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Filter Efficiency Measurement with Optical Particle Counters—Limitations and Error Sources

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ABSTRACT

Filter efficiency determination by means of the optical particle counting technique can yield information about the filter performance from a single set of upstream and downstream measurements. *In principle*, optical particle counters provide the possibility of a real time determination of particle size distribution and concentration. However, substantial errors in determination of the filtration efficiency may result from the performance characteristics of optical particle counters. This article presents results of a theoretical and experimental study analyzing the effects of variation of particle refractive index and coincidence on the sizing accuracy and counting ability of optical particle counters and the impact on the efficiency determination. Since both parameters, size and count, are critical for the determination of fractional collection efficiency and the most penetrating particle size, exemplary filter efficiency measurements with optical particle counters under various conditions are presented and critically discussed. Depending on the experimental conditions chosen, the efficiency of the same filter can differ by more than an order of magnitude. The limitations reported here may numerically vary for different instruments; however, they are inherent to this technique, in gaseous as well as in liquid media, and must be taken into account during measurements and data evaluation.

* To whom correspondence should be addressed. This work was done in part during this author's visit at the National Chiao Tung University.

INTRODUCTION

Collection efficiency and most penetrating particle size information are important indicators of filter performance (1). They describe particle removal characteristics of a filter and also allow an objective comparison of different products. In order to obtain representative efficiency data or time-dependent filter performance characteristics under circumstances which are as close to the actual filter working conditions as possible, it is advantageous to challenge a filter with polydisperse particles having a rather broad size distribution and made of various materials to assess the "real life" filter performance (2). The size specific quantification of a filter's performance requires both particle sizing and concentration measuring instrumentation, hence an optical particle counting technique would seem to be an interesting and possible approach (3–5). However, among the parameters which may affect the sizing capability of optical particle counters (OPC) are not only the particle size and the material it is made of, but also the specific instrumental design (6–8).

Assuming that particle sampling and transport is not biasing the measurement (9), the quality of information from an OPC measurement depends primary on the ability of the instrument to detect all particles in question, particularly important issue at the lower sensitivity limit of the instrument (10). Furthermore, the measuring characteristics of an OPC may greatly depend on the refractive index of the particulate material. Also the range of particle concentrations in which reliable size and concentration information can be obtained must be established. The determination of all these experimental conditions is crucial, since instruments such as OPCs will "merrily report erroneous data and not notify the user" (11). Laboratory measurements with well-defined particles of various chemical compositions and size distributions allow the limits of this technique to be explored. Based on the above identified criteria, particle sizing and counting characteristics of OPCs have been investigated and the impact of possible measurement errors on the fractional filter efficiency data analyzed.

PRINCIPLE OF OPTICAL PARTICLE COUNTING TECHNIQUE AND MODELING OF THE RESPONSE

Optical particle counters (OPC) detect scattered light from a particle while it is passing through a sensing volume of the instrument illuminated by a light source, such as a laser. The scattered light intensity is used as a measure of the particle size. In an ideal instrument a monotonic relationship between the particle size and the scattered light intensity, independent of the particle material, would allow a unique particle sizing. In reality, this is not the case.

The instruments' response, representing the amount of light scattered into a given solid angle, is not necessarily a monotonic function of particle size, and this behavior will vary not only with the refractive index of the particulate material but will also depend on the angular range in which the scattered light is collected. These effects were reported previously (6, 7, 12), so here only the relevant key aspects will be briefly given.

The response R is usually defined as the scattered light flux normalized to the incident radiation. For an unpolarized laser beam irradiating a particle, the response R is given by

$$R = \frac{\lambda^2}{8\pi^2} \int_{\theta_1}^{\theta_2} \{I_1(x, m, \theta) + I_2(x, m, \theta)\} \sin \theta \, d\theta$$

where I_1 and I_2 are the scattered light intensities perpendicularly polarized to each other, m is the refractive index of the particle material, given by a complex number, and x is the nondimensional particle size parameter defined as $\pi D_p/\lambda$, with D_p being the particle diameter and λ the wavelength of the illuminating beam. θ_1 and θ_2 are angles limiting the solid angle for scattered light collection. The model calculations are based on the Mie theory of light scattering (13) and on the assumption that the particles are spherical. Substantial deviation from the sphericity of particles complicates the prediction of the response (14, 15). An unpolarized He-Ne laser ($\lambda = 633$ nm) was assumed to be the illumination source for our calculations. The OPC performance analysis given below is not confined to the instruments used in this study, but is representative to any particle counting system with a similar optical geometry.

In general, there are two basic designs of OPCs with regard to optical geometry. The scattered light is typically collected either in the narrow forward direction with respect to the direction of propagation of the illumination beam of light or into a wide angular range. Forward scattering systems are very suitable for sizing absorbing particulate matter, since for such particles they exhibit a monotonic response versus size. However, for nonabsorbing particles the response becomes multivalued around and above 1 μm in diameter (12), resulting in ambiguous sizing.

The second scattering geometry collects light scattered by a particle into a large angular range, oriented perpendicularly or axial with respect to the beam direction. In this case the response is usually a monotonic function of size for nonabsorbing particles, so that the instrument's calibration typically performed with nonabsorbing polystyrene latex particles ($m = 1.59 - 0i$) usually holds for optically transparent particles (7). For that reason this scattering geometry is widely used in various optical particle counting systems. The presence of absorptivity, however (the imaginary part of the refractive index

is nonzero), reduces the sizing sensitivity of such a system remarkably. This behavior is shown clearly in Fig. 1. The lines represent the theoretical response calculated for an OPC used in this study (Particle Measuring Systems, LAS-X). The test particles were generated by the electrostatic size classification method described extensively elsewhere (16). Based on Fig. 1, it is also obvious that for absorbing particles in the size range from 0.3 to about 1 μm, the size resolution is practically nonexistent. Additionally, above 1 μm in diameter, due to the shift of the response with respect to the curve for nonabsorbing particles, measured absorbing particle sizes will be severely undersized, e.g., a 1-μm NaCl particle is optically equivalent to about a 0.4-μm carbon particle. The very good agreement between the experimental data and theory proves the feasibility of using theoretical performance predictions for OPCs, hence the effect of absorptivity of the particle material was studied numerically. The results, shown in Fig. 2, indicate the changes of the response of the system as a function of particle size and the imaginary part of the

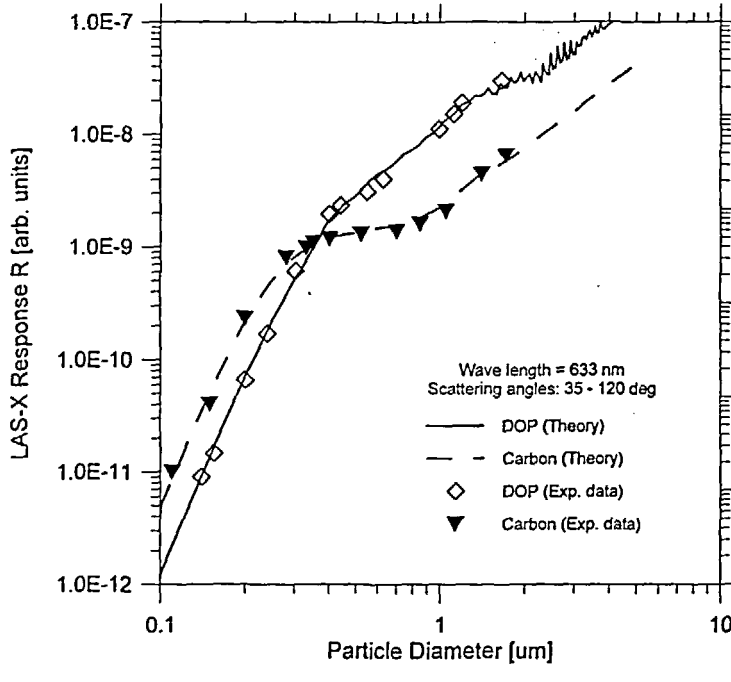

FIG. 1 Experimental response of the LAS-X optical particle counter to di-octyl-phthalate (DOP) particles with $m = 1.48 - 0i$ and carbon particles with $m = 1.77 - 0.6i$ shown with the corresponding theoretical curves.

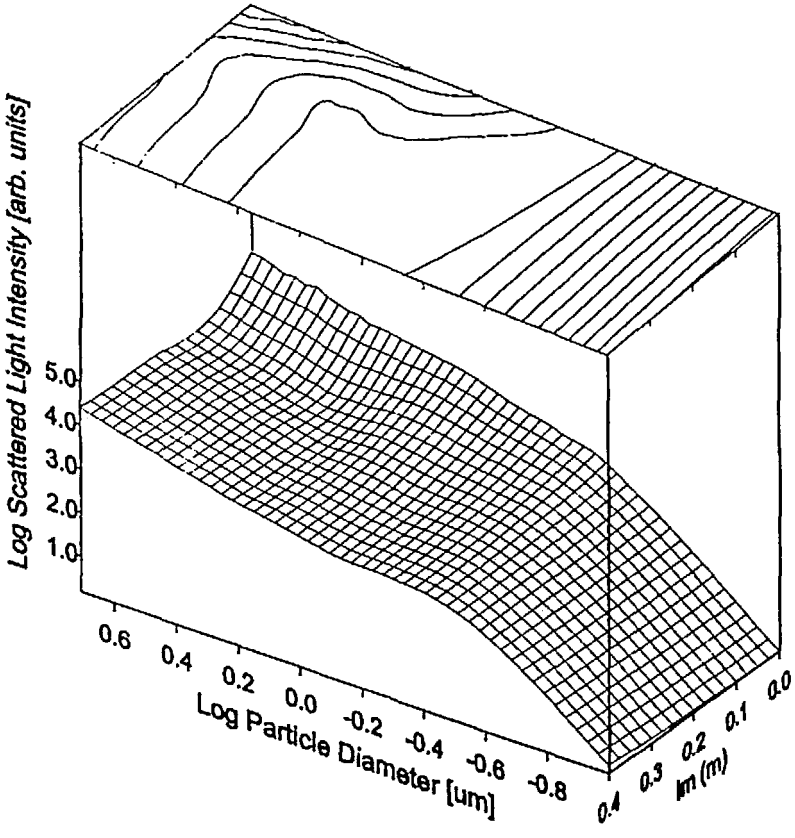

FIG. 2 Theoretical prediction of the response of the wide angle OPC used in this study for particulate material with varying absorptivity. The real part of the refractive index is here equal to 1.54. Note the choice of the logarithmic scales necessary to emphasize the instrument's performance.

refractive index, representing the absorption characteristics of particles. The real part of the refractive index was kept constant and equal to 1.54. The three-dimensional data presentation shows the response as a function of particle diameter and its absorptivity. The contour map is particularly illustrative because it indicates the change of the OPC response with increasing particle size. The contour lines represent the intensity level of scattered light in equal intensity increments as a function of particle diameter. It is apparent that for submicrometer particles, the intensity increase is more rapid (i.e., much better size resolution), resulting in a narrower spacing of the contour lines. One can

also see the continuously increasing response for Im(m) = 0, observed also in Fig. 1. However, with increasing Im(m), the resolution of the instrument changes and deteriorates, showing for Im(m) larger than 0.2 virtually no increase in the response in the logarithmic size interval from about -0.5 to 1.0, corresponding to particle diameters of 0.3 to 1.0 μm. Moreover, for a weak absorption [Im(m) $\leq$ 0.2], the undersizing will be different and even more severe than for stronger absorbing particles. These effects will have an impact on the numerical values of filter efficiency should a filter be challenged with particulate matter which varies in chemical composition with regard to absorption.

EXPERIMENTAL ARRANGEMENT AND METHOD

A schematic diagram of the experimental setup is depicted in Fig. 3. Test particles were generated from solutions [NaCl in water; di-octyl-phthalate (DOP) in isopropyl alcohol] or water suspensions (in the case of carbon) by means of an atomizer. Subsequently, the particles were dried in a diffusion dryer. The resulting polydisperse particles were brought to charge equilibrium using a radioactive Kr-85 source and then allowed by means of an electrostatic classifier (TSI, Inc. Model 3071) to generate quasi-monodisperse particles of various, well-defined sizes (12, 16). Since the particle-size-determining voltage in the classifier is limited to 10 kV, the flow rates through the classifier had to be varied to generate test particles covering more than one decade in size. However, the flow-rate ratio (aerosol to sheath air) in the classifier was always set to 1:10, resulting in aerosols with a geometric standard deviation of the order of 1.05. The concentration of these filter challenge particles was controlled using particle-free dilution air. To obtain very high concentrations of polydisperse filter challenge material, the electrostatic classifier was bypassed. The total particle concentration was monitored by means of a condensation nuclei counter (CNC; TSI, Inc., Model CPC 3022) simultaneously with the OPC measurement. CNC data were used here as a concentration reference for size-classified particles. CNCs are capable of counting high particle concentrations of particles typically above about 10 nm, but they provide no size information. In this study two wide angle OPCs of the LAS-X type (Particle Measuring Systems, Inc.) were used. They are identical with regard to their optical geometry, but they were designed to work in different particle size ranges and at different sample flow rates. The first instrument has a particle sizing range from 90 nm to 3.05 μm particle diameter divided into 16 sizing channels and was operated at 1 cm^3/s flow rate. The second instrument is capable of particle sizing in the diameter range from 100 nm to 7.5 μm. The size range is also divided into 16 channels and has a higher sample flow rate, selected for these experiments to 2 and 6 cm^3/s. Both instruments have the

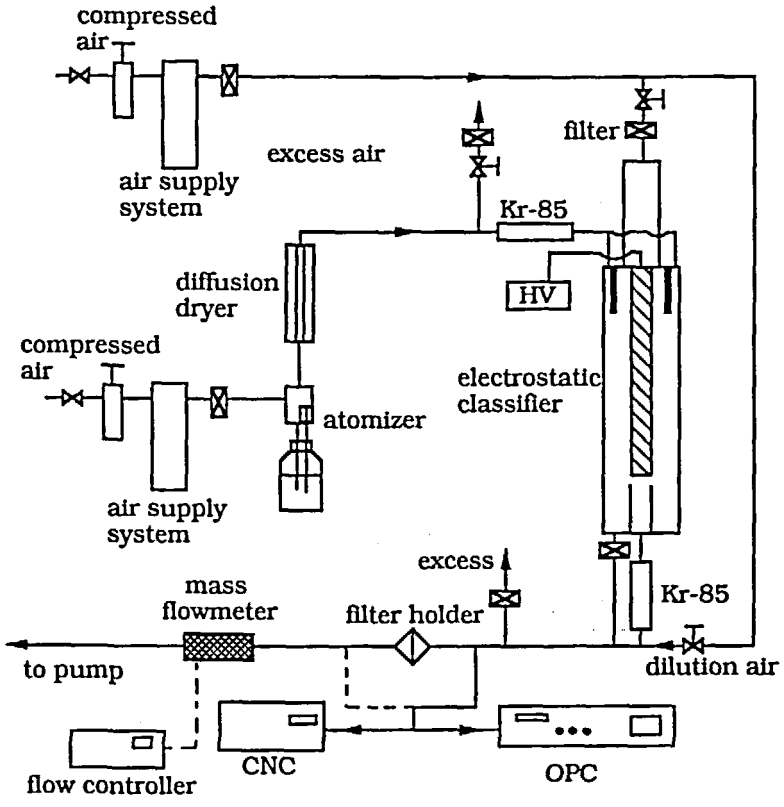

FIG. 3 Schematic diagram of experimental set-up for particle generation and filter efficiency measurement.

option of using subranges for particle sizing in order to improve size resolution; this option was not used in this investigation.

RESULTS AND DISCUSSION

Besides their particle-sizing capability, OPCs are also suitable for particle number concentration measurement. However, at higher concentrations these measurements suffer from coincidence errors which always occur at levels near or above the design limits of an instrument. In such a situation the measured concentration becomes increasingly underestimated and at the same time the measured particle size distribution becomes distorted. Two types of coincidence errors can occur in OPCs. The "spatial" coincidence, when more

than one particle is present in the sensing volume, and the "electronic" coincidence, when the pulses resulting from the particles passing through the viewing volume occur too rapidly for the electronics to be detected as separate events. Based on a theoretical performance analysis of particle-counting instrumentation (e.g., Refs. 17–19), one can conclude that for measuring situations where the product from the number particle concentration (N) and the size of the viewing volume (V_s) is $\ll 1$, the indicated deviation in relation to the actual particle concentration is caused mainly by the "electronic" coincidence.

In the first stage of this work we investigated the concentration range in which an accurate, coincidence-free particle concentration measurement with OPCs is possible. In these experiments the particle sizes were kept well within the sensing range of the instruments, typically between 0.15 and 0.5 μm, with concentrations that were varied from about 100 to about 100,000 cm^{-3}.

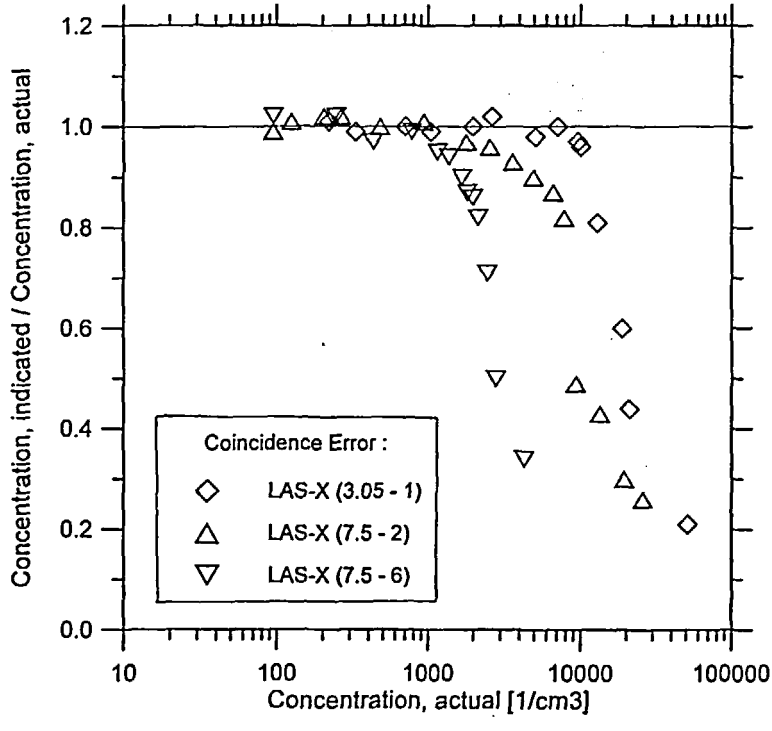

FIG. 4a Experimentally determined optical coincidence error resulting from high particle concentrations for LAS-X optical counters (number in parentheses relate to the upper sizing limit and sample flow rate) presented as a function of concentration.

The particle number concentration from an OPC measurement can be determined from the particle count frequency dN/dt [number of detected events (particles) per unit time] and the sample flow rate Q_s:

$$C = \frac{dN}{dt} \cdot \frac{1}{Q_s}$$

Figure 4a shows the concentration ratio measured by the OPC (concentration, indicated) and by the CNC (concentration, actual) as a function of the increasing actual particle concentration. For lower concentrations this ratio is about unity. Increasing the particle concentration causes an increasing coincidence error, resulting in an underestimation of the concentration. This behavior appears to be dependent on the sample flow rate when the data are presented as a function of the actual concentration. From data shown in Fig. 4b it is apparent that the concentration ratio does not depend on the flow rate, hence

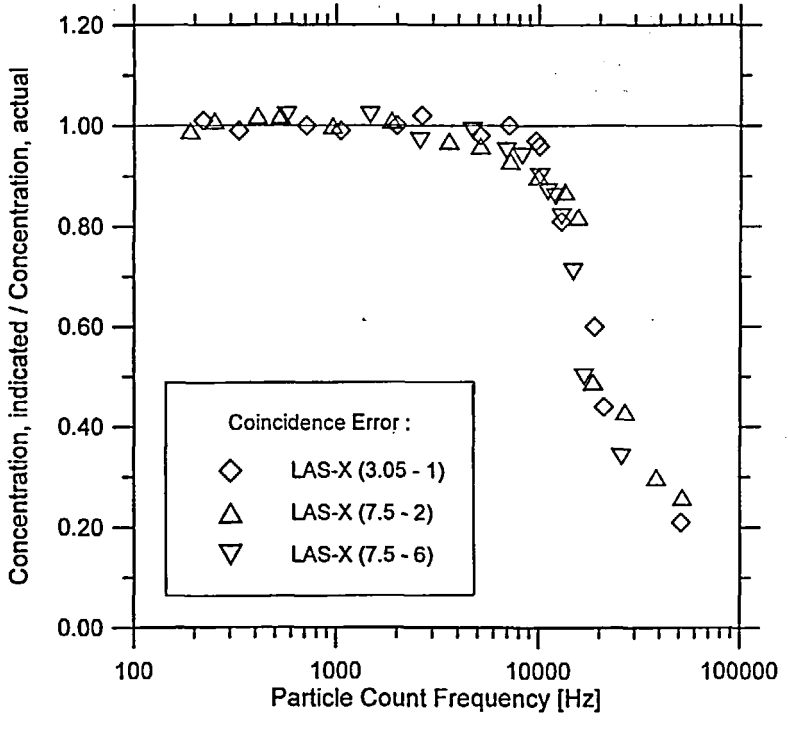

FIG. 4b Experimentally determined optical coincidence error resulting from high particle concentrations for LAS-X optical counters presented as a function of particle count frequency.

the indicated effect is mainly a result of the electronic limitation of the investigated counters. This behavior can also be deduced from the theoretical analysis based on the size of the viewing volume of the instruments used in this study and the chosen particle concentrations. Since the size of the viewing volume can be estimated to about 1.5×10^{-5} cm^{-3}, it is evident that for most concentrations the product $V_s N \ll 1$.

Considering these data, one can easily define a maximum concentration value which can be measured with a given coincidence error. It can be seen that with the OPCs used in this study, reliable concentration measurements with coincidence errors below 10% can be performed for particle count frequencies up to 10 kHz.

From the results presented it is evident that an uncritical application of the optical counting technique for filter efficiency measurement will probably lead to unrealistic results which may be caused by sizing errors due to changes in the index of refraction of the test particles and/or by erroneous sizing and counting due to coincidence errors.

Particular caution has to be applied if polydisperse particles are used for efficiency measurements. The advantage of a combination of polydisperse test particles together with the use of an OPC is obvious. Not only can one obtain information regarding the fractional filter efficiency in real time, but also testing with the polydisperse material is a much better representation of the possible filter operating conditions, hence a more realistic performance indication is provided.

Based on the reasons given above, an investigation of the suitability of OPCs for filtration efficiency measurement under extreme experimental conditions in order to scrutinize possible errors and their magnitude was performed.

First, the fractional filter efficiency of a glass fiber filter was determined under no coincidence conditions ($dN/dt < 10$ kHz) with size-defined NaCl particles using the electrostatic particle size classification system (Fig. 3). The filter efficiency curve was obtained from the OPC data:

$$E(D_p) = \frac{N_1(D_p) - N_2(D_p)}{N_1(D_p)}$$

where N_1 and N_2 are the upstream and downstream concentrations of a given particle diameter D_p, respectively. The results, shown in Fig. 5, indicate the highly efficient filter performance and the most penetrating particle size at 0.4 μm with penetration:

$$P(D_p) = 1 - E(D_p) = 8.7 \times 10^{-3}$$

The same filter was then tested with polydisperse NaCl particles at a high concentration with $N_{1,\mathrm{CNC,tot}} = 5.3 \times 10^6$ cm^{-3}. In this case it was necessary to by-pass the electrostatic classifier. Such test particles had a mean diameter

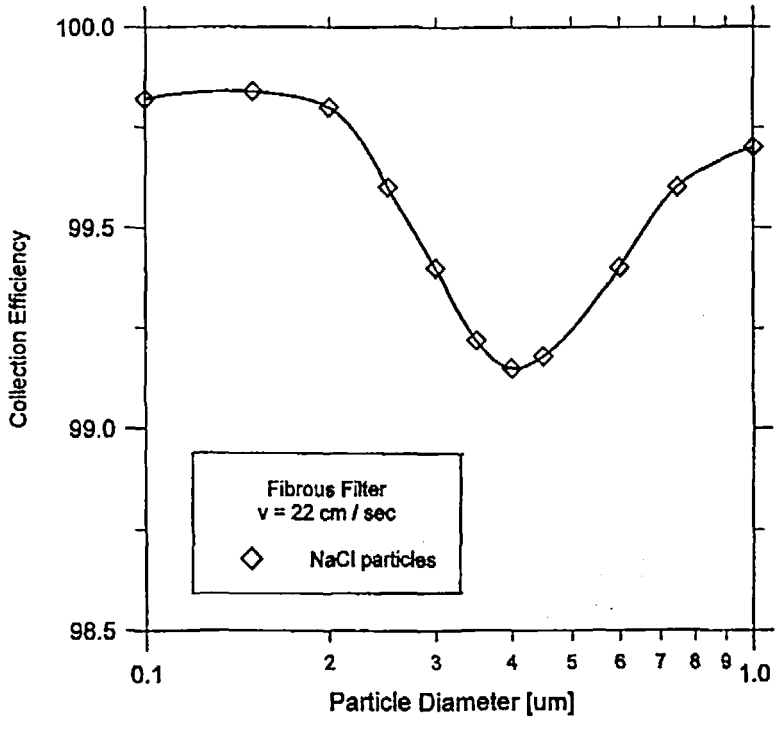

FIG. 5 Actual fractional collection efficiency of a glass fiber filter determined by LAS-X using electrostatically size-classified sodium chloride particles at concentrations below the determined coincidence level.

of about 0.05 μm and a geometric standard deviation of the order of 2.0. These values were assessed from the performance characteristics of the atomizer (20) and from the aqueous NaCl solution strength (0.025 g NaCl per cm^3 of water). The total downstream concentration measured by the CNC, amounted to $N_{2,\,CNC,\,tot} = 6.55 \times 10^3\ cm^{-3}$. The total collection efficiency obtained from the CNC data was 99.87%, in good agreement with the results presented in Fig. 5. Results of the upstream and downstream measurements with the OPC are shown in Fig. 6. The sizing range of the instrument and the nominal sizing intervals are indicated by the scale shown below the channel number. A very high upstream concentration results in severe errors due to coincidence effects—a virtual shift of the size distribution by about one order of magnitude toward larger particle diameters, and an underestimation of the upstream concentration for particles larger than 100 nm (lower sizing limit of the OPC) by a *factor* of

about 30. The downstream concentration, as well as the size distribution, are measured correctly because the particle concentration was decreased due to the filtration effect. Attempting to obtain the fractional efficiency from such a set of data is meaningless.

A melt-blown, electrically charged filter material was used for further measurements. In order to experimentally establish the impact of the particle refractive index on the measurement of filter efficiency, polydisperse sodium chloride ($m = 1.54 - 0i$) and carbon particles ($m = 1.78 - 0.6i$) were used. The particles were electrically neutralized by bipolar ions created by a Kr-85 radiation source (16). The filter efficiency data were obtained at upstream concentrations below 5000 cm^{-3}, which guaranteed a coincidence-

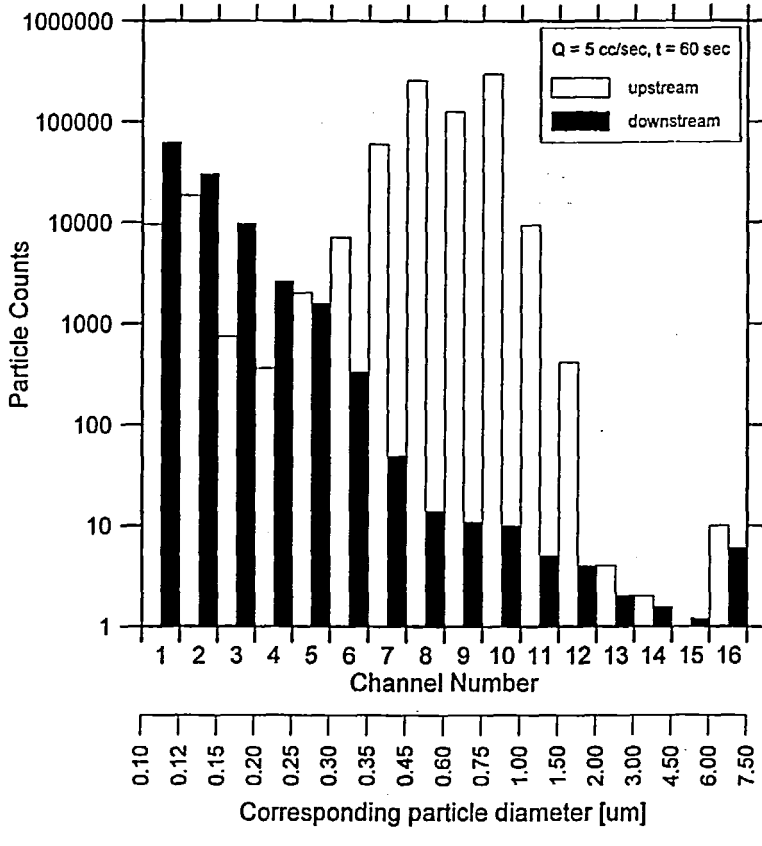

FIG. 6 Virtual changes in the measured particle size distribution upstream (extreme coincidence conditions) and downstream of the glass fiber filter.

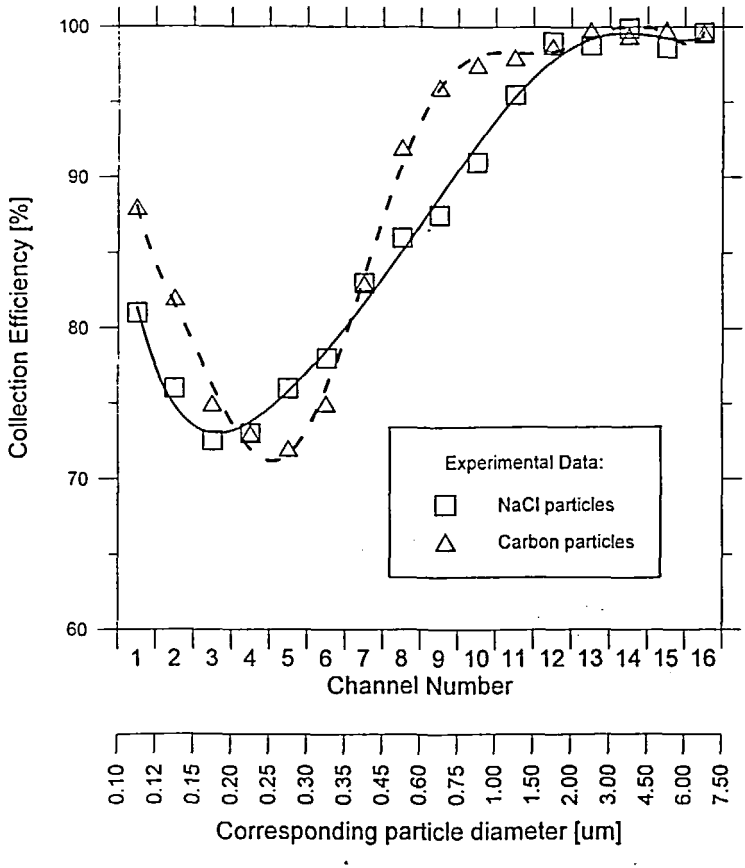

FIG. 7 Change of the shape of the efficiency curve of a melt-blown electrically charged fiber filter resulting from the response change of the OPC due to the difference in the optical properties of particle material.

free measurement. The results are presented in Fig. 7. Due to the fact that the OPC used in this study has a monotonic response and sizes nonabsorbing particles quite accurately (7), the efficiency data obtained with NaCl particles represent the actual filter efficiency curve. In comparison, the efficiency curve obtained with absorbing material has quite a different shape. The apparent better filtration efficiency for carbon particles below 0.3 μm results from particle oversizing due to the refractive index, e.g., 0.15 μm carbon particles have about the same response as 0.2 μm NaCl particles (see Fig. 1), hence they will be classified by the OPC as 0.2 μm particles, thus causing an artifi-

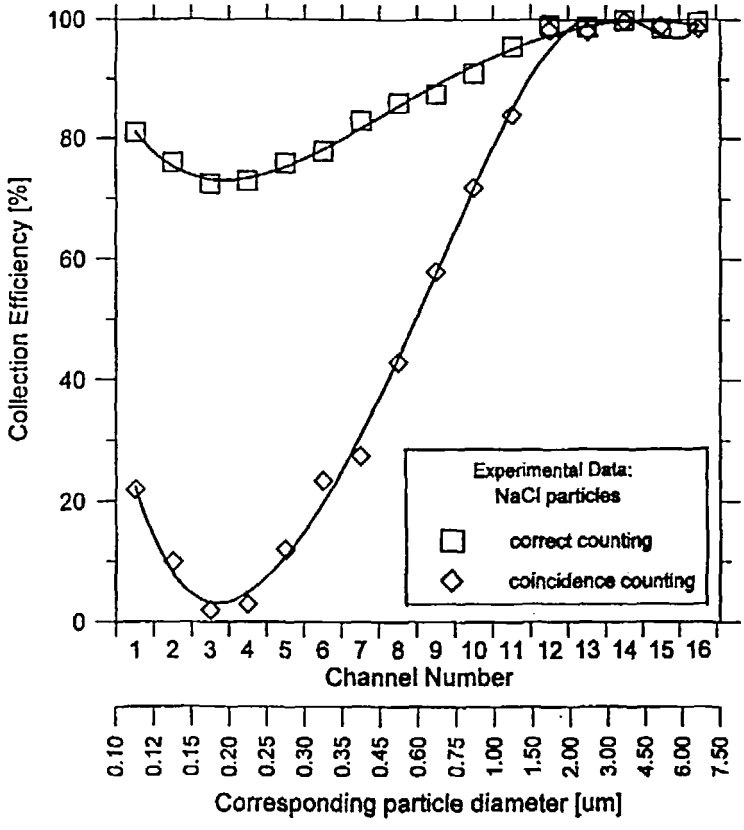

FIG. 8 Unrealistic decrease of the fractional filter collection efficiency of a melt-blown electrically charged filter caused by coincidence errors.

cial shift of the left part of the efficiency curve to larger particles. Between about 0.3 and 1.0 μm the increase in the response for absorbing particles is very weak (see Fig. 2); it improves above 1 μm. This results in agglomeration of the data in the instrument's lower channels, resulting in a much steeper apparent increase of the efficiency curve for these particles sizes. Taken together, these effects result in a virtual shift of the most penetrating particle size from about 0.15 μm (NaCl) to about 0.3 μm (carbon).

Finally, a filter's collection efficiency was measured with polydisperse NaCl particles by choosing a concentration which, for one set of upstream measurements, would lead to a moderate counting coincidence ($dN/dt \sim 18$ kHz). For such conditions the change of the measured polydisperse particle

size distribution is not as obvious as seen previously (Fig. 6). The filter efficiency curves so obtained are shown in Fig. 8. The upper curve corresponds to the actual filter efficiency. The lower curve results from the fact that the upstream concentration was underestimated due to the coincidence effect. These data, combined with the proper downstream concentration measurement, imitates an extremely low filter efficiency. Due to unfortunate choices of particle concentrations, filter materials, and instrument design, this error may gradually produce a variety of virtual filter collection efficiency curves.

CONCLUSIONS

Filtration efficiency measurement using real time particle sizing and counting instruments, such as optical particle counters, can in principle yield a fast, simple, and reliable indication of filter performance. However, the instrument's performance depends on the optical properties of the particulate material and is prone to substantial errors for higher concentrations. By knowing the optical properties of the material being tested, it is possible to explain and correct the data based on the theoretical model, and consequently obtain the actual filter characteristics for given particles. However, with the increasing tendency to test filters with various model aerosols, simulating, e.g., atmospheric particles with regard to their size distribution, concentration, and chemical composition, great experimental care has to be taken for such measurements. Variation of the refractive index of particulate matter may affect the shape of the filter efficiency curve, usually simulating better filtration efficiency, especially for the smallest measurable particles. Ignoring the coincidence effects will result in an erroneous particle penetration determination, where errors of an order of magnitude can easily occur. A possible indicator of the reliability of filter efficiency data obtained with optical particle counters should be an efficiency measurement at various upstream particle concentrations. The occurrence of coincidence effects would result in various efficiency curves and provide information regarding the reliability of data.

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