

Using the Pr, P1, P2 Gas Delivery Manifold

(draft 04/07/16 SWV)

INTRODUCTION

The original Teflon lines going from individual gas cylinders to the Pr, P1, and P2 inlets on the back of the STA449 instrument were prone to oxygen diffusion through the Teflon. The lines have been replaced with a stainless-steel manifold constructed using ¼" OD tubing and bellows-style shut-off valves. All fittings are standard ¼" Swagelok except for short adapter pieces (6 mm Swagelok) that attach to the instrument.

When switching between purge gases, or when minimizing O₂ in the purge gases is required, some of the steps presented below should be followed. Some will be optional, depending upon your needs. It is important that you understand the concepts discussed in this document and the choices available to you in this protocol.

CONCEPT: Understanding the Gas Delivery Manifold

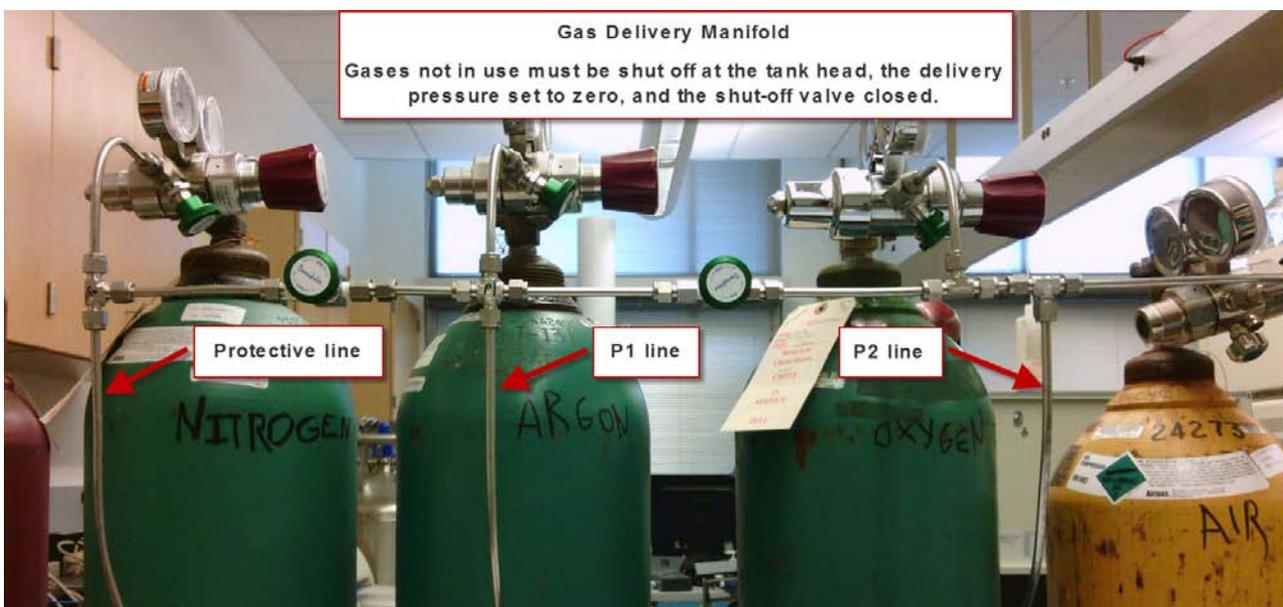
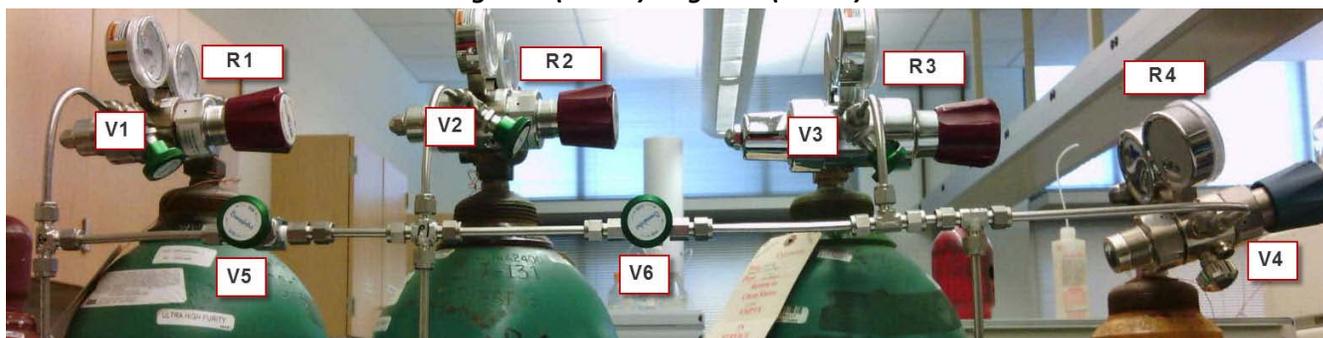


Figure 1 (above) Figure 2 (below)



Normally four cylinders are attached to the manifold. These include Ultra High Purity (UHP) Nitrogen, Argon, Oxygen, and “breathable air”. All four cylinders have high-quality two-stage regulators (R1 – R4). All of the regulators *should* be “low pressure” versions, making it easy to set the delivery pressure at 5-10 psi.

Unfortunately the O₂ and “breathable air” regulators are the normal pressure versions at the moment, so be extremely careful setting the pressure. *NOTE: Pressure must not exceed 10 psi or it will damage the mass flow controllers (MFC’s) in the instrument.*

Become familiar with the manifold. Note where the Protective, Purge 1 and Purge 2 lines are located. Note that the “bellows style” shut-off valves V1, V2, and V3 associated with regulators R1, R2, and R3 cannot leak across the valve to the room air. The V4 shut-off valve associated with the air regulator (R4) is the normal, less expensive style.

Note that separation valves V5 and V6 provide the flexibility to choose between the various gases desired for Pr, P1 and P2.

The protocol for selecting different Pr, P1, P2 gases will involve the following sequence:

- Close the regulator shut-off valves and tank valves for the gases that will no longer be used. The regulators should also be backed off (depressurized).
- Verify that the regulator for the new purge gas is depressurized (backed off).
- Open the tank valve and the regulator shut-off valve for the new purge gas.
- Open the separation valves V5 and V6 so that the Pr, P1 and P2 lines can all be flushed with the new gas.
- Sequentially loosen the three purge plugs at the tee-s that connect Pr, P1, and P2 lines to the instrument. See Figure 3.
- Engage the regulator for the new purge gas until you are able to hear or feel the purge gas escaping around the loosened plugs.

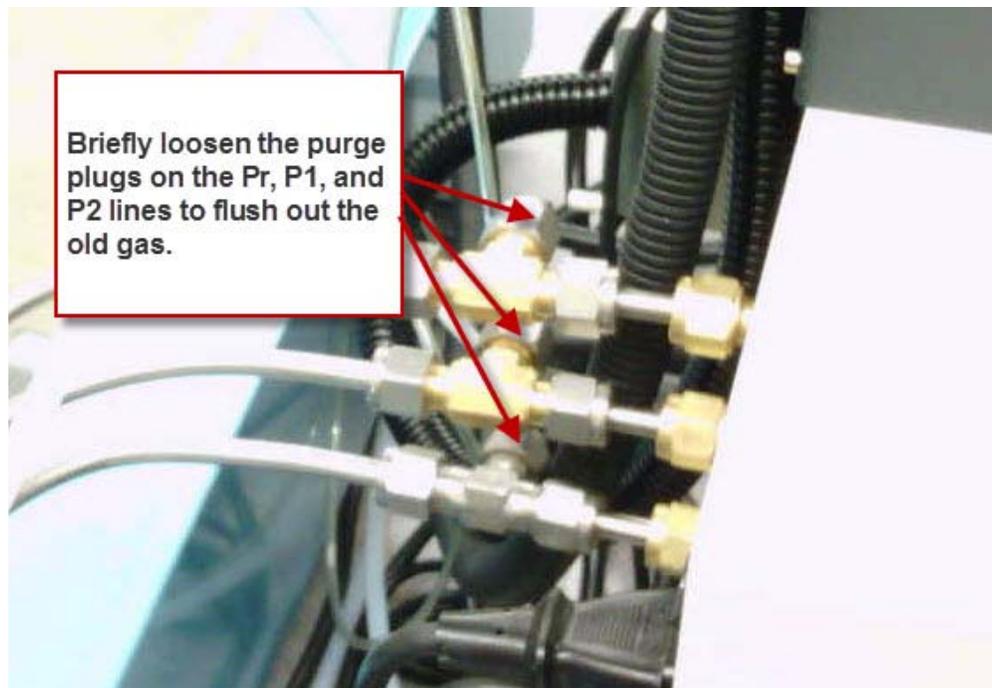


Figure 3

- Purge each line for approximately 30 seconds in order to remove traces of the previously used purge gases, then retighten each purge plug. Do not overtighten. During this process, be sure that the pressure in the lines does not exceed 10 psi or you may damage the MFC’s.

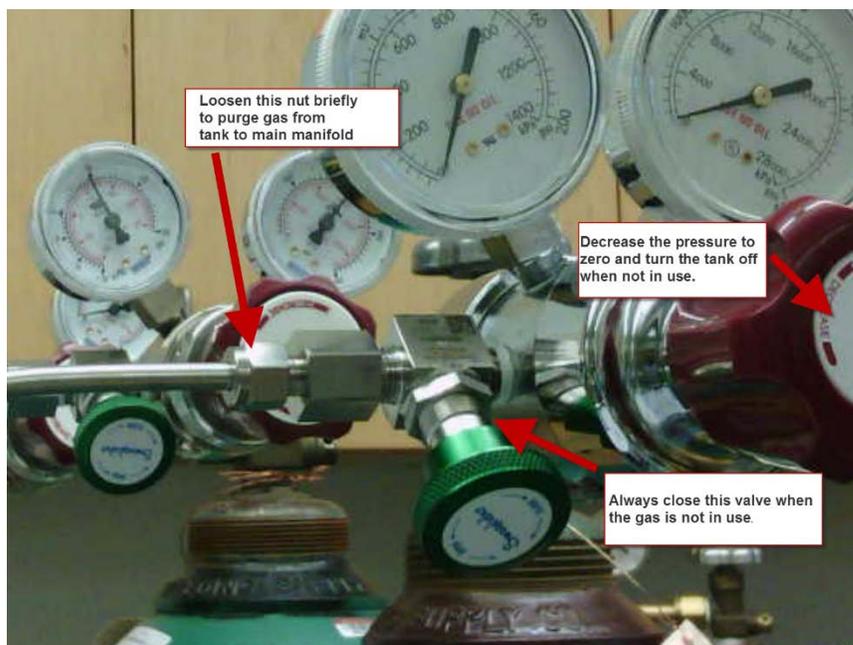


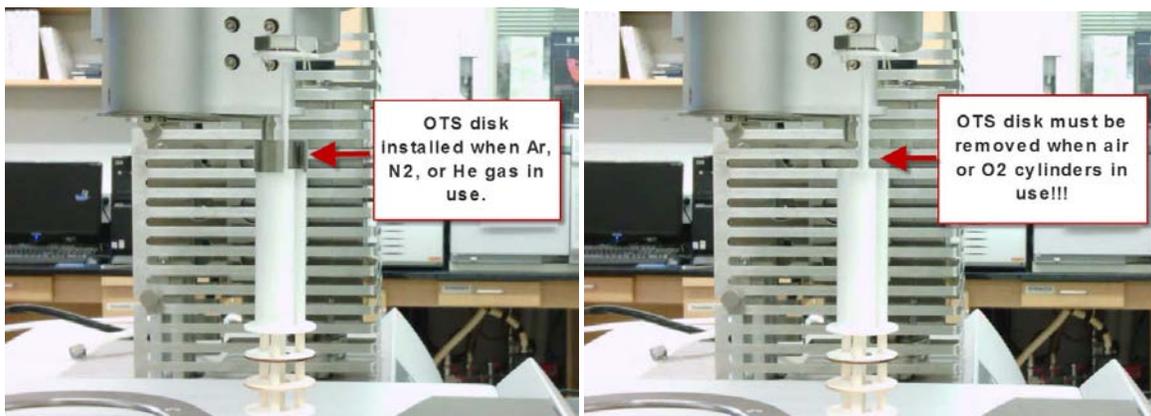
Figure 4

Note that the procedure above does not remove small amounts of gas trapped between the regulator shut-off valve and the main Pr, P1, P2 manifold line. This can be remedied by carefully loosening the Swage nuts that connect to each regulator shut-off valve and purging for 30 seconds. See Figure 4. This part of the purging procedure is optional. The small volume of trapped gas will naturally purge out over 30 minutes of normal instrument operation during the pre-run phase, i.e. after you've selected gases in the STA449F1 program, but haven't yet implemented the macro.

CONCEPT: Using the Oxygen Trap System (OTS)

The OTS system removes traces of residual oxygen in the gas atmosphere inside the instrument. The system becomes effective at temperatures above 400C. If you are using the OTS system, all steps must be taken to purge air from the instrument and all gas lines and connections must be air-tight.

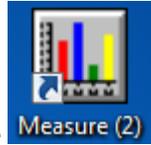
When switching from O₂ or "breathable air" to N₂ or Ar, it is advisable to manually perform an "Evac and Fill" cycle, especially if the OTS system will be used for your measurements. See relevant section below. The OTS disk should only be inserted or removed by Steve. See Figures 5 and 6.

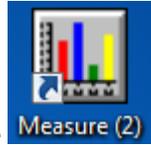


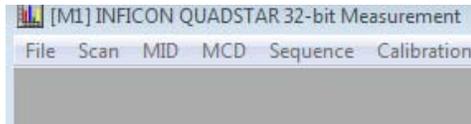
Figures 5 and 6

CONCEPT: Monitoring Purge Gas Impurities using the Mass Spec

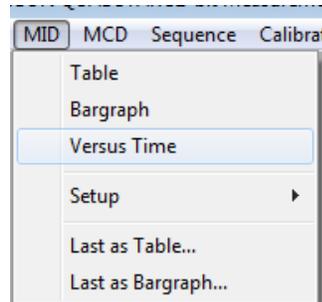
A Selected Ion Monitoring (SIM) parameter set has been created that can be used with the Aeolos "Measure" program located on the desktop. The masses include 18 (H₂O), 28 (N₂), 32 (O₂), 40 (Ar), and 44 (CO₂). This parameter set can be used to view the level of impurities or residual purge gas, and will help you successfully switch-over to a new purge gas.



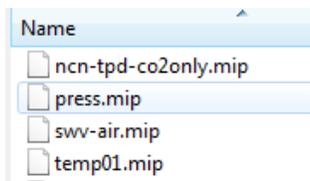
1. Start MS Measure  ->



2. From the MID pulldown, select Versus Time



3. Select the parameter set **swv-air.mip**



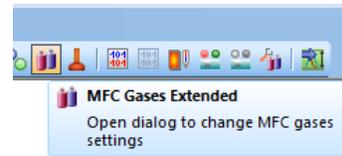
The MS program should start right away. *NOTE: It might present you with a dialog box to turn on the MS filament and the MS multiplier. Do so.* You will see the various m/z traces displayed vs time. The intensity shown in the Y-axis will be a logscale. Note that when a proper purge is established, the ion intensity related to the purge gas should be about 2E-7, while the other ions should all be clustered at about 5E-9, which is the detection limit in this instance.

CONCEPT: Purging Using the MFC Gases Extended Program

To complete the switchover to a different purge gas, it will be necessary to take appropriate action in the STA449F1 program. You are already familiar with this protocol; it's part of the normal training guide.



- Start STA449F1 . Select MFC Gases Extended.



- Set the three gas flows to 0; then select the new gas type you are switching to.
- After selecting the new gas, initially set flows to Pr=60, P1=20, P2=20 to facilitate purging.

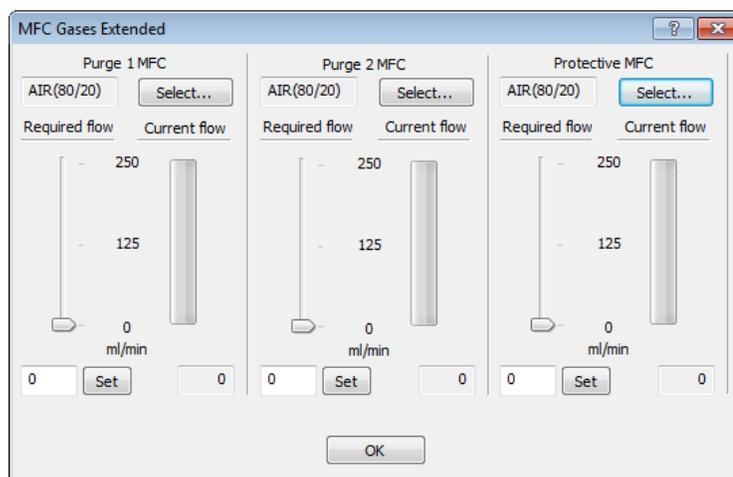


Figure 7

In this example (switching from Ar to Air) the Ar (40) will begin to come down, and the Air 28/32 will begin slowly to rise. Without assistance, it will take a long time to purge out the large volume of residual argon in the balance area. Conversely, the residual volume in the furnace area will purge out in 3-5 minutes.

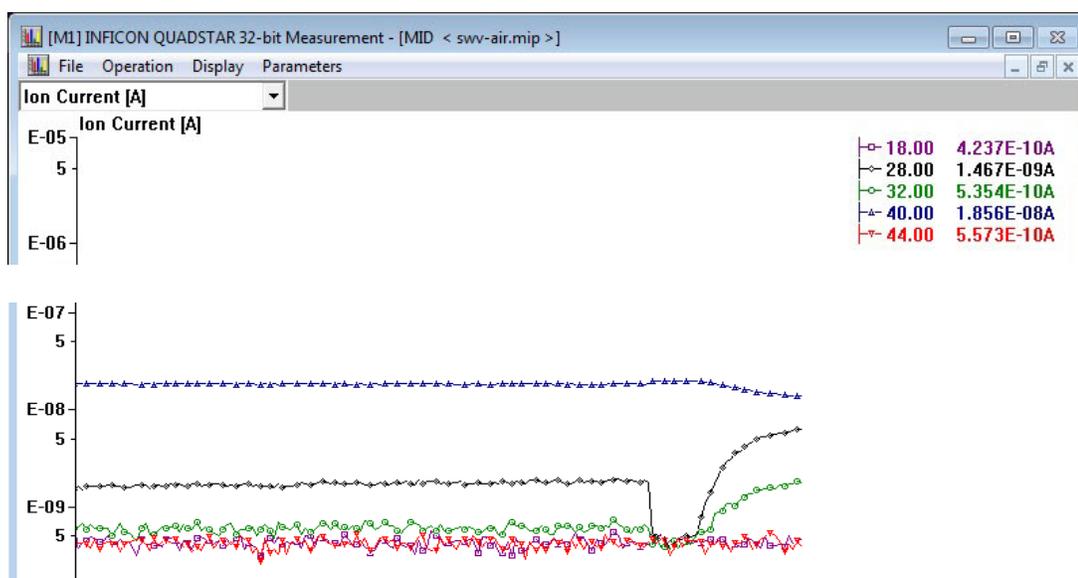


Figure 8

CONCEPT: Manually Controlled Evac and Fill Cycles

Normally “Evac and Fill” is implemented as part of your *autosampler macro*, but there are definitely times when you should use this capability PRIOR to starting an autosampler run.

The normal sequence after changing purge gases, is to:

- Purge the gas manifold lines (discussed earlier).
- Use *STA449F1* -> *MFC Gases* to begin purging within the instrument (discussed earlier).
- Monitor the MS ions; you will still show impurities from the old purge gas trapped in the balance and furnace regions.

To assist the removal of residual gas from the balance area, in STA449F1, select *Diagnosis* -> *Vacuum Controller*. This will bring up the vacuum control panel.

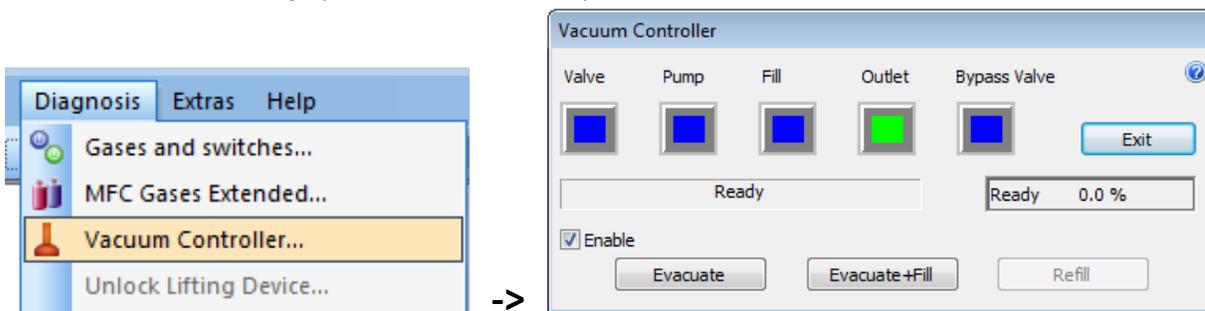


Figure 9

In order to use the Vacuum Controller, you must first select “Enable”. Then select *Evacuate*. All of the ion traces in the MS display should begin to drop precipitously!

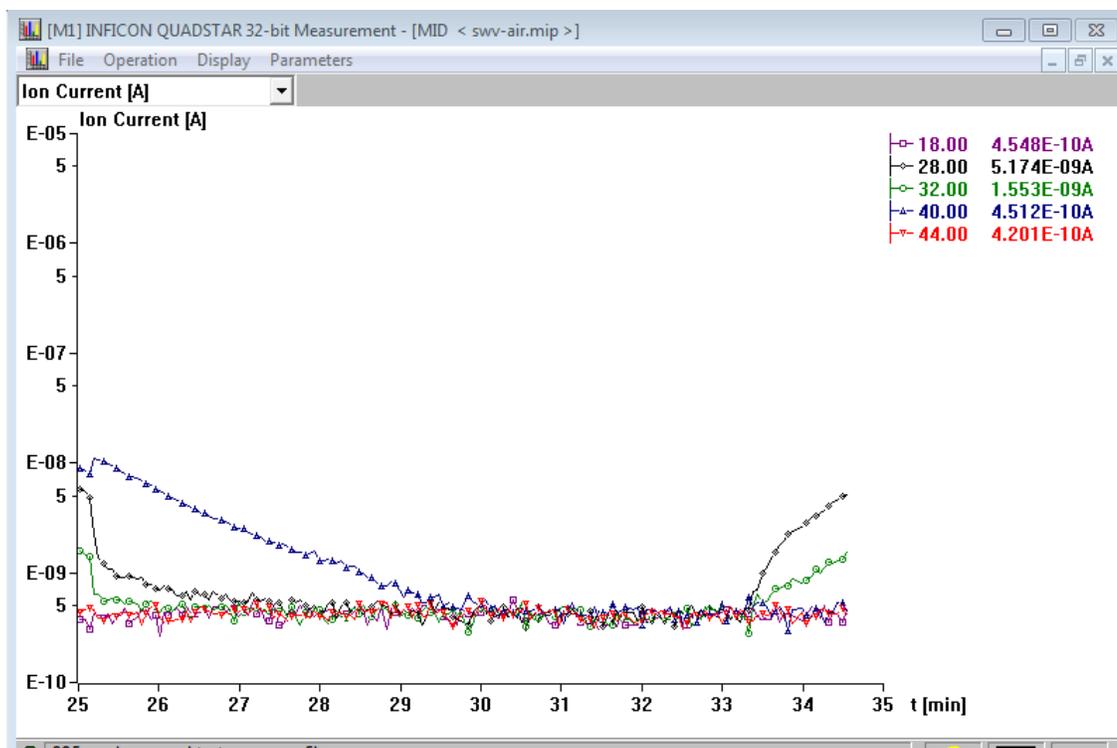


Figure 10

After 4-7 minutes, when all of the ion traces are at the baseline, click on “Refill”. This will also take 4-7 minutes, but now there should be no trace of the previous purge gas and the ions from the new purge gas will maximize. Note that if you have not properly purged the gas manifold lines you may still see residual impurities for a period of time. Set Pr, P1, and P2 to fairly high flows for a few minutes, then do another Evac and Fill cycle. *NOTE: The sum of Pr, P1, and P2 flows should never be set higher than 120 mls/min.*

When finished, be sure to “disable” the Vacuum Controller and use MFC Gases Extended to set the proper flows, e.g. Pr/P1/P2 typically set to 40/10/10.