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Green coffee — Procedure for calibration of moisture meters — Routine method

Café vert — Mode opératoire d'étalonnage des humidimètres — Méthode de routine

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Foreword

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ISO 24115 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 15, *Coffee*.

Green coffee — Procedure for calibration of moisture meters — Routine method

1 Scope

This International Standard specifies a procedure for adjustment and subsequent calibration of moisture meters for green coffee beans with reference samples (RSs).

The RSs are green coffee beans of various moisture contents, determined by a standard method (ISO 6673).

NOTE This method of determining the loss in mass can be considered, by convention, as a method for determining the water content and can be used as such by agreement between the interested parties.

This International Standard is applicable to green coffee as beans.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3509, *Coffee and coffee products — Vocabulary*

ISO 6673:2003, *Green coffee — Determination of loss in mass at 105 °C*

ISO/IEC Guide 99, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3509, ISO/IEC Guide 99, and the following apply.

3.1
reference sample
RS
sample of green coffee beans, sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties

NOTE See Annex A.

4 Principle

From green coffee beans, a set of samples with different moisture contents is prepared, to be taken as reference samples (RS). Their individual moisture content (or mass fraction loss) values are obtained by the respective determinations of loss in mass by applying ISO 6673.

These RSs with assigned moisture values are used for calibration of moisture meters.

5 Equipment and material

5.1 Green coffee as beans, in a quantity sufficient to prepare n reference samples, RSi, $i = 1 \ldots n$ (the minimum is 5), with moisture mass fraction ranges between 8,5 % and 13,5 %, prepared according to A.1.

5.2 **Thermometer**, range between 0 °C to 120 °C, scale division 0,1 °C, provided the moisture meter does not display the sample temperature.

5.3 **Moisture meter**, equipped with all the accessories specified by the manufacturer. It shall include a channel for green coffee, which will be the subject of calibration.

5.4 **Balance**, capable of being read to the nearest 0,1 g.

5.5 **Equipment necessary for determinations according to ISO 6673**.

6 Procedure

6.1 Test conditions

The procedure shall be carried out at ambient temperature and at a relative humidity from 40 % to 70 %.

Prior to the test, allow conditioning of the green coffee bean RSs to ambient temperature.

6.2 Alignment of instrument deviation

6.2.1 Before adjustment, verify the equipment in accordance with the manufacturer's instructions.

NOTE The verification is necessary to ensure the electronic gauging.

6.2.2 For moisture meters with direct reading, select green coffee beans channel.

6.2.3 For each RSi, obtain the difference between the reference sample moisture mass fraction, $w_{\text{RS}i}$, determined according to ISO 6673, and the moisture meter reading, $w_{\text{r}i}$, where i is the reference sample number assigned. Record them as $\Delta w_i = w_{\text{RS}i} - w_{\text{r}i}$ in the column headed "Difference" of Table 1.

6.2.4 Calculate the average of the differences, $\overline{\Delta w}$, and adjust the moisture meter bias according to the manufacturer's operation manual.

Table 1 — Input values to obtain the bias for moisture meter adjustment

Reference sample RSi, $i = 1 \ldots n$	Moisture mass fraction of reference sample[a] $w_{\text{RS}i}$	Equipment reading on sample $w_{\text{r}i}$	Difference $\Delta w_i = w_{\text{RS}i} - w_{\text{r}i}$
RS1			
RS2			
...			
RS(n – 1)			
RSn			
		$\overline{\Delta w}$	

[a] The moisture content (or mass fraction loss), $w_{\text{RS}i}$, is obtained using ISO 6673.

6.3 Calibration of moisture meter

6.3.1 For calibration, use the moisture meter after adjustment (6.2).

6.3.2 Take the oven moisture values of the RSs, obtained according to A.2, and insert the figures into the appropriate w_{RSi} cells in Table 2.

6.3.3 Take a test portion from RS1 in accordance with the equipment manufacturer's instructions or the equipment capacity, and pour it into the moisture meter. Record the reading from the equipment in the cell matching the first row, RS1, and column, $w_{ri,1}$, of Table 2; this cell corresponds to $w_{r1,1}$.

Provided the equipment requires a defined test portion quantity, use the balance specified in 5.4.

6.3.4 Repeat the the procedure specified in 6.3.3 with the same test portion twice and record the readings in the subsequent cells which correspond to $w_{r1,2}$, and $w_{r1,3}$ for RS1.

At least three determinations shall be performed.

6.3.5 Calculate the arithmetic average of the RS1 readings, $w_{r1,1}$, $w_{r1,2}$, and $w_{r1,3}$, and record it in the corresponding cell of column $\overline{w_{ri,j}}$, $i = 1 \ldots n$; $j = 1,2,3$ of Table 2.

6.3.6 Calculate the experimental standard deviation, s_1, of $w_{r1,1}$, $w_{r1,2}$, and $w_{r1,3}$ and insert it into the corresponding cell of column s_i of Table 2.

6.3.7 Measure the temperature of the test portion RS1 and record it in the corresponding cell of column T_i of Table 2.

6.3.8 Repeat the procedure as specified in 6.3.3 to 6.3.7 with the other reference samples, RS2 to RSn and their test portions.

6.3.9 Determine a correction term, C_{ri}, to compensate for an estimated systematic effect of moisture for each w_{ri} moisture reading by applying Formula (1):

$$C_{ri} = w_{RSi} - \overline{w_{ri\,j}} \qquad i = 1 \ldots n \qquad j = 1,2,3 \tag{1}$$

NOTE For an example, see Table D.2.

Table 2 — Input values to obtain the calibration of moisture meter

Reference sample	Sample temperature °C	Equipment readings on RSs and corresponding mean				Standard deviation of reading	Reference sample moisture[a]	Moisture meter correction
RSi	T_i	$w_{ri,1}$	$w_{ri,2}$	$w_{ri,3}$	$\overline{w_{ri,j}}$	s_i	w_{RSi}	$C_{ri} = w_{RSi} - \overline{w_{ri\,j}}$
RS1								
RS2								
...								
RS(n – 1)								
RSn								

[a] The moisture content (or mass fraction loss), w_{RSi}, is obtained using ISO 6673; see A.2.

6.4 Temperature correction

6.4.1 Calculate the moisture reading correction term, C_T, due to bean temperature (if not automatically done by the instrument itself) based on equipment manufacturer instructions; otherwise follow steps 6.4.2 to 6.4.5.

NOTE Some manufacturers provide an automatic temperature correction programme with their instruments.

6.4.2 Determine the moisture content of the RSs with the moisture meter at a given temperature.

6.4.3 Increase the temperature of the samples by about 5 °C to 10 °C and determine the new values at the moisture meter.

6.4.4 For each RS, take the difference in moisture readings at test temperatures, and the difference of temperatures, and calculate the respective ratio

$$\xi_i = \frac{\Delta w_{\mathrm{r}i}}{\Delta T} \tag{2}$$

6.4.5 Calculate the arithmetic mean of the ratios, $\bar{\xi}$, to obtain the resulting moisture reading correction term C_T due to bean temperature.

$$C_T = \bar{\xi}\left(T_{\mathrm{ref}} - T_{\mathrm{s}}\right) \tag{3}$$

NOTE For an example, see Table D.4.

6.5 Calculation of uncertainty

This approach is based on that set out in ISO/IEC Guide 98-3:2008.[1]

When reporting the result of a measurement of a physical quantity, it is obligatory that some quantitative indication of the quality of the result be given so that those who use it can assess its reliability. Without such an indication, measurement results cannot be compared, either among themselves or with reference values given in a specification or standard.

It is therefore necessary that there is a readily implemented, easily understood, and generally accepted procedure for characterizing the quality of a result of a measurement, that is, for evaluating and expressing its uncertainty.1)

The components of moisture measurement uncertainty are:

a) $U_{\mathrm{RS}i}$: uncertainty of the (assigned) value of the reference sample;

b) $s_{\mathrm{r}i}/\sqrt{j}$: uncertainty type A given by the moisture meter readings obtained from the experimental standard deviation $s_{\mathrm{r}i}$ using reference material i, with $j =$ 3 repetitions (see 6.3.6);

c) u_{Ba}: uncertainty type B due to moisture meter accuracy;

d) u_{Br}: uncertainty type B due to moisture meter resolution;

e) $u_{\mathrm{B}Ti}$: uncertainty type B due to the temperature (see 6.4 and D.4).

Calculate the expanded uncertainty of the moisture meter, $U_{\mathrm{m}i}$, for each moisture point i using Formula (4):

$$U_{\mathrm{m}i} = k\sqrt{\left(\frac{U_{\mathrm{RS}i}}{k}\right)^2 + \left(\frac{s_{\mathrm{r}i}}{\sqrt{3}}\right)^2 + u_{\mathrm{Ba}}^2 + u_{\mathrm{Br}}^2 + u_{\mathrm{B}Ti}^2} \tag{4}$$

where k is the coverage factor; generally $k = 2$ for a probability of 95 %, according to ISO/IEC Guide 98-3:2008.[1]

For each individual case, it is necessary to check every possible source of uncertainty.

1) In most cases, regressions are linear, however, it can prove necessary to apply another type, e.g. quadratic, regression.

6.6 Calibration curves for indirect reading equipment

If the equipment does not give a measurement result directly in moisture content, but as a dimensionless value, generate a calibration curve by comparing the equipment readings for each sample with the moisture of the RSs.

For a graphic calibration, plot the n values of the reading means, $\overline{w_{\mathrm{r}i,j}}$, on the abscissa against the n values of sample moisture content, $w_{\mathrm{RS}i}$ on the ordinate, with $i = 1 \ldots n$ for the samples and $j = 1, 2, 3$ for the replicate readings to obtain the calibration curve.

The calibration curve relates the equipment readings to moisture content (or mass fraction loss) of the reference samples.

For calculation, a linear regression is suitable in most cases.[1]

7 Expression of results

The moisture content of a sample, $w_{\mathrm{H_2O}}$, determined using a moisture meter which has been calibrated with the method described in this International Standard, shall be expressed as

$$w_{\mathrm{H_2O}} = \left(w_{\mathrm{r}} + C_{\mathrm{r}} + C_T \right) \pm U_{\mathrm{m}} \tag{5}$$

where

w_{r} is the moisture content meter reading;

C_{r} is the correction for the moisture meter reading;

C_T is the correction for the moisture meter due to the temperature;

U_{m} is the expanded uncertainty for the moisture mass fraction in the sample.

All values are given as percentage mass fractions.

NOTE D.6 gives an example of the final calculation.

Annex A
(normative)

Reference sample preparation and determination of moisture content as a mass fraction loss

A.1 Reference sample preparation

A.1.1 Decide the number, n ($n \geq 5$), of RSs to be used in the calibration process.

A.1.2 Select the starting material, green coffee beans of single species and homogeneous characteristics, with an initial moisture mass fraction between 14 % and 16 %.

NOTE 1 Depending on the measurement principle of the moisture meter to be calibrated and the precision required, it can prove necessary to perform individual calibrations for coffees of different bulk or intrinsic characteristics of the beans, like shape, size or any steaming pretreatment used.

NOTE 2 Some moisture meters allow different parallel installations of green coffee channels, adapted to the specific coffees to be analysed.

A.1.3 Obtain a total mass of green coffee quantity necessary to prepare n RSs, i.e. $n \times 600$ g, since, for each RS, 500 g is required for the moisture meter and an additional 100 g for the replicate moisture determinations according to ISO 6673.

The 500 g mass is recommended due to mass loss during the drying process, taking into consideration that most commercially available moisture meters accept up to 400 g.

A.1.4 Place one sample portion in the oven (5.5) at a temperature of 40 °C ± 2 °C. After drying, label it RS1.

A.1.5 Repeat the same procedure of A.1.4 with the other RSs, applying different drying times to obtain the RSi ($i = 1 \ldots n$) series with different moisture contents. Label the subsequent RSs as RS2, RS3 ... RSn until the desired moisture range is covered.

NOTE The validated mass fraction range with ISO 6673 is 8,5% to 13,5%.

Differences in moisture content, Δw, as percentage mass fractions, between two consecutive RSs should be in the range $0,7 \leq \Delta w \leq 1,3$.

A.1.6 Place each RS separately into an airtight container or a self-sealing double plastic bag and store them for 72 h to allow a homogeneous moisture distribution throughout each RS to be achieved.

A.2 Determination of oven moisture content (mass fraction loss)

A.2.1 When ready, take from each of RS1, RS2 ... RSn at least three test portions to perform the procedure in ISO 6673:2003, Clause 7. Mark each test portion as $O_{1,1}$, $O_{1,2}$, $O_{1,3}$, $O_{2,1}$, $O_{2,2}$, $O_{2,3}$ ... $O_{n,1}$, $O_{n,2}$, $O_{n,3}$, where O denotes “oven”.

A.2.2 Determine the moisture content (or mass fraction loss) of RS1 in accordance with ISO 6673. Use Table A.1 to record the values $w_{O_{1,1}}$, $w_{O_{1,2}}$, and $w_{O_{1,3}}$ in the appropriate mass fraction columns.

A.2.3 Calculate the arithmetic mean of the three values for RS1 obtained in A.2.2, and record it in Table A.1, column $\overline{w_{\mathrm{RS}i}}$.

A.2.4 Calculate the standard deviation, s_{O_1}, of $w_{\mathrm{O}_{1,1}}$, $w_{\mathrm{O}_{1,2}}$, and $w_{\mathrm{O}_{1,3}}$ and record it in Table A.1, column $s_{\mathrm{RS}i}$.

A.2.5 Calculate the standard uncertainty, u_1, of $w_{\mathrm{O}_{1,1}}$, $w_{\mathrm{O}_{1,2}}$, and $w_{\mathrm{O}_{1,3}}$ as $u_1 = s_{\mathrm{O}_1} / \sqrt{3}$ and record it in Table A.1, column u_i.

A.2.6 Calculate the expanded uncertainty: U_{RS1} as ku_1 and record it in Table A.1, column $U_{\mathrm{RS}i}$.

A.2.7 Repeat the procedure specified in A.2.2 to A.2.6 with RS2 to RSn.

NOTE Table B.1 gives an example of the use of Table A.1.

A.2.8 Pack the remaining material from each RS in an airtight container or a self-sealing double plastic bag. Label them as specified in A.3.

A.2.9 If there is a delay between oven moisture determination and usage of the RSs for moisture meter calibration, a re-checking of oven moisture may become necessary. The expiry time depends on storage conditions.

A.3 Reference sample labelling

The labels for the reference samples shall clearly indicate:

a) the coffee species (e.g. *Coffea arabica* or *C. canephora*);

b) whether decaffeinated or non-decaffeinated;

c) the moisture content of each reference sample and the standard method used to determine it;

d) the uncertainty of reference sample moisture content;

e) the date of reference sample preparation;

f) the expiry time of the reference sample.