



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 3282

Low-Calorie Cranberry Juice Cocktail

This Standard Reference Material (SRM) is intended primarily for use in validating analytical methods for the determination of organic acids and nutrient elements in cranberry juice cocktails and similar matrices. This SRM can also be used for quality assurance when assigning values to in-house control materials. A unit of SRM 3282 consists of five ampoules, each containing approximately 1.2 mL of liquid.

The development of SRM 3282 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 of selected organic acids and elements are provided in Tables 1 and 2, respectively. 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 investigated or taken into account [1]. Values were derived from the combination of results provided by NIST using two methods (organic acids) and NIST data and data provided by collaborating laboratories (elements). The certified values in this material are the equally weighted means of the means of individual sets of results (organic acids) and the equally weighted mean of results provided by NIST and the collaborating laboratories' median (elements); the associated uncertainties are expanded uncertainties at the 95 % level of confidence [2-4]. Values are reported in mass fraction units [5].

Reference Mass Fraction Values: Reference mass fraction values for additional organic acids, anions, elements, and sugars are provided in Tables 3 through 5. Reference values are noncertified values that are the best estimate of the true values based on available data; however, the values do not meet the NIST criteria for certification [1] and are 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.

Expiration of Certification: The certification of **SRM 3282** is valid, within the measurement uncertainty specified, until **30 April 2015**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Warning and 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) will facilitate notification.

Support for the development of SRM 3282 was provided in part by NIH-ODS. Technical consultation was provided by J.M. Betz (NIH-ODS).

The overall direction and coordination of the technical measurements leading to the certification of this SRM were performed by L.C. Sander, K.E. Sharpless, and S.A. Wise of the NIST Analytical Chemistry Division.

Acquisition of the material was performed by K.E. Sharpless of the NIST Analytical Chemistry Division.

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Analytical Chemistry Division

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Measurement Services Division

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Certificate Issue Date: 14 September 2010

Analytical measurements at NIST were performed by M.M. Phillips, M.A. Pichon, and L.J. Wood of the NIST Analytical Chemistry Division. Results were also provided by analysts participating in an interlaboratory comparison exercise involving members of the Grocery Manufacturers Association (GMA, Washington, DC) Food Industry Analytical Chemists Committee (FIACC); participants are listed in Appendix A.

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 Measurement Services Division.

WARNING AND INSTRUCTIONS FOR STORAGE AND USE

Warning: For laboratory use only. Not for human consumption.

Storage: The material should be stored under refrigeration (4 °C), in unopened ampoules, until needed. The certification does not apply to contents of previously opened and stored ampoules as the stability of analytes has not been investigated.

Use: Prior to removal of a test portion for analysis, the contents of an ampoule should be mixed thoroughly. For certified values to be valid, test portions equal to or greater than 0.5 g for organic acid analyses and 1.4 g for element analyses should be used. The stability of organic acids and elements in opened ampoules has not been investigated.

PREPARATION AND ANALYSIS¹

Material Acquisition and Preparation: The low-calorie cranberry juice cocktail used for production of SRM 3282 was obtained from a commercial source. The material was ampouled under argon at NIST.

Analytical Approach for Determination of Organic Acids: Value assignment of the concentrations of organic acids in SRM 3282 was based on the combination of measurements from two different methods: liquid chromatography with absorbance detection (LC-UV) and ion chromatography with conductivity detection (IC-CD). Duplicate test portions of approximately 0.5 g were taken from each of six ampoules for analysis using each of the methods. Samples were diluted with a solution containing potassium phosphate monobasic and DL-dithiothreitol for stabilization of ascorbic acid prior to analysis.

For analysis by LC-UV, two organic acid columns connected in series were held at 43 °C. An aqueous mobile phase adjusted to pH 2.0 using concentrated hydrochloric acid was used under isocratic conditions. The flow rate was set to 0.5 mL/min and the sample injection volume was 1 µL. Ultraviolet absorbance detection was carried out at 210 nm. Quantitation was based on an external calibration model [6].

For analysis by IC-CD, 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 [6].

Analytical Approach for Determination of Elements: NIST and collaborating laboratories' data were combined to provide certified values. For NIST analyses, single 1.4 g test portions from each of six ampoules of SRM 3282 were analyzed for calcium, copper, iron, magnesium, manganese, potassium, sodium, and zinc by using inductively coupled plasma optical emission spectrometry (ICP-OES). Test portions were digested in sealed vessels with nitric acid 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.

Collaborating Laboratories' Analyses: The GMA FIACC laboratories (Appendix A) were asked to use their usual methods to make single measurements on test portions taken from each of two ampoules of SRM 3282. The median of the collaborating laboratories' means were combined with NIST data for calculation of certified values of elements. Collaborating laboratories' data alone were used to assign reference values for sugars.

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.

Homogeneity Assessment: The homogeneity of organic acid and element contents was assessed at NIST by using the methods described above. An analysis of variance did not show inhomogeneity for the test portions analyzed; see “Warning and Instructions for Storage and Use.”

Value Assignment: The equally weighted means of the mean results from each set of data available were used to calculate the assigned values. The results of the GMA FIACC data are the medians of the collaborating laboratory means.

Table 1. Certified Mass Fraction Values for Organic Acids in SRM 3282^(a)

	Mass Fraction (mg/g)
Citric Acid	3.221 ± 0.053
Malic Acid	2.133 ± 0.042
Quinic Acid	2.672 ± 0.048

^(a) Each certified mass fraction value is an equally weighted mean of results provided by LC-UV and IC-CD. The uncertainty provided with each value is an expanded uncertainty about the mean to cover the measurand with approximately 95 % confidence; it expresses both the observed difference between the results from the methods and their respective uncertainties, consistent with the ISO Guide and its Supplement 1 [2-4]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the effects of the combined components of uncertainty, and k is a coverage factor corresponding to approximately 95 % confidence for each analyte. For the certified values shown above, $k = 2$.

Table 2. Certified Mass Fraction Values for Elements in SRM 3282^(a)

	Mass Fraction (mg/kg)
Calcium	26.3 ± 1.6
Copper	0.23 ± 0.06
Magnesium	12.97 ± 0.84
Manganese	0.493 ± 0.016
Potassium	247 ± 12
Sodium	201 ± 20

^(a) Each certified mass fraction value is an equally weighted mean of results provided by NIST and the GMA FIACC laboratories' median. The uncertainty provided with each value is an expanded uncertainty about the mean to cover the measurand with approximately 95 % confidence; it expresses both the observed difference between the results from the methods and their respective uncertainties, consistent with the ISO Guide and its Supplement 1 [2-4]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the effects of the combined components of uncertainty, and k is a coverage factor corresponding to approximately 95 % confidence for each analyte. For the certified values shown above, $k = 2$.

Table 3. Reference Mass Fraction Values for Organic Acids and Anions in SRM 3282^(a)

	Mass Fraction (mg/g)
Ascorbic Acid	0.16 ± 0.03
Galacturonic Acid	0.348 ± 0.005
Glycolic Acid	0.046 ± 0.002
Isocitric Acid	0.0198 ± 0.0007
Oxalic Acid	0.096 ± 0.003
Phosphate	0.0266 ± 0.0006
Shikimic Acid	0.051 ± 0.001
Sulfate	0.0101 ± 0.0004

^(a) Each reference mass fraction value is the mean of results provided by IC-CD, except for shikimic acid and ascorbic acid, which are the means of results provided by LC-UV. The uncertainty provided with each value is an expanded uncertainty about the mean to cover the measurand with approximately 95 % confidence [2]. The uncertainty incorporates within-method uncertainty. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the effects of the combined components of uncertainty, and k is a coverage factor corresponding to approximately 95 % confidence for each analyte. For the reference values shown above, $k = 2.2$.

Table 4. Reference Mass Fraction Values for Elements in SRM 3282^(a)

	Mass Fraction (mg/kg)
Iron	0.54 ± 0.14
Zinc	0.15 ± 0.06

^(a) Each reference mass fraction value is the mean of results provided at NIST by using ICP-OES. The uncertainty provided with each value is an expanded uncertainty about the mean to cover the measurand with approximately 95 % confidence [2]. The uncertainty incorporates within-method uncertainty. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the effects of the combined components of uncertainty, and k is a coverage factor corresponding to approximately 95 % confidence for each analyte. For the reference values shown above, $k = 2.57$.

Table 5. Reference Mass Fraction Values for Sugars in SRM 3282^(a)

	Mass Fraction (%)	k
Total Sugars	2.86 ± 0.05	2.57
Fructose	2.08 ± 0.10	2.45
Glucose	0.85 ± 0.06	2.45

^(a) Each reference mass fraction value is the median of results provided by the GMA FIACC laboratories. The uncertainty provided with each value is an expanded uncertainty about the median to cover the measurand with approximately 95 % confidence, consistent with the ISO Guide [2]. The uncertainty incorporates within-method uncertainty. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the effects of the combined components of uncertainty, and k is a coverage factor corresponding to approximately 95 % confidence for each analyte.

REFERENCES

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- [6] Phillips, M.M.; Case, R.J.; Rimmer, C.A.; Sharpless, K.E.; Wise, S.A.; Sander, L.C.; *Determination of Organic Acids in Vaccinium Berry Standard Reference Materials*; Anal. Bioanal. Chem., Vol. 398, pp. 425-434 (2010).

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) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.

APPENDIX A

Analysts at the laboratories listed below performed measurements that contributed to the value assignment of SRM 3282 Low-Calorie Cranberry Juice Cocktail.

Campbell Soup Company; Camden, NJ
Covance, Inc.; Madison, WI
Eurofins Scientific, Inc.; Des Moines, IA
Hormel Foods Corporation; Austin, MN
Krueger Food Laboratories; Cambridge, MA
The Coca-Cola Company, Apopka, FL
The National Food Laboratory; Livermore, CA