

NO MORE TEARS OVER  
TRACE ELEMENTS:  
How Our PCRMs  
Keep Os, Ir, and Ru  
in Line

THURSDAY, MARCH 30 | 9:00–9:30AM EST

PRESENTED BY:

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INORGANIC VENTURES  
2023 WEBINAR SERIES

**BE BOLD**

# What is different about Os, Ir, & Ru?

- No National Metrology Institute has SI traceable solution standards for these elements.
- SI traceability is critical to Inorganic Ventures as an ISO:17025 & ISO:17034 accredited Certified Reference Material (CRM) manufacturer.
- Creating a **primary SI traceable solution standard** for these elements will ensure the accuracy of our CRMs.



# Introducing IV's Primary Certified Reference Materials (PCRM<sup>TM</sup>s)

- The PCRM<sup>TM</sup> Product Line will differentiate **Primary SI Traceable Solution Standards** from our normal CRMs.
- These PCRM<sup>TM</sup>s may not be applicable to every use, but they are designed for users that need extreme levels of accuracy.



# What is a Primary Solution Standard?

1. Measured value is obtained by means of a primary reference measurement procedure. <sup>A, B</sup>
2. Gravimetry, or measurement by weight, is a primary SI traceable measurement procedure.

A. VIM\_JCGM\_200\_2012, section 5.4

B. VIM\_JCGM\_200\_2012, section 2.8

# What is SI Traceability?

Your measured value is linked or traced to the International System of Units, the SI.



The SI was established in 1960.



There are 7 base units in the SI.

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

# Why Does Traceability Matter?



To maintain clear communication about your results.



Accuracy, Accuracy, Accuracy




*Always try to **shorten** your chain of traceability.*

# How Do PCRM<sup>TM</sup>s Establish SI Traceability?

Our primary measurement procedure was **Gravimetry**, or measurement by weight.



The kilogram (kg) is one of the 7 base units of the SI.



Thus, we obtain traceability to the SI through the kg.

# What Are The Requirements for SI Traceability?

Primary  
Measurement  
Procedure

Purity Analysis of the  
Candidate Starting  
Materials



# Experimental Design – Primary Measurement Procedure

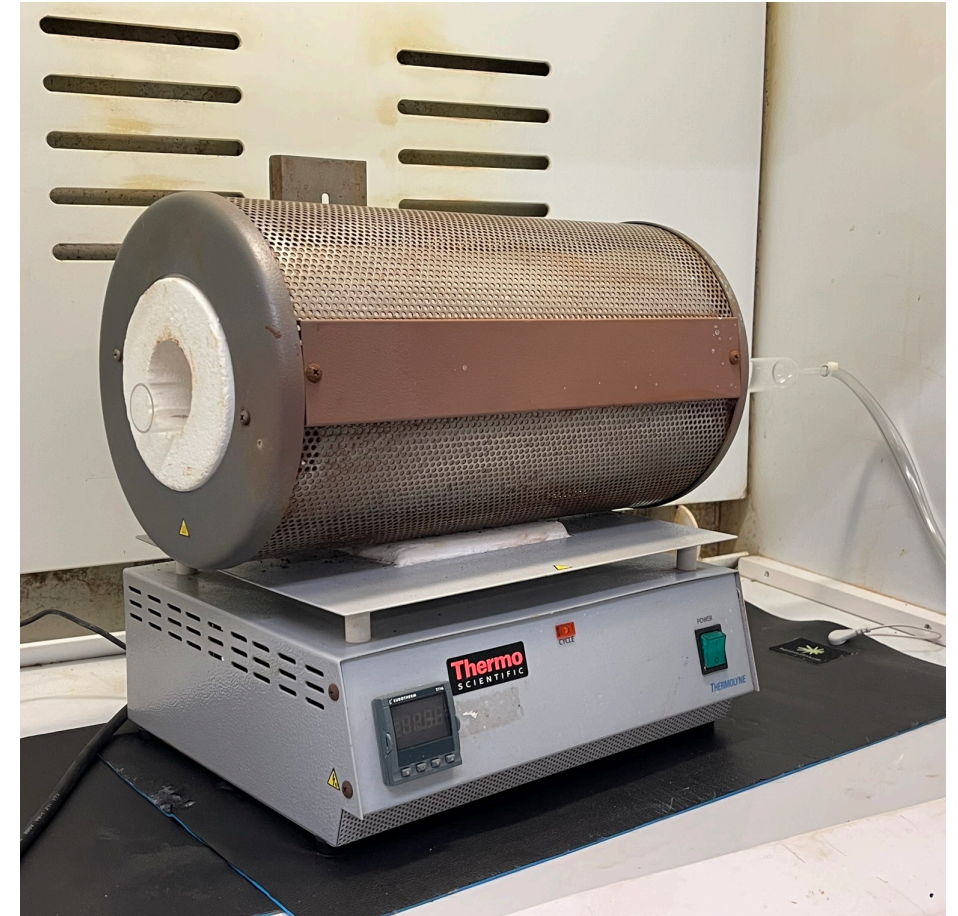
- We followed the approach used by Beck, Salit et al. at NIST when they developed the Rh SRM.<sup>1</sup>
- We needed to determine the Os or Ir mass fraction in the candidate metal salt.
  - Gravimetric Reductions of the salt to the metal
  - Establishes SI traceability of the candidate starting material

<sup>1</sup>Beck, C. M., II; Salit, M. L. Preparation and Certification of a Rhodium Standard Reference Material Solution. Anal. Chem. 1993, 65, 2899–2902, <https://doi.org/10.1021/ac00068a030>



# Gravimetric Reductions

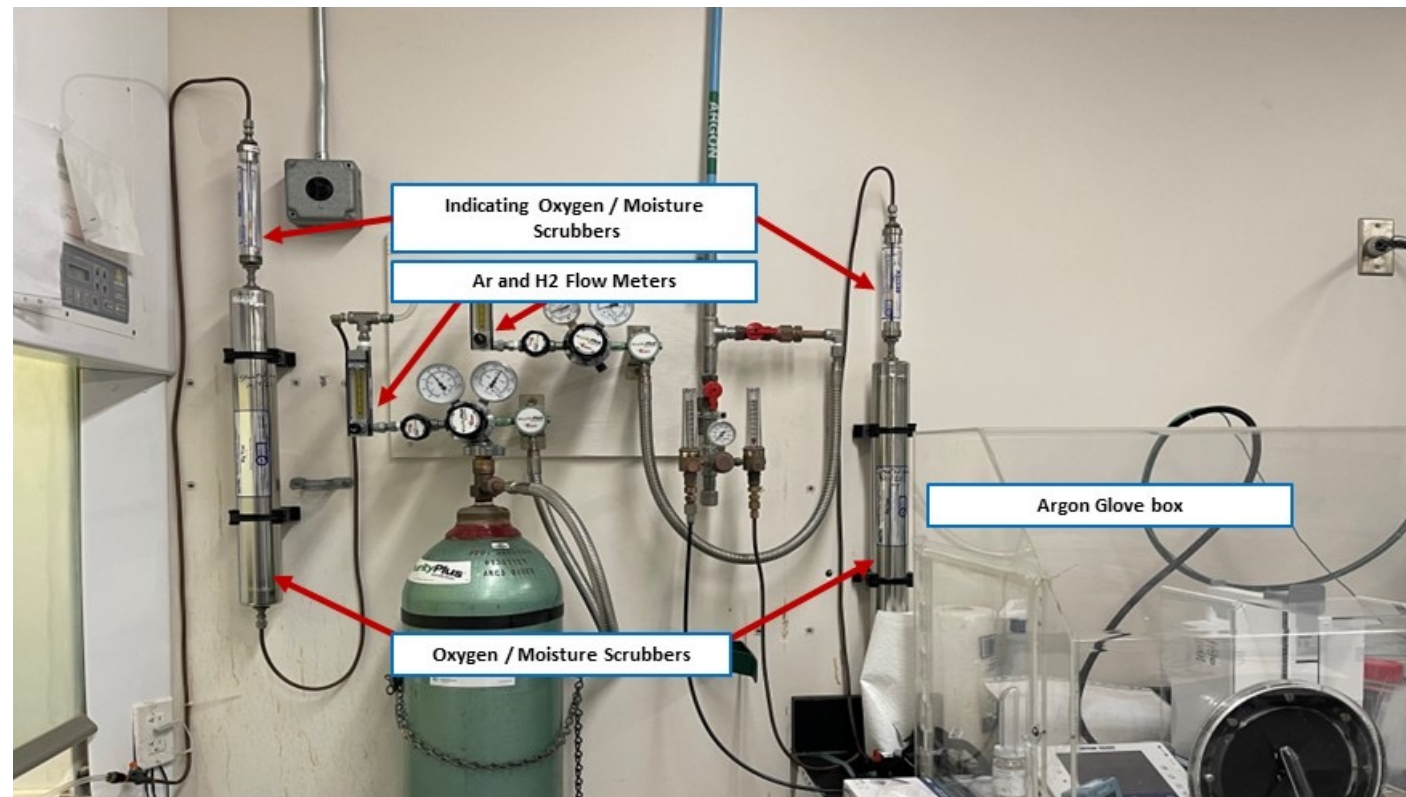
- Reduce the starting material (Os or Ir salt) to the metal under  $\text{H}_2$  at high temperature.
- SI Traceability partially achieved through weighing the salt and metal gravimetric reduction products.
  - This determines the mass fraction of metal in the salt.



# Eliminating Systematic Errors

- Major Systematic Errors

- Exposure to air/moisture
- Static



# What Are The Requirements for SI Traceability?

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# Starting Material Purity Analysis

1. Trace Metallic Impurities (TMI) analysis was performed by ICP-OES & ICP-MS on solutions of the candidate SI traceable salts.
2. Inert Gas Fusion (IGF) analysis was performed on the reduced metals to determine estimates of the O, N, and H impurities.

## Final Purity Calculation

The combined impurities from TMI and IGF analysis were applied as corrections to the mass fraction of Ir or Os in the starting material.



# Checking Our Work

How can we know that the gravimetric reduction and purity calculations produce an accurate **primary SI traceable solutions standard**?

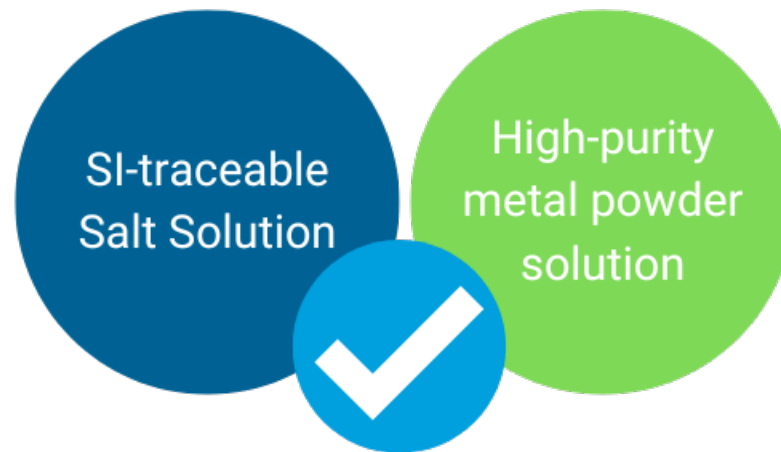
- We used the purity calculations to make a new standard solution from the salt and compare it against a solution made from a high-purity metal powder.
  - The comparison of these two independent starting materials provide a suitable check that our Ir/Os mass fractions in the salts, corrected for purity values, are accurate.

# ICP-OES Comparison of Solution Standards

- Accurate matrix matching and/or internal standardization of SI traceable solution standard to comparison solution standard
- Careful monitoring of metal washout
- Natural aspiration function of the nebulizer
- Large number of replicates
- Bracketing of samples

# ICP-OES Comparison Results

- Results for candidate SI solution standards were **< 0.1%** from the calculated values.
- Comparison demonstrates the **accuracy** of the candidate SI traceable solution standards.





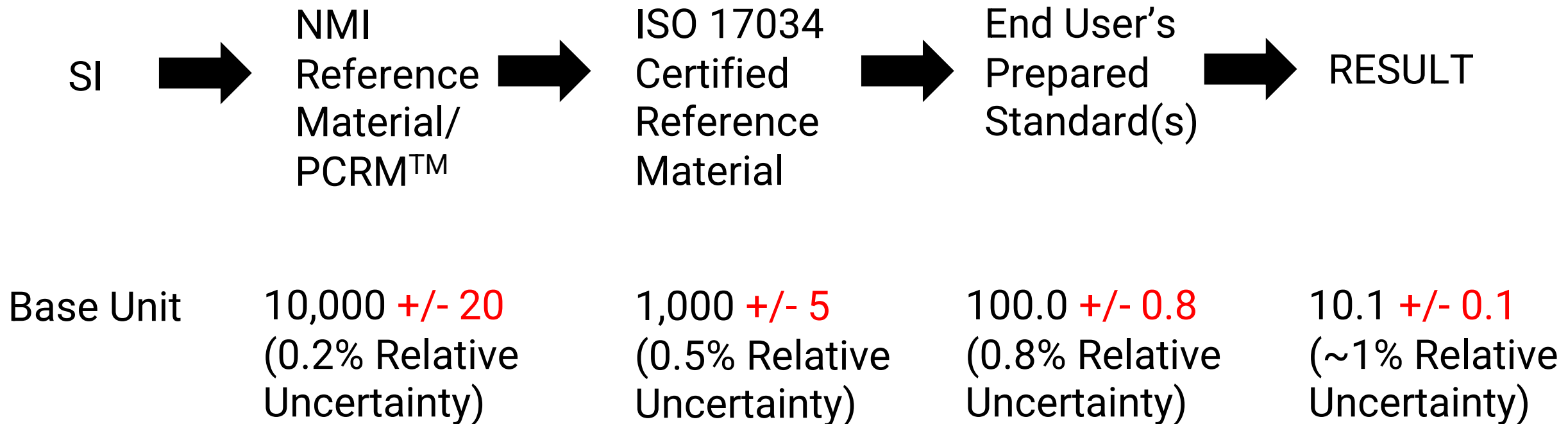
# Estimating the Uncertainty of PCRMs

ISO:17034 and ISO:17025 accreditation requires reporting the uncertainty of a certified value.

We make uncertainty estimates using:

1. Cause and effect diagrams
  - Map your sources of error
2. Error budget analysis
  - Quantify error based on gravimetric and purity measurements

# Understand Your Chain of Traceability

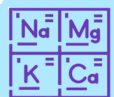


# Summary

- Primary SI traceable solution standards available for Os and Ir (Ru in progress).
- Short chain of traceability to ensure accurate results.
- Certified value and uncertainty from gravimetry and purity analysis.



# Any Questions?



**Interactive Periodic Table**

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## **Guides**

- ICP Operations Guide
- Sample Preparations Guide
- Trace Analysis Guide
- Periodic Table Guide
- Instrument Cross reference guide

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