

Establishing Sample Degassing Conditions for the FlowSorb

Sample preparation is crucial in obtaining reliable, repeatable results with the FlowSorb just as it is with all physical adsorption instruments. Users typically establish the temperature-time parameter that gives consistent results by heating samples of the material to be analyzed to various temperatures for different times and then measuring surface areas, or they merely choose temperatures and times that are thought to be more than sufficient. However, one can define what conditions are really required by only a few tests with the FlowSorb itself.

Certain precautions need to be noted before applying the procedure to be described. Do not use this procedure with samples that are contaminated with such things as light hydrocarbons, waxes, or other materials that can decompose and foul the FlowSorb's detector. These materials must be degassed in the usual manner because then they are downstream of the detector and their effluent poses no hazard to the detector. Also certain materials, notably zeolites, must be treated by a several-step procedure because they are known to undergo a structural change if allowed to reflux in their contained water vapor. The procedure with this type of material must be started at no more than 90 °C and increased in increments of approximately 50 °C.

These instructions require the heating mantle to be at temperature when the actual test begins because that is most likely to be the case when the FlowSorb is used for routine work. The result is thus a minimum time for sample degassing. The time for degassing will increase by the 10 to 20 minutes it takes for the mantle to come to temperature if mantle heating is started as the test begins.

1. Plug a heating mantle into its power socket and set its temperature to be that of the first test, say 200 °C.
2. Establish gas flow through the instrument with a clean, empty sample tube at the TEST port. Note that it is necessary to have the cold trap tube installed to obtain gas flow.
3. Fill a sample tube with a typical quantity of sample and set it and its holder aside for the moment.
4. Clear the display meter and then depress the DET. push button. If necessary, adjust the display to zero with the FINE or COARSE ZERO knobs. Be sure the PATH select is on SHORT.
5. As soon as the heating mantle reaches its set temperature: exchange the empty sample tube holder on the TEST port with the sample tube holder containing sample, place the heating mantle around the sample tube, and record the time of day.

6. The display will register for the first four to five minutes the air peak introduced with the sample tube. After that it is only the moisture coming from the sample that is registered. When the display (after minutes or maybe hours) returns to a nearly steady reading of 0.05 to 0.07, record again the time of day. A reading of a little more than zero is acceptable because the system itself typically requires an extra 15 to 20 minutes to return all the way to zero after exposure to water vapor.
7. The difference in the two times of day is the heating interval required at the set temperature, in this example 200 °C.

The numbers displayed for water vapor effluent rarely rise to more than 2 or 3 because the detector is not as sensitive to water vapor as it is to nitrogen and helium. These numbers can rise rapidly and decay rather abruptly if the moisture is only on the sample surface, or they can rise and decay more slowly if much of the moisture is held in small pores. In any event, a test establishes a possible set of temperature-time conditions for degassing. If the time of the first test is undesirably long and the sample can tolerate a higher temperature without degradation, then a trial with a new sample and a higher temperature is in order. If it cannot, then the longer time at the lower temperature establishes the required conditions.