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Inorganic & Organic Certified Reference Materials

Your Science is Our Passion.™

Certified Reference Materials: Beyond the Basics

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Housekeeping

- Everyone will receive a copy of the presentation slides
- The webinar is being recorded and will be posted to our YouTube account
- Questions will be answered at the end of the presentation
  - *Type any questions you may have into the question box and we will answer them during the Q&A session*

Challenges to Analysts and CRM manufacturers

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- Accreditations & Regulations (ISO, CPSC, EPA …)
  - *Challenges:*
    - Compliance
    - Decreasing detection levels
    - Increasing # compounds
  - *What do the accreditations mean and where do they come from?*
  - *What really defines a standard, certified reference material?*

Challenges to Analysts and CRM manufacturers

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- Accurate Analyses
  - *What are traceability, homogeneity, uncertainty and error?*
  - *How are each defined and measured?*
- Error
  - *How do we reduce error and contamination in analyses?*
  - *What practices, procedures or habits lead to increased error?*

Accreditations

Accredited to 17025 & Guide 34

ISO

Certified to 9001

What is ISO 9001:2000?

- **ISO 9000** family of standards
  - *Quality management systems*
    - Meet the needs of customers
    - Meet statutory and regulatory requirements of product
- Published by International Organization for Standardization,
  - *Third party certification bodies*
  - *Over a million organizations worldwide certified*

ISO 9001 History

- Origins in US Military: Mil-Q-9858 (1959)
- NATO (1969) AQAP series of standards
- British Standard (BS) 5750 from NATO (1979)
- International Organization of Standards (1946-47)
  - 1987: ISO 9001:2000
  - 1994: 2nd edition
  - Latest edition 2008

ISO and Reference Materials

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- 1991 ISO/REMCO Guide 34 draft
  - ***General requirements for the competence of reference material producers***
- 1996: Guide 34 published
  - *Latest revision 2009*
- 1999: ISO/IEC Guide 25
  - ***General Requirements for the competence of testing and calibration laboratories***
- 2000: Became ISO/IEC 17025
  - *Re-issued in 2005*

ISO 17025

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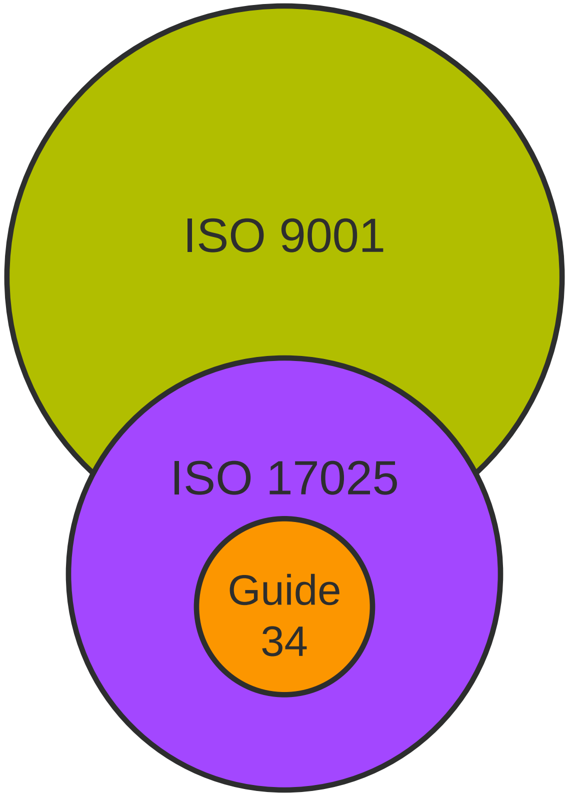
- Laboratories must demonstrate
  - *Management system*
  - *Technical competency*
  - *Technically valid results*
- Harmonization of standards & procedures.
  - *Comply to International Standard*
  - *Equivalent results among testing bodies*
  - *Equivalent accreditation bodies in countries using International Standard.*

ISO Guide 34

- Allows transfer of results from CRM to labs for:
  - *Calibration of measuring equipment*
  - *Evaluation or validation of measurement procedures.*
- Requires CRM producers to demonstrate:
  - *Scientific & technical competence*
  - *Ability to supply information about their materials*

Quality Systems for CRM

- ISO 9001
  - *General requirements: Quality Management System*
- ISO 17025
  - *Testing and calibration laboratories*
- Guide 34
  - *CRM producers*
  - *Requirements for manufacturer of CRM*



Standard

Reference material

CRM

Uncertainty

Traceability

Homogeneity

Stability

Error

What is a Standard?

- A standard is a ‘KNOWN’ to compare an ‘Unknown’
- An acknowledged measure of comparison for quantitative or qualitative value – American Heritage Dictionary
- ISO definitions
  - *Primary standard:*
    - highest metrological quality
    - value is accepted w/o reference to other same quality standards
      - Example: Le Grand K
  - *Secondary standard:*
    - value is assigned by comparison with same quantity of a primary standard

Image credit: International Bureau of Weights and Measures

The Role of Standards

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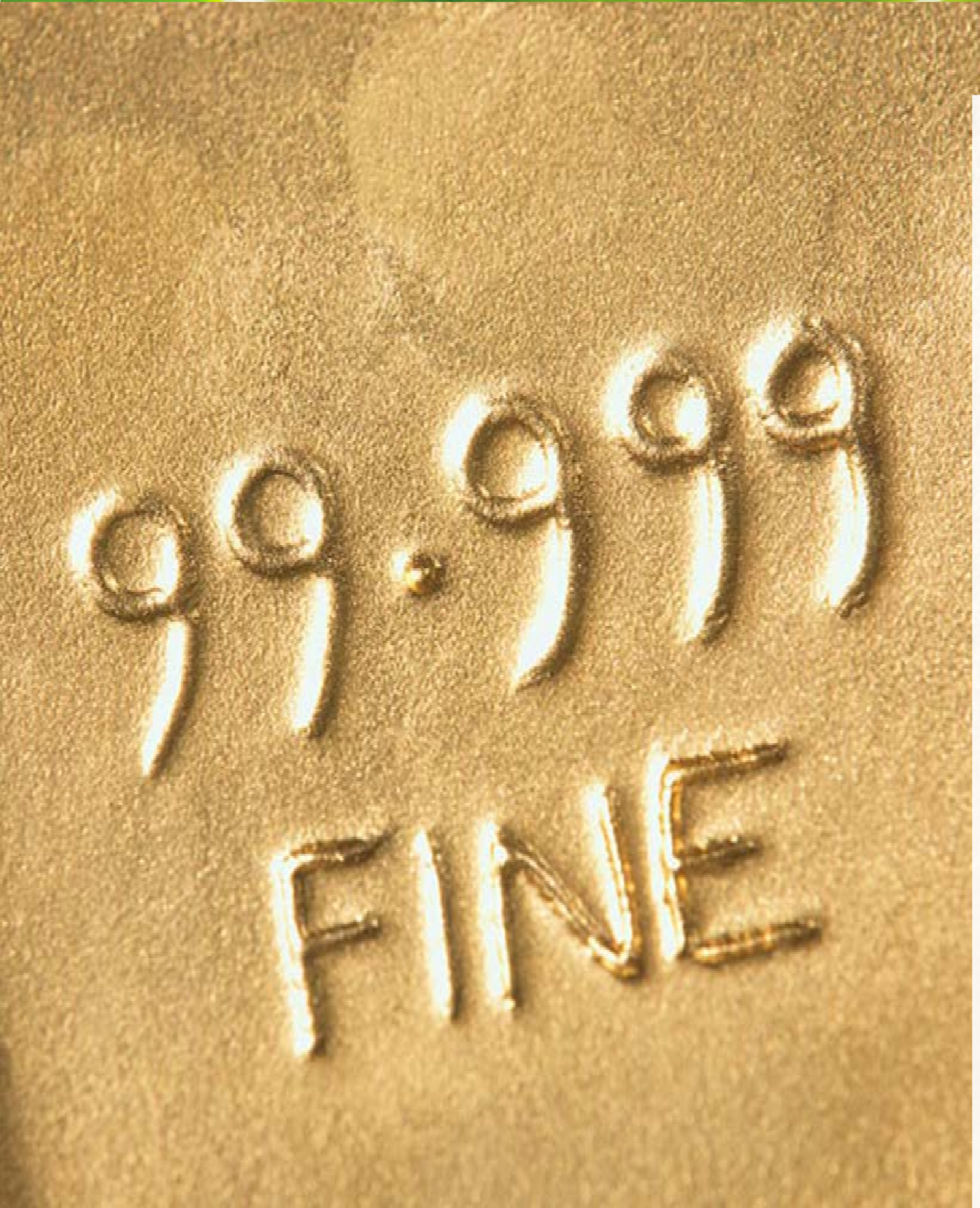
- **Quantitation**
  - *ESTD*
  - *ISTD*
  - *Std Addition*
- Find, Calculate and Correct for **Uncertainty** in process
  - *Calculate instrument uncertainty*
  - *Calibration*
  - *Calculate recovery*
- Find & Eliminate **Error** in process

What is a Reference Material?



- ISO definition
  - *A material or substance whose property value(s) are:*
    - Sufficiently **homogeneous** & well established
    - Used for:
      - Calibration of an apparatus
      - Assessment of a measured method
      - For assigning values to materials.

What are Certified Reference Materials?

- ISO definition:

A reference material accompanied by a certificate

– *One or more property values are certified by a procedure that establishes:*

- **Traceability**
- **Uncertainty**

– *Contains supplementary information*

- Certifying bodies
- Methods of Measurement
- Material preparation
- Statistical treatment of results

Certificates

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SPEXertificate®
Certificate of Reference Material

Reference Materials Producer
CERT #2495.01
Chemical Testing
CERT #2495.02

Catalog Number: 5511-PH **Lot No.** T1121024013

Description: Halogenated Pesticides and Herbicides Mix

Matrix: Acetone

Ship Date: 11-5-2012
Expiration Date: 5-4-2013

This **SPEXOrganics®** Certified Reference Material, CRM, is intended primarily for use as a calibration standard or quality control standard for organic chromatography instrumentation such as GC, GC-MS, LC, and LC-MS. It can be employed in USEPA, ASTM and other methods relevant to the certified properties listed below.

Certified Compounds:

Compound	CAS #	Purity	Labeled	Actual†
Alachlor	15972-60-8	99.5%	2000 µg/mL	2000 µg/mL
Atrazine	1912-24-9	98.9%	2000 µg/mL	1990 µg/mL
Bladex	21725-46-2	99.5%	2000 µg/mL	2000 µg/mL
Bromacil	314-40-9	99%	2000 µg/mL	1990 µg/mL
Endrin	72-20-8	98.9%	2000 µg/mL	2000 µg/mL
Endrin aldehyde	7421-93-4	99%	2000 µg/mL	1990 µg/mL
Endrin ketone	53494-70-5	99%	2000 µg/mL	1990 µg/mL
gamma-BHC	58-89-9	97%	2000 µg/mL	1990 µg/mL
Heptachlor	76-44-8	99%	2000 µg/mL	1990 µg/mL
Heptachlor epoxide (Isomer B)	1024-57-3	99%	2000 µg/mL	1990 µg/mL
Hexachlorobenzene	118-74-1	99%	2000 µg/mL	2000 µg/mL
Hexachlorocyclopentadiene	77-47-4	98%	2000 µg/mL	2020 µg/mL
Methoxychlor	72-43-5	95%	2000 µg/mL	1990 µg/mL
Metolachlor	51218-45-2	98%	2000 µg/mL	2000 µg/mL
Metribuzin	21087-64-9	99.5%	2000 µg/mL	1990 µg/mL
Trifluralin	1582-09-8	99.5%	2000 µg/mL	2000 µg/mL

Final Solution Verification:

Final solution integrity verified by Gas Chromatography/Mass Spectrometry. The mass spectrum of each compound was confirmed against the NIST mass spectral database.

† Actual concentration based on gravimetric weights and corrected for the purity of the compound(s) used to prepare the standard. Analytical balance calibration is verified daily with C1 weight set #23-190006 which is registered with Atlantic Scale, and traceable to NIST and NJ Division of Weights and Measures.

This CRM is guaranteed stable and accurate to +/- 0.6% of the certified value. This includes uncertainty components due to preparation, homogeneity, short term and long term stability. During the stated period of validity, the purchaser will be notified if this product is recalled due to any significant changes in the stability of the solution. For further information, contact the Sales Support Department at crmsales@spexcsp.com.

Date of Certification: 11-5-2012 Certifying Officer:

- **Certificate**
  - *Traceability*
  - *Uncertainty*
  - *Use as Calibration*
  - *Homogeneity*
  - *Stability*
  - *Certified values*
  - *Detailed Supplementary information*

Traceability & Stability

- Traceability - trace a product: from origin to manufacture & delivery/receipt.
  - *For CRMs ensures can be traced to a primary standard*

- Stability -
  - *not reactive during normal use*
  - *retains properties in expected timescale in the presence of expected conditions of application.*
  - *Unstable material*
    - corrode, decompose, polymerize, burn or explode under the ‘normal’ conditions

Homogeneity

- Homogeneity - uniform in composition or character
  - *In-bottle Homogeneity*
    - Checks product for stratification, precipitation etc.
    - Materials which can settle out of solution are still homogenous if they can be re-dissolved into solution by the instructions with the material (i.e. sonication, heating, shaking etc.)
  - *Between-bottle Homogeneity*
    - Samples multiple containers from each lot to check for homogeneity

What are Error and Uncertainty?

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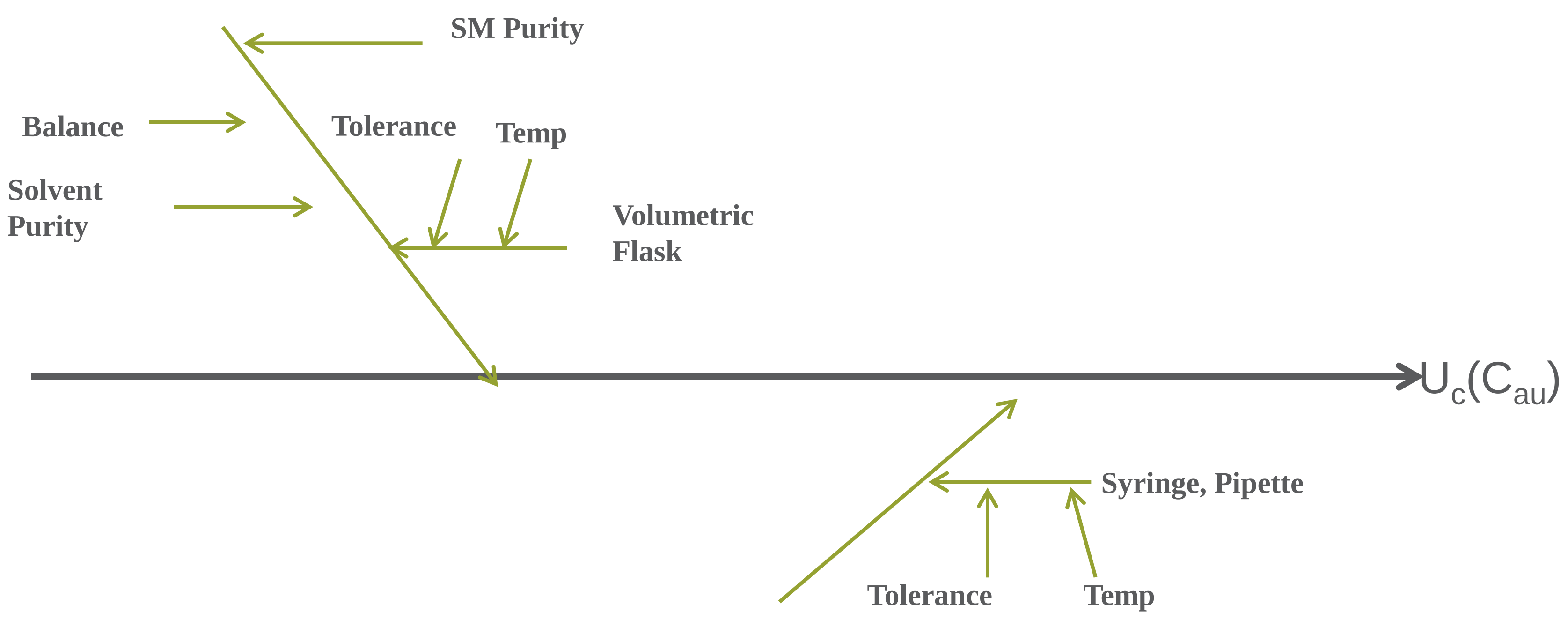
- **Error**: difference between measurement & 'TV' of the measurand.
  - *Doesn't include mistakes (explain & exclude)*
  - *Causes values to differ when a measurement is repeated*
    - None of the results preferred over others
  - *Impossible to completely eliminate error but it can be controlled and characterized*
- **Uncertainty**: estimate attached to a certified value which characterizes the range of values where "true value" lies within a stated confidence level.
  - *Random effects, short-term fluctuations in temperature, humidity, static electricity, and air-pressure*
  - *Variability in the performance of the analyst*
  - *Drift than can be accounted for by a correction*
- Error vs. Uncertainty:
  - *Error: Usually can't be estimated*
  - *Uncertainty: Can be estimated*

Uncertainty Sources

Identify all major uncertainty sources
Understand their effect on the analyte and its uncertainty



Uncertainty Estimation

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- Determine what is to be measured
- Outline the various processes
- Identify sources of uncertainty
- Estimate uncertainties from each source
- Combine and expand all uncertainty

Types of Uncertainty

Type A

- Associated with repeated measurements
- Type A uncertainty is expressed as:

$$u_i = \frac{s}{\sqrt{n}}$$

Where s is standard deviation and n is the number of replicates

Type B

- Based on scientific judgment made from previous experience, manufactures' specifications, etc.
- Three common models are:
  - *Rectangular*
  - *Triangular*
  - *Normal*

Type B: Rectangular Distribution

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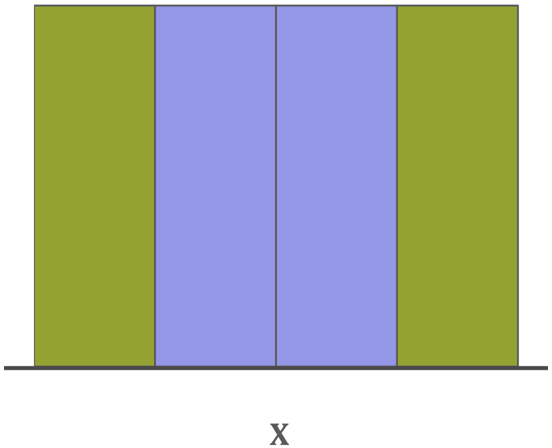
- Use when a certificate or other specification gives limits without specifying a level of confidence
- Normalizing factor for converting to Standard Uncertainty is $\frac{Listed\ u}{\sqrt{3}}$



Type B: Triangular Distribution

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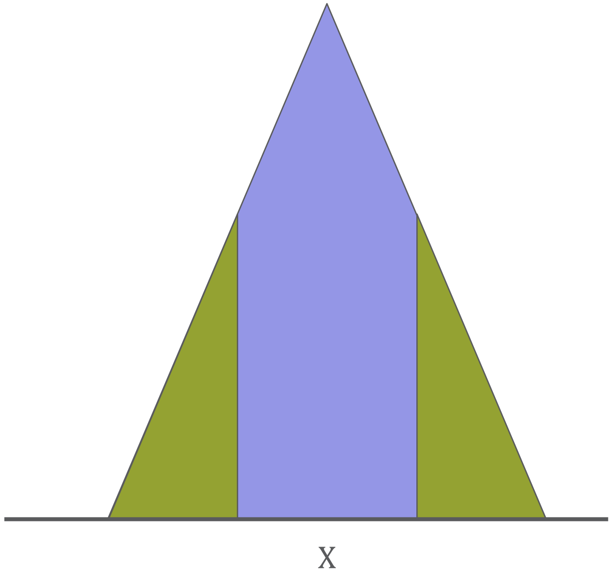
- Used when distribution is symmetric and when values close to the target value are more likely than near the boundaries
  - *Example: Uncertainty associated with volumetric glassware*
- Normalizing factor for converting to Standard Uncertainty is $\frac{Listed\ u}{\sqrt{6}}$



Type B: Normal Distribution

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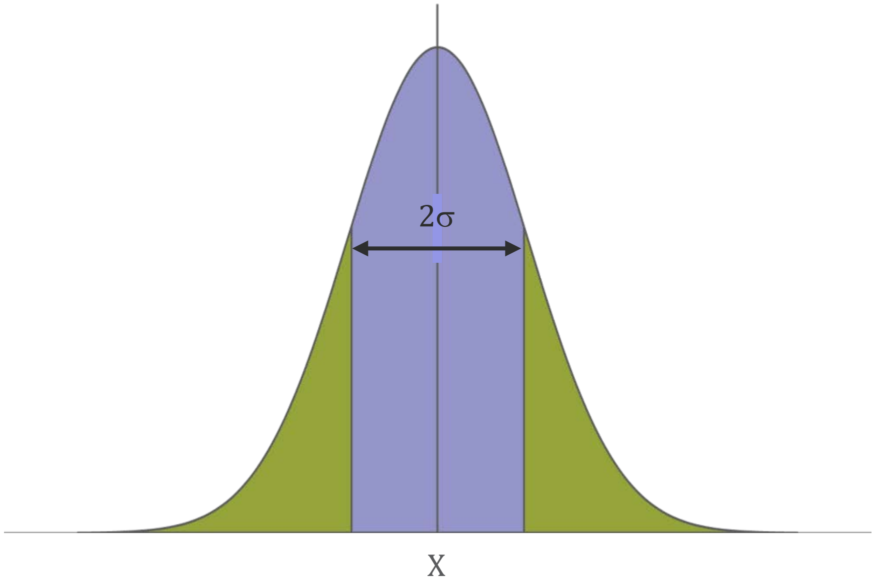
- Used when an estimate is made from repeated observations of a randomly varying process and an uncertainty is associated with a certain confidence interval.

  – *Example: A calibration certificate with stated level of confidence.*

- Normalizing factor for converting to Standard Uncertainty is $\frac{Listed\ u}{k}$



Combined Uncertainty (u_c)

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We will use the following model for calculating ***interim*** u_c within a process:

$$u_c(y) = \sqrt{u(x_1)^2 + u(x_2)^2 + u(x_3)^2}$$

We will be using the following model to ***combine*** overall uncertainties from all of the processes:

$$u_c(y) = y\sqrt{\left[\frac{u(x_1)}{x_1}\right]^2 + \left[\frac{u(x_2)}{x_2}\right]^2 + \left[\frac{u(x_3)}{x_3}\right]^2}$$

Expanded Uncertainty (U)

- The Expanded Uncertainty defines the interval within which lies the value of the measurand
- To calculate, multiply the combined standard uncertainty with a Coverage Factor (k):

$$U = ku_c$$

- Coverage factor k depends on the level of confidence and the degree of freedom:
- k= 2 for 95% confidence level

True Value (X) = $\overline{X}$ +/- U

Analysis Challenges & Issues

The Analytical Threshold Keeps Decreasing

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- Modern instrumentation LOD to PPB & PPT
  - *ICP*
  - *ICP-MS*
  - *GFAA*
  - *LCMS*
  - *GCMS*
- Lower detection limits > importance of eliminating trace contaminations
- Eliminate trace impurities present in:
  - *Reference Materials*
  - *Samples*
  - *Reagents*
  - *Environment*

Trace Analysis Concentrations

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Just how much is a part per billion or trillion?

Unit	1 PPM	1 PPB	1 PPT
Time	1 second / 11.6 days	1 second / 32 years	1 second / 320 centuries
Money	1 cent / $10 k	1 cent / $10 million	1 cent / $10 billion
Volume	1 drop vermouth / ½ barrel of gin	1 drop vermouth / 500 barrels gin	1 drop vermouth / 500,000 barrels gin
Length	1 inch / 16 miles	1 inch / 16,000 miles (diameter around the earth twice)	1 inch / 16 million miles (farthest distance between Earth and Venus)

The Compendium of Compounds regulated keeps Increasing

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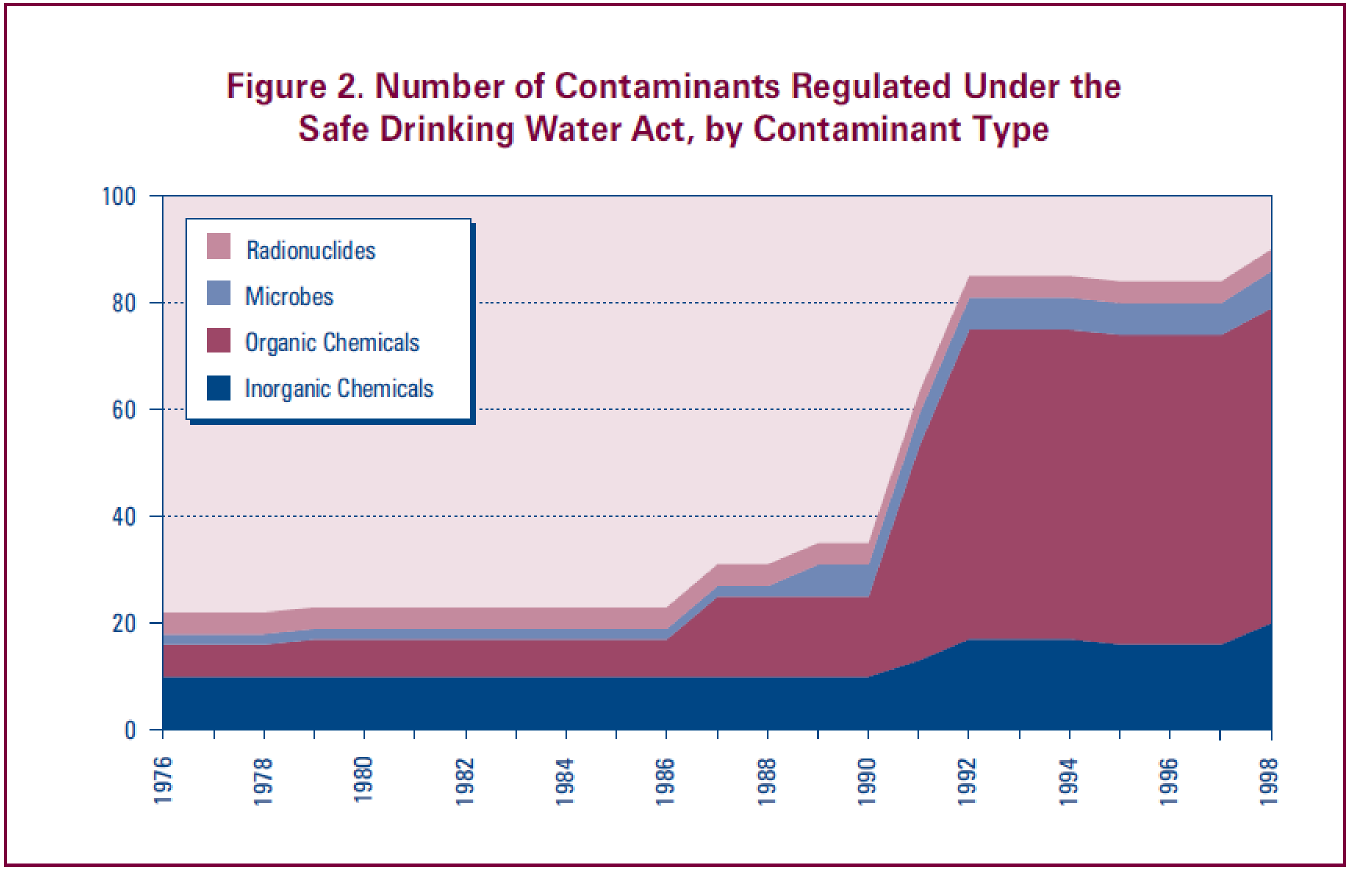
- First US drinking water standards in US – 1914 (Bacteriological standards)
- 1962- First chemical standards: 28 substances & 2 Organic Compounds
- Since 1974 – 3x increase in compounds, and decrease in limits



From: 25 Years of the Safe Water Drinking Act: History and Trends: EPA

Sources of Potential Contamination or Error Everywhere

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- Chemical Components
  - *Solvents, Reagents, Additives*
- Laboratory Components
  - *Syringes, Pipettes, Glassware, Storage containers, Scales*
- Sample preparation techniques
  - *Grinding, Extraction, SPE, Dilutions, Esterification*
- Standard preparation techniques
  - *Serial dilutions, Weighing out & Preparation*
- Instrumentation Contamination
  - *Columns, Mobile Phases, Gases, Carryover*

Sources of Potential Contamination or Error

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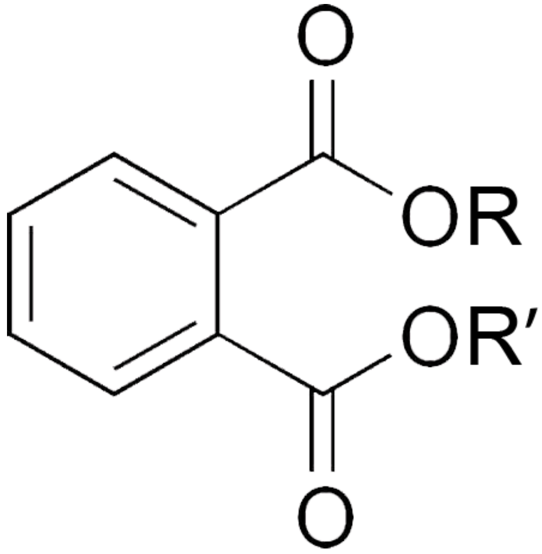
- Laboratory environment
  - *Lab surfaces: Residue & Dust*
  - *Ventilation & Hoods*
  - *Waste containers*
- Laboratory Personnel
  - *Cosmetics*
  - *Glove contamination*
  - *Lab Hygiene practices*
  - *Human Error*

The Chemical Components

- Starting materials
  - *tested for trace impurities*
  - *Impurities create:*
    - overlap of spectra
    - incorrect calibration curve
    - inaccurate results
- Common Laboratory contamination:
  - *Metals: Silicon, Calcium, Aluminum*
  - *Phthalates*
  - *Impurities*
  - *VOA: Chloroform, Acetone*
  - *Laboratory Solvents*

Solvents

- Source of contamination
  - *Persistent Solvents in Lab*
    - DCM & Carbon Disulfide
- Solvent Contamination includes
  - *Particles, Gases, Preservatives and additives*
  - *Leach or dissolve compounds from their containers*
    - Sodium (glass), Boron (glass), Silica (pH >10)
    - Phthalates (plastics and liners)



Solvents

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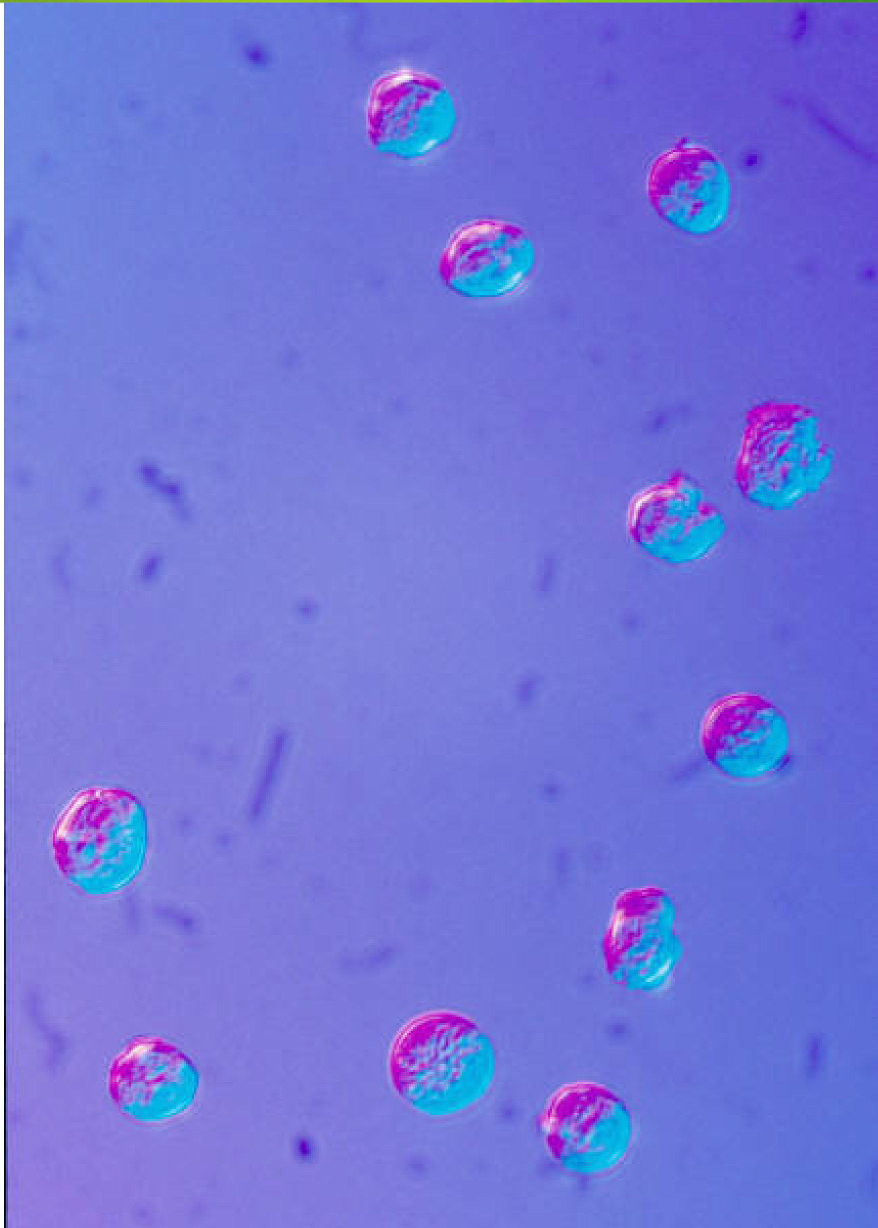
Grade	Application/Use	Analytical
ACS	General Procedures	Meets or Exceeds ACS specifications
Anhydrous	Water sensitive reactions and synthesis	Low water levels (10-30 ppm)
Biotechnical	Biotechnical applications	Low water, low residue, low UV
Environmental	Environmental Analysis, HPLC, Trace Organic	
Food/FCC Grade	Food & Drug Applications	Meets specifications of Food Chemicals Codex (FCC).
GC	GC applications	PPB levels of contamination
HPLC	HPLC applications	Sub-micron filtration, some low UV absorbance
LCMS	LCMS applications	Low ionic impurities <0.1 ppm
Pesticide Residue	Pesticide, Environmental Analysis, Trace analysis & GC-ECD, FID, MS	Meets or Exceeds ACS Pesticide Specifications
Reagent	General Lab Use	>95% Purity
Spectrophotometric	UV applications	UV, Vis, IR
Technical	General Lab Use	Non-critical tasks
USP	Food & Drug Applications	Meets or Exceeds USP Specifications

Acids

- Any part of analytical process must use high purity acids
  - *Dissolution of materials and samples*
  - *Digestions*
  - *Dilutions*
- Contaminants in acid increase error:
  - *An aliquot of 5 mL of acid*
  - *100 ppb of Ni as contaminant*
  - *used for diluting a sample to 100 mL*
  - *introduce 5 ppb of Ni into the sample*
- Use High purity acid for dilution but costly



Water

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- The major component of an aqueous standards, LC & LCMS mobile phases
- Quality & Accuracy of analysis depends on water quality

Specifications of Four Types of ASTM Water

Requirement	ASTM Type			
	I	II	III	IV
Use	Critical laboratory applications and processes	General Lab grade used for pH, buffers, feeding other water polishing systems	Cleaning Glassware, Feeding Water polishing systems, water baths	Not good for Lab Use
Specific Resistance (megohm/cm) (max)	**18**	1	4	0.2
pH	**N/A**	N/A	N/A	5 - 8
Sodium (max)	**1 µg/L**	5 µg/L	10 µg/L	50 µg/L
Total Silica (max)	**3 µg/L**	3 µg/L	500 µg/L	high
Total Organic Carbon (max)	**50 µg/L**	50 µg/L	200 µg/L	N/A

Laboratory Water

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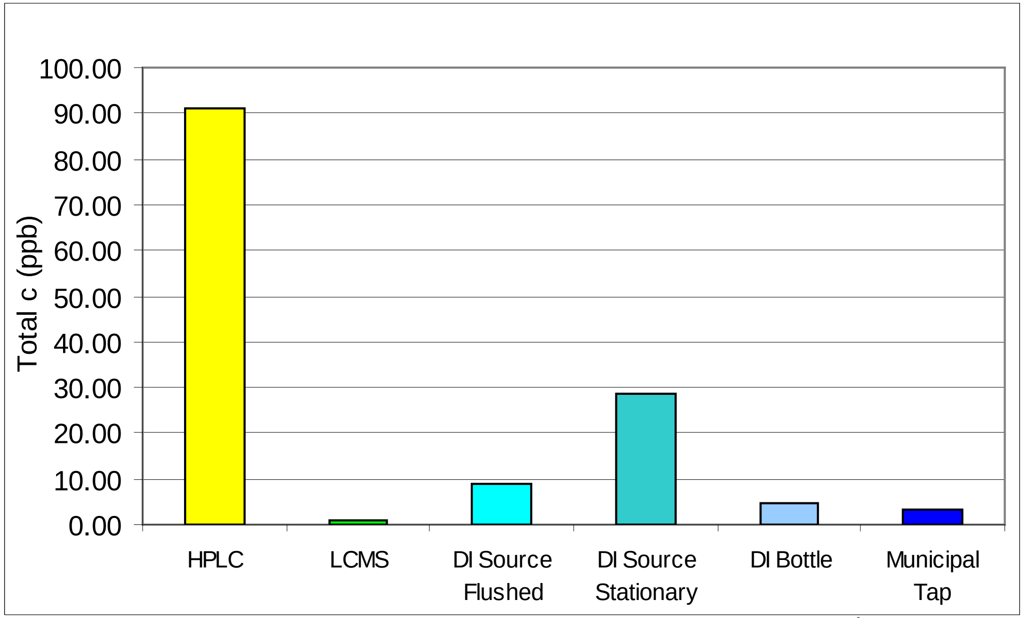
	HPLC	LCMS	DI Source Flushed	DI Source Stationary	DI Bottle	Municipal Tap
DEP	6.28	0.18	0.00	0.30	0.50	0.00
DIBP	3.52	0.16	0.88	1.36	0.52	0.00
DBP	16.72	0.00	0.00	0.00	0.54	0.00
BPA	3.16	0.00	0.00	0.00	0.00	0.00
BBP	44.74	0.20	2.32	0.63	0.47	1.29
DCP	1.00	0.00	0.00	0.00	0.00	0.00
DEHP	15.60	0.63	5.92	26.41	2.44	1.94
Total c (ppb)	**91.02**	**1.17**	**9.12**	**28.70**	**4.47**	**3.23**
Total # compounds	**7**	**4**	**3**	**4**	**5**	**2**



Reducing Contamination and Error from Laboratory Water

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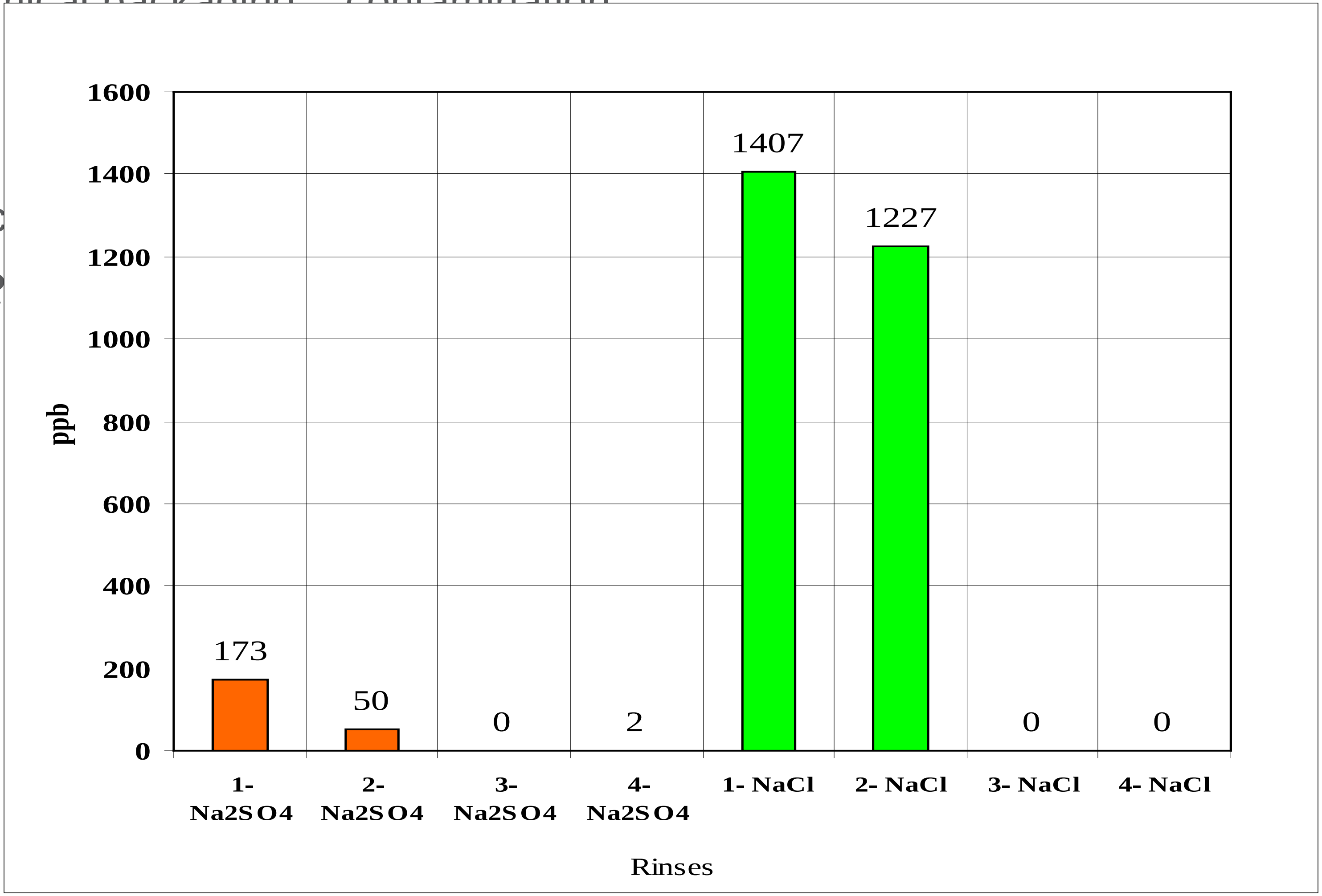
- Bottle Water has an expiration date
- Water quality can change
  - *Storage bottles can leach contaminants*
  - *Microbial growth*
    - Change water once a day
    - Flush LC system if final conditions have >80% water
    - A clean flush mixture can be run through an HPLC system to remove microbial growth or clean an LCMS source.
      - MeOH, Acetonitrile, Acetone, IPA, and Cyclohexane

Chemical Stock Contamination

- Chemical packaging = contamination
  –
  –
- Diffic
  – *R*



Laboratory Equipment

- The most common sources of contamination, and uncertainty is labware.
  - *Syringes, pipettes, dispensers*
    - Carryover/Cleanliness
    - Accuracy
    - Calibration & Maintenance
  - *Laboratory glassware*
    - Calibration/Class
    - TC or TD
  - *Storage containers*
    - Material
    - Cleanliness

How Clean Are Your Pipettes?

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2% nitric acid run through 5 mL pipettes that were cleaned manually and scanned on ICP-MS

Element	Conc. (PPB)	Element	Conc. (PPB)
Ag	**2.33**	Mn	1.72
Al	**6.43**	**Na**	**19.1**
Be	2.62	Ni	0.96
Bi	1.07	**Pb**	**5.4**
Ca	**18.8**	Sn	0.55
Co	2.02	Th	0.24
Cr	0.91	Ti	0.56
Fe	**1.62**	Tl	1.53
Mg	2.56	Zn	9

How Clean are Your Syringes?

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1000 uL syringe Carryover

	Naphthalene-d8		Phenanthrene-d10		Chrysene-d12	
Injection Wash #	**ppm**	**% Carryover**	**ppm**	**% Carryover**	**ppm**	**% Carryover**
1	16.15	0.81	15.58	0.77	24.40	1.22
2	0.59	0.03	0.61	0.03	0.69	0.03
3	0.06	0.00	0.06	0.00	0.06	0.00
4	0.04	0.00	0.02	0.00	0.03	0.00
5	0.04	0.00	0.04	0.00	0.04	0.00
7	0.01	0.00	0.02	0.00	0.01	0.00
10	0.02	0.00	0.03	0.00	0.03	0.00
15	0.01	0.00	0.00	0.00	0.02	0.00
20	0.00	0.00	0.00	0.00	0.01	0.00

Manufacturer suggest rinse 2-3 times to waste and between 5-20 times overall

Syringe Carryover

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100 uL syringe Carryover

	Naphthalene-d8		Phenanthrene-d10		Chrysene-d12	
Injection Wash #	**ppm**	**% Carryover**	**ppm**	**% Carryover**	**ppm**	**% Carryover**
1	25.21	1.26	24.71	1.26	64.47	3.22
2	0.59	0.03	0.50	0.03	0.93	0.05
3	0.06	0.00	0.01	0.00	0.13	0.01
4	0.02	0.00	0.01	0.00	0.07	0.01
5	0.02	0.00	0.01	0.00	0.07	0.01
7	0.01	0.00	0.01	0.00	0.06	0.00
10	0.00	0.00	0.00	0.00	0.04	0.00
15	0.00	0.00	0.01	0.00	0.04	0.00
20	0.00	0.00	0.01	0.00	0.04	0.00

Syringe Carryover

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10 uL syringe Carryover

	Naphthalene-d8		Phenanthrene-d10		Chrysene-d12	
Injection Wash #	**ppm**	**% Carryover**	**ppm**	**% Carryover**	**ppm**	**% Carryover**
1	175.67	8.78	180.72	8.30	360.51	18.03
2	26.21	1.31	25.70	1.26	46.96	2.35
3	6.12	0.31	4.98	0.27	8.12	0.41
4	1.71	0.09	1.74	0.10	4.10	0.21
5	0.69	0.03	0.70	0.04	2.02	0.10
7	0.25	0.01	0.36	0.03	1.37	0.07
10	0.10	0.01	0.04	0.02	1.37	0.07
15	0.09	0.00	0.17	0.02	1.13	0.06
20	0.03	0.00	0.07	0.01	0.43	0.02

Reducing Syringe Contamination & Error

- Smaller volume syringe = more rinses needed
  - *10 uL syringe 5-10 rinses should go to waste in addition to another 5-10 rinses to clean syringe*
  - *Viscous samples may need much more cleaning*
    - Pulling solvent through a vacuum system & taking the syringe fully apart
- Additional Cleaning tips:
  - *Don't heat fixed needle syringes above 60 C*
  - *Do not submerge syringes in solvent to clean*
  - *Take syringe apart and clean plunger and air dry*

Reducing Syringe Error

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Syringe Size	uL Dispensed	Water
10	2	23.15%
10	5	8.16%
10	10	2.72%
25	2	8.82%
25	5	5.47%
25	10	2.37%
25	20	1.05%
25	25	1.25%
100	10	6.09%
100	25	1.67%
100	50	0.64%
100	100	0.61%
1000	250	1.05%
1000	500	1.14%
1000	1000	0.47%

- Use calibrated syringes of appropriate range for level decanting
  - Measured volume should be no lower than 20% of syringe volume (1000 & 100 uL syringes) for 1% or lower error
  - Measured volume needs to be >20% for smaller syringes to decrease error.

How Clean are Your Storage Containers?

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- Bottles
  - *Various sizes, shapes, and materials of construction*
- Contaminants present in the materials of construction can leach into the solution
- Active sites can bind analytes

Summary of Average Element Content in Storage Containers

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Material	Total No. of Elements	Total PPM	Major Impurities
Polystyrene-PS	8	4	Na, Ti, Al
TFE*	24	19	Ca, Pb, Fe, Cu
Low Density PE-LDPE	18	23	Ca, Cl, K, Ti, Zn
Polycarbonate-PC	10	85	Cl, Br, Al
Polymethyl Pentene-PMP	14	178	Ca, Mg, Zn
FEP†	25	241	K, Ca, Mg
Borosilicate Glass	14	497	Si, B, Na
Polypropylene-PP	21	519	Cl, Mg, Ca
High Density PE-HDPE	22	654	Ca, Zn, Si

* TFE-Tetrafluoroethylene

† FEP-Fluorinated ethylene propylene

Do you have a Clean Laboratory?

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- Walls, ceilings, and floors are sealed and dust free
- HEPA filters mounted in the ceiling
- No exposed metal parts
- Contamination sources:
  - *Ceiling tiles, paints, cements, and dry walls*
  - *Dust and rusts on shelves, equipment, and furniture*
  - *Temperature control systems*

Clean Room Packaging Comparison

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Comparison of a solution made in clean room environment and packaged in regular lab and clean lab

Elem	Regular Lab	Clean Lab	Elem	Regular Lab	Clean Lab
Al	5 ppb	0.1 ppb	**Na**	**6.0**	**0.1**
As	0.05	<0.01	Ni	0.1	<0.01
Co	0.2	<0.01	Sb	1.0	<0.01
Fe	**7.0**	**<0.7**	Sn	2.0	<0.01
Mn	0.08	<0.01	**Zn**	**8.0**	**<1.0**
Mo	0.02	<0.01	Zr	0.1	<0.01

Controlling Laboratory Environment Contamination

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- Minimize exposure:
  - Equipment, samples, blanks or standards opened in clean room, clean bench, or glove box
  - Apparatus should be covered well in plastic bag or box
- Clean work surfaces:
  - Surfaces cleaned with reagent water
- Wear powder-free gloves:
  - Sweat contains K, Pb, Ca, Mg, SO_4, PO_4, and NH_4 ions, Na and Cl
  - Powder gloves contain Zn
- Use metal free containers:
  - Volumetric flasks, beakers made out of FEP, polycarbonate, and polypropylene

Controlling Laboratory Environment Contamination

- If clean room is not available sample prep performed in a class 100 clean bench or glove box with a flow of air or N2
- Use adhesive mats at entry points to control dust and dirt from shoes
- Change shoes and / or wear shoe coverings to reduce bringing in dirt from the outside

Controlling Laboratory Environment Contamination

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- Jewelry, cosmetics, or lotions
  - *Cosmetics and lotions*
    - Fragrances, solvents, phthalates
    - Al, Be, Ca, Cd, Cu, Cr, K, Fe, Mn, Ni, Pb, Ti, & Zn
  - *Hair dyes: lead acetate*
  - *Calamine lotion: ZnO*
  - *Anti-dandruff shampoo: Se*
  - *Jewelry: Ni, Pb, Zn, Co, Au, Ag*
- Wash lab coats regularly
  - *Dust: Phthalates, Solvent residue,* Ca, Na, K, Mg, & Si
  - *Cigarette Smoke: Cd, VOA, SVOA*

Final Tips

- How do you determine if you have a clean lab?
  - By running blanks and standards!
  - *Blanks have to be clean to avoid false positive and false negative results*
  - *Carry blanks and standards through all steps of an analytical procedure*

Want to see more ?

- White Paper on BPA & Phthalates in Water
- Webinar recordings (youtube.com/spexcertiprep)
  - *Clean Lab techniques*
  - *BPA & Phthalates*
  - *Uncertainty*
- Articles
  - *Spectroscopy*
- ISO Website
- Accreditation of reference material producers: Let’s get it right! Henery F. Steger Accreditation and Quality Assurance (2003)
- Accreditation of reference material producers: an update Henery F. Steger Accreditation and Quality Assurance (2005)

References

- ISO/IEC 17025, General requirements for the competence of testing and calibration laboratories, 2005.
- ISO Guide 34, General requirements for the competence of reference material producers.
- ISO Guide 30, Terms and definitions in connection with reference materials, 2nd Ed., 1992.
- ISO Guide 31, Reference materials — Contents of certificates and labels, 2nd Ed., 2000.
- ISO Guide 35, Reference materials — General and statistical principles for certification, 3rd Ed., 2006.
- EPA Guide: 25 Years of the Clean Water Drinking Act

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Thank You!

Questions?