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

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This document was developed by the EURAMET e.V., Technical Committee for Flow.
Authors: Andrea Malengo (INRIM, Italy), Elsa Batista (IPQ, Portugal), Matjaz Gaber (MIRS, Slovenia), Erik Smits (VSL, Netherlands), Gunn Kristin Svendsen (JV, Norway)

The authors of the current version acknowledge the contributions from Corinna Kroner (PTB, Germany) and Isabelle Care (LNE-CETIAT, France).

EURAMET e.V.
Bundesallee 100
38116 Braunschweig
Germany

E-Mail: secretariat@euramet.org
Phone: +49 531 592 1960

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Version 3.0 (02/2024)

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

Purpose

This document provides guidance to the calibration of a standard capacity measure or proving tank utilising the volumetric calibration method and to the evaluation of the measurement uncertainty.

The guide aims to harmonise the procedures used by organisations that apply this method of calibration in their laboratories or in the field of activity.

The current version reflects the actual practice applied in European National Metrology Institutes in terms of calibration procedures and calculation models for the calibration of Standard Capacity Measures (SCM) with the volumetric method. In particular, the changes to the previous version include new equations for the scale calibration and uncertainty evaluation, more detailed description of the calibration procedure as well as additional information in the example.

Content

1 INTRODUCTION

The accurate measurement of domestic and industrial consumption of water, fuels and other liquids 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 and mutual recognition of the measurements. These volume standards are standard capacity measures (SCM) and, depending on their nominal volume, can be divided in three categories: standard test measures, standard flasks and proving tanks [1].

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

- 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 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 for loading and unloading ships, rail and road tankers,
- large proving tanks.

The standard capacity measures can be calibrated at a higher level of accuracy by using the gravimetric method [2, 3]. At a working level, where the required accuracy of the measurement is lower or when the capacity of a standard capacity measure is so large that using weighing instruments is impracticable, the volumetric method can be used.

The volumetric method consists of delivering a known quantity of liquid to or from a calibrated standard (reference standard), to or from a standard capacity measure. This method can be used to calibrate SCM up to 10 000 L capacity, but other higher capacities can also be tested if technically possible.

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 TERMINOLOGY AND SYMBOLS

Symbols whose meaning are not self-evident, will be explained where they are first used.

The terminology used in this document is mainly based on existing documents, GUM [4], VIM [5], VIML [6], ISO 8222 [7], but there are some specific definitions that are explained below.

2.1 Volumetric method principle

In the volumetric method a known amount of liquid is delivered into a container up to a certain point (usually corresponding to a graduation mark on a scale) and this volume refers to a reference temperature applicable for the intended use of the measure under

calibration. When the measure is equipped with an adjustable indicating device or scale the calibrated volume can be adjusted to the nominal volume of the measure.

In the majority of cases the volumetric method is faster and easier than the gravimetric or geometric method provided that certain laboratory set up arrangements are available. 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 (at least 3 times) than the standard capacity measure to be calibrated.

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 (at least 3 times) than the standard capacity measure to be calibrated.

2.4 Measures used “to contain” or “In”

The term refers to a standard capacity measure whose capacity is equal to the volume of water that it contains, at the reference temperature, when filled to its reference graduation mark.

Strictly speaking, the volume should be determined from a dry vessel condition (“dry contained”). However, in some cases, the “contained volume” of a standard capacity measure can be further distinguished in “dry contained” and “wet contained” volume, respectively. The “dry contained” volume of a SCM refers to the volume determination of a completely empty and dry vessel while the “wet contained” volume is determined after a preliminary filling and emptying of the SCM to be calibrated under prescribed conditions of delivery and drain of the contained liquid.

2.5 Measures used “to deliver” or “Ex”

The term refers to a standard capacity measure which delivers at the given reference temperature the quantity of water that corresponds to the capacity defined by its reference graduation mark, during a predefined delivery and dripping time.

The volume delivered is always less than the “dry contained” volume, 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. On the contrary the delivered volume of a vessel is equal to the “wet contained” volume under the same delivery and drain time and the same liquid.

2.6 Dripping time

It is the time necessary to wait after the main flow ceases and starts dripping. It is also called the drainage time. This value should be stated in the calibration certificate of the standard capacity measure and should be strictly applied during calibration and use of the SCM, as well.

2.7 Valve opening and closing

Some types of discarge valves do not allow a complete discharge with the valve fully open (because the holes of the sphere and of the valve are different), this entrained water is discharged at the time of closing the valve, and the quantity discharged during the closing phase depends on the closing speed of the valve, if the valve is closed with a slow movement, the water inside will flow, but if the closing movement is fast some water will remain inside. In such cases, it is recommended to perform at least one closing and subsequent reopening of the valve during dripping time. This should be performed after more than half of the dripping time, and the applied procedure is to be reported on the calibration certificate.

2.8 Delivery time

It is the time necessary to empty completely the standard capacity measure. Delivery time is the time between the opening and closing of the drain valve including dripping time. This information should be reported in the calibration certificate and depends on the size and shape of the vessel and the size of the bottom drain valve. For convenience, it can also be indicated separately in terms of main flow drain time and dripping time.

2.9 Residual volume

Volume or quantity remaining in the measure after closing the drain valve that depends on the different liquids used and inner walls' surface properties of the construction material of the measure. It could also depend on the closing speed of the valve, see clause 2.7.

2.10 Reference standard

Is a calibrated volumetric standard with known "In" or "Ex" volume (depending on applicable method) at its reference conditions.

2.11 Standard capacity measure

Is the measuring instrument under test for which the volume at its reference conditions is going to be determined applying appropriate, "In" or "Ex" method.

3 GENERAL TECHNIQUES

3.1 Choice of type of calibration

Preferably the calibration laboratory, should calibrate the standard capacity measure so that the calibrated volume fits the way the customer uses it (either as measure "to deliver" or "to contain" in a wet or dry mode) choosing either the filling or the withdrawing method.

3.2 Standard capacity measure

The standard capacity measure (SCM) is the instrument to be calibrated.

There are three types of standard capacity measures: standard test measures, standard flasks and proving tanks. The capacity of standard test measures can vary from 1 L up to 20 L. Proving tanks are larger vessels with capacities up to several thousands of liters and are provided with drain valves at the bottom. The maximum capacity of a SCM that can be calibrated by the volumetric method is implicitly defined as the volume that can be reasonably calibrated within a typical working day or alternatively that volume over which other methods (e.g. geometrical, optical) are considered more suitable or appropriate for the calibration.

Measures may be filled from the top or from the bottom. The standard test measures and standard flasks are emptied by pouring so that the liquid flows out from only one point of the rim.

Proving tanks are always drained from the bottom through the drain valve.

Inspection and cleaning of the artefact must be performed prior to calibration of the SCM. The readability and security of the scale, the levelling mechanism and any relevant seals should be checked. 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 is performed before the start of measurement in case of measures with discharge valve and/or scales with glass gauges, to verify the tightness of the instrument. Usually this can be done when prewetting of SCM is perfomed, checking if there is a water level decrease over time. There might be only a small difference in the water level, that can be a consequence of evaporation or buble extraction.

Volume standards must allow a precise and repeatable measurement of the quantity of liquid (water). The shape of the measure must ensure that problems regarding the trapping of liquid or vapor are avoided 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 levelled 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 ensure that problems regarding the trapping of liquid or vapor are avoided 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 reference standards (RS) must be calibrated with an uncertainty at least 3 times smaller than the uncertainty of the measure being calibrated.

There are two basic types of reference standards: reference standards with graduation line (neck scale) and overflow pipettes.

3.4 Reference temperature

The value of the reference temperature to which the volume of a SCM refers to depends on the purpose of its use and should be clearly defined. A reference temperature of 20 ºC is typical for standard flasks, but alternative temperatures are specified for different applications (e.g. 15 ºC for petroleum industry [7], 4 ºC for milk industry). This information

should be specified, included in calculations, stated in the calibration certificate and preferably marked on the identification plate on the measure.

3.5 Calibration liquid

The liquid used in volumetric calibrations shall be clean water, without dirt, particles, air, contaminants or corrosive chemicals. In general potable or good quality tap water can be used as long it is free of air bubbles and is stored in a reservoir located preferably in the same room as the rest of the calibration equipment for temperature stabilisation. When using potable water direct from a pipeline (e.g. when calibrating on site), it is important to ensure it is free of entrained air. The removal of the entrained air can be facilitated by using a buffer storage tank for the water in on site calibrations and ensuring an appropriate acclimatisation period.

In special applications (like use of the SCM with liquids of viscosity much different than water's) it may be advisable to calibrate the SCM (≤ 100 L) with the actual liquid of use (or similar one) in order to account for viscosity effects (e.g. increased residual volume for the same drain time applied in water calibration).

If liquids other than water are used for the calibration, care should be taken regarding evaporation and viscosity. These liquid properties can lead to an incorrect reference volume and large measurement uncertainties. Also, the corrections for liquid expansion, the different dripping and delivery time and film formation on the inner walls (2.9) should be considered in the measurements.

3.6 Water temperature

The water temperature should be measured in both reference standard and standard capacity measure.

In large proving tanks (capacities greater than 500 L) the water temperature should be measured in at least two locations due to possible temperature gradients inside the tank [1]. For more than 2 000 L the water temperature should be measured in 3 representative locations across the liquid volume in order to estimate the mean temperature of the liquid inside the tank.

When performing calibrations in the laboratory it is recommended that the maximum water temperature variation during the tests be within ± 1 °C and be as close as possible to ambient air temperature, this intended to reduce the contribution of uncertainties due to temperature gradient and the difference between air and water temperature.

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 measure 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. Measures to limit the exposure of the calibration equipment and standard capacity measure under calibration to direct sunlight, wind and precipitation (rain) should be taken in order to keep temperature gradients within the water inside the standards as low as possible and within the above-mentioned range.

3.7 Ambient conditions

During calibration it is recommended that air temperature shall be stable. If the calibration is perfomed in the laboratory in order to obtain more accurate results and better thermal equilibrium between SCM, RS and calibration liquid the variation of air temperature should

be within ± 3 °C. Ambient humidity, temperature and pressure should be recorded. The range of the air temperature, ambient humidity and ambient pressure during calibration should be stated in the calibration certificate.

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 for at least 6 hours before calibration. This is especially important in places where large temperature variations take place during the day. During on site calibration, exposure to direct solar radiation should be avoided. Care must also be taken for wind and rain. For calculation purposes it is assumed that the instrument temperature is at the liquid temperature and uncertainty components are evaluated to reflect this assumption.

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 and powered up in advance following the manufacturer's recommendations, usually at least one hour before calibration of the SCM is enough.

3.10 Adjusting the volume of the standard capacity measure

Usually, metal standard capacity measures have a removable scale that can be adjusted. Any scale adjustment done by the laboratory during calibration should be agreed with the customer/owner. When adjustment is performed the calibration results before and after adjustment or adjustment value must be reported in the calibration certificate.

3.11 Cleaning

The standard capacity measure must be sufficiently clean to permit uniform wetting of its internal surface. When clean, the walls will be uniformly wetted, and the water will adhere to the interior surface forming a uniform film. Lack of cleanliness causes irregularities in capacity by distorting the free water surface as well. If the standard is dirty and the calibration is done without cleaning this should be stated in the calibration certificate.

The cleaning should be carried out by the customer or by the laboratory in agreement with the customer.

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 detergents which will attack, discolour or swell the material of the surface on the measure). Always follow the instructions of the manufacturer. After cleaning with the cleaning solution, if applicable, the measure should be rinsed with ethyl alcohol, then thoroughly rinsed with tap water and dried at room temperature.

It is not necessary to dry any measure to be calibrated to provide a volume “to deliver”.

3.12 Meniscus reading

Meniscus reading is critical for the volume determination in neck scale type of capacity measures and one of the most important contributions to measurement uncertainty in volumetric calibration, especially for volumes greater than 200 L. In those cases, neck diameter is usually 20 cm or more and often the water level is fluctuating due to the

unstable base rendering the definition of the position of the meniscus with respect to the graduation line difficult.

Meniscus reading has a big influence on measurement and affects consequently the repeatability of the result. The variability of meniscus settings and scale interval readings made by a single operator depend upon his/her individual expertise (Fig. 1).

Depending on how clean the standard capacity measure is, the meniscus can be curved up (convex) or down (concave). Usually with clean water and with measures with small necks the meniscus is curved downwards. For measures with wider necks, it can be almost impossible to define the meniscus clearly, so this contribution should definitely be included in uncertainty calculations.

In case of convex curving, the meniscus shall be set considering that the eyes are raised to the same plane as the graduation lines (in front and behind) and the upper edge of the graduation line is tangential to the bottom edge of meniscus (see Fig. 2).

In order to improve the reading, methods can be applied to render the meniscus bottom more visible, for example by using a black and white card placed behind the scale reading, with the black and white edge aligned with the graduation line (Fig. 3) turns the meniscus bottom black. 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 observing the meniscus at the same level of the graduation line (Fig. 2). The observation of the meniscus is again greatly facilitated when a black shading strip is placed just under the bottom of the meniscus. The black background turns the bottom of the meniscus to black rendering its position clearly visible with respect to the graduation line (Fig. 3). [2].

Figure 1. Standard test measure meniscus

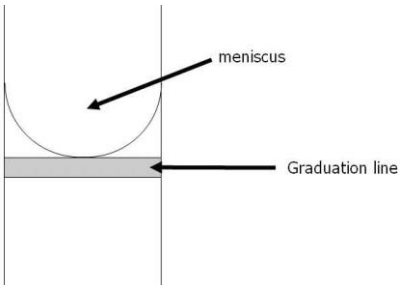

Figure 2. Meniscus setting

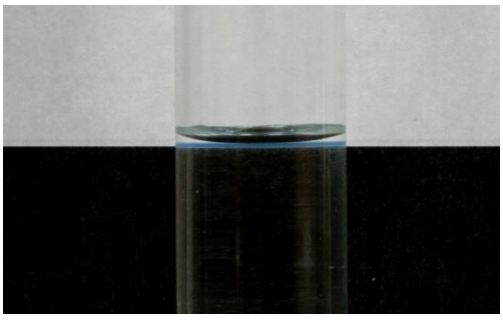

Figure 3. Meniscus reading

3.13 Coefficient of cubical thermal expansion of water

The coefficient of thermal expansion of water β in °C^{-1} can be determined using equation (1) developed according to the data presented in [8]:

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

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

t_{RS} - the temperature of water in the filled RS before pouring, in °C,
t_{SCM} - the temperature of water in the SCM after its filling, in °C.

Equation (1) can be used at normal ambient condition with typical differences in water temperature $t_{RS} - t_{SCM}$ less than 10 °C, for other conditions, or for a more accurate evaluation the formula is:

$$\beta = \frac{\frac{\rho_w(t_{RS})}{\rho_w(t_{SCM})} - 1}{t_{SCM} - t_{RS}} \quad (2)$$

where:

$\rho_w(t_{RS})$ - water density at temperature t_{RS};
$\rho_w(t_{SCM})$ - water density at temperature t_{SCM};

The water density can be determined from the Tanaka equation [9].

3.14 Coefficient of cubical thermal expansion of the material

Standard test measures are constructed of materials which should be resistant to corrosion by water and any other liquid which may be used, including cleaning liquids. Stainless steel is the most common material, other type of materials, however, may be used. The coefficient of cubical thermal expansion depends on the material that the standard capacity measure is made of. The coefficients for the most common materials are given in the Table 1 [2,10,11]:

Table 1. Coefficient of cubical thermal expansion of standard materials

Standard capacity measure material	Coefficient of cubical thermal expansion of the material ºC^{-1}
Carbon fiber	1×10^{-6}
Borosilicate glass 3,3	$9{,}9 \times 10^{-6}$
Borosilicate glass 5,0	15×10^{-6}
Soda-Lime Glass	27×10^{-6}
Steel	33×10^{-6}
Mild carbon	$33{,}5 \times 10^{-6}$
Stainless Steel grade 304	$51{,}8 \times 10^{-6}$
Stainless Steel grade 316	$47{,}7 \times 10^{-6}$
Stainless Steel 17-4 PH	$32{,}4 \times 10^{-6}$
Copper – zinc alloy (brass)	54×10^{-6}

Standard capacity measure material	Coefficient of cubical thermal expansion of the material $^{o}C^{-1}$
Aluminium	69×10^{-6}
PVC	80×10^{-6}

The material used should be documented and the coefficient of cubical thermal expansion should be given on the design, identification plate and calibration certificates.

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.

Perform an inspection and cleaning process on the standard capacity measure, note any defects such as bumps or dents, leaking valves and leaking tubes.

Level the SCM and RS when empty either by attached or built-in levels or by placing a level across the top of the open neck and check again when they are filled with water. 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 opposite sides.

If the standard capacity measure is to be calibrated to “dry contained” (In type) volume, the internal surfaces of the vessel must be completely dry.

For “wet contained” (Ex type) volume the internal surface must be wetted according to the prewetting conditions and the corresponding delivery and dripping time defined for every specific vessel. This prewetting of the SCM, beyond the establishment of the residual volume, is also a very important priming step for the thermal equilibration of the measure with the water temperature and the prevailing ambient conditions.

After the above mentioned “priming” of the equipment and 6 hours of equilibration time is guaranteed the calibration can be started.

The volumetric method of calibration may be used in two different approaches: withdrawing or filling.

4.2 Calibration using the filling method

Utilising this method, the standard capacity measure is calibrated by filling from a reference standard that has a calibration certificate with a known “Ex” volume at reference conditions. The calibration steps are the following:

a) Measure and record the ambient conditions (air temperature, humidity and barometric pressure).
b) Fill the RS with water up to a selected point of the neck scale or until it overflows. Agitate the water in the gauge tube to get a uniform meniscus in the case of a neck scale RS. Measure the water temperature and record it. In case of a neck scale RS, remove the temperature sensor if temperature is measured with a removable sensor through RS’s neck, adjust the meniscus and record the volume of the RS at the current temperature.
c) Deliver the water from RS into the levelled SCM, which has been properly prepared (by prewetting for “Ex or Wet contained” type or cleaning and drying for “Dry contained or

In type"), as described in the previous paragraphs. Keep the dripping time indicated on the RS.

d) In case of multiple fillings, fill the reference standard again in the same way. Measure the water temperature in the reference standard, adjust the meniscus and deliver the liquid into the standard capacity measure. Keep the dripping time indicated on the reference standard.
e) Measure and record the temperature(s) of the water in the SCM. Measure and record the ambient conditions (air temperature, humidity and barometric pressure). Remove the temperature sensor (the volume variability caused by the water drops that remain in the sensor are considered in the uncertainty budget as additional factors).
f) Repeat the procedure as many times as required in order to obtain an estimate of the repeatability, depending on the uncertainty required. It should be noted that with a SCM larger than 100 L, 2-3 repeats should be adequate while for SCM smaller than 100 L, 3 times minimum are recommended.

According to OIML R120, the calibration of a SCM shall be carried out such that the expanded uncertainty is within one-fifth of the maximum permissible error on pattern approval tests and one-third of the maximum permissible error on verification tests [1].
If the SCM is equipped with a correction device/scale and upon customer request, it is possible to adjust the volume to obtain $V_{\mathrm{read}} = V_t$, that is $V_{0\mathrm{SCM}} = V_N$. After the adjustment the standard must be calibrated again.

In case of adjustment of the SCM, the scale must be fixed with appropriated tools in order to avoid possible changes.

The indication error E of the SCM will be the difference between the value of the volume read on its scale V_{read} and V_t, where V_t, (equation (15)) is the volume delivered from the reference measure at the same reference temperature t (after the application of appropriate thermal corrections):

$$E = V_{\mathrm{read}} - V_t \tag{3}$$

The estimated volume of the SCM $V_{0\mathrm{SCM}}$, at the reference temperature t, at the nominal value V_N (assuming linearity of the scale) will be:

$$V_{0\mathrm{SCM}} = V_N - E \tag{4}$$

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 one or more reference standard measures. The calibration steps are the following:

a) Measure and record the ambient conditions (air temperature, humidity and barometric pressure).
b) Prime the receiving RS according to its type of use ("In" (dry or wet) or '"Ex") using the appropriate dripping time according to its calibration certificate where necessary.
c) Level and fill the SCM. Measure the water temperature, remove the temperature sensor, if the temperature is measured with a removable sensor through SCM's neck, adjust the meniscus to the reference line (V_N) and deliver the liquid into the RS. Keep the delivery or dripping time indicated on the SCM.

d) Measure and record the temperature of the water in the RS (the volume variability caused by the water drops that remain in the sensor are considered in the uncertainty budget as additional factors).
e) Record the neck scale reading of the RS V_{read}. Alternatively determine the delivered volume at the reference line (V_N) and the error (E) of the SCM by removing or adding a known quantity of water (ΔV) until the volume corresponds to the nominal volume mark (V_t) of the RS.
f) Repeat the procedure as many times as required obtaining an estimate of repeatability. It should be noted that with SCM larger than 200 L 2-3 repeats should be adequate, while for SCMs smaller than 200 L minimum 3 repetitions are recommended.

In case of adjustment of the SCM, the scale must be fixed with appropriate tools in order to avoid possible changes.

The indication error E of the SCM will be the difference between the value of the volume read in its scale $V_{\text{read}} = V_N$ and V_t, where V_t, is the volume contained or captured by the reference measure at the same reference temperature t:

$$E = V_{\text{read}} - V_t \tag{5}$$

The estimated volume of the SCM at the reference temperature t, $V_{0\text{SCM}}$, at the nominal value V_N (assuming linearity of the scale) will be:

$$V_{0\text{SCM}} = V_N - E \tag{6}$$

4.4 Multiples fillings

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 maximum of 10. The cumulative volume is calculated and the average temperature of the water of all fills is determined. In the case of multiple fills with RS of different volumes, equation (15) is not applicable.

The withdrawing method should be preferably applied in cases where the volumes of the SCM and the RS have a 1:1 relation.

4.5 Neck scale calibration

The volume corresponding to any scale mark at reference temperature can be calculated if requested by the custumer and especially in cases where the SCM is used in the frame of legal metrology for inspection and verification of e.g. fuel pumps where during the relevant test the level of the liquid transferred to the SCM is never expected to correspond to one specific mark of the scale, but on the contrary is expected to be anywhere between the frame of visual inspection of the scale.

The results of the neck scale calibration in all the marks can be plotted in a diagram as shown in Figure 4, where on the y-axis is the measured volume and, on the x-axis, the corresponding scale marks. In this diagram the x-axis data can easily be replaced by the length if the scale length between successive marks is measured. The sensitivity of the scale of the SCM is obtained by the slope of the regression line through the plotted points. The error obtained for each mark will then be applied directly during use.

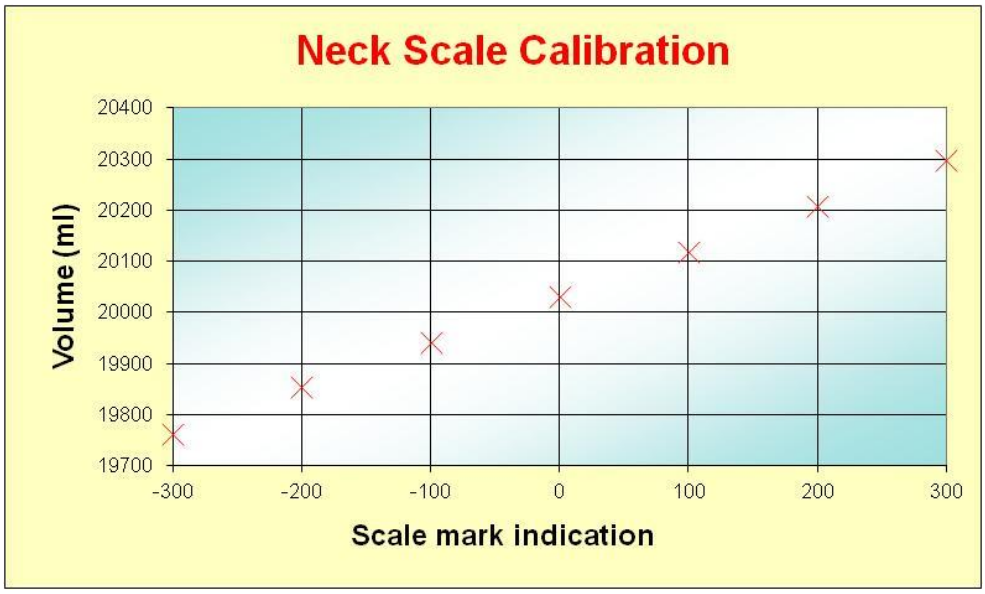

Figure 4. Neck scale calibration

Another procedure to perform a neck scale calibration is to start from the calibrated nominal volume and add liquid with a calibrated pipette or burette until the next mark its reached.

In many cases, where it can be assumed that the scale markings are linear and the neck section constant, only the measurement of the volume of the total scale is enough to evaluate the sensitivity of the scale.

The neck scale correction factor K can be calculated as:

$$K = \frac{V_{SM}}{V_{SN}} \tag{7}$$

and the associate uncertainty is given by:

$$u(K) = \frac{u(V_{SM})}{V_{SN}}, \tag{8}$$

where:

V_{SN} - nominal total neck scale volume
V_{SM} - measured neck scale volume
$u(V_{SM})$ - uncertainty of the measured volume.

The error E_R determined at the reading volume V_{read} is:

$$E_R = (V_{read} - V_N)(1 - K) + E \tag{9}$$

and

$$u(E_R) = \sqrt{u^2(V_{read})(1-K)^2 + u^2(K)(V_{read} - V_N)^2 + u^2(E)}, \tag{10}$$

where $u(V_{read})$ is the uncertainty due to the scale reading.

Depending on the measured error E related to the nominal volume V_N and the sensitivity of the scale K, the equation giving the correct volume V_R as a function of the reading V_{read} is:

$$V_R = a\,V_{read} + b. \tag{11}$$

The coefficients a and b are calculated as follows:

$$a = K \tag{12}$$

$$b = V_N(1 - K) - E \tag{13}$$

and the associated uncertainty is evaluated from:

$$u(V_R) = \sqrt{u^2(K)V_{read}^2 + u^2(V_{read})K^2 + V_N^2 u^2(K) + u^2(E)}\ . \tag{14}$$

5 DETERMINATION OF THE VOLUME

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

$$V_t = NV_0[1 - \gamma_{\mathrm{RS}}(t_{0\mathrm{RS}} - t_{\mathrm{RS}}) + \beta(t_{\mathrm{SCM}} - t_{\mathrm{RS}}) + \gamma_{\mathrm{SCM}}(t - t_{\mathrm{SCM}})] + \Delta V \tag{15}$$

V_t	-	volume of the standard capacity measure at t;
N	-	whole number ratio between nominal volumes of SCM an RS;
V_0	-	volume of the reference standard at the reference temperature $t_{0\mathrm{RS}}$;
$t_{0\mathrm{RS}}$	-	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: $(t_{\mathrm{RS}} + t_{\mathrm{SCM}})/2$;
γ_{SCM}	-	coefficient of cubical thermal expansion of the material of the SCM;
ΔV	-	quantity of water added or removed. Once filling has been completed, in order to reduce the meniscus reading uncertainty, it is preferable to adjust the level exactly to the reference scale mark, by adding or removing liquid.

The average temperature of liquid in the RS, t_{RS}, is calculated from:

$$t_{\mathrm{RS}} = \frac{1}{N}\sum_{i=1}^{N} t_{\mathrm{RS}i}\,, \tag{16}$$

where $t_{\mathrm{RS}i}$ is the temperature of the liquid in RS for each individual filling i out of N fillings.

Note: If a combination of more than one different standard is used for the calibration, equation (15) can be expanded accordingly.

The approach for volume determination described in ISO 8222 [7] can also be used as an alternative to equation (15).

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 of a volumetric calibration of standard capacity measures are the following.

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 required uncertainty.

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 into the standard capacity measure, before adjusting the volume reading (filling method) or just after its volume reading is done (withdrawing method).

In the case where more fillings are necessary to fill the SCM (using the same reference standard), the water temperature of each fill is recorded, and the average temperature value is used to determine the volume.

The thermometer used should have a resolution of at least 0,1 ºC.

6.1.3 Water temperature of the standard capacity measure

Water temperature in the standard capacity measure being calibrated must be measured after each volume reading (filling method) or before delivery, before adjusting the volume reading (withdrawing method).

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 temperature 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, the 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 thermal expansion coefficient of the material, must also be considered.

6.1.5 Water expansion coefficient

The uncertainty of the water thermal expansion coefficient needs to be taken into account.

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 or water drops remaining on the temperature sensor. 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 [4]. The method consists of the following steps:

a) Expressing, in mathematical terms, the relationship between the measurand and its input quantities;
b) Determining the expectation 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 covariances between the input quantities;
f) Calculating the expectation 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.

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

It is relevant to point out that special conditions can arise where the GUM uncertainty framework might not be the best approach to evaluate the measurement uncertainty. This is particularly relevant when there is a dominant source of uncertainty with a non-Gaussian distribution. In such cases alternative methods may provide a better approach, e.g., GUM supplement 1 [12] or a Bayesian method [13].

6.3 Procedure for calculating uncertainty in volumetric determination of volume

6.3.1 Mathematical expression of the volume V_t

$$V_t = NV_0[1 - \gamma_{\mathrm{RS}}(t_{0\mathrm{RS}} - t_{\mathrm{RS}}) + \beta(t_{\mathrm{SCM}} - t_{\mathrm{RS}}) + \gamma_{\mathrm{SCM}}(t - t_{\mathrm{SCM}})] + \\ + \Delta V + \delta V_{\mathrm{men}} + \delta V_{\mathrm{rep}} + \delta V_{\mathrm{add}} \tag{17}$$

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 (17), 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;
- Quantity of volume added or removed, ΔV;
- Meniscus reading, δV_{men};
- Measurement repeatability, δV_{rep};
- Additional factors, δV_{add}.

Note: For this uncertainty evaluation it is considered that all systematic errors in the measuring values obtained through the use of the reference equipment used for the calibration are previously corrected.

6.3.3 Standard uncertainty of each input quantity

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

6.3.3.1 Reference standard

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

$$u_{\mathrm{cal}}(V_0) = \frac{U_{\mathrm{cal}}(V_0)}{k}\,, \tag{18}$$

where:

$U_{\mathrm{cal}}(V_0)$ is the expanded measurement uncertainty of the reference standard, in volume units (the value is obtained from the last calibration certificate of the reference standard);
k is the coverage factor. If the uncertainty in the calibration certificate has been estimated for a 95 % confidence level, then $k = 2$ with 50 degrees of freedom is taken into account.

Also, reference standards may drift between calibrations. This fact adds an additional uncertainty contribution given by:

$$u_{\mathrm{drift}}(V_0) = \frac{\delta_{\mathrm{drift}}(V_0)}{\sqrt{3}}\,, \tag{19}$$

where $\delta_{\mathrm{drift}}(V_0)$ is the difference for reference values between consecutive calibrations, in L.

In addition, in case the reference standard has a scale, the uncertainty of the scale interval should also be added. This is not necessary if the uncertainty of the reading of the user is not greater than that taken into account during calibration, which has already been taken into account in the uncertainty estimation of the reference standard $U_{\mathrm{cal}}(V_0)$, see section 6.3.3.7.

The overall uncertainty for the reference standard, which is used *N* times, to measure the total volume $N\,V_0$, is given by:

$$u(N\,V_0) = N\sqrt{u_{\mathrm{cal}}^2(V_0) + u_{\mathrm{drift}}^2(V_0)}\,. \tag{20}$$

For a more accurate uncertainty evaluation, the correlations due to the reference standard need to be taken into account:

$$u(N\,V_0) = \sqrt{N\,s^2(V_0) + N^2[u_{\mathrm{B}}^2(V_0) + u_{\mathrm{drift}}^2(V_0)]}\,, \tag{21}$$

where $u_B(V_0)$ is the Type B uncertainty of the reference standard RS, which can be evaluated by

$$u_B(V_0) = \sqrt{u_{\mathrm{cal}}^2(V_0) - s^2(V_0)/n}\,,$$

where

- $u_{\mathrm{cal}}(V_0)$ (from equation (18)) is the combined standard uncertainty of the RS,
- the second term the Type A uncertainty of the RS, usually evaluated from the standard deviation of the measurements $s(V_0)$ and the number of repeated measurements *n* reported on the calibration certificate.

6.3.3.2 Water temperature of the reference standard

Equation (22) is a possible expression for this uncertainty component:

$$u(t_{\mathrm{RS}}) = \sqrt{u_{\mathrm{cal}}^2(t_{\mathrm{RS}}) + u_{\mathrm{res}}^2(t_{\mathrm{RS}}) + u_{\mathrm{drift}}^2(t_{\mathrm{RS}}) + u_{\Delta t}^2(t_{\mathrm{RS}}) + u^2(\delta ts_{\mathrm{RS}})}\,, \tag{22}$$

where:

$u_{\mathrm{cal}}(t_{\mathrm{RS}})$	-	standard uncertainty of the thermometer in the reference standard, in ºC,
$u_{\mathrm{res}}(t_{\mathrm{RS}})$	-	resolution of the used thermometer, in ºC,
$u_{\mathrm{drift}}(t_{\mathrm{RS}})$	-	estimate of the uncertainty caused by possible drift and ageing of the temperature measuring system after its calibration, in ºC,

$u_{\Delta t}(t_{\mathrm{RS}})$ - 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.

$u(\delta ts_{\mathrm{RS}})$ - estimate of the uncertainty contribution caused by the difference between air temperature t_a and water temperature t_{RS}. Following a conservative approach, it can be evaluated as [14]:

$$u(\delta ts_{RS}) = \frac{|t_{RS} - t_a|}{8\sqrt{3}} \tag{23}$$

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, $u_{\Delta t}(t_{\mathrm{RS}}) = (t_{\max} - t_{\min}) / \sqrt{12}$ 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).

6.3.3.3 Water temperature of the standard capacity measure

Equation (24) is a possible expression for this uncertainty component:

$$u(t_{\mathrm{SCM}}) = \sqrt{u_{\mathrm{cal}}^2(t_{\mathrm{SCM}}) + u_{\mathrm{res}}^2(t_{\mathrm{SCM}}) + u_{\mathrm{drift}}^2(t_{\mathrm{SCM}}) + u_{\Delta t}^2(t_{\mathrm{SCM}}) + u^2(\delta ts_{\mathrm{SCM}})}\,. \tag{24}$$

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}, then 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 $\delta t = t_{\mathrm{SCM}} - t_{\mathrm{RS}}$, equation (17) can be expressed as:

$$\begin{aligned} V_t &= NV_0[1 - \gamma_{\mathrm{RS}}(t_{0\mathrm{RS}} - t_{\mathrm{RS}}) + \beta\delta t + \gamma_{\mathrm{SCM}}(t - \delta t - t_{\mathrm{RS}})] + \\ &\quad + \Delta V + \delta V_{\mathrm{men}} + \delta V_{\mathrm{rep}} + \delta V_{\mathrm{add}} = \\ &= NV_0[1 - \gamma_{\mathrm{RS}} t_{0\mathrm{RS}} + \gamma_{\mathrm{SCM}} t + (\beta - \gamma_{\mathrm{SCM}})\ \delta t + (\gamma_{\mathrm{RS}} - \gamma_{\mathrm{SCM}})\ t_{\mathrm{RS}}] + \\ &\quad + \Delta V + \delta V_{\mathrm{men}} + \delta V_{\mathrm{rep}} + \delta V_{\mathrm{add}} \end{aligned} \tag{25}$$

The uncertainty components for t_{RS} will be the same as the ones in equation (22).

For δt the uncertainty contribution is given by:

$$u(\delta t) = \sqrt{u_{\mathrm{lin}}^2(\delta t) + 2u_{\mathrm{res}}^2(\delta t) + u_{\Delta t}^2(\delta t)}\ , \tag{26}$$

where:

$u_{\mathrm{lin}}(\delta t)$ - standard uncertainty due to the linearity and sensitivity of the thermometer between t_{RS} and t_{SCM}, in ºC,

$u_{\mathrm{res}}(\delta t)$ - 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 depend 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 coefficient of water can be determined with formula (1) with the standard uncertainty of 2×10^{-6} ºC^{-1}, or formula (2), with the standard uncertainty of 1×10^{-6} ºC^{-1}. These uncertainties are evaluated considering the uncertainties due to the Tanaka equation and to the interpolation.

6.3.3.6 Quantity of water added or removed

The uncertainty contribution for the added/removed volume is given by:

$$u(\Delta V) = \frac{U_{\mathrm{cal}}(\Delta V)}{k}\ , \tag{27}$$

where $U_{\mathrm{cal}}(\Delta V)$ is the expanded measurement uncertainty of the added or removed quantity necessary for the adjustment, determined by gravimetric or volumetric method.

6.3.3.7 Meniscus reading

The variability of meniscus settings and scale readings made by a single operator depends upon his/her individual expertise and experience. 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 artefact) with reference to the ideal position defined in [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 the uncertainty separately declared in calibration certificates, in order to allow users (who are responsible for evaluation of 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 meniscus uncertainty of both, SCM and RS [3].

6.3.3.8 Resolution of the standard capacity measure

In the case where the transferred volume is adjusted by removing or adding a known quantity of water until the volume corresponds to the nominal volume mark of the SCM the reading uncertainty is evaluated as described in EURAMET cg 19, clause 7.3.7.1 b). In the case where the transfer volume is not adjusted, the reading uncertainty is evaluated as described in EURAMET cg 19, clause 7.3.7.1 a).

6.3.3.9 Measurement repeatability

Equation (28) is a possible expression for this type A uncertainty component:

$$u(\delta V_{\mathrm{rep}}) = \frac{s(V_t)}{\sqrt{n}}\,, \tag{28}$$

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, $u(\delta V_{\mathrm{rep}})$ 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 $u(\delta V_{\mathrm{rep}})$, but the standard deviation of the whole population of possible measurements, which is evaluated by $s(V_t)$.

6.3.3.10 Additional uncertainty factors

There are some additional uncertainty factors that can 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 remaining drops on the temperature sensor or evaporation (to avoid loss by evaporation a good laboratory recommendation is to close the top of the standard capacity measure with a cap; where the calibration is performed using multiple deliveries from the reference standard the filling should be undertaken through a smaller aperture).

Following a conservative approach, these additional uncertainties can reach a (standard) uncertainty of 0,01 %.

Based on practical experience some values of additional uncertainty factors can be recommended (see Table 2).

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

According to equation (17), the sensitivity coefficients of each input quantity can be determined as follows:

6.3.4.1 Reference standard

$$\frac{\partial V_t}{\partial (NV_0)} = [1 - \gamma_{\mathrm{RS}}(t_{0\mathrm{RS}} - t_{\mathrm{RS}}) + \beta(t_{\mathrm{SCM}} - t_{\mathrm{RS}}) + \gamma_{\mathrm{SCM}}(t - t_{\mathrm{SCM}})] \approx 1 \quad (29)$$

6.3.4.2 Water temperature in the reference standard

$$\frac{\partial V_t}{\partial t_{\mathrm{RS}}} = NV_0(\gamma_{\mathrm{RS}} - \beta) \quad (30)$$

6.3.4.3 Water temperature in the standard capacity measure

$$\frac{\partial V_t}{\partial t_{\mathrm{SCM}}} = NV_0(\beta - \gamma_{\mathrm{SCM}}) \quad (31)$$

6.3.4.4 Coefficient of cubical thermal expansion of the reference standard

$$\frac{\partial V_t}{\partial \gamma_{\mathrm{RS}}} = -NV_0(t_{0\mathrm{RS}} - t_{\mathrm{RS}}) \quad (32)$$

6.3.4.5 Coefficient of cubical thermal expansion of the standard capacity measure

$$\frac{\partial V_t}{\partial \gamma_{\mathrm{SCM}}} = NV_0(t - t_{\mathrm{SCM}}) \tag{33}$$

6.3.4.6 Coefficient of cubical thermal expansion of the water

$$\frac{\partial V_t}{\partial \beta} = NV_0(t_{\mathrm{SCM}} - t_{\mathrm{RS}}) \tag{34}$$

6.3.4.7 Quantity of water added or removed

$$\frac{\partial V_t}{\partial \Delta V} = 1 \tag{35}$$

6.3.4.8 Meniscus reading

$$\frac{\partial V_t}{\partial \delta V_{\mathrm{men}}} = 1 \tag{36}$$

6.3.4.9 Measurement repeatability

$$\frac{\partial V_t}{\partial \delta V_{\mathrm{rep}}} = 1 \tag{37}$$

6.3.4.10 Additional factors

$$\frac{\partial V_t}{\partial \delta V_{\mathrm{add}}} = 1 \tag{38}$$

For equation (25), the sensitivity coefficients of each input quantity are given in Appendix.

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:

$$u^2(V_t) = \sum_i \left(\frac{\partial V_t}{\partial x_i} u(x_i) \right)^2 \tag{39}$$

Using the expressions presented in the section 6.3, the resulting combined standard uncertainty of the measurand is:

$$u(V_t) = \sqrt{\begin{aligned}&\left(\frac{\partial V_t}{\partial (N\,V_0)}u(N\,V_0)\right)^2 + \left(\frac{\partial V_t}{\partial t_{\mathrm{RS}}}u(t_{\mathrm{RS}})\right)^2 + \left(\frac{\partial V_t}{\partial t_{\mathrm{SCM}}}u(t_{\mathrm{SCM}})\right)^2 + \left(\frac{\partial V_t}{\partial \gamma_{\mathrm{RS}}}u(\gamma_{\mathrm{RS}})\right)^2 + \\ &+\left(\frac{\partial V_t}{\partial \gamma_{\mathrm{SCM}}}u(\gamma_{\mathrm{SCM}})\right)^2 + \left(\frac{\partial V_t}{\partial \beta}u(\beta)\right)^2 + \left(\frac{\partial V_t}{\partial \Delta V}u(\Delta V)\right)^2 + \\ &+u^2(\delta V_{\mathrm{men}}) + u^2\left(\delta V_{\mathrm{rep}}\right) + u^2(\delta V_{\mathrm{add}})\end{aligned}} \tag{40}$$

6.3.6 Evaluation of any existing covariances

Equation (39) and Equation (40) 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 [4]:

$$\nu_{\mathrm{eff}} = \frac{u_V^4}{\sum_{i=1}^{N}\frac{u_i^4}{\nu_i}} \tag{41}$$

where:

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 $u(V_t)$, the expanded uncertainty is:

$$U(V_t) = k \times u(V_t) \tag{42}$$

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 with 1 L resolution was calibrated at a reference temperature of 20 ºC using an overflow pipette reference standard of 500 L ($N = 4$) by the filling method. We consider the case of multiple fillings where all additions are correlated (since we are using the same reference standard). Once the SCM has been filled with the four transfers, the scale reading was slightly above the nominal value mark of the SCM, therefore, in order to obtain a better reading uncertainty, the level was adjusted by removing liquid, adjusting

the reading to exactly the reference mark of the nominal value. The volume removed was ΔV = 0,556 L. 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**	**Uncertainty equation number**
Calculated reference volume (V_t)	2000,50 L	(40)
Read volume (V_{read})	2000,00 L	
Indication error of SCM (E)	- 0,50 L	
Air temperature (t_a)	21 ºC	
Reference standard volume at 20 ºC ($N\ V_0$)	(4 × 500,26) L	(20)
Removed volume (ΔV)	0,556 L	(27)
Reference standard water temperature (t_{RS})	20,45 ºC	(22)
Reference standard expansion coefficient (γ_{RS})	51,8 × 10^{-6} ºC^{-1}	
Standard capacity measure water temperature (t_{SCM})	20,50 ºC	(24)
Water expansion coefficient (β)	2,125 × 10^{-4} ºC^{-1}	
Standard capacity measure expansion coefficient (γ_{SCM})	51,8 × 10^{-6} ºC^{-1}	
Uncertainty contribution	**Value**	**Distribution**
Meniscus reading of the SCM (δV_{men})	0,0249 L	Uniform
Measurement repeatability ($s(V_t)$)	0,05 L	Gaussian
Additional factors (δV_{add})	0,14 L	Gaussian

After analysing 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 summarised 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 $U_{\mathrm{cal}}(V_0) = 0{,}19\ \mathrm{L}$. This RS was used 4 times ($N = 4$). Considering that the reference standard used does not have any drift between consecutive calibrations the overall uncertainty for the total reference standard volume is:

$$u(N\ V_0) = 4\sqrt{\left(\frac{U_{\mathrm{cal}}(V_0)}{k}\right)^2 + u^2_{\mathrm{drift}}(V_0)} = 4\sqrt{\left(\frac{0{,}19}{2}\right)^2 + 0\ \mathrm{L}} = (4 \times 9{,}5 \times 10^{-2})\mathrm{L} = 0{,}38\ \mathrm{L}$$

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 $U_{\mathrm{cal}}(t_{\mathrm{RS}}) = 0{,}01$ ºC, using a coverage factor of $k = 2$. If we consider the drift 0,01 ºC, a resolution of 0,01 ºC and temperature gradient 0 ºC then:

$$u(t_{\mathrm{RS}}) = \sqrt{u_{\mathrm{cal}}^2(t_{\mathrm{RS}}) + u_{\mathrm{res}}^2(t_{\mathrm{RS}}) + u_{\mathrm{drift}}^2(t_{\mathrm{RS}}) + u_{\Delta t}^2(t_{\mathrm{RS}}) + u^2(\delta ts_{RS})}$$

$$= \sqrt{\left(\frac{0{,}01}{2}\right)^2 + \left(\frac{0{,}01}{2\sqrt{3}}\right)^2 + \left(\frac{0{,}01}{2\sqrt{3}}\right)^2 + 0 + \left(\frac{0{,}55}{8\sqrt{3}}\right)^2}\ ℃ = 4{,}02 \times 10^{-2}\ ℃$$

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 $U_{\mathrm{cal}}(t_{\mathrm{SCM}}) = 0{,}01$ ºC, using a coverage factor of $k = 2$. The temperature is only measured after all the filing is completed. If we consider that the drift is 0,01 ºC, the resolution is 0,01 ºC and temperature gradient is 0,03 ºC then:

$$u(t_{\mathrm{SCM}}) = \sqrt{u_{\mathrm{cal}}^2(t_{\mathrm{SCM}}) + u_{\mathrm{res}}^2(t_{\mathrm{SCM}}) + u_{\mathrm{drift}}^2(t_{\mathrm{SCM}}) + u_{\Delta t}^2(t_{\mathrm{SCM}}) + u^2(\delta ts_{SCM})}$$

$$= \sqrt{\left(\frac{0{,}01}{2}\right)^2 + \left(\frac{0{,}01}{2\sqrt{3}}\right)^2 + \left(\frac{0{,}01}{2\sqrt{3}}\right)^2 + \left(\frac{0{,}03}{2\sqrt{3}}\right)^2 + \left(\frac{0{,}50}{8\sqrt{3}}\right)^2}\ ℃ = 5{,}22 \times 10^{-2}\ ℃$$

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 $\gamma = 51{,}8 \times 10^{-6}$ ºC^{-1}, with a standard uncertainty of 5 %
The relevant standard uncertainty is therefore:

$$u(\gamma_{\mathrm{RS}}) = u(\gamma_{\mathrm{SCM}}) = (51{,}8 \times 10^{-6} \times 5\ \%) = 2{,}59 \times 10^{-6}\ ºC^{-1}$$

7.2.5 Coefficient of cubical thermal expansion of the water

The thermal expansion coefficient of the water is given by equation (1) for $t = \frac{t_{RS} + t_{SCM}}{2}$. In this case study $\beta = 2{,}125 \times 10^{-4}$ ºC^{-1}, and the standard uncertainty is:

$$u(\beta) = 2 \times 10^{-6}\ ºC^{-1}$$

7.2.6 Quantity of water added or removed

The expanded uncertainty for the calibration of the standard used to remove the extra water (using a 1 L cylinder) is $0{,}28 \times 10^{-3}$ L. Considering that the reference standard used does not have any drift between consecutive calibrations the overall uncertainty for the standard is:

$$u(\Delta V) = \frac{U_{\mathrm{cal}}(\Delta V)}{k} = \frac{2{,}8 \times 10^{-4}}{2}\ \mathrm{L} = 1{,}4 \times 10^{-4}\ \mathrm{L}$$

7.2.7 Meniscus reading of the standard capacity measure

The meniscus position of the standard capacity measure was determined taking into consideration the neck diameter and the width of the scale indication. With a rectangular distribution with a half width of 0,0249 L, the value of the standard uncertainty of the meniscus reading $u(\delta V_{\mathrm{menSCM}})$ is therefore 1,44 × 10^{-2} L:

$$u(\delta V_{\mathrm{menSCM}}) = \frac{0{,}0249}{\sqrt{3}}\mathrm{L} = 1{,}44 \times 10^{-2}\ \mathrm{L}$$

7.2.8 Measurement repeatability

Following equation (28), the type A uncertainty component can be determined by:

$$u(\delta V_{\mathrm{rep}}) = \frac{0{,}05}{\sqrt{3}}\mathrm{L} = 2{,}89 \times 10^{-2}\ \mathrm{L}$$

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

7.2.9 Additional uncertainty factors

These additional uncertainties $u(\delta V_{\mathrm{add}})$ 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

$$\frac{\partial V_t}{\partial (N\,V_{0})} = N[1 - \gamma_{\mathrm{RS}}(t_{0\mathrm{RS}} - t_{\mathrm{RS}}) + \beta(t_{\mathrm{SCM}} - t_{\mathrm{RS}}) + \gamma_{\mathrm{SCM}}(t - t_{\mathrm{SCM}})] = 1$$

7.3.2 Water temperature in the reference standard

$$\frac{\partial V_t}{\partial t_{\mathrm{RS}}} = NV_0(\gamma_{\mathrm{RS}} - \beta) = -3{,}22 \times 10^{-1}\ \mathrm{L/°C}$$

7.3.3 Water temperature in the standard capacity measure

$$\frac{\partial V_t}{\partial t_{\mathrm{SCM}}} = NV_0(\beta - \gamma_{\mathrm{SCM}}) = 3{,}22 \times 10^{-1}\ \mathrm{L/°C}$$

7.3.4 Coefficient of cubical thermal expansion of the reference standard

$$\frac{\partial V_t}{\partial \gamma_{\mathrm{RS}}} = -NV_0(t_{0\mathrm{RS}} - t_{\mathrm{RS}}) = 9 \times 10^{2}\ \mathrm{L°C}$$

7.3.5 Coefficient of cubical thermal expansion of the standard capacity measure

$$\frac{\partial V_t}{\partial \gamma_{\mathrm{SCM}}} = NV_0(t_{0\mathrm{SCM}} - t_{\mathrm{SCM}}) = -1 \times 10^{3}\ \mathrm{L°C}$$

7.3.6 Coefficient of cubical thermal expansion of the water

$$\frac{\partial V_t}{\partial \beta} = NV_0(t_{\mathrm{SCM}} - t_{\mathrm{RS}}) = 1 \times 10^2 \text{ L°C}$$

7.3.7 Quantity of water added or removed

$$\frac{\partial V_t}{\partial \Delta V} = 1$$

7.3.8 Meniscus reading

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

7.3.9 Measurement repeatability

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

7.3.10 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 (39). The individual input quantity values are collected and substituted into this expression to obtain:

$$u(V_t) = \begin{array}{l} \sqrt{\left(\frac{\partial V_t}{\partial (N\,V_0)} u(N\,V_0)\right)^2 + \left(\frac{\partial V_t}{\partial t_{\mathrm{RS}}} u(t_{\mathrm{RS}})\right)^2 + \left(\frac{\partial V_t}{\partial t_{\mathrm{SCM}}} u(t_{\mathrm{SCM}})\right)^2 + \left(\frac{\partial V_t}{\partial \gamma_{\mathrm{RS}}} u(\gamma_{\mathrm{RS}})\right)^2 +} \\ + \left(\frac{\partial V_t}{\partial \gamma_{\mathrm{SCM}}} u(\gamma_{\mathrm{SCM}})\right)^2 + \left(\frac{\partial V_t}{\partial \beta} u(\beta)\right)^2 + \left(\frac{\partial V_t}{\partial \Delta V} u(\Delta V)\right)^2 + \\ + u^2(\delta V_{\mathrm{men}}) + u^2(\delta V_{\mathrm{rep}}) + u^2(\delta V_{\mathrm{add}}) \end{array}$$
$$= 41 \times 10^{-2} \text{ L}$$

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 is necessary to estimate the effective degrees of freedom, ν_{eff}, using the Welch-Satterthwaite formula:

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

This corresponds to the coverage factor of $k = 2$ and the coverage probability of approximately 95 %.

7.7 Expanded uncertainty

The expanded uncertainty is calculated as follows:

$U(V_t) = k \times u(V_t) = 2 \times 0{,}41\ \text{L} = 0{,}82\ \text{L}$

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

Table 4. Uncertainty budget

Standard uncertainty component $u(x_i)$	**Source of uncertainty**	**Value of standard uncertainty** $u(x_i)$	$\boldsymbol{c_i \equiv \frac{\partial V_t}{\partial x_i}}$	$u_i(V_0) \equiv \lvert c_i \rvert u(x_i)$ (L)	ν_{eff}
$u(N\ V_0)$	Volume of the RS	3,80 × 10^{-1} L	1	3,80 × 10^{-1}	50
$u(t_{\text{RS}})$	Water temperature of RS	4,02 × 10^{-2} ºC	-3,22 × 10^{-1} L/°C	1,29 × 10^{-2}	∞
$u(t_{\text{SCM}})$	Water temperature of SCM	5,22 × 10^{-2} ºC	3,22 × 10^{-1} L/°C	1,68 × 10^{-2}	∞
$u(\gamma_{\text{RS}})$	Coefficient of cubical thermal expansion of the RS material	2,59 × 10^{-6} ºC^{-1}	9 × 10^{2} L·°C	2,33 × 10^{-3}	∞
$u(\gamma_{\text{SCM}})$	Coefficient of cubical thermal expansion of the SCM material	2,59 × 10^{-6} ºC^{-1}	-1 × 10^{3} L·°C	2,59 × 10^{-3}	∞
$u(\beta)$	Coefficient of cubical thermal expansion of the water	2 × 10^{-6} ºC^{-1}	1 × 10^{2} L·°C	2,00 × 10^{-4}	∞
$u(\Delta V)$	Quantity of water added or removed	1,4 × 10^{-4} L	1	1,40 × 10^{-4}	50
$u(\delta V_{\text{menSCM}})$	Meniscus reading of the SCM	1,44 × 10^{-2} L	1	1,44 × 10^{-2}	∞
$u(\delta V_{\text{rep}})$	Measurement Repeatability	2,89 × 10^{-2} L	1	2,89 × 10^{-2}	2
$u(\delta V_{add})$	Additional factors	1,4 × 10^{-1} L	1	1,40 × 10^{-1}	∞
$u_c(V_{20}) = 4{,}1 \times 10^{-1}\ \text{L}$ $\nu_{eff}(V_{20}) = 65,\ k = 2$ $U(V_t) = 8{,}2 \times 10^{-1}\ \text{L}$					

The calibration result shows an error E= -0,5 L (Table 3), if required, according to the customer, an adjustment of the standard could be carried out to reduce this error. After the adjustment, the calibration has to be repeated. The calibration results, in terms of error or volume of the SCM at

the marks/volume calibrated and the respective uncertainty have to be shown in the calibration certificate.

Assuming that the volume has been adjusted, and the result of the new calibration has a zero error and all uncertainties are the same as the initial calibration, the results will be shown as in Table 5.

Table 5. Example of a calibration cetificate results at nominal volume

Serial Number	Nominal volume (L)	Volume before adjustment (L)	Volume after adjustment (L)	Expanded uncertainty (L)	*k*	ν_{eff}	Standard uncertainty of the reading (L)	Measurement repeatability (L)	Number of repeated measurements
XXX	2000	2000,50	2000,00	0,82	2	65	0,014	0,050	3

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9 APPENDIX. Sensitivity coefficient of each input quantity for equation (25)

According to equation (25), the sensitivity coefficients of each input quantity can be determined as follows:

Reference standard

$$\frac{\partial V_t}{\partial (NV_0)} = 1 \tag{43}$$

Water temperature in the reference standard

$$\frac{\partial V_t}{\partial t_{\mathrm{RS}}} = NV_0\left(\gamma_{\mathrm{RS}} - \gamma_{\mathrm{SCM}}\right) \tag{44}$$

Water temperature in the standard capacity measure

$$\frac{\partial V_t}{\partial \delta\, t} = NV_0\left(\beta - \gamma_{\mathrm{SCM}}\right) \tag{45}$$

Coefficient of cubical thermal expansion of the reference standard

$$\frac{\partial V_t}{\partial \gamma_{\mathrm{RS}}} = -NV_0\left(t_{0\mathrm{RS}} - t_{\mathrm{RS}}\right) \tag{46}$$

Coefficient of cubical thermal expansion of the standard capacity measure

$$\frac{\partial V_t}{\partial \gamma_{\mathrm{SCM}}} = NV_0\left(t - t_{\mathrm{RS}}\right) \tag{47}$$

Coefficient of cubical thermal expansion of the water

$$\frac{\partial V_t}{\partial \beta} = NV_0\delta\, t \tag{48}$$

Quantity of water added or removed

$$\frac{\partial V_t}{\partial \Delta V} = 1 \tag{49}$$

Meniscus reading

$$\frac{\partial V_t}{\partial \delta V_{\mathrm{men}}} = 1 \tag{50}$$

Measurement repeatability

$$\frac{\partial V_t}{\partial \delta V_{\mathrm{rep}}} = 1 \tag{51}$$

Additional factors

$$\frac{\partial V_t}{\partial \delta V_{\mathrm{add}}} = 1 \tag{52}$$

EURAMET e.V.
Bundesallee 100
38116 Braunschweig
Germany

Phone: +49 531 592 1960
Fax: +49 531 592 1969
E-mail: secretariat@euramet.org