



National Institute of Standards & Technology

Report of Investigation

Reference Material 8851

Zeolite A

This Reference Material (RM) is intended to provide a source of Zeolite A for measurement comparisons. The zeolite was donated to the National Institute of Standards and Technology (NIST) by a zeolite-producing company. A unit consists of approximately 35 g to 40 g of material in a sealed container.

Reference and information values derived from chemical, diffraction and particle size measurements are provided in Tables 1 through 14.

Reference Values: Reference values are a best estimate of the true value provided by NIST where all known or suspected sources of bias have not been fully investigated by NIST [1]. Reference values for major components (major elements, loss on ignition [LOI], loss on fusion [LOF]) are provided in Table 1, Si/Al and Na/Al ratios in Table 2, and trace elements in Table 3.

Information Values: Information values are noncertified values that may be of interest and use to the RM user, but insufficient information is available to assess an uncertainty associated with the value [1]. An information value for the Si/Al ratio determined from nuclear magnetic resonance (NMR) is provided in Table 4. Information values for enthalpy of formation of constituent oxides and liquid water, and elements are provided in Table 5. Information values for unit cell parameters for hydrated and dehydrated Zeolite A are provided in Tables 6 and 7; information values for the fractional coordinates, site occupancy, isotropic thermal parameters and site multiplicity for atoms used for Rietveld refinement of dehydrated zeolite are provided in Table 8. Information values for particle size distributions are provided in Table 9 and Tables 11 through 14; and for refractive index determination in Table 10. In addition, spectra for three types of NMR analyses are provided in Figures 1 through 3; plots for particle size distribution are provided in Figures 4 through 7; electron microscope images are provided in Figure 8 and information concerning the variation in sample mass with change in ambient humidity is provided in the Appendix.

Expiration of Value Assignment: RM 8851 is valid indefinitely, within the measurement uncertainty specified, provided the RM is handled and stored in accordance with instructions given in this Report of Investigation (see "Instructions for Use"). Periodic validation of this RM is not required. This report is nullified if the RM is damaged, contaminated, or otherwise modified.

Maintenance of Reference Values: NIST will monitor this RM over the period of its validity. If substantive technical changes occur that affect the value assignment before the expiration of this report, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Overall direction and coordination of the analyses were performed at NIST by S. Turner and R.R. Cavanagh.

Analytical measurements were performed at NIST by D.A. Becker, R.A. Fletcher, J.F. Kelly, A.F. Marlow, M.G. Moreno-Ramirez, J.R. Sieber, E.B. Steel, B.H. Toby, S. Turner, J.R. Verkouteren, T.W. Vetter, E.S. Windsor, and R. Zeisler.

NMR studies were performed by M.E. Davis at the California Institute of Technology and by G.J. Kennedy and W.S. Borghard at ExxonMobil Research & Engineering (Annandale, NJ). Heat of enthalpy studies were performed by S. Yang and A. Navrotsky at the University of California (Davis, CA).

Statistical consultation for the experimental design and analysis of data was provided at NIST by S.D. Leigh, W.F. Guthrie, and K.R. Eberhardt.

Carlos A. González, Chief
Chemical Sciences Division

Gaithersburg, Maryland 20899
Report Issue Date: 11 December 2020
Report Revision History on Page 12

Steven J. Choquette, Director
Office of Reference Materials

The zeolite was prepared and packaged by NIST and by Laboratory Quality Services International (LQSI)⁽¹⁾.

Support aspects involved in the issuance of this RM were coordinated through the NIST Office of Reference Materials.

INSTRUCTIONS FOR USE

Chemical and Diffraction Analyses: For comparison to chemical and diffraction values, samples should be equilibrated in a constant relative humidity (RH) of 54 % ± 2 % for at least 48 h. A hydrator (e.g., glove box) containing a saturated solution of Mg(NO₃)₂·6H₂O with excess salt can attain a constant RH of approximately 54 % when the temperature is 20 °C. ASTM Standard Practice E 104-02 gives more details on maintaining constant RH by means of aqueous solutions [2]. Samples should be in relatively thin layers (< 0.5 cm) in the hydrator.

If samples are to be weighed for chemical analyses, the samples should be equilibrated to approximately 54 % RH and the samples then weighed in the same ambient humidity. This can be accomplished by weighing samples in a glove box containing a scale. For preparation of samples for diffraction analysis, samples should be equilibrated to approximately 54 % RH in a glove box and then sealed within the glove box. The RH of the glove box should be monitored with a calibrated hygrometer. Samples should not be exposed to ambient laboratory humidity prior to weighing for chemical analyses or during the collection of diffraction data. An example of the variation of sample mass with exposure to a different humidity is given in the Appendix. Refer to section on “Chemical Homogeneity Testing by XRF” for minimum recommended mass for chemical analysis.

Particle Sizing: For particle sizing experiments, special care must be exercised to obtain subsamples that are representative of the particle size distribution of the entire bottle [3–5]. Spin riffling (whole stream rotary sampling) was used to obtain subsamples for characterization of the particle size distribution of this RM. Other methods for subsampling the zeolite material have not been investigated in this study and may result in altered particle size distributions or different uncertainties. In general, the zeolite is highly agglomerated. Therefore, once a representative subsample is obtained, it is recommended that the sample be deagglomerated prior to particle size analysis. Deagglomeration can be achieved by ultrasonification of a suspension of the sample in filtered water. Tests should be run using increasing ultrasonification times to determine the time required to reach a stable particle size distribution. An ice bath may be necessary to reduce the temperature of the suspension during ultrasonification.

The container should be resealed tightly after sampling.

PREPARATION

Sample Preparation: The zeolite material came to NIST in five paper bags. Samples were scooped out of the original containers and placed into aluminized bags using an alternate shovel method. By this method, a scoop was taken from a container and placed in a bag; the next scoop taken from the container was placed in another bag, etc. The procedure was repeated until the original containers were empty. This procedure was done to ensure that each aluminized bag contained samples from all parts of the original containers. Each aluminized bag contained less than 9 kg (20 lbs) of material. Sample spin riffling and bottling was contracted out to LQSI, which is a division of Commercial Testing & Engineering Co. (CT&E).

ANALYSES

Methods Used: Chemical analyses were performed by X-ray fluorescence spectrometry (XRF), gravimetry, instrumental neutron activation analysis (INAA) of short, medium and long-lived isotopes, magic angle spinning (MAS) NMR, and calorimetric methods [6].

Unit cell parameters were determined for hydrated samples using synchrotron X-ray diffraction and for dehydrated samples using neutron diffraction [6].

Particle size analyses were performed using laser light scattering, laser light extinction, electric sensing zone, and sedimentation methods [6]. In order to obtain representative samples for the analyses, a series of mass reduction steps were done to randomly chosen units of material using a spin riffling apparatus. Bottles of zeolite were split into 16 subsamples using a spinning riffler. One of the 16 subsamples was then randomly selected and further split into 8 subsamples using a smaller spinning riffler. An overall riffle split mass reduction of 128 yielded samples of

⁽¹⁾ Certain commercial equipment, instruments, materials, and entities are identified in this report to adequately specify the analytical procedures. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment, instruments, materials, and entities identified are the best available for the purpose.

approximately 0.2 g, which is the subsample size used for particle analysis. Tests were performed to determine the amount of sonication necessary to ensure deagglomeration of particles and samples were ultrasonified prior to particle size measurements. Images of the particles were obtained by scanning transmission electron microscopy (STEM). Electron microscopy samples were not deagglomerated prior to imaging.

Chemical Homogeneity Testing by XRF: Chemical homogeneity was tested for the zeolite for Na, Al, Si, K, S, Ca, Fe, Zn, and Zr using XRF analysis. No statistically significant differences among bottles were observed for Na, Al, Si, K, S, Ca, Fe, and Zr. A statistical F-test (5 % significance) applied to the data for Zn indicates heterogeneity between bottles at the level of precision of the test data, 0.3 % relative standard deviation for the estimated 0.5 g of material analyzed by the spectrometer. Zinc is present at the mg/kg mass fraction level. For the major elements, Na, Al and Si, specimens as small as 10 mg will be representative of the bulk material. For the determination of trace elements, the heterogeneity of Zn must be taken into account and it is recommended that at least 0.5 g of material be analyzed to obtain results representative of the bulk material.

REFERENCE VALUES: CHEMISTRY

Major Components:

Table 1. Reference Values for Major Components of Zeolite A

Component	Mass Fraction (%) ^{(e)(f)}	Methods
Sodium (Na) ^(a)	12.732 ± 0.066	XRF, INAA
Aluminum(Al) ^(a)	14.766 ± 0.076	XRF, INAA
Silicon (Si) ^(a)	15.27 ± 0.10	XRF, gravimetry
Si ^(b)	19.541 ± 0.045	gravimetry
LOI ^{(a)(c)}	21.464 ± 0.085	gravimetry
LOF ^{(a)(d)}	22.1 ± 1.7	XRF

^(a) Value relative to the hydrated sample mass.

^(b) Value relative to sample mass ignited at 1000 °C.

^(c) Ignited at 1000 °C.

^(d) Fused with Li borates at 1100 °C.

^(e) The reference values for Na, Al, and Si (relative to hydrated mass) are an unweighted mean of the results from two analytical methods. The associated uncertainties are expanded uncertainties about the mean, with coverage factor $k = 2$ (95 % confidence), calculated by combining a between-method variance [7] with a pooled, within-method variance following the ISO/JCGM and NIST Guides [8]. Where appropriate, components of uncertainty attributable to fit calibration, balance calibration, blank replication, contamination, mechanical loss, and hydration have been included.

^(f) The reference values for Si (relative to the ignited sample mass), LOI and LOF are the mean of results obtained by NIST using one analytical method. The expanded uncertainties, U , for Si (relative to ignited sample mass) and LOI are calculated as $U = ku_c$ where u_c is intended to represent, at the level of one standard deviation, the combined standard uncertainty calculated according to the ISO/JCGM and NIST Guides [8]. The coverage factor, k , is determined from the Student's t -distribution with the appropriate associated degrees of freedom for 95 % confidence for each analyte. The expanded uncertainty for LOF is calculated as $U = ku_c$ where u_c represents the standard uncertainty of the mean for the NIST values, and $k = 2.12$ is the Student's t multiplier corresponding to 95 % confidence for 16 determinations.

Element Ratios:

Table 2. Reference Values for Atomic Ratio of Si (Relative to Hydrated Sample Mass) to Al and Na to Al^(a)

Elements	Ratio	Methods
Si/Al	0.9937 ± 0.0082	XRF, INAA, gravimetry
Na/Al	1.0120 ± 0.0074	XRF, INAA

^(a) The reference atomic ratio values for Si/Al and Na/Al are calculated using the reference values for the elements from Table 1 and the atomic mass of the elements. The corresponding uncertainties are determined by propagation-of-error [9] for ratios from the unexpanded uncertainties of Table 1, and then expanded by a coverage factor $k = 2$ (95 % confidence).

Trace Elements:

Table 3. Reference Values for Detectable Trace Elements

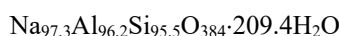
Element ^(a)	Mass Fraction (mg/kg) ^(c)		
Iron (Fe)	90.8	±	5.2
Cerium (Ce)	1.069	±	0.039
Cobalt (Co) ^(b)	0.064 6	±	0.002 8
Chromium (Cr)	0.513	±	0.033
Cesium (Cs)	0.005 60	±	0.000 52
Europium (Eu)	0.024 87	±	0.000 78
Hafnium (Hf)	0.803	±	0.025
Lanthanum (La)	0.534	±	0.026
Scandium (Sc)	0.059 1	±	0.001 9
Samarium (Sm)	0.102 8	±	0.006 1
Thorium (Th)	0.141 3	±	0.004 6
Zinc (Zn)	18.73±		0.98

^(a) Trace element analyses by INAA were conducted for Ag, As, Cd, Ce, Co, Cr, Cs, Eu, Fe, Hf, La, Mo, Sb, Sc, Se, Sm, Sn, Th, W, and Zn. Several elements were at or below the detection limit achieved for this material with the procedure used. These elements and their detection limits are: Ag (10 µm/kg), As (0.3 mg/kg), Cd (4 mg/kg), Mo (1.3 mg/kg), Sb (10 µg/kg), Se (10 µg/kg), Sn (1.5 mg/kg), and W (2 µg/kg).

^(b) Results from one of sixteen analyses for Co were excluded (sample had a nugget containing high Co and measurable Ag [31 µg/kg ± 1 µg/kg]).

^(c) The reference value for Fe (relative to hydrated mass) is an unweighted mean of the results from two analytical methods (XRF and INAA). The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor $k = 2$ (95 % confidence), calculated by combining a between-method variance [7] with a pooled, within-method variance following the ISO/JCGM and NIST Guides [8]. The reference values for trace elements other than Fe are the mean of results obtained by NIST using one analytical method (INAA applied to 16 samples). Expanded uncertainties, U , are calculated as $U = ku_c$ where u_c is intended to represent, at the level of one standard deviation, the combined standard uncertainty calculated according to the ISO/JCGM and NIST Guides [8]. The coverage factor, k , is determined from the Student's t -distribution with the appropriate associated degrees of freedom for 95 % confidence for each analyte.

Comment 1: The compositional formula of RM 8851 determined from chemical analyses is:



The formula is of the bulk RM 8851 and therefore includes a minor contribution from the impurity identified by diffraction work—sodalite ($\text{Na}_8\text{Al}_6\text{Si}_6\text{O}_{24}\text{Cl}_2$) (see “Diffraction” section). The sodalite content elevates the apparent Na content and depresses the water content relative to the main Zeolite A component of RM 8851. The water is derived from the LOI mass fraction in Table 1 with the assumption of no other components. The uncertainties for the elemental and water content in the formula are: Na ± 0.5, Al ± 0.5, Si ± 0.6, H₂O ± 0.8. The uncertainties are propagated from the uncertainties in mass fractions given in Table 1. Trace elements have not been included in the formula.

INFORMATION VALUES: CHEMISTRY

NMR Analyses for Si/Al and Si coordination:

Table 4. Si/Al Atomic Ratio Determined from ²⁹Si MAS NMR Analyses

Lab	Ratio	Number of samples
1	1	1
2	1	3

The ²⁹Si MAS NMR spectrum for Zeolite A (Figure 1) shows a single peak at −89.5 ppm corresponding to Si(OAl)₄.

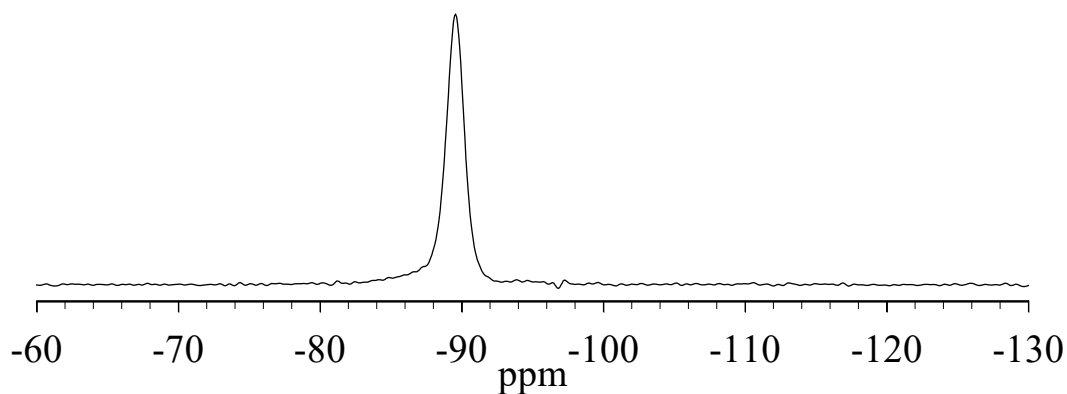


Figure 1. ^{29}Si MAS NMR spectrum obtained from Zeolite A.

NMR Analyses for Al Coordination: The ^{27}Al MAS NMR spectra show a single Lorentzian peak at 58.4 ppm (lab 1, Figure 2) or 60.4 ppm (lab 2) corresponding to tetrahedral, i.e., framework, aluminum. There is no evidence for nonframework, octahedrally coordinated aluminum in this zeolite.

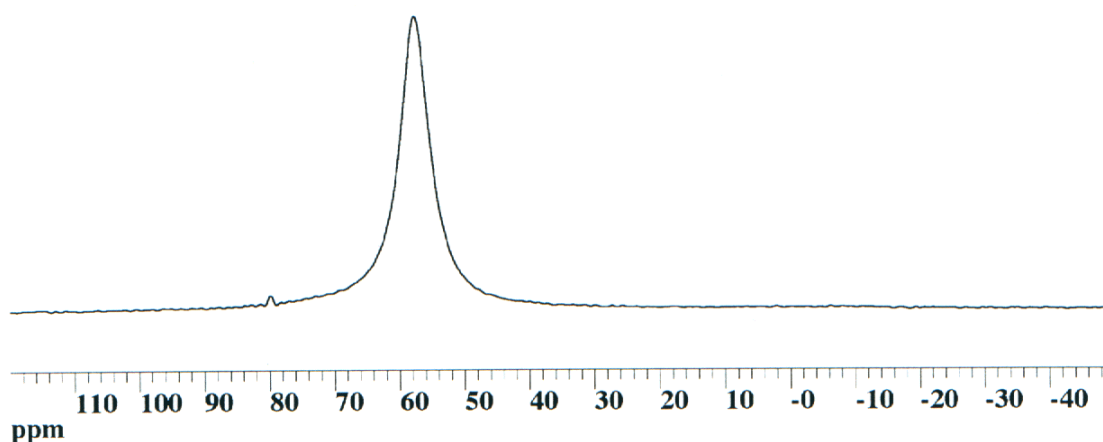


Figure 2. ^{27}Al MAS NMR spectrum obtained from Zeolite A.

NMR Analyses for H in Proximity to Si: An example ^{29}Si cross polarization (CP) MAS NMR spectrum is shown in Figure 3. The CP experiment enhances the signals from those silicon nuclei that are in close proximity to hydrogen, such as covalently bound silanols or trapped hydrocarbons. The signal to noise ratio of a CP-MAS NMR spectrum when compared to that of the ^{29}Si MAS NMR experiment is a qualitative indicator of the presence of these species.

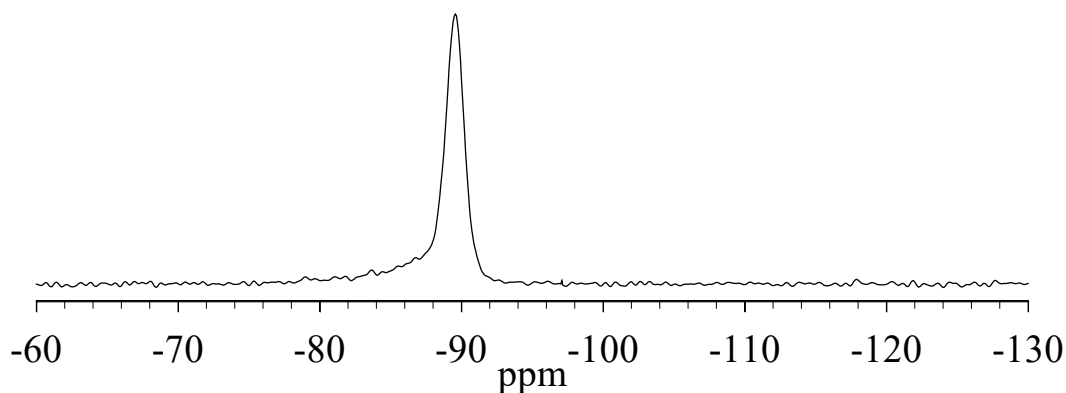


Figure 3. ^{29}Si CP-MAS NMR spectrum obtained from Zeolite A.

Enthalpy of Formation:

Table 5. Enthalpy of Formation from Constituent Oxides and Liquid Water, and Constituent Elements at 25 °C

Constituents	Enthalpy of Formation (kJ/mol) ^(a)
Oxides and liquid water	-74.24 ± 0.65
Elements	-1365.4 ± 0.9

^(a) The uncertainties are propagated from calorimetric data and from literature values for the constituent oxides or elements (coverage factor $k = 2$, 95 % confidence).

INFORMATION VALUES: DIFFRACTION

Synchrotron X-ray Diffraction of Hydrated Samples:

Table 6. Unit Cell Parameter of Hydrated Zeolite A (Space Group *Fd-3c*)

Axis	Value (nm) ^(a)
a	2.4589 ± 0.0002

^(a) The uncertainty is one standard deviation based on 13 samples.

Note: Peaks from an impurity phase were noted, where the strongest impurity peak was $\approx 1\%$ of the strongest Zeolite A peak. The impurity phase was assigned as sodalite.

Neutron Diffraction of Dehydrated Samples:

Table 7. Unit Cell Parameter of Dehydrated Zeolite A (Space Group *Fd-3c*)

Axis	Value (nm) ^(a)
a	2.4560 ± 0.0006

^(a) The uncertainty is one standard deviation based on 11 samples.

Note: Peaks from a sodalite impurity phase could not be detected. Pattern simulation shows that peaks due to a 1 % to 2 % sodalite impurity (by mass) would be difficult to detect, setting an upper limit for the impurity level.

Table 8. Parameters used in Rietveld Refinement of Dehydrated Zeolite A^(a)

Atom	$x/a^{(b)}$	$y/a^{(b)}$	$z/a^{(b)}$	Fractional Occupancy ^(b)	$U_{iso} \times 100^{(b)}$	Site Multiplicity
Si	0	0.094 10 (27)	0.183 91 (26)	1	1.46 (5)	96
Al	0	0.187 51 (34)	0.089 29 (34)	1	1.46 (5)	96
O1	0	0.113 34 (8)	0.247 91 (28)	1	2.46 (3)	96
O2	0	0.143 94 (22)	0.146 35 (23)	1	2.46 (3)	96
O3	0.054 60 (13)	0.057 85 (12)	0.172 48 (6)	1	2.46 (3)	192
Na1	0.099 79 (9)	0.099 79 (9)	0.099 79 (9)	0.928 (11)	2.11 (16)	64
Na2	0	0.232 6 (5)	0.203 1 (6)	0.248 (5)	2.11 (16)	96

^(a) Column headings correspond to atoms and their fractional coordinates, site occupancy, isotropic thermal parameters (U_{iso}) and site multiplicity.

^(b) Values in parentheses represent standard uncertainties in the associated value, where the uncertainty is applied to the final digit(s) of the value. Standard uncertainty is a crystallographic statistic derived from experimental counting statistics propagated via the least-squares covariance matrix [10].

INFORMATION VALUES: PARTICLE SIZING

The distributions reported for the particle size measurement methods reported below should be considered valid only for the specific method used. Variations in values among methods may result from effects of particle characteristics such as morphology.

Laser Light Scattering:

Table 9. Cumulative Volume Percent Less Than Particle Diameter of Sixteen Samples^(a)

Cumulative Volume (%)	Mean Diameter (μm)	Standard Deviation of the Mean (μm)
5	1.104	0.002
10	1.285	0.003
15	1.431	0.003
20	1.564	0.003
25	1.691	0.004
30	1.815	0.004
35	1.940	0.005
40	2.068	0.005
45	2.199	0.006
50	2.339	0.006
55	2.488	0.007
60	2.649	0.008
65	2.832	0.008
70	3.037	0.010
75	3.276	0.012
80	3.569	0.014
85	3.959	0.016
90	4.541	0.021
95	5.754	0.043

^(a) Measured by the laser light scattering method.

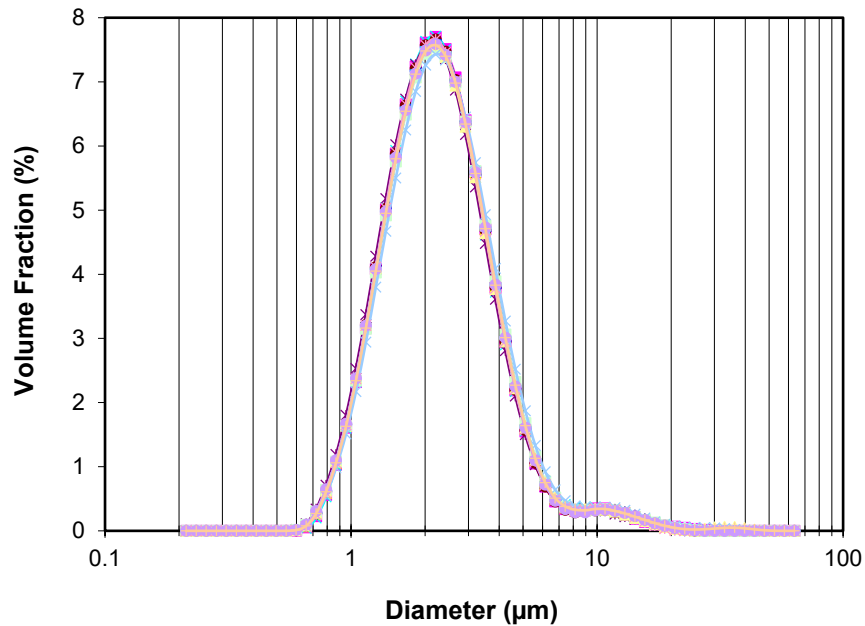


Figure 4. Distribution of differential volume fraction (%) vs. particle diameter as measured by the laser light scattering method. Each curve represents the analysis for a different bottle of the eight bottles analyzed.

Refractive Index Determination for Mie Optical Model:

Table 10. Refractive Index Determination

Refractive Index^(a)
1.446

^(a) A value of 1.45 was used in Mie modeling of the particle size distribution for the laser light scattering method since values to 2 decimal places are considered sufficient for this purpose.

Electric Sensing Zone:

Table 11. Cumulative Volume Percent Less Than Particle Diameter of Six Samples^(a)

Cumulative Volume (%)	Mean Diameter (μm)	Standard Deviation of the Mean (μm)
5	1.71	0.01
10	1.93	0.01
15	2.08	0.01
20	2.21	0.01
25	2.32	0.01
30	2.42	0.01
35	2.52	0.01
40	2.61	0.02
45	2.71	0.02
50	2.81	0.02
55	2.91	0.02
60	3.02	0.02
65	3.14	0.02
70	3.28	0.03
75	3.44	0.03
80	3.67	0.03
85	4.02	0.04
90	4.85	0.09
95	7.45	0.22

^(a) Measured by the electric sensing zone method.

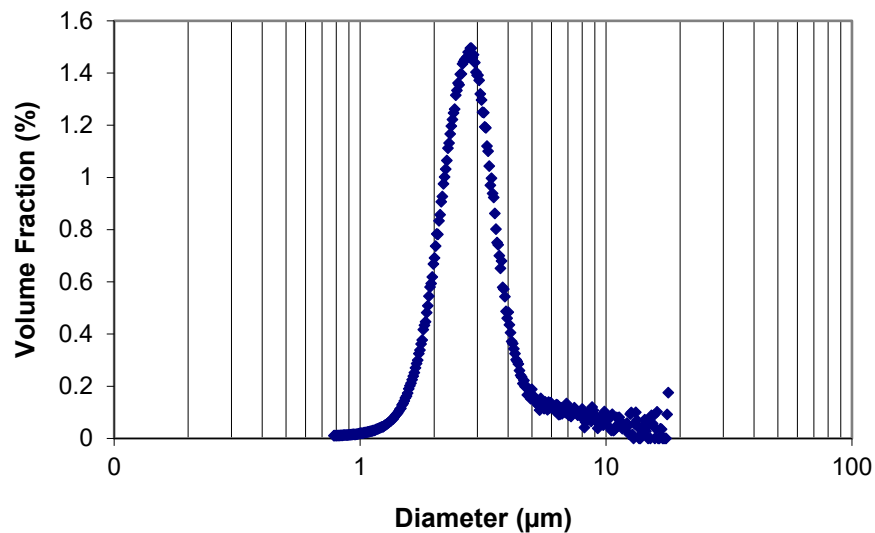


Figure 5. Distribution of differential volume fraction (%) vs. particle diameter as measured by the electric sensing zone method (distribution is the average of six runs).

Laser Light Extinction:

Table 12. Cumulative Number of Particles Greater Than Nominal Particle Diameter per μg Zeolite^(a)

Diameter (μm)	Cumulative Number	Standard Deviation	Cumulative Number (%)
1.0	62 468	552.9	100.
1.5	35 564	430.0	56.93
2.0	15 542	232.1	24.88
2.5	9 554	145.5	15.29
3.0	6 837.5	111.8	10.95
3.5	4 190.1	70.7	6.71
4.0	2 135.4	52.7	3.42
4.5	1 032.9	31.6	1.65
5.0	583.9	24.6	0.94
5.5	403.4	16.9	0.65
6.0	306.5	14.5	0.49
6.5	233.5	12.1	0.37
7.0	172.5	10.1	0.28
7.5	118.8	8.0	0.19
8.0	82.1	7.6	0.13
8.5	57.7	5.9	0.09
9.0	40.5	5.4	0.06
9.5	29.4	4.8	0.05
10.0	22.0	4.9	0.04
10.5	16.4	3.9	0.03
11.0	12.4	3.2	0.02
11.5	9.3	2.9	0.02

^(a) Measured by the laser light extinction method.

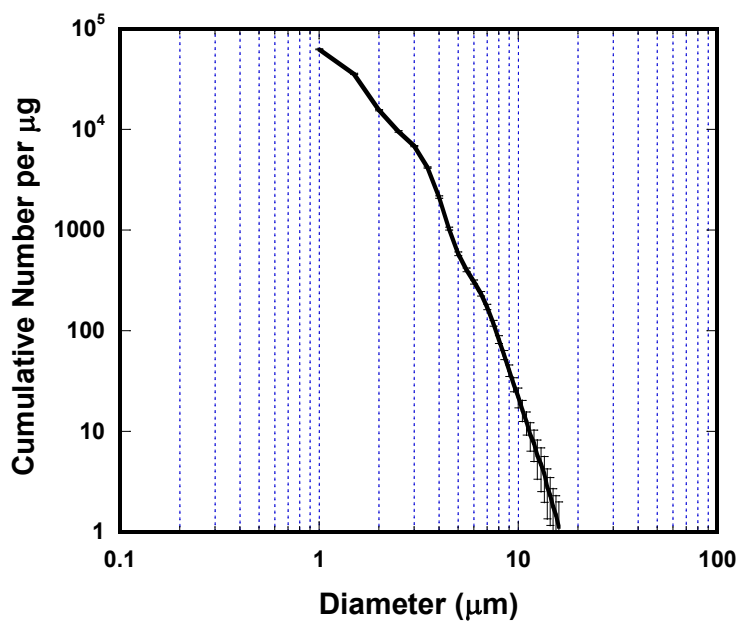


Figure 6. Distribution of the cumulative number of particles per μg greater than particle diameter as measured by the laser light extinction method. The uncertainty bars correspond to the standard deviation of the measured values.

Table 13. Cumulative Volume Percent Less than Particle Diameter^(a)

Cumulative Volume (%)	Diameter (μm)	Uncertainty (μm) ^(b)
5	1.53	0.04
10	1.75	0.04
15	2.03	0.04
20	2.30	0.05
25	2.69	0.06
30	3.03	0.06
35	3.30	0.06
40	3.42	0.07
45	3.64	0.07
50	3.86	0.07
55	4.08	0.07
60	4.25	0.07
65	4.47	0.08
70	4.75	0.07
75	5.30	0.08
80	6.36	0.10
85	7.36	0.08
90	8.97	0.06
95	10.85	0.04

^(a) Measured by the laser light extinction method.

^(b) Uncertainties are the expanded uncertainties with coverage factor $k = 2$ (95 % confidence).

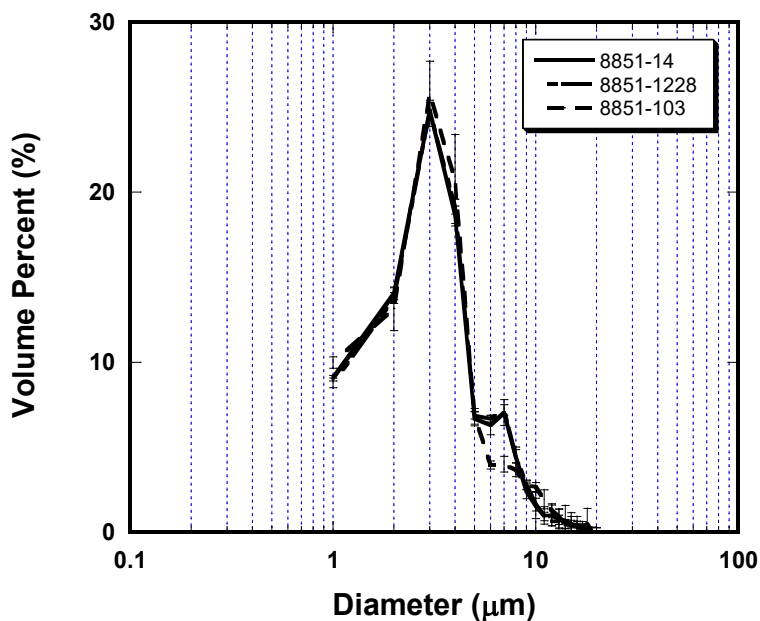


Figure 7. Distribution of differential volume fraction (%) vs. particle diameter as measured by the laser light extinction method. Each of the three curves (one each for the three bottles analyzed) represents the mean value ($n = 5$ to 10) of the volume distribution. The uncertainty bars correspond to the standard deviations for each of the three sets of measurements.

X-ray Sedimentation:

Table 14. Information Values Obtained by X-ray Sedimentation^(a)

Cumulative Volume (%)	X-ray Sedimentation Mean Diameter (μm)	Laser Light Scattering Mean Diameter (μm) ^(b)	Electric Sensing Zone Mean Diameter (μm) ^(b)	Laser Light Extinction Mean Diameter (μm) ^(c)
10	1.48	1.285 ± 0.003	1.93 ± 0.01	1.75 ± 0.04
25	1.87	1.691 ± 0.004	2.32 ± 0.01	2.69 ± 0.06
50	2.37	2.239 ± 0.006	2.81 ± 0.02	3.86 ± 0.07
75	3.10	3.276 ± 0.012	3.44 ± 0.03	5.30 ± 0.08
90	4.45	4.451 ± 0.021	4.85 ± 0.09	8.97 ± 0.06

^(a) Values from other methods for comparison.

^(b) Uncertainty values represent the standard deviation of the mean.

^(c) Uncertainty values represent expanded uncertainties with coverage factor $k = 2$ (95 % confidence).

STEM:

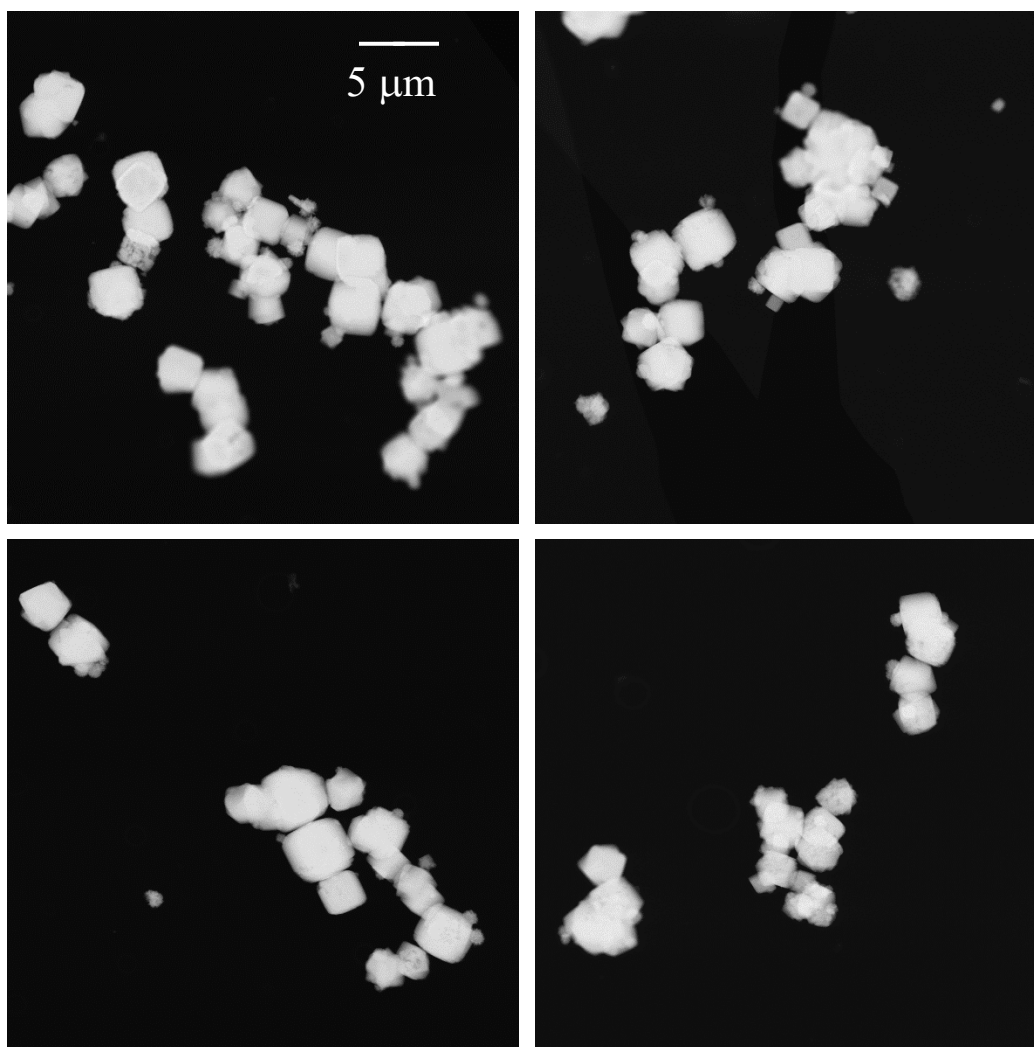


Figure 8. Dark field STEM images of Zeolite A. All images were taken at the same magnification.

REFERENCES

- [1] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at <https://www.nist.gov/system/files/documents/srm/SP260-136.PDF> (accessed Dec 2020).
- [2] ASTM E104-02; *Standard Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions*; Annu. Book ASTM Stand., Vol. 11.03 (2012).
- [3] Kaye, B.H.; *Investigation into the Possibilities of Developing a Rate Method of Sieve Analysis*; Powder Metall., Vol 5, pp. 213–234 (1962).
- [4] Allen, T.; Khan, A.A.; *Critical Evaluation of Powder Sampling Procedures*; Chem. Engr., Vol. 238, pp. CE108–CE112 (1970).
- [5] British Standard 3406-1; *Methods for Determination of Particle Size Distribution - Part 1: Guide to Powder Sampling*; British Standards Institution, London UK (1986).
- [6] Turner, S.; Sieber, J.R.; Vetter, T.W.; Zeisler, R.; Marlow, A.F.; Moreno-Rameriz, M.G.; Davis, M.E.; Kennedy, G.J.; Borghard, W.G.; Yang, S.; Navrotsky, A.; Toby, B.H.; Kelly, J.F.; Fletcher, R.A.; Windsor, E.S.; Verkouteren, J.R.; Leigh, S.D.; *Characterization of Chemical Properties, Unit Cell Parameters and Particle Size Distribution of Three Zeolite Reference Materials: RM 8850 – Zeolite Y, RM 8851 – Zeolite A and RM 8852 – Ammonium ZSM-5 Zeolite*; Micropor. Mesopor. Mater., Vol. 107, pp. 252–267 (2008).
- [7] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results From Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105, pp. 571–579 (2000); available at <https://www.ncbi.nlm.nih.gov/pmc/articles/PMC4877152/> (accessed Dec 2020).
- [8] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement (GUM 1995 with Minor Corrections)*; Joint Committee for Guides in Metrology (JCGM) (2008); available at https://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Dec 2020); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at <https://www.nist.gov/pml/nist-technical-note-1297> (accessed Dec 2020).
- [9] Ku, H.H.; *Notes on the Use of Propagation of Error Formulas*; J. Res. Natl. Bur. Stand., Vol. 70C, pp. 263–273 (1966); available at https://nvlpubs.nist.gov/nistpubs/jres/70C/jresv70Cn4p263_A1b.pdf (accessed Dec 2020).
- [10] Schwarzenbach, D.; Abrahams, S.C.; Flack, H.D.; Prince, E.; Wilson, A.J.C.; *Statistical descriptions in crystallography. II. Report of a Working Group on Expression of Uncertainty in Measurement*; Acta Crystallogr. A51, pp. 565–569 (1995).

Report Revision History: 11 December 2020 (Correction to revision history; editorial changes); 28 September 2020 (Change of expiration date; clarification for LOF Method in Table 1; editorial changes); 08 September 2006 (Editorial changes); 26 July 2006 (Original Report of Investigation).

Users of this RM should ensure that the Report of Investigation in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; e-mail srminfo@nist.gov; or via the Internet at <https://www.nist.gov/srm>.

APPENDIX A

Variation in Sample Mass with Change in Ambient Humidity

Experiments were conducted to determine the magnitude of variation in sample mass with change in ambient humidity. One of the experiments is described and its results given as information values so that users of the RM will be aware of the magnitude of mass change with exposure to two different relative humidities ($33\% \pm 2\% \text{ RH}$ and $54\% \pm 2\% \text{ RH}$).

Approximately 0.8 g of zeolite was placed in an aluminum weighing dish. The sample was placed in a hydrator (in this case, a glass desiccator containing a saturated solution of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ with excess salt) and allowed to equilibrate at $33\% \pm 2\% \text{ RH}$ over several days. The hydrator with the sample was placed in a glove box containing a weighing scale and several pans of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ salt in solution. After the glove box reached $33\% \pm 2\% \text{ RH}$, the sample was removed from the hydrator and the sample mass was determined. The sample was placed back in the hydrator. The pans of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ salt solutions in the glove box were exchanged for several pans containing $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ salt solutions. After the glove box was stable at $54\% \pm 2\% \text{ RH}$, the sample was removed from the hydrator, placed on the scale in the glove box and its mass monitored at one minute intervals for approximately the first half hour and periodically over the next several days.

The change in mass is shown in Figure A1. The results show a mass difference of approximately 0.4 % between the $33\% \pm 2\% \text{ RH}$ and $54\% \pm 2\% \text{ RH}$ provided by the two salt solutions. The greatest change in mass occurred in the initial few minutes of exposure to $54\% \pm 2\% \text{ RH}$. The mass gain was stable at approximately 0.4 % over a period of several days.

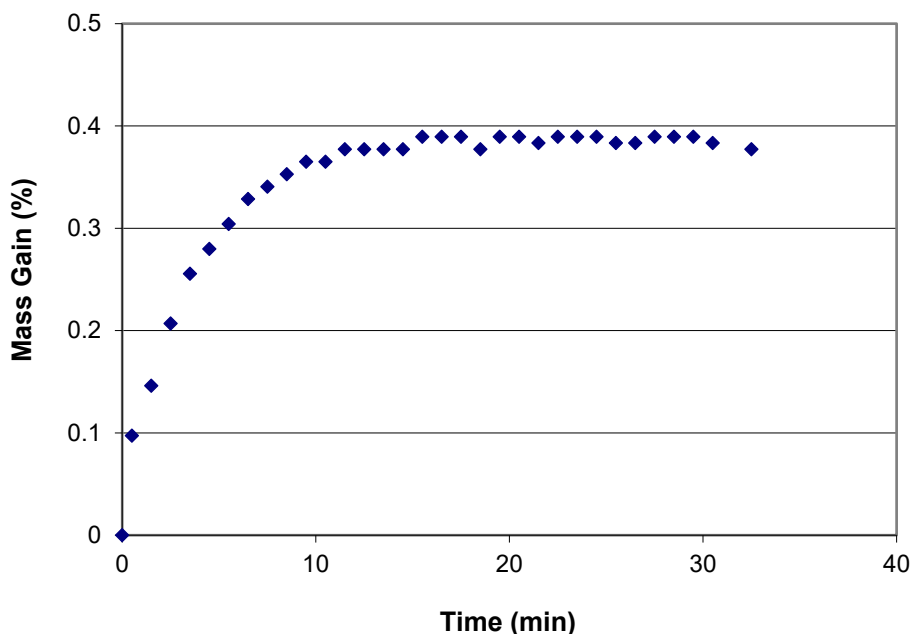


Figure A1. Variation in mass of Zeolite A as the ambient humidity was changed from $33\% \pm 2\% \text{ RH}$ (time = 0) to $54\% \pm 2\% \text{ RH}$ (time > 0 min).