# Update on Manual Method for Sampling and Analysis of Condensable Particulate

Ray Merrill – ERG

Ron Myers – EPA

# Overview

- Methods background
- Method 202 performance (bias)
- Methods Comparison
  - Hardware & preparation
  - Sampler operation
  - Recovery requirements
  - Analytical finish
- Dry Impinger Method performance
  - Bias
  - precision
- Schedule

# Background
# What is Condensable PM ?

- **Vapors and gases at stack temperature that form liquid or solid aerosols upon cooling**
- **All condensable PM <1µM**

# Background
# How was Condensable PM Measured?

- **Existing Method 202**
  - **Promulgated in 1991**
  - **Over 6 options in method**
  - **Artifact issue**

# Background
## What's Changing with Condensable PM Measurement?

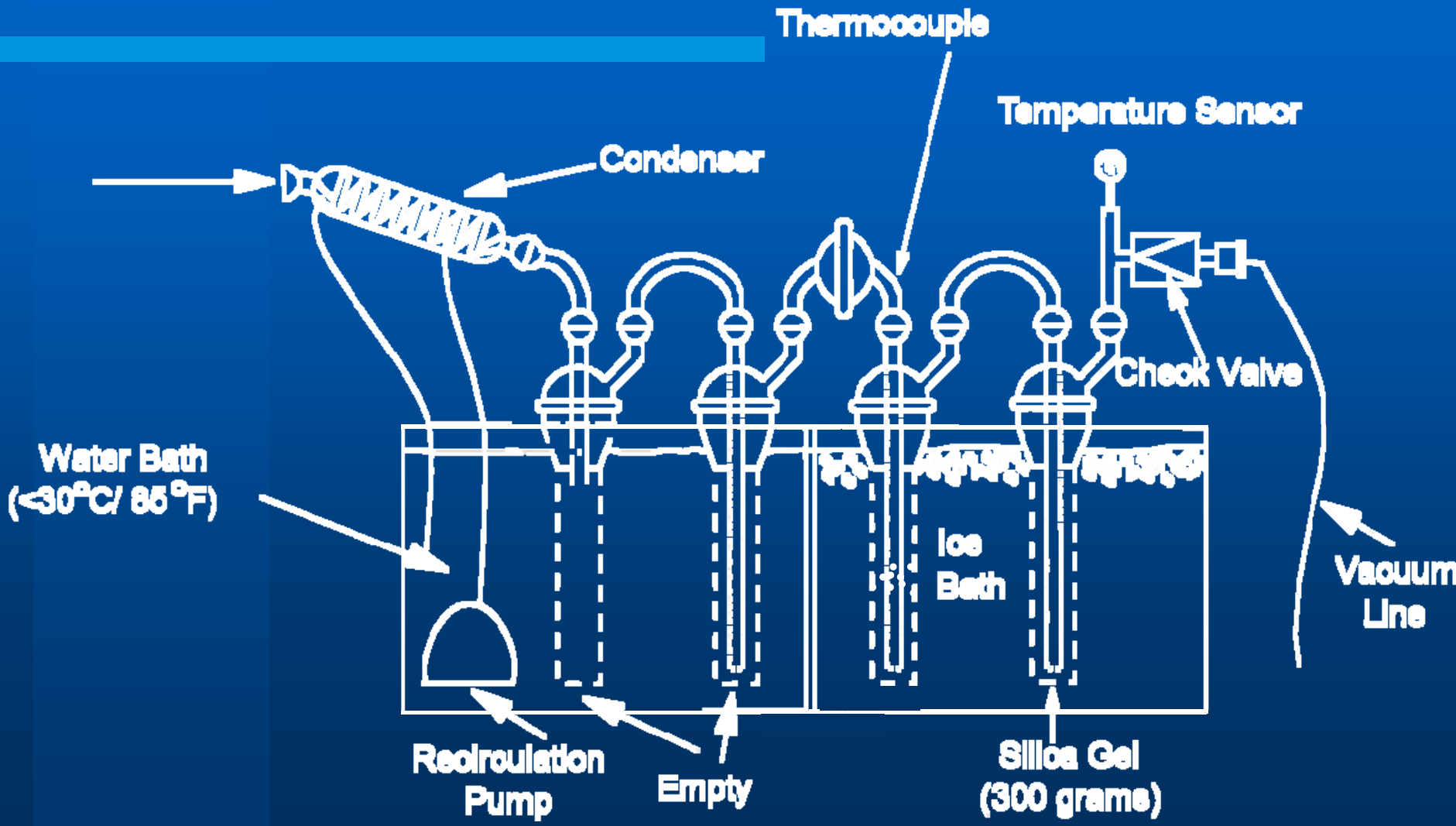
- **Dry Impinger Method (OTM-28)**
  - **Originally proposed by John Richards (Method 202 variant)**
  - **Single standard procedure (no options)**
  - **Minimizes $SO_2$ artifact**
  - **Low level blank management**
  - **Will replace EPA Method 202**

# Method 202 Artifact (bias)

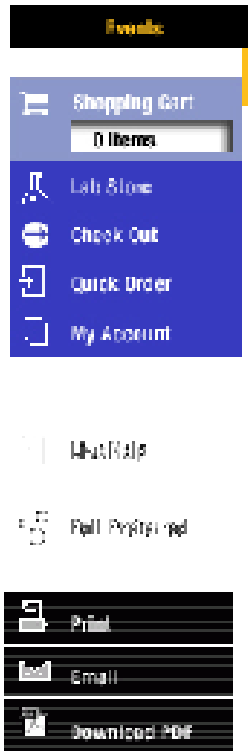
| $SO_2$ ppm | Test duration | $H_2O$ volume | Artifact Mass (mg) | |
|---|---|---|---|---|
| | | | No Purge | Purge |
| 300 | 1 Hr | 400 ml | 180 ± 6 | 10 ± 0.5 |
| 300 | 3 Hr | 800 ml | 400 ± 25 | 20 ± 5 |
| 50 | 6 Hr | 1400 ml | 200 ± 10 | 20 ± ?? |

# OTM-28 Sampling Train

## Dry Impinger Glassware



# OTM-28 CPM Filter Media




## PTFE Membrane Disc Filters

Order Online Now

Description | Applications | Specifications | Ordering Information

| Description | Zefluor™ Membrane | Teflo Membrane |
|---|---|---|
| Filter Media/Support | PTFE with PTFE support | PTFE with PMP (polymethylpentene) support ring |
| Typical Thickness | 0.5 µm: 178 µm (7 mils)<br>1 µm: 165 µm (6.5 mils)<br>2 and 3 µm: 152 µm (6 mils) | 1 µm: 76 µm (3 mils)<br>2 µm: 46 µm (1.8 mils)<br>3 µm: 30.4 µm (1.2 mils) |
| Typical Air Flow Rate (L/min/cm² at 0.7 bar (70 kPa, 10 psi)) | 0.5 µm: 1<br>1 µm: 14.6<br>2 µm: 25.3<br>3 µm: 53 | 1 µm: 17<br>2 µm: 53<br>3 µm: 90 |
| Minimum Bubble Point - IPA bar (psi) | Not Applicable | Not Applicable |
| Water Breakthrough bar (psi) | Not Applicable | Not Applicable |
| Typical Aerosol Retention* | 0.5, 1, and 2 µm: 99.99%<br>3 µm: 99.98% | 1 and 2 µm: 99.99%<br>3 µm: 99.79% |

# Method Comparison:
## Glassware Preparation

| Existing Method 202 | Dry Impinger Method |
|---|---|
| Soap & Tap Water Wash | Soap & Tap Water Wash |
| Tap Water Rinses | Tap Water Rinses |
| | Ultrafilter water rinse |
| Acetone Rinse | Acetone Rinse |
| MeCl Rinse | MeCl Rinse |
| | Heat @ 300 °C – 6 Hrs |

# Method Comparison:
## Glassware Set Up

Option #1

| Existing Method 202 | Dry Impinger Method |
|---|---|
| | Method 23 Condenser |
| 2 G/S Impingers<br>w/ tipped stems<br>100ml DI water | G/S Impinger<br>w/ short stem, no water |
| 1 G/S Impinger<br>w/ 100 ml DI water | G/S Impinger<br>w/ stem w/o water |
| Optional filter | Filter |
| | G/S Impinger w/ 100 ml water |
| G/S Impinger<br>w/ Silica Gel | G/S Impinger<br>w/ Silica Gel |

# Method Comparison:
## Sampling Temperature

| Existing Method 202 | Dry Impinger Method |
|---|---|
| Maintain ALL four impingers in ice water bath | First two impingers in ≈ 85° F water bath |
| | Filter Gas @ ≤ 85° F |
| | Last two impingers in ice water bath |

# Method Comparison:
## Post Test Recovery

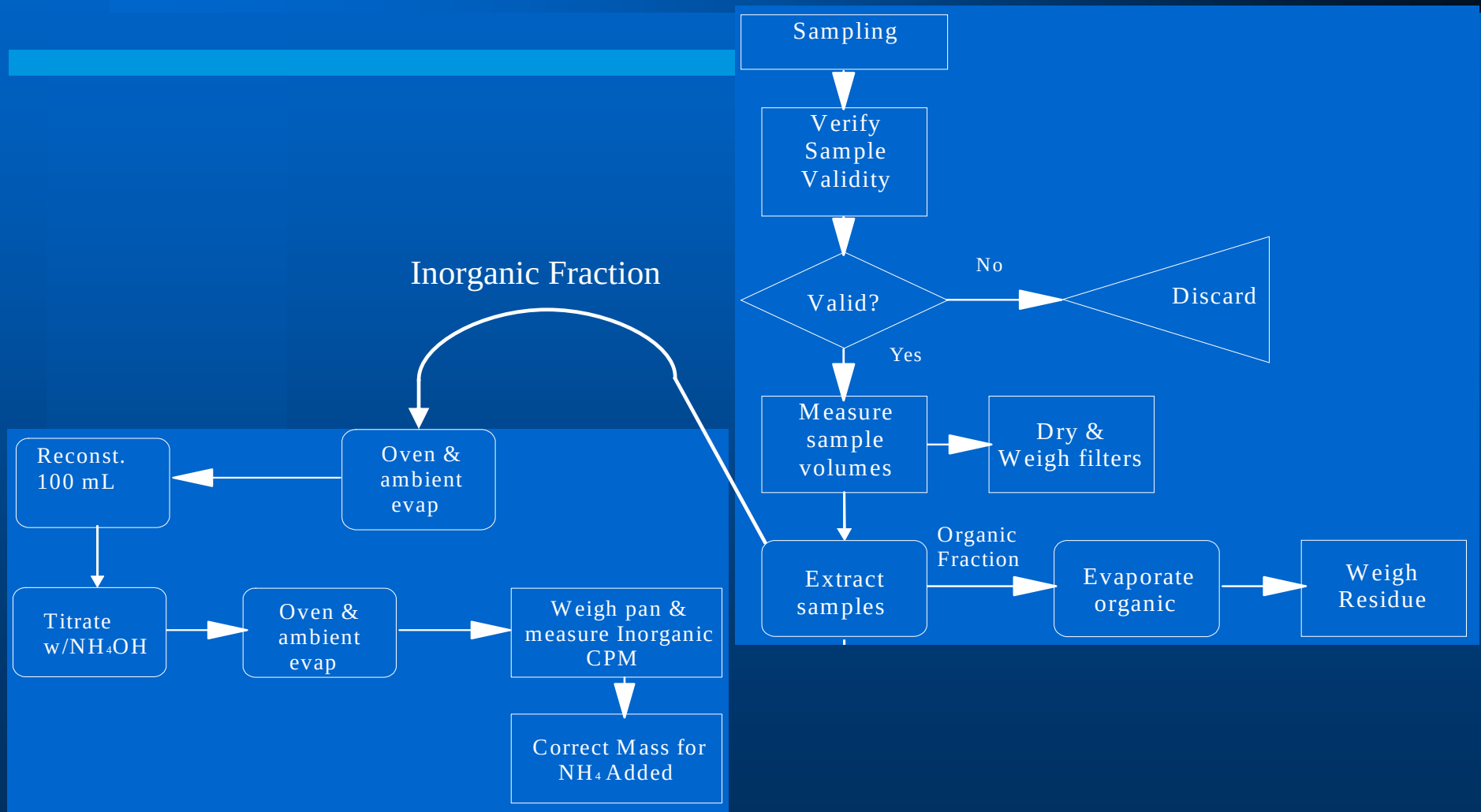
| Existing Method 202 | Dry Impinger Method |
| --- | --- |
| Nitrogen Purge<br>Air Purge<br>No Purge } Optional | Mandatory Purge with filtered UHP Nitrogen |

**Option #2**

# Recovery Requirements:
## Reagent Quality Requirements

| Existing Method 202 | Dry Impinger Method |
|---|---|
| DI Water – ASTM Type II | DI Water – Ultra filtered ASTM Type II |
| Acetone – not used | Acetone - < 1 ppm residue |
| MeCl – ACS Grade <0.001% residue | MeCl – ACS Grade < 1 ppm residue |
| $NH_4OH$ – 14.8 Molar | $NH_4OH$ – NIST traceable 0.1 Normal |

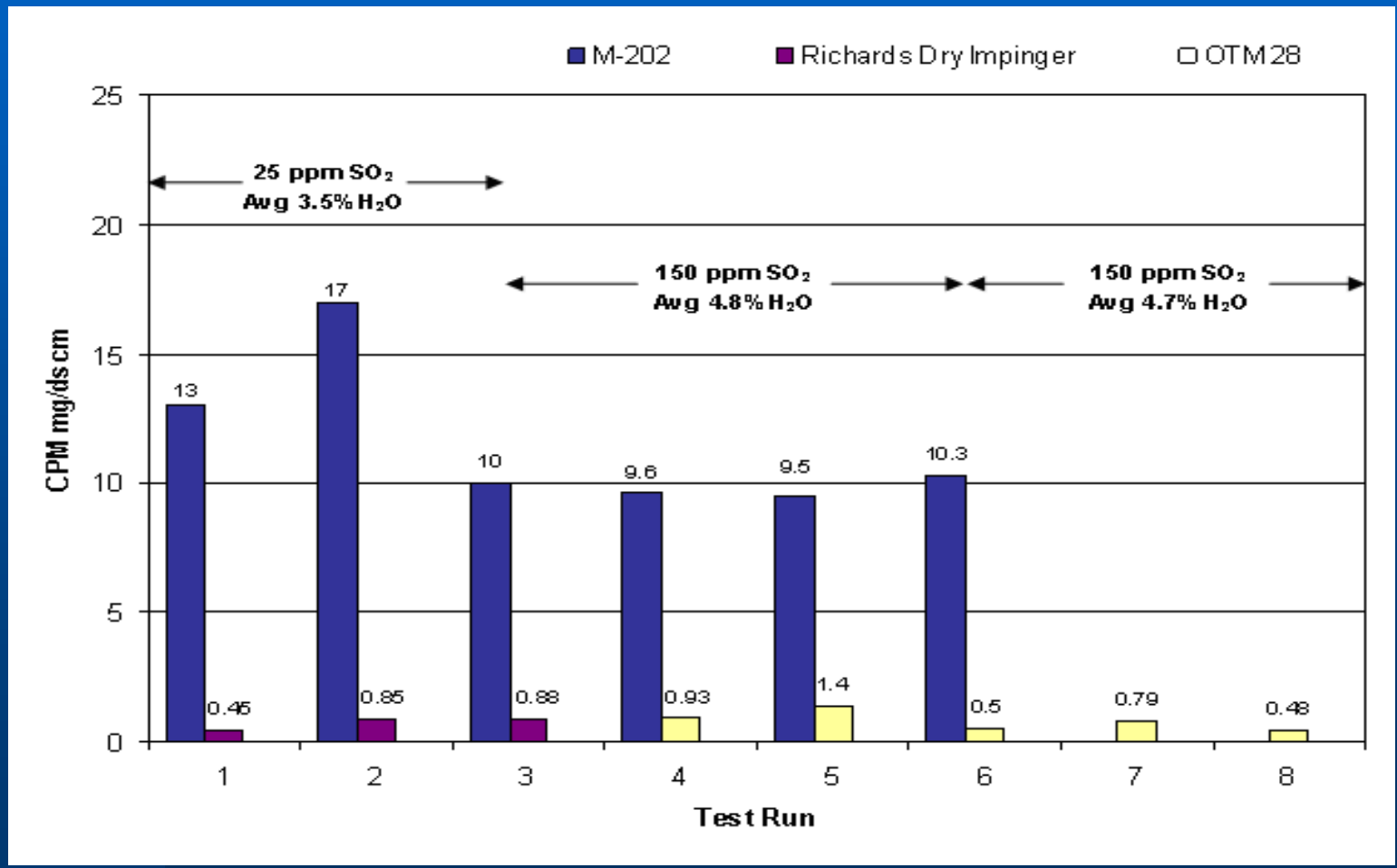
# Dry Impinger Method



# Method OTM-28 Comparisons

- **OTM – 28 Compared to**
  - **Method 202**
  - **Controlled Condensate**
  - **Low Temperature Filter Train**

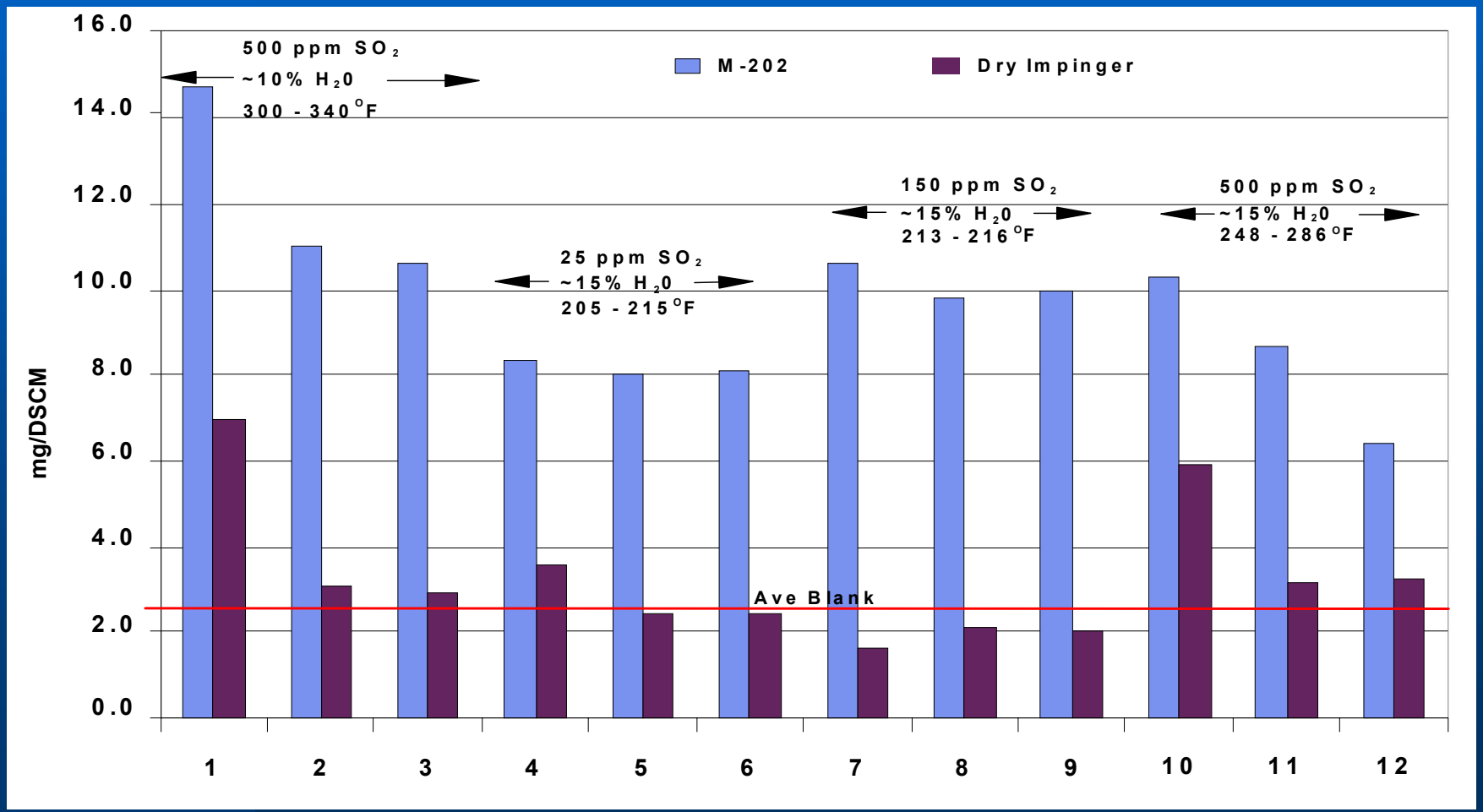
# OTM – 28 to Method 202 Comparison
## (EPA Laboratory Study)



# OTM - 28 vs. M202

## (Gravimetric Comparison)

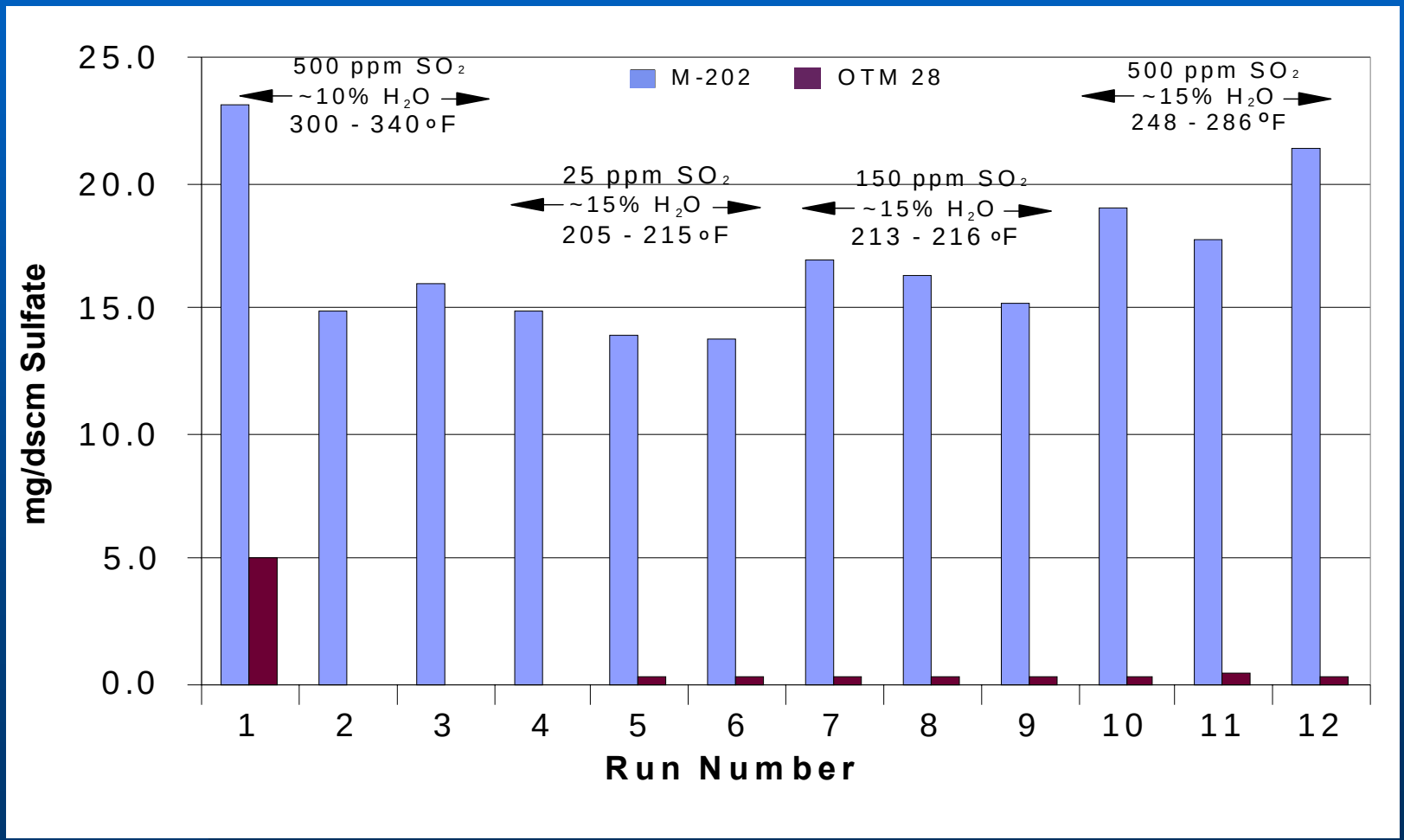
Source: Electric Power Research Institute, 2008



# OTM - 28 vs. M202

## (Titration for Acid Artifact)

Source: Electric Power Research Institute, 2008



# OTM – 28 Method Performance

MDL =1.60

STD = 0.53

# OTM – 28 Method Performance

## (EPA Laboratory Replicate Study)

| Run | Organic (mg/dscm) | Inorganic (mg/dscm) | Filter (mg/dscm) | Total (mg/dscm) |
|---|---|---|---|---|
| 1 | 0.12 | 2.36 | -0.36 | 2.34 |
| 2 | 0.16 | 2.99 | -0.06 | 3.03 |
| 3 | 0.09 | 1.44 | 0.00 | 1.46 |
| 4 | 0.31 | 1.98 | 0.00 | 2.22 |
| 5 | 0.17 | 1.65 | 0.07 | 1.77 |
| 6 | 0.39 | 2.58 | -0.20 | 2.52 |
| 7 | 0.08 | 1.24 | 0.31 | 1.56 |
| 8 | 0.02 | 1.94 | 0.18 | 2.06 |
| Blank | -0.02 mg | 0.21 mg | 0.00 mg | 0.68 mg |
| Average | 0.17 | 2.02 | -0.01 | 2.18 |
| Std Dev | 0.12 | 0.59 | 0.21 | 0.53 |
| MDL | 0.37 | 1.78 | 0.63 | 1.60 |

# OTM – 28 Method Performance

## Ammonia and Sulfur Dioxide

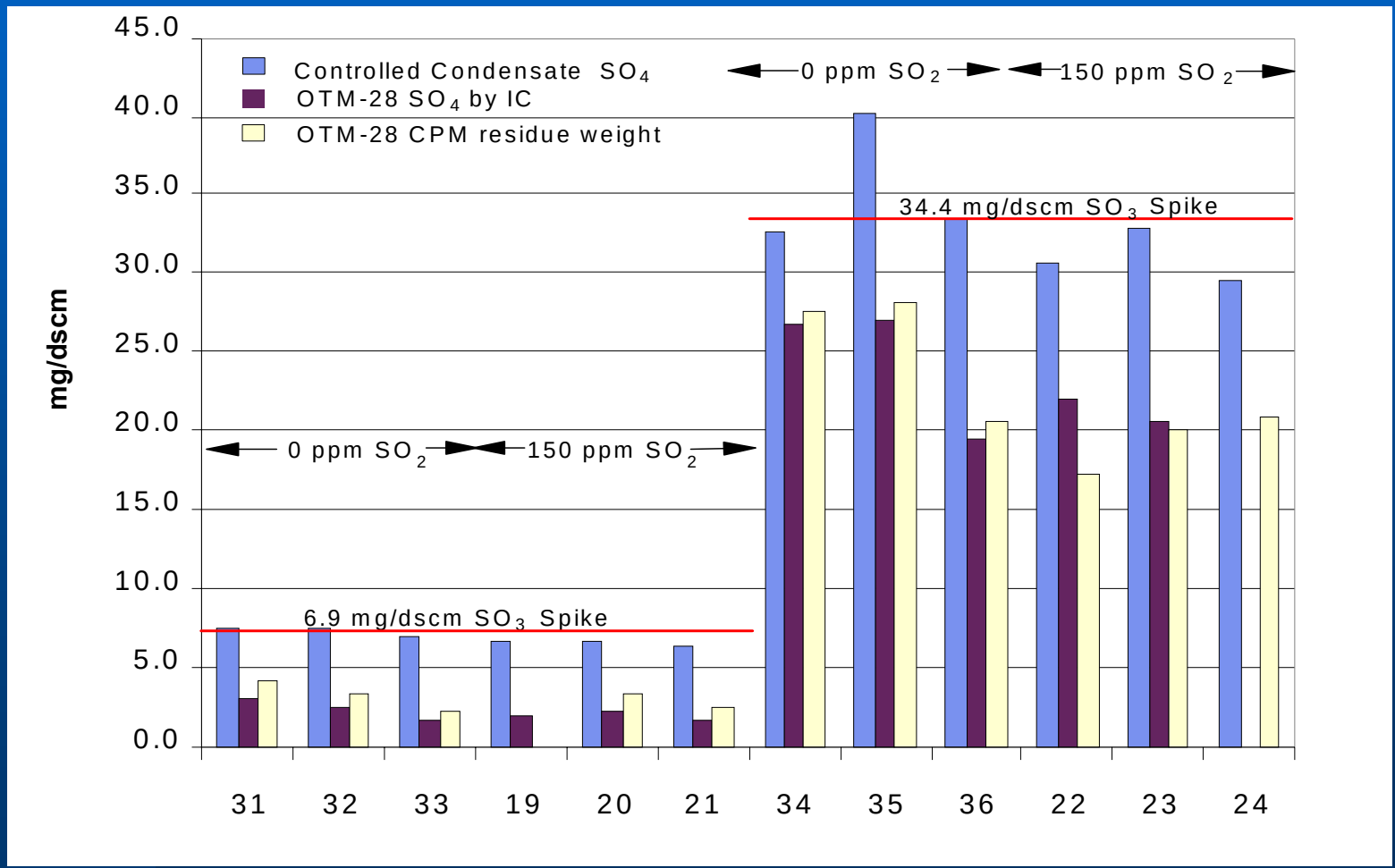
(EPA Laboratory Replicate Study)

| Phase I Test Run | Method 5 filter (160 °C) (µg) | Low-temp filter (71 °C) (µg) | Aqueous (µg) | Organic (µg) | Ambient filter (30 °C) (mg) | Total (µg) |
|---|---|---|---|---|---|---|
| | | | **$NH_3$** | | | |
| 16 | ND | ND | ND | NA | ND** | ND |
| 17 | ND | ND | ND | NA | ND** | ND |
| 18 | ND | ND | ND | NA | ND** | ND |
| Train Blank 1 | ND | NA | ND | NA | NR | ND |
| Train Blank 2 | ND | NA | ND | NA | NR | ND |
| Train Blank 3 | ND | NA | ND | NA | NR | ND |
| Reagent Blank | NA | NA | ND | NA | NR | ND |
| | | | **$SO_4$** | | | |
| 16 | 180 | 160 | ND | NA | ND* | 340 |
| 17 | 146 | 201 | ND | NA | ND* | 347 |
| 18 | 181 | 156 | ND | NA | ND* | 337 |
| Train Blank 1 | 147 | 142 | ND | NA | NR | 289 |
| Train Blank 2 | 147 | 102 | ND | NA | NR | 249 |
| Train Blank 3 | 150 | NA | ND | NA | NR | 150* |
| Reagent Blank | NA | NA | ND | NA | NR | ND |

ND = Not detectable above 27.6 total micrograms
ND** = Not detected in gravimetric analysis of filter
NR = Not run

# OTM - 28 vs. Controlled Condensate $SO_4$ Recovery

Source: Electric Power Research Institute, 2008



# Dry Impinger Method Recap

- No Options
- Minor glassware changes
- No water at start of test
- Short stem impinger insert replaced with tipped insert for purging
- Degassed Type III water added if needed
- Both organic & inorganic evaporations at room temperature
- Significant reduction of sulfate artifact

# Schedule Update

- FR Proposal – April 2009
- Comment Period Close – June 2009
- FR Promulgation – December 2009

# QUESTIONS