



National Institute of Standards & Technology

Report of Investigation

Reference Material 8850

Zeolite Y

This Reference Material (RM) is intended to provide a common source of Zeolite Y 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 15.

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 homogeneously distributed trace elements in Table 3a.

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]. Information values for inhomogeneously distributed trace elements are given in Table 3b. An information value for the Si/Al ratio determined from nuclear magnetic resonance (NMR) is provided in Table 4 and example peak assignments for a ^{29}Si magic angle spinning (MAS) NMR spectrum are provided in Table 5. Information values for enthalpy of formation of constituent oxides and liquid water and elements are provided in Table 6. Information values for unit cell parameters for hydrated and dehydrated Zeolite Y are provided in Tables 7 and 8. Information values for the fractional coordinates, site occupancy, isotropic thermal parameters and site multiplicity for atoms used for Rietveld refinement of dehydrated Zeolite Y are provided in Table 9. Information values for particle size distributions are provided in Tables 10 and 12 through 15; and for refractive index determination in Table 11. 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 Figures 8 and 9 and information concerning the variation in sample mass with change in ambient humidity is provided in the Appendix.

Expiration of Value Assignment: RM 8850 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 of NIST.

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).

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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.

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 % \pm 2 % for at least 48 h. A hydrator (e.g., glove box) containing a saturated solution of $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 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.

PREPARATION

Sample Preparation: The zeolite material came to NIST in a single metal barrel with a metal coating. Samples were scooped out of the original container and placed into aluminized bags using an alternate shovel method. By this method, a scoop was taken from the 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 container was empty. This procedure was done to ensure that each bag contained samples from all parts of the original container. Each 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, 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. 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

⁽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.

8 subsamples using a smaller spinning riffler. An overall riffle split mass reduction of 128 yielded samples of 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) and scanning electron microscopy (SEM). 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, Ca, Fe, Zn, and Zr using XRF analysis. No statistically significant differences among bottles were observed for Na, Al, Si, Ca, Fe, Zn, and Zr. A statistical F-test (5 % significance) applied to the data for K indicates heterogeneity between bottles at the level of precision of the test data, 0.8 % relative standard deviation for the estimated 0.03 g of zeolite material analyzed by the spectrometer. Potassium 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 K must be taken into account and it is recommended that at least 0.05 g of material be analyzed to obtain results representative of the bulk material.

REFERENCE AND INFORMATION VALUES: CHEMISTRY

Major components:

Table 1. Reference Values for Major Components of Zeolite Y

Component	Mass Fraction (%) ^{(e)(f)}	Methods
Sodium (Na) ^(a)	7.225 ± 0.094	XRF, INAA
Aluminum (Al) ^(a)	8.49 ± 0.11	XRF, INAA
Silicon (Si) ^(a)	22.52 ± 0.16	XRF, gravimetry
Si ^(b)	30.336 ± 0.054	gravimetry
LOI ^{(a)(c)}	25.679 ± 0.095	gravimetry
LOF ^{(a)(d)}	25.37 ± 0.67	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 and 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 $k = 2.145$ is the Student's t multiplier corresponding to 95 % confidence for 14 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	2.547 ± 0.037	XRF, INAA, gravimetry
Na/Al	0.997 ± 0.018	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 3a. Reference Values for Homogeneously Distributed Trace Elements

Element ^(a)	Mass Fraction (mg/kg) ^(b)
Iron (Fe)	174.3 ± 2.0
Cerium (Ce)	2.836 ± 0.066
Europium (Eu)	0.0345 ± 0.0009
Hafnium (Hf)	0.816 ± 0.019
Lanthanum (La)	1.666 ± 0.048
Rubidium (Rb)	0.335 ± 0.042
Scandium (Sc)	0.1350 ± 0.0037
Samarium (Sm)	0.1861 ± 0.0068
Thorium (Th)	0.3601 ± 0.0086
Zinc (Zn)	19.45 ± 0.99

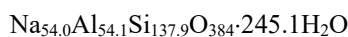
Table 3b. Information Values for Inhomogeneously Distributed Trace Elements

Element ^(a)	Mass Fraction (mg/kg) ^(b)
Cobalt (Co)	0.1308 ± 0.004
Chromium (Cr)	1.212 ± 0.053
Cesium (Cs)	0.0298 ± 0.0031

^(a) Trace element analyses by INAA were conducted for Ag, As, Cd, Ce, Co, Cr, Cs, Eu, Fe, Hf, La, Mo, Rb, 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 (20 µm/kg), As (0.3 mg/kg), Cd (4 mg/kg), Mo (1.3 mg/kg), Sb (10 µg/kg), Se (20 µg/kg), Sn (2 mg/kg), and W (2 µg/kg).

^(b) 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). The 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 8850 determined from chemical analyses is:



The formula is of the bulk RM 8850 and therefore includes a contribution from an unknown impurity phase discussed in the “Diffraction” section. 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 for are: Na \pm 0.7, Al \pm 0.7, Si \pm 1.0, H₂O \pm 0.9. The uncertainties are propagated from the uncertainties in mass fractions given in Table 1. Trace elements have not been included in the formula.

NMR Analyses for Si/Al and Si Coordination:

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

Ratio^(a)
2.60 \pm 0.06

^(a) The uncertainty value for Si/Al is an expanded uncertainty about the mean (coverage factor $k = 2$, 95 % confidence).

Example peak assignments and ²⁹Si MAS NMR spectrum are provided in Table 5 and Figure 1 below:

Table 5. An Example of Assignment of Peaks to ²⁹Si MAS NMR Spectrum

Chemical Shift	Assignment
-89.5	Si(OAl) ₃ (OSi)
-94.5	Si(OAl) ₂ (OSi) ₂
-99.2	Si(OAl)(OSi) ₃
-101.4	Si(OAl)(OSi) ₃
-105.6	Si(OSi) ₄

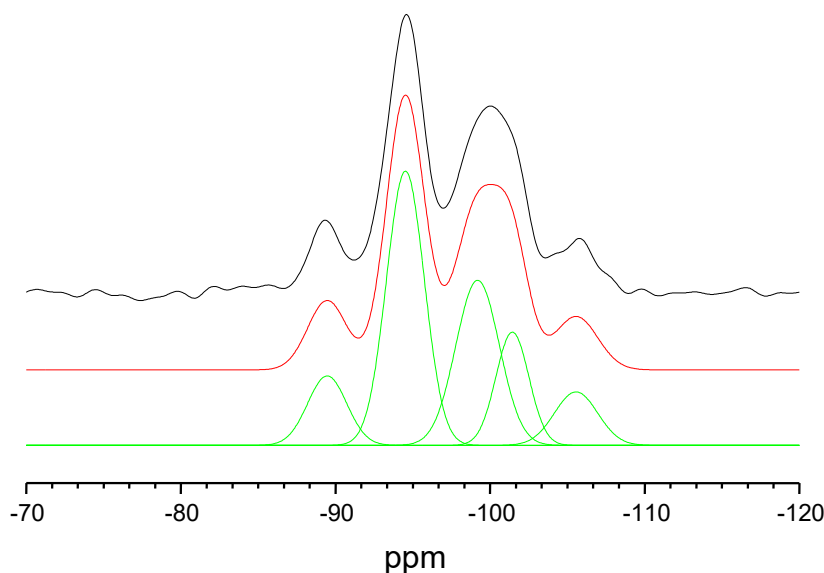


Figure 1. ²⁹Si MAS NMR spectrum (upper), simulated spectrum (middle) and fitted Gaussian peaks (lower).

The Si(OAl)(OSi)₃ region is best fit in the line shape simulation using two Gaussian peaks. Using the peak assignments given above and simulated peak areas (Gaussian peaks), Si/Al ratios were calculated for each spectrum.

NMR Analyses for Al Coordination: The ^{27}Al MAS NMR spectra show a single Lorentzian peak at 60.2 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 sample.

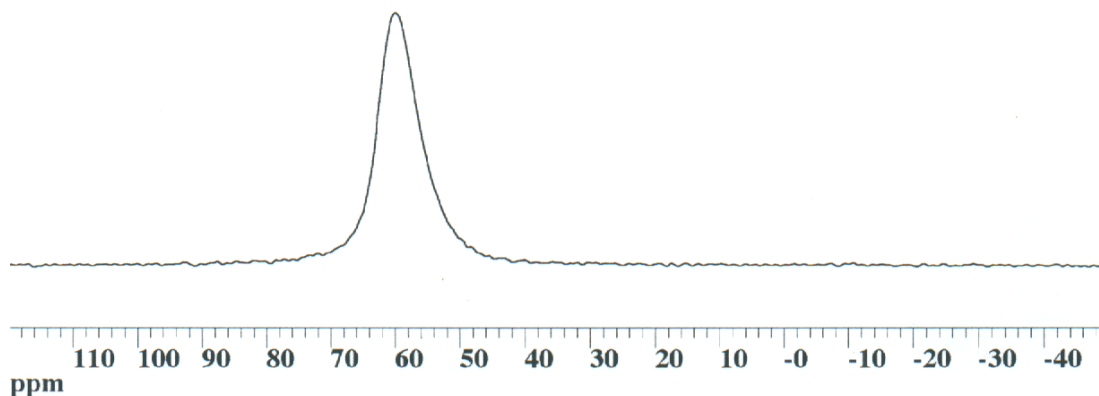


Figure 2. ^{27}Al MAS NMR spectrum obtained from Zeolite Y.

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.

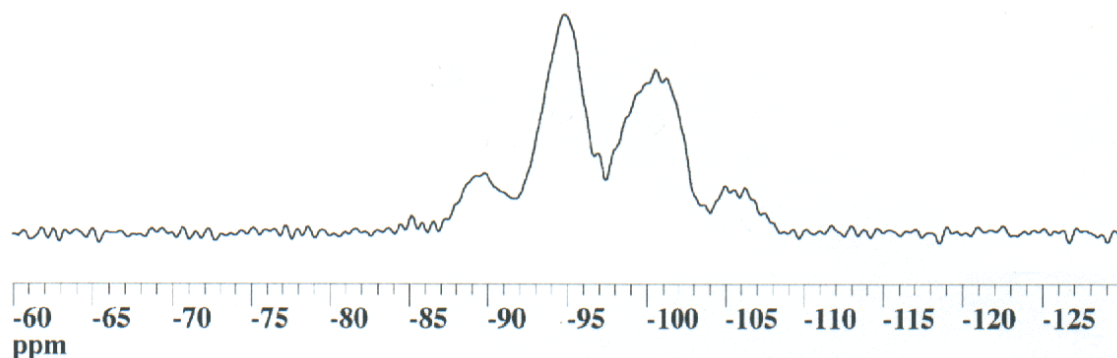


Figure 3. ^{29}Si CP-MAS NMR spectrum obtained from Zeolite Y.

Enthalpy of Formation:

Table 6. Information Values for Enthalpy of Formation at 25 °C

Constituents	Enthalpy of Formation (kJ/mol) ^(a)
Oxides and liquid water	-46.74 ± 0.81
Elements	-1360.1 ± 1.1

^(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 7. Unit Cell Parameter of Hydrated Zeolite Y (Space Group *Fd-3m*)

Axis	Value (nm) ^(a)
a	2.4697 ± 0.0002

^(a) The uncertainty is one standard deviation based on 14 samples.

Note: Peaks from an unknown impurity phase were noted, where the strongest impurity peak was ≈ 0.5 % of the strongest zeolite Y peak. The amount of impurity was not quantified but is presumed small.

Neutron Diffraction of Dehydrated Samples:

Table 8. Unit Cell Parameter of Dehydrated Zeolite Y (Space Group *Fd-3m*)

Axis	Value (nm) ^(a)
a	2.4791 ± 0.0003

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

Note: Peaks from an impurity phase could not be detected.

Table 9. Parameters Used for Rietveld Refinement of Dehydrated Zeolite Y^(a)

Atom	$x/a^{(b)}$	$y/a^{(b)}$	$z/a^{(b)}$	Fractional Occupancy ^(b)	$U_{iso} \times 100^{(b)}$	Site Multiplicity
T	0.125 52 (21)	0.945 45 (20)	0.035 82 (25)	1	1.38 (10)	192
O1	0.176 14 (19)	0.176 14 (19)	0.967 03 (25)	1	3.5 (2)	96
O2	0.177 28 (20)	0.177 28 (20)	0.317 90 (25)	1	2.5 (2)	96
O3	0.252 04 (17)	0.252 04 (17)	0.142 09 (23)	1	2.5 (2)	96
O4	0.106 58 (15)	-0.106 58 (15)	0	1	1.4 (2)	96
Na1	0.055 0 (5)	0.055 0 (5)	0.055 0 (5)	0.637 (33)	2.8 (9)	32
Na2	0	0	0	0.41 (5)	5.6 (24)	16
Na3	0.234 6 (4)	0.234 6 (4)	0.234 6 (4)	1	3.0 (4)	32

^(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 10. Cumulative Volume Percent Less Than Particle Diameter of Sixteen Samples^(a)

Cumulative Volume (%)	Mean Diameter (μm)	Standard Deviation of the Mean (μm)
5	0.411	0.003
10	0.566	0.004
15	0.714	0.005
20	0.861	0.006
25	1.012	0.006
30	1.169	0.007
35	1.334	0.007
40	1.510	0.008
45	1.700	0.008
50	1.908	0.009
55	2.139	0.010
60	2.397	0.011
65	2.694	0.013
70	3.042	0.015
75	3.455	0.017
80	3.968	0.021
85	4.633	0.026
90	5.587	0.036
95	7.309	0.060

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

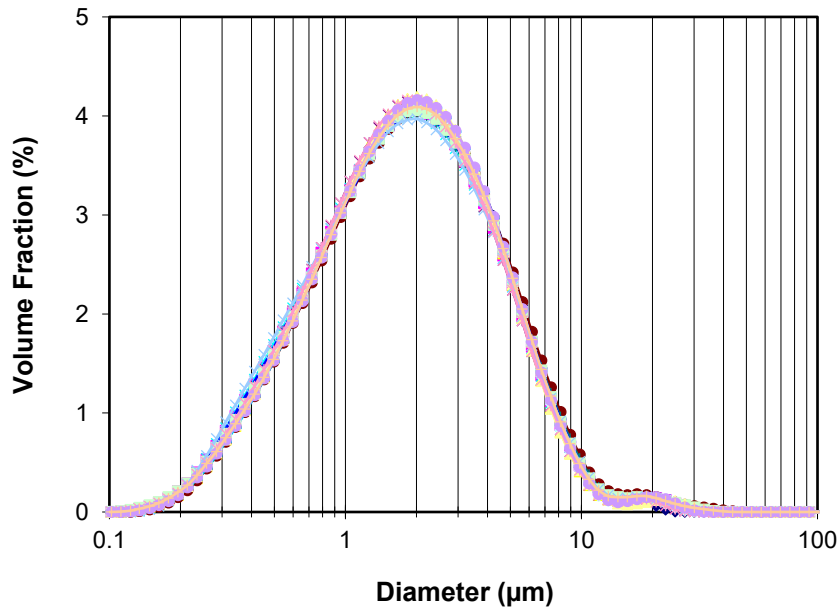


Figure 4. Distribution of differential volume fraction (%) vs. particle diameter as measured by the laser light scattering method. Each curve represents the analysis for a different bottle of the eight bottles analyzed.

Refractive Index Determination for Mie Optical Model:

Table 11. Refractive Index Determination

Refractive Index^(a)
1.442 to 1.444

^(a) A value of 1.44 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 12. Cumulative Volume Percent Less Than Particle Diameter of Six Samples^(a)

Cumulative Volume (%)	Mean Diameter (μm)	Standard Deviation of the Mean (μm)
5	0.99	0.02
10	1.12	0.02
15	1.24	0.02
20	1.34	0.02
25	1.44	0.03
30	1.53	0.02
35	1.64	0.02
40	1.74	0.02
45	1.86	0.02
50	1.99	0.02
55	2.14	0.02
60	2.32	0.01
65	2.54	0.02
70	2.83	0.02
75	3.19	0.03
80	3.66	0.04
85	4.34	0.07
90	5.43	0.11
95	7.51	0.20

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

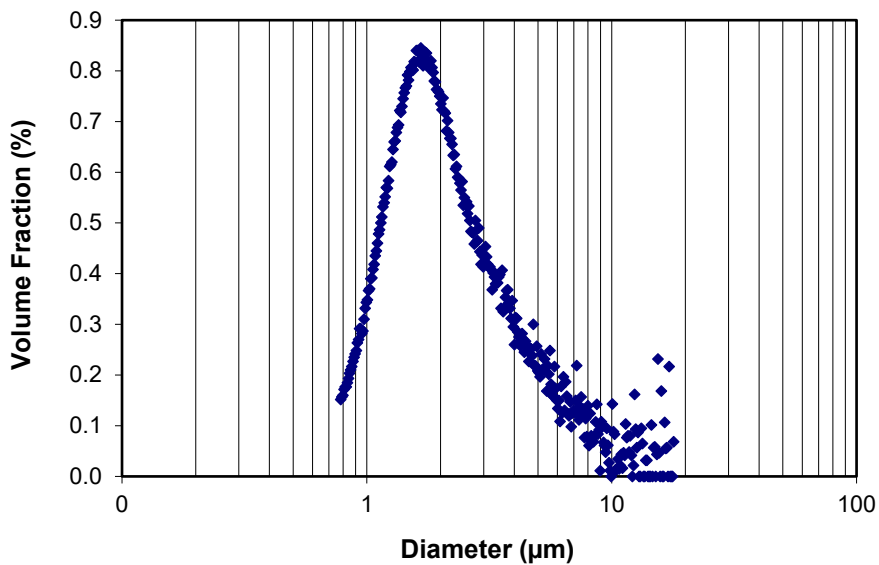


Figure 5. Distribution of differential volume fraction (%) vs. particle diameter as measured by the electric sensing zone method (distribution is the average of six runs).

Laser Light Extinction:

Table 13. Cumulative Number of Particles Greater than Nominal Particle Diameter per μg Zeolite^(a)

Diameter (μm)	Cumulative Number	Standard Deviation	Cumulative Number (%)
1.0	62 297	176.6	100
1.5	13 134	58.7	21.08
2.0	5 168.7	29.0	8.30
2.5	3 467.1	23.0	5.57
3.0	2 719.6	18.1	4.37
3.5	1 944.9	15.9	3.12
4.0	1 221.5	13.8	1.96
4.5	727.6	10.1	1.17
5.0	471.8	8.2	0.76
5.5	349.6	8.4	0.56
6.0	275.4	7.7	0.44
6.5	212.6	6.3	0.34
7.0	152.4	5.1	0.24
7.5	101.2	4.2	0.16
8.0	65.3	3.3	0.10
8.5	42.0	2.9	0.07
9.0	27.3	2.1	0.04
9.5	18.6	1.6	0.03
10.0	13.0	1.4	0.02
10.5	9.6	1.2	0.02

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

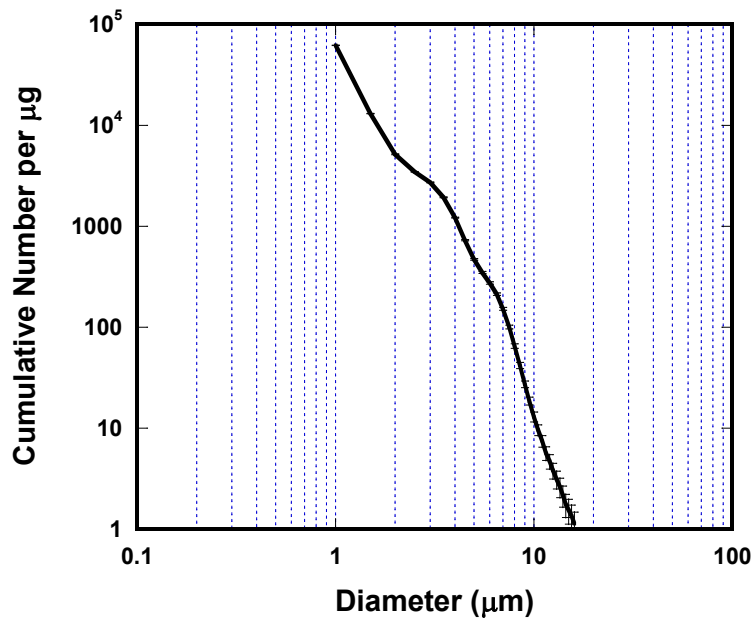


Figure 6. Distribution of the cumulative number of particles per μg greater than particle diameter as measured by the laser light extinction method. The uncertainty bars correspond to the standard deviation of the measured values.

Table 14. Cumulative Volume Percent Less Than Particle Diameter^(a)

Cumulative Volume (%)	Diameter (μm)	Uncertainty (μm) ^(b)
5	1.21	0.04
10	1.37	0.04
15	1.62	0.05
20	1.90	0.04
25	2.50	0.06
30	3.17	0.06
35	3.58	0.07
40	3.83	0.07
45	4.14	0.07
50	4.42	0.08
55	4.75	0.07
60	5.17	0.06
65	5.83	0.08
70	6.50	0.10
75	7.00	0.10
80	7.58	0.10
85	8.17	0.08
90	9.08	0.06
95	11.80	0.03

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

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

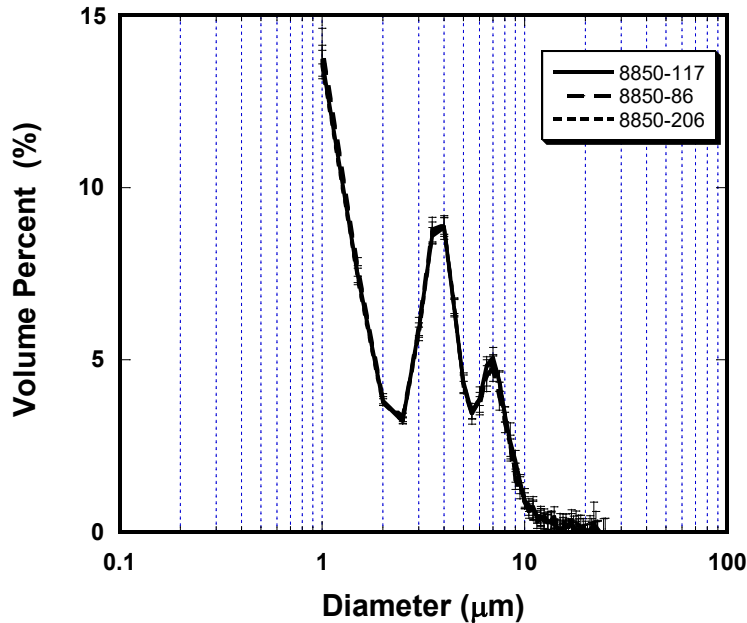


Figure 7. Distribution of differential volume fraction (%) vs. particle diameter as measured by the laser light extinction method. Each of the three curves (one each for the three bottles analyzed) represents the mean value ($n = 5$ to 10) of the volume distribution. The uncertainty bars correspond to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

X-ray Sedimentation:

Table 15. 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	0.77	0.566 \pm 0.004	1.12 \pm 0.02	1.37 \pm 0.04
25	1.13	1.012 \pm 0.006	1.44 \pm 0.03	2.50 \pm 0.06
50	1.66	1.908 \pm 0.009	1.99 \pm 0.02	4.42 \pm 0.08
75	2.70	3.455 \pm 0.017	3.19 \pm 0.03	7.00 \pm 0.10
90	4.56	5.587 \pm 0.036	5.43 \pm 0.11	9.08 \pm 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).

Note: The data in the particle sizing section are for information only. The uncertainties for the reported data do not include all sources of bias. For this material, there is an apparent difference in results obtained by the laser light extinction method and the other particle sizing methods.

STEM:

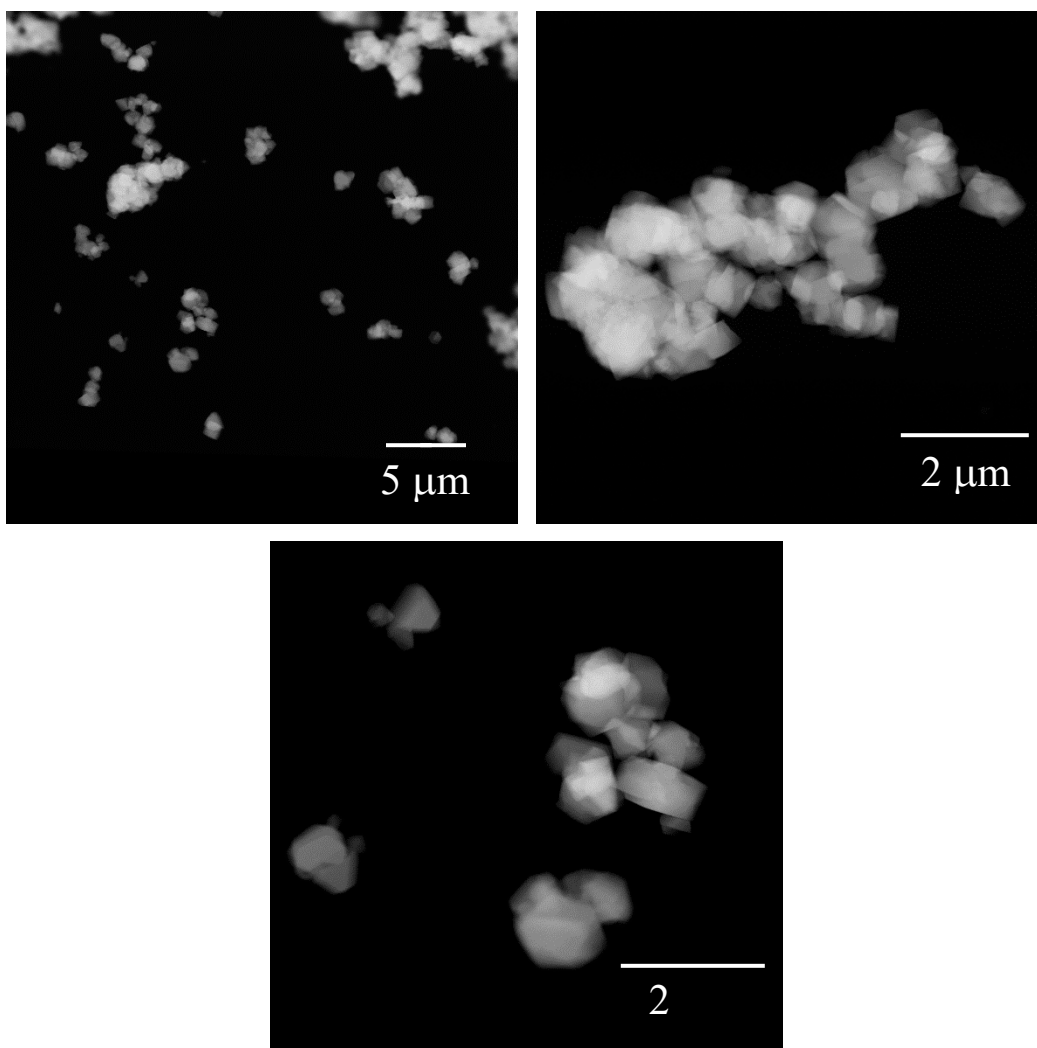


Figure 8. Dark field STEM images of Zeolite Y.

SEM:

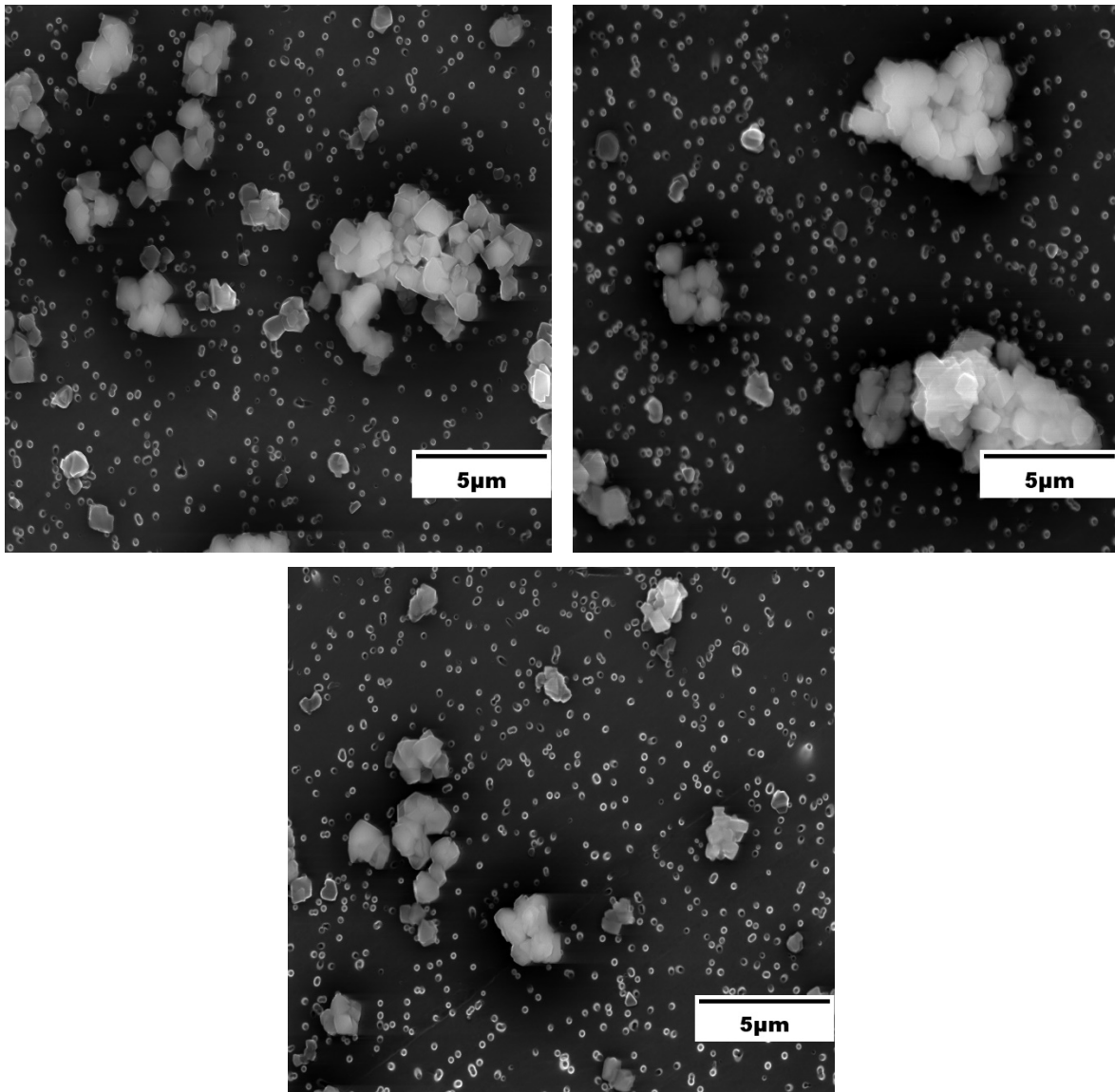


Figure 9. SEM images of Zeolite Y. Small holes in background are images of the pores in the polycarbonate filter upon which the zeolite was deposited.

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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 ($33\% \pm 2\%$ RH and $54\% \pm 2\%$ RH).

Approximately 1.1 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 salt solution of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ with excess salt) and allowed to equilibrate at $33\% \pm 2\%$ 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 $33\% \pm 2\%$ 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 $54\% \pm 2\%$ 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 hour and periodically over the next several days.

The resultant mass change for the first two hours is shown in Figure A1. After 48 h, the experiment shows a mass gain of approximately 0.55 % between the $33\% \pm 2\%$ RH and $54\% \pm 2\%$ RH provided by the two salt solutions. The data show slight additional mass gain ($< 0.1\%$) over an extended period of exposure (several days) to $54\% \pm 2\%$ RH.

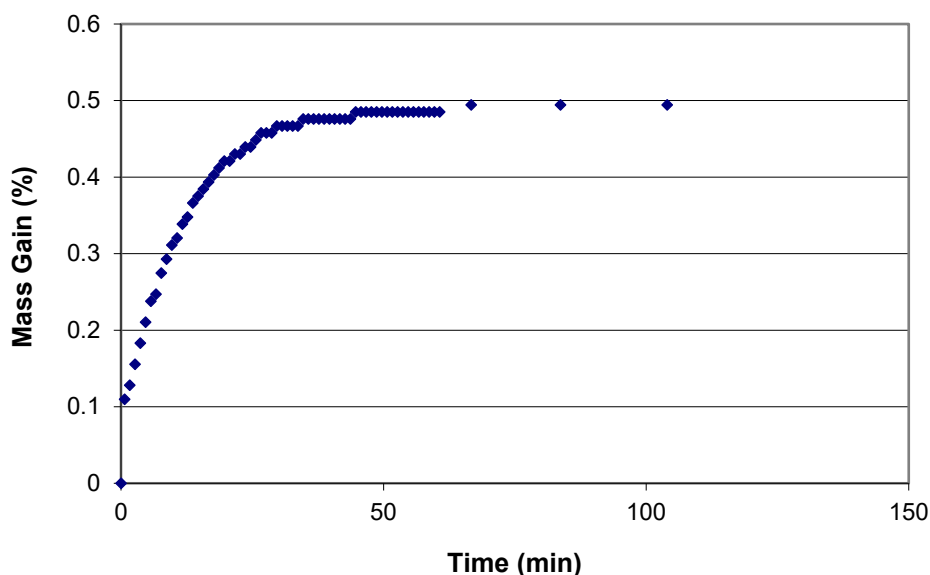


Figure A1. Variation in mass of Zeolite Y as the ambient humidity was changed from $33\% \pm 2\%$ RH (time = 0) to $54\% \pm 2\%$ RH (time > 0 min).