Aridus Desolvating Sample Introduction System Operator's Manual

Product Warranty Statement

SD Acquisition, Inc., DBA CETAC Technologies ("CETAC"), warrants any CETAC unit manufactured or supplied by CETAC for a period beginning on the date of shipment and ending on the sooner to occur of: (a) the date that is twelve (12) months from the date of installation, or (b) the date that is thirteen (13) months from the date of shipment. Units found in the reasonable judgement of CETAC to be defective in material or workmanship will be repaired or replaced by CETAC without charge for parts and labor. CETAC reserves the right to change or improve the design of any unit without assuming any obligation to modify any unit previously manufactured.

This warranty does not cover any unit that has been subject to misuse, neglect, negligence, or accident. The warranty does not apply to any damage to the unit that is the result of improper installation or maintenance, or to any unit that has been operated or maintained in any way contrary to the instructions specified in the CETAC instruction and operation manual. Operation of the CETAC unit inside a laboratory fume hood is contra-indicated and will void the warranty. Any attempt to repair or alter any CETAC unit by anyone other than by CETAC authorized personnel or agents will void this warranty. If any non-CETAC component is installed in the CETAC manufactured unit without the approval of CETAC, the warranty will be voided. In addition, this warranty does not extend to repairs made necessary by the use of parts, accessories or fluids which are either incompatible with the unit or adversely affect its operation, performance or durability. CETAC'S obligation under this warranty is strictly and exclusively limited to repair or replacement of defective CETAC parts, and no claim of breach of warranty shall be cause for cancellation or recission of the contract of sale of any unit.

The foregoing express warranty is in lieu of all other warranties, expressed or implied, including warranties of merchantability and fitness for a particular purpose. CETAC shall not be bound by any representations or statements on the part of its employees or agents whether oral or in writing and including any made in catalogues and other promotional material including technical details and specifications except where such representations and statements are expressly made part of this contract. CETAC assumes no responsibility for incidental, consequential or other damages, even if advised of such a possibility, including but not limited to loss or damage of property, loss of revenue, loss of use of the unit, loss of time, or inconvenience. CETAC's liability on any claim for loss or damage arising out of the sale, resale or use of any of its products shall in no event exceed the selling price of the unit.

Purchaser shall indemnify CETAC against any claim or liability which may be asserted as relates to the following: (i) the use to which any product supplied hereunder is put infringes the patent, copyright or other intellectual property rights of any third party; or (ii) any liability resulting from the failure by Purchaser to observe the terms of this Warranty.

Returned Product Procedures

Claims for shipment damage (evident or concealed) must be filed with the carrier by the buyer. CETAC must be notified within ninety (90) days of shipment of incorrect materials. No product may be returned, whether in warranty or out of warranty, without first obtaining approval from CETAC. No replacements will be provided nor repairs made for products returned without such approval. Any returned product must be accompanied by a return authorization number. The expense of returning the unit to CETAC for service will be paid by the buyer. The status of any product returned later than thirty (30) days after issuance of a return authorization number will be subject to review. Shipment of repaired products will generally be made forty eight (48) hours after the receipt.

Products may not be returned which are contaminated by radioactive materials, infectious agents, or other materials constituting health hazards to CETAC employees.

Returned Product Warranty Determination

After CETAC'S examination, warranty or out of warranty status will be determined. If a warranted defect exists, the product will be repaired at no charge and shipped prepaid back to the buyer. If the buyer desires an air freight return, the product will be shipped collect. Warranty repairs do not extend the original warranty period.

If an out of warranty defect exists, the buyer shall be notified of the repair cost. At such time the buyer must issue a valid purchase order to cover the cost of repair and freight, or authorize the products to be shipped back as is, at the buyer's expense. Failure to obtain a purchase order number approval within fifteen (15) days of notification will result in the products being returned as is, at the buyers expense.

CETAC Technologies
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E-mail custserv@cetac.com

REVISIONS

CETAC Technologies strives to provide the scientific community with an unparalleled combination of effective technology and continuing value. Modular upgrades for existing instruments will continue to be a prime consideration as designs progress.

CETAC Technologies reserves the right to revise this document and/or improve products described herein at any time without notice or obligation. Warranty registration entitles the named owner exclusively to manual change pages/new editions as they are published.

SAFETY

Instruments, accessories, components or other associated materials **may not** be returned to CETAC Technologies if contaminated with biohazard or radioactive materials, infectious agents, or any other materials and/or conditions that could constitute a health or injury hazard to CETAC employees. Call Customer Service and Support if there is any question or doubt relative to decontamination requirements. CAUTION and WARNING statements, as applied in this document, shall be interpreted consistent with the following context: CAUTION applies only to potential property damage conditions; WARNING applies to potential personal injury conditions, in combination with or exclusive of potential property damage.

All user-serviceable components are specifically identified in this document as such; the balance shall be assumed to require the expertise of a factory service technician/engineer for adjustment, repair, replacement, modification, etc. Others not so qualified and performing these actions shall do so at their own risk. Furthermore, never operate the instrument without first reading and understanding the *Aridus Desolvating Sample Introduction System Operator's Manual* and ensuring that it is operated safely and properly.

ORIGINAL PACKAGING

Retain original factory packaging for moves and factory return shipments. Shipping in anything other than the original fitted foam and container can result in incidental damage from which the purchaser will not be protected under warranty.

WARNING

Under all conditions the user must observe safe laboratory procedures during the operation of this product.

FEDERAL COMMUNICATIONS COMMISSION (FCC) NOTICE

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to Part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference in a commercial installation.

This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instructions, may cause harmful interference to radio communications. Operation of this equipment in a residential environment is likely to cause harmful interference, in which case the user will be required to correct the interference at his or her own expense.

MODIFICATIONS

The FCC requires the user to be notified that any changes or modifications made to this device that are not expressly approved by CETAC Technologies may void the user's authority to operate the equipment.

CABLES

Connections to this device must be made with shielded cables with metallic RFI/EMI connector hoods to maintain compliance with FCC Rules and Regulations.

CANADIAN NOTICE

This digital apparatus does not exceed the Class A limits for radio noise emissions from digital apparatus as set out in the interference-causing equipment standard entitled "Digital Apparatus." ICES-003 of the Department of Communications.

AVIS CANADIEN

Cet appareil numerique respecte les limites de bruits radioelectriques applicables aux appareils numeriques de Classe A prescrites dans la norme sur le materiel brouilleur: "Appareils Numeriques," NMB-003 edictee par le ministre des Communications.

POWER CORD SET REQUIREMENTS

The power cord set supplied with your instrument meets the requirements of the country where you purchased the instrument.

If you use the instrument in another country, you must use a power cord set that meets the requirements of that country.

WARNING

This equipment is designed for connection to a grounded (earthed) outlet. The grounding type plug is an important safety feature. To reduce the risk of electrical shock or damage to the instrument, do not disable this feature.

CAUTION

To reduce the risk of fire hazard and electrical shock, do not expose the unit to rain or humidity. To reduce the risk of electrical shock, do not open the cabinet. All maintenance is to be performed by an Authorized CETAC Service Provider.

Protection provided by the equipment may be impaired if the equipment is used in a manner not specified by the manufacturer.

CLEANING INSTRUCTIONS

To clean the exterior surfaces of the instrument, complete the following steps:

1 Shut down and unplug the instrument.

2 Wipe the instrument exterior surfaces only using a towel dampened with a lab-grade cleaning agent.

3 Repeat step 2, using a towel dampened with clear water.

4 Dry the instrument exterior using a dry towel.

WARNING

Do not allow any liquid to enter the instrument cabinet, or come into contact with any electrical components. The instrument must be thoroughly dry before you reconnect power, or turn the instrument on.

COOLING FAN OBSTRUCTION

The instrument cooling fan(s) shall remain unobstructed at all times. Do not operate the instrument if the cooling fan(s) are blocked or obstructed in any manner.

ENVIRONMENTAL

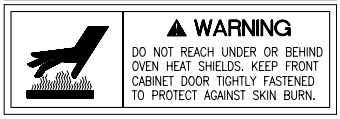
Operating Temperature:	10° to 30°C
Relative Humidity	0% to 95%

WARNING
FOR CONTINUED PROTECTION AGAINST RISK OF FIRE, REPLACE ONLY WITH FUSES OF THE SPECIFIED TYPE AND CURRENT RATING.

FOR CONTINUED PROTECTION AGAINST RISK OF FIRE, REPLACE ONLY WITH FUSES OF THE SPECIFIED TYPE AND CURRENT RATING.

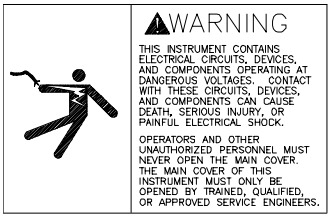
⚠ AVERTISSEMENT

POUR UNE PROTECTION CONTINUÉ CONTRE LES RISQUES D'INCENDIE, REMPLACER UNIQUEMENT PAR DES FUSIBLES DE MÊME TYPE ET AMPÈRAGE.



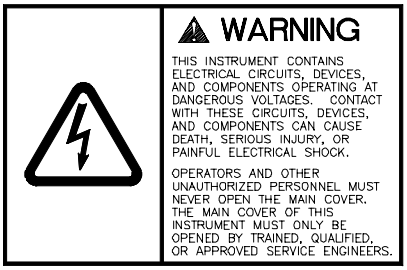
⚠ AVERTISSEMENT

NE PAS GLISSER LA MAIN SOUS OU DERIERE LES ECRANS THERMIQUES DU FOUR. GARDER LA PORTE D'ACCES AU DEVANT DU BOITIER BIEN FERMEE POUR ASSURER LA PROTECTION CONTRE LES BRULURES



⚠ AVERTISSEMENT

TOUT CONTACT AVEC LES HAUTES TENSIONS PEUT ENTRAINER LA MORT OU DES BLESSURES SÉVÈRES. CE PANNEAU NE DOIT ÊTRE ENLEVE QUE PAR UN RÉPARATEUR QUALIFIÉ.



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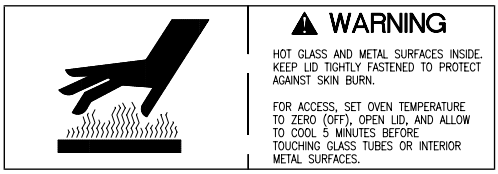
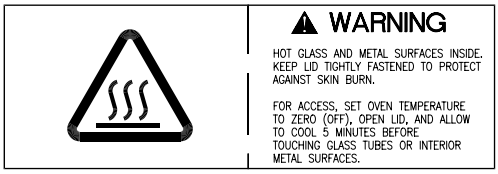
⚠ **WARNING**
HIGH LEAKAGE CURRENT - ENSURE PROPER GROUNDING

⚠ AVERTISSEMENT

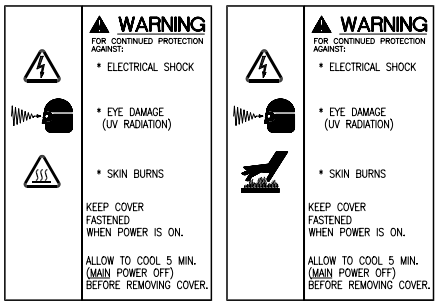
COURANT DE FUITE ÉLEVÉ — FORNIR UNE MISE À LA TERRE EFFICACE.





⚠ AVERTISSEMENT

SURFACES CHAUDES, LAISSER LE COUVERCLE HERMÉTIQUEMENT FERMÉ.
POUR ACCÉDER, METTRE LA TEMPÉRATURE DU FOUR À ZÉRO, OUVRIR LE COUVERCLE ET LAISSER REFROIDIR 5 MINUTES AVANT DE TOUCHER LA VERRERIE OU TOUTE SURFACE MÉTALLIQUE INTÉRIEURE.



⚠ AVERTISSEMENT

POUR LA PROTECTION PERMANENTE CONTRE UN CHOC ÉLECTRIQUE, UNE BRÛLURE DES YEUX (RADIATION UV) OU DE LA PEAU, LAISSER LE COUVERCLE HERMÉTIQUEMENT FERMÉ LORSQUE L'APPAREIL EST SOUS TENSION.
LAISSER REFROIDIR 5 MINUTES (APPAREIL ÉTEINT) AVANT D'ENLEVER LE COUVERCLE.

Table of Contents

Table of Contents

Preface

Preface

The *Aridus Operator's Manual* explains the procedures for installing, using, and maintaining the CETAC Aridus Desolvating Sample Introduction System. It also provides information about troubleshooting Aridus problems and describes the design of the system.

Who Should Read This Book

The primary audience for the *Aridus Operator's Manual* consists of analytical chemists and lab technicians. To use this manual effectively, you should have a strong knowledge of chemistry, a basic knowledge of electronic sampling equipment, at least a beginning level of computer experience, and working knowledge of an inductively coupled plasma mass spectrometer (ICP-MS).

How to Use This Book

The *Aridus Operator's Manual* contains seven chapters. You should read the chapters sequentially the first time. Thereafter, refer to the chapters separately as needed.

The *Aridus Operator's Manual* contains the following chapters:

Chapter 1, "Introduction," provides you with an overview of the Aridus's function and design.

Chapter 2, "Preparing for Installation," discusses space, power, and gas requirements that must be met before the Aridus is installed. It also provides instructions for unpacking the instrument and ICP-MS requirements.

Chapter 3, "Installing the Aridus," provides step-by-step procedures for installing the Aridus and connecting it to the analytical instrument.

Chapter 4, "Verifying Installation," explains initial operation of the Aridus, ICP-MS operation, and system optimization.

Chapter 5, "Using the Aridus," describes the tasks you perform during daily operation of the Aridus.

Chapter 6, "Maintaining the Aridus," explains daily, weekly, and periodic maintenance tasks.

Chapter 7, "Troubleshooting the Aridus," describes how to diagnose and correct minor Aridus and ICP-MS problems.

Conventions Used in This Book

This book uses certain conventions to distinguish different types of information easily. This section describes these conventions.

Instructions

All step-by-step instructions are numbered and in bold type, as in the following example.

1 Mount the sample probe assembly on the cover...

Many numbered instructions are followed by more detailed explanations.

Terminology

This book frequently uses the following terms:

Aridus	Desolvating Sample Introduction System.
ICP-MS	An inductively coupled plasma mass spectrometer.
Hz	Hertz.
ID	Inside diameter.
LED	Light-emitting diode.
PEEK	Polyetheretherketone.
PTFE	Polytetrafluoroethylene.
PSI	Pounds per square inch.
PVC	Polyvinyl chloride.
Trademarks	Teflon™ and Tefzel™ are registered trademarks of E.I. du Pont de Nemours.
VAC	Volts alternating current.
VDC	Volts direct current
T1H	Teflon™ microconcentric nebulizer.
Low-flow	Sample flow-rate of approximately 60 µL/min.
Sweep gas	Ar gas flowing counter-current to aerosol flow.

Notes

Notes contain a reminder about the effect of particular actions. Notes are indicated as follows:

> **Note:**
>
> This example shows how a note is displayed.

Cautions

Cautions indicate situations that require immediate attention to prevent harm to the Aridus desolvating sample introduction system. Cautions are indicated as follows:

CAUTION This example shows how a caution is displayed.

Warnings

Warnings indicate situations that could cause bodily harm. Warnings are indicated as follows:

WARNING **This example shows how a warning is displayed.**

Where to Go for More Information

In addition to the *Aridus Operator's Manual*, you can refer to the following resources:

- Software and hardware manuals for the ICP-MS instrument you are using.
- CETAC Technologies Customer Service and Support:

  1 (800) 369-2822

  1 (402) 733-2829

  1 (402) 733-1932 (Fax)

  custserv@cetac.com

1

Introduction

Introduction

The Aridus is a sample introduction system designed primarily for ICP mass spectrometry. The Aridus incorporates a Teflon™ T1H microconcentric nebulizer which yields low sample flow rates, dense aerosol production, and enhanced sensitivity. The T1H nebulizer is constructed entirely of inert materials and is suited for use with acidic and alkaline solutions. Solutions may contain nitric acid (HNO_3), hydrochloric acid (HCl), hydrofluoric acid (HF), or ammonium hydroxide (NH_4OH).

Sensitivity is improved with the Aridus by enhancing analyte transport efficiency and reducing solvent loading to the plasma. With conventional pneumatic nebulization, injected water vapor causes oxide and hydride polyatomic ion interferences in ICP-MS; organic solvent vapor loading causes carbide polyatomic ion interferences as well as plasma instability and carbon deposition on ICP-MS sampling cones. Using the Aridus for sample introduction greatly reduces these problems. It is strongly noted that sensitivity enhancement is dependent on the type of ICP-MS used with the area of application.

Functionally, liquid sample is nebulized into a PTFE spray chamber using a Teflon™ T1H microconcentric nebulizer operating at a sample uptake rate of approximately 60µL/min and an argon nebulizer gas flow rate of 1 L/min. The spray chamber is heated to help reduce the formation of solvent droplets inside the spray chamber. The spray chamber is also drained by a built-in peristaltic pump. The nebulizer gas flow then transports the aerosol from the spray chamber to a heated microporous PTFE tubular membrane. Solvent vapor passes through the membrane and is removed by an exterior flow of Ar gas (sweep gas). Analyte continues through the middle of the membrane tube and to the ICP-MS. An external view of the Aridus system is shown in Figure 1-1.

Aridus System Overview

Figure 1–1. Aridus Desolvating Sample Introduction System.

The following standard components/accessories are also included with each Aridus.

- **ICP-MS interface kit:** All parts to interface to the ICP-MS, including torch adapter, fittings and tubing.
- **Rinse kit:** Contains membrane rinse accessories and tubing kit that connects to the necessary ports.
- **Spare fuse kit:** Contains replacement fuses for the Aridus.
- **Gas-line tubing kit:** Contains all the necessary tubing to interface the argon sweep gas and nitrogen addition gas to the Aridus.
- **T1H Nebulizer**: Two (2) low-flow Teflon™ microconcentric nebulizers.
- **Nebulizer Flush Kit**: Flush kit for cleaning T1H nebulizer.

Note:

Contact CETAC Technologies, ICP Product/Application Department if you need additional accessories or added features to integrate the Aridus and your analytical system or if your laboratory has unique requirements. Research and development of new features and accessories for the Aridus, often inspired by customer requests, is a continuing activity of CETAC Technologies.

2

Preparing for Installation

Preparing for Installation

This chapter discusses the requirements for choosing a location for the Aridus Sample Introduction System. It also describes how to unpack the Aridus before installation.

Unpacking the Aridus

Inspect external packaging upon receipt for signs of physical damage from rough handling or abuse during shipment. Inspect all items during unpacking and notify the carrier immediately of any visible or concealed damage.

Remove packing checklist from the shipping container, and check off items against it. Leave accessories in the packing unit until you are ready to install them on the Aridus.

Aridus Packaging

The shipping container unpacks in two layers from the top down:

- Installation Kits for Aridus and ASX-100 Micro Auto-Sampler (if ordered)
- Aridus Desolvating Sample Introduction System and ASX-100 Micro Auto Sampler (if ordered)

> **Note:**
>
> Do not throw away the factory packaging. Keep it for possible future use. This is one of the warranty conditions.

CAUTION

If water vapor condensation has formed on or inside the Aridus, allow it to dry thoroughly before connecting it to an AC power source and operating it. Failure to do so may cause equipment damage.

Choosing a Location

Choosing a location for the Aridus involves evaluating the laboratory environment for the availability of space and power. For the Aridus to function optimally, the location you select must meet specific requirements associated with each of these items. The following sections discuss space and power requirements.

Space Requirements

Most analytical applications benefit from the shortest sample flow path. A one meter length of Teflon™ lined PVC tubing is provided to connect the Aridus to the ICP-MS torch. Place the Aridus sample out port less than one meter from the ICP-MS torch. The system without the ASX-100 Micro Auto-Sampler is 17 x 12 x 12 inches (43.2 x 30.5 x 30.5 cm). Adequate space should be allowed for proper ventilation.

Power Requirements

Place the Aridus within 1.2 meters of a power outlet. Two versions of the Aridus are provided to accommodate either 100-120 VAC ± 10%, 50/60 Hz, 6A, or 220-240 VAC ± 10%, 50/60 Hz, 3A.

ICP-MS Requirements

To achieve optimum performance with the Aridus, the ICP-MS system must meet factory specifications from your vendor and be in good operating condition. Check the ICP-MS performance using the standard sample introduction system before Aridus installation. If the sensitivity, oxide ratios and overall performance do not meet instrument specifications, consult the ICP-MS manufacturer for assistance. If these parameters are within the manufacturer's specifications, begin installation of the Aridus system. In order for the T1H nebulizer to properly self-aspirate, a minimum argon gas pressure of 420kPa (60psi) is required in the nebulizer gas line. Ensure that the Ar gas supply for the T1H nebulizer is from a known, good supply.

Gas Requirements

The argon gas supply for the Aridus sweep gas may be teed in from the main ICP-MS instrument argon gas supply. Nitrogen gas must be supplied from an external source at a pressure of at least 420kPa (60psi). Nitrogen with a minimum purity of 99.995% should be used.

3

Installing the Aridus

Installing the Aridus

To install the Aridus, you must complete the following tasks:

WARNING

Ensure that the AC power is off (0 showing at the top edge of the power switch) on the Aridus before proceeding with installation.

Establishing External Connections

The first step in the installation process involves connecting the Aridus to the power source and to the ICP-MS instrument. The following sections explain how to establish these connections.

Connecting the Aridus to the Power Source

Voltage-specified power cords are supplied with each Aridus.

WARNING

Use only these power cords or exact replacements.

To connect the Aridus to a power source, plug the cord into the power module located on the back panel of the Aridus. Then plug the cord into an appropriate AC outlet (110 or 220 VAC ±10%, 50/60 Hz depending on the model).

Connecting the Aridus to the ICP-MS

Depending on the ICP-MS manufacturer and model, a torch adapter is supplied for interfacing the Aridus to the ICP-MS. Adapters are available for all ICP-MS instruments.

1 **A length of ¼" O.D. Teflon™-lined PVC tubing is connected to the torch adapter. Place the other open end of the this tubing onto the SAMPLE OUT port located on the back of the Aridus.**

CAUTION:

Make certain that the Teflon™ lining in the sample out tubing is not blocking the aerosol path to the ICP-MS torch. This can result in instability and lack of analyte signal from the ICP-MS instrument.

Attach the other end, containing the torch adapter, to the ICP-MS torch/injector (Figure 3-1).

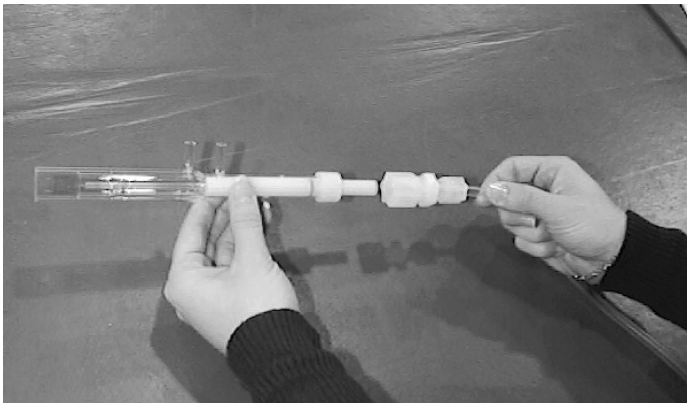


Figure 3-1. Connecting the 3/8" - ¼" torch adapter to the optional sapphire injector on the ICP-MS torch.

2 The rinse port is shipped with a fingertight Teflon™ plug installed. The rinse port should remain plugged except during cleaning/rinsing of the membrane.

3 Place the ½" adapter end of the corrugated Teflon™ tubing into the custom fitted waste reservoir on the back of the Aridus, and tighten the retaining nut. Place the other end of this tubing in an exterior exhaust vent. The corrugated tubing carries solvent vapors to waste. Exhaust should be appropriately vented at atmospheric pressure.

4 A minimum argon and nitrogen supply of 420kPa (60 psig) is required to operate the Aridus. The argon may be teed in from

the instrument argon supply using the ¼" union tee. Connect the ¼" O.D. tubing, labeled "ARGON" and "NITROGEN", to the respective ports on the Aridus. The valves are supplied to turn off these gas supplies when the Aridus is not in use (see Figure 3-2).

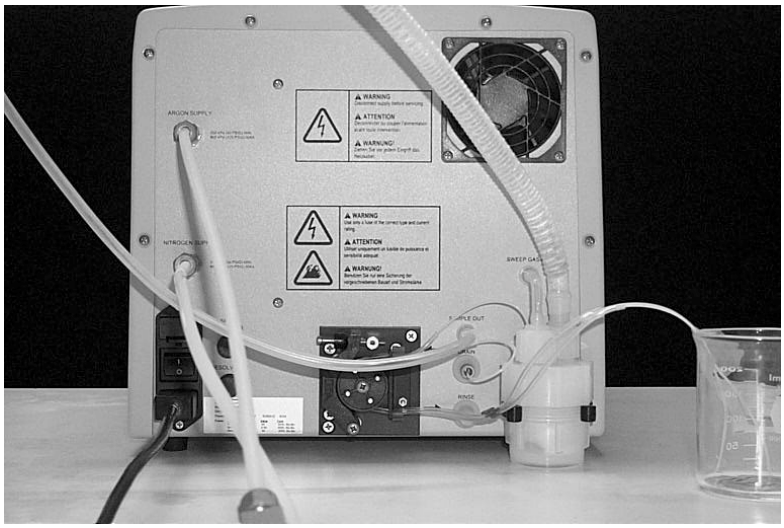


Figure 3-2. Attachment of argon and nitrogen supply lines.

Connecting the Aridus Drain Tubing

There are two drain lines to install on the back of the Aridus unit. To set up these drain lines:

1 **Obtain the peri-pump tubing kit from the Aridus completion kit.**

2 **One bag inside the peri-pump tubing kit contains pre-cut tubing attached to gray-gray color-coded peristaltic tubing.**

Remove this tubing from the bag. Note that there are two separate tubing assemblies.

3 **One of the tubing assemblies contains a tan-colored PEEK nut. Install the PEEK nut into the threaded port labeled "DRAIN" on the back of the Aridus unit. Tighten the PEEK nut so it is finger-tight.**

4 **Install the gray-gray color-coded peristaltic tubing (already attached to the PEEK nut via a length of Teflon™ tubing) to the peristaltic pump mounted in the back of the Aridus unit. Note that liquid from the drain port should flow to the top of the peristaltic pump and out the bottom to waste. This is shown by the arrows next to the pump.**

5 **The second drain line does not contain a PEEK nut. Insert the Teflon™ line from one end of this line into the narrow port on top of the sweep gas out reservoir. Carefully ensure that the line reaches the bottom of the reservoir.**

6 **Again, attach the gray-gray color-coded peristaltic pump tubing to the peristaltic pump as described previously in Step Number 4.**

7 **The outlet of both lines may be placed in a suitable waste receptacle (such as a beaker) as shown in Figure 3-2.**

8 **Close the shoe of the peristaltic pump and adjust the tension with the spring-loaded screw.**

Connecting the ASX-100 Auto Sampler to the Power Source

To connect the auto sampler to a power source:

1 **Plug the AC power cord receptacle into the AC/DC power module.**

2 Plug the AC/DC power module's 24 VDC coaxial plug into the auto sampler's 24 VDC-IN jack.

3 Plug the AC power cord into a 100-240 VAC ± 10%, 50/60Hz utility power outlet.

Connecting the ASX-100 Auto Sampler to the Host Computer

To operate the ASX-100 Auto Sampler, first establish a communications interface between the autosampler and host computer. It is through this interface that the host computer directs the operation of the ASX-100. The serial interface kit provided with the ASX-100 includes an interface cable equipped with two modular port adapters. Use the interface kit to establish a serial communications interface with the host computer. Complete the following steps:

1 Plug one end of the cable into the host computer's serial (COM) port selected for auto sampler communications. Make sure the COM port you select matches the port selected in the host computer's software.

2 Finger tighten both screws.

3 Connect the other end of the cable to the ASX-100 auto sampler, COM 1 port.

4 Finger tighten both screws.

CAUTION

Ensure that you are connecting the adapter to the COM 1 port. Connecting the adapter to the COM 2 port on the auto sampler will cause the auto sampler (and possibly the host computer) to malfunction. The auto sampler's COM 2 port is used only for communications to devices other than the host computer and cannot be user reconfigured.

5 Insert the T1H nebulizer into the spray chamber (Figure 3-3).

Figure 3-3. Inserting T1H nebulizer into the spray chamber.

6 Attach the sample probe assembly to the ASX-100 cover. (Refer to the ASX-100 Operator's Manual for further instructions.)

7 Insert the stainless steel sample probe through the Z-axis cap slider block and sample probe assembly on the ASX-100. Retain the sample probe with the PEEK thumbscrew and finger-tighten. (Refer to the ASX-100 Operator's Manual for further instructions.)

8 Finally, connect the nebulizer gas from the ICP-MS instrument to the T1H nebulizer using the nebulizer gas line kit supplied in the Aridus completion kit. The gas line attaches to the TIH nebulizer as shown in Figure 3-4.

Figure 3-4. Connecting the nebulizer gas line.

Setting the Sample Probe Depth

1 **Turn the slider rotor fully clockwise to move the probe to the maximum up position. (The set screw on the rotor should be in the six o'clock position.)**

2 **With the probe in the up position, set the probe depth such that the tip of the probe has clearance above the largest standard vial, but does not come above the plane of the autosampler lid.**

3 **This position may be finely adjusted to ensure maximum sampling from the sample vials.**

4 **Finger-tighten the PEEK screw to secure the probe to the slider.**

4

Verifying Installation

Verifying Installation

Once installation of the Aridus is complete, it is important to verify that you have installed the system correctly. Attempting to use the Aridus before ensuring that it is installed correctly may result in damage to the system.

Verifying installation of the Aridus consists of three parts:

- Initial operation procedure
- ICP-MS operation under normal conditions
- System Optimization

This chapter explains initial operation of the Aridus, ICP-MS operation`, and the optimization of the Aridus.

Initial Operating Procedure

1 Plug the supplied power cord into the Aridus and then into the AC supply outlet.

2 Turn on the power switch and allow the heaters to preheat. After approximately 5-10 minutes, all stages should be operating at a steady state as indicated by temperature readings of 160°C ± 2° on the heater for the desolvation system and 70°C ± 2° on the heater for the spray chamber.

Standard Conditions				
Sweep Gas L/min	**Nitrogen mL/min**	**Spray Chamber °C**	**Desolvator °C**	**Nebulizer Gas Flow L/min**
2 - 4	0 – 20	70°	160°	0.7 - 1.0

3 Open the valves on the ARGON and NITROGEN gas lines. Turn the gas flow control knobs on the Aridus clockwise and allow the system to purge. The Argon flow should be set to approximately 3.0 L/min and the nitrogen flow should be set to approximately 20 mL/min. Note that a nitrogen flow of 20mL/min will read as "020" on the flow meter.

> **Note:**
>
> All temperature controllers are factory programmed and preset. Do not exceed controller settings of 160°C for the desolvation system and 70°C for the spray chamber. If a different temperature setting is desired, the user must contact CETAC Customer Service. Unauthorized changes in the temperature settings will immediately terminate the warranty.

4 Adjust SWEEP GAS to 2.5L/min and NITROGEN to zero (fully counterclockwise).

5 Nebulize 2-propanol (isopropyl alcohol, also abbreviated IPA) for 15 - 20 minutes to condition the membrane. Use a nebulizer gas flow of 0.7 to 1.0 L/min. Verify that the nebulizer is self-aspirating by watching the liquid flow through the sample line toward the nebulizer body.

6 Remove the sample probe from the IPA and place it in deionized water (Type I water, >18 MΩ/cm). Nebulize deionized water for at least 10 minutes.

ICP-MS Operation

1 Set the SWEEP GAS and NITROGEN gas flows to zero by adjusting the gas flow control knobs located on the front of the Aridus. The SWEEP GAS flow may read a non-zero number from 0.5 to 1.0, but no gas will be flowing.

> **Note:**
>
> It may be possible to ignite the plasma while the **SWEEP GAS** and **NITROGEN GAS** are flowing. However, it is recommended that the gas flows be turned down during plasma ignition.

2 Ignite the plasma as instructed in the ICP-MS operating manual.

3 After the plasma has stabilized, increase the SWEEP GAS to approximately 2.5 L/min. NITROGEN flow should remain at the zero setting.

4 Prepare and aspirate an appropriate tuning solution for the ICP-MS instrument.

System Optimization

It is necessary to optimize the ICP-MS instrument after installation of the Aridus. Optimization procedures may include adjustment of gas flows, plasma viewing or sampling positions, ion lens settings, and other ICP-MS parameters. Typically, the signal-to-noise or signal to-background ratio is the primary criterion for optimization. For detailed instructions on system optimization for aqueous or organic samples, perform the ICP-MS/Aridus Optimization Procedures. After the system has been optimized, the Aridus is ready for routine operation.

Aridus Optimization Procedure

1 After the Aridus and ICP-MS have reached a stable operating temperature, set the nebulizer flow rate to 0.9 L/min and aspirate an appropriate tuning solution which includes the element cerium (Ce).

2 Monitor elemental signal intensity of the selected set of tuning elements in time resolved mode and adjust the argon SWEEP

GAS flow to produce desired signal intensity. The SWEEP GAS flow is one of the most important parameters in determining sensitivity and stability. Optimize the SWEEP GAS flow to maximize signal and minimize element oxide/element ratio. Note that the element Ce is often used for this purpose. The normal operating range for the SWEEP GAS is 2 - 4 L/min.

3 **Gradually increase the NITROGEN flow to further increase the signal. Based upon the type of ICP-MS instrument, little or no NITROGEN may need to be added. The normal operating range for the NITROGEN flow is 0 - 20 mL/min.**

> **Note:**
>
> The addition of NITROGEN will cause an increase in nitrogen-containing polyatomic interferences. Signal enhancement upon NITROGEN addition should be weighed against these interferences.

4 **Other parameters such as ICP sampling position, other gas flows, ICP power, and ion lens settings may need to be adjusted to achieve best performance. When properly optimized, CeO/Ce should be ≤ 0.05%.**

> **Note:**
>
> Organic solvents (e.g., 2-propanol, toluene, hexane) may be run directly through the Aridus to the ICP-MS. To analyze organic solvents, a low flow of oxygen (~10mL/min) is added into the sample line leading from the membrane desolvator to the ICP torch. This small amount of oxygen helps prevent carbon buildup on the ICP-MS sampling cones. **The CETAC BGX-100 Blend Gas Accessory can be used to simplify the gas addition. Please contact CETAC Technologies at 800-369-2822 or 402-733-2829 for further information and assistance with regard to gas addition.**

5

Using the Aridus Desolvating Sample Introduction System

Using the Aridus

It is important to establish sound laboratory practices, analytical environment and ICP-MS performance before using the Aridus. The best performance can then be expected when these conditions are met.

Establishing Optimal Conditions

Malfunction or damage can occur if specific operating conditions are not met. Meeting these conditions requires establishing the proper laboratory environment and replacement of ICP-MS and Aridus components that wear out during normal usage. The following sections explain how to meet these conditions.

> **Note:**
>
> Damage or malfunction that results from unsatisfactory operating conditions may constitute misuse and abuse and can be excluded from warranty coverage.

Establishing the Laboratory Environment

To establish satisfactory operating conditions in your laboratory environment, follow these guidelines:

- **Operate the Aridus in a conventional laboratory environment where the temperature is 50-86°F(10-30°C), the humidity is 20-70% non-condensing; and the unit is not exposed to excessive flammable or corrosive materials.**
- **Avoid rough handling of the Aridus. If possible, do not expose the system to vibration or shock.**

- **Protect the Aridus from long-term exposure to condensation, corrosive materials, solvent vapor, continual standing liquids, or large spills. Exposures of this type can damage the electronics.**

- **Observe the same general electrostatic discharge precautions as with any other integrated circuit electronic device. Low humidity environments, especially when combined with static-generating materials require maximum care.**

WARNING

Discharge static buildup and ground to the Aridus cabinet before performing any maintenance. Do not touch or short-circuit bare contacts.

Avoid using the Aridus if strong electromagnetic interference or radio frequency interference is present. In environments with very low humidity, high static charges may effect the stability of the analyte signal, causing a transient drop in intensity within the proximity of the charged object.

Replacing Aridus Components

The following components for the Aridus system need to be replaced periodically during normal usage. Failure to replace these supplies will result in deterioration of analytical performance.

- **Sample Transfer Line: Teflon™-lined PVC Tubing**

The sample transfer line should be replaced when it becomes grossly discolored or when it contains residues/condensation. See Chapter 6, "Maintaining the Aridus System" for further details.

- **Peristaltic Pump Tubing: 3-stop gray/gray 1.3mm i.d.**

The peristaltic pump tubing that drains the PTFE spray chamber and the sweep gas out reservoir should be replaced whenever the tubing becomes flat or misshapen due to pump shoe pressure. See Chapter 6, "Maintaining the Aridus System" for further details.

Startup Procedure

1 **If the Aridus has been turned off for an extended period of time, turn on the AC power switch and allow the HEATER temperatures to reach operating values and stabilize (approximately 5-10 minutes), purge the system with argon (3 L/min) and nitrogen (20 mL/min) for 5 minutes.**

2 **Decrease the sweep gas flow (0.5L/min) and nitrogen flow (0 mL/min).**

3 **Ignite the plasma according to standard operating procedures for the ICP-MS instrument. Re-adjust Aridus operating parameters to the optimized settings. If the system has not been optimized, see the previous section on Optimization.**

4 **The Aridus system is now ready to use.**

Shutdown Procedure

1 **<u>Aqueous Analysis:</u> Aspirate dilute nitric acid (1-3%) for approximately 5-10 minutes to provide a thorough spray chamber rinse. Follow the acid-rinse by aspirating deionized water for approximately 3-5 minutes.**

2 Remove the sample probe from the deionized water and aspirate the solution remaining in the sample line. It is important to dry the sample uptake line prior to shutting down the system.

3 Shut down the ICP-MS using the procedures recommended by the manufacturer.

4 Close the valves on the argon and nitrogen gas lines.

5 Turn off the power switches on the Aridus and ASX-100 micro auto sampler (when applicable).

Temperature Controller Operation

The temperature controllers for the spray chamber and the membrane desolvation system display the actual operating temperatures of the two modules. The setpoint for each temperature controller can be viewed by simply pressing the button labeled SET on the respective temperature controller. When the button is released, the actual temperature is again displayed. The setpoint temperature can be changed by following the steps below:

1 Press and hold the SET button and press the up or down arrow until the desired setpoint is reached.

2 Release the SET button and the actual temperature will be displayed.

Note:

The temperature controllers are set to allow adjustment over a certain range. You must contact CETAC Customer Support if you desire an operating temperature outside the specified range. Unauthorized changes in the temperature settings will immediately terminate the warranty.

Switching from Organic Samples to Aqueous Samples and Aqueous Samples to Organic Samples

When switching from analyzing organic solvents to aqueous samples, nebulize 2-propanol (isopropyl alcohol, IPA) for 10 minutes before running the aqueous samples. This will condition the spray chamber and membrane desolvation system.

When returning to organic samples, use 2-propanol to re-condition the Aridus.

WARNING

Carefully rinse the Aridus with copious amounts of deionized water when switching between strong acids (e.g. nitric acid) and organic solvents. If heated residue from nitric acid contacts organic solvents, a charring reaction can occur in the desolvation system. Failure to carefully rinse the membrane system between organics and strong acid (or bases) samples can cause damage to the system and may void warranty conditions. (Refer to Chapter 7, "Rinse Procedure for the Membrane Desolvation System").

6

Maintaining the Aridus

Aridus Maintenance

Routine maintenance of the Aridus consists of daily, weekly and monthly procedures for specific system components and expendable supplies used in the system. Maintenance includes checking the Aridus system components for leaks or other problems/damage. Some applications may require frequent maintenance procedures to be followed, e.g. flushing the membrane desolvation system after prolonged exposure to high dissolved solids.

WARNING

The Aridus must be turned off and the AC power cords unplugged before performing any maintenance on the system.

Maintaining the Nebulizer and Spray Chamber

The T1H Nebulizer may be removed from the unit while the plasma is on. If droplets collect on the nebulizer tip, remove the sample uptake tubing from the solution and allow the nebulizer to aspirate air 1-2 minutes. Dry the tip with a lint-free cloth.

If the aspiration rate is extremely slow or if the T1H nebulizer does not self-aspirate, the capillary may be plugged. To remedy this condition backflush the nebulizer line by placing the black cap that came with the nebulizer over the T1H nebulizer tip while the nebulizer gas is on. This will redirect the gas pressure through the sample line and push out any obstruction. A series of bubbles, such as when the sample begins to run dry, may also cause slow or stopped aspiration. If this procedure is not effective, then use the T1H flush kit. (See "Using the T1H Flush Kit".)

The PTFE spray chamber may be removed from the Aridus by grasping the endcap and pulling directly out. The chamber may be disassembled for cleaning if samples containing high dissolved solids were introduced.

Using the T1H Flush Kit

The T1H flush kit consists of a syringe and appropriate tubing for the T1H nebulizer. To use the flush kit, fill the syringe with rinse solution (1 – 3% HNO_3) Insert the sample line from the T1H into the tubing provided with the flush kit. Connect the tubing to the syringe and apply steady pressure to force the rinse solution through the sample line capillary.

Main Fuse Replacement

The main fuses are located in the AC power module fuse drawer located at the right rear of the Aridus. To replace blown fuses:

1 **Turn the AC power switch to off (0) and disconnect the AC power cord.**

2 **Use a small flat blade screwdriver to unlatch the fuse holder.**

3 **Replace the defective fuse with a GMC 5A, 250V slo-blo if operating on 100/115 VAC or a GMC 2.5A, 250V slo-blo if operating on 220/230 VAC.**

WARNING

Use of a different fuse other than those specified can damage the electrical components of the Aridus, constitute a fire hazard, or result in personal injury.

4 **Insert the fuse drawer, replace the power cord, and turn the AC power switch on. If the new fuse blows, do not attempt to operate the unit. Contact your authorized service representative or CETAC Technologies for assistance.**

Rinse Procedure for the Membrane Desolvator

The membrane may need to be thoroughly rinsed after the analysis of organic samples containing high concentrations of nonvolatile residues or after analysis of aqueous samples containing high concentrations of dissolved solids. The length of time between rinses in a routine operation depends on the types of samples analyzed. Analysis of samples containing less than 5% of dissolved solids is recommended. Best performance will be achieved with 0.1% to 0.5% dissolved solids.

When the solvent removal efficiency decreases, the oxide levels increase or if the signal becomes unstable, the membrane should be rinsed. The following procedure describes how to rinse the membrane desolvator. Please refer to Figure 6-1 for the rinse tubing setup.

Note:

If aqueous samples were analyzed, 1% HNO_3 may be used to rinse the membrane. If organic solvents such as toluene or hexane were analyzed, rinse the membrane with 2-propanol. If non-volatile organic samples (e.g., oils diluted in an organic solvent) were analyzed, rinse the membrane with toluene or hexane. After rinsing with such a solvent, perform a second rinse with 2-propanol.

WARNING

Please observe any necessary safety precautions (safety glasses, gloves, etc.) when handling HNO_3 solutions and organic solvents.

Figure 6-1. Membrane system rinse diagram.

1 Turn the AC power switch to off (0) and disconnect the AC power cords. Allow the unit to cool to room temperature.

WARNING

Power should be turned off at this time. If left on, the following procedure could cause permanent damage to the internal gas flow controllers.

2 Disconnect the SWEEP GAS OUT tubing from the barb on the back of the Aridus unit by pulling gently.

3 Disconnect the SWEEP GAS IN line and the NITROGEN GAS IN line from the back of the Aridus unit.

4 Disconnect the SAMPLE OUT Teflon™-lined PVC tubing in the same manner as above.

5 Disconnect the nebulizer gas line to the T1H nebulizer, and remove the T1H by pulling it out of the spray chamber.

6 Using the adapters and tubing provided in the Aridus rinse kit, connect the two ends from the "Y" of the rinse tubing to the SWEEP GAS OUT port and the T1H port on the spray chamber respectively. Connect the remaining end to the provided rinse bottle containing the appropriate rinse solvent (toluene or hexane for organic residues, 1-5% HNO_3 for aqueous residues). Heated (70°C) 1-3% HNO3 may enhance removal of contaminants. The HNO_3 rinse solution may be preheated and then added to the rinse bottle.

7 Connect the two ends from the "Y" of the other length of rinse tubing to the RINSE and SAMPLE OUT ports. Direct the remaining end into a suitable waste container.

8 Hold the rinse bottle above the unit and begin a siphon action by squeezing the bottle. Loosen the cap on the bottle and allow the solvent to siphon into the unit until the solvent emerges from the T1H port on the spray chamber. The flush volume of the Aridus system is approximately 340 mL.

9 Allow the solvent to soak the membrane for a minimum of ten minutes. The solvent can be allowed to soak for longer time periods without adverse affects.

10 To drain, lower the rinse bottle and allow the solvent to siphon back into the bottle until most of the solvent has been recovered.

11 If the solvent is significantly discolored, repeat the rinse procedure until it drains clear. As much of the solvent flush should be recovered prior to proceeding to step 11.

12 Locate the "Y" connector that leads to the SWEEP GAS OUT port, rinse bottle, and spray chamber adapter. Remove the line from the SWEEP GAS OUT port at the "Y", leaving the other end attached to the SWEEP GAS OUT port. Place the open end in a suitable waste container.

13 Reconnect the power cord and SWEEP GAS IN line to the Aridus. Turn the power on and set the SWEEP GAS flow to 3.0L/min. Let the SWEEP GAS run through the unit for at least 30 minutes to remove most traces of the rinse solvent.

14 Remove the rinse tubing and adapters. Reconnect the SWEEP GAS OUT, rinse port plug, and SAMPLE OUT LINE. Finally, replace the T1H nebulizer and the nebulizer gas line.

15 Longer drying times may ensure removal of virtually all traces of the rinse solvent. The Aridus may be turned on, SWEEP GAS flow set to 3L/min, and nebulizer gas from the ICP-MS instrument sent through the TIH nebulizer/spray chamber. Drying may be done overnight (8 to 12 hours). In addition, 2-propanol (isopropyl alcohol, IPA) may be aspirated for at least 1 hour to further enhance drying.

7

Troubleshooting the Aridus

Troubleshooting the Aridus

The Aridus is both easy to operate and reliable. However, problems with the system may occur. If good performance is not obtained, try to isolate the problem to determine if it originates in the ICP-MS instrument, sample preparation procedure, or the Aridus.

This chapter explains how to troubleshoot Aridus problems. If you cannot solve a problem using the steps given in this chapter, contact CETAC Technologies Customer Service and Support.

Phone: (800) 369-2822

(402) 733-2829

Fax: (402) 733-1932

E-Mail: custserv@cetac.com

Temperature Controller Problems

1 Temperature controllers and gas flow meters do not illuminate.

- The power cord may not be plugged into the AC wall outlet. Plug in power cord.
- Main fuses may be blown. Replace main fuses. See page 6-3 "Main Fuse Replacement".

2 Display of temperature controller reads "Er 4".

- Open thermocouple, bad connection, or broken wire. Replace/repair thermocouple wire. Contact CETAC Customer Service.

3 Temperature controller reads room temperature.

- Oven fuse blown. Replace fuse. Contact CETAC Customer Service.

4 Temperature controller stops illuminating.

- Thermal safety switch tripped due to excessive temperature. Heat controller or solid-state relay is defective. Contact CETAC Customer Service.

Gas Flow Controller Problems

1 Sweep gas controller does not read zero.

- It is normal for the sweep gas controller to read a value greater than zero because of the inherent design of the controller. Even though the reading is slightly more than zero (0.5 – 1.0) the flow is completely off.

2 Sweep gas controller will not provide 3.0 L/min Argon flow or flow is unstable.

- Argon supply is low. Replace Argon supply.

3 Nitrogen gas controller will not provide 20 mL/min of nitrogen flow or flow is unstable.

- Nitrogen supply is low. Replace Nitrogen supply.

Nebulizer/Plasma Problems

1 Poor and/or unstable mist generation.

- **Insufficient nebulizer flow or pressure.** The Argon nebulizer gas flow should be in a 0.7 - 1.0 L/min range. If such a flow range

cannot be achieved, there may be insufficient nebulizer gas pressure. Increase inlet pressure (not to exceed 100 psi or 700kPa.). Normal operating pressure for the Aridus is 60 psi argon pressure. If the ICP-MS cannot achieve 60 psi, alternative plumbing may be required.

- **Capillary may be plugged.** Backflush the nebulizer line by placing the black cap that came with the nebulizer over the T1H tip while the nebulizer gas is on. This will redirect the gas pressure through the sample line and push out any obstruction. If this procedure is not effective then use the T1H flush kit.
- **Leaking in the nebulizer gas line.** Inspect the nebulizer gas line to make certain that all connections are sound and leak-free. If the line is leaking, repair with appropriate tubing.
- **No signal from the ICP-MS.** Check the sample out line to make certain the Teflon™ lining is not blocking aerosol flow to the ICP torch. If the lining is in the path, recut, clean edge and reinstall.

2 Sample Flow Rate Problems

- **The liquid flow rate is too high.** The liquid flow rate is determined by the nebulizer gas flow. Decreasing the nebulizer gas flow will decrease the liquid flow rate. Consequently, signal intensity may decrease as a result. Proper nebulizer gas flow rates should be optimized based on each individual application. Normal criterion for assessing performance are signal to background ratios and elemental oxide levels.

- **Liquid accumulation in spray chamber.** Replace the peristaltic pump tubing and adjust the pump shoe pressure to be slightly firm. Inspect the pump tubing on a daily basis and replace as required especially when it becomes worn and flattened. **Proper Aridus performance requires that the pump tubing is fresh, maintaining a consistent shoe pressure on the tubing.**

3 Poor precision.

- Poor precision is often due to improperly set gas flows. If the imprecision is characterized by large, periodic drops in signal

intensity, the cause may be an excessive sample flow rate. Poor short-term precision can be caused by a non-optimum sweep gas flow. More concentrated samples (>1% dissolved solids) may also cause poor precision. In addition, such samples may require longer rinse-out times. Finally, readjustment or replacement of the peristaltic pump drain tubing may be necessary to improve precision.

4 High elemental oxide levels.

- High levels of oxides are caused by non-optimum sweep gas flow rate or solids buildup on the membrane. Repeat the sweep gas optimization procedure. If this does not correct the problem, the membrane may require cleaning. Refer to the rinsing procedure. Under normal usage (dilute acid samples), the membrane should be thoroughly flushed approximately every 3 months.
- **Non-volatile membrane build-up.** Make certain that solvents are not analyzed which have a boiling point > 160° C. These solvents will not be removed from the membrane and will require that the membrane be rinsed and the Aridus re-optimized.

5 Excessive nitrogen-containing polyatomic ion interferences.

- Decrease nitrogen flow.

6 Nebulizer self-aspiration stops.

- **The capillary may be clogged.** Backflush the nebulizer capillary by placing the black cap that came with the nebulizer over the T1H tip while the nebulizer gas is on. This will redirect the gas pressure through the sample line and push out any obstruction. A series of bubbles, such as when the sample begins to run dry, may also cause slow or stopped aspiration. If this procedure is not effective then use the T1H flush kit. Refer to Chapter 6, "*Maintaining the Aridus.*"
- Make certain that the nebulizer gas is turned on, the nebulizer gas line is leak-free and the proper amount of gas pressure is being delivered to the T1H nebulizer.

Plasma Problems

Plasma flickers excessively or is unstable.

- The most likely cause of plasma instability is a leak in the Aridus nebulizer / membrane system. Check all gas line connections. Make sure all connections are tight and correct. Very high gas flow may also cause plasma instability. Refer to "*System Optimization*" in Chapter 4, "*Verifying Installation.*"