National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3140

Platinum (Pt) Standard Solution

Lot No. 140930

This Standard Reference Material (SRM) is intended for use as a primary calibration standard for the quantitative determination of platinum. A unit of SRM 3140 consists of five 10 mL sealed borosilicate glass ampoules of an acidified aqueous solution prepared gravimetrically to contain a known mass fraction of platinum. The solution contains hydrochloric acid at a volume fraction of approximately 10 %, which is equivalent to a concentration (molarity) of approximately 1.2 mol/L.

Certified Value of Platinum: 9.996 mg/g ± 0.017 mg/g

The certified value is based on (1) gravimetric preparation using high-purity platinum and (2) inductively coupled plasma optical emission spectrometry (ICP-OES) calibrated using six primary standards independently prepared from high-purity platinum [1,2].

The uncertainty associated with the certified value, stated as a symmetric interval with a level of confidence of 95 %, was evaluated in accordance with Supplement 1 to the ISO/JCGM Guide [3]. The uncertainty can be expressed as:

$$U = ku_c$$

where $k = 1.98$ is the coverage factor for a 95 % confidence interval and 107 effective degrees of freedom. The quantity u_c is the combined standard uncertainty which represents, at the level of one standard deviation, the combined effect of uncertainty components associated with the gravimetric preparation, the ICP-OES determination, any difference between the methods' results, and stability of the actual platinum mass fraction.

Expiration of Certification: The certification of **SRM 3140 Lot No. 140930** is valid, within the measurement uncertainty specified, until **30 July 2027**, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Storage, Handling, and Use"). This 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 SRM 3140 Lot No. 140930 was provided by J.L. Molloy of the NIST Chemical Sciences Division.

This SRM was prepared by T.A. Butler of the NIST Chemical Sciences Division. The ICP-OES analysis was performed by J.L. Molloy and T.A. Butler using primary calibration standards prepared by B.R. Norman of the NIST Chemical Sciences Division.

Statistical consultation was provided by A. M. Possolo of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 16 October 2015

Robert L. Watters, Jr., Chief
Office of Reference Materials

METROLOGICAL TRACEABILITY

Metrological traceability of measurement results to a given reference must be established through an unbroken chain of calibrations and/or comparisons, each having stated uncertainties [4], using measurement standards that are appropriate for the physical or chemical property being measured. Comparisons may include validation measurements using various spectroscopic, chromatographic or classical methods of analysis. Gravimetric or volumetric dilution is also a method of comparison, where the mass or volume of a solution before and after dilution is measured.

For this SRM, the measurand is the total concentration of platinum, expressed as mass fraction and the certified value is metrologically traceable to the SI unit for mass. This SRM can be used to establish traceability of the results of platinum measurements to NIST measurement results and standards. One approach is to calibrate analytical instruments or procedures for the determination of platinum using standards whose values are traceable to the certified value of platinum in this SRM. When the traceable values of such standards are assigned using this SRM for calibration, the uncertainties assigned to those values must include the uncertainty of the certified value of this SRM, appropriately combined with the uncertainties of all calibration measurements.

INSTRUCTIONS FOR STORAGE, HANDLING, AND USE

CAUTION: This SRM is an acid solution contained in tip-sealed borosilicate glass ampoules with pre-scored stems. Therefore, all appropriate safety precautions, including use of gloves during handling, should be taken. Unopened ampoules should be stored under normal laboratory conditions in an upright position inside the original container supplied by NIST.

Opening an Ampoule: When an ampoule is to be opened, that area of the stem where the pre-scored band is located (≈ 5 mm below the encircling metallic band) should be carefully wiped with a clean, damp cloth and the body of the ampoule wrapped in absorbent material. Then holding the ampoule steady and with thumb and forefinger grasping the stem at the metallic band, **minimal** thumb pressure should be applied to the stem to snap it. Correctly done, the stem should break easily where pre-scored. Use of a metal file to break the stem is **NOT** recommended.

Working Standard Solutions: After opening the ampoule, the entire contents should be transferred immediately to another container and *working standard solutions* should be prepared. Working standard solutions in the range of 10 mg/kg to 100 mg/kg are recommended, from which more dilute standards can be prepared. The user should establish internal laboratory procedures that specify a maximum shelf life for a working standard solution. Two procedures for the preparation of working standard solutions follow.

Preparation of Working Standard Solutions by Mass: Each working standard solution should be prepared by emptying one or more ampoules of the SRM into an empty, dry, preweighed, polyethylene bottle and then reweighing the bottle. An appropriate dilute acid must be added by mass to bring the solution to the desired dilution. The dilution need not be exact since the mass of the empty bottle, mass of the bottle plus SRM aliquot, and the final diluted mass of the solution will permit calculation of the exact mass fraction (mass of platinum per mass of solution) of the working standard solution. Dilutions prepared gravimetrically as described will need no correction for temperature and no further correction for true mass fraction in vacuum.

Preparation of Working Standard Solutions by Volume: Volumetric dilutions are **NOT** recommended due to uncertainties in volume calibrations and variations in density. However, for user convenience, a procedure for volumetric preparation that will minimize the major sources of error is given. Each working standard solution should be prepared by emptying one or more ampoules of the SRM into an empty, dry, polyethylene bottle and then weighing the bottle. The solution must now be transferred to a Class A volumetric flask and the polyethylene bottle reweighed to determine the exact mass of SRM solution transferred. The solution in the flask is then diluted to 99 % + volume using an appropriate dilute acid, mixed thoroughly, and the remaining few drops needed to dilute to exact volume carefully added. The concentration (in milligrams per milliliter) of the resulting working standard solution can then be calculated by multiplying the mass (in grams) of the SRM solution amount by the SRM certified value (in milligrams per gram) and dividing the numerical product by the calibrated volume (in milliliters) of the flask used for dilution. If this procedure is followed, no correction for density is needed. Although the concentration of the resulting working standard solution may be an uneven fraction of the original SRM concentration, it will be known as accurately as a volumetric dilution permits.

Possible Presence of Other Elements: Studies conducted by NIST have shown that components of borosilicate glass ampoules may leach into solution. In *undiluted* solutions, Si and Na mass fractions as large as 20 mg/kg, B and La mass fractions in the range 1 mg/kg to 5 mg/kg, and Mg, Al, Mn, As, Ce, Zn, Rb and Ca mass fractions in the range 0.05 mg/kg to 1 mg/kg have been found. When diluted to prepare working standard solutions, the levels of these elements become negligible for most purposes. Nevertheless, possible effects should be considered when this SRM is used.

REFERENCES

[1] Rukhin, A.L.; *Weighted Means Statistics in Interlaboratory Studies*; Metrologia, Vol. 46, pp. 323–331 (2009).

[2] DerSimonian, R.; Laird, N.; *Meta Analysis in Clinical Trials*; Control. Clin. Trials, Vol. 7, pp. 177–188 (1986).

[3] JCGM 101:2008; *Evaluation of Measurement Data — Supplement 1 to the "Guide to the Expression of Uncertainty in Measurement" — Propagation of Distributions using a Monte Carlo Method*; Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Oct 2015).

[4] JCGM 200:2012; *International Vocabulary of Metrology - Basic and General Concepts and Associated Terms (VIM)*, (2008 version with Minor Corrections); 3rd ed.; JCGM (2012); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_200_2012 (accessed Oct 2015).

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