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TOWARDS THE RE-VERIFICATION OF PROCESS TANK CALIBRATIONS

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Abstract. Re-verification is needed to ensure that the calibration (the relationship between measured level and measured volume) that is obtained during commissioning hasn't changed over time. This can be achieved, in part, by metering in solution and correlating with marks identified *a priori*. Mark identification and correlation are discussed and possible error sources are outlined.

1. Introduction

Conventionally the volume of solution contained in a vessel is estimated on the basis of pressure, density and temperature measurements. These measurements are combined to calculate a normalised level, which is input into a calibration equation to obtain a volume, which is subsequently temperature corrected. Accuracy therefore relies heavily on the calibration equation and on the measurement base. Issues relating to accuracy are of particular importance in nuclear fuel reprocessing plants, because of the special nuclear materials involved. Although there is probably less concern in other industries, clearly accuracy might be an issue also.

During plant commissioning, the calibration equation is obtained empirically from data collected whilst known quantities of calibration liquid are poured into the vessel. The generation of such calibration data is a costly and time consuming exercise, because it requires that a specific set of experiments should be performed on each vessel under well-determined conditions. There is no guarantee that the calibration equation will remain the same over many years of operation, so procedures must be put in place to ensure that a valid calibration is always referenced. Repeating the same exercise that was carried out during commissioning represents a considerable work load, and would be financially prohibitive if all vessels in a plant were to be re-calibrated at regular intervals. As an example of scale, the International Atomic Energy Agency (IAEA) now monitor the contents in about 60 tanks at the Rokkasho Reprocessing Plant in Japan [Ehinger et. al., 2004]. The term tank is often used instead of vessel, to reflect the fact that they are relatively large storage devices. Indeed they can be very large. Fortunately, from a nuclear materials perspective, certain tanks are more important than others. Tanks are installed in reprocessing plants for at least four purposes: to provide temporary storage for solutions that are fed into, and exit out from, unit processes; to act as 'buffers' to de-couple, to some extent, the operation of the various stages; to provide long-term storage and to provide a means of accounting material that passes through the plant. Only certain tanks need regular re-calibration, re-verification should suffice for the vast majority. That is it should be sufficient to confrim that the original calibration still holds.

A re-verification procedure is proposed here, which is based on the way that volume measurement is performed traditionally with chemistry laboratory equipment like flasks and pipettes. These containers are deemed to contain specific volumes, when they are filled to designated marks. To verify that this is indeed the case, one simply has to confirm that the apparatus does what it is intended to do. If one wants to verify that a 100 ml flask does indeed contain 100ml, for instance, one simply puts 100ml water into the flask and confirms that the water level now aligns with the mark drawn horizontally on the side of the flask. Although simple in concept, when this is extrapolated to full-size commercial tanks, issues arise because the tank is not transparent and hence is difficult to mark, and because of difficulties in metering in an accurate reference volume.

A mark can only be 'seen' if there is appropriate instrumentation available. Vision is required to detect a mark that is drawn on the side of a transparent tank, hydrostatic pressure instrumentation (or level instrumentation, through calculation) can detect changes in cross sectional area. Such changes are in-built, either because of the internal structure of the tank, or because of items that are often inserted into it. For instance horizontal support plates might be inserted to guide instrument tubes and pipework in general. Certain features might be so small that they are not visible in the calibration equations themselves, but nevertheless might still provide a 'crisp' reference to look for. Such features might be identified from engineering drawings, or more likely, from the first application of the experimental procedure described here. Even if a feature is first identified from drawings, experimentation would still be required to obtain their locations, because, as highlighted by De Ridder et al [2004], the height in a tank calibration is not necessarily the same as the geometric height. In other words it is more sensible to determine locations experimentally, preferably during commissioning, and then to re-verify their locations subsequently. Re-verification then reduces to correlation.

For financial reasons, it would be preerable if re-verification could be performed as part of normal operation, for instance by adapting a common procedure. The acid or water imported into a tank during washouts might be metered accurately at a constant flowrate, monitored and level/temperature trends evaluated. Monitoring during an emptying operation is not considered to be practicable, because of the complications that might have to be overcome to meter solution out of a radioactive tank..

It is shown that, with flow metering that is generally available, it should be possible to detect gross changes in the calibration, for instance as a result of solid material build-up in the heal. Greater detail should be attainable with the installation of more accurate flow metering.

2. The Experimental Procedure

One of the major concerns when performing high quality, calibration experiments relates to thermal equilibrium [Higuchi et. al., 2001]. Ideally the calibrating solution should be at one temperature, and this temperature should be the same as the temperature of the receiving tank and of the surroundings. In addition the temperature of the surroundings should not change significantly during the filling operation. This, in part, explains why calibration exercises can be so demanding. Since the experiments envisaged in this paper are unlikely to be performed under such conditions, it is worth bearing in mind that errors arising from thermal non-equilibrium are likely to arise. Whenever possible, plant operators should seek to take every opportunity to minimise thermal equibilibrium. For instance, it is preferable that experiments are set-up during the previous evening by filling the target vessel with 'calibration solution' and leaving the rest of the 'calibration solution' in a holding vessel overnight so that the temperatures in both vessels converge to the ambient temperature.

Two separate sets of experiments can be identified: 1) a number of quick, relatively uncontrolled experiments, merely to ascertain the existence and approximate location of a feature, with which to mark a volume; and 2) a set of experiments to verify, or re-verify, that this mark is reached when the same volume is added. Ideally the first set would be performed as part of the calibration experiments that would be carried out during commissioning. Clearly it would be preferable if at least one feature is near the top of the vessel, as the re-assessment of its location would help to re-verify the calibration of the entire vessel.

In either set, and in-line with the practice that would be adopted in a chemistry laboratory, the following procedure would be adopted: starting with an emptied target vessel, solution would be added quickly until the volume was a little below the mark, say ΔV . A quick fill is preferable to avoid any change in the temperature of the environment. The 'top-up , say $2\Delta V$, would now be added slowly and carefully, this time metering at a constant mass flowrate, W, and recording the level history, L(t). The level histories obtained during the first set of experiments would be analysed to provide a reference, r, with which to compare with the experiments performed later.

3. Data Analysis

The data analysis procedure that would be adopted would depend on certain factors, particularly with the instrumentation. Here we assume that the level history is to be compared with measurements taken previously using the same instrumentation and analysis, because this avoids issues that relate to phase shifts that occur in both the instrumentation and the analysis. Conventionally a liquid level measurement, *L*, is obtained using a dip-tube pneumacator system, which compares the pressure near the bottom of the tank (p_b) with the pressure in the vapour space at the top of the tank (p_v). The level is estimated on the basis of the standard hydrostatic equation that assumes knowledge of the

gravitational constant, g, and liquid density, $\rho: p_b - p_v = L\rho g$. In practice the bottom pressure measurement signal has a small periodic content, attributable to bubble formations. This corruption can be approximated by a Fourier series with fundamental frequency, ω_0 .

The rate at which a tank level rises will change as it passes a crisp feature like a support plate (i.e. the mark). This rate change should be observable at the same location in all the bottom pressure and level time histories collected, provided that the solution is metered into the vessel with the same flow rate. Unfortunately, as is well-known, slope change detection is sensitive to unwanted high frequency variations, and this is particularly problematic here because of the bubble induced corruption. If the tank is filled at a constant flowrate, α , then the derivative (i.e. slope) of its bottom pressure measurement can be approximated by

$$\frac{dp_b}{dt} = \rho g \left[\frac{\alpha}{\frac{\delta V}{\delta L}} + \omega_o \sum_{i=1}^k i\beta_i \cos i\omega_o t \right]$$
[1]

where β_i are the Fourier series coefficients associated with the bubbles. The influence of the bubble term is now frequency dependent, the higher the harmonic the greater its influence. It is therefore important that measurement records are processed carefully, in particular avoiding aliasing as much as possible and attentuating high frequencies where possible. However the degree of attentuation must not be so excessive that it 'masks' a change in slope: the period of bubble formation is typically 2 or 3 seconds, whilst the transient response observed as a result of a sudden change in cross sectional area typically lasts about 10 seconds. Thus any filtering needed to ameliorate bubble and noise effects will affect the fidelity of the technique. Figure 1 gives an example of a filter that has been found to be suitable: this provides differentiation at low frequencies and attentuation at high frequencies, the frequency at which the plot turns is slightly below the bubble fundamental frequency, ω_0 . A typical output from this filter is shown in Figure 2, which pertains to filtered data that was collected from a small tank that was filled at a rate of about 60 litres per hour. The sudden change at about 70 seconds pertains to a sudden change in horizontal cross sectional area: the transient lasts about 10 seconds.

Suppose that a reference filtered data set, y, is generated for a particular feature during commissioning. The location of the feature can then be re-verified by cross correlating, y, with a set of filtered data, x, obtained later. Clearly the collection of both datasets would have to start from the same volume and the tank would have to be filled at the same constant flow rate.

The following form of cross correlation function is recommended for tank sections that have a uniform cross-sectional area:

$$R_{xy}(k) = \sum_{i=f-M}^{f+M} \left[\left(x_i - \overline{x} \right) - \left(y_{i+k} - \overline{y} \right) \right]^2$$
[3]

where $k:-k < N \le k$ and \overline{x} and \overline{y} provide (horizontal) baselines. The shift is then located at the minimum of this function. The choice of focus, *f*, window length *M* and range *N*, and the preparation of the time series, *x* and *y*, would depend on the particular tank and instruments involved. A modified version of Equation 3 is needed to perform the cross-correlation for tanks with a non-uniform cross-sectional area:

$$R_{xy}(k) = \sum_{i=f-M}^{f+M} \left[(x_i - b_i) - (y_{i+k} - c_{i+k}) \right]^2$$
[4]

The choice of baselines b and c is not that straightforward. The most intuitive approach would be to produce theoretical equations for these baselines by considering the curvature in the calibration equation, and then to evaluate these at the heights that are measured. This approach is outlined in Section 5. Unfortunately (bubble) noise corruption of these height measurements makes this route difficult. Instead, a long-time constant, bi-directional EWMA filter is found to perform adequately.



FIG. 1. Typical filter requirements.



FIG. 2. A typical filter output.

3.1. A test case

The approach was applied to the location of a thin, horizontal support plate positioned in the top most section of a 420 litre DTank (Figure 3). Water was metered into the DTank at a rate of 45 kg/hr and its diptube pressures were recorded using standard PPMD instrumentation [5], which outputs highly accurate, filtered data. Figure 4 shows filtered time series pertaining to 3 separate experiments: the bottom plot is the average of these three time series. Focusing on the top trend, it can be seen that the noise levels are quite high, so that the thin, horizontal support plate is barely visible at around 400 seconds. It was for this reason that the experiment was repeated a further two times, so that an averaged series could be obtained with slightly reduced noise levels. Note the feature initially goes the other way, which is similiar to that observed in Figure 2. A correlation accuracy of about 10 seconds was possible, which was equivalent to about 0.15 kg of solution (Figure 5).



FIG. 3. The DTank & its regions.

4. Error Analysis

There are a number of sources of inaccuracy: with estimating the alignment of the two datasets, with metering, and with thermal non-equilibrium.

4.1.1. Estimating the alignment of the two datasets

The alignment of the two datasets can be estimated fairly precisely, provided that the same instrumentation and analysis algorithms have been used in the production of both datasets. For instance a mass error equivalent to about 10 seconds was obtained in the DTank experiment above assuming that the tank was of uniform cross-sectional area in the region examined.

4.1.2. Metering

The metering device installed on the DTank is typical of what is available commercially. It's operation is based on the Coriolis principle, which enables accurate mass flow metering that is independent of density. A conversion is therefore required to obtain the equivalent volumetric flow rate, which might vary with time if the temperature of the solution fluctuates significantly. The metering device installed on the DTank is rated at a maximum flowrate of 300 kg/hr with a repeatability of "0.1% of rate" and an accuracy of "0.5% of rate". The metering device forms part of a local proportional plus integral control system that regulates the flowrate to a setpoint. For a 400 litre tank, the total quantity added into the tank could therefore be in error by as much as 2.4 kg. There are number of ways of reducing this error, the most obvious being to repeat the experiment many times.



FIG. 4. Three DTank processed time series + their average.

4.1.3. Thermal non-equilibrium

An idea of the scale of inaccuracy caused by thermal non-equilibrium can be obtained by seeing what happens if temperature variations are neglected. Normally a tank calibration is corrected for:

shrinkage/swelling:

$$V = \left(\frac{\rho_{ref}}{\rho}\right) V_{ref}$$

and metal expansion:

$$V = V_{ref} \left(1 + \alpha_m \left(\theta - \theta_{ref} \right) \right)^3$$
$$\simeq V_{ref} \left(1 + 3\alpha_m \left(\theta - \theta_{ref} \right) \right)$$

on the basis that the tank was originally calibrated whilst in thermal equilibrium, at a temperature θ_{ref} and with a solution density ρ_{ref} , but is now operated in thermal equilibrium, at a different temperature, θ and density, ρ . Table 1 quantifies the effect of failing to make these corrections, for a 400 litre tank, calibrated at 25°C, but operated at 5°C, 15°C or 20°C.

Table 1. Shrinkage/swelling/expansion errors in litres.

Temperature difference	Shrinkage/swelling/expansion	Expansion only
5°C	0.572	0.103

10° <i>C</i>	1.015	0.206
20° <i>C</i>	1.348	0.413



FIG. 5. The result of shifting the 'new' series by the optimum lag.

5. Feature Identification

To reiterate, although the identification of suitable crisp features might be based on knowledge of the construction of the tank, experimentation is still required to produce their signatures. Clearly this experimentation might be computationally intensive, unless the search can be restricted to particular regions of the tank. Features in ceratin regions are more 'visible' than features in others. This is because a feature will only be visible if the changes in the associated differentiated, filtered time series can be seen in the background of noise. These changes can be estimated from the theoretical, noise free, equation for the differentiated level history, L'(t):

$$L'(t) = \frac{dL}{dt} = \frac{dL}{dV}\frac{dV}{dt} = \alpha \frac{dL}{dV}$$
[5]

if the solution is metered into a vessel at a constant volumetric flowrate, α . If a feature causes a 'sudden' or crisp change, ΔA , in the cross sectional area of a tank that is, locally, of constant area, A, then L'(t) at the feature will be:

$$L'(t) = \frac{\alpha}{\left(A + \Delta A\right)} \approx \frac{\alpha}{A} \left(1 - \frac{\Delta A}{A}\right)$$
^[6]

In other words the absolute change in the differentiated time series will be proportional to the relative change in area. It has already been demonstrated that the top support plate in the D-tank was just about discernible in data collected with standard tank calibration equipment and when filled at 45 kg/hr. It is therefore unlikely that a small feature would be discernible in regions of the tank where the cross sectional area is much larger, unless the instrumentation is improved. In addition, the fill rate may also be a practical issue. For instance the DTank would take in excess of 8 hours to fill, if filled at a rate of 45 kg/hr. Figure 6 shows averaged, differentiated, filtered time series obtained when the DTank was filled, twice, at a rate of 100 kg/hr, where the regions are as those defined in Figure 3. Height information has been added to the right-hand graphs to give an idea of the location in the tank. The thin, horizontal plate is located at a height of about 1830mm. Note that the slope estimate for the top most region (E) is about a factor of 10 times larger than that for Regions B & D, and about a factor of 2 for Region C. Note also that the slopes change significantly over Regions B & D and that ('absolute' sized) noise is seen to dominate these plots more than in the other two. Thus any feature identification strategy must be sensitive to these limitations by restricting the search to those regions that are thought to give a realistic chance of finding something suitable.



FIG. 6. Regional slopes

6. Conclusions

Although it should be possible to verify the location of crisp features accurately, inaccuracy will still centre on issues relating to metering and thermal non-equilibrium. Ideally the testing should be performed under the same environmental conditions, with the same instrumentation and following the same procedures. This should still be far less stringent than with conventional tank calibration, because most of the solution is added as one batch, and subsequent to this, a wait period can be implemented so that the tank is encouraged to reach a state of thermal equilibrium.

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