



Interlaboratory study of the quality of water vapor sorption data for wood from automated sorption balances

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Abstract

Automated sorption balances are widely used for characterizing the interaction of water vapor with hygroscopic materials. This paper is part of an interlaboratory study investigating the stability and performance of automated sorption balances. A previous paper in this study investigated the mass, temperature, and relative humidity (RH) stability of automated sorption balances by looking at the mass change of a non-hygroscopic sample over time. In this study, we examine the mass stability of wood samples held at constant RH for seven to ten days after a step change. The reason for the long hold times was to collect data to “operational equilibrium” where the change in mass is on the order of the inherent operational stability of the instrument. A total of 80 datasets were acquired from 21 laboratories covering absorption with final RH levels ranging from 10 to 95%. During these long hold times, several unusual behaviors were observed in the mass-vs-time curves. Deviations from expected sorption behavior were examined by fitting the data to an empirical sorption kinetics model and calculating the root mean square error (RMSE) between the observed and smoothed behavior. Samples that had a large RMSE relative to the median RMSE of the other datasets often had one of several types of errors: abrupt disturbances, diurnal oscillations, or long-term mass decline during an absorption step. In many cases, mass fluctuations were correlated with changes in the water reservoir temperature of the automated sorption balance. We discuss potential errors in sorption measurements on hygroscopic materials and suggest an acceptable level of RMSE for sorption data.

Keywords Water vapor sorption · Dynamic vapor sorption · Sorption isotherms · Interlaboratory study

1 Introduction

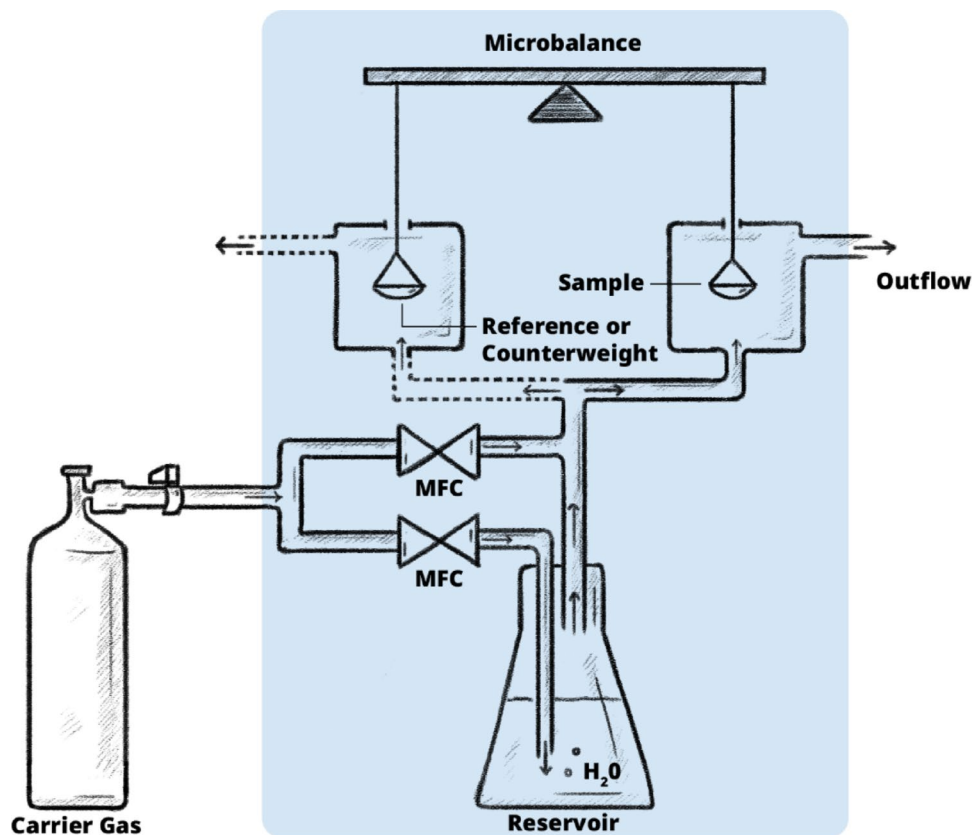
Investigating moisture sorption in materials is important for a wide range of research fields from food science to construction and materials science. Commonly, moisture sorption is investigated by gravimetric techniques where the relative humidity (RH) is held constant at a specific temperature and the mass is measured over time until an equilibrium criterion is met.

While there are many ways to measure sorption isotherms, one popular technique utilizes an automated sorption

balance (often referred to as “Dynamic Vapor Sorption” or “DVS”) ¹. In these instruments, a small material sample (milligram range) is located inside a temperature-controlled chamber, and the surrounding RH is controlled by mass flow controllers that mix dry and water vapor-saturated streams of carrier gas (often nitrogen or dry air) (see Fig. 1). The

¹ Note that there are also automated vacuum sorption balances where the sample is under vacuum and water vapor is the only gaseous component introduced to the sample. However, this paper focuses on automated sorption balances that operate with continuous flow of carrier gas under atmospheric pressure.

Fig. 1 Generic schematic of an automated sorption balance. Note that some instruments isolate the microbalance counterweight whereas in others an empty reference pan is exposed to the same environment as the sample (illustrated by the dotted lines in the figure). The abbreviation MFC refers to “mass flow controller”. Illustration by Dorothy Punderson of the US Forest Service, Forest Products Laboratory



water-saturated stream is generated by bubbling the carrier gas through a reservoir of water. In these experiments, the sample mass is continuously recorded by a microbalance which allows for the sorption kinetics to be observed [1–6].

To construct a sorption isotherm, automated sorption balances can be programmed to collect data at a constant RH until predefined equilibrium conditions are met and then automatically move on to collect data at the next desired RH. Automated sorption balances therefore require the operator to decide how long to take data by programming stop criteria. Most commonly, stop criteria are based off the rate of change in mass (dm/dt) or moisture content (du/dt); however, automated sorption balances can also be programmed to collect data for a predetermined amount of time before moving to the next sorption step.

For wood and other lignocellulosic materials, a du/dt of $0.002\% \text{ min}^{-1}$ (or equivalently, $20 \mu\text{g g}^{-1} \text{ min}^{-1}$) over 10 min was widely used as a stop criterion [7–19]. Early research claimed that the equilibrium moisture content measured stopping at $20 \mu\text{g g}^{-1} \text{ min}^{-1}$ was within 0.001 g g^{-1} (0.1% MC, absolute error) of the true equilibrium moisture content [13]. However, when sorption steps were carried out for much longer periods of time (4–7 days at each RH step), it was found that the $20 \mu\text{g g}^{-1} \text{ min}^{-1}$ criterion produced

errors as large as 0.012 g g^{-1} (1.2% MC, absolute error) [20, 21].

The research of Glass et al. [20, 21] has highlighted the potential errors of using a $20 \mu\text{g g}^{-1} \text{ min}^{-1}$ stop criterion for wood and other lignocellulosic materials. However, collecting data for up to 7 days at each RH step in a sorption balance is impractical for most circumstances. Glass et al. [21] showed that short hold times cause a systematic error (underprediction of equilibrium moisture content in absorption, overprediction of equilibrium moisture content in desorption). Furthermore, they showed that these systematic errors appear to vary linearly with RH and suggested how a correction factor could be used to better predict equilibrium moisture content even with short hold times. However, more data is needed to develop these correction factors which may depend upon the material.

In 2021, a worldwide interlaboratory study was initiated to gather sorption data on wood with long hold times (to equilibrium²). The first report from this study concerned the mass stability of the instruments as determined from measurements with an inert sample consisting of aluminum

² Glass et al. [21] defined “equilibrium” for sorption balance measurements as a change in mass less than or equal to the instrument stability over a 24-h period measured with a non-hygroscopic reference material.

[22]. Several laboratories had high levels of instability in their instrument. In many of these cases the performance was improved until it met what was considered an acceptable level of mass stability of $4 \mu\text{g day}^{-1}$, based on a 24-hour slope. The temperature and RH of the automated sorption balances were also characterized in these experiments; however, these variables were not correlated to the mass stability of the inert sample.

In this second paper from the interlaboratory study we present mass stability data during sorption measurements with wood samples. The goal of the paper is to develop tools to evaluate the data quality of sorption measurements on wood samples. This requires isolating the expected mass change due to sorption from mass changes due to environmental disturbances, such as noise, vibrations, or temperature and humidity changes. We tie these evaluation tools to previous work that evaluated the quality of literature sorption data for wood. In addition, we highlight various types of mass instabilities, identify correlations between mass changes and other instrument parameters, and make suggestions on sorption balance operation to achieve the most stable readings possible.

2 Materials and methods

2.1 Materials

Laboratories were provided with a non-hygroscopic material to test the stability of the instrument and wood samples

to examine sorption behavior. 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 tested in the study.

Wood samples consisted of 1 mm thick cross sections of western larch (*Larix occidentalis* Nutt.) heartwood. Samples were cut from a single US nominal “2×4”, a board with dimensions of 38 mm by 89 mm by 2.44 m. The board was chosen from a large pallet of visually graded #1 boards of the “Douglas Fir-Larch” sawn lumber commercial species combination [23]. The board was chosen because it was free of knots and had a straight grain pattern (Fig. 2a).

Hand chisels were used to shape a section of the board according to its true radial and tangential anatomical directions (Fig. 2b and c). From the original 38 mm by 89 mm cross section, a 20 mm (radial) by 40 mm (tangential) straight-grained section was obtained (Fig. 2d). This 20 by 40 mm block was then cross sectioned on a precision table saw into 1 mm thick slices (Fig. 2e). From these 20 by 40 by 1 mm slices, 6 by 6 by 1 mm samples were cut (Fig. 2f). A custom jig was made to cut these smaller, square samples repeatably. The jig consisted of a 3D-printed plastic block wherein two razor blades could be held approximately 6 mm (0.25 inches) apart. This tool was then placed on top of the 20 by 40 mm samples which, when struck by a hammer, cut them into 6 by 20 mm sections at which point the jig was turned 90 degrees and the process was repeated. Samples had a dry mass of around 20 mg. Samples were placed in

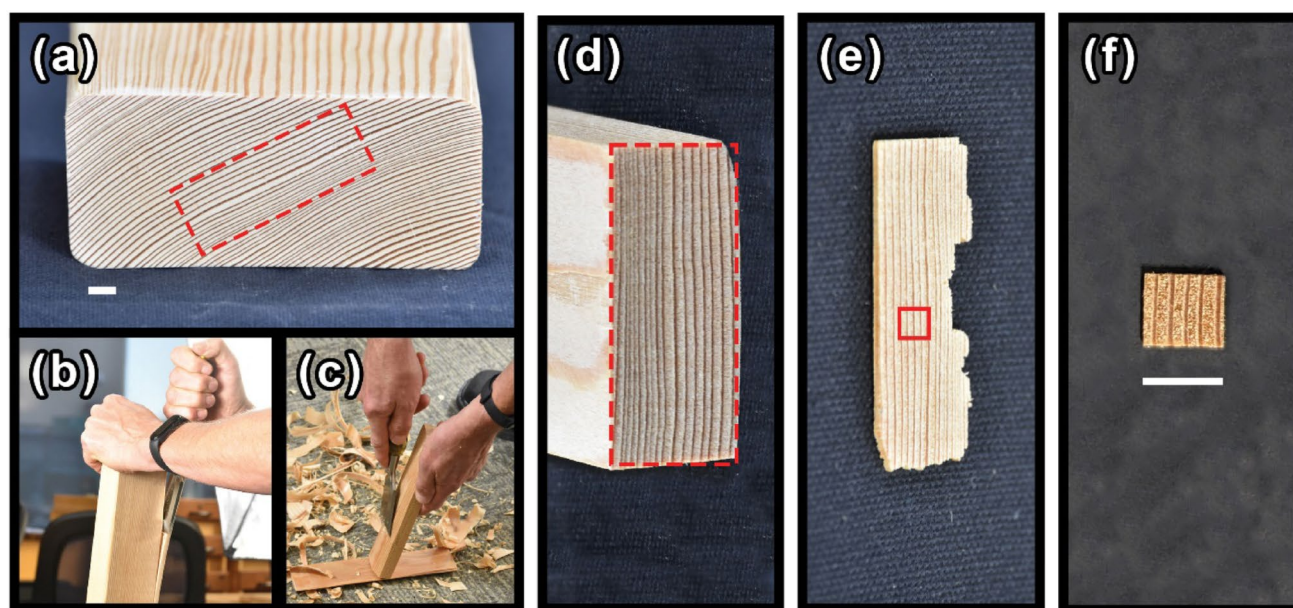


Fig. 2 Photographs showing how the sample (f) was created from a commercial “2×4” (a). The board was first chiseled (b–c) into its true radial and tangential directions (d) before being cut into 1 mm thick

cross sections (e). The sample dimensions in (a) are 38×90 mm and 6×6 mm in (f). The scale bar is 6 mm in both (a) and (f)

Table 1 List of sorption balances that supplied data on larch specimens for the interlaboratory study

Manufacturer	Model(s)
Hidden Isochema Ltd.	IGAcorp
Surface Measurement Systems Ltd.	DVS Advantage, DVS Advantage Plus, DVS Endeavour, DVS Intrinsic, DVS Resolution
TA Instruments–Waters LLC	Discovery SA, Q5000 SA

polyethylene bags filled with molecular sieve (4Å) desiccant. Laboratories were asked to keep the samples in the sealed bags prior to testing.

2.2 Experimental methods

Each laboratory was asked to contribute two types of measurements: a measurement of the instrument stability using a non-hygroscopic aluminum sample pan at 50% RH, and measurements on larch samples with a long hold time (7 or 10 days depending on the RH) with the goal of achieving operational equilibrium within that timeframe. Laboratories were asked to contribute at least two datasets on larch samples.

In total, 21 labs contributed larch sorption data to the interlaboratory study using instruments of several different models and manufacturers (Table 1).

Instrument stability measurements were described in our previous publication [22]. In summary, an aluminum sample pan was loaded into the instrument at 25 °C with a carrier gas flow rate of 200–250 cm³ min⁻¹. Data were collected for 3000 min with a relative humidity of 50% RH with data recorded at intervals of 1 min or less.

The same data acquisition rate, carrier flow rate, and temperature were used to collect the data on larch specimens. Prior to the sorption steps, the sample was first held at 0% RH (dry carrier gas) at 25 °C for 24 h, yielding a preliminary dry mass, m_{dry} . Sorption sequences had one “long” step held at a final RH value listed in Table 1, preceded by anywhere from zero to nine “short” steps at each of the RH levels below the final RH. The “long” step had a specified duration of seven days based on time to reach operational equilibrium for previous data [21]. For final RH values of 90% and 95%, the duration of the “long step” was revised to 10 days part way into the study based on data received. The stop criterion for the “short” steps was a du/dt value [= $(1/m_{\text{dry}})dm/dt$] of 0.002% min⁻¹ within a 10 min window, with a maximum time of 600 min per step. An example protocol for a “long step” at 40% RH is as follows:

Table 2 Number of datasets taken at each relative humidity (RH) step along with the number of instruments used to collect the data

Initial RH (%)	Final RH (%)	Instruments	Datasets
0	10	4	5
10	20	6	16
20	30	4	6
30	40	4	6
40	50	4	7
50	60	4	4
60	70	4	8
70	80	4	6
80	90	5	11
90	95	5	11

- Target: 0% RH, Stop criterion: Time, Period: 24 h (= 1440 min).
- Target: 10% RH, Stop criterion: du/dt , Value: 0.002% min⁻¹.
- Target: 20% RH, Stop criterion: du/dt , Value: 0.002% min⁻¹.
- Target: 30% RH, Stop criterion: du/dt , Value: 0.002% min⁻¹.
- Target: 40% RH, Stop criterion: Time, Period: 168 h (= 10080 min).

The original study plan called for at least five replicates of long hold times for each RH step and at least one replicate from each instrument manufacturer. As is common in interlaboratory studies, some laboratories were unable to complete the full range of measurements, but other laboratories provided additional data. In addition, some laboratories were asked to repeat measurements with noticeable abnormalities in the sorption data. In total 80 measurements were taken to operational equilibrium. For each relative humidity step, at least four datasets (replicates) were collected, from at a minimum of four different instruments (laboratories). The distribution of the measurements across the different RH steps is given in Table 2.

2.3 Data analysis

For this study, data quality was determined by observing deviations from expected behavior. For the non-hygroscopic materials tested in the first part of the interlaboratory study [22], measurement instabilities could easily be identified since the tested material did not absorb moisture. For hygroscopic materials, one must separate mass changes caused by the sorption process from mass changes caused by limitations in the measurement technique. We found that

a discrete exponential model with five components, i.e. a penta-exponential kinetics model (which we abbreviate to 5EK) worked best for this purpose (see *Supplementary Information* for a detailed explanation and justification and comparison against other potential techniques). The 5EK model can be described by

$$m_{5EK} = m_0 + (m_\infty - m_0) \sum_{n=1}^5 A_n \left[1 - \exp\left(-\frac{t}{\tau_n}\right) \right] \quad (1)$$

where m is mass (g), t is time (s), A_n is the amplitude of the n^{th} component (-), τ_n is the time constant of the n^{th} component (s), and subscripts “0” and “ ∞ ” refer to the initial and final mass, respectively; note that the sum of all A_n must equal 1 and A_n are greater than 0. The model is thus constrained to give a positive first derivative that gradually decreases with time, consistent with expected sorption behavior when the temperature and RH are held constant after a step change in RH.

The 5EK model was fitted to each dataset by minimizing the sum of squared residuals between model and measured mass using the Excel Solver function (Microsoft Corp., Redmond, WA, USA). Full details are in the *Supplementary Information*. Allowing more than five components did not improve the fit. In many cases, the best fit included five components, but the routine allowed the model to effectively collapse to fewer components (by making one or more time constants practically identical, or by adjusting the amplitude of a given component to nearly zero).

After the expected mass change from sorption was determined by the 5EK model, the residuals were calculated as the difference between measured data and 5EK fit. The

overall mass instability was quantified by the root mean square error (RMSE).

Instabilities in the temperature and RH measurements during each sorption step, as well as instabilities in the mass measurements of the non-hygroscopic aluminum sample pan were assessed by calculating the standard deviations (SDs) from their mean values. For a parameter with an expected constant value, the RMSE is equivalent to the SD. The SD calculations for RH excluded the first 5 min of data to avoid transitional delays in RH measured close to the samples.

3 Results and discussion

3.1 RMSE as an indicator of data quality

Figure 3 presents an example of experimental data with a 5EK fit along with the residuals plotted two different ways. The data in Fig. 3 had the median RMSE (1.1 μg) of all data sets analyzed and was from a 50–60% RH step. The left panel shows the mass gain along with the 5EK curve fit over the 7-day absorption step. The middle panel shows the residuals as a function of time; there are no general trends in the residuals that would suggest a structural bias in the model fit. Instead, the data are more or less evenly distributed within a 4 μg band of the curve fit. Finally, the right panel plots a histogram of the residuals along with a normal distribution, with the data following the general shape of this curve.

Further support for the suitability of the 5EK model can be found in Figure S6, where examples are shown of the

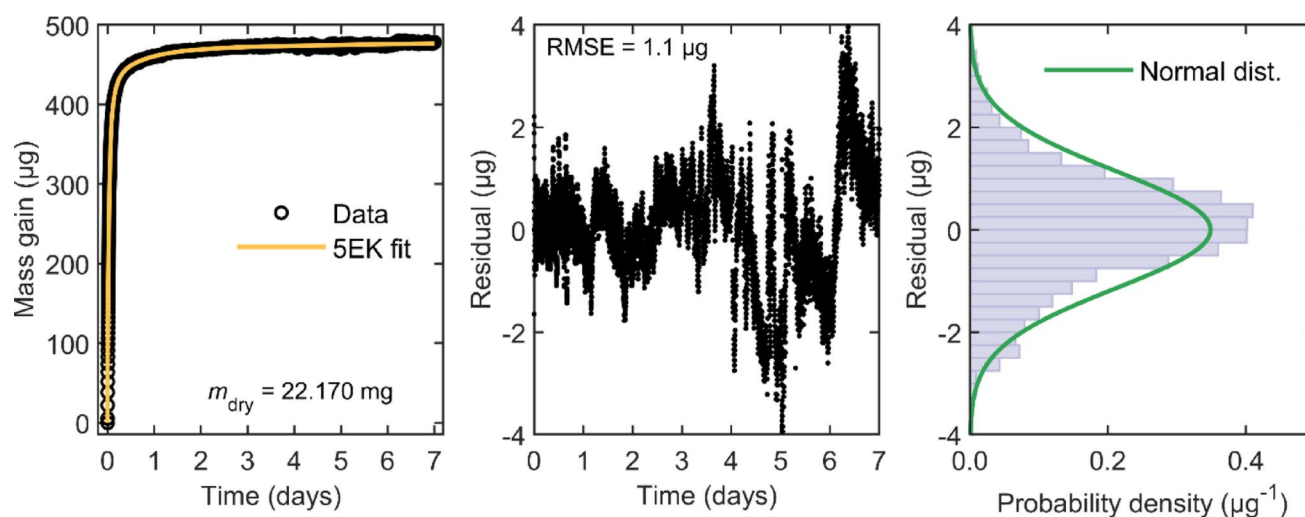


Fig. 3 Example of an absorption step with the 5EK fit overlaid in yellow. The RH step was 50–60% and this example had the median RMSE of all analyzed data. The left panel shows the raw data and the curve fit. The middle panel plots the residuals as a function of time.

The right panel plots the residuals as a histogram according to their probability density. The normal distribution is overlaid on the histogram for reference.

best data collected on three different instruments, one from each instrument manufacturer involved in this study.

It is important to emphasize that while the 5EK model can be used to identify abnormalities in sorption data, the parameters of the 5EK model fit do not have physical significance. Many sorption steps contain fewer than five time constants. In these cases, the fitting routine can find many ways to partition the amplitudes and time constants. Additionally, in some instances the resulting model parameters could vary depending on initial values. Similar RMSEs could be obtained with different ensembles of time constants and weights.

Figure 4a shows the cumulative distribution of RMSE values obtained for larch in this study from all of the laboratories and RH steps (see Appendix for tabulated values). The data are plotted along with the cumulative distribution of standard deviations found using the non-hygroscopic aluminum sample pans [22]. The larch RMSEs ranged from 0.2 μg to 46.9 μg , in contrast with the Al pan SDs, which ranged from 0.1 μg to 2.2 μg . The 90th percentile value (indicated by dotted lines in the inset of Fig. 4a) was 1.5 μg for the Al pan and 6.0 μg for larch. Figure 4b plots the larch RMSEs vs. the Al pan SDs from the same instruments. Mass instabilities observed in the larch data are not correlated with the basic operational stability of the automated sorption balances. The greater range of mass instability in the larch data likely reflects the response of the hygroscopic sample to instabilities in the temperature or relative humidity near the sample, as discussed further below.

3.2 Classification of mass instabilities

This section focuses on datasets that have large RMSE values indicative of random or systematic mass changes that differ from the expected sorption behavior for larch. We first discuss the types of patterns found in the mass measurements before a deeper discussion of the likely causes for these patterns.

3.2.1 Abrupt disturbances

The first type of issue that can be easily identified is a random spike or offset in the mass, as shown in Fig. 5. These issues could be caused by a disturbance to the microbalance such as a vibration or shock to the laboratory floor or instrument itself. Another possibility is that a disturbance caused a change to the mass flow controllers that affected the RH near the sample. In the case of a shock to the mass flow controller, the change in mass would not be instantaneous but instead exhibit a lag as the sorption process occurred. Depending on the type and severity of the disturbance, the data may either return to a very similar sorption pattern or exhibit a permanent offset in the sorption curve. Two further examples are shown in Figure S7.

3.2.2 Diurnal oscillations

Another common issue we identified in the data was diurnal oscillations in the mass, as shown in Fig. 6. The magnitude of these diurnal oscillations varied widely across datasets,

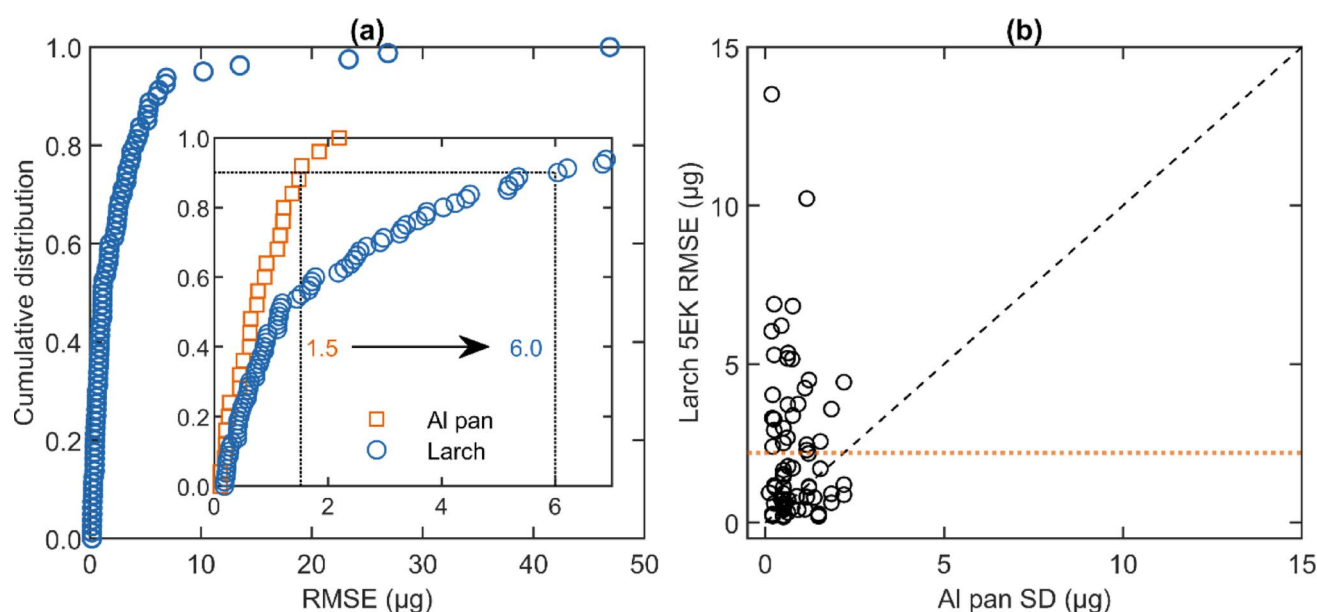


Fig. 4 (a) Cumulative distribution of RMSE values. Inset: Comparison with aluminum pan SD values. Dotted lines represent 90th percentile values. (b) Larch RMSE values vs. Al pan SD values from the same

instrument; orange horizontal dotted line indicates the range of aluminum pan SD values (2.2 μg)

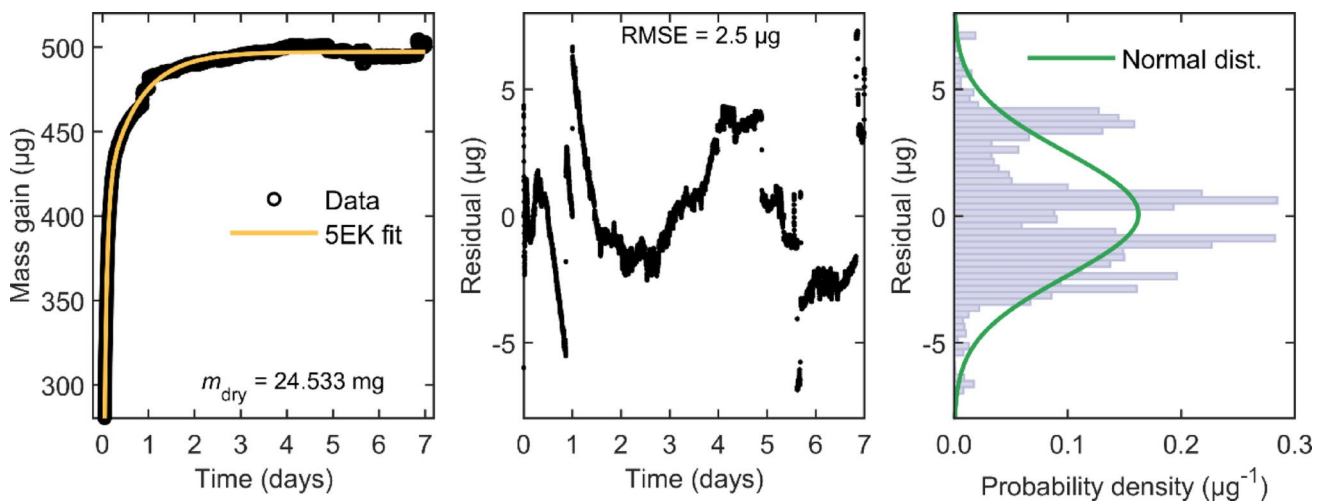


Fig. 5 Example of data with offsets in the residuals from a 30–40% RH step. The left panel shows the raw data and the curve fit. The middle panel plots the residuals as a function of time. The right panel plots

the residuals as a histogram according to their probability density. The normal distribution is overlaid on the histogram for reference

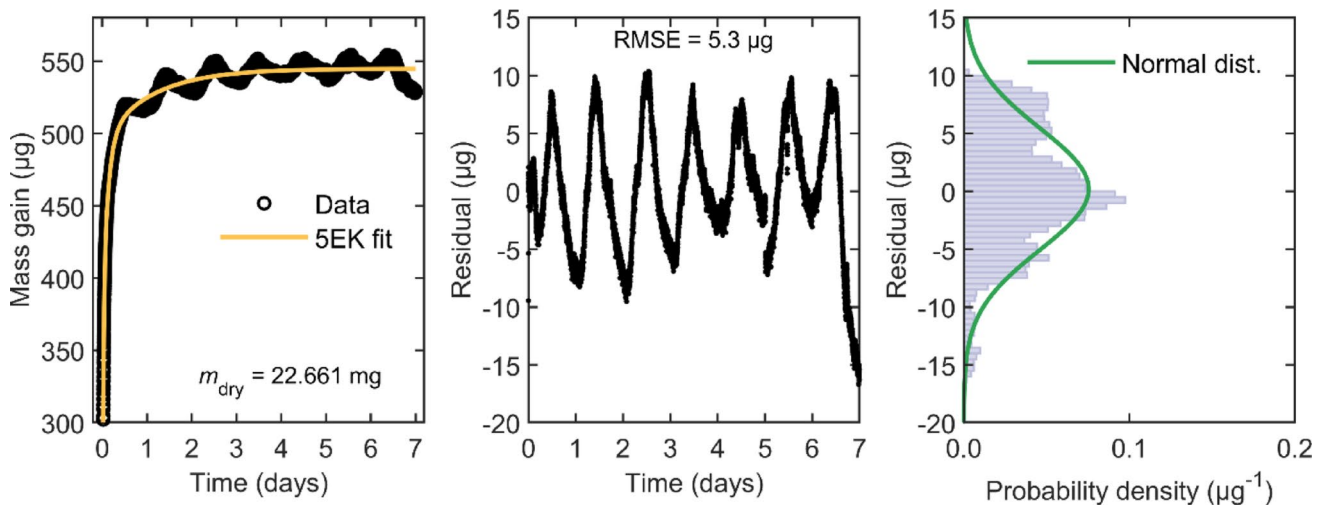


Fig. 6 Example of data with oscillating patterns in the residuals from a 60–70% RH step. The left panel shows the raw data and the curve fit. The middle panel plots the residuals as a function of time. The

right panel plots the residuals as a histogram according to their probability density. The normal distribution is overlaid on the histogram for reference

with examples in Figure S8 (*Supplementary Information*) having RMSE values from 0.8 μg to 10.2 μg .

While in some cases the magnitude of the RMSE is small, the fact that there are patterns in the residuals suggests that the mass is not following an expected behavior for sorption. In many cases, these oscillations arise from fluctuations in the relative humidity around the sample (see Sect. 3.3). Since the sorption isotherm for wood has a very steep slope at high RH, it is not surprising that the RMSE

from these oscillating patterns (or RH disturbances in general) increases with relative humidity [24]. For the example shown above and further examples in the *Supplementary Information*, the RMSE increases with increasing RH:

- 0.8 μg for 0–10% RH (Figure S8).
- 1.7 μg for 40–50% RH (Table S1).
- 5.3 μg for 60–70% RH (Fig. 6).
- 10.2 μg for 80–90% RH (Figure S8).

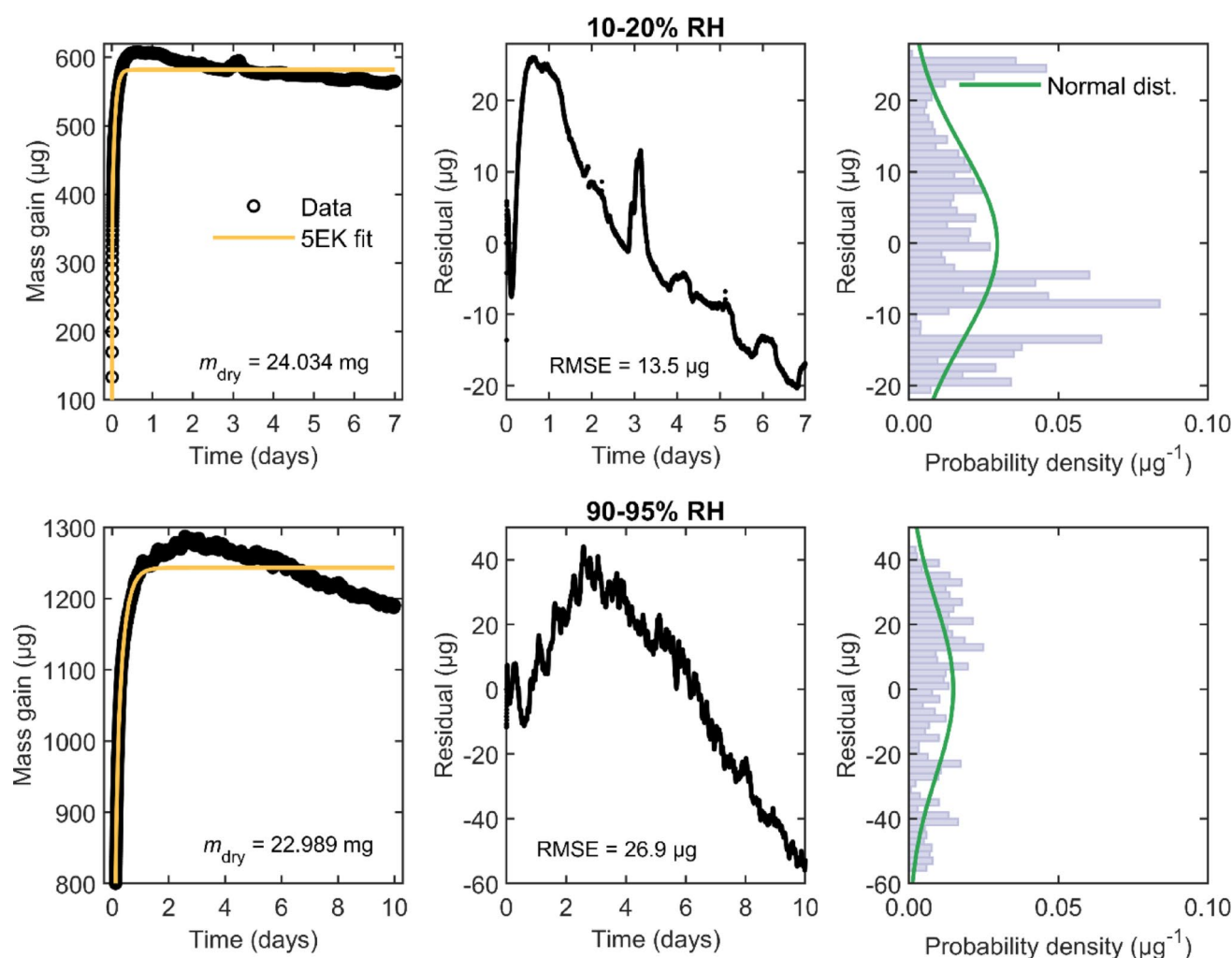


Fig. 7 Examples of data with long-term mass decline from two different relative humidity (RH) steps. The left panel shows the raw data and the curve fit. The middle panel plots the residuals as a function

of time. The right panel plots the residuals as a histogram according to their probability density. The normal distribution is overlaid on the histogram for reference

3.2.3 Long-term mass decline

Finally, Fig. 7 illustrates datasets that had appreciable decreases in the mass over time. There is no physical reason for the mass to decrease in an absorption experiment. These datasets could be identified as unphysical from the shape of their mass vs. time curves. However, the RMSEs of these datasets are also much higher than the median RMSE value. In these datasets, the 5EK model does not fit the data well, but instead minimizes the error by fitting an asymptote between the maximum value reached in the sorption test and the final value at the end of seven or 10 days. Similar to Fig. 6, the data in Fig. 7 suggest that there may be an issue with the local relative humidity near the sample (see Sect. 3.3).

3.3 Correlations between mass instabilities and instrument conditions

Figure 3 and Figure S6 showed that the 5EK model could fit high quality sorption data well. In the cases with large RMSE values, the data suggested that the mass changes were caused by either (1) a physical disturbance to the instrument or (2) a disturbance in the environmental conditions surrounding the sample.

In addition to recording the mass, automated sorption balances also record the temperature and relative humidity as a function of time. Since the local environmental conditions around the sample appeared to contribute to many of the high-RMSE data sets, it seems logical to see if there are any correlations between the instrument temperature and humidity variations and mass fluctuations.

Fig. 8 Larch RMSE vs. SD of sample temperature and relative humidity

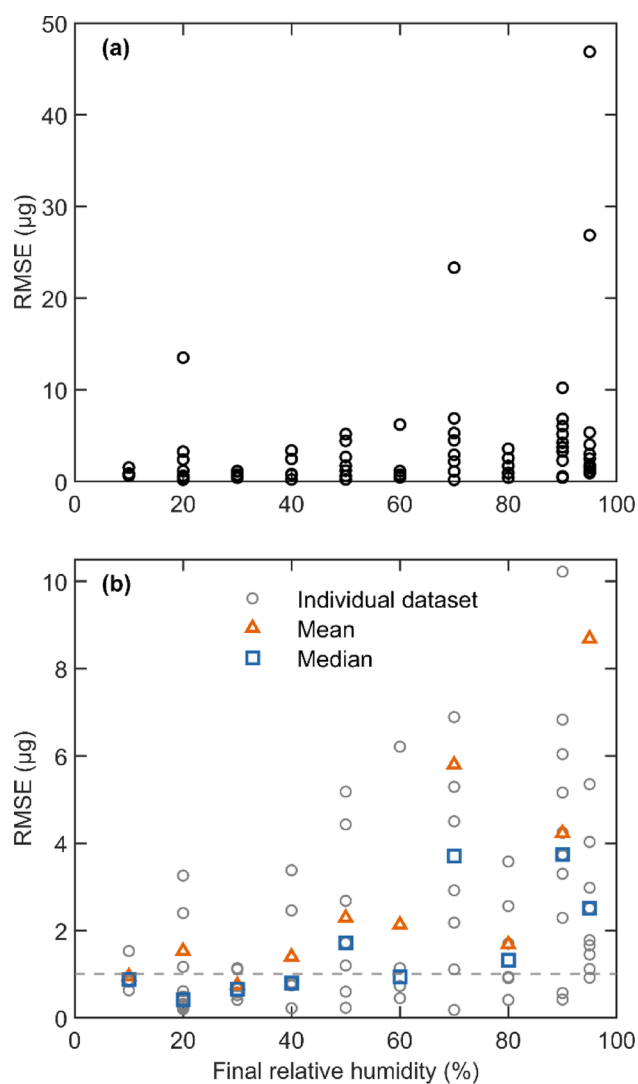
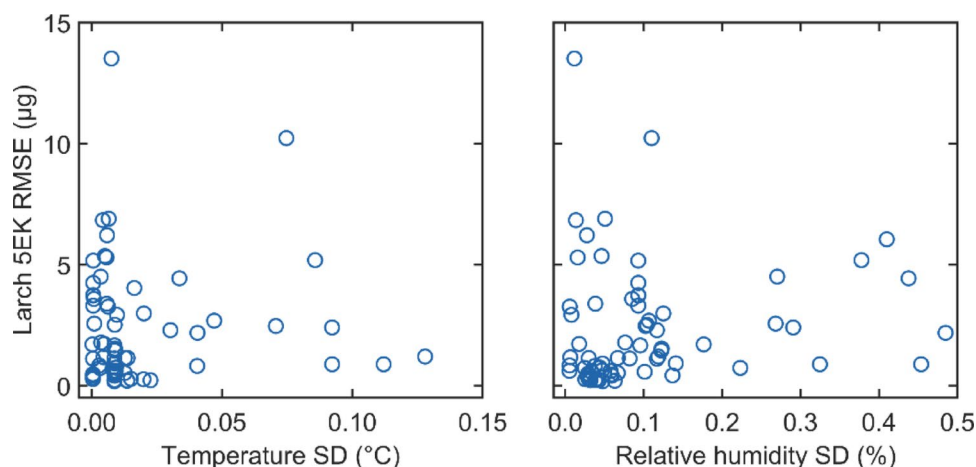


Fig. 9 (a) RMSE for each dataset in the study plotted as a function of the final relative humidity in the RH step of interest; (b) Same data as in (a) with truncated y-axis, along with mean and median values; horizontal dashed line shows that an RMSE of 1 μg or less is attainable across the full RH range

Figure 8 shows lack of correlation between the larch RMSE and the SD of the sample temperature and relative humidity. At first glance the lack of correlation may suggest that the mass oscillations or other instabilities during sorption were not caused by environmental fluctuations. However, when the same data are plotted as a function of the final RH (Fig. 9), the RMSE generally increases with increasing RH. The individual datasets exhibit a wide range of RMSE values (Fig. 9a), but an RMSE of 1 μg or less is attainable across the full range of RH values (dashed line in Fig. 9b). For comparison, the 90th percentile SD value for the mass of a non-hygroscopic sample was 1.5 μg (Fig. 4a). The mean RMSE values are consistently greater than the median RMSE values (Fig. 9b) because the median is less sensitive to extremes. Given the small sample of different instruments represented at each RH level (between 4 and 6 instruments; see Table 1) and the potential for multiple types of mass instabilities discussed above, one should not expect a monotonic increase in the mean or median RMSE with increasing RH. While the global trend in Fig. 9 does not establish correlation between mass fluctuations and environmental conditions, closer examination of individual samples can help explain the high RMSE values in the sorption data, as can be seen in the following examples.

Figure 10 shows the sample temperature (a), sample relative humidity (b), and residual between the sample mass and the 5EK fit (c) for an RH step from 80 to 90%. This sample exhibited diurnal temperature fluctuations, with a magnitude of approximately 0.25 $^{\circ}\text{C}$. These spikes are notated with a gray vertical dashed line that extends to subfigures (b) and (c). The spikes are correlated with an RH change of approximately 0.2% and a mass change of approximately 30 μg . Figure 10 illustrates how very small changes in temperature can be correlated with large swings in absorbed mass.

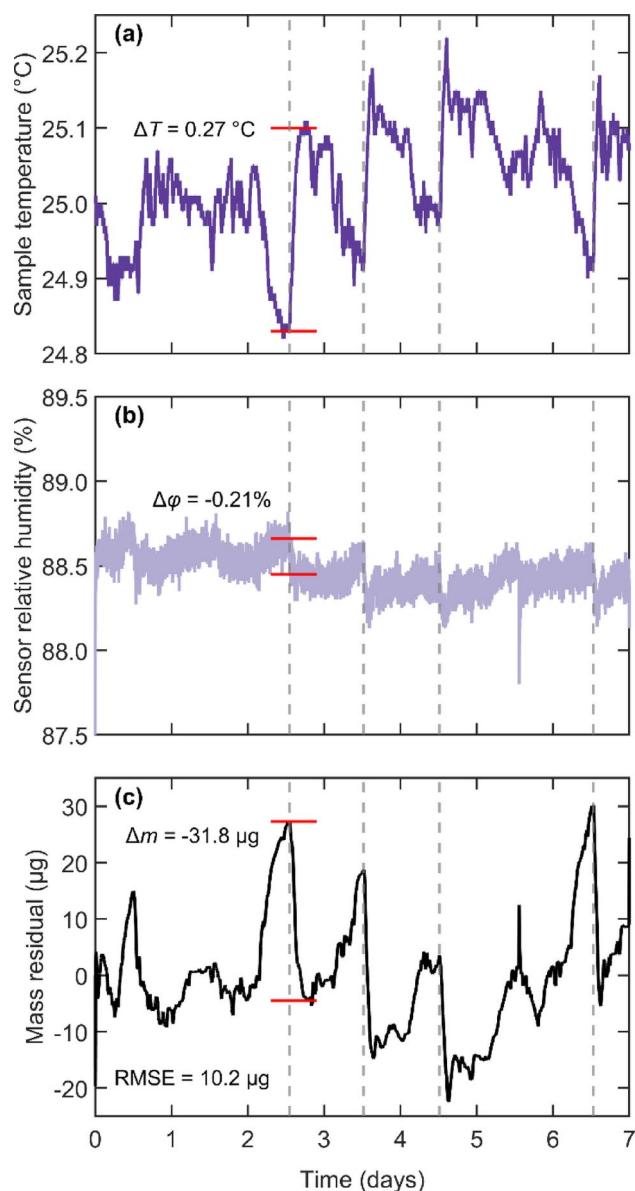


Fig. 10 Time series of sample temperature (a), sensor relative humidity (b), and mass residuals from the 5EK model fit to sample mass (c) for a step from 80–90% RH, with vertical dashed lines at select time values to show correlation and horizontal bars indicating changes in each parameter for a representative fluctuation

In contrast to Figs. 10 and 11 is an example with almost no variation in the sample temperature, yet exhibits appreciable mass fluctuations (an additional example of this behavior is given in the *Supplementary Information*, Figure S9). In this case the sample temperature remained within ± 0.02 °C of the 25.00 °C target. Despite the extremely stable sample temperature, the relative humidity sensor readings fluctuated between 78.3% and 79.3% with several prominent dips that correlate with corresponding dips in the mass residuals (subfigure d, see vertical dashed lines).

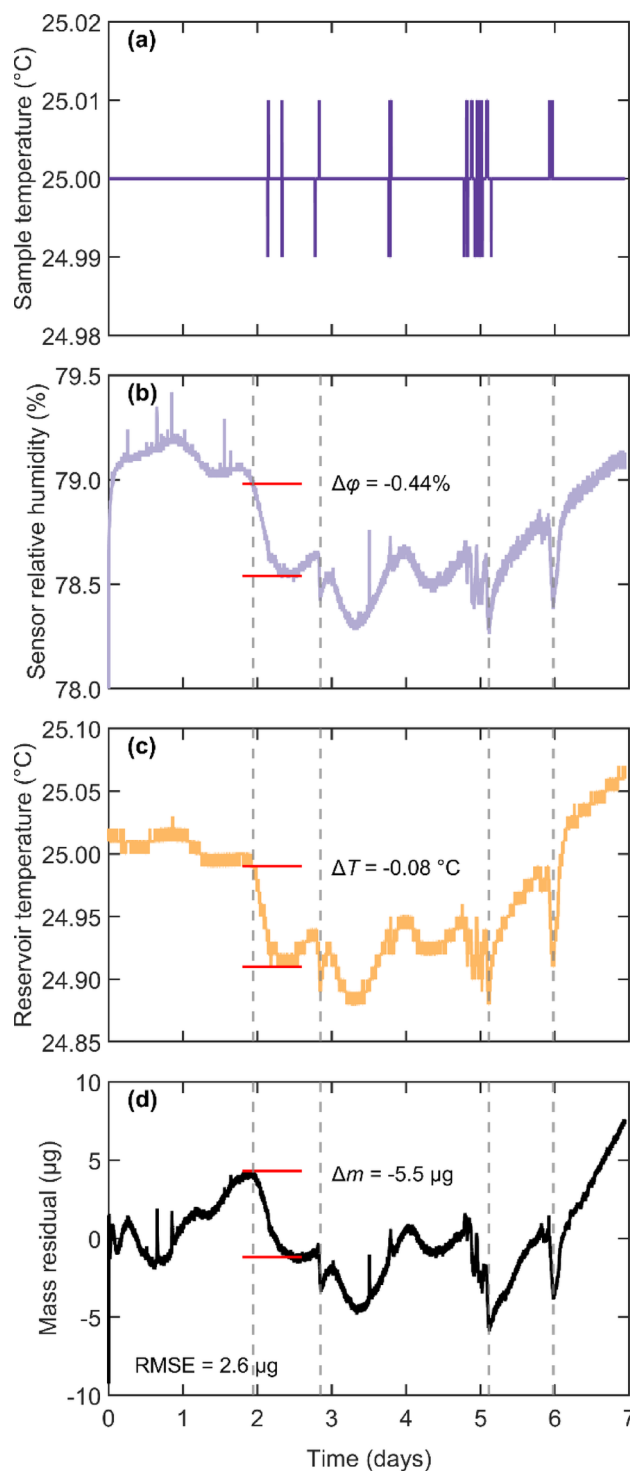


Fig. 11 Time series of sample temperature (a), sensor relative humidity (b), reservoir temperature (c), and mass residuals from the 5EK model fit to sample mass (d) for a step from 70–80% RH, with vertical dashed lines at select time values to show correlation and horizontal bars indicating changes in relevant parameters for a representative fluctuation

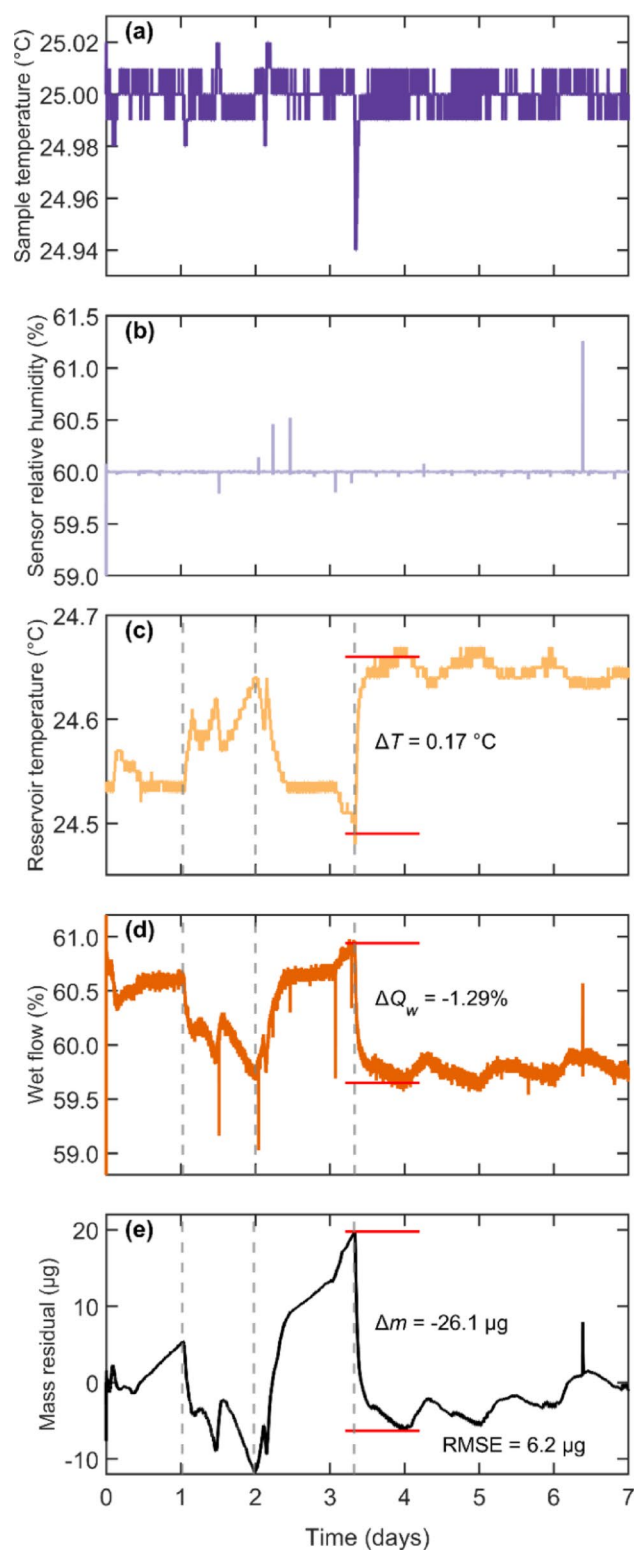


Fig. 12 Time series of sample temperature (a), sensor relative humidity (b), reservoir temperature (c), wet flow (d) and mass residuals from the 5EK model fit to sample mass (e) for a step from 50–60% RH, with vertical dashed lines at select time values to show correlation and horizontal bars indicating changes in relevant parameters for a representative fluctuation

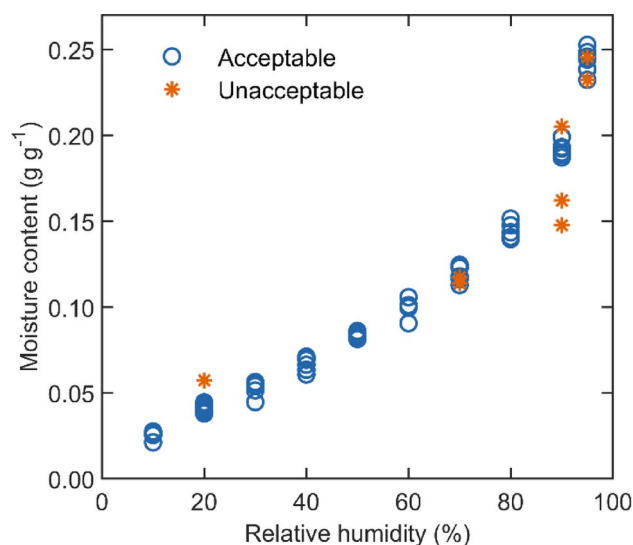


Fig. 13 Absorption isotherm data for western larch at 25 °C based on 80 datasets from 21 laboratories (unacceptable values have moisture content uncertainty greater than 0.0005 g g^{-1})

It may be surprising that RH fluctuates with a constant sample temperature. However, the reason for the changes in RH can be understood by examining the temperature of the water reservoir which provides the water vapor-saturated stream of carrier gas (subfigure c). The reservoir temperature varies from 24.88 °C to 25.07 °C. The relative humidity is strongly correlated with the reservoir temperature. Both curves exhibit a similar shape, and the sharp dips in RH occur at the same times as dips in the reservoir temperature. There is a strong correlation between reservoir temperature fluctuations and mass fluctuations; however, it is unclear if these temperature changes are the sole cause of the mass fluctuations or if the local air pressure or flow rate also fluctuated with reservoir temperature. In either case, the changes in reservoir temperature are most likely caused by fluctuations in laboratory ambient temperature, with the instrument adjusting to maintain the sample temperature reading at its set point.

Finally, Fig. 12 shows data from an instrument that was operating in “closed loop” mode, i.e., using RH sensor feedback to adjust the mass flow controllers to maintain a stable relative humidity. As expected for a closed loop system, both the sample temperature and RH sensor readings were very stable. However, like the data in Fig. 11, the reservoir temperature was not stable, varying between 24.48 °C and 24.67 °C with several notable peaks. In this case, the reservoir temperature changes did not directly influence RH. However, to achieve a stable RH at the sensor, the wet stream mass flow percentage (or simply “wet flow”) varied

with time (subfigure d). Upward trends in the reservoir temperature were matched with downward trends in the wet flow and downward trends in mass. This implies that the actual RH conditions at the sample are not identical to the RH sensor reading. Like the previous open-loop examples, fluctuations in the reservoir temperature in this closed-loop example resulted in appreciable fluctuations in the mass of the sample.

Overall, many of the observed mass fluctuations were a result of temperature and humidity changes that altered the environment to which the samples were exposed. In many cases, the temperature sensor closest to the sample displayed a stable value. However, the water reservoir temperatures fluctuated, which changed the humidity and therefore the sorption conditions. In several cases, improving the temperature stability of the ambient laboratory environment and running the same RH step resulted in smaller mass oscillations and smaller RMSE values (data not shown).

3.4 Proposal for an acceptable level of mass instability

This paper has presented a methodology for characterizing the quality of sorption data by comparing the data to a 5EK model fit. The residuals from these graphs are useful for identifying potential root causes for different types of mass instabilities. However, to this point, we have not discussed how to evaluate whether the quality of the sorption data quantified by the RMSE is acceptable.

We consider two approaches for setting an acceptable RMSE threshold. The first is driven by the data from this interlaboratory study. From the cumulative distribution (Fig. 4a), 80% of the datasets had an $\text{RMSE} \leq 4 \mu\text{g}$, and 90% of the datasets had an $\text{RMSE} \leq 6 \mu\text{g}$. Larger RMSEs were mainly associated with substantial diurnal oscillations that could be remedied by improved control of laboratory ambient temperature, or anomalous behavior such as long-term mass decline during an absorption step. Therefore, a threshold RMSE in the 4–6 μg range is broadly attainable.

The second approach is based on prior literature. Zelinka et al. [25] evaluated published sorption data across five different categories:

- temperature stability,
- relative humidity stability,
- number of replicates,
- moisture content uncertainty,
- mass change at the end of the sorption experiment.

Each of these five categories was rated on a scale of “very good”, “good”, “fair”, and “poor”. The RMSE found from the 5EK fit represents one measure of the uncertainty in the

moisture content. Other sources that contribute to uncertainty in moisture content are not addressed here, such as uncertainty in dry mass or balance calibration. Zelinka et al. [25] set a level of less than 0.0001 g g^{-1} for “very good”, 0.0001 g g^{-1} to 0.0015 g g^{-1} for “good”, 0.0015 g g^{-1} to 0.005 g g^{-1} for “fair”, and greater than 0.005 g g^{-1} uncertainty in the moisture content as “poor”.

An acceptable RMSE threshold in the sorption data could be set based on one of these absolute levels of uncertainty from the paper of Zelinka et al. [25] Of over 27 studies reviewed by the authors that presented data at three or more temperatures on wood, only three studies were found to have achieved a rating of “good” or “very good” across all categories. One of these three studies was given a moisture content uncertainty rating of “very good” and two studies were given a rating of “good”. Therefore, it is reasonable to select a “good” value of moisture content uncertainty for the determination of an acceptable level of RMSE.

The data of Weichert [26] had an absolute moisture content uncertainty of 0.0005 g g^{-1} . For the approximately 20 mg sample dry mass used in this study, a moisture content uncertainty of 0.0005 g g^{-1} translates to 10 μg uncertainty in the absorbed mass. Since approximately 95% of normally-distributed residuals fall within plus or minus twice the RMSE value around zero, the translation from a “good” level of moisture content uncertainty of 0.0005 g g^{-1} results in an RMSE of less than or equal to 5 μg . Selecting the moisture content uncertainty of 0.0005 g g^{-1} as the stability criterion for each dataset, the corresponding RMSE threshold would then depend on the specimen dry mass. For example, a 20 mg dry mass corresponds to an RMSE threshold of 5 μg , whereas a 25 mg dry mass corresponds to an RMSE threshold of 6.25 μg .

While the 0.0005 g g^{-1} moisture content uncertainty and corresponding RMSE threshold for acceptable data was based off previous studies on sorption in wood (including gravimetric techniques other than automated sorption balances), it correlates well with the first approach discussed above. Of the 80 sorption datasets analyzed, 72 (90%) are considered acceptable based on the proposed stability criterion. However, it is also important not to rely too much on RMSE as the data may contain an anomaly not captured by the RMSE. Visually inspecting all mass vs. time curves along with the RMSE can help to ensure high quality data sets.

3.5 Sorption isotherm

The sorption isotherm is plotted in Fig. 13, with each of 80 datasets represented by a point. The moisture content values were calculated using the mass at the end of the long hold (i.e., 7 or 10 days) and the dry mass from the initial

24-h drying sequence. The relative humidity values are the nominal target values. The sorption isotherm is sigmoidal, as expected for water vapor sorption in wood [25].

Individual datasets are differentiated based on whether they passed the stability criterion proposed in Sect. 3.4. Some of the unacceptable datasets in Fig. 13 appear to be outliers (i.e., values at 20% RH and 90% RH), whereas other unacceptable datasets fall within the range of the acceptable datasets (i.e., values at 70% RH and 95% RH). Mass stability is not the only factor affecting data quality (see Sect. 3.4). Excessive mass instability by itself makes the data unreliable, and it might coincide with other sources of measurement error but not necessarily.

The quality of the sorption isotherm for the datasets that passed the mass stability test is evident. The mean coefficient of variation in moisture content (for the acceptable datasets) over all RH levels is 5.0%. Variation in moisture content may stem from RH error or differences in the (trace) amount of water vapor in the carrier gas [24]. Since the RH accuracy of each instrument was not verified in this study, the data in Fig. 13 should not be considered a reference isotherm. However, this interlaboratory study is unique in several respects: the total number of datasets for water vapor sorption in wood (80); the breadth of different automated sorption balances represented across RH steps (Tables 1, 2, and 3); the extended duration of the sorption kinetic data (7–10 days); the high percentage of datasets with moisture content uncertainty of 0.0005 g g^{-1} or less (90%); and the prior verification of instrument mass stability using a non-hygroscopic sample [22]. The findings from this study shed light on ways to identify and reduce mass instabilities and thereby improve data quality.

4 Conclusions and recommendations

Several different types of deviations from expected sorption behavior could be observed in the data.

- Spikes and offsets appeared in some datasets. The most likely cause for these errors was physical disturbances to the microbalance or mass flow controllers. As hold

times are increased, the statistical likelihood of a disturbance increases. Most automated sorption balances are already on vibration isolation tables, and it may be impossible to completely eliminate these errors.

- Some data had oscillations in the mass. These oscillations could often be correlated with changes in the reservoir temperature. Maintaining a constant temperature in the laboratory and in the reservoir is critical for stable mass measurements.
- The temperature readings from the automated sorption balances were often stable, even though the reservoir temperature had fluctuations. Looking at the sample temperature alone may not give a full picture of the temperature stability of the instrument.
- Mass fluctuations were more pronounced at higher relative humidities. Sorption measurements at high RH are complicated by two compounding factors. Firstly, the sorption measurements at high RH take longer than at low RH, which allows more time for a spike in temperature, humidity, or vibration. Secondly, the mass is more sensitive to slight changes in relative humidity at high RH. It can be challenging to collect stable mass data to equilibrium at or above 90% RH.
- The 5EK model provides a way to identify discrepancies from expected sorption behavior. For the sample size of 20 mg used in this study, a $5 \text{ }\mu\text{g}$ RMSE between the 5EK model and the sorption data translates to a moisture content uncertainty of 0.0005 g g^{-1} , which was determined to be a “good” level of moisture content uncertainty in previous work on sorption data. At the same time, while RMSE can be a powerful tool for examining whether the data has a high level of instability, users are encouraged to visually examine the mass vs. time curves to detect other potential issues in the data.

Appendix

See Table 3.

Table 3 Summary of 80 datasets on water vapor sorption in larch from 21 laboratories, with corresponding aluminum pan stability data

Laboratory	RH step	Dry mass (mg)	5EK RMSE (μg)	Sample T SD ($^{\circ}\text{C}$)	RH SD (%)	Reservoir T SD ($^{\circ}\text{C}$)	Al pan SD (μg)
A	20–30%	22.64518	1.11	0.0005	*	No data	0.27
B	10–20%	21.2776	1.17	0.0045	0.006	No data	0.25
	10–20%	22.7902	3.26	0.0063	0.006	No data	
	10–20%	25.8392	0.61	0.0084	0.005	0.0127	
	60–70%	22.6610	5.29	0.0057	0.016	No data	
	60–70%	21.3559	6.89	0.0065	0.051	No data	
	60–70%	23.8143	23.32	0.0077	0.857	No data	
	60–70%	26.7412	2.92	0.0097	0.008	0.0128	
C	30–40%	24.09037	0.79	*	*	No data	1.37
D	70–80%	19.47619	0.94	*	*	No data	0.11
E	50–60%	19.44409	0.73	*	0.223	No data	0.45
F	20–30%	25.3249	0.52	0.0127	0.067	No data	No data
	20–30%	31.9829	1.14	0.0127	0.067	No data	No data
	50–60%	22.1703	1.14	0.0138	0.030	No data	1.22
	60–70%	22.9019	1.11	*	0.117	No data	
	60–70%	23.6046	4.50	0.0035	0.270	No data	
G	0–10%	23.110	0.88	0.1120	0.325	No data	2.20
	0–10%	21.601	0.88	0.0922	0.454	No data	
	40–50%	22.952	1.20	0.1279	0.119	No data	
	40–50%	20.287	4.43	0.0336	0.438	No data	
H	10–20%	24.5772	2.40	0.0922	0.291	*	0.21
	10–20%	23.7148	0.21	0.0137	No data	No data	
	90–95%	24.6900	4.03	0.0163	No data	No data	
I	70–80%	22.5382	2.56	0.0010	0.268	0.0481	1.54
	70–80%	23.9650	1.70	0.0003	0.176	0.0390	
J	10–20%	24.0340	13.51	0.0076	0.012	0.0271	0.18
	80–90%	23.7922	6.04	No data	0.410	No data	
K	60–70%	24.199	2.18	0.0406	0.485	No data	1.21
L	30–40%	23.1534	3.38	0.0057	0.038	No data	0.77
	40–50%	24.4829	1.72	0.0048	0.018	No data	
	80–90%	23.3399	6.83	0.0043	0.014	No data	
M	30–40%	24.5333	2.46	0.0706	0.102	No data	1.16
	30–40%	22.2698	0.81	0.0405	0.038	No data	
	80–90%	24.4777	10.22	0.0747	0.110	No data	
	80–90%	23.1618	2.29	0.0302	0.117	No data	
N	0–10%	22.2113	1.53	0.0089	0.123	0.0111	0.51
	10–20%	22.9604	0.49	0.0087	0.029	0.0086	
	20–30%	23.1166	0.58	0.0088	0.033	0.0159	
	20–30%	22.0288	0.41	0.0087	0.028	0.0135	
	30–40%	25.5336	0.74	0.0092	0.044	0.0168	
	30–40%	23.6491	0.22	0.0086	0.032	0.0059	
	40–50%	25.3694	0.60	0.0092	0.058	0.0171	
	40–50%	24.7323	0.23	0.0087	0.038	0.0083	
	50–60%	23.1057	0.45	0.0087	0.058	0.0097	
	60–70%	23.9074	0.18	0.0087	0.048	0.0054	
	70–80%	22.8875	0.41	0.0089	0.059	0.0125	
	80–90%	23.5103	0.57	0.0088	0.101	0.0093	
	80–90%	23.5230	0.42	0.0087	0.137	0.0129	
	90–95%	25.5547	0.92	0.0087	0.141	0.0101	
	90–95%	24.7038	1.12	0.0087	0.082	0.0079	
	90–95%	24.5899	2.51	0.0088	0.104	0.0122	
	90–95%	24.4384	1.45	0.0087	0.122	0.0164	
	90–95%	28.4989	2.98	0.0200	0.125	0.0037	
	90–95%	22.8284	1.66	0.0087	0.096	0.0132	

Table 3 (continued)

Laboratory	RH step	Dry mass (mg)	5EK RMSE (μg)	Sample T SD ($^{\circ}\text{C}$)	RH SD (%)	Reservoir T SD ($^{\circ}\text{C}$)	Al pan SD (μg)
O	10–20%	22.9890	0.22	0.0226	0.043	No data	1.49
	10–20%	23.5237	0.28	0.0150	0.041	No data	
	10–20%	23.2178	0.26	0.0199	0.026	No data	
	10–20%	22.9890	0.20	0.0136	0.063	No data	
	90–95%	22.9890	26.87	0.0133	0.246	No data	
P	10–20%	23.2411	0.42	0.0005	0.030	No data	0.20–1.11
	10–20%	28.0766	0.47	0.0005	0.030	No data	
	10–20%	23.1660	0.41	0.0005	0.030	No data	
	10–20%	24.5722	0.32	0.0005	0.030	No data	
	10–20%	23.9449	0.27	0.0005	0.030	No data	
	80–90%	23.2411	4.24	0.0006	0.093	No data	
	80–90%	28.0766	5.16	0.0006	0.093	No data	
	80–90%	23.1660	3.74	0.0006	0.093	No data	
	80–90%	24.5722	3.72	0.0006	0.093	No data	
	80–90%	23.9449	3.30	0.0006	0.093	No data	
Q	0–10%	22.4121	0.63	*	0.047	No data	1.85
	70–80%	23.2250	0.91	*	0.048	No data	
	70–80%	22.8696	3.58	0.0008	0.085	No data	
R	50–60%	26.8187	6.21	0.0059	0.028	0.0500	0.45
S	0–10%	21.9102	0.83	0.0029	0.006	0.0303	0.88
	90–95%	22.5509	46.87	0.2087	0.535	0.2711	
T	40–50%	24.1421	5.18	0.0857	0.378	No data	0.62
	40–50%	23.8222	2.68	0.0470	0.107	No data	
U	20–30%	21.8953	0.73	0.0032	0.025	0.0232	0.64
	90–95%	24.0161	1.78	0.0036	0.076	0.0130	
	90–95%	23.7163	5.35	0.0051	0.046	0.0308	

RH: relative humidity; 5EK: five-component exponential kinetics curve fit; RMSE: root mean square error; T: temperature; SD: standard deviation

*Data were reported with insufficient precision to calculate a non-zero standard deviation

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Declarations

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

Ethical approval This study did not involve human or animal partici-

pants.

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