Colorado State University

CHEM 477 Physical Chemistry Laboratory - II

Notes for Determination of Surface Area of a Solid by the Dynamic Flow Method

The following is a set of short notes to outline the experiment in question and to provide helpful guidance to those executing the experiment.

- A. The determination of the surface area of a solid is an important on for modern surfaceactive systems. The determination is most often performed by the physisorption (as contrasted with the chemisorption) of a non-reactive gas (nitrogen in this case) onto the surface at low temperature and desorption of those absorbed molecules at ambient temperature. Measurement of the amount of gas absorbed by a known mass of solid is used to compute the surface are of the solid (and in certain cases more details of the surface morphology).
- **B.** Setup and demonstrate the proper operation of the surface area analyzer. Read the instrument brochure and select sections (chapters 2, 3 and 4) of the manual. Because the instrument has been severely modified by an earlier student the operation of the detector and analysis reported in the manual are no longer relevant and must be ignored. The recording of data will be performed by a more modern USB-based analog-digital acquisition system, data logging program and subsequently analyzed using Igor Pro.
- C. Perform a "one-point" surface area measurement (absorption, desorption and calibration) on a sample of amorphous silica gel using a nitrogen mole fraction of 0.3. Record the local atmospheric pressure as reported by the on-campus weather station at http://ccc.atmos.colostate.edu/. Transfer the raw instrumental data to Igor for visualization and analysis.
- **D.** Degas the same sample at 200° C for 15 minutes, permit it to return to room temperature and repeat the surface area measurement. Analyze.
- E. Perform several additional surface area measurements at varying nitrogen concentration. Perform a multi-point BET determination and analysis. Collect data at a total of four nitrogen concentrations.
- **F.** Report on the instrumentation and the underlying mechanism of its operation. Compare the determined surface area values with the literature.

Surface Area of Solids