

Gemini 2360 Surface Area Analyzer

Updated June 2, 2008

Instrument instructions can be found at:

<http://academic.bowdoin.edu/chemistry/resources/instructions.shtml>

If you have any problems with the instrument or would like to get trained, please contact Celeste Moody (725-3756 / cmoody@bowdoin.edu / Druckenmiller 256)

1. Protocol

- a. **Read instructions carefully before using this instrument.** Please insure that you understand the full procedure before continuing. If you do not, please ask the appropriate questions.

2. Startup Procedure

- a. **Sample vials need to be cleaned under the following procedure:**
 - i. Sonicate the vials in a solution of 10g laboratory detergent per liter of warm water for 15 minutes.
 - ii. With rubber gloves on, rinse the vials in tap water then thoroughly in D.I. water. Dry in an oven for a minimum of 2 hours.
 - iii. Remove from the oven, again with gloves, and allow cooling. Fill with dry Nitrogen then stopper with a cork that has been wiped with a lint free cloth. (Note: quickly stopper to keep the Nitrogen from escaping)

3. Weighing the sample

- a. **Place the sample weighing support onto the scale and zero.**
 - i. Place the sample tube with the stopper into the support. (*Always wear gloves when handling the sample tube*) and record the weight.
- b. **In a separate container, or on a piece of weighing paper, weigh out the approximate amount of sample desired.**
 - i. Remove the stopper and pour the sample into the tube using the aluminum funnel.
 - ii. Re-stopper the tube and transport to the FlowPrep 060 unit.
- c. **Make sure the Nitrogen source for the FlowPrep 060 is turned on.**
 - i. There are two rows of holes, one for heating, and one for cooling. Place the tube and sample into one of the heating holes.
 - ii. Remove the stopper and place a de-gassing probe into the tube and turn on the switch to that particular de-gassing probe.
 - iii. Adjust the temperature to the desired level and wait the appropriate amount of time.
 - iv. Remove the tube from the heating port and place in the cooling port of the 060,

- v. Allow to come to room temperature while still de-gassing with Nitrogen then re-stopper.
- d. Weigh the sample, stopper and tube and record the weight.**
 - i. Find the weight of the de-gassed sample by subtracting from the weight of the tube and stopper.

4. Creating and Running the Method

- a. All components of the instrument must be on to have the keypad be operational.**
- b. The first step is to attach the sample tubes to the instrument.**
 - i. Inside the sample compartment there are two ports, the sample goes to the right; the reference tube (empty) goes on the left.
 - ii. Unscrew the collar rings and retrieve the “o” rings.
 - iii. Place the collar and the “o” rings onto the sample tube and reference tube.
 - iv. Carefully insert the tubes into the open port a screw the collar snug. (*do not over tighten*)
- c. Open the valve to the helium tank, turn on the vacuum pump and turn on the Gemini unit.**
 - i. Allow the components to warm up and purge for 20 minutes.
- d. Press the white key then 4 on the keypad to begin the process.**
 - i. Enter the “set-up” group number (the number under which your method will be stored. Try and not use one currently being used)
 - ii. Put in a number for your sample identification and press enter.
 - iii. Type in your sample weight and press enter.
 - iv. Put in the rest of the information when prompted according to the method you are using
- e. On the last step, when asked to start the experiment, DO NOT press enter at this time. You must first fill the Dewar with liquid Nitrogen.**
 - i. Liquid Nitrogen is dangerous and can cause severe frost burns.
 - ii. Either be trained on how to get the liquid Nitrogen or have someone else get it for you.
 - iii. Wear safety glasses and gloves when getting the liquid Nitrogen.
- f. When the Dewar of liquid Nitrogen is on the pedestal, you may press enter to begin the experiment.**

5. Shutdown

- a. Close the valves to the Nitrogen and Helium tanks.**
- b. Turn off the vacuum pump.**
- c. Turn off the FlowPrep and the Gemini unit.**