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

**Part 1:
Procedural overview**

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*Technologie du combustible nucléaire — Étalonnage et détermination
du volume de cuve pour la comptabilité des matières nucléaires —
Partie 1. Aperçu général de la procédure*
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ISO 18213-1:2007

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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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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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.

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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.

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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 ISO 18213-1:2007

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):

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

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.

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, $V_r = f^{-1}(H_r)$, 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):

$$V_r = f^{-1}(H_r) = \int_{-\varepsilon}^{H_r} A_r(H) dH \tag{2}$$

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where

- H is the elevation of the liquid surface above the reference point;
- $A_r(H)$ 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 $A_r(H)$ 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):

$$V_r = V_0 + \int_{H_0}^{H_r} A_r(H) dH \tag{3}$$

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

$$V_0 = \int_{-\varepsilon}^{H_0} A_r(H) dH \quad (4)$$

If the tank can be completely emptied, then $H_0 = -\varepsilon$ 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

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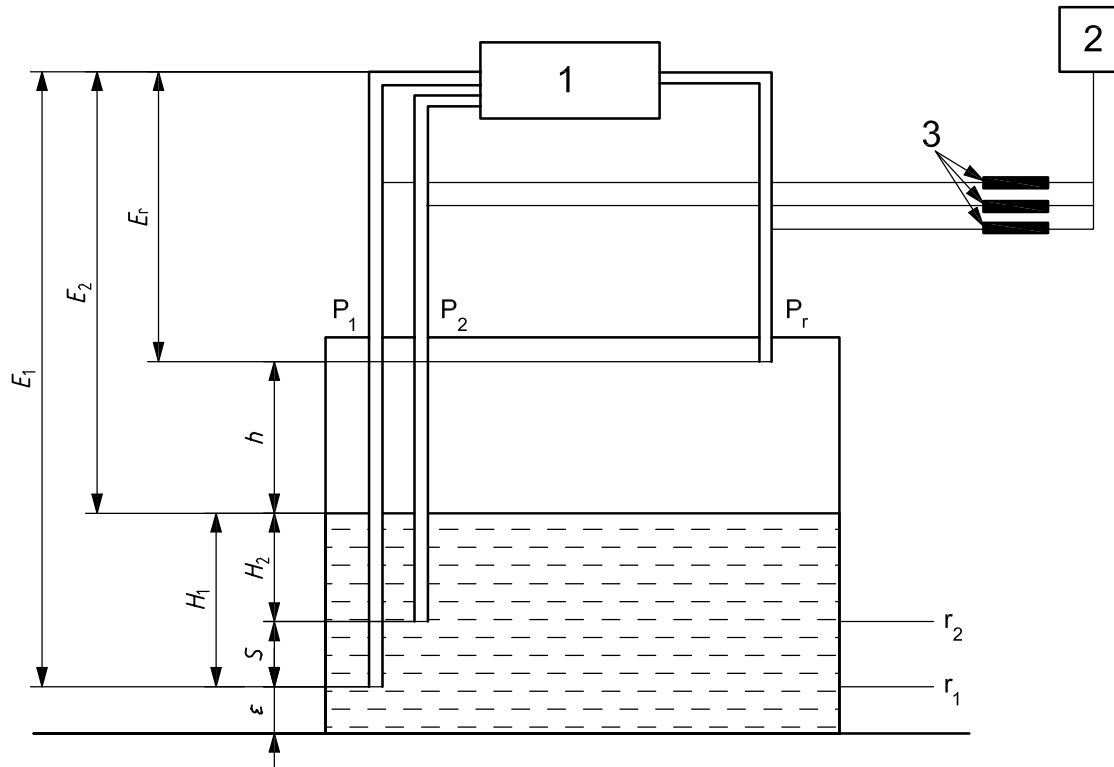
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The elements of a typical pressure-based measurement system for determining liquid content (height) are shown schematically in Figure 1. These include the tank, its bubbler probes, its temperature probes and the manometer(s) used to measure pressure. 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⁾

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.



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Key

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

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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	$\epsilon + S^a$	—

^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.

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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.