

# Accuracy Improvements in Service Life Testing Using FTIR Spectrometry

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# What is FTIR?

Fourier Transform Infrared Spectrometry.

*Fourier Transform:* Math used to convert Signal to a Spectrum

*Infrared:* Wavelengths beyond visible light  
(where almost everything absorbs)

*Spectrometry:* Using light absorption.

FTIR uses an “interferometer” -- a device which splits a beam of light, and the recombines it to produce an *Interferogram*. Software converts the Interferogram to an *Infrared Absorbance* spectrum.

A computer Model fits the absorption spectrum and generates  
*Gas Concentration Readings.*

# Why FTIR is Better than IR

- Most chemicals have a distinct IR absorption spectrum
  - With multiple IR Absorption Bands
- Absorption bands of different chemicals overlap
  - Water (H<sub>2</sub>O) has lots of bands that overlap
- FTIR makes IR absorption bands Narrower
  - Improved Resolution
- FTIR absorption bands are more intense
  - Improved Sensitivity
- Chemo-metrics Software Curve Fitting Algorithm
  - Simultaneous measurement of 5 or more chemicals in a mixture

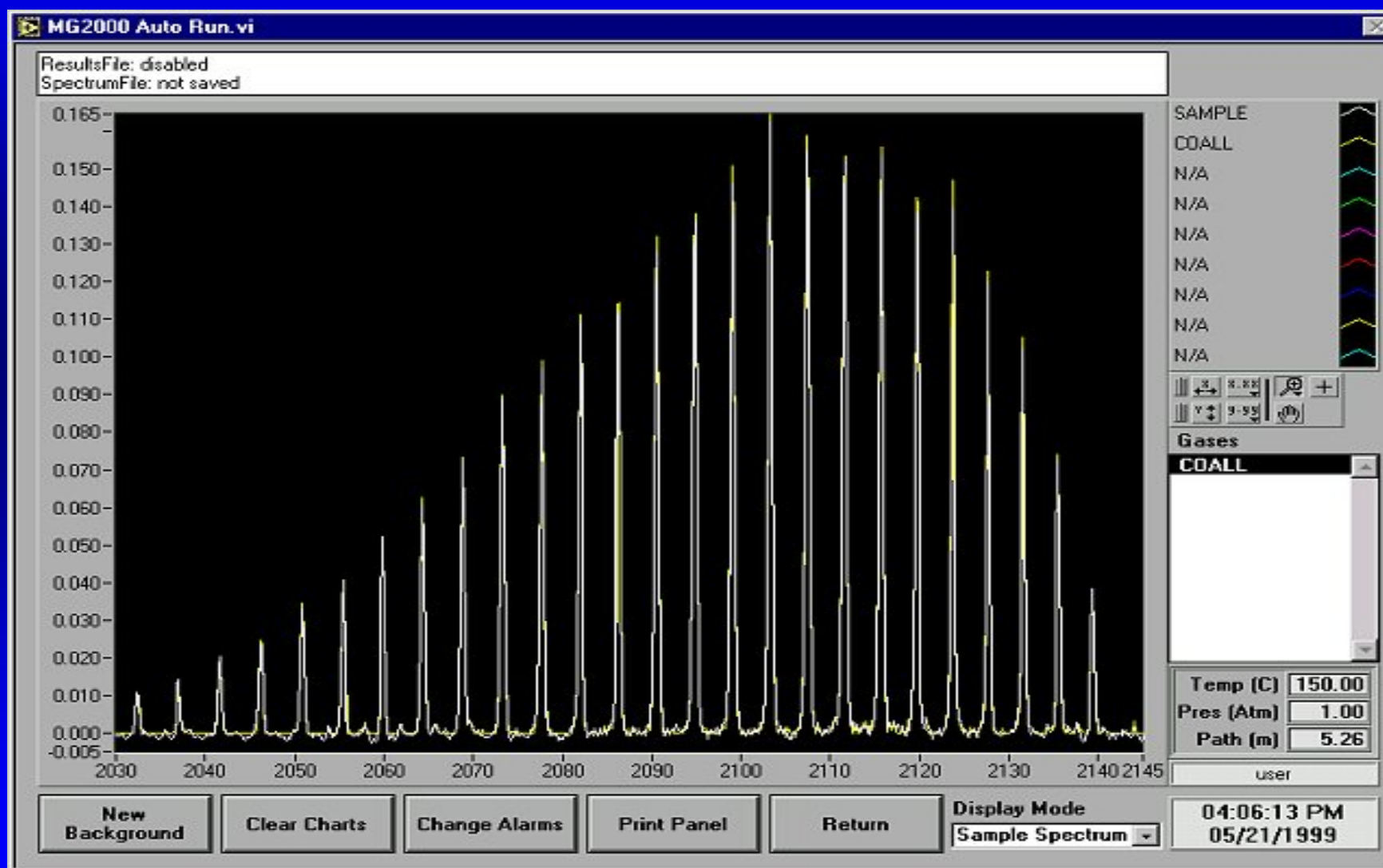
# Features & Benefits of Detectors

Measurement of ...	EC	TLD	IR	FTIR
Different Gases & Vapors	1	12+	Many	Many
In presence of similar species	C	C	B	A
In low ppm range	Yes	Yes	No	Yes
In high ppm to % Range	No	No	Yes	Yes
Continuously	Yes	Yes	Yes	Yes
Calibration Frequency	Daily	Rarely	Daily	Rarely

EC = Electrochemical Sensor

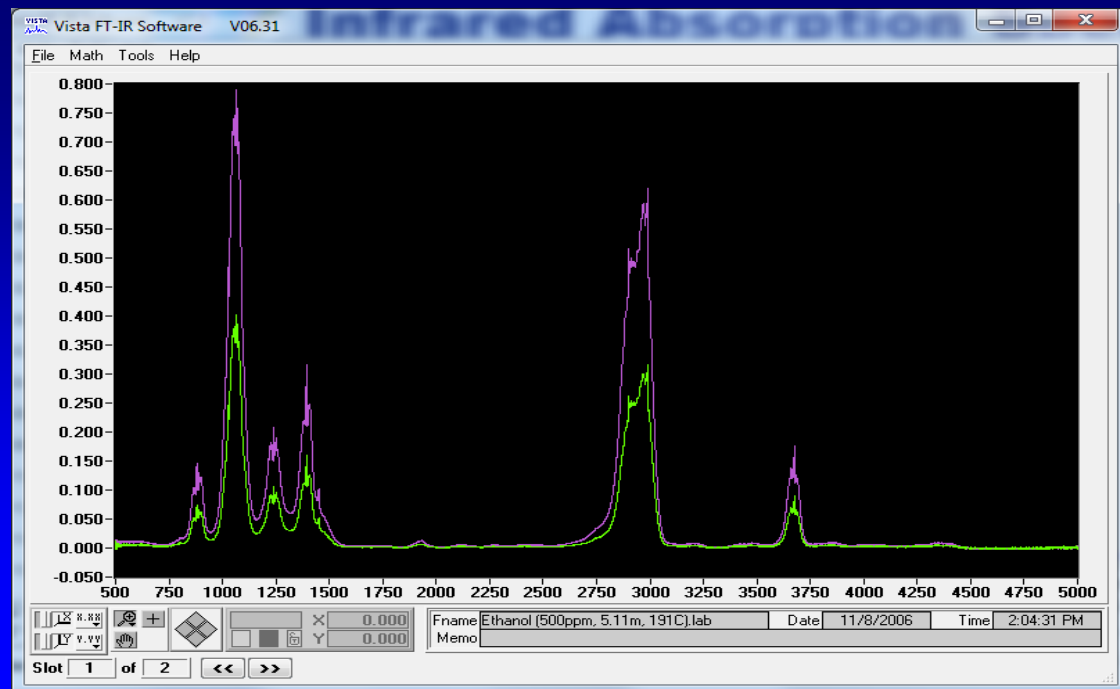
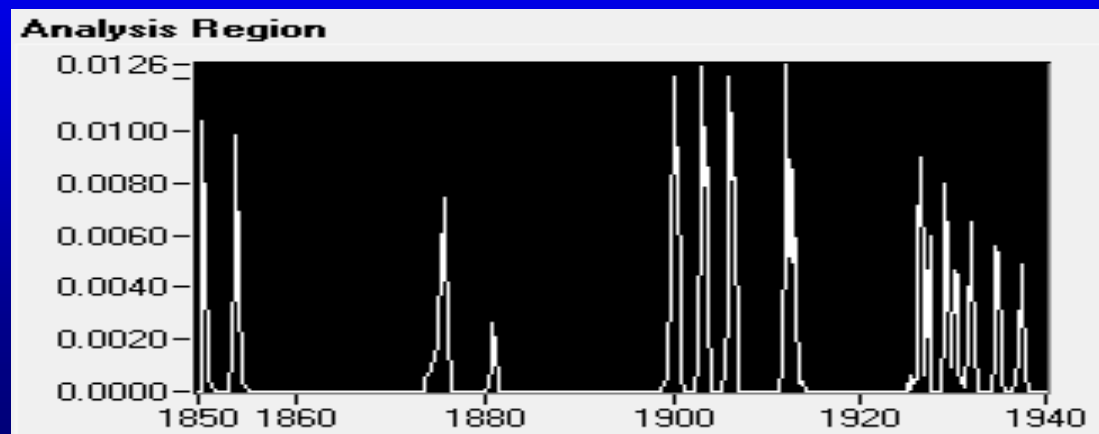
TLD = Colorimetric Tape Reader  
ChemCassette

# FTIR Spectrum of CO



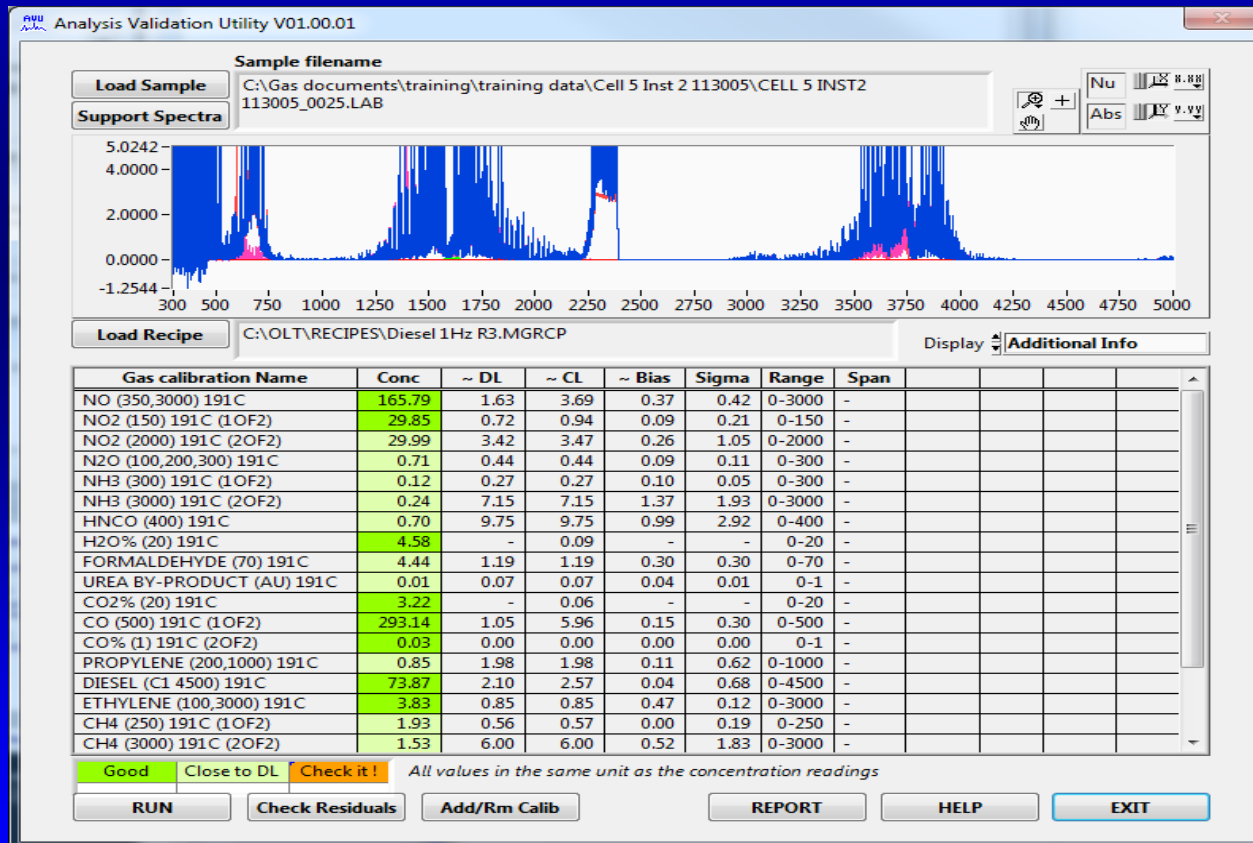
# FTIR Spectra

NO<sub>2</sub>



Ethanol

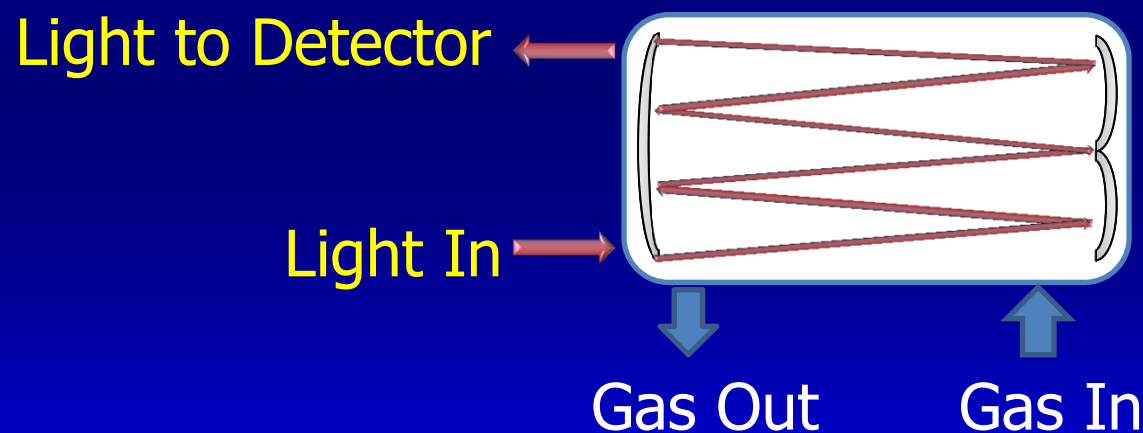
# Single Method Includes Multiple Gas Calibrations



# Gas Measuring Cell

Path Length = 5.11 Meter

Cell Volume = 0.2 Liter



Cell Material = Dursan-coated SS



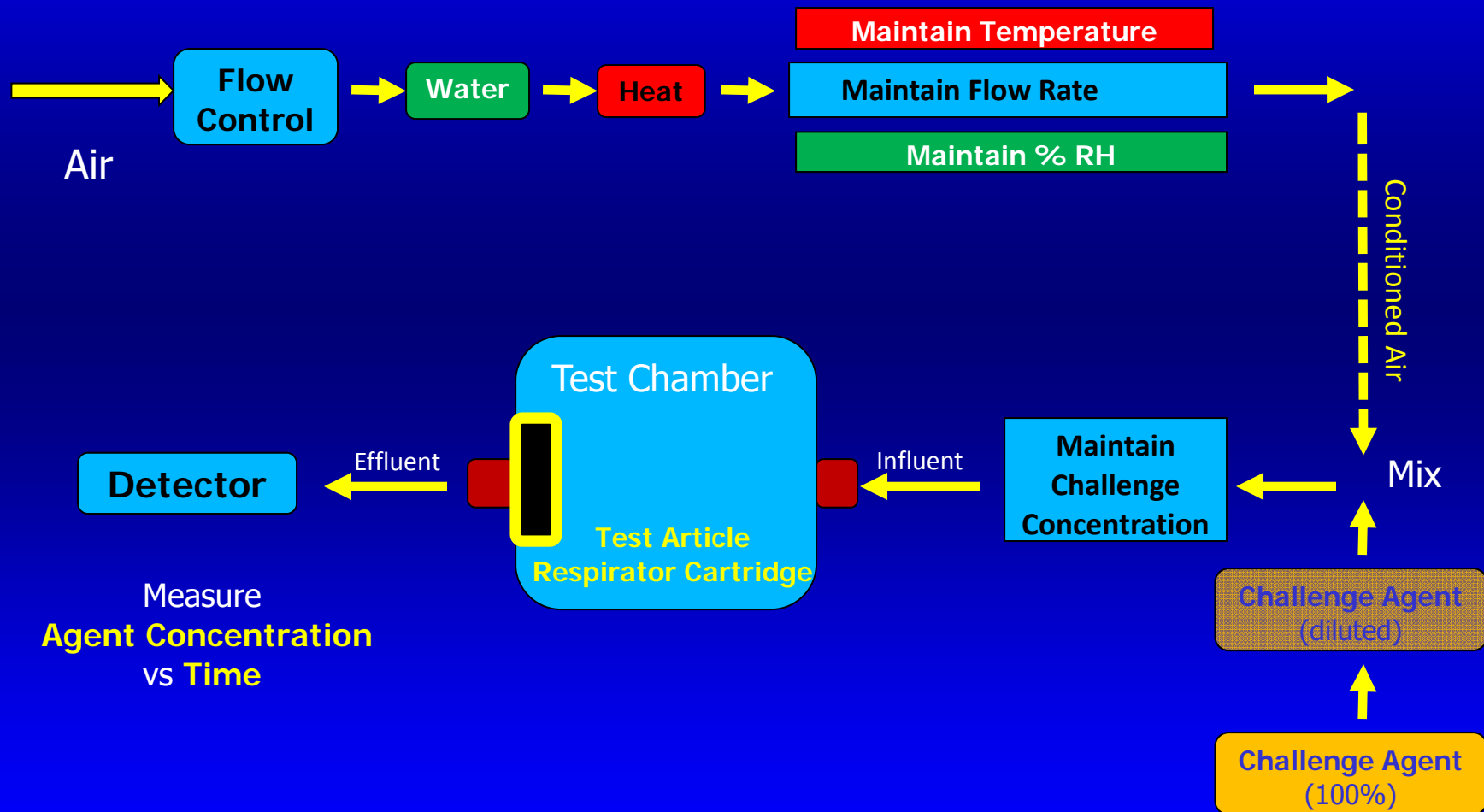
# FTIR Spectrometer (front)



# FTIR Spectrometer (rear)



# Anatomy of a Chemical Challenge Test



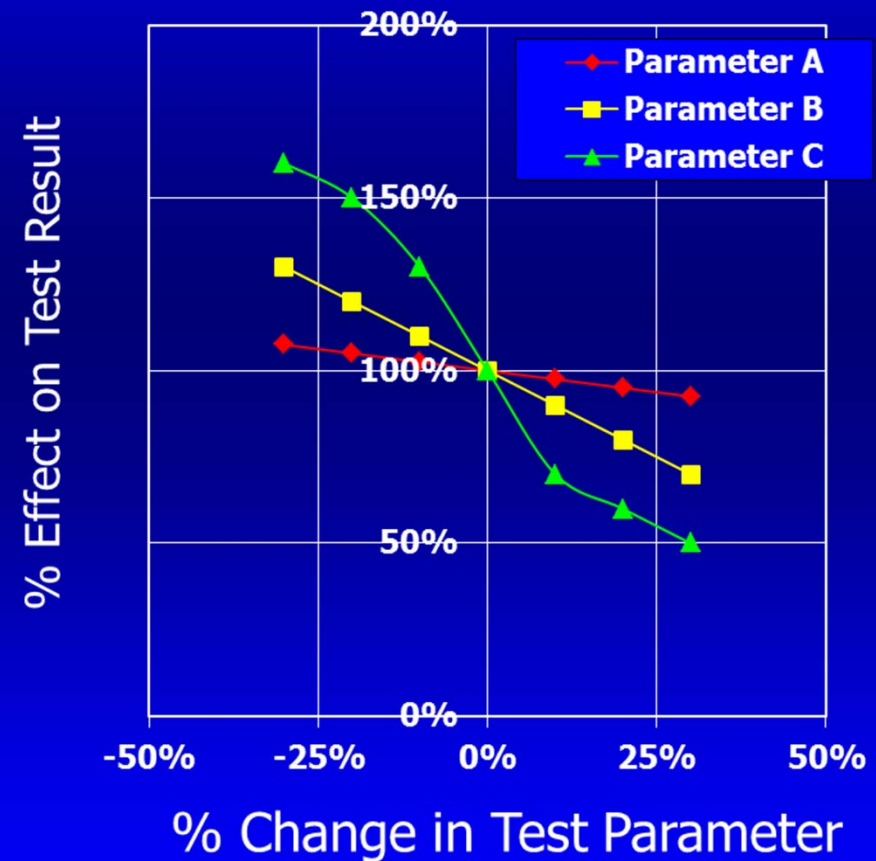
# Sensitivity of Test Result to Test Parameters

- The Test Result is more sensitive to variations in some Test Parameters than in others

A – Not Very Sensitive

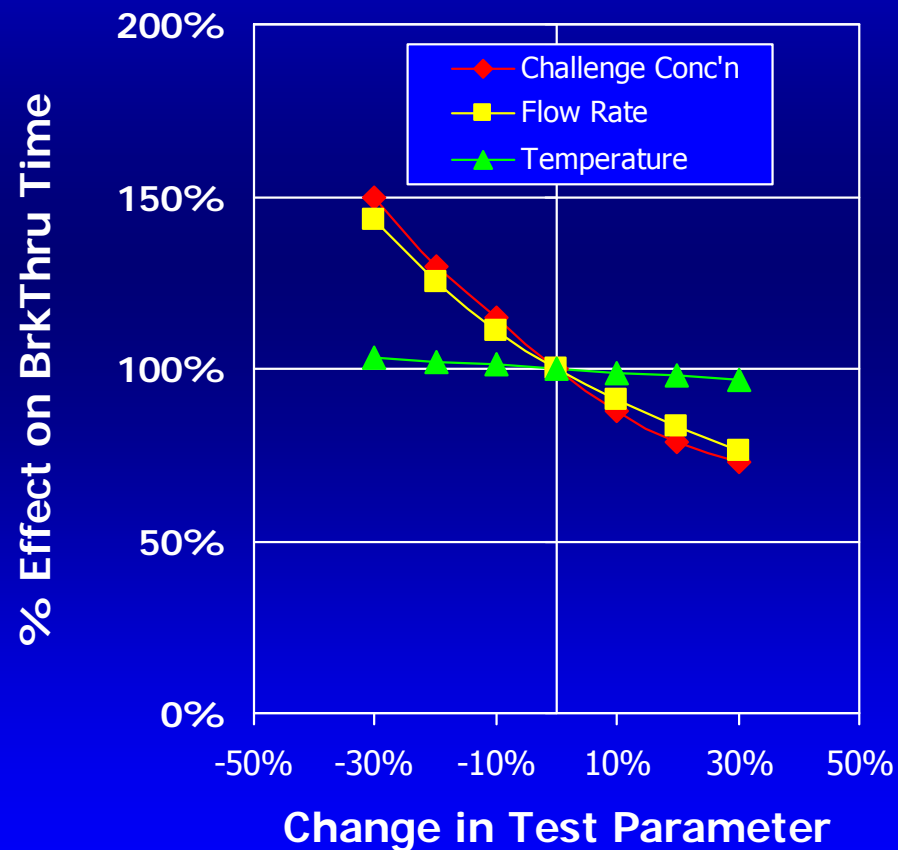
B – Proportional

C – Very Sensitive



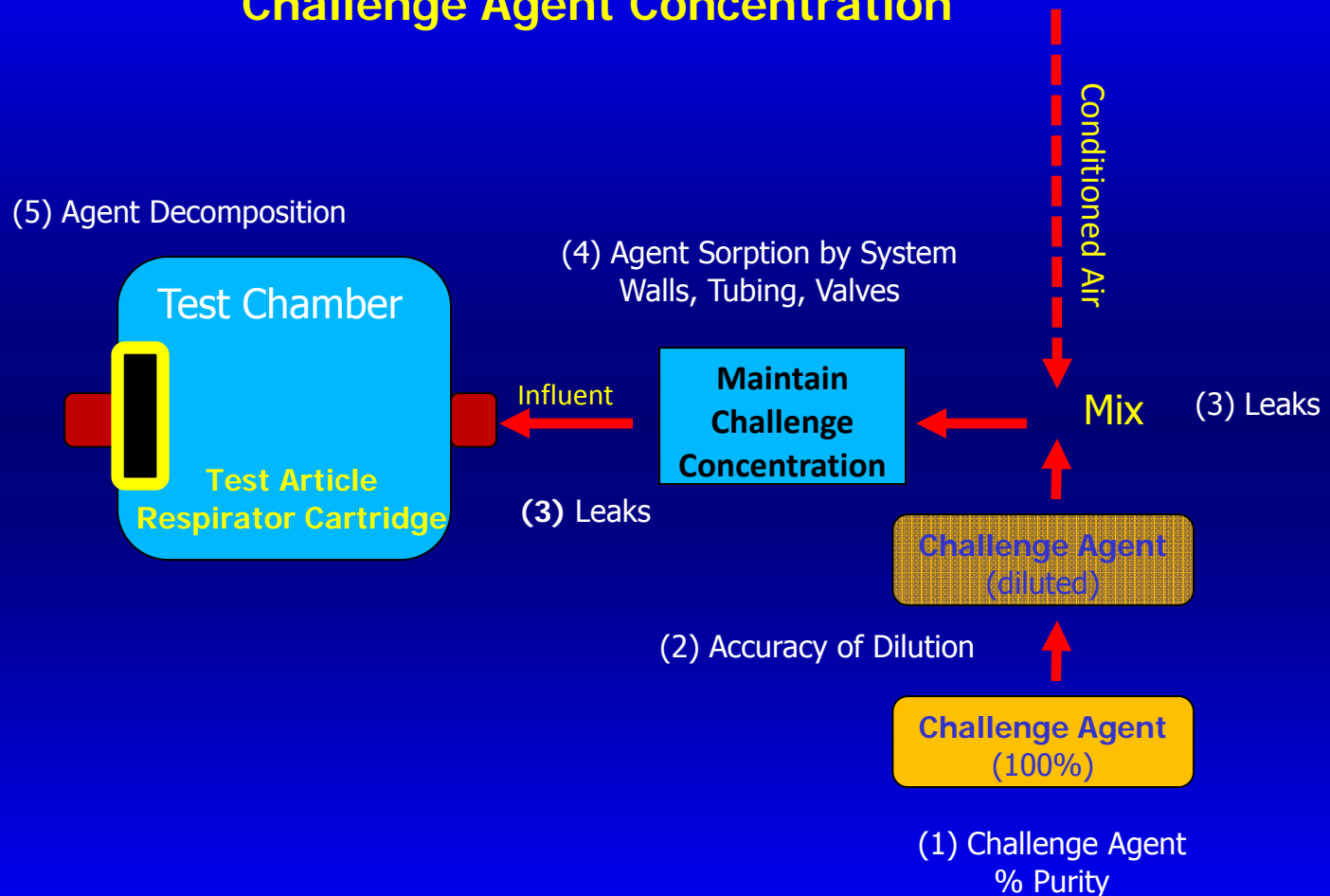
# Sensitivity of Test Result to Challenge Agent Concentration

- 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)



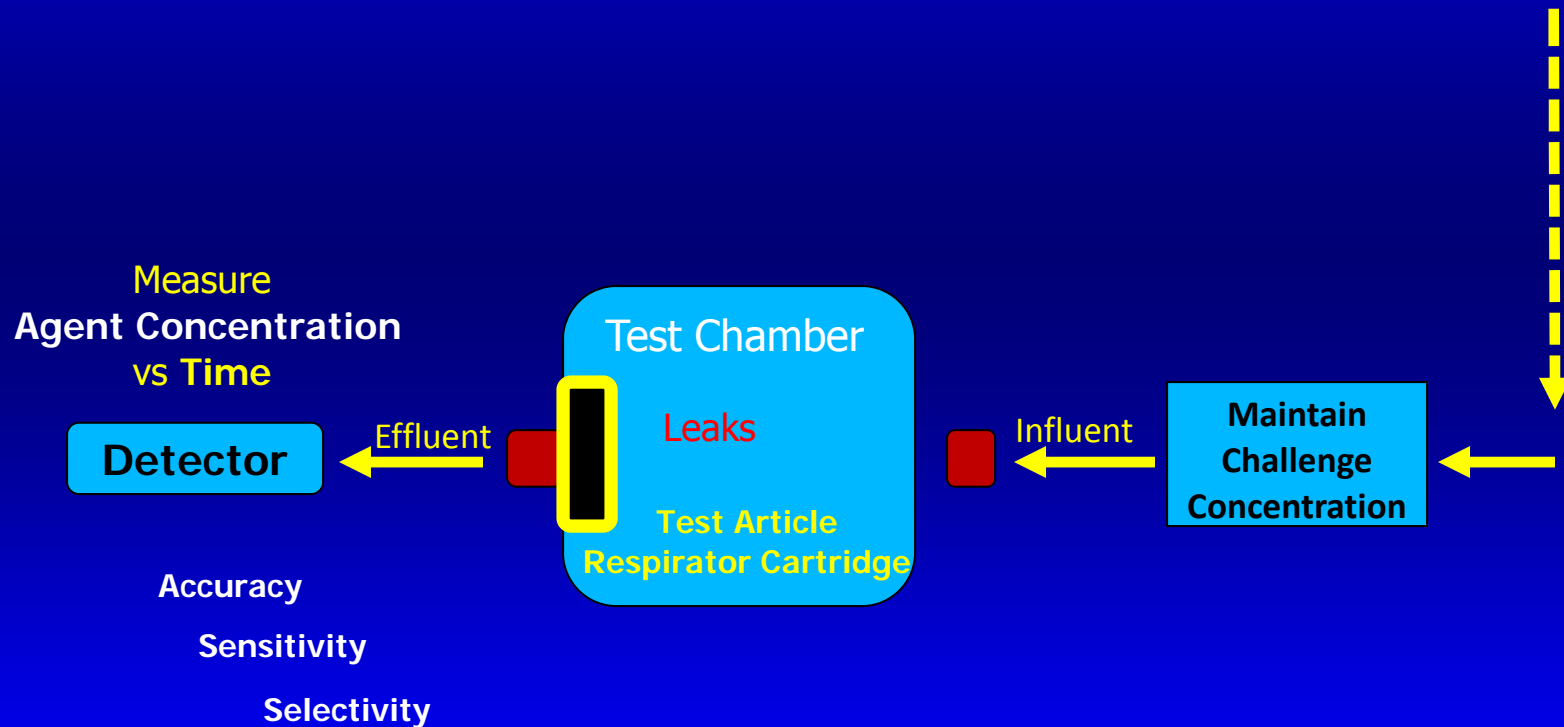
# Controlling a Chemical Challenge Test

## Challenge Agent Concentration



# Controlling a Chemical Challenge Test

## Chamber and Detector



# Control of Challenge Concentration

## Test Parameter

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

## Control Criteria

- Cylinder Gas Conc'n
  - Vendor Certificate
  - Wet Chem or FTIR Analysis
- Measure Gas *in situ*
  - Wet Chem Analysis
    - Prior to Test
  - FTIR
    - Continuous



# Best FTIR Applications

## Direct & Continuous Measurement of ...

- Cylinder Gas
  - As Received
  - After Storage
- Cylinder Gas Concentration Accuracy
  - Control Accuracy of Challenge Level
  - 10% error leads to 10% error in BT Time

# Best FTIR Applications

Direct & Continuous Measurement of ...

Influent Challenge Agent  
– Just Prior to Test

- Challenge Gas Accuracy
  - 10% error leads to 10% error in BT Time
  - Errors Can Multiply
    - Cyl Gas Accuracy Error + Sorption Loss Error

# Best FTIR Applications

## Direct & Continuous Measurement of ...

- **Effluent Agent**
  - Breakthrough
- **Agent Concentration Error**
  - 25-50% error leads to 10% BT Time error
- **Agent Detection Error**
  - Insensitive Detection of Agent
  - **Misidentification of Agent**
  - Fail to Detect Reaction Products

# Best FTIR Applications

## Direct & Continuous Measurement of ...

- **Diluted Cylinder Gas**
  - As Received & Prior to Use
- **Influent Challenge Agent**
  - Just Prior to Test
- **Effluent Agent** or Reaction Products
  - Breakthrough

# What Agents Can Be Analyzed by FTIR?

**Direct & Continuous Measurement**

**In the Presence of Similar Agents**

# FTIR Calibrations

(availability)

## Now AVAILABLE

- Formaldehyde
- Sulfur Dioxide
- CO & CO<sub>2</sub>
- Ammonia
- NO & NO<sub>2</sub>
- Chlorine Dioxide
- Carbon Tetrachloride
- Benzene
- HCl, HF, HBr
- Cyclohexane
- Phosphine
- Phosgene
- Ozone
- Acrolein ... and others

## To Be Developed

- $\alpha$ -chloroacetophenone (CN)
- o-chlorobenzylidene  
malononitrile (CS)
- Cyanogen Chloride

## Can't Do

- Cl<sub>2</sub>
- Br<sub>2</sub>
- No Dipole Moment

# Summary of FTIR Applications

- **Correct Challenge Agent Concentration**
  - Unstable Cylinder Gas
  - Gas Sorption during delivery to test
  - Gas Decomposition during delivery
- **Improve Breakthrough Detection**
  - Agent Difficult to Detect
  - Detect Reaction Products
    - Detect Agents in presence of Reaction Products

# Summary of Value

(delivered by FTIR)

- Improve Overall System Accuracy
  - Reduce Error
- Improve Inter-Laboratory Test Agreement
  - Improve overall Quality Assurance



# AT Respirator and Filter Chemical Challenge Test Lab



**Finis**