INTERNATIONAL STANDARD

ISO 8655-6

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Piston-operated volumetric apparatus —

Part 6: Gravimetric methods for the determination of measurement error

Appareils volumétriques à piston —

Partie 6: Méthodes gravimétriques pour la détermination de l'erreur de mesure

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 8655 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 8655-6 was prepared by Technical Committee ISO/TC 48, *Laboratory glassware and related apparatus*, Subcommittee SC 1, *Volumetric instruments*.

ISO 8655 consists of the following parts, under the general title *Piston-operated volumetric apparatus*:

- *Part 1: Terminology, general requirements and user recommendations*
- *Part 2: Piston pipettes*
- *Part 3: Piston burettes*
- *Part 4: Dilutors*
- *Part 5: Dispensers*
- *Part 6: Gravimetric methods for the determination of measurement error*

The following part is under preparation:

- *Part 7: Non-gravimetric methods for the determination of measurement error*

Annex A forms a normative part of this part of ISO 8655. Annex B is for information only.

Introduction

ISO 8655 addresses the needs of:

— suppliers, as a basis for quality control including, where appropriate, the issuance of supplier's declarations;

— test houses and other bodies, as a basis for independent certification;

— users of the equipment, to enable routine checking of accuracy.

The tests specified should be carried out by trained personnel.

Piston-operated volumetric apparatus —

Part 6:
Gravimetric methods for the determination of measurement error

1 Scope

This part of ISO 8655 specifies the reference method for conformity testing of piston-operated volumetric apparatus, whereby errors of measurement are determined gravimetrically. The tests are applicable to complete systems comprising the basic apparatus and all parts selected for use with the apparatus, disposable or reusable, involved in the measurement by uptake (In) or delivery (Ex) process.

NOTE General requirements and definitions of terms of piston-operated volumetric apparatus are given in ISO 8655-1. For the metrological requirements, maximum permissible errors, requirements for marking and information to be provided for users for piston-operated volumetric apparatus, see ISO 8655-2 for piston pipettes, see ISO 8655-3 for piston burettes, see ISO 8655-4 for dilutors and see ISO 8655-5 for dispensers. Alternative test methods such as photometric and titrimetric methods will be the subject of a future Part 7 to ISO 8655.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 8655. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 8655 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 8655-1:2002, *Piston-operated volumetric apparatus — Part 1: Terminology, general requirements and user recommendations*

ISO 8655-2:2002, *Piston-operated volumetric apparatus — Part 2: Piston pipettes*

ISO 8655-3:2002, *Piston-operated volumetric apparatus — Part 3: Piston burettes*

ISO 8655-4:2002, *Piston-operated volumetric apparatus — Part 4: Dilutors*

ISO 8655-5:2002, *Piston-operated volumetric apparatus — Part 5: Dispensers*

ISO/TR 20461:2000, *Determination of uncertainty for volume measurements made using the gravimetric method*

ISO/IEC Guide 2, *Standardization and related activities — General vocabulary*

OIML R 76-1:1992, *Non-automatic weighing instruments — Part 1: Metrological and technical requirements — Tests*

3 Terms and definitions

For the purposes of this part of ISO 8655, the terms and definitions given in ISO 8655-1, ISO/IEC Guide 2 and OIML R 76-1 apply.

4 Apparatus

4.1 Analytical balance or equivalent weighing device, with a resolution appropriate to the selected volume of the apparatus under test (see Table 1).

Table 1 — Minimum requirements for balances

Selected volume[a] of apparatus under test	Resolution	Repeatability and linearity	Standard uncertainty of measurement
V	mg	mg	mg
1 µl $\leqslant V \leqslant$ 10 µl	0,001	0,002	0,002
10 µl $< V \leqslant$ 100 µl	0,01	0,02	0,02
100 µl $< V \leqslant$ 1 000 µl	0,1	0,2	0,2
1 ml $< V \leqslant$ 10 ml	0,1	0,2	0,2
10 ml $< V \leqslant$ 200 ml	1	2	2

[a] For practical purposes, the nominal volume may be used to choose the balance.

If the standard uncertainty of measurement of the balance is known (e.g. from the balance calibration certificate), this may be used instead of the repeatability and linearity. The standard uncertainty of measurement shall not be more than two to three times the resolution.

4.2 Liquid reservoir, with sufficient capacity for all the test liquid likely to be required for the complete series of tests.

4.3 Weighing vessel, suitable for the test procedure selected from clause 7. Care shall be taken regarding the loss of water by evaporation during the dispensing and weighing procedure.

It is recommended that, especially for testing apparatus of the lowest volume, the height-to-diameter ratio of the weighing vessel be at least 3:1 or that a weighing vessel with a lid be used.

4.4 Timing device, with a standard uncertainty of $\leqslant$ 1 s (see note to 4.7).

4.5 Thermometer, with a standard uncertainty of $\leqslant$ 0,2 °C (see note to 4.7).

4.6 Hygrometer, with a standard uncertainty of $\leqslant$ 10 % (see note to 4.7).

4.7 Barometer, with a standard uncertainty of $\leqslant$ 0,5 kPa (see note to 4.7).

NOTE All uncertainties are specified using a coverage factor k of 1.

5 Test liquid

Use distilled or deionized water conforming grade 3 as specified in ISO 3696, degassed or air-equilibrated. The water shall be at room temperature (see 6.2).

6 Test conditions

6.1 General

Apparatus that is routinely dismantled and reassembled within the scope of its intended use (e.g. for cleaning purposes) shall be dismantled and reassembled at least once prior to the test in accordance with the supplier's operation manual.

Apparatus shall be operated as specified in the supplier's operation manual.

6.2 Test room

The test shall be carried out in a draught-free room with a stable environment. The test room shall have a relative humidity above 50 % and a constant (± 0,5 °C) temperature between 15 °C and 30 °C. Prior to the test, the apparatus to be tested and the test water shall have stood in the room for a sufficient time, at least 2 h, to reach equilibrium with the room conditions.

NOTE See 8.3 for corrections to be made when the balance readings are converted to volumes.

6.3 Evaporation

Especially for small volumes below 50 µl, errors due to evaporation of the test liquid during weighing shall be taken into consideration. Apart from the design of the weighing vessel (4.3), the test cycle time is important.

In order to keep the error due to evaporation as small as possible, the following additional items can be considered, if volumes below 50 µl are tested:

— a balance with appropriate accessories such as an evaporation trap could be used; or

— the test liquid to be weighed could be delivered into a capillary tube, although this method does not replicate the normal method of use and the user should verify for himself that correlation exists.

Regardless of these items, the error due to evaporation during the measuring series can be determined experimentally (see 7.2.8) and compensated mathematically (see 8.1). The uncertainty of this compensation should be added to the uncertainty of measurement.

6.4 Test cycle time

The test cycle time (time required to complete the weighing of one dispensed volume) shall be kept to a minimum. It should ideally not exceed 60 s. It is important that it is regular, both within each cycle and as far as possible from cycle to cycle, so that a reliable mathematical compensation of the error due to evaporation during the measuring series can be applied.

7 Procedure

7.1 General

7.1.1 Test volume

In the case of a fixed-volume apparatus, the test volume is the nominal volume. In the case of a variable-volume (user-selectable volume) apparatus, at least three volumes shall be tested:

— the nominal volume,

— approximately 50 % of the nominal volume,

— the lower limit of the useful volume range or 10 % of the nominal volume (whichever is the greater).

Measurement of further volumes is optional. The setting devices of the apparatus (e.g. dials, scales) shall be sufficient for the selection of the test volume.

7.1.2 Number of measurements per test volume

If the gravimetric methods of this part of ISO 8655 are used as conformity tests or type tests, e.g. prior to a declaration or certification of conformity, or if the gravimetric method is used as a reference method, 10 measurements for each test volume shall be carried out. These measurements are used to calculate the systematic and the random error of measurement in accordance with clause 8.

For re-establishing conformity, e.g. after repairs not performed by the supplier, 10 measurements at each volume shall also be performed.

If the gravimetric method is used for other purposes, such as supplier's quality control or supplier's after-sales service,

— the number of test volumes (see 7.1.1),

— the number of measurements per volume and,

— where applicable, the number of channels tested

may be changed to an appropriate number. Alternative test methods may also be used for this purpose, provided that they can be proven to correlate with the reference method specified in this part of ISO 8655, in which case the user should choose a number of measurements for his metrological confirmation based on his accuracy requirements.

7.1.3 Weighing procedure

Weighing for apparatus designed to deliver (Ex) shall always involve dispensing of the test liquid into the weighing vessel. Weighing for apparatus designed to contain (In) shall always involve the removal of test liquid from the weighing vessel. An example of the latter is the sample uptake step in the use of a dilutor.

7.1.4 Test conditions during the weighing procedure

At the start and at the end of the weighing procedure, the temperature of the test liquid in its container shall be recorded to the nearest 0,2 °C. The barometric pressure in the test room shall be recorded to the nearest 1 kPa and the relative humidity to the nearest 10 %.

7.2 Single-channel piston pipettes with air interface (in accordance with ISO 8655-2)

7.2.1 In the case of power-driven piston pipettes, aspiration and delivery of test liquid are automatic. The remainder of the procedure is carried out following the procedure described below.

7.2.2 Place test liquid from the water container in the weighing vessel to a depth of at least 3 mm. Record the temperature of the test liquid and the barometric pressure and relative humidity in the test room (see 7.1.4). If the weighing vessel has a lid, fit it.

NOTE Temperature and barometric pressure are necessary for the choice of the correction factor Z (see 8.3 and annex A); the relative humidity is not necessary for the evaluation as the correction factors Z in annex A apply to relative humidities ranging from 20 % to 90 % but are necessary for documentation in the test report [see clause 9, item d)].

7.2.3 If using a variable-volume piston pipette, select the test volume; this setting shall not be altered during the test cycle of 10 measurements.

7.2.4 Prepare the piston pipette and the test cycle as follows:

a) Fit the selected tip to the piston pipette.

b) Fill the tip with test liquid and expel to waste five times to reach a humidity equilibrium in the dead air volume (see ISO 8655-1:2002, 3.1.8) of the air-displacement piston pipette.

c) Place the weighing vessel with its added water on the balance pan.

7.2.5 Perform the following test cycle (see Figure 1 and Figure 2):

a) Replace the disposable tip of the piston pipette.

b) Fill the piston pipette with test liquid, immersing its delivery orifice 2 mm to 3 mm below the surface of the water. Release the operating button slowly, if hand operated, and withdraw the pipette vertically and carefully from the surface of the water. Touch the delivery orifice against the side wall of the container with the test liquid.

c) Expel the water to waste in order to pre-wet the tip and refill the piston pipette as described in b).

d) Record the mass m_0 of the weighing vessel to the nearest readable graduation as in Table 1, or tare the balance to zero ($m_0 = 0$). Start the timing device (this may be omitted if using a weighing vessel with lid).

e) If the weighing vessel has a lid, remove it. Deliver the contents of the pipette into the weighing vessel, touching the delivery end of the pipette tip against the inside wall of the vessel just above the liquid surface at an angle of approximately 30° to 45° and draw it approximately 8 mm to 10 mm along the inner wall of the weighing vessel to remove any droplets at or around the tip orifice. Replace the lid if applicable.

Where applicable, use the blow-out feature of the piston pipette to expel the last drop of liquid before drawing the delivery end of the tip along the inner wall of the weighing vessel.

If it is necessary to remove the weighing vessel from the balance pan to permit delivery of the dispensed volume, avoid excessive handling and possible contamination by the use of lint-free gloves. Return the weighing vessel to the balance pan after delivery.

f) Record the mass m_1 of the weighing vessel, or if tared in step 7.2.4 c) the mass m_i of the quantity delivered.

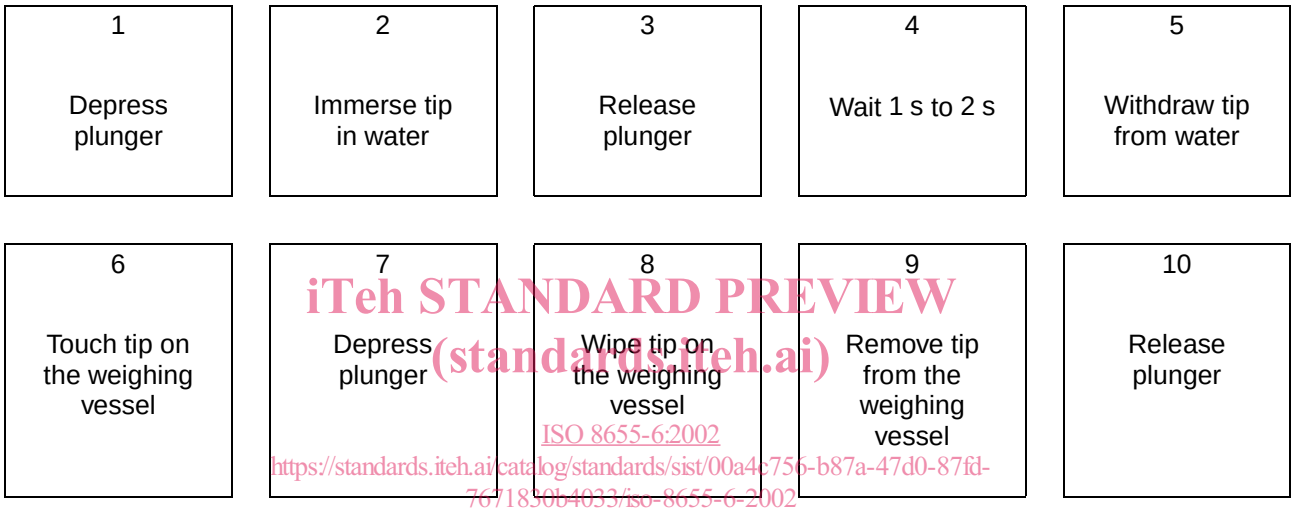

Figure 1 — Pipetting of testing volume into the weighing vessel

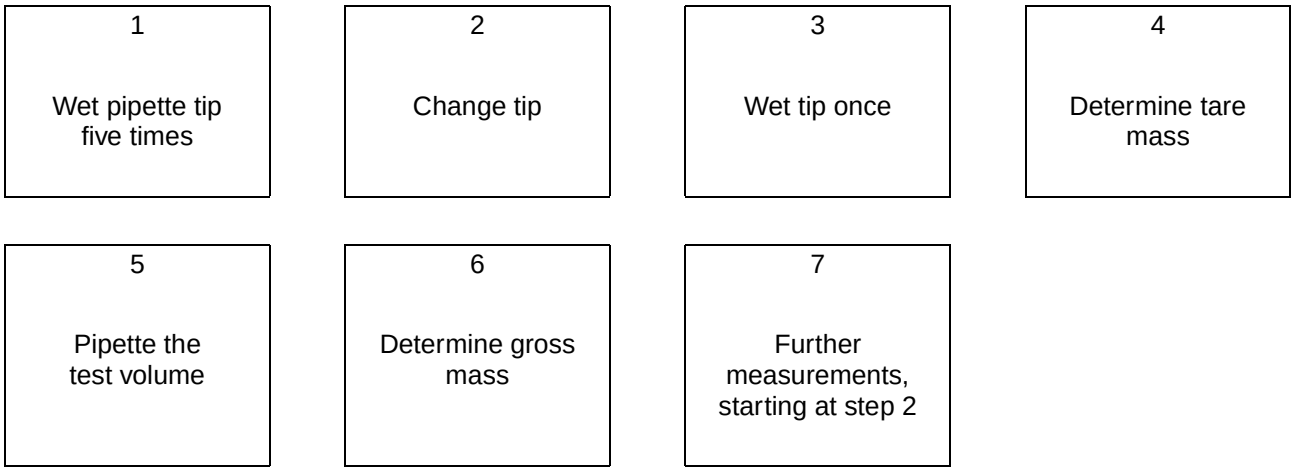

Figure 2 — Scheme of test procedure for piston pipettes with air interface

7.2.6 Repeat the test cycle described in 7.2.5 until 10 measurements have been recorded as a series of masses m_1 to m_{10}.

7.2.7 Note the time to the nearest second taken to complete the 10 test cycles.