INTERNATIONAL STANDARD

**ISO
8222**

Petroleum measurement systems — Calibration — Volumetric measures, proving tanks and field measures (including formulae for properties of liquids and materials)

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Foreword

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

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

This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*, Subcommittee SC 2, *Measurement of petroleum and related products*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 19, *Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 8222:2002), which has been technically revised. The main changes to the previous edition are as follows:

— revision of the title and scope to allow the document to cover the design, calibration and use of a wide range of volumetric measures comprising proving tanks, test measures, field and standard measures;

— provision of revised, updated and extended formulae to allow calculation of temperature correction including the addition of formulae for saline water, other liquids and material properties.

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

Introduction

Volumetric, or capacity, measures are used to provide an accurate measure of volume, thereby providing a calibration reference for other volume-measuring devices, such as pipe displacement provers or flowmeters.

Volume measures are categorized in terms of capacity, test measures being below 20 l. Measures above 20 l are categorised as prover tanks. Standard measures are designed to comply with regulatory guidance and hence have specified volumes. Other measures have non-standard volumes specifically designed to suit an application, for example measures to accompany a small volume prover.

Volumetric measures can be used to calibrate flowmeters, both duty and reference meters. They can also be used to calibrate secondary volume measures, displacement (pipe) provers and storage tanks.

Annex A provides the recommended formulae used in the calibration and use of volumetric measures and for other volumetric measurements. This includes pure and saline water properties, the properties of hydrocarbon liquids and the materials of construction of volumetric measuring devices.

Petroleum measurement systems — Calibration — Volumetric measures, proving tanks and field measures (including formulae for properties of liquids and materials)

WARNING — The use of this document could involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices.

1   Scope

This document describes the design, use and calibration of volumetric measures (capacity measures) which are intended for use in fixed locations in a laboratory or in the field. This document gives guidance on both standard and non-standard measures. It also covers portable and mobile measures. This document is applicable to the petroleum industry; however, it may be applied more widely to other applications.

This document excludes measures for cryogenic liquids and pressurized measures as used for liquid petroleum gas (LPG) and liquefied natural gas (LNG).

Volumetric measures are classified as test measures or prover tanks depending on capacity and design.

Measures described in this document are primarily designed, calibrated and used to measure volumes from a measure which is wetted and drained for a specified time before use and designated to deliver. Many of the provisions, however, apply equally to measures which are used to measure a volume using a clean and dry measure and designated to contain.

Guidance is given regarding commonly expected uncertainties and calibration specifications.

The document also provides, in Annex A, reference formulae describing the properties of water and other fluids and materials used in volumetric measurement more generally.

2   Normative references

There are no normative references in this document.

3   Terms, definitions, symbols and units

3.1   Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

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

3.1.1
accuracy

closeness of the agreement between a measured quantity value and a true quantity value of a measurand

Note 1 to entry: The concept 'measurement accuracy' is not a quantity and should not be given a numerical value. The quantitative expression of accuracy should be in terms of uncertainty. "Good accuracy" or "more accurate" implies small measurement error. Any given numerical value should be taken as indicative of this.

[SOURCE: VIM:2012, 2.13]

3.1.2
adjustment

set of operations carried out on a measuring system so that it provides prescribed indications corresponding to given values of a quantity to be measured

Note 1 to entry: Adjustment should not be confused with calibration, which is a prerequisite for adjustment.

Note 2 to entry: After adjustment, a recalibration is usually required.

[SOURCE: VIM:2012, 3.11]

3.1.3
automatic pipette

overflow pipette
high precision measure, where the volume withdrawn is defined by a top overflow weir rather than a gauge scale

3.1.4
brim measure

field measure where the volume is defined by an overflow from the top edge of the neck

3.1.5
calibration

operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and in a second step uses this information to establish a relation for obtaining a measurement result from an indication

Note 1 to entry: A simplified definition is 'set of operations that establish, under specified conditions, the relationship between quantities indicated by an instrument and the corresponding values realized by standards'.

Note 2 to entry: Calibration should not be confused with adjustment of a measuring system.

Note 3 to entry: The word "proving" is used in the oil industry and has the same meaning but can include a check of the results against specified acceptance criteria.

[SOURCE: VIM:2012, 2.39]

3.1.6
calibrated volume

volume of a measure between a top and bottom datum as determined by calibration and expressed at a standard temperature

3.1.7
clingage

film of liquid that adheres to the inside surface of a volumetric measure after it has been emptied, resulting in a residual volume

3.1.8
correction factor
numerical factor by which the uncorrected result of a measurement at the measured conditions is multiplied

Note 1 to entry: Correction factors to standard conditions are used to convert a volume at observed conditions to the volume at another (standard) condition.

3.1.9
plunger
displacement plunger
device consisting of a piston which is used for adjusting the volume of a volumetric measure

3.1.10
drain time
total time taken to empty the measure or tank to leave a consistent residual volume

Note 1 to entry: Drain time commences when the drain valve is opened and ceases when closed after a defined time or condition. Drain time may be divided into two parts: first drain time and final drain time.

Note 2 to entry: A dry measure can be employed where the product evaporates quickly, for example petrol. These measures will not have a drain time and the means to ensure they are dry will be specified in documents regulating their use.

3.1.11
final drain time
time which follows the cessation of the first drain time and finishes at a defined time, or condition, such as rate of dripping

3.1.12
first drain time
time to drain the majority of the fluid from the measure

Note 1 to entry: The first drainage flow commences when the drain valve is opened and ends at a defined level, time or when flow breaks into a defined trickle or drip rate. This is prior to the start of the final drain time.

3.1.13
dry measure
contents measure which is calibrated and used with the internal surface completely dry (i.e. no clingage or residual volume)

Note 1 to entry: The volume of a dry measure may be marked and designated to contain.

3.1.14
error
measured quantity value minus a reference quantity value

Note 1 to entry: Note1 to entry: Relative error is error divided by a reference value. This can be expressed as a percentage.

[SOURCE: VIM:2012, 2.16]

3.1.15
field measure
measure designed to be used in the regular calibration of other devices

Note 1 to entry: While most field measures are portable, some can be in a fixed location.

3.1.16
gauge glass
clear tube in parallel with, or a window set into, the neck of a measure to show the level of liquid in the neck

Note 1 to entry: There is an associated scale to indicate the measured volume.

3.1.17
nominal volume
design volume of a volumetric measure

3.1.18
primary measure
reference measure calibrated gravimetrically

Note 1 to entry: Primary measures should be calibrated by a national measurement institute (NMI) or a competent laboratory meeting ISO/IEC 17025.

3.1.19
proving
calibration with comparison to defined acceptance criteria

Note 1 to entry: The term "proving" is used in the oil industry and is similar to verification.

Note 2 to entry: Proving is a calibration, sometimes of limited measurement range, according to methods defined by standards, regulations or procedures, providing a determination of the errors of a device and showing (proving) it performs to defined acceptance criteria.

3.1.20
proving tank
volumetric measure which generally has capacity greater than 20 l and has a bottom drain

Note 1 to entry: A prover tank can be free-standing or mounted on a truck or trailer.

3.1.21
pour
individual quantities of liquid poured from, or received into, a volumetric measure

3.1.22
range
range of values
difference between the maximum and minimum values of a set of values

Note 1 to entry: This can be expressed as a half range (±) number. Relative range is normally expressed as a percentage of a specified value, for example mean, minimum or other calculated value.

3.1.23
reference conditions
operating conditions prescribed for evaluating the performance of the measure

Note 1 to entry: This is the range of ambient and fluid conditions with which the measure is evaluated, verified or operated.

3.1.24
reference measure
volumetric measure calibrated, used and maintained to provide traceability to other volume measures and devices, including pipe provers and reference flowmeters

Note 1 to entry: A reference measure can be calibrated gravimetrically (primary measure) or volumetrically by means of a primary measure which itself has been calibrated gravimetrically.

3.1.25
repeatability
measurement precision
closeness of agreement between indications or measured quantity values obtained by replicate measurements under specified conditions

Note 1 to entry: Specified conditions normally implies the same reference, same conditions, same operators and procedures and that the data are obtained sequentially over a short period of time.

Note 2 to entry: Repeatability can be expressed as the range (difference between the maximum and minimum) values of error or K-factor. Alternatively, repeatability can be expressed as a function of the standard deviation of the values.

Note 3 to entry: Dividing repeatability by the mean gives the relative value which can be expressed as a percentage. It is noted some standards suggest dividing by the minimum value.

[SOURCE: VIM:2012, 2.15]

3.1.26
residual volume
volume or quantity remaining in the measure after draining for the defined drain time

3.1.27
resolution
quantitative expression of the ability of an indicating device to distinguish meaningfully between closely adjacent values of the quantity indicated

3.1.28
scale datum
fixed reference point or mark, established at manufacture or initial calibration, from which subsequent adjustments to the scale can be referred

Note 1 to entry: This may be an engraved mark on the neck, or another defined fixed point such as a support bracket. The location should be referenced on calibration certificates.

3.1.29
secondary measure
volumetric measure which is calibrated by a primary measure

3.1.30
standard condition
base condition
condition of temperature and pressure to which measurements of volume or density are referred to standardize the quantity

Note 1 to entry: These are the specified values of the conditions to which the measured quantity is converted.

Note 2 to entry: For the petroleum industry, these are usually 15 °C, 20 °C or 60 °F and 101,325 kPa.

Note 3 to entry: Standard conditions can refer to the liquid or the volume of the measure. These may be different.

Note 4 to entry: Quantities of volume expressed at standard conditions may be indicated by prefixing the volume unit by "S", for example 4 Sm^3 or 700 kg/Sm^3. This abbreviation is used in place of the unit m^3 (standard conditions) where there is limited space and there is no risk of confusion regarding the unit.

Note 5 to entry: Standard conditions should not be confused with the reference (operating) conditions prescribed for evaluating the measure.

3.1.31
standard measure
volumetric measure which is designed to meet the requirements of regulatory standards

Note 1 to entry: Examples of regulatory standards are OIML R 120, Measurement Instruments Directive (MID) and NIST 105-3.

3.1.32
standard volume
base volume
volume expressed as being at standard conditions

3.1.33
strike measure
brim measure where the volume is defined from the top edge of the neck which has been designed to be struck by sliding a ground glass disk over it to leave a consistent volume within the measure

3.1.34
test measure
hand portable volumetric measure up to 20 l capacity

Note 1 to entry: A test measure can be inverted to drain or be fitted with a bottom drain.

Note 2 to entry: A test measure can also be non-portable, in a fixed frame or on a vehicle. It may in some cases be classified as a prover tank.

3.1.35
thermowell
metal pocket which protrudes through, or is attached to, the wall of a pipe or volumetric measure to hold a temperature-measuring device

3.1.36
volume to contain
standard volume of liquid a measure contains with respect to its reference line or datum when filled from a clean dry condition

3.1.37
volume to deliver
standard volume of liquid which can be withdrawn from or filled into a pre-wetted measure with respect to its reference line or datum and following specified drainage times and procedures

Note 1 to entry: A wet condition is obtained by filling the measure and draining it for the specified drain time and procedure.

Note 2 to entry: The volume to deliver is always less than the volume to contain due to the volume of residual liquid left on the walls of the measure after the specified drain time.

Note 3 to entry: A measure marked with a volume to deliver can be used either to withdraw or to fill volumes as long as the wetting and drainage procedures are followed.

3.1.38
fill
receive
In
technique for using or calibrating a volumetric measure by filling from top or bottom with liquid from the device under test or the reference

Note 1 to entry: The reference may be volumetric or gravimetric.

Note 2 to entry: 'In' is the term adopted by EURAMET guide cg21[2].

3.1.39
withdraw
water draw
Ex
technique for using or calibrating a volumetric measure by withdrawing liquid from the measure into the device under test or the reference

Note 1 to entry: The reference may be volumetric or gravimetric.

Note 2 to entry: 'Water draw' is usually applied to the calibration of pipe provers.

Note 3 to entry: 'Ex' is the term adopted by EURAMET guide cg21[2].

3.1.40
traceability
metrological traceability
property of a measuring result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty

[SOURCE: VIM:2012, 2.41]

3.1.41
transfer point
point or location in a fluid transfer where the quantity and accountability of the fluid passes from one measurement system to another

3.1.42
uncertainty
non-negative parameter characterizing the dispersion of the quantity values attributed to a measurand based on the information used

[SOURCE: VIM:2012, 2.26]

Note 1 to entry: The uncertainty is normally expressed as a half width range along with the probability distribution with that range. It can be expressed as a value or as a percentage of the perceived true value.

3.1.43
volumetric measure
measure used to provide an accurate measure of a volume, hence providing a calibration reference for other volume-measuring devices, such as pipe displacement provers or flowmeters

3.1.44
water pour
technique for calibrating a measure by decanting liquid from a reference measure or a gravimetric system to a device under test

3.1.45
wet measure
volumetric measure which has been wetted and drained before use in accordance with defined drain times and procedures given in the calibration certificate and specification

3.1.46
wetted
portion of the internal surface of a volumetric measure which has been in contact with the liquid during use

3.1.47
weir
device, usually a horizontal edge, where a consistent liquid level is established to provide a datum

3.2 Symbols and units

NOTE 1   The preferred unit for kinematic viscosity is metres squared per second (m^2/s) or millimetres squared per second (mm^2/s). The practical unit used in this document is the industry-recognized unit centistoke (cSt); 1 cSt = 1 mm^2/s.

NOTE 2   The preferred unit for a volume expressed at a standard condition is m^3 (standard condition). In practice this is conventionally abbreviated to Sm^3 where there is limited space and there would be no confusion of units used.

Symbol	Quantity	Unit
C_{tl}	volume correction factor for thermal expansion of liquid from measured temperature to the standard temperature	
C_{dtl}	volume correction factor for thermal expansion of liquid from reference device (measure) temperature to the temperature at the device under test	
C_{tlr}	volume correction factor for thermal expansion of liquid from reference device (measure) temperature to the standard temperature	
C_{tlt}	volume correction factor for thermal expansion of liquid from test device temperature to the standard temperature	
C_{pl}	volume correction factor for liquid compressibility from measured pressure to standard pressure	
C_{plt}	volume correction factor for liquid compressibility from pressure to standard pressure from a device under test	
C_{ts}	volume correction factor for the thermal expansion of the device (material) to volume at standard temperature	
C_{tsr}	volume correction factor for the thermal expansion of the reference measure material to the volume at standard temperature	
C_{tst}	volume correction factor for the thermal expansion of the device under test material to the volume at standard temperature	
C_{ps}	volume correction factor for the pressure expansion of the device under test to the volume at standard pressure	
F	meter factor of a flowmeter at operating temperature and pressure	
M	mass of water collected in weigh container	kg
t_r	temperature of the liquid in the reference device	°C
t_t	temperature of the liquid in the device under test	°C
t_{sr}	standard temperature for the reference measure	°C
t_{st}	standard temperature of the device under test	°C
V_m	indicated volume from a flowmeter at actual conditions	m^3 or l
V_r	volume measured by a reference, measure or flowmeter, at actual conditions of pressure and temperature	m^3 or l
V_{rs}	calibrated volume of a measure corrected for scale indication, i.e. the indicated volume, at standard conditions, from a reference measure, corrected for known calibrations errors	m^3 or l (standard conditions)
V_{ts}	volume of device under test at standard conditions	m^3 or l (standard conditions)
V_t	volume transferred to or from a reference to the device under test	m^3 or l
W_a	weight of liquid collected in the weigh container during gravimetric calibration	kg
α_r	linear expansion coefficient of the reference device	°C^{-1}
α_t	linear expansion coefficient of the device under test	°C^{-1}
ρ_a	density of air during weighing	kg/m^3
ρ_w	nominal density of weights used to calibrate weighing machines (normally defined as 8 000 kg/m^3)	kg/m^3

Symbol	Quantity	Unit
ρ_r	density of liquid in reference device at t_r	kg/m^3
ρ_t	density of liquid in device under test at t_t	kg/m^3

4 Traceability

All the volumetric measures described have a unique hierarchy in the traceability chain. All are traceable to standards of mass combined with the derived density of pure water. There is a hierarchy for traceability which is followed to provide the uncertainty required by the final application.

A primary measure would be calibrated by weighing the water withdrawn to a tank on a weigh scale or to directly weigh the measure and determine the weight of liquid filled or withdrawn. The weigh scale is calibrated with mass standards of the required class[3] and uncertainty. The mass and the density of the water are then combined to give the volume.

A primary measure can be used to determine the volume of a secondary measure. This can require multiple measurements fills. A secondary measure can be used to determine the calibration factor of a flowmeter. This can be a reference flowmeter used to measure volume in a pipe prover or large volume measure or to calibrate other meters.

Figure 1 shows schematically a primary measure being used to determine the volume of a small volume prover, which in turn is used to calibrate a reference flowmeter used to determine the volume of a large pipe prover.

Primary and reference measures are calibrated using pure water or clean drinking water. The water density is determined from the recommended formulae relating density to temperature and corrected where required through a measurement of relative density. The water density can also be experimentally determined by the use of a calibrated densitometer.

Some secondary measures, particularly large fixed measures, may need to be calibrated with impure water, seawater or a hydrocarbon product.

Applicable liquid property formulae are given in Annex A.

The uncertainty for volume determination should be specified according to the application. Indicative values of uncertainty applicable to different volumetric measures are given in Annex E.
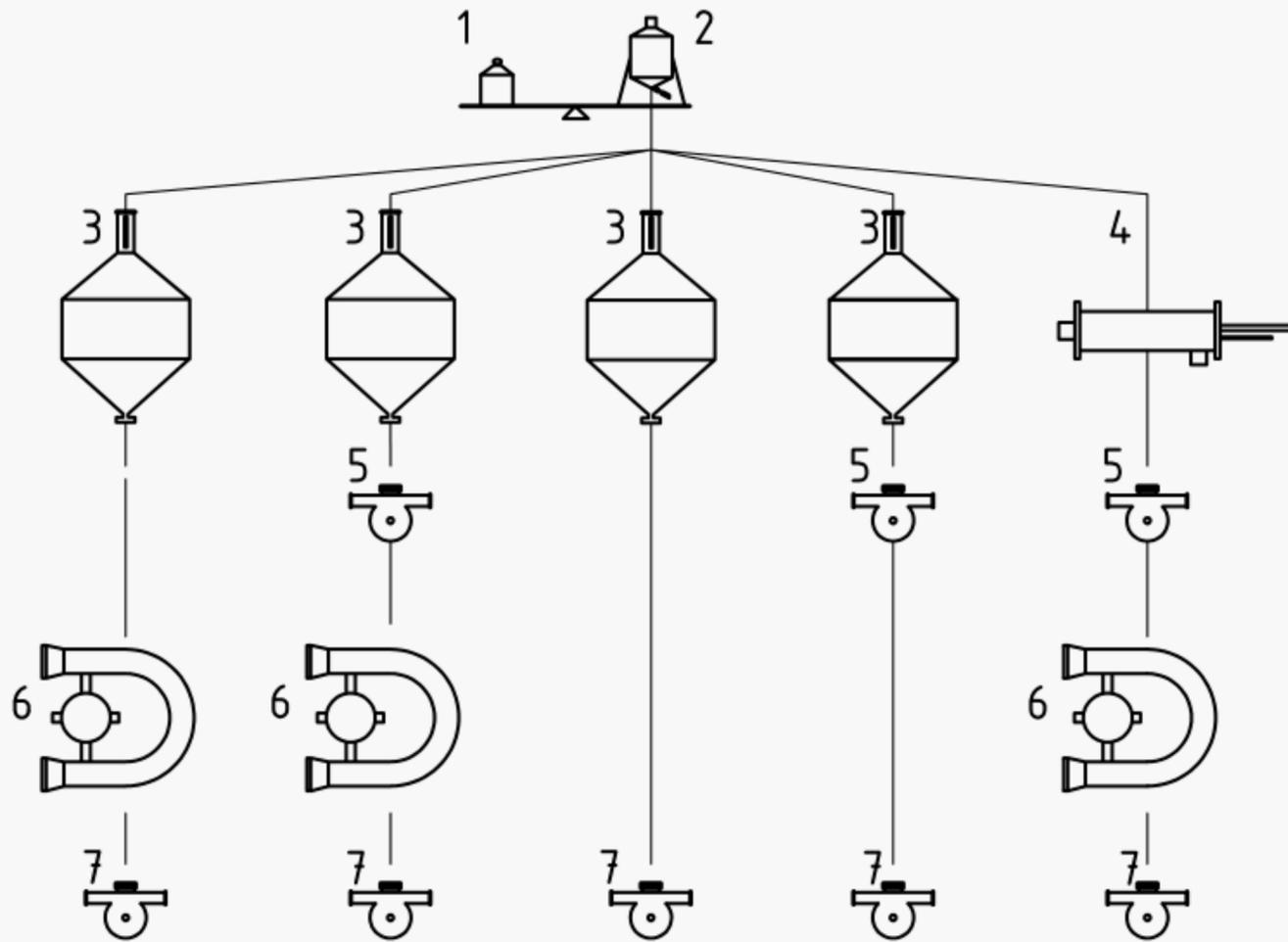
Key

1   mass standard

2   primary reference measure

3   primary measure

4   small volume piston prover

5   reference flowmeter

6   pipe prover

7   secondary measure

Figure 1 — Traceability: hierarchy of volumetric measures

5 General design characteristics of volumetric measures

5.1 General design

Volumetric measures are containers of defined internal volume. The nominal volume is derived by design and manufacture with the calibrated volume subsequently determined by calibration. The volume is defined between a top and bottom datum. The top datum can be a reference line on a scale or an overflow weir. The bottom datum can be an overflow weir, the internal plane of a drain valve (when closed) or a scale reading in a bottom sump or neck.

The volume is expressed as being the volume of liquid in the measure when it is at a defined standard condition. Nominal and calibrated volume(s) may be given for one or different standard conditions.

The volume to deliver is the standard volume of a measure which can be withdrawn from a full measure with a defined drain time. It is also the volume which can be filled into a measure which has been pre-wetted by filling then draining for the defined drain time.

The volume to contain is the standard volume of a clean dry measure. The volume to contain is always larger than the volume to deliver due to residual liquid remaining on the walls of a wet measure.

As nominal and calibrated volumes can have different conditions, both should always be clearly identified as being to contain or to deliver and the standard temperature referenced.

Volumetric measures are classified into two categories separated by what is considered to be a safe weight for manual handling. This division follows guidance given by OIML-R120[4], NIST-105-3[5] and API-MPMS chapters 4.4[6] and 4.7[7].

1) Test measures have volumes between 5 l and 20 l. However, smaller measures including 3,8 l (1 US gallon) and measures down to 1 l, manufactured in stainless steel or carbon fibre, are also included in the classification. Test measures produced following the design, specifications and volumes defined in regulatory standards are classified as standard measures. Test measures can also be referred to as field measures when designed to be portable.

2) Proving tanks are measures with larger volumes. Proving tanks can have volumes above 20 l and extend to tanks up to 20 m^3 or above.

Volumetric measures in glass are a category of cylinders or flasks with volumes less than 10 l. These are not covered by this document but may be used as additional volume measures for use in the calibration process. Standard sizes for glass measures are shown in Annex C. Specifications for such measures are given in ISO 4788.

Measures can be top filled or bottom filled.

Drainage of test measures can be by inverting the measure and pouring out the contents or by draining through a bottom connection and valve. Proving tanks are drained from the bottom.

Volumetric measures are constructed of materials which should be resistant to corrosion by water and any other fluid which may be used, including cleaning fluids. Stainless steel is the most common material; however, other metals can be used, particularly in older measures. Carbon steel can be utilized for large secondary measures if internally protected from corrosion through coating with epoxy resin, phenolic or glass. The use of unprotected carbon steel should be avoided as it is subject to corrosion and deposition even if hydrocarbons are the only fluid to be used. A low or well-defined coefficient of expansion of the material is chosen, hence the use of materials such as aluminium and plastic is not normally acceptable (carbon fibre composite being the exception as it has a very low coefficient of expansion).

The material used needs to be documented and the coefficient of expansion given in the design documentation and calibration certificates. This should also be engraved on the marking plate where possible. Suggested default coefficients of expansion of materials are given in Annex A.

Construction and design are such that there is minimal distortion of the measure when filled or emptied. This is achieved through adequate thickness of the materials. Strengthening bands and shaped end closures provide adequate stiffness to the measure. Measures are exclusively cylindrical in shape.

The design and construction should ensure all liquid drains from the measure when it is emptied and all air is expelled when it is filled. The cylindrical section should be vertical or at a slope, with ends constructed with a domed or coned shape. Slopes on cones and cylinders should be approximately 30° to the horizontal. Standard proving tanks and test measures have specified slopes for top and bottom cones.

Internal welds and joints should be smooth and constructed to avoid retention of fluid or air. The surface finish should be smooth to minimize liquid clingage and contamination through deposition.

Any inspection flanges or joints between sections of the measure should be constructed with metal-to-metal face joints sealed with "O" rings or non-compressible gaskets to ensure no change in volume in use, or minimal change if disturbed. Similarly, any insertions into the measure such as thermowells, connections to sight tubes, windows and valves should be of welded construction or metal-to-metal faced joints and angled to avoid liquid or air entrapment.

There should be a top vertical neck section with a diameter designed to provide suitable resolution of volume measurement. In proving tanks there can also be a bottom neck.

All measures should have means of indicating the volume through the use of a sight glass or window with a scale, or alternatively a weir, associated with the top neck. This provides the top datum to indicate the volume. A bottom datum is established at a fixed location which can be the plane of the drain valve, a weir or a level in a sight glass or window on a bottom neck.

Any valves used with a volumetric measure, for filling or draining, should be demonstrably bubble-tight on low differential pressure tests. Depending on the duty, valves used for filling should also be leak-tight at the appropriate pressure. A method for checking valve leakage in the system, usually by means of visual inspection, is required.

One practical leak-testing method is to first have the measure in a thermally stable environment. It is then filled and the level recorded, with all surfaces under the measure dried. It is then left undisturbed for a period of 1 h or more before rechecking the level has remained constant and the surrounding area shows no signs of leakage.

Valves should be sized to suit the filling and drainage flowrates required by the measure. Double block valves can be required in some locations.

It is important that the operation of the valve, particularly drain valves, has no demonstrable effect on the measured volume by displacing fluid during operation.

A means and method of providing for calibrations, including connections and valves, should be defined to allow reproduction of the original calibration conditions.

Examples of the different aspects of design are given in subsequent clauses of this document.

5.2 Design to indicate the volume — Resolution

5.2.1 Neck size

All measures rely on a means to indicate or define the volume contained when full. This can be by attaining a level at the top, a weir or overflow, or by observing the level in a window and comparing to a scale.

A measure can require a large diameter to contain the required volume but this provides a poor resolution of the level. To provide the required resolution, measures are designed to have a reduced area in the form of a vertical neck. This allows a large volume to be collected in the main body but the level in the neck to be observed to the required resolution. The narrower the neck, the higher the resolution.

While providing adequate resolution, the cross-sectional area of the neck should not provide a significant back pressure when the measure is being filled and air vented out. The neck should be long enough to allow the rising level to be observed and the flow to be stopped within the scale.

For high-accuracy laboratory test measures the neck should be relatively long and narrow; field test measures have a shorter and relatively wider neck. The neck area is the sum of the cross-sectional areas of the neck, the sight tube and any external top filling tube.

As a guide the neck diameter should be such that 3 mm change in level equates to the maximum uncertainty in volume required from the measure. For standard measures, the neck volume should be in the order of ± 1 % of the total volume of the tank. Where the filling flowrate is high, or when a high resolution is required, the neck, and any sight glass and scale, can be longer. Suggested neck diameters for standard measures are given in OIML-R120[4].

5.2.2 Gauge glass

Measures not employing a top weir should have means of indicating the volume contained. This can either be a separate sight tube in parallel with the neck or a window inserted into the neck. There should be a scale associated with the gauge glass. The gauge glass may be longer than the scale associated with it to allow observation of the level in the tank above and below the measuring section.

Gauge glass should be manufactured from borosilicate glass unless there is an incompatibility with the fluids used.

The gauge glass scale allows reading of the level in the neck and has a scale marked or engraved legibly and indelibly on the scale. Although scale marking could be engraved on the glass, a separate scale alongside the glass is preferred. A separate scale allows better clarity when reading a meniscus and allows for adjustment.

Additional information on how to read the level when a meniscus is formed against a gauge glass is given in Annex D.

The diameter of a sight tube should be such that the desired level of resolution is achieved, taking into account the formation of a meniscus forming at the tube wall. Standard measures have recommended tube diameters given in OIML-R120[4].

Sight tubes should have a clear, unimpeded entry to allow the fluid to flow evenly into the tube without restriction. The top of the tube should vent into the top of the neck, below the top, hence allowing free venting of vapour and overflow to the neck. There can be an access to the top of the tube to allow cleaning without disturbing the tube and hence the volume of the measure.

In adverse environmental conditions, it should be ensured that sight tube temperature does not differ significantly from the tank temperature. Such a difference can introduce a volume error due to differential temperature expansion of the fluid within the tube.

In conditions of gusting wind, it can be difficult to obtain a settled level reading in the gauge glass due to pressure variations acting on the surface of the liquid in the neck of the tank. Precautions should then be taken to protect the top of the neck from the wind.

Measures can have an overflow weir to set the datum level rather than a gauge glass and scale. A weir should have similar requirements for resolution and the diameter (length) of the weir. Weir edges should be machined horizontal and the edge chamfered outward to a sharp, defined edge.

5.2.3   Scales

Examples of scales and markings are shown in Figure 2. Scales should be fitted either side of a gauge glass or window in a manner that the level can be seen and related to the scale reading in an accurate and consistent way. This should maximize clarity and minimize error due to misreading, identification of meniscus and operator interpretation. A single scale should be fitted to one side of a gauge glass tube; however, a frame design with scale marks either side is preferred. Scales should be positioned between 2 mm and 5 mm from the side of the window or tube and slightly in front.

A single scale should be positioned to ensure there is no bias in the reading while a scale with frame design and units on each side should be of one-piece construction. Scale marks should extend to the edge of the scale beside the tube or window.

Where dual scales in different units are fitted, for example marking in metric and US customary units, the right-hand scale should be the metric scale and the second scale engraved appropriately on the left-hand side. It would be normal to have a single nominal value determined at calibration on one scale with the secondary scale marked according to a predetermined conversion factor.

Scales should be clearly and indelibly marked and manufactured in materials which should not be subject to wear or corrosion. Marking or engraving should be differentiated from the scale material by a different colour.

Scales should be securely attached but may be adjustable to bring the nominal scale mark in line with the calibrated volume. The adjustment should be secure and sealed to prevent tampering.

Scales for standard measures should be marked with the nominal volume mark centred on the scale. Offsetting the nominal volume mark to allow a longer graduated scale either above or below the nominal mark is allowable for specific applications. The nominal volume mark should be extended across the whole width of the scale.

For standard measures the scale length are normally ± 0,5 % of the tank volume. A longer scale can be required for some applications.

Scales should be clearly marked and major and minor unit markings are advised. Graduation marks should extend to the edge closest to the gauge glass.

As good practice it is recommended that graduations should be spaced at 2 mm but no less than 1,5 mm apart. For standard measures, graduations should be set to indicate an increment relating to 0,05 % of the volume of the measure. For primary measures this interval should be reduced to 0,02 % and for high resolution primaries reduced further to 0,01 %.

It is important that scales are oriented to reduce the potential for parallax error when reading. Provision to allow an operator to attain a horizontal eye line at any point across the range of the scale length with safe and secure access is required.

The scale should indicate the value and units of the nominal volume and the units of increment. The value of nominal volume may be shown anywhere on the scale or against the nominal scale mark. Scale increments should be values of volume or as ± volume increments from the nominal volume. Marking as ± percentage from the nominal volume is not advised. The prime importance is clarity of scale increment markings to reduce any chance of misreading. Dual scales should show units on both sides.

The scale plate should show the nominal volume as indicating the volume to contain or volume to deliver. It is important, however, to recognize the calibrated volume could have been derived differently from the nominal volume and steps should be taken to avoid confusion.

It can also be useful to show the standard temperature on the scale in addition to showing on the marking plate.
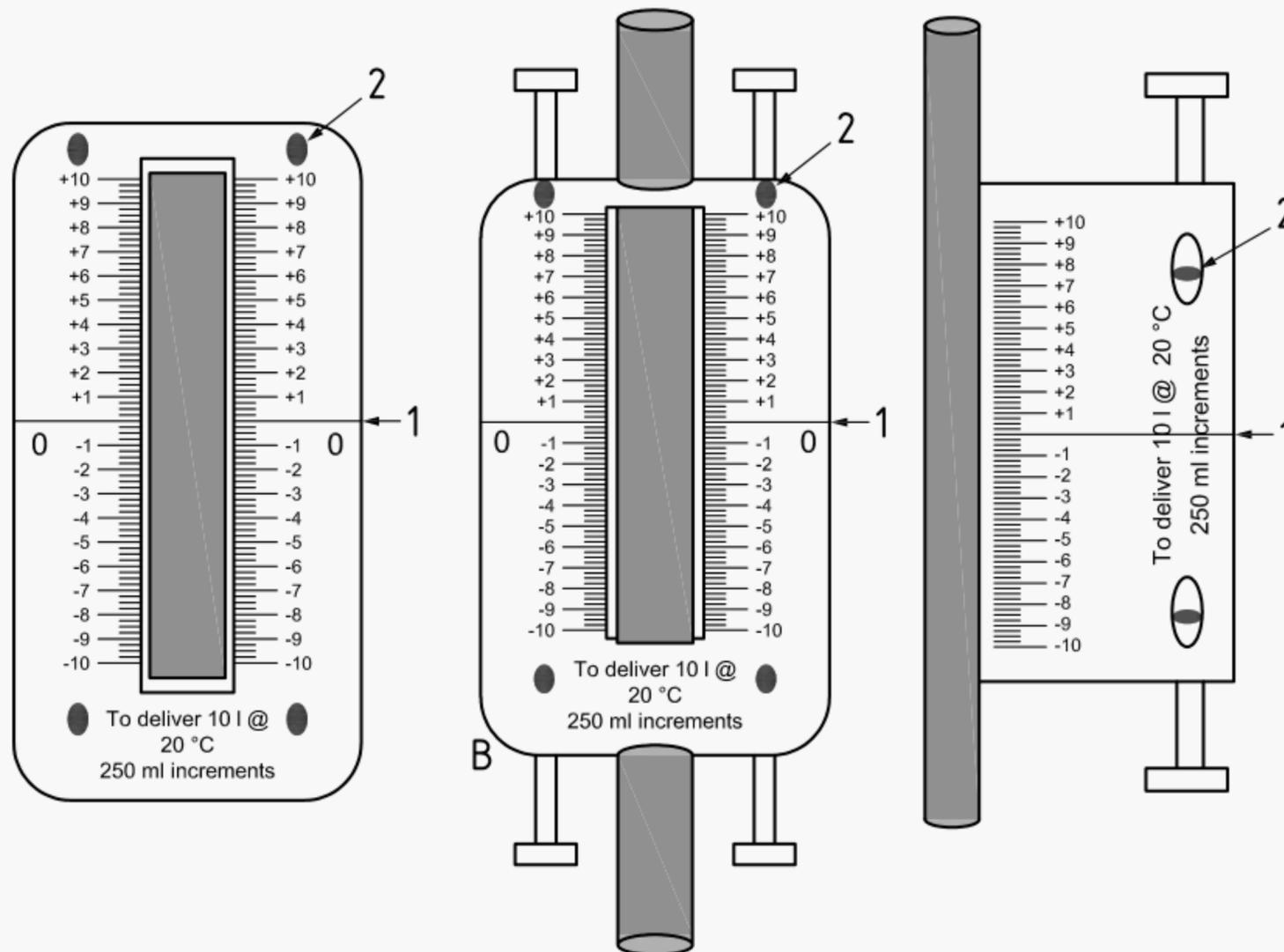
Key

1   nominal volume mark

2   fixing screw and seal

Figure 2 — Examples of scales

5.2.4   Adjustment of scale and calibrated volume

Scales can be fitted to allow adjustment of the nominal scale mark to the calibrated volume. Such adjustment should be secured and sealed to prevent unplanned or unauthorized adjustment.

It is good practice at the time of manufacture, or first calibration, to designate a fixed reference mark as a scale datum to provide a reference to which subsequent scale adjustments can be referred. This can be an engraved mark next to the scale or some permanent fixture on the neck to define the position of the original nominal or calibrated volume mark on the scale. This scale datum should be adequately described on calibration certificates.

Although it is unusual, any adjustment to a weir should have similar requirements for the setting and securing the weir.

A means for adjusting the nominal volume of the measure can be provided, normally by an adjustable plunger inserted into the side or top cone of a measure. The plunger displaces volume when inserted and adjusted. The plunger should be demonstrably leak-tight and the entry into the measure should not provide a trap for air or liquid. Normally the displacement adjustment will not exceed 0,1 % of the nominal volume. The adjustment should be able to be locked, secured and sealed.

5.2.5   Levelling

To obtain accurate measurement, the neck of the measure, external gauge glass and filling tube should be vertical. The error introduced by an incorrect level can be determined by examining the height differential across the neck, relative to difference from the vertical.

Brim measures and automatic pipettes should have the weir or edge horizontal and at right angles to the neck.

Establishing the neck to be vertical is vitally important before each use and should be checked between readings to ensure no change has occurred. As the level may change in use it should be established when the measure is empty and again when full. Increasing weight might cause movement.

A spirit level or similar device positioned across the top of a neck is not generally suitable. Level should be established by checking the neck section is vertical. Where the neck is of large diameter and the manufacturer of the measure has ensured the top edge is machined at right angles to the neck, as is the case for brim measures, a spirit level positioned across the top of the neck may be acceptable.

Permanent level gauges can be fitted to measures. These can consist of a two-axis level gauge, two level gauges set at 90° orientation, on a single plate or two plates at right angles. Alternatively, a single "bullseye" level mounted on a plate can be used. The plate(s) should be securely fixed to the tank and of suitably robust construction. The plate surface should be horizontal with reference to the neck being vertical. An adjustment mechanism can be provided to ensure this orientation is established. Level gauges should be securely fixed and sealed. The resolution of the level gauges should reflect the required uncertainty of the measure. Levels with sensitivity of 2,5 mm graduation per 45 min of tilt angle, or better, are usually required.

5.3   Additional design aspects

5.3.1   Temperature measurement

For small test measures, the temperature of the liquid and the measure material can be measured by inserting a thermometer probe into the neck and extending into the body of the measure. This method should follow a procedure to ensure inserting and removal of the probe does not affect the measured volume.

Temperature in larger measures is determined by providing one or more fixed thermowells permanently inserted into the body of the measure. Thermowells are preferred over directly inserted probes as they have a fixed displacement within the measure and allow the measurement probe to be removed for calibration.

Thermowells should have adequate immersion depth into the body of the measure. Larger measures might require multiple measurements to determine and then provide an average temperature. The minimum number of thermowells recommended is given in Table 1.

Table 1 — Number of thermowells required

Size of measure litres	Minimum number of thermowells
Up to 500	1
500 to 2 000	2
More than 2 000	3

Thermowells should be installed to ensure there is no pocket to trap air or liquid at the wall inside the measure. It is advisable to install with a downward angle to ensure liquid drains off the surface.

Thermometer probes should be a tight fit to the thermowell, extend to the bottom of well and minimize the exposed length outside of it. The thermowell should be filled with conductive fluid or paste.

It is recommended that the uncertainty of thermometers used should be 0,2 °C or less. However, for the highest accuracy applications and for primary measures, uncertainty of 0,05 °C is required.

For the calibration of a primary measure where an intrinsically safe thermometer is required, an uncertainty of 0,1 °C would be acceptable if a suitable thermometer is unavailable.

It is accepted that, in all but the most extreme conditions, the measure material can take up the same temperature as the fluid. However, in adverse environmental conditions, such as wind, rain or sun, protection should be given to the measure through screens or insulation. This ensures the material and fluid temperature stabilizes and avoids temperature differentials around and within the measure. Variations in temperature can create significant changes in the volume leading to errors and poor repeatability.

More detail on thermometer specification and temperature stability conditions is given in Annex B.

5.3.2   Valves and connections

All components of the calibration and proving system, including the connecting pipes, valves and manifolds, shall be in accordance with applicable pressure codes. Provision shall be made for expansion and contraction, vibration and reaction to pressure surges. Valves or provision for blind flanges to isolate the measure from line pressure when not in use can be provided. Valves should be securely supported to ensure no undue force is applied when a manual valve is operated.

Provision should be made for the safe disposal of liquids or vapours drained or vented from a measure. The potential for spillage or overflow from measures should be recognized and provision be made to capture and safely dispose of any spillage. Portable measures should be emptied, cleaned and all vapour removed before transporting.

Use of measures with volatile fluids, such as petrol, requires special consideration regarding the flammable nature of the fluid and the potential for static electricity discharge. Change of fluid between volatile and non-volatile fluids, such as petrol and diesel, can produce a flammable vapour mixture within the measure. This use is sometimes referred to as switch loading. Electrical connection of the measure and associated pipework to earth is required when using hydrocarbon or inflammable liquids.

Connecting pipework should have diameters selected to allow for the flowrates within the operating range of the measure. This should minimize variation of fluid retention when draining. This applies particularly if the measure is filled or emptied and liquid is drained from connecting pipework before and after each use (empty pipe configuration).

Establishing a defined transfer point between the measure and the device under test is recommended. The transfer point defines where fluid to be measured during a test passes from one measurement device to another. For any system, running with a full or empty pipe, the quantity of liquid on each side

of the transfer point should be the same at the start of the test as at the end. Any difference in volume of liquid left in the connecting pipe can create a difference between the quantities measured by the two devices. The transfer point is typically the drain valve body or a weir.

Valves should be leak-tight. They should enable fast and repeatable operation allowing flow to be stopped with the correct level in the neck. They should also ensure reproducible drain times.

Some valve designs can displace a volume of liquid when operating or have an internal difference in volume between open and closed position. The potential for this should be recognized when a valve is chosen for the application and tested to ensure there is no effect on the measured volume introduced by valve action.

As bottom valves become part of the measure volume, security seals are normally applied to the valve body and connections to the pipework.

5.3.3   Size of measures

Volumetric measures may be manufactured to any size to suit a particular purpose, for example measures to accompany a small volume pipe prover. For standard measures, specific volumes are defined. Using defined volumes allow measures to be calibrated using single or multiple fills from reference measures without resorting to smaller make-up measures or flasks.

Except for special applications, the nominal capacity should be of the form 1×10^n or 2×10^n or 5×10^n, where n is a whole number, either positive or zero. Units should be ml, l or m^3. For non-metric tanks a similar size sequence would be established.

After manufacture or repair, the calibrated volume of a measure would be expected to be within ± 2 % of the designed nominal volume.

Standard measures are recommended or specified by OIML-R120[4] and NIST-105-3[5] with volumes given in Table 2.

Table 2 — Recommended sizes for standard measures

Standard test measures	Small proving tanks	Large proving tanks	Very large proving tanks
1 l[a]		*1 m³*	*1 000 m³*
2 l[a]	2 l	2 m³	2 000 m³
3,8 l[b]	3,8 l[b]	—	↓
5 l	5 l	5 m³	Continued
10 l	10 l	10 m³	
20 l	20 l	20 m³	
	50 l	50 m³	
	100 l	100 m³	
	200 l	200 m³	
	500 l	500 m³	

Sizes in italic are specified in OIML R120[4].

a   Small standard measures in carbon fibre or stainless steel are available.

b   1 US gallon.

Glass volumetric measures, flasks or cylinders are not covered by this document but may be used, usually with volumes less than 3,8 l, as additional volumes in the calibration process. Standard sizes for glass flasks are given in Annex C following guidance given in ISO 4788·

The restriction of a test measure size to 20 l reflects manual handling limitations. Some older standards allowed for test measures up to 100 l.

5.4 Filling and drainage

5.4.1 Drainage times and fluids

Volume measures have a defined capacity between the full and empty datum. This defines the nominal volume of a measure to contain when dry. When a measure is filled then emptied, residual fluid clings to the wall, slowly draining away over time. Most test measures and proving tanks are used to deliver, hence wetted. The calibrated volume is therefore to deliver. Initially the nominal volume of a measure may be shown as either the volume to contain or the volume to deliver, which could differ from the calibrated volume to be referenced in use.

To obtain reliable and consistent measurements the measure is first filled then emptied for a consistent length of time. This drain time is defined during the initial calibration of the tank and then used for subsequent calibrations and when the measure is in use. The drain time is recorded in the tank records and calibration certificates and should also be clearly defined on the marking plate.

Drainage of invertible test measures is specified by both a time and a method. Draining an invertible measure is done by pouring from one side of the neck until cessation of the main flow then inverting vertically for the specified final drain time. For standard test measures the final drain time should be 10 s. Some regulations for standard measures and particular liquids require a first drain (pour) time to be 30 s. For this and other specifications the main flow time and angle of pour should be consistent and specified in the operating procedures, calibration certificates and reports of calibrations using the measure.

Many test measures and smaller proving tanks with bottom drains have a single-stage drain time. This requires the drainage to be through a defined valve setting and drain pipe arrangement. This is usually draining through a fully open valve and short straight length of pipe to a container without restriction. Such measures should have no restrictions or alterations made to the outlet from the drain valve. The drain time is specified as the time between opening the valve to when the valve is closed. It can be a specified time or be defined in a similar way to a final drain time.

Other measures and proving tanks may have the drain time divided into two stages: i) the first drain time where the majority of the liquid is drained at a high flowrate; ii) the final drain time where the last of the liquid is drained out to leave a consistent residual volume. Each stage can have a defined time, tolerance on the time and a defined method assigned to it.

The first drain time reflects the time allowed to empty the tank of most of the content. A significantly fast or slow drain time due to variations and differences in the drain pipework can affect the residual volume in the tank. For this reason it is advisable to drain the fluid with the drain valve fully open. The first drain time is not usually critical and a wide tolerance is allowed. It is good practice to specify the allowable range of first drain time in the design and in the operating procedures. It is also good practice to record the first drain time and description of the pipework on the calibration certificate and subsequently when using the measure.

Typical first drain times lie in the range of 10 s to 180 s for prover tanks. It is also advised to have the fall rate of the level of liquid in the body of a measure exceed 1 cm/s.

Cessation of the first drain time, hence the start of the final drain time, should be identified, defined and recorded in the calibration certificate and the operating procedure.

Specifying the end of the first drain time varies with tank design and use. Common options to define the end of the first drain time are as follows:

a) For an invertible measure, main flow is defined by pouring from one edge until the flow breaks into drops.

b) For a proving tank, where there is an open drain, main flow ceases when the flow breaks into drops.

c) For a proving tank with a closed drain and a window, sight glass or weir, the main flow ceases when the flow breaks into drops.

d) For a proving tank with a bottom neck and sight glass, main flow is stopped when the level is seen within the scale and the drain valve closed. The level is either read or lowered through a smaller drain valve to a datum after the defined drain time.

e) For a proving tank with a bottom weir, the main flow is considered to be completed when it is observed to break into drops. The final flow to the weir is completed after the defined drain time.

f) For a measure with a fixed restriction in the drain outlet and consistent fluid viscosity, there may be a single drain time from open valve to closed valve.

It is good practice to define and document the criteria used to decide when the flow is observed to break into drops as this is open to interpretation. For example, this could be defined as one drop every second.

The final drain time of a two-stage drain commences immediately after cessation of the first drain time and finishes with the closing of the drain valve or retuning an invertible measure to upright position.

Final drain time may finish at a defined condition, i.e. a very slow dripping rate, but is almost always a specified time. Final drain time of 30 s is commonly specified for standard test measures but can be longer for larger proving tanks.

Where a single drain time is specified this has to satisfy the criteria of both first and second drain times. Single drain time is usually applicable to measures which drain directly to an open container and where no restriction can be placed on the drain outlet.

Drain time is not only a specified time but also a description of how it is achieved. Where a measure is drained through variable drain restrictions, the resistance introduced by the pipework should be recognized as potentially reducing the flowrate, leading to a longer first drain time.

The clingage of fluid increases with increasing viscosity and surface tension. Measures are normally calibrated using water with viscosity around 1 mPa s. It is normally accepted that subsequently using a measure with fluids of up to 5 mPa s does not introduce undue error, although for the highest accuracy applications the effect of variations in viscosity, even variations in water viscosity with temperature, should be assessed.

Use of a measure with fluids above 5 mPa s or below 1 mPa s should be accompanied by an assessment resulting in a correction factor applied to the volume or an adjusted drain time. To assess or derive the correction the measure should be filled with water then drained for the specified time(s). At the end of the drain time the valve is not closed but any further water draining from the vessel collected and the quantity recorded as a variable with time. The exercise is repeated with the higher viscosity fluid. The difference in the volumes collected after the required drain time allows an estimate of the potential error. It is to be noted that viscosity varies significantly with temperature and hence this exercise may have to be repeated at different viscosities and temperatures.

The defined drain time is used for all applications using the measure. If, however, a different drain time from that specified on the measure calibration certificate has to be used, this should be clearly stated in the procedure and on any certificate issued along with the assessment of additional uncertainty.

NOTE   NIST have developed a formula to correct drainage time for viscosity applicable to changes in water viscosity. This is only of interest to the highest accuracy laboratory work. It is not currently extended to apply to higher viscosity fluids.

5.4.2   Filling and drainage methods

To allow drainage without entrainment of air, an anti-vortex device, plate or vortex breaker can be installed in the measure at, or above, the outlet. This should be designed to ensure no air or liquid can be trapped around or within the device when the measure is filled or emptied. It should be permanently and securely attached inside the measure.

To drain and fill a measure from the bottom, pipe(s) can be permanently fitted to the measure and are part of the measure volume. Pipes should be joined to the base of the measure by welding or flanged with metal-to-metal seals to ensure the volume cannot be altered. Installation should be robust and the

pipes supported. Pipes should be of material similar to that of the measure. Any pipe should be angled downward by at least 15° to ensure good drainage and have adequate diameter to allow free flow at the required rate.

A means to observe cessation of the drainage flow should be provided. This can be an open pipe discharge to atmosphere or a window in the drain pipe. Reliance should not be placed on audible means to detect cessation of flow. If drainage is by pumping, the suction and flowrate should be controlled to maintain the flowrate within specifications for the first drain time.

Bottom fill can be into the bottom neck, share an entry with the drain or be through a separate entry into the bottom cone. A filling entry should be positioned to minimize swirl and agitation within the tank to reduce aeration and evaporation.

To fill a measure from the top (top filling), the entry should avoid splashing or excessive flow down the inside wall of the measure. Test measures and smaller proving tanks, particularly for mobile installations, are usually filled directly through the top of the neck. Care should be taken to avoid bubble or foam formation during filling.

Where evaporation or foaming could occur, the installation of a filling tube is recommended. A filling tube allows top filling but discharges below the surface of the liquid in the measure. A filling tube would terminate far enough above the bottom of the measure to avoid restriction to the flow and reduce swirl and agitation.

Filling tubes can be installed as an insert inside the neck or directly into the top cone. The tube should have a large enough diameter not to restrict the flow into the measure while allowing air and vapour to vent from the neck and the tube. A vent connection should be made between the top of the filling tube and the top of the neck to ensure levels are equalised.

The filling tube becomes part of the measure and therefore should be made of the same material as the measure and should be fixed securely. A filling tube entering to the upper cone gives an additional volume and should be carefully considered when determining the resolution of the neck.

An open measure vents to the atmosphere from the top of the neck.

A closed measure prevents vapours venting to the atmosphere and therefore vents through a controlled vent arrangement or a vapour recovery system. This should not produce a pressure significantly above or below atmospheric pressure during filling or emptying. The top of the measure should be fitted with both pressure relief and vacuum break valves to prevent overpressure when filling or a vacuum when draining. While considering the design and strength of the measure, a pressure relief valve should be set from 20 kPa(g) to 35 kPa(g) (3 psig to 5 psig) and a vacuum break set to –20 kPa(g).

An assessment should be made to quantify and minimize the risk of loss of product through evaporation.

5.5 Markings

All volume measures should carry identification marking and information on an attached plate. The marking plate should be permanently fixed to the measure and be made of corrosion and wear-resistant material. The marking should be engraved or otherwise indelible. The marking should provide the following information as a minimum:

— identification or serial number;

— nominal volume at reference temperature.

All other required information should be available in the design documentation and on calibration certificates. Some or all of the additional information may be included on the marking plate as space allows.

Additional information should be:

— marking to show nominal volume is to deliver or to contain;

— manufacturer's name;

— date of manufacture or modification;

— material of construction;

— coefficient of expansion of the material (linear or cubical);

— reference temperature(s);

— first drainage time (with range of times or flowrates);

— final drainage time;

— alternative drain times for other fluids;

— definition of drainage time or cessation of first flow;

— bolt torque values for any removable openings;

— description of scale datum location.

Calibrated volume is not normally recorded on the marking plate unless the marking plate is to be changed at each calibration.

6 Test measures

6.1 Overview

Test measures are volumetric measures with capacities between 5 l and 20 l; 20 l being the largest tank deemed acceptable for manual handling. Smaller measures of 3,8 l (1 US gallon) are included in the definition as are smaller measures as low as 1 l manufactured in stainless steel or carbon fibre.

Except for special applications, the nominal volumes for standard measures should be of the form 1×10^n or 2×10^n or 5×10^n, where n is a whole number with the units being litres. A similar sequence is recommended for tanks using non-metric units.

Test measures may be designed to have the nominal volume to contain or to deliver.

The calibrated to contain volume should be determined, particularly for a measure to be used with volatile products where the measure is allowed to dry, through evaporation, between fills.

More commonly, measures are calibrated to deliver but may be designed and marked with the nominal volume to contain. The calibrated volume to deliver will be given in the calibration certificate.

Test measures can be used to calibrate flowmeters, other measures and devices and configured to fill. They can also be used as references to calibrate other measures and configured to withdraw. Test measures are predominantly used to define the volume delivered from other volume measures, pipe provers or storage tanks. This can be directly, or as a link in the traceability chain, for example to calibrate/verify a reference flowmeter.

Test measures can be of invertible or bottom drain design. They can be filled from the bottom or the top. Top filling can be directly into the neck or through a filling tube, as is recommended when foaming or aeration in the liquid is likely.

Test measures follow the general design and construction criteria given in Clause 4.

Examples of three test measure designs are given in Figure 3.

Figure 3 a) shows an invertible test measure with an upper neck and scale. The measure is top filled until the level is shown in the gauge glass.

Figure 3b) shows a standard test measure with a bottom drain and top neck and scale.

Figure 3 c) shows an invertible brim measure. The volume is defined by filling to the brim then pouring the contents for the appropriate drain times into the receiving vessel. Some designs establish the level by wiping the top surface using a plate to ensure consistency of the meniscus (strike measure). This type of measure is not discussed further in this document.

The features of a test measure designed as an automatic pipette are described in 8.2.
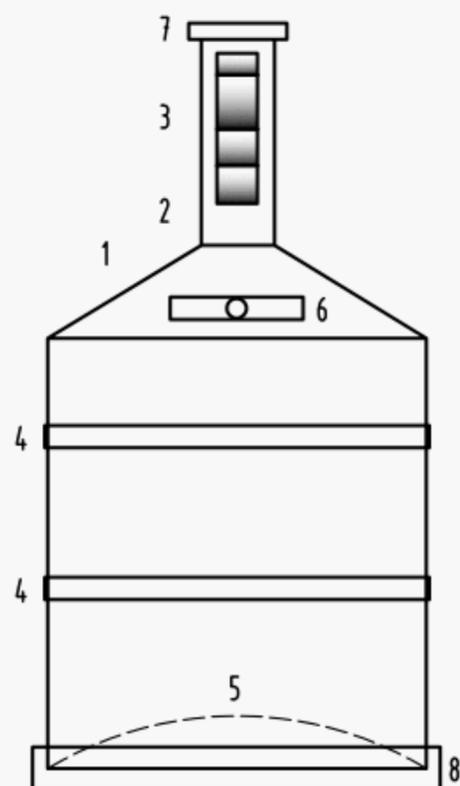
a) Invertible test measure
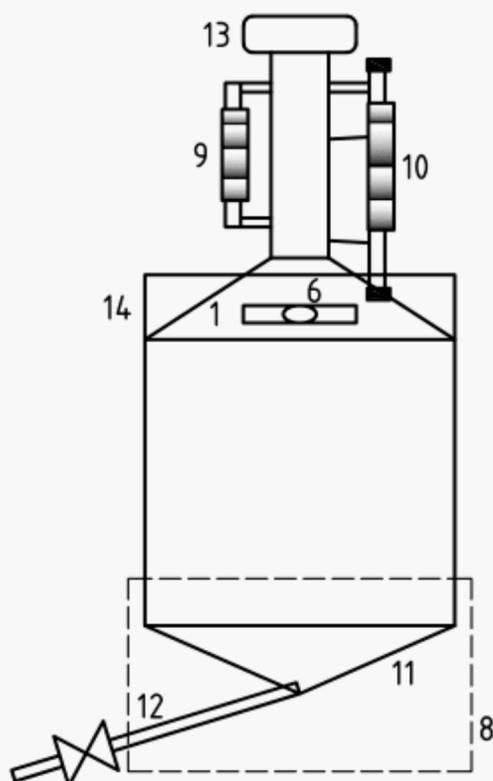
b) Bottom drain test measure
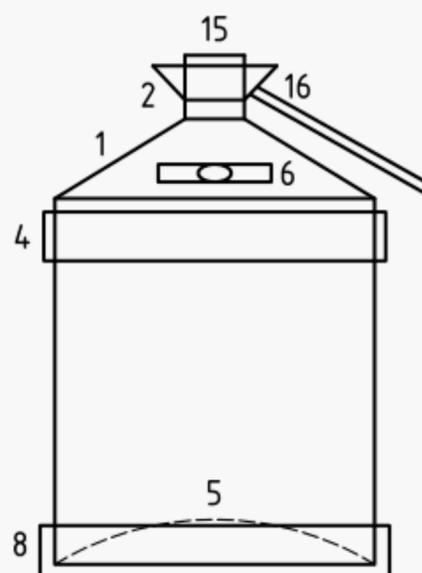
c) Brim measure

Key

1	upper cone	9	side tube and scale (option)
2	neck	10	sight tube and scale (option)
3	window scale	11	bottom cone
4	strengthening bands	12	drain pipe and valve
5	convex bottom	13	wide top
6	two axis level	14	overflow catchment
7	rolled top	15	brim-sharp edge
8	base	16	overflow and drain

Figure 3 — Test measures

6.2 Design and construction of test measures

Test measures should be constructed taking account of the following guidelines:

a)   The measure shall have the general features given in Clause 5 and in 6.1.

b)   The body should be a vertical cylinder with a top conical section leading to the neck. The base angle of the cone shall be approximately 30° to the horizontal. For standard measures the top cone angle should be 35°.

c)   There should be a ring around the base of the body to support the measure and three legs, or other means provided to support and level the measure.

d)   For bottom drain measures there should be a cone leading down to the bottom drain pipe with an angle approximately 30° to the horizontal. For standard measures the bottom cone angle should be 20°.

e)   The bottom of invertible measures can be convex upwards to ensure rigidity and that no distortion occurs when filling.

f)   The scale should be as described in Clause 5. For invertible measures a separate sight gauge tube is not advised due to the vulnerability to damage.

g)   The top of the neck above the measuring section can be expanded to reduce the rate of level rise, hence reducing the chance of overflow. Generally, there can be a strengthening band or rolled metal lip to the rim to give strength. For brim measures the top of the neck shall be machined at right angles to the vertical axis, be chamfered towards the bore and can have an integral overflow and catch volume which can be drained to a collection point or reservoir.

h) There can be an extended body section or trough above the start of the upper cone to act as a catchment if there is an overflow.

i) Lifting handles should be securely fixed to the body or the top of the neck to facilitate lifting and pouring. The design of such handles is not regulated; however, there are significant safety risks should a handle fail in service.

j) There can be a swivel-type handle fitted to the neck to allow vertical lifting and holding while pouring. This should be attached securely and in such a way as to not affect or alter the volume of the neck. Similarly, fixed handles on the body or in the base ring should be such that there is no distortion of the vessel when lifted.

k) There should be a means to ensure the measure is level in use and is checked and adjusted after every fill. The use of adjustable feet or shims could be required for field use. An adjustable base plate can be used to provide a level surface to place the measure during use. The sensitivity and stability is discussed in 5.2.5.

l) Bottom drain test measures do not normally have a bottom neck. The drain pipe is connected or welded into the lowest point of the bottom cone in such a way as to avoid any retention of fluid. The pipe should be vertical, or vertical with an extension pipe at a downward angle at no less than 15° to terminate in a drain valve which may be outside the circumference of the measure body.

m) As the bottom datum is defined by the valve, a means to monitor for leakage and for observation of the cessation of the first drain time should be provided.

n) Bottom-drain test measures should have a base robust enough to raise the measure above the level of the drain valve termination and have means to level the measure. The drain pipe should be supported and protected from damage when being transported, connecting pipework and when operating the valve.

o) Inspection and cleaning of larger test measures can be done by splitting the tank at the join between the top cone and the body. The joint shall be metal-to-metal and incorporate an "O" ring or a non-compressible gasket with specific thickness and material. Flange bolt torque should be specified and marked on the design records, procedures and the calibration certificate. The flange join is designed so that no air can be trapped when filling, nor any water retained upon draining. The measure should be recalibrated after any break of the joint. Where a measure is designed to be split or hinged on a regular basis for transportation, it should be demonstrated that no change in volume occurs when following the specified procedures. Such records give confidence that recalibration is not required after each reassembly.

p) As the displacement and volume of a top fill filling tube, if fitted, forms a significant part of the overall neck volume, the design should be carefully considered. The principles are described in 5.4.2.

q) The handling of vapour recovery or venting is described in 5.4.2. For portable measures it is specific to the application.

r) For safety reasons any measure carried in or on a vehicle should be operated to prevent any vapour or fumes entering the cab of the vehicle. It should be fully vented and purged after use and before being transported.

7 Proving tanks

7.1 Overview

Proving tanks are generally volumetric measures with a capacity greater than 20 l but measures designed as proving tanks are available with smaller volumes. Except for special applications, the nominal capacity for standard proving tanks should be of the form 1×10^n, 2×10^n or 5×10^n, where n is a whole number with the units being either m^3 or litres. A similar sequence is recommended for tanks using non-metric units.

These devices are designed for calibrating larger volumetric measures, displacement provers or the calibration (proving) of flowmeters. Proving tanks are almost exclusively to deliver measures. For the calibration of very large proving tanks, smaller proving tanks may be used to withdraw, i.e. to fill the device under test. Most proving tanks are, however, used configured to fill, i.e. to receive liquid from the device under test.

Proving tanks are usually calibrated by higher accuracy measures, often by multiple fills. Where a suitable weighing capability is available they can be calibrated gravimetrically. Very large tanks may be calibrated using a reference flowmeter.

The volume is determined by measuring the liquid level in the neck against scale graduations indicating incremental volume. Table 3 is given as guidance to the critical dimensions which are expected for proving tanks up to 5 000 l and the principles can be extrapolated to larger sizes. This table should be used as guidance and any tank should be designed to meet the criteria required for the particular application.

Table 3 — Suggested critical dimensions for proving tanks

Nominal volume	Minimum metal thickness	Neck diameter (minimum)	Neck diameter (maximum)	Drain diameter	Weir diameter (if used) (maximum)
l	mm	mm	mm	mm	mm
20 to 100	2	30	100	37–56	—
101 to 500	3	50	250	50–75	15
501 to 1 000	3	100	300	50–75	20
1 001 to 2 500	4	150	450	75–100	20
2 501 to 5 000	5	250	500	100–150	25
NOTE The size of the necks can be different depending on the required sensitivity and use of the tank.					

There are two basic designs of proving tank:

1) Volumetric measure with a cylindrical body, top and bottom cone sections and a top and bottom neck, with drain being taken from the bottom of the bottom neck. Tanks can drain through weirs as shown in Figure 4 or have a direct drain as shown in Figure 5. The latter has the bottom neck with a graduated bottom scale.

2) Volumetric measures where the body is an inclined cylinder with a top graduated neck located at the highest point of the measure have a bottom drain or neck at the lowest point, as shown in Figure 6.
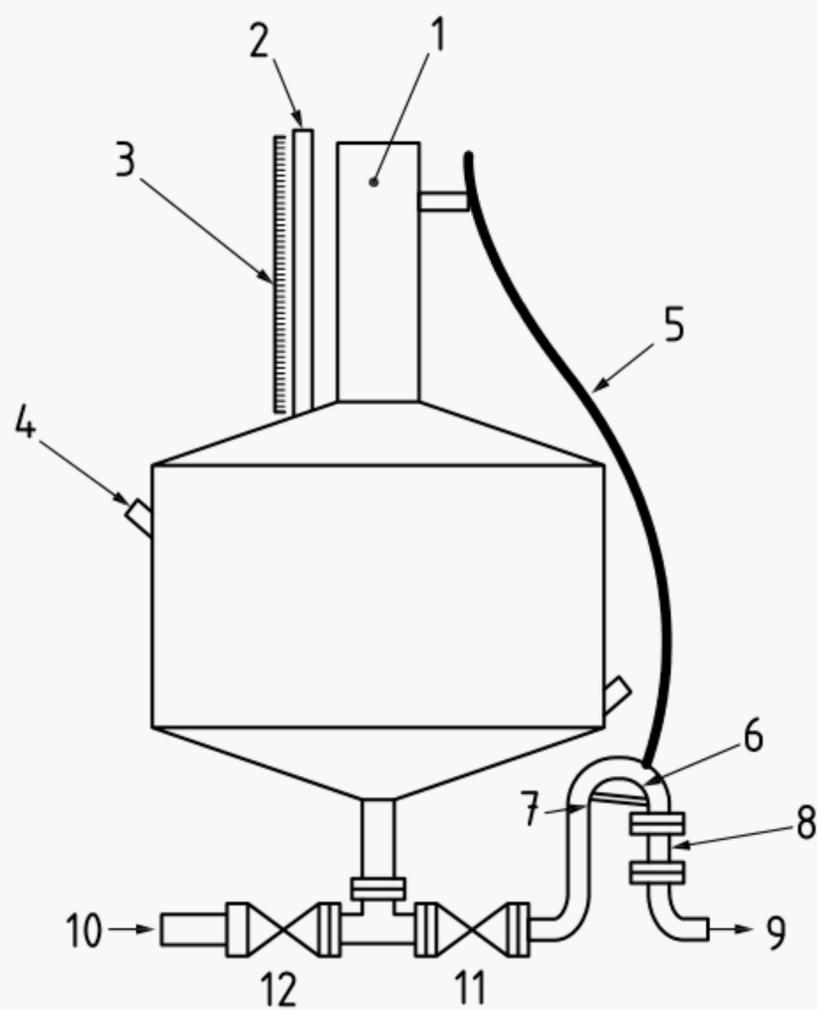
Key

1	neck	7	minor weir
2	sight tube	8	sight glass
3	scale	9	drain
4	thermowells	10	supply
5	syphon – break	11	drain valve
6	major weir	12	inlet valve

Figure 4 — Proving tank with bottom neck and draining through weirs
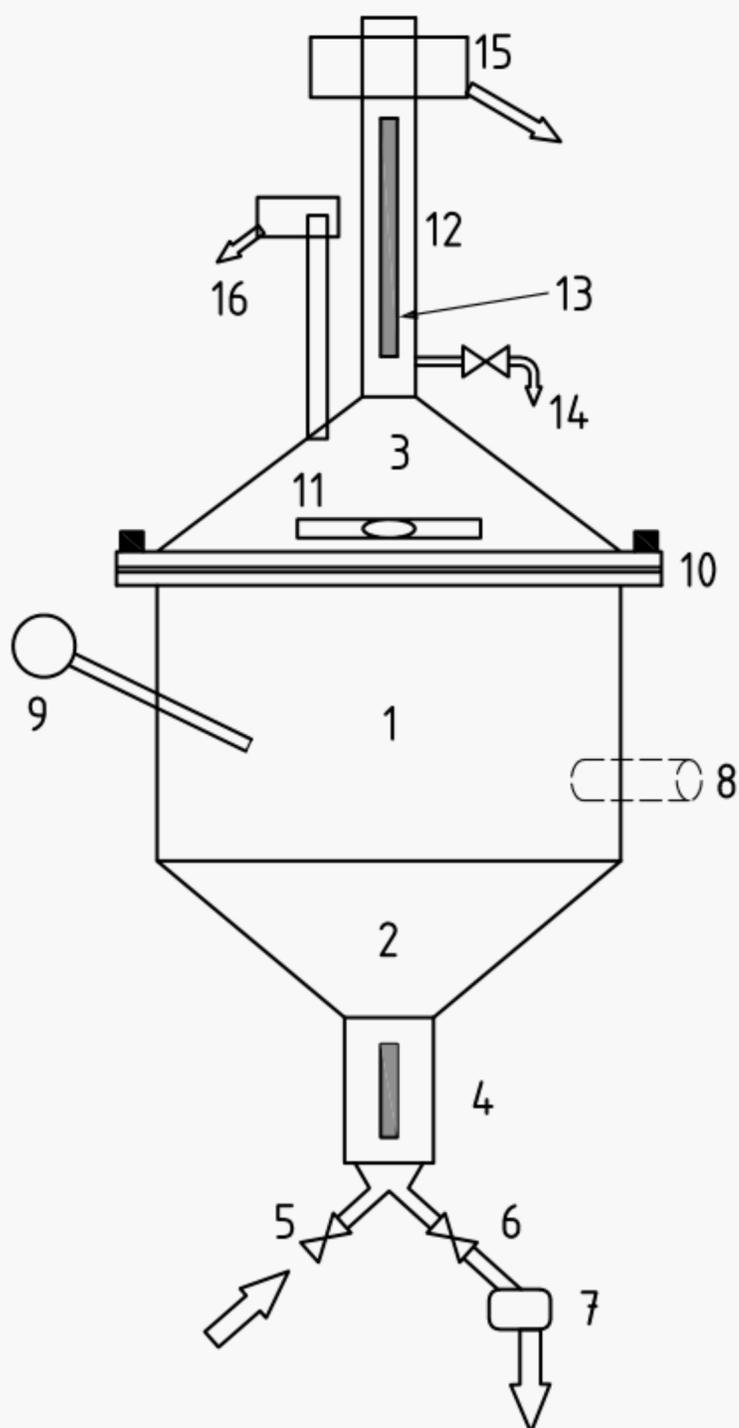
Key

1	body	9	thermowell
2	bottom cone	10	split tank (metal-metal join) (optional)
3	top cone	11	level
4	bottom neck with optional scale	12	top neck
5	bottom fill valve	13	scale window (optional)
6	delivery/drain valve	14	neck drain (optional)
7	window to observe delivery	15	top overflow or weir
8	plunger adjuster (optional)	16	side tube with minor weir (optional)

Figure 5 — Proving tank with bottom neck
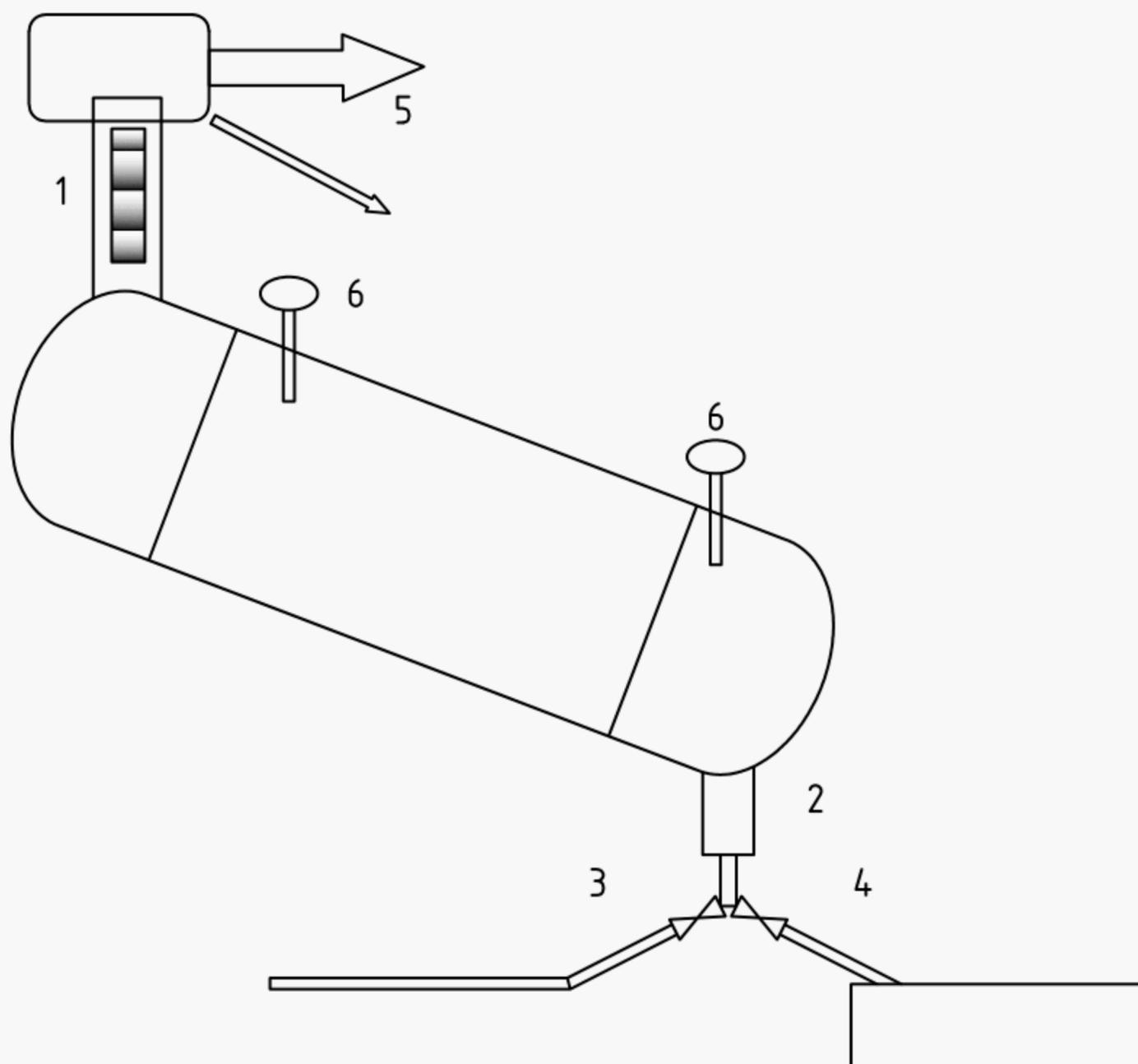
Key

1   neck with integral window scale

2   sump drain

3   bottom fill

4   drain

5   vapour vent and overflow drain

6   thermowells

Figure 6 — Inclined cylindrical proving tank

7.2   General construction

Prover tanks shall have the general features described in Clause 5. As the definition of a prover tank and a bottom drain test measure is based on size (weight), the designs referenced in Clause 6 can also be applicable.

7.3   Bottom neck

Proving tanks can be drained from the base of the bottom cone as described in 6.1 and 6.2. They can also be designed to have a bottom neck prior to the drain valve.

A tank with a bottom neck should have the lower datum below or within the neck. If the datum is within the neck, the bottom neck should be of a diameter not greater than that of the top neck. The lower datum shall be in the form of either valve(s) in the drain and fill pipes at the bottom of the lower neck, or a weir establishing a level within the lower neck. When a weir is used as the lower datum, a

syphon breaker of not less than 25 mm diameter shall be fitted to a hump in the outlet pipe. The lower internal level of the hump shall be above the datum weir. All valves employed shall be capable of being monitored for leakage.

Alternatively, a gauge glass may be installed to show a level in the lower neck.

A drain cock can be fitted at the bottom of the lower neck to completely drain the measure neck or to establish a level in a lower neck sight gauge during calibration. This valve should be closed, provided with a means to ensure it is leak tight and a seal or protection against opening when the measure is in service.

7.4 Strength

The proving tank should be robust and the metal thickness adequate to prevent distortion when the tank is filling or being emptied. While standard proving tanks up to 5 000 l have recommendations on steel thickness for some sizes suggested in Table 3, larger tanks require detailed structural design.

7.5 Support

The supports for the tank should allow for levelling and be designed to avoid any undue stress on the mounting and fixing to the tank body or base. Trucks or trailers with tanks fitted should have jacks or supports to stabilize and level the vehicle with final levelling being carried out at the tank.

7.6 Size

While standard proving tanks have recommended or mandatory neck sizes and lengths, non-standard and larger tanks are designed to suit the application and the available height.

7.7 Mobility

Where the tank has to be mobile or transported, lifting points should be easily accessible and located such that lifting cannot damage, distort or stress the tank. Protection should be provided for gauge glasses.

Where a measure is designed to be split or hinged on a regular basis for transportation it should be demonstrated and recorded that no change in volume occurs when split and reassembled following the specified procedures or that recalibration is required after each reassembly.

7.8 Overflow and vapour recovery

Due to the larger volumes and higher flowrates, care should be taken with the design of the filling arrangements to avoid overflow.

If specified, vapour should be extracted from just below the top of the neck. Any system should be designed to avoid over-pressure when filling the tank or under-pressure (vacuum) when draining. Under-pressure can lead to evaporation loss or, in extreme cases, the vacuum damaging the tank. Pressure relief valves can be considered and sized to accommodate the potential flowrates of gas when the tank is being filled. More information is given in 5.4.

The potential for spillage, especially from overfilling large tanks, should be considered as high flowrates could lead to large quantities of liquid being lost. Precautions should be taken to safely contain spillage, should overflow occur.

7.9 Filling and drainage

Tanks can be drained by gravity or by a pumped system. Pumped systems should be regulated to avoid a reduced pressure in the tank. Flowrate should be controlled to keep within the range of the first drain time specified for the tank, hence allowing for a reproducible drain time.

Top fill tubes should be sized with the resolution of the neck fully considered along with the potential fill rate and vapour/gas release.

8 Alternative designs

8.1 High-accuracy designs

Measures can be manufactured with accuracy higher than that achievable with a standard test measure or standard proving tank. To design a high-accuracy measure significant care should be taken with finish and quality of all joins and connections.

Additional care should be given to the neck to ensure consistent diameter and roundness along its length. Levelling should ensure the neck is vertical in service.

The neck, gauge glass and scale should be designed to achieve the desired resolution. This requires a longer and narrower neck section than would be required for a standard measure. In a narrower neck the effect of a meniscus should be recognized.

A narrower neck can place a limitation on the filling flowrate.

8.2 Automatic pipettes

Automatic pipettes are measures which are filled to a level determined by a weir at a set position in the top neck. The weir can be the top edge of the neck, a pipe parallel to the neck with a defined top edge or another weir device which defines a level within the neck. A common arrangement for a top overflow weir of an automatic pipette consists of a vertical overflow tube discharging into a transparent overflow chamber fitted with a drain and an air vent.

The weir may be in one or two parts. The top edge of the neck can provide the first weir from which fluid overflows. If in two parts, on stopping the fill the level drops from the top weir to the level of a second weir. This second weir is formed by a small-bore vertical tube in parallel with the neck, the top being below the top of the neck and the first weir.

Automatic pipettes are generally high-accuracy devices designed to withdraw liquid to a device under test. They are normally calibrated gravimetrically to deliver by draining directly into a weighing vessel (calibrated to withdraw).

An automatic pipette is filled until fluid overflows from the weir(s). It is then allowed to settle until no more liquid overflows and a meniscus is formed. The liquid is then drained out to the device under test following the defined drain times. It is usual to use a pipette to calibrate other measures.

An alternative arrangement is described as a semi-automatic pipette. In this case there is a drain valve placed at the base of the neck allowing, after filling, the level to be reduced to a consistent volume reading or datum mark on the scale.

8.3 Proving tanks with bottom sight gauge

Proving tanks with a bottom neck define the calibrated volume as being between the top and bottom datum marks. The volume is measured by the difference between the volume read on the bottom scale and the top scale. The bottom scale is marked in a similar way to the top scale. For a standard measure the bottom scale should have a range of 0,5 %.

To use a measure with a bottom scale there can be a main drain valve. However, a secondary (smaller) valve can be fitted to allow more controlled drainage, hence establishing a level within the range of the bottom scale. The drainage rate is reduced by throttling the main drain valve then closing it just as, or just before, the level is observed in the bottom sight gauge. The small drain valve is then opened and draining continues until the level is below the datum line and the valve closed. The drain time commences when both valves are closed and the level in the gauge scale is read and recorded as the empty volume after the drain time has elapsed.

In the time between reading the empty volume and starting to fill the measure, the observed level in the bottom scale rises due to liquid draining down the walls of the tank. This increased level is not to be used in the measurement; however, it could be used as a good measure of correction factors to be applied for different viscosity fluids.

9 Calibration

9.1 General

Regular calibration of measures is required to provide confidence in the volume, particularly where mobile or portable measures are used. The frequency of calibration can be defined by regulation or policy from the user. It is good practice, however, not to rely on defined calibration intervals alone but to examine the risk of a change in volume developing while in use and recalibrate at intervals to give the required confidence.

Common practice in industry suggests that high-accuracy reference measures are calibrated annually. Fixed or laboratory measures and proving tanks require recalibration every three years, while mobile field measures which are carefully controlled require recalibration every two years. Recalibrations are, however, required after any incidence of damage or contamination.

There are several techniques for calibrating a volumetric measure. This document deals with three of them:

1) gravimetric by withdrawing (water-draw or Ex) or by fill (In) outlined in 9.3;

2) volumetric by withdrawing (water-draw or Ex) or by fill (In) outlined in 9.4;

3) reference flowmeter by fill (In) outlined in 9.5.

9.2 Common calibration procedures

To calibrate a measure there are a number of generic steps which can be followed:

a) On receipt, and prior to starting the calibration, the external features of the tank should be inspected. Any damage to the outside of the tank should be recorded. Seals on valves, scales and adjusters should be inspected for breakage or tampering. The scale datum position and the difference from the nominal volume mark should be recorded and compared with the previous calibration certificate.

b) The marking plate should be inspected and the necessary information recorded and compared with the manufactured specification and previous calibration certificates. Any discrepancy or missing information should be investigated.

c) Without opening the measure, inspect the interior as far as practicable. Observe for debris, corrosion or deposits on the wall. Clean the sight window/tube if possible without altering the volume. If the measure has to be cleaned, determine if a pre-cleaning and post-cleaning calibration has to be performed. At the appropriate time, clean the measure internally with the appropriate solvents, water or detergent. Flush the measure thoroughly to remove any trace of cleaning fluids as these can reduce surface tension, hence altering residual volume.

d) For primary and reference measures calibrated in a laboratory the measure should be cleaned of all contamination before any work commences.

e) Check all valves and connections for leakage. Clean out thermowells and refill with appropriate conductive fluid.

f) Level the measure. Confirm accuracy of fitted level indicators.

g) Determine the method of calibration and the required uncertainty and acceptance criteria. Record this on the certificate.

h) Set up the calibration circuit with the appropriate reference devices. Ensure these have valid calibration certificates. Install temperature measurement devices in both the measure under test and at the reference. Ensure adequate supply of calibration liquid and means to recirculate and dispose of it. Provide environmental protection from sun, wind and cold to ensure a stable environment for the system. Ensure all required reference conditions, i.e. influence factors such as ambient temperature, can be maintained throughout the calibration period.

i) Allow the measure and the circuit to stabilize to the environmental temperature. For primary and reference measures this should never be less than 1 h and, if practicable, at least 6 h to stabilize conditions (see Annex B).

j) Confirm the drain times to be used and record this along with details of any drainage pipework and restrictions. A sketch drawing can be the best record. Record if the calibration is to be by fill or withdraw.

k) Fill the measure and check the valves and all connections for leakage. Drain for the specified drainage time. Repeat if contamination is observed in the liquid drained out. Repeat as required until the measure is seen to be clean and temperatures are stable. This process is to stabilize the circuit, test for leakage, establish filling and drainage times and establish a wet measure.

l) If monitoring water purity, either relative density or conductivity should be measured and compared with defined acceptance criteria. A sample can be taken and sealed for subsequent laboratory determination of (relative) density if necessary. This should be repeated at the end of the test from a second sample.

m) Record ambient conditions and other influence factors (e.g. barometric pressure and humidity). This should be repeated at intervals during the calibration and again on completion.

n) Carry out a number of repeat calibrations. Calculate the repeatability and the mean value. The repeatability can be compared against an acceptability criterion.

o) The number of repeat points depends on the required uncertainty. Regulatory requirements and common practice usually specify three or five consecutive points to fall within a stated range. This can be ± 0,01 % for reference measures or ± 0,02 % for working field standards (see Annex E). For high precision, measures up to 10 points (but no more than 15) may be repeated and the repeatability defined as the standard deviation of the mean.

NOTE   If the conditions are stable it is usually satisfactory to use the measured volumes rather than the calculated volumes to indicate an acceptable repeatability is achieved during the test. This can then be confirmed retrospectively after the calculation is completed.

p) The mean value is compared with the previous calibration if available. If the difference is considered significant then an investigation is initiated and where remedial actions are deemed necessary, for example cleaning the tank, the calibration should be repeated in full. For most applications a significant difference can be 0,02 % of the volume.

q) Verification of the neck volume as indicated by the scale marks, i.e. mm/l, is best carried out before the calibration and the result recorded and checked against acceptability criteria.

r) The final calculations are completed using corrected water densities and a calibrated volume and scale error established.

s) On completion, the scale or adjustment plunger may be adjusted to align the nominal scale mark with the measured calibrated volume. A confirmation calibration fill is then performed to verify the adjustment. The adjustment and the result of the confirmation should be recorded on the certificate.

t) If the results are dependent on a laboratory-determined relative water density, a preliminary certificate can be provided with a final certificate given after recalculation.

9.3 Gravimetric calibration

9.3.1 Principle

Gravimetric calibration provides the most direct traceability to standards of mass. As such it is the preferred method for the calibration of primary measures and automatic pipettes. Pure or drinking water (see Annex A) is the preferred liquid for gravimetric calibration. The purity of the water can be monitored by measurement of density or conductivity during the duration of the test.

Water density should be measured using a calibrated density meter providing the required uncertainty. This can be from a sample taken at the start of the test and a second sample taken on completion of the test. It is good practice to seal and retain samples in case results are questioned. From the measured density, relative density can be used to indicate water purity and also used to correct a calculated density based on pure water density formulae (see Annex A).

The liquid withdrawn from or filled into the measure under test is weighed. The weight is corrected for buoyancy to give the mass. Volume is calculated from mass and density.

The measure under test can be mounted directly on a weighing machine. The weight of the wet measure is recorded empty and again after the measure is filled to the datum. Alternatively, the measure can be filled, the full weight recorded and weighed again after emptying for the required drain time(s).

When the measure is not mounted on the weighing machine it can be filled from or drained into a separate tank or container mounted on a weighing machine. It is unusual to fill a measure from a container mounted on a weighing machine and it is difficult to provide for a bottom fill for a measure from a weighing container.

The weighing machine should be chosen with a range to suit the weight of the empty and full measure or container. The measure or container should be level, stable and secure on the weighing machine and mounted in such a way as to prevent uneven loading of the weighing machine when filled or empty.

The scale should be pre-calibrated using a suitable class of mass standards, for example as described in OIML R111-1[3], with calibration certificates from or traceable to a competent laboratory meeting ISO/IEC 17025 or an NMI. A further post-test calibration of the weigh scale can be carried out.

When weighing there should be no external connections such as drain or fill hoses, or thermometer cables which might influence the weighing.

Both the measure and the weighing machine should be shielded from environmental effects such as wind, sun, vibration and rain. The weighing machine in particular should be located in a draft- and vibration-free environment which is thermally stable. For more information on the use and calibration of weighing scales, EURAMET cg 18[10] can be consulted.

The humidity should be below that where condensation is possible on surfaces of the weighing container or the measure, taking into consideration the possibility of lower than ambient liquid temperature.

NOTE   For ambient conditions, see Annex B.

The connecting pipework between the measure and the container should allow complete and consistent transfer of liquid without retention, leakage or splashing.

Automatic pipettes require overflow water to be drained away in such a way as not to affect the weighing.

9.3.2 Calibration circuit and equipment

A circuit and pump is arranged to allow the measure to be filled from top or bottom, as required in a suitable time period. This can be from a pumped source or a gravity feed. A means to drain the measure to the weighing container, or to a drain or reservoir, should be established. Such a drain should not allow liquid hold-up in the pipework or restrict the flow.

Instruments to measure temperature in the measure, and optionally the weighing container, should be provided. Instruments to measure ambient temperature, atmospheric pressure and humidity should also be provided.

9.3.3 Procedure for calibrating a measure gravimetrically

There are a number of steps which can be followed to provide a gravimetric calibration of a measure.

a) Fill the measure under test to the nominal volume mark, brim or weir. Allow to stand until the temperature is stable. Note the water temperature.

b) Note the weight of the weighing container, or the measure, with drain valves closed.

c) Drain the measure, for the appropriate drain time(s), into the weighing container or drain sump. Close the measure drain valve(s) and wait until any residual water in the drain pipework drops out and weight is stable. Record the weight and, optionally, the temperature in the weigh container. Record the ambient air temperature, pressure and humidity.

d) Large measures which, when full, exceed the capacity of the scale can be calibrated by withdrawing the water to, or filling from, a weighing tank in stages. It is recommended that larger prover tanks requiring multiple weighings are calibrated by filling.

e) If a measure is to be calibrated by withdrawal, the drain time, particularly the first drain time, should be considered. Withdrawing slowly, especially if multiple withdrawals are required, extends both the total and the first drain times significantly, hence reducing the clingage volume. It should be ensured that the total drain time remains within specification. Times between withdrawals should be minimized and the last withdrawal should include all of the final drain time. For this reason it is recommended that larger proving tanks are calibrated by filling.

f) The indicated volume from the measure is corrected to the required standard temperature. The volume weighed is calculated from mass and water density and corrected to standard volume. An error value is calculated.

g) The procedure given in steps a to e is now repeated a further number of times as specified to establish repeatability and an acceptable mean value for the calibrated volume.

h) The first fill is considered as the "wetting" fill and pre-test result. The result should be calculated, recorded and retained. This result would not be reported on the final certificate or used to calculate the mean calibrated volume. It should be used, however, to identify if there is an unacceptable error. If the error is unacceptable an investigation should be initiated to the measure and the reference method and equipment.

i) A representative sample of the water should be taken mid-way through the test to allow a measurement of the density. Optionally, two samples are taken: one after the first reportable fill and one after the last reportable fill.

j) Calculate the mean error from the nominal volume.

9.4 Volumetric calibration

9.4.1 Principle

This method of calibration involves transferring a volume of liquid between the measure under test and the reference volume measure. The method requires the use of calibrated volume measures with suitable traceability to national standards, preferably provided by a competent laboratory meeting ISO/IEC 17025 or an NMI.

If primary reference measures are used as the reference, only pure or clean drinking water should be used. For the calibration of working standard and field measures, particularly large measures, another characterized and stable liquid may be used. Filtered brackish, saline or seawater may be used if

compatible with the measures. Light refined non-volatile hydrocarbon products, for example diesel or similar liquids, may also be used as long as the measure material is compatible. Highly saline water, brine or viscous hydrocarbons should not be used.

NOTE   For ambient conditions see Annex B.

A preliminary or first fill should be carried out as suggested in 9.3.

The result can also be used to match the total reference volume to the volume of the measure under test, and to identify any small volume measures required to add or withdraw liquid to achieve a volume reading close to the nominal volume mark.

For any liquid, a representative sample should be taken for determination of purity and relative density. Two samples are taken, one after the first reportable fill and one after the last reportable fill. These should be retained for future analyses. Monitoring of the purity and density can be undertaken at intervals during the test to ensure stability.

For liquids other than water it should be established that the expansion factors follow the appropriate algorithm.

9.4.2   Calibration circuit and equipment

As volume measures have a very limited range of measured volume it is important to have reference volume measures available to match the volume of the measure under test. Where a standard test measure or proving tank is to be calibrated, a reference of the same size should be used. Where the measure is large, the calibration can be by multiple fills of a suitable reference. It is recommended that no more than 10 fills be employed. For non-standard sized measures, or where the available reference does not provide the full number of complete fills, additional reference measures should be available to make up the full volume. This can require additional small volume glass measures or a weighing capability.

It is advisable, particularly for a first calibration or after a refurbishment, to have additional small measures and flasks available to measure any gross error identified in the calibration prior to adjustment. For multiple fills from larger measures it can reduce the time and improve repeatability if two reference measures are employed alternatively, one being filled while the other is emptying and draining.

The reference measure(s) should be located on a firm stable base and levelled. Provision should be made to handle any overflow or vapour recovery from the reference measures. The measure under test should be located in a similar manner. The reference measures should be located as close to the test measure as practicable and connected in a way to minimize or eliminate any residual liquid between the measures. If the measure under test is being calibrated by filling, the references can usually be located above the measure under test. If calibrating by withdrawal, the references should be below the measure under test.

The measures should be protected from environmental effects such as wind, sun and rain.

The humidity should be below that where condensation is possible on internal and external surfaces of the measures, taking into consideration the possibility of lower-than-ambient liquid temperature.

Calibrating by filling requires a circuit and pump arranged to allow the reference measure to be filled from top or bottom as required. The measure under test should have drain arrangements to allow drainage at a rate within the specified drain times. The measure under test can usually be top filled by draining the reference measures into it. This transfer pipework should be short and minimize any liquid retention in the pipe.

If calibrating by withdrawal the drain times for the measure under test should be considered when establishing the transfer of liquid from the measure to the reference(s). The measure under test can be filled from top or bottom. It is normal to have the reference top filled. Establishing transfer pipework for bottom fill without liquid retention would be difficult. The transfer pipework should be short and

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minimize any liquid retention. A means to drain the reference measure(s) safely for the required drain times should be established.

A means to measure the temperature of both the measure under test and the reference measures should be in place. If the temperature sensors are permanently installed, either directly or in thermowells, as part of a measurement system, these should be used for the calibration but should be calibrated and/or verified before the calibration starts. The reference measure temperature measurement should be calibrated and fully traceable.

9.4.3   Procedure for calibrating a measure volumetrically (water pour)

There are a number of steps which can be followed to provide a volumetric calibration by water pour:

a)   Fill the reference measure(s) and allow to stand. Check for any leakage and that the measure(s) are level.

b)   Fill the measure under test to the nominal volume mark. Allow to stand until the temperature is stable. Check for any leakage and that the measure is level.

c)   Drain the measure under test for the specified drain times and close the drain valves.

d)   Record the temperature of the reference measure (fluid). If the temperature is measured by inserting a thermometer directly into the neck, this should be done and the probe removed. The level is then re-established and read.

e)   Drain the measure for the specified drain time(s) by pouring into the measure under test then close the drain valve. The reference measure can then be refilled.

f)   This process can be repeated for multiple fills until the measure under test is full to show a level in the gauge scale. Recommendations given in 9.4.5.2 should be considered for multiple fills.

g)   Record the volume in the measure under test, along with the (fluid) temperature. The measure under test can be now be drained for the specified drain time(s).

h)   The procedure given in steps a to g is now repeated a further number of times as specified to establish repeatability and an acceptable mean value for the calibrated volume.

i)   Calculate the sum of the standard volumes of each reference fill. Calculate the standard volume for the measure under test and by comparison with the reference volume the mean error can be calculated.

9.4.4   Procedure for calibrating a measure volumetrically (water withdraw)

There are a number of steps which can be followed to provide a volumetric calibration by water withdraw:

a)   Fill the reference measure(s) and allow to stand. Check for any leakage and that the measure(s) are level.

b)   Fill the measure under test to the nominal volume mark. Allow to stand until the temperature is stable. Check for any leakage and that the measure is level.

c)   Drain the reference measure(s) for the specified drain times and close off the drain(s). Note that a reference measure not being used immediately may be left full and then drained just before use.

d)   Record the temperature of the measure under test. If the temperature is measured by inserting a thermometer directly into the neck, this should be done and the probe removed. The level is then re-established and read.

e) The measure under test is now drained into the reference measures for the specified drain time with the drain closed after completion. For multiple withdrawals the recommendations given in 9.4.5.2 should be considered.

f) When each reference measure is filled, the volume is read from the scale and recorded and the temperature read and recorded. The measure is then drained for the specified times.

g) This process can be repeated for multiple withdrawals with the last withdrawal conforming with the drain time specified.

h) The procedure given in steps a to f is now repeated a further number of times as specified to establish repeatability and an acceptable mean value for the calibrated volume.

i) Calculate the sum of the standard volumes of each withdrawal. Calculate the standard volume for the measure under test and by comparison with the reference volume the mean error can be calculated.

9.4.5   Additional notes on procedures

9.4.5.1   Repeat points

The number of repeat points depends on the required uncertainty and regulatory requirements. In general, three or five consecutive points are necessary. Repeatability acceptability can be ± 0,01 % for reference measures and ± 0,02 % for working field standards (see Annex E).

9.4.5.2   Multiple and additional fills

For a calibration by filling, the delivered volume should be less than the sum of the chosen reference measures. Smaller reference measures or graduated flasks may be used to add liquid to bring the level within the scale or up to the nominal mark of the measure under test.

For a calibration by withdrawal, the volume should be greater than the sum of the reference volumes. This then requires some liquid to be removed and measured using a smaller measure or graduated flask.

The volume collected, corrected to standard conditions, in additional measures or flasks will be added or subtracted from the reference volume. The use of a weighing vessel, rather than a measure or flask, can be advantageous as it has a much wider range to cover an initially indeterminate volume.

For multiple fills of, or withdrawals from, reference measures, each volume is individually measured and calculated. The temperature in each reference fill is allowed to stabilize, then the volume and temperature recorded. The volume for each reference measure is recorded and calculated separately before being totalled.

If a measure is to be calibrated by multiple withdrawals, care should be taken not to compromise the drain times. The drain time, particularly the first drain time, should be considered. Withdrawing slowly with time between withdrawals can extend both the total and the first drain times significantly, hence reducing the clingage volume. It should be ensured that the total drain time remains within specification. Times between withdrawals should be minimized and the use of two or more reference measures should be considered, one filling while the others drain. The last withdrawal should include all of the final drain time from the measure under test. For these reasons it is recommended that larger proving tanks are calibrated by filling.

9.4.5.3   Temperature measured by insertion in neck

Temperature can be measured by inserting a thermometer directly into the neck then the probe removed.

For a measure being filled this is done after the volume and the weight – if the measure is mounted on a weighing machine – is recorded. For a measure calibrated by withdrawal the water temperature is recorded the volume read or re-established after the thermometer is removed.

9.4.5.4 First fill

The first test fill is considered as the wetting fill. This test ensures all measures are filled and drained to the specified times, ensures there are no leaks or measurement issues and provides a pre-test result to identify if there is an unacceptable error in the expected volume which can be investigated before proceeding. If the error is unacceptable an investigation should be initiated. This investigation would include the condition of the measure under test, the reference measures and the method.

The first fill is used to confirm the reference volume matches that of the measure under test and identify additional measures which can be used to match the volumes to the reference. A representative sample of the water should be taken at this time to ensure purity, salinity and relative density. The ambient conditions should be recorded.

Following the first test fill, the required number of reportable results are carried out to establish a mean value and repeatability. The result of the first fill should be retained within the records of the calibration.

The result of the first fill is not reported on the final certificate or used to calculate the mean calibrated volume but retained in the records of the calibration.

9.5 Calibration by reference meter

9.5.1 Principle

Large volumetric measures can be calibrated by delivering known quantities of oil or water through a reference meter. For large tanks, the use of primary or even secondary test measures or proving tanks would involve a very large number of fills of the primary measure, increasing uncertainty and taking an impractical length of time. It is therefore acceptable to use a pre-calibrated reference flowmeter to fill the measure.

There is potential to draw air into the flowmeter if a reference meter is used to withdraw liquid from the measure. Reference meter calibration is therefore almost always restricted to fill.

The reference meter should have a valid calibration applicable to the liquids and flowrates to be used. Alternatively, the meter should be calibrated prior to and post use often at the site of the test and using the same fluid and circuit. This requires a reference volume measure, displacement prover or weighing system to be provided.

9.5.2 Calibration circuit

It might be necessary to calibrate or verify the reference meter before and/or after the calibration, in which case the installation should allow the connection of the reference meter to a reference measure or displacement prover and the measure under test.

Install the meter in line with the measure under test and a pump, drawing the liquid from a storage reservoir. The liquid should be stabilized to a temperature as close as practicable to the standard temperature. A control valve and quick-acting valve shall be installed downstream of the meter and pump to allow the flow to be controlled and the stopped when the level is observed within the neck of the measure. Precautions should be taken to prevent damage to the pump and circuit through water hammer and overheating when the flow is stopped.

Facilities for circulating the liquid through, and to, the reference proving tank and then changing this to fill the proving tank under test should be provided, ensuring the filling pipework in each case allows a clear transfer point to be established. Suitable drainage facilities for both the measure under test and the reference should be provided, ensuring total drainage times can be achieved within specification. A safe working area with a firm and solid base to allow levelling is required for both the measure under test and the reference equipment.

9.5.3   Equipment

9.5.3.1   Flowmeter

The reference meter can be of any type, with response time, resolution and linearity suitable for the test. High-quality positive displacement meters are normally specified; however, Coriolis meters used either as mass meters or volume meters have been employed successfully. The meter should provide a quantity-indicating mechanism reading in litres, which can be set to zero. The resolution of the indicator shall be at least 0,005 % of the quantity delivered to both the reference and the test measure. Alternatively, a meter with a pulse generator of equivalent resolution fixed directly to the output shaft can be used.

The flowmeter should have a calibrated flowrate range to match the tank volume and the flowrate being used to fill the measure. The maximum flowrate should not exceed that required to fill the measure, or reference measure, in 1 min. No minimum flowrate is specified; however, it should not be less than the minimum flowrate specified for the particular meter or less than that expected to be employed when the measure is in use.

9.5.3.2   Valves

The valve(s) used to control the flow into the tank should allow for the control of the flow during the fill, to slow the flow down to observe the level appearing in the sight gauge and to stop the flow at the required level. When closed, valves should provide a complete seal and a means to verify integrity. A double-seal type can be selected so that any leakage can be monitored or some other mechanism for demonstrating its integrity employed.

To achieve the requirements, multiple valves may be considered. A flow control valve can be installed upstream of the main fill valve and this does not require a high-integrity seal leaving the main fill valve to provide a simpler on-off function. A second, smaller valve can be installed in parallel with the main fill valve to provide a fast shut-off capability at a lower flowrate. This should be a high-integrity valve.

The valves should be sized to maintain the flowrate within the calibrated range of the reference flowmeter.

It is not acceptable to use the valve to reduce the flowrate below that specified for the meter to achieve a desired level in the neck of the measure (creeping the meter). The shut-off should be smooth and continuous.

9.5.3.3   Pumps

The pump shall provide a flowrate of not less than 50 % of the maximum flowrate of the meter to ensure the test can be carried out within the linear and calibrated range of the meter. Pressure shall be high enough to overcome system losses and avoid cavitation or flashing, but not high enough to require pressure corrections to the meter measured volumes. Provision should be made to ensure the pump is not over-pressured, overheated or damaged when the flow to the measure is stopped, for example a pump bypass line and pressure relief valve.

9.5.3.4   Instrumentation

Temperature should be measured in the measure under test, at the flowmeter and at any reference used to calibrate the meter. The thermometers should have valid calibration and an uncertainty suitable for the application.

Pressure should be measured in the pipe adjacent to the meter and this carried out with a calibrated pressure gauge or transmitter with a resolution of at least 10 kPa.

Ambient conditions of temperature, pressure and humidity should also be measured.

9.5.4   Procedure for calibration by reference meter

There are a number of steps which can be followed to provide a volumetric calibration of a measure using a reference meter:

a)   Fill the calibration circuit with the chosen liquid and circulate using the pump so as to remove any air in the system. Allow enough time to ensure the system becomes thermally stable. Circulation should include both the reference proving tank and the proving tank under test.

b)   Once the circuit is full and stable, first prove the reference meter against the calibrated reference proving tank. The flowrate or range of flowrates should be chosen to minimize any changes between the three stages of the calibration.

c)   Set the flow into the reference proving tank to the required rate.

d)   Fill the reference proving tank and stop the flow. Empty the proving tank and allow it to drain for the specified draining time.

e)   Set the meter readout indicator to zero.

f)   Flow a quantity of liquid through the meter into the reference proving tank so that the liquid level can be read in the neck. Record the temperature and pressure at the meter and the temperature of the liquid in the proving tank. Record the observed volumes of both the meter and proving tank. Calculate the meter factor.

g)   Repeat the procedure for a minimum of five determinations of meter factor. The tests are repeated until the repeatability meets the agreed acceptance criteria. This can be five consecutive values falling within a range (often specified as 0,02 %) or a repeatability based on uncertainty. Calculate the mean meter factor or error of the acceptable test runs.

h)   Direct the flow from the reference proving tank to the prover tank under test. Purge all air from the connection lines. Fill the prover tank and allow it to drain for the specified time.

i)   Zero or read the flowmeter indicator. Fill the prover tank at the same flowrate as that used to calibrate the meter until the liquid level is observed in the gauge glass. Read the pressure and temperature at the meter during the fill. When the level is seen in the sight gauge, stop the flow and read the meter, the volume from the gauge glass and the temperature in the prover tank. Drain the tank for the specified time.

j)   This procedure should be repeated until the required repeatability in the calculated volume error falls within the required acceptance criteria. This can be five consecutive determinations of base volume falling within a specified range (usually 0,02 %) or within a specified uncertainty.

k)   As confirmation, the meter is re-proved after the calibration. The difference between the meter factor before the calibration shall be within specification (usually 0,02 %). It is good practice to then recalculate the prover tank volume based on the mean of the two derived meter factors.

l)   Calculate the mean error of the nominal volume mark.

9.6   Calibration of neck scales

The neck scale can be calibrated after the tank is filled and stable and can be best carried out as part of the initial fill.

Neck scales can be verified or calibrated for volumes equal to the major scale marks or simply at the highest, nominal and lowest marks.

The measure is filled to the bottom of the gauge scale. Water is added through the neck from a calibrated measure, flask or weighing tank until the level in the gauge glass reaches the required mark. The volume added is compared with the difference between the levels.

The process is repeated for each chosen mark.

This procedure can be accomplished in the same way by filling the measure to the highest scale mark then withdrawing water into calibrated measures, flasks or the weighing tank. Withdrawal method is particularly suitable for measures with a drain at the bottom of the neck or a small drain valve at the bottom of the measure.

A bottom neck scale is calibrated in the same way.

10 Calculations

10.1 Overview

The calculations are to provide the calibrated standard volume of a measure using a gravimetric reference, a reference measure or reference flowmeter. They also provide for calculating the volume measured by a device under test, including a displacement prover, using a measure as a reference.

The calculations can be broken down into three distinct steps: the determination of the volume as measured by the reference device or measure, the determination of the volume passed to the device under test and finally the volume of the device under test (measure or pipe prover) expressed as volume at standard conditions. If the device under test is a flowmeter this final step is not required.

The volume determined can then be used to calculate the error or performance indicator of the device under test, to establish the standard calibrated volume or to adjust the scale.

In a number of industry sectors and standards, for example ISO 4267-2, the calculation procedures have been combined into a single calculation formula and these are given in 10.7, 10.8 and 10.9.

10.2 Reference volume

The reference volume is the actual volume as measured by the reference device. The reference device may be a volumetric measure or a flowmeter. Volume as measured by a gravimetric method is discussed in 9.3 and the calculation in 10.4.

For a flowmeter, the reading from the meter (V_m) is recorded then corrected for any predetermined fluid property effects, error or meter factor to give the reference volume. If the meter has a pulsed output, the K-factor and the collected pulses provide the reference volume. It is generally not recommended to further correct this volume for temperature or pressure effects on the meter body unless this correction has been established by testing. The reference volume (V_r) is therefore the measured volume or, optionally but not recommended, the measured volume further corrected for predetermined meter body expansions.

For a volumetric reference measure, the volume indicated (V_{rs}) denotes the volume corrected for any known scale adjustments or errors. As this is the volume at standard conditions it requires an additional correction by multiplying by the factor C_{tsr} to give the volume at the measure temperature as shown in Formula (1) with the correction shown in Formula (2).

$$V_r = V_{rs} \times C_{tsr} \tag{1}$$

where V_{rs} is the volume at standard conditions as indicted by the reference measure after correction for errors determined by calibration.

$$C_{tsr} = 1 + 3\alpha_r (t_r - t_{sr}) \tag{2}$$

where

α_r is the linear expansion coefficient of the reference device (see Annex A);

t_r is the temperature of the liquid within the reference measure;

t_{sr} is the standard temperature of the reference measure.

NOTE   It is possible that the standard temperature and materials of the reference measure and the device under test are different.

10.3 Transferred volume (volumetric method)

When the reference volume is transferred from the reference (measure or flowmeter) to the device under test, it can expand or contract due to changes in temperature and pressure.

The transferred volume is calculated from Formula (3).

$$V_t = V_r \times C_{dtl} \times C_{pl} \tag{3}$$

where

V_r is the volume measured by the reference at the actual conditions of pressure and temperature;

C_{pl} is the volume correction factor for liquid compressibility from measured pressure to standard pressure. This correction is only required when the reference is a flowmeter operating at a pressure significantly higher than atmospheric pressure;

C_{dtl} is the volume correction factor for thermal expansion of liquid from the reference device to the device under test, i.e. expansion due to the temperature difference.

C_{dtl} may be calculated in one of two ways:

If the density of the liquid can be calculated directly from temperature, for example if water is the test liquid, C_{dtl} is the ratio of the densities calculated at each temperature as shown in Formula (4).

$$C_{dtl} = \frac{\rho_r}{\rho_t} \tag{4}$$

where

ρ_r is the density of the liquid at the reference device at temperature, t_r;

ρ_t is the density of the liquid at the device under test at temperature t_t;

t_r is temperature of the reference device;

t_t is the temperature of the device under test;

NOTE   Annex A provides formulae relating density to temperature for water.

Alternatively, if the density cannot be calculated directly from temperature, but formulae are available to give the volume correction factor from a given temperature to a standard temperature, volume at the first temperature can be converted to volume at standard temperature then back to volume at the second temperature using Formula (5).

$$C_{dtl} = \frac{C_{tlr}}{C_{tlt}} \tag{5}$$

where

C_{tlr} is the volume correction factor for thermal expansion of liquid from the reference device temperature to standard temperature;

C_{tlt} is the volume correction factor for thermal expansion of liquid from the test device temperature to standard temperature.

The standard temperatures used should be the same and should be that of the device under test.

When a flowmeter or a pipe prover is the device under test or the reference, the pressure can be significantly higher than the atmospheric pressure in a measure. A correction for the compressibility of the liquid should therefore be made by applying a correction factor C_{pl} calculated using Formula (6).

$$C_{\text{pl}} = 1 - (\beta \times P) \tag{6}$$

where

β is the isothermal compressibility of the liquid, usually at atmospheric pressure;

P is the pressure.

Isothermal compressibility for water and hydrocarbons are given in Annex A. Care is to be taken to ensure the units of pressure and compressibility are the same and liquid property data references give compressibility in many different units.

10.4 Transferred volume (gravimetric method)

Calculation of the transferred volume by the gravimetric method requires the density of the liquid to be known. For water, the formulae given in Annex A should be used to calculate density from the measured temperature. If another liquid, for example hydrocarbon oil, is used, the density at the test temperatures can be either measured, a bespoke formulae used or the correction factor C_{tl} as described in Annex A used.

The measured weight is corrected for any known error in the weighing and then the mass of water is calculated by correcting for the air buoyancy as shown in Formula (7).

$$M = W_{\text{a}} + W_{\text{a}} \times \rho_{\text{a}} \times \left(\frac{1}{\rho_{\text{t}}} - \frac{1}{\rho_{\text{w}}} \right) \tag{7}$$

where

M is the mass of liquid collected (kg);

W_{a} is the indicted weight of the liquid collected (kg);

ρ_{a} is the density of air, calculated from the formula given in Annex A (kg/m^3);

ρ_{t} is the density of the liquid in the device under test (kg/m^3);

ρ_{w} is the conventional density of the weights used to calibrate the weighing machine (8 000 kg/m^3).

In theory, ρ_{t} should be the density of the liquid in the weighing container. In practice, utilizing the density at the temperature in the measure under test does not provide significant error unless the very highest accuracy is required.

The volume of water transferred is given by Formula (8).

$$V_t = \frac{M}{\rho_t} \qquad (8)$$

where

$\quad M \quad$ is the mass of water collected in the weighing tank (kg);

$\quad \rho_t \quad$ is density of liquid based on the temperature in the device (measure) being calibrated (kg/m^3).

10.5 Calibrated volume of test device

When using a measure to calibrate a flowmeter, the transferred volume is the reference volume used to determine the performance indicator of the meter.

When calibrating a volume measure or pipe prover the calibrated volume has to be derived. This is the volume at the chosen standard condition.

The volume relating to the volume indicated by the device under test is calculated from Formula (9).

$$V_{rs} = V_t \cdot \frac{1}{C_{tst}} \cdot \frac{1}{C_{ps}} \qquad (9)$$

where

$\quad V_t \quad$ is the volume of liquid transferred to, or from, a reference to the device under test;

$\quad C_{tst} \quad$ is the volume correction factor for the thermal expansion of the device under test (material) to the volume at standard conditions;

$\quad C_{ps} \quad$ is the volume correction factor for the pressure expansion of the device to the volume at standard conditions.

The thermal expansion correction factor is calculated from Formula (10)

$$C_{tst} = 1 + 3\,\alpha_t \left(t_t - t_{st} \right) \qquad (10)$$

where

$\quad \alpha_t \quad$ is linear expansion coefficient of the device under test (see Annex A);

$\quad t_t \quad$ is the temperature of the liquid in the device under test;

$\quad t_{st} \quad$ is the standard temperature of the device under test.

NOTE 1   It is possible that the standard temperature and materials of the reference and the device under test can be different.

For the calibration of a small volume prover with an external rod, the thermal expansion of the prover is calculated using Formula (11).

$$C_{tst} = 1 + \left(2\alpha_t \left(t_t - t_{st} \right) + \alpha_e \left(t_{te} - t_{st} \right) \right) \qquad (11)$$

where

α_t is linear expansion coefficient of the prover body (see [Annex A](#));

α_e is linear expansion coefficient of the external rod material (see [Annex A](#));

t_t is the temperature of the liquid in the prover body (liquid temperature);

t_{te} is the temperature of the external rod;

t_{st} is the standard temperature.

When the device under test is at a pressure significantly higher than atmospheric pressure and the calibrated volume has to be expressed at the standard condition of 101 325 Pa, or another defined standard pressure, the expansion of the device body has to be allowed for. Within the scope of this document this applies to the calibration of pipe provers using a volumetric measure as a reference.

The correction factor, C_{ps}, for the expansion of the device body due to pressure is given by [Formula (12)](#).

$$C_{ps} = 1 + \frac{p}{E} \times \frac{D}{w} \tag{12}$$

where

p is the gauge pressure (bar);

E is the modulus of elasticity of the body material (e.g. most steels are approximately $2,1 \times 10^6$ bar);

D is the pipe prover internal diameter (mm);

w is the pipe prover wall thickness (mm).

10.6 Multiple fills

When a calibration requires more than one pour or fill using one or more reference measures, the volume should be the sum of each corrected. The calculation cannot be carried out until all the pours are completed as it is only then that the temperature of the device under test is determined by summation as shown in [Formula (13)](#).

$$V_{ct} = \Sigma \left(V_t \right) \tag{13}$$

10.7 Calibration of a measure using a reference measure

When calibrating a measure using another measure as reference and water as the liquid, the previous calculations can be combined into a single formula given in two forms in [Formula (14)](#).

$$V_{ts} = V_{rs} \times \frac{C_{dtl} \cdot C_{tsr}}{C_{tst}} = V_{rs} \times \frac{\rho_r \times \left[1 + 3\alpha_r \left(t_r - t_{sr} \right) \right]}{\rho_t \times \left[1 + 3\alpha_t \left(t_t - t_{st} \right) \right]} \tag{14}$$

where

α is linear expansion coefficient of the relevant measure material;

t_r is the temperature of the liquid in the reference measure;

t_t is the temperature of the liquid in the measure under test;

t_{sr} is the standard or base temperature of the reference measure;

t_{st} is the standard or base temperature of the measure under test;

ρ_r is the density of the liquid in reference device at t_r;

ρ_t is the density of the liquid in device under test at t_t.

10.8 Calibration of a flowmeter using a measure as reference

To calibrate a flowmeter which can be at a pressure higher than atmospheric pressure and using a volume measure as reference, the meter factor can be derived at the conditions of temperature and pressure of the meter under test. The calculation is shown in Formula (15).

$$F = \frac{V_t}{V_m} = \frac{V_{rs}}{V_m} \times C_{tsr} \times \frac{C_{tlr}}{C_{tlt}} \times C_{pl} \tag{15}$$

In this case the reference is the measure or proving tank and the device under test is the flowmeter.

To calibrate a volumetric measure using a pre-calibrated flowmeter at pressure, Formula (16) gives the calibrated volume of the measure at standard conditions.

$$V_{ts} = F \times V_m \times C_{tsr} \times \frac{C_{tlr}}{C_{tlt}} \times \frac{1}{C_{pl}} \tag{16}$$

In this case, the reference is the flowmeter and the device under test is the volume measure (proving tank).

10.9 Calibration of a displacement (pipe) prover using a measure as reference

To calibrate a displacement or pipe prover, including small volume provers, at pressure against a reference measure, Formula (17) gives the calibrated volume of the prover at standard conditions.

$$V_{ts} = V_{rs} \times \frac{C_{dtl} \times C_{tsr}}{C_{tst}} \times \frac{1}{C_{pl} \times C_{pst}} \tag{17}$$

11 Calibrating and setting the neck and scale

11.1 Calibrating the neck

The neck scale should be calibrated as part of an initial calibration and thereafter at intervals specified by the application. This is often specified as every fifth measure calibration.

A neck scale calibration should cover the length of the scale with a minimum of six intervals being calibrated.

Verifications can be carried out as part of every calibration and will usually only verify two intervals. Verification can be carried out by ensuring one tank fill of a calibration uses the highest part of the scale and one tank fill the lowest part. A significant difference in the volume error indicates a possible error in the scale.

As the calibration of a neck scale is dependent on the size and volume of the measure, a detailed procedure for the location and access to reference equipment should be prepared for individual calibrations. A guide to the process is given.

The neck scale can be calibrated by fill or by withdrawal, depending on the measure design and size. Neck scales can be verified or calibrated for volumes equal to the major scale marks or by establishing the volume per unit length of the scale (l/mm).

The reference to calibrate a neck scale may be provided by glass measuring flasks, reference test measures or a gravimetric system. The capacity of the reference should be chosen to reflect the volume between the chosen intervals to be calibrated in the neck. Whereas a volume reference provides calibration intervals fixed by the volume of the reference, a gravimetric reference allows calibration to accurately match to the scale markings.

To calibrate by filling, the measure is filled to the top of the neck and allowed to stabilize in temperature. A water supply is provided and allowed to fill the reference to the desired level and again allowed to stabilize.

The measure is slowly drained until a level in the neck scale is coincident with the lowest major mark. It can be easier to allow the level to drop below this level then add water to the neck until the mark is reached.

Measure the temperature in the reference, refilling if required after removing the thermometer. Empty the reference measure into the neck and allow it to drain for the specified time. Read the volume registered on the neck scale and/or measure the distance up from the lowest mark. Calculate the actual volume filled into the measure using the thermal correction factor. If a gravimetric reference is used the fill can be controlled to coincide with the neck scale marks.

The process is repeated at the chosen intervals across the range of the neck scale.

When the last fill is complete the temperature in the measure neck is measured.

The volume errors or the volume per unit length across the scale can then be calculated for each interval.

A similar procedure is adopted if the neck is calibrated by withdrawal. Withdrawal is particularly suitable for larger tanks where there is a small drain either at the bottom of the top or bottom neck which can be utilized to take water to the reference.

The measure would be filled above the top scale mark and the temperature measured. Water is withdrawn until the level is coincident with the top scale mark. Water is then withdrawn into the pre-wetted reference or gravimetric reference. It is noted that only reference measures with a neck and scale can be used making brim measures or automatic pipettes unsuitable. This again suggests withdrawing is best used for larger proving tanks. The process is again repeated for each chosen mark.

A bottom neck scale is calibrated in the same way as described above.

The results of the calibration are examined to identify variability in volume measured across the scale range. A comparison with previous neck calibrations is made to identify any change in the neck volume or scale.

11.2 Setting the scales

The height difference between the nominal volume mark on the scale and the scale datum should be checked and recorded prior to starting a calibration, and again confirmed prior to setting or adjusting a scale. If a scale datum has not previously been identified or defined this can be established at this time and recorded.

NOTE   This value of height difference can be used between calibrations to ensure the scale has not been moved from the previously set position.

From the calibration result, the error or difference between the nominal volume and the calibrated volume, is determined. A temporary mark can be placed next to the scale corresponding to the difference.

The scale can now be adjusted or moved so that the nominal volume mark coincides with the temporary mark. The scale then should be fixed and sealed.

The new height difference between the scale datum and the nominal mark is recorded on the calibration certificate indicting the amount of adjustment carried out.

Errors permissible on the scale compared with the calibration of a volumetric measure should be within 0,01 % of the nominal volume. This should be compatible with the uncertainty (see Annex E).

12 Safety

All operations using a volumetric measure should be aware of applicable international, national and local regulation and safe working practices. In particular the following specific aspects should be noted.

All measures should be designed to be structurally sound and all supports, supporting structures and lifting points designed and inspected to ensure there is no structural failure.

Access platforms should allow safe filling and draining and the reading of scales carried out safely, particularly when working at height.

Steps should be taken to avoid any spillage. All precautions should be taken in advance to plan for the safe containment and recovery of spilled fluids should spillage or overflow occur. This is particularly important if hydrocarbon liquids are being used to prevent any risk of ignition or pollution of water courses.

Where hydrocarbons are being used, consideration of electrical safety and earth bonding is strongly recommended.

If a measure is used for both volatile and non-volatile hydrocarbons, recognition should be given to the danger of creating an explosive gas mixture within the measure when a change of hydrocarbon is undertaken.

A means to allow the safe venting of hydrocarbon vapours from a measure or calibration circuit should be provided.

Any measure carried in or on a vehicle should be operated to prevent any vapour or fumes entering the cab of the vehicle. A measure should be fully vented and purged after use and before being transported.

Annex A
(informative)

Properties of fluids and materials

A.1 General

ISO 8222:2002 provided formulae to calculate the density of water from temperature when calibrating volumetric measures and proving tanks. This standard is referenced and cited by other international, national and regulatory standards along with contract and custody transfer documents. The formulae given within ISO 8222:2002 have been retained in this document where they remain appropriate for use, allowing external references to ISO 8222:2002 and undated references to remain unchanged.

ISO 8222:2002 is also found to be referenced for applications beyond the scope of the document and the formulae are also frequently being referenced for use beyond their applicable range, particularly for use with saline water at high temperatures.

The formulae given in this document provide current internationally accepted formulae for water while retaining reference to those previously referenced, but remain suitable for the applications. Formulae covering the properties of all expected materials and fluids used with volumetric measuring devices have been included, along with formulae for impure and saline water and the industry-standard volume correction factor formulae for hydrocarbon liquids.

A.2 Volumetric thermal correction factor for liquids

The volumetric thermal correction factor is used to correct the volume of liquid at one temperature to the volume which it would occupy at a second temperature due to the thermal expansion.

The volume correction factor for liquid, C_{dtl}, is the ratio of the densities of liquid at temperatures t_1 and t_2. To correct a volume from one temperature to the volume at another, the volume at t_1 is multiplied by C_{dtl} to give the volume at t_2 as shown in Formula (A.1).

$$C_{dtl} = \frac{\rho_1}{\rho_2} \tag{A.1}$$

where

ρ_1 and ρ_2 are densities at t_1 and t_2, respectively;

t_1 and t_2 are the two temperatures.

Formula (A.1) applies to any liquid with the densities being measured or calculated from traceable formulae or equations of state.

Where the volume has to be corrected to the volume at a standard temperature condition, the correction is abbreviated to C_{tl} where t_2 is the defined standard temperature.

A.3 Density of pure (distilled or deionised) water

A.3.1 General

The density of water is not a linear function with temperature; however, the relationship has been well defined for pure water. There are a number of different formulae and the most common are summarized

by Batista and Paton[15]. The International Committee for Weights and Measures (CIPM) currently recommends the Tanaka formula for use at temperatures up to 40 °C and the IAPWS equation of state for applications where the temperature exceeds 40 °C.

A.3.2   Tanaka formula

Following publication in 2001, the formula for the density of pure water accepted by the CIPM for use in metrology is that of Tanaka et al.[16]. This is the recommended formula to be used when calibrating volume measures, see Formula (A.2).

$$\rho_{\text{Tanaka}} = a_0 \times \left[1 - \frac{(t+a_1)^2 (t+a_2)}{a_3 (t+a_4)} \right] \tag{A.2}$$

where

ρ_{Tanaka} is density of pure water from Tanaka formula (kg/m^3);

t is temperature (°C).

and the coefficients are:

a_0 = 999,974 950 a_1 = −3,983 035 a_2 = 301,797 a_3 = 522 528,9 a_4 = 69,348 81

This formula is for pure water and a range of validity for temperatures of 0 °C to 40 °C and atmospheric pressure.

A.3.3   Patterson and Morris formula (ISO 8222:2002)

Formally recommended by CIPM, and given in ISO 8222:2002, the formula proposed by Patterson and Morris[17] is extensively referenced. To give consistency with older standards this formula has been reproduced as Formula (A.3).

$$\rho_{\text{P\&M}} = \rho_0 \left\{ 1 - [c_1 (t-t_0) + c_2 (t-t_0)^2 + c_3 (t-t_0)^3 + c_4 (t-t_0)^4 + c_5 (t-t_0)^5] \right\} \tag{A.3}$$

where

$\rho_{\text{P\&M}}$ is density of pure water from the Patterson and Morris formula (kg/m^3);

t is temperature (°C);

t_0 is temperature of maximum density (°C);

t_0 = 3,981 8;

ρ_0 is the maximum density of water (kg/m^3);

ρ_0 = 999,973 58;

c_1 = 7,013 4 × 10^{-8};

c_2 = 7,926 504 × 10^{-6};

c_3 = −7,575 677 × 10^{-8};

c_4 = 7,314 894 × 10^{-10};

c_5 = −3,596 458 × 10^{-12}.

This formula is specified to have a range of validity for pure water at temperatures of 0 °C to 40 °C and at atmospheric pressure.

For the calibration of volumetric measuring devices the difference between Tanaka and Patterson and Morris is unlikely to be significant except at the lowest uncertainty levels.

A.3.4 IAPWS 95 equation of state

The Tanaka and the Patterson and Morris formulae are specified for use at temperatures up to 40 °C. Extrapolating to 80 °C, the Patterson and Morris formula gives an error in the density of water of around 0,04 %, while the Tanaka formula is in error by around 0,01 %.

For applications where the temperature is between 0 °C and 100 °C, CIPM recommends the International Association for the Properties of Water and Steam (IAPWS) formulation. IAPWS provides two equations of state, IAPWS releases 1995 and 1997, the release 95[18] being advised for metrology. CIPM recognizes the IAPWS–R6-95[18] formulation as equivalent to Tanaka in the temperature range up to 40 °C and recommends its use if the application has a temperature exceeding 40 °C.

IAPWS 95[18] formulation is an equation of state for the water substance and is a complex formula which includes many thermodynamic properties, including compressibility. The formulation is available from a number of sources as computer implementation code which can be used where practicable.

Where the full implementation is unsuitable, or unavailable, a rational polynomial formula, Formula (A.4), has been fitted to values calculated from the IAPWS equation of state at atmospheric pressure. This was proposed by Batista and Paton[15]. This formula is specified to have a range of validity from 0 °C to 85 °C at atmospheric pressure. It reproduces the IAPWS calculated values to within 0,001 kg/m^3 at temperatures of 1 °C to 90 °C, rising to 0,002 kg/m^3 at 95 °C.

$$\rho_{IAPWS} = c_0 \cdot \left(\frac{1 + c_1\, t_n + c_2\, t_n^2 + c_3\, t_n^3}{1 + c_4\, t_n + c_5\, t_n^2} \right) \tag{A.4}$$

where

ρ_{IAPWS} density of pure water from equation fit to IAPWS 95 (kg/m^3);

t_n is normalized temperature, $t/100$;

t is temperature (°C);

c_0 = 999,843 82;

c_1 = 1,463 938 6;

c_2 = −0,015 505;

c_3 = −0,030 977 7;

c_4 = 1,457 209 9;

c_5 = 0,064 893 1.

A.3.5 Correction for air-saturated water and isotopic composition

Variations in isotopic composition provide variations in density; however, this is not significant for the purposes of volumetric measurement.

If the water used is air-saturated, the density is lower and hence a correction could be applied. The correction is small and determining if the water is fully or only partially saturated is difficult. As a result, the correction is not generally recommended for anything other than laboratory primary calibrations and inter-comparisons.

Where a correction is required, different formulae have been summarized by Batista and Paton[15]. The correction by Bignell is usually acceptable where a correction is considered to be required, see Formula (A.5).

$$\rho_c = -(4,612 - 0,106\,t) \times 10^{-3} \tag{A.5}$$

where

ρ_c is the correction to the density (kg/m^3);

t is the temperature of the water (°C).

NOTE   This was derived for water in the temperature range 0 °C to 25 °C. However, extrapolation to 40 °C is acceptable with the correction becoming insignificant above 40 °C.

A.4 Density of impure and saline water

A.4.1 Description of impure water

It is assumed that any water has been filtered to remove solid impurities to a level which is insignificant. For applications involving volumetric measures, filtration to < 20 micron is recommended.

Ideally pure water, distilled, deionised or otherwise treated, is used for volume calibrations where practicable. While initially specifying pure water to be to a standard, for example ISO water quality grade 3 as specified in ISO 3696, in practice this quickly degrades from the purity specified when used for volume measurement. For the purposes of volumetric metrology pure water will include some level of impurity. This may be measured by salinity or conductivity up to the level classified as pure shown in Table A.1. This is a purity level coincident with the best-quality drinking water and allows for insignificant error to be introduced when using the pure water density formulae.

Impure water is more commonly used in all but the highest-level laboratory calibrations. Impure water ranges from good-quality drinking water with small impurity levels through to poor-quality drinking water and extends to include brackish and seawater, i.e. salinity range 0,5 to 40 g/kg.

Although volumetric measurements in brine and at temperatures above 40 °C is discouraged, the definition and properties of impure water have been extended to cover a salinity range of 0 g/kg to 140 g/kg and a temperature range of 5 °C to 90 °C (extended to 0 °C to 100 °C) in this annex. This allows this document to cover applications where ISO 8222:2002 was previously being referenced outside the scope and range of applicability.

Impurities are predominately dissolved salts, but can also contain other dissolved materials such as biological contaminants, hydrocarbons or non-ionic materials. They can also include small amounts of fine solids in suspension.

In this document, seawater composition is considered to provide the best descriptor for impurity on which property formulae can be defined. Reference seawater has been defined and standardized in terms of composition ratio as described by Mellero[20]. This also defines the unit of absolute salinity (S_a) as the ratio of the mass of all impurities to the mass of the water. The normal units used are g/kg.

Other definitions of salinity were used prior to 2008, particularly the unit of practical salinity (S_p), which is based on conductivity. Confirmation is required to ensure the correct value is being used.

NOTE   An alternative measure of impurity, mainly used to describe fresh water, is Total (Dissolved) Solids (TDS or TS), being the ratio of the mass of impurity to a volume of water. The normal units used are mg/l. Knowledge of the density of the water at a given temperature allows conversion to salinity.

Salinity, and thus density, can be measured by electrical conductivity with the unit Siemens per metre (S/m) but mS/cm or µS/cm are more commonly used. Conductivity is a function of temperature and is by default usually quoted as specific conductivity at 25 °C. Conductivity is also a function of the ratio of

anion and cation concentrations and does not recognize any non-conductive components. Due to these variables, conductivity is therefore not recommended as a definitive measure to calculate density or salinity unless the water conductivity has been characterized. Conductivity does, however, provide the most sensitive method of indicating purity.

Salinity can be measured by evaporation of a water sample, following specific procedures or by determining the chemical (mol ratio) composition.

Relative density, the ratio of the density of the water to the density of reference water at defined temperature(s), refers to the ratio at the same temperature in this document. Relative density can be used as a measure or estimate of salinity and is calculated using Formula (A.6).

$$\rho_{RD} = \frac{\rho_s}{\rho_{rw}}$$ (A.6)

where

ρ_s is the measured density of the sample at the given temperature;

ρ_{rw} is the calculated density of a reference water at a given temperature.

The given temperature is normally the standard temperature, i.e. 15 °C, for most hydrocarbon industry applications. Reference water is pure water unless otherwise specified.

NOTE   Subscripts to RD are used to indicate alternative temperatures of determination. Normally a standard temperature (15 °C, 20 °C, 25 °C, 27 °C or 60 °F) is used. An additional subscript, 'sw', indicates density of reference seawater rather than density of pure water as the reference.

To assist in describing impure water, it can be classified. Classification is not standardized across industries. Table A.1 shows descriptive terms for different water qualities used in this document along with approximate values for the maximum measurements characterizing each category.

Table A.1 — Water categories with increasing impurity

Water category description			Maximum salinity g/kg	Conductivity @15 °C µS/cm	Relative density @ 15 °C	Error in converting density from 15 °C to 40 °C %
Fresh	Pure	Distilled or deionised	< 0,05	< 50	< 1,000 04	0,000 1
Fresh	Potable or drinking quality	Good quality	0,5	800	1,000 39	0,001
Fresh	Potable or drinking quality	Poor quality	1	1 000	1,000 77	0,003
Saline	Brackish	Slightly saline	5	2 000	1,038 4	0,014
Saline	Brackish	Very saline	20	5 000	1,015 28	0,054
Saline	Seawater	Ref. at 35 g/kg	40	40 000	1,030 62	0,103
Saline	Brine	Produced water	120	150 000	1,093 26	0,22

Conductivity has been given at a temperature of 15 °C and not at 25 °C, which is the more common standard temperature quoted. This, however, gives consistency with standard conditions used in the petroleum industry.

Pure water has been given an upper limit for salinity which reflects insignificant error when using pure water density functions and is significantly higher than that specified for pure water for laboratory use.

The last column in Table A.1 describes the error found if pure water formula is used to correct the density of impure water from 40 °C to 15 °C. This is further explained in A.4.6.

A.4.2 Formulae for density of impure water at atmospheric pressure

Reference seawater[20] is defined in terms of the ratio of the impurities and the concentration of the impurity (salinity).

The definitive equation of state for reference seawater was released as IAPWS-R13-08[21] and fully adopted as the Thermodynamic Equation Of Seawater 2010 (TEOS-10)[22] by UNESCO-IOC in 2009. The open-source code for TEOS-10 is available in FORTRAN and VB6 and includes software implementations of the IAPWS-R13 formulation. It includes conversion software to convert salinity units, conductivity and some other functions. These functions have been taken from a previous seawater standard issued by UNESCO as EOS-80[23] and are still available separately from TEOS[24].

TEOS-10 currently provides the most comprehensive and theoretically based equation of state for seawater and is fully compatible with IAPWS 95 for pure water. Density, as a function of temperature and pressure, can be predicted with an uncertainty within 0,001 % across the range 0 °C to 40 °C and 0 °C to 40 g/kg. Outside this range the equations have been extrapolated and show error in density of around 2 % at 80 °C and salinity 120 g/kg. An extension to the basic formula has been provided by Feistal[25]. This reduces the error to an uncertainty of 0,02 % at 90 °C and 120 g/kg. The extension is not available in the IAPWS implementation; however, it is included in the TEOS documentation but requires to be enabled in TEOS computer code.

Where it is impractical to implement the full TEOS code, a rational formula has been generated from the TEOS-10 formula and proposed by Batista and Paton[15]. This covers the temperature range 5 °C to 90 °C and salinity from 0 g/kg to 120 g/kg and at pressure of 101 325 Pa. The density of reference seawater is expressed in Formula (A.7).

$$\rho = \frac{a + b \cdot t + c \cdot S_a + d \cdot S_a{}^2}{1 + e \cdot t + f \cdot t^2 + g \cdot S_a + h \cdot S_a{}^2} \qquad (A.7)$$

where

ρ is density (kg/m^3);

t is temperature (°C);

S_a is salinity (absolute) (g/kg);

a = 999,952 012 7;

b = 4,766 828 864;

c = 7,980 982 355;

d = 3,906 984 × 10^{-3};

e = 4,726 51 × 10^{-3};

f = 6,765 82 × 10^{-6};

g = 7,172 015 × 10^{-3};

h = −1,653 2 × 10^{-6}.

Agreement with the TEOS-10 formulation is within ± 0,005 % in the range 10 °C to 40 °C and salinities 0 g/kg to 40 g/kg. Agreement across the wider range, 5 °C to 90 °C and 0 g/kg to 120 g/kg is within ± 0,01 %.

Extrapolation to 0 °C and 100 °C and salinities above 120 g/kg can incur increased uncertainty.

TEOS-10 has been formulated for reference seawater. There is additional uncertainty introduced through variations in the nature and ratio of impurities compared with that of reference seawater. This

additional uncertainty can be minimized through a direct measurement of density of the water from a sample and the resultant relative density (to that of reference seawater), used to determine and correct the density.

A.4.3 Thermal expansion correction factor

It is assumed that the thermal expansion of any commonly used saline water is not significantly different from that of reference seawater across the full salinity range.

Thermal expansion factor can be calculated from the density of the water at the two relevant temperatures as given in Formula (A.2).

An alternative formula relating thermal volumetric expansion correction factor, C_{tl15} to temperature and salinity is given in API MPMS Chapter 20.1, Annex A[26]. This is the volume (or density) correction factor from the measured temperature to 15 °C and given in Formula (A.8). The original source of this formula is not known.

When compared with values of C_{tl15} calculated from TEOS-10, agreement within ± 0,1 % in the range 15 °C to 40 °C and salinities of 0 g/kg to 40 g/kg is found. This rises to show a difference of up to 0,3 % in the range 15 °C to 90 °C and 0 g/kg to 120 g/kg.

The agreement shows the form of the formula is valid and, by utilizing the TEOS 10 formula, the coefficients given for the API MPMS Chapter 20.1 formula have been modified. This now shows agreement with TEOS-10 within ± 0,02 % in the range 10 °C to 40 °C and 0 g/kg to 40 g/kg, and within ± 0,03 % across the range 5 °C to 90 °C and 0 g/kg to 120 g/kg.

$$C_{tl15} = 1 - (a + b \cdot S_a) \cdot \Delta t - (c - d \cdot S_a) \cdot \Delta t^2 + (e - f \cdot S_a) \cdot \Delta t^3 \tag{A.8}$$

where

C_{tl15} is the volume correction factor to 15 °C;

S_a is absolute salinity (g/kg);

t is temperature (°C);

Δt is the temperature difference from standard temperature (t–15) °C.

The coefficients are as follows:

Coefficient	Original coefficients for C_{tl15} from API 20.1 1995[a]	Revised coefficients for C_{tl15} from API 20.1 and TEOS-10
a	$1{,}856\ 2 \times 10^{-4}$	$1{,}615\ 6 \times 10^{-4}$
b	$1{,}288\ 2 \times 10^{-6}$	$1{,}249\ 8 \times 10^{-6}$
c	$4{,}115\ 1 \times 10^{-6}$	$5{,}022\ 7 \times 10^{-6}$
d	$1{,}446\ 4 \times 10^{-8}$	$2{,}070\ 7 \times 10^{-8}$
e	$7{,}192\ 6 \times 10^{-9}$	$1{,}539\ 0 \times 10^{-8}$
f	$1{,}308\ 5 \times 10^{-11}$	$7{,}340\ 5 \times 10^{-11}$

[a] API MPMS chapter 20.1 gives the coefficients based on salinity units of 'weight ratio %'. The coefficients given have been modified to match the salinity unit g/kg. The coefficients for C_{tl60F} are also given in MPMS 20.1.

A.4.4 Other referenced formulae

API MPMS chapter 4.9.1[9] has an algorithm for density as a function of conductivity. Being a formula derived from now superseded seawater formulae, and then applying it to both fresh and impure water, the use of this formula is not recommended.

Similarly, the formulae for use with saline water and brine which were proposed by MacGillivray et al.[27], having been proposed before the release of the IAPWS and TEAOS formulae, are not now recommended

A.4.5 Salinity from relative density

To use the formulae in the previous subclauses, a measurement of salinity is required. If this is not available, a relative density, relative to the density of pure water, can be estimated.

Formula (A.9) has been proposed by Batista and Paton[15]. Using the density values of reference seawater calculated from TEOS-10 at 15 °C and the density of pure water, the relative density is calculated across a salinity range of 0 g/kg to 120 g/kg. The formula to calculate salinity has then be derived.

$$S_a = \left(-0,025\,98 + 1\,316,498 \times (\rho_{RD} - 1) - 321,408\,18 \times (\rho_{RD} - 1)^2\right) \tag{A.9}$$

where

S_a is salinity (g/kg);

ρ_{RD} is relative density, relative to pure water at 15 °C.

This provides a reasonable estimate of salinity in the range 0 g/kg to 120 g/kg where the density of the saline water has been measured at 15 °C.

A.4.6 Application of impure water formulae

The formulae referenced have been compiled for reference seawater. This has a standardized composition ratio for defined salts and impurities. Where the water used has different impurities, additional impurities or a different impurity ratio, the formulae might not provide the uncertainty in density required for gravimetric calibrations or volume-to-mass calculations. In this situation the density calculated from the formulae should be corrected through application of a measured relative density (relative to reference seawater).

Density of a water sample can be measured with uncertainty better than 0,010 kg/m^3 using high-quality commercially available laboratory instruments and to 0,1 kg/m^3 using hand-held vibrating tube density meters suitable for field and industrial determinations.

If the formulae relating reference seawater density to temperature are used, and an uncertainty in density of < 0,005 % is required, salinity has to be measured to an uncertainty of less than 0,1 g/kg.

If, however, the formulae are used to calculate a volume expansion factor, and not absolute density, they may be used without correction for all but the most accurate applications. In this case, uncertainty in salinity only requires to be measured with an uncertainty of less than 5 g/kg.

The practice of using pure water density formulae to calculate volume expansion factors for impure water is discouraged. There is a significant relationship between C_{tl} and salinity which gives rise to increasing error in density as salinity and temperature increase. Tl errors introduced are shown in Table A.1, based on correcting density from 40 °C to 15 °C.

For some applications, however, this error can be acceptable and insignificant within the uncertainty required.

For drinking-quality water and at temperatures up to 40 °C the potential error found by using pure water formulae does not exceed 0,003 %. This suggests that pure water formulae may be acceptable for most applications using and calibrating volumetric measures. For seawater this error rises to > 0,1 %; for brine, found in produced water applications, the error introduced is around 0,2 % at 40 °C but rises to 3 % at 90 °C.

A.5   Density of hydrocarbons

The density of hydrocarbons approximates to a linear function with temperature and is significantly dependant on the composition. For a hydrocarbon liquid, a sample can be taken and the density at base conditions measured. It is also possible to characterize the density by measuring across a range of temperatures. For most industrial purposes the thermal expansion coefficient can be derived from the formulae provided in ISO 91. Alternatively, a specific hydrocarbon liquid can be characterized against temperature and the results used to generate the constants used in the formulae given in ISO 91.

A.6   Liquid compressibility correction factor

A.6.1   General

Liquid compressibility is not a significant factor in the calibration of volume tanks at atmospheric pressure. Where a flowmeter, pipe prover or pressurized volume measure is at an elevated pressure, the liquid is subject to compression and a volume or density correction is required.

The liquid compressibility correction factor, C_{pl}, is given by Formula (A.10):

$$C_{pl} = \frac{1}{(1 - \beta \cdot P)}$$
(A.10)

where

   β is the isothermal secant compressibility of the liquid. (bar^{-1});

   P is the pressure of the liquid, expressed in bar ($Pa \times 10^5$).

A.6.2   Pure water compressibility

Pure water compressibility (β) is discussed by Batista and Paton and three formulae are identified. Compressibility can be determined with the lowest uncertainty from the IAPWS R6-95[18] formulation.

The simpler formula is found in a number of different standards issued by both API and the energy institute (EI). This is an unattributed polynomial relating compressibility at atmospheric pressure to temperature and is given in Formula (A.11).

$$\beta = \frac{1}{\left(19{,}69 + 0{,}141\,8 \cdot t - 1{,}934 \times 10^{-3} \cdot t^2 + 5{,}866 \times 10^{-6} \cdot t^3\right)} \times 10^{-3}$$
(A.11)

where

   β is isothermal compressibility at atmospheric pressure (bar^{-1});

   t is temperature (°C).

Comparison with values calculated from the IAPWS-R6-95 formulation shows agreement within 0,2 % across a temperature range of 0 °C to 80 °C, confirming that this formula is suitable for most volumetric measurement purposes.

API MPMS chapter 4.9.1[9] recommends the use of a constant value of $B = 4{,}64 \times 10^{-7}$ kPa^{-1}, (i.e. B at 15 °C). This can be compared with values calculated from IAPWS R6-95 which range between $(4{,}9 \times 10^{-7})$ kPa^{-1} and $(4{,}4 \times 10^{-7})$ kPa^{-1} across the temperature range 5 °C to 90 °C, with the minimum being at 50 °C. This indicates the constant value can be acceptable for most applications with due regard to the required uncertainty for any particular application.

A.6.3 Impure water compressibility

Compressibility of water is affected by the presence of impurities. Compressibility of impure water can be estimated from seawater formula, TEOS-10[22]. While being accurate in the range 0 °C to 40 °C and salinity 0 g/kg to 40 g/kg, there is an increasing extrapolation error above these ranges. The extension to correct thermal density extrapolation does not apply to compressibility.

As reported by Batista and Paton[15], the isothermal compressibility, Beta (β) at atmospheric pressure for pure water taken from TEOS 10 was combined with values from the UNESCO EOS-80[23] and extrapolated to 95 °C and 140 g/kg. A formula was fitted to this data set. While recognizing this is an artificial method of generating a formulation, the resultant curve provides an estimate of the compressibility to within 0,5 % up to 40 °C and 40 g/kg rising to 2 % at 95 °C and 120 g/kg.

Formula (A.12) is therefore proposed for the isothermal compressibility of saline water at atmospheric pressure.

$$\beta_{sw} = \left(a + b \cdot t + c \cdot S + d \cdot t^2 + e \cdot S^2 + f \cdot t \cdot S \right) \tag{A.12}$$

where

β_{sw} is the compressibility of seawater (GPa^{-1});

t is temperature (°C);

S_a is the absolute salinity (g/kg);

a = 0,499 841;

b = −2,408 56 × 10^{-3};

c = −1,162 09 × 10^{-3};

d = 2,350 96 × 10^{-5};

e = 1,222 53 × 10^{-6};

f = 8,385 00 × 10^{-6}.

A.6.4 Oil compressibility

The accepted formula to provide compressibility of oil is given in ISO 9770[1]) and as Formula (A.13).

$$\beta = \text{EXP}(-1,620\,8 + 2,159\,2 \times 10^{-4} \cdot t + 0,870\,96 \cdot \rho_{15}^{-2} + 4,209\,2 \times 10^{-6} \cdot t \cdot \rho_{15}^{-2}) \times 10^{-4} \tag{A.13}$$

where

β is isothermal compressibility of oil at atmospheric pressure (bar^{-1});

t is the temperature, expressed in °C;

ρ_{15} is the density in kg/l, at 15 °C and 1,013 25 bar.

NOTE 1   The density is input as kg/l not kg/m^3 as elsewhere in this document.

NOTE 2   API MPMS chapter 11.1 (2004 onwards) and ISO 91 include the pressure correction within the implementation to provide a combined C_{tl} and C_{pl} correction factor.

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1)   This document has been withdrawn and replaced by ISO 91.

A.7 Shell and pipe materials

The volume of a measure or pipe prover changes with temperature. The volume correction factor for the thermal expansion of the shell material is given by Formula (A.14).

$$C_{ts} = 1 + 3 \cdot \alpha (t_1 - t_2) \tag{A.14}$$

where

t_1 is the actual temperature (°C);

t_2 is the temperature to which the volume is referred (°C), which could be the standard temperature if the correction is to standard conditions;

α is the coefficient of linear expansion of the material (°C^{-1}).

For a volumetric device the material used and the coefficient of expansion should be known and recorded in documentation.

If the material is not known, the coefficients of linear expansion for the two most common materials used can be assumed to be:

Mild steel: 11×10^{-6} °C^{-1}

Stainless steel: 17×10^{-6} °C^{-1}

NOTE   The coefficients of linear expansion for the 300 series of stainless steels varies with grade. Values of between $(14,5 \times 10^{-6})$ °C^{-1} and (18×10^{-6}) °C^{-1} are found. In this document the value (17×10^{-6}) °C^{-1} [cubical expansion (51×10^{-6}) °C^{-1}] has been adopted for consistency with ISO 4269 and API MPMS standards. The errors arising from any deviation from the value for the stainless steel used is unlikely to be significant.

Coefficients of cubical thermal expansion of the common materials used to construct volume measures, provers and flasks are taken from EURAMET cg21[2] and given in Table A.2.

Table A.2 — Coefficient of cubical thermal expansion of construction materials

Standard measure material	Linear thermal expansion coefficient °C^{-1}	Cubical thermal expansion coefficient °C^{-1}
Carbon fibre	$0,3 \times 10^{-6}$	1×10^{-6}
Borosilicate glass 3.3	$3,3 \times 10^{-6}$	$9,9 \times 10^{-6}$
Borosilicate glass 5.0	5×10^{-6}	15×10^{-6}
Soda-lime glass	9×10^{-6}	27×10^{-6}
Stainless steel (410)	10×10^{-6}	30×10^{-6}
Mild steel	11×10^{-6}	33×10^{-6}
Stainless steel (316)	16×10^{-6}	48×10^{-6}
Stainless steel (304)	17×10^{-6}	51×10^{-6}
Copper – zinc alloy (brass)	18×10^{-6}	54×10^{-6}
Aluminium	23×10^{-6}	69×10^{-6}

A.8 Density of air

When calibrating gravimetrically, the mass of liquid has to be calculated from the measured weight by applying a buoyancy correction factor.

The buoyancy correction factor, providing a correction of 0,1 % to 0,15 % to the determined volume, is given by Formula (A.15).

$$C_B = 1 + \rho_a \left(\frac{1}{\rho_l} - \frac{1}{\rho_w} \right) \tag{A.15}$$

where

ρ_a is the density of air during weighing (kg/m^3);

ρ_w is 8 000 kg/m^3, i.e. the conventional density of the weights used to calibrate the weighing machine;

ρ_l is the density of the liquid (water) in the weightank (kg/m^3).

For convenience the temperature at the prover or measure may be assumed unless significantly different from that of the weigh tank.

The density of air is dependent on the air temperature, the air pressure and the humidity.

Density of air may be taken as a constant, 1,22 kg/m^3, or determined based on the average conditions of pressure, temperature and humidity for the location. Using a constant is acceptable if the uncertainty has been assessed with regard to the potential variations in conditions at the location.

To give improved uncertainty it is recommended that a more accurate determination of air density is carried out for each calibration.

The formulation accepted and recommended by CIPM is that from Picard et al.[31] and should be used where practicable and for laboratory determinations of mass and volume.

The approximation to the recommended formula is given in OIML R111[32] and in Formula (A.16). It is recommended for use where the CIPM formulation is not employed.

$$\rho_a = \frac{0,348\,48 \times P_a - 0,009 \times H_r \times \text{EXP}(0,061 \times t_a)}{T_a} \tag{A.16}$$

where

ρ_a is density of air (kg/m^3);

P_a is atmospheric pressure (hPa or mbar);

H_r is relative humidity (%);

t_a is temperature (°C);

T_a is absolute temperature (K) = t+273,15.

There are other formulae recommended, for example Spieweck[33] and Jager[34], and their use does not provide a significantly different uncertainty for volumetric calibrations.

A.9 Viscosity of water

Although not required in volumetric measurement, the viscosity of water is often used to define flowrate in terms of Reynolds number.

The viscosity of pure water at atmospheric pressure can be obtained from IAPWS R12-08[35] for the Viscosity of Ordinary Water Substance. However, Harvey et al.[36] provides a simpler formula, Formula (A.17), recognized by IAPWS as supplementary release IAPWS SR6-08[37] for the dynamic viscosity of pure water.

This formula requires input of absolute temperature and returns viscosity in units Pa s.

$$\eta = \sum_{i=1}^{4} a_i \times (T^*)^{b_i}$$ (A.17)

where

H is dynamic viscosity of water (Pa·s);

T^* is = $T/300$ (K);

T is temperature (K).

and the coefficients a_i and b_i are as follows:

i	a_i	b_i
1	280,68	−1,9
2	511,45	−7,7
3	61,131	−19,6
4	0,459 03	−40,0

The range of applicability is 0 °C to 110 °C (273,15 K < T < 383,15 K) and pressures up to 0,3 MPa. It should not be extrapolated outside these limits. The uncertainty is stated as 1 % across the range. Within the range 15 °C to 40 °C the formula agrees with the ISO/TR 3666 values within the stated uncertainty of 0,17 %, and so it is not unreasonable to assume an uncertainty of 0,2 % over the range 10 °C to 40 °C and 0,5 % between 5 °C and 80 °C.

Viscosity is also a function of pressure. The effect can be determined from the IAPWS-R12-08 viscosity formulation. From 0 °C to 25 °C the change in viscosity with pressure is insignificant. From 30 °C to 80 °C, an increase in viscosity of up to 6 % is observed at a pressure of 100 MPa. ISO/TR 3666 gives coefficients for pressure correction at the fixed points between 15 °C and 40 °C. These vary from $−6,14 \times 10^{-4}$ at 15 °C to $−1,22 \times 10^{-4}$ at 30 °C, rising to $+1,2 \times 10^{-4}$ at 40 °C with units of MPa^{-1}. This increase can also be considered as being insignificant for most flow and volume measurements where the highest pressures normally observed are around 1 MPa to 2 MPa. Further considerations would, however, have to be made if higher pressures were to require a lower uncertainty.

For seawater, Sharqawy et al.[39] reviewed and compared a number of formulae predicting the dynamic viscosity of saline water and showed that viscosity is a function of both salinity and temperature. In this paper a formula for the dynamic viscosity of saline water was proposed, see Formula (A.18).

$$\eta_{sw} = \eta_w \left(1 + A \cdot S_a + B \cdot S_a^2 \right)$$ (A.18)

where

η_{sw} is the viscosity of seawater (Pa s);

S_a is absolute salinity (g/kg);

η_w is the viscosity of pure water (Pa s) at temperature t;

A = $1,541 \times 10^{-3} + 1,998 \times 10^{-5} \times t + 9,52 \times 10^{-8} \times t^2$;

B = $7,974 \times 10^{-6} + 7,561 \times 10^{-8} \times t + 4,724 \times 10^{-10} \times t^2$;

t is the temperature (°C);

S_a is salinity (g/kg).

Sharqawy gives the range of validity of 0 °C to 180 °C and salinity 0 g/kg to 150 g/kg with an uncertainty of 1,5 %.

Annex B
(informative)

Temperature measurement and thermometers

B.1 Thermometers

It is recommended that thermometers are class A, platinum resistance detectors, to IEC 60751:2008[12] specification. These would be connected to a suitable readout. Other options are possible; however, liquid in glass thermometers is discouraged.

Probes should have adequate length to reach to the bottom of a thermowell or have an immersion (in liquid) depth of at least 300 mm if inserted into a neck.

Both probes and readouts should be regularly calibrated along with the associated readout traceable to national standards.

B.2 Thermometer pockets (thermowells)

Where appropriate, thermowells should be fitted protruding into the body of the measure.

Thermowells should be installed at time of manufacture and be fitted by welding or, if removable, be sealed in such a way as not to alter the calibrated volume if disturbed.

Thermowells should be installed to ensure that there is no air or liquid trapped at the entry to measure. They should be angled downward to allow filling with conductive liquid and allow consistent draining of liquid within the measure.

In large proving tanks (capacities greater than 500 l) the water temperature should be measured at more than one location due to possible temperature gradients.

The pockets or thermowells should be arranged so that the sensor within the thermometer probes is evenly distributed volumetrically in the lower, middle and upper zones of the measure.

Each pocket should preferably be arranged so that the sensor within the thermometer coincides with the axis of the measure or not less than 300 mm from the wall of the measure.

B.3 Water temperature

The water temperature should be measured in both the reference and test measures.

When performing calibrations in the laboratory the water temperature shall be stable to within ± 1 °C between fills and as close as possible to ambient air temperature.

For standard capacity measures installed in fixed systems it is recommended that a calibration be carried out on site and within a period so that the water temperature in the measure being calibrated does not vary by more than 2 °C during the filling.

B.4 Ambient conditions and air temperature

During the calibration in a laboratory situation air temperature shall be stable to at least ± 3 °C.

In order to avoid uncontrolled expansions and consequentially changes to the volume, the measures shall be stored in the calibration area for at least 1 h and preferably 6 h before calibration. This is especially important in locations with large temperature differences during the day.

During on-site use exposure to direct solar radiation, wind and rain should be avoided.

Annex C
(informative)

Standard glass contents measures

Standard glass measures are specified in ISO 4788 for graduated cylinders and OIML R43[13] for flasks. The nominal capacity is the volume is contained at 20 °C and read, as described in the standard, with the top of the scale mark tangential to the bottom of the meniscus. Capacities of glass measures are summarized in Tables C.1 and C.2

Table C.1 — Nominal capacity of glass cylinders, class A

Nominal capacity ml	Volume of a subdivision of scale ml	Limits of error ml
5	0,1	0,05
10	0,2	0,1
25	0,5	0,25
50	1	0,5
100	1	0,5
250	2	1,0
500	5	2,5
1 000	10	5,0
2 000	20	10

Table C.2 — Nominal capacity of flasks, class A

| Nominal capacity ml	Limits of error ml
1	0,025
2	0,025
5	0,025
10	0,025
20	0,040
25	0,040
50	0,060
100	0,100
200	0,150
250	0,150
500	0,250
1 000	0,400
2 000	0,600
5 000	1,200

Annex D
(informative)

Meniscus reading

When a water level is established in a site tube or window a meniscus is formed. Meniscus reading is one of the most important contributions to measurement uncertainty in the use and calibration of volumetric measures. Meniscus reading has a big influence on measurement reading and consequently repeatability of the result.

The variability of meniscus setting and scale readings made by a single operator depends upon his or her individual expertise. The formation of a meniscus is dependent on the cleanliness of the sight tube/window and the purity of the water.

A meniscus formed with clean water and a small tube is curved upward. Other liquids do not form a meniscus or have a meniscus curved downward. Impurities in the water reduce the meniscus. For measures with broader necks it can be almost impossible to see the meniscus clearly. If the meniscus cannot be discerned, any potential effect should be included in the uncertainty calculation.

The meniscus shall be set so that the plane of the upper edge of the viewing line is horizontally tangential to the lowest point of the meniscus, the line of sight being in the same plane (Figure D.1).
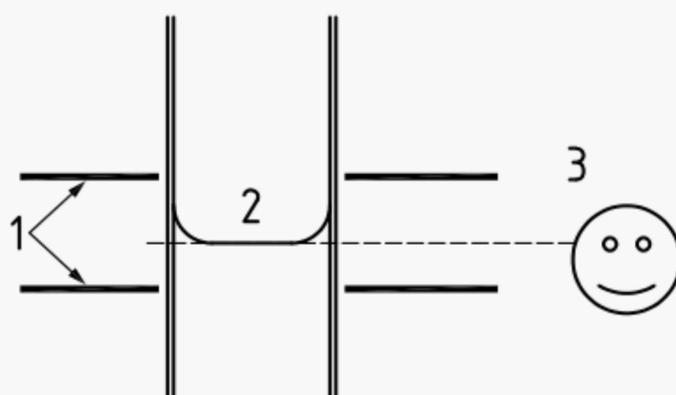
Key

1   graduation lines

2   surface with meniscus

3   align viewing

Figure D.1 — Reading a meniscus

It is important that the operator is provided with a safe and secure platform which allows line of sight to be established at right angles to the line of the liquid level, without undue stretching or bending. Significant parallax error can be introduced if the line of sight is difficult to achieve or is not established.

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 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.

Annex E
(informative)

Accuracy and uncertainty of volumetric measures

Table E.1 provides the uncertainty expected in the volume measured by different classification of measure designed, built and operated according to the guidance given in this document.

Table E.1 — Expected uncertainty of measures

Type of measure	Design	Expected range of expanded uncertainty in base volume, expressed with coverage factor $k = 2$ (approximating to a probability of 95 %)
Primary or reference measure	Automatic pipette	0,005 % to 0,01 %
Primary or reference measure	Top neck and closed bottom	0,01 % to 0,02 %
Field measure and proving tanks	Top neck and bottom cone (proving tank)	0,01 % to 0,02 %
Field measure and proving tanks	Top and bottom neck (proving tank)	0,01 % to 0,02 %
NOTE The overall uncertainty includes all uncertainties, including repeatability of the mean.		

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