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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.
WARNING

The handling of organomercurial concentrates which may be used in the preparation of process standards presents a substantial (potentially lethal) safety hazard. Only an experienced, professionally trained organo-metallic chemist, knowledgeable and skilled specifically in the safe handling of organomercurials (using approved apparatus and approved protection measures in an approved facility) should attempt to prepare diluted organomercurial process standards from concentrates.

NOTE

SD Acquisition, Inc., DBA CETAC Technologies assumes no liability for the handling of organomercurial concentrates or the preparation, handling, or use of diluted organomercurial process standards. Instead, CETAC Technologies recommends use of appropriate standard reference materials to validate sample preparation (dissolution/digestion) and use of inorganic mercury standards for instrument calibration.

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 *QuickTrace™ Mercury Analyzer Operator 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.
Notices and Compliance Declarations

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 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-001 of the Department of 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.

The AC power cord is the power mains disconnect for this instrument or accessories.

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.
Notices and Compliance Declarations

WARNING
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 clean 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.

ENVIRONMENTAL

Operating Temperature: 10° to 30°C
Relative Humidity: 0% to 95%
Operating Altitude: 0 to 2000 m

Other Compliance Declarations

Over-voltage Category: Over voltage category II
Pollution Category: Pollution Category II
Rated Degree of Protection: IEC 60529
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 FERMÉE POUR ASSURER LA PROTECTION CONTRE LES BRULURES

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

Caution (refer to accompanying documents)
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.

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.

AVERTISSEMENT
POUR UNE PROTECTION ININTERROMPUE CONTRE LES RISQUES DE DOMMAGES AUX YEUX (DES RAYONS UV "INVISIBLES" SONT PRÉSENTS LORSQUE LE COUVERCLE AINSI QUE LA CARTE DE CIRCUIT SONT ENLEVÉS), LAISSER LE COUVERCLE EN PLACE LORSQUE L'APPAREIL EST EN MARCHE.
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Preface
0 Preface

The QuickTrace™ M-6100 Mercury Analyzer Operator’s Manual provides an overview and explains the theory of operation of the CETAC QuickTrace™ M-6100 Mercury Analyzer. It also supplies QuickTrace™ M-6100 installation and operation information, technical specification data about the systems and sub-systems, and it details troubleshooting and maintenance procedures.

0.1 Who Should Read This Book

The primary audience for the QuickTrace™ M-6100 Mercury Analyzer Operator’s Manual consists of laboratory managers, chemists, technicians, field-service engineers and owners of the QuickTrace™ M-6100. To use this manual (and product) safely and effectively, at least a general knowledge of chemistry, electronic or laboratory equipment, and basic chemical handling procedures are required.

Before operating the QuickTrace™ M-6100, Autosampler, or optional ADX-500, it is important to read this manual, the QuickTrace™ M-6100 Mercury Analyzer Software Manual, the Autosampler Operator’s Manual, and (if applicable) the ADX-500 Autodilutor Accessory Operator’s Manual.

0.2 How to Use This Book

The QuickTrace™ M-6100 Mercury Analyzer Operator’s Manual contains six chapters. Read the chapters sequentially the first time. Thereafter, refer to the chapters separately as needed. The first chapter provides an introduction, system features and performance specifications for the QuickTrace™ M-6100 Mercury Analyzer. Subsequent chapters detail the installation, theory of operation, operation, maintenance, and
troubleshooting procedures associated with the QuickTrace™ M-6100. These six chapters are followed by a Glossary of terms and spare parts catalog.

0.3 Conventions Used in This Book

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

Instructions

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

1 Remove the Gas-Liquid Separator.

Many numbered instructions are followed by more detailed explanations.

Menu Items

This book uses the following format for referring to menu items in the software:

Setting | Communication

The text before the arrow symbol is the name of the menu; the text after the arrow symbol is the menu choice.
Notes

Notes contain a reminder about the effect of particular actions. They 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 QuickTrace™ M-6100 Mercury Analyzer 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.

0.4 Terminology

Terminology used in this manual may be found in the Glossary at the end of the manual.
0.5 Where to Go for More Information

In addition to the QuickTrace™ M-6100 Mercury Analyzer Operator’s Manual, the analyst can refer to the following short list of resources:

The QuickTrace™ M-6100 Software Manual

The CETAC Autosampler Operator’s Manual

The ADX-500 Autodilutor Manual (optional)

U.S. EPA Method 245.1; method for Hg determination in drinking water

U.S. EPA Method 245.7; Mercury in Water by Atomic Fluorescence Spectrometry

U.S. EPA, Office of Solid Wastes. SW846 Method 7470A; Mercury in Liquid Waste (Cold-Vapor Technique)

U.S. EPA, Office of Solid Wastes, SW846 Method 7471A; Mercury in Solid or Semisolid Waste (Cold-Vapor Technique)

American Society for Testing and Methods. ASTM D3223-91; Standard Test Method for Total Mercury in Water

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Introduction
1 Introduction

1.1 Overview

The QuickTrace™ M-6100 Mercury Analyzer is specifically designed to measure trace levels of mercury in aqueous solution by Cold Vapor Atomic Absorption Spectrometry (CVAAS) (i.e. without use of flame, plasma, furnace, etc.). Modular design permits remarkably easy maintenance access and a reduced countertop footprint. Sturdy construction, drift-stabilized double beam optics, thermal and electro-optical lamp stabilization, and an unusually stable “non-foaming” Gas-Liquid Separator (U.S. Patent #5,792,663) collectively afford exceptional structural integrity and signal stability. The QuickTrace™ M-6100 exhibits ultra-low signal noise and detection limits for an absorbance system that is fully compliant with EPA method #245.1.

1.2 System Features

Principal features of the QuickTrace™ M-6100 Mercury Analyzer:

The QuickTrace™ M-6100 incorporates the following features to form an automated, integrated Mercury analysis system.

- Computer-controlled four-channel high-performance peristaltic pump (12-roller pump head).
- Ozone-free Hg Lamp. No lamp ventilation is needed.
- Thermally controlled Hg lamp housing (for a stabilized Hg vapor lamp).
- Stable high performance Gas-Liquid Separator (GLS). (U.S. Patent #5,792,663). Non-foaming/non-bubbling "thin liquid film" GLS design, which allows trouble-free direct analysis of blood, urine, and fish tissue digests as well as standard water and waste analysis.
• Rigid, shock and vibration-isolated optical rail (mounting the Hg lamp, collimator lens, absorption tubes, camera, photo detector, and A/D converter).

• Precise, self-aligning optical mounts, no optical alignment required, maximizing the convenience of instrument baseline zeroing. This design extends maintenance intervals without loss of performance.

• Long path (220 mm) absorbance cells.

• Hg lamp electro-optical feedback beam utilizing a high-performance solid-state detector for ultra-fine lamp stabilization.

• Fixed optical interference filters, three each (254 ± 2nm wavelength, 20%T, 12.7 mm dia.). No moving parts.

• Standard Nafion® dryer cartridge eliminates the need for Mg(ClO₄)₂ drying agent.

• Stabilized double beam optics - traditional double-beam (sample and reference) with a CCD detector.

• Internal ADC (Analog-to-Digital Conversion).

• High-rate data sampling.

• Computer controlled system shutdown/standby routines.

• Integrated optional Autosamplers for accommodation of calibration standards and up to 720 samples.

• RS-232 serial communications.

• Gas exhaust Hg vapor safety trap (solid crystalline KMnO₄).
1.3 System Performance Specifications

Principal performance specifications of the QuickTrace™ M-6100 Mercury Analyzer:

- Low detection limits: typically < 10ppt at 50mL / min. carrier gas flow\(^1\). (Direct steady state absorbance mode, without preconcentration by gold amalgamation).
- Wide dynamic linear working range, \(\approx 5\) orders of magnitude.
- Short term precision, maximum 1.5% RSD, typical \(\leq 1\% \text{ RSD}^{**}\).
- Ultra-low drift rates \(\leq 300\mu\text{Abs/hr}\) (after warm-up) raw uncorrected analog baseline on-screen drifts.
- Ultra-low short-term absorbance noise \(\leq 100\mu\text{Abs}\) (10\(^{-5}\) Abs).
- 0.1% "raw" Hg lamp stability (single beam output).
- Unusually fast washout \(\approx 240\) sec. from 1ppm Hg, at 350mL/min gas flow.
- Mercury Response: \(\geq 6000\mu\text{Abs / ppb}\)\(^2\) at 100mL/min carrier gas flow.

---

\(^1\) One hour minimum warm-up using the standard Nafion® dryer and a gas flow equal to 50mL/min along with prescribed tubing and reagents. Using pump speed, uptake and rinse times specified for standard Nafion® dryer in Table 4-2A, and \(\geq 12\) s integration cycle selected on the "flattest" portion of the peak time profile.

\(^2\) Using prescribed tubing, reagents, and pump speed.
Preparing for Installation
2 Preparing for Installation

Installing the QuickTrace™ M-6100 requires preparation. This chapter discusses what requirements must be met when obtaining supplies and choosing a location for the QuickTrace™ M-6100. It also describes how to successfully unpack the system prior to installation. Before installing the mercury analyzer, first obtain the necessary supplies listed below. Next evaluate the laboratory layout to choose a suitable location. Once a location is chosen, carefully unpack the QuickTrace™ M-6100 prior to installation.

2.1 Supplies

2.1.1 Necessary Supplies

- **Inert Gas Regulator.**
  
  Two-stage, 10-200 psig secondary pressure gauge, with plumbing couple for either a cylinder or dewar capable of delivering 150 psig.

- **AC Power Strip (surge protected) with six outlets.**

- **Cylinder or Dewar, UHP Nitrogen or Ar gas.**

  Ultra-high purity, dry, research grade N₂ or 99.999% purity Ar. The QuickTrace™ M-6100 has a user replaceable 2-micron filter, which prevents damage from particulates to the internal gas control components.

- **Mercury Standard Solution.**

  1000 ppm (minimum order quantity).
• **Hydrochloric Acid Trace Metal Grade (37%).**
  Trace metal HCl will be used in the preparation of Hg standards, SnCl₂ reagent and in some method applications. If an application is more demanding, a better grade of acid may be needed (e.g. double distilled).

• **Nitric Acid Trace Metal Grade (68-70%).**
  Trace metal HNO₃ will be used in sample preparation, cleaning glassware (lab glassware and the QuickTrace™ Gas-Liquid Separator) and added to the QuickTrace™ rinse solution to help maintain the cleanliness of the system during operation.

• **Stannous Chloride (Crystals, Di-Hydrate).**
  Two 500g containers minimum order, “suitable for Hg determination.” The stock SnCl₂ is introduced into the QuickTrace™ at a steady flow rate and therefore any Mercury contamination will be negated during the instrument zero.

• **Potassium Permanganate. Solid, crystalline.**
  The least expensive available grade at a minimum quantity is sufficient unless it is also to be used for oxidative sample preparation. This low-grade reagent stock is sufficient to fill a safety trap for retention of Hg vapor exhaust from the instrument.

• **2-propanol. High purity, “spectrophotometric” grade.**
  2-propanol will be used for cleaning the optical cells and cell windows.

• **Kimwipes®.**

• **Additional Chemical Compounds.**
  The sample preparation procedures of the intended analytical method may require additional chemical compounds. Refer to published method specifications.
2.1.2 Recommended Supplies

- Volumetric flasks 100mL class A (TC) six each.
- Volumetric flasks 1000mL class A (TC) two each.
- Precision air displacement micropipettes, 10 to 10,000 μL (TD).
- Replacement tips for micropipettes.
- Disposable plastic dropping pipettes.
- Graduated cylinders, 10 and 100 mL.
- Polypropylene or polyethylene bottle with Cap, 1 L.
- Weighing balance, top loading, 0.1g readability (or better), any available capacity will work (1.1 Kg capacity is good).
- Laboratory scoopula and large spatula.
- Stopwatch (for measuring liquid uptake rates).
- Stirring rod.
- Powder funnel, wide bore stem, small overall size.
- Wrenches, adjustable 12” and 6”.
- Screw drivers:
  - One (1) small Phillips
  - One (1) medium Phillips
  - One (1) long-shank medium flat-blade
  - One (1) small thin flat-blade
- Deionized water.
- Flow meter 0 - 500 mL/min. with 1 mL/min. readability, calibrated to user’s choice of carrier gas (Ar or N₂).
2.2 Choosing a Location

Choosing a location for the QuickTrace™ M-6100 involves evaluating the laboratory environment for the availability of space, ventilation and power. For the QuickTrace™ M-6100 to function optimally, the location selected must meet specific requirements associated with each of these items. The following sections discuss space, ventilation and power requirements.

2.3 Space Requirements

The QuickTrace™ M-6100 Mercury Analyzer System includes the base unit, peristaltic pump and PC with monitor and requires a minimum footprint for countertop installation of 5’ (152cm) X 2’ (31cm) X 3’ (91cm) (W x D x H). A floor space of 1’ (30cm) X 1’ (30cm) is required for the liquid waste receptacle. The space for the waste can be directly below the analyzer, or directly in front of the lab bench and inline with the peristaltic pump.

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Figure 2-1  Footprint of QuickTrace™ M-6100 Mercury Analyzer System Shown with the ASX-400.
Figure 2-2  Footprint of QuickTrace™ M-6100 Mercury Analyzer System Shown with the ASX 520 and ASX Platform.

Figure 2-3  Footprint of QuickTrace™ M-6100 Mercury Analyzer System Shown as Manual System and ASX Platform.

Figure 2-4  Footprint of QuickTrace™ M-6100 Mercury Analyzer System Shown with the ASX 260 and ASX Platform.

Figure 2-5  Footprint of QuickTrace™ M-6100 Mercury Analyzer System Shown with the ASX 130.
2.4 Ventilation

During operation, the QuickTrace™ M-6100 internally contains trace amounts of Mercury vapor. To prevent inhalation of the vapor, the QuickTrace™ M-6100 uses a solid KMnO₄ absorbent trap located on the back of the instrument. This trap absorbs the mercury vapor prior to final exhaust; therefore no extra ventilation is required beyond that of a standard laboratory environment.

**WARNING**

Gases exhausting from the QuickTrace™ M-6100 cabinet, prior to the external Hg vapor trap (affixed to the rear cabinet panel) contain traces of mercury vapor and must be treated as such. Do not run the QuickTrace™ M-6100 unless exhausted gas is properly “scrubbed” or removed. Fill, maintain and use the provided KMnO₄ absorbent trap or run a transfer line to a fume hood.

Locating the QuickTrace™ M-6100 directly in the path of an air conditioner or heater vent may cause baseline drift, and is not recommended.

**Notice:**

Due to the likelihood of accelerated damage from corrosion and dust, locating the QuickTrace™ M-6100 in a fume hood with stagnant air automatically voids the warranty.
2.5 Power Requirements

The QuickTrace™ M-6100 base unit, autosampler, and peristaltic pump use an external switching power supply. They accept an AC input from 100-240V, 50/60Hz. They supply power at +24VDC to the system. The specific specifications are shown in Table 2-1.

<table>
<thead>
<tr>
<th></th>
<th>M-6100</th>
<th>Autosampler</th>
<th>Peristaltic Pump</th>
<th>Maximum</th>
</tr>
</thead>
<tbody>
<tr>
<td>AC Input</td>
<td>100-240VAC 1.4A</td>
<td>100-240VAC 1.9A</td>
<td>100-240VAC 1.4A</td>
<td>100-240VAC 4.7A</td>
</tr>
<tr>
<td></td>
<td>50/60Hz</td>
<td>50/60Hz</td>
<td>50/60Hz</td>
<td>50/60Hz</td>
</tr>
<tr>
<td>DC Output</td>
<td>+24VDC 2.9A</td>
<td>+24VDC 3.33A</td>
<td>+24VDC 2.9A</td>
<td>+24VDC 9.13A</td>
</tr>
</tbody>
</table>

Table 2-1 Voltage and power requirements

Place the QuickTrace™ M-6100 within 1.2 meters of a standard power outlet. The QuickTrace™ M-6100 instrument and peristaltic pump power input requirements are specified when ordering, either 115 VAC (50/60 Hz) or 220-240 VAC (50/60 Hz). Five power outlets may be required, one each for the QuickTrace™ M-6100 Mercury Analyzer, peristaltic pump, Autosampler, computer, and monitor. (An AC surge protected power strip with six or more outlets will suffice).

The power cord set supplied with the QuickTrace™ M-6100, Autosampler, and peristaltic pump meets the requirements of the country where the instrument was purchased. If the instrument is to be used in a country other than the one specified at the time of ordering, obtain a new 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. For continued protection against electrical shock or damage to the instrument, do not disable this feature.
2.6 Unpacking the QuickTrace™ M-6100

Inspect the external packaging upon receipt for holes, tears, smashed corners, water damage or any other outward signs of damage from rough handling or abuse during shipment. Notify the shipping carrier of the damage to the shipping container and open immediately to inspect for damage. Inspect all items during unpacking and notify the carrier immediately of any concealed damage.

If the QuickTrace™ M-6100 or any of its components are shipped or removed from storage during cold weather, allow the packaged equipment to attain room temperature before opening and exposing to warm, humid air. It is usually sufficient to provide four to eight hours for this purpose.

If water damage has occurred (en route or at destination), or if condensation forms on or inside the QuickTrace™ M-6100 Mercury Analyzer System, allow it to dry thoroughly before connecting it to an AC power source and operating it. Failure to do so may cause equipment damage. Also, notify CETAC Customer Service if any damage is revealed upon inspection.

Remove the packing checklist from the shipping container, and check off items against it. Leave accessories in the packing until needed.

Note:
Do not throw away the factory packaging. Keep it for possible future use.
Preparing for Installation
Installing the QuickTrace™ M-6100
3 Installing the QuickTrace™ M-6100

Refer to Figure 3-1 through Figure 3-10.

![Figure 3-1](image) Front assembly of QuickTrace™ M-6100, Autosampler, and peristaltic pump.
Figure 3-2  Front assembly of QuickTrace™ M-6100, autosampler stand and peristaltic pump. (Manual Operation)

Figure 3-3  Front assembly of QuickTrace™ M-6100, ASX-130, and peristaltic pump.
Installing the QuickTrace™ M-6100

Figure 3-4  Front assembly of QuickTrace™ M-6100, ASX-260, autosampler stand and peristaltic pump.

Figure 3-5  Front assembly of QuickTrace™ M-6100, ASX-520, autosampler stand and peristaltic pump.
Installing the QuickTrace™ M-6100

Figure 3-6  Rear view of QuickTrace™ M-6100, Autosampler, and peristaltic pump.

Figure 3-7  Rear view of QuickTrace™ M-6100 and peristaltic pump.
Installing the QuickTrace™ M-6100

Figure 3-8  Rear view of QuickTrace™ M-6100, ASX-130, and peristaltic pump.

Figure 3-9  Rear view of QuickTrace™ M-6100, ASX-260, autosampler stand and peristaltic pump.
3.1 Placement of the Autosampler Platform

Autosampler and Peristaltic Pump

1 Place the Autosampler on top of the QuickTrace™ M-6100 base unit or Autosampler Platform.

Assemble the autosampler platform and place the M-6100 beneath the completed assembly. (Refer to Figure 3-11, Figure 3-12, Figure 3-2, Figure 3-4 and Figure 3-5) The proper placement of the ASX-130 autosampler should have the tray touching the front cabinet of the base unit. (Refer to Figure 3-3)
2 Place the analytical Peristaltic Pump directly to the left of the QuickTrace™ M-6100 base unit and or platform assembly.

Leave an ~1cm gap. The pump should also be set back so that its front edge is approximately in line with the boundary between the QuickTrace™ M-6100 base unit and the front cabinet or so that the pump tubing bridge stop is lined up with tube pass through hole of the autosampler platform assembly.
3.2 Electrical Connections

Refer to Figure 3-13 through Figure 3-18.

Figure 3-13  M-6100, Autosampler Electrical Connections
Installing the QuickTrace™ M-6100

Figure 3-14  M-6100 Manual mode Electrical Connections
(A and D, power in) (B Computer SCSI cable) (C Pump Manual Override)

Figure 3-15  M-6100 Cables and Accessories
(A, power supply) (B Computer SCSI cable) (C, auxiliary cable gender changer) (D, Pump Manual Override) (E, autosampler COM cable or a USB cable for ASX communication) (F, auxiliary cable)
Installing the QuickTrace™ M-6100

Figure 3-16
M-6100, ASX-130 Electrical Connections
(A, auxiliary cable)

Figure 3-17
M-6100, ASX-260 Electrical Connections
(A, COM or USB cable from ASX COM 1 or USB port to PC COM port or USB port)
Power cords and universal 24 VDC power adapters are included with the QuickTrace™ M-6100 System.

1. Place the on/off switches on the power adapters in the off position.
2. Check the base unit’s lamp power switch immediately below item is off (released “out” rather than pushed “in”).
3. Ensure the main power switch to the Autosampler is off (released “out” rather than pushed “in”).
4. Make sure the main power switch to the peristaltic pump is in the off position.
5. Insert the male jack plug from the power adapter into the female 24 VDC-in jack on the Autosampler.
6. Insert the male six-pin plugs from the power adapters into the female 24 VDC-in connectors on the M-6100 base unit and Peristaltic Pump.
7. Connect the power cords to the 24 VDC power adapters, then insert the other ends into a grounded surge protected power strip.
8. Plug the surge protection power strip into the AC outlet receptacle.

Do not apply power to the QuickTrace™ M-6100 at this time.
3.3 PC Interface

Refer again to Figure 3-13 through Figure 3-18.

The system utilizes a nine pin male RS232 serial communication port (BLUE) or a USB cable for the Autosampler and a high-density 50-pin (SCSI) connector (ORANGE) for the QUICKTRACE™ M-6100 Mercury Analyzer base unit. These can be found in the completion kits.

1 If supplied with a COM cable connect the blue labeled cable to the Autosampler (COM1) port. If a USB cable is supplied connect the USB cable to the USB port on the autosampler.

2 Connect the other end of the blue labeled cable to the computer COM1 or connect the USB cable to a free USB port on the PC.

3 Connect the orange-labeled cable to the M-6100 orange SCSI port.

4 Connect the other end of the orange labeled cable to the computer orange SCSI port.

3.4 Autosampler and Peristaltic Pump Auxiliary Cables

Refer again to Figure 3-13 through Figure 3-18.

Locate the purple-yellow-green (PYG) auxiliary cable in the QuickTrace™ M-6100 completion kit.

1 Connect the green end (37-pin Female) of the PYG cable to the Autosampler green (AUXILLIARY 37-pin Male) port on the ASX-520. If connecting to an ASX-130 or ASX-260 (37-pin Female) the gender changer (37-pin Male-Male) must be used to connect the auxiliary cable to the autosampler.

2 Connect the yellow end of the PYG cable (9-pin Female) to the M-6100 yellow (AUXILLIARY 9-pin Male) port.

3 Connect the purple end (9-pin Male) of the PYG cable to the peristaltic pump purple (CONTROL 9-pin Female) port.
3.5 Installing the Autosampler Z-Drive and Sample Probe

See the Autosampler Operator’s Manual, and follow the instructions carefully.

3.6 Verifying the Installation

Once installation of the QuickTrace™ M-6100 system is complete, it is important to verify that the system is installed correctly.

CAUTION

Attempting to use the QuickTrace™ M-6100 before ensuring that all components are installed correctly may result in damage to the system.

3.6.1 Testing the Interface

1) Power on the QuickTrace™ M-6100, Autosampler, and peristaltic pump.

2) Power on the M-6100 Lamp.

3) Check to ensure that the auxiliary port cables are connected to the equipment according to section 3.3 and section 3.4.

4) Start the QuickTrace™ Software.

5) When the software is initializing, it will test the connections to the peristaltic pump, QuickTrace™ M-6100 System (base unit) and the Autosampler.

The QuickTrace™ M-6100 Software runs a test routine at startup to test the various interfaces throughout the system. The software will give a report on the status of the interface if there is a failure.
3.7 Verify the Software Configuration of COM Ports Manually

If the software does not find the QuickTrace™ M-6100 base unit or the Autosampler it will be necessary for the analyst to test them using the QuickTrace™ M-6100 Mercury Software and the Hyperterminal.exe Program provided with Windows.

**Note:**

If the autosampler fails to communicate and a USB cable was supplied try closing the software and connecting the cable to another free USB port on the PC. Restarting the software will indicate if the communication to the autosampler is restored. USB drivers and port configurations were installed and designated during the factory quality control testing of the system.

To check or change the configuration in the QuickTrace™ M-6100 Hg software:

1. **Open the Specify Installed Hardware application, in the Start menu.**

2. **Note the COM port designated to the autosampler and verify that it is connected to the correct COM port.** The default configuration for a 9-pin serial cable is Autosampler on COM1. If the configuration differs and you are using a 9-pin serial cable set the autosampler to COM1. If using a USB cable the autosampler setting will be a COM port designation of COM port 3 or higher.

3. **Press okay to save changes and exit the application.**

4. **Restart the QuickTrace™ Software.**

   If errors persist, the Windows HyperTerminal Program can be used to manually verify the communication through each serial port.
3.7.1 Using Windows HyperTerminal Program

HyperTerminal (if installed) can be found in the Start menu. Select the Programs | Accessories | Communications folder to find the HyperTerminal program.

Note:
This manual assumes the reader and operator of the QuickTrace™ M-6100 Mercury Analyzer has a working knowledge of using standard Windows applications. For more information on how to use Windows, see your computer's documentation.

1. Click on Hypertrm.exe. (It may be designated HyperTerminal.)

2. If this is the first time that HyperTerminal has been run on this computer, you may be asked to enter an area code and other dialing information. This information will not be used for our procedure, so enter any number you desire. Furthermore you may be prompted to install a modem – this is not necessary and can be ignored.

3. A dialog box titled "Connection Description" will appear. Enter a name significant to you, such as "QuickTrace™ M-6100."

4. A dialog titled "Phone number" or "Connect to" will appear. The only important field is the "connect using" field. Select the desired COM port such as "Direct to com1" or "COM1."
5 A COM port properties dialog box will appear. Be sure the settings are as follows: 9600-8-N-1 (9600 baud, eight data bits, no parity, one stop bit) with no flow control. (As illustrated in the Figure 3-1).

![Figure 3-19 COM port properties dialog box.](image)

6 Under the file menu select “Properties.”

7 A properties dialog box will appear.

8 Click on the setting tab. Click on the “ASCII Setup...” button. A dialog box will appear. Make sure the following settings are checked (Figure 3-20).

![Figure 3-20 ASCII Setup dialog box](image)
9 “Echo typed characters locally”
10 “Append line feeds to incoming line ends”
11 Click OK to confirm these settings.
12 Go to file menu and select Save.

If you have changed the COM port properties, you may have to sever your connection and reconnect to have the new settings take effect. This can be done using two buttons on the toolbar – the one with the phone off the hook disconnects, the one with the phone on the hook reconnects.

The Autosampler will respond to the command VER. Typing VER into the text box followed by pressing the enter key (capitalization is important).

The Auto Sampler will respond ‘CETAC Technologies ASROM...’. If the response is not received as expected on the expected COM ports, then this will indicate a hardware or Windows configuration problem.

### 3.8 Checking the Autosampler Components

See the *Autosampler Operator’s Manual* to verify configuration.

### 3.9 Testing the Sample Probe

Refer to the *Autosampler Operator’s Manual* and the *QuickTrace™ M-6100 Software Manual* to verify proper alignment and movement of the Autosampler.
3.10 Carrier Gas Connection

Refer to Figure 3-21. Locate the plastic-shipping bag labeled “Completion Kit – QuickTrace™ M-6100.” It contains various small parts, tubing, fittings, computer CD-ROM, etc. In this bag, find the brass 2-micron gas filter, with associated brass Swagelok™ fittings, and a short section of ETFE tubing (attached to the filter) which has been formed into a partial loop.

Install the looped end of the ETFE tube into the bulkhead fitting labeled “GAS INLET,” and tighten the fitting very securely. Make sure that the flow arrow on the gas filter is pointing in the direction to the gas in fitting.

Determine how far the QuickTrace™ M-6100 is located from the gas supply (UHP nitrogen or argon). Allow a generous service loop of 1/8” Nylon® tubing from the roll provided the likely event of system placement changes or maintenance. This will allow the system to be slid forward for cell maintenance without disconnecting the gas lines. Connect one end of this tube to the gas inlet side of the 2-micron brass filter and tighten the Swagelok™ fitting securely.

Note:

A 2-micron in-line filter must always be used. The 2-micron filter has been selected for minimal pressure drop and minimal flow fluctuation. Do not substitute other filters.

Figure 3-21 Expanded rear view of QuickTrace™ M-6100.
Connect the other end of the nylon tube to the gas supply regulator, using ¼” NPT 1/8” Swagelok™ fitting provided.

**Note:**

Exceeding 120 psig gas supply pressure to the QuickTrace™ M-6100 may rupture the bulkhead fittings, causing the unit to malfunction.

**CAUTION**

Use only “research-grade”, “dry” UHP Nitrogen or Argon. Do not use “welding” grade gases - these may permanently damage the QuickTrace™ M-6100.

### 3.11 Mercury Trap (KMnO₄)

In the plastic bag labeled "Completion Kit - QuickTrace M-6100," find the polyethylene tube with a seven inch (17.8 cm) length of dark Viton® tubing attached to one end. When filled with crystalline potassium permanganate, this will serve as the mercury vapor trap. The vapor trap will clean the QuickTrace™ M-6100 exhaust vapors, to prevent the release of mercury vapor into the lab atmosphere.

Refer back to Figure 3-21. Remove one end cap from the polyethylene tubular body. Do NOT remove the heatshrink wrapped Luer fitting from the end cap. Inspect both end cap interiors to ensure that the ends are lightly plugged with fine glass wool. If not, lightly pack a small loose wad of fine glass wool into the small i.d. section of each cap. Pack enough glass wool to stop the potassium permanganate from filtering through, but not restrict the gas flow. Once the glass wool is in place, use a powder funnel to fill this tube with dry crystalline solid potassium permanganate (KMnO₄). While filling, have one end fully capped, hold the other end straight upward, and use the powder funnel to guide the KMnO₄ crystals into the tube. Fill to the top, tapping a finger lightly on the tube to settle the KMnO₄, and finally place the end cap on securely.
Be sure to wear protective eyewear and safety gloves when handling chemicals.

Refer again to Figure 3-21. Snap the filled mercury trap into the black holder’s under the silk-screen label "MERCURY TRAP (KMnO₄)." Attach the black Viton® tube with fitting to the connection labeled “GAS EXHAUST.” The Mercury vapor trap needs to be cleaned and refilled when the brown color approaches the open end. This is the formation of the MnO₂ as the KMnO₄ is reduced. The potassium permanganate may last at least one year depending on frequency of use, except in the unlikely event of a major overflow accident in the QuickTrace™ M-6100.

**Note:**
So long as the KMnO₄ is dry, free flowing (not caked), dark purple crystals, it is perfectly OK.

### 3.12 Nafion® Dryer Cartridge

CETAC’s Nafion®-based dryer cartridge¹ (Figure 4-1) is designed as the standard recommended drying system for all CETAC Mercury Analyzers (it replaces the older magnesium perchlorate drying tube). Using the Nafion® dryer substantially reduces daily maintenance and setup time, since daily water saturation will not occur with the Nafion® membrane as it may with the earlier magnesium perchlorate system. It also lowers detection limits and yields more consistent performance. Nafion® cartridges typically last three to six months, whereas older magnesium perchlorate systems must be replaced/recharged daily.

Nafion® Dryer benefits:

- Efficient drying capability.
- No maintenance required.

¹Nafion® is a registered trademark of DuPont (E.I. du Pont de Nemours and Company), which trademark is licensed to Perma Pure, Inc. and used (with permission) herein by SD Acquisition, Inc. DBA CETAC Technologies.
• Improved long-term precision.
• Higher sensitivity.
• Lower detection limits.
• User can easily interchange Nafion® and perchlorate cartridges, if desired (but the Nafion® cartridge is recommended for all applications).
• Replace Nafion® cartridge every three to six months, depending on use.

The CETAC Nafion® dryer cartridge contains a tubular inner Nafion® membrane housed within an outer tube and coiled inside the cartridge. The argon or nitrogen carrier gas containing mercury and water vapor is swept along the inner Nafion® membrane, allowing water vapor to permeate the membrane selectively, whereas the membrane is not permeable to mercury vapor. On the “waste” side of the membrane, a counter gas flow, split from the carrier gas supply, selectively sweeps the water vapor out of the system, whereas non-permeating Mercury vapor proceeds to the sample cell.

The CETAC Nafion® dryer cartridge and associated plumbing is already pre-installed in your new QuickTrace™ M-6100 factory shipment. No further installation is required.
3.13 Plumbing Connections

Refer to Figure 3-23 through Figure 3-26.

3.13.1 Installing the Peristaltic Pump Tubing

1. Release all four peristaltic pump channel clamps.

2. Locate the peristaltic pump tubing in the completion kit. There are two sets of pump tubing. The tubes with the yellow bridge stops are for the sample and drain channels. The tubes with the black bridge stops are for the reagent channel.

3. Install tubes with yellow bridge stops in the first three channels.

4. Install a tube with black bridge stops in the top channel.

![Figure 3-23: Pump tubing placement.](image)
3.13.2 Installing the Mixing Tee and Drain Tees

1 Locate the smallest (1/16” dia.) polypropylene provided in the completion kit. Install this tee on the destination side of the pump. It connects the top two channels.

2 Connect the remaining two larger (3/32” dia.) tees to the two peristaltic pump drain tubes.

Figure 3-24 Tees.
3.13.3 Connecting the Liquid Mix and Drain Lines

1 Connect the Liquid Mix line to the small tee on the outlet side of channels three and four.

2 Connect the Drain line to the ‘large’ tee on the inlet side of channels one and two.

3 Locate the three feet long waste line (1/8” o.d. Tygon® tubing) in the completion kit, and connect it to the ‘large’ tee on the outlet side of channels one and two.

4 Find a Luer male barbed fitting (look for this in the 10 liter waste container). Replace one of the luer caps on the waste container lid with the barbed fitting. Attach the other end of the waste line to this fitting.

To prevent pressure buildup in the 10-liter waste container, be sure that at least one of the two vents on the 10-liter waste container are open (uncapped) during operation.

Figure 3-25 Connect the liquid mix, drain, and waste lines.
3.13.4 Connecting the Reagent Sipper Tube and Sample Probe

1. Find the 11 inch (28cm) length of 1/16” Teflon® tubing inside the reagent bottle. This is the reagent sipper tube.

2. Fill and cap the reagent bottle with 10% SnCl₂ w/v (in 7% HCl v/v). The reducing agent will be used with the QuickTrace™ M-6100 system during operation of the system. Cap the Luer fitting when not in use to preserve the SnCl₂.

3. Connect the 11 inch reagent sipper tube to the inlet side of channel number four.

   When in use, to ensure that no precipitated solids are pumped through the system, the reagent tube should not touch the bottom of the reagent bottle.

4. Connect the 1/16” Teflon® tubing from the sample probe to the inlet side of channel number three.

   ![Figure 3-26](image-url) Connect the reagent sipper and sample probe.
3.13.5 Connect the Autosampler Rinse Station Tubing

The Autosampler peristaltic rinse pump rotates clockwise. The rinse station is always filled from the Fill (bottom) port and drained from the Drain (top) port. For a full description of how to set up the Autosampler rinse station tubing, see the *Autosampler Operator’s Manual*.

**Figure 3-27**  Rinse Station and Rinse Station Pump.

Fill the rinse bottle with trace metal grade 1% HCl / 2% HNO₃ v/v.

**Note:**

When analyzing samples and standards of high concentration such as 20ppb or greater use a stronger concentration of acid i.e., 5% HCl / 2% HNO₃ or similar mix but do not exceed 10% HNO₃.

Locate in the completion kit the ¼” OD Tygon® tubing and refer to Figure 3-27 through Figure 3-31. The tubing will need to be cut to the appropriate lengths.
1 Cut a length of tubing to connect channel one outlet to the fill (bottom) port on the rinse station.

![Figure 3-28 Channel one outlet routing.](image)

2 Cut a length of tubing to connect channel two inlet to the Drain (top) port of the rinse station.

![Figure 3-29 Channel two inlet routing.](image)
3 Cut and connect a length of tubing to connect the channel one inlet so that channel one will pull its supply from the bottom of the Rinse bottle.

Figure 3-30   Rinse Uptake.

4 Cut and connect a length of tube to extend from channel two outlet so extends return a few inches inside the Rinse bottle.

Figure 3-31   Rinse Return.
3.13.6 Peristaltic Pump Tubing Clamp Tension

1. Disconnect “11→HG VAPOR→12” tube from the GLS vapor outlet.

2. Open the QuickTrace software.

3. Open the QuickTrace hardware Controls (see QuickTrace Software Manual) by pressing the Instrument button, or select Window|Instrument.

4. With zero clamp tension on the tubing (screws nearly unscrewed), snap all four clamps into place and start the Peristaltic Pump.

   a) Set gas pressure to 30 psi

---

**CAUTION**

Read through steps 5-8 before proceeding. It is extremely important to set the peristaltic pump drain clamps in a timely fashion after the GLS begins to fill with liquid. Failing to do so can cause an overflow and spillage.

---

**WARNING**

Do not start liquid flow without the carrier gas being on and pressure set to 30 psi. Otherwise, fluid backfill can occur.

Refer to software manual for instrument control.

5. Place the Autosampler sipper tube into rinse station. Use the mouse and click the “Up” button, then the “Park” button. Visually verify the sipper's movement.

6. Manually increase the clamp tension on the Santoprene® sample tubing (Channel three) until liquid uptake begins to flow with a jerky motion in the sipper tube, rotate the tension screw ¼ turn past this point and verify that the flow is steady.

7. Watch the liquid flow as it makes its way from the Autosampler rinse station, to the pump, through the mixing tee, and to the Gas-Liquid Separator.

The GLS will begin to fill with rinse solution.
8 Increase the clamp tension on both of the drain tubing clamps (Channels one & two) until flow begins from the drain port of the GLS and the liquid level begins to drop.

Do this quickly before the GLS fills to the point where fluid overflows the GLS. The GLS should slowly drain to empty, even though sample continues to be delivered to the top of the frosted center post. The GLS is intended to operate “empty” with only a thin film of liquid continuously wetting the frosted center post and exiting the drain.

9 Tighten both drain tube clamps equally to ensure even flow to each.

Check this by observing the segmented flow at both drain tees. The rate of flow in and out should be balanced through both Santoprene drain lines. Adjust the clamp tension to keep the GLS empty and to achieve a smooth, balanced, segmented flow. Unstable drain flow can cause baseline noise in the system. The drain tube tension should exactly match the sample flow tension.

10 Start the SnCl₂ flow.

a Place the SnCl₂ uptake tube in the reagent bottle

b Close the Channel 4 clamp.

c Increase the reagent clamp tension until reagent uptake begins in the tube.

d Adjust the clamp so that the flow from the SnCl₂ bottle is smooth, with no jerks in the flow.

11 Once liquid is running through the QuickTrace M-6100, note that the drain tubing clamp tension is properly adjusted by watching the flow through the Gas-Liquid Separator.

The GLS should remain empty and liquid exiting the GLS should appear nearly motionless, with no flutter or instability.
12 The flow into the GLS should be as smooth and pulse free as possible.

View this closely at the top of the GLS frosted center post. The liquid should stream continuously from the capillary tip to the top of the post, and the liquid column spanning the gap between the capillary tip and post should be nearly “motionless,” with minimal fluctuation and no jerkiness or discontinuity. If this is not the case check that the “gap” between the bottom of the GLS capillary insert tube and the top of the GLS frosted center post is ~0.5 mm (range of 0.3-0.6 mm). If not, very carefully slide this insert up or down, as needed. Refine the clamp tension of the sample and reagent channels as needed to stabilize the liquid flow to the GLS.

A flow check with a 10ml graduated cylinder (less than 100mm tall) and stopwatch should yield a sample uptake rate of ~5 mL/min, and a reagent uptake rate of ~1.8 mL/min. Check liquid flow stability at the drain exit of the GLS after final adjustments of clamp tension to sample and reagent pump tubing. The pump tension will not need further adjustments, do not adjust pump tension to compensate for worn pump tubing.
Flow Check Procedure – Reagent:
1 Fill the 10ml graduated cylinder with 10ml DI water.
2 Simultaneously place the reagent sipper tube in the graduated cylinder and start the stopwatch.
3 After 30 seconds, remove the uptake tube from the cylinder.
4 Measure the water remaining in the cylinder, and calculate the reagent flow rate.

Flow Check Procedure – Sample Probe:
1 Fill the 10ml graduated cylinder with 10ml DI water.
2 Move the Sample probe to the middle of the sample rack. (1:35 if set for a 60 position rack).
3 Position the graduated cylinder beneath the sample probe.
4 Simultaneously click the Down button and start the stopwatch.
5 After 30 seconds press the Up button.
6 Measure the water remaining in the cylinder, and calculate the flow rate.
7 Park the sample probe.

Note:
When properly adjusted the tension on the bottom three peristaltic pump tubes should be the same and the tension screw for the top pump tube (reagent tube) should be screwed 1 to 2 mm in farther.

Once the clamp tension on the pump tubing is established, relieve their stretch. Unclamp reagent and sample clamps. Park the sample probe. Press the Up button to remove the sample probe from the rinse station. Remove the sipper tube from the SnCl₂. Allow the waste line to empty. Unclamp waste line clamps. Turn the pump off using the software controls.

Do not leave tubes clamped in place when the system is not being used. The next time the system is used, hook the tubes and close the quick-release mechanisms. No screw adjustments will be needed. Previous clamp tension is “remembered” as the quick release is engaged and disengaged.
Installing the QuickTrace™ M-6100
Using the QuickTrace™ M-6100
4 Using the QuickTrace™ M-6100

Operation of the M-6100 is mostly through the QuickTrace™ Software interface.

For a detailed description of the software see the QuickTrace™ help file and the QuickTrace™ Mercury Analyzer Software Manual.

The following chapter covers physical operation instructions to achieve optimum performance.

4.1 Theory of Operation

4.1.1 Autosampler

The autosampler is prepared for operation by loading sample vials of digested samples, into selected positions of the sample racks. Vials of calibration standards are placed in user-selected positions of the standards rack. Rinse solution, for sample-to-sample probe decontamination, fills the rinse station and re-circulates to the rinse bottle.

After a method worksheet is prepared/loaded, the system is ready for unattended operation to begin. The Autosampler operates under computer control to move the sample uptake probe to any sample position; the rinse station, reference standard, blank, etc., in a user-programmed sequence. The sample uptake probe (sipper tube) supplies the multi-channel peristaltic pump's sample inlet.

For further information, see the Autosampler Operator's Manual.
Figure 4-1  QuickTrace™ M-6100 Systems Functional Block Diagram

4.1.2 QuickTrace™ M-6100 Automated Mercury Analyzer

Sample Introduction & Stannous Chloride Reactor

Refer to Figure 4-1 to trace the path of liquids through the M-6100 System. An acidified digested aqueous sample from the autosampler is introduced, via peristaltic pump as Hg²⁺ dissolved in solution. A reducing agent (10% stannous chloride in 7% HCl), is introduced via a parallel pump channel. The sample and reagent (SnCl₂) streams join at
the mixing tee (1), and immediately enter the QuickTrace™ M-6100 tubing reactor (“Liquid Mix”). Sn2+ reduces Hg2+ in solution to Hg0 while the mixture is en route to the Gas-Liquid separator (GLS). At this stage and prior to the GLS, the analyte is present as a finely dispersed emulsion of liquid (metallic) Hg0 micro-droplets, in excess SnCl₂ solution.

**NOTE:**
The CETAC QuickTrace™ M-6100 Mercury Analyzer measures inorganic Mercury (free Hg²⁺ or HgCl₂, which is subject to efficient stannous chloride reduction in the QuickTrace™ M-6100 tubing reactor); using inorganic Mercury standard solutions for instrument calibration. If insoluble Mercury, bound Mercury, or organomercurials are present in samples, an appropriate sample dissolution/digestion procedure will have to be employed to convert these other forms to free inorganic Hg²⁺ or HgCl₂, prior to analysis with the QuickTrace™ M-6100.

**Gas-Liquid Separation**

The finely dispersed Hg⁰/SnCl₂ emulsion is introduced into the top of the GLS (Gas-Liquid Separator (2)). The Hg⁰/SnCl₂ emulsion flows over the frosted GLS center post in a relatively thin film, covering the entire post from top to bottom. A carrier gas simultaneously enters the bottom of the GLS tangentially (10). The carrier gas (Ar or N₂) swirls around the wetted center post and upwards toward the GLS gas exhaust port (11).

Hg⁰ droplets in the thin emulsion film quickly evaporate into the gas vortex surrounding the post. The carrier gas stream efficiently sweeps this Hg⁰ vapor (along with some evaporated water) upward and out of the GLS gas exhaust (11), and on to the drying (12, 13) and optical section (14, 15) of the QuickTrace™ M-6100 for CVAAS analysis.

The liquid water, containing excess reducing agent, acid, any non-participating "spectator ions", and reaction by-products, finally drains
out the bottom of the GLS (3) and is pumped to waste (4), (channels one
& two).

<table>
<thead>
<tr>
<th>Note:</th>
</tr>
</thead>
<tbody>
<tr>
<td>The GLS operates “empty” with no liquid level. The liquid spreads out as a film that wets the center post. At the bottom of the post, the film collects at a single point and is then continuously pumped to waste, so the “liquid level” should not rise in the GLS.</td>
</tr>
</tbody>
</table>

**Carrier Gas**

Refer again to Figure 4-1 to trace the path of the carrier gas. A clean, dry carrier gas, such as UHP N₂ or Argon, must be supplied to the back of the instrument. The gas passes through fixed restrictors to produce primary flow rates in the range of 20-350 mL/min @ 10-100 psig. The carrier gas first enters the reference cell (6) to facilitate measurement of the incident radiant power ($P_0$) at 253.7 nm. It exits (7) and passes through the GLS (10) to pick up Hg⁰ vapor from the reduced sample. The carrier gas and Hg⁰ vapor exit the GLS (11) and enter (12) a Nafion® drying cartridge where water vapor is removed (13). For the Nafion® dryer, an auxiliary sweep gas from a restrictor (5A) enters an auxiliary port (12A) and selectively removes water vapor from the dryer cartridge at 13A.

Finally, the dry Hg⁰/carrier gas mixture exits the dryer (13) and enters the sample cell (14) for measurement of transmitted radiant power ($P$) at 253.7 nm.

Ultimately, the gas stream (carrier gas and Hg⁰) exits the sample cell (15) and is exhausted to a solid KMnO₄ trap (16) where Hg⁰ is absorbed, and clean carrier gas passes to the lab atmosphere.

**Optics and Cold Vapor AAS**

Refer again to Figure 4-1 to trace the optical path of the QuickTrace™ M-6100. The Cold Vapor AAS (Atomic Absorption Spectrometry) process
within the sample cell begins with a low pressure, high frequency, thermally stabilized, electro-optically regulated Hg vapor lamp, which produces the Hg emission spectrum. Emitted light is collimated (L₁) and projected in two parallel, isolated beams one each through the reference and sample cells. Absorbance of 253.7 nm radiation by Hg⁰ vapor (derived from the chemically reduced sample and GLS) occurs only in the sample cell. P is thereby decreased, relative to P₀.

Light from the cells enter the binocular camera, where both collimated beams are independently focused (L₂) and filtered (F) before reaching the Charged Coupled Device (CCD) detector. Narrow band 254 +/- 2 nm interference filters (F) remove all radiation but the strong 253.7 nm Hg⁰ "resonance line" from both the sample (P) and reference (P₀) beams. By a photovoltaic effect, the CCD converts the light beams into electrical signals, proportional to radiant power (P and P₀). These outputs are processed to yield an electrical signal proportional to optical absorbance (Abs = -log (P/P₀)).

4.2 Software

In the host computer, the sample absorbance value is drift corrected, blank subtracted, if through blank is desired. The absorbance value is then measured against a calibration curve derived from previously obtained absorbance values of calibration standards.

The QuickTrace™ Software operates under a Windows environment. The QuickTrace™ Software provides complete instrument, autosampler control. The QuickTrace™ Software also provides a variety of EPA compliant quality control functions, display features, report generation and diagnostic routines.

The user interface is sufficiently powerful that it will satisfy the requirements of experienced technically advanced analysts and scientists.

The reader is referred to the separate QuickTrace™ Help file and QuickTrace™ Mercury Analyzer Software Manual for a detailed
description of the software features, functions, and operation instructions.

4.3 Preparing Reagents and Calibration Standards

Always use high purity gas, chemicals, acids, water, standards, and clean glassware for analysis. It may be necessary to acid wash and rinse all glassware more than once to eliminate contamination for the most sensitive mode of operation (<20ppt).

Recommended Chemical Concentrations:

<table>
<thead>
<tr>
<th>Reagent</th>
<th>10%(w/v) SnCl₂ in 7%(v/v) HCl.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standards for Instrument</td>
<td>All instrument calibration standards (Hg²⁺) are prepared in 7%(v/v) HCl then an aliquot of standard is treated to the same procedure as a sample prior to instrument calibration.</td>
</tr>
<tr>
<td>Rinse Solution</td>
<td>1% v/v HCl / 2% v/v HNO₃ trace metal grade in the autosampler rinse reservoir bottle.</td>
</tr>
</tbody>
</table>

**NOTE:**

HCl media must be used for stock standard preparation! Hg standards in nitric acid (only) are unstable at ppb levels and below. With 7% HCl media (in glass at room temperature), the standards are stabilized for weeks at the ppb level, and for several days at the ppt level. Standards must be 7%(v/v) HCl even after final dilution.
NOTE:
Acidified rinse prevents carryover contamination from high samples and standards to subsequent low samples. (Deionized water (alone), as rinse does **not** ensure this.)

NOTE:
The CETAC QuickTrace™ M-6100 Mercury Analyzer measures **inorganic** Mercury (free Hg\(^{2+}\) or HgCl\(_2\), which is subject to efficient stannous chloride reduction in the QuickTrace™ M-6100 tubing reactor). Inorganic Mercury standard solutions are used for instrument calibration. If insoluble Mercury, bound Mercury, or organomercurials are present in samples, an appropriate sample dissolution/digestion procedure will have to be employed to convert these forms to free inorganic Hg\(^{2+}\) or HgCl\(_2\) prior to analysis with the QuickTrace™ M-6100. If it is desired to confirm the oxidative digestion procedure accuracy (recovery) regarding organomercurials, then organomercurial standards or appropriate standard reference materials would have to be carried through the digestion as “process standards”.

WARNING
The handling of organomercurial concentrates, which may be used in the preparation of process standards, presents a substantial (potentially lethal) safety hazard. Only an experienced, professionally trained organo-metallic chemist, knowledgeable and skilled specifically in the safe handling of organomercurials (using approved apparatus and approved protection measures in an approved facility) should attempt to prepare diluted organomercurial process standards from concentrates. Always be sure to obtain and carefully read the MSDS (Material Safety Data Sheets) before handling organomercurials!
NOTE:

SD Acquisition, Inc., DBA CETAC Technologies assumes no liability for the handling of organomercurial concentrates or the preparation, handling, or use of diluted organomercurial process standards.

In most cases, CETAC Technologies recommends that samples be oxidized following standard, safe, well known, approved sample dissolution or digestion procedures, and that the QuickTrace™ M-6100 instrument calibration standards be prepared only from inorganic Mercury concentrates or diluted from commercially available inorganic Mercury standard solution concentrates. Where possible, the recommended means of overall process (dissolution/digestion + QuickTrace™ M-6100 analysis) validation should be through use of commercially available standard reference materials (SRM's) of composition matching (or similar to) the samples and containing certified, known mercury levels in a concentration range similar to the samples. (Being by far the safest alternative, this SRM approach to overall process validation should be used whenever possible, and is nearly always preferred to preparing diluted process standards from hazardous organomercurial concentrates!)

4.4 Gas Parameters

The QuickTrace™ M-6100 has a manual gas control. The user adjusts the gas pressure to achieve a desired flow rate.

**Carrier Gas**  
N₂ UHP, high purity grade cylinder (dry, research grade) or Argon, high purity grade (e.g. liquid dewar boil-off or cylinder).

**Gas Pressure**  
20-100 psig

**Gas Flow Rate**  
20 - 350 ml/min
QuickTrace™ M-6100 Mercury Analyzer Operator’s Manual

Using the QuickTrace™ M-6100

<table>
<thead>
<tr>
<th>Pressure setting (psig)</th>
<th>Nominal Flow (ml/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>20</td>
</tr>
<tr>
<td>20</td>
<td>55</td>
</tr>
<tr>
<td>30</td>
<td>95</td>
</tr>
<tr>
<td>40</td>
<td>140</td>
</tr>
<tr>
<td>60</td>
<td>240</td>
</tr>
<tr>
<td>80</td>
<td>295</td>
</tr>
<tr>
<td>100</td>
<td>350</td>
</tr>
<tr>
<td>120</td>
<td>415</td>
</tr>
</tbody>
</table>

Table 4-1    M-6100 Carrier Pressure vs. Flow

For exact pressure and flow parameters for your instrument, please consult the final test documentation, which accompanied the instrument.

See Table 4-2 and Table 4-3 for a more complete listing of optimal instrument setups.

4.5 Start-up and Analysis of Samples

Power on the QuickTrace™ M-6100, Autosampler, system lamp and peristaltic pump. Open the QuickTrace™ Software. The lamp will require about 15 minutes to stabilize. Other instrument prep may be performed during this time.

Once the QuickTrace™ M-6100 has powered up, check that the four channel peristaltic pump is plugged in. Make sure the pump tubing is installed and tension is adjusted per Section 3.13. Check also that supply gas is connected and 60psig pressure is applied to the unit. This is the pressure at which we will perform the “Wetting of the GLS,” described below.

Ensure that the KMnO4 trap is filled per Section 3.11.
4.5.1 Wetting of the GLS

At the beginning of each day or after any period of pump inactivity and prior to analysis ensure that the GLS center post is fully wetted.

Refer to Figure 4-2 while reading the procedure below.

1. Disconnect “11→HG Vapor→12” tube from the GLS vapor outlet.
2. Set the gas pressure to 60 psig. This should result in a gas flow of about 240 mL/min.
3. Check that the bottle supplying the Autosampler rinse station is filled with clean trace metal grade acidified rinse solution.
4. Place the reagent sipper in a beaker of DI water.
5. Turn peristaltic pump on. The instrument controls in the software control the peristaltic pump on off and standby functions.
6. Clamp the pump quick release mechanisms.

Figure 4-2  Wetting the GLS
7 Using the quick-release mechanisms, fully release the clamp tension on channels one & two (drain channels) of the peristaltic pump.

8 Pinch the drain tube as shown in Figure 4-2.

9 With the drain pump tubes unclamped, the GLS should begin to fill with liquid. Once the liquid level rises, gas will bubble through it.

10 Allow the GLS to fill until a gas bubble propels a "meniscus" upward to wet the post all along its length, including its top. (THE POST IS NOW WETTED)

11 When this happens, re-engage the quick-release clamps on the drain pump tubes and un-pinch the drain line. With the drain tube clamps properly re-engaged and the pump running, the liquid will begin draining.

12 Once the GLS has "emptied", leave the pump running (keep liquid flowing), reconnect "11→HG Vapor→12” tube to the GLS vapor outlet.

13 Place reagent sipper in the SnCl₂.

14 The post is now wetted and the QuickTrace™ M-6100 is ready to run samples.

The analyst may now operate the QuickTrace™ M-6100 to perform analysis of samples. The QuickTrace™ M-6100 Mercury Analyzer help file has been developed specifically to assist the analyst in this task. Refer to the help file to perform the desired analytical tasks. Once finished, the analyst should place the QuickTrace™ M-6100 instrument in either Standby (See the QuickTrace™ M-6100 Mercury Analyzer help file) or Cold Shutdown condition.
4.6 QuickTrace™ M-6100 Startup Summary

1. Initiate QuickTrace™ M-6100 software (if software was left open and in standby, open the QuickTrace™ M-6100 controls and start the autosampler rinse pump (click pump on and probe down)).

2. Turn on lamp and initiate the carrier gas flow. A minimum of a 15-minute warm-up time is required.

3. Clean and rinse the 2L rinse bottle with DI water and refill with the desired trace metal grade HCl / HNO₃ solution.

4. Place the autosampler rinse tubing into the rinse bottle.

5. Prepare a fresh 10% SnCl₂·H₂O w/v 7% HCl v/v solution if old solution is yellow (oxidized) or precipitated. Prepare only what you need to complete the calibration and sample run including all QC checks and spikes. The reagent flow is ≈ 1.5 ml/min.

6. Verify that the sample capillary (inlet insert) is 0.5mm above the Gas-Liquid Separator (GLS) center post.
7 Open vents on waste container.

8 Inspect peristaltic pump tubing for wear and flat spots (replace if necessary). Place the peristaltic pump tubing in their appropriate shoes and holder clips. **Do not lock shoe clamps at this time.**

9 Place the reagent capillary in a beaker of DI water.

10 Lock down the peristaltic shoe clamps.

11 **Inspect liquid flows.** The GLS drain should be flowing smoothly with no build up or pulsing of liquid. The waste line from the peristaltic pump to the waste container should be liquid/gas etc... with no vibration. **If this is not the case upon inspection, stop immediately and change GLS drain line and or waste line.**

12 Wet the GLS center post per Section 4.5.

13 Inspect the rinse station for a convex liquid bubble adhering to the sample probe. If this is not the case, change the rinse pump peristaltic tubing.

14 Open the appropriate worksheet **(See QuickTrace™ M-6100 help file)** and set the gas pressure to match the method.

15 Record lamp V monthly (See section 5.14 and Figure 5-16) in the daily instrument logbook.

16 **Zero the QuickTrace™ M-6100 using the auto zero.**

17 Peak profile the high standard and verify baseline and sample integration times. Record μAbs and concentration of the peak profile standard in a daily instrument logbook. **Note: This operation should be performed on the highest standard.**

18 Calibrate instrument and analyze samples.
Summary of Gas and Liquid Flows for Analytical Ranges of the QuickTrace™ M-6100

<table>
<thead>
<tr>
<th>RANGE #1: M-6100 PPT</th>
<th>&lt;0.01 - 1 ppb Hg</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gas Pressure</td>
<td>20 psig</td>
</tr>
<tr>
<td>Peristaltic Pump Speed</td>
<td>High</td>
</tr>
<tr>
<td>Sample Flow Rate</td>
<td>~ 5 mL/min</td>
</tr>
<tr>
<td>Sample Time (for Liquid Uptake or Autosampler &quot;Sip&quot;)</td>
<td>50 s</td>
</tr>
<tr>
<td>Rinse Time</td>
<td>100 s</td>
</tr>
<tr>
<td>Read Delay</td>
<td>65 s</td>
</tr>
<tr>
<td>Replicate Read Time</td>
<td>3 s</td>
</tr>
<tr>
<td>Replicates</td>
<td>4</td>
</tr>
<tr>
<td>Baseline Correction Method</td>
<td>2 point (15-25s &amp; 136-146s)</td>
</tr>
</tbody>
</table>

**Expected Results:** 100 ppt | 7% HCl
~1400 uAbs

Detection Limit (nominal): < 0.010 ppb
Sample Throughput Rate (minutes/sample) 2.5 min/sample
Dryer Cartridge Life 3-6 months

**Table 4-2** PPT Parameters to Optimize.
Figure 4-3  Typical Results from PPT Settings.

<table>
<thead>
<tr>
<th>RANGE #2: M-6100 PPB</th>
<th>0.1 – 20 ppb Hg</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gas Pressure</td>
<td>40 psig</td>
</tr>
<tr>
<td>Peristaltic Pump Speed</td>
<td>High</td>
</tr>
<tr>
<td>Sample Flow Rate</td>
<td>~5 mL/min</td>
</tr>
<tr>
<td>Sample Time (for Liquid Uptake or Autosampler &quot;Sip&quot;)</td>
<td>30 s</td>
</tr>
<tr>
<td>Rinse Time</td>
<td>70 s</td>
</tr>
<tr>
<td>Read Delay</td>
<td>49 s</td>
</tr>
<tr>
<td>Replicate Read Time</td>
<td>1.5 s</td>
</tr>
<tr>
<td>Replicates</td>
<td>4</td>
</tr>
<tr>
<td>Baseline Correction Method</td>
<td>1 point (26-30s)</td>
</tr>
<tr>
<td><strong>Expected Results:</strong></td>
<td>10 ppb</td>
</tr>
<tr>
<td>Detection Limit (nominal):</td>
<td>&lt; 0.05 ppb</td>
</tr>
<tr>
<td>Sample Throughput Rate (minutes/sample)</td>
<td>&lt; 2 min/sample</td>
</tr>
<tr>
<td>Dryer Cartridge Life</td>
<td>3-6 months</td>
</tr>
</tbody>
</table>

**Table 4-3**  PPB Parameters to Optimize
4.7 Placing the QuickTrace™ M-6100 in Standby Mode

To prolong instrument life, it is not recommended to leave the system fully on (with lamp lit) overnight, or when not in use. However, to speed the next day’s startup, the QuickTrace™ M-6100 can be left on with the lamp off overnight without significantly shortening its life. When analysis is done for the day, rinse with 10% HNO₃ for several minutes through the reagent uptake tube. Then rinse with deionized water through the reagent uptake tube for several minutes.
Note:
Failure to perform this “shutdown rinse” may result in a system clog.

Next, withdraw the reagent uptake tube and using the QuickTrace™ M-6100 Software Controls withdraw the autosampler probe from the rinse station. Continue pumping until the drain tubing runs fully empty. Turn off the pump, unclamp all pump tubes and unhook them from one side of their tubing bridge. Turn off the gas (main supply) and disconnect the Hg vapor tube from the GLS.

Leave the system mains power on. The Hg lamp sent with the system has an operation life of ~5000 hours, but internal optical filter life may be substantially extended by turning off just the lamp whenever analyses are not being performed. Leaving the main power on leaves the lamp block heaters on, and consequently the lamp block remains thermally stable. To reactivate the instrument to “run” status, simply turn the lamp on again, re-establish appropriate gas and liquid flows and operate the instrument normally. The system will be stable and ready to run within 5-10 minutes.

4.8 Cold Shutdown

For a total system shutdown (to cold condition), prepare the pump tubing as described in Section 4.6 above. Exit the QuickTrace™ Software, shutdown Windows and turn off the computer. Turn off the Hg lamp, gas, Autosampler, pump, and QuickTrace™ M-6100 main power.
4.8.1 Summary of QuickTrace™ M-6100
Shut Down
Shutting down the QuickTrace™ M-6100 and autosampler.

1. Place the reagent capillary in a beaker of 10% HNO₃ and cap the reagent bottle. Rinse the system for a minimum of ten minutes.
2. Place the reagent capillary in a beaker of DI water and rinse the system for one minute.
3. Raise sample probe via QuickTrace™ M-6100 controls.
4. Remove reagent capillary from DI water.
5. Allow the drain and waste lines to run completely dry.
6. Turn off peristaltic pump.
7. Release peristaltic shoe clamps and release the pump tubing from the tubing bridge.
8. Close vents on waste container.
9. Disconnect GLS exhaust line from GLS.
10. Turn off gas and lamp.
11. If you are going to use the instrument the next day or in the near future, leave the instrument in this condition. It will then be ready for a warm start.
12. If you are not going to be using the instrument in the near future then exit the QuickTrace™ Software and turn off the autosampler and QuickTrace™ M-6100.
Note:
Before shutting down the instrument to either Standby or Cold condition, remember to run 10% HNO₃ and deionized water through the SnCl₂ reagent lines. This will clean out any chemicals from the peristaltic pump and sample tubing and prevent residue encrustation in the Gas-Liquid Separator and its drain. Remember to pump all lines completely dry after rinsing.

CAUTION
Always remember to release all clamps and unhook the pump tubing from the peristaltic pump. Failure to release clamps and unhook the tubing when the pump is off, will cause tube fatigue and lead to poor results (bad % RSD) when used for analysis the next time.
Maintaining the QuickTrace™
M-6100
5 Maintaining the QuickTrace™ M-6100

5.1 Daily Maintenance (Always Check Before Analysis)

- Ensure the autosampler rinse bottle is rinsed with DI water and refilled with acidified rinse solution. For concentration of standards and samples greater than 20ppb a 5% HCl / 2% HNO₃ v/v should be sufficient.

- Ensure the rinse bottle tubes are completely submerged in rinse solution. The rinse station supply tubing should be at the bottom of the rinse bottle and the rinse station return tubing should be at the top of the rinse bottle. This will ensure that the rinse is a true recirculating rinse. Inspect the rinse station flow and ensure that the rinse is not removed via the sample probe faster than it is supplied. Replace the ASX rinse pump tubing periodically for best performance (See the ASX Operator’s Manual).

- Inspect the sample peristaltic pump tubing for fatigue and wear. Replace if too worn or fatigued.

- If the pump tubing was left clamped overnight, install new tubing.

- Pre-wet the GLS center post and be sure it remains completely wet during operation.

- Check that the liquid flows, to and from the GLS, are smooth. Verify by close inspection the inlet to the GLS center post and drain exit points.

- Be sure the waste bottle will not overflow during the run.
• Check that the reagent bottle is sufficiently full for the number of samples to analyze.

• Check that the SnCl₂ is fresh and not precipitated, crystallized, yellowed, or oxidized or that the small cap on reagent bottle was left open overnight. Replace if necessary.

• For autosampler maintenance, see the appropriate ASX Operator’s Manual.

5.2 Weekly Maintenance

• Remove the GLS and clean if residue is building up. This is described later in section 5.9.

• Clean the SnCl₂ reagent bottle weekly or before refilling.

• Change the pump tubing if it is too worn, appreciably “flattened,” or left in place overnight.

• Empty the waste bottle. Cap all Luer fittings to carry this bottle.

• Check the cells and cell windows for cleanliness.

5.3 Monthly Maintenance

• Clean the GLS. See Section 5.9.

• Clean the cells and cell windows. See Sections 5.6, 5.7 and 5.8.

• Replace the GLS inlet tubing and capillary insert. See Section 5.11.1.
• Replace the GLS drain tube if it is clogged or dirty. This is described in Section 5.11.2.

• Check that the Nafion® dryer cartridge is still good. A failing Nafion® cartridge may be indicated by loss of mercury absorbance sensitivity and an increase in the baseline of more than 1000 μabs during a short run of 30 minutes or less. If the Mercury absorbance for a given standard solution drops to 50% or more of its original value, change the cartridge. See Section 5.12.

5.4 Yearly Maintenance

• Replace the Nafion® dryer cartridge bi-yearly, or as needed. (See Section 5.12). A failing Nafion® cartridge may be indicated by loss of Mercury absorbance sensitivity and an increase in the baseline of more than 1000 μabs during a short run of 30 minutes or less. If the Mercury absorbance for a given standard solution drops to 50% or more of its original value, change the cartridge.

• Replace the 2-micron filter (See Figure 3-6).

5.5 Autosampler Yearly Maintenance

• Replace the sample probe.

• Replace the rinse peristaltic pump tubing.

See the Autosampler Operator’s Manual.
5.6 Removal or Inspection of the Sample Cell

5.6.1 Opening the Optics Door

The instrument is shipped with the optics access door secured with thumbscrews (Figure 5-2).

**WARNING**

For continued protection against hazards indicated on the warning labels, always retighten these four metal screw-knobs securely.

Refer to Figure 5-2. For access to the optics interior, first turn off the main power. Use a flat-blade screwdriver to loosen the thumbscrew on the optical access door.
Refer to Figure 5-3. Remove the thumbscrews on the optical cell clamps.

5.6.2 Removing the Sample Cell

Refer to Figure 5-3 and Figure 5-4. The cells are designed for simple removal and cleaning. When removing the cells, be careful to not touch the cell windows at the ends of the optical cells. If the cells and cell windows are dirty, use a clean foam swab and isopropyl alcohol (spectrophotometric grade only) to clean the surfaces (See Section 5.7). If needed, the windows can be taken out by removing the end caps and the O-rings.

Once the cell clamps have been removed, disconnect the carrier gas lines from the cell end caps. Remove the cell end caps by holding the glass cell, pull, and rotate the end cap until it slides off the glass cell. Repeat this procedure for the reference cell. Inspect and/or clean the cell and its windows per the instructions in sections 5.7 and 5.8, or perform tubing maintenance as described in section 5.10.
5.7 Cleaning the Cell Windows

Refer to Figure 5-4. There are two ways to clean cell windows:

- **Quick exposed surface cleaning (without dismantling).**
- **Dismantling for total cleaning.**

The need for cleaning (or re-cleaning) is determined by close inspection of the window (D), visible through the hole in the window cap (A), while maintaining a low-angle total surface reflection of room light on the window. Any film, fingerprint, dust, or dirt will show up dramatically against the “white” background of a low-angle surface reflection of room light from the window.

5.7.1 Quick Exposed Surface Cleaning

Cleaning the exposed surface of the window requires the following: a clean foam swab, Kimwipes®, and a bottle of isopropyl alcohol (use only spectrophotometric grade).

**Note:**

Do NOT use cotton swabs. They will leave small bits of lint, which can offset the absorbance baseline and add a great deal of noise, if the lint moves or flutters in the optical beam. Use only clean foam swabs. To pre-clean the foam swab, rinse in alcohol and dry with a Kimwipes®. Do not dip the swab in the alcohol supply (when new, the swabs may be dirty and contaminate the alcohol supply). Instead, squirt alcohol onto the swab with a wash-bottle that is for alcohol only and dry with the Kimwipes®. Rinse and only lightly blot the swab with Kimwipes® when cleaning, this will leave the swab moist with alcohol, which will be enough to clean the cell windows.
Using the pre-cleaned, alcohol-moistened swab, gently swab the outside of the cell window. Allow to air dry or blow-dry with UHP Argon or Nitrogen. Re-check the surface reflection to see if the window is completely clean. If residue, fingerprints, or particles remain, repeat the process with another pre-cleaned alcohol-moistened foam swab until the windows are clean. If this quick procedure fails, it may be necessary to dismantle the assembly for more rigorous “total” cleaning, as described below.

**Figure 5-4**

Cell assembly diagram.
A - Window cap  C - Phillips screws  E - Cell end cap
B - Window O-ring  D - Sapphire windows  F - Glass cell
5.7.2 Dismantling for Total Cleaning

Refer again to Figure 5-4. Total cleaning requires a small Phillips screwdriver, clean Kimwipes®, isopropyl alcohol, and a clean plastic forceps. Carefully pull the cell end caps (E) off the cell (F), using a twisting motion, next remove the Phillips screws (C) and the window caps (A). This will allow the sapphire windows to be removed. Grip the window (D) with the forceps or wear powder free gloves when cleaning. Squirt the window with alcohol or use an alcohol wetted foam swab, then rub the surface of the window clean with a Kimwipes®. Rotate the forceps to a different position on the window and repeat the cleaning. Blow-dry with clean UHP Nitrogen or Argon.

Clean the entire end cap (E) including the O-ring (B) and gas ports with alcohol. Do not handle the cleaned parts with your fingers; use clean forceps or powder free gloves. It will be necessary to blow dry the end cap; gas orifice and fitting with clean gas before assembly. Be sure not to touch the windows after cleaning.

5.8 Cell Assembly

Reassemble the sapphire window (D), O-ring (B), and a window retainer (A) onto the cell end cap (E) with three flat-head Phillips screws (C), as shown in Figure 5-4. Be sure not to touch the clean O-ring (B). Handle it instead with clean forceps.

Grip the cell (F) near one end and insert the cell into the open end of the cell-end cap (E) with a pushing twisting motion. From Figure 5-5, which shows the “open” end, note that each cell end cap has two imbedded O-rings (A, B). Firmly push (with twisting motion) the cell into the open end of the cell end cap and continue pushing until both O-rings (A, B) are fully engaged.
Inspect the assembled cell to determine that both O-rings (A, B) are fully engaged as shown in Figure 5-6. In Figure 5-6, shown without the window and cell cap, area “C” reveals no O-ring gap. This indicates that both O-rings A and B of Figure 5-5 are fully engaged. Figure 5-7, shown with window and cell cap, also reveals no O-ring gap at point C, (the boundary between the cell end (E) and the cell end cap.)

Figure 5-5 Open end of cell end cap. A - O-ring   B - O-ring

Figure 5-6 Engaged O-ring. Shown without the window and cell cap. C - No gap visible
Figure 5-7. Engaged O-ring. Shown with the window and the cell cap.
C - No O-ring visible   D - Window O-ring   E - Cell

Note:
The O-ring “D” visible in Figure 5-7 seals against the cell window.

If an O-ring is NOT engaged, as in Figure 5-6, the O-ring “B” Figure 5-8 is visible in the gap immediately at the end of the glass cell “E” Figure 5-8. This should look, instead like region “C” in Figures 5-5 and Figure 5-7. If the O-rings are not engaged correctly (as in Figure 5-8), then the system may drift and perform poorly. Assemble and attach the remaining cell end cap to the other end of the glass cell.
An alternate means of checking complete engagement of all O-rings in both cells is to measure the overall length of the fully assembled cell with a ruler. If the overall assembled cell length is: 8 29/32 inches (226.5mm), then both O-rings are engaged; 8 31/32 inches (228mm) indicates one O-ring is not engaged; 9 1/32 inches (229.5mm) indicates that two O-rings are not engaged (one in each end).

**Note:**

The glass tubing is sufficiently thick-walled that there is almost no danger of breakage (provided you have gripped near the end being inserted). However, to error on the side of caution, grip the glass tube with a sufficient thickness of cloth or paper towel to protect your hands in the unlikely event of glass breakage. Never insert or try to use a cracked or chipped glass tube.
Once the cell has been completely assembled, with both O-rings fully engaged, place the cell on a flat surface with both cell end cap “flats” facing downward. Rotationally adjust the cells until both “flats” are flat against the surface and parallel with each other. Recheck O-ring engagement (as above) and re-inspect both windows under low-angle reflection illumination to verify that no residual dust, lint, fingerprints, or other smudges exist on the windows. If both windows are “clean”, attach the appropriate Viton® interconnect tubing and reinstall the cell into the oven.

To reinstall the clean (and/or re-tubed) sample cell, first check that the two cell holder "flats" are parallel to each other. A simple check will reveal both cell end cap flats to be completely “tight down” against a flat surface with no gap visible between the end cap and the flat surface, when parallel.

Finally, reconnect the tubing and the optical cabinet cover.
5.9 Cleaning the Gas-Liquid Separator

Periodically it will be necessary to clean the Gas-Liquid Separator (Figure 4-2). Try pumping 10% HNO₃ through the system for 30 minutes continuously, followed by a deionized water rinse. For more aggressive cleaning, disconnect all tubing from the GLS. Be careful not to pull hard on the tubing; this can break the glass side arms off. Instead, use a fingernail to gently work the tubing off the glass arms.

Refer to Figure 5-10. Loosen the white, plastic retainer screw, and carefully remove the GLS by rotating the vapor outlet to the front and slide the GLS down through the clamp and exiting at the clamp bottom.

Once the GLS is removed, place it in a beaker containing 50% HNO₃ v/v in DI water. If an ultrasonic bath is available, place the beaker in the bath, sonicate for 30 minutes followed by a repeat of the cleaning with fresh acid. Otherwise, let the GLS soak for two hours in the 50% HNO₃. If excessively dirty immerse the GLS in a mixture of 20 % nitric and
20\% sulfuric acid and heat on a hot plate for several hours or until clean.

Next, rinse with DI water and dry. Reassemble GLS as shown in section 5.11. Tighten the plastic screw finger tight only.

**WARNING**

Hot concentrated acids may cause severe burns, severe fume inhalation trauma and/or death. They should be handled only by professionally trained chemists, who employ proper safety precautions and equipment (hoods, goggles, gloves, tongs, etc.).

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### 5.10 Changing the External Gas Tubing

To do this, begin by pulling the instrument forward on the bench, and remove the optical front cabinet.

Next, replace the tubing as shown in Figure 5-11 and Figure 5-12. The replacement tubes, which are in the external tubing kit, are labeled and precut to length. See Figure 5-11 for label designations. Match these numbers and letters with Figure 5-12 and the existing tubing, to see where each labeled tube should go. It is best to replace one tube at a time.

In Figure 5-11 connect the number printed on each tube end label (See “Tubing Label Schedule”) to the same circled number in Figure 5-12 diagram. Refer to Figure 5-12 to see the exact tube routing.
Figure 5-11  Tubing diagram and connection table.
To remove an old tube from a plastic connector, grip it near its connector and pull firmly. For glassware, it is better to slit the old tube with a razor blade or sharp knife, before removing. Alternatively, you may use the edge of a fingernail to ease the tube off its glass arm.

A simple way to avoid making wrong connections is to remove only one tube at a time, and replace this tube with the appropriate, labeled new one, before proceeding to the next tube.

Drying cartridges are replaced as assemblies (replacements with tubing already attached).

![Figure 5-12 Tube routing.](image)

When finished, be sure no tubing is pinched when the covers are replaced.

Replace the Tygon® drain waste as described in Chapter 3 Installation.
Sample in and drain lines need to be replaced monthly. If needed an external tubing kit is available from CETAC with the correct labeled tubing included, pre-cut to correct length.

**Note:**

Do not use waste tubing other than that provided by CETAC. The ID and length (3 feet) of the drain tube are optimized for maximum system stability, and should not be altered. Other tubes are similarly optimized and substitutions/alterations should not be made.
5.11 Retubing the Gas-Liquid Separator

This procedure should be followed once a month, unless the samples are excessively "dirty", in which case the procedure should be followed more often, as needed.

5.11.1 GLS Inlet

Refer to Figure 5-13. Note tubing routing and then remove all tubing, drain sleeve (J), inlet capillary (C) and silicone sleeve (D) from the GLS using the same procedure as for Viton® tubing above.

Figure 5-13  Assembled Gas-Liquid Separator.

A  –  Liquid mix tube
B  –  Capillary heat-shrink
C  –  Teflon capillary
D  –  Silicon inlet sleeve
E  –  Sample inlet guide
F  –  Hg vapor outlet
G  –  Frosted center post
H  –  Carrier gas inlet
I  –  Teflon drain tubing
J  –  Silicon drain sleeve
Select a new translucent white silicone inlet sleeve (D), and push it down over the glass sample inlet guide (E) until ≈ 6mm (1/4 inch) of silicone tubing protrudes above the top of the glass inlet guide (E).

Select a replacement GLS Teflon inlet capillary assembly, (C). Carefully direct the capillary end of the insert into the top protruding end of the silicone tube (D), and GENTLY push "straight" down.

The capillary insert (C) should go down inside the inlet guide (E). Continue pushing gently downward until the exposed capillary end (C) protrudes below the glass guide (E). Stop pushing when the bottom edge of the capillary is about 0.5 mm (range of 0.3 - 0.6 mm) above the top of the GLS frosted center post (G). The two parts should never touch! Finally, select a replacement liquid mix tube (A). Carefully slide the end of the Viton® liquid inlet tube labeled "2 <- Liq. Mix <---" onto the protruding upper end (yellow heat-shrink, B) of the GLS inlet insert assembly. Continuously watch the lower end of the insert, to be sure that its position does not change. Finally, check that the lower end of the insert is still spaced 0.3 · 0.6 mm above the frosted center post (G).

Install the assembled GLS into its holder on the front of the analyzer and gently tighten the GLS screw clamp (Figure 5-10). Do not overtighten, or the GLS may crack. Connect the drain tube and route the drain and liquid mix tubes in their appropriate tubing clamps. The above procedure may be done with the GLS in its holder, if caution is used so the fragile glass ports are not broken.

5.11.2 GLS Drain

Select a new GLS drain tube. Prior to installation, the Teflon® drain tube should be pushed all the way into the silicone drain tube-sleeve. Do not kink the Teflon® drain tube. To install, line up the end of the assembly against the glass drain and push. Refer again to Figure 5-13. The silicone tube-sleeve (J) should automatically slide over the outside of the glass tube, and the Teflon® drain tube (I) should slide inside the glass drain tube as seen in Figure 5-13. Keep pushing until the Teflon® tube is approximately flush with the interior wall curvature of the GLS, or about 2 mm withdrawn from this point.
Inspect the drain area closely. If the liquid pulsates or segments immediately prior to the Teflon® drain tube end, then try a different position for this tube end. Try it further inserted (about 1 mm into the chamber), or try it with less insertion (several mm withdrawn from the chamber). Select the position that eliminates or minimizes segmentation and/or pulsation of the flow at the entry point of the Teflon® insert. A steady, high-speed stream of tiny bubbles should form within the Teflon® drain tube, and larger bubble segments should emerge from the remote end, where a longer silicone tube is attached.

5.12 Replacing the Nafion® Dryer Cartridge

Replace this cartridge if mercury absorbance diminishes to less than 50% of original value. Refer to Figure 5-11, Figure 5-12, Figure 5-13, and Figure 5-14, which show a replacement Nafion® cartridge and its installation.

1 Open the instrument front door for access to the dryer cartridge.

2 To remove the old Nafion® dryer cartridge, refer to Figure 5-11 and Figure 5-12. Disconnect the following tubes: “11>Hg Vapor>12” from the GLS arm (11), “>Sample Gas>14” from the bulkhead (14), and “<Dryer Supply<17”, from the bulkhead (17).

3 Carefully unhook the remaining Nafion® dryer tube, “20<Dryer Exhaust” from the clips on the side of the instrument. Detach the Nafion® dryer from the two black clamps (Figure 5-12); pull cartridge forward, and set aside.

4 Install a new Nafion® Dryer cartridge and reattach tubes described above. (When reconnecting Luer lock fittings, be careful not to kink the tubing, which could cause gas flow constriction.) Remember to route the tube 20 (“20<Dryer Exhaust) up through the cabinet cover cut out.
5.13 GLS Overflow Recovery

Observation of a full or overflowing GLS, absorbance over-range, loss of gas flow or liquid in the sample cell may indicate a GLS overflow accident. This could happen if the sample probe is left in the park position for an extended period and rinse siphons into the GLS. The sample probe, when not in use should not be submerged in the rinse station. In addition, during operation an overflow might occur. The latter could occur if the GLS drain outlet-tubing clogs from non-filtered digested samples during unattended operation. In addition, it may occur if the waste pump tubes fail, or the clamp tension is too loose and or not clamped. When any of the above symptoms occur, liquid may have overflowed the Nafion® dryer cartridge into the optical sample cell, and possibly beyond.

If the overflow is discovered quickly, it is possible that the Nafion® dryer can be cleaned and dried (See Step 11) before the membrane pores become saturated and enlarged, which render the dryer useless.

Follow the steps below to correct the problem and bring the instrument performance back to normal. Also, refer to section 5.7.2.
1 Shut off the instrument main power and unplug the QuickTrace™ M-6100.

2 Open the optical access panel. Inspect the sample cell (in place) and judge whether any liquid is likely to have passed through the sample cell to the "gas exhaust."

3 Remove the sample cell, GLS, and Nafion® cartridge (with all tubing still attached). Place all parts on a clean lab cloth (or equiv.) on the lab bench.

4 Dismantle the sample cell completely. See Section 5.7.2.

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**CAUTION**

Do NOT remove the cell window blocks over the optical rail if the cell is wet or full of water. Do this instead outside the instrument to avoid spillage onto the optical components.

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5 Dump out all water and brine from the sample cell glass tube.

6 Rinse the sample cell glass tube with deionized water and oven dry. Alternatively, dry by rinsing with alcohol (recommended spectrophotometric isopropyl alcohol (isopropanol)) and blowing dry with clean air or Nitrogen.

7 Rinse and dry all remaining cell holder parts, fittings and transfer tubing by steps similar to five and six. However, do not oven dry; use the alcohol rinse/blow-dry procedure, instead. Inspect closely to be sure all water, and/or all residual alcohol is completely eliminated from all fittings, tubes, parts, and gas ports.

8 Clean the sapphire window first with water and then as described in Section 5.7.2.

9 Reassemble the window and cell end caps. Handle the window with clean forceps or hold by the edge with fingertips while wearing cloves (verify cleanliness by inspection with low-angle room light reflection).
10 Install the glass tube into the cell end caps, and seat firmly to fully engage both O-rings.

11 For Nafion® dryer cartridges that have gotten wet, if not already disconnected, disconnect the cartridge “sample gas” from sample cell. Attach a 10mL Luer lock syringe filled with DI water to tube 14 and gently push the water through the Nafion® dryer cartridge. The water will exit through the cartridge at tube 11 (Figure 5-14, tube numbered 11). Hold tube 11 so it is positioned over an empty beaker (do not pull on tube 11 it may disconnect from the cartridge and render it useless). Repeat the flushing procedure again followed by a syringe filled with air, which will flush the water out of the Nafion®. Next, adjust carrier gas pressure to 10 psig. Reconnect tube 11 to the GLS. Turn on the gas and allow GLS and cartridge to blow dry for one hour with flowing gas. Be sure to engage all peristaltic pump tube clamps before initiating gas flow (otherwise the gases will leak out the pump tubing and bypass the Nafion® dryer cartridge).

12 **Turn off gas!** Reattach cartridge sample outlet tube to the sample cell and reinstall the sample cell into the QuickTrace™ M-6100, close front door, and reattach all tubes except for the final tube end labeled “20Å GAS EXHAUSTÅ”.

**Note:**

More than likely, water saturation of the Nafion® dryer will destroy it, making replacement necessary. However, if the overflow accident is quickly caught, cleaning and drying the Nafion® membrane immediately (per above procedures) may save it.

13 Determine whether any rinse solution (acidic stannous chloride dissolved in water) got past the sample cell (during the original accident), and into any portion of the remaining gas exhaust lines and KMnO₄ trap. Determine this by dismantling all fittings "en route" and inspecting for the presence of any liquid or salt encrustation in any of the fittings or tube ends. Make
sure to check the dark purple potassium permanganate powder to see if it is wet and/or no longer "free flowing" in any part of the trap tube.

14 If no undesirable conditions are found in the above plumbing inspection, reconnect all the system plumbing and check the gas flow. Be sure all peristaltic pump tubes are engaged by their clamps before checking gas flow (otherwise the gases will leak out the pump tubing and bypass the flow meter).

15 If ANY undesirable condition was found during the above plumbing inspection proceed to the next step. Otherwise, skip to step 25.

16 Remove the potassium permanganate Mercury trap from the rear of the instrument. If it is completely dry, simply set it aside. If the vapor trap is wet, empty the KMnO₄ and dismantle the trap. Remove the glass wool plugs; rinse all parts, fittings and tubes with deionized water and then Hydroxylamine Hydrochloride. The Hydroxylamine Hydrochloride will clean any remaining purple color from the vapor trap. Dry by means of rinsing with alcohol and blowing dry with clean air or nitrogen. Reinstall loose glass wool plugs into the endcaps, install one cap, and refill tube body with potassium permanganate powder (crystals). Install the remaining end cap and set the trap aside. Do not reinstall the permanganate trap on the QuickTrace™ M-6100 instrument at this time.

17 It is still necessary to rinse residual acidic stannous chloride brine out of remaining internal gas exhaust tubing. Refer to Figures 5-9 and Figure 5-12. Locate the sample cell exit. Disconnect the exhaust tube at the sample cell.

18 Disconnect the “liquid mix” at the tee on the output side of the peristaltic pump. Connect the exhaust tube to this tee. Alternately, a syringe filled with water could be connected the sample exhaust tube for the water flush.
19 Place a waste receptacle (>100 mL) immediately under the "gas exhaust" fitting on the rear of the instrument. Alternatively, use the appropriate Luer fittings and hook another transfer tube from the rear gas exhaust fitting to the drain bottle on the floor.

20 Pump 100mL of deionized water through the exhaust line, until it all passes through to the waste collection receptacle. Refer to the *QuickTrace™ M-6100 Mercury Analyzer Software Manual* for use of the QuickTrace™ M-6100 instrument controls to place the autosampler sample probe in 50mL tubes of deionized water located in standard positions. This will wash all residual perchlorate salt encrustation and/or acidic stannous chloride brine out of the internal gas exhaust lines and fittings.

21 Using QuickTrace™ M-6100 instrument controls, lift the autosampler sipper tube out of the deionized water tube and allow the peristaltic pump to push air through the exhaust tube, until no more water enters the waste receptacle.

22 Reconnect the exhaust tube to the sample cell exit. Reconnect the liquid mix line to the tee on the outlet side of peristaltic pump channels three and four.

23 Reinstall all covers and close all doors.

24 Initiate a reasonable gas flow, then pump rinse solution through the GLS continuously, and let the system "purge,” "dry” and thermally stabilize for a period of 90 minutes.

25 Reinstall the permanganate trap onto the back of the instrument.

26 Operate instrument normally.
5.14 Replacing the Hg Lamp Bulb

The effect of lamp current on data quality (absorbance and noise) is minimal over the range 0.6 – 1.5 VDC. When a lamp is new, the normal operating lamp current is 0.65 – .75 VDC. As the lamp ages, the lamp current will automatically adjust to maintain constant emission intensity reaching the EOFM filter/detector.

Use a voltmeter to check the lamp current. Wait until the lamp current reaches 1.5 VDC to order a new lamp. At this point, use a dentist mirror and flashlight to check that the EOFM filter is not "smudged" Refer to Figure 5-15). If it’s clean, order a replacement lamp. If the EOFM filter is dirty, clean in place using the cell cleaning procedure. After cleaning, recheck the lamp current. Install the new lamp when the current of the old lamp reaches 1.8 VDC, or if you need greater absorbance sensitivity than the old “high current” lamp can provide.

To change the bulb, turn off the main power, unplug the QuickTrace™ M-6100 completely. Disconnect all power and communication cables and
cords to the autosampler. Remove the autosampler from atop the QuickTrace™ M-6100 and set aside. Remove the cabinet screws from the electrical cabinet cover of the QuickTrace™ M-6100 and remove the cover. Allow the instrument to cool five minutes. Locate the "heavy" yellow/orange colored lamp cord, on the left-hand side of the cabinet interior. Trace the cord backward and unplug it from the lamp controller board. On the top surface of the lamp block, push the edge of the gray foam toward the left side of the instrument. Under the edge of this foam is a small "Allen" set screw. Insert a 0.050 inch Allen wrench into the setscrew head and loosen the screw.

Grab the old bulb where it attaches to its yellow cord and pull it towards the right. The bulb will slide out easily. Clean the new bulb by wiping clean with a Kimwipes® or optical tissue moistened with high purity (spectrophotometric grade) isopropanol, and blow dry with argon gas. Don't touch the bulb face, once it is clean.

Holding it by the base, carefully insert the bulb into the lamp block until it stops. Rotate the bulb base until the scratch mark scribed on the lamp body faces upward and lines up exactly with a matching scratch mark or setscrew on the lamp block housing. This scratch mark may be an arrow or a date on the yellow power cord. Hold this position carefully while tightening the Allen set screw. Check that the two scratch marks match up after the setscrew has been tightened. Plug the yellow lamp cord into the lamp controller board.

Turn on the unit; allow a 15-minute warm-up. Check the lamp current. If the lamp current is in the range of 0.6 – 0.85 VDC, operate the instrument normally. If the current is not in the range 0.6 – 0.85 VDC an adjustment is necessary to get the longest possible life cycle from your new CETAC lamp.

The lamp current can be adjusted using the potentiometer located on back of the lamp manager.
1. Attach voltmeter to pins 6 & 7 on the auxiliary connector on the back of the M-6100. Attach the positive to pin #6 and the ground to pin #7 as shown in Figure 5-16:

![Figure 5-16](image1.png)

*Figure 5-16* Checking lamp current with voltmeter on auxiliary pins 6 & 7.

2. Use a 5/16 nut driver to remove the 4 nuts from the lamp manager while holding each screws with a #2 Phillips as shown in Figure 5-17:

![Figure 5-17](image2.png)

*Figure 5-17* Removing lamp manager.
3 Use a jewelers standard screwdriver to adjust the lamp current (LAMP INTENSITY) to read .700 volts DC as shown in Figure 5-18.

![Image of lamp manager voltage control](image)

**Figure 5-18** Adjustment of lamp manager's voltage control.

4 Clock-Wise will increase the voltage and counter Clock-Wise will decrease the voltage.

5 Rotate the potentiometer fully Clock-Wise until a faint clicking sound is heard.

6 Rotate the potentiometer counter Clock-Wise until a faint clicking sound is heard. Both of the LED lights on the lamp manager will be out at this time and the lamp manager potentiometer is now ready for the final adjustment.

7 Rotate the potentiometer slowly until the current reads 0.7 volts DC.

8 Let the voltage stabilize and make fine adjustments until the system stabilizes between 0.65 and 0.75 volts DC.

You have completed the adjustment of the M-6100 lamp manager after installation of a new mercury lamp. Reassemble the system covers and cables and operate the system normally.
Troubleshooting the QuickTrace™ M-6100
6 Trouble-shooting the QuickTrace™ M-6100

6.1 Cannot Zero Instrument

Perform the following steps:

- Check that the Hg lamp is on in the software and that the main power switch on the back of the instrument is in the on position (up). Verify the yellow light on the front panel is on.

- Be sure instrument is fully warmed up. 15 minutes is long enough.

- Check that both cells (sample & reference) and cell end caps are clean and "dry" (no liquid or dried stannous chloride obstructing the gas flow or the optical beam. If so, see Section 5.7).

- Check that both cell windows are clean (See Section 5.6.2).

6.2 Drifting Baseline

The system might not be thermally stable because of insufficient warm up time. Wait longer.

- Check that gas pressures are stable and correct. Variable gas flow can cause the baseline to drift. Check that no stannous chloride encrustation exists in gas lines/fittings after a GLS overflow accident. See Section 5.6.2.

- Check that the gas hoses are not pinched.

- Check that the lamp block heater works. The lamp block should be hot (56°C) to touch. Turn the lamp power off. Remove lamp-
housing cover and touch the lamp block momentarily to verify that it is hot.

- With the Hg lamp off, check that the EOFM filter is not dirty (inspect it with a dentist’s mirror and low angle flashlight).

- Check that the lamp current is not too high using the QuickTrace™ M-6100 instrument controls. High current indicates a worn-out lamp, if all the windows and optics are clean.

- Replace the Nafion® dryer cartridge.

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**6.3 Low Absorbence or No Mercury Response**

Ensure the Hg lamp is on.

Check all liquid uptake rates and gas flow. If there is no liquid or gas flow, see Section 6.4 below.

- Check that the reagent tube is in the reagent bottle.

- Check that SnCl₂ is active, not empty, not oxidized or precipitated.

- Check that standards have the correct Hg concentrations in them.

- Check the liquid lines for kinks or clogs.

- Check that standards have 7% HCl in them.

- Check the gas flow at the GLS outlet.

- Check the gas flow at the sample cell outlet.

- Check that the gas flow at the KMnO₄ trap outlet; does not drop in pressure or flow, this indicates an upstream block or a leak.

- Check all plumbing connections for correct location and proper seal.
Troubleshooting the QuickTrace™ M-6100

- Replace the Nafion® dryer cartridge.

- Mechanically block the sample beam optical path (e.g. with a business card) and see if absorbence goes full scale (≥ 1,000,000 μabs).

- Reboot the system: shut down the software, and power down the QuickTrace™ M-6100 and Autosampler. Restart, and check the signal.

6.4 No Liquid or Gas Flow

6.4.1 If No Sample or Rinse Flow

- Increase the tension on the sample pump tubing to start flow.

- Be sure the sample, SnCl₂, and “Liq. Mix” tubes are not pinched off anywhere and restricting flow.

- Make sure neither tube is pinched under the autosampler foot.

- Ensure the rinse station is filled with acidified rinse.

- Ensure all pump tubing is centered in clamps.

- Check for clogs in sample tubing.

- Check for kinks in the Autosampler sample probe and sipper tube.

- Check for excessive pump tubing wear. Replace if needed.
6.4.2 If No SnCl₂ Flow

- Increase tension on the reagent pump tubing to start flow.

- Check to see that no precipitate has formed and clogged the reagent sipper and/or pump tubing.

- Check for excessive pump tubing wear. Replace if needed.

6.4.3 If No Drain Flow

- Increase tension on the drain pump tubing to start flow.

- Check that there is no clogging of the drain outlet tubing of the Gas-Liquid Separator. If clogged, clean or replace the drain outlet tubing.

- Be sure the drain tube is not pinched off and restricting flow. Ensure it is not pinched under the autosampler foot.

- Check that the vent port on the waste bottle is open, and that the bottle is not overflowing.

6.4.4 No Gas Flow or Low Gas Flow

- Check that the in-line gas filter is not clogged. Remove the threaded connection downstream from the filter and check for gas flow at the filter outlet.

- Check that all gas supply lines are connected correctly.

- Check that no gas tube is kinked or pinched.

- Be sure that the KMnO₄ trap is not packed too tightly (with either the glass wool plugs or the reagent crystals) and restricting flow. Repack if too tight.
Check for leaks/clogs throughout the gas system, especially after a GLS overflow accident. Check flow after each fitting/component to isolate the bad section.

6.5 Double Peak with Low Absorbence

This may indicate a problem with not enough (or none at all) reagent (stannous chloride) uptake. Check the following items:

- The reagent sipper tube is in the reagent bottle (rather than sitting in a deionized water container or loose in air).
- There is liquid in the reagent bottle.
- The sipper tube is submerged below the liquid level.
- The liquid is 10% stannous chloride solution in 7% HCl.
- The reagent is not "old", precipitated, yellowed, or otherwise oxidized (for example, by leaving the bottle open overnight).
- There is no clog, kink, pinch, or other obstruction in the reagent-tubing pathway.
- The reagent liquid uptake rate is at least 1.5 mL/min.
- The Autosampler sipper tube is the right size, no longer than 3.5 feet and no smaller than 1.0mm i.d.
- The sample uptake is at least 4.5 mL/min.
- The Autosampler probe, reagent sipper tube, QuickTrace™ M-6100 mixing tee and GLS liquid/mix capillary inlet is not under pressure from a partial clog.
6.6 Poor Reproducibility

- Always be sure to matrix match standards and samples as closely as possible (excluding the 7% HCl in the standards), and rinse solution should also be acidified.

- Inspect the liquid flow into and out of the Gas-Liquid Separator. If either the sample in or waste out is pulsing, adjust the clamp tension on the corresponding tubing in the peristaltic pump to smooth out flows. If unable to stop the pulsing, check to see if the pump tubing is worn out. If so, replace the pump tubing. Be sure to check all the pump tubes.

- Ensure the center post is fully “wet.” If partially dry anywhere on post surface, wet the post. See Section 4.

- Check to see if the reagent tube is in the reagent bottle.

- Ensure that the stannous chloride has not been emptied or oxidized. Old SnCl₂ can lead to poor results. Replace if yellow, precipitated, or just too old.

- Ensure that the Autosampler rinse station and rinse bottle are filled with acidified rinse.

- Inspect both cell windows for fingerprints, films, or debris. If dirty, clean the windows following the procedure outlined in Section 5.7.

- Make sure gas pressure to the QuickTrace™ M-6100 is 20-100 psig.

- Check the output gas flow after the KMnO₄ gas trap with a flow meter (to check this flow, all pump tubes must be clamped or plugged). This gas flow should be the same as set in the software. Check all the seals and recalibrate if necessary. Note calibration and flow stability.

- Check the gas flow at the GLS exit.
QuickTrace™ M-6100 Mercury Analyzer Operator’s Manual

Troubleshooting the QuickTrace™ M-6100

• Check the gas flow at the sample cell exit.

• Check that the optimal instrument settings are employed. See the QuickTrace™ M-6100 help file, chapter four of this manual for and the QuickTrace™ M-6100 Mercury Analyzer Software Manual for more details.

• Check that the peristaltic pump rollers are not severely worn. Inspect all rollers with tubing removed. Roller facets should not be “grooved.” All rollers should spin freely when turned by sliding your thumb quickly across them. None should feel “gritty” or slow in spinning. Replace the head if any one of the 12 rollers are grooved or fail to move freely.

• Ensure the baseline is not drifting severely (See Section 6.2).

• Check that the raw analog system noise is ≤ 400 μAbs peak to peak. If not, call CETAC Support.

6.7 Noisy Baseline

• Check that flows into and out of the Gas-Liquid Separator are not pulsing. Pulsation indicates improperly adjusted pump clamps.

• Make sure the gas pressures are correct.

• Be sure the SnCl₂ is fresh and not oxidized or precipitated.

• Ensure the cell windows are clean.

• Check that the EOFM filter is clean. Turn the Hg lamp off and the EOFM filter is not dirty (inspect it with a dentist’s mirror and low angle flashlight).

• Check that nothing has been spilled on the binocular camera lenses. Turn the Hg lamp off and the camera lenses are not dirty (inspect
them with a dentist’s mirror and low angle flashlight. Call CETAC Customer Service and Support if the camera lenses are dirty.

- Ensure the lamp current is not excessive (>15 mA). Do this with the QuickTrace™ M-6100 Software. For more information see Section 5.

### 6.8 Bad DL

- Check Low Absorbance. See Sections 6.3 and 6.5.

- Check noisy baseline. See Section 6.7.

### 6.9 Sudden Standard Absorbence Rise During Run

- Ensure the rinse bottle has acidified rinse.

### 6.10 Poor Accuracy

- Verify good reproducibility (e.g. ~1 % RSD on standard replicates).

- If reproducibility is poor, see Section 6.6.

- Be sure the standards contain 7% HCl (v/v).

- Be sure the samples are properly digested.

- Utilize an appropriate process standard to validate digestion and container storage.

- Check process (digestion blanks, containers, and rinse solution) for Mercury contamination.
Troubleshooting the QuickTrace™ M-6100

- Check standard solution accuracy, and all gravimetric/volumetric process steps and equipment for accuracy and calibration.

- If very low samples are run immediately following high samples or standards, the rinse time may not have been long enough and the result may be reading low. (Increase rinse times when sample and/or standard concentrations are widely spread).

- Be sure that the rinse solution contains at least 1% HCl / 1% HNO₃. If it only contains deionized water, very low samples (acidified) may read erroneously high if they immediately follow the high standard or a high sample, regardless of allocated deionized water rinse time. The problem is avoided by a acidified rinse solution in the rinse solution bottle.
Spare Parts
## Spare Parts

<table>
<thead>
<tr>
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<th>DESCRIPTION</th>
</tr>
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<td>SP5595</td>
<td>BOTTLE KIT, RINSE; M6000, M6100, M7500, M8000</td>
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<tr>
<td>SP5594</td>
<td>BOTTLE KIT, STANNOUS CHLORIDE REAGENT; M6000, M6100, M7500, M8000</td>
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<td>Part No.</td>
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<td>SP5593</td>
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<td>SP8070</td>
<td>CABLE Y, PUMP INTERFACE; M6100 /ASX-400</td>
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<td>CABLE Y, PUMP INTERFACE; M6100 /ASX-130, 260, 520</td>
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<td>CABLE, RS232 SERIAL</td>
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<td>CAMERA 254 Hg FILTERS (SET OF 2); M6000, M6100, M7500</td>
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<td>CAMERA 50mm FOCAL LENS (SET OF 2); M6000, M6100, M7500</td>
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<td>CAMERA BLOCK; M6000, M6100, M7500</td>
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<td>CELL O-RING KIT</td>
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<td>SP6050</td>
<td>DRYER MOUNTS, SPRING CLIPS KIT</td>
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<td>SP5615</td>
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<td>GLS DRAIN TUBE (PACK OF 3); M6000, M6100, M7500, M8000</td>
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<td>ISA INTERFACE BOARD; M6100</td>
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<td>SP5614</td>
<td>MERCURY LAMP HOUSING ASSEMBLY; M6000, M6100, M7500</td>
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<td>PERISTALTIC PUMP MOTOR DRIVE COUPLER</td>
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<td>PERISTALTIC PUMP</td>
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<td>SP5705B</td>
<td>PUMP TUBING, STANNOUS CHLORIDE REAGENT TUBING, BLK-BLK, (PACK OF 12)</td>
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<td>SP6033</td>
<td>SAMPLE PROBE, 1.0MM I.D.; M6000, M6100, M7500, M8000</td>
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<td><img src="image3.png" alt="Image of SAMPLE PROBE, 1.0MM I.D." /></td>
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<td>SCSI INTERNAL RIBBON CABLE ADAPTER; M6100</td>
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<td>SP5558</td>
<td>SWABS, FOAM TIP</td>
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<td>SP8024</td>
<td>TIMING BOARD; M6100</td>
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<th>Description</th>
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<td><img src="image2.png" alt="Waste Tubing" /></td>
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Glossary

This manual frequently uses the following terms:

A Amperes, electrical current
AAS Atomic Absorption Spectrometry
Abs Absorbance (-\log_{10} T or 2-\log_{10} %T)
ADC or A/D Analog-to-digital converter
ADX-500 Optional Autodilutor accessory
ASX-500 The ASX-500, Model 510 Autosampler
Bar Unit of pressure. 1 bar = 100 kPa ≈ 14.5 psi
Ar Argon carrier gas, chemical formula
CH$_3$HgCl Methyl mercuric chloride (or “methyl mercury”), chemical formula of a common organo-mercurial
CLP Contract Laboratory Protocol (analysis protocol of U.S. EPA)
cm Centimeter (10$^{-2}$ meter), unit of length
Cold Vapor Direct Atomic Absorption Spectrometric analysis (at 253.652 nm) of “head-space” gas from a stannous chloride or stannous sulfate reactor using neither flame, nor plasma, nor furnace nor any other electro-thermal atomizer. CVAAS works only for the element mercury (Hg)
DBA Doing Business As
Dia. Diameter
DL  Detection limit. Smallest statistically detectable concentration, where the absorbance, Abs (produced by that concentration), equals 3 times the standard deviation $\sigma$ of the blank Abs

DSP  Digital Signal Processor

each  Each

EOFM  Electro-Optic Feedback Module; used to stabilize the Hg lamp

EPA  U.S. Environmental Protection Agency

EPA-245.1  The standard EPA method of water quality analysis for measuring mercury (Hg)

ETFE  Ethylenetetrafluoroethylene (Tefzel), a polymeric tubing material

g  Gram, unit of mass or “weight”

GCU  Gas Control Unit, sets and regulates carrier gas flow rate

GLS  Gas-Liquid Separator

HCl  Hydrochloric Acid, chemical formula

Hg  Mercury, chemical symbol

Hg$^0$  Mercury, elemental (reduced) state

Hg$^{2+}$  Mercuric ion, mercury in $+2$ (oxidized) state, typically HgCl$_2$

HgCl$_2$  Mercuric chloride, chemical formula

HNO$_3$  Nitric acid, chemical formula

i.d.  Inside diameter
**Glossary**

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Definition</th>
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<tr>
<td>IDL</td>
<td>Instrument Detection Limit. DL in ultra-clean, high purity acid media (e.g. 7% HCl, “Ultrex II” grade). IDL is generally measured under “favorable” operating conditions and does not involve sample digestion or preparation steps. IDL indicates what the instrument is capable of doing, if not subjected to contamination, digestion loss, storage loss, or other sample collection/preparation errors or limitations</td>
</tr>
<tr>
<td>KMnO₄</td>
<td>Potassium permanganate, chemical formula of oxidizing reagent, and Mercury Exhaust Trap agent</td>
</tr>
<tr>
<td>L</td>
<td>Liter, unit of volume</td>
</tr>
<tr>
<td>LED</td>
<td>Light-Emitting Diode</td>
</tr>
<tr>
<td>QuickTrace™</td>
<td>Specifically the CETAC Mercury Analyzer instrument that sits below the ASX-500 Autosampler</td>
</tr>
<tr>
<td>QuickTrace™</td>
<td>Specifically the entire Mercury Analyzer system including the QuickTrace™, ASX-500, peristaltic pump, etc.</td>
</tr>
<tr>
<td>mA</td>
<td>Milliamperes (10⁻³ amperes), electrical current</td>
</tr>
<tr>
<td><strong>MDL</strong></td>
<td>Method Detection Limit; DL measured under actual reagent purity, sample preparation, and storage conditions for samples, reagents, and containers in question. Calibration standards are generally prepared in the sample media and are carried through all sample digestion/preparation, storage and transfer steps, etc., as are samples. In the presence of significant contamination, small concentration detectability gets worse and the actual MDL should be redefined as 1/3 the contamination, but <strong>not less than the statistical MDL</strong>!</td>
</tr>
<tr>
<td><strong>mL</strong></td>
<td>Milliliter (cubic centimeter, cc, $10^{-3}$ L), unit of volume</td>
</tr>
<tr>
<td><strong>mm</strong></td>
<td>Millimeter ($10^{-3}$ meter), unit of length</td>
</tr>
<tr>
<td><strong>MSDS</strong></td>
<td>Material Safety Data Sheet specifying chemical hazard type and level</td>
</tr>
<tr>
<td><strong>N₂</strong></td>
<td>Nitrogen carrier gas, chemical formula</td>
</tr>
<tr>
<td><strong>Nafion</strong>®</td>
<td>Registered trademark of DuPont (E.I. du Pont de Nemours and Company), which trademark is licensed to Perma Pure, Inc. and used (with permission) herein by SD Acquisition, Inc. DBA CETAC Technologies. The trademark is descriptive of DuPont/Perma-Pure’s porous membrane which passes water vapor, but not Hg vapor.</td>
</tr>
<tr>
<td><strong>nm</strong></td>
<td>Nanometer ($10^{-9}$ meter), wavelength unit.</td>
</tr>
<tr>
<td><strong>ng</strong></td>
<td>Nanogram ($10^{-9}$ gram), mass or weight unit</td>
</tr>
<tr>
<td><strong>o.d.</strong></td>
<td>Outside diameter</td>
</tr>
<tr>
<td><strong>P</strong></td>
<td>Transmitted radiant power, photon flux at sample detector (after passing through sample)</td>
</tr>
<tr>
<td>Term</td>
<td>Description</td>
</tr>
<tr>
<td>----------</td>
<td>-------------------------------------------------------------------------------------------------</td>
</tr>
<tr>
<td>( P_0 )</td>
<td>Incident radiant power, photon flux at reference detector (before passing through sample)</td>
</tr>
<tr>
<td>PC</td>
<td>Personal Computer</td>
</tr>
<tr>
<td>PEEK</td>
<td>Polyetheretherketone; a machined polymeric construction material</td>
</tr>
<tr>
<td>pg</td>
<td>Picograms (( 10^{-12} ) g), mass or weight unit</td>
</tr>
<tr>
<td>PID</td>
<td>Proportional Integral Differential. Description of a type of precision heater control device</td>
</tr>
<tr>
<td>ppb</td>
<td>Parts per billion (( \text{ng/mL, } 10^{-9} \text{ g/mL, } \mu\text{g/L, } 10^{-6} \text{ g/mL} )), concentration unit</td>
</tr>
<tr>
<td>ppm</td>
<td>Parts per million (( \mu\text{g/mL, } 10^{-6} \text{ g/mL, } \text{mg/L, } 10^{-3} \text{ g/L} )), concentration unit</td>
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<tr>
<td>ppt</td>
<td>Parts per trillion (( \text{pg/mL, } 10^{-12} \text{ g/mL, } \text{ng/L, } 10^{-9} \text{ g/L} )), concentration unit</td>
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<tr>
<td>psi</td>
<td>Pounds per square inch. Pressure. 1 psi ( \approx ) 0.068 bar. 1 bar = 100 kPa</td>
</tr>
<tr>
<td>psig</td>
<td>Pounds per square inch. Gauge reading (above atmospheric pressure)</td>
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<tr>
<td>PTU</td>
<td>Precision-Timed Uptake</td>
</tr>
<tr>
<td>Pump or PP</td>
<td>Peristaltic Pump</td>
</tr>
<tr>
<td>P-P</td>
<td>Peak to Peak. A description of how signal noise is measured (One method)</td>
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<tr>
<td>RMS</td>
<td>Root Mean Square. A description of how signal noise is measured. RMS = 0.707 of peak amplitude (another method), approximately one standard deviation unit</td>
</tr>
<tr>
<td>RSD</td>
<td>Relative Standard Deviation. A measure of data precision or reproducibility</td>
</tr>
<tr>
<td>SCR</td>
<td>Stannous Chloride Reactor</td>
</tr>
<tr>
<td>Term</td>
<td>Definition</td>
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<tr>
<td>------</td>
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</tr>
<tr>
<td>Sn</td>
<td>Tin, chemical symbol. Typically as SnCl₂ reagent</td>
</tr>
<tr>
<td>SnCl₂</td>
<td>Stannous chloride, chemical formula of reducing agent</td>
</tr>
<tr>
<td>SRM</td>
<td>Standard Reference Material, containing a certified, known mercury level</td>
</tr>
<tr>
<td>T</td>
<td>Transmittance (P/P₀), often %T or percent transmittance (P/P₀ x 100%)</td>
</tr>
<tr>
<td>TC</td>
<td>“To Contain” Designation of a type of volumetric flask calibrated to accurately contain a specified volume of liquid</td>
</tr>
<tr>
<td>TD</td>
<td>“To Deliver” Designation of a type of volumetric flask or pipet calibrated to accurately deliver a specified volume of liquid</td>
</tr>
<tr>
<td>UHP</td>
<td>Ultra High Purity</td>
</tr>
<tr>
<td>UV</td>
<td>Ultraviolet; short wavelength region of spectrum below 370 nm (e.g. 253.7 nm)</td>
</tr>
<tr>
<td>VAC</td>
<td>Volts Alternating Current</td>
</tr>
<tr>
<td>VDC</td>
<td>Volts Direct Current</td>
</tr>
<tr>
<td>XS</td>
<td>A substantial concentration “excess” of one chemical reactant (over another)</td>
</tr>
<tr>
<td>μg</td>
<td>Micro-gram (10⁻⁶ g), unit of mass or weight</td>
</tr>
<tr>
<td>μL</td>
<td>Micro-liter (10⁻⁶ L), unit of volume</td>
</tr>
<tr>
<td>μAbs</td>
<td>Micro-absorbence units. (10⁻⁶ Abs)</td>
</tr>
</tbody>
</table>