



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 1880a

#### Portland Cement

This Standard Reference Material (SRM) is intended primarily for use in evaluating chemical methods of analysis and in the calibration of instrumental methods for analysis of cements and materials of similar matrix. A unit of SRM 1880a consists of four sealed vials, each containing approximately 5 g of Portland cement ground to pass a 75  $\mu\text{m}$  (200 mesh) sieve.

**Certified Values:** The certified values for SRM 1880a are provided in Table 1, expressed as mass fractions [1] on an as-received basis. A NIST certified value is a value for which NIST has the highest confidence in its accuracy, in that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified values listed are based on the results of analyses performed at NIST and at Construction Technology Laboratories, Inc. (CTL) using x-ray fluorescence spectrometry, atomic absorption spectrophotometry, and reference methods given in ASTM C 114-97 Standard Test Methods for Chemical Analysis of Hydraulic Cement [2]. Homogeneity testing was performed using x-ray fluorescence spectrometry. The uncertainty listed with each value is an expanded uncertainty, with coverage factor 2, calculated by combining a between-method variance [3] with a pooled, within-method variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurement [4].

**Reference Values:** The reference values for ZnO, Cl, and Loss On Ignition (LOI) are provided in Table 2, expressed as mass fractions on an as-received basis. Reference values are noncertified values that are the best estimate of the true value; however, the values, which are based on determinations done by a single reliable method, do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision and may not include all sources of uncertainty.

**Information Values:** Information values for F, Insoluble Residue, and Free CaO are provided in Table 3. These are noncertified values with no uncertainty assessed. In addition, data from the Cement and Concrete Reference Laboratory Proficiency Sample Program (CCRL), Sample 126, are provided in Table 4 in order to demonstrate user experience with this material using conventional methods.

**Expiration of Certification:** The certification of this SRM is valid until **01 July 2014**, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see Use and Handling). However, the certification will be nullified if the SRM is damaged, contaminated, or modified.

The coordination of technical measurements for certification was accomplished under the direction of J.R. Sieber of the NIST Analytical Chemistry Division. Analytical measurements for certification of this SRM were performed by J.R. Sieber, A.F. Marlow, and P.R. Seo of the NIST Analytical Chemistry Division and by D. Broton, S. Nettles, M. Bharucha, and S. Padiyara of CTL, Skokie, IL.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald.

Willie E. May, Chief  
Analytical Chemistry Division

Gaithersburg, MD 20899  
Certificate Issue Date: 29 July 1999

Thomas E. Gills, Director  
Office of Measurement Services

Statistical consultation for this SRM was provided by S.D. Leigh of the NIST Statistical Engineering Division.

**Stability:** This material is considered to be stable during the period of certification. NIST will monitor this material and will report any significant changes in certification to the purchaser. Return of the attached registration card will facilitate notification.

**Use and Handling:** Cement powder is hygroscopic and the following procedure is recommended. Samples should be used immediately after opening. To relate analytical determinations to the certified value in this Certificate of Analysis, a minimum sample mass of 500 mg should be used. The vial should be recapped immediately and stored in a desiccator over magnesium perchlorate or phosphorus pentoxide. When a sample is used after storage in a previously opened vial, the LOI for that sample should be determined in accordance with ASTM C 114 and the weight of the sample corrected for any additional moisture above the LOI value reported in this certificate.

**Calibration of X-ray Methods:** To obtain the most accurate results by x-ray fluorescence methods of analysis, it is recommended that the user employ calibration procedures utilizing corrections for interelement effects to minimize biases. Alternatively, the user may compare samples to the particular SRM that is most similar in overall chemical composition.

**Cooperating Analysts:** In order to help evaluate conventional wet test methods using this SRM, a number of industry laboratories provided confirmatory analyses. The following analysts provided information:

G. Choy, Hawaii Department of Transportation, Highways Division, Materials Testing and Research Branch, Honolulu, HI, USA

W. Medford, North Carolina Department of Transportation, Materials & Tests Unit, Raleigh, NC, USA

K. Maylin, Blue Circle Cement, Bowmanville, Ontario, Canada

D. Kerridge, Hercules Cement Company, Stockertown, PA, USA

J.R. Arnold, Lafarge Corporation, Paulding Plant, Paulding, OH, USA

A. Godek, Ash Grove Cement Company, Nephi, UT, USA

**Reporting:** The constituents listed in this Certificate of Analysis are expressed as the chemical forms, and in the order given, in ASTM C 114-97, Section 3, Table 1.

Table 1. Certified Values for SRM 1880a, Portland Cement

Constituent	Mass Fraction (%)	Constituent	Mass Fraction (%)
SiO <sub>2</sub>	20.31 ± 0.28	K <sub>2</sub> O	0.92 ± 0.05
Al <sub>2</sub> O <sub>3</sub>	5.18 ± 0.10	TiO <sub>2</sub>	0.25 ± 0.03
Fe <sub>2</sub> O <sub>3</sub>	2.81 ± 0.04	P <sub>2</sub> O <sub>5</sub>	0.22 ± 0.02
CaO	63.83 ± 0.46	Mn <sub>2</sub> O <sub>3</sub>	0.127 ± 0.005
MgO	1.72 ± 0.04	SrO	0.083 ± 0.021
SO <sub>3</sub>	3.25 ± 0.06	Cr <sub>2</sub> O <sub>3</sub>	0.007 ± 0.002
Na <sub>2</sub> O	0.19 ± 0.02		

Table 2. Reference Values for SRM 1880a, Portland Cement

Constituent	Mass Fraction (%)	Constituent	Mass Fraction (%)
ZnO	0.005 ± 0.001	Cl	0.007 ± 0.001
LOI at 950 °C	1.32 ± 0.02		

Table 3. Information Values for SRM 1880a, Portland Cement

Constituent	Mass Fraction (%)	Constituent	Mass Fraction (%)
F	0.06	Insoluble Residue	0.22
Total <sup>a</sup>	100.31	Free CaO	0.84

<sup>a</sup> A correction has been made for the amount of fluoride present. This correction, which was subtracted from the gross total, was determined by multiplying the percent fluoride by the ratio of the atomic weight of oxygen to the molecular weight of fluorine (0.421).

**CCRL Round Robin:** This material was used as sample number 126 in the CCRL program. As many as 198 laboratories provided analysis results for the constituents of this material. The results are summarized in Table 4, which reports the values calculated by a statistical analysis that identified and removed outliers beyond three standard deviations from the mean. Round robin analyses were performed on the material prior to packaging as an SRM and were not used in the certification of SRM 1880a.

Table 4. CCRL Proficiency Sample Program, Sample No. 126, Portland Cement  
Results from the Final Report Dated September 1997 (with labs eliminated)  
All values in mass fraction (%)

Test	# Labs	Average	Standard Deviation
Silicon Dioxide	173	20.347	0.22
Al Oxide (P <sub>2</sub> O <sub>5</sub> & TiO <sub>2</sub> included)	125	5.699	0.25
Al Oxide (P <sub>2</sub> O <sub>5</sub> & TiO <sub>2</sub> not included)	94	5.133	0.14
Ferric Oxide	169	2.806	0.064
Calcium Oxide	174	63.725	0.54
Magnesium Oxide	158	1.747	0.092
Sulfur Trioxide	170	3.255	0.084
Loss on Ignition	188	1.310	0.087
Sodium Oxide	138	0.182	0.028
Potassium Oxide	137	0.937	0.032
Insoluble Residue	175	0.215	0.077
Free Lime	152	0.782	0.18

## REFERENCES

- [1] Taylor, B.N., "Guide for the Use of the International System of Units (SI)," NIST Special Publication 811, 1995 Ed., (April 1995).
- [2] ASTM C 114-97, Standard Test Methods for Chemical Analysis of Hydraulic Cement, *Annu. Book ASTM Stand. Vol. 04.01*, West Conshohocken, PA.
- [3] Rukhin, A.L. and Vangel, M.G., "Estimation of a Common Mean and Weighted Means Statistics," *J. Amer. Stat. Assoc. (JASA)*, **93 (441)**, pp. 303-308, (1998).
- [4] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993); see also Taylor, B.N. and 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://physics.nist.gov/Pubs/>).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov), or via the Internet <http://ts.nist.gov/srm/>.