



Specific Accreditation Criteria

ISO/IEC 17025 Application Document Calibration - Annex

Mass and related quantities

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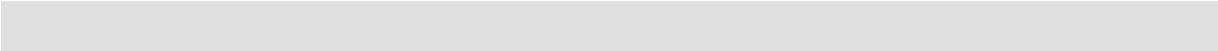


Table of Contents

Purpose.....	4
Criteria and recommendations for all types of mass and related measurements.....	4
Acceleration due to gravity.....	4
Criteria and recommendations for specific types of measurement equipment	4
Weighing Device calibration.....	4
Pressure measuring devices.....	7
Flow meters	8
Force Measuring Systems	10
Piston operated volumetric apparatus (POVA) calibration	11
References.....	12
Amendment Table	12

Mass and related quantities

Purpose

In addition to the *ISO/IEC 17025 Standard Application Document (SAD)* and the accompanying Calibration - Appendix, this document provides interpretative criteria and recommendations for facilities performing measurements of mass and related quantities for both applicant and accredited facilities.

Facilities must comply with all relevant documents in the NATA Accreditation Criteria (NAC) package for Calibration (refer to *NATA Procedures for Accreditation*).

For ease of use and to avoid fragmentation of the information, the relevant clause numbers of ISO/IEC 17025 have not been included.

Criteria and recommendations for all types of mass and related measurements

Acceleration due to gravity

Where the value of acceleration of gravity is required for force, mass, pressure or other calibration purposes, this value must be determined with an uncertainty appropriate for application at the working location. Geosciences Australia and State based geological or mineral organisations can provide a value of acceleration of gravity estimated at a nominated location or can provide a more accurate measured value when needed. It is the responsibility of the facility using this value to estimate an uncertainty for the value used. Acceleration of gravity values obtained from AS 1349 *Bourdon tube pressure and vacuum gauges*, internet or web based searches are approximate values and can only be considered suitable for calibration traceability where the final reported uncertainty of the measurand is not less than 0.05%.

Records on how the acceleration of gravity has been determined and how the associated measurement uncertainty has been estimated must be maintained.

Criteria and recommendations for specific types of measurement equipment

Weighing Device calibration

Calibration location

Weighing devices or balances are sensitive to transportation, their environment and changes in gravity. Consequently, balances should be calibrated at the location at which they are to be used. When moved, a weighing device is to be recalibrated or have the calibration and performance verified. This is important for high resolution (10 mg or better) weighing machines. The NATA publication, *General Accreditation Guidance: User checks and maintenance of laboratory balances* contains recommendations for the relocation of weighing devices.

Calibration method

Chapters 6 and 7 of NMI Monograph 4, *The Calibration of Weights and Balances*, provide an accepted method for the calibration of high resolution (10mg or better) weighing devices. For devices with a lesser required accuracy, such as industrial weighing appliances and hopper weighing systems, the shorter test methods as found in the NMI *National Instrument Test Procedure* (NITP) 6.1 to 6.4, have traditionally been accepted as a basis of a calibration method. It is acknowledged however that the NITP document is intended for the verification of equipment used in trade and thus does not make up a complete calibration method.

Whichever test method is used, the facility must be able to demonstrate the method is valid.

Preliminary

Reference masses/weights must have reached thermal equilibrium. The weighing device should have been turned on for the time specified by the manufacturer or at least 30 minutes if this period is not known. Prior to calibration, the auto-calibration or other adjustment feature used by the end-user must be run. Where weights are used for adjusting the device and the calibrated value cannot be entered, then these weights shall have a departure from nominal value that is appropriate to the accuracy required and/or specification. After exercising the device, the error close to full capacity must be recorded. If the device appears to require physical adjustment or repair, the user must be consulted to determine if a full set of before adjustment readings is required.

It should be ensured that the device is correctly levelled and any zero-tracking feature is temporarily disabled.

Handling of masses

Weights used for the calibration of balances should never be touched with bare hands. Small weights should be handled with plastic tipped tweezers and large weights with clean gloves (chamois, cotton or plastic) or with a lifting tool. For devices which include a chamber, the calibrator's hands should not enter the chamber during the loading and unloading of the weights as the resulting air currents and temperature effects can affect the measurements.

Corrections to reading

For higher resolution devices a minimum of 10 approximately evenly spaced calibration points must be taken over the range with no less than 5 points taken for devices with a lesser required accuracy. For balances with more than one range an equivalent number of calibration points must be taken in each range. If an instrument has more than 2 ranges and no independent adjustment for each range, a minimum of 5 points per range and one repeatability test in each range would be suitable.

For higher resolution devices, the reading sequence for each calibration point must be carried out twice and consist of zero/mass/mass/zero readings.

Note: The mass is lifted off the balance between the two mass readings.

In some cases the user may request a limited calibration range. This is permitted, provided it is stated on the report and on any calibration label attached to the balance. Alternatively where the user requires a higher precision within a limited section of the range (e.g. at the lower end of range), more points would be accordingly taken in this section.

Effect of off-centre loading / eccentricity

The effect of off-centre loading must be determined. This may be achieved by placing a weight on the centre of the pan and then lifting and placing it successively to the front, rear, left and right positions on the pan. For those balances with a higher resolution range this should be activated (e.g. by taring).

Hysteresis

Hysteresis must be carried out on the first calibration of a new device or after a device has undergone a repair to its weighing mechanism.

Repeatability of measurement

The repeatability of measurement must be determined usually at close to both half load and full load. As repeatability usually increases with larger loads, the full-load repeatability test would be carried out as close as practical (usually within 20%) to the full capacity of the instrument and using the minimum number of masses/weights. For example, it would not be appropriate to use a 2 kg mass to determine the repeatability of a 3.2 kg balance. In that case a 2 kg and 1 kg mass would be used together and care taken in placing them in the same spot each time.

For balances with more than one range, the repeatability must be carried out close to full capacity of the balance and also close to the maximum capacity of each range. The half load repeatability tests are not required unless the actual measuring system is different for each range.

For high resolution devices or where the customer requires a higher level of confidence, a minimum of 10 measurements must be taken for determining repeatability. For devices with a lesser required accuracy, such as industrial weighing appliances and hopper weighing systems, fewer measurements may be taken with the facility required to demonstrate the value calculated for repeatability is statistically valid and supports the required measurement uncertainty.

Limit of performance

Where the NATA *General Accreditation Guidance: User checks and maintenance of laboratory balances* is used, the Limit of Performance must be calculated and reported for each range using the formula:

$$F = 2.26 \times S_r(max) + C_{max} + U(C_{max})$$

Where:

$S_r(max)$ is the maximum value of the repeatability of measurement of the device for that range (taken from a minimum of 10 measurements) or 0.41 of the resolution in that range, whichever is greater.

C_{max} is the magnitude of the maximum correction to the reading for any of the calibration points measured in the range under consideration.

$U(C_{\max})$ is the expanded uncertainty associated with the maximum correction in the range under consideration.

Calibration report

Reports shall include:

- resolution of the device;
- description of the device;
- the precise location of the weighing device;
- pre-adjustment readings. As a minimum, the correction or error close to full load must be reported;
- repeatability of measurement;
- corrections to readings at each calibration point and the associated uncertainty of measurement;
- off-centre loading (description of test and results);
- hysteresis (where verified);
- uncertainty of weighing at each calibration point;
- Limit of Performance (when required by the customer);
- for balances with more than one range the repeatability, corrections and Limit of Performance for each range must be reported.

Pressure measuring devices

The facility must unambiguously specify the range of capability over which calibration can be conducted as a gauge pressure range and/or an absolute pressure range. This capability will be included in the facility's their Scope of Accreditation. Where not otherwise specified, the range will be taken as a gauge pressure only. Reporting measurements in absolute pressure can only be done if a Calibration and Measurement Capability (CMC) for absolute pressure is stated in the Scope of Accreditation.

The measurement uncertainty (as stated in the CMC), if reported as a percentage, shall be a percentage of reading and not full scale. For gauge pressure, ranges that cross zero must include a minimum value (in SI units) in conjunction with a percentage of reading.

For negative gauge pressure measurements, the maximum negative range must take into account at least the vacuum generating equipment and the reference calibrator range and operation. As the achievable negative gauge pressure will depend on the atmospheric pressure at any one point in time, a maximum negative pressure of -101 kPa should be considered the best case achievable.

Flow meters

A flow meter may be regarded as one of two distinct types of device.

- 1) An instrument that indicates or generates a signal, representing a volume or mass of a fluid passed through it (for example, the volume in L or m³ or mass in kg or tonnes) when operating at a flow condition (for example, 15 L/s or 15 kg/s).
- 2) An instrument that indicates or generates a signal, representing a flow rate or velocity.

In either case the instrument may include a flow computer which provides flow outputs.

Calibration report

To allow unambiguous declaration of the calibration of a flow meter, the report must include:

- the fluid used during calibration describing relevant physical properties which are not otherwise defined and can impact the calibration. including but not limited to: pressure, temperature, relative humidity, electrical conductivity;
- the calibrated volume or mass and the nominal rate of delivery for a flow meter described as type 1 above;
- the calibrated flow rate and the nominal measured volume or test time for a flow meter described as type 2 above;
- environmental conditions;
- a description of the instrument under test (IUT), including the size of the meter and any upstream pipework such as flow straighteners, filters, gas eliminators or pulsation dampeners.

NATA scope of accreditation

The SoA will specify:

- type of fluid. Facilities may also state the relevant delivery condition to inform potential customers (for example CNG at 10 bar to 300 bar);
- either a test volume range or flow rate range which can be delivered from the reference system;
- an applicable uncertainty of measurement for a best typical instrument under test following the guidance for key uncertainty terms below must be declared either in volume or flow units or where uncertainty is declared in % units, a minimum delivery must be specified;
- the facility's measurement capability as either a volume or a flow rate capability or both when relevant;
- when uncertainty is specified in % units, the SoA will indicate if the uncertainty relates to volume or flow rate.

For example, if a facility provides calibration of the volume attribute of fuel oil meters, the specification of capability will include the facility's nominal flow rate range and the minimum test volume which can be accurately delivered.

*Fuel oil in the flow rate range 1 L/s to 50 L/s with a least uncertainty of measurement of:
0.1% of volume with a minimum 100 L test volume.*

It is the facility's responsibility to determine its measurement capability and to request of NATA what the SoA is to define. NATA can only confirm during the assessment the facility's capability and adjust the SoA as necessary.

Key uncertainty terms for flow meters

Uncertainty analysis should include certain key terms such as:

- calibration of the reference meter, collection volume or gravimetric apparatus;
- difference in calibration of the reference meter arising from its use with different fluids or at different pressures than those with which it was calibrated;

Note: This type-B uncertainty could, for example, be estimated using manufacturer's information or published literature but preferably from direct measurements.

- meter-under-test repeatability;

Note: This can be assessed by either repeat testing of one or more calibration points or by a type B analysis for that meter class estimated from prior testing, manufacturer's data, accepted industry knowledge or other demonstrated means.

- uncertainty of relevant physical properties which are not otherwise defined and can impact the calibration including but not limited to: pressure; temperature; relative humidity; electrical conductivity associated with the flow difference between the reference equipment and the Item Under Test (IUT);
- terms specified in industry specific standards.

Key operational characteristics of flow meters

Due to IUT design and test method employed, a minimum test volume may be required for accurate calibration.

It is essential to consider the change in volume of the test fluid between the reference equipment and the IUT due to relevant physical properties. For highest accuracy work, these properties must be measured, both at the reference and at the IUT and appropriate corrections applied.

Any working fluid loss between the reference equipment and the IUT will result in measurement errors and the facility must have processes to manage this. For highest accuracy work, appropriate leak testing must be considered as part of each test setup. Valid statistical processes may be acceptable for a type B estimate of this error to provide an uncertainty contribution. Acceptable processes would include:

- perform and record standard leak testing at regular intervals (for example, start and end of a shift) using the same type of meter and mounting system as is being routinely tested;
- visually examine the IUT/test rig seals;
- a system of testing during the shift with an interval that maintains confidence in the system.

Force Measuring Systems

(for testing including force testing machines)

In addition to the uncertainty contribution associated with the reference standard, the uncertainty calculation must also include contributions from the drift of the reference load cell (during its calibration interval or from manufacturer's specifications), the resolution and the repeatability of the force measuring system under test.

Where a force measuring system cannot provide stable force application, factors such as operator reaction bias and response time of the working force standard and/or the force measuring system must also be included in the uncertainty calculation.

Contributions from temperature effect, creep, linearity deviation, misalignment of forces, adaptors and fittings, and the effect of worn or unparallelled platens must also be considered when these contributions apply.

Refer to Appendix E, AS 2193 *Calibration and classification of force-measuring systems* as a guide.

Working force standards

(for the calibration of force measuring systems)

The calibration of working force standards must also consider contributions from deflections and the zero deviation as well as a full determination of the repeatability and hysteresis. Refer to Section 4 and Appendix F of AS 2193 as a guide.

A separate CMC to differentiate force measuring systems (to be used for testing) and working force standards (to be used for calibration) will be listed in the scope of accreditation of facilities that calibrate working force standards.

The classification of working standards to AS 2193 must follow the criteria in Section 4 of that standard. Both continuous class and point class method may be used on a single load cell, however the classification reported must only apply to the section of the range application to the classification method used. For example, when using the point class method, the classification can only be reported for the forces used during calibration. When reporting a range of classification the continuous class method must be used.

Piston operated volumetric apparatus (POVA) calibration

Currently there are two recognised standard methods in use for the calibration of POVAs by gravimetric method, ISO 8655-6:2002 and AS 2162.2-1998, with noted technical differences between the two publications.

When reporting compliance to the Maximum Permissible Errors (MPEs) published in the normative references to ISO 8655-6, the higher criteria contained in ISO 8655 Parts 2 and 6 shall be applied. For example, in the case of piston pipettes, Tables 1 and 2 of ISO 8655-2 apply.

Examples of noted differences between the ISO and AS standards include:

- for volumes at 10 μl , a balance with minimum resolution of 0.001 mg (ISO 8655-6) is required instead of minimum resolution 0.01 mg (AS 2162-2);
- the MPEs stated in ISO 8655-2 are based on 10 aliquot repeats and not the option of fewer repeats contained in AS 2162.2;
- for variable volume apparatus, clause 7.1.1 of ISO 8655-6 requires at least three volumes to be tested, unless a reduced user range has been agreed to with the end user and subsequently reported;
- for multichannel apparatus, clause 7.5 of ISO 8655 requires each channel to be considered independently against the specifications.

When stating conformance to the systematic error and random error MPEs contained in Parts 2 to 5 of ISO 8655, the procedure as stated in clause 8.4.2 and 8.5.2 of ISO 8655-6 shall be applied, that being the measurement uncertainty is not added or subtracted from the result. This follows the Accuracy Method of ISO GUM 98-4, clause 8.2.4, where the well characterised test method minimises sources of uncertainty by use of instruments with maximum permissible errors within specified limits (clause 4 of ISO 8655-6).

As with all calibrations, the measurement uncertainty for the volume dispensed is to be reported. Additional information on the calibration of measurement uncertainty may be found in the EURAMET Guideline No. 19 *Guidelines on the Determination of Uncertainty in Gravimetric Volume Calibration*.

References

This section lists publications referenced in this document. The year of publication is not included as it is expected that only current versions of the references shall be used.

Standards

AS 1349	Bourdon tube pressure and vacuum gauges
AS 2162.2	Verification and use of volumetric apparatus - Guide to the use of piston-operated volumetric apparatus (POVA)
AS 2193	Calibration and classification of force-measuring systems
ISO 8655-6	Piston-operated volumetric apparatus
ISO/IEC 98-4 (GUM 98-4)	Uncertainty of measurement - Part 4: Role of measurement uncertainty in conformity assessment
ISO/IEC 17025	General requirements for the competence of testing and calibration laboratories

NATA Publications

NATA Accreditation Criteria (NAC) package for Calibration

General Accreditation Criteria	ISO/IEC 17025 Standard Application Document
Specific Accreditation Criteria	ISO/IEC 17025 Standard Application Document, Calibration - Appendix
General Accreditation Guidance	User checks and maintenance of laboratory balances

Other Publications

EURAMET cg-19	Guidelines on the Determination of Uncertainty in Gravimetric Volume Calibration
NMI Monograph 4	The Calibration of Weights and Balances, E C Morris and K M K Fen
NMI, National Instrument Test Procedure (NITP) 6.1 to 6.4	

Amendment Table

The table below provides a summary of changes made to the document with this issue.

Section or Clause	Amendment
Whole document	General editorial revision.
Piston operated volumetric apparatus (POVA) calibration	New section.

