Measuring Mixed Cellulose Ester (MCE) Filter Mass Under Variable Humidity Conditions

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Mixed cellulose ester (MCE) filters, used routinely to collect dust samples from air for fiber analysis, are the only filter type that can be prepared for both phased contrast microscopy and transmission electron microscopy analyses. However, whenever fiber counts require collecting dust masses <100 μg on a single filter under variable relative humidity (RH) conditions, historically noted effects of humidity on MCE filter mass can hinder accurate estimates of dust mass, measured as loaded minus unloaded filter mass (M). In this study, a baseline set of hundreds of paired measures of change in RH versus M over different time intervals were obtained over a 5-day period for replicate series of 40 unloaded 37-mm MCE filters under varying RH conditions at a nearly constant temperature. Similar baseline data were obtained for 25-mm MCE filters. Linear regressions fit to these data allow improved estimates of dust mass loaded onto MCE filters from measures of M and RH made before and after loading occurs. Using established theory, these relationships were generalized to address temperature variation as well, and examples of numerical applications are provided.

Keywords: absolute humidity; asbestos; balance; modeling; moisture; regression; temperature

INTRODUCTION

Overview

Mixed cellulose ester (MCE) filters have long been used to measure suspended solids mass in water samples and suspended particulate mass in air samples. Currently, MCE filters are a standard medium used to collect dust samples from air for analysis of fibers including asbestos [e.g. National Institute of Occupational Safety and Health (NIOSH), 1994a,b]. For asbestos analysis, MCE filters are the only filter type that can be prepared for both phased contrast microscopy (PCM) and transmission electron microscopy (TEM) analyses because while different types of filters may be analyzed by TEM, MCE filters (which, after chemical preparation, become optically transparent) are required for PCM analysis. Unless the amounts of dust collected are very small (e.g. <100 μg for 25-mm filters), excessive total-particulate loading (i.e. dust ‘overloading’) may obscure fibers counted by either PCM or TEM analysis, and so interfere with fiber detection and quantitation (NIOSH, 1994a,b). However, any dust mass this small can be measured accurately (as the mass gained by a filter after dust loading) only if the mass of the filter material itself, including its moisture content, remains relatively constant between the times at which that filter is weighed (first blank, then after dust has been loaded).

As discussed below, measurements of MCE filter mass are particularly sensitive to humidity. Traditionally, mass instability of MCE filters under different humidity conditions has not been an issue for fiber analyses because such analyses typically report fibers per unit of air volume sampled, not fibers per unit of dust mass collected. However, it is sometimes
necessary to characterize asbestos fibers and dust mass collected together on the same MCE filter. For example, such analysis of fibers and dust mass on the same MCE filter is required in studies that use PCM (or both PCM and TEM) to characterize fibers per unit mass of dust measured (depending on application) either as total suspended dust, respirable dust, or specific size fractions of dust (e.g. Berman and Kolk, 1997, 2000). In such studies, to the extent MCE filter masses are affected by changes in humidity when adequate humidity control is not feasible, accurate estimation of dust mass collected on MCE filters requires adjusting the mass of each filter measured both before and after dust-sample collection, to account for expected humidity effects. This paper describes how to make such adjustments and thereby facilitate reliable determination of particulate masses deposited on MCE filters.

**Background**

Moisture-related factors can substantially affect measured weights of filters composed of hygroscopic materials (such as cellulose esters, glass, and quartz) that are used for personal exposure sampling, more so than for filters made of relatively non-polar materials such as teflon, polycarbonate, polyvinyl chloride (PVC), or polytetrafluorethylene that have little or no capacity to absorb and may even repel water, although even some of these apparently non-hygroscopic materials can absorb moisture and influence weights measured after sustained desiccation (Charell and Hawley, 1981; Lawless and Rodes, 1999). For more than five decades, researchers have known that MCE filters are particularly hygroscopic, that the mass of these filters is affected by moisture in the environment in which such filters are stored and weighed, and that filter weight changes due to changes in humidity are sufficiently large to affect the validity of mass determinations obtained using this type of filter (Winneberger et al., 1963; Strand et al., 1978; Lowry and Tillery, 1979; Charell and Hawley, 1981; Beaulieu et al., 1986; Lawless and Rodes, 1999). By the early 1960s, weights of Millipore HA 47-mm MCE filters were reported to increase by more than 400 µg (i.e. by ~0.1%) when relative humidity (RH) increased from 52.5 to 63% in weighing-room air. On removal from desiccated conditions, moisture contained in these filters equilibrated rapidly with that in room air in 5–10 min, with ~50% of this equilibration occurring in the first minute (Winneberger et al., 1963; Beaulieu et al., 1986). Among other things, this suggests that ‘conditioning’ of filters by desiccating them prior to weighing will not solve the mass measurement problem because the filters will respond to moisture in the air within the time period required to move the filter from the desiccators to the balance.

A comparison of 10 different filter types used to determine the mass concentration of airborne particulate matter (PM) sponsored by the NIOSH showed that MCE and nitrocellulose filters have the largest variation in mass when weighed on 30 separate days, and also when exposed to different RH and temperature conditions, compared to other filter types such as PVC and Teflon filters (Lowry and Tillery, 1979). This study produced several recommendations to minimize filter-weight variability, including using weight-stable filters when possible and when not, use of blank control filters subject to the same conditions a sample filters to provide an (otherwise unspecified) empirical basis for adjusting measured filter mass. US Environmental Protection Agency (EPA) National Ambient Air Quality Standards (NAAQS) for PM subsequently required that rooms suitable for PM_{2.5} filter weighing have environmental controls allowing temperature to have a mean value of 20–23°C and vary by ≤2°C over 24 h, and RH to be controlled ideally in the range of 30–40% and in any case to within 5% of the RH at which samples were taken (Lawless and Rodes, 1999). These EPA guidelines do not quantify the likely magnitude of error expected to arise even if the recommended environmental controls are followed.

Charell and Hawley (1981) estimated air-moisture-related mass gains on Millipore HA 47-mm MCE filters to range from 1.2 to 1.5 mg per 1% increase in the absolute moisture (as %) in air, with ~75% of this change occurring in approximate linear proportion to elapsed time over a period of ~70–80 s. They observed that these linear patterns differed for MCE filters made by different manufacturers and were far (>10-fold) greater in slope for MCE than for PVC filters. Because at typical room temperatures (21 ± 5°C), there is only a relatively small (~0.07%) gain in absolute air moisture per degree Celsius (Charell and Hawley, 1981; Tables 1 and 2), observations of Charell and Hawley (1981) imply that a change in filter mass (ΔM = M_2 – M_1) should be linearly related to the corresponding change in RH (ΔRH = RH_2 – RH_1) for any given type and dimension of MCE filter.

Using rigorously controlled temperature and RH combinations from the ranges 13–32°C and 60–65%, respectively, Charell and Hawley (1981) demonstrated experimentally that reversible exchange of moisture content between filters and air is determined not by RH per se, but rather by absolute humidity (i.e. total water content of air, defined as
The vapor pressure of water at a particular temperature multiplied by the observed RH at that temperature divided by the atmospheric pressure. In contrast, noting that measured masses of 37-mm Gelman MCE filters exhibited higher correlations with RH (0.985–0.987) than with absolute humidity (0.828–0.906); Beaulieu et al. (1986) derived a model predicting that filter-masses $M_1$ and $M_2$ measured at RH values $RH_1$ and $RH_2$, respectively, are related as $M_2/M_1 = (RH_2 + a)/(RH_1 + a)$, where $a$ is a filter-specific constant defined in terms of the initial rate of weight gain after being removed from a fully desiccated state. They applied this relationship to sets of 21–55 repeated measures of the mass of a total of nine different filters, obtained under conditions of varying RH at unspecified temperatures, and found that it reduced apparent filter-specific standard deviations (~150 µg) of measured filter mass by ~50%. The constant $a$ was evidently estimated for each filter, but relevant details were omitted. The authors did not discuss the discrepancy between their filter-weight model, involving a desiccation-related filter-specific constant, and conclusions by Charell and Hawley (1981) that MCE filter weight is linearly and reversibly determined by absolute humidity, and thus that ‘desiccation of filters prior to weighing has no effect on the final filter weight or the amount of moisture ultimately adsorbed’.

Rigorous moisture-related controls, such as humidity-controlled measurement chambers (Strand et al., 1978), were not routinely implemented historically to measure asbestos-related dust on MCE filters and are cumbersome as well as expensive to implement in current field and laboratory settings. The present study was therefore undertaken to better characterize the accuracy with which fiber-mass determinations for material collected on MCE filters can be accomplished using the relatively simple approach implied by Charell and Hawley (1981) when filter-mass data are collected together with data on temperature and RH, but without RH controls. Our analysis included estimating a hypothesized RH-averaging time interval during which moisture adsorbed onto unloaded filters effectively equilibrates with ambient humidity. Using established physical theory, we then generalized the approximate linear, RH-dependent relationship we observed at a controlled and nearly constant temperature, to predict how unloaded filter mass is expected to be influenced additionally by changes in temperature outside the small temperature range in our study. We provide numerical examples of how these relationships may be applied by the practitioner to estimate dust mass collected on MCE filters under conditions of varying RH and/or temperature.

### METHODS

**Overview**

We weighed each of 40 individually identified, unloaded (i.e. blank) 37-mm MCE filters, weighing each every 2 h for a total of 6 h, yielding four consecutively measured values of mass (M) per filter. We then repeated this series twice, yielding three such
measurement series over about 5 days, recording changes in RH. We then repeated this ‘baseline’ experiment using 25-mm MCE filters. For each filter size, we estimated an approximately linear relationship between 2-hourly change ($D_RH$) in RH and corresponding change ($D_M$) unloaded filter mass. To investigate potential long-term stability of the relationship estimated for 37-mm filters, 14 additional ‘comparison’ sets of 37-mm filter-mass measurements were made over a period of 5 months, each involving 10 or 20 individually identified filters, and 14 corresponding linear regressions were fit. Each of the comparison regressions was compared to the baseline regression fit to the 37 mm data. Details are provided below.

**Filter mass and humidity measurements**

In a laboratory room maintained at ~20–21°C by a typical building-wide ventilation system without any RH control, three replicate series of experiments were done in which a total of 40 unloaded filters were each weighed sequentially using a closed-chamber balance system with 1-µg sensitivity (Sartorius Filter Balance MES-F), then protected from overhead ventilation airflow and dust by placement under an inverted box for 2 h, after which this process was repeated three times. To undertake each such series of measurements, new 0.45-µm MCE filters (Zefon International) were first removed from their original package, set on a foil tripod on a tray next to the balance (Fig. 1), and the filters and balance were covered by a five-sided inverted cardboard box (open side down) to protect the filters from overhead ventilation airflow and dust deposition. After 30 min, the box was removed and temperature and RH were recorded initially and again at 1- or 2-min intervals starting 5 min prior to each weighing period, and then at the time each of the 40 filters was weighed. The 5-min pre-weighing period during which all filters were uncovered and exposed to room air was expected to allow even the first filter weighed to effectively equilibrate with ambient humidity (Winneberger et al., 1963; Charell and Hawley, 1981; Beaulieu et al., 1986). These measurements were made using a VWR Digital Hygrometer/Thermometer (Model 35519-041) with an accuracy/resolution of ±1.5%/0.01% for RH and ±0.4°C/0.01% for temperature (NIST traceable Certification No. 4085-2291280, 28 May 2009; Control Company, Friendswood, TX). The filters were weighed individually within this weighing period, with weights separated only by the time required to: (i) remove each filter from its foil tripod support, (ii) place it on an antistatic device (NRD Model 2U500) for 30 s, then place it within the closed balance chamber, (iii) allow initial weight stabilization (requiring ~7 s), and finally (iv) return the filter to its tripod support. After the last filter was weighed, the inverted box was returned to its original position over the filters until the start of the next set of 40 filter-mass measurements.

In each of three replicate sets of ‘baseline’ experiments, conducted over a period of ~5 days, 40 individually identified 37-mm MCE filters were weighed as described above and immediately re-covered by the inverted box for a holding period of 2 h. Filters were reweighed every 2 h for a total of 6 h, yielding a series of 4 consecutive weights per filter. For each filter, three corresponding values were obtained for change $\Delta M = M_2 - M_1$ in filter mass M (in µg) measured initially ($M_1$) compared to that measured later ($M_2$) at corresponding RH values of $RH_1$ and $RH_2$, respectively, resulting in three corresponding sets of $\{\Delta RH, \Delta M\}$ data points, where $\Delta RH = RH_2 - RH_1$. The baseline experiments described were repeated using 25-mm MCE filters. Finally, to characterize the consistency of the relationship between $\Delta M$ and $\Delta RH$ over longer periods of time, over a 5-month period following baseline data collection a series of 14 ‘comparison’ measurement runs were done, each involving ten or twenty 37-mm MCE filters that were weighed at either two or three time points over an 8-h period.

**Statistical analysis**

For each size filter, the pooled (and, by definition, statistically independent) $\{\Delta RH, \Delta M\}$ data points were modeled by least-squares linear regression (Selvin, 1995), using RH values calculated for

![Fig. 1. Filter-weighing set-up, showing foil tripods on which individually identified filters were each placed prior to and after weighing.](image-url)
filter $j$ ($j = 1, \ldots, 40$) as a linearly interpolated time-weighted average (TWA) value over the period $[t_j - \Delta t, t_j]$, where $t_j$ is the time at which filter $j$ was weighed, and $\Delta t$ is the estimated 'averaging time' used to define each TWA average value of RH. The averaging time $\Delta t$ was hypothesized to reflect the possibility that filters each may absorb moisture from and release moisture back into air over some effective duration of time that is determined by average filter thickness, density, and absorptivity. The averaging time $\Delta t$ thus represents an effective interval over which humidity was assumed to be absorbed into the MCE filters. The value of $\Delta t$ was (for all filters combined) estimated by numerically maximizing the coefficient of determination ($R^2$) for the resulting estimated linear model $\Delta M = a + b \Delta RH$, fit to abscissa values ($\Delta RH = RH_2 - RH_1$) each defined conditional on a corresponding specified value of $\Delta t$ as explained above. Values of $\Delta t$ were examined over the range $0 \text{ min} \leq \Delta t \leq 5 \text{ min}$, using 0.1-min intervals.

To assess potential departure from a strictly linear relationship between $\Delta M$ and $\Delta RH$ per se over the RH range experienced, the residuals ($\Delta M_{\text{res}}$) about the linear fit obtained to the baseline 37-mm \{$\Delta RH, \Delta M$\} data were examined in relation to initial RH ($RH_1$), and to values of average RH defined as $(RH_1 + RH_2)/2$. A significant positive $RH_1$-related trend ($\Delta M_{\text{res}} = a' + b' RH_1$) was detected (see Results). To improve the final regression of $\Delta M$ on $\Delta RH$, this trend was removed by subtracting from $\Delta M$ all negative residuals ($\Delta M_{\text{res}}$) estimated at relatively low values of $RH_1$. For each filter size, standard $F$-tests from analysis of covariance for linear regression (ANOCOVAR) (Selvin, 1995) were used to analyze slope and intercept homogeneity in regressions of data sets analyzed separately by day and also by time interval (2, 4, or 6 h) between initial and final weighing of a filter.

As mentioned above, a total of 14 comparison sets of 37-mm filter-weight data were obtained over a 5-month period to characterize long-term consistency of corresponding \{$\Delta RH, \Delta M$\} regressions obtained with that estimated from the 37-mm baseline data. The estimated values of $\Delta RH$ slope ($b$) and $\Delta M$ intercept ($a$) for each of the comparison data set regressions were compared to those estimated from baseline data using standard $F$-tests from ANOCOVAR (see above). Outliers from fitted regressions were assessed by nested $F$-test (Selvin, 1995). Calculated $P$ values $< 10^{-10}$ were reported as $\sim 0$. All calculations were done using Mathematica® 7.0.1 (Wolfram Research, 2010) and related RiskQ software (Bogen, 2002).

RESULTS

Baseline data obtained using 37-mm filters are summarized in Table 1. Associated RH data plotted in Fig. 2a indicate a strongly periodic pattern of ventilation-driven variation in humidity, with a coefficient of variation (CV) of 8.7% about an overall mean RH value of 56.2%. Over the corresponding cumulative total period of $\sim$24 h during which filter masses were measured, there was substantially less variation in temperature, which maintained a mean value ($\pm 1$ SD) of $21.0 \pm 0.3^\circ C$ (CV = 1.3%), and a 95% confidence interval (CI) (20.5–21.6°C) equal to approximately $\pm 0.5^\circ C$ about the mean temperature.

A $\Delta M$ range of approximately $\pm 200 \mu g$ was observed in the raw 37-mm baseline \{$\Delta RH, \Delta M$\} data, indicating that filter mass varied by less than $\pm 0.5\%$ over time (Table 1, Fig. 3). The linear fit obtained to these data is highly predictive ($n = 360$, $R^2 = 0.954$), with an approximate 95% CI of $\pm 32 \mu g$ for a new single observation. After removing one significant outlying point ($P < 10^{-5}$), recalculation of $\Delta RH$ using an optimized lag interval of $\Delta t = 1.0 \text{ min}$ improved the fit obtained: $\Delta M = a + b \Delta RH$.

![Fig. 2.](image-url) RH (as %) in relation to time over which measures were made of weights of individually identified MCE filters of diameter (a) 37 mm and (b) 25 mm.
with estimated parameters (±1 SE) \( a = 4.61 (±0.72) \mu g \) and \( b = 11.6 (±0.11) \mu g \%^{-1} \) (\( n = 359, \ R^2 = 0.967, 95\% CI = ±27 \mu g \)). Over the range of \( R^2 \) values examined, \( R^2 \) was an inverted, approximately parabolic (i.e. unimodal, and thus clearly meaningfully maximized) function of \( \Delta \tau \). A significant positive linear trend (\( P < 10^{-8} \)) was found between \( \Delta M \) residuals (\( \Delta M_{\text{res}} \)) and 120 values of \( RH_1 < 53\% \). To adjust for this trend, values of \( \Delta M \) observed at \( RH_1 < 51\% \) (associated with negative values of \( \Delta M_{\text{res}} \)) were first replaced by the corresponding values \( \Delta M^* = \Delta M - (-173 + 3.40 \, RH_1) \). This adjustment resulted in a fit shown in Fig. 3 (\( n = 359, \ R^2 = 0.971, 95\% CI = ±25 \mu g \)) given by \( \Delta M = a + b \, \Delta RH \), with estimated parameters (±1 SE) \( a = 5.74 (±0.68) \mu g \) and \( b = 11.9 (±0.11) \mu g \%^{-1} \). It is unknown if the \( \Delta M^* \) adjustment estimated can meaningfully be applied to \( RH_1 \) values below those (<46%) experienced during these baseline experiments (Fig. 2a).

Baseline data obtained using 25-mm MCE filters are summarized in Table 2, and associated RH data plotted in Fig. 2b again show much greater relative variation in RH (CV = 8.7%) than in temperature (CV = 1.3%). Over the corresponding cumulative total period of ~24 h during which filter weights were measured, temperature varied about a mean (±1 SD) of 20.9 ± 0.3°C, with a 95% CI of 20.4–21.6°C. Corresponding \{ \Delta RH, \Delta M \} data were fit conditional on \( \Delta \tau = 1.0 \) min, based on results obtained for 37-mm control filters. A \( \Delta M \) range of approximately ±100 \mu g was observed in this data set, again indicating that filter mass varied by less than ±0.5% over time (Table 2, Fig. 3). The resulting estimated linear-fit, with parameters (±1 SE): \( a = 3.94 (±0.38) \mu g \) and \( b = 6.58 (±0.05) \mu g \%^{-1} \), is shown in Fig. 3 (\( n = 320, \ R^2 = 0.981, 95\% CI = ±13 \mu g \)). Because this fit is even better than the final baseline fit obtained for 37-mm filters, it was not further optimized.

Analysis of covariance showed that values of \( \Delta M \) versus \( \Delta RH \) slope (\( b \)) estimated for data subsets specific to each interweight duration used (2, 4, or 6 h) did not differ significantly (\( P > 0.10 \)) for either 37- or 25-mm filters. Corresponding intercepts also did not differ for the 37-mm filters (\( P = 0.13 \)) but did (albeit only over a range of 2.4–5.5 \mu g) for the 25-mm filters (\( P = 0.000042 \)). Slopes similarly estimated for filter-size-specific control data subsets obtained on each of three separate days differed significantly, albeit with CV values that were relatively small, for 37-mm filters (\( p = 0.11 \)) and for 25-mm filters (\( P = 0.015, \ CV = 0.034 \)).

Table 3 summarizes linear regression results for the 14 comparison sets of \{ \Delta RH, \Delta M \} data, and corresponding ANOCOVAR comparisons to the linear fit obtained for baseline data previously obtained for 37-mm MCE filters. Only 1 of the 14 linear fits included an estimated slope that did not differ significantly from zero (Table 3, Run 13), and only 2 of the 14 estimated intercepts were significantly negative (Table 3, Runs 6 and 13). Only 2 of the 13 non-zero-slope fits had estimated slopes that clearly differed significantly (\( P > 0.01 \)) from that estimated for baseline filters, and both of these slope-inconsistent fits had negative estimated intercepts. Most (8 of 11) of the slope-consistent also had intercepts consistent (\( P > 0.05 \)) with that previously estimated for baseline filters. Of the slope-inconsistent fits, the slope from Run 7 differed most significantly from the baseline estimate, exceeding the latter by a factor of 1.5. The absolute difference between the Run 7 and baseline fits is \( 7.72 \mu g - 6.01 (\mu g \%^{-1}) \Delta RH \), which, for example, amounts to ~52 or 68 \mu g if \( |\Delta RH| = 10\% \).

Our study results indicated that >96% and >98% of \( \Delta M \) variance measured in 37- and 25-mm MCE filters, respectively, at RH values in the range of ~50–70% was explained by a linear relationship \( \Delta M = a + b \, \Delta RH \), independent of RH magnitude per se. Conditional on the conclusion by Charell and Hawley (1981) that unloaded filter weights are linearly and reversibly determined by absolute humidity (H), the fixed physical relationship between H, RH, and temperature (\( T \)) (see Lowe, 1977; Buck, 1981) implies that the linear relationship between \( \Delta M \) and \( \Delta RH \) for an unloaded MCE filter can be re-expressed in terms of absolute humidity (H) and temperature (\( T \)) as:
Table 3. Consistency of linear fits, $\Delta M = a + b \Delta RH$, estimated for 14 comparison sets of 37-mm MCE filters weighed over a 5-month period, with those previously estimated for baseline data obtained over a period of 3 days for 359 filters of the same type.

<table>
<thead>
<tr>
<th>Run</th>
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<td>Slope, $b$ (g %$^{-1}$)</td>
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$^a$—, ‘not applicable’; $\Delta$, difference with respect to time interval of measurement; M, MCE filter mass (micrograms); n, (total number of filters) $\times$ (total number of time intervals); p, two-tail significance of deviation from the null hypothesis of independence ($b = 0$); $R^2$, fraction of $\Delta M$ variance explained by the fitted regression; RH, relative humidity (%); SE, standard error of estimated regression parameter $a$ and $b$ estimated by least-squares linear regression.

$^b$Results from analysis of covariance for linear regressions (ANOCOVAR). $P$ value for different intercepts is meaningful and listed only if that for different slopes is significant ($P \leq 0.05$).

$^c$Estimated intercept is significantly negative ($P \leq 0.05$, by t-test).

$^d$Values of $\Delta M$ were pre-adjusted to $\Delta M^* = [RH_1 > 51\%$, $\Delta M = a + b' RH_1]$ with $a' = -173$ and $b' = 3.40$, to account for a significant ($p = ~0$) positive association between $\Delta M$ and $RH_1$ for $RH_1 < 53\%$ exhibited among the baseline data.

$^e$One data point dropped as a significant outlier from the estimated linear fit ($P = 0.023$).

\[
M_2 = M_1 + a + b \Delta RH \\
= a + [b/h(T_o)](H_2 - H_1) \quad \text{if} \quad T_1 = T_2 = T_o
\]

\[
M_2 = M_1 + a + [b/h(T_o)]h(T_2) \quad RH_2 \\
- h(T_1)RH_1 \quad \text{generally,}
\]

in which $H$ = absolute humidity (i.e. density of water vapor in air) in units of mg per liter (l), $T$ = temperature in degree Celsius at time $t$, $T_o$ = is the average temperature attained in our study (21°C), and $h(T) = H/RH$ in units of mg per percent per liter expressed as a function of temperature $T$ in degree Celsius, where approximations for $h(T)$ are readily derived from corresponding approximations for vapor pressure in mbar as a function of $T$ (e.g. Lowe, 1977; Buck, 1981) by applying the gas law equation using the gas constant for water vapor (4.615 mbar m² kg⁻¹ Kelvin⁻¹). For example, we find the approximation

\[
h(T) = \left( \sum_i c_i T_i \right) / 100, i = 0, 1, \ldots, 4,
\]

which to have <1% relative error in the range 0°C $\leq T \leq 50°C$ using the constants: \{c_0, c_1, c_2, c_3, c_4\} = \{4.851, 0.3326, 0.01065, 1.332 $\times 10^{-4}$, 2.888 $\times 10^{-6}$\}. Note that this relationship implies an approximately constant and fairly small (1.06-fold) relative variation in $H$ per °C within the typically relevant temperature range 16°C $\leq T \leq 26°C$. Equations (2) and (3) together imply that dust mass collected on an MCE filter can be estimated as

\[
\text{Dust Mass} = M_2 - [M_1 + a + b(h_2 RH_2 - h_1 RH_1)],
\]

in which the temperature-dependent ratios $h_j$ of relative variation in $H$ are defined as $h_j = h(T_j)/h(T_o)$ with $j = 1, 2$, and RH is calculated as a TWA value over a 1-min averaging period preceding measures $M_j$ (where $M_j$ and $a$ must have the same units).
DISCUSSION

Consistent with observations from previous studies, results from the baseline experiments conducted showed a substantial amount of variation in measured masses of hundreds of individually identified 37- and 25-mm Zefon International MCE filters, repeated every 2 h under typical office-building AC/ventilation conditions involving relatively low temperature variation (CV = ~1.3%) and larger, uncontrolled, periodic humidity variation (CV = ~8.7%). Consistent with observations by Charell and Hawley (1981), it was observed that when measured at a nearly constant temperature, ΔM was approximately linearly proportional to ΔRH for both sizes of MCE filter investigated ($R^2 > 0.96$) as expressed by equation (1), in which parameters $a$ and $b$ depend on filter size. In this study, it was determined that the best linear fit was obtained when RH is calculated as a TWA value over an averaging period of 1 min preceding each corresponding measure of filter mass, which is also the duration required for MCE filter moisture to attain 50–90% equilibration with ambient water vapor (Winneberger et al., 1963; Charell and Hawley, 1981; Beaulieu et al., 1986). The RH range we investigated (46–70%) is ~5-fold greater than that (60–65%) investigated by Charell and Hawley (1981).

For both the 25-mm and the 37-mm baseline data sets, the estimated, relatively small intercept term ($a$) of this linear model was determined to differ from zero highly significantly (p = ~0.0), indicating that an unidentified positive bias (equal to several micrograms) was associated with each repeated weight measurement per se in this series of measurements. The same was generally true of the 14 comparison data sets, the estimated intercepts for which were significantly negative in only two cases (Table 3). The positive estimated intercepts may indicate that an unidentified positive bias (equal to several micrograms) was associated with each repeated weight measurement per se in this series of measurements.

Although the linear association between ΔM and ΔRH estimated for baseline 37-mm MC filters was generally consistent with individual linear models fit to each of 14 comparison{ΔRH, ΔM} data sets gathered over a subsequent 5-month period, fits to the later data sets included several that differed significantly in slope and/or intercept (albeit by small magnitudes) compared to the fit obtained to the control data (Table 3). Consequently, the accuracy of estimated dust mass on MCE filters measured under varying RH conditions may be improved slightly by obtaining at least 20 {ΔRH, ΔM} measures for individually identified blank ‘concurrent control’ filters processed in parallel with filters on which dust is collected. If one or both regression parameters ($a$ and $b$) estimated from the concurrent data differ significantly from those we report for our baseline data for 25- or 37-mm Zefon International MCE filters, the concurrent control parameter estimate(s) should be used; otherwise, the estimates we report can be used.

The same ‘concurrent control’ procedure should be applied if dust mass is measured using MCE filter sizes other than those used in this study, given the size dependence of regression parameters estimated in this study. The same recommendation applies if MCE filters used are not manufactured by Zefon International since filters from different sources may exhibit very different linear patterns of filter-air moisture equilibration (Charell and Hawley, 1981). This recommendation is similar to, but (by providing explicit default fits for 25- and 37-mm Zefon International MCE filters, and data pertaining to the consistency of these fits over many months) more specific than, earlier recommendations that concurrent unloaded MCE filters can improve estimates of dust mass obtained using MCE filters (Lowry and Tillery, 1979; Charell and Hawley, 1981).

Conditional on the conclusion by Charell and Hawley (1981) that unloaded filter weights are linearly and reversibly determined by absolute humidity, equations (3) and (4) are expected to predict the joint effect of RH and temperature variation on unloaded filter mass. Numerical examples summarized in Table 4 illustrate how equations (3) and (4) can be used by an industrial hygienist to estimate dust mass collected on an MCE filter, using measures of filter mass ($M_1$ and $M_2$), RH (RH$_1$ and RH$_2$), and temperature ($T_1$ and $T_2$) made before and after dust collection (respectively). The examples show how estimates for parameters $a$ and $b$ presented above for 25- or 37-mm Zefon International MCE filters may be used to apply equation (4) to estimate dust mass collected on these filters. As mentioned above, significantly different estimates of parameters $a$ and/or $b$ may be obtained using the same type of blank filters weighed concurrently with another such set.
on which loaded dust mass is to be estimated using M, T, and RH data collected before and after dust loading. If this occurs, or if MCE filters of other sizes or filters made by other manufacturers are used, then new estimates obtained from such concurrent measures should be used in equation (4) to estimate dust mass.

The results obtained indicate the size of potential error associated with EPA NAAQS recommendations that PM$_{2.5}$ mass measures on MCE filters be done at mean temperatures of 20–23°C varying by ≤2°C day$^{-1}$ and with RH controlled to within 5% of that at which samples were taken (see Introduction). Whereas these recommendations are consistent with temperatures maintained in our study, we allowed ambient RH to vary in a closed, air-conditioned laboratory room in a range of ~50–70% (Fig. 2), and thus at all times substantially exceeded the ideal ‘30 to 40%’ range recommended by EPA. Under these conditions in our study, >96% of observed ΔM variance in unloaded 25- and 37-mm Zefon International MCE filters was explained as an approximately linear function of ΔRH independent of RH magnitude per se. The 95% confidence limits we report on linear relationships for ΔM (Fig. 3) imply that the EPA recommendation that RH be controlled ‘to within 5%’ may lead to errors as great as ~60 and 120 μg for the 25- and 37-mm MCE filters used, respectively, and the significance of errors of this magnitude would depend on the total mass of dust collected on the filter. The methods and results described herein can thus be used for improved estimation of low levels of dust mass on MCE filters, particularly those analyzed for both dust mass and fiber content.

**Table 4. Numerical examples illustrating applications of equations (3) and (4) to estimate dust mass collected on Zefon International MCE filters$^a$**

<table>
<thead>
<tr>
<th>Example</th>
<th>Filter size (mm)</th>
<th>Filter mass, M (mg)</th>
<th>Temperature, T (°C)</th>
<th>RH (%)</th>
<th>Absolute humidity ratio, $h_j$ (unitless)</th>
<th>Dust mass (μg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>37</td>
<td>46.103</td>
<td>21.0</td>
<td>61.70</td>
<td>1</td>
<td>89.175</td>
</tr>
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<td>46.103</td>
<td>21.0</td>
<td>71.21</td>
<td>1</td>
<td>316.227</td>
</tr>
<tr>
<td>3</td>
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<td>46.103</td>
<td>19.0</td>
<td>61.70</td>
<td>1.124</td>
<td>309.722</td>
</tr>
<tr>
<td>4</td>
<td>37</td>
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<td>19.0</td>
<td>71.21</td>
<td>1.124</td>
<td>532.815</td>
</tr>
<tr>
<td>5</td>
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<td>61.70</td>
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<td>38.013</td>
</tr>
<tr>
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<td>71.21</td>
<td>1</td>
<td>163.559</td>
</tr>
<tr>
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<td>19.0</td>
<td>61.70</td>
<td>1.124</td>
<td>159.963</td>
</tr>
<tr>
<td>8</td>
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<td>19.0</td>
<td>71.21</td>
<td>1.124</td>
<td>283.320</td>
</tr>
</tbody>
</table>

$^a$Dust mass calculated using equation (4) with filter mass in micrograms, and parameters \(\{a, b\} = \{5.74 \text{ μg}, 11.9 \text{ μg g}^{-1}\}\) for 37-mm filters or \(\{a, b\} = \{3.94 \text{ μg}, 6.58 \text{ μg g}^{-1}\}\) for 25-mm filters.

**REFERENCES**


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