National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3287

Blueberry (Fruit)

This Standard Reference Material (SRM) is intended primarily for use in validating analytical methods for the determination of organic acids, anthocyanidins, and nutrients in blueberries and similar materials. This SRM can also be used for quality assurance when assigning values to in-house control materials. A unit of SRM 3287 consists of five packets, each containing approximately 5 g of freeze-dried, powdered fruit.

The development of SRM 3287 was a collaboration between the National Institute of Standards and Technology (NIST) and the National Institutes of Health Office of Dietary Supplements (NIH-ODS).

Certified Mass Fraction Values: Certified mass fraction values for quinic acid, free water-soluble vitamins, and elements in SRM 3287, reported on a dry-mass basis, are provided in Tables 1 through 3. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been taken into account [1]. Analyses for value assignment were performed by NIST and collaborating laboratories. Certified values were calculated as the mean of the mean values from NIST methods and the median of the mean values of results provided by collaborating laboratories, where appropriate. The associated uncertainties are expressed at an approximately 95 % level of confidence [2–4].

Reference Mass Fraction Values: Reference mass fraction values for anthocyanidins, additional organic acids, anions, proximates, amino acids, and additional nutrients are provided in Tables 4 through 7. A NIST reference value is a noncertified value that is the best estimate of the true value based on available data; however, the value does not meet the NIST criteria for certification [1] and is provided with an associated uncertainty that may reflect only measurement reproducibility, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods. The reference mass fraction values were derived from results reported by NIST and/or collaborating laboratories.

Expiration of Certification: The certification of **SRM 3287** is valid, within the measurement uncertainty specified, until **01 December 2027**, provided the SRM is stored in accordance with instructions given in this certificate (see "Instructions for Storage and Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM 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 this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Overall direction and coordination of the technical measurements leading to the original certification of this SRM were performed by L.C. Sander, K.E. Sharpless, and S.A. Wise of the NIST Chemical Sciences Division and S. Ehling of the Grocery Manufacturers Association (GMA, Washington, DC). Revision of this certificate was coordinated by C.A. Rimmer and L.J. Wood of the NIST Chemical Sciences Division.

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Certificate revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

Support for the development of SRM 3287 was provided in part by the NIH-ODS. Technical consultation was provided by J.M. Betz (NIH-ODS). Acquisition and preparation of the material were coordinated by K.E. Sharpless of the NIST Special Programs Office.

Analytical measurements at NIST were performed by G.E. Hahm, M.M. Phillips, B.J. Porter, and L.J. Wood of the NIST Chemical Sciences Division.

Analyses for value assignment were also provided by analysts participating in a GMA Food Industry Analytical Chemists Committee (GMA FIACC) interlaboratory comparison exercise: Campbell Soup Company (Camden, NJ); ConAgra Foods Analytical Laboratory, Omaha, NE; Covance, Inc., Madison, WI; Eurofins Scientific, Inc. (Des Moines, IA); Eurofins Chemical Control (Cuneo, Italy); Eurofins – Strassburger and Siegel (Hanover, MD); General Mills, Inc. (Minneapolis, MN); Hormel Foods Corporation (Austin, MN); Krueger Food Laboratories (Billerica, MA); McCormick & Company, Inc. (Hunt Valley, MD); National Center of Food Safety and Technology (Summit-Argo, IL); Ocean Spray (Lakeville, MA); Silliker Inc. (Chicago Heights, IL); The Hershey Company Technical Center (Hershey, PA); The J.M. Smucker Company (Orrville, OH); The National Food Laboratory (Livermore, CA); The Schwan Food Company (Salina, KS); and Welch's (Billerica, MA).

Statistical analysis was provided by J.H. Yen of the NIST Statistical Engineering Division.

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

NOTICE AND WARNINGS TO USERS

SRM 3287 IS INTENDED FOR RESEARCH USE; NOT FOR HUMAN CONSUMPTION.

INSTRUCTIONS FOR STORAGE AND USE

Storage: The SRM should be stored at controlled room temperature (20 °C to 25 °C), in unopened packets, until required for use. For elemental analyses, the packet can be opened and resealed; test portions can be removed and analyzed until the material reaches its expiration date. For other analyses, the packet can be opened and resealed; test portions can be removed and analyzed for two weeks after the packet was first opened.

Use: Prior to use, the contents of the packet should be mixed thoroughly. Allow the contents to settle for one minute prior to opening to minimize the loss of fine particles. Homogeneity of the material has not been evaluated for sample sizes smaller than those used by NIST methods described below. Therefore, the certified and reference values may not be valid for test portions smaller than those described in the sections below: 0.1 g for organic acid analysis, 0.5 g for elemental analysis, 2.5 g for vitamin analysis, 1.0 g for anthocyanidin analysis. Results obtained should include their own estimates of uncertainty and can be compared to the certified and reference values using procedures described in reference 5. The moisture conversion factor below can be used for the sample(s) when using an unopened packet for the first time. If using a previously opened and resealed packet, sample(s) need to be dried using one of the recommended techniques (see "Determination of Moisture"). The moisture content should be determined on a separate sample from that used for analysis, and the analytical result corrected to a dry-mass basis for comparison with values in this certificate.

Determination of Moisture: Moisture content of SRM 3287 was determined at NIST by (1) freeze-drying to constant mass over 7 d; (2) drying over magnesium perchlorate in a desiccator at room temperature for 28 d; and (3) drying for 1 h in a forced-air oven at 80 °C. Unweighted results obtained using all three techniques were averaged to determine a conversion factor of (0.9859 ± 0.0065) gram dry mass per gram as-received mass, which was used to convert data from an as-received to a dry-mass basis; the uncertainty shown on this value is an expanded uncertainty. A relative uncertainty component for the conversion factor (0.32 %) obtained from the moisture measurements is incorporated in the uncertainties of the certified and reference values, reported on a dry-mass basis, that are provided in this certificate.

SOURCE, PREPARATION, AND ANALYSIS[(1)]

Source and Preparation: The material for production of SRM 3287 was a combination (approximately 50/50) of Tifblue and Rubel (highbush and rabbiteye varieties) acquired from the U.S. Highbush Blueberry

[(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.

Council (Folsom, CA), which provided freeze-dried powdered blueberries (40 mesh) in nitrogen-flushed cans. The material was shipped to High-Purity Standards (Charleston, SC), where it was blended, aliquoted, and heat-sealed inside nitrogen-flushed 4 mil polyethylene bags, which were then sealed inside nitrogen-flushed aluminized plastic bags along with two packets of silica gel each. Following packaging, SRM 3287 was irradiated (Neutron Products, Inc., Dickerson, MD) to an absorbed dose of 7.2 kGy to 8.4 kGy.

Analytical Approach for Determination of Organic Acids: Value assignment of the mass fractions of organic acids in SRM 3287 was based on the combination of measurements from two different methods: isotope dilution liquid chromatography with mass spectrometric detection (ID-LC-MS) and ion chromatography with conductivity detection (IC-CD).

NIST Analysis for Organic Acids Using ID-LC-MS: Mass fractions of organic acids were determined by ID-LC-MS from duplicate, nominal 0.1 g test portions taken from each of six packets of SRM 3287. Organic acids were extracted into water and the solutions from four successive extractions were combined. For analysis by ID-LC-MS, an organic acid column was held at 40 °C and an aqueous mobile phase containing 0.5 % (volume fraction) formic acid was used under isocratic conditions at a flow rate of 0.5 mL/min. The mass spectrometer was operated in negative ion mode, with electrospray ionization. Each organic acid was matched with a ^{13}C- or ^{2}H-labeled internal standard, and quantitation was based on response factors calculated from the relative peak areas and concentrations. Calibrants were prepared gravimetrically, at levels intended to approximate the levels of the organic acids in the SRM following extraction. The purity of the neat organic acid calibrant materials was determined as an average of the manufacturer data and NIST data, including LC-absorbance at 210 nm, LC with evaporative light scattering detection (ELSD), differential scanning calorimetry (DSC), and Karl Fischer titration [6].

NIST Analysis for Organic Acids Using IC-CD: Mass fractions of organic acids were determined by IC-CD from duplicate, nominal 0.1 g test portions taken from each of six packets of SRM 3287. Organic acids were extracted into water and the solutions from four successive extractions were combined. A hydroxide-selective anion exchange column was held at 30 °C and a flow rate of 1.5 mL/min was used for the separation. Ultrapure water was used for generation of a hydroxide gradient, and a current of 186 mA was applied for suppression of the background conductivity from the hydroxide mobile phase. Quantitation was based on relative peak areas with trifluoroacetic acid (TFA) as an internal standard. Calibrants were prepared gravimetrically, at levels intended to approximate the levels of the organic acids in the SRM following extraction. The purity of the neat organic acid calibrant materials was determined as an average of the manufacturer data and NIST data, including LC-absorbance at 210 nm, LC-ELSD, DSC, and Karl Fischer titration [6].

Analytical Approach for Determination of Free Water-Soluble Vitamins: Value assignment of the mass fractions of free water-soluble vitamins in SRM 3287 was based on the combination of results from NIST with confirmation from data provided by one collaborating laboratory. NIST provided measurements by using ID-LC-MS. Duplicate, nominal 2.5 g aliquots were taken from each of six packets for analysis. Vitamins were extracted into water that contained acetic acid, and the solutions from four such successive extractions were combined. For analysis by ID-LC-MS, a C18 column with a gradient consisting of a mobile phase of methanol and 20 mmol/L ammonium formate in water was used with a flow rate of 0.8 mL/min. The mass spectrometer was operated in the positive ion mode with electrospray ionization. Each vitamin was matched with a ^{13}C-, ^{15}N-, or ^{2}H-labeled internal standard, and quantitation was based on response factors calculated from the relative peak areas and concentrations. Calibrants were prepared gravimetrically, at levels intended to approximate the levels of the vitamins in the SRM following extraction. The purity of calibrant materials was determined at NIST using LC-absorbance and DSC.

Analytical Approach for Determination of Elements: Value assignment of the mass fractions of elements in SRM 3287 was based on the combination of results from NIST and collaborating laboratories, where available [7]. NIST provided measurements by using inductively coupled plasma optical emission spectrometry (ICP-OES). Duplicate, nominal 0.5 g test portions from each of six packets of SRM 3287 were analyzed for calcium, copper, iron, magnesium, manganese, phosphorus, potassium, sodium, and zinc. Test portions were digested in sealed vessels with a HNO_3/HF mixture using a microwave digestion system. Quantitation was based on the method of standard additions using calibration solutions prepared from the SRM 3100 series of single-element standard solutions.

Analytical Approach for Determination of Anthocyanidins: Value assignment of the mass fractions of anthocyanidins in SRM 3287 was based on measurements at NIST using LC-absorbance. Mass fractions of cyanidin, delphinidin, malvidin, peonidin, and petunidin were measured by LC-absorbance using duplicate, nominal 1.0 g test portions from each of six packets of SRM 3287. Anthocyanidins were isolated from SRM 3287 and hydrolyzed by using a 24-h Soxhlet extraction into 225 mL of 6 % (volume fraction) hydrochloric acid in methanol. Pelargonidin chloride was used as an internal standard. Following extraction, 50 mL of the extract was reduced to approximately 10 mL and analyzed by LC-absorbance using a C18 column and detection at 520 nm. Gradient elution was used with a 25 mmol/L potassium phosphate/methanol mobile phase and a flow rate of 1 mL/min. Calibrants were prepared

gravimetrically at levels intended to approximate the levels of the anthocyanidins in the SRM following extraction. The purity of the neat calibrant materials was determined at NIST using LC-absorbance at 520 nm.

Collaborating Laboratories' Analyses: The GMA FIACC laboratories were asked to use their usual methods to make single measurements on test portions taken from each of two packets of SRM 3287 for measurements of nutrients. Because of the variability among data provided by laboratories participating in an interlaboratory comparison exercise, the median of the means is used. The median of the collaborating laboratories' means was combined with NIST data for calculation of certified values of elements. Collaborating laboratories' data alone were used to assign reference values for proximates, sugars, and amino acids.

Homogeneity Assessment: The homogeneity of anthocyanidins, organic acids, free water-soluble vitamins, and element mass fractions was assessed at NIST using the methods and test portion sizes described above. The uncertainties for delphinidin, malvidin, petunidin, and peonidin incoporate an uncertainty component for possible inhomogeniety based on standard deviation for the test portion size analyzed (see "Source, Preparation, and Analysis"). Homogeneity of constituents measured solely by collaborating laboratories (e.g., proximates, amino acids) was not assessed, although the data were treated as though these analytes were homogeneously distributed.

Value Assignment: For calculation of assigned values for analytes that were measured only by NIST, the mean of the mean values from NIST results were used. The collaborating laboratories reported the individual results for each of their analyses for a given analyte. The mean of each laboratory's results was then determined. For calculation of assigned values for analytes that were measured only by the GMA FIACC laboratories, the median of the laboratory means was used. For analytes that were also measured by NIST, the mean of the individual sets of NIST data were averaged with the median of the individual GMA FIACC laboratory means. In some cases, the calculation of assigned values is the mean of the means of NIST results with confirmation provided by collaborating laboratories.

Certified Mass Fraction Value for Quinic Acid: The certified mass fraction value, reported on a dry-mass basis, is the combined mean from the means of NIST ID-LC-MS data and the mean of NIST IC-CD data. Values are expressed as $x \pm U_{95\%}(x)$, where x is the certified value and $U_{95\%}(x)$ is the expanded uncertainty of the certified value. The true value of the analyte is believed to lie within the interval $x \pm U_{95\%}(x)$ with 95 % confidence. To propagate this uncertainty, treat the certified value as a normally distributed random variable with mean x and standard deviation $U_{95\%}(x)/2$ [2–4]. The measurand is the total mass fraction of quinic acid in blueberry (fruit) as listed in Table 1 on a dry-mass basis. Metrological traceability is to the measurement unit as realized through the purity determined for the primary chemical standards employed in the NIST methods.

Table 1. Certified Mass Fraction Value for Quinic Acid in SRM 3287

	Mass Fraction (mg/g)
Quinic Acid	25.53 ± 0.73

Certified Mass Fraction Values for Free Water-Soluble Vitamins: Each certified mass fraction value, reported on a dry-mass basis, is the mean from NIST ID-LC-MS data with confirmation by collaborating laboratories. Values are expressed as $x \pm U_{95\%}(x)$, where x is the certified value and $U_{95\%}(x)$ is the expanded uncertainty of the certified value. The true value of the analyte is believed to lie within the interval $x \pm U_{95\%}(x)$ with 95 % confidence. To propagate this uncertainty, treat the certified value as a normally distributed random variable with mean x and standard deviation $U_{95\%}(x)/2$ [2–4]. The measurand is the total mass fraction of free water-soluble vitamins in blueberry (fruit) as listed in Table 2 on a dry-mass basis. Metrological traceability is to the measurement unit as realized through the purity determined for the primary chemical standards employed in the NIST methods.

Table 2. Certified Mass Fraction Values for Free Water-Soluble Vitamins in SRM 3287

	Mass Fraction (mg/kg)
Free Thiamin	1.679 ± 0.030
Free Niacin[(a)]	2.864 ± 0.090
Free Pantothenic Acid	3.36 ± 0.19
Free Pyridoxine[(b)]	1.263 ± 0.020

[(a)] Measured as niacinamide and converted to niacin by multiplication by the ratio of the relative molecular masses.
[(b)] Measured as the sum of pyridoxine and pyridoxal, which was converted to pyridoxine by multiplication by the ratio of the relative molecular masses.

Certified Mass Fraction Values for Elements: Each certified mass fraction value, reported on a dry-mass basis, is the combined mean from the means of NIST data and the median of the mean results provided by collaborating laboratories. Values are expressed as $x \pm U_{95\%}(x)$, where x is the certified value and $U_{95\%}(x)$ is the expanded uncertainty of the certified value. The true value of the analyte is believed to lie within the interval $x \pm U_{95\%}(x)$ with 95 % confidence. To propagate this uncertainty, treat the certified value as a normally distributed random variable with mean x and standard deviation $U_{95\%}(x)/2$ [2–4]. The measurand is the total mass fraction of elements in blueberry (fruit) as listed in Table 3 on a dry-mass basis. Metrological traceability is to the SI derived unit for mass fraction (expressed as milligrams per kilogram).

Table 3. Certified Mass Fraction Values for Elements in SRM 3287

	Mass Fraction (mg/kg)
Calcium (Ca)[(a,b)]	323 ± 16
Copper (Cu)[(a,b)]	2.22 ± 0.16
Iron (Fe)[(a,b)]	12.20 ± 0.74
Magnesium (Mg)[(a,b)]	313.7 ± 7.2
Manganese (Mn)[(a,c)]	8.47 ± 0.59
Phosphorus (P)[(a,d)]	671 ± 21
Potassium (K)[(a,d)]	4490 ± 220
Zinc (Zn)[(a,e)]	6.49 ± 0.61

[(a)] NIST ICP-OES.
[(b)] Collaborating Laboratories. Reported methods included atomic absorption spectroscopy (AAS), direct current plasma optical emission spectrometry (DCP-OES), and ICP-OES.
[(c)] Collaborating Laboratories. Reported methods included inductively coupled plasma mass spectrometry (ICP-MS), colorimetry, and ICP-OES.
[(d)] Collaborating Laboratories. Reported methods included AAS, ICP-MS, DCP-OES, and ICP-OES.
[(e)] Collaborating Laboratories. Reported methods included AAS, ICP-MS, and ICP-OES.

Reference Mass Fraction Values for Organic Acids and Anions: Each reference mass fraction value, reported on a dry-mass basis, is the mean from NIST IC-CD data, except for shikimic acid which was determined by NIST ID-LC-MS. Values are expressed as $x \pm U_{95\%}(x)$, where x is the estimated value and $U_{95\%}(x)$ is the expanded uncertainty of the value. The method-specific value of the analyte is believed to lie within the interval $x \pm U_{95\%}(x)$ with about a 95 % confidence [2–4]. The measurand is the mass fraction for each organic acid and anion listed in Table 4 on a dry-mass basis as determined by the methods indicated. Metrological traceability is to mass fraction (expressed as milligrams per gram), as realized by the method used.

Table 4. Reference Mass Fraction Values for Organic Acids and Anions in SRM 3287

	Mass Fraction (mg/g)
Galacturonic Acid	0.1297 ± 0.0064
Glycolic Acid	0.1707 ± 0.0047
Isocitric Acid	0.2252 ± 0.0076
Oxalic Acid	0.0892 ± 0.0056
Shikimic Acid	0.438 ± 0.015
Phosphate	0.777 ± 0.044
Sulfate	1.17 ± 0.26

Reference Mass Fraction Values for Proximates, Sugars, Total Dietary Fiber, Calories, and Sodium: Each reference mass fraction value, reported on a dry-mass basis, is the median of the mean values provided by collaborating laboratories. Collaborating laboratories did not report methods used to determine proximates, sugars, and total dietary fiber. Values are expressed as $x \pm U_{95\%}(x)$, where x is the estimated value and $U_{95\%}(x)$ is the expanded uncertainty of the value. The value of the analyte is believed to lie within the interval $x \pm U_{95\%}(x)$ with about a 95 % confidence [2-4]. For proximates and sugars, the measurands are the mass fractions listed in Table 5, on a dry-mass basis, as determined by the collaborating laboratories. Metrological traceability is to mass fraction (expressed as grams per 100 grams) as realized by the methods used. For calories, the measurand is the caloric content (expressed as kilocalories per 100 grams), listed in Table 5 on a dry-mass basis as determined by the method indicated and metrological traceability is to the derived unit for energy. For sodium, the measurand is the mass fraction listed in Table 5 on a dry-mass basis as determined by the method indicated. Metrological traceability is to the SI derived unit for mass fraction (expressed as milligrams per kilogram), as realized by the method used.

Table 5. Reference Mass Fraction Values for Proximates, Sugars, Total Dietary Fiber, Calories, and Sodium in SRM 3287

	Mass Fraction (g/100 g)
Solids	98.59 ± 0.65
Ash	1.126 ± 0.084
Fat	1.40 ± 0.37
Protein	3.43 ± 0.30
Carbohydrate	91.92 ± 0.83
Total Sugars	60.4 ± 3.3
Fructose	30.5 ± 1.5
Glucose	30.5 ± 1.4
Total Dietary Fiber	18.4 ± 1.3
	Energy (kcal/100 g)
Calories[(a)]	392 ± 10
	Mass Fraction (mg/kg)
Sodium (Na)[(b)]	16.39 ± 0.74

[(a)] If the proximate values above are used for calculation, with caloric equivalents of 9, 4, and 4 for fat, protein, and carbohydrate, respectively, the mean caloric content is 394 kcal/100 g.
[(b)] NIST ICP-OES

Reference Mass Fraction Values for Amino Acids: Each reference mass fraction value, reported on a dry-mass basis, is the median of the mean values provided by collaborating laboratories. Values are expressed as $x \pm U_{95\%}(x)$, where x is the estimated value and $U_{95\%}(x)$ is the expanded uncertainty of the value. The method-specific value of the analyte is believed to lie within the interval $x \pm U_{95\%}(x)$ with about a 95 % confidence [2-4]. The measurand is the mass fraction for each amino acid listed in Table 6 on a dry-mass basis. Metrological traceability is to mass fraction (expressed as grams per 100 grams), as realized by the method used.

Table 6. Reference Mass Fraction Values for Amino Acids in SRM 3287

	Mass Fraction (g/100 g)
Alanine	0.167 ± 0.095
Arginine	0.342 ± 0.037
Aspartic Acid	0.279 ± 0.087
Cysteine	0.056 ± 0.023
Glutamic Acid	0.402 ± 0.079
Glycine	0.165 ± 0.006
Isoleucine	0.110 ± 0.034
Leucine	0.211 ± 0.022
Lysine	0.149 ± 0.022
Methionine	0.061 ± 0.007
Phenylalanine	0.134 ± 0.012
Proline	0.121 ± 0.019
Serine	0.141 ± 0.019
Threonine	0.121 ± 0.015
Tyrosine	0.088 ± 0.020
Valine	0.147 ± 0.060

Reference Mass Fraction Values for Anthocyanidins: Each reference mass fraction value is the mean result of NIST analyses using LC-absorbance. Values are expressed as $x \pm U_{95\%}(x)$, where x is the estimated value and $U_{95\%}(x)$ is the expanded uncertainty of the value. The method-specific value of the analyte is believed to lie within the interval $x \pm U_{95\%}(x)$ with about a 95 % confidence [2–4]. The measurand is the mass fraction for each anthocyanidin listed in Table 7, on a dry-mass basis, as determined by the method indicated. Metrological traceability is to mass fraction (expressed as milligrams per kilogram) as realized by the methods used.

Table 7. Reference Mass Fraction Values for Anthocyanidins in SRM 3287

	Mass Fraction (mg/kg)
Cyanidin	294 ± 24
Delphinidin	1180 ± 230
Malvidin	1390 ± 280
Petunidin	650 ± 120
Peonidin	286 ± 51

REFERENCES

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Certificate Revision History: 30 May 2019 (Editorial changes); 29 March 2019 (Removal of reference values for antioxidant capacity since methods used for analyses provided on previous certificates are no longer considered valid; addition of reference values for anthocyanidins; editorial changes); 19 December 2016 (Change of expiration date; editorial changes); 27 March 2013 (Removal of certified mass fraction values for citric acid and malic acid; update of storage and use information; editorial changes); 22 December 2011 (Addition of certified mass fraction values for water-soluble vitamins; addition of reference mass fraction values for antioxidant capacity; editorial changes); 14 September 2010 (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 https://www.nist.gov/srm.