

Agilent Auxiliary Gas Module AGM 2

User's Guide



Notices

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CAUTION

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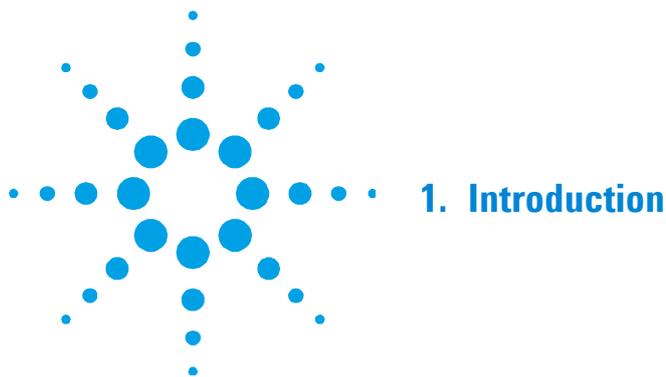
WARNING

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.

Contents

1. Introduction	5
2. Installation	7
Gas Requirements	8
Tubing Attachments	8
Installing the AGM 2 Unit	9
3. Operation	11
The AGM 2 Unit	11
On/Off Switch	11
Mono Flow Meter	11
Snout flow meter	12
Purging Procedure	12
4. Measuring Samples	17
Liberty DOS Software	17
5. Spare Parts	19

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At wavelengths below 189 nm, oxygen in the optical system of the Liberty ICP-AES begins to absorb the energy emitted from the plasma. To perform measurements below 189 nm requires the purging of the optical system with an inert gas. This removes any oxygen present, allowing the far UV wavelengths to be used.

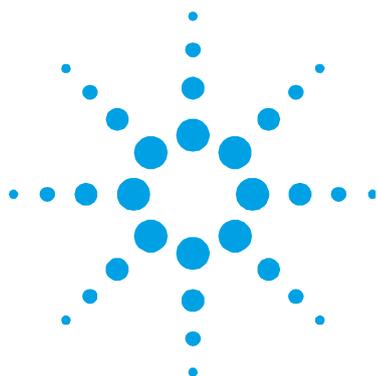
The Auxiliary Gas Module (AGM 2) is an accessory designed to extend the wavelength range of the Liberty 100 ICP-AES spectrometer. It does this by purging both the monochromator and the pre-optics snout assembly with nitrogen. This extends the wavelength range down to 175 nm, allowing the Liberty 100 to be used for the determination of important additional elements, such as sulfur.

The Liberty 100, fitted with the AGM 2 accessory is the best alternative if the far UV region is used infrequently. The Liberty 200 vacuum monochromator provides the best detection limits and stability in the far UV if that region is often used.

NOTE

If you order the AGM 2 after you get your Liberty Series II you need a service call to install the gas outlets on the back of the instrument.

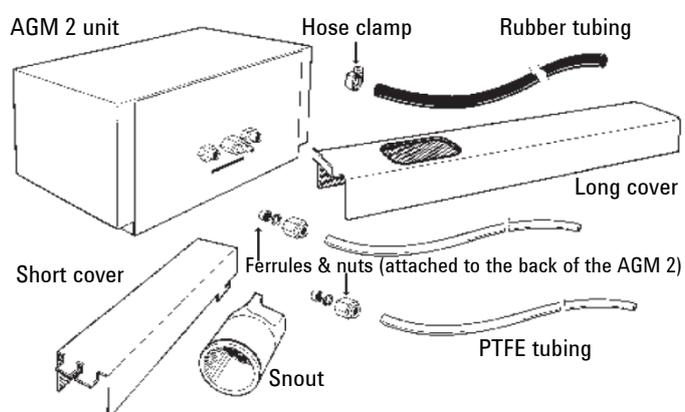
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2. Installation

Gas Requirements	8
Tubing Attachments	8
Installing the AGM 2 Unit	9

You should receive the following components:



You will need

- 1 9/16 in. AF open end wrench
- 1 flat blade screw driver
- 1 brass standard barbed-tail gas hose connector to suit your nitrogen gas outlet
- Access to the back of the Liberty spectrometer

Gas Requirements

All gas supply installations must comply with rules and/or regulations that are imposed by the local authorities responsible for the use of compressed gas in the workplace. The following points should be considered:

We recommend the use of instrument grade nitrogen.

Permissible pressure range: 300-700 KPa

Recommended pressure: 400 kPa

Required flow rates: Up to 30 liters per minute

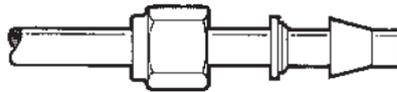
NOTE

Liquid nitrogen is recommended (in conjunction with a heat exchanger) because it is generally cheaper than compressed nitrogen and of better quality. Where compressed nitrogen must be used, the gas must be dry, oil-free and uncontaminated. Do not use compressed nitrogen from a supplier who uses oil or water in the compression process. Only use nitrogen from a supplier who fills containers from immersion pumps lubricated with liquid nitrogen.

Tubing Attachments

Before you install the AGM 2, you need to attach the various fittings to the ends of the AGM 2 tubing.

- 1 Remove the 3 lengths of tubing from the AGM 2 packing box.
- 2 Remove a hose clamp from the box.
- 3 Unscrew the brass nuts attached to the back of the AGM 2 unit. Remove the ferrules from inside the nuts.
- 4 Thread a brass nut and the ferrules onto each piece of PTFE tubing, as shown below:



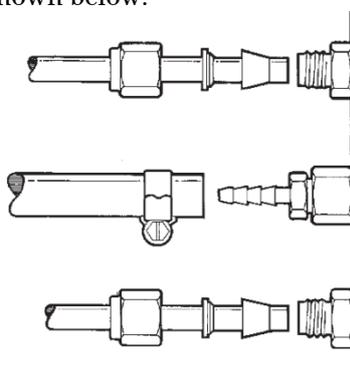
- Slide the hose clamp onto the rubber piece of tubing (do not tighten the clamp at this stage). Fit a gas hose fitting, suitable for your laboratory nitrogen supply, on the other end.



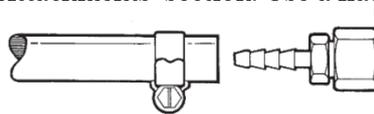
Installing the AGM 2 Unit

To install the AGM 2:

- Remove the AGM 2 unit from the box and place it on a bench.
- Attach the two PTFE tubes to the back of the AGM 2 unit, as shown below.



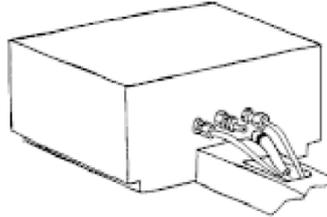
- Fasten the tubing by tightening the nuts with the 9/16 in. wrench.
- Fit the rubber tubing over the barb at the back of the AGM 2 unit.
- Tighten the hose clamp that you fitted in the 'Tubing Attachments' section. Use a flat blade screw driver to do this.



- Standing in front of the Liberty, place the AGM 2 unit on top of the Liberty 100, at the right side of the instrument.
- Lay the hoses on top of the instrument and allow them to reach behind the instrument to the elbow fittings.

Installation

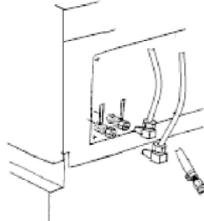
- 8 Remove the lock nut from the brass elbow fittings at the back of the Liberty. Remove the rod and ferrules from inside the fittings.
- 9 Attach the brass ferrules and nuts that you removed in Step 8 to the bare end of both pieces of PTFE tubing, as shown in the 'Tubing Attachments' section.
- 10 Fasten the PTFE tubing to the elbow fittings at the back of the instrument. Tighten with the 9/16 in. wrench.



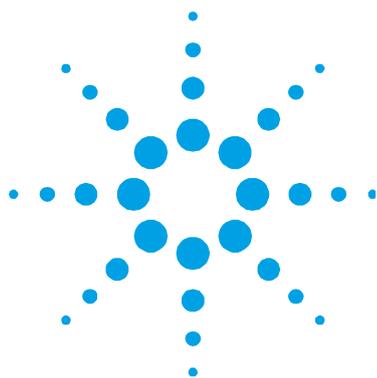
CAUTION

Make sure that you have connected the tubing so that 'snout' goes to 'snout' and 'mono' goes to 'mono'.

- 11 Attach the rubber hose to your laboratory nitrogen outlet (use a brass barbed-tail connector). Do not turn on the gas supply at this stage.



- 12 Move around to the front of the Liberty. Take the 'snout' from the AGM 2 box. Install it by following the instructions in the Liberty operation manual.
- 13 The AGM 2 is now ready for use.

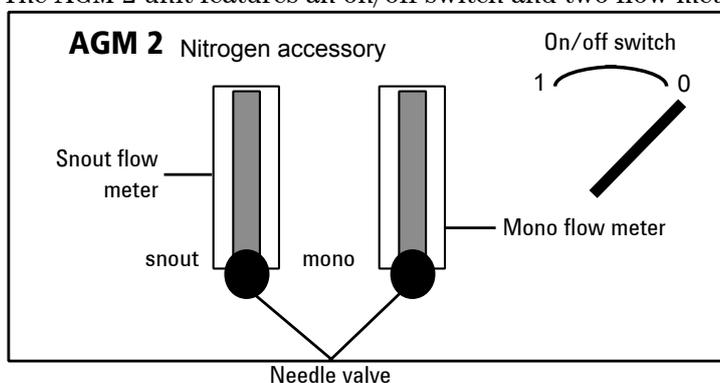


3. Operation

The AGM 2 Unit	11
On/Off Switch	11
Mono Flow Meter	11
Snout flow Meter	12
Purging Procedure	12

The AGM 2 Unit

The AGM 2 unit features an on/off switch and two flow meters:



On/Off Switch

The on/off switch controls gas flow to both flow meters. The gas flow to the monochromator ('mono') and the snout is regulated by the needle valves at the base of the flow meter.

Mono Flow Meter

This meter controls the flow of nitrogen into the monochromator of the instrument. The flow is controlled by the needle valve at the bottom of the meter.

The monochromator requires a high initial purge (20-30 L/min) for 2 to 3 hours, followed by a lower level purge (10 L/min) during operation.

Snout Flow Meter

This meter controls the flow of nitrogen into the 'snout', fitted in the torch box. This is the link between the torch and the instrument optics. The flow is controlled by the needle valve at the bottom of the meter.

NOTE

The two flow meters are different. The 'mono' flow meter allows higher flows. The scales on each are not the same.

The 'snout' requires a relatively low flow (approximately 3 L/min) and short purge times (approximately 15-20 min).

The rates and duration of purging required depend upon:

- The wavelength to be measured—the lower the wavelength, the longer the purge.
- The signal intensity—the lower the intensity, the longer the purge.
- The time since the instrument was last purged—the purge time can be reduced if the instrument was only recently purged.

Purging Procedure

Prior to using wavelengths below 189 nm, the monochromator must be purged with nitrogen. The optimum settings for a particular analysis should be determined experimentally. The following is a typical procedure for the analysis of sulfur at 180.731 nm.

NOTE

The instrument can be used at wavelengths above 189 nm while the monochromator is purging.

- 1 Turn the laboratory nitrogen supply on. Set the gas pressure to 400 kPa (the range is 300-700 kPa).
- 2 Ensure that both flow meter valves are shut.
- 3 Turn on the gas flow to the flow meters by turning the switch to the '1' position.
- 4 Set the 'mono' gas flow to 30 L/min by adjusting the needle valve.

NOTE

The AGM 2 gas meters are marked with arbitrary units. Use the calibration graphs below to determine what the actual gas flow is.

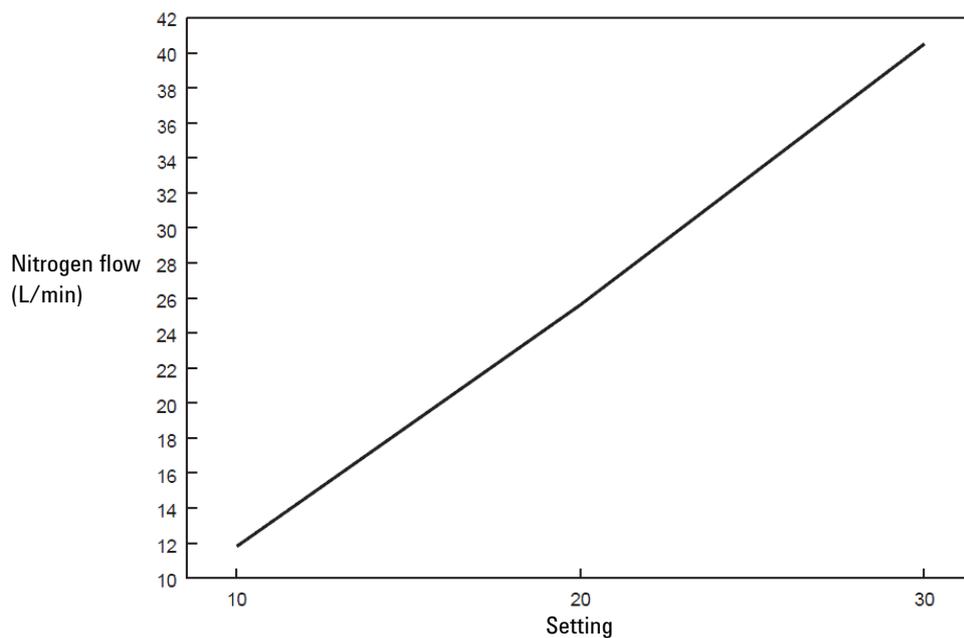


Figure 1. Calibration graph for the 'mono' flow meter

- 5 Purge the monochromator for 3 hours.

NOTE The instrument can be used for normal operation while the monochromator is being purged.

NOTE If you need to perform a quick measurement below 189 nm you can purge the monochromator for a shorter period. The stability of the measurement increases as the purge time is increased. If this is not important for your measurement then you may wish to purge for 1 or 2 hours only.

- 6** After the initial purge time, reduce the 'mono' setting on the AGM 2 to 10 L/min (refer to the 'mono' calibration graph above). Allow an equilibrium time of at least 15 minutes at this setting before calibration or analysis.
- 7** Set the 'snout' flow to 3 L/min (refer to 'snout' calibration graph). The snout flow should be set at least 5 minutes before instrument calibration (this can be the last 5 minutes of the time required for equilibration of the mono).

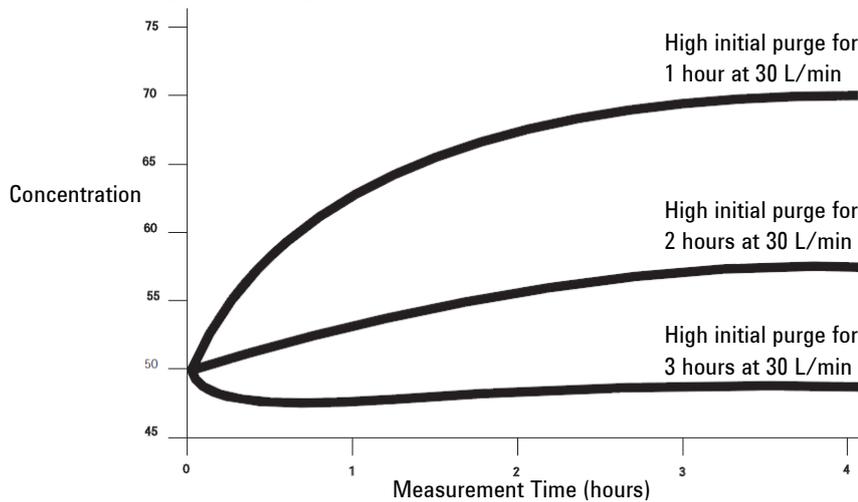
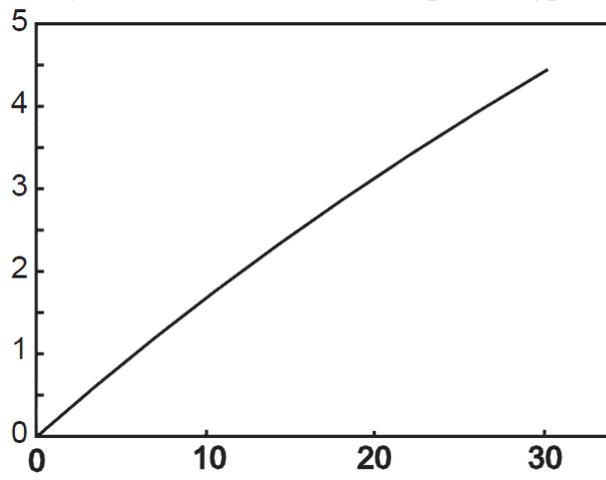
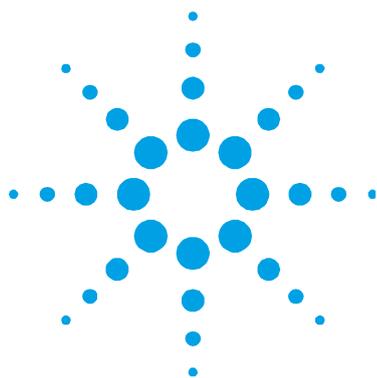


Figure 2. Using 10 L/min measurement purge after various initial purge times

- 8 Proceed with your calibration and measurements in the normal way. Section 4 contains an example of a typical far UV analysis.



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4. Measuring Samples

Liberty DOS Software 17

Liberty DOS Software

The Liberty 100, fitted with the AGM 2, allows you to use the 180.731 nm sulfur line. The following example describes how to measure the sulfur content of a sample using this technique.

NOTE

Refer to the Liberty operation manual for further explanation of the steps described below.

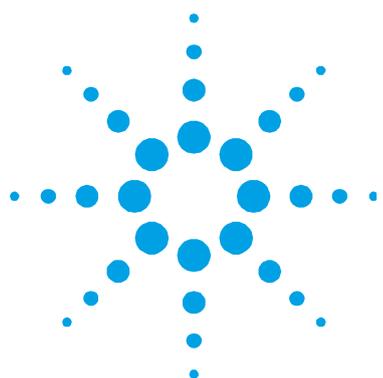
- 1 Purge the monochromator and snout by following the instructions in Section 3 of this manual.
- 2 With the nitrogen gas flowing, setup the instrument as follows:
- 3 On the Instrument Setup page, press **D** to develop a program. This will clear any previous program selections and take you to the Element Selection page.
- 4 On the Element Selection page the highlight will appear in the Element field. Type **S** and press **Enter**.
- 5 The wavelength field is automatically filled with the most prominent emission wavelength for S (180.731 nm).
- 6 Press **Esc** to exit the data block and press **O** to go to the Program Conditions page.
- 7 Turn the plasma on by pressing **Shift + F12**.

- 8** Press **F2** and setup the following conditions:

Viewing Height	8 mm	
Integration	3 s	
Replicates	3	
Power	1.5 kW	
Plasma	12 L/min	
Auxiliary	0.75 L/min	
Pump speed	15 rpm	
Nebulizer	150 kPa*	
Stabilization time	15 s	
Filter 1	Order 1	PMT 650 V

* Set for the nebulizer you are using

- 9** Place the sample capillary into a suitable sulfur standard, allow the solution to reach the plasma and press **F5**.
- 10** The instrument will scan at the S wavelength and display the peak on the screen. Optimization may be performed if required.
- 11** Press **C** to access the Corrections page. Press **F2** to enter the data block. The highlight will be in the mode field.
- 12** Press the **space bar** to select 'Dynamic' background correction, then press **Esc**.
- 13** Press **S** to go to the Standards page and press **F2** to enter the data block.
- 14** Use the **tab** and **arrow keys** to move the cursor to the required position in the table.
- 15** Enter the concentration of the calibration standard in the table. Press **Esc** when finished.
- 16** Press **Enter** to go to the Program Directory page and save the program.
- 17** You can now use either the Rapid Measurement or Sequence Run to measure your samples.



5. Spare Parts

Refer to the Agilent Technologies Web site at www.agilent.com for spare parts ordering information.

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