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Automated Substitution Weighing Apparatus for Liquid Volume Measurement

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The custody transfer of petroleum worth \$1 x 10^{12} / year is traced to the volume delivered from provers and liquid test measures. Mistakes as small as 0.02 % in a custody meter calibration can lead to multi-million dollar corrections in bills. NIST presently uses the direct weighing method to measure the volume delivered from a test measure, by weighing (on a calibrated balance) the test measure when it is full of pure water and again after it has been drained. Recently, NIST reduced the uncertainty of its calibrations of volumes between 3.8 L and 40 L by constructing an automated substitution-weighing standard. The new standard reduces the mass measurement uncertainty by alternately placing 1) the test measure (unknown mass) and 2) approximately equal reference masses on the balance. Automated, pneumatically-driven hardware moves heavy liquid-filled volumes and reference masses on and off the weigh scale, thereby protecting the operator's safety and comfort. We describe the system's design, operation, uncertainty, and repeatability. The new standard was validated by comparison to NIST's well established direct weighing approach and by repeated calibration of a 38 L pipette during more than 2 years. The results are fully consistent with the 95 % confidence level uncertainty estimate of 0.007 % for the substitution weighing standard. Correlation between temperature and the volume of the 38 L pipette shows the need for improved environmental temperature control in the laboratory. We also describe the design of a set of four pipettes with an overflow filling system that can be used as references in an automated volume transfer standard.

1. Introduction

The NIST Fluid Metrology Group measures the volume of water delivered from test measures to provide impartial, System International traceability for petroleum custody transfer. NIST presently uses the direct weighing method for these calibrations, but has developed an automated substitution weighing standard for test measures from 3.8 L to 40 L that has uncertainty at least 6 times smaller. This paper describes the new standard and validates its performance by directly comparing it to the existing direct-weighing standard.

For the direct weighing method, a balance (weigh scale) is calibrated with reference masses and the balance is subsequently used to weigh the test measure: 1) when it is full of pure water and 2) after it is drained [1]. At NIST, a 60 kg balance is used to calibrate volumes 40 L or smaller and a 600 kg balance is used for volumes up to 380 L. Between periodic calibrations of the balance, it is evaluated with a check mass at mid-scale, and an error larger than 3 g triggers a new balance calibration. The 95 % confidence level uncertainty of the full and drained mass measurements is based on the 3 g tolerance and a rectangular probability distribution [$u(m) = 2(3/\sqrt{3}) = 3.46$ g]. Usually, this 3.46 g uncertainty is the largest uncertainty component for test measures smaller than 40 L. The 95 % confidence level uncertainty of the delivered volume of a 3.8 L test measure is 0.32 % and for a 40 L test measure it is 0.04 %.

Single substitution weighing alternately places an unknown mass and a reference mass on a balance and uses the buoyancy-corrected reference mass and the ratio of the two balance readings to determine the unknown mass [2]. Substitution weighing is equivalent to calibrating the balance immediately before it is used to weigh the unknown mass and provides lower mass uncertainty. Our automated substitution weighing standard has uncertainty of 0.007 % or less (95 % confidence level).

We also describe a set of pipettes designed to serve as references for calibrations of test measures by volume transfer [1]. The pipettes have nominal volumes of 3.8 L, 19 L, 38 L, and 190 L. Our plan is to use the pipettes in various combinations to calibrate customer's test measures that are larger than 40 L. The pipette design is bottom-filled, has an overflow system for filling that produces a reproducible delivered volume, and is designed for automation. We present calibration results for the 38 L pipette from the substitution weighing standard that demonstrate the suitability of the design for low uncertainty volume transfer calibrations.

2. The Automated Substitution Weighing Standard

Figures 1 and 2 show the arrangement of equipment in the substitution weighing standard. Three aluminum plates can be placed on or off a 60 kg Mettler* balance by pneumatic actuators under the control of a data acquisition computer and a Labview program. The "bucket plate" is used to place a plastic bucket large enough to receive the delivered volume from a volume under test (VUT) on the balance. Plate #2 holds reference masses that produce approximately the same balance reading as the bucket plate when the bucket is drained. Plate #3 holds masses that produce a balance reading approximately equal to the bucket plate when the bucket is filled with water from the VUT. The pneumatic actuators (Motion Controls Inc, D12SERC SL1.5 RA1) are air driven in both directions (not spring return) and needle valves on the air inlets allow one to tune the speed that they move, thereby avoiding sudden impacts on the balance when the plate is lowered. Two wires run from a support post to the bucket plate to: 1) open or close the electrically-actuated drain valve (Plast-o-matic ¾ inch) on the collection bucket, and 2) measure the temperature of the water in the bucket. We hang the wires in a catenary shape to avoid significant changes in the forces imposed by these wires when the bucket plate is on the balance in the lowered position. Other temperature sensors measure the temperature in the volume under test. The reference masses used on Plates #2 and #3 are rectangular blocks of 316 stainless steel that were calibrated by substitution weighing and are traceable to the System International through US mass standards. A Vaisala PTU200 measures the room pressure, temperature, and relative humidity. These measurands are used to calculate the air density for buoyancy correction to mass.

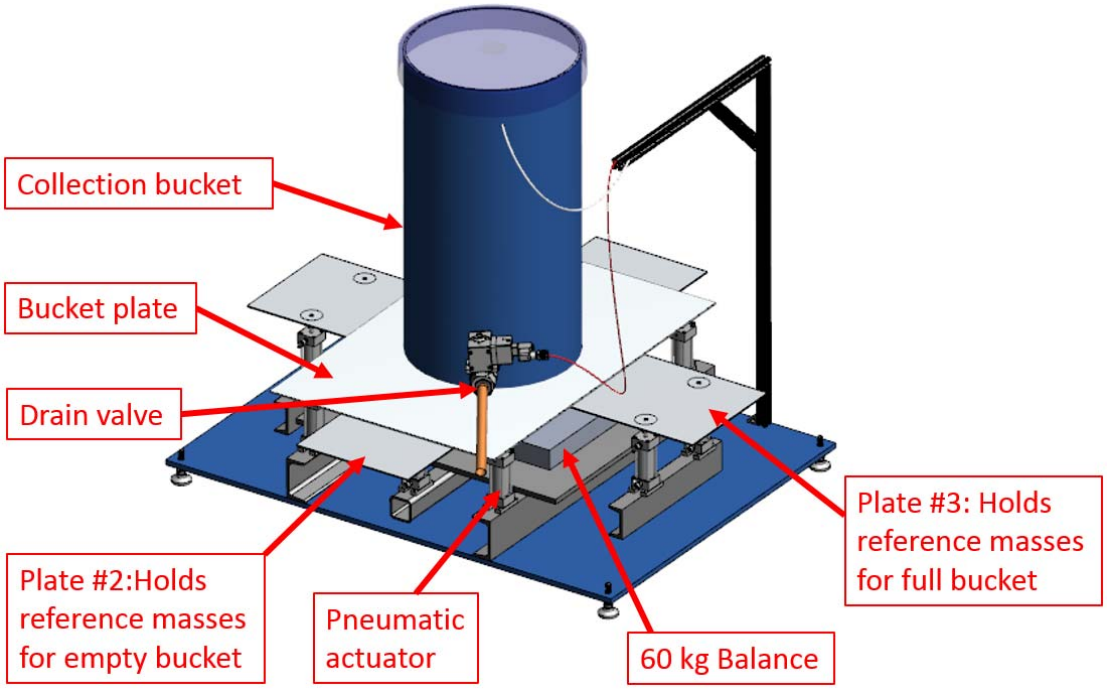

Figure 1. A drawing of the automated substitution weighing standard.

* Certain commercial entities, equipment, or materials may be identified in this document in order to describe an experimental procedure or concept adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the entities, materials, or equipment are necessarily the best available for the purpose.

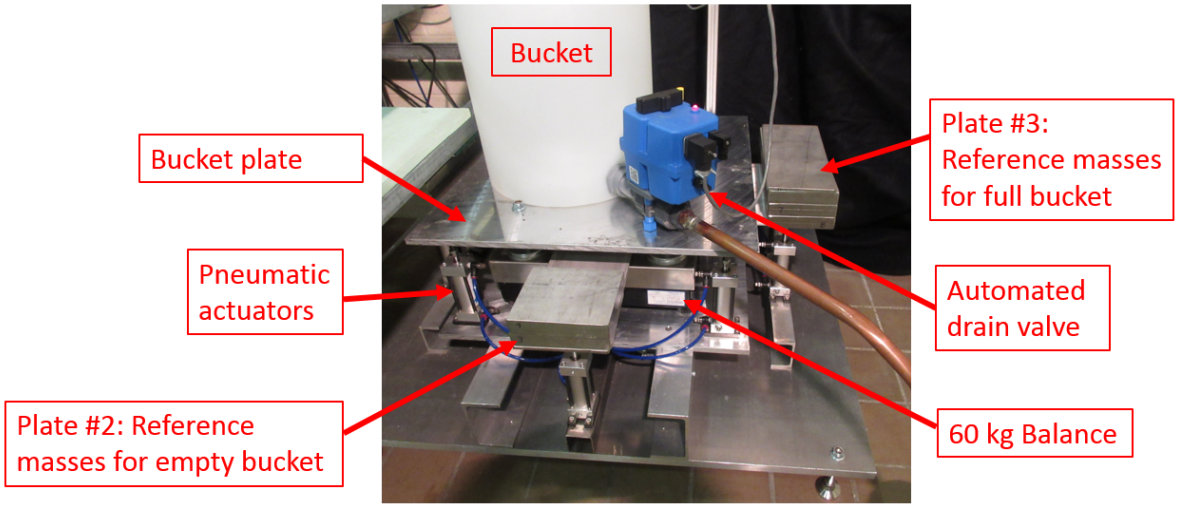

Figure 2. A picture of the automated substitution weighing standard.

A single volume calibration run using the substitution weighing standard has the following steps:

1. Place the VUT on a platform above the substitution weighing standard so that it can be drained by gravity into the collection bucket. Note that the VUT should be levelled, filled, and drained in the same manner as in normal use by the customer. The same drip time and if practical, the same plumbing on the drain should be used so that residual volume of liquid on the interior walls of the VUT is consistent during calibration and usage [3].
2. Check that the collection bucket is drained and the drain valve is closed. Lift all plates off the balance, wait 17 s or more for the balance reading to stabilize, re-zero the balance, and record the balance zero reading (R_{z1}).
3. Lower the bucket plate (and the bucket) onto the balance. When stable, record the balance reading (R_{buc1}).
4. Raise the bucket plate and when stable, record the balance zero reading (R_{z2}).
5. Place reference masses of approximately equal weight as the bucket plate and empty bucket on Plate #2.
6. Lower Plate #2 and when stable, record the balance reading (R_{ref1}).
7. Fill the VUT with pure water. NIST uses filtered, reverse-osmosis water so that literature correlations of water density as a function of temperature apply [4].
8. Wait for the VUT temperature sensor to reach thermal equilibrium with the water, and record the VUT water temperature. Record the volume registered by the VUT.
9. Transfer water from the VUT to the collection bucket. Take care that no water splashes out of the collection bucket during transfer from the VUT. The bucket has a lid with minimal openings to the room to reduce water loss by evaporation.
10. Raise Plate #2 and when stable, record the balance zero reading (R_{z3}).
11. Lower the bucket plate onto the balance and when stable, record the balance reading (R_{buc2}). Record the bucket water temperature and the environmental conditions: barometric pressure, room temperature, and relative humidity (for buoyancy corrections). Because the mass measurements are only weakly sensitive to the environmental conditions and change slowly, these values can be used for all mass buoyancy corrections for this run.
12. Raise the bucket plate and when stable, record the balance zero reading (R_{z4}).
13. Place reference masses of approximately equal weight as the bucket plate and full bucket on Plate #3.
14. Lower Plate #3 and when stable, record the balance reading (R_{ref2}).
15. Open the bucket valve to drain the bucket to prepare for the next run.

In a typical calibration, the appropriate steps above are repeated to produce 5 or more independent volume determinations to assess repeatability of the process.

The apparent mass of the drained bucket ($m_{A,buc1}$) is calculated from the ratio of the zero-corrected readings for the bucket and reference masses multiplied by the apparent mass of the reference masses (Equation 1).

Note that the reference mass is both the stainless steel blocks and the aluminum plate supporting them, leading to:

$$m_{\mathrm{A,buc1}} = \frac{(R_{\mathrm{buc1}} - R_{\mathrm{z1}})}{(R_{\mathrm{ref1}} - R_{\mathrm{z2}})}\left[m_{\mathrm{ref,ss1}}\left(1 - \frac{\rho_{\mathrm{air}}}{\rho_{\mathrm{ss}}}\right) + m_{\mathrm{ref,Al1}}\left(1 - \frac{\rho_{\mathrm{air}}}{\rho_{\mathrm{Al}}}\right)\right], \quad (1)$$

where ρ_{air} is the density of the room air, $m_{\mathrm{ref,ss1}}$ and ρ_{ss} are the mass and density of the stainless steel reference masses, and $m_{\mathrm{ref,Al1}}$ and ρ_{Al} are the mass and density of the aluminum plate. Analogously, the apparent mass of the full bucket is:

$$m_{\mathrm{A,buc2}} = \frac{(R_{\mathrm{buc2}} - R_{\mathrm{z3}})}{(R_{\mathrm{ref2}} - R_{\mathrm{z4}})}\left[m_{\mathrm{ref,ss2}}\left(1 - \frac{\rho_{\mathrm{air}}}{\rho_{\mathrm{ss}}}\right) + m_{\mathrm{ref,Al2}}\left(1 - \frac{\rho_{\mathrm{air}}}{\rho_{\mathrm{Al}}}\right)\right]. \quad (2)$$

The density of air is calculated from the environmental pressure, temperature, and relative humidity measurements made during the calibration run. Once the apparent masses of the full and empty bucket are calculated, the volume of delivered water is calculated in the same manner as for the direct weighing method using Equations 4 and 5 in reference [1]. The 95 % confidence level uncertainty of the mass measurements for the direct weighing method is 3.46 g while the 95 % confidence level uncertainty via substitution weighing is less than 0.6 g, dominated by the uncertainty of the reference masses and balance non-linearity (10 parts in 10^6). The improvement in mass measurement reduces the expanded uncertainty (k = 2) of the delivered volume measurement to 0.007 % or less.

We have compared volume measurements made by direct weighing and by the automated substitution weighing standard using three 20 L test measures, and the results are shown in Figure 3. Volume “A” is a slicker plate, Volume “B” is a pipette, and volume “C” is a neck scale test measure. The points in Figure 3 are based on averages of 5 or more volume measurements. The slicker plate and pipette show a six-fold improvement in uncertainty for the substitution weighing method. The neck scale test measure uncertainty is reduced by a factor of two: the poorer repeatability of the neck scale test measure relative to the slicker plate and pipette hamper uncertainty improvement somewhat. The volume measurements made by direct weighing and substitution weighing agree within 170 parts in 10^6, 22 parts in 10^6, and 6 parts in 10^6 for volumes A, B, and C respectively.

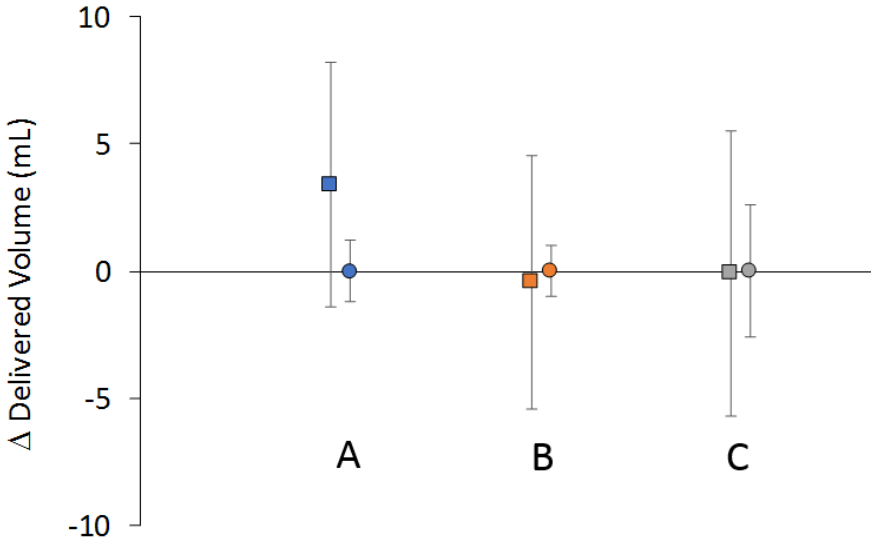

Figure 3. The difference in delivered volume from the direct weighing method (squares) and the automated substitution weighing standard (circles) for three 20 L test measures. Volume A is a slicker plate, volume B is a pipette, and volume C is a neck scale test measure.

3. Volume Transfer Pipettes

The NIST Fluid Metrology Group purchased four Seraphin Series P pipettes with nominal volumes of 3.8 L, 18.9 L, 38 L, and 189 L. With a suitable platform, we plan to calibrate these pipettes gravimetrically and then use them (in various combinations) to do volume transfer calibrations [1] of customer test measures. Figure 4(a) is a general view of the 38 L pipette where one can see the main body of the volume, the overflow system for filling, and one of the two thermowells for temperature sensors used to measure the water and pipette material

temperature. This temperature is used to calculate water density and to make thermal expansion corrections and give the VUT volume at the customer-specified reference temperature. Figure 4(b) shows the lower half of the pipette and the pneumatically actuated valves used to fill or drain the pipette via computer control. The valve is a weir-type valve made by ITT Industries that is designed to prevent any trapped air or liquid volumes in either the open or closed states. Figure 4(c) shows the overflow system that sets up a reproducible meniscus (and hence liquid volume) with the pipette. The pipette is bottom filled to reduce bubble formation. When the pipette is full, water flows out of the pipe labelled “meniscus” in Figure 4(c) and the float switch triggers the Labview program to close the fill valve. Water gravity-flows out of the overflow system through the pipe labelled “overflow” and later through a 3 mm “drain hole”. The small diameter drain hole slows the draining of the water from the overflow system and sets up a reproducible liquid level at the position labelled “meniscus”.

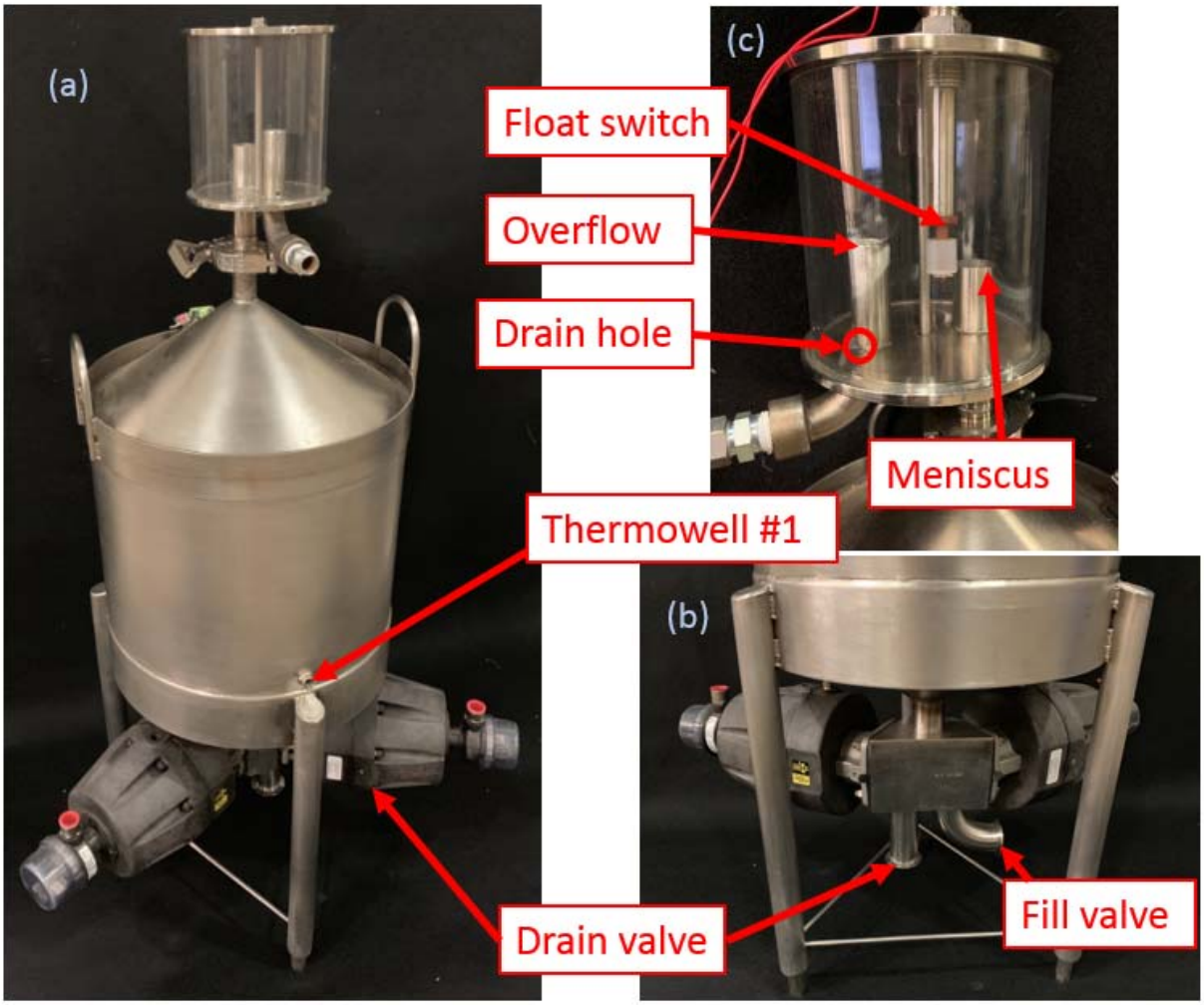

Figure 4. An overall view of the 38 L pipette (left), the actuated fill and drain valves (bottom right), and the overflow system (top right).

The 38 L pipette has been calibrated using the substitution weighing standard on 12 occasions spanning more than 2 years. The results are shown in Figure 5. The uncertainty bars in Figure 5 are the 95 % confidence level uncertainty of the volume calibrations (0.007 %). The standard deviation of the 5 measurements averaged to make each point in Figure 5 is 17 parts in 10^6 or less.

While the 12 volume values in Figure 5 agree within the uncertainty expectations, we searched the data for explanations for the 0.015 % changes over time. The room where the substitution weighing standard is presently located has poor environmental controls and the temperature in the room ranged from 17.9 °C to 26 °C depending on the season of the year. The reverse osmosis water is continuously pumped through filters and an ultraviolet light system to prevent bacterial growth in the water. The continuous pumping keeps the water about

5 °C warmer than the room temperature. We found strong correlation between the volume changes in Figure 5 and the 1) room temperature, 2) water temperature, and 3) difference between the room and water temperatures during calibrations. The temperature difference is plotted on the secondary axis of Figure 5 to illustrate the correlation.

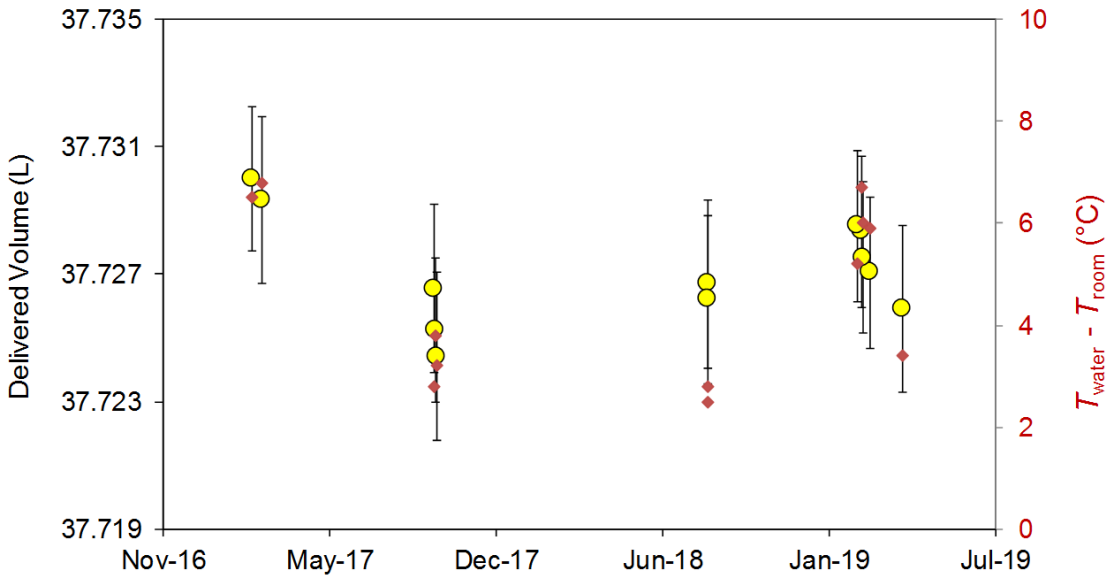

Figure 5. Calibrations of the NIST 38 L pipette performed with the automated substitution weighing standard. Volume differences correlated with temperature show the need for better environmental controls.

After the initial set up of the pipette and the placing of masses on Plates #2 and #3 of the substitution weighing standard the calibrations of the 38 L pipette were performed by the data acquisition system and Labview program: No operator actions were required to collect the calibration data. This level of automation was achieved using 1) the actuated fill and drain valves, 2) the float switch to stop filling, 3) the overflow filling system (no reading of a neck scale required), 4) temperature sensors in thermowells (no need to install and remove them by hand to avoid volume errors), and 5) the previously described automation of the substitution weighing standard. At present, when setting up a run, the operator measures and enters the drain time into the Labview program so that the computer can implement the necessary delays. In the future, we will install a sensor to detect the cessation of main flow from the pipette in order to initiate the 30 s drip interval.

4. Summary and Conclusions

We described an automated substitution weighing system designed to calibrate the volume delivered from test measures between 3.8 L and 40 L. We validated the performance of the new standard by comparison to NIST's existing direct weighing volume standard. The uncertainty estimate for the new standard is 0.007 % at a 95 % confidence level. This uncertainty is 6 or more times smaller than the direct weighing approach but there are numerous opportunities for refinement of the new standard that could reduce the uncertainty further. For example, for the data we have presented herein, the reference masses and the unknown masses differed by as much as 7.2 %. One uncertainty component of the substitution weighing result is directly related to the magnitude of this difference and the linearity of the balance. Using a reference mass set with smaller mass increments will reduce this uncertainty component. As a second example, Figure 5 shows correlation between the 38 L pipette volume results and the room and water temperature. We conclude that better laboratory environmental controls (particularly temperature) would improve the calibration uncertainty.

We described the design of a pipette with an automated and repeatable (< 17 parts in 10^6) filling system. A set of these pipettes of various sizes can be placed on an elevated platform to perform highly automated and low uncertainty volume transfer calibrations of a customer's test measure.

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[3] Wright, J. D., Sheckels, S. D., Lachance, C., Sharifi, F., Falicki, A., and Ondoro, D., *The Effect of Liquid Kinematic Viscosity and Drain Time on the Volume Delivered from a Test Measure*, Proceedings of the 9th International Symposium on Fluid Flow Measurement, Arlington, Va, USA, April 14 to 17, 2015.

[4] Tanaka, M., Girard, G., Davis, R., Peuto, A., and Bignell, N., *Recommended Table for the Density of Water between 0 ℃ and 40 ℃ Based on Recent Experimental Reports*, Metrologia, **38**, 2001.