Standardization of Procedures and Controls
in Chemical Challenge Testing of Respirator Cartridges and Canisters

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Service Life Testing
with Chemical Challenge Agents

• Conducted by a small community of expert labs who have developed esoteric test methods containing complex elements that are not well-understood or discussed outside a small circle of aficionados.
  – Could be considered by some as a “Cult”

• Perhaps, some aspects of practices in our cult could be improved by an increased dialogue leading to increased …
  – Standardization of Procedures and Controls
Aspects of Chemical Challenge Agent Testing

• Difficult & Highly Specialized
  – Methods are not taught in any Schools

• Small No. of Qualified Laboratories
  – Majority are Government Labs

• Small No. of Vendors of Test Equipment
  – Custom-made Equipment is Prevalent

• Few Forums for Idea-Sharing Between Labs
  – No Specialist Journals or Technical Meetings
Analytical Chemistry Testing
Food and Drug Testing, Blood Testing, Air Sampling

• Difficult & Highly Specialized
  - But Methods ARE taught in Schools

• Large No. of Qualified Laboratories
  - Accreditation of Labs is common

• Large No. of Vendors of Test Equipment
  - Example: Pittsburgh Conference (Pittcon)

• Many Forums for Idea-Sharing Between Labs
  - Many Journals and Technical Meetings
Technical Associations who have more standardized methods (procedures & controls)

- College of American Pathologists (blood chemistry)
  - Proficiency Testing

- United States Pharmacopeia (drug chemistry)
  - Standard Methods & Proficiency Testing

- Association of Analytical Communities (food chemistry)
  - Standard Methods & Proficiency Testing

- American Industrial Hygiene Assn (air sampling)
  - Proficiency Testing
Aspects of Standardization

• Standardization of Procedures & Controls
  – Writing and Communicating Methods (Procedures & Controls) among many users
  – Round Robin Testing Protocols
  – Test Method Evaluation
  – General Improvement in Test Methods

• Round Robin (Proficiency) Testing
  – Uniform Test Articles distributed to Labs.
  – Compare Results from different Labs.
What Happens

When Standardization of Procedures & Controls is Implemented

• Standard Test Methods
  - Users evaluate methods more closely
  - Users argue about method details
  - Users publish articles about Methods

• Round Robin (Proficiency) Testing
  - Labs are supposed to be evaluated
  - Test Methods are actually evaluated
Need for Standardization

How do we know if Standardization and Proficiency Testing are necessary?

• A Lab testing replicate articles (believed to be “identical”) obtains results that seem to be significantly different.

• Two different Labs testing seemingly “identical” articles obtain results that seem to be significantly different.
To Make a Case

For Standardization of Procedures & Controls

• We will talk a little about Test Method Evaluation which looks into the variation of procedures we use in Chemical Challenge Testing.

• Listeners can reflect upon whether or not, in your experience, replicate tests of (seemingly) identical articles often lead to significantly different results.
Test Result Variations

- Test Variations can be observed in Round Robin Tests using
  - Different Labs
  - Different Analysts (Same Lab)
  - Different Procedures
Test Method Evaluation

*Scientific approach ...*

- Seeks to analyze methods rather than blame people or labs for differences in test results.

- Control of Test Parameters contained within each Test Methods lead to control of Test Results.
Terms in Test Method Evaluation

Meaning of “Error”...

- ERROR = VARIABILITY Due to Extraneous Factors
  - Factors other than the TEST ITEM
  - Observed in REPLICATE TESTS

Significance ...

- In Evaluation of APR Service Life …
  VARIABILITY due to Extraneous Factors needs to be minimized.
Test Variations observed when identical Items are Tested multiple times

To evaluate Items fairly, Test Variations must be minimized
Test Method Variability

Questions...

- Do variations that are allowed in the current Tests cause substantial variations in measured Service Lives independent of the Test Item?

- How much does each parameter contribute to overall measured variation in Service Life?

- Is there a way to control such variations?
How Test Variations Arise

Causes...

• The Test Result (Service Life, min) has a characteristic sensitivity to each Test Parameter

• Five (5) or more Test Parameters must be accurately and precisely applied during the Test

• Some Test Parameters are more difficult to Control than others

• The Test Result is more sensitive to the some Test Parameters than others
**Chemical Challenge Tests**

*Basic Test Parameters ...*

- Challenge Agent Conc’n (ppm)
- Air Flow Rate (L/min)
- Time of Test (min)
- Break-Through Conc’n (ppm)
- Air Conditioning (Temp & RH)
- Pre-Conditioning (Temp, RH, and Flow Rate)
Chemical Challenge Tests

Secondary or “Hidden” Test Parameters …

• Temperature effect on Relative Humidity  
  - an error in one propagates to the other

• Gas Concentrations (at various times)

• Accuracy of purchased Gas Standards

• Instrument Accuracy (at various times)

• Instrument Selectivity (interferences)

• The Written Test Method (itself) - Whether all Test Parameters are clearly specified
Sensitivity of Test Result to Test Parameters

- The Test Result is more sensitive to variations in some Test Parameters than in others:
  - A - Not Very Sensitive
  - B - Proportional
  - C - Very Sensitive
Sensitivity of Test Result to Test Parameters

- The Test Result is more sensitive to some Test Parameters
  - Control of Challenge Agent Concentration
  - Control of Flow Rate

- Not so sensitive to
  - Temperature
    - (Measured as °C)
Sensitivity of Test Result to Test Parameters

• The Test Result is more sensitive to some Test Parameters
  - Humidity Variation above 80%

• Not so sensitive to
  - Measurement of Break Through Concentration
  - Variation in control of RH at 25-50%
## Estimated Variation in Generating Challenge Agents

<table>
<thead>
<tr>
<th>Type of Challenge Agent</th>
<th>Agent Concentration</th>
<th>Control &amp; Measurement</th>
<th>Estimated Variation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Stable, Compressed Gas</td>
<td></td>
<td>± 5 - 50 %</td>
</tr>
<tr>
<td></td>
<td>Stable, Volatile Liquid</td>
<td></td>
<td>± 5 %</td>
</tr>
<tr>
<td></td>
<td>Reactive Liquid</td>
<td></td>
<td>± 10 %</td>
</tr>
<tr>
<td></td>
<td>Non-Volatile Liquid or Solid</td>
<td></td>
<td>± 10-50 %</td>
</tr>
</tbody>
</table>
Error Budgeting in Test Methods

Estimates …

<table>
<thead>
<tr>
<th>Test Parameter</th>
<th>Allowed Parameter Variation</th>
<th>Induced Test Result Variation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Agent Conc’n</td>
<td>± 10 %</td>
<td>± 10 %</td>
</tr>
<tr>
<td>Flow Rate</td>
<td>± 3 %</td>
<td>± 3 %</td>
</tr>
<tr>
<td>Temp</td>
<td>± 5 %</td>
<td>± 1 %</td>
</tr>
<tr>
<td>RH</td>
<td>± 5 %</td>
<td>± 1 %</td>
</tr>
<tr>
<td>Break-Through Measurement</td>
<td>± 5 %</td>
<td>± 1 %</td>
</tr>
</tbody>
</table>

TOTAL Test Variation \(\text{estimated}\) = ± 16 %
Effect of Moisture Load on Measured Service Life

Example…

CCI4 Service Life as a function of H2O Loading of Cartridge (Product A)

CCI4 Service Life (min)

0 5 10 15 20

Qty H2O Sorbed During Pre-Cond (gm)
Effect of Humidity on Measured Service Life

Example...

CCI4 Service Life as a function of Temp & RH Variations in Pre-Conditioning
(Product B)
Making a Case

For Standardization of Procedures & Controls

• We have now looked into the variation of some of the processes and procedures we use in APR Cartridge and Canister Testing.

• Listeners have had time to reflect on whether, in your experience, replicate tests of (seemingly) identical articles often have led to significantly different results.
Aspects of Standardization

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How Would We Do Proficiency (Round Robin) Testing?

• Acquire a significant quantity of APR Cartridges believed to be uniform in content and character (i.e. a “good lot”).
  – Document SOP for Cartridge Preparation.

• Submit samples to different labs for the same type of test.
  – Specific, Control, or at least Document the Test Methodology that is actually used.

• Record test results in a format that facilitates comparison among the different labs performing the tests.
  – Specify how data is to be reported and analyzed.
# Possible Round-Robin Tests

<table>
<thead>
<tr>
<th>Agent</th>
<th>Possible Method Issues</th>
<th>Possible Lab-to-Lab Variation</th>
</tr>
</thead>
<tbody>
<tr>
<td>SO2</td>
<td>No significant problem</td>
<td>± 7%</td>
</tr>
<tr>
<td>HCN</td>
<td>Must Detect Breakthrough of 2 gases</td>
<td>± 10%</td>
</tr>
<tr>
<td>Acrolein</td>
<td>Reactive, Very Volatile Liquid</td>
<td>± 25%</td>
</tr>
<tr>
<td>ClO2</td>
<td>Must generate with Reactor using Cl2</td>
<td>± 25%</td>
</tr>
<tr>
<td>CN (Tear Gas)</td>
<td>Hugh boiling, difficult to volatilize</td>
<td>± 25%</td>
</tr>
</tbody>
</table>
Evolution

Toward future improvement ...

• Conduct Round Robin Testing.
• Publish more details of Test Methods.
• Publish discussions of Procedures & Controls and their effect on Test Results.
• Refine Test Methods.
Final Comments

- Standardization Procedures and Controls is required in order for Laboratories to comply with ISO 17025 (general standard for “testing and calibration labs”).

- ISO 17025 is incorporated into the AIHA’s current LQAP program for accreditation of Laboratories engaged in Air Sampling and analysis.

- Extension of this approach to Chemical Challenge Testing for Respirator Service Life is feasible.