Calibration of Aerosol Electrometers for PMP

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In this paper we discuss the traceable calibration of a commercial Aerosol Electrometer- a key component in the primary calibration of Condensation Particle Counters (CPC) to meet Particle Measurement Programme (PMP) specifications. Calibration of the Aerosol Electrometer is essential for ensuring traceability and comparability between measurements. We focus on the electrical performance of the instrument.

The electrometer current is dictated by the flow rate and number concentration of the sample. For a CPC calibration, where the particle flux may be switched on and off, a current difference of about 3 fA must be reliably measured. The electrometer used (Grimm 5.705) consists of a primary amplifier followed by three secondary amplifiers. Its performance was evaluated using both the manufacturer’s control electronics (Grimm 5.170) and independently using a multi-channel Digital Voltmeter (Keithley 2700).

Experimental
Known currents between +1000 and –1000 fA were injected into the electrometer using a current source, which had been traceably calibrated against the NPL reference low current facility. Calibrations were carried out every six months, and additional experiments conducted on single occasions.

Results
An example calibration (figure part a) shows a good best fit with slope about -0.96. Close examination of the differences between the two measurements shows an offset on points below ~400 fA (figure part b), which is likely to be associated with a switch between gain modes.

The calibration factors determined from several calibrations (See table) show no obvious instrument ageing or significant drift. This is an important outcome for validating CPC calibration results in the medium and long term.
A similar calibration was carried out using the alternative control electronics. Good agreement was seen between the different gain channels but the slopes were ~5% different from that obtained using the manufacturer’s electronics. The intercept values changed consistently with previous results. The magnitude of the offset is 3 fA and would have a worst case effect of the order 1% when measuring at the 400 fA level, far beyond the scope of PMP calibrations. However, these artifacts demonstrate the need to calibrate entire instruments as ‘black boxes’ instead of just key components within them.

To investigate the linearity of the electrometer over the required 3 fA current change, a constant current ramp of 1 fA/s was applied. By comparing many pairs of data points collected with a time separation of 3 seconds, the co-linearity of the current source and electrometer may be determined. The mean current change was 2.86 fA. Inspection of time series showed no indication of deviation from linear behavior, except at the 400 fA gain change point.

Finally, the electrometer noise was evaluated with all shielding intact. A 30 minute time series yielded a mean current of –0.213 fA with standard deviation 0.145 fA. Therefore, the expected electrometer noise for an ideal current difference measurement is 0.205 fA, some 15 times smaller than the 3 fA target.

<table>
<thead>
<tr>
<th>Elapsed Time (approx)</th>
<th>Calibration Factor</th>
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<tbody>
<tr>
<td>--</td>
<td>-0.964</td>
</tr>
<tr>
<td>6 months</td>
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Calibration of aerosol electrometers for PMP

Paul Quincey, Richard Gilham
ETH Conference on Combustion Generated Nanoparticles
3 August 2010
Talk outline

1  NPL background
2  Calibrating CPCs with electrometers
3  Results from an evaluation of aerosol electrometer calibration
<table>
<thead>
<tr>
<th>Year</th>
<th>Event</th>
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<tbody>
<tr>
<td>1995</td>
<td>QA/QC of UK network PM$_{10}$ (TEOMs)</td>
</tr>
<tr>
<td>1998</td>
<td>CEN PM$_{2.5}$ WG 15 starts</td>
</tr>
<tr>
<td>2002</td>
<td>Filter conditioning and weighing facility</td>
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</tbody>
</table>
| 2003 | CEN trial of 20 PM instruments  
Particle metals analysis by ICP-MS |
| 2004 | UK metals network won  
UK PM$_{10}$ equivalence trial started at NPL |
| 2005 | UK particle network (ambient CPC and SMPS) won  
Nanoparticle and PM speciation work started |
# NPL Particle History

<table>
<thead>
<tr>
<th>Year</th>
<th>Activities</th>
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| 2006 | Second UK PM$_{10}$ equivalence trial at NPL  
Participation in ISO 24 SC 4 (particle counting and sizing) |
| 2007 | Routine OC/EC and anion analysis for UK network  
Move to purpose built laboratory  
Role in PMP (vehicle emission) programme  
UK Black Smoke network won |
| 2008 - | Participation in EUSAAR (European atmospheric aerosol group)  
CPC calibration service  
EURAMET comparison  
Joint PM equivalence trials with TUV  
CEN WG 32 (number/size),  
WG 34 (anions), WG 35 (EC/OC)  
Robot filter weigher … |
Calibration of CPCs

Measurement units: (particles) cm$^{-3}$

Traceability required for:

- flow (+ time)
- particle detection efficiency
Calibration of CPCs

Measurement units: (particles) cm$^{-3}$

Traceability required for:
- flow (+ time)
- particle detection efficiency
  - as a function of particle size
Calibration of CPCs

Measurement units: (particles) cm$^{-3}$

Traceability required for:

- flow (+ time)
- particle detection efficiency
  - as a function of particle size
  - as a function of particle concentration
Calibration of CPCs

Measurement units: (particles) cm$^{-3}$

Traceability required for:

- flow (+ time)
- particle detection efficiency
  - as a function of particle size
  - as a function of particle concentration
  - as a function of particle material / type

ISO NWI 27891 Aerosol particle number concentration – calibration of condensation particle counters
CPC calibration procedures

PMP procedures and ISO NWI 27891 describe comparison with an aerosol electrometer. Measures charge concentration: C cm\(^{-3}\)

Key parameters: Current (0 – ~1000 fA) Flow Particle collection efficiency

*The mean charge per particle must be known by other mechanisms.*
CPC calibration procedures

PMP procedures and ISO NWI 27891 describe comparison with an aerosol electrometer. Measures charge concentration: C cm$^{-3}$

Key parameters:
- Current (0 – ~1000 fA)
- Flow
- Particle collection efficiency

The mean charge per particle must be known by other mechanisms.
Aerosol Electrometer- Current calibration

Grimm model 5.705

Main amplifier + 3 gain stages:
Maximum 4000 fA, 400 fA, 40 fA

Electrometer output monitored by Grimm software (5.170) and independent DVM (Keithley 2700)
Aerosol Electrometer- Current calibration

NPL Reference Low Current System

Transfer Standard (Keithley 6430)

Aerosol Electrometer- Direct Current Injection

Reference current applied:
-1000 fA to 1000 fA

Aerosol Electrometer- Current calibration

\[ y = -0.9617x \]

\[ R^2 = 0.9998 \]
Effect of amplifier gain change
## Electrometer / calibration system stability

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Use of independent DVM

3 gain channels “raw” signals monitored simultaneously:

<table>
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<tr>
<th>Gain channel</th>
<th>1</th>
<th>2</th>
<th>3</th>
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<tbody>
<tr>
<td>Slope</td>
<td>-1.014</td>
<td>-1.012</td>
<td>-1.016</td>
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<tr>
<td>Offset / fA</td>
<td>+0.293</td>
<td>-2.610</td>
<td>-2.714</td>
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NB compare
-0.96

NB offset change
Small scale linearity

Current source set to sweep repeatedly between
–1000 and 1000 fA at 1 fA/s
Differences at 1s intervals logged.
Differences at 1s and 3s intervals collated.
Small scale non-linearity would be expected to show as broadening of the “constant” difference.

– but this will be combined with other noise.
Small scale linearity

1 second intervals

3 second intervals

Interpreted as random noise

SD ~ 1 fA
Realistic noise – 30 minute constant zero source, shielding replaced

Mean –0.213 fA
SD 0.145 fA
Summary

Calibration of CPCs needs forethought about the required uses, hopefully helped by standardised procedures.

Calibration of aerosol electrometers also needs forethought, especially for high accuracy – beware amplifier gain changes and hidden software features.

Procedures and facilities for calibrating aerosol electrometers exist.

Other sources of uncertainty are usually more important.