# Analytical Issues You Don't Need to be a Chemist to See
## (...and Some You Do)

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# Data Errors Happen!

- High-quality, correct analytical is critical to good decision-making
- Samples are subject to many field and laboratory handling steps that may introduce error
- 'Reasonability' review may help spot issues that require further investigation

# What is a Reasonability Review?

- Simply: do the numbers make sense?

- Total results vs. Dissolved results
- Field Duplicates comparison
- Comparison between parameters
- Blank Results
- Consistency between lab report and EDD
- Comparison to historical results for same location*

** Useful to identify anomalies, but be careful not to make assumptions about what results 'should' be*

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# Not a Chemist? Be a Detective.

- Incorrect data or suspect data may be identified by carefully looking for clues
- Inquiries to field personnel and laboratory partners may be needed to resolve apparent issues

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# Clue: Mismatched Results

| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS N |
|---|---|---|---|---|---|---|---|---|
| **6020B MET ICPMS, Dissolved** | Analytical Method: EPA 6020B Preparation Method: EPA 3020A | | | | | | | |
| Thallium, Dissolved | ND | ug/L | 0.10 | 0.041 | 1 | 12/17/21 09:50 | 12/22/21 15:15 | 7440-28- |
| Vanadium, Dissolved | 0.43J | ug/L | 1.0 | 0.16 | 1 | 12/17/21 09:50 | 12/22/21 15:15 | 7440-62- |
| Zinc, Dissolved | 3.1J | ug/L | 5.0 | 2.0 | 1 | 01/07/22 05:57 | 01/07/22 17:04 | 7440-66- |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | Thallium, Dissolved | 7440-28-0 | D | ug/L | | U | |
| | Vanadium | 7440-62-2 | T | ug/L | 0.55 | J | RL |
| | Vanadium, Dissolved | 7440-62-2 | D | ug/L | 0.43 | J | RL |
| | Zinc | 7440-66-6 | T | ug/L | 2.5 | J | RL |
| | Zinc, Dissolved | 7440-66-6 | D | ug/L | 63.9 | J | M- |
| SW846 7470A | Mercury | 7439-97-6 | T | ug/L | | U | |

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# Clue: Mismatched Results

- What did we see?
  - Mismatched results between the lab report and the EDD
- Why is that a clue?
  - Laboratory report and EDD must match exactly

# Clue: Mismatched Results

- The lab indicated that the result reported in the data package was correct and that the result in the EDD was incorrect.
- A revised EDD was provided, and data were reloaded.

# Clue: Inconsistent Field Duplicates

| Sample ID: | MW-12 | | | | Duplicate Sample ID: | | MW-12D | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Analyte | Sample Concentration | Qual | QL | MDL | Duplicate Concentratio | Qual | QL | MDL | Difference | RPD | Flag |
| arsenic | 22 | | 1 | 0.28 | 3.4 | | 1 | 0.28 | 18.6 | NA | J |
| barium | 68 | | 10 | 3.1 | 50 | | 10 | 3.1 | NA | 31% | J |
| calcium | 120000 | | 500 | 130 | 64000 | | 500 | 130 | NA | 61% | J |
| cobalt | 11 | | 0.5 | 0.26 | 0.98 | | 0.5 | 0.26 | 10.02 | NA | J |
| lead | 0.36 | J | 1 | 0.17 | 0.81 | J | 1 | 0.17 | NA | NA | |
| lithium | 36 | | 5 | 0.83 | 25 | | 5 | 0.83 | NA | 36% | J |
| molybdenum | 17 | | 5 | 0.61 | 39 | | 5 | 0.61 | 22 | NA | J |
| thallium | 0.56 | J | 1 | 0.47 | 0.47 | U | 1 | 0.47 | NA | NA | |
| TDS | 1200 | | 20 | 16 | 940 | | 20 | 16 | NA | 24% | J |
| chloride | 300 | | 5 | 1.4 | 210 | | 5 | 1.4 | NA | 35% | J |
| fluoride | 200 | | 50 | 24 | 29 | J | 50 | 24 | 171 | NA | J |
| sulfate | 240 | | 1 | 0.35 | 150 | | 1 | 0.35 | NA | 46% | J |

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# Clue: Inconsistent Field Duplicates

- What did we see?
  - Disparity between parent and FD results
- Why is that a clue?
  - Imprecision between most or all parameters can indicate potential sample switch or incorrect association to parent
  - Homogeneous matrices (like GW) should be consistent with good sampling practices

# Clue: Inconsistent Field Duplicates

- Additional similar inconsistencies observed for other field duplicates submitted on the same day
- Field duplicate had been switched with a field duplicate from a different SDG
  - 3 SDGs delivered to the laboratory on the same day; FDs in each SDG were named identically
- 3 reports and EDDs were revised
- Corrective action requested

# Clue: Comparison Between Parameters

| Parameter | Result | Qualifier | MDL | QL | Units |
|---|---|---|---|---|---|
| Total Dissolved Solids | 3.40 | U | 3.40 | 14.3 | mg/L |
| Chloride | 4.71 | | 0.0670 | 0.200 | mg/L |
| Fluoride | | U | 0.0330 | 0.100 | mg/L |
| Sulfate | 0.685 | | 0.133 | 0.400 | mg/L |
| Alkalinity, Total | 16.0 | | 16.0 | 16.0 | mg/L |
| Bicarbonate alkalinity | 16.0 | | 16.0 | 16.0 | mg/L |
| Carbonate alkalinity | | U | 1.45 | 4.00 | mg/L |
| Calcium | 3640 | | 80.0 | 200 | ug/L |
| Magnesium | 1670 | | 10.0 | 30.0 | ug/L |
| Potassium | 120 | J | 80.0 | 300 | ug/L |
| Sodium | 3480 | | 80.0 | 250 | ug/L |

# Clue: Comparison Between Parameters

- What did we see?
  - “Not-detected” TDS result not supported by other parameters
- Why is that a clue?
  - TDS results can be checked using results for cations, anions, and alkalinity when available
    - Calculated TDS for this sample ~3500 mg/L
  - Cation/anion balance can also help identify potential errors

# Clue: Comparison Between Parameters

- Standard Methods 1030E provides several equations for checking results
- Laboratory did not find any errors with analysis; sample was reanalyzed to confirm
- Reanalysis did NOT confirm original results, sample data were revised to report reanalysis

# Clue: Suspicious Consistency in Rad Data

| Analyte | Result<br>pCi/l | Qualifier | Uncertainty<br>+ / - | MDA<br>pCi/l |
|---|---|---|---|---|
| Radium-226 | 0.450 | | 0.312 | 0.0500 |
| *(T) Barium* | *92.1* | | | *30.0-143* |

| Analyte | Result<br>pCi/l | Qualifier | Uncertainty<br>+ / - | MDA<br>pCi/l |
|---|---|---|---|---|
| Radium-226 | 0.694 | | 0.393 | 0.0500 |
| *(T) Barium* | *89.6* | | | *30.0-143* |

(MB) R3786317-2 04/27/22 15:26

| Analyte | MB Result<br>pCi/l | MB Qualifier | MB Uncertainty<br>+ / - | MB MDA<br>pCi/l |
|---|---|---|---|---|
| Radium-226 | 0.189 | | 0.140 | 0.0500 |
| *(T) Barium* | *90.1* | | *90.1* | |

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# Clue: Suspicious Consistency in Rad Data

- What did we see?
  - Identical Minimum Detectable Activity (MDA) values for several samples and a method blank
- Why is that a clue?
  - Results, uncertainty, and MDAs are calculated on a sample-specific basis for radium analyses

# Clue: Suspicious Consistency in Rad Data

- Upon inquiry, laboratory indicated that sample and QC results did not take detector background into account
- Results were revised for 2 investigatory samples, the field duplicate, field blank, and laboratory method blank
- Impacted radium-226 and combined radium-226+228 results

# Clue: Multiple Lines of Evidence

- Reported copper result of 106 ug/L was inconsistent with historical data
  - Historical data < 10 ug/L, often ND
- Result was a new UPL exceedance (not previously observed at that location
- Result did not agree with FD result (< 0.50 ug/L)

# Clue: Multiple Lines of Evidence

- What did we see?
  - FD imprecision
  - Results significantly out of line with historical data
- Why is are those clues?
  - Multiple issues indicated a potential problem
  - Significant differences from historical data may indicate need for further investigation

# Clue: Multiple Lines of Evidence

- Laboratory requested to check bottles to confirm labeling
- Laboratory reported distinct color difference between parent and field duplicate
- Both bottles indicated as preserved with nitric acid


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# Clue: Multiple Lines of Evidence

- Coloration of parent sample bottle was consistent with other unpreserved bottles for that location
- Laboratory requested to check pH of both bottles
- Parent sample bottle determined to be pH 7

- Laboratory added preservative and reanalyzed sample for all metals and mercury
  - Copper was 2.8 ug/L upon reanalysis

# Data Errors Happen…

- …and you don't necessarily need to be a chemist to spot them!
  - Relatively simple reasonability reviews can identify issues
- Use the clues in the data to identify results that may need further investigation
  - Work with field sampling personnel and laboratory partners to investigate suspect data
- When a result doesn't "feel" right but you can't find an issue – call a chemist friend!

# Thank You

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