Laboratory Variation in Service Life Test Results Using the NIOSH Carbon Tetrachloride Test

A Pain in the Ankle for Test Labs

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Service Life Tests

GOALS ...

• To measure Effective Service Life of Air Purifying Respirator Cartridges

• To demonstrate Differences in Performance among APR Cartridges

• To provide a Basis for Specifications
  – “Effective” vs “Ineffective” Cartridges
Service Life Tests

Desirable ATTRIBUTES ...

• Measures Effective Life of APR Cartridges as described under GOALS

• Convenient to Perform
  - rapid and cost-effective

• Low Systematic and Random Error
  - Low Variation in replicate tests from known or unknown causes
Service Life Tests

(Organic Vapor)

Representative Challenge Agents ...

• It is not practical to Test EVERY Organic Vapor

• CARBON TETRACHLORIDE has been taken to “represent” all OVs
  • Popular industrial chemical
  • Well-adsorbed on carbon
  • Adsorption sensitive to moisture
Service Life Test Variability

(Organic Vapor)

**Issues...**

- CCl4 “represents” all organic vapors under **42CFR84** in lab evaluation of Industrial APR Cartridges

- Carbon Tetrachloride Service Life is *very sensitive* to Humidity in Pre-Conditioning

- **VARIABILITY** is observed in CCl4 Tests
  - **Test-to-Test variation**
  - **Lab-to-Lab variation**
Questions & Test Method Evaluation

Concept...

If a test seems to give different results from repeat tests of similar Items, the Test Method may be questioned.

If questions are posed scientifically, They can form the basis of a Test Method Evaluation.
Service Life Test Variability

Questions...

• Do variations in Humidity and Temperature typically allowed in the current NIOSH Test 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 minimize any such errors?
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 Cartridges ... VARIABILITY due to Extraneous Factors needs to be minimized.
• Variation observed when the same Item is Re-Tested

• To evaluate Items fairly, Variability due to extraneous factors needs to be minimized

*Test Method Error*

(variation)
STUDY DESIGN

- Pre-Condition and Test APR Cartridges as described in 42CFR84
- Study the range of Temperature and Humidity variation commonly permitted
- Plot the effects of allowed Temperature and Humidity variation on Test Results
4.3.4. Two cartridges or pairs of cartridges will be equilibrated at room temperature by passing 85 percent RH air through them at 25 lpm for 6 hours and then testing them at 50 percent RH, approximately 25°C, and 32 lpm continuous air flow rate containing 1000 ppm CCl₄.
**PROTOCOL**
(Pre-Conditioning)

<table>
<thead>
<tr>
<th>25 L/min, 25°C, &amp;</th>
<th>25 L/min, 22.5°C, &amp;</th>
<th>25 L/min, 27.5°C, &amp;</th>
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<tbody>
<tr>
<td>- 80.0 %RH</td>
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<tr>
<td>- 90.0 %RH</td>
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*Each separate pair of APR Cartridges pre-conditioned as per NIOSH STP 0046 with variations shown above.*
PROTOCOL
(Testing)

- All Cartridges tested Identically as per 42CFR84 except ...

- each separate APR Cartridge tested separately at half the Flow Rate specified for the Pair.

1000 ppm CCL4 at 16 L/ min
25°C / 50 % RH
DEFINITIONS

• Relative Humidity
  - % of Saturation (Water in Air)

• Absolute Humidity
  - mg of Water per Liter of Air

\textit{at fixed Absolute Humidity, Relative Humidity increases when Temperature decreases}
Qty of H₂O Sorbed as a function of Humidity During Pre-Conditioning (Product A)
CCl4 Service Life as a function of H2O Loading of Cartridge
(Product A)

CCl4 Service Life (min)

Qty H2O Sorbed During Pre-Cond (gm)
CCI4 Service Life as a function of Temp & RH Variations in Pre-Conditioning (Product A)
CCl4 Service Life as a function of Temp & RH Variations in Pre-Conditioning (Product B)
CCl4 Service Life as a function of Temp & RH Variations in Pre-Conditioning (Product C)
SUMMARY of RESULTS

- Test Variation induced by RH Variation of 85±5% RH
  - + 25% variation (error) in measured Service Life

- Test Variation induced by Temp Variation of 25±2.5°C
  - + 25% variation (error) in measured Service Life

- Test Variation induced by combined Temp & RH Variation
  - + 50% variation (error) in measured Service Life
SUMMARY of RESULTS

• Test Variation induced by RH Variation of 85±3% RH
  • + 15% variation (error) in measured Service Life

• Test Variation induced by Temperature Variation of 25±1°C
  • + 10% variation (error) in measured Service Life

• Test Variation induced by combined Temp & RH Variation
  • + 25% variation (error) in measured Service Life
Recommendations
(from Test Method Evaluation)

• Reduce allowed Temp variation during preconditioning and test to ± 1°C

• Reduce allowed %RH variation during preconditioning and test to ± 3% RH

• Adopt a new “representative” Challenge Agent
Afterward

It would be good to ... 

• …have more evaluation, and discussion of Chemical Challenge Test Methods for cartridges and canisters

• …establish an APR Cartridge and Canister Proficiency Testing System