NIST Special Publication 260-169

Certification of SRM 46H

Chiara F. Ferraris
William Guthrie
Max Peltz
Robin Haupt

NIST National Institute of Standards and Technology • Technology Administration • U.S. Department of Commerce

The National Institute of Standards and Technology was established in 1988 by Congress to "assist industry in the development of technology ... needed to improve product quality, to modernize manufacturing processes, to ensure product reliability ... and to facilitate rapid commercialization ... of products based on new scientific discoveries."

NIST, originally founded as the National Bureau of Standards in 1901, works to strengthen U.S. industry's competitiveness; advance science and engineering; and improve public health, safety, and the environment. One of the agency's basic functions is to develop, maintain, and retain custody of the national standards of measurement, and provide the means and methods for comparing standards used in science, engineering, manufacturing, commerce, industry, and education with the standards adopted or recognized by the Federal Government.

As an agency of the U.S. Commerce Department's Technology Administration, NIST conducts basic and applied research in the physical sciences and engineering, and develops measurement techniques, test methods, standards, and related services. The Institute does generic and precompetitive work on new and advanced technologies. NIST's research facilities are located at Gaithersburg, MD 20899, and at Boulder, CO 80303. Major technical operating units and their principal activities are listed below. For more information visit the NIST Website at http://www.nist.gov, or contact the Public Inquiries Desk, 301-975-NIST.

Office of the Director
- National Quality Program
- International and Academic Affairs

Technology Services
- Standards Services
- Technology Partnerships
- Measurement Services
- Information Services
- Weights and Measures

Advanced Technology Program
- Economic Assessment
- Information Technology and Applications
- Chemistry and Life Sciences
- Electronics and Photonics Technology

Manufacturing Extension Partnership Program
- Regional Programs
- National Programs
- Program Development

Electronics and Electrical Engineering Laboratory
- Microelectronics
- Law Enforcement Standards
- Electricity
- Semiconductor Electronics
- Radio-Frequency Technology[1]
- Electromagnetic Technology[1]
- Optoelectronics[1]
- Magnetic Technology[1]

Materials Science and Engineering Laboratory
- Intelligent Processing of Materials
- Ceramics
- Materials Reliability[1]
- Polymers
- Metallurgy
- NIST Center for Neutron Research

Chemical Science and Technology Laboratory
- Biotechnology
- Process Measurements
- Surface and Microanalysis Science
- Physical and Chemical Properties[2]
- Analytical Chemistry

Physics Laboratory
- Electron and Optical Physics
- Atomic Physics
- Optical Technology
- Ionizing Radiation
- Time and Frequency[1]
- Quantum Physics[1]

Manufacturing Engineering Laboratory
- Precision Engineering
- Manufacturing Metrology
- Intelligent Systems
- Fabrication Technology
- Manufacturing Systems Integration

Building and Fire Research Laboratory
- Applied Economics
- Materials and Construction Research
- Building Environment
- Fire Research

Information Technology Laboratory
- Mathematical and Computational Sciences[2]
- Advanced Network Technologies
- Computer Security
- Information Access
- Convergent Information Systems
- Information Services and Computing
- Software Diagnostics and Conformance Testing
- Statistical Engineering

[1]At Boulder, CO 80303
[2]Some elements at Boulder, CO

NIST Special Publication 260-169

Certification of SRM 46H

Chiara F. Ferraris
Materials and Construction Research Division
Building and Fire Research Laboratory

William Guthrie
Statistical Engineering Division
Information Technology Laboratory

Max Peltz
Materials and Construction Research Division
Building and Fire Research Laboratory

Robin Haupt
Materials and Construction Research Division
Construction Materials Reference Laboratory

National Institute of Standards and Technology
Gaithersburg, MD 28099

January 2008

U.S. Department of Commerce
Carlos M. Gutierrez, Secretary

National Institute of Standards and Technology
James M. Turner, Acting Director

Certain commercial entities, equipment, or materials may be identified in this document in order to describe an experimental procedure or concept adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the entities, materials, or equipment are necessarily the best available for the purpose.

National Institute of Standards and Technology Special Publication 260-XXX
Natl. Inst. Stand. Technol. Spec. Publ. 260-169, 32 pages (January, 2008)
CODEN: NSPUE2

U.S. GOVERNMENT PRINTING OFFICE
WASHINGTON: 2005

For sale by the Superintendent of Documents, U.S. Government Printing Office
Internet: bookstore.gpo.gov — Phone: (202) 512-1800 — Fax: (202) 512-2250
Mail: Stop SSOP, Washington, DC 20402-0001

Abstract

The standard reference material (SRM) for fineness of cement, SRM 114, is an integral part of the calibration material routinely used in the cement industry to qualify cements. Since 1934, NIST has provided SRM 114 for cement fineness testing and it will continue to do so as long as the cement industry requires it. Different lots of SRM 114 are designated by a unique letter suffix to the SRM number, e.g., 114a, 114b, …. ,114q. A certificate that gives the values obtained using the standard tests ASTM C204 (Blaine), C115 (Wagner), and C 430 (45 μm sieve residue) for cement fineness and the particle size distribution by laser diffraction (LD) is included with each lot of the material.

In 2005, NIST issued the SRM 114q and it was immediately clear that the cement selected was too fine to be easily usable in standard test ASTM C 430 (45 μm sieve residue) test method. The value for SRM 114q was about 10 times smaller than that of the SRM 114p. As a result, when the sieve used is only slightly larger than 45 μm, the residue measured can be zero, a value which cannot be used for sieve calibration.

To respond to the industry demand to have an SRM viable for C 430 (45 μm sieve residue), Staff from NIST and the Cement and Concrete Reference Laboratory (CCRL) identified a cement that would have a residue in the range of 6 % to 10 %. This cement was then packaged in small vials, and the values obtained by ASTM C 430 test were determined at NIST using calibrated sieves. This new SRM is labeled SRM 46h.

The purpose of this report is to provide a detailed description of the process used to package and certify SRM 46h. All measurements used for the certifications are provided along with descriptions of the statistical analyses. To accelerate the development of SRM 46h, the chemical analysis, the density, the Blaine value, and the particle size distribution (PSD) are tabulated but should not be considered certified values. This SRM is complementary to SRM 114q and not a replacement.

Acknowledgements

We would like to thank Stephen Small and the staff of the Cement and Concrete Reference Laboratory (CCRL), who were instrumental in providing the samples and helping in the selection of the cement.

The authors would also like to thank some key NIST staff persons without whom this certification could not have been completed: Mark Cronise and Curtis Fales for packaging all the materials; Theodore Doiron of the Manufacturing Engineering Laboratory for the calibration of the sieves used and John Winpigler for some of for information only values reported here.

Table of Contents

List of Tables

List of Figures

1 Introduction

A standard reference material (SRM) is a material that has been well characterized with regard to its chemical composition, or physical properties, or both. The National Institute of Standards and Technology (NIST) provides over 1300 different SRMs to industry and academia. These materials are used in quality assurance programs, for calibration, and to verify the accuracy of experimental procedures. Every NIST SRM is provided with a certificate of analysis that gives the official characterization of the material's properties. In addition, supplementary documentation, such as this report, that describes the development, analysis, and use of SRMs is also often published by NIST to provide the context necessary for effective use of these materials.

There are several SRMs related to cement (http://ts.nist.gov/ts/htdocs/230/232/232.htm). SRM 114 is related to the fineness of cement, as measured by various indirect methods giving its surface area and by passing the material through a fine sieve. This SRM is an integral part of the calibration material routinely used in the cement industry to qualify a cement. Being a powder, the main physical properties of cement are its surface area and particle size distribution (PSD). Since 1934, NIST has provided SRM 114 for cement fineness and it will continue to do so as long as the cement industry requires it. Different lots of SRM 114 are designated by a unique letter suffix to the SRM number. A certificate that gives the values obtained using ASTM C 204 (Blaine) [1], C 115 (Wagner) [2] and C 430 (45 μm residue) [3] is included with each lot of the material.

In 1934, only the Wagner test and the 45 μm residue were listed. In 1944, the Blaine measurement was added to the certificate of SRM 114. In 2003, the PSD measured by laser diffraction was added as an information value, i.e., not certified. The PSD was obtained under the sponsorship of ASTM Task Group C01.25.01 [4, 5, 6]. In 2007, the PSD was given as a certified value for SRM 114q [7].

In 2006, NIST was made aware that the 45μm residue reported for SRM 114q [8] was too low (0.79 % ± 0.19 %) to be of practical use in the correction of measurements as described in C 430. Therefore, to response to industries' demand to have an SRM viable for the C 430 (45 μm sieve residue), NIST identified, with the help of Cement and Concrete Reference Laboratory (CCRL), a cement that would have a 45 μm sieve residue in the range of 6 % to 10 %. This cement was then packaged in small vials, and the certified value for ASTM C 430 was determined at NIST using calibrated sieves. This new SRM is labeled SRM 46h.

The purpose of this report is to provide a detailed description of the process used to package and certify SRM 46H. All measurements used for the certifications are provided along with descriptions of the statistical analyses.

To accelerate the development of SRM 46h, the chemical analysis, the density, the Blaine value, and the particle size distribution (PSD) are given as information items only and are not certified values. This SRM is complementary to SRM 114q and not a replacement.

2 Description of Sieve Residue ASTM C 430

The principle of this test is to measure the residue or amount of cement retained on a calibrated sieve. The sieve was selected as having a 45 μm opening (No. 325[1]). Since a direct certification of sieve openings is impractical and expensive for production-scale work, sieves are calibrated by using a reference material, such as SRM 114 or now SRM 46h. A sieve correction factor is calculated by measuring the SRM on the selected sieve and dividing the result by the certified value of the SRM.

To avoid propagating the SRM 114q uncertainty in the certification of the sieve residue for the SRM 46h material, three sieves with nominal openings of 38 μm, 45 μm, and 56 μm were directly calibrated for use in reference material certification. Interpolation was then used to obtain the value at 45 μm. This is the same method used for the certification of SRM 114q.

[1] Sieve numbers follow the USA definition given in ASTM E11

3 Materials

3.1 Packaging

Upon its arrival at NIST, the cement was blended in a 1.7 m^3 (60 ft^3) V-blender and then transferred to 0.2 m^3 (55 gal) drums lined with 0.15 mm (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 (35.27 lb) of cement. The foil bags were stored, and subsequently packaged as described below into vials, in a climate-controlled area.

Each foil bag was packaged into vials and capped and boxed. Each box contained approximately 500 sealed vials and the boxes were sequentially labeled from 1 to 120. Usually about five boxes were filled per day. The more than 60 000 glass vials produced, each containing approximately 5 g of cement, were subsequently sealed into smaller individual foil bags. Vials were randomly selected (see section 4) for testing sieve residue. After the analysis of the results was completed, the vials were packaged in boxes containing 10 vials each.

3.2 Chemical analysis of the cement

Based on the properties of past lots of SRM 114, CCRL and NIST identified a plant with suitable cement for SRM 46h. The material selected was Type I according to the ASTM C 150 Standard Classification, and had a mass fraction of 8.4 % tricalcium aluminate (C_3A) as defined by ASTM C 150. This requirement was the same as for SRM 114q. Material was collected directly from the finish mill process stream into bags and then shipped to NIST.

The approximate chemical composition has been determined by ASTM Standard Test Method C 114-02 to provide additional information on this cement. Analyses of this cement (CCRL Portland Cement Proficiency Sample No. 164; (www.ccrl.us)) were performed by 70 to 170 laboratories. The composition, which is not certified but is provided for information only, is shown in Table 1.

Calculation of cement compounds from this chemistry, according to ASTM C 150-02 and obtained from the CCRL Portland Cement Proficiency Sample report (www.ccrl.us), are shown in Table 2, again for information only.

Table 1: Chemical composition

	CaO	SiO_2	$A1_2O_3$	Fe_2O_3	SO_3	K_2O	TiO_2	P_2O_5	Na_2O	MgO	loss on ignition
Mass Fraction, %	63.9	20.6	4.9	2.8	2.9	0.7	0.3	0.21	0.19	1.9	1.46

Table 2: Cement compounds according to ASTM C150

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

3.3 Physical characteristics

3.3.1 Density

The density of the cement was measured using the ASTM C 188 [9] method. The medium was kerosene, and a calibrated Le Chatelier flask was used as described in the ASTM test. Seventeen vials were blended together to obtain the material needed for the tests. Two measurements were done: 3.27 g/cm^3 and 3.30 g/cm^3. These values are given for information only and are not certified values.

3.3.2 Surface area by Blaine

The Blaine measurements were performed as described in ASTM C 204 [1]. The calculations were performed using SRM 114q as the reference material and 3.15 g/cm^3 as the density. Three Blaine determinations of three measurements each were done on four samples of cement from different vials. The average values observed for each vial are: 3696 cm^2/g, 3685 cm^2/g, 3737 cm^2/g, and 3762 cm^2/g. This leads to an average specific surface area by the Blaine method of 3720 cm^2/g with a standard uncertainty of ±18 cm^2/g (372.0 m^2/kg ± 1.8 m^2/kg). These values are given for information only and are not certified values. For a Blaine SRM, select SRM 114q (or the current SRM 114).

3.3.3 Particle size distribution by laser diffraction

To determine the particle size distribution (PSD), laser diffraction (LD) measurements were performed on samples from same vials used for ASTM C 204 with the cement dispersed in a liquid (suspension). The medium was isopropanol and the method was as described in [7]. The parameters used in the analysis were:

The refractive index of isopropanol was set to be 1.39
Refractive index of cement: 1.7
Imaginary index of cement: 1 (In Ref. [10], it has been shown that the selection of an imaginary index larger than 0.1 does not affect the results for cement. Therefore, the same results would be obtained for imaginary index values of 0.1 to 1.)

The four specimens were measured 6 times and the distributions obtained for each vial are shown in Figure 1. From these results it is clear that the reproducibility of these PSD determinations for SRM 46h is very good. Some statistics are shown in Table 3.

To simplify the data interpretation the cumulative particle size distribution were reduced to the following sizes: (1, 1.5, 2, 3, 4, 6, 8, 12, 16, 24, 32, 48, 64, 96, 128) μm. This is the same procedure used for SRM 114q [7]. The PSD results for SRM 46h are presented

in Table 4 and Figure 2. The relative standard uncertainties of each mean cumulative volume fraction determined is estimated to be about 0.5 %, but ref. [7] provides a more comprehensive uncertainty analysis of PSD measurements. These values are given for information only and are not certified values. For a PSD SRM, select SRM 114q (or the current issue of SRM 114). SRM 46h is coarser than SRM 114q as desired to ensure that the C 430 test would give larger 45μm sieve residues.

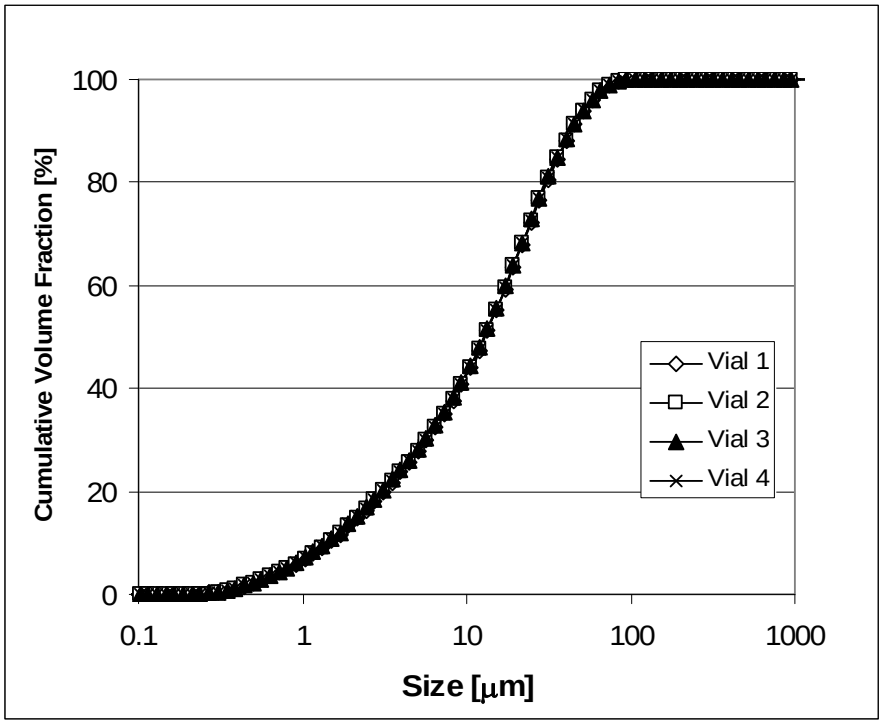

Figure 1: Cumulative volume fraction distributions for 4 different vials of SRM 46h

Table 3: Some statistics on the SRM 46h particle size distribution. For definitions refer to Ref. [7]

	10 % diameter	50 % diameter	90 % diameter	Span
Average [μm]	1.42	12.84	43.06	3.24
Standard deviation	0.02	0.08	0.25	0.01

Table 4: PSD of SRM 46h. The relative standard uncertainties of each mean cumulative volume fraction is estimated to be about 0.5 %. (Values for information only)

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, [%]	6.7	10.6	14.0	19.6	24.1	31.1	37.2	47.9	57.1	71.4	81.3	92.5	97.4	99.9	100.0

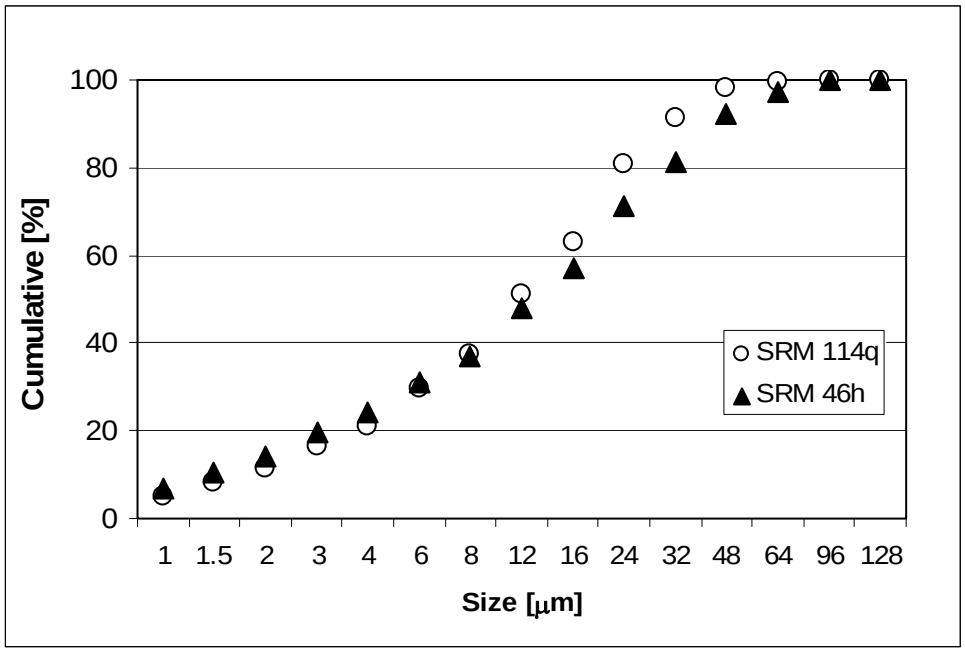

Figure 2: Cumulative particle size distributions of SRM 46h and SRM 114q (Values for information only)

4 Experiment Design and Data Analysis for the Sieve Residue ASTM C 430

To determine the mass fraction of cement that would be retained in a 45 μm sieve, determinations of sieve residue were made using calibrated sieves of different sizes and a quadratic model was fit to that data to predict the residue at 45 μm. Based on their calibrations, the sizes of the three sieves used were 37.95 μm, 46.3 μm, and 57.8 μm, each with a standard uncertainty of ± 0.707 μm. The data obtained for analysis are mass fractions of cement residue using procedures consistent with standard test method ASTM C430.

In addition to including residues from different size sieves, the experimental design used to collect this data was also set up to allow the estimation of potential material heterogeneity between vials and between boxes of vials and potential day-to-day differences in the measurements. Overall, data were collected on 15 days on duplicate vials randomly sampled from 30 boxes of the candidate cement. Section 7 (Appendix A) shows the experimental design in the randomized run order used to collect the data. A few data points for each sieve appeared to be outliers (as shown in Section 7: Appendix A) and were not used in further analysis. Given the form of the final model that was found to describe the data (discussed below), the fact that the outlying points were always observed the first time when the sieve was used probably indicates a difference in behavior between a new, unused sieve and a sieve that has been used.

Because there was significant uncertainty in both the sieve calibrations and the measured sieve residues, the quadratic model was fit to the data using Bayesian methods [11]. The flexibility of the Bayesian approach makes it relatively easy to account for these two sources of variation and also to estimate day-to-day and material heterogeneity effects. These effects can be either random or systematically related to such variables as the number of times each sieve has been used or the position of a vial in the vialing sequence. The Bayesian approach also provides results with a clear cut statistical interpretation when incorporating systematic sources of uncertainty evaluated using Type B methods, which were significant in the uncertainty of the sieve calibrations.

The main difference between a Bayesian analysis and the frequentist statistical methods that people are often more familiar with arises in the way the true physical quantities (called parameters) are incorporated in the statistical model of the data. In a frequentist analysis, the parameters are treated as fixed, unknown constants while in the Bayesian analysis one's knowledge of the unknown parameter value is modeled with a probability distribution. In both approaches the probability of the data given the parameters (called the likelihood of the data) is also used when fitting the model. In a frequentist analysis, a statistical criterion, such as the least squares, is used to obtain estimates of each parameter in the model and their associated uncertainties. These are then propagated as necessary, typically according to the methods outlined in the ISO *Guide to the Expression of Uncertainty in Measurement* [12], to obtain an estimate of the parameter of interest (the measurand) and its uncertainty. In the Bayesian approach, prior probability

distributions for one's knowledge of each parameter are specified before analyzing the data and Bayes' theorem [11] is used to update the prior distributions using the information in the data. The resulting posterior distributions for each parameter in the model, including the measurand, represent our knowledge of the parameter values given the data that was observed. Because the measurand has a probability distribution in the Bayesian setting, direct probability statements about its uncertainty can be made based on its distribution. In particular, 95 % probability intervals can be computed.

Several models were actually fit to this data to try to determine which of many potential sources of uncertainty were needed to accurately describe the data. The appropriateness of these different models was assessed using posterior predictive residuals [13, 14]. Based on the results obtained with this series of models, the material appears to be homogeneous but to have systematic changes in the residue left behind on the sieves with each use. The effect of sieve use looked linear in each case, so the final model fit to obtain the certified values was actually quadratic in the original sieve size and included linear terms for each sieve that described how much the sieve size was effectively reduced each time the sieve was used.

Each model was fit to the data using a technique called Markov Chain Monte Carlo simulation using an open-source software package called WinBUGS [15]. The model used to obtain the certified values is shown in Figure 3, as implemented in WinBUGS.

The first block of code in the model defines the prior distributions for the parameters in the model and related parameters needed to specify the probability distributions used to describe the data. The first set of prior distributions, for the vector of parameters, `mu.s`, that describe the size of the openings in each sieve, are each normal distributions determined from the sieve calibration reports. These distributions are informative prior distributions based the calibrations of the sieves. The remaining prior distributions, for `sigma.wv` and `b`, are all uniform distributions with relatively large variances that are sometimes referred to as vague or non-informative distributions. Use of relatively non-informative prior distributions means that we are not incorporating prior knowledge of these parameter values in the analysis but are relying on the data alone to determine their values. The parameter `sigma.wv` describes the random variation in the residues measured using each sieve while the vector of parameters denoted `b` are the regression coefficients. The first three components of `b` are the regression coefficients that described the relationship between the sieve sizes and the residues while the latter three describe how much the sieve residues changed with each use of the sieve (one parameter for each size).

The next block of code describes the probability distributions for the data `y38`, `y46`, and `y58` (by sieve), which are assumed to be normal with different means for each measurement and different levels of variation for each set of data. This block of codes also defines the interrelationships of the parameters in the model, including the quadratic relationship between sieve size and residue and the linear change in sieve residue with repeated use of each sieve. Finally, predicted values, residuals, and individual p-values are also defined in the loop that specifies the likelihood of each data point.

In the last block of code the distribution of the residue predicted for a sieve with openings of exactly 45 μm sieve, `mr45`, is defined. Note that the quadratic relationship between the sieve sizes and the sieve residues is specified using an orthogonal polynomial to more easily relate the prior distributions of the parameters to the values of cement residue expected for the sizes of sieves used and to potentially improve the numerical stability of the computations. The data, in the format used in the fit of the model, is shown in Figure 4.

Residuals from the fit of the model are shown in Figure 7, Figure 6, and Figure 7 for `y38`, `y46`, and `y58` respectively. Since the simulation generates a distribution of predicted values associated with each data point, the distributions of residuals are shown using box plots rather than as individual points as would be done in an analysis of variance or regression analysis. The fact that these distributions randomly cluster around zero and the distributions all overlap zero indicates that the model seems to fit the data reasonably well.

The results of the analysis are shown in Figure 8. From those numeric results, the 2.5 % and 97.5 % quantiles of the distribution for `mr45`, can be seen to be 6.64 % and 8.14 %. The posterior mean of the distribution is 7.43 %. Computing upper and lower expanded uncertainties from these values indicates that an expanded uncertainty of ± 0.79 % can be used since the upper and lower values are fairly close and the larger of the two is 0.79 %. A standard uncertainty of $u_c = 0.38$ % should be used for future uncertainty computations based on the sieve residue of this material when passed through a 45 μm sieve.

model

```
{
for(i in 1:3)
{
pr.cv.s[i] <- 1/(uc.cv.s[i]*uc.cv.s[i])
mu.s[i]~dnorm(cv.s[i],pr.cv.s[i])
mu.r[i] <- b[1]+b[2]*(mu.s[i]-47.35)+b[3]*(mu.s[i]*mu.s[i]-
96.25751893891726*mu.s[i]+2249.549355091065)
sigma.wv[i]~dunif(0,10)
tau.wv[i] <- 1/(sigma.wv[i]*sigma.wv[i])
}
b[1]~dunif(0,20)
b[2]~dunif(-2,0)
b[3]~dunif(-1,1)
b[4]~dunif(-1,1)
b[5]~dunif(-1,1)
b[6]~dunif(-1,1)

for(i in 1:40)
{
y38[i]~dt(mu38[i],tau.wv[1],df[1])
y46[i]~dt(mu46[i],tau.wv[2],df[2])
y58[i]~dt(mu58[i],tau.wv[3],df[3])
mu38[i] <- mu.r[1]+b[4]*(i-1)
mu46[i] <- mu.r[2]+b[5]*(i-1)
mu58[i] <- mu.r[3]+b[6]*(i-1)
p.y38[i]~dt(mu38[i],tau.wv[1],df[1])
p.y46[i]~dt(mu46[i],tau.wv[2],df[2])
p.y58[i]~dt(mu58[i],tau.wv[3],df[3])
res[i] <- y38[i]-p.y38[i]
res[i+40] <- y46[i]-p.y46[i]
res[i+80] <- y58[i]-p.y58[i]
pv[i] <- step(res[i])
pv[i+40] <- step(res[i+40])
pv[i+80] <- step(res[i+80])
}

mr45 <- b[1]+b[2]*(45-47.35)+b[3]*(45*45-
96.25751893891726*45+2249.549355091065)
}
```

Figure 3: WinBUGS model developed to predict the sieve residue at 45 μm.

```
list(
cv.s=c(37.95,46.3,57.8),
uc.cv.s=c(0.7071068,0.7071068,0.70710
68),
df=c(200,200,200),
y38=c(
12.4663140033936,12.4550179928029,
12.2204472843450,12.5212181727409,
              NA,12.5062431325542,
              NA,12.0336100830249,
12.9922046771937,13.3766233766234,
12.9396482813749,12.8784094315116,
12.9687031296870,13.2067932067932,
12.9061281615515,13.4951553291379,
13.2122542660413,13.4907926341073,
13.4265734265734,13.1649865256014,
13.1728752621592,13.3000000000000,
13.3286671332867,13.9662573624838,
13.8819534605013,13.7951686963466,
13.8000000000000,13.7834598481822,
13.4792166266986,13.5483226837061,
13.5561097256858,13.8492261607589,
14.5251396648045,13.8875012488760,
14.0349127182045,13.8403117817528,
14.3085531574740,13.5064935064935,
13.7920703085988,13.6941082324697),
y46=c(
              NA,7.08936595107339,
6.61536924153093,6.78457234212630,
6.60207750699161,6.55262104841937,
6.79660169915043,6.88622754491018,
6.65866826634673,7.14214264279284,
6.90723710515794,6.97139427885577,
6.75918530351438,6.81908945686901,
7.03156212544946,7.19424460431655,
7.03015777910925,7.36337296433210,
7.28053530410466,7.20855828834233,
7.24478594950604,7.10147822612864,
7.24492855001499,6.95513140801439,
7.23072307230723,7.27037000099731,
7.40037950664137,7.30028921910841,
7.44362402714029,7.26691954481933,
7.36747529200359,7.21567059764142,
7.22205573868744,7.17282717282717,
7.50075007500750,7.44223266980094,
7.31756014774883,7.40296118447379,
7.31122652816620,7.38335498051753),
y58=c(
3.39592489013184,3.4889533140058
,
NA,3.42349535881825,
3.29703267059646,3.4255467891740
7,
3.22355289421158,3.4613845538215
3,
3.87116134840452,3.7307461492298
5,
3.52070414082817,3.5831919353228
9,
3.68815592203898,3.6681659170414
8,
3.52647352647353,3.7732082251946
5,
3.71368673255466,3.8169464428457
2,
3.67558929284858,3.9288213535939
2,
3.67485520271620,3.6874188068352
2,
3.70296436770137,3.8307661532306
5,
3.84000000000000,4.0639041437843
2,
3.79544546544147,3.9748327174672
9,
3.80581360503446,3.8100000000000
0,
3.78092577813248,3.9940089865202
2,
3.96960303969603,3.8937699680511
2,
3.93763741754947,4.0075954427343
6,
3.93842463014794,4.0720360180090
0,
4.09672262190248,4.1870690516638
4))
```

Figure 4: Data in *WinBUGS* format for the model shown Section 7 (Appendix A) used for prediction of the certified value. The data have been separated by sieve for convenience of model specification and potential outliers omitted from the analysis are indicated by NA.

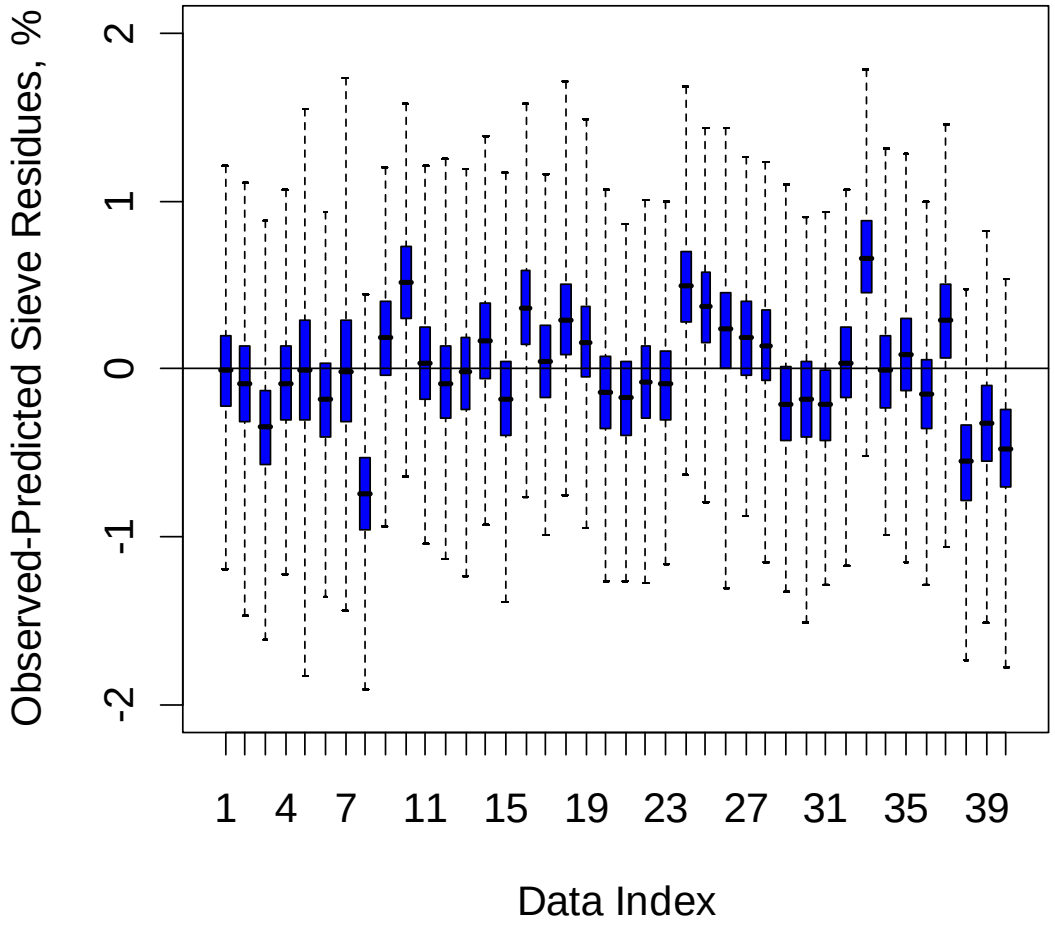

Figure 5: Box plots of the posterior predictive residuals from the fit of the model to the data from the 38 μm sieve. The whiskers indicate the ranges of the residual distributions. Note that in this model the variation of the residual distributions depends on the sieve size.

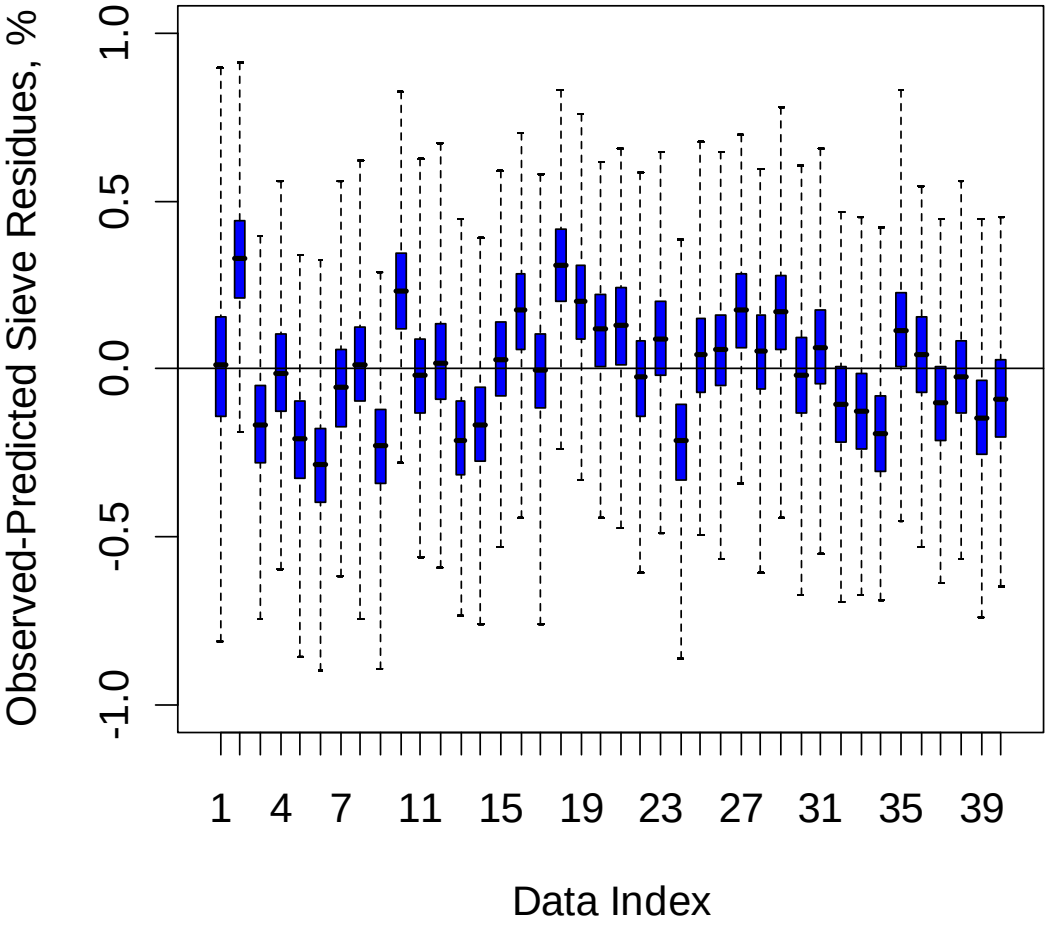

Figure 6: Box plots of the posterior predictive residuals from the fit of the model to the data from the 46 μm sieve. The whiskers indicate the ranges of the residual distributions. Note that in this model the variation of the residual distributions depends on the sieve size.

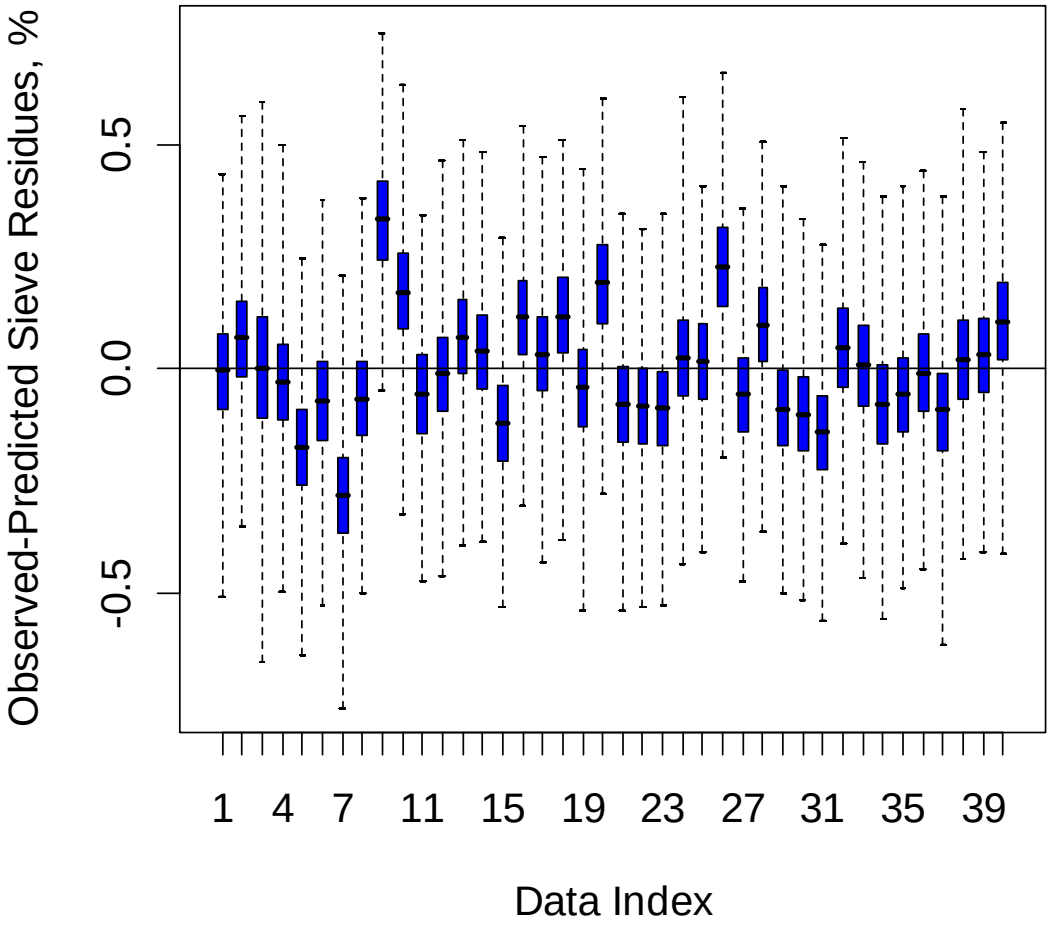

Figure 7: Box plots of the posterior predictive residuals from the fit of the model to the data from the 58 μm sieve. The whiskers indicate the ranges of the residual distributions. Note that in this model the variation of the residual distributions depends on the sieve size.

node	mean	sd	MC error	2.50%	median	97.50%	start	sample
b[1]	7.5650	0.2517	0.003729	7.0990	7.5530	8.0830	4001	4000
b[2]	-0.4541	0.0315	0.000439	-0.5227	-0.4514	-0.4027	4001	4000
b[3]	0.0211	0.0061	0.000089	0.0116	0.0204	0.0349	4001	4000
b[4]	0.0430	0.0045	0.000070	0.0343	0.0429	0.0521	4001	4000
b[5]	0.0188	0.0023	0.000039	0.0144	0.0188	0.0231	4001	4000
b[6]	0.0174	0.0017	0.000025	0.0141	0.0174	0.0208	4001	4000
mr45	7.4280	0.3790	0.005860	6.6430	7.4420	8.1390	4001	4000
mu.r[1]	12.4800	0.1040	0.001753	12.2700	12.4800	12.6800	4001	4000
mu.r[2]	6.7410	0.0531	0.000862	6.6340	6.7410	6.8470	4001	4000
mu.r[3]	3.4040	0.0394	0.000590	3.3260	3.4030	3.4820	4001	4000
mu.s[1]	38.0200	0.7049	0.009964	36.6500	38.0200	39.4100	4001	4000
mu.s[2]	46.2700	0.7221	0.011200	44.8600	46.2700	47.6800	4001	4000
mu.s[3]	57.7200	0.7136	0.012400	56.3000	57.7100	59.1500	4001	4000
sigma.wv[1]	0.3071	0.0382	0.000528	0.2429	0.3032	0.3931	4001	4000
sigma.wv[2]	0.1590	0.0195	0.000319	0.1262	0.1573	0.2015	4001	4000
sigma.wv[3]	0.1207	0.0146	0.000217	0.0960	0.1195	0.1529	4001	4000

Figure 8: Numerical results from the fit of the Bayesian model for certification of sieve residue.

5 Summary of Values

5.1.1 Certified values

The certified value for the SRM 46h certificate is:

Measurement and ASTM Method	Result
Sieve Residue C 430-96 (2003) (45 μm sieve)	7.43 % ± 0.79 %

5.1.2 Information only values

Measurement and ASTM Method	Result
Specific Surface Area C 204-07 (Blaine)	3720 cm^2/g ± 18 cm^2/g (372.0 m^2/kg ± 1.8 m^2/kg)
Density by Le Chatelier Flask	3.27 g/cm^3 and 3.30 g/cm^3

The particle size distribution (PSD) is given in Table 5 and Figure 9.

Table 5: PSD results (the relative standard uncertainties of each mean cumulative volume fraction are estimated to be about 0.5 %)

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, [%]	6.7	10.6	14.0	19.6	24.1	31.1	37.2	47.9	57.1	71.4	81.3	92.5	97.4	99.9	100.0

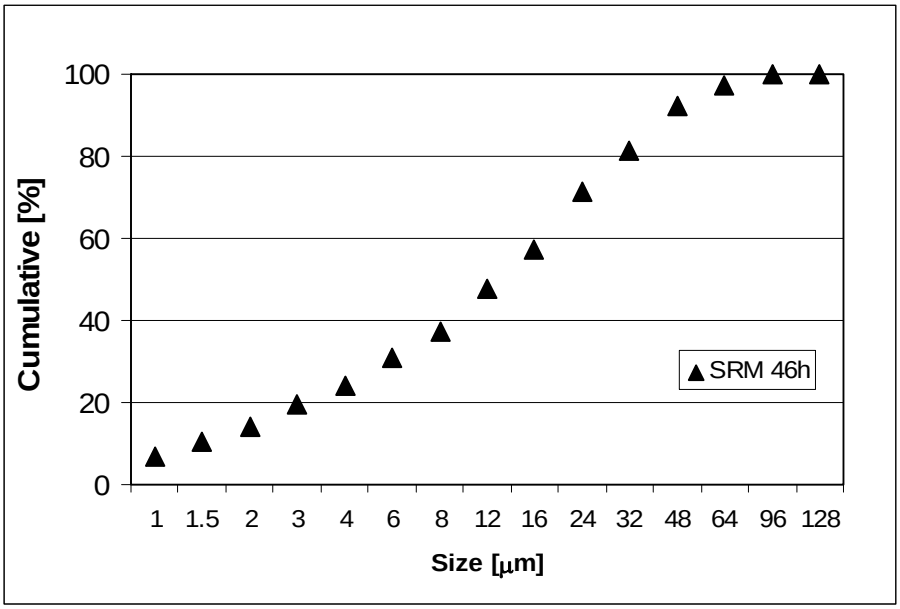

Figure 9: Cumulative particle size distribution for SRM 46h.

6 References

1 "Standard Test Method for Fineness of Hydraulic Cement by Air Permeability Apparatus", ASTM C204-07 (2007) Volume: 04.01

2 "Standard Test Method for Fineness of Portland Cement by the Turbidimeter", ASTM C115-96a (2003) Volume: 04.01

3 "Standard Test Method for Fineness of Hydraulic Cement by the 45-μm (No. 325) Sieve", ASTM C 430-96 (2003) Volume: 04.01

4 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)

5 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

6 Ferraris C.F., Hackley V.A., Avilés A.I., "Measurement of Particle Size Distribution in Portland Cement Powder: Analysis of ASTM Round-Robin Studies", Cement, Concrete and Aggregate Journal, Dec. 2004, vol. 26 #2, pp 71-81

7 Ferraris C.F., Guthrie W., Avilés A.I., Peltz M., Haupt R., MacDonald B. S. "Certification of SRM 114q; Part II (Particle Size distribution)", NIST SP260-166, November 2006

8 Ferraris, C.F., Guthrie W., Avilés A.I., Haupt, R., McDonald B., "Certification of SRM 114q; Part I", NIST SP260-161, July 2005

9 "Standard Test Method for Density of Hydraulic Cement", ASTM C188-95(2003) Volume: 04.01

10 Hackley V., Lum L-S., Gintautas V., Ferraris C., "Particle Size Analysis by Laser Diffraction Spectrometry: Applications to Cementitious Powders", NISTIR 7097, March 2004

11 Lee, Peter M. *Bayesian Statistics: An Introduction*, 2nd ed, Arnold Publishers, London (1997) 344 p

12 ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993).

13 Gelman, A., *A Bayesian Formulation of Exploratory Data Analysis and Goodness-of-fit Testing*, International Statistical Review (2003), 71, 2, 369–382.

14 Bayarri, M.J., Berger, J.O., *P Values for Composite Null Models*, Journal of the American Statistical Association, Vol. 95, No. 452. (Dec., 2000), pp. 1127-1142.

15 WinBUGS, http://www.mrc-bsu.cam.ac.uk/bugs/.

7 Appendix A: Data obtained for Residue

Values with bold black outlines were determined to be outliers and were not used in the analyses (see discussion Section 4)

Test Day	Vial selection				Mass measured [g]		Residue [%]
	Day vial prepared	Box #	Vial #	Sieve #	Before	After	
1	9	36	1	1	1.0024	0.1021	**10.19**
1	9	33	2	1	1.0016	0.1224	12.22
1	9	36	2	1	1.0026	0.0986	**9.83**
1	9	36	2	1	0.9997	0.1203	12.03
1	9	33	1	1	1.0019	0.1249	12.47
1	9	36	1	1	1.0011	0.1252	12.51
1	9	33	2	1	1.0015	0.1254	12.52
1	9	33	1	1	1.0004	0.1246	12.46
2	18	70	2	3	1.0017	0.0299	2.98
2	18	71	1	3	1.0009	0.0330	3.30
2	18	70	1	3	1.0012	0.0340	3.40
2	18	70	1	3	1.0003	0.0349	3.49
2	18	71	2	3	1.0020	0.0323	3.22
2	18	71	1	3	1.0013	0.0343	3.43
2	18	71	2	3	0.9996	0.0346	3.46
2	18	70	2	3	1.0019	0.0343	3.42
3	23	89	1	2	1.0020	0.0594	5.93
3	23	91	1	2	1.0012	0.0661	6.60
3	23	91	2	2	1.0005	0.0680	6.80
3	23	91	1	2	0.9996	0.0655	6.55
3	23	89	2	2	1.0007	0.0662	6.62
3	23	89	2	2	1.0008	0.0679	6.78
3	23	91	2	2	1.0020	0.0690	6.89
3	23	89	1	2	1.0015	0.0710	7.09
4	22	85	2	3	0.9998	0.0352	3.52
4	22	88	1	3	1.0005	0.0369	3.69
4	22	88	1	3	1.0005	0.0367	3.67
4	22	85	2	3	1.0019	0.0359	3.58
4	22	88	2	3	1.0010	0.0353	3.53
4	22	85	1	3	0.9997	0.0387	3.87
4	22	88	2	3	1.0018	0.0378	3.77
4	22	85	1	3	0.9998	0.0373	3.73
5	24	95	2	1	1.0003	0.1291	12.91
5	24	93	2	1	1.0008	0.1295	12.94
5	24	95	1	1	1.0001	0.1297	12.97
5	24	93	1	1	1.0006	0.1300	12.99
5	24	93	2	1	1.0009	0.1289	12.88
5	24	95	1	1	1.0010	0.1322	13.21
5	24	93	1	1	1.0010	0.1339	13.38
5	24	95	2	1	1.0011	0.1351	13.50

Test Day	Vial selection				Mass measured [g]		Residue [%]
	Day vial prepared	Box #	Vial #	Sieve #	Before	After	
6	6	23	1	2	1.0002	0.0666	6.66
6	6	24	2	2	1.0012	0.0704	7.03
6	6	24	1	2	1.0016	0.0677	6.76
6	6	23	2	2	1.0004	0.0691	6.91
6	6	24	1	2	1.0016	0.0683	6.82
6	6	23	2	2	0.9998	0.0697	6.97
6	6	23	1	2	0.9997	0.0714	7.14
6	6	24	2	2	1.0008	0.0720	7.19
7	7	25	1	2	1.0014	0.0704	7.03
7	7	26	2	2	1.0007	0.0725	7.24
7	7	25	2	2	1.0013	0.0729	7.28
7	7	26	1	2	1.0021	0.0726	7.24
7	7	26	2	2	1.0007	0.0696	6.96
7	7	25	1	2	1.0009	0.0737	7.36
7	7	25	2	2	1.0002	0.0721	7.21
7	7	26	1	2	1.0012	0.0711	7.10
8	11	43	1	1	1.0013	0.1319	13.17
8	11	41	1	1	1.0021	0.1324	13.21
8	11	41	2	1	1.0010	0.1344	13.43
8	11	43	1	1	1.0000	0.1330	13.30
8	11	41	2	1	1.0019	0.1319	13.16
8	11	43	2	1	1.0001	0.1333	13.33
8	11	43	2	1	1.0017	0.1399	13.97
8	11	41	1	1	0.9992	0.1348	13.49
9	13	51	1	1	1.0008	0.1349	13.48
9	13	51	1	1	1.0016	0.1357	13.55
9	13	51	2	1	1.0025	0.1359	13.56
9	13	49	1	1	1.0013	0.1390	13.88
9	13	49	2	1	1.0000	0.1380	13.80
9	13	51	2	1	1.0015	0.1387	13.85
9	13	49	2	1	1.0012	0.1380	13.78
9	13	49	1	1	1.0018	0.1382	13.80
10	17	66	2	1	1.0025	0.1407	14.03
10	17	66	1	1	1.0024	0.1456	14.53
10	17	68	1	1	1.0008	0.1432	14.31
10	17	68	2	1	1.0013	0.1381	13.79
10	17	66	1	1	1.0009	0.1390	13.89
10	17	68	2	1	0.9997	0.1369	13.69
10	17	66	2	1	1.0007	0.1385	13.84
10	17	68	1	1	1.0010	0.1352	13.51
11	1	1	2	3	1.0012	0.0368	3.68
11	1	3	2	3	1.0019	0.0371	3.70
11	1	1	1	3	1.0017	0.0372	3.71
11	1	3	2	3	0.9998	0.0383	3.83
11	1	3	1	3	1.0014	0.0368	3.67
11	1	1	2	3	1.0003	0.0393	3.93
11	1	3	1	3	1.0007	0.0369	3.69
11	1	1	1	3	1.0008	0.0382	3.82

Test Day	Vial selection				Mass measured [g]		Residue [%]
	Day vial prepared	Box #	Vial #	Sieve #	Before	After	
12	16	63	1	3	1.0011	0.0381	3.81
12	16	63	1	3	1.0000	0.0381	3.81
12	16	62	1	3	1.0000	0.0384	3.84
12	16	63	2	3	1.0024	0.0379	3.78
12	16	63	2	3	1.0015	0.0400	3.99
12	16	62	2	3	1.0012	0.0380	3.80
12	16	62	1	3	1.0015	0.0407	4.06
12	16	62	2	3	1.0013	0.0398	3.97
13	27	106	1	3	1.0001	0.0397	3.97
13	27	108	1	3	1.0004	0.0394	3.94
13	27	106	1	3	1.0016	0.0390	3.89
13	27	106	2	3	1.0006	0.0394	3.94
13	27	108	2	3	1.0008	0.0410	4.10
13	27	108	1	3	0.9995	0.0407	4.07
13	27	106	2	3	1.0006	0.0401	4.01
13	27	108	2	3	1.0007	0.0419	4.19
14	30	118	2	2	1.0013	0.0741	7.40
14	30	118	1	2	0.9999	0.0723	7.23
14	30	120	1	2	1.0022	0.0746	7.44
14	30	120	1	2	1.0018	0.0728	7.27
14	30	118	1	2	1.0027	0.0729	7.27
14	30	118	2	2	1.0027	0.0732	7.30
14	30	120	2	2	1.0017	0.0738	7.37
14	30	120	2	2	1.0006	0.0722	7.22
15	8	32	1	2	1.0017	0.0733	7.32
15	8	31	1	2	1.0011	0.0723	7.22
15	8	32	2	2	1.0012	0.0732	7.31
15	8	31	1	2	1.0010	0.0718	7.17
15	8	32	2	2	1.0009	0.0739	7.38
15	8	31	2	2	0.9999	0.0750	7.50
15	8	31	2	2	0.9997	0.0744	7.44
15	8	32	1	2	0.9996	0.0740	7.40

8 Appendix B: Particle size distribution measurements

Size [μm]	Cumulative Volume Fractions [%]				Statistics		
	Vial 1	Vial 2	Vial 3	Vial 4	**Average Cumulative Volume Fraction [%]**	Standard Deviation of Average Cumulative Volume Fraction, [%]	Coefficient of Variation, [%]
1	6.651	6.535	6.819	6.830	**6.71**	0.071	1.1%
1.5	10.535	10.368	10.725	10.750	**10.59**	0.089	0.8%
2	13.971	13.799	14.191	14.165	**14.03**	0.092	0.7%
3	19.577	19.439	19.864	19.699	**19.64**	0.090	0.5%
4	23.975	23.868	24.307	24.051	**24.05**	0.093	0.4%
6	31.007	30.925	31.370	31.063	**31.09**	0.097	0.3%
8	37.083	37.006	37.446	37.143	**37.17**	0.096	0.3%
12	47.862	47.772	48.209	47.909	**47.94**	0.095	0.2%
16	57.071	56.948	57.405	57.098	**57.13**	0.097	0.2%
24	71.383	71.202	71.685	71.441	**71.43**	0.100	0.1%
32	81.276	81.094	81.527	81.410	**81.33**	0.093	0.1%
48	92.386	92.312	92.540	92.570	**92.45**	0.062	0.1%
64	97.350	97.390	97.459	97.441	**97.41**	0.025	0.0%
96	99.990	99.993	99.993	99.766	**99.94**	0.056	0.1%
128	100.000	100.000	100.000	99.873	**99.97**	0.032	0.0%

9 Certificate of SRM 46h

The certificate for SRM 46h is provided below for information only. The official version of the certificate is the available online at:
https://srmors.nist.gov/certificates/view_cert2gif.cfm?certificate=46h

Select “Standard Reference Materials” from the website www.nist.gov and typing in “46h”.

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 46h

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 46h contains 10 glass vials of approximately 5 g of powdered cement. Each vial has a plastic snap off cap and is contained in a sealed foil bag.

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 accounted for by NIST. The certified value for sieve residue, given in Table 1, was calculated from NIST data using three calibrated sieves having openings ranging from 38 μm to 56 μm. The expanded uncertainty for the sieve residue was computed using a Bayesian statistical analysis and is given at the 95 % probability level. The expanded uncertainty accounts for the variability due to random measurement errors and sieve calibrations. Material homogeneity was assessed and no evidence of significant heterogeneity was found.

Table l. Certified Values

Measurand	ASTM Method	Certified Value and Expanded Uncertainty
Sieve Residue (45 μm residue)	C 430-96(2003)[a]	7.43 % ± 0.79 %

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

Expiration of Certification: The certification of SRM 46h is valid, within the measurement uncertainty specified, until **31 December 2017**, provided the SRM is 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) will facilitate notification.

The preparation of the material and the coordination of the technical measurements leading to certification were performed by C. Ferraris of the NIST Materials and Construction Research Division.

Statistical consultation on measurement design and analysis of the certification data was performed by W.F. Guthrie of the NIST Statistical Engineering Division.

Support aspects involved in the preparation of this SRM were coordinated through the NIST Measurement Services Division.

Jonathan Martin, Chief
Materials and Construction Research Division

Gaithersburg, MD 20899
Certificate Issue Date: 28 April 2008

Robert L. Watters, Jr., Chief
Measurement Services Division

INSTRUCTIONS FOR USE

Stability and Use: This material is considered to be extremely hygroscopic. Based on measurements in 1993 of several 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 3.4, allow the cement to settle for 2 minutes, and then perform the measurement.

Material Selection and Packaging[1]: The desired properties were selected for 46h to be coarser than 114q so as to obtain a 45μm 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 and had a mass fraction of 8.4 % 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 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.

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 ref. [4].

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 for SRM 46h are Chemical composition (Tables 2), Chemical Compounds (Table 3), surface area by Blaine (ASTM C204), density, and particle size distribution. **These values cannot be used for calibration purposes. For Blaine and particle size use SRM 114q or the current SRM 114.**

Chemical composition determined by ASTM Standard Test Method C114-02 is shown in Table 2. 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 3 provides the calculation of cement compounds according to ASTM C 150-02.

[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.

Table 2. Information Values 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 3. Information Values 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

Blaine: Analyses for specific surface area using ASTM Standard Test Methods C 204-00 were performed on four samples at NIST. SRM 114q *Portland Cement Fineness Standard* was used for calibration and the density was assumed to be 3.15 g/cm^3 as recommended in the SRM 114q certificate. The Blaine value was determined as 3720 cm^2/g ± 36 cm^2/g (372.0 m^2/kg ± 3.6 m^2/kg).

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

Particle Size Distribution (PSD): The SRM 46h particle size distribution (PSD) was determined using laser diffraction (LD) in liquid medium (LD-W) techniques at NIST. The results calculated a mean PSD for LD-W, and are shown graphically in Figure 1 and tabulated in Table 4. A complete discussion of the test procedures is provided in references 3 and 4. The particle size distribution provided here is given for information only. For a SRM on PSD, use SRM 114q or later version. The parameters used to develop the PSD were:

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

Table 4. The Particle Size Distribution of SRM46h Using LD Methodology (wet dispersion) [4]

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 (%)	6.7	10.6	14.0	19.6	24.1	31.1	37.2	47.9	57.1	71.4	81.3	92.5	97.4	99.9	100.0

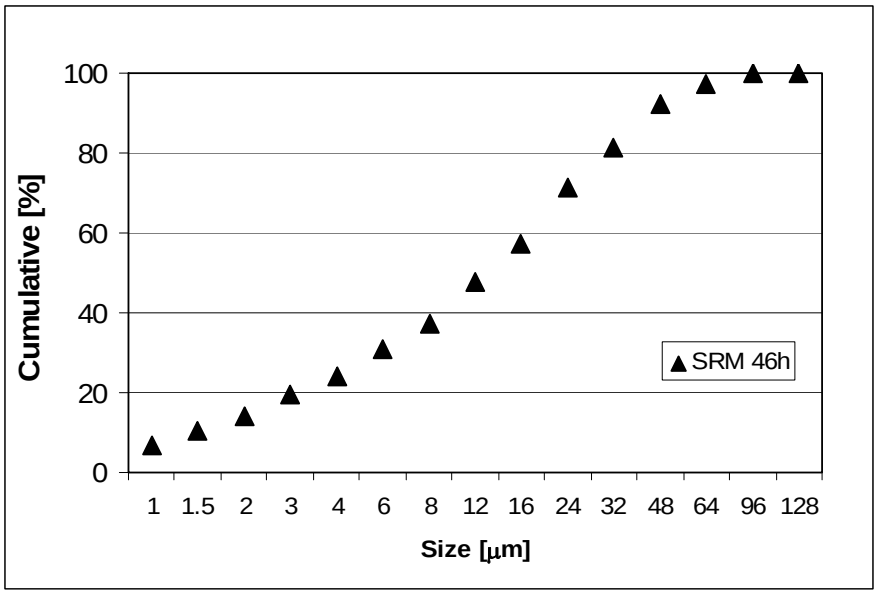

Figure 1. Graphical depiction of the particle size distribution of SRM 46h using LD (wet dispersion) [4].

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 (1995).

[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).

[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)

[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)

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 at http://www.nist.gov/srm.

NIST Technical Publications

Periodical

Journal of Research of the National Institute of Standards and Technology—Reports NIST research and development in metrology and related fields of physical science, engineering, applied mathematics, statistics, biotechnology, and information technology. Papers cover a broad range of subjects, with major emphasis on measurement methodology and the basic technology underlying standardization. Also included from time to time are survey articles on topics closely related to the Institute's technical and scientific programs. Issued six times a year.

Nonperiodicals

Monographs—Major contributions to the technical literature on various subjects related to the Institute's scientific and technical activities.

Handbooks—Recommended codes of engineering and industrial practice (including safety codes) developed in cooperation with interested industries, professional organizations, and regulatory bodies.

Special Publications—Include proceedings of conferences sponsored by NIST, NIST annual reports, and other special publications appropriate to this grouping such as wall charts, pocket cards, and bibliographies.

National Standard Reference Data Series—Provides quantitative data on the physical and chemical properties of materials, compiled from the world's literature and critically evaluated. Developed under a worldwide program coordinated by NIST under the authority of the National Standard Data Act (Public Law 90-396). NOTE:The Journal of Physical and Chemical Reference Data (JPCRD) is published bimonthly for NIST by the American Institute of Physics (AIP). Subscription orders and renewals are available from AIP, P.O. Box 503284, St. Louis, MO63150-3284.

National Construction Safety Team Act Reports—This series comprises the reports of investigations carried out under Public Law 107-231, the technical cause(s) of the building failure investigated; any technical recommendations for changes to or the establishment of evacuation and emergency response procedures; any recommended specific improvements to building standards, codes, and practices; and recomendations for research and other approprate actions to help prevent future building failures.

Building Science Series—Disseminates technical information developed at the Institute on building materials, components, systems, and whole structures. The series presents research results, test methods, and performance criteria related to the structural and environmental functions and the durability and safety characteristics of building elements and systems.

Technical Notes—Studies or reports which are complete in themselves but restrictive in their treatment of a subject. Analogous to monographs but not so comprehensive in scope or definitive in treatment of the subject area. Often serve as a vehicle for final reports of work performed at NIST under the sponsorship of other government agencies.

Voluntary Product Standards—Developed under procedures published by the Department of Commerce in Part 10, Title 15, of the Code of Federal Regulations. The standards establish nationally recognized requirements for products, and provide all concerned interests with a basis for common understanding of the characteristics of the products. NIST administers this program in support of the efforts of private-sector standardizing organizations.

*Order the **following** NIST publications—FIPS and NISTIRs—from the National Technical Information Service, Springfield, VA 22161.*

Federal Information Processing Standards Publications (FIPS PUB)—Publications in this series collectively constitute the Federal Information Processing Standards Register. The Register serves as the official source of information in the Federal Government regarding standards issued by NIST pursuant to the Federal Property and Administrative Services Act of 1949 as amended, Public Law 89-306 (79 Stat. 1127), and as implemented by Executive Order 11717 (38 FR 12315, dated May 11, 1973) and Part 6 of Title 15 CFR (Code of Federal Regulations).

NIST Interagency or Internal Reports (NISTIR)—The series includes interim or final reports on work performed by NIST for outside sponsors (both government and nongovernment). In general, initial distribution is handled by the sponsor; public distribution is handled by sales through the National Technical Information Service, Springfield, VA 22161, in hard copy, electronic media, or microfiche form. NISTIR's may also report results of NIST projects of transitory or limited interest, including those that will be published subsequently in more comprehensive form.