



Interlaboratory study of the operational stability of automated sorption balances

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Abstract

Automated sorption balances are widely used for characterizing the interaction of water vapor with hygroscopic materials. These instruments provide an efficient way to collect sorption isotherm data and kinetic data. A typical method for defining equilibrium after a step change in relative humidity (RH) is using a particular threshold value for the rate of change in mass with time. Recent studies indicate that commonly used threshold values yield substantial errors and that further measurements are needed at extended hold times as a basis to assess the accuracy of abbreviated equilibration criteria. However, the mass measurement accuracy at extended times depends on the operational stability of the instrument. Published data on the stability of automated sorption balances are rare. An interlaboratory study was undertaken to investigate equilibration criteria for automated sorption balances. This paper focuses on the mass, temperature, and RH stability and includes data from 25 laboratories throughout the world. An initial target for instrument mass stability was met on the first attempt in many cases, but several instruments were found to have unexpectedly large instabilities. The sources of these instabilities were investigated and greatly reduced. This paper highlights the importance of verifying operational mass stability of automated sorption balances, gives a method to perform stability checks, and provides guidance on identifying and correcting common sources of mass instability.

Keywords Water vapor sorption · Interlaboratory investigation · Measurement uncertainty

1 Introduction

The study of water vapor sorption in materials has been revolutionized by automated instruments. Automated gravimetric instruments fall into two categories: vacuum sorption balances, in which water vapor is the only component in the gas phase, and continuous-flow sorption balances, in which relative humidity (RH) is controlled by mixing dry and water-saturated streams of carrier gas (e.g., nitrogen or dry air) using mass flow controllers. The latter technique is often called Dynamic Vapor Sorption (DVS). In both instrument types, the mass of a small material sample (milligram

range) is continuously measured by an electronic microbalance located within a temperature-controlled chamber [3, 5, 6, 15, 22, 25].

Gravimetric sorption balances have a century-old history in sorption science [13], going back to the early apparatus by McBain et al. [16]. Since then a variety of apparatuses have been contrived to measure sorption of a single sample [21] or multiple samples simultaneously [18], even achieving a mass resolution of $< 1 \mu\text{g}$ in some systems [4, 11]. The RH of the conditioning environment has been maintained through controlling the temperature of a liquid reservoir [16] or by saturated salt solutions [14] in vacuum systems. Alternatively, continuous flow of pre-conditioned gas using saturated salt solutions has for instance been employed in the

Extended author information available on the last page of the article

Fig. 1 Geographical locations of the laboratories who participated in the study



non-vacuum sorption balance by Seborg et al. [23]. Automation has greatly reduced the labor required to perform sorption experiments. Therefore, with the adoption of automated sorption balances in laboratories across the world, the last two decades have seen an explosion in published studies on water vapor sorption. They have been used to characterize a variety of materials such as food ingredients [7, 20, 24], pharmaceutical components [2], nanoporous carbon [19], textile and composite fibers [1, 12, 26], and construction materials [8, 17].

Despite the benefits offered by automated sorption balances in terms of mass resolution and automated control of the conditioning environment, they can only provide quality sorption data through carefully programmed experiments. The appropriate experimental protocol depends on sample geometry and sorption properties of the material investigated as well as the required level of data accuracy. A high accuracy in equilibrium moisture content requires sufficiently long conditioning times in an accurately controlled conditioning environment.

To reduce measurement time, a typical method for defining equilibrium after a step change in relative humidity is using a particular threshold value for the rate of change in mass over time. However, commonly used threshold values can yield errors that are much larger than usually assumed for automated sorption balances, as documented for water

vapor sorption in wood and other cellulosic materials [9, 10]. While more data are needed at extended measurement times as a basis to assess the accuracy of abbreviated equilibration criteria or systematic corrections to such criteria, the mass measurement accuracy at extended times depends on the operational stability of the instrument. The sensitivity of electronic microbalances makes them vulnerable to drift and external disturbances such as vibrations and accidental bumps to the instrument during the extended measurement time, but published data on the operational stability of automated sorption balances are scarce.

Therefore, an interlaboratory study was initiated to investigate equilibration criteria for automated sorption balances. The first step to developing equilibrium criteria is to understand the inherent stability of instruments used in practice.. This comprehensive approach not only emphasizes the technical aspects but also brings to light the regulatory and compliance considerations, especially given the diverse applications of these instruments across industries.

This paper focuses on the mass, temperature, and RH stability of automated sorption balances and includes data from 25 laboratories across the world (Fig. 1). We highlight the importance of verifying operational mass stability, present a method to perform stability checks, and offer guidance on identifying and correcting common sources of mass instability.

Table 1 Instruments used in the study

Manufacturer	Model(s)
CI Electronics Ltd	Vacuum microbalance
Hidden Isochema Ltd	IGAsorp
Surface Measurement Systems Ltd	DVS Advantage, DVS Advantage Plus, DVS Intrinsic
TA Instruments – Waters LLC	Discovery SA, Q5000SA, VTI-SA +

2 Materials and methods

2.1 Materials

To check the stability of the instruments, a non-hygroscopic sample material was selected to eliminate any potential interference from a sorption response that would complicate the interpretation of results due to RH instability. The non-hygroscopic samples consisted of aluminum Differential Scanning Calorimetry (DSC) sample pans (Tzero sample pan, TA Instruments, New Castle, DE, USA), which were low cost, easily obtainable, and had a relatively uniform mass (ca. 41 mg) close to the mass of the wood samples frequently used in automated sorption balance studies [9, 10].

2.2 Experimental methods

All automated sorption balances in the study had a mass resolution of $\leq 1 \mu\text{g}$; most laboratories had a resolution of $\leq 0.1 \mu\text{g}$. Both continuous-flow instruments and vacuum instruments were included. A list of manufacturers and models of the instruments used is provided in Table 1.

All laboratories in this study measured the operational mass stability of their instrument using the aluminum pan provided to them. The pan was loaded into the sample holder with the temperature set to 25 °C. For continuous-flow instruments, the carrier gas flow rate was between 200 $\text{cm}^3 \text{min}^{-1}$ and 250 $\text{cm}^3 \text{min}^{-1}$. The data collection interval was less than or equal to 60 s. The mass, temperature, and RH were recorded with a constant target of 50% RH for a duration of 50 h. Although calibration accuracy for temperature, relative humidity, and mass is essential, this was outside the scope of this study. The focus here was on operational stability.

The initial mass stability target was a maximum 24-h slope less than or equal to 4 $\mu\text{g day}^{-1}$ (see below for further description). The 4 $\mu\text{g day}^{-1}$ criterion was twice the largest mass instability noted by Glass et al. [9]. If the first attempt exceeded this threshold, the source of the instability was investigated and corrected if possible, and the experiment was repeated.

2.3 Data analysis

Data were analyzed for the temperature stability, relative humidity stability, and mass stability.

2.3.1 Temperature stability

The temperature stability was analyzed by calculating the difference between the 99th percentile measured temperature value and the 1st percentile value over the 50 h experiment. This difference is denoted ΔT_{1-99} . For instruments that reported the temperature at multiple locations within the instrument, only the temperature nearest to the sample was analyzed.

2.3.2 Relative humidity stability

The relative humidity stability was similarly determined by calculating the difference between the 99th percentile measured RH value and the 1st percentile value. This difference is denoted $\Delta \phi_{1-99}$ with values given as percentages.

2.3.3 Mass stability

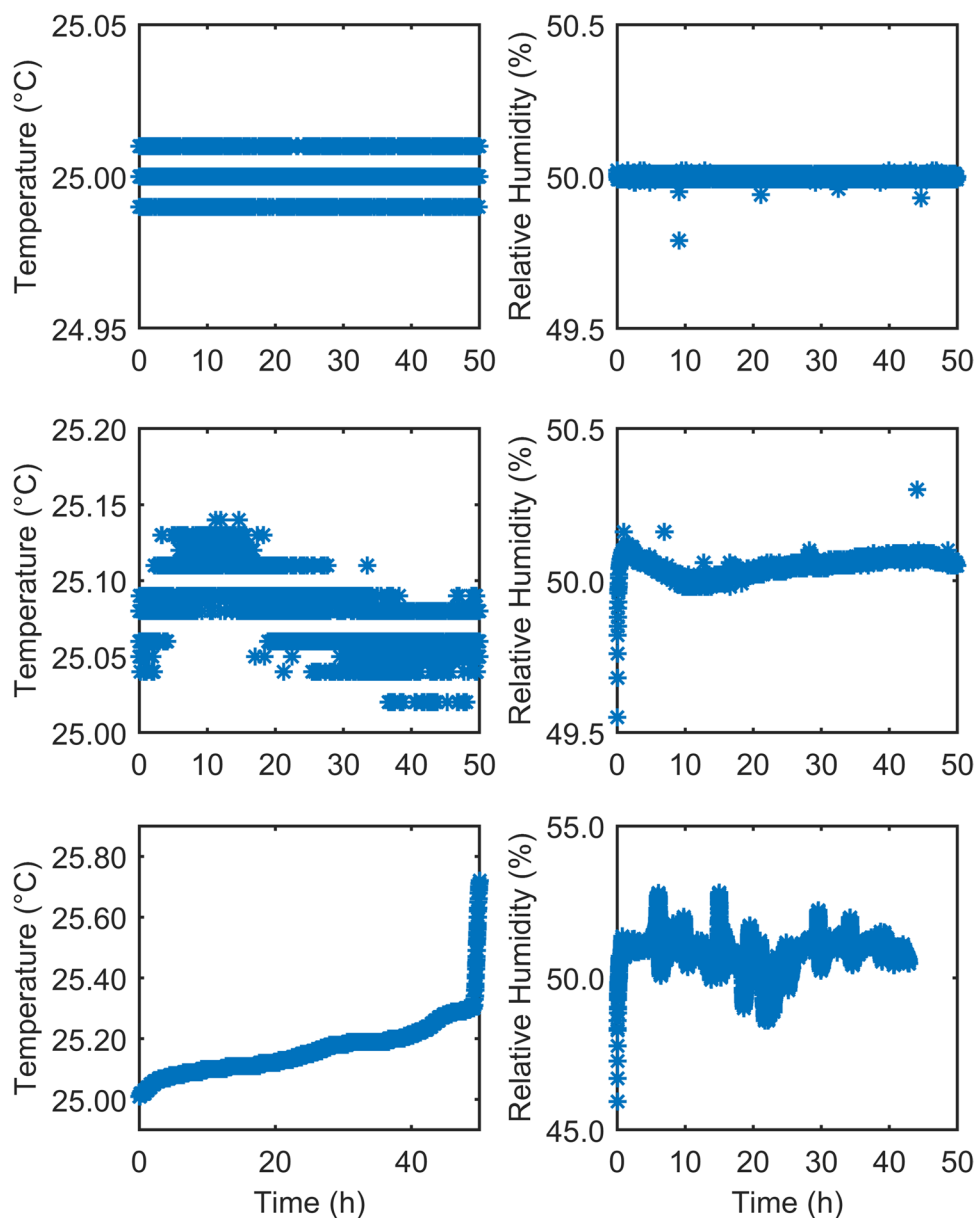
The mass stability was calculated in several different ways. First, like temperature and relative humidity, the difference between the 99th percentile measured mass value and the 1st percentile value was calculated and denoted Δm_{1-99} . In addition, the slope of the mass vs. time data was analyzed and reported using time windows of the previous 1 h and 24 h. The latter was used previously by Glass et al. [9] to characterize the mass stability of an automated sorption balance and also an operational definition for sample equilibrium.

Since every laboratory had different data acquisition intervals, the raw data was first treated with the “interp1” function in MATLAB (Natick MA, USA). This applied a linear interpolation to convert the raw data into (time, mass) ordered pairs with the time in even 1-min intervals. These ordered pairs were then input into an Excel spreadsheet.

The “SLOPE” function was applied to 1440 rows of data which resulted in a linear regression over the previous 24 h of data (1440 min), which we hereafter refer to as the 24-h running slope. The 24-h running slope was calculated at 1-min intervals from 24 to 50 h. The maximum value of the absolute value of this 24-h running slope (from 24 to 50 h) was identified and denoted $|S_{24\text{-h}}|_{\text{max}}$.

The “SLOPE” function was also applied to 60 rows of data which resulted in a linear regression over 1 h (60 min) of data, which we hereafter refer to as the 1-h running slope. The 1-h running slope was calculated at 1-min intervals from 1 to 50 h. The maximum value of the absolute value of this 1-h slope (from 1 to 50 h) was identified and denoted $|S_{1\text{-h}}|_{\text{max}}$.

Fig. 2 Left column) Example temperature vs. time data from the laboratory with the most stable (top row), median (middle row), and least stable dataset (bottom row). (right column) Example relative humidity vs. time data from the laboratory with the most stable (top row), median (middle row), and least stable dataset (bottom row). (Note the different vertical axis scales in each row)



3 Results and discussion

3.1 Temperature stability

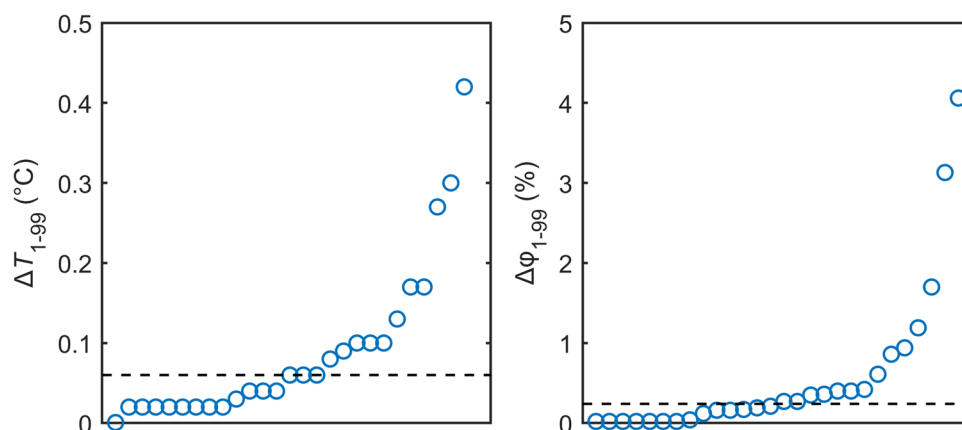
Figure 2 is a plot showing example time series from different laboratories, with measured temperature values shown in the left column. The top row is the dataset with the most stable temperature control (smallest non-zero ΔT_{1-99}); the middle row is the median; and the bottom row is the least stable dataset (note the different vertical axis scales in each row). While the primary goal of these measurements was to examine the stability of the microbalance, temperature stability is important for hygroscopic materials as the temperature may affect both the relative humidity in the chamber

and the amount of absorbed moisture, or may also directly affect the balance stability.

A comparison across all laboratories can be seen in the left panel of Fig. 3, which plots the difference between the 99th percentile and 1st percentile measured temperatures. The graph includes multiple measurements from some laboratories that repeated the mass stability step. The graph also excludes several laboratories that reported no temperature fluctuations. The reported temperatures from these laboratories were displayed as “25” with no decimal places following the integers making it difficult to discern the true range of the temperature measurement.

In general, the temperature was quite stable across laboratories. The median value of ΔT_{1-99} was 0.06 °C. Only

Fig. 3 Left) Difference between 99th and 1st percentile measured temperatures across datasets. (right) Difference between 99th and 1st percentile measured relative humidity (ϕ) values across datasets. The dashed line represents the median value in each subfigure



6 measurements (out of 27) had a value of ΔT_{1-99} that exceeded 0.1 °C.

3.2 Relative humidity stability

Example time series of relative humidity from different laboratories are shown in the right column of Fig. 2 (note the different vertical axis scales in each row). While the relative humidity was not expected to affect the mass of the inert sample used in these experiments, it is critical for hygroscopic materials, and the results give insight into the performance of automated sorption balances used worldwide.

The right panel of Fig. 3 plots the difference between the 99th percentile and 1st percentile values of measured relative humidity during the experiment for different laboratories. Similar to the temperature curve, some laboratories reported a relative humidity of “50” with no decimal places. These measurements were excluded from the figure as it was impossible to ascertain the number of significant digits in the humidity measurement. For laboratories that supplied multiple datasets, the relative humidity stability from all measurements is included in Fig. 3.

As expected, most laboratories had very tight control of the relative humidity near the sample. 21 of the 28 measurements in the study (75%) had a $\Delta\phi_{1-99}$ value less than 0.5%. However, four of the remaining seven laboratories had a $\Delta\phi_{1-99}$ value greater than 1%. The largest value was 4.06% RH. These data do not exhibit a long-term drift upward or downward in RH but instead show large fluctuations around a median RH of 50.9% RH.

3.3 Mass stability

The key data from each laboratory are the mass vs. time curves for the 50 h experiment. An example mass vs. time curve for the laboratory with the most stable balance is shown in Fig. 4a. Figure 4b shows the 24-h running slope. The top panel also graphically illustrates how the 24-h slope

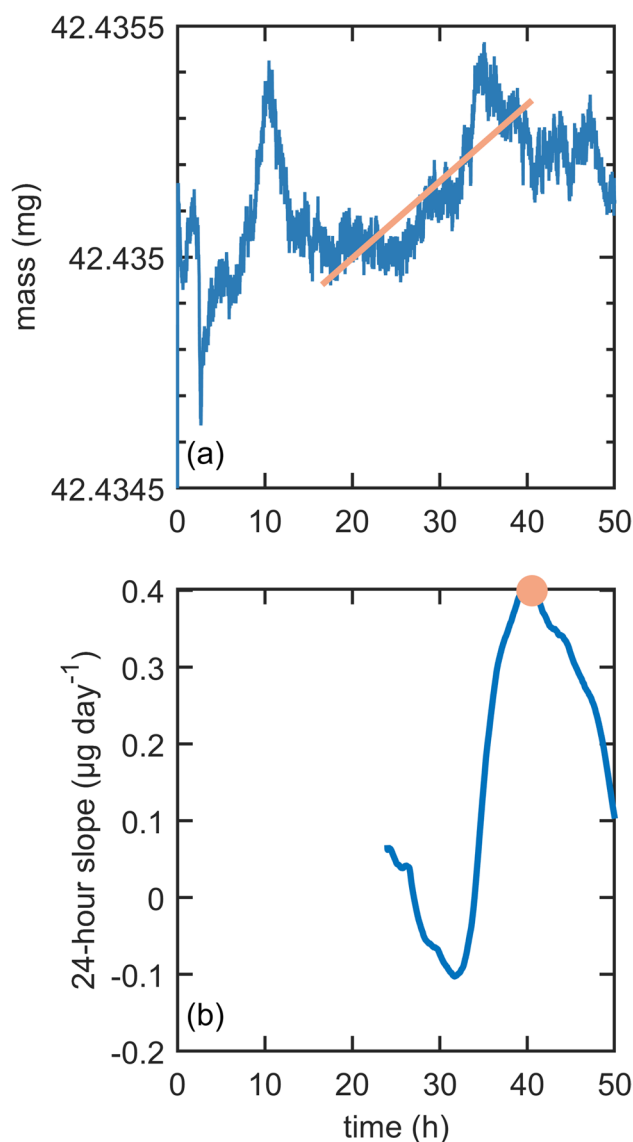
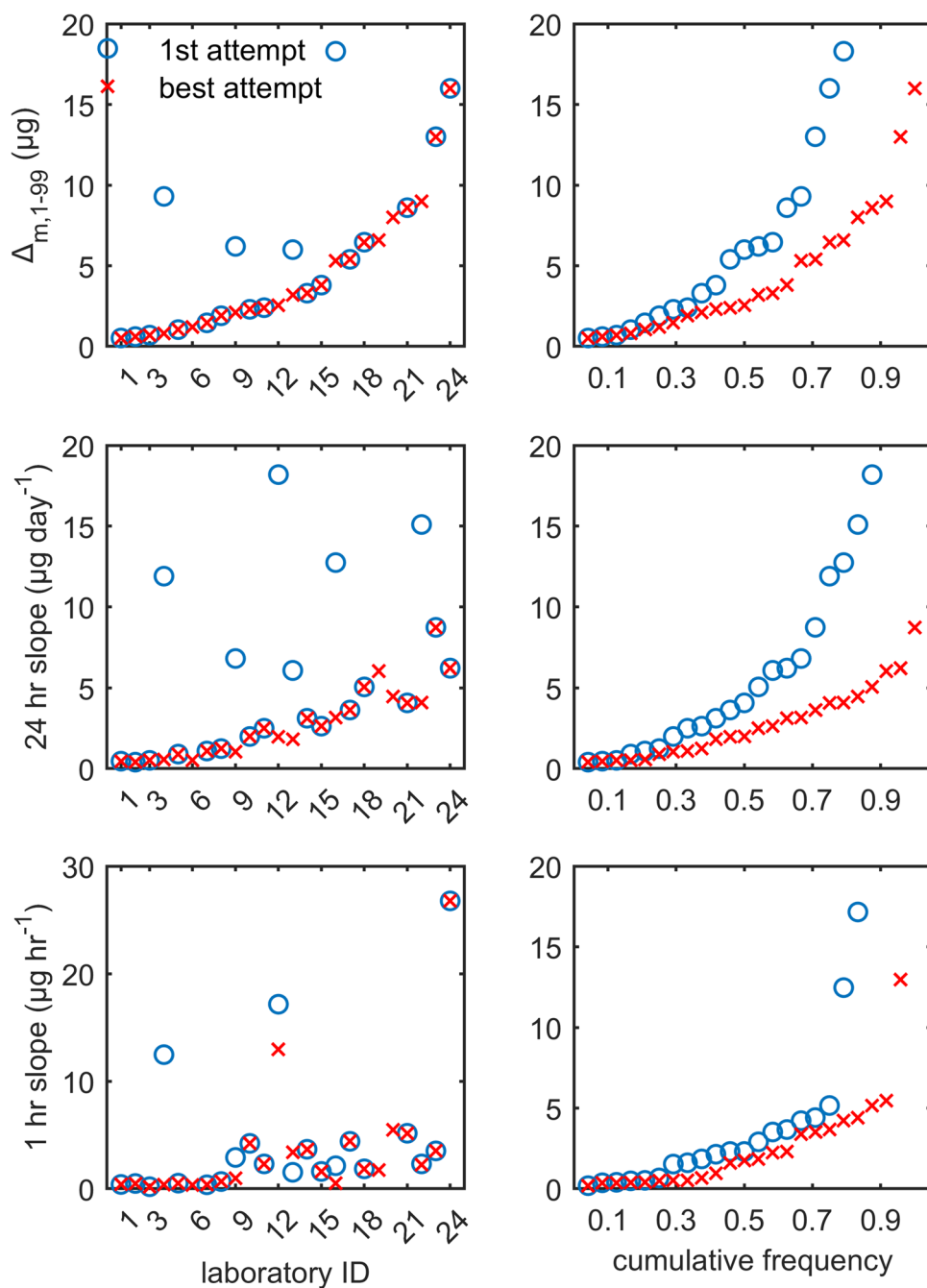


Fig. 4 A) Example mass vs. time data for the most stable balance. The orange region of the graph corresponds to the 24 h used to calculate the slope (orange trendline) at the orange dot in the bottom panel. (b) 24-h running slope calculated from the data above

Fig. 5 Left column) Mass stability across datasets as characterized by difference between 99th percentile and 1st percentile (top), 24-h slope (middle), and 1-h slope (bottom) for the 25 participating laboratories. Note that the laboratory ID is not related to the author affiliation number. (right column) Same data from the left column presented as cumulative frequency. The blue circles represent the first attempt by each laboratory. A difference between the blue circle and the red 'x' in the left column indicates the experiment was repeated and stability was improved



was calculated for the point with a orange dot in the bottom subfigure. The preceding 24 h of data are plotted in orange along with the trendline taken from that region of $0.4 \mu\text{g day}^{-1}$.

Figure 5 summarizes the results of the mass stability checks performed at each laboratory for the three different measures of mass stability: difference between the 99th and 1st percentiles (top), 24-h slope (middle), and 1-h slope (bottom). These data are replotted in terms of the cumulative frequency in the right column. The cumulative frequency shows the number of laboratories with a mass stability value

less than or equal to that number; a point at (0.7, 5) in the top right panel would mean that 70% of laboratories have a Δm_{1-99} less than or equal to $5 \mu\text{g}$. These data are useful for understanding the total number of laboratories with errors less than or equal to a certain mass difference. The data are also tabulated in the Appendix Table 2. Given that the mass stability checks were conducted on a non-hygroscopic material, there should be no actual mass gain or loss throughout the experiment. In other words, the data in Fig. 5 represent a measure of the inherent short-term and long-term stability of the instruments. Examples of the data with the highest

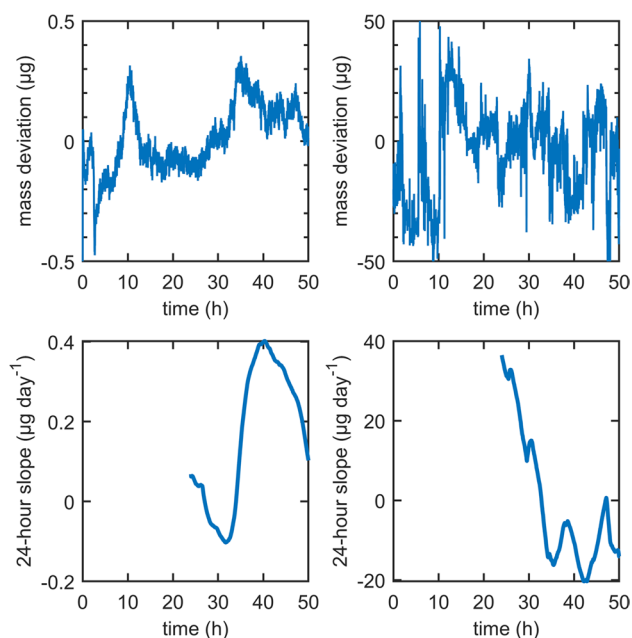


Fig. 6 Comparison of the most stable (left) and least stable (right) mass measurements with the 24-h slopes shown in the subfigures below. The mass deviation from the median mass is plotted rather than the absolute sample mass since the mass of the DSC sample pans was slightly different. Note the difference in scales on the y axes

and lowest mass stability are given in Fig. 6 along with their corresponding 24-h slopes.

Many laboratories provided data on the first attempt that did not meet the initial stability target ($|S_{24-h}|_{\max} \leq 4 \mu\text{g day}^{-1}$). In these cases, the source of the instability was investigated and corrected if possible, and the experiment was repeated. For example, one laboratory was able to reduce their mass instability (as characterized by $|S_{24-h}|_{\max}$) from more than $35 \mu\text{g day}^{-1}$ to $0.52 \mu\text{g day}^{-1}$.¹ It can be easily seen from the cumulative frequency figure that while the median value only slightly improves between the first and best runs, the maximum values are greatly lowered and the width of the distribution is reduced. Figure 5 does not include data from a laboratory with an initial value of $845 \mu\text{g day}^{-1}$. Although this was subsequently reduced to $21 \mu\text{g day}^{-1}$, the laboratory decided not to invest more time in further improvements during this study. These datasets were therefore not included in the statistics reported in the remainder of the paper or the Appendix Table 2.

Examining only the best dataset supplied by each laboratory, the value of $|S_{24-h}|_{\max}$ ranged from $0.40 \mu\text{g day}^{-1}$ to $8.73 \mu\text{g day}^{-1}$. The median value was $2.25 \mu\text{g day}^{-1}$, and the mean value was higher at $2.76 \mu\text{g day}^{-1}$ on account of several

extreme values visible in Fig. 5. The median value of $|S_{1-h}|_{\max}$ was $1.80 \mu\text{g h}^{-1}$ and the mean value was $3.51 \mu\text{g h}^{-1}$. The median value of Δm_{1-99} was $2.90 \mu\text{g}$ and the mean value was $4.43 \mu\text{g}$. Comparing across these metrics, we note that both the 1-h and 24-h slopes had similar magnitude mass changes over different time periods, and both were about 50% smaller than the Δm_{1-99} over the entire experiment. This suggests that a large portion of the instability over a 1-day period happens within the hour with the biggest mass fluctuation.

While several laboratories showed a high initial mass instability (reflected in $|S_{24-h}|_{\max}$) that was later corrected, there was not one universal fix to reduce the instability. These corrections also improved the short-term mass stability (i.e., the 1-h slope). Several different sources of instability were encountered; three of these are further discussed below: instrument warm-up, vibration, and contamination.

3.3.1 Instrument warm-up

Upon turning on the instrument after a period of being shut down, the mass instability was seen to be high. This was observed across three different laboratories, whose first experiments are shown Fig. 7. It can be seen in the figure that absolute value of the 24 h slope decreases (stability improves) during the 50 h hold time in these datasets. When these laboratories repeated the stability test after leaving the instrument on for several days, the mass stability greatly improved. This long time needed before stabilization should be noted.

3.3.2 Vibration

The operational mass stability of sorption balances can be degraded by vibrations or other disturbances to the balance. In general, there are two types of vibrations that caused issues in mass stability. An example of each of these types of vibration are shown in Fig. 8 from two different laboratories. In some cases, there was a one-time spike in the mass, possibly from someone touching the instrument itself or the table the instrument was placed on, or an unusual event in the laboratory (someone dropping something, a door slamming, etc.). An example is shown in the upper left of Fig. 8. For these one-time mass spikes, a repeated measurement showed much better mass stability (data not shown).

In other cases, the sorption balance was not properly isolated from the environment and the mass readings indicated drift as well as abrupt instabilities (see upper right of Fig. 8). In this case the stability was greatly improved after the instrument was moved onto a vibration isolation table (data not shown).

3.3.3 Contamination

An interesting observation was that the mass decreased in one instrument when the RH was increased from near

¹ This laboratory was able to improve their instrument stability by replacing the counterweight.

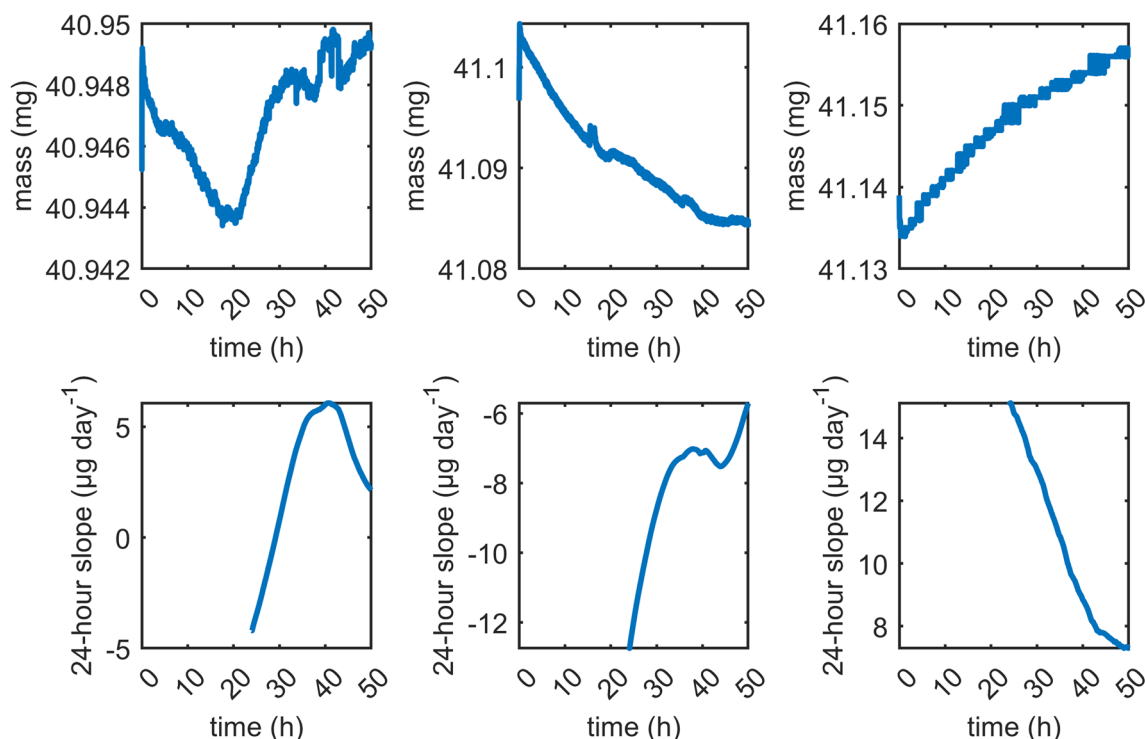


Fig. 7 Top) Example mass vs. time data from laboratories that were able to improve their mass stability by letting their instruments warm up before re-running the mass stability experiment. (bottom) Corre-

sponding 24-h slopes showing a clear trend towards smaller absolute values over the 50 h experiment

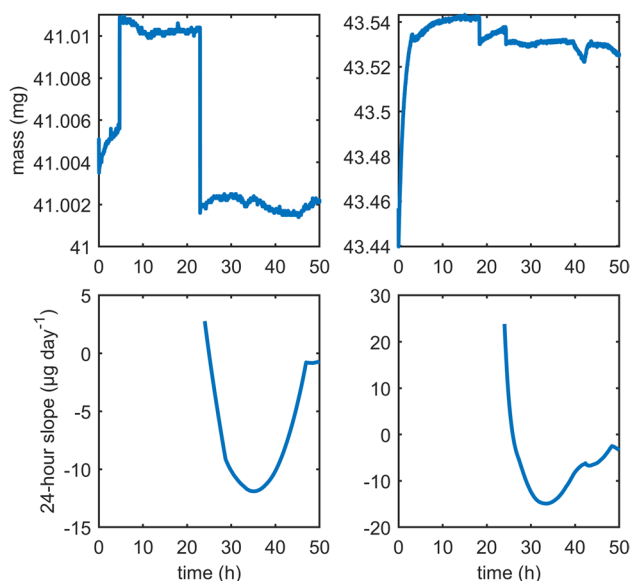


Fig. 8 Top) Example mass vs. time data from laboratories that had instability from vibrations. In the top left case, the table upon which the instrument sat was unintentionally bumped. In the top right case, the vibrations were caused by improper vibration isolation of the instrument. This laboratory was only able to improve their stability after moving the instrument to a new location. (bottom) Corresponding 24-h slopes

0% to 50% RH (Fig. 9). The material appears to have an unphysical “negative sorption” behavior where the (inert) sample dries out as the humidity is increased. These results can be explained by contamination on the reference pan, which is exposed to the same RH conditions as the sample. Since the microbalance measures the difference between the sample and reference (or counterweight depending on the instrument design), if the reference is contaminated with a hygroscopic material, the sorption of water vapor by the contaminant will result in the apparent decrease in sample mass with increasing RH. In this case, contamination was found both on the reference pan and the chain which held the pan. After this issue was identified and the reference pan and chain² were cleaned, the experiment was repeated with much improved mass stability.

3.4 Relationship between temperature, relative humidity, and mass stability

To better understand whether there was a correlation between mass and temperature or relative humidity stability, these

² Other laboratories have noted that microcracks in the chain or a sample pan can allow capillary condensation and give rise to unusual sorption behavior on inert samples.

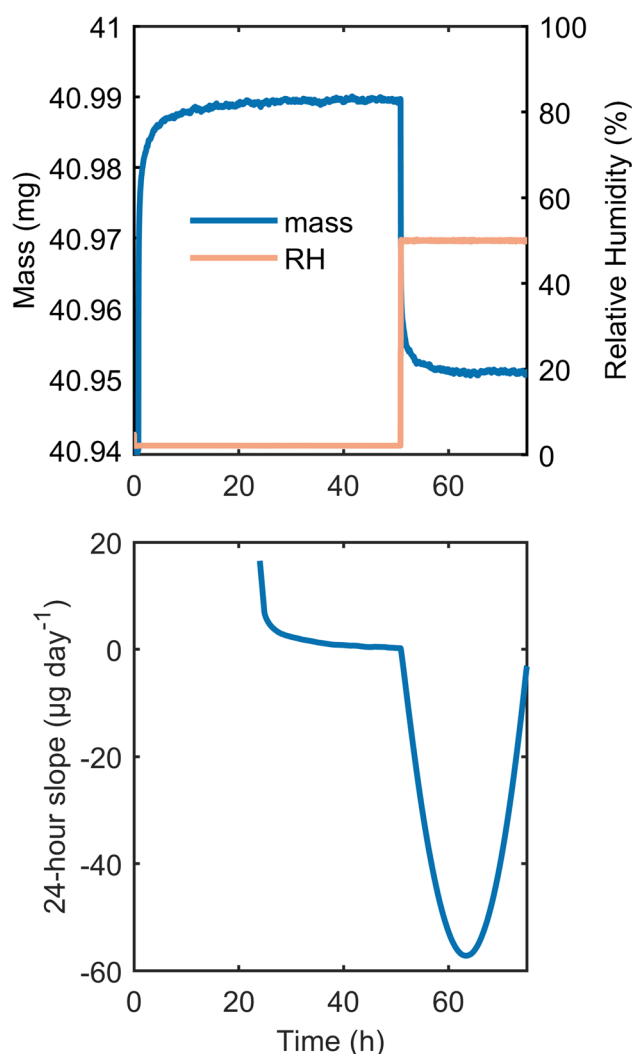


Fig. 9 Top) Example mass vs. time and RH vs. time data indicating contamination of the reference pan. (bottom) Corresponding 24 h slope

variables are plotted against each other in Fig. 10. From Fig. 10 it can be seen that there are laboratories with very stable mass measurements that have high temperature or humidity instabilities. In the same manner, laboratories with the largest mass instabilities did not necessarily have an unusually high temperature or relative humidity instability. Near the origin there may be some correlation between the temperature or humidity stability and the mass instability. However, for hygroscopic samples, temperature and humidity stability become much more important to overall mass stability.

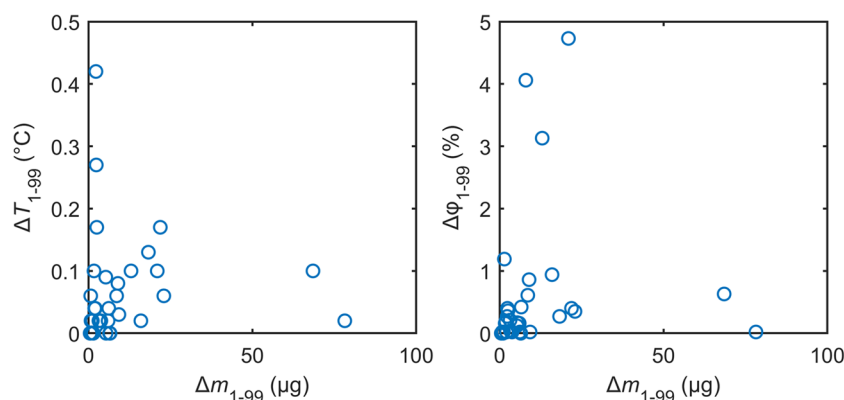
4 Conclusions

This study provides important data on the operational stability of automated sorption balances in laboratory environments around the world. Temperature, relative humidity, and mass stability were analyzed based on 50 h of data at constant temperature and RH with a non-hygroscopic sample. Nearly half of the laboratories reported data with mass instability greater than an initial target of $4 \mu\text{g day}^{-1}$.

Mass stability is important for automated sorption balances because accurate measurements of the water sorption process at extended times rely on it. Mass stability is rarely reported in the literature. This interlaboratory study is the first formal investigation of stability using automated sorption balances across a variety of instruments and laboratory conditions. Although many laboratories were able to meet an initial target for instrument mass stability on the first attempt, some instruments were found to have unexpectedly large instabilities.

Although a mass instability of $10 \mu\text{g}$ may seem insignificant when compared to the total mass of the inert sample used, the relative error in an absorption experiment would be much higher. At low RH steps, a 20–40 mg hygroscopic sample may absorb less than 1 mg of water. At long absorption times and low relative humidities, instrument instability may have an appreciable effect on the absorption measurement.

Fig. 10 Comparison of the mass stability against the temperature and relative humidity stability



Several different sources of mass instability were observed across laboratories, including apparent instrument warm-up, disturbances from vibration or accidental bumps, and contamination of the reference pan or counterweight. Most laboratories improved their instrument mass stability after correcting the issue and repeating their measurements. These observations highlight the need for awareness of potential sources of error. In addition to regular verification of balance calibration, which is common practice, this study highlights the need for regular stability checks to confirm that the instrument is performing as intended.

Given the number of laboratories that did not meet the initial mass stability target, we recommend that laboratories include mass stability data as supplementary information when publishing new sorption data in the literature. The supplementary information should include a mass stability check using a calibration weight or other non-hygroscopic object.

The results of this study will inform the uncertainty analysis for ongoing work on water vapor sorption measurements in hygroscopic materials.

Appendix

Table 2 Mass stability data from the 25 laboratories

Laboratory	Δm_{1-99} (μg)		$IS_{1-h} _{\text{max}}$ ($\mu\text{g h}^{-1}$)		$IS_{24-h} _{\text{max}}$ ($\mu\text{g day}^{-1}$)	
	First dataset	Best dataset ^a	First dataset	Best dataset	First dataset	Best dataset
1	0.52	0.52	0.40	0.40	0.46	0.46
2	0.61	0.61	0.49	0.49	0.40	0.40
3	0.7	0.7	0.19	0.19	0.51	0.51
4	9.3	0.8	12.47	0.38	11.90	0.57
5	1.04	1.04	0.52	0.52	0.90	0.90
6	78.2	1.2	80.79	0.36	36.45	0.52
7	1.46	1.46	0.37	0.37	1.09	1.09
8	1.9	1.9	0.67	0.67	1.24	1.24
9	6.2	2.1	2.91	0.97	6.80	1.04
10	2.3	2.3	4.22	4.22	1.99	1.99
11	2.4	2.4	2.31	2.31	2.50	2.50
12	21.93	2.55	17.16	12.97	18.19	1.98
13	6.0	3.2	1.53	3.38	6.07	1.83
14	3.3	3.3	3.67	3.67	3.12	3.12
15	3.8	3.8	1.61	1.61	2.63	2.63
16	18.3	5.3	2.14	0.52	12.74	3.16
17	5.4	5.4	4.41	4.41	3.63	3.63
18	6.46	6.46	1.85	1.85	5.06	5.06
19	68.5	6.6	54.79	1.75	23.85	6.04
20	106.3	8.0	75.24	5.47	58.19	4.47
21	8.6	8.6	5.16	5.16	4.07	4.07
22	23	9	2.31	2.24	15.10	4.09
23	13	13	3.52	3.52	8.73	8.73
24	16.0	16.0	26.77	26.77	6.21	6.21
25	1929	21	845.55	20.48	845.59	21.59
Mean	16.88	4.43	12.73	3.51	9.66	2.76
Median	6.1	2.9	2.61	1.80	4.57	2.25
Maximum	106.3	16.0	80.79	26.77	58.19	8.73
Minimum	0.52	0.52	0.19	0.19	0.40	0.40

^aNote: The “best dataset” was determined from the dataset submitted from each laboratory with the lowest Δm_{1-99} , which also corresponded with the lowest $IS_{24-h}|_{\text{max}}$. In some cases, this dataset had a higher $IS_{1-h}|_{\text{max}}$ than the first reported run

Table 2

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Author contributions CRediT Taxonomy:

Conceptualization: SLZ, SVG, EET, Investigation: All authors, Data Curation: SLZ, SVG, EET, EQDL, Formal Analysis: SLZ, SVG, EET, EQDL, Visualization: SLZ, EET, SVG, Writing—Original Draft: SLZ, SVG, EET, Writing—Review & Editing: All authors, Project administration: SLZ, SVG, EET.

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Data availability Data files are available at the US Forest Service Data Archive Repository at the following URL <https://www.fs.usda.gov/rds/archive/catalog/RDS-2024-0025>. It has been assigned a DOI number: <https://doi.org/10.2737/RDS-2024-0025>.

Declarations

Ethical approval This study does not involve human or animal studies, so no there is nothing to declare.

Competing interests Several coauthors work for companies that sell automated sorption balances. All data have been anonymized to prevent a comparison across instrument type or models to address potential conflicts due to commercial interests.

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