INTERNATIONAL STANDARD

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

Part 1:
Procedural overview

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

Partie 1: Aperçu général de la procédure

Reference number
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Foreword

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ISO 18213-1 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 data to determine liquid volumes in process tanks for the purpose of nuclear materials accountability. This part of ISO 18213 complements the other parts, which include ISO 18213-2 (data standardization), ISO 18213-3 (statistical methods), ISO 18213-4 (slow bubbling rate), ISO 18213-5 (fast bubbling rate), and ISO 18213-6 (in-tank determination of liquid density).

Accurate determinations of volume are a fundamental component of any measurement-based system of control and accountability in a facility that processes or stores nuclear materials in liquid form. Volume determinations are typically made with the aid of a calibration or volume measurement equation that relates the response of the tank's measurement system to some independent measure of tank volume. 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. The steps carried out to acquire data for estimating the tank's calibration or volume measurement equation are collectively described as the process of tank calibration.

The methods presented in this part of ISO 18213 apply to tanks equipped with bubbler probe systems for measuring liquid content. With such systems, gas (air) is forced through a dip tube (probe) submerged in the tank liquid. Measurements of the pressure required to induce bubbling are used to determine the height of the column of liquid in the tank above the tip of the probe. During the calibration process, these determinations of liquid height are related to an independent measure of the tank's liquid content for some (calibration) liquid whose density has been precisely determined. An estimate of the volume measurement equation obtained from these data is subsequently used to determine process liquid volumes from measures of the pressure that these liquids exert at the tip of the dip tube.

This part of ISO 18213 is intended to serve as a procedural overview for the tank calibration and volume determination process, the main elements of which are presented. Selected steps that require further amplification are discussed in detail in other parts of ISO 18213 as noted.

Tank calibration and volume measurement data are sensitive to variations in measurement conditions and especially to changes in liquid and air temperatures. Therefore, it is necessary to standardize these data to a fixed set of reference conditions to minimize variability and ensure comparability. Standardization is required whenever measurement conditions change during a calibration exercise. Standardization is also necessary for comparing or combining data obtained during several calibration periods over which the measurement conditions are not constant. Finally, it is essential to standardize measurements of process liquid used to determine volumes for accountability purposes, because process measurement conditions are typically quite different from those that prevail during the calibration exercise. Data standardization steps are presented in ISO 18213-2.

A key step for both calibration and volume determination is to determine the height of a column of liquid above some reference point from a measure of the pressure that liquid exerts at the tip of a submerged probe. Procedures for making accurate liquid height determinations from pressure measurements are presented for slow and fast bubbling rates in ISO 18213-4 and ISO 18213-5, respectively.

Statistical methods for (i) examining the consistency of a set of data obtained during the calibration process, (ii) deriving an estimate of a tank's measurement or calibration equation from a set of calibration data and (iii) estimating the uncertainty of a volume determination obtained from this equation are presented in ISO 18213-3.

In tanks equipped with two or more dip tubes, the procedures of this part of ISO 18213 can be used to obtain (differential) pressure measurements for each probe. These measurements can, in turn, be used to make very accurate determinations of liquid density. Methods for making accurate determinations of density from in-tank measurements are presented in ISO 18213-6.

Taken together, the six parts of ISO 18213 provide a comprehensive state-of-the-art methodology that addresses all the factors known to significantly affect the uncertainty of volume determinations obtained by means of a tank calibration equation. This methodology can be used to produce high-quality calibrations for tanks from which very precise volume determinations are required, such as key input and output accountability tanks. For various reasons (inadequate instrumentation, lack of time or other resources), it might not be possible for an operator to meet all the prescribed conditions set forth herein, even for key accountability tanks. Moreover, it is typically not necessary for the operator to meet these conditions for all the tanks in a facility. Under these circumstances, this part of ISO 18213 provides a starting framework from which to develop a suitable "reduced" calibration model for each tank.

The first step for any calibration is to establish appropriate uncertainty limits for the resulting volume determinations. Next, each potentially significant factor is evaluated relative to its effect on calibration results, and specifically for its contribution to the total uncertainty of volume determinations (see ISO 18213-3:—, Annex D). A reduced model is obtained by ignoring factors found to have a negligible effect on total uncertainty in subsequent calculations pertaining to that calibration [possibly by fixing them at suitable constant values; see either ISO 18213-4:—, Annex A (slow bubbling) or ISO 18213-5:—, Annex A (fast bubbling) for examples]. Other factors are, of course, retained. Thus, for a key accountability tank for which very precise volume measurements are required, a suitable model retains (nearly) all potentially significant factors in subsequent standardization and uncertainty calculations. For tanks with less restrictive measurement requirements, a model that includes terms which involve only one or two of the most influential factors, such as temperature and density, is often sufficient. The user is reminded at numerous points throughout this International Standard that it is required of the user to determine whether or not to retain a particular variable.

INTERNATIONAL STANDARD **ISO 18213-1:2007(E)**

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

Part 1:
Procedural overview

1 Scope

This part of ISO 18213 describes procedures for tank calibration and volume determination for nuclear process tanks equipped with pressure-measurement systems for determining liquid content. Specifically, overall guidance is provided for planning a calibration exercise undertaken to obtain the data required for the measurement equation to estimate a tank's volume. The key steps in the procedure are also presented for subsequently using the estimated volume-measurement equation to determine tank liquid volumes.

The procedures presented apply specifically to tanks equipped with bubbler probe systems for measuring liquid content. Moreover, these procedures produce reliable results only for clear (i.e. without suspended solids), homogeneous liquids that are at both thermal and static equilibrium.

2 Physical principles involved

The pressure measurement systems for determining liquid height described in this part of ISO 18213 are based on the fundamental hydrostatic principle which states that the pressure, P, exerted by a column of liquid at its base is related to the height of the column and the density of the liquid as given in Equation (1):

$$P = gH_{\mathrm{M}}\rho_{\mathrm{M}} \tag{1}$$

where

H_{M} is the height of the liquid column (at temperature T_{m})[1];

ρ_{M} is the average density of the liquid in the column (at temperature T_{m});

g is the local acceleration due to gravity.

If the density of the liquid is known, Equation (1) can be used to determine the height of the liquid column above a given point from (a measure of) the pressure the liquid exerts at that point. Therefore, process tanks are typically equipped with bubbler probe systems to measure pressure. With a bubbler probe system, gas is forced through a probe whose tip is submerged in the tank liquid until bubbling occurs. At this point, the pressure exerted at the tip of the probe by the bubbling gas equals that exerted by the liquid column. The pressure required to induce bubbling is measured with a manometer located above the tank at some distance from the tip of the probe.

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[1] The subscript "M" is used to indicate the value of a temperature-dependent quantity at temperature T_{m}.

In practice, many factors 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 is quite sensitive to variations in temperature. Moreover, differences between the actual pressure at the tip of the probe and the observed pressure at the manometer can result from the buoyancy effect of air, the mass of gas in the probe lines, flow resistance, and the effects of bubble formation and release at the tip of the probe. A general algorithm for standardizing pressure measurements that compensates for temperature differences and other measurement factors is presented in ISO 18213-2. The pressure-to-height calculation step required for each measurement depends on the bubbling rate. The calculation is discussed in more detail in ISO 18213-4 and ISO 18213-5, respectively, depending on whether a slow or fast bubbling rate is employed.

3 The calibration model

The calibration equation for a process tank expresses the response of its measurement system (e.g. pressure or liquid height determined from pressure) as a function of its liquid content (e.g. mass or volume). The measurement equation, which gives the volume of the tank as a function of height, is the inverse of the calibration equation.

At a fixed reference temperature, T_r, the measurement equation, $V_\mathrm{r} = f^{-1}(H_\mathrm{r})$, gives the volume of the tank below some point at elevation, H_r, above a selected reference point (typically the tip of the major probe). The measurement equation can be written as given in Equation (2):

$$V_\mathrm{r} = f^{-1}(H_\mathrm{r}) = \int_{-\varepsilon}^{H_\mathrm{r}} A_\mathrm{r}(H)\,\mathrm{d}H \qquad (2)$$

where

H is the elevation of the liquid surface above the reference point;

$A_\mathrm{r}(H)$ is the free cross-sectional area of the tank (the cross-sectional area of the tank minus the area occupied by internal apparatus) at elevation, H, above the selected reference point (at temperature T_r);

ε is the vertical distance between the selected reference point and the lowest point in the tank.

Note that if the lowest point in the tank is chosen as the reference point, then $\varepsilon = 0$.

The form of the measurement equation given in Equation (2) is generally not used directly because the functional form of $A_\mathrm{r}(H)$ can be quite complex and estimates obtained from engineering drawings are not sufficiently accurate for safeguard purposes. Therefore, a calibration exercise is undertaken to obtain data from which a sufficiently accurate estimate of the height-volume relationship given by Equation (2) [or Equation (3)] can be made. The estimate of Equation (2) [or Equation (3)] derived from these calibration data is typically expressed in the form of several low-degree polynomial equations, each of which has been fitted to a particular segment of the overall calibration equation.

If a tank cannot be completely emptied, a calibration begins at some unknown elevation $H_0 > -\varepsilon$ determined by the residual liquid that remains in the tank (i.e. the tank's heel). In terms of H_0, Equation (2) can be written as Equation (3):

$$V_\mathrm{r} = V_0 + \int_{H_0}^{H_\mathrm{r}} A_\mathrm{r}(H)\,\mathrm{d}H \qquad (3)$$

where V_0 is the heel volume of the tank, as given in Equation (4):

$$V_0 = \int_{-\varepsilon}^{H_0} A_{\mathrm{r}}\left(H\right)\mathrm{d}H \tag{4}$$

If the tank can be completely emptied, then $H_0 = -\varepsilon$ et and $V_0 = 0$. In general, however, the tank cannot be completely emptied, in which case the heel volume, V_0, cannot be determined directly with the tank's measurement system. In this latter case, the heel volume cannot be measured as part of the calibration process (except possibly during the very first calibration run, and then only if the tank is initially empty), so it is necessary to determine it in some other manner (see 6.6.6 and ISO 18213-2:2007, Annex C).

4 Equipment required

4.1 General

For accountability purposes, a tank's liquid content is measured in order to determine its volume. This requires that the tank first be calibrated, i.e. that the relationship between the elevation of a given point in the tank and the volume of the tank below that point be established. During the calibration process, increments of some calibration liquid of known density are added to the tank. The content of each increment is measured (independently of the tank's measurement system) and, after it is added to the liquid in the tank, the corresponding response of the tank's measurement system is observed. The independent measurements of tank content are obtained by means of a suitable prover system. The tank's measurement and measurement support systems are discussed in 4.2. The major components of a calibration system, which consists of the prover system, the calibration liquid and the requisite software, are discussed in 4.3, 4.4 and 4.5, respectively.

4.2 The tank and its measurement systems

4.2.1 Overview

The elements of a typical pressure-based measurement system for determining liquid content (height) are shown schematically in Figure 1. These include the tank, its bubbler probes, its temperature probes and the manometer(s) used to measure pressure. Figure 1 also gives the nomenclature that is used throughout the six parts of ISO 18213.

The bubbling pressure depends not only on the height of the 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 the gas in the major (or minor) probe and the pressure of gas in the reference probe.

In the configuration shown in Figure 1, the major (minor) probe is connected at the high-pressure side of the manometer and the reference probe is connected at the low-pressure side. This configuration, although typical, is not the only one possible. In another widely-used configuration, for example, the major probe is connected at the high-pressure side of the manometer while the minor and reference probes are connected at the low-pressure side. Minor modifications in the methods and nomenclature of this part of ISO 18213 can be required when these methods are applied to configurations differing from that shown in Figure 1.[2]

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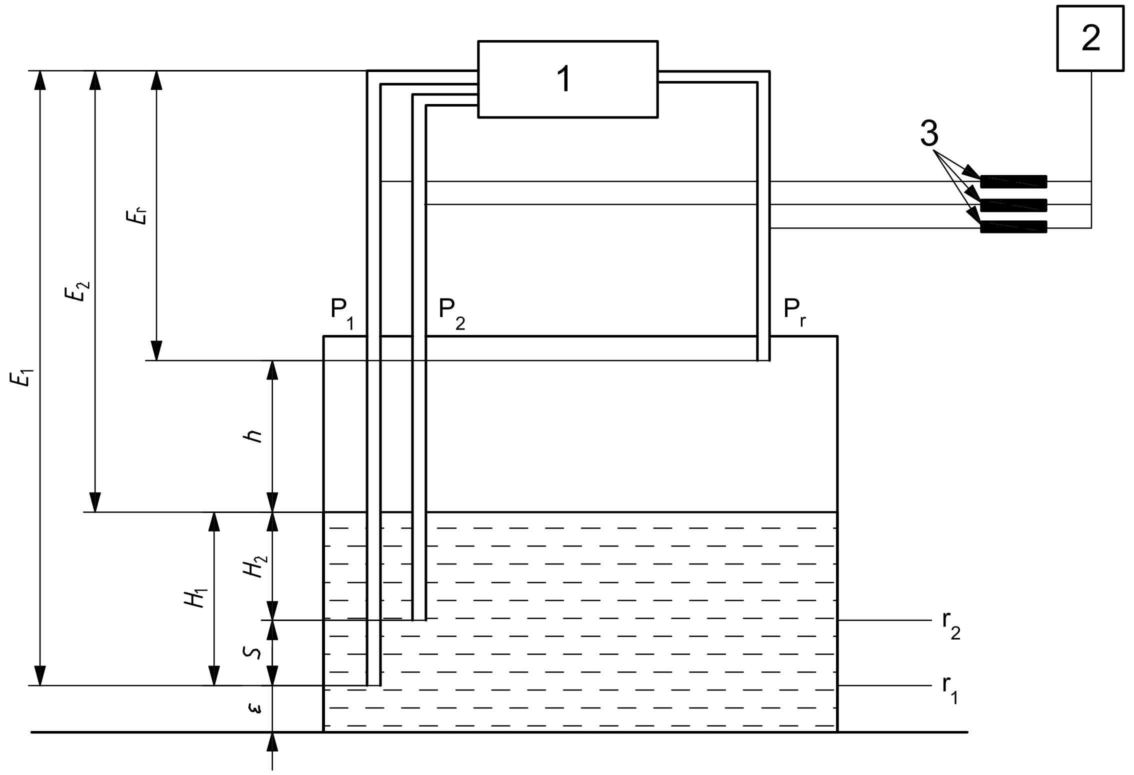
[2] The advantage of the configuration shown in Figure 1 is that, once the minor probe is submerged, it yields duplicate measures of liquid height. The alternative configuration yields one measure of liquid height and a measure of the difference in pressure between the major and minor probes.



Key

1 manometer

2 gas supply (N_2 or air)

3 flowmeters

Probe	Major probe	Minor probe	Reference probe
Probe designation	P_1	P_2	P_r
Reference point	r_1 (primary)	r_2 (secondary)	—
Height of the liquid above the reference point	H_1	H_2	—
Elevation of the pressure gauge (manometer) above the reference point	E_1	E_2	E_r
Elevation of the 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 the tank	ε	$\varepsilon + S$ [a]	—

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

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

4.2.2 Tank

The tank in which liquid height is measured should be equipped with at least two tubes (probes) of small diameter (< 15 mm). One of the probes (the major probe) should extend as close to the bottom of the tank as possible (without touching it). This probe should be rigidly mounted so that its position relative to the tank is fixed and it is not in contact with any point on the wall of the tank. The second probe (reference probe) shall also extend into the tank, but it should be as short as possible (or mounted on the vent pipe), so that its tip is above the maximum filling level.

Each probe should be connected to two rigid tubes (pipes) of small diameter, one of which is connected to a gas supply and one of which is connected to a pressure gauge (manometer). The two tubes for each probe should be of the same diameter and as close to the same length as possible (and preferably co-located). The tubes should be installed (mounted) so that they are not subject to vibrations that can adversely affect the measurement quality.

Changes in temperature can significantly affect the reliability of data for calibration and volume determination, especially through their effect on liquid density. Therefore, the tank should be equipped with temperature probes that are calibrated to ensure measurements with an accuracy of at least 0,5 °C.

The tank shall also be equipped with instrumentation (spargers, agitators, etc.) to ensure that its contents are homogeneous and at uniform temperature at the time of measurement. These instruments shall be capable of operating at rates that maintain the homogeneity and uniformity of the liquid without causing excessive motion or evaporation during a calibration run. It shall be possible to turn these instruments off on demand to make the necessary measurements.

To ensure stable measurement conditions, the tank, together with its operating and measurement systems, should be isolated insofar as possible from other elements (e.g. surrounding tanks) of the plant process.

4.2.3 Manometers

Tanks equipped with pressure measurement systems for determining liquid content shall also be equipped with manometers for measuring the pressure of the bubbling gas flowing through the probe lines. The selected manometer(s) should be equipped with a digital readout or connected to a digital voltmeter so it/they can be interfaced electronically with other components of the tank measurement and calibration systems.

The manometer system shall be sensitive enough to measure pressure with sufficient precision to meet safeguard requirements imposed on the tank. If it is necessary, for example, to resolve 1-l volumes, this in turn imposes a requirement on manometer resolution (see 6.3.3). Generally, a manometer system that can resolve pressure differences of 1 Pa to 2 Pa or less is suitable for safeguards purposes. The manometer system should also have a differential range that is appropriate for its intended use. While a manometer with a differential range of 50 000 Pa can be required for a large input tank, one with a narrower range of 20 000 Pa can be suitable for a smaller output tank.

The electronic acquisition and transfer of data is important both for eliminating data recording errors and for ease of operation, especially during calibration exercises (see 4.5). The system should be capable of measuring continuously, or with a frequency of at least 5 Hz.

4.2.4 Bubbling gas

A supply of gas is required. The supply shall be sufficient, not only to maintain flow in the reference probe that vents into the tank above the liquid surface, but also to maintain bubbling at the tip of the submerged probe(s) throughout the established measurement periods. Instrumentation for delivering and controlling the flow of gas through the probe lines shall be capable of maintaining a constant flow rate during calibration and measurement activities. The delivery system should allow the gas to reach thermal equilibrium within the facility so that large thermal gradients are avoided.

The selected bubbling gas shall be inert with respect to the calibration and process liquids. Moreover, a gas should be selected whose physical properties (especially density) are well known so that necessary standardization calculations can be carried out (see ISO 18213-4 or ISO 18213-5). Compressed air and nitrogen are widely used in practice. Nitrogen is easy to use. Compressed air, on the other hand, has the advantage that many of the data-standardization calculations are somewhat simpler (see ISO 18213-2), provided that air is compatible with the process. When selecting a bubbling gas, it is useful to consider that dry air has the tendency to increase evaporation, whereas saturated (wet) air has the tendency to increase condensation.

With fast bubbling rates, flow rates between 6 l/h and 20 l/h are typically used during measurement periods. The optimal flow rate depends on the diameter of the dip tube: greater flow rates are required for dip tubes of larger diameter. A mass flow meter whose set point can be fixed at 0,1 l/h is required for making measurements at slow bubbling rates (see ISO 18213-4:—, Annex C).

4.2.5 Ambient conditions

Measurements for tank calibration and volume determination are sensitive to changes in ambient conditions. Therefore instrumentation is required to measure ambient temperature, barometric pressure and relative humidity. Data on ambient conditions are required to standardize a series of measurements to a fixed set of reference conditions (see ISO 18213-2).

4.3 Prover system

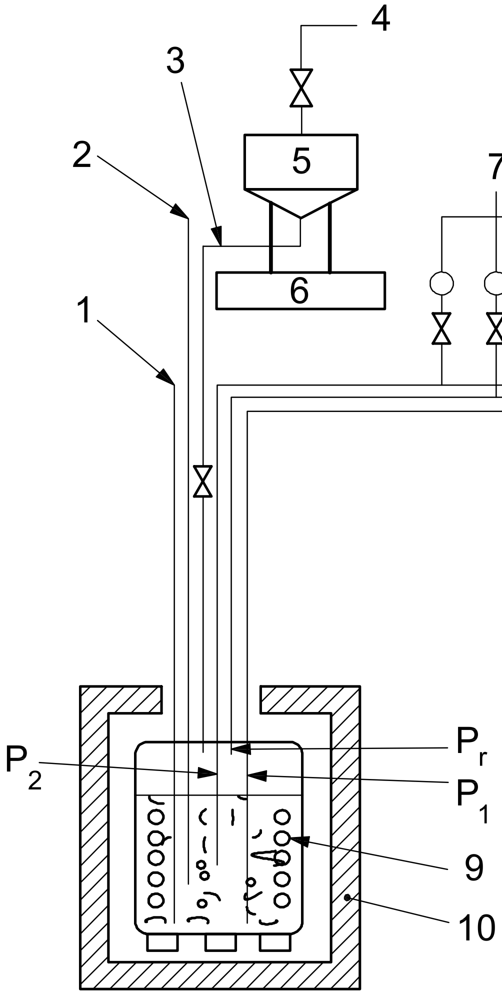
One of two basic prover systems, gravimetric or volumetric, may be employed to make independent measurements of the liquid added to the tank via the calibration process. A gravimetric prover, which is essentially a container on a scale, provides a measure of the mass of liquid added to the tank. A volumetric prover system consists of one or more containers of differing capacity, each of which has been fabricated to deliver a single fixed volume of liquid at some predefined temperature. A combined gravimetric-volumetric prover system (essentially a volumetric prover on a scale) may be employed to provide a redundant measurement capability. Gravimetric systems are more widely used in practice, but it is possible to obtain high quality calibration measurements with either system. A typical tank calibration setup is shown schematically in Figure 2.

Several considerations enter into the selection of a suitable prover system for tank calibration. The prover system shall not only be capable of delivering calibration increments in sizes that are consistent with the capability of the tank's measurement system, but it shall also deliver increments that meet other accountability requirements and procedural constraints.

Increment size(s) should be large enough to affect a change that is at least five times the resolution of the tank's measurement system (see 6.3.3), but small enough to permit adequate resolution of important structural features in the tank (see 6.6.3).

Subject to this system resolution constraint, it is generally desirable to plan for as many calibration increments as time and resources allow. For larger tanks, the total time required for a calibration run can become a consideration. The selected prover system should be designed to fill and empty rapidly enough to deliver a sufficient number of increments (at least 50 and preferably more) to obtain the required resolution within approximately 12 h (see 5.2).

To meet resolution and time constraints, it can be necessary to use increments of several sizes during a calibration run. For this purpose, it is possible to construct a volumetric prover system that delivers a range of increment sizes by fabricating several different-sized containers, each of which delivers a single, fixed volume of liquid. However, the change from one container to another can be time-consuming, especially if it is necessary to disconnect and reconnect drain lines. Moreover, if it is necessary to move calibration containers, they shall be leveled after each move. These inconveniences can be circumvented with a gravimetric prover which, being essentially a container on a scale, can deliver a continuous range of volume increment sizes. Another advantage of a gravimetric prover is that it is possible to make multiple readings for each measurement. On the other hand, a gravimetric prover is sensitive to environmental conditions (e.g. air currents) and requires two measurements for each calibration increment (e.g. the mass of the container before and after the increment is delivered to the tank). Regardless of which type of prover system is selected, however, the decision to use several increment sizes during a calibration should be made with care because statistical analysis of the data can be more difficult when different-sized calibration increments are used.



Key

1	liquid temperature probe(s)
2	process lines (vent, fill, empty, decontamination, sparge, sample, etc.)
3	supply-line calibration liquid
4	callibration liquid supply
5	prover vessel
6	scale
7	purge gas supply
8	differential pressure manometers
9	tank internals (coils, braces, agitator, etc.)
10	isolation barrier
P_1	major probe
P_2	minor probe
P_r	reference probe
[a]	Level 2 (“density”)
[b]	Level 1 (“level”).

Figure 2 — Elements of a typical tank calibration setup

4.4 Calibration liquid

A supply of calibration liquid sufficient to complete a planned calibration exercise, which can consist of several calibration runs, is required. It is desirable to move the liquid into the facility far enough in advance of the calibration exercise for its temperature to equilibrate with that of its surroundings.

Factors that should be considered when selecting a calibration liquid are discussed in 6.5.4.

4.5 Calibration software

It is strongly recommended that the tank calibration and volume measurement systems be connected to a computer that controls the operation of the calibration process and processes the acquired data. A computerized system can help to simplify the calibration procedure, improve the consistency with which it is carried out and substantially reduce the work involved in performing a calibration exercise. At a minimum, a computer should be used for acquiring the data from the tank's measurement instruments (manometers) and transferring it to a suitable data base for analysis. The ability to acquire and transfer calibration data electronically eliminates the possibility of transcription errors and reduces the effort required for data verification, especially if it is necessary to assemble the calibration data set from data collected at several reading stations. The development of a program that can control the entire calibration operation, acquire and record the required data, perform the necessary data standardization operations, and provide on-line checks of data validity is strongly recommended.

Calibration software should be capable of accepting multiple instrument readings for each calibration measurement (see 6.6.4). This capability is particularly important for instruments that measure the primary calibration variables, such as manometers (that measure pressure) and scales (that measure mass). With multiple measurements, the effect of one or two erroneous readings can be minimized or eliminated. Moreover, replicate measurements are required to estimate the statistical properties of the measurement process. Although it is helpful to compute summary statistics (e.g. average and standard deviation) for each set of multiple readings at the time they are made, it is advisable to archive all individual readings for subsequent detailed analysis. All measurements are sensitive to temperature variations, so the software should be capable of recording the temperature of the relevant liquid in both the tank and the prover for each calibration measurement. Finally, the software should be capable of recording and archiving information on ambient conditions, such as temperature, barometric pressure and relative humidity (see 6.6.2).

It is recommended that the data standardization calculations (see especially ISO 18213-2, ISO 18213-4, and ISO 18213-5) be done as the data are acquired. This practice is not only convenient, but also helps to provide procedural consistency. Moreover, comparisons involving data collected under varying measurement conditions are more reliable if the data are standardized to a fixed set of reference conditions before the comparison is made. Although special-purpose software has been developed for data standardization, all of the calculations indicated in ISO 18213-2, ISO 18213-4, ISO 18213-5, and ISO 18213-6 can be done with the aid of commercially available software.

With more advanced special-purpose calibration software, procedures can and should be established for routinely checking the validity of the data collected for each volume increment as they are acquired. Such validation procedures should, for example, ensure that the prover is filling and draining properly. A variety of interrelated consistency checks involving liquid height and volume measurements can be devised for a particular calibration system. These checks serve to identify erroneous readings and can provide an early indication of possible instrument malfunctions.

5 A typical tank calibration procedure

5.1 Calibration procedure

With adequate preparation, the procedural steps involved in conducting a tank calibration run are relatively straightforward. A tank may be calibrated either by making incremental additions of liquid to it or by making incremental removals of liquid from it. Both methods are equally valid. However, for the sake of clarity and because the practice is more common, it is assumed that calibration is done by means of incremental additions. Under this assumption, a calibration run involves making a series of additions of carefully measured quantities of calibration liquid to the tank and, after each addition, recording the measurement system response, the temperature of the liquid in the tank and current ambient conditions (or changes therein). As indicated in 4.5, appropriate data standardization calculations may be performed in connection with each increment at the time of acquisition.

The following steps are carried out during a typical calibration run.

a) Preparatory steps are carried out. These include isolating, flushing, and drying the tank, and performing a pre-calibration leak test of the pneumatic systems.

b) Initial steps are completed. These include such activities as making a final system check, zeroing measurement instruments, and recording data on reference and ambient conditions.

c) The prover is filled with calibration liquid.

d) Prover-related data are recorded as specified in the calibration protocol (see Clause 4 and 6.7). For a gravimetric prover system, the before-delivery mass of the prover and calibration increment is required. For a volumetric prover, the volume and the temperature of each calibration increment are required (see Clause 4 and 6.5). Ambient conditions or changes therein are also recorded.

e) The liquid is transferred from the prover to the tank and sufficient time is allowed for drainage (see procedural notes in 5.2 below). For a gravimetric prover system, the after-delivery mass of the prover is recorded.

f) Sufficient time is allowed for conditions in the tank to stabilize. Steps, such as mixing and waiting for the release of trapped gas, are taken to ensure that the liquid in the tank is homogeneous and free of significant thermal gradients. After mixing, sufficient time is allowed for the liquid level to reach static equilibrium (see 5.2 below).

g) Measurements required to determine tank content are made. The response of the tank's measurement system, the temperature of tank liquid, and other information required to clarify or interpret these data are recorded.

h) Steps 3 to 7 are repeated for each calibration increment specified in the calibration plan (see 6.7). Typically, the increments are designed to calibrate the entire tank or a particular region of interest. The region of interest and the sizes of the calibration increments are specified in a comprehensive calibration plan.

i) Closing verification and confirmatory steps such as leak tests (see 6.3.5) are conducted.

A tank calibration exercise typically consists of several calibration runs. A single run can be sufficient to verify an existing calibration or volume measurement equation.

If the tank cannot be completely emptied and dried between calibration runs, it can be necessary to determine its heel volume prior to the start of the calibration exercise (see Clause 3 and ISO 18213-2:2007, Annex C).

5.2 Procedural notes

The various waiting times (between increments, after mixing, etc.) encountered during the calibration process depend on factors that are specific to a particular tank, its operating systems, and the selected calibration system. In preparation for the calibration exercise, appropriate waiting times should be determined experimentally within each facility, and suitable measurement stability criteria should be incorporated into the calibration software.

Excessive mixing and waiting times should generally be avoided to minimize evaporation losses, changes in measurement conditions, and the effects of condensation.

A calibration run should continue to completion without major interruptions or time delays after it has been started. Calibration runs of more than 12 h should generally be avoided unless special precautions are taken to minimize the effects of evaporation and changes in ambient conditions.

The key to a successful calibration exercise is a complete and comprehensive calibration plan. Items that should be addressed in a comprehensive calibration plan are discussed in Clause 6.

6 Calibration planning and pre-calibration activities

6.1 The calibration plan

The goal of the calibration exercise is to obtain a high-quality set of data that can be used to estimate a tank's calibration or volume measurement equation, together with the necessary ancillary data to analyze and interpret these calibration data. Thorough planning is crucial to the achievement of this goal and its importance cannot be over-emphasized. The calibration planning effort should culminate in a detailed written calibration protocol (calibration plan) that specifies both the procedural details for the calibration steps of the previous section and the conditions under which they are carried out. Careful planning, as reflected in the calibration protocol, establishes a context in which the required measurement accuracy for the calibration (or volume measurement) equation can be weighed against the effort devoted to the calibration exercise.

A comprehensive calibration planning exercise should include each of the following:

- assessment of the required measurement accuracy and a preliminary error analysis;
- review of the tank and its measurement (manometer) and support systems;
- preparations for calibration of the tank calibration equipment (prover system);
- specification of reference operating conditions under which calibration measurements [e.g. pressure (tank) and mass (prover)] are to be made;
- preparations and plans for the acquisition, verification and subsequent detailed analysis of calibration data.

Finally, the calibration plans should culminate in a pour schedule that specifies the number and size of the calibration increments to be made during each calibration run.

In addition to the items specifically listed above, the calibration plan should address any other factors that are specific to the particular calibration exercise being planned. In short, the planning exercise should result in a thorough understanding of the tank, its measurement and support systems, the calibration equipment and the relationships among them. This understanding is the basis for a smooth calibration exercise.

6.2 Measurement requirements and preliminary error analysis

A clear statement of the required volume measurement accuracy should be developed for the tank. The maximum acceptable error limits for volume determinations should take account of the tank's role in the overall accountability plan for the facility, a major consideration being the amount of nuclear material involved. For primary accountability tanks, relative standard deviations as small as 0,1 % or less may be prescribed for individual volume determinations. Such limits are achievable with state-of-the-art measurement systems operated under favourable conditions. Of course, if specified error limits subsequently prove to be inconsistent with system capability, some corrective action (e.g. upgrading the measurement system or relaxing measurement requirements) can be required.

A preliminary error analysis should be performed after a review of the tank and the calibration system. This analysis is an item-by-item assessment of measurement uncertainty for the components of the volume-measurement-and-tank-calibration process.[3] This preliminary error analysis is intended to identify possible inconsistencies between measurement capability and required measurement accuracy. The analysis should also help to identify any system components or procedural steps that require special attention because they are particularly vulnerable to error. Whenever possible, uncertainty estimates should be based on in-plant observations that reflect actual operating conditions, rather than upon manufacturer's specifications or other "standard" estimates. The choice of an appropriate statistical model will depend on the relative magnitudes of uncertainties associated with specific error sources.

6.3 The tank and its measurement support systems

6.3.1 Engineering review

Personnel responsible for tank calibration should perform an engineering review of the tank to become familiar with its construction and installation; its physical environment; and the operation of its measurement, transfer, and support systems. The entire measurement domain of the tank, which includes all operating and support systems that can affect measurement system response, should be evaluated during the engineering review. Plans should also be made at this time for isolating the tank during the calibration exercise.

6.3.2 Equipment checkout

The integrity of the tank, its process and instrument lines, and all calibration equipment should be verified prior to the calibration exercise. A checklist should be compiled to ensure that necessary preliminary operation steps (e.g. flushing the tank to be calibrated and isolating it from adjacent tanks) are performed prior to the start of a calibration exercise and that all normally active auxiliary systems (off-gas service, bubbler air flow, etc.) are operational and operated according to standard procedures during the calibration. The availability of suitable connections to the tank should be checked, and steps should be taken to ensure that temperatures will be reasonably stable during calibration.

Transfer procedures should be reviewed to identify possible points of hold-up in sparge, sampling and cold chemical lines. If possible, all hold-up should be eliminated, especially in transfer lines used for calibration. Otherwise, plans should be made to estimate the volume of liquid at hold-up points or to otherwise control the effect of hold-up on liquid height and volume determinations (see 6.4.3).

6.3.3 Measurement system resolution

The resolution of the tank's measurement (manometer) system is required not only for the preliminary error analysis, but also to establish a lower limit for the size (volume) of a calibration increment. The product of the measurement system resolution and the cross-sectional area of the tank gives the detection threshold (minimal detectable volume) for the tank and its measurement system. The size of minimum calibration increment should, in turn, be at least five times the detection threshold (see 4.3). For example, in a tank with a cross-sectional area of 2 m^2 and a measurement system capable of resolving a 0,5 mm column of water (approximately 5 Pa), the detection threshold is approximately 1 l, so the volume of a calibration increment (of water) should be at least 5 l.

6.3.4 Tank profile

Potential transition regions (regions of the tank in which the cross-sectional area changes rapidly with height) should be identified during the engineering review because they can require special attention during a calibration run. Knowledge of transition regions is also helpful for developing the tank's calibration or volume measurement equation because calibration segments are determined largely by examining the tank's profile to identify transition regions. The locations of any protrusions and internal equipment are especially helpful and can be obtained from "as-built" engineering drawings of the tank. These points can require special treatment (e.g. smaller calibration increments) during the calibration and they can also serve as reference

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[3] See ISO 18213-3:—, Annex C, for guidance in conducting a preliminary error analysis.

points for aligning the data from several runs. Alignment is necessary if the tank cannot be completely emptied because successive calibration runs begin at an unknown level that varies from run to run (see ISO 18213-2:2007, Annex D).

6.3.5 Leaks

All leaks shall be eliminated from pneumatic lines, the transfer and sparging systems and the tank itself prior to the start of a calibration run. The effect of the leaks cannot be adequately quantified, so all calibration data acquired when detectable leaks are present shall be declared invalid and discarded. Therefore, a leak test should be performed (i) before each calibration run to verify that leaks are not initially present and (ii) after each run to verify that calibration data have not been invalidated by leaks which appeared during the run. Pneumatic lines can easily be checked for air leaks without transferring liquid simply by turning off the air flow to the probes and observing whether the pressure readings remain constant.

Specifically, the following procedure can be used to check for leaks in pneumatic lines at the start of a calibration run. First, the tank is emptied (if it is not already empty). Next, the air flow meters are balanced and enough liquid is added to the tank to cover normally submerged probes. Then, the air to the probes is turned off and the observed pressures are recorded. A leak is indicated if the readings do not remain constant throughout a time period of 15 min to 20 min or more.

Similarly, leaks can be detected at the end of a calibration run by observing pressures for the nearly filled tank for an extended period of time. Instruments such as the air sparge and the recirculating sampler should be activated in accordance with established operating procedures during the observation period. However, extended operation of the tank’s sparging, off-gas, and sampling systems should be avoided during leak testing because these practices can also cause a decrease in pressure (primarily due to evaporation losses). Any unexpected changes in pressure during this test indicate the presence of leaks. If several calibration runs are planned in succession, it can be procedurally convenient to take the leak test at the end of one run as the initial leak test for the next run.

Leak tests are not conclusive unless the tank’s pneumatic systems can be isolated from other plant systems and activities. Moreover, when a leak is indicated, the leak test cannot distinguish the source of the leak. Additional testing can be required to find a particular leak because leaks in the tank, in its operating systems, and in pneumatic lines all produce a decrease in pressure.

6.3.6 Instrument calibration

Instruments used to measure volume, pressure, temperature, and related ancillary variables should either be in calibration at the time of a tank calibration exercise or be calibrated prior to its start. In planning for the calibration of these instruments, the manufacturer’s manuals and required equipment should be procured as necessary.

6.4 Calibration equipment (prover system)

6.4.1 Equipment review

In preparation for a calibration exercise, personnel should become familiar with the prover system. Personnel should also become familiar with existing equipment for calibrating provers or make plans to acquire new calibration equipment, as appropriate.

6.4.2 Prover calibration

If the prover system is not in calibration at the time of the planned tank calibration exercise, arrangements should be made to calibrate it prior to the start of the exercise. For a gravimetric prover, this involves calibrating the scale(s) used to measure volume increments. Calibration of a volumetric prover involves making a very precise determination of its contained volume at some reference temperature. A facility may choose to calibrate its own provers, or to engage a recognized outside agency (e.g. a state or federal bureau of weights and measures). Either alternative is acceptable, provided that recognized calibration procedures are used and valid measures of uncertainty are given for the values assigned to test measures. Typically, a scale calibration is done on-site by the appropriate instrument group at the facility and a volumetric prover is calibrated off-site by a recognized state or national authority.

For in-house calibrations, only test weights and instruments whose calibration and uncertainty values are traceable to suitable national or international standards should be used. These weights and instruments should be calibrated prior to their use for prover calibration and all certifications should be valid at the time the instruments are used. All weighings made for calibration should be corrected for the effect of air buoyancy (see ISO 18213-2:2007, Annex B).

Calibration procedures should be reviewed to ensure that they are compatible with the method to be used for transferring calibration liquid to (or from) the tank. For example, to calibrate a tank by means of incremental additions, the prover should be calibrated for the volume it delivers. Regardless of the decision to calibrate scales and provers in-house or elsewhere, a schedule should be established that allows ample lead time.

6.4.3 Location of equipment

The prover should be located as close to the tank as possible and should be maintained in a level position. All calibration equipment should be located where it will not be moved or disturbed during a calibration exercise, and should be installed so that it is free of vibrations and electronic interference. Lines used to introduce calibration liquid into the tank should be completely free of the prover and should not come in contact with it.

Lines used to deliver calibration liquid from the prover to the tank should be arranged so that there is no liquid holdup. If this is impossible, an initial increment to wet lines and fill holdup points should be considered. It is highly desirable to jet excess liquid introduced by this “wetting” increment from the tank before the start of the calibration run.

6.5 Reference operating conditions

6.5.1 General

To ensure the comparability of a series of calibration measurements, steps should be taken to ensure that consistent operating procedures are maintained throughout the measurement period. Moreover, insofar as possible, steps should be taken to ensure that measurements of process liquid are made under the same conditions that prevail at the time of calibration (or *vice versa*). The goal is to minimize measurement variability by standardizing operating procedures and minimizing variability in ambient conditions.

Factors that can affect measurement system response are identified in this clause. Suitable operating procedures are discussed, and possible corrective actions are suggested to compensate for variations in ambient conditions that cannot be controlled.

6.5.2 Operating variables

Procedures should be established to ensure that standard settings on all operating and support systems are maintained throughout the calibration process. The same settings used for calibration should subsequently be used for measurements made to determine process liquid volumes. Operational factors that can affect measurement system response include the gas purge (flow) rate, off-gas vacuum, air sparge and the elevation of the manometer above the tank.

6.5.2.1 Gas purge rate

Gas purge is the flow of gas (e.g. air) through the pneumatic lines. For fast bubbling rates, flow resistance can cause back pressure in the lines that can bias measurement results. The back pressure depends upon the purge (flow) rate and the length and diameter of the lines. To minimize the effect of flow resistance, all accountability measurements should be made at a fixed flow rate. In particular, all measurements of pressure for calibration or volume determination should be made at the same flow rate that is used during routine plant operation.

If pressure drop due to flow resistance is found to be significant, corrections can be calculated as indicated in ISO 18213-5:—, Annex A. Corrections for flow resistance should be considered unless pneumatic lines are sufficiently large (at least 5 mm or 0,25 in in diameter), the purge rate is relatively small (less than 20 l/h or 0,75 f^3/h under standard conditions), and line configurations are identical for calibration and volume measurements. Corrections should be made for tall tanks (4 m or more), especially if they are expected to receive liquids (including the calibration liquid) that vary significantly in density (e.g. by more than 50 %). Regardless of their importance for a particular situation, it is good practice to apply the corrections indicated in ISO 18213-5 to all measurements as a matter of course.

6.5.2.2 Off-gas vacuum

Fluctuations in off-gas vacuum can cause anomalous or highly variable measurement system responses. Therefore, pressure readings should be made only when the off-gas vacuum is stable. Vapour head (reference) pressure should be established relative to an outside reference and monitored to ensure that pumping operations in adjacent tanks do not affect pressure measurements.

6.5.2.3 Air sparge

The air sparge should be off when pressure readings are made to ensure that calibration measurements are not excessively variable. If sparging is necessary, the air sparge should be operated for as short a time as possible to minimize the loss of calibration liquid by evaporation. To minimize measurement times, it is convenient to operate the sparge while calibration liquid is added to the tank.

6.5.2.4 Manometer elevation

The difference between the pressure at the tip of the probe and at the manometer depends not only on the purge rate, but also on the mass of the purge gas in the line and the elevation of the manometer above the tip of the probe. Manometers used to measure pressure for tank calibration or volume determination should be kept at a constant elevation to minimize these effects.

Corrections that compensate for the mass of gas in the probe lines and differences in manometer elevation are given in ISO 18213-4 or ISO 18213-5 for slow and fast bubbling rates, respectively. These corrections should be made if the elevation of the manometer above the tip of the probe is large (e.g. greater than 1 m). Regardless of the elevation difference in a particular situation, it is good practice to apply the corrections indicated in ISO 18213-4 or ISO 18213-5 to all measurements as a matter of course.

6.5.3 Temperature

Temperature variations have a significant effect on measurement results because they affect nearly all aspects of the measurement process. Temperature changes affect both the response of the tank's measurement system and the prover system. They affect the dimensions of the tank and, more importantly, they affect the densities of all liquids involved in the tank calibration and volume determination process. The combined effect of these changes can be quite complex.

Even moderate temperature changes can have a significant adverse effect on a series of calibration measurements unless changes in liquid density are taken into account. For example, a change of 3 °C in the temperature of water near 25 °C induces a change of nearly 0,1 % in its density. Failure to compensate for this change directly affects the height calculations based on Equation 1. This effect is transmitted in turn to the volume determinations made with the calibration (measurement) equation derived from these height determinations. Therefore, it is important to use an accurate measure of the density of the liquid at the time it was measured (i.e. at its measurement temperature) when calculating liquid height from pressure (see 6.5.4).

It can be difficult to maintain a constant temperature for a series of measurements and temperatures can differ significantly between two measurement periods (e.g. between two calibration runs). Therefore, a pre-selected reference temperature should be established and provision should be made to routinely adjust all measurements of liquid content to this reference temperature. The statement applies equally to calibration measurements and to measurements of process liquids made for accountability purposes. It is convenient to select a reference temperature, such as 25 °C or 30 °C, that is close to the ambient temperature in the facility or in the laboratory.

A procedure is presented in ISO 18213-2 for standardizing a set of calibration data to a fixed reference temperature. One step in the algorithm is an adjustment to each measurement that compensates for dimensional changes in the tank and its dip tubes caused by temperature variations. To implement the algorithm, a measurement of liquid temperature is required for each measurement of liquid content. As noted above, it is good practice to routinely apply the standardization corrections indicated in ISO 18213-5 to all measurements as a matter of course.

6.5.4 Liquid density

According to Equation 1, the height of a column of liquid, H, is directly proportional to P/ρ_M, the ratio of the pressure that the liquid exerts at the base of the column and its density (the constant of proportionality is g^{-1}). Since the density of a given liquid is very sensitive to changes in temperature, a crucial pre-calibration planning step is to select a calibration liquid whose density is adequately characterized.

Any liquid may be used for calibration, provided that it meets certain criteria. First, it shall be compatible with the process liquids in the tank. Second, the liquid should be safe to use. Third, an adequate supply should be readily available. Finally, the selected liquid shall be one whose density either is or can be adequately characterized. This means that the density of the liquid either has been or can be determined with sufficient accuracy at all measurement temperatures so that density measurement uncertainty does not have a significant adverse effect on volume measurement uncertainty. It is desirable, but theoretically not necessary, to choose a liquid whose density does not differ too much (e.g. by more than 50 %) from the density of the process liquids the tank will contain.

De-mineralized water is widely used for calibration because operating risks are minimal and its density has been well characterized. Tables of the density of water are available at a wide range of temperatures [7] and convenient formulas have been developed for computing the density of water at all temperatures of interest (see ISO 18213-2:2007, Clause 3, and ISO 18213-2:2007, Annex A, ISO 18213-4:—, Annex A, or ISO 18213-5:—, Annex A). Alternative liquids, such as nitric acid and uranyl nitrate, may be used in situations where water is incompatible with the process, provided they meet the criteria given in the previous paragraph.

Steps should be taken to ensure that calibration data are not biased by differences in density between the calibration liquid and the tank's heel. The bias may be eliminated in either of two ways: (i) by determining the volume (see ISO 18213-2:2007, Annex C) and density of the heel so that the volume and density of the tank's contents can be accurately determined when calibration liquid is introduced, or (ii) by eliminating any liquid from the tank whose density differs from that of the calibration liquid. The latter alternative involves flushing the tank thoroughly with calibration liquid and jetting this liquid from the tank prior to the start of the calibration. The latter alternative has the advantage that the liquid in any remaining holdup regions is replaced with calibration liquid of known density (see 6.4.3).

When a tank is calibrated with some liquid other than process liquid (e.g. water), it is good practice to verify the calibration with process liquid (e.g. unanyl nitrate) to ensure that the calibration does not introduce undetected biases or other measurement anomalies.

6.6 Data acquisition and analysis

6.6.1 Completeness and accuracy

The completeness of the data and the accuracy with which they are recorded affect the computational effort required for data standardization and analysis. More importantly, they also affect the reliability of the resulting estimate of the tank's calibration or measurement equation. The acquisition of data should be planned to produce a complete and accurate set of data for each calibration run that includes the primary calibration variables, together with all ancillary information needed to interpret these data.

6.6.2 Required data

Raw calibration data are collected from one or more calibration runs. Each calibration run consists of a series of incremental additions (or removals) of calibration liquid to the tank. Additions typically continue until the tank is filled. Basic calibration data to be acquired for each increment include a measure of its mass (or volume), the corresponding response of the tank's measurement system and the temperature of all liquids at the time of measurement.

Data on ambient conditions should be recorded at the start of each calibration run and periodically thereafter as conditions warrant. Ancillary data include observations of ambient temperature, barometric pressure and relative humidity, together with any other information needed to standardize the primary calibration data or to verify that reference conditions were in effect during calibration. It is also useful to record such information as prover identification and related constants, the names of persons performing the calibration, and to note any unusual conditions or events that occur during the calibration run.

If not previously done, arrangements should be made to ensure that the density of the calibration liquid at all anticipated measurement temperatures is available as needed (see 6.5.4).

Arrangements should be made for the electronic acquisition, transfer and storage of calibration data. If a computer is used to acquire the calibration data, it is possible to make on-line checks of data consistency (see 4.5). Moreover, with a computer, nearly all required data standardization steps and computations, especially the pressure-to-height calculation indicated in ISO 18213-4 or ISO 18213-5 and the data standardization calculations given in ISO 18213-2, can be done at the time of acquisition. To fully realize the advantages of electronic data acquisition, the data shall be put into a format that is suitable for subsequent statistical analysis.

Other issues that shall be resolved in developing a calibration plan and the software procedures for implementing it pertain to the number and size of volume increments, the number of replicate observations of each measurement variable, the number of calibration runs that are planned and the determination of heel volume. These issues are considered in turn in the following subclauses.

6.6.3 Number and size of calibration volume increments

The number and size of calibration volume increments depend not only upon the geometry of the tank and established uncertainty limits for volume determinations, but also upon the type and size of provers used, available manpower and time constraints. Each calibration run should contain a sufficient number of calibration increments to detect significant structural features (spargers, coils, etc.) and transition regions in the tank. Uncertainty limits should be considered when the minimum number of calibration increments per run is established. Since calibration increments should be at least five times the detection threshold of the manometer system (see 4.2.3, 4.3 and 6.3.3), the maximum number of increments is limited by the size of the tank and the detection threshold of its measurement system. Subject to this limit, it is generally desirable to plan as many increments as time and manpower constraints allow. It is necessary to balance the desire to have a large number of increments against the length of time required to complete a run. As a rule of thumb, a calibration run should not take more than 10 h or 12 h to complete.

A study of the tank's engineering drawings, or profile plots and incremental slope plots of data from a previous calibration run (see ISO 18213-3), can be used as aids for determining the proper sizes for calibration increments. Generally speaking, the smallest calibration increments should be used in regions where the change in height per unit volume is the greatest, such as in the initial (bottom) regions of the tank.

The first calibration run should be made with sufficiently small increments to detect all significant cross-sectional irregularities in the tank. Volume increments for succeeding calibration runs can be chosen with the aid of information obtained from the first run. If there are regions where the cross-sectional area of the tank is not adequately resolved or where the data are extremely variable, an offsetting increment (e.g. half the volume of the increment used in the first run) may be added at the beginning of a subsequent run. Another option is to make smaller incremental additions in regions of interest. It is unnecessary to offset successive runs if calibration increments are sufficiently small.

6.6.4 Replicate observations

To obtain accurate estimates of replicate (pure) measurement error, provision should be made to obtain repeated observations of calibration variables (e.g. measurement system response, mass, volume) at each increment whenever possible. A sufficient number of observations of each calibration variable should be made to produce a measurement whose variability is not significantly greater than the resolution of the relevant measurement device. Experimentation can be required to determine an adequate number of observations and an optimum observation time. With the electronic systems currently available, it is not difficult to collect 50 or more observations from the tank's measurement system in a matter of seconds. Every effort should be made to eliminate potential sources of error such as operator bias from all observations.

6.6.5 Number of calibration runs

The variability of the tank's profile, as reflected in the functional form of the calibration or measurement equation, should be considered in deciding how many calibration runs are required. A sufficient number of successful calibration runs should be made to obtain both (i) a reliable estimate of tank's measurement equation and (ii) the contribution of uncertainty in this equation to total volume determination uncertainty within the context of the overall accountability plan for the facility (see ISO 18213-3). The variability of liquid height determinations and volume measurements made during the calibration exercise contribute both directly (as measurement error) and indirectly (via the estimated calibration equation) to the uncertainty of future volume determinations. However, run-to-run differences also contribute to model uncertainty and are often the largest component thereof[8]. At least two calibration runs are needed to estimate run-to-run variability, but three to five runs are preferable, especially for the initial calibration of a tank.

6.6.6 Heel volume

In many cases, a tank cannot be completely emptied and the tank contains some liquid from previous activities at the start of a calibration run. Ideally, the volume of this remaining heel should be determined at the start of each calibration run so that measurement system responses can be matched with the actual (or contained) volume of the tank, rather than with only the volume above the heel. In practice, it may be acceptable to determine the heel volume once at the start of a calibration exercise consisting of several runs. A spiking-and-dilution method for determining heel volume is described in of ISO 18213-2:2007, Clause C.2 (see also 7.5 of this part of ISO 18213).

6.7 The calibration plan

All calibration planning activities should culminate in a formal written calibration protocol, or plan. A comprehensive calibration plan should address all essential steps of the calibration process. In particular, the calibration plan should specify what calibration variables are to be measured, what data are to be collected for each calibration increment, the operating conditions under which data are to be collected, what data are required to document ambient conditions at the time of each measurement, and it should specify standard reference conditions.

A very important component of the calibration plan is a schedule that specifies the number of calibration runs, the number of calibration increments for each run and the size of each calibration increment. The calibration protocol should identify regions in the tank of special interest, where small calibration increments are needed to define the effect of some obstruction or to determine the precise location of a transition region. For tanks equipped with siphons, it is especially important to obtain a very accurate calibration in the region surrounding the tip of each siphon probe.

Procedures for estimating heel volumes should be specified, together with potential alignment points that are common to all runs. Finally, the plan should make provision for documenting any background information that pertains to the calibration exercise, such as the results of instrument calibrations and the names of participating personnel.

In summary, the calibration plan should specify all procedural details for all aspects of the calibration exercise. Moreover, the plan should specify what information is required to interpret the data acquired during the calibration exercise, together with the documentation that is required to establish the validity of the calibration.

7 Volume determination

7.1 Overview

The ultimate purpose of a tank calibration exercise is to estimate the tank’s measurement equation. This resulting estimate is then used to determine the volume of process liquid that produces an observed response in the tank’s measurement system (manometer).

Starting with a given measurement of pressure, the volume of process liquid in the tank can be determined from the tank’s estimated measurement equation by means of the following steps.

- Obtain a measurement of the differential pressure produced by the liquid of interest.
- Calculate the height of liquid that corresponds to the observed pressure measurement.
- Calculate the corresponding height at the reference temperature established for the calibration (or measurement) equation.
- Use the tanks estimated measurement equation to determine the volume, at the calibration reference temperature, that corresponds to the height determined at the previous step.
- Compute the volume of liquid at its measurement temperature.
- Compute an estimate of uncertainty for the computed volume.

The steps given here for computing the standardized liquid height used (in the estimated measurement equation) for determining process liquid volume are analogous to those used to compute liquid height for calibration. The main difference is that, it is necessary to measure the density of a process liquid because it is not *a priori* known (see ISO 18213-2 for a complete discussion of the data standardization algorithm, and see ISO 18213-6 for a method for making in-tank determinations of density).

7.2 Steps for determining reference height

7.2.1 Observe differential pressure, ΔP

The first step in obtaining a measure of process liquid volume is to observe the measurement system response for the liquid in question. The differential pressure, ΔP, should be measured under the same operating conditions that were in effect during the calibration exercise (see 6.5). With pressure measurement systems, it is particularly important to obtain the temperature and density of the process liquid (see 6.5.3 and 6.5.4). The ambient conditions at the time of measurement should also be recorded (see 6.6.2).

Operating procedures should specify regions of the tank in which measurements should be avoided because of structural anomalies. Such regions often correspond to segments in which the calibration or measurement equation does not conform well to the tank profile. Measurements of volume should be avoided in regions where the calibration or measurement equation does not fit the tank profile because volume measurement uncertainty can be unacceptably large for accountability purposes. The identification of appropriate regions in which to make measurements for volume determination does not apply to tanks equipped with siphons, because the levels at which measurements can be made are *a priori* fixed.

7.2.2 Determine liquid height, H_M, at measurement temperature, T_m

The second step is to determine the height, H_M, of the column of liquid that yields the observed value of ΔP. This computation involves dividing ΔP (after it is corrected for factors that can result in differences between the pressure at the tip of the dip tube and that observed at the manometer) by the measured density of the observed liquid (corrected for air buoyancy) as given in Equation (5):

$$H_\text{M} = \left(\Delta P - c_\text{M}\right) \Big/ \left[g\left(\rho_\text{M} - \rho_\text{a,s}\right)\right] \tag{5}$$

where

ΔP is the observed differential pressure at the manometer;

c_{M} is the “corrections” for differences between the observed pressure at the manometer and the actual pressure at the tip of the probe;

g is the local acceleration due to gravity;

ρ_{M} is the average density of the liquid at its measurement temperature, T_{m};

$\rho_{\mathrm{a,s}}$ is the average density of the air in the tank above the liquid surface at the prevailing pressure.

The ancillary variables (such as ambient temperature, barometric pressure, etc.) required for determining the correction, c_{M}, depend upon the bubbling rate. Corrections appropriate for very slow and fast bubbling rates are given in ISO 18213-4 and ISO 18213-5, respectively.

In any case, a precise determination of the density of the liquid at its measurement temperature, T_{m}, is required to obtain a height determination that is sufficiently accurate for safeguards purposes. Individual facilities often develop solution-specific equations for process liquids that relate density to temperature. These equations may be used to estimate the density of the process liquid at operating temperatures from density determinations made at some reference temperature.

In tanks equipped with two or more probes, it is possible to make accurate determinations of the density of process liquid from in-tank measurements, provided that the separation of the probes has been determined with a (calibration) liquid whose density has been adequately characterized (i.e. determined with sufficient accuracy at all measurement temperatures). When both probes are submerged in a homogeneous liquid, a measure of the density can be obtained from the (standardized) difference in the two pressure readings, divided by the product of their separation and the local acceleration due to gravity (see ISO 18213-6 for details). Density determinations made from pressure measurements should be verified by laboratory analysis.

7.2.3 Determine reference height, H_{r}, at reference temperature, T_{m}

It is now necessary to compute the reference height, H_{r}, of a point that is at height, H_{M}, at the time of measurement. This calculation is carried out as given in Equation (6):

$$H_{\mathrm{r}} = H_{\mathrm{M}} \big/ \left(1 + \alpha \Delta T_{\mathrm{m}}\right) \tag{6}$$

where

$\Delta T_{\mathrm{m}} = T_{\mathrm{m}} - T_{\mathrm{r}}$ is the difference between the measured temperature of the liquid and the reference temperature for the estimated calibration or measurement equation;

α is the linear coefficient of (thermal) expansion of the tank and its probes (see either ISO 18213-4:—, Clause 5, or ISO 18213-5:—, Clause 5, for details).

7.3 Steps for determining volume

7.3.1 Determine tank volume, V_{r}, at reference temperature, T_{r}

The tank’s estimated volume measurement equation, $V_{\mathrm{r}} = \hat{f}^{-1}\left(H_{\mathrm{r}}\right)$, is now used to determine the volume, at the calibration reference temperature, T_{r}, that corresponds with the reference height, H_{r}, determined at the previous step. This involves substituting the height determination, H_{r}, into the equation, $V_{\mathrm{r}} = \hat{f}^{-1}\left(H_{\mathrm{r}}\right)$ and performing the indicated calculation. The resulting value, V_{r}, is the volume (capacity) of the tank, at the reference temperature, T_{r}, below a point at elevation, H_{r}.[4]

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[4] Note that because liquid density changes with temperature, the volume, V_{r}, obtained from the measurement equation is not the volume at temperature, T_{r}, of the *liquid* that was measured at temperature, T_{m}.

Statistical methods for estimation the tank's calibration or volume measurement equation from a valid set of calibration data are presented in ISO 18213-3.

7.3.2 Determine liquid volume, V_{M}, at measurement temperature, T_{m}

The volume of the tank at temperature, T_{m}, can now be calculated by means of Equation (7):

$$V_{\mathrm{M}} = V_{\mathrm{r}}\left(1+3\alpha\Delta T_{\mathrm{m}}\right) \tag{7}$$

where α and ΔT_{m} are defined in 7.2.3 (see ISO 18213-2:2007, Clause 5). At temperature T_{m}, the volume of the tank below a point at elevation, H_{M}, and the volume of the liquid that determined, H_{M}, are equal, and both are given by Equation (7).

Note that, beginning with H_{M}, it is possible to combine all the indicated calculations into a single equation as given by Equation (8):

$$V_{\mathrm{M}} = \left(1+3\alpha\Delta T_{\mathrm{m}}\right)\hat{f}^{-1}\left[H_{\mathrm{M}}/\left(1+\alpha\Delta T_{\mathrm{m}}\right)\right] \tag{8}$$

Thus, the volume, V_{M}, of process liquid at its measured temperature can be obtained directly from the measurement equation at reference temperature by means of Equation (8).

Equation (8) yields the volume, V_{M}, of the process liquid at its measurement temperature, T_{m}. In practice, it can be necessary to relate the volume of this liquid to its volume at some other temperature (e.g. the ambient temperature in the laboratory), say T_3. This calculation requires the density of the liquid at both temperatures, T_{m} and T_3, and is accomplished by means of the equation for the conservation of mass as given by Equation (9):

$$V_3 = V_{\mathrm{M}}\rho_{\mathrm{M}}/\rho_3 \tag{9}$$

7.4 Compute uncertainty estimates

The final step is to compute a suitable uncertainty estimate for the volume determination, V_{M}. Major components of this estimate are the uncertainties in the measurements of ΔP and $\rho_{\mathrm{M},}$ and the statistical uncertainty resulting from the estimated measurement equation.

Uncertainty in the measurement, ΔP, depends upon the capabilities of the measurement system and is determined at the time of measurement. If the same equipment and procedures are used for calibration as for volume determination, then the "random error" component associated with the calibration or measurement equation can be used as an estimate of uncertainty in ΔP for process liquids. Otherwise, an alternative method of estimation is required.

Uncertainty in the density estimate, ρ_{M}, depends on the method used to make the estimate. If the density is determined analytically, then an estimate of uncertainty appropriate for the method should be available from the laboratory. If the density is determined from in-tank measurements, the associated uncertainty can be calculated by means of the propagation of variability methods presented in ISO 18213-6.

Uncertainty estimates for the estimated calibration (or measurement) equation depend both upon the data and the statistical method used to estimate this equation and should be derived at the time the equation is estimated. Statistical methods that yield suitable estimates of the calibration (or measurement) equation and accompanying uncertainty estimates are presented in ISO 18213-3:—, Clause 6. Suitable methods for combining the various components of uncertainty to obtain an estimate of overall volume measurement uncertainty are also presented in ISO 18213-3. The methods are necessarily somewhat general and can require adaptations to fit the needs of a particular facility.

7.5 Final note on heel volume

If the heel volume of the tank has been incorporated into the volume measurement equation, then the reference volume, V_r, is a measure of the actual (total, contained) volume of the tank below the point, H_r. Otherwise, V_r is only a measure of the calibrated volume of the tank (the volume of liquid added during the calibration) and not its contained volume. In particular, the initial amount of liquid in the tank at the time of calibration is not included in the estimate of V_r.

Determinations of transfer volumes are independent of the heel volume because the heel volume cancels when the difference between pre- and post-transfer volume determinations is computed. However, determinations of contained volume are required for inventory purposes. It is, therefore, necessary to incorporate a determination of the tank's heel volume into the calibration (or measurement) equation that is used to make inventory measurements.

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