ADVANCING THE UNDERSTANDING OF PM FILTER MASS STABILITY: UNVEILING THE INFLUENCE OF HUMIDITY AND TEMPERATURE

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Abstract

The intricate interplay between humidity and temperature on PM filter performance has remained a subject of profound scientific interest. By employing fully-automated weighing method and a two-factor regression model, the study aimed to evaluate mass variations of unloaded PM filters under diverse humidity and temperature conditions. Existing guidelines, as posited by the US EPA and the European Standard (EN), have demonstrated a pronounced disparity. The US EPA advocates a narrow humidity range of 30–40% RH, while the EN suggests a broader range of 40–50% RH. The results of this investigation confirm the US EPA's guidelines, which exhibited superior filter mass stability under varying humidity. In controlled stable temperature conditions of 20°C, the research unveiled significant variations in filter mass as RH% increased from 50% to 55%. It has been proven that under stable temperature conditions (20°C) inside a weighing robot, an increase in relative air humidity from 50% to 55% results in filters mass addition by 15 µg (Q, quartz fibre filters); by 93 µg (PTFE filters); by 9 µg (G, glass fibre filters); by 112 µg (PA, polyamide filters) and by 20 µg (PC, polycarbonate filters). Further exploration of the conditioning ranges prescribed by the EN 12341:2014 standard, the study highlighted glass fibre filters as being notably stable, while polyamide membrane filters posed intriguing challenges potentially related to ionization insufficiencies during the equilibration stage. These findings bear significance for filter manufacturing and conditioning protocols, potentially enhancing the precision of air quality monitoring practices.

Keywords: PM filters; mass deviation; mass measurement, weighting, robotic weighing system

1. Introduction

The methodology for establishing PM (particulate matter) mass concentrations involves PM sampling, transportation to the analysis site and mass determination through weighing methods, with the gravimetric method widely recognized as the most precise one (Barba-Lobo et. al., 2022). Proper conditioning of PM filters before and after exposure significantly impacts the accuracy of PM mass concentration (NIST 2019; Lacey and Faulkner 2015; US EPA 2008). Particular attention is required with respect to weighing conditions, conditioning periods and filter handling to minimize potential weight loss. Undesirable phenomena such as water condensation or the loss of PM-bound volatile components have been noted (Barba-Lobo et. al., 2022; Widziewicz-Rzonca and Tytła 2020; Montgomery et al., 2015). To mitigate those effects, PM filters should be conditioned under stable humidity and temperature conditions (Raynor et al., 2011; Rasmussen, 2010; Bureau Veritas UK, 2009). Ensuring filter equilibration during pre-sampling and post-sampling weighing is critical for precise PM net mass loading calculation and subsequent mass concentration establishment.

The European Standard EN 12341:2014 specifies different filter equilibration requirements as compared to US EPA guidelines. The CFR (40 CFR Part 50, Appendix L) provides general conditioning specifications for PM2.5 sample filters, with recommended temperature and humidity ranges. EN 12341:2014 specifies alternate ranges. Given the hysteresis effects on filter material, the choice between those conditioning requirements and the extent to which temperature and humidity affect mass deviations become pertinent issues. Notably, EN 12341:2014 identifies air moisture content (Relative Humidity, RH%) as the most relevant factor affecting mass measurements for unsampled filters, impacting both unloaded and loaded filters. Different filter batches, and even filters within the same batch, may respond differently to changing humidity conditions (Raynor et al., 2011; Charell and Hawley, 1981). Air humidity impacts not only the equilibration between pre- and post-sampled filters but also varies among filters of different materials (Brown et al., 2006). Therefore, maintaining stable equilibration conditions during conditioning and weighing is essential for accurate mass determinations. Unfortunately, achieving such conditions in practice is challenging, as various factors, including direct sunlight, air currents, vibration, temperature fluctuations, open doors, static electricity and even breathing, can significantly affect the weighing process (Harris 2019; Su et al., 2008; US EPA 2008). This emphasizes the preference for solutions that quickly establish the best equilibration conditions, reducing PM mass uncertainty. Traditional manual balances often face limitations in addressing these issues, such as limited speed control and programming capabilities (Ren et al., 2013; Frohlich et al., 2009). Only a few research groups have attempted to compare PM mass results using automated gravimetric measurements and traditional manual balances (Presler-Jur et al., 2016; Ogden et al., 2015; Carlton and Teitz 2002; Allen et al., 2001). The automation of mass standard calibration

processes caused lower uncertainty when using automatic mass comparators, further improving repeatability. Recent developments include Robot Weighing Systems (RWS), such as the PFS-ONE Horiba (PFS-ONE, Austria), Mettler Toledo (2021) Switzerland, AWS-RE1 Comde-Derenda GmbH, Germany and Automatic Weighing System by Radwag, Poland. These systems provide highly reliable and precise PM weight measurements. Professional devices like these feature climate control units for temperature regulation, along with evaporators to maintain specified relative humidity levels precisely.

The automation of weighing processes offers a key advantage by eliminating the human factor. However, widespread adoption of these systems hinges on their comparability to manual weighing in various conditions, as discrepancies can lead to significantly different results. The EN12341:2014 standard lacks recommendations regarding the preferable gravimetric method for filter mass measurements, with the only requirement being to maintain calibration uncertainty within the range of ±25 µg. Notably, advanced systems like PFS-ONE (Horiba) or UMA 2.4 YF.C (Radwag) achieve a remarkable 0.2 µg uncertainty, outperforming manual balances, which typically exhibit slightly higher uncertainties (e.g., MYA 5.4 YF model (Radwag) at approx. 0.8 µg), indicating that robotic weighing offers superior accuracy. Different regulatory agencies have diverse requirements regarding approaches, balance performance and filter conditioning. Comparing these requirements, including ambient conditions, electrostatic discharge, filter handling and storage, is crucial for precise, repeatable and comparable gravimetric measurements.

This study aimed to investigate discrepancies in filter mass measurements under varying conditioning parameters using an automatic weighing system (RB 2.4 YF, Radwag). It included an adaptation of manual weighing procedures to ensure comparability, mass measurement of unloaded filters using the automatic system and an analysis of equilibration conditions' influence on mass measurements for various filter types (quartz fibre, glass fibre, Teflon, polyamide and polycarbonate, Table S1 in the Supplement) through two-factor regression analysis.

Testing filter mass deviations using robotic systems is rarely documented in the literature, underscoring the importance of such measurements (Frohlich et al., 2009; Su et al., 2008). Only one study by L'Orange Ch. (2021) was found, which explored PM measurements using an Automated Air Analysis Facility (AIRLIFT) system and also examined the impact of humidity. While a summary of existing studies on PM and filter measurements under changing conditions is provided, no publications were found specifically addressing filter mass deviations under varying conditioning parameters with automatic weighing systems. This paper presents mass deviations due to variable conditioning criteria, details the weighing procedure's methodology and its adaptation for automation, reports filter mass measurements and deviations, and discusses sources of uncertainty in automatic weighing.

2. Materials and Methods

2.1. Conditioning, weighing procedure, and weighing environment description

In this study, weight measurements utilized the RB 2.4Y.F robotic filter weighing system by Radwag Electronic Scales, Poland (Fig. S1 in Supplementary materials). Complying with standard guidelines, the RB 2.4Y.F system, based on MYA 4Y microbalances, offered a readability of 1 µg and a maximum capacity of 2.1 g, meeting AQG-Air Quality Guidelines (WHO, 2021) specifications and detection requirements for various filter masses. The system maintained constant climatic conditions within the chamber to ensure proper filter equilibration, aligning with EN12341 standard criteria for weighing filters, as employed in this study. Upon receipt, each filter underwent examination for discoloration, pinholes and surface non-uniformity; filters with defects were rejected in accordance with the standard. Weighing commenced 48 hours after the conditioning process began, with subsequent weightings performed after an additional 12 hours. The unloaded filter mass was determined as the average of two separate measurements, rejecting filters with measurement differences exceeding 40 µg. RMCS software facilitated calibration procedures, weighing commands, and overall process management. The RB 2.4Y.F robotic system autonomously recorded air humidity and temperature conditions (temperature resolution: 0.001°C; humidity resolution: 0.001% RH). A filter deionization system was employed to stabilize electrostatic charges during measurements. The system ensured specific climatic conditions during conditioning and weighing with uncertainties of ± 0.5°C (1-hour average) for temperature and ± 2.5% (1-hour average) for humidity within the ranges of 20-22°C and 45-50%, respectively. Various filter types were tested, including those known for their resistance to weight deviations (quartz fibre, glass fibre) and those susceptible to variations in the weighing environment (Teflon, polyamide, polycarbonate filters) (Table S2). The primary study objective was to determine weighing precision (filter mass deviation) during repeated weightings. Testing encompassed transitions from the extremes of recommended humidity and temperature ranges (~35–50%, ~19–21°C) to middle values (defined as optimal in the EN123401:2014 standard), such as filter mass deviation when shifting from 45% RH to 50% RH. The study also examined in what way increasing balance time positively impacted this deviation. Experimental schemes illustrated in Figures 1 and 2 evaluated the influence of variable equilibration conditions on mass measurement precision. In the first scheme (Fig. 1), stabilization occurred under different conditions than weighing, while the second scheme (Fig. 2) involved stabilization under the same conditions as weighing. "Stabilization" denoted a return to conditions specified in the mid-point of the optimum temperature/humidity range defined in the EN 12341:2014 standard (50% RH and 20°C). Variation testing in Scheme No. 1 (Fig. 1) slightly extended to humidity ranges outlined in US EPA regulations (US EPA, 2016) but
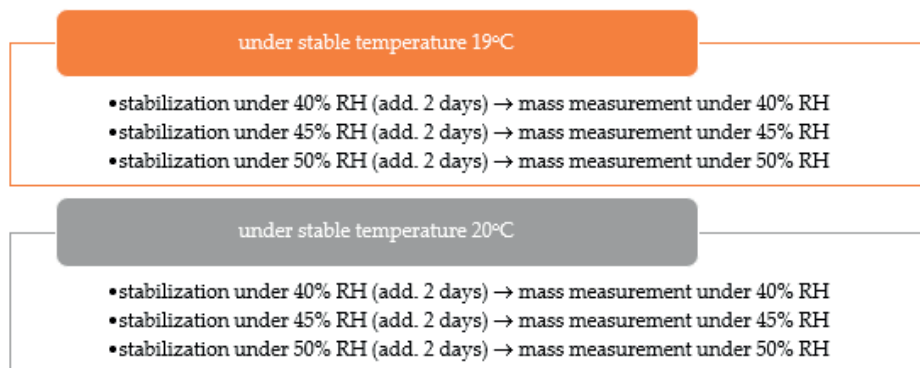
remained optimal for filter equilibration. Under scheme No. 1, stabilization was performed under 50% RH and three separate temperature points (19°C, 20°C, 21°C), while mass measurement occurred under 35%, 45% and 50% RH. Each experiment involved ten filters of each type (Q-quartz fibre; PTFE-Teflon; G-glass fibre filters; PA-polyamide membrane; PC-polyester (polycarbonate) membrane).
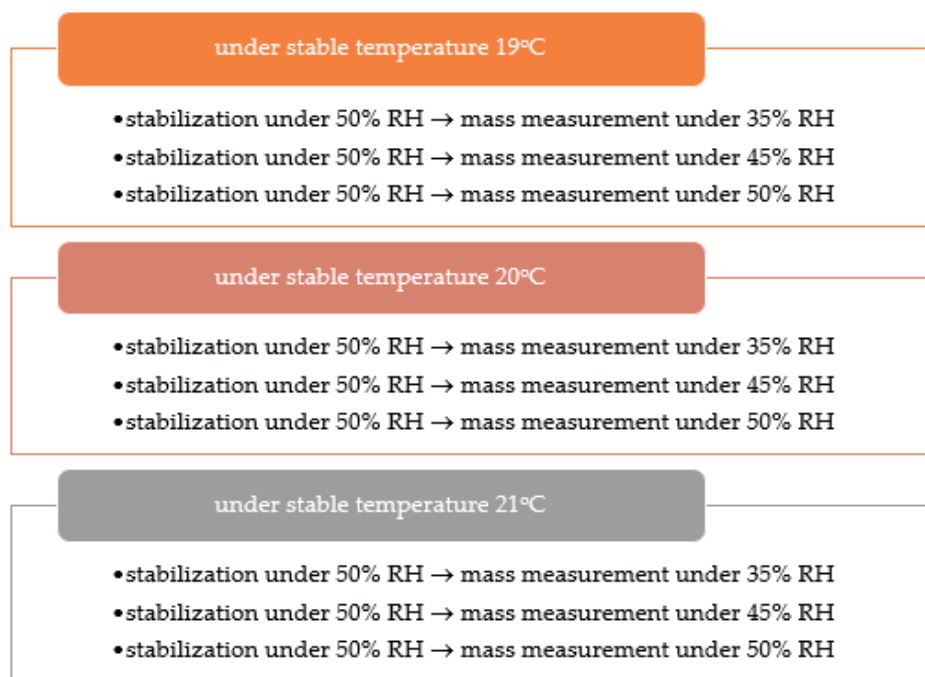
Figure 1. Experimental scheme No. 1 for testing mass deviations of the unloaded filters depending on the set temperature (19°C, 20°C, 21°C) and humidity (35%, 45%, 50%) variations where stabilization is being performed in different conditions compared to weighing. A total of 57 weighing cycles have been performed for each type of filter media (10 filters of each type).

In the later stages of the research, experimental scheme No. 1 was modified to create experimental scheme No. 2 (Fig. 2), introducing additional filter stabilization periods before actual mass measurements. Typically, the first weighing occurs 48 hours after starting the conditioning process, but in scheme No. 2, this period was extended to 96 hours. Both stabilization and mass measurement now occurred under identical temperature and humidity conditions to assess the impact of a longer stabilization process on mass measurement precision. Fig. 2 illustrates experimental scheme No. 2, focusing on mass deviations of unloaded filters under varying temperature (19°C, 20°C) and humidity (40%, 45%, 50%) settings, with stabilization occurring in the same conditions as weighing. Experimental scheme No. 1 involved a 48-hour stabilization period before initiating the next series of

measurements under altered RH% and temperature conditions, whereas scheme No. 2 included an additional 48-hour period within the robot enclosure area to ensure filters reached a stable moisture state.

under stable temperature 19°C

- stabilization under 40% RH (add. 2 days) → mass measurement under 40% RH
- stabilization under 45% RH (add. 2 days) → mass measurement under 45% RH
- stabilization under 50% RH (add. 2 days) → mass measurement under 50% RH

under stable temperature 20°C

- stabilization under 40% RH (add. 2 days) → mass measurement under 40% RH
- stabilization under 45% RH (add. 2 days) → mass measurement under 45% RH
- stabilization under 50% RH (add. 2 days) → mass measurement under 50% RH

Figure 2. Experimental scheme No. 2 for testing mass deviations of the unloaded filters depending on the set temperature (19°C, 20°C) and humidity (40%, 45%, 50%) variations when stabilization was performed under the same conditions as weighing. For the temperature of 19°C, a total of 39 weighing cycles were performed (10 filters for each type) and at the temperature of 20°C, the number of cycles was 28 (10 filters of each type).

Additionally, repeatability measurements of balance indications were conducted during 29 measurements of the standard mass (a stainless steel weight shaped like a Mercedes badge with a quartz filter) under varying RH% and temperature conditions. Repeatability measurements of standard mass indications are presented in Table S3 in the Supplementary materials. The total mass of the standard weight and the reference quartz fibre filter ranged from 243.95 ± 0.0022 mg. This means that under conditioning parameters close to those set in the EN12341:2014 standard, considered hypothetically "constant," the mass of quartz filters changed only due to balance indication repeatability error, which fell within the range of ±2.2 µg. This error closely aligned with the device producer's specified repeatability of 1 - 2 µg. To assess quantitatively the influence of temperature and humidity variations within the standard-defined ranges on the mass of each filter type [mg], a two-factorial regression evaluation was performed. Filter mass deviations resulting from temperature and relative humidity variations within the weighing robot were visualized on 3D graphs (Fig. 5a–Fig. 5e). While the analysis covered a wider range of relative humidity (35.67–58.78% RH) and temperature (19.2–20.5°C) than specified in the standard, the changes in filter weight for the standard-provided temperature and humidity ranges (50±5%, 20±1°C) were plotted for easier result comparison.

2.2. Statistical analysis

After weight measurements of each ten filters (from one type), the standard deviation was calculated according to the formula:

$$s = \sqrt{\frac{\sum_{i=1}^{n}\left(x_i - \overline{x}\right)^2}{\left(n-1\right)}}$$ (eq. 1)

where:
n – the number of repetitions (measurements),
x_i – the result of the weight measurement,
x – the average value for n repeated weight measurements.

To study the simultaneous effect of two factors: temperature and humidity on the outcome variable – filter mass, two factorial regression analyses was performed. For this purpose, a statistical package – ''Statistica 13'' was used. The model was created by a linear relationship between dependent variable-filter mass and two or more independent variables - temperature and humidity. Before performing two-factorial regression the linear relationship between the individual variables is tracked. By drawing scatter plots and correlation matrices the linear relationship may be checked between filters mass and each of the independent variables. Since the first assumption was meet, multicollinearity was further tested. Temperature and humidity show a small collinearity, with the r^2 value=0.16. It was also found that residuals are independent and rests were normally distributed (tested based on histogram).

The air-conditioning system controlled the airflow inside the chamber, ensuring stable working conditions, adjusted by heating and cooling. The convection unit controlled the required relative humidity. Due to uncertainty of the temperature and RH% sensor, the given conditions (19–21°C; 35–60% RH) were slightly different than the ones obtained in the course of measurement (19.2-20.5°C; 35.67-58.78% RH). Regression equations (eq. 2–6) were therefore plotted for the following ranges of temperature and humidity: 19.2–20.5°C; 35.67–58.78% RH and the joint effect of those variables on filters mass variability was presented on three dimensional graphs (Fig. 5).

3. Results

Based on the results presented in Table S2, only slight deviations in the mass of the standard weight plus reference filter can be noticed under the equilibration conditions typical for the ranges put into the EN 12341:2014 standard, i.e. 45%–55% RH, and 19°C–21°C. The mass of the standard weight + reference filter changed within the range of 243.95 ± 0.0022 mg~2.2 μg. This means that under

hypothetically "constant" equilibration conditions, the mass of quartz filters was changing only as a result of balance indication error, and this error was within the range of ± 2.2 µg.

Temperature and humidity conditions in the weighing chamber and the accompanying fluctuations in filter weight under those conditions have been presented in Table 1 and Fig 3. It is worth noting that the pre-set temperature and humidity values (19°21°C and 35°55% RH), were slightly different as compared to the measured one (Table 1). This is easily observable when comparing the set temperature and humidity conditions in experimental schemes No. 1 and No. 2 and the feedback from the sensors (Table 1,2; Fig. 3, 4). It must be remembered that the robotic system, although maintaining stable operating conditions in certain ranges, gives the actual temperature and moisture slightly different to those set as input variables. The readings will depend on the distribution of the conditioning parameters inside the weighing chamber, as well as the location and uncertainty of the sensors coupled with the weighing device.

Table 1. Descriptive statistics of temperature and humidity conditions inside the weighing robot over filter equilibration for various types of filter material (conditions: 19.2–20.5°C; 35.67–58.78% RH).

Parameter	RH [%]	Temperature [°C]
mean	45.60	19.81
median	46.88	19.79
std. dev.	3.90	0.30
N	4218	4218
Min.	35.67	19.204
Max.	58.78	20.518
25%	42.34	19.502
75%	48.4	20.11

The computational base for Scheme No. 1 contained 4,218 important measurements and the coverage of the results for the data series ranged from 86% to 96%. It was estimated that the standard deviation of RH% was 3.9%, while for the average temperature of 19.81°C it was ca. ±0.3°C. The average humidity was 45.60%, while the average temperature accounted to 19.81°C (Table 1). As shown in Figure 3, except for polyamide membrane filters, all of the tested filter materials were characterized by a rather small weight deviation under temperature and RH% conditions defined by Scheme No 1. The average deviations of the filter mass under those conditions amounted to: 146 ± 0.32 mg (quartz fibre filters); 135.59 ± 3.19 mg (PTFE filters); 91.91 ± 0.51 mg (glass fibre filters); 101.09 ± 7.13 mg (polyamide membrane); 35.22 ± 0.75 mg (polyester membrane – polycarbonate). The largest deviation was observed for the polyamide membrane, which probably stemmed

from insufficient ionization of the filter surface at the stage of stabilization. The influence of variable equilibration conditions on the weight deviations of filters depended on the type of filter material. In case of quartz fibre, glass fibre and polycarbonate filters, mass measurements met the condition of normal distribution, while the occurrence of outliers and extremes in the measurements series was found for PTFE and polyamide filters (Fig. 3).
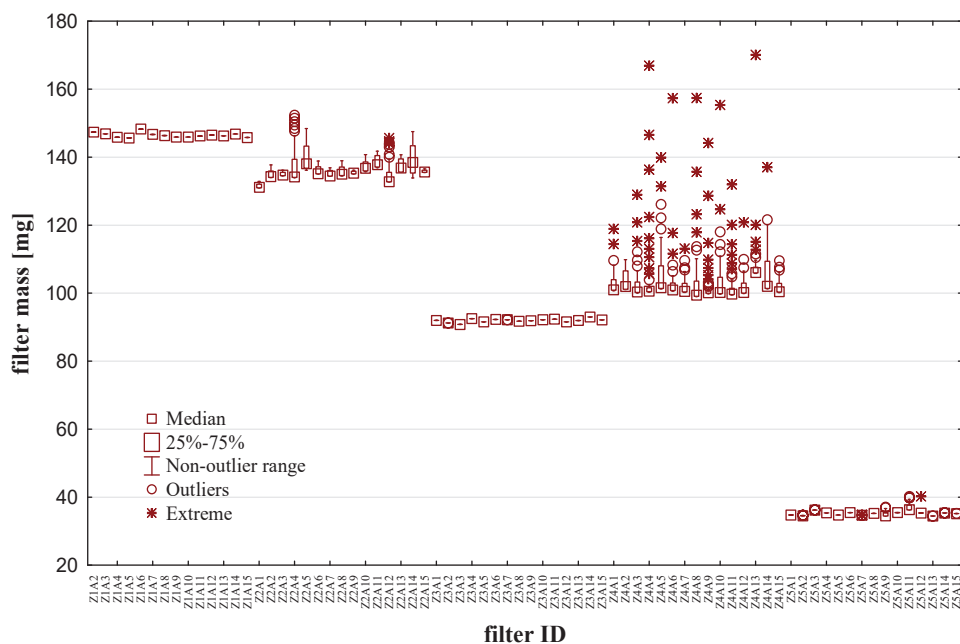
Figure 3. The influence of equilibration conditions on the mean and weight deviations of unloaded filters [mg] (conditions: 19.2–20.5°C; 35.67–58.78% RH). *Note: ID – Z1A2 to Z1A15 quartz fibre; ID – Z2A1 to Z2A15 PTFE; ID – Z3A1 to Z3A15 glass fibre filters; ID – Z4A1 to Z4A15 polyamide membrane, ID – Z5A1 to Z5A15 polyester (polycarbonate) membrane.

In the second part of the research (experimental scheme No 2), to check whether extending stabilization improves mass measurement precision, both stabilization and measurement of the filter mass was carried out at identical temperature and humidity. The descriptive statistics presenting conditioning parameters during this stage of the research are summarized in Table 2. The standard deviation of the relative air humidity, in the scheme No 2, was 2.34%, while in case of temperature these fluctuations amounted to approximately ±0.09°C. With an average humidity of 40.63%, the 25% percentile was 38%, while with an average temperature of 19.75°C, the 75% percentile was 19.79°C.

Table 2. Descriptive statistics of temperature and humidity conditions inside the weighing robot over filter equilibration for various types of filter material (conditions 19.36–19.81°C; 38–43% RH).

Parameter	RH [%]	Temperature [°C]
mean	40.63	19.75
median	40	19.78
std. dev.	2.34	0.09
N	1989	1989
Min.	38	19.36
Max.	44	19.81
25%	38	19.76
75%	43	19.79

The influence of equilibration conditions on the mean weight and deviations of unloaded PM filters [mg] were tested against temperature and relative humidity variability in the ranges of 19.36–19.81°C and 38–43% RH. Obtained data consisted of 1,989 important data. Some results from this database were rejected (missing data in the box plot), since mass difference between first and second weighing of PTFE and polyamide filters failed to fulfil the requirement ≤40 µg, recommended by the standard (Fig. 4). The influence of equilibration conditions on the mean weight and mass deviations of filters [mg] for each of the tested filter media was characterized by the stability of the mass measurements. It was observed that in the described ranges both humidity , and temperature fluctuations have little effect on the filters mass variations.

Within the ranges 19.36–19.81°C; 38–43% RH, the mean and standard deviation of filters mass amounted to: 149.48 ± 0.47 mg (quartz fibre filters); 135.07 ± 0.63 mg (PTFE filters); 91.09 ± 0.86 mg (glass fibre filters); 74.07 ± 0.66 mg (polyamide membrane); 35.28 ± 0.70 mg (polyester-polycarbonate membrane). The largest deviations were observed in the case of glass fibre filters. The range of filters weight were as follows: glass fibre filters (89.59–92.53 mg); quartz fibre filters (148.81–150.32 mg); PTFE filters (134.37–136.26 mg); polyamide membrane filters (73.16–75.26 mg) and polyester membrane filters (polycarbonate) (34.14–37.87 mg). The most stable measurements in the entire tested range of humidity changes were characteristic of quartz fibre filters, while the largest spread of weight was ascertained for glass fibre filters. The average and SD of filters mass in case of polycarbonate filters was (35.28 ± 0.66 mg), while for PTFE filters (135.07 ± 0.63 mg). Scheme No. 2 was preferable when speaking about filters mass equilibration. By comparing Fig. 3 and 4 we can clearly state that lowering air humidity during conditioning from even 58.78% (Scheme No. 1) to 44% (Scheme No. 2) and equilibrating filters under the same conditions as their weighing helps in a tremendous reduction of the filter's mass deviations.
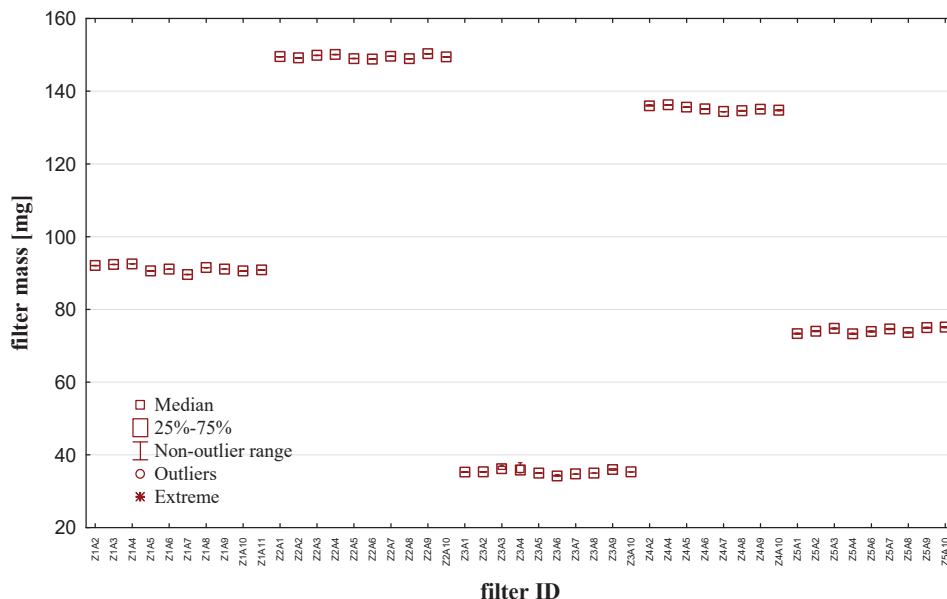
Figure 4. The influence of equilibration conditions on the mean and weight deviations of unloaded filters [mg] (conditions: 19.36–19.81°C; 38–44% RH). *Note: ID – Z1A2 to Z1A11 glass fibre filters; ID – Z2A1 to Z2A10 quartz fibre filters; ID – Z3A1 to Z3A10 polyester membrane (polycarbonate); ID – Z4A1 to Z4A10 PTFE, ID – Z5A1 to Z5A10 polyamide.

Mass variations of unloaded filters during conditioning in the ranges specified in the EN 12341:2014 standard (50±5%, 20±1°C) were presented in Fig. 5. It is easily observed that glass and quartz fibre filters keep their weight stable in the mentioned ranges (Fig. 5a, Fig. 5c), while PTFE and polyamide filters are most susceptible to humidity and temperature variations (Fig. 5b, Fig. 5d). To mathematically describe those relations, a two-factorial linear regression model was adopted to the obtained results. This was further used to predict for example which extent a 5% increase in RH% or 1°C increase in temperature could deviate filter mass (eq. 2–6).
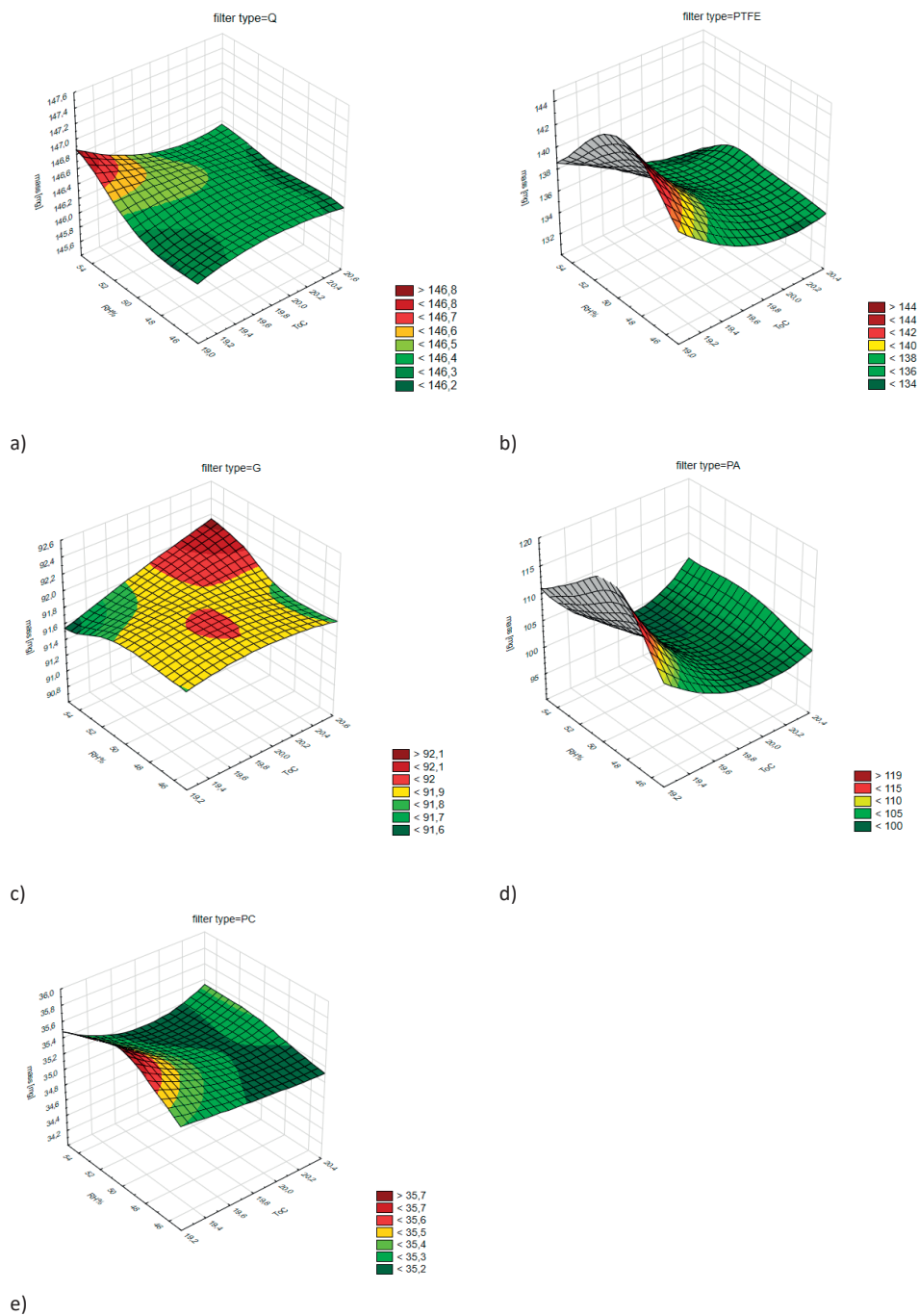
The linear regression equations can be written as follows:

Quartz fibre filters mass [mg] = 146.01 + 0.003 RH + 0.015 Temp.   (eq. 2)

PTFE filters mass [mg] = 157.28 + 0.186 RH − 1.473 Temp.   (eq. 3)

Glass fibre filters mass [mg] = 90.038 + 0.018 RH + 0.055 Temp.   (eq. 4)

Polyamide filters mass [mg] = 51.799 + 2.242 RH − 2.907 Temp.   (eq. 5)

Polycarbonate filters mass [mg] = 35.76 + 0.004 RH − 0.046 Temp.   (eq. 6)

a)

b)

c)

d)

e)

Figure 5. Filter mass variations [mg] about changing equilibration conditions
in the range of 50 ± 5%, 20 ± 1°C

These equations are applicable to filters of the same type with identical Lot numbers (Table S2). Deviations from these factors could significantly affect weighing results. The statistical significance of the results, as indicated by a probability value of $p<0.05$, confirms the suitability of the presented regression models. The regression equations reveal that increasing the relative humidity (RH%) by 1% while conditioning quartz filters results in a minimal mass increase of 0.003 mg. In contrast, for polyamide membranes, this increase is substantial, reaching 2.242 mg. Based on these equations, for quartz fibre filters equilibrated and weighed at a constant temperature of 20°C and a relative humidity of 50%, the average filter weight is 146.460 mg. With an increase in humidity to 55%, the mass of quartz filters (Whatman, QMA, 2.2 μm) reaches 146.475 mg. The estimated weight gain for the quartz filter when transitioning from 50% to 55% RH at constant air temperature of 20°C is 15 μg. For other filter types used in this study, a 5% increase in relative humidity (under 20°C) leads to the following weight changes: PTFE filters increase by 0.93 mg (93 μg), glass fibre filters by 0.09 mg (9 μg), polyamide membrane filters by 11.21 mg (112 μg), and polyester membrane filters (polycarbonate) by 0.02 mg (20 μg). Importantly, the calculated mass additions are within the standard requirement for maximum difference from repeated weighing of a single unsampled filter (≤40 μg) for quartz fibre, glass fibre and polycarbonate filters, but not for PTFE and polyamide filters. When the conditioning temperature decreases while air humidity remains constant, the regression formulas (eq. 2-6) indicate that under a constant temperature of 19°C and relative humidity of 50%, the mass of quartz fibre filters should be approximately 146.445 mg. A temperature decrease of 1°C (from 20°C to 19°C) at constant humidity results in a 15 μg weight change. Interestingly, for quartz fibre filters, this temperature change has an equivalent impact on weight measurements as increasing relative air humidity from 50% to 55%. However, for polyamide and PTFE membranes, the relationship between filter weight and temperature and humidity variations is more complex, leading to greater mass deviations depending on the stabilization conditions.

4. Discussion

Individual sources of uncertainty in PM sampling and concentration measurement are specified in the EN 12341:2014, and their size can be estimated using the provisions and guides on uncertainty in metrology (JCGM, 2008). Those sources have a decisive influence on the accuracy of the PM mass and concentration (Lacey and Faulkner, 2015; Rasmussen, 2010), however, the extent to which those factors can affect weighing performance and measurement accuracy often remains underestimated. This can lead to measurements that fail to fulfil the requirements of applicable regulations or relevant quality standards. Without information on uncertainty of filters mass measurements, the results for i.e. PM concentrations

cannot be compared with each other, or with reference values given in specifications or standards (US EPA, 2008).

Automatic weighing systems offer several advantages over manual methods, including a sealed environmental housing that safeguards the measuring system and filters from potential contamination, ensuring stable ambient conditions during filter conditioning and mass measurement pre- and post-exposure. These systems feature a robotic arm fitted with an infrared sensor for filter detection in the magazine and robot arm holder, complemented by a programmable control system managing the entire weighing process. Additionally, they actively monitor microbalance stability and chamber relative humidity. In our study, we confirmed that the performance metrics for temperature and humidity control and weight stability met the requirements stipulated by the US Environmental Protection Agency (US EPA, 2016) and the EN123401:2014 standard. We also quantified ionizer activity for each measurement session using RMC Filter software. The deionization module in RB 2.4. YF, positioned on the microbalance cover side, effectively neutralized electrostatic charges immediately before filter placement on the pan. Notably, the RB 2.4Y.F. robot consistently maintained stable relative humidity and air temperature conditions during PM filter weighing, remaining within the allowable range as per EN 12341:2014 standard (50±5%; 20±1°C). Our research revealed that variations in equilibration conditions directly impacted filter mass deviations. Therefore, we emphasize the importance of monitoring temperature and humidity parameters throughout the weighing process, facilitated by environmental condition reports generated by the RCMS software for each measurement period. Any deviations beyond standard ranges are typically linked to operational activities such as placing filters in the storage magazine, necessitating documentation in the weighing report.

We presented two experimental equilibration schemes, clearly demonstrating that conditioning filters at 19.36–19.81°C; 38–43% RH yields superior mass stabilization compared to 19.2–20.5°C; 35.67–58.78% RH. The discrepancies in the directional changes of PTFE filters and polyamide membranes' mass, resulting from varying temperature and humidity conditions, can be attributed to insufficient conditioning time. To gain a comprehensive understanding of these dynamics concerning temperature and RH% variability, we investigated the influence of weighing precision relative to changes in filter conditioning time. This influence will be detailed in a separate study (Widziewicz-Rzońca et al., 2022), which can also be found in the report (Weighing procedure, 2021). The research outlined in the cited report led to the formulation of substantial and practical guidelines for conducting gravimetric measurements using the RB 2.4Y.F automatic weighing system, assessing the impact of environmental conditions during weighing based on the filter media type. Within the temperature range of approximately 19-21°C and relative humidity of ca. 35-55% RH, the filters exhibited average and weight deviations as follows: 146 ± 0.32 mg (quartz fibre filters); 135.59 ± 3.19 mg (PTFE filters); 91.91 ± 0.51 mg (glass fibre filters); 101.09 ± 7.13 mg (polyamide membrane); 35.22 ± 0.75 mg (polyester-polycarbonate membrane). These results align well with

prior publications concerning deviations in filter mass due to atmospheric water presence in PM filters (Widziewicz-Rzońca and Tytła, 2020; Widziewicz-Rzońca et al., 2020). In a study by Barba-Lobo et al. (2022), they assumed that changes in relative humidity do not significantly affect the adsorption or loss of PM-bound water, as the mass of PM-bound water is usually much smaller than the mass of loaded filters. Widziewicz-Rzońca and Tytła (2020) found that the addition of water to filter blanks under specific temperature and humidity conditions ranged from 20.8 to 3401.6 µg for different filter types. The mass deviation of the filters, aside from water absorption, was linked to insufficient ionization of the filter surface during equilibration, with the most significant deviation observed in polyamide filters. Stable operation and precise measurements within the PN-EN 12341:2014 standard conditions (45%–55% RH, 19°C–21°C) were emphasized. Rasmussen et al. reported that the magnitude of blank correction increased at higher relative humidity levels, reaching 2.9 µg at 59.6% RH. The study by Kraus and Juhásová Šenitková (2017) indicated that a 10% decrease in relative humidity led to a 10 µg/m^3 increase in PM10 concentration inside buildings, highlighting the greater impact of water addition on PM mass compared to filters. US EPA guidelines (2016) recommended 30–40% RH for filter equilibration, with allowances for variations in ambient conditions. Longer equilibration times were needed for Teflon and polycarbonate filters (Widziewicz-Rzońca et al., 2022), making them more time-consuming. In summary, US EPA humidity regulations appeared more suitable for filter equilibration than the PN EN 12341:2014 standard. Humidity and temperature can affect filter and weighing device performance. Rapid humidity changes can disrupt equilibrium by settling additional water molecules on the mass comparator's mechanical design, gradually affecting indication repeatability. Quantifying this change is challenging, necessitating further research on water absorption, weighing repeatability, and automated versus manual weighing.

5. Conclusions

The authors succeeded in adapting manual weighing procedures for filter weight measurements using a weighing robot. The mass of the reference filter remained stable, with variations limited to the balance indication repeatability error (±2.2 µg) under standard conditions (temperature: 19.64–20.6°C, humidity: 38-44%). Air temperature and humidity remained stable within the weighing robot chamber during filter measurements. Under varying conditions (humidity: 35.67–58.78% RH, temperature: 19.2–20.5°C), average filter weights were as follows: Quartz fibre filters: 146 ± 0.32 mg; PTFE filters: 135.59 ± 3.19 mg; Glass fibre filters: 91.91 ± 0.51 mg; Polyamide membrane: 101.09 ± 7.13 mg; Polyester-polycarbonate membrane: 35.22 ± 0.75 mg. Notably, PTFE and polyamide membranes exhibited reduced mass deviation when humidity decreased from 45.6% to 40.63%. Introducing 48-hour filter stabilization periods before mass measurement reduced

standard deviation by approximately 0.15 mg (quartz filters) and 0.35 mg (glass filters), suggesting that longer equilibration may enhance mass stabilization compared to EN 12341:2014 standards.

However, PTFE, polyamide, and polycarbonate filters were unable to meet the EN 12341:2014 standard (40 µg mass difference), even with the extended equilibration period. This may be attributed to filter material ionization variations, slow neutralization, electric field effects by charged filters, and chamber opening-induced differences in air buoyancy and electric fields. Pre-weighing filter charge is recommended for quality control. In conclusion, weighing robots are suitable for routine PM filter mass measurements. Future research should explore different filter materials and compare weighing system accuracy and stability with various robots under diverse conditions.

Acknowledgments

Conflicts of Interest

The authors declare no conflict of interest

References

1. Barba-Lobo, A. et al, (2022). Methodology for Determining Particulate Matter Mass in Atmospheric Filters. *Environ. Res.*, 214, Part 1, 113817, https://doi.org/10.1016/j.envres.2022.113817
2. Lacey, R.E., and Faulkner, W.B., (2015). Uncertainty in Gravimetric PM Concentration Measurement. *J. Air & Waste Manag. Assoc.*, 65(7): 887-894.
3. NIST (2019). Good Measurement Practice for Weighing Operations: https://doi.org/10.6028/NIST.IR.6969-2019.
4. US EPA (2008). Particulate Matter Gravimetric Analysis Procedure. RTI Int. Res. Triangle Park, NC.
5. Widziewicz-Rzonca, K. and M. Tytla (2020). Water Sorption by Filter Media for PM Collection. Int. J. Environ. Res. Public Health 17(14).
6. Montgomery J.F., et al., 2015. Impact of Relative Humidity on HVAC Filters. Aerosol Sci. Technol. 49, pp. 322-331.
7. Bureau Veritas UK (2009). Trends in Gravimetric Particulate Mass Measurements. R. Maggs et al.
8. EN 12341:2014 - Gravimetric Measurement Method for PM10 and PM2.5.
9. CFR Appendix L to Part 50 - Reference Method for PM2.5 Determination.

10. US EPA (2016). Quality Assurance for PM2.5 Monitoring. EPA-454/B-16-001.

11. Charell, P.R., Hawley, R.E. (1981). Water Adsorption on Air Sampling Filters. Am. Ind. Hyg. Assoc. J. 42, 353–360.

12. Raynor PC, Leith D, Lee K. W., Mukund (2011). Sampling and Analysis Using Filters. Aerosol Measurement: Principles, Techniques, and Applications.

13. Brown, A. S., et al. (2006). Effect of Humidity on Filter Materials for PM Measurements. Atmos. Environ. 40(25): 4670-4678.

14. Harris G.L. (2019). Laboratory Practices for Mass Calibrations (2019 Edition).

15. Su, W., et al. (2008). Effect of Humidity on Filter and Gravimetric Measurement of PM. Chin. J. Ind. Hyg. Occup. Dis. 26, 198–202.

16. Frohlich T., Fehling T, Heydenbluth D (2009). Mass Dissemination using a Robot System. Int. Conf. on Precision Measurement.

17. Ren, XP et al., (2013). Mass Metrology Based on Robotic Mass Measurement System.

18. Allen, R., et al. (2001). Cost-effective Weighing Chamber for Particulate Matter Filters. J. Air & Waste Manag. Assoc. 51(12): 1650-1653.

19. Carlton, A. M. and A. Teitz (2002). Design of a Weighing Facility for PM 2.5 Quality Assurance. J. Air & Waste Manag. Assoc. 52: 506-510.

20. Ogden, M.W., et al. (2015). Robotic System for Filter Weighing.

21. Presler-Jur, A. P., et al. (2016). Robotic Weighing System Performance. Aerosol Air Qual. Res. 16(10): 2438-2451.

22. PFS-ONE CHAM. https://static.horiba.com/fileadmin/Horiba/Products/Automotive/Emission_Measurement_Systems/PFS-ONE/PFS-ONE_Brochure_ENG.pdf

23. Mettler Toledo (2021). Automated Filter Weighing. https://www.mt.com/tw/zt/home/products/Laboratory_Weighing_Solutions/mettlerproduct-collaboration/Automated-Filter-Weighing.html

24. Comde-Derenda. https://www.comde-derenda.com/en/products/automatic-weighing-system-aws-1re/

25. RB 2.4Y.F Filter Weighing Robot, https://radwag.com/.

26. L'Orange Ch., Neymark G., Carter E., Volckens J (2021). High-throughput, Robotic System for Aerosol Sampling Filters. Aerosol Air Qual. Res. 210037.

27. Rasmussen,P.E; Gardner,H.D; and Niu,J.J; (2010). Buoyancy-Corrected Gravimetric Analysis of Filters. J. Air & Waste Manag. Assoc. 60 (9), pp.1065-1077.

28. World Health Organization. (2021). Global Air Quality Guidelines. https://apps.who.int/iris/handle/10665/345329.

29. JCGM 100:2008 - Guide to Uncertainty in Measurement.

30. Widziewicz-Rzońca K., et al. (2020). Water in Ambient Particulate Matter. Sustainability 12(15): 6196.

31. Weighing Procedure (2021). Mass Determination of Filters With RB 2.4Y.F Robot. https://radwag.com/pl/.

32. Kraus Michal and Juhásová Šenitková Ingrid 2017. PM Mass Concentration in Buildings. IOP Conf. Ser.: Mater. Sci. Eng. 245 042068.

Supplement

Figure S1. RB 2.4Y.F automatic robotic system, manufactured by Radwag Electronic Scales, Poland (https://radwag.com/en/).

Table S1. Summary presenting PM filter mass deviations due to variable conditioning parameters during equilibration

Aim	Filter type	PM fraction	Measurement type	Mass deviation	Conditioning parameters	Reference
Testing changing temperature on buoyancy-corrected mass measurements	47-mm-diameter blank PTFE (Teflon) filters	dust control powder	Archimedes M3 buoyancy-corrected gravimetric analysis facility	0.9 - 0.6 RSD 0.7 - 0.7 RSD 0.4 - 0.6 RSD 0.2 - 0.5 RSD 1.3 - 0.4 RSD 2.9 - 1.5 RSD	15.9 /21.5 °C, 20.6 /21.5 °C 30.6 /21.5 °C 40.5 /21.5 °C 49.9 /21.5 °C 59.6 /21.5 °C	[Rasmussen et al., 2010]
To design and construct a PM2.5 weighing facility	47-mm stretched PTFE Teflon (lab blank) filters	-	PM2.5 weighing room and chamber in Edison, NJ., chamber (Electro-Tech Systems, Inc. (ets) model 518)	MDL for mass determination MDL= 6.3 µg	34.6%-36.8% and 21.8–23.3 ºC	[Carlton and Teitz, 2002]
Creating a novel, simple methodology to determine the corrections of the filter mass due to humidity changes	Quartz fiber filters (QF20 Schleicher and Schuell, 25.4 cm × 20.3 cm)	PM10	Nd.	Uncertainty of the particulate matter mass deposited on the filters (σ_m) Method 1. σ_m = 1.4 mg Method 2. σ_m =1 mg	Relative humidity for: Method 1. RH~18-62% Method 2. RH ~50%	[Barba-Lobo et al, 2022]

Aim	Filter type	PM fraction	Measurement type	Mass deviation	Conditioning parameters	Reference
Testing the effects of the alteration of humidity and (or) temperature on weight of filters without and with ambient particulate matter in a balance room			Nd.	0.07-0.1 mg and 0.04-0.08 mg under the temperatures of (18+/-1) ºC and (28 +/-1) ºC, respectively 0.09-0.14 mg under the temperatures of (18+/-1) ºC and (28+/-1) ºC, respectively and under RH% varying from 35 – 100 %RH	(18 +/- 1)ºC and (28 +/- 1) ºC; humidity 35 – 100 %RH	[Su et al., 2008]

*Nd: No data

Table S2. Characteristics of Whatman® filters used in weight measurements

No	Filter type	Product no.	Pore size μm	Purity	Thickness μm	Water retention	Filter effectiveness [%]
1.	Quartz fibre	1851-047	2.2	QMA	450	Nd.	98
2.	Glass fibre	1820-047	1.6	GF/A	220	Nd.	98
3.	Polyamide	10414012	0.2	Nd.	110	Nd.	Nd.
4.	PTFE**	7592-104	2	Nd.	30-50	Mass addition <10 μg [1]	99.7
5.	Polycarbonate	7060-4702	0.2	Cyclo-sporine PC	7-20	Nd.	Nd.

*Nd: No data.
**PTFE - (polytetrafluoroethylene) with support ring for PM2.5.
[1] after 24h of exposure under 40% RH in relation to the mass addition after 24h of exposure under 35%.

Table S3. The influence of equilibration conditions on the deviation of the mass of the standard mass and reference filter.

Variable	N	Mean	Min.	Max.	Std. Dev.
Average reference mass [mg]	29	243.95	243.95	243.96	0.0022
RH [%]	29	47.37	41	49	1.45
Temp. [°C]	29	19.95	19.77	20.14	0.07

ANALIZA STANU WIEDZY NA TEMAT STABILNOŚCI MASY FILTRÓW DO POBIERANIA CZĄSTEK PYŁU ZAWIESZONEGO: WPŁYW WILGOTNOŚCI I TEMPERATURY

Abstrakt

Skomplikowana zależność pomiędzy wilgotnością i temperaturą a masą filtrów do pobierania pyłu zawieszonego pozostaje przedmiotem zainteresowania społeczności naukowej. Celem badania była ocena zmian masy nieobciążonych filtrów do pobierania pyłu zawieszonego w różnych warunkach wilgotności i temperatury, wykorzystując w pełni zautomatyzowaną metodę ważenia i dwuczynnikowy model regresji. Istniejące wytyczne, jak postulują EPA i norma europejska (EN), wykazują w tej kwestii wyraźną rozbieżność. Amerykańska Agencja Ochrony Środowiska EPA zaleca węższy zakres wilgotności wynoszący 30–40% RH, podczas gdy EN sugeruje szerszy zakres 40–50% RH. Wyniki tego badania potwierdzają wytyczne amerykańskiej Agencji Ochrony Środowiska (EPA), które wykazały doskonałą stabilność masy filtra przy zmiennej wilgotności. W kontrolowanych, stabilnych warunkach temperaturowych wynoszących 20°C analizy wykazały znaczne różnice w masie nieobciążonych filtrów wraz ze wzrostem wilgotności względnej z 50% do 55%. Wykazano, że w stabilnych warunkach temperaturowych (20oC) wewnątrz robota ważącego wzrost wilgotności względnej powietrza z 50% do 55% powoduje wzrost masy filtrów o 15 μg (Q, filtry z włókien kwarcowych); o 93 μg (filtry PTFE); o 9 μg (G, filtry z włókna szklanego); o 112 μg (PA, filtry poliamidowe) i o 20 μg (PC, filtry poliwęglanowe). Analiza zakresów kondycjonowania określonych w normie EN 12341:2014 wykazała, że filtry z włókna szklanego są szczególnie stabilne, podczas gdy filtry z membraną poliamidową stwarzają intrygujące wyzwania, potencjalnie związane z niedostateczną eliminacją ładunków elektrostatycznych na etapie kondycjonowania. Odkrycia te mają znaczenie dla protokołów aplikacyjnych stosowanych przy produkcji filtrów i protokołów kondycjonowania wdrożonych w laboratoriach grawimetrycznych, potencjalnie zwiększając precyzję monitorowania jakości powietrza.

Słowa kluczowe: filtry do pobierania pyłu zawieszonego, odchylenia masy, pomiar masy, ważenie, ważenie automatyczne