Inside the “Black Box” of an Environmental Testing Laboratory – Basic Analytical Procedures

Karla Buechler – Corporate Technical Director
Outline of Environmental Analysis

**Part 1** – Review of Environmental Statutes.

**Part 2** – The Black Box – Laboratory functions. What items should be considered during the method selection process? Overview of methodology including advantages and disadvantages.

**Part 3** – When and what should I communicate to the lab? What does the lab need from me? What do I need from the lab?
Congress writes a bill

The President approves [or vetoes] the bill

The Act is codified in the United States Code

Government agencies create regulations

EPA proposes a regulation; it is listed in the Federal Register for comment.

EPA reviews all comments and can revise regulation. It is then issued as a final rule.

Regulation is completed, printed as a final rule in the Federal Register & codified in the Code of Federal Regulation [CFR].

CFR is the official record of all regulations created by the federal government, divided in different volumes. Title 40 is environmental regulations.
Part 1- Regulatory Process

“Acts” refers to Acts of Congress that are made into law

- Title 40 of the Code of Federal Regulations (CFR)
- Citations are often noted as 40 CFR Part...136, 141, 761...
- Regulations Resources http://www.ecfr.gov/

In many cases, states environmental statutes implement the federal requirements

- State can go beyond the federal requirement and stand as independent state laws
State Statutes

While federal statutes have established national standards for the transportation, emission, discharge and the disposal of harmful substances, the implementation and enforcement of many large programs have been delegated by the U.S. EPA to the States.

States apply national standards to sources within their borders through permit programs that control the release of substances into the environment.
State Statutes

U.S. EPA maintains an overarching role to the states by establishing federal standards and approving state programs but most of the implementation and enforcement occurs at the state and local levels.

Examples of programs which have been delegated to the states include: National Pollution Discharge and Elimination System [NPDES] program under the Clean Water Act, Hazardous Waste Program under RCRA, and the Drinking Water Programs under the Safe Drinking Water Act.
Part 1 - Regulatory Process

- Federal & State Environmental Statutes
- Federal & State Regulations
- Drives the Method Selection for Environmental Testing Programs
Part 1- Federal Statutes

**Safe Drinking Water Act [SDWA]**

- 40 CFR Part 141
  - 500 method series for organics [524.2; 504.1]
  - 200 method series for metals [200.7; 200.8]
  - EPA, ASTM & Standard Methods for wet chemistry
  - 900 method series for radiochemistry [900.0; 903.0]

**Clean Water Act [CWA]**

- 40 CFR Part 136
  - 600 and 1600 method series for organics
  - 200 method series for metals [200.7; 200.8]
  - EPA, ASTM & Standard Methods for wet chemistry
  - 900 method series for radiochemistry [900.0; 903.0]
Part 1- Federal Statutes

Resource Conservation & Recovery Act (RCRA)

U.S. EPA SW-846 Methods

3500, 3600, 5000 & 8000 series for organics [3540C & 3660B & 8082A or 5035 & 8260B]

3000 & 7000 series for metals [3050 & 6020B]

9000 series for wet chemistry [9060]

1000 for waste characterization & leaching methods [1311 for leaching method followed by analytical metals]
Part 1 - Federal Statutes

Toxic Substance Control Act [TCSA] for PCBs

- 40 CFR Part 761
- Refers to SW-846 for PCB extraction & analysis

Comprehensive Environmental Response, Compensation & Liabilities Act [CERCLA]

- Contract Laboratory Program [CLP] Analytical Statements of Work
- Organic
- Inorganic
- PCB Congeners & Dioxins/Furans by HR/MS
Part 1- Other Federal Statutes

- Clean Air Act [CAA]
- Marine Protection, Research & Sanctuaries Act [MPSRA]
- Federal Insecticide, Fungicide & Rodenticide Act [FIFRA]
- National Environmental Policy Act [NEPA]
- Oil Pollution Act
- And others…
Part 1 - Project Definition

- What is the law?
- Why must I do the testing?
- What decision do I need to make?
- What are my method selection considerations?
Part 2 - Method Selection Considerations

Project Needs and Method Selection

- Regulatory Programs
- Detection Levels
- Project Objectives
- Levels of certainty
- Previous Analytical Activities
- Subsequent Analytical Activities
- Costs
Part 2 - Continue – Method Selection Criteria

Laboratory Capabilities and Method Selection

- Sensitivity - Method Detection Levels
- Selectivity (identification)
- Laboratory method offerings
- Laboratory Certifications
- Precision and Accuracy
- Reproducibility
- Reporting Capabilities
- Costs/value additions
DDT – Method Comparison
8081 vs 8270 vs 1699
Perchlorate – Method Comparison 314 vs 331 vs 6850
Arsenic – Method Comp 6010 vs 6020 vs 6020 collision cell

Selectivity | Sensitivity | Cost

6010
6020
6020 w/cell
Part 2 - Project Initiation

- Involve the lab immediately.
- What methods do you need?
- What type of samples?
- What type of containers?
- What analytes at what sensitivity?
Part 2 - Communication and Documentation

- Quotation
- SOW
- SAP
- QAPP
- Project kick-off call
- COC
- Bottle orders
- Sample confirmation
Part 2 – Who Makes up the Black Box?

General and Administrative Functions
Management and Business
Quality Assurance
Project Management & Customer Service
Sales and Marketing
Field Services
Sample Receipt and Login
Report Production
Part 2 - What Makes up the Black Box?

**Technical and Analytical Functions**
- Organic Sample Prep
- Metals Sample Prep
- Classical Chemistry
- Metals Analyses
- GC Organics Analyses
- GCMS Analyses
- Specialty Analyses (Rad, Micro, Geotech, Dioxin, PFAS, etc..)
Part 2 – Organic Methods Performed in the Black Box

1. Sample collection and shipment to the lab.

2. Sample pre-extraction steps.

3. Sample extraction and clean up.

4. Sample extract Concentration.

5. Sample analysis.


End report to client.
Part – 2 Sample Collection

- Field quality control samples
- Bottles, preservatives & holding times
- Chain-of-custody (CoC)
- Packing a cooler
- Sample acceptance at the laboratory
- Sample acknowledgements

Let the analysis/fun begin!!!
Part 2 - Sample Pre-extraction Steps

Goal - We need a small representative sample of a larger sample.

- Homogenization
- Subsampling
- Drying
- Grinding
- Sieving
Part 2 - Organic Sample Extraction Methods

Goal – To selectively remove the target analytes from other interfering components in the sample.

- Liquid/liquid separatory funnel extraction (Aq)
- Solid phase extraction (SPE) (Aq)
- Sonication (Solid)
- Soxhlet (Solid)
Part 2 - Organic Sample Cleanups

**Goal** – To further isolate our target analytes from the sample matrix interferences.

- Gel Permeation Chromatography (GPC)
- Solid Phase Extraction (SPE) including silica, florisil, C18
- Sulfuric Acid
- Mercury
Part 2 - Organic Sample Concentration

Goal – To increase the sensitivity of the method by decreasing the extract final volume.

- Kuderna Danish (KD)
- Rotary evaporators (RotoVap)
- TurboVap/RapidVap
## Organic Instrumentation

<table>
<thead>
<tr>
<th>Inst.</th>
<th>Pros</th>
<th>Cons</th>
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</thead>
<tbody>
<tr>
<td>GC</td>
<td>Cost effective, rugged, flexible, sensitive</td>
<td>Interferences, selectivity, need site history</td>
</tr>
<tr>
<td>GCMS</td>
<td>Selectivity, flexible, long analyte list, identification</td>
<td>Cost, sensitivity, expertise needed</td>
</tr>
<tr>
<td>HPLC</td>
<td>Same as GC</td>
<td>Same as GC</td>
</tr>
<tr>
<td>LCMS</td>
<td>Same as GCMS</td>
<td>Same as GCMS</td>
</tr>
</tbody>
</table>
Part 2 - Organic Analytical Methods

- Pesticides – including DDT (Method 8081, 8270, and 1699)
- Perchlorate (314.0, 314.1, 8321, 6850, 6860, 331)

PCBs, Herbicides, Volatiles, Hydrocarbons, PAHs, PFAS (PFOA and PFOS), Dioxins, etc.
1. Aqueous samples are collected in glass and shipped chilled to the lab.

2. Sample pre-extraction steps. Consult client if ½ inch of sediment present.

3. Sep funnel extraction with 3X60 mls of DCM. Routine cleanups include Florisil and Sulfur.

4. Concentrate the extract using Kuderna-Danish followed by N-evap.

5. Sample analysis by GC/ECD with dual column confirmation.

Part 2 - Additional Analyses

Goal – Get useful data the first time.

- Re-extractions
- Re-injections
- Dilutions
- Confirmations
- QC failures
Part 2 – Inorganic Methods Performed in the Black Box

1. Same

2. Same

3. Sample extraction (no cleanups).

4. No sample extract concentration.

5. Same

6. Same
Part 2 - Inorganic Sample Preparation

Goal – To break down the sample matrix while leaving the elements of interest in tact and in solution.

- Acid Digestion
- Microwave Digestion
- Distillation
- Leaching procedures
### Part 2 - Inorganic Instrumentation

<table>
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<tr>
<th>Instrument</th>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>ICP</td>
<td>Speed, cost effective, automation, identification, higher concentration ranges, broader range of matrix types historically</td>
<td>Sensitivity, spectral interferences</td>
</tr>
<tr>
<td>ICPMS</td>
<td>Increased selectivity and sensitivity, As interference</td>
<td>Intended for trace analyses</td>
</tr>
<tr>
<td>ICPMS (w/Collision cell)</td>
<td>Increased selectivity and increased sensitivity for all matrices, no As interference</td>
<td>Intended for trace analyses</td>
</tr>
</tbody>
</table>
Part 2 - Inorganic Analytical Methods

- Method 6010/EPA 200.7
- Method 6020/EPA 200.8
- Method 6020 with collision cell
- 7470A for Mercury
- 7196A for Hex Chrom
- 9040B for Alkalinity
- And many many more
Part 2 – Method Selected for (Aq) Arsenic by Method SW-846 6020A

1. Sample collection in nitric acid preserved plastic or glass.

2. Sample pre-extraction steps. Mix by shaking and pour aliquot immediately.

3. Digest the sample by adding HNO3 and HCL. Heat to reduce volume to 20 mL.

4. Sample extract Concentration. None

5. Sample analysis by ICP/MS.

Part 3 – Client Laboratory Communication Exchange

Phase 1 – You tell us what you need. We tell you what we can do.

Phase 2 – We tell you what we did and explain why in the case narrative.

Phase 3 – You tell us if we met your needs.
Part 3 - How do I get a price quotation?

- Contact your CRM team
- Contact your PM
- Contact your AE
- Submit a request on our website
- Call (866) 785-LABS (5227)
Part 3 - Why does TAT change?

- Change orders
- Sample matrix related concerns
- Positive target analyte levels
- Laboratory QC outside limits
- Instrument maintenance
Part 3 - The Case Narrative

- Sample condition upon receipt
- Chain-of-custody (CoC) discrepancies
- Unusual sample preparation issues
- Unusual sample analysis issues
- Depends on regulatory program
- If you want to see something ask for it
Part 3 - The Final Deliverables

You will get a report.

You will get a case narrative.

You will get an EDD.

You get to give us feedback.

You will get an invoice.
The next webinar in this basic series is May 17th!
Please join us for our next webinar!
Thank you for attending

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