National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1880b

Portland Cement

This Standard Reference Material (SRM) is intended primarily for the calibration or evaluation of methods for analysis of cements and materials of similar matrix. A unit of SRM 1880b consists of four sealed vials, each containing approximately 5 g of portland cement ground to pass through a 75 µm (No. 200) sieve.

Certified Values: Certified values for 13 constituents in SRM 1880b are reported in Table 1 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 taken into account [2]. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST and collaborating laboratories using the instrumental and classical test methods listed in the Appendix.

Reference Values: Reference values for seven constituents are reported in Table 2. Reference values are non-certified values that are the present best estimates of the true values. However, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty [2].

Information Values: Information values for three constituents are reported in Table 3 along with a calculated "total" value accounting for all determined constituents. An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [2].

Expiration of Certification: The certification of SRM 1880b is valid, within the measurement uncertainty specified, until **01 November 2023**, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Use"). The certification is 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.

The coordination of technical measurements for certification was performed by J.R. Sieber of the NIST Analytical Chemistry Division.

Analyses leading to the certification of this SRM were performed at NIST by A.F. Marlow and J.R. Sieber of the NIST Analytical Chemistry Division and M. Stair of the Cement and Concrete Reference Laboratory. Analytical determinations for value assignments of SRM 1880b were performed by Construction Technology Laboratories, Inc., Skokie, IL.

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

Support aspects involved in the preparation of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 25 May 2010
See Certificate Revision History on Page 4

Robert L. Watters, Jr., Chief
Measurement Services Division

INSTRUCTIONS FOR USE

Cement powder is hygroscopic and the following procedure is recommended. Samples should be used immediately after opening. To relate analytical determinations to the certified values 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. When a sample is used after storage in a previously opened vial, the Loss on Ignition (LOI) at 950 ºC for that sample should be determined in accordance with ASTM C 114 and the mass of the sample corrected for any additional moisture, combined water, or carbonate above the value reported in this certificate for LOI at 950 ºC.

Preparation and Analysis[1]: The material for SRM 1880b was obtained in the form of powder prepared using a typical industrial process. The material was blended and packaged at NIST. Homogeneity testing was performed at NIST using X-ray fluorescence spectrometry. Material heterogeneity was low and fit for the purpose of value assignment. Quantitative determinations done by NIST included X-ray fluorescence spectrometry [6] and thermogravimetric analysis and by Construction Technology Laboratories, Inc. using X-ray fluorescence spectrometry, inductively coupled plasma optical emission spectrometry, and reference methods given in reference [3].

Reporting: The constituents listed in this Certificate of Analysis are expressed as the chemical forms and in the order given in ASTM C 114-07, Section 3, Table 1 [3].

Table 1. Certified Values for SRM 1880b

Constituent	Mass Fraction [(a)] (%)		
SiO_2	20.42	±	0.36
Al_2O_3	5.183	±	0.073
Fe_2O_3	3.681	±	0.023
CaO	64.16	±	0.40
MgO	1.176	±	0.020
SO_3 [b]	2.710	±	0.099
Na_2O	0.0914	±	0.0052
K_2O	0.646	±	0.014
TiO_2	0.236	±	0.012
P_2O_5	0.2443	±	0.0027
Mn_2O_3	0.1981	±	0.0020
Cl	0.01830	±	0.00057
Cr_2O_3 [(b)]	0.01927	±	0.00042

[(a)] Each certified value is the unweighted mean of the results from two to four methods. The uncertainty of a certified value is expressed as an expanded uncertainty, U, and is calculated according to the method described in the ISO Guide [4,5]. The expanded uncertainty is $U = ku_c$, where u_c is calculated, at the level of one standard deviation, by combining a between-method variance with a pooled, within-method variance. The coverage factor, $k = 2$, was used corresponding to an approximately 95 % confidence level.

[(b)] The uncertainty estimates for SO_3 and Cr_2O_3 include an additional component of uncertainty of 2 % (relative) to account for greater than expected heterogeneity observed during testing of the material after packaging.

[1]Certain commercial organizations, services, equipment, or materials are identified in this certificate in order 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 organizations, services, materials, or equipment identified are necessarily the best available for the purpose.

Table 2. Reference Values for SRM 1880b

Constituent	Mass Fraction (%)		
LOI at 950 ºC [a]	1.666	±	0.011
ZnO [a]	0.01054	±	0.00034
Sulfide Sulfur [b]	0.0131	±	0.0021
Insoluble Residue [b]	0.487	±	0.014
Free CaO [b]	1.567	±	0.059
SrO [b]	0.0272	±	0.0016
Fluoride (F^-) [b]	0.0539	±	0.0012

[a] Each reference value is the unweighted mean of the results from two to four methods. The uncertainty of the reference value is expressed as an expanded uncertainty, *U*, and is calculated according to the method described in the ISO Guide [4,5]. The expanded uncertainty is $U = ku_c$, where u_c is calculated, at the level of one standard deviation, by combining a between-method variance with a pooled, within-method variance. The coverage factor, $k = 2$, was used corresponding to approximately 95 % confidence level.

[b] Each reference value is the mean of results obtained by a single laboratory using one analytical technique. The expanded uncertainty is calculated as $U = ku_c$ where u_c is one standard deviation of the analyte mean, and the coverage factor, $k = 2$, was used corresponding to approximately 95 % confidence level for each analyte.

Table 3. Information Values for SRM 1880b

Constituent	Mass Fraction (%)
LOI at 550 ºC	1.026
LOI at 220 ºC	0.478
Total [a]	100.49

[a] Three corrections have been made to the calculated total of analyzed constituents: 1) the amount of fluorine present, 2) the amount of chlorine present, and 3) the overestimation of oxygen by expressing total S as SO_3 when a quantifiable amount of sulfide sulfur is present. All three corrections were subtracted from the gross total. The correction for F was determined by multiplying the percent fluorine by the ratio of the relative atomic mass of oxygen to two times the relative atomic mass of fluorine (0.421). The correction for chlorine was determined by multiplying the percent chlorine by the ratio of the relative atomic mass of oxygen to two times the relative atomic mass of chlorine (0.226). The correction for sulfide sulfur was determined by multiplying the percent sulfide sulfur by the ratio of three times the relative atomic mass of oxygen to the relative atomic mass of sulfur (1.50).

REFERENCES

[1] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at: http://ts.nist.gov/WeightsAndMeasures/Metric/mpo_pubs.cfm (accessed May 2010).

[2] 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: Gaithersburg, MD (2000); available at http://ts.nist.gov/MeasurementServices/ReferenceMaterials/PUBLICATIONS.cfm (accessed May 2010).

[3] ASTM C 114-07, Standard Test Methods for Chemical Analysis of Hydraulic Cement, *Annu. Book ASTM Stand.*, Vol. 04.01, West Conshohocken, PA.

[4] 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).

[5] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed May 2010); 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 http://physics.nist.gov/Pubs/ (accessed May 2010).

[6] Sieber, J.; Broton, D.; Fales, C.; Leigh, S.; MacDonald, B.; Marlow, A.; Nettles, S.; Yen, J.; *Standard Reference Materials for Cement*, Cement and Concrete Res., 32 (12), pp 1899–1906 (2002).

Certificate Revision History: 25 May 2010 (Correction of reference value for Free CaO and minor editorial changes); 09 March 2009 (Original certificate issue date).

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: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

Constituent	Methods
SiO_2	Total Si determined using XRF, ICP-OES, and gravimetry
Al_2O_3	Total Al determined using XRF and ICP-OES
Fe_2O_3	Total Fe determined using XRF and ICP-OES
CaO	Total Ca determined using XRF, ICP-OES, and gravimetry
MgO	Total Mg determined using XRF and ICP-OES
SO_3	Total S determined using XRF, ICP-OES, and gravimetry
Na_2O	Total Na determined using XRF and ICP-OES
K_2O	Total K determined using XRF and ICP-OES
TiO_2	Total Ti determined using XRF and ICP-OES
P_2O_5	Total P determined using XRF, ICP-OES, and UV absorbance
Mn_2O_3	Total Mn determined using XRF and ICP-OES
Cl	Total Cl determined using XRF[(a)] with standard additions at NIST and ion-selective electrode at the collaborating laboratory
Cr_2O_3	Total Cr determined using XRF and ICP-OES
ZnO	Total Zn determined using XRF
Sulfide S	KIO_3 titration after reaction with HCl
Insoluble Residue	Gravimetry
SrO	Total Sr determined using XRF, ICP-OES
Free CaO	ASTM C 114-07 method performed by the collaborating laboratory
F	Ion-selective electrode at the collaborating laboratory
Loss on Ignition (LOI)	Thermogravimetric Analysis performed by both NIST and the collaborating laboratory with mass loss measured at 220 ºC, 550 ºC, and 950 ºC.

[(a)] Borate fusion was not used for Cl.

Key to Methods:

XRF	X-ray fluorescence spectrometry after borate fusion at NIST and the collaborating laboratory.
ICP-OES	Inductively coupled plasma optical emission spectrometry at the collaborating laboratory.
Gravimetry	Indicates the specific gravimetric method found in ASTM C 114-07 performed by the collaborating laboratory.
UV absorbance	The colorimetric method found in ASTM C 114-07 performed by the collaborating laboratory.