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Measurement of Aerosol Absorption Coefficient from Teflon Filters Using Integrating Plate and Integrating Sphere Techniques

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Optical transmission of aerosol samples collected on Teflon filters measured by the integrating plate method (IPM) has been used as a means of determining the aerosol absorption coefficient, b_{ap}, by the Air Quality Group at Crocker Nuclear Lab for over 9 years. The results of an intensive quality assurance program, using an integrating sphere photometer configured for measurement of the transmission of filters, demonstrate that the IPM is highly precise (±1%) and does not suffer from inherent overestimation of particle absorption due to internal scattering, a problem commonly attributed to IPM measurements of aerosols collected on Nuclepore filters. If the appropriate correction is applied when filter loading exceeds a monolayer of particles, b_{ap} can be accurately determined by the IPM for aerosol samples collected on Teflon filters.

INTRODUCTION

Establishment of b_{ext} and hence visual range can be accomplished through measurement of both b_{scat} and b_{abs}. It has been shown that the coefficient of absorption due to aerosol particles, b_{ap}, can be extracted from aerosols collected on transparent or translucent filter media through the integrating plate technique (Lin et al., 1973). The method has been calibrated and compared to aerosol absorption determined by difference in an extinction cell, which simultaneously measured transmission and scattering, and found to be reasonably accurate (Weiss and Waggoner, 1984). Since then the integrating plate method (IPM) has come into wide use in many laboratories, including ours at the Air Quality Group of Crocker Nuclear Lab. Since 1984 over 40,000 stretched Teflon membrane filters (Gelman Teflo™) have been analyzed by the IPM as part of the IMPROVE program (Malm et al., 1994). With reasonable care, measurements of b_{ap} by the IPM can be one of the most precise measurements in the entire arsenal of physical, optical, and compositional measurements made from aerosols collected on filters, often approaching precision of 1% in repeated measurements of the same deposit.

Despite the wide use of the technique and the highly precise data that can be obtained from the measurements, questions still remain about the absolute accuracy of the values obtained from IPM as it is applied in most laboratories. At the heart of these problems lies the fact that the theoretical assumptions behind the IPM are routinely violated when b_{ap} is measured from the same samples destined for non-destructive compositional analyses such as x-ray fluorescence (XRF) and particle induced x-ray emission (PIXE). The IPM assumes that less than a mono-

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layer of particles is present on the filter surface (Waggoner et al., 1981) in order to avoid particles shadowing other particles, but such lightly loaded filters generate poor minimum detectable limits for elemental species analysis. A second problem involves the question of light scattering within the filter and deposit, which is only approximately ascertained in the IPM when one has highly scattering substrates such as Nuclepore or stretched Teflon filters.

We have investigated the first problem through a series of calibration tests involving side-by-side collection of aerosols on filters with a range of mask sizes, thus creating samples differing only in areal density. The results indicated that, for Teflon filters, it is necessary to correct absorption measurements from more heavily loaded filters back to a higher value representative of a monolayer sample (Campbell et al., 1989). The magnitude of this 'loading correction' has been determined to be dependent on the optical properties of the particular aerosol and can only be determined from empirical data. This problem appears to be inherent in all measurements of aerosol absorption made from filter samples but it is minimized by keeping filter loading light.

The second problem involves the approximate correction for scattering from the blank and loaded filter media. IPM is generally considered to overestimate particle absorption due to internal scattering (Lin et al., 1973; Weiss and Waggoner, 1984; Horvath and Habenreich, 1989; Hitzenberger, 1993). Nuclepore filters, the substrate for which the IPM was originally developed, have been used in most studies of the method but it has been suggested by some researchers that the inherent overestimation of b_{ap} is common to all highly scattering substrates (Waggoner et al., 1981). Unfortunately, evaluating the results of these studies is complicated by the differing instrument configurations used for the IPM. In this paper we present the results of a calibration study of IPM measurements using an integrating sphere that demonstrate that the method is free from significant overestimation errors, even for heavy particle loading, when aerosol samples are collected on Teflon filters.

EXPERIMENTAL METHOD

The integrating plate system used is similar in design to that described by Lin et al. (1973) except for the light source which is a diffused Ne-He laser. In house tests have shown that the values of b_{ap} derived from the laser integrating plate method agree with values for the same system using a white light source within the range of experimental error. A schematic of the IPM is shown in Fig. 1. To obtain b_{ap} the light intensity transmitted through the Teflon filters is measured before and after sampling and the results combined as:

$$b_{ap} = \frac{1}{L} \ln(I_0/I),$$

where I_0 is the transmitted intensity for the blank filter, I is the transmitted intensity for the exposed filter, and L is the length of the air column sampled (L = sample volume/filter area). The detector output is calibrated against a series of standard filters daily. Overall precision for measurements of b_{ap}, including estimated error in the sample volume measurement, is within 6%.

In order to evaluate the accuracy of our IPM a separate system was designed to do laser integrating sphere analysis (LISA). This system consists of a commercially available integrating sphere (Labsphere, Inc.) with sample holders that can position slides in front of ports located at various positions around the equator of the sphere, which is coated internally with a highly reflective, diffusing surface. A

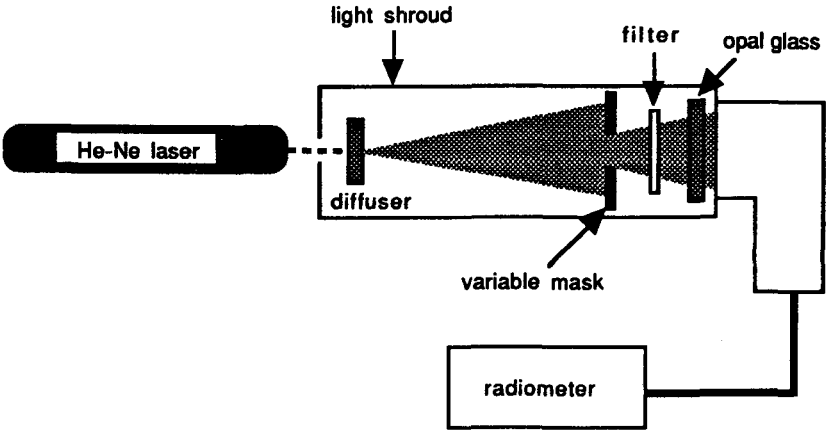

FIGURE 1. Integrating plate method (IPM) configuration.

detector positioned at the top of the sphere measures the intensity of light reflected inside. As in the IPM, the illumination source used is a diffused He-Ne laser. Detector response was calibrated using reflectance and absorption standards traceable to NIST. The source intensity was monitored during all measurements by means of a beam splitter and laser power meter. By positioning the filter, supported in a photographic slide frame, first at the entrance and then at the exit of the sphere both the total optical transmission and reflectance may be measured as shown in Fig. 2. Since Teflon filters have no measurable absorption coefficient all losses of incident intensity are due to absorption by aerosol particles. The value $I_T/(1-I_R)$, where I_T is the transmitted intensity and I_R is the reflected intensity, is equivalent to I/I_0 from the IPM in the equation for b_{ap}. All measurements were made with the side of the filters containing the particle deposit facing away from the laser beam.

The two major advantages of LISA over the IPM for measuring b_{ap} are the reduced possibility of scattering losses between the filter and detector, since the integrating sphere is essentially a 2π radiometer, and the ability to measure the back reflectance of the filter after sampling obviating the need for any presampling measurements of the blank filters. While the first advantage may represent only a very small improvement in accuracy over a well-configured IPM system the second point is potentially of great significance. Not only is the elimination of presampling transmission measurements a convenience, it also accounts for a characteristic of translucent filter substrates that has been overlooked by some groups using the IPM; that the presence of particles on, and within, the substrate changes the back reflectance even with the sample deposit facing away from the light source. This effect has been characterized by Clarke (1982) for Nuclepore filters using a reflectometer apparatus and is apparently related to the inherent error in IPM with Nuclepore filters since the change in substrate transmission due to the sample is not accounted for.

Using the integrating sphere in a similar fashion we measured the reflectance of Teflon and Nuclepore filters before and after sampling. Our results for the Nuclepore substrate were very similar to Clarke's, in that the decrease in re-

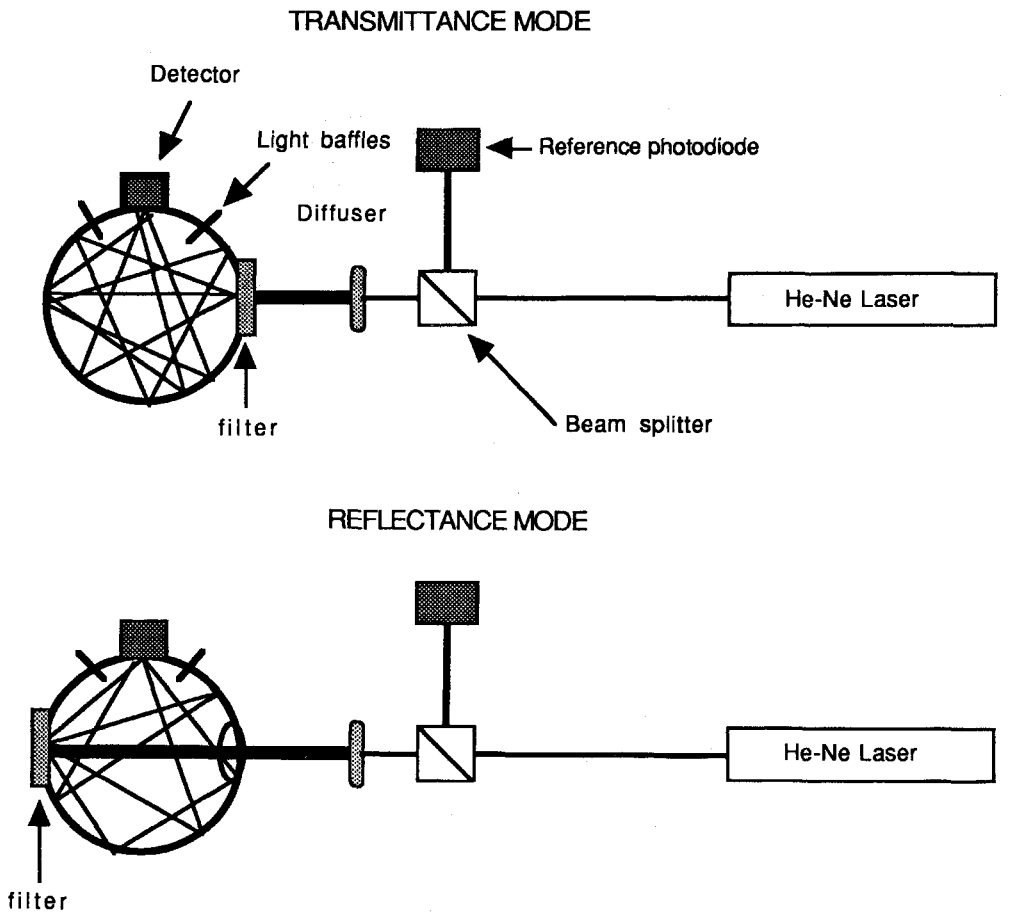

FIGURE 2. Measurement of sample optical absorption by LISA.

flectance correlated well with the measured decrease in transmission for a group of filters containing aerosols from the same source. However, the results for a group of Teflon filters were quite different, as shown in Fig. 3. Not only is the relative decrease in reflection (dR/R) consistently less for the Teflon filters, it shows only a very weak correlation to either the change in transmittance or the sample mass. Although the two sets of samples were not from the same sources, both were ambient aerosols with approximately the same range of filter loading as shown in Fig. 4.

In order to compare b_{ap} measured by LISA with the IPM a set of Teflon filters containing ambient aerosol samples collected in Davis and at various locations throughout North America, including the set represented in Figs. 3 and 4, were analyzed by both techniques. These samples were chosen to represent a wide range of sample loading and aerosol mixtures. These values were converted to b_{ap} and are compared to the IPM results in Fig. 5. Agreement between the two techniques is remarkably good, with a very high correlation and a slope not significantly different from unity. This demonstrates that mea-

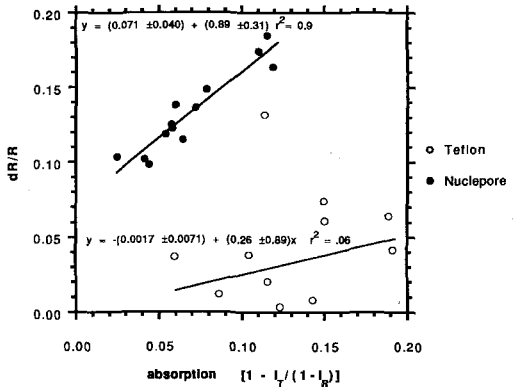

FIGURE 3. Decrease in filter reflectance of back side of Nuclepore and Teflon filters vs. measured absorption.

surements of aerosol absorption from the two techniques are equivalent and the IPM is not subject to chronic overestimation when Teflon filters are used.

DISCUSSION

The optical interactions that occur when well-diffused light is incident on a highly scattering material such as the stretched membrane of a Teflon filter are complex, particularly when the presence of small particles of unknown composition and

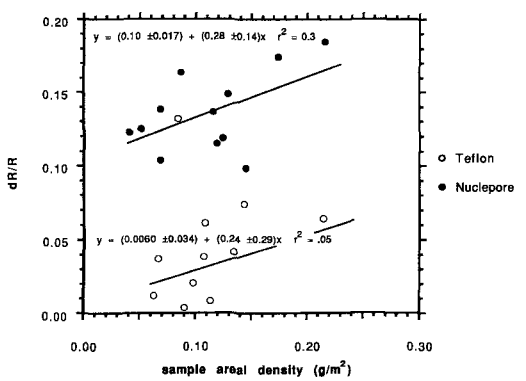

FIGURE 4. Decrease in filter reflectance vs. sample density.

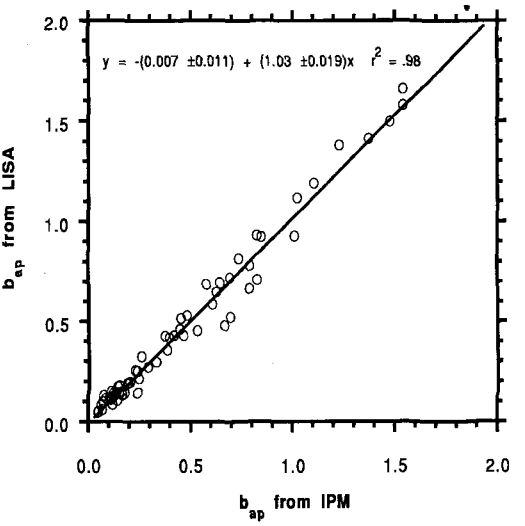

FIGURE 5. Comparison of aerosol absorption coefficient measured by integrating plate and integrating sphere techniques.

morphology present within the membrane is included. To accurately model such a system whould be extremely difficult, and no such attempt will be made here, but with the data presented in this paper we are able to make some conclusions as to the significance of the undefined optical interactions that occur within the filter/particle system. The task is much simplified by the integrating sphere. Since LISA, as described here, is capable of accounting for essentially all scattered and transmitted light the only possible mechanism for significant attenuation of the incident intensity is absorption by aerosol particles captured on the filter.

A possible source of inaccuracy still exists, however, since the high porosity of the Teflon mesh allows some particles to be captured within the filter as well as on the surface. Multiple scattering within the substrate could result in an increased probability of individual photons interacting with particles thereby causing absorption to be overestimated. The results shown in Figs. 3 and 4 suggest that this is

not a significant problem since an increase in the number of potential absorbers (particles) would be expected to decrease the intensity of both forward and back scattered light, as it clearly does for the Nuclepore filters, but there is little correlation of sample absorption or density to the magnitude of the decrease in reflectance after sampling (dR/R). Apparently, the type of internal surface to surface reflections postulated by Clarke (1982) for the Nuclepore substrate are not prevalent in the Teflon membrane.

CONCLUSIONS

A purely empirical study designed to determine the accuracy of aerosol optical absorption coefficients (b_{ap}) derived from transmission measurements of aerosol samples collected on Teflon membrane filters using the integrating plate method has produced the following results.

1. The internal reflectance of Teflon filters is not affected by the presence of light absorbing particles to the same extent, or in the same manner, as Nuclepore filters and therefore it cannot be assumed that IPM measurements of b_{ap} from Teflon filters will be subject to the same overestimation errors.
2. IPM measurements of Teflon filters are equivalent to transmission/reflection measurements made with the best available technique, laser integrating sphere analysis, which is believed to be free from any losses of incident light intensity external to the filter and particle deposit.

In light of these results we believe that Teflon membrane filters remain the optimal substrate choice for point measurement of aerosol light absorption, particularly where elemental analysis by x-ray methods is also desired.

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