

**Australian Government** 

National Measurement Institute

## NMI M 8

## Pattern Approval Specifications for Protein Measuring Instruments for Grain

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#### PREFACE

In the absence of a recommendation of the International Organisation on Legal Metrology (OIML) on grain protein measurement, this national standard is based on the generic requirements of *OIML D 11. General Requirements for Electronic Measuring Instruments* and current practice within the grain industry in Australia. Reference has also been made to Publication 14 on near infrared grain analysers published by the National Conference on Weights and Measures in the USA.

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## SECTION I — GENERAL

## 1 SCOPE

This document applies to protein measuring instruments used for trade at grain receival and other sites. It specifies the metrological and technical requirements for the pattern approval and verification of instruments used for the measurement of protein content of grain.

## 2 TERMINOLOGY

The terminology includes terms applicable to protein measuring instruments and some general terms included in the *International Vocabulary of Basic and General Terms in Metrology* (VIM, 1993).

## 2.1 Adjustment

Operation of bringing a measuring instrument into a state of performance suitable for its use (VIM, 4.30).

## 2.2 Certified Measuring Instrument

A measuring instrument certified under the National Measurement Regulations.

## 2.3 Certified Reference Material

A sample of grain whose protein content has been certified under the National Measurement Regulations.

## 2.4 Error of Indication

The indication of a measuring instrument minus the (conventional) true value of the measurand (VIM, 5.20).

## 2.5 Fault

The difference between the error of measurement and the intrinsic error of the measuring instrument.

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

## 2.6 Grain

For the purpose of this document, the term grain is taken to mean wheat or barley.

## 2.7 Indicator

A device that displays the measured protein concentration.

## 2.8 Influence Quantity

A quantity that is not the measurand but that affects the result of the measurement (VIM, 2.7).

## 2.8.1 Influence Factor

An influence quantity having a value within the rated operating conditions of the measuring instrument, specified in this document.

## 2.8.2 Disturbance

An influence quantity having a value within the limits specified in this document, but outside the specified rated operating conditions of the measuring instrument.

Note: An influence quantity is a disturbance if for that influence quantity the rated operating conditions are not specified.

## 2.9 Initial Intrinsic Error

The intrinsic error of a measuring instrument as determined prior to performance tests.

#### 2.10 Intrinsic Error

The error of a measuring instrument determined under reference conditions (VIM, 5.24).

## 2.11 Maximum Permissible Error

The extreme values (positive and negative) of the error of measurement permitted by regulation. The absolute value of the maximum permissible error is the same value without sign (VIM, 5.21).

## 2.12 Measuring Device

A device that makes each individual measurement of protein concentration.

## 2.13 Networked Instrument

An instrument that is linked, either electronically or manually under a quality system, to a certified measuring instrument and/or a certified reference material and/or the reference method of Annex A so that its performance may be monitored on a daily basis.

## 2.14 Protein Measuring Instrument

An instrument that determines the concentration of protein in a sample of grain.

## 2.15 Rated Operating Conditions

Conditions of use (giving the range of values of influence quantities) for which the metrological characteristics of a measuring instrument are intended to lie within the specified maximum permissible errors (VIM, 5.5).

## 2.16 Reference Conditions

Conditions of use prescribed for testing the performance of a measuring instrument or for comparison of results of measurements (VIM, 5.7).

## 2.17 Significant Fault

A fault greater than the absolute value of the maximum permissible error for the quantity delivered.

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

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

## 2.18 Test

A series of operations intended to verify the compliance of the equipment under test with certain requirements.

## 2.18.1 Test Procedure

A detailed description of the tests.

## 2.18.2 Test Program

A description of a series of tests for certain type of equipment.

## 2.18.3 Performance Test

A test intended to verify whether the equipment under test is able to accomplish its intended functions.

## SECTION II — METROLOGICAL REQUIREMENTS

## 3 UNITS OF MEASUREMENT

Australian legal units of measurement have not been prescribed generally for the physical quantity concentration. However, units of measurement, including percentage (by weight), expressed as %, will be prescribed for the concentration of protein in grain.

## 4 MAXIMUM PERMISSIBLE ERROR

For all grains, the maximum permissible error at verification, certification and reverification is with reference to the Dumas method (see Annex A).

The maximum permissible error for wheat is 0.4%, and for barley is 0.5%. These maximum permissible errors apply to all instruments irrespective of their principles of operation.

For the determination of the maximum permissible error:

- (a) Where a standard or reference material is used, the value of such standard or reference material should ideally be known to an expanded uncertainty (coverage factor two) of not greater than one-third of the maximum permissible error specified for that value.
- (b) Where the Dumas reference method is used, the number of repetitions of the complete measurement cycle should be such that the expanded uncertainty conforms with (a).

## 5 PERFORMANCE WITH VARIOUS GRAINS

## 5.1 Moisture Content

The protein content shall be converted to the value at the following moisture content:

- wheat 11%; and
- barley 0%.

The reference method for moisture content determination is Method 44-15A Moisture —

Air Oven Methods, from *AACC Approved Methods*, published by the American Association of Cereal Chemists.

## 6 INFLUENCE QUANTITIES AND DISTURBANCES

## 6.1 Reference Conditions

For reference conditions see clause B.2.

#### 6.2 Rated Operating Conditions for Influence Factors

Instruments shall be designed and manufactured so that all functions continue to operate as designed and that they do not exceed the maximum permissible errors when tested over the following ranges of influence factors:

- (a) mains power voltage variations: -15% to +10% of nominal voltage; and
- (b) air temperature variations:  $5^{\circ}$ C to  $40^{\circ}$ C.

A battery-operated instrument shall either continue to function correctly, or not provide a measurement, when the voltage is below the battery manufacturer's specified nominal voltage.

## 6.3 Humidity

Instruments shall be designed and manufactured so that all functions continue to operate as designed and that they do not exceed the maximum permissible errors when subjected to a damp heat, steady state test as described in clause B.3.3.

## 6.4 Disturbances

Instruments shall be designed and manufactured such that when exposed to the disturbances listed in Annex B, the following apply:

 (a) all functions continue to operate as designed and significant faults do not occur, that is the difference in the measured quantity without the disturbance applied and with the disturbance applied shall not exceed the absolute value of the maximum permissible error; or (b) significant faults are detected and the instrument made inoperative automatically or a visual or audible indication is provided automatically and shall continue until such time as the user takes action or the fault disappears.

The choice whether (a) or (b) is applied is left to the manufacturer.

## 6.5 Tests

A pattern of an instrument is presumed to comply with the requirements of clause 6 if it has passed the examination and tests specified in Annex B.

# SECTION III — TECHNICAL REQUIREMENTS

## 7 OPERATIONAL REQUIREMENTS

## 7.1 Suitability

7.1.1 Suitability for Purposes

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.

## 7.1.2 Suitability for Verification

An instrument shall be designed to enable the performance requirements of these rules to be applied.

## 7.2 Operational Safeguards

## 7.2.1 Fraudulent Use

Instruments shall not facilitate fraudulent use by either accidental means or by deliberate means when using the instrument in the normal manner. Sealing shall be applied as described in clause 10.

## 7.2.2 Operational Controls

Stand alone and networked instruments shall be subject to verification and reverification as described in clause 15.

## 7.2.3 Operational Adjustment

Networked instruments that are subject to a quality control system may be adjusted within the range of the maximum permissible error to improve the accuracy of the instrument in accordance with the procedures of the document referenced in clause 15.1.

An audit trail of such adjustments must be available for inspection by the relevant trade measurement authorities.

## 7.3 Sampling

Sampling shall be as described in Annex C.

## 7.4 Instrument Monitoring

When receiving grain using networked instruments, the following number of samples per day shall be retained and sealed for subsequent analysis by a reference instrument or by the Dumas method:

- up to 5 000 t/day one
- 5 000 t/day to 10 000 t/day two
- 10 000 t/day and above three

Information obtained in this way may be used to justify operational adjustments under clause 7.2.3.

## 8 INDICATIONS

## 8.1 Form of Indication

The figures forming the display shall be of a size, shape and clarity for easy reading under normal conditions of use.

## 8.2 Visibility

The display, if not permanent, shall remain visible until the next measurement is made or until the sample is removed from the instrument.

## 8.3 Style

Numbers and symbols of units shall be presented in accordance with AS ISO 1000– 1998. The International System of Units (SI) and its Application.

## 9 MARKING

## 9.1 Information to be Marked

Instruments shall be clearly and permanently marked with the following:

- (a) manufacturer's name or mark;
- (b) model designation;
- (c) serial number; and
- (d) NMI approval number.

## 9.2 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.

## 9.3 Style of Markings

The letters and numbers shall be marked in a font whose capitals are not less than 2 mm high.

Numbers and symbols of units shall be presented in accordance with AS ISO 1000-1998. The International System of Units (SI) and its Application.

## 10 VERIFICATION/CERTIFICATION MARKS AND SEALING

## 10.1 General

Provision shall be made for the application of a verification/certification mark and for sealing any adjustment device or control device which could affect the measurement.

Sealing shall be by password or any other acceptable means.

## 10.2 Position

A verification/certification mark shall be easily accessible and situated such that:

- (a) the part on which it is located cannot be removed from the instrument without damaging the mark;
- (b) it can be easily affixed without affecting the metrological properties of the instrument; and
- (c) it is visible without moving the instrument when it is in use.

## 10.3 Mounting

The area provided for the verification/ certification mark shall be at least 200  $\text{mm}^2$ , with an aspect ratio of approximately 2:1.

## 10.4 Electronic Sealing

If the adjustment or control of any function which affects the measurement is provided by electronic means (e.g. computer software) the following shall apply:

- (a) access by authorised persons shall be protected by some form of physical key, a password or access code (e.g. a four digit code);
- (b) access to alter protected parameters shall be automatically recorded (e.g. by means of an audit trail);
- (c) the audit record shall be readily accessible;
- (d) the audit record shall be readily identifiable as such and shall not be easily confused with other indications of the instrument; and
- (e) the audit record shall also be stored in a separate location.

## SECTION IV — METROLOGICAL CONTROL

## 11 LIABILITY

All protein measuring instruments to be used for trade shall be marked and verified under the uniform trade measurement legislation of the States and Territories. One of the requirements for verification is that the instrument is of a pattern approved by the Chief Metrologist.

## 12 APPLICATION FOR PATTERN APPROVAL

General application requirements are given in *NMI P 106. Approval and Certification Procedures for Measuring Instruments for Trade Use.* The application for pattern approval shall be accompanied by:

- (a) at least one sample instrument representative of the submitted pattern; and
- (b) descriptive documents and drawings.

## 12.1 Sample Instrument

The sample instrument shall be in full working order and shall include all functions to be examined for pattern approval.

Note: If the measuring instrument is part of a system which includes other than metrological functions, only that part which controls the metrological functions may be submitted for evaluation. A consultation service is provided prior to submission to determine those parts required for evaluation.

## 12.2 Descriptive Documents

Descriptive documents shall include:

- (a) drawings of the general arrangement and details of metrological interest including details of any interlocks, safeguards, auxiliary devices etc;
- (b) a short functional description of the instrument;
- (c) a short technical description including, if necessary, schematic diagrams of the method of operation; and
- (d) list of grain types for which approval is required.

## 13 PATTERN EVALUATION

The submitted documents shall be examined to verify compliance with the requirements of this document.

The instrument shall be tested in accordance with these procedures and Annex B. If testing of a complete instrument is not possible, tests may, as agreed by NMI and the applicant, be performed on a simulated set-up or on modules or main devices separately.

NMI may, in special cases, require the applicant to supply test equipment to perform the tests.

## 14 LABORATORY TESTS

## 14.1 Instrument Tests

All instruments shall be tested for maximum permissible errors (see clause 4) and the effect of influence factors. Annex B specifies the performance tests. Samples of grain having at least three protein concentrations (high, medium and low) shall be used for approval testing.

## 14.2 Repeatability

Measurements of the above samples of grain shall be repeated five times under reference conditions. For each sample the difference between the highest and lowest determination of protein concentration shall not exceed half of the maximum permissible error.

## 14.3 Test Standards

Standards with the required uncertainty (see clause 4.1) for the measurement shall be used. They shall be graduated so that the maximum permissible errors can be determined.

## 15 VERIFICATION

## 15.1 Verification Tests

#### 15.1.1 Networked Instruments

Networked instruments will be the subject of verification procedures specified in a separate document endorsed by State and Territory trade measurement authorities.

#### 15.1.2 Stand Alone Instruments

Where a stand alone instrument is used for trade it shall be verified individually using grain samples approved by the relevant trade measurement authority for that purpose.

Instruments shall be adjusted to within the verification maximum permissible error and as close to zero error as practicable across the protein concentration range.

#### 15.2 Verification Maximum Permissible Errors

The verification maximum permissible errors are given in clause 4.

# ANNEX A. DUMAS COMBUSTION — TOTAL NITROGEN DETERMINATION (REFERENCE METHOD)

This method is the approved Royal Australian Chemical Institute – Cereal Chemistry Division method.

## A.1 Field of Application

Principally to determine the protein content of grain and grain products.

## A.2 Principle

The sample is incinerated in an oxygen-rich environment, at approximately 1 000°C, to produce oxides of nitrogen which are catalytically reduced to molecular nitrogen. Interfering combustion products are removed by selective absorption. The concentration of nitrogen in the carrier gas mixture is measured by a thermal conductivity detector which is calibrated against a standard of known nitrogen content. Crude protein is calculated from nitrogen content using a known factor for each particular product.

## A.3 Reagents

## A.3.1 Gases

In various combustion nitrogen analysers, different gases are required to drive parts within the instrument or as carrier (such as helium or carbon dioxide). All gases used in the instrument must be of suitable purity stipulated by the instrument manufacturer. Storage and handling procedures must follow respective safety regulations and guidelines.

## A.3.2 Calibration Standard

It is recommended to use high purity ethylenediaminetetracetic acid. The standard should be dried before use in a vacuum desiccator over phosphorus pentoxide or oven dried at 130°C for 3 h.

## A.4 Apparatus

Grinder or mill that produces ground material with particle size  $\leq 0.8$  mm and

with minimal heat generation. To test the effectiveness of grinding for particular samples see clause A.9.1

Combustion nitrogen analyser that employs the Dumas combustion principle with a thermal conductivity detector to measure nitrogen. To test the performance accuracy see clause A.9.2

Analytical balance accurate to at least 0.000 5 g.

## A.5 Sample Preparation

Grind grain samples to pass a  $\leq 0.8$  mm sieve.

## A.6 Procedure

- A.6.1 Follow the manufacturer's guidelines to set up the combustion nitrogen analyser and the operating gas systems. Perform the necessary adjustments for gas flows and pressures, combustion temperatures and times, and start-up equilibrium times to ensure optimal analysis conditions for the type of sample to be analysed.
- A.6.2 Calibration of the instrument should follow manufacturer's guidelines using an appropriate nitrogen standard. It is recommended that the calibration is cross-checked against a second high purity standard (e.g. TRIS). Blanks, as stipulated by the manufacturer, should be run prior to analysis to establish the baseline. These should include consideration of an atmospheric blank factor or a sample blank, similar to samples under test.
- A.6.3 Grind an amount of sample sufficient to represent the original material and to perform a number of nitrogen determinations as required.

- A.6.4 Weigh accurately to 0.001 g an amount of ground sample, as recommended by the manufacturer, into the appropriate sample capsule and place the sample into the instrument for analysis.
- A.6.5 If presenting the sample to the instrument in a pellet form, adjustments may be required to burn temperatures, times and blanks to compensate for the absence of a sample capsule.
- A.6.6 Blank and standard control/check samples should be repeated periodically (e.g. every 10 samples) during each analytical run to monitor any drift. Standard drift corrections and recalculation of samples should be made after analysis, if the drift exceeds specification.

## A.7 Calculations

A.7.1 Calculation of nitrogen content is usually performed automatically by the instrument data processing system or associated software.

- A.7.2 Results should be expressed as percent nitrogen to two decimal places. For conversion to protein content 'as is' multiply wheat nitrogen by 5.70 and all other cereals by 6.25, unless otherwise stated. Convert protein content to a respective moisture basis for the nitrogen/protein values, where necessary.
- A.7.3 Analysis should be repeated if the differences between duplicate results exceed the respective repeatability values (r) shown in Table 1 (95% confidence level).
- A.7.4 The difference between single test results from two laboratories should not exceed the reproducibility values (R) shown in Table 1 (95% confidence level).

## A.8 Precision

Table 1 shows the mean nitrogen contents (as is) and precision statistics from analysis by the Dumas method of a range of products in a collaborative study.

Grain	Mean % N	Repeatability		Reproducibility	
Grain		r	RSD (%)	R	$RSD_{R}(\%)$
Barley	1.85	0.06	1.22	0.11	2.09
Barley malt	1.49	0.04	0.99	0.08	1.97
Lupin	5.10	0.08	0.57	0.18	1.29
Oats	1.04	0.05	1.59	0.07	2.35
Sorghum	1.47	0.05	1.15	0.07	1.69
Soymeal	7.85	0.10	0.47	0.22	1.00
Wheat (Durum)	2.09	0.04	0.64	0.08	1.32
Wheat	1.97	0.03	0.61	0.09	1.69
Wheat (American prime hard)	2.54	0.03	0.46	0.08	1.15
Wheat flour	2.03	0.03	0.46	0.09	1.56

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Table 1. Mean nitrogen	contents in a range	of grains (from	Willgiord and Fox. 19	991
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## A.9 Quality Assurance

- A.9.1 Suitable fineness of grind gives a relative standard deviation of ≤2.0% for 10 successive determinations of nitrogen in ground test material (Sweeney, 1989). A larger relative standard deviation indicates the need for a finer grind or a larger analytical test weight assuming that the instrument has been properly set up.
- A.9.2 For each analytical batch the accuracy of the system is demonstrated by making 10 successive determinations of nitrogen in nicotinic acid or tryptophan (different materials from calibration standard). Means of determinations must be  $\leq \pm 0.15$  of respective theoretical values with standard deviation  $\leq 0.15$ . Failure to achieve these values indicates the need for recalibration or optimisation of instrumental settings (see clauses A.6.1 and A.6.2).

- A.9.3 Accuracy checks should be carried out:
  - on instrument installation and reinstallation following repairs and service;
  - when a new batch of working reference material is used;
  - after experiencing problems in instrumental setup.
- A.9.4 Dumas (combustion) nitrogen values may be greater than corresponding Kjeldahl values, particularly at higher nitrogen levels.

## ANNEX B. PERFORMANCE TESTS FOR ELECTRONIC MEASURING SYSTEMS

## B.1 General

This mandatory annex is taken from OIML D 11. It defines the program of performance tests intended to ensure that electronic protein measuring instruments perform and function as intended in a specified environment and under specified conditions. Each test indicates, where appropriate, the reference conditions under which the intrinsic error is determined.

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.

## B.2 Reference Conditions

Ambient temperature:  $20^{\circ}C \pm 2^{\circ}C$ Relative humidity:  $60\% \pm 10\%$ Atmospheric pressure: 86 kPa to 106 kPaPower voltage: nominal voltage,  $U_{\text{nom}}$ Power frequency: nominal frequency,  $f_{\text{nom}}$ 

During each test the temperature and relative humidity shall not vary by more than 5°C and 10% respectively within the reference ranges.

## B.3 Performance Tests

The following performance tests can be carried out in any order:

- (a) dry heat (influence factor) see clause B.3.1;
- (b) cold (influence factor) see clause B.3.2;
- (c) damp heat, steady state (influence factor) see clause B.3.3;
- (d) power voltage variation (influence factor) see clause B.3.4;
- (e) short time power reductions (disturbance) — see clause B.3.5;
- (f) bursts (disturbance) see clause B.3.6;
- (g) electrostatic discharge (disturbance) see clause B.3.7;

- (h) electromagnetic susceptibility (disturbance) — see clause B.3.8; and
- (i) disturbances on d.c. voltage powered equipment see clause B.3.9.

## B.3.1 Dry Heat

## **Test Method**

Dry heat (non-condensing).

## **Object of the Test**

To verify compliance with the provisions in clause 6.2 under conditions of high temperature.

## References

IEC 60068-2-2 (1974). Background information concerning dry heat tests is given in IEC 60068-3-1 (1974) and first supplement IEC 60068-3-1A (1978). General background information on basic environmental testing procedures is given in IEC 60068-1 (1988), which is equivalent to AS 1099.1-1989.

## Test Procedure in Brief

The test consists of exposure of the equipment under test to a temperature of 40°C under free air conditions for a 2 h period after the equipment under test has reached temperature stability. The equipment under test shall be tested for at least 10 measurements:

- (a) at the reference temperature of 20°C following conditioning;
- (b) at the temperature of 40°C, 2 h after temperature stabilisation; and
- (c) after recovery of the equipment under test at the reference temperature of 20°C.

## **Test Severities**

Temperature:40°CDuration:2 h

## Number of Test Cycles

One cycle.

#### **Maximum Allowable Variations**

All operational functions shall operate as designed (e.g. indicators).

All errors shall be within the maximum permissible errors.

#### B.3.2 Cold

#### **Test Method**

Cold.

## **Object of the Test**

To verify compliance with the provisions in clause 6.2 under conditions of low temperature.

#### References

IEC 60068-2-1 (1990). Background information concerning cold tests is given in IEC 60068-3-1 (1974) and first supplement IEC 60068-3-1A (1978). General background information on basic environmental testing procedures is given in IEC 60068-1 (1988) which is equivalent to AS 1099.1–1989.

#### **Test Procedure in Brief**

The test consists of exposure of the equipment under test to a temperature of 5°C under free air conditions for a 2 h period after the equipment under test has reached temperature stability. The equipment under test shall be tested for at least 10 measurements:

- (a) at the reference temperature of 20°C following conditioning;
- (b) at the temperature of 5°C, 2 h after temperature stabilisation; and
- (c) after recovery of the equipment under test at the reference temperature of  $20^{\circ}$ C.

#### **Test Severities**

Temperature: 5°C Duration: 2 h

#### Number of Test Cycles

One cycle.

#### **Maximum Allowable Variations**

All operational functions shall operate as

designed (e.g. indicators).

All errors shall be within the maximum permissible errors.

#### B.3.3 Damp Heat, Steady State

## Test Method

Damp heat, steady state (non-condensing).

#### Object of the Test

To verify compliance of the electronic measuring instrument with the provisions in clause 6.3 under conditions of high humidity and high temperature.

#### References

IEC 60068-2-3 (1969) which is equivalent to AS 1099.2.3-1990. Background information is given in IEC 60068-3-4 (2001).

#### **Test Procedure in Brief**

The test consists of exposure to the specified high temperature and relative humidity for a period of two days. The handling of the equipment under test shall be so that no condensation of water occurs on the equipment.

The power supply is on when the influence factor is applied. The equipment under test shall be tested for at least 10 measurements before and after the application of the damp heat and at the specified damp heat after two days at these conditions.

#### **Test Severities**

Upper temperature:40°CHumidity:85%Duration:two days

#### **Number of Test Cycles**

One cycle.

#### **Maximum Allowable Variations**

All operational functions shall operate as designed (e.g. indicators).

All errors shall be within the maximum permissible errors.

## B.3.4 Power Voltage Variation

#### **Test Method**

Variation in a.c. mains power supply (single phase).

## **Object of the Test**

To verify compliance with the provisions in clause 6.2 under conditions of varying a.c. mains power supply.

## References

No reference to an international standard can be given at the present time.

#### **Test Procedure in Brief**

The test consists of exposure of the equipment under test to power voltage variations, while the equipment under test is operating under normal atmospheric conditions.

## **Test Severities**

Mains voltage:

- upper limit:  $U_{\text{nom}} + 10\%$
- lower limit:  $U_{\rm nom} 15\%$

## Number of Test Cycles

One cycle.

## **Maximum Allowable Variations**

All operational functions shall operate as designed (e.g. indicators).

All errors shall be within the maximum permissible errors.

#### B.3.5 Short Time Power Reduction

#### **Test Method**

Short time interruptions and reductions in mains voltage.

## **Object of the Test**

To verify compliance with the provisions in clause 6.4 under conditions of short time mains voltage interruptions and reductions.

#### References

No reference to an international standard can be given at the present time.

#### **Test Procedure in Brief**

The test consists of subjecting the equipment under test to voltage interruptions from nominal voltage to zero voltage for a duration equal to half a cycle of line frequency, and from nominal voltage to 50% of nominal for a duration equal to one cycle of line frequency. The mains voltage interruptions and reductions shall be repeated with a time interval less than the time required for a single measurement so that at least one voltage interruption occurs per delivery.

## **Test Severities**

100% voltage interruption for a period equal to half a cycle.

50% voltage reduction for a period equal to one cycle.

## Number of Tests

At least 10 deliveries shall be made with the voltage interruptions applied.

#### **Maximum Allowable Variations**

All operational functions shall operate as designed (e.g. indicators).

The effect of the disturbance shall not exceed the significant fault or the instrument shall detect and react to the fault (see clause 6.4).

## B.3.6 Bursts

#### **Test Method**

Electrical bursts.

## **Object of the Test**

To verify compliance with the provisions in clause 6.4 under conditions where electrical bursts are superimposed on the mains voltage.

#### Reference

#### IEC 61000-4-4 (1995)

## **Test Procedure in Brief**

The test consists of subjecting the equipment under test to bursts of double exponential waveform transient voltages. Each spike shall have a rise time of 5 ns and a half amplitude duration of 50 ns. The burst length shall be 15 ms, the burst period (repetition time interval) shall be 300 ms. All these bursts shall be applied during the same measurement in symmetrical mode and asymmetrical mode.

## **Test Severities**

Amplitude (peak value): 1 000 V

## **Number of Test Cycles**

At least 10 positive and 10 negative randomly phased bursts shall be applied at 1 000 V.

The burst are applied during all the time necessary to perform a measurement. At least 10 measurements shall be made with the bursts applied.

#### **Maximum Allowable Variations**

All operational functions shall operate as designed (e.g. indicators).

The effect of the disturbance shall not exceed the significant fault or the instrument shall detect and react to the fault (see clause 7.3).

## B.3.7 Electrostatic Discharge

#### **Test Method**

Electrostatic discharge.

#### **Object of the Test**

To verify compliance with the provisions in clause 6.4 under conditions of direct and indirect electrostatic discharges.

#### Reference

IEC 61000-4-2 (1995)

#### **Test Procedure in Brief**

A capacitor of 150 pF is charged by a suitable d.c. voltage source. The capacitor is then discharged through the equipment under test 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.

## **Test Severities**

8 kV for air discharges and 6 kV for contact discharges.

## Number of Test Cycles

At least one direct discharge or one indirect discharge shall be applied during the one measurement. At least 10 deliveries shall be made with the discharges applied.

#### **Maximum Allowable Variations**

All operational functions shall operate as designed (e.g. indicators).

The effect of the disturbance shall not exceed the significant fault or the instrument shall detect and react to the fault (see clause 6.4).

#### B.3.8 Electromagnetic Susceptibility

#### **Test Method**

Electromagnetic fields (radiated).

#### **Object of the Test**

To verify compliance with the provisions in clause 6.4 under conditions of electromagnetic fields.

#### Reference

IEC 61000-4-3 (2002)

## Test Procedure in Brief

The equipment under test shall be exposed to electromagnetic field strength as specified by the severity level.

The field strength can be generated in various ways:

- (a) the strip line is used at low frequencies
   (below 30 MHz or in some cases
   150 MHz) for small equipment under test;
- (b) the long wire is used at low

frequencies (below 30 MHz) for larger equipment under test;

(c) dipole antennas or antennas with circular polarisation placed at least 1 m from the equipment under test are used at high frequencies.

The specified field strength shall be established prior to the actual testing (without equipment under test in the field).

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.

When the test is carried out in a shielded enclosure to comply with international laws prohibiting interference to radio communications, care needs to be taken to handle reflections from the walls. Anechoic shielding might be necessary.

#### **Test Severities**

Frequency range:	26 to 1 000 MHz, field strength 3 V/m
Modulation:	80% AM, 1 kHz sine wave

## Number of Tests

Carry out deliveries throughout the application of the electromagnetic field. The tests will have to be initiated by remote control.

#### **Maximum Allowable Variations**

All operational functions shall operate as designed (e.g. indicators).

The effect of the disturbance shall not exceed the significant fault or the instrument shall detect and react to the fault (see clause 7.3).

#### B.3.9 Disturbances on d.c. Voltage Powered Instruments

Electronic measuring systems supplied with d.c. voltage shall fulfil the tests B.3.1 to B.3.8 with the exception of B.3.4 to B.3.6 which are replaced by the following provisions:

- (a) for under-voltages or over-voltages all errors shall be within maximum permissible errors when the instrument is still working; and
- (b) the under-voltage or over-voltage is applied for a complete measurement or part of a measurement.

## C.1. Scope

This annex applies to all types of sampling. The procedures contained here are based on ISO 13690:1999.

## C.2 Sampling System

## C.2.1 Manual Sampling

Research carried out in South Australia indicates that vacuum sampling has better repeatability than other manual sampling methods. Accordingly, of the manual sampling systems, vacuum systems are preferred. However, where vacuum systems are unavailable conventional manual spears may be used.

## C.2.1.1 Design of Vacuum Sampling Spear

The sampling spear comprises a 2 m long, 32 mm diameter stainless steel tube connected by a flexible hose to a sample container with a 0.5 mm mesh to ensure that fine material is retained in the sample container. The spear has a vacuum bypass that allows the operator to control the sampling.

## C.2.1.2 Quantity of Grain Sampled

Size of rail or road wagon	Minimum composite sample quantity
10 t or less	3 L
Over 10 t to 20 t	4 L
Over 20 t to 30 t	5 L
Over 30 t to 40 t	6 L
Over 40 t to 50 t	7 L
Over 50 t to 60 t	8 L
Over 60 t to 70 t	9 L
Over 70 t to 80 t	10 L

## C.2.1.3 Sampling Plans

For each bulk unit tendered for delivery, the following number of sampling points are to be used, evenly spaced throughout the load. Approximately equal sub-samples are to be taken from each sampling point in the road or rail vehicle.

Size of rail or road wagon	Number of sampling points
10 t or less	3
Over 10 t to 20 t	4
Over 20 t to 30 t	5
Over 30 t	7

## C.2.2 Mechanical Sampling

Mechanical sampling devices shall use the same sampling plans and produce the same sampled quantities of grain but the method of sampling is not manual.

## C.3 Collection and Packaging of Samples for Daily Monitoring

Each composite sample shall be thoroughly mixed prior to analysis. Samples to be returned to the laboratory for analysis shall be sealed in moisture-tight containers, labelled and stored appropriately.

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