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# Inter-laboratory comparison with mobility particle sizers

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# Interlaboratory Comparison with Mobility Particle Sizers

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As you can see in the announcement of this contribution our company changed its name. Now the institute is called METAS, which stands for Metrology and Accreditation Switzerland.

This contribution presents the results from a comparison carried out during the last September in Dortmund at the “Institut für Gefahrstoff-Forschung der Bergbau Berufsgenossenschaft” IGF.

At this place I like to thank the organiser of this comparison, Mr. Dahmann and his team.

# Objectives

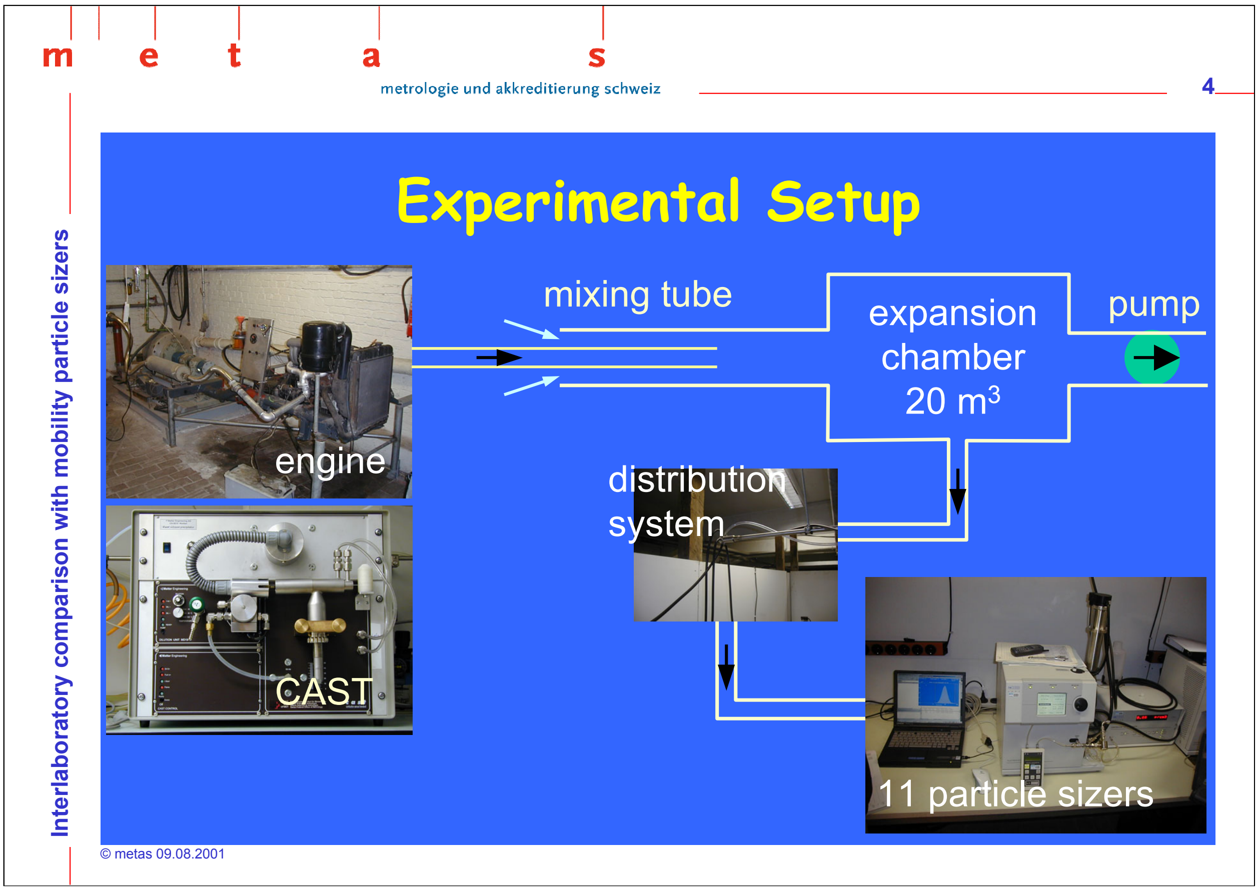
comparison of mobility particle sizers (SMPS):

- at real field measuring conditions
- with real combustion aerosol
- all instruments operated by the users

and evaluation of systematic deviations

# The Participants

- ÖSBS Leoben (A)
- FHA Aargau (CH)
- IfT Leipzig (DE)
- ETH Zürich (CH)
- IMKO Magdeburg (DE)
- BIA St. Augustin (DE)
- ÖZL Langenbruck (CH)
- FAL Braunschweig (DE)
- VW Wolfsburg (DE)
- IGF Bochum (DE)
- metas Wabern (CH)




The figure shows the experimental set-up. The exhaust is sucked together with ambient air through a mixing tube into the expansion chamber. The probe for all the particle sizers was taken at the same place in the chamber and distributed with tubes of the same length.

Two sources were used for the comparison:

• Diesel engine.

• Combustion aerosol Standard (CAST), that was developed in corporation with metas

The following particle sizers were used:

• 2 TSI DMA 3071

• 8 TSI DMA 3080

• 1 Twin Hauke DMA’s

The following particle counters were used:

• 4 TSI CPC 3022A

• 4 TSI CPC 3010

• 4 TSI CPC 3025

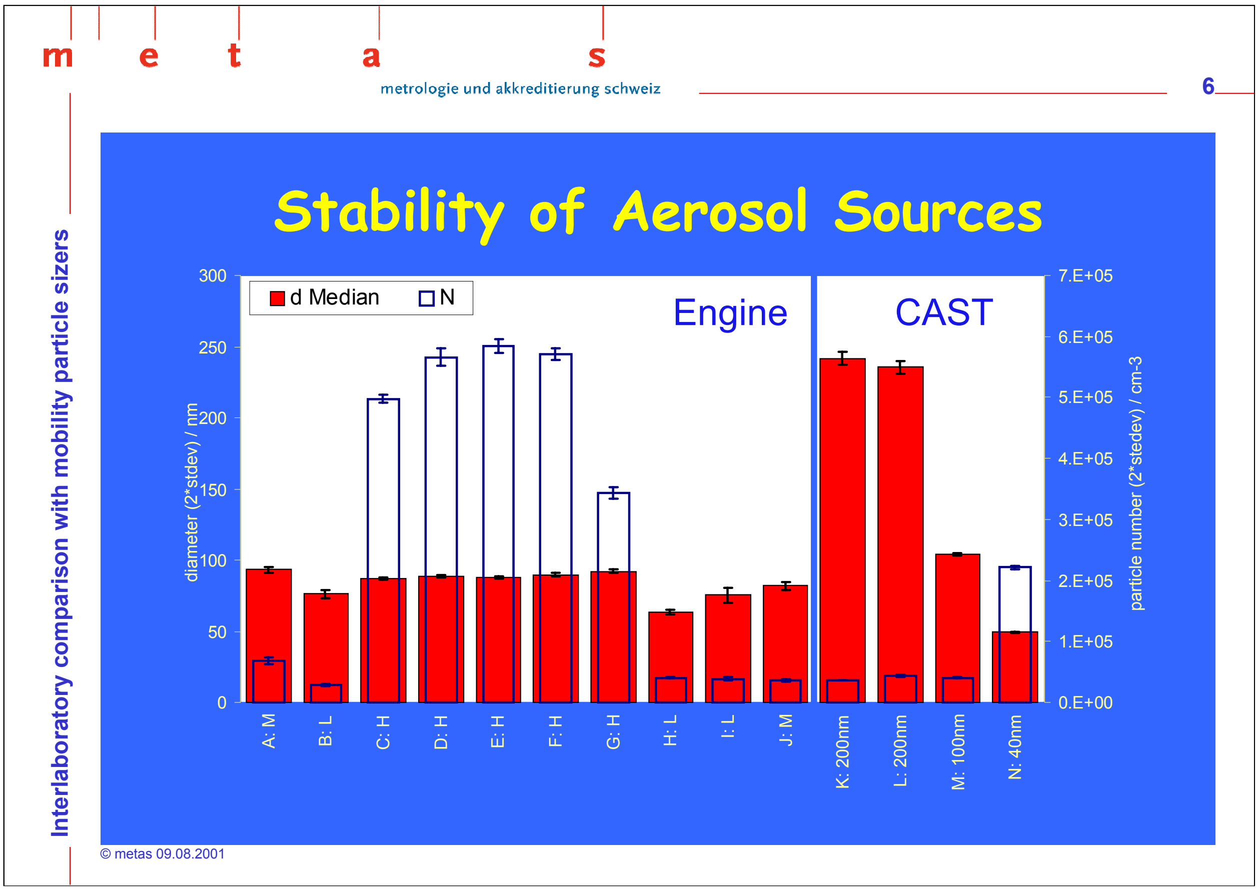
# The Protocol

- 3 concentration levels, one particle size (engine)
- 3 particle sizes: 40 nm, 100 nm, 150 nm (CAST)
- SMPS$^{TSI}$ scantimes, upscan: 120 s or 300 s
- SMPS$^{TSI}$ flowrates, sheath air: 1, 3 or 10 l/min
- at least 10 scans per run
- SMPS Software$^{TSI}$, version 3.2
- analysis: generally 16 to 220 nm,
- results: median, concentration (raw data, fitted)



The comparison was carried out in 14 runs.

During each run of about 1/2 hour, specific settings at the measuring instruments and at the particle sources were kept constant.



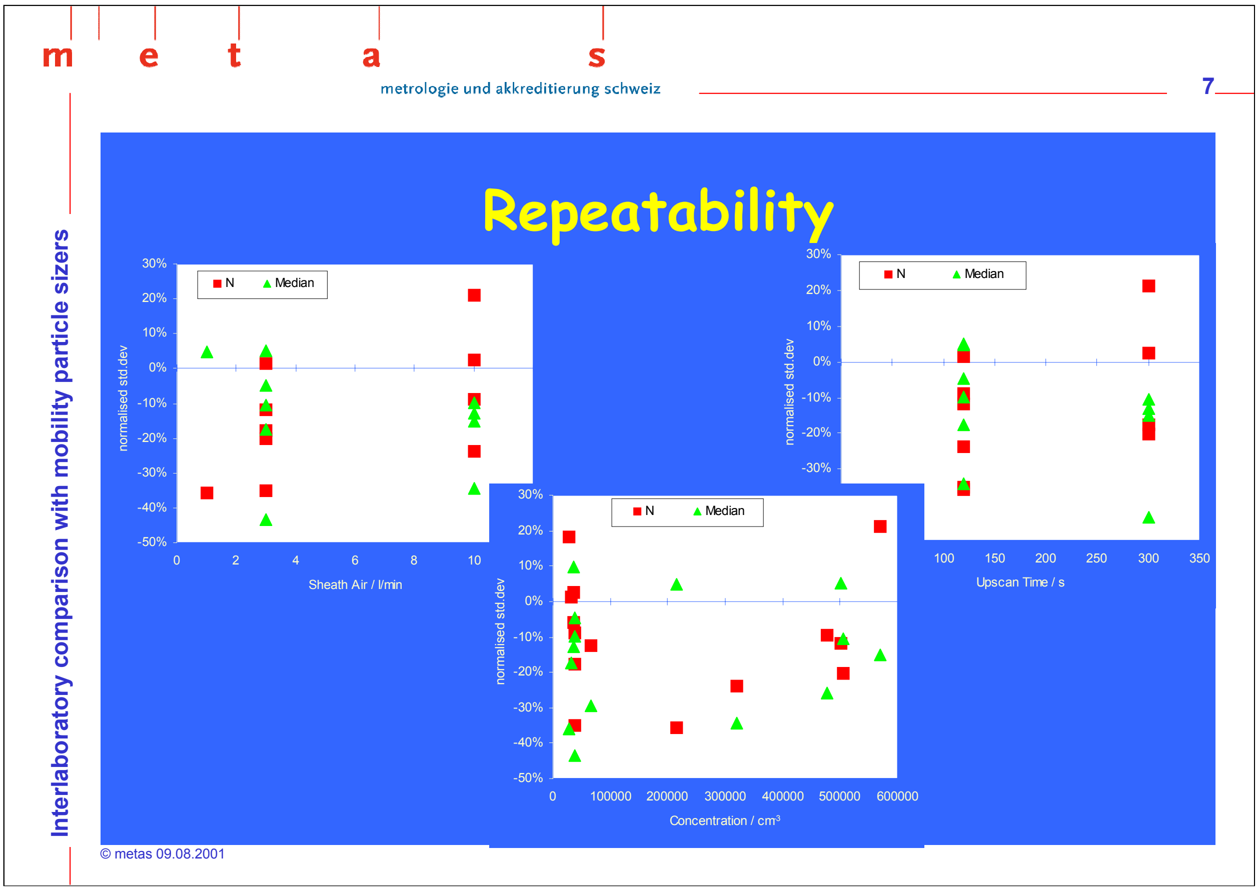

One instruments was kept at the same settings during all 14 runs of the experiment. This allows the comparison of the different settings of the particle generators:

• The engine produces particles with just one size distribution (median of diameter is at 80 nm)

• The concentration could be varied from $3 \cdot 10^4$ to $6 \cdot 10^5$ cm$^{-3}$

• The CAST gave a broader size range (50 to 250 nm) but lower concentrations ($4 \cdot 10^4$ to $2 \cdot 10^5$ cm$^{-3}$)

• The experimental standard deviation of the measurement is indicated as error bars. The averaged relative standard deviation was for the particle size 2 % and the particle conentration 3 %. Contributions to this STD come from the particle source and from the measuring instrument.




The experimental relative standard deviation (RSD) for each instrument for one run is calculated first, then the average of the standard deviations of all instruments is built. But this average standard deviation of one run cannot be compared  to that of another run because of the variation of the particle source.

Therefore we kept one instrument at the same settings and used its result to normalise the average standard deviation of all other instruments. With these normalised standard deviations, it can be seen if specific settings have an influence on the repeatability of the measurement.

The three figures show the influence of the sheath air flow, the particle concentration and the upscan time to the experimental standard deviation for a repeated measurement.

The dots for the particle size and the concentration don’t correlate with any of the chosen parameters. There is no systematic influence on the repeatability.

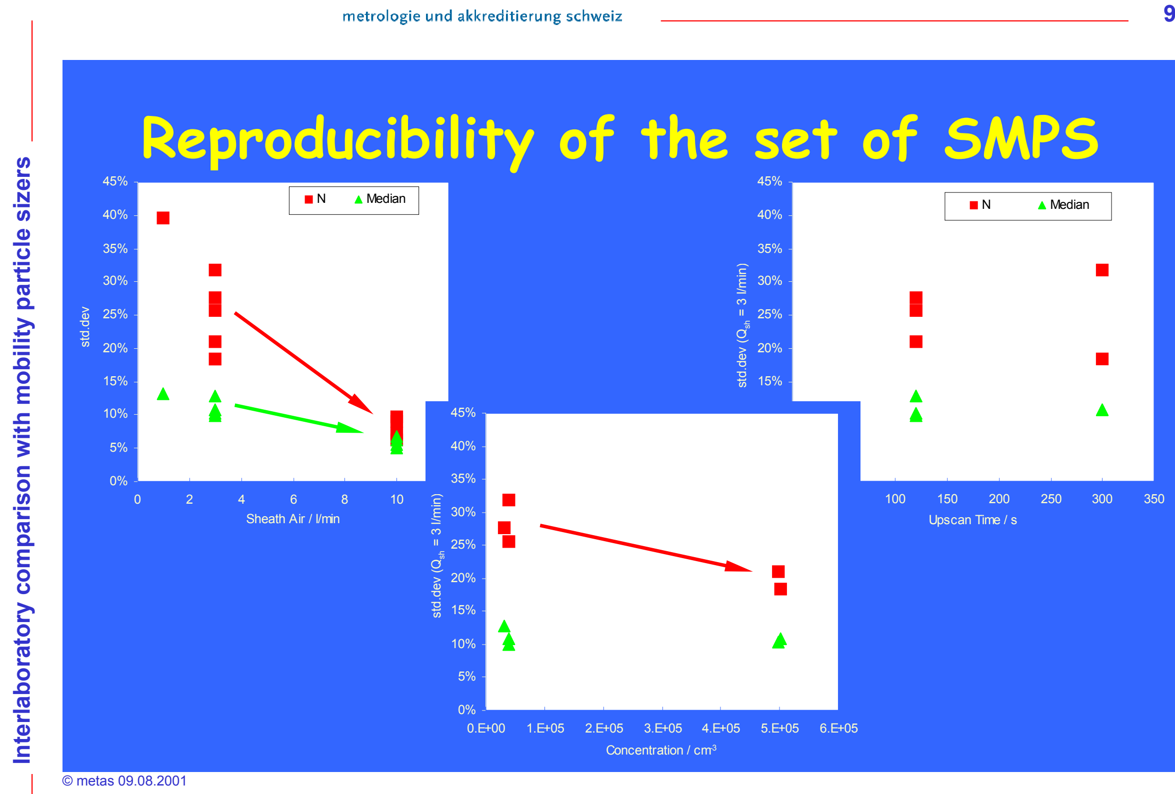
This means, that the measurements don’t need to be repeated more often, if specific settings are chosen.

# Repeatability

repeatability is independent from:

- particle concentration ($10^4$ to $10^6$ cm$^{-3}$)
- flow setting ($Q_{sh}$: 1 to 10 l/min)
- scan time (Upscan: 120 to 300 s)

Note: The repeatability is only one contribution to the uncertainty of the measurement !




This figure shows the experimental standard deviations from the comparison of 9 SMPS with the same settings.

The **figure to the left** shows that the RSD depends on the sheath air setting. The higher the sheath air flow is, the closer together are the particle size and the particle concentration measurements. The standard deviation for particle concentration diminishes from 40 % to 10 % and for particle diameter from 15 % to 5 %, when the flow for SheathAir is increased from 1 to 10 l/min.

The **figure in the middle** shows the RSD of the selected measurements with sheath air set to 3 l/min at different concentrations. The deviation for the particle concentration depends on the concentration. The deviation for particle size does not.

The **figure to the right** shows no influence for the upscan time.

# Reproducibility of the Set of SMPS'

deviations between the instruments are caused by

#1 flow adjustment for Sheath Air (size and concentration)

#2 flow adjustment for CPC (concentration)

#3 adjustment of DelayTime and FlowTime in SMPS[TSI] Software (size)

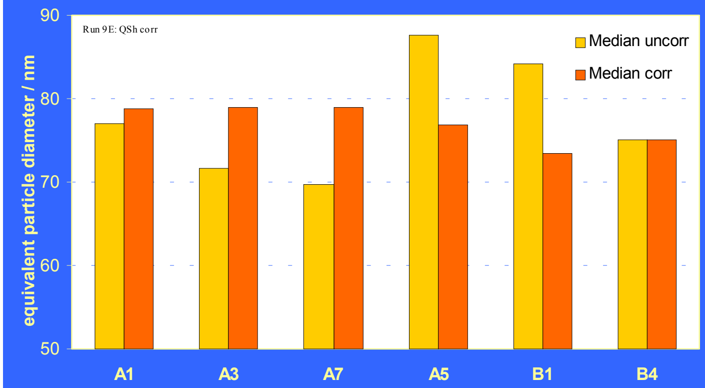
#4 adjustment of photometric mode in CPC 3022 (concentration)

#5 lower and upper limit of analysis (size and concentration)

# #1 Flow Adjustment for SheathAir

correct adjustment of $Q_{Sh\,V}$ results in better values:

$$Q_{ShV}(T,p) = Q_{Sh0} \cdot \frac{T}{273\,\mathrm{K}} \cdot \frac{1013\,\mathrm{hPa}}{p}$$




**averaged values (exp. std. dev.):**

uncorr: (77.6±7.1) nm

corr: (77.0±2.3) nm



The flow of SheathAir differs from instrument to instrument. It must be calibrated periodically. For the adjustment it must be considered that the differential mobility analyser demands a defined volumetric flow ($Q_{Sh\,V}$). This means, the flow changes if the ambient pressure or the temperature of the aerosol changes.

Some results from the comparison could be corrected due to the calibrated Sheath Air flow. The slight change in the average of particle size is accompanied by much smaller standard deviation. Reduced by a factor of 3.

## #2 Flow adjustment for CPC

- with CPC 3022 in the mode HighFlow a flow of 1.5 l/min is assumed. But the flow cannot be independently adjusted.
- adjustment of CPC is performed as a volume flow
- the indication of the concentration must be normalised to common conditions (e.g. 1013 hPa, 273 K)
- consequence: external measurement of mass flow is recommended



In order to perform the correct measuring using the differential mobility analyser (DMA) the flow of the CPC must be adjusted as volume flow.

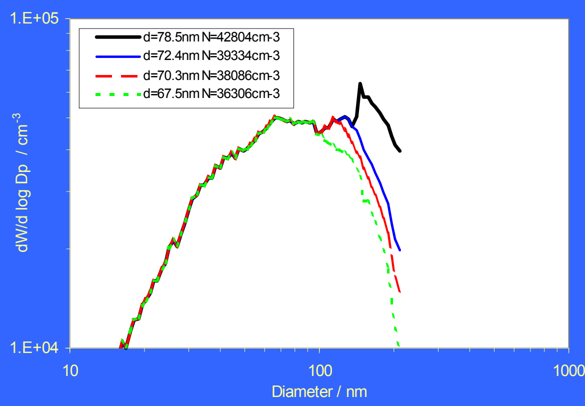
But the results of particle measurements can only be compared, if they are referenced to common gas conditions as e.g. a pressure of 1013 hPa and temperature of 273 K.

# #3 Adjustment of $t_d$ and $t_f$

- default values for DelayTime $t_d$ and FlowTime $t_f$ are assumed by the SMPS software
- attention: $t_d$ and $t_f$ depend on the tubing and the settings of the instrument
- attention: wrong settings cause systematic errors in particle size measurement
- solution: $t_d$ can be measured with SMPS software

# #4 Adjustment of photometric mode

- CPC 3022A switches from counting to photometric mode at $10^4$ cm$^{-3}$
- different assumptions lead to significant different results (see figure)




recommendation:
avoid the photometric mode



The figure shows the result, when the photometric mode does not fit to the counting mode at a concentration of $10^4$ cm$^{-3}$. Even after recalibration and service at the manufacturer a concentration shift of about 10 % to 20 % could be observed.

A correction of that shift cannot be carried out afterwards. The example shows differences in particle size and concentration of up to 15 %.

The only useful measure: Avoid the photometric mode

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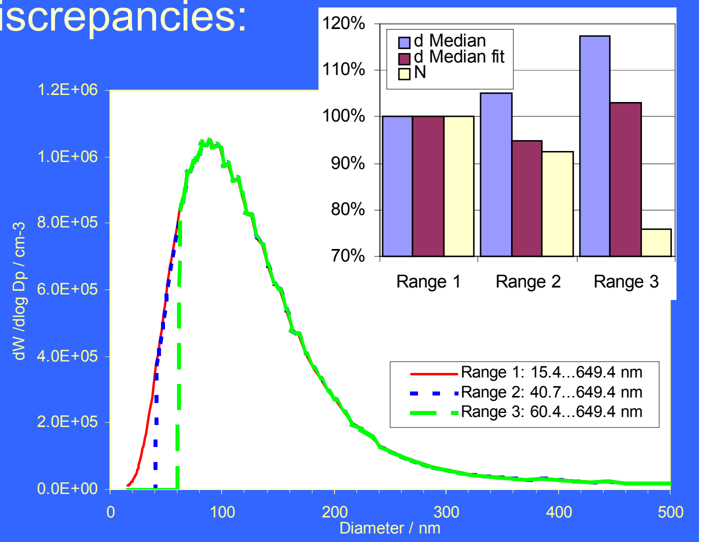
# #5 Procedure for analysis

measures to avoid discrepancies:

- central analysis of data
- agreement for measuring range
- agreement for range of analysis
- agreement for log-norm-fit






Already in advance the participants of this comparison were aware of the importance of harmonised data collection and harmonised data handling.

The measures taken (as in the list) avoided further discrepancies in the results.

The figures show the consequences if e.g. the measuring range was chosen differently. Cutting the size distribution at one end, all the calculated parameters for the particle size distribution show significant changes. This is valid at least for the median of particle diameter and the particle concentration, independently if they are calculated from the raw data or the fitted log-normal curve. As it can be seen in the small figure the difference easily exceed 10 %.

## Summary

- repeatability independent of instruments settings
- relative STD high at low particle concentration and low SheathAir flow
- flow adjustments (SheathAir, CPC) are essential
- no report of particle size distribution without following indications: Range of analysis, (referenced) ambient conditions, type of approximation (fit)



Further information on this comparison can be found in the October issue of the journal “Staub - Gefahrenstoffe der Luft”.