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Determination of uncertainty for volume measurements of a piston-operated volumetric apparatus using a gravimetric method

Détermination de l'incertitude de mesure pour les mesurages volumétriques des appareils volumétriques à piston au moyen de la méthode gravimétrique

Reference number
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Contents

Page

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 48, *Laboratory equipment*.

This second edition cancels and replaces the first edition (ISO/TR 20461:2000), which has been technically revised and cancels ISO/TR 20461:2000/Cor 1:2008.

The main changes are as follows:

— the term “standard deviation of the mean delivered volume” has been replaced in this document by “repeatability” according to ISO/IEC Guide 99 ;

— a new uncertainty calculation example has been supplied;

— new uncertainty components have been added, namely, reproducibility, air cushion and resolution;

— a new Annex A concerning approaches for the estimation of uncertainty in use of a single delivered volume has been added;

— a new Annex B concerning volume correction due to pressure changes has been added.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The example given in this document is informative and supports the requirements found in ISO 8655-6:2022, 9.6 and ISO 8655-7:2022, 4.2, to perform an estimation of measurement uncertainty when calibrating POVA according to the measurement procedures described in these documents and the principles of ISO/IEC Guide 98-3.

The revision of this document coincides with a major revision of the ISO 8655 series in 2022, reflecting the state-of-the-art measurement procedures and approaches for the estimation of measurement uncertainty.

Determination of uncertainty for volume measurements of a piston-operated volumetric apparatus using a gravimetric method

1 Scope

This document gives detailed information regarding the evaluation of uncertainty for the gravimetric reference measurement procedure specified in ISO 8655-6[1] and the gravimetric procedure specified in ISO 8655-7:2022[1], Annex A, according to the ISO/IEC Guide 98-3[16].

This document also includes the determination of other uncertainty components related to the liquid delivery process of a piston-operated volumetric apparatus (POVA), e.g. repeatability and handling. Furthermore, it provides examples for the calculation and application of the uncertainty of the mean delivered volume and the uncertainty in use of a single delivered volume.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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

ISO/IEC Guide 99, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8655-1, ISO/IEC Guide 2 and ISO/IEC Guide 99 apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Modelling the measurement

In the gravimetric reference measurement procedure, a quantity of water is delivered by the instrument under calibration (POVA) into a vessel that is weighed on a balance. Ambient conditions are recorded so that the liquid density and air density can be determined and, consequently, the delivered volume can be calculated from this data.

Furthermore, the influence of possible evaporation and possible temperature difference of the POVA from the reference calibration temperature are taken into consideration as corrections in the mathematical model of the calibration.

The general formula for calculation of the volume at the reference temperature of 20 °C, V_{20} (at a reference temperature of 27 °C, V_{27}), from the balance indication of the delivered water as described in

ISO 4787[2] and the ISO 8655 series[1], is given by Formula (1). When a liquid other than water is used, Formula (1) is modified accordingly.

$$V_{ref} = (m_L - m_E + m_{evap}) \times \frac{1}{\rho_W - \rho_A} \times \left(1 - \frac{\rho_A}{\rho_B}\right) \times [1 - \gamma(t_W - t_{ref})] \quad (1)$$

where

V_{ref} is the calculated volume at the reference temperature, in ml;

m_L is the balance indication of the weighing vessel after water delivery, in g;

m_E is the balance indication of the weighing vessel before water delivery, in g (m_E = 0 in case the balance was tared with the weighing vessel);

m_{evap} is the estimated evaporated mass within a test cycle, in g;

ρ_A is the density of air, in g/ml, at the temperature, humidity and atmospheric pressure of the test, see Formula (3);

ρ_B is the density of the reference weights (typically 8 g/ml);

ρ_W is the density of water at the test temperature (in units of °C), in g/ml, calculated with the "Tanaka" Formula (4);

γ is the combined cubic thermal expansion coefficient of the POVA under test, in °C^{-1};

t_W is the temperature of the POVA, assumed to be equal to the temperature of the water used in the test, in °C;

NOTE The temperature of the POVA can be different from the temperature of the water, however the POVA temperature cannot be measured directly. The effect of a potential temperature difference can be taken into account in the uncertainty model.

t_{ref} is the reference temperature of the POVA (20 °C or 27 °C).

This model shows that the measured volume V_{ref} is a function of $m, t_W, \rho_A, \rho_B, \rho_W, \gamma$.

Formula (1) can be simplified by using the Z correction factor according to the following:

$$V_{ref} = m_i \times Z \times [1 - \gamma(t_W - t_{ref})] \quad (2)$$

where

m_i (i=1 to n) is each balance indication;

Z is a correction factor as a function of pressure and temperature that is given in ISO 8655-6:2022, Table A.1.

The simplified formula for the air density ρ_A, Formula (3), can be used at temperatures between 15 °C and 27 °C, barometric pressure between 600 hPa and 1 100 hPa, and relative humidity between 20 % and 80 %:

$$\rho_A = \frac{1}{1\,000} \times \frac{0{,}348\,48 \times p - 0{,}009 \times h_r \times e^{(0{,}061 \times t_A)}}{t_A + 273{,}15} \quad (3)$$

where

ρ_A is the air density, in g/ml;

t_A is the ambient temperature, in °C;

p is the barometric pressure, in hPa;

h_r is the relative air humidity, in %.

At other environmental conditions, Formula (3) is replaced with the calculations described in CIPM-2007[3].

Another commonly used formula for air density is described in Spieweck's work[4].

The density of pure water ρ_W is normally provided from formulae given in the literature. Formula (4) given by Tanaka[5] can be used:

$$\rho_W = a_5 \left[1 - \frac{(t_W + a_1)^2 (t_W + a_2)}{a_3 (t_W + a_4)} \right] \tag{4}$$

where

ρ_W is the density of water, in g/ml;

t_W is the water temperature, in °C;

a_1 −3,983 035 °C;

a_2 301,797 °C;

a_3 522 528,9 (°C)^2;

a_4 69,348 81 °C;

a_5 0,999 974 950 g/ml.

Atmospheric pressure corrections can also be applied to the volume delivered according to Annex B and Formula (B.1).

5 General procedure for the uncertainty calculation

The evaluation of measurement uncertainty in this document follows the ISO/IEC Guide 98-3. The method described has the following steps:

a) Expressing, in mathematical terms, the relationship between the measurand and its input quantities.

b) Determining the expected value of each input quantity.

c) Determining the standard uncertainty of each input quantity.

d) Determining the degree of freedom for each input quantity.

e) Determining all covariance between the input quantities.

f) Calculating the expected value for the measurand.

g) Calculating the sensitivity coefficient of each input quantity.

h) Calculating the combined standard uncertainty of the measurand.

i) Calculating the effective degrees of freedom of the combined standard uncertainty.

j) Choosing an appropriate coverage factor, *k*, to achieve the required confidence level.

k) Calculating the expanded uncertainty.

In this document, the uncertainty of the measurement associated with the volume is separated in three different clauses: the uncertainty components associated with the gravimetric measuring system, the uncertainty components associated with the device under test (POVA) and the uncertainty components associated with the liquid delivery process.

6 Standard uncertainty components associated with the measuring system (gravimetric measurement procedure)

6.1 General information on standard uncertainty components estimation

It is possible to experimentally estimate the standard uncertainty of measurement, $u(x)$, for a quantity x, by performing repeated measurements of x under identical experimental conditions. This is called a type A evaluation according to reference ISO/IEC Guide 98-3. The standard deviation of the obtained values is a measure of the repeatability of the measurement. The standard uncertainty associated with x can be the standard deviation (in the case where a single measurement of x is made), or the standard deviation of the mean equal to stdev(x)/sqrt(n) (in the case where x is the average of n readings).

See ISO/IEC Guide 98-3:2008, 4.2 for more information on type A evaluation of standard uncertainty.

As an alternative to repeated measurements, the uncertainty of measurement, $u(x)$, for a quantity x, can be estimated by other means. This is called a type B evaluation according to ISO/IEC Guide 98-3. For example, information can be obtained for that estimation by considering the manufacturer's specifications of the POVA (e.g. resolution, linearity, drift, temperature dependence, etc.).

Often the manufacturer's specifications are given in the form of an interval covering the measurement value, with no additional information regarding distribution or coverage. In those cases, the measurement is assumed to follow a uniform or rectangular distribution. This distribution is characterized by a constant probability inside the interval while the probability outside the interval is zero.

The interval can be used to give the variance of x in the form (type B evaluation according to ISO/IEC Guide 98-3) of:

$$u^2(x_i) = \frac{\left[\frac{1}{2}(a_{i+} - a_{i-})\right]^2}{3} = \frac{a_i^2}{3} \tag{5}$$

where a_{i-} and a_{i+} give the lower and the upper limits of the interval of the variable i.

a_i is half of this interval, typically the interval is denoted as $\pm a_i$ in this case. The standard uncertainty is given as the square root of the variance.

In addition to uniform rectangular, other distributions are also possible when preforming type B evaluations. See ISO/IEC Guide 98-3:2008, 4.3 for more information on type B evaluations of standard uncertainty.

The different expressions for the standard uncertainty of each input quantity related to the gravimetric reference measurement procedure are presented in the following formulas.

6.2 Standard uncertainty of weighing (balance indication)

The standard uncertainty $u(m)$ related to the balance indication (m) is calculated as follows:

$$u(m)=\left[u^2(m_{\mathrm{L}})+u^2(m_{\mathrm{E}})+u^2(\delta m)+u^2\left(m_{\mathrm{evap}}\right)\right]^{\frac{1}{2}} \tag{6}$$

where

$u(m_{\mathrm{L}})$ is the standard uncertainty associated with the balance indication of the weighing vessel after water delivery, in g;

$u(m_{\mathrm{E}})$ is the standard uncertainty associated with the balance indication of the weighing vessel before water delivery, in g;

$u(\delta m)$ is the drift of the balance, in g;

$u(m_{\mathrm{evap}})$ is the standard uncertainty of the estimated mass of the evaporated quantity of water within a delivery cycle, in g. This is determined experimentally in each laboratory.

NOTE 1 The uncertainty of the balance indications can be estimated according to References [10] and [11] at the value corresponding to the selected volume.

The uncertainty of the balance indications can be taken from the balance calibration certificate if the expanded uncertainty in use is expressed. Otherwise, it can be calculated by using the uncertainty at calibration and including non-corrected errors, as well as possible drift and environmental effects to balance sensitivity.

The uncertainty calculation for the weighing is determined considering that the weighing vessel is not removed during the test. Additional uncertainties can arise if the vessel is removed from the balance.

NOTE 2 The correlations found in mass measurements are, in the case of this gravimetric measurement procedure, negligible.

6.3 Standard uncertainty of temperature

The standard uncertainty $u(t)$ related to the temperature (water and POVA), t, is calculated as follows:

$$u(t)=\left[u^2(t_{\mathrm{W}})+u^2(\delta t_{\mathrm{s}})\right]^{\frac{1}{2}} \tag{7}$$

where

$u(t_{\mathrm{W}})$ is the uncertainty of the temperature of the water;

$u(\delta t_{\mathrm{s}})$ is the estimation of the uncertainty caused by the variation between the water temperature and the temperature of the POVA;

and

$$u(t_{\mathrm{W}})=\left[\left(\frac{U_{\mathrm{ther}}}{k}\right)^2+u^2(\mathrm{res})+u^2(\delta t)\right]^{\frac{1}{2}} \tag{8}$$