



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 3281

#### Cranberry (Fruit)

This Standard Reference Material (SRM) is intended primarily for validation of methods for determining quinic acid, anthocyanidins, and elements in the fruit of cranberries and similar materials. This SRM can also be used for quality assurance when assigning values to in-house control materials. A unit of SRM 3281 consists of five packets, each containing approximately 6 g of freeze-dried, powdered fruit.

The development of SRM 3281 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:** The certified mass fraction values of quinic acid and elements, reported on a dry-mass basis, are provided in Tables 1 and 2. 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 provided by collaborating laboratories, where appropriate. The associated uncertainties are expressed at the 95 % level of confidence [2–4].

**Reference Mass Fraction Values:** Reference mass fraction values for anthocyanidins, proximates, and sugars, reported on a dry-mass basis, are provided in Tables 3 and 4. 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 associated uncertainties 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 3281** is valid, within the measurement uncertainty specified, until **01 December 2027**, provided the SRM is handled and 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 certification of this SRM were performed by L.C. Sander, and S.A. Wise of the NIST Chemical Sciences Division; and K.E. Sharpless of the NIST Special Programs Office. Revision of this certificate was coordinated by C.A. Rimmer and L.J. Wood of the NIST Chemical Sciences Division and W. Koshute of the Grocery Manufacturers Association (GMA, Washington, DC).

Support for the development of SRM 3281 was provided in part by NIH-ODS. Technical consultation was provided by J.M. Betz of the NIH-ODS. Acquisition of the material was performed by K.E. Sharpless.

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Certificate Issue Date: 04 April 2019  
*Certificate Revision History on Last Page*

Steven J. Choquette, Director  
Office of Reference Materials

Analytical measurements at NIST were performed by J.F. Browning, G.E. Hahm, R. Oflaz, 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 three GMA Food Industry Analytical Chemists (GMA FIAC) share group interlaboratory comparison exercises: Campbell Soup Company (Camden, NJ); Chelab Silliker (Resana, Italy); ConAgra Foods Analytical Laboratory (Omaha, NE); Covance, Inc. (Battle Creek, MI); Covance, Inc. (Harrogate, North Yorkshire, United Kingdom); Covance Inc. (Madison, WI); Del Monte Foods (Walnut Creek, CA); Eurofins Central Analytical Laboratories (New Orleans, LA); Eurofins Chemical Control (Cuneo, Italy); Eurofins Scientific, Inc. (Des Moines, IA); Eurofins S&S (Hanover, MD); General Mills (Golden Valley, MN); Hormel Foods Corporation (Austin, MN); Krueger Food Laboratories (Billerica, MA); Land O'Lakes (Arden Hills, MN); McCormick and Company (Hunt Valley, MD); National Center of Food Safety and Technology (Summit-Argo, IL); NSF International (Ann Arbor, MI); Ocean Spray (Lakeville, MA); Schwan Food Company (Salina, KS); Silliker Canada Co (Markham, ON, Canada); Silliker Food Science Center (Crete, IL); Silliker Iberica (Barcelona, Spain); The Coca-Cola Company (Rio de Janeiro, Brazil); The Coca-Cola Company (Apopka, FL); The Coca-Cola Company (Shanghai, China); The Hershey Company Technical Center (Hershey, PA); The J.M. Smucker Company (Orrville, OH); The National Food Laboratory (Livermore, CA); 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 WARNING TO USERS

SRM 3281 IS INTENDED FOR RESEARCH USE; NOT FOR HUMAN CONSUMPTION.

## INSTRUCTIONS FOR STORAGE AND USE

**Storage:** The material should be stored at controlled room temperature (20 °C to 25 °C) in an unopened packet until needed. 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, 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").

**Determination of Moisture:** Moisture content of SRM 3281 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 21 d; and (3) drying for 2 h in a forced-air oven at 80 °C. Unweighted results obtained using all three techniques were averaged to determine a conversion factor of  $(0.9761 \pm 0.0067)$  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. An uncertainty component for the conversion factor (0.34 %) 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.

## PREPARATION AND ANALYSIS<sup>(1)</sup>

**Material Acquisition and Preparation:** Frozen cranberries for production of SRM 3281 were freeze-dried and ground to 420 µm (40 mesh; Van Drunen Farms, Momence, IL) and shipped to NIST where they were further ground and sieved to 180 µm (80 mesh). 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

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<sup>(1)</sup> 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.

nitrogen-flushed, aluminized, plastic bags, along with two packets of silica gel each. Following packaging, SRM 3281 was irradiated (Neutron Products, Inc., Dickerson, MD) to an absorbed dose of 7.1 kGy to 8.9 kGy.

**Analytical Approach for Determination of Quinic Acid:** Value assignment of the mass fraction of quinic acid in SRM 3281 was based on the combination of measurements from two different methods: isotope dilution liquid chromatography with mass spectrometric detection (ID-LC-MS) and isotope dilution gas chromatography with mass spectrometric detection (ID-GC-MS).

*NIST Analysis for Quinic Acid Using ID-LC-MS:* The mass fraction of quinic acid was determined by ID-LC-MS from duplicate, nominal 0.1 g test portions taken from each of six packets of SRM 3281. Quinic acid was 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. <sup>13</sup>C<sub>3</sub>-quinic acid was used as internal standard, and quantification 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 quinic acid in the SRM following extraction. The purity of the neat quinic acid calibrant material 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 Quinic Acid Using ID-GC-MS:* The mass fraction of quinic acid was determined by ID-GC-MS from duplicate, nominal 0.1 g test portions taken from each of six packets of SRM 3281. Quinic acid was extracted into water, and the solutions from four successive extractions were combined. Ten milliliter aliquots of the combined water extracts were dried and mixed with N-methyl-N-(trimethylsilyl)trifluoroacetamide (MSTFA) containing 1 % (volume fraction) trimethylchlorosilane (TMCS) for derivatization prior to analysis by GC-MS using a GC×GC-MS instrument. A 5 % diphenyl/95 % dimethyl polysiloxane (mole fractions) column was used for the analysis; a second column, 50 % diphenyl/50 % dimethyl polysiloxane (mole fractions), was installed, but was not utilized in two-dimensional mode. <sup>13</sup>C<sub>3</sub>-quinic acid was used as internal standard, and quantification was based on response factors calculated from the relative peak areas and concentrations. Although full spectra were acquired, extracted ion chromatograms were used for quantification [6].

**Analytical Approach for Determination of Elements:** Value assignment of the mass fractions of the elements in SRM 3281 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), inductively coupled plasma mass spectrometry (ICP-MS), and instrumental neutron activation analysis (INAA).

*NIST Analyses for Ca, Cu, Fe, K, Mg, Mn, Na, P, and Zn Using ICP-OES and ICP-MS:* Mass fractions of calcium, copper, iron, magnesium, manganese, phosphorus, potassium, sodium, and zinc were measured by ICP-OES using duplicate, nominal 0.5 g test portions from each of six packets of SRM 3281. Mass fractions of copper and manganese were also measured by ICP-MS using duplicate, nominal 0.5 g test portions from each of six packets of SRM 3281. Test portions were digested in sealed vessels with nitric and hydrofluoric acids using a microwave digestion system. Quantification was based on the method of standard additions using calibration solutions prepared from the SRM 3100 series of single-element standard solutions.

*NIST Analyses for Al, Ca, Cl, Fe, Mg, Mn, Na, and Zn Using INAA:* Mass fractions of aluminum, calcium, chlorine, iron, magnesium, manganese, sodium, and zinc were measured by INAA using duplicate, nominal 0.2 g test portions taken from each of six packets of SRM 3281. Powders were pressed into cylindrical pellets, and samples, standards, and controls were packaged individually in clean polyethylene bags. For analysis of aluminum, calcium, chlorine, magnesium, manganese, and sodium, the packages were irradiated with one flux monitor foil for 2 min at a reactor power of 20 MW. The count was done for 5 min after a 10 min decay. For the analysis of iron and zinc, standards and controls were irradiated for 6 h, with irradiation capsules inverted 180 degrees at the midpoint. The count was done for 7 h to 24 h after a 2-week decay. Quantification for INAA was based on solutions made using pure metal foils, pure compounds, and 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 3281 was based on measurements at NIST using liquid chromatography with absorbance detection (LC-absorbance). Mass fractions of cyanidin, delphinidin, and peonidin were measured by LC-absorbance using duplicate, nominal 1.0 g test portions from each of six packets of SRM 3281. Anthocyanidins were isolated from SRM 3281 and hydrolyzed by using a 24h -Soxhlet extraction into 250 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 Share Group laboratories were asked to use their usual methods to make single measurements on test portions taken from each of two packets of SRM 3281 for measurements of elements and sugars. Because of the variability among data provided by laboratories participating in an interlaboratory comparison exercise, the median of the means was 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, and sugars. Commercial laboratories were used to provide measurement values for proximates.

**Homogeneity Assessment:** The homogeneity of quinic acid, anthocyanidins, and elements in SRM 3281 was assessed at NIST by using the methods and test portion sizes described above. An analysis of variance with 5 % significance level did not show inhomogeneity for the test portions analyzed (see "Instructions for Storage and Use"). Homogeneity of constituents measured solely by collaborating laboratories (e.g., proximates, sugars) 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 was 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 collaborating 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 was averaged with the median of the individual collaborating laboratory means. For calculation of assigned values determined by commercial laboratories, the mean of the method means of each laboratory was used.

**Certified Mass Fraction Value for Quinic Acid:** The certified mass fraction value, reported on a dry-mass basis, is the combined mean from the mean of NIST ID-LC-MS data and the mean of NIST ID-GC-MS 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 cranberry (fruit) as listed in Table 1 on a dry-mass basis. Metrological traceability is to the SI derived unit for mass fraction (expressed as milligrams per gram).

Table 1. Certified Mass Fraction Value for Quinic Acid in SRM 3281

|             | Mass Fraction<br>(mg/g) |
|-------------|-------------------------|
| Quinic Acid | 47.8 ± 6.8              |

**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 for each element listed in Table 2 on a dry-mass basis. Metrological traceability is to the SI derived unit for mass fraction (expressed as milligrams per kilogram).

Table 2. Certified Mass Fraction Values for Elements in SRM 3281

|                                     | Mass Fraction<br>(mg/kg) |
|-------------------------------------|--------------------------|
| Calcium (Ca) <sup>(a,b,c)</sup>     | 514 ± 18                 |
| Copper (Cu) <sup>(a,b,c,d)</sup>    | 3.65 ± 0.15              |
| Iron (Fe) <sup>(a,b,c)</sup>        | 26.3 ± 1.5               |
| Magnesium (Mg) <sup>(a,b,c)</sup>   | 429 ± 20                 |
| Manganese (Mn) <sup>(a,b,c,d)</sup> | 21.59 ± 0.44             |
| Phosphorus (P) <sup>(a,e)</sup>     | 807 ± 56                 |
| Potassium (K) <sup>(a,b,c)</sup>    | 7680 ± 400               |
| Sodium (Na) <sup>(a,b,c)</sup>      | 263 ± 10                 |
| Zinc (Zn) <sup>(a,b,c)</sup>        | 6.69 ± 0.51              |

<sup>(a)</sup> NIST ICP-OES

<sup>(b)</sup> NIST INAA

<sup>(c)</sup> Collaborating Laboratories. Reported methods included atomic absorption spectroscopy (AAS), ICP-MS, and ICP-OES.

<sup>(d)</sup> NIST ICP-MS

<sup>(e)</sup> Collaborating Laboratories. Reported methods included ICP-MS, colorimetry, and ICP-OES.

**Reference Mass Fraction Values for Proximates, Sugars, and Calories:** Each reference mass fraction value, reported on a dry-mass basis, is the median of the mean values provided by collaborating laboratories or the mean of the means provided by commercial 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]. For proximates and sugars, the measurands are the mass fractions listed in Table 3, on a dry-mass basis, as determined by the methods indicated. 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 3 on a dry-mass basis as determined by the method indicated and metrological traceability is to the derived unit for energy.

Table 3. Reference Mass Fraction Values for Proximates, Sugars, and Calories in SRM 3281

|                             | Mass Fraction<br>(g/100 g) |
|-----------------------------|----------------------------|
| Solids <sup>(a)</sup>       | 90.9 ± 1.2                 |
| Ash <sup>(b)</sup>          | 4.89 ± 0.18                |
| Protein <sup>(c)</sup>      | 2.10 ± 0.51                |
| Carbohydrate <sup>(d)</sup> | 81.6 ± 3.5                 |
| Total Sugars <sup>(e)</sup> | 24.4 ± 3.6                 |
| Fructose <sup>(e)</sup>     | 4.27 ± 0.48                |
| Glucose <sup>(e)</sup>      | 20.1 ± 3.2                 |
|                             | Energy<br>(kcal/100 g)     |
| Calories <sup>(d)</sup>     | 376 ± 28                   |

<sup>(a)</sup> AOAC 925.09 Solids (Total) and Moisture in Flour

<sup>(b)</sup> AOAC 923.03 Ash of Flour

<sup>(c)</sup> AOAC 922.15 Crude Protein in Meat and Meat Products Including Pet Foods.

<sup>(d)</sup> Calculation

<sup>(e)</sup> Collaborating Laboratories. Reported methods included LC with refractive index and LC with evaporative light scattering detection.

**Reference Mass Fraction Values for Elements:** Each reference mass fraction value is the mean result of NIST analyses using INAA. 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 element listed in Table 4 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 4. Reference Mass Fraction Values for Elements in SRM 3281

|               | Mass Fraction<br>(mg/kg) |
|---------------|--------------------------|
| Aluminum (Al) | 14.14 ± 0.66             |
| Chlorine (Cl) | 796.4 ± 9.4              |

**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 analyte listed in Table 5, 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 5. Reference Mass Fraction Values for Anthocyanidins in SRM 3281

|             | Mass Fraction<br>(mg/kg) |
|-------------|--------------------------|
| Cyanidin    | 119 ± 31                 |
| Delphinidin | 5.18 ± 0.68              |
| Peonidin    | 121 ± 22                 |

## REFERENCES

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- [2] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at [https://www.bipm.org/utis/common/documents/jcgm/JCGM\\_100\\_2008\\_E.pdf](https://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf) (accessed Apr 2019); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at <https://www.nist.gov/pml/nist-technical-note-1297> (accessed Apr 2019).
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**Certificate Revision History:** 04 April 2019 (Removal of reference values for antioxidant capacity since methods used for analyses provided in previous certificates are no longer considered valid; correction to chlorine reference value; editorial changes); 28 August 2017 (Change of element reference values to certified values; addition of reference values for proximates, aluminum, chlorine, and anthocyanidins; removal of certified values for citric acid, malic acid, and shikimic acid; change of expiration date; editorial changes); 29 February 2012 (Addition of reference 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](mailto:srminfo@nist.gov); or via the Internet at <https://www.nist.gov/srm>.