

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)

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. Start-up Procedure

- 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)

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 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.

4. Degassing the Sample and Gemini 2360 Prep

- 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.

- iii. On the keypad, push the white key and then [Po] and “enter” to measure reference pressure.
- iv. After the Gemini has taken Po, top off the dewar with LN₂.

5. 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. (*Wear gloves during this procedure.*) Do these steps as quickly as possible to minimize sample exposure to air.**
 - i. Inside the sample compartment there are two ports, the sample goes to the right; the reference tube (empty) goes on the left. The reference, or balance tube should be degassed before the first time it is used.
 - ii. Place the connector nut, ferrule, and O-ring onto the sample and reference tube stem.
 - iii. Carefully insert the tubes into the open port, making sure they are fully seated, and screw the connector nut onto the port. Hand tighten only. (*Do not over tighten!*)
 - iv. Close the door to the sample compartment.
 - v. **Note**—the reference tube does not need to be replaced between analyses unless it has been contaminated or you are using a different size sample tube.
- c. **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) (NONE)
 - 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
- d. **On the last prompt, press “ENTER” to begin the analysis. You can cancel the analysis while it is running by pressing the white key then “CLEAR.”**
- e. If the analysis is lengthy, more than one hour, you will need to re-fill the Dewar with liquid nitrogen.
- f. When sample analysis is finished, place the sample back in the heat block of the FlowPrep with outgassing needle in case the sample needs to be re-run.
- g. Repeat steps b through f for remaining samples.

6. Shutdown

- a. **Close the valve to the Nitrogen tank.**
- b. **Turn off the vacuum pump.**
- c. **Turn off the FlowPrep and the Gemini unit.**
- d. **Discard excess liquid nitrogen by evaporating.**
- e. **Empty and clean sample tubes.**