Protocol
Accelerated Surface Area and Porosimetry analyzer (ASAP)

1. Method

The ASAP 2010 (Accelerated Surface Area and Porosimetry System) provides versatility in gas selection and high vacuum for high-resolution low surface area measurements. It uses the principle of physical adsorption to obtain adsorption and desorption isotherms and information about the surface area and porosity of a solid material. It performs surface area analyses plus pore size and pore volume distributions, using nitrogen as the standard gas. The observations are interpreted following the model of Brunauer, Emmett and Teller (BET Method), or Langmuir, to calculate the surface areas, the average and total pore volume, the BJH pore size distribution and performs micro-pore analysis.

2. Equipment

- Instrument: Micromeritics ASAP 2010 (http://www.micromeritics.com);
- Analytical balance (precision 0.1 [mg]);
- Spatula for powder samples;
- Sample tube suitable for the instrument;
- Glass pipe, introduced inside the test tube, to decrease the dead volume;
- Specific lid for the test tube.

3. Protocol

The measurement of a complete adsorption/desorption isotherm for one sample must be planned over 2 days.

Tube degassing: 1 hour

- Put carefully a glass pipe in an empty test tube. Put the lid on the top of the tube;
- Install these onto the instrument (choose left or right position);
- Install the heating bag around the bottom of the tube;
- To start the degassing: press “Load” on the keyboard, then “Left or Right” depending on the tube position, and “Begin”;
- Choose the temperature of heating, depending of the nature of the sample which will be measured. For most ceramics, 200 °C is the standard temperature. For an unknown material the temperature must be determined by a preliminary thermal analysis (thermogravimetric analysis TGA);
- Start the heating by pressing “Enable heating”;
- Choose the vacuum pressure depending on the nature of the sample which will be measured. For volatile powders (apparent densities <0.5), choose 200 or 300 mmHg. For heavy powders, choose 500 or 600 mmHg.

Stop the degassing

- After one hour of degassing, check the vacuum: press “Check” on the keyboard, then “Left or Right” depending on the tube position, and “Begin”. If there is no variation of the pressure in the “Vacuum” window, the system is ready to work.

- To stop the heating: press “Begin”, then “Enable heating”. Remove the heating bag from the tube;
- To stop the degassing: press “Unload” on the keyboard, then “Left or Right” depending on the tube position, and “Begin”;
- Let the tube cool during 10 minutes.

Introduction of the sample

- Remove the sample tube from the apparatus;
- Weigh the empty sample tube together with its rack, the lid and the glass pipe inside with the analytical balance (accuracy 0.1 [mg]). Carefully write down the result W_T [g];
- Remove carefully the lid, and the glass pipe;
- With the spatula add a sufficient amount of powder in the test tube (the total surface available should range from 10 to 20 [m^2]);
- Replace carefully the glass pipe inside the test tube, and close it with the lid;
- Install these onto the instrument, at the same position as before for the degassing (left or right);
- Install the heating bag around the bottom of the tube.

Sample degassing: 16 hours

- Press “Load” on the keyboard, then “Left or Right” depending on the tube position, and “Begin”;
- Start the heating by pressing “Enable heating”.

After one hour, check that “Ready” is lit. If not, that means the vacuum cannot be achieved, or the sample cannot be easily degassed.

If “Error” is lit, stop the degassing, replace the tube correctly, and start the procedure again.

Stop the degassing

- After 16 hours (minimum) of degassing, check the vacuum: press “Check” on the keyboard, then “Left or Right” depending on the tube position, and “Begin”. If there is no variation of the pressure in the “Vacuum” window, the system is ready for analysis;
- To stop the heating: press “Begin”, then “Enable heating”. Remove the heating bag from the tube;
- To stop the degassing: press “Unload” on the keyboard, then “Left or Right” depending on the tube position, and “Begin”;
- Let the tube cool during 10 minutes.

Start the measurement

- Remove the sample tube from the apparatus;
- Place the sample tube with its lid on its rack and weigh (accuracy 0.1 [mg]). Carefully write down the result W_C [g];
- The mass of powder to insert to the program is calculated from:

$$W_P=W_C-W_T[g]$$

- Place the white protection around the sample tube. Place these on the measurement system;
- Carefully fill in the two Dewars with liquid nitrogen; work with gloves and safety glasses. Control the level of nitrogen;
- Place them on the platforms. Place the protection sponges on the top.

- Open the program
- Go to "File/Open/***Sample information***"
- Choose the directory in the list, in the right side of the window
- Put a new name in "File name", then OK, and Yes
- Add some information for Sample, Operator, Submitter
- Introduce W_P in "Sample weight"
- Go to ***Analysis conditions***
- Click on "Replace", and in the list, choose "Powders.anc Porous powders"
- Go to ***Adsorptive properties***, and control that Nitrogen is selected
- Go to ***Report options***
- Click on "Replace", and in the list, choose "ltpstand1.rpo pore size analys-1"
- Click on OK, Save, and Close

- Go to "Analyse"¨
- Select "Report after analyse"
- Choose Screen in the Destination window
- Select the name of your sample in "filename", and OK; the measurement starts, and lasts around 12 hours.

Remark: as soon as a sample is being measured, a new test tube could be put for degassing.

- Once the measurement is finished, remove the powder from the sample tube, wash the tube with water using the ultrasonic bath, rinse with ethanol and dry at 60 [°C] in the oven.

4. Presentation of the results, data storage and data treatment

Export the results

- Go to "Report/Start report"
- Choose "File" in the Destination window
- Choose the directory, and your file. Click on OK and save as Powder-Lotn°-ASAP-Experimentn°-Operator.rpt
- Open this file. Go to File/Printer setup, and choose PdfCreator. Save it as Powder-Lotn°-ASAP-Experimentn°-Operator.pdf.

Data storage

- Copy the report, and the PDF report.
- Go to \\Ltppc40\powderfiles. Copy the folder *Powderfiles*. Paste it in your project folder, and change its name into Powder-Lotn°

- Paste the RPT and PDF files respectively in the folders Project/Powder-Lotn°/ASAP/Data and PDF.

Data treatment

- Go to \\Ltppc40\powderfiles. In the folder Project/Powder-Lotn°, open the Excel sheet "Powdersheet.xls"
- Click on the *ASAP* button, and follow the instructions given in the Excel sheet