Lab Symposium Breakout: Advanced Instrumentation

DMACC Wellness Center
Ankeny, Iowa
September 26, 2019
Method Detection Limits
Revision 2

- Clean Water Act, 2017 MUR – not applicable to SDWA
- \( \text{MDL}_B \) = MDL determined from method blanks
- \( \text{MDL}_S \) = MDL determined from matrix spikes
- Your MDL is the larger of \( \text{MDL}_S \) or \( \text{MDL}_B \)
- Samples used to calculate the MDL are representative of laboratory performance throughout the year, rather than on a single date.
- Option to pool data from multiple instruments to calculate one MDL that represents multiple instruments.
MDL’s – Ongoing Verification

At least once every 13 months:

- Calculate MDL\(_S\) and MDL\(_B\)
- Use data generated within last 24 months – include initial MDL\(_S\) if within 24 months.
- Only use data associated with acceptable calibrations and batch QC.
- Option: Use MB’s from last six months or most recent 50, whichever is greater.
- Option: If verified MDL is within 0.5 to 2.0\times\) existing MDL, no change required.
MDL’s – Initial Verification

New method or rarely used

• Process minimum of seven spikes and seven blanks.
• Prepared on three separate calendar dates.
• Analyzed on three separate calendar dates.
• Can be prepared and analyzed on the same day.
Comparison Studies

Demonstrate equivalency between methods.

• Example: Compare GFAA and ICPMS for analysis of lead in blood.

• Analyzed 52 samples by both methods.

• Tabulate results w/sample identifiers.
Comparison Studies

• Excel: Data tab, Data Analysis (load Data Analysis Toolpak if necessary)
• t-test: Paired Two Sample for Means

<table>
<thead>
<tr>
<th>Lab #</th>
<th>Tube #</th>
<th>Type</th>
<th>AA600 Results</th>
<th>NexION Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>589269</td>
<td>AGI777</td>
<td>V</td>
<td>0.06</td>
<td>1.94</td>
</tr>
<tr>
<td>589270</td>
<td>AGG478</td>
<td>V</td>
<td>0.28</td>
<td>0.697</td>
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<tr>
<td>589271</td>
<td>AGA882</td>
<td>V</td>
<td>5.35</td>
<td>5.318</td>
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<tr>
<td>589272</td>
<td>AEG500</td>
<td>V</td>
<td>0.56</td>
<td>0.715</td>
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<tr>
<td>589273</td>
<td>AGE206</td>
<td>V</td>
<td>0.70</td>
<td>0.861</td>
</tr>
<tr>
<td>589274</td>
<td>AGE207</td>
<td>V</td>
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<td>589275</td>
<td>AGE900</td>
<td>V</td>
<td>3.64</td>
<td>3.34</td>
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</table>
Comparison Studies

http://www.statisticshowto.com/how-to-do-a-t-test-in-excel/

<table>
<thead>
<tr>
<th>t-Test: Paired Two Sample for Means</th>
<th>AA600 Results</th>
<th>NexION Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>2.592307692</td>
<td>2.382711538</td>
</tr>
<tr>
<td>Variance</td>
<td>24.1783083</td>
<td>15.6236648</td>
</tr>
<tr>
<td>Observations</td>
<td>52</td>
<td>52</td>
</tr>
<tr>
<td>Pearson Correlation</td>
<td>0.988384871</td>
<td></td>
</tr>
<tr>
<td>Hypothesized Mean Difference</td>
<td>0</td>
<td>Null hypothesis: Sample means are the same.</td>
</tr>
<tr>
<td>df</td>
<td>51</td>
<td></td>
</tr>
<tr>
<td>t Stat</td>
<td>1.285815878</td>
<td></td>
</tr>
<tr>
<td>P(T&lt;=t) one-tail</td>
<td>0.102157483</td>
<td></td>
</tr>
<tr>
<td>t Critical one-tail</td>
<td>1.67528495</td>
<td></td>
</tr>
<tr>
<td>P(T&lt;=t) two-tail</td>
<td>0.204314966</td>
<td>p-value &gt; alpha (0.05) -- null hypothesis supported</td>
</tr>
<tr>
<td>t Critical two-tail</td>
<td>2.00758377</td>
<td>t-Stat&lt; t-critical value -- null hypothesis supported</td>
</tr>
</tbody>
</table>
Interelement Correction Factors

• Spectral overlap vs. Background correction.
• +/- 20% acceptable?
• Vendor software tools
• Manual methods
Collision/Reaction cells

- Cannot be used for drinking water.
- Can be used for wastewater provided that you document that the performance specs are met in collision mode. (40 CFR Part 136.6 allows modifications that improve performance of CWA methods without EPA review.)
Triple Quad

- ICP-QQQ-MS = ICP-MS + $100,000
- Interference and matrix removal
- Unleash potential of reaction cell
Contact

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