National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 114q

Portland Cement Fineness Standard

This Standard Reference Material (SRM) is intended for use in calibrating fineness testing equipment according to ASTM Standard Methods. A unit of SRM 114q consists of 20 glass vials with plastic caps containing powdered cement (each vial is contained in a sealed foil bag). Each vial contains approximately 5 g of cement.

Certified Values and Uncertainties: 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. The certified values for specific surface area and sieve residue are given in Table 1. The certified values for particle size distribution are given in Table 4. 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 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 [1], and are 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The surface area uncertainties also include material variability and the uncertainty of the surface area values for the superseded SRM 114p *Portland Cement Fineness Standard*, which was used as the calibrant for this material.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 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[a]
Specific Surface Area (Blaine)	C 204-96a[b]	3818 cm^2/g ± 78 cm^2/g (381.8 m^2/kg ± 7.8 m^2/kg)
Specific Surface Area (Wagner)	C 115-96a[c]	2183 cm^2/g ± 160 cm^2/g (218 m^2/kg ± 16 m^2/kg)
Sieve Residue (45 μm residue)	C 430-96[d]	0.79 % ± 0.19 %

[a] The measurands are the specific surface area and the sieve residue (45 μm residue). The certified values are metrologically traceable to the 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 Portland Cement by the Turbidimeter [Wagner].
[d] Standard Test Method for Fineness of Hydraulic Cement by the 45 μm (No. 325) Sieve.

Expiration of Certification: The certification of **SRM 114q** is valid, within the measurement uncertainty specified, until **31 December 2021**, provided the SRM is stored and 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 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 certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Jason Averill, Chief
Materials and Structural Systems Division

Gaithersburg, MD 20899
Certificate Issue Date: 30 August 2016
Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

Coordination of the preparation of the material, and 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 W.F. Guthrie A.I. Avilés formerly of the NIST Statistical Engineering Division. Additional statistical consultation was provided 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.

INSTRUCTIONS FOR USE

Stability and Use: This material is considered to be extremely hygroscopic. Based on measurements in 1993 of several earlier renewals of SRM 114, 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 two minutes, and then perform the measurement.

Material Selection and Packaging: The desired properties were determined to be generally the same as those selected for the previous issues of SRM 114; however, in order to better represent current cements, the material selected for SRM 114q consists of a finer particle size distribution than previously issued. The Cement and Concrete Reference Laboratory (CCRL) and NIST identified a plant with suitable cement: Lehigh Cement Company (Union Bridge, Maryland)[(1)] donated 1300 kg of appropriate cement for this SRM. The material selected was Type I according to the ASTM C 150 Standard Classification and had a mass fraction of less than 8 % tricalcium aluminate (C_3A). 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 118. About five boxes were filled per day. Nearly 59 000 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 stratified random sampling [2] for both homogeneity and certification analyses. Selected vials were shipped to the participating laboratories for 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 48 random-selected samples. Measurements of the loss of ignition (LoI) showed no reversible moisture take-up by the cement during packaging. The data received from the round-robin participants were also checked for laboratory-to-laboratory (or day-to-day in the case of sieve residue) variability, box-to-box variability, and vial-to-vial variability. No significant box-to-box or vial-to-vial variability was detected except for the Wagner or sieve residue tests, and therefore it was determined that the samples were homogeneous for the ASTM measurements. Significant vial-to-vial variability was observed using the Blaine test and the certified values reflect this source of uncertainty.

Certification analyses for specific surface areas using ASTM Standard Test Methods C 115-96a and C 204-00 were performed on two samples at each of the participating laboratories. SRM 114p *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. The density was measured twice at NIST: the results were 3.255 g/cm^3 and 3.248 g/cm^3.

Certification analyses according to ASTM Standard Test Method C 430-96 for the 45 μm sieve residue were performed at NIST on 40 samples from 20 vials of cement.

Laboratories performing certification analyses are members of the CCRL (http://www.ccrl.us) proficiency program. The full list is provided in the report describing the details of the certification process [2].

[(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.

Information Values: NIST information values are considered to be of interest to the SRM user, but are not certified because insufficient information is available to assess adequately the uncertainty associated with the values or only a limited number of analyses were performed. Information values cannot be used to stablish metrological traceability. Information values for SRM 114q are given in Tables 2 and 3. Table 2 provides the approximate chemical composition determined by ASTM Standard Test Method C114-02. The analysis of this cement (CCRL Portland Cement Proficiency Sample No. 150) was performed by 70 to 170 laboratories; the number of participating laboratories depends on the value measured.

Table 2. Information Values for Chemical Composition

Compound	Mass Fraction (%)	Compound	Mass Fraction (%)
CaO	64.0	K_2O	0.70
SiO_2	20.7	TiO_2	0.30
$A1_2O_3$	4.7	P_2O_5	0.12
Fe_2O_3	3.2	Na_2O	0.07
SO_3	2.4	MgO	2.2
Loss on Ignition	1.67		

Table 3 provides the calculation of cement compounds according to ASTM C 150-02.

Table 3. Information Values for Cement Compounds (Calculation from Table 2)

	Compound	Mass Fraction (%)
C_3S	(tricalcium silicate)	60
C_2S	(dicalcium silicate)	14
C_3A	(tricalcium aluminate)	7
C_4AF	(tetracalcium alumino-ferrite)	10

Particle Size Distribution (PSD): The SRM 114q particle size distribution (PSD) was determined using laser diffraction (LD) techniques in a round-robin evaluation. 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). The values given in this addendum were obtained through a round-robin inter-laboratory study by volunteer participants from companies participating in the CCRL certification program. Because the results obtained from the two methods were not found to be statistically different, the results were combined to calculate a mean PSD for LD, shown graphically in Figure 1 and tabulated in Table 4. A complete discussion of the test procedures and statistical analysis is provided in reference 3.

This particle size distribution could be used as a reference to validate methodology and instrument operation as described in reference 3 and notes below.

The parameters used to develop the PSD were:

- For LD-D and LD-W: the complex refractive index for the cement used had a real part of 1.7 and an imaginary part of 1.0
- For LD-W: IPA was used as the medium and the refractive index (real) used for IPA was 1.39; the imaginary part was zero.

Table 4. The Particle Size Distribution of SRM 114q Using LD Methodology (either wet or dry dispersion) [3]

Particle Size (μm)	1.0	1.5	2.0	3.0	4.0	6.0	8.0	12.0	16.0	24.0	32.0	48.0	64.0	96.0	128.0
Mean Cumulative Volume Fraction (%)	5.1	8	11.2	16.3	21	29.6	37.6	51	62.8	80.8	91.2	98.4	99.7	99.9	99.9
95 % Lower Expanded Uncertainty Bound	3.8	6.2	9.1	13.9	18.1	26.1	33.8	46.8	57.9	78	89.4	97.4	98.9	99.1	99.1
95 % Upper Expanded Uncertainty Bound	6.5	9.8	13.3	18.8	24	33.3	41.5	55.6	68	83.6	92.9	99.4	100.0	100.0	100.0

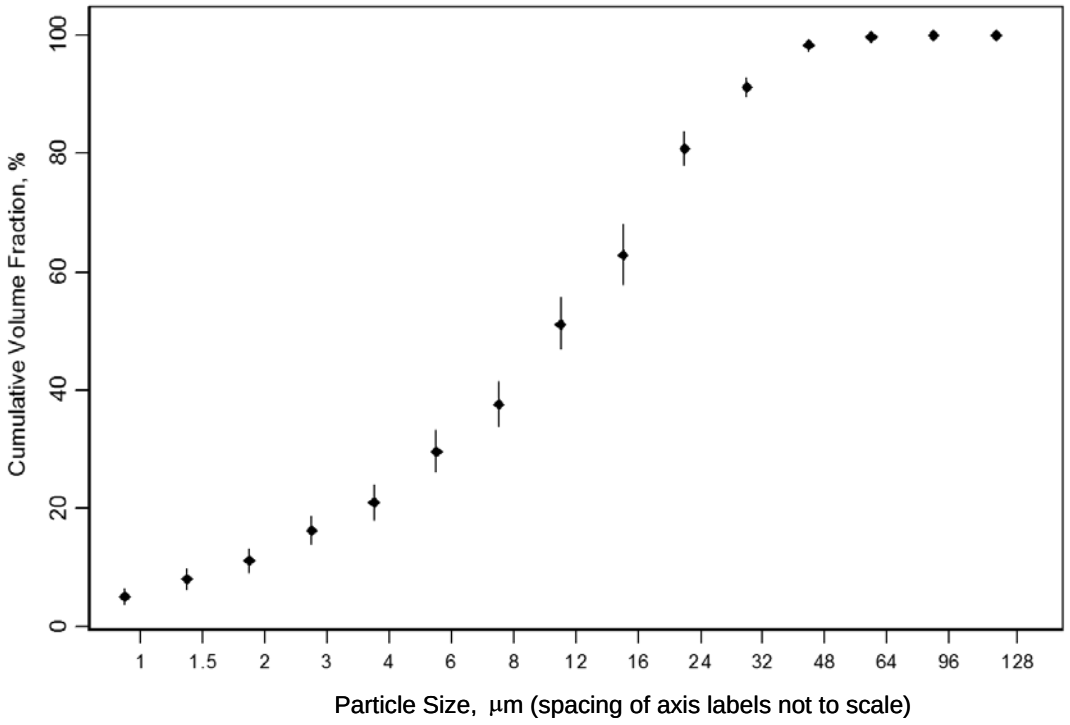

Figure 1. Graphical depiction of the particle size distribution of SRM 114q using LD (either wet or dry dispersion) [3].

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.

The approach suggested to compare the data obtained with the SRM 114q value is presented in the next section on "Conformity Determination for PSD". If the data are found to be statistically different from the SRM 114q, 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 reference 3.

Conformity Determination for PSD: The operator should start by measuring the SRM 114q at least three times using his own procedure and an instrument based on LD. These results can then be used to determine conformity at the following levels:

- General agreement of the results with other laboratories: Because there is no standard test method, a relatively large amount of between-lab variation is allowed in this situation
- Agreement to within-laboratory reproducibility: This level of agreement indicates that the user's results will not differ from the certified value of SRM 114q by more than expected based on the within-lab reproducibility of a typical laboratory.

Table 5. Simultaneous Expanded Uncertainties for Conformance Assessment with SRM 114q by LD Based Method [3]

Particle Size (µm)	Cumulative Volume Fraction (CVF) of SRM 114q (%)	Simultaneous 95 % Expanded Uncertainties for the Difference Between a Typical Lab and the Certified Value of SRM 114q (%)	Simultaneous 95 % Expanded Uncertainties for the Difference Between a Typical Lab and the Certified Value of SRM 114q Including Between Lab Variation (%)
1	5.1	2.0	7.6
1.5	8.0	2.8	9.9
2	11.2	3.2	11.8
3	16.3	3.8	13.8
4	21.0	4.6	15.7
6	29.6	5.5	18.2
8	37.6	5.9	19.0
12	51.0	6.7	19.7
16	62.8	7.6	20.2
24	80.8	4.4	15.2
32	91.2	2.8	10.5
48	98.4	1.6	4.2
64	99.7	1.2	2.0
96	99.9	1.2	1.8
128	99.9	1.2	1.8

As stated above, these expanded uncertainty intervals allow for two levels of conformance assessment. The first level indicates agreement with the LD results typically obtained by laboratories that participated in the interlaboratory study used for certification of the particle size distribution of SRM 114q. Because there is no currently agreed-upon standard test method for obtaining particle size distributions using laser diffraction (LD), a relatively large amount of between-lab variation is allowed for in these uncertainties, given in column 4 of Table 5.

For laboratories that would like to assure tighter agreement with SRM 114q, the uncertainties in column 3 of Table 5 can be used instead. This level of agreement indicates that the LD user's results will not typically differ from the certified value of SRM 114q by more than the within-lab reproducibility of a typical laboratory.

To use these uncertainties to assess agreement with other laboratories, the user should compute the absolute difference in cumulative volume fraction between his or her LD results and the certified values for SRM 114q for each particle size. These differences should then be compared to the appropriate expanded uncertainties in columns 3 or 4 of Table 5 to determine conformance. If the observed absolute difference between the LD user's results and the certified values for SRM 114q is always less than the corresponding expanded uncertainty, then the user can conclude that his or her results are in agreement with other laboratories with a confidence level of approximately 95 %. If, on the other hand, one or more of the observed absolute differences is larger than the corresponding expanded uncertainty, this is evidence that the user's results are not in agreement with the results of other laboratories and that changes to the measurement procedures are needed.

For more details on how the values were obtained, refer to reference 3.

REFERENCES

[1] 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 (1994); available at http://www.nist.gov/customcf/get_pdf.cfm?pub_id=200349 (accessed Aug 2016).

[2] 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 http://www.nist.gov/srm/publications.cfm (accessed Aug 2016).

[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 http://www.nist.gov/srm/publications.cfm (accessed Aug 2016).

Certificate Revision History: 30 August 2016 (Change of expiration date; editorial changes); 15 September 2008 (Changed the ASTM Standard from 3.4 to 4.3); 21 February 2007 (This technical revision includes the addition of the sections “Particle Size Distribution” and “Conformity of PSD”); 24 March 2005 (This technical revision corrects the certified values and expanded uncertainties for the measurand); 23 March 2005 (Original certificate 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 http://www.nist.gov/srm.