Sample Preparation, Why do I Care?

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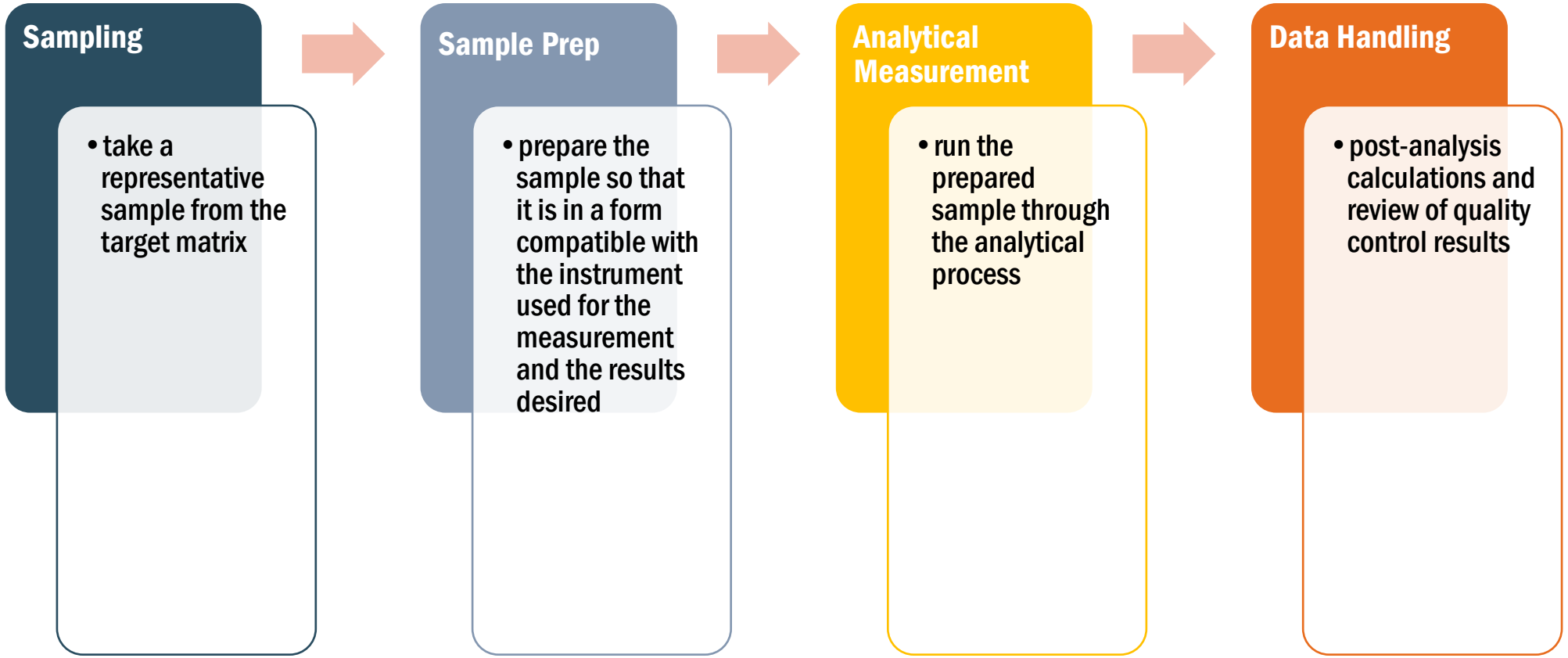
Agenda

- Why is Sample Prep Important
- Uncertainty/Variability/Sources
- Challenges in quality control of the sample prep step
- Examples of issues observed

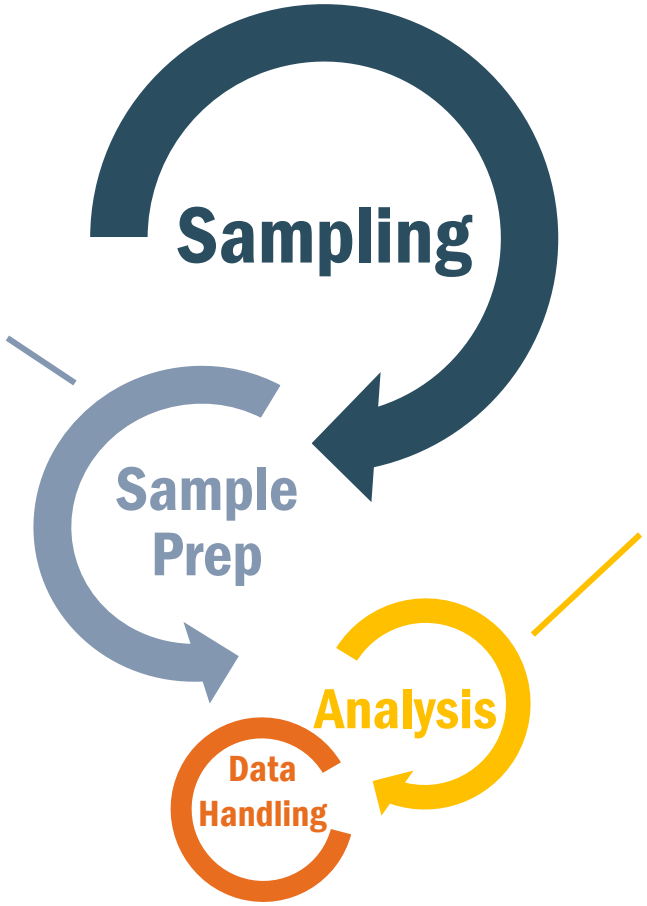
Typical Process to Obtain an Analytical Result



Estimated Uncertainty for Analytical Major Steps

Analyst, 2003, **128**, 1391–1398,
Jennifer A. Lyn, Michael H. Ramsey, Richard J. Fussell and Roger Wood

- For a variety of pesticides in a study using normal laboratory variation the overall sample preparation process contributed up to 20% to the total variability



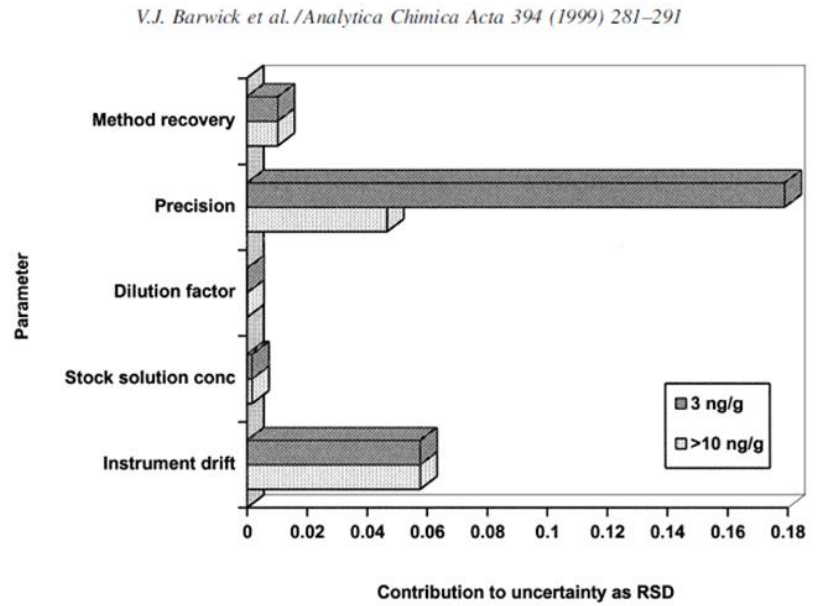
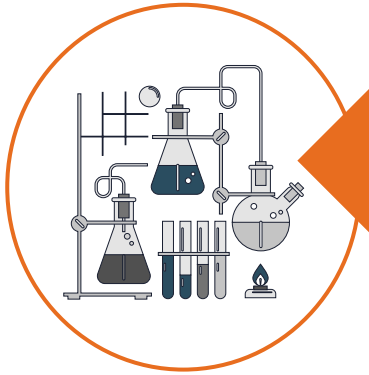


Fig. 2. Illustration of contributions to the uncertainty budget.

Example contributions from ICP-MS Instrument in 1999

Estimated Uncertainty for Major Steps

Uncertainty for the sample collection and preparation steps is large and challenged in detecting and determining the cause of failures



Uncertainty for the analysis step is small compared with other steps, with the continued effort to develop appropriate QC approaches, including SOP, ICV, CCV, etc.

Sample Preparation Challenges

- Challenges become bigger for sample prep as analytical instrumentation capability delivers more performance
- Detection limits lower on new technology, also allowing regulatory limits to be lowered
- Cleaner reagents and preparation procedures may be required
- Sample cleanup may be necessary for good results and better productivity

- Laboratory efficiency further pressed to maintain profitability
- Additional training may be needed for less-skilled technicians

How to Achieve?

Automation of prep step to reduce variability

Cleaner prep, such as microwave digestion for metals

Couple the prep with instrumentation

Simplify prep (QueCHers)

Examples of Sample Prep Problems - 1

Having a Strong Quality Management System Prevents Faulty Results

July 6, 2023, The NELAC Institute

Case Study 13: Incorrect Reagent

Some methods require use of reagents of specified purity (e.g., EPA 1664 requires 85% purity for hexane). The laboratory violated requirement in 40 CFR 136 to follow the method exactly as written. The result was likely accurate, but not acceptable.

Reference

1. State accreditation body, personal observation

QMS Failure

5.9.3– Mandated Methods. *The laboratory shall ensure that the essential standards outlined in Technical Modules or mandated methods or regulations (whichever are more stringent) are incorporated into their method manuals. When it is not apparent which is more stringent, the mandated method or regulations is to be followed.*

TNI QMS can help prevent serious sample prep failures

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Examples of Sample Prep Problems - 2

- Low level selenium measurements varied more than expected
- Se uncertainty tied to prep technician Selsun Blue shampoo usage
- Source of issue found by the observation of the sample prep process

Examples of Sample Prep Problems - 3

- Pharmaceutical company with a new lab and instrument complained to the instrument provider that the instrument had high metal blank readings
- The local application specialist carried his own clean water sample to the lab and demonstrated a low, acceptable blank
- Contaminated blank tied to improper material in a section of the water system piping from the high-quality water system to the plastic spigot in the laboratory found by tracing the water piping end-end

Examples of Sample Prep Problems - 4

- Cannabinoids in flower results from outside lab were very different than results from internal lab

- When they raised the concern with the lab, they did not get the expected brush-off, but the lab designed a study for their sample prep folks to do replicates and other measures they could compare. Analysis of the data showed one sample prep technician was different than the others and they sent him for re-training

How to Avoid these Issues?

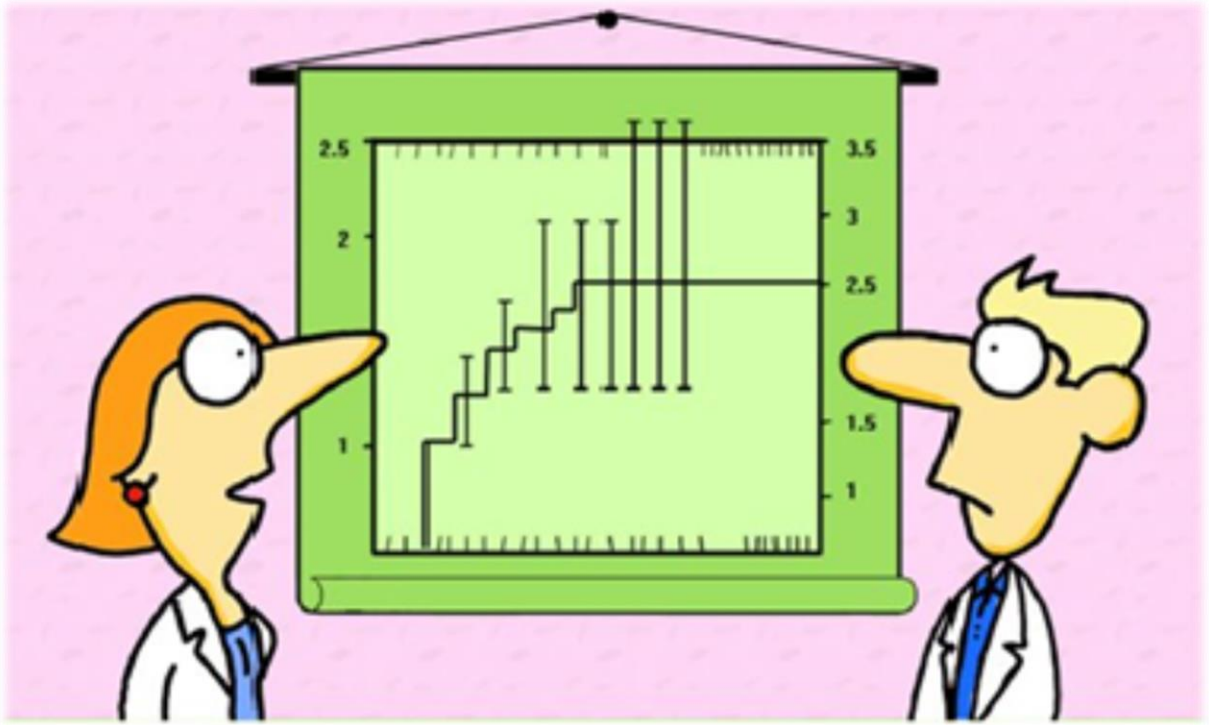
Have a strong Quality Management System in place (or use a certified Lab)

Acknowledge that the system is not perfect, ask questions

Understand what total uncertainty is tolerable

Total Uncertainty – Real Life Example

- Acetone 17 µg/L
- IC = R^2 0.989 / RSD 21.1 %
- CCV = -27.9%D [23 µg/L]
- LCS = 56 %R [42 µg/L]
- MS/MSD = 26 %R/40%R
  - 45 %RPD
  - [105 µg/L – 161 µg/L]



"Did you really have to show the error bars?"

Automation and Innovation for Sample Preparation – NEMC 2024 Oral Session

- **Sample Preparation, Why do I Care?, P. Newbold[1], Z. Grosser[2]**
  1 - ddms, Inc, 370 Wabasha St N, Suite 1310, St. Paul, MN, 55102, United States
- **Inline Filtration and Treatment for Ion Chromatographic (IC) Methods, K. Nash, S. Giraud, L. Ramirez, J. Davis, D. Tran, P. Parmar**
  Orange County Water District, PO Box 8300, Fountain Valley, CA, 92728-8300, United States
- **Advancements in EPH Fractionation: Overcoming Challenges and Enhancing Efficiency, C. Mitchell**
  Biotage, 2350 Bering Drive, San Jose, California, 95131, United States
- **The Automated Extraction of 40 PFAS Compounds from Tissue Samples, A. Stell, B. Liu**
  CEM Corporation, 3100 Smith Farm Rd., Matthews, NC, 28106, United States
- **Fully Automated Determination of PFAS in Liquid and Solid Matrices Via Online SPE Coupled to LC-MS/MS: Application to EPA Method 1633 Compound List, L. Ispiryan[1], H. Korb[2]**
  1 - Trajan Scientific and Medical, Axel Semrau, Stefansbecke 42, Sprockhoevel, Northrine westfalia, 45549, Germany
  2 - iChrom Solutions, 542 Cross Keys Rd, Sicklerville, NJ, 08081, United States
- **Analysis of Semi Volatiles Method by GC/MS/MS with Liquid-liquid Extractions Using ePrep Full Automation for EPA 3511, A. Willey[1], A. Minett[2], B. Van Middlesworth[3]**
  1 - Agilent Technologies, 2850 Centerville Road, Wilmington, DE, 19808, United States
  2 - ePrep Pty Ltd, 3 Kingston Town Close, Oakleigh, VID, 3166, Australia
  3 - ePrep Pty Ltd, , CA, United States
- **Semi-Automated Solid Phase Extraction and Analysis of Wastewater with EPA Method 625, T. Hall, R. Addink**
  Fluid Management Systems, 900 Technology Park Dr, Billerica, MA, 01821, United States

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Discussion or Questions