Project Report 1588
Explaining the process of revision of volume standards ISO 8655 and ISO 4787

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Content

1 INTRODUCTION

This report was developed by EURAMET's TC Flow Subcommittee Volume with the cooperation of several members of ISO TC 48/WG4 - Liquid Handling Devices – Manual and Semi-Automatic.

The objective of this report is to provide information on the changes performed in two of the most important volume standards that were revised in 2021 and 2022, respectively ISO 4787 and ISO 8655. These standards are used by both National Metrology Institutes, Accredited laboratories and end users, all over the world. Also, information is given regarding the changes on the ISO/TR 20641 and ISO/TR 16153 performed in 2023.

2 ISO 8655:2022 series, ISO/TR 20641:2023 and ISO/TR 16153:2023

2.1 Objective of the ISO 8655:2022 revision and the correspondent Technical Reports ISO/TR 20641:2023 and ISO/TR 16153:2023

This revision aimed to improve the metrological content of the documents reflecting the improvements and development of technology and to develop two new parts: one for the photometric reference method (part 8) and another for syringes (part 9). Another part is still under development (part 10). The concurrent revision and development of new standards allowed the harmonisation of the content of all parts within the ISO 8655 series.

The work was accomplished in ISO TC48/WG4 – Laboratory equipment - Liquid Handling Devices – Manual and Semi-Automatic, WG Convenor Valentin Lütke-Börding.

- ***Part 1:*** *Terminology, general requirements and user recommendations* [1], revised.
- ***Part 2:*** *Pipettes* [2], revised.
- ***Part 3:*** *Burettes* [3], revised.
- ***Part 4:*** *Dilutors* [4], revised.
- ***Part 5:*** *Dispensers* [5], revised.
- ***Part 6:*** *Gravimetric reference measurement procedure for the determination of volume* [6], revised.
- ***Part 7:*** *Alternative measurement procedures test methods for the for the determination of volume* [7], revised.
- ***Part 8:*** *Photometric reference measurement procedure for the determination of volume* [8], new.
- ***Part 9:*** *Manually operated precision laboratory syringes* [9], new.
- ***Part 10:*** *User guidance, and requirements for competence, training, and POVA suitability*, under development.

- **ISO/TR 20461** – Determination of uncertainty for volume measurements of a piston-operated volumetric apparatus using a gravimetric method [10], revised.
- **ISO/TR 16153** - Determination of uncertainty for volume measurements of a piston-operated volumetric apparatus using the photometric method [11], revised.

2.2 ISO 8655 Revision steps

The ISO 8655 series was originally published in 2002. The review work began in 2014, with more than 30 experts involved from various institutions, namely National Metrology Laboratories, Accredited Laboratories and manufacturers. Experts convened in 44 working group meetings in order to get to the final and published versions. Seven documents were revised, two new ones developed, and one new part of the standard is still under development. The focus of this new part 10 is handling by the user.

- EURAMET actively participated as a liaison organisation for every part of the standard.
- Each part of the standard had a project leader:
  - Elsa Batista from IPQ, Portugal, parts 1, 6 and 9.
  - A. Bjoern Carle from Artel, USA, parts 7, 8, and 10.
  - Valentin Lütke-Börding from Webers, Germany, part 2, 3, 4 and 5.

In the next subchapters are explained the changes to each part of ISO 8655 from 2022 and the content of the new parts also from 2022.

2.2.1 ISO 8655-1:2022 – terminology, general requirements and user recommendations

- The standards ISO 8655-7, ISO 8655-8 and ISO 8655-9 were added as normative references.
- The definitions and terms were arranged to be in alphabetical order.
- Abbreviated terms have been introduced in Clause 4.
- Metrological terms referring to the VIM [12] were included.
- 20 new terms were included, the most relevant were:
  - Correction
  - Measurement
  - Repeatability
  - Accuracy
  - Metrological confirmation
  - Test
  - Reference procedure
  - Nominal volume – maximum volume

- General requirements for measurement error reporting, calibration frequency, metrological confirmation, consumables, routine testing and maintenance and repair were added.
- Information on the determination of acceptance criteria and the adequacy of the performance statements were added in Clause 6 of this part 1.
- Information was included that error determination is performed differently than described in the VIM [12], resulting in a reversed sign of the error.

2.2.2 General changes to ISO 8655:2022 parts 2,3,4,5

- The standards ISO 8655-7, ISO 8655-8 were added as normative references.
- Requirements for the metrological performance of micropipette tips were developed.
- Tables 1 and 2 were changed, mainly:
    - The maximum permissible errors (systematic and random) are now only given in percentages (relative errors).
    - The tables are organized in nominal volume ranges.
    - For each nominal value, the maximum permissible error at 10 %, 50 % and 100 % is described.

2.2.2.1 ISO 8655-2:2022 – Piston pipettes

- New tables with random and systematic errors for multichannel micropipettes.
- New informational annex on motorised micropipettes.
- Former annex A with manufacturer information, is now included in clause 10 of part 2.
- Increase volume range up to 20 mL.

2.2.3 ISO 8655-6:2022 – Gravimetric reference measurement procedure for the determination of volume

- The expanded uncertainty of the test equipment, given in Tables 1 and 2, has been changed according to ISO/TR 20461:2023 requirements.
- New information added to Table 1 – minimum requirements for balances namely:
    - The choice of the balance is done based nominal volume instead of select volume.
    - Information on special requirements for multichannel pipettes.
    - Reference to EURAMET Calibration Guide 18 - Guidelines on the Calibration of Non-Automatic Weighing Instruments [13] in the notes.
- New Table 2 – minimum requirements for the measuring devices was added.
- Annex B has been deleted from this part 6.

- Clause 4 “General requirements” was added.
- The Z Tables of appendix A are now informative.
- More detailed standard dispense procedures for each POVA are given.
- Environmental/test conditions are now stricter and more controlled.
    - Humidity between 45% to 80% (in 2002 version >50%).
    - Air temperature between 20 °C ± 3 °C, not varying by more than 0.5 °C. per hour (in the 2002 version the air temperature was from 15 °C and 30 °C).
    - The water temperature must not vary by more than 0.5 °C during the tests and must be recorded at the beginning and at the end of the tests.
    - New Note: the stabilisation time should be approx. 2 h and could be much longer in dependency from the temperature gradient between POVA and air temperature in the laboratory.
    - Recording of ambient temperature, relative humidity and air pressure
- The reference temperature of 20 ºC or 27 ºC was added in clause 7.2.
- Test cycle concept that corresponds to a weighing – a measurement.
- New table 3 of immersion depth of the tips and aspiration waiting time.
- The procedure for calibrating piston pipettes was changed to include at least one tip change in 10 tests.
- All channels of a multichannel pipette must be tested individually, filled and emptied at the same time.
- Inclusion of the syringe calibration procedure.
- Focus on the manufacturer's instructions.
- An example of evaporation calculation was added has Formula (1).
- Formula (2) for calculating the delivered volume was added based on ISO 4787 [14].
- Formula (3) of the CIPM density [15] of air was added.
- Formula (4) of the Tanaka [16] density of water was added.
- Inclusion of information on the calculation of uncertainties in the determination of volume in a new subchapter 9.6
- Change of content of test reports/calibration certificates including measurement uncertainty.
- Include in the bibliography the EURAMET Guide 19 - Guidelines on the determination of uncertainty in gravimetric volume calibration.

2.2.4 ISO 8655-7:2022 – Alternative measurement procedures for the determination of volume

- Five normative procedures for the determination of volume are provided, as well as a batch testing method. Each procedure states clear requirements for the test equipment to be used and the preparation of the test liquids. The following procedures are described in detailed, normative annexes:

  - Gravimetric procedure.
  - Dual-dye ratiometric photometric procedure.
  - Single-dye photometric procedure.
  - Hybrid photometric/gravimetric procedure.
  - Potentiometric titration procedure.
- It is permitted to use test liquids other than water or water-like solutions, provided that the liquid properties are described in sufficient detail to allow for these tests to be reproducible.
- A clear distinction between calibrations and routine tests is drawn.
- Calibration of POVA according to one of the procedures in Part-7 requires validation of the measurement procedure against one of the reference measurement procedures described in ISO 8655-6 and ISO 8655-8.
- Calibrations require an estimation of the measurement uncertainty – either according to ISO/IEC Guide 98-3 (GUM) [18] or following ISO/TR 16153 (for the dual-dye ratiometric photometric procedure) or ISO/TR 20461 (for the gravimetric procedure). The estimation of measurement uncertainty for routine tests is optional.
- Routine tests allow testing at fewer than three volume settings and performing fewer than ten (but at least four) replicate measurements per volume setting.
- Particular consideration is given to multi-channel pipettes. Volumetric performance may be determined by using multi-channel balances, performing dual-dye ratiometric photometric measurements in micro plates, or performing the photometric/gravimetric hybrid procedure using micro plates.
- This standard encourages calibration and routine tests of POVA under the environmental conditions in which it is used, e.g., by performing the volume measurements in the laboratory in which the POVA is used.
- Standardised dispense procedures for the different POVA types are now included in this standard and harmonised with the standardised dispense procedures in ISO 8655-6 and ISO 8655-8.
- Requirements for the qualification of POVA users have been added.
- Annexes A, B and C of the 2005 edition have been replaced.

2.2.5 ISO 8655-8:2022 (NEW) - Photometric reference measurement procedure for the determination of volume

- This new standard adds a second reference measurement procedure to the ISO 8655 series of standards. Both reference measurement procedures provide the best possible volume measurements under tightly controlled conditions.
- The dual-dye ratiometric photometric procedure forms the basis of this reference measurement procedure, measuring the absorbance of Ponceau S and copper (II) chloride solutions.

- The test liquids are aqueous Ponceau S solutions, exhibiting water-like liquid properties.
- The standard provides details on minimum performance requirements for all test equipment and reagents to be used in this procedure.
- Environmental reference conditions are specified: temperature: 20 °C ± 3 °C, and maximum variation of 0.5 °C during the test; relative humidity: 45% to 80% (non-condensing).
- The reference temperature of 20 ºC or 27 ºC was added.
- Standardised dispense procedures for each POVA type are described and harmonised with ISO 8655-6 and -7.

2.2.6 ISO 8655-9:2022 (NEW) – Manually operated precision laboratory syringes

This new standard includes information of the principle of operation of the precision syringes, the construction requirements, and the metrological performance requirements.

2.3 ISO/TR 20461:2023 – Determination of uncertainty for volume measurements of a piston-operated volumetric apparatus using a gravimetric method

This document was published in February 2023, Elsa Batista from IPQ, Portugal was the project leader.

The main changes of the document when compared with the previous edition are:

- Structure according to GUM.
- Components similar to the ones described in EURAMET cg 19 and DKD-E 8-2.
- New uncertainty components were added e.g.:
    - Standard uncertainty related to air cushion effects.
    - Standard uncertainty of resolution.
    - Standard uncertainty of setting.
    - Uncertainty of reproducibility (0.1%).
- Include a new detailed calculation example for a piston pipette of 100 μL.
- Introduce information about the uncertainty in use of single delivered volume in a dedicated informative Annex. In this Annex A information for the end user and not to be used for traceability and further uncertainty calculations is described.
- Include calculation of volume variation with altitude, Annex B informative
- Reference is made to the EURAMET Calibration Guide 19 - Guidelines on the determination of uncertainty in gravimetric volume calibration.

2.4 ISO/TR 16153:2023 - Determination of uncertainty for volume measurements of a piston-operated volumetric apparatus using the photometric method

This document was published in February 2023, A. Bjoern Carle from Artel, USA was the project leader and the main changes are similar as for ISO/TR 20461:2023.

2.5 ISO 8655 and correspondent TRs revision conclusions

- More precise technical requirements for gravimetric calibration by the gravimetric reference procedure in part-6.
- A second reference procedure is specified in part-8: the photometric reference measurement procedure.
- Users have the option to use one of the alternative methods described in part 7 for calibrations, testing, and routine tests.
- Distinction between routine tests, testing and calibration: calibrations require an estimation of measurement uncertainty, while this is optional for routine tests and testing.
- New standard on precision syringes (part-9).
- TRs revised according to GUM and EURAMET cg 19 information.

3 ISO 4787:2021 - Laboratory glass and plastic ware - Volumetric instruments- Methods for testing of capacity and for use

3.1 Objective of the ISO 4787:2021 revision

This revision aimed to improve the metrological content of the document according to the development of technology and to include information about uncertainty and repeatability.

The work was done in ISO TC48/WG 7 – Laboratory equipment - Volumetric apparatus made of glass and plastic, WG Convenor Isabel Faria.

3.2 Revision steps

The 1st edition of ISO 4787 was in 1984 - Laboratory glassware - Volumetric glassware - Methods for testing of capacity (0,1 mL – 2000 mL).

The 2nd edition was in 2010 - Laboratory glassware - Volumetric instruments - Methods for testing of capacity and for use (0,1 mL – 10000 mL).

The 3rd edition was in November 2021 - Laboratory glass and plastic ware - Volumetric instruments- Methods for testing of capacity and for use.

The revision of the 3rd edition started in March in 2020, with about 10 experts involved from various institutions, namely National Metrology Laboratories, Accredited Laboratories and manufacturers. In less than 2 years it was possible to make the revision of the document and publish it.

EURAMET actively participated as a liaison organisation.

Elsa Batista from IPQ was the coordinator of this project.

3.2.1 Main revisions of ISO 4787:2021 - Laboratory glass and plastic ware - Volumetric instruments- Methods for testing of capacity and for use

- The volumetric plastic ware has been included in the scope of this document.

- The requirements of Table 1 have been improved and expanded uncertainty of the balances added including a reference to EURAMET Calibration Guide 18 - Guidelines on the Calibration of Non-Automatic Weighing Instruments [14].
- New Table 2 for minimum requirements for the measurement devices used in the calibration has been added, all the requirements are better than the previous version and the timing device was included.
- New information on meniscus adjustment of a convex meniscus has been added; namely, “Upper edge of the graduation line is horizontally tangential to the highest point of meniscus” was altered as compared to older procedure “Upper edge of the graduation line is horizontally tangential to the lowest point of the meniscus”.
- The figures for meniscus adjustment have been provided for different type of liquids and shapes.
- Liquid temperature with a maximum variation of ± 1 °C during the test.
- A variation between the liquid temperature and the air temperature of ±0.5 °C was included.
- The cleaning clause now also contemplates plastic instruments.
- A new clause for conditions of use was added, in the previous edition some of this information was in Annex A.
- The requirements for the test room ambient conditions changed significantly. The temperature range is now (20 ± 3) °C or (27 ± 3) °C with a maximum variation of ± 1 °C during the test. Also, the temperature variation of the room should not be more than 1 °C per hour.
- The humidity variation is also described and should be between 30 % and 80 % due to the influence in the balance’s performance.
- The time for equilibrium was removed and a new sentence added “Prior to the test, the apparatus to be tested, all test equipment, and water shall have stood in the test room for a sufficient time to reach equilibrium with the test room conditions”.
- Information on how to calibrate Pipettes adjusted to contain and Pycnometers was added.
- The weighing procedure was described in detail and included two options for subsequent tests in instruments used to contain, e.g. flasks.
- Formula (C.1) has been changed to Formula (1).
- A new clause on volume and uncertainty calculation was added. The calculation formula for volume is now in the main text. Not in an Annex.
- In clause 10 the pycnometers were included.
- A new annex for Cleaning volumetric plasticware was added, Annex B.
- Annex C has now all the support information, tables and formulas for volume calculation according to Formula (1).
- Tables for Z factor are now only for 20 °C and 27 °C.
- Annex D was created to include all the relevant coefficients of cubic thermal expansion. Before only 3 materials were included, now there are 10.

- New information regarding repeatability and uncertainty has been added in Annex E. Three types of operations are referred: batch, verification and calibration.
- A reference to EURAMET Calibration Guide 19 - Guidelines on the Determination of Uncertainty in Gravimetric Volume Calibration [17] was included.

3.3 ISO 4787 revision Conclusions

- Plastic ware was included.
- New and more precise technical requirements for volume determination. New information on meniscus adjustment of convex meniscus has been added.
- Ambient conditions of the test more restrictive.
- The use and calibration of pycnometers were included.
- Uncertainty information is now available in this document with references to EURAMET guides.
- This document is now harmonised with the ASTM E542:2022 [19] where some members of the volume subgroup were also invited to participate in the revision of this document.

4 - EURAMET contributions

Several members of the EURAMET TC Flow Volume subgroup actively participated in the revision of the described volume standards, and coordinate the revision of ISO 8355-2, ISO 8655-6, ISO 8655- 6, ISO 4787 and TR 20461. The main points addressed by the volume subgroup members were:

- Improved measurement procedures
- Use of more accurate instrumentation
- More control of the test conditions
- Adequate calculation formulas
- Improvement of uncertainty estimation information

Overall, the revised documents allow more accurate and reliable volume measurement results, which will benefit all members of laboratories and industry, end users and the global economy.

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