



# Certificate of Analysis

## Standard Reference Material<sup>®</sup> 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 evaluation of equipment, and the validation of methodology used to measure electrophoretic mobility. A unit of SRM 1980 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 nm  $\times$  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 measurements, and the narrow particle size distribution in the suspension [1,2]. Additionally, this material was the subject of a previous interlaboratory analysis during which goethite was suggested as a potential electrophoresis standard [3,4].

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 [5]. It includes uncertainty due to the measurement process and due to material variability, and has an approximate level of confidence of 95 %. The instruments used by NIST and round robin participants, and mean values of their measurements, are shown in Table 2.

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

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

**Expiration of Certification:** The certification of **SRM 1980** is valid, within the measurement uncertainty specified, until **01 September 2016** provided the SRM is handled and stored in accordance with the instructions given in the certificate (see "Instructions for Use"). However, the certification will be nullified if the SRM is damaged, contaminated, or otherwise modified.

**Maintenance of SRM Certification:** NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

**Stability:** The material was last validated/stability tested in October 2012.

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

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

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

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*Certificate Revision History on Last Page*

Robert L. Watters, Jr., Director  
Office of Reference Materials

Participation by the University of Florida (Gainesville, FL), University of Wisconsin (Madison, WI), PenKem Inc. (Bedford Hills, NY), and Coulter Corp. (Miami, FL) in the development of this SRM is gratefully acknowledged. Participation by Beckman-Coulter Corp. (Brea, CA), Brookhaven Instruments Corp. (Holtville, NY), and Malvern Instruments, Inc. (Worcestershire, UK) in the evaluation of stability of this SRM is also acknowledged.

## PREPARATION AND ANALYSIS<sup>(1)</sup>

**Preparation:** The starting material was synthesized in several batches using the procedure of Atkinson et al. [6] and then washed with ultrapure water several times, freeze dried and stored in an air tight polyethylene bottle. The combined powder obtained from different batches has a BET surface area of  $84 \text{ m}^2/\text{g} \pm 3 \text{ m}^2/\text{g}$ . Ten grams of powder was suspended in 20 L of  $5 \times 10^{-2} \text{ mol/L}$  sodium perchlorate solution adjusted to pH 2.5. To this well-stirred suspension,  $100 \text{ }\mu\text{mol/g}$  potassium dihydrogen phosphate was added. The suspension was dispersed by sonication for 10 minutes and then aged for 60 days. From the aged suspension,  $40 \text{ cm}^3$  aliquots were transferred into 500 polyethylene bottles using a unidispenser. Maximum care was taken to avoid inhomogeneity while dispensing the samples.

**Analysis:** A set of 12 bottles from the 500 bottles was selected at random for homogeneity testing. In order to prepare individual test samples for analysis, two  $10 \text{ cm}^3$  aliquots were taken by pipette from each bottle and each diluted to  $100 \text{ cm}^3$  with deionized water. Additionally, randomly chosen samples from the group of 500 bottles 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.

## INSTRUCTIONS FOR USE

**Sample Preparation Procedure:** Shake the SRM bottle vigorously for 1 minute with wrist action, then transfer a  $10 \text{ cm}^3$  aliquot by pipette to a  $100 \text{ cm}^3$  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 minute at 40 W. After cooling to  $20 \text{ }^\circ\text{C}$  to  $25 \text{ }^\circ\text{C}$ , measure the sample pH. The pH should fall within the range  $3.5 \pm 0.1$ ; if not, adjust the value using  $0.1 \text{ mol/L}$  nitric acid or  $0.1 \text{ mol/L}$  sodium hydroxide.

**Sample Analyses Procedure:** The following procedures should be used for sample analysis. Shake the sample mildly with wrist action before each analysis. Prior to the first run, precondition the electrophoresis cell with the sample for 1 minute. Then introduce a fresh sample for analysis and allow to equilibrate for 10 s to 15 s, and make three consecutive mobility measurements. Take the average (unweighted) of these values.

**Warning:** Water used for dilution must be ultrapure (deionized,  $18 \text{ M}\Omega$  resistance); use of tap water may lead to contamination of sample; use of distilled water is not certified, but should produce similar results to deionized water if no other contamination is present. Glassware and plasticware should be meticulously clean (wash with copious amounts of ultrapure water before using). Avoid phosphate-containing soaps, sulfuric acid, and hydrochloric acid as cleaning agents.

## Application Notes

- (1) Temperature dependence: Application range is  $20 \text{ }^\circ\text{C}$  to  $25 \text{ }^\circ\text{C}$ . Sample mobility should not vary more than  $0.02 \text{ }\mu\text{m}\cdot\text{cm}/\text{V}\cdot\text{s}$  per  $^\circ\text{C}$  over this range.
- (2) Sample appearance after dilution should be translucent bright yellow with no visible sediment. If this condition is not manifest, the sample may be contaminated and should be discarded. Repeat dilution procedure. If poor appearance recurs, check “Warning” above before proceeding.
- (3) After initially unsealing the SRM bottle, the undiluted material should remain viable for up to 7 days if tightly capped between uses and stored at room temperature. Therefore, several diluted samples can be obtained from a single bottle over multiple days.

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<sup>(1)</sup> Certain commercial equipment, instruments, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 1. Sources of Uncertainty Due to Material and Measurements

Sources of Uncertainty	Degree of Freedom	Standard Uncertainty
Material	5.1	0.045
Measurements	14.3	0.014
Combined	5.6	0.047
Coverage Factor	2.5	
Expanded Uncertainty	0.117	

To compute the overall uncertainty, the responses were modeled as a function of bottle, sample within bottle, and measurement error. The standard uncertainty ( $u_c$ ) 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,  $U$ , is the product of the coverage factor ( $k$ ) and the combined standard uncertainty ( $U = ku_c$ ).

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

Table 2. Instruments Used by Participating Laboratories and Mean Values of Mobilities and Uncertainty in Mobility Data

Laboratory	Instrument	Number of Measurements	Mean Mobility ( $\mu\text{m}\cdot\text{cm}/\text{V}\cdot\text{s}$ )	Uncertainty ( $\mu\text{m}\cdot\text{cm}/\text{V}\cdot\text{s}$ )
NIST	Malvern ZetaSizer III	96	2.54	0.06
Univ. of Florida	Coulter Delsa 440	8	2.49	0.09
Univ. of Wisconsin	Pen Kem 3000	4	2.56	0.13
Coulter Corp.	Coulter Delsa 440	4	2.45	0.13
Pen Kem, Inc.	Pen Kem 3000	4	2.57	0.13

## REFERENCES

- [1] Hackley, V.A.; Anderson, M.A.; *Langmuir*; Vol. 5, p. 191 (1989).
- [2] Tejedor-Tejedor, M.I.; Anderson, M.A.; *Langmuir*; Vol. 6, p. 602 (1990).
- [3] Ceramic Technology Project, Department of Energy/Oak Ridge National Laboratory, Contract No. DE-AC05-84OR21400.
- [4] Zeltner, W.A.; Wang, J.; Omatete, O.O.; Janney, M.A.; Tejedor-Tejedor, M.I.; Anderson, M.A.; Riman, R.E.; Shanefield, D.J.; Adair, J.H.; *Characterization Techniques for the Solid-Solution Interface*; Adair, J.H.; Casey, J.A.; Venigalla, S., Eds.; American Ceramic Society, Westerville, OH, p. 87 (1993).
- [5] 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 <http://www.nist.gov/pml/pubs/index.cfm> (accessed Oct 2012).
- [6] Atkinson, R.J.; Posner, A.M.; Quirk, J.P.; *J. Inorg. Nucl. Chem.*; Vol. 30, p. 2371 (1968).

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Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 948-3730; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.