DESIGN, CALIBRATION, AND OPERATION OF VOLUME STANDARDS
Class #4040

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Introduction

Test measures are designed to deliver a known liquid volume when drained. Their primary use is to calibrate displacement and tank provers in the field by the waterdraw method.[1, 2] Accurate test measure volume calibrations are critical to achieving low uncertainty calibrations with flow provers in the field. Test measures can either be invertible or bottom-drain type. Invertible test measures are usually less than 40 L (10 gal)[†] while bottom-drain test measures are larger than 40 L (10 gal). Each year, approximately 100 test measures used for field calibrations are calibrated at the National Institute of Standards and Technology (NIST) to comply with API standards.[3] NIST uses several calibration methods depending on the size of the test measure: 1) the gravimetric method, 2) the gravimetric transfer method, and 3) the volumetric method.[4] NIST calibrations include a neck scale calibration at five levels spaced over the range of the neck scale. To ensure accurate customer calibrations and to maintain an ISO 17025 compliant quality system[5] NIST regularly performs calibrations of the 60 kg and 600 kg balances used during the calibration and calibrates volume check standards.

Test Measure Design

Manufacturers of test measures follow the specifications and tolerances outlined in NIST Handbook 105-3.[6] Test measures are generally selected in multiples of 4 L, 8 L and 20 L (1 gal, 2 gal and 5 gal) for nominal volumes. They are generally made of corrosion resistant 300 series stainless steel. Test measures are round in shape and vary in height depending upon the volume. The horizontal cross section of a test measure is circular and its shape permits complete draining. Test measures must be rigid to prevent distortion when full of water. Reinforcement bands are used at the transition from the body to the top and bottom cones. Larger test measures sometimes have a band around the middle for extra support from distortion. The inside must be smooth and clean. All welds must be smooth, dent free, and there must be no crevices or pockets where air bubbles, liquid, or foreign material can be entrapped. To prevent distortion to the neck, the opening at the top is reinforced by a rolled bead or welded band. Two types of test measures are commonly used 1) invertible test measures that are drained by pouring through the neck and 2) bottom drain test measures that are drained by opening a valve (see Figure 1).

Contained volume is the volume of liquid required to fill a dry test measure to the full reference mark. Delivered volume is the volume of liquid that drains from a test measure, originally filled to the full reference mark, under specified conditions. For a test measure with a bottom drain, it is normally the volume that drains from the test measure during the time from the opening of the valve to the closing of the valve 30 seconds after the cessation of the main flow. The correct method to drain an invertible test measure is to pour the full contents in 30 seconds, with a 10 second final drain with the test measure held at a 75 to 80 ° angle from the horizontal.[3, 6] Delivered volume is also the volume of water required to fill a test measure to its full reference mark once it has been pre-wetted by being filled to the full reference mark and then drained under the specified conditions. The contained volume is always larger by a few hundredths of a percent than the delivered volume for a given test measure because water clings to the wall after draining. Since test measures are used more than once to accumulate the total volume of large provers, delivered volume is the quantity of interest to most users. Contained volume is of interest because it is not affected by

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† The US gallon (231 in^3, 0.003785412 m^3, 3.785412 L) is the common unit of liquid volume used in commerce in the United States. Accordingly, to better communicate with the intended audience, in this publication US gallons are used in descriptions of test measures for which the manufacturers have used US gallons, rather than the equivalent of gallons expressed in the less familiar m^3 of the International Systems of Units (SI). SI units are normally used in publications of the National Institute of Standards and Technology.

the amount of water that clings to the walls and therefore is independent of the cleanliness of the test measure and the properties of the liquid used.

Bottom drain test measures have a cone shaped top with a cylindrical neck, and a cone shaped bottom with a drain line and valve. Installed in the bottom of the test measure is an anti-vortex plate, a vertical stainless steel plate mounted inside the test measure to remove swirl from the draining liquid. A drain line is attached to the bottom cone at a downward slope of at least 5° from horizontal in order to provide complete drainage. The drain valve is an important component of the calibrated volume. Test measure drain valves shall be sealed to indicate whether the valve has been changed, modified, replaced or repaired in any way that affects the integrity of the volume determined at the last calibration. Many test measures have drain valves that are simply screwed into the drain line. The volume is altered if this valve is turned to a different number of threads, either more or less. The importance of this valve to the calibrated volume cannot be overemphasized and properly sealing drain valves should be an absolute priority for volumetric test measures. A metal-to-metal flange arrangement between the drain line and the drain valve is the preferred method of drain valve connection[3].

There are two common types of neck designs for test measures, normal or high sensitivity. Normal sensitivity necks have a larger diameter neck with less scale resolution. A high sensitivity neck has smaller diameter and therefore greater neck scale resolution. For example a 380 L (100 gal) test measure with a normal sensitivity neck will have graduations of 164 ml (10 in^3) and a high sensitivity neck will have graduations of 33 ml (2 in^3).

Test measures are equipped with a sight glass made of borosilicate glass, mounted to the side of the neck, normally directly above the drain valve. The sight glass is sealed using O-rings in the top and bottom brackets (as opposed to using a sealant). The scale plate is made of corrosion resistant metal, usually stainless steel or aluminum. It is mounted to the front of the sight glass, no more than 6 mm (0.236 in) from the glass. The scale divisions should not be less than 1.6 mm (1/16 in) apart or the lines will be too close together to get accurate readings of the meniscus. The zero mark is normally in the middle of the neck of the test measure. The zero mark represents the nominal volume of the test measure. The scales are mounted to yokes with two adjusting rods running parallel to the sight glass. The scale is wired and sealed in place upon calibration. The seal must be installed in such a way that the scale can not be adjusted vertically without removing the seal. After calibration, NIST seals the scale in the upper left corner. If the seal is missing, the test measure must be recalibrated.

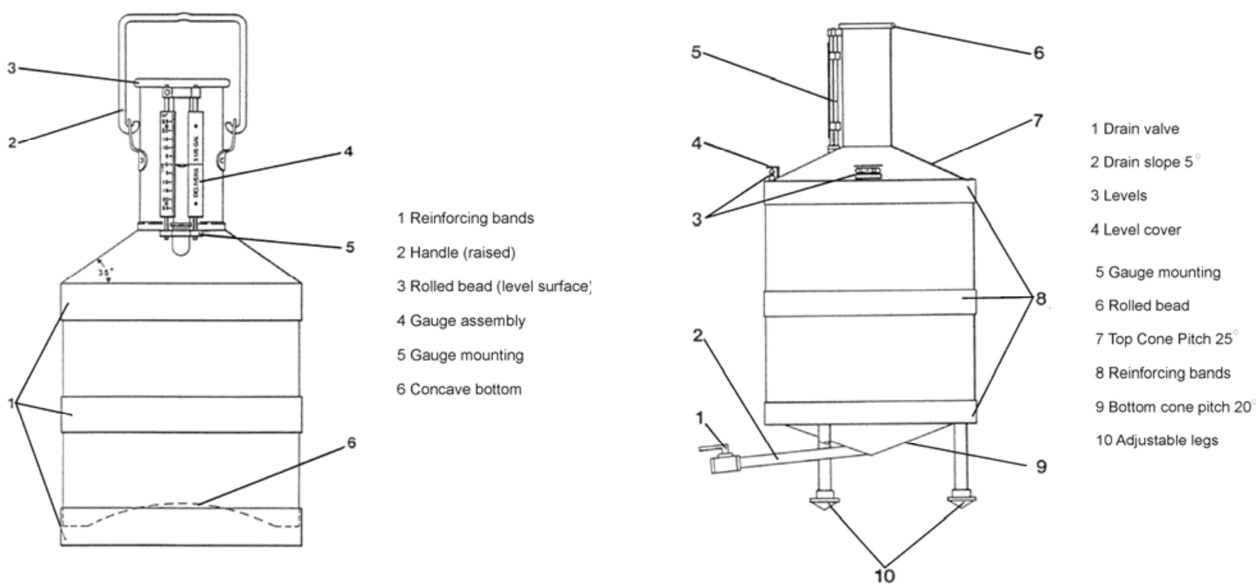

Figure 1: Invertible test measure (left) and bottom drain test measure (right).[6]

Leveling of a bottom drain test measure is done by placing a precision machinist's spirit level on the side of the neck in two places, one in the same plane as the sight glass, the other 90 degrees around the neck. This is done to check the attached spirit levels on the test measure. When calibrating an invertible test measure, it is placed on a flat, leveled surface so that the test measure is level across its bottom.

Test measures are precision liquid measurement instruments vital to the calibration of meter provers and tank provers in the field. Therefore, great care should be taken to keep them free from dents and debris. Depending on size, a protective case is a good method of protecting and storing test measures.

Figure 2: Photographs of 4 L, 400 L, and 2000 L (1 gal, 100 gal, 500 gal) test measures.

NIST Calibration of Test Measures

NIST provides calibration services for metal test measures for volumes up to 7600 L (2000 gal). Test measures with volumes up to 1900 L (500 gal) are calibrated gravimetrically. Test measures over 1900 L (500 gal) are too large to fit into our gravimetric laboratory and are calibrated by volume transfer using NIST test measures that have been calibrated gravimetrically.

The volume transfer method was used at NIST for all customer calibrations prior to 1997. In 1996 NIST and American Petroleum Institute worked together to develop a new facility to enable test measures to be calibrated by the gravimetric method. This laboratory came online in 1997 and the gravimetric method has been the primary method used since then.

Gravimetric Method

In the gravimetric method, the volume of distilled water required to fill a test measure up to its full reference mark is determined by measuring the mass and the temperature of the water, calculating the density of the water from an established equation, and then calculating the volume from the definition of density.[4] Described step by step, the temperature sensors are inserted through the top of the test measure and it is filled with water to approximately the zero mark on the neck scale. The temperature of the water is taken and the sensors are removed, leaving only the test measure and its contents on the scale. Then the weight of the test measure and the visually read neck scale reading are recorded. The test measure is drained and the drained weight is recorded. This process is repeated five times. From these measurements, we calculate the delivered volume for the test temperature via the following equations:

$$V_{deliver}(T_w) = \frac{m_f - m_d}{\rho(T_w) - \rho_{air}} + h \quad , \tag{1}$$

and,

$$V_{deliver}\left(T_{ref}\right) = V_{deliver}\left(T_W\right)\left[1 - \beta_t\left(T_W - T_{ref}\right)\right] \quad , \qquad (2)$$

where T_w is the water temperature, m is mass (f represents full condition, d represents drained condition), ρ is the density of the water, ρ_{air} is the room air density, h is the neck scale reading, β_t is the thermal expansion coefficient of the test measure metal, and T_{ref} is the reference temperature (normally 15.56 °C or 60 °F). The first equation gives the volume of the test measure at the particular temperature of the water during the calibration while equation 2 uses the metal thermal expansion coefficient to give the test measure volume at the reference temperature. The density of air, ρ_{air} is calculated from measurements of the room temperature, pressure, and relative humidity.

Gravimetric Transfer Method

The gravimetric transfer method is used for test measures that are too heavy to weigh on our 600 kg scale. In this case, the scale is moved from the floor to a raised platform in the volume lab. A vessel that holds approximately 400 L (120 gal) of water is placed on the scale. Batches of water are weighed in the 400 L (120 gal) vessel and then drained into the test measure being calibrated. The sum of the masses of these batches along with the water temperature and neck scale reading in the test measure and the room air density are used to calculate the volume. It is important that the drain spout from the 400 L (120 gal) vessel be well inside the test measure neck to avoid loss of water by splashing. Also, the drain spout must not touch the test measure or this will lead to mass measurement errors.

Volume Transfer Method

In the volume transfer method, a test measure is calibrated by filling it to its full reference mark with a known volume of tap water determined using working standard test measures which were previously calibrated by the gravimetric method. In this method, it is necessary to measure temperature in both the working standard test measures and the test measure being calibrated in order to account for thermal expansion of the water and the metal of the test measure.

Neck Scale Calibration

Every test measure that comes to NIST for calibration also has a neck calibration performed. During the fifth run, the test measure is filled to five levels over the range of the neck scale. At each level, the test measure weight and a visual neck scale reading are recorded. The measurements are used to calculate the delivered volume represented by each of the five neck scale positions tested and to calculate a correction for each. This data is presented in graphical form in the report of calibration (see figure 3).

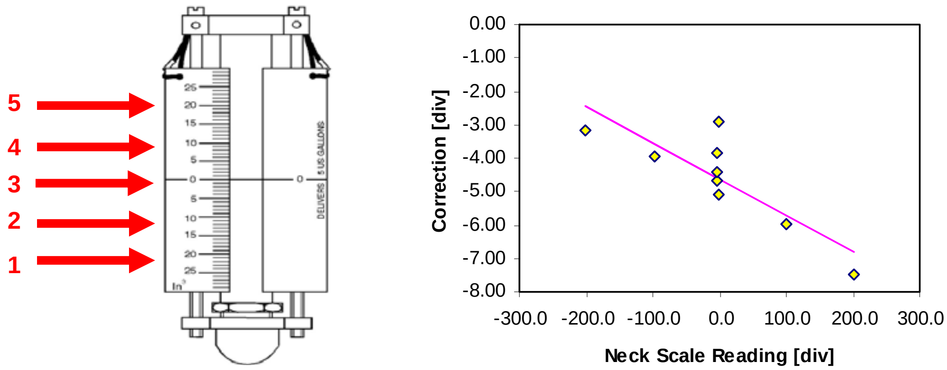

Figure 3: Neck scale calibration locations and sample results.

Uncertainties

The uncertainty of a NIST volume calibration depends upon the method used as well as the design and size of the test measure. For the most commonly used gravimetric method, the uncertainty components are 1) the operator's reading of the neck scale, 2) the full and drained masses of the test measure, 3) the density of the water, 4) the thermal expansion coefficient of the test measure metal, 5) the temperature of the water, 6) the density of the room air, 7) drainage effects, and 8) the repeatability of the five volume measurements.[4] A unique uncertainty calculation is made for each test measure that NIST calibrates, using data from the actual calibration. The most significant of the uncertainty sources are the repeatability, mass measurements, and drainage effects. Which of them is most significant depends on the design and cleanliness of the test measure and what portion of the balance range is being used. For instance, a high sensitivity neck scale will normally have better repeatability than one with normal sensitivity. A test measure that is poorly cleaned will show larger differences between the contained and delivered volume leading to a larger uncertainty due to drainage effects. A test measure that is slightly too heavy for the 60 kg scale (40 L (10 gal)) must be tested at the low end of the 600 kg scale where its uncertainty as a percent of reading is larger.

Typical uncertainties for neck scale test measures are 0.015 % + 0.003 gal or 0.015 % + 0.012 L [about 0.3 % for a 3.8 L (1 gal) test measure and 0.02 % for a 380 L (100 gal) test measure]. This specification is shown as the solid curve in figure 4. NIST uses a coverage factor $k = 2$ in the calculations to give approximately 95% confidence level for the uncertainty. The symbols in figure 4 are unique uncertainty calculations for approximately 100 customer test measures.

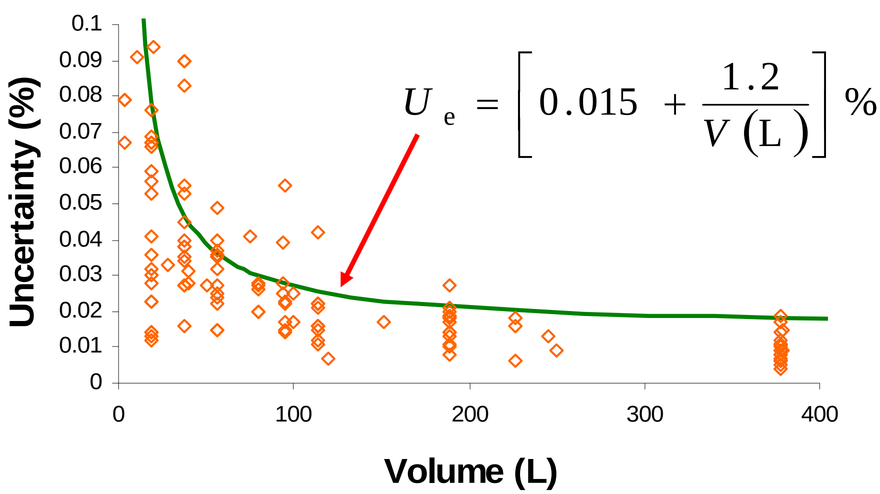

Figure 4: Uncertainty plot for NIST calibration of test measures

Check Standards and Comparisons

Check standards serve to verify the quality of results from the NIST volume calibration service. Check masses are used on the 60 kg and 600 kg balances before they are used to perform volume calibrations. A stack of six 45 kg masses is placed on the 600 kg balance and if the buoyancy corrected measurement does not agree with the true mass within 10 g (22 parts in 10^6), we perform a calibration with 12 points over the range of the balance and determine a new balance calibration equation. A 20 kg check mass with a 3 g tolerance triggers a full calibration of the 60 kg balance. Also anytime that the balance is moved (for instance from the floor to the raised platform) the balance undergoes a full calibration.

Figure 5 is a control chart for our 380 L (100 gal) check standard test measure. This check standard is used periodically to detect problems anywhere in the volume calibration process. Points outside of the volume service specifications trigger a root cause analysis investigation and corrective actions.

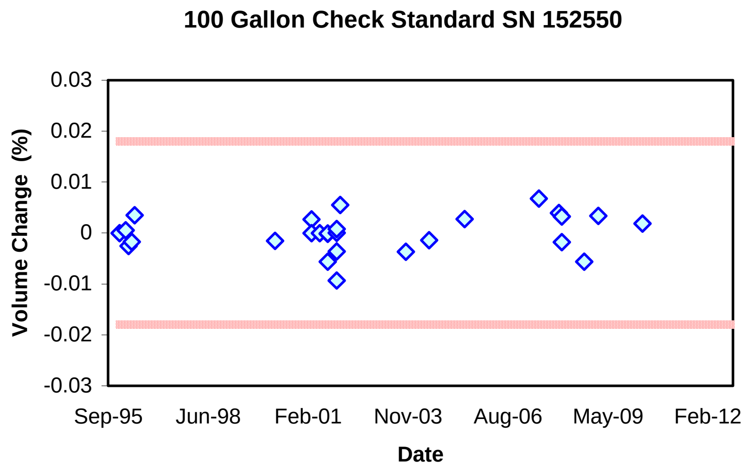

Figure 5: Control chart for the NIST 380 L (100 gal) test measure (Serial Number 152550). Change between current volume and prior volume measurements in percent. Red horizontal lines represent 95 % confidence level tolerance for this size test measure.

In addition to the internal checks, NIST participates in international comparisons with other national metrology institutes. Figure 6 presents results of a volume key comparison between the NIST and seven other national labs: CENAM (Mexico), MC (Canada), SP (Sweden), PTB (Germany), IMGC (Italy), NMIA (Australia), and INMETRO (Brazil).[7] The comparison was performed with three 20 L test measures specially designed for good repeatability, represented by the sets of three diamonds for each participant. The square for each lab indicates the average result for each lab and shows NIST in agreement with the reference value within 26 parts in 10^6 (0.0025 %). Error bars represent approximately 95% confidence level uncertainties. Figure 7 presents the results of a regional comparison covering North and South America completed in 2008 using a 20 L volume. The NIST results agree with the comparison reference value within 50 parts in 10^6 (0.005 %).[8]

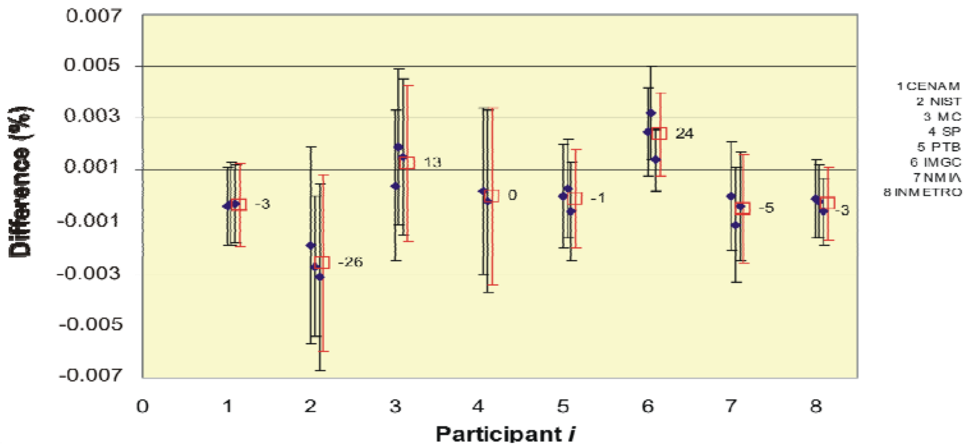

Figure 6: Results CCM.FF K4, a key comparison between 8 national laboratories performed in 2005.

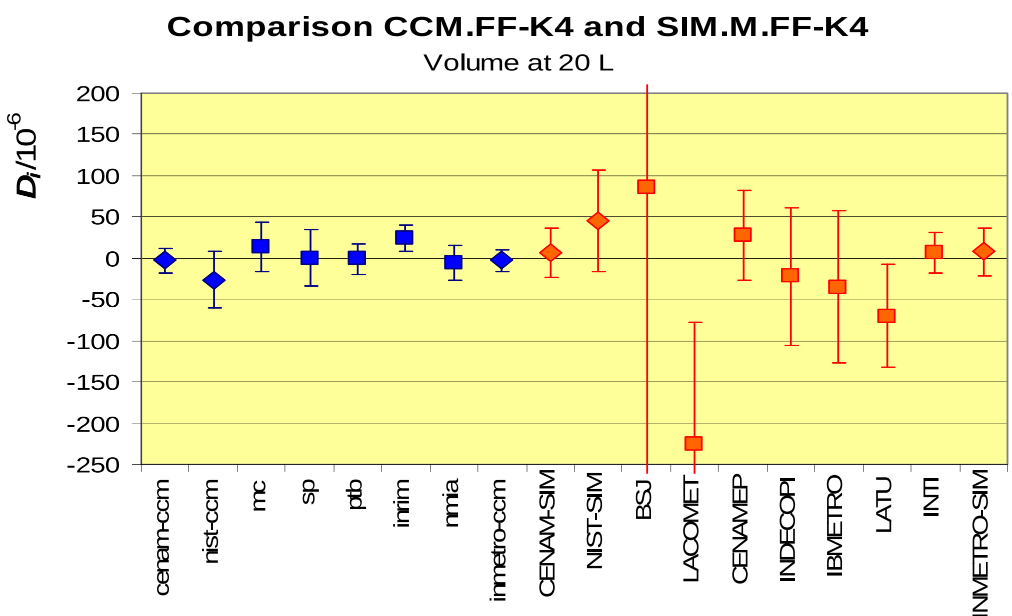

Figure 7: Degrees of Equivalence related to CCM.FF-K4 and SIM.M.FF-K4. *DoE*s were calculated for the corresponding reference value (KCRV for CCM.FF-K4 and RCRV for SIM.M.FF-K4). Diamond marks corresponds to the linking NMIs. Conducted January 2007 to December 2008

Operation and Use

Test measures are used to determine the volume of pipe provers, small volume provers, and tank provers by the waterdraw method of calibration. Detailed procedures for field use of these test measures for waterdraw calibration can be found in API MPMS, Ch 4.9.2 “Determination of the Volume of Displacement and Tank Provers by the Waterdraw Method”.[9] Test measures are also used in the calibration of metering systems that measure the quantity of material delivered through a pipeline or into a transport vehicle for petroleum products and other materials such as milk, water, and chemicals. Test measures come in various sizes anywhere from 4L up to 6000 L (1 gallon up to 1500 gal). Test measures are often made to be the exact size of small volume provers for convenience.

With the graduated neck test measures it is not necessary to operate at the zero level. Therefore, the test measure can be filled to any location on its scale, the liquid level read, and its certified capacity then adjusted mathematically using a plus or minus scale reading. A minus signifies that the water level is below the certified volume, and a plus indicated that the water is above the zero mark on the scale.

When calibrating a meter prover by the waterdraw method, multiple fills of one or more test measure may be necessary for each run. It is recommended that the number of fills be kept to a minimum and should not exceed 15 times, in order to keep the uncertainty of the calibration within reasonable limits.

1. The test measure is filled with water to an upper scale reading.
2. The level of the test measure is verified.
3. The water level in the sight glass, from the bottom of the meniscus, is read on the scale plate and is recorded as a (+) or (-) volume, depending on whether the reading is above or below the zero line on the scale.
4. The temperature of the water in the test measure is recorded by immersing a thermometer in the measure or by putting the thermometer in the stream of flow while it is draining.
5. Drain the test measure in the prescribed manner to reestablish the wet down condition, with the consistent amount of clingage, for the next run.

Conclusion

Test measures are precision liquid volumetric measurement instruments used to calibrate dynamic provers or tank provers by the waterdraw method. They are designed and constructed to detailed specifications outlined by NIST Handbook 105-3 and are calibrated for their “to deliver” volume at NIST. They are carefully manufactured to avoid air or liquid entrapment that might increase the volume uncertainty or increase the repeatability of successive tests. Although very durable, they should be handled and transported with care, to avoid jeopardizing the integrity of their calibration.

NIST reports contained and delivered volume for test measures at a specified reference temperature. These test measures are used to perform field calibrations of tank provers and dynamic provers (ball provers and small volume provers). This provides NIST traceability to petroleum flow measurements. Reliable, low uncertainty volume calibrations are critical for obtaining low uncertainty flow measurements for petroleum custody transfer.

References

[1] Design, Calibration and Operation of Field Standard Test Measures, Class 4040, Richard C. Jordan, Seraphin Test Measure Company.

[2] Design, Calibration and Operation of Field Standard Test Measures, Class 4040, William R. Young, Jr., Meter Engineers, Inc.

[3] API Manual of Petroleum Measurement Standards, Chapter 4, Section 7 – Field Standard Test Measures, Third Edition, April 2009.

[4] V.E. Bean, P.I. Espina, J.D. Wright, J.F. Houser, S.D. Sheckels, and A.N. Johnson, NIST Calibration Services for Liquid Volume, NIST Special Publication 250-72, National Institute of Standards and Technology, Gaithersburg, MD, November 24, 2009.

[5] General Requirements for the Competence of Testing and Calibration Laboratories, ISO 17025, International Organization for Standardization, Switzerland, 1999.

[6] Handbook 105-3, Specifications and Tolerances for Graduated Neck Type Volumetric Field Standards National Institute of Standaards and Technology, Gaithersburg, MD, 2010.

[7] Arias, R., Maldonado, M., Wright, J., Jacques, C., Lachance, C., Lau, P., Tobben, H., Cignolo, G., Lorefice, S., Man, J., and Aibe, V., *Results of the Key Comparison CCM.FF-K4 for Volume of Liquids at 20 L and 100 mL*, Technical Supplement to Metrologia, 43, 2006.

[8] Arias, R., Maldonado, M., Wright, J., Wallace, T., Rodriguez, S., Pinzon, O., Morales, A., Vega, M., Santo, C., Kornblit, F., and Malta, D., *Final Report on Regional Key Comparison for Volume of Liquids at 20 L and 100 mL*, BIPM Key Comparison Database, January, 2010.

[9] API Manual of Petroleum Measurement Standards, Chapter 4, Section 9 Methods of Calibrations for Displacement and Volumetric Tank Provers. First Edition, October 2005.