HPP7 & ProbeDirect[™] Manual

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B. Declaration of Conformity

Application of Council Direct	tive(s):
Standard(s) to which conform	ity is declared:
Manufacturer's Name:	Scientific Instrument Services, Inc.
Manufacturer's Address:	1027 Old York Rd. Ringoes, NJ 08551 U.S.A.
Importer's Name:	MS Wil GmbH
Importer's Address:	Postfach 4 CH-9500 Wil 1 Switzerland

Type of Equipment: Laboratory instrument

Model No.: HPP7

Serial No.:_____

Year of Manufacture: _____

I, the undersigned, hereby declare that the equipment specified above conforms to the above Directive(s) and Standards(s).

Place: Ringoes, NJ

(Signature)

Date: _____

(Full Name)

(Position)

C. Safety Information

Please read this section before proceeding further.

The following warning symbols are used in this manual to call attention to procedures which, if not adhered to or correctly performed can 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 Direct Insertion Probe and Electronics Console.



The Direct Insertion Probe Electronics Console must be connected to a power source with a protective earth contact. The absence of a protective earth ground can create a shock hazard for the operator and can damage the instrument.



Fuses with the required voltage and current rating and of the specified type must be used. The use of incorrect or makeshift fuses or short-circuiting of fuse holders creates a shock hazard for the operator and can damage the instrument. See Appendix A for more information.



The case for the Direct Insertion Probe Electronics Console should only be opened and serviced by a trained and qualified technician. There are no user serviceable parts. The power source to the Direct Insertion Probe Electronics Console must be disconnected before opening the electronics console.



The probe rod or tip may be HOT and should never be touched by hands. The Direct Probe should only be touched or picked up by the probe handle.



Compressed air is required for the cooling of the direct probe. The air pressure should be set to **40 psi** for optimum use. In NO case should the air pressure exceed **60 psi**.



Only DRY compressed air should be used to cool the probe rod. If house compressed air is used a moisture trap is required. For both house air and tank compressed air the SIS Part Number MT2002D air moisture filter must be used to remove traces of water from the cooling air. This compressed air moisture trap should be checked frequently and replaced when expired.



The Direct Probe and Electronics Console must be installed exactly as described in this manual. If not correctly installed damage to the probe or the Agilent mass spectrometer system or personal injury may occur.



The Direct Probe Electronics Console is shipped set up for either 110 or 220 VAC voltage. Check setting on the rear panel before use.



Use only sample vials supplied by SIS for the HPP7 probe. Other vials may damage the retaining spring or fall out and damage pumping systems. See Appendix D for SIS part numbers.



Mechanical vacuum pump exhaust must be filtered or vented external to the laboratory. SIS Part Numbers FK251 is suggested.

D. Warranty

Scientific Instrument Services, Inc. (SIS) warrants its scientific products, software and equipment against defects in materials and workmanship for a period of 12 months commencing from the date of shipment. During the warranty period, SIS will at its option, repair or replace products that prove to be defective.

The above-described warranty from SIS shall not apply to defects resulting from:

1. User negligence or use of the equipment in a manner contrary to the procedures and warnings outlined in this manual.

2. Improper or inadequate installation, maintenance, service, adjustment or calibration of the equipment by the customer.*

3. Unauthorized modifications of the equipment or misuse.

4. Water or moisture damage.

5. Operation of the equipment at electrical and environmental parameters outside of the specifications outlined in this manual.

6. Use of the probe with sample vials other than those specifically supplied by SIS for use with this equipment.

If physical damage is found, or the equipment does not perform as specified when the instrument is first received, please notify the carrier and SIS immediately. SIS will arrange for the repair or replacement of the equipment as soon as possible.

Scientific Instrument Services' liability on products manufactured, sold or repaired or the use of these products, whether on warranties or claim of negligence, or otherwise, is limited to the cost of correcting the defect in the product supplied by Scientific Instrument Services, Inc. In no case shall Scientific Instrument Services be liable for consequential or special damages. Scientific Instrument Services will not correct defects caused by the buyers negligence.

Scientific Instrument Services is not responsible for any damages or consequential damages that are due to attempted customer service or repair of the system.

All service including warranty service and repairs are available only at SIS facilities in Ringoes, NJ USA.

Warranty of the mechanical pump is supported by the pump manufacturer.

*The Direct Insertion Probe and Electronics Console are designed to be user installable utilizing this manual. SIS does not normally install these components at a users site. However, optional on site installation is available at an additional cost.

E. Service

The Direct Insertion Probe and Electronics Console are serviced and repaired only at the repair facilities of SIS in Ringoes, NJ. No on-site service is available. Do **not** contact Agilent for service on the HPP7 Direct Probe. The customer should retain packing and shipping materials.

If equipment is in need of service or repair, contact SIS and obtain a Return Authorization Number (RA Number).

Phone: (908) 788-5550 (USA) FAX: (908) 806-6631 WEB/E-Mail: http://www.sisweb.com

Return and Repair information can be found in **Appendix F**. The proper documentation is **required** for returns or they will be refused at our loading dock. Upon receipt of the equipment, SIS will contact you to notify you of the estimated repair costs.

Service agreements are available for service beyond the 1-year warranty period. This agreement covers all materials and labor for repairs conducted at the repair facilities of SIS in Ringoes, NJ. All equipment must be returned to SIS for servicing. Details of these agreements and pricing are available from Scientific Instrument Services.

Scientific Instrument Services is not responsible for any damages or consequential damages that are due to attempted customer service or repair of the system.

Do not return Direct Insertion Probe, Electronics Console, or Mechanical Pump to Agilent for repairs. All components are exclusively supported by Scientific Instrument Services, Inc.

Service and Warranty of the mechanical pump are supported by the pump manufacturer. See the pump manual.

Section I

Introduction – Chapter 1 Probe Installation - Chapter 2 Probe Operation - Chapter 3 Probe Maintenance - Chapter 4

Chapter 1 – Introduction

- Theory of Operation
- How this Manual is Organized

A. Theory of Operation of the Direct Probe and Probe Inlet System

The direct probe technique has been routinely used for the analysis of samples via the mass spectrometer for more than 30 years. It had its beginnings in the early stages of the development of the mass spectrometer before the GC was interfaced to the mass spec. The technique has the advantage of being a quick method of introducing solid



matrix samples directly into the mass spectrometer. It has been widely used by many laboratories for the routine analysis or screening of a large number of samples in a short period of time since the analysis time is usually only about 1 minute per sample. The major applications have been in the analysis of forensic drug samples, pharmaceuticals and polymers. It is also very useful as a quality control method where large volumes of samples need to be analyzed. This high productivity is a major advantage of the technique. In addition direct probe permits the analysis of many samples that would be impossible to chromatograph or introduce into the mass spectrometer via other techniques. The direct probe has now been interfaced to the HP 5973 MSD by Scientific Instrument Services. Through the use of this highly sensitive mass spectrometer and Agilent (HP) ChemStation software, which permits fast sample analysis and background subtraction, mixtures of samples can be analyzed and the components resolved and identified. The direct probe in conjunction with the HP 5973 is a proven powerful and highly productive method for the analysis of a large number of samples.



Scientific Instrument Services has designed the new direct probe and probe inlet specifically for the HP 5973 MSD. It will not interface to other models of

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the MSD. The use of a direct probe on previous versions of the MSD was not practical because the sources of these instruments (Models 5970, 5971 and 5972) were indirectly heated by the GC/MS interface. When the GC/MS interface was removed to allow for the introduction of a probe, another source heating system was needed to heat the MSD source. The new HP 5973 has a directly heated source, which now permits the development of this new direct probe.

The direct probe system consists of four major components: the direct probe, the probe inlet system, the electronics console, and the PC software for probe control. The probe inlet system mounts onto the HP 5973 MSD transfer line port where the GC normally was interfaced to the MSD. This requires the venting of the MSD and the removal of the GC and GC/MS interface in order to install the probe inlet system. After venting the MSD, this installation can be completed in about 30 minutes.



The probe inlet system mounts onto the side of the MSD and provides the probe valving and pumping system to permit the direct probe to enter the MSD vacuum system with minimal air introduction into the MSD vacuum system. An auxiliary vacuum pump is attached to the ¹/₄" tube at the end of the roughing valve to enable the evacuation of the probe inlet system. The probe inlet consists of two quarter turn valves. The main isolation valve permits the probe to pass through the valve and mate to the MSD source. The second roughing valve enables the auxiliary vacuum pump to partially evacuate the area in the probe inlet system between the tip of the probe sealed against the first vacuum seal and the isolation valve. The SIS dual PTFE sealing system provides for exceptional vacuum sealing against the probe rod as well as long seal life. The probe inlet system contains an indexed dual guide rod.



The SIS Probe Inlet System mounts on the GC/MS Transfer line port on the HP 5973 MSD to permit the quick and easy introduction of the SIS 1/4" diameter direct probe through the MSD Vacuum manifold and into the MSD source. It incorporates an indexed dual guide rod system that accurately guides the probe through the vacuum system valves. The guide rods are indexed at three positions in addition to the fully inserted and fully extracted positions:

- Load fully extended position probe loading position
- Stop 1 at the first vacuum seal to permit the rough pump out of the probe inlet seals
- Stop 2 at the second vacuum seal to enable the user to open the isolation valve
- Stop 3 1 cm from the source cooling position after the sample is finished
- Run fully injected position Run or sample analysis position

The normal procedure for the introduction of the probe into the MSD vacuum system has the following steps:

- Initially both the isolation and roughing vacuum valves are closed
- The direct probe is inserted into the probe sealing system and through the first vacuum seal (Stop 1)
- The roughing valve is opened to evacuate the pump seal area and probe tip
- After pumping for 30 seconds the probe is inserted further to seal against the second vacuum seal (Stop 2)
- The isolation valve is opened to the MSD
- The direct probe is inserted into the mass spectrometer past Stop 3 and inserted against the MSD source

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- The probe is heated and data analysis begins
- After analysis is complete automatic cooling begins
- The probe is extracted to Stop 3 and allowed to continue cooling below 100 degrees C
- The probe is extracted to Stop 2
- Both the isolation and roughing vacuum valves are closed
- The probe is fully extracted past Stop 1
- The sample vial is removed and the system prepared for the next sample

The direct probe is only ¹/₄" in diameter. This small diameter provides for minimal surface area and therefore less resistance when inserting it through the PTFE vacuum seals. The small size of the probe and handle also make it easy to handle and use unlike other probes which are big and bulky. The probe tip is heated via a rapid response resistance heater and temperature feedback and control is accomplished with a thermocouple attached to the probe tip inside the probe rod. Also inside the probe rod are two gas lines for cooling. The gas IN line extends to the tip of the probe to provide cooling directly on the heater coils. The electrical, thermocouple and gas lines are directly connected to the probe and into the electronics console.

Flared glass sample vials are used to contain the samples for analysis. The flared glass sample vials permit the efficient introduction of sample (both solid and liquid samples) into the glass vials and are easy to insert and remove from the direct probe tip without vial breakage. The direct probe tip contains a spring contact to gently hold the flared glass sample vials in place. For easy sample setup, a 12 hole sample vial block is included with the system. This block can be placed onto a hot plate to gently evaporate solvents from samples injected into the sample vials.





User setup and control of the probe heating and cooling is accomplished via a ProbeDirectTM software window. ProbeDirect may be operated independently of your data system (via the standalone mode), or it may be fully integrated into the Agilent ChemStation software (via the integrated mode). The direct probe can be ballistically heated to 450 degrees at ramp rates in excess of 500 degrees C per minute. This provides for fast removal of the samples on the probe and into the mass spec for analysis. In addition the probe tip can be programmed at ramp rates from 1 to 400 degrees per minute. At the end of the ramp the probe temperature can be held at this temperature for a timed interval before a second ramp is applied. Up to 14 ramp steps can be programmed via the PC window controller. After the sample analysis is complete the heater current to the probe is turned off and the compressed air cooling gas is turned on to lower the probe tip temperature to enable the removal of the probe through the PTFE vacuum seals of the probe inlet system. The PC control software fully integrates with the Agilent ChemStation software to not only control the probe parameters but also to store the probe parameters within the ChemStation method files and data analysis files.

The user inputs the probe temperature, ramp rate and time parameters into a PC window, however the actual control of the direct probe heating and cooling is accomplished via the Direct Probe Electronics Console. The direct probe electronics console contains its own microprocessor, which receives input from the PC to tell it what temperatures to heat the probe to and at what rate as well as the starting and stopping of the heating and cooling cycles. The direct probe electronics console uses this information from the PC to control the actual heating and cooling of the probe as well as the operation of the

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solenoid valve to permit the introduction of cooling air into the probe. The electronics console is interfaced to the PC via an RS232 plug, which plugs into a serial port in the PC. The electronics console in turn has a cable interfaced to the direct probe that contains the heating cable, thermocouple cable, air cooling and air exhaust lines.

A typical sample analysis proceeds as follows:

- 1. The sampling method in ChemStation is set up. This normally is a short analysis time of 1 to 3 minutes.
- 2. The Probe setup window is set up with the appropriate temperatures, ramp rate and time of analysis
- 3. The sample is set up in the sample vial, solvent evaporated if necessary and then inserted into the probe tip.
- 4. The probe is inserted through the vacuum lock as described above.
- 5. As soon at the probe meets the MSD source, data collection begins
- 6. After sample analysis the probe is backed out of the source to the third index stop (approximately 1 cm from the source) and the cooling cycle, which began automatically, continues until the probe cools to the point where it can be removed from the inlet system.
- 7. The probe is removed through the vacuum lock system, closing both the isolation and roughing valves.
- 8. The data is analyzed.

In studies completed at Scientific Instrument Services to date, the complete injection analysis and removal of a sample can be accomplished in less than 3 minutes per sample (data analysis and interpretation takes longer). The sensitivity of the Agilent 5973 has proven to be phenomenal when used with the direct probe. For the analysis of Caffeine we have been able to analyze samples with detection limits (full scan mode) down to 1.0 ng injected. Routinely we analyze 50 ng sample sizes via the direct probe technique and get library searchable and identifiable spectra of the known and unknown analytes. This sensitivity level is comparable to the older 5971 and 5972 MSD instruments for the analysis of GC samples. In addition we have been able to analyze and resolve mixtures of drugs using the direct probe technique. Multiple analytes in a sample will have different boiling points that will be thermally removed from the probe sample vial at slightly different times. Therefore the analytes can be separated and analyzed via the ChemStation software. Granted the total ion chromatograms look like poorly resolved packed GC column data, but the ChemSation software can separate the components using the background subtraction techniques. For additional details check out the direct probe application notes on http://www.sisweb.com.

B. How this Manual is Organized

Chapter 2, 3, and 4 describe the installation, operation, and maintenance of the probe from a hardware perspective. Chapter 5 and 6 describe the installation and operation of the control software.

Chapter 2 - Probe Installation

- Preparation of the Agilent 5973 MSD for Probe Installation
- Installation of the HPP7 Direct Probe Interface
 Socket
- Installation of the Vacuum Pump
- Installation of the Probe Controller
- Initial Insertion of the HPP7 Direct Probe
- Installation of the ProbeDirect Software

A. Preparation of the Agilent 5973 MSD for Probe Installation

The SIS Direct Insertion Probe, Model HPP7, is designed to be mounted onto the GC/MS Interface port on the Agilent (HP) 5973 MSD. Before you can install the Probe Inlet system onto the Agilent 5973 MSD, the GC and GC/MS interface must be disconnected from the MSD.

The following procedures are described in detail in the Agilent 5973 Mass Selective Detector Manual, HP# G1099-90001, Copyright 1996.

1. Vent the MSD (see Agilent 5973 MSD Manual Chapter 2).

Separate the MSD from the GC (see Agilent 5973 MSD Manual Chapter
 Be sure that the source and interface temperatures are cooled to below 50 degrees C before proceeding.

3. Remove the interface assembly cover from the GC/MSD Interface Assembly (see Agilent 5973 MSD Manual Chapter 4).

4. Remove the three screws holding the GC/MS Interface to the vacuum manifold. Store all removed parts in a safe location for future reinstallation. The probe inlet assembly will be mounted to these same boltholes using new bolts supplied with the probe inlet system.



5. Remove the sealing o-ring from the vacuum manifold flange where the GC/MSD interface was removed. Do not discard. This o-ring must be reused for the installation of the probe inlet system.

6. Open the MSD vacuum manifold to allow entrance to the MSD source. (See Agilent 5973 MSD Manual Chapter 2)

7. Remove the GC interface socket from the MSD source using the interface socket tool supplied with the probe system. Insert the center pin of the tool through the socket, and turn the tool counter-clockwise to remove the interface socket. Note that the center pin of the insertion tool is reversible. The smaller end is designed to fit the GC interface socket and the larger end is designed to fit the new Direct Probe interface socket. Store the removed interface socket with the GC/MS interface.



The MSD source is shown removed from the MSD system for clarity. It should not be necessary to remove the source from the source radiator in order to remove the interface socket.

B. Installation of the HPP7 Direct Probe Interface Socket

1. Install the new Probe Interface Socket by screwing it into the 5973 MSD source in place of the GC interface socket. Note that the new Probe Interface Socket is longer and has 4 flat sides. This socket should only be used in EI (Electron Impact Ionization) mode. The socket is needed in order to accurately guide the probe to mate with the MSD source.



The GC Interface Socket must be replaced with the new Probe Interface Socket in order to avoid breaking sample vials in the MSD. Glass chips may damage the pumping system.







Warning: The above, all metal, Probe Interface Socket is for use in EI (Electron Impact Ionization) mode only.



2. Reinstall the o-ring. This o-ring was removed in Step A.5. Make sure that the o-ring and o-ring groove are clean. Use no grease. Replacement o-rings are available from SIS.

3. Using the three (3) bolts (1/4"dia. x 0.5" long) and Allen wrench supplied, attach the probe inlet to the MSD vacuum manifold. Note the correct orientation of the probe inlet system. The probe guide rods should be facing down and the vacuum pump out line should be facing towards the rear of the MSD. Evenly tighten the three bolts to securely fasten the probe inlet system to the vacuum manifold flange.



Do not over-tighten!

4. After the probe inlet system has been attached to the MSD, close the MSD vacuum manifold plate in order to seal the MSD source. Replace the MSD cover. Do not pump down the MSD yet.

5. Attach the new MSD side panel to the Agilent 5973 MSD. This panel should be slid in place by angling it 45 degrees with the U slot just under the stem of the probe inlet system and then slowly slide it into place.





The side cover is required to prevent accidental contact with HOT surfaces within the MSD.



C. Installation of the Vacuum Pump

The direct probe inlet system requires an auxiliary vacuum pump to remove air from the probe inlet system. This "roughing out" must be done before the probe interface is opened to the vacuum system of the MSD. An Edwards 1.5 vacuum pump or equivalent is used for this purpose. The probe vacuum roughing pump can either be located behind the probe inlet system on the bench or alternately can be placed on the floor or at some other location close to the MSD. The following steps are required in order to set up the roughing vacuum pump.



Two separate vacuum pumps are required. One for the MSD and one for the probe. Do NOT try to use a single pump.

1. Prepare the vacuum pump according to the manufacturers directions. (See appropriate manuals)



The vacuum pump may or may not be shipped with oil. Be sure the pump is charged with new oil before proceeding. See pump manual.

2. Depending on the location of the vacuum pump, cut a piece of the $\frac{1}{4}$ " I.D. Tygon® vacuum tubing (supplied with the probe system) to the required length.

3. Attach one side of the vacuum hose to the $\frac{1}{4}$ " diameter tube on the vacuum pump nipple. Use one of the vacuum hose clamps (supplied) to securely fasten the hose. See illustration next page.

4. Clamp this vacuum pump fitting to the intake flange on the roughing pump using the centering ring with o-ring and clamp supplied with the pump.



Vacuum pump exhaust must be properly filtered or exhausted. Refer to vacuum pump manual. A filter kit is available from SIS (part number FK251).

5. Attach the other end of the vacuum hose to the $\frac{1}{4}$ inch stem of the tube extending out the back of the probe inlet assembly and again clamp in place with a vacuum hose clamp.

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6. Close the valves on the probe inlet system and plug in the mechanical vacuum pump to test the pump and check for vacuum leaks. Turn on the mechanical vacuum pump. (Pumps shipped within the United States and to selected locations include a switch on the pump power cord to simplify turning the pump on and off.) After a few seconds, the pump should run smoothly without a gurgling or chugging sound. If not, re-examine connections and pump installation procedures.

7. Turn off the vacuum pump.



D. Installation of the Probe Controller



Be sure that there is no power supplied to the electronics console and computer during installation of the air and computer connections.

The Probe Controller is the interface between the personal computer and the Direct Insertion Probe. The personal computer will transfer the temperature and control parameters to the probe electronics console. The probe electronics console will then control the operation of the direct probe heating and cooling through its own microprocessor. The following steps will interface the probe electronics console to the personal computer.





Units shipped within North America have been set for 120 VAC. Check voltage selection before proceeding. For other voltage selections see Appendix A.

1. A supply of clean and dry compressed air is required for certain probe operations. House compressed air or tank compressed air can be used for this purpose. If the air is damp, a moisture trap (SIS part number MT2002D) is 30

Chapter 2 - Probe Installation

required to minimize the introduction of water into the probe. Using a length of the 1/8 inch black nylon tubing supplied with the system, connect the air supply to the rear of the probe controller at the Quick Connect fitting marked "Air In". The Quick Connect attaches by pushing the outer sleeve of the fitting mounted on the electronics controller towards the controller. The male part of the fitting can then be inserted into its mate. When the two fittings are separated a valve within the male part prevents the flow of gas. The moisture trap includes 1/8-inch fittings that are compatible with the tubing attached to the Quick Connect. The trap can be installed by cutting the tubing and attaching the two new tubing ends to the connections on the trap. These connectors should be tightened 1/4 turn past finger tight. The air pressure should be set for 40 psi for use with the direct probe. Other pressures may effect the calibration of ramp rates.

Do not exceed 60-psi air pressure for the safe operation of the probe.

2. Turn on the air supply and check for leaks. Use Snoop (SIS part number SNOOP-8) or soap solution to check for leaks.

3. Install the RS232 cable between the Probe Controller and the personal computer. The RS232 cable should be attached to an RS232 (serial) port on the personal computer. Note which port (COM1, COM2, etc.) is used. This information will be required during software installation. A 9-pin to 25-pin adapter is provided if required for your particular computer. The opposite end of this cable attaches to the rear of the probe electronics console at the RS232 socket.

4. Finish the installation of the Probe Controller by making sure all fittings and plugs are secure. The Controller fits well under the probe inlet system on the right side of the MSD.

5. Connect the power cord at the rear of the Probe Controller. After checking the correct voltage setting plug the electronics console into the wall outlet. (Power switch is OFF.)

6. To check status of the controller, turn on the power switch. The front white button will illuminate to indicate that the system power is ON. This light will flash approximately once every five seconds until communication is established between the computer software and the Probe Controller.



View of Probe Controller Rear Panel

E. Initial Insertion of the Direct Probe

The system is now ready for the attachment of the direct probe to the probe inlet system.



Remove the plastic cover from the probe shaft before proceeding.

1. Attach the probe to the probe inlet system by sliding it into the U-shaped Probe Mount on the probe inlet system. Make sure that the sliding probe bracket is fully extended to the right before attaching the probe. It may be necessary to lift the Lock Release on the probe bracket to position the probe bracket.



For the following Procedures the Mass Spectrometer Vacuum system must be turned OFF and the probe Vacuum Pump must be Unplugged before proceeding. All vacuum systems must be OFF and the MSD must be vented. See the Agilent 5973 MSD manual for instructions on performing these operations.





The U-shaped Probe Mount must be fully extended before inserting or removing the probe or damage to the probe will occur.

Isolation Value (Closed Position)





The MSD must have been properly vented before performing the next step.

2. Open the Isolation Valve on the probe inlet system.

3. Gently slide the probe through the probe inlet system and seals until it touches the MSD source. It will be necessary to lift up the Lock Release on the Probe Mount to push the probe past the automatic stops. These stops are provided to allow accurate positioning of the probe during insertion and removal. They also prevent damage of the probe or probe sample vial as well as to minimize the inadvertent venting of the MSD during probe removal.



Lock Release

4. The probe should slide smoothly through the inlet system. If it is too loose or tight either loosen or tighten the black End Cap on the end of the probe inlet system. This will adjust the sealing properties of the probe seals inside the probe inlet system. With extended use this inlet system nut can be adjusted to allow for seal wear and prevent small vacuum leaks.

5. The probe should not hang up at the MSD source. It should slide smoothly and seat against the probe interface socket on the MSD source. If it does not

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seat smoothly, it will be necessary to open the MSD source housing and determine the problem.

Never insert a probe into the mass spectrometer if the probe or any part of the inlet system is bent or damaged. This could irreversibly damage the probe inlet system or the mass spectrometer. Misalignment of the probe shaft and mass spectrometer source could result in vial breakage and damage to pumping systems.

6. The distance between the probe handle and the probe inlet nut will be about 1/4" when the probe is in the proper location against the MSD source. This final position is past all 3 stops on the indexed probe guide.



7. Remove the probe from the Probe Inlet by lifting the lock release and sliding the Probe Mount to the end of the Guide Rods. Be sure that the U-shaped Probe Mount is at the far end of the Guide Rods before lifting the probe from the mount. Lifting the probe earlier will bend the probe shaft.

8. Close the Isolation Valve and pump down the MSD as per the instructions in the Agilent 5973 manual.

F. Installation of the ProbeDirect Software

Installation procedures for the probe control software are included in Section II of this Manual. Please follow those instructions before operating the HPP7 Direct Insertion Probe.
Chapter 3 - Probe Operation

- Software Operation
- Sample Preparation
- Insertion of the Direct Probe into the Mass Spectrometer
- Sample Analysis
- Removal of the Direct Probe from the Mass Spectrometer
- Summary of Probe Insertion and Removal

A. Software Operation

See Section II of this manual for complete description of software installation and use before operating the probe.



SIS ProbeDirect control window.

B. Sample Preparation

Both liquid and solid samples can be analyzed via the direct probe. In all cases, extreme care must be taken so as not to overload the mass spectrometer. Overloaded samples will cause contamination of the mass spectrometer source and will shorten detector lifetime. Sample sizes should be adjusted such that individual components are in the range of 1 to 200 nanograms per analyte in the sample.

Flared glass sample vials are used to introduce samples into the direct probe for analysis in the mass spectrometer.



Chapter 3 - Probe Operation



To Prevent damage to the mass spectrometer or probe only glass sample vials supplied by SIS should be used. (P/N S12A, S12B, or S12C) These vials are produced to precise tolerances. Other vials may damage the retaining spring at the probe tip or the pumping system of the mass spectrometer.

The flared glass vials allow for the easy introduction of samples into the direct probe. The flared tip provides increased strength and permits simple insertion and removal without breakage. Special grooved tweezers are provided for handling the flared sample vials.

Samples are normally prepared by dilution in a volatile solvent such as chloroform, acetone or methanol. The liquid samples are then injected into the sample vials using a syringe. Solvent can then be evaporated, leaving the solid analytes behind for subsequent direct probe analysis. This technique permits the accurate and quantitative introduction of known sample sizes and concentrations into the mass spectrometer.

Note: Sensitivity and reproducibility are enhanced when efforts are made to inject liquids samples to the bottom of the vial. This assures an even heating of the sample.

A sample vial block is included with the probe system for holding sample vials during the solvent evaporation process. The aluminum sample vial block can be placed on a hot plate at low temperature or other warm surface to facilitate solvent evaporation.

The flared sample vials are inserted into the end of the direct probe. This is most easily accomplished using the grooved tweezers supplied with the system. The sample vial is held in place via a small spring at the tip of the direct probe.



See Chapter 4 for care and maintenance of this spring.





Warning: Vials should fit securely into the probe tip. If they are loose or can slide, then the retaining spring needs adjustment. See Chapter 4.

Solid samples can also be placed directly into the glass sample vials. However extreme care must be used with this technique because it is very easy to overload the mass spectrometer in this manner. Normally, the vial is overloaded if sample material is visible within the vial.

C. Insertion of the Direct Probe into the Mass Spectrometer and Sample Analysis

1. Place the probe on to the U-shaped slot in the probe mount of the inlet assembly. This is performed after the sample has been loaded into the tip of the direct probe.





Before attaching the probe, be sure that the Probe Mount is fully extended to the right. It may be necessary to lift the lock release on the bracket to slide it to the correct position. The probe should easily slip into the bracket and the probe tip should not touch the inlet.

2. Move the probe to Stop Position 1.



Stop Position 1

The lock release will click when the probe is in position. At this point the probe tip should be extended into the Inlet Nut and will be sealing at the first of two probe vacuum seals. An illustration of the probe seals is shown in Chapter 4.A.

3. Open the probe Pump Out Valve by turning the valve handle onequarter turn counter clockwise.

This pumps out the volume between the two seals of the probe inlet system and minimizes the amount of air that enters the mass spectrometer. Since the probe tip is between the two inlet seals the area around the sample is now evacuated.



NOTE: Allow the vacuum pump to pump in this position for at least 10 to 15 seconds.

4. Lift the lock release lever and move the probe to Stop Position 2.

Chapter 3 - Probe Operation

This positions the probe tip just past the second probe vacuum seal in the inlet assembly. This stop position prevents the probe tip and sample vial from hitting the isolation valve during probe insertion. The probe now passes through both seals and will prevent excess air from entering the mass spectrometer when the isolation valve is opened.



5. Slowly open the isolation valve.

Perform this opening of the valve slowly to minimize any surge of pressure in the mass spectrometer vacuum system that might be caused by the small amount of air which was not evacuated by the roughing pump in the initial pumping described above.



6. Lift the lock release lever and insert the probe to Stop Position 3.

7. Close probe pump out valve by turning the roughing valve handle onequarter turn clockwise.



This will prevent the collection of pump oil in the interface region.

8. Release the lock release lever and insert the probe until it touches the mass spectrometer source.



Note: Do not force the probe. This will result in damage to the mass spectrometer source.



For some applications it may be best to begin data acquisition before the probe tip contacts the source. A heated source will affect the temperature of the probe tip.

D. Sample Analysis

9. Start the Direct Probe Sample Analysis by one of the following two methods. (This should not be attempted until the user has read and followed the instructions in Chapters 5 and 6.)

(1) Push the start button (large green button labeled Start) on the probe electronics console.

(2) From the main ProbeDirect screen, start the analysis by clicking on the "Start" button.

Note that the "Status" panel on the ProbeDirect screen must show "Connected" for a method to start. If the panel says "Not-Connected" communication between the probe controller and computer must be established. Other messages such as "Cooling" or "Ramping" etc. indicate processes that must be stopped before a method can start.

The probe tip will begin to heat according to the parameters established in the probe temperature program. A vertical bar on the probe setup screen will indicate the progress of the probe programs. The heater power and probe temperature are also indicated.

After the sample analysis is complete, the probe tip will stop heating and the cooling cycle will begin. The cooling gas may be turned on to accelerate this cooling process.

Chapter 3 - Probe Operation

For information on heating rates and the accuracy of temperature measurements see Appendix A.

E. Removal of the Direct Probe from the Mass Spectrometer



Great care must be used when removing the probe to avoid "venting" the instrument to the atmosphere. Venting the instrument will damage pumping systems and the detector.

10. Slide the probe to stop position 3 for cooling.



Stop Position 3

This position enables the probe tip to cool to a temperature less than 100 °C without being effected by the temperature of the source. It is necessary to cool the probe below 100 °C so that it can be withdrawn through the interface without damaging the Teflon® vacuum seals. While cooling, the cool light on the electronics console should be illuminated and the cooling gas should be flowing through the probe. The probe can be permitted to cool without this cooling gas, but will require significantly more time.

11. Slide the probe to Stop Position 2.

After the probe tip has cooled below 100 °C, a message on the computer screen will appear and the probe can be withdrawn to the Stop Position 2. Pull the lock release lever and slide the probe to the right until it clicks into position. This once again positions the probe just past the second vacuum seal in the probe inlet system. The probe is positioned so that it will not interfere with movement of the isolation valve.



Do not move the probe past Stop Position 2 without closing the Isolation valve. Doing so would vent the instrument.



Stop Position 2

12. Close the isolation valve to seal off the probe tip from the mass spectrometer vacuum system.



13. After closing the Isolation Valve, push the lock release lever to slide the probe to the Stop Position 1.



14. <u>Check</u> to be sure the Isolation Valve is closed. Push the lock release lever again to permit the probe to be fully withdrawn from the mass spec interface.

F. Summary of Probe Insertion and Withdrawal

Note: An action is required after every movement of the probe. Keeping this in mind will reduce the possibility of mistakes.

- 1. Load Sample into Probe
- 2. Place Probe on the Probe Mount
- 3. Move to Stop Position 1 Open Pumpout Valve, wait 15 seconds
- 4. Move to Stop Position 2 Open Isolation valve
- 5. Move to Stop Position 3 Close Pump Out Valve
- 6. Move Probe against Source Acquire Data
- 7. Move to Stop Position 3 (Cooling) Wait for probe to cool below 100 degrees C
- 8.Move to Stop Position 2 Close isolation Valve
- 9. Move to Stop Position 1 Check to be sure Isolation Valve is closed
- 10. Move Probe to end of rails *Remove Probe*

Chapter 4 - Probe Maintenance

- Replacement of Inlet Vacuum Seals
- Probe Spring Adjustment
- Care of the Probe
- Care of the Probe Controller
- Mechanical Pump

A. Replacement of Inlet Vacuum Seals

Location of probe seals.

There are two Teflon[®] probe seals within the probe interface. These are separated by a vent spacer.



These seals must be replaced if they have been damaged. Damage may occur through the use of a nicked, dirty, or scratched probe or by removing the probe when it is too hot. See Appendix A, Troubleshooting, for identification of problems with the probe seals.

The End Cap should be screwed on until a slight resistance is felt. Further tightening may be used to temporarily improve the function of the seals but it will also increase the rate of wear and decrease the lifetime of the seals.

Removal of Probe Seals

Probe Seals are a disposable item and will have to be replaced periodically. Once removed, seals cannot be reused. The removal process will damage the o-rings.

Seals may be removed with the Probe Interface on a clean workbench or attached to the mass spectrometer. If work is done with the interface on a workbench care must be taken to protect the o-ring surface on the flange that connects to the MSD. If this surface is scratched it may no longer seal properly to the mass spectrometer. Tools for replacing probe seals are shown on the following page. Only these devices should be used in the probe interface.



1. Begin the seal replacement process by unscrewing the End Cap (nut) at the entrance to the probe interface. Remove the Guide Bushing and place these two parts in a safe location.





Never use a damaged Seal Tool. The front edge of the tool must be smooth and round. A damaged tool will damage the interior of the Probe Interface and the replacement seals.

2. Remove the first seal by inserting the Seal Tool into the Probe Interface. Apply significant pressure to push the tool over the first seal. If the tool does not slide over the seal, rotate the tool slightly. The tool should slide approximately 1 cm.



3. Remove the Seal Tool from the interface and use the Seal Plunger to eject the first seal from the Seal Tool. This is done by inserting the Plunger through the back end of the Seal Tool.



4. Use the blunt pick to remove the Vent Spacer from the inlet.



Never place a sharp object within the probe interface. Scratches on the interior of the probe interface cannot be repaired.

5. Use the Seal Tool to remove the second Probe Seal using the same procedure as above. Clean the interface with a cotton swap or equivalent soft device. It is acceptable to use a mild solvent such as Acetone. Check for bits of o-ring that may have been cut by the Seal Tool.

Seal Insertion Procedure

6. Remove the new seals from their storage bag. They have been lubricated with a vacuum compatible material. Avoid skin and eye contact with the lubricant. See Material Safety Data Sheet.

7. Place new seal on the narrow tip of the Seal Plunger. Be sure that both parts are clean. To load the seal into the Seal Tool, insert the plunger into the back end of the Seal Tool as shown below.



Push the seal into the Seal Tool until the edge of the seal is flush with the front edge of the Seal Tool. Leave the Seal Plunger in this position.



Chapter 4 - Probe Maintenance

8. Now insert the Seal Tool all the way into the interface as shown below. Hold down the Seal Plunger so that it cannot move. At the same time pull the Seal Tool out. This will leave the Probe Seal in proper position within the interface. Now remove the plunger from the interface.



9. Insert the Vent Spacer back into the Probe Interface.

10. Load second Probe Seal into Seal Tool using the technique described above. Insert the Seal Tool into the Probe Interface using the same procedure that was used for the first seal. Use the Seal Plunger to hold the seal in place while the Seal Tool is removed.

Replace the Guide Bushing and screw the End Cap back on. Turn the End Cap only until a slight resistance is felt. Further tightening should not be necessary and will shorten the life of the seals.

Discard the old seals. They have been damaged by the removal process and are not reusable.

B. Probe Spring adjustment

The probe tip includes a spring to hold vials in place. This spring is a loop of metal ribbon welded to tabs at the end of the probe tip.



The spring may be tightened by squeezing gently on the exposed sides. The spring should not be compressed beyond the edges of the support tabs. It is suggested that the tool used be large enough so that the support tabs stop its movement.





Never use a probe that does not hold vials securely. Loose vials may damage pumping systems within the mass spectrometer. SIS will not be responsible for damage caused by loose or broken vials.

C. Care of The Probe

Storage

The Direct Insertion Probe should be stored on the Probe Mount on top of the Probe Controller. For long term storage the acrylic cover and rubber cap should be placed on the probe shaft for protection. These items were on the probe when shipped. Be sure the rubber and plastic parts are removed before the probe is heated again.

Cleaning

The probe shaft should be cleaned periodically. This may be done with a volatile solvent such as acetone. When cleaning use a soft lint-free cloth.



Never immerse the probe handle in liquids.

D. Care of the Probe Controller

The case of the Probe Controller may be wiped with a dilute soap and water solution. Do not use organic solvents. Do not clean with electrical power connected. Do not immerse the Probe Controller in liquids.

E. Mechanical Pump

The mechanical pump supplied with the HPP7 Direct Insertion Probe requires regular maintenance. See the pump manual for further information.

Section II Software Installation -Chapter 5 Software Operation -Chapter 6

Chapter 5 - Software Installation

- Introduction
- Connecting the Probe-to-PC communications Cable
- Preparing ChemStation
- ProbeDirect Installation
- Program Removal

A. Introduction

Overview. The probe temperatures, ramp rates, hold times, and start and stop events are all computer controlled through the use of the SIS ProbeDirectTM software. The ProbeDirect software is an easy-to-use Windows program that operates from a personal computer and directly communicates with the probe controller through a serial communications port. ProbeDirect provides a graphical user interface environment to create, store and download probe control methods, and it provides all status and run information for the probe. The software allows the SIS HPP7 probe to be heated ballistically at ramp rates in excess of 500 degrees per minute or temperature programmed with up to 14 ramp and hold steps.

ProbeDirect optionally can be fully integrated with the Agilent ChemStation software so that MS-probe operation is synchronized, and the probe temperature parameters are stored directly in ChemStation sample data (.D) files and method (.M) files.

Installation CD. The CD provided with this manual includes a copy of the ProbeDirect software. Before installation, it is recommended that you make a backup copy of the original ProbeDirect diskette and store the original in a safe location.

Installation Mode.

ProbeDirect can be installed in one of two operating modes:

- ChemStation integrated mode
- Standalone mode

The selection between these two modes of the ProbeDirect software is made during the installation process.

The **integrated mode** is designed to integrate the ProbeDirect software with the Agilent ChemStation software that controls the 5973 MSD mass spectrometer. The integrated mode will allow the two programs to communicate with each other, and the user will only have to give a single start command to begin both the probe ramp program and the MS analysis. The integrated mode will also cause the probe method parameters to be stored directly within the ChemStation method (.M) file. This is important for proper documentation of analysis procedures.

The **standalone mode** is designed to run independently from ChemStation or any data system. This "standalone" version of the software requires that the probe program and data acquisition be started independently. Probe parameters (methods) may be saved and recalled; however, they are not saved with the MS data system method files.



Installation Version.

The ProbeDirect software also comes in two versions, the newer **32-bit version** and the older **16-bit version**. The older 16-bit version is used only when using the HPP7 with ChemStation versions 1701AA and 1701BA in integrated mode only. **Except in this case, you should always install the newer 32-bit version**.

Software/OS Compatibility. Refer to the "Installation Notes" on the documentation on your installation disk for up-to-date information on software compatability.

Communication. The ProbeDirect software communicates to the Probe Controller through a free RS232 serial port on your personal computer. Check to make sure you have an unused serial port (9 or 25 pin) available on your personal computer that may be utilized for this function. If you do not have a free serial port, it will be necessary to add an additional serial port input/output (I/O) interface board to your personal computer. Contact SIS for more details on adding additional computer hardware.

Before continuing with the ProbeDirect installation, make sure you can locate the following items shipped to you as part of your probe purchase:

- Copy of the ProbeDirect software on CD
- 9/25-pin RS232 adapter
- RS232 communication cable

All of these items are important and will be utilized during the installation process. If you cannot locate any of these items, contact SIS for assistance.

Registration. A registration card has been included in your shipment. Please fill this out and mail it back to SIS. As a registered customer, you will be able to take advantage of SIS customer support programs and will receive information on future software products and revisions. You will need your registration number whenever you contact us for technical support.

This section. This section of the manual describes the installation and operation of the ProbeDirect software. It begins by giving you helpful hints for preparing your data system for ProbeDirect hardware and software installation. This is followed by instructions on hardware setup and installation of the ProbeDirect software. Upon successful installation, you can start to use the probe and ProbeDirect software by following the instructions in Chapter 6.

B. Connecting the Probe-to-PC communications cable.

In order for the probe to communicate with a personal computer, the probe controller must be connected to a free serial port on the personal computer. This port must be able to operate at 19200 baud (typically true). The following items have been included in your shipment for connection of the controller to your computer:

- 9 to 25 pin RS232 adapter
- RS232 communication cable

To connect the Probe Controller to your personal computer:

Step 1. Locate a free serial (COM) port on your computer.

Note: If you do not currently have a free serial (COM) port, please contact SIS for assistance. An additional serial communications card often can be installed.

Step 2. If the serial (COM) port has a 25 pin male connector, attach the 9 to 25-pin RS232 adapter to the port. Otherwise, skip to Step 3.

Step 3. Connect one end of the RS232 communication cable to the serial port.

Step 4. Connect the other end of the RS232 communication cable to the RS232 port on the rear of the Probe Controller.



Warning: Some video ports use a 9-pin configuration. Do Not use this connector. Doing so will damage electronics.

Note: The maximum allowable distance between the Computer and Probe Controller is 50 ft.

C. Preparing ChemStation

If you have not already done so, set up a ChemStation instrument to use with the HPP7 probe. This is done with the ChemStation MS Config program. Refer to your Agilent ChemStation manuals for details.



If using <u>integrated mode</u>, you must set up your ChemStation instrument <u>before</u> installing the ProbeDirect software. If you add an instrument for use with the HPP7 probe after having installed ProbeDirect, simply reinstall the ProbeDirect software. Failure to do so may result in either the ProbeDirect only being able to operate in standalone mode or your probe configuration not being saved with your instrument.

<u>Technical details</u>: The ProbeDirect installation program sets up program icons specific to your instrument and adds an instrumentspecific configuration file (sisprobe.ini) into each ChemStation instrument directory (e.g. <chemstation>\1\sisprobe.ini, for instrument #1).

D. ProbeDirect Installation

You are now ready to install the SIS ProbeDirect[™] software on your Agilent ChemStation GC/MS data system. This section contains information on hardware configuration, cabling and software installation of the ProbeDirect software to allow your PC to completely control the Probe Controller.

Important note: Before installing, make sure you have read the previous part of this chapter.

This program will occupy approximately 7 MB of hard drive space when installed. Make sure you have at least 7 MB of free space available before starting the installation process.

To install the ProbeDirect software:

- 1. **Place the ProbeDirect CD into your computer.** The installation web page typically will display automatically. This page will contain important information since the printing of this manual. Please follow the instructions provided on the computer screen to complete the software installation process.
- If the installation web page does not start automatically, you will need to start it manually as follows. Open the Windows Control Panel. Select the "Run..." option from the Windows Task bar and then enter "D:\README.html", where "D" is replaced by the drive letter for your system's CD-ROM drive. (Typical drive letters are D and E). If unsure about your drive letter, click the "Browse..." button to locate your drives.

Chapter 5 - Software Installation



3. Read the installation notes and determine whether you wish to install the latest (32-bit) version of the software or the older (16-bit) version of the software. (The only reason to install the 16-bit version is if you are running ChemStation version G1701AA or G1701BA **and** wish to use the integrated mode.) Click the appropriate link above to begin the installation. Windows may display some warnings at this point, in which case just respond by selecting "Open" or "Run."

Chapter 5 - Software Installation

🕞 Setup - SIS ProbeDirect	
	Welcome to the SIS ProbeDirect Setup Wizard
	This will install SIS ProbeDirect version 2.1 on your computer.
	It is recommended that you close all other applications before continuing.
	Click Next to continue, or Cancel to exit Setup.
	<u>N</u> ext > Cancel

- 4. Follow the instructions on your screen. You can usually just accept the default settings.
- 5. During installation, you will need to select between "**standalone**" and "**integrated**" mode. Refer to Section A of this chapter on the meaning of this mode. If you do install the software in integrated mode, you will still be able to run the software in standalone mode (but not vice versa).

<u>Technical details</u>: When you select integrated mode, a few DLLs (auxinlet.dll and sisrun.dll) are copied into your ChemStation's msexe directory, and the ChemStation instctl.mac is modified. A backup of your original instctl.mac is stored in the directprobe.backup subdirectory, and instctl.mac will be fully restored following software uninstallation.

6. The installation program copies the files from the ProbeDirect install disk to your hard drive and completes the installation. If any errors are reported, please contact SIS for assistance.

Before starting to utilize the ProbeDirect software, make sure you have completed all of the necessary hardware configuration and connections described in the previous sections.

E. Program Removal

The ProbeDirect software can be uninstalled via the "Add/Remove Programs" icon in the Windows Control Panel. (The Windows Control Panel can be opened from the "<u>Start | Settings | Control Panel</u>" menu item in the Windows NT/2000 Taskbar or just the "<u>Start | Control Panel</u>" menu item in the Windows XP Taskbar.) Alternately, you may execute the "uninstall.exe" program in the ProbeDirect program directory.

Note: For the older **16-bit version** of the software, double-click the "Removepd.bat" to uninstall.



For additional software information, especially technical information, refer to the documentation that comes on the installation CD and is installed on your computer.

- Getting Started
- Creating a new ProbeDirect Method
- Saving a Method as a File
- Loading a pre-existing ProbeDirect Method
- Printing a ProbeDirect Method
- Running a ProbeDirect Method
- Modifying a Method in Progress
- Ending a Method Run

A. Getting Started

Once you have completed installation, you are now ready to utilize the probe and the ProbeDirect software. This section contains instructions and suggestions for using the ProbeDirect software.

During installation of the standalone mode, a new program group folder called "SIS ProbeDirect" is created in the Programs folder on the Windows Taskbar menu. It contains start icons for the ProbeDirect software, as well as for the manual in PDF format.



If you installed the ProbeDirect in standalone mode, click the "ProbeDirect – Standalone Version" icon in the "SIS ProbeDirect" menu in the Windows Taskbar.

If you installed the ProbeDirect in integrated mode, your program group folder will contain the "ProbeDirect – Standalone Version" icon for starting the software in standalone mode as well as **one icon for each** ChemStation instrument on your system. For example, you might have an icon named "Probe Direct for Instrument #1 (Instrument #1)" for starting the ProbeDirect software integrated mode using ChemStation instrument #1. To start the **ProbeDirect software, simply double click on the appropriate ProbeDirect icon. Subsequently, start ChemStation** (for the same instrument number).

Note: if you create new ChemStation instruments and wish to use those instruments with the DirectProbe in integrated mode, you should reinstall the ProbeDirect software so that the appropriate icons can be created. Refer to Chapter 5 for details.

(<u>16-bit version</u>: For the older **16-bit version** of the software, the integrated ProbeDirect icons are instead located inside the program group folders for each instrument.)

Configuration. When the program is started for the first time, the software will display the ProbeDirect configuration panel (also available via the "<u>Probe</u> | <u>Probe Setup</u>..." menu on the ProbeDirect Window). This allows selection of the appropriate COM port that serves as the communication port between the PC and the Probe Controller. These settings may be changed at a later time using the "<u>Probe</u> | <u>Probe Options</u>" menu item. After this initial configuration, starting the ProbeDirect software will move directly to the main ProbeDirect display panel.

ProbeDirect Setup	×	
<u>Communication</u> Port:	ОК	
COM2	X Cancel	
Probe Air:	? <u>H</u> elp	
✓ Use <u>A</u> ir to maintain target temperature.		
Probe Type:		
DIP1 Direct Insertion Probe		

Once the ProbeDirect software is loaded, the main display panel for the software will appear. This display panel has been segmented into main control regions. Each region has a specialized function for either editing probe parameter information or reporting temperature and status information for the probe.



The Function of each region is defined below:

Menu Bar - Access to all main features including:

• Probe: All file and print related functions

- Control: Method execution and instrument control
- Options: Display and Hint options
- Help: Help information (Includes descriptions of all commands and error messages.)

Profile Display - Indicates temperature profile and total length of temperature program. Colors indicate different stages in the ramp cycle.

Probe Control Region - Set of easy-to-use control buttons to connect, start, cool and hold probe temperature control program.

Information Region - Provides status information and helpful hints on how to utilize the software. In addition, hints can be provided on each section of the main panel by selecting the SHOW HINTS command from the OPTIONS menu.

Method Editing Region - Edit probe temperature program with full graphical display of probe program. Allows up to 14 separate temperature segments with ballistic heating and cooling options. Simple spreadsheet control with detailed graphics view of temperature program.

Status region - Information on probe electronic status and temperature. When communication is established between the probe controller and computer a "Status" other than "Not Connected" will be displayed. Temperature values will also be shown in the "Target" and "Actual" fields. The "Heating Load" bar indicates the power applied to the heater.

Note: In order to reduce the area of the personal computer display that is occupied by ProbeDirect a reduced version of the main ProbeDirect screen is available. This option can be selected by choosing the "Options | Show Condensed" items from the menu bar. The condensed view only displays the Status region.

B. Creating a new ProbeDirect Method

Once the probe hardware has been configured, one can now proceed with developing a probe temperature program. The probe temperature program specifies how the probe tip will heat as a function of time. This information is entered in the Method Editing region and can be stored in a probe method file (.PRB) for later recall.

A probe method file can be created by simply specifying the probe temperature program within the Method Editing region of the ProbeDirect software and then selecting the <u>Save</u> command under the <u>File</u> menu. When using the standalone version of the code it is suggested that these files be saved in the same directory as the Agilent ChemStation method.

<u>Technical note:</u> The ChemStation integrated mode of ProbeDirect automatically stores the method in a file named SISProbe.Prb in the ChemStation method (.M) directory upon the user saving the ChemStation method. Whenever you load a different ChemStation method, the corresponding probe method is automatically loaded into the ProbeDirect window just as if the probe settings were part of the standard ChemStation method.

The Method Editing region is divided into two sections:

- Profile display (visual display of temperature program.)
- Probe temperature table



Warning: The probe tip will become HOT. Never touch the probe tip. Operation of the probe outside of a vacuum system is not recommended due to the risk of burns.

To specify the probe temperature program:

Step 1. Move to the Probe Temperature Program table and enter in the temperature program for the probe. To enter a value, select the table cell to edit and type in the appropriate temperature or rate value. Press <Enter> on your keyboard after entering a value. <u>A value of 0 in the Rate (°C/min) column indicates the end of heating steps in the program</u>. With the exception of cooling, all subsequent rows will be ignored.

Listed below are explanations of the various columns within the Method Editing Region.

Rate ($^{\circ}C/min$): Rate of heating or cooling. Not available for initial temperature period.

: Enables ballistic (>500 °C/min) heating or cooling for this section of the probe temperature program. Ballistic (air) cooling is enabled if the box in the last (Cool) row is checked. Ballistic heating is used to reach the initial temperature.

Target ($^{\circ}C$): Target temperature

Duration (min): Length of time to hold after reaching target temperature

• : If checked, the temperature program will hold indefinitely at the target temperature until either:

- The Resume button is pushed.
- The Cool button is pushed.

The scroll bar on the right side of the Method Editing Region can be used to examine all fourteen temperature ramp segments.

Note: Important information concerning allowed temperature ranges and temperature calibration is found in Appendix C.

As you enter the temperature program, the graphics region above the table will update to show the temperature program in graphical format.

Normally, at the end of a probe temperature program, you will want to ballistically cool the probe tip temperature to allow removal of the probe and analysis of the next sample. The probe tip must be below 100 °C before passage through the seals.

The last entry in the probe temperature table is reserved for a COOL program. This is normally used to ballistically cool (-400 $^{\circ}$ C/min) the probe to a desired temperature. (Rapid cooling is only available if air is used to cool the probe.)

To disable the ballistic cooling function simply un-check the \clubsuit column. In this case compressed air will not be used to cool the probe.

The following illustration shows an example method entry.



Target Temperature / Final Cooling Target



Warning: The probe temperature must be below 100 °C before the probe is removed from the mass spectrometer. Higher temperatures will damage the vacuum seals in the probe interface.
Integrated Mode and Standalone Mode

If the **integrated mode** of the ProbeDirect software is used, ensure that "Injection Source" variable is set to "Valve/Immediate Start". These parameters can be changed manually by selecting **Instrument** on the main ChemStation Instrument Control menu and then clicking on **Inlet/Injection Types**.

0	Inlet and Injection	Parameters	:	×
1	Sample <u>I</u> nlet	SIS DIP1	ŀ] [
<u> </u>	Injection <u>S</u> ource	Valve/Imme	ediate Start 💌] -
7	Injection Location		7] -
	₩ Use <u>M</u> S			
	OK	Cancel	<u>H</u> elp	2

When the **standalone mode** of ProbeDirect is used, these parameter options will not be available. The Sample Inlet parameter should be set to "Other/None."

e s	ample Name: 👘			Á
	Inlet and Injection	Parameters	×	
	Sample <u>I</u> nlet	Other/None	•	-+
	Injection <u>S</u> ource	Manual	F	
	Injection Location		7	
	₩ Use <u>M</u> S			2
	ОК	Cancel	<u>H</u> elp	

C. Saving a Method as a File

A method can be saved as a Probe Method File (.PRB) in one of three ways:

- 1. Select the <u>Probe</u> command from the menu and choose the <u>Save</u> command to save the changes made to the current file (file name is displayed in title bar at the top of the window).
- 2. In standalone mode, select the <u>Probe</u> option from the menu and choose the <u>Save As</u> command to save the changes made to the current file loaded in the ProbeDirect main panel to a different or new file name. Changes are not made to the original file.

3. In integrated mode, save your ChemStation method. Upon the ChemStation method being saved, the probe settings will also be saved.

D. Loading a pre-existing ProbeDirect Method

A probe method (.PRB) file can be loading in one of three ways:

- 1. In standalone mode, select the "<u>Probe</u> | <u>Open</u>" menu item. This will bring up a dialog from which you can select another (.PRB) file.
- 2. In integrated mode, load a ChemStation method. Upon doing so, any corresponding probe method settings will automatically be loaded into the ProbeDirect window. Changes are not made to the method imported from. If no probe settings exist in that ChemStation method, then default settings will be used.
- 3. In integrated mode, select the "<u>Probe</u> | <u>Read</u>" menu item. This will allow you to import probe parameters from another ChemStation method into the current ChemStation method.

E. Printing a ProbeDirect Method

To print a report detailing the probe method, simply choose the "<u>Probe</u> | <u>Print</u>" menu item. Use the "<u>Probe</u> | <u>Print Options</u>" command to set up output printer and print parameters before generating the report.

F. Running a ProbeDirect Method

Now that you have developed a method file for the probe, you are now ready to use this information to control the probe.

Running a probe method involves these basic steps:

- Establishing a communication link to the probe
- Defining or loading a method
- Starting the run
- Optionally, aborting the run

Before running a probe method, serial communication must be established between the computer and probe controller electronics. This step "connects" the two devices and allows the flow of information between them. Chapter 6 - Software Operation

Starting a communications session. Select the "<u>Control</u> | <u>Connect</u>" menu item or press the Connect button on the right side of the ProbeDirect main display panel. This will attempt to start a serial communication session between the personal computer and the controller.

If the connection is successful, the Information and Status Regions within the main panel will update and show a completed connection and the current temperature and status of the probe will become active. The probe temperature will also start to heat to the initial temperature entered in the table.

🕅 SIS ProbeDir	ect			
<u>P</u> robe <u>C</u> ontrol <u>(</u>	<u>D</u> ptions <u>H</u> elp			
Status:	Connected	Actual: 48 *C	Heating	E%
Run Time:	0.00	Target: 50 *C	Load:	0%

If the connection is not successful, you will receive a message:

ProbeDirect Fault			
	Fault during reconnect. Will try again(8).		OK
		?	<u>H</u> elp

The software will try several times on the first attempt at connection.

Note: A failed connection to the device usually results from one of the following conditions:

- 1. The connection was attempted during a short dead time within the Probe Controller communications software. An automatic second attempt will usually solve this problem. The dead time occurs for 0.5 seconds every 5 seconds so this is a common occurrence.
- 2. The Probe Controller power is not on. (White light should be on).
- 3. Improper serial port selection in ProbeDirect software configuration. (See the "<u>Probe | Probe Setup</u>" menu item.)
- 4. Conflict with another software program.
- 5. Improper cabling.
- 6. Bad serial port or cable.



Warning: The probe will heat to the initial temperature once connection is made.

Starting the run. After a successful connection, the Probe Controller device waits for a start signal before starting the temperature program. This start

Chapter 6 - Software Operation

signal can be from the ProbeDirect software or the green "Start" button on the front of the probe controller.

If the ChemStation **integrated mode** of ProbeDirect is used, the start of the probe temperature program is used to initiate MS data acquisition. First, be sure that the proper Inlet/Injection Types parameters have been set as described above. To execute the method, go to the main ChemStation window and select <u>Run</u> from the <u>Method</u> menu. Enter the sample analysis parameters as described in the Agilent Manual. Click the <u>Run</u> button. Starting the SIS Direct Insertion Probe will now initiate data acquisition. To start the probe program either press the green "Start" button on the front of the Probe Controller or click on the "Start" button on the ProbeDirect screen. This initiation method will coordinate the start of data acquisition and the probe program to within approximately 1 second.

When using the **standalone mode** of ProbeDirect, the probe control software and ChemStation function as separate programs. First, be sure that the proper the Inlet/Inject Type parameters have been set as described above ("Other/None"). The two software programs are then started independently. For instructions on starting the ChemStation software see the Agilent ChemStation Manual. To start the probe program either press the green "Start" button on the front of the Probe Controller or click on the "Start" button on the ProbeDirect screen.

To start the probe method in the ProbeDirect software, simply select the "<u>Control</u> | <u>Start</u>" menu item or press the Start button,



on the right side of the ProbeDirect main display panel. The green "Start" button on the front of the Probe Controller performs the same function. Note that the "Status" panel must show "Connected" for the program to begin. Other processes, such as Cooling, must be stopped before a new program can be started.

The information and status region on the bottom of the ProbeDirect main display panel will automatically update to show you the current run time.

G. Modifying a Method in Progress

A probe method can be changed during a run in one of two ways:

1. Using the HOLD command or button. This will set the current temperature as a new Target temperature. An overshoot should be expected when this command is used while executing fast ramp rates. The method will continue when the Resume button is pressed.



2. A method can also be changed by entering new data in the probe temperature table. Only changes made to rows not yet started will be recognized during the current run. Be sure to push the <Enter> key after entering data.

H. Ending a Method Run

1. Normal completion

If the last segment in the probe temperature table is set to cool to some initial temperature, the probe will attempt to return to this temperature at the end of the run. If the cooling air is too warm to cool the probe to the final target temperature the software will stop the flow of air after 6 minutes.

2. Manual User Abort

You can immediately abort a run manually by selecting the "**Control | Stop**" or "**Control | Cool**" menu items. You may also select the STOP or COOL buttons on the right side of the ProbeDirect Screen. The COOL button on the front of the Probe Controller performs the same function as the COOL button on the software screen. The COOL command initiates the cooling sequence while the STOP command halts the run but does not initiate air cooling.



Section III - Appendix

A – Troubleshooting

- **B** Specifications
- C Temperature Ranges &

Calibration

D – Parts List

E – Trademarks

F-Repairs, Returns, and

Registration

- Voltage Selection
- Fuse Selection
- Error Messages
- Problem Solving



There are no user serviceable components within the HPP7 probe and controller assembly. Refer to a qualified technician or contact Scientific Instrument Services, Inc. The SIS Direct Insertion Probe model HPP7 is not serviced by Agilent.

A. Voltage Selection

Unit is shipped with voltage identification tag attached to rear of controller. Check this tag and voltage indicator on power input module before connecting power cord.

Input Voltage Ranges:

Input Voltage	Fuse	Voltage Range Selection
95 - 130 VAC	3AF 250V	120
200 - 250	1.5AF 250V	240



Remove power cord before opening power input module.

Input voltage is selected from within the power input module. This device can be accessed by inserting a screwdriver in the small slot just below the power cord connector (See top illustration next page). Prying with the screwdriver will remove the cover plate and fuse holder. The voltage selector tab can then be removed by pulling with a small pair of pliers or tweezers. To change the voltage selection, rotate the plastic indicator so that it faces away from the numbers indicating the desired voltage. (See bottom illustration next page) Reinsert the voltage selector tab fully into the power input module. The text on the voltage selector tab should be facing the power on/off switch. Proceed to fuse selection.



Changing the input voltage requires a change in power line fuse. Complete next section before closing power input module.

Appendix A - Troubleshooting



B. Fuse Selection

Power Line Fuses:

Fuse selection for the power line input is made within the power input module. See previous section of this chapter for instructions on accessing this device.



Remove power cord before opening power input module.

Fuse requirements may depend on local ordinances. Some regions (e.g. European Community) require two 5 x 20 mm fuses on the power input lines. Others (e.g. United States) require only a single $\frac{1}{4}$ x $\frac{1}{4}$ -inch fuse on the "Hot" line of the power input. Consult your local requirements before proceeding.

The dual or single fuse option may be changed by flipping the fuse holder attached to the cover of the power input module. This is accomplished by loosening of the screw indicated below. The fuse holder has two sides. One includes a channel for a $\frac{1}{4} \times 1^{\frac{1}{4}}$ -inch fuse and a metal bar. The other includes two channels for 5 x 20 mm fuses. The side fully exposed when the fuse holder is attached to the cover of the power input module is the active side. Only one set of fuses can be used at a time.



Select fuses based on the chart below:

Input Voltage	Fuse	Voltage Range Selection
95 - 130 VAC	3AF 250V	120
200 - 250	1.5AF 250V	240
Fuses in the power in	put module sho	ould be "Fast-Blo."

After the proper fuses have been installed reinsert the fuse holder and cover in the power input module. The cover should fit flush with the surface of the power input module. If not, check the positioning of the voltage selector tab. It should slide fully into its slot.



At this point check that the plastic tip to the voltage indicator is protruding through the proper opening on the power input module cover. (See illustration page A-2.) If not, review the previous section on voltage selection.

Heater Fuse:

The heater fuse limits the current passing to the heater coil within the probe tip. It is located in the fuse holder on the rear of the controller. (See figure is section A of this chapter. This fuse is labeled "3AT 125V" and must be replaced with a $\frac{1}{4} \times \frac{11}{4}$ inch 3 Amp Slo Blo fuse with a voltage rating of 125V or higher. These fuses are available from SIS (P/N 313003).

C. Error Messages

A complete description of error messages can be found in the documentation on the installation disk and installed on your computer.

D. Problem Solving

1. Software

1.a. Communication cannot be established

Check that the Probe Controller power is on and the power cord is properly connected. The white light on front panel should be on. Check the connection of RS232 cable from controller to COM port. Check the identity of the COM port (COM1, COM2, etc.). Software selection of the COM port can be made under "Probe | Probe Setup..." menu. Try using different COM ports. Other programs may interfere with use of the COM port. Stop all programs other than ProbeDirect and then try making connection. Do not forget modem and network programs that start automatically. If a conflict is found the two programs must be set up to use different COM ports. Check that the COM port is capable of communicating with the following settings:

19200 baud, one stop bit, even parity, 7 bit data size, CTS/RTS off

This is the factory setting for communications with the controller firmware. Some very old communications cards cannot operate at 19200 baud.

1.b. Communication lost

The probe controller and computer attempt to talk to each other once every second. If this communication is interrupted any methods in progress will be halted and the controller will reset to its initial state (and beep). This will stop heating of the probe. Please allow time for the probe to cool (10 min.) before

touching or removal from the mass spectrometer. Communication may be reestablished by clicking on the "Connect" button.

Loss of communication can occur from interruptions in the connection between the computer and controller (loose cable). It may also result from electrical noise or loss of electrical power to the probe. Other software may interfere with the operation of the probe by taking control of the computer communications interrupts for more than 1 second. In some cases this may occur only when the conflicting software is initialized. If so establish the controller computer connection after starting the conflicting software.

1.c. Method does not start.

The "Status" panel on the ProbeDirect screen must show "Connected" for a method to start. If the panel says "Not-Connected," communication between the probe controller and computer must be established. Other messages such as "Cooling" or "Ramping" etc. indicate processes that must be stopped before a method can start. (Click on the STOP button.)

1.d. Data acquisition does not wait for signal from ProbeDirect under integrated mode.

Be sure that the Auxiliary Inlet is set to "SIS DIP1" and the Injection Source is "Value/Immediate Start". If an Agilent ChemStation method is executed while ProbeDirect is executing a temperature program the data acquisition will start immediately. Be sure that the probe status is "Connected" before starting the ChemStation method if you want the ChemStation to wait for a start signal from ProbeDirect.

1.e. Sudden interruption of Method.

This will typically result from a loss of communication or power. It is also possible that temperature limits have been exceeded or improper data has been entered in the method table. Check the values entered. Other problems will be accompanied by an appropriate error message.

Note that the method ends when a zero is found in the "Rate (°C/min)" column. Subsequent rows are ignored.

1.f. Cannot close ChemStation or ChemStation is stuck in "Run" mode.

Under integrated mode, when a ChemStation method is started it will enter a "PreRun" mode as indicated near the upper left corner of the ChemStation acquisition window. At this point it is waiting for a start signal from ProbeDirect. ChemStation must get this signal before operating properly. If the ChemStation method is aborted before receiving a start signal from ProbeDirect it may go into "Run" mode but not take data. It still needs a start signal from ProbeDirect before operating properly. Give a start signal by running a probe method.

2. Heating and Cooling

2.a. Probe does not heat

If the "Heating Load" display indicates that power is being applied to the heater but the temperature is not rising above ambient temperatures this may be a problem with either the heater or the temperature measurement.

Warning: The probe may be HOT and the problem may be with the temperature measurement. Reset controller and allow 10 min. in cooling position before removing probe from mass spectrometer.

In many cases the software will detect this problem and notify the user. Check and replace if needed the heater fuse on the rear of the controller. See Fuse Selection (above) for specific instructions. If the problem persists the probe and controller should be returned to SIS for repair or replacement.

The thermocouple used in the probe tip should be isolated from ground potential. If the insulation fails an incorrect temperature will be measured. This failure mechanism can be tested by touching the probe shaft to a grounded surface, such as the inlet, and observing if the indicated temperature changes. This problem must be fixed by SIS.

2.b. Air is on while heating

This may not be a problem. If the "Use air to maintain target temperature" option is checked (See "Probe | Probe Setup..." Menu) air will be used when the target temperature is 200 °C or less. This prevents the source of the 5973 MSD from heating the probe above the target temperature.

If the Air is still on when the probe is attempting to heat above 200 °C there is a problem with the air valve. Contact SIS concerning repair of this problem.

Airflow can be checked by placing one's finger near the "Air Out" port on the back of the controller.

2.c. Probe cools too slowly

See Appendix C for expected cooling times. Be sure that the probe is in the "Cooling Position" (Stop Position 3).

When the "Status" panel on the ProbeDirect screen shows "Cooling" air should be flowing. The airflow can be checked by placing one's finger near the "Air Out" port on the back of the controller.



Check the air pressure. System is calibrated for use at 40 psi and 22 °C. (60 psi maximum). Note that the Quick-Connect at the air input to the controller has a check valve so that air will not flow when the male part of the connector is not inserted into the female part. To check air flow at this point press the tip of the male connector onto a solid surface. The check valve should open.

If sufficient air pressure is present at the Quick-Connect but air is not flowing when the software indicates "Cooling" Status, SIS should be contacted regarding repair or replacement of the unit.

2.d. Probe is heating but probe temperature does not reach target temperature

Check the voltage input setting. If the controller expects 240 VAC but only receives 120 VAC the probe will not heat properly. Check that air is not flowing if target temperature is above 200 °C. Airflow can be checked by placing one's finger near the "Air Out" port on the back of the controller. Check that the 5973 MSD source temperature is above 200 °C.

2.e. Probe temperature significantly above target

Check the voltage input setting. If controller expects 120 VAC but receives 240 VAC the probe will overheat. This will damage the probe heater. Check that air is flowing when the target temperature is below 200 °C and the "Use air to maintain target temperature" option has been selected. Airflow can be checked by placing one's finger near the "Air Out" port on the back of the controller. Note that the temperature ramp is designed to overshoot the target temperature during fast ramps. Temperature calibration is discussed in Appendix C.

3. Controller

3.a. White light does not illuminate when controller is turned on

One of the two front panel lights should always be on or flashing. If not, check that power switch is on and power cord is properly connected. Check the line fuses (See fuse selection on page A-3.) Attempt to make a software connection between computer and controller. If the connection works the problem is in the light or associated electronics. The green light should illuminate when a method is run. Contact SIS for repair information.

The white light will flash off once every 5 seconds when controller is on but not communicating with the personal computer.

3.b. Green light does not illuminate when controller method is executed.

The green light should be lit when probe status is "Soaking", "Ballistic", or "Ramping". If not, and system is otherwise working well, problem is in the light or wiring to the light. Contact SIS for repair information.

3.c. Front buttons do not work

The "Start" and "Cool" buttons on the front panel should behave in a manner similar to the "Start" and "Cool" buttons on the ProbeDirect software screen. If both buttons are pushed within 2 seconds the controller will be reset. When a button is pushed it should light.

Test to see that these buttons are working by comparing their function with their counterparts on the computer screen. Contact SIS for repair information.

3.d. Air leaks

If air leaks from the Quick-Connect fitting on the rear of the controller, review the connection instructions. Watch for dirt or contamination. If air leaks from other fittings, review their respective installation instructions. Contact SIS for the replacement of damaged parts or tubing. If you believe that there may be an air leak within the controller contact SIS for repair information.

4. Vacuum interface

4.a. Poor vacuum in 5973 MSD

A poor vacuum may be the result of insufficient pumping or a leak in the vacuum seals. Consult the 5973 MSD manual for troubleshooting vacuum problems within the instrument.

- Leaks before the isolation valve is opened

This indicates that there may be a leak in the seal between the vacuum interface and the mass spectrometer or in the isolation valve on the interface. Check to see that the isolation valve is fully closed. Insert the probe following the instructions in Chapter 2. If this improves the vacuum within the mass spectrometer there is a leak in the isolation valve. Contact SIS concerning repair or replacement.

If insertion of the probe did not improve the vacuum the problem is not in the isolation valve or there is more than one leak. Slightly tighten the End Cap on the entrance to the interface and check that the mechanical pump is on. If there is no improvement, continue by venting the instrument per the instructions in the 5973 MSD manual. Remove the interface from the instrument. Examine carefully the o-ring and sealing surfaces. They must be clean and smooth. If the o-ring is damaged (it should feel smooth and have no chips, cracks, or deformities) replace it and remount the Probe Inlet on the instrument following the instructions in Chapter 2. If there are any scratches or marks on the surfaces in contact with the o-ring these are likely to be the source of a leak. Contact SIS about repair or replacement. If no problem is found with the o-rings, replace the original GC interface or blank flange that came with the 5973 MSD. Then test the vacuum within the instrument. If no

problems are found there may be more than one leak in the probe interface. This may, for example, occur when a piece of damaged o-ring from the probe seals is trapped in the isolation valve. Replace the probe seals or contact SIS regarding repair.

- Leaks when interface valve is opened

The pressure within the mass spectrometer should not rise by more than ~ 50 times when the interface value is opened. If this occurs confirm that the probe was in the proper position, sufficient time was given for pumping out the interface, and the mechanical pump is working properly. Consult the maintenance records for the mechanical pump and compare these with the recommendations in the pump manual. Service the pump if required.

If the mass spectrometer pressure returns to its original value within 1 minute, the problem is likely to be with the mechanical pumping system. This can be confirmed by slowly opening the Pump Out valve. If the pressure rises again then there is a problem between the mechanical pump and the Pump Out valve.

If the mass spectrometer pressure does not return to its original value within 1 minute after the interface valve is opened the problem is likely to be in the probe seals. Slightly tighten the End Cap at the entrance to the probe interface. If this does not solve the problem replace the probe seals. (SIS part number HPP7007)

- Leaks while inserting probe

If the mass spectrometer pressure suddenly jumps as the probe is slid into the interface there may be a problem with the probe shaft or probe seals. Carefully examine the probe shaft. It must be smooth and straight without scratches or nicks. If the shaft is damaged contact SIS about replacement or repair. If there are no problems with the probe shaft replace the probe seals. (SIS part number HPP7007) In addition, if there are any rough scratches or nicks on the probe shaft the seals have probably been damaged and should be replaced.

5. Mechanical pump

Consult the mechanical pump manual for troubleshooting and maintenance of the mechanical pump. This device requires regular care.

6. Probe

6.a. Probe does not align with front of Probe Inlet.

First check that the probe shaft is straight. Never use a bent probe. Second examine the slide rods on the interface. If either of these components are bent or damaged contact SIS about replacement.

6.b. Probe sticks or is blocked

Carefully examine the probe shaft. It must be smooth and straight without scratches or nicks. Never use a bent probe. If the shaft is damaged contact SIS about replacement or repair. If a good probe does not slide smoothly into the interface the probe seals may be too tight. Loosen the knurled knob on the front of the interface. If this does not improve movement of the probe, examine the seals for dirt or contamination. If no problems are found replace the probe seals. (SIS part number HPP7007)

6.c. Probe shaft is bent.

Never use a bent probe. This may cause damage to the Probe Inlet or to the MSD pumping system if a vial is broken while attempting to insert the probe. Contact SIS about replacement.

6.d. Guide rods on the Probe Inlet are bent.

Never use an interface with bent parts. Contact SIS about replacement.

6.e. Scratched probe shaft.

Never use a damaged probe. Contact SIS about replacement.

6.f. Dirty or contaminated probe.

Clean the probe shaft with soap and water or organic solvent. Do not use abrasive materials. Never immerse the probe handle in liquids.

6.g. Air leaks from probe handle.

If air is leaking from the probe handle or the line between the controller and probe, contact SIS for repair or replacement information.

6.h. Problems with sample vials.

Be sure to use only vials supplied by SIS. Other vials may damage the retaining spring. There is a discussion of spring tightening in Chapter 4.

Never use a probe that does not hold vials securely. Loose vials may damage pumping systems within the mass spectrometer. SIS will not be responsible for damage caused by loose or broken vials.

Never use a damaged vial.

6.i. Broken sample vial in probe tip.

Broken vials must be carefully removed. Do not use anything that could damage the spring or probe tip. Never use a probe with glass fragments in the probe tip. Vials should fit snugly in probe tip.

6.j. Spring does not hold vials properly.

See the discussion of spring tightening in Chapter 4. If vials cannot be made to fit properly contact SIS.

6.k. Vial does not go in at all.

The retaining spring has been damaged or over tightened. Use a pointed instrument to spread the sides of the spring apart. Use caution. Note that a broken spring may be repairable but damage to the tabs used to support the spring will require probe replacement. See diagram at the end of Chapter 4. If a spring no longer functions reliably contact SIS for repair or replacement.

6.1. Vial does not go in all the way.

There may be material from a broken vial within the probe tip. Never use a probe with glass fragments in the probe tip. See above for discussion of removing broken vials. Vials should fit in probe tip as shown in Chapter 4.

Appendix B -Specifications

Electrical

Power Requirements

95-130 or 200-250 VAC, Ground lug required Input Frequency 50/60 Hz 75 Watts Max Shielded Power cord required to meet emission requirements. (Supplied) Installation Category II

Fuses

Input VoltageFuse		Voltage Range Selection
95-130 VAC	3AF 250V	120
200-250	1.5AF 250V	240

Note: heating rates are calculated for 120 and 240 VAC. Other voltages will result in increased deviation from target temperatures.

Fuse and voltage selection are made within the power input module. See Appendix A.

Direct Insertion Probe Heater

Voltage: 8 VAC with 120 or 240 VAC line input Fuse: 3AT 125V (on rear panel) Heater Resistance: 5.5 ohms +/- 10%

Environmental

Designed for Indoor Use Only Operating Environment: 15 to 35 degrees C Operating Humidity: 25 to 50% relative humidity Non-Operating Humidity: 10 to 90% relative humidity, noncondensing Grooves in the front of the controller serve as air vents, do not block Pollution Degree 2

Compressed Air

Compressed air is required for rapid cooling of the direct probe. The air pressure should be set to 40 psi for optimum use. In NO case should the air pressure exceed 60 psi.

Only DRY compressed air should be used to cool the probe rod. If house compressed air is used a moisture trap is required. For both house air and tank compressed air the SIS Part # MT2002D air moisture filter must be used to remove traces of water from the cooling

Appendix B - Specifications

air. This compressed air moisture trap should be checked frequently and replaced when expired.

Venting

All mechanical vacuum pump exhaust must be filtered or vented external to the laboratory. This is a required safety precaution. Oil mist generated by mechanical pumps can be harmful. A mist eliminator is available from SIS, part numbers 68316 and ED2.

Communications

RS-232 Cable (supplied) 9-pin to 25-pin adapter (supplied)

Appendix C -Temperature Ranges & Calibration

Temperature ranges:

Probe tip:

Maximum temperature: 450 °C Minimum temperature: 0 °C (Temperatures below 0 °C will be interpreted as a thermocouple error.)

Probe tip when passing through seals: Maximum temperature: 100 °C

Cooling target:

Maximum temperature: 100 °C Minimum temperature: 10 °C

Ramp Rate:

Ballistic: >1200 °C/Min. for the probe tip corresponds to ~500 °C/Min. inside the sample vial

Maximum selectable: 500 °C/Min. Minimum selectable: 1 °C/Min.

Calibrated Ramp Rates: (°C/Min.)

Ramp rates have been calibrated for a MSD source at 350 °C and a cooling gas pressure of 40 psi. For lower source temperatures the gas pressure may be reduced if desired.

With air option:

Target temperature	Max Rate	Min Rate
<=200 °C	400 °C/Min	1
201-450 °C	500	~100
Without air option:		
Target temperature	Max Rate	Min Rate
<=450 °C	500 °C/Min	~100

Temperature Calibration:

Temperature measurements are made using a thermocouple within the tip of the probe. These measurements are accurate within 2 % and are displayed on the ProbeDirect screen. However, the temperature of the sample may be different than that of the thermocouple since they are separated by a glass vial. The temperature of the front (flared) end of the vial will also be a function of the temperature of the MSD source. Thus there may be a temperature gradient within the vial. To obtain the most reproducible results it is best to deposit the sample completely at the bottom of the vial.

Temperature variations are found within all direct insertion probes. Fortunately, in almost all applications reproducibility is more important that 96

Appendix C - Temperature Ranges & Calibration

obtaining an exact temperature for the sample. While most probe manufacturers report the temperature of a thermocouple or platinum resistance thermometer, SIS believes that it is important to point out that the temperature of the sample may be different. The most accurate temperature readings for the SIS HPP7 Direct Probe are obtained when air is not used to stabilize the temperature of the probe tip. This is because the airflow tends to increase the magnitude of the temperature gradient within the probe tip. The thermocouple is cooled more efficiently than the heater coil. Thus, when the air is flowing the thermocouple may be cooler than the interior of the vial. (By as much as $25 \ ^{\circ}C$ at 100 $^{\circ}C$.) The use of air to stabilize the temperature at values below $200 \ ^{\circ}C$ therefore increases the precision of the sample temperature at the expense of accuracy. When air is not used to stabilize the probe temperature the thermocouple and inside of the vial thermally equilibrate within 1 second. The temperature of the vial increases more quickly than the thermocouple reading.

Ramp Calibration:

Measured temperatures during temperature ramps are expected to be within approximately ± 4 °C or ± 2 seconds of the target temperature.

Temperature Overshoot:

In most applications a rapid temperature ramp is more important than a gradual approach to the target temperature. In addition, the entire sample vial does not overshoot the target temperature to the extent that the thermocouple does. The Probe controller has therefore been adjusted to overshoot the target temperature by a reproducible amount. This is followed by an oscillation and a quick stabilization near the target temperature.

Temperature Overshoot when using the Hold button:

Stopping a rapid ramp without prior notice is difficult. The ProbeDirect software may be calculating heating rates several seconds in advance. A large overshoot and oscillation can therefore be expected as part of normal operation of this function.

Appendix D - Parts List



B. Part List

The HPP7 Probe Package includes the following components:

Part number:	
HPP7003	Probe Controller
HPP7004	Probe
HPP7005	Inlet
HPP7006	Side Cover for 5973 MSD
MT2002D	Air Filter (moisture trap)
HPP7009	Tool Kit
	Box #1 Parts Kit
	Box #2 Parts Kit

Tool Kit Includes:

1 of	SVB712	12 Hole Vial Block
1	HPP7001	Socket Tool
1	HPP7002	Seal Tool
1	HPP7014	Washer & Nut Set
1	5503A39	Allen Driver
1	HPP7012	Blunt Pick
1	HPP7013	Vial Tweezers
1	HPP7008	Probe Socket
1	S12A	Sample Vials (100 count)

Box #1 Parts Kit Includes:

2	53175K71	Hose Clamp
6	61210B	foot 1/8" Nylon Tubing
6	14TT	foot 1/4" ID Tygon® Vacuum Tubing
1	NS10025	Hose Nipple
1	17600	Shielded Power Cord
1	CBLMNT9	6' RS232 Cable
1	GENDER259	9 to 25 pin Adapter

Box #2 Parts Kit Includes:

1	HPP7M	Manual
1	HPP7010	ProbeDirect [™] Software
1	HPLit	SIS literature pack for 5973 MSD users.

Shipped Separately:

1 HPP7011 Mechanical Vacuum Pump

Additional Accessories Available:

S12C Sample Vials 500 count

Appendix D - Parts List

Sample Vials 1000 count
Oil Mist Eliminator Kit
Adapter for 68316 and Edwards Pump
Replacement Inlet Seal Set

Appendix E -Trademarks

Windows® is a registered trademark of Microsoft Corporation.

Teflon® is a registered trademark of E.I. Dupont.

Swagelok® is a registered trademark of Swagelok Co.

Tygon® is a registered trademark of Norton Co.

ProbeDirectTM is a registered trademark of Scientific Instrument Services, Inc.

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Appendix F- Repairs, Returns, And Registration

Do not return the HPP7 Direct Insertion Probe or related parts to Agilent.



All Returns require a Return Authorization Number (RMA) Items without an RMA number will not be accepted from shipper.

Warranty Returns: The SIS HPP7 probe will be repaired or replaced at SIS's discretion.

Repair costs: Current repair or replacement costs may be obtained by contacting Scientific Instrument Services.

1027 Old York Rd. Ringoes, NJ 08551 USA 1-(908)-788-5550 1-(908)-806-6631 FAX Web/E-Mail: http://www.sisweb.com

Use photocopy of RMA form on next page and ship to:

RMA#___

Repairs Dept. Scientific Instrument Services, Inc. 1027 Old York Rd Ringoes, NJ 08551 U.S.A.

Please include the following on the shipping label:

 RMA#_____

 Sender_____

 Sender Address______

 Sender Phone #_____

 Sender e-Mail______

RMA number is required on shipping label Shipping insurance is recommended.

Note: **Do not** ship the probe mounted in the U-shaped bracket. This will increase the possibility that it will be bent. It is best to use original packaging to insure safe shipping.

A. SIS Return Authorization Form

Send	er
Send	er Address
Send	er Phone #
Send	er Fax #
Send	er e-mail
Mode	el #
Seria	1 #
Purcl	hase date Ship date
Purcl	hase order number
Warr	anty Service Y N
Desc	ription of Problem
Pavn	nent Method:
,	
A.	Credit Card: Type: Amex MC Visa
	Number
	Expiration Date
	Credit card holder's name
	Cardholder's Signature
R	PO
D.	(SIS Account # required)
	(SIS Recount # required)
Send	er's Signature:Date
B. SIS Registration Card

HPP7 Direct Insertion Probe and ProbeDirect[™] Software

Purchase date
P/O number used to purchase HPP7
HPP7 Serial number
End User: Name Address
Phone FAX e-mail
Institution: Name Primary business
I would like to receive information on the following SIS products:

- ____ 450-page Scientific Supplies Catalog
- ____ ChemStation Enhancements
- ____ Thermal Desorption
- ___ Cryo-Traps
- ____ Scientific Software
- ____ Filament Repairs
- ____ Mass Spectrometer Supplies

Please mail this page in the enclosed envelope to: Scientific Instrument Services, Inc. 1027 Old York Rd. Ringoes, NJ 08551 USA

Registering your product helps us provide technical support and allows us to notify you of important updates.