



PROCEDURE TO TEST NIST SRM-2206 AND SRM-2207 WITH HORIBA SA-9600

NIST certified standards are commonly used to verify accuracy and proper operation of instruments. The NIST Standard Reference Materials SRM-2206 and 2207, high-purity granulated controlled-pore glass with a nominal pore diameter of 300 nm and 18 nm respectively, are intended for the calibration and performance testing of instruments used for the determination of the Brunauer-Emmett-Teller (BET) specific surface area (SSA). When these standards are used on HORIBA SA-9600, it is important to use the method developed for the HORIBA SA-9600 to obtain meaningful results.

Analytical test method

Applicable instruments: HORIBA SA-9600 series, including SA-9601, SA-9601MP, SA-9603, SA-9603MP, etc.

Procedure:

1. Prepare sample
 - a. Record the weight of an empty cell (tare weight).
 - b. Mix sample in original sample bottle by shaking and scoop recommended amount of sample into cell as indicated by the table below.
 - c. Record the weight of the cell with sample in it. The difference between the cell with sample in it and the empty cell is the pre-degas sample weight.
 - d. Put the cell in the cell holder and connect to a prep station.
 - e. Fit the heating mantle around the cell and connect the heating mantle wires to the prep station.
 - f. Degas the sample following the degas conditions in the table below.
 - g. After degas is complete, remove the heating mantle and let the cell cool down at room temperature for 15 minutes.

Standard	Degas Temperature and Time	Recommended Weight
SRM 2206	290 °C For 120 minutes	~ 0.3 g
SRM 2207	290 °C For 120 minutes	~ 0.1 g

2. Take single-point measurements (for all instrument models)
 - a. Set up test conditions for single-point measurement, i.e. single relative pressure, typically 30% N₂.
 - b. Carefully fill dewar approximately 2/3 full of liquid nitrogen.
 - c. Place dewar on tray under measurement station.
 - d. Lock in degassed sample cell and holder into measurement station.



Analytical Test Method

Specific Surface Area Analyzer

HORIBA SA-9600

ATM117

NIST SRM 2206 and 2207

- e. On control panel of instrument, press "V" for to see bridge voltage. Allow several minutes for purge voltage reading to pass by and wait for voltages to stabilize.
 - f. Take measurement.
 - g. After measurement is complete, reweigh sample to obtain post-degas sample weights.
 - h. Input post-degas sample weight to recalculate the specific surface area.
3. Take multi-point measurements (for model SA-9601MP and SA-9603MP)
- a. Set up test condition for multi-point measurement, either 3 points or 6 points of relative pressure.
 - b. Follow same steps b. through h. for single-point measurement.

Test Results

Verify that the results are within the following specification:

Standard	Nominal* m ² /gram	Spec Range m ² /gram
SRM 2206	10.86 +/- 0.81	+/- 10% (9.04-12.84)
SRM 2207	174.72 +/- 3.1	+/- 10% (155.61-197.01)

* Nominal value is the average of certified values for single-point measurements and multi-point measurements provided by the certificate of analysis from NIST.

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