

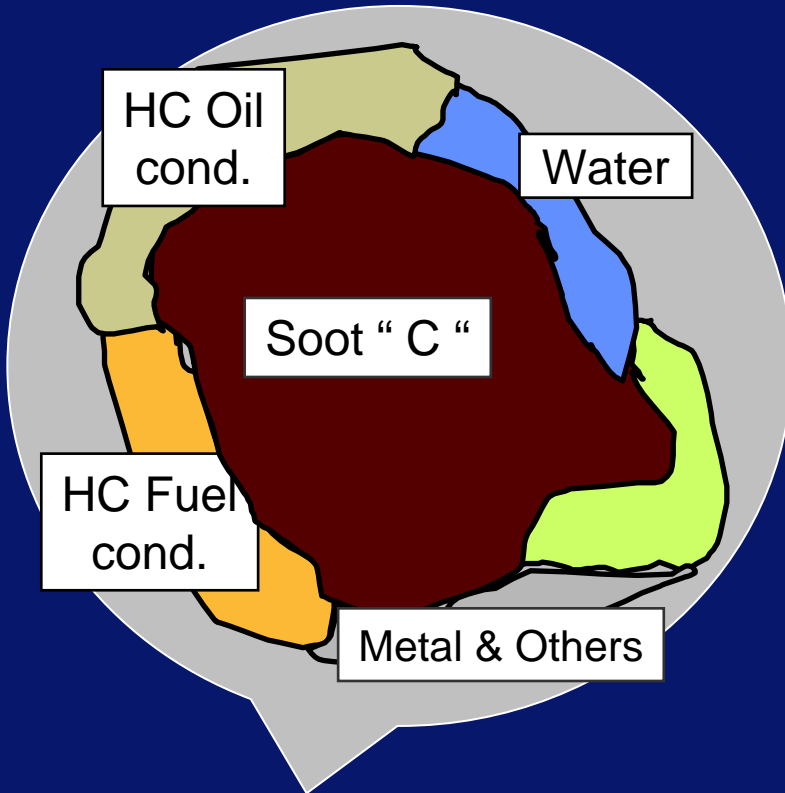


Notes on „Soot“ Measurement of Diesel Engines

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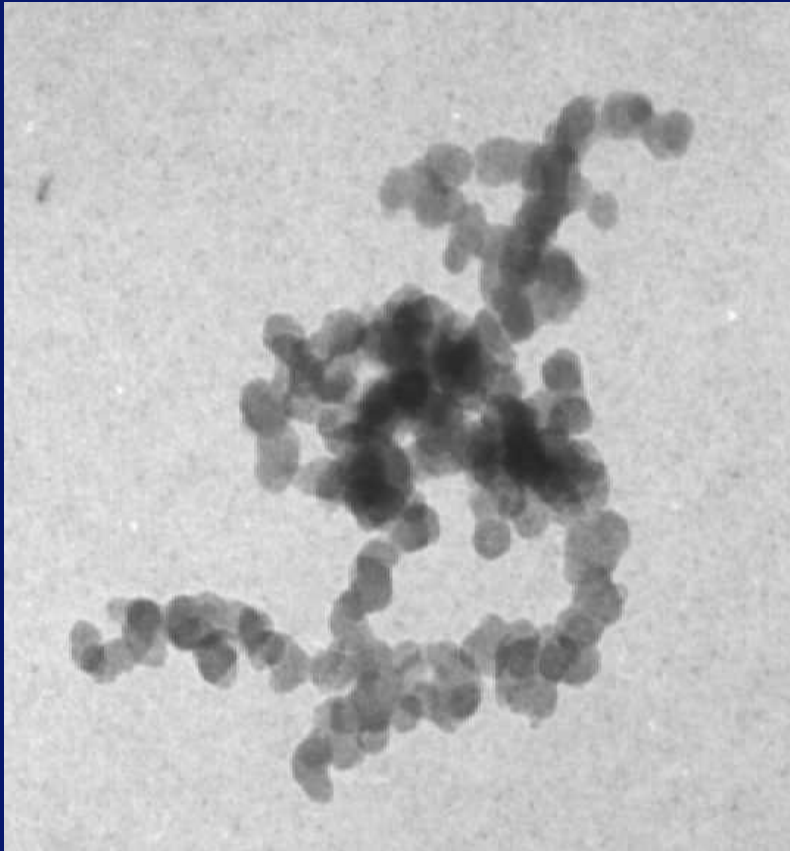
AVL List GmbH, Graz, Austria

Why bother about Soot Measurement ?



Gravimetric measurement of total particulate mass after partial-flow or full-flow dilution

- The measurement of “Soot” is a widely accepted method in Engine R&D since several decades.
- It provides information about the quality of the combustion process
- PMP has decided that agglomeration mode particles should be counted - Such particles typically consist of “soot” in Diesel exhaust
- There is some evidence that soot is more relevant with regard to health concerns than the homogeneous, volatile “Nanoparticles”



- Soot is a combustion product. (“Carbon black” is no “soot”).
- Soot consists of “Carbonaceous Particles”
- Soot has a combustion temperature in air of $>450\text{ }^{\circ}\text{C}$, it is not volatile or liquid. (compare the measuring proposal of PMP)
- The size is mainly in the “agglomeration mode” range, the structure irregular (fractal dimension 1.8 to 2.5)
- **Soot is “really black”**

■ Analysis of filtered Diesel Particulates

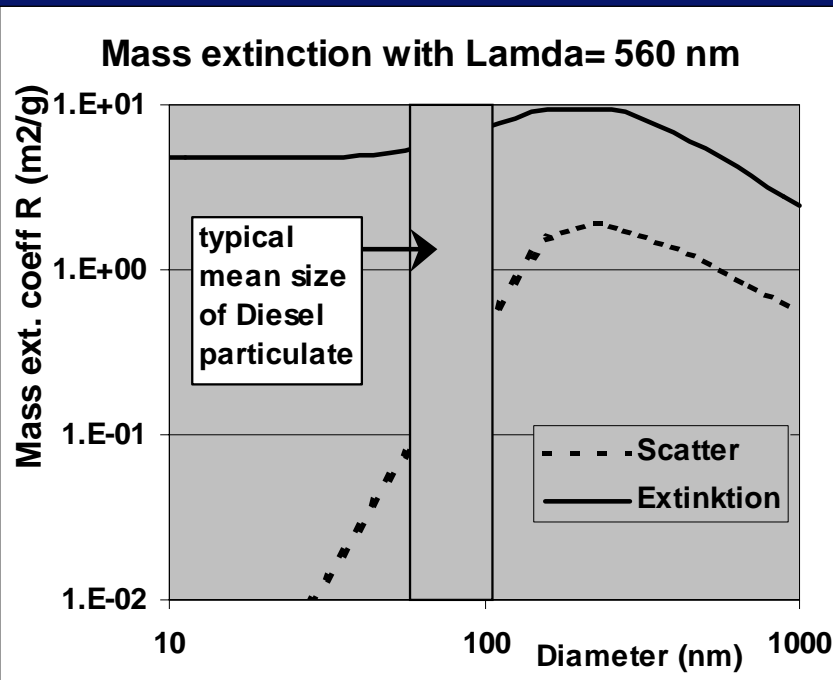
- chem. extraction
- thermogravimetry
- Coulometry

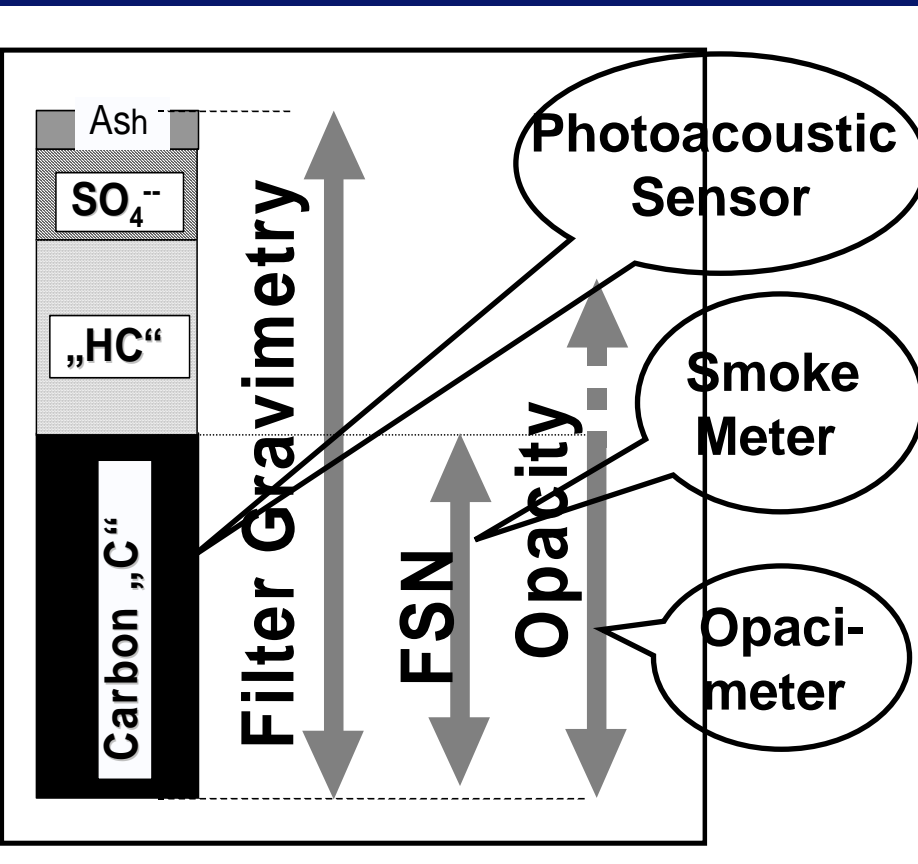
■ PMP method:

Thermodesorption and particle counting

■ Via its optical properties:

- The mass extinction coefficient of Soot is nearly constant for $D < 100$ nm
- The scattering coefficient goes with D/λ^{-4}



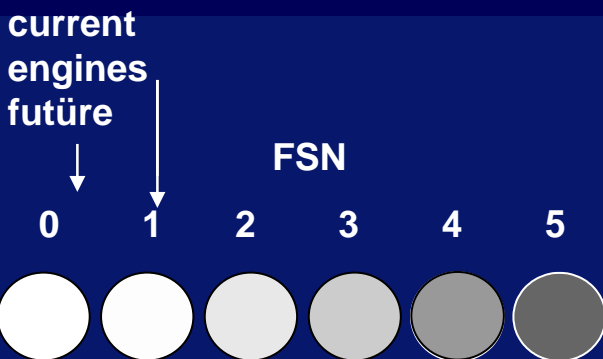


Criteria:

- accuracy - selectivity - measuring artefacts
- concentration:
 - resolution, detection limit
 - dynamic range
- repeatability, reproducibility
- time resolution

Target:

to assess the physical limits of various methods and the practical problems encountered when measuring Diesel Soot.



■ Advantages:

- Simple method, proven reliability, widely used in R&D
- Selectivity: for soot content >15%, FSN is clearly related to Soot concentration
- Detection limit: with high sampling volume $20 \mu\text{g}/\text{m}^3$ or 0,002 FSN achievable
⇒ dynamic range > 1: 10000 (concentration)
- Repeatability: 5% achievable (see below)

■ Challenges:

- Due to the simplicity of the method it is often used uncritically without observing the basic principles of particle measurement.
- A realistic theory of the relation between FSN and Soot concentration does not exist

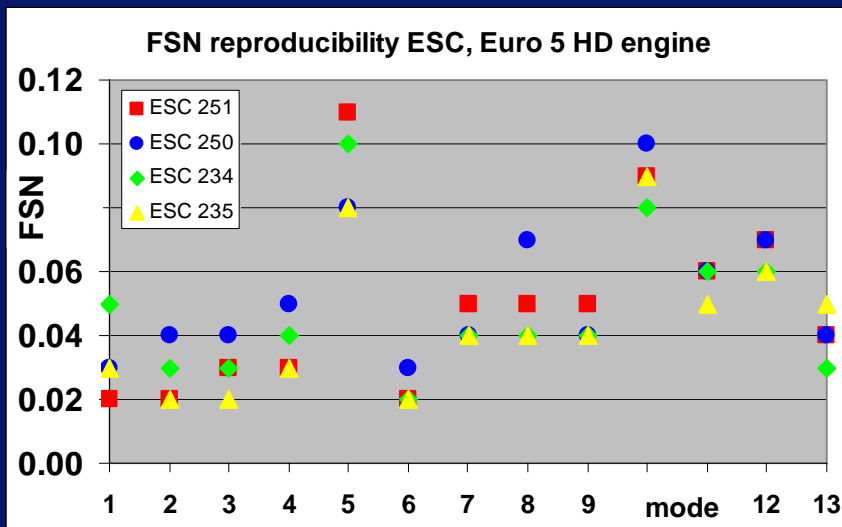
■ Drawback:

time resolution >10 sec by principle



Influences to FSN Measurement:

- Sample Conditioning. FSN is measured from the raw exhaust \Rightarrow concentrations are high, particle deposition is of considerable concern.
- Hangup after measurement of large FSN: purging is required to avoid re-entrainment during “white level determination” ($<2 \mu\text{g}$ allowed !)
- Filter paper: if two different filter papers, at opposite ends of the ISO 10054 specification band are used, results may vary by $>20\%$. (AVL makes a special paper check to guarantee $\pm 5\%$).
- Soot content: in aerosols with a very small soot core ($<15\%$ by mass) “the absorption coefficient is increased”



If measurements are carried out carefully, even the emissions of a Euro 5 HD engine can be measured reproducibly.



■ Advantages:

- Good time resolution, ≤ 0.1 sec
- Theoretically well defined (for fixed R)
- Detection limit: $k = 0.002 \text{ m}^{-1}$ corresponding to $\approx 300 \mu\text{g}/\text{m}^3$ (sufficient for non-trap engines)

■ Challenges:

- To achieve a stable “Zero” value I_0
- The mass extinction coefficient R of soot is slightly size dependent (typical uncertainty $\pm 20\%$)
- The influence of NO_2 on the signal is substantial, the influence of HC and SO_4 only at high concentrations

■ Drawback:

- At the current stage the technology is at its limits

Challenges for good Opacity Sensitivity

$$I/I_0 = e^{-k \cdot L} = (1 - N/100)$$

N [%]

Opacity

k [m⁻¹]

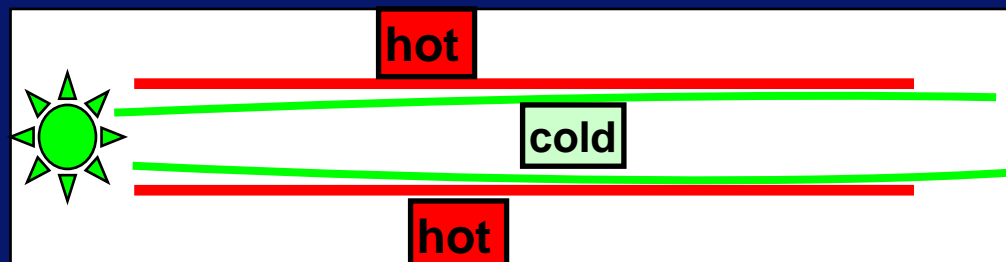
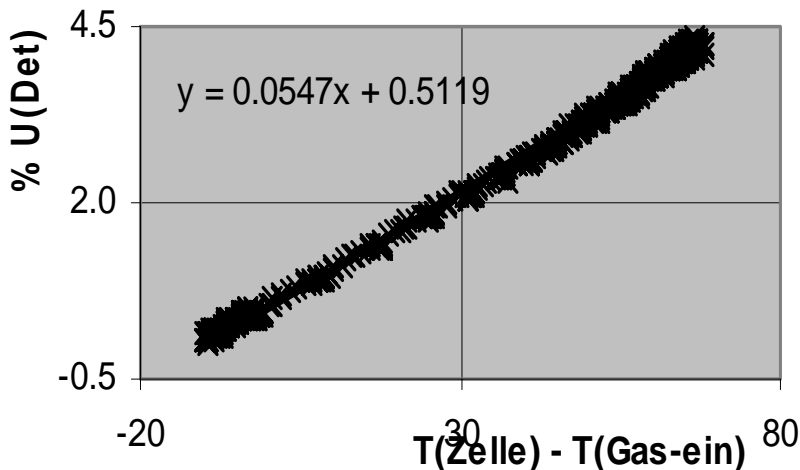
Absorption

Good resolution in k is achieved if:

- I₀ is stable
- The measuring length L is large

To some extent the two requirements contradict each other:

U(Det) Änderung mit Gas-ein T



If the temperature of gas and chamber walls are not stable, the **thermal lens** („mirage effect“) changes I₀

Temperature changes of 1.5 °C result in an I₀ change of 0.1%

“Accuracy” - is Opacity related to Soot?

In other words: is
 $k (m^{-1}) = R \cdot \text{Conz} (mg/m^3)$
with konstant R?

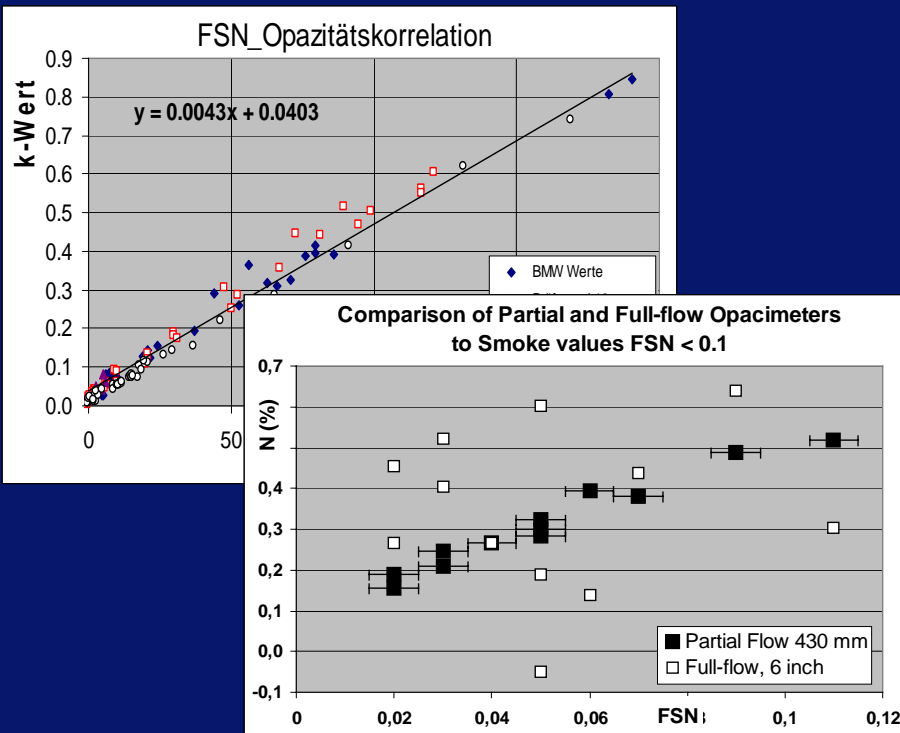
Quantities influencing R:

- Particulate size.
- Particulate composition: (HC and SO_4)

Practical experience:

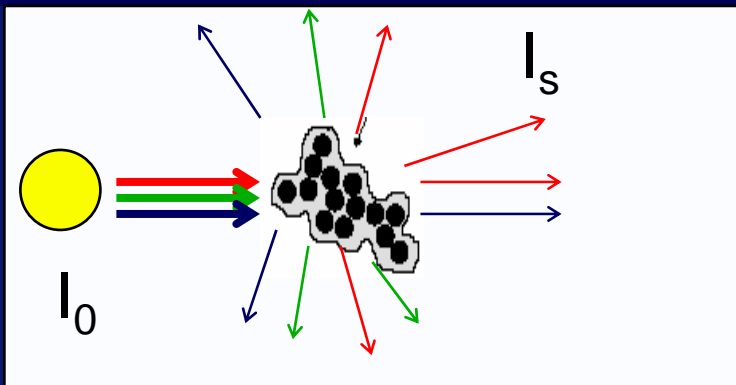
A constant ($\pm 20\%$) relation between Opacity and Soot is given if:

- NO_2 concentration is $< 100ppm$
- The particulates consist to less than 50% of HC and SO_4
- The exhaust temperature in the measuring chamber is $\geq 100^\circ C$
- The particle size is in the “typical” Diesel exhaust accumulation mode range



The Particulate size dependence of R decreases with increasing λ
-> IR extinction (drawback: R decrease with increasing wavelength)

How about light Scattering?



$$I_s = Fkt(\vartheta, \varphi, d/\lambda, n, ff)$$

$$\text{with } n = m+ik,$$

ff = form faktor

$$I_s \propto d^{-6}$$

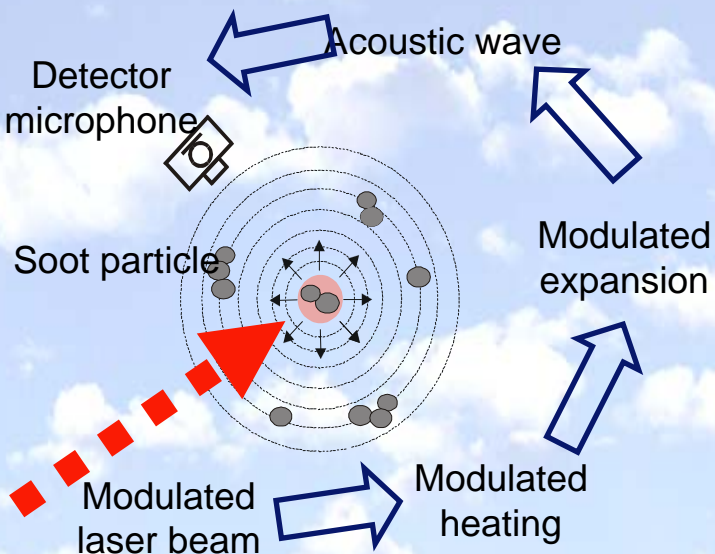
In Principle: The measurement of I_s at different ϑ , φ and at different λ can yield a wealth of information about size, size distribution, number, shape.....

- In Practice:**
- Single particle scattering: with reasonable effort the lower size limit is ≈ 100 nm due to low scattering intensity.
 - Multi particle scattering: large particles dominate the Signal.
 - Multi-wavelength and -angle scattering: the addition of measuring uncertainties introduces ambiguities into the data evaluation.

⇒ A robust and accepted light scattering instrument has not made its way to the market despite substantial and repeated effort in the last 20 years



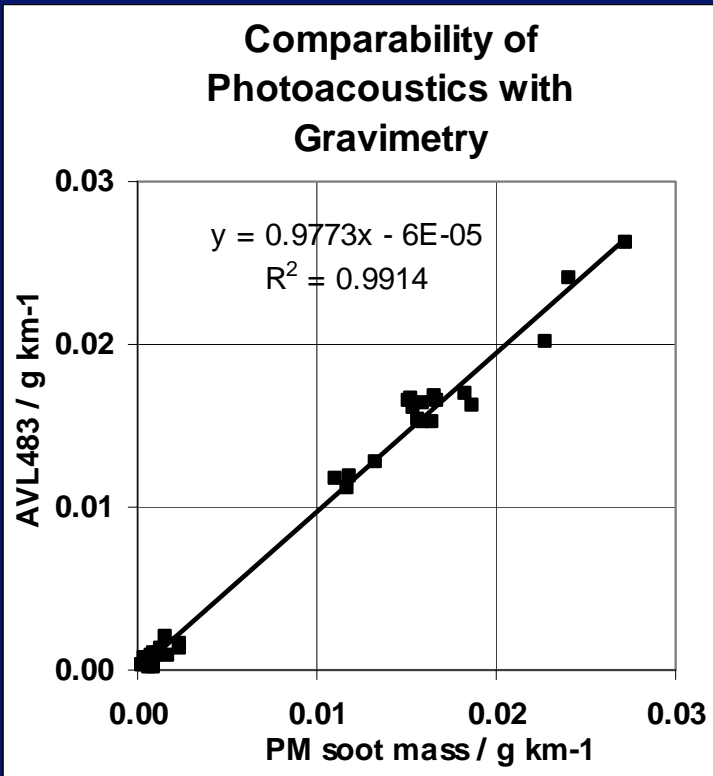
- **Periodic heating by a modulated laser beam and subsequent cooling generates an acoustic signal that is detected by a microphone**
- **Advantages:**
 - Sensor is “directly” sensitive to soot concentration
 - high sensitivity (detection limit $< 10 \mu\text{g}/\text{m}^3$)
- **Challenges:**
 - signal evaluation at low concentrations
 - repeatability / reproducibility / calibration
 - currently for diluted exhaust only



Accuracy: Selectivity, Linearity and Comparability

- **Selectivity:**

No cross-sensitivity from typical or untypical exhaust gases, if the proper near IR Laser wavelength is used (H₂O, HC, SO₄, NO₂.....).



- **Linearity:**

Due to the physical principle the signal is linear with soot concentration

- **Comparability:**

According to experience in various laboratories the signal is comparable to the soot determined with gravimetric soot mass determination

Courtesy V. Scheer, FFA



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Reproducibility, Repeatability, Calibration

- **Reproducibility:**

The near IR frequency of the laser used guarantees, that D/λ is small for typical Diesel particulates.

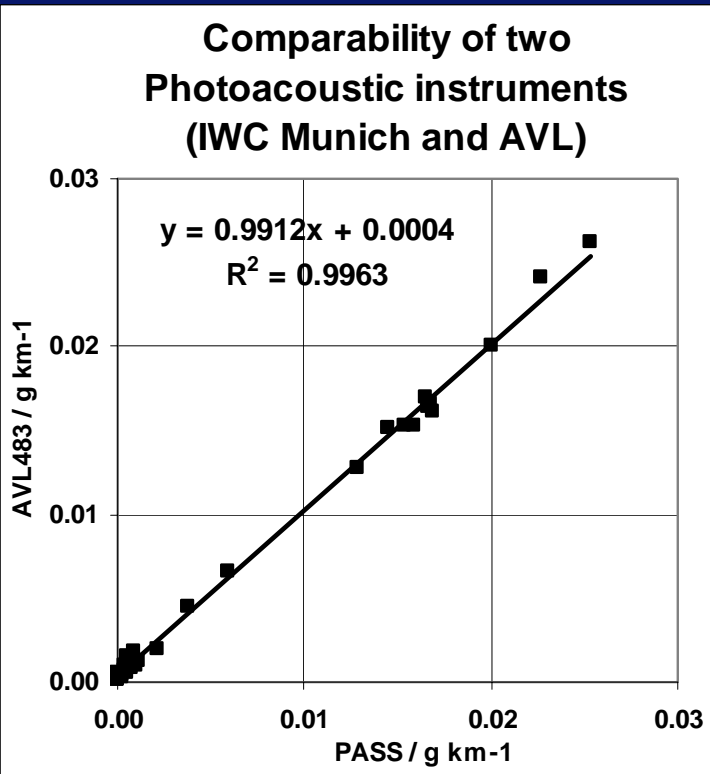
Therefore the mass extinction coefficient R is nearly constant

- **Repeatability:**

The experiments show that two photoacoustic systems of different build and different calibration methods show equal results

- **Calibration:**

Calibration must be performed in comparison to gravimetric methods. However, the calibration factor in the „PASS“ has not been changed in the last 2 years, and yields the same results as the AVL Micro Soot Sensor



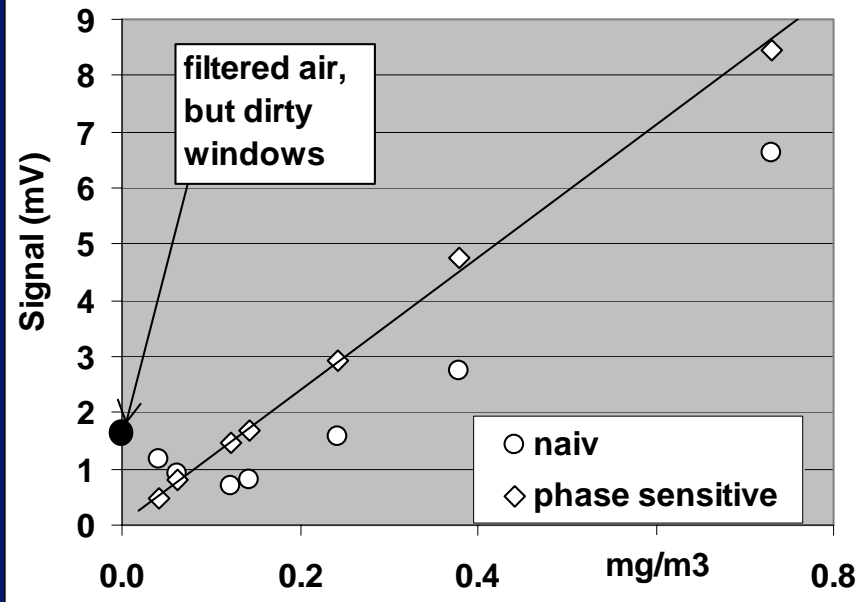
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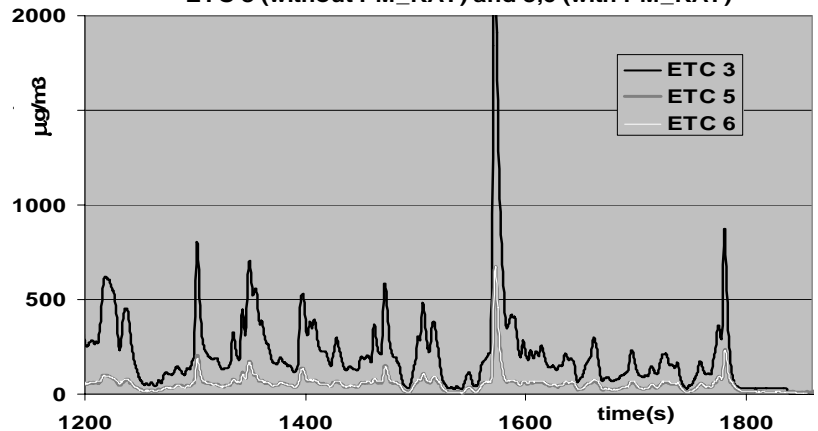
Resolution (concentration, time)

Photoacoustic Signal Evaluation

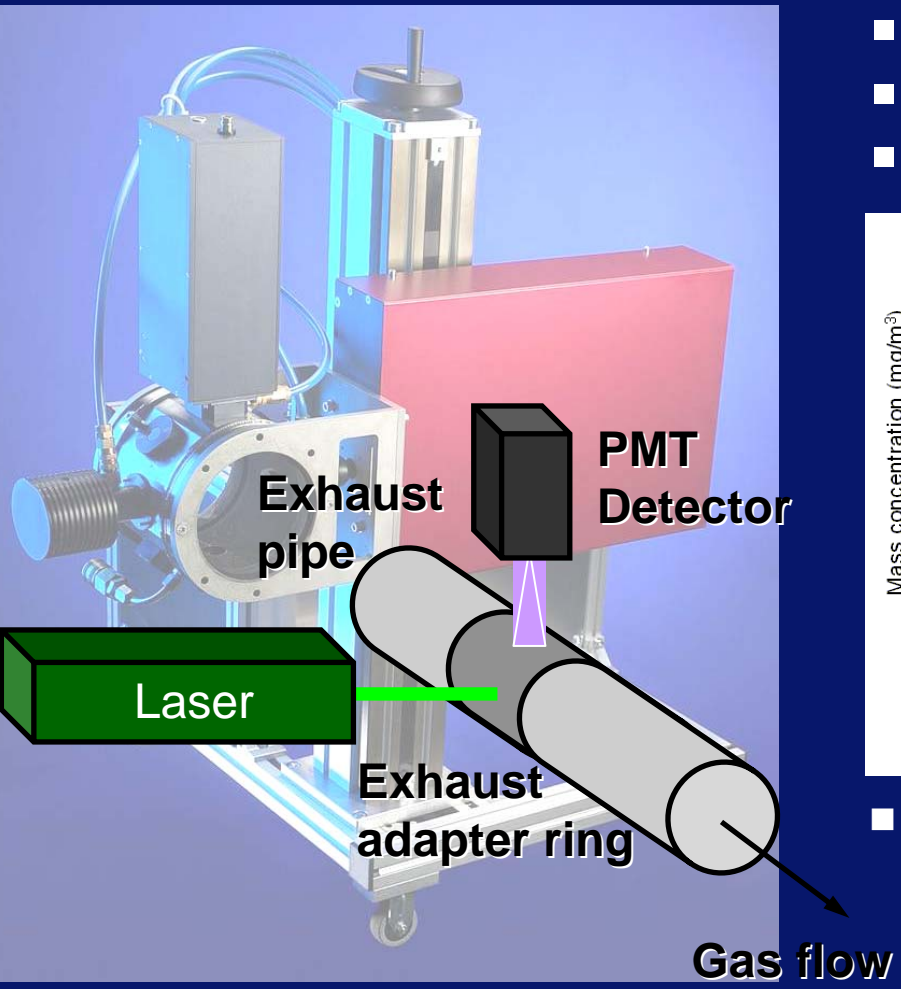


- **Resolution:**
Challenges: Good resolution (below 10 $\mu\text{g}/\text{m}^3$) can be obtained by:
 - Signal evaluation with high frequency selectivity
 - Low noise in the gas path
 - Correct signal evaluation, subtracting the baseline signal from sooted windows phase-corrected
- **Dynamic range:**
Photoacoustics allows a dynamic range of 1: 10000 (important to detect peaks on low background)
- **Time resolution:**
1 sec τ_{90} is easily achievable, better time resolution currently sacrifices concentration detectivity

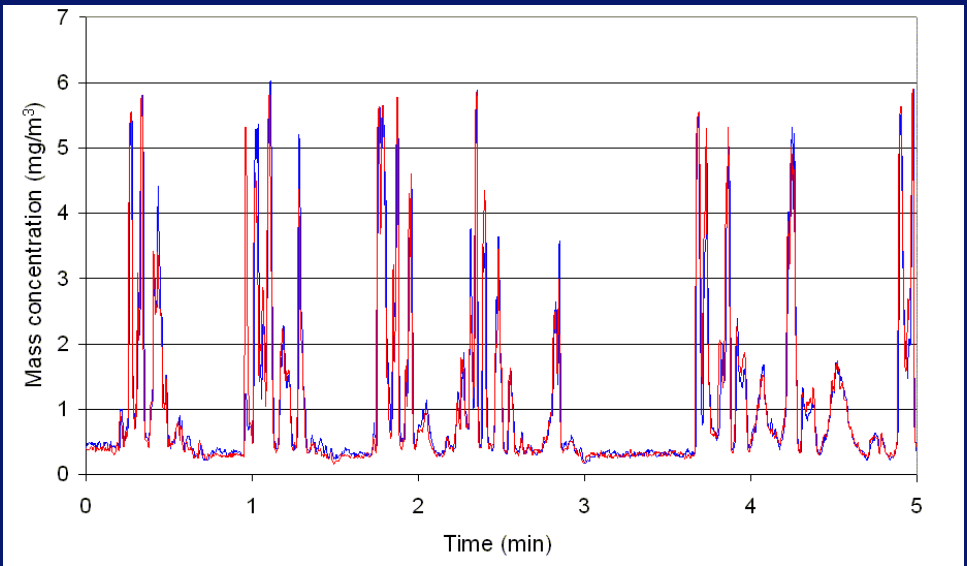
ETC 3 (without PM_KAT) and 5,6 (with PM_KAT)



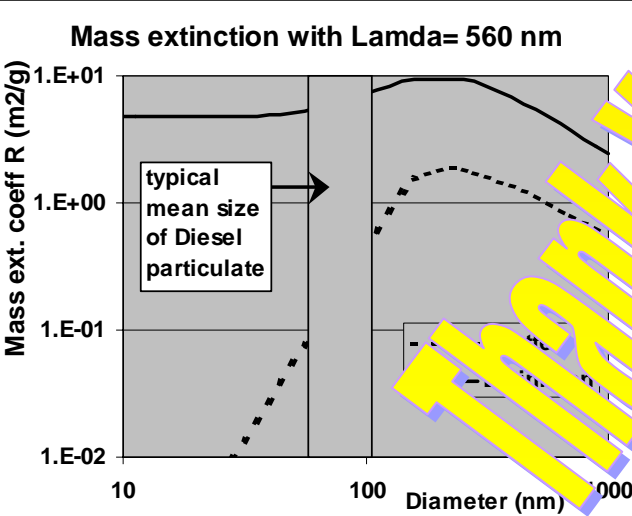
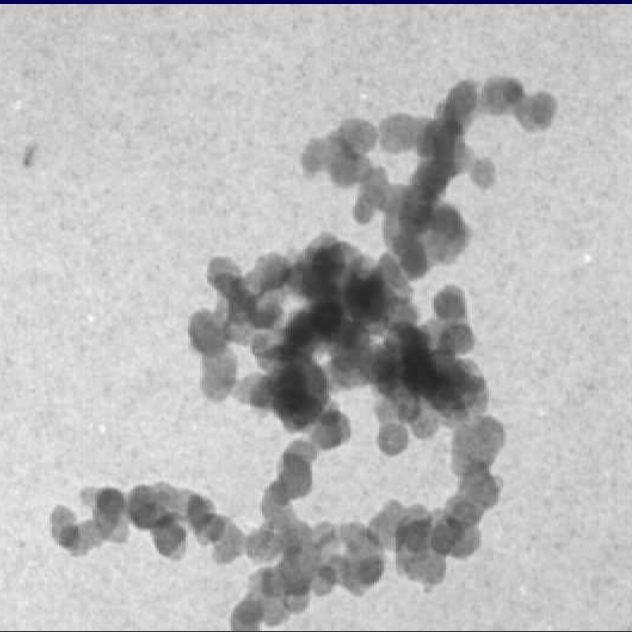
Laser Induced Incandescence (LII)



- High sensitivity for ultra-low emission
- good time resolution for transient testing
- In-situ raw exhaust gas measurement



- However:
 - substantial costs, due to expensive laser
 - serious laser hazards



- Several Methods exist to measure Soot on the basis of its strong absorption coefficient
- If these methods are used with attention and some basic knowledge about particulate measurement they can give accurate soot data
 - even for very low soot concentration in the exhaust
- The method proposed by PMP for measuring particles seems feasible - the interlaboratory exercise has not yet started - but practically it measures only the soot fraction of the Diesel exhaust
- A second thought on mass-proportional soot measurement methods as a viable alternative seems appropriate.

THANK YOU FOR YOUR ATTENTION