Development of Improved Criteria for Chemical Challenge Test Methods for Air-Purifying Respirators

Aimed at Improving Inter-Laboratory Agreement of Test Results

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If a Test Method Results seem to vary

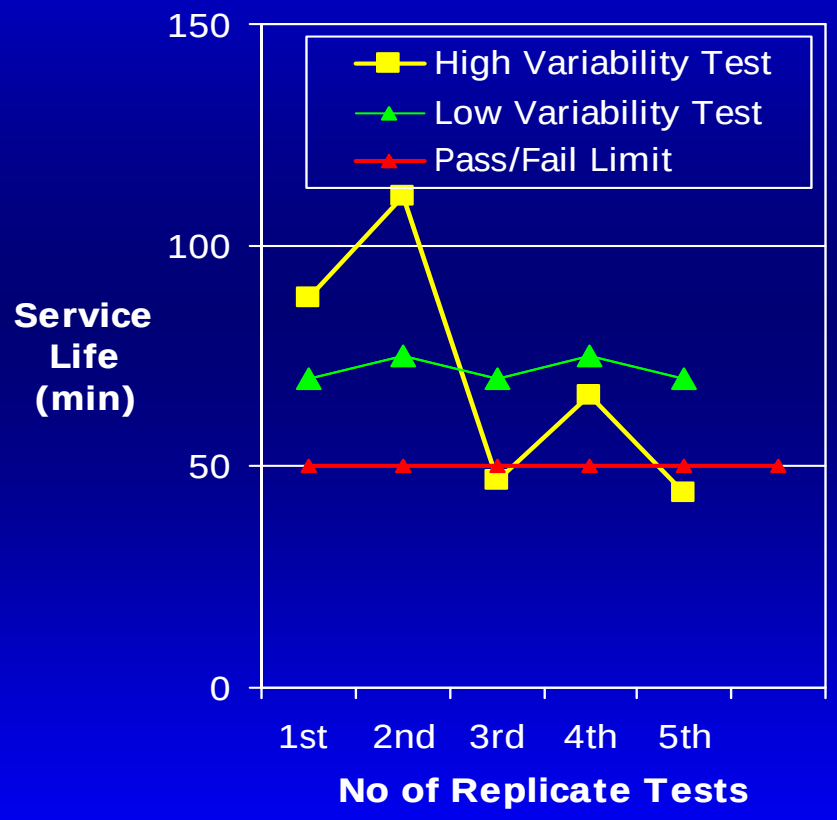
Lab-to-Lab or Test-to-Test

(withinLab)

Then, we would like to improve the Test Criteria

Assessing Method Variability

- Test Variations can be measured when Identical Items are Tested by
  - Different Labs
  - Different Analysts in same Lab
  - Different Procedures



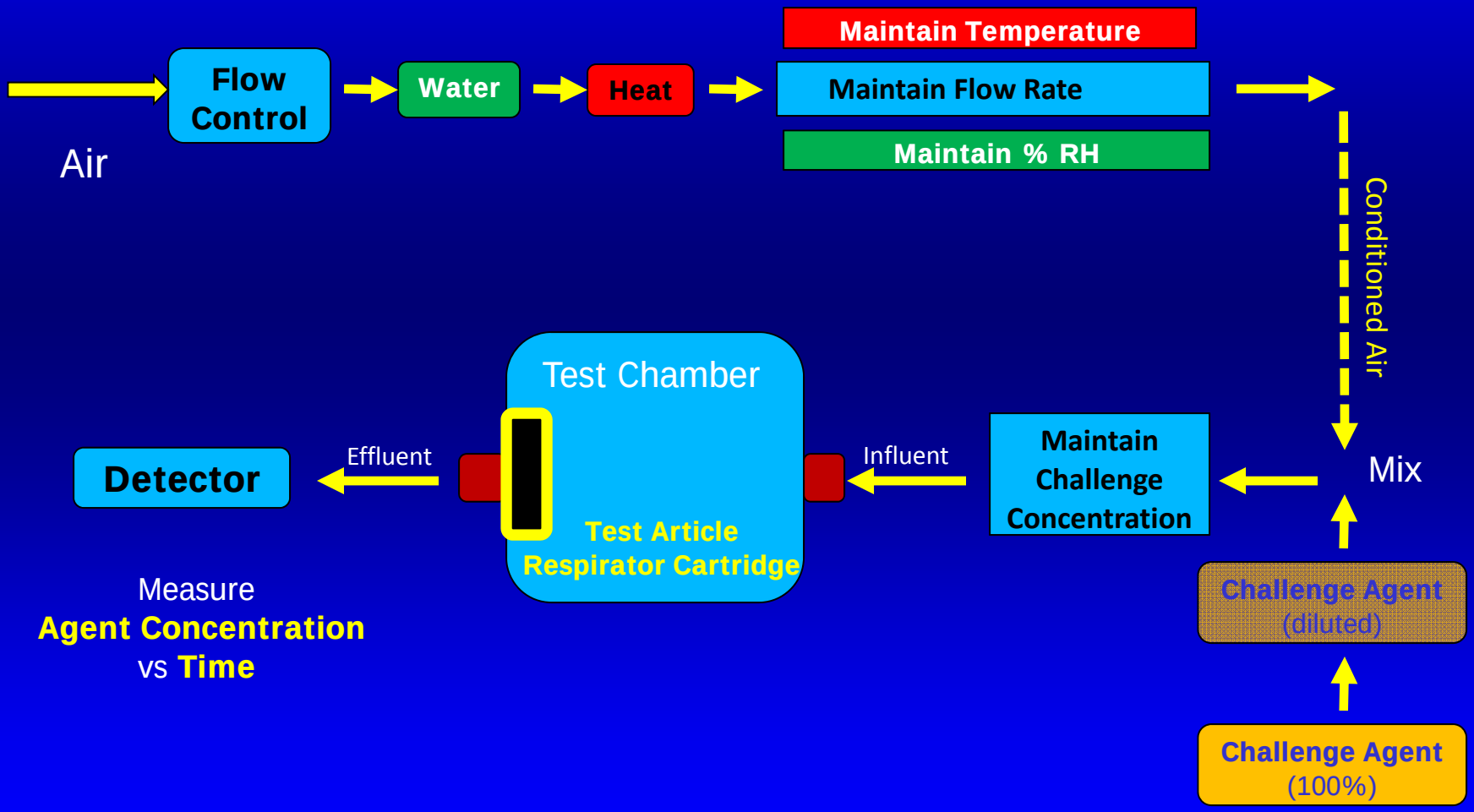
To Improve "High Variation" Methods

We need to Analyze Inside the Method

Questions...

- Do variations that are allowed in the current Tests cause substantial variations in measured Service Lives independent of the Test Item?
- Is there a way to minimize any such errors?

To begin the analysis, let's look at Anatomy of a Chemical Challenge Test



Chemical Challenge Tests

Test Parameters in Chemical Challenge …

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

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 variation of the some Test Parameters than others

Bottom Line Questions

In a specific test ...

Which test parameters are the most difficult to control?

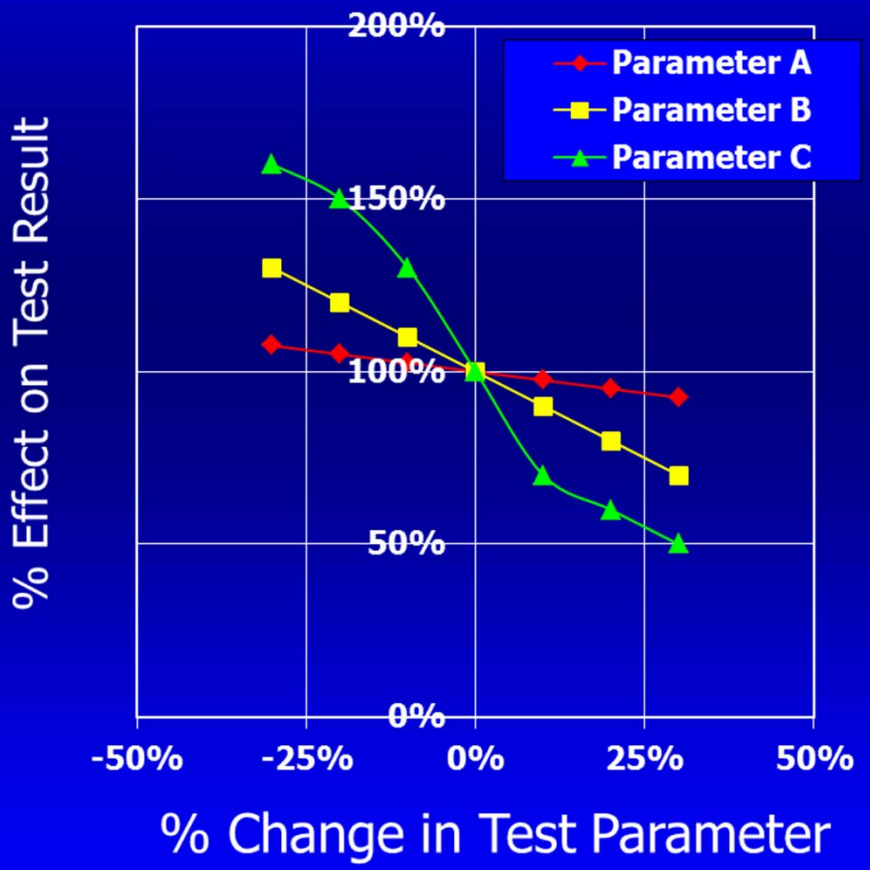
Which parameters have the greatest effect on the final result?

How Would We Measure the Effects of Test Parameter Variations on Results?

- Try to Hold Parameters Constant
- Intentionally Vary one Parameter
  - Measure Change in Results

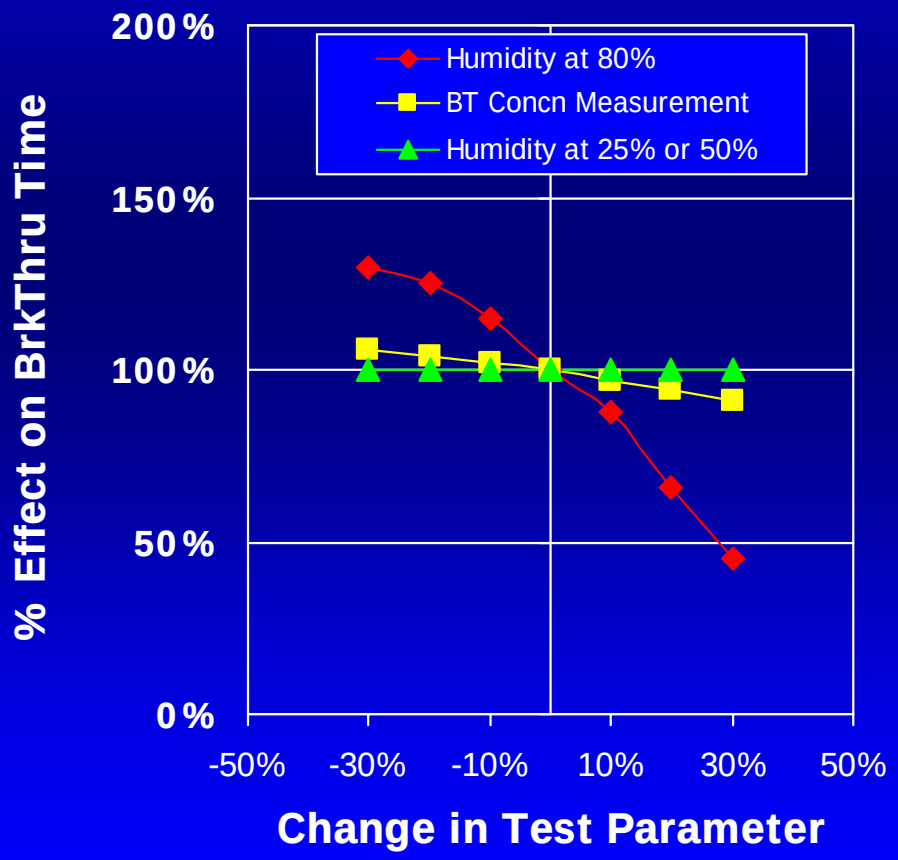
Sensitivity of Test Result to Test Parameters

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



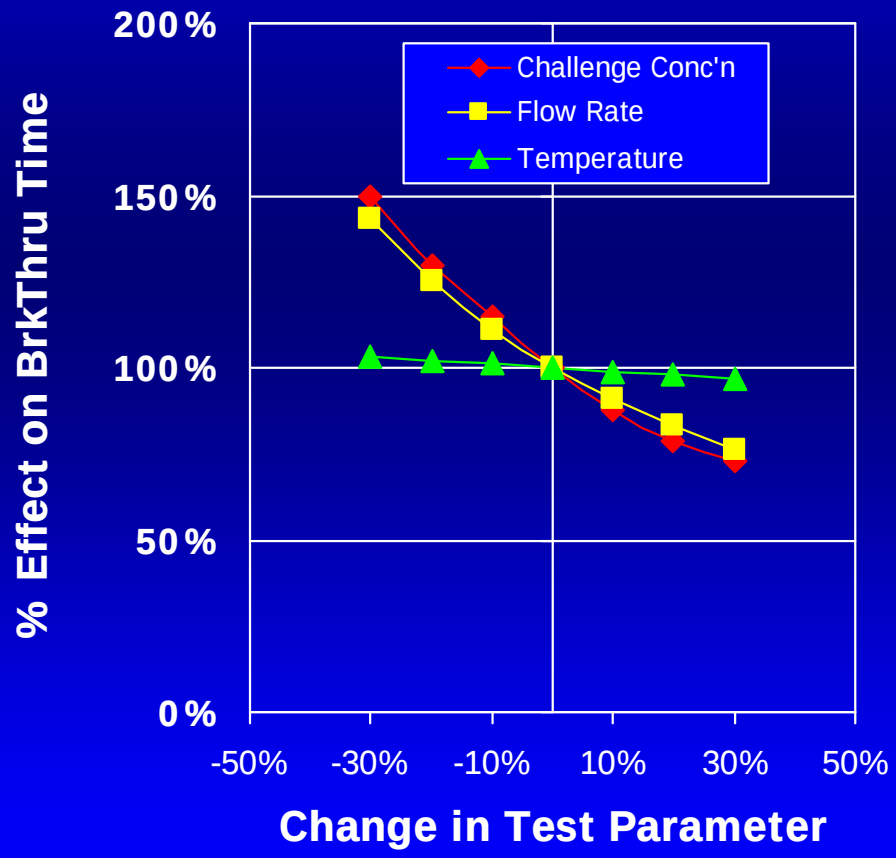
Sensitivity of Result to "Real" Test Parameters

- 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%



Additional “real world” 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 ^{o}C)



Estimated Variation
in Generating Different Challenge Agents

Type of Challenge Agent	***Estimated Variation***
Agent Concentration Control & Measurement	± 5 - 50 %
Stable, Compressed Gas	± 5 %
Stable, Volatile Liquid	± 10 %
Reactive Liquid	± 10-50 %
Non-Volatile Liquid or Solid	± 10-50 %

Error Budgeting in Test Methods

Estimates ...

Test Parameter	*Allowed Parameter Variation*	→ *Induced Test Result Variation*
Agent Conc'n	± 10 %	± 10 %
Flow Rate	± 3 %	± 3 %
Temp	± 5 %	± 1 %
RH	± 5 %	± 1 %
Break-Through Measurement	± 5 %	± 1 %

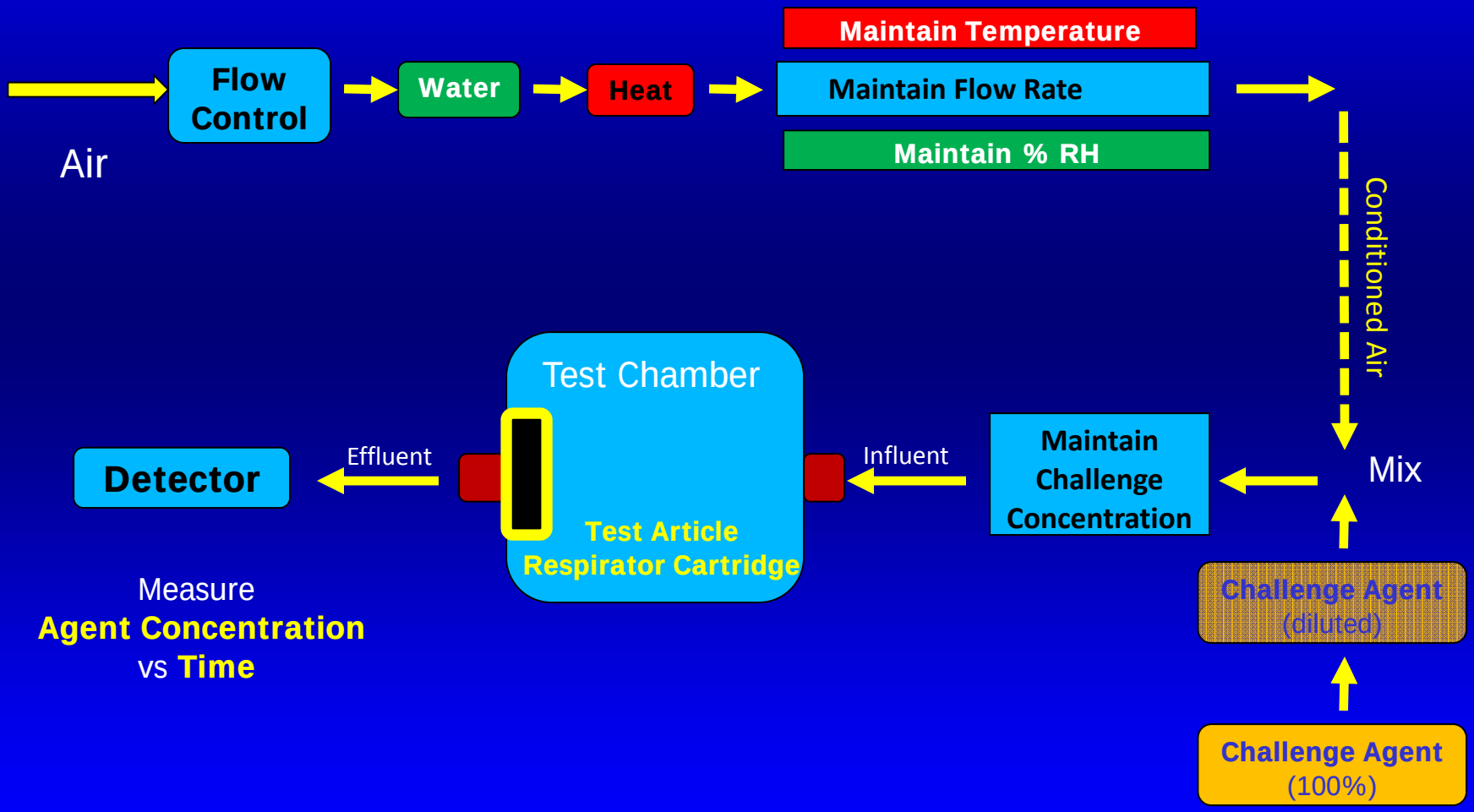
TOTAL Test Variation (estimated) = **± 16 %**

Chemical Challenge Tests

Test Parameters in Chemical Challenge …

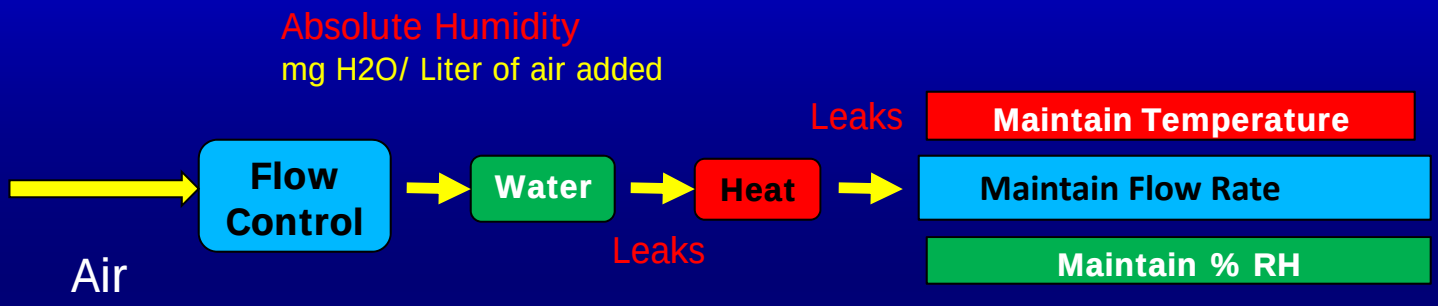
- Air Flow Rate (L/min)
- Air Conditioning (Temp & RH)
- Pre-Conditioning (Temp, RH, and Flow Rate)
- Challenge Agent Conc'n (ppm)
- Break-Through Conc'n (ppm)
- Time of Test (min)

Controlling a Chemical Challenge Test



Controlling a Chemical Challenge Test

Flow, Temperature, Humidity

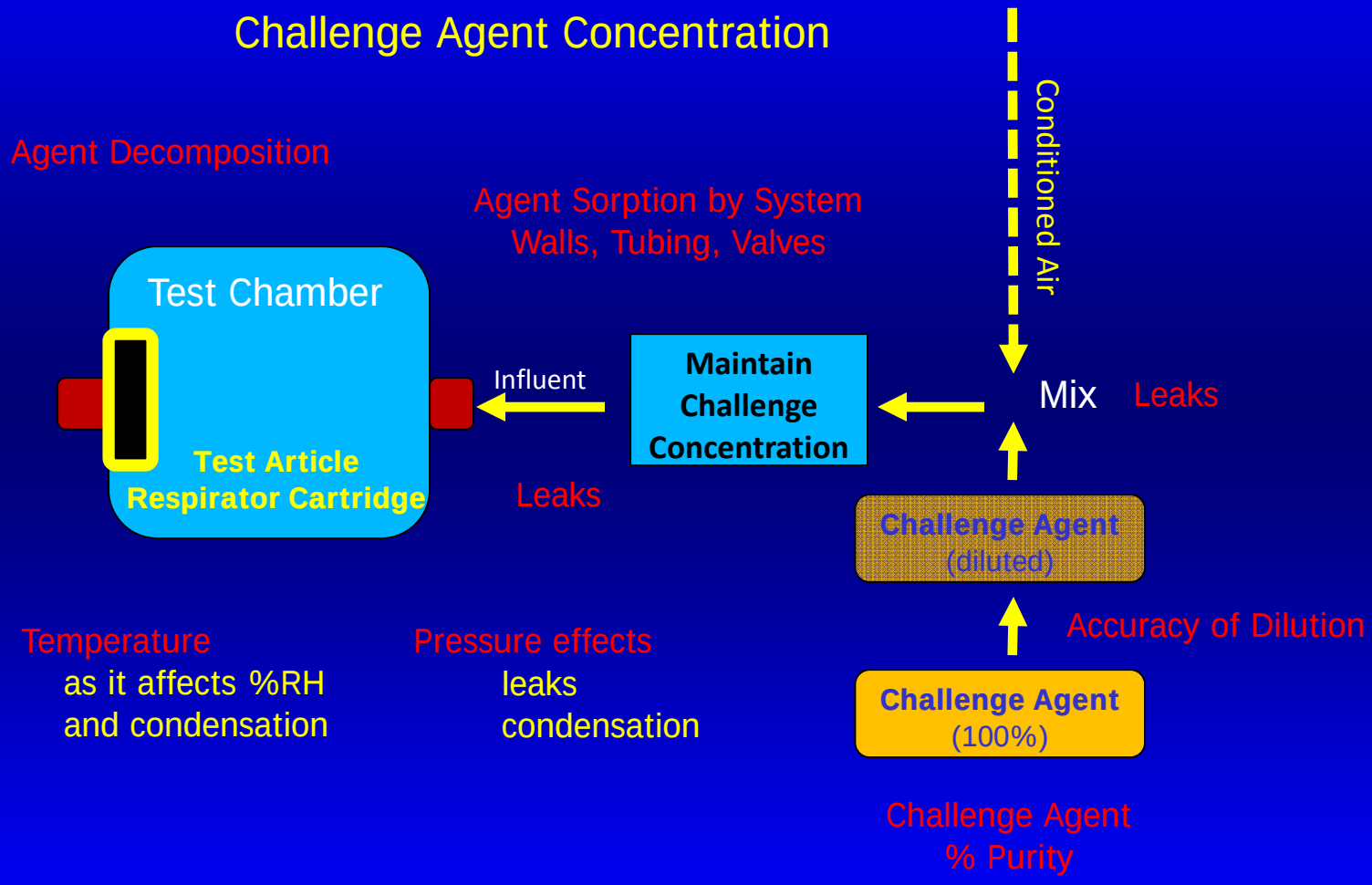


Mass Flow Control Accuracy

Temperature effects
as it affects %RH
and condensation

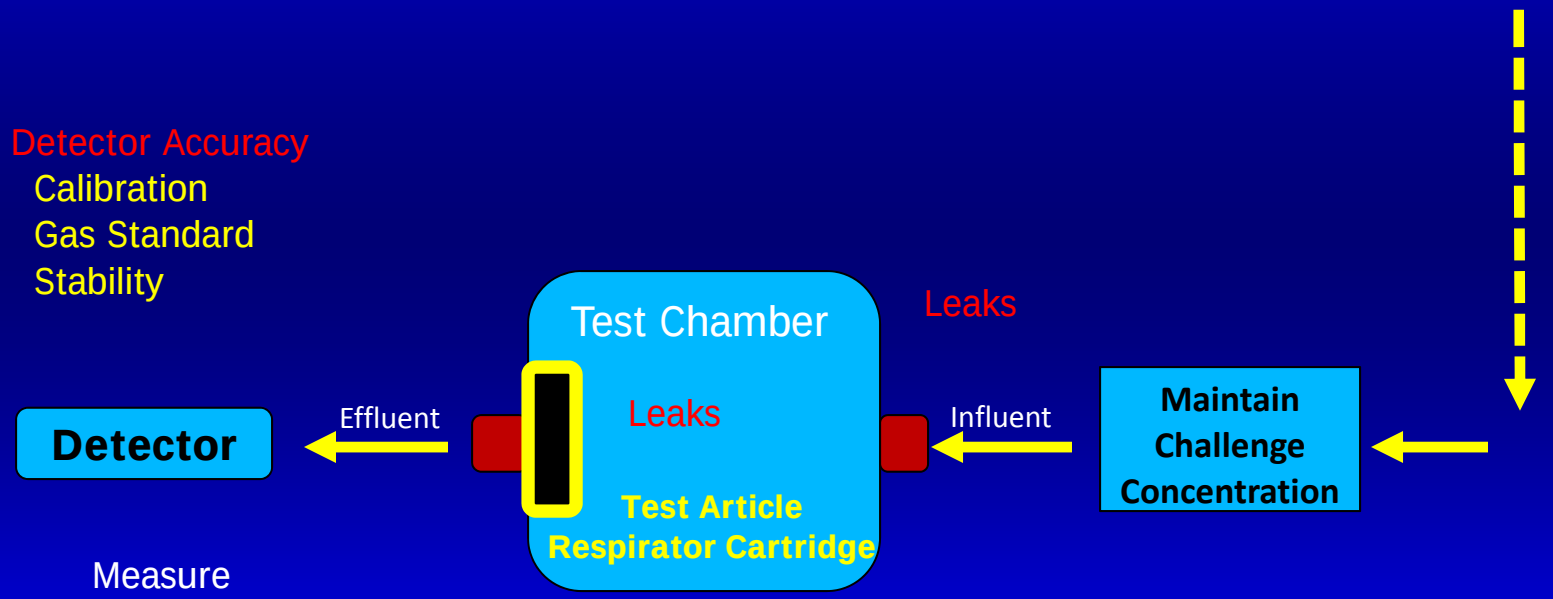
Pressure effects
leaks
condensation

Controlling a Chemical Challenge Test



Controlling a Chemical Challenge Test

Chamber and Detector Functions



Recommendations
(parameter controls)

Test Parameter

- Air Flow Rate
  - (L/min)
- Air Conditioning
  - (Temp & RH)
- Pre-Conditioning
  - (Temp, RH, and Flow Rate)

Control Criteria

- Measure Flow Rate
  - Use Mass Flow Meter
  - Calibrate vs Bubble Meter
  - Measure Downstream
    - Detect leaks
- Measure Temperature
  - Thermocouple or RTD
  - Calibrate vs Std
- Measure Humidity
  - Hygrometer
  - Chilled Mirror
  - Calibrate vs Salt Solns

More Recommendations
(parameter controls)

Test Parameter

- Challenge Agent Conc'n
  - Purchased Gas
  - Diluted gas
- Challenge Agent Conc'n
  - Loss by Decomposition
  - Loss by Sorption

Control Criteria

- Measure Potency
  - Vendor Certificate
  - Lab Analysis
- Measure *in situ*
  - Instrument
    - Continuous
  - Wet Chemical Analysis
    - Beginning & End
- Chamber & Tubing
  - Inert Components
  - Minimize Tube Length
  - Check for Agent Loss
  - Check for Leaks

Even More Recommendations
(parameter controls)

Test Parameter

- Break-Through Concentration
  - ppm Agent in Effluent as function of Time
- Time of Test
  - Time as function of ppm Agent in Effluent

Control Measure

- Use Selective Monitor
  - Spectroscopy
  - Electrochemistry
  - Ionization Detector
- Calibration Scheme
  - Gas Standard
  - Stability of Monitor
- Record vs Time

Summary of Recommendations

- Measure Flow Rate
  - Use Mass Flow Meter
  - Calibrate vs Bubble Meter
  - Measure Downstream
    - Detect leaks
- Measure Temperature
  - Thermocouple or RTD
  - Calibrate vs Std
- Measure Humidity
  - Hydrometer
  - Chilled Mirror or Calibrate vs Salt Solns

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  - Spectroscopy
  - Electrochemistry
  - Ionization Detector
- Calibration Scheme
  - Gas Standard
  - Stability of Monitor
- Record vs Time

Implementation of Recommendations

- Within a Single Organization
  - Communicate with Lab Mates
  - Write New Criteria into Procedures
- Across Many Organizations
  - Share Information
    - Communication is Much More Complicated

Collaborative Approaches

- Standardize Control Criteria
  - Communication Among Users
  - Technical Meetings
    - Establish Common Goals
    - Work Toward Consensus
- Round Robin Testing
  - Uniform Test Articles distributed to Labs
  - Compare Results from different Labs

Collaboration on Methods

What Actually Happens

- Standard Test Methods
  - Users evaluate methods more closely
  - Users discuss method details
  - Users propose criteria for Methods
- Problem
  - Different Organizations Do Things Differently
  - Difficult to Achieve a Consensus

Current Situation
(inter-laboratory agreement)

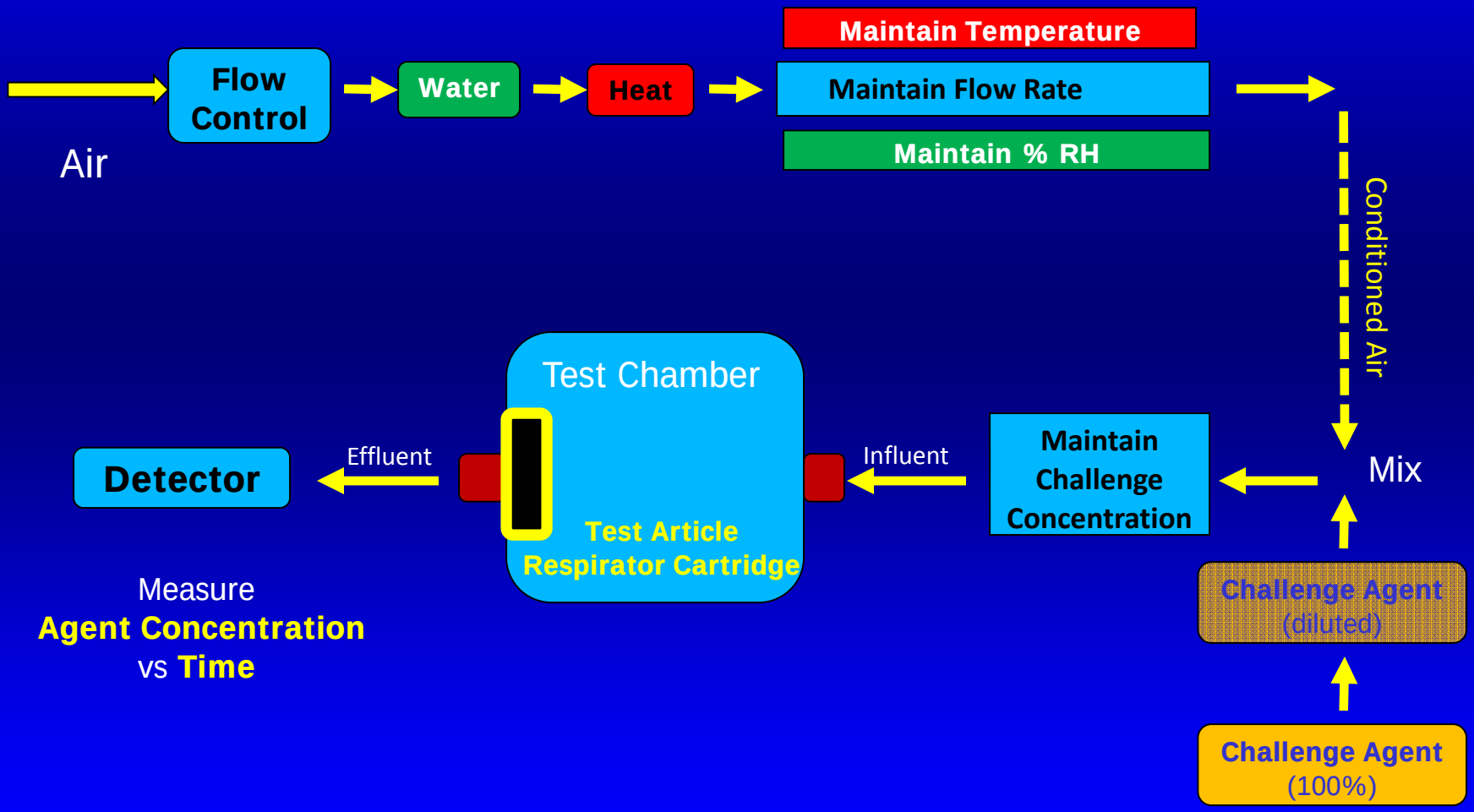
Good Agreement

- Sulfur Dioxide
- Ammonia
- Mineral Acids
- Organic Vapor
- Phosphine

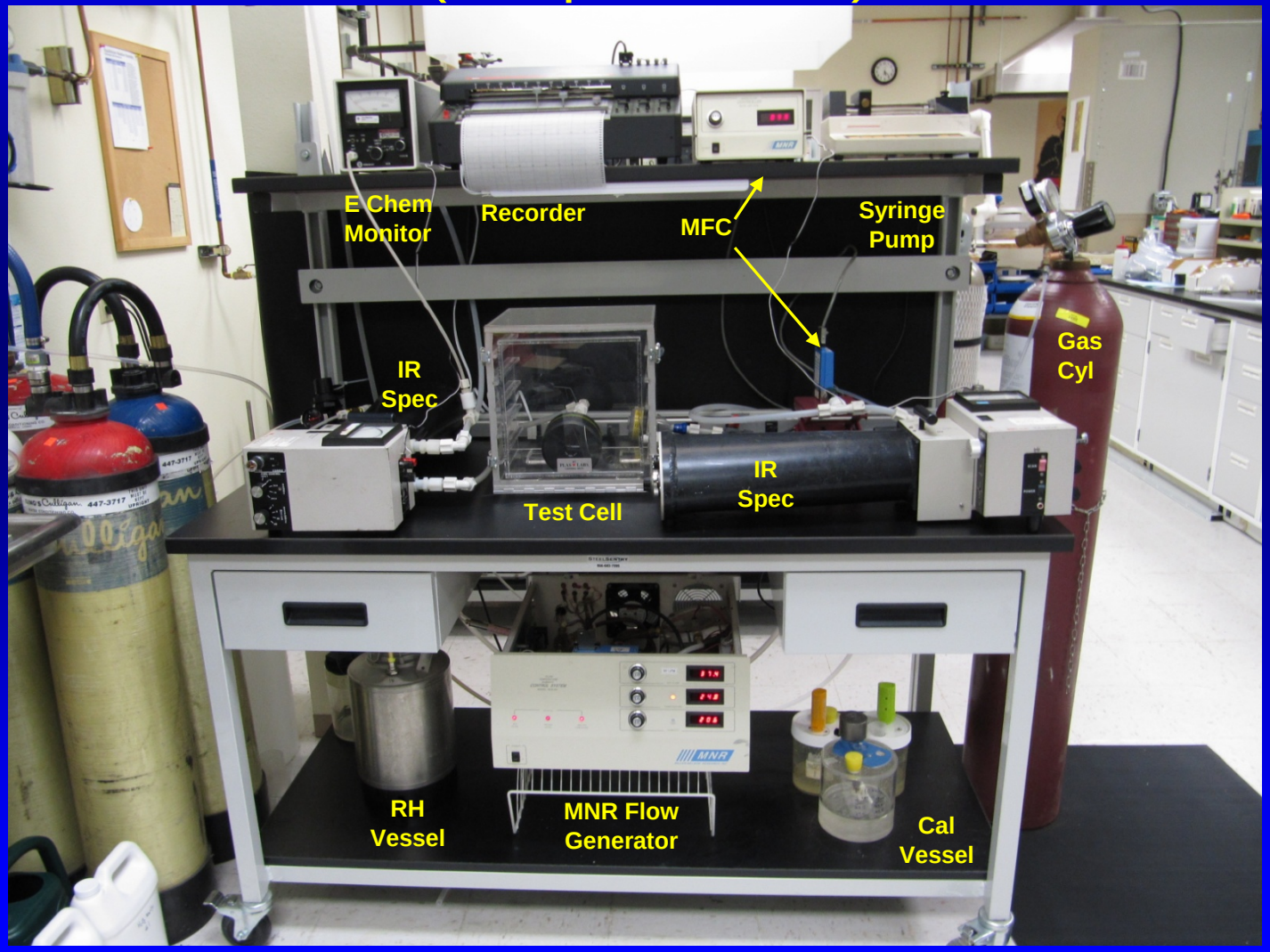
Inter-Lab Agreement
Not So Good

- Chlorine Dioxide
- Tear Gas
- Nitrogen Dioxide
- Cyanogen Chloride

Controlling a Chemical Challenge Test



Lab Bench
(except for Hood)



AT Respirator and Filter Chemical Challenge Test Lab

Work to be Done

Final Thought

Test Method Evaluation…

- Seeks to analyze methods rather than blame people for differences in test results.
- Control Criteria for Test Parameters leads to more uniform Test Results.

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