U. S. Department of Commerce
Philip M. Klutznick
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards
Certificate
Standard Reference Material 4422L
Radioactivity Standard
Chlorine-36

This Standard Reference Material consists of chlorine-36 in 5.05604 grams of approximately 1.52 M hydrochloric acid in a flame-sealed borosilicate-glass ampoule. The density of this solution is 1.024 ± 0.002 g/ml at 24.4°C.

The beta-ray-emission rate of the chlorine-36 in β^- per second per gram of solution, in 1980, is

$$*4.146 \times 10^5 \pm 1.58\%*.$$

The beta-ray-emission rate of this Standard Reference Material was determined by comparative liquid-scintillation-counting measurements of dilutions of the master solution and Standard Reference Material 4943, chlorine-36 as sodium chloride. The standard, SRM 4943, had been calibrated by $4\pi\beta$ proportional counting.

The uncertainty in the beta-ray-emission rate of this Standard Reference Material, 1.58 percent, is the linear sum of 0.07 percent, half the 99-percent confidence interval of the mean for the comparative liquid-scintillation-counting measurements (4.604 times the standard error computed from five independent comparisons), and 1.51 percent, the overall uncertainty ascribed to SRM 4943. The overall uncertainty associated with the calibration of SRM 4943, 1.51 percent, is the linear sum of the following: half the 99-percent confidence interval of the mean for the $4\pi\beta$ measurements, 0.41 percent; background, 0.1 percent; dead time, 0.1 percent; stability, 0.2 percent; self absorption, 0.2 percent; film absorption, 0.1 percent; solution density, 0.1 percent; effect of any impurities, 0.1 percent; plateau, 0.2 percent.

The solution from which this Standard Reference Material was prepared was examined for photon-emitting impurities with a germanium-spectrometer system and cobalt-60 was found to be present. At 1444 EST February 5, 1980, the ratio of the beta-ray-emission rate of cobalt-60 to that of chlorine-36 was $2.2 \times 10^{-7} \pm 1.1 \times 10^{-7}$. Photons at any energy between 90 and 1900 keV with an emission rate greater than 10^{-4} that of the beta-ray-emission rate of the chlorine-36 should have been detected.

This Standard Reference Material was prepared in the Center for Radiation Research, Nuclear Radiation Division, Radioactivity Group, W.B. Mann, Principal Scientist.

Washington, D.C. 20234
April, 1980

George A. Uriano, Chief
Office of Standard Reference Materials

SRM 4422L-13

PRIME Lab Standards

Prime Lab	User	Ratio [xE-15]	Error [%]	Primary Source	Supplier	Comments	Status
Z91-0283	STD Cl 10000	10000		NBS	K. Nishiizumi		used up
Z91-02844	STD Cl 4000	4000		NBS	K. Nishiizumi		O.K.
Z91-0285	STD Cl 1600	1600		NBS	K. Nishiizumi		O.K.
Z91-0286	STD Cl 2003	2003		NBS	K. Nishiizumi	also: Z92-123	O.K.
Z92-0102	STD Cl 154000	154000	0.5	SRM 4422	NBS/K.N.		O.K.
Z92-0103	STD Cl 520	520	3.3	SRM 4422	NBS/K.N.		O.K.

PRIME Lab Standards

Prime Lab	User	Ratio [xE-15]	Error [%]	Primary Source	Supplier	Comments	Status
Z91-0281	STD Cl 10000	10000		NBS	K. Nishiizumi		used up
Z92-0282	STD Cl 4000	4000		NBS	K. Nishiizumi		O.K.
Z91-0283	STD Cl 1600	1600		NBS	K. Nishiizumi		O.K.
Z91-0286	STD Cl 2003	2003		NBS	K. Nishiizumi	also: Z92-123	O.K.
Z92-0104	STD Cl 154000	154000	0.5	SRM 4422	Vogt		O.K.
Z92-0103	STD Cl 520	520	3.3	SRM 4422	Vogt		O.K.
Z93-0001	STD Cl E-9	1442000		SRM 4422	Vogt		O.K.
Z93-0002	STD Cl E-10	282200		SRM 4422	Vogt		O.K.
Z93-0003	STD Cl-E-11	41640		SRM 4422	Vogt		O.K.
Z93-0004	STD Cl E-12a	4423		SRM 4422	Vogt		O.K.
Z93-0005	STD Cl E-12b	1203		SRM 4422	Vogt		O.K.
Z93-0006	STD Cl E-13	408		SRM 4422	Vogt		O.K.

} calibrated

E-9 & E-10 STD from NH4Cl 99.99% Aldrich kz Lot No 00524 HT
all others DY Lot No 06327DX

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SPACE SCIENCES LABORATORY
BERKELEY, CALIFORNIA 94720

Dec. 10, 1993

Dear Stephan,

I am enclosing certificate of 4422L.
I opened the ampule at May 19, 1983 and diluted.

original solution 5.0119 g → (0.5N HCl) dilute 59.0958 g

↑ 1st dilution
($\sim 1.3\times10^{-3}$ $^{36}Cl/Cl$)

2nd dilution
1.3715 g of 1st dil → (0.5N HCl) 80.4278 g
3.667 x 10⁴ dpm / 18.1 mg Cl / g soln.
(2.7×10^{-3})

3rd dilution
1.6174 g of 2nd dil. → (0.5N HCl) 79.6190 g
745.0 dpm / 18.1 mg Cl / g soln
($\sim 5.5\times10^{-5}$)

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SPACE SCIENCES LABORATORY
BERKELEY, CALIFORNIA 94720

at Nov. 29, 1991 I transfered 11.1659 g of 3rd dilution to an ampule and shipped to Purdue. the solution contains 763.7 dpm/g (~0.5N HCl). the concentration was different from original 3rd dilution (745.0 dpm) due to evaporation. (8 yrs)

The dilution (1st, 2nd and 3rd) was carried out by 0.5 N HCl. If I add Cl from original solution (1.52 M) and 0.5 N HCl, The third dilution (original) contains (745.0 dpm — 18.1 mg Cl — 5.5 x 10^{-5} ^{36}Cl/Cl).

The original calculation, I didn't add 1.52 M HCl, but the error is negligible after further dilution.

My old AMS ^{36}Cl stds ($\lesssim 10^{-10}$) which were used at Rochester since 1979 were calculated based on 3.0 x 10^5 y half-life. Later, I changed to 3.01 x 10^5 y half life. The calibration of my old UCSD stds and New UCSD-NIST standards (Sharma et al 1991) was used the half-life of 3.01 x 10^5 yrs

Sincerely yours.

Kuni

11/29/91

^{36}Cl standard from 3rd dilution

original NBS SRM4422L diluted 5/19/83

glass ampule

$1.74309 \cdot 10^{14}$ at ^{36}Cl / g $T_{1/2} = 3.01 \cdot 10^5$

11.1659 g soln. 763.7 dpm ^{36}Cl / g soln ($3.09607 \cdot 10^{20}$ at Cl^-/g)

0.018227 g Cl^-

($5.6_3 \times 10^{-7}$ $^{36}Cl/Cl$)

KUNIHIKO NISHIIZUMI

$\lambda_{36}(3.01) = 7.302177 \cdot 10^{-14}\ s^{-1}$

$= 4.3959 \cdot 10^{-12}\ min^{-1}$

$763.7\ dpm/g * 11.1659\ g * \frac{60\ sec}{min} = 5.116\ dps$

Cl-36

5.-2. = weight ampoule after transfer 12.52832

12.52832 − 12.30003 = 0.2280 ⇒

10.93765 + 0.2280 = 11.16565

PREPARED BY / DATE

kuni said 11.1659 g

I weighed open, dried ampul after months (diff. calibration? loss of glass?) 12.30033

1. Ampule closed 23.84914 ✓
2. Beaker empty 54.90610 ✓ } 78.75524

3. Apule i. beaker 78.75580 ✓

4. Ampule open i. beaker: 78.37207 ✓

5. Ampule open i. beaker after transfer: 67.43442 ✓ — Δ = 10.93765 g transfered taken

Δ: 10.93765 g ✓

4.a. PE bottle empty, no label, ~~with~~ cap 6.89263

Δ = 10.93507 ✓

4.b. Bottle no label with cap after transfer 17.82770 — Δ = 10.93507 g transfered received

---

I. 1. Dilution: PE 1-l bottle: + 67.8449 g dried NH_4Cl 99.999%

MW NH_4Cl 53.463

67.8449 g NH_4Cl ✓ (PE bottle: 17.82770; after transf: 17.15003; .67767) + 0.674? g of undiluted solution (kuni 10^{-7})

Cl^- (NH_4Cl) = 66.31%

0.674? g kuni

994.848 g H_2O + 92.6000 ✓

$7.642006 \cdot 10^{23}$ at Cl^- + 92.1356 ✓

^{36}Cl $T_{1/2} = 3.01 \times 10^5$ a

67.845 * .6631 = 44.99025 g Cl^- + 90.3966 ✓

$\lambda_{36} = 4.381306 \cdot 10^{-12}$ min^{-1}

0.674 ≙ 514.7 dpm ^{36}Cl ≙ $1.174841 \cdot 10^{14}$ at ^{36}Cl + 93.7538 ✓

994.848 + 92.1041 ✓

Σ_{TOT}: 1,063.367 g

$$\frac{36}{Cl} = \frac{1.174841 \cdot 10^{14}}{7.642006 \cdot 10^{23}} = 1.537_{35} \cdot 10^{-10}$$ + 94.5720 ✓

+ 90.7400 ✓

Ratio: $1.537_{35} \cdot 10^{-10}$ 36/S/S + 94.1145 ✓

$^{36}Cl/g$: $1.17848 \cdot 10^{14}$ / 1,063.37 g = $1.1049 \cdot 10^{11}$ at/g soltn + 92.6805 ✓

+ 92.4407 ✓ — 17.4275

+ 86.7378

PAGE NO. 2

02-21-92

PREPARED BY

DATE

SUMMARY

±0.1%	67.845 g NH_4Cl	≙ 44.99 g Cl^- = $7.642 \cdot 10^{23}$ at Cl^-
±0.6%	0.674 g kumi E-7	≙ 514.7 dpm ^{36}Cl ⇒ ($\lambda = 4.3813 \cdot 10^{-12}$) $1.1748_4 \cdot 10^{14}$ at ^{36}Cl
± 0.1%	994.848 g H_2O	

bottle empty ~97.… g

1063.3_7 g solution

containing 44.99 g Cl^- ≙ $7.642 \cdot 10^{23}$ at Cl^-

514.7 dpm ^{36}Cl ≙ $1.1748 \cdot 10^{14}$ at ^{36}Cl

RATIO: $\frac{36}{Cl^-} = 1.537_{35} \cdot 10^{-10}$

A : $1.1049 \cdot 10^{11}$ at $g^{-1}_{solution}$

B : $2.611_3 \cdot 10^{12}$ at ^{36}Cl / g Cl^-

C : $4.23 \cdot 10^{-2}$ g Cl^- / g solution = 42.31 mg Cl^- / g soln

executive

743-1122
Lisa Gordinchee

PAGE NO. 3

STD 520 ± 0.6% 3.ck

PREPARED BY SU
DATE 02-21-92

Dilution to STD 500 (3658)

2.9575 g of solution I (E-10) ✓ $\hat{=} 1.1748 \cdot 10^{14}$ at ^{36}Cl * $\frac{2.9575g}{1063.37g}$ = $3.267_4 \cdot 10^{11}$ at
60.6290 g NaCl ✓ $\hat{=} 36.780 \cdot$ at Cl^- * $\frac{35.453gl}{mol}$ * N_A = $6.247_4 \cdot 10^{23}$ at
954.32 g H_2O
Σ 1017.9 g of solution ✓ (92.64 + 92.19 + 90.84 + 92.66 + 93.71 + 93.21 + 94.10 + 93.14 + 91.83 + 87.56 + 32.44)

1017.9g solution

containing. RATIO: $\frac{3.27 \cdot 10^{11}}{6.25 \cdot 10^{23}}$ = 5.2300×10^{-13} STD 523

A : $3.20955 \cdot 10^{8}$ at ^{36}Cl / g solution

B : $8.88_3 \cdot 10^{9}$ at / g Cl^-

C : 36.13 mg Cl^- / g solution

Ray Middleton
$T_{1/2}$ =

→ 146 mg AgCl / g solution

executive

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SRM 3230 - Iodine-129 Isotopic Standard (Low Level)

ordered 11/18/05

Related Materials: 104.9 - Stable Isotopic Materials (solid and solution forms)
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Description:	Iodine-129 Isotopic Standard (Low Level)
Unit Price*:	$549.00
Unit of Issue:	5x5 mL
Status:	Now Selling
Expiration Date:	3/31/13
Lot:	

Shipping Information:

Perishable:	No
Hazardous:	No
Hazardous Shipping Code:	N/A

Documentation:

Certificate Date:	10/22/03
MSDS Date:	N/A
Data Updated:	4/12/05

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Technical Contacts by Category

Keyword(s):
Iodine, Isotopic

Date created: 3/10/2002
Last updated: 8/28/2005

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3230

Iodine-129 Isotopic Standard. (Low 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 3230 consists of five amber borosilicate glass ampoules containing approximately 5 mL of iodide solution (0.007 mol/L); two ampoules of isotopic standard having a nominal $^{129}I/^{127}I$ ratio of 5 x 10^{-10}, two ampoules of isotopic standard having a nominal $^{129}I/^{127}I$ ratio of 1 x 10^{-12}, and one ampoule of blank iodide solution, which contains no added ^{129}I.

5049

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	4.920 x 10^{-10} ± 0.062 x 10^{-10}
$^{129}I/^{127}I$ Isotope Ratio, Level II	0.985 x 10^{-12} ± 0.012 x 10^{-12}

[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 × 10^{-15} ± 5 x 10^{-15}
Solution Density	1.000 ± 0.001 g/mL (21.2 °C)

[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 3230 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 3230 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.

REFERENCE

[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).

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[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.