



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material 1980

#### Positive Electrophoretic Mobility ( $+\mu_E$ ) Standard

This Standard Reference Material (SRM) contains 500 mg/L goethite ( $\alpha$ -FeOOH) suspension saturated with 100  $\mu\text{mol/g}$  phosphate in a  $5 \times 10^{-2}$  mol/L sodium perchlorate electrolyte solution at a pH of 2.5. The suspension is intended for use in the calibration and evaluation of equipment used to measure electrophoretic mobility. The SRM consists of a single 60-cm<sup>3</sup> polyethylene bottle containing 40 cm<sup>3</sup> of suspension, which is to be diluted prior to analysis. The goethite powder consists of acicular particles with an average dimension of 60 x 20 nm as determined from electron microscopy and is consistent with previously reported data. [1] The characteristics used for the selection of goethite as a standard were its long term stability, ease of mobility control from positive to negative polarity by adjusting the phosphate concentration or pH, reproducibility of mobility measurement, and the narrow particle size distribution in the suspension. Additionally, this material has been the subject of a previous interlaboratory analysis during which goethite was examined as a potential electrophoresis standard. [2]

The certified value and uncertainty for the positive electrophoretic mobility of SRM 1980, is

$$2.53 \pm 0.12 \mu\text{m}\cdot\text{cm}/\text{V}\cdot\text{s}$$

The starting material was synthesized in several batches using the procedure of Atkinson et al., and then washed with ultrapure water several times, freeze dried and stored in an air tight polyethylene bottle. [3] The combined powder obtained from different batches has a BET surface area of  $84 \pm 3 \text{ m}^2/\text{g}$ . Ten g of powder were suspended in 20 L of  $5 \times 10^{-2}$  mol/L sodium perchlorate solution adjusted to pH 2.5. To this well-stirred suspension, 100  $\mu\text{mol/g}$  potassium dihydrogen phosphate was added. The suspension was dispersed by sonication for 10 min and then aged for 60 days. From the aged suspension, 40 cm<sup>3</sup> aliquots were transferred into 500 polyethylene bottles using a unidispenser. Maximum care was taken to avoid inhomogeneity while dispensing the samples. A set of 12 bottles from the 500 was selected at random for homogeneity testing. In order to prepare individual test samples for analysis, two 10 cm<sup>3</sup> aliquots were taken by pipette from each bottle and each diluted to 100 cm<sup>3</sup> with deionized water. Additionally, randomly chosen samples from the group of 500 were sent to four other laboratories for interlaboratory data comparison. The data from these laboratories were obtained using three different electrophoretic measuring systems following the procedure specified by NIST. The certified mobility value is based on measurements by the five laboratories.

Certification measurements and technique development were performed by S.G. Malghan, R.S. Premachandran, and V.A. Hackley of the NIST Ceramics Division.

Statistical analyses were performed by S.B. Schiller of the NIST Statistical Engineering Division.

Participation by the University of Florida, University of Wisconsin, PenKem Incorporated and Coulter Corporation in the development of this SRM is gratefully acknowledged.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by N.M. Trahey.

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## Results of Statistical Analysis of Material and Measurement Variability

The certified value was computed using an equally weighted mean of the injections for NIST and round robin data. The uncertainty is calculated according to NIST Technical Note 1297 "Guidelines for Evaluating and Expressing Uncertainty of NIST Measurement Results." [4] It includes uncertainty due to the measurement process and due to material variability, and has an approximate level of confidence of 95 %.

To compute the overall uncertainty, the responses were modeled as a function of bottle, sample within bottle, and measurement error. The standard uncertainty due to material is the between-bottle standard deviation, and the standard uncertainty due to the measurement process is the standard deviation of the mean response. These standard uncertainties, as well as their associated degrees of freedom, are shown in Table 1. The combined standard uncertainty is the root-sum-of-squares of the material and measurements standard uncertainties, and the coverage factor is the t-multiplier for a two-sided 95 % confidence interval with degrees of freedom listed for the combined standard uncertainty. The expanded uncertainty is the product of the coverage factor and the combined standard uncertainty.

Table 1

Sources of uncertainty due to material and measurements

| <u>Sources of<br/>Uncertainty</u> | <u>Degree of<br/>Freedom</u> | <u>Standard<br/>Uncertainty</u> |
|-----------------------------------|------------------------------|---------------------------------|
| Material                          | 5.1                          | 0.045                           |
| Measurements                      | 14.3                         | 0.014                           |
| Combined                          | 5.6                          | 0.047                           |
| Coverage Factor                   | 2.5                          |                                 |
| Expanded Uncertainty              |                              | 0.117                           |

Note that the SRM is certified for electrophoretic mobility only, and not for pH measurement. The procedure described in the Appendix should be followed to obtain the certified value.

The instruments used by NIST and round robin participants, and mean values of their measurements, are shown in Table 2.

Table 2

Instruments used by participating laboratories and mean values of mobilities and uncertainty in mobility data

| <u>Laboratory</u>  | <u>Instrument</u>                  | <u>Number of Measurements</u> | <u>Mean Mobility, in <math>\mu\text{m}\cdot\text{cm}/\text{V}\cdot\text{s}</math></u> | <u>95% Confidence in <math>\mu\text{m}\cdot\text{cm}/\text{V}\cdot\text{s}</math></u> |
|--------------------|------------------------------------|-------------------------------|---------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------|
| NIST               | Malvern ZetaSizer III <sup>1</sup> | 96                            | 2.54                                                                                  | 0.06                                                                                  |
| Univ. of Florida   | Coulter Delsa 440 <sup>1</sup>     | 8                             | 2.49                                                                                  | 0.09                                                                                  |
| Univ. of Wisconsin | Pen Kem 3000 <sup>1</sup>          | 4                             | 2.56                                                                                  | 0.13                                                                                  |
| Coulter Corp.      | Coulter Delsa 440 <sup>1</sup>     | 4                             | 2.45                                                                                  | 0.13                                                                                  |
| Pen Kem, Inc.      | Pen Kem 3000 <sup>1</sup>          | 4                             | 2.57                                                                                  | 0.13                                                                                  |

<sup>1</sup>Certain manufacturers are mentioned for information purposes only and do not represent an endorsement by NIST.

#### REFERENCES

- [1] Hackley, V.A. and Anderson, M.A. *Langmuir* **5**, 191 (1989).
- [2] Ceramic Technology Project, Department of Energy/Oak Ridge National Laboratory, Contract No. DE-AC05-84OR21400.
- [3] Atkinson, R.J., Posner, A.M., and Quirk, J.P. *J. Inorg. Nucl. Chem.* **30**, 2371 (1968).
- [4] Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results", NIST Technical Note 1297, National Institute of Standards and Technology, Technology Administration, U.S. Department of Commerce, 1993.

#### APPENDIX

**SAMPLE PREPARATION PROCEDURE:** Shake the SRM bottle vigorously for 1 min with wrist action, then transfer a 10 cm<sup>3</sup> aliquot by pipette to a 100 cm<sup>3</sup> volumetric flask and make to volume with deionized water. Mix the resulting suspension ("the sample") thoroughly and transfer to a polyethylene (non-glass) bottle. Ultrasonicate for 1 min at 40 W. After cooling to 20-25 °C, measure the sample pH. The pH should fall within the range 3.5  $\pm$  0.1; if not, adjust the value using 0.1 mol/L nitric acid or sodium hydroxide.

**SAMPLE ANALYSES PROCEDURE:** The following procedures should be used for sample analysis. Shake the sample with wrist action before each analysis. Prior to the first run, precondition the electrophoresis cell with the sample for 1 min, introduce the sample for analysis and allow to equilibrate for 10-15 s, and make three consecutive mobility measurements. Take the average (unweighted) of these values. Each measurement should be of approximately 20 s duration.

**WARNING:** Water used for dilution must be ultrapure (deionized, 18 megohm resistance); use of tap water may lead to contamination of sample. Glassware and plasticware should be meticulously clean (wash with copious amounts of ultrapure water before using). Do not use phosphate-containing soaps or hydrochloric acid as cleaning agents.

*Application Notes*

- (1) Temperature dependence: Application range is 20 to 25 °C. Sample mobility should not vary more than 0.02  $\mu\text{m}\cdot\text{cm}/\text{V}\cdot\text{s}$  per °C over this range.
- (2) Sample appearance after dilution and over an extended period of time (24 h) should remain translucent bright yellow with no visible sediment. If this condition is not manifest, the sample is contaminated and should be discarded. Repeat dilution procedure. If poor appearance recurs, check WARNING above before proceeding.