
**Nuclear fuel technology — Tank
calibration and volume determination for
nuclear materials accountancy —**

Part 4:

**Accurate determination of liquid height in
accountancy tanks equipped with dip
tubes, slow bubbling rate**

*Technologie du combustible nucléaire — Étalonnage et détermination
du volume de cuve pour la comptabilité des matières nucléaires —*

*Partie 4: Détermination précise de la hauteur de liquide dans une cuve
bilan équipée de cannes de bullage, bullage lent*



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Contents

Page

Foreword.....	iv
Introduction	v
1 Scope	1
2 Physical principles involved.....	1
3 Required equipment, measurement conditions, and operating procedures.....	6
3.1 General.....	6
3.2 Tank and its measurement system	6
3.3 Software	6
3.4 Operating procedures	8
4 Determination of height from measurements of pressure	8
4.1 Differential pressure	8
4.2 Pressure sensor calibration drift	9
4.3 Buoyancy effects	9
4.4 Bubbling overpressure.....	10
4.5 Liquid height	11
5 Results	11
Annex A (informative) Estimation of quantities that affect the determination of liquid height.....	13
Annex B (informative) Bubbling overpressure.....	17
Annex C (informative) Operating procedure for making pressure measurements	19
Bibliography	21

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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ISO 18213-4 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

ISO 18213 consists of the following parts, under the general title *Nuclear fuel technology — Tank calibration and volume determination for nuclear materials accountancy*:

- *Part 1: Procedural overview*
- *Part 2: Data standardization for tank calibration*
- *Part 3: Statistical methods*
- *Part 4: Accurate determination of liquid height in accountancy tanks equipped with dip tubes, slow bubbling rate*
- *Part 5: Accurate determination of liquid height in accountancy tanks equipped with dip tubes, fast bubbling rate*
- *Part 6: Accurate in-tank determination of liquid density in accountancy tanks equipped with dip tubes*

Introduction

ISO 18213 deals with the acquisition, standardization, analysis, and use of calibration to determine liquid volumes in process tanks for the accountancy of nuclear materials. This part of ISO 18213 is complementary to the other parts, ISO 18213-1 (procedural overview), ISO 18213-2 (data standardization), ISO 18213-3 (statistical methods), ISO 18213-5 (fast bubbling rate) and ISO 18213-6 (in-tank determination of liquid density).

The procedure presented herein for determining liquid height from measurements of induced pressure applies specifically when a very slow bubbling rate is employed. A similar procedure that is appropriate for a fast bubbling rate is given in ISO 18213-5.

Measurements of the volume and height of liquid in a process accountancy tank are often made in order to estimate or verify the tank's calibration or volume measurement equation. The calibration equation relates the response of the tank's measurement system to some independent measure of tank volume.

Beginning with an empty tank, calibration data are typically acquired by introducing a series of carefully measured quantities of some calibration liquid into the tank. The quantity of liquid added, the response of the tank's measurement system, and relevant ambient conditions such as temperature are measured for each incremental addition. Several calibration runs are made to obtain data for estimating or verifying a tank's calibration or measurement equation. A procedural overview of the tank calibration and volume measurement process is given in ISO 18213-1. An algorithm for standardizing tank calibration and volume measurement data to minimize the effects of variability in ambient conditions that prevail during the measurement period is given in ISO 18213-2. The procedure presented in this part of ISO 18213 for determining the height of calibration liquid in the tank from a measurement of the pressure it induces in the tank's measurement system is a vital component of that algorithm.

In some reprocessing plants, the volume of liquid transferred into or out of a tank is determined by the levels of two siphons. The high level corresponds to the nominal volume, and the low level to the heel volume. If the transfer volume cannot be measured directly, then it is necessary to calibrate this volume (as described in the previous paragraph), because the difference between the actual volume and that used for inventory calculations will appear as a systematic error.

The ultimate purpose of the calibration exercise is to estimate the tank's volume measurement equation (the inverse of the calibration equation), which relates tank volume to measurement system response. Steps for using the measurement equation to determine the volume of process liquid in the tank are presented in ISO 18213-1. The procedure presented in this part of ISO 18213 for determining the height of process liquid in a tank from a measurement of the pressure it induces in the tank's measurement system is also a key step in the procedure for determining process liquid volumes.

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Nuclear fuel technology — Tank calibration and volume determination for nuclear materials accountancy —

Part 4:

Accurate determination of liquid height in accountancy tanks equipped with dip tubes, slow bubbling rate

1 Scope

This part of ISO 18213 specifies a procedure for making accurate determinations of the liquid height in nuclear-materials-accountancy tanks that are equipped with pneumatic systems for determining the liquid content. With such systems, gas is forced through a probe (dip tube) whose tip is submerged in the tank liquid. The pressure required to induce bubbling is measured with a manometer located at some distance from the tip of the probe. This procedure applies specifically when a very slow bubbling rate is employed.

A series of liquid height determinations made with a liquid of known density is required to estimate a tank's calibration equation (see ISO 18213-1), the function that relates the elevation (height) of a point in the tank to an independent determination of tank volume associated with that point. For accountability purposes, the tank's measurement equation (the inverse of its calibration equation) is used to determine the volume of process liquid in the tank that corresponds to a given determination of the liquid height.

2 Physical principles involved

The methodology in this part of ISO 18213 is based on measurements of the difference in hydrostatic pressure at the base of a column of liquid in a tank and the pressure at its surface, as measured in a bubbler probe inserted into the liquid. Specifically, the pressure, P , expressed in pascals, exerted by a column of liquid at its base is related to the height of the column and the density of the liquid, in accordance with Equation (1)¹⁾:

$$P = gH_M\rho_M \quad (1)$$

where

H_M is the the height of the liquid column (at temperature T_m), in m;

ρ_M is the average density of the liquid in the column (at temperature T_m), in kg/m³;

g is the local acceleration due to gravity, in m/s².

1) The subscript "M" is used to indicate the value of a temperature-dependent quantity at the temperature T_m .

For a liquid of known density, ρ , Equation (1) can be used to determine the height, H , of the liquid column above a given point from (a measurement of) the pressure, P , exerted by the liquid at that point. Therefore, process tanks are typically equipped with bubbler probe systems to measure pressure. Components of a typical pressure measurement system (see Figure 1) are discussed in detail in ISO 18213-1, together with a description of the procedural aspects of a typical calibration exercise.

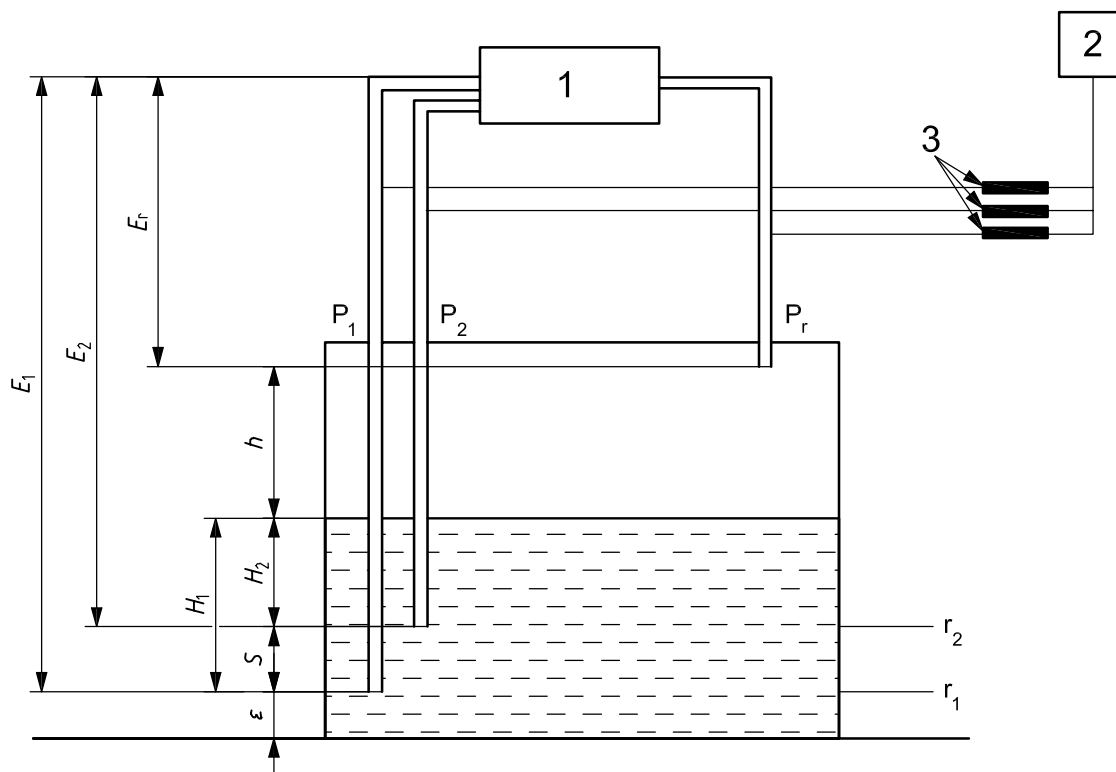
In practice, it is not absolute pressure that is measured, but rather the difference in pressure between the bottom and top of the liquid column. Gas is forced through two probes to measure this differential pressure. The tip of one probe (the long or major probe) is located near the bottom of the tank and immersed in the liquid. The tip of the second probe (reference probe) is located in the tank above the liquid surface.

To measure the pressure, P , exerted by a column of liquid, the pressure of gas in the probe immersed in the liquid should be measured while the gas-liquid interface is at static equilibrium. In practice, it is not possible to measure this pressure directly because it is difficult to maintain a stable and reproducible gas-liquid interface level in the probe. Therefore, a dynamic system is used to make measurements under conditions as close to equilibrium as possible: Gas is forced through the probe at a very low and constant flow rate, and its pressure is measured continuously. The fluctuation with time of these measurements (around some central value) depends on the bubbling frequency.

Provided the gas flow rate is low and constant, the gas pressure at the tip of the major probe first increases with time during the formation of a bubble. The release of a bubble from the tip of the probe causes a sudden increase in the level of the bubble-liquid interface at the tip of the probe and a corresponding decrease in pressure. For a probe with a small diameter (less than 8 mm), the pressure reaches a maximum and then decreases slightly before the sudden drop associated with bubble separation. For probes with larger diameters (greater than 8 mm), the maximum pressure that occurs just before bubble separation may not be accompanied by a decrease, but may instead show a short period of relative stability followed by a sudden drop in response to bubble separation. The dynamics of bubble formation and release, together with their effect on pressure in the probe, are shown in Figures 2 and 3.

Measurements of pressure are made at its maximum in the bubble formation-and-separation cycle because this is the point at which pressure is most stable. Measuring the maximum pressure results in an overpressure (a positive bias), denoted by $(\delta p)_{\max}$, relative to the actual pressure at the tip of the probe. A formula for computing the overpressure, $(\delta p)_{\max}$, is given in 4.4.

Various factors, in addition to bubbling overpressure, can affect the accuracy of the height determinations that follow from Equation (1). Temperature variations potentially have the greatest effect, especially on the comparability of two or more measurements (such as those taken for calibration), primarily because liquid density changes with temperature. Moreover, differences between actual pressures at the tip of the probes and observed pressures at the manometer can result from the buoyancy effect of air and the mass of gas in the probe lines. A general algorithm for standardizing pressure measurements that compensates for temperature variations and other measurement factors is presented in ISO 18213-2. For the case in which pressure measurements are made with a very slow bubbling rate, details of the pressure-to-height calculation step of this standardization algorithm are presented in Clause 4 of this part of ISO 18213. Analogous calculations that apply for a fast bubbling rate are given in ISO 18213-5. Procedures for estimating the uncertainty of the resulting height determinations are given in ISO 12813-3.



NOTE This configuration is typical but other configurations are possible, see Reference [11] for examples.

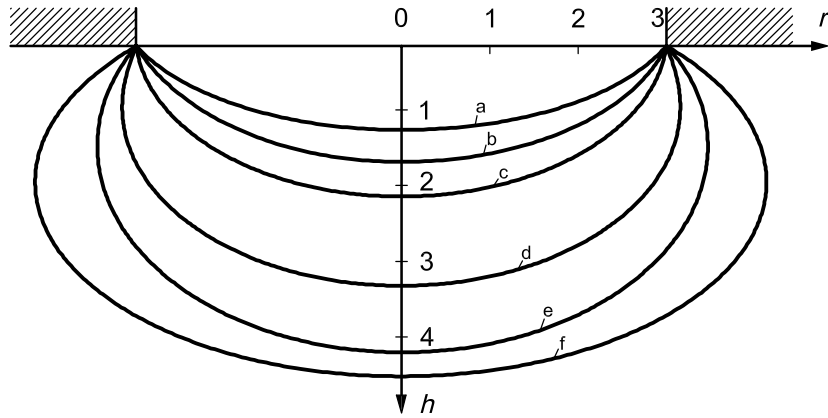
Key

- 1 manometer
- 2 gas supply (N₂ or air)
- 3 flowmeters

	Major probe	Minor probe	Reference probe
Probe designation	P ₁	P ₂	P _r
Reference point	r ₁ (primary)	r ₂ (secondary)	—
Height of liquid above reference point	H ₁	H ₂	—
Elevation of pressure gauge (manometer) above reference point	E ₁	E ₂	E _r
Elevation of reference probe above liquid surface	$h = E_1 - E_r - H_1$	$h = E_2 - E_r - H_2$	—
Elevation of reference point above bottom of tank	ε	ε + S ^a	—

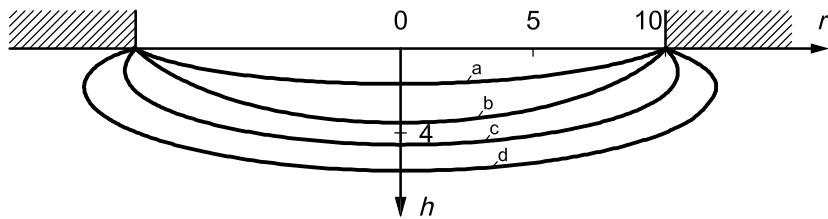
^a Vertical distance (probe separation): $S = H_1 - H_2$.

Figure 1 — Elements of a typical pressure measurement system for determining liquid content



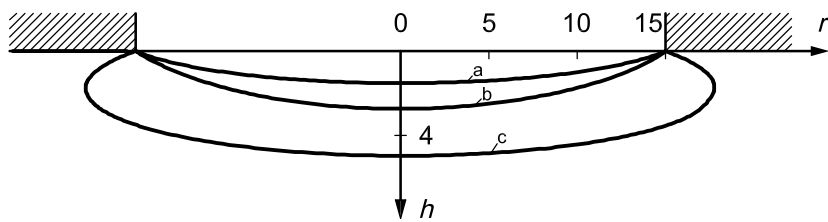
a) Radius of the bubbler probe, $r = 3$ mm

- | | | | |
|---|------------------|---|------------------|
| a | $\Delta P = 3,7$ | d | $\Delta P = 7,1$ |
| b | $\Delta P = 4,8$ | e | $\Delta P = 7,2$ |
| c | $\Delta P = 5,7$ | f | $\Delta P = 7,4$ |



b) Radius of the bubbler probe, $r = 10$ mm

- | | | | |
|---|------------------|---|------------------|
| a | $\Delta P = 2,0$ | c | $\Delta P = 5,4$ |
| b | $\Delta P = 4,4$ | d | $\Delta P = 5,9$ |



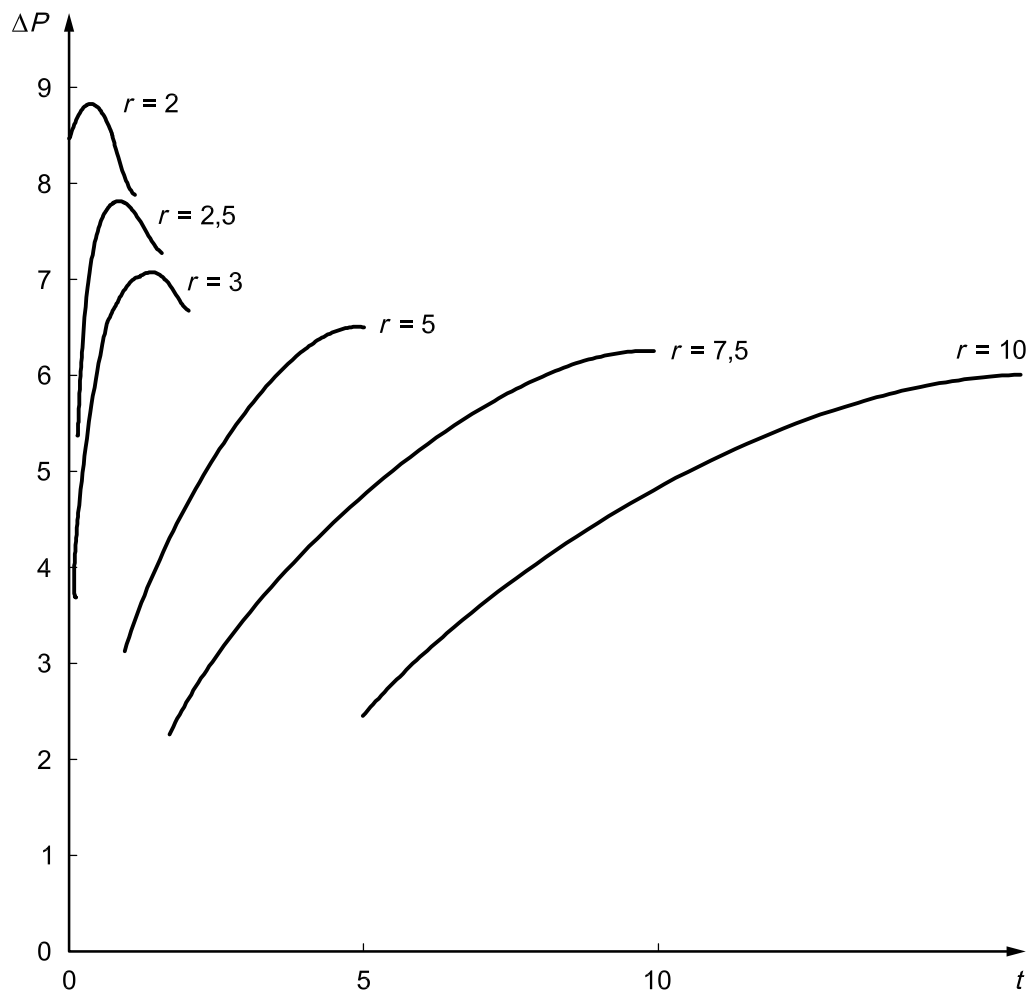
c) Radius of the bubbler probe, $r = 15$ mm

- | | |
|---|------------------|
| a | $\Delta P = 1,8$ |
| b | $\Delta P = 2,8$ |
| c | $\Delta P = 5,7$ |
- $\Delta P = \text{mm of H}_2\text{O}$.

Key

- r radius of the bubbler probe, mm
- h bubble height, mm

Figure 2 — Evolution of a bubble in water

**Key** t time, s ΔP overpressure, mm of H_2O r radius of the bubbler probe, mm**Figure 3 — Evolution of bubbling overpressure in water**

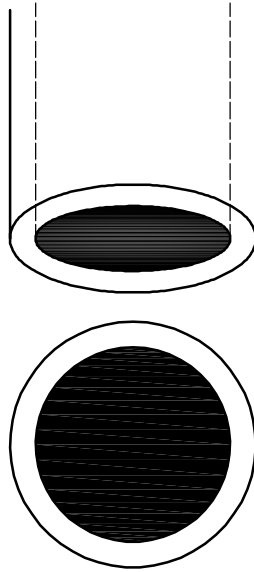


Figure 4 — End of the dip tube

3 Required equipment, measurement conditions, and operating procedures

3.1 General

The pressure measurements to which this part of ISO 18213 applies are made either to calibrate a tank or to determine the volume of process liquid it contains. The same equipment, operating procedures, and standardization steps are used for both purposes. The elements of a pressure measurement system for determining the liquid content of a process tank are described in detail in Clause 4 of ISO 18213-1:2007. Measurement conditions and operating procedures for making pressure measurements to determine liquid height are described in detail in Clause 6 of ISO 18213-1:2007.

Only aspects of equipment, measurement conditions and operating procedures that differ from those described in ISO 18213-1 and that are specific to a slow bubbling rate are discussed in 3.2 to 3.4.

3.2 Tank and its measurement system

The tank should be connected to an air flow system that ensures a steady slow bubble rate (e.g. 2 to 4 bubbles per minute for a 15 mm diameter probe; see ISO 18213-1:2007, 4.2). For a slow bubbling rate, the submerged probes should have a cylindrical geometry relative to the vertical axis (see Figure 4).

Experience with differential electromanometers has shown that they exhibit measurement drift. It is therefore recommended that the instrument “zero” (i.e. the reading when the same pressure is applied at both inlets) be read and recorded periodically. If the drift proves to be significant, this information can be used to correct the raw data as necessary before other standardization steps are carried out.

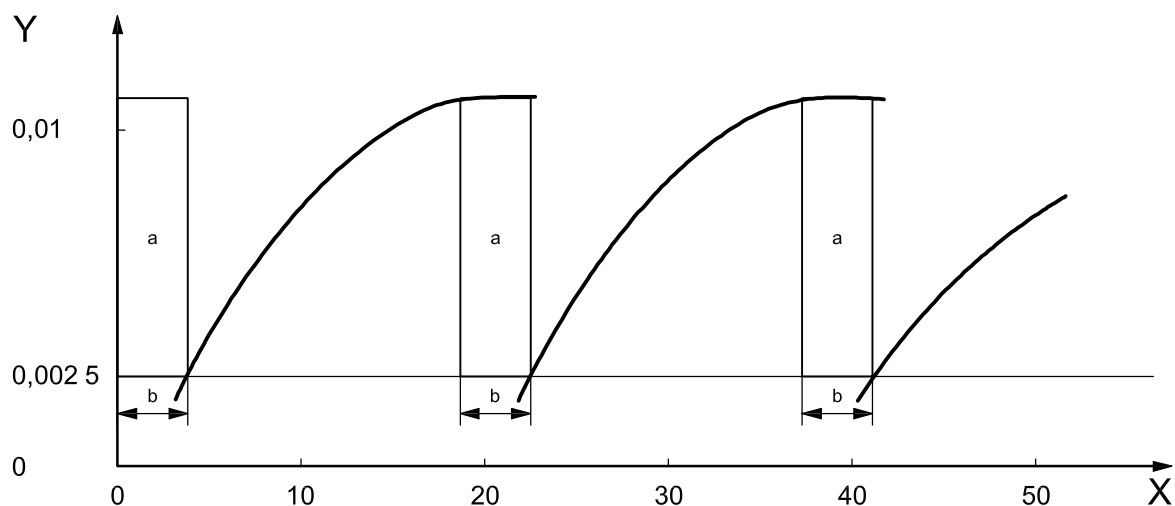
3.3 Software

The measurement system should be connected to a micro-computer that controls operations and processes the data into requisite form (see ISO 18213-1:2007, 4.5). The software should be adapted to the measurement system and it should meet the following requirements.

- A pressure measurement should be made under conditions as close to equilibrium as possible, therefore at a slow enough bubbling rate (say one bubble every 15 s to 25 s for a 15 mm probe) so that the maximum overpressure is nearly independent of the bubbling rate. The software should include a subroutine that is capable of measuring bubbling frequency.

- The pressure should be measured at the point of maximum overpressure (see Figures 5 and 6). The software should therefore contain a subroutine to monitor the minimum and maximum overpressure during the bubble cycle.
- The software should monitor the pressure values in the upper third of the fluctuation range, determine the maximum value, and select the value(s) to be retained for the height calculation. In practice, ten rapid (5 Hz) measurements near the maximum pressure are retained to minimize the effect of measurement fluctuations. The criteria for selecting points to be retained depend on the “bubble profile”. Therefore a subroutine is required to record and select suitable readings. A graphical display of all pressure measurements is most helpful for selecting suitable readings. If the maximum pressure is obtained before the bubble breaks away, as is the case for small diameter probes (see Figure 3), ten values are selected from the readings around this maximum. For larger diameter probes, the bubble separation causes a rapid drop in pressure: the pressure falls below the monitoring range. In this case, the 6th through the 15th readings prior to the point of separation are used in the calculation. In all cases, the average of the selected values is retained.
- The previous three steps are carried out for five successive bubbles. The average and standard deviation of these five points are calculated and stored.

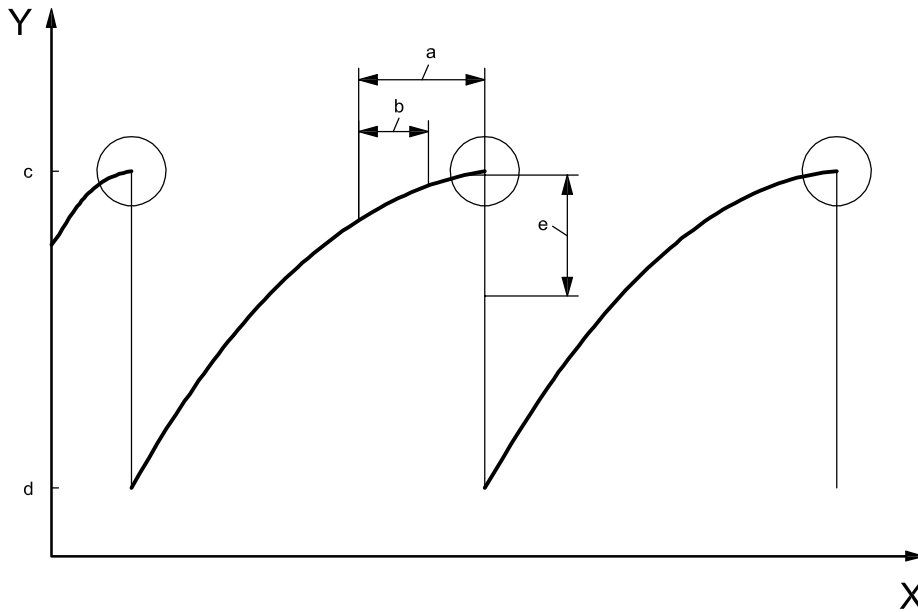
The total time required to make the measurements for a determination of liquid height is closely linked to the time required for the liquid surface to reach equilibrium (i.e. to stabilize) following the addition of liquid to the tank. Less than 5 min is required to actually make the required measurements.



Key

- X time, s
- Y differential pressure, ΔP , mV
- a Overpressure, δP .
- b 0,25 s.

Figure 5 — Bubble profile with a maximum before separation



Key

- X time
- Y differential pressure, ΔP
- a 15 points.
- b 10 points.
- c Maximum, M.
- d Minimum, m.
- e $1/3 (M-m)$.

Figure 6 — Bubble profile without a maximum before separation

3.4 Operating procedures

Unlike the situation for a fast bubbling rate, operating procedures are required to switch the air flow from a fast bubbling rate that is used during routine operations to a slow bubbling rate that is required during measurement periods. This can be accomplished by means of the steps described in Annex C.

4 Determination of height from measurements of pressure

4.1 Differential pressure

When gas flows at a constant, slow rate through a dip tube immersed in liquid, a periodic fluctuation of pressure is observed at a pressure sensor (usually located at some distance above the tank). As a bubble forms, the pressure at the tip of the dip tube increases continuously, and then decreases abruptly when the bubble breaks away. Therefore, if accurate measurements of pressure are required, they shall be taken under well-defined conditions. The point at which the pressure achieves its maximum is selected because pressure is relatively stable at this point and measurements have well-defined physical significance. A very slow gas flow rate (2 to 4 bubbles per minute for a 15 mm diameter probe) is required to achieve a state of quasi-equilibrium.

The bubbling pressure depends not only on the height of liquid above the tip of the dip tube, but also on the pressure in the tank at the liquid surface. What is measured in practice is the difference between the pressure of gas inside the submerged tube, $P_1(E_1)$, and the pressure of the same gas flowing into a second tube that vents into the vapour space at the top of the tank above the liquid surface, $P_r(E_1)$:

$$\Delta P_1 = P_1(E_1) - P_r(E_1) \tag{2}$$

The differential pressure, ΔP_1 , is measured by a manometer located at some elevation, E_1 , above the tip of the major probe. One inlet of the manometer is connected to the dip tube whose tip is submerged in the tank liquid and a second is connected to the reference probe that vents into the air space at the top of the tank.

As noted in Clause 2, various factors can affect the accuracy of the calculation for determining height from pressure based on Equation (1). Therefore, measurements of differential pressure shall be adjusted to compensate for variations in ambient conditions during the measurement period before they can be converted into accurate measures of liquid height. Appropriate corrections are discussed in 4.2 to 4.4.

4.2 Pressure sensor calibration drift

Pressure fluctuations (e.g. drift) over time may result from a zero shift in the pressure sensors (manometers). Therefore, it is necessary to make measurements of the instrument “zero” before and at regular intervals during a series of measurements (e.g. every hour for instance depending on the instrument). This is done by equalizing the pressure at both inlets of the manometer and recording the results. These measurements should be used to correct pressure measurement as necessary for the effect of zero drift (which can exceed 10 Pa). Excellent results can often be obtained simply by making a linear adjustment (shift) to the observed pressure measurements.

In general, however, the response of the pressure sensor and its measurement chain (sensor and voltmeter) is not a linear function of pressure. Thus, it may be necessary to develop a suitable model of measurement system response. A low-order polynomial will typically be adequate for this purpose.²⁾

4.3 Buoyancy effects

After pressure measurements have been corrected for instrument drift, they can in principle be converted into determinations of height, $H_{1,M}$, by means of Equation (1) rewritten in accordance with Equation (3):

$$H_{1,M} = \Delta P_1 / (g \rho_M) \quad (3)$$

where

ρ_M is the average density of the liquid in the tank at its measurement temperature, T_m .

However, according to the principle of Archimedes, it is in fact more accurate to use Equation (4):

$$H_{1,M} = \Delta P_1 / [g(\rho_M - \rho_{g,r})] \quad (4)$$

where

$\rho_{g,r}$ is the density of the medium (gas) in which the measurements are made, typically air in the tank above the liquid surface.

Moreover, the differential pressure, ΔP_1 , is measured by a pressure sensor that is not located at the liquid surface, but typically at a markedly different elevation. Thus, the weight of the gas column in the pneumatic lines should also be taken into account. Because pressure equilibrium exists on both sides of the liquid-gas interface, one can write

$$P_1(E_1) + gE_1\rho_{g,1} = P_r(E_1) + gE_r\rho_{g,r} + g(E_1 - E_r - H_{1,M})\rho_{a,s} + gH_{1,M}\rho_M + (\delta p)_{\max} \quad (5)$$

2) With Crouzet 43 or 44 manometers, for example, the response is described by a quadratic polynomial that reduces possible bias of several pascals to less than one pascal.

where

- $H_{1,M}$ is the height of liquid in the tank above the tip of the bubbling (major) probe ³⁾;
- E_1 is the elevation of the manometer (pressure sensor) above the tip of the major probe;
- E_r is the elevation of the manometer above the tip of the reference probe;
- $\rho_{g,1}$ is the density of the bubbling gas at pressure P_1 in the major probe;
- $\rho_{g,r}$ is the density of the bubbling gas at pressure P_r in the reference probe;
- $\rho_{a,s}$ is the density of air in the tank above the liquid surface at pressure P_r ;
- $(\delta p)_{\max}$ is the maximum overpressure, relative to that at the tip of the probe, observed during the bubbling process.

It follows from Equation (5) that

$$\begin{aligned}
 H_{1,M} &= [P_1(E_1) - P_r(E_r) + gE_1\rho_{g,1} - gE_1\rho_{a,s} - gE_r\rho_{g,r} + gE_r\rho_{a,s} - (\delta p)_{\max}] / [g(\rho_M - \rho_{a,s})] \\
 &= [\Delta P_1 + gE_1(\rho_{g,1} - \rho_{a,s}) - gE_r(\rho_{g,r} - \rho_{a,s}) - (\delta p)_{\max}] / [g(\rho_M - \rho_{a,s})]
 \end{aligned}
 \tag{6}$$

If the bubbling gas is air, then $\rho_{g,1} = \rho_{a,1}$ and $\rho_{g,r} = \rho_{a,r}$. In this case, Equation (6) can be written as

$$H_{1,M} = [\Delta P_1 + gE_1(\rho_{a,1} - \rho_{a,s}) - gE_r(\rho_{a,r} - \rho_{a,s}) - (\delta p)_{\max}] / [g(\rho_M - \rho_{a,s})]
 \tag{7}$$

The expression for $H_{1,M}$ in Equation (7) includes adjustments to the measured differential pressure, ΔP_1 , that compensate for the buoyancy of the medium (air) in which the measurements are made, the weight of the gas in the pneumatic lines, and the maximum bubbling overpressure at the tip of the submerged probe. A formula for computing the maximum overpressure, $(\delta p)_{\max}$, is given in 4.4. Other quantities on the right-hand side of Equation (7) may be computed by means of formulae given in Annex A.

Equation (7) is used to determine liquid heights from measurements of pressure for both tank calibration and volume determination.

4.4 Bubbling overpressure

Measurements of pressure are made at the maximum in the bubble formation-and-separation cycle because the pressure is most stable at this point. In Equation (7), the maximum overpressure, relative to the actual pressure at the tip of the probe, is denoted by $(\delta p)_{\max}$. For aqueous solutions, it is shown in Annex B that this overpressure can be described by the following empirical relationship:

$$(\delta p)_{\max} = (gr\rho_M) / [rc^{0,5}/2 - 0,14] = (2gr\rho_M) / [rc^{0,5} - 0,28]
 \tag{8}$$

where

- r is the radius of fixation of the bubble;
- $c = g(\rho_M - \rho_g) / \sigma$

3) The subscript "1" is used in this part of ISO 18213 to indicate "1" quantities that refer to the major probe (see Figure 1). The steps for standardizing data for a second probe are completely analogous.

where

ρ_g is the the density of gas in pressure line ($\rho_g = \rho_{a,1}$ if the bubbling gas is air);

σ is the the surface tension at the liquid-gas interface.

The normalized overpressure (expressed in terms of liquid height), given by Equation (8), depends on the liquid in the tank through c , as given by Equation (9). This quantity is essentially independent of pressure and is therefore also independent of the height of liquid in the tank. The quantity c varies with temperature as the ratio $(\rho_M/\sigma)^{0,5}$.

4.5 Liquid height

Taken together, Equations (7) to (9) yield an estimate of liquid height, $H_{1,M}$, that is valid at temperature T_m , the temperature of the liquid in the tank at the time of measurement.

The accuracy of height determinations obtained by means of Equations (7) to (9) is limited by how well the density of the measured liquid is determined at the prevailing temperature. It is also important to note that $H_{1,M}$ is the height of the liquid in the tank only at the measurement temperature. In particular, $H_{1,M}$ is *not* the height of the same liquid at some other temperature.

Some of the effects identified in Equations (7) to (9) may be quite small. Whether or not they must be taken into account in a particular situation depends on the capability of the tank's measurement system (e.g. manometer) and established measurement accuracy requirements. If the quantities in these equations must be taken into account, they should be measured whenever possible. However, an algorithm is given in Annex A for estimating these quantities when measurements are unavailable. Under normal operating conditions, use of the suggested default values in lieu of actual measurements will provide acceptable results in nearly all situations.

5 Results

Starting with a measure of the pressure required to induce bubble formation at the tip of a probe submerged in the liquid in an accountancy tank, the standardization procedure described in Clause 4 yields an accurate measure of the height of the column of liquid exerting the pressure. With high-precision manometers and good technique applied under stable conditions, it is possible to achieve relative accuracies for individual height determinations in the range of 0,01 % to 0,02 % ⁴⁾, ⁵⁾ for pressures of approximately 10 000 Pa or greater. This degree of accuracy corresponds to 1 Pa to 2 Pa, or approximately 0,1 mm to 0,2 mm, for a 1 m column of water.

The accuracy of liquid height determinations obtained from Equations (7) to (9) is limited by the accuracy of available measurements of liquid density. Thus, to successfully employ the methods of this part of ISO 18213, the density of the measured liquid must be determined with sufficient accuracy at its measurement temperature. Therefore, a liquid (such as water) whose density has been very accurately determined at all measurement temperatures is required for calibration. In tanks equipped with two or more dip tubes of differing lengths, it is possible to make accurate determinations of the densities of process liquids from in-tank measurements. The first step is to accurately determine the vertical separation between the two probes (i.e. to calibrate their separation) using a suitable calibration liquid. The probe separation calibration can in turn be used to determine the density of the process liquid in question. Details of this two-stage procedure are presented in ISO 18213-6.

4) In ISO 18213, all estimates of accuracy are expressed in terms of the half-width of two standard deviation (95 %) confidence intervals. Thus, the assertion here is that relative standard deviations for individual measurements in the range 0,005 % to 0,01 % are possible.

5) Depending on the resolution of the manometer, it may be necessary to average the results of several height determinations to achieve this level of precision (see ISO 18213-1:2007, 6.6.3).

Because liquid density changes with temperature, a height measurement, $H_{1,M}$, obtained from Equations (7) to (9), corresponds to the height of liquid in the tank only at the measurement temperature, T_m . In particular, the height of the liquid used to determine $H_{1,M}$ is not equal to $H_{1,M}$ at any other temperature. Moreover, process tanks do not in general have constant cross-sectional areas, so heights determined for a liquid at one temperature are not directly comparable to those determined at other temperatures, even for the same liquid. Therefore, except in very special cases, it is not appropriate to use an equation of the form $H_2 = H_1 \cdot \rho_1 / \rho_2$ to make thermal adjustments. In particular, the ratio of the densities of water at two temperatures should not be used to infer the height of process liquid at one temperature from its height at another because unacceptably large errors can result. To ensure that the resulting height determinations are comparable, the standardization steps in Clause 4 should be applied individually to each measurement of pressure.

The value of $H_{1,M}$ obtained from Equations (7) to (9) is valid only at the measurement temperature, T_m . Therefore, it is necessary to standardize the height measurements made at differing temperatures to a fixed reference temperature to compensate for thermally induced changes in the tank and dip tubes. Standardization of several measurements at a fixed reference temperature is accomplished as follows. When the liquid in the tank is at temperature T_m , then $H_{1,M}$ determines a point on the tank wall at the liquid surface⁶⁾. If the tank temperature now changes to T_r , then the elevation of the indicated point (but not the height of the liquid used to determine $H_{1,M}$) above the tip of the probe changes to

$$H_{1,r} = H_{1,M} / (1 + \alpha \Delta T_m) \tag{10}$$

where $\Delta T_m = T_m - T_r$ and α is the linear (thermal) coefficient of expansion for the tank and its probes. To ensure comparability in the presence of temperature variations, all height determinations obtained from Equations (7) to (9) should be standardized to a convenient reference temperature (e.g. $T_r = 25\text{ °C}$ or $T_r = 31\text{ °C}$) by means of Equation (10). See ISO 18213-2:2007, 5.3 for additional details.

An equation has been developed by Jones^[8] that relates the differential pressure exerted by a column of water at one temperature to the pressure it exerts at another temperature. A paper by Jones and Crawford^[10] describes an experiment which shows that the calculated results are in good agreement with the observed results. For water, this eliminates the need to apply the standardization steps of Clause 4 individually to each pressure measurement. Similar equations can be developed for other liquids, but a safe alternative is to always make the corrections indicated by Equations (7) to (9) for each measurement of pressure.

An algorithm is presented in ISO 18213-2 for standardizing a set of data and using these (standardized) data to calibrate a tank (i.e. to estimate the relationship between the response of the tank's measurement system and some independent measure of its liquid content). The procedure specified in this part of ISO 18213 for determining liquid height from pressure is a key step in the overall standardization-and-calibration process. Steps in this part of ISO 18213 are also required when the calibration equation (or its inverse) is subsequently used to determine process liquid volumes (see Clause 7 of ISO 18213-1:2007).

6) $H_{1,M}$ denotes the height of the point determined by means of Equations 7 to 9 for a liquid at temperature T_m .

Annex A (informative)

Estimation of quantities that affect the determination of liquid height

A.1 Introduction

Procedures are presented in this annex for estimating the quantities required to determine liquid height, $H_{1,M}$, from differential pressure, ΔP_1 , by means of Equations (7) to (9). In these equations, heights are expressed in metres, pressures are expressed in pascals, and densities are expressed in kilograms per cubic metre. The local acceleration due to gravity, g , is expressed in metres per second squared.

A.2 Liquid density, ρ

Any liquid compatible with the process liquid can be used for tank calibration, provided that accurate measurements of its density can be obtained at all measurement temperatures. Demineralized water is a preferred calibration liquid because its density is well known and can be accurately determined at all temperatures of interest. Equation (A.1) gives very accurate determinations of the density of air-free (freshly distilled) water, ρ_M , in kilograms per cubic metre, for temperatures $T = T_m$ between 4 °C and 40 °C:

$$\rho_M = A + BT + CT^2 + DT^3 + ET^4 + FT^5 \quad (\text{A.1})$$

where

$$A = 999,843\ 22$$

$$B = 6,684\ 416 \times 10^{-2}$$

$$C = -8,903\ 070 \times 10^{-3}$$

$$D = 8,797\ 523 \times 10^{-5}$$

$$E = -8,030\ 701 \times 10^{-7}$$

$$F = 3,596\ 363 \times 10^{-10}$$

For temperatures between 3 °C and 30 °C, the estimated residual standard deviation for this fit is less than 0,001 kg/m³. For other temperatures between 1 °C and 40 °C, the reported standard deviation does not exceed 0,001 4 kg/m³.

Water can become saturated after being exposed to air for a relatively short period of time (approximately 15 h). If necessary, the density of air-saturated water at 1 atm can be calculated by adding the following correction to the estimate obtained from Equation (A.1):

$$\Delta\rho_M = -4,873 \times 10^{-3} + 1,708 \times 10^{-4}T - 3,108 \times 10^{-6} T^2 \quad (\text{A.2})$$

Equation (A.2) is applicable for temperatures between 0 °C and 20 °C. The correction for air saturation is -0,002 70 kg/m³ at 20 °C and its effect diminishes with increasing temperature. The estimated total uncertainty of values calculated with Equation (A.2) is reported as 2×10^{-4} kg/m³ at the 99 % confidence level. Thus, the effect of air saturation at temperatures greater than 20 °C can safely be ignored for most safeguards applications.

Equations (A.1) and (A.2) are based on a recent re-determination of the density of water [11]. Equation (A.1), or Equation (A.1) and Equation (A.2) in combination, can be used to compute the density of water with sufficient accuracy and precision for safeguards purposes.

If some liquid other than water is used for calibration, then its density must be determined with suitable accuracy at all measurement temperatures before Equations (7) to (9) (see 4.3 and 4.4) can be successfully applied.

Likewise, the use of Equations (7) to (9) to determine the height of some process liquid in the tank requires an accurate measure of its density at the measurement temperature. A method for making accurate determinations of liquid density from in-tank measurements is presented in ISO 18213-6.

A.3 Density of gas in the pressure lines

A.3.1 General formula for air density

The density of the gas in the pressure lines is required to evaluate some of the terms in Equations (7) to (9). If the gas is air, then its density, ρ_a , in kilograms per cubic metre, can be determined from its temperature, pressure and relative humidity by means of Equation (A.3) [9]:

$$\rho_a = \frac{(0,003\ 484\ 7)}{(T + 273,15)} \left[P - 6,653\ 06 \times 10^8 \times U \times e^{\left(\frac{-5\ 315,56}{T + 273,15} \right)} \right] \quad (\text{A.3})$$

where

- P is the pressure, in pascals;
- U is the relative humidity of the air, in percent saturation;
- T is the average temperature of the gas, in degrees Celsius.

If the bubbling gas is not air (e.g. N_2), then a suitable alternative to Equation (A.3) is required. In any case, Equation (A.3) can be used to estimate the density of the air in the tank above the liquid surface.

A.3.2 Density of air in the major probe line, $\rho_{a,1}$

Measurements of P (Pa), U (% saturation), and T (°C) are required to use Equation (A.3) for estimating the density of air in the major probe line. If measurements are not available, the following default values will yield acceptable results in nearly all cases.

A suitable default value for P is

$$P = \Delta P_1 + P_s \quad (\text{A.4})$$

where

- ΔP_1 is the observed differential pressure at the manometer;
- P_s is the barometric pressure minus off-gas pressure.

Standard atmospheric pressure at sea level is $1,013\ 25 \times 10^5$ Pa, and typical off-gas pressure is 500 Pa, equivalent to the pressure exerted by a 50 mm column of water. If these values are used in Equation (A.4), the result is

$$P = \Delta P_1 + 1,008\ 25 \times 10^5 \quad (\text{A.5})$$

In practice, when standard atmospheric pressure is used in Equation (A.4) (and elsewhere) to estimate P_s , it should be adjusted to account for the elevation of the facility above sea level and also, if possible, for the actual off-gas pressure. Since variations in atmospheric pressure will generally have the greatest effect on results, it is desirable to use actual measurements of atmospheric pressure whenever possible.

Suitable default values for U and T are

$U = 20\%$ saturation (if the bubbling gas is dry), or $U = 80\%$ saturation (if the bubbling gas is wet),

$T = 25\text{ }^\circ\text{C}$

The average of the ambient temperature and the temperature of the tank liquid can be used as an alternative to a standard value for T .

A.3.3 Density of air in the reference probe line, $\rho_{a,r}$

As with the major probe, suitable measurements of P , U , and T are required to use Equation (A.3) for estimating the density of air in the reference probe line. The following default values for P (Pa), U (% saturation), and T ($^\circ\text{C}$) will yield acceptable results in nearly all cases:

$P = P_s = 1,008\ 25 \times 10^5\ \text{Pa}$;

$U = 20\%$ saturation (dry bubbling gas), or $U = 80\%$ saturation (wet bubbling gas);

$T = 25\text{ }^\circ\text{C}$.

The current ambient temperature can be used as an alternative to a standard value for T .

A.3.4 Density of air in the tank, $\rho_{a,s}$

The density of air in the tank above the surface of the liquid can be estimated from Equation (A.3) with the aid of the following values for P (Pa), U (% saturation), and T ($^\circ\text{C}$):

$P = P_s = 1,008\ 25 \times 10^5\ \text{Pa}$;

$U = 50\%$ saturation (if the bubbling gas is dry), or $U = 90\%$ saturation (if the bubbling gas is wet);

$T =$ temperature of the liquid in the tank.

A.4 Quantities associated with bubble formation, r and σ

Quantities associated with the bubbling overpressure, $(\delta p)_{\text{max}}$, given by Equations (8) and (9) include the radius of fixation of the bubble, r , and the surface tension at the liquid-gas interface, σ . Densities required for determining the parameter c in Equation (9) may be estimated as indicated in Clauses A.2 and A.3. If the liquid is water, its density may be estimated by means of Equation (A.1). If the bubbling gas in the major probe is air, then its density may be estimated as indicated in A.3.2.

A suitable estimate of the radius of fixation, r , is $d/2$, where d is the inner diameter of the submerged probe, in metres.

If the liquid in the tank is water and the bubbling gas is air, then the surface tension at the air-water interface, σ , in newtons per metre, may be estimated by means of Equation (A.6) [8]:

$$\sigma = 75,675 \times 10^{-3} - 1,376\ 2 \times 10^{-4}T - 3,938 \times 10^{-7}T^2 + 1,076 \times 10^{-9}T^3 \quad (\text{A.6})$$

where T is the temperature of the tank liquid, in degrees Celsius.

A.5 Elevations, E_1 and E_r

It is necessary that the elevations, E_1 and E_r , of the manometer above the tips of the major and reference probes, in metres, respectively, be measured or determined from engineering drawings. There are no default values for these quantities.

A.6 Local acceleration due to gravity, g

The local acceleration due to gravity depends on elevation and latitude, and should be determined at the measurement site. In lieu of an on-site determination, the local acceleration due to gravity may be determined with suitable accuracy from charts in Appendix 7 of Reference [12]. An erroneous value will produce a bias in the resulting height determination that is approximately proportional to the error in the determination of the local acceleration due to gravity. In any event, the same value should be used for all measurements of both calibration and process liquids to ensure internal consistency among measurements.

A.7 Coefficient of linear expansion, α

The linear (thermal) coefficient of expansion for the tank and its probes is required to standardize height determinations to a convenient reference temperature as indicated in Clause 5. If it is not possible to determine the type of material from which the tank is constructed, then the coefficient of linear expansion for Type 304 stainless steel may be used (provided the tank is made of steel). This coefficient has the value $\alpha = 1,728 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ [13].

A.8 Note on use of default values

Whenever possible, the quantities required to apply Equations (7) to (9) should be measured. However, where measurement is impossible or impractical, the default values suggested in this annex will give acceptable results under normal operating conditions in nearly all cases. For certain parameters, such as atmospheric pressure and the local acceleration due to gravity, systematic biases are introduced when standard values are used in place of actual measurements. These biases have no effect on comparisons within a facility, provided that the same values are used for all calculations, but they may become significant when measurements from facilities at several locations are compared or combined. In this case, local determinations of the relevant parameters should be employed to ensure that the results generated by means of Equations (7) to (9) remain comparable.

Annex B (informative)

Bubbling overpressure

Equations (7) to (9) in Clause 4 contain the following term for the normalized bubbling overpressure (i.e. the overpressure due to the bubble at the tip of the probe), expressed in terms of height:

$$(\delta p)_{\max} / [g(\rho_M - \rho_{a,s})]$$

In the case where the tip of the probe is symmetrical about its vertical (central) axis, Hartland and Hartley^[6] show that the same equation can be used to describe the bubbling overpressure for all liquids and gases. If M is a point of the bubble surface with elevation, Z , above the base of the bubble, then the bubbling overpressure, δP , at M is given by Equation (B.1):

$$\delta P = 1/R_1 + 1/R_2 = 2/B + Z \quad (\text{B.1})$$

In Equation (B.1), B is a parameter that varies with bubble form and R_1 and R_2 are the principal radii of curvature (in the vertical and horizontal planes) at the point M, expressed in length units equal to $c^{-0.5}$, where

$$c = g(\rho_M - \rho_g) / \sigma \quad (\text{B.2})$$

In Equation (B.2), σ is the surface tension at the liquid-gas interface, ρ_M is the density of the liquid and ρ_g is the density of the gas.

In Equation (B.1), $Z = zc^{0.5}$, where the elevation is expressed in the same units as R_1 and R_2 , and

$$\delta P = \delta p / [\sigma c^{0.5}] \quad (\text{B.3})$$

In Equation (B.3), δp is the bubbling overpressure expressed relative to the hydrostatic pressure at the tip of the dip tube. Let X equal the distance between M and the axis of symmetry (in units of $c^{-0.5}$), Hartland and Hartley give tables that, for each value of B , contain values of X and Z that correspond to a given value of M, together with corresponding values of δP .

Using the tables of Hartland and Hartley, it is possible to determine $(\delta P)_{\max}$, provided that the radius of the fixation, X , of the (circular) bubble is given. The Equation (B.4) is given for relating X and $(\delta P)_{\max}$:

$$X / (\delta P)_{\max} = X/2 - 0,14 \quad (\text{B.4})$$

Equation (B.4) may be written as

$$(\delta p)_{\max} / [g(\rho_M - \rho_g)] = r / [r c^{0.5}/2 - 0,14] = 2r / [r c^{0.5} - 0,28] \quad (\text{B.5})$$

where

$$c = g(\rho_M - \rho_g) / \sigma$$

is given by Equation (B.2). Equation (B.5) depends on the radius of fixation of the bubble, r , the density of the tank liquid, ρ_M , the surface tension at the liquid/gas interface, σ , and the density of the bubbling gas, ρ_g . The quantities in Equation (B.5) can be determined as indicated in Annex A.

For a given liquid, the expression for normalized overpressure given by Equation (B.5) is independent of pressure, and therefore of the height of liquid above the tip of the tube. (This statement is only correct as a first approximation because ρ_g depends on liquid height, as can be seen from Equation (B.5), but this effect is very small.) Thus, the effect of this term is the same as if the origin of the height determinations were shifted by a constant amount from the tip of the dip tube. For water and air, and for a dip tube with an inner diameter of 14 mm, the estimated normalized overpressure is approximately 6,1 mm at 20 °C, a quantity which is certainly not negligible.

The normalized overpressure does change with liquid (through its density and its surface tension), however. If for instance, the calibration is carried out with water and the calibration equation is to be used to measure uranyl nitrate, the change in normalized overpressure should be taken into account. This quantity should be estimated for the appropriate conditions, or at a minimum, the deviation of this quantity from its calibration value should be estimated. The difference in this term between water and a typical input solution is approximately 1 mm for a dip tube with inner diameter of 14 mm.

The normalized overpressure varies with temperature through ρ_M and σ . For water between 20 °C and 30 °C, the variation is approximately 0,09 mm for a dip tube with an inner diameter of 14 mm.

The above calculations are based on the assumption that the bubble is fixed at the inner wall of the dip tube, permitting r to be identified with the inner radius of the dip tube. This assumption should be verified for uranyl nitrate, and in particular it should be verified that the bubble does not extend beyond the thickness of the dip tube wall (i.e. the outer radius of the dip tube). It should also be verified that the overpressure reaches its maximum value before the bubble separates from the tip of the dip tube. If this is not the case, then it is necessary to evaluate the effect of this phenomenon on the pressure measurements.

Hosoma *et al.* [7] have proposed an alternative model for the maximum bubble overpressure. The authors show that their results are in good agreement with those of Hartland and Hartley, and further, that both are in good agreement with experimental results for several liquid-gas combinations over the following range of values for c :

$$3,6 \times 10^{-6} \leq c^{-1} \leq 7,4 \times 10^{-6} \text{ or } 12,35 \times 10^5 < c < 2,8 \times 10^5 \quad (\text{B.6})$$

Their paper should be consulted for complete details.

Annex C (informative)

Operating procedure for making pressure measurements

C.1 General

The procedure described in this annex is suitable for making pressure measurements with a differential electromanometer (see Figure C.1). It involves the steps in C.2 and C.3.

C.2 Initial steps

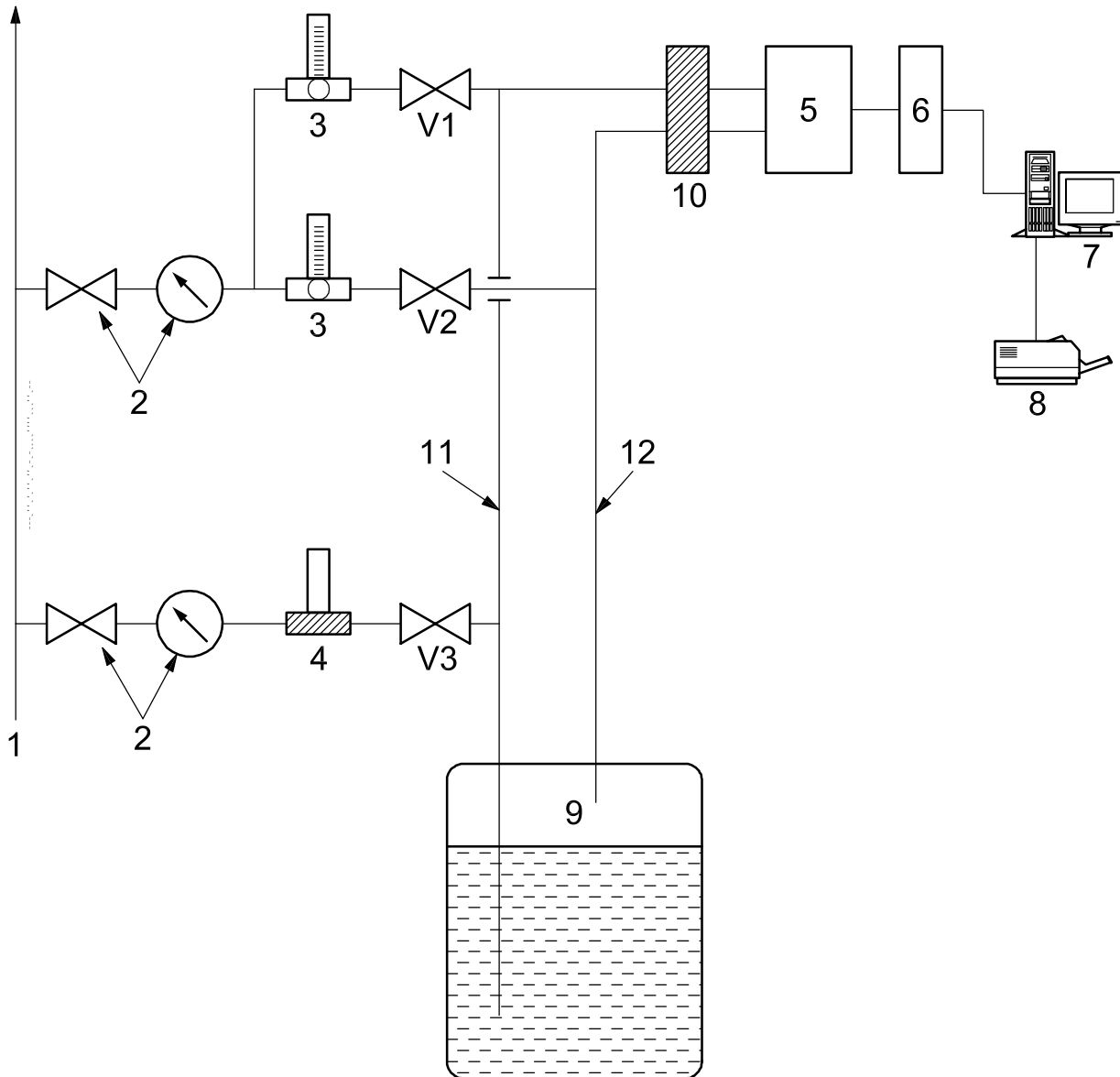
Before any measurement or series of measurements is made, the measurement system should be checked under normal conditions (the solution is at rest and its level is stable) to ensure that no leaks are present. The pressure readings in the major probe (immersed) should be stable for at least 10 min after valves V1, V2 and V3 are closed.

C.3 Procedure

- a) **Step 1:** keep (or set) both bubbling circuits in operation by keeping open (or opening) valves V1, V2 and V3.
- b) **Step 2:** ensure that the solution is at rest and that its level is stable:
 - turn off the cooling system; it should be turned off before the mixing system (agitator) is turned off to ensure that thermal equilibrium is achieved;
 - turn off the mixing system;
 - wait for oscillations in the liquid surface to subside (for instance approximately 10 minutes for a 16 m³ tank with a diameter of 3 m).
- c) **Step 3:** ensure that bubbling is taking place (by checking minimum and maximum pressures on the voltmeter display or on the graphical display produced by the computer software).
- d) **Step 4:** change to slow bubbling mode by closing valve V1. Use the software to check the bubbling rate and, if necessary, adjust the set point of the mass flowmeter to achieve a rate of 2 to 4 bubbles per minute.
- e) **Step 5:** initiate the steps to measure pressure for a series of 5 bubbles (see Clause 3). Examine the results with the aid of the data acquisition software. Repeat this step if the data are unsatisfactory for some reason (e.g. distorted bubble profile, large standard deviation).
- f) **Step 6:** measure the zero pressure. To do this, the two valves on the pipes are turned off (see Figure C.1, reference 10) and the internal valve (between the two inlets) is turned on. After the measurement, the internal valve should be turned on and the two others turned off.

Note that this measurement is useful for correcting the zero drift of the manometer (see Clause 3). The drift is not very fast and, if a series of measurements are being made (for example during a calibration run), the zero measurement can be made approximately every 2 h. After this measurement is made, it should be verified that no leaks are present in the pneumatic system.

- g) **Step 7:** print the pressure measurements for each bubble, especially the average and the standard deviation of all readings, the zero measurement, and the temperature of the solution. Save this information in suitable electronic form and, if possible, save all intermediate measurements. Ancillary data such as ambient temperature, barometric pressure, and *in situ* density measurements should also be recorded and stored.
- h) **Step 8:** change to fast bubbling mode by opening valve V1.



- Key**
- 1 air supply
 - 2 adjustment to 1,4 bar (140 000 Pa)
 - 3 rotameter for fast bubbling (~ 6 l/h)
 - 4 mass flowmeter controlled by a set point (~ 0,1 l/h)
 - 5 sensor
 - 6 voltmeter
 - 7 computer
 - 8 printer
 - 9 tank fitted with bubbling tubes
 - 10 pressure equilibrium chamber for zero measurement (same pressure in both probe lines)
 - 11 major bubbling tube
 - 12 reference bubbling tube
 - V1, V2, V3 valves

Figure C.1 — Typical unit for level measurement

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