

Gemini 2360 Surface Area Analyzer

Updated November 14, 2017

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 Morin

(x3756 / cmorin@bowdoin.edu / Druckenmiller 243)

- 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.

- a. **Sample vials need to be cleaned under the following procedure:**
 - i. Sonicate the sample tubes in a solution of 10g laboratory detergent per liter of warm water for 15 minutes. Make sure sample tubes are entirely covered with water.
 - ii. With rubber gloves on, clean the interior of the samples tubes with brush if necessary. Rinse the sample tubes in hot tap water, then thoroughly in D.I. water, then with isopropyl alcohol. Dry the inside of the sample tubes with nitrogen, then place them upside down on a tube rack and dry in an oven for a minimum of 2 hours @ 75C.
 - iii. Remove from the oven, again with gloves, and allow cooling. Fill with dry Nitrogen then stopper with a rubber stopper that has been wiped with a lint free cloth. (**Note:** Quickly stopper to keep the Nitrogen from escaping)

- 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 as the empty sample tube weight.
- b. **Place a sample weighing container, or a piece of weighing paper, onto the balance and zero. Weigh out the approximate amount of sample desired $\sim 4\text{m}^2$ total surface area, or tube $>1/2$ full. Record the weight of the sample as the sample weight prior to degassing.**
 - 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.

- a. Make sure the Nitrogen source for the FlowPrep 060 is turned on ~6psi which is 20cm³/min flow. (Wear gloves during this process.)**
- 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 gently place a degassing probe into the tube and place the rubber stopper lightly on the top of the tube (should NOT be tight). Turn on the switch to that particular degassing probe.
 - iii. Adjust the temperature to the desired level and wait the appropriate amount of time. 105C for ~4 hrs, or ~60C overnight. (It takes ~2hrs. to degas at 200C)
 - iv. Remove the sample tube from the heating port and place in the cooling port of the 060, keeping the gas delivery tube and stopper in the sample tube to prevent contamination from the atmosphere.
 - v. Allow to come to room temperature while still de-gassing with Nitrogen. Pull the gas delivery tubes out, then immediately re-stopper.
 - vi. Wipe the gas delivery tube with a lint-free cloth and place back into the cooling station.
 - vii. Turn off the flow control valve.
 - viii. Weigh the stoppered sample tube w/sample and record the weight. Find the weight of the degassed sample by subtracting from the weight of the tube and stopper to determine if adequate degassing has occurred. Record the weight as the sample plus sample tube/stopper weight.
- b. While sample is degassing, open the valve to the nitrogen tank (15-18 psi -- min. tank pressure no less than 200psi), turn on the vacuum pump and turn on the Gemini unit.**
- i. Allow the components to warm up for at least 40 minutes before performing analysis.
- c. Fill the Dewar with liquid nitrogen ~ 1 cm from the top.**
- 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.
- d. Measure Reference Pressure**
- i. Perform each day before running samples.
 - ii. Place empty tubes in both the reference (left) and sample (right) tube holders on the Gemini analyzer. Make sure they are tight. <10 in first 3 min. and <5 in next 3 min. To do this, take the metal nut off as well as the rubber gasket, and place them over the end of the tube. Place the tube flush against the sample holder and screw on the nut and gasket until finger tight. A tube should not wobble or be able to be pulled from the holder if it is inserted properly.

