INTERNATIONAL RECOMMENDATION

OIML R 146-1

Edition 2016 (E)

Protein measuring instruments for cereal grain and oilseeds

Part 1: Metrological and technical requirements

Instruments de mesure de protéine pour grains de céréales et graines oléagineuses

Partie 1: Exigences métrologiques et techniques

ORGANISATION INTERNATIONALE DE MÉTROLOGIE LÉGALE

INTERNATIONAL ORGANIZATION OF LEGAL METROLOGY

Contents

Foreword

The International Organization of Legal Metrology (OIML) is a worldwide, intergovernmental organization whose primary aim is to harmonize the regulations and metrological controls applied by the national metrological services, or related organizations, of its Member States. The main categories of OIML publications are:

- **International Recommendations (OIML R),** which are model regulations that establish the metrological characteristics required of certain measuring instruments and which specify methods and equipment for checking their conformity. OIML Member States shall implement these Recommendations to the greatest possible extent;
- **International Documents (OIML D),** which are informative in nature and which are intended to harmonize and improve work in the field of legal metrology;
- **International Guides (OIML G),** which are also informative in nature and which are intended to give guidelines for the application of certain requirements to legal metrology; and
- **International Basic Publications (OIML B),** which define the operating rules of the various OIML structures and systems.

OIML Draft Recommendations, Documents and Guides are developed by Project Groups linked to Technical Committees or Subcommittees which comprise representatives from the Member States. Certain international and regional institutions also participate on a consultation basis. Cooperative agreements have been established between the OIML and certain institutions, such as ISO and the IEC, with the objective of avoiding contradictory requirements. Consequently, manufacturers and users of measuring instruments, test laboratories, etc. may simultaneously apply OIML publications and those of other institutions.

International Recommendations, Documents, Guides and Basic Publications are published in English (E) and translated into French (F) and are subject to periodic revision.

Additionally, the OIML publishes or participates in the publication of **Vocabularies (OIML V)** and periodically commissions legal metrology experts to write **Expert Reports (OIML E)**. Expert Reports are intended to provide information and advice, and are written solely from the viewpoint of their author, without the involvement of a Technical Committee or Subcommittee, nor that of the CIML. Thus, they do not necessarily represent the views of the OIML.

This publication – reference OIML R 146-1, Edition 2016 (E) – was developed by Project Group 1 in Technical Subcommittee TC 17/SC 8 *Instruments for quality analysis of agricultural products*. It was approved for final publication by the International Committee of Legal Metrology in 2016 and was submitted to the International Conference on Legal Metrology in 2016 for formal sanction.

OIML Publications may be downloaded from the OIML web site in the form of PDF files. Additional information on OIML Publications may be obtained from the Organization's headquarters:

Bureau International de Métrologie Légale
11, rue Turgot - 75009 Paris - France
Telephone: 33 (0)1 48 78 12 82
Fax: 33 (0)1 42 82 17 27
E-mail: biml@oiml.org
Internet: www.oiml.org

Part 1: Metrological and technical requirements

1 Scope

This Recommendation includes metrological and technical requirements and test methods for the metrological control of digital, self-indicating measuring instruments used to determine the protein content of grain and oilseeds for commercial transactions. It specifies accuracy requirements including maximum permissible errors (MPEs) for verification and other error limits for type evaluation tests.

The provisions in this Recommendation are based on the performance of instruments that estimate the mass fraction of protein constituents in grain and oilseeds via inferential means. Instruments that modify the test sample in order to infer the protein content are not specifically covered, but may still qualify for type approval if the requirements in the Recommendation are met. This Recommendation is not intended to preclude the application of new technologies to grain protein measurement.

2 Terminology

2.1 General metrology and legal metrology terms

The basic terminology used in this Recommendation is consistent with the definitions in OIML V 2-200 *International Vocabulary Metrology – Basic and General Concepts and Associated Terms* (VIM) [1] and OIML V 1 *International Vocabulary of Terms in Legal Metrology* (VIML) [2].

Table 1 is limited to additional information such as the context in which the general terms are used in this Recommendation. Some definitions from the VIM and VIML have been reproduced in alphabetical order in Annex D.

Table 1: Additional notes on the general terms in this Recommendation

General term	R 146 additional notes	Reference
accuracy; measurement accuracy	--	VIM 2.13
adjustment	For protein measuring instruments, alignment with the reference method is typically accomplished through a bias adjustment to the calibration equation. Other mechanisms that require a higher level of expertise (e.g. adjustment of the calibration equation slope, modification of hardware/ software components or settings) may be less accessible due to increased security requirements.	VIM 3.11
certified reference material (CRM)	Refer to Annex B for guidelines on producing whole-grain CRMs. Further general information is in OIML D 18:2008 [3].	VIM 5.14
maximum permissible error (MPE); limit of error	The MPE and other limits for tests on the type of instrument and various grain calibrations are listed in 4.5, Table 4.	VIM 4.26
measurement error; error	--	VIM 2.16
measured quantity value, measured value; indication	Unless specified otherwise, the measured quantity value is a single P_{MB} indication on a sample.	VIM 2.10
rated operating condition	--	VIM 4.9
reference condition	--	VIM 4.11
reference material (RM)	--	VIM 5.13
reference quantity value	In this Recommendation, the P_{MB} of the whole-grain CRM is the reference quantity value used to assess the measurement accuracy at verification and to assess the accuracy of calibrations at type evaluation. Where a CRM is not used, the reference quantity value is the mean P_{MB} at reference conditions prior to a test.	VIM 5.18
repeatability; measurement repeatability	--	VIM 2.21
repeatability condition of measurement	--	VIM 2.20
reproducibility; measurement reproducibility	In this Recommendation, the reproducibility of measurements between units of the same type of instrument under reference conditions is assessed by the standard deviation of differences (SDD_I). The reproducibility of measurements from one instrument when selected influence factors are varied is assessed by the magnitude of the error shift or fault.	VIM 2.25
reproducibility condition of measurement	For the tests in this Recommendation, the conditions changed and unchanged are summarized in R 146-2, Annex A, A.3.4.	VIM 2.24
type approval	--	VIML 2.05
type (pattern) evaluation	--	VIML 2.04
verification of a measuring instrument	--	VIML 2.09

2.2 Other definitions

This subclause defines terms applicable to grain protein measuring instruments, the assessment of multivariate calibrations and also includes definitions from OIML D 11:2013 [4] and OIML D 31:2008 [5].

2.2.1 accuracy of a grain protein calibration; calibration accuracy

performance characteristic of a calibration assessed at reference conditions

Note: The assessment requires calculation of $\bar{y}$, the bias over a set of test samples or the 'calibration bias', and the standard error of prediction (SEP) which is the standard deviation of measurement errors from the same sample set.

Refer to R 146-2, Annex A, A.7.1 for the calculation of $\bar{y}$ and SEP from measured values. The limiting values for $\bar{y}$ and SEP in 4.5, Table 4 shall be observed in order to deem a calibration as sufficiently accurate.

2.2.2 average error shift

algebraic mean of error shift values calculated from samples of the same grain type with different protein (P_{MB}) levels

Note: The resulting 'average' value is indicative of the average variation over the encompassed measurement range, as opposed to the variation in measured values at one point of the range.

In this Recommendation, reference to a resulting 'mean' value is reserved for the mean of replicated measurements, i.e. the mean of measured values on the same test sample (usually taken under repeatability conditions).

2.2.3 calibration equation; calibration

set of calibration coefficients for one type of grain to convert raw instrument data into a protein content measurement

Note: Both these terms are used in the same context as 'calibration function' in Note 1 of VIM 2.39.

2.2.4 cryptographic means [further information in OIML D 31:2008, 3.1.11]

encryption of data by the sender (storing or transmitting program) and decryption by the receiver (reading program) with the purpose of hiding information from unauthorized persons

electronic signing of data with the purpose of enabling the receiver or user of the data to verify the origin of the data, i.e. to prove their authenticity

2.2.5 error shift

difference between the mean error of indication while one or more influence quantities are varied within the rated operating conditions and the mean intrinsic error of a measuring instrument with reference to a certified measurement standard

Refer to Table 2 for the relevant measured values in the calculation of errors.

Note: If a certified measurement standard is not used, the error shift is the difference between two measured values: the indication under rated operating conditions and the mean indication at reference conditions prior to test.

Table 2: Measured values for calculating the error shift exhibited by the instrument

Mean error of indication		Mean intrinsic error	
Measured quantity value	Reference quantity	Measured quantity value	Reference quantity
Mean of P_{MB} indications under rated operating conditions	If CRM is used - P_{MB} of CRM	Mean of P_{MB} indications at reference conditions prior to test	If CRM is used - P_{MB} of CRM

2.2.6 fault [OIML D 11:2013, 3.9]

difference between the error of indication (during or after exposure to a disturbance) and the mean intrinsic error of a measuring instrument with reference to a certified measurement standard

OIML D 11:2013 Notes

1 Principally, a fault is the result of an undesired change of data contained in or flowing through an electronic measuring instrument.

2 From the definition it follows that a 'fault' is a numerical value which is expressed either in a unit of measurement or as a relative value.

Note: Refer to Table 3 for the relevant measured values in the calculation of errors.

Note: If a certified measurement standard is not used, a fault is the difference between a single indication during or after a disturbance and the mean indication at reference conditions prior to test.

Table 3: Measured values for calculating the fault exhibited by the instrument during or after a disturbance

Measurement error (error of indication)		Mean intrinsic error	
Measured quantity value	Reference quantity	Measured quantity value	Reference quantity
Single P_{MB} indication during or after the disturbance	If CRM is used - P_{MB} of CRM	Mean of P_{MB} indications at reference conditions prior to test	If CRM is used - P_{MB} of CRM

2.2.7 grain

for the purpose of this Recommendation, the term "grain" is taken to mean those cereal grains and oilseeds listed in column 1 of Table 4 with samples that comply with any limits specified by the national responsible body for the sample temperature (see 4.3)

2.2.8 integrity of programs, data or parameters

assurance that the programs, data or parameters have not been subjected to any unauthorized or unintended changes while in use, transfer, storage, repair or maintenance

2.2.9 intrinsic error [OIML D 11:2013, 3.7]

error of a measuring instrument, determined under reference conditions

Note: The grain sample would also be at the reference conditions.

2.2.10 legally relevant [OIML D 31:2008, 3.1.29]

software/hardware/data or part of the software/hardware/data of a measuring instrument which interferes with properties regulated under legal metrology, e.g. the accuracy of the measurement or the correct functioning of the measuring instrument

2.2.11 basis moisture content; moisture basis (M_B)

basis moisture concentration, expressed as a percentage by mass, specified by the national responsible body for reporting protein content of the particular grain type

Note: When the specified M_B is 0 %, the reported protein content is at 'dry basis'.

2.2.12 open network [OIML D 31:2008, 3.1.35]

network of arbitrary participants (electronic devices with arbitrary functions)

Note: The number, identity and location of a participant can be dynamic and unknown to the other participants. This is in contrast to a closed network [OIML D 31:2008, 3.1.6] which is a network of a fixed number of participants with a known identity functionality and location.

2.2.13 protein content (P_{MB})

concentration of protein in a grain sample, expressed as a percentage by mass, calculated at the basis moisture content (M_B)

Note: When the specified M_B is 0 %, the reported protein content is at 'dry basis' (i.e. $P_{0\%}$).

2.2.14 protein measuring instrument; instrument; unit

instrument that infers the protein content in grain samples that are within the scope of its calibration

Note: An instrument may be approved with multiple calibrations in order to analyze more than one type of grain.

2.2.15 sample temperature sensitivity (STS)

measurement variation (relative to the P_{MB} values obtained at reference conditions) resulting from the range of grain sample temperatures permitted in commercial measurements

Note: STS is controlled in approved P_{MB} calibrations. During assessment, a limit is placed on the value of the average error shift caused by allowable temperature variations.

2.2.16 significant fault

fault exhibited by the equipment under test that is greater than the values listed in 4.5, Table 4, column 10

The following faults are not considered to be significant, even when they exceed the maximum value:

(a) faults arising from a simultaneous and mutually independent cause (e.g. EM fields and discharges) originating in a measuring instrument or in its checking facilities;

(b) faults implying the impossibility to perform any measurement; and

(c) transitory faults being momentary transitions in the indication, which cannot be interpreted, memorized or transmitted as a measurement result.

2.2.17 universal computer [OIML D 31:2008, 3.1.54]

computer that is not constructed for a specific purpose but that can be adapted to the metrological task by software

Note: In general this software is founded on an operating system that permits loading and execution of software for specific purposes.

2.2.18 (software) validation [OIML D 31:2008, 3.1.56]

confirmation by examination and provision of objective evidence (i.e. information that can be proved true, based on facts obtained from observations, measurement, tests, etc.) that the particular requirements for the specific intended use are fulfilled

Note: In the present case the related requirements are those of this Recommendation.

2.3 Abbreviations and acronyms

AC	alternating current
AM	amplitude modulation
ASD	acceleration spectral density
CRM	certified reference material
EM	electromagnetic
EMC	electromagnetic compatibility
ESD	electrostatic discharge
EUT	equipment under test
IEC	International Electrotechnical Committee
ISO	International Organization for Standardization
M	actual 'as is' moisture content of a sample
M_B	basis moisture content specified by the national responsible body (see 2.2.11)
MPE	(also $MPE_{MB>0\%}$ for the purpose of Equation 2) maximum permissible error scaled to the relevant M_B
$MPE_{0\%}$	maximum permissible error at dry basis (i.e. $M_B = 0$ %)
OIML	International Organization of Legal Metrology
P_M	mass percentage protein content at the actual moisture content
P_{MB}	mass percentage protein content calculated at the basis moisture content (see 2.2.13 and 3.2)
RF	radio frequency
RH	relative humidity
RM	reference material
RMS	root mean square
SEP	standard error of prediction (see Part 2, A.7.1)
SD	standard deviation (see Part 2, A.7.1)
SDD_I	SD of differences (see Part 2, A.7.1)
STS	sample temperature sensitivity
t	actual temperature during a test
t_{ref}	reference temperature during a test
Δt	magnitude of the temperature difference between a sample and an instrument at t_{ref}
Δt_{max}	maximum Δt specified by the national responsible body for type testing
$\Delta t_{C,\,max}$	maximum permitted Δt_{max} below t_{ref} (applicable only if unequal to $\Delta t_{H,\,max}$)
$\Delta t_{H,\,max}$	maximum permitted Δt_{max} above t_{ref} (applicable only if unequal to $\Delta t_{C,\,max}$)
t_C	minimum environmental temperature specified by the national responsible body for type testing
t_H	maximum environmental temperature specified by the national responsible body for type testing
$t_{C,sample}$	minimum grain sample temperature specified by the national responsible body type testing
$t_{H,sample}$	maximum grain sample temperature specified by the national responsible body type testing
$\bar{y}$	bias of a calibration (see Part 2, A.7.1)

2.4 Additional symbols and subscripts used in equations

d_i difference in $\bar{x}_i$ from two units (same type)

$\bar{d}$ d_i averaged over n test samples

i identifier for a test sample in a set of n samples

j identifier for a measured value in a series obtained by repeated measurements

n number of test samples with different P_{MB} levels of the same grain type used in a test

r_i certified P_{MB} value for test sample i

x measured P_{MB} value for test sample i

$\bar{x}_i$ mean x for test sample i

y_i error of measured P_{MB} value for test sample i

$\bar{y}_i$ mean y for test sample i

$\bar{y}$ $\bar{y}_i$ averaged over n test samples (i.e. the bias of a calibration)

3 Units of measurement

3.1 The unit of measurement used for the protein content of a grain sample is percentage protein by mass (see 2.2.13). The abbreviation for percentage by mass is % w/w. Conventionally, the percentage symbol alone (%) is used.

3.2 P_M is the protein content at the actual moisture content of the sample. To allow comparison across samples with varying moisture levels, P_M, must be converted to P_{MB}, which is the protein content at the basis moisture content.

$$P_{MB} = P_M \times \frac{100 - M_B}{100 - M} \qquad \textbf{Equation 1}$$

where: M = the moisture content of the sample
M_B = the basis moisture content for the type of grain

3.3 The national responsible body shall clearly specify the basis moisture content (M_B) for all applicable grain types.

4 Metrological requirements

4.1 Applicable grains and P_{MB} measuring ranges – specification

4.1.1 Due to climatic and crop variability, the national responsible body shall specify commercially important P_{MB} ranges for the types of grain listed in Table 4. These are examples of the grain calibrations for which a protein measuring instrument manufacturer may seek national approval.

4.1.2 The manufacturer shall specify the types of grain and respective P_{MB} measuring ranges that the instrument can analyze. Measuring ranges as specified by the manufacturer shall include any commercially important P_{MB} ranges specified by the national responsible body.

4.2 Instrument environmental operating temperature – specification

4.2.1 The errors on the measured values displayed by a protein measuring instrument shall be below the MPE in Table 4 regardless of the environmental temperature, unless the national responsible body permits limitations on the conditions in which the instrument can be used.

4.2.2 In the latter case, the national responsible body shall specify the range of ambient temperatures (t_C to t_H) in which the instrument can be used to take P_{MB} measurements for commercial purposes. The t_C to t_H specified shall include the temperature range 10 ºC to 30 ºC.

4.2.3 The manufacturer may specify a wider temperature range than the t_C to t_H required by the national responsible body in order to meet international requirements. The manufacturer may request type testing and approval over the wider environmental operating temperature range (i.e. for that particular type approval application, the manufacturer specified ranges are adopted as t_C to t_H).

4.3 Grain sample operating temperature – specification

4.3.1 Specification of the sample temperature range

The errors on the measured values displayed by a protein measuring instrument shall meet the MPE in Table 4 regardless of the sample temperature unless the national responsible body permits limitations on the temperature of grain that can be tested.

In the latter case, the national responsible body shall specify the temperature range ($t_{C,sample}$ to $t_{H,sample}$) allowed for grain samples tested for commercial purposes. For each type of grain, the $t_{C,sample}$ to $t_{H,sample}$ specified shall include the minimum temperature range 2 ºC to 40 ºC.

In order to meet international requirements, the manufacturer may specify a wider sample temperature range for each type of grain than $t_{C,sample}$ to $t_{H,sample}$ specified by the national responsible body. The manufacturer may request type testing and approval over the wider sample temperature range (i.e. for that particular type approval application, the manufacturer specified ranges are adopted as $t_{C,sample}$ to $t_{H,sample}$).

4.3.2 Specification of the sample and instrument maximum temperature differential (Δt_{max})

An instrument at reference temperature (t_{ref}) shall be able to analyze cooler or warmer samples within the range $t_{C,sample}$ to $t_{H,sample}$ regardless of the magnitude of the sample and instrument temperature differential (Δt). This requirement may be limited in effect if the national responsible body permits a limit on the temperature differential (i.e. Δt_{max} or $\Delta t_{C,\ max}$ and $\Delta t_{H,\ max}$ for cold and hot samples if unequal) in which the instrument is used.

In the latter case, the national responsible body shall specify the value of Δt_{max} (or $\Delta t_{C,max}$ and $\Delta t_{H,max}$) for taking P_{MB} measurements for commercial purposes. The instrument shall be able to take into account a Δt of at least 10 °C.

In order to meet international requirements, the manufacturer may specify a maximum allowable Δt that is larger than the Δt_{max} specified by the national responsible body. The manufacturer may request type testing and approval at the larger maximum allowable Δt (i.e. for that particular type approval application, the manufacturer specification is adopted as Δt_{max}).

4.3.3 Provisions in the absence of a manufacturer-specified sample temperature range

A declaration by the manufacturer regarding the sample temperature range or a maximum allowable Δt may not be feasible if the submitted type is not able to measure sample temperature and/or the calibration does not account for sample temperature variations. The operating procedure and/or metrological tests defined by the national responsible body shall ensure that the requirements in 4.7.2 and 4.8.1 are met.

4.4 Influence quantities – specification

The following influence quantities have the potential to reduce the accuracy or cause a grain protein measuring instrument to malfunction. An influence quantity that is not within any of the ranges in 4.4.1 also qualifies as being a disturbance, although its effect on measurements is not specifically assessed at type evaluation.

4.4.1 Rated operating conditions

(a) Ambient temperature: Operating range specified by the national responsible body (t_C to t_H). See 4.2 for requirements

(b) Relative humidity: Below 85 %, non condensing

(c) Atmospheric pressure: 86 kPa to 106 kPa

(d) Power voltage: –15 % to +10 % of nominal mains or test voltage

(e) Power frequency: Nominal frequency, f_{nom}

(f) Instrument tilt position: Up to 5 % or the maximum limit on the level indicator where one is present

(g) Battery voltage*: 9 V to 16 V (nominally 12 V), or 16 V to 32 V (nominally 24 V)

(*) These ranges apply to instruments used for commercial purposes while powered by a vehicle battery.

4.4.2 Disturbances

(a) AC mains voltage dips, short interruptions and voltage variations: reduction to 0 % (0.5 cycle), reduction to 0 % (1 cycle), reduction to 70 % (25 / 30[(1)] cycles), reduction to 0 % (250 / 300[(1)] cycles).

(b) Bursts (transients) on AC mains: Amplitude 1 kV, repetition rate 5 kHz

(c) Radiated radio-frequency fields, electromagnetic fields: 26[(2)] MHz to 2 GHz, 10 V/m

(d) Conducted radio-frequency fields: 0.15 MHz to 80[(3)] MHz, 10 V (e.m.f.)

(e) Electrostatic discharge – direct application: Up to 6 kV contact discharge

(f) Electrostatic discharge – indirect application: Up to 8 kV air discharge

(g) Storage temperature (extreme shipping conditions): –20 ºC to 55[(4)] ºC

(h) Random vibration[(5)]: total frequency range 10 Hz to 150 Hz, total RMS level 7 ms^{-2}, ASD level 10 Hz to 20 Hz: 1 m^2s^{-3}, ASD level 20 Hz to 150 Hz: 13 dB/octave

Notes:

(1) The cycle counts apply for 50 Hz / 60 Hz respectively.

(2) Testing from 80 MHz is permitted. Refer to R 146-2, A.6.3.

(3) Testing up to 26 MHz is permitted. Refer to R 146-2, A.6.4.

(4) The national responsible body may apply a lower maximum limit.

(5) Only applicable to portable instruments that are designed not to require reverification after transportation between different sites.

4.5 Maximum permissible error (MPE) and other accuracy requirements

4.5.1 Overview

Provided that the error on each P_{MB} measurement is less than the MPE in Table 4, column 3, an instrument with a P_{MB} calibration that has been approved for a particular grain type may be verified as sufficiently accurate under the presented conditions of use.

The error shift or fault shall not exceed the limit specified in either column 9 or column 10 of Table 4 during type evaluation tests. This result supports the presumption that a calibrated instrument can comply with the MPE over the rated operating ranges or in the event of a disturbance. In this Recommendation, error shifts and faults are primarily attributed to variations in the performance of the instrument hardware, not the calibration.

The calibration assessment and sample temperature sensitivity (STS) test shall be performed on every P_{MB} calibration submitted to ensure the calibrations are sufficiently accurate and robust. The P_{MB} errors shall be averaged over different samples of the grain type. This results in a single value for the following parameters that represents the bias or imprecision of the calibration over the legally relevant measurement range:

- $\bar{y}$, the bias of the mean P_{MB} over a set of samples spanning the P_{MB} measurement range;
- *SEP*, the standard deviation of the errors for the set of samples;
- averaged *SD*, the standard deviation of repeated measurements averaged over the set of samples;
- SDD_I, the standard deviation of differences in the mean P_{MB} from two units over the set of samples;
- average error shift, the error shifts calculated from the STS test, averaged over a set of high or low moisture content samples in the P_{MB} measurement range.

Refer to Table 4 for the limits on the values calculated for $\bar{y}$, *SEP*, averaged *SD*, SDD_I, and the STS average error shift.

4.5.2 Accuracy requirements for various types of grain – specification

The MPE and other requirements (e.g. accuracy) for measurements of protein content in various grain types are presented in Table 4. These are recommended values that apply to measurements of P_{MB} including the protein content at dry basis (i.e. $P_{0\%}$). They apply to all instruments used in commercial transactions, irrespective of their principles of operation.

Note: The same MPE is applied across all test samples of the same grain type regardless of the P_{MB} level.

4.5.3 Conversion of dry basis MPE and limits for measurements at other M_B

The dry basis MPEs in Table 4 shall be appropriately scaled down when the indicated protein content is not at dry matter basis, i.e. the specified M_B is greater than zero. The MPE at any $M_B > 0$ % can be calculated from the relevant values of $MPE_{0\%}$ using Equation 2:

$$MPE_{MB>0\%} = MPE_{0\%} \times (1 - \frac{M_B}{100}) \qquad \textbf{Equation 2}$$

where: M_B = basis moisture content for the type of grain.

The limiting values for *SEP*, averaged *SD*, SDD_I, error shift and fault in Table 4 are scaled accordingly for the relevant M_B.

For type evaluation, the adjusted values for the MPE shall be rounded to two decimal places, e.g. 0.275 becomes 0.28 and –0.275 becomes –0.28. Adjusted values for the MPE at verification shall be rounded to one decimal place, e.g. 0.356 becomes 0.4 and –0.356 becomes –0.4.

4.5.4 Reference method – specification

The errors during verification and the values of $\bar{y}$ and *SEP* used to assess the calibration accuracy at type evaluation are calculated with reference to certified reference materials (CRMs), i.e. whole-grain measurement standards with certified P_{MB} values.

The national responsible body shall define the reference method for calculating the protein content of grain applicable to the certification of whole-grain reference materials (RM). Where possible, methods based on international standards such as ISO publications shall be used (see Annex A). Additional guidance on the production and handling of whole-grain RMs and CRMs are included in Annex B.

Table 4: MPE and other accuracy requirements expressed in percentage protein by mass (%) at dry basis or M_B[(1)(3)]

Test sample		Field tests	Type evaluation tests						
Grain type[(2)]	M_B	Verification	Calibration assessment (instrument at reference conditions)					Reproducibility assessment	
		Rated operating condition	Accuracy		Repeatability	Reproducibility (two units)	Sample temp sensitivity (STS)	Influence factor rated operating condition	Disturbance
		MPE (%)	Max $\bar{y}$ (%)	Max *SEP* (%)	Max averaged *SD* (%)	Max SDD_I (%)	Max average error shift (%)	Max error shift (%)	Fault limit (%)
column 1	column 2	column 3	column 4	column 5	column 6	column 7	column 8	column 9	column 10
Wheat	0 %	±0.4	±0.34	Absolute value of col 4	Absolute value of col 4 × 0.5	Absolute value of col 4 × 0.6	col 4	col 4 × 0.7	col 4
	M_B > 0 %	$\pm 0.4 \times (1 - \frac{M_B}{100})$	$\pm 0.34 \times (1 - \frac{M_B}{100})$						
Barley	0 %	±0.5	±0.40						
	M_B > 0 %	$\pm 0.5 \times (1 - \frac{M_B}{100})$	$\pm 0.40 \times (1 - \frac{M_B}{100})$						
Rice	0 %	±0.6	±0.50						
	M_B > 0 %	$\pm 0.6 \times (1 - \frac{M_B}{100})$	$\pm 0.50 \times (1 - \frac{M_B}{100})$						
Corn	0 %	±0.8	±0.50						
	M_B > 0 %	$\pm 0.8 \times (1 - \frac{M_B}{100})$	$\pm 0.50 \times (1 - \frac{M_B}{100})$						
Soybean	0 %	±0.8	±0.63						
	M_B > 0 %	$\pm 0.8 \times (1 - \frac{M_B}{100})$	$\pm 0.63 \times (1 - \frac{M_B}{100})$						
Lupins	0 %	±1.2	±1.0						
	M_B > 0 %	$\pm 1.2 \times (1 - \frac{M_B}{100})$	$\pm 1.0 \times (1 - \frac{M_B}{100})$						

Notes:

(1) Refer to 4.5.3 for instructions on rounding calculated values and the equation converting the MPE/ limits at dry basis to another M_B.

(2) Recommended MPEs for other grain types (e.g. triticale and rye) can be added in future revisions of this Recommendation.

(3) The limits in Table 4 apply to values (e.g. $\bar{y}$, *SEP*, averaged *SD*, SDD_l, error shift, fault) that are calculated according to *Test result* in R 146-2, Annex A.

4.6 MPE at verification

Instruments shall be designed, manufactured and used with appropriate calibrations so that during verification, errors do not exceed the value for the MPE shown in Table 4, column 3.

In-field surveillance is under the control of the national responsible body. In-field conditions are represented by the rated operating conditions specified in 4.4. The instrument in service shall not be operated while exposed to influence quantities outside the ranges applied during type evaluation.

4.7 Requirements for calibrations

4.7.1 Calibration accuracy and precision requirements at reference conditions

Under the reference test conditions specified in R 146-2, Annex A, each calibration submitted for approval with the type of protein measuring instrument shall be statistically tested for accuracy with a set of whole-grain CRMs. Each set shall encompass the legally relevant P_{MB} range and represent all the varieties of grain in the scope of the calibration under test.

Instruments shall be designed, manufactured and used with appropriate calibrations so that the calculated measures of bias and imprecision, i.e. $\bar{y}$, *SEP*, averaged *SD* and SDD_{I}, do not exceed the limits in Table 4.

4.7.2 Limited sample temperature sensitivity (STS)

Each calibration submitted for approval with the type of instrument shall be tested for STS with high and low moisture samples at the $\Delta t_{\max}$ specified by the national responsible body or the manufacturer (see 4.3.2).

Instruments shall be designed, manufactured and used with appropriate calibrations so that the average error shift for a set of samples does not exceed the limit shown in Table 4, column 8.

4.8 Error due to variations in influence quantities

4.8.1 Variation of selected influence factor(s) within the rated operating ranges

Instruments shall be designed and manufactured so that all functions continue to operate as designed and the error shift does not exceed the limit in Table 4, column 9 when selected influence factors are varied within the rated operating ranges shown in 4.4.1.

4.8.2 Effect of disturbances on instruments

In the event of disturbances as severe as those specified in 4.4.2, significant faults as defined in 2.2.16 shall either not occur or shall be detected and acted upon by means of checking facilities as described in 5.1.

4.9 Error due to changes in the instrument over time

Changes within the instrument over time shall not compromise the measurement accuracy.

4.9.1 The error shift on measurements taken immediately after the instrument is switched on shall be within the limit in Table 4, column 9 where no warm-up time is specified. If a warm-up time is specified, the error shift on measurements taken after this warm-up time shall be within the limit.

4.9.2 Any error shift due to instrumental drift over a period of at least four weeks shall remain stable (i.e. not exceed the limit shown in Table 4, column 9).

5 Technical requirements

5.1 Checking facilities

5.1.1 Suppression of P_{MB} measured values in the event of a significant fault

A protein measuring instrument shall automatically prevent further measurements and clearly indicate when a significant fault has occurred by an appropriate error message, unambiguous warning or blanking the display.

5.1.2 Suppression of P_{MB} measured values outside of operating ranges

5.1.2.1 A protein measuring instrument shall automatically and clearly indicate when one of the following type-approved operating ranges is exceeded, by an appropriate error message, unambiguous warning or blanking the display:

(a) instrument environmental temperature range, t_C to t_H (see 4.2);

(b) sample temperature range, $t_{C,sample}$ to $t_{H,sample}$, for each grain type (see 4.3.1);

(c) maximum sample and instrument temperature differential (Δt_{max}) for each grain type (see 4.3.2).

5.1.2.2 The instrument shall automatically prevent further measurements as long as the respective influence factor or sample characteristic remains outside the type-approved ranges.

5.1.2.3 The operator shall not be required to judge the precise ambient temperature and the temperature of the sample in order to make an accurate measurement.

5.1.3 Suppression of P_{MB} values or warnings outside of the approved measuring range

P_{MB} measured values that are outside of the type approved range for the calibration (see 4) shall be suppressed unless they are accompanied by an appropriate error message or unambiguous warning.

5.1.4 Instrument warm-up period

When a protein measuring instrument is turned on, it shall not display or record any measured values until the operating temperature necessary for accurate measurement has been attained. This requirement may not be necessary for instruments which do not require any warm-up time.

5.2 Manufacturer's manual

The manufacturer shall provide with each protein measuring instrument, a manual that describes the installation, operation, and routine maintenance of the instrument and its accessories. In addition, the manual shall include the following information:

(a) name and address of the manufacturer;

(b) type or pattern of the instrument with which it is intended to be used;

(c) date of issue;

(d) type or varieties of grain for which the instrument is designed to be used within the scope of national requirements;

(e) limitations of use, including, but not confined to the P_{MB} measurement range(s), grain sample temperature, maximum allowable temperature difference between grain sample and instrument, instrument operating temperature range, voltage and frequency ranges, electromagnetic interferences and electromagnetic compatibility.

This manual shall be supplied to the owner/user of the instrument in the official language(s) of the countries in which it is used or in a language accepted by the national responsible body.

5.3 Markings

5.3.1 General markings

Instruments shall be clearly and permanently marked for the purpose of identification with the following:

(a) manufacturer's name or mark;

(b) model designation;

(c) serial number given by the manufacturer; and

(d) approval marking of the national responsible body, if the instrument is approved.

5.3.2 Location of markings

Markings shall be grouped together in a clearly visible location, either on a permanently attached nameplate or on part of the instrument. The required information shall be readily observable without disassembly.

5.3.3 Marking operational controls, indications and features

All operational controls, indications, indicating switches, features, light displays and push buttons shall be clearly identifiable. Keys that are visible only to the operator need only be marked to the extent that a trained operator can understand the function of each key.

5.4 Sample input and calibration selection

5.4.1 Selection of calibration on the instrument

5.4.1.1 On protein measuring instruments that have a different calibration for each grain type, it shall be possible to select the calibration applicable to the test sample, for example, via a user menu listing the grain types that the instrument is approved to measure.

5.4.1.2 To prevent misuse, the calibration selected via the user interface shall be unambiguous and visible to all parties present, i.e. the displayed calibration name shall correspond with the grain type to be analyzed.

5.4.2 Sampling and minimum sample size

5.4.2.1 The operator shall not be required to judge the precise volume or weight required by the instrument to make an accurate P_{MB} measurement.

5.4.2.2 The minimum allowable sample size for measurement of P_{MB} shall be 100 g or 400 kernels or seeds whichever is smaller, except where the national responsible body determines otherwise.

5.4.2.3 The national responsible body shall specify minimum guidelines for the sampling of bulk or packed cereals for testing. These may be based on voluntary international standards (e.g. ISO 24333:2009 *Cereals and cereal products – Sampling* [6]).

5.5 Instrument construction

5.5.1 Grain protein measuring instruments and all accessory equipment shall be of such materials, design and construction as to make it probable that, under normal service conditions

(a) accuracy will be maintained,

(b) operating parts will continue to function as intended, and

(c) adjustments will remain secure and stable.

5.5.2 Undue stresses, deflections or distortions of parts shall not occur to the extent that accuracy is detrimentally affected.

5.5.3 The housing shall be constructed so that the main components of the instrument are protected from dust and moisture.

5.5.4 When the process of measurement requires the use of a grinding mill, the mill shall be considered an integral part of the measurement process. The appropriate mill type shall be designated by the instrument manufacturer. A milling unit shall accompany the submitted instrument so its suitability for the measurement process may be assessed during type evaluation tests.

5.6 Level indicating means

5.6.1 If tilting the instrument in any upright direction by up to 5 % (approximately 3º) reduces the accuracy of the instrument, it shall be equipped by a level indicator and level adjustment means to reduce the likelihood that it will be tilted when placed in service.

5.6.2 The level indicating means shall be readable without any disassembly of the instrument.

5.7 Presentation of the measured value

5.7.1 Grain protein measuring instruments shall be equipped with a digital indicating element which shall not display any protein content values before the end of the measurement cycle.

5.7.2 Measurement results shall be displayed as percent protein by mass (%) at the M_B. Subdivisions of this unit shall be in terms of decimal subdivisions (not fractions).

5.7.3 The display shall permit protein content value determination to a resolution of at least 0.1 % P_{MB}. The 0.1 % P_{MB} resolution is for commercial transactions; the display on sample instruments submitted for type evaluation shall permit 0.01 % P_{MB} resolution.

5.7.4 On multi-constituent instruments (e.g. instruments which also measure grain moisture content), provision shall be made for displaying and recording the constituent label to make it clear which constituent is associated with each of the displayed values.

5.7.5 The minimum height for the digits used to display protein content shall be 10 mm. Numbers and symbols of units shall be presented in accordance with OIML D 2:2007 [7].

6 Requirements for software-controlled devices and security

The software requirements are based on OIML D 31:2008 [5].

The risk associated with the software used in protein measuring instruments is level I. Validation in accordance with Procedure A in 6.3 and 6.4 of OIML D 31:2008 is adequate for solutions implemented to fulfil requirements at the normal severity level.

Commercial application of P_{MB} measured values typically occurs at the same time and place as the measurement.

6.1 Specification of software requirements

6.1.1 For instruments and modules operated by software, the manufacturer shall describe or declare how the software is implemented within the instrument or module, i.e. if it is installed in a fixed hardware and software environment (embedded) or on a universal computer system (implemented into the housing or external).

6.1.2 Legally relevant software shall be clearly identifiable via a unique software version or a checksum. In the normal operation mode of the instrument, the software version or the checksum shall be displayed or printed out on command or shall be displayed during the start-up procedure of the instrument.

6.1.3 Legally relevant measuring algorithms and functions shall be appropriate and functionally correct as evidenced by the instrument correctly displaying and recording the measurement result and the required accompanying information. It shall be possible to validate algorithms and functions where required by metrological tests.

6.1.4 The conformity of the legally relevant software used in each instrument to that in the approved type shall be at level (b) described in OIML D 31:2008, 5.2.5. In types where selected functions or parts of the source code can be modified, it shall be possible to detect software variations, e.g. via checksum values.

6.1.5 Further measurements shall not be possible when a significant fault is detected.

6.1.6 If the software of the instrument is separated into legally relevant and non-relevant parts, the requirements of OIML D 31:2008, 5.2.1.2 shall be fulfilled.

6.1.7 For instruments/measuring systems using an internal or external universal computer, the legally relevant software shall be operated only in the environment specified to ensure appropriate functioning. If necessary to secure the functioning of the legally relevant software, the operating system shall be fixed to a defined invariant configuration.

Note: A fixed environment for software is also required for instruments where cryptographic data protection is implemented or when software changes on a verified instrument are permitted without an appointed verifier onsite (i.e. the 'Traced Updates' described in OIML D 31:2008, 5.2.6.3).

6.1.8 The national responsible body may require instruments to be equipped with an internal recording element and/or a communication interface that permits interfacing with an external recording element, for example a printer. In this case, correspondence between the displayed information and the remote recording element shall be verified.

6.1.9 The national responsible body may apply the requirements in 6.2 and 6.3, if measurement data has to leave the measuring instrument and be stored or transmitted in an insecure environment before it is used for commercial purposes.

6.2 Data storage

6.2.1 If storage of legally relevant data is required by the national responsible body, the measurement data must be stored automatically when the measurement is finished.

Note: A recording element shall not record any protein content values before the end of the measurement cycle.

The storage device must have sufficient permanency to ensure that the data are not corrupted under normal storage conditions and there shall be sufficient memory storage for any particular application.

6.2.2 The measurement value stored shall be accompanied by all relevant information necessary for future legally relevant use. The measurement records shall include as a minimum: unambiguous identifier of the measurement, measurement date and time, unique identification of the instrument,

grain type, P_{MB} results and units, calibration version identification, error messages and constituent labels (on multi-constituent meters). Acceptable examples of a measurement identifier include consecutive numbers enabling assignment to values printed on an invoice, or a test sample ID.

6.3 Data transmission

Where the transmission of legally relevant measurement data in open networks presents opportunities for fraud or misuse with serious consequences for an important market of the country, the additional requirements below may be applied.

6.3.1 The data shall be protected by software means described in OIML D 31:2008, 5.2.3.2 to guarantee the authenticity and integrity.

6.3.2 If cryptographic protection of data as indicated in OIML D 31:2008, 5.2.3.3 is employed to achieve protection at the severity II level, Procedure B (Table 2 in 6.4 of OIML D 31:2008) methods are recommended for validating this aspect of the software.

6.3.3 The measurement shall not be inadmissibly influenced by a transmission delay.

6.3.4 If a transmission interruption occurs because the network services become unavailable, no measurement data shall be lost. The measurement process should be stopped to avoid the loss of measurement data.

6.4 Provision for software and calibration security

6.4.1 Sealing

Provision shall be made for appropriate sealing by mechanical, electronic and/or cryptographic means, in order to make any change that affects the metrological integrity of the instrument impossible or evident. Calibrations, zero-setting and test point adjustments are considered to affect metrological characteristics and must therefore be sealed.

Refer to Annex E which contains practical guidance for sealing protein measuring instruments, including consideration of sealable parameters and details of sealing mechanisms, e.g. metrological audit trails.

6.4.2 Safeguards against fraudulent use

For protection against fraudulent use, the requirements below shall be fulfilled.

6.4.2.1 The legally relevant software shall be secured against unauthorized modification, loading or changes by swapping of the memory device. In addition to mechanical sealing, technical means may be necessary to secure measuring instruments that have an operating system or an option to load software.

6.4.2.2 Only clearly documented functions are allowed to be activated by the user interface, which shall be realized in such a way that they do not facilitate fraudulent use.

6.4.2.3 Parameters that fix the legally relevant characteristics of the measuring instrument shall be secured against unauthorized modification. It shall be possible to display or print the current parameter settings.

6.4.2.4 National responsible bodies may restrict access to any of the device-specific parameters.

Note: Device-specific parameters may be adjustable or selectable only in a special operational mode of the instrument. Type-specific parameters have identical values for all specimens of a type and are fixed at type approval.

6.4.2.5 The national responsible body may require protein measuring instruments in service to be positioned such that all interested parties present have the possibility of seeing all the measurement operations, not limited to the indicating or recording device(s).

6.5 Software documentation

In addition to the requirements in R 146-2, 1.1.2 the manufacturer shall submit the software documentation described in

Table 5.

Table 5: Examples of software documentation and application notes

Documentation	Application notes and/or examples
Description of the legally relevant software, incorporating how the requirements are met	
Description of the operating system security	For e.g. password protection
Description of the software sealing method(s)	
Overview of the system hardware, highlighting any hardware components that are deemed legally relevant or performing legally relevant functions	For e.g. topology block diagram, type of computer(s), type of network
Description of the accuracy of the algorithms	Example algorithms – filtering of A/D conversion results, rounding algorithms
Declaration of the hardware and software environment, including minimum resources and configuration necessary for correct functioning of the instrument	Applicable for types of instrument requiring a universal computer
Description of the user interface, menus and dialogues	
Description of the software identification which has to be clearly assigned to the legally relevant functions	If applicable, include a description of all encryption means
Clear instructions on how to check the actual software identification against the reference number as listed in the type approval certificate	This reference may be additionally marked on or displayed by the instrument
List of commands of each hardware interface of the measuring instrument/electronic device/sub-assembly	Include a statement of completeness
List of durability errors that are detected by the software, e.g. for a spectrometer – an alert for the user to clean lenses/windows or to replace LED when radiation intensities fall below threshold values.	If necessary, include a description of the detecting algorithms
Description of data sets stored or transmitted	
List of significant faults that are detected and a description of the detecting algorithm	Applicable where fault detection is achieved by software means
Operating manual which clearly identifies all operational controls, indications, and features	Example features – switches, lights, displays and push buttons

Annex A

Protein content calculation from nitrogen determination

(Mandatory)

The national responsible body shall specify the relationship between the measured nitrogen content and protein content assigned to whole-grain reference materials used to test protein measuring instruments.

The moisture content measurements that are used to adjust the calculated protein content to the relevant M_B shall meet the requirements of OIML R 59 or international reference methods therein.

Two examples are given below:

A.1 Dumas combustion – total nitrogen determination

ISO/DTS 16634.2 (2014) Food products – Determination of the total nitrogen content by combustion according to the Dumas principle and calculation of the crude protein content – Part 2: Cereals, pulses and milled cereal products. [11]

Note: Dumas (combustion) nitrogen values may be greater than the corresponding Kjeldahl values, particularly at higher nitrogen levels.

A.2 Improved Kjeldahl method — total nitrogen determination

For example: ISO 20483 (2013) Determination of the nitrogen content and calculation of the crude protein content – Kjeldahl method [12]

Annex B

Whole-grain measurement standards

(Mandatory)

B.1 Whole-grain reference material (RM) certified for P_{MB}

Whole-grain RMs with certified P_{MB} values (i.e. CRMs for P_{MB}) shall be used to provide reference quantity values during verification of protein measuring instruments and in type evaluation tests concerned with the accuracy of P_{MB} calibrations at reference conditions. These shall be sufficiently homogenous, moisture-stable samples representative of the grain traded in the region with P_{MB} values certified using a reference test method.

B.1.1 Reference method traceability

The national responsible body shall specify the reference method, according to the options listed in Annex A, for assigning a P_{MB} value to each whole-grain RM.

Essentially, a reference method allows the P_{MB} of a sample to be inferred from direct measurement of the nitrogen mass fraction in the sample and a direct measurement of the moisture content. The procedure applied shall contain provisions for verifying the calibration of the reference method using a nationally recognized measurement standard for nitrogen (i.e. a CRM for nitrogen concentration).

Note: Systematic errors in the execution of the reference method may be reduced by having traceability to the results of a collaborative survey of several reference method laboratories (i.e. an interlaboratory test). Refer to ISO/TS 16634-2 Annex E [11] for example results.

B.1.2 Suitability of the whole-grain CRM for use in verification

Third party laboratory accreditation of the reference method and sampling systems may be pursued to ensure that the whole-grain CRMs generated are adequate for determining the error during verification of protein measuring instruments.

The size of grain sample analyzed by the reference method and a protein measuring instrument may differ and the resulting whole-grain CRMs are not always used on a protein measuring instrument immediately. It is therefore important to consider the level of spatial inhomogeneity and any compositional variations over time when assigning a P_{MB} value to an RM and evaluating the uncertainty.

Refer to ISO Guide 35 *General and statistical principles for certification* [13] for further guidance on development of valid methods to traceably assign values to the properties of reference materials (RMs) and evaluation of the associated uncertainty.

To produce a CRM, the grain protein reference method shall be applied with sufficient repetitions of the complete measurement cycle, on representative portions of the whole-grain RM. The expanded uncertainty of the mean P_{MB} assigned to the whole-grain CRM, calculated with a coverage factor of two, should ideally be within a third of the MPE for the grain type.

B.1.3 Suitability of the whole-grain CRM for assessment of calibrations at reference conditions

SEP is the standard deviation of at least 30 measurement errors from different CRMs. A limit for the uncertainty associated with certified P_{MB} values in the calculation of *SEP* is difficult to specify compared to the certified P_{MB} values at verification, which is concerned with the measurement errors on individual CRMs.

However, poor reproducibility in the reference method and/or a lack of consideration for the level of inhomogeneity can result in enlarged *SEP* values that can be wrongly attributed to the calibrations under test. It is recommended that the expanded uncertainty associated with the P_{MB} value of any

CRM used in the assessment of calibrations is limited to one third of the value in 4.5, Table 4, column 3.

Note: The values calculated for averaged *SD,* and SDD_I are not dependent on certified P_{MB} values.

B.2 Practical instructions for test samples

B.2.1 Source

The characteristics of the standards (reference materials) used as test samples shall be representative of the grain being traded in the region. This is particularly important for the assessment of calibrations in R 146-2, A.7. Foreign produce, i.e. samples based on the grain harvested in another country or region, may not be suitable for the assessment of calibrations due to climatic and crop variability.

B.2.2 Moisture content

Unless dried or moistened grain is commonly traded, all test samples shall be naturally occurring grain, i.e. the moisture should not be adjusted by soaking or spraying the sample with water or by extended exposure to high humidity air. Before storing a sample for any length of time, ensure that the moisture level does not make it susceptible to mold, which can occur at relatively low levels for certain types of grain e.g. over 13 % moisture for wheat.

B.2.3 Sample records

The sample records should include the identification number assigned, the date received, source, grain type, protein content, moisture, and other pertinent information.

B.2.4 Sample handling and storage

Upon receipt the integrity of the moisture-tight sample enclosure should be checked and a new enclosure used if necessary. Most grain samples are to be stored at 2 °C to 8 °C prior to use.

Prior to testing, samples are removed from cold storage and equilibrated to room temperature.

Note: Except during analysis, a test sample is returned to its enclosure during the performance tests.

B.2.5 Sample cleaning

The sample must be visibly free from insects, foreign seeds and any other foreign material. The condition of the sample (odor, appearance, damage, etc.) is recorded on the sample record. Spatial inhomogeneity in a bulk sample is minimized as much as possible by mixing.

B.2.6 Sample size

Unless the certificate of analysis permits otherwise, the entire CRM shall be analyzed. Where RMs are permitted in a test, the cleaned bulk sample that has been mixed must be divided into representative portions slightly in excess of the amounts needed for analysis using the protein measuring instrument or where necessary, the reference method.

Annex C

Bibliography

(Informative)

1 ISO/IEC Guide 99; OIML V 2-200:2012 *International Vocabulary of Metrology – Basic and General Concepts and Associated Terms* (VIM)

An international agreement on terminology, prepared as a collaborative work of experts appointed by the BIPM, IEC, IFCC, ISO, IUPAC, IUPAP and OIML. This Vocabulary covers subjects relating to measurement and includes information on the determination of physical constants and other fundamental properties of materials and substances.

2 OIML V 1:2012 *International Vocabulary of Terms in Legal Metrology* (VIML)

3 OIML D 18:2008 *The use of certified reference materials in fields covered by metrological control exercised by national services of legal metrology. Basic principles*

4 OIML D 11:2013 *General requirements for electronic measuring instruments*

The primary aim of this International Document is to provide OIML Technical Committees and Subcommittees with guidance for establishing appropriate metrological performance testing requirements for influence quantities that may affect the measuring instruments covered by International Recommendations.

5 OIML D 31:2008 *General requirements for software controlled measuring instruments*

Specifies the general requirements applicable to software-related functionality in measuring instruments and gives guidance for verifying the compliance of an instrument with these requirements.

6 ISO 24333:2009 *Cereals and milled cereals – Sampling*

Specifies requirements for the dynamic or static sampling, by manual or mechanical means, of cereals and cereal products, for assessment of their quality and condition.

7 OIML D 2:2007 *Legal units of measurement*

8 OIML D 16:2011 *Principles of assurance of metrological control*

9 OIML D 20:1998 *Initial and subsequent verification of instruments and processes*

10 OIML D 9:2004 *Principles of metrological supervision*

11 ISO/TS 16634-2:2014 *Food products – Determination of the total nitrogen content by combustion according to the Dumas principle and calculation of the crude protein content – Part 2: Cereals, pulses and milled cereal products*.

12 ISO 24083:2013 *Determination of the nitrogen content and calculation of the crude protein content – Kjeldahl method*

Specifies a method for the determination of the nitrogen content of cereals, pulses and derived products, according to the Kjeldahl method, and a method for calculating the crude protein content. The method does not distinguish between protein nitrogen and non-protein nitrogen.

13 ISO Guide 35:2006 *General and statistical principles for certification*

Statistical principles to assist in the understanding and development of valid methods to assign values to properties of a reference material, including the evaluation of their associated uncertainty, and establish their metrological traceability. Reference materials (RMs) that undergo all steps described in ISO Guide 35:2006 are usually accompanied by a certificate and called a certified reference material (CRM).

14 IEC 60068-2-1:1990-05 with Amendment 1:1993-02 and Amendment 2:1994-06 *Environmental testing, Part 2: Tests, Test A: Cold*

Concerns cold tests on both non-heat-dissipating and heat dissipating specimens.

15 IEC 60068-3-1:1974-01 + Supplement A:1978-01 *Environmental testing Part 3 Background information, Section 1: Cold and dry heat tests*

Gives background information for Tests A: Cold (IEC 68-2- 1), and Tests B: Dry heat (IEC 68-2-2).

16 IEC 60068-2-2:1974-01, with Amendment 1:1993-02 and Amendment 2:1994-05 *Environmental testing Part 2: Tests. Test B: Dry heat*

Contains Test Ba: Dry heat for non-heat-dissipating specimen with sudden change of temperature; Test Bb: Dry heat for non-heat-dissipating specimen with gradual change of temperature; Test Bc: Dry heat for heat-dissipating specimen with sudden change of temperature; Test Bd: Dry heat for heat-dissipating specimen with gradual change of temperature.

17 IEC 60068-2-78:2001-08 *Environmental testing – Part 2-78: Tests -Test Cab: Damp heat, steady state*

(IEC 60068-2-78 replaces the following withdrawn standards: IEC 60068-2-3, test Ca and IEC 60068-2-56, test Cb)

Provides a test method for determining the suitability of electrotechnical products, components or equipment for transportation, storage and use under conditions of high humidity. The test is primarily intended to permit the observation of the effect of high humidity at constant temperature without condensation on the specimen over a prescribed period.

18 IEC 60068-3-4:2001-08 *Environmental testing – Part 3-4: Supporting documentation and guidance – Damp heat tests*

Provides the necessary information to assist in preparing relevant specifications, such as standards for components or equipment, in order to select appropriate tests and test severities for specific products and, in some cases, specific types of application.

19 IEC/TR 61000-2-1:1990-05 *Electromagnetic compatibility (EMC) Part 2: Environment Section 1: Description of the environment – Electromagnetic environment for low frequency conducted disturbances and signaling in public power supply systems*

Has the status of a technical report, and gives information on the various types of disturbances that can be expected on public power supply systems.

20 IEC 61000-4-1:2000-04 Basic EMC Publication *Electromagnetic compatibility (EMC) Part 4: Testing and measurement techniques Section 1: Overview of IEC 61000-4 series*

Gives applicability assistance to the users and manufacturers of electrical and electronic equipment on EMC standards within the IEC 61000-4 series on testing and measurement techniques. Provides general recommendations concerning the choice of relevant tests.

21 ISO 16750-2:2006 *Road vehicles – Environmental conditions and testing for electrical and electronic equipment Part 2: Electrical loads*

Specifies electrical loads and corresponding tests and requirements for the mounting of electric and electronic systems and components on road vehicles. It is applicable to environmental conditions and tests affecting electrical and electronic equipment mounted directly on or in the vehicle. It does not cover electromagnetic compatibility (EMC).

22 IEC 61000-4-11:2004-03 *Electromagnetic compatibility (EMC) – Part 4-11: Testing and measuring techniques - Voltage dips, short interruptions and voltage variations immunity tests*

Defines the immunity test methods and range of preferred test levels for electrical and electronic equipment connected to low-voltage power supply networks for voltage dips, short interruptions, and voltage variations.

23 IEC 61000-6-1:1997-07 *Electromagnetic compatibility (EMC) – Part 6: Generic standards – Section 1: Immunity for residential, commercial and light-industrial environments*

Defines the immunity test requirements in relation to continuous and transient, conducted and radiated disturbances, including electrostatic discharges, for electrical and electronic apparatus intended for use in residential, commercial and light-industrial environment, and for which no dedicated product or product-family standard exists.

24 IEC 61000-6-2:1999-01 *Electromagnetic compatibility (EMC) – Part 6-2: Generic standards – Immunity for industrial environments*

Applies to electrical and electronic apparatus intended for use in industrial environments, for which no dedicated product or product-family immunity standard exists. Immunity requirements in the frequency range 0 Hz to 400 GHz are covered, in relation to continuous and transient, conducted and radiated disturbances, including electrostatic discharges. Test requirements are specified for each port considered.

25 IEC 61000-4-4:2004-07 *Electromagnetic compatibility (EMC) Part 4-4: Testing and measurement techniques – Electrical fast transient/burst immunity test*

Establishes a common and reproducible reference for evaluating the immunity of electrical and electronic equipment when subjected to electrical fast transient/burst on supply, signal, control and earth ports. The test method documented in this part of IEC 61000-4 describes a consistent method to assess the immunity of an equipment or system against a defined phenomenon.

26 IEC 61000-4-3 consolidated Edition 2.1:2002-09 with amendment 1 (2002-08) *Electromagnetic compatibility (EMC) Part 4: Testing and measurement Techniques Section 3: Radiated, radio-frequency, electromagnetic field immunity test*

Applies to the immunity of electrical and electronic equipment to radiated electromagnetic energy. Establishes test levels and the required test procedures. Establishes a common reference for evaluating the performance of electrical and electronic equipment when subjected to radio-frequency electromagnetic fields.

27 IEC 61000-4-6 (2003-05) with amendment 1:2004-10 *Electromagnetic compatibility (EMC) Part 4: Testing and measurement techniques Section 6: Immunity to conducted disturbances, induced by radio-frequency fields*

Relates to the conducted immunity requirements of electrical and electronic equipment to electromagnetic disturbances coming from intended radio-frequency (RF) transmitters in the frequency range 9 kHz up to 80 MHz. Equipment not having at least one conducting cable (such as mains supply, signal line or earth connection), which can couple the equipment to the disturbing RF fields is excluded.

28 IEC 61000-4-2 (1995-01) with amendment 1:1998-01 and amendment 2 (2000-11) *Basic EMC Publication Electromagnetic compatibility (EMC) Part 4: Testing and measurement techniques Section 2: Electrostatic discharge immunity test.*

Consolidated Edition: IEC 61000-4-2 (2001-04) Ed. 1.2

This publication is based on IEC 60801-2 (second ed: 1991). It relates to the immunity requirements and test methods for electrical and electronic equipment subjected to static electricity discharges, from operators directly, and to adjacent objects.

29 IEC 60068-2-47 Ed 3.0:2005-4 *Environmental testing Part 2-47: Tests mounting of specimens for vibration, impact and similar dynamic tests*

Provides methods of mounting components, and mounting requirements for equipment and other articles, for the families of dynamic tests in IEC 60068-2, that is impact (Test E), vibration (Test F) and acceleration, steady-state (Test G).

30 IEC 60068-2-64 Ed 2.0:2008-04 *Environmental testing – Part 2-64: Test methods, Test Fh: Vibration, broad-band random and guidance*

Determines the adequacy of specimens to resist dynamic loads without unacceptable degradation of its functional and/or structural integrity when subjected to the specified random vibration test requirements.

31 IEC 60068-3-8 Ed. 1.0:2003-08 *Environmental testing – Part 3-8: Supporting documentation and guidance - Selecting amongst vibration tests*

Provides guidance for selecting amongst the IEC 60068-2 stationary vibration test methods Fc sinusoidal, Fh random and F(x) Mixed mode vibration. The different steady-state test methods and their aims are briefly described in clause 4. Transient test methods are not included.

32 WELMEC Guide 7.2, March 2012 Issue 5 *Software Guide (Measuring Instruments Directive 2004/22/EC)*

This Guide provides guidance to all those concerned with the application of the Measuring Instruments Directive (European Directive 2004/22/EC; MID), especially for software-equipped measuring instruments. It addresses both manufacturers of measuring instruments and notified bodies which are responsible for conformity assessment of MID instruments. By following the Guide, compliance with the software-related requirements contained in the MID can be assumed.

Annex D

General metrology & legal metrology terms

(Informative)

D.1 VIM definitions

D.1.1 accuracy; measurement accuracy [VIM 2.13]

closeness of agreement between a measured quantity value and a true quantity value of the measurand

Notes

1 The concept of 'measurement accuracy' is not a quantity and is not given a numerical quantity value. A measurement is said to be more accurate when it offers a smaller measurement error.

2 The term "measurement accuracy" should not be used as a synonym for "measurement trueness" and the term "measurement precision" should not be used as a synonym for "measurement accuracy", which, however, is related to both concepts.

3 'Measurement accuracy' is sometimes understood as closeness of agreement between measured quantity values that are being attributed to the measurand.

D.1.2 adjustment [further information in VIM 3.11]

set of operations carried out on a measuring system so that it provides prescribed indications corresponding to given values of a quantity to be measured

D.1.3 calibration [VIM 2.39]

operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication

Notes

1 A calibration may be expressed by a statement, calibration function, calibration diagram, calibration curve, or calibration table. In some cases, it may consist of an additive or multiplicative correction of the indication with associated measurement uncertainty.

2 Calibration should not be confused with adjustment of a measuring system, often mistakenly called "self-calibration", nor with verification of calibration.

3 Often, the first step alone in the above definition is perceived as being calibration.

D.1.4 certified reference material; CRM [further information in VIM 5.14 and OIML D 18]

reference material, accompanied by documentation issued by an authoritative body and providing one or more specified property values with associated uncertainties and traceabilities, using valid procedures

D.1.5 maximum permissible error (MPE); limit of error [further information in VIM 4.26]

extreme value of measurement error, with respect to a known reference quantity value, permitted by specifications or regulations for a given measurement, measuring instrument, or measuring system

D.1.6 measurement error; error [further information in VIM 2.16]

measured quantity value minus a reference quantity value

D.1.7 measured quantity value; measured value [further information in VIM 2.10]

quantity value representing a measurement result

D.1.8 rated operating condition [VIM 4.9]

operating condition that must be fulfilled during measurement in order that a measuring instrument or measuring system performs as designed

Note: Rated operating conditions generally specify intervals of values for a quantity being measured and for any influence quantity.

D.1.9 reference condition [VIM 4.11]

operating condition prescribed for evaluating the performance of a measuring instrument or measuring system or for comparison of measurement results

Notes

1 Reference conditions specify intervals of values of the measurand and influence quantities.

2 In IEC 60050-300, item 311-06-02, the term "reference condition" refers to an operating condition under which the specified instrumental measurement uncertainty is the smallest possible.

D.1.10 reference material; RM [further information in VIM 5.13]

material, 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

D.1.11 reference quantity value; reference value [further information in VIM 5.18]

quantity value used as a basis for comparison with values of quantities of the same kind

D.1.12 repeatability; measurement repeatability [VIM 2.21]

measurement precision under a set of repeatability conditions of measurement

D.1.13 repeatability condition of measurement [VIM 2.20]

condition of measurement in a set of conditions including the same measurement procedure, same operator, same measuring system, same operating conditions, same location and replicate measurements over a short period of time

D.1.14 reproducibility; measurement reproducibility [VIM 2.25]

measurement precision under reproducibility conditions of measurement. Relevant statistical terms are given in ISO 5725-1:1994 and ISO 5725-2:1994

D.1.15 reproducibility conditions of measurement [VIM 2.24]

condition of measurement, out of a set of conditions that includes different locations, operators, measuring systems, and replicate measurements on the same or similar objects

Notes

1 The different measuring systems may use different measurement procedures.

2 A specification should give the conditions changed and unchanged to the extent practical.

D.2 VIML definitions

D.2.1 type (pattern) evaluation [VIML 2.04]

conformity assessment procedure on one or more specimens of an identified type (pattern) of measuring instruments which results in an evaluation report and/or an evaluation certificate

Note: 'Pattern' is used in legal metrology with the same meaning as 'type'; in the entries below, only 'type' is used.

D.2.2 type approval [VIML 2.05]

decision of legal relevance, based on the review of the type evaluation report, that the type of a measuring instrument complies with the relevant statutory requirements and results in the issuance of the type approval certificate

Note: See also A.1.26.

D.2.3 verification of a measuring instrument [VIML 2.09]

conformity assessment procedure (other than type evaluation) which results in the affixing of a verification mark and/or issuing of a verification certificate

Note: See also OIML V 2-200:2012, 2.44.

Annex E

Principles for sealing

(Informative)

This Annex highlights considerations for determining which parameters on a grain protein measuring instrument require sealing. It also provides examples of sealing methods, such as metrological audit trails, and the minimum requirements for an effective seal.

E.1 Terminology specific to this Annex

E.1.1 adjustment mode

operational mode of a measuring instrument which enables the user to make adjustments to sealable parameters, including changes to configuration parameters

E.1.2 adjustment

change in the value of the sealable calibration parameters or the sealable configuration parameters of an instrument

E.1.3 audit trail

electronic count and/or information record of the changes to the values of the calibration or configuration parameters of a measuring instrument

E.1.4 enabling/inhibiting sealable hardware

physically sealable hardware, such as a two-position switch located on a remotely configurable instrument that enables and inhibits the sealable parameters of the instrument from being changed from a remote device

E.1.5 event

while in adjustment mode, an action in which

- one or more changes are made to configuration parameters, or
- adjustments are made to one value (or values for a set of values) for a calibration parameter (e.g. adjustments for a set of calibration factors to linearize device output)

Note: If no adjustment is made, then there is no event. In the case of a centralized audit trail, the same values for the same parameter sent to multiple devices shall be considered to be the same event. In the case of a centralized event logger, the event logger must identify both the device and the parameter that was changed.

E.1.6 event counter

non-resettable counter that increments once each time the mode that permit changes to sealable parameters is entered and one or more changes are made to sealable parameters of the instrument

E.1.7 event logger

form of audit trail containing a series of records where each record contains the number from the event counter corresponding to the change to a sealable parameter, the identification of the parameter that was changed, the time and date when the parameter as changed, and the new value of the parameter

E.1.8 physical seal

physical means, such as lead and wire, used to seal a device to detect access to those adjustable features that are required to be sealed

E.1.9 remote configuration capability

ability to adjust a measuring instrument or change its sealable parameters from or through some other device that is not itself necessary to the measurement operation or is not a permanent part of the instrument

E.1.10 remote device

device that (1) is not required for the measurement operation of the instrument or for computing the transaction information in one or more of the available operating modes for commercial measurements, or (2) is not a permanent part of the measuring instrument. In the context of this Annex, a remote device has the ability to adjust a measuring instrument or change its sealable configurable parameters

E.1.11 remotely configurable device

any measuring instrument with remote configuration capability that permits sealable configuration or calibration parameter values to be deleted, appended to, modified, or substituted in whole or in part by downloading over any type of communications link from another device, such as a geographically local or remote console or computer

E.1.12 seal

as a verb, to seal a device is to render a device secure so that access to adjustments and other sealable parameters will be detectable

E.1.13 sealable parameters

calibration and configuration parameters that are required to be sealed

E.1.14 unrestricted access to sealable parameters

unrestricted access means that a physical security seal is not present, so that access to the sealable parameters is available from a remote device at any time at the request of an authorized operator subject to the operating status of the receiving device

E.2 Principles for determining features to be sealed

The need to seal some features depends on both the following:

- the ease with which the feature or the selection of the feature can be used to facilitate fraud; and
- the likelihood that the use of the feature will result in fraud being undetected.

Features or functions which the operator routinely uses as part of device operation, such as selecting the grain calibration to be used, are not sealable parameters and shall not be sealed.

If selection of a parameter or set of parameters would result in performance that would be obviously in error, such as the selection of parameters for different countries, then it is not necessary to seal the selection of these features.

If individual device characteristics are selectable from a “menu” or a series of programming steps, then access to the “programming mode” must be sealable.

Note: If an audit trail is the only means of security, then it shall update only after at least one sealable parameter has been changed; simply accessing the sealable parameters via a menu shall not update the audit trail.

For parameters protected by physical means of security, once a physical security seal is applied to the instrument, it should not be possible to make a metrological change to those parameters without breaking that seal. For parameters protected by electronic means of security, it should not be possible to make a metrological change to those parameters without that change being reflected in an audit trail. Since this philosophy addresses provisions for protecting access to any metrological adjustment, the philosophy should be applied consistently to all electronic device types.

If a device must undergo a physical act, such as cutting a wire and physically repairing the cut to reactivate the parameter, then this physical repair process would be considered an acceptable way to select parameters without requiring a physical seal or an audit trail.

E.3 Typical features and parameters to be sealed

The following provides examples of parameters that are to be sealed. The examples are provided for guidance and are not intended to cover all possible parameters.

E.3.1 Calibration parameters

Calibration parameters are those adjustable parameters which can affect measurement or performance accuracy, and whose values need to be updated on an ongoing basis to maintain device accuracy. Calibration parameters can be classified into three categories:

(1) Those parameters which are adjusted to standardize or normalize instrument response to changes in the physical parameter being measured. Examples include zero-setting and test point adjustments, temperature sensing element zero and span adjustments, amplifier gain settings, optical wavelength standardization adjustments, etc. These are parameters normally set by the manufacturer or a competent service representative.

(2) Those parameters which are common to all instruments of the same type for a given grain type (e.g. grain P_{MB} calibration coefficients). The approval certificate lists the calibration coefficients (or a unique identifier) for each grain type which has been approved for use on a particular type of grain protein measuring instrument.

(3) Those parameters which are adjusted for each grain type to standardize P_{MB} readings on like instruments (e.g. slope and bias settings).

E.3.2 Configuration parameters

Configuration parameters are those adjustable or selectable parameters which can affect the accuracy of a transaction or can significantly increase the potential for fraudulent use of the device, and whose values only need to be updated during instrument installation or upon replacement of a component and which are not expected to change after initial installation settings have been made:

(1) System date and time (only if used by an event logger as audit trail information);

(2) P_{MB} value resolution;

(3) Sample size and/or number of sub portions measured (if not determined by individual calibrations);

(4) Password for access to sealable parameters (if used);

(5) Enable/disable display of constituent values that are not legally relevant;

(6) Format for results display and recording;

(7) Operating range limits (e.g. temperatures);

(8) Enable/disable display or recording of results for out-of-limits conditions.

E.4 Metrological audit trails

E.4.1 Scope

The ability of users to make changes which affect the metrological integrity of the device (e.g. slope, bias, etc.) in normal operation and the remote configuration capability of commercial protein measuring instruments has led to new, more appropriate means of sealing being implemented. These instruments must be either physically sealed or must incorporate an approved form of audit trail.

Included below are the requirements for the acceptable forms of metrological audit trail, which are recognized as providing acceptable security.

E.4.2 Event loggers: An acceptable form of audit trail

The event logger is the minimum form of audit trail for instruments that allows unrestricted access whether by an operator or a remote device, to the configuration or calibration parameters.

(1) The event logger shall contain the following information: event counter; date and time; parameter ID; new value.

Note: For calibration changes consisting of multiple calibration constants, the calibration version number is to be used as the new value rather than the calibration constants.

(2) This information shall be automatically entered into the event logger by the measuring instrument. Additional relevant information is permitted (e.g. the identification of the person who made the adjustment or the old value of the parameter that was changed).

(3) The date and time shall be presented in an understandable format. The date shall include the month, day and year. The time shall include the hour and minutes.

(4) A hard-copy printout of the contents of the event logger shall be available upon demand from the instrument or an associated device on the site of the instrument installation. The printing of the event logger contents shall exclude other information not relevant to the changes logged such as transaction data, number of measurements performed, etc.

(5) An event logger shall have a capacity of at least 25 times the number of sealable parameters; however, it is not required to retain more than 1000 events for all parameters combined.

E.4.3 General requirements for metrological audit trails

The following general requirements for metrological audit trails must be satisfied:

(1) The adjustment mode shall address only sealable parameters in order to avoid entering the adjustment mode to access non-sealable parameters that must be routinely changed as part of the normal use of the device.

(2) An event counter shall have a capacity of at least 1000 values (e.g. 000 to 999).

(3) In the case of the event logger, the event counter will increment once for each change to a sealable parameter since each new value must be retained in the event logger. If an adjustment mode is entered but no changes are made, this does not constitute an event and the counter must not increment.

(4) When the storage memory of the event logger has been filled to capacity, any new event shall cause the oldest event to be deleted. The event counter used in the event logger shall continue to increment to its capacity, although the event logger may retain fewer records than the count capacity of the event counter. The event counter provides the necessary information to indicate the number of records that have been overwritten in the event logger as new information overwrites the old records.

(5) The audit trail data shall be

  (a) stored in non-volatile memory and shall be retained for at least 30 days if power is removed from the device, and

  (b) protected from unauthorized erasure, substitution, or modification.

(6) Access to the audit trail information for the purpose of printing the contents must be “convenient” for an enforcement official of the national responsible body.

  (a) Accessing the audit trail information for review shall be separate from the calibration mode so there is no possibility for the official to change or corrupt the device configuration or the contents of the audit trail.

  (b) Accessing the audit trail information shall not affect the normal operation of a device before or after accessing the information.

  (c) A key (for a panel lock) may be required to gain access to the means to view the contents of the audit trail. Access may be through the supervisor’s mode of operation of the device.

(d) Accessing the audit trail information shall not require the removal of any additional parts other than normal requirements to inspect the integrity of a physical seal.

(7) The printed form of the audit trail information shall be readily interpretable by an official.

(8) The information from an event logger shall be printed in order from the most recent event to the oldest event. If a device is not capable of printing all the information for a single event on one line or at one time, the information shall be displayed in blocks of information, which are readily understandable.