S Series Atomic Absorption Spectrometers Operators Manual

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Issue 3d

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INTRODUCTION

SOLAAR S Series User Documentation

Operators Manual

This is 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.

Pre-Installation Manual

This Manual provides details of the services and bench space that must be available before an S Series Atomic Absorption Spectrometer System can be installed in your laboratory. This Manual should have been supplied to you with the confirmation of the order for the S Series instrument that you placed with your supplier.

Accessory Installation Manuals

Several of 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 S Series instruments, which supersedes that provided in the Accessory Manuals.

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

Data Station Software Manual

This Manual is supplied with the SOLAAR Data Station Software.

It describes:

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

Data Station On-line Help System

The Data Station software contains a comprehensive On-line 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 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.

Local Control Software Manual

The Local Control Software Manual is supplied with the AA Series Local Control Module.

It describes:

- the features and functions of the software.
- the purpose of every command.
- the purpose and values of every parameter.
- a series of familiarisation procedures to get you up and running quickly with your new instrument.

Methods Manual

This 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.

Applications Library

The SOLAAR Series Software Installation CD-ROM also contains a large amount of applications information to help you get the best from your S Series instrument. This will be installed with the Data Station Software, and can be accessed from the Applications Documentation command in the SOLAAR program group.

The Applications Information includes:

- **Method Sheets**, providing detailed descriptions of various complex analyses.
- **Method Guides**, providing brief descriptions of various simple analyses.
- **Performance Sheets**, providing details of the analytical performance of the S Series instruments
- **Technical Articles**, providing in-depth discussion of various aspects of the technique and instrumentation.

Safety in Atomic Absorption Spectrometry

READ THIS PAGE 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.

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 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 operating and service personnel follow generally accepted safety procedures in addition to the specific precautions specified in this manual.

Specific Warning and Caution statements are included in the relevant sections of this manual.

Warning and Caution statements and symbols are marked on the apparatus where appropriate. The symbols used are described in the figure.

Unless otherwise stated in this manual, the covers of the instrument and accessories should only be removed by qualified, trained Service personnel.

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

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.

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

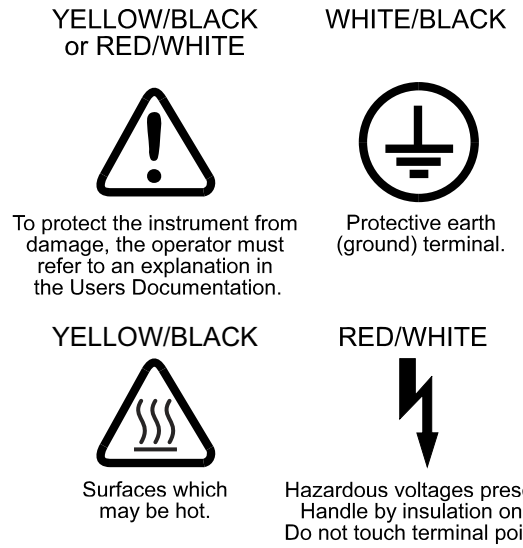
It is the ***user's*** responsibility to carry out appropriate cleaning and decontamination of the equipment if hazardous material is spilt on or inside the equipment.

- 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.



Warning and Hazard symbols

- 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 Electron 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 Electron's compliance with these Directives, the recyclers in your country, and information on Thermo Electron products which may assist the detection of substances subject to the RoHS Directive are available at www.thermo.com/WEEERoHS.

Sicherheit in der Atom Absorptionsspektrometrie

LESEN SIE DIESE SEITEN DESONDERS SORGFÄLTIG VOR DER INSTALLATION UND DEM GEBRAUCH DES GERÄTES.

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.

Spezielle Vorsichts- und Warnungshinweise finden Sie in der Bedienungsanleitung besonders vermerkt.

Außderdem befinden sich am Gerät, wo erforderlich, Hinweisschilder.

Die Geräteabdeckung sollte ausschließlich durch einen qualifizierten Thermo Electron Corporation Service-Ingenieur entfernt werden.

Alle Ersatzteile und verbrauchbaren benutzten Einzelteile müssen durch Thermo Electron Corporation genehmigt werden.

Einige der in der AA-Spektroskopie zur Anwendung kommenden Chemikalien sind korrosive, leight entzüdbar, radioactiv, infektiös oder toxisch.

- Daher muß dafür Sorge getraden werden, daß die normalen Laborrichtlinien zur Handhabung dieser Chemikaline zur Anwendung kommen.

Elektrische Sicherheit

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

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.

Reinigung und Dekontamination der Geräte

Es liegt in der direkten Verantwortung des ANWENDER und nicht des Geräteherstellers, eine Dekontamination des Gerätes durchzuführen, falls sich toxische Substanzen im oder dem System befinde.

- Reinigungs- und Dekontaminationsarbeiten sind in der Bedienungsanleitung beschrieben.
- Bevor Sie eine Reinigungs-bzw. Dekontaminierungsmethode einsetzen, die nicht vom Hersteller empfoohlen wird (vgl. Wartungshinweise dieser Bedienungsanleitung), sollten Sie zusammen mit demhersteller sicherstellen, daß diese Vorgehensweise da Systemnicht beschädigt.

Beeinträchtigung der Sicherheitseintichtungen.

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

- Eine Sicherheitsbeinträchtigung liegt z.B. dann vor, wenn nicht mehr die erwarteten Ergebnisse oder eine sichtbare Beschädigung vorliegen.

Symbole - Sicherheit

- Wenn das Gerät nicht gemass 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 Electron 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 Electron, die Verwerter und Hinweise die Ihnen nützlich sein können, die Thermo Electron Produkte zu identifizieren, die unter diese RoHS Anweisung fallen, finden Sie unter www.thermo.com/WEEERoHS.

Sécurité en Spectrométrie d'Absorption Atomique

LIRE TRES ATTENTIVEMENT CETTE PAGE AVANT D'INSTALLER ET D'UTILISER L'INSTRUMENT ET SES ACCESSOIRES.

LES NORMES DE SECURITE CONTENUES DANS CE MANUEL SONT CONFORMES AUX RECOMMANDATIONS DU «HEALTH AND SECURITY AT WORK ACT 1974».

Introduction

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

Le réglage, la maintenance et la réparation de l'équipement, impliquant qu'un capot soit retiré, ne doit être réalisée que par des Ingénieurs de Service qualifiés qui sont prévenus des risques encourus.

Précautions de Sécurité

Pour l'utilisation correcte et en toute sécurité de l'instrument et de ses accessoires, il est essentiel que les utilisateurs et le personnel de service suivent les procédures de sécurité généralement acceptées en plus des précautions spécifiques indiquées dans ce manuel.

Les indications spécifiques de Mise en Garde et de Précaution sont inclus dans les sections appropriées de ce manuel.

Les indications de Mise en Garde et de Précaution et les symboles sont marquées sur l'instrument lorsque cela est approprié. Les symboles utilisés sont décrit figure 1

Les capots de l'instrument et des accessoires ne doivent être retirés que par un ingénieur qualifié et entraîné de Thermo Electron Corporation.

Tous les pièces de rechange et articles consommables utilisés doivent être approuvés par Thermo Electron Corporation.

Certains produits chimiques utilisés en Spectrométrie d'Absorption Atomique sont corrosifs et/ou inflammable, et les échantillons peuvent être radioactifs, toxiques ou potentiellement inflammables.

- Les procédures et règlements normaux de laboratoire pour la manipulation de tels matériels doivent être suivis.

Sécurité Electrique

Tous les équipements alimentés par le courant électrique sont conçus pour fonctionner avec une alimentation électrique avec prise de terre. La connexion de mise à la terre de l'équipement doit être réalisée, dans le cas contraire, la sécurité ne pourrait être assurée.

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

- Suivre les normes nationales du pays d'utilisation lors de l'installation des prises électriques. 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

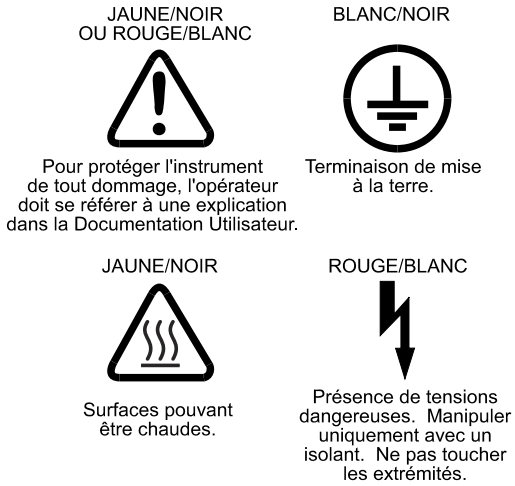
Il est de la responsabilité de l'**utilisateur** de procéder au nettoyage et à la décontamination appropriés de l'équipement si des matériaux dangereux ont été répandus sur ou dans l'équipement.

- Les procédures de nettoyage et de décontamination sont spécifiées dans les sections appropriées de ce manuel.
- Avant d'utiliser toute autre procédure, les **utilisateurs** doivent contrôler avec le constructeur que la méthode proposée ne peut endommager l'équipement.

Altération de la Protection de Sécurité

Si jamais la Protection de Sécurité a été altérée, l'instrument et les accessoires doivent être rendus inopérants et protégés contre toute utilisation involontaire. La cause du problème doit alors être communiquée à l'organisation de Service Après Vente.

- La Protection de Sécurité peut avoir été altérée si l'instrument n'arrive pas à fonctionner normalement ou s'il présente des dommages visibles.



Symboles de Mise en Garde et de Danger

- Si l'équipement est utilisé d'une manière non spécifiée par le constructeur, la protection de sécurité fourni par l'équipement peut avoir été altérée.

Conformité DEEE:

Ce produit doit être conforme à la directive européenne (2002/96/EC) des Déchets d'Equipements Electriques et Electroniques (DEEE). Il est marqué par le symbole suivant:

Thermo Electron 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 Electron à ces directives, les recycleurs dans votre pays et les informations sur les produits Thermo Electron qui peuvent aider la détection des substances sujettes à la directive RoHS sont disponibles sur www.thermo.com/WEEERoHS

Regulatory Notices

Regulatory Compliance

The S Series Spectrometers and accessories are CE marked, indicating compliance with the following European Directives:

89/336/EEC Electromagnetic Compatibility Directive (EMC Directive)
72/23/EEC Electrical Equipment designed for use within certain voltage limits (Low Voltage Directive).

For further details, refer to the regulatory notice for the S Series spectrometers and accessories, which is reproduced here.

Übereinstimmung mit Regularien

Alle Spektrometer der S-Serie tragen das CE Zeichen und entsprechen damit den europäischen Regelwerken.

89/336/EEC Electromagnetische Kompatibilität (EMC)
72/23/EEC Elekrische Geräte, die für den Einsatz innerhalb bestimmter Spannungsgrenzen konsipert sind (Regularien zur Minimalspannung)

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

Conformité Normative

Tous les Spectromètres Série S et accessoires sont marqués CE, indiquant leur conformité avec les Directives Européennes suivantes :

89/336/EEC Directive de Compatibilité Electromagnétique (Directive EMC).
72/23/EEC 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 S, reproduite sur la page suivante.

EC DECLARATION OF CONFORMITY

No. : UAA090201

The undersigned, representing the following manufacturer

manufacturer :	Thermo Elemental (Unicam Ltd)
address :	SOLAAR House, 19 Mercers Row, Cambridge CB5 8BZ, UK

herewith declares that the product

product identification :	SOLAAR S Series Flame and Furnace Atomic Absorption Spectrometer Systems.

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

reference no	title
73/23/EEC	Low Voltage Directive
89/336/EEC	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 : 02

Place : Cambridge, CB5 8BZ, UK **Date :** 31st January 2002

Signature : M. P. Wassall

Name : Dr Michael P Wassall **Function :** Product Director

EC DECLARATION OF CONFORMITY

No. : UAA090201

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

- harmonized standards :

number	title	result
safety:		
BS EN 61010-1:2001	General Requirements	Pass
IEC1010-2-061:1995	Safety Requirements for Electrical Equipment for Measurement, Control and Laboratory Use	Pass
EMC:		
Conducted & Radiated Emissions		
EN61326-1 Class B	Conducted & Radiated Emissions	Pass
EN55022 Class A	Conducted & Radiated Emissions	Pass
EN61000-3-2	Harmonic Currents	Pass
Immunity		
IEC1000-4-3	Radiated Field Immunity	Pass
IEC1000-4-6	Conducted RF Immunity	Pass
IEC1000-4-4	Electrical Fast Transients	Pass
IEC1000-4-2	Electrostatic Discharge	Pass
IEC1000-4-5	Surges	Pass
IEC1000-4-11	Voltage Dips & Interruptions	Pass
IEC1000-4-8	Power Frequency Magnetic Field	Pass

- other standards and/or technical specifications :

number	title	result
FCC CFR47 Part 15/B Class A	Radio Frequency Devices - Unintentional Radiators	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 Elemental approved computer and accessories
2. About Shielded Cables: Use only shielded cables supplied by Thermo Elemental when connecting this instrument to the computer and other accessories

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.
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		

Lamp	UV Hazard	Cathode material hazard
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		
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		

SPECTROMETER OPERATION

Spectrometer Installation

Pre-Installation

The laboratory facilities necessary to safely install and use your S 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 and accessories will be installed by a trained Service Engineer.

Accesories

Connect accessories to the appropriate sockets on the Connection Panel. Refer to the relevant sections of this manual for detailed instructions. Note that accessories that require serial RS232C connections can be connected to either of the Accessory ports, or to the Aux/Download port as required.

Local Control Printer Installation

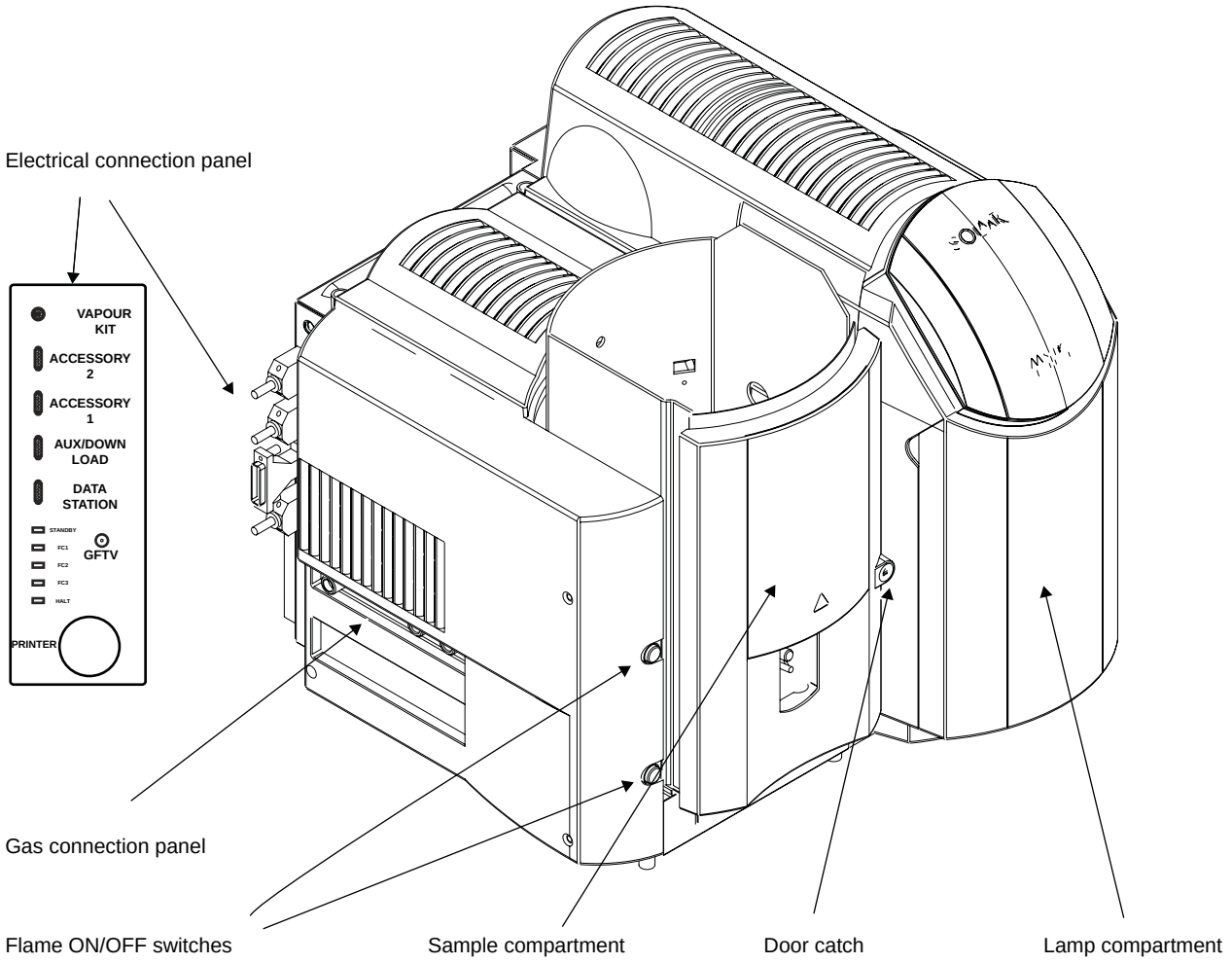
If you have the Local Control module fitted, connect a suitable printer to the pre-installed printer cable. The printer must have a parallel interface connection, and must support the Epson ESC/P printer control protocol.

Data Station Installation

If you have a Data Station, connect a 9-way RS232C cable between a serial port on your Data Station and the Data Station port on the Spectrometer Connection Panel; software installation procedures are described in the Software Manual

Data Station Printer Installation

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.



S 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 S 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.

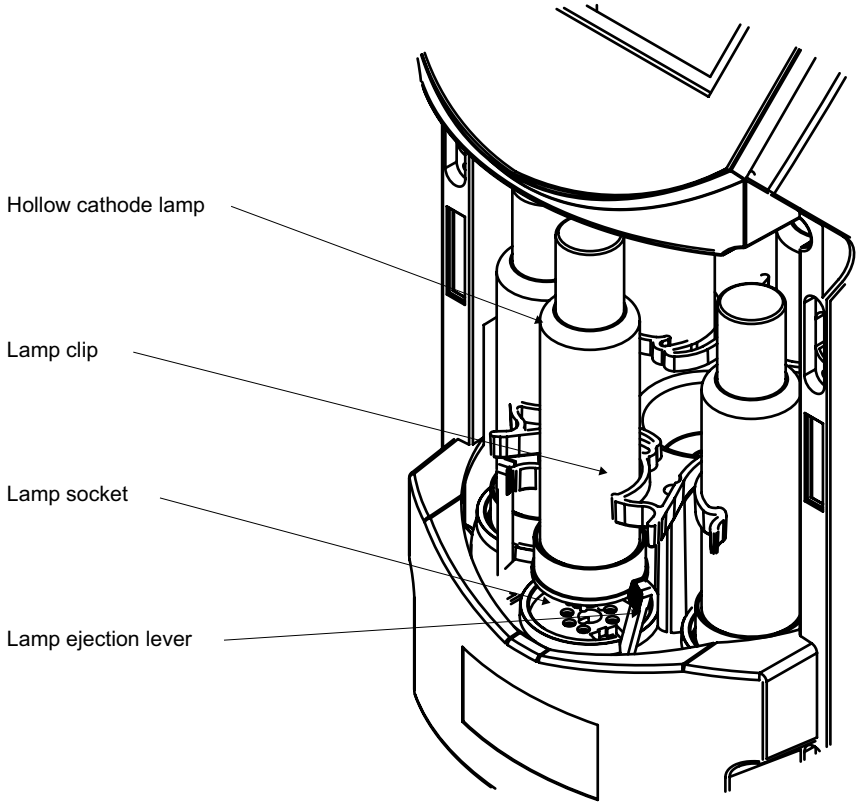
Hollow Cathode Lamps

- Coded or uncoded lamps supplied by Thermo Electron Corporation or their agents are recommended.
- Most 37mm diameter uncoded lamps supplied by third party manufacturers may be used.
- Most 37mm 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.

Warning: Thermo Electron Corporation does not guarantee that the instrument will work, nor that the published specification will be met when using such third party lamps.

- 50mm 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 S Series spectrometer; they could cause severe damage to the instrument.



Lamp compartment - general view

Lamp installation

1. Turn on the system and start the Data Station software if required.
2. Open the lamp carousel door.
3. Use the software lamp commands 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.
5. Secure it in position with the Lamp Clip.
6. 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.

Installing Hollow Cathode Lamps (continued)

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. Unclip the Lamp Clip, then press the lamp ejection lever downward to eject the lamp.

Lamp alignment

The S Series spectrometers provide automatic lamp alignment facilities, with manual overrides if required. You can access these facilities from the Lamp pages of the Data Station or Local Control software.

The Sample Compartment Universal Accessory Mount

Introduction

This is a Universal Mount for fitting several accessories in the S Series Sample Compartment.

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 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 Sample Compartment Universal Mount

1. Use the appropriate Software commands to move the Burner to the Parked position.
2. Refer to the Flame section of this manual, and remove the Burner Head.
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.

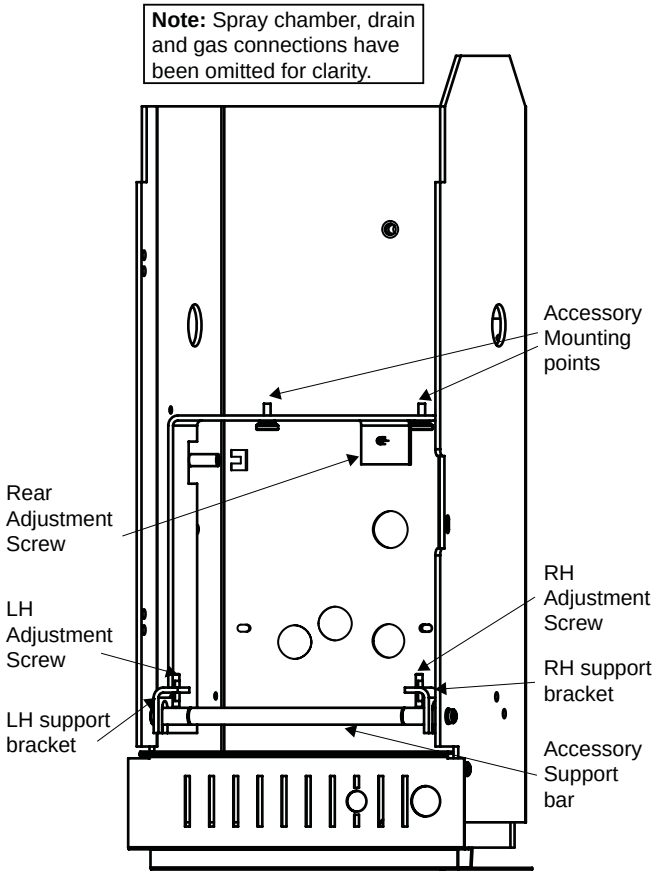
Reverse this procedure to remove the assembly.

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 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. Do ***not*** select any form of background correction.
   - use the Software to display the live absorbance signal.
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 3mm 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.



The Sample Compartment Universal Accessory Mount

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 low level performance of your spectrometer. The iSQ Module is fitted in the Sample Compartment of the spectrometer using the Universal Accessory Mount.

The iSQ package consists of :

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

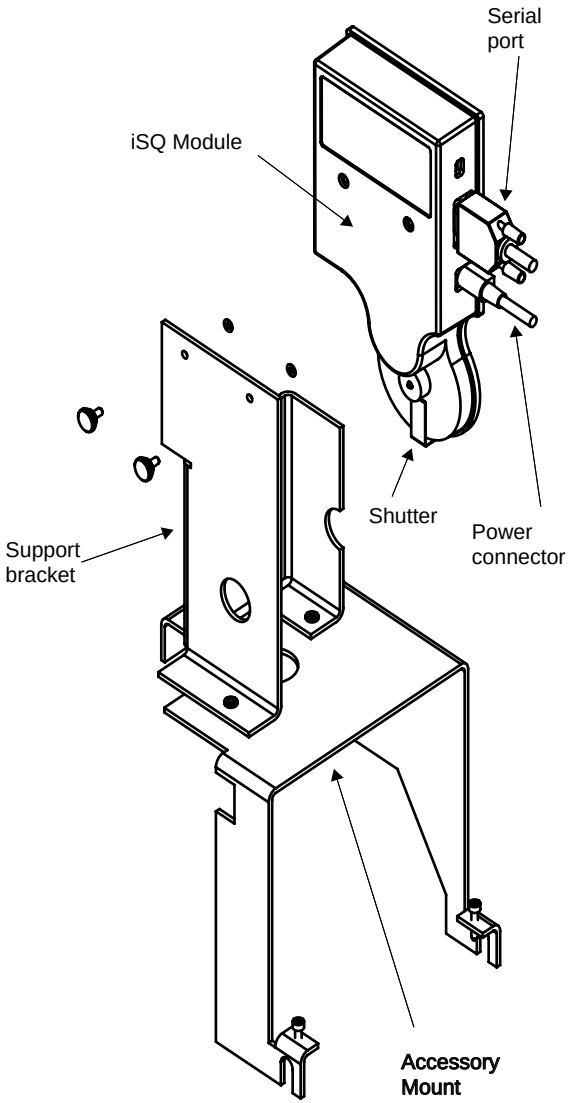
Safety

Observe electrical safety precautions.

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 figure.
2. Attach the assembly to the Accessory 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.



iSQ Module Assembly (S Series)

Installation

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

1. If a GFS97 graphite furnace is fitted in the spectrometer sample compartment:
   a. Remove the GFS97 Furnace and Autosampler Assembly.
2. Fit the Accessory Mount and iSQ Module assembly in the spectrometer sample 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.

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.

The iSQ Module (continued)

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 Universal 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 its Lamps database

Thermo Hollow Cathode Lamps are guaranteed to emit spectra for a period of 24 months from date of dispatch or 5000 milliampere.hours usage, which ever occurs first. If either of these criteria is not met, 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. If the module needs repair, it must be returned to Thermo Electron Atomic Absorption, Cambridge, UK, 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.

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 S Series instrument covers are made of ABS plastic material, which can be damaged by strong solvents and concentrated acids.

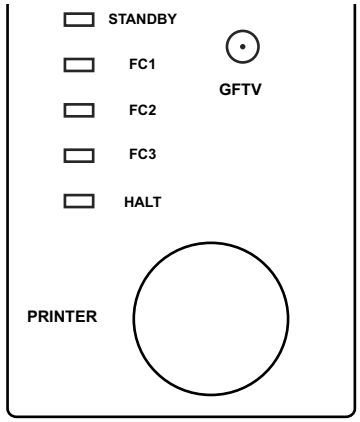
- Any spillage on the external covers or within the kitchen areas should be cleaned up immediately, using appropriate safety protection if necessary.
- Stains and marks on the covers should be removed with a soft cloth moistened with dilute detergent solution. Do NOT use any type of solvent based cleaners.

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 or local controller the status LED will instead flash slowly, once a second, otherwise it will flash rapidly, 5 times a second.



Connection panel and Status Indicators

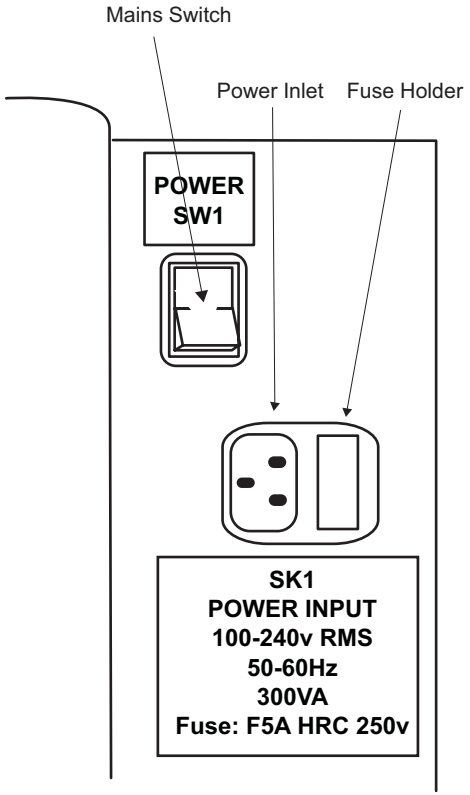
- The flash checksum failure may be corrected by downloading the spectrometer software, as described in the Software Manual. The other errors and the display of any code not shown in the table will require service attention.

Spectrometer fuse replacement

The S 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 right hand rear panel of the instrument.
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.



Mains Inlet and Fuse
(upper rear right corner of spectrometer)

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

Deuterium Lamp Alignment

The Deuterium (D2) lamp is located under the right hand panel, at the side of the Spectrometer.

The D2 Lamp should not normally require routine align-

Spectrometer Maintenance (continued)

ment. 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 - 200nm.
3. Use the system software to display the optical status of the spectrometer. The D2 Energy Bar Graph should be live.
4. Remove the right hand spectrometer panel by removing the four screws and lifting the panel away. 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.
7. Replace the right hand spectrometer panel.

Deuterium Lamp Replacement

The Deuterium Lamp 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 can replace it.

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. Refer to the figure, and remove the right hand spectrometer panel. Locate the Lamp Housing.
4. Disconnect the Lamp Cable.
5. Slacken the Lamp Securing Screws 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.

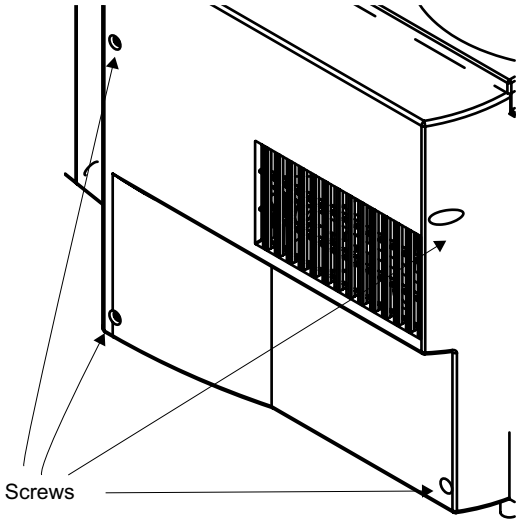
8. Fit the new Lamp into the mount.
9. Rotate it slightly clockwise, then tighten the Lamp Securing Screws.
10. Connect the Lamp Cable to the socket.
11. Align the new Lamp as described above.
12. Replace the right hand spectrometer panel.
13. Refer to your local Regulations, and dispose of the old deuterium lamp in a safe manner.

Spectrometer re-calibration

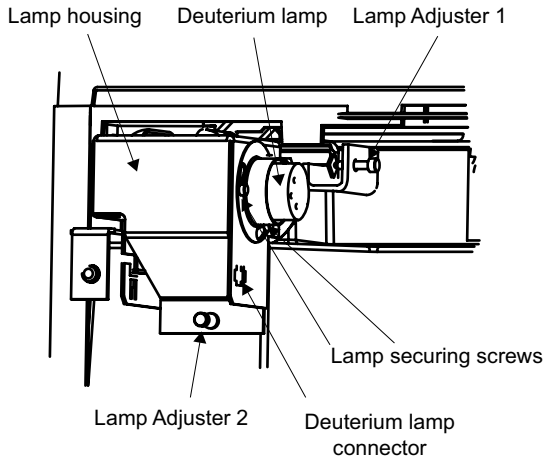
All S Series spectrometers are fully calibrated and tested before they leave the factory, and are again tested, and re-calibrated if necessary on installation.

However, facilities are provided for you to re-calibrate the monochromator and the burner height mechanism of the spectrometer, should it become necessary.

Monochromator re-calibration may be required if the wavelength displayed after an optical setup has been performed is consistently more than 0.1nm away from the nominal wavelength, or if error messages indicating



Spectrometer RHS panel



Deuterium lamp housing and adjustment

Spectrometer Maintenance (continued)

that the nominal wavelength cannot be found are repeatedly displayed.

Burner height re-calibration may be required if any major component of the flame atomisation system, such as the spray chamber itself, have been replaced.

To re-calibrate your spectrometer:

1. Refer to section 6.2.1 of the SOLAAR Software Manual to learn how to access the Customer Diagnostic facilities.
2. Follow the instruction provided to run the Calibrate Wizard.

Spares and Consumables

Deuterium Lamp	9423 420 30004

FLAME OPERATION

Flame Safety

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.

A FLAME AAS INSTRUMENT MUST NEVER BE LEFT UNATTENDED WHILE THE FLAME IS BURNING.

Flames

Nitrous oxide supported flames emit intense radiation which can damage your eyes if you view them 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 flame for at least 10 minutes before use.
- If carbon deposits appear, the flame must be extinguished immediately and the deposits removed.

Hydrogen fuelled flames are virtually invisible. To confirm that a flame is established, aspirate a solution containing a small amount of sodium to colour the flame.

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 or explosion occurs, complete the Flashback Report form included in the documentation supplied with your instrument, and return it to the address given on the form.

If you can establish and rectify the cause of the flashback, dismantle and clean the spray chamber, and replace any damaged components before using the instrument again.

If you cannot establish the cause of the flashback, or if the instrument shows signs of damage, disconnect all gas lines and power supplies, and do not use the instrument again until it has been inspected and repaired by a qualified Service Engineer.

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 6.9 bar (100psi), 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 protect yourself 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.
  - 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.

Flame Safety (continued)

- 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 and hydrogen supplies are fitted with flash back arrestors.

Acetylene

- is highly inflammable and forms an explosive mixture with air.
- has a strong garlic-like odour caused by impurities.
- if supplied or used above 0.62bar (9psi) 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 should therefore 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 (100psi).

Warning: Some specialist grades of acetylene may be supplied in solvents other than acetone. The S Series gas control systems have not been designed to withstand carryover of these solvents, which may cause severe damage to the instrument.

Hydrogen

- is highly inflammable and forms an explosive mixture with air
- is colourless and odourless
  - it is therefore very difficult to detect, and extra care should be taken to ensure that the system is leak free.

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 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 components may reach significantly elevated temperatures. Take sensible precautions to avoid touching them.

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.

Flame Safety (continued)

- 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.

Setting up the Flame System

Introduction

The Flame System consists of:

- the fuel and oxidant gas supplies and connectors.
- the Spectrometer 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 and:

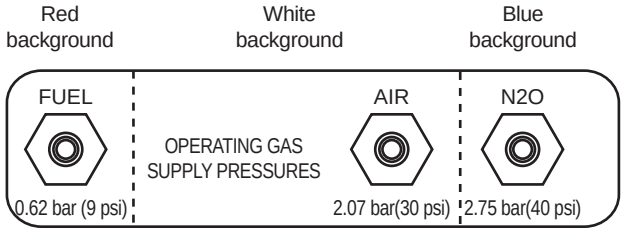
- inspect and, if necessary, replace the Over-Pressure Disc at the rear of the Spray Chamber.
  - a small dentist's mirror is provided to help you make this inspection.
- inspect and, if necessary, clean the Burner.
- inspect and, if necessary, clean the Spray Chamber and Nebuliser.
- confirm that the 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 250mm length of 0.5mm ID sample uptake tubing to the nebuliser.
- Use a longer length, and/or 0.4mm ID tube to reduce the uptake rate and 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:



Spectrometer Gas Connection Panel

Support gases

- Air 2.07 bar (30psi)
- Nitrous oxide 2.75 bar (40psi)
- Inert gas 2.07 bar (30psi)

Warning: Never attempt to use oxygen as a support gas.

Fuel gases

- Acetylene 0.62 bar (9psi)
- Hydrogen 0.7 bar (10psi)

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.

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_2O, using the BLUE hose supplied.

Inert gas-hydrogen flame

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

Air-hydrogen flame

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

Nitrous oxide-hydrogen flame

- connect the AIR supply to the connector marked AIR, using the CLEAR hose supplied.
- connect the HYDROGEN supply to the connector marked FUEL, using the RED hose supplied.
- connect the NITROUS OXIDE supply to the connector marked N_2O, using the BLUE hose supplied.

Burners

Two types of burner are available for your S Series spectrometer:

- 5cm slot Universal Titanium Burner
  - suitable for general purpose use with all flame types
- 10cm slot Titanium Burner
  - suitable for air-acetylene flames only. Sensitivity for elements measured with this flame will be improved compared to the Universal Burner.

Setting up the Flame System (continued)

To install a Burner
Refer to the figure and:

1. Open the 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 it 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.

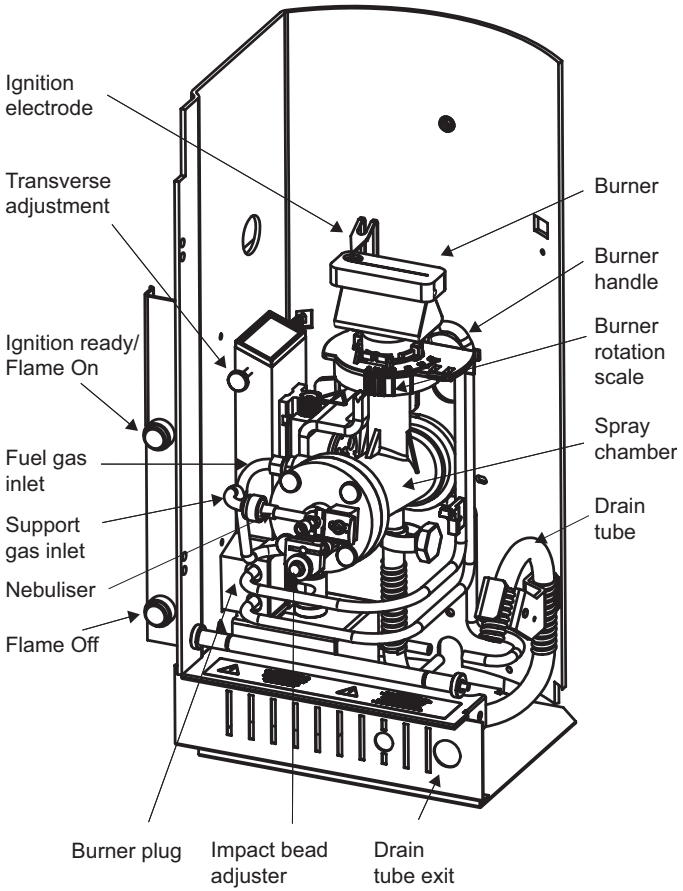
Organic Solvents

To measure samples in an organic solvent, you **must** first refer to the Flame Safety pages above. 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 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 drain.



Burner Compartment - general view

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 free from leaks, 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.

Automatic Gas Control System

Your S Series Spectrometer may be fitted with a fully automatic gas control system. In this case, there will be NO rotameter fitted to the front face of the left hand panel of the instrument.

Setting up the Flame System (continued)

To light the flame with an Automatic Gas Control System:

1. Confirm that the Ignition Ready light is flashing.
2. If you intend to use a hydrogen flame, and have connected hydrogen to the Fuel Gas inlet, you must select the hydrogen fuelled flame type, and execute the Flame Setup command in the system software before lighting the flame.
2. Press and hold the Flame On button 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.

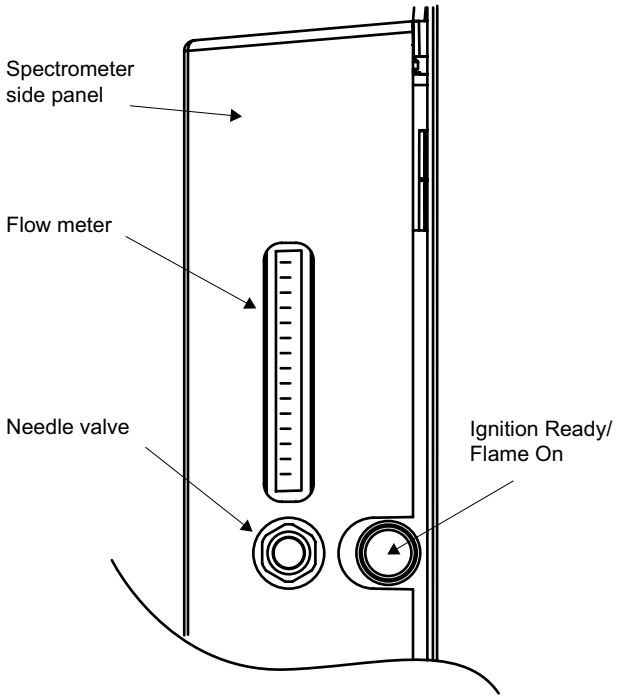
Warning: If you cannot ignite a flame after two attempts, it is likely that there is a problem with the burner, ignition system, or gas supplies. Refer to the Flame Maintenance section below and identify and rectify any problems before making another attempt to light the flame.

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 fuel rich flame.
   - the flame will then be shut off.



Semi-automatic Gas Control System

   - the flow of air will be maintained for approximately 30 seconds to ensure that all flammable gases are flushed away.

Semi-automatic Gas Control System

Your S Series Spectrometer may be fitted with a semi-automatic gas control system. In this case, there will be a Rotameter and Fuel Flow Control valve fitted to the front face of the left hand panel of the instrument.

You must use the Fuel Flow Control valve at the left hand side of the Flame compartment, and the Fuel Flow rotameter to adjust the fuel gas flow rate.

   - The Rotameter is approximately calibrated in L/min of acetylene, and for an air-acetylene flame, the indicated flow rate should correspond to the flow rate values given in the Cookbook.
   - For a nitrous oxide-acetylene flame, the fuel flow rate to the flame will be automatically boosted by the Gas Control system, and this boosted flow will NOT be shown on the Rotameter. To correlate the flow rate displayed on the Rotameter with the values given in the Cookbook, you should add 3.6L/min to the displayed value.

Air will flow through the instrument as soon as the air supply is turned on. The gas control system will automatically change over to nitrous oxide and increase the fuel flow rate under control of the system software when required, and will automatically change back to air before extinguishing the flame.

To light the flame with a Semi-automatic Gas System:

1. Confirm that the Ignition Ready light is flashing.
2. If you intend to use a hydrogen flame, and have connected hydrogen to the Fuel Gas inlet, you must select the hydrogen fuelled flame type, and execute the Flame Setup command in the system software before lighting the flame.
2. Press and hold the Flame Off button.
3. Adjust the Fuel Flow Control valve until the Fuel Flow Rotameter reads approximately 1.2L/min.
4. Release the Flame Off button and wait until the gas flow ceases, and the Ignition Ready light starts to flash again.
5. Press the Flame On button until the flame is established.

Setting up the Flame System (continued)

- 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.

6. Use the system software to select the oxidant gas type you want to use, and then perform a flame setup operation if necessary. Adjust the Fuel Flow Control valve to obtain the type of flame you require.

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 fuel rich flame.
   - the flame will then be shut off.
   - air will continue to flow through the instrument until the air supply is turned 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 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.
   - Do not forget to set the Fuel Gas flow rate back to the original value 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 burning, as described above.
2. Change the flame type to Nitrous Oxide-Acetylene, and set the Fuel Gas flow rate to 3.6 - 3.8l/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.
   - Do not forget to set the Fuel Gas flow rate back to the original value before starting your analysis.

Burner Alignment

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

If you intend to work with an organic solvent, you should align the burner using aqueous solutions first, to minimise the hazards of working with Sample Compartment Door open in the vicinity of flammable solvents.

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 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 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. 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.

Setting up the Flame System (continued)

Impact Bead alignment

Align the Impact Bead by aspirating a suitable sample solution, and adjusting the Impact Bead position to obtain the maximum absorbance signal.

To align the Impact Bead:

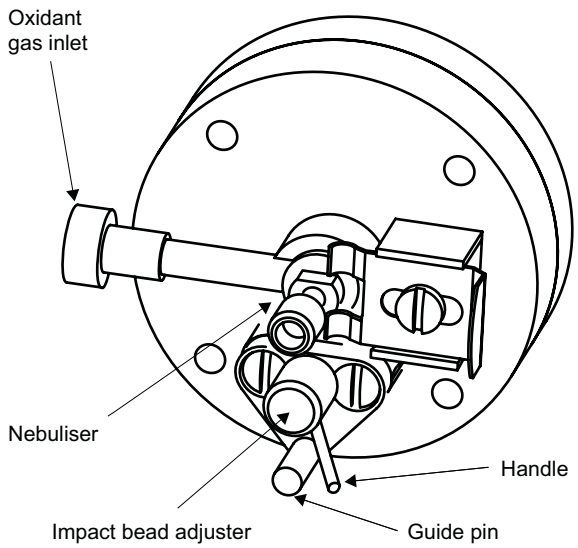
1. Set up a suitable analysis with the system software, install a hollow cathode lamp, and perform an Optical Setup. Ensure that the live absorbance signal is displayed.
2. Light the flame, and allow a few minutes for it to stabilise.
3. Align the Burner, as described above.
4. Aspirate deionised water 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.
   - S Series Spectrometers may be fitted with either a Micro-adjustable Impact Bead control or a Standard Impact Bead control. Refer to the figure above to identify the type of control and the adjustment required.

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

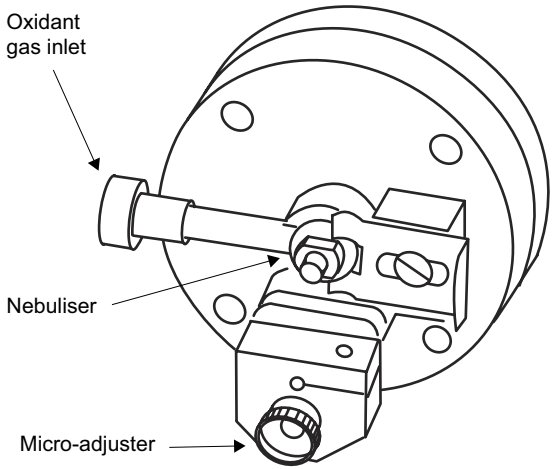
Warning: Do *not* attempt to adjust the Impact Bead position too far towards the Nebuliser as it may damage either the Nebuliser or the Impact Bead surface.

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.



Standard Impact Bead Adjuster



Micro-adjustable Impact Bead

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 here a click and the gases will be vented through the Burner.
4. Release the Flame Off button.

Flame System Spares and Consumables

Universal Titanium Burner (50mm)	9423 420 31011
Air/acetylene Ti Burner (100mm)	9423 420 31021
0.4mm sample uptake tube	9423 390 05411
0.5mm sample uptake tube	9423 390 05421
Acetylene gas pressure regulator	9423 352 43881
Hydrogen gas pressure regulator	9435 179 21191
Nitrous oxide gas pressure regulator	9423 354 03111
Acetylene flash back arrestor	9423 393 46061
Hydrogen flash back arrestor	9423 393 46071

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; system cleanliness is most important.

S Series Spectrometers can accept both the Universal (50mm) and the 100mm Titanium Burners. Both of these are cleaned and dismantled in the same way.

Safety

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

Burner

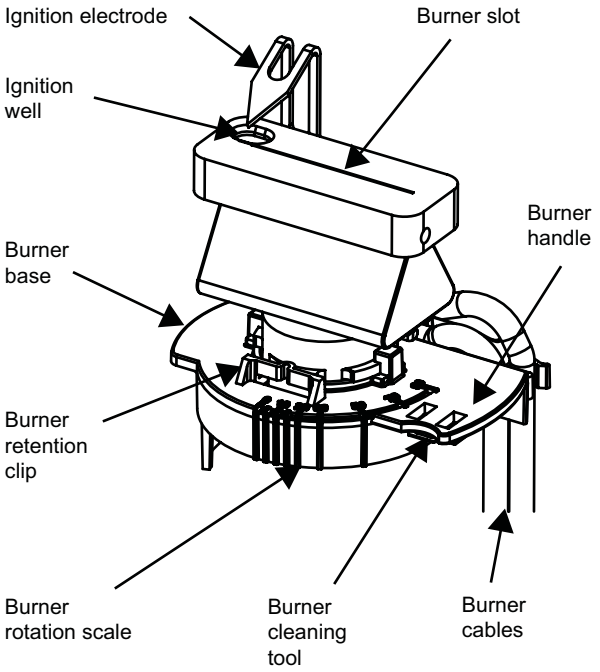
DO NOT ATTEMPT TO CLEAN THE BURNER WHILE A FLAME IS BURNING, AND ENSURE THAT THE BURNER HAS COOLED BEFORE HANDLING IT.

To clean the external surfaces of the Burner

- Clean and polish the top surfaces using a mild abrasive soap cleaner.
- 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.
- Wash the Burner with detergent solution and rinse with deionised water. Dry the Burner carefully before using it again.

To dismantle the Burner

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



Universal titanium burner head

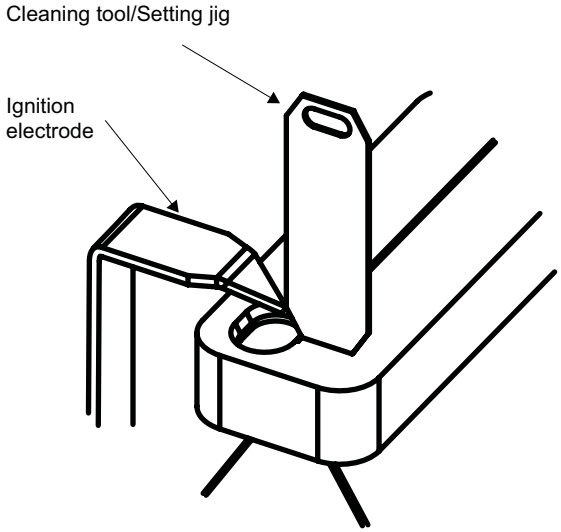
To clean the internal surfaces of the Burner

- 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.

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

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.



Ignition electrode adjustment

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.

To align the Ignition Electrode

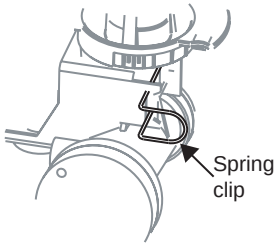
1. Refer to the figure, and place the Cleaning Tool/Setting Jig as shown, aligning the bottom corner of the chamfer with the edge of the Ignition Well.
2. Gently bend the Electrode until the tip touches the top corner of the chamfer on the Setting Jig, as shown above.

Flame System Maintenance (continued)

Spray Chamber

To clean the Spray Chamber

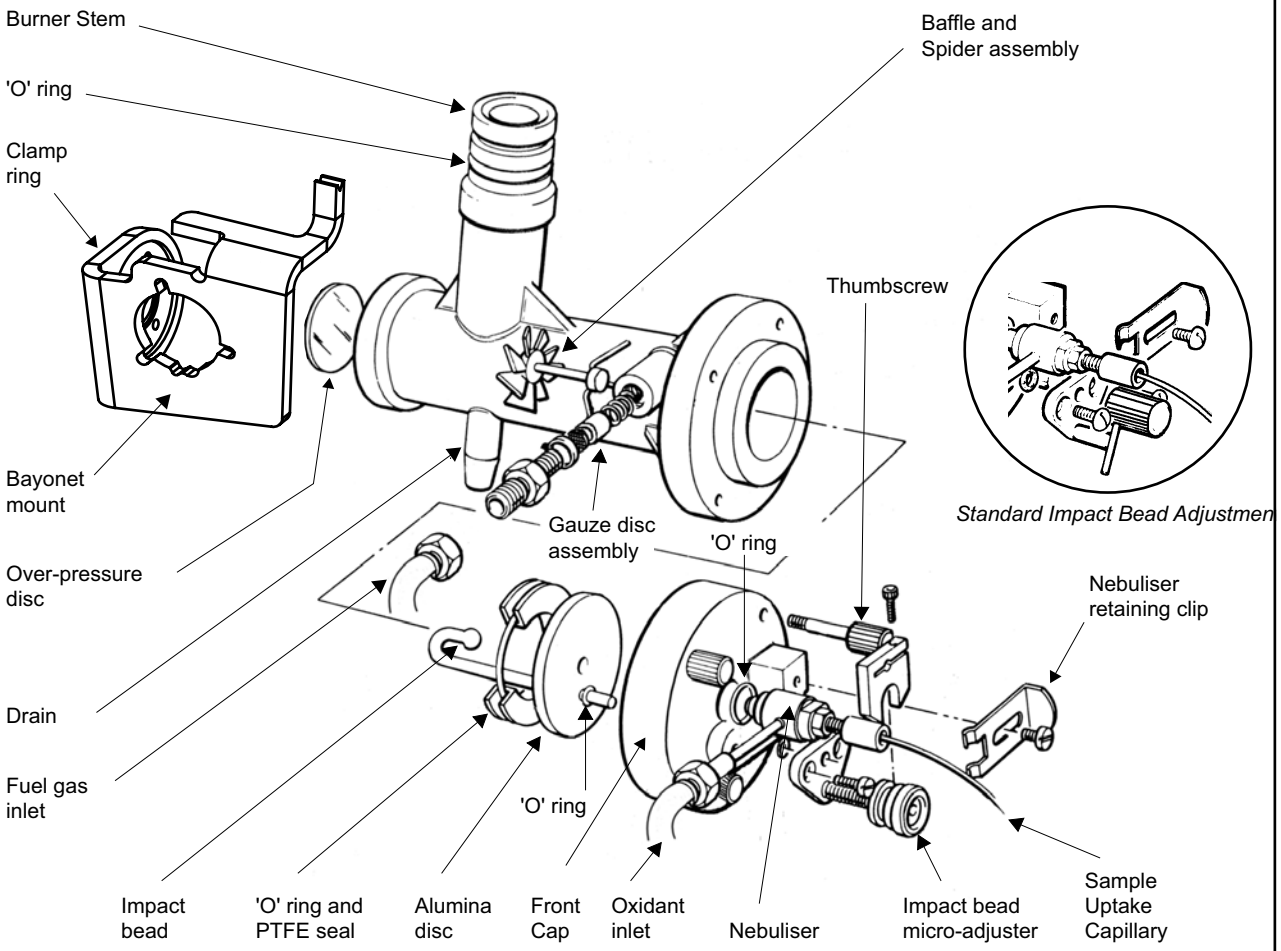
- Aspirate a solution of dilute (1% v/v) hydrochloric acid, or a solution of laboratory detergent, whichever is most appropriate. Aspirate the solution for 5 minutes, then aspirate deionised water.
- For more thorough cleaning, dismantle the Spray Chamber, clean the individual components with laboratory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



Spring Clip Spray Chamber Mount

To dismantle the Spray Chamber

1. Use the system software cammnds to raise the Burner until the Buner Height is less than 10mm.
2. Remove the Burner.
3. Empty the Spray Chamber Drain.
4. Disconnect the gas lines to Spray Chamber and disconnect the Drain Tube.
5. Refer to the figure above to identify the type of Burner Assembly fitted to your instrument.
   - If your Spray Chamber is secured with a spring clip, the squeeze the spring clip to release it, and remove the Spray Chamber.



Nebuliser and Spray Chamber Components

Standard Impact Bead Adjustmen

Flame System Maintenance (continued)

- If your Spray Chamber is fitted with a bayonet mount, then rotate the Spray Chamber anti-clockwise about about the mount axis to release it and the Clamp Ring/Mounting Bracket from the bayonet mount, and remove the assembly.

5. Remove the Spray Chamber.
6. Unscrew the 4 thumbscrews, and remove the Spray Chamber Front Cap.
7. Unscrew the 3 Rear Clamp Ring screws, and remove the Clamp Ring and Over-Pressure Disc.
8. Push the Baffle Assembly out of the Spray Chamber body towards the front.
9. Unscrew the Fuel Gas Coupling from the Spray Chamber body, and remove the Gauze Disc assembly.

To dismantle the Cap Assembly fitted with the Micro-adjustable Impact Bead control

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 both O- rings.

To dismantle the Cap Assembly fitted with the standard Impact Bead control

1. Using a suitable Allen key, slacken the grub screws securing the Adjustment Knob to the Impact Bead shaft.
2. Remove the Adjustment Knob and withdraw the Bead from the Alumina Disc side of the cap.
3. Remove the Seal and the Alumina Disc.
4. Undo the securing screws, and remove the Bead Adjustment Assembly.
5. Slacken the Nebuliser Retaining Clip screw, withdraw the Clip, and then remove the Nebuliser and both O- rings.

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. 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.
6. Either:
   - Adjust the Micro adjustable mechanism to its fully out position, then tighten the clamping screw to secure the adjuster to the bead shaft.

   Or:
   - Slide the Bead Adjuster Knob on to the bead shaft, and orient it so that the handle rests against the support on the left hand side of the moulding, then tighten the grub screws to secure the Knob to the shaft.
7. Operate the mechanism, and confirm that the Impact bead slides freely in and out, while remaining in line with the Nebuliser Nose Cone.
8. Refit the 'O' ring and PTFE Seal to retain the Alumina disc.

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.
7. 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 the blockage.

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 10ml measuring cylinder with deionised water.
2. Fit a standard length (250mm) of 0.5mm ID Nebuliser Capillary.
3. Light an air/acetylene flame.

Flame System Maintenance (continued)

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 between 4.5 and 5.5ml/min.

Drain

- 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.

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.
   - Air will flow through a spectrometer fitted with a Semi-automatic Gas System under these conditions.
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.
   - The air flowing through a Semi-automatic Gas System will cause the contents of the Drain to be blown out to waste.
6. Vent the Gas System of a spectrometer fitted with an Automatic 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.
7. Remove the bung from the top of the Spray Chamber, and 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 four years.
- Run the Gas Leak Test at least once every six months, or whenever the instrument is moved, and rectify any leaks found.

Gas Leak Test

The software commands necessary to run the Gas Leak test are included in the SOLAAR OQ Tests Data Station application, and on the Utilities page of the Local Control software.

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

Instructions for performing the Gas Leak Test with the Local Control software are given in the Local Control Software Manual.

Spares and Consumables

S Series User Spares Kit	9423 450 30001
Universal (50mm) Titanium Burner	9423 420 31011
100mm Titanium Burner	9423 420 31021
Nebuliser	9423 390 05481
Nebuliser Probe	9423 390 05441
Spray Chamber Renovation Kit	9423 390 05461
Replacement 'O' ring Kit	9423 390 05151

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

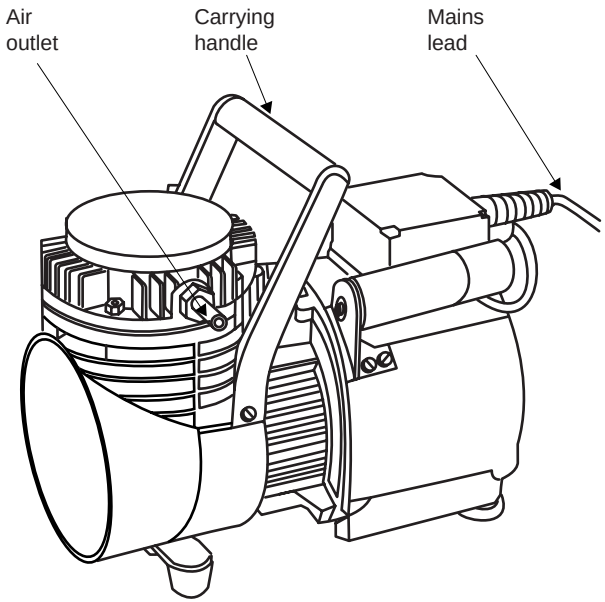
- Observe electrical safety precautions.
- Use the accessory in a well ventilated position.
- Ensure that the accessory is not close to a heat source.
  - This may cause poor removal of water vapour from the air, causing flame instability.
- Do not use the accessory as a pump for combustible liquids 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 ± 10%
9423 393 34226	220V/60Hz ± 10%
9423 393 34115	110V/50Hz ± 10%
9423 393 34116	110V/60Hz ± 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:



Air Compressor

   - 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's Air inlet connector.
7. The Compressor is now ready to use.

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 (30psi).
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 (2psi), 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.)

Gilson 221XL and 222XL Flame Autosamplers

Introduction

These are autosampling accessories for flame and vapour systems.

- 221XL - 14 to 60 sample capacity.
- 222XL - 14 to 254 sample capacity.
- The 222XL autosampler will also accept carriers for four 96 well microplates.

They are compatible with

- Normal flame operation.
- Flame dilution with the ID100 Autodilutor Accessory.
- Vapour operation with the VP90/VP100 accessories.

A Wash Kit is also available, to provide wash facilities.

Safety

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

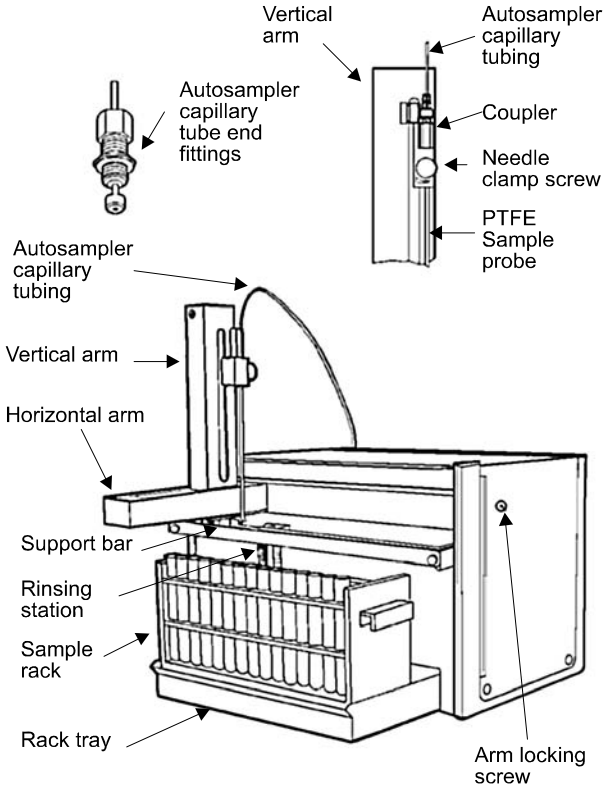
Installation

Assembly

1. Unpack and assemble the unit as detailed in the Installation Manual supplied, fitting the Teflon Sample Probe and associated components in place of the standard stainless steel Probe.
2. Position the Autosampler on the Accessory Trolley, or on the bench in front of the Sample Compartment.
3. Install the polypropylene Rack Tray.

System interconnections

1. Set the microswitches on the rear panel of the unit as shown in the figure.
2. Connect the 9-25 way RS232C Interface Cable supplied between one of the Accessory connections on the Spectrometer, and the 25 pin RS232C socket on the rear of the unit.

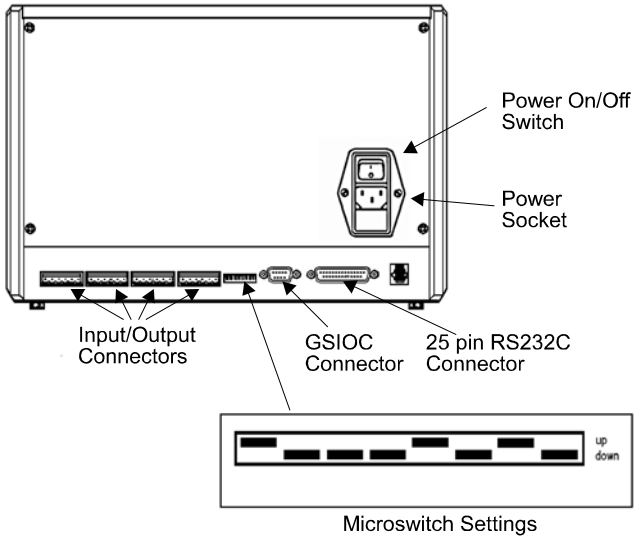


Gilson 221XL Sample Changer - General View

3. Connect a suitable mains cable to the mains inlet connection on the unit.

Capillary Tubing connections

1. Select the appropriate Capillary Tubing Interface.
2. Connect the Capillary Tubing to the Threaded Coupler on the top of the Autosampler Teflon Sample Probe.
3. Connect the free end of the Capillary as follows:



Gilson 221XL - Rear View

- **Normal Flame operation.** Cut back the Capillary to the minimum convenient length, and connect the free end to the Nebuliser.
- **Other Accessories.** Refer to the appropriate accessory page.

Sample Racks

1. Fit the required Sample Rack(s) into the Rack Tray.
   - The **221XL** will take one type 21, 22 or 24 Rack.
   - The **222XL** will take one Rack 21, and up to 4 type 21, 22 or 24 Racks. It will also take two Type 81 carriers, each of which will hold two 96 well microplates.
2. The Rack capacities are:
   - Type 21 - 60 positions, each of 8ml volume.
   - Type 22 - 44 positions, each of 12ml volume.
   - Type 24 - 14 positions, each of 50ml volume.
   - Type 81 - 4 x 96 positions, each of 2ml volume.

Gilson 221XL and 222XL Flame Autosamplers (continued)

222XL with 96 well microplates

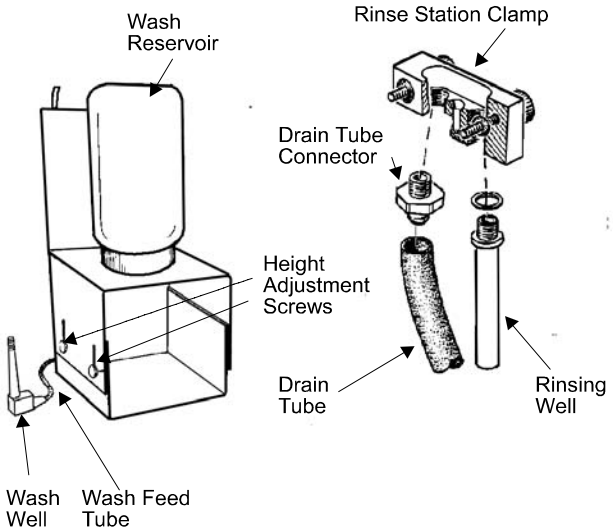
To use the 222XL with 96 well microplates, you will need the 96 well Microplate Update kit, part number 9423 470 03481. This kit contains two Type 81 support platforms which locate in the rack tray. Each support platform can accommodate two 96 well microplates. You will also require one Type 14 racks to contain the standard solutions, which should be fitted at the left hand position in the rack tray.

Note that:

- Operation of the 222XL with 96 well microplates is only supported by the SOLAAR Data Station software v8.13 and above. It is NOT supported by earlier versions, nor is it supported by the Local Control software.
- The maximum capacity of the individual wells on the 96 well microplate is around 2ml. Remembering that the nebuliser uptake rate is approximately 5ml/min, select the signal measurement time, number of resamples and nebuliser uptake delay carefully to ensure that you do not run out of solution before the measurement is finished.

Wash Kit Installation

1. Place the Wash Kit to the left of the Autosampler.
2. Locate and remove the Rinsing Station from the Autosampler Support Bar.
   - Only the left hand Rinsing Station of the 222XL is used.
3. Unscrew the Rinse Well, and replace it with the Wash Well supplied with the Wash Kit.
4. Connect the black Drain Tube provided with the Autosampler to the Drain Tube Connector, and place the free end in a suitable drain container.
5. Refit the Rinsing Station to the Support Bar, with the Wash Feed tube running to the left of the autosampler.



Wash Kit - General View

6. Fill the wash reservoir with a suitable wash liquid (deionised water, or dilute acid to match your standard solution composition). Allow the liquid to flow into Wash Well.
7. Adjust the height of the Wash Reservoir so that the liquid fills the Wash Well without overflowing.
8. The Wash Kit is now ready to use.

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 or Local Control software facilities to define the type and number of Sample Racks, and the number of solutions required.
4. Fit the Rack(s) to the Autosampler.
5. Refer to the Autosampler Loading Guide facility in the software, and place the solutions in the correct positions in the Rack(s).
6. The system is now ready for you to start the analysis.

Maintenance

- Ensure that the Autosampler is switched off and isolated from the mains supply before carrying out any maintenance.
- Wipe the exterior surfaces with 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 Gilson 221XL and 222XL Autosamplers do NOT require routine lubrication.
- The Autosamplers must never be picked up by either the Vertical or Horizontal Arms, but only by the Support Bar.
- If the Autosampler is to be transported, we advise that the Vertical Arm is removed, and the Horizontal Arm is locked with the Locking Screw provided. These procedures are described in the Installation Manual supplied with the units.

Spares and Consumables

Rack Type 21 (60 positions)	9423 470 03301
Rack Type 22 (44 positions)	9423 470 03391
Rack Type 24 (14 positions)	9423 470 03381
Sample Tubes - Rack 21	
Glass (100 off)	9423 470 03321
Polypropylene (1000 off)	9423 470 03311
Sample Tubes - Rack 22	
Glass (100 off)	9423 470 03341
Polypropylene (10 off)	9423 470 03331
Sample Tubes - Rack 24	
Glass (30 off)	9423 470 03361

Gilson 221XL and 222XL Flame Autosamplers (continued)

Polypropylene (10 off)	9423 470 03351
Rack Tray for 222XL	9423 470 03471
Teflon Sample Probe	9423 470 03211
Autosampler to Nebuliser Connector Tube Assembly	9423 393 68121
Accessory Trolley	9423 393 46131
96 well microplate support kit	9423 470 03481

The ASX-510/520 and ASX-260 Flame Autosamplers

Introduction

The ASX-510/520 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 and VP90 accessories.

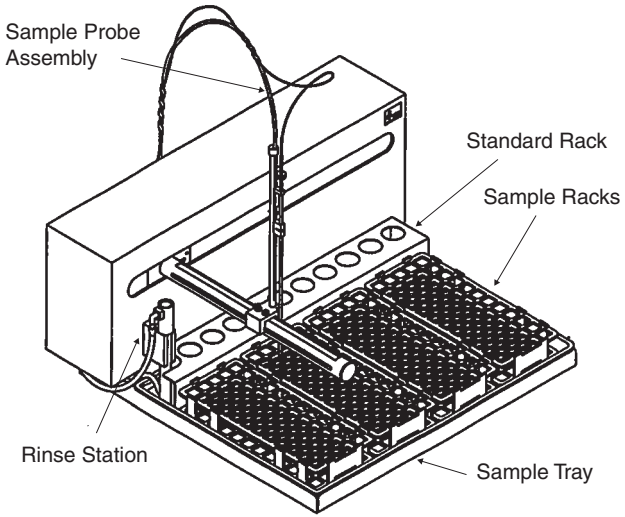
Safety

- 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.

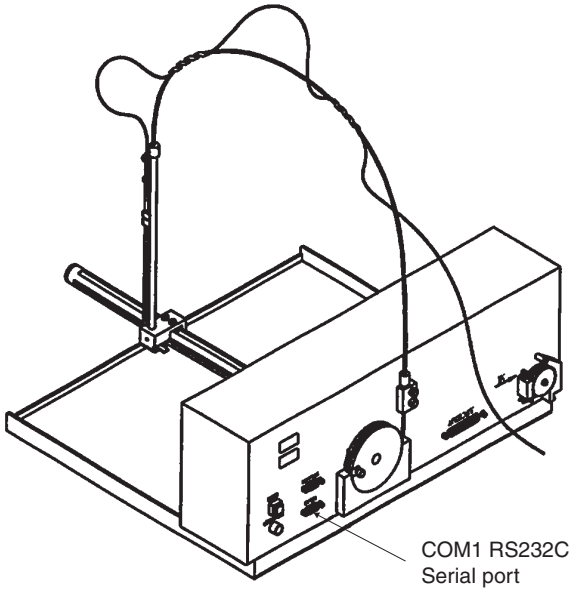


Cetac ASX-510 Sample Changer - General View

   - Two types of uptake tube are supplied - one with an internal diameter of 0.5mm, and one with an internal diameter of 0.8mm. The 0.5mm 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 0.8mm ID uptake tube will restore the sensitivity.
4. Connect the Rinse Station as described in the Autosampler Manual. 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.



Cetac ASX-510 - Rear View

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.

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.

Note: If you choose to use the 0.8mm ID uptake tube

supplied, you will find a short length of 0.5mm 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 0.5mm 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:

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

3. Place the Standard Tubes into the rear Standards Rack.

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 or Local Control 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.

Maintenance

- Ensure that the Autosampler is switched off and isolated from the mains supply before carrying out any maintenance.
- 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-510/520 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.

Spares and Consumables

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 Assembly	9423 470 03981
Sample Probe (0.5mm)	9423 470 03991
Sample Probe (0.8mm)	9423 470 04171
Tray Sub-assembly with Standard Rack	9423 470 03951
Rinse Station	9423 470 03961
Drain Pump Tubing/Connector Kit	9423 470 03971
Standard Tubes (50ml polypropylene, 500 off)	9423 470 04151
50ml Polypropylene Sample Tubes (for 21 position rack, 500 off)	9423 470 04101
30ml Polypropylene Sample Tubes (for 24 position rack, 500 off)	9423 470 04111
18ml Nalgene Sample Tubes (for 40 position rack, 100 off)	9423 470 04121
14ml Polypropylene Sample Tubes (for 60 position rack, 1000 off)	9423 470 04131
8ml Polypropylene Sample Tubes (for 90 position rack, 1000 off)	9423 470 04141
Accessory Trolley	9423 393 46131

The ID100 Autodilutor Accessory

Introduction

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

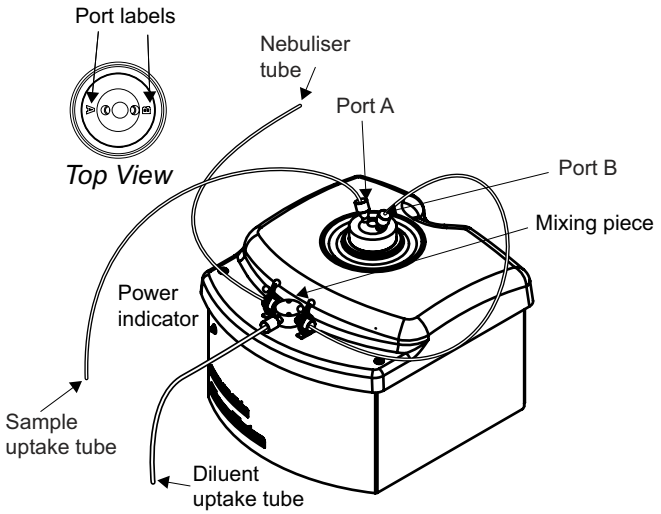
- It can be used manually or with any of the Flame Autosamplers.
- It is compatible with both Data Station and Local Control systems.
- It is not compatible with the VP90 and VP100 Vapour Generation accessories.

Installation

Before installing your Autodilutor, refer to the figure and familiarise yourself with the parts shown. Check the version numbers of your SOLAAR software - the SOLAAR Data Station software must be v9.1 or higher, the spectrometer firmware must be v1.2 or higher, and the Local Control software must be v2.2 or higher.

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 1.5mm OD, 0.5mm 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 1.5mm OD, 0.8mm 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 minature fitting at the Autodilutor end.



ID100 Autodilutor - General View

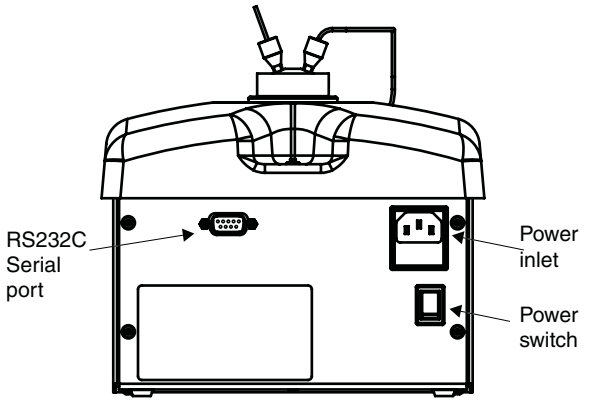
6. Locate the 3.2mm OD wide bore PTFE tubing supplied, and fit it to the remaining free port of the mixing piece, using the red finger nut and ferrule connector. Trim the tube to a suitable length and place the free end in the diluent container.

Manual Sampling

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

Gilson Autosamplers

Fit a suitable length of 0.8mm ID PTFE tubing to Port A of the Autodilutor using a grey minature 10/32 connector, then fit the other end to the Gilson PTFE Sample Probe using the spare white finger nut and ferrule connector, or a Gripper Fitting, if one is available.



ID100 Autodilutor - rear view

Cetac Autosampler

Either fit a spare grey minature 10/32 connector to the free end of the Cetac Sample Probe tube, and fit that to Port A of the Autodiluter, or fit a 10/32 connector to a short piece of 0.8mm 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

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

To fit a minature 10/32 connector:

1. Use a sharp blade to trim the end of the 1.6mm 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.

The ID100 Autodilutor Accessory (continued)

5. Trim the tapered end from the tubing, leaving 0.5 - 1mm 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, and place it in the suitable container. Place the Diluent Uptake tube in this container.
   - 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. If you are working with the SOLAAR Data Station software, 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. If you are using the SOLAAR Data Station software, you must set the Pre-Fill volume on the Sampling tab of the View.Options dialogue.
   - A Pre-Fill volume of 1200 - 1500µl will be suitable for use with manual sampling, and with the 221XL and ASx-260 autosamplers. Up to 2000µl will be needed for the larger 222XL and ASX-510 autosamplers.
   - 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, using either the Data Station or the Local Control.
   - 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 the 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 while it is operating, it is possible that a small amount of liquid will be ejected from the Leak Port. If this happens, stop the Dilutor 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's 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 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

The Slotted Tube Atom Trap (STAT)

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 50mm Universal Burner.
- 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:

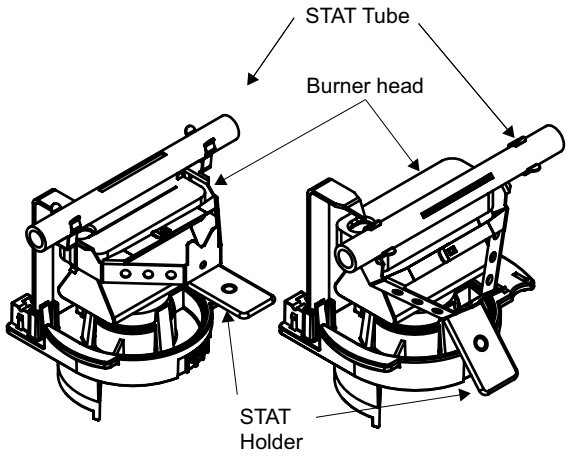
1. Remove the Burner from the Spectrometer.
2. Orientate the burner with the Burner Handle to the right and the Ignition Electrode to the left.
3. Gently push the STAT Holder assembly on to the Burner until the Securing Clips click into the Locating holes on the Burner.
4. Refit the Burner to the Spectrometer.

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.

Alignment

1. Fit the Burner and STAT Holder assembly to the Spray Chamber Stem and insert the Burner Plug into its socket on the left of the Sample Compartment.



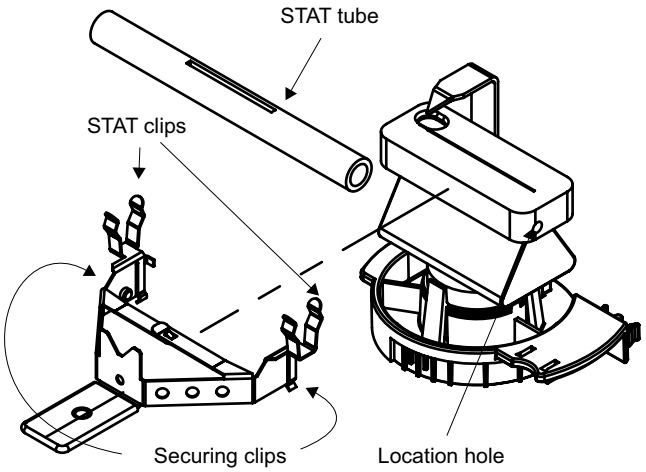
Operating position Parked position

STAT Tube and Holder

2. Fit a STAT Tube to the Holder, then move the Holder so that the Tube is away from the Burner slot.
3. Install a suitable hollow cathode lamp, and perform an optical set up.
4. Move the Holder so that the STAT Tube is 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 STAT Tube.
7. Move the STAT Tube away from the Burner slot.

Operation

1. Ensure that the STAT Tube is away from the Burner slot.
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.



STAT Assembly

4. When the flame is established, move the STAT Tube into its operating position over the Burner slot.
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 STAT Tube from the Holder after using it. The holder can be left fitted to 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 STAT Tube in its operational position, the Tube 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 off)	9423 393 35021

The Manual Aliquot Microsampling Accessory

Introduction

- This is a simple accessory to allow you to use manual Aliquot Microsampling to measure your samples.
- Aliquot Microsampling is particularly useful when:
  - the available volume of sample is limited
  - the sample is too viscous, or contains too high a concentration of dissolved solids for conventional aspiration.

Safety

There are no additional safety hazards associated with this accessory, but you should review the Safety sections concerned with normal flame operation before using it.

Installation

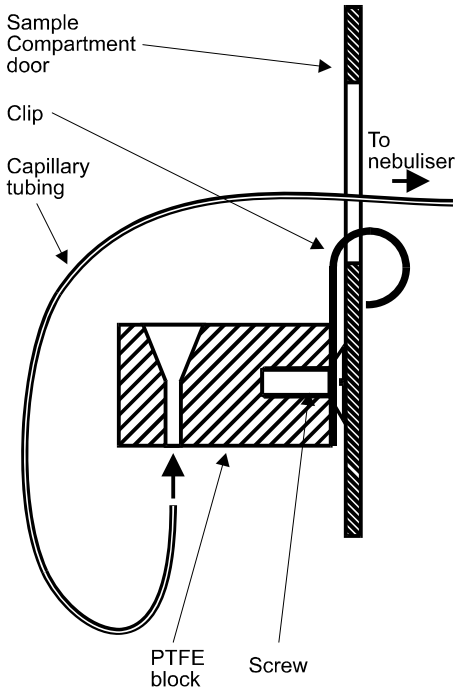
The accessory is fitted to the lower edge of the aperture in the door of the left hand sample compartment, and secured with a small Plastic Clip.

To install the Manual Aliquot Microsampling Accessory

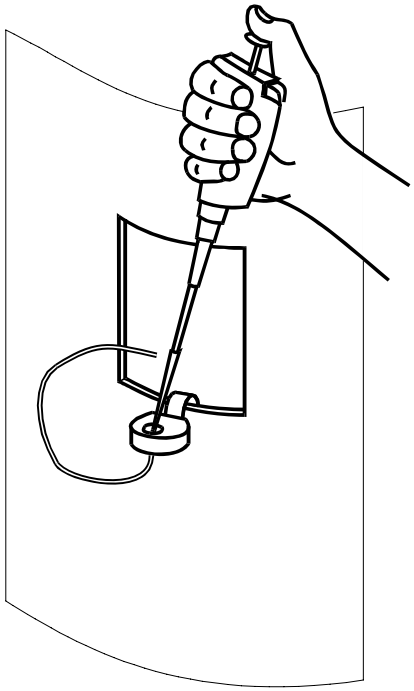
1. Attach the Plastic Clip to the PTFE Block using the countersunk screw provided.
2. Cut a 230mm length of the Capillary Tube provided with the accessory.
3. Push one end of the Capillary Tube into the hole on the base of the PTFE Block.
4. Remove the standard Uptake Tube from the Nebuliser.
5. Fit the PTFE Block and its Clip to the lower edge of the aperture in the Sample Compartment Door.
6. Feed the Capillary Tube through the Door aperture, and fit it to the Nebuliser.
7. Ensure that the Door is closed before using the accessory.

Operation

1. Set up and optimise the instrument in the normal way.



The Aliquot Microsampling Accessory



Sample Injection

2. Set up your Method.
   - Note that you must set the Signal Type parameter on the Spectrometer page to Transient Height or Transient Area.
3. Start a measurement cycle. When prompted, use a micropipette to inject a suitable volume (50 - 300µl) into the funnel.
4. Adjust the injection volume to obtain a clean, peak shaped signal, and optimise the Spectrometer measurement time to ensure that all the peak is captured.
5. Start your analysis in the normal way, and inject the solutions when prompted.
   - you can rinse the accessory with one or more injections of clean water between measurements.
6. When you have finished your analysis, clean the accessory and Spray Chamber by injecting several aliquots of clean water.

Maintenance

- Rinse the accessory thoroughly with clean water before and after use.

Spares and Consumables

Spare Capillary Tubing	9423 390 05421

The Variable Flow Auxiliary Oxidant Accessory

Introduction

- An auxiliary supply of Oxidant gas to the air/acetylene flame is necessary when using non-aqueous solvents to maintain the optimum flame chemistry.
- 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.
- This accessory is NOT compatible with S2 spectrometers fitted with semi-automatic gas control systems.
- 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

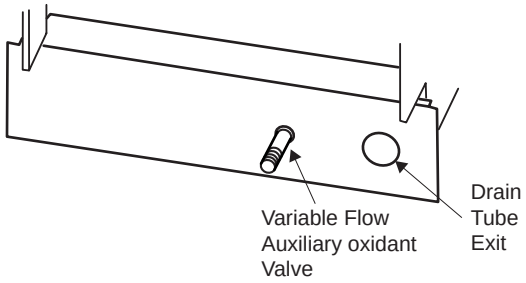
1. Set up the instrument in the normal way.
   - It may be beneficial to use a longer and/or narrower length of nebuliser uptake tube to reduce the aspiration rate of the sample, although this will also reduce the chemical sensitivity.
2. Ensure that the Auxiliary Oxidant parameter on the Flame page of the Method is turned on.
   - 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.

Spares and Consumables

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



Flame Compartment Valance withVariable Flow Auxiliary Oxidant Accessory fitted.

The Solvent Resistant Flame Kit

Introduction

This kit provides replacements for certain parts in the standard flame atomisation system that are susceptible to damage if subject to long term exposure to organic solvents. If you want to use your instrument to analyse this type of sample on a regular basis, we recommend that your instrument should be fitted with the Solvent Resistant Flame Kit.

Installation

The Solvent Resistant Flame Kit will have been installed at our factory, if you specified it on the order for your spectrometer.

It can also be installed as a field upgrade by a properly trained Service Engineer, authorised by Thermo to perform this work.

Do NOT attempt to install the Solvent Resistant Flame Kit yourself.

Components

Spray Chamber

The top of the Spray Chamber stem has been modified with a Teflon sleeve. The prevents the top of the stem from softening when ketone solvents such as methyl isobutyl ketone are run for long periods with a high temperature nitrous oxide supported flame.

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

Drain Tube

The Kit includes an extended length of convoluted PTFE Drain Tube that extends down to the drain vessel and so 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 perfluoroelastomer 'O' rings which are resistant to attack by certain solvents that cause the standard Viton 'O' rings to swell and degrade. The replacement 'O' rings are fitted at the Spray Chamber neck, the Spray Chamber Drain, and the Nebuliser to Spray chamber seal.

0.4mm 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 optimise the flame chemistry. This can be conveniently achieved by replacing the standard 0.5mm ID sample uptake tube with the 0.4mm uptake tube supplied in the kit.

Operation

The presence of the Solvent Resistant Flame Kit itself does not require any special operating procedures. However, using organic solvents with a flame Atomic Absorption Spectrometer is inherently hazardous. You MUST, therefore, refer to the relevant sections of this manual that describe the hazards and the procedures to minimise them, and then carry out an appropriate and comeprehensive Risk Assessment before starting your analysis.

A User Manual is supplied with the Solvent Resistant Flame Kit. This contains the material included in this section, and provides more detailed procedures for assessing and reducing the hazards, and for setting up and optimising your analysis.

Maintenance

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

Spares and Consumables

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

FURNACE OPERATION

Furnace Safety

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.

The GFS97 Furnace Head

- 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 spectrometer 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, excessively high temperatures can be generated, 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.

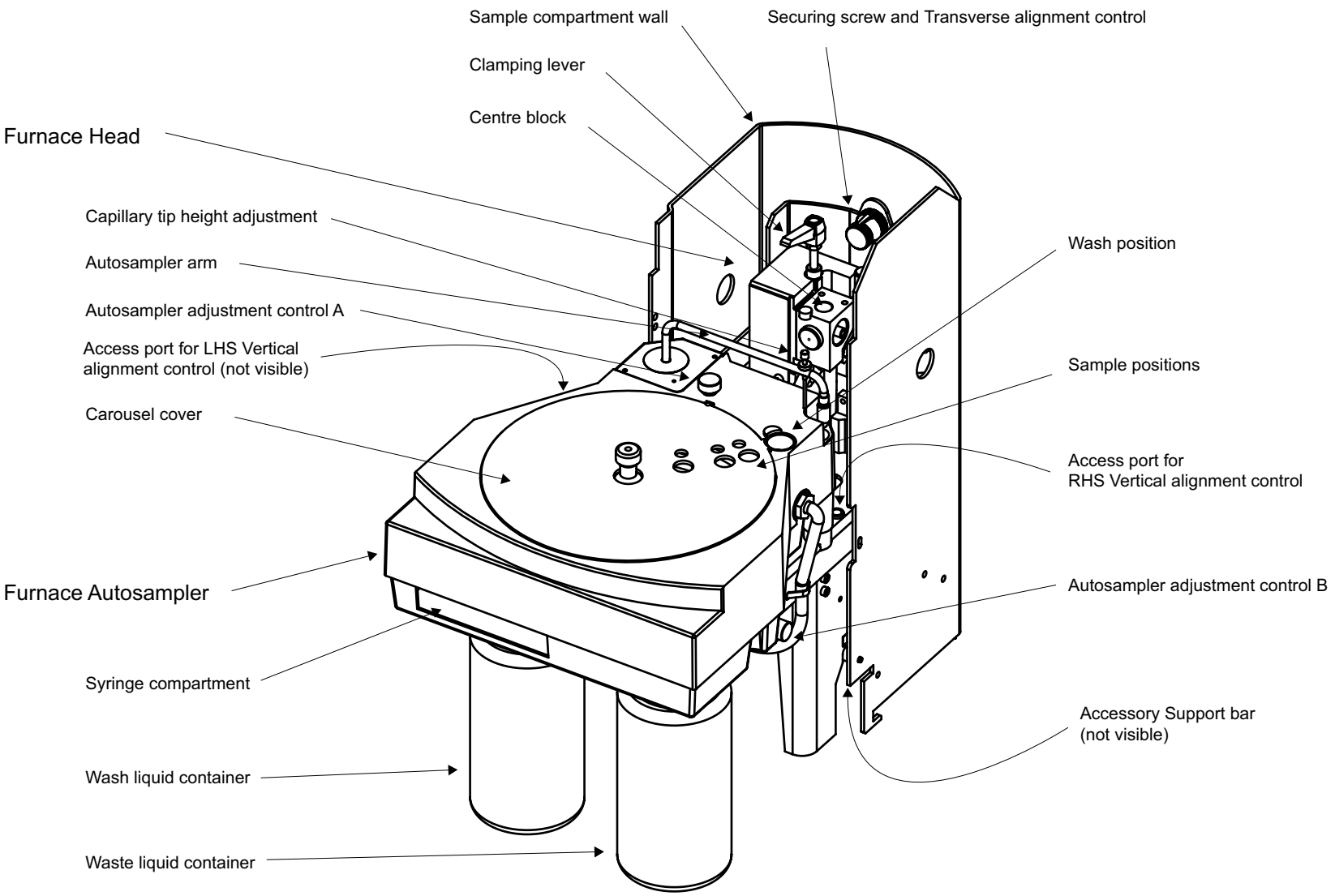
Fume Extraction

- 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.

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.

The GFS97 Graphite Furnace System.



GFS97 Integrated Graphite Furnace and Autosampler - general view

The GFS97 Graphite Furnace System

Introduction

- The GFS97 Graphite Furnace system is major accessories that can be fitted in the Sample Compartment of the S Series spectrometers.
- It replaces the flame atomiser with an electrically heated graphite cuvette, enhancing the analytical sensitivity by 100-1000 times and reducing the sample volume required to a few microlitres.
- The GFS97 accessory includes an integrated Furnace Autosampler, to automatically inject the sample solutions into the Graphite Furnace.

Installation

Your GFS97 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.

GFS97 Fitting and Removal

You will have to remove the GFS97 assembly if you want to use the flame or vapour atomisation systems on your spectrometer. When not fitted in the Sample Compartment of the spectrometer, the GFS97 should be parked between the LHS of the spectrometer and the Furnace Power Supply.

To remove the GFS97:

1. Refer to the figure, and identify the Securing Screw at the rear of the assembly.
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 fit the flame or vapour atomisation system that you want to use.
6. Refit the Sample Compartment Door.

To fit the GFS97 in the Sample Compartment:

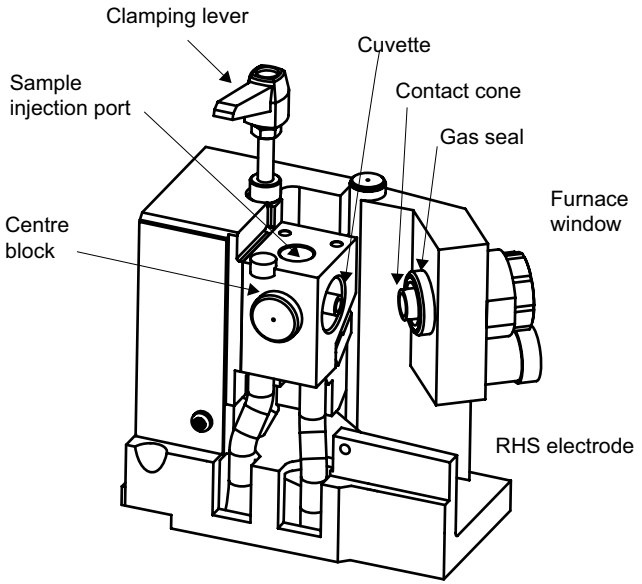
1. Use the system software to move the Burner to the Parked position.

Note: The Local Control software does not provide a Park Burner command. The burner can be parked by switching the spectrometer off and of again.

2. Refer to the appropriate section of this manual, and remove the Burner or Vapour atomisation accessory.
3. Remove the Sample Compartment Door, by lifting it off its hinges.
4. Move the GFS97 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 GFS97 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 GFS97 is now ready for use.

Operation - Graphite Furnace

The GFS97 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.



GFS97 Graphite Furnace Head

Graphite Cuvettes

The GFS97 will accept all types of Cuvette supplied by Thermo Electron Corporation 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.

The GFS97 Graphite Furnace System (continued)

- 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 uppermost.
4. Position the Cuvette in the Furnace Head, and carefully close the Head. The Cuvette will be automatically aligned as the Head is closed.
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 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.

Cuvette Clean
The system software provides a Cuvette Clean facility to remove any dust and contamination from the cuvette before it used. A new cuvette may require several Cuvette Clean cycles before it is ready for analytical use.

Operation - Furnace Autosampler

Autosampler 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 contamination is acceptable, by analysing a sample of the liquid before use.

Autosampler 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
Three types of Sample Cup are available for use with the GFS97 Autosampler. These are:

- **Polypropylene Cups** for general use. These will contain approximately 2ml of liquid, and are normally not re-used.
- **Fluoroplastic Cups** for critical applications. These also contain approximately 2ml, 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 1.5ml. However, the bottom of the cup is “V” shaped, allowing sample volumes down to 100µl 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 25ml.

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 critical applications.

Spares and Consumables

Normal Electrographite Cuvettes (10 off)	9423 393 95031
Pyro-Coated Electrographite Cuvettes (10 off)	9423 393 95071
Extended Lifetime Cuvettes (ELCs) (10 off)	9423 393 95041
Extended Lifetime Cuvettes (ELCs) (20 off)	9423 393 95051
Omega Platform Extended Lifetime Cuvettes (10 off)	9423 490 20101
Pyro-Coated Unridged Cuvettes (10 off)	9423 393 95091
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
Floor Standing 5L Waste Vessel	9423 390 05471

Graphite Furnace System Installation and 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 GFS97 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 reinstall it.

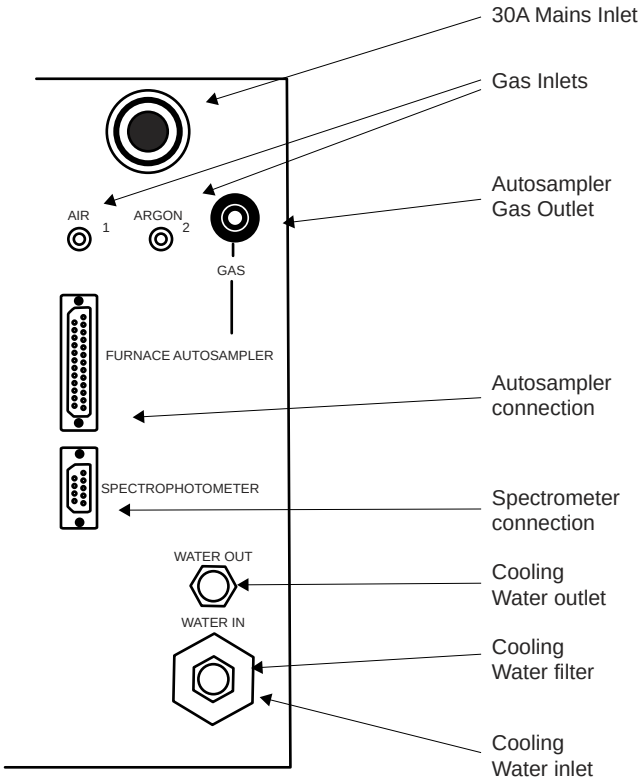
Services

Inert Gas

- This protects the hot cuvette from atmospheric oxygen, and flushes sample vapours from the cuvette interior.
- The Autosampler Wash facility is pressurised by the inert gas supply routed through the Furnace Power Supply unit.
- Argon is recommended; nitrogen can be used with some loss of performance for some elements.

To connect the Inert Gas Supply:

1. 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 1.1±0.14 bar (15±2 psi).
2. Locate the Autosampler Gas Inlet tube.



Furnace Power Supply Connections Panel

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

Alternate Gas

- An alternate type of gas can be passed through the cuvette to modify the sample behaviour.

To connect an Alternate Gas Supply:

1. 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 1.1±0.14 bar (15±2 psi).
   - The normal Inert Gas Supply must also be connected and turned on when the Alternate Gas is used.

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.

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 - 100psi), capable of providing a minimum flowrate of 0.7l/min is required.
  - Do ***not*** allow the water pressure to exceed 6.9 bar (100 psi).

To connect the Cooling Water Supply:

1. Connect the cooling water inlet and outlet hoses to the Water In and the Water Out 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.

Graphite Furnace System Maintenance (continued)

- Set the recirculating water temperature to about 5ºC above ambient temperature, providing that this is less than 30ºC.

Electrical Connections

- The GFS97 Furnace Head and Autosampler are controlled by the Spectrometer, via an RS232C serial connection.
- The GFS97 requires a single phase mains supply capable of providing 7.2kVA, as specified in the Pre-Installation Manual.

To make the electrical connections:

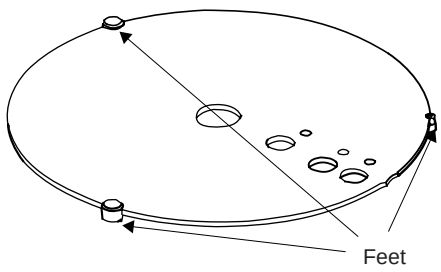
1. Connect the 9 pin serial port on the Furnace Power Supply unit to an Accessory port on the Spectrometer Connection Panel, using a standard RS232C cable.
2. Locate the Autosampler Connection cable.
3. Fit the free end of the cable to the Autosampler socket on the Furnace Power Supply connection panel.
4. Connect the Furnace Power Supply mains lead to a suitable mains outlet.

Autosampler Drain Assembly

The GFS97 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.



FS95 Cover

Autosampler Carousel Cover

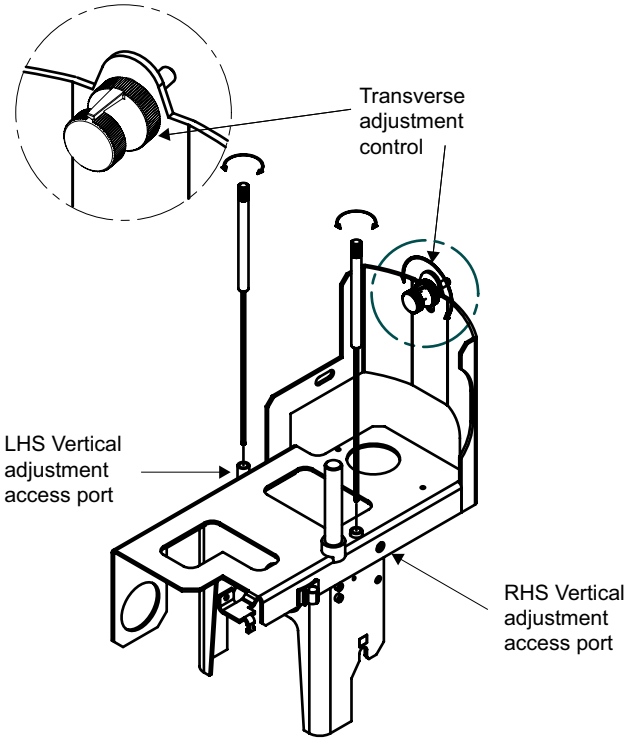
The Carousel Cover protects your sample solutions from airborne dust, and prevents contamination. It will also reduce evaporation of the sample solutions. In some ambient conditions, condensation can form on the lower surface of the cover, which can also contaminate the samples. You can fit small feet to the Carousel Cover to reduce the condensation on the under surface, although this will increase the sample evaporation rate. We recommend that you do ***not*** fit these feet, unless 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.

Alignment

Routine alignment of the GFS97 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.



GFS97 - Furnace Adjustment Controls

To align the Furnace Head

1. Remove the GFS97 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.

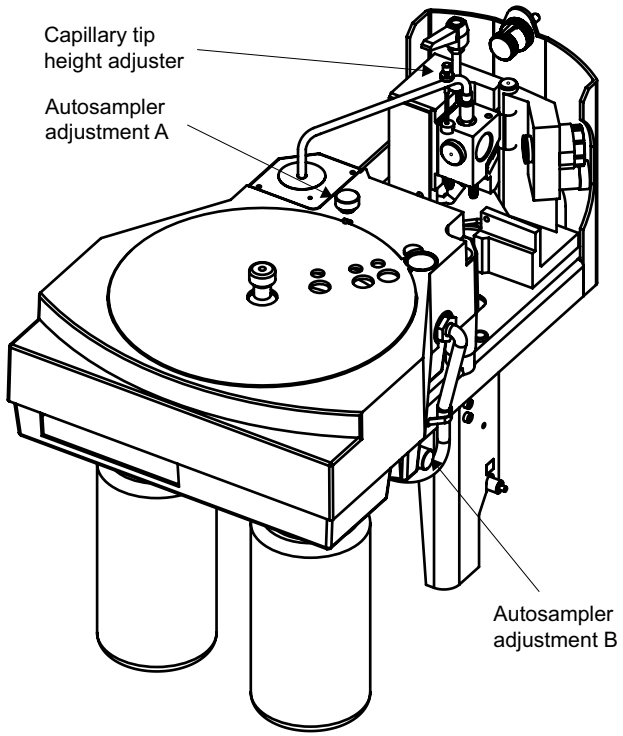
Graphite Furnace System Maintenance (continued)

- Ensure that you do NOT select any form of background correction.

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 GFS97 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 8mm 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.



GFS97 - Autosampler adjustments

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 1mm 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.

Maintenance - Graphite Furnace

Cleaning the 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.

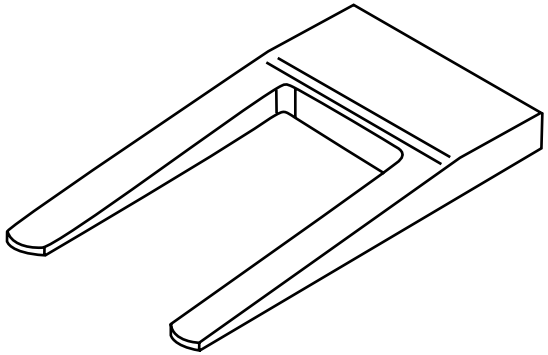
Furnace Head Windows

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

To clean the Furnace Head Windows

1. Remove the GFS97 from the Sample Compartment, and place it in a secure, accessible position in front of the spectrometer.

Graphite Furnace System Maintenance (continued)



Furnace Window Removal Tool

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 it's 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.
3. 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.
4. 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.

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.

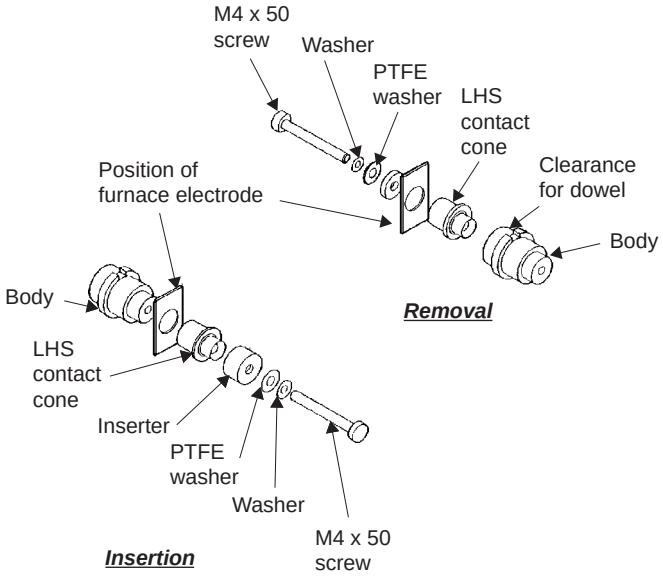
If a Cuvette has failed in use, it is likely that soot and debris will have been deposited on the Temperature Control Window. The Window should be cleaned before fitting a new Cuvette.

To clean the Temperature Feedback System

1. Remove the GFS97 from the Sample Compartment, and place it in a secure, accessible position in front of the spectrometer
2. Open the Furnace Head, and remove the Cuvette.
3. Pull the Centre Block gently to the right, to free it from the locating spigots.
4. Gently pull the Centre Block, with the attached hoses and connections, out from the body of the Furnace, to expose the Temperature Control Window in the base of the Centre Block, and the Lens in the body of the Furnace.
5. Clean the Lens with a cotton bud.
6. Unscrew the Window Holder from the rear of the Centre Block.
   - Take care that the Window does not fall out and get lost.
7. Inspect and clean the Window.
8. Replace the Window and Window Holder.
9. Reverse the procedure to refit the Centre Block.
   - When feeding the hoses and connection back into the Furnace body, ensure that none are trapped, kinked, or otherwise damaged.

Cuvette Contact Cones

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



GF95 Contact Cone Replacement

Note that:

- 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. Remove the GFS97 and position it securely in front of the spectrometer.
2. Remove the Sample Injection Port Sleeve, and both Furnace Head Window Holders.
3. Remove the Centre Block.
4. Remove the Rubber Seal from the right hand Electrode.
5. Referring to the figure, assemble the Cone Replacement Tool, and insert it into one of the Electrodes.

Graphite Furnace System Maintenance (continued)

- 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

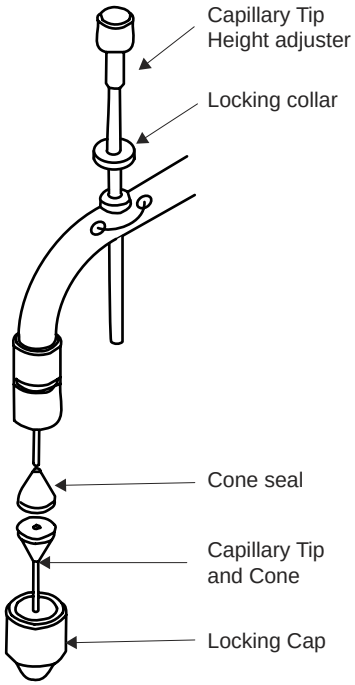
1. Referring to the figure, assemble the new Cone on the Cone Replacement Tool.
2. Insert the assembly into the Electrode.
3. Fit the screw, then gently tighten it until the Cone is fully home.
   - Take care not to over tighten the screw, which may damage the Cone.
4. Repeat the procedure for the other Electrode.
5. Refit the Rubber Seal in the right hand Electrode.
6. Refit the Centre Block.
7. Refit the Window Holders and Sample Injection Port sleeve.
8. Fit a new Cuvette, and replace the GFS97 in its normal position in the Spectrometer Sample Compartment.
9. Check the alignment of the GFS97 before using it again.

Leak Testing

Leaks may occur in the gas handling system or the water cooling system. Inspect your GFS97 regularly for leaks in both these areas.

Gas System

1. Set the inert gas supply pressure to 1.1 bar (15psi).
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.

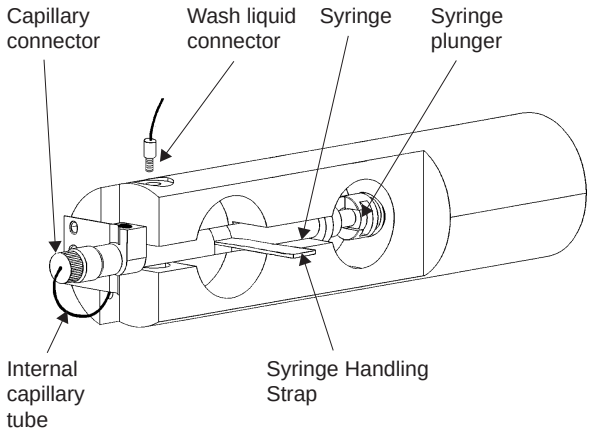


Capillary Tip detail

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 usually self-evident. Rectify them by tightening or remaking the connection.



FS95 Syringe Assembly

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.

Maintenance - Autosampler

Capillary Tip

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

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.

Graphite Furnace System Maintenance (continued)

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.

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 in 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 internal capillary tubing 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.

Spares and Consumables

Furnace Head Window (1 off)	9423 393 68561
GF95 Contact Cone set	9423 393 95011
GF95 Contact Cone Replacement Tool	9423 393 95101
GF95 User spares Kit	9423 450 20002
Capillary Tips (10)	9423 393 81261
Internal Capillary Tubing	9423 393 82261
Spare Carousel and Tray	9423 393 83261
FS95 User Spares Kit	9423 450 20004
Floor Standing 5L Waste Vessel	9423 390 05471

VAPOUR OPERATION

Vapour Safety

Introduction

- The gaseous products of the hydride reaction used in Vapour 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.

Reagents

- Sample solutions and reagents used in Vapour 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 Vapour Accessory.

Introduction

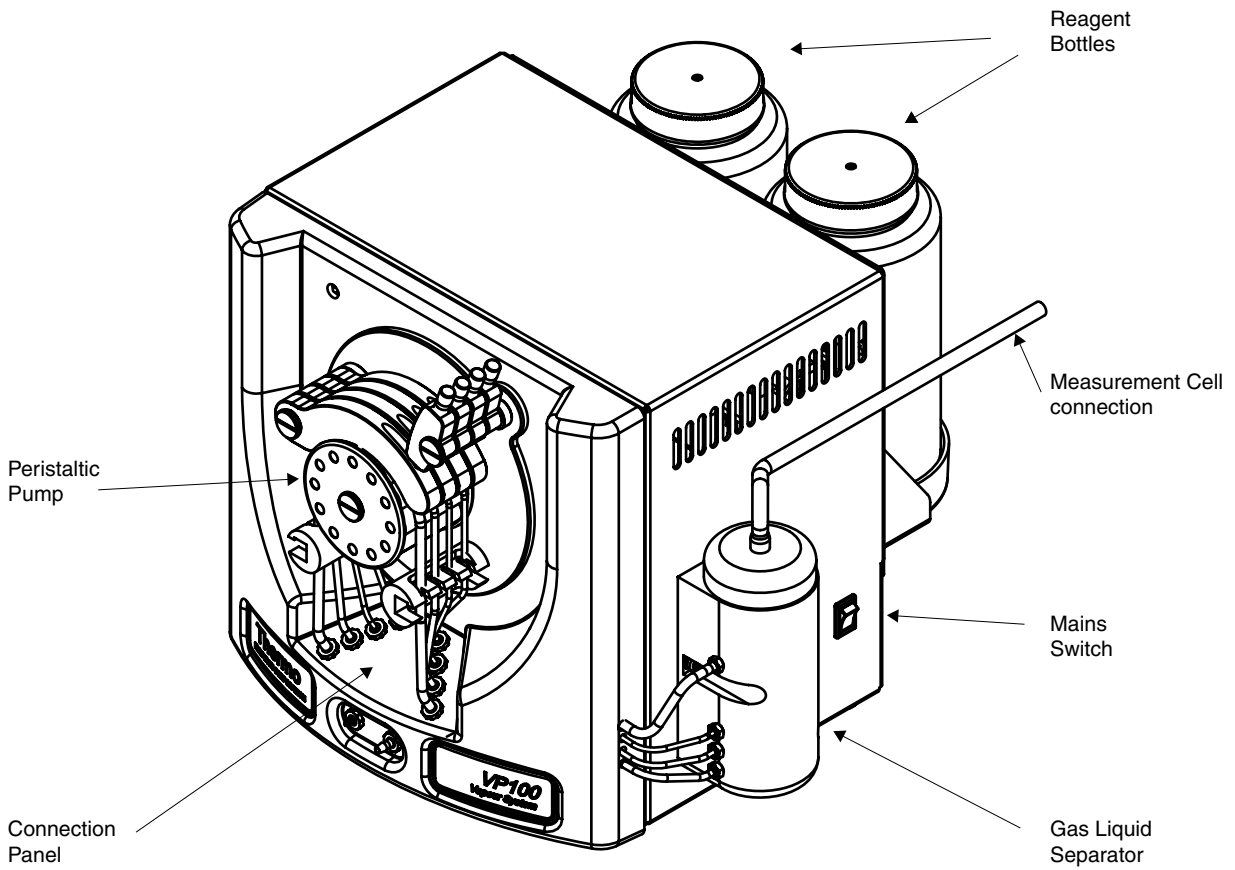
- The VP100 is an accessory for measuring the hydride forming elements and mercury with better analytical sensitivity than can be obtained from flame atomisation.
- The analyte elements in the sample solution are reduced to volatile hydrides using sodium borohydride, and are carried in a carrier gas 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 is not necessary to heat the Measurement Cell 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 1.3x relative to the standard 'T' cell.

Installation

System software

The VP100 is NOT compatible with the Local Control software. It can only be used with the SOLAAR Data Station software.

The VP100 requires at least version 10.1 of the SOLAAR Data Station software, and version 1.24 of the spectrometer firmware.



VP100 General View

Power

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

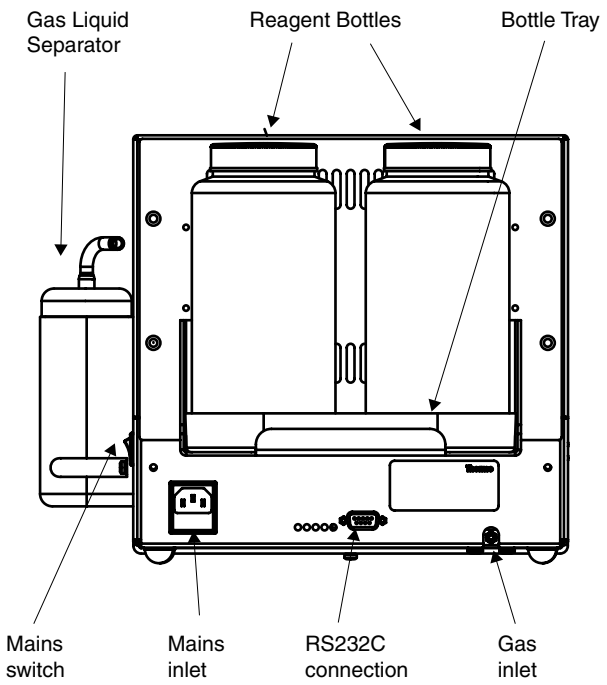
Control Signals

- 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 between 0.34 - 1 bar (5 - 14 psi), to the gas inlet at the rear of the unit.

The VP100 Continuous Flow Vapour Accessory (continued).



VP100 Rear View

Drain

- Connect a suitable length of the 3.2mm 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.

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.
- The VP100 can conveniently be supported on the Accessory Trolley (part number 9423 393 46131) if the bench is not deep enough.

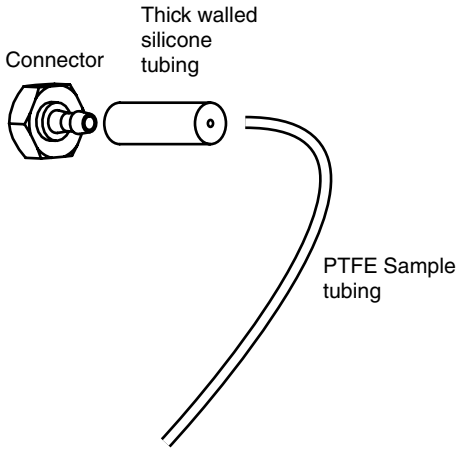
Sample Uptake

The Sample Uptake tubing must be attached to the GREEN Sample Uptake connector on the front panel of the ID100 using a short piece of the thick walled silcone tube supplied.

- **Manual sampling.** Take a suitable length of the PTFE Sample Uptake tubing supplied, and connect it to the ID100 Sample Uptake connector.
- **Autosampler.** Connect the tube from the autosampler sample probe to the ID100 Sample Uptake Connector.
  - If you are using a CETAC autosampler, we recommend that you use the larger 0.8mm 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 15-20mm long.
2. Grip the sample uptake tube firmly and push it into one end of the silicone tubing, until 10mm 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 ID100 front panel.



Sample Uptake tubing connection

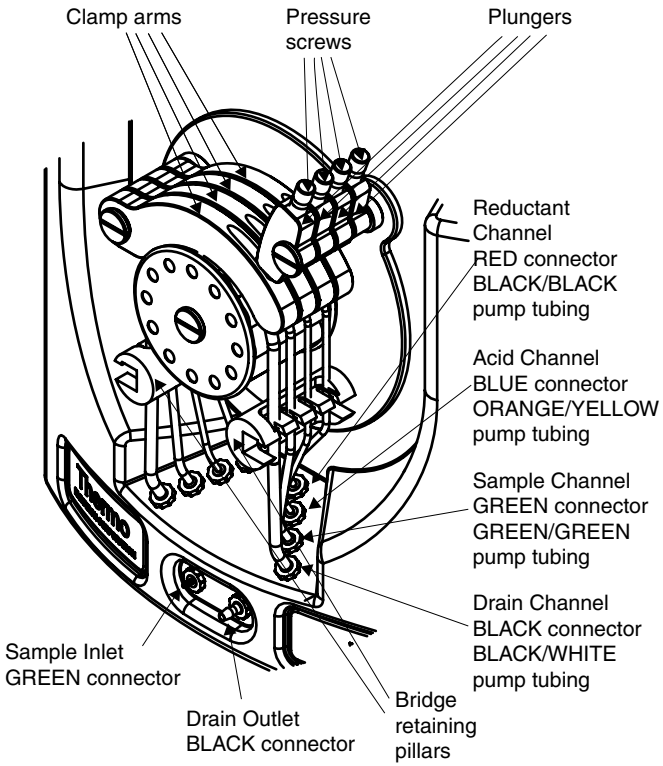
Pump Tubing

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 3.17mm and and OD of 4.85mm.
- The GREEN (sample) channel must be fitted with pump tubing with two GREEN bridges. This tubing has an ID of 1.85mm and an OD of 3.53mm.

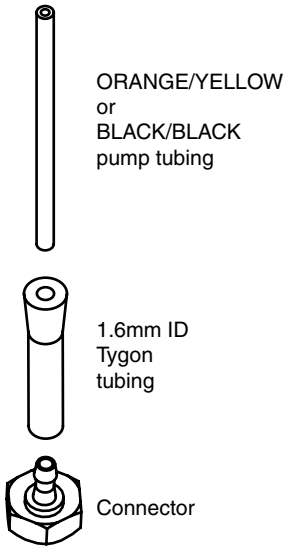
The VP100 Continuous Flow Vapour Accessory (continued).



VP100 Connection Panel and Peristaltic Pump

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

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 ID100 connection panel. You must fit short length of the 1.6mm ID Tygon tubing supplied over the ends of the pump tubing, then fit that to the ID100 connectors.



Pump tubing connection

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

1. Take a piece of 1.6mm 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 - 5mm 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.
3. Trim back the Tygon tubing to 15mm 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 ID100 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.
4. Ensure that the clamp arms are correctly positioned, and that the plunger arm pressure screws have been fully released.

The VP100 Continuous Flow Vapour Accessory (continued).

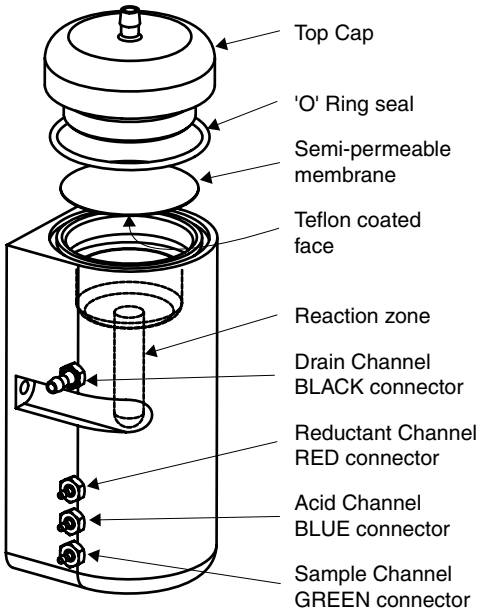
5. Use the system software to set up a Vapour Method using the VP100, and set an appropriate value for the Carrier Gas flow rate. Set the Pump Speed to 30RPM.
6. Use the commands on the Action menu in the system software to turn on the VP100.
7. Tighten the pressure screws in each plunger arm in turn 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, therefore, there will be bubbles present in the pumped fluid in the Drain channel.
   - The flow rates in the Acid and Reductant channels are quite 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 4mm glass beads, which minimise the dead volume of the zone and ensure proper mixing of the carrier gas and liquid reagents. The Gas Liquid Separator also contains a semi-permeable 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.

To prepare the Gas Liquid Separator

1. Refer to the figure, and unscrew the Top Cap of the Gas Liquid Separator.
2. Add a sufficient quantity of the 4mm glass beads supplied to fill the Reaction Zone. There should be no beads on the floor of the Expansion Volume.



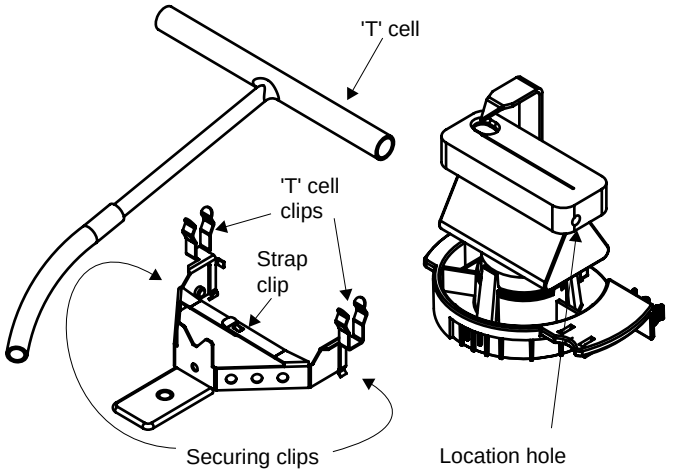
VP100 Gas Liquid Separator

   - Take care to prevent the glass beads from falling into the Drain, where they may cause blockage.
3. Take one of the 47mm 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.



'T' Cell Holder Assembly

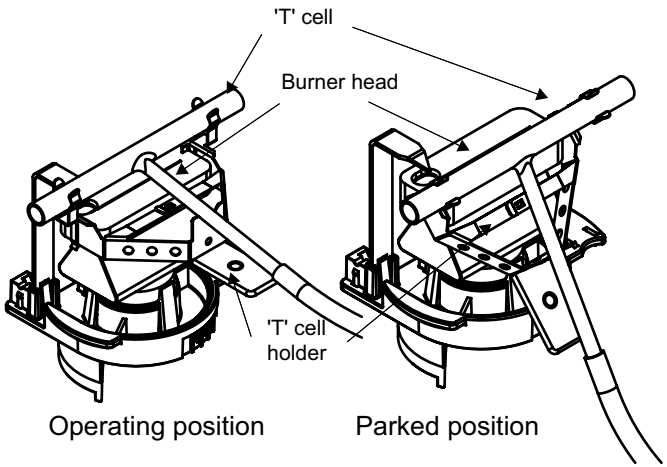
To fit the Cell Holder to the burner:

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.

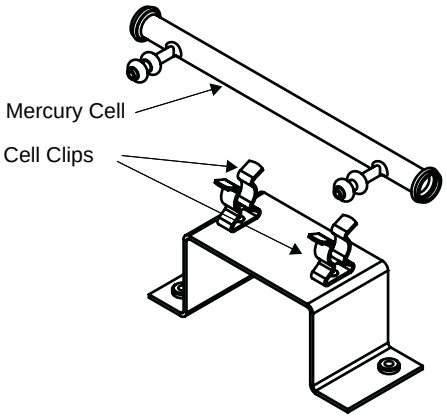
'T' Cell Alignment

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.

The VP100 Continuous Flow Vapour Accessory (continued).



'T' Cell and Holder



Mercury Absorption Cell and Cell Holder

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 in either 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 ID100.

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.

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 ID100.

To connect the Absorption cell:

1. Cut a suitable length of the 8mm 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.
   - It is good practice to loosely plug the other arm of the Mercury Cell with a small piece of cotton wool, to prevent the ingress of dust.

'T' Cell Conditioning

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

To condition a new 'T' cell

Either

- Run a high concentration standard (typically 1-10 mg/L) for at least 5 complete measurement cycles.

The VP100 Continuous Flow Vapour Accessory (continued).

Or

- Soak the 'T' cell in 50% v/v hydrofluoric acid for 15 minutes, then rinse it thoroughly in water and dry it before use.

WARNING: HYDROFLUORIC ACID IS EXTREMELY DANGEROUS TO HANDLE. IT MUST BE USED IN A FUME HOOD, AND PROTECTIVE CLOTHING (RUBBER GLOVES, RUBBER APRON AND FACE SHIELD) MUST BE WORN.

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(III) (sodium borohydride, $NaBH_4$), stabilised in 0.5% m/v sodium hydroxide (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 preferable to prepare it immediately before use.

Stannous Chloride Reductant reagent.

This reagent is suitable for mercury analyses only.

- 0.1 - 10% m/v of stannous chloride ($SnCl_2$) 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.
  - Some grades of stannous chloride form a milky suspension, not a clear solution, particularly at the higher concentrations. Providing that the suspension is stable, and shows no tendency for the solids to settle out, it can be used without affecting the analysis.

Parameter Optimisation

The VP100 has three parameters that control it's operation:

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

Default values are provided for these parameters, which you can alter in the System software, on the Vapour 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, and, except for mercury, offer no benefits. 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 - 200mL/min, and it is unlikely that flow rates higher than 300mL/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 30RPM, and good results can be obtained with pump speeds up to 45RPM.

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 gives a signal of between 0.1 and 0.4A.

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.

The VP100 Continuous Flow Vapour Accessory (continued).

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 default value of 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 Vapour Status Window.
6. Use the commands on the VP100 submenu on the Actions menu to start the VP100.
   - The Vapour 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 0.000A.
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 Vapour 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 Vapour 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. An 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} Se^{IV}, 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 therfore 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 Vapour Kit

1. Install and align all parts of the VP100 Vapour Kit.
2. Prepare your samples, standards and blank solutions.
3. Prepare the Reagents required and place them in the Reagent bottles.

The VP100 Continuous Flow Vapour Accessory (continued).

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 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 cannot check the amount of Reagents in the Reagent bottles, nor detect if the Reagents are actually 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 interrupt it, 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.

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

To shut down the VP100

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 over the Plungers to release the pressure on the pump tubes.
5. Empty and rinse the Reagent bottles. Wipe up an 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 off)	9423 390 60101
VP100 Pump Tubing set	9423 460 10011

VP100 Maintenance

Introduction

- Routine maintenance is mainly concerned with keeping the apparatus clean. If it 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 that 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 30RPM, and allow it to run until the water has filled the Gas Liquid Separator, and is being discharged from the Drain.
3. Take a 10mL measuring cylinder, and fill it to the 10mL 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:

Acid channel	0.7mL/min
Reductant channel	1.6mL/min
Sample channel	7.5ml/min
Drain channel	14mL/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. However, 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 Semi-Permeable Membrane.
2. Inspect the internal volume of the Gas Liquid Separator, and remove any deposits.
3. Confirm that the Reaction Zone is filled with glass beads.
4. Fit a new Semi-Permeable 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 it 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 Semi-Permeable Membrane	9423 460 10021
Spare Glass Beads for GLS	9423 460 10031

The EC90 Electrically Heated Atomiser

Introduction

- This is an accessory for use with the VP90 AND VP100 Continuous Flow Vapour Accessories.
- It replaces the Flame Heated Atomisation Cell with an Electrically Heated Atomisation Cell, so that Vapour measurements can be made without a flame.
- It is fitted in the Sample Compartment, using the 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.

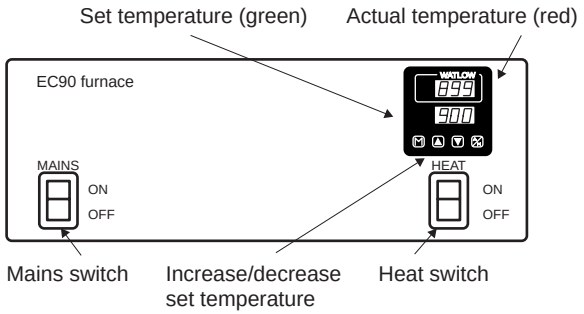
Installation

The EC90 Power Supply

1. Place the Power Supply on the right hand 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.

The EC90 Furnace Head

1. Fit the Furnace Head to the Universal Mount using the two securing screws on the base of the Furnace.
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 Sample Compartment of the Spectrometer.
4. Connect the Power Cable from the Furnace Head to the Head socket on the back of the Power Supply.



EC90 Front Panel

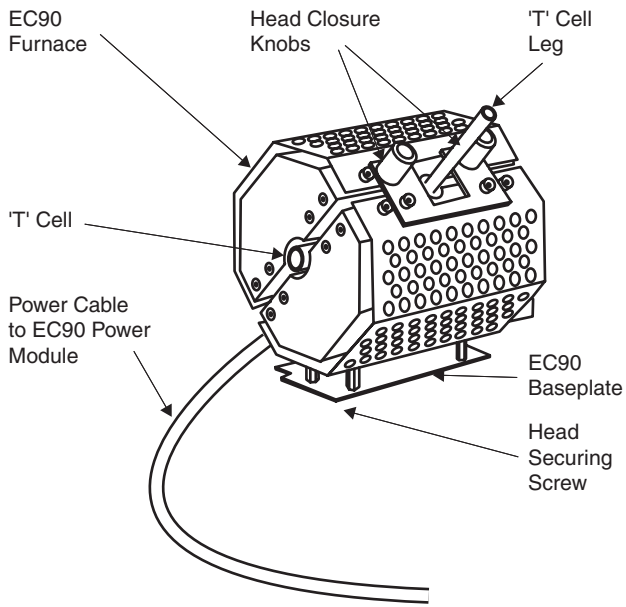
- Push the plug into the socket, then give it a quarter turn clockwise, until it clicks into position.
- To disconnect the plug from the socket, pull the metal locking piece towards the cable to release the lock, then rotate the plug anti-clockwise and pull it from the socket.

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 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 Bulkhead connector of the vapour accessory.

Operation

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

1. Set a suitable atomisation temperature
2. Turn on the EC90 Furnace and allow the temperature to stabilise.



EC90 Furnace Head

3. Set up the vapour accessory and perform the analysis.

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 EC90 Electrically Heated Atomiser (continued)

- The upper (red) display will indicate the approximate ambient temperature.

4. Ensure that the vapour system Carrier Gas is flowing through the Atomisation Cell.
3. Turn on the **Heat** power switch.
   - Power will be applied to the Furnace Head, and the red temperature display will increase.
4. 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.

The Cold Vapour Mercury Kit

Introduction

- This is a manual accessory that will allow you to perform simple, cost effective Cold Vapour Mercury analyses.
- Relatively large volumes of sample solution can be used, so that concentration sensitivities below 1µg/l can be obtained.
- The Mercury Absorption Cell is used, which is mounted on the Universal Accessory Mount in the Sample Compartment.

Installation

1. Refer to the HS90 section of this manual, and fit and align the Mercury Absorption Cell in the Sample Compartment.
2. Place the Pump and Conical Flask on the bench in front of the Sample Compartment.
3. Locate the Sinter Filter, and place a small plug of dry cotton wool in one side.
4. Using the minimum suitable lengths of the supplied Tygon tubing, connect:
   - the Pump Outlet to the Dreschel Head Inlet.
   - the Dreschel Head Outlet to the side of the Sinter Filter containing the cotton wool..
   - the other side of the Sinter Filter to the Mercury Absorption Cell Inlet.
   - the Mercury Absorption Cell Outlet to the Pump Inlet.
5. Connect the Pump to a suitable switched mains outlet.

Reagents

Stannous Chloride Reductant.

- Prepare a solution containing 10% m/v of stannous chloride ($SnCl_2$) in 10%v/v hydrochloric acid.

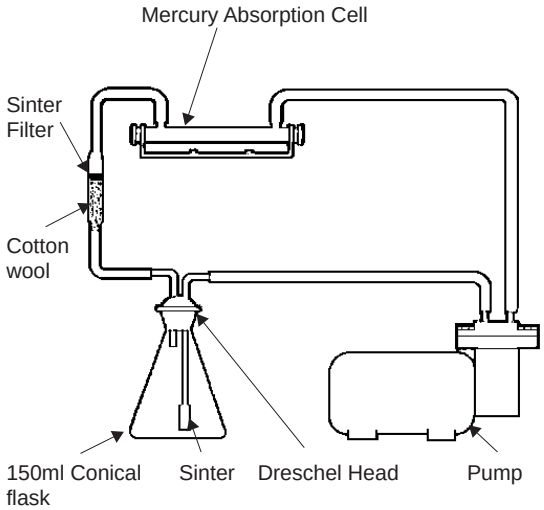
Hydroxylamine Hydrochloride Pre-reductant.

- Prepare a solution containing 10% m/v of hydroxylamine hydrochloride ($HO.NH_3Cl$) in water.

Note: Both stannous chloride and hydroxylamine hydrochloride are available as 'Low in mercury' grade reagents. We strongly recommend that you use this grade of chemicals if they are available.

Operation

1. Install a mercury hollow cathode lamp, and set up your analysis using the system software. Ensure that you select Vapour analysis with the Cold Vapour Mercury Kit.
2. Start the Pump.
3. Start the analysis from the system software in the normal way. Ensure that the live absorbance signal is displayed. When the prompt to measure the first solution appears:
   - Measure the required volume of sample into the conical flask, and add deionised water as necessary to bring the liquid level to approximately 10mm above the top of the sinter.
   - Add sufficient Pre-reductant Reagent to clear any oxidising residues from the sample solution.
   - Add 1 - 2ml of the Stannous Chloride Reagent, then immediately attach the flask to the Dreschel Head.
   - Observe the displayed absorbance signal, and when it is stable, acknowledge the software prompt to start the measurement.
4. When the measurement has finished, remove the flask, and rinse it with clean water ready for the next sample.



The Cold Vapour Mercury Kit

Maintenance

- Clean the flask and Dreschel Head thoroughly after use. Be especially careful to remove any residues of tin oxide, as they can block the sinter in the Dreschel Head.
- Replace the cotton wool plug in the Sinter Filter when it becomes damp, and use a new piece of cotton wool for each analysis.

Consumables and Spares

Mercury Absorption Cell	9423 168 05641
Sinter Filter	9423 393 68581
Dreschel Head and sinter	9423 393 68591
150ml Conical Flask	9423 393 68601