



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 3530

Iodized Table Salt (Iodide)

This Standard Reference Material (SRM) is intended primarily for validation of methods for the determination of iodide in an iodized table salt or similar material. This SRM can also be used for quality assurance when assigning values to in-house reference materials. A unit of SRM 3530 consists of one bottle containing approximately 200 g of a commercially available iodized table salt sealed inside an aluminized polyethylene terephthalate (PET) pouch containing an oxygen absorber pack.

The development of SRM 3530 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 value of iodine as iodide in SRM 3530, reported on a dry-mass basis, is provided in Table 1. 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]. Analyses for value assignment were performed at NIST and Centro Nacional De Metrología, Mexico (CENAM). The certified mass fraction value is the mean of the reported method means. The associated uncertainties are expressed at an approximately 95 % level of confidence [2–5].

Expiration of Certification: The certification of **SRM 3530** is valid, within the measurement uncertainty specified, until **30 November 2020**, provided the SRM is handled and stored in accordance with the 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.

Coordination of the technical measurements leading to the certification of this SRM was performed by K.E. Murphy of the NIST Chemical Sciences Division.

Analytical measurements were performed by W.C. Davis, S.E. Long, Y. Nuevo Ordonez and K.W. Pratt of the NIST Chemical Sciences Division and J.L. Ortiz-Aparicio and J. Montero-Ruiz of the CENAM Inorganic Analysis Division.

Statistical analysis was provided by H.K. Iyer of the NIST Statistical Engineering Division.

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Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
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Steven J. Choquette, Acting Director
Office of Reference Materials

NOTICE AND WARNING TO USERS

SRM 3530 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) and maximum humidity of 65 % in the original sealed aluminized PET pouch. Once the pouch is opened, the bottled should be stored tightly capped in the aluminized PET pouch at controlled room temperature (20 °C to 25 °C) and maximum humidity of 65 %. Test portions can be removed and analyzed until the material reaches its expiration date.

Use: Before use, the contents of the bottle should be mixed for 30 s by rotating and/or rolling. For the certified value to be valid, a minimum test portion of 0.25 g should be used (see descriptions of analysis methods below). Test portions should be analyzed as received. Separate test portions should be dried as recommended below (see “Determination of Moisture”) to determine residual moisture content. Results obtained in analyses should include their own estimates of uncertainty and can be compared to the certified value using procedures described in NIST Special Publication 260-181 [6].

SOURCE, PREPARATION, AND ANALYSIS⁽¹⁾

Source and Preparation: Material for SRM 3530 was originally manufactured for retail food sale and was obtained from a commercial supplier of table salt. The table salt contains high purity sodium chloride, sodium silicoaluminate, calcium sulfate, dextrose and potassium iodide. Five bags, each containing 11 kg of iodized table salt, were blended and packaged in individual units at NIST. Each unit consists of 200 g of table salt packaged in a nitrogen-flushed, 6.5 oz, opaque, high-density polyethylene bottle. The bottle is tightly capped, sealed with plastic shrink-wrap, and further sealed inside a nitrogen-flushed aluminized PET pouch along with an oxygen absorber packet.

Determination of Moisture: Moisture content of SRM 3530 was determined at NIST in duplicate samples from each of six bottles by heating in a thermogravimetric analyzer under an air atmosphere at (1) 110 °C for 3 h and (2) at 250 °C for 3 h. No mass loss was observed for SRM 3530 samples dried at 110 °C, indicating the material did not contain measurable moisture at the time of packaging. A discoloration was observed for SRM 3530 samples dried at 250 °C; drying at this temperature is not recommended. It is recommended that the user determine moisture content on test portions separate from those used for iodine content by heating the separate test portions for 3 h at 110 °C. An uncertainty component for the conversion to a dry-mass basis is not incorporated in the uncertainty of the reported certified value because there was no observed moisture in SRM 3530 at the time of value assignment.

Analytical Approaches for Determination of Iodide: Samples of SRM 3530 were assayed by the following methods. Results from the individual experiments were combined to form a method group mean.

Gravimetric Titrimetry: Samples were assayed for iodide by gravimetric titrimetry at NIST using a procedure adopted by the Association of Official Analytical Chemists (AOAC) [7] which is based on the Winkler titration for iodide [8]. Duplicate 10 g test portions of SRM 3530 from each of six bottles were accurately weighed and dissolved in water in pre-cleaned titration flasks. Gaseous bromine (Br₂) prepared from stoichiometric quantities of commercial bleach, potassium bromide and hydrochloric acid and transferred to the titration flask by use of a gas generator, was used to oxidize iodide to iodate. This was followed by iodimetric titration of the iodate with sodium thiosulfate. The sodium thiosulfate titrant was standardized against ground, dried potassium iodide that was assayed by high-accuracy titration against ceric ammonium nitrate that had previously been assayed by coulometry. Additional assays were performed by gravimetric titrimetry in a second experiment conducted eleven months later in order to assess material stability (see “Stability Assessment”).

⁽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.

Inductively Coupled Plasma Mass Spectrometry (ICP-MS): Samples of SRM 3530 were assayed by ICP-MS using the schemes listed below.

Quadrupole (Q) ICP-MS: Samples were assayed for total iodine by Q-ICP-MS at NIST using the standard additions method. Duplicate 0.25 g test portions of SRM 3530 from each of six bottles were accurately weighed into pre-cleaned jars and a ruthenium (Ru) internal standard was added. An additional set of 0.25 g test portions were accurately weighed into pre-cleaned jars and a known amount of SRM 3180 *Iodide Anion (I⁻) Standard Solution* (Lot No. 110530) was added along with the Ru internal standard, resulting in six iodine-spiked and six unspiked samples. Samples were diluted with Milli-Q water and placed in an ultrasonic bath for 10 min to facilitate complete dissolution of the salt material prior to ICP-MS analysis.

Sector ICP-MS: Samples were assayed for total iodine by sector ICP-MS at NIST using the standard additions method. Single 0.25 g test portions of SRM 3530 from each of six bottles were accurately weighed and processed as described above (see Q-ICP-MS).

Ion Chromatography (IC) Q-ICP-MS: Samples were assayed for iodide by IC/Q-ICP-MS at NIST using the standard additions method. Single 0.25 g test portions of SRM 3530 from each of six bottles were accurately weighed and processed as described above (see Q-ICP-MS). An isocratic chromatographic method utilizing a 50 mM nitric acid mobile phase at 1.5 mL·min⁻¹ was used to separate the iodine species with an IonPac AG11 (4 mm x 50 mm) guard column and a Dionex IonPac AS11 (4 mm x 250 mm) analytical column directly interfaced to the Q-ICP-MS. Iodide was the only iodine species observed in the chromatographic separations of the spiked and unspiked SRM 3530 test portions.

Linear Voltammetry: Samples were assayed for iodide by linear voltammetry at CENAM using the standard additions method in which the potassium iodide calibrator solution was added to the electrolyte in the electrochemical cell. The potassium iodide calibration solution was prepared gravimetrically from a concentrated solution of potassium iodide, which was standardized using gravimetric titration against a silver nitrate solution with potentiometric end-point detection. The silver nitrate solution was standardized with a solution of NIST SRM 999b *Potassium Chloride (Primary Chemical)*. Two sample preparation schemes were utilized:

Method A: Two salt samples per bottle were weighed and added directly to the electrochemical cell for analysis.

Method B: An aliquot of two gravimetrically prepared solutions from each bottle were added to the electrochemical cell for analysis.

Homogeneity Assessment: The homogeneity of iodine was assessed at NIST in duplicate samples from at least six bottles of SRM 3530 using the methods and test portion sizes described above. Analysis of the variance showed the between bottle heterogeneity to be negligible.

Stability Assessment: Samples of SRM 3530 from both unopened and previously opened bottles were assayed for iodide by gravimetric titrimetry at two time points eleven months apart. Single factor analysis of variance (ANOVA) for comparing results from the two time points resulted in $p = 0.79$ which may be taken as an indication of iodide stability for this material.

Certified Mass Fraction Value for Iodine as Iodide: The certified mass fraction value is a consensus mean from the mean of results from analyses provided by NIST and CENAM. Results from the same method group (gravimetric titrimetry, ICP-MS, and linear voltammetry) were first combined, using equal weights, to obtain mean values for each method group. These were then combined, again using equal weights, to yield an overall consensus mean. The provided uncertainty is an expanded uncertainty about this mean which is expected to cover the measurand with an approximately 95 % level of confidence. The expanded uncertainty is calculated as $U = ku_c$, where u_c incorporates the observed differences among the results from the methods and their respective uncertainties, consistent with the ISO/JCGM Guide, and k is a coverage factor corresponding to an approximately 95 % level of confidence [2–5]. The measurand in Table 1 is the total mass fraction of iodine (as iodide); metrological traceability is to the SI derived unit for mass fraction (expressed as milligrams per kilogram) on a dry-mass basis [9].

Table 1. Certified Mass Fraction Value for Iodine as Iodide in SRM 3530

	Mass Fraction (mg/kg)	Coverage Factor, k
Iodine (as Iodide)	52.2 ± 4.2	2.00

NOTICE TO USERS

NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of this SRM as a primary benchmark for the quality and accuracy of the user's in-house reference materials and working standards. As such, the SRM should be used to validate the more routinely used reference materials in a laboratory. Comparisons between the SRM and in-house reference materials or working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

REFERENCES

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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 <http://www.nist.gov/srm>.