The Effect of Liquid Kinematic Viscosity and Drain Time on the Volume Delivered from a Test Measure[*]

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Abstract

Volumetric test measures are used for calibration and traceability of petroleum measurements and their low uncertainty is important for the equitable trade of valuable liquids. The volume delivered from a test measure depends in part on the volume of liquid retained on the test measure walls after the draining procedure is complete. Mixtures of propylene glycol and water with kinematic viscosity ranging from 1 mm²/s to 55 mm²/s were used to measure the delivered volumes of three test measures (19 L, 20 L, and 38 L) for 30 s drip times. The retained volume in a 60 L test measure was determined using water at 7 °C to 35 °C to change the liquid kinematic viscosity. Our experiments demonstrate that van Rossum's [1] drainage theory applies to test measures: the retained volume is proportional to the square root of the ratio of kinematic viscosity to total drain time ($V_{\text{retain}} \propto (v/t_{\text{d}})^{1/2}$). We use van Rossum's theory and retained volume data from more than 300 test measures to estimate changes in the delivered volume due to drainage effects for various liquids and test measures up to 400 L. For example, neglecting other effects (e.g. evaporation, and surface tension), we estimate that a 20 L test measure calibrated in water will deliver 0.01 % more volume when used in gasoline and 0.05 % less volume when used in diesel fuel.

1. Introduction

Test measures are well-established tools for maintaining traceability of petroleum measurements to national standards and ensuring fair trade of valuable liquids. For example, they are often used to calibrate the volume of liquid between the start and stop sensors in a piston or ball prover [2]. In this way, they are tied to the equitable trade of 10^{12} dollars of petroleum products between buyers and sellers each year. Test measures (usually 5 gallons) are used in the U. S. by weights and measures inspectors to verify gasoline dispensers are delivering the correct quantity of fuel to consumers; within maintenance tolerance of 0.5 % [3].

Test measures can be calibrated for their contained volume or for their delivered volume. The contained volume is the quantity of liquid inside a test measure when it is filled to the zero mark on its neck scale. The delivered volume is the quantity of liquid that is discharged from the test measure when it is drained in a specified manner, i.e. by opening a valve (or pouring through the neck) until the "cessation of main flow" and then allowing the liquid to drip for a specified time before closing the valve (or tipping the hand-held test measure back to an upright position). Delivered volume is of greater interest for

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[*] May 9, 2019 Errata: Equation 5 had an error in the original version of this paper and it is corrected in this version.

users of test measures because the same test measure is often used repeatedly and it is impractical to dry the test measure between each usage.

Calibration laboratories use water to calibrate test measures by volume transfer or gravimetric methods [4] and achieve uncertainties as low as 0.01 % for test measures with neck scales. Water is also the calibration liquid when test measures are used to determine the volume of small volume or ball provers. But the test measures that are used to check fuel dispensers are calibrated with water and subsequently used with a different liquid, i.e. gasoline, diesel, or heavier petroleum products. A recurring question is: how much uncertainty is introduced when one calibrates the delivered volume of a test measure with water and then uses it in a fluid with different properties?
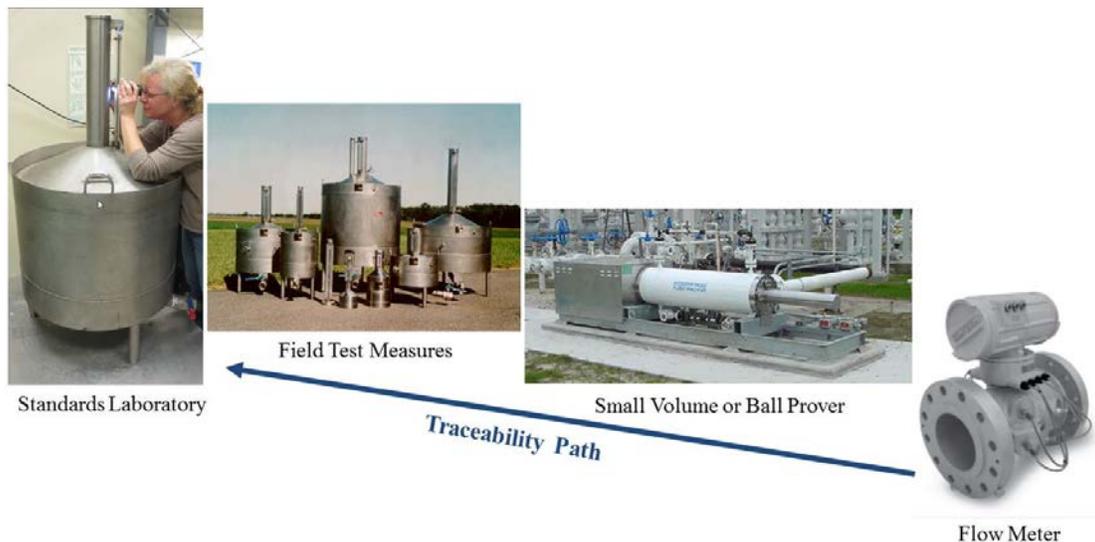
Figure 1. Volume test measures provide traceability for petroleum sales in pipelines and for retail gasoline and diesel dispensers.

It is understood that both the drainage procedure as well as the viscosity of the liquid will have an influence on the amount of retained volume for volumetric standards that are calibrated on a "To Deliver" basis. Variations in the retained volume cause variations in the delivered liquid volume and therefore the accuracy of the volumetric standard. It is evident that smaller test measures are prone to more relative variability due to drainage effects than large test measures because of their higher ratio of surface area to volume.

Drainage effects are an often overlooked concern in the uncertainty of laboratory volume calibrations. Calibrations are corrected for thermal expansion of the test measure material to give the volume at a reference temperature via the equation:

$$V(T_{\mathrm{ref}}) = V(T)[1 - \beta(T - T_{\mathrm{ref}})] \tag{1}$$

where the normally used reference temperature $T_{\mathrm{ref}} = 15.56\ °C$ in the U. S., β is the volumetric thermal expansion coefficient of the test measure material, and T is the temperature of the material during the calibration. But there is an additional thermal correction that is not generally applied. For example, if a test measure is calibrated a 23 °C, the water used to calibrate it will be less viscous that it would be at

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the reference temperature of 15.56 °C and less water will remain on the walls of the drained test measure after the 30 s drip time.

In this publication we will review the theory of drainage effects, verify the theory experimentally, and assess the significance of drainage effects for test measures in custody transfer applications.

2. Prior Work

In 1958, van Rossum [1] presented the theory of viscous drainage and showed excellent agreement with experimental data from Morey [5]. Van Rossum calculated the mean liquid film thickness on a *plane vertical plate* lifted at a *constant speed* from a pool of liquid assuming laminar flow, negligible inertia, and *negligible surface tension effects*. He found that the profile of the film as a function of time was a family of parabolas and that the mean film thickness is,

$$\bar{\xi} = \frac{2}{3}\left(\frac{\nu}{g}\frac{L}{t}\right)^{\frac{1}{2}},$$
(2)

where ν is the kinematic viscosity[1], g is the acceleration due to gravity, L is the length of the film, and t is the time since the vertical motion of the plate started. In the context of a test measure, t is the time since the drain valve was opened until the stage of drainage considered, not the 30 s drip time.

van Rossum's problem is similar to the draining test measure but there are important differences: 1) the speed of the falling liquid surface slows as the test measure drains and 2) the test measure wetted surfaces are not all vertical, and 3) drops may remain on surfaces due to surface tension effects. Based on Equation 2, for a particular total drain time t_{d}, we might expect a linear relationship between the amount of liquid remaining in a test measure and $(\nu/t_{\mathrm{d}})^{1/2}$. More recent studies of draining films have examined the tear drop and wavey structures that occur over certain ranges of Reynolds and Kapitza numbers [6], but we anticipate that the theory of van Rossum will serve our purposes and allow prediction of the retained volume remaining on the walls of a test measure for a given change in liquid properties or drain time.

3. Drain Times

The total drain time t_{d} is the interval between the beginning of draining and the termination of dripping. We divide the interval into two parts: (1) the duration of the main flow, t_{cess}, and (2) the time allotted for dripping, t_{drip}:

$$t_{\mathrm{d}} = t_{\mathrm{cess}} + t_{\mathrm{drip}} .$$
(3)

The time until the cessation of main flow, t_{cess}, is the interval from opening of the prover outlet valve at the beginning of delivery to the time when the liquid level has dropped to the test measure drain pipe, the flow "breaks", and the flow in the drain pipe is no longer completely water but carries air too. This is usually detected by listening for the sound of the flow breaking but some test measures have clear sections in the drain pipe that allow a visual assessment. The measurement of the time to cessation of

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[1] Kinematic viscosity $\nu = \mu/\rho$ where μ is the dynamic viscosity and ρ is the mass density.

main flow is subjective, especially for highly viscous liquids and this is one source of irreproducibility for test measures.

The drip time, t_{drip}, is a specified interval from when the liquid flow breaks to the closing of the test measure outlet valve. American Petroleum Institute Chapter 4.7 [7] specifies a 30 s drip time after the flow breaks, but other procedures are also used. For instance, in Canada the drip time varies with the size of the test measure and can be up to 5 min long. For a longer drip time, drainage is more complete, correctly detecting the time of flow breakage is less critical, and the delivered volume is more reproducible. For the data shown herein, the drip time at NIST was 30 s after cessation of main flow and the drip time at Measurement Canada (MC) was 65 s after the cessation of main flow.

The design of the test measure and the configuration of the drain can have significant influence on drain time t_{cess}. Aspects of the test measure that influence drain times and retained volume are: height to diameter ratio, internal baffles, top and bottom cone angles, drain pipe angle, internal wall material, cleanliness, and surface roughness. Tests at MC show that when a hose is attached to the test measure outlet, it can reduce t_{cess} due to increased liquid head. For a 60 L test measure without a hose connected to the drain valve, t_{cess} was 23.5 s ± 1 s (95 % confidence). But when a fully-flowing 1.8 m hose with outlet 1.2 m below the test measure outlet was connected to the drain, t_{cess} was cut nearly in half to 13.5 s. Drain rate will be reduced when piping with a restriction is attached to the test measure outlet. For example, the 19 L and 20 L test measures shown in Figure 3 have t_{cess} of 128 s and 80 s respectively when used with water: the 19 L test measure takes longer to drain because the long radius elbow on the exit reduces the flow through the outlet. Hence, it is important that the piping configuration during calibration match that during usage and not accelerate or decelerate the drain rate. Generally this means that the drain pipe and valve on the test measure must be maintained as the most significant restriction and to avoid increased head, only an oversized hose with an air vent can be used to channel flow away from the drain valve (Figure 2).
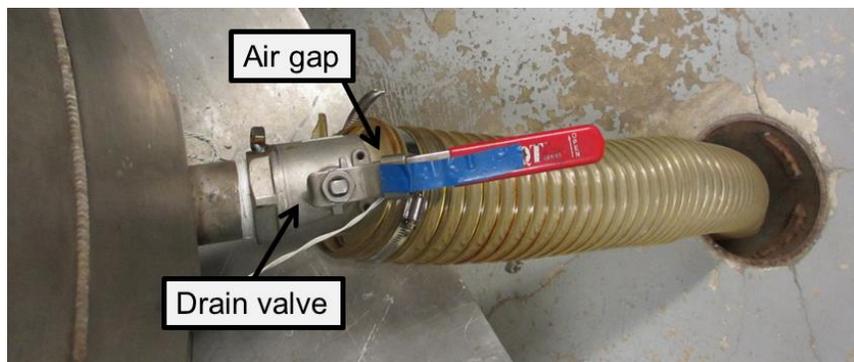
Figure 2. A drain piping configuration to avoid head effects on drain times during calibration.

4. Experimental Measurements

In this work, NIST and MC measured the retained volume for different drain times and liquid kinematic viscosities in order to verify that van Rossum's model applies to test measures. To calculate the contained volume, V_{contain}, the change in mass between the dry and water-filled test measure is

measured during the first run of the calibration. Subsequent runs give the delivered volume, V_{deliver}, using the mass difference between the full and drained (but still wet) test measure. The details of these measurements are given in the description of the NIST liquid volume calibration service [4]. The volume of liquid retained in the test measure is,

$$V_{\text{retain}}(T) = V_{\text{contain}}(T) - V_{\text{deliver}}(T) \quad . \tag{4}$$

Reference [4] includes a section on viscosity corrections that is based on van Rossum's theoretical model. The NIST liquid volume calibration service includes an uncertainty component for the retained volume due to the temperature of the water during calibration T differing from the reference temperature, T_{ref}. NIST assumes a rectangular distribution and van Rossum's drainage theory to arrive at a 95 % confidence level uncertainty due to drainage effects:

$$U_v = \frac{2}{\sqrt{3}}[V_{\text{contain}}(T) - V_{\text{deliver}}(T)]\left(1 - \left[\frac{v(T_{\text{ref}})}{v(T)}\right]^{1/2}\right). \tag{5}$$

The uncertainty component, U_v only accounts for the change in water kinematic viscosity at the time of calibration from water at T_{ref}. It does not account for the temperature conditions during customer usage or the effects of using the test measure with a liquid other than water.

Figure 3. Four test measures used to measure retained volume dependence on kinematic viscosity.

Figure 3 shows four test measures used to determine retained volume in this study. In November 2013, Measurement Canada used a gravimetric method [8] to conduct drainage experiments on test measures

using water at various temperatures. As shown in Figure 4, for the MC 60 L test measure, the retained water varied between 37 cm^3 and 16 cm^3 for temperatures ranging from 7 °C to 35 °C (0.75 cm^3 / °C). The thermal expansion corrections of Equation 1 are applied to this data, so the changes in retained volume are due to drainage effects and the dependence of the water kinematic viscosity on temperature.
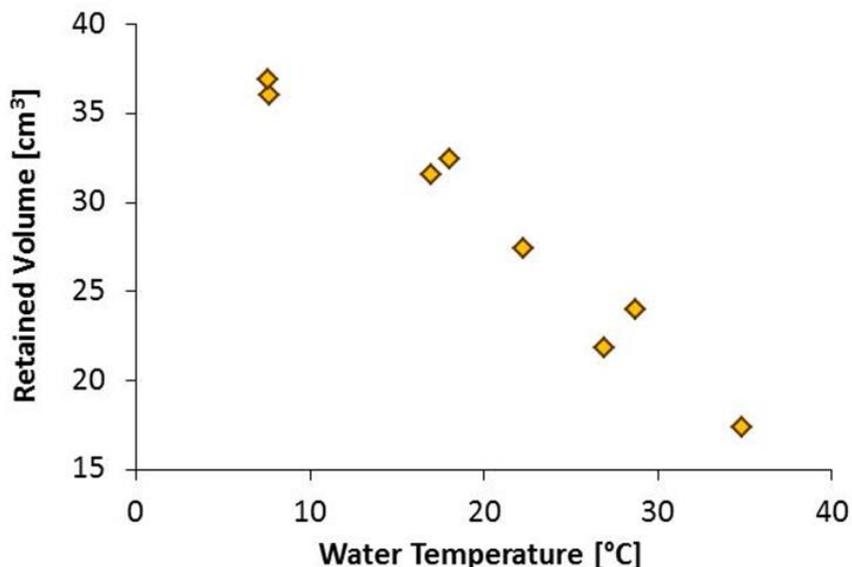
Figure 4: Retained volume of water versus water temperature for a 60 L test measure: $t_{cess} = 49$ s, $t_d = 65$ s.

The NIST Fluid Metrology Group measured the retained volume for three test measures with volumes of 19 L, 20 L, and 38 L using mixtures of USP[2] grade propylene glycol and water (PG&W) with kinematic viscosity ranging from 0.8 mm^2/s to 58 mm^2/s. The drip time was 30 s for all of the NIST tests. The retained volume was measured by two methods on different occasions: 1) gravimetrically as described above, and 2) by collecting the retained liquid. The collection method was performed as follows. The drain valve was closed after the prescribed drip time and the neck opening was covered to prevent significant evaporation. After 6 h or more, the drain valve was opened and liquid collected in a beaker. The mass and density of the liquid in the beaker were used to calculate the retained volume.

The density of the PG&W mixtures was measured using a Rudolph Research Analytical Model DDM 2911 Plus vibrating tube density meter.[3] The instrument was calibrated with pure water and air and the PG&W mixture densities were measured between 18 °C and 26 °C in 2 °C increments and fitted with a linear function of temperature. The temperature of the PG&W mixtures during the retained volume measurements was between 19 °C and 26 °C. The water used in the retained volume measurements was reverse osmosis water and the density was calculated from the temperature using reference [9]. Considering uncertainty components due to calibration, resolution, repeatability, temperature

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[2] United States Pharmacopeial Convention.

[3] In order to describe materials and procedures adequately, it is occasionally necessary to identify commercial products by manufacturers' name or label. In no instance does such identification imply endorsement by the National Institute of Standards and Technology, nor does it imply that the particular product or equipment is necessarily the best available for the purpose.

uncertainty, imperfect cleaning, and fit residuals, we estimate the uncertainty of the density measurements of the liquids in this study to be 0.01 % with a 95 % confidence ($k = 2$).

The kinematic viscosity of the PG&W mixtures was measured with an Anton Paar AMVn falling-ball viscometer also between 18 °C and 26 °C in 2 °C increments. The data for each mixture were fitted with a 2nd order function of temperature. Considering components of reproducibility, temperature, reference liquid, viscometer cleaning, and fit residuals, the uncertainty of the viscosity for the liquids in this study was 0.75 % ($k = 2$). The viscosity of water for the MC experiments was calculated using references [10] and [11].

Small bubbles in the PG&W mixtures were a significant problem: for hours after stirring or pumping the mixtures, bubbles 0.1 mm or less in diameter were present in the liquid. Hence, gravimetric measurements were not reliable unless we waited many hours for bubbles to rise to the surface.
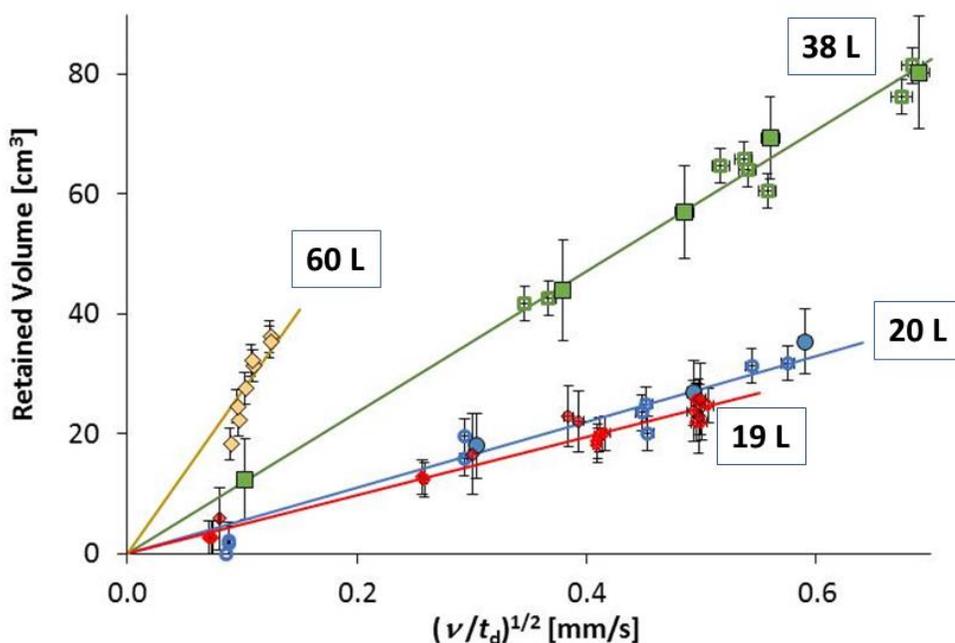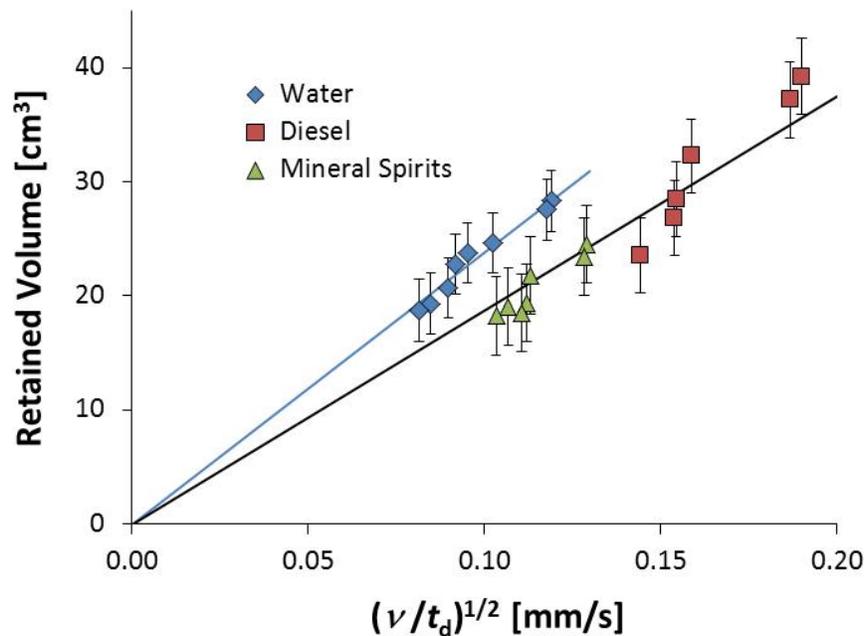

Figure 5. Retained volume in four test measures versus the square root of the ratio of kinematic viscosity to total drain time.

The retained volumes for the four test measures are plotted versus $(v/t_d)^{1/2}$ in Figure 5. Measurements made by the gravimetric method are shown as filled symbols and those made by the collection method are shown by unfilled symbols. Vertical and horizontal error bars indicate the 95 % confidence level uncertainties of the measurements. The vertical (retained volume) error bars for the NIST gravimetric data are approximately 0.03 % (6 cm^3 to 12 cm^3). They are the root-sum-of-squares (RSS) of the uncertainty presented in reference [4] and an extra uncertainty due to the density of the PG&W mixture (0.01 %). The uncertainty of the liquid collection method is smaller (approximately 3 cm^3) and is based on our estimate of the amount of liquid that might evaporate or remain on the test

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measure walls even after 6 h of waiting and the liquid density uncertainty. The gravimetrically measured retained volume has larger uncertainty than the liquid collection method because of the much larger full scale of the balance required (60 kg versus 200 g) and the resulting larger mass uncertainty. The horizontal error bars are based on the uncertainty of the kinematic viscosity (0.75 %) and the drain time (2 s). (For some data, the horizontal error bars are too small to be visible.) The agreement between the gravimetric and collection methods is within uncertainty expectations and the retained volumes for the four test measures are linear with respect to $(\nu/t_d)^{1/2}$ as one would expect from the theory of van Rossum. The lines in Figure 5 are linear best fits to each data set with intercept forced to zero. A zero intercept is a physical boundary condition unless liquid either pools or forms non-flowing drops on the test measure surfaces (which can certainly happen). Note that the drip time for the 60 L data was 65 s and it was 30 s for the other three test measures.

Measurement Canada gravimetrically measured the retained volume in a 60 L test measure using water, diesel fuel, and mineral spirits over a range of temperature conditions to obtain a range of kinematic viscosities. The results of these tests are shown in Figure 6. The data for the hydrocarbon liquids and for water show the expected linearity with respect to $(\nu/t_d)^{1/2}$, but do not collapse to a single line. (The slopes differ by 26 %.) This may be due to the high surface tension of water and a greater tendency for water to form drops on the test measure surfaces.

Figure 6. Retained volume versus $(\nu/t_d)^{1/2}$ for water, mineral spirits, and diesel fuel.

We have insufficient data over a range of test measure sizes in these well controlled experiments to form conclusions about a general relationship between contained volume and retained volume. We expect complications such as internal baffles to change the slopes displayed in Figures 5 and 6. The NIST Fluid Metrology Group has data from more than 300 gravimetrically measured contained and delivered

volumes from customer calibrations performed during the last 20+ years. The retained volume from this database is shown in Figure 7.

The variance of the retained volume is large, partly because there is a wide variety of test measure designs and aspect ratios in the sample population. Also, the uncertainty of the retained volume is large ($\sqrt{2}\,(0.00015\,V_{\mathrm{deliver}}[\mathrm{L}] + 0.012\,[\mathrm{L}])$), because its measurement is based on a small difference in mass measured with balances of 60 kg or 600 kg full scale. (The 60 kg balance is used for test measures of 38 L or smaller and the 600 kg balance is used for test measures between 38 L and 400 L.) A 3rd order polynomial fit to the data allows us to estimate the retained volume for this population of test measures when used with water near room temperature:

$$V_{\mathrm{retain}}(24.5\,°\mathrm{C})[\mathrm{cm}^3] = 0.489\,V_{\mathrm{contain}}[\mathrm{L}] - 0.001139\,V_{\mathrm{contain}}^2[\mathrm{L}] + 1.018 \times 10^{-6}\,V_{\mathrm{contain}}^3[\mathrm{L}] \quad (6)$$

The average water temperature for the data in Figure 7 is 24.5 °C. To reduce the impact of outliers and to counteract unequal data set sizes, the 3rd order polynomial was fitted to the medians at each nominal test measure size, represented in Figure 7 by the triangles at 3.8 L, 19 L, 38 L, 57 L, 94 L, 113 L, 189 L, and 378 L. The results for water for the test measure data in Figure 5 are also plotted with the same color key as Figure 5 for comparison.
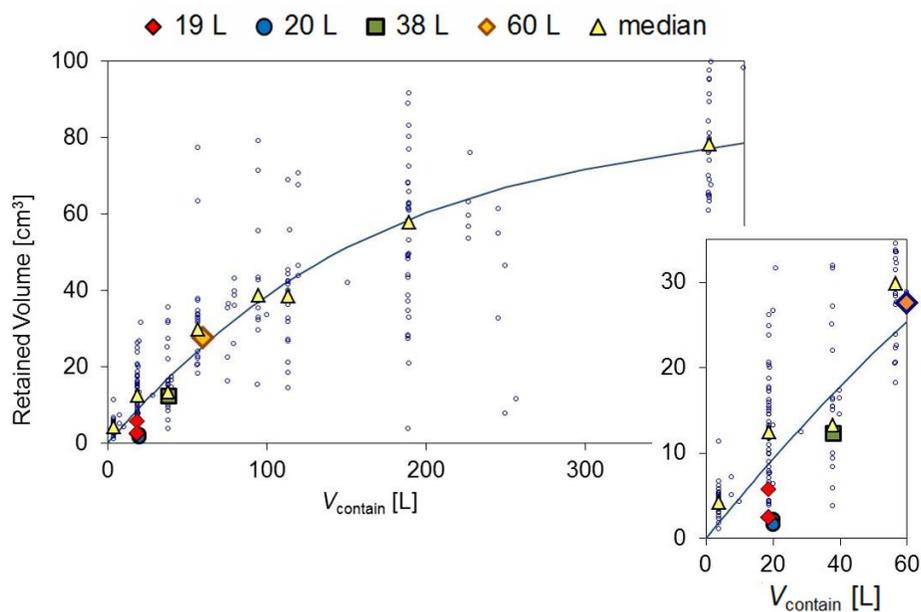
Figure 7. Retained volume from more than 300 test measure calibrations performed at NIST over a 20+ year interval.

Work remains before we can generalize the retained volume for test measures of various shapes and sizes. Fortunately, generalization across different test measures is not necessary for practical application of the drainage theory. A calibration that provides both contained and delivered volumes for specified liquid kinematic viscosity and drain time gives sufficient information to allow predictions of retained volume under other conditions. If we assume that the retained volume versus $(\nu/t_{\mathrm{d}})^{1/2}$ intercept is

zero, we can predict the retained volumes for varying fluid properties from a single experimental measurement and the van Rossum model, i.e.

$$V_{\text{retain}}[\nu(T)] = V_{\text{retain}}[\nu_{\text{ref}}(T_{\text{ref}})] \left[\frac{\nu(T)}{\nu_{\text{ref}}(T_{\text{ref}})} \frac{t_{\text{d}}[\nu_{\text{ref}}(T_{\text{ref}})]}{t_{\text{d}}[\nu(T)]} \right]^{1/2}, \tag{7}$$

where $V_{\text{retain}}[\nu_{\text{ref}}(T_{\text{ref}})]$ depends on a particular test measure (its geometry, drain configuration, draining procedure, etc.). Of course, there may be significant uncertainty in this estimate, especially if Equation 7 is applied for extrapolation rather than interpolation. Note that the drain times are not a strong function of the kinematic viscosity within the range tested and if the drain times for the two liquids are nearly equal, the final term in brackets in Equation 7 can be approximated by $[\nu/\nu_{\text{ref}}]^{1/2}$.

5. Estimates of Drainage Effects in Practical Applications

In this section, we will use the data in Figure 7 and van Rossum's theory to estimate the magnitude of drainage effects for various kinematic viscosity and drain time scenarios. In order to do so we will assume that 1) Equation 6 gives $V_{\text{retain}}[\nu_{\text{ref}}(T_{\text{ref}})]$ for water at 24.5 °C for test measures between 3.8 L and 400 L, 2) Equation 7 predicts the retained volume for a change in kinematic viscosity, and 3) drain times do not change significantly due to the changes in kinematic viscosity. Regarding the first assumption we emphasize that the retained volume for this set of test measures does not predict the retained volume for a particular test measure. For instance, some test measure designs have large diameter and short height and they will retain more liquid than a tall test measure of the same volume. Note, the data for the tall 19 L and 20 L test measures shown in Figure 3 have less retained volume than predicted by Equation 6 (see the inset in Figure 7). Regarding the second assumption, the data in Figure 6 indicates that there are drainage effects not captured in Equation 7, probably due to differences in surface tension between water and hydrocarbon liquids. Therefore the changes in retained volume for hydrocarbon liquid may be overestimated in the following analysis.

In Figure 8, we show the estimated change in retained volume that would result if a test measure where calibrated using water at 24.5 °C and the test measure was subsequently used with liquids with different kinematic viscosity. Note that for a given test measure, the total drain time will increase as the kinematic viscosity increases. Drain time data for the 19 L, 20 L, and 38 L test measures (the data in Figure 5) show that drain time increases approximately linearly with slope of 0.75 mm^{-2}. But the kinematic viscosity changes we examine below are relatively small and will not lead to significant drain time changes (less than 5 s out of $t \approx 100$ s), hence drain time was considered a constant in the following estimates and the retained volume considered proportional to $[\nu/\nu_{\text{ref}}]^{1/2}$.

Figure 8 displays results for the following liquids (and kinematic viscosities) : #2 fuel oil (7 mm^2/s), #2 diesel (4 mm^2/s), gasoline (0.5 mm^2/s), and water at 14.5 °C (1.13 mm^2/s). The uncertainty of the NIST calibration service for neck scale test measures ($U_{V_{\text{deliver}}}[\text{L}] = 0.00015\, V_{\text{deliver}}[\text{L}] + 0.012\,[\text{L}]$) is also shown in Figure 8 to aid the reader in assessing the significance of the drainage effects. A 5 gallon test measure, the size normally used to verify that fuel dispensers are calibrated within maintenance tolerance of 0.5 %, will retain approximately 0.01 % less liquid when used with gasoline than with water at 24.5 °C. When used with diesel fuel, it will retain approximately 0.05 % more liquid.

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If the total drain time t_d for a particular liquid is doubled between calibration and usage (e.g., due to changes in the piping configuration, drain valve not fully open, or not using the same drip time), Equation 7 predicts that the retained volume will be reduced by a factor of $1/\sqrt{2}$. If the drain time is halved (e.g., due to a hose attached to the drain increasing the head), the retained volume increases by a factor of $\sqrt{2}$. These scenarios are plotted in Figure 9 for a range of test measure sizes. For a 100 L test measure, these scenarios change the delivered volume by approximately +0.03 % and -0.06 % respectively.
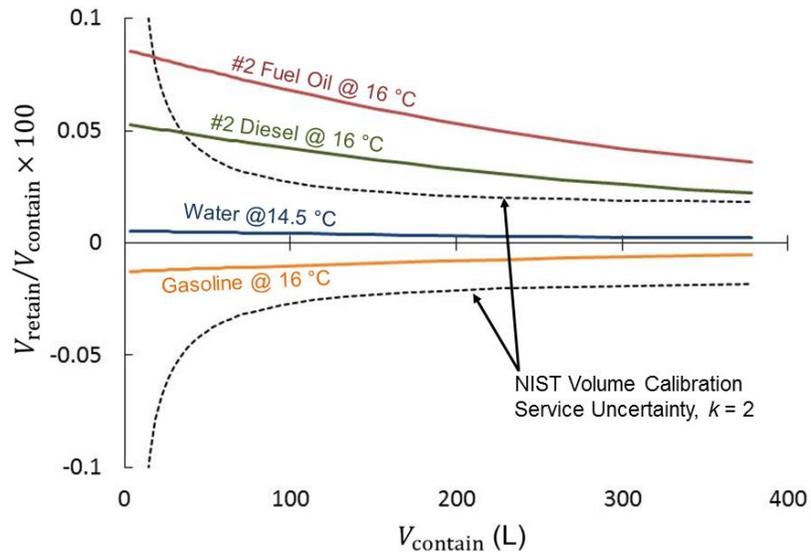
Figure 8. Change in retained volume, as a percent of the test measure contained volume, for test measure up to 400 L and various liquids. The uncertainty of the NIST liquid volume calibration service is shown for comparison purposes.
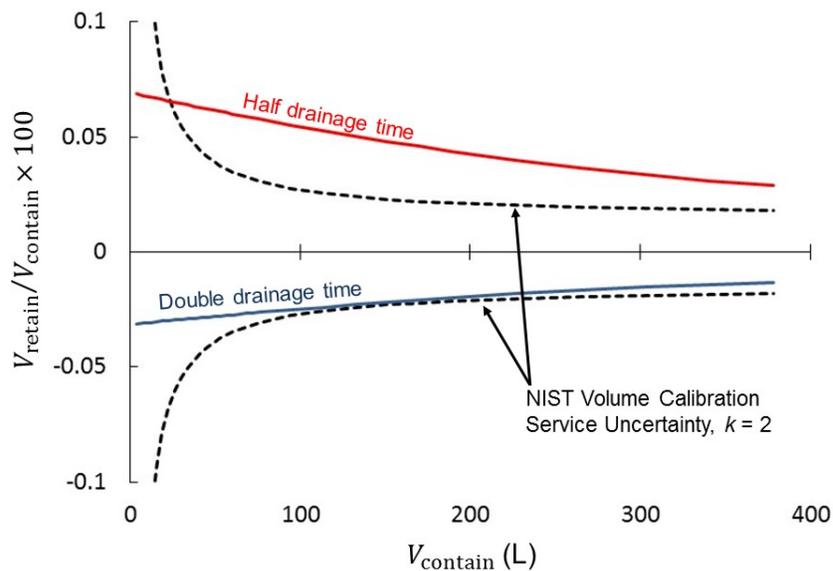
Figure 9. Change in retained volume, as percent of contained volume, for halved or doubled drain times.

6. Conclusions

A test measure is a simple but precise volume measurement instrument. Special test measures designed to drain to nearly dry condition have demonstrated long term reproducibility of less than 5 µL/L [12]. But for typical test measure designs, inconsistent operating methods, a change in liquids, or different environmental conditions between calibration and usage introduce uncertainties due to drainage effects that are on the order of 0.01 % to 0.05 %. One can reduce drainage-caused uncertainties by using the same drain piping configuration, liquid with the same kinematic viscosity, and the same drain and drip times. Calibrating a test measure with water and then using it with a different liquid introduces a bias from drainage effects that can be reduced by accounting for the change in the liquid's kinematic viscosity. We have experimentally verified the drainage theory of van Rossum for four test measures: retained volume is proportional to $(v/t_{\mathrm{d}})^{1/2}$. The drainage theory allows estimation of uncertainty due to drainage effects and predictions of delivered volumes for different drain times.

A database of retained volumes for more than 300 customer calibrations was fitted with Equation 6. We used this equation to estimate drainage effects for test measures up to 400 L. One can estimate the retained volume for a test measure by combining the retained volume for water at 24.5 °C from the NIST sample of test measures with the $(v/t_{\mathrm{d}})^{1/2}$ theoretical dependence. This approach was applied (assuming constant drain time) and the magnitude of drainage effects was as large as 0.08 % for the scenarios we considered in Figures 8 and 9. Note that the results for a particular test measure can differ markedly from these estimates. For example, there is a large variance of retained volumes used to obtain Equation 6, probably because of the wide range of shapes and surface conditions for the test measures in this data set.

The theoretical model for retained volume that we employed does not consider wetting effects (surface tension and contact angle). The differences in retained volumes for water and hydrocarbon liquids in Figure 6 imply that the theoretical model could be improved by including wetting effects.

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