INTERLABORATORY STUDY MEASURING Water Vapor SORPTION ISOTHERMS ON BAM-P109

STUDY PROTOCOL

# INTRODUCTION

This study is organized under the auspices of the Versailles Project on Advanced Materials and Standards ([VAMAS](http://www.vamas.org/)) Technical Working Group № 39 ([TWG 39)](http://www.vamas.org/twa39/) and is coördinated by the Facility for Adsorbent Characterization and Testing ([FACT Lab](https://www.nist.gov/mml/fact/)) at the National Institute of Standards and Technology ([NIST](https://www.nist.gov/)), Gaithersburg, Maryland, USA.

The objectives of this interlaboratory study are to compare measured water/BAM-P109 sorption isotherms at 25 °C as a function of P/P0 or percent relative humidity (% RH) and to extract a reference isotherm from these measure­ments.

Study Participants are asked to execute the [Study Agreement.](https://www.nist.gov/document/study-agreement)

# MATERIALS

A certified reference material, BAM-P109 (nanoporous carbon[[1]](#footnote-1)), will be used in this study. A bottle of the material (≈3 g) will be provided to each Study Participant upon enrollment in this exercise. Material is limited, so additional materials cannot be provided.

Study Participants shall be responsible for providing all other materials required for the study. Gases shall have a purity of 99.999 % as determined by the supplier. Water shall be LC/MS grade.

# SAMPLE HANDLING, STORAGE & SAFETY

The sample should be handled as prescribed in the BAM-P109 certificate, which will be shipped along with the sample. The sample should be stored at room temperature, preferably in a desiccator, and should be sealed when not in use. Any spilled material should be handled in accordance with established safe-operating procedures.

# SUPPLIED DOCUMENTATION

The certificate for BAM-P109 will be provided. The [Safety Data Sheet](https://www.nist.gov/document/safety-data-sheet) for BAM-P109 will be shipped with the material.

# INSTRUCTIONS FOR PERFORMING THE MEASUREMENTS

# Sample Size

The sample mass should be optimized to achieve the best signal-to-noise ratio for the instrument being used and measured using an analytical balance.

# Sample Activation

Before starting the adsorption measurements, the sample must be activated. The sample is to outgassed using the BAM prescribed procedure, i.e., outgas the sample in vacuum at room temperature, then heat over a period of 1 h to 200 C and hold at that temperature for 3 h under vacuum with continuous pumping (final pressure < 0.1 Pa). If the outgassing is performed in a separate manifold, minimize exposure to air when transferring to the analysis port and re-outgas in the instrument to 140 C for at least 6 h to remove any adsorbed gases.

# Measurement of Adsorption Isotherms

**Study Measurements (Water & BAM-P109).** Water (H2O) adsorption and desorption isotherms are to be measured on BAM-P109 at 25 C as a function of P/P0 or percent RH. Isotherms should be measured for two separate sample aliquots, activated individually, resulting in a total of two isotherms. See below for recommended P/P0 and relative humidity values.

It is recommended that data be recorded at the following interval: 0.05, 0.1, 0.15, 0.2, 0.25, 0.3, 0.35, 0.4, 0.45, 0.5, 0.55, 0.6, 0.65, 0.7, 0.75, 0.8, 0.85, 0.9, 0.95 P/P0, or 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90, and 95 % RH.

For pure vapor sorption measurements (i.e., static, no carrier gas is used), uptake should be reported as a function of P/P0. For dynamic vapor sorption measurements, nitrogen (99.999 % purity) should be used as the carrier gas, and data should be reported as a function of percent RH.

**Blank Run.** A blank run is required. A blank run is a measurement of an “isotherm” without an adsorbent. The units of this blank will be amount-of-substance, i.e., millimoles. The values of the blank run are subtracted from the uptake of the isotherms measured for the absorbent, also in units of millimoles. The values of the isotherm are then divided by the mass of the aliquot, yielding an isotherm with units of amount-of-substance over mass, i.e., mmol/g. The same set of relative humidity values used in the adsorption measurements should be used in the blank run measurements (see above).

# DATASETS

A total of three isotherms are to be measured:

* + Two isotherms for water ad-/desorption on BAM-P109
  + A blank run using no adsorbent

# REPORTING: DEADLINES & TEMPLATES

# Water/BAM-P109

The Experimental Report and Data Submission Spreadsheet for the study measurements should be submitted no later than February 14, 2022.

The fully processed isotherms (i.e. after all processing, corrections, blank subtraction etc.) should be reported using the supplied spreadsheets (more details on the spreadsheets below). The data should be reported in units of millimoles of adsorbed water vapor per gram of activated adsorbent (mmol/g) as a function of P/P0 (for static measurements) or percent relative humidity (for dynamic measurements).

Using the Excel® spreadsheet either named [Study\_Results\_RH.xlsx](https://www.nist.gov/document/study-results-rh) or [Study\_Results\_PP0.xlsx](https://www.nist.gov/document/study-results-pp0) insert the adsorption isotherm for Aliquot № 1, adsorption into columns A and B and for desorption in columns D and E. For Aliquot № 2, enter adsorption into columns G and H and desorption in columns J and K. These should be the fully processed isotherms (i.e. after all processing, including blank subtraction). Insert the blank run in columns M and N. Data will appear in the associated graphs.

Details about the experimental parameters should be reported using the Word® document named [Study\_Report.doc](https://www.nist.gov/document/study-report). Please complete relevant sections.

These documents should be submitted to [FACTLab@nist.gov,](mailto:FACTLab@nist.gov?subject=NIST/VAMAS%20ILS%20Documents:%20Water/BAM-P109%20Results) with the subject line “NIST/VAMAS ILS Documents: Water/BAM-P109 Results,” or simply click the e-mail link above.

# OUTPUTS

The output of this exercise is intended to be one or more articles published in the peer-reviewed literature, describing the study, lessons learned from this exercise, and the reference vapor adsorption isotherm for water on BAM-P109 derived from this exercise. The reference isotherm will be determined using statistical metrics. All published isotherms will also be made available on-line through the NIST/ARPA-E Database of Novel and Emerging Adsorbent Materials.[[2]](#footnote-2) Results of participating labs will be anonymized, though Study Participants will have their data identified to them.

# QUESTIONS or COMMENTS?

Please direct any questions or comments regarding this study to [FACTLab@nist.gov,](mailto:FACTLab@nist.gov?subject=NIST/VAMAS%20ILS:%20Question) with the subject line “NIST/VAMAS ILS: Question,” or simply click the e-mail link above.

1. Sample generously provided by the German Federal Institute for Materials Research and Testing (BAM). [↑](#footnote-ref-1)
2. adsorption.nist.gov [↑](#footnote-ref-2)