DR EHRENSTORFER™

Reference Materials -In a nutshell

Presented by Don Shelly

NACRW 2018

THE ELEPHANT IN THE ROOM

The Second Source

LGC DR EHRENSTORFER™

What is a second source and why should I need one?

A second source is used to verify the integrity of the primary source. What defines a second source?

- **The answer depends on your industry**
  - **Different starting material?**
    - **Different lot of starting material?**
    - **Different supplier of starting material?**
  - **Different preparation?**
  - **Preparation from a different vendor?**

The NELAC definition

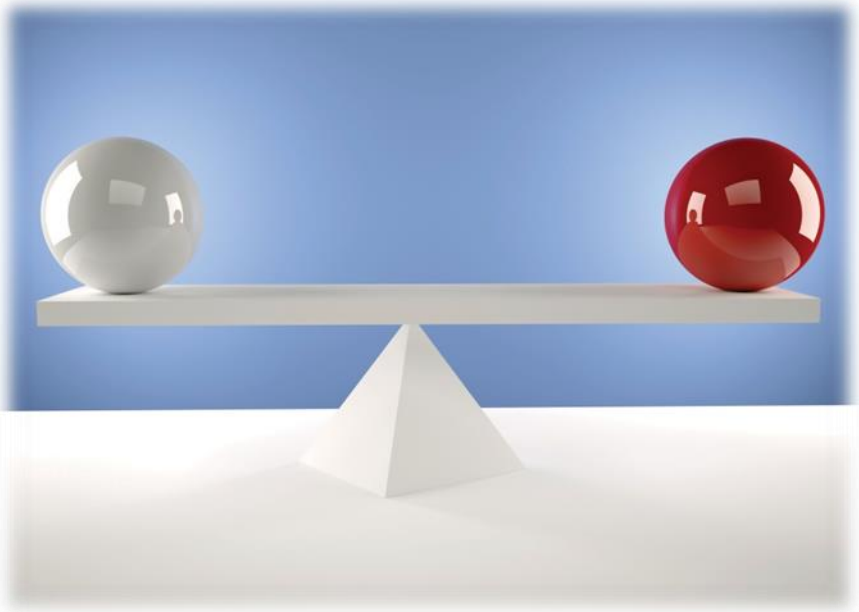
Independent preparation of a standard

- Different lot numbers from same starting material
- Different lot numbers from different starting materials
- Prepared from different vendors

Starting Materials

- The joy of sharing
  - There is often only one manufacturer of a neat material, 2 at most.
  - Blenders (provide mixes) generally acquire neats from vendors that can provide consistent quality at a competitive price.

Stability



ISO 17034:2016
Reference Material (RM)

- **Material, sufficiently homogeneous and stable with respect to one or more specified properties**

- **Established to be fit for its intended use in a measurement process.**

ISO 17034:2016 Certified Reference Material (CRM)

- Reference material characterized by a metrologically valid procedure for one or more specified properties

- Accompanied by a reference material certificate
  - provides the value of the specified property
  - its associated uncertainty
  - a statement of metrological traceability

    - Note: The uncertainty is the Total Combined Uncertainty at a specified coverage factor

Uncertainty Determination

- **RM**
  - **Characterization – Manufacturing Process**
    - **Weights**
    - **Volumes**
    - **Neat material purity**

Uncertainty Determination

- **CRM (Total Combined Uncertainty)**
  - **Characterization – Manufacturing Process**
  - **Homogeneity – Manufacturing Process**
  - **Transportation – Shipping, impacts shelf life**
  - **Long term stability – Chemistry, impacts shelf life and is the major contributor to uncertainty**

Shelf Life Determination

- Classical Method – Real time storage data
  - Hold product and test at various intervals until product fails or desired time elapses
  - Use when product development and/or life cycle allows
- Historical data/Product knowledge
- Accelerated Method – Estimate shelf life
  - Stress product with appropriate environmental variables to accelerate failure
  - Valuable when product development cycle is short

Accelerated Methods

- **Assumes that product failure, rate of degradation, increases if environmental conditions are made more harsh than the stated storage conditions**

- **Ensuring that the certified values for the analytes are within the stated uncertainties for the specified shelf life is critical**

Gravimetrically Prepared Analytical Reference Materials

- Most of our organic reference materials are sealed in ampoules so the predominate variables we measure are temperature and composition
- Composition changes primarily through chemical reactions
  - Analyte - Analyte
  - Analyte - Solvent
- Chemical reaction rates change with temperature – can use Arrhenius' Equation

Accelerated Stress Experiment

- Put one unit at storage temperature
- Heat stress additional units at three or more temperatures between the storage temperature and 100 °C for a given time
- Analyze all samples sequentially

Other Factors Which Influence Shelf Life Determination

- **Analyte stability for a long period does not necessarily mean that the solution is not changing**
  - **Analyte – Solvent interaction can impact solution pH and redox potential over time**
  - **Chlorinated hydrocarbons in methanol (methanol becomes more acidic with time)**
  - **Linear ketones degrade within hours (acetone, MEK, 2-hexanone)**

Solvent – Analyte Interaction

- Benzaldehyde – Benzidine reaction in methylene chloride to form the hydrozone like derivative
  - As methylene chloride ages, it becomes more acidic and catalyzes this reaction
- Urea based pesticides in acetonitrile
  - Acetonitrile goes basic as it ages and causes urea based pesticides to degrade

Environmental Conditions

- Storage conditions
- Transportation conditions

We are here to help

Contact

- DON.SHELLY@LGCGROUP.COM
- lgcstandards.com
- +1 (603) 622 7660
- lgcusa@lgcstandards.com

Science for a safer world