Field Calibrations

PQAO Training 2017

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Calibrations

- Right now: Introduction, Big Picture
- This afternoon:
  - Demo: Ozone Analyzer Calibration
  - Some additional slides (nitty gritty)
  - More time for Q&A

Session: Station Operations & Documentation “B”
Room: Mountain Vista 2
Times: 2:35 to 3:15 and again 4:20 to 5:00
Some Definitions

“Calibration” is defined as:

The comparison of a measurement standard, instrument, or item with a standard or instrument of higher accuracy to detect and quantify inaccuracies and to report or eliminate those inaccuracies by adjustment.

(EPA QA Handbook Vol II)

“Verification”: comparison, but no adjustment made

We’ll focus on gas instruments, but concepts apply to PM and meteorology, too.
How

- Calibrations should be carried out
  - at the field monitoring site
  - by allowing the analyzer to sample test atmospheres containing known pollutant concentrations

- The analyzer should be warmed up:
  - at least several hours but preferable overnight
  - Warm-up periods (per API):
    - 2 hours (O3)
    - 24 – 48 hours (NOx)
    - 4 – 7 days (TCO)
During the calibration, the analyzer should be operating in its normal sampling mode, including:

- Filters / scrubbers / conditioners
- Through as much of the ambient air inlet system as is practicable. ➔ Ideally, through the probe.

Keep this in mind when:

- Designing new station
- Purchasing calibration equipment
When

- EPA QA Vol II, Section 12.3:
  - Installation
  - Relocation
  - Repairs or Service
  - Interruption
  - Upon any indication of potential malfunction
  - At least every 6 or 12 months (see below)
  - Station shutdown

- Minimum frequency of calibration (EPA QA Vol II, App D)
  - Every 6 months
  - Or annually, if zero/span check is done daily

- Keep track of when calibrations are happening!
Audits:
- No calibrations within 30 days before an audit (CARB QA)

Ambient Conditions:
- Pollution: not during pollutant episodes, (i.e., $O_3$ not in afternoon)
- $O_3$: Not during $O_3$ season
- Met / Particulates: not during high wind conditions or adverse weather conditions due to safety
How Many Points?

- EPA requirements (EPA QA Vol II. App D):
  - Ozone, SO\textsubscript{2}, Carbon Monoxide:
    - Zero
    - 4 Upscale Points
  - NO, NO\textsubscript{2}, NO\textsubscript{x}:
    - Zero
    - 4 Upscale Points for NO & NO\textsubscript{x}
    - 3 NO\textsubscript{2} Titration Points
No EPA/ARB requirements, but:
- Highest point should be 80% of full scale and >NAAQS (EPA QA Vol II)
- Keep limits of calibrator (i.e. MFC ranges) in mind
- Good idea to bracket audit levels
- ARB proposed 2017 audit levels (ppm):

<table>
<thead>
<tr>
<th>Audit Point</th>
<th>O3</th>
<th>CO</th>
<th>NO2</th>
<th>SO2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.130</td>
<td>17.0</td>
<td>0.147</td>
<td>0.120</td>
</tr>
<tr>
<td>2</td>
<td>0.080</td>
<td>12.0</td>
<td>0.075</td>
<td>0.080</td>
</tr>
<tr>
<td>3</td>
<td>0.055</td>
<td>6.0</td>
<td>0.035</td>
<td>0.040</td>
</tr>
<tr>
<td>4</td>
<td>0.030</td>
<td>2.0</td>
<td>0.014</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>0.015</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
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</tr>
</thead>
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<tr>
<td>1</td>
<td></td>
<td>2.00</td>
<td></td>
<td>0.035</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>0.70</td>
<td></td>
<td>0.015</td>
</tr>
<tr>
<td>3</td>
<td></td>
<td>0.11</td>
<td></td>
<td>0.004</td>
</tr>
<tr>
<td>4</td>
<td></td>
<td>0.04</td>
<td></td>
<td>0.002</td>
</tr>
</tbody>
</table>
### What Levels (Concentrations)?

- **EPA audit levels:**

<table>
<thead>
<tr>
<th>Audit Level</th>
<th>$O_3$ (ppm)</th>
<th>$SO_2$ (ppm)</th>
<th>$NO_2$ (ppm)</th>
<th>$CO$ (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.004-0.0059</td>
<td>0.0003-0.0029</td>
<td>0.0003-0.0029</td>
<td>0.020-0.059</td>
</tr>
<tr>
<td>2</td>
<td>0.006-0.019</td>
<td>0.0030-0.0049</td>
<td>0.0030-0.0049</td>
<td>0.060-0.199</td>
</tr>
<tr>
<td>3</td>
<td>0.020-0.039</td>
<td>0.0050-0.0079</td>
<td>0.0050-0.0079</td>
<td>0.200-0.899</td>
</tr>
<tr>
<td>4</td>
<td>0.040-0.069</td>
<td>0.0080-0.0199</td>
<td>0.0080-0.0199</td>
<td>0.900-2.999</td>
</tr>
<tr>
<td>5</td>
<td>0.070-0.089</td>
<td>0.0200-0.0499</td>
<td>0.0200-0.0499</td>
<td>3.000-7.999</td>
</tr>
<tr>
<td>6</td>
<td>0.090-0.119</td>
<td>0.0500-0.0999</td>
<td>0.0500-0.0999</td>
<td>8.000-15.999</td>
</tr>
<tr>
<td>7</td>
<td>0.120-0.139</td>
<td>0.1000-0.1499</td>
<td>0.1000-0.2999</td>
<td>16.000-30.999</td>
</tr>
<tr>
<td>8</td>
<td>0.140-0.169</td>
<td>0.1500-0.2599</td>
<td>0.3000-0.4999</td>
<td>31.000-39.999</td>
</tr>
<tr>
<td>9</td>
<td>0.170-0.189</td>
<td>0.2600-0.7999</td>
<td>0.5000-0.7999</td>
<td>40.000-49.999</td>
</tr>
<tr>
<td>10</td>
<td>0.190-0.259</td>
<td>0.8000-1.000</td>
<td>0.8000-1.000</td>
<td>50.000-60.000</td>
</tr>
</tbody>
</table>

- Regular analyzers: 3 **yellow** levels
- Trace analyzers: 2 **pink** & 2 **yellow** levels
Standards

- Must be certified and traceable
  - EPA QA Vol II, Section 12.1.2
  - CARB Standards Lab: http://www.arb.ca.gov/aaqm/qa/stdslab/stdslab.htm

- Check expiration dates:
  - Cal Gas Tanks
  - Mass Flow Controllers
  - Ozone transfer standard (6 months)
  - Flow Standards (1 year)
  - etc

- Properly warm-up/equilibrated:
  - Flow Standards
  - Ozone transfer standard
  - etc
Zero Air

- Zero Air Generator is part of calibration system
- Per EPA’s TSA of ARB, we should:
  - Verify that our zero is clean
  - Document ZAG maintenance

- In depth presentations on this topic:
  - Wednesday, 10:35 – 11:00
    Yousef Hameed
  - Wednesday, 1:45 & 3:30, Mountain Vista 2
    David Roque
    Zero Air Generator Certification.
Response Plateauing

- General practice is to allow concentrations to stabilize (plateau) for at least 10–15 minutes
- Teledyne API Analyzers:
  Stability should be <0.5 ppb (non-trace level)
Documentation

- Usual stuff: name, data, initials/signature
- Information for standard(s):
  - Serial number(s)
  - Calibration/certification date(s)
  - Concentrations, correction factors, etc.
- Information for instrument:
  - Serial number
  - As-Is and Final slope/intercept/correction factor
- Calibration info:
  - Calibration points
  - Instrument response
Criteria for PASS/FAIL

- **EPA** says (QA Vol II sec 12.2 & App D; 40 CFR 50 Apps A, C, D or F):
  - All points within 2% of calibration scale
  - For NO$_2$: NO$_x$ convertor efficiency > 96%

- **ARB** says (SOPs):
  - “Overall % Accuracy”: 100% * ($\sum$true$_i$ – $\sum$resp$_i$) / $\sum$true$_i$
    - Ozone: <2%
    - NO/NO$_2$/No$_x$: <5%, convertor efficiency > 96%

- Check your SOPs and QAPPs!!
Failed Calibration?

- Whatever happens: Document Document Document!!
- Double check calibration equipment, data logger, and calculations
- Corrective Action:
  - Rezero and/or Respan
  - Fix or replace instrument
  - Perform calibration/verification prior to placing monitor back online.
- Inform upstream data users
- Effect on Data: Depends
  - Note: “post-processing” of data no longer accepted
Questions?
Proper use of Standards

- Response plateauing
- Purging regulators
- Warm-up period
NO$_2$ Example

4 NO/NO$_x$ points

3 NO$_2$ points

15 min

1 zero

Time →
Purging a Regulator
Z/S/P with Properly Purged Reg

[Graph showing NO, NO₂, NO_x concentrations over time]
Purging a Regulator
Autocal with Improperly Purged Reg
Calibration: Purging a Regulator Demo
Purging a Regulator

1. Attach the regulator to the cylinder valve.
2. Open the cylinder valve to flow gas into the regulator, then immediately close the cylinder valve.
3. Increase the regulator outlet pressure several PSI.
4. Then open the regulator outlet valve to vent the gas trapped in the regulator.
5. Close the regulator outlet valve.
6. Repeat steps 2 through 5 (4–5 times).

- **CAUTION:** Many gas mixtures contain harmful or toxic substances. Suitable vents must be used to avoid breathing these substances.