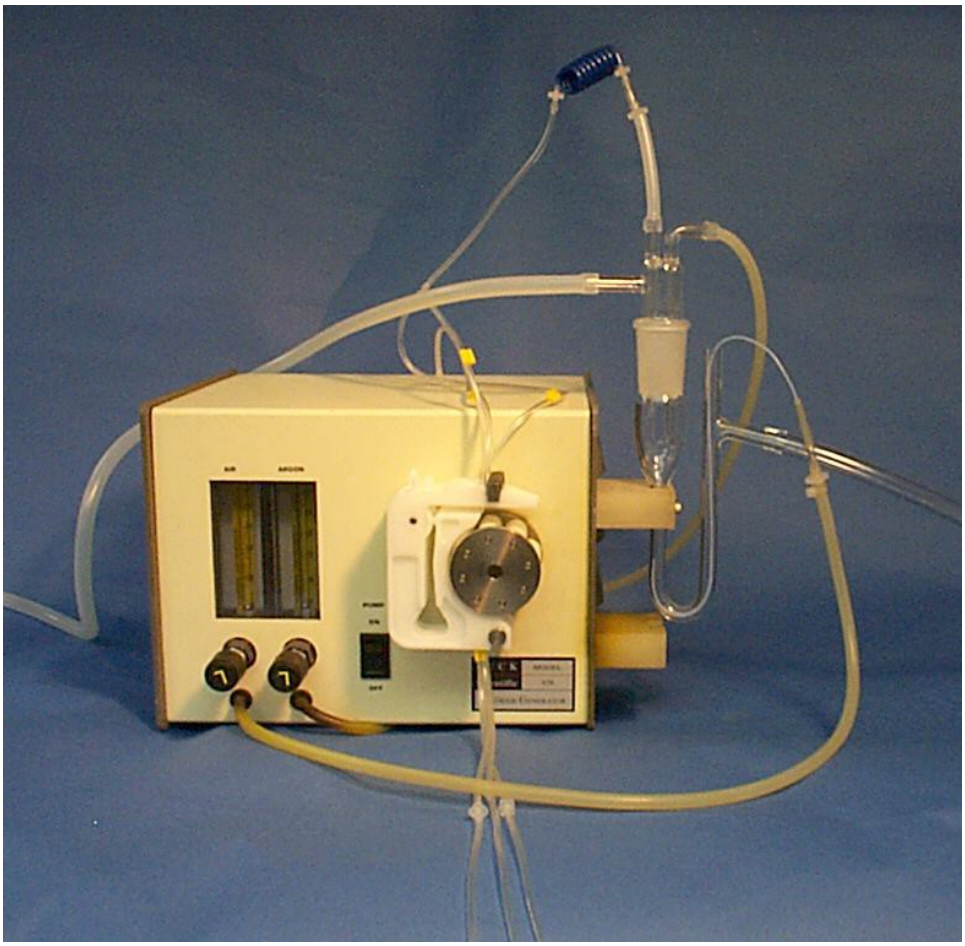
Buck Scientific

Model 420

Continuous Flow Hydride
for low-level determination of Arsenic and Selenium

INSTALLATION AND OPERATION MANUAL

March 2009 / Revision-2



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INTRODUCTION:

Thank you for purchasing the model-420 Continuous Flow Hydride Generator for low-level (ppb) Arsenic and Selenium determinations.

DISCLAIMER: Due to the inconsistencies from performing continuous flow Cold-Vapor reactions for Mercury, the model-420 is not recommended for this work. Please review the performance specifications for the model-1018 Batch-mode system for both Hydride and Cold-Vapor Generation.

The unit is designed for optimal operation with the BUCK Scientific line of atomic absorption spectrophotometers, the model 200A and model 210VGP, but can function with other brands off AA instruments. These modifications are left to the end user, since it would be impossible to accommodate everyone's specific needs.

The model-420 unit is a compact, easy to use system. The glassware and procedures are included for Hydride techniques. If you are missing any parts according to the bill of materials list, please call and we will expedite the material to you.

The model-420 system you have purchased is for continuous flow work on prepared liquid samples. Buck Scientific also manufactures a stand alone Batch-mode unit called the model-1018 Combination Hydride Generator which has options for both Hydride analysis and Cold Vapor Mercury analysis. Due to its more controlled nature, hydride generation is typically done in the continuous flow mode for ease of use.

Regardless of the technique that you are going to use for your samples, it is critical that the samples be properly prepared so the metals are present in a free, ionic form and not bound to organic materials in a complex form. This is the primary reason for low and inaccurate results. A section on generalized sample preparation methods and techniques is present at the back of this manual.

Chemistry also plays an important part in the proper reactions for good Hydride Generation of Arsenic and Selenium. Both the quality and the age of reagents used in the analysis are important to good reproducibility. This is discussed in the operations section.

If you have any questions or problems on the installation or operation after viewing all of the information provided here please give us a call.

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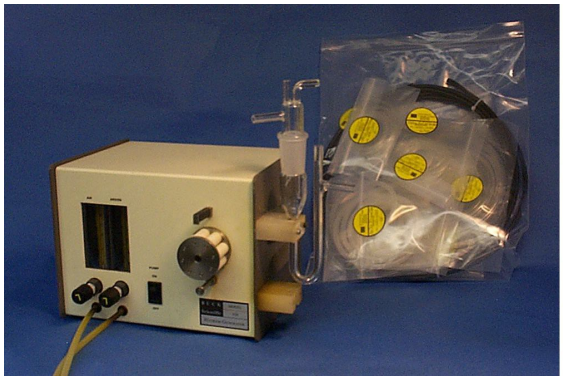
INSTALLATION (Hydride for Arsenic / Selenium):

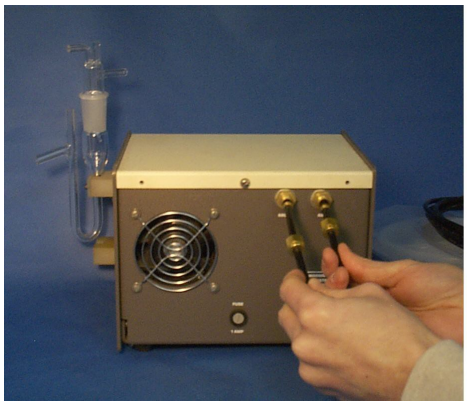
NOTE: Please refer to the pictures for each section for additional installation help

1. Unpack all components from the shipping container. Do not lose small bags with Tubing and Fittings. Check the items against the shipping list shown below to verify all the correct parts have been received.



Model-420 unit with Glassware mounted and bag of Installation components

2. Connect Argon and Air lines to the back using the brass Swagelok Fittings and the black nylon Tubing enclosed with the unit. Connect to the Gas cylinders or Air supply. Recommended inlet pressures are ~15psi, though pressures up to 50psi can be used.



Connect AIR and ARGON Lines to back of unit using included 1/4" Swagelock Compression Fittings on Nylon tubing

3. Loosen the screws holding the white plastic Locking Plate on the right side of the unit. Carefully position the U-shaped Drain Tube and Phase Separator into the UPPER white plastic Mounting Block and secure with the locking plates.

Attach U-Tube and Phase Separator Glassware CAREFULLY! It is fragile and can be easily damaged if bumped!

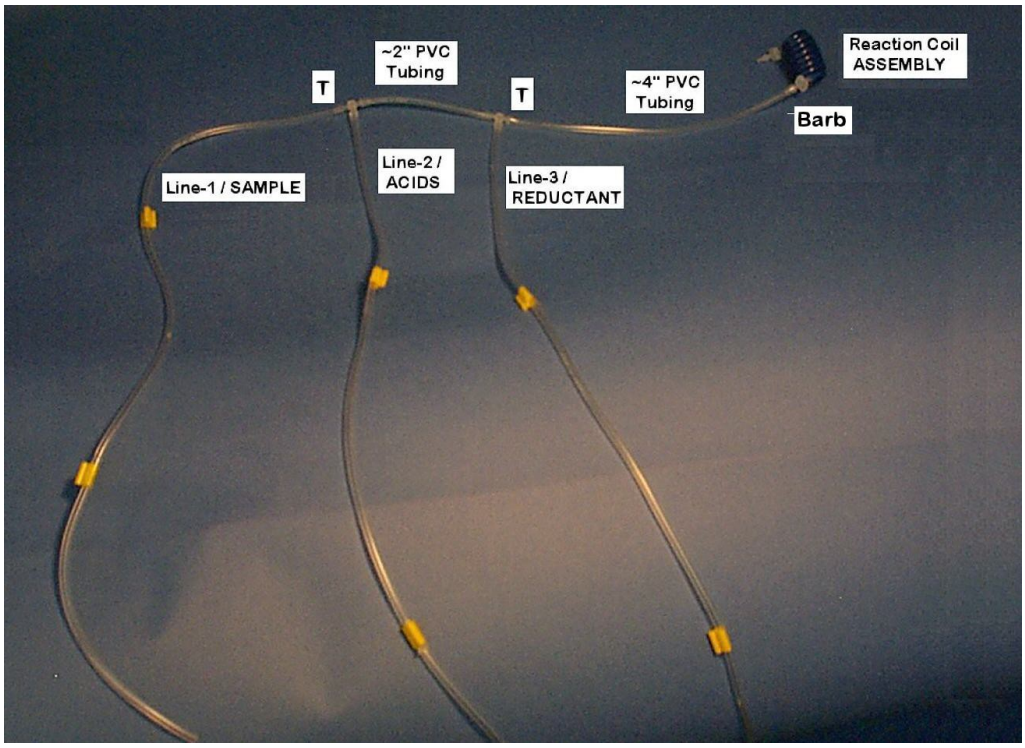
4. The peristaltic sampling Pump has 3 lines available on it. For use with Hydride Generation, the tubing connections are as follows:

Inside line (1) Sample – for introducing Samples and Standards -- This line brings the solution from the Sample bottle to a T-fitting with line 2 then to another T-fitting for line3 and then to the straight arm INLET on the Phase Separator.

Center line (2) Acid -- for Mixed Hydrochloric Acid / Sulfuric Acid diluent to allow reaction to occur. -- This line brings the solution from the Acid bottle to a T-fitting with line 1 and then to another T-fitting for line 3 and then to the INLET.

Outside line (3) Reagent -- for Sodium Borohydride / Sodium Hydroxide reductant -- This line brings the solution from the Reductant Reagent bottle to a T-fitting with a piece of tubing coming from the T-fitting for lines 1 & 2 and then to INLET.

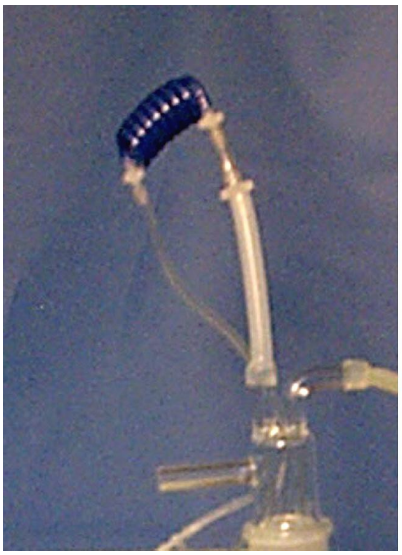
* To the tops of lines 1 & 2 connect together with a small plastic T-fitting. Connect this "T" with ~ 2-4" of clear 1/8" PVC tubing to the other end of the second T-fitting.
* To the top of line 3 connect one leg of the T-fitting then attach the ~2-4" extension tubing from the first T-fitting (lines 1 & 2).



From the ***outlet*** of the second T-fitting connect an ~4-6" piece of clear 1/8" PVC tubing which is connected to the Reaction Coil Assembly with the straight Barb fitting.

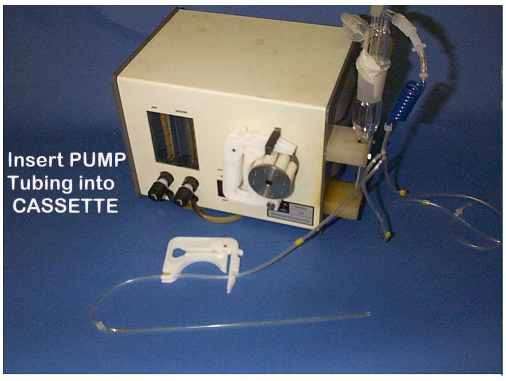
NOTE: Use clear PVC tubing with the yellow tabs (2.54mm ID, pn: 420-1024) for setting up lines 1, 2 & 3. Replace as needed when deformed or brittle.

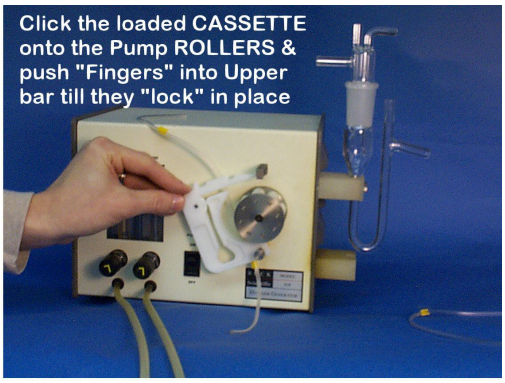
The Reaction Coil assembly OUTLET is connected to the STRAIGHT arm INLET on the Phase Separator by moistening the arm with water, then carefully stretching the ~2" section of white Silicone Tubing over the arm so it sits securely by ~1/2" on Glass arm.

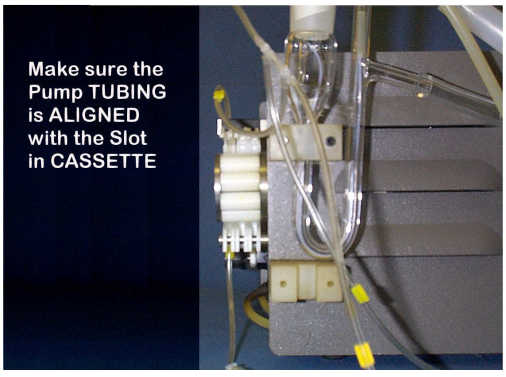


Slide Silicone Tubing from Reaction Coil Assembly over straight Glass arm of Phase Separator so it sits ~1/2" on the arm

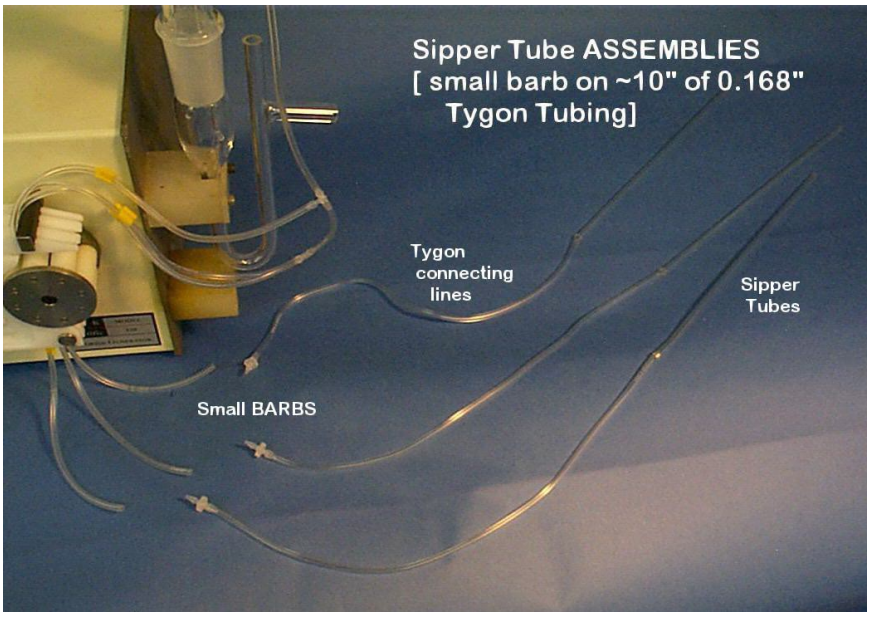
To connect the Pump Tubing, remove the white Tubing Cassette cartridges by pressing down on the fingers above and to the right of the rollers and pushing the Cassette to the left. Insert the appropriate Tubing into each channel and lock the Yellow tab into the lower hole in the Cassette and thread the tubing through to the upper hole. Slide the Cassette back onto the holder and snap it in over the Pump rollers.





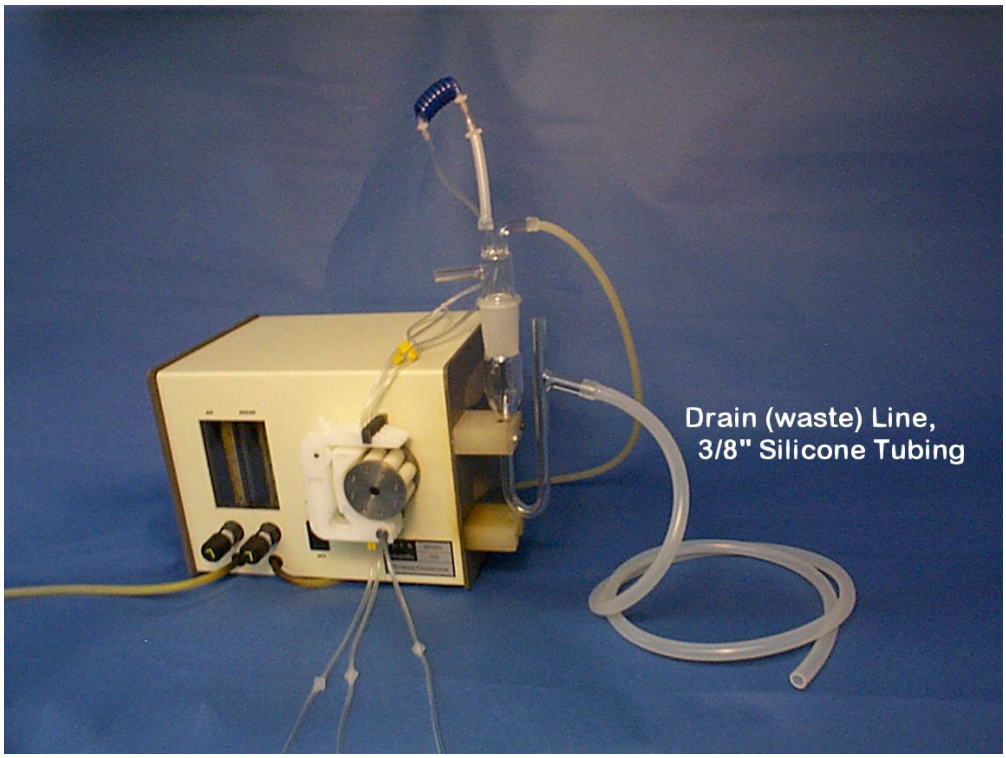


5. The bottoms of lines 1, 2 & 3 are connected to the long Sipper Tube Assemblies with the small, straight Barb fitting. The Sipper Tubes are placed in 1L plastic Bottles containing the appropriate solutions (Samples, Acids and Reductants).

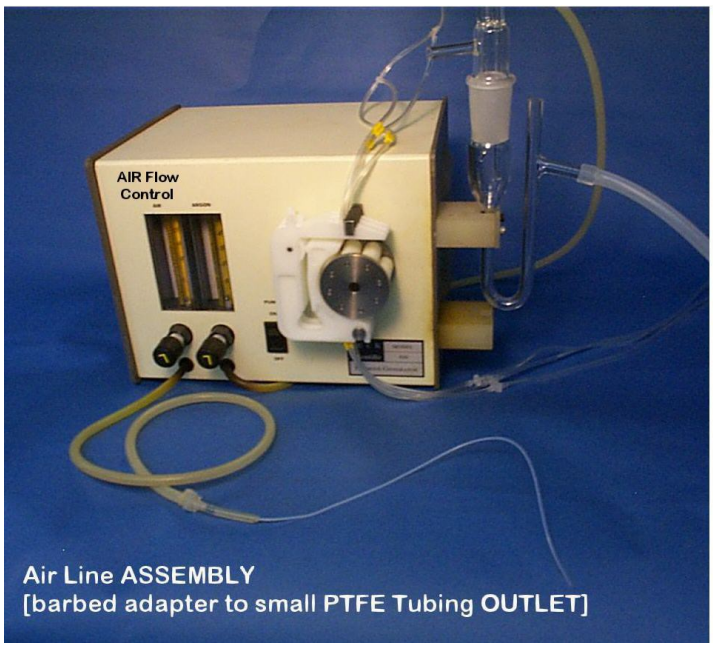


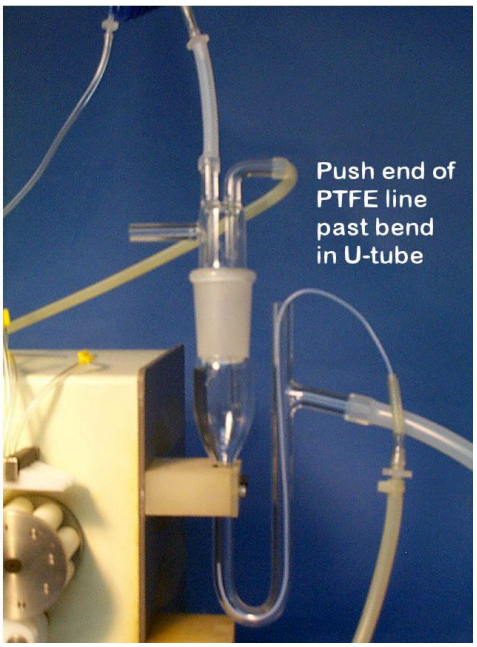
6. Connect the Argon purge line to ANGLED arm of Phase Separator. Set FLOW at ~250 on the Flowmeter, but adjust as needed to optimize individual Response & Stability.

6. Connect the Drain/Waste OUTLET arm to 3/8” Silicone tubing or soft Tygon tubing and place end in the waste container (do not let it get submerged, keep ABOVE liquid level).

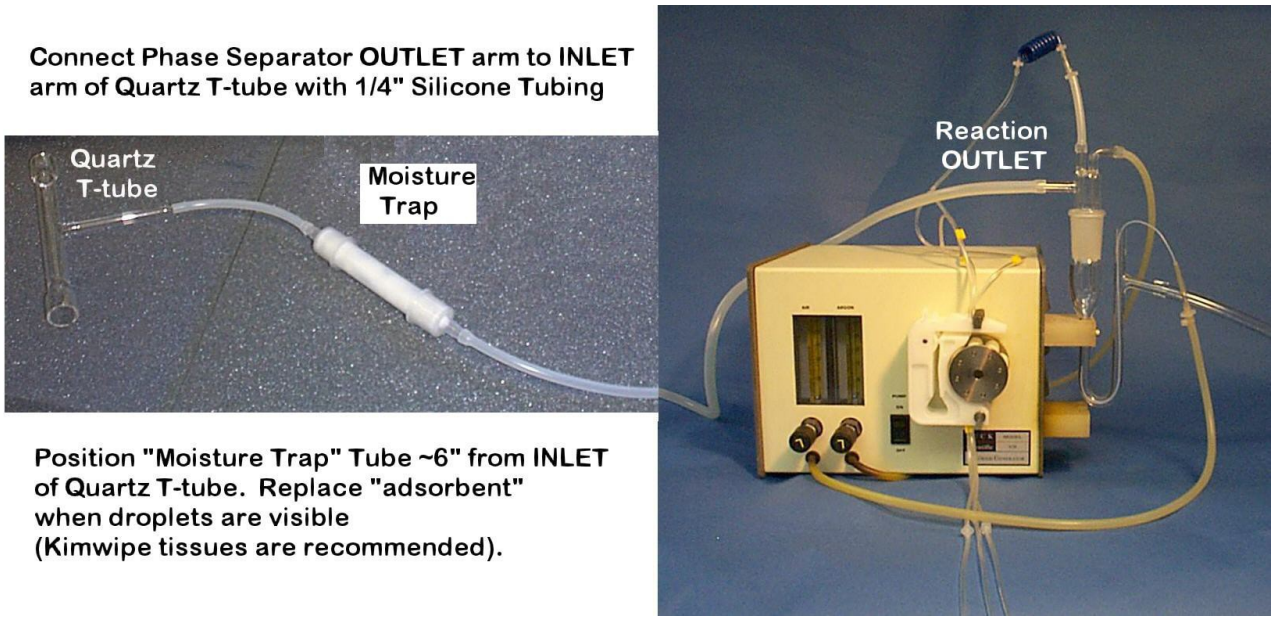


7. Insert the small PFTE Tubing from the Air Line Assembly into the U-portion of the Phase Separator glassware. Set FLOW at ~25 on the Flowmeter, but adjust as needed to optimize individual Response & Stability.

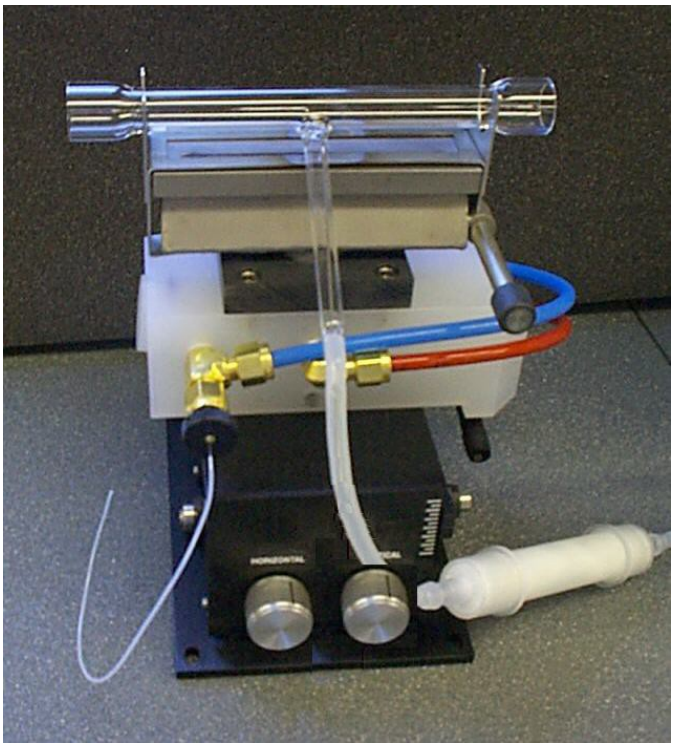




8. Connect the OUTLET arm on the side of the Phase Separator to the Moisture Trap then to the INLET arm of the Quartz T-tube with ¼" Silicone tubing.



9. Place the Quartz T-tube over the Burner Head into the proper Holder, adjusting the Horizontal / Vertical Burner Head positional controls to maximize Hollow-Cathode Lamp ENERGY through the T-tube.



<u>END of HARDWARE ASSEMBLY / INSTALLATION PROCEDURES!</u>

OPERATION:

1. Place the Arsenic or Selenium lamp in holder and allow to warm up for ~ 30 minutes. Set the current (4ma for As / 6ma for Se), wavelength (193.7nm for As / 196nm for Se). Connect the Hydride delivery (Outlet) line to T-tube and place T-tube in the metal Holder bracket over the Burner Head. Maximize the energy going through the center of T-tube. Once the system is aligned and solutions are ready, light the flame and adjust fuel for a lean flame condition (slightly leaner than you would normally use for AA flame work).

2. Fill bottle #1 with Sample solution, acidify to 5% HCl (Hydrochloric Acid), add 0.1% KI (potassium iodide) if needed to reduce any As+5 to the lower As+3 valence state. Repeat for all Sample solutions and put aside for analysis.

3. Fill bottle #2 with 10% HCl (Hydrochloric Acid) / 3% H_2SO_4 (Sulfuric Acid) acid mixture for Arsenic or 4%HCl / 1% H_2SO_4 acid mixture for Selenium.

4. Fill bottle #3 with 1.5% $NaBH_4$ (Sodium Borohydride) in 0.5% NaOH (Sodium Hydroxide). NOTE: This Reductant is NOT stable and needs to be made fresh every 2-4 weeks; depending on Environmental conditions.

5. Calibrate by putting line #1 (inner pump channel) in a DI water Blank (0 ppm), line #2 (center pump channel) in the Acid mix and line #3 (outer pump channel) in the Borohydride solution. Turn on pump switch, wait for signal to equilibrate (~10-30 seconds) and press ***A/Z*** to set your Auto-Zero. Repeat procedure with 2 or 3 Standards from 1-250ppb and use the ***READ*** key to run your Calibration Curve.

   NOTE: Please refer to the model 210-AAS Manual or Mini-Guide (or other AAS manual) for the complete procedure for establishing a Calibration Curve.

6. Aspirate your Blank to flush line #1 and place in Sample solution. Let reading stabilize and press ***READ*** to get ppb concentrations of As / Se in the solution.

7. Recheck several Standards throughout the course of the analysis and use the ***A/Z*** and ***RESLOPE*** keys if necessary to correct for any drift or instability in the system.

PACKING/PARTS LIST:

1	Hydride flow control unit	
1	Instruction Manual	
1	Power cord	
2	1 amp fuse (slow blow)	999-1045
1	Tee tube holder	420-1034
1	Tee tube	420-1016
1	Drying Tube	400-0400
1	Hydride reaction vessel	420-0033
1	Air adapter	420-0040
2	Gas tubing nycoil (10')(w/nuts & ferrules)	990-1855
1	7' 1/4" O.D. silicone tubing	400-0500
1	4” 3/8" O.D. silicone tubing (w/fitting)	420-1603
1	5’ 3/8” O.D. pvc tubing	115-1024
1	14' .1681 O.D. tubing	420-1604
3	Mini Cassette Cartridge	420-1023
6	Yellow-yellow pump tubing (sample)	420-1024
2	10 turn mixing coil	420-0026
6	1/16" straight barb fitting	420-1036
4	1/16" tee fitting	420-1037
6	10" uptake straws	420-1027
3	1 liter bottle	991-1038