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Guidelines on the Calibration of Standard Capacity Measures using the Volumetric Method

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Version 1.0 (04/2013)

Calibration Guide

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GUIDELINES ON THE CALIBRATION OF STANDARD CAPACITY MEASURES USING THE VOLUMETRIC METHOD

Purpose
This document provides guidance in the volumetric calibration procedure and in determination of the uncertainty in volumetric volume calibration of standard capacitiy measures and it has been produced to improve harmonization between the calibration laboratories.

Authorship
This document was developed by the EURAMET e.V., Technical Committee for Flow.

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Guidance Publications
This document gives guidance on measurement practices in the specified fields of measurements. By applying the recommendations presented in this document laboratories can produce calibration results that can be recognized and accepted throughout Europe. The approaches taken are not mandatory and are for the guidance of calibration laboratories. The document has been produced as a means of promoting a consistent approach to good measurement practice leading to and supporting laboratory accreditation.

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1. Introduction

The accurate measurement of domestic and industrial consumption of water, fuels and other fluids is essential to carry out business transactions in a clear and unequivocal way. Therefore it is necessary to use the correct volume standards, calibrated by competent entities that will ensure the traceability of the measurements. These volume standards are standard capacity measures (SCM) [1] and, depending on the nominal volume, can be divided in two types: standard test measures from 1 L to 20 L and proving tanks for more than 20 L.

The calibrated standard capacity measures are used as working standards for calibration and verification of the following measuring instruments:

- fuel dispensers and adblue dispensers,
- measuring systems (dynamic or static) on road tankers (delivery, or collected),
- measuring systems at truck loading facilities,
- measuring systems for the loading and unloading of tank containers,
- measuring systems for milk and beer,
- standard metering equipment for wet calibration of storage tanks,
- measuring systems for refueling aircrafts,
- measuring systems on ships,
- pipe provers,
- large proving tanks.

The standard capacity measures can be calibrated at a higher level by using the gravimetric method [2] or at a working level by using the volumetric method.
The volumetric method consists of delivering a quantity of liquid to or from a calibrated standard (reference standard), to or from a standard capacity measure. This method is normally used where the capacity of a standard capacity measure is so large that using weighing instruments is impracticable or if the necessary accuracy of the measurements is lower.
In this Guide the volumetric calibration procedure is presented in detail along with the evaluation of the measurement uncertainty.
The procedure and formulae suggested in this Guide are not intended to, nor can they replace the personal judgment and responsible evaluation individually made by the metrologists in any particular application and laboratory.

2. Definitions

2.1 Volumetric method

In the volumetric method a certain amount of liquid is delivered into a container. The volume is determined at a reference value denoted by a graduation mark. When the standard capacity measure is equipped with an adjustable device or scale the volume can be adjusted to the nominal volume. The calibration can be performed adjusting the level exactly to the reference value, by adding or removing water until the level corresponds to the graduation mark, or calculating the actual volume at the reference value on the basis of the scale reading.
In the majority of the cases the volumetric method is faster, easier and with lower cost than the gravimetric or geometric method. It is considered a method of direct comparison, requiring specific structures such as overflow pipettes or volumetric containers.
The volumetric method may be used in two different approaches: withdrawing or filling.

2.2 Filling method

The filling method consists of filling the standard capacity measure being calibrated with water, from a smaller or equally large, reference standard, which has been calibrated to an accuracy level significantly higher than the measure under calibration.

2.3 Withdrawing method or delivery method

The withdrawing method involves the determination of the volume of water drained by gravity or air pressure, from the standard capacity measure being calibrated, into one or several, smaller or equally large, reference standards, which have been calibrated to an accuracy level significantly higher than the measure under calibration.

2.4 Measures “to contain” or “In”

Standard capacity measure whose capacity is equal to the volume of water that it contains, at the reference temperature, when filled to its reference line.

2.5 Measures “to deliver” or “Ex”

Standard capacity measure whose capacity, with respect to it’s reference line, is equal to the volume of water that it delivers, during a predefined dripping time, at the reference temperature.
The volume delivered is always less than the volume contained, due to the film of liquid left on the internal walls of the vessel. The volume of this film depends on the time taken to deliver the liquid. The volume delivered decreases with decrease of delivery time.

2.6 Dripping time

It is the time necessary to wait after the main flow ceases and starts to drop. It is also called the drainage time. This value should always be stated in the calibration certificate of the standard capacity measure.

2.7 Delivery time

It is the time necessary to empty the standard capacity measure completely. Delivery time is time from the beginning to the end of main flow plus dripping time. This information should be described in the calibration certificate and depends on the size and shape of the vessel and the size of the bottom valve.

3. General techniques

3.1 Definition of type of calibration

Preferably the calibration laboratory, should use the same method as the customer (either as measure "to deliver" or "to contain" in a wet or dry mode) during its calibration, because this will determine the chosen calibration method, the filling method or the withdrawing method.

3.2 Standard capacity measure

The standard capacity measure (SCM) is the instrument to be calibrated.
There are two types of standard capacity measures; standard test measures and proving tanks. The standard test measures are emptied by pouring so that the liquid flows out from only one point of the rim. The capacity of these measures can vary from 1 up to 20 L. Proving tanks are provided with drain valves at the bottom, the capacity of these measures can go up to 5000 L.
An inspection and cleaning process must be performed to the SCM prior to calibration. The readability and security of the scale, the level determination system and any relevant seals should be noted. The condition and leak tightness of the discharge valve in the case of proving tanks, and the general condition of the measure e.g. existence of bumps, leaking tubes or damages should be noted.
It is recommended that a leak-check be performed before the start of measurement in case of measures with discharge valve and/or scales with glass displays. Volume standards must allow a precise and repeatable quantity of liquid (water). The shape of the measure must avoid problems regarding the trapping of liquid or vapor and should allow for easy cleaning. It shall be ensured, that liquids are easily delivered to and from the standard and no pockets, dents or crevices capable of trapping the liquid, air or vapor are present.
The standard capacity measure shall be leveled before calibration commences.

3.3 Reference standard

Volume standards, especially reference standards, must allow "to contain" or "to deliver" a precise and repeatable quantity of liquid. The shape of the measure must avoid problems with regard to the trapping of liquid or air or vapor or to the cleaning of the measure. It shall be ensured that the liquids are easily transferred to and from the standard and that pockets, dents or crevices capable of trapping the liquid, air or vapor are not present.
The reference standards (RS) must be calibrated with an uncertainty significantly smaller than that of the measure being calibrated.
There are two basic types of reference standards; reference standard with graduation line and overflow reference standard.

3.4 Reference temperature

The value of the reference temperature of the SCM depends on the purpose it is used for. These measures are usually used for a reference temperature of 20 ºC, but sometimes the customer needs 15 ºC (petrol industry) [3], 4 ºC (milk industry), or some other different temperature. This information should be specified, included in calculations, clearly visible in the calibration certificate and preferably marked on the identification plate on the measure.

3.5 Quality of water

The water used for volumetric calibrations shall be clean, without dirt, air, contaminants or corrosive chemicals. In general potable water can be used as long it is free of air and stored in a reservoir for temperature stabilization. When using potable water direct from pipeline (e. g. when calibrating on site), it is important to mechanically remove the air, by stirring the water.
If small SCM are used (100 liter and smaller) the delivered volume of liquids with high viscosities (higher than 5 mm^2/s) can be different from water. In this case the reference standard also needs to be calibrated with this liquid or a liquid with a similar viscosity range used for calibration.

Note: If other liquids are used care should be taken regarding evaporation and viscosity. These liquid proprieties can lead to an incorrect reference volume and large measurement uncertainties. Also, the corrections and calculations must be determined based on the properties of the alternative liquid.

3.6 Water temperature

The water temperature should be measured in both the reference standard and the standard capacity measure.
In large proving tanks (capacities greater than 500 L) the water temperature should be measured in more than one location due to possible temperature gradients [1].
When performing calibrations in the laboratory the water temperature shall be stable to within ± 1 °C and as close as possible to air temperature [1].
For standard capacity measures installed in fixed systems it is recommended that this method shall be carried out on site and within a period so that the water temperature in the standard capacity being calibrated will not vary by more than 2 ºC during the filling [1]. In this case a calibrated reference standard in delivery mode ("Ex") should be used.

3.7 Air temperature

During the calibration in the laboratory air temperature shall be stable to at least ± 3 °C and its value recorded.

3.8 Temperature of the standard capacity measure and reference standard

In order to avoid uncontrolled expansions and consequentially changes to the volume, the measures shall be stored in the calibration area at least 6 hours before calibration. This is especially important in places with large temperature differences during the day. During on site calibration exposure to direct solar radiation should be avoided.

3.9 Conditions for auxiliary equipment used during calibrations

Other auxiliary equipment, such as equipment for measuring environmental conditions shall be in the calibration area at least one hour before calibration of the SCM.

3.10 Adjusting the volume of the standard capacity measure

Usually metal standard capacity measures have a removable scale that can be adjusted. If the laboratory determines that adjustment shall be done, this must be agreed with customers/owner. When adjustment is performed the calibration results before and after adjustment must be reported.

3.11 Cleaning

The standard capacity measure must be sufficiently clean to permit uniform wetting of the internal surface. When clean, the walls will be uniformly wetted and the water will adhere to the interior surface in a uniform film. Lack of cleanliness causes irregularities in capacity by distorting the water surface. If the calibration is done without cleaning this should be stated in the calibration certificate.
The liquids usually used for cleaning are cleaning solutions (commercially available from laboratory suppliers), alcohol, and water. The choice of cleaning agent to be used depends on the nature of the contaminant (do not use materials which will attack, discolor or swell the material, always follow the instructions of the manufacturer) and the dimensions of the SCM. After cleaning with the cleaning solution, if applicable, the measure should be rinsed with ethyl alcohol, then thoroughly rinsing with tap water and dried at room temperature.
It is not necessary to dry any measure marked “to deliver.”

3.12 Meniscus reading

Meniscus reading is one of the most important contributions to measurement uncertainty in volumetric calibration of standard capacity measures, being critical for volumes greater than 200 L. In those cases neck thickness are 20 cm or more and often the water level is fluctuating due to the unstable base.
Meniscus reading has a big influence on measurement reading and consequently repeatability of the result. The variability of meniscus settings and scale readings made by a single operator depends upon his/her individual expertise (figure 1).
Depending on how clean the standard capacity measure is, the meniscus can be curved up or down. Usually with clean water used for calibrations and with measures with small necks the meniscus is curved downwards. For measures with broader necks it can be almost impossible to see the meniscus clearly, so this contribution should be included in calculations.
The meniscus shall be set so that the plane of the upper edge of the graduation line is horizontally tangential to the lowest point of the meniscus, the line of sight being in the same plane (see Figure 2).
The lighting should be arranged so that the meniscus appears dark and distinct in outline. For this purpose, it should be viewed against a white background and shaded from undesirable illumination. This can be achieved, for example, by securing a strip of black or blue paper directly below the level of the graduation line or ring mark or by using a short section of thick black rubber tubing cut open at one side and of such size as to clasp the tube firmly. Parallax is avoided when the graduation lines are of sufficient length to be seen at the front and back of the volumetric instrument simultaneously.
On volumetric instruments which have graduation lines on the front only, parallax can be made negligible when making a setting on the top edge of the line by using the black shading strip, taking care that the top edge of this is in a horizontal plane. In this case, the eye shall be placed so that the front and back portions of the top edge appear to be coincident [2].

Figure 1 – Standard test measure meniscus

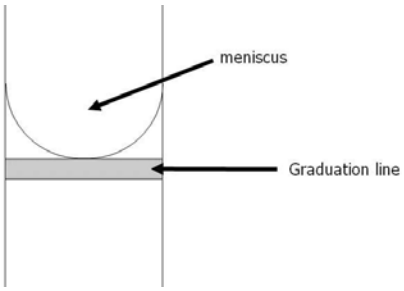

Figure 2 – Meniscus Setting

3.13 Coefficient of cubical thermal expansion of water

The coefficient of thermal expansion of water can be determined using equation (1) developed according to the data presented at [4]:

$$\beta = (-0{,}1176 \times t^2 + 15{,}846 \times t - 62{,}677) \times 10^{-6} / ^{o}\mathrm{C} \qquad (1)$$

Where :

$$t = \frac{t_{RS} + t_{SCM}}{2}$$

and
t_{RS} is the temperature of water in the filled Reference Standard (RS) before pouring
t_{SCM} is the temperature of water in the SCM after its filling

A more complex formula, derived from the Tanaka equation of the water density [5], with a standard uncertainty of 2×10^{-6} /°C, can be used:
If t_0 = reference value temperature, $t - t_0 = \Delta t$ and $a'_i = a_i + t_0$ we have:

$$\beta = \frac{\left[(\Delta t + a_1')^2 + a_2' \times \left(\Delta t + a_1' \times \left(2 - \frac{a_1'}{a_4'}\right)\right)\right]}{a_3 \times (\Delta t + a_4') - (\Delta t + a_1')^2 \times (\Delta t + a_2')} \quad (2)$$

Where,
t = water temperature, in °C
a_1 = -3,983035 ºC
a_2 = 301,797 ºC
a_3 = 522528,9 (ºC)^2
a_4 = 69,34881 ºC
a_5 = 0,999974950 g/mL

3.14 Coefficient of cubical thermal expansion of the material

The coefficient of cubical thermal expansion depends on material that the standard capacity measure is made. The most common materials are described in the following table [2,6,7]:

Table 1 – Coefficient of cubical thermal expansion of standard materials

Standard capacity measure material	Coefficient of cubical thermal expansion of the material / °C
Carbon fiber	1×10^{-6}
Borosilicate glass 3.3	$9{,}9\times10^{-6}$
Borosilicate glass 5.0	15×10^{-6}
Soda-Lime Glass	27×10^{-6}
Steel	33×10^{-6}
Mild carbon	$33{,}5\times10^{-6}$
Stainless Steel grade 304	$51{,}8\times10^{-6}$
Stainless Steel grade 316	$47{,}7\times10^{-6}$
Stainless Steel 17-4 PH	$32{,}4\times10^{-6}$
Copper – zinc alloy (brass)	54×10^{-6}
Aluminum	69×10^{-6}
PVC	80×10^{-6}

Where the cubical expansion coefficient is given on the type plate of the standard capacity measure then this value should be used.

4. Calibration procedure

4.1 Preparation

Select a reference standard of known volume at a specified reference temperature and a known coefficient of cubical thermal expansion.
An inspection and cleaning process is performed on standard capacity measure, noting any defects such as bumps, leaky valves and leaky tubes.
Level the SCM and RS either by attached or built-in levels or by placing a level across the top of the open neck. In case of a SCM with double scale (or window) on the neck, the front window has to be observed

by looking at it from the back window, and vice-versa. The levelling of the tank is adjusted so that both scales must appear correct when viewed from alternate sides.
If the standard capacity measure is to be calibrated to contain (In type), the internal surfaces of the vessel must be dry; for delivery (Ex type) the internal surface must be wet.
The volumetric method of calibration may be used in two different approaches: withdrawing or filling.

4.2 Calibration using the filling method

In this method the standard capacity measure is calibrated by filling it with a pre-determined volume of water delivered from a reference standard measure.

a) Measure and record the air temperature.
b) Level the reference standard. Fill the reference standard to a selected point of the neck scale or until it overflows with water from a storage tank. Measure the water temperature, in ºC, and record it. Agitate the water in the gage tube to get a uniform meniscus in the case of a neck scale reference standard. Record the volume at the working temperature of the reference standard.
c) Deliver the liquid into the levelled standard capacity measure for wetting purposes (only Ex type). Wait for the dripping time indicated on the reference standard (eg.30 s).
d) Empty the SCM and wait for the dripping time. Both standards are now preconditioned to correspond to the "empty" state for their intended use.
e) Fill the reference standard again in the same way. Measure the water temperature in the reference standard and deliver the liquid into the standard capacity measure. Wait the time indicated on the reference standard.
f) Measure and record the temperature of the water in the SCM.
g) Record the neck reading of the SCM and adjust the volume (if the SCM is equipped by correction device/scale and upon costumer request) to its nominal volume at the reference temperature (e.g. 20 ºC), or determine the error of the SCM by removing or adding a known quantity of water until the volume corresponds to the nominal volume mark of the SCM.
h) Repeat the procedure as many times as required (2 times minimum recommended).
i) In case of adjustment of the scale, it must be fixed with appropriated tools in order to avoid possible changes in time.

The indication error of the SCM will be the difference between the value of the volume read in its scale and V_t, where V_t, is the volume delivered from the reference measure at temperature t.

4.3 Calibration using the withdrawing method

In this method the standard capacity measure is calibrated by determining the volume of water it delivers by emptying it into a reference standard measure.

a) Measure and record the air temperature.
b) Level the standard capacity measure (SCM). Fill the SCM (pre-wetted or dry depending on the use). Measure the water temperature and deliver the liquid into the pre-wetted "contained" reference standard (RS). Wait the time indicated on the SCM.
c) Measure and record the temperature of the water in the RS.
d) Determine the delivered volume at the reference line and the error of the SMC by removing or adding a known quantity of water until the volume corresponds to the nominal volume mark of the RS.
e) Repeat the procedure as many times as required (2 times minimum recommended).
f) In case of adjustment of the scale, it must be fixed with appropriated tools in order to avoid possible changes in time.

The indication error of the SCM will be the difference between the value of the volume read in the reference standard scale and V_t, where V_t, is the volume delivered from the reference measure at temperature t.

In a situation where a smaller RS is used to calibrate a larger SCM, each step described above in 4.2 and 4.3 must be repeated the appropriate number of times, n, up to a max of 10. The cumulative volume is calculated and the average temperature of the water of all fills is determined.

5. Determination of the volume

Determine the volume at a reference temperature t (usually 20 º C), for each calibration value, using the following formula:

$$V_t = V_0\left[1-\gamma_{RS}(t_{0RS}-t_{RS})+\beta(t_{SCM}-t_{RS})+\gamma_{SCM}(t-t_{SCM})\right] \quad (3)$$

V_t - Volume of the standard capacity measure at t °C
V_0 - Volume of the reference standard at the reference temperature t_{0RS}
t_{0RS} - Reference temperature of the RS
t - Reference temperature of the SCM
t_{RS} - Temperature of the liquid in the RS
t_{SCM} - Temperature of the liquid in the SCM
γ_{RS} - Coefficient of cubical thermal expansion of the material of the RS
β - Coefficient of cubical thermal expansion of the liquid (water) at the average test temperature: 0,5 (t_{RS} + t_{SCM})
γ_{SCM} - Coefficient of cubical thermal expansion of the material of the SCM

For multiple fillings $V_0 = n \times V_{0i}$, where V_{0i} is the reference standard volume and t_{RS} is the average temperature of the n measurements.

6. Procedure for estimating measurement uncertainty

6.1 Parameters that affect the uncertainty in volumetric determination of volume

The main parameters that can influence the quality of the result during the volumetric calibration of standard capacity measures are described.

6.1.1 Reference standard

The reference standard uncertainty is one of the most important components in the determination of the uncertainty of the volume of the standard capacity measure. This reference standard must be calibrated using the gravimetric or volumetric method depending on the needed uncertainty accuracy.

6.1.2 Water temperature of the reference standard

Water temperature in the reference standard must be measured before the water from the reference standard is poured to the standard capacity measure or just after its volume reading is done.
In the case where more fillings are necessary to fill the SCM, the water temperature of each fill is recorded and the average value used to determine the volume.
The thermometer used should have a resolution of at least 0,1 ºC or larger uncertainties can be expected.

6.1.3 Water temperature of the standard capacity measure

Water temperature in the standard capacity measure being calibrated must be measured every time before the volume reading is done or before delivery.
Due to the duration of the calibration procedure in some circumstances (especially in bigger standard capacity measures) there may be no way to avoid temperature differences between various parts of the measure. This can be reduced to negligible values (0,02 ºC) if the water is effectively stirred with a rod just before the volume reading is taken. When using a stirring rod, the temperature of the rod shall be as close as possible to the temperature of the water in order to avoid heat transfer. If this is not possible,

temperature can be measured in different, representative locations and the average between the measured temperatures used.
The thermometer used should have a resolution of at least 0,1 ºC.

6.1.4 Standard capacity measure features

The characteristics of the standard capacity measure under calibration, e.g. the scale resolution or the expansion coefficient of the material, must also be considered.

6.1.5 Water expansion coefficient

The uncertainty of the water expansion coefficient should be determined.

6.1.6 Operator

The operator can directly influence the measurement in the meniscus reading, in the filling and emptying procedure or in the handling of the equipment.

6.1.7 Other influences

There are some additional factors that can contribute to the quality of the results like air bubbles in the water, the variation in the amount of liquid residue (in case of calibration of standard capacity measure in delivery mode "Ex") and liquid loss due to evaporation. The uncertainty of these additional factors must be determined.

6.2 General procedure for the uncertainty calculation

In this document, the evaluation of measurement uncertainty follows the methods described in JCGM 100:2008 [8]. The method consists of the following steps:

1. Expressing, in mathematical terms, the relationship between the measurand and its input quantities;
2. Determining the expectation value of each input quantity;
3. Determining the standard uncertainty of each input quantity;
4. Determining the degree of freedom for each input quantity;
5. Determining all covariances between the input quantities;
6. Calculating the expectation value for the measurand;
7. Calculating the sensitivity coefficient of each input quantity;
8. Calculating the combined standard uncertainty of the measurand;
9. Calculating the effective degrees of freedom of the combined standard uncertainty;
10. Choosing an appropriate coverage factor, k, to achieve the required confidence level;
11. Calculating the expanded uncertainty.

It should be noted that for steps 6 to 11 suitable computer programs exist which can replace manual calculation. Step 1 is the most important part in the whole GUM procedure.

6.3 Procedure for calculating uncertainty in volumetric determination of volume

6.3.1 Mathematical expression of the volume V_t

$$V_t = V_0\left[1-\gamma_{RS}(t_{0RS}-t_{RS})+\beta(t_{SCM}-t_{RS})+\gamma_{SCM}(t-t_{SCM})\right]+\delta V_{men}+\delta V_{rep}+\delta V_{add} \quad (4)$$

6.3.2 Sources of uncertainty in volumetric volume determination

When the input quantities of the measurand, i.e. the volume V_t, in equation (4), are identified it is then possible to identify the sources of uncertainty coming from the different input quantities; these are:

- Reference standard, V_0
- Water temperature of the reference standard, t_{RS}

- Water temperature of the standard capacity measure, t_{SCM}
- Coefficient of cubical thermal expansion of the reference standard material, γ_{RS}
- Coefficient of cubical thermal expansion of the standard capacity measure material, γ_{SCM}
- Coefficient of cubical thermal expansion the water, β
- Meniscus reading, δV_{men}
- Measurement repeatability, δV_{rep}
- Additional factors, δV_{add}

6.3.3 Standard uncertainty of each input quantity

In the following, the different expressions of these uncertainties are displayed.

6.3.3.1 Reference standard

The uncertainty contribution for the calibration of the reference standard will be given by:

$$u_{cal}(V_0) = \frac{U_{cal}(V_0)}{k} \tag{5}$$

where:
$U_{cal}(V_0)$ is the expanded measurement uncertainty of the reference standard, in volume units (the value is from the last calibration certificate of the reference standard); k is the coverage factor. If the uncertainty in the certificate has been estimated for a 95,45% confidence level, then k =2.
If the same reference standard is used more than once the following formula can be used:

$$U_{cal}(V_0) = n \times U_{cal}(V_{0,i}) \tag{6}$$

where:
n - the times the reference standard is used.
$U_{cal}(V_{0,i})$ - the volume uncertainty of the reference standard i used n times, in L.
This equation (6) expresses a conservative uncertainty estimate (total correlation).
On the other hand reference standards may drift between calibrations. This fact adds an additional uncertainty contribution given by:

$$u_{drift}(V_0) = \frac{\delta_{drift}(V_0)}{\sqrt{12}} \tag{7}$$

Where $\delta_{drift}(V_0)$ is the difference for reference values between consecutive calibrations, in L.
The overall uncertainty for the reference standard would be given by equation (8):

$$u(V_0) = \sqrt{u_{cal}^2(V_0) + u_{drift}^2(V_0)} \tag{8}$$

6.3.3.2 Water temperature of the reference standard

Equation (9) is a possible expression for this uncertainty component:

$$u(t_{RS}) = \left[u^2{}_{cal}(t_{RS}) + u^2{}_{drift}(t_{RS}) + u^2{}_{\Delta t}(t_{RS})\right]^{1/2} \tag{9}$$

where:
$u_{cal}(t_{RS})$ - standard uncertainty of the thermometer in the reference standard, in ºC
$u_{drift}(t_{RS})$ - estimate of the uncertainty caused by possible drift and ageing of the temperature measuring system after its calibration, in ºC
$u_{\Delta t}(t_{RS})$ - estimate of the uncertainty of the average water temperature caused by temperature differences (and temperature gradients) that can be measured or estimated between bottom and top of the instrument under calibration, in ºC

Note: the maximum temperature difference between various parts of the measure can be reduced if the water is effectively stirred with a rod (care has to be taken to ensure the rod is at the same temperature as the water before use to avoid heat transfer). If this is not possible, temperature can be measured in different, representative locations; having defined $t_{\max}$ and $t_{\min}$ as the highest and lowest temperatures found, the standard deviation of a rectangular distribution, namely, $u_{\Delta t}(t_{RS}) = (t_{\max} - t_{\min})/\sqrt{12}$ is an upper limit for the uncertainty of the mean temperature.
Temperature gradients can occur in any direction within the measure. The personal judgment of the operator could be a guide towards the direction of a realistic estimation of this uncertainty depending on the prevailing environmental conditions (e.g. exposure to sunlight, air streams, air conditioning outlets, etc).
If the same reference standard is used more than once the mean average of the temperature of each filling should be used and therefore the standard deviation of the temperature measurements should be taken into consideration in the uncertainty budget. In this case the following formula should be used for the water temperature of the reference standard:

$$u(t_{RS}) = \left[u^2_{cal}(t_{RS}) + u^2_{rep}(t_{RS}) + u^2_{drift}(t_{RS}) + u^2_{\Delta t}(t_{RS})\right]^{1/2} \quad (10)$$

where:
$u_{rep}(t_{RS})$ - standard deviation of the mean of a series of independent temperature measurements, in ºC

6.3.3.3 Water temperature of the standard capacity measure

Equation (11) is a possible expression for this uncertainty component:

$$u(t_{SCM}) = \left[u^2_{cal}(t_{SCM}) + u^2_{drift}(t_{SCM}) + u^2_{\Delta t}(t_{SCM})\right]^{1/2} \quad (11)$$

The components are the same ones as in the case for the reference standard, but applied for the standard capacity measure.
If the same thermometer is used for measuring t_{RS} and t_{SCM} there is a strong correlation, which is difficult to calculate. In order to avoid this problem, there is the possibility to redefine the quantities to be measured and change the model. If $\delta t = t_{SCM} - t_{RS}$ equation (4) can be expressed as:

$$\begin{aligned} V_t &= V_0\left[1 - \gamma_{RS}(t_{0RS} - t_{RS}) + \beta \cdot \delta t + \gamma_{SCM}(t - \delta t + t_{RS})\right] + \delta V_{men} + \delta V_{rep} + \delta V_{add} = \\ &= V_0\left[1 - \gamma_{RS} \cdot t_{0RS} + \gamma_{SCM} \cdot t + (\beta - \gamma_{SCM}) \cdot \delta t + (\gamma_{SCM} + \gamma_{RS}) \cdot t_{RS}\right] + \delta V_{men} + \delta V_{rep} + \delta V_{add} \end{aligned} \quad (12)$$

The uncertainty components for t_{RS} will be the same as the ones in equation (9).

For δt the uncertainty contribution is given by

$$u(\delta t) = \left[u^2_{lin}(\delta t) + 2 \times u^2_{res}(\delta t) + u^2_{\Delta t}(\delta t)\right]^{1/2} \quad (13)$$

where
$u_{lin}(\delta t)$ – standard uncertainty due to the linearity of the thermometer between t_{RS} and t_{SCM}, in ºC
$u_{res}(\delta t)$ – estimate of the uncertainty caused by the finite resolution of the instrument, in ºC
$u_{\Delta t}(\delta t)$ - estimate of the uncertainty of the average water temperature caused by temperature differences and temperature gradients in the standard capacity measure that can be measured or estimated between bottom and top of the instrument under calibration, in ºC.

6.3.3.4 Coefficient of cubical thermal expansion of the material of the reference standard and standard capacity measure

The thermal expansion coefficients are dependent on knowledge of the actual material of the standard and on the source of data which provides the user with an appropriate value. Data from the literature or

manufacturer should be used and this would be expected to have a (standard) uncertainty between 5 % and 10 %. If the upper and lower limits of this table values are known, the standard uncertainty can also be determined applying a rectangular probability distribution at these limits.

6.3.3.5 Coefficient of cubical thermal expansion of the water

The thermal expansion coefficients of the water are dependent on the source of data which provides the user with an appropriate value. Data from the literature should be used and this would be expected to have an (standard) uncertainty between 5 % and 10 %. If the upper and lower limits of this table values are known, standard uncertainty can also be determined applied rectangular probability distribution at these limits. If alternatively formula (2) is used, a standard uncertainty of 2×10^{-6} $^{o}C^{-1}$, can be used.

6.3.3.6 Meniscus reading

The variability of meniscus settings and scale readings made by a single operator depends upon his/her individual expertise. This reading directly influences the experimental standard deviation; therefore only type B components of meniscus and scale reading uncertainty should be estimated and compiled. These components are intended to take into account the unavoidable bias (or average deviations of the positioning of meniscus that is characteristic of a given operator in a given artifact) with reference to the ideal position defined by ref. [2] ("the meniscus shall be set so that the plane of the upper edge of the graduation line is horizontally tangential to the lowest point of the meniscus, the line of sight being in the same plane"). It is recommended that the estimate of this contribution to uncertainty be separately declared in calibration certificates, in order to allow users (who are responsible for evaluating actual uncertainties occurring during the use of their own instrument) to estimate and compose a supplementary contribution if they are unable to approximate, the correct meniscus positioning within the same uncertainty limits. Several approaches can be used to determine the uncertainty of the meniscus [9].

6.3.3.7 Resolution of the standard capacity measure

In the case where the transferred volume should be adjusted by removing or adding a known quantity of water until the volume corresponds to the nominal volume mark of the SCM the sensitivity of the scale should be taken in to account (0,5 mm of the neck), and therefore the uncertainty of the resolution should be added to the uncertainty budget.

6.3.3.8 Measurement repeatability

Equation (14) is a possible expression for this type A uncertainty component:

$$u\left(\delta V_{rep}\right)=\frac{s\left(V_t\right)}{\sqrt{n}} \tag{14}$$

where:
$s(V_t)$ - standard deviation of a series of independent volume measurements, in L
n - number of measurements

Note: the value of volume that will be given as a result of n repeated measurements is the arithmetic mean of the n results, therefore the type A uncertainty component is the standard deviation of the mean, $u(\delta V_{rep})$ as defined above. However, it is recommended that the number of measurements n and their standard deviation $s(V_t)$ be quoted in calibration reports or certificates, because if the user is going to make single, not averaged measurements, its type A uncertainty contribution will not be $u(\delta V_{rep})$, but the standard deviation of the whole population of possible measurements, whose best estimate can be determined knowing n and $s(V_t)$.

6.3.3.9 Additional uncertainty factors

There are some additional uncertainty factors that can be contribute to the combined uncertainty:

- Air bubbles in the water;

- Variation in the amount of liquid residue (in case of calibration of standard capacity measure in delivery mode "Ex");
- Loss due to evaporation (in order to avoid loss by evaporation it`s a good laboratory recommendation to close the top of the standard capacity measure with a cap; where the calibration is performed using multiple deliveries from the reference standard than the filling should be undertaken through a smaller aperture).

This additional uncertainties would be expected to have a (standard) uncertainty of 0,01 %.
Based on practical experience some values of additional uncertainty factors can be recommended.

Table 2 – Standard uncertainty values for additional factors

Additional uncertainty factors	**Nominal volume**										
	2 L	5 L	10 L	20 L	50 L	100 L	200 L	400 L	500 L	1000 L	2000 L
Air bubbles in the water (mL)	0,02	0,05	0,1	0,2	0,5	1	2	4	5	10	20
Loss by the evaporation (mL)	0,03	0,014	0,25	0,51	1,3	2,6	5,2	10,4	13	26	52
Variation in the amount of liquid residue (mL)	0,24	0,45	0,55	0,68	1,7	3,4	6,8	13,6	17	34	68

6.3.4 Sensitivity coefficient of each input quantity

The sensitivity coefficients of each input quantity can be determined as following:

6.3.4.1 Reference standard

$$\frac{\partial V_t}{\partial V_0} = \left[1 - \gamma_{RS}\left(t_{0RS} - t_{RS}\right) + \beta\left(t_{SCM} - t_{RS}\right) + \gamma_{SCM}\left(t - t_{SCM}\right)\right] \quad (15)$$

6.3.4.2 Water temperature in the reference standard

$$\frac{\partial V_t}{\partial t_{RS}} = \left[V_0\left(\gamma_{RS} - \beta\right)\right] \quad (16)$$

6.3.4.3 Water temperature in the standard capacity measure

$$\frac{\partial V_t}{\partial t_{SCM}} = \left[V_0\left(\beta - \gamma_{SCM}\right)\right] \quad (17)$$

6.3.4.4 Coefficient of cubical thermal expansion of the reference standard

$$\frac{\partial V_t}{\partial \gamma_{RS}} = \left[-V_0\left(t_{0RS} - t_{RS}\right)\right] \quad (18)$$

6.3.4.5 Coefficient of cubical thermal expansion of the standard capacity measure

$$\frac{\partial V_t}{\partial \gamma_{SCM}} = \left[V_0 (t_{0SCM} - t_{SCM})\right] \tag{19}$$

6.3.4.6 Coefficient of cubical thermal expansion of the water

$$\frac{\partial V_t}{\partial \beta} = \left[V_0 (t_{SCM} - t_{RS})\right] \tag{20}$$

6.3.4.7 Meniscus reading

$$\frac{\partial V_t}{\partial \delta V_{men}} = 1 \tag{21}$$

6.3.4.8 Measurement repeatability

$$\frac{\partial V_t}{\partial \delta V_{rep}} = 1 \tag{22}$$

6.3.4.9 Additional factors

$$\frac{\partial V_t}{\partial \delta V_{add}} = 1 \tag{23}$$

6.3.5 Combined standard uncertainty of measurand

Within the hypothesis of the applicability of the propagation law of uncertainties, the combined standard uncertainty of the measurand is expressed as:

$$u^2(V_t) = \sum_i \left(\frac{\partial V_t}{\partial x_i} \times u(x_i) \right)^2 \tag{24}$$

Using the expressions of the parts 6.2 and 6.3, the resultant combined standard uncertainty of the measurand is:

$$u(V_t) = \left[\begin{array}{l} \left(\frac{\partial V_t}{\partial V_0} \right)^2 \times (u(V_0))^2 + \left(\frac{\partial V_t}{\partial t_{RS}} \right)^2 \times (u(t_{RS}))^2 + \left(\frac{\partial V_t}{\partial t_{SCM}} \right)^2 \times (u(t_{SCM}))^2 + \left(\frac{\partial V_t}{\partial \gamma_{RS}} \right)^2 \times (u(\gamma_{RS}))^2 \\ + \left(\frac{\partial V_t}{\partial \gamma_{SCM}} \right)^2 \times (u(\gamma_{SCM}))^2 + \left(\frac{\partial V_t}{\partial \beta} \right)^2 \times (u(\beta))^2 + (u(\delta V_{men}))^2 + (u(\delta V_{rep}))^2 + (u(\delta V_{add}))^2 \end{array} \right]^{\frac{1}{2}} \tag{25}$$

6.3.6 Evaluation of any existing covariances

Equation (24) and Equation (25) do not include any covariances terms. If some other correlations are identified they must be evaluated and introduced if influential.

6.3.7 Choice of an appropriate coverage factor (*k*)

Having computed the standard uncertainty of the measurand through the composition of all contributions, assuming that the distribution of the standard uncertainty is normal, its number of degrees of freedom ν_{eff}, can be estimated by means of the Welch-Satterthwaite formula [8]:

$$\nu_{eff} = \frac{u_V^4}{\sum_{i=1}^{N} \frac{u_i^4}{\nu_i}} \tag{26}$$

u_V – combined uncertainty of the determined volume
u_i – standard uncertainty of each component
ν_i – degrees of freedom

This allows the calculation of an appropriate coverage factor (*k*) for a given level of confidence, the most usual is 95%.

6.3.8 Expanded uncertainty

With the value of the coverage factor (*k*) and of the combined standard uncertainty of the measurand, the expanded uncertainty is deduced by:

$$U = k \times u(V_t) \tag{27}$$

7. Practical application

7.1 Measurement problem

In order to apply numerical values to the uncertainty calculation procedure described above, a 2000 L proving tank was calibrated using an overflow reference standard of 500 L (4 times). The data is summarized in table 3.

Table 3 – Summary of data for volumetric calibration of a 2000 L proving tank (average values)

Input Quantity x_i	**Value of the input quantity**
Indication error of SCM	0,50 L
Reference standard volume at 20 ºC	500,26 L
Reference standard water temperature	20,50 ºC
Reference standard expansion coefficient	$51{,}8\times10^{-6}$ /ºC
Standard capacity measure water temperature	20,45 ºC
Water expansion coefficient	$2{,}125\times10^{-4}$ /ºC
Standard capacity measure expansion coefficient	$51{,}8\times10^{-6}$ /ºC
Meniscus reading of the RS	-
Meniscus reading of the SCM	0,025 L
Measurement repeatability	0,05 L
Additional factors	0,14 L

After analyzing the measurement problem and determining the indication error of the SCM based on V_t, it is necessary to determine the standard uncertainty of each input quantity, the sensitivity coefficients, the combined uncertainty, the degrees of freedom and corresponding k factor and finally the expanded uncertainty. The pertinent aspects of this example as discussed in this and the followings subclauses are summarized in table 4.

7.2 Determination of the standard uncertainty of each input quantity

7.2.1 Reference standard

The expanded uncertainty for the calibration of the reference standard is 0,19 L. This RS was used 4 times. Considering that the reference standard used does not have any drift between consecutive calibrations the overall uncertainty for the reference standard is:

$$u(V_0) = \sqrt{n^2 \times u_{cal}^2(V_0) + u_{drift}^2(V_0)} = \sqrt{\left(\frac{4 \times 0{,}19}{2}\right)^2 + 0} = 0{,}38 \text{ L}$$

7.2.2 Water temperature of the reference standard

The standard uncertainty of the water temperature was obtained from the calibration certificate of the thermometer calibration $U_{cal}(t_{RS})$ = 0,005 ºC, using a coverage factor of 2. The standard deviation of the mean of the 4 temperature measurements was 0,035 ºC. If we consider that the drift and temperature gradient are zero then:

$$u(t_{RS}) = \left[u^2{}_{cal}(t_{RS}) + u^2{}_{rep}(t_{RS}) + u^2{}_{drift}(t_{RS}) + u^2{}_{\Delta t}(t_{RS})\right]^{1/2} = \left[\left(\frac{0{,}005}{2}\right)^2 + (0{,}035)^2 + 0 + 0\right]^{1/2} = 0{,}0354\ ^{\circ}\text{C}$$

7.2.3 Water temperature of the standard capacity measure

The standard uncertainty of the water temperature in the SCM was obtained from the calibration certificate of the thermometer calibration $U_{cal}(t_{SCM})$ = 0,01 ºC, using a coverage factor of 2. The temperature is only measured after all the filing is completed and therefore a mean value is not used. If we consider that the drift and temperature gradient are zero then:

$$u(t_{SCM}) = \left[u^2{}_{cal}(t_{SCM}) + u^2{}_{drift}(t_{SCM})^2 + u^2{}_{\Delta t}(t_{SCM})\right]^{1/2} = \left[\left(\frac{0{,}01}{2}\right)^2 + 0 + 0\right]^{1/2} = 0{,}005\ ^{\circ}\text{C}$$

7.2.4 Coefficient of cubical thermal expansion of the material of the reference standard and standard capacity measure

The thermal expansion coefficient of the reference standard and of the standard capacity measure is given by table 1 as γ = 51,8×10^{-6} /ºC, with an expanded uncertainty of 5 %; in the lack of a more informative statement, a rectangular probability distribution is assumed. The relevant standard uncertainty is therefore:

$$u(\gamma_{RS}) = u(\gamma_{SCM}) = \frac{2{,}59 \times 10^{-6}}{\sqrt{3}} = 1{,}50 \times 10^{-6}\ /^{\circ}\text{C}$$

7.2.5 Coefficient of cubical thermal expansion of the water

The thermal expansion coefficient of the water is given by equation (1) for $t = \frac{t_{RS} + t_{SCM}}{2}$ with an expanded uncertainty of 5 %. In this study case β = 0,0002125 /ºC. A rectangular distribution is assumed. The relevant standard uncertainty is therefore:

$$u(\beta) = \frac{1{,}06 \times 10^{-5}}{\sqrt{3}} = 6{,}13 \times 10^{-6} \ /^{o}\mathrm{C}$$

7.2.6 Meniscus reading of the standard capacity measure

The meniscus position of the standard capacity measure was determined using geometrical method calculation taking into consideration the neck diameter and the width of the scale mark [9, eq.9]. The value of the standard uncertainty of the meniscus reading is therefore 0,0144 L, with a rectangular distribution:

$$u(\delta V_{menSCM}) = \frac{0{,}0249}{\sqrt{3}} = 0{,}0144 \ \mathrm{L}$$

7.2.7 Measurement repeatability

Following equation (14), the type A uncertainty component can be determined by:

$$u(\delta V_{rep}) = \frac{s(V_o)}{\sqrt{n}} = \frac{0{,}05}{\sqrt{3}} = 0{,}0289 \ \mathrm{L}$$

In this practical application the calibration of the 2000 L tank was repeated 3 times, therefore n = 3.

7.2.8 Additional uncertainty factors

This additional uncertainties would be expected to have a (standard) uncertainty of 0,14 L according to table 2.

7.3 Sensitivity coefficient of each input quantity

7.3.1 Reference standard

$$\frac{\partial V_t}{\partial V_0} = \left[1 - \gamma_{RS}(t_{0RS} - t_{RS}) + \beta(t_{SCM} - t_{RS}) + \gamma_{SCM}(t - t_{SCM})\right] = 1$$

7.3.2 Water temperature in the reference standard

$$\frac{\partial V_t}{\partial t_{RS}} = \left[V_0(\gamma_{RS} - \beta)\right] = -3{,}22 \times 10^{-1} \ \mathrm{L/^{o}C}$$

7.3.3 Water temperature in the standard capacity measure

$$\left(\frac{\partial V_t}{\partial t_{SCM}}\right) = \left[V_0(\beta - \gamma_{SCM})\right] = 3{,}22 \times 10^{-1} \ \mathrm{L/^{o}C}$$

7.3.4 Coefficient of cubical thermal expansion of the reference standard

$$\frac{\partial V_t}{\partial \gamma_{RS}} = \left[-V_0(t_{0RS} - t_{RS})\right] = 900 \ \mathrm{L\,^{o}C}$$

7.3.5 Coefficient of cubical thermal expansion of the standard capacity measure

$$\frac{\partial V_t}{\partial \gamma_{SCM}} = \left[V_0(t_{0SCM} - t_{SCM})\right] = -1000 \ \mathrm{L\,^{o}C}$$

7.3.6 Coefficient of cubical thermal expansion of the water

$$\frac{\partial V_t}{\partial \beta} = \left[V_0 (t_{SCM} - t_{RS})\right] = 100 \text{ L } ^{\circ}\text{C}$$

7.3.7 Meniscus reading

$$\frac{\partial V_t}{\partial \delta V_{men}} = 1$$

7.3.8 Measurement repeatability

$$\frac{\partial V_t}{\partial \delta V_{rep}} = 1$$

7.3.9 Additional factors

$$\frac{\partial V_t}{\partial \delta V_{add}} = 1$$

7.4 Combined standard uncertainty of measurand

The combined uncertainty $u(V_t)$ is calculated from equation 25. The individual input quantity values are collected and substituted into this expression to obtain:

$$u(V_t) = \left[\begin{array}{l} \left(\frac{\partial V_t}{\partial V_0}\right)^2 \times \left(u(V_0)\right)^2 + \left(\frac{\partial V_t}{\partial t_{RS}}\right)^2 \times \left(u(t_{RS})\right)^2 + \left(\frac{\partial V_t}{\partial t_{SCM}}\right)^2 \times \left(u(t_{SCM})\right)^2 + \left(\frac{\partial V_t}{\partial \gamma_{RS}}\right)^2 \times \left(u(\gamma_{RS})\right)^2 \\ + \left(\frac{\partial V_t}{\partial \gamma_{SCM}}\right)^2 \times \left(u(\gamma_{SCM})\right)^2 + \left(\frac{\partial V_t}{\partial \beta}\right)^2 \times \left(u(\beta)\right)^2 + \left(u(\delta V_{menRS})\right)^2 + \left(u(\delta V_{rep})\right)^2 + \left(u(\delta V_{add})\right)^2 \end{array} \right]^{\frac{1}{2}} = 0{,}406 \text{ L}$$

7.5 Evaluation of any existing covariances

There are no significant covariances.

7.6 Choice of an appropriate coverage factor (k)

To calculate the coverage factor (k), it's necessary to estimate the degrees of freedom, ν_{eff}, using the Welch-Satterthwaite formula:

$$\nu_{eff} = \frac{u_V^4}{\sum_{i=1}^{N} \frac{u_i^4}{\nu_i}} = 65$$

This gives a coverage probability of approximately 95 %, the corresponding coverage factor is k = 2

7.7 Expanded uncertainty

The expanded uncertainty is calculated from:

$U = k \times u(V_t)$ = 2 × 0,406 = 0,81 L

A summary of the uncertainty calculation can be found in table 4.

Table 4 – Summary for the standard uncertainty components

Standard uncertainty component $u(x_i)$	**Source of uncertainty**	**Value of standard uncertainty $u(x_i)$**	$c_i \equiv \frac{\partial f}{\partial x_i}$	$u_i(V_0) \equiv \lvert c_i \rvert u(x_i)$ **(L)**	ν_{eff}
$u(V_0)$	Volume of the RS	0,38 L	1,00	$3{,}8 \times 10^{-1}$	50
$u(t_{RS})$	Water temperature of RS	$3{,}54\times10^{-2}$ ºC	$-3{,}22\times10^{-1}$	$1{,}14 \times 10^{-2}$	3
$u(t_{SCM})$	Water temperature of SCM	5×10^{-3} ºC	$3{,}22\times10^{-1}$	$1{,}61 \times 10^{-3}$	50
$u(\gamma_{RS})$	Coefficient of cubical thermal expansion of the RS material	$1{,}5 \times 10^{-6}$ /ºC	900	$1{,}35 \times 10^{-3}$	∞
$u(\gamma_{SCM})$	Coefficient of cubical thermal expansion of the RS material	$1{,}5 \times 10^{-6}$ /ºC	-1000	$1{,}50 \times 10^{-3}$	∞
$u(\beta)$	Coefficient of cubical thermal expansion of the water	$6{,}13 \times 10^{-6}$ /ºC	100	$6{,}14 \times 10^{-4}$	∞
$u(\delta V_{menSCM})$	Meniscus reading of the SCM	0,014 L	1	0,014	∞
$u(\delta V_{rep})$	Measurement Repeatability	0,0289 L	1	0,025	2
$u(\delta V_{add})$	Additional factors	0,14 L	1	0,14	∞
				$u_c(V_{t20}) = 0{,}406$ L $\nu_{eff}(V_{20}) = 65,\ k = 2$ $U(V_t) = 0{,}81$ L	

8. References

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