National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 114p

Portland Cement Fineness Standard

This Standard Reference Material (SRM) is intended for use in calibrating fineness testing equipment according to ASTM Standard Methods. The SRM unit consists of approximately 200 g of powdered cement, packaged in 20 sealed laminated barrier film pouches, each containing approximately 10 g of cement.

Table I. Certified Values

Measurement	ASTM Method	Result
Specific Surface Area	C 115-93[1]	2086 ± 45 cm^2/g (208.6 ± 4.5 m^2/kg)
Specific Surface Area	C 204-92[2]	3774 ± 58 cm^2/g (377.4 ± 5.8 m^2/kg)
Sieve Residue	C 430-92[3]	8.24 ± 0.37 wt %

[1]Standard Test Method for Fineness of Portland Cement by the Turbidirneter [Wagner].

[2]Standard Test Method for Fineness of Portland Cement by Air Permeability Apparatus [Elaine].

[3]Standard Test Method for Fineness of Hydraulic Cement by the 45 μm (No. 325) Sieve.

The certified values for surface area are the means of results from certification analyses performed by cooperating laboratories. The certified value for sieve residue was predicted from the straight line fit of residue data measured at one laboratory using seven sieves having openings ranging from 42 μm to 46 μm.

The expanded uncertainties of the certified values were computed according to the NIST uncertainty policy, as described in NIST Technical Note 1297 [1], and are at the 95 % level of confidence. 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 SRM 114n, which was used as the calibrant for this material.

The preparation of the material and the coordination of the technical measurements leading to certification were performed under the direction of H.M. Kanare, Construction Technology Laboratories, Inc. (CTL), a subsidiary of the Portland Cement Association, Skokie, IL.

Statistical consultation on measurement design and analysis of the certification data was performed by S.B. Schiller, of the NIST Statistical Engineering Division.

The technical and 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 of the NIST Measurement Services Division.

Sivaraj Shyam-Sunder, Chief
Materials and Construction Research Division

Gaithersburg, MD 20899
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See Certificate Revision History on Page 3

John Rumble, Jr., Chief
Measurement Services Division

INSTRUCTIONS FOR USE

Stability and Use: This material is considered to be extremely moisture-sensitive. Based on measurements in 1993 of several earlier renewals of SRM 114, the properties certified are stable as long as the barrier film pouch remains intact. The specific surface area of cement changes on exposure to the moisture in air. Therefore, this cement should be used as soon as possible after opening a pouch.

To open, allow the sealed laminated pouch to equilibrate to testing temperature. Hold the pouch at one end and gently tap to let the cement settle to the bottom before cutting off the end of the pouch with scissors. Immediately after opening, transfer the cement to a clean four-ounce glass screw-top jar. Fluff the cement in accordance with C204, Section 3.4 and allow the cement to settle for 2 min, then measure without delay.

Material Selection and Packaging: ASTM Committee CO1.25 identified the desired properties for SRM 114p and CTL identified cement plants from which a candidate cement could be obtained from mill test reports in their possession. The Calmat Cement Plant, Mohave, CA donated 1800 kg of appropriate cement for this SRM. Material was collected for shipment to CTL directly from the finish mill process stream into eight 55 gallon barrels lined with 0.015 cm (6 mil) polyethylene liners to minimize hydration of the cement in transit and in storage prior to preparation and packaging. The material selected was Type II according to the ASTM C 150 Standard Classification, and had less than 8 wt. % tricalcium aluminate (C_3A).

At CTL, the cement was blended and cross-blended in an Abbe ribbon blender for a total of 3 h in batches of 2 to 3 barrels and then sieved to remove particles greater than 600 μm (#30 sieve) as well as to disperse the cement agglomerates. After sieving, the cement was blended for another 3 h to assure homogenization. Finally the cement was packaged into 125,000 ten g laminated barrier film pouches, from which samples were randomly selected for both homogeneity and certification analyses.

Homogeneity Assessment and Certification Analyses: Homogeneity testing of the material was performed on 48 randomly selected samples, following ASTM C 204-92. The material variability detected is a component of the combined overall uncertainty of each of the certified surface area values.

Certification analyses for specific surface areas using ASTM Standard Test Methods C 115-93 and C 204-92 were performed on ten samples at each of the participating laboratories. SRM 114n was used for calibration. Raw data was submitted by each laboratory to CTL for tabulation and calculation of surface areas, which for the Blaine test assumed a density of 3.15 g/cm^3. The measured density of 114p is also 3.15 g/cm^3, while that for SRM 114n is 3.20 g/cm^3.

Certification analyses according to ASTM Standard Test Method C 430-92 for the 45 μm sieve residue were performed at CTL by two operators, each of whom made measurements on 48 samples.

Laboratories performing certification analyses include:

R. Peck: Blue Circle Atlantic; Ravena, NY
P. Hawkins: Calmat Co.; Colton, CA
E. Shkolnik: J. Berant, CTL, Inc.; Skokie, IL, L. Hills
D. Levonian: Davenport Cement Co.; Davenport, IA
J. Bangeman: Dragon Products Co.; Thomaston, ME
M. Amoury: Gifford-Hill Cement Co.-Texas; Midlothian, TX
C. Hardin: Kaiser Cement Corp.; Permanente, CA
A.D. McCandless: Medusa Cement Co.; Charlevoix, MI
W. Klemm: Southwestern Portland Cement Co.; Victorville, CA

Additional Uncertified Results on the Fineness and Chemical Composition of SRM 114p

1. Air permeability measurements using Lea and Nurse apparatus were made on SRM 114p by CTL and by the Cement and Concrete Association, Wexham Springs, UK. These measurements, leading to a specific surface area of 3923 cm^2/g were made according to EN 196, Part 6, Annex NB to identify whether or not the European (Lea and Nurse) and the American (Blaine) standard methods provide compatible results. The data suggests a possible bias between the two methods.

2. Residue remaining on a 30 μm sieve cannot be reported, because the material binds in the sieve to such a degree that the residue cannot be dislodged for accurate weighing of the residue.

3. The approximate chemical composition has been determined by X-ray fluorescence spectrometry to provide additional information on this cement. The composition, which is not certified but provided for information only, is:

	Percent by Wt.		Percent by Wt.
CaO	63.6	K_2O	0.51
SiO_2	21.6	TiO_2	0.23
Al_2O_3	4.2	P_2O_5	0.19
Fe_2O_3 (total)	3.0	Na_2O	0.19
SO_3	2.7	SrO	0.14
MgO	2.4	loss on ignition	1.21

4. Calculation of cement compounds from this chemistry, according to ASTM C 150-92, gives:

Compound		Percent by Wt.
C_3S	(tricalcium silicate)	53
C_2S	(dicalcium silicate)	23
C_3A	(tricalcium aluminate)	7
C_4AF	(tetracalcium alumino-ferrite)	9

REFERENCE

[1] Taylor, B.N.; Kuyatt, C.E.; NIST Technical Note 1297, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results* (January 1993).

Certificate Revision History: 17 June 2003 (Addendum with particle size distribution added); 19 May 1994 (Unit size corrected); 6 April 1994 (Original certificate date).

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.

Addendum

Standard Reference Material® 114p

Portland Cement Fineness Standard

ASTM Committee C01.25.01 Particle Size Distribution Round Robin Results and Correction Procedure: The SRM 114p particle size distribution (PSD) was determined using laser diffraction (LAS) techniques in a round-robin evaluation. Two LAS methods were included in the tests: LAS-W, in which the powder was dispersed in a liquid medium (wet) and LAS-D in which the powder was measured in a dry dispersed state as an aerosol (dry). Two round-robins [1,2] were sponsored by ASTM Committee C01.25.01. The first round-robin was preliminary in nature, and was used to refine test procedures and to assess the overall state-of-the-art for cement industry powder measurements. The second round-robin included a total of 38 participants using LAS techniques: 25 participants used LAS-W and 13 participants used LAS-D. Data from this second round-robin was analyzed and used to calculate mean PSDs for LAS-W and LAS-D, and these mean distributions are shown graphically in Figure 1 and in tabulated form in Tables 2 and 3. A complete discussion of the test procedures and statistical analysis is provided in reference 2. The particle sized distribution round-robin results and correction procedure provided in this addendum are for information purposes only.

These curves could be used as a reference to "calibrate" measurement results as described in reference 2 and below, or to validate methodology and instrument operation.

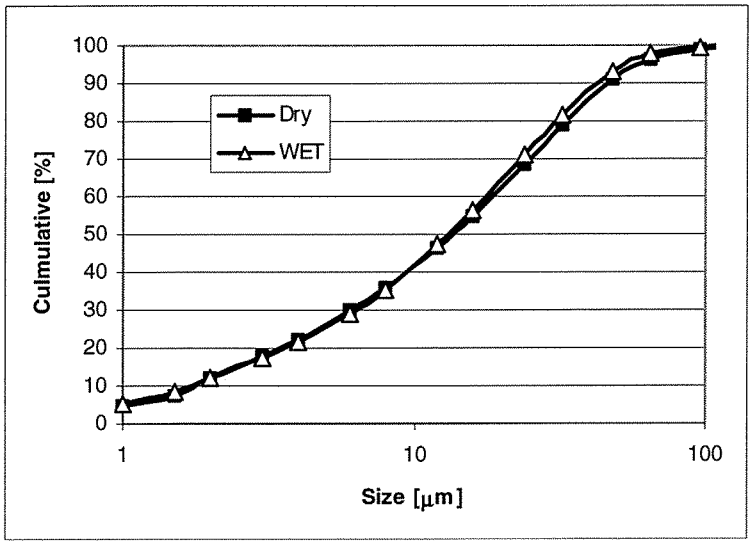

Figure 1. Graphical comparison between distributions calculated from each LAS method separately (LAS-W, Table 2 and LAS-D, Table 3). For clarity, standard uncertainties are not shown, but can be found in the corresponding tables.

Table 2. Statistical Results for the LAS-W based on the Bootstrap (data-based simulation) Method [1,2]

Size [μm]	1	1.5	2	3	4	6	8	12	16	24	32	48	64	96	128
Mean	5.4	8.7	12.2	17.5	21.8	29.1	35.5	47.2	56.2	71.0	81.7	93.4	97.6	99.6	99.9
Low	4.2	7.2	10.9	16.2	20.4	27.7	34.1	45.4	54.2	69.5	80.3	92.5	97.1	99.3	99.8
High	6.7	10.0	13.5	19.0	23.2	30.6	37.0	49.1	57.9	72.6	83.1	94.2	98.1	99.8	100.0

Table 3. Statistical Results for the LAS-D based on the Bootstrap (data-based simulation) Method [1,2]

Size [μm]	1	1.5	2	3	4	6	8	12	16	24	32	48	64	96	128
Mean	4.7	7.3	12.1	17.7	22.3	29.9	35.9	46.4	54.6	68.4	78.7	91.1	96.3	99.1	99.5
Low	3.3	5.4	10.6	16.1	20.3	27.8	33.9	44.4	52.8	66.9	77.3	89.9	95.3	98.3	98.8
High	6.1	9.5	13.4	19.3	24.2	32.0	38.1	48.4	56.7	70.1	80.2	92.2	97.3	99.6	100.0

Correction Procedure

The purpose of a reference PSD based on an easily accessible reference material is twofold: 1) to verify the efficacy of the instrument or method being used, and 2) to calibrate measurement results by applying a set of correction factors. A methodology using the mean PSD curve to calibrate measured data obtained using various instruments would work in the following manner:

- Calculate the correction factor for each size, defined as the ratio between the customer's measured value using SRM 114p and the mean value as shown in Table 2 and Table 3. The selection of the table depends on the method used.
- Multiply all subsequently measured data for unknown samples by this correction factor.

If the target measurement results themselves (not the calibration curve) contain outliers, (i.e., data points that are more than 5 % (absolute value) outside the confidence limits obtained with the statistical bootstrap method), the correction is not sufficient to bring the entire curve within the confidence limits of the calibration curve. On the other hand, if the data set lies completely within the confidence limits defined by the reference curve, the correction factor will reduce the spread of the data. Therefore, the reference material SRM 114p could be used in two ways:

- To check that LAS measurements are within the confidence limit range of the reference. This will allow the operator to determine if sample preparation problems or a malfunctioning instrument should be considered (i.e., as a validation reference).
- To calibrate measurement results by correcting the data obtained using the reference cement, after the method has been validated. (i.e., as a calibration reference).

REFERENCES

[1] Ferraris, C.F.; Hackley V.A.; Aviles A.I.; Buchanan C.E.; *Analysis of the ASTM Round-Robin Test on Particle Size Distribution of Portland Cement: Phase I*; NISTIR 6883 (May 2002). (http://ciks.cbt.nist.gov/~garbocz/nist6883/nistir6883.htm).

[2] Ferraris, C.F.; Hackley V.A.; Aviles A.I.; Buchanan C.E.; *Analysis of the ASTM Round-Robin Test on Particle Size Distribution of Portland Cement: Phase II*; NISTIR 6931 (December 2002).