

SIS AP2000 AutoProbe™ Manual

An Automated Direct Exposure Probe (DEP) for the Thermo Finnigan PolarisQ™ and DSQ™ Mass Spectrometers



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Publication No. 886000M – Document Revision 2.0.0 (February 2004)

Covering the AutoProbe 1.0, Cycle Composer 1.5, PAL Driver for Xcalibur 1.3.1, Xcalibur 1.1/1.2/1.3/1.4, Open Access 1.1/1.3, Thermo Finnigan PolarisQ/DSQ.

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A. Introduction



Safety Information and Warnings

Improper use of the AutoProbe system may result in equipment damage or personal injury. The operator must thoroughly understand the material in this manual before using the AutoProbe system.

The following warning symbols are used in this manual to alert the reader of procedures that if not adhered to or correctly performed are most likely to result in damage to equipment or personal injury:



Warning: Important Point



Warning: Electrical Hazard



Warning: Burn Hazard

The following safety precautions must be adhered to for the safe operation of the AutoProbe Console and the AutoProbe System.



The AutoProbe Console must be connected to a power source with a protective earth contact, and the AutoProbe must share a common ground with the Thermo Finnigan PolarisQ or DSQ Mass Spectrometers. Shock, fire, or instrument damage may occur if the AutoProbe System is connected to a power source that is not equipped with a protective earth ground contact.



Use only fuses with the required current rating and of the specified type for replacement. Shock, fire, or instrument damage may occur if fuse holders are short-circuited or incorrect or makeshift fuses are used.



The AutoProbe Console should be opened and serviced only by a trained and qualified service technician. The power source to the AutoProbe Console must be electrically disconnected before the AutoProbe Console is opened. Removal of AutoProbe Console covers may expose dangers due to hazardous voltages, excessive temperatures, and moving parts.



Do not operate the AutoProbe System with AutoProbe Console panels removed except as required during instrument calibration (page 43). The AutoProbe Console is equipped with electric switches that prevent probe movement and heating when the panels are removed. Do not short out or bypass these switches.



The AutoProbe filament tip is extremely hot under normal operating conditions. Keep hands and fingers outside of the AutoProbe Console. The probe tips can become red hot, even when outside of the vacuum system. Exercise caution at all times while using the system.



Compressed air is required for the operation of the air-powered cylinders inside of the AutoProbe Console. 60 psi of pressure is recommended for optimal performance. **In NO case shall the air pressure exceed 100 psi.**



The AutoProbe system is designed to operate with the Thermo Finnigan PolarisQ and DSQ MS in conjunction with the Thermo Finnigan Xcalibur™ software. It is not intended to operate on other manufacturers' instruments or software.



If the equipment is used in a manner not described in this manual, the safety protection provided by the equipment may be impaired.



General Description of the AP2000 AutoProbe

The SIS Model AP2000 AutoProbe™ provides fast, automated, multi-sample introduction of liquid samples into the Thermo Finnigan PolarisQ and DSQ MS via the direct exposure probe (DEP) technique, also known as desorption chemical ionization (DEP). The AutoProbe is fully integrated with the Thermo Finnigan Xcalibur software environment: AutoProbe system setup, methods setup, system operation, and data storage are seamlessly integrated into the data system.



Figure 1 - AutoProbe attached to the Thermo Finnigan PolarisQ MS

The DEP or DCI technique is used for the analysis of samples that are not volatile enough to be introduced into the mass spectrometer via a GC and therefore must be introduced directly into the mass spectrometer source. This technique is known as a “soft” ionization technique because thermally labile samples can be ionized with less fragmentation compared to conventional forms of EI (Electron Impact) mass spec analysis. The technique is normally used for single component samples since component resolution is low; samples with two or more components are difficult to interpret. However, sample analysis with the DEP technique is fast: 1 to 5 minutes per sample.

Frequently DEP sample analysis is performed utilizing CI (Chemical Ionization) mass spectrometer techniques, which produces less fragmentation in the mass spectrometer. The increased abundance of a molecular ion and reduced fragmentation in the CI techniques permits the analysis and interpretation of mixtures.

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The Direct Exposure Probe (DEP) on the AutoProbe uses a platinum wire on the end of a probe for the analysis of samples. Samples for analysis are dissolved in a suitable solvent and 0.125 to 1.0 μL of the prepared sample solution is injected onto a small platinum wire loop on the tip of the probe. After the probe has been inserted into the mass spec source, the platinum wire is heated with a small current to desorb the sample into the mass spec source for analysis. This DEP probe filament can be set to a constant current or can be programmed in up to three ramp steps. This versatility of analysis enables the user to optimize the analysis depending on the sample being analyzed.



Figure 2 - DEP Probe Tip

The AutoProbe adds the capability of automatic sample loading and unattended analysis to the DEP probe technique with the inclusion of a CTC PAL Autosampler. A wide range of sample trays can be fitted to the CTC PAL Autosampler from which the samples can be loaded onto the DEP probe wire via a syringe. The PAL permits the accurate and reproducible delivery of 0.125 to 1.0 μL samples onto the DEP Probe wire and can clean the syringe between samples.

AutoProbe samples can be analyzed at the rate of 2.0 to 5.0 minutes per sample. Sample analysis time can vary, but a typical sample analysis time is composed of the steps listed below:

1. Position Probe for Sample Loading	15 seconds
2. Load Sample onto Probe Tip	30 seconds
3. Solvent Removal	10 seconds
4. Take probe through isolation valve and into MS source	35 seconds
5. DEP analysis of sample	45 seconds
6. Clean Probe tip with high temperature bakeout	15 seconds
7. Return Probe to position for next sample analysis	30 seconds
TOTAL TIME	180 seconds

Several of the step times can be increased or decreased depending on the samples being analyzed.



AutoProbe Console Description

The AutoProbe Console is packaged in a white plastic cabinet (Figure 1) that attaches to the PolarisQ or DSQ MS at the GC inlet port. Inside, there is a probe rod attached to a servo linear actuator that drives the probe rod through the isolation valve and into the mass spectrometer source (Figure 3). The DEP probe tip can easily be removed and replaced.

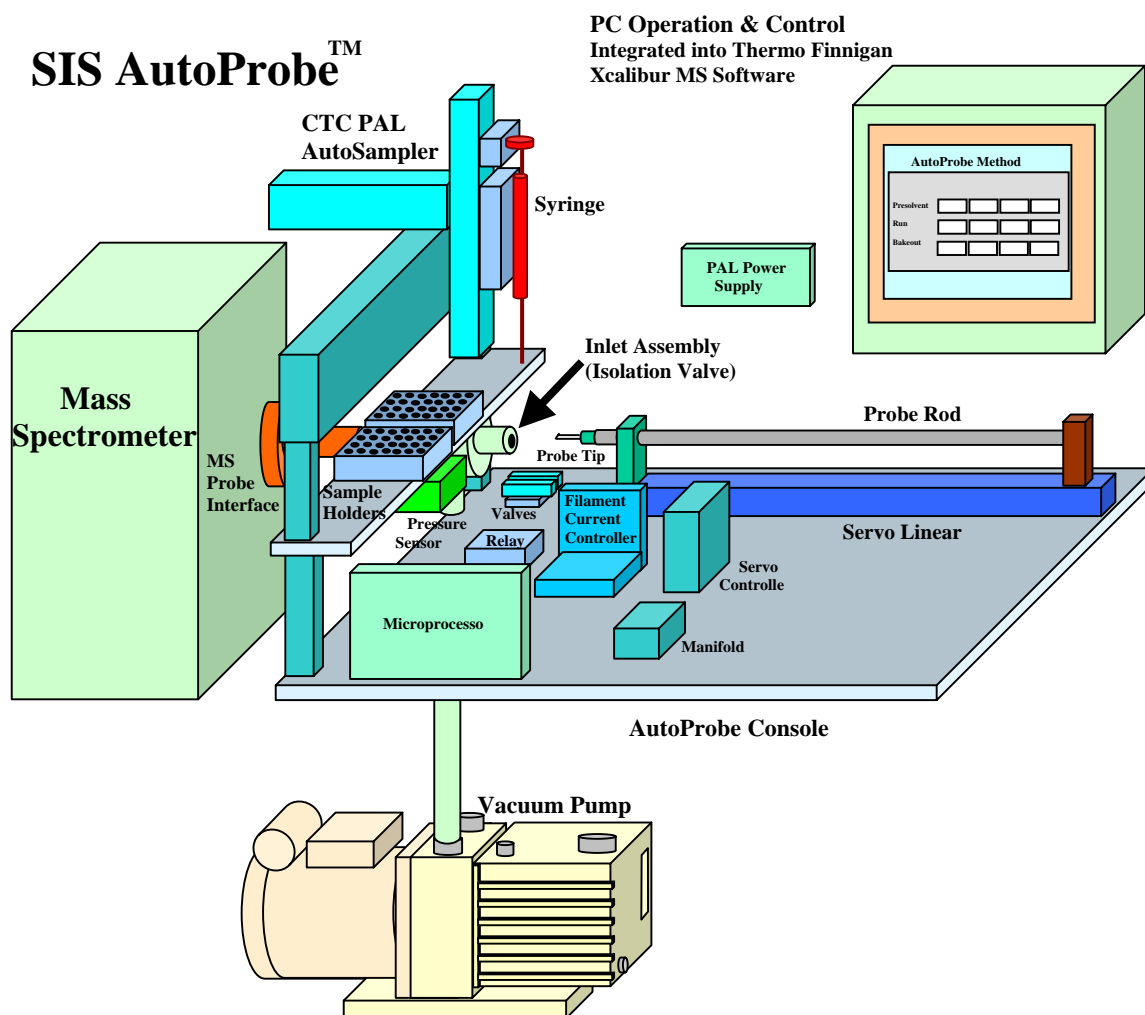


Figure 3 - Components of the AutoProbe Console

An isolation valve allows the probe to be introduced into the mass spectrometer vacuum system while still maintaining a good vacuum in the MS. When the probe is inserted into the first probe seal of this isolation valve, a vacuum valve opens so that the accessory vacuum pump evacuates the probe inlet. A vacuum gauge monitors this vacuum, and the probe is advanced to the second seal when the vacuum measurement falls below 200 millitorr. A rotary ¼ turn pneumatically operated ball valve then opens, allowing the probe to insert through the isolation valve and into the mass spectrometer (Figure 4 and Figure 5).



The two drawings below show the insertion of the probe through the isolation valve.

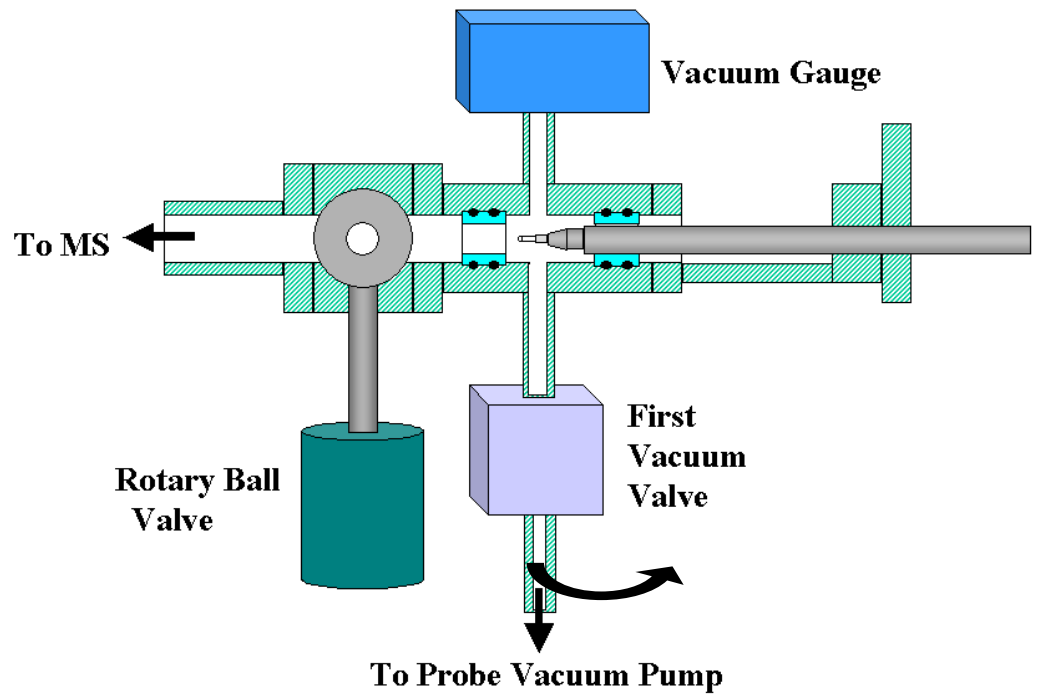


Figure 4 - Probe Positioned at the first seal in the isolation valve

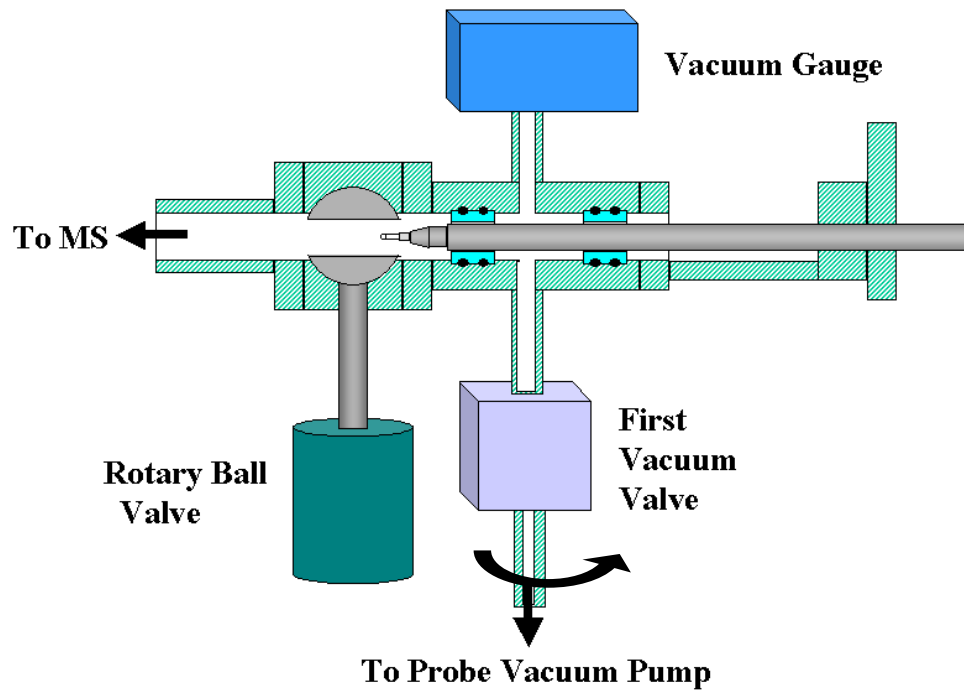


Figure 5 - Probe passing through the rotary ball valve



Included inside the AutoProbe Console (Figure 3) are the following:

1. 24-volt main power supply
2. power supply for the CTC PAL (Auxiliary Box)
3. current-controlled power supply for heating the probe filament
4. microprocessor for operating the AutoProbe hardware
5. A servo controller and linear actuator for moving the probe
6. DEP probe with replaceable probe tips
7. two solenoid valves for the isolation valve
8. relay switch for the probe
9. vacuum pressure sensor
10. pneumatic rotary ball isolation valve
11. electrically controlled vacuum pump rough out valve



Figure 6 - Front Panel of the AutoProbe Console

The panel on the front of the AutoProbe Console has a series of three LED lights that indicate the status of the AutoProbe: Power On, Running, Fault or Door Ajar. Also on this display is an emergency Pause button, which will halt the operation of the AutoProbe so that the probe will not move or heat until the button is pressed a second time to continue operation. This switch only pauses the AutoProbe Console; it does not stop or pause the CTC PAL Autosampler.



Figure 7 - Side Panel on the AutoProbe Console

The panel on the right side of the AutoProbe Console has a series of plugs for attaching various cables used for interfacing the AutoProbe to the CTC PAL Autosampler, the PC, and the mass spectrometer. The main power cord connector and ON/OFF Switch are located below and under this panel.



Figure 8 - Vacuum Pump Hose attachment to the AutoProbe Console

The vacuum pump is attached to a pump flange nipple on the bottom of the AutoProbe Console.

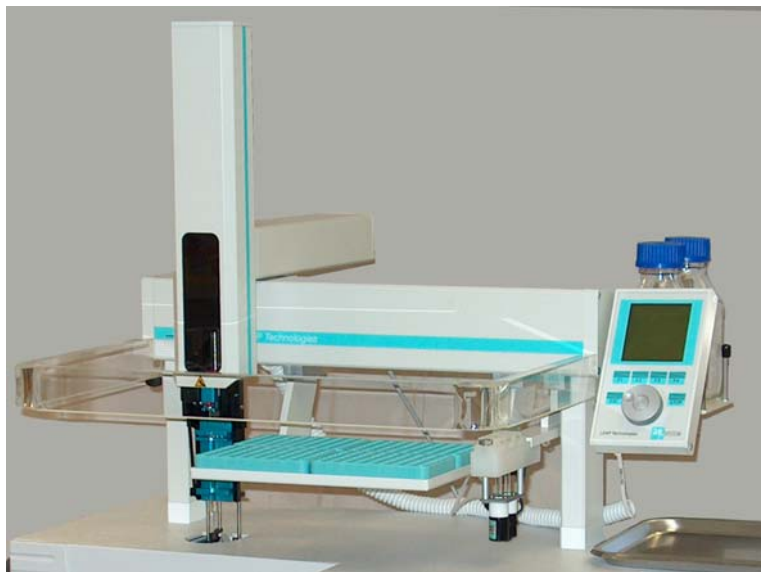


Figure 9 - CTC PAL Autosampler attached to the AutoProbe

The CTC PAL Autosampler is mounted on the top of the AutoProbe Console. This PAL Autosampler system can be removed and converted back to a standard GC or LC autosampler if necessary. However, in order to make this change, you will need to purchase an additional power supply, cord, and set of mounting feet for the CTC PAL Autosampler.



AutoProbe System Components

The AutoProbe System as shipped with the following components (one item each unless otherwise noted). Upon receipt, check the packaging to ensure that all components are included.

1. AutoProbe Console with Adjustable Base
2. CTC PAL Autosampler
3. Software
 - a. SIS AutoProbe Software CD
 - b. PAL Driver for Xcalibur with Cycle Composer CD
4. Edwards Vacuum Pump
5. MS-Probe Interface Tube
6. AutoProbe Accessories
 - a. DEP Filament (2 included)
 - b. Source Position Calibration Tip (1 included)
 - c. Probe Seals (1 spare set included)
 - d. Isolation Valve Seal Set (1 spare set included)
7. One Sample Tray and one Pump Tray
8. Compressed Air Gas Line - 1/8" O.D. x 20 feet flexible nylon air line
9. AutoProbe Cables
 - a. Serial Cable - PC to AutoProbe
 - b. Serial Cable - PC to CTC PAL Autosampler
 - c. Remote Start Cable - AutoProbe to MS
 - d. AutoProbe IO Cable - AutoProbe to CTC PAL Autosampler
 - e. CTC Low-Voltage Power Cord
 - f. Main Power Cord for AutoProbe
10. Accessory Tools
 - a. Allen Wrenches
 - b. Probe Seal Removal Tool
11. Manuals
 - a. SIS AutoProbe Manual
 - b. CTC PAL Autosampler Manual
12. Optional Accessories - (must be purchased separately)
 - a. PC Serial Port
13. Recommended Spare Parts (must be purchased separately)
 - a. DEP Probe Filaments
 - b. Probe Seals
 - c. Isolation Valve Seal Set



AutoProbe System Specifications and Requirements

Voltages

- AutoProbe Console: 110 VAC, 10 amps
- Edward Vacuum Pump: 110 VAC

Fuses

- Main Fuse: 10 amp slow blow

Compressed Air

- Minimum Pressure: 60 psi
- Maximum Pressure: 100 psi

Mass Spectrometer System Requirements

- Thermo Finnigan PolarisQ or DSQ MS System operating under Xcalibur Software (PolarisQ and DSQ tested with version 1.3 and 1.4) and a Windows NT-based operating system (tested under NT4, 2000, and XP). Open Access is optional (tested under version 1.1 and 1.3).
- Polaris Q or DSQ MS Source with Ion Volumes modified for the AutoProbe (see parts section E of this manual)

Dimensions

- Console (excluding PAL Autosampler): 42" wide x 17" Deep x 15" High
- Console (with PAL Autosampler): 42" wide x 17" Deep x 37" High
-

Weight

- Console Weight: 150 pounds
- Total Shipping Weight: 300 pounds



AutoProbe System Features

The following are the main features of the AutoProbe:

- Automated Direct Probe for continuous analysis of samples
- Uses Direct Exposure Probe (DEP) Technique (also called DCI)
- Contains CTC PAL Autosampler to load liquid samples onto probe tip
- Can run 200 to 500 samples per day unattended
- Analyzes samples at the rate of 2 to 5 minutes per sample
- Uses plug-in replaceable DEP probe filaments
- Has long filament life (expected life >1000 samples per filament)
- Constant DEP filament current or programmable in up to three temperature ramps
- Automatic high temperature filament cleaning between samples
- Loads four sample trays with 54 or more samples each
- Fully integrated with the Thermo Finnigan Xcalibur software
- Probe method stored as part of the sample method
- Can use different methods for subsequent samples in a series
- Can be used in Open Access mode of operation
- Manual Control Window permits manual positioning of probe
- Long life probe seals with expected life in excess of 2000 injections
- Probe seals easily replaced by user
- Automatic injection of probe through a pneumatically operated inlet valve
- Vacuum gauge measures vacuum in probe inlet to eliminate insertion of probe into source if seals are leaking
- Automatic sequence stop programmed into system if leaks, broken filament or other problems occur
- Automatic repositioning of probe without vacuum leaks if a power outage occurs when the probe is in the MS vacuum



B. Hardware Installation



AutoProbe System Installation Outline

The installation of the AutoProbe requires a number of steps that should be followed in the sequence outlined below.

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Adding a Serial Port to the PC

Two serial ports are required on the PC for the operation of the AutoProbe System: one serial port for the AutoProbe and a second serial port for the CTC PAL Autosampler attached to the AutoProbe. The Thermo Finnigan MS System computer is normally supplied with two serial ports, one of which is normally utilized for the GC, and the second of which is intended for an autosampler. If one or more of these serial ports are already used, then an additional serial port card will need to be added to the computer. One and two port serial cards for the PC are available from several computer accessory suppliers.

Follow the manufacturers instructions when installing these cards. When installing the serial port card, it may be necessary to configure the PC in the CMOS setup using the following settings. The IRQ settings can vary depending on what is available on the PC.

Serial Port	Address
1	3F8H
2	2F8H
3	3E8H
4	2E8H



Preparing the Thermo Finnigan MS System

Vent MS System and Remove MS Source

The AutoProbe is attached to the Finnigan DSQ or PolarisQ mass spectrometer through the GC transfer line inlet. Therefore to install the AutoProbe, the GC and the GC/MS transfer line must be removed from the mass spectrometer.



Refer to the Thermo Finnigan MS manual and vent the mass spectrometer as outlined in the manual. Remove the source from the mass spectrometer.

After the MS has been shut down and vented, remove the GC/MS transfer line using a 2 mm allen wrench and set aside. Install the AutoProbe Transfer line in the same location using the same "O" Ring and screws to attach it to the Mass Spectrometer. The small 1/16" stainless steel line on the side of the transfer line is for the attachment of the CI reagent gas for the mass spectrometer.

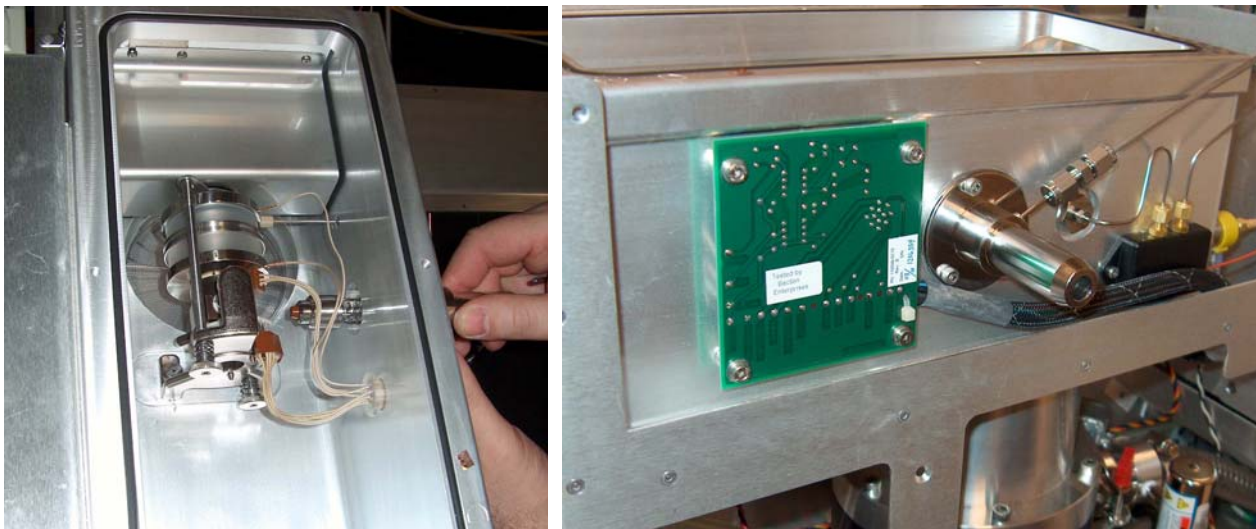
Installing the MS-Probe Interface

After the MS has been shut down and vented, separate the GC from the Mass Spectrometer and remove the MS covers



Figure 10 – Preparing the Thermo Finnigan PolarisQ

Next remove the GC/MS transfer line by unscrewing the three socket screws holding it to the manifold housing. Also disconnect the CI gas line where it goes into the transfer line. The three socket screws, the "O" Ring seal and the CI gas line will be used to connect to the new AutoProbe transfer line. Installation of the AutoProbe transfer line on the Polaris Q and DSQ are very similar as shown below.



Attaching the AutoProbe Transfer line to the Polaris Q MS

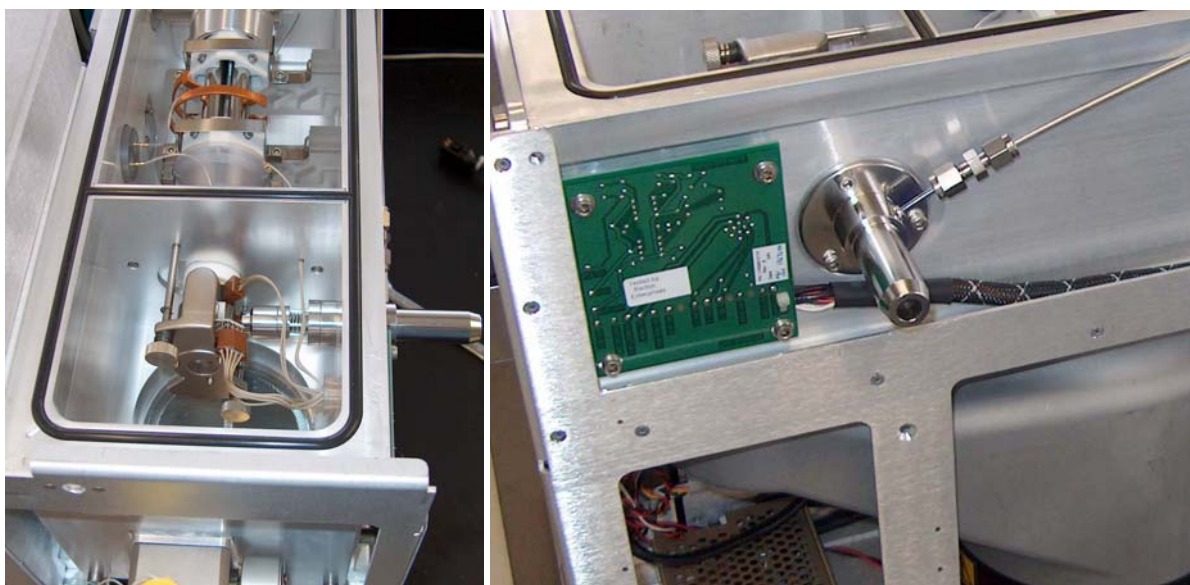


Figure 11 - Attaching the AutoProbe Transfer line to the DSQ MS.

Install the AutoProbe Transfer line in the same location. Attach the AutoProbe MS Probe interface tube to the MS source flange using the three hex screws without washers that are supplied with the GC/MS transfer line. Reuse the O-ring from the GC/MS Transfer Line. Use a new 1/8" Swagelok nut on the CI gas line to attach it to the new AutoProbe transfer line.

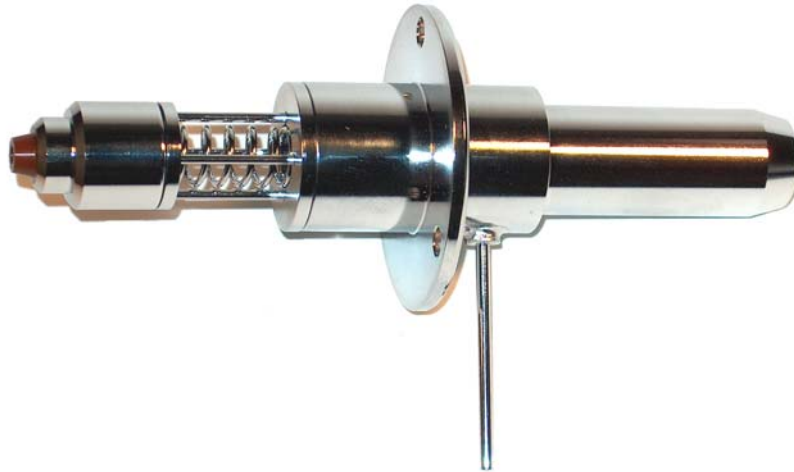


Figure 12 - AutoProbe Transfer Line for the Finnigan MS

The AutoProbe Transfer line includes a spring which provides tension to hold the transfer line parts in place and snugly against the mass spectrometer source. This spring location is open to the mass spectrometer vacuum and is the location where bake-out of the probe filament occurs. See the Maintenance Section (Section D) for details on the assembly of the transfer line and for the replacement of the "O" Ring Seals.



Inspect the probe source to insure that the source wires and other parts will not interfere with the probe as it passes through the Adaptor Tube and into the mass spec source. Also make sure the parts of the transfer line are perfectly aligned and that the transfer line assembly fits snugly against the mass spectrometer source.



DO NOT TURN ON THE MASS SPEC SYSTEM - The MS System is still open to air since the probe inlet is open.



Installing the AutoProbe System

Alignment of the AutoProbe

The AutoProbe Console is attached to the MS Interface Tube with a locking compression nut. The AutoProbe height is normally adjusted by the SIS technician during installation and does not normally need to be readjusted. If adjustment is required, please contact SIS directly for additional instructions.

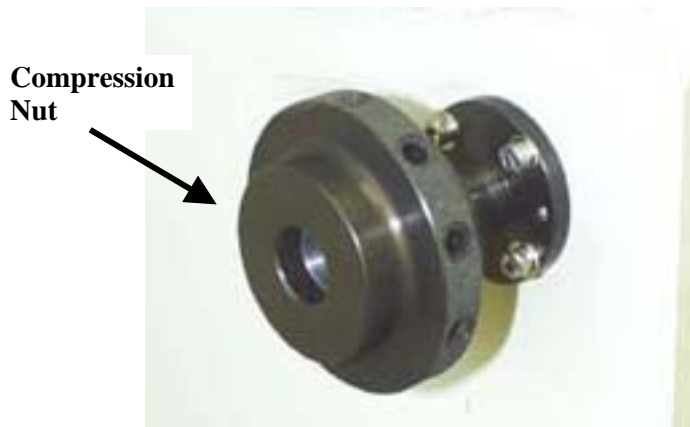


Figure 12 - Compression Nut on the AutoProbe left side panel

Attach the large Compression Nut to the Inlet Assembly on the side of the AutoProbe Console.

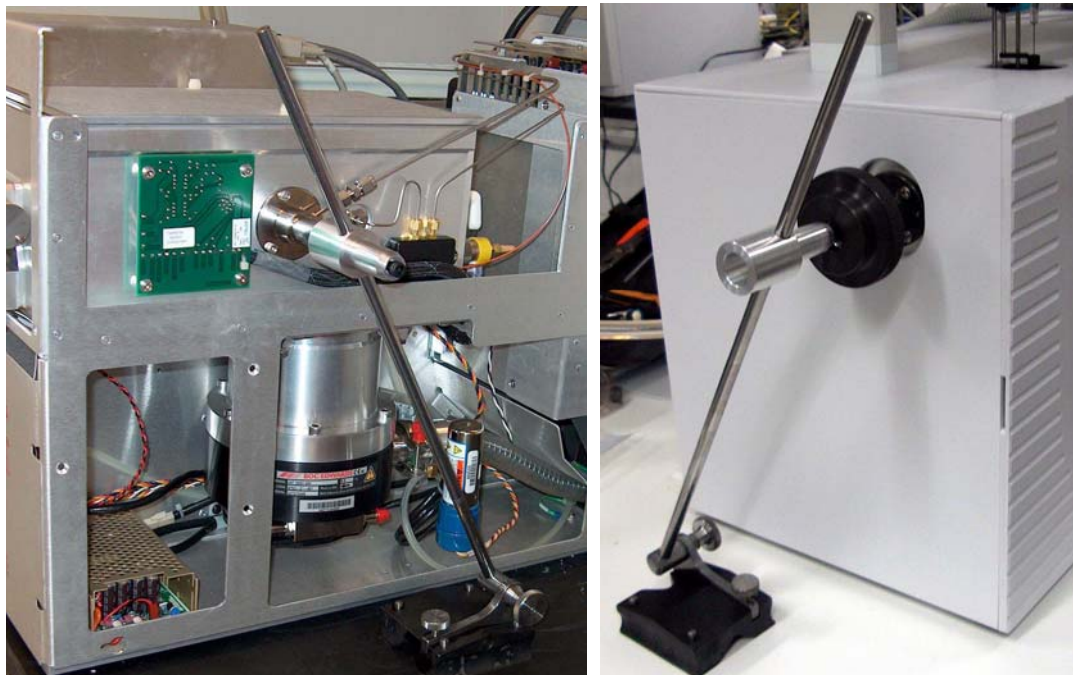


Figure 13 - Alignment of AutoProbe with the MS Interface Tube using the special alignment tool. (This tool is NOT supplied with your system)



A special alignment tool is used to set the height of the centerline of the AutoProbe to the center of the MS source (Figure 13 and Figure 14). This alignment procedure is done by the technician at the time of installation and does not normally need to ever be performed or redone by the user.



Figure 14 – Adjustment of the AutoProbe height (to be performed ONLY by a trained technician)

Attaching the AutoProbe to the MS System

Remove the Compression Nut from the Inlet Assembly and insert it on the AutoProbe MS-Probe Interface Tube. Also attach the compression sleeve and O-ring onto the MS-Probe Interface tube as shown. All the covers for the AutoProbe and the Mass Spectrometer can be replaced at this time. It is preferable that the top cover and the source plate cover on the mass spectrometer are not reinstalled at this time. This will make it easier to assure the proper alignment and operation of the AutoProbe before the mass spec is put under vacuum.

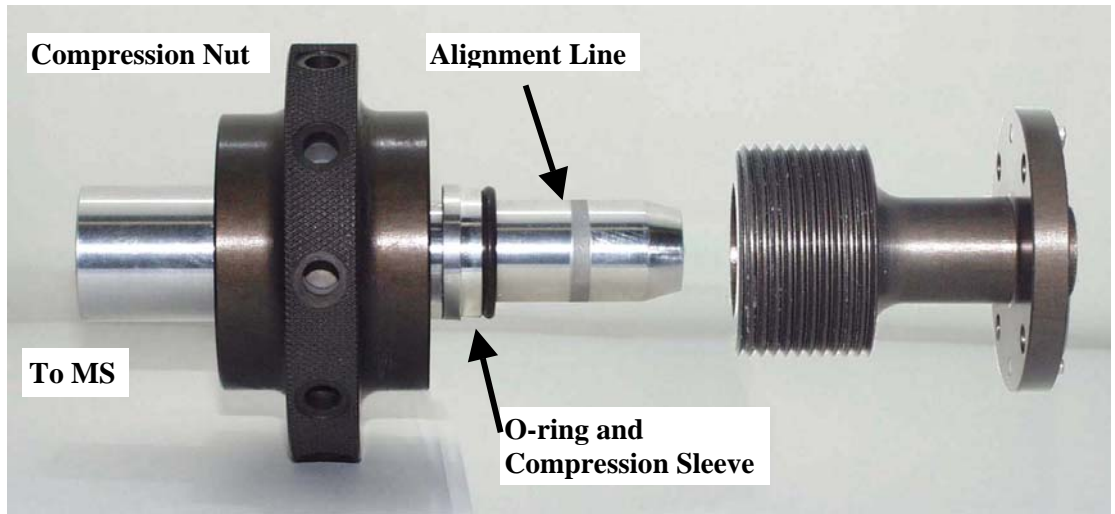


Figure 15 - Alignment of the AutoProbe to MS-Probe Interface

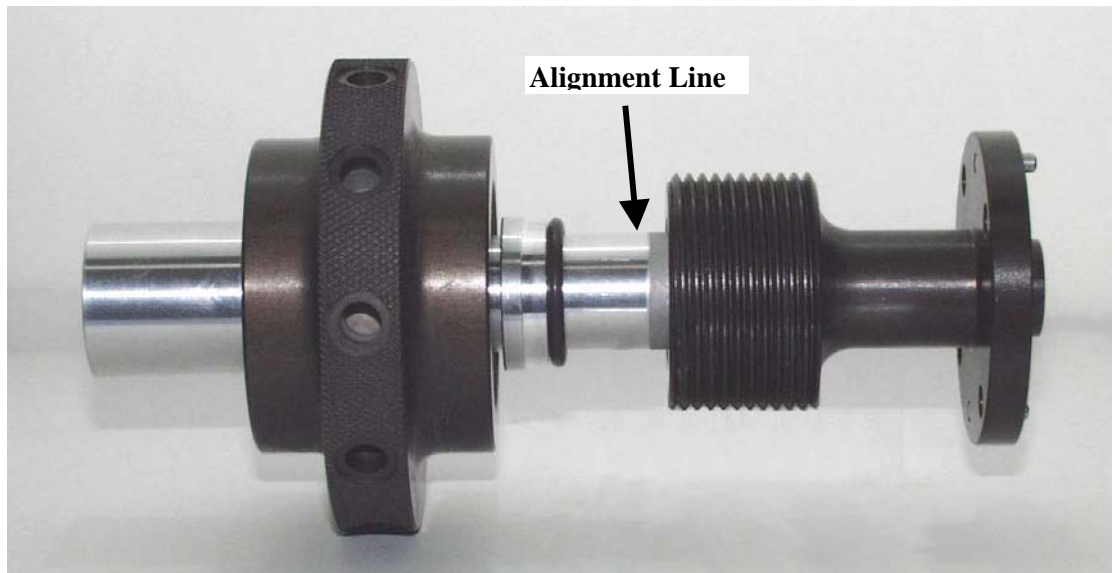


Figure 16 - Attachment of the AutoProbe to the MS-Probe Interface

Move the AutoProbe Console and Base to align the large compression nut with the AutoProbe MS Probe Interface tube on the MS Source flange. Make sure the AutoProbe is square with the MS and then slowly push the two systems together until the AutoProbe MS-Probe Interface tube is seated fully inside the Inlet Assembly. The alignment groove on the AutoProbe MS-Probe Interface tube should be barely visible. Screw on the Compression Nut with its compression sleeve and O-ring. Screw on this fitting until firmly sealed.



Figure 17 - AutoProbe attached to the MS with Compression Nut

Make sure that the AutoProbe Console is parallel to the MS and adjust if necessary.



Figure 18 – AutoProbe attached to the Thermo Finnigan PolarisQ.



Installation of the Vacuum Pump

Be sure that the vacuum pump is charged with pump oil to the fill line. Attach the vacuum pump hose to the vacuum pump using the centering ring and clamp. Then attach the other end of the vacuum hose to the vacuum flange adaptor on the base of the AutoProbe Console using the appropriate centering ring and clamp. Make sure the clamps are tight and that both centering rings and O-rings are properly installed on all flanges.

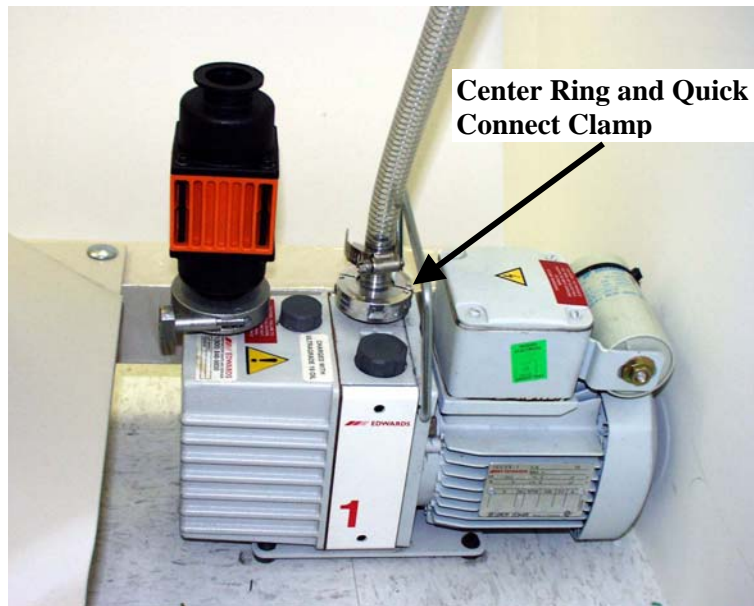


Figure 19 - Installation of the Vacuum Pump



Be certain that all vacuum pump exhaust is properly filtered. A SIS Vacuum Pump Filter Kit (Part # FK-251) is recommended.



Figure 20 - Installation of the Vacuum Pump Hose to AutoProbe



Cables and Gas Lines

A number of cables and gas lines are required for the operation of the AutoProbe System. These Cables and Gas Lines are as follows:

1. Serial Cable - PC to AutoProbe (Fig 20)
2. Serial Cable - PC to CTC PAL Autosampler (On the PAL System)
3. Remote Start Cable - AutoProbe to MS (Fig 20)
4. PAL Interface Cable - AutoProbe to CTC PAL Autosampler (Fig 20)
5. CTC Low Voltage Power Cord (On Pal System)
6. Main Power Cord for AutoProbe (On the AutoProbe Main Chassis)
7. Compressed Air Gas Line - 1/8" x 20 feet flexible gas line (Fig 20)

These cables attach to the back panel of the AutoProbe Console as shown below.



Figure 21 - AutoProbe Right Side Panel for cable hookup

Attach each of the cables as listed above. Most of the cables have unique ends and can only be connected in one configuration. However do take note as to which serial cable is attached to which serial port on the PC for the AutoProbe and CTC PAL Autosampler, since these port identities will be needed to configure these devices in the instrument setup screens.

The power supply for the CTC PAL Autosampler is located outside of the AutoProbe and a cable from the AutoProbe provides this low voltage power. An additional I/O Cable (Pal Interface) from the CTC PAL Autosampler provides additional communication to the AutoProbe.



A source of compressed air is needed for the operation of the isolation valve in the AutoProbe. A 1/8" flexible nylon gas line is provided for this gas. Either house compressed air or tank compressed air or nitrogen can be used as long as at least 60 psi but less than 100 psi of pressure can be supplied to the AutoProbe.



Make sure that all gas fittings are tight and check the air lines for leaks before proceeding. A supply of dry clean air is required for the operation of the AutoProbe. It is advisable to install an air dryer in the gas line if considerable water vapor is present in your air supply.

Power On the AutoProbe System

After the AutoProbe system has been physically attached to the Mass Spec and the vacuum system has been installed, the systems can be powered up. Do **NOT** power up the mass spectrometer at this time. The alignment of the AutoProbe with the Mass Spectrometer will be checked before the mass spectrometer is put back in service.

You can also turn on the AutoProbe and CTC PAL Autosampler at this time. Turn on both the CTC Power switch and the AutoProbe Power switches on the rear of the AutoProbe Console.

After the software has been installed and the alignment and operation of the AutoProbe has been tested, the mass spectrometer source cover plate and the top cover of the mass spectrometer can be reinstalled. The mass spectrometer can then be powered up and made ready for the analysis of samples.



Installing the CTC PAL Autosampler

Refer to the CTC PAL Autosampler manual for additional details and to perform the installation of the CTC Autosampler.



Install a 10- μ L LC style syringe. **Use only the 10- μ L LC style syringe with a blunt squared end (CTC part # SYRC L10-225-3).** Other style needles such as 20-degree point will NOT deliver samples to the probe filament wire. Also the maximum sample size that can be delivered to the probe wire tip is 1.0 μ L, so using a larger syringe is not practical. Sample sizes from 0.125 μ L up to 1.0 μ L can be efficiently delivered to the DEP probe tip wire.



Figure 22 - CTC PAL Autosampler Syringe injecting sample

Also install the appropriate CTC sample trays for your needs and configure the CTC PAL Autosampler as per the CTC Autosampler manual.

Refer to the CTC PAL Autosampler manual to set up the CTC PAL system including, wash positions, inject position, tray types and other variables if these values have not been previously set up. Normally these values have been preset at SIS when your system was set up and tested. If these values need to be changed, refer to the CTC manual. These values need to be set before the CTC PAL system is configured in Xcalibur.



C. Software Preparation and System Calibration



Background

Overview.

For the AutoProbe to function properly, certain software must first be installed on your PC. The software installation process is slightly involved because the system is composed of multiple distinct components, and each component is highly customizable and versatile in its own right. The individual components may be used together in the manner described here for regular AutoProbe analyses, or a different combination or configuration of the components may be used for custom applications (e.g. PAL without the AutoProbe Console).

Preparation Roadmap

The steps required for getting a new system configured and running can be outlined as follows.

Instrument preparation

- Install software components.
- Configure software components.
- Calibrate instruments.

Analysis preparation

- Set up instrument methods.
- Set up processing methods.
- Create a sequence in Xcalibur
- Set up Open Access.

Installing Software Components

Initial Considerations

Computer to install to.

Most (or all) of the below software will be installed on a single “acquiring” PC. The acquiring PC is the PC to which the various instruments (MS, PAL Autosampler, and AutoProbe Console) are physically connected via communication links (e.g. Ethernet, serial/RS-232, TDAT, etc.). You will need to install Xcalibur and various instrument drivers on this computer.

File system location to install to.

During the installation process, you will be prompted for locations of file system folders in which to install the software. Be aware that although Xcalibur 1.2 can be installed in any location, a bug in Open Access 1.3 for Xcalibur 1.2 requires that Xcalibur be installed in either “c:\xcalibur” or “d:\xcalibur.” Other software can be installed anywhere, but the author prefers installing all other components inside the same folder as Xcalibur for easier system maintenance. This ensures that all related software is in one place.

Acquiring PC requirements.

The acquiring PC must have an operating system installed that is compatible with the version of Xcalibur and other software you will use. In addition, if you wish to generate sample reports, Xcalibur requires Microsoft Office be installed. The AutoProbe software has been tested on Windows NT 4.0, 2000, and XP, and it will probably work on any later Windows operating system. However, Thermo Finnigan typically tests a given version of Xcalibur



only on a single version of Windows with a single version of Office. Just because it software wasn't tested on other operating systems does not necessarily mean that it will not work otherwise; however, there are known instances where it indeed will not work otherwise. In any case, review the release notes that come with each software component for respective system requirements. Release notes are typically available with each software component.

Uninstalling if reinstalling.

If you are reinstalling AutoProbe, CTC, or Xcalibur software, it is good practice to first completely purge the old version of the software from your system and then reboot your computer. It is a general case for existing Windows-based software that strange system behavior and conflicts can occur with a new copy of software if pieces of the old copy of the software linger in the system. It is strongly recommended that you use the "Start | Settings | Control Panel | Add/Remove Programs" feature in Windows to uninstall software from your system. Do not simply delete the file system directory of the given software package since registry settings and other changes may linger in the system. If the folder is deleted (or moved) manually, then the "Start | Settings | Control Panel | Add/Remove Programs" feature in Windows may fail, in which case fully uninstalling the software might only be possible by manual means, which often is not a straightforward task. Rebooting your computer at times may be necessary to release any lingering locks system resources or files.

Outline of the installation process

The order in which components are installed is important. Some components refuse to install if certain prerequisites are not met, such as other components first being installed. Although there is some flexibility in the installation sequence, SIS recommends that you install the components in the order given below unless there is reason otherwise. Thermo Finnigan systems frequently ship with a number of these components preinstalled, so you do not have to reinstall those components.

- **Operating System Service Packs.** Operating system service packs contain bug and security fixes for your operating system. As good practice, SIS highly recommends that you install the latest service pack available for your specific operating system. The latest service pack can be downloaded from the Microsoft web site. Alternately, you likely will find a service pack for your operating system on the Xcalibur CD, although the service pack might not be recent. In Windows NT, 2000, and XP, you can determine which service pack (if any) is already installed on your operating system by selecting "Start | Run..." from the Windows Task Bar and then entering "winver" in the Run dialog box. You will then see a dialog box containing some text such as the following (emphasis added):

Microsoft (R) Windows
Version 5.1 (Build 2600....*Service Pack 1*)
Copyright (C) 1981-2001 Microsoft Corp.

A service pack may be installed at any time, although it is best to install it before installing other software.

- **The Xcalibur Core.** The Xcalibur Core is the central software component of Xcalibur. The Xcalibur Core is quite generic since it allows third party vendors (such as SIS, CTC, or Thermo Finnigan themselves) to attach their own instrument-specific software into the Xcalibur Core thereby permitting a multi-instrument analysis. The



Xcalibur Core can be found on the Xcalibur CD. Versions 1.1, 1.2, 1.3, and 1.4 of the Xcalibur Core are known to work with the AutoProbe. Your mass spectrometer may have its own requirements (the PolarisQ and DSQ require Xcalibur 1.3, 1.4, or above). If the Xcalibur Core is already installed, you can determine which version of the Xcalibur Core you are currently using by selecting “Help | About Home Page...” from the Xcalibur Home Page window. As good practice, you may wish to check the Xcalibur section on the Thermo Finnigan web site for any “software updates” (bug fixes) that can be freely downloaded for your version of Xcalibur. Xcalibur must be installed prior to installing the PolarisQ/DSQ MS Driver for Xcalibur, Cycle Composer, PAL Driver for Xcalibur, SIS AutoProbe software, and Open Access.

- **PolarisQ or DSQ MS Driver for Xcalibur.** This software attaches to the Xcalibur Core to allow Xcalibur to manage the MS. The MS Driver can be found on the Xcalibur CD. The MS Driver can only be installed after installing the Xcalibur Core. The default program folder of “c:\xcalibur” is ok. Check your mass spec software manual for configuration details.
- **Cycle Composer.** This software controls the PAL Autosampler from the PC. It can provide standalone control the PAL, and it can interface to various data systems via data system drivers. Cycle Composer can be found on the PAL Driver for Xcalibur CD. Version 1.5 is known to work with the AutoProbe. Cycle Composer must be installed before the PAL Driver for Xcalibur is installed. The default installation folder is “c:\Program Files\PAL\Cycle Composer”, but the author prefers “c:\xcalibur\PAL\Cycle Composer”. Be sure to install the component named “Cycle Composer for HTS, HTC, and LC PAL”. The default program folder is “PAL System”, but the author prefers “Xcalibur” (however, the installation program in version 1.5 seems to ignore this choice).

PAL Macro and Method files for the AutoProbe. By default, the PAL is configured to behave like an LC autosampler. For the most part, the AutoProbe is content that the PAL behaves like an LC autosampler. However, some fine adjustments should be made to the PAL for optimal operation with the AutoProbe. For this reason, SIS has created a special PAL method file (SISAUTOPROBE.PME) and PAL macro file (SISAUTOPROBE-Inject.pma) that should be installed into Cycle Composer. You can copy these files from the AutoProbe CD to the “c:\Program Files\PAL\Cycle Composer\HTC PAL” (or, if applicable, “c:\xcalibur\PAL\Cycle Composer\HTC PAL”) folder. The SISAUTOPROBE method will be visible the next time you open Cycle Composer or the PAL section of the Xcalibur Instrument Method Setup window (Figure 36).

- **PAL Driver for Xcalibur.** This software interfaces the PAL Cycle Composer software to the Xcalibur Core software so that Xcalibur can manage the PAL. The PAL Driver can be found on the PAL Driver for Xcalibur CD. Version 1.3.1 is known to work with the AutoProbe. Although previous versions partly worked, it is highly recommended that a newer version be obtained. This software may only be installed after the Xcalibur Core software and Cycle Composer software are installed. The default destination of “c:\xcalibur” is ok. The “Cycle Composer root folder” must be set to the same folder you specified in the Cycle Composer set up. This defaults to and should be “c:\Program Files\PAL\Cycle Composer” (or “c:\xcalibur\PAL\Cycle Composer” if applicable).



Side Note: The A200S Autosampler Driver provided with Xcalibur can also control the PAL in lieu of the PAL Driver and Cycle Composer, but this A200S driver has some limitations, such as only supporting a single tray and single method. The method must be named “A200-S” and must be stored on the PAL itself..

- **SIS AutoProbe Software.** This software can run alone, for standalone control of the AutoProbe system, or it can attach to the Xcalibur Core, for integrated management of the AutoProbe system from Xcalibur. The SIS AutoProbe Software can be found on the SIS AutoProbe CD. This software must be installed after the Xcalibur Core software is installed. The default destination folder is “c:\Program Files\SIS\autoprobe”, but the author prefers “c:\xcalibur\autoprobe”. Accept the default for installing all components. At the end of the installation a dialog box pops up permitting you to configure the global AutoProbe settings (Figure 23). Specify the communications port on your computer to which the AutoProbe is connected to and click "Test" to verify the communication link to the AutoProbe System. You may need to press this button a second time for the entropy on the communications line to be cleared. You can safely ignore the advanced settings for now.

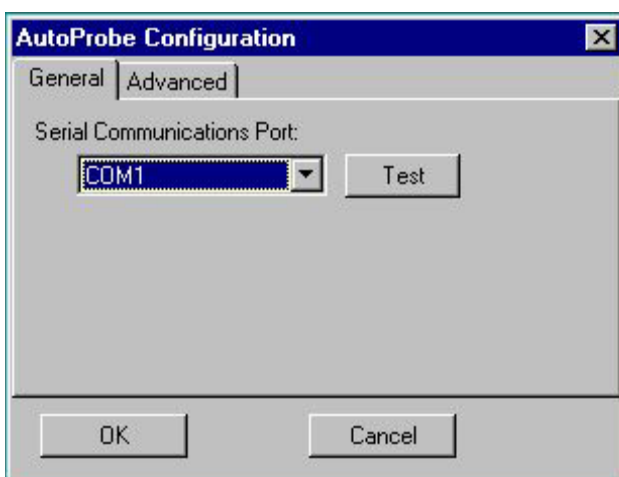


Figure 23 - AutoProbe Instrument Configuration Window

In addition to the above required components, you may wish to install the following optional software components:

- **Xcalibur Open Access.** This can be found on the Xcalibur Open Access CD. It provides a much simplified and multi-user graphical interface to the Xcalibur Core. It is suitable in a so-called “Open Access” lab setting, where the end user requires minimal knowledge of the system. There are two software components to Open Access: Open Access Server and Open Access Login. The server component talks to the Xcalibur Core, maintains a queue of pending samples, and provides a user interface for the data system administrator to configure the system and prepare standard Open Access methods for the end users. The login client component interacts with the end user and provides a simplified user interface to checking in samples for analysis. The server must be installed on the same computer as Xcalibur is installed. The login client can be installed on that same computer, on another computer, or on multiple other computers. Note: There is an Open Access version 1.3 designed to work with Xcalibur 1.2 (numbers do not always match). During installation, the default “Xcalibur” program folder is ok.



- **Any other Xcalibur components.** Other software on the Xcalibur CD such as NIST or Product Information may be installed as needed after installing the Xcalibur Core. These software components are independent of and do not affect the AutoProbe.



Global Software Configuration

Now that the software is installed, you are ready to configure the software. Each component you installed in the previous section must be configured, so you can use that list as a checklist. You can configure each component roughly in the order in which they were installed with the exception that some of the Xcalibur Core configuration can only be performed after the instrument drivers are configured. The components requiring configuration are as follows:

- Xcalibur Core.
- PolarisQ or DSQ MS Driver for Xcalibur
- PAL Driver for Xcalibur / Cycle Composer
- SIS AutoProbe Software
- Cycle Composer
- Xcalibur Open Access (if installed)

Xcalibur Core

In order for the autosampler, AutoProbe, and MS instruments to operate within the Xcalibur environment, Xcalibur must be told which instruments to use. To do so, start the Xcalibur Instrument Configuration program. The following window appears.

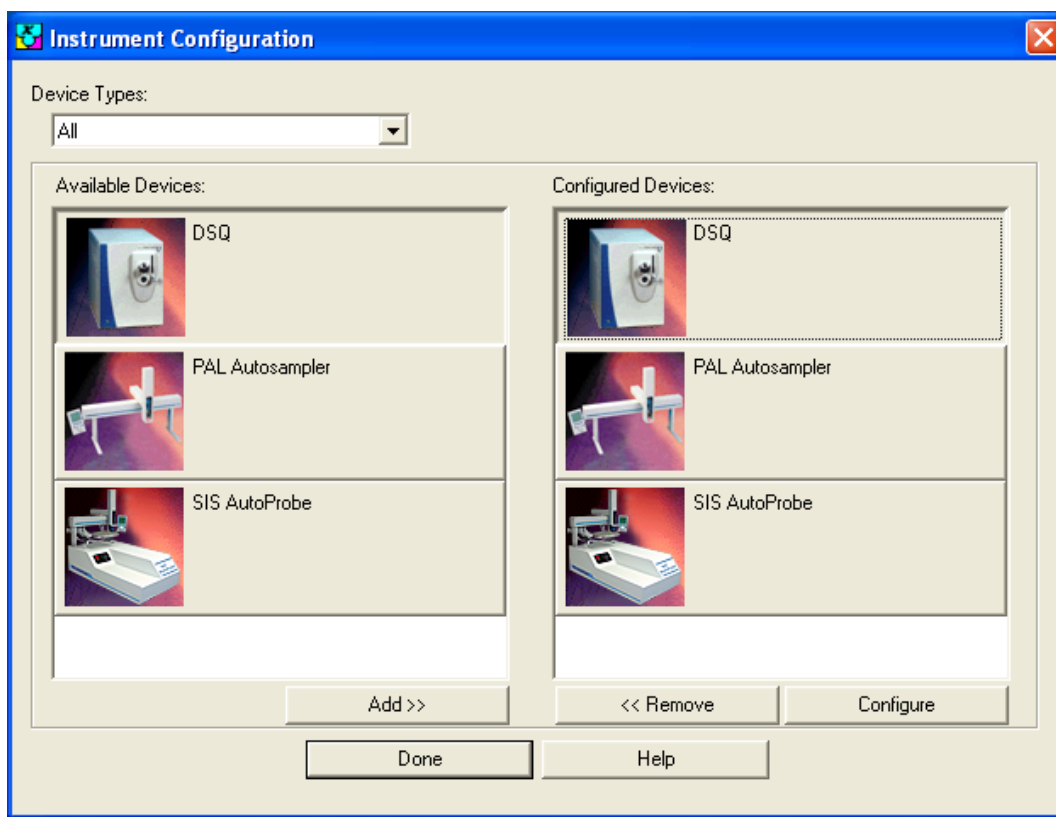


Figure 24 - Instrument Setup Window



Add each of the following instruments by clicking on the instrument in the left panel and then clicking on the “Add >>” button so that the instrument appears in the right panel.

1. PolarisQ or DSQ MS (whichever one you have)
2. PAL Autosampler
3. SIS AutoProbe

The next step is to configure each instrument.

PolarisQ or DSQ MS Driver for Xcalibur

In the Xcalibur Instrument Setup window (Figure 24), ensure that your MS is installed in the right panel and double click your MS button to configure it. Refer to your MS software manual for configuration options.

PAL Driver for Xcalibur / Cycle Composer

In the Xcalibur Instrument Setup window (Figure 24), ensure that the PAL is installed in the right panel and double click the PAL Autosampler button to configure it. It is important that the method and communications port are set correctly. First, set the method folder. This is by default “c:\Program Files\PAL\Cycle Composer\HTS PAL” (although the author prefers “c:\xcalibur\PAL\Cycle Composer\HTS PAL”). The communications port is the serial port on your computer to which the PAL is connected. It is necessarily different than the communications port that the AutoProbe uses. You can test whether your choice of communications port is correct by clicking the “get configuration from PAL” button. This will download the configuration set on the PAL keypad from the PAL to the PC. You may need to click this button again at a later time if you sometime make changes to the PAL configuration (e.g. tray type) from the key pad, otherwise the PAL driver for Xcalibur might not be aware of those changes, and odd behavior might result. Refer to your PAL Autosampler manual for specifics on configuring this dialog.

SIS AutoProbe Software

In the Xcalibur Instrument Setup window (Figure 23 and Figure 25), ensure that the SIS AutoProbe is installed in the right panel and double click the SIS AutoProbe button to configure it. This dialog was previously configured during the AutoProbe software installation, but it’s a good idea to double-check it now. It is important that the communications port is set correctly. The communications port is the serial port on our computer to which the AutoProbe is connected. It is necessarily different than the communications port that the PAL uses. You can test whether your choice of communications port is correct by clicking the “Test” button. You may need to press this button twice to clear out any entropy on the communications line.

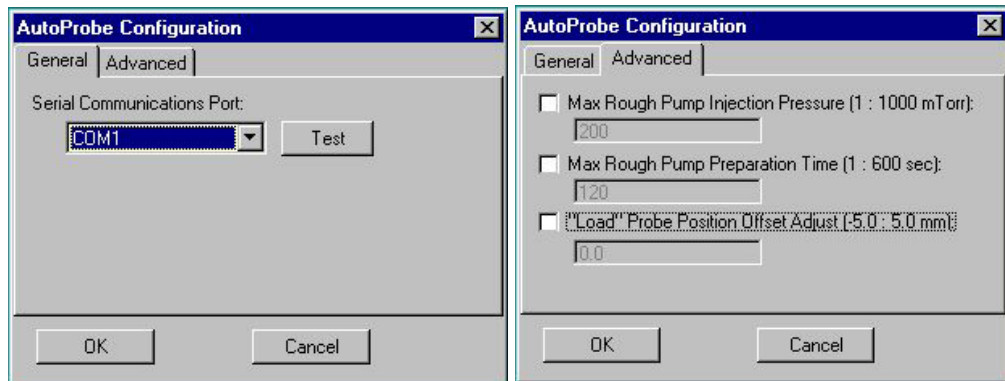


Figure 25 - AutoProbe Configuration Windows

The settings in the Advanced tab might not need to be changed, but be aware of their existence. Defaults are used when corresponding checkboxes are unchecked.

The setting most likely needed to be changed is the **“Load’ Probe Position Offset Adjust.”** This is a fine adjustment on the horizontal position of the probe filament with respect to the syringe needle during sample loading. If the alignment of the needle is not correct, the horizontal position of the probe can be adjusted here to position the syringe needle precisely over the center of the DEP probe tip filament coil. Page 47 describes how to accurately position the syringe needle onto the filament.

The **“Max Rough Pump Injection Pressure”** is the pressure to which the isolation valve vacuum must fall before the isolation valve opens and the probe proceeds in to the source. It is recommended that the value of 200 millitorr be used. The next line, **“Max Rough Pump Pressure Time”** is the time limit that the vacuum pump has to achieve the above set pressure. It is recommended that this time be set to 120 seconds. If the set vacuum pressure is not reached in the operation of the probe within the 120 seconds time (2.0 minutes), the probe will withdraw from the isolation valve and a vacuum leak warning message is displayed in the status panel of Xcalibur on the AutoProbe Manual control window pending user action.



Calibrate Instruments

Calibrate PAL object types and positions.

Use the PAL keypad to configure the types of trays and syringes and the X-Y-Z coordinates of tray holders, trays, injection ports, and wash stations. Details of these procedures are provided in the PAL manual. Note that the injection port is called “LC Vlv1”. These positions are preset before shipment, but fine adjustment may be required. In addition, these values must be reset/rechecked if the PAL system is removed from the AutoProbe Console and reinstalled onto the AutoProbe Console.

Many other settings such as speeds, signals, and events can be configured on the AutoProbe keypad, but doing so is not required since SIS preconfigures all such required settings prior to AutoProbe shipment.

Calibrate Source Position with AutoProbe

This procedure will determine the exact point at which the probe tip meets the source of the mass spectrometer. This contact point will serve as the end point for the AutoProbe inject cycle; it is where the probe filament will heat the sample for MS analysis.

First remove the right rear side panel on the AutoProbe by pulling the panel straight out to expose the probe.

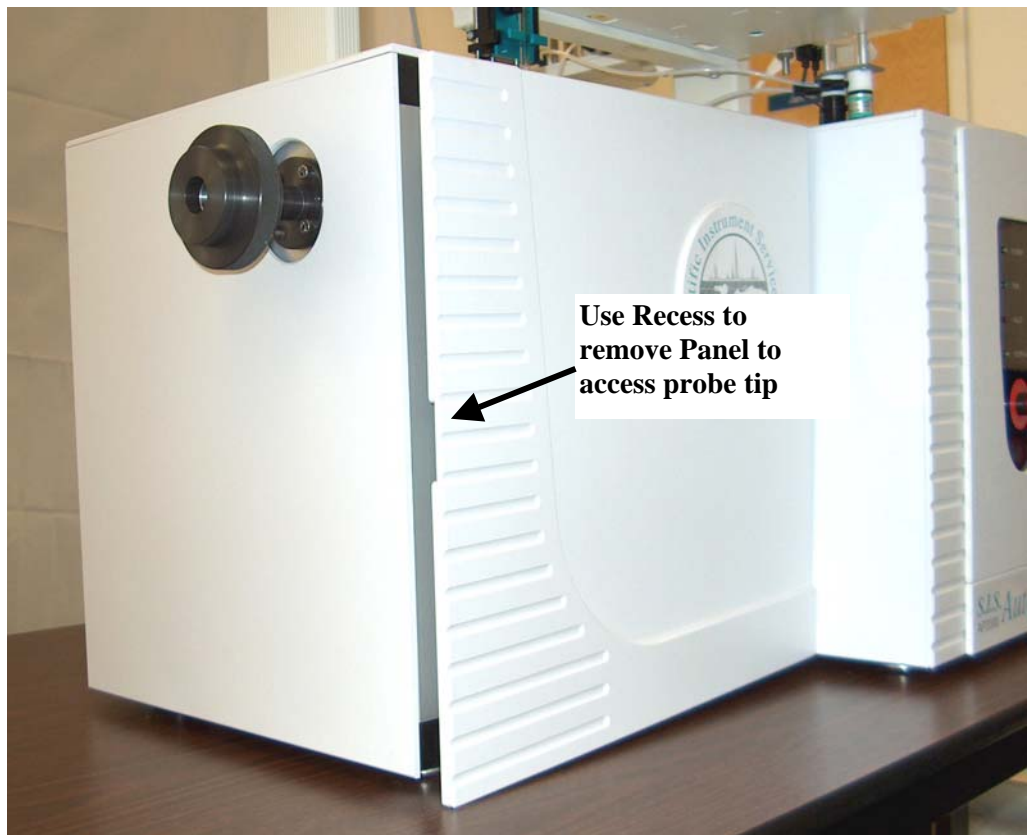
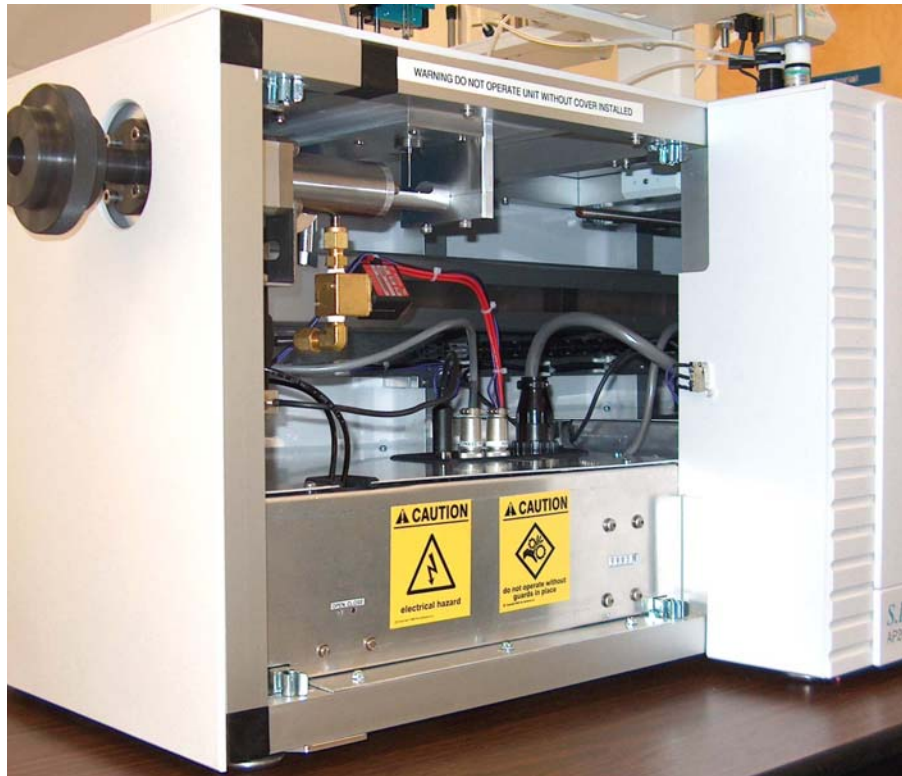


Figure 26 - Front Panel Removal





Next remove the DEP filament in the end of the probe by pulling it straight in the direction of the length of the probe. Plug the Source Position Calibration Tip snugly into the end of the probe as shown below.

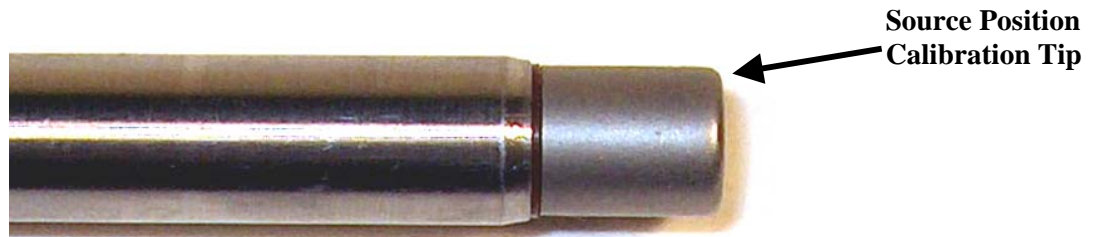


Figure 27 - Installation of the Source Position Calibration Tip

The two electrical connector pins of this tip are electrically connected to the sensor part of the probe tip. When the tip touches the source inside the MS, the tip will become electrically grounded, at which time the AutoProbe software records the position. This procedure should be repeated every time the AutoProbe is removed and reinstalled onto the MS.



The Source End Position must be determined every time that the AutoProbe is removed and reinstalled onto the MS.

Start the AutoProbe Manual Control window from either the windows desktop or the Start button on the Windows Taskbar (**Start | Programs | SIS AutoProbe | Manual Control**). The following window appears:

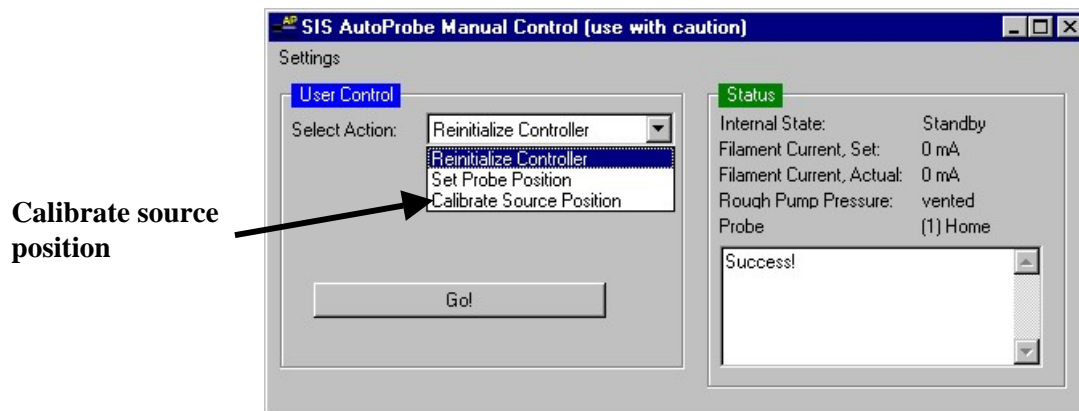


Figure 28 - AutoProbe Manual Control Window

Press the down arrow at “Select Action:”, select the “Calibrate Source Position” program, and press “Go!”



A series of dialogs will appear that will guide you through the Calibrate Source Position procedure. The program will automatically take the probe through the isolation valves and into the mass spec and slowly advance the probe until it touches the source. The AutoProbe Console will then record this end of stroke position in its battery-backed memory, and the AutoProbe Console will hereafter move to this position when it is told to inject against the source. The procedure requires one to two minutes to complete. Follow the directions as specified in these dialogs:

1. Install the Source Position Calibration Tip as described above.
2. Turn on the AutoProbe with the rear switch if not done so already. Make sure that the AutoProbe vacuum pump is turned on and that compressed air is present at the AutoProbe.
3. *Touch the ground wire inside the cabinet to the metal end of the probe tip.*
4. *Do NOT proceed until a Beep is heard, otherwise damage could occur to the MS source from jamming.*
5. Finish the calibration.

When done, the probe will uninject from the mass spec and the status box in the Manual Control window should indicate “Success!” as shown in Figure 28 above.



Remove the Source Position Calibration Tip and install a standard AutoProbe DEP Filament tip. This probe tip is required for the sample loading position calibration.



Figure 29 - Remove the Source Calibration Tip and Install the DEP Filament Probe Tip



Calibrate Sample Loading Position—Horizontal Offset

It is necessary to correctly position the syringe to accurately deliver the liquid sample onto the wire loops on the end of the AutoProbe DEP filament. To accomplish this, a needle with a flat or blunt end is required. The needle must be positioned directly in the middle of the wire coil and as close to the wire coil as possible without touching the wire.

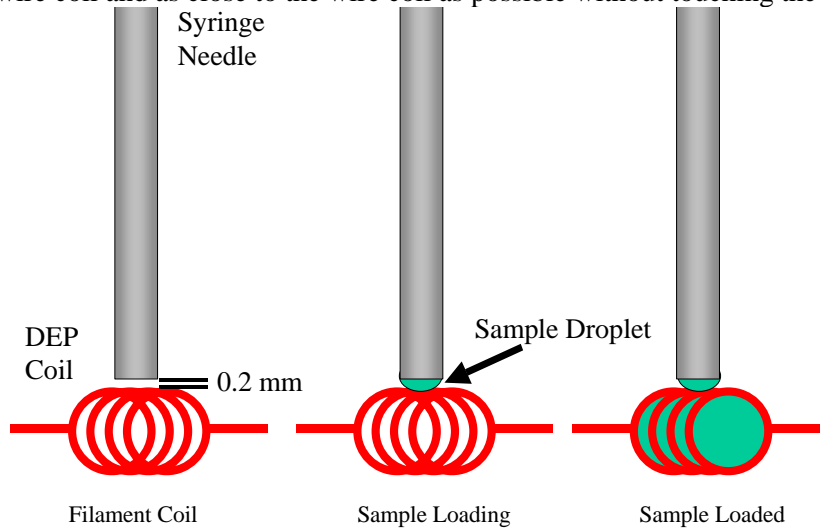


Figure 30 - Loading Sample onto DEP Wire Coil



If the needle is not positioned correctly, the sample drop will not be delivered to the wire coil but instead will ride up the side of the syringe needle.

If not already started from the previous procedure, start the AutoProbe Manual Control window from either the Windows desktop or the Start menu on the Windows Taskbar. The following window appears:

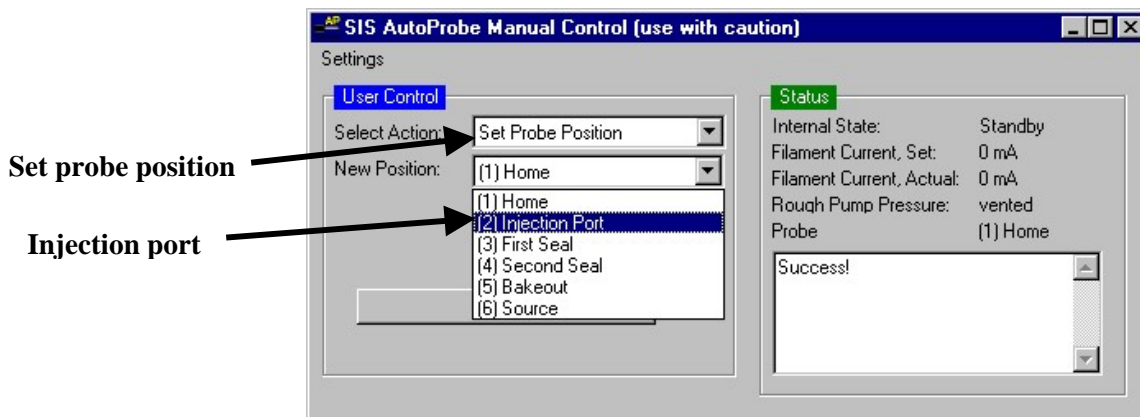


Figure 31 - AutoProbe manual Control Window for Positioning Probe

First be sure that a DEP coil filament is installed in the AutoProbe (Figure 29).



In the Select Action drop down list box, press the down arrow and then select “Set Probe Position.” Then select “(2) Injection Port” for New Position. Finally press “Go!” This will advance the probe to the sample loading position directly under the syringe. If the syringe does not properly align horizontally with the filament DEP coil, adjust the linear position of the probe by opening the Xcalibur Instrument Configuration window, double clicking the AutoProbe button, selecting the Advanced tab, specifying an offset in the “load’ position offset adjust”, saving the results, **restarting all the AutoProbe software**, and repeating the above procedure. Note: a positive offset will align the filament further toward the MS; a negative offset will adjust the filament in the opposite direction.

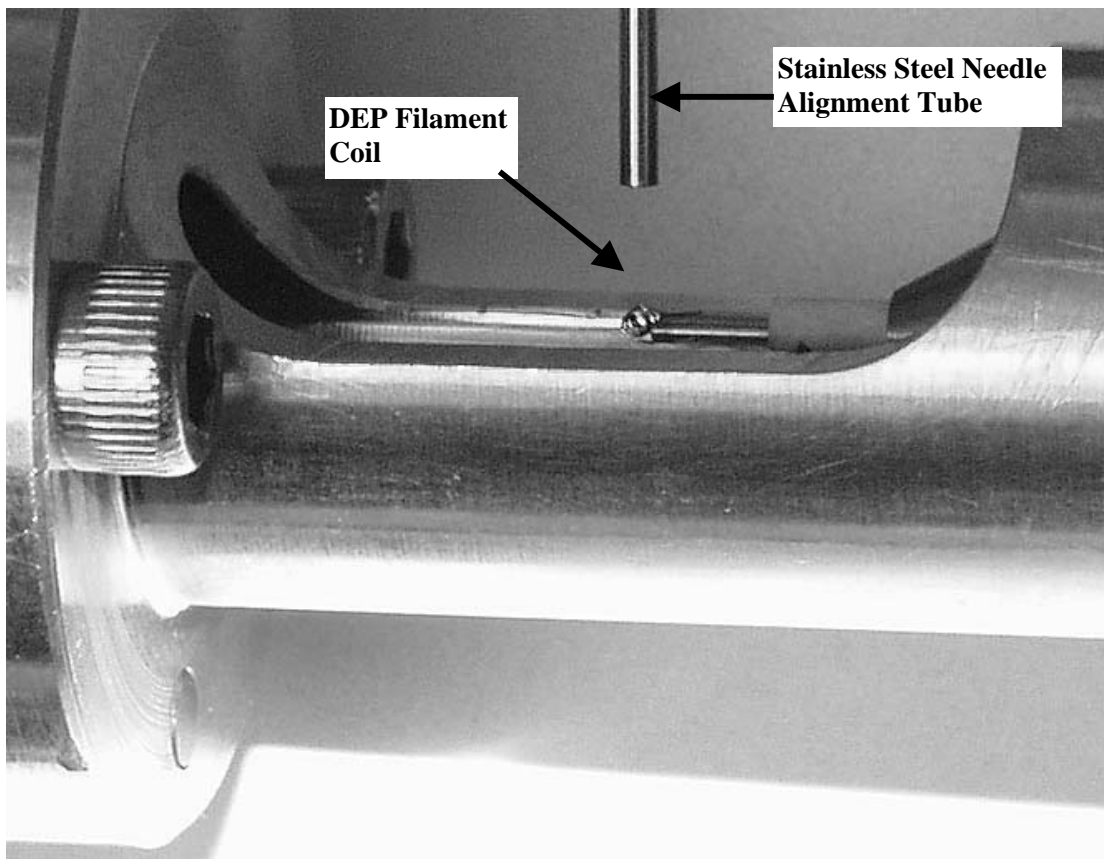


Figure 32 - DEP Filament in Position ready for Syringe Calibration

Calibrate Sample Loading Position—Vertical Offset

You will now need to calibrate the vertical position of the syringe needle against the filament.

While the AutoProbe is still in the sample load position, apply the following command sequence on the PAL keypad to move the syringe position to the injection port. The following assumes that the tray and wash positions have been previously set up for the CTC PAL Autosampler as mentioned in the section “Calibrate PAL object types and positions.” on page 43.

1. Menu <F1>
2. Setup <Enter>
3. Objects <Enter>
4. Injectors <Enter>



5. LC VIv1 <Enter>
6. Scroll down to Needle Penetr and **ensure that this value is no greater than 40 mm**. If the value is greater than 40 mm, press <Enter>, turn the dial so that the value becomes 40 mm, and then press <Enter>. A high value could cause the needle to hit and damage the filament during step 8.
7. Check Position <F1>
(Note: at this time, you may adjust the X-Y-Z positions of the valve if it is misaligned.)
8. Needle Penetr <Enter>

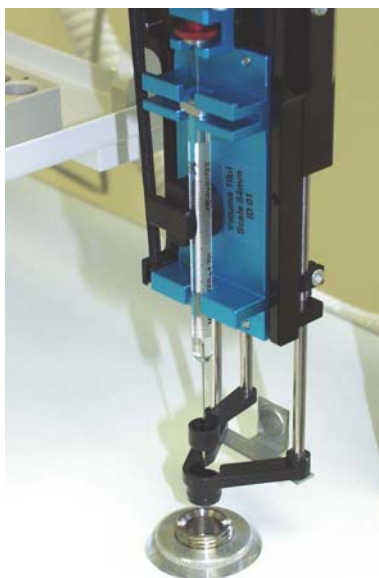


Figure 33 - Syringe Ready for Injection and Calibration of Loading Position

This will position the syringe over the injection area for the AutoProbe. If not done so already, turn the knob on the CTC PAL Keypad so that the needle penetration is at about 40 mm. Then slowly increase the setting one step at a time until the syringe needle just touches the Filament coil wire on the DEP filament (if the syringe tip is not electrically isolated, you will hear a beep), then back off one setting (0.1 to 0.2 mm) from the filament coil. This will position the syringe needle so that as soon as a liquid droplet begins to form at the end of the syringe needle, it will immediately be transferred to the DEP filament coil as shown above (Figure 30). Press <Enter> on the PAL controller to make this setting active. Then press <ESC> multiple times to exit out of the menus.

Tune the Mass Spec

Refer to the MS manuals for auto-tune procedures. It is recommended that the MS be tuned with the probe at the source position.

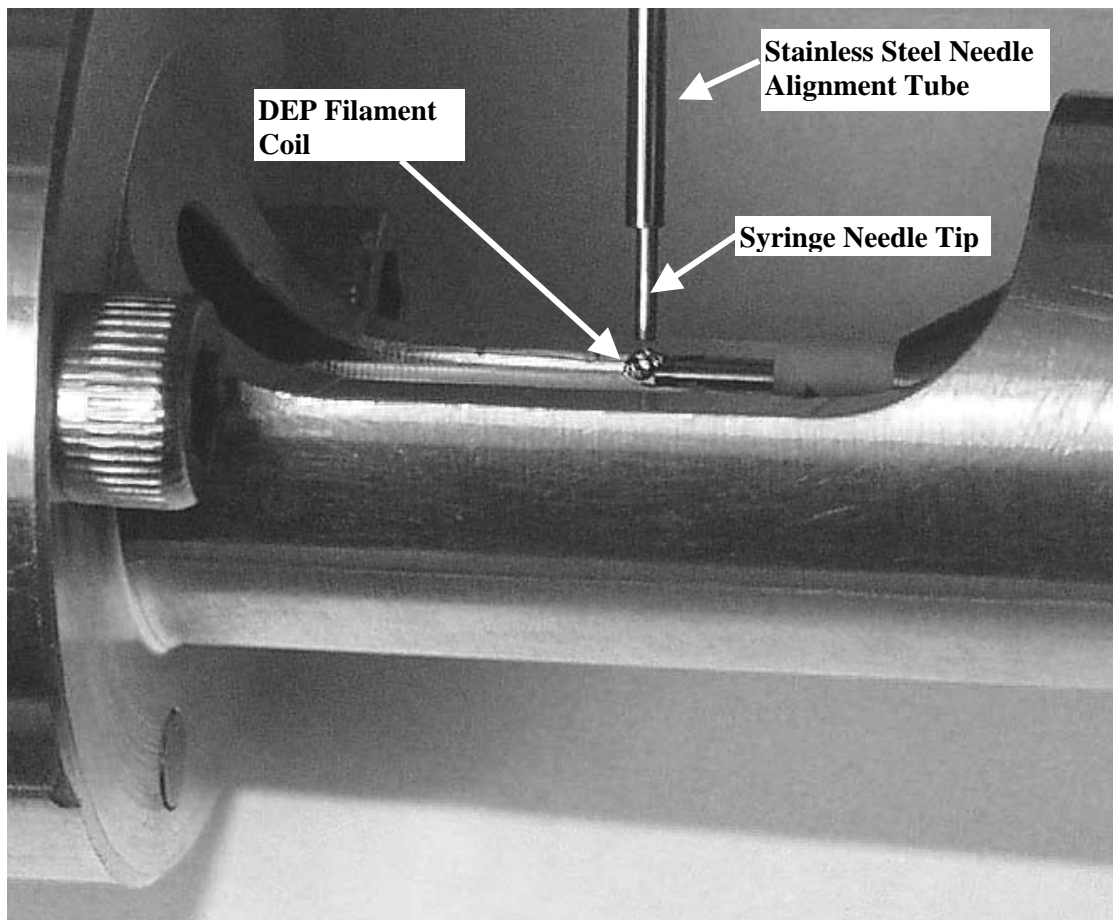


Figure 34 - Syringe Needle Correctly aligned over DEP Filament Coil



Setting Up Instrument Methods

AutoProbe Instrument Parameters

We are now ready to prepare an instrument method. The instrument method defines parameters for one or more sample analysis using the connected instruments. You can prepare multiple methods, and during a sequence of sample analysis, you can (if desired) assign different methods to each sample. The instrument method is different from the instrument configuration prepared in the previous section in that the instrument configuration is designed for “global” settings that change infrequently and pertain to all sample runs.

Start the Xcalibur Home Page. The following window appears.

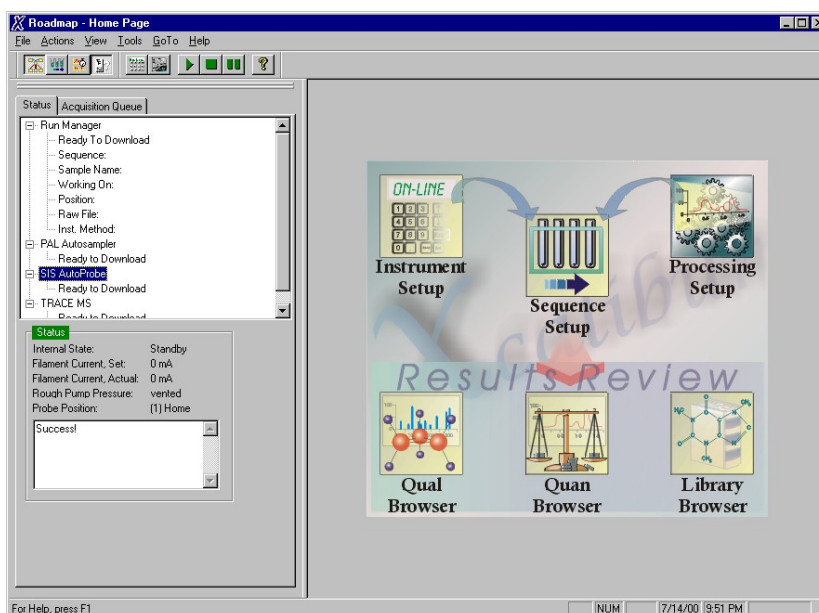


Figure 35 - Xcalibur “Home Page”

Select the “Instrument Setup” icon to bring up the following window.

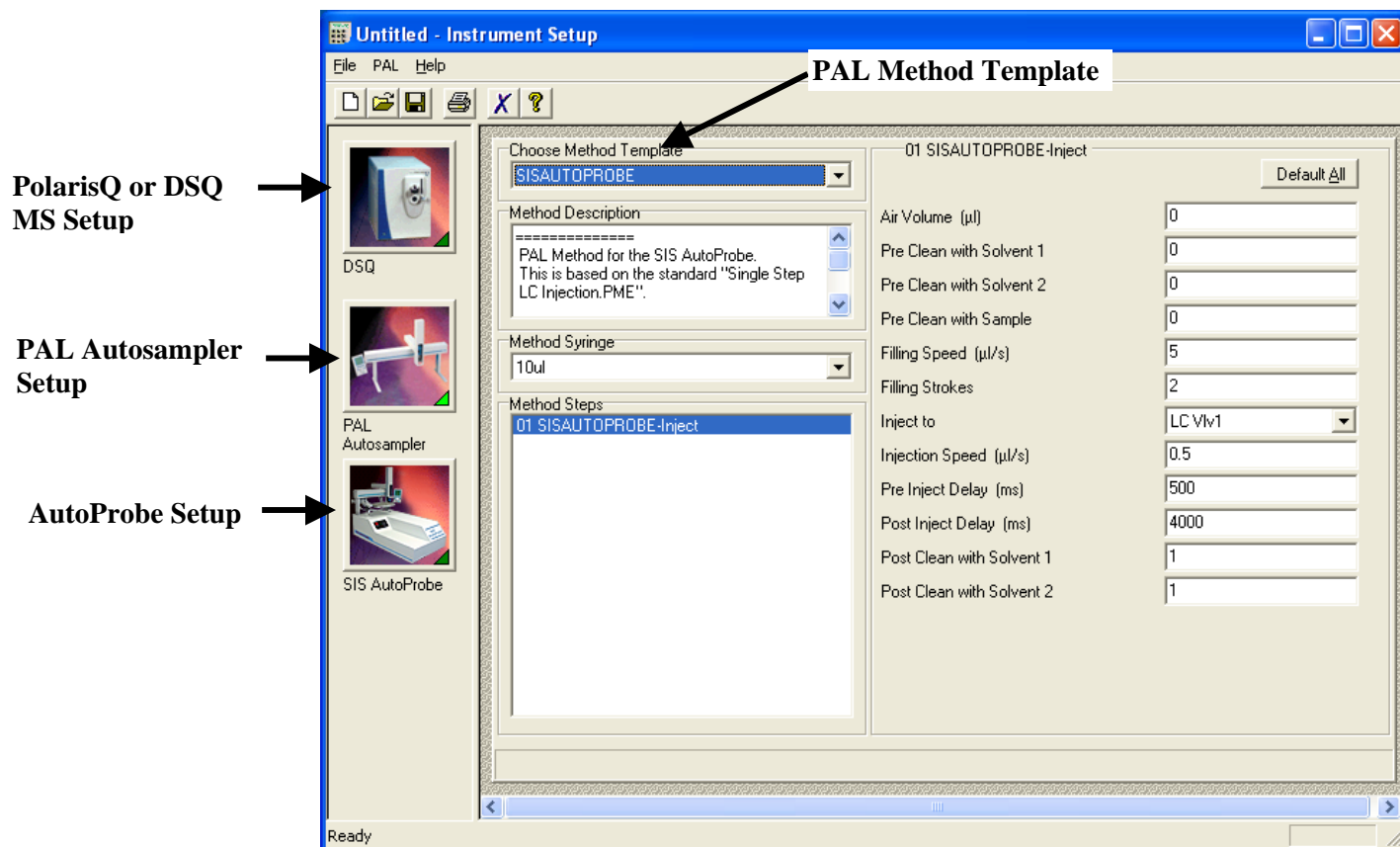


Figure 36 - Xcalibur Instrument Setup Window for PAL Autosampler

In the Choose Method Template Box, select “SISAUTOPROBE”. The defaults should be sufficient, but you may wish to adjust some settings. Refer to the CTC PAL Autosampler manual for the configuration of the above window for the PAL Autosampler. The following settings are critical to the proper operation of the AutoProbe:



CTC PAL Setup parameter	Recommended Value for AutoProbe
Injection Speed (uL/sec)	0.5
Post Inject Delay (ms)	4000
Inject to	LC Vlv1

The rate of injection is slowed down from a normal (LC) injection speed to permit the sample to be delivered to the DEP Probe Tip wire coil. If the speed of injection is too fast, the sample will splatter and not be efficiently delivered to the wire coil.

The long Post Inject Delay delays the retraction of the syringe from the DEP filament wire coil for a short time to allow the liquid sample to be efficiently delivered to the wire coil.



Do not increase the value of the Post Inject Delay beyond 4000 milliseconds or the probe will inject before the syringe is removed from the probe path.





AutoProbe Instrument Method Parameters

Next press the SIS AutoProbe button to set up the AutoProbe method. The following window appears.

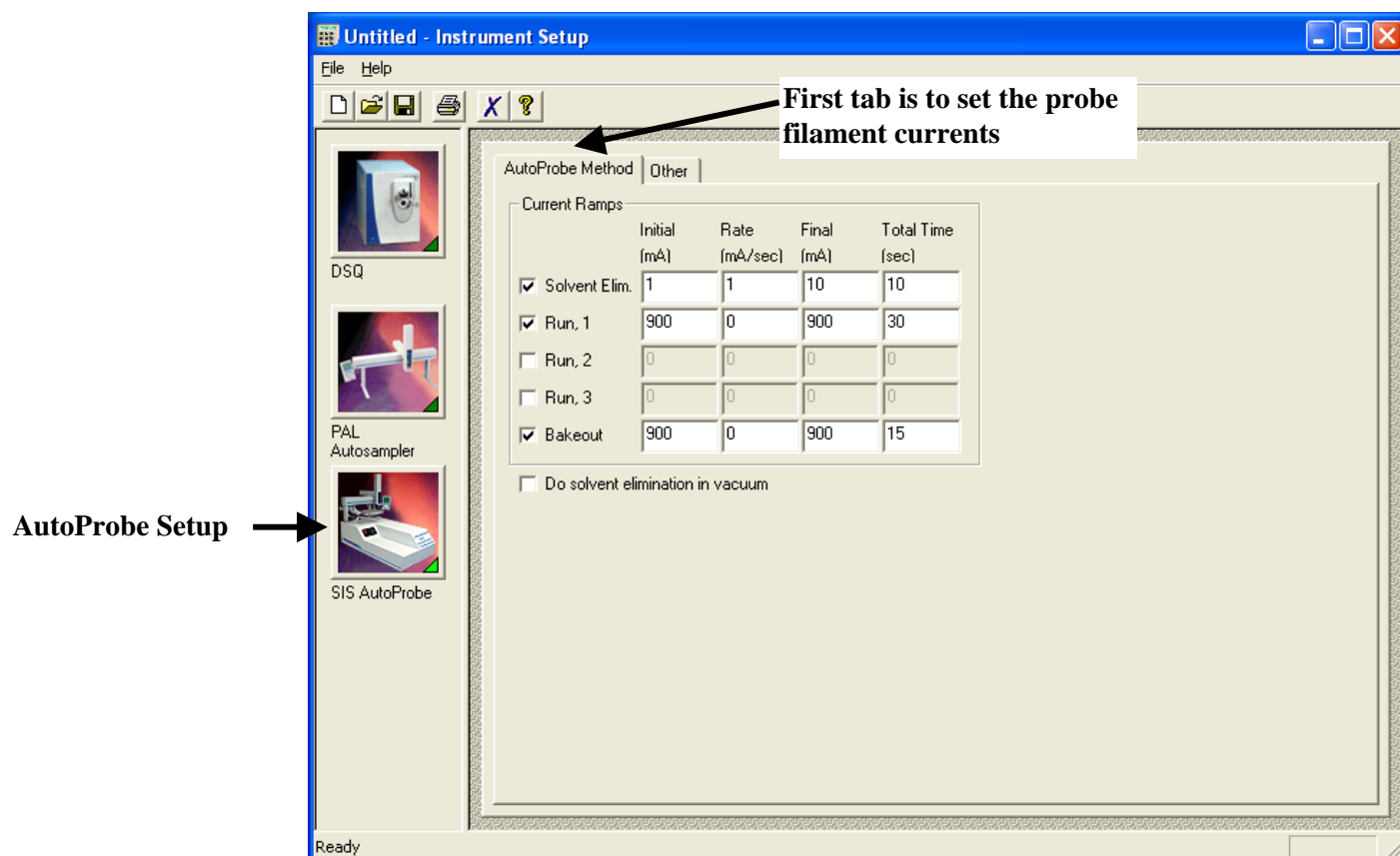


Figure 37 - AutoProbe Method Setup Window

This first tab on the right panel is used to set up the DEP probe filament currents. The second tab on this screen can be used to open the manual control window described previously.

The probe filament is heated at three different times during a sample run.

Step 1.	Solvent Elimination	Low heat to remove solvent
Step 2.	Sample Analysis	Up to 3 steps of hold and ramp
Step 3	Bakeout	High temperature cleaning

The maximum current recommended for the standard DEP Probe Filament is 1000 mA. At this temperature the platinum wire is glowing red-hot and has a surface temperature in excess of 1000 degrees C in vacuum. Higher currents will result in shortened DEP filament life.



Recommended settings for the Filament Currents



	minimum value	maximum value
Solvent Elimination	5 mA	50 mA
Sample Analysis	20 mA	1000 mA
Bakeout	500 mA	1000 mA

Solvent Elimination. Before the sample is analyzed, a small current can be passed through the DEP filament wire to slightly heat the wire and remove the sample solvent. If solvent removal is not required, uncheck the “Solvent Elim.” checkbox. This step of solvent removal can be done either in atmosphere (before the probe is injected into the vacuum system), or in the vacuum inside the mass spec (before the probe is mated against the MS source). If the solvent removal is to be done inside the vacuum, then check the “Do solvent elimination in vacuum” checkbox, else leave this box unchecked to perform solvent removal at the sample load position (at atmospheric pressure).

Sample Analysis. The DEP filament wire can be run at a constant current rate or with a ramp. Up to three different ramp steps are available. These multiple current ramps can be used in some applications to separate analytes in a sample based on boiling points. Just check the current steps desired in the boxes on the left.

Good results can be obtained by running the filament at a single ballistic constant high current of between 500 and 900 mA. This will provide for a quick heating and desorption of the samples into the mass spec source in the minimum amount of time. The result will be sharp total ion chromatograms and therefore the highest level of sensitivity.

Bakeout. The bakeout mode is used to clean the DEP probe tip between samples to prevent cross contamination of samples. After sample analysis, the probe retracts about 1.5 inches out of the source (which is still within the mass spec vacuum) and the bakeout temperature program is performed. Normally an isothermal setting of between 800 and 1000 mA for 15 seconds will thoroughly clean the DEP probe tip wire.

Manual Control. The second tab on the AutoProbe method panel allows the “Manual Control” window to be opened. This window can also be accessed from the Windows desktop or the start menu on the Windows taskbar.

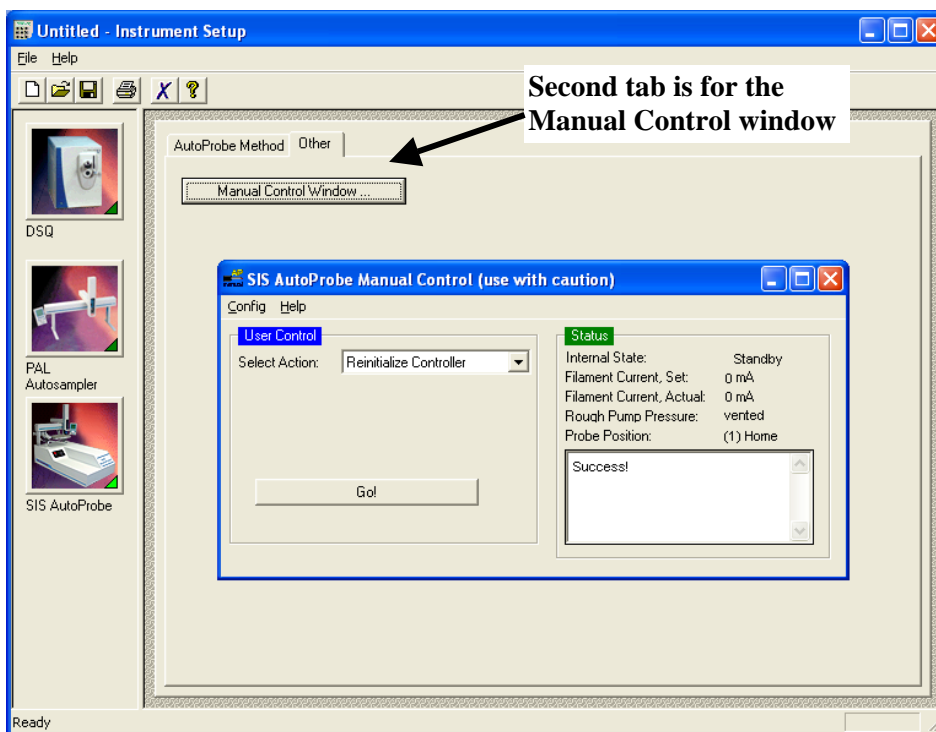


Figure 38 - AutoProbe Manual Control Screen

As described previously, this window is used to manually set the probe position. It is used to position the probe in the load position to align the syringe needle and calibrate the probe stroke length with the MS source. It can also be used to position the probe against the source when the MS is calibrated or when other samples need to be analyzed via the GC. It is especially important in CI tuning that the probe be positioned tightly against the source to assure a high pressure of the reagent gas.



MS Instrument Settings for the AutoProbe

When the PolarisQ or DSQ MS button is pressed on the Xcalibur Instrument Setup window, the following window appears.

PolarisQ or DSQ
MS Setup

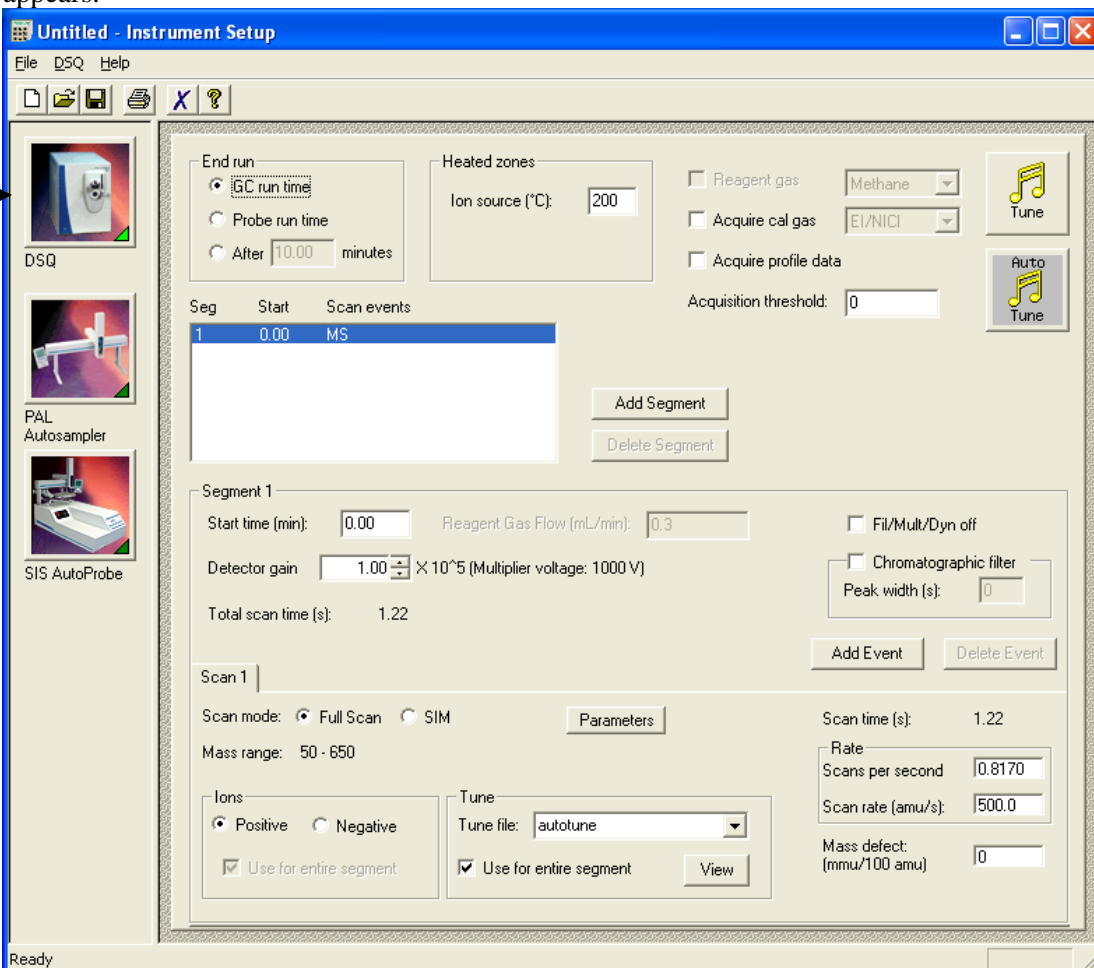


Figure 39 – PolarisQ or DSQ Instrument method Setup Window

Refer to the PolarisQ or DSQ MS manuals for details on these screens.



As shown above, the run is listed as stopping when the “GC run time” finishes. **Be sure that the radio button “GC run time” is checked.** The AutoProbe software works just like the GC. It will stop the collection of MS data at the end of the probe run.



Create a Sequence in Xcalibur

Refer to the Xcalibur and PAL software manuals for creating a sequence of sample runs. A few special notes are in order. Vial positions take the forms Tray01:1, Tray01:2, ... Tray01:54, Tray02:1, ..., Tray04:54. Injection volumes are in μL .

AutoProbe →

AutoProbe Status →

Display Screen →

Sample	Type	File Name	Sample ID	Path	Inst Meth	Proc Meth	Position	Inj Vol
1	Unknown	cholestane01	1	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:1			0.50
2	Unknown	cholestane02	2	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:2			0.50
3	Unknown	cholestane03	3	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:3			0.50
4	Unknown	cholestane04	4	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:4			0.50
5	Unknown	cholestane05	5	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:5			0.50
6	Unknown	cholestane06	6	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:6			0.50
7	Unknown	cholestane07	7	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:7			0.50
8	Unknown	cholestane08	8	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:8			0.50
9	Unknown	cholestane09	9	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:9			0.50
10	Unknown	cholestane10	10	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:10			0.50
11	Unknown	cholestane11	11	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:11			0.50
12	Unknown	cholestane12	12	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:12			0.50
13	Unknown	cholestane13	13	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:13			0.50
14	Unknown	cholestane14	14	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:14			0.50
15	Unknown	cholestane15	15	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:15			0.50
16	Unknown	cholestane16	16	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:16			0.50
17	Unknown	cholestane17	17	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:17			0.50
18	Unknown	cholestane18	18	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:18			0.50
19	Unknown	cholestane19	19	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:19			0.50
20	Unknown	cholestane20	20	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:20			0.50
21	Unknown	cholestane21	21	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:21			0.50
22	Unknown	cholestane22	22	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:22			0.50
23	Unknown	cholestane23	23	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:23			0.50
24	Unknown	cholestane24	24	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:24			0.50
25	Unknown	cholestane25	25	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:25			0.50
26	Unknown	cholestane26	26	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:26			0.50
27	Unknown	cholestane27	27	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:27			0.50
28	Unknown	cholestane28	28	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:28			0.50
29	Unknown	cholestane29	29	C:\Xcalibu C:\Xcalibur\ C:\Xcalibur\m	Tray01:29			10.00

Figure 40 - Xcalibur Sequence Setup Window

Status Window

If the “SIS AutoProbe” item in the Status window is selected, the parameters for the AutoProbe will appear in the Status panel on the lower left of the Xcalibur Home Page. The following values for the AutoProbe are displayed in this panel.

Internal Status	Status of the AutoProbe (Initializing, standby, running, shutdown, not connected)
Filament Current, Set	The Programmed set value for the DEP Filament
Filament Current Actual	The Actual Filament Current (feedback reading)
Rough Pump Pressure	The vacuum measured in the isolation valve
Probe Position	The position of the DEP Probe
Display Screen	Any messages and system errors

This panel can be helpful during the operation of the AutoProbe. If an error occurs, this screen will display the cause so that the problem can be corrected.

Running

When the “Run Sequence” toolbar button on the Xcalibur Home Page is pressed, the following window appears.

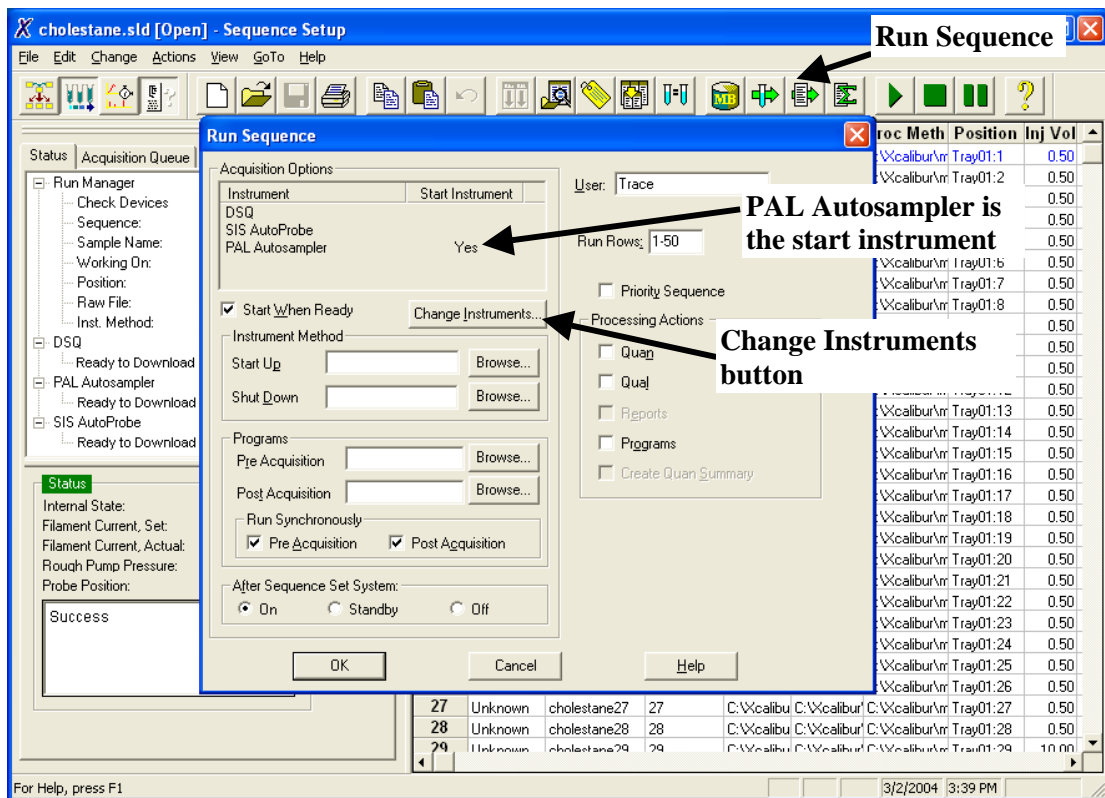


Figure 41 - Xcalibur Run Sequence Window

Before proceeding, make sure that the correct instrument starts the sequence. For the operation of the AutoProbe system, the Xcalibur software will first start the CTC PAL Autosampler and tell it what sample to inject. After the sample is injected, the PAL system signals the AutoProbe to begin its injection. At the completion of the injection, the AutoProbe signals the MS to start collecting data.



It is important that the PAL Autosampler is indicated as the “Start Instrument”.

So, from the window above, select the “Change Instruments” Button. The following window appears.

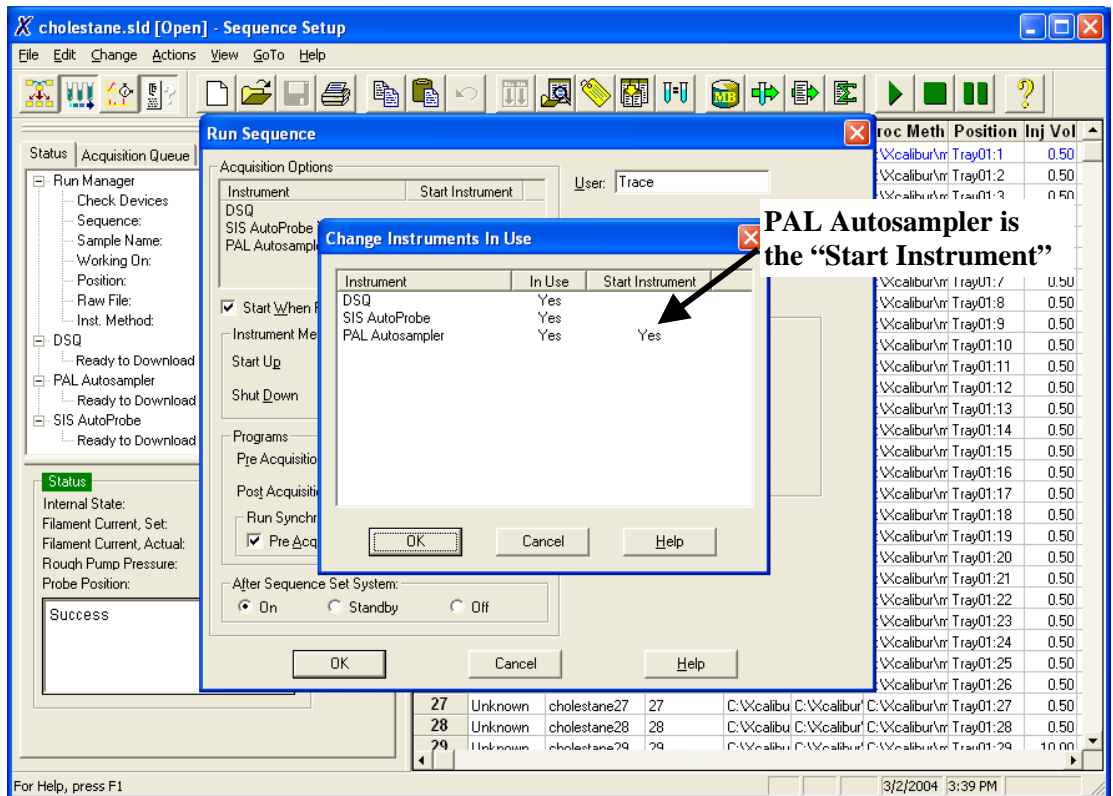


Figure 42 - Xcalibur Change Instruments in Use Window

As shown above, all three instruments must be indicated as “In Use”. The PAL Autosampler must be indicated as the “Start Instrument”.

When finished, press OK, and then press OK on the “Run Sequence” window. The samples will run in the sequence indicated, and the required reports will be generated.

SIS recommends that a suitable standard be run upon first preparing the AutoProbe.



Open Access Operation

For details on the use of the Open Access, refer to the Thermo Finnigan Xcalibur manual. Some main points are presented here.

Open the “Open Access Instrument Server” Program from the Windows desktop. The following window appears (Open Access 1.1 is shown in the screenshots, but Open Access 1.3 is functionally similar).

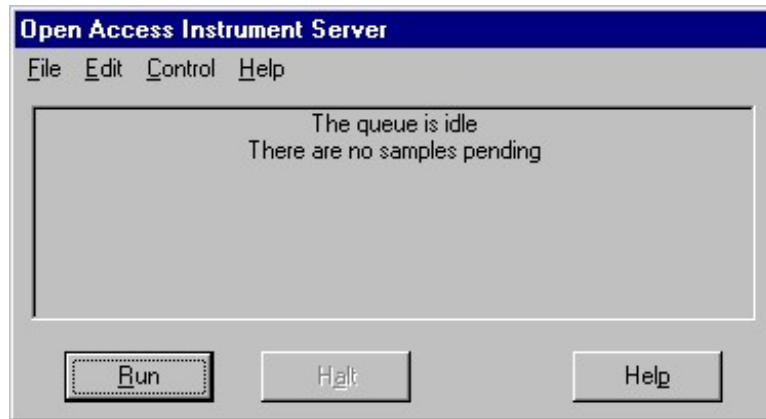


Figure 43 - Xcalibur Open Access Instrument Server

Select the “Edit” menu, and then select “Methods”. The following window appears.

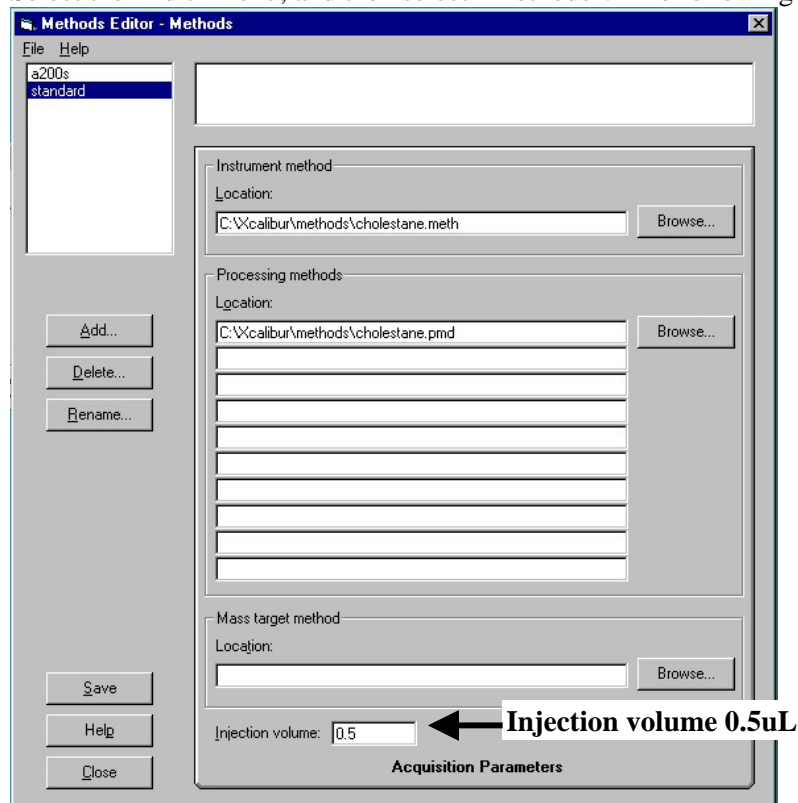


Figure 44 - Xcalibur Open Access Instrument Method Setup



Fill in the fields for the instrument and the processing method files. It is recommended that the “Injection Volume” should be set to 0.5 μ L for most applications. Save the change, and then close the window.

From the “Open Access Instrument Server” window, select Run.

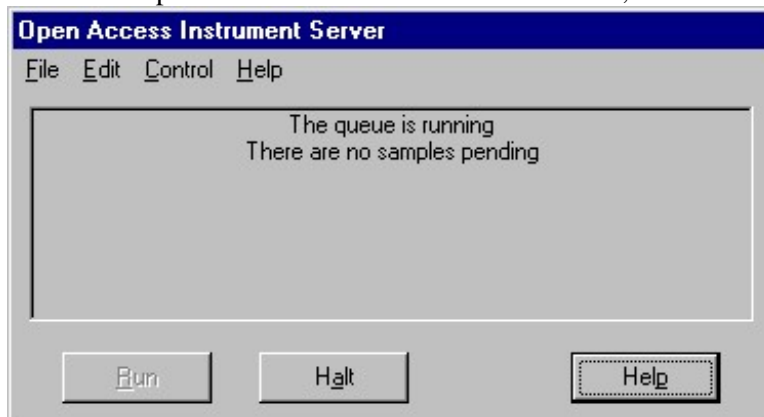


Figure 45 - Xcalibur Open Access Instrument Method Run

Open the “Open Access Login” program from the Windows desktop. The following window appears.



Figure 46 - Xcalibur Open Access Login Window

Set up the “Supervisor” options as outlined in the Thermo Finnigan manual.

To run a sample, activate the “Log in Sample” button in the above window. The following window appears. Fill in the appropriate information as outlined in the Thermo Finnigan



manual to run your sample. To run a single sample press the “Accept Sample” button or to run additional samples press the “Next Sample in Batch” button on this window.

Open Access - Log in a Sample

Entry Number: 1

Vial Position: 3 Use Profile

UserName: Trace

Lab:

Sample ID: chol1

Method Name: standard

Target molecular weight:

Reporting: E-Mail

Next Sample in Batch...

Accept Sample...

Help

Cancel

Figure 47 - Xcalibur Open Access Login Sample Screen

After completing the logging in of samples and pressing “Accept Sample”, the following window appears. Place the samples to be analyzed in the vial positions indicated and select “OK”. The samples will be run, and any reports will be generated. Refer to the Xcalibur and Open Access manuals for preparing reports and having reports printed or e-mailed to Open Access users.

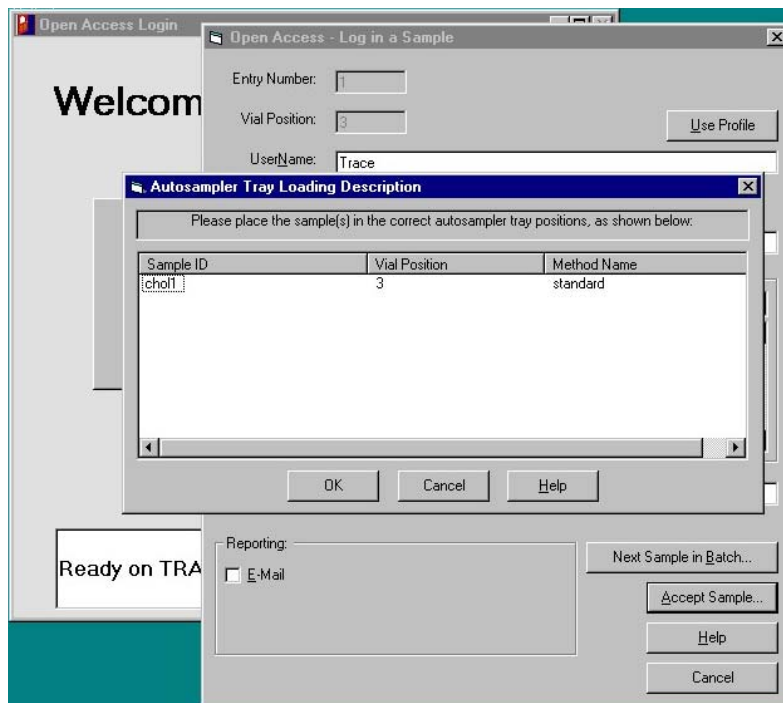


Figure 48 - Xcalibur Open Access Log of Samples



D. Maintenance



Remove the AutoProbe from the MS

The AutoProbe will need to be removed from the MS for one or more of the following reasons.

1. MS Source cleaning or service
2. MS Filament replacement
3. Replacing probe seals in the AutoProbe



The following steps must be followed sequentially to remove the AutoProbe. Be sure the MS turbo and vacuum pumps are turned off and the system is vented before removing the AutoProbe.

1. Turn off the Turbo pump and rough pumps on the MS by following the procedures in the Thermo Finnigan MS manual.
2. Allow the MS source to cool.
3. Turn off the Autoprobe Vacuum Pump.
4. Loosen the Compression Nut on the Inlet Assembly and the AutoProbe MS-Probe Interface Tube (Figure 49).
5. Slide the AutoProbe away from the MS while maintaining the MS and AutoProbe parallel to each other (Figure 50).
6. Remove the Compression Nut, O-ring and compression fitting.

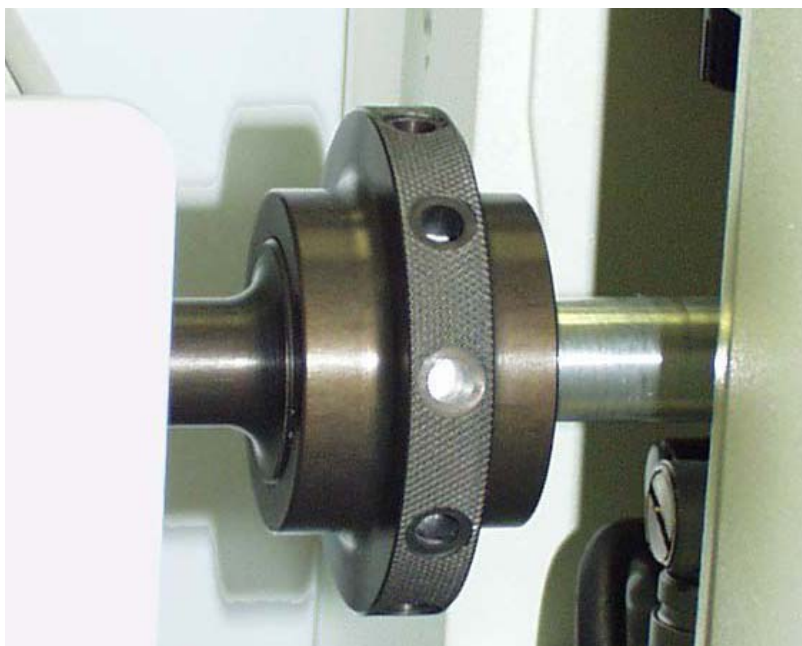


Figure 49 - Compression Nut Connecting AutoProbe to MS

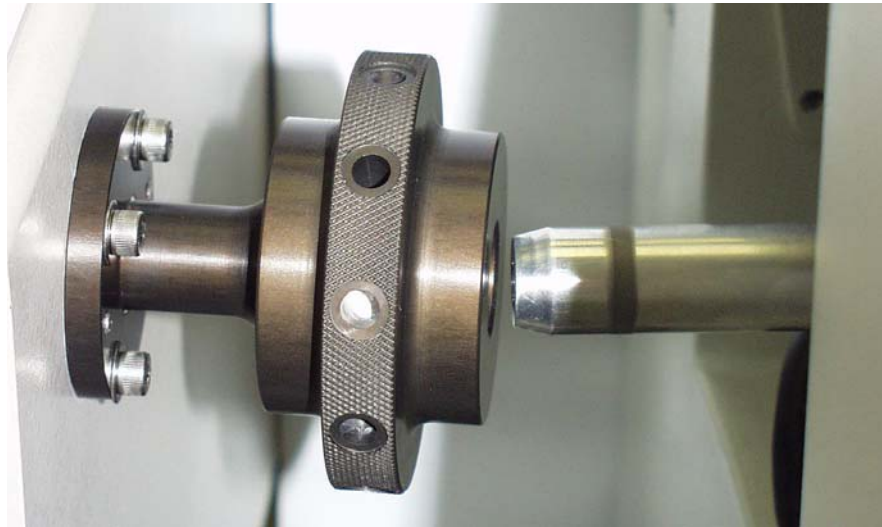


Figure 50 - AutoProbe Disconnected from the MS



Reinstall the AutoProbe to the MS

To reinstall the AutoProbe on the MS follow the instructions on the following pages.

B. Hardware Installation	20
C. Software	34



Replacing the DEP Probe Filament

To replace the DEP Probe Filament tip, grab the metal base of the probe filament with your fingers or a small pair of pliers and slide the tip to the right in the direction of the probe shaft until the plug in leads are clear of the probe shaft.

+



Figure 51 - Replaceable DEP Probe Filament



Figure 52 - Probe Shaft with DEP Probe Filament removed

Install a new filament by inserting the two pins on the backside of the filament into the two receptacles in the end of the probe rod. Push down until the filament is firmly seated against the probe shaft

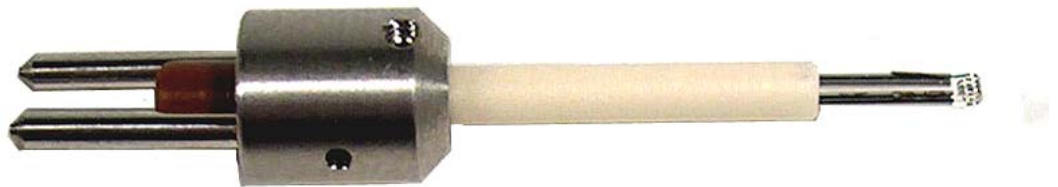


Figure 53 - Plug- In DEP Probe Filament



Removing the AutoProbe Inlet Assembly

The AutoProbe Inlet Assembly consists of the following components:

1. Pneumatic Ball Isolation Valve
2. Probe Sealing System
3. Electric actuated vacuum roughout valve
4. Vacuum pressure sensor flange

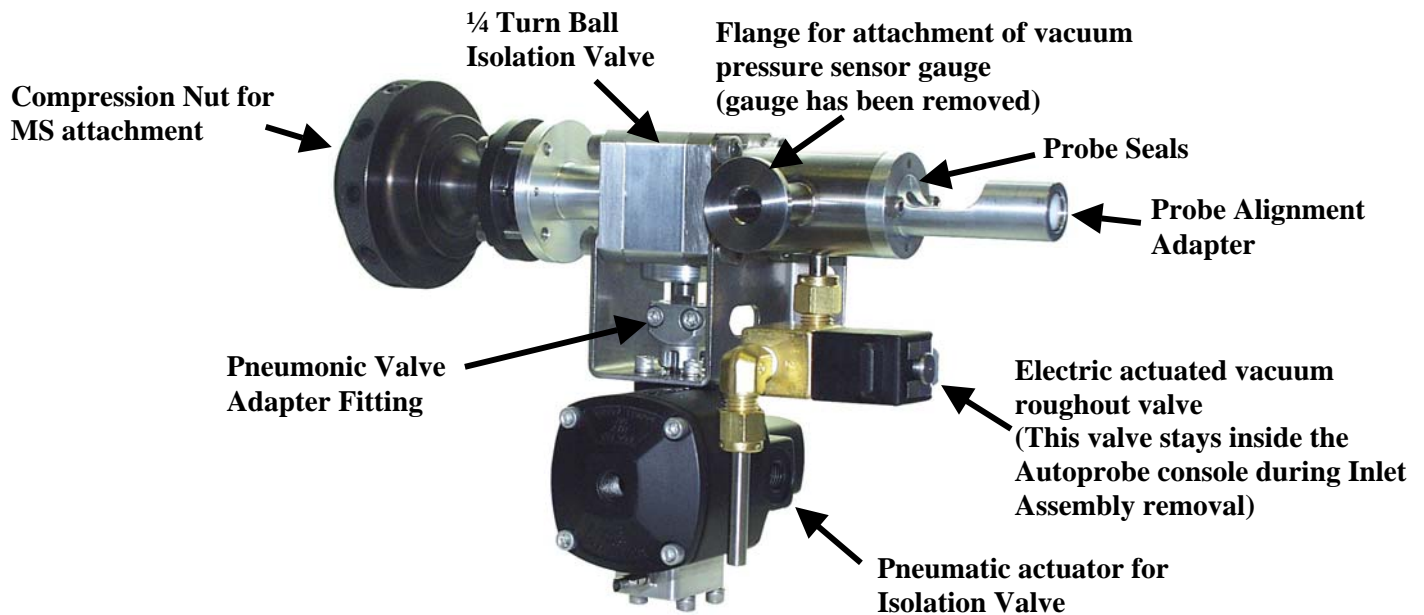


Figure 54 - AutoProbe Inlet Assembly

The AutoProbe Inlet Assembly will need to be removed from the AutoProbe Console for any of the following reasons:

1. To replace the probe seals
2. To replace the seals in the Ball isolation valve
3. To service the vacuum sensor gauge
4. To send out the AutoProbe Inlet Assembly for service or replacement



Before removing the AutoProbe Inlet Assembly, the mass spectrometer must be shut down and vented and the AutoProbe must be disconnected from the MS as described on page 67.

The AutoProbe Inlet Assembly will need to be removed whenever the probe seals or isolation valve ball seals need to be serviced. The operator can either perform this service himself as described in the next section, or the assembly can be returned to SIS for service. A spare AutoProbe Inlet Assembly (which does not include the vacuum pressure sensor gauge) can be purchased from SIS, alternatively, SIS can service your gauge with a 24-hour turnaround time.



Figure 55 - Removing the Inlet Assembly Coupling

After disconnecting the AutoProbe from the MS, remove the AutoProbe Inlet Assembly Coupling on the back panel of the AutoProbe Console as shown above. It is best to first remove the compression nut along with the compression sleeve and O-ring. Next remove the four bolts (#10-32 x 7/8" long) holding the inlet assembly coupling to the back panel using a 5/32 Allen hex driver. Remove the coupling and set aside being careful not to lose the O-ring attached to the backside of the fitting.

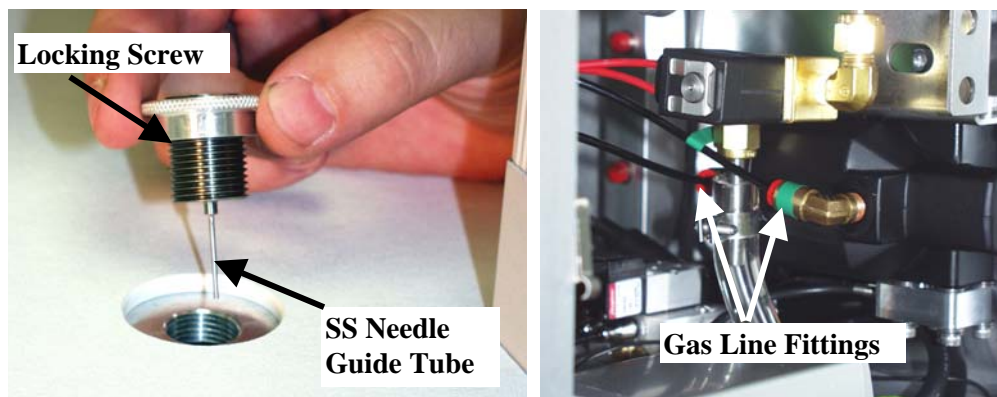


Figure 56 - Removing the Syringe Adaptor and Pneumatic Gas Lines

Remove the Syringe Adaptor from the top of the AutoProbe Console. This is necessary so that the stainless steel needle guide tube is not damaged when the inlet assembly is later removed. First loosen the locking nut and then unscrew the syringe adaptor until it is removed from the console top.

Next remove the compressed air gas lines for the pneumatic ball isolation valve. These lines are accessed from the right rear panel of the AutoProbe Console. Two gas lines must be disconnected. Note that the front gas line and fitting are marked with a green tape. This is for identification purposes. Remove the lines by pushing the red nipple in towards the fitting with one hand and then gently pulling the black gas line out with the other hand.



Electric Actuated Valve

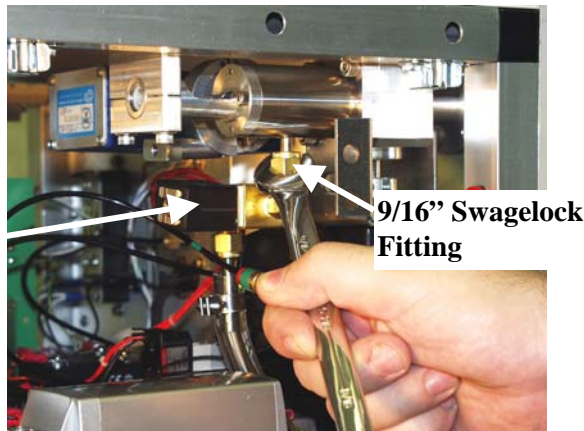


Figure 57 - Removing the Electric Actuated Vacuum Roughout Valve

Next loosen the fitting on the electric actuated roughout valve using a 9/16" open end wrench as shown above. This valve stays inside the AutoProbe Console due to its electrical cable connections. Move the electric valve down and out of the way in order to facilitate the removal of the Inlet Assembly.

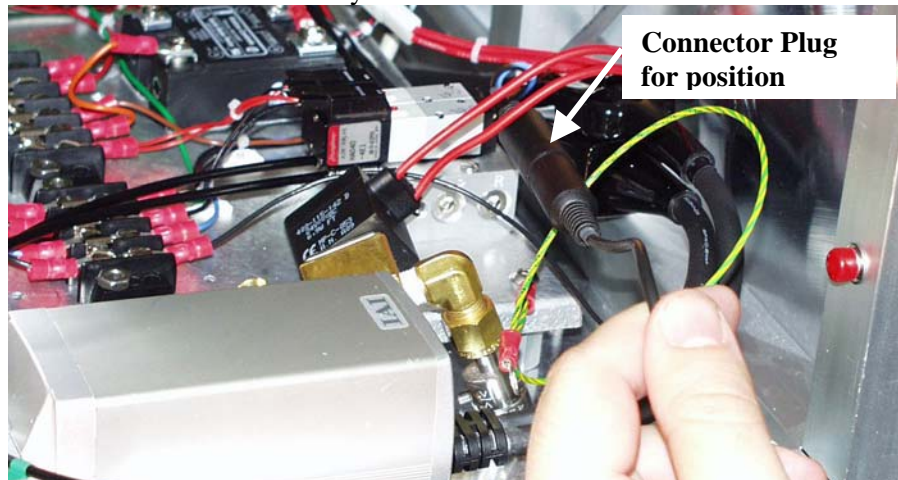


Figure 58 - Disconnect the Pneumatic Valve Position Sensor

Disconnect the pneumatic valve position sensor by unplugging the black connector for this cable. This cable and connectors are located at the bottom of the AutoProbe Console under the Inlet Assembly. It may be hard to get to this fitting and it may be easier to disconnect this cable after the Inlet Assembly has been lowered from its mount but not fully removed from the AutoProbe Console

One additional cable still connects the Inlet Assembly (the electronics connection for the vacuum sensor gauge). However this cable is located on the right side of the vacuum gauge and cannot be disconnected until the Inlet Assembly has been lowered from its mounting position inside the AutoProbe Console.



Figure 59 - Disconnect clamp Holding Inlet Assembly

Finally remove the two screws holding the inlet assembly to the top plate of the AutoProbe Console using a 5/32” Allen wrench. It will be necessary to hold the Inlet Assembly up while removing the two screws and clamp to prevent it from falling down.

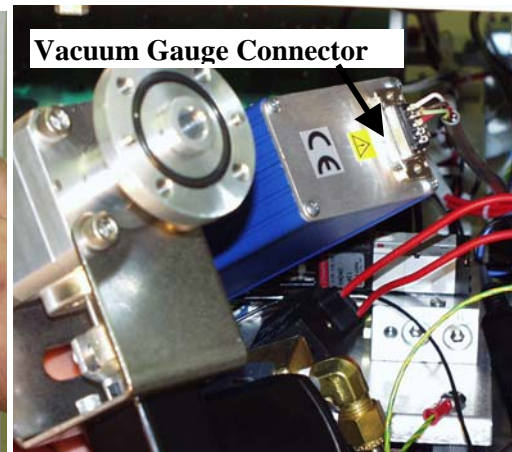
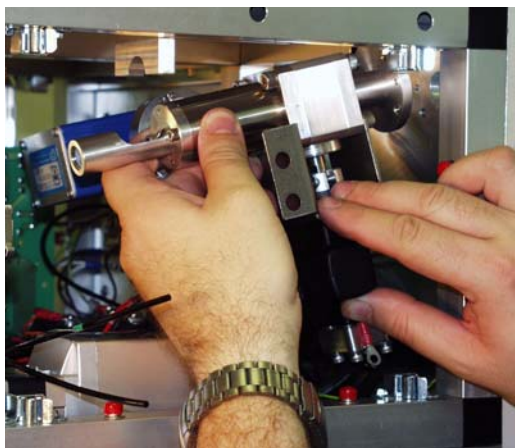


Figure 60 - Lower the Inlet Assembly and Disconnect the Vacuum Gauge Connector Plug

After removing the mounting clamp, carefully lower the inlet assembly and position the inlet assembly to access the vacuum gauge connector plug. Disconnect the vacuum gauge connector plug as shown above. There should be no further cables or lines holding the Inlet Assembly in place. Carefully remove the Inlet Assembly from the AutoProbe Console taking care not to damage any components.

The removal of the inlet assembly is now complete. The Inlet assembly can now be repaired as described below or returned to SIS for service and repair.



Replacing Probe Seals & Isolation Valve Seals – Disassembly

If the probe seals begin to leak, they will need to be replaced. This can either be accomplished according to the procedures outlined below, or, alternatively, the Inlet Assembly can be returned to SIS for restoration of the assembly. Restoration includes replacement of all seals, cleaning of the inlet assembly and leak checking. The procedure for replacing the probe seals and isolation valve seals is fully described below.

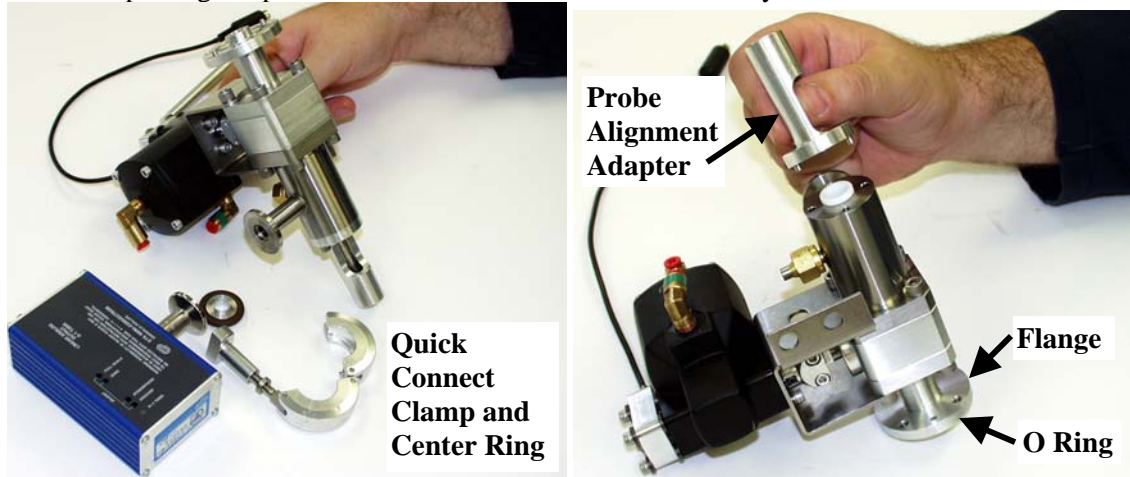


Figure 61 - Removing Vacuum Gauge and Probe Alignment Adaptor

Remove the quick connect clamp holding the Vacuum Gauge on the Inlet Adaptor. Also remove the centering ring. The Inlet Assembly can be returned to SIS for service at this stage of disassembly.

Remove the two screws holding the probe alignment adaptor on the Inlet Assembly using a 9/64" hex wrench. Also remove the O-ring on the flange of the probe alignment adaptor. Next remove the PTFE locating centering bushing by gently sliding out from the Inlet Assembly as shown below.

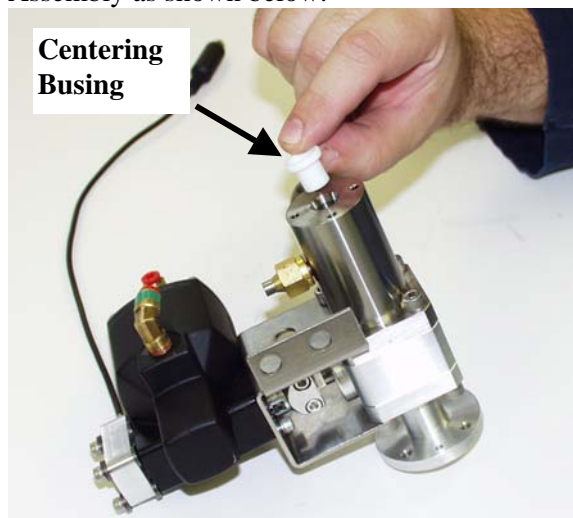


Figure 62 - Removing PTFE Locating Centering Bushing

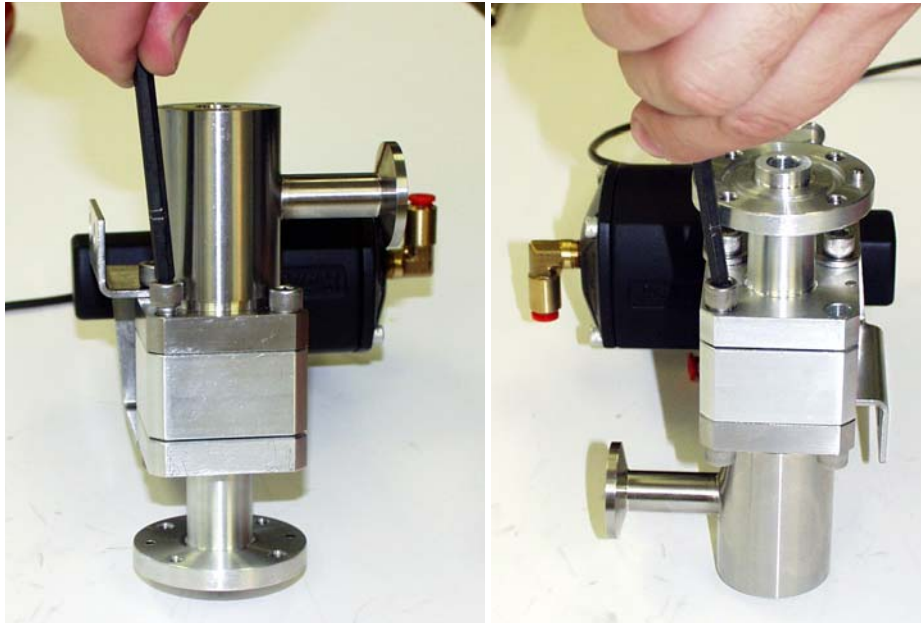


Figure 63 - Removing Screws on Ball Isolation Valve

Remove the 4 screws on both sides of the inlet assembly ball valve using a 3/16" hex wrench as shown above.

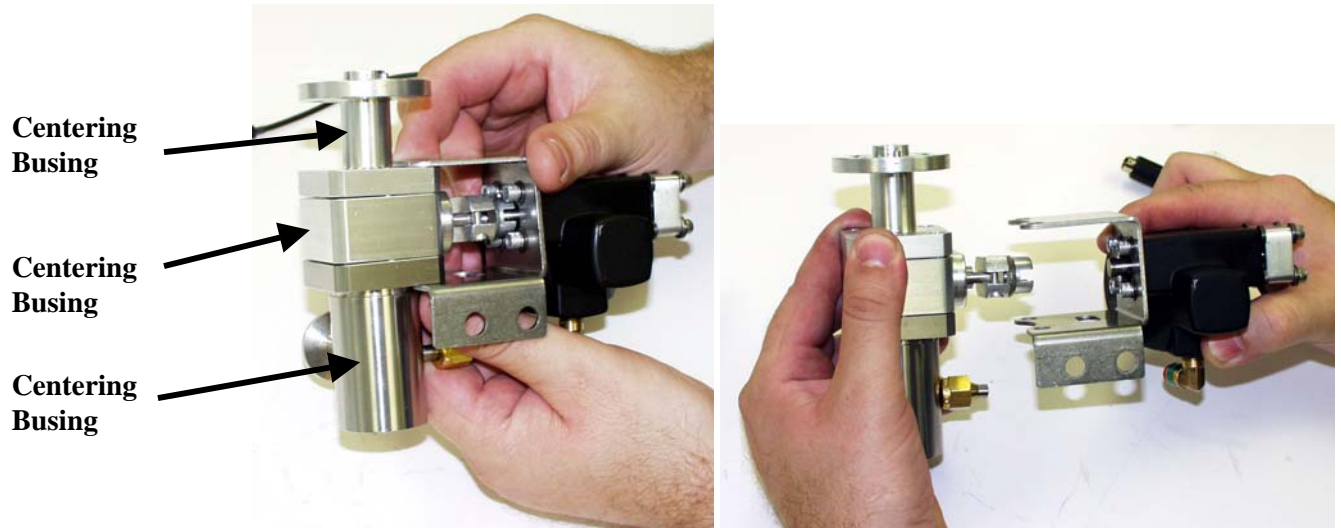


Figure 64 - Removing the Pneumatic Actuator for the Isolation Valve

Slide the pneumatic actuator for the isolation valve away as shown above. The three parts of the Inlet assembly can then be separated for service as shown above (left).

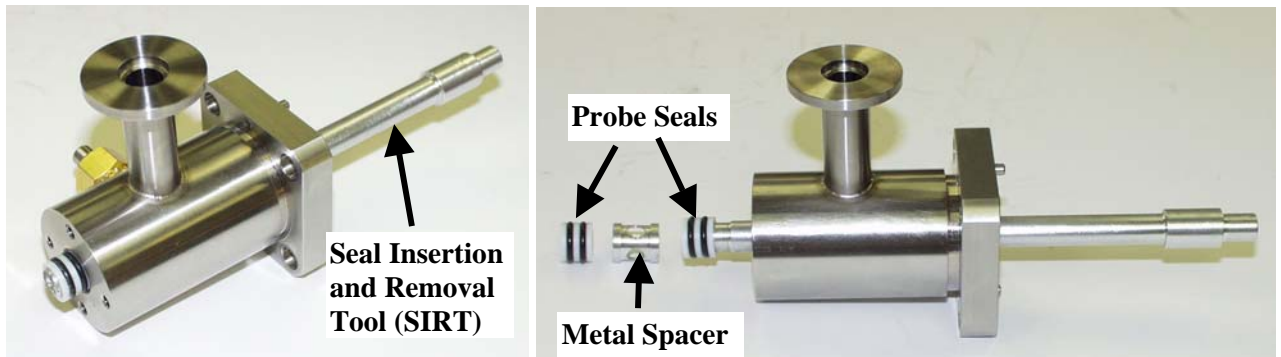


Figure 65 - Removing Probe Seals from the Probe Sealing Chamber

Insert the Seal Insertion and Removal Tool (SIRT) into the flange end of the probe sealing chamber. Gently push the SIRT until the first seal is pushed through the probe sealing chamber as shown above. Continue to push the SIRT until the metal spacer is removed as well as the second probe seal.

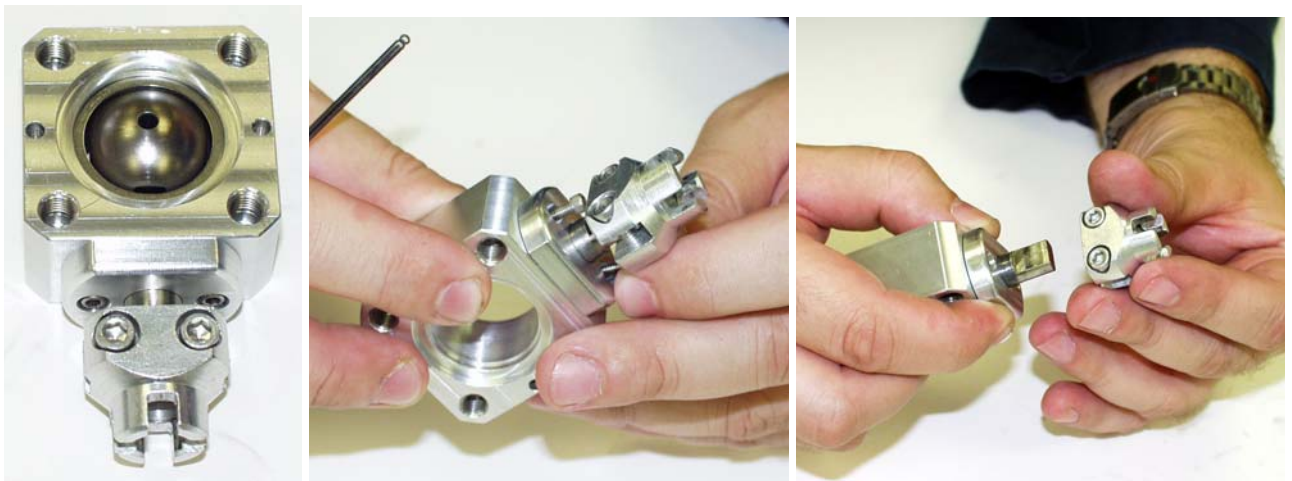


Figure 66 - Removing Pneumatic Valve Adaptor Fitting

Loosen the two hex nuts on the pneumatic valve adaptor fitting and slide away from the ball isolation valve body as shown above using a 9/64" hex wrench.

Note that the steel metal ball from the ball isolation valve has also been removed from the isolation valve body above. In addition the PTFE seals and metal spacers for the ball isolation valve have also been removed. The PTFE seals will be replaced. All other parts will be cleaned and reused as described below.

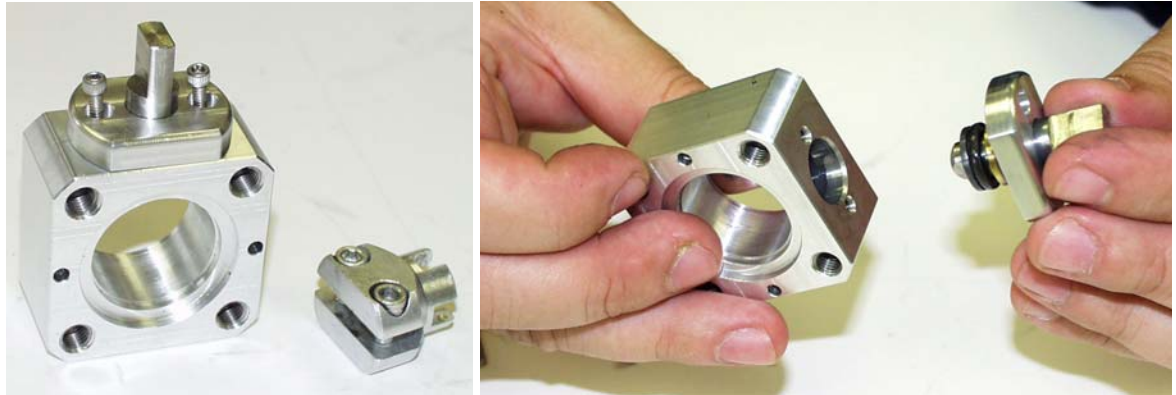


Figure 67 - Removing Valve Stem from Ball Isolation Valve Body

Remove the two screws securing the cap holding the valve stem in place on the ball isolation valve body as shown above using a 3/32" hex wrench. Slide away the valve stem making sure that the Viton seal is not lost on the bottom of the valve stem.

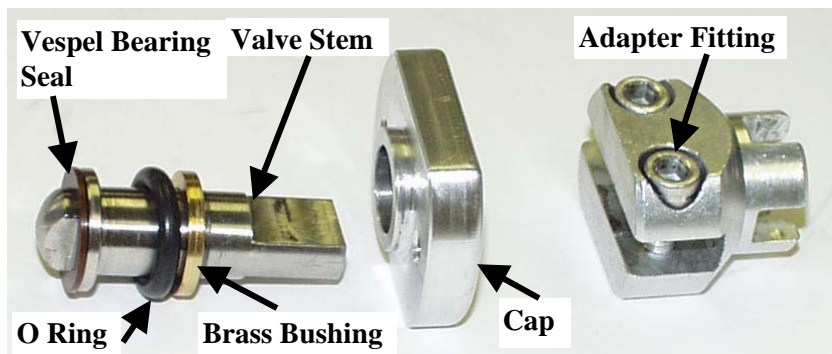


Figure 68 - Disassembly of the Valve Stem for the Isolation Valve

Disassemble the valve stem components including the O-ring, brass bushing and Vespel bearing seal as shown above.

Clean all components from the Inlet Assembly using clean lint free cloths and cotton swabs. The use of solvents is optional. However if solvents are used, then only volatile solvents such as methanol or acetone should be used and all parts should be baked out in an oven at 100 degrees C for 15 minutes before the parts are reassembled. Do not wash O-rings in acetone.

The disassembly of the Inlet Assembly is now complete. After all parts have been cleaned, the reassembly of the inlet Assembly can begin as outlined below.



Replacing Probe Seals & Isolation Valve Seals –Reassembly

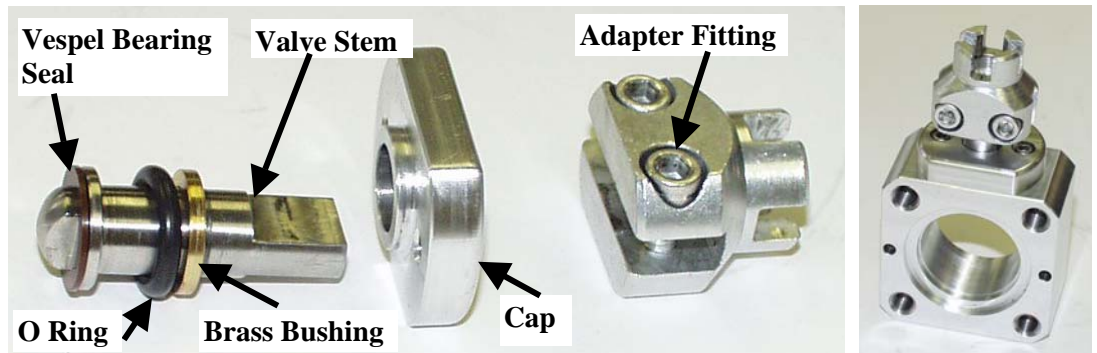


Figure 69 - Reassembly of the Valve Stem for the Isolation Valve

Reassemble the valve stem for the ball valve. Install a new O-Ring that has been lightly greased with Apiezon M grease. Use only a light coating of grease and do not get any grease on the Vespel Bearing Seal or ball surface of the valve stem. Place the brass bushing on top of the O-ring. Also attach the cleaned Vespel Bearing Seal to the surface of the valve stem or if preferred, insert the Vespel Bearing Seal into the opening on top of the ball valve body. Insert the valve stem into the ball valve body, attach the cap and secure in place with the two hex head screws using a 3/32" hex wrench. Be sure that the Vespel Bearing Seal was inserted first. Next attach the adaptor fitting but do not tighten at this time.



Figure 70 - Reassembly of the Ball Valve

Carefully drop the steel ball into the isolation valve body. Make sure that the 1/8" vent hole is facing UP. This vent hole must face towards the probe (opposite side of the MS) when the system is assembled. Next insert the thin metal washer in the groove on the valve body. The driver slot in the ball should engage on the valve stem previously installed into the valve body.

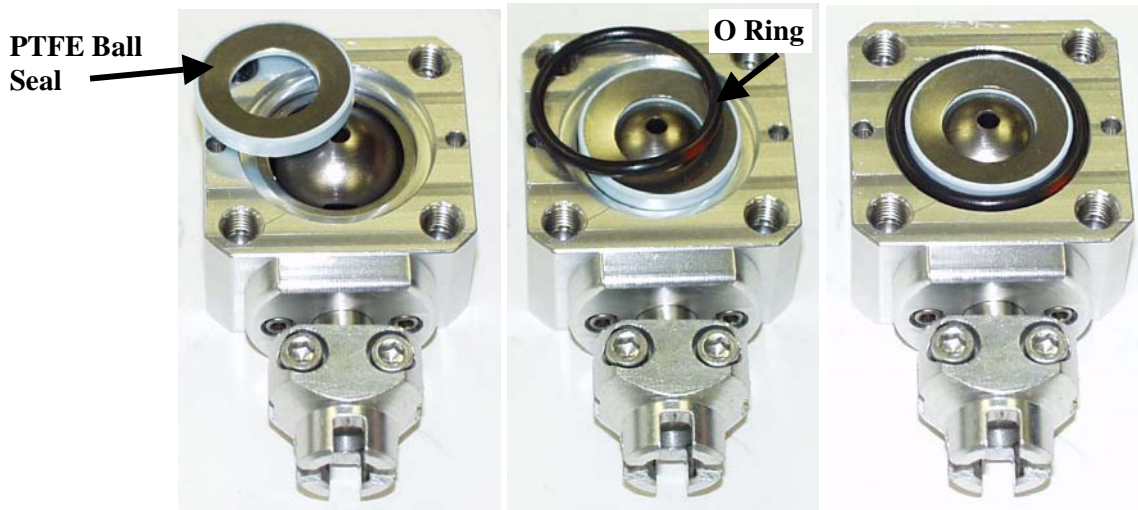


Figure 71 - Reassembly of the Ball Valve Seals

After the metal washer has been installed, install a new PTFE ball seal. Before installing the PTFE ball seal, lightly coat the ball seal with Apiezon M grease. Do not use excessive grease, only a thin coating is needed. Install a clean or new O-ring into the groove on the valve body.

Repeat this operation for the other side of the valve.

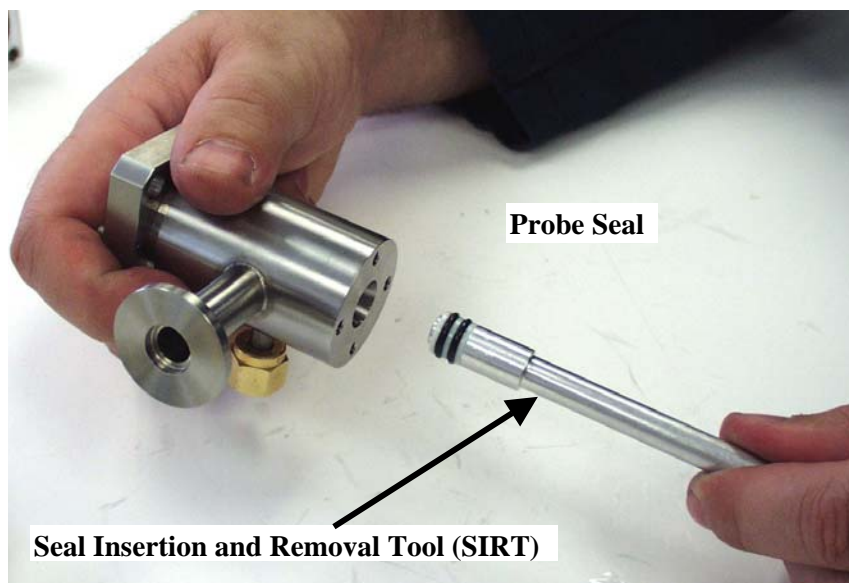


Figure 72 - Installing New Probe Seals

Attach the first probe seal to the Seal Insertion and Removal Tool (SIRT). Coat the outside of the probe seal with a light layer of Apiezon M grease. Then using the SIRT, slowly insert the seal into the probe sealing chamber. Push all the way in until this seal is seated against the stop position inside the probe sealing chamber. Use care when inserting this first seal so



as not to damage the O-rings during insertion. If the seal or O-ring is damaged, the seal will need to be replaced, since it will not seal properly if damaged.

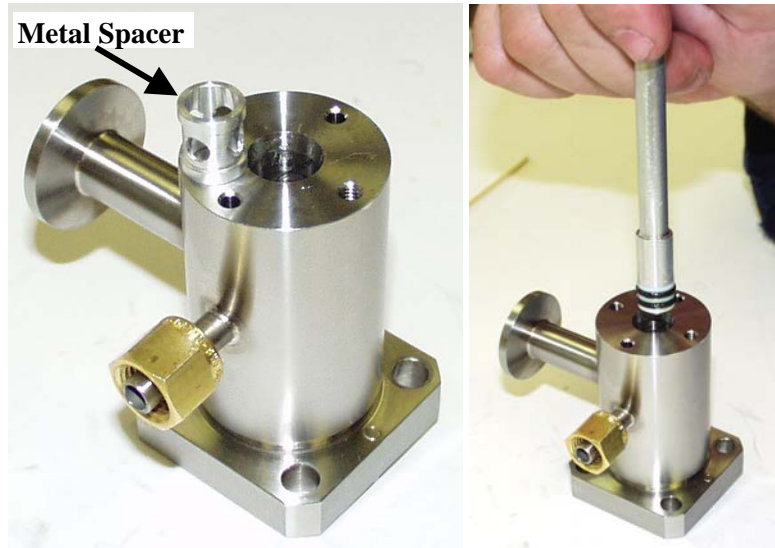


Figure 73 - Finish Installing New Probe Seals

Next drop the metal spacer on top of the first seal in the probe sealing chamber. Finally attach the second seal to the SIRT, lightly coat it with a layer of Apiezon M grease and insert the seal into the probe sealing chamber. Push this seal in until fully seated.

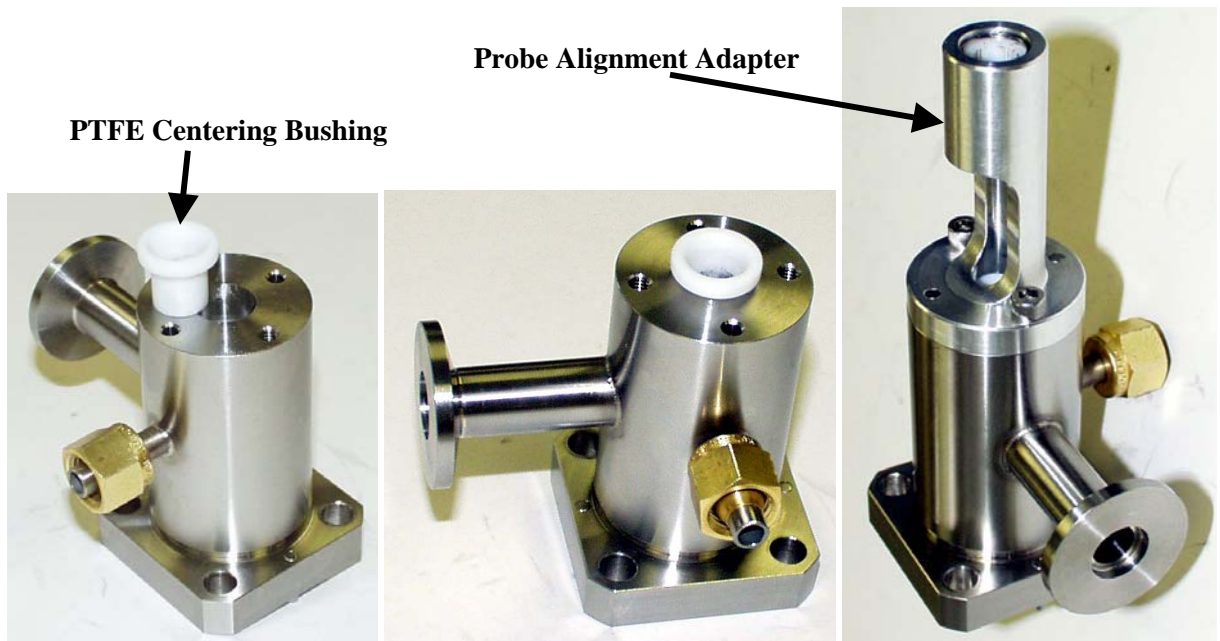


Figure 74 - Install Centering Bushing and Probe Alignment Adaptor



Insert the PTFE locating centering bushing on top of the probe seals in the probe sealing chamber. Attach the probe alignment adaptor on top of the probe sealing chamber and screw in place using the two hex head screws. Note the orientation of the probe alignment adaptor in relation to the fittings on the probe sealing chamber. Do not reverse the orientation.



Figure 75 - Assembly of the Ball Isolation Valve

Complete the assembly of the ball isolation valve. First attach the previously assembled probe sealing chamber with the probe alignment adaptor. Note that this section is attached to the side of the ball valve with the 1/8" vent hole. Note also that the valve stem should be facing the same direction as the fitting for the electric pumpout valve (the 1/4" Swagelok® fitting side). When attaching this first side, install all four bolts to seat the new PTFE seal. It will be necessary to subsequently remove two of these bolts to attach the pneumatic valve below. In a likewise manner, attach the connector tube flange to the other side of the valve body using the 4 screws.

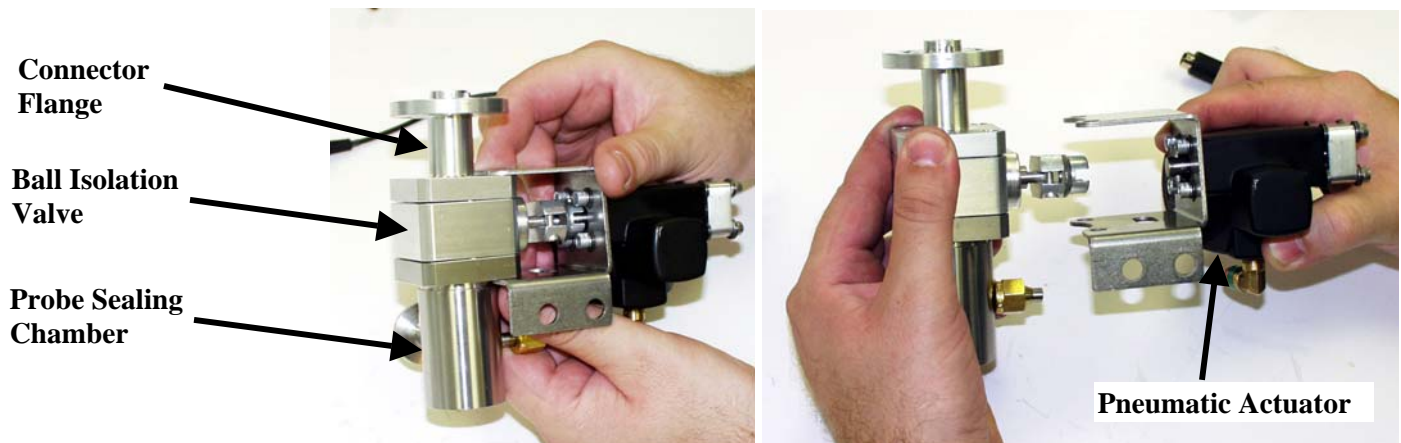


Figure 76 - Attachment of the Pneumatic Actuator

Remove the four bolts (2 on each side facing the 1/4" Swagelok fitting) for the pneumatic actuator. Slide the clamp for the pneumatic actuator in place over top the bolt holes, align the



valve stem adaptor with the key in the pneumatic valve and then replace the bolts in the holes and screw in place.

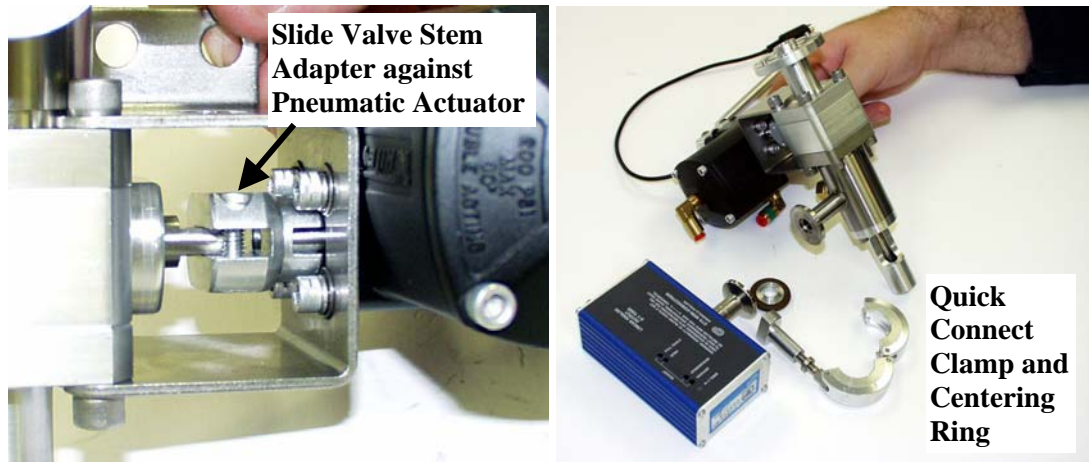


Figure 77 - Attachment of the Valve Stem Adaptor and Installation of the Vacuum Gauge

Slide the valve stem adaptor snugly against the shaft and alignment pin on the pneumatic actuator. Then lock in place with the two hex head screws on the valve stem adaptor. These screws can be accessed via the slot in the mounting bracket for the pneumatic valve.

Reattach the vacuum gauge to the flange on the inlet assembly using the centering ring and quick connect clamp. Align the gauge horizontally as previously positioned with the connecting fitting facing towards the back.



Reinstallation the AutoProbe Inlet Assembly

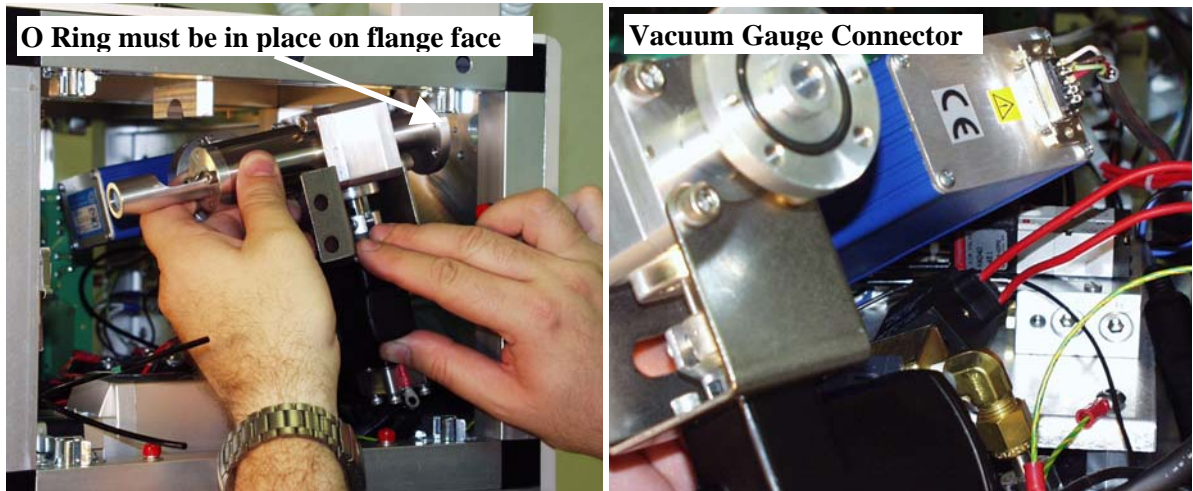


Figure 78 - Installation of AutoProbe Inlet Assembly

After the Inlet Assembly has been serviced and repaired, it can be reinstalled in the reverse fashion it was removed previously.

First position the Inlet Assembly inside the AutoProbe Console as shown above. Before installing, attach the vacuum gauge connector plug onto the end of the vacuum gauge sensor.

Make sure that the O-ring is in place on the flange face of the Inlet Assembly. This O-Ring is needed to make a vacuum tight connection to the MS. Slide the Inlet assembly into place and then attach the clamp with two bolts to hold the Inlet Assembly in place. Do not tighten these bolts at this time.

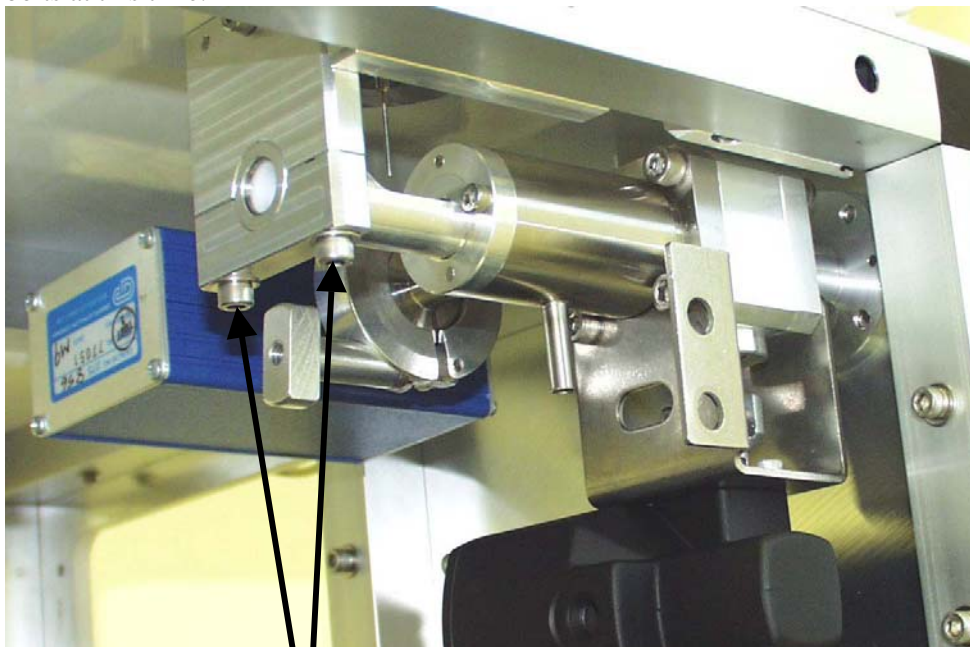


Figure 79 - Attachment of Inlet Assembly with clamp and bolts

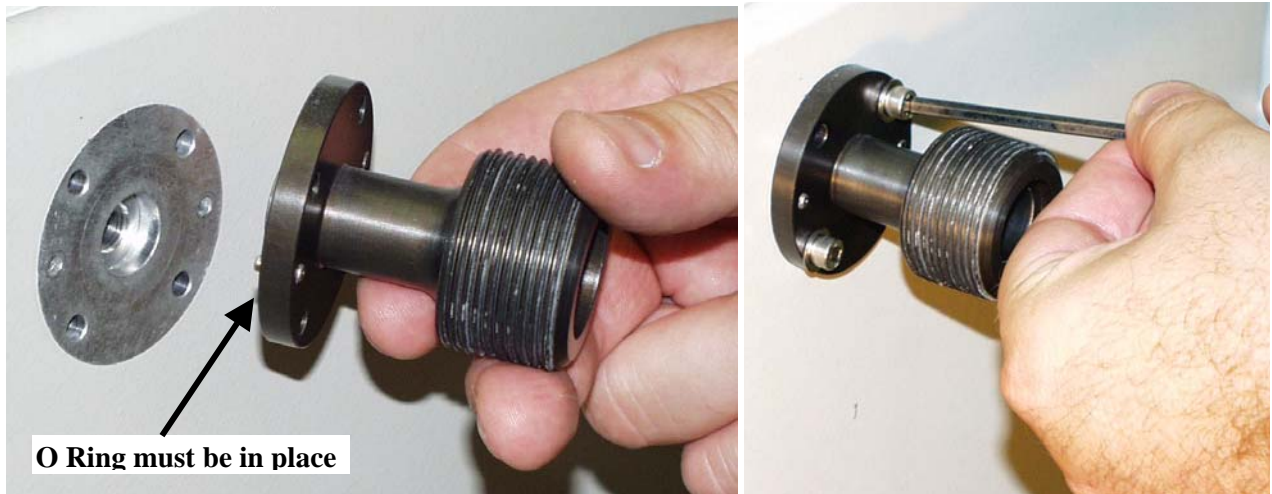


Figure 80 - Attachment of Inlet Assembly Coupling

Next attach the inlet assembly coupling to the outside back panel of the AutoProbe Console. Make sure that the O-ring is in place on the back side of this fitting before attaching. This O-ring is needed to make a good vacuum seal to the MS. Attach the 4 bolts and tighten snugly.

Next go back and tighten the two bolts on the clamp holding up the inlet assembly inside of the AutoProbe Console (Figure 79).

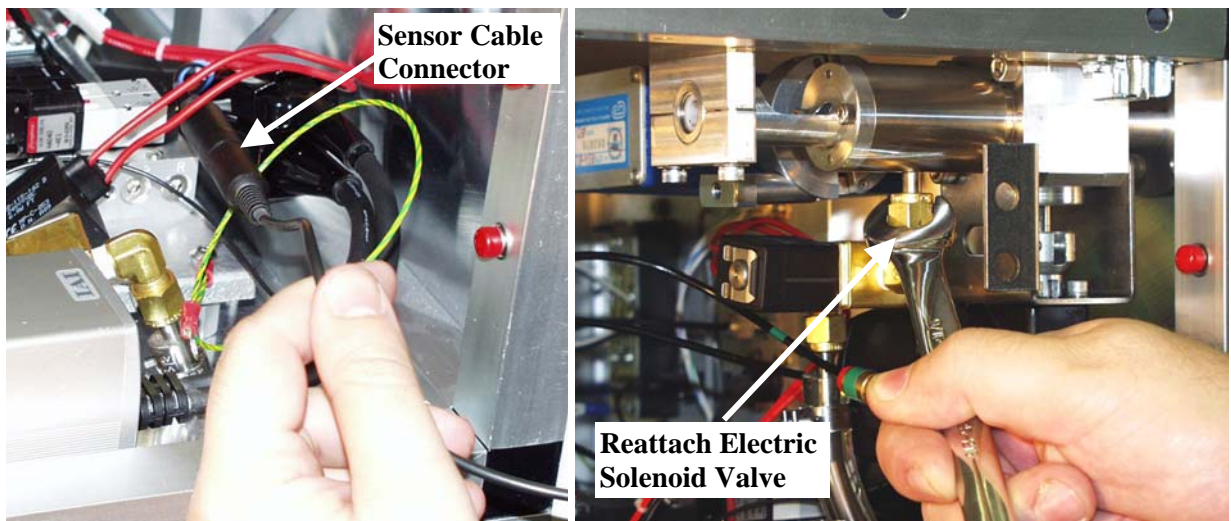


Figure 81 - Attachment of pneumatic sensor cable and electric solenoid vacuum valve

Reattach the sensor cable connectors as shown above. Plug the two black connector plugs together. This sensor is needed to determine if the isolation valve is open or closed.

Reattach the electric vacuum solenoid valve to the 1/4" Swagelok fitting. This valve provides for the vacuum evacuation of the probe inlet seal system.

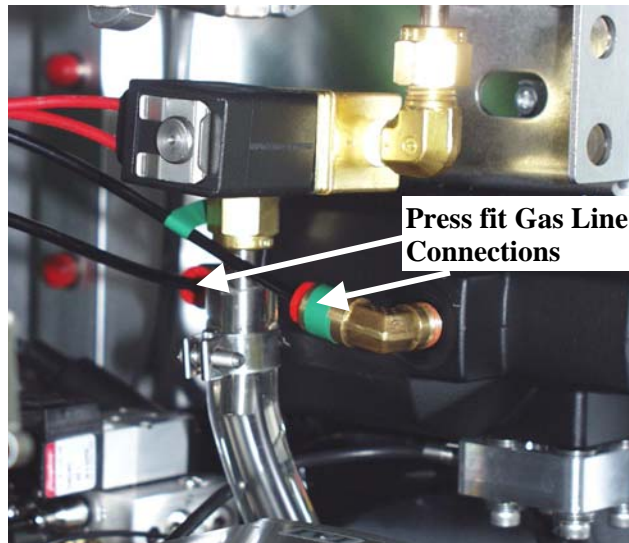


Figure 82 - Attachment of pneumatic gas lines

Reattach the compressed air lines by pushing the 1/8" nylon tubing into the fittings on the isolation valve. The front fitting is marked green and the corresponding cable with green tape should be attached to this fitting. These lines are connected with push on fittings. Just push the 1/8" nylon tubing into the fitting until firmly seated. The tubing should stay in the fitting and not come loose.

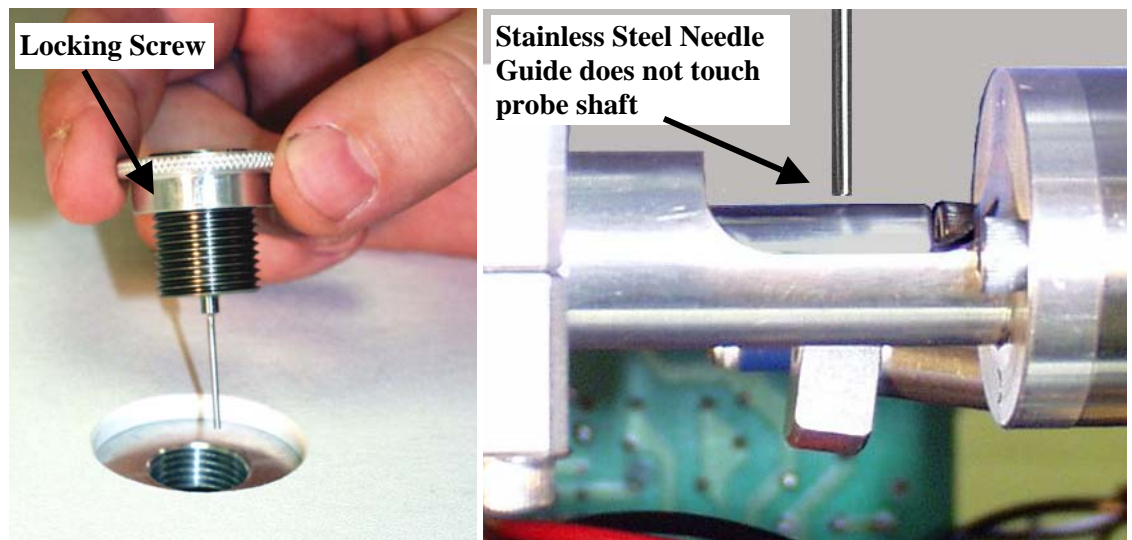


Figure 83 - Attachment and Alignment of Injector Needle Guide

Attach the injector needle guide by screwing the fitting into the top of the AutoProbe Console as shown above. Manually move the probe rod into position through the probe alignment adaptor. Screw the needle guide down until the stainless steel needle guide just clears the probe rod. This guide should not touch the probe rod, otherwise scoring of the probe rod will occur. Lock the needle guide in position using the locking screw on the needle guide.

The installation of the Inlet Assembly is now complete.



Vacuum Pump Maintenance

A rotary vacuum pump is required for the evacuation of the isolation valve on the AutoProbe. A vacuum gauge in the isolation valve measures the pressure between the two probe seals and when this vacuum gets below 200 millitorr, the rotary ball isolation valve opens.



Figure 84 - Rotary Pump for the evacuation of the pump isolation valve

Vacuum Pump Oils



For optimum life of the rotary vacuum pump, the pump oil should be drained and replaced every 3 months.

If the isolation valve fails to achieve a low enough vacuum to activate the rotary ball isolation valve, the problem may be due to the vacuum pump. However the most obvious cause of a poor vacuum is leaking at the probe seals. It is recommended that the probe seals in the isolation valve be changed before the vacuum pump is suspected as the cause of the problem.



Fuse Replacement

The power module on the back of the AutoProbe Console contains a fuse. If this fuse needs to be replaced, use a standard 10-amp slow blow fuse.



Moving Probe Against MS Source

In some instances it may be necessary to move the probe against the MS source in order to seal the source tightly. This is required for CI operations, where the source pressure must be maintained at a high pressure with the CI reagent gas. It is also normally recommended to tune the MS with the probe against the source in the EI mode.

To move the probe against the source, use the following procedures:

1. Place a DEP filament at the end of the probe. (Do **NOT** use the Source Position Calibration Tip - this will ground out the MS source.)
2. Start the AutoProbe Manual Control Mode from either the Windows desktop or Windows taskbar Start menu. The following window appears:

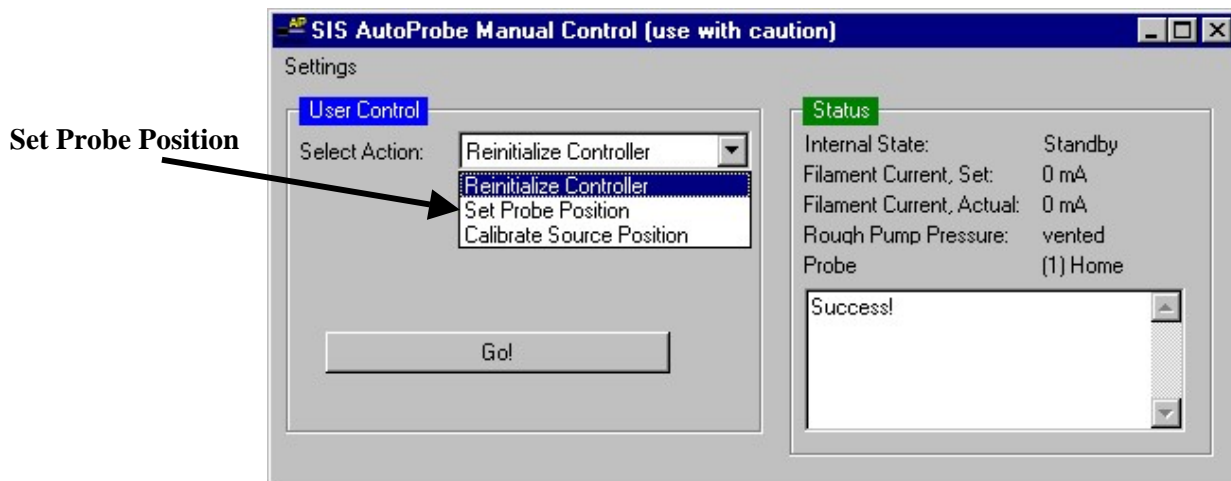


Figure 85 - Manual Control Window

3. Select the Set Probe Position and then select “Source”

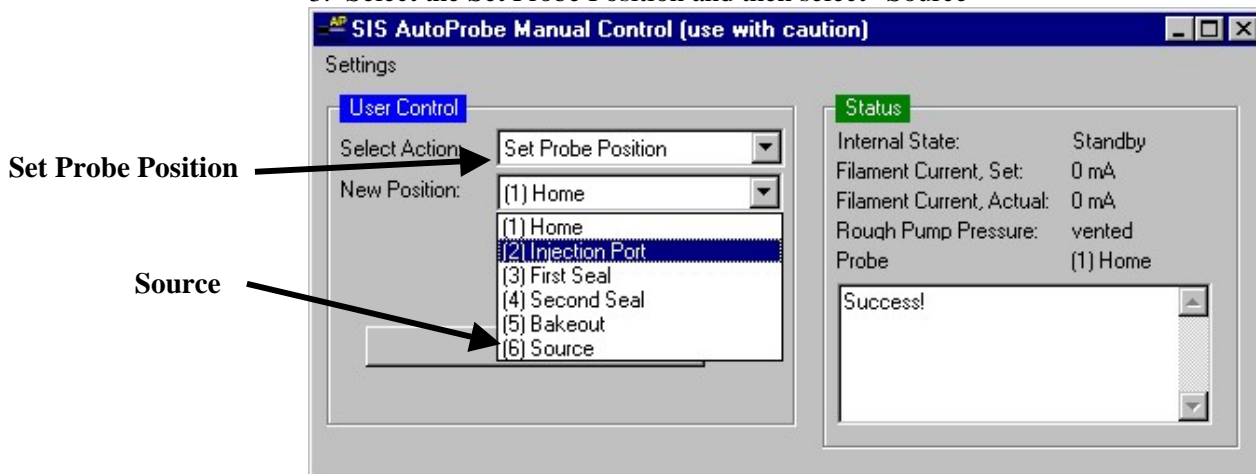


Figure 86 - Screen to position probe against MS source

4. Finally select “GO.” The probe will position itself against the source and remain there until manually removed.



Replacing "O" Rings in the AutoProbe Transfer Line

In some instances it may be necessary to replace the two Viton "O" Rings in the AutoProbe Transfer Line. These seals provide the seal against the probe shaft to permit the CI gas to properly pressure the source when the mass spectrometer is used in the CI mode. If the "O" Rings begin to leak, the CI reagent gas will leak into the vacuum housing instead of being directed into the mass spectrometer source. The 2 Viton "O" Rings are located underneath the Large and Small Cover Plates in the AutoProbe Transfer Line Assembly, which must be disassembled to replace these seals. If the mass spectrometer is used in the EI mode, these seals are not critical to the performance of the mass spectrometer.

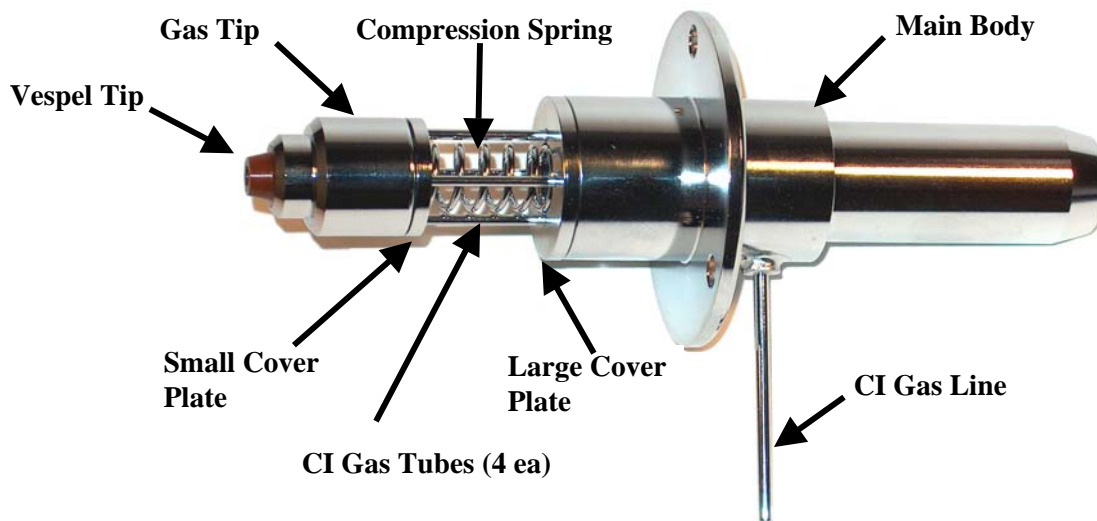
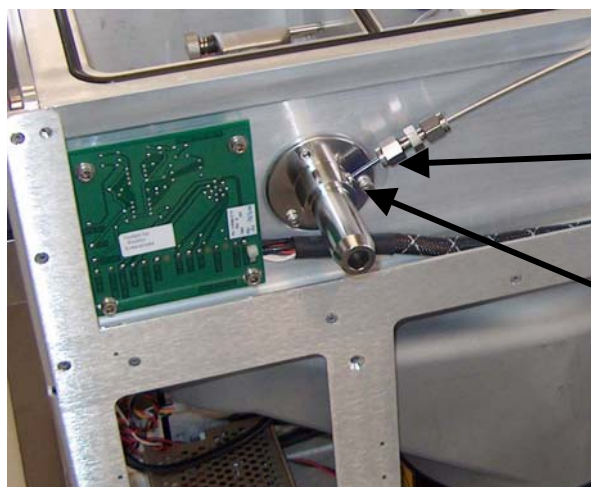


Figure 87 - AutoProbe Transfer Line

Begin the operation by removing the AutoProbe transfer line from the mass spectrometer. Remove the three allen hex screws using a 2 mm allen hex driver and also remove the 1/8" Swagelok nut which attaches the CI reagent gas line.



Loosen Swagelok nut to disconnect CI gas line from the transfer line

3 allen hex screws hold the transfer line onto the vacuum manifold

Figure 88 - Remove the AutoProbe Transfer Line from the mass spectrometer.



The components of the AutoProbe Transfer line are not physically attached together. They are held together by the force of the compression spring when the AutoProbe transfer line assembly is attached to the MS housing and clamped in position against the mass spectrometer source. Therefore, when removing the transfer line assembly use care so that the transfer line parts do not come apart and fall to the floor.

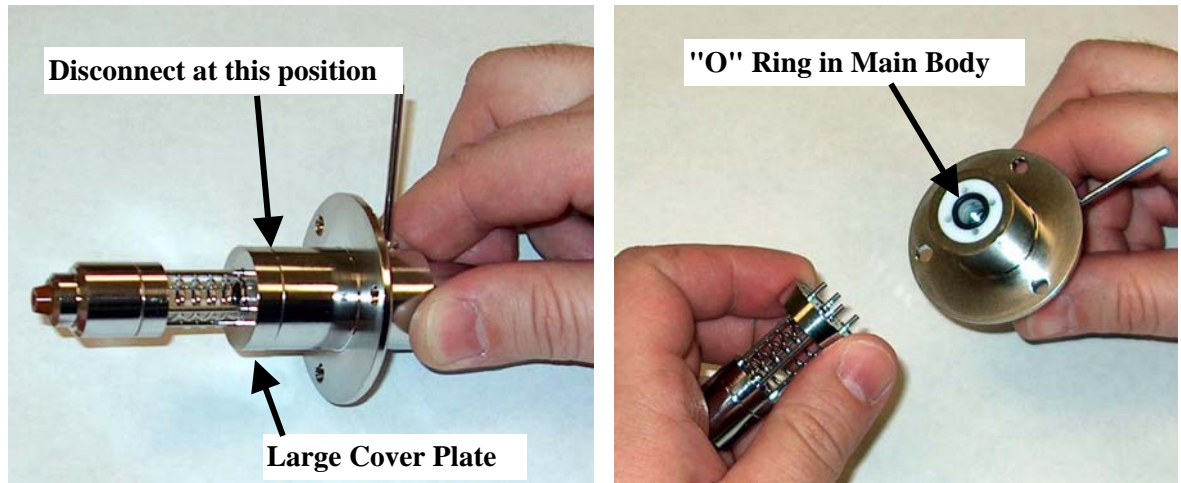


Figure 89 - Initial disassembly of the Transfer Line

Begin the disassembly by disconnecting the Main Body of the transfer line from the Large Cover Plate. The parts should easily slide apart. The first "O" Ring seal is then easily visible on the Main Body assembly.

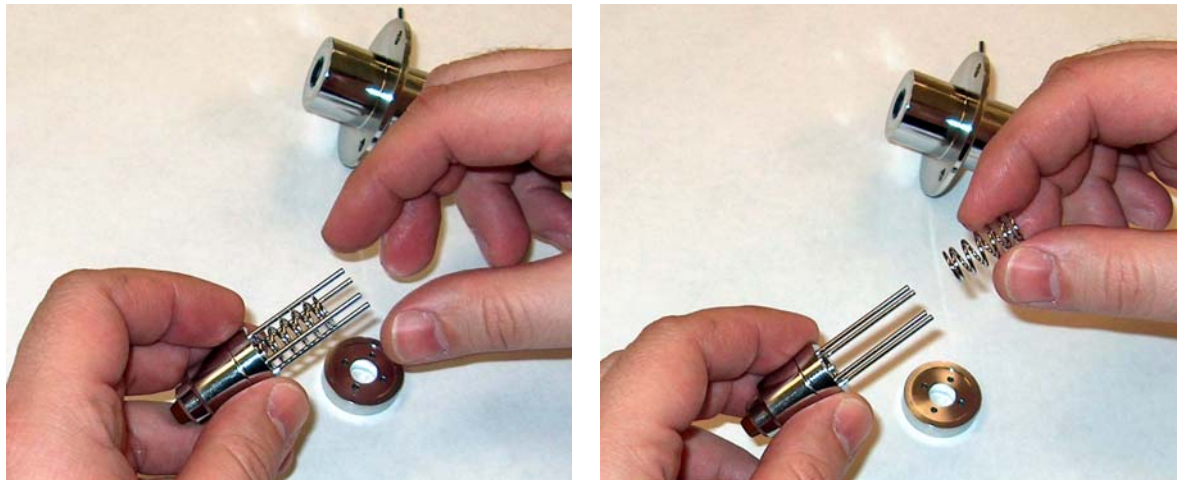


Figure 90 - Removing Compression Spring from Transfer Line

Continue the disassembly of the transfer line by removing the Large cover plate by sliding out over the CI gas lines. It should slide out easily over these tubes. Then remove the Compression Spring.



Figure 91 - Removal of cover Plate to access the "O" Rings in the Transfer Line

Next, remove the small cover plate by sliding it along the CI Gas Tubes to access the 'O' Ring in the Gas Tip. Both "O" Rings should now be easily visible - one in the Gas Tip and the second in the Main Body.

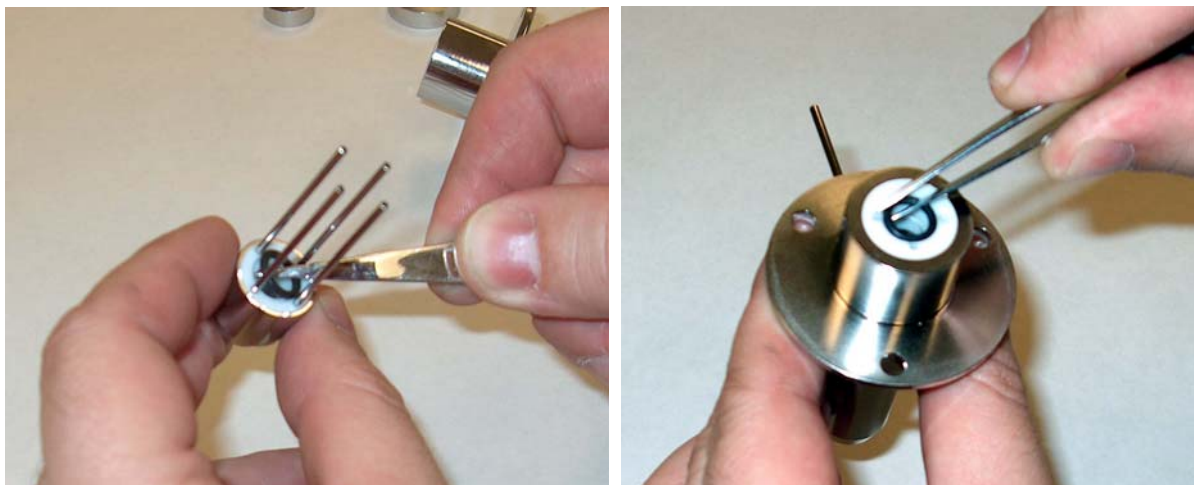


Figure 92 - Removal of "O" Rings in the Transfer Line

Remove and replace the two "O" Rings in the transfer line. The "O" Rings can most easily be removed using a pair of forceps. Replace the "O" Rings with two new Viton "O" Rings (Part Number V-011). Use care so as not to scratch the "O" rings and be sure the "O" rings are firmly seated in the two grooves in the PTFE sleeve in both the Gas Tip and the Main Body of the transfer line assembly.

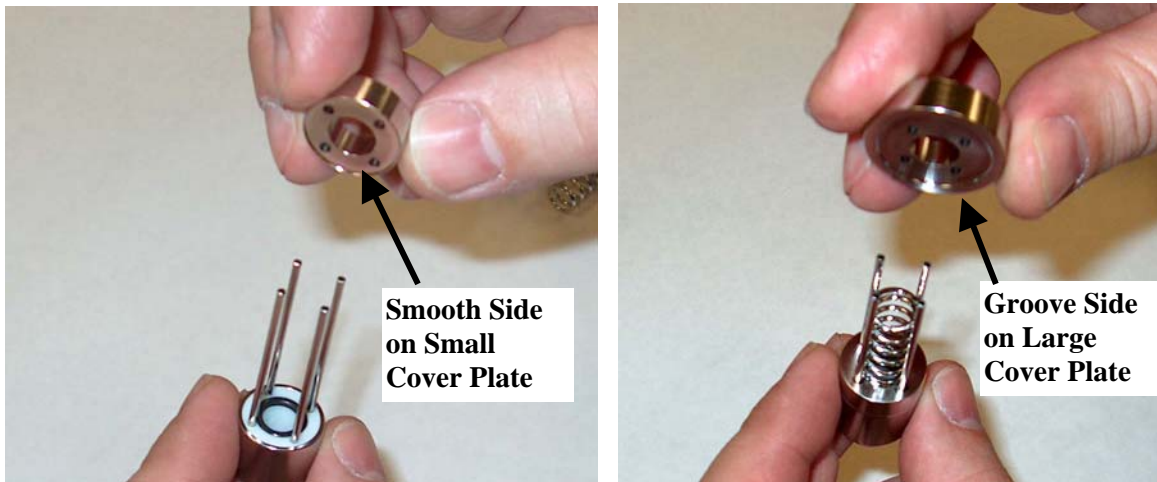


Figure 93 - Reassembly of the Transfer Line

Reassemble the transfer line in the reverse order that it was disassembled. First slide the Small Cover Plate over the CI Gas Tubes to cover the "O" Ring. Note that the sides of the Small Cover Plate are different. One side has a small groove. This groove is used to hold the Compression Spring in place, so be sure that the groove side of the Small Cover Plate is in the direction of the Compression Spring. Next insert the Compression Spring followed by the Large Cover Plate. Again note that the side of the Large Cover Plate with the small groove must be in the direction of the Compression Spring.

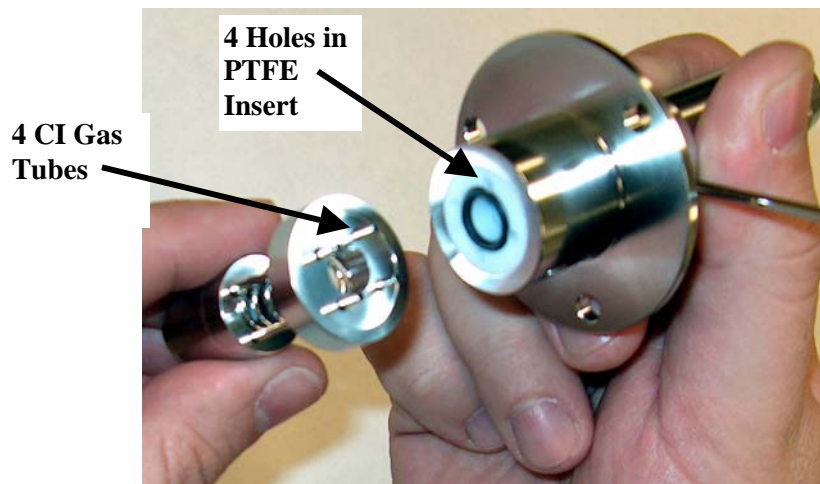


Figure 94 - Final Assembly of the Transfer Line

Finish the assembly of the transfer line by sliding the 4 CI Gas Tubes into the 4 holes in the PTFE insert in the Main Body of the transfer line assembly. Reinstall into the mass spectrometer using care that the parts do not come apart during the assembly. When the Transfer Line is installed into the vacuum housing and screwed into place, the compression spring holds the assembly in place between the vacuum housing and the mass spectrometer source.



E. Troubleshooting



Error Messages

This description describes various error messages you may encounter.

“The source position must be recalibrated.”

You must perform the source calibration procedure mentioned in page 43.

“The inlet could not open (close) or the sensor did not detect it to be open (closed).”

Determine if the isolation valve is actually opening (or closing) by temporarily removing the side panel near the injection port and visually observing whether the isolation valve fully opens (or closes). If the valve doesn't fully switch position, then there could be a problem in the air pressure or resistance in the isolation valve itself. If it does switch position, then there is probably a problem with the sensor (likely a misalignment problem that prevents the sensor from being triggered at the correct time). A misalignment problem is indicated by the sensor light flashing on while the valve is opening but then turning back off once the valve fully opens.

“The rough pump pressure was not obtained.”

The vacuum inside the isolation valve was not sufficient for injection. Typically, the required vacuum pressure is 200 mTorr, although this value may be adjusted in the AutoProbe Instrument Configuration window. Check that the rough pump is on and working properly and that there are no leaks. A leak could be caused by worn out probe seals. Seals are expected to last 2000-3000 injections.

“Communications lost.” / “Could not retrieve controller status.”

Ensure that the PC's serial communications port that is connected to the AutoProbe is configured correctly, that the cable is connected, and that the AutoProbe is on. Ensure that the “serial communications port” setting in the AutoProbe Instrument Configuration window matches the actual port that the AutoProbe is connected to. The Instrument Configuration window has a Test button to test each serial port for connectivity. Ensure that the serial port works. You can do this by testing another instrument on that serial port.

“... bad checksum ...”

There was interference in the communication between two devices. If the problem persists, remove any possible cause of electric interference or call SIS.

“Unknown actuator error 130”

The AutoProbe may be trying to recover to the home position but for some reasons already be at the home position. Turn the power off and manually push the arm forward a bit. Then turn the power back on and reattempt moving to the home position.



Fault Light

The fault light (red light) on the front of the AutoProbe Console may come on during an AutoProbe run. A corresponding error message will be displayed on the AutoProbe status panel of the Xcalibur Home Page and in the status panel of the AutoProbe manual control window.

Possible Causes:

- Filament Burnout
- Isolation valve does not pump down to the required vacuum in the allotted time.
 - o Probe seals are leaking
 - o Vacuum pump not turned on not operating poorly
- A communications glitch
- If the isolation valve does not open or close
- If the probe does not hit the source during source probe position calibration



CTC PAL System Errors

“GET_SAMPLE(...): error: Object not found.”

This indicates that some setting/item does not exist or is not properly configured on the PAL. It may be caused by either an invalid parameter on the PC (such as an invalid/nonexistent tray or vial name) or a misconfigured setting on the PAL’s hand-held controller (such as a misconfigured tray, syringe, wash station, injection port, etc.). Ensure that the PAL is configuration correctly, and then insure that these settings are in sync with the PC by performing the “reload PAL configuration” function from the PAL section of the Xcalibur Instrument Configuration window.



F. Appendix



Consumable Parts and Supplies

The following is a list of the parts that may be expected to be in need of replacement on the Model AP2000 AutoProbe. The quantity of these units needed per year, depends on the number of samples analyzed on the AutoProbe.

Part #	Description	Required in the System
AP20001	AutoProbe Filament Assembly.	2
AP20002	AutoProbe Calibration Tip	1
AP10003	AutoProbe Shaft Seal Set, package of 2	1 Spare Set
AP10004	AutoProbe Isolation Valve Seal Set	1 Spare Set
V-011	Viton "O" Ring for Transfer Line Seal	2
28406201	Ion Volume , AP2000 - CI	1
28406202	Ion Volume, AP2000 - EI	
FK251	Vacuum Pump Exhaust Filter Kit	1
KA-1	Koby® Charcoal Replacement Filter	1

The isolation valve will need to be serviced after 2000 or more sample analysis. This service includes replacement of the seals listed above and cleaning of the isolation valve as described in the Maintenance section of this manual. The user may wish to service the isolation valve himself or return it to SIS for 24-hour turnaround service. As an alternative the user may wish to purchase a spare isolation valve to avoid down time due to the time required to service this component. The following parts and services relating to the isolation valve are available from SIS:

AP20005	AutoProbe Isolation Valve Assembly (A complete isolation valve for replacement into your AutoProbe—does not include vacuum gauge or electronic solenoid valve)
AP2000C	AutoProbe Isolation Valve Cleaning and Seal Service (Send in your Isolation Valve and it will be serviced and repaired within 24 hours.)



Trademarks

The following list is an attempt to give recognition to the trademarks and their respective owners.

AutoProbe	Scientific Instrument Services
DEP Probe	Thermo Finnigan (Finnigan)
DSQ	Thermo Finnigan (Finnigan)
PAL	CTC Analytics
PolarisQ	Thermo Finnigan (Finnigan)
Teflon	E. I. Dupont
TRACE MS	Thermo Finnigan (Finnigan)
Thermo Finnigan	Thermo Finnigan (Finnigan)
Vespel	E.I. Dupont
Viton	E.I. Dupont
Windows	Microsoft
Xcalibur	Thermo Finnigan (Finnigan)



Additional Notes

The PAL Backdoor Feature.

A backdoor feature is available on the PAL system that permits you to configure parameters that are otherwise not visible in the PAL keypad menu. During factory installation, SIS has made some configuration changes via the backdoor feature. For details on the backdoor feature and its functionality, contact LEAP Technologies or SIS. Be aware that incorrect parameters set with the backdoor feature can potentially damage the system.

Terminating unresponsive processes.

A given software component may fail to respond to user input due to a misconfiguration or an obscure system state. If any software component fails to respond, a dependable way to fix this is to reboot the computer. However, a quicker and often equally effective method is to kill individual computer processes (programs) by holding down the CTRL-ALT-DELETE keys simultaneously, selecting Task Manager, selecting the Processes tab, selecting the name of the process to kill, and then selecting End Process. Below is a list of AutoProbe, PAL, and Xcalibur related processes. The exact list may depend on your version and configuration of Xcalibur.

cyc_comp.exe	Cycle Composer
palvi.exe	PAL Driver for Xcalibur
TRACEM~2.EXE	TRACE MS Driver for Xcalibur
ACQSER~1.EXE	Xcalibur acquisition server
QUADInst.EXE	DSQ Driver for Xcalibur
HomePage.exe	Xcalibur Home Page
InstSetup.exe	Xcalibur Instrument Setup
ProcSetup.exe	Xcalibur Processing Setup
QualBrowser.exe	Xcalibur Qual Browser
QuanBrowser.exe	Xcalibur Quan Browser
OALogin.exe	Xcalibur Open Access Login
OACtrl.exe	Xcalibur Open Access Server
ap_vi.exe	SIS AutoProbe Virtual Instrument
ap_x_controller	SIS AutoProbe Driver for Xcalibur

Uninstalling the software

Note: The Xcalibur uninstall program does not delete your Xcalibur data and methods even if you stored these files inside the c:\xcalibur directory. The uninstall program only deletes the files originally created by during the Xcalibur installation. Note, therefore, that the “c:\xcalibur” directory remains even following an uninstall. This directory may be safely deleted only after Xcalibur is uninstalled, all instrument drivers are uninstalled, and all valuable data and methods are backed up elsewhere.



Frequently asked questions.

Can I use the same MS source for AutoProbe and GC applications?

Yes, SIS can modify your existing source to permit dual AutoProbe and GC operation. However, during use in an Open Access environment, end users will not be able to switch between AutoProbe and GC operations; this requires action by the system administrator.

How can I secure the Open Access window so that end users cannot modify system settings?

The Open Access Server and Open Access Login provide a feature that allows the Open Access configuration to be “locked” unless a password is provided (although this feature may be turned off by the system administrator). However, even if Open Access is “locked,” the user may very well still be able to access the Windows Start Menu and from there modify the file system or run Xcalibur directly. There are two known ways to prevent this. One is run the Open Access Login on a computer other than the computer running the Open Access Server and to prevent the end user from accessing the computer running the Open Access Server (by physically isolating the computer or locking the computer when not in use by holding down CTRL-ALT-DELETE and then clicking “Lock Computer”). Open Access Login may even be run from end users’ own computers. The other solution is to set up a Windows user account with limited permissions and log into that account. This solution permits Open Access Server and Open Access Login Client to be installed on the same computer, but it requires some knowledge of Windows security to set up.

How long does a sample analysis take?

Three minutes, but this varies if you decide to cut or extend corners by modifying system configuration. Refer to the section “General Description of the AutoProbe” (page 10) for a breakdown of the analysis time.

In an Open Access Environment, how can users receive data results?

The Open Access Server allows the administrator to configure users and assign e-mail accounts to those users. For each user, you can specify whether results will be printed or sent to them via e-mail. Refer to the Xcalibur Open Access and processing method/report manuals for further information.

It appears that the rinses from the syringe cleaning are being injected back into the cleaning solvent reservoir.

This appears the case, but it is not. The valve system is set up such that waste is indeed being diverted into the waste collection vial. When many samples are run, waste will accumulate into the waste collection vial. The PAL port marked “Waste” is unused.