Alliance Source Testing, LLC

Stack Testing 101
Alliance Source Testing is focused solely on providing accurate and consistent source testing solutions that allow our partners to make informed decisions and succeed responsibly.

We provide Stack Testing Services to partners with permit, MACT, or NSPS testing requirements; CEMS Evaluations in support of Part 60, 53 and 75 programs; Onsite Analytical Services in support of testing projects including FTIR, GC/FID, GC/FPD, IC, gravimetric and multiple titrations; and Fugitive Emissions Evaluations in support of LDAR and Greenhouse Gas monitoring programs.

Specializing in compliance, engineering and performance specification testing... Stack Testing is Our Business!

**Testing You Can Rely On... Partners You Can Trust**
Stack Testing Is Our Business

Alliance provides emissions testing services for multiple purposes including, but not limited to, the following:

- Permit Compliance Demonstrations
- P60 & P75 CEMS Evaluations (RATA, CGA, Linearity)
- PM CEMS Evaluations (RCA, RRA, ACA)
- Comprehensive Performance Testing
- Boiler/Turbine/Engine Optimization
- Process Evaluations & Optimization
- Control System Evaluations & Optimization
- Destruction/Reduction Efficiency Evaluations
- Particle Size Evaluations
- Trial Burns/Risk Assessments
• Receive a cost proposal within **two business days** following the development of a firm scope of work

• Receive a test plan within **two business days** following the receipt of documentation to proceed

• Receive a test report within **two weeks** following the completion of field work or within **two business days** following receipt of laboratory data
Testing You Can Rely On… Partners You Can Trust
Student Introductions
Murphy’s Law  
(The Stack Testing Version)

“Anything that can go wrong, will go wrong…

especially if you have scheduled a stack testing project.”
Common Pitfalls

• Technical Review
• Electrical Power Availability
• Stack Set-up
• Site Safety
Technical Review

PROCESS DESCRIPTION

• Source Type (i.e. furnace, boiler, engine, reactor, etc.)
• What is being produced? How is it produced?
• Continuous or batch process? If a batch process, what is the cycle time?
• What process data is monitored and recorded by the plant?
• What operating parameters will be set by the performance test (if applicable).
Technical Review (cont.)
CONTROL DEVICE DESCRIPTION

• What type of control device is used (i.e. baghouse, ESP, wet or dry scrubber, SCR)?
• What data from the control device is monitored by the plant?
• What control system parameters are being established by the performance test (if applicable).
Technical Review (cont.)

STACK GAS DATA

- Temperature (personnel safety, sampling equipment)
- Estimated flow rate (measurement system, nozzle, sampling time)
- Type of flow (induced or natural draft; fixed or variable drive fan)
- Static pressure (personnel safety, sampling approach)
- Moisture Content (test design, method selection)
Technical Review (cont.)

STACK GAS DATA

- O2 & CO2 concentrations (gas MW, pollutant corrections)
- Target parameters (specific parameters, define PM, VOC)
- Expected concentrations and permit limits of target parameters (method selection, test duration, interferences)
- Are any hazardous pollutants present (personnel safety)
ELECTRICAL POWER AVAILABILITY

Disclaimer: All Mobile Laboratories are not created equal…check with your tester to verify their power requirements.

- How close is power supply to the mobile laboratory staging location?
- Is 480 power available? 240 power? 120 power?
- Can generators be used as an alternative to plant power?
- Is 120 power available on the stack or in close proximity to the sampling location?
STACK SET-UP
STACK DATA

• Is the stack vertical or horizontal?
• Height to test ports from ground?
• Is the stack circular or rectangular?
• Stack diameter (circular) or width/depth (rectangular)?
• Are cyclonic flow conditions anticipated?
STACK SET-UP (cont.)

TEST PORTS

- Are test ports installed? How many?
- Are they located 90 degrees apart or in center of equal areas?
- What are the diameters of the test ports?
- What are the nipple lengths?
- What is the height of the test ports?
- Are monorail supports present? D-rings?
- Are they large enough to accommodate testing equipment?
Test ports located a minimum of 0.5 duct diameters (2 diameters is preferred) upstream of the nearest flow disturbance. Stock exit = flow disturbance.

Test ports apply a minimum of 2 duct diameters (6 diameters is preferred) downstream of the nearest flow disturbance. Disturbance = construction, bars, damper, etc.

Note for rectangular ducts:
Area equivalent diameter = \((2 \times \text{Depth} \times \text{Width}) / (\text{Depth} + \text{Width})\)

Alliance Source Testing
EPA Method 7 Reference Diagram

(Methology is applicable to all duct orientations)
Site Safety

- Is there a safe means available to access the test ports (i.e. ladder, stairs, man lift, elevator)?
- Is there a safe location to conduct testing (i.e. platform, scaffolding, man lift)?
- Can the area below test location be secured?
- How high are the handrails? Are handrails cut?
- Any site specific safety issues (heat stress, respiratory concerns)?
- Work area conditions due to plant operations?
<table>
<thead>
<tr>
<th>Stack Testing Information Sheet</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Company Name:</strong></td>
</tr>
<tr>
<td><strong>Company Address:</strong></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td><strong>City:</strong></td>
</tr>
<tr>
<td><strong>Site Description:</strong> <em>attach sketch if design is unusual</em></td>
</tr>
</tbody>
</table>

**No. of Stacks**
- (list description for each if different)

**Source**

**Control Device(s)**

**Pollutants**

**No./Diameter of Ports**

**Monorail Attachments**

**Stack Height to Ports**
- ft

**Stack Diameter**
- in

**Duct Distances**
- upstream
- downstream

**Platform, Lift or Scaffolding**

**Electrical**
- Type (i.e. 15A, 20A)
- Distance from Port
- Distance from Trailer
- (Trailer) No. of Outlets

**Project Description (please provide detailed process description)**

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**Project Scope/Desired Schedule**

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**Project Requirements**

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Source Testing Basics

- Extract Gas from Source
- Filtration
- Condition Gas Sample
- Analyze or Collect Gas Sample
Source Testing Basics

• Extract Gas from Source
  • Representative Sample
  • Leak free system
  • Constant Rate, Isokinetic Rate
  • Teflon, Stainless Steel, Glass, Quartz

• Filter
  • In stack or out of stack
  • Glass fiber, Quartz, Teflon
Source Testing Basics

• Condition Gas Sample
  • Remove Moisture
  • Filtration for Instruments
  • Direct vs Non-Contact

• Analyze or Collect Gas Sample
  • Instrumental Testing
  • Direct Interface Testing
  • Wet Chemistry Testing
Understanding Gases

- Flow properties
- Pressure
- Temperature
- Gas Laws
- Isokinetic Sampling
Gas Flow

- **Turbulent – RANS**
  - Reynolds averaged Navier Stokes (time averaged equation of flow)
- **Laminar**
  - Parallel layers, no disruption
- **Cyclonic**
  - Swirling due to baffles or other disturbance causing an angle of incident
- **Conservation of mass, conservation of momentum**
  - The rate of change of mass must be equal to the net rate of fluid flow in (or out)
  - Decreasing area = increasing velocity
Gas Pressure & Temperature

• Gas Pressure
  • Barometric (Atmospheric)
  • Gauge (Location)
  • Source = Barometric + Gauge (Absolute)
  • Standard Pressure – ?

• Gas Temperature
  • Measured at each traverse point
  • Absolute (R or K)
  • Standard Temperature – ?
Gas Volume & Ideal Gas Law

• Gas Volume – 3 variables
  – Pressure
  – Temperature
  – Amount/Composition of Gas

• Ideal Gas Law – $PV = nRT$
  – $P$ – absolute pressure
  – $V$ – volume
  – $n$ – number of moles
  – $R$ – universal gas constant
  – $T$ – absolute temperature
Other Gas Laws

• Boyle’s Law
  – When temperature is constant, the volume of an ideal gas varies inversely proportional to absolute pressure

• Charles’s Law
  – At constant pressure, the volume of an ideal gas is directly proportional to the absolute temperature
Sampling Rate

• Isokinetic
  • Entrained Particles
  • Response to pressure boundaries

• Constant Rate
  • Homogenous gas
  • Instrumental sampling
  • Manual method sampling
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Gas Flow Rate

- EPA Method 1
  - Sampling Location
  - Traverse (Sampling) Locations
  - Cyclonic Flow

- EPA Method 2
  - Gas Velocity Pressure
  - Static Pressure
Figure 1-3. Example showing circular stack cross section divided into 12 equal areas, with location of traverse points.

<table>
<thead>
<tr>
<th>Traverse Point</th>
<th>% of Diameter Distance</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.4</td>
</tr>
<tr>
<td>2</td>
<td>14.7</td>
</tr>
<tr>
<td>3</td>
<td>29.5</td>
</tr>
<tr>
<td>4</td>
<td>70.5</td>
</tr>
<tr>
<td>5</td>
<td>85.3</td>
</tr>
<tr>
<td>6</td>
<td>95.6</td>
</tr>
</tbody>
</table>

Figure 1-4. Example showing rectangular stack cross section divided into 12 equal areas, with traverse points at centroid of each area.
Duct Diameters that Measurement Site is Upstream from Flow Disturbance¹ (Distance A)

Higher Number is for Rectangular Stacks or Ducts

Minimum Number of Traverse Points

24 or 25 a points
20 points
16 points
12 points
8 or 9 a points

¹ From Point of Any Type of Disturbance (Bend, Expansion, Contraction, etc.)

Stack Diameter > 0.61 m (24 in.)

Stack Diameter = 0.30 to 0.61 m (12 - 24 in.)

Duct Diameters that Measurement Site is from Downstream to Flow Disturbance¹ (Distance B)

Figure 1-1. Minimum number of traverse points for particulate traverses
Duct diameters that measurement site is upstream from flow disturbance (Distance A)

Higher number is for rectangular stacks or ducts.

- For stack diameter > 0.61 m (24 in.):
  - Minimum number of traverse points: 16 points
  - From point of any type of disturbance (bend, expansion, contraction, etc.)

- For stack diameter = 0.30 to 0.61 m (12 - 24 in.):
  - Minimum number of traverse points: 12 points
  - Stack diameter = 0.30 to 0.61 m (12 - 24 in.)

Figure 1-2. Minimum number of traverse points for velocity (nonparticulate) traverses.
<table>
<thead>
<tr>
<th>Traverse point number on a diameter</th>
<th>Number of traverse points on a diameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>14.6 6.7 4.4 3.2 2.6 2.1 1.8 1.6 1.4 1.3 1.1 1.1</td>
</tr>
<tr>
<td>2</td>
<td>85.4 25.0 14.6 10.5 8.2 6.7 5.7 4.9 4.4 3.9 3.5 3.2</td>
</tr>
<tr>
<td>3</td>
<td>75.0 29.6 19.4 14.6 11.8 9.9 8.5 7.5 6.7 6.0 5.5</td>
</tr>
<tr>
<td>4</td>
<td>93.3 70.4 32.3 22.6 17.7 14.6 12.5 10.9 9.7 8.7 7.9</td>
</tr>
<tr>
<td>5</td>
<td>85.4 67.7 34.2 25.0 20.1 16.9 14.6 12.9 11.6 10.5</td>
</tr>
<tr>
<td>6</td>
<td>95.6 80.6 65.8 35.6 26.9 22.0 18.8 16.5 14.6 13.2</td>
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<tr>
<td>7</td>
<td>89.5 77.4 64.4 36.6 28.3 23.6 20.4 18.0 16.1</td>
</tr>
<tr>
<td>8</td>
<td>96.8 85.4 75.0 63.4 37.5 29.6 25.0 21.8 19.4</td>
</tr>
<tr>
<td>9</td>
<td>91.8 82.3 73.1 62.5 38.2 30.6 26.2 23.0</td>
</tr>
<tr>
<td>10</td>
<td>97.4 88.2 79.9 71.7 61.8 38.8 31.5 27.2</td>
</tr>
<tr>
<td>11</td>
<td>93.3 85.4 78.0 70.4 61.2 39.3 32.3</td>
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<td>12</td>
<td>97.9 90.1 83.1 76.4 69.4 60.7 39.8</td>
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<tr>
<td>13</td>
<td>94.3 87.5 81.2 75.0 68.5 60.2</td>
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<tr>
<td>14</td>
<td>98.2 91.5 85.4 79.6 73.8 67.7</td>
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<td>15</td>
<td>95.1 89.1 83.5 78.2 72.8</td>
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<td>23</td>
<td>96.8</td>
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<tr>
<td>24</td>
<td>99.9</td>
</tr>
</tbody>
</table>
**TABLE 1-1 CROSS-SECTION LAYOUT FOR RECTANGULAR STACKS**

<table>
<thead>
<tr>
<th>Number of tranverse points layout</th>
<th>Matrix</th>
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<tbody>
<tr>
<td>9</td>
<td>3×3</td>
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<tr>
<td>12</td>
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<tr>
<td>16</td>
<td>4×4</td>
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<tr>
<td>20</td>
<td>5×4</td>
</tr>
<tr>
<td>25</td>
<td>5×5</td>
</tr>
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<td>30</td>
<td>6×5</td>
</tr>
<tr>
<td>36</td>
<td>6×6</td>
</tr>
<tr>
<td>42</td>
<td>7×6</td>
</tr>
<tr>
<td>49</td>
<td>7×7</td>
</tr>
</tbody>
</table>
Figure 2-1. Type S Pitot Tube Manometer Assembly.
Figure 2-5. Standard pitot tube design specifications.
Gas Flow Rate

• EPA Method 3
  • Gas Composition
  • Gas Molecular Weight

• EPA Method 4
  • Gas Moisture Content
  • Measured and Theoretical
Flow Measurement
Hands On Training

- Method 1
- Method 2
Quantifying Emissions in 1971
Quantifying Emissions in 2015
Quantifying Low Level Emissions

• Wet Chemistry Testing
  • Pretest Planning (flow rate, target rate, DL, QL)
  • Test Method Bias – zero does not exist
  • Sample Volume (directly proportional)
  • Increased Time & Cost
  • Modified Sample Recovery
  • Modified Analytical Techniques

• Instrumental Analyzers, FTIR
  • High Resolution Analyses
  • FTIR – liquid nitrogen, detectors
Wet Chemistry - Detection 101

Emission Rates are based on 3 Variables

- Stack Gas Flow (Qs)
- Gas Meter Volume (Vm)
- Reported Mass (Mi)
  - Measured Concentration (Ci)
  - Sample Volume (SV)
- Linear Relationship with variables
  - \( Mi = Ci \times SV \)
  - \( ER = Mi \times Qs / Vm \)
Instrumental Parameters

• Homogenous Components
• Low/No solubility in water
• Low boiling point
• High vapor pressure
• Simple means of detection
• Destructive and non-destructive analysis
Common Components

- Oxygen (O\textsubscript{2})
- Carbon dioxide (CO\textsubscript{2})
- Carbon monoxide (CO)
- Oxides of Nitrogen (NO, NO\textsubscript{2})
- Sulphur dioxide (SO\textsubscript{2})
- Total Hydrocarbons (THC)
Means of Detection

• Paramagnetic
  • Oxygen attracted to strongest part of a magnetic field
  • Presence of oxygen displaces nitrogen filled spheres causing a mirror to move
  • Incident light on the mirror is reflected to a photovoltaic cell
  • Moving the mirror changes the amount of light, triggering a feedback signal
  • Current flows through wires on the spheres, generating magnetic field to counter the movement.
Means of Detection

• Infra-Red
  • Primary Infra-red absorbing species - CO, CO2
  • An Infra-red beam is directed through the sample gas
  • Specific frequencies correspond to specific components
  • Infra-red light causes molecules to vibrate or rotate, absorbing photons
  • The loss of photons directly corresponds to the gas concentration
Means of Detection

• Ultraviolet
  • Used for SO₂ measurement
  • An ultraviolet beam is directed through the sample gas.
  • Ultraviolet light disrupts electrons within orbitals. Match the wavelength to the correct orbital energy level.
  • UV photons are absorbed as electrons are excited to higher energy levels.
  • The loss of photons directly corresponds to the gas concentration.
Means of Detection

- Chemi-luminescence
  - Two step process for NO\(_x\)
  - Gas is passed over a heated catalyst to convert all NO\(_2\) to NO
  - NO is oxidized to NO\(_2\) in the presence of ozone
  - This oxidation results in a released photon
    - NO + O\(_3\) → NO\(_2\) + O\(_2\) + hv
    - Photomultiplier “counts” photons directly correlating to the amount of NO oxidized.
Means of Detection

• Flame Ionization Detector (FID)
  • Sample Gas is burned in a oxygen rich atmosphere with a hydrogen fuel source
  • Hydrocarbons undergo complete oxidation, generating formylium (CHO⁺)
  • Positive ions are collected on an negatively charged collector
  • Ionic interaction generates a current directly proportional to the number of ions.
  • The number of ions is directly tied to the number of carbon atoms oxidized
Calibration Principles

- Zeroing
- Spanning
- Low / Mid-point Calibration
- Drift Checking
- Bias checking
- Operational checking
Types of measurements

- Oxygen correction
  - 3%, 7%, 15%
- Emission rates
  - lb/hr, lb/ton, tpy
- Performance Specification
  - RATA and CGA
Specific Methods

- EPA Method 3A
- EPA Method 6C
- EPA Method 7E
- EPA Method 10
- EPA Method 25A
Particulate Matter 101

• Types of Particulate Matter
  • Filterable PM (FPM)
  • Filterable PM$_{10}$ (FPM$_{10}$)
  • Filterable PM$_{2.5}$ (FPM$_{2.5}$)
  • Condensable PM (CPM)

  • PM$_{10}$ (FPM$_{10}$ plus FPM$_{2.5}$ plus CPM)
  • PM$_{2.5}$ (FPM$_{2.5}$ plus CPM)
Particulate Matter 101

- Particulate Matter is defined by Method
  - Filterable PM - Filtration Temperature
    - In-stack
    - 248°F or 320°F
  - Condensable PM - Back half Train Operating Temperature
    - 65 - 85°F
    - As close to 85°F as possible
Particulate Matter Test Methods

- EPA RM 5 or 17
  - PM
  - FPM

- EPA RM 201A
  - FPM_{10}
  - FPM_{2.5}

- EPA RM 202
  - CPM

- EPA RM 201A/202
  - PM_{10}
  - PM_{2.5}
EPA Methods 5 & 17
Parameters - PM, FPM, TSP

- Method 5
  - PM Method for the first group of NSPS
  - Proposed 1971 – included FPM & CPM
  - Promulgated 1971 – included only FPM
  - Quantifying only FPM shifted focus from atmospheric emissions to performance of pollution control equipment

- FPM
  - Temperature defined by Method 5
  - Method 17 (and 5I) involve filtering at stack temperature
  - Filtration temp accounts for major difference in PM collected
EPA RM 5 Diagram

Figure 5-1. Particulate Sampling Train.
EPA Method 201A

Parameter – “filterable” PM$_{10}$ and PM$_{2.5}$

- Old RM 201A, Promulgated 1990
- New RM 201A (f/k/a OTM-27), Promulgated 2010
- Challenges
  - Sampling Rate
  - Sampling Port Size
  - Small Diameter Stacks
  - Variable Gas Characteristics – temp, moisture, velocity
  - Wet Stacks – use RM 5 until method is developed
EPA RM 201A Diagram

Figure 1. In-stack PM$_{10}$ and PM$_{2.5}$ Sampling Train
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M17 & M201A Systems
PM$_{10}$ & PM$_{2.5}$ Cyclones
EPA Method 202
Parameter – Condensable PM (CPM)

• What is CPM?
  • Vapors & gases at stack (filtration) temperature
    • Form liquid or solid aerosols at ambient temperature
    • Semi-volatile organic compounds
    • Semi-volatile inorganic compounds (i.e. SO3, ammonium nitrate)
  • Acid or basic gases
    • Chemically react at reduced temperatures
    • NH3, HCl, HF, Cl2, SO2
• Characteristics
  • Final particle size achieved up to 4 minutes after cooling
  • Typical final particle size is ~ 0.75 μm
Figure 1. Schematic of Condensable Particulate Sampling Train
EPA RM 202 Challenges

• Low Target Rates & Decreased Sampling Rate with 201A
• Positive Bias
  • Pretest Cleaning, Baking
  • Blanks - Reagent, Proof, Field
  • Hexane Squirt Bottles
    • Teflon - 0.1 mg after 4 days
    • HD Polypropylene - 1-2 mg after 4 days
    • LD Polypropylene - 20-25 mg after 4 days
    • Polyethylene - 2 mg after 30 minutes, 50 mg after 4 days
EPA RM 202 Challenges

- Precision - ~ 4.0 mg
  - Organic CPM - ~ 0.5 mg
  - Inorganic CPM - ~ 3.5 mg
Highlight: Estimating CPM
• Hands on Training

• Method 4
• Method 5
Advanced Analytical Techniques

Jordan Laster
Technical Director
jordan.laster@stacktest.com
(610) 500-3615
Advanced Analytical Techniques

• Direct Interface FTIR

• Direct Interface GC

• Laboratory Analysis
Up Next –

Fourier Transform Infra-Red Spectroscopy
FTIR Outline

• Intro to FTIR
  • What is Spectroscopy?
  • What makes FTIR different from other instruments?

• Common and Uncommon Applications
  • Where can I use it?
  • What can I use it for?

• General Perceptions
  • What’s the answer?
  • Why not?
What is Spectroscopy?

• Light is measured in discrete packets of energy
  • Particle / Wave duality

• Light can interact with matter
  • Electron Excitement
  • Molecular Vibration & Rotation

• Ways to measure the interaction
  • Ultraviolet frequencies
  • Visible frequencies
  • Infrared frequencies
Quick Physics Lesson

- Wave Properties
  - Wavelength and frequency are measurable and can be manipulated
  - Constructive and Destructive interference

- Particle Properties
  - Can alter physical conditions by impact

- Type of interaction is determined by wavelength
  - UV: 10-400 nm
  - Vis: 400-700 nm
  - IR: 700-1,000,000 nm
    - Near: 780-3,000 nm
    - Mid: 3000-50,000 nm
    - Far: Up to 1,000,000 nm
Absorption Spectrometer

Light Source

Band Filter

Sample

Detector

Electronics

Display
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What makes FTIR Different?

- Completely unique response for every compound
  - Must exhibit IR absorbance (no homo-nuclear diatomics)
  - No “Cross-talk”
- Recorded spectra for follow-up analysis
  - Sample spectra can be re-analyzed later for additional compounds
- Minimal need for calibration gases on-site
  - Calibration spectra are all stored on the computer for reference
  - Individual compounds do not need direct calibration in the field
- Analysis performed on a “hot/wet” basis
  - Filtration is the only gas conditioning required
  - Analysis can be done on a dry basis if desired
FTIR Spectrometer

Light Source

Michelson Interferometer

Sample

Detector

Electronics

Computer
Sample FTIR Spectrum
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Absolute Measurement

• Stored references can be used on and spectrometer with the same resolution
  – Samples of pure compounds and dilutions in nitrogen or zero air
  – PNNL, NIST, EPA, or commercially available libraries
  – Internally generated references for custom projects

• Calibration transfer standard
  – Measures the optical pathlength so references can be applied correctly
  – Confirms analyzer performance
Field Confirmation

- EPA FTIR Protocol
- EPA Method 320
- EPA Method 321
- EPA Method 318
- ASTM D6348-03
- Sample system spiking
  - Proves that the sampling system is delivering the target compound
  - Proves that the analytical method is accurately accounting for interference
- Sample flow rate
  - Unique sample volume versus flow, sample changeover time
Certainties

• If the pathlength is verified, the only possible accountability for incorrect values is a sample system issue or poor method configuration
• Traditional manual methods have more sources of error
• Quick spot checks and hand-held analyzers may not meet the same rigor
• Many other measurement approaches not based on first order measurement principles.
Common Applications

• Compliance Testing
  - Increasingly requested or allowed by EPA and state Agencies
    • CAA Section 114 ICR Letters
    • MACT Rules
    • State Permits

• Investigative / Diagnostic Testing
  - Control System – sizing, optimizing, troubleshooting
  - General research
  - Mass balance
Common Control Devices

- Wet Scrubber
- Adsorption tower
- RCO/RTO
- TO
- Dry Condenser
- Catalyst beds
Common Industries

• NG aggregation & compression
• Petroleum Refining
• Specialty petro-chemical
• Cement Manufacturing
• Coal / Diesel EGU
• Building Material Manufacturing
• Aluminum Recycling
• Iron/Steel milling
• Non-petroleum based specialty chemical
• Waste to Energy & Incineration
Common Target Analytes

- Alcohols & Aldehydes
- Alkanes, -enes, -ynes
- Aromatics
- Acid Gases
- Fluorocarbons
- Reduced Sulphurs
Other Applications

- Estimating CPM contributions
  - NH$_3$, HCl
- Material off-gassing
  - Typically HAPs
- Capture Efficiency
  - SF$_6$
- Ambient air “hotspot sniffing”
  - Any compounds of interest
Highlight – Estimating CPM

- Facility was concerned about CPM emissions
  - Based on analysis of previous CPM samples, ammonium sulfate was identified as a significant portion of the catch
  - Based on the process, it was determined that ammonia may be combining with $SO_2/SO_3$
Cost/Benefit Summary

- Multi-component real time data
  - Power to know compliance on the spot
  - Power to monitor real time process changes
  - Monitor 4, 5, 6..18..? components
  - Revisit previous data
- Stack Testers with internal knowledge and experience
- Highly trained for complicated or unusual situations
- Expensive equipment to own and to operate
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FTIR Sampling & Response Demonstration
Up Next –

Gas Chromatography
GC Outline

• Intro to Gas Chromatography
  • What is Chromatography?
  • What makes GC different from other instruments?
  • What are the different types of GCs?

• Common and Uncommon Applications
  • Where can I use it?
  • What can I use it for?
Chromatography Refresher

• Chromatography in general
  – Separation
  • Physical Properties
  • Electrochemical Properties

• Gas Chromatography (GC)
  • Gas phase injection
  • Consists of injection port, heated oven, column, and detector
  • Column options based on application
  • Detector based on application
Other types of Chromatography

• Liquid Chromatography (LC)
  • Liquid phase injection
  • Consists of injection port, column, and detector
  • Column options based on application
  • Detector based on application

• High Performance Liquid Chromatography (HPLC)
  • Essentially the same but using higher pressure to expedite elution
GC Detail

• Sampling Frequency
  • Requires discrete injections of sample gas

• Injection run time
  • Dependent on target analyte(s)

• Calibration
  • Method dependent
  • Typically 3-5 calibration points per analyte
  • Direct calibration eliminates the need for response factor corrections
Detection Principles

• Flame ionization detector
  • Most common, standard organics, most versatile
• Flame Photometric detector
• Thermal Conductivity detector
• Nitrogen Phosphorus detector
• Photoionization Detector
• Electrolytic Conductivity Detector
• Mass Spectrometer
Detector Highlight

- Flame ionization detector
  - Compounds are burned in a H₂ rich atmosphere
  - Carbon compounds produce ions which are detected
  - Typically used for HAPs
  - EPA Method 18

- Flame photometric detector
  - Compounds are burned in a H₂ rich atmosphere
  - Sulphur and Phosphorous emit light at specific frequencies
  - Photomultiplier measures the light at specific wavelengths
  - EPA Methods 15 and 16
Common Applications

• Volatile Organic Compounds (VOCs)

• Hazardous Air Pollutants (HAPs)
  • Wood HAPs
  • Steel HAPs
  • Ethanol HAPs
  • PVC HAPs

• Reduced Sulphurs

• Bulk Gas Analysis
Up Next –

Off Site (Laboratory) Analysis
Off-Site Analysis

- When should an external lab be involved?
- How do I select the proper laboratory?
- What kinds of samples will be collected?
- What happens to the samples?
- When can I expect results?
- Who can help translate the lab report?
Reasons to Involve a Laboratory

- No suitable direct interface approach
- NSPS, NESHAP, Permit restrictions
- Cost differential
- Project timeline & availability
Let the Stack Testers do it!

- Knowledge of services offered by a variety of labs
- Vendor/Client relationships can mitigate surprises and issues
- Understanding of regulations calling for accreditation
- Special circumstances
Types of Samples

- Compliance vs. Investigative
  - QA/QC Measures
    - Spikes, Blanks, Duplicates
    - Did the collection and analysis work?
  - Number of samples
    - Typically 3 per source or operating condition
    - Which one is an outlier?
  - Collection duration
    - Typically not less than one hour, could be 8 or more
    - Grab samples vs. Integrated samples, how low can it go?
QA/QC for samples

• Different methods call for different QC sets
  • Spiking for Tubes and Condensates
  • Spiking for bags (EPA Method 18)
  • Duplicate samples
  • Laboratory duplicates & Multiple injections
  • Field Blanks
  • Laboratory Blanks
Common Options

• Blank analysis

• Multiple Fractions

• Expedited analysis

• Multiple laboratories
Sample Media

- Filters & Rinses
  - Mostly particulate samples
- Condensates
  - Polar organics, high Bp, low vapor pressure, entrained liquids
- Bags
  - Low Bp / high vapor pressure, non-reactive
- Adsorbent Tubes
  - Most organics, low detection limits needed
- Canisters
  - Ambient air and flammable gases
Most common combinations

• EPA Method 201A/202
• EPA Method 18 Tubes
• EPA Method 18 Bags
• SW-846 Method 0010
• NCASI 99.02
Method 5/202

Figure 1. Schematic of Condensable Particulate Sampling Train
Method 18 Tubes

Used with permission from Enthalpy Analytical, Inc.
Method 0010
Sample Journey

- Collection
  - Initiation of Chain of Custody
- Transportation
  - Different samples, different requirements
- Receipt at laboratory
  - Verification of Chain of Custody
- Sample Analysis
  - Extraction/Recovery, preparation, analysis & reporting
Analytical Approaches

- Gravimetric
- GC (FID, TCD, NPD, ECD, PID, FPD)
- HPLC (UV, ECD)
- GCMS (SIM, SCAN)
- Spec (UV/VIS)
- Phospholuminescence
- SEM-XRF
- Atomic Absorption
General Protocol Notes

• Due Dates
  – Part 60 Testing – 30 Days Prior
  – Part 63 Testing – 60 Days Prior
  – Permit Testing – Varies by State

• Test Plan Requirements – SSTP, State Specific Protocol Requirements, Etc.

• Facility Information / Site Contact

• Current Permit & Limits

• Process Operating / Control System Data
General Reporting Notes

- Basic Requirements
  - Summary of Results
  - Facility & Source Information
  - Methodology
  - Field Data & QA/QC Data
  - Process/Control System Data
    - Feed Rate, Fuel, Production Rate, etc.
    - Scrubber Flow, Baghouse DP, etc.
What is the ERT

- Stand alone Microsoft Access application
  - One application for those with MS Access
  - One “Run Time” program for those without MS Access
- Standardized format
- Incorporates all information identified in Source Test Plan and Report Guidance
- Initiated by source or source test contractor
- Guides EPA/State review, assessment & comment entry and stores entries with information provided by source
- Generates export file for transmission to external data systems
### Current Standards requiring ERT

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How is the ERT used by Source

- Source fills out test plan and test report portions of ERT
  - May attach additional documentation
  - Limited number of “Required fields”
- EPA or State establishes required “data fields”
  - ERT requires some critical fields to get past test plan
  - State or EPA fields flexible
- Several fields are drop down menus
- Some fields are open free text fields
- Current ERT provides a print version of a summary test plan and test report
- Project Data file produced by ERT is transmitted to Agency or data system (e-mail, FTP site, CDX)
How is the ERT used by EPA

- EPA or State review is by check list with option for text comment (both test plan and test report)
- Review items provided but reviewer decides which items to evaluate
- Compare electronic and printed version of summary
- ERT generates an XML file of selected data elements for use by WebFIRE (the Emissions Factor data system)
- WebFIRE will import the data elements and the complete test file for factor calculation and support documentation
- ERT has many additional data elements that are available for use in several data systems (AIRS, NEI, etc.) but not currently exported
Resource Expenditure (future)

- Industry source testing increasing
  - MACT, NSPS, NSR/PSD
  - SIP, State compliance
- Increased emissions accuracy demand
  - Use of test data in inventories
  - More and better emissions factors
- State resources stagnant or decreasing
- Federal emissions factors development resources decreasing
- Possible increased public scrutiny of test data quality
EF Stakeholder Recommendations

- Improve quality of test data while maintaining or reducing burdens
  - Standardize reporting
  - Assess quality quantitatively
  - Automate manual transcription and evaluation processes
- Employ electronic data management
  - Increase transportability of data
  - Reduce storage space
  - Improve ability to search
Electronic Data Resource Implications

- Report generation by source
  - Same information required
  - Format same for all States
  - Potential reduction in resubmitted plans & reports
- Report review by EPA or State
  - Reduce data transcription time
  - Reduce data transcription errors
  - Allow for open sharing of data
    - Within State agency
    - Between States
    - Between States, Feds and sources
  - Reduce physical file storage space
  - Response times reduced
Possible Data Applications

- Improve Emissions Factors
- Improve test report assessment for compliance evaluation
- Initiator of non-EF data flow
  - Population of internal data systems
  - AIRS/AFS submissions
  - Emissions Inventory submissions
- Use for emissions standards data
  - State limits (non Federal, SIP)
  - Federal NSPS, MACT, NSR/PSD
Recent ERT Improvements

• ERT is CROMERR compliant
• Improved linking of field data spreadsheets
• Added option to link instrumental test and isokinetic tests ($O_2$, $CO_2$, flow)
• Additional data fields for export routine
• Expanded test methods submitted by ERT
  • Improved Custom test method procedures
  • RATA’s ($CO$, $CO_2$, NOx, $O_2$, SO$_2$)
Future ERT Improvements

- Improve Test Completeness Assessment
- Improve State Review Guide Sheet
- Link State Review with published method
- Additional data fields for export routine
- Develop data definitions and formatting for import of lab data and export of data fields
- Expand test methods submitted by ERT
  - Methods 30B, 18, 320, 321, 0010, 0031
  - Additional RATA’s (TRS, PM, Hg, etc)
Questions & Discussion