Traceability Issue in PM$_{2.5}$ and PM$_{10}$

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Traceability Issue in PM$_{2.5}$ and PM$_{10}$ Measurements

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Abstract: Nowadays particle size and mass concentration measurements are the important parameter of the ambient air quality standards of several countries. The regulatory limits of mass concentration of particulate matter (PM) for the size classes of PM$_{2.5}$ and PM$_{10}$, i.e., particle sizes of less than or equal to 2.5 and 10 μm in aerodynamic diameter, respectively in air are defined on yearly and hourly time-weighted-average basis. However, these limits are different in different regulations of the countries. Both of the parameters relate with the human health, climate and other issues, therefore accurate and precise measurement of these parameters are very important. Despite this, so far not much work has progressed in national metrology institutes (NMIs) worldwide on calibration and traceability issue of PM measurements. In this paper in context of PM measurement traceability, we present systematically the (1) air quality regulation in different countries, (2) reference methods for size and mass measurements, (3) variation/error and limitations of PM measurements based on the current results in this study and previously published results, (4) current status of PM size and mass calibration facility, (5) expected uncertainty in PM measurements, (6) add-on uncertainty in other parameters of national ambient air quality standards due to PM measurements, (7) where does traceability of PM issue stand against other parameters of air quality standards and its impact on health and climate, (8) NMIs working on this issue, (9) status at Bureau International des Poids et Mesures (BIPM), France and (10) conclusion. The aim of this paper is to better understand the importance of international system of units (SI) traceability issue in PM measurements, so wherever and whenever it is measured, should be acceptable everywhere, and data should be comparable for improving air quality and thus the quality of life. Funding agencies should be aware of this issue, and accept the results from the principle investigators and team only when their results have the traceability link to SI. NMIs should make program to involve industries in gas and aerosol metrology work to fulfill the requirement of calibration and standards. The regulatory authorities/ministry should work together with NMIs to improve the data quality of ambient measurements. This will greatly help to better make the policies and decisions on the related impacts. These were also the ultimate goals of “one-day pre-AdMet workshop” organized at National Physical Laboratory, New Delhi, India on February 20th, 2013.
Focus…

1. Air quality regulation in different countries,
2. Reference methods for size and mass measurements,
3. Variation/error and limitations of PM measurements based on the current results in this study and previously published results,
4. Current status of PM size and mass calibration facility,
5. Expected uncertainty in PM measurements,
6. Add-on uncertainty in other parameters of national ambient air quality standards due to PM measurements,
7. Where does traceability of PM issue stand against other parameters of air quality standards and its impact on health and climate,
8. NMIs working on this issue,
9. Status at Bureau International des Poids et Mesures (BIPM), France,
10. Conclusion
Atmospheric aerosols...

Size: Few nanometers to >100 µm
Primary and secondary aerosols

Major concerns:
• Health effect
• Climate effect
Aerosol effects: Climate effects…

Aerosol characterization

Physical
- Size, Shape
- CCN
- Hygroscopicity

Optical
- Scattering
- Absorption

Chemical
- At bulk level
- At molecular level

Aerosol radiative forcing...
(Direct and indirect effects, cooling or warming)

Atmospheric Particle Mass and Size Distribution
Health effects...

Particle Transport in the Respiratory Tract

- Particle characteristics
- Geometry of the respiratory tract
- Fluid dynamics

Particle density: 1 g cm⁻³
Respiratory flow rate: 300 cm³ s⁻¹
Breathing cycle period: 5 s
<table>
<thead>
<tr>
<th>Countries</th>
<th>Criteria pollutants</th>
<th>PM$_{10}$ (µg m$^{-3}$)</th>
<th>PM$_{2.5}$ (µg m$^{-3}$)</th>
<th>Pb (µg m$^{-3}$)</th>
<th>As (µg m$^{-3}$)</th>
<th>Ni (µg m$^{-3}$)</th>
<th>BaP (µg m$^{-3}$)</th>
<th>O$_3$ (ppm)</th>
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<td>Ecologically Sensitive Area (notified by Central Government)</td>
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<td>24 hours**</td>
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<td>5</td>
<td>Ozone (O₃) μg/m³</td>
<td>8 hours**</td>
<td>100</td>
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<td>- UV photometric - Chemiluminescence - Chemical Method</td>
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<td>1 hour**</td>
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<td>6</td>
<td>Lead (Pb) μg/m³</td>
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<td>AAS/ICP method after sampling on EPM 2000 or equivalent filter paper - ED-XRF using Teflon filter</td>
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<td>Carbon Monoxide (CO) mg/m³</td>
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http://cpcb.nic.in/National_Ambient_Air_Quality_Standards.php
NAAQS: Particle mass measurement:

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<th></th>
<th>Particulate Matter (size less than 10(\mu)m) or PM(_{10})(\mu)g/m(^3)</th>
<th>Annual*</th>
<th>24 hours**</th>
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</table>

- Gravimetric
- TOEM
- Beta attenuation

PM\(_{10}\) Sampling Inlet

Flow splitter

By-pass Flowline

Sensor Unit

In-line Filters

Flow Controllers

Vacuum Pump

Beta Attenuation

Tapered Element Oscillating Microbalance (TEOM)

PM\(_{10}/PM_{2.5}\) sampler
Comparison between all three techniques: PM\textsubscript{2.5} mass measurements

More than 30% differences in mass measurement were reported in these three techniques.

These differences are regarded because of:

1. difference in measurement principles, i.e. different calibration methods those may not always be traceable to SI.
2. for segregation of PM\textsubscript{2.5} (or PM\textsubscript{10}), an impactor or a cyclone is used at the inlet of the air flow to the sampler/s (filter/s). Hence calibration of these impactor and cyclone as well as precise control (calibration) of drawn air flow through the sampler are the major issues (cutoff size calibration).
Impactor theory combines the parameters for size segregation of particles called Stokes number:

\[ d_{50} = \left( \frac{9 \mu W St k_{50}}{p_{s} V_{50} C} \right)^{\frac{1}{2}} \]

\[ St = \frac{\rho d_{p}^{2} C V}{9 \mu W} \]

where:  
\( \rho \) = particle density  
\( d_{p} \) = particle Stokes diameter  
\( C \) = Cunningham slip factor, as defined in Equation 2  
\( V \) = mean velocity in the jet  
\( \mu \) = air viscosity  
\( W \) = jet diameter or width
Cyclone theory

Cutoff diameter derived by Lapple (1950):

\[ Dp_{50} = 3 \sqrt[3]{\frac{\mu b}{2\pi \rho_p U_i CN_t}} \]

where \( U_i \) is the gas velocity at the inlet; \( \mu \) is the air dynamic viscosity; \( \rho_p \) is the particle density; \( C \) is the slip correction factor of the particle corresponding to \( Dp_{50} \). The number of turns \( N_t \) can be calculated as \( N_t = \frac{tU_i}{\pi D} \) and the residence time \( t \) is equal to the volume of the cyclone divided by the volumetric flow rate, \( Q \).

\[ Dp_{50} = \frac{3}{U_{\text{max}}} \sqrt[3]{\frac{\mu Q}{\pi \rho_p C(H - S)}} \]

Iozia and Leith (1989):

\[ Dp_{50} = \frac{3}{U_{\text{max}}} \sqrt[3]{\frac{\mu Q}{\pi \chi_c \rho_p}} \]

\[ U_{\text{max}} = 6.1 \times U_i \times \left( \frac{ab}{D^2} \right)^{0.61} \times \left( \frac{De}{D} \right)^{-0.74} \times \left( \frac{H}{D} \right)^{-0.33} \]

and \( \chi_c \) is the length of the central core.
PM10 mass measurement results by three TEOM samplers (from a manufacturer).

Size/mass calibration is needed…

Comparison results of aerosol mass distribution measured using QCMs from different groups

Size distributions obtained from 5 QCMs in parallel under the same conditions

November 28, 2004 at NPL rooftop
Comparison of PM2.5 and PM10 mass

PM sampler (gravimetric technique) Vs. Dust monitor system (particle counting by scattering based technique)

RH = 72%  
RH = 87%
Comparison between all three techniques: possible causes of bias

- Techniques vs. environmental conditions
- Evaporation/volatilization losses, diffusion losses, charge effect
- Flow calibration vs. cutoff size calibration
- Balance calibration/sensitivity, Beta absorption calibration, frequency calibration (mass-frequency relationship)
Where does traceability of PM issue stand against other parameters of air quality standards and its impact on health and climate

- **Gravimetric measurement:** Yes
  (large uncertainty due to: water content, evaporation loss, volatilization, charge effect)

- **Cyclone/Impactor**
  - Air flow: Yes
  - Cutoff size: No

- **Beta Attenuation measurement:** not clear
  (large uncertainty due to: water content, compositional changes, evaporation loss, volatilization)

- **TOEM measurement:** not clear
  (large uncertainty due to: environmental condition changes, evaporation loss, volatilization)
Add-on uncertainty in other parameters of national ambient air quality standards due to PM measurements

**NAAQS: Chemical Analysis**

- **Metals: As, Ni, Pb**  
  (Using ICP-MS)

- **PAHs**  
  (Using GC-MS)
**Metal determination:**

As, Ni, Pb

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Pollutant</th>
<th>Time Weighted Average</th>
<th>Concentration in Ambient Air</th>
<th>Methods of Measurement</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Industrial, Residential, Rural and Other Area</td>
<td>Ecologically Sensitive Area (notified by Central Government)</td>
</tr>
<tr>
<td>6</td>
<td>Lead (Pb)</td>
<td>Annual*</td>
<td>0.50</td>
<td>0.50</td>
</tr>
<tr>
<td></td>
<td>µg/m³</td>
<td>24 hours**</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>11</td>
<td>Arsenic (As), ng/m³</td>
<td>Annual*</td>
<td>06</td>
<td>06</td>
</tr>
<tr>
<td>12</td>
<td>Nickel (Ni), ng/m³</td>
<td>Annual*</td>
<td>20</td>
<td>20</td>
</tr>
</tbody>
</table>

**Analysis:** Microwave digestion followed by ICP-MS analysis
# Chemical Analysis: Metals

<table>
<thead>
<tr>
<th>PM$<em>{2.5}$/PM$</em>{10}$ sampling</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz filter: Baked, conditioned and weighed</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Filter conditioning and weighing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Storing: -20 °C in glass bottle</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Take a filter cut in plastic/ Teflon vessel</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>Add HNO$_3$+H$_2$O$_2$+HF</th>
</tr>
</thead>
<tbody>
<tr>
<td>(5 ml, 3 ml, 1 ml)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Perform microwave/open digestion</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>Transfer the extract in a plastic beaker by washing (~50 ml)</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>Transfer the extract by filtering into a pre-weighed plastic bottle, and weight the sample solution finally</th>
</tr>
</thead>
</table>

- Why Quartz, pre-baked
- Why -20 °C in glass bottle
- Why not HCl
- Why HF
- Microwave/open digestion
- Use ultrapure (sub-boiled) acids
- Use elemental fee Milli-Q water
- Final solution → why gravimetric preparation
- Use SI traceable calibration solutions
Reality: Accuracy of different commercial calibration solutions (according to the measurements done by NMIs)

There is need to develop PM CRM
Uncertainty budget for the arsenic determination in aerosol samples using ICP-HRMS

Source of uncertainty | Distribution, Type A or B | Relative standard uncertainty | SC | DOF
--- | --- | --- | --- | ---
Calibration solution | Normal, Type B | 0.003 | 1 | ∞
Instrumental calibration (linearity) | Normal, Type A | 0.029 | 1 | 2
Repeatability | Normal, Type A | 0.005 0.005 0.021 0.015 0.001 0.005 0.008 0.002 0.007 0.003 0.000 | 1 | 2
Air volume | Rectangular, Type B | 0.029 | 1 | ∞
Sample digestion | Rectangular, Type A | 0.032 | 1 | 2

Combined uncertainty (ng/m³): 0.963 0.481 0.350 0.808 0.429 0.690 0.939 1.738 0.773 0.548 0.374


Expanded uncertainty (k=2): 1.926 0.961 0.699 1.617 0.857 1.379 1.878 3.477 1.545 1.096 0.748

Kumar, Aggarwal et al., 2014
Current status of aerosol metrology: Traceability issue in PM measurements
Development of aerosol wind tunnel and its application for evaluating the performance of ambient PM$_{10}$ inlets

Sangil Lee$^1$, Miae Yu$^1$, Hun H. Kim$^2$

$^1$ Center for Gas Analysis, Korea Research Institute of Standards and Science, Daejeon, Republic of Korea
$^2$ Center for Analytical Measurement Service, Korea Research Institute of Standards and Science, Daejeon, Republic of Korea
Progress in Development of Russian National Measurement Standards in the Field of Mass Concentration Measurement of Suspended Particles

Y. A. Kustikov* and B. I. Popov

Scientific Research Laboratory of the State Standards in the Field of Disperse Medium Parameters Measurements, D.I. Mendeleyev Institute for Metrology, Moskovskiy pr. 19, 190005 St. Petersburg, Russia

Structure chart of the transportable (mobile) working standard. 1 Generator, 1a compressor, 1b nebulizer, 2 cyclone, 3 dehumidifier, 4 column mixer, 5 dryer, 6 air blower, 7 power unit, 8 DUST1-E, 9 instrument under verification
MiC workshop on “Gas and aerosol metrology”, as pre-Admet-13 international conference, was organized during Feb.20, 2013 at NPL, New Delhi

About 80 delegates, including 6 invited speakers from other countries

Special issue on this workshop

http://link.springer.com/journal/12647/28/3/page/1
Our setup for counter’s calibration check…

We used scanning mobility particle sizer (SMPS) consists of a DMA (TSI 3081) and CPC (TSI 3788).
Our approach… (CPC derived volume vs. filter based volume)

This approach is based on the comparison of particle volumes derived from scanning mobility particle sizer (SMPS) with corresponding particle volume, which are collected on filter and determined by mass (gravimetric method).

Dried ammonium sulfate particles were introduced to SMPS and particles of size range 14 - 615 nm were segregated by DMA and number concentration was measured by CPC.
Conclusion... (1)

- Less work has been done so far for PM measurement traceability dissemination, especially for its size and mass related calibration.

- The inlets (size cutoff) of most of the commercially available PM samplers are not traceable to SI.

- Few NMIs are now taking initiatives for setting up the calibration facility of PM2.5 and PM10 impactors.

- Awareness programs on PM measurement and traceability issue should be organized more often.
Industries and manufacturers should participate in “traceability to SI through NMI program in ambient measurements” to improve the data quality which have significant impact on several issues, e.g. health, climate, societal, economy and other developments.

NMIs should make program to involve industries in gas and aerosol metrology work to fulfill the requirement of calibration and standards.

The regulatory authorities/ministry should work together with NMIs to improve the data quality of ambient measurements.

This will greatly help to better make the policies and decisions on the related impacts.
Thank you....!