National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 3144

Spectrometric Standard Solution

Rhodium

Batch Code 490906

This Standard Reference Material (SRM) is intended for use in atomic absorption spectrometry, inductively-coupled plasma optical emission spectrometry, spectrophotometry, or any other analytical technique that requires aqueous standard solutions for calibrating instruments. SRM 3144 is a single element solution prepared gravimetrically with a rhodium salt in 10 percent (V/V) hydrochloric acid. The certified value is based on the gravimetric preparation of the solution and the assay of the rhodium salt. The density of the solution at 22 °C is 1.0177 g/mL.

Metal	Concentration (mg/mL)	Source Purity, %	Acid Conc. (V/V) Approximate
Rh	0.9996 ± 0.0011	$(NH_4)_3RhCl_6$*	HCl, 10%

*This material was reduced to rhodium metal which was then analyzed by direct current arc optical emission spectrometry and found to contain 85 μg/g Ir, 20 μg/g Pt, 70 μg/g Ru, 60 μg/g Si, and 10 μg/g Ag.

The uncertainty in the certified value is calculated as

$$U = ku_c$$

where u_c is the combined standard uncertainty calculated according to the ISO Guide [1]. The value u_c is intended to represent, at the level of one standard deviation, the combined effects of any errors attributable to weighing and uncertainty associated with the dilution as well as those associated with the gravimetric assay of the ammonium hexachlororhodate salt for percent rhodium.

SRM 3144 was prepared by T.A. Butler of the NIST Inorganic Analytical Research Division. The gravimetric assay procedures were performed by C.M. Beck II of the NIST Inorganic Analytical Research Division. Direct-current arc spectrometry analyses were performed by C.L. Maul, formerly of Ledoux & Co., Teaneck, NJ, and oxygen and nitrogen analyses were performed by L.W. Ollila and M.P. Ollila of Luvak Inc., Boylston, MA. Technical advice on the gravimetric assay procedures for the rhodium salt was given by P. Blumberg of Ledoux & Co. and E.W. Hobart, Jr., formerly of Ledoux & Co.

The technical aspects involved in the preparation and certification of this SRM were coordinated by R.L. Watters, Jr. of the NIST Inorganic Analytical Research Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.S. Kane

Gaithersburg, MD 20899
August 11, 1994

Thomas E. Gills, Chief
Standard Reference Materials Program

(over)

Procedures for Use

Stability: This certification is valid for one year from the shipping date provided the bottle is kept tightly capped and stored under normal laboratory conditions. Before recapping, the user should carefully remove any solution adhering to the bottle lip and the inside of the cap with a clean wipe. NIST will monitor the stability of representative solutions from this SRM lot and if any changes occur that invalidate this certification, NIST will notify purchasers.

Preparation of Working Standard Solutions: All materials used in the preparation of working standard solutions should be brought to 22 $\pm$1 °C before use and all glass or plastic surfaces coming into contact with the materials must have been previously cleaned. A working standard solution can be prepared from the SRM solution by serial dilution. Dilutions should be made with certified class A volumetric flasks and 5 mL or 10 mL class A pipets. All volumetric transfers of solutions should be performed using a proven analytical technique. Each dilution should be acidified with an appropriate high-purity acid and diluted to calibrated volume using high-purity water. The stability of the working standard solution will depend on the final acid concentration; therefore, care should be exercised to ensure that the final acid concentration of the dilution closely approximates that of the SRM. To achieve the highest accuracy, the analyst should prepare daily working solutions from 100 μg/mL dilutions of the original SRM solution.

The water-soluble rhodium salt used to prepare this solution standard was gravimetrically assayed for rhodium by first oxidizing the salt to rhodium oxide and then reducing the oxide to rhodium metal with hydrogen in a Rose crucible. Impurities were determined in the resulting rhodium metal and an appropriate deduction made. Independently, high-purity rhodium metal was used to prepare a different water-soluble rhodium salt by chlorination of the metal and subsequent reaction with sodium chloride and chlorine in a tube furnace. Inductively-coupled plasma emission spectrometric comparisons of a previous batch of rhodium solutions prepared from each salt were shown to be indistinguishable to 0.2% relative. The details of these procedures are published in reference [2].

REFERENCES

[1] "*Guide to the Expression of Uncertainty in Measurement*", ISBN 92-67-10188-9, 1st Ed. ISO, Switzerland, 1993.

[2] C.M. Beck II, M.L. Salit, R.L. Watters, Jr., T.A. Butler, and L.J. Wood, "The Preparation and Certification of a Rhodium Standard Reference Material Solution," *Anal. Chem.*, **1993**, *65*, 2899-2902.