National Institute of Standards and Technology
U.S. Department of Commerce

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Standard Reference Material® 46h
Portland Cement Fineness Standard
CERTIFICATE OF ANALYSIS

Purpose: The certified values delivered by this Standard Reference Material (SRM) are intended for use in calibrating fineness testing equipment according to ASTM Standard Methods.

Description: A unit of SRM 46h contains 10 glass vials of approximately 5 g each of powered cement. Each vial has a plastic snap off cap and is contained in a sealed foil bag.

Certified Values: These values are traceable to International System of Units (SI). A NIST certified value is a value for which NIST has the highest confidence in its accuracy and that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified values for specific surface area and sieve residue are given in Table 1. The certified values of particle size distribution are given in Table 2. The certified values for the surface area and the particle size distribution (PSD) are the mean of results from analyses performed by cooperating laboratories. The certified value for the sieve residue was calculated from NIST data using three calibrated sieves having openings ranging from 38 μm to 56 μm. The measurands are listed in Table 1 and 2. Metrological traceability is to the derived SI units for area per unit of mass; expressed as square meters per kilogram, square centimeters per gram; and derived SI units for mass fraction, expressed as percent; respectively.

The expanded uncertainties of the certified values for specific surface area were calculated according to the NIST uncertainty policy [1], and are at the 95 % confidence level. The uncertainties include measurement variability within and between laboratories. The expanded uncertainty for the sieve residue was computed using a Bayesian analysis and is also at the 95 % probability level. The expanded uncertainty accounts for the variability of random measurement effects, sieve calibrations, and material inhomogeneity. The uncertainty of the PSD is discussed below.

Table l. Certified Values

Measurand	ASTM Method	Certified Value and Expanded Uncertainty
Specific Surface Area (Blaine)	C204-17[(a)]	364.4 m^2/kg ± 2.68 m^2/kg (3644.0 cm^2/g ± 26.8 cm^2/g)
Sieve Residue (45 μm residue)	C 430-96 (2003)[(b)]	7.43 % ± 0.79 %

[(a)] Standard Test Method for Fineness of Portland Cement by Air Permeability Apparatus [Blaine].
[(b)] Standard Test Method for Fineness of Hydraulic Cement by the 45 μm (No. 325) Sieve.

Additional Information: Values of potential use to customers and additional information are provided in Appendix A.

Period of Validity: The certified values delivered by **SRM 46h** are valid within the measurement uncertainty specified until **31 December 2025**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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Certified Values for Particle Size Distribution (PSD): The SRM 46h PSD was determined using laser diffraction (LD) in liquid medium (LD-W) and in air (LD-D) techniques following AASHTO T353-14. The results calculated a mean PSD of the combined value obtained by LD-D and LD-W, and are shown graphically in Figure 1 and tabulated in Table 2, with 95 % uncertainty bounds. The parameters used to develop the PSD were a) The complex refractive index for the cement used had a real part of 1.7 and an imaginary part of 1.0 and b) IPA was used as the liquid medium and the refractive index (real) used was 1.39; the imaginary part was zero.

Table 2. Certified Values for the Particle Size Distribution of SRM 46h Using LD Methodology [2], combination wet and dry

Particle Size [µm]	Mean Cumulative Volume Fraction [%]	Lower 95 % Uncertainty	Upper 95 % Uncertainty
1	4.5	3.95	5.08
1.5	7.6	7.02	8.17
2	10.5	9.945	11.14
3	15.9	15.26	16.56
4	20.7	19.96	21.34
6	28.5	27.72	29.36
8	34.9	34.0	35.84
12	45.1	44.14	46.17
16	53.8	52.69	54.85
24	67.9	66.78	69.02
32	78.3	77.1	79.46
48	90.2	88.94	91.57
64	95.4	94.05	96.84
96	98.6	97.11	100
128	99.1	97.68	100

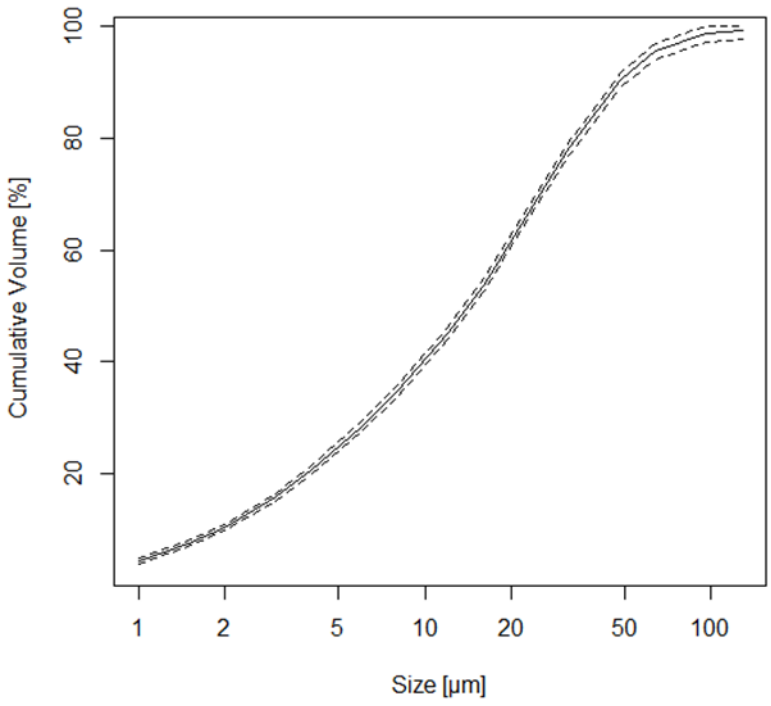

Figure 1. Graphical depiction of the particle size distribution of SRM 46h using LD [2]

NOTES: The purpose of a certified PSD based on an easily accessible reference material is to verify the efficacy of an instrument and the procedure being used.

The approach suggested to compare the data obtained with the SRM 46h value is presented in the next section on "Conformity Determination for PSD". If the data are found to be statistically different from the SRM 46h, the operator should check the performance of the device, the parameters used (such as the refractive indices) or the procedure used (dispersion, ultrasound, duration of measurement, etc.). For more details on the procedure, refer to references 2 and 3.

Conformity Determination for PSD: The operator should start by measuring the SRM 46h at least three times using his own procedure and an instrument based on LD, and computing his average cumulative particle size distribution. If the results fall between the lower and upper bounds of the predictive interval given in Table 3 for all sizes, then the user's results do not differ from the certified value of SRM 46h by more than expected based on the within-lab reproducibility of a typical laboratory at 95 %. If, on the other hand, one or more of the observed cumulative volume fractions does not fall within the lower and upper bounds, this is evidence that the user's results are not in agreement with the certified value of SRM 46h and that changes to the measurement procedures are needed. The 95 % predictive intervals (Table 3) are wider than the 95 % uncertainty intervals given in Table 2 because they include the additional uncertainty of the user's reproducibility estimated as the typical reproducibility uncertainty of a lab that participated in the certification inter-laboratory study. For more details on how the values were obtained, refer to reference 2.

Table 3. Certified Predictive Intervals for the Combined PSD Determined by LD-D and LD-W

Particle Size [µm]	Mean Cumulative Volume Fraction [%]	Lower 95 % predictive interval	Upper 95 % predictive interval
1	4.5	1.8	7.2
1.5	7.6	4.7	10.4
2	10.5	7.6	13.5
3	15.9	12.7	19.2
4	20.7	17.2	24.1
6	28.5	24.3	32.7
8	34.9	30.4	39.5
12	45.1	40.1	50.2
16	53.8	48.5	59.1
24	67.9	62.3	73.5
32	78.3	72.0	84.5
48	90.2	83.3	97.1
64	95.4	88.1	100
96	98.6	90.9	100
128	99.1	91.4	100

INSTRUCTIONS FOR USE

Stability and Use: This material is considered to be extremely hygroscopic. This SRM must be stored in the original container in a cool (10 °C to 25 °C), dry (relative humidity ≤60 %) environment away from light and fumes. Based on measurements at NIST on SRM 46h, the properties certified are stable as long as the foil bag remains sealed. The specific surface area of cement changes on exposure to the moisture in air. Therefore, this cement should be used immediately after opening the outer foil bag.

Allow the sealed foil bag to equilibrate to testing temperature before opening. To open the pouch, cut off the end with scissors. Fluff the cement in accordance with ASTM Standard C204, Section 4.3, allow the cement to settle for 2 minutes, and then perform the measurement.

REFERENCES

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[2] Ferraris C.F.; Peltz M.; Toman B.; *Certification of Standard Reference Material® 46h: Fineness of cement - Addition of Blaine and Particle Size Distribution*; NIST SP260-190, National Institute of Standards and Technology, U.S. Department of Commerce: Gaithersburg, MD (2018); available at https://www.nist.gov/publications/certification-standard-reference-materialr-46h-fineness-cement-addition-blaine-and (accessed May 2023).

[3] Ferraris, C.F.; Guthrie, W.; Ivelisse Avilés, A.; Peltz, M.; Haupt, R.; MacDonald, B.S.; *Certification of SRM 114q; Part II (Particle Size distribution)*; NIST SP260-166, National Institute of Standards and Technology, U.S. Department of Commerce: Gaithersburg, MD (2006); available at https://nvlpubs.nist.gov/nistpubs/Legacy/SP/nistspecialpublication260-166.pdf (accessed May 2023).

[4] Ferraris, C.F.; Guthrie, W.; Peltz, M.; Haupt, R.; *Certification of SRM 46h*; NIST SP260-169, National Institute of Standards and Technology, U.S. Department of Commerce: Gaithersburg, MD (2008); available at https://www.nist.gov/system/files/documents/srm/SP260-169.pdf (accessed May 2023).

[5] ASTM International; CCLR, West Conshohocken, PA, available at http://www.ccrl.us (accessed May 2023)

[6] Ferraris, C.F.; Avilés A.I.; Guthrie W.; Haupt, R.; *Certification of SRM 114q*; *Phase I*, NIST SP260-161, National Institute of Standards and Technology, U.S. Department of Commerce: Gaithersburg, MD (2005); available at https://www.nist.gov/system/files/documents/srm/SP260-161.pdf (accessed May 2023).

If you use this SRM in published work, please reference:

Ferraris CF, Guthrie W, Peltz M, Haupt R (2008) Certification of SRM 46h. (National Institute of Standards and Technology, Gaithersburg, MD), NIST Special Publication (SP) 260-169. https://doi.org/10.6028/NIST.SP.260-169

Ferraris CF, Peltz M, Toman B (2018) Certification of Standard Reference Material® 46h: Fineness of cement - Addition of Blaine and Particle Size Distribution. (National Institute of Standards and Technology, Gaithersburg, MD), NIST Special Publication (SP) 260-190. https://doi.org/10.6028/NIST.SP.260-190

Certificate Revision History: 16 May 2023 (Change of period of validity, editorial changes); 09 September 2022 (Change of period of validity; updated format; updated storage conditions; editorial changes); 13 June 2018 (Addition of Blaine and particle size distribution certified values; editorial changes); 30 August 2016 (Change of expiration date; editorial changes); 30 May 2008 (Changed ASTM Standard from 3.4 to 4.3); 28 April 2008 (Original certificate issue date).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis 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.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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APPENDIX A

Values of Potential Interest: Values of potential use to customers for SRM 46h are provided below. **These values cannot be used for calibration purposes.** Chemical composition determined by ASTM Standard Test Method C114-02 is shown in Table A1. The analysis of this cement (CCRL Portland Cement Proficiency Sample No. 164) was performed by 70 to 170 laboratories; the number of participating laboratories depends on the value measured. Table A2 provides the calculation of cement compounds according to ASTM C 150-02.

Table A1. Values of Potential Interest for Chemical Composition

Compound	Mass Fraction (%)	Compound	Mass Fraction (%)
CaO	63.9	K_2O	0.68
SiO_2	20.6	TiO_2	0.30
$A1_2O_3$	4.9	P_2O_5	0.21
Fe_2O_3	2.8	Na_2O	0.19
SO_3	2.9	MgO	1.9
Loss on Ignition	1.5		

Table A2. Values of Potential Interest for Cement Compounds (Calculation from Table 2)

Compound		Mass Fraction (%)
C_3S	(tricalcium silicate)	59
C_2S	(dicalcium silicate)	15
C_3A	(tricalcium aluminate)	8
C_4AF	(tetracalcium alumino-ferrite)	8

Values of Potential Interest -Density of cement: The density was measured twice at NIST: the results were 3.27 g/cm^3 and 3.30 g/cm^3.

Additional Information

Material Selection and Packaging: The desired properties were selected for SRM 46h to be coarser than SRM 114q so as to obtain a 45 μm residue of about 6 % to 10 %. The Cement and Concrete Reference Laboratory (CCRL) and NIST identified a plant with suitable cement for this SRM. The material selected was Type I according to the ASTM C 150 Standard Classification. The material was collected for shipment to NIST directly from the finish mill process stream into bags. Upon arrival at NIST, the cement was blended in a V-blender (1.68 m^3) and then transferred to 208 L (55 gallon) drums lined with 0.015 cm (6 mil) polyethylene liners to minimize hydration of the cement in storage prior to preparation and packaging. Over the next two days, the cement from each drum was sealed in foil bags, each containing about 16 kg of cement. The foil bags were stored in a climate-controlled area. The contents of each bag were subsequently packaged into vials. The vials were then capped and packaged in boxes of about 500 vials per box. The boxes were sequentially labeled from 1 to 120. About 5 boxes were filled per day. Nearly 60 000 glass vials, each containing approximately 5 g of cement, were produced. Each vial was then individually sealed in a foil bag. Vials were selected from the lot by random sampling [4] for certification analyses at NIST for the Sieve residue. The remaining vials were packaged into SRM unit boxes of 20 vials each. In 2017, randomly selected vials were shipped to the participating laboratories for measurements to determine the Blaine and the PSD.

Homogeneity Assessment and Certification Analyses: During the certification process using the same data obtained from the sieve residue analysis, material homogeneity was assessed and no evidence of significant heterogeneity was found.

Certification analyses according to ASTM Standard Test Method C 430-96 for the 45 μm sieve residue were performed at NIST on 120 samples from 60 vials of cement. Analysis and details are reported in reference 4.

Certification analyses for specific surface areas using ASTM Standard Test Method C 204-00 was performed on two samples at each of the participating laboratories. SRM 114q Portland Cement Fineness Standard was used for calibration. Raw data were submitted by each laboratory to NIST for tabulation and calculation of surface areas, which for the Blaine test, assumed a density of 3.15 g/cm^3. Analysis and details are reported in reference 2.

Laboratories performing certification analyses are members of the Cement and Concrete Reference Laboratory (CCRL) [5] proficiency program. The full list is provided in the report describing the details of the certification process [2,6].

Preparation of the material and the coordination of the technical measurements leading to certification were performed by C. Ferraris of the NIST Materials and Structural Systems Division.

Statistical consultation on measurement design and analysis of the certification data was performed by B. Toman 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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