Reply to anonymous referee #3

In blue we copied the comments of the reviewer, in black our replies.

I am reviewing the manuscript of César Dionisio Jiménez–Rodríguez and colleagues, entitled “comparison of water vapor sampling techniques for stable isotope analysis” submitted as technical note and currently under discussion for HESS. The authors investigated in the laboratory the short to mid-term (1-16 days) reliability of different plastic sampling bags for isotopic analysis of water vapor, as an affordable alternative to the online analysis with laser-based spectrometers. They highlight that only one commercial product (made out of MPE) provided reliable results, independently from storage time. The authors don’t say clearly what could have been the cause of the change with time in isotopic composition of the sampled water vapor in the two other types of bags, although they show the influence of the laboratory air during measurement.

If the bags were not tight, then the influence of the laboratory air would not be restricted to the time of measurement, but would spread from sampling to measurement times (i.e., over the whole storage duration). To me, this points to a tightness problem during measurement. The laser spectrometer seemed to have sampled from a mix of laboratory air and bag air and measured some average-weighed water vapor isotopic composition. This should be thoroughly addressed by the authors.

Reply: there are three possibilities of exchange with the laboratory air:

1. Through the bag material
2. Through the valve during analysis
3. Through both, valve and material.

We think based on the results in figure 2 that #1 and #3 are minor in comparison to #2. Because MPE and PVF have the same valve system, where MPE is still performing well after 9 days whereas PVF follows the laboratory air. This is likely due to the higher Water Vapor Transmission Rate of the bag material.

So you are right that PVF is not only disturbed during analysis, but also during storage. However, it’s surprising that PVF is seemly so much affected by the laboratory air during analysis. Since we did not measured the water vapor mixing ratio on all days we cannot concluded whether the air in the PVF bag is affected during storage and/or only during analysis. This should be investigated in further research. To clarify this point, we added the following paragraphs:

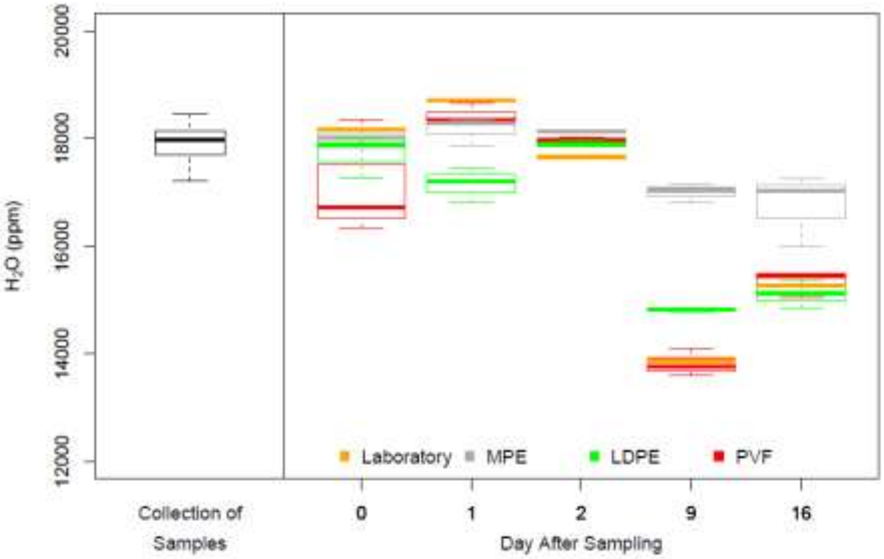
Following the reviewer’s suggestion to elaborate more on this issue we proposed to add the following:

Page 7, Line 2:

“Water vapor mixing ratio during the collection of samples had a mean value of 17 930 ± 369 ppm. This concentration changes with time during the different days of measurement (Figure 2). Between the measuring days 2 and 9 the water vapor mixing ratio drops from 18000 ppm to less than 14000 ppm, while towards the measuring day 19 it increased with 1000 ppm more. This trend is tightly followed by the PVF sampling bags, followed by the LDPE with a larger difference and the MPE with small variations respect to the water vapor mixing ratio of the samples collection. This data shows that all the sampling bags exchange water vapor from and towards the atmosphere with a different degree of magnitude.”

Page 8, Line 1:

"The tendency of drift towards the signature of the laboratory air could be linked to other factors such as welding quality between bag material and the valve (for MPE and PVF bags), fitting issues between the tubing connecting the sample bags to the MIU unit (all sample bags) or the inlet connection for the LDPE bags. In the case of MPE and PVF bags, the manufacturer states that the bags should not be filled more than 90% of their capacity. This practice could lead to the development of fissures between the air valve and the bag material that in the case of PVF bags due to their brittle properties respect to MPE of LDPE bags. An increment on the air pressure within the MPE bags can lead to the detachment of the air valve from the layers in which it is welded. LDPE bags are susceptible to leaking as a consequence of the inlet built with in-house materials that the presence of different joints can induce the filtering of the laboratory air. "



In new manuscript: Figure 2. Boxplots describing the water vapor mixing ration in ppm during the collection of samples and the posterior measurement of samples.

I also urge the authors to remove the part on cryogenic extraction from the benchmarking, as it was not working well enough during the experiment and is off-topic.
Reply: the reviewer is right that the cryogenic bath during the collection of air with the sample bags was not carried out properly and gave some errors. However, the second time we did run the cryogenic bath and compared against the WVIA run simultaneously the data show the capacity of this sampling method. The second text provides information about another sampling procedure widely used for sampling air water vapor that requires and removing it will reduce the discussion about collection methods.

Aiming to skip this, we proposed to remove the cryogenic samples collected during the first test due to their lower performance and small number of samples. Also, we propose to keep the second test explained in page 6, lines 25-28 and the results from figure 2.B (submitted manuscript) as the proper evaluation of the cryogenic bath method against the WVIA. In this way, the analysis is still enriching with the evaluation of the cryogenic bath with a larger number of samples.

I found the text to be difficult to read at times and generally not well structured. I am afraid it did not undergo sufficient internal review before it was submitted to HESS. I provide the authors with many comments and corrections below.
Reply: the authors thank the reviewer for the suggestions to improve the manuscript.

Technical comments:

Throughout the manuscript: - stick to past tense for things that happened in the past. - provide unit/dimension for all parameters and variables present in the different equations

Reply: we surveyed and checked the manuscript to improve homogenized the verb tenses accordingly with the suggestion.

Abstract P1L1. The first two sentences read awkward. I propose something like:

"The isotopic characterization of water vapor samples can help describing evaporation processes. However the collection of water vapor, which historically involves phase change, may be associated with fractionation in case of incomplete sampling. "

Reply: we improved the sentences accordingly.

"The isotope signature of a parcel of air was continuously monitored with a cavity. . ."

Reply: we improved accordingly.

P1L4-5. You did not sample the same air parcel with different methods simultaneously, rather you sampled from the same air parcel simultaneously with different methods.

Reply: this sentence was improved as follows:

"… (benchmark). Sampling from the same air parcel simultaneously with 3 types of sampling bags made of …"

1 Introduction

P1L11. The isotopes or isotopologues are the tracers, not their isotopic signatures.

Reply: the δ was removed to refer only to the isotopologues. Also, we check the manuscript to fix this issue.

P1L12. "Water stable isotopes..". Also move the deltas to the previous sentence. You are not talking about their signatures here.

Reply: done.

P1L14. Consider citing a more recent review on plant water use isotopic applications,
e.g., Rothfuss and Javaux (2017).

Reply: thanks for the recommendation. This reference (Rothfuss and Javaux, 2017) was checked and included on the manuscript together with Schwendenmann *et al* (2015) and Wang *et al* (2017).

P1L15. Remove "only" from this sentence: sampling precipitation conservatively is not that straightforward actually.

Reply: done.

P1L17. Some sample cannot describe evaporation processes, please rephrase.

Reply: this sentence was modified as follows:

"… samples. Water vapor samples such as transpired water or atmospheric water are good descriptors of the evaporation process , …"

P1L18-19. "It is formed by water vapor originated from evapotranspiration (plant transpiration and soil evaporation) and of intercepted water..." What about free water surfaces (streams, lakes, etc.)?
Reply: we include the free water surfaces to this definition and the respective reference as follows:

"… It is formed by water vapor originated from open water evaporation, plant transpiration, soil evaporation and the evaporation of intercepted water on wet surfaces (Abtew and Melesse, 2013; Savenije, 2004). …"

P1L20-22. This should be shortened: go straight to keywords evapotranspiration partitioning.
Reply: it was changed as follows:

"… Evaporation partitioning has been carried out …"

P1L22. "has been. . ."?. It is still the case and will always be, right?
Reply: verb tense was changed for present: "is"

P2L4-15. This level of detail in this section is not needed. Also it is confusing: at this point, it is not clear anymore what you are referring to "water vapor"; is it the atmospheric water vapor or the evaporated water vapor? Since you are addressing the potential isotopic effects of the sampling bags during/following collection/storage of atmospheric water vapor, this first "technique" is off-topic.
Reply: aiming to prevent this type of confusion, we simplified this paragraph as follows:

"Currently, the estimation of the isotope signatures of water vapor can be performed with three different methods. Firstly, the Craig-Gordon model (CG–model) (Craig and Gordon, 1965) determine the water vapor signature of evaporation originated from open waters (Horita et al., 2008) and has also been applied in transpiration and soil evaporation studies (Dubbert et al.,2013; Ferrio et al., 2009; Williams et al., 2004). The high sensitivity of ^{18}O to temperature makes some assumptions of this model unreliable for the application in soil evaporation or plant transpiration processes (Dubbert et al., 2013). The cryogenic bath is the second …"

P2L17. "collects"
Reply: done

P2L17-23. These two sentences are hard to read, should be merged and shortened, e.g. the "second method consists in. . ." (it is the operator who collects); "conveyed at. . ."; "requires sublimating...". What you need to say is that we have two different classes of measuring techniques: 1- online, direct and continuous and 2- offline. The second technique is then subdivided in two sub classes: a-direct (with sampling bags) and indirect (via sublimation).
Reply: see next reply.

P2L20-23. Again, these sentences are hard to read: a sample does not collect itself; there is no such thing as "heavier isotope signatures". An isotopic signature does not "occur". You should write something like: "The collection of water vapor in cold-traps for isotopic analysis is only a reliable technique when recovery rate tends towards 1; an incomplete recovery of water vapor fractionates water stable isotopes following the Rayleigh distillation model."
Reply: according with the suggestions from the reviewer, we improved these lines as follows:

“The second method consists in a cryogenic bath that allow the collection of atmospheric water vapor within a canister immerse in a cooling agent (e.g,liquid nitrogen) (IAEA, 2016; Kool et al., 2014), freezing the water vapor conveyed at a constant air flow into the collection canister (He and Smith, 1999; Sheppard, 1958; Wen et al., 2016). This sampling method for isotopic analysis is only a reliable technique when recovery rate tends towards 1 (Griffis, 2013). An incomplete recovery of water vapor fractionates the water stable isotopes following the Rayleigh distillation model (Kendall and Caldwell, 1998). The consequences of incomplete sample recoveries are similar to the effects as studied by Orlowski et al. (2018) for soil water extraction.”

P2L25-26. We measure the isotopic composition of water vapor, not the “water stable isotopes”.
Reply: this sentence was changed as follows:

“The most recent method involves direct measurements of the isotopic composition of water vapor using mass spectrometers or laser-based spectroscopy.”

Change “cavity output spectroscopy” to the general term “laser-based spectroscopy”.
Reply: we followed this recommendation and the manuscript was updated accordingly with the suggestion.

P2L31. What is a “storage unit”?
Reply: here we refer to the sampling bags.

P3L1-3. I think there is a conceptual misunderstanding here: the equilibration bags for soil water vapor are water-tight (due to the hydrophobic nature of the plastic) but let the water vapor diffuse through their walls. The observed problems have not to do with “[liquid] water diffusing through the container wall”
Reply: this sentence was fixed as follows:

“… Wassenaar et al., 2008). They underline the risk of water vapor diffusion through the wall container when using equilibrium bags of different materials to determine the soil water isotope signature. If their findings for equilibrium bags used in soil water measurements hold for air water vapor samples as well, is still unknown. The aim …”

P3L3-4. Why are you referring to “units” here? In the abstract you say “sampling bag”. You should stick to one lexical field/terminology.
Reply: following the recommendation from the reviewers, we changed the term “storage units” for “sampling bags” in the whole document.

2 Methodology
2.1 Instrumentation and Measurements
P3L12. Why is mentioning the voltage of the water pump of interest to the reader?
How do they relate to the “controlled water vapor concentration”? Please explain.
Reply: the voltage pump is the only parameter that the user can adjust on the WVSS, because the built-in software controls the mixing water ratio by the power provided to the pump (Page 3, lines 12-13). Aiming to provide more details about this item, we followed the recommendation of Reviewer 1. Then we add Appendix C

Also you mean water vapor “mixing ratio”, and not “concentration”.

Reply: yes, it should be “water vapor mixing ratio”. Aiming to homogenize the manuscript terminology, we checked and change this term accordingly.

P3L13. What is a “sample set here”?
Reply: we refer to every round that the MIU unit measures from the different inlets.

P3L17-18. “allows the connection to different sampling devices. . .”. It is “inlet” or “port”? Stick to wording for ease of reading.
Reply: it is “inlet”. We checked and fixed this through the whole document.

P3L18-21. Why do you need the altered water vapor source?
Reply: The idea behind adding an altered air source during the sampling is to be able to differentiate between samples during the laboratory procedure and post-processing of the data. This helped to carry out the data analysis because after the air passed by the drying element, the ^{2}H and ^{18}O signatures will change drastically. To clarify this, we proposed to improve the lines 18 and 19 of page 3 as follows:

“… post-processing of the data. The data obtained from this inlet was not used during the analysis as it was used only as a distinction mark between samples. The altered air …”

P3L23-24. "The first 3 minutes were discarded from the analysis to account for memory of the instrument to the previous sample". Users know that memory effect is due to the turnover rate of the volume ahead and inside the cell of the laser. No need to say this.
Reply: we modified the sentence and attached to the previous paragraph as follows:
“… with sampling intervals of 5 s. The first 3 minutes were discarded by the memory effect, calculating the average and standard deviation of each measurement based on the last 2 minutes of measurements per sample.”

P3L24-25. Please revise grammar
Reply: see previous reply.

Figure 1. I don’t think the drawing is necessary. Text is enough to me. Also both liquid and vapor modes are missing the part of the analyzer itself. They should be renamed e.g. vapor and liquid "peripheral devices".
Reply: the authors want to keep the diagram because it will help to people new in the topic to understand how the system is integrated. Also, we changed the diagram according to the reviewer’s suggestion.

P4L1-5 & Eq. (1). This is also not needed. Also terminology is not correct: "hydrogen and oxygen isotopic ratios are expressed relatively to those of the SMOW" not "Isotope signatures (2H and 18O) [...] were expressed in respect to the Vienna Standard Mean Ocean Water (VSMOW)". A signature is the relative deviation of some ratio with respect to another, meaning it remains a ratio, only on another scale. So why is a "signature" termed as "relative concentration" when "ratio" isn’t? They are all ratio of concentrations.
Reply: aiming to simplify this section to the reader, it was changed as follows:

“Stable water isotope signatures of air vapor and liquid samples were expressed in δ values (‰), representing the relative deviation from Vienna Standard Mean Ocean Water (VSMOW) (Craig, 1961).”

2.2 Water Vapor Correction

You address here the problem of the nonlinear response of the laser spectrometer to changes of water vapor mixing ratio, which translates into a dependency of the raw isotopic readings to water vapor mixing ratio in the measuring range. This section's text is overall not well structured. Choose your wording carefully and stick with it. It starts with the title: it should read something like "Water vapor isotopic calibration". The isotopic terminology is sometimes not adequate (e.g. "The raw signatures of δ2H and δ18O"). I advise you to be as precise as possible and write "water vapor mixing ratio" and not "concentration of water molecules" (does not apply here: the LGR measures water vapor mixing ratio, not concentration) or "ppm concentration" (does not mean anything).

Reply: this section was upgraded as follows:

"2.2 Water Vapor Isotopic Calibraton

Measurements of water vapor isotope signatures depend on the water vapor mixing ratio (ppm) and the specific drift of the laser spectrometer of the WIA unit, which makes it essential to correct each individual measurement (Aemisegger et al., 2012; Rambo et al., 2011; Kurita et al., 2012; Steen-Larsen et al., 2013, 2014). The drift of the used laser spectrometer was negligible, because the measurement period was not longer than six hours every day. In addition, the thermal control within the laser chamber provides stable measurements with a negligible drift as it is stated by the manufacturer (LGR, 2019). The calibration was performed with a standard water ($\delta^{18}O_{standard}$: -14.4, $\delta^{2}H_{standard}$:-104.9) injected into the WIA at different pumping rates depending on the pump voltage (see Section 2.1). The injection is controlled by a built–in software package that managed the WVISS pump and the DAS. This system allows the use of only one standard water to calibrate the isotope signatures carried out with the WVIA. The calibration procedure was performed every time the MIU start a new round of measurements (see Appendix D). The measured isotope signatures ($\delta^{2}H_{raw}$ and $\delta^{18}O_{raw}$) were calibrated using the correction factors (ϕ_O and ϕ_H) determined based on the dependency of the isotope signatures of standard water ($\delta^{2}H_{standard}$ and $\delta^{18}O_{standard}$) to their water vapor mixing ratio (w) in ppm. The polynomial coefficients a, b and c in equations 2 and 3 were determined for every set of measurements per experiment (Rambo et al., 2011; Kurita et al., 2012; Steen-Larsen et al., 2013, 2014). Each data point used in equations 2 and 3 corresponds to the last minute of measurements for each voltage, obtaining an average based on 12 individual measurements for both stable isotopes and water vapor concentrations. The calibrated values of each stable isotope ($\delta^{18}O$ and $\delta^{2}H$) were determined with equations 4 and 5 (Rambo et al., 2011; Steen-Larsen et al., 2013)."

P4L8-10. Revise sentence structure. It is hard to read.

Reply: see previous reply about Section 2.2.

P4L10-12. Revise sentence as well: how did you measure evaporated standard water at different water vapor mixing ratio? Is it by changing the water pump flow rate (hence the different voltage values mentioned earlier?) or by adapting the dry air flow rate?

Reply: see previous reply about Section 2.2.

I understand you did a one-point calibration rather than a two-point (span) calibration?

Reply: yes, it is one point calibration due to the settings of the device. However, we followed the recommendations given by Rambo et al. (2011), Kurita et al. (2012) and Steen-Larsen et al. (2013) for this procedure (Page 5, Lines 4-8).

Have you investigated the laser dependency to water vapor mixing ratio at another value of isotopic composition?

Reply: yes, we had checked with one additional isotopic composition ($\delta^{18}O$: -5.6 and $\delta^{2}H$: -40.8). The device follows the polynomial tendency as described by Rambo et al. (2011) but the coefficients a, b and c have to be constantly determined. Figure ## shows the results of that test.

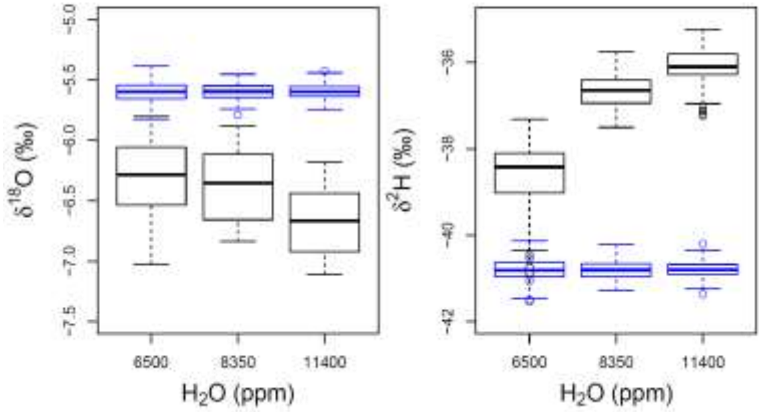


Figure ##. Isotope signatures of the raw measurements (black boxplots) and calibrated values (blue boxplots) of the known water ($\delta^{18}O$: -5.6 and $\delta^{2}H$: -40.8).

Eqs (2-5): they yield to δ18O=δ18Ostandard and δ2H=δ2Hstandard, so something does not add up here. Please revise. A "good" instrument is characterized by a slow time drift of a, b and c; is this the case?

Reply:

The LGR analyser used during this experiment did not experience a significant drift during the measurements due to the little time running on a daily basis (less than six hours every day). Additionally, the model of water isotope analyser (IWA) keeps a "negligible drift" as it is stated by the manufacturer (LGR, 2019).

To clarify this, we proposed to add the following sentence on Page 4, line 10:

"… Steen-Larsen et al., 2013, 2014). **The drift of the used laser spectrometer was negligible, because the measurement period was not longer than 6 hours every day. In addition, the thermal control within the laser chamber provides stable measurements with a negligible drift as it is stated by the manufacturer (LGR, 2019).** The correction of water vapor measurements was …"

Also, the authors agreed with the suggestion of one of the reviewers of adding an additional plot to the manuscript showing the relationship of the correction procedure applied. This plot shows the negligible drift of the laser in terms of the calibration factors ϕ_O and ϕ_H. The plot will be added as an appendix to the manuscript as follows:

Appendix C: Plots of the Water Vapor Correction Procedure

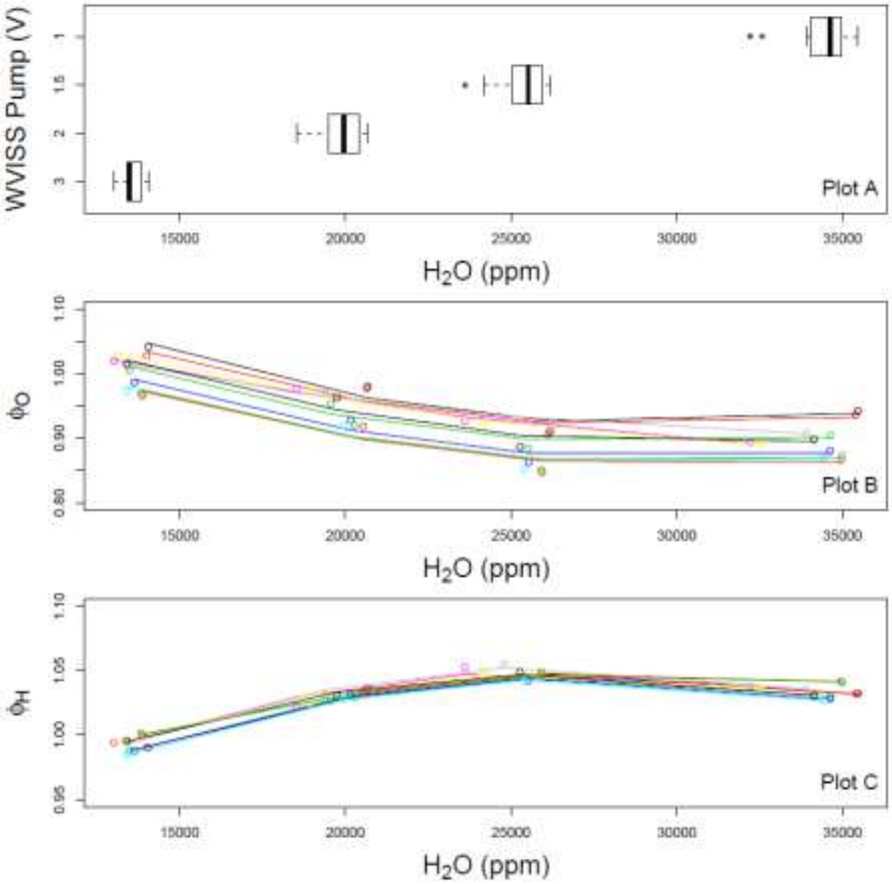
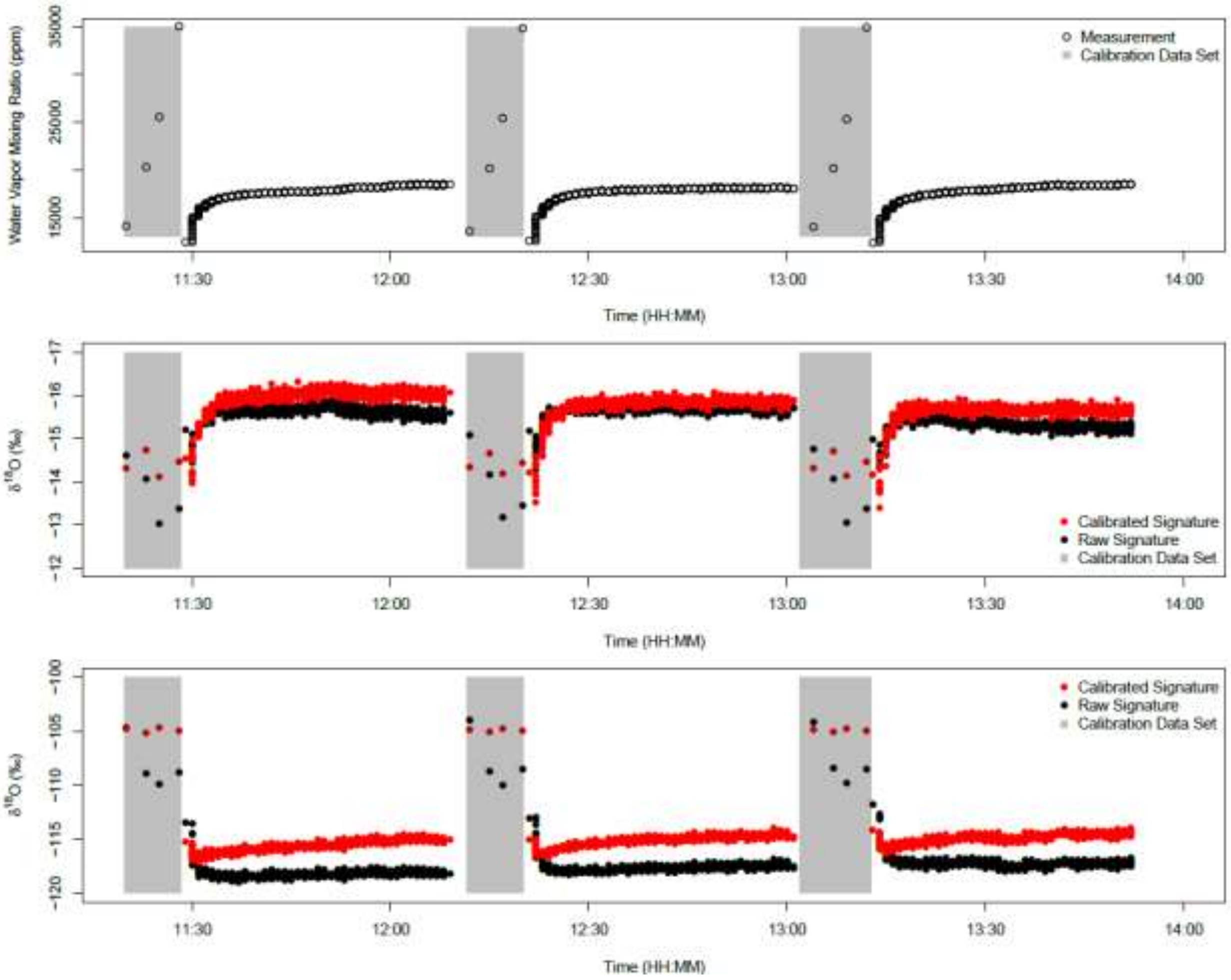


Figure C1. Water vapor correction plots showing the variation of the water vapor mixing ratio (ppm) and the calibration factors ϕ_O and ϕ_H. Plot A shows the variation of water vapor missing ratio in ppm for each voltage used on the WVISS pump during the correction procedure. Plots B and C show the polynomial relationships between the water vapor missing ratio and the calibration factors ϕ_O and ϕ_H, respectively.

Also, equations 2 to 5 are uses to correct the raw isotope signatures given by the WVIA according with their dependency on the water vapor mixing ratios (Rambo et al, 2011). The following figure shows the calibration process during the water vapor sampling procedure of three hours. The grey areas correspond to the controlled water vapor mixing ratio of the known standard ($\delta^{18}O$: -14.4 and $\delta^{2}H$: -104.9). The polynomial relationship of the data set in between grey areas (equations 2 and 3) were used to correct the raw signatures of the sampled air based on the dependency of the water vapor mixing ratio. This calibration was done estimating the φ coefficient with the equations 2 and 3 for the raw measurements of the samples, applying later on equations 4 and 5 to retrieve the corrected values. As it is shown in the figure, this procedure does not change the raw measurements into the standard water. The coefficients a, b and c do not refer to slow time drift, they refer to the linear relationship showed by the mixing ratio and the raw isotope signature measured by the device. We proposed to add the following image as an Appendix to the manuscript to better understand the calibration process.



2.3 Experimental Design

P5L15-16. ". . .the isotope signature of the stored air water vapor samples remains. . ." or the "the water vapor isotope signature of the stored air samples remains. . ."

Reply: we changed it for:

"… the water vapor isotope signature of the stored air samples remains …"

However, this line was move up to improve the objectives section in the introduction following the recommendation of one reviewer.

Points1.-3. Avoid starting each sentence with "these"

Reply: each point was improved as follows:

"1. MPE: bags of 1L made of methalized polyethylene and manufactured with …
2. PVF: bags of 1L made of …
3. LDPE: bags of 1L made of …"

P5L18. "According to the supplier,. . ."

Reply: correction done.

P6L3. Until now, it wasn't clear that the experiment took place in the laboratory. It should be stated early on (abstract + listing of objectives in the introduction).
Reply: aiming to inform the reader earlier about the location of the sampling, we add the following:

Page 1, Line 3-4: "…The isotope signature of a parcel of air was continuously monitored in the laboratory with a laser-based spectroscopy …"

Also, the objectives in the introduction were improved according with the suggestions of all the reviewers as follows:

"The aim of this work is to evaluate different sampling procedures to collect atmospheric water vapor and analyze the stable water isotopes. This experiment tested whether the stored mass of water vapor sampled in the laboratory remained unchanged as well as whether the isotope signature of the stored water vapor remains consistent in time. We included three sampling bags to determine their suitability for sampling, storing and analyzing water vapor isotopes. The results were compared against a set of cryogenic samples and direct measurements performed with laser-based spectroscopy."

Did you check for complete recovery for the fast (3 L min-1) option by, for, instance measuring the water vapor mixing ratio with the laser spectrometer at the outlet of the trap, or installing a second trap in series to observe if water vapor was collected in it?
Reply: no, we did not evaluate the complete recovery of the sampled air.

P6L8. Was it a simple test tube or something more elaborated (i.e. with an inner collecting wall)? This needs further detail as this greatly impacts the ability of the trap to capture moisture.
Reply: following the suggestion of one of the reviewers, we add a diagram of the cryogenic bath used during this experiment as an appendix.

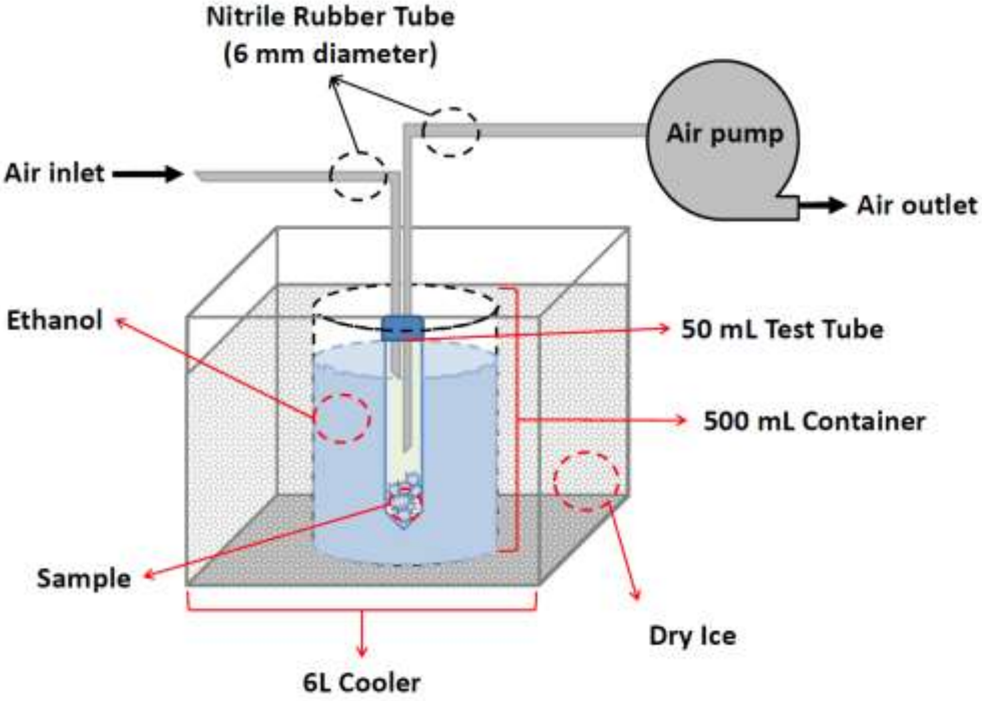


Figure C1. Cryogenic bath diagram for the collection of liquid samples of atmospheric water vapor.

2.4 Analysis

This section needs some streamlining: you are unnecessarily repeating yourself (P6L18: “The cross comparison was performed with the Z-score analysis” vs P6L28: The comparison was performed with the Zscore analysis”).

Reply: we removed the repeated sentence in Page 6, line 28.

P6L17. Revise sentence, e.g., “you compare the isotopic signature of the samples to that of the benchmark”, not “to the benchmark”.

Reply: we improved the sentence as follows:

“The consistency analysis of the isotopic signatures was performed comparing the isotope signatures of the samples against the Benchmark. …”

P6L19. “(Orlowski et al., 2016; Wassenaar et al., 2012), where S is the isotope signature (δ2H or δ18O) of the bags air water vapor and cryogenic water samples, B is the benchmark water vapor isotope signature (WVIA), and μ is the target variability.”

Reply: we add the correction to the sentence. Thanks!

3 Results and Discussion

P7L2-3. Revise sentence (“the isotope signature of the benchmark . . . had an isotope signature”)

Reply: the sentence was improved as follows:

“The stable isotope signatures of the benchmark during the three hours of the experiment were -15.61±0.14‰ and -115.12±0.47‰ for δ^{18}O and δ^{2}H, respectively. The benchmark represents the center point of both graphs in Figure …”

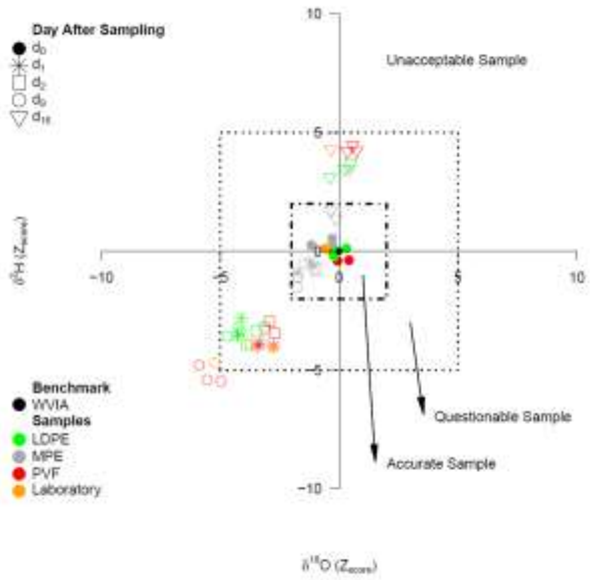
P7L5-6. Please revise (edit grammar).

Reply: the sentence was edited as follows:

“The isotope signature of the laboratory air water vapor was not constant on the different days when the measurements were performed.”

Also: use letter “d” for day, or “t” for time, but not “T” (stands for temperature usually).

Reply: we modified Figure 2 using d instead of T when we refer to days as follows:



P6L25-26. Here, you are not testing the ability of the association trap+cryogenic bath to fully collect the air moisture, rather you are testing its reproducibility. See my previous comment on P6L3.

Reply: you are right. Here we are not testing the collection capacity, however the results from the second test shows the capacity of using the cryogenic bath as benchmark when the technique is carried out properly.

P7L15-24 and Fig. 2B. I don't understand the difference between "cryogenic bath" and "cryogenic test sample". There is no distinction made in the text here and before. . .but see my general comment: I don't see how cryogenic water extraction is relevant in your study.

Reply: there is no difference on the collection method between both samples. We choose different words to differentiate between the two samplings. Again, the authors strengthen the need to keep the second test where the cryogenic bath is tested carefully against the WVIA.

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