THE EXCEPTIONAL QUALITY OF

Primary Certified Reference Materials (PCRM™s)

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Our Challenge

At Inorganic Ventures, we are excited to announce the development of a completely new product line: Primary Certified Reference Materials (PCRM™s). The first questions that you might ask are: "What are Primary Certified Reference Materials and how do they differ from the Certified Reference Materials (CRMs) currently available from Inorganic Ventures?" These are valid and valuable questions that we will answer below along with presenting information on reference material traceability.

We created the PCRM product line because for certain elements, like osmium (Os), iridium (Ir) and ruthenium (Ru), there are no Standard Reference Materials available from our National Metrology Institute (NMI), the National Institute of Standards and Technology (NIST). After investigation, to the best of our knowledge, there is no NMI worldwide, that has the equivalent of NIST SRMs for those elements. Dr. Paul Gaines, the Founder and Chairman of Inorganic Ventures, has long been concerned with the lack of primary standards against which to test our solution standards of Os, Ir and Ru. So, we set out to formulate these primary standards ourselves.

Background

The definition of a Primary Standard comes from the International Vocabulary of Metrology (VIM) which in turn comes from the source of all metrological philosophy and practice, the International Bureau of Weights and Measures (BIPM[1]), which makes its home in Sevres, Saint-Cloud, Paris. The BIPM was founded on May 20, 1875, after the signing of the Meter Convention, which was a treaty, creating the basis for international agreement on units of measure. That was at the time of the industrial revolution, when new modes of manufacturing were expanding, and railroads had already become important means of conveying materials and people. Uniform units of distance, weight, and time became increasingly important to support the manufacture of goods and their distribution across great distances.

In 1960, the International System of Units (SI)[2] was established by the governing body of the BIPM. The SI became the preferred system of units and created an international language for the communication of measurement information. There are seven base units defined by the SI[3].

BASE QUANTITY	BASE UNIT	SYMBOL
Time	Second	s
Length	Meter	m
Mass	Kilogram	kg
Electric Current	Ampere	A
Thermodynamic Temperature	Kelvin	K
Amount of Substance	Mole	Mol
Luminous Intensity	candela	cd

The SI base unit, the kilogram, serves as the focus of our work in the PCRM product line.

Primary Certified Reference Materials vs. Certified Reference Materials

So, returning to our question: "How do Primary Certified Reference Materials (PCRMs) differ from the Certified Reference Materials (CRMs) currently available from Inorganic Ventures?" The short answer is that PCRMs achieve a higher level of metrological traceability than CRMs. Metrological traceability is a basic requirement for establishing an accurate value for a Certified Reference Material. Specifically, for a Certified Reference Material manufacturer, ISO:17034 and ISO:17025 standard accreditation is essential, because those certifications tell our customers that we have the highest accuracy CRMs available. The claim to the highest level of accuracy is possible because ISO:17034 and ISO:17025 require the measurement results for CRMs to be traceable to the International System of Units (SI) by a documented, unbroken chain of comparisons, where each of these comparisons contribute to the measurement uncertainty[4].

For the majority of our CRMs, we gain the SI traceability by analyzing our products directly against NIST SRMs, that trace their certified values and uncertainties directly to the SI. NIST SRMs are primary measurement standards[5], meaning, they establish their certified values through primary reference measurement procedures[6]. A primary reference measurement procedure is one that produces measurement results without reference to another measurement standard of the same kind. We needed to create the SI traceability for Os and Ir ourselves, because no NMI has SI traceable solution standards for these elements. Up until now, no CRM producer could rigorously fulfil the definition of a CRM for Os and Ir because no one has demonstrated the SI traceability of their solution standards by a primary method.

Establishing Traceability to the SI

We established the SI traceability of our new Os and Ir PCRMs by tracing the mass fraction of the elements in their salts, directly to the kilogram base unit of the SI. Weighing is a primary method, because the weights of the salts and the metals, i.e., the measurement results, were obtained without reference to another measurement standard of the same kind. This procedure makes the Os and Ir PCRMs primary reference materials because they were traced to the SI through a primary measurement procedure, gravimetry, along with additional requirements which will be discussed later. As mentioned earlier, most CRMs, that have the ISO:17034 and ISO:17025 standard accreditation, gain their SI traceability through the analysis of the CRM against a NIST SRM. NIST SRMs are traced to the SI through primary reference measurement procedures. When a CRM is traced to the SI through a NIST SRM, the SI traceability chain of the CRM becomes longer, because you now have the intermediate SRM between the CRM and the SI. That process makes the CRM a secondary measurement standard[7] because its certified value is traced to the SI through the primary measurement standard, the NIST SRM. See **Figure 1**.

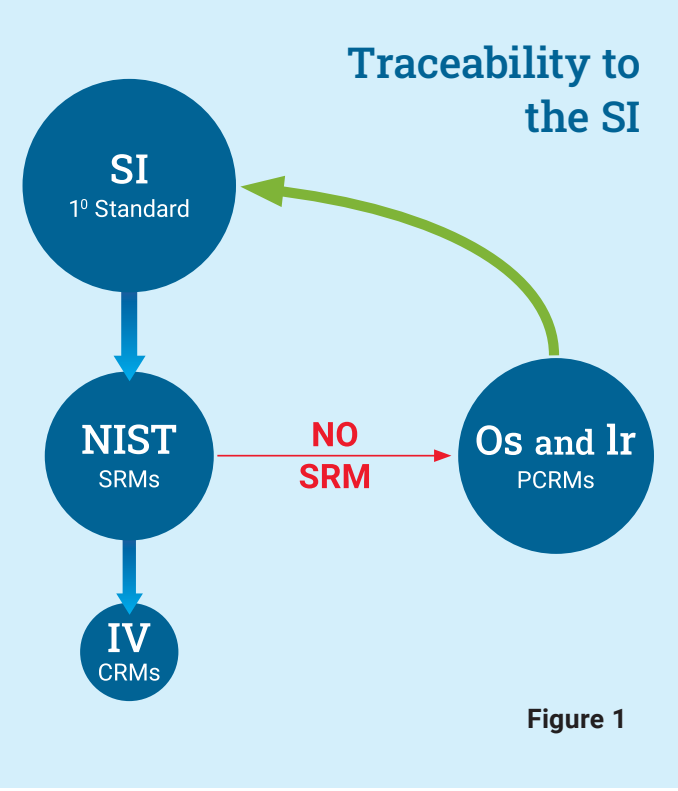

Figure 1

Historically at Inorganic Ventures, we have had ISO:17034 and ISO:17025 standard accreditation for our Ir CRMs as a result of a gravimetric assay performed on an Ir standards solution back in 2001. The current work on the PCRM product line has yielded far more rigorous gravimetric assay methods than previously performed.

Gravimetric Assays & Purity Analysis

Our Ir PCRM and Os PCRM both established SI traceability following the guidance provided by the work of Charles Beck, Marc Salit, and their associates at NIST, in developing the SRM for Rh in 1993[8]. For the Os and Ir PCRMs, determining the mass fraction of each element in the salts by the primary gravimetric method is the foundation of the SI traceability for those elements, but there is more work needed to fulfil the requirements for establishing SI traceability. In the NIST document, *SI Traceability and Primary Standards*[9], it is stated, "Results from a series of measurements can be linked to the SI through an unbroken chain of comparisons, i.e., calibrations. At the apex of such a calibration hierarchy are accurate mass determinations of calibrants and purity assessments that realize SI units."

The purity assessments of the Os and Ir starting materials are an essential component in establishing the SI traceability of these elements. The purity assessments were accomplished by performing trace metallic impurities (TMI) analysis on solutions of the metal salts on ICP-OES and ICP-MS. Inert gas fusion (IGF) testing provided data on the O, H, and N content of the salts. The combined purity data from these analyses gave us the needed information to quantify the purity of the salt.

The story does not end there. It is necessary to confirm the accuracy of the gravimetric determination of the metal mass fractions. Solutions of the potential SI traceable solution standards were made from the salts, based on the gravimetrically determined metal mass fraction that had been corrected for impurities. As a standard for comparison a separate solution made from the high purity metal of that element was put into solution. Purity assessments were also performed on the high purity metal. The comparison of these two solutions from different sources of the metal provided confirmation that the concentration of the SI traceable solutions of Os or Ir, made from their respective salts, were accurate. See **Figure 2**, pg.4.

There is one more piece of information that tells us about the value of the PCRMs. As mentioned earlier, having a solution standard with direct traceability to the SI, by a primary method, means the chain of comparisons for the PCRM has only one link! The more links you add to a chain of comparisons, the greater the uncertainty in your result, because each link in the chain adds another uncertainty component to the final result. The smaller the uncertainty, the greater confidence you will have in your result.

The details of these processes get quite involved, so they will not be discussed here. You may read about these details in the paper, *Tracing an Osmium Solution Standard to the International System of Units (SI)*, Madeline Gozzi, Paul Gaines, Thomas Kozikowski, and Brian Alexander.
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Conclusions

In summary, Os and Ir PCRMs are, to the best of our knowledge, the only rigorously developed SI traceable solution standards for these elements. These solution standards, with SI traceability by means of a primary method, give the analyst the highest degree of confidence in their results. In fact, PCRMs can be used with the same degree of confidence you have when using solution standards from National Metrology Institutes.

[1] https://www.bipm.org/en/
[2] https://www.bipm.org/en/measurement-units
[3] The SI Brochure, The International System of Units (SI), 9th Edition, 2019. https://www.bipm.org/en/publications/si-brochure
[4] ISO17025, Metrological Traceability, Sections 6.5.1, and 6.5.2
[5] VIM_JCGM_200_2012, section 5.4
[6] VIM_JCGM_200_2012, section 2.8
[7] VIM_JCGM_200_2012, section 5.5
[8] Beck, C. M., II; Salit, M. L.; et al. Anal. Chem. 1993, 65, 2899–2902.
[9] https://www.nist.gov/mml/csd/organic-chemical-metrology/primary-focus-areas/fundamental-chemical-metrology/si

The Steps in Development of a PCRM™

Figure 2