

Water content determination in green coffee – Method comparison to study specificity and accuracy

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Abstract

Green coffee behaves very differently at high and low water content with a number of unwanted consequences like microbial growth, mycotoxin formation, altered sensorial quality of end product, instable production conditions and unclear trade issues. Generally, a water content ranging between 8.0% and 12.5% is considered to be adequate to avoid the mentioned issues. ISO has therefore issued a number of standards for reference, routine and rapid methods. Nevertheless, on-going discussions on how effective the methods are capable to principally determine the water content lead to modifications of the official approach. This work was therefore focused on clarifying the specificity and accuracy of several available methods. We could demonstrate that only ISO 1446 exclusively measures water but leaves some residual water content difficult to extract from the dried coffee matrix. For all drying oven based methods we observe degradation of the product contributing to the overall weight loss. We used near-infrared spectroscopy and color measurement to establish the degree of degradation and the completeness of the drying process. Repeatability was found excellent for all methods despite degradation and incomplete drying which should negatively affect accuracy. © 2005 Elsevier Ltd. All rights reserved.

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1. Introduction

Water content is certainly the most critical quality parameter of green coffee, as it governs fermentation and mould growth during storage and transport, which could lead either to the development of off-flavors at the cup level or to the formation of mycotoxins. It is also important for economical reasons as coffee is paid by weight and buyers are obviously more interested to buy solid coffee materials than water. In this connection, ISO had issued three official standards. ISO 1446 is the basic P₂O₅ reference method (slow drying at 48 °C) for the determination of the water content in green coffee. ISO 1447 (rapid drying at 130 °C) is the routine oven method. ISO 1446 and 1447 are supposed to give identi-

cal results. The third procedure, ISO 6673 (medium drying at 105 °C, is the practical method for the determination of the loss of mass, in which coffee is dried in an oven with forced ventilation. It is clearly stated in ISO 6673 that the method gives results, which are about 1.0% lower than ISO 1446.

ISO recently decided to withdraw standard ISO 1447, which was the method used up to now to calibrate all water content analyzers based on indirect methodologies such as capacity, conductivity or near-infrared spectroscopy. Consequently, the withdrawal imposes a recalibration of all analyzers. The purpose of this study was to precisely assess the performance of ISO 6673 and to further test the robustness of the method.

The water content of green coffee beans is most often determined using rapid instrumentation. Capacitance-based (Clarke, 1985) or microwave-based (Wiseman, 1999) analyzers are the most widespread and cheapest

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whereas near-infrared spectroscopy (Guyot, Davrieux, Manez, & Vincent, 1993) the probably most accurate indirect method for water content determination in green coffee beans. Proper calibration is the most important step and ISO is currently working on guidelines for the calibration of this type of instrument (ISO AWI 24115, 2003). The reference method used for calibration is one of the key elements determining the final performance and often the main source of variation. Lacking accuracy of the reference method will therefore translate into an increased error of prediction of the indirect technique. Understanding the behavior of the reference method will therefore help to adapt existing calibrations to the new reference method.

2. Materials and methods

The present work is based on the comparison of the following methods and practices. Near-infrared spectroscopy and color measurement are used to support conclusions on the specificity and accuracy of the water content determination methods. We did not include the Karl Fischer method into this study despite its very high selectivity for water. The method requires the grinding of the beans and the removal of the water by dioxane distillation prior to the final titration. This method is described in the official German Standard DIN 10766.

3. Drying method ISO 1446

ISO 1446 describes the basic reference method for the determination of the water content in green coffee. It is based on the desiccation of about 3–4 g of ground green coffee under phosphorus pentoxide (P_2O_5) at 48 ± 2.0 °C up to weight constancy. The grinding step needs to be performed with care to avoid losses due to increasing temperature in the grinder.

The glass tube was evacuated under water pump vacuum in order to facilitate the water release. According to the standard the required drying time is between 150 and 200 h. The reported results were taken after 317 h (about 13 days) checking weight constancy before. The P_2O_5 was changed a few times in order to keep the water uptake high. A constant temperature was guaranteed by placing the end of the tube where the sample is placed into an oil bath. Due to the large heat capacity of the oil the temperature is very stable and most often better than ± 1.0 °C. The analysis was performed at least in duplicate and the results averaged.

The method is obviously insensitive to the laboratory temperature and relative humidity. However, the drying kinetics was found very sensitive to the particle size of the ground coffee.

4. Drying method ISO 1447

ISO 1446 describes the routine method for the determination of the water content in green coffee. Approximately 5 g of complete green coffee beans are dried for $6 \text{ h} \pm 15 \text{ min}$ at 130.0 ± 2.0 °C. The sample is then removed from the oven and weighed after it has cooled down to ambient conditions. In a second drying step the sample is put under identical drying condition for $4 \text{ h} \pm 15 \text{ min}$. After an identical weighing procedure the water content is calculated as sum of the weight loss after the first drying step and half the weight loss of the second drying step.

All drying ovens were placed in an air-conditioned laboratory with controlled temperature (23.0 ± 2.0 °C) and relative humidity ($40 \pm 5\%$). The number of samples in the oven was limited to six to provide comparable amounts of released volatiles during all measurement series. We used model T6060 ovens (Heraeus, Germany). The temperature in the ovens was measured using a temperature controller (Hamster, 2000; Herni AG, Switzerland), which is checked twice a year against a certified thermometer. Analysis were performed at least in duplicate and results averaged.

5. Drying method ISO 6673

ISO 6673 measures the mass loss of complete green coffee beans. ISO 6673 is using an electrically heated drying oven with forced air ventilation controlled at 105.0 ± 1.0 °C. Approximately 10 g of whole green coffee beans are dried for 16 ± 0.5 h. We used model Salvis Thermocenter (Renggli AG, Switzerland), which was checked identically to the standard ovens. The analysis was performed in duplicate and the results averaged.

Additionally, we carried out the same analysis but using an oven without forced air ventilation. We used the T6060 oven from Heraeus above described. The number of samples in the oven was kept at maximum six.

It is known that, for food powders, the residual water content at the end of the drying is sensitive to the laboratory temperature and relative humidity (reproducibility issue). In order to investigate the influence of the laboratory conditions on the final results some samples were additionally measured in a climatic chamber at 32 ± 1 °C and $63 \pm 2\%$ RH.

6. Near-infrared spectroscopy

All the samples were scanned using a scanning near-infrared spectroscopy (NIRS) spectrometer model Infralyzer 500 from Bran and Luebbe, Germany. The measurements were done using small ring cups. Samples

were scanned over the whole range of wavelength (400–2500 nm) with a resolution of 2 nm. However, an averaged spectrum was used for further work. For the measurement all samples were rapidly ground.

For the normalization procedure all NIR spectra were exported as JCAMP format from the instrument and converted into absorption values corresponding to each peak using a home made program. Further, these absorption values were normalized to minimize texture effects. The first order normalization was performed using an EXCEL sheet at the wavelengths of 1680 and 2232 nm. For further evaluation of the residual water content we principally focus on the water band, which can be found at around 1940 nm.

7. Color measurement

The color measurement was performed with a HunterLab ColorFlex (CLFX) benchtop spectrophotometer (Brimelow & Joshi, 2000). The granular milled samples were presented in an opti-glass cylinder placed directly onto the measurement aperture. The sample was sieved in order to achieve the volume fraction between 1 and 2.5 mm. This was done to keep the influence of the particle size distribution at a minimum. Results output was expressed according to the CIE $L^*a^*b^*$ system where L^* is always positive ranging between 0 (black) and 100 (white). Colors with $a^* > 0$ possess the attribute of redness, those with $a^* < 0$ greenness, those with $b^* > 0$ yellowness and those with $b^* < 0$ blueness.

7.1. Samples

Arabica and Robusta green coffees from different geographic origin were selected in order to cover a wide range of water content. Humidification of samples to obtain higher water content levels was not necessary. After reception the green coffee beans were placed into cans and allowed to thermo-dynamically equilibrate for minimum one month. We selected four samples of green coffee and applied all described methods. For a given can, all the measurements were then performed by the different methods on the same day.

8. Results and discussion

Table 1 summarizes the results. ISO 1446 shows the lowest results of all tested methods. The new ISO 6673 gives results, which are approximately 0.3% w/w higher than ISO 1446. ISO 1447 gives results, which are about 1.3% w/w higher than ISO 1446 and about 1% w/w than ISO 6673. We almost did not find any differences when we exposed the drying ovens operated according ISO 6673 to a more tropical climate.

Drying processes are mainly driven by temperature, particle diameter and partial pressure of water on the surface of the particle. For ISO 1446 based on P_2O_5 desiccation, the principle driving force for the drying process is the almost complete absence of water in the gas phase combined with the reduced pressure, which accelerates the water transfer from the bean surface to the desiccant agent. The restricting process is the diffusion of the water molecules to the particle interface. Decreasing the particle size, which modifies the distance the water has to diffuse, speeds up the drying. The second effect of the particle size reduction is the increase of the surface area, which facilitates the mass transfer into the gas phase. The other main variable for the P_2O_5 method is temperature. At 48 °C, diffusion processes are quite slow but degradation reactions are considered to be absent for the majority of the known food products. This is supported by our color measurements. The significant residual water content that we observed by the NIR spectra indicates that the drying is not complete under the chosen conditions. On the other hand, the color measurement only shows little change principally due to the difference absorbance of the product when water has left the product. The efficiency of the P_2O_5 method could be either improved by increasing the temperature onto a level (about 85 °C) where degradation is still absent or minimal compared to the overall water content, or by reducing the average particle size. This could lead to a significant reduction of the drying time.

The major driving force for the air oven drying based methods used in this study is the temperature. Particle influences also this type of methods strongly but due to operational reasons the grinding step is left out. Increasing the temperature speeds up the diffusion of the water molecules to the surface but also triggers degradation. We observed significantly more degradation at 130 °C supported by the color and near-infrared measurements. Therefore, the ISO 1447 procedure interrupts the drying and accounts only 50% of the weight loss during the second drying step to the water content. It should be pointed out that this degradation not only occurs in the second but also in the first drying step. We even estimate more loss by degradation in the first step, as the presence of water is required for most of the occurring reactions. Near-infrared spectra showed an almost complete drying but the comparison of the results suggests a significant contribution of the degradation to the overall weight loss. At the end, water loss and degradation explains the 1.3% w/w difference observed between ISO 1447 and ISO 1446.

We have chosen one Robusta sample to assess the effects of the grinding and the drying processes in more detail. Enough beans were ground and rapidly sieved for obtaining about 100 g of particles ranged between

1 and 5 mm. Table 2 and Figs. 1 and 2 summarize the results. For the sample dried according ISO 1446 we measured 12.24% w/w as extracted water and we found 0.53% w/w residual water content (according to a NIR calibration). Assuming no weight loss due to degradation we can calculate “true” water content of 12.77% w/w. For ISO 6673, extracted water was 12.53% w/w. Based on the assumption that the near-infrared absorption at 1940 nm is in linear correlation to the residual water content (ranged between 0% and 1.5% w/w) we could determine the residual water content as 0.63% w/w. The sum of both extracted and residual water by-pass the true water content determined as 12.77 by 0.39% w/w. We attribute this weight loss to degradation reactions during the drying process. Accordingly we also performed the calculation for ISO 1447. After the first drying step we determined a residual water content of 0.12% w/w and extracted water content of 13.16% w/w. We calculated therefore a weight loss by degradation of 0.51% w/w. The second drying step leads to an overall weight loss of 13.30% w/w and ISO 1447 would report 13.23% w/w as final result. The additional weight loss of 0.14% w/w is at the lower end of what we have observed during our study and all differences given for this sample are not statistically representative for the whole population. Nevertheless, they allow understanding the occurring phenomena during the drying process.

Of this additional weight loss 0.06% w/w were attributed to water loss based on the near-infrared spectra. The additional weight loss for degradation in the second drying step would be in this example 0.08% w/w. Compared to the overall weight loss related to degradation of 0.59% w/w this amount stays quite small, meaning that the most significant weight loss related to degradation occurs in the first drying step. This also supports the theory that water is required for some of the degradation reactions. Globally, we can conclude that ISO 1447 almost completely removes the water but also includes significant degradation of the sample. Here, the degradation at 130 °C in the ground particles was less extensive than in the whole beans due to the more efficient drying kinetics. Overall the weight loss related to degradation is larger for ISO 1447 than for ISO 6673. This would be an argument supporting the change of method versus ISO 6673. Degradation is clearly observed in Fig. 2 by changes in color of the samples after drying. With increased degradation samples were measured to be more red (a^* increased) and yellow (b^* increased).

In some additional experiments we grinded the green coffee and fractionated the ground material according to their particle size. By controlled drying conditions at 48 °C and 1% relative humidity, we observed that the drying process was more rapid when the particle size

Table 1
Determination of moisture (% w/w) in green coffee by different ISO standards

Sample	Arabica 512 966	Arabica 513 048	Robusta 513 032	Robusta 512 927	Average
<i>ISO 6673 105 °C</i>					
1 With ventilation HR 40% 16 h	12.18	12.40	12.41	13.07	
2 With ventilation HR 63% 16 h	12.11	12.36	12.32	13.02	
Difference 1–2	0.07	0.04	0.09	0.05	0.06
<i>ISO 1447 130 °C</i>					
3 RH 40% 6 h	12.91	13.30	13.26	13.99	
4 RH 40% 6 h + 4 h	13.40	13.74	13.57	14.35	
Difference 4–3	0.49	0.44	0.31	0.36	0.40
5 Final result ISO 1447	13.15	13.52	13.42	14.16	
<i>ISO 1446 48 °C</i>					
6 P205 317 h	11.74	11.93	12.14	12.92	
Difference 1–5	–0.97	–1.12	–1.01	–1.09	–1.05
Difference 1–6	0.44	0.48	0.27	0.15	0.33
Difference 5–6	1.41	1.59	1.28	1.24	1.38

Table 2
Repartition of residual water, extracted water and weight loss by degradation for a typical sample for tested drying techniques

Robusta 513032			NIR absorption (abs)	Residual water	Extracted water	Overall weight	Weight loss by
Method	Temperature (°C)	Time (h)	at 1940 nm	(% w/w)	(% w/w)	loss (% w/w)	degradation (% w/w)
ISO 1446	48	316	0.489	0.53	12.24	12.24	0.00
ISO 6673	105	16	0.495	0.63	12.14	12.53	0.39
ISO 1447	130	6	0.460	0.12	12.65	13.16	0.51
		6 + 4	0.453	0.06	12.71	13.30	0.59
Result						13.23	

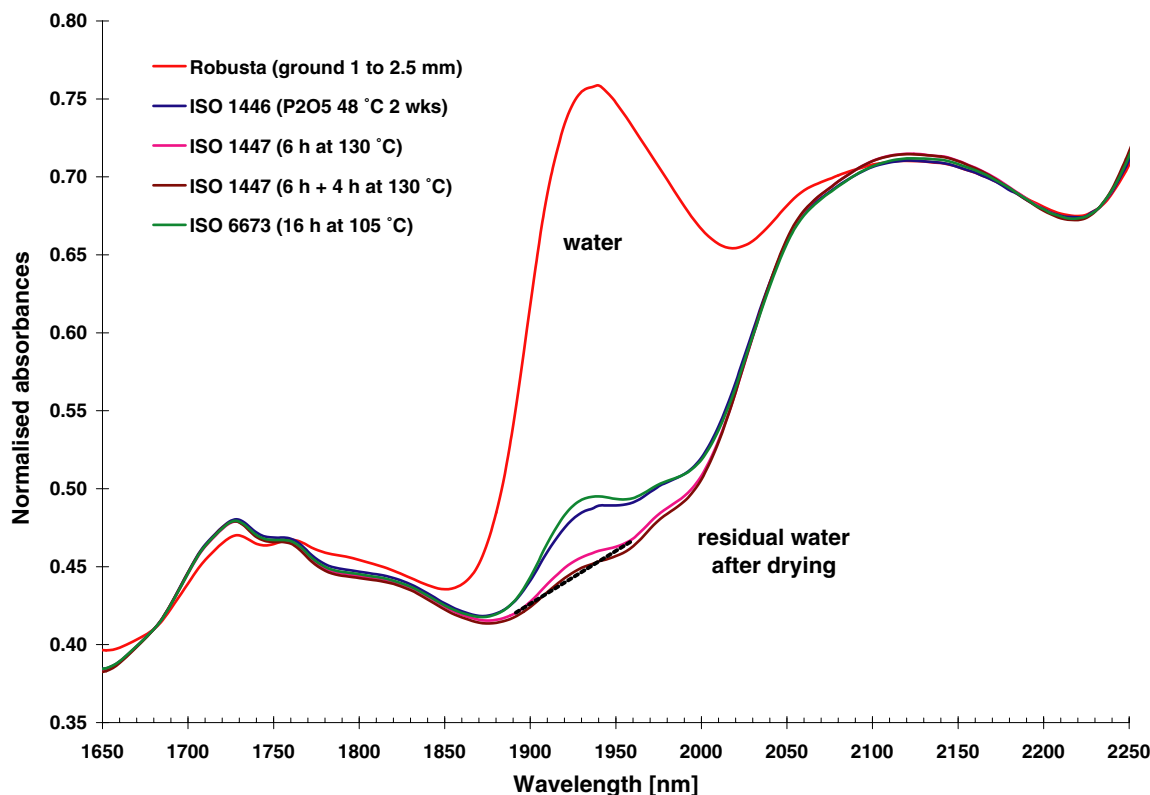


Fig. 1. Green coffee wet and dried under different conditions.

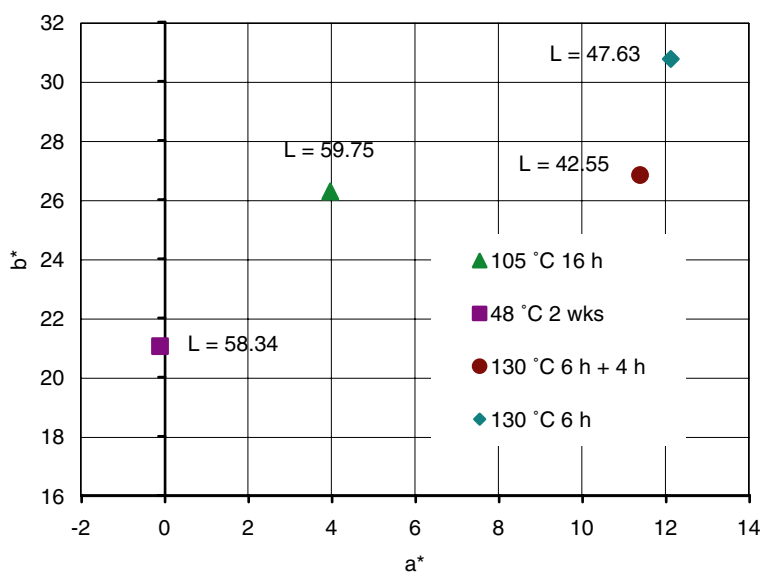


Fig. 2. Color differences after drying.

was smaller. This indicates a strong influence of the diffusion of the water molecules through the matrix on the drying time, especially for the dry region below 3% residual water content. Based on color measurements we additionally observed less degradation reactions when the particle size was smaller. The explanation is that for most of the temperature related degradation

reactions occurring in the green coffee water molecules are required. As the water content level of smaller particles decreases faster they are also less affected by degradation.

The second part of the study was focused on the expected change from using ISO 1447 to ISO 6673. Different samples covering 7–13% water content were

Table 3
Comparison between ISO 1447 and ISO 6673

Method	ISO 6673 with ventilation			ISO 6673 no ventilation			ISO 1447			Difference ISO 6673 with – no	Difference ISO 6673 – ISO 1447
	Sample	1	2	Average	1	2	Average	1	2		
1	9.35	9.44	9.40	9.83	9.74	9.79	n.d.	n.d.	n.d.	–0.39	n.d.
2	9.24	9.17	9.21	9.00	9.09	9.05	n.d.	n.d.	n.d.	0.16	n.d.
3	8.98	8.95	8.97	8.31	8.40	8.36	n.d.	n.d.	n.d.	0.61	n.d.
4	7.96	7.99	7.98	8.04	8.10	8.07	n.d.	n.d.	n.d.	–0.10	n.d.
5	7.64	7.64	7.64	7.85	7.78	7.82	8.71	8.75	8.73	–0.18	–1.09
6	7.54	7.50	7.52	7.52	7.55	7.54	8.54	8.63	8.58	–0.02	–1.06
7	8.01	8.01	8.01	8.08	8.17	8.13	9.24	9.19	9.21	–0.12	–1.20
8	7.81	7.81	7.81	7.95	7.93	7.94	8.89	8.87	8.88	–0.13	–1.07
9	8.60	8.56	8.58	8.69	8.60	8.65	9.49	9.56	9.52	–0.07	–0.94
10	7.90	7.87	7.88	7.92	7.88	7.90	8.78	8.86	8.82	–0.02	–0.94
11	7.78	7.83	7.81	7.86	7.85	7.86	8.75	8.73	8.74	–0.05	–0.93
12	7.78	7.69	7.74	7.87	7.81	7.84	8.83	8.77	8.80	–0.10	–1.06
13	7.72	7.73	7.72	7.77	7.79	7.78	8.77	8.84	8.80	–0.06	–1.08
14	7.38	7.41	7.39	7.51	7.43	7.47	8.51	8.48	8.49	–0.08	–1.10
15	12.15	12.25	12.20	12.28	12.23	12.26	13.15	13.25	13.20	–0.06	–1.00
16	12.53	12.63	12.58	12.58	12.68	12.63	13.64	13.57	13.60	–0.05	–1.02
17	12.86	12.78	12.82	12.82	12.78	12.80	13.88	13.95	13.92	0.02	–1.10
18	12.11	12.02	12.06	12.18	12.13	12.16	13.15	13.23	13.19	–0.10	–1.13
19	10.98	11.07	11.02	11.02	11.02	11.02	12.15	12.14	12.14	0.00	–1.12
20	11.08	10.98	11.03	11.00	11.07	11.04	12.23	12.15	12.19	–0.01	–1.16
21	11.10	11.20	11.15	11.26	11.22	11.24	12.20	12.25	12.22	–0.09	–1.07
22	11.79	11.89	11.84	11.93	11.96	11.95	12.80	12.71	12.76	–0.11	–0.92
Average			9.47			9.51			10.65	–0.04	–1.05

analyzed by both methods. Table 3 summarizes the results. The regression analysis between ISO 6673 and ISO 1447 shows a very good correlation with R^2 of 0.999 and a standard error of differences of 0.078% w/w (Fig. 3). Based on the 18 samples we found a significant bias of 1.05% w/w. This bias is constant over the water content range and independent of the origin of the green coffee.

Table 3 also compares the results obtained by ISO 6673, using an oven with (as recommended in the standard) and without forced ventilation. The regression analysis between the two procedures shows a very good correlation with R^2 of 0.992 and a standard error of differences of 0.05% w/w. Based on 18 samples we found a bias of 0.065% w/w, which due to the excellent repeatability is statistically significant. This bias is very

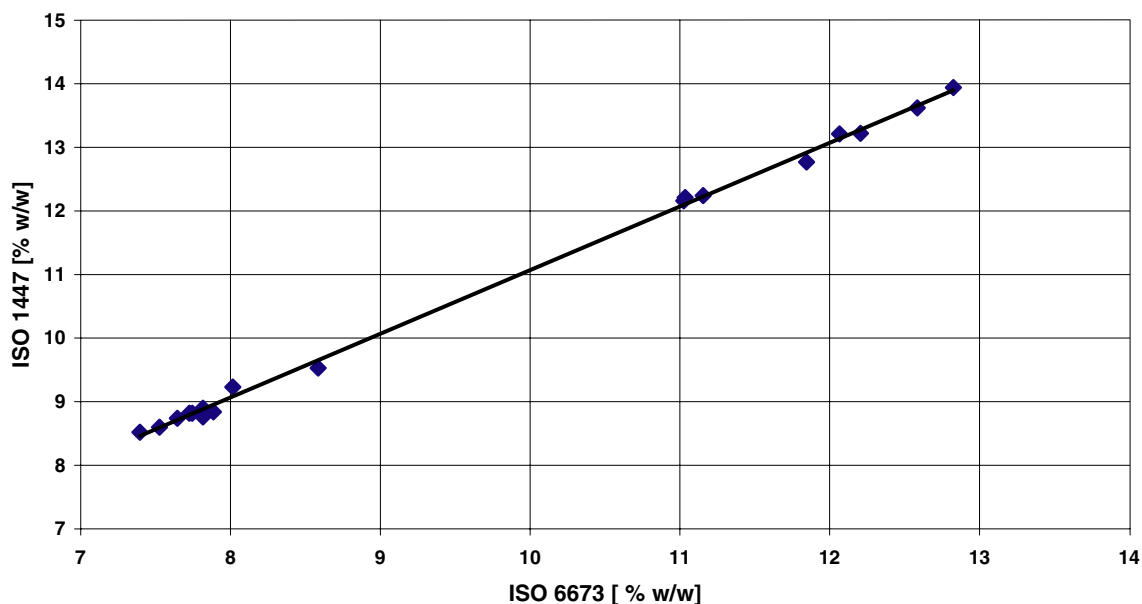


Fig. 3. Correlation between ISO 1447 and ISO 6673.

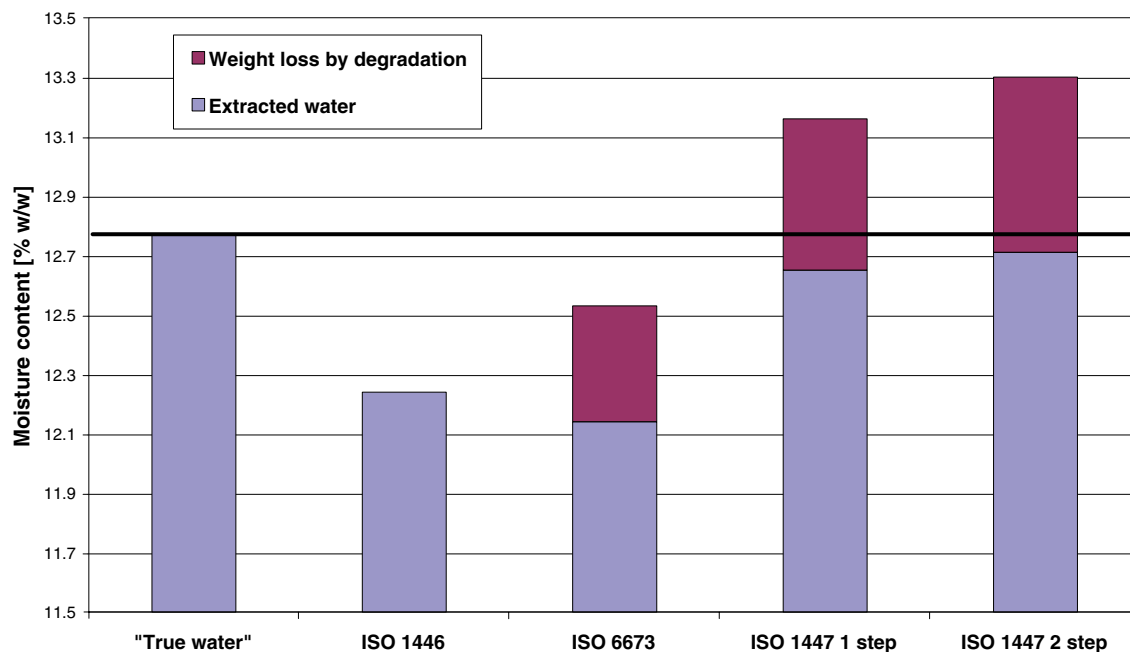


Fig. 4. Comparison of results.

small and is difficult to be explained especially as we expected that the more effective air movement around the sample due to the forced ventilation should lead to a better drying. From a practical point of view the oven without ventilation is cheaper and more robust in the operation. As results are very comparable we suggest that in routine application both types of oven can be used.

9. Conclusions

Fig. 4 summarizes the overall results by well illustrating the repartition of the extracted water and the water loss by degradation. The reference method ISO 1446 has proved to let residual water content at the end of the drying (e.g., 2 weeks). This residual value was very sensitive to the particle size, due to the very low water diffusion in dehydrated green coffee material. Results obtained with ISO 6673 present a significant bias of 0.3% w/w compared to ISO 1446. The bias is constant over the whole water content range. All methods are highly correlated with each other with R^2 values above 0.990. This will allow the upgrade of the calibrations of the water content analyzers by a simple bias adjustment procedure.

ISO 1447 method surprisingly gave not results similar to ISO 1446 method, but higher values (average difference: 1.38%). However, the usual average difference of 1.05% was measured between ISO 1447 and ISO 6673.

Fig. 4 summarizes the overall results by well illustrating the repartition of the extracted water and the water

loss by degradation. The repeatability standard deviation is very good and similar for all the tested methods: about 0.06% w/w.

Near-infrared spectroscopy (NIRS) was applied to study the different drying methods. Due to the specificity of the 1940 nm wavelength band for water molecules the drying process can be indeed easily monitored. By comparing the weight loss results with the residual NIRS absorption left after drying, interesting conclusion can be drawn on the losses due to water evaporation under different oven and surrounding conditions or due to degradation reaction products. Neither ISO 1446 nor ISO 6673 methods allow complete drying of green coffee.

Further trials showed that the results obtained with ISO 6673 were independent of the climate conditions and the presence of forced ventilation. This would allow the use of cheaper standard ovens in laboratories where climatic conditions are not necessarily well controlled. ISO 6673 requires the least input of labor as no grinding is required and only one drying step is applied. It is therefore well suited for routine purposes.

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