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

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Standard Reference Material® 114r

Portland Cement Fineness Standard

CERTIFICATE OF ANALYSIS

Purpose: This Standard Reference Material (SRM) is intended for use in calibrating fineness testing equipment according to ASTM Standard Methods.

Description: A unit of SRM 114r consists of 20 glass vials with plastic caps containing powdered cement. Each vial is sealed in a foil bag and contains approximately 5 g of cement.

Certified Values: 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 taken into account [1]. The certified values for specific surface area and sieve residue are given in Table 1. The certified values for the surface area are the mean of results from analyses performed by cooperating laboratories. The certified value for sieve residue was calculated from a quadratic fit of NIST data using three sieves having openings ranging from 38 μm to 56 μm.

The expanded uncertainties of the certified values for specific surface area were calculated according to the NIST uncertainty policy described in the NIST Technical Note 1297 [2] at the 95 % confidence level. The uncertainties include measurement variability within and between laboratories. The surface area uncertainties also include material variability and the uncertainty of the surface area values for the SRM 46h *Portland Cement Fineness Standard*, which was used as the calibrant for this material. The expanded uncertainty for the sieve residue was computed using a Bayesian analysis at the 95 % probability level. The expanded uncertainty accounts for the variability of random measurement effects, sieve calibrations, and material inhomogeneity.

Table l. Certified Values

Measurand	ASTM Method	Certified Value and Expanded Uncertainty[(a)]
Specific Surface Area (Blaine)	C 204-18[(b)]	3932 cm^2/g ± 17 cm^2/g (393.2 m^2/kg ± 1.7 m^2/kg)
Sieve Residue (45 μm residue)	C 430-17[(c)]	5.97 % ± 0.48 %

[(a)] The measurands are the specific surface area and the sieve residue (45 μm residue). The certified values are metrologically traceable to the International System of Units (SI) unit for length and mass; expressed as square centimeters per gram (square meters per kilogram) and percentage, respectively.
[(b)] Standard Test Method for Fineness of Portland Cement by Air Permeability Apparatus [Blaine].
[(c)] Standard Test Method for Fineness of Hydraulic Cement by the 45 μm (No. 325) Sieve.

Non-Certified Values: Non-certified values for particle size distribution are provided in Appendix A, Table A1.

Additional Information: Additional information is available in Appendix B.

Period of Validity: The certified values delivered by **SRM 114r** are valid within the measurement uncertainty specified until **31 December 2030**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Jason Averill, Chief
Materials and Structural Systems Division

Steven J. Choquette, Director
Office of Reference Materials

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).

Safety: SRM 114r is intended for research use. Consult the Safety Data Sheet (SDS) for hazard information.

Stability and Use: This material is hygroscopic; however, the properties remain stable as long as the foil bag remains sealed. The cement should be used immediately after opening the 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 5.3 and then perform the measurement.

Material Selection and Packaging: The ideal properties were provided by ASTM Subcommittee C01.25, Fineness and are similar as those for the previous issues of SRM 114. The Cement and Concrete Reference Laboratory (CCRL) and NIST identified a plant with suitable material and obtained 1000 kg of cement. The entire lot of cement was blended in a V-blender by CCRL 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. The contents of each drum were packaged into vials each containing approximately 5 g of cement and packaged in boxes of about 500. Each vial was then individually sealed in a foil bag. Vials were selected from the lot by stratified random sampling [3] for both homogeneity and certification analyses. Selected vials were shipped to the participating laboratories for Blaine and laser particle size distribution measurements. The remaining vials were packaged into SRM unit boxes of 20 vials each.

Homogeneity Assessment and Certification Analyses: Homogeneity testing of the material was performed on randomly-selected samples. The data received from the inter-laboratory participants were also checked for laboratory-to-laboratory variability, box-to-box variability, and vial-to-vial variability.

Certification analyses for specific surface areas using ASTM Standard Test Methods C 204 were performed on two samples at each of the participating laboratories using SRM 46h *Portland Cement Fineness Standard* for calibration. Raw data were submitted by each laboratory to NIST for tabulation and calculation of surface areas, which for the Blaine test, used a density of 3.15 g/cm^3. Four density measurements performed at NIST yielded a mean density of 3.147 g/cm^3.

Certification analyses according to ASTM Standard Test Method C430 for the 45 mm sieve residue were performed at NIST on 36 vials of cement. Three sieves with nominal openings of 38 μm, 45 μm, and 54 μm were directly calibrated ASTM E11-20^d for use in reference material certification. Interpolation was then used to obtain the value at 45 μm.

REFERENCES

[1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication (NIST SP) 260-136, 2021 edition; U.S. Government Printing Office: Washington, DC (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Jul 2022).

[2] 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 https://www.nist.gov/pml/special-publication-811 (accessed Jul 2022).

[3] Stutzman, P.; Votri, E.; Toman, B.; *Certification of Standard Reference Material® 114r Portland Cement Finess*; NIST SP260-226, National Institute of Standards and Technology, U.S. Department of Commerce: Gaithersburg, MD (2022); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-226.pdf (accessed Jul 2022)

[4] AASHTO T 353; *Standard Method of Test for Particle Size Analysis of Hydraulic Cement and Related Materials by Light Scattering*; American Association for State Highway Transportation Officials (01 January 2014).

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

Stutzman P, Votri E, Toman B (2022) Certification of SRM 114r, Particle Size Distribution; (National Institute of Standards and Technology, Gaithersburg, MD), NIST Special Publication (SP) 260-226. https://doi.org/10.6028/NIST.SP.260-226

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

Non-Certified Values: Non-certified values are suitable for use in method development, method harmonization, and process control but do not provide metrological traceability to the International System of Units (SI) or other higher order reference system. Non-certified values are provided below.

Particle Size Distribution (PSD): The SRM 114r particle size distribution (PSD) was determined using laser diffraction (LD) techniques provided through an inter-laboratory study. Two LD methods were included in the tests: LD-W, in which the powder was dispersed in a liquid medium (wet) and LD-D in which the powder was measured in a dry dispersed state as an aerosol (dry). Results were combined to calculate a mean PSD for LD and tabulated in Table A1. A complete discussion of the test procedures and statistical analysis is provided in reference 3 and 4.

Table A1. The Particle Size Distribution of SRM 114r Using LD Methodology (either wet or dry dispersion) [4]

Particle Size [µm]	Mean Cumulative Volume Fraction [%]	Standard uncertainty of cumulative %	95 % Lower Expanded Uncertainty Bound	95 % Upper Expanded Uncertainty Bound
1	5.97	1.12	3.59	7.92
1.5	9.02	1.27	6.41	11.32
2	11.82	1.32	9.06	14.26
3	16.82	1.37	13.97	19.36
4	21.18	1.43	18.25	23.88
6	28.42	1.66	25.12	31.54
8	34.49	1.83	30.94	38.04
12	44.54	2.10	40.42	48.6
16	52.74	2.16	48.55	56.9
24	66.78	2.18	62.52	70.97
32	77.51	2.24	73.11	81.89
48	91.29	2.74	85.94	96.66
64	96.84	2.87	91.23	100
96	99.54	2.91	93.88	100
128	100	2.92	94.42	100

NOTES: The purpose of a reference PSD based on an easily accessible reference material is to verify the efficacy of an instrument and the procedure being used. If the data are found to be statistically different, the operator should check the performance of the device, the parameters used (such as the refractive indices) or the procedure (dispersion and duration of the measurement). For more details on these measurements refer to the references 3 through 5 provided above.

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Certificate of Analysis 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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APPENDIX B

Coordination of the preparation of the material, and the technical measurements leading to certification were performed by P. Stutzman and E. Votri 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.

M. Faijt of Buzzi Unicem assisted with material acquisition.

S. Makens, Chair of ASTM C01.25 and K. Niedzielski of CCRL helped identify ideal material properties and prospective suppliers.

M. Cronise of the NIST Office of Reference Materials coordinated packaging. T. Doiron of the NIST Sensor Science Division provided sieve calibrations. R. Eason of the NIST Materials and Structural Systems Division performed density measurements.

Support aspects involved in the preparation of this SRM were coordinated through the NIST Office of Reference Materials.

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