Technical Support of NCore Implementation
Gas Instrument Support

Dennis Mikel
EPA Office of Air Quality, Planning and Standards
• Overview of NCore Requirements
• Mass Flow Controllers (MFC) Calibrators
• Compressed Gas Cylinders
• Zero Air Generators
• Summary
Overview of NCore Requirements

• Quality Control (QC), i.e., Precision Checks
• Multipoint Calibration
• Level I/Zero Span Checks
• Method Detection Limit (MDL) Test
• Zero Air Certification Test

• To perform these test you will need:
  – Mass Flow Calibrators (MFCs)
  – Compressed Gas Standards
  – Recommends Zero Air Systems
NCore Calibration and QC Checks

• QC Checks (Precision):
  – Required (40 CFR 58 Appendix A)
  – Minimum: Once every two weeks
• Multipoints: 1 in 6 months, repair or startup
• Level I Zero Spans: Recommend Daily
• MDL Tests: Annually
• Zero Air Certification Test: Annually
# NCore Calibration and QC Checks

<table>
<thead>
<tr>
<th>Item</th>
<th>CO</th>
<th>SO2</th>
<th>NOy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Full Scale Range</td>
<td>0 to 5000 ppb</td>
<td>0 to 100 ppb</td>
<td>0 to 200 ppb</td>
</tr>
<tr>
<td>Cylinder Concentration</td>
<td>200 – 300 ppm</td>
<td>10 - 15 ppm</td>
<td>10 – 30 ppm</td>
</tr>
<tr>
<td>Calibration Ranges</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>zero</td>
<td>40 ppb</td>
<td>0.100 ppb</td>
<td>0.050 ppb</td>
</tr>
<tr>
<td>Level I Span</td>
<td>4500 ppb</td>
<td>90 ppb</td>
<td>180 ppb</td>
</tr>
<tr>
<td>Mid Point Span</td>
<td>2500 ppb</td>
<td>50 ppb</td>
<td>100 ppb</td>
</tr>
<tr>
<td>Precision Level</td>
<td>500 ppb</td>
<td>.20 ppb</td>
<td>40 ppb</td>
</tr>
</tbody>
</table>
Method Detection Limit Test

• Use a concentration of 2.5 to 5 times the instrument signal/noise
• Run zero gas through analyzer
• Dilute the calibration gas to estimated concentration level and collect readings for a predetermined length of time:
  – Suggested: 20-25 1-minute observations, repeated 7 times over the course of 5-14 days. Average the concentration from these readings.

• Calculate the MDL as:

\[ MDL = t_{0.01,(n-1)} \cdot s \]

Where \( t_{0.01,(n-1)} \) represents the 99th quantile of a Student’s t distribution with (n-1) degrees of freedom and n represents the number of replicate measurements and s is the standard deviation.
Zero Air Certification Test

- Replace the zero air generator with certified zero air cylinder
- Program Calibrator to perform a zero test
- Compare the results of the cylinder to air generator.

If response to generator and cylinder are equal to or below:
  - NO  50 ppt
  - SO2 100 ppt
  - CO   40 ppb

Then, generator is operating within tolerance for Precursor Gas instruments.
MFC Theory

- MFC technology works on a very simple principle!
- Each MFC has a Thermister (a Thermal Resistor)
  - Thermisters are sensitive to heat
  - As air passes over it, it loses heat, the more air flow, the more heat that is lost
  - This action changes the resistance of the thermister
  - The change in resistance is monitored by a sensitive electronic feedback loop. The resistance is converted to voltage and controlled by computer.
MFC Theory-Calibrator Diagram
MFC Calibrator Features

Thermo (TEI) Model 146

Enviroricons Model 9100
MFC Calibrator Critical Features

• Gas Flow – 0 to 100 cc/min
• Air Flow – 0 – 20 L/min
• Multiple Gas ports - optional
• Built in traceable ozone generator
• Accuracy +/- 1% Full Scale
• Precision +/- 1% Full Scale
• Linearity +/- 1% Full Scale
# MFC Calibrator Matrix

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Ecotech 1100</td>
<td>0-500 sccm</td>
<td>0-20 lpm</td>
<td>4 Std.</td>
<td>Yes, 0 – 1ppm</td>
<td>+/- 1.0% Full Scale</td>
<td>+/- 0.15% Full Scale</td>
<td>0.15% Full Scale</td>
</tr>
<tr>
<td>Environics 9100</td>
<td>0-100 sccm</td>
<td>0-20 lpm</td>
<td>2 Std.</td>
<td>Yes, 0.5 – 1.25 ppm</td>
<td>+/- 1.0% Full Scale</td>
<td>+/- 1.0% Full Scale</td>
<td>1.0% Full Scale</td>
</tr>
<tr>
<td>Sabio 4010L</td>
<td>0-1000 sccm</td>
<td>0-20 lpm</td>
<td>Yes</td>
<td>Yes, 0 – 2 ppm</td>
<td>+/- 1.0% Full Scale</td>
<td>+/- 0.15% Full Scale</td>
<td>0.5% Full Scale</td>
</tr>
<tr>
<td>Tanabyte 300</td>
<td>0-100 sccm</td>
<td>0-10 lpm</td>
<td>Yes</td>
<td>Yes, 0 – 2 ppm</td>
<td>+/- 0.5% Full Scale</td>
<td>+/- 0.1% Full Scale</td>
<td>0.5% Full Scale</td>
</tr>
<tr>
<td>Teledyne-API 700E</td>
<td>0-200 sccm</td>
<td>0-20 lpm</td>
<td>4 Std.</td>
<td>Yes, 0.1 – 10 ppm</td>
<td>+/- 1.0% Full Scale</td>
<td>+/- 0.2% Full Scale</td>
<td>0.5% Full Scale</td>
</tr>
<tr>
<td>Thermo 146C</td>
<td>0-200 sccm</td>
<td>0-20 lpm</td>
<td>NA</td>
<td>Yes</td>
<td>+/- 1.0% Full Scale</td>
<td>+/- 1.0% Full Scale</td>
<td>1.5% Full Scale</td>
</tr>
</tbody>
</table>

* Highest optional ranges.
MFC Calibrator Critical Features

- Programmable Scheduled Tasks
- Front Panel Display
- Integrated Switch Closure System
- Calibrators should be remote access/control ready
- Digital Inputs – controlled by remote access
- Digital Outputs – signals to remote device
## MFC Calibrator Matrix (cont.)

<table>
<thead>
<tr>
<th>Calibrator</th>
<th>Programmable Calibrations</th>
<th>Display</th>
<th>Digital Inputs</th>
<th>Digital Outputs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ecotech 1100</td>
<td>Yes, 20 sequences cycled through 5 points</td>
<td>4 line LCD</td>
<td>Relay contact closures or TTL logic</td>
<td>8 digital outputs for sequence indication</td>
</tr>
<tr>
<td>Environics 9100</td>
<td>Yes, 21 sequences (3/day) through 5 points</td>
<td>LCD</td>
<td>Optional RS-232 serial data interface</td>
<td>8 digital outputs for sequence indication</td>
</tr>
<tr>
<td>Sabio 4010L</td>
<td>20 timer driven cal routines, user defined sequences on a 7 day calendar</td>
<td>Bright active matrix color display</td>
<td>24 bit Digital Input, TTL logic levels, RS-232</td>
<td>24 bit output, 2 serial ports for communication</td>
</tr>
<tr>
<td>Tanabyte 300</td>
<td>Menu operation allows auto or manual calibrations</td>
<td>LCD</td>
<td>TTL contact closures allows remote operation</td>
<td>Transparent pass serial I/O after programmable pass-code is received</td>
</tr>
<tr>
<td>Teledyne- API 700E</td>
<td>Readout and control by front panel</td>
<td>2 line LCD</td>
<td>Via RS-232 12 opto-isolated inputs</td>
<td>Via RS-232 12 opto-isolated outputs</td>
</tr>
<tr>
<td>Thermo 146C</td>
<td>10 calibration events</td>
<td>4 line LCD</td>
<td>RS-232 remote access</td>
<td>RS-232 remote access</td>
</tr>
</tbody>
</table>
MFC Calibrator Issues

- Certify your MFC against a NIST traceable flow device (such as a Bios DryCal, Gilibrator or Hastings Bubble Kit or other flow device)
- NIST Traceable flow device should be certified annually (or if you suspect a problem)
- Perform quarterly checks of your MFCs (or until establish trend)
- Calculate True flow vs Flow Set Points
  - Air Flow: 0 – 20 lpm (recommended)
  - Gas Flow 0 – 100 cc/min (recommended)
- Corrections should be made to STP (25°C and 760 Torr)
# MFC Calibrations

## Air Flow Results (lpm)

<table>
<thead>
<tr>
<th>Setting</th>
<th>Display</th>
<th>BIOS</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.000</td>
<td>4.986</td>
<td>5.187</td>
</tr>
<tr>
<td>6.000</td>
<td>5.995</td>
<td>6.194</td>
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<tr>
<td>8.000</td>
<td>7.995</td>
<td>8.106</td>
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<tr>
<td>10.000</td>
<td>10.000</td>
<td>10.160</td>
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<tr>
<td>12.000</td>
<td>12.000</td>
<td>12.104</td>
</tr>
<tr>
<td>14.000</td>
<td>14.000</td>
<td>14.080</td>
</tr>
<tr>
<td>16.000</td>
<td>15.990</td>
<td>16.170</td>
</tr>
<tr>
<td>18.000</td>
<td>18.000</td>
<td>18.154</td>
</tr>
<tr>
<td>20.000</td>
<td>19.990</td>
<td>19.328</td>
</tr>
</tbody>
</table>

## Gas Flow Results (cc/min)

<table>
<thead>
<tr>
<th>Setting</th>
<th>Display</th>
<th>BIOS</th>
</tr>
</thead>
<tbody>
<tr>
<td>10.000</td>
<td>9.933</td>
<td>7.616</td>
</tr>
<tr>
<td>13.000</td>
<td>12.913</td>
<td>10.546</td>
</tr>
<tr>
<td>20.000</td>
<td>19.957</td>
<td>17.073</td>
</tr>
<tr>
<td>30.000</td>
<td>29.975</td>
<td>26.616</td>
</tr>
<tr>
<td>40.000</td>
<td>39.941</td>
<td>36.540</td>
</tr>
<tr>
<td>50.000</td>
<td>49.958</td>
<td>46.424</td>
</tr>
<tr>
<td>60.000</td>
<td>59.976</td>
<td>56.122</td>
</tr>
<tr>
<td>70.000</td>
<td>69.994</td>
<td>67.697</td>
</tr>
<tr>
<td>80.000</td>
<td>80.090</td>
<td>78.670</td>
</tr>
<tr>
<td>90.000</td>
<td>89.978</td>
<td>89.505</td>
</tr>
<tr>
<td>100.000</td>
<td>99.995</td>
<td>97.403</td>
</tr>
</tbody>
</table>
MFC Calibrations

Air Flow Calibration
Y = 0.9694x + 0.424
R² = 0.9981

Gas Flow Calibration
Y = 1.0138x - 3.323
R² = 0.9992
Compressed Gas Cylinders - Features

• Come in variety of sizes (size 50 or 150)
• Get EPA Protocol certification
• Use Stainless Steel regulators and cylinder valves
• Use Stainless steel or Teflon lines from Regulator to MFC (Teflon recommended)
Compressed Gas Cylinders - Issues

- Recommend reputable vendors
- Handle with Care
- Make sure the cylinders are secure!!
- Read your MSDS Sheets
- Leaks!!
Zero Air Source - Features

TEI Model 111

T_API Model 701
Zero Air Source - Features

Materials utilized for pollutant removal

- Cooler and Water Trap
- Hopcalite, Palladium or Carulite (scrubs CO)
- Purafil (scrubs NO)
- Activated Charcoal (scrubs SO2, O3 and NO2)
Zero Air Systems - Features
Zero Air Source - Issues

The Zero Generator or Cylinders should be able to provide air below the stated Lower Detection Limits of the instruments you are testing. How clean is clean??

• NO  50 ppt
• SO2  100 ppt
• CO   40 ppb

Check the specifications before you purchase.
Summary

- QC Checks and Calibrations are required for NCore Monitoring Stations
- The MFC systems available today are compatible with the PG instruments
- Lower concentrations cylinders are required since PG instruments have lower ranges and levels of detection
- Gas cylinders should be certified – EPA Protocol
- Zero air generators should be able to "scrub" below the LDLs of the PG instruments
Technical Support of NCore Implementation
Manifold and Inlet Design

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Outline

• Sample Manifold Issues
• Residence Time Determination
• Types of Manifolds
• Line Placement
• Ambient/Calibration Manifold Interface
• Through the Probe (TTP) Audits
• Summary
Sample Manifold Issues

There are important variables affecting the ambient air gaseous instruments sampling manifold design:

- residence time of gases
- construction materials
- diameter, length
- flow rate
- pressure drop
Sample Manifold Issues

Construction materials

Code of Federal Regulations (CFR), Title 40 Part 58, Appendix E.9a states,

“For the reactive gases, SO2, NO2 and O3 special probe material must be used for point analyzers…. Of the above materials, only Pyrex® glass, and Teflon® have found to be acceptable for use as intake sampling lines for all the reactive gaseous pollutants…. Therefore, borosilicate glass, FEP Teflon or their equivalents must be the only material in the sampling train…”

- Borosilicate Glass
- Teflon
  - fluorinated ethylene propylene (FEP)
  - Polytetrafluoroethylene (PTFE)*
- Aluminum or Steel OK if glass or Teflon lined

*Not in the CFR, however, it is an equivalent material
Residence time Determination

Code of Federal Regulations (CFR), Title 40 Part 58, Appendix E.9c states,

“Ozone in the presence of NO will show significant losses even in the most inert probe material when the residence time exceeds 20 seconds. Other studies indicate that 10-second or less residence time is easily achievable.” Therefore, sampling probes for reactive gas monitors at NCORE must have a sample residence time less than 20 seconds.”

Total Volume = \(Cv + Mv + Lv\)

Where:
- \(Cv\) = Volume of the sample cane and extensions
- \(Mv\) = Volume of the sample manifold and trap
- \(Lv\) = Volume of the instrument lines from the manifold to the instrument bulkhead
Residence Time Determination

Each of the components of the sampling system must be measured individually. To measure the volume of the components, use the following calculation:

\[ V = \pi \times \left(\frac{d}{2}\right)^2 \times L \]

Where:

- \( V \) = volume of the component
- \( \pi \) = 3.14
- \( L \) = length of the component
- \( d \) = inside diameter
Residence Time Determination

Hot Wire Flow Device

- Real-time flow
- Thermistor Type
- Allows operator to track flow deviations
Residence Time Determination
Types of Manifolds

There are a number of different types of manifolds available

• Laminar Flow
• Teflon lines
• ”T” Type – Horizontal Modular
• CARB - Octopus
• Vertical Manifold
Types of Manifolds

Laminar Flow

- High Flows
- Difficult to Clean
- Temperature Difference
- Can’t be audited by TTP
- Not Allowed in New Regulation (40 CFR 58)
Types of Manifolds

Teflon Lines

• Teflon lines can deteriorate in sun and weather (dry winds)
• Difficult to Clean
• Insect Accumulation
• Pressure differentials
Types of Manifolds

Conventional Manifold

- Borosilicate Glass or Teflon
- Modular
- Use of Drop Out
- Moisture issues
- Fan/Blower
- Heating optional
Types of Manifolds
Types of Manifolds
Types of Manifolds
Types of Manifolds

CARB/Octopus Style

- Borosilicate Glass
- Low Profile
- Drop Out
- No need for Blower
Types of Manifolds
Types of Manifolds
Types of Manifolds

Vertical Flow Manifold

- Borosilicate Glass
- Moisture issues
- Heating optional
Line Placement is very important!!

- Cal gas must flow past the instruments inlets before being exhausted

- Optimize your line placement!
Ambient/Calibration Manifold Interface

Burden’s Creek Monitoring Setup

- Solenoid Switching
- Calibration Manifold
- Interface to DAS
Ambient/Calibration Manifold Interface

**Burden’s Creek**

- Solenoid Switching
- Calibration Manifold
- Interface to DAS
Ambient/Calibration Manifold Interface

**Burden’s Creek Calibration System**
Through the Probe Audit
TTP Audit Design

- Audit Gas should pass through all inlets, manifolds, solenoids and instrument lines
- Auditor should make sure the gas is not pressurizing the instruments!
- Your manifold must be compatible with TTP system
Summary

• Numerous issues to be considered
• There are a number of designs
• The sample manifold must be “integrated” into the monitoring system
• Solenoids can be the link between the calibration/ambient systems
• DAS allow the user more freedom to control the monitoring systems
• Your manifolds will need to be compatible with TTP audits