National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3231

Iodine-129 Isotopic Standard (High Level)

This Standard Reference Material (SRM) is primarily intended for use in instrument calibrations and for evaluating the accuracy of mass spectrometry measurements of $^{129}I/^{127}I$ isotope ratios. A unit of SRM 3231 consists of five amber borosilicate ampoules containing approximately 5 mL of iodide solution (0.007 mol/L), together with sodium hydroxide and sodium sulfite preservatives; two ampoules of isotopic standard having a nominal $^{129}I/^{127}I$ ratio of 1×10^{-6}, two ampoules of isotopic standard having a nominal $^{129}I/^{127}I$ ratio of 1×10^{-8}, and one ampoule of blank iodide solution, which contains no added ^{129}I.

Certified Values: The certified isotope ratios of both levels of the standard are summarized in Table 1. The certified isotope ratios were calculated from the gravimetric combination of well-characterized sources of ^{129}I and natural ^{127}I and confirmed by Accelerator Mass Spectrometry.

Table 1. Certified Isotopic Compositions and Uncertainties for $^{129}I/^{127}I$ Isotopic Standards

Standard	Certified Value[a]
$^{129}I/^{127}I$ Isotope Ratio, Level I	$0.981 \times 10^{-6} \pm 0.012 \times 10^{-6}$
$^{129}I/^{127}I$ Isotope Ratio, Level II	$0.982 \times 10^{-8} \pm 0.012 \times 10^{-8}$

[a]The uncertainty of the certified value is ku_c, where k is the coverage factor for a 95 % confidence interval and u_c is the combined standard uncertainty calculated according to the ISO Guide [1].

Information Values: The isotope ratio of the blank solution and the density of the SRM solution are provided in Table 2 for information purposes only.

Table 2. Information Values for Isotopic Composition of Blank[b] and Density of the SRM Solution

$^{129}I/^{127}I$ Isotope Ratio, Blank	$16 \times 10^{-15} \pm 5 \times 10^{-15}$
Solution Density	1.000 ± 0.001 g/mL (21.2 ºC)

[b]The blank isotope ratio is an information value with estimated uncertainty provided by the collaborating laboratory.

Expiration of Certification: The certification of this SRM is valid until **31 March 2013**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. If substantive changes occur that affect the reference values before expiration, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

The overall technical direction and coordination of the preparation and measurements leading to the certification of this SRM were provided by S.E. Long of the NIST Analytical Chemistry Division.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 22 October 2003
See Certificate Revision History on Last Page

John Rumble, Jr., Chief
Measurement Services Division

This SRM was prepared in the NIST Analytical Chemistry Division by C.M. Beck, and by L.L. Lucas of the Radioactivity Group, Physics Laboratory. Confirmatory Accelerator Mass Spectrometry measurements of the isotope ratios were made by M.J. Bourgeois, D. Elmore, T. Kubley, and S. Ma of PRIME Lab, Purdue University, West Lafayette, IN.

Ampouling of this SRM was performed by M.P. Cronise of the NIST Measurement Services Division.

Statistical analysis was provided by S.D. Leigh of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

INSTRUCTIONS FOR USE

Radiological Hazard: An ampoule of SRM 3231 contains an extremely small amount of ^{129}I. The total activity is less than 0.1 Bq, and the material is therefore **NOT** considered to be radioactive. Transport of the material is **NOT** subject to DOT transport regulations for radioactive substances.

Chemical Hazard: Each ampoule of SRM 3231 contains sodium hydroxide at a concentration of approximately 0.01 mol/L of solution and sodium sulfite at a concentration of approximately 0.006 mol/L of solution. The solution is mildly corrosive. Contact with eyes or skin should be avoided. Use gloves when opening ampoules and manipulating contents. See "Instructions for Use".

Silica in the SRM Ampoule: The pH of solution in each ampoule is approximately 11, and will slowly etch small quantities of silica from the interior surface of the ampoule. The silica has a density greater than the density of the solution and will tend to settle at the bottom of the ampoule. Tests have shown that the silica does **NOT** affect the iodine content of the solution. If silica is likely to interfere with the measurement, the solution should be removed from the top of the ampoule, or filtered if the silica has been dispersed in the solution by movement of the ampoule.

Stability and Storage: This SRM should be stored at a temperature between 4 °C and 25 °C. It should **NOT** be frozen or exposed to sunlight or ultraviolet radiation.

Opened Ampoules: After opening the ampoule, the contents should be used immediately. Any unused material should be transferred to a tightly closed container, the headspace purged with an inert gas such as nitrogen or argon, and stored in a refrigerator. Teflon® containers are **NOT** recommended for this purpose.

Use: When opening ampoules, wear appropriate eye protection, gloves, and protective clothing. Check that all of the liquid has drained out of the neck of the ampoule. If needed, gently tap the neck to facilitate drainage. Open the ampoule by snapping off the top at the score line in the narrowest segment of the neck. It is advisable to wrap the neck of the ampoule with an absorbent paper towel prior to opening, in order to reduce the hazard from broken glass if the ampoule should break unevenly. Ampoules should not be resealed. Once opened, the contents of the ampoule should be used as soon as possible as the stability of the solution cannot be guaranteed. Transfer the solution from the ampoule using a suitable transfer pipette. **DO NOT PIPETTE BY MOUTH**. Pouring solution out of the ampoule is **NOT** recommended as the narrow cross section of the neck does not facilitate easy exchange of liquid and air.

PREPARATION AND CERTIFICATION[1]

Source of Material: Natural ammonium iodide was obtained from Deepwater Chemicals, Inc., Woodward, Oklahoma. The material was obtained from a "Deep Well" location, which minimized the amount of ^{129}I. The material, as well as the preservative reagents, sodium sulfite and sodium hydroxide, were screened for ^{129}I content by Accelerator Mass Spectrometry at PRIME Lab, Purdue University, West Lafayette, IN.

The ^{129}I stock solution was prepared by serial gravimetric dilution of NIST SRM 4949c *Iodine-129 Radioactivity Standard*. The concentration of the solution was calculated from the original massic activity, and confirmed by reverse isotope dilution inductively coupled plasma mass spectrometry (ID-ICP-MS) calibration using potassium iodide primary standard.

Preparation of Material: The isotopic mixtures were prepared by accurate gravimetric combination of a preserved *Woodward* ammonium iodide solution with a calibrated stock solution of SRM 4949c.

REFERENCES

[1] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland (1993): 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 http://physics.nist.gov/Pubs/.

Certificate Revision History: 22 October 2003 (Editorial changes); 10 July 2003 (Original certificate date).

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

[1] Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately 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.