

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1884a

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 1884a consists of four sealed vials, each containing approximately 5 g of portland cement ground to pass a 75 µm (200 mesh) sieve.

Certified Values: The certified values for SRM 1884a expressed as mass fractions [1] on an as-received basis are provided in Table 1. 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)¹ 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 at NIST 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: Reference values for SRM 1884a expressed as mass fractions on an as-received basis are provided in Table 2. Reference values are noncertified values that are the best estimates of the true values; however, the values, which are based on determinations done by reliable methods, 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, Loss On Ignition (LOI), Free Calcium Oxide (CaO), and the Total of all analyzed constituents 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 127, are provided in Table 4 in order to demonstrate user experience with this material using conventional methods. CCRL results should **NOT** be used as substitutes for NIST values.

Expiration of Certification: The certification of this SRM is valid until **01 July 2016**, 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.

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 and A.F. Marlow of the NIST Analytical Chemistry Division and by D. Broton, S. Padiyara, M. Bharucha, R. Naamane, S. Nettles, and C. Wedzicha of CTL, Skokie, IL.

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.

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¹Certain commercial organizations, services, equipment, or materials are identified in this certificate to specify adequately 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.

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 is recommended. 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.

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 1884a Portland Cement

Constituent	Mass Fraction (%)	Constituent	Mass Fraction (%)
SiO_2	20.57 ± 0.11	K_2O	0.997 ± 0.013
Al_2O_3	4.264 ± 0.032	TiO_2	0.186 ± 0.010
Fe_2O_3	2.695 ± 0.078	P_2O_5	0.1278 ± 0.0048
CaO	62.26 ± 0.56	Mn_2O_3	0.0853 ± 0.0015
MgO	4.475 ± 0.080	SrO	0.2984 ± 0.0024
SO_3	2.921 ± 0.030	Cr_2O_3	0.0166 ± 0.0015
Na_2O	0.2161 ± 0.0020		

Table 2. Reference Values for SRM 1884a Portland Cement

Constituent	Mass Fraction (%)	Constituent	Mass Fraction (%)
ZnO	0.0101 ± 0.0009	Cl	0.0037 ± 0.0003

Table 3. Information Values for SRM 1884a Portland Cement

Constituent	Mass Fraction (%)	Constituent	Mass Fraction (%)
F	0.11	Insoluble Residue	0.25
LOI at 950 °C	1.06	Free CaO	0.71
Total ^a	100.25		

^a 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). The Total does not include Insoluble Residue or Free CaO.

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CCRL Round Robin: This material was used as sample number 127 in the CCRL program. 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 1884a.

Table 4. CCRL Proficiency Sample Program, Sample No. 127, Portland Cement Results from the Final Report Dated March 1998 (with labs eliminated)

All values in mass fraction (%)

Test	No. of Labs	Average	Standard Deviation
Silicon Dioxide	157	20.718	0.24
Aluminum Oxide (P ₂ O ₅ & TiO ₂ not include	ed) 171	4.352	0.15
Ferric Oxide	169	2.6956	0.056
Calcium Oxide	164	62.165	0.44
Magnesium Oxide	171	4.5367	0.17
Sulfur Trioxide	161	2.8845	0.093
Loss on Ignition	113	0.8919	0.068
Sodium Oxide	153	0.2158	0.037
Potassium Oxide	165	1.0014	0.035
Manganic Oxide	74	0.0849	0.011
Phosphorus Pentoxide	91	0.1259	0.018
Titanium Dioxide	108	0.1875	0.017

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.*, **04.01**, West Conshohocken, PA.
- [3] Levenson, M.S., Banks, D.L., Eberhardt, K.R., Gill, L.M., Guthrie, W.F., Liu, H.K., Vangel, M.G., Yen, J.H., and Zhang, N.F., "An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM," J. Res. Natl. Inst. Stand. Technol., **105**, No. 4, 571 (2000).
- [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; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.

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