/min
<b>Reductant channel</b>	1.6 mL/min
<b>Sample channel</b>	7.5 ml/min
<b>Drain channel</b>	14 mL/min

To check the flow rate in the Drain channel, you can remove the Top Cap and Semi-Permeable Membrane of the Gas Liquid Separator, then pour clean water into the body of the Gas Liquid Separator and measure the volume of liquid discharged from the Drain outlet in 1 minute. It is unlikely that the Drain will become blocked in normal operation.

## **Gas Liquid Separator**

The Gas Liquid Separator is unlikely to become worn or damaged in normal operation, but may become dirty, and so require cleaning.

### **To clean the Gas Liquid Separator**

1. Remove the Top Cap and Hydrophobic Membrane.
2. Inspect the internal volume of the Gas Liquid Separator, and remove any deposits.
3. Confirm that the Reaction Zone is filled with 4 mm glass beads.
4. Fit a new Hydrophobic Membrane, and refit the 'O' ring seal and Top Cap.

## **Fuse Replacement**

The VP100 contains two main fuses located in the mains inlet connector. It is unlikely that these fuses will fail in normal operation, but if they do, you can replace them. The specification of the fuses is detailed on the label adjacent to the mains inlet connector. You must ensure that the replacement fuse matches this specification.

If the replacement fuse fails immediately, or if the equipment does not work normally after the fuse has been replaced, it is likely that a serious fault exists. Isolate the unit from the mains power and gas supplies, and ensure that it is not used until the fault has been diagnosed and rectified by a trained Service Engineer.

## **Consumables and Spares**

Set of VP100 Pump Tubing	9423 460 10011
Spare Hydrophobic Membrane	9423 460 10021
Spare 4 mm Glass Beads for GLS	9423 460 10031

## The EC90 Electrically Heated Atomiser

- Introduction**
- This is an accessory for use with the VP100 Continuous Flow Vapor Accessory.
  - It replaces the Flame Heated Measurement Cell with an Electrically Heated Atomisation Cell, so that Vapor measurements can be made without a flame.
  - It can be fitted in either Sample Compartment, using either the Right Hand or Left Hand Sample Compartment Universal Mount.
  - The accessory consists of two parts:
    - the EC90 Furnace Head.
    - the EC90 Power Supply.

- Safety**
- The Electrically Heated Atomisation Cell can reach temperatures in excess of 1100 °C during use. Do not open the Furnace or handle the Cell for at least 15 minutes after the power to the Furnace has been switched off.
  - Open the furnace head using the insulated furnace handles only. Remove the T-cell with great care, handling only the end of the silica inlet tube or Tygon hydride tubing.

**WARNING:** The T-cell wide body may still be hot - avoid contact. ▲

## Installation The EC90 Power Supply

1. Place the Power Supply at the front or side of the Spectrometer.
2. Confirm that the voltage selector on the rear panel matches your mains voltage.
3. Connect the Power Supply to a mains socket.

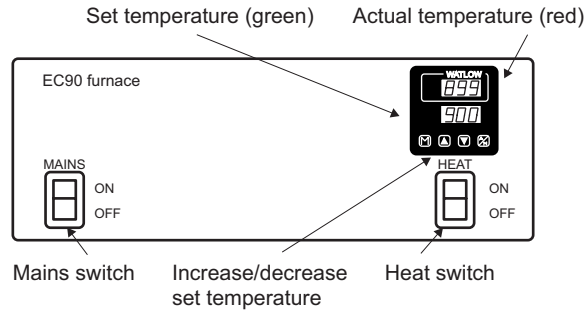


Fig 6 - 10. EC90 Front Panel

## The EC90 Furnace Head

1. Fit the Furnace Head to the appropriate Mount using the two securing screws on the base of the Furnace. Ensure that the power cable is fed through the hole in the RH Universal Mount, to emerge from the front of the instrument.
2. Locate the pink Sensor Lead, and connect it to the pink Sensor Plug on the Furnace Head.
3. Refer to the Spectrometer section of this manual, and fit the Furnace Head and Mount assembly in the required Compartment of the Spectrometer.

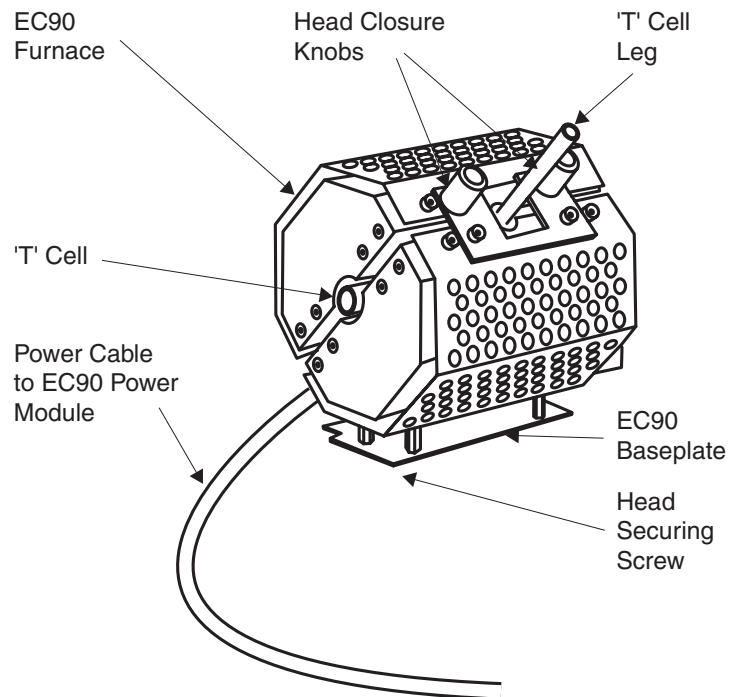


Fig 6 - 11. EC90 Furnace Head



4. Connect the Power Cable from the Furnace Head to the Head socket on the back of the Power Supply.
  - Push the plug into the socket, then give it a quarter turn clockwise, until it clicks into position.
  - To disconnect the plug from the socket, pull the metal locking piece towards the cable to release the lock, then rotate the plug anti-clockwise and pull it from the socket.
5. Fit the Sensor Lead to the Sensor socket on the back of the Power Supply.
6. Loosen the Furnace Head Closure knobs, and open the Furnace Head.
7. Fit a quartz 'T' Cell, then close the Furnace Head.
8. Refer to the relevant Universal Mount section of this manual, and align the assembly with the optical system of the Spectrometer.
9. Connect the 'T' cell to the Gas Liquid Separator outlet connector of the vapour accessory.

### **Operation** To perform a vapour analysis with the EC90, you must:

1. Set a suitable atomisation temperature (typically 900 °C).
2. Turn on the EC90 Furnace and allow the temperature to stabilise.
3. Set up the vapour accessory and perform the analysis. Please refer to the appropriate section of this manual to learn how to do this.

### **To set the atomisation temperature:**

1. Ensure that the Heat power switch is turned off, then turn on the main power switch on the front panel of the Power Supply.
  - The Controller Display will then be illuminated.
2. Set the required temperature on the lower (green) display, using the up and down arrow buttons.
  - Recommended values for the atomisation temperature for each element are provided in the On-Line Cookbook and Method Manual.
  - The upper (red) display will indicate the approximate ambient temperature.
3. Ensure that the vapour system Carrier Gas is flowing through the Atomisation Cell.
4. Turn on the Heat power switch.
  - Power will be applied to the Furnace Head, and the red temperature display will increase.
5. When the red and green temperature displays are the same, the Furnace Head is ready to use.

**Maintenance** The EC90 Power Supply is fitted with thermal overload protection. If this should trip, the power supply to the Furnace Head will be cut off, and the Overload Reset switch on the back of the Power Supply will move to the Off position.

### **To reset the thermal overload protection**

1. Ensure that the Heat power switch is turned off.

2. Move the Overload Reset to the On position.
3. Turn on the Heat power switch.

If the thermal overload protection repeatedly trips out, it is likely that there is a serious fault with the accessory. Disconnect all power to the accessory, remove it from the Spectrometer, and do not use it again until the fault has been rectified by a trained Service Engineer.

## Optimisation **Furnace Operation Temperature**

For default operation temperatures for your particular vapour analysis refer to Table I.

**Table I**

Default operating Temperatures for the EC90 in the determination of Hydride forming elements by vapour generation.

Element	EC90 Operating Temperature (Default)
Arsenic As	900 °C
Antimony Sb	900 °C
Selenium Se	900 °C
Germanium Ge	900 °C
Tellurium Te	900 °C
Lead Pb	900 °C
Tin Sn	900 °C
Mercury Hg	Ambient EC90 non operational

Further improvements in system performance may be possible through manual optimisation of the atomisation temperature. Using a standard solution expected to give an absorbance of approximately 0.20, vary the furnace operating temperature until the maximum signal response is observed. Ensure that the furnace temperature has achieved equilibrium prior to each Vapor measurement.

## Trouble Shooting **Furnace Non Operational:**

- Check that the power supply is connected and switched ON.
- Check that the power cable is connected between the furnace head and the back of the furnace power supply/control box
- Check that the thermocouple cable is connected between the furnace head and the back of the furnace power supply / control box.
- Check that the correct operating temperature is programmed into the local controller.
- Check that the power/controller unit has been switched ON. Refer to MAINS and HEAT ON/OFF Switches.
- Check that the power trip on the rear panel of the power supply is in the operating position.

## Consumables and Spares **Silica Atomisation T Cells (Pack of 2) 9423 390 60101 14**

# Chapter 7 Specifications

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## **iCE 3400 / 3500 Series Spectrometer Specification Models**

### **iCE 3500 Spectrometer**

Dual Atomiser Zeeman AA Spectrometer with wide range photomultiplier.  
Flame Atomisation in LH Sample Compartment.  
Zeeman or standard Furnace Atomisation in RH Sample Compartment.  
Vapour atomisation in either Sample Compartment.

### **iCE3400 Spectrometer**

Vapor atomisation in the RH Sample Compartment  
Zeeman Furnace AA Spectrometer in RH Sample Compartment with wide range photomultiplier.

### **Light Source**

Single or multi-element hollow cathode lamps.  
Uncoded and Data Coded hollow cathode lamps.  
6 independent lamp supplies, each providing 0 to 20 mA.

### **Wavelength Range**

180 nm to 900 nm.

### **Data/Control Systems**

#### **Spectrometer**

512kB Flash PROM.  
256kB RAM.  
3 general purpose RS232C ports.  
USB Port

#### **Data Station**

Refer to data station control section.

### **iCE 3500 Configuration**

Double Beam Stockdale optical system.

### **iCE 3400 Configuration**

Self-referencing Single Beam Zeeman system.

### **Monochromator**

Echelle monochromator and prism post-monochromator.  
Nominal 1.0, 0.5 and 0.2 nm spectral bandpass with 0.1 nm available below 400 nm.  
0.5 nm/mm reciprocal linear dispersion at 200 nm.

### **Absorbance Range**

-0.150 A to 3.000 A (including background signal).

### **Background Correction**

Quadline (continuum source) is standard on all spectrometers.  
Background signals <2 A corrected to <2 %.  
Total signal up to 3 A.

**Gas Control**

Fuel Flow Range  
Air/Acetylene - 0.8 to 2.3 l/min.  
Nitrous Oxide/Acetylene - 3.6 to 5.1 l/min.

**Power**

100 to 240 V ( $\pm 10\%$ ) at 50 or 60 Hz.

**Power consumption**

300 VA.

**Dimensions (mm)**

788W x 527H x 595D.

**Weight**

Approximately 54 kg.

**iCE 3300 Series  
Spectrometer Specification**

**iCE 3300 Spectrometer**

AA Spectrometer with wide range photomultiplier.  
6 Lamp Automatic Carousel.  
Flame and/or Furnace options available.

**Light Source**

Single or multi-element hollow cathode lamps.  
Uncoded and Data Coded hollow cathode lamps.  
6 independent lamp supplies, each providing 0 to 20 mA.

**Wavelength Range**

180 nm to 900 nm.

**Data/Control Systems**

Spectrometer  
512 kB Flash PROM.  
256 kB RAM.  
3 general purpose RS232C ports.  
USB Port

**Data Station**

Refer to data station control section.

**Optics**

**iCE 3300 Configuration**

Double Beam Stockdale Optical system

Monochromator

Ebert configuration with nominal 0.2, 0.5 and 1.0 nm spectral bandwidths.  
Half height slit settings available.  
Reciprocal linear dispersion 1.5 to 2.0 nm/mm.  
Focal length 270 mm.

**Grating**

1800 l/mm

**Absorbance Range**

-0.150 A to 3.000 A (including background signal).

**Background Correction**

Quadline (continuum source) is standard on all spectrometers.

Background signals &lt;2 A corrected to &lt;2 %.

Total signal up to 3 A.

**Gas Control****Fuel Flow Range**

Air/Acetylene - 0.8 to 2.3 l/min.

Nitrous Oxide/Acetylene - 3.6 to 5.1 l/min.

**Power**100 to 240V ( $\pm 10$  %) at 50 or 60 Hz.**Power consumption**

300 VA.

**Dimensions (mm)**

575W x 510H x 595D.

**Weight**

Approximately 33 kg.

**Description of AA Systems**

Fully functional AA system with complete multi-element capability, controlled by a Data Station running SOLAAR Software under Windows. Flame absorption/emission systems can be extended to Graphite Furnace or Vapour absorption modes by ordering the appropriate accessory atomiser.

**Optical system**

All systems (except the iCE 3400) are fitted with Stockdale double beam optics giving maximum signal-to-noise ratio during the signal measurement. All mirrors are silica coated, and the optics are sealed to prevent dust ingress. The self-calibrating Echelle monochromator and prism post-monochromator provide automatic wavelength and bandpass set-up. Quadline background correction is fitted as standard to all instruments. Both the Zeeman and the high energy Quadline background correction system correct for up to 2 A of background with <2 % error. All iCE 3000 Series spectrometers provide a 6 position data-coded, auto aligning lamp carousel, with 6 independent power supplies modulated at 200/240Hz. Automatic analyses with up to 16 element Methods are possible, and separate Methods can be sequentially run in each Sample Compartment as a Dual Analysis. The wide range PMT covers the wavelength range from 180 nm to 900 nm. GFTV is available as an option on all furnace systems.

**Flame System** All AA flames are supported by the Universal Finned 50 mm titanium burner with chamfered slot design giving high solids handling, excellent flame stability and low carbon build-up. The burner height is automatically adjustable, and controls are provided to adjust the burner rotation and transverse position. An optional 100 mm burner for air/acetylene flames is also available. Automatic flame ignition is provided by a high tension electric spark. Flame systems are controlled by a fully automatic gas control system using programmable array state logic and binary flow switching technology for reliability. Oxidant gas changeover, and fuel and oxidant flow rates are software controlled and interlocked to prevent operation with incorrect burners and gas flows. A flame sensor, responding to all flame types ensures safe gas handling if the flame goes out. All active gas lines are fitted with flame arrestors and blow back valves, with pressure sensors to provide automatic safe shutdown if the line pressure falls below the safe limits. The gas control system shuts down safely if the power fails. The totally enclosed, draught proof "kitchen" area is fitted with a safety door containing a heat and UV absorbing window.

The inert fluoroplastic spray chamber contains an adjustable impact bead, flow spoiler and low memory hydrophilic disc front end, with built in over pressure relief and automatic empty drain protection. The standard spray chamber configuration is suitable for all flame and sample types. The standard inert nebuliser has a Pt/Ir capillary and PTFE venturi, and is factory optimised.

**Graphite Furnace Systems** Graphite Furnaces are fitted in the RH sample Compartment of the iCE 3400/3500 Series Spectrometers and the LH sample Compartment of the iCE 3300. The iCE 3500 will accept either the GFS35 or GFS35Z Furnace systems while the iCE 3400 Spectrometer will only accept the GFS35Z Furnace. The iCE 3300 will only accept the GFS33.

The GFS35Z Zeeman Furnace is mounted on a pre-aligned tilt mount, which can be tilted forward out of the sample compartment to allow access to the furnace for maintenance purposes, and to allow other accessories to be fitted into the sample compartment.

The GFS35 furnace is fitted with a pre-aligned fixed mount as standard, but can be optionally fitted with the tilt mount if required.

The GFS Furnace Autosampler is mounted in front of the RH Sample Compartment with a simple self-locating 'push in' mount on the iCE 3400 / 3500. The GFS Furnace Autosampler is integrated into the GFS33 and does not require separate mounting.



**Graphite Furnace TeleVision (GFTV)**

Standard feature on the iCE 3400 / 3500 and the furnace only iCE 3300 (3300 GF). GFTV is optional on the flame only iCE 3300 (iCE 3300 FL) Spectrometers. The GFTV accessory provides high definition images of events inside the Graphite Furnace cuvette, allowing monitoring of the sample injection, dry and ash phases of the furnace program. A CMOS camera mounted in the instrument produces live video for display on the Data Stations screen.

**Electronics**

Spectrometer electronics are based on the Motorola 68340 microprocessor, with the system firmware residing in Flash PROM. Firmware can be downloaded to the Flash PROM from an external PC without removing any covers. Interfacing to the Data Station is via USB. Accessories including autosamplers, furnace and VP100 interface to the spectrometer is via RS232C at 9600 baud.

**Data Station Control**

The spectrometer system and accessories are controlled via a Data Station running the SOLAAR AA Software Package under the Microsoft Windows XP Professional, Vista Ultimate or Windows 7 Professional operating systems. The Data Station is a Personal Computer, with a minimum configuration of:

800MHz Pentium processor or equivalent.

512MB RAM.

CD ROM Drive.

Fixed Disk Drive with at least 1GB free space

19" SVGA Colour Monitor.

USB Port,

Parallel Port.

Windows XP Professional SP2, Vista Ultimate or Windows 7 Professional.

**Data Station Software**

The SOLAAR Data Station Software will control all iCE 3000 Series spectrometers. The software is aware of the type of spectrometer that it is controlling, and will only provide relevant parameters and options for each instrument type and configuration. The Data Station Software supports all the facilities provided by all the AA Series Accessories,

The software features a range of Wizard based procedures and a tabbed Methods dialogue to make setting up even complex analyses fast and easy. All raw data, results and parameters are stored in a single database for easy retrieval. True Windows multi-tasking is available, so that SOLAAR can be iconised while running an analysis, allowing concurrent use of other applications. Methods can be created, edited and saved, and earlier results can be reviewed, edited and printed, while an analysis is underway.

SOLAAR software enables full 16 element Methods to be performed automatically. The flexible and comprehensive tabbed Methods dialogue box guides the user through setting up complex, multi-element analyses. Data coded HCLs are fully supported, and facilities are provided to record lamp usage. Up to 10 calibration standards and a blank can be used, with segmented curve, linear or quadratic least squares fit

## Specifications

calibration algorithms. Normal, standard addition and standard curve calibration methods are supported, with full graphical display and print out of the calibration curve. Full alpha-numeric sample details including sample mass and dilution correction can be entered for each sample, or imported via text files.

Comprehensive furnace signal graphics are provided, and automated ash-atomise plots produce graphical data for furnace program optimisation, and will even suggest optimum values. For flame systems, fully automatic fuel gas flow and burner height optimisation with graphical presentation are provided.

Extensive online, context sensitive Help facilities are available. Carefully structured "books" allow easy browsing and searching as well as direct access to any Help topic. Trouble-shooting and diagnostics sections describe simple experiments to first locate the problem then to rectify it. A full "Cookbook" with default parameters for all elements, common interferences, and the methods to overcome them is included.

SOLAAR software provides comprehensive printer support through the Windows operating system. Report formatting enables customisation of the sample details, results, summaries, methods, system parameters and graphical displays that are to be included in the Report. All Reports, as well as individual results, are time and date stamped. Flexible and comprehensive Results database filters select and display the required data, which can be exported to other applications for further processing. Fully reversible results editing is available and is automatically audit trailed. Password protection and the Audit Trail maintain the validity of the data for accreditation purposes.

Integrated Auto QC software provides comprehensive Quality Control protocols automation with checking, testing and re-analysis options, SPC graphical results display, report generation and data filing. QC Blanks, QC Checks, QC Duplicates and two forms of QC Spikes are all included, each with user definable test criteria and failure actions. Full QC reporting, including pass/fail results and time and date stamps is provided on the Results display, and hard copy reporting of all QC protocols, parameters, results and actions is available. QC data is linked to the results to which it applies, and can be exported with the results.

SOLAAR software is available in English, USA English, French, German, Russian, Spanish, Japanese, Chinese and Polish languages.

An additional software package, SOLAARsecurity, is available, which adds to the SOLAAR Data Station software the tools, functions and facilities required to comply with the Food and Drug Administration 21 CFR Part 11 Rule "Electronic Records, Electronic Signatures" .

**ASX 520 Flame Autosampler****Standard Items**

Random access x-y autosampler mechanism.  
Standards rack (10 positions).  
4 sample racks (60 positions each).  
10 standard and 240 sample polypropylene sample tubes.  
0.5 mm ID and 0.8 mm ID PTFE sample probes.  
Pumped wash facility.  
RS232C adaptor.

**Necessary Additional Items**

None.

**Power**

100V - 240 V at 50 or 60 Hz.

**Power consumption**

50 VA.

**Description**

Random access, large capacity flame and vapour autosampler with 84 to 360 maximum sample capacity, dependent on rack configuration. Optional racks for 21, 24, 40, 60 or 90 samples.

**ASX-260 Flame Autosampler****Standard Items**

Random access x-y autosampler mechanism.  
Standards rack (10 positions).  
2 sample racks (60 positions each).  
10 standard and 240 sample polypropylene sample tubes.  
0.5 mm ID and 0.8 mm ID PTFE sample probes.  
Pumped wash facility.  
RS232C adaptor.

**Necessary Additional Items**

None.

**Power**

100V - 240 V at 50 or 60 Hz.

**Power consumption**

50VA.

**Description**

Random access, medium capacity flame and vapour autosampler with 42 to 180 maximum sample capacity, dependent on rack configuration. Optional racks for 21, 24, 40, 60 or 90 samples.

**ID100 Autodilutor**

**Standard Items**

ID100 Autodilutor.

**Necessary Additional Items**

None.

**Power**

110V - 240 V at 50 or 60 Hz.

**Power consumption**

85 VA.

**Description**

The ID100 Autodilutor provides rapid, in-line preparation of calibration standards and intelligent or fixed ratio dilution of samples for flame measurements. It is based on a high precision multi-piston pump that is calibrated for life, and does not require any consumable items. It can be used with manual sampling or with any of the flame autosamplers.

**GFS33, GFS35 and GFS35Z  
Furnace Head and Power Supply**

**Standard Items**

Standard (GFS33/35) or Zeeman (GFS35Z) Furnace Head.  
Power supply.  
Tilt Mount (GFS35Z) or Fixed Mount (GFS35).  
Intergrated Furnace and Auto Sampler mount (GFS33)  
Normal Electrographite cuvettes (10).

**Necessary Additional Items**

Additional cuvettes as required.

**Contacts**

End-loaded graphite.

**Containment**

All graphite.

**Cuvettes**

Normal Electrographite.  
Extended Lifetime (ELC).  
Pyrolytically Coated Electrographite.  
Omega Platform Extended Lifetime.

**Cuvette Volume**

0 - 70  $\mu$ l.

**Temperature Control System**

Choice of Voltage Control or Optical Temperature Control with a stabilised sensor and fibre optic transmission system.

**Gas System**

Fixed external flow.  
Adjustable internal flow with choice of inert or alternate gas.

**Gas Flow Rate**

3.5 l/min external flow.  
0, 100, 200, 300 ml/min internal flow.

**Gas Pressure**

1.03 ± 0.14 bar (15 ± 2 psi)

**Cooling Water**

Mains or Recirculator.  
Minimum flow rate 0.7 l/min.  
Pressure 1.4 - 6.9 bar (20 -100 psi).

**Cooling Time**

20 seconds (fixed).

**Interlocks**

Water pressure and flow rate.  
Inert gas pressure.  
Power.  
Power supply temperature.  
Magnet temperature (GFS35Z only).  
Furnace open.  
Magnet open (GFS35Z only).  
Cuvette present.

**Power**

200/220/240 V at 50/60 Hz, 30A Single phase.  
GFS35Z requires additional 200-240V 50/60 Hz single phase supply.

**Power consumption**

7.2k VA.  
GFS35Z - additional 1.5 kVA

**Dimensions (mm)**

265W x 390H x 495D.

**Weight**

GFS33 - 58kg, GFS35 - 50kg, GFS35Z - 66kg.

**Description**

All cuvettes mount directly in an all-graphite containment with end loaded contacts. Cuvettes are self aligning, and can be rapidly exchanged with a single lever movement.

The binary flow controlled internal gas system, with gas stop, offers a choice of the inert gas or an alternate gas, and the fixed external inert gas flow protects the cuvette and purges the optical temperature sensor.

**Furnace Control**

**Control System**

From Data Station.

**Program Phases**

20 time + temperature phases and 20 ramp (5 of each with Local Control).

**Phase Temperature Range**

Ambient to 3000 °C in 1 °C increments.

**Phase Time**

0 to 200.0 seconds in 0.1 second increments.

**Ramping Modes**

Linear and non-linear.

**Ramp Rates**

1 to 2000 °C/sec in 1 °C/sec increments.

**Injection Temperature**

Programmable up to 200 °C when used with the furnace autosampler.

**Signal Display**

Data Station VDU.

**Program Storage**

Unlimited disk storage with Data Station.

**Description**

Control of the furnace is from the SOLAAR Data Station software via the spectrometer. A full handshaking RS232C link is provided to the furnace power supply. Furnace cycles are set up within a rapid fill table allowing up to 20 phases to be programmed for temperature, time, ramp rate, gas type and flow and special functions such as optical temperature control, read phase and non linear ramp. Individual temperature look-up tables for all cuvette types ensure accurate temperature calibration. A lifetime display enables the cuvette to be changed before failure becomes likely. A pre-programmed cuvette clean cycle is available, culminating in maximum temperature for 5 seconds to ensure complete contamination removal. Comprehensive storage of the furnace program, together with spectrometer and autosampler parameters, is provided. Full furnace and autosampler status can be displayed, together with the results during the analysis. Complete monitoring of all furnace interlocks is provided.

**GFS Furnace Autosampler**

**Standard Items**

- Polypropylene Sample cups (120).
- Polypropylene Reagent cups (12).
- Spare capillary tips (10).

**Necessary Additional Items**

None.

**Carousel Capacity**

60 sample cups, 6 reagent cups.

**Cup Types**

Polypropylene sample cups.

Fluoroplastic sample cups.

Reduced Volume sample cups.

Polypropylene reagent cups.

**Cup Volume (nominal)**

Sample cups 2 ml.

Reduced Volume cups 1.5 ml.

Reagent cups 25 ml.

**Matrix Modifiers**

Up to 6 per Method.

**Resamples**

1 to 99 per measurement.

**Sampling System**

100 µl syringe (side port).

**Sampling Volume**

1 to 70 µl.

**Reproducibility of Injected Volume**

Better than 1 % (by mass) at volumes equal to and exceeding 10 µl.

**Accuracy of Injected Volume**

±0.5 µl.

**Inert Gas Pressure**

0.34 bar (5 psi).

**Wash and Waste Container Volume**

1 litre each.

**Dimensions (mm)**

296W x 380H x 320D.

**Weight**

Approximately 8 kg.

**Description**

The Autosampler will accommodate up to 60 samples in the large carousel. Carousel changeover allows up to 120 samples per element run. 6 large reagent cups enable up to 6 matrix modifiers to be

employed. Using the cup reducing ring, smaller cups can be used to reduce modifier consumption. The sample is collected and dispensed via an inert PTFE capillary with rapidly interchangeable tips. Viscous and Normal modes of injection ensure that samples are handled appropriately and an enhanced wash program eliminates contamination. Fast furnace operation provides concurrent operation of the furnace, spectrometer, and autosampler sample uptake procedures, minimising overall furnace cycle time. The syringe is visible and easily accessible for routine maintenance. A large, 1 litre capacity wash vessel for long, unattended analysis is complemented by an on-board waste vessel. All facilities are programmed from the SOLAAR software and sampling facilities such as matrix modification (wet or dry mixing), standards addition, dilution, re-concentration and automatic standards preparation can be rapidly set up. Automatic re-scaling and re-calibration functions are provided. If a sample falls outside the calibration range the system automatically and intelligently calculates the most appropriate dilution factor required to bring the sample back within range. Autosampler loading guides indicate the optimum carousel layout.

**VP100 Continuous Flow Vapour System**

**Standard Items**

'T' cell (2).  
Spare pump tubing.

**Necessary Additional Items**

None

**Absorption Cell**

Open ended, T-shaped silica cell.  
120 mm long.  
8 mm internal diameter.  
Long path Mercury absorption cell.

**Cell Heating**

Air/acetylene flame on 50 mm Universal burner.  
Electrical heating using EC90.

**Carrier/Purge Gas**

Argon or nitrogen.

**Gas Inlet Pressure**

0.34 bar (5 psi).

**Gas Consumption**

Up to 500 ml/min.

**Reducing Agents**

Sodium borohydride  
Tin (II) chloride may be used for mercury analysis.



**Solution Transport**

Variable speed, 4 channel peristaltic pump, using continuous flow principle.

**Solution Flow Rates**

Reductant - 3.2 ml/min.

Sample - 7.8 ml/min.

**Carrier Gas Flow Rates**

Variable 50 - 500 mL/min.

Electronic control via Mass Flow Controller.

**Reagent Containers**

Two 1L plastic bottles.

**Power**

220/240 V, 50 Hz or 110/120 V, 60 Hz.

**Power consumption**

30 VA.

**Dimensions (mm)**

475W x 300H x 200D.

**Description**

The VP100 Continuous Flow Vapour System performs hydride and mercury vapour measurements with significant sensitivity improvement over normal flame techniques. An air/acetylene flame or an electrically heated cell is used for atomisations. The VP100 unit incorporates the reagent reservoirs, a variable speed 4 channel peristaltic pump, control electronics and gas-liquid separator. An integrated Mass Flow Controller allows the carrier gas flow to be controlled and monitored through the system software, and the continuous flow principle eliminates the need to clean the reaction vessel after each sample. The VP100 operates automatically under Data Station control and can provide full autosampling operation with a suitable autosampler. When used with the EC90 Electrically Heated Atomisation Cell, unattended operation is possible.

**EC90 Electrically Heated  
Atomisation Cell****Standard Items**

EC90 Furnace Head.

EC90 Furnace Power Supply.

2 Silica T Cells.

**Necessary Additional Items**

VP100 Continuous Flow Vapour System.

Appropriate Accessory Mount.

**Control System**

From external power supply.

## Specifications

### Temperature Range

Ambient to 1000 °C in 1 °C steps.

### Power

220/240 V, 50 Hz or 110/120 V, 60 Hz.

### Power consumption

720 VAC.

### Dimensions (mm)

280W x 140H x 290D (Power supply).

### Weight

Approximately 13 kg.

### Description

The EC90 is an electrically heated atomisation cell for flameless vapour generation AAS. It provides improved analytical performance, unattended operation and reduced operating costs. The EC90 must be used with the VP100 and is suitable for all hydride forming elements.

### AA Validator Major Contents (depends on kit type):

- SOLAAR Validator Logbook
- Ca/Mg Hollow Cathode Lamp
- Ni/Cr/Mn Hollow Cathode Lamp
- Pyrolytically Coated Cuvettes
- Manganese Validation Standard
- Nickel Validation Standard
- Chromium Validation Standard
- Calibration Validation Unit (CVU)
- Water Blank Validation Standard

### Description

Available in three separate kits designed for Flame, Furnace or Flame and Furnace installations, the unique AA Validator kits provide full system validation for iCE 3000 Series AA systems, including comprehensive qualification plans. Full SOP and check sheets, together with complete supplier assessment questionnaires, are supplied in the Validator Logbook for recording and demonstrating compliance. All necessary consumables are provided and even method validation guidelines are included.

### Calibration Validation Unit Standard Items

- Calibration Validation Unit (CVU).
- Power Supply.
- RS232 Data Lead.

### Power

110V - 240 V at 50 or 60 Hz.

**Power consumption**

10VA

**Description**

The CVU is provided as part of the AA Validator kits and contains a set of traceable and certified filters. It is fitted in the spectrometer and controlled by the OQ Test software, and tests the spectrometer resolution, wavelength accuracy, photometric accuracy and stability, and D2 background correction efficiency.

The integrated PQ Test software contains a complete set of SOPs and a Wizard to lead the user through the tests needed to validate the instrument analytical performance against the manufacturer's specification.

**LH Universal Accessory Mount****Description**

Accessory Mount for use in the iCE 3000 Series Sample Compartments. This is necessary for mounting the EC90 Furnace Head, the Calibration Validation Unit, and the Mercury Absorption Cell in these Sample Compartments.

**RH Universal Accessory Mount****Description**

Accessory Mount for use in the right hand iCE 3400 / 3500 Series Sample Compartment. This is necessary for mounting the EC90 Furnace Head, the Calibration Validation Unit, and the Mercury Absorption Cell in this Sample Compartment.

**iSQ Enhancement****Standard Items**

Intelligent Spectrometer Qualification module.  
Coded Ca/Mg Hollow cathode lamp  
RH Accessory mount

**Necessary Additional Items**

None

**Power**

110V - 240V at 50 or 60Hz.

**Power consumption**

10VA

**Description**

The iSQ module is mounted on either the RH or LH sample compartment of iCE 3000 Series instruments. It provides facilities for automatically testing the Spectrometer hardware.

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# **iCE 3000 Series Pre-Installation Manual**

Version 1.3

Part number 9499 500 30031

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# Chapter 1

## Introduction

This manual is designed to help ensure that your iCE 3000 Series AA Spectrometer will be installed efficiently and will be able to meet your requirements quickly and completely.

It will minimise installation time if the services and facilities detailed here are available before the equipment is delivered.

Properly installed and maintained, your system will provide you with years of reliable service. Please inform your local Thermo Fisher organisation what aspects of support are most important to you and a tailored agreement can be provided to prolong the life of your instrument. Contact details for your local support office are at the end of this document.

This manual describes the environment and the resources required at the installation site of the iCE 3000 Series AA Spectrometer and associated equipment. Safety requirements for the installation are also detailed.

**WARNING:** 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 at the installation site. An installation engineer is not responsible for the fitting, or compliance of the facilities, or services.

The choice of an operating site for the instrument will be influenced by local considerations, for example ease of access and availability of electrical power.

Logical planning of the installation can save both time and money. The objective of this manual is to provide information that will enable the best site to be chosen and to highlight the essential requirements.

If further information or advice is required, contact your local Thermo Fisher organisation. A list of Thermo Fisher organisations and agents can be found on <http://www.thermoscientific.com>

Installation of the system will include some familiarisation training; however comprehensive method development and software training will require additional training that should be ordered separately.

## Chapter 2

# System Accessories

Required:

- Data Station (see specification)

Optional:

- GFS33 Integrated Graphite Furnace and Autosampler
- GFTV for iCE 3300
- SOLAAR Validator Package
- SOLAAR*security* Software
- ID100 Auto Dilutor
- VP100 Continuous Flow Vapour System
- EC90 Electrically Heated Atomisation Cell
- Slotted Tube Atom Trap
- Air Compressor
- Cetac ASX-520 Autosampler (requires dedicated RS232 on Data Station)
- Cetac ASX-260 Autosampler (requires dedicated RS232 on Data Station)
- Water Recirculator

Please request site requirement guides for any purchased accessories

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# Chapter 3

## Transportation

The spectrometer is supplied with all compatible accessories that have been purchased to meet the customer's system requirements. Items may be shipped in separate packages.

**WARNING:** All items should be transported to the installation site on pallets, in their original packaging and the right way up. Under no circumstances may any package be moved without its pallet unless under the direct control of an authorised Thermo Fisher service engineer.

Proposed routes from the customer receiving area to the installation site must be checked for suitability. The largest pallet size should be used to calculate clearances.

**WARNING:** The boxed Spectrometer and the Graphite Furnace and Power Supply should be moved using a mechanical lift, and to distribute the weight, four people should be used to lift the equipment from the box. Care should be taken in doorways, corridors or when lowering to avoid trapping fingers.

### Package Size

The instrument and accessories will be supplied inside especially designed packaging. The dimensions are:

**Table 3–1.** Spectrometer Packaged Dimensions and Weight

	Width	Depth	Height	Weight
iCE 3500	1070mm (42.2in)	740mm (29.2in)	760m (30in)	64 kg (141lbs)
iCE 3400	1070mm (42.2in)	740mm (29.2in)	760m (30in)	60 kg (132lbs)
iCE 3300	660mm (26in)	700mm (27.6in)	760mm (30in)	55 kg (121 lbs)
iCE 3300 GF	660mm (26in)	700mm (27.6in)	760mm (30in)	48 kg (106 lbs)

**Table 3–2.** Accessory Packaged Dimensions and Weight

	<b>Width</b>	<b>Depth</b>	<b>Height</b>	<b>Weight</b>
Furnace	715mm (28.2in)	550mm (21.7in)	620mm (24.5in)	57Kg (126lbs)
Zeeman Furnace	715mm (28.2in)	550mm (21.7in)	720mm (28.4in)	71Kg (157lbs)
Furnace Autosampler	510mm (20.1in)	420mm (16.6in)	530mm (20.9in)	10Kg (22.1lbs)
EC90	720mm (28.4in)	620mm (25.5in)	350mm (13.8in)	20Kg (44.1lbs)
VP100	690mm (27.2in)	500mm (19.7in)	400mm (15.8in)	9.5Kg (21lbs)

Transportation to some destinations may necessitate additional packaging, for example a packing crate. Dimensions and exact specifications may vary.



## Short Term Storage

To maintain the instrument in serviceable condition and to comply with the conditions of warranty, ensure that storage of each item is maintained within the stated parameters detailed for the Working Area Requirements below. Make sure that the packages are stored the correct way up.

Note: Do not open the Packages without the permission of the Thermo Fisher engineer

When the packages are opened prior to installation, the contents will be checked against the packing list(s).

Spectrometers ordered with Validation packages should not be opened other than by the trained Service Engineer performing the installation.

## Transit Damage

Before accepting delivery of any equipment, the packages should be inspected for signs of obvious damage. The nature of the damage should be noted on the delivery notice and signed by the carrier's representative.

Within the time stated in the Thermo Fisher terms and conditions an inspection should be made for concealed damage. The local Thermo Fisher organisation should be advised of any damage in writing and, on receipt of specific instructions, the customer should return the equipment complete and in the original packing material.

## Warranty

The instrument is warranted against defects in material and workmanship for a period of 12 months from installation, or 15 months from shipment, whichever comes first. This warranty does not cover damage sustained as a result of improper storage by the customer prior to installation, or resale to a third party.

**Note:** Warranty is not transferable to a third party without Thermo Fisher approval.

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# Chapter 4

## Working Area Requirements

The choice of site will be influenced by the dimensions and weights of the spectrometer and 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.

Pre-installation visits can be made on request, but may be chargeable.

### Introduction

Prior to 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 condensation forming. Sample preparation activities and corrosive materials should be located in a separate room to avoid problems due to corrosive fumes.

A comprehensive 'Risk Assessment' should be carried out that is specific to the handling of solvents, samples and sample preparation.

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

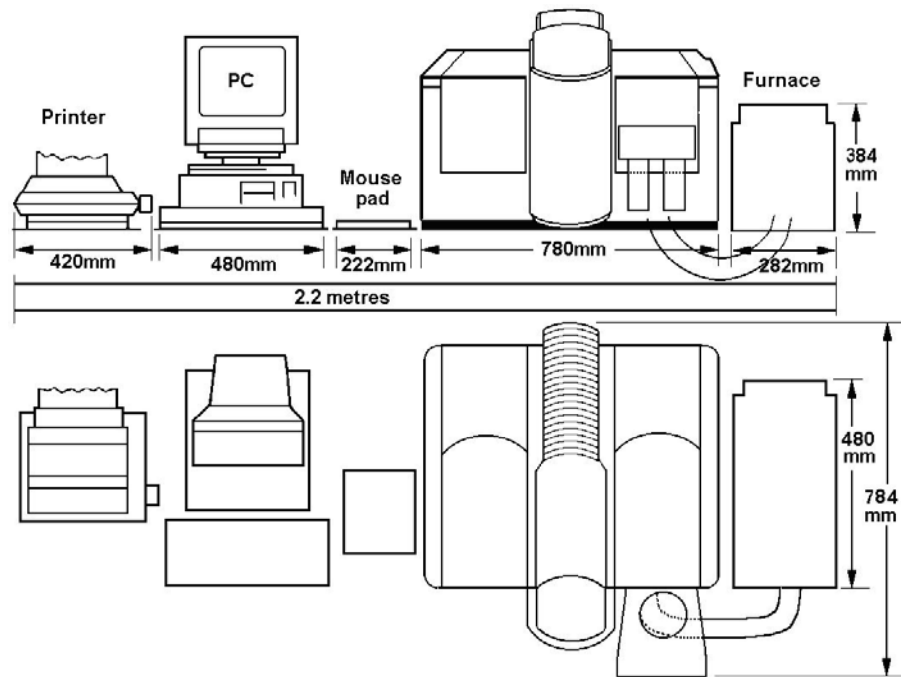
### Location

The instruments are designed for use on a normal laboratory bench. Ideally the instrument should be placed on a moveable bench with 0.5 meters 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.

The mounting arrangements should 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 may affect the optical alignment of the spectrometer and accessories.

To avoid the possibility of liquid ingress into the top of the spectrometer the location should ensure it is not possible to store sample or other liquids directly above the instrumentation.

Organic or volatile solvents should not be stored, even for a short time, near the instrument.



**Figure 4–3.** iCE 3500 Series typical location diagram.

Do not position the equipment so 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 5 to 40 °C (41 to 104 °F), The temperature should not change by more than 2 °C per hour.

Atmospheric humidity should be 20 to 80 % m/v for an ambient temperature between 5 and 30 °C and 20 to 60 % m/v for an ambient temperature between 30 and 40 °C. Atmospheric conditions must be non-condensing. The instrument room should be at a positive pressure with respect to rooms with a corrosive atmosphere.

## Electrical

The spectrometer will require an electrical supply at 100 to 240VAC, 300VA, 50/60Hz. The spectrometer will be supplied with a 2m mains cord with USA type or Non USA (rest of world) type coding. USA coding is black (X – ac high), white (Y – ac low), green (G – earth/ground). Rest of World coding is blue (neutral), brown (live), green/yellow (earth/ground).

The furnace power supply unit will require a single phase mains supply of 200/240VAC, 7.2KVA, 50/60Hz. In normal operation the furnace power supply may draw surge currents in excess of the nominal rating. A dedicated supply should be used which may require the inclusion of delayed action protection devices such as circuit breakers or motor start fuses.

Within the EU the furnace power supply unit should be connected to a supply with an impedance of 0.124 Ohms or less. It is the responsibility of the user of the unit to ensure by consultation with the local supply authority if necessary, that the unit is connected to this type of supply.

An additional 220/240VAC, 1.5KVA, 50/60Hz is required for a Zeeman furnace power supply unit.

Other accessories will require a single phase mains supply 110/120V, or 200/240V at 50/60Hz. The requirements are:

EC90, 720VAC

VP100, 125VAC

ID100, 100VAC

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

Multiple installations will require separate mains supplies.

Additional standard mains sockets will be required for the Data Station and printer, for an extended autosampler, for a chiller and for each additional accessory used.

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.

## Spectrometer Size

**Table 4–1. Spectrometer Dimensions and Weight**

	Width	Depth	Height	Weight
iCE 3500	780mm (30.1in)	577mm (22.7in)	520mm (20.5in)	54Kg (119lbs)
iCE 3400	780mm (30.1in)	577mm (22.7in)	520mm (20.5in)	54Kg (119lbs)
iCE 3300	567mm (22.3in)	589mm (23.2in)	510mm (20.1in)	45Kg (99lbs)
iCE 3300GF	567mm (22.3in)	589mm (23.2in)	510mm (20.1in)	38 Kg (84 lbs)

**Table 4-2. Accessory Dimensions and Weight**

	Width	Depth	Height	Weight
Furnace	265mm (10.4in)	495mm (19.5in)	390mm (15.4in)	50Kg (110.2lbs)
Zeeman Furnace	265mm (10.4in)	495mm (19.5in)	390mm (15.4in)	66Kg (145.5lbs)
Furnace Autosampler	296mm (11.7in)	320mm (12.6in)	380mm (15in)	7.3Kg (16.1lbs)

	<b>Width</b>	<b>Depth</b>	<b>Height</b>	<b>Weight</b>
EC90	305mm (12in)	500mm (19.7in)	215mm (8.5in)	16Kg (35.3lbs)
VP100	340mm (13.4in)	360mm (14.2in)	280mm (11in)	8.5Kg (18.7lbs)

# Gas Requirements

## General

The spectrometer will use a flame or furnace to provide atoms that are analysed to indicate the concentration of an element in an unknown sample. A flame will use acetylene as the fuel and either air or nitrous oxide will be required as the oxidant for the flame. A nitrous oxide flame will also require air to allow the system to stabilize before a nitrous oxide flame is used.

Care should be taken to ensure that laboratory gas lines are gas tight, free from oil and grease, and do not contaminate the gases used for the spectrometer.

Gas cylinders must be stored and operated in a vertical position and in an environment free from naked lights and sparks. Cylinders should be left to stabilize overnight before use.

Only the gas tubing supplied with the spectrometer should be used. The gas tubing supplied will terminate in 3/8in BSP thread connections.

All gas supplies should be regulated and connected within 1 meter of the instrument.

A comprehensive 'Risk Assessment' should be carried out that is specific to the gas requirements.

## Acetylene

A flame spectrometer will require acetylene at 0.62 bar (9 psi) with flow rates of between 0.8 and 5.1 lmin<sup>-1</sup>. The purity must be at least 98.5%, with impurities for sulphur and phosphorus of less than 15 ppm, and water of less than 100 ppm. The balance of the gas should be inert gas.

The instrument will be supplied with acetylene gas tubing terminating in 3/8in BSP Left Hand tighten thread connections.

Black iron is recommended for internal plumbing of acetylene.

It is recommended that banks of cylinders be used with switch over valves so that the gas supplies can be used continuously. The cylinders must be fitted with regulators which can provide an outlet pressure of at least 0.84 bar (12 psi).

A flashback arrestor must be fitted to pressure regulator outlets.

Acetylene is usually supplied in cylinders containing about 6.16m<sup>3</sup> (220ft<sup>3</sup>) of the gas dissolved in acetone. As the acetylene is used the pressure in the cylinder will fall. This can result in release of acetone that can lead to erratic results and possible instrument damage. A cylinder must be considered exhausted at 7 bar (100 psi).

The acetylene in the cylinder must be dissolved in acetone. Do not use acetylene that is dissolved in other solvents (for example DMF).

The cylinders should be stored and operated in a vertical position as far away from the instrument as the length of the fuel hose will permit and in an environment free from naked lights or sparks.

**Caution:** If pressures above 0.62 bar (9 psi) acetylene are used in the UK, the Health and Safety Executive of HM Factory Inspectorate must be informed and an Exemption Certificate obtained. Local and national regulations and guidelines must be followed at all installation sites.

**Warning:** Acetylene is a highly flammable gas that can form explosive compounds. Contact with copper, silver, and alloys containing these metals must be avoided. Fittings, filters, or tubing containing copper, silver, or other acetylide forming metals, must not be used.

## Air

The spectrometer requires clean dry compressed air at a steady pressure at 2.1 bar (30 psi), with a flow up to 10 lmin<sup>-1</sup>.

If the graphite furnace is using air as an oxidant it requires a pressure of 1.1 bar (15 psi) and a flow rate of 0.2 lmin<sup>-1</sup>.

The air should be supplied by an oil free compressor at a pressure up to 4.14bar (60psi). A filter/ regulator unit is required that incorporates a 5 micron filter, a pressure regulator and a pressure gauge. A clear plastic Terylene reinforced link hose, of at least 3.5m (12ft) is required to cool the air and permit efficient water removal by the filter/regulator unit.

## Argon and Nitrogen

A graphite furnace will require argon, or nitrogen, gas at 1.04bar (15psi) with a purity of 99.999%. Argon gas is the preferred purge gas.



A vapour system will require a pressure of 0.34bar (5psi).

**Warning:** Nitrogen and argon gas may cause asphyxiation at high concentrations. Appropriate care should be taken.

### **Nitrous Oxide**

A flame spectrometer may require nitrous oxide gas for some applications. Nitrous oxide must be supplied at 2.75bar (40psi) at a flow of 8lmin<sup>-1</sup>. Special grade AA nitrous oxide must be used with oxygen impurities of less than 150ppm; nitrogen less than 400ppm; carbon dioxide of less than 50ppm; oxides of nitrogen less than 2ppm; carbon monoxide less than 1ppm and water less than 15ppm. A two stage regulator is required to provide good precision and stable flame conditions. It maybe necessary to fit a heater to the regulator to prevent freezing, which would cause flow rate variations and an unstable flame.

**Warning:** Nitrous oxide is an anaesthetic and if inhaled in large amounts can be fatal. The gas supports combustion to a higher degree than air and will produce highly combustible mixtures very readily.

## **Water Temperature Control**

A recirculating chiller should be used to remove waste heat from the graphite furnace. The water temperature should be set to 5°C below the ambient temperature.

The flow requirement of the furnace is 0.7 l/min and the pressure should be between 1.4 and 6.9bar (20 to 100psi). The chiller must have a cooling capacity in excess of 900W.

A suitable chiller (ThermoFlex 900) can be supplied from your local Thermo Fisher organisation.

Requirements for ThermoFlex 900:

Power: 230V, 50Hz, 7A; or 115V, 60Hz, 13A

Dimensions: 696 x 361 x 627mm (27.4 x 14.2 x 24.7in)

The furnace will be supplied with plastic connecting tubing that has a 6.5mm **inside** diameter.

Connection to the water chiller should be within 3 meters of the instrument. If greater distance is required insulated tubing must be used.

A suitable fungicide, or biocide, should be added to the water. This can be recommended by the supplier of the chiller.

## Waste Storage

The analysis of a sample, by flame atomic absorption, usually involves production of a fine mist from a liquid sample. Waste will be produced that could be corrosive and toxic, or an organic solvent. An appropriate container is required that is solvent proof, shatterproof and vented away from the instrument. Ensure the waste container does not constitute a spill or trip hazard.

The waste vessel must have a wide neck, be made of a shatterproof material and resistant to attack by the chemicals used in the analysis. Polythene is suitable in most cases.

It may be necessary to neutralise waste to prevent any toxin build up. Appropriate facilities should be provided for the disposal of any waste. Waste should be disposed of following local procedures and regulations.

**Warnings:** The lower end of the drain extension must always be above the level of the liquid in the drain vessel.

Glass or metal drain vessels must not be used.

The drain extension tube must provide a free flowing drain outlet, to prevent the formation of any additional 'U' traps, and must be free of kinks and sharp bends.

## Spectrometer Fume Extraction

The spectrometer is designed to operation in clean air conditions. The laboratory must be free of all contaminants that could have a degrading effect on the instrument components.

Dust, acid and organic vapours must be excluded from the work area. The warranty will be void if the equipment is operated in substandard conditions.

**WARNING:** The spectrometer must never be operated without an effective fume extraction system installed above the sample compartment. The fume extraction hood must not be attached to the chimney and an air gap of 150mm (6in) to 230mm (9in) must be made. For dual atomizer instruments with two sample compartments, two extractions may be required.

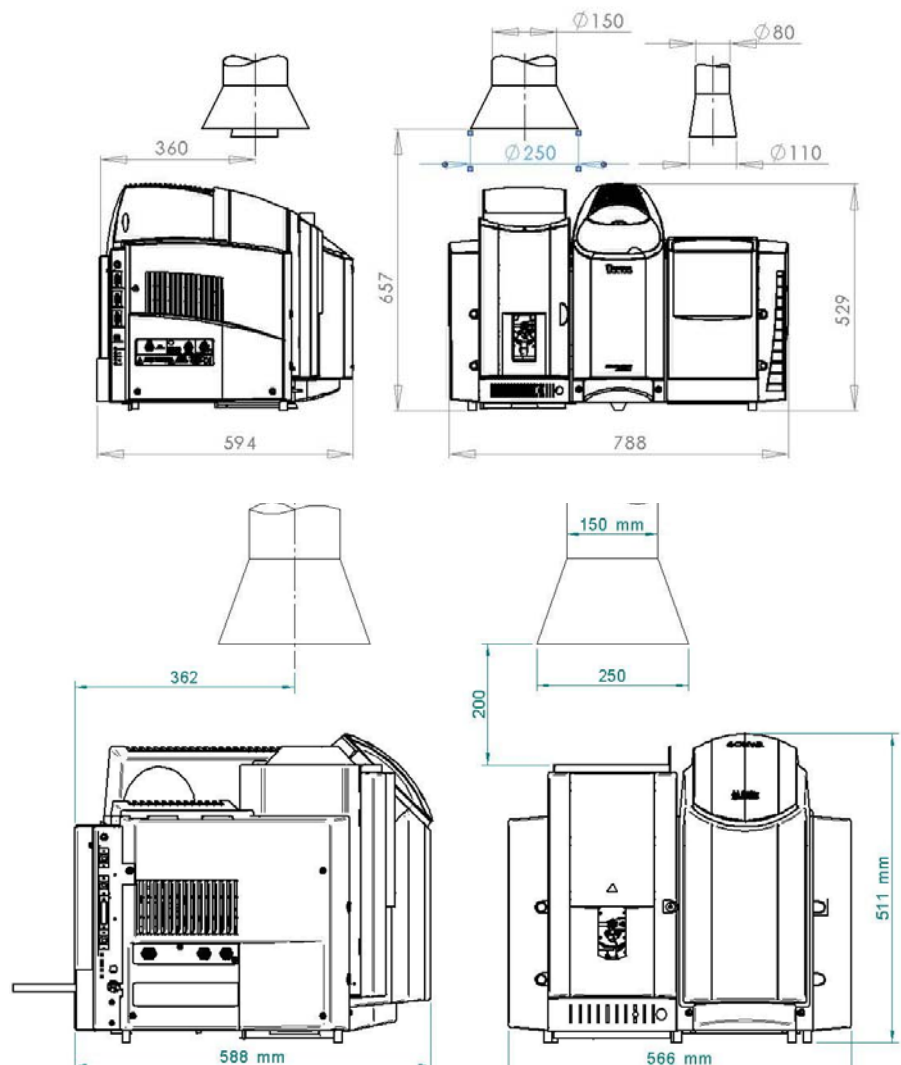
An acetylene flame can generate between 3 and 5kW with temperatures up to 3000°C. The fumes from the flame, which may be corrosive and toxic, are discharged from the instrument chimney during operation. To ensure a safe working environment and safe removal of waste combustion products an effective extraction system must be installed; this should include appropriate filtering of hazardous toxic fumes.

It is recommended that the hood of the extraction system and ducting be made of stainless steel with a circular cross section of at

least 150mm (6in). No compounds that may form acetylides should be in contact with exhaust fumes.

A centrifugal fan, preferably with plastic coated metal blades, extracting at least  $2800\text{lmin}^{-1}$  ( $100\text{ft}^3\text{min}^{-1}$ ) must be fitted into ducting connected to the hood over the flame. The fan must be at least 3m from the hood. The hood over the furnace must extract  $200\text{lmin}^{-1}$ . Suitable extraction should also be used for the EC90.

**Caution:** The instrument must not be installed in a normal chemical fume cupboard as the ventilation is likely to be inadequate and any other chemical operation may damage the spectrometer.



**Figure 4-4.** iCE 3000 Series, extraction positions.

The extraction system must not be affected by external weather conditions, or other uses the system may be used for.

**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, even those in an autosampler.

# Chapter 5

## Communications Interface

### On the Data Station

The Data Station requirements will normally be met by a PC meeting the following minimum specification:

- Windows XP Professional, Windows Vista Ultimate or Windows 7 Professional.
- 20 Gb Hard drive
- 512 Mb RAM
- A USB connection for spectrometer communication
- Some accessories require a dedicated RS232 port
- A separate Ethernet card will be required for networking
- A printer port may be required

A suitable PC is available from your local Thermo Fisher organisation.

The installation engineer is not responsible for network and customer specific set-up of the PC.

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# Chapter 6

## Pre-Installation Checklist

This checklist is to be filled in by the customer to confirm that all relevant factors concerned with the installation have been considered and dealt with. On completion of the list, a copy should be forwarded to your local Thermo Fisher organisation with a request for installation to be carried out. Failure to send the checklist may result in delays with the instrument installation; any delays may incur extra charge.

**Table 6–2.** Pre-Installation Checklist

Action	Completed?
Work area prepared	<input type="checkbox"/>
Location of units planned	<input type="checkbox"/>
Suitable work benches provided	<input type="checkbox"/>
Fume extraction system installed and commissioned.	<input type="checkbox"/>
Electrical socket outlet available for:	
Spectrometer (domestic)	
Furnace (conditional – see Chapter 4 Electrical)	<input type="checkbox"/>
Data station (domestic)	<input type="checkbox"/>
Chiller (domestic)	<input type="checkbox"/>
Autosampler (domestic)	<input type="checkbox"/>
Other accessories (domestic)	<input type="checkbox"/>
Drain/waste container available	<input type="checkbox"/>
Gas supplies available and tested with correct size fittings (3/8in BSP): 2.1bar (30psi) air; 0.62bar (9psi) acetylene; 2.1bar (30psi) for argon/nitrogen; 2.75bar (40psi) nitrous oxide.	<input type="checkbox"/>
Cooling water (6.5mm id plastic tubing supplied with instrument)	<input type="checkbox"/>
18 MΩ/cm <sup>3</sup> water required for testing	<input type="checkbox"/>
Test and set-up solution.	<input type="checkbox"/>
SolaarSecurity (optional) and Validation (optional)	
The following manuals are required by the customer to enable preparation prior to instrument installation IQ	
1) SolaarSecurity pre-installation procedure	<input type="checkbox"/>
2) SolaarSecurity Software installation manual	<input type="checkbox"/>
3) Security Administration Users Guide	<input type="checkbox"/>

Contact details for your local Thermo Fisher Organisation should be below, or are available from:

[technicalsupport.sid.cambridge@thermofisher.com](mailto:technicalsupport.sid.cambridge@thermofisher.com)

Customer Support contact details:



## Chapter 7

# Special Agreements

This section should detail any special agreements, which have been arranged with your local Thermo Fisher organisation.

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