

Agilent Sample Introduction Pump System SIPS 10/20

User's Guide



Agilent Technologies

Notices

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Safety Notices

CAUTION

A **CAUTION** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

WARNING

A **WARNING** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a **WARNING** notice until the indicated conditions are fully understood and met.

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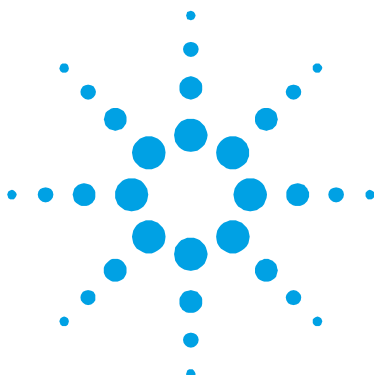
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General

Operation of the Sample Introduction Pump System (SIPS) accessory involves the use of high voltage and moving parts. Careless, improper or unskilled use of this accessory can cause death or serious injury to personnel, or severe damage to equipment and property.

Appropriate safety practices have been included in this operation manual and your spectrometer operation manual to help you operate the equipment safely. Read all safety practices thoroughly before attempting to operate your system.

If the equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired.

Verifying Safe State

The following general safety precautions must be observed during all phases of operation, maintenance and service of this instrument.

To ensure continued safety of the instrument after maintenance or service procedures, verify the instrument is returned to a safe state for the user. This includes running performance checks to verify the instruments safety systems are functioning correctly. Check the general condition of the instrument during operation for wear or signs of corrosion that are likely to inhibit function or safety.

Failure to comply with these precautions or with specific warnings elsewhere in this manual violates safety standards of design, manufacture, and intended use of the instrument. Agilent Technologies assumes no liability for the customer's failure to comply with these requirements.

Hazard Warnings

In addition to the hazard warnings specified in your spectrometer operation manual, specific hazard warnings have been included in this operation manual. These warnings state the hazard, describe how to avoid it and specify the possible consequences of not following the instructions. Read all warnings carefully and observe them at **all** times.

Electrical Hazards

The SIPS electronic control module contains electrical circuits, devices and components operating at dangerous voltages. Contact with these circuits, devices and components can cause death, serious injury or painful electric shock. Panels or covers retained by screws on the SIPS may be opened only by an Agilent field service engineer.

A blown fuse should be replaced only with one of the size and rating specified in the text adjacent to the fuse holder. Always ensure that the accessory is turned off and disconnected from the mains supply before attempting any replacement.

Moving Parts Hazard

During normal operation of the SIPS accessory, there are rotating and automatic clamping components. To avoid injury or damage ensure that all fingers, clothing etc. are kept well clear of these parts during operation.

Chemical Hazards

The SIPS accessory may involve the use of materials that are corrosive or otherwise hazardous. Careless, improper or unskilled use of these materials can cause serious personal injury or an environmental hazard.

Always ensure that laboratory safety practices governing the use, handling and disposal of hazardous materials are strictly observed. These safety practices should include wearing the appropriate safety clothing.

Always ensure that the sample introduction system is thoroughly flushed with distilled water and that the contents of the drain or waste container are known and disposed of if necessary before touching any part.

Other Precautions

The pump module of the SIPS accessory weighs approximately 5.5 kg (12 lb). To avoid injury or damage to the pump module, take care when lifting or moving the accessory so as not to drop it.

The use of the SIPS in any manner other than that specified by Agilent may impair the protection provided by the product.

Notes and Tips

A Note or Tip message is used to give advice or additional information

NOTE

This is a note.

Warning Symbols

The following is a list of symbols that appear in conjunction with warnings in this manual and on the spectrometer. The hazard they describe is also shown.

A triangular symbol indicates a warning. The meanings of the symbols that may appear alongside warnings in the documentation or on the instrument itself are as follows:



Corrosive liquid



Electrical shock



Fire hazard



Hot surface



Moving parts



Noxious gas



*Heavy weight
(danger to feet)*







*Heavy weight
(danger to hands)*

The following symbol may be used on warning labels attached to the instrument. When you see this symbol, refer to the relevant operation or service manual for the correct procedure referred to by that warning label.



Information Symbols

The following symbols appear on the instrument for your information.

I	Mains power on
0	Mains power off
	Fuse
	Single phase alternating current
	Caution, disconnect all supplies, risk of electric shock
	High leakage current – ensure proper grounding

Color Coding

The various indicator lights appearing on the instrument and any associated accessories have been color coded to represent the status of the instrument or accessory:

- A green light indicates the instrument is in normal standby condition.
- A blue light indicates that operator intervention is required.
- An orange light indicates a potential hazard.
- A red light indicates danger or an emergency.

CE Compliance

Your SIPS has been designed to comply with the requirements of the Electro-magnetic Compatibility (EMC) Directive and the Low Voltage (electrical safety) Directive (commonly referred to as the LVD) of the European Union. Agilent has confirmed that each product complies with the relevant Directives by testing a prototype against the prescribed EN (European Norm) standards.

Proof that a product complies with the Directives is indicated by:

- The CE Marking appearing on the rear of the product.
- The documentation package that accompanies the product, containing a copy of the Declaration of Conformity. This Declaration is the legal declaration by Agilent that the product complies with the Directives, and also shows the EN standards to which the product was tested, to demonstrate compliance.

Electromagnetic compatibility

EN55011/CISPR11

Group 1 ISM equipment: group 1 contains all ISM equipment in which there is intentionally generated and/or used conductively coupled radio- frequency energy which is necessary for the internal functioning of the equipment itself.

Class A equipment is equipment suitable for use in all establishments other than domestic and those directly connected to a low voltage power supply network which supplies buildings used for domestic purposes.

This device complies with the requirements of CISPR11, Group 1, Class A as radiation professional equipment. Therefore, there may be potential difficulties in ensuring electromagnetic compatibility in other environments, due to conducted as well as radiated disturbances.

Operation is subject to the following two conditions:

- 1 This device may not cause harmful interference.
- 2 This device must accept any interference received, including interference that may cause undesired operation.

If this equipment does cause harmful interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try one or more of the following measures:

- 1 Relocate the radio or antenna.
- 2 Move the device away from the radio or television.

- 3** Plug the device into a different electrical outlet, so that the device and the radio or television are on separate electrical circuits.
- 4** Make sure that all peripheral devices are also certified.
- 5** Make sure that appropriate cables are used to connect the device to peripheral equipment.
- 6** Consult your equipment dealer, Agilent Technologies, or an experienced technician for assistance.

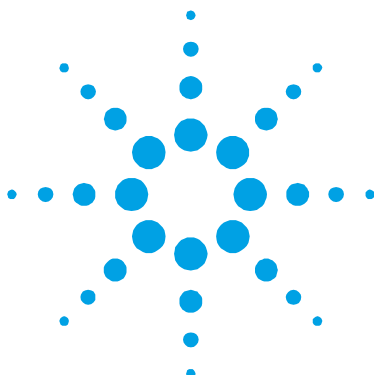
Changes or modifications not expressly approved by Agilent Technologies could void the user's authority to operate the equipment.

ICES/NMB-001

This ISM device complies with Canadian ICES- 001.

Cet appareil ISM est conforme à la norme NMB-001 du Canada.

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2. Introduction

About this Manual

20

The Sample Introduction Pump System (SIPS) is an integrated, efficient dilution system for flame AA and is suitable for use with most Agilent (flame) atomic absorption spectrometers.

In some instruments the control module is built in to the instrument (e.g., the Agilent 280FS AA). For these instruments, installation instructions for the control module can be ignored. It is still necessary to unpack and install the SIPS pump module.

The unique range of capabilities and applications provided by the SIPS includes:

- Online 10 point calibration from a single standard
- Online standard additions calibration from a single standard
- Intelligent online dilution of overrange samples with 'Smart Rinse' to reduce memory effects
- Online preparation of analytical spikes, using a single standard
- Online addition of chemical modifiers such as ionization suppressants, releasing agents or internal standards
- An extended dynamic range for flame AA, approaching that of ICP-OES

Introduction

The SIPS accessory is available as a single pump (SIPS 10) or dual pump system (SIPS 20). The SIPS 10 provides online calibration and sample dilution, while the SIPS 20 provides the added capabilities possible with dual pump operations, including online standard additions calibration, spiking of samples and addition of modifiers.

CAUTION

The SIPS is not suitable for use with organic solvents because the tubing is not resistant to these solvents.

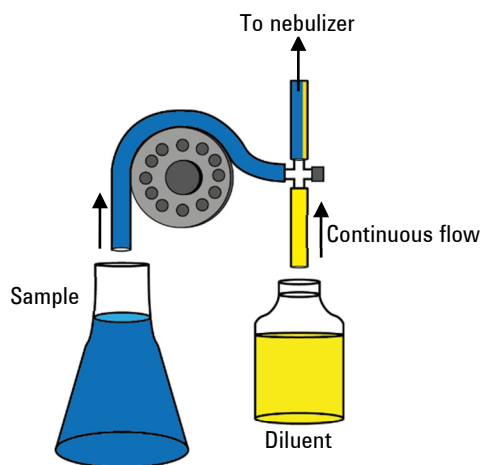


Figure 1 The SIPS 10 single pump unit

The SIPS 10 uses a single pump to combine the sample (or a bulk standard) with a small flow of diluent. Reducing the pump speed results in a reduced flow of sample. With the nebulizer uptake rate constant, the diluent flow is increased, effectively diluting the sample. Using accurate computer control of the pump speed, online dilution and automatic calibration are possible.

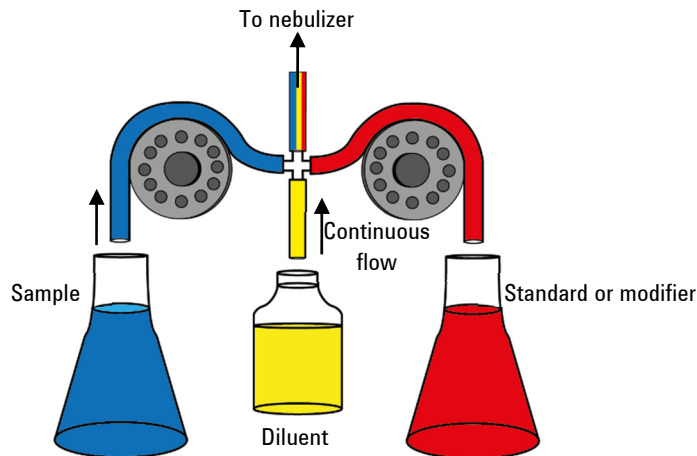


Figure 2 The SIPS 20 dual pump system

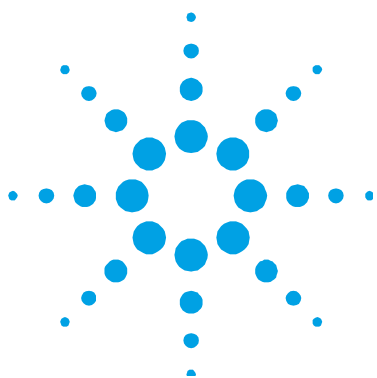
The SIPS 20 uses two pumps to deliver sample and a standard or modifier to the nebulizer. Controlling the relative speeds of both pumps in the SIPS 20 allows the amount of standard or modifier added to the sample to be precisely regulated, facilitating online standard additions calibration, spiking of samples and addition of modifiers. Samples can still be diluted online by reducing the sample pump speed relative to the speed of the second pump.

SIPS supports both manual and automatic sampling with the Agilent flame autosamplers. The diluter option for the Agilent SPS 3 autosampler is not required and cannot be used in conjunction with SIPS, since the SIPS automatically provides all dilution operations.

About this Manual

This manual includes instructions for installing, using and maintaining the SIPS. It is intended for use by laboratory personnel responsible for the day-to-day use of an Agilent AA instrument fitted with a SIPS unit.

The manual does not include instructions for setting up and operating your spectrometer. For these instructions refer to the Agilent Atomic Absorption spectrometer (AA) operation manual supplied with your instrument.



3. Specifications

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Environmental

Your accessory is designed for indoor use only. In addition, this accessory is suitable for the following categories:

- Installation category II
- Pollution degree 2
- Equipment class I

Condition	Altitude	Temp t (°C)	Humidity (%RH) non-condensing
Non-operating (transport)	0–2133 m (0-7000 ft)	5–45	20–80
Operating within performance specifications	0–853 m (0–2800 ft) 853–2133 m (2800–7000 ft)	10–35 10–25	8–80

NOTE

For optimum analytical performance, it is recommended that the ambient temperature of the work area be between 20 and 25 °C (68 and 77 °F) and be held constant to within ± 2 °C throughout the entire working day.

Specifications

Power

NOTE

If you are using an Agilent 280FS AA or 140/240 AA¹, the electronic module is built into the instrument. A separate power supply is not required. The electronic module is powered from the main spectrometer power supply.

Information is correct at time of printing – information on the back of the accessory is the most up to date.

Voltage	100–240 VAC ±10%
Frequency	50-60 Hz ± 1 Hz
Consumption	70 W

NOTE

For safety reasons, any other internal fuse or circuit breaker is not operator accessible, and should be replaced only by an Agilent field service engineer.

External Connections

Mains power cord connector:

Australia	10 A 250 VAC	Complies with AS3112
USA	10 A 125 VAC	Complies with NEMA 5-15P
Europe	6A 250 VAC	Complies with CEE7 sheet vii or NFC61.303VA

Do not replace the power cord with one of a lower rating than specified. Use only Agilent supplied power cords for your country.

Other connections:

- IEEE-488
- Pump module interconnection: 25-pin D-range connector
- Mains inlet coupler: 6 A 250 VAC IEC type.

¹ Factory fitted option

Weights and Dimensions

Weight

Packed 15 kg (33 lb)

Unpacked

- **pump module** 5.5 kg (12 lb)

- **electronic control module²** 3 kg (6.6 lb)

Dimensions (W × D × H)

Packed 620 × 530 × 360 mm
(21 × 24.4 × 14.2 in.)

Unpacked

- **pump module** 275 × 285 × 215 mm
(10.8 × 11.2 × 8.5 in.)

- **electronic control module²** 225 × 100 × 385 mm
(8.9 × 4 × 15.2 in.)

Performance

Typical dilution error is < 2%.

Guaranteed performance (based on 3 × 3 second replicate readings using Integration with 30 second read delay time and 10 samples) is:

Dilution Factor < 50

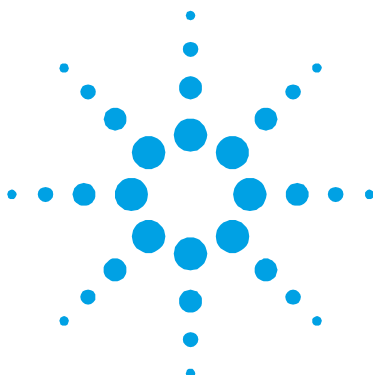
Dilution Error < 3.5%

Precision (% RSD) < 2.5%

² The optional electronic module is built into the Agilent 280FS AA or 140/240 AA, as a factory fitted option.

Specifications

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4. Installation

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Those sections covering the installation of the electronic control module are not relevant when the control module is built into the instrument, such as in the Agilent 280FS AA. It will still be necessary to unpack and install the SIPS pump module.

Installation Checklist

Use the following checklist to help you install the SIPS.

- ❑ Unpack the SIPS according to the instructions and inspect the parts for damage (refer to Page 26).
- ❑ Check that the environmental conditions are correct (refer to Page 21).
- ❑ Check that the correct power supply is available and the correct fuses are installed (refer to Page 27).
- ❑ Install the pump unit and electronic control module (refer to Page 28).
- ❑ Connect the pump unit to the Agilent AA instrument or connect the pump unit to the electronic control module (refer to Page 30).
- ❑ Install the pump bands (refer to Page 32).
- ❑ Prepare rinse and diluent solutions of 0.01% Triton X-100 (refer to Page 39).
- ❑ Install the SIPS diluent and pump tubing (refer to Page 33).
- ❑ Calibrate the SIPS pressure arm force (refer to Page 35).
- ❑ Condition the pump tubing (refer to Page 38).
- ❑ Fit the dust cover (refer to Page 38).
- ❑ Connect the SIPS to an autosampler (if required) (refer to Page 38).

Unpacking

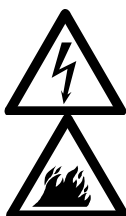
Open the shipping case and remove the upper cardboard sheet. Open each of the enclosed packages with care, inspecting the individual parts for damage which may have occurred in transit. Any damage should be reported to Agilent immediately. Please refer to the accessory by part number and serial number.

NOTE

The serial number of the pump unit is on the underside of the control module. The serial number of the electronic control module can be found on its side, next to the fuses.

Please refer to the packing list that is shipped with the SIPS to confirm that you have received all of the components shipped.

Power

WARNING**Electrical Shock – Fire Hazard**

Application of the wrong supply voltage, connection of the accessory to an incorrectly wired supply outlet, or lack of proper electrical grounding can create a fire or shock hazard that can cause death, serious injury or serious damage to equipment. Do not connect the SIPS to the mains power until you have checked the power requirements as specified on Page 22.

Checking the Fuses

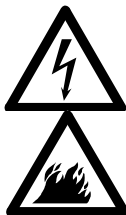
The stand alone electronic control module contains two 5 × 20 mm IEC fuses, located on the side of the module along with the various connection ports.

NOTE

The internal power supply module (280/240 series AA) fuse can only be replaced by an Agilent field service engineer.

Fuses have a code marked on the cap (e.g., T2AH250V). This refers to the fuse characteristic (T - time lag, F - fast acting), the current rating (x amperes), the breaking capacity (H - heavy, L - low), and the voltage rating (y volts).

WARNING



Electrical Shock – Fire Hazard

To prevent reduced safety protection or unwanted fusing, always ensure that the marking on the fuse matches the value screen-printed next to the fuseholder.

To check a fuse:

- 1 Disconnect the accessory from the mains power supply.
- 2 Undo the fuse cap by pressing the cap and turning it counter-clockwise.
- 3 Pull the cap out carefully; the fuse should be held in the fuseholder in the fuse cap.
- 4 Check that the fuse is the correct type and is not damaged. If necessary, replace the fuse in the holder.
- 5 Place the fuse into the cap, push the cap in, then turn the cap clockwise.
- 6 Reconnect the accessory to the mains power supply.

Always ensure both fuses are of the same type and rating before operation.

Installing the Pump Unit and Electronic Control Module

To install the SIPS pump unit on your Agilent AA instrument, insert the hooks on the back of the unit into the slots located on either side of the Agilent AA instrument sample compartment.

WARNING**Heavy Weight**

The SIPS pump unit weighs 5.5 kg (12 lb) and may cause injury or damage if dropped. Always use care when installing or lifting the pump unit.

Before installing the electronic control module, check that it is correctly configured for your Agilent AA instrument. The electronic control module is shipped from the factory configured for installation on most Agilent AA instrument instruments (refer to Figure 3).

NOTE

If you are using an Agilent 280FS AA or 140/240 AA³, the electronic module is built into the instrument. If so, you will not need to follow the steps for installing this module.

To attach the electronic control module to your Agilent AA instrument, insert the hooks on the mounting plate into the vent slots on the rear panel of the instrument. Position the module so it does not cover any of the charts or WARNING information on the rear of the instrument. The top of the electronic control module should be level with the upper surface of the instrument.

NOTE

For Agilent 50/55/140/240 AA instruments, which do not have vent slots, insert the hooks on the mounting plate in the two slots in the rear panel of the instrument.

³ Factory fitted option

Installation

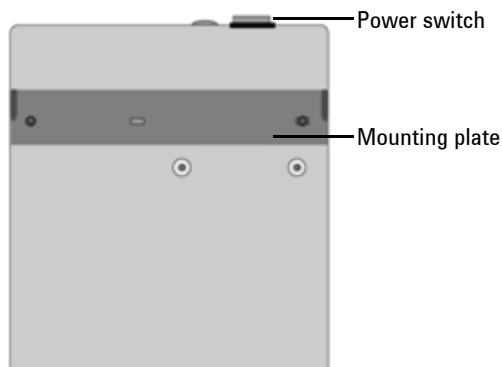


Figure 3 The rear of the electronic control module

Cabling

Connecting to the Agilent AA/Computer

The electronic control module is connected to the Agilent AA by an IEEE-488 cable. Attach one end of the IEEE-488 cable supplied to the electronic control module (the appropriate port is clearly labeled), and the other end to the port on the back of the computer. For Agilent 50/55 AA instruments, which do not require a computer, plug the IEEE-488 cable into the IEEE port on the rear of the instrument.

NOTE

If you are using an Agilent 280FS AA or 140/240 AA⁴, the IEEE-488 connection is built into the instrument.

Connecting to the SIPS Unit

The SIPS pump unit is connected to the electronic control module via an interconnecting cable with 25-pin D-range connectors at each end.

⁴ Factory fitted option

To connect the SIPS unit to the electronic control module:

- 1 Connect one end of this cable to the appropriate port on the electronic control module.
- 2 Secure the cable in place by sliding the metal bracket on the connector to the right, over the studs located on either side of the connector port.
- 3 Connect the other end of the cable to the port located underneath the pump unit on the right-hand side, sliding the bracket on the connector to one side to secure it in place. The cable may be routed around the bottom edge of the instrument, or underneath it.

CAUTION

The SIPS is not suitable for use with organic solvents because the tubing is not resistant to these solvents.

NOTE

If you are using an Agilent 280FS AA or 140/240 AA⁴, the power for the electronic control module is supplied from the spectrometer. The following steps in this procedure are not applicable.

- 4 The SIPS is shipped with three power cables. Select the appropriate one for your location.
- 5 Ensure that the power switch on the electronic control module is turned off (to '0').
- 6 Plug one end of the power cable into the electronic control module and the other end into the mains power outlet.
- 7 You may now turn the SIPS on by pressing the power switch to 'I'. The green indicator lamp should light.

Installing the Pump Bands

To install the pump bands on the SIPS 10/20:

- 1 Select a pump band.
- 2 Loop the band around the (left⁵) roller drum.
- 3 Anchor both ends of the band (attaching the right end first) to the small post located on the left side of the T-piece, as shown in Figure 4.

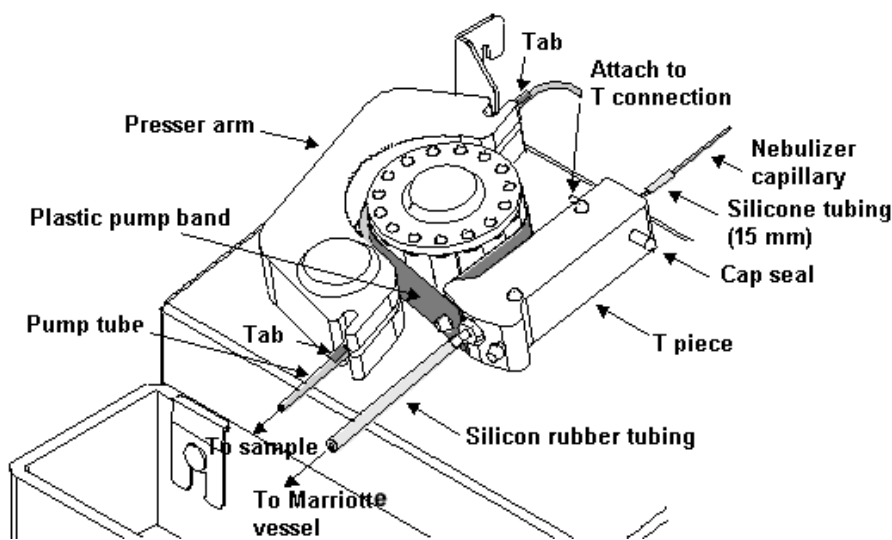


Figure 4 Installing the pump band and tubing on the SIPS 10/20

NOTE

If you are installing pump bands on a SIPS 10, there is only one roller. The following step in this procedure is not applicable.

- 4 For a SIPS 20, repeat the process for the right roller drum, anchoring the two ends of the band to the post on the right side of the T-piece

⁵ The SIPS 20 has two rollers but the SIPS 10 has only one, which has the same layout as the left roller of a SIPS 20.

Installing the Diluent Tubing

Attach a piece of silicon rubber diluent tubing (supplied with the SIPS) between the barbed tube at the bottom of the T-piece (see Figure 4) and the lid of the Mariotte vessel. Ensure the tubing is long enough to avoid kinks or sharp bends.

NOTE

Use one of the Mariotte vessels supplied with the SIPS. Do not use any other container as the Mariotte vessels have been designed to maintain a constant pressure head. The use of any other diluent vessel may introduce dilution errors.

Connecting the SIPS to the Nebulizer

Use a 15-mm piece of silicone tubing (1 mm ID) as supplied with the SIPS, to connect the upper end of the T-piece to a length of capillary tubing leading from the nebulizer (as shown in Figure 4). The length of capillary used should be as short as possible. Ensure that there is no gap between the two ends of the tubing.

Installing the SIPS Pump Tubing

CAUTION

Use only pump tubing supplied and recommended by Agilent. Only this tubing will provide accurate performance.

NOTE

New pump tubing must be conditioned after it has been installed. For details of how to condition new pump tubing, refer to Page 38.

Installation

To install the pump tubing on the SIPS 10/20:

- 1 Remove one of the pump tubes from the plastic bag supplied with the SIPS module.
- 2 Place one end of the tube in the sample vessel (alternatively connect it to your autosampler).

NOTE

It may be necessary to connect a piece of capillary tubing to the end of the tube so that it can reach the bottom of your sample vessel.

- 3 Ensure the power is switched off.
- 4 Gently open the (left⁶) presser arm with the power off.
- 5 Slide the tubing between the front slots in the presser arm, allowing the lower orange tab to hold the tubing in position.
- 6 Fit the tube around the left side of the roller drum, positioning it between the presser arm and the pump band.
- 7 Hook the tube into the rear slot in the presser arm using the other orange tab to hold it in place as shown in Figure 4.
- 8 Fit the free end of the tubing over the left outlet of the T-piece.
- 9 Ensure a cap seal is in place over the right outlet of the T-piece.

NOTE

If you are installing pump bands on a SIPS 10, there is only one roller. The following step in this procedure is not applicable.

- 10 Repeat the process for the right pump, connecting the upper end of the tube to the right outlet of the T-piece (removing the cap seal if one is in place).

⁶ The SIPS 20 has two rollers but the SIPS 10 has only one, which has the same layout as the left roller of a SIPS 20.

CAUTION

Never operate the SIPS without the pump tubing and bands in place, as this may damage the presser arms.

Extending the Pump Tubing

If it is necessary to extend the length of the pump tubing to sample from your test tubes (e.g., for manual sampling), use a suitable length of wide bore capillary tubing (part number 9910024800). This should be connected directly to the pump tube to avoid any unnecessary connectors or junctions, which can cause carryover.

Calibrating the SIPS Pressure Arm Force

This procedure should be completed once only, when the SIPS is first installed – or after the SIPS has been repaired by an Agilent field service engineer. This procedure may be completed by the service engineer at the time of installation.

NOTE

This must be carried out using a new or never used dry tube.

This process optimizes the pressure that is applied to the SIPS tubing during operation. This aims to ensure maximum tube life and correct pumping of solution.

Computer-controlled Instruments

To calibrate the force of the SIPS pressure arm(s):

- 1 Start the SpectrAA software. Turn on the Agilent AA instrument.
- 2 Create a new method called "SIPS PRIMING" which includes an element that uses air/acetylene, such as Cu or Fe. On the Type/Mode page of the Methods dialog select the Use SIPS checkbox. Also set the Sampling mode to Manual. All other parameters can be left at their presets. Save the method.
- 3 Light the instrument flame.

Installation

- Place the inlet capillary tube in a beaker of de-ionized water. Place the outlet tube of the capillary in an empty beaker.
- From the Administration page, select SIPS from the Configuration group. Set the following parameters:

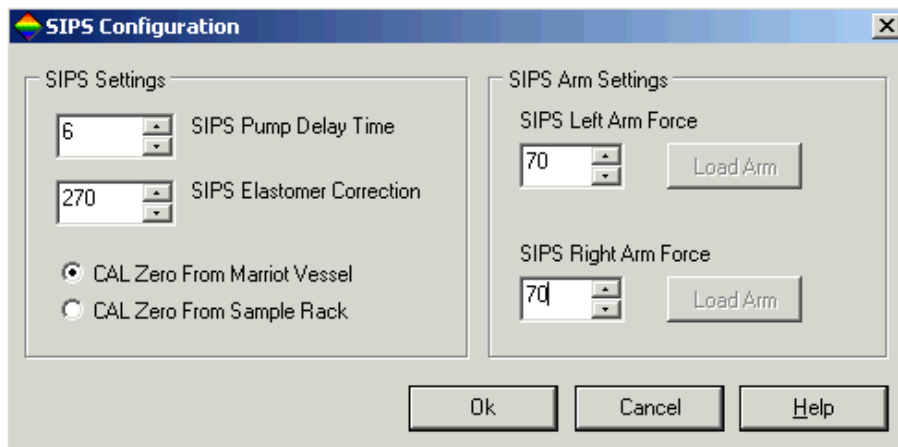


Figure 5 The SIPS configuration page

NOTE

If you are using a SIPS 10, there is only one SIPS Arm Force field.

- Click **Load Arm** to start the SIPS operating. Observe if the de-ionized water in the inlet line starts to pulse or flow up the line. If the liquid in the tube is not moving follow the steps below. If it is moving then go to Step 9.
- Unload the arm by clicking Unload Arm.
- Repeat Steps 5 and 6, each time increasing the: SIPS Arm Force/s by five units until the de-ionized water in the line starts to pulse.
- Increase the SIPS Arm force setting by an additional 5 units.
- Click OK to confirm the settings.

Now condition the pump tube as described on Page 38.

Standalone Agilent 50/55 AA Instruments

NOTE

Calibration of the SIPS pressure arm is only provided in version 1.12 (and later versions) of the on-board software for the Agilent 50/55 AA. The version number is displayed with the Agilent logo as part of the initialization sequence.

If you do not have version 1.12 (or later), contact your local service organization to obtain this upgrade.

To calibrate the force of the SIPS pressure arm(s):

- 1 Turn on the Agilent 50/55 AA instrument but not the SIPS unit. Wait for the initialization sequence to complete.
- 2 Load a method that has the SIPS unit enabled. Fit an appropriate hollow cathode lamp.
- 3 Place the inlet capillary tube in a beaker of de-ionized water. Place the outlet tube of the capillary in an empty beaker.
- 4 Light the flame.
- 5 Access the Service diagnostic page 54 by holding the 'Alt' key down while you type 54 on the numeric keypad. This page is titled _ SIPS (Installation).
- 6 The SIPS arm pressure field on this page has a default value of 120. Type in the number '70' using the numeric keypad and press ENTER to save the value.
- 7 Turn on the SIPS control module.
- 8 Press the Results key to go to the Results page.
- 9 Press the Read key and observe if the liquid pulses in the capillary tubing.
- 10 If the liquid is not moving, switch off the SIPS control unit.
- 11 Repeat Steps 5–10, increasing the SIPS arm pressure setting by 5 units each time until liquid starts to flow.
- 12 Once you have established the setting which allows liquid to flow, add 5 to the value. Access the SIPS Installation page (Alt+54) and type in that number. Press Enter to save this value. This value will now be used every time the instrument is operated.

NOTE

This value should typically be between 95 and 105.

Now condition the tube as described in the following section.

Conditioning the SIPS Pump Tubing

Before each use of a new tube, you should condition the tubing using the following procedure:

- 1 Connect the outlet of the pump tube to a waste vessel.
- 2 Replace the de-ionized water at the inlet tube with 1% Decon or Triton detergent solution. From the instrument page, select 'Condition pump tubes' from the menu. Set the time limit to 15 minutes and press OK. Pump this solution until the time has lapsed.
- 3 Replace the detergent solution with de-ionized water and pump for an additional 30 minutes. This will complete the conditioning of the tube.
- 4 Connect the outlet tube to the SIPS T-piece. The SIPS unit is now ready for regular operation.

Fitting the Dust Cover

To attach the protective dust cover to the pump unit, clip the hooks on the cover over the posts located on the sides of the pump unit. Ensure all tubing is positioned under the opening in the dust cover.

Connection to an Autosampler

If automatic sampling is required, the SIPS can be used with the Agilent SPS 3, SPS 4 or SPS-5 autosampler. Connect the end of the sample pump directly to the PTFE tubing attached to the SPS probe.

NOTE

Ensure that the connection is leak-free to avoid air bubbles entering the flow of sample. Check that the tubing is of long enough to prevent any kinks forming. Try to have a continuous length (i.e., no joins) between the SIPS and autosampler. Fit the small bore probe supplied with the autosampler to the autosampler probe arm when using the autosampler in conjunction with the SIPS.

Rinse Solution

To minimize nebulizer blockage from “spalling” (breakdown of the tube material), you should add Triton X-100⁷ to the rinse and make-up diluent in the Mariotte vessel at a concentration of 0.01 % (mass/volume). This helps wetting of the tubing and ensures that particles pass into the flame.

Replacing the Pump Tubing

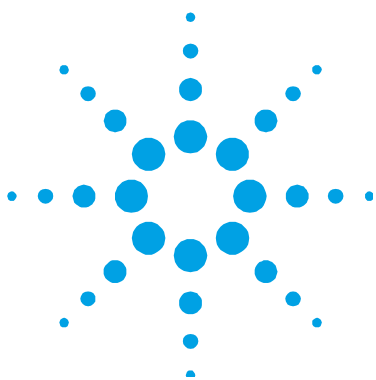
When you need to replace the pump tubing, you should follow these additional maintenance procedures and operational.

- 1 Release the plastic pump band(s) from the T-piece.
- 2 Remove the nebulizer capillary and silicon rubber tubing from the T-piece.
- 3 Lift the T-piece off the SIPS unit and rinse it with water. Use a nebulizer cleaning wire to clean the inside of the metal connectors in the T-piece.
- 4 Place the T-piece in an ultrasonic bath with laboratory detergent for 5 minutes and then rinse it with de-ionized water.
- 5 Test the flow from each of the tantalum arms of the T-piece by using a wash bottle of de-ionized water to force water down each arm. Watch the flow from the other two arms while blocking the large inlet with your finger. You should see the same flow from all outlets.

⁷ Triton X-100 is also known as octylphenol decaethylene glycol ether, CAS No. 9002-93-1, Merck 1.08603.1000.

Installation

- 6** Clean the plastic pump band by wiping it with a tissue moistened with alcohol.
- 7** Using an alcohol-moistened cloth, wipe the pump rollers. Ensure they all free of corrosion and dirt, and rotate freely.
- 8** Clean the nebulizer and Pt/Ir capillary using the standard procedure shown in the SpectrAA Online Help - Cleaning a Blocked Nebulizer.
- 9** Assemble the SIPS system and install the pump tube in place.
- 10** Now condition the pump tube as described on Page 38.

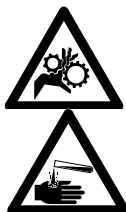


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This chapter assumes you have correctly set up your spectrometer and optimized the system for flame operation. The examples in this chapter are for the PC-based SpectrAA system (refer to the online Help for additional information).

WARNING



Moving Parts – Corrosive Liquid

During normal operation of the SIPS accessory, there are rotating and automatic clamping components. To prevent injury, ensure that all fingers, clothing etc. are kept well clear of any moving parts during operation. The SIPS accessory uses materials which may be corrosive or otherwise hazardous. To prevent injury, always wear appropriate protective clothing when handling hazardous chemicals.

Operational Tips

Whenever new pump tubes have been installed it is necessary to condition the tubing prior to analysis, otherwise signal drift may be observed as the tubing settles in during operation.

TIP

Follow the steps outlined in the 'Conditioning the SIPS Pump Tubing' section, Page 38, to condition new tubing after it has been installed.

Use the following checklist to help you install the SIPS.

- ❑ Never run the tubing dry – it will collapse and accuracy will be affected.
- ❑ Regularly check the diluent in the Mariotte vessel to ensure there is enough solution in the bottle.
- ❑ Check accuracy every 50/60 samples by measuring a standard that has to be diluted.
- ❑ Ensure that the bulk standard has been accurately prepared. This will determine the accuracy of your calibration and your sample results.
- ❑ Make the calibration cover an extended Absorbance range, approaching 1 Abs. This will give a wider dynamic range and a better curve fit.
- ❑ As with all AAs, it is good practice to operate the flame for approximately 30 min to thermally equilibrate the system. During that time, also run the SIPS pumps for 15–30 min to condition the pump tubing.
- ❑ You should edit the SpectrAA configuration file under the SIPS heading, to specify the SipsPumpDelay (in seconds). This allows the solution flow to stabilize after starting the pump or changing pump speeds. This delay must be long enough to allow solution to move from the T-piece to the nebulizer. Recommended settings are 8 s for manual sampling and 12 s for autosampling. Refer to the online help for more information on configuring the SpectrAA configuration file.

Condition previously used pump tubing before each use, using the following procedure:

- 1 Prepare the rinse solution of 0.01% (mass/volume) Triton X-100.
- 2 Dip the inlet tube for the pump in the rinse solution.
- 3 From the **Analysis** page, select **Condition pump tubes** from the menu. Set the time limit to 15 minutes and click **OK**.
- 4 Pump this solution until the time has lapsed. The SIPS unit is now ready for regular operation.

TIP

This procedure can be completed while waiting for the hollow cathode lamp and burner to warm up and stabilize.

Nebulizer Setup

For correct operation of the SIPS it is essential that the nebulizer uptake rate is correctly adjusted. The following procedure must be performed each time new tubing is installed on the SIPS and checked on a regular basis (e.g. daily).

NOTE

This procedure assumes the spectrometer is fully operational, (lamp optimized, flame on, etc.).

- 1 In the **Optimize** window, select to optimize the signal and click the **Start SIPS/Pumps** button.
- 2 Set the nebulizer to the high solids position (wound all the way in).
- 3 Using your finger, block the air vent (the small hole in the lid) on the diluent Mariotte vessel. Very quickly disconnect and then reconnect the diluent tubing from the Mariotte vessel. This will create a small air bubble in the diluent tubing.

NOTE

Make sure that you do not let too much air into the diluent tubing when you remove it from the Mariotte vessel. Allow sufficient time for the air to clear before remeasuring.

- 4 Observe the movement of the bubble in the diluent line. If it is not travelling towards the nebulizer, slowly wind out the nebulizer thimble until the bubble travels towards the nebulizer. It should take approximately 25–30 seconds for the bubble to travel 100 mm in the diluent tubing with the pump running at 100%.

TIP

If the nebulizer blocks, replace the diluent in the Mariotte vessel because blocking causes the flow of sample to be reversed, towards the Mariotte vessel, leading to contamination of the diluent and incorrect results.

Automatic Calibration from a Single Standard

Automatic calibration from a single standard can be performed using either the single pump system of the SIPS 10 or the dual pump system of the SIPS 20.

SIPS 10

To calibrate a method using the SIPS 10:

- 1 Develop or modify a method (flame analysis) as per your analytical requirements (refer to the online help for details).
- 2 To incorporate SIPS operation into the method, enable the Use SIPS check box on the **Type/Mode** page (the first page in the **Edit Methods** dialog).
- 3 On the **SIPS** page:
 - a Set the **Right Pump** setting to **None**.

Automatic Calibration and Dilution of Overage Samples

Overrange dilution can be performed online using either the SIPS 10 or the SIPS 20.

SIPS 10

To perform overrange dilution using SIPS 10:

- 1 Develop or modify a method, following Steps 1–7 in ‘Automatic Calibration from a Single Standard’ section (Pages 44–45).
- 2 Click **OK** to save the method and close the **Method** window.
- 3 On the **Options** page in the **Sequence Parameters** window, select the required **Smart rinse on overrange** error action and the required **Flush time** for the **Smart rinse**. This is the error action that is executed if any contamination is detected after an overrange sample.
- 4 Click **OK** and switch to the **Analysis** window.
- 5 Ensure the diluent tubing is connected to the Mariotte vessel and press **Start**.

NOTE

Follow Steps 6–7 only if the **Sampling Mode** selected for the method is manual (i.e. there are no prompt messages for an Autonormal method).

-
- 6 At the prompt for the standard, place the pump tubing in the bulk standard and press **Read**.
 - 7 At the prompt for Sample 1, remove the pump tubing from the bulk standard, place it in the first sample and press **Read**.

If the sample is overrange, the message 'Solution result over calibration range' appears in the status line after the first replicate. SIPS automatically dilutes the sample online and measurement continues. The dilution factor used is given in the **Datalog** window and the final dilution corrected concentration is reported. When using manual sampling and when the final concentration has been obtained, or if the solution is still out of the calibration range, you will be prompted to present the rinse. Place the pump tubing in the blank solution and press **Read**. SIPS pumps the blank, flushing the overrange solution from the pump tubing.

SIPS 20

To perform overrange dilution using SIPS 20:

- 1 Develop or modify a method as described for SIPS 10 with the following exception: on the SIPS page, select **Bulk Standard** in the **Right Pump** group.
- 2 On the **Options** page in the **Sequence Parameters** window, select the required **Smart rinse on overrange** error action and the required **Flush time** for the **Smart rinse**.
- 3 Press **OK** and switch to the **Instrument** window.
- 4 Ensure the diluent tubing is connected to the Mariotte vessel, and the standard pump tubing is in the bulk standard, then press **Start**.

NOTE

Follow Steps 5–6 only if the **Sampling Mode** selected for the method is manual (i.e. there are no prompt messages for an Autonormal method).

- 5 At the prompt, place the sample pump tubing in the bulk standard and press **Read** (this calibrates the two pumps against each other).
- 6 At the prompt for Sample 1, place the sample pump tubing in the first sample and press **Read**.

If the sample is overrange, an error message, 'Solution result over calibration range' appears in the status line after the first replicate. SIPS automatically dilutes the sample online and measurement of the sample continues. The dilution factor is given in the results window and the final diluted corrected concentration is reported.

NOTE

The following sequence of events is expected when overrange samples are encountered while using SIPS.

- If the sample is out of the calibration range, SIPS reverses the pump for a short period, then stops.
- Diluent is aspirated to rinse the atomization system and the absorbance is monitored as the signal decays.
- When contamination has been removed, SIPS pumps forward at full speed for a short period, then reduces the pump speed to automatically dilute the sample.
- If any contamination is remaining after 30 s, an error message appears and the SIPS Purge error action (specified on the Sequence Error Protocol page) is executed.

Autorun with QC Spikes (SIPS 20)

The automatic introduction of QC spikes is only possible using the dual pump system of the SIPS 20.

To perform automatic addition of QC spikes using the SIPS 20:

- 1 Develop or modify a method as per your analytical requirements (refer to the online help for details).
- 2 To incorporate SIPS operation into the method, enable the 'Use SIPS' checkbox on the **Type/Mode** page in the **Method** window.
- 3 On the SIPS page, select **Bulk Standard** from the **Right Pump** group. If you will be running a very long autorun, with many samples, it may be necessary to periodically recalibrate the pumps during the run. Select the appropriate option from the **Dual Pump Recalibration** group.

- 4 On the **Measurement** page, enter a value for the **Read Delay**. This represents the time required to pump solutions introduced with SIPS through the pump tubing to the T-piece.
- 5 To introduce QC spikes to the method, enable the **QC spike** test in the table on the **QCP** page, and specify:
 - a The required spike concentration in the **Concentration** field.

NOTE

The value entered in the **Concentration** field must not exceed the bulk standard concentration.

- b The error action from the drop-down list.
- 6 On the **Options** page in the **Sequence Parameters** window, select the required **Smart rinse on overrange** error action and the required **Flush time** for the **Smart rinse**.
- 7 Click **OK** and switch to the **Analysis** window.
- 8 Ensure the diluent tubing is connected to the Mariotte vessel and the standard pump tubing is in the bulk standard, then press **Start**.

NOTE

Follow Steps 9–10 only if the **Sampling Mode** selected for the method is manual (i.e. there are no prompt messages for an Autonormal method).

- 9 At the prompt, place the sample pump tubing in the same bulk standard being supplied to the second pump and press **Read** (this calibrates the two pumps against each other).
- 10 At the prompt for Sample 1, place the sample pump tubing in the first sample to be measured and press **Read**. Present each subsequent sample when prompted.

The second pump automatically spikes the samples online, depending on the rate specified in the method.

Online Addition of Chemical Modifier or Internal Standard (SIPS 20)

The online addition of a chemical modifier such as an ionization suppressant can only be performed using the dual pump system of the SIPS 20.

To perform automatic addition of QC spikes using the SIPS 20:

- 1 Develop or modify a method as per your analytical requirements (refer to the online help for details).
- 2 To incorporate SIPS operation into the method, enable the **Use SIPS** checkbox on the **Type/Mode** page in the **Method** window.
- 3 On the SIPS page:
 - a Select **Modifier or Internal standard** from the **Right Pump** group.
 - b Set the sample pump speed by modifying the **Nebulizer Uptake Rate** and the modifier **Pump speed** (in the **Right Pump** group) according to the amount of modifier or Internal standard you wish to add to your samples.

NOTE

To prevent the contamination of the diluent by pumping solution through both pumps at a combined flow which exceeds the nebulizer uptake rate, the sum of the sample pump speed and the modifier pump speed cannot exceed 100%.

-
- 4 On the **Measurement** page, enter a value for the **Read Delay**. This represents the time required to pump solutions introduced with SIPS through the pump tubing to the T-piece.
 - 5 On the **Standards** page:
 - a Set the **Bulk standard concentration**.
 - b Enter the **Standard count**.
 - 6 Fill in the remaining pages as required and click **OK**.
 - 7 On the **Options** page of the **Sequence Parameters** window, select the required Smart **rinse on overrange** error action and the required **Flush time** for the **Smart rinse**.
 - 8 Click **OK** and switch to the **Analysis** window.

- 9 Ensure that the diluent tubing is connected to the Mariotte vessel and that the pump tube for the right hand pump is in the modifier solution, then press **Start**.

NOTE

Follow steps 10–11 only if the **Sampling Mode** selected for the method is manual (i.e. there are no prompt messages for an Autonormal method).

- 10 At the prompt for the standard, place the left pump tubing in the bulk standard and press **Read**.
- 11 At the prompt for Sample 1, remove the left pump tubing from the bulk standard, place it in the first sample and press **Read**. Present each subsequent sample when prompted.

The second pump automatically adds the modifier to the sample on-line, in the quantity specified in the method.

NOTE

The speed of the modifier pump will remain constant throughout the analysis, even when diluting overrange samples, to ensure consistent blank correction and matrix modification. Use the **Flame Facilities** dialog to ensure that the RH Modifier pump is primed before starting an analysis run.

Using the Autosamplers and the SIPS 10

To use automatic sampling with SIPS:

- 1 Connect the sample pump tubing to the PTFE tubing attached to the SPS probe (see the ‘Connection to an Autosampler’ section on Page 38 of this manual).
- 2 Develop or modify a method according to your analytical requirements (refer to the online help for details).
- 3 On the **Type/Mode** page of the **Method** window, select Autonormal from the Sampling Mode group box.

- 4 On the **Measurement** page, enter a value for the **Read Delay**. This represents the time required to pump solutions introduced with SIPS through the pump tubing to the T-piece.
- 5 Fill in the **Standards** and **Sampler** pages as required, then save the method.
- 6 On the **Sampler** page, specify the position of the bulk standard.
- 7 On the **Options** page of the **Sequence Parameters** window, select the required SIPS **Smart rinse on overrange** error action and the required **Flush time** for the **Smart rinse**.
- 8 Develop the remainder of the sequence as desired (refer to the online help for details) and click **OK**.
- 9 Switch to the **Analysis** window and press **Start**.

Single Pump SIPS 20 Operation

To use only one pump of the SIPS 20 unit:

- 1 Develop or modify a method according to the instructions for the SIPS 10 (see the 'Automatic Calibration from a Single Standard' section on Pages 44–45 of this manual).

NOTE

Selecting **None** from the **Right Pump** group on the **SIPS** page when developing a method specifies that the second pump (the right-hand pump) will not be operating.

- 2 If the second pump does not have a pump tube fitted to it, you will need to place a cap seal over the right outlet of the T-piece as shown in Figure 4, Page 32. This is necessary to prevent air bubbles entering the system. If the second pump does have a pump tube in place, it may be left connected. Both presser arms will be closed during operation, and the pressure applied will prevent any solution leaking through the standard pump tubing.

NOTE

Single pump SIPS 20 operation cannot facilitate online standard additions, QC spiking or the online addition of chemical modifiers.

Standard Additions Calibration

SIPS 20

The dual pump system of the SIPS 20 performs standard additions calibration using one pump to deliver sample at a constant rate, while the other delivers standard solution over a range of rates.

To use automatic standard addition calibration with a SIPS 20:

- 1 Develop or modify a method according to your analytical requirements (refer to the online help for details).
- 2 To incorporate SIPS operation in the method, enable the **Use SIPS** checkbox on the **Type/Mode** page of the **Method** window.
- 3 On the **Measurement** page:
 - a Enter a value for the **Read Delay**. This represents the time required to pump solutions introduced with SIPS through the pump tubing to the T-piece.
 - b Select **Standard Additions** as the calibration mode.
- 4 On the **SIPS** page:
 - a Select **Bulk Standard** from the **Right Pump** group. If you will be running a very long autorun, with many samples, it may be necessary to periodically recalibrate the pumps during the run. Select the appropriate option from the **Dual Pump Recalibration** group.
 - b Select the **Calculate LRR during autorun** checkbox if you want to determine the LRR (Linear Reading Range) during the run. Otherwise leave this box unchecked and the value in the entry field will be used as the LRR for that method.
- 5 On the **Standards** page, set the **Bulk standard concentration** and the **Standard count**.
- 6 Fill in the remaining pages in the **Method** window as desired and click **OK**.
- 7 Insert the method into a sequence, developing the sequence according to your requirements (as described in the online help).
- 8 On the **Options** page of the **Sequence Parameters** window, select the required **Smart rinse of overrange** error action and the required **Flush time** for the **Smart rinse**.

Operation

- 9 Click **OK** and switch to the **Analysis** window.
- 10 Ensure the diluent tubing is connected to the Mariotte vessel and the standard pump tubing is in the bulk standard, then press **Start**.

NOTE

Follow steps 11–12 only if the **Sampling Mode** selected for the method is manual (i.e. there are no prompt messages for an Autonormal method).

- 11 At the prompt, place the sample pump tubing in the same bulk standard being supplied to the second pump and press **Read** (this calibrates the two pumps against each other).
- 12 At the prompt for Sample 1, remove the sample pump tubing from the bulk standard, place it in the first sample and press **Read**.

The top addition standard will be checked first to see if it is within the linear reading range at maximum pump speed. If the absorbance of the highest addition standard is greater than the LRR, the sample will be diluted so that the absorbance is less than the LRR. All addition standards will be determined using the same dilution factor. A standard addition calibration will then be performed online.

NOTE

Any subsequent samples analyzed against that calibration (with a recalibration rate greater than one) will be run at maximum pump speed to minimize loss of sensitivity.

SIPS 10

Standard additions with the single pump system of the SIPS 10 is effectively the same as in a non-pump system, however with the added possibility of online dilution. This means that standard additions solutions will still need to be pre-mixed.

To perform standard addition calibration with a SIPS 10:

- 1 Develop or modify a method as described the online help.
- 2 To incorporate SIPS operation in the method, enable the **Use SIPS** checkbox on the **Type/Mode** page of the Method window.
- 3 On the **Measurement** page:
 - a Enter a value for the **Read Delay**. This represents the time required to pump solutions introduced with SIPS through the pump tubing to the T-piece.
 - b Select **Standard Additions** as the calibration mode.
- 4 On the **SIPS** page, select **None** from the **Right Pump** group, then set the **Linear Reading Range** (refer to the online help for more information).
- 5 On the **Standards** page, enter the concentrations of the pre-mixed standards in the table as per non-SIPS operation.

NOTE

The **Bulk standard concentration** and the **Standard count** is disabled since the standard additions are pre-mixed.

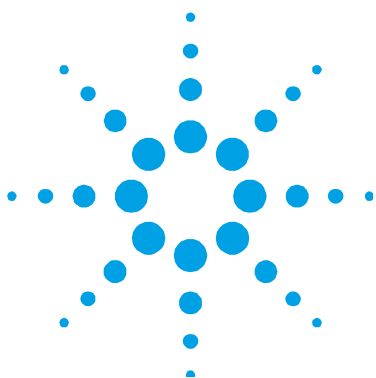
- 6 Fill in the remaining pages as desired and save the method.
- 7 Insert the method into a sequence, developing the sequence according to your requirements (refer to the online help for additional information).
- 8 On the Options page of the Sequence Parameters window, select the required **Smart rinse of overrange** error action and the required **Flush time** for the **Smart rinse**.
- 9 Click **OK** and switch to the **Analysis** window.
- 10 Ensure the diluent tubing is connected to the Mariotte vessel and press **Start**.

NOTE

Follow steps 11–13 only if the **Sampling Mode** selected for the method is manual (i.e. there are no prompt messages for an Autonomal method).

Operation

- 11 At the prompt for the standard, place the pump tubing inlet in the highest concentration pre-mixed standard addition and press **Read**. (This will check the top standard addition to see if it is within the linear reading range at maximum pump speed.)
- 12 At the next prompt, place the pump tubing inlet in the reagent blank and press **Read**.
- 13 At the prompt for the first standard addition, place the pump tubing in the pre-mixed standard addition # 1 and press **Read**. Follow the prompts for the remaining standard additions and subsequent samples.



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The Agilent SpectrAA software contains controls which are specific to SIPS. This chapter provides an overview of these SIPS-specific controls; refer to the SpectrAA Help for additional information on the software.

Method Window

Four pages in the **Method** window for flame operation contain settings for controlling SIPS:

- Type/Mode
- Measurement
- SIPS
- Standards

Type/Mode Page

Select the **Use SIPS** checkbox in the **Online diluter type** group if you wish to use the SIPS unit to introduce your samples to the spectrometer. Enabling this checkbox enables the settings on the SIPS page and various other SIPS related controls, including automatic online overrange sample dilution.

Measurement Page

Two values are relevant to SIPS operation on the **Measurement** page in the Method window.

Read Delay

This field defines the time required to pump solution from the container (vessel or test tube) to the T-piece. This should be approximately 5–10 seconds for manual sampling and 15-20 seconds for automatic sampling with the autosamplers.

Minimum Reading

This field defines the minimum residual absorbance level used in the **Smart Rinse** process. So if the **Minimum reading** entered is 0.1, dilution of a sample that is overrange will start when the absorbance reading during **Smart Rinse** falls below 0.1 abs.

SIPS Page

This page contains the following controls:

Nebulizer Uptake Rate

This field defines the uptake rate of the nebulizer, controlling the maximum sample pump speed to prevent the sample solution being pumped into the make-up vessel.

If your nebulizer has an uptake rate exceeding 5 mL/min you should adjust the nebulizer to reduce the uptake rate which will avoid unnecessary sample dilution and loss of sensitivity.

Right Pump Group

This group allows you to specify what type of solution the second pump (right-hand pump) on the SIPS 20 unit will be pumping.

None – Select this radio button if you have a SIPS 10 unit or are not going to use the second pump of a SIPS 20 unit.

Bulk Standard – Select this radio button if you want to use the second pump on the SIPS 20 to perform all calibration operations. This includes standard instrument calibration, the automatic preparation of QC spikes or performing online Standard Additions calibration.

NOTE

Selecting Bulk standard will force the preparation of standards to be automatic, rather than pre-mixed. When Bulk standard is selected, the Dual Pump Recalibration group becomes active (see Dual Pump Recalibration, Page 62).

Modifier – Select this radio button if you want the second pump on the SIPS 20 unit to add another solution (which is not a standard) to your samples during an autorun.

NOTE

To enable the Pump speed field, the Modifier checkbox must be checked.

Command Reference

You can specify the amount of this solution to be added by nominating a pump speed for the second pump in the **Pump speed** entry field. For example, if you wanted to add 1 mL of modifier to 9 mL of sample (a total of 10 mL), you should set:

$$(9/10) \times 100 = 90\% \text{ for the sample pump speed}$$

$$(1/10) \times 100 = 10\% \text{ for the modifier pump speed.}$$

NOTE

To maintain proper blank correction, the second pump speed will remain fixed throughout the analysis. The first pump speed may be reduced when diluting overrange samples and performing calibrations.

Pump Speed – This entry field controls the speed of the second pump relative to the first pump on the SIPS 20 unit. Refer to the previous section (Modifier) for details.

NOTE

The actual pump speed is dynamic and will change depending upon the value entered for the **Nebulizer uptake rate**.

Internal Standard – Select this button if you want to use the internal standards method. Refer to the online help for a description of this method.

Standard Additions Group

This group is activated only when you select **Standard Additions** as the **Calibration Mode** on the **Measurement** page.

Linear Reading Range – The linear reading range (LRR) is the absorbance range within which the relationship between concentration and absorbance is effectively linear. This defines the point where the calibration deviates significantly from a straight line of best fit. You can either accept the default LRR value or enter your own.

Calculate LRR During Autorun – If this checkbox is selected, the LRR determination procedure is started automatically at the start of each relevant method in the autorun. This limit is determined by generating a 10 point calibration and determining the point at which Chi squared exceeds 0.0002 or the value set in the SpectrAA configuration file. If the determination fails, the default value in the **Linear reading range** field is used. You can, however, perform a manual LRR determination by selecting **Linear reading range** from the **Instrument** menu in the Instrument window. The LRR determined will be displayed in the **Linear reading range** field.

NOTE

If the checkbox is not selected, an LRR determination will not be performed at the start of each relevant method in the autorun and the value in the **Linear reading range** field is used. You can either accept the default LRR value or enter your own.

Calibration Mode

Autoset Standard Concentrations – In this mode, SIPS will automix the standards by diluting the bulk standard. The concentration of these will be set by the bulk standard concentration and the number of standards. The top standard concentration will always match the concentration of the bulk standard.

Manually Entered Standards – Select this checkbox to if you wish to manually enter the concentration of the standards that SIPS will use. SIPS will prepare these standards by diluting the bulk standard. The concentration of the top standard must be \leq the concentration of the bulk standard.

Use Premixed Standards — In this mode the user can set the concentrations for the individual standards on the **Standards** page, these must be premixed by the operator. The location of these is found on the **Sampler** page. In this mode, the SIPS is used for overrange sample dilution only.

Dilute Sample By

Select this checkbox and enter an initial dilution factor to be used for all samples.

Overrange dilution can be performed online using SIPS. If it is expected that samples are over the calibration range, SIPS automatically uses this dilution factor to dilute all samples before measurement.

Dual Pump Recalibration Group

This group is enabled only when the selection for the **Right Pump** group has been set to **Bulk Standard**. It allows you to periodically recalibrate the pumps during a long autorun, to correct for any possible drift which may have occurred, particularly if there is any significant difference in the ages of the sample pump tubing and the standard pump tubing.

NOTE

If the periodic pump recalibration fails, the error message "Periodic pump recalibration failed" appears and the calibration error action selected on the **Options** page of the **Sequence Parameters** window will be executed.

With each Recalibration – Select this checkbox if you want a pump recalibration to occur at the start of each recalibration.

With each Reslope – Select this checkbox if you want a pump recalibration to occur at the start of each reslope.

Standards Page

The following controls are relevant to SIPS:

Bulk Std Conc

This field defines the concentration of the bulk standard solution that the SIPS will use to prepare standards, (in the units specified in the **Units** field on the **Type/Mode** page). The value in this field will be the concentration of the highest standard in the calibration. For example, if the bulk standard is set to 10.0 mg/L, SIPS will prepare a calibration covering the range from 0 to 10.0 mg/L.

Standard Count

The value in this field defines the number of standards SIPS will create for the calibration.

Standards Table

If you selected the Use SIPS checkbox on the Type/Mode page, and did not check Manually Entered Standards on the SIPS page (Default is off), the Concentration column in the Standards table will be unavailable. The SIPS will calculate the concentration of the standards from the Bulk standard concentration and the Standard count such that the standards will be evenly spaced over the calibration range. The concentrations will be displayed in the standards table, but not editable. (Standard additions using SIPS 10 requires the standards to be pre-mixed, hence the Concentration column on the Standards page will be enabled).

QC Tests Page

This page allows you to enable QC spikes, which can be automatically prepared when using the SIPS 20.

The operation of the error action selections for the failure of a QC spike are affected by having the SIPS installed. If the QC spike recovery fails the minimum recovery limit specified on the QC Tests page, the nominated error action will be executed.

Refer to the online help for more information on how to spike samples using SIPS.

Sequence Parameters Window

The only page in the Sequence Parameters window containing SIPS controls is the Options page.

Options Page

This page includes an error action drop down list, **On flame/vapor overrange** in the Smart rinse in the Overage group, which allows you to select the action to occur when a SIPS **Smart rinse** fails.

The **Flush time** defines the maximum period allowed for **Smart rinse** is 30 seconds. If, at the end of this period, the absorbance reading is still greater than the minimum reading value, the message "Unable to achieve blank reading" appears and the error action selected in this list is executed.

Analysis Window

The following SIPS functions are available in the **Analysis** window:

Calibrate Pumps

Selecting **Calibrate pumps** from the **Instrument** menu in the **Analysis** window starts the SIPS pump calibration procedure, allowing you to calibrate the pumps when you are not running an autorun. This button is only appropriate for a SIPS 20 unit (when the **Right Pump** group is set to **Bulk standard** on the **SIPS** page) and will compare the volume pumped by the two pumps when the same pump speed is set for both. The comparison is made by measuring the absorbance level of the same solution. You should place the inlet tubes for both pumps into the same solution. The SIPS unit will then automatically adjust the pump speeds to achieve the same absorbance reading.

NOTE

The pumps will be automatically calibrated at the start of an autorun or at the start of every new method where the **Right Pump** group on the **SIPS** page has been set to **Bulk standard**.

Determine SIPS LRR

Selecting **Determine SIPS LRR** from the Analysis menu of the Instrument window displays the **Determine SIPS LRR** dialog. This dialog allows you to start the Linear Reading Range determination procedure for applicable methods.

This will only be available for those methods where SIPS is selected AND the calibration mode selected is standard additions.

Flame Facilities Dialog

Selecting Flame Facilities from the Analysis menu or pressing F7 in the Instrument window produces the Flame Facilities dialog box. The dialog contains the following controls pertaining to SIPS.

Load

Clamps the presser arm on the pump tubing. Use this button to clamp the pump tubing into place before a manual run.

Unload

Releases the presser arm on the pump tubing. Use this button to release the pressure on the tubing. The tubing lifetime will be extended if you release the presser arm whenever the SIPS unit is not expected to be used again.

NOTE

The presser arm will be automatically loaded before an automatic run.

Purge RH Pump

Pressing this button runs the pump for the rinse time specified in the method to flush out standard or modifier from the tubing of the right pump. This button is enabled for a SIPS 20 unit only.

Purge LH Pump

Pressing this button runs the pump for the rinse time specified in the method to flush out standard or modifier from the tubing of the left pump. This button is enabled for a SIPS 20 unit only.

Stop Pumps

Stops the SIPS pumps.

Flame Optimization

The Optimize Signal dialog box, accessed via the Optimize window (Flame/Vapor) allows you to specify the state of the SIPS for optimization. It is only displayed if the method you are optimizing is a flame SIPS method.

Start Pump

This button starts the SIPS pump running.

Stop Pump

This button stops the SIPS pump.



7. Maintenance

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Keep a maintenance log with details of the maintenance tasks that need to be performed and record when each check is completed.

Clean up spills immediately, using a sponge or cloth dampened with warm water or a mild detergent. Do not use organic solvents or abrasive cleaning agents. If left to evaporate, the spill will leave an acid residue which will eventually corrode the affected surfaces. The acid residue may also be activated by moisture on the hands of the next person to touch that surface.

For details of fuse replacement, refer to Page 27.

Daily

If a new pump tube has been fitted, condition the tube as described in ‘Conditioning the SIPS Pump Tubing’ section (Page 38).

NOTE

When you are using new tubing do NOT connect the tubing to the T-piece for the first 10 minutes of the pump’s operation.

To condition previously used pump tubing before each use:

- 1 Prepare the rinse solution of 0.01% (mass/volume) Triton X-100. Dip the inlet tube for the pump in the rinse solution.
- 2 From the **Analysis** page, select **Condition pump tubes** from the menu. Set the time limit to 15 minutes and click **OK**.

- 3 Pump this solution until the time has lapsed. The SIPS unit is now ready for regular operation.

TIP

This procedure can be completed while waiting for the hollow cathode lamp and burner to warm up and stabilize.

Check that the silicon rubber tubing connecting the T-piece to the Mariotte vessel is secure at both ends and that there are no leaks. If necessary, cut the ends of the tubing by a small amount to improve the connection. When the tubing becomes too short, replace the tubing.

Check that the pump tubes are securely fitted to the inlets on the T-piece. Also check the connections between the silicone tubing and the T-piece at the outlet (top of the T-piece) and those between the silicone tubing and the nebulizer capillary to ensure all connections are firm and there are no leaks. If necessary, cut the ends of the tubing by a small amount to improve the connection. When the tubing becomes too short, replace the tubing.

Inspect the pump tubing before each run. If it is badly stretched or deformed when removed from the pump, or its cross section is oval-shaped, the tubing must be replaced. To ultimately determine if the tubing requires replacement, observe the accuracy and precision obtained when a solution of known concentration is measured.

Check that the nebulizer uptake rate is correctly adjusted using the procedure described in the 'Nebulizer Setup' section, Page 43. Check that there is a small positive flow of diluent from the Mariotte vessel when the SIPS is running at full speed.

To check the uptake rate:

- 1 Go to the **Optimize** page and select the appropriate option for optimizing the signal.
- 2 Present a sample to SIPS and click the **Start SIPS/Pump** button.
- 3 Briefly disconnect the tubing connecting the Mariotte vessel to the T-piece (at the Mariotte vessel) to introduce an air bubble into the tube.

- 4 Reconnect the tubing at the Mariotte vessel again and observe what happens with the air bubble. With SIPS running at full pump speed, the air bubble should be moving slowly towards the T-piece.

If the air bubble is moving quickly, the nebulizer uptake rate is excessive. Although this will not affect SIPS operation, sensitivity will be reduced and diluent consumption will be high. Adjust the nebulizer to reduce the uptake rate as described in the 'Nebulizer Setup' section, Page 43. Check that it is in the high solids position, or an intermediate setting between the high solids and the high vac position.

If the air bubble is not moving at all, or moving back towards the Mariotte vessel, the nebulizer uptake rate is less than the recommended level of 5 mL/min. Adjust the nebulizer to increase the uptake rate (e.g. adjust the capillary so that it is closer to the high vac position). Alternatively, and if it is not possible to increase the nebulizer uptake rate as described previously, modify the method being used to reduce the pump speed from the maximum speed setting (100%), i.e., modify the **Nebulizer Uptake Rate** field on the **SIPS** page.

After making the necessary adjustments, introduce another air bubble in the tubing from the Mariotte bottle and observe if the air bubble is moving slowly towards the T-piece.

To check the nebulizer uptake rate:

- 1 Remove the capillary tubing connecting the T-piece to the nebulizer.
- 2 Fit a normal capillary tube to the nebulizer and measure the uptake rate of the nebulizer.

Record the result obtained. If necessary, modify the methods being used to change the entry for the sample pump speed, i.e., modify the **Nebulizer Uptake Rate** field on the **SIPS** page. This will ensure that there is always a positive delivery of diluent, even with SIPS running at full speed.

Weekly

Wipe clean the rollers on each pump and the pump band (if necessary) with a dry (lint free) cloth. You should also wipe the pump bands with a cloth dampened with alcohol.

Check that the presser arms and other areas in the vicinity of the pump are free from dust, dirt, spills etc. Clean as required using a sponge or cloth dampened with warm water or mild detergent. Do not use organic solvents or abrasive cleaning agents.

Check that each roller rotates freely. If not, contact your Agilent field service engineer for assistance.

Routinely determine a QC Solution of known concentration that is outside the calibration range (i.e. a solution of known concentration that will require dilution). Record the result obtained after dilution with SIPS and monitor how this changes with time. When the result is no longer acceptable in terms of accuracy and precision, this is an indication that the pump bands should be replaced. Once replaced, repeat the measurement. If this still has not restored the accuracy/precision of measurement, the pump tubes also need replacement.

Remove the sample tray at the front of the SIPS and clean with warm soapy water. Dry and refit.

Remove the T-piece by disconnecting the pump tubes, the pump bands and all other tubing. Place the T-piece in an ultrasonic bath containing 0.5% Triton X-100 wetting agent for approximately 5–10 minutes. Using a wash bottle, rinse distilled water through all of the inlets in the reverse direction to normal flow. Refit the tubing and reassemble.

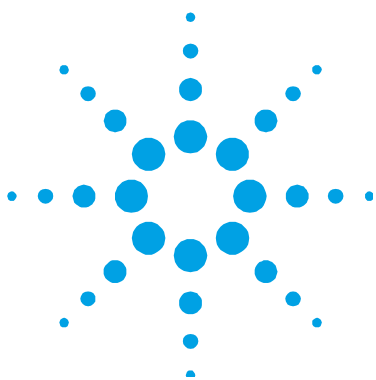
Monthly

If the SIPS has passed the weekly performance checks (using a QC solution with known concentration outside the calibration range) and the SIPS has been used on a regular basis (e.g. at least two to three times per week) replace the pump tubes and pump bands. Remember to condition new pump tubes using the procedure described in 'Conditioning the SIPS Pump Tubing' section, Page 38.

Switch off the mains power to the control module on the rear of the instrument. Check that the control module is firmly seated in the ventilation slots on the rear of the instrument. Check that the connecting cable from the power module to the SIPS pump module is secured. If not, disconnect and refit. When complete, switch the power back on again.

Touch up any scratches on the epoxy painted surfaces. Use a cotton bud (Q Tip) or small brush to apply a small amount of color matched automotive touch-up paint to seal the scratch and prevent corrosion

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8. Troubleshooting

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This chapter discusses the various SIPS related error messages, and offers solutions to problems you may encounter while using SIPS. If the suggestions provided do not resolve the problem, or if you encounter other problems with SIPS, contact your local Agilent office or field service engineer for assistance.

Two Pumps Not Connected

The error message “Two pumps not connected” appears during an autorun.

You may encounter this error message if you have selected an action that requires the dual pump system but only a single pump system has been detected by the software.

SIPS 10

If you are using the SIPS 10, the requested action cannot be performed. The settings for the Right pump group (on the **SIPS** page) should be set to none.

SIPS 20

If you have a SIPS 20 unit, recall the method and check that the setting for the second pump is correct on the **SIPS** page.

Pump Calibration Failed! Switch to Single Pump Operation

The error message “Pump Calibration failed! Switch to single pump operation” appears during an autorun.

The SIPS 20 system attempts to calibrate the pumps so that each pump delivers the same volume of solution when running at maximum pump speed. This message appears when the variation in the standard and sample pumps is too great and the system cannot calibrate the standard pump against the sample pump. The autorun pauses.

- Check all pump tubes and connections for leaks or blockage and replace any worn tubing.
- Ensure that:
 - both pump tubes are placed in the same solution during pump recalibration
 - the pump bands are fitted correctly
 - all pump tubes are the correct type, replacing them if necessary

- the current Read Delay (set on the Measurement page) is being used.

Dual Pump SIPS or Diluter Required to Prepare Standard Additions

The error message "Dual pump SIPS or Diluter required to prepare Standard additions" appears during an autorun

You may encounter this error if you have selected **Switch to Std Additions** as the error action for the failure of a QC spike and you do not have the hardware to prepare QC standard additions installed.

- If you have a SIPS 20 unit, change the option for the right pump to Bulk standard.

Unable to Achieve Blank Reading

The error message "Unable to achieve blank reading" appears during an autorun.

This error message will appear if the final absorbance reading during a **Smart Rinse** fails to fall below the user-defined minimum absorbance limit within the rinse period. This suggests the diluent has been contaminated or some memory effects are remaining.

The SIPS Purge error action specified on the **Sequence** page will be performed next.

This error may also be caused by one of the following:

- Nebulizer blockage, preventing the aspiration of diluent.
 - Check the nebulizer uptake rate and remove the nebulizer for cleaning if necessary.
- The nebulizer is adjusted incorrectly or the maximum pump speed is set too high. This results in the contamination of the diluent since the volume of solution being pumped is greater than the uptake rate of the nebulizer.

- Check the nebulizer uptake rate, adjusting it if necessary to increase it to the required rate of 5 mL/min. You can also reduce the Maximum pump speed to prevent flooding of the nebulizer. Disconnect the Mariotte vessel and replace the diluent solution.
- The value entered for the minimum absorbance reading, used as the contamination test limit, is set too low.
 - Recall the method and check that the minimum absorbance reading is set to a realistic value e.g. 0.005 abs.

No Flame Detected – SIPS Operation Cannot Continue

The error message “No flame detected – SIPS operation cannot continue” appears.

You will encounter this message if you attempt to start SIPS without the flame on, or if the flame goes out while SIPS is pumping. This is to prevent the contamination of the diluent, which occurs if SIPS is allowed to pump solution without the flame on.

Dilution Factor > 50

The error message “Dilution factor > 50” appears during an autorun.

To ensure accurate results, all samples which have been diluted by a factor greater than 50 will be flagged and this error message displayed. The message is only a warning that diluting the sample by such a large factor may introduce unacceptable errors in the results.

Dilution factor > 200

The error message “Dilution factor > 200” appears during an autorun.

The maximum dilution factor possible with SIPS is 200. This message informs you that a dilution factor exceeding 200 is required to get the current solution into range, so the dilution was not performed.

LRR Calibration Failed – Default LRR Value Used

The error message "LRR Calibration Failed – Default LRR Value Used" appears.

This message appears if the linear reading range could not be determined because even the first two readings were nonlinear.

You may use the default LRR value, or dilute the standard.

SIPS Offline or Pump Not Connected

The error message "SIPS offline or pump not connected" appears.

You will encounter this message if you have selected SIPS operation in the method but do not have a SIPS unit installed correctly.

- Ensure all cabling to the SIPS is connected properly.
- Turn the SIPS off and on again to reset communications.

The SIPS Doesn't Pump

- Ensure that:
 - the flame is on
 - SIPS is powered up and online
 - the power indicator light on the electronic control module is lit (if not, call Agilent for service assistance)
 - the pump bands are fitted correctly
 - the IEEE cable is not faulty (try another cable if you have one available).
- Turn the SIPS off and on again to reset communications.
- Check the connection between the nebulizer capillary and the tubing.
- Check the nebulizer or capillary tubing for blockage by disconnecting the capillary from the T-piece to see if any signal is obtained when SIPS is bypassed. Rectify if required.

- If you can hear the SIPS running, but the pump isn't turning, the hub may be slipping. Call Agilent for service assistance.

The Absorption Signal Drifts Over Time

- Check the gas supplies to the instrument—adjust them to near the maximum recommended supply, rather than at the lower end of the range.
- Ensure that:
 - you have conditioned the pump tubing
 - you have allowed the system to warm up prior to starting the autorun.
 - Triton X-100 has been added to the rinse (make-up) and diluent solutions at a concentration of 0.01%.
- If using a SIPS 20, select periodic pump recalibration, particularly if running many samples.
- Check the nebulizer or capillary for blockage by disconnecting the capillary from the T-piece to see if any signal is obtained when SIPS is bypassed. Rectify if required.

Poor Sensitivity

- Check the uptake rate of the nebulizer. If it is greater than 5.0 mL/min, adjust the nebulizer until the uptake rate is between 5.0 and 5.5 mL/min. This gives optimum sensitivity.
- Replace the pump tubes and bands with new ones.
- Check the nebulizer or capillary for blockage by disconnecting the capillary from the T-piece to see if any signal is obtained when SIPS is bypassed. Rectify if required.

Poor Precision

As for poor sensitivity.

- Check the signal graphics. A signal that shows some pulsations (as shown in Figure 6, Page 80) is normal at high dilutions. Increase the integration time or switch to PROMT mode with a long read time to improve precision.

There are Spikes in the Absorbance Signal

Air bubbles may be leaking into the system.

- Check all of your connections, particularly around the T-piece. Ensure that the tubing is pushed completely over the barb.

The Accuracy of the Result is Poor

- Ensure that:
 - you are using the pump tubes supplied and recommended by Agilent
 - you are using one of the Agilent supplied Marriotte vessels to hold your diluent and/or standard
 - the pump bands are fitted correctly
 - your bulk standard is accurately prepared. The accuracy of this standard determines the accuracy of your whole calibration and thus your sample results.
 - there are no leaks at any of the connections in the tubing.
- Check the signal graphics. It is normal to see a signal that shows some pulsation at high dilutions, as shown in Figure 6. Increase the integration time. If using the PROMT measurement mode, reduce the **Precision** and increase the **Read time**.

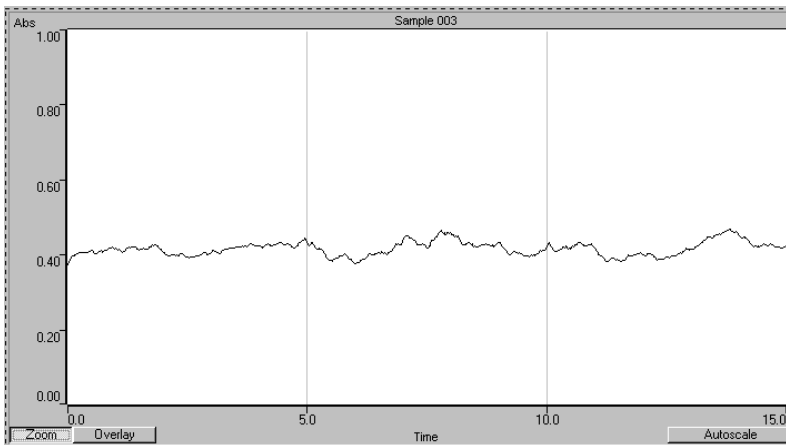


Figure 6 A normal pulsating signal

- Check the nebulizer or capillary for blockage by disconnecting the capillary from the T-piece to see if any signal is obtained when SIPS is bypassed. Rectify if required.
- Check the uptake rate of the nebulizer. If it is less than 5.0 mL/min, increase the nebulizer uptake rate to avoid contaminating the diluent.

In This Guide

The guide describes the following:

- Safety Practices and Hazards
- Introduction
- Specifications
- Installation
- Operation
- Command Reference
- Maintenance
- Troubleshooting

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