Validation of Novel Light Scattering Sensors for Soot Opacity and Soot Mass

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Outline of presentation

➢ Legal bases and framework for soot opacity
  • In Europe and Germany

➢ Research project
  • Challenges and objectives
  • New method for measurement of soot opacity and soot mass concentration
  • Experimental setup for validation and special dilution system

➢ Results of research project
  • Correlation for high soot concentration
  • Correlation for low soot concentration

➢ Conclusion
**Periodical emission control of vehicles**

- Vehicles with type approval since 2006: On-Board-Diagnostic
- Older vehicles: regular - particle emission controlled via opacimeter

Measuring the opacity $N$ (in %) or the light absorption coefficient $k$ (m$^{-1}$)

\[
N = 1 - \frac{I}{I_0}
\]

\[
k = \frac{1}{L_A} \ln \left( \frac{I}{I_0} \right)
\]

Maximum limit for exhaust of modern engines: $k = 1.5$ (error limit ± 0.3)
Framework for vehicle inspection in Germany

- Type approval of opacimeters required verification ordinance 18.9 at PTB
- Traceability of reference opacimeter to the SI via optical filters that are calibrated at PTB
- Verification in the field via calibrated optical filters

Points of criticism since years:
- Insufficient resolution of type approved opacimeter
- \( k = 0.5 \pm 0.3 \) as lower limit is too high in contrast to the regular motor type approval (\( k < 0.3 \))
- No signal when measuring exhaust of modern diesel engines with particle filters
Research project with ASA\(^1\)-association

- Project duration: 3 years (finished in Nov. 2013)
- Goal:
  - Development of a metrological background for future type approvals of new soot sensors based on light scattering
- Challenges:
  - German verification act requires devices to measure the opacity and the light absorption coefficient
  - Indication on the screen of devices must be \(k\) in \(m^{-1}\) or \(N\) in \%
  - Verification method in the field should be similar to transmission filters (easy to use, traceable)

\(^1\) Bundesverband der Hersteller und Importeure von Automobil-Service Ausrüstungen e.V.
PTB – ASA Research cooperation

Objectives

➢ *Traceable and improved correlation between soot opacity and soot mass concentration are needed for the novel devices → experimental validation!*

➢ *Investigate if a general correlation between the soot opacity and the soot mass concentration*
  
  – for different particle sizes / particle number size distributions
  
  – for different light scattering sensor types
  
  – for improved uncertainty
PTB-ASA research cooperation
New method for particle measurement: light scattering

Basic physical concept:

- The Mie Theory for light scattering
  - Light is scattered by small particles
  - Scattered light intensity and angle distribution depends on the sensor angle between laser beam and detector
  - Detected signal of sensors depends on particle size, shape and number concentration

- Typically, higher sensitivity than opacimeter at low particle concentrations
Experimental Setup for Research Cooperation

Tasks:
- stable soot aerosol source about longer time scale
- accurate and precise characterization between reference systems
- stable and controlled aerosol size distribution to DUT

**Modified HighMass-CAST:**
- approx. 460 l/min
- $10^5$-$10^8$ part./cm³
- 1 mg/m³ to 500 mg/m³
- 70 to 220 nm, monomodal

**Closed loop-SMPS:**
- flow rate: 1 to 5
- size range: 8 to 880 nm

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Simulation of particle trajectories

A boundary layer and a developing of string jet could only be avoided by the sphere

→ Best results for aerosol mixture by a counter flow mixer, especially for the geometry of the sphere
Overview of parameter for intercomparison workshop

- Gravimetric soot mass \((m_1)\) via filter loading
- Online optical soot mass \((m_2)\) via MSS AVL 483
- Light absorption coefficient \((k)\) via reference opacimeter AVL 439 \((k \text{ in } \text{m}^{-1})\)
- Particle number size distribution via non-commercial SMPS \((\rightarrow \text{Mean Dp, nm})\)
- CVS tunnel parameter \((T, \text{flow})\)

<table>
<thead>
<tr>
<th>Number of participants and DUTs</th>
<th>Number of measured points</th>
<th>Analyzed data of intercomparison workshop</th>
</tr>
</thead>
<tbody>
<tr>
<td>6 (14)</td>
<td>117</td>
<td>(k, m_1, m_2, \text{Dp, CVS})</td>
</tr>
</tbody>
</table>

- Average means values of all parameters are predefined by gravimetrical filter loading time
- Only measurements for stationary conditions (no dynamical response check)
Results of intercomparison workshop
PTB internal traceable systems

- Very good correlation for reference systems could be observed about 3 orders of magnitude for \( k \) and \( m_1 \)

\[
y = 110.07x \\
R^2 = 0.9768
\]

117 data points

About stability of setup: see Poster Session 5: Instrumentation, no. 40: Margit Hildebrandt
Results of intercomparison workshop  
PTB internal systems

- Also very good correlation could be observed for gravimetric soot mass vs. optical soot mass (MSS 483)
- For correlation between optical soot mass and reference opacimeter are some discrepancy for the slope of the linear correlation

\[
y = 0.9869x \\
R^2 = 0.9641
\]

83 data points

Less data density for MSS, because of leakage in measuring cell for two days
Results of intercomparison workshop reference systems vs. new sensors for high soot loading

- Quite different deviation for DUTs could be observed, one device with very good correlation (master device?)

  \[
  y = 1.55313x, \ R^2 = 0.9283, \ N = 25(35) \\
  y = 1.37079x, \ R^2 = 0.8973, \ N = 25(35) \\
  y = 0.94335x, \ R^2 = 0.9901, \ N = 22(35)
  \]

- Higher variance of data points for \(m > 150 \mu g/m^3\) and \(k > 1.6 \text{ m}^{-1}\) stronger variance as well as very rare data points
Results of intercomparison workshop
reference systems vs. new sensors for low soot loading

- For data points for $m < 150 \, \mu g/m^3$ and $k < 1.6 \, m^{-1}$ could observed very good correlation, especially for $k$

For $m$ were observed different slopes for the linear correlation in different directions
Conclusion

- A new stable soot reference source was installed at PTB for a large range of soot mass concentration.
- The new light scattering sensors are a 100x more sensitive than commercial opacimeter on the market for vehicle inspection.
- In general, the correlations with respect to light absorption coefficient showed for low soot concentration ($k < 1.6 \, \text{m}^{-1}$) significantly better results than for high soot concentration ($k > 1.6 \, \text{m}^{-1}$).
- For $k < 1.6$ mostly very good correlation coefficient were determined between 0.98 to 0.99 for all devices.
- Just few sensors with good correlation for soot mass during these campaign, especially below 150 mg/m³.

→ So far, no universal calibration method according to verification ordinance 18.9 is available to validate the sensors during field measurements → no type approval.
Thanks for your attention
Counter flow mixer

Scale with filters

Gravimetical unit

Entire setup

17.07.2013
Outlook: Link to real soot data (MIRA report\textsuperscript{1})

Fitted linear correlation vs. measured data

- The linear fit of the MIRA report was used to calculate the MIRA soot mass based on the k-values of the intercomparison workshop → Implementation of conversion factor is needed

\[ y = 110.07x \]
\[ R^2 = 0.9768 \]

\[ y = 162.55x \]

\( R^2 = 0.9768 \)

\[ k, \text{ reference opacimeter} \]

\[ k, \text{ reference opacimeter AVL 439} [\text{m}^{-1}] \]

\textbf{Source:}
Outlook: Link to real soot data

Difference between diesel and propane soot

- TEM analyze showed more clustered structure for diesel soot than propane soot (longer aggregates) → Differences in density
- Effect on optical parameter like refraction index, primary particle sizes, fractional dimension
- Influence of relative humidity in the exhaust

→ Further research project is needed
Quantification of mixing process behind the mixing unit about $\Delta M = M_{\text{max}} - M_{\text{min}}$

→ Best results for gas mixture by a counter flow mixer including a sphere
Comparison between $k$-value of reference opacimeter (AVL 439) and mass concentration from gravimetrical mass and Gaussian fit parameter of PNSD for mode diameter.