



INTERNATIONAL ORGANIZATION OF LEGAL METROLOGY

Second Committee Draft of a Recommendation on

## **Protein Measuring Instruments for Cereal Grain and Oil Seeds**

Part 1: Metrological and technical requirements

Part 2: Metrological controls and performance tests

Part 3: Report format for type evaluation

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## FOREWORD

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## **PART 1: METROLOGICAL AND TECHNICAL REQUIREMENTS**

### **1 SCOPE**

This Recommendation includes metrological and technical requirements and the test methods for metrological control of digital, self-indicating measuring instruments used to determine the protein content of grain and oilseeds for commercial transactions. It specifies maximum permissible errors (MPEs) for type evaluation, initial in-field verification and subsequent reverification.

The provisions in this document are based on the performance of instruments that estimate the mass fraction of protein constituents in grain and oilseeds via inferential means. This Recommendation is not meant to preclude the application of new technologies to grain protein measurement.

### **2 TERMINOLOGY**

The terms and definitions used in this document, where possible are consistent with the OIML V2 International Vocabulary Metrology – Basic and General Concepts and Associated Terms (VIM:2007) [1], OIML V1 International Vocabulary of Terms in Legal Metrology [2], and OIML D 11 General Requirements for Electronic Measuring Instruments [3]. This section also includes terms applicable to protein measuring instruments.

#### **2.1 Definitions**

##### **2.1.1 calibration equation**

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

##### **2.1.2 certified reference material; CRM [VIM, 5.14]**

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.

##### **2.1.3 error shift**

Same definition as fault (see clause 2.1.4)

See clause 4.5 Table 1 columns 5 and 6 for the maximum error shift allowed on specific grain types.

##### **2.1.4 fault [OIML D11, 3.9]**

Difference between the error of indication and the intrinsic error of a measuring instrument.

#### **NOTES**

1 Principally, a fault is the result of an undesired change of data contained in of 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.

### **2.1.5 grain**

For the purpose of this document, the term grain is taken to mean those cereal grains and oilseeds listed in column 1 of Table 1.

### **2.1.6 intrinsic error [OIML D 11, 3.7]**

Error of a measuring instrument, determined under reference conditions.

### **2.1.7 initial intrinsic error [OIML D 11, 3.8]**

Intrinsic error of a measuring instrument as determined under reference conditions prior to performance tests and durability evaluations.

### **2.1.8 maximum permissible error; limit of error [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.

#### **NOTES**

1 Usually the term “maximum permissible errors” or “limits of error” are used, where there are two extreme values.

2 The term “tolerance” should not be used to designate ‘maximum permissible error’.

The maximum permissible errors for various kinds of grain are listed in clause 4.5.

### **2.1.9 measurement error; error [VIM, 2.16]**

Measured quantity value minus a reference quantity value.

Refer to the VIM for NOTES 1-2

### **2.1.10 moisture basis ( $M_B$ )**

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

### **2.1.11 networked instrument**

An instrument that is linked, either electronically or manually under a quality system, to an instrument aligned with a whole-grain certified reference material so that its performance may be monitored on a daily basis or according to a schedule set by the quality system administrator.

### **2.1.12 protein content ( $P_{MB}$ )**

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

### **2.1.13 protein measuring instrument**

An instrument that infers the content of protein in grain samples.

### **2.1.14 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.

#### **2.1.15 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 Rated operating conditions generally specify intervals of values for a quantity being measured and for any influence quantity

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.

#### **2.1.16 reference material; RM [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.

Refer to the VIM for NOTES 1-8

#### **2.1.17 reference quantity value [VIM, 5.18]**

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

Refer to the VIM for NOTES 1-2

In this Recommendation, the protein content of the whole-grain CRM used for testing is the reference quantity value.

#### **2.1.18 significant fault**

An error shift exhibited by the equipment under test that is greater than the values listed in clause 4.5 Table 1 columns 5 and 6.

The following error shifts are not considered to be a significant fault, even when they exceed the maximum value:

- a) error shifts arising from simultaneous and mutually independent cause (e.g. EM fields and discharges) originating in a measuring instrument or in its checking facilities;
- b) error shifts implying the impossibility to perform any measurement;
- c) transitory error shifts being momentary transitions in the indication, which cannot be interpreted, memorised or transmitted as a measurement result; and
- d) error shifts giving rise to variations in the measurement result so serious that they are bound to be noticed by all those interested in the result of the measurement.

## **2.2 Abbreviations and acronyms**

AC: alternating current

DC: direct current

EM: electromagnetic

EMC: electromagnetic compatibility

e.m.f.: electromotive force

ESD: electrostatic discharge

EUT: equipment under test

IEC: International Electrotechnical Committee

ISO: International Organization for Standardization



MPE: maximum permissible error  
 OIML: International Organization of Legal Metrology  
 RH: relative humidity  
 SD: standard deviation  
 SDD: standard deviation of differences

### 3 UNITS OF MEASUREMENT

The unit of measurement used for protein content of a grain sample is percentage protein by mass (see clause 2.1.12). The abbreviation for percentage by mass is % w/w. Conventionally, the percentage symbol alone (%) is used.

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

$$P_{MB} = P_m \times \frac{100 - M_B}{100 - m} \quad \text{Equation 1}$$

where:  $m$  = the actual moisture concentration of the sample  
 $M_B$  = the moisture basis for the grain type

The national responsible body shall clearly specify the moisture basis for all applicable grain types.

### 4 METROLOGICAL REQUIREMENTS

#### 4.1 Measuring ranges

The manufacturer shall clearly specify the types of grain that the instrument can analyse with respective measuring and indication ranges for the protein content. These ranges shall encompass commercially important protein content ranges specified by the national responsible body (see clause 5.1).

#### 4.2 Reference conditions

During type evaluation, reference environmental conditions for the protein measuring instrument shall be as follows:

- \*Ambient temperature ( $T_{ref}$ ):  $22^{\circ}\text{C} \pm 5^{\circ}\text{C}$
- \*Relative humidity: 30-70%
- Atmospheric pressure: 86 kPa to 106 kPa (see note below)
- Power voltage: nominal mains or test voltage,  $V_{nom}$  or  $U_{nom}$
- Power frequency: nominal frequency,  $F_{nom}$
- Instrument tilt: level at  $0^{\circ} \pm 0.1^{\circ}$
- Dust ingress: negligible (see note below)

Note: As these can expect to be fulfilled without specific measures in normal laboratory conditions, it is not deemed necessary to measure/monitor these values.

Reference conditions applicable to grain samples have been marked by asterisks (\*).

During each test at the reference conditions the temperature and relative humidity shall not vary by more than  $5^{\circ}\text{C}$  and 10% respectively within the allowable ranges.

### 4.3 Rated operating conditions for influence factors

#### 4.3.1 Verification

The instrument in service shall not be operated while exposed to influences outside the ranges applied during type evaluation.

#### 4.3.2 Type evaluation

The following influence factor ranges apply during type evaluation testing:

- |   |   |
|---|---|
| a) Ambient temperature:   | Operating range specified by the national responsible body.<br>Minimum range: 10°C to 30°C (see clause 4.3.3) |
| b) Relative humidity:   | Up to 85%, no condensation  |
| c) Atmospheric pressure:  | 86 kPa to 106 kPa   |
| d) Power voltage:   | -15% to +10% of mains or test voltage   |
| e) Power frequency:   | nominal frequency, $F_{nom}$  |
| f) Instrument tilt position:  | 5% or maximum allowable on level indicator where indicator is present   |
| g) Dust ingress:<br>(Composition of the particles as specified in clause 3.2.1 of IEC 60512-11-8) | Air velocity: 3 m/s<br>Particle concentration: 5 g/m <sup>3</sup>   |
| h) Grain sample temperature   | Manufacturer specified operating range.<br>Minimum range: 0°C to 40°C (see clause 4.3.4)                      |
| i) Sample and instrument temperature differential:  | Manufacturer specified operating range.<br>Minimum differential: 10°C (see clause 4.3.5)                      |

#### 4.3.3 Environmental operating temperature

A protein measuring instrument shall meet the protein content accuracy specification over the environmental operating range specified by the national responsible body. The specification shall encompass a minimum range of 20 °C and shall include the temperature range 10 °C to 30 °C.

The manufacturer may specify a wider temperature range in order to meet various national requirements. The instrument shall not indicate a measurement result or shall provide a warning when it operates outside the manufacturer specified temperature range.

#### 4.3.4 Grain sample temperature

The minimum sample temperature range for each grain shall be 0 °C and 40 °C.

The manufacturer shall specify the temperature range for each grain or seed for which the protein measuring instrument is to be used. The instrument must operate within the relevant MPEs specified in clause 4.5 Table 1 for grain samples within this temperature range. The instrument shall not indicate a measurement result or shall provide a warning if the sample temperature is outside the manufacturer specified range.

If the instrument is not able to measure sample temperature, then the operating procedure shall be defined by the national responsible body.

#### **4.3.5 Sample and instrument temperature differential**

The manufacturer shall specify the maximum allowable difference in temperature between the instrument and the sample for which an accurate protein content determination can be made. The instrument shall be able to take into account a temperature difference of at least 10 °C. No protein content value may be displayed or an appropriate error message shall be displayed when the difference in temperature between the meter and the sample exceeds the specified difference.

If the instrument is not able to measure sample temperature, then the operating procedure shall be defined by the national responsible body.

#### **4.4 Whole-grain measurement standards**

Whole-grain certified reference materials (CRMs) shall be used to provide reference quantity values during verification and in type evaluation accuracy and precision tests. These shall be homogenous samples of naturally occurring grain or seed and shall be certified via a reference test method or measurement campaign. Both shall be traceable to a nationally certified nitrogen standard.

Details on practical instructions for the selection and handling of whole-grain standards to use in type evaluation tests are in clause 7, Whole-grain test samples.

##### **4.4.1 Whole-grain CRM generated by a reference method**

The national responsible body shall specify the reference method, according to the options listed in Annex B, for generating whole-grain reference materials.

Essentially, a reference method allows the protein content of a sample to be inferred from a direct measurement of the mass fraction nitrogen in the sample. The procedure applied shall contain provisions for verifying the calibration of the instrument using a nationally recognised nitrogen CRM.

Laboratory accreditation of the reference method and sampling systems may be pursued to ensure that the whole-grain reference materials generated are adequate for the purpose of calibration and verification of protein measuring instruments.

##### **4.4.2 Whole-grain CRM generated by measurement campaign**

Where a measurement campaign is used, each whole-grain CRM shall be based on repeated protein content measurements on a bulk sample of grain on an ensemble of instruments according to the measurement campaign approach of ISO Guide 35 [4].

The instrument ensemble can be comprised of direct nitrogen determination methods listed in Annex B. All measurements shall be traceable to the protein content calculated for the nationally recognised nitrogen CRM. Propagation of measurement uncertainty shall be controlled. An example of the infrastructure that can be developed is included in Annex C.

The measurement campaign shall be applied with sufficient repetitions of the complete measurement cycle such that the uncertainty of the mean for the whole-grain CRM, calculated with a coverage factor of two, should be appropriate for verification (i.e. within a third of the relevant MPE for accuracy).

#### 4.5 Maximum permissible errors (MPEs)

##### 4.5.1 General

The MPEs for various types of grain presented in Table 1 are recommended values that apply to all instruments irrespective of their principles of operation.

The MPEs for protein measuring instruments do not vary as a function of protein content; hence the same MPE is applied across all test samples of the same grain type.

The MPEs for accuracy, repeatability and reproducibility in Table 1 columns 2, 3, 4 and 7 are with reference to the reference quantity value for protein content assigned to whole-grain measurement standards (see clause 4.4).

The maximum limit for the error shift in Table 1 columns 5 and 6 are with reference to the intrinsic error prior to each test.

**Table 1: Maximum permissible errors (MPEs) for type approval and verification expressed in percentage protein by mass (%)**

Grain type	Type evaluation					Verification
	Repeatability	Reproducibility	Accuracy	Error Shift Limit		Accuracy
	<i>SD</i> MPE	<i>SDD<sub>t</sub></i> MPE	MPE	Majority of tests	Sample temp test	MPE
Column 1	Column 2	Column 3	Column 4	Column 5	Column 6	Column 7
Wheat	0.2	0.3	± 0.3	± 0.2	± 0.4	± 0.4
Barley	0.3	0.4	± 0.4	± 0.2	± 0.4	± 0.5
Rice	0.25	0.5	± 0.5	± 0.3	± 0.68	± 0.6
Corn	0.25	0.5	± 0.5	± 0.3	± 0.68	± 0.8
Soybean	0.5	0.55	± 0.55	± 0.3	± 0.8	± 0.8
Canola	0.9	1.0	± 1.0	± 0.5	± 1.0	± 1.2
Lupins	0.9	1.0	± 1.0	± 0.5	± 1.0	± 1.2

Col 5 = Col 4 x 0.5 [CD5 R 59, 5.4.1]

Col 6 = Col 5 x 2.25 (corn, rice, sorghum, sunflower pulses and oats) [CD5 R 59, Annex A, A.3]

Col 6 = Col 5 x 2 (all other grains) [CD5 R 59, Annex A, A.3]

##### 4.5.2 Verification

In-field inspection is under the control of the national responsible body. Accuracy errors shall not exceed the values in Table 1 column 7 during initial verification and subsequent in-field reverification.

#### 4.6 Accuracy and precision requirements for the type at reference conditions

The error of a protein measuring instrument on a whole-grain measurement standard is the algebraic difference between the average of repeat measurements and the reference quantity value (the protein content assigned to the whole-grain CRM).

Protein measuring instruments shall be statistically tested for accuracy, repeatability, and reproducibility with whole-grain measurement standards for each grain type. All grain types will be tested at consecutive intervals (approximately 2%  $P_{MB}$ ) across the entire  $P_{MB}$  range and shall fulfil the MPEs listed in Table 1. Refer to clause 8.2 in Part 2 for test procedures and details on the mathematical analysis.

##### 4.6.1 Accuracy and precision tests and intrinsic error determination

The type of instrument is presumed to comply with the above mentioned requirements if it passes the following examinations in Part 2.

Accuracy	clause 8.2.1
Repeatability	clause 8.2.2
Reproducibility	clause 8.2.3

If these tests are carried out prior to any influence or disturbances tests, the initial intrinsic error of the instrument at reference conditions (as defined in clause 2.1.7) can be determined from these tests.

##### 4.6.2 Grain sample evaluation

Due to the natural variability of grain and oil seeds, all standards used in tests shall be pre-screened for homogeneity. The whole-grain standards used for testing are presumed to comply if the samples pass the following examinations in Part 2.

Sample homogeneity evaluation	clause 7.4
Sample stability evaluation	clause 7.5

NOTE: The sample stability evaluation applies to select test samples only.

Variation of the ambient temperature as required for some tests, may inadvertently affect the sample moisture content during testing. This can impact on the protein content. Instability in the sample protein content over the limit for the error shift in Table 1 columns 5 is undesirable in the assessment of instrument performance. Accordingly, verification of the sample protein content stability during testing via a second submitted instrument (maintained at reference conditions) is necessary. This shall take place in conjunction with the type evaluation tests listed below:

- a) Dry heat;
- b) Cold; and
- c) Damp heat, steady-state (non-condensing)

#### 4.7 Influence factors

Instruments shall be designed and manufactured so that all functions continue to operate as designed and that the error shift limit in Table 1 column 5 and 6 is not exceed when tested over the ranges of influence factors shown in clause 4.3.

#### 4.7.1 Influence factor tests

The type of instrument is presumed to comply with this requirement if it passes the basic influence tests and the following examinations in Part 2.

Instrument levelling	clause 8.3.3
Sample temperature sensitivity	clause 8.3.4
Storage temperature (extreme shipping conditions)	clause 8.3.5
Dry heat	clause 8.3.6
Cold	clause 8.3.7
Damp heat, steady-state (non-condensing)	clause 8.3.8
Dust ingress	clause 8.3.9
AC mains voltage variation	clause 8.3.10

#### 4.7.2 Basic instrument tests

Instrument instability and a warm-up time result from the design and construction of the instrument instead of solely external factors. Similar to the other influence factors, their effect on the measurement shall not exceed the limit for the error shift when determined according to the following tests:

Instrument drift and instability	clause 8.3.1
Instrument warm-up time	clause 8.3.2

#### 4.8 Disturbance tests for electronic instruments

Significant faults as defined in clause 2.1.18 shall either not occur (i.e. all functions continue to operate as designed) or shall be detected and acted upon by means of checking facilities in case of the following disturbances:

a) AC mains voltage dips, short interruptions and voltage variations	0.5 cycle	reduction to 0%
	1 cycle	reduction to 0%
	25/30 <sup>(1)</sup> cycles	reduction to 70%
	250/300 <sup>(1)</sup> cycles	reduction to 0%
b) Bursts (transients) on AC mains	Amplitude 1kV	
	Repetition rate 5 kHz	
c) Radiated radiofrequency, electromagnetic susceptibility	26 MHz - 2 GHz, 10 V/m	
d) Conducted radio-frequency fields	0.15 MHz - 80 <sup>(2)</sup> MHz, 10 V (e.m.f.)	
e) Electrostatic discharges	Up to 6 kV contact discharge	
	Up to 8 kV air discharge	
f) Mechanical shock	1 fall up to 50mm on a bottom edge	

NOTES:

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

(2) Testing up to 26 MHz is permitted. Refer to clause 8.4.4 for conditions.

The type of instrument is presumed to comply with above mentioned requirements if it passes the following examinations in Part 2.

AC mains voltage dips, short interruptions and voltage variations	clause 8.4.1
Bursts (transients) on AC mains	clause 8.4.2
Radiated radiofrequency, electromagnetic susceptibility	clause 8.4.3
Conducted radio-frequency fields	clause 8.4.4

Electrostatic discharges  
Mechanical shock

clause 8.4.5  
clause 8.4.6

## **5 TECHNICAL REQUIREMENTS**

### **5.1 Applicable grain types and protein content ranges**

Due to climatic and crop variability, the national responsible body shall specify commercially important protein content ranges (minimum range of 6%  $P_{MB}$ ) for the grain types in Table 1 and the types of grain types for which a protein measuring instrument manufacturer may seek national approval.

The grain types specified are typically those which:

- a) are of greatest economic importance;
- b) are significantly different in their physical structure to adequately test the instrumentation (e.g., large grain, small grain, and oil seeds); and
- c) are variable and are typically grown in regions of the national responsible body.

Refer to clause 7, Whole-grain test samples, for further requirements on the selection of samples for testing.

### **5.2 Sample preparation and input**

#### **5.2.1 Selection of grain type on the instrument**

Protein measuring instruments shall permit the selection of grain or seeds being measured and the selection of grain shall be clearly identified and visible to all parties present.

#### **5.2.2 Sampling and preparation**

The grain protein measuring system shall not require the operator to judge the precise volume or weight and temperature needed to make an accurate protein content determination.

The national responsible body shall specify minimum guidelines for the sampling of bulk or packed cereals for inspection or testing . These may be based on voluntary international standards (e.g. ISO or ISTA) and regional practice.

### **5.3 Instrument construction**

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.

Undue stresses, deflections, or distortions of parts shall not occur to the extent that accuracy or permanence is detrimentally affected. The housing shall be constructed so that the main components of the instrument are protected from dust and moisture.

The grain protein content may be a quantity or a function of various quantities such as: mass, volume, temperature, electrical resistance, spectral data or capacitance.

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

#### **5.4 Instrument warm up period**

When a protein measuring instrument is turned on, it shall not display or record any usable values until the operating temperature necessary for accurate determination has been attained. Otherwise, the instrument shall bear a conspicuous statement adjacent to the indication stating that the instrument needs to be turned on for a time period specified by the manufacturer prior to use. This requirement may not be necessary for instruments which do not require any warm up time.

#### **5.5 Level indicating means**

The instrument shall be equipped with a level indicator and levelling adjustments if its performance is changed by an amount greater than the tolerance requirement when the instrument is moved from a level position into a position that is out of level in any upright direction by up to 5% (approximately 3°). The level-indicating means shall be readable without removing any instrument parts requiring a tool.

#### **5.6 Presentation of the measured value**

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

The display shall permit protein concentration value determination to a resolution of at least 0.1%  $P_{MB}$ . The 0.1%  $P_{MB}$  resolution is for commercial transactions; at the national responsible body's option the display and printout shall also permit 0.01%  $P_{MB}$  resolution for type evaluation.

Measurement results shall be displayed as percent protein by mass at the moisture basis. Subdivisions of this unit shall be in terms of decimal subdivisions (not fractions). On multi-constituent instruments (e.g. instruments which also measure grain moisture content), provision shall be made for displaying and recording the constituent label (such as protein, moisture, etc.) to make it clear which constituent is associated with each of the displayed values.

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 ISO 1000:1998 The International System of Units (SI) and its Application [5].



## **5.7 Durable recording of measured values**

Some national bodies may require instruments to be equipped with a communication interface that permits interfacing with a recording element and transmitting the date, grain type, grain protein content results, and calibration version identification. Correspondence between displayed information and remote recording element shall be verified.

Measurement results shall be recorded as percent protein by mass at the reference moisture content.

A recording element shall not record any protein concentration values before the end of the measurement cycle. To prevent ambiguity, constituent labels and units accompanying the measured values must also be recorded if the instrument is not limited to measuring the protein content.

## **5.8 Checking facilities**

A protein measuring instrument shall automatically and clearly indicate if a significant fault has occurred or when a manufacturer specified operating range has been exceeded by either an error indication, or blanking the display.

Examples of operating ranges:

- a) Instrument measuring or indication range (see clause 4.1)
- b) Environmental temperature range (see clause 4.3.3)
- c) Sample temperature range (see clause 4.3.4)
- d) Sample and instrument temperature differential (see clause 4.3.5)

## **5.9 Operational safeguards**

### **5.9.1 Fraudulent use**

Instruments shall not facilitate fraudulent use by either accidental means or by deliberate means when using the instrument in the normal manner.

### **5.9.2 Operational controls**

Stand alone and networked instruments shall be subject to verification and reverification as described in clause 6.2.

### **5.9.3 Provision for electronic sealing and calibration security**

Provision shall be made for applying a security seal in a manner that requires the security seal to be broken, or for using an audit trail, or other approved means of providing security, before any change that affects the metrological integrity of the instrument can be made.

Note: Zero-setting and test point adjustments are considered to affect metrological characteristics and must be sealed.

If calibration constants are digitally stored in an electronically alterable form, the instrument shall be designed to make automatic checks to detect corruption. An error message must be displayed if calibration constants have been electronically altered and no further measurement shall be possible.

## **5.10 Software-controlled electronic devices and security**

Note: More requirements and advice on software-controlled measuring instruments and devices exist in OIML D 31 [6], particularly for instruments and applications at increased risk.

### **5.10.1 Instruments with embedded software**

For instruments and modules with embedded software, the manufacturer shall describe or declare that the software of the instrument or module is embedded, i.e. it is used in a fixed hardware and software environment and cannot be modified or uploaded via any interface or by other means after securing and/or verification. In addition to the documentation required in clause 6.1.3 the manufacturer shall submit the following documentation:

- a) Description of the legally relevant functions;
- b) Software identification that is clearly assigned to the legally relevant functions;
- c) Securing measures foreseen to provide for evidence of an intervention.

The software identification shall be provided by the instrument and noted in the type approval certificate.

### **5.10.2 Acceptable solutions for software identification**

The software identification is provided in the normal operation mode by either:

- a) a clearly identified operation of a physical or soft key, button, or switch; or
- b) a continuously displayed version number or checksum, etc.

accompanied in both cases by clear instructions on how to check the actual software identification against the reference number (as listed in the type approval certificate) marked on or displayed by the instrument.

### **5.10.3 Grain calibrations and integrity**

#### **5.10.3.1 Seasonal variability of crops and effect on calibration**

Grain protein measuring instruments determine protein levels indirectly, e.g. in NIR instruments the intramolecular properties of the nitrogen compounds cause measurable changes in the energy of radiation. Radiation is affected to some extent by other constituents and physical properties of the grain, besides the nitrogen in protein.

So in NIR instruments, a correlation based on the discrete properties of a particular grain crop and its interaction with radiation, may change on subsequent grain crops due to seasonal variability in chemical and physical properties. As such, some national authorities update the grain calibrations based on grain data collected during the year.

This data is used to adjust the grain calibrations to accommodate for the seasonal and crop year variations. The grain calibration data in many cases are downloaded to the instrument using an RS232 port. These are not considered software changes that would require a change to the software identification. Changes to the grain calibrations of the device shall be recorded in an audit trail or event logger

#### 5.10.3.2 Calibration Version

A protein measuring instrument must be capable of displaying calibration constants, a unique calibration name, or a unique calibration version number for use in verifying that the latest version of the calibration is being used to make protein content determinations.

#### 5.10.3.3 Calibration Corruption

If calibration constants are digitally stored in an electronically alterable form, the meter shall be designed to make automatic checks to detect corruption of calibration constants. An error message must be displayed if calibration constants have been electronically altered.

#### 5.10.3.4 Calibration Transfer

The instrument hardware/software design and calibration procedures shall permit calibration development and the transfer of calibrations between instruments of like models without requiring user slope or bias adjustments.

Note: Only the manufacturer or the manufacturer's designated service agency may make standardization adjustments on the protein measuring instruments. This does not preclude the possibility of the operator installing manufacturer-specified calibration constants under the instructions of the manufacturer or its designated service agency. Standardization adjustments (not to be confused with grain calibrations) are those physical adjustments or software parameters which make meters of like type respond identically to the grain(s) being measured.

### 5.10.4 Correctness of algorithms and functions

The measuring algorithms and functions of a measuring device shall be correct.

### 5.10.5 Software protection

#### 5.10.5.1 Prevention of misuse

A protein measuring instrument – especially the software – shall be constructed in such a way that the possibilities for unintentional accidental misuse are minimal.

#### 5.10.5.2 Fraud protection

Metrological critical software shall be secured against unauthorized modification, loading, or changes by swapping of hardware

## 5.11 Manufacturers 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 must include the following information:

- a) name and address of the manufacturer;
- b) the type or pattern of the instrument with which it is intended to be used;
- c) date of issue;
- d) the kind or varieties of grain for which the instrument is designed to be used;
- e) the limitations of use, including, but not confined to the protein content measurement range, grain or seed temperature, maximum allowable temperature

difference between grain sample and instrument, instrument operating temperature range, voltage and frequency ranges, electromagnetic interferences and electromagnetic compatibility. In addition this manual shall be supplied to the owner/user of the instrument in the official language(s) of the countries where it is used or in a language accepted by the national responsible body.

## **5.12 Markings**

### **5.12.1 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.

### **5.12.2 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 necessity of disassembly of a part requiring the use of any means separate from the device.

### **5.12.3 Marking operational controls, indications, and features**

All operational controls, indications, and features (e.g. indicating switches, lights displays and push buttons) shall be clearly and definitely identified. Keys visible only to the operator need only be marked to the extent that a trained operator can understand the function of each key.

## **5.13 Suitability**

### **5.13.1 Suitability for Purpose**

An instrument shall be designed to be suitable for the purpose for which it is intended to be used and shall be constructed to be suitable for service in normal conditions of use.

### **5.13.2 Suitability for Verification and Testing**

An instrument shall be designed (and installed) such that initial verification, subsequent reverification and metrological supervision can be carried out onsite without unreasonable effort.

Protein measuring instruments in service shall be so placed that all parties present have the possibility of seeing simultaneously all the measurement operations. The indicating or recording device should be seen at the same time, and all necessary steps shall be taken to eliminate any possibility of error or fraud.

## **PART 2: METROLOGICAL CONTROLS AND PERFORMANCE TESTS**

### **6 METROLOGICAL CONTROLS**

#### **6.1 Type approval**

##### **6.1.1 Application**

General application requirements are available from national authorities. The application for type approval shall be accompanied by:

- a) at least two sample instruments representative of the submitted type; and
- b) descriptive documents and drawings.

A manufacturer may also provide data and other information that support a determination of whether the performance of the instrument meets requirements according to this Recommendation.

##### **6.1.2 Sample instruments**

Manufacturers shall provide the national responsible body with at least two instruments and an operating manual. The sample instruments shall be in full working order and shall include all functions to be examined for pattern approval.

The second instrument is required for testing reproducibility and following selected influence factor tests, for confirming sample stability. It is also possible to accelerate the test program with a second instrument as the testing laboratory may carry out different tests simultaneously on different units.

Note: If the measuring instruments are part of a system that includes other than metrological functions, only that part which controls the metrological functions may be submitted for evaluation.

##### **6.1.3 Documentation**

The documentation submitted with the application for type approval shall include:

- a) description of its general principle of measurement;
- b) lists of the essential sub-assemblies, components (in particular electronics and other essential ones) with their essential characteristics;
- c) mechanical drawings;
- d) electric/electronic diagrams;
- e) installation requirements;
- f) security sealing plan;
- g) panel layout;
- h) software documentation;
- i) test outputs, their use, and their relationships to the parameters being measured;
- j) operating instructions that shall be provided to the user, documents or other evidence that supports the assumption that the design and characteristics of the measuring instrument comply with the requirements of this Recommendation;
- k) a list of grains and protein content ranges to be approved on the instrument.

Software documentation may include:

- a) a description of the legally relevant software and how the requirements are met.
- b) a description of suitable system configuration and minimal required resources;
- c) a description of security means of the operating system (e.g. password);
- d) a description of the sealing methods (e.g. physical, software encryption);

- e) an overview of the system hardware, e.g. topology block diagram, type of computer(s),
- f) type of network, etc.
- g) where a hardware component is deemed legally relevant or where it performs legally relevant functions, this should also be identified;
- h) a description of the accuracy of the algorithms (e.g. filtering of A/D conversion results, price calculation, rounding algorithms, etc.);
- i) a description of the user interface, menus and dialogues;
- j) the software identification and instructions for obtaining it from an instrument in use;
- k) list of commands of each hardware interface of the measuring instrument electronic devices including a statement of completeness;
- l) list of durability errors that are detected by the software and if necessary for understanding, a description of the detecting algorithms;
- m) a description of data sets stored or transmitted;
- n) if fault detection is realized in the software, a list of faults that are detected and a description of the detecting algorithm;
- o) the operating manual.

#### **6.1.4 Measurement Standards**

Whole-grain standards used in type evaluation tests shall fulfil the requirements in clause 7 and where applicable, clause 4.4.

#### **6.1.5 Tests and examinations**

The national responsible body shall review the operating manual for its completeness and clarity of operating instructions and shall visually inspect the instrument in conjunction with a review of its specifications by the manufacturer to determine that the technical requirements are met. Refer to the test report format guide in Part 3 clause 9 for the compulsory inclusions of the visual inspection regime for type approval.

The instrument shall be tested in accordance with these requirements and the test procedures of Part 2 clause 0. If testing of a complete instrument is not possible, tests may, as agreed by the national responsible body and the applicant, be performed on a simulated set-up or on modules or main devices separately.

Where more than multiple unit are submitted with the intention of accelerating the test program, all accuracy and influence tests shall be performed on the same unit, but disturbance tests may be carried out on no more than 2 additional instruments.

If a unit does not pass a particular test and as a result it has to be modified or repaired, the applicant shall carry out this modification to all instruments submitted for testing. After this modification, at least two different units shall be subjected to the previously failed test. If the testing laboratory has sound reason to fear that the modification will have a negative effect on tests that have passed, these tests shall be repeated.

#### **6.1.6 Test report**

The report on the tests carried out at type approval shall contain, as a minimum, the items of information according to the format provided in Part 3. A specific form may

be developed according to national preference. The manufacturer shall be provided specific comments about any test failures.

## **6.2 Initial verification**

Before being taken into service, the initial verification of each individual instrument is intended to verify compliance with the requirements of clauses 4.3 and 4.5. A new instrument shall undergo initial verification only after type approval.

### **6.2.1 Legal status of the instrument submitted for verification**

Production of measuring instruments shall be in conformance with the approved type. Initial verification of a measuring instrument includes a procedure to ensure that the individual measuring instruments conform to the approved type. But, notwithstanding this initial verification carried out by the appropriate Legal Authority or under its responsibility, the manufacturer has the full responsibility that the instrument complies with all the applicable requirements according to this Recommendation and other relevant requirements.

### **6.2.2 Verification examination and tests**

#### **6.2.2.1 Inspection**

Before starting the tests, the examinations to be performed include:

- a) installation of the instrument and surroundings
  - i. conform with the requirements given in the manual provided by the manufacturer.
  - ii. allow all interested parties a clear and unobstructed view of the indicator
- b) visual inspection to determine conformance with the approved type and to obtain a general appraisal of its design and construction;
- c) completeness and the correctness of the inscriptions;
- d) presence, the completeness, and the language of the documentation meant for the user;
- e) compliance of the power supply voltage and frequency at the location of use to with the specifications on the measuring instrument's label;
- f) conformance with requirements in Part 1:
  - i. units of measurement;
  - ii. measuring range;
  - iii. scale interval;
  - iv. construction;
  - v. presentation of the measured value (including validation of multiple indicators);
  - vi. adjustment facilities;
  - vii. protection against fraud;
  - viii. durable data storage and/or printer;
  - ix. compliance of the software with the approved type;
  - x. sealing devices; and
  - xi. provisions for stamping.

#### **6.2.2.2 Measurement standards**

The verification shall be carried out using suitable whole-grain standards that fulfil the requirements of clauses 4.4 and where applicable, clause 7.

### 6.2.2.3 Tests

The national responsible body shall specify the test procedure to determine if the performance of a protein measuring instrument meets the accuracy and repeatability requirements, or whether the instrument requires servicing or adjustment.

The number of test samples and measurement repetitions in the verification test procedure shall be determined by the national responsible body. The number of different test samples shall enable commercially important concentration ranges to be verified. There shall be sufficient measurement repetitions to generate a statistically significant test result.

If the verification test process is interrupted to make a necessary repair or adjustment, the instrument shall be reverified.

### 6.2.3 Verification marks, seals and document

After successful initial verification, the verification marks and the sealing shall be attached and/or an accompanying document shall be made up according to national regulations.

## 6.3 Metrological supervision

### 6.3.1 Reverification frequency

The obligation of subsequent verification and the interval is subject to national legislation. This Recommendation however, suggests instruments are reverified annually, with the interval not to exceed 16 months.

For countries without a system of mandatory reverification, metrological supervision consists of randomly checking the following:

- the presence of the correct, valid and undamaged verification marks and seals
- evidence of regular maintenance according to the manufacturers' instructions

The national responsible body may require the user of a protein measuring instrument to carry out a routine performance check before and/or after a series of protein content measurements. For regular performance checks, a sub-set of the verification procedure may be adopted and secondary standards used, as routine monitoring is not intended to be as thorough as a verification. The purpose is to ensure consistency of networked instruments and to monitor instrument drift so that corrective adjustments are carried out before the measurement error exceeds the MPE.

Verification is mandatory after any repair, adjustment or change that affects metrological performance.

### 6.3.2 Reverification examination and tests

Reverification shall only be performed if it can be proved that:

- a) Earlier verification has been successfully performed and the appropriate verification marks are undamaged;



- b) The period elapsed since the previous verification does not exceed the prescribed period; and
- c) Seals are not broken.

Reverification shall be carried out according to clause 6.2.2.

While consideration of instrument design, installation and suitability was afforded at initial verification, reverification shall focus on the following:

- a) instrument and calibration integrity
- b) appropriateness of use; and
- c) adequacy of maintenance.

After successful reverification, pre-existing marks must be removed or obliterated and replaced.

## **7 WHOLE-GRAIN TEST SAMPLES**

### **7.1 Practical instructions for test samples**

#### **7.1.1 Sample records**

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

#### **7.1.2 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, unless tested within 24 hours of receipt.

Prior to testing, samples are removed from cold storage and equilibrated overnight to room temperature. Samples over 18% moisture content are equilibrated to room temperature over a time period of at least 4 hours on the day of testing.

#### **7.1.3 Sample cleaning**

The sample must be clean. The condition of the sample (odour, appearance, damage, remaining foreign material, etc.) is recorded on the sample record. The sample is mixed.

#### **7.1.4 Sample size**

The sample must be divided into representative portions slightly in excess of the amounts needed for the protein measuring instrument to allow for sample stability check, where necessary, via the reference method or analysis on a master instrument.

### **7.2 Sample selection general requirements**

Guidance to be provided by the national responsible body with regards to grain type selection and applicable concentration ranges is covered in clause 5.1. National authorities shall specify reference methods and provide guidance regarding measurement campaigns for production of whole-grain CRMs traceable to a nationally certified chemical standard (see clause 4.4).

The standards used as test samples shall be naturally occurring grain or seed, i.e. the moisture should not be adjusted by soaking the sample in water or by spraying with water or by extended exposure to high humidity air. The moisture level must not make the sample susceptible to mould. This can occur at relatively low levels for certain grain types e.g. over 13% moisture for wheat.

Methods of pre-screening grain and oilseed samples for type approval tests are indicated in clause 7.4.

The testing laboratory shall choose well performing moisture-stable whole-grain standards from at least 3 adjacent 2% protein content intervals, e.g. 9% to 11%, 11% to 13%, 13% to 15%. The magnitude of the  $P_{MB}$  range selected should bracket commercially important  $P_{MB}$  levels for the grain type as specified by the national responsible body (see clause 5.1).

Selected tests will also require the grain moisture content to be distributed across adjacent 2% moisture content intervals (at least 3). The magnitude of the  $m$  range selected should bracket commercially important moisture levels for the grain type as specified by the national responsible body. For consistency of application in the OIML certificate system, it is recommended that each 2% moisture interval should begin and end with an even number, e.g. 10% to 12%, 12% to 14%, 14% to 16%. For details, refer to OIML R 59 [7].

### 7.3 Selection of sample sets for tests

#### 7.3.1 Sample set for accuracy, repeatability and reproducibility tests

The sample set for accuracy, repeatability and reproducibility tests may be identical and shall consist of at least 27 different whole-grain standards for each grain type. At least three grain types are recommended for each test.

For each grain type, a minimum of 3 different whole-grain standards shall be taken from distinct protein and moisture concentration combinations based on the consecutive 2%  $P_{MB}$  intervals and consecutive 2%  $m$  intervals specified by the national responsible body. Refer to Table 2 for an example sample set with 3 grain types, each with 6%  $P_{MB}$  and 6%  $m$  intervals.

**Table 2. Minimum number of different samples required for accuracy, repeatability and reproducibility tests (for 3 grain types, each with 6%  $P_{MB}$  and 6%  $m$  intervals)**

Grain type*		$P_{MB}$ (%)*	$m$ (%)*		
Number	Name		6-8	8-10	10-12
1	Soft white wheat	9-11	3	3	3
		11-13	3	3	3
		13-15	3	3	3
			6-8	8-10	10-12
2	Barley	10-12	3	3	3
		12-14	3	3	3
		14-16	3	3	3
			12-14	14-16	16-18
3	Corn	8-10	3	3	3
		10-12	3	3	3
		12-14	3	3	3

\*Listed values are for example only. The national responsible body may select grain types and constituent ranges in accordance with clause 5.1.

### 7.3.2 Sample set for testing instrument stability, sample temperature sensitivity, effect of heat and cold

The criterion for passing these tests is that the limit for error shift must not be exceeded. Moisture stable, homogenous samples with protein content within the specified  $P_{MB}$  intervals are to be used for influence and disturbance tests.

For a single grain type, 3 different whole-grain standards are required. Only one grain type is required to test for instrument stability, and the influence of dry heat, damp heat and cold. For the sample temperature sensitivity test, 3 grain types are required, as well as duplicates for each sample.

There shall be one sample from distinct protein and moisture concentration combinations based on the consecutive 2%  $P_{MB}$  intervals and consecutive 2%  $m$  intervals specified by the national responsible body. Refer to Table 3 for an example sample set with 3 grain types, each with 6%  $P_{MB}$  and 6%  $m$  intervals.

**Table 3 Minimum number of different samples required for testing the effect of instrument stability, heat and cold (for 3 grain types, each with 6%  $P_{MB}$  and 6%  $m$  intervals)**

Grain type*		$P_{MB}$ (%)*	$m$ (%)*		
Number	Name		6-8	8-10	10-12
1	Soft white wheat	9-11	1		
		11-13			1
		13-15		1	
			6-8	8-10	10-12
2	Barley	10-12			1
		12-14		1	
		14-16	1		
			12-14	14-16	16-18
3	Corn	8-10		1	
		10-12	1		
		12-14			1

\*Listed values are for example only. The national responsible body may select grain types and constituent ranges in accordance with clause 5.1.

### 7.3.3 Sample for tests on the effect of disturbances and other influence factors

For testing the effect of disturbances and influence factors not mentioned in clause 7.3.2, only one sample from a single grain type is required.

Effectively, it can be from the same bulk as one of the samples described in clause 7.3.2. If not, the protein and moisture concentration of the sample shall fall within the relevant  $P_{MB}$  and  $m$  intervals specified by the national responsible body.

## 7.4 Sample homogeneity evaluation

### 7.4.1 Accuracy, repeatability and reproducibility test samples

Grain or oilseed sample sets used for type approval accuracy and repeatability and reproducibility tests shall be nationally recognised whole-grain CRMs..

#### 7.4.2 Samples for testing disturbances and other influence factors

Grain or oilseed samples used for all other type approval tests will be pre-screened for homogeneity by assessing the repeatability. Each sample will be measured six times on the submitted instrument under reference conditions. For each sample the difference between the highest and lowest determination of  $P_{MB}$  shall not exceed the values listed in Table 4.

**Table 4: Homogeneity assessment criteria for samples and sample sets by type of test**

Grain type	Max difference between any two $P_{MB}$ determination for sample
Wheat	0.2
Barley	0.25
Rice	0.3
Corn	0.4
Soy bean	0.4
Canola	0.6
Lupins	0.6

Table 4 = Table 1 column 7 divided by 2 [CD2 N6 of this document May 2007, Annex B, 7.2]

#### 7.5 Sample stability evaluation

The results from the following tests will require correction for possible changes in the sample during the test:

- a) Dry heat;
- b) Cold; and
- c) Damp heat, steady-state (non-condensing)

Prior to each test, the samples to be tested shall be analysed on both submitted instruments under the reference test conditions (see clause 4.2). Immediately after analysis of any sample on the EUT, that sample shall be analysed on the second submitted unit. The second instrument shall be maintained at the reference conditions.

Particularly for a sample that has been analysed on the EUT whilst subjected to an influence factor, the sample shall be transferred in the second instrument for analysis in the shortest space of time possible. This is to prevent to the sample taken from the EUT from equilibrating at the reference test conditions prior to analysis in the second instrument.

The correction factor to be deducted from the EUT error shift is the difference between the mean  $P_{MB}$  measurements of the same grain sample on the second submitted unit.

$$\text{Corr. factor} = \text{mean } P_{MB,inst2}(\text{after test}) - \text{mean } P_{MB,inst2}(\text{prior test}) \quad \text{Equation 2}$$

## 8 TYPE APPROVAL TEST PROCEDURES

This section defines the program of performance tests intended to ensure that protein measuring instruments perform and function as intended in a specified environment and under specified conditions.

## 8.1 General

### 8.1.1 Reference test conditions

Unless otherwise specified by the test procedure the influence ranges in clause 4.2 define the reference conditions under which the intrinsic error is determined for each influence or disturbance test. When the effect of one influence quantity or disturbance is being evaluated, all other influence quantities and disturbances are to be held relatively constant, at values close to reference conditions.

### 8.1.2 Instrument preconditioning, conditioning and recovery

The guidelines for preconditioning, conditioning and recovery listed in the relevant test standard shall be observed. Where there are no preconditioning guidelines, the instrument shall be stabilised according to the manufacturer's specifications. If the manufacturer does not recommend a warm-up time, assume that accurate results will be provided immediately after the instrument is turned on.

Particularly during disturbance tests, the EUT shall not be re-adjusted at any time during the test except to reset if a significant fault has been indicated.

### 8.1.3 Instrument mounting

Unless otherwise specified, the instrument must be placed on a stable, level surface (e.g. work bench or table) during testing.

### 8.1.4 Maximum Permissible Errors

The relevant MPE referenced in each test are listed in clause 4.5 Table 1.

## 8.2 Accuracy and precision tests at reference conditions

Grain analysers will be tested for accuracy, repeatability, and reproducibility over the applicable constituent concentration ranges shown in Table 5. Instrument and calibration performance will be individually tested for each grain type.

The sample set to be used for the following tests is defined in clause 7.3.1.

### 8.2.1 Accuracy

This test will also indicate the 'initial' intrinsic error if it is carried out prior to exposing the instrument to influences and disturbances.

The two tests for accuracy are protein error,  $\bar{y}$ , (measurement result minus the certified value) and the Standard Deviation of the Differences,  $SDD$ , between the measurement result and the certified value of whole-grain measurement standards in each of the 2%  $P_{MB}$  intervals. The results from one instrument will be used. Testing of two instruments for accuracy is necessary for assessment of Reproducibility. The equations for  $\bar{y}$  and  $SDD$  follow:

$$\bar{y} = \frac{\sum_{i=1}^n (\bar{x}_i - r_i)}{n}$$

Equation 3

$$SDD = \sqrt{\frac{\sum_{i=1}^n (y_i - \bar{y})^2}{n-1}}$$

Equation 4

where,

$\bar{y}$  = average over all  $y_i$

$y_i = \bar{x}_i - r_i$

$\bar{x}_i$  = average measured  $P_{MB}$  for sample  $i$  (3 replicates)

$r_i$  = conventional true value of  $P_{MB}$  for sample  $i$

$n$  = number of samples per 2%  $P_{MB}$  interval ( $n = 9$ )

Accuracy acceptance requirements for both  $\bar{y}$  and  $SDD$  in each 2%  $P_{MB}$  interval are defined by the Type Evaluation Accuracy MPE listed in Table 1 column 4.

### 8.2.2 Repeatability

The repeatability of a meter is defined as the Standard Deviation,  $SD$ , of the three replicates. It shall be calculated for each sample in a 2%  $P_{MB}$  interval and pooled across samples. Each instrument is to be tested individually. The equation used to calculate  $SD$  is:

$$SD = \sqrt{\frac{\sum_{i=1}^n \sum_{j=1}^3 (x_{ij} - \bar{x}_i)^2}{2n}} \quad \text{Equation 5}$$

where,

$x_{ij}$  = measured  $P_{MB}$  value for sample  $i$  and replicate  $j$ ,

$\bar{x}_i$  = average of the three  $P_{MB}$  for sample  $i$  (3 replicates),

$n$  = number of samples per 2%  $P_{MB}$  interval ( $n = 9$ )

Repeatability requirement for  $SD$  in each 2%  $P_{MB}$  interval is defined by the Type Evaluation  $SD$  MPE listed in Table 1 column 2.

### 8.2.3 Reproducibility

Reproducibility between submitted instruments is estimated by calculating the standard deviation of differences,  $SDD_I$ . The equation used to calculate instrument reproducibility is:

$$SDD_I = \sqrt{\frac{\sum_{i=1}^n (d_i - \bar{d})^2}{n-1}} \quad \text{Equation 6}$$

where,

$d_i = \bar{x}_i^{(1)} - \bar{x}_i^{(2)}$

$\bar{x}_i^{(1)}$  = mean of three  $P_{MB}$  replicates for sample  $i$  on instrument 1

$\bar{x}_i^{(2)}$  = mean of three  $P_{MB}$  replicates for sample  $i$  on instrument 2

$\bar{d}$  = mean of the  $d_i$

$n$  = number of samples in all 2%  $P_{MB}$  ranges

Reproducibility requirement for the 6%  $P_{MB}$  range is defined by the Type Evaluation  $SDD_I$  MPE listed in Table 1 column 3.

### 8.3 Influence Factor Tests

The purpose of the following tests is to verify compliance with the provisions under clause 4.9 when subjected to the influence factor in the heading.

#### 8.3.1 Instrument drift and instability

Sample	Sample set described in clause 7.3.2 for one grain type only. Consecutive measurements per sample: 5
Test procedure in brief	Minimum time period for assessing instrument stability shall be 4 weeks. Each sample shall be repeatedly analysed through all submitted EUTs under reference conditions, prior to all influence and disturbance tests. The mean protein content ( $P_{MB}$ ) and the mean moisture content obtained of all results (no. samples x no. repeat analyses) shall be recorded. All samples shall be stored and retested once all other type evaluation testing has been completed.
Requirements	The maximum permitted difference between the mean $P_{MB}$ of the two tests is defined by the Error Shift Limit in Table 1 column 5.

#### 8.3.2 Instrument warm-up time

Objective	To ensure that the warm-up time recommended by the manufacturer is sufficient.
Sample	Sample described in clause 7.3.3. Consecutive measurements per sample at each condition: 5
Test procedure in brief	The EUT is powered off and stabilised at reference conditions overnight. The EUT is then powered on, and a test conducted after waiting for the specified warm-up time. Another test is conducted after waiting for one hour or twice the manufacturer recommended warm-up time (whichever is greater). For EUT where no warm-up time is specified, the sample would be tested immediately upon the instrument being powered on and then again after 1 hour.
Requirements	The difference between the mean $P_{MB}$ of the two tests is shall be within the Error Shift Limit in Table 1 column 5.

#### 8.3.3 Instrument levelling

Sample	Sample described in clause 7.3.3. Consecutive measurements per sample at each orientation: 5
Reference tilt	Instrument levelled to 0.1°.
Test procedure in brief	The sample is measured with the instrument mounted in a level surface (reference alignment); then in each of the four orientations of tilt; returning to the reference alignment for the final test. A minimum of 2 orientations of tilt shall be applied e.g. front to back and left to right (choose the direction with the greatest effect). The mean of replicate measurements at each orientation is determined.
Degree of tilt	Instruments without level indicators: 5% Instruments with level indicators: max allowable tilt limit on the indicator
Requirements	The difference between the mean $P_{MB}$ of each tilt orientation from the mean $P_{MB}$ at reference orientation shall be within the Error Shift Limit in Table 1 column 5.

#### 8.3.4 Sample temperature sensitivity

Objective	To verify compliance with the provisions in clause 4.7 under conditions
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	when the sample and instrument are at different temperatures
Sample	Sample set described in clause 7.3.2. A duplicate sample set is required. Consecutive measurements per sample at each temperature: 3
Test procedure in brief	The samples at reference temperature ( $T_{ref}$ ) will be repeatedly analysed under reference conditions prior to any treatment. During testing both EUT shall be under reference conditions with temperature $T_{ref}$ and temperature of samples varying from to $T_{ref} - \Delta T_C$ to $T_{ref} + \Delta T_H$ . The mean $P_M$ of all the samples of each grain type at the following temperatures shall be determined: $T_{ref}$ , $T_{ref} - \Delta T_C$ , $T_{ref} + \Delta T_H$ .
Sample temperatures	Sample set #1: 1) Heat to $\Delta T_H$ above $T_{ref}$ then analyse on instrument 1 2) Cool to $\Delta T_C$ below $T_{ref}$ then analyse on instrument 1 3) Cool again to $\Delta T_C$ below $T_{ref}$ , analyse on instrument 2 4) Heat to $\Delta T_H$ above the $T_{ref}$ then analyse on instrument 2 Sample set #2: 1) Cool to $\Delta T_C$ below $T_{ref}$ then analyse on instrument 1 2) Heat to $\Delta T_H$ above $T_{ref}$ then analyse on instrument 1 3) Reheat to $\Delta T_H$ above $T_{ref}$ , analyse on instrument 2 4) Cool to $\Delta T_C$ below the $T_{ref}$ then analyse on instrument 2 $\Delta T_H$ is the magnitude of the manufacturer-specified maximum difference between instrument and sample above reference temperature. $\Delta T_C$ is the magnitude or the manufacturer-specified maximum difference between instrument and sample below reference temperature. The two temperature differences $\Delta T_H$ and $\Delta T_C$ need not be equal. Reference temp $+ \Delta T_H < 45^\circ C$ In absence of a manufacturer specification for temperature differential, a minimum $\Delta T$ of $\pm 10^\circ C$ shall be used.
Requirements	The difference between the mean $P_{MB}$ of tests with $\Delta T$ applied to samples and the tests with samples at reference temperature shall be within the Error Shift Limit for Sample Temperature Sensitivity in Table 1 column 6.

### 8.3.5 Storage temperature (extreme shipping conditions)

Sample	Sample described in clause 7.3.3. Consecutive measurements per sample: 10
Test procedure in brief	The sample is repeatedly analysed at reference conditions prior to temperature cycling. The EUT is then powered down and placed in the environmental chamber. The chamber temperature is then increased to maximum temperature over a 1 hour period and maintained at that temperature for 3 hours. The chamber temperature is then decreased to minimum temperature over a 1 hour period and maintained at that temperature for 3 hours. The temperature cycle is repeated. The EUT is equilibrated under reference conditions for at least 12 hours unpowered. The EUT is switched on for the specified warm-up period and the test sample is repeatedly analysed for the second time. The mean of each replicate measurement is to be determined before and after temperature cycling.
Test severity	Minimum temperature: $-20^\circ C$ Maximum temperature: $50^\circ C$ As this test aims to simulate extreme shipping conditions, national authorities may specify different temperature limits.
Requirements	The difference between the mean $P_{MB}$ at reference conditions prior to tests and the mean $P_{MB}$ after temperature cycling shall be within the Error Shift



	Limit in Table 1 column 5.
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### 8.3.6 Dry heat

Standards	IEC 60068-2-2 [8], IEC 60068-3-1 [9]
Test method	Dry heat (non-condensing)
Sample	Sample set described in clause 7.3.2. Consecutive measurements per sample at each condition: 10
Test procedure in brief	The test consists of exposure to the specified high temperature under “free air” conditions for a specified time (after the EUT has reached temperature stability). The change of temperature shall not exceed 1 °C/min during heating up and cooling down. The samples shall be repeatedly analysed under the following conditions: a) at the reference temperature following conditioning; b) at the maximum temperature, after the required exposure time; and c) after recovery of the EUT at the reference temperature. Sample stability evaluation shall be carried out simultaneously using a second instrument at reference conditions as detailed in clause 7.5. The sample stability correction shall be applied to the difference between the mean EUT $P_{MB}$ at reference conditions prior to tests and the mean EUT $P_{MB}$ during exposure.
Test severity	Number of test cycles: 1 Maximum temperature: $T_H$ or 30 °C $T_H$ is the maximum temperature in the operating range specified by the national responsible body (see clause 4.3.3). If an operating range is not declared then the minimum operating range from 10 °C to 30 °C will apply. Exposure duration (after stabilisation): 2 h
Requirements	All operational functions shall operate as designed (e.g. indicators). The difference between the mean $P_{MB}$ at reference conditions prior to tests and the mean $P_{MB}$ during and after exposure shall be within the Error Shift Limit in Table 1 column 5.

### 8.3.7 Cold

Standards	IEC 60068-2-1 [10], IEC 60068-3-1 [9]
Test method	Cold
Sample	Sample set described in clause 7.3.2. Consecutive measurements per sample at each condition: 10
Test procedure in brief	The test consists of exposure to the specified low temperature under “free air” conditions for a specified time (after the EUT has reached temperature stability). The change of temperature shall not exceed 1 °C/min during heating up and cooling down. IEC specifies that the power to the EUT shall be switched off before the temperature is raised. The samples shall be repeatedly analysed under the following conditions: a) at the reference temperature following conditioning; b) at the minimum temperature, after the required exposure time; and c) after recovery of the EUT at the reference temperature. Sample stability evaluation shall be carried out simultaneously using a second instrument at reference conditions as detailed in clause 7.5. The sample stability correction shall be applied to the difference between the mean EUT $P_{MB}$ at reference conditions prior to tests and the mean EUT $P_{MB}$ during exposure.
Test severity	Number of test cycles: 1 Minimum temperature: $T_C$ or 10 °C $T_C$ is the minimum temperature in the operating range specified by the

	national responsible body (see clause 4.3.3). If an operating range is not declared then the minimum operating range from 10 °C to 30 °C will apply. Exposure duration (after stabilisation): 2 h
Requirements	All operational functions shall operate as designed (e.g. indicators). The difference between the mean $P_{MB}$ at reference conditions prior to tests and the mean $P_{MB}$ during and after exposure shall be within the Error Shift Limit in Table 1 column 5.

### 8.3.8 Damp heat, steady-state (non-condensing)

Standards	IEC 60068-2-78 [11], IEC 60068-3-4 [12]
Test method	Damp heat, steady-state
Sample	Sample described in clause 7.3.2. Consecutive measurements per sample at each condition: 10
Test procedure in brief	The test consists of exposure to the specified high level temperature and the specified constant relative humidity for a specified time frame. The EUT shall be handled such that no condensation of water occurs on it. The power supply is on when the damp heat is applied. The samples shall be repeatedly analysed under the following conditions: a) before application of the damp heat; b) whilst still in the damp heat, after the specified exposure time; and c) after recovery of the EUT at the reference temperature. Sample stability evaluation shall be carried out simultaneously using a second instrument at reference conditions as detailed in clause 7.5. The sample stability correction shall be applied to the difference between the mean EUT $P_{MB}$ at reference conditions prior to tests and the mean EUT $P_{MB}$ during exposure.
Test severity	Number of test cycles: 1 Maximum temperature: $T_H$ or 30 °C $T_H$ is the maximum temperature in the operating range specified by the national responsible body (see clause 4.3.3). If an operating range is not declared then the minimum operating range from 10 °C to 30 °C will apply. Humidity: 85% Exposure duration: 2 h
Requirements	All operational functions shall operate as designed (e.g. indicators). The difference between the mean $P_{MB}$ at reference conditions prior to tests and the mean $P_{MB}$ during and after exposure shall be within the Error Shift Limit in Table 1 column 5.

### 8.3.9 Dust ingress

Standards	IEC 60512-11-8 [13], IEC 60529 [14], and IEC 60721-2-5 [15].
Test method	Sand and dust
Sample	Sample described in clause 7.3.3. Consecutive measurements per sample at each condition: 10
Test procedure in brief	The test consists of exposure to cyclic temperature variation between 30 °C and 65 °C, maintaining the following conditions: Relative humidity: less than 25% Air velocity: 3 m/s Particle concentration: 5 g/m <sup>3</sup> Composition of the particles: as specified in clause 3.2.1 of IEC 60512-11-8 The samples shall be repeatedly analysed under the following conditions: a) before exposure to sand and dust; and b) after recovery of the EUT at reference conditions.

Test severity	Number of test cycles: 1
Requirements	All operational functions shall operate as designed (e.g. indicators). The difference between the mean $P_{MB}$ at reference conditions prior to tests and the mean $P_{MB}$ after exposure to sand and dust shall be within the Error Shift Limit in Table 1 column 5.

### 8.3.10 AC mains voltage variation

Standards	IEC/TR 61000-2-1 [16], IEC 61000-4-1 [17]
Test method	Variation in AC mains power voltage (single phase)
Sample	Sample described in clause 7.3.3. Consecutive measurements per sample at each condition: 10
Test procedure in brief	The test consists of exposure to the specified power condition for a period sufficient for achieving temperature stability and for performing the required measurements. The samples shall be repeatedly analysed under the following conditions: a) nominal voltage, b) upper limit c) lower limit; and d) recovery (nominal voltage).
Test severity	Number of test cycles: 1 Stabilising period after voltage change: 30 min Test voltage upper limit: $U_{nom} +10\%$ Test voltage lower limit: $U_{nom} -15\%$
Requirements	All operational functions shall operate as designed (e.g. indicators). The difference between the mean $P_{MB}$ at reference conditions prior to tests and the mean $P_{MB}$ during voltage variation and on recovery shall be within the Error Shift Limit in Table 1 column 5. The maximum allowable standard deviation of repeat measurements at any voltage level is 0.10%.
Notes	In case of three phase mains power, the voltage variation shall apply for each phase successively. The values of $U_{nom}$ are those marked on the measuring instrument. In case a range is specified, the “-” relates to the lowest value and the “+” to the highest value of the testing range.

## 8.4 Disturbance Tests

The purpose of the following tests is to verify compliance with the provisions under clause 8.4 when subjected to the disturbance named in the heading. Instruments that do not contain any active electronic circuits (e.g. transistors, IC's, radio tubes), are presumed to comply without being subjected to these tests. This justification shall be mentioned in the test report.

### 8.4.1 AC mains voltage dips, short interruptions and voltage variations

Standards	IEC 61000-4-11 [18], IEC 61000-6-1 [19], IEC 61000-6-2 [20]
Test method	Short-time reductions in mains voltage
Sample	Sample described in clause 7.3.3. Consecutive measurements per sample at each condition: 10
Test procedure in brief	Over four tests, the EUT shall be subjected to voltage reductions and interruptions of varying intensity and duration. A test generator suitable to reduce for a defined period of time the amplitude of the AC mains voltage is used. The performance of the test generator shall be verified before connecting to the EUT. The mains voltage interruptions and reductions shall be repeated with a

	<p>time interval less than the time required for a single measurement so that at least one voltage interruption occurs per measurement. At least 10 cycles are necessary for each test to enable the required number of measurements.</p> <p>Record the following prior and during each test:</p> <p>a) <math>P_{MB}</math> measurements;</p> <p>b) indications and errors; and</p> <p>c) functional performance.</p>
Test severity	<p>Test a) <math>U_{nom}</math> to zero for a duration equal to half a cycle of frequency</p> <p>Test b) <math>U_{nom}</math> to zero for a duration equal to one cycle of frequency</p> <p>Test c) <math>U_{nom}</math> to 70% reduction for a duration equal to 25/30* cycles of frequency</p> <p>Test d) <math>U_{nom}</math> to zero for a duration equal to 250/300* cycles of frequency</p>
Notes	*Values are for 50 Hz and 60 Hz respectively
Requirements	<p>All operational functions shall operate as designed (e.g. indicators).</p> <p>The effect of the disturbance on <math>P_{MB}</math> measurements shall not exceed a significant fault or the instrument shall detect and react to the fault (see clauses 2.1.18 and 4.8).</p>

#### 8.4.2 Bursts (transients) on AC mains

Standards	IEC 61000-4-1 [17], IEC 61000-4-4 [21]
Test method	Electrical bursts
Sample	<p>Sample described in clause 7.3.3.</p> <p>Consecutive measurements per sample at each condition: 10</p>
Test procedure in brief	<p>The test consists of subjecting the EUT to bursts of double exponential waveform transient voltages. All bursts shall be applied during the same measurement in symmetrical mode and asymmetrical mode.</p> <p>The characteristics of the burst generator shall be verified before connecting the EUT.</p> <p>The duration of the test shall not be less than 1 min for each amplitude and polarity. The injection network on the mains shall contain blocking filters to prevent the burst energy being dissipated in the mains.</p> <p>Record the following prior to test and during the application of bursts:</p> <p>a) <math>P_{MB}</math> measurements;</p> <p>b) indications and errors; and</p> <p>c) functional performance.</p>
Test severity	<p>Amplitude (peak value): 1 kV</p> <p>Repetition rate: 5 kHz</p>
Number of test cycles	At least 10 positive and 10 negative randomly phased bursts shall be applied at 1000 V. The bursts are applied during all the time necessary to perform a measurement. At least 10 measurements shall be made with the bursts applied.
Requirements	<p>All operational functions shall operate as designed (e.g. indicators).</p> <p>The effect of the disturbance on <math>P_{MB}</math> measurements shall not exceed a significant fault or the instrument shall detect and react to the fault (see clauses 2.1.18 and 4.8).</p>

#### 8.4.3 Radiated radiofrequency, electromagnetic susceptibility

Standards	IEC 61000-4-3 [22]
Test method	Radiated electromagnetic fields
Sample	<p>Sample described in clause 7.3.3.</p> <p>Consecutive measurements per sample at each setting: as many as possible over the sweep across the frequency range.</p>

Test procedure in brief	<p>The test procedure involves the exposure of the EUT to electromagnetic field strength as specified by the severity level and field uniformity as defined by the referred standard.</p> <p>The specified field strength shall be established prior to the actual testing (without EUT in the field).</p> <p>The field shall be generated in two orthogonal polarisations and the frequency range shall be scanned slowly. If antennas with circular polarisation (i.e. log-spiral or helical antennas) are used to generate the electromagnetic field, a change in the position of the antennas is not required.</p> <p>When the test is carried out in a shielded enclosure to comply with international laws prohibiting interference on radio communications, care needs to be taken to handle reflections from the walls.</p> <p>The frequency ranges to be considered are swept with the modulated signal, pausing to adjust the RF signal level or to switch oscillators and antennas as necessary. Where the frequency range is swept incrementally, the step size shall not exceed 1% of the preceding frequency value.</p> <p>The dwell time of the amplitude modulated carrier at each frequency shall not be less than the time necessary for the EUT to be exercised and to respond, but shall in no case be less than 0.5 s.</p> <p>The sensitive frequencies (e.g. clock frequencies) shall be analysed separately.</p> <p>Record the following prior to test and then with radiated EM fields:</p> <ol style="list-style-type: none"> <li><math>P_{MB}</math> measurements;</li> <li>indications and errors; and</li> <li>functional performance.</li> </ol>
Test severity	<p>EM frequency range: 26 MHz– 2 GHz</p> <p>Field strength: Radiated 10 V/m</p> <p>Modulation: 80 % AM, 1 kHz sine wave</p>
Requirements	<p>All operational functions shall operate as designed (e.g. indicators).</p> <p>The effect of the disturbance on <math>P_{MB}</math> measurements shall not exceed a significant fault or the instrument shall detect and react to the fault (see clauses 2.1.18 and 4.8).</p>
Notes:	<p>The EM field can be generated in different facilities, however the use of which is limited by the dimensions of the EUT and the frequency range of the facility:</p> <ol style="list-style-type: none"> <li>the strip line is used at low frequencies (below 30MHz or in some cases 150MHz) for small EUT;</li> <li>the long wire is used at low frequencies (below 30 MHz) for larger EUT;</li> <li>dipole antennas or antennas with circular polarisation placed at least 1 m from the EUT are used at high frequencies.</li> </ol>

#### 8.4.4 Conducted radio-frequency fields

Standards	IEC 61000-4-6 [23]
Test method	Conducted electromagnetic fields
Sample	<p>Sample described in clause 7.3.3.</p> <p>Consecutive measurements per sample at each setting: as many as possible over the sweep across the frequency range.</p>
Test procedure in brief	<p>The test procedure involves the use of radio frequency EM current, simulating the influence of EM fields coupled or injected into the power ports and I/O ports of the EUT using coupling/decoupling devices as defined in the referred standard.</p> <p>The performance of the test equipment consisting of an RF generator, (de-</p>

	coupling devices, attenuators, etc. shall be verified. Record the following prior to test and then with conducted EM fields: a) $P_{MB}$ measurements; b) indications and errors; and c) functional performance.
Test severity	EM frequency range: 0.15 – 80* MHz *For the frequency range 26-80 MHz, the testing laboratory may carry out the test according to clause 8.4.3. However, in case of dispute, the result from the test according to clause 8.4.4 shall prevail. RF amplitude (50 $\Omega$ ): 10 V (e.m.f) Modulation: 80 % AM, 1 kHz sine wave
Requirements	All operational functions shall operate as designed (e.g. indicators). The effect of the disturbance on the $P_{MB}$ measurement shall not exceed a significant fault or the instrument shall detect and react to the fault (see clauses 2.1.18 and 4.8).

#### 8.4.5 Electrostatic discharges

Standards	IEC 61000-4-2 [24]
Test method	Electrostatic discharge (ESD)
Sample	Sample described in clause 7.3.3. Consecutive measurements per sample at each condition: 10
Test procedure in brief	A capacitor of 150PF is charged by a suitable d.c. voltage source. The capacitor is then discharged through the EUT by connecting one terminal to ground (chassis) and the other via 330 $\Omega$ to surfaces which are normally accessible to the operator. The test includes the paint penetration method, if appropriate. For direct discharges, the air discharge shall be used where the contact discharge method cannot be applied. Before starting the tests, the performance of the ESD generator shall be verified. For EUT not equipped with a ground terminal, the EUT shall be fully discharged between discharges. Direct application: In the contact discharge mode to be carried out on conductive surfaces, the electrode shall be in contact with the EUT. In the air discharge mode on insulated surfaces, the electrode is approached to the EUT and the discharge occurs by spark. Indirect application: The discharges are applied in the contact mode to coupling planes mounted in the vicinity of the EUT. Record the following prior to test and then during application of ESDs: a) $P_{MB}$ measurements; b) indications and errors; and c) functional performance.
Test severity	Air discharge voltage: 2,4,6, 8 kV Contact discharge voltage: 2, 4, and 6 kV
Number of test cycles	At least one direct discharge and one indirect discharge shall be applied during the one measurement. At least 10 deliveries shall be made with the discharges applied. The time interval between successive discharges shall be at least 10 seconds.
Requirements	All operational functions shall operate as designed (e.g. indicators). The effect of the disturbance on the $P_{MB}$ measurement shall not exceed a significant fault or the instrument shall detect and react to the fault (see

	clauses 2.1.18 and 4.8).
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#### 8.4.6 Mechanical shock

Standards	IEC 60068-2-31 [25]
Test method	Dropping on to a face
Sample	Sample described in clause 7.3.3. Consecutive measurements per sample at each condition: 10
Test procedure in brief	The EUT, placed in its normal position of use on a rigid surface, is tilted towards one bottom edge and then is allowed to fall freely onto the test surface. The height of fall is the distance between the opposite edge and the test surface. However, the angle made by the bottom and the test surface shall not exceed 30°. Record the following prior and after the test when the instrument has been returned to the upright position: a) $P_{MB}$ measurements; b) indications and errors; and c) functional performance.
Test severity	Height of fall: 50 mm Number of falls (on bottom edge): 1
Requirements	All operational functions shall operate as designed (e.g. indicators). The effect of the disturbance on the $P_{MB}$ measurement shall not exceed a significant fault or the instrument shall detect and react to the fault (see clauses 2.1.18 and 4.8).

## ANNEX A. BIBLIOGRAPHY (MANDATORY)

1 ISO/IEC Guide 99; OIML V 2-200 (2007) International Vocabulary of Metrology – Basic and General Concepts and Associated Terms (VIM: 2007)	An international agreement on terminology, prepared as a collaborative work of experts appointed by 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. In practice, these publications are usually referred to as the "VIM".
2 OIML V1 (2000) International Vocabulary of Terms in Legal Metrology (VIML)	No abstract available
3 OIML D 11 (2004) 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.
4 ISO Guide 35 (2006), Reference materials - General and statistical principles for certification	
5 ISO 1000 (1998) The International System of Units (SI) and its Application.	
6 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.
7 OIML R 59 (20xx) Moisture Meters for Cereal Grain and Oilseeds	TC17/SC8 CD5
8 IEC 60068-2-2 (1974-01), with amendments 1 (1993-02) and 2 (1994-05) Environmental testing Part2: 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. The 1987 reprint includes IEC No. 62-2-2A
9 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). Includes appendices on the effect of: chamber size on the surface temperature of a specimen when no forced air circulation is used; airflow on chamber conditions and on surface temperatures of test specimens; wire termination dimensions and material on surface temperature of a component; measurements of temperature, air velocity and emission coefficient. Supplement A gives additional information for cases where temperature stability is not achieved during the test.
10 IEC 60068-2-1 (1990-05) with amendments 1 (1993-02) and 2 (1994-06) Environmental testing, Part2:	Concerns cold tests on both non-heat-dissipating and heat dissipating specimens



Tests, Test A: Cold	
<p>11 IEC 60068-2-78 (2001-08) Environmental testing - Part 2-78: Tests -Test Cab: Damp heat, steady state</p> <p>(IEC 60068-2-78 replaces the following withdrawn standards: IEC 60068-2-3, test Ca and IEC 60068-2-56, test Cb)</p>	<p>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. This test provides a number of preferred severities of high temperature, high humidity and test duration. The test can be applied to both heat-dissipating and non-heat dissipating specimens. The test is applicable to small equipment or components as well as large equipment having complex interconnections with test equipment external to the chamber, requiring a set-up time which prevents the use of preheating and the maintenance of specified conditions during the installation period.</p>
<p>12 IEC 60068-3-4 (2001-08) Environmental testing - Part 3-4: Supporting documentation and guidance - Damp heat tests</p>	<p>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. The object of damp heat tests is to determine the ability of products to withstand the stresses occurring in a high relative humidity environment, with or without condensation, and with special regard to variations of electrical and mechanical characteristics. Damp heat tests may also be utilized to check the resistance of a specimen to some forms of corrosion attack.</p>
<p>13 IEC 60512-11-8 (1995-11) Electromechanical components for electronic equipment - Basic testing procedures and measuring methods - Part11: Climatic tests - Section 8: Test 11h -Sand and dust</p>	<p>Defines a standard test method to assess the ability of a connector to withstand driving fine sand and dust.</p>
<p>14 IEC 60529 (2001-02) Ed. 2.1 Degrees of protection provided by enclosures (IP Code) Corr.1 (2003-01) Ed. 2.1 Am1 (1999-11) Amendment 1 Consolidated Edition</p>	<p>Applies to the classification of degrees of protection provided by enclosures for electrical equipment with a rated voltage not exceeding 72.5 kV. Has the status of a basic safety publication in accordance with IEC Guide 104.</p>
<p>15 IEC 60721-2-5 (1991-07) Classification of environmental conditions - Part 2: Environmental conditions appearing in nature - Section 5: Dust, sand, salt mist</p>	<p>Presents characteristics of dust, sand and salt mist appearing in nature, and describes the influences from these environmental factors to which products are liable to be exposed during storage, transportation and use.</p>
<p>16 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 signalling in public power supply systems</p>	<p>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. The following disturbance phenomena are considered: - harmonics - inter-harmonics - voltage fluctuations - voltage dips and short supply interruptions - voltage unbalance - mains signalling – power frequency variation - DC components</p>

<p>17 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</p>	<p>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</p>
<p>18 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</p>	<p>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. This standard applies to electrical and electronic equipment having a rated input current not exceeding 16 A per phase, for connection to 50 Hz or 60 Hz AC networks. It does not apply to electrical and electronic equipment for connection to 400 Hz AC networks. Tests for these networks will be covered by future IEC standards. The object of this standard is to establish a common reference for evaluating the immunity of electrical and electronic equipment when subjected to voltage dips, short interruptions and voltage variations. It has the status of a Basic EMC Publication in accordance with IEC Guide 107.</p>
<p>19 IEC 61000-6-1 (1997-07) Electromagnetic compatibility (EMC) - Part 6: Generic standards - Section 1: Immunity for residential, commercial and light-industrial environments</p>	<p>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. Immunity requirements in the frequency range 0 kHz to 400 GHz are covered and are specified for each port considered. This standard applies to apparatus intended to be directly connected to a low-voltage public mains network or connected to a dedicated DC source which is intended to interface between the apparatus and the low-voltage public mains network.</p>
<p>20 IEC 61000-6-2 (1999-01) Electromagnetic compatibility (EMC) - Part 6-2: Generic standards – Immunity for industrial environments</p>	<p>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. Apparatus intended to be used in industrial locations are characterized by the existence of one or more of the following: – a power network exists powered by a high or medium voltage power transformer dedicated for the supply of an installation feeding a manufacturing or similar plant; – industrial, scientific and medical (ISM) apparatus; – heavy inductive or capacitive loads are frequently switched; and – currents and associated magnetic fields are high.</p>
<p>21 IEC 61000-4-4 (2004-07) Electromagnetic compatibility (EMC) Part 4-4: Testing and measurement techniques - Electrical fast transient/burst immunity test</p>	<p>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</p>

	<p>describes a consistent method to assess the immunity of an equipment or system against a defined phenomenon. The standard defines:</p> <ul style="list-style-type: none"> <li>– test voltage waveform;</li> <li>– range of test levels;</li> <li>– test equipment;</li> <li>– verification procedures of test equipment;</li> <li>– test set-up; and</li> <li>– test procedure.</li> </ul> <p>The standard gives specifications for laboratory and post-installation tests.</p>
<p>22 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</p>	<p>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.</p>
<p>23 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</p>	<p>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. This standard does not intend to specify the tests to be applied to particular apparatus or systems. Its main aim is to give a general basic reference to all concerned product committees of the IEC. The product committees (or users and manufacturers of equipment) remain responsible for the appropriate choice of the test and the severity level to be applied to their equipment.</p>
<p>24 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.</p> <p>Consolidated Edition: IEC 61000-4-2 (2001-04) Ed. 1.2</p>	<p>This publication is based on IEC 60801-2 (second edition: 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. It additionally defines ranges of test levels which relate to different environmental and installation conditions and establishes test procedures. The object of this standard is to establish a common and reproducible basis for evaluating the performance of electrical and electronic equipment when subjected to electrostatic discharges. In addition, it includes electrostatic discharges which may occur from personnel to objects near vital equipment</p>
<p>25 IEC 60068-2-31 (1969-01) with amendment 1 (1982-01) Environmental testing Part 2: Tests Test Ec: Drop and topple, primarily for equipment-type specimens</p>	<p>Determines the effect on a specimen of simple standard treatments which are representative of the knocks and jolts likely to occur during repair work or rough handling on a table or bench. Has the status of a basic safety publication in accordance with IEC Guide 104.</p>
<p>26 ISO/DIS 16634.2 Cereals, pulses, milled cereal products, oilseeds and animal feeding stuffs — Determination of the total nitrogen content by combustion according to the Dumas</p>	

principle and calculation of the crude protein content	
27 EN ISO 5983-2. Animal feeding stuffs — Determination of nitrogen content and calculation of crude protein content — Part 2: Block digestion/steam distillation method	
28 AACC Approved Methods, 10th edition, American Association of Cereal Chemists	
29 Leco Corporation Organic Application Note 203-821-026 & 203-821-146	
30 NIST Certificate of Analysis SRM 723d (2003) National Institute of Standards and Technology	
31 NMI M 8 Pattern Approval Specifications for Protein Measuring Instruments for Grain (2004) National Measurement Institute	
32 NMI V 10 2nd Edition Uniform Test Procedures for the Verification, Certification and In-service Inspection of Protein Measuring Instruments for Grain (2007) National Measurement Institute	
33 RACI Official Testing Methods of the Cereal Chemistry Division, 4th Edition, The Royal Australian Chemical Institute	
34 Australian Grains Industry Grain Quality Laboratories Certified National Reference Samples Instructions for Participants (2008-09) Wheat and Barley; Lowien, J and NMI Grain Quality Committee: 2008	
35 ISO 13690 (1999) Cereals, pulses and milled products - Sampling of static batches	

## **ANNEX B. REFERENCE METHODS (MANDATORY)**

### **B.1 Protein content calculation from nitrogen determination**

#### **B.1.1 Dumas Combustion — Total Nitrogen Determination**

For example: "ISO/DIS 16634.2" [26]

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

#### **B.1.2 Improved Kjeldahl Method — Total Nitrogen Determination**

For example: ISO standards using the Kjeldahl method, i.e. "EN ISO 5983-2 Animal feeding stuffs – Determination of nitrogen content and calculation of crude protein content – Part 2: Block digestion/steam distillation method [27]"

### **B.2 Moisture content measurements**

Examples:

AACC International Method 44-15-02 – Moisture – Air-Oven Methods

ISO 6540:1980 – Maize – Determination of moisture content (of milled grains and on whole grains)

ISO 665:2000 – Oilseeds – Determination of moisture and volatile matter content

ISO 771:1977 – Oilseed residues – Determination of moisture and volatile matter content

ISO 6496:1999 – Animal feeding stuffs – Determination of moisture and other volatile matter content

ISO 711:1985 – Cereals and cereal products – Determination of moisture content – Basic reference method

ISO 712:1998 – Cereals and cereal products – Determination of moisture content – Routine reference method

## **ANNEX C. MEASUREMENT CAMPAIGN FOR GRAIN (INFORMATIVE)**

The procedure contained in this annex for generating whole-grain certified reference materials (certified national reference samples - CNRS) for verification of near infrared (NIR) protein measuring instruments is used in Australia. The instructions below were issued for the 2008 season and the referenced documents [see Annex A, items 28 - 33] were the current versions at that time.

### **Australian Grains Industry Grain Quality Laboratories Certified National Reference Samples Instructions for Participants Wheat and Barley [34]**

#### **C.1 Introduction**

This process is being undertaken to create a set of Certified National Reference Samples (CNRS) that can be used to verify the correct operation of NIR/NIT instruments. A number of samples are exchanged between participants at a predetermined time for reference analysis. Results are collated and, in conjunction with the National Grain Certification Committee, a final result for each sample determined.

#### **C.2 Participants**

List of participants meeting eligibility requirements (See clause C.16 Eligibility Requirements):

Laboratory ID Code (e.g. Lab A, B, C ... H)

Name of the company, laboratory address and contact details

#### **C.3 Coordinator**

Name and contact details of the coordinator.

#### **C.4 Timetable for Sample Dispatch**

Sample dispatch – Week beginning Monday 21st April 2008

CNRS Round begins – Monday 28th April 2008

Results Due to Coordinator – No later than Friday 16th May 2008

Final Results Due Out – No later than Friday 23rd May 2008

#### **C.5 Reference Methods**

The following reference methods must be used for protein and moisture determination of each sample.

##### **C.5.1 Protein (as is)**

Royal Australian Chemical Institute (RACI) – Cereal Chemistry Division (CCD) method 02-03 (Dumas combustion method).

- I. 2-Amino-2-(hydroxymethyl)-1,3-propanediol commonly referred to as Tris must be used as the standard certified reference material (CRM) to set up Dumas instruments. (Note: EDTA is not a recognised CRM).
- II. Tris should be dried at room temperature (22 °C to 23 °C) for 24 h in a vacuum desiccator over anhydrous magnesium perchlorate (or equivalent) before use.

- III. Tris should be ground to a fine powder using a mortar and pestle before use. Any remaining ground Tris should be stored separately to unground Tris or disposed of.
- IV. Where possible 3-Pyridinecarboxylic Acid commonly referred to as Nicotinic Acid should be used as a cross check of Dumas instrument setup.

### C.5.2 Moisture

American Association of Cereal Chemists (AACC) method 44-15A.

### C.5.3 Protein (CM)

The following equation may be used to convert protein (As Is) to a corrected moisture (CM) basis:

$$P_{CM} = P_{AsIs} \times \frac{100 - M_{CM}}{100 - M}$$

Where:

- $P_{CM}$  = Protein corrected moisture basis
- $P_{AsIs}$  = Protein content of the grain (Dumas)
- $M$  = Moisture content of the grain
- $M_{CM}$  = Corrected moisture basis  
(11% wheat; 0% barley)

## C.6 Standardised Setup of Dumas Instruments

Various setup differences of the Dumas (Leco) instrument can influence the results of samples analysed. The following standardised setup must be used.

### C.6.1 Instrument Setup

**Table C 1. Required settings for different instruments**

Set up	Truspec, FP528, FP428	FP2000			
Furnace temperature	950 °C	1050 °C			
Oxygen flow profile	All high	Burn cycle	Lance flow	Purge flow	Time (sec)
		1	OFF	ON	5
		2	ON	ON	15
		3	ON	ON	15
		4	ON	ON	15
		5	ON	OFF	END
Atmospheric blank	0.04	-			
Protein factor	Wheat= 5.7; Barley= 6.25	Wheat= 5.7; Barley= 6.25			
Sample weight	0.2 to 0.35 g	0.5 to 1.25 g			

### C.6.2 Calibration Setup

- I. Ensure the system is leak free and all ambients are at operating levels
- II. Define the standard settings using the theoretical N value for Tris (See 15.Theoretical Value of Tris)
- III. Run blank samples until results are stable near zero
- IV. Adjust blank if necessary
- V. Run standard samples
  - If using a two point curve
    - Run 3-5 Tris standards of approximate weight 0.2-0.35g
    - Standards should be run in sealed tin foil cups (Part No. 502-186)

If using a multi point curve

- Run 5 Tris standards in duplicate at weights of 0.4, 0.3, 0.2, 0.1 and 0.05g
- Duplicate weights should give similar results but different weights may give different results
- Standards should be run in sealed tin foil cups (Part No. 502-186)
- Curve fitting should be linear or linear (fixed at origin)

VI. Adjust calibration if necessary

Note: If the above weights for Tris standards can not be analysed by the instrument, weights within the instrument capabilities should be used.

### C.7 Level of Reporting

All results are to be reported to two (2) decimal places.

### C.8 Sample Preparation, Homogeneity Check and Dispatch Procedure

#### C.8.1 Sample Preparation

##### C.8.1.1 Wheat

An initial standardised sample should be selected from any uniform source that complies with the following prerequisites.

- The sample should weigh at least twenty (20) kg
- The sample should be in the protein range allocated to your laboratory (Table C 2)

Note: If a sample can not be obtained in the protein range specified for your laboratory contact the coordinator as soon as possible.

- The sample should be homogeneous of a similar protein range
- The sample should be a milling wheat grade (not durum)
- The sample should be free from insects, foreign seeds and any other foreign material

**Table C 2. Allocated laboratory protein ranges and sample numbers for wheat**

Sample Code	Lab Code	Protein Range (%)
1	A	10.0 – 10.4
2	B	12.5 – 12.9
3	C	13.5 – 14.0
4	D	11.5 – 12.4
5	E	9.0 – 9.9
6	F	11.0 – 11.4
7	G	13.0 – 13.4
8	D	10.5 – 10.9

Note: Lab D is required to produce two (2) samples. Lab H is not required to produce a wheat sample.

The sample is to be divided into eight (8), 1.8 kg sub-samples using a suitable mechanical divider. Each sub-sample should be placed in a non-permeable, completely sealable container (plastic bag or similar) and labelled with the sub-sample number and the date of dispatch. The sub-sample number is a combination of the sample code followed by the lab code (eg. Lab G is required to produce sample 7 for each of the 8 laboratories. Sub-sample numbers would be 7A, 7B, 7C, 7D, 7E, 7F, 7G and 7H).



### C.8.1.2 Barley

An initial standardised sample should be selected from any uniform source that complies with the following prerequisites.

- The sample should weigh at least twenty (20) kg
- The sample should be in the protein range allocated to your laboratory (Table C 3)

Note: If a sample can not be obtained in the protein range specified for your laboratory contact the coordinator as soon as possible.

- The sample should be homogeneous of a similar protein range
- The sample should be a malting barley grade or a premium feed grade if the protein range allocated is outside the normal malt barley range
- The sample should be free from insects, foreign seeds and any other foreign material

**Table C 3. Allocated laboratory protein ranges and sample numbers for wheat**

Sample Code	Lab Code	Protein Range (%)
1	A	8.0 – 8.9
2	C	12.0 – 12.9
3	D	10.0 – 10.9
4	E	9.0 – 9.9
5	B	11.5 – 11.9
6	H	11.0 – 11.4

Note: Lab F and Lab G are not required to produce a barley sample.

The sample is to be divided into eight (8), 1.8 kg sub-samples using a suitable mechanical divider. Each sub-sample should be placed in a non-permeable, completely sealable container (plastic bag or similar) and labelled with the sub-sample number and the date of dispatch. The sub-sample number is a combination of the sample code followed by the lab code (eg. Lab C is required to produce sample 2 for each of the 8 laboratories. Sub-sample numbers would be 2A, 2B, 2C, 2D, 2E, 2F, 2G and 2H).

### C.8.2 Homogeneity Check

Each sub-sample is to be checked for homogeneity before dispatch by analysing three (3) times through a NIR/NIT instrument (Infratec or similar) and results recorded (See clause C.11 Reporting of Results to Coordinator). If any results vary by more than +/- 0.10 from the mean for wheat or +/- 0.20 from the mean for barley, or by more than +/- 0.30 between sub-samples, the entire sample should be remixed, sub-sampled again and reanalysed by the NIR/NIT instrument.

### C.8.3 Dispatch Procedure

Each sub-sample should be sealed and dispatched to the laboratory indicated by its lab code to the address listed (See clause C.2 Participants). You should retain the sample(s) with your lab code. All grain left over from the original 20 kg sample should be stored in cold storage in an air-tight container clearly labelled with the sample code and date.

Note: If foreign contamination is detected in a sample by AQIS officers the dispatching laboratory may be requested to submit a fresh, clean sample.

### C.9 Dumas Verification

To ensure correct setup and operation, the Dumas (Leco) instrument must be verified as working correctly before any new season samples are analysed. Six (6) sub-samples of EDTA should first be analysed in tin foil cups (Part No. 502-186) for protein only and results recorded (See clause C.11 Reporting of Results to Coordinator).

As a further check 4 samples from the 2007-08 CNRS (i.e. last year) should be run. Sample 1 (P 10.08 ± 0.11%), sample 2 (P 12.67 ± 0.09%), sample 3 (P 13.90 ± 0.09%) and sample 4 (P 11.30 ± 0.10%) should be allowed to equilibrate to room temperature. Then 1 x 50g sub-samples should be taken from each sample and labelled appropriately. Each sub-sample should then be analysed for protein and moisture 6 times and results recorded (See clause C.11 Reporting of Results to Coordinator). Note: Correct operation of the Leco must be verified before continuing with sample validation.

### C.10 Sample Validation

You should wait until you have received all eight (8) samples of wheat and all six (6) samples of barley. Once received in the laboratory, 6 x 50g sub-samples should be taken from each of the 8 samples of wheat and 6 samples of barley and labelled appropriately (8 samples x 6 sub-samples each = 48 sub-samples wheat; 6 samples x 6 sub-samples each = 36 sub-samples barley; 84 sub-samples total). Each sub-sample should then be analysed for protein and moisture (See clause C.5 Reference Methods) and the results recorded (See clause C.11 Reporting of Results to Coordinator).

Samples should be stored in cold storage in an air-tight container clearly labelled with the sample number (See clause C.13 Sample Labelling and Storage Requirements). Samples should not be used until a final result for each sample is determined (See clause C.12 Reporting of Results by Coordinator).

### C.11 Reporting of Results to Coordinator

Results should be e-mailed to the coordinator as an Excel spreadsheet. The result template named *Certified National Reference Samples Results Template 2008-09.xls* is to be used for this purpose. The appearance of this template is shown below.

**Australian Grains Industry  
Grain Quality Laboratories  
Certified National Reference Samples  
2008-09**

Your Lab Code

Please enter your lab code in the blue box. This will automatically enter information onto the next 3 sheets. By clicking on the continue button you will be taken to the next sheet.

Lab Code	Lab	Wheat Sample	Barley Sample
A	name undisclosed A	1	1
B	name undisclosed B	2	5
C	name undisclosed C	3	2
D	name undisclosed D	4 & 8	3
E	name undisclosed E	5	4
F	name undisclosed F	6	-
G	name undisclosed G	7	-
H	name undisclosed H	-	6

**Figure C 1. Laboratory details input page**

Australian Grains Industry Grain Quality Laboratories Certified National Reference Samples 2008-09  Homogeneity Check - Wheat Testing Lab name undisclosed A								Australian Grains Industry Grain Quality Laboratories Certified National Reference Samples 2008-09  Homogeneity Check - Barley Testing Lab name undisclosed A							
Protein % CM (NIR Method (N x 5.7, 11% MB))								Protein % CM (NIR Method (N x 6.25, 0% MB))							
Result	Sample	Sample	Sample	Sample	Sample	Sample	Sample	Result	Sample	Sample	Sample	Sample	Sample	Sample	Sample
1	1A	1B	1C	1D	1E	1F	1G	1	1A	1B	1C	1D	1E	1F	1G
2								2							
3								3							
Average							Average	Average							Average

NA							
NA	NA	NA	NA	NA	NA	NA	NA
NA	NA	NA	NA	NA	NA	NA	NA
NA							
NA							
NA							
NA							Average

WARN!

Will be given if the sample is not homogenous.

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Continue

**Figure C 2. Homogeneity check result page**

Australian Grains Industry Grain Quality Laboratories Certified National Reference Samples 2008-09  Leco Verification Testing Lab name undisclosed A					
Protein % As Is (Dumas Method (N x 5.7))					
Sub Sample	Sample	Sample	Sample	Sample	Sample
1	EDTA %N	1E	2E	3E	8E
2					
3					
4					
5					
6					
Average					

Moisture % (Reference Method)					
Sub Sample	Sample	Sample	Sample	Sample	Sample
1	EDTA	1E	2E	3E	8E
2	NA				
3	NA				
4	NA				
5	NA				
6	NA				
Average	NA				

Protein % CM (Dumas Method (N x 5.7, 11% MB))					
Sub Sample	Sample	Sample	Sample	Sample	Sample
1	EDTA %N	1E	2E	3E	8E
2					
3					
4					
5					
6					
Average					

Reference Result	
EDTA	9.58
Sample 1	10.08
Sample 2	12.67
Sample 3	13.90
Sample 4	11.30

Please enter the EDTA %N value from the bottle here.

WARN!

Will be given if the Leco check fails.

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Continue

**Figure C 3. Reference instrument check results page**

Australian Grains Industry Grain Quality Laboratories Certified National Reference Samples 2008-09								Back	Australian Grains Industry Grain Quality Laboratories Certified National Reference Samples 2008-09						
Validation - Wheat								Validation - Barley							
Testing Lab name undisclosed A								Testing Lab name undisclosed A							
Protein % As Is (Dumas Method (N x 5.7))								Protein % As Is (Dumas Method (N x 6.25))							
Sub Sample	Sample 1A	Sample 2A	Sample 3A	Sample 4A	Sample 5A	Sample 6A	Sample 7A	Sample 8A	Sub Sample	Sample 1A	Sample 2A	Sample 3A	Sample 4A	Sample 5A	Sample 6A
1									1						
2									2						
3									3						
4									4						
5									5						
6									6						
Moisture % (Reference Method)								Moisture % (Reference Method)							
Sub Sample	Sample 1A	Sample 2A	Sample 3A	Sample 4A	Sample 5A	Sample 6A	Sample 7A	Sample 8A	Sub Sample	Sample 1A	Sample 2A	Sample 3A	Sample 4A	Sample 5A	Sample 6A
1									1						
2									2						
3									3						
4									4						
5									5						
6									6						
Protein % CM (Dumas Method (N x 5.7, 11% MB))								Protein % CM (Dumas Method (N x 6.25, 0% MB))							
Sub Sample	Sample 1A	Sample 2A	Sample 3A	Sample 4A	Sample 5A	Sample 6A	Sample 7A	Sample 8A	Sub Sample	Sample 1A	Sample 2A	Sample 3A	Sample 4A	Sample 5A	Sample 6A
1									1						
2									2						
3									3						
4									4						
5									5						
6									6						

Figure C 4. CNRS validation results page

**C.12 Reporting of Results by Coordinator**

Results from all participants will be collated in an Excel spreadsheet by the coordinator and e-mailed to each member of the National Grain Certification Committee for their perusal. A telephone conference will then be conducted by the National Grain Certification Committee for discussion and acceptance of the results. The final result for each sample, as determined by the National Grain Certification Committee, will then be e-mailed to participants.

Note: Laboratories with outliers will be contacted for investigation prior to the final release of results.

**C.13 Sample Labelling and Storage Requirements**

**C.13.1 Labelling**

Once final results are received samples are required to be labelled as certified national reference samples. The labels will contain the following information that allows each sample to be identified;

- The name of the producer
- The name of the material (eg wheat or barley)
- The producers code for the sample (eg CNRS 7A)
- The sample result and uncertainty
- The batch number
- Any relevant health and safety warnings

If a certificate has been issued for the material the above details should match those shown on the certificate.

Where adhesive-backed labels are used they must be capable of remaining intact for the lifespan of the reference material.

### C.13.2 Storage

CNRS samples will be packaged and stored in such a way as to minimise the risk of container breakage and atmospheric exposure (for example in airtight jars or heat sealed impervious plastic).

The collaborating laboratory will minimise the risk of insect activity by storing the reference material in its sealed containers in a cool-store, but must ensure that the samples equilibrate to room temperature before further handling or exposure to the atmosphere.

### C.14 Copies of Documents

Copies of the following documents are held by the coordinator and are available to participants on request.

- Reference methods
- Standard reference material Certificate of Analysis
- Related MSDS

### C.15 Theoretical Value of Tris

Chemical Formula (HOCH<sub>2</sub>)<sub>3</sub>CNH<sub>2</sub>

	Atomic Mass	n	M (g mol <sup>-1</sup> )	%
H	1.00794	11	11.08734	9.1529
O	15.9994	3	47.9982	39.6237
C	12.0107	4	48.0428	39.6605
N	14.0067	1	14.0067	11.5629
Total			121.13504	100.0000

Certified Value 99.924 % ± 0.036 % (from Certificate of Analysis)

Therefore Tris contains 11.554 % ± 0.004 % N

### C.16 Eligibility Requirements

To be eligible to participate, a participant must;

- Hold NATA accreditation in the field of chemical testing of cereal grains by oven moisture and Dumas protein, or equivalent, and;
- Satisfy any other requirements as determined by the National Grain Certification Committee.

## **PART 3: TEST REPORT FORMAT FOR TYPE EVALUATION**

### **9 TEST REPORT FORMAT**

#### **9.1 Introduction**

This Report Format applies for any kind of protein measuring instrument for grain (independent of its technology). It presents a standardized format for the results of the various tests and examinations, described in Part 2 clause 0 of OIML R xxx (20xx), to which a type of protein measuring instrument for grain shall be submitted with a view to its approval based this OIML Recommendation.

It is recommended that all metrology services or laboratories evaluating and/or testing types of protein measuring instrument for grain according to OIML R xxx (20xx), or to national or regional regulations based on that Recommendation, use this Report Format, directly or after translation into a language other than English or French. In case of a translation, it is highly recommended to leave the structure and the numbers of the clauses unchanged: in this case most of the contents are also understandable for those who can not read the language of the translation.

It is also recommended that this Report Format in English or in French (or in both languages) be transmitted by the country performing the tests to the relevant authorities of another country, when requested for issuing a national or regional type-approval.

In the practical application of the Report Format, in addition to a cover page by the Issuing Authority, as a minimum clauses XXX- XXX (as necessary) shall be included

#### **9.2 Applicability of this Test Report Format**

In the framework of the *OIML Certificate System for Measuring Instruments*, and the *OIML Mutual Acceptance Arrangement (MAA)* applicable to protein measuring instrument for grain in conformity with OIML R xxx (20xx), use of this report format is mandatory, in French and/or in English with translation into the national languages of the countries issuing such certificates, if appropriate.

Implementation of this Report Format is informative with regard to the implementation of OIML Recommendation OIML R xxx (20xx) in national regulations.

### 9.3 Guidance for the application of this Test Report Format

Meaning of symbols where used

EUT:	==equipment under test
$\Delta$ :	difference in measured value and reference value (measured at reference test conditions).
$m$ :	the “as is” moisture content of a sample determined using a moisture meter (the meter may be a component of the protein measuring instrument).
$M_B$ :	the basis moisture concentration, expressed as a percentage by mass, specified by the national responsible body for reporting protein content of the particular grain type.
$P_{MB}$ :	the protein content of a grain sample, expressed as a percentage by mass, calculated at the moisture basis
MPE:	maximum permissible error
$n_m$ :	number of measurements
$n_s$ :	number of samples
$U$ :	test voltage
$V$ :	voltage

The name(s) or symbol(s) of the unit(s) used to express test results shall be specified in each form.

For each test, the “SUMMARY OF TYPE EVALUATION” and the “TYPE EVALUATION CHECKLIST” shall be completed according to this example:

PASSED	FAILED	Remarks
X		
	X	
NA	NA	

when the instrument has passed the test or inspection:

when the instrument has failed the test or inspection:

when the test or inspection is not applicable:

The white spaces in the headings of the report should always be filled in according to the following example:

	At start	At end	
Temp:	20.1	20.3	°C
RH:			%
Date:			yy:mm:dd
Time:			hh:mm:ss

Where:

Temp: temperature

RH: relative humidity

Date / time the date and time that the test is performed

In case a prescribed test is not relevant for the type of instrument to be tested, the reason why the test is omitted shall be clearly stated in the field “Remarks”

The number of the report and the page numbers shall be completed in the heading.

#### 9.4 Authority, responsible for this Report

Name	
Address	
Report number	
Application number	
Period of tests	
Date of issuing this Report	
Name and signature of the responsible person	
Stamp(s) (if applicable)	

#### 9.5 Synopsis of the results of the examination and tests

The tested specimen fulfils ALL the applicable requirements in OIML R XXX (200x)	
<input type="checkbox"/>	Yes
<input type="checkbox"/>	No
Remarks:	



## 9.6 Summary of the results of the examination and tests

### 9.6.1 Visual examinations

Clause(s) in R xxx	Metrological and technical requirements	PASSED	FAILED	Location of details in Report
3	Units of measurement			9.9
4	Metrological requirements			
4.1	Measuring ranges			9.9
4.4	Whole-grain measurement standards			9.9
4.6	Accuracy and precision requirements for the type at reference conditions	Refer to 9.6.2 checklist		
4.7	Influence factors	Refer to 9.6.2 checklist		
4.8	Disturbance tests for electronic instruments	Refer to 9.6.2 checklist		
5	Technical requirements			
5.1	Applicable grain types and protein content ranges			9.9
5.2	Sample preparation and input			
5.3	Instrument construction			9.9
5.4	Instrument warm up period	Refer to 9.6.2 checklist		
5.5	Level indicating means	Refer to 9.6.2 checklist		
5.6	Presentation of the measured value			9.9
5.7	Durable recording of measured values			9.9
5.8	Checking facilities			9.9
5.9	Operational safeguards			9.9
5.10	Software-controlled electronic devices and security			9.9
5.11	Manufacturers manual			9.9
5.12	Markings			9.9
5.13	Suitability			9.9

### 9.6.2 Type evaluation tests

Clause(s) in R xxx	Type approval tests	PASSED	FAILED	Location of details in Report
8.2.1	Accuracy			9.10.3
8.2.2	Repeatability			9.10.4
8.2.3	Reproducibility			9.10.5
8.3.1	Instrument drift and instability			9.11.4
8.3.2	Instrument warm-up time			9.11.5
8.3.3	Instrument levelling			9.11.6
8.3.4	Sample temperature sensitivity			9.11.8
8.3.5	Storage temperature (extreme shipping conditions)			9.11.7
8.3.6	Dry heat			9.11.9
8.3.7	Cold			9.11.10
8.3.8	Damp heat, steady-state (non-condensing)			9.11.11
8.3.9	Dust ingress			9.11.12
8.3.10	AC mains voltage variation			9.11.13
8.4.1	AC mains voltage dips, short interruptions and voltage variations			9.12.3
8.4.2	Bursts (transients) on AC mains			9.12.4
8.4.3	Radiated radiofrequency, electromagnetic susceptibility			9.12.5
8.4.4	Conducted radio-frequency fields			9.12.6
8.4.5	Electrostatic discharges			9.12.7
8.4.6	Mechanical shock			9.12.8

Note that all type approval test samples required to pass the following tests in order for the test result to be valid.

Clause(s) in R xxx	Sample tests	PASSED	FAILED	Complete	Location of details in Report
7.4	Homogeneity evaluation – accuracy and precision tests				9.10.2
	Homogeneity evaluation and initial intrinsic error – influence tests				9.11.2
	Homogeneity evaluation – disturbance tests*				9.12.2
7.5	Sample stability - dry heat test				9.11.3.1
	Sample stability - cold test				9.11.3.2
	Sample stability - damp heat steady state test				9.11.3.3

\*Not required if same sample used is from set used the influence tests.

## 9.7 General information about the application

### 9.7.1 Manufacturer

Company	
Address	

### 9.7.2 Applicant

Company	
Representative	
Address	
Reference	
Date of application	
Applicant authorized by the manufacturer (documented)	<input type="checkbox"/> Yes <input type="checkbox"/> No
Statement that no concurrent application for OIML type evaluation has been made to any other OIML Issuing Authority (see OIML B 003, 3.1.2)	<input type="checkbox"/> Yes <input type="checkbox"/> No

Remarks:
----------

**9.7.3 Information concerning the type**

and the sample(s) supplied for the tests  
(as stated on the instrument/provided by the manufacturer)

Information, indicated on the instrument	
Manufacturer's trade mark	
Year of manufacture	
Type designation	
Model number ( <i>if applicable</i> )	
Serial number(s) of the sample(s)	
Type description of the <u>main</u> transducer	
Serial number of the <u>measuring</u> transducer	
etc	
etc	
Electrical power	
Identification of software	

Relevant external/internal photographs taken during the examination and tests:

Remarks:

#### 9.7.4 Accessories, supplied by the applicant

Battery (if applicable)	type	
	nominal voltage	
	number required	
Operating instructions		
Data printer (if applicable)		
Data storage		
Cables		
etc		
etc		
Other accessories:		

#### 9.7.5 Additional information concerning the type

Additional remarks and/or information (connection equipment, interfaces, etc.):

#### 9.7.6 Results of previous tests that were taken into account

Details:

## 9.8 Test samples and equipment

### 9.8.1 Provision of sample selection guidelines by national responsible body

Indicate grain types to be used to test the submitted type in Table 5 column 5.

**Table 5 Protein content ranges for type evaluation**

Grain Type*	$P_{MB}$ Range (%)*	$M_B$ (%)*	$m$ range (%)	Used for type approval Indicate Y or N
Durum Wheat	10 - 18	12	Refer to OIML R 59 [7] for guidance	
Hard Red Spring Wheat	10 - 19			
Hard Red Winter Wheat	8 - 18			
Hard White Wheat	9 - 16			
Soft Red Winter Wheat	9 - 12			
Soft White Wheat	8 - 15			
"All Class" Wheat Calibration	8 - 19			
Two-rowed Barley	8 - 17	0		
Six-rowed Barley	8 - 17			
"All Class" Barley Calibration	8 - 17			
Corn	8 - 12			
Soybeans	30 - 40	13		
Canola				
Lupin				
Column 1	Column 2	Col 3	Column 4	Column 5

\*Listed values are for example only. The national responsible body may select grain types and constituent ranges in accordance with clause 5.1.

### 9.8.2 Selection of sample(s)

9.8.2.1 Justification for samples used:

9.8.2.2 Reference method used:

9.8.2.3 Adjustments, modifications, and repairs made to the samples during the testing:

**9.8.3 Testing laboratories involved in the tests**

(This table to be completed for each test laboratory)

Name			
Address			
Application number			
Tests by this laboratory			
Date/period of tests			
Name(s) of test engineer(s)			
Accredited by		Number:	Expires (date):
Accreditation includes R XXX	<input type="checkbox"/> Yes	Edition: <input type="text"/>	<input type="checkbox"/> No
Details of relevant peer assessment or assessment by other means			
In case tests have been performed on an other location than the premises of this laboratory, give details here			
Name of the responsible person			
Date of signature			
Stamp (if applicable) and signature of the responsible person			

Remarks:

#### **9.8.4 Information concerning the test equipment used for the type evaluation**

(including details of simulations and the way uncertainties are taken into account, including the level of "risk" like for instance 95% or  $k=2$ )



### **9.9 Visual examination details**

Section to be completed once sections on Metrological and Technical Requirements have been finalised

## 9.10 Report format for accuracy and precision tests

### 9.10.1 Accuracy, repeatability and reproducibility test samples

List sample ID of whole-grain CRMs to be used under the relevant 2%  $P_{MB}$  and 2%  $m$  interval

Grain type*		$P_{MB}$ (%)*	$m$ (%)*		
Number	Name		6-8	8-10	10-12
1	Soft white wheat	9-11	Sample A Sample B Sample C	Sample D Sample E Sample F	Sample G Sample H Sample I
		11-13	Sample J Sample K Sample L	etc.	etc.
		13-15	etc.	etc.	etc.
			6-8	8-10	10-12
2	Barley	10-12	etc.	etc.	etc.
		12-14	etc.	etc.	etc.
		14-16	etc.	etc.	etc.
			12-14	14-16	16-18
3	Corn	8-10	etc.	etc.	etc.
		10-12	etc.	etc.	etc.
		12-14	etc.	etc.	etc.

\*Listed values are for example only. The national responsible body may select grain types and constituent ranges in accordance with clause 5.1.

Attach certificates or list sample properties determined by the reference method. Include measurement uncertainty.

Sample ID	Reference method analysis		
	Date	$P_{MB}$ (%)	$m$ (%)
Sample A			
Sample B			
etc.			

### 9.10.2 Homogeneity evaluation – accuracy and precision tests

Reference test the method applied to the whole-grain CRMs to confirm homogeneity.

### 9.10.3 Accuracy test

Observer:	
Instrument 1 ID	
Instrument 2 ID	

	At start	At end	
Temp:			°C
RH:			%
Date & Time:			yy:mm:dd hh:mm:ss

Instrument	Grain type*	Consecutive 2% $P_{MB}$ intervals*	Sample ID $n_s$ per $P_{MB}$ int = 9	Results													
				EUT $P_{MB}$ $n_m$ per sample=3			CRM Ref $P_{MB}$	Error at ref conditions		MPE {4.5} Table 1 col 4	PASSED	FAILED	Remarks				
				1	2	3		$\bar{y}$	SDD								
1	Soft white wheat	9-11	Sample A														
			Sample B														
			Sample C														
			Sample D														
			Sample E														
			Sample F														
			Sample G														
			Sample H														
			Sample I														
		11-13	Sample J														
	Sample K																
	Sample L																
	etc.																
	etc.																
	etc.																
	13-15	etc.															
etc.																	
etc.																	
	Barley	12-14	$n_s=9$	$n_m=3$													
14-16		$n_s=9$	$n_m=3$														
16-18		$n_s=9$	$n_m=3$														
	Corn	8-10	$n_s=9$	$n_m=3$													
10-12		$n_s=9$	$n_m=3$														
12-14		$n_s=9$	$n_m=3$														
2	SWW	9-11															
		11-13															
		13-15															
		Barley	12-14														
	14-16																
	16-18																
		Corn	8-10														
	10-12																
	12-14																

\*Listed values are for example only. The national responsible body may select grain types and constituent ranges in accordance with clause 5.1.

$$\bar{y} = \frac{\sum_{i=1}^n (\bar{x}_i - r_i)}{n}$$

$$SDD = \sqrt{\frac{\sum_{i=1}^n (y_i - \bar{y})^2}{n-1}}$$

$\bar{y}$  = average over all  $y_i$   
 $y_i = \bar{x}_i - r_i$   
 $\bar{x}_i$  = average measured  $P_{MB}$  for sample  $i$  (3 replicates)  
 $r_i$  = CRM reference  $P_{MB}$  for sample  $i$   
 $n$  = number of samples per 2%  $P_{MB}$  interval ( $n = 9$ )

Check if $\bar{y}$ and SDD $\leq$  MPE	<input type="checkbox"/> Passed	<input type="checkbox"/> Failed
--	---------------------------------	---------------------------------

### 9.10.4 Repeatability test

Observer:	
Instrument 1 ID	
Instrument 2 ID	

	At start	At end	
Temp:			°C
RH:			%
Date & Time:			yy:mm:dd hh:mm:ss

Instrument	Grain type*	Consecutive 2% $P_{MB}$ intervals*	Sample ID $n_s$ per $P_{MB}$ int = 9	Results												
				EUT $P_{MB}$ $n_m$ per sample=3			Standard deviation SD		MPE {4.5} Table 1 col 2	PASSED	FAILED	Remarks				
				1	2	3	Sample	Pooled								
1	Soft white wheat	9-11	Sample A													
			Sample B													
			Sample C													
			Sample D													
			Sample E													
			Sample F													
			Sample G													
			Sample H													
			Sample I													
			11-13	Sample J												
		Sample K														
		Sample L														
		Barley	12-14	etc.												
	etc.															
	etc.															
	14-16		$n_s=9$	$n_m=3$												
			16-18	$n_s=9$	$n_m=3$											
				Corn	8-10	$n_s=9$	$n_m=3$									
	10-12	$n_s=9$			$n_m=3$											
		12-14	$n_s=9$		$n_m=3$											
2			SWW	9-11												
	11-13															
	13-15															
		Barley	12-14													
	14-16															
	16-18															
		Corn	8-10													
	10-12															
	12-14															

\*Listed values are for example only. The national responsible body may select grain types and constituent ranges in accordance with clause 5.1.

$$SD = \sqrt{\frac{\sum_{i=1}^n \sum_{j=1}^3 (x_{ij} - \bar{x}_i)^2}{2n}}$$

$x_{ij}$  = measured  $P_{MB}$  value for sample  $i$  and replicate  $j$ ,  
 $\bar{x}_i$  = average of the three  $P_{MB}$  for sample  $i$  (3 replicates),  
 $n$  = number of samples per 2%  $P_{MB}$  interval ( $n = 9$ )

Check if $SD \leq  MPE $	<input type="checkbox"/> Passed	<input type="checkbox"/> Failed
--------------------------	---------------------------------	---------------------------------

**9.10.5 Reproducibility test**

Observer:	
Instrument 1 ID	
Instrument 2 ID	

	At start	At end	
Temp:			°C
RH:			%
Date & Time:			yy:mm:dd hh:mm:ss

Grain type*	Consecutive 2% $P_{MB}$ intervals*	Sample ID $n_s$ per $P_{MB}$ int = 9	Results										MPE {4.5} Table 1 col 3	PASSED	FAILED	Remarks
			EUT $P_{MB}$ $n_m$ per sample=3						Reproducibility measure							
			1			2			$d_i$	mean $d_i$	$SDD_1$					
			1	2	3	1	2	3								
Soft white wheat	9-11	Sample A														
		Sample B														
		Sample C														
		Sample D														
		Sample E														
		Sample F														
		Sample G														
		Sample H														
		Sample I														
	11-13	Sample J														
		Sample K														
		Sample L														
		etc.														
		etc.														
		etc.														
13-15	etc.															
	etc.															
	etc.															
	etc.															
	etc.															
	etc.															
Barley	12-14	$n_s=9$	$n_m=3$			$n_m=3$										
	14-16	$n_s=9$	$n_m=3$			$n_m=3$										
	16-18	$n_s=9$	$n_m=3$			$n_m=3$										
Corn	8-10	$n_s=9$	$n_m=3$			$n_m=3$										
	10-12	$n_s=9$	$n_m=3$			$n_m=3$										
	12-14	$n_s=9$	$n_m=3$			$n_m=3$										

\*Listed values are for example only. The national responsible body may select grain types and constituent ranges in accordance with clause 5.1.

$$SDD_1 = \sqrt{\frac{\sum_{i=1}^n (d_i - \bar{d})^2}{n-1}}$$

$$d_i = \bar{x}_i^{(1)} - \bar{x}_i^{(2)}$$

$\bar{x}_i^{(1)}$  = mean of three  $P_{MB}$  replicates for sample  $i$  on instrument 1

$\bar{x}_i^{(2)}$  = mean of three  $P_{MB}$  replicates for sample  $i$  on instrument 2

$\bar{d}$  = mean of the  $d_i$

$n$  = number of samples in all 2%  $P_{MB}$  ranges

Check if $SDD_1 \leq  MPE $	<input type="checkbox"/> Passed	<input type="checkbox"/> Failed
-----------------------------	---------------------------------	---------------------------------

## 9.11 Report format for influence factor tests

### 9.11.1 Samples for influence factor tests

List ID of samples to be used under the relevant 2%  $P_{MB}$  and 2%  $m$  intervals

Grain type*		$P_{MB}$ (%)*	$m$ (%)*		
Number	Name		6-8	8-10	10-12
1	Soft white wheat	9-11	Sample AA		
		11-13			Sample AB
		13-15		Sample AC	
			6-8	8-10	10-12
2	Barley	10-12			Sample AD
		12-14		Sample AE	
		14-16	Sample AF		
			12-14	14-16	16-18
3	Corn	8-10		etc.	
		10-12	etc.		
		12-14			etc.

\*Listed values are for example only. The national responsible body may select grain types and constituent ranges in accordance with clause 5.1.

Attach certificates or list sample properties determined by the reference method or a master instrument. Include measurement uncertainty.

Sample ID	Reference method analysis			OR Master instrument analysis		
	Date	$P_{MB}$ (%)	$m$ (%)	Date	$P_{MB}$ (%)	$m$ (%)
Sample AA						
Sample AB						
etc.						

### 9.11.2 Homogeneity evaluation and initial intrinsic error – influence tests

Sample ID	Summary of 6 $P_{MB}$ measurements				MPE max-min {9.10.2}	PASSED	FAILED	Remarks
	mean $P_{MB}$ prior to tests	min	max	max-min difference				
Sample AA								
Sample AB								
etc.								

### 9.11.3 Sample stability evaluation

Report is to be completed simultaneously with the corresponding influence factor test. Values in fields marked with asterisks (\*) are for example only, but must correspond with the samples in the corresponding influence tests.

#### 9.11.3.1 Sample stability - dry heat test

Instrument	Grain type*	Consecutive 2% $P_{MB}$ intervals*	Reference conditions $P_{MB}$				Sample stability correction factor	
			Prior to test (ref)		After high temp test		Diff mean $P_{MB}$ HT& Prior to test	Remarks
			$n_m=10$	mean	$n_m=10$	mean		
2	Soft white wheat	9-11						
		11-13						
		13-15						

#### 9.11.3.2 Sample stability - cold test

Instrument	Grain type*	Consecutive 2% $P_{MB}$ intervals*	Reference conditions $P_{MB}$				Sample stability correction factor	
			Prior to test (ref)		After low temp test		Diff mean $P_{MB}$ LT& Prior to test	Remarks
			$n_m=10$	mean	$n_m=10$	mean		
2	Soft white wheat	9-11						
		11-13						
		13-15						

9.11.3.3 Sample stability - damp heat steady state test

Instrument	Grain type*	Consecutive 2% $P_{MB}$ intervals*	Reference conditions $P_{MB}$				Sample stability correction factor		
			Prior to test (ref)		After damp heat test		Diff mean $P_{MB}$ DH & Prior to test	Remarks	
			$n_m=10$	mean	$n_m=10$	mean			
2	Soft white wheat	9-11							
		11-13							
		13-15							



**9.11.4 Instrument drift and instability**

Observer:	
Instrument 1 ID	
Instrument 2 ID	

Stage 1	At start	At end	
Temp:			°C
RH:			%
Date & Time:			yy:mm:dd hh:mm:ss

Observer:	
-----------	--

Stage 2	At start	At end	
Temp:			°C
RH:			%
Date & Time:			yy:mm:dd hh:mm:ss

Instrument	Grain type*	Consecutive 2% $P_{MB}$ intervals*	EUT $P_{MB}$				Results summary				
			Prior to influence and disturbance tests (Stage 1)		After test program (Stage 2)		Diff mean $P_{MB}$	Error Shift Limit {4.5} Table 1 col 5	PASSED	FAILED	Remarks
			$n_m$ per sample=5	mean	$n_m$ per sample=5	mean					
1	Soft white wheat	9-11									
		11-13									
		13-15									
2	Soft white wheat	9-11									
		11-13									
		13-15									

\*Listed values are for example only. The national responsible body may select grain types and constituent ranges in accordance with clause 5.1.

Check if $\Delta \text{ mean } P_{MB} \leq \text{Error Shift Limit}$ All instrument functions operated as designed	<input type="checkbox"/> Passed <input type="checkbox"/> Failed
---	---

**9.11.5 Instrument warm-up time**

Observer:	
Instrument 1 ID	
Instrument 2 ID	
Sample ID	

	At start	At end	
Temp:			°C
RH:			%
Date & Time:			yy:mm:dd hh:mm:ss

Instrument	EUT $P_{MB}$				Results summary				
	After warm up		1 hour after being switched on or 2x the warm up time (whichever is greater)		Diff mean $P_{MB}$	Error Shift Limit {4.5} Table 1 col 5	PASSED	FAILED	Remarks
	$n_m$ per sample=5	mean	$n_m$ per sample=5	mean					
1									
2									

Check if $\Delta \text{mean } P_{MB} \leq \text{Error Shift Limit}$ All instrument functions operated as designed	<input type="checkbox"/> Passed <input type="checkbox"/> Failed
--	---

**9.11.6 Instrument levelling**

Observer:	
Instrument ID	
Sample ID	
Level indicator	Y / N

	At start	At tilt	At end	
Temp:				°C
RH:				%
Date & Time:				yy:mm:dd hh:mm:ss
Degree of tilt*				°

\*Without a level indicator, tilt to 5%. With a level indicator, tilt instrument to the limits of the indicator

Instrument	Orientation	EUT $P_{MB}$				Results summary				
		During tilt position		Returned to reference level		Diff mean $P_{MB}$ (ref-tilt)	Error Shift Limit {4.5} Table 1 col 5	PASSED	FAILED	Remarks
		$n_m$ per sample=5	mean	$n_m$ per sample=5	mean					
1	Reference level									
	Left or right tilt*									
	Front or back tilt*									

\*Choose direction with the most influence

Check if $\Delta \text{ mean } P_{MB} \leq \text{Error Shift Limit}$ All instrument functions operated as designed	<input type="checkbox"/> Passed <input type="checkbox"/> Failed
---	---

**9.11.7 Storage temperature**

Observer:	
Instrument ID	
Sample ID	

	At start	At end	
Temp:			°C
RH:			%
Date & Time:			yy:mm:dd hh:mm:ss

Note details about the influence settings (e.g. heating/cooling cycles here):

Instrument	EUT $P_{MB}$				Results summary				
	Before temperature cycles		After temperature cycles		Diff mean $P_{MB}$	Error Shift Limit {4.5} Table 1 col 5	PASSED	FAILED	Remarks
	$n_m$ per sample=5	mean	$n_m$ per sample=5	mean					
1									

Check if  $\Delta \text{mean } P_{MB} \leq \text{Error Shift Limit}$   
 All instrument functions operated as designed

Passed       Failed

**9.11.8 Sample temperature sensitivity**

Observer:	
Instrument 1 ID	
Instrument 2 ID	
$\Delta T_C$	
$\Delta T_H$	

	At start	At end	
Temp:			°C
RH:			%
Date & Time:			yy:mm:dd hh:mm:ss

Note details about the influence settings (e.g. sample heating/cooling cycles) here:

Instrument	Grain type*	Consecutive 2% $P_{MB}$ intervals*	EUT $P_{MB}$ at sample temp $n_s=2$ (duplicates), $n_m=3$									Results summary					
			Reference (22°C)			COLD Ref $-\Delta T_C$			HOT Ref $+\Delta T_H$			Diff mean $P_{MB}$		Error Shift Limit {4.5} Table 1 col 6	PASSED	FAILED	Remarks
			1	2	mean	1	2	mean	1	2	mean	COLD Ref and Ref $-\Delta T_C$	HOT Ref and Ref $+\Delta T_H$				
1	SW wheat	9-11															
		11-13															
		13-15															
	Barley	10-12															
		12-14															
		14-16															
	Corn	8-10															
		10-12															
		12-14															
2	SW wheat	9-11															
		11-13															
		13-15															
	Barley	10-12															
		12-14															
		14-16															
	Corn	8-10															
		10-12															
		12-14															

\*Listed values are for example only. The national responsible body may select grain types and constituent ranges in accordance with clause 5.1.

Check if $\Delta$ mean $P_{MB} \leq$ Error Shift Limit All instrument functions operated as designed	<input type="checkbox"/> Passed <input type="checkbox"/> Failed
---	---

**9.11.9 Dry Heat**

Observer:	
Instrument 1 ID	
Instrument 2 ID (sample stability)	

		Influence				
		At start	Start	End	At end	
Temp:						°C
RH:						%
Date & Time:						yy:mm:dd hh:mm:ss

Note other details about the influence settings (e.g. heating/recovery cycle) here:

Instrument	Grain type*	Consecutive 2% $P_{MB}$ intervals*	EUT $P_{MB}$						Results summary						
			Prior to test (ref)		High temp test		Recov		Diff mean $P_{MB}$			Error Shift Limit {4.5} Table 1 col 5	PASSED	FAILED	Remarks
			$n_m=10$	mean	$n_m=10$	mean	$n_m=10$	mean	HT & Ref	Stability corrected HT& Ref	Recov & Ref				
1	Soft white wheat	9-11													
		11-13													
		13-15													

\*Listed values are for example only. The national responsible body may select grain types and constituent ranges in accordance with clause 5.1.

Report format for calculating the sample stability correction factor to be deducted from the EUT  $P_{MB}$  error shift (HT – Ref) is located in clause 9.11.3.1

Check if $\Delta$ mean $P_{MB} \leq$ Error Shift Limit All instrument functions operated as designed Ensure that stability correction is applied to EUT result	<input type="checkbox"/> Passed <input type="checkbox"/> Failed
--	---

**9.11.10 Cold**

Observer:	
Instrument 1 ID	
Instrument 2 ID (sample stability)	

		Influence				
		At start	Start	End	At end	
Temp:						°C
RH:						%
Date & Time:						yy:mm:dd hh:mm:ss

Note other details about the influence settings (e.g. cooling/recovery cycle) here:

Instrument	Grain type*	Consecutive 2% $P_{MB}$ intervals*	EUT $P_{MB}$						Results summary									
			Prior to test (ref)		Low temp test		Recov		Diff mean $P_{MB}$			Error Shift Limit {4.5} Table 1 col 5	PASSED	FAILED	Remarks			
			$n_m=10$	mean	$n_m=10$	mean	$n_m=10$	mean	LT & Ref	Stability corrected LT & Ref	Recov & Ref							
1	Soft white wheat	9-11																
		11-13																
		13-15																

\*Listed values are for example only. The national responsible body may select grain types and constituent ranges in accordance with clause 5.1.

Report format for calculating the sample stability correction factor to be deducted from the EUT  $P_{MB}$  error shift (LT – Ref) is located in clause 9.11.3.2

Check if $\Delta$ mean $P_{MB} \leq$ Error Shift Limit All instrument functions operated as designed Ensure that stability correction is applied to EUT result	<input type="checkbox"/> Passed <input type="checkbox"/> Failed
--	---

**9.11.11 Damp heat steady state**

Observer:	
Instrument 1 ID	
Instrument 2 ID (sample stability)	

	Influence			
	At start	Start	End	At end
Temp:				°C
RH:				%
Date & Time:				yy:mm:dd hh:mm:ss

Note other details about the influence settings (e.g. heating/recovery cycle) here:

Instrument	Grain type*	Consecutive 2% $P_{MB}$ intervals*	EUT $P_{MB}$						Results summary						
			Prior to test (ref)		Damp heat test		Recov		Diff mean $P_{MB}$			Error Shift Limit {4.5} Table 1 col 5	PASSED	FAILED	Remarks
			$n_m=10$	mean	$n_m=10$	mean	$n_m=10$	mean	DH & Ref	Stability corrected DH& Ref	Recov & Ref				
1	Soft white wheat	9-11													
		11-13													
		13-15													

\*Listed values are for example only. The national responsible body may select grain types and constituent ranges in accordance with clause 5.1.

Report format for calculating the sample stability correction factor to be deducted from the EUT  $P_{MB}$  error shift (DH – Ref) is located in clause 9.11.3.3

Check if $\Delta \text{ mean } P_{MB} \leq \text{Error Shift Limit}$ All instrument functions operated as designed Ensure that stability correction is applied to EUT result	<input type="checkbox"/> Passed <input type="checkbox"/> Failed
--	---





**9.11.13 Mains voltage variations**

Observer:	
Instrument ID	
Sample ID	

	Influence			At end $U_{nom}$	
	At start $U_{nom}$	$U_{nom} + 10\%$	$U_{nom} - 15\%$		
Temp:					°C
RH:					%
Date & Time:					yy:mm:dd hh:mm:ss
Test voltage					V
Test frequency					Hz

Note other details about the influence settings here:

EUT $P_{MB}$								Results summary						
Prior test		Over V $U_{nom} + 10\%$		Under V $U_{nom} - 15\%$		Recovery		Diff mean $P_{MB}$ $U_{nom}$ &-			Error Shift Limit {4.5} Table 1 col 5	PASSED	FAILED	Remarks
$n_m = 10$	mean	$n_m = 10$	mean	$n_m = 10$	mean	$n_m = 10$	mean	over V	under V	recovery				

Check if  $\Delta \text{mean } P_{MB} \leq \text{Error Shift Limit}$   
 All instrument functions operated as designed

Passed       Failed

## 9.12 Report format for disturbance tests

### 9.12.1 Samples for disturbance tests

If not already supplied in clause 9.11.1, provide details for the grain sample to be used in disturbance tests.

Attach certificates or list sample properties determined by the reference method or a master instrument. Include measurement uncertainty.

Sample ID	Grain type	Reference method analysis			OR Master instrument analysis		
		Date	$P_{MB}$ (%)	$m$ (%)	Date	$P_{MB}$ (%)	$m$ (%)
Sample X							

### 9.12.2 Homogeneity evaluation – disturbance tests

Sample ID	Summary of 6 $P_{MB}$ measurements				MPE max-min {9.10.2}	PASSED	FAILED	Remarks
	mean $P_{MB}$ prior to tests	min	max	max-min difference				
Sample X								

**9.12.3 AC mains voltage dips and short interruptions**

Observer:	
Instrument ID	
Sample ID	

	At start	At end	
Temp:			°C
RH:			%
Date & Time:			yy:mm:dd hh:mm:ss

	$n_m=10$	mean
Reference $P_{MB}$		
No disturbance		
Error Shift Limit {4.5}		
Table 1 col 5		

Note other details about the disturbance settings (voltage cycling) here:

Settings				Results				
Test	Voltage reduction		Duration (cycles)	EUT $P_{MB}$ $n_m=10$	$\Delta P_{MB}$ (Measured - ref)	$\Delta P_{MB} <$ Error Shift Limit	Sig fault detected & acted upon	Remarks
	new V	% reduction						
a	0	100	0.5					
b	0	100	1					
c		70	25 / 30					
d	0	100	250 / 300					

Check if: $\Delta P_{MB} \leq$ Error Shift Limit Checking facility detected and acted upon significant fault All instrument functions operated as designed	<input type="checkbox"/> Passed <input type="checkbox"/> Failed
--	---

**9.12.4 Electrical Bursts**

Observer:	
Instrument ID	
Sample ID	

	At start	At end	
Temp:			°C
RH:			%
Date & Time:			yy:mm:dd hh:mm:ss

L = phase, N = neutral,  
PE = protective earth, G = ground

Note other details about the disturbance settings here:

Error Shift Limit {4.5}	
Table 1 col 5	

Settings		EUT $P_{MB}$			Results summary			
Connection	Test V (kV) & Polarity	Prior to disturbance (reference)		Test	$\Delta P_{MB}$ (Measured - ref)	$\Delta P_{MB} <$ Error Shift Limit	Sig fault detected & acted upon	Remarks
		$n_m=10$	mean	$n_m=10$				
L ↓ G	+1							
	-1							
N ↓ G	+1							
	-1							
PE ↓ G	+1							
	-1							

Check if: $\Delta P_{MB} \leq$ Error Shift Limit Checking facility detected and acted upon significant fault All instrument functions operated as designed	<input type="checkbox"/> Passed <input type="checkbox"/> Failed
--	---

**9.12.5 Radiated, radio-frequency, electromagnetic fields**

Observer:	
Instrument ID	
Sample ID	

	At start	At end	
Temp:			°C
RH:			%
Date & Time:			yy:mm:dd hh:mm:ss

Note: V = vertical, H = horizontal

Note other details about the disturbance settings here:

Error Shift Limit {4.5}	
Table 1 col 5	

Settings		$P_{MB}$ prior disturbance (reference)
EUT side	Antenna polarisation	$n_m=10$
Front	V	
		mean
	H	$n_m=10$
Left	V	$n_m=10$
	H	$n_m=10$
Right	V	$n_m=10$
	H	$n_m=10$
Rear	V	
	H	

Test		Results summary			
Frequency (MHz)	EUT $P_{MB}$	$\Delta P_{MB}$ (Measured - ref)	$\Delta P_{MB} <$ Error Shift Limit	Sig fault detected & acted upon	Remarks
26					
2000					
26					
	2000				
26					
	2000				
26					
	2000				
26					
	2000				
26					
	2000				

Check if: $\Delta P_{MB} \leq$ Error Shift Limit Checking facility detected and acted upon significant fault All instrument functions operated as designed	<input type="checkbox"/> Passed <input type="checkbox"/> Failed
--	---



### 9.12.7 Electrostatic discharge

#### 9.12.7.1 Direct application

Observer:	
Instrument ID	
Sample ID	

	At start	At end	
Temp:			°C
RH:			%
Date & Time:			yy:mm:dd hh:mm:ss

Contact discharge (Y / N)  
 Paint penetration (Y / N)  
 \*Air discharge (Y / N)  
 Error Shift Limit {4.5}  
 Table 1 col 5

Note other details about the disturbance settings (e.g. repeat interval) here:

Settings		EUT $P_{MB}$			Results summary			
Test V (kV)	Polarity	Prior to disturbance (reference)		Test	$\Delta P_{MB}$ (Measured - ref)	$\Delta P_{MB} <$ Error Shift Limit	Sig fault detected & acted upon	Remarks
		$n_m=10$	mean	$n_m=10$				
2	+							
	-							
4	+							
	-							
6	+	$n_m=10$		$n_m=10$				
	-	$n_m=10$		$n_m=10$				
8*	+	$n_m=10$		$n_m=10$				
	-	$n_m=10$		$n_m=10$				

Check if: $\Delta P_{MB} \leq$ Error Shift Limit Checking facility detected and acted upon significant fault All instrument functions operated as designed	<input type="checkbox"/> Passed <input type="checkbox"/> Failed
--	---



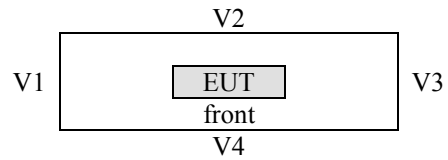
9.12.7.2 Indirect application

Observer:	
Instrument ID	
Sample ID	

	At start	At end	
Temp:			°C
RH:			%
Date & Time:			yy:mm:dd hh:mm:ss

Notes: H = horizontal, V = vertical  
Refer to diagram for vertical coupling plane positions

Polarity (+ / -)  
Error Shift Limit {4.5}  
Table 1 col 5



Settings		EUT $P_{MB}$			Results summary			
Coupling plane position	Test V (kV)	Prior to disturbance (reference)		Test	$\Delta P_{MB}$ (Measured - ref)	$\Delta P_{MB} <$ Error Shift Limit	Sig fault detected & acted upon	Remarks
		$n_m=10$	mean	$n_m=10$				
H	2							
V1	4	$n_m=10$		$n_m=10$				
	6	$n_m=10$		$n_m=10$				
	2	$n_m=10$		$n_m=10$				
V2	4	$n_m=10$		$n_m=10$				
	6	$n_m=10$		$n_m=10$				
	2	$n_m=10$		$n_m=10$				
V3	4	$n_m=10$		$n_m=10$				
	6	$n_m=10$		$n_m=10$				
	2	$n_m=10$		$n_m=10$				
V4	4	$n_m=10$		$n_m=10$				
	6	$n_m=10$		$n_m=10$				
	2	$n_m=10$		$n_m=10$				

Check if: $\Delta P_{MB} \leq$ Error Shift Limit Checking facility detected and acted upon significant fault All instrument functions operated as designed	<input type="checkbox"/> Passed <input type="checkbox"/> Failed
--	---

**9.12.8 Mechanical shock**

Observer:	
Instrument ID	
Sample ID	

	At start	At end	
Temp:			°C
RH:			%
Date & Time:			yy:mm:dd hh:mm:ss

Note other details about the disturbance settings here:

Error Shift Limit {4.5} Table 1 col 5	
--	--

EUT $P_{MB}$			Results summary			
Prior to disturbance (reference)		After disturbance	$\Delta P_{MB}$ (Measured - ref)	$\Delta P_{MB} <$ Error Shift Limit	Sig fault detected & acted upon	Remarks
$n_m=10$	mean	$n_m=10$				

Check if: $\Delta P_{MB} \leq$ Error Shift Limit Checking facility detected and acted upon significant fault All instrument functions operated as designed	<input type="checkbox"/> Passed <input type="checkbox"/> Failed
--	---