



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material 3144

#### Spectrometric Standard Solution

#### Rhodium

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 to contain 1.000 mg/mL of rhodium with a hydrochloric acid concentration (V/V) of 10 percent. The certified value is based on gravimetric procedures i.e., weight per volume composition of the high-purity rhodium salt dissolved in NIST high purity reagents.

Metal	Concentration <sup>a</sup> (mg/mL)	Source Purity, %	Acid Conc. (V/V) Approximate
Rh	1.000 ± 0.005	(NH <sub>4</sub> ) <sub>3</sub> RhCl <sub>6</sub> *	HCl, 10%

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

<sup>a</sup>The uncertainty listed for an element is based on judgement and represents an estimate of the combined effects of any errors, attributable to weighing, dilutions, interelement effects, and purity of the metal or compound. (No attempt was made to derive exact statistical results as the imprecisions of most analytical methods are much larger than the errors listed above).

#### Procedures for Use

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

**Preparation of Working Standard Solutions:** All materials used in the preparation of working standard solutions should be brought to 22 ± 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.

Gaithersburg, MD 20899  
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William P. Reed, Chief  
Standard Reference Materials Program

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

**Notice to Users:** The same acid mixture listed on this SRM certificate should be used in making appropriate dilutions and working standards. For some instrumental techniques, small differences between the standard and the sample in acid type and/or acid concentration may lead to erroneous results.

SRM 3144 was prepared by T.A. Butler of the NIST Inorganic Analytical Research Division. Inductively-coupled plasma optical emission spectrometry analyses and gravimetric analyses were made by C.M. Beck II, M.L. Salit, and L.J. Wood of the NIST Inorganic Analytical Research Division. Direct-current arc spectrometry analyses were performed by C.L. Maul 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 and E.W. Hobart, Jr., of Ledoux & Co.

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 spectrometry comparison of 1,000  $\mu\text{g/mL}$  rhodium solutions prepared from each salt were indistinguishable to 0.2% relative.