



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 five sealed vials, each containing approximately 5 g of portland cement ground to pass through a 75 μm (No. 200) sieve.

Certified Mass Fraction Values: Certified values for 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 Mass Fraction Values: Reference values for 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 Mass Fraction Values: Information values for 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 or register online) will facilitate notification. Information values cannot be used to establish metrological traceability.

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

Analyses leading to the certification of this SRM were performed at NIST by A.F. Marlow and J.R. Sieber of the NIST Chemical Sciences 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., (CTL Group, 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 Office of Reference Materials.

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Certificate Issue Date: 04 October 2018
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INSTRUCTIONS FOR USE

Cement powder is hygroscopic. Samples should be used immediately after opening the vial to minimize changes from reaction with moisture and carbon dioxide in air. To relate analytical determinations to the certified values in this Certificate of Analysis, a minimum test portion of 500 mg should be used. The vial should be recapped immediately, placed back in the labeled foil pouch, and stored in a desiccator.

When a sample is used after storage in a previously opened vial, the loss on ignition (LOI) at 950 °C value for that sample should be determined in accordance with ASTM C114 Standard Test Methods for Chemical Analysis of Hydraulic Cement [3] and the mass of the sample corrected for any increase above the value reported in Table 2 for LOI total at 950 °C. See Appendix A for more information about LOI of portland cement.

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]. 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].

Certified Mass Fraction Values: The measurands are the mass fractions of the elements in cement listed in Table 1. Metrological traceability is to the SI derived unit for mass fraction (expressed as a percent). 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/JCGM 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.

Table 1. Certified Mass Fraction Values for SRM 1880b Portland Cement

Constituent	Mass Fraction (%)		
SiO ₂	20.42	±	0.36
Al ₂ O ₃	5.183	±	0.073
Fe ₂ O ₃	3.681	±	0.023
CaO	64.16	±	0.40
MgO	1.176	±	0.020
SO ₃ ^b	2.710	±	0.099
Na ₂ O	0.0914	±	0.0052
K ₂ O	0.646	±	0.014
TiO ₂	0.236	±	0.012
P ₂ O ₅	0.2443	±	0.0027
Mn ₂ O ₃	0.1981	±	0.0020
Chlorine (Cl)	0.01830	±	0.00057
Cr ₂ O ₃ ^(a)	0.01927	±	0.00042

^(a) The uncertainty estimates for SO₃ and Cr₂O₃ 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 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.

Reference Mass Fraction Values: The measurands are the mass fractions of the chemical constituents in cement listed in Table 2 as determined by the methods indicated in Table 4. Metrological traceability is to the SI derived unit for mass fraction (expressed as a percent).

Table 2. Reference Mass Fraction Values for SRM 1880b Portland Cement

Constituent	Mass Fraction (%)
Insoluble residue ^(a)	0.487 ± 0.014
Free CaO ^(a)	1.567 ± 0.059
Fluoride (F ⁻) ^(a)	0.0539 ± 0.0012
SrO ^(a)	0.0272 ± 0.0016
Sulfide Sulfur ^(a)	0.0131 ± 0.0021
ZnO ^(b)	0.01054 ± 0.00034
Measurand	Mass Fraction (%)
LOI at 950 °C	1.666 ± 0.011

^(a) 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.

^(b) 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/JCGM 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.

Table 3. Information Values for SRM 1880b

Measurand	Mass Fraction (%)
LOI at 550 °C	1.026
LOI at 220 °C	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 SO₃ 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).

Table 4. Analytical Methods Used on SRM 1881b Portland Cement

Constituent	Methods ^(a)
SiO ₂	Total Si determined using XRF, ICP-OES, and gravimetry
Al ₂ O ₃	Total Al determined using XRF and ICP-OES
Fe ₂ O 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 ₂ O	Total Na determined using XRF and ICP-OES
K ₂ O	Total K determined using XRF and ICP-OES
TiO ₂	Total Ti determined using XRF and ICP-OES
P ₂ O ₅	Total P determined using XRF, ICP-OES and UV absorbance
ZnO	Total Zn determined using XRF
Mn ₂ O ₃	Total Mn determined using XRF and ICP-OES
Cr ₂ O ₃	Total Cr determined using XRF and ICP-OES
SrO	Total Sr determined using XRF and ICP-OES
Sulfide S	KIO ₃ titration after reaction with HCl
Insoluble Residue	Gravimetry
Free CaO	ASTM C114-07 method performed by the collaborating laboratory
Chlorine (Cl)	Total Cl determined using XRF ^(b) with standard additions at NIST, and an ion-selective electrode method at the collaborating laboratory
Fluoride (F ⁻)	Ion-selective electrode at collaborating laboratory
Loss on Ignition (LOI)	Thermogravimetric analysis performed at NIST and the collaborating laboratory with mass loss measured at 220 °C, 550 °C, and 950 °C.

^(a) Key to Methods:

XRF	X-ray fluorescence spectrometry after borate fusion at NIST [5] 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 114-15 performed by the collaborating laboratory
UV absorbance	The colorimetric method found in ASTM C 114-07 performed by the collaborating laboratory.

^(b) Borate fusion was not used at NIST for Cl by the standard additions calibration.**NOTICE TO USERS**

NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of this SRM as a primary benchmark for the quality and accuracy of the user's in-house reference materials and working standards. As such, the SRM should be used to validate the more routinely used reference materials in a laboratory. Comparisons between the SRM and in-house reference materials or working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

REFERENCES

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Certificate Revision History: 04 October 2018 (Corrected unit size; editorial changes); 25 May 2010 (Correction of reference value for Free CaO and 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) 948-3730; e-mail srminfo@nist.gov; or via the Internet at <https://www.nist.gov/srm>.