How accurate are automated sorption balances? An analysis of errors in wood moisture content from uncertainties in the conditioning environment

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Automated sorption balances are increasingly used to determine sorption isotherms of hygroscopic materials like wood. The sorption isotherms are constructed based on a number of equilibrium states between a specimen and the surrounding conditioning environment. In recent years, the uncertainty of these equilibrium states has been thoroughly discussed related to the time at constant environmental conditions needed to reach an adequate mass stability. However, uncertainties related to the conditioning environment are rarely discussed. This study describes a framework for analyzing and quantifying the effect of these uncertainties on the error in determined equilibrium moisture content. Various effects of the conditioning environment are analyzed, and the most important effects relate to the relative humidity (RH) in the instruments, both in terms of fluctuations in RH and an offset from the target value. The moisture error for these effects was found within the range 0.001-0.01 g g^{-1}.

Keywords: DVS, moisture, wood, temperature, relative humidity, hysteresis, sorption balance

Introduction

Moisture sorption in wood significantly affects the properties of the material, and has hence been a central topic in wood research for 100+ years [1, 2]. One of the most common and fundamental ways of characterizing water in wood is by the sorption isotherm, which describes the equilibrium moisture content as function of relative

humidity at a constant temperature. In wood science, moisture content is defined as the mass of water in the wood divided by the dry mass of the material.

Sorption isotherms are increasingly being determined based on measurements from automated sorption balances (also called Dynamic Vapor Sorption, DVS or vapor analyzers) [3-9]. These instruments allow easy control of the conditioning environment (RH, temperature) around a small sample mass which can be continuously monitored with in-situ mass recordings as equilibrium is approached. Thus, this setup allows the sample material to be left undisturbed in the instrument after loading. The conditioning environment is maintained by thermostats controlling the temperature and mass flow controllers generating a specific level of RH by mixing saturated and dry streams of carrier gas (typically nitrogen or air). Many instruments contain a built-in RH sensor that can be used to monitor the RH and automatically adjust the flow rates to obtain the desired target level. Alternatively, the RH is generated based on predefined mass flow controller settings, and the RH sensor is just used to monitor the RH level generated.

An advantage of automated sorption balances is that the instruments can be programmed to automatically change the conditioning environment when specified criteria are met [7, 10]. Hereby, sorption data are collected for constructing a sorption isotherm based on a specified number of equilibrium moisture states. The mass stability is often used to indicate a close-to-equilibrium moisture state and to automatically switch the instrument to the next pre-programmed step in RH. Alternatively, the conditioning period at each RH levels is pre-defined [11]. Whatever the criterion used to control the conditioning period, interrupting sorption before equilibrium is attained introduces an uncertainty (error) in the determined mass after conditioning, which will affect the determined equilibrium moisture content at the specific RH [7, 10].

Uncertainty in the dry mass will also affect the equilibrium moisture content, since the mass of water is determined by the difference between the mass after conditioning and the dry mass. Any additional mass from residual moisture in the wood will therefore affect the determined moisture contents of the entire sorption isotherm. Thus, this effect relates both to the decreased perceived mass of water in equilibrium with a given RH and to the increased perceived dry mass of the sample [12, 13]. Appropriate drying protocols are therefore necessary to limit the amount of residual moisture after drying [12]. Most studies in literature do not specify how dry the carrier gas is, but often refer to it as "dry nitrogen gas" or "dry air" without further specification [10, 14, 15]. Few studies provide information on the concentration of water or other impurities in the carrier gas, e.g. the grade of nitrogen [12, 16] or the dew point of compressed air [17]. The RH of carrier gas defines the lowest obtainable RH in the sorption balance and ranges from 0.002% to 0.6% at room temperature for the various qualities of carrier gas reported in literature (Supplementary Material). Drying the sample material without increasing the temperature will result in residual moisture in the assumed "dry mass" of the sample, corresponding to equilibrium at the lowest obtainable RH (Fig. 1a).

Offsets in RH and temperature will result in errors in the moisture content. Since the equilibrium moisture content at a given RH decreases with increasing temperature (Fig. 1b), it means that an offset in temperature compared with the expected value will shift the determined moisture content at all RH levels. Similarly, an offset in RH will shift the moisture content (Fig. 1b), since equilibrium is established at a different RH than expected. Furthermore, the equilibrium state is also path dependent (sorption hysteresis). The equilibrium moisture content at a given RH is thus lower if reached by absorption instead of desorption (Fig. 1a). The largest attainable difference in

equilibrium moisture content is found between the desorption and absorption isotherms, also known as boundary curves. The desorption isotherm describes equilibrium states reached by desorption from water-saturation, either directly or in successive desorption steps. Similarly, the absorption isotherm describes equilibrium states reached by absorption from dry state, either directly or in successive absorption steps. If equilibrium is not reached only by absorption steps from dry state or only by desorption steps from water-saturation, the equilibrium moisture content at a given RH is found on a scanning curve somewhere between the two boundary curves (Fig. 1a). Fredriksson and Thybring [15] discussed this with respect to how to determine sorption hysteresis of wood appropriately, but did not analyze the error in equilibrium moisture content.

Fluctuations in RH and/or temperature also affect the equilibrium moisture state attained. This is not only a result of the fact that the equilibrium moisture content depends on both RH and temperature (Fig. 1b), but also because sorption hysteresis may lead to a gradual drift in the equilibrium moisture state (Fig. 1c). Several studies have shown that cyclic RH variations can lead to gradually increasing or decreasing moisture content [18-23]. Thus, hysteretic drift in moisture content arises since the scanning curves are not as steep as the absorption and desorption isotherms. Therefore, a cyclic conditioning environment will cause the moisture content to drift slightly away from the moisture state attained by absorption or desorption (Fig. 1c). For the commercially available automated sorption balances, the temperature stability is rarely specified by the instrument manufacturers, but the data available ranges from ± 0.02 °C to ± 0.2 °C in temperature stability (Supplementary Material). Specification of the RH stability is even rarer for commercially available instruments with only one instrument having a promised RH stability of ± 0.1% (Supplementary Material). The RH stability depends partly on the temperature stability and partly on the mode of RH generation. The

saturation vapor pressure used to calculate the RH depends directly on the temperature, and therefore temperature fluctuations will lead to variations in the saturation vapor pressure of the air. In a "closed loop mode" of RH generation where the instrument is continuously adjusting the RH level based on RH sensor data, temperature fluctuations will cause the instrument to vary the flow rates of the gas streams in an attempt to maintain the desired RH level. In an "open loop mode" where RH is generated based on preset mass flow controller settings, temperature fluctuations will result in RH variations in the fixed mix of gas streams in accordance with the variation in saturated vapor pressure. In this case, the magnitude of RH variation will depend only on the temperature fluctuations. Zelinka et al. [24] calculated the effect of temperature fluctuations on the RH stability when moisture conditioning is done with saturated salt solutions to generate a specific RH. Using a similar approach, it is shown in the Supplementary Material that at 95% RH, temperature variations of ± 0.1 °C and ± 0.5 °C will result in RH variations of ± 0.6% and ± 3%, respectively, highlighting the importance of keeping the temperature stable.

The error in moisture state related to the conditioning environment of automated sorption balances is seldomly discussed in literature. Therefore, the purpose of this study is to analyze and quantify the errors in equilibrium moisture content related to the conditioning environment of automated sorption balances, i.e. quantify the error introduced by the potential error sources that are visualized in Fig. 1. This was done based on sorption data both from the literature and from sorption balance measurements performed in the present study.
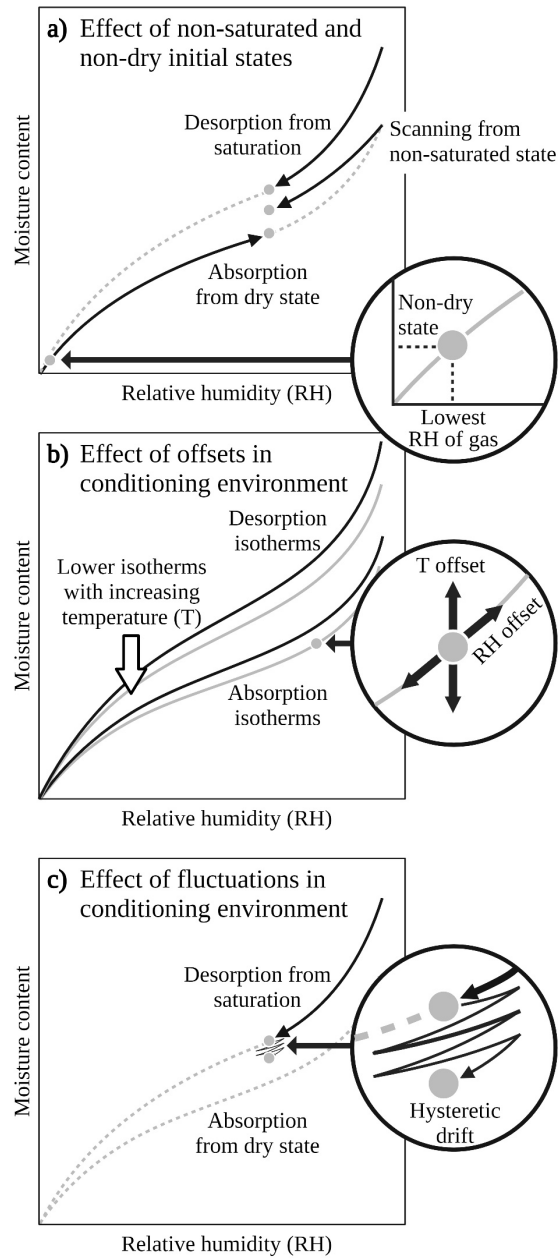
Figure 1: Factors that can generate errors in the equilibrium moisture content related to a) non-saturated and non-dry initial moisture states, b) offsets in conditioning environment (relative humidity, temperature), and c) fluctuating conditioning environment causing hysteretic drift of moisture content. Created with BioRender.com.

Materials and Methods

Sorption data at different levels of temperature

Untreated Norway spruce (*Picea abies* (L.) Karst.) from an experimental forest in Northern Sweden was used [25]. The material was initially water-saturated by vacuum-impregnation in a desiccator. Afterwards, it was cut into samples with a dry mass of approximately 7-10 mg. Before loading the sample in the sample pan of the sorption balance (DVS Advantage, Surface Measurement Systems Ltd., London, UK), excess surface water was removed by wiping with a moist microfiber cloth (Wettex, Freudenberg Home & Cleaning Solutions AB, Malmö, Sweden). The balance was located in an air conditioned laboratory where the most extreme temperature fluctuations are below ± 0.3 °C.

To determine sorption isotherms at two temperatures, the RH was controlled using a fixed conditioning time at each level after the following schedule: 95% (60 h) – 80% (24h) – 65% (12 h) – 50% (12 h) – 35% (12 h) – 0% (24 h) – 35% (12 h) – 50% (12 h) – 65% (12 h) – 80% (24 h) – 95% (24 h). This schedule was followed at the two temperature levels studied: 20 and 40 °C. The sample was finally dried at 60 °C for 8 h using the built-in preheater followed by a 2 h temperature stabilization period. The conditioning times at each RH level were selected based on previous experiments with the aim of obtaining adequate mass stability at the end of each sorption step (Fig. S1 in the Supplementary Material).

Sorption data in fluctuating RH

To investigate the effect of a fluctuating conditioning environment, a sample was loaded and conditioned at 95% RH, 20 °C for 60 h. This was followed by seven cycles in humidity where the RH was ramped up 2% over 6 h, ramped down 4% over 12 h, and ramped up again 2% over 6 h. Furthermore, a non-saturated sample was loaded and

dried at 0% RH, 20 °C for 24 h before conditioning at 95% RH for 24 h. Thereafter, the same protocol for varying RH was followed in which a full cycle of ± 2% RH is completed over 24 h simulating a daily variation in temperature around the sample of approximately ± 0.46 °C. This could for instance occur in laboratories with inadequate temperature control for regulating differences in heat production during the day and night. The protocol was used instead of changing the temperature in the instrument for two reasons: 1) it is difficult to implement temperature variations in an instrument designed for isothermal conditions, and 2) the mass flow controller settings depend on the target instrument temperature. Therefore, it would not have been possible to mimic RH variations from temperature fluctuations in the external lab climate by programmed temperature cycles, and instead cycles in RH were used. Finally, the sample was conditioned at 95% RH for 24 hours before it was dried using the same protocol as for the other sorption measurements. The entire RH history is shown in Fig. S6 (Supplementary Material).

Sorption data from previous studies

In order to validate the magnitude of the effects of offsets in RH and temperature, the experimental data of Weichert [26, 27] and Djolani [28, 29] were used. While these data were not collected on automated sorption balances, they were meticulously recorded using careful protocols to ensure moisture equilibrium in a very stable conditioning environment [24]. The Weichert study includes sorption isotherm data at four temperatures (25, 50, 75, and 100 °C) for spruce and beech, whereas the data of the Djolani study concerns sugar maple (*Acer saccharum* Marsh) at four temperatures (5, 21, 35, and 50 °C). The sorption properties of these materials can of course deviate from the properties of the Norway spruce materials investigated in the present study and other data from our labs included here. However, the Weichert and Djolani data are included

as examples of high-quality data that can validate the overall trends and magnitudes of the results derived from our data.

Sorption measurements on Norway spruce have been conducted in a number of published studies from our labs. These data were also compiled and analyzed in the present study. This includes the data on Norway spruce (*Picea abies* (L.) Karst.) from Southern Sweden reported by Fredriksson and Thygesen [3] on isolated early- and latewood tissue in desorption from water-saturation and absorption from dry state at 20 °C. It also includes the data on the same material reported by Fredriksson and Thybring [15] on isolated earlywood tissue in desorption from water-saturation, absorption from dry state, and scanning desorption after conditioning to either 80% or 95% RH at 20 °C. As for the new data collected for this paper, the data from the two previously published studies were also collected using a DVS Advantage (Surface Measurement Systems Ltd., London, UK).

Data analysis

The experimental sorption isotherm data from the present study as well as the literature data were fitted with the ABC isotherm [30], Eq. 1. This equation is mathematically equivalent with the Hailwood-Horrobin, Dent, and Guggenheim-Anderson-de Boer sorption isotherm models, which have been found to fit well to sorption isotherm data for wood. However, these models have been found to yield invalid physical parameters values for water in wood [31]. Therefore, we used the ABC isotherm that does not imply to provide any physically meaningful parameters regarding water in wood [30].

$$u = \frac{\varphi}{A\varphi^2 + B\varphi + C} \tag{1}$$

where u (g g^{-1}) is moisture content, φ (-) is RH, and A, B, and C are fitting parameters. All fits of the ABC isotherm to experimental absorption and desorption isotherms

analyzed in this study are shown in Fig. S5 and the fitting parameters in Table S1 of the Supplementary Material. From the fits of the ABC isotherm (Eq. 1) to the experimental data, several effects of the conditioning environment on the error in equilibrium moisture content can be found.

The moisture error when using conditioning at non-saturation was found as the difference in ABC fits of the desorption isotherm and scanning curves initiated after absorption to either 80% or 95% RH.

The moisture error induced by residual moisture in the perceived dry mass of the sample was calculated as

$$\Delta u(\varphi) = \frac{1}{1+u_{\text{res}}}(u(\varphi) - u_{\text{res}}) - u(\varphi) \tag{2}$$

where u_{res} (g g^{-1}) is the residual moisture content in the determined dry mass. The residual moisture content was determined as the predicted moisture content from the ABC isotherm at the lowest obtainable RH in the sorption balance.

The moisture error from an offset in RH can be determined from the derivative of moisture content with respect to RH

$$\Delta u(\varphi) = \frac{du}{d\varphi}\Delta\varphi \tag{3}$$

where Δu (g g^{-1}) is the moisture error, $\Delta\varphi$ (-) is the offset in RH, and the derivative is calculated from the ABC isotherm (Eq. 1) as

$$\frac{du}{d\varphi} = \frac{-A\varphi^2 + C}{(A\varphi^2 + B\varphi + C)^2} \tag{4}$$

The moisture error of an offset in temperature can be determined from the derivative of moisture content with respect to temperature

$$\Delta u(\varphi) = \frac{du}{dT}\bigg|_{\varphi} \Delta T \tag{5}$$

where Δu (g g^{-1}) is the moisture error at fixed φ, and ΔT (K) is the offset in temperature. The derivative is calculated from linear regression of the ABC fits to data at more than one level of temperature.

$$u_i(\varphi) = \beta_0(\varphi) + T_i\beta_1(\varphi) + \varepsilon_i \ \ with \ \ i = 1, \dots, n \tag{6}$$

where u_i (g g^{-1}) is the dependent variable, T_i (K) is the regressor, ε_i (g g^{-1}) is random error, and β_0 (g g^{-1}) and β_1 (g g^{-1} K^{-1}) are the intercept and regression coefficient of the linear regression at the specific φ, respectively. The latter term is the derivative of moisture content with respect to temperature in Eq. 5.

The moisture error from conditioning in a fluctuating environment was calculated based on the difference in moisture content between the initial and final conditioning periods at 95% RH, which were intermitted by seven RH cycles

$$\Delta u = u_{\text{final}} - u_{\text{initial}} \tag{7}$$

where u_{initial} (g g^{-1}) and u_{final} (g g^{-1}) are the moisture contents found after conditioning at 95% RH before (initial) and after (final) seven cycles in humidity.

Results and Discussion

To calculate the moisture error from offsets in RH and temperature, the derivatives of the moisture content with respect to these two parameters is first determined (Fig. 2). It can be seen that the derivative with respect to RH is highest at both low and high levels of RH, i.e. where the sorption isotherms are steepest. Therefore, an offset in RH from

the target value will result in the highest moisture error in these two ranges. The derivative with respect to temperature becomes increasingly negative with increasing RH, except for in absorption at high RH. Thus, a higher temperature will result in a lower moisture content than at the target temperature. The high-quality literature data from Weichert [26, 27] and Djolani [28, 29] supports the trends of the two derivatives as seen in Fig. 2. In particular, the derivative with respect to RH follows closely literature data, whereas the magnitude of the derivative with respect to temperature is similar.
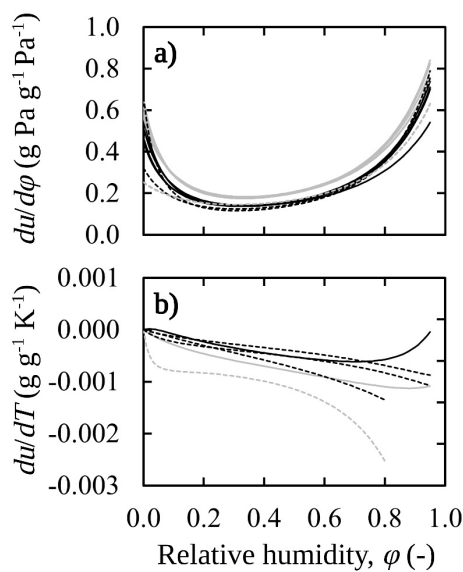

Figure 2: Derivatives of sorption isotherms in terms of a) relative humidity (g Pa g^{-1} Pa^{-1}) and b) temperature (g g^{-1} K^{-1}) in absorption (black) and desorption (grey). For a) the solid lines represent data from this study, Fredriksson and Thygesen [3], and Fredriksson and Thybring [15], all at 20 °C, whereas the dashed lines represent data of Weichert [26, 27] at 25 °C and Djolani [28, 29] at 21 °C. For b) the solid lines represent data of this study and dashed lines represent data of Weichert [26, 27] and Djolani [28, 29]. Created with BioRender.com.

The moisture error by initiating desorption measurements from a non-saturated state, i.e. the deviation from the desorption isotherm, is shown in Fig. 3a. The error is largest

at the initial RH level, where the moisture content corresponds with a point (equilibrium state) on the absorption isotherm. As the RH is decreased, the deviation from the desorption isotherm decreases.

Fig. 3b shows the moisture error stemming from residual moisture after drying in a non-dry carrier gas of 0.6% RH without elevated temperature. The error increases with RH and is larger in desorption than absorption, which is because the moisture error is larger with a higher moisture content. Also, the error is of course larger the more residual moisture is present when the dry mass is determined. Thus, Fig. S11 in the Supplementary Material shows the moisture error from residual moisture after drying in carrier gas with 0.01% RH reflecting nitrogen gas of grade 5 commonly used for automated sorption balances.

The moisture error from slow fluctuations in RH between 93% and 97% over several cycles is shown in Fig. 3c. The initial state in this plot is reached by absorption. It is clear that the first cycle has the largest effect and subsequent cycles leads to fluctuations around a new quasi-equilibrium state. Fig. S9 in the Supplementary Material depicts the same plot as Fig. 3c after initial desorption to equilibrium (Fig. S8). In this case, the fluctuations lead to variations in moisture content around a gradually decreasing quasi-equilibrium state. As described previously, the RH fluctuations simulate the effect of daily temperature variations of $\pm$ 0.46 °C using fixed mass flow controller settings for the automated sorption balance. This level of temperature variation will only affect the equilibrium moisture content by approximately $\pm$ 0.001 g g^{-1} as seen from Fig. 2b. However, it will cause the saturation vapor pressure to vary and with it the RH with $\pm$ 2% if the mass flow controllers run on fixed settings. Such fluctuations in RH will lead to a change in the equilibrium moisture content of around $\pm$ 0.01 g g^{-1} as seen from Fig. 2a, i.e. one order of magnitude larger effect on the

equilibrium moisture state approached by the sample than the temperature variations themselves.
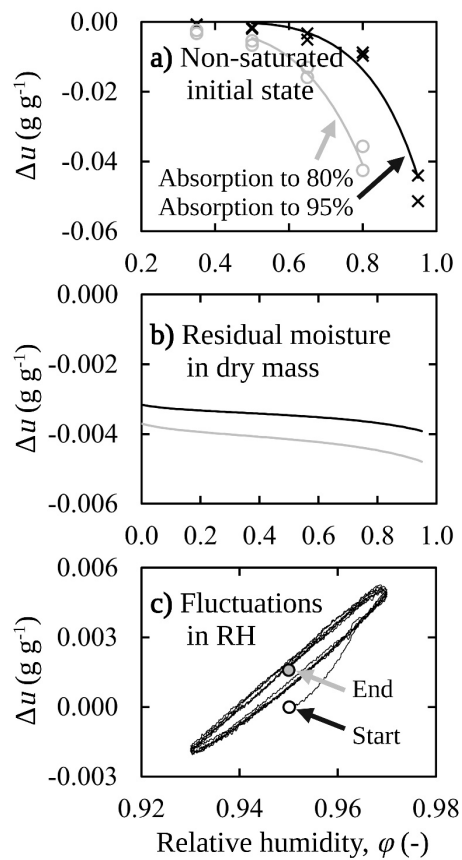

Figure 3: a) Deviation from equilibrium moisture content on desorption isotherm, Δu (g g⁻¹), by conditioning in absorption to either 80% (grey) or 95% RH (black) instead of from water-saturation (circles and crosses mark experimental data points), b) deviation from absorption (black) and desorption (grey) isotherms because of residual moisture in determined dry mass after drying at 0.6% RH, and c) deviation from equilibrium moisture content on absorption isotherm, Δu (g g⁻¹), by RH fluctuations of ±2% after conditioning in stable climate at 95% RH in absorption from dry state. Arrows mark the start and end of the fluctuating RH period. Created with BioRender.com.

In Fig. 4, the magnitudes of the various moisture errors at 95% RH are compiled. Data at lower RH are shown in Fig. S12-S14 and Tables S7-S8 in the Supplementary

Material. Fig. 4 shows the absolute error in moisture content on a logarithmic scale to illustrate the differences in magnitude. Moreover, the errors were partitioned into four categories (major, moderate, minor, negligible) based on their magnitude.
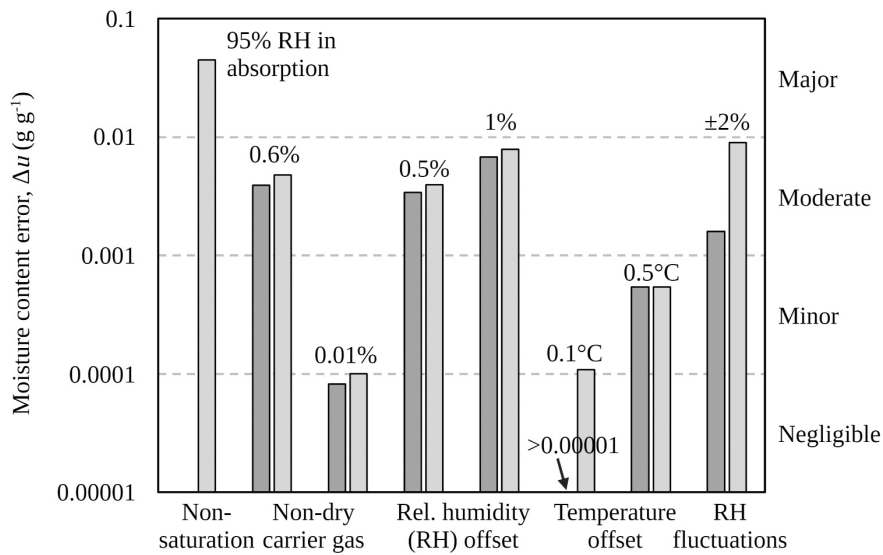

Figure 4: Error in equilibrium moisture content at 95% relative humidity (RH) as a result of desorption from non-saturation (absorption to 95% RH), drying in non-dry carrier gas, offsets in RH and temperature, and RH fluctuations. Dark grey = absorption, light grey = desorption. Please note the logarithmic scale on the y-axis for distinguishing the order of magnitude of the moisture content error. Created with BioRender.com.

Desorption from non-saturation caused a major error > 0.01 g g^{-1} at 95% RH in the equilibrium state on the supposed desorption isotherm. Several moisture errors were in the moderate range (0.001-0.01 g g^{-1}) which all relate to the humidity of the conditioning gas; how stable the RH is, how accurately it is generated (offset), and if inadequately dry carrier gas was used without raising the temperature. The smallest moisture errors were found in the minor to negligible range < 0.001 g g^{-1} and includes potential temperature offset as well as drying at very low, non-zero RH. While the

major and moderate moisture errors decreased with decreasing RH, except for residual moisture corresponding to an RH of 0.6%, the magnitude and hence severity of the various errors persist even at 80% and 65% RH in most cases (Fig. S12-S14).

Whereas all these errors from uncertainties in the conditioning environment have been investigated separately, it should be remembered that several effects may operate at the same time during sorption measurements, e.g. an offset in RH and fluctuations in RH. In this case, their combined effect may either increase or decrease the error in moisture content, depending on the sign of the effect. In such cases, this study provides a framework for how moisture error from uncertainties in the conditioning environment can be analyzed and quantified. Importantly, this study illustrates the importance of tight control and monitoring of the RH for performing high-quality measurements. Thus, the RH should be monitored by calibrated RH sensors to avoid or be aware of an offset RH. Moreover, a stable temperature should be maintained around the automated sorption balance to avoid RH fluctuations. Reducing these two effects would greatly limit the error in moisture content and improve data quality in sorption measurement with automated sorption balances.

Conclusion

This study developed a framework for analysis and quantification of errors in equilibrium moisture content determined with automated sorption balances related to the conditioning environment. Various effects of the conditioning environment on the moisture content error were analyzed and quantified. The most important parameter was the RH, both in terms of fluctuations in RH and an offset from the target value. The moisture content error was within the range 0.001-0.01 g g^{-1}. Moreover, residual moisture from drying in inadequately dry carrier gas yielded an error of similar

magnitude. An offset in temperature only resulted in minor moisture content error, but may yield larger errors indirectly by causing fluctuations in RH.

Disclosure statement

The authors report there are no competing interests to declare.

Data availability

https://doi.org/10.17894/ucph.9f90c50c-2610-4c88-a627-12bc9d600cc5

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