CERTIFICATE OF ANALYSIS

ERM®-FD107

Faujasite type zeolite

Certified Values		
Quantity	Certified value [1]	Uncertainty [2]
Specific micropore volume in $cm^3\ g^{-1}$	0.217	± 0.002
Median pore width in nm	0.86	± 0.02

[1] Unweighted mean value of the means of 11 and 13 accepted sets of data, each set obtained in a different laboratory.

[2] Estimated expanded uncertainty *U* with a coverage factor *k*=2, corresponding to a level of confidence of about 95 %, as defined in the Guide to the expression of uncertainty in measurement, ISO, 1993.

The sample mass to be analyzed should be about 0.2 g, depending on the instrument used (see instructions of instrument producer).

NOTE

European Reference Material ERM®-FD107 was originally certified as BAM-P107. It was produced and certified under the responsibility of Bundesanstalt für Materialforschung und -prüfung (BAM) according to the principles laid down in the technical guidelines of the European Reference Materials® co-operation agreement between BAM-LGC-IRMM. Information on these guidelines is available on the Internet (http://www.erm-crm.org).

Accepted as an ERM®: Berlin, 2004-04-14

This certificate is valid for three years after purchase.

Sales date:

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Additional Material Information		
Quantity	Informative value [3]	Uncertainty [4]
Langmuir specific surface in $m^2\ g^{-1}$	610.6	± 13.8
Density in $g \cdot cm^{-3}$	2.34	–

[3] Unweighted mean value of the means of accepted sets of data respectively, each set being obtained in a different laboratory.

[4] Estimated expanded uncertainty *U* with a coverage factor *k*=2, corresponding to a level of confidence of about 95 %, as defined in the Guide to the expression of uncertainty in measurement, ISO, 1993.

[5] The density (apparent density of the material including closed and inaccessible pores) was estimated by helium pycnometry at room-temperature (296 K = 23 °C) after drying at 393 K (120 °C) for 24 h.

DESCRIPTION OF THE SAMPLE

The reference material consists of pellets (binder: 1 – 2 % clay content). Additional information on the preparation, the certified and indicative values are given in the certification report.

ANALYTICAL METHOD USED FOR CERTIFICATION

Micropore analysis by gas adsorption according to DIN 66134-4.

Sorptive: dry nitrogen, not less than 99.99 % purity

Coolant: liquid nitrogen, not less than 99 % purity

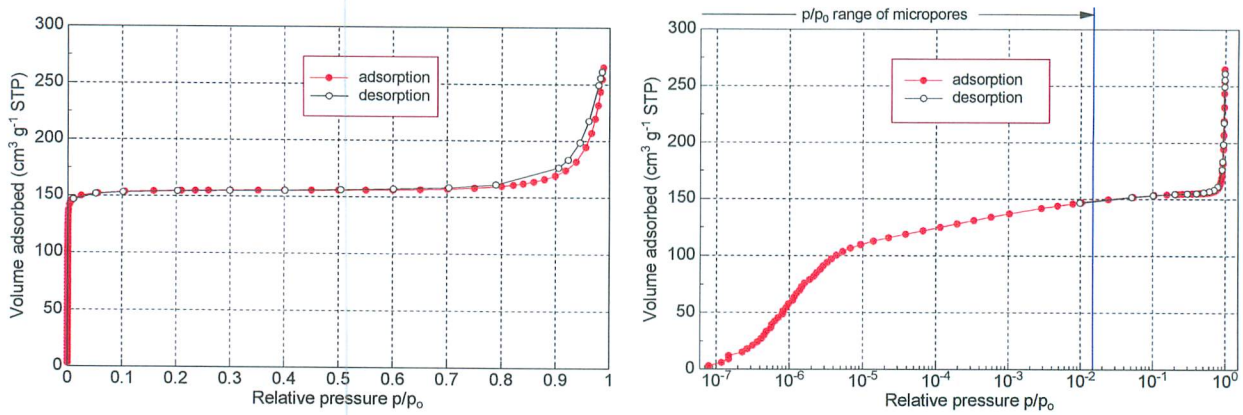

Figure 1: Linear and logarithmic plot of the nitrogen adsorption isotherm (non-certified) at 77.3 K and p/p_0-range for micropores (width ≤ 2 nm) according to the physical parameters applied in the Saito/Foley model with cylindrical pore geometry given on page 3.

DATA EVALUATION

- Median pore width:

  Determined as a d_{50} diameter from the cumulative pore voume curve up the pore diameter of 2 nm (corresponding to the relative pressure $p/p_0 = 0.015$) calculated by the Saito/Foley model (cylindrical pore geometry, see DIN 66135 part 4) with the physical parameters according to the Table below.

- Specific micropore volume:

  Cumulative pore volume at the pore diameter of 2 nm, corresponding to the isotherm data point nearest to $p/p_0 = 0.0150$, but within the range $p/p_0 = (0.0150 \pm 0.0076)$.

- Langmuir specific surface area (non-certified):

  Calculated from the slope of a Langmuir plot ($p/p_0 \leq 0.015$).

Table: Physical parameters for the Saito/Foley cylindrical pore model

Adsorbent	**Zeolite**
width d	0.304 nm
polarizability α	8.5×10^{-25} cm^3
magnetic susceptibility χ	1.94×10^{-29} cm^3
density N	3.75×10^{15} molecules/cm^2

Adsorptive	**Nitrogen**
temperature T	77.15 K
diameter d	0.300 nm
polarizability α	1.76×10^{-24} cm^3
magnetic susceptibility χ	3.6×10^{-29} cm^3
density N	6.71×10^{14} molecules/cm^2

range of micropores	$p/p_0 \leq 0.015$
molecular cross-sectional area for nitrogen	$\sigma_{N_2} = 0.162$ nm^2
volume conversion factor for nitrogen	$V_{N_2,\,\text{gas},\,\text{STP}} \times 0.0015468 = V_{N_2,\,\text{liquid}}$

PARTICIPANTS

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- VTT Energy, Espoo, FI

INSTRUCTIONS FOR USE

Prior to using ERM®-FD107, an outgassing procedure is necessary.

Starting at room temperature, the zeolites are to be heated up to about 350 K (≈ 80 °C) under vacuum. When a residual pressure of 10^{-2} Pa or lower has been achieved at 350 K (≈ 80 °C), carefully increase the temperature up to 390 K (≈ 120 °C) with a rate of about one Kelvin per minute. In this area the main portion of water is expelled. When a residual pressure of 10^{-2} Pa or lower has been achieved, heat in vacuum up to 620 K (≈ 350 °C) in steps of 50 K per half an hour. After a residual pressure of 10^{-2} Pa or lower has been reached at 620 K (≈ 350 °C), continue evacuating at 620 K (≈ 350 °C) for at least 5 hours.

To avoid contamining the analysis manifold with water vapour, it is preferable to outgas on a separate manifold. However, if outgassing has been carried out separately, a re-outgassing of the sample on the analysis port may be required (620 K) after the sample has been transferred across.

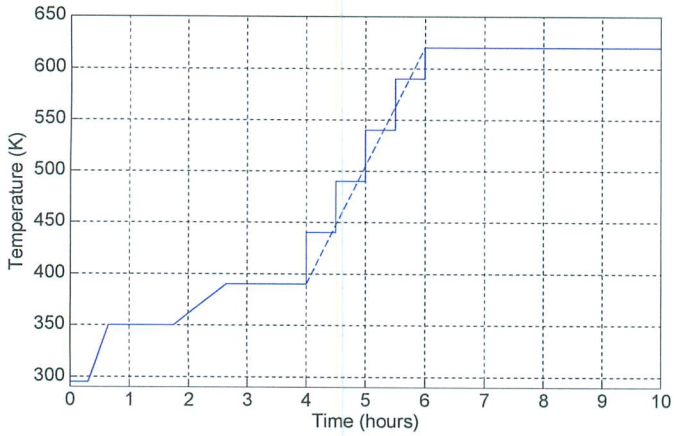

Figure 2: Recommended heating curve for the microporous material ERM®-FD107

Attention:

It is recommended to determine the dead space / free space system volume by means of helium after completing the adsorption measurement.

STORAGE

The material should be stored at normal laboratory temperature (20 - 25 °C).

TECHNICAL REPORT

A detailed technical report (in German) describing the analysis procedures and the treatment of the analytical data used to certify ERM®-FD107 is available on request.

REFERENCES

Guidelines for the production and certification of BAM reference materials

BCR/01/97 Guidelines for the production and certification of BCR reference materials

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Pure & Appl. Chem. **66** (1994) 8, 1739 - 1758

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