

Removing the Hydrocarbon Liquid from Hydrocarbon Liquid Flow Standards

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Abstract

NIST and other laboratories calibrate meters that measure the flow of valuable liquids such as petroleum products. Historically, these calibrations have been conducted using Stoddard solvent. Laboratories using Stoddard solvent for liquid-flow calibrations must contend with flammability, inhalation toxicity, and hazardous waste disposal requirements. In response to health and safety concerns, we modified NIST's piston-prover flow standards so that calibrations are now conducted with mixtures of propylene glycol and water (PG + W). In this paper, we describe the modifications to NIST's calibration systems and our experiences in using the modified systems to calibrate turbine, positive displacement, and coriolis meters.

Introduction

NIST has successfully changed the liquid in its piston-prover Liquid Flow Standards (LFS) from Stoddard solvent to a mixture of propylene glycol and water (PG + W) to eliminate problems resulting from Stoddard solvent's flammability, inhalation toxicity, and hazardous waste disposal requirements. The change in calibration fluid required us to modify the LFSs to resist rusting. The impact of the change on meter calibrations is negligible. We discuss the required modifications and we present data for turbine, positive displacement and coriolis meters that show their compatibility with PG + W and the validity of calibrations conducted with PG+W.

Flow meters are widely used to measure the flow of valuable fluids such as natural gas and petroleum products. Therefore it is important to have primary calibration standards with low uncertainty. NIST has two primary LFSs that are piston provers; the 2.5 L/s LFS and the 0.1 L/s LFS with uncertainties of 0.06 % and 0.03 % ($k=2$) respectively. Historically the fluid used in these standards was Stoddard solvent. Stoddard solvent is a light mineral oil with a kinematic viscosity of ≈ 1.2 cSt at 20 °C. ($1 \text{ cSt} = 10^{-6} \text{ m}^2\text{s}^{-1}$). Because Stoddard solvent is a hazardous chemical [1], the substitution of it with PG + W has been of interest [2,3,4]. PG + W mixtures are benign fluids [5] and a mixture containing 5 % PG by volume has a kinematic viscosity that matches the kinematic viscosity of jet fuel (≈ 1.2 cSt at 20 °C). Calibration laboratories are utilizing PG + W mixtures to decrease the cost of maintaining multiple standards with fluids of various kinematic viscosities and to reduce: 1) the danger associated with flammable fluids, 2) pollution and 3) the expense and risk of disposal of harmful substances.

Water Compatibility of the Standards

Piston prover systems have long been accepted as primary flow calibrators for liquid flowmeters such as turbine, positive displacement and coriolis flowmeters [6,7]. In its most basic form, the piston prover consists of a circular cylinder of known internal diameter, which encompasses a sealed piston. This piston strokes through measured lengths, at a constant speed, to produce a volumetric flow. The volumetric flow is calculated by dividing the volume displaced by the moving picture by the time it takes the piston to traverse the measured length. Figure 1 is a schematic of NIST's piston provers. The valve assembly allows for the piston to move back and forth while the flow at the meter under test (MUT) is unidirectional.

The LFSs were constructed by Flow Dynamics Inc, in Scottsdale, AZ¹ and were designed to be used in hydrocarbon liquid and therefore were susceptible to rust. Both LFSs had rusted components that we replaced. Figure 2 shows the LFSs with the plumbing that was replaced outlined in red and the added filters outlined in white.

Four sources of rust were found in the 2.5 L/s LFS: 1) filters made of steel wire screen inside steel canisters (the main source of rust), 2) the exposed ends and threads of galvanized pipe, where the zinc had been removed by cutting, 3) The fitting for the hose connection for the fluid intake, which was made of steel and 4) the inlet to the system from the fill pan under the MUT, which was made of steel.

To minimize rust, we 1) replaced filters with filters designed for water, 2) replaced fittings (where possible) and pipes made of steel with similar components made of stainless steel or reinforced polyethylene, 3) cleaned the inside of the cylinder, 4) cleaned the piston and replaced both seals, 5) replaced both shaft seals and 6) added a filter on the inlet from the fill pan. Figure 3 shows the cylinder of the 2.5 L/s LFS disassembled for cleaning and seal replacement. Following the cleaning and seal replacement, temporary tubes and a recirculating pump were installed to flush each branch of the LFS's plumbing system individually with pure water. A filter in the return line to the pump captured the rust removed by flushing.

We modified the 0.1 L/s LFS after modifying the 2.5 L/s LFS; therefore, the smaller system benefitted from our

¹ Certain commercial equipment is identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by NIST, nor does it imply that the equipment identified is necessarily the best available for the purpose.

experience. We used a magnet to identify ferrous components. Those components were replaced with polyethylene tubing and polypropylene compression tube fittings (Shown in Figure 2B outlined in red). Filters were placed at the fill pan inlet and at the exit of the reservoir tank such that all the PG + W mixture flows through the filters before entering the test section. The 0.1 L/s LFS uses a pump² to move the liquid that, in turn, moves the piston. Therefore, the pump was replaced with a water compatible one.

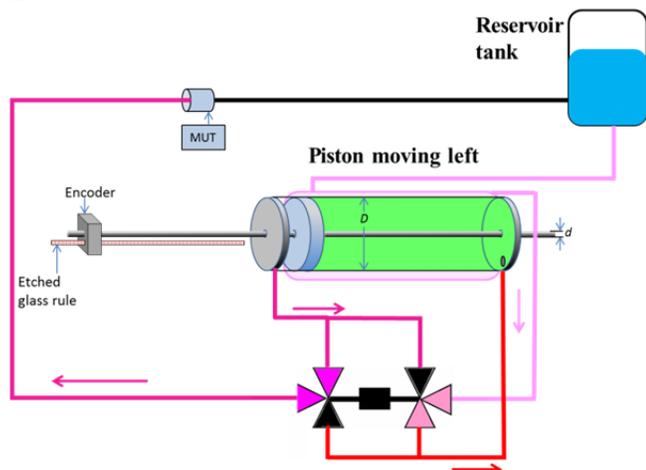


Figure 1. Sketch of the piston prover with the piston stroking left.

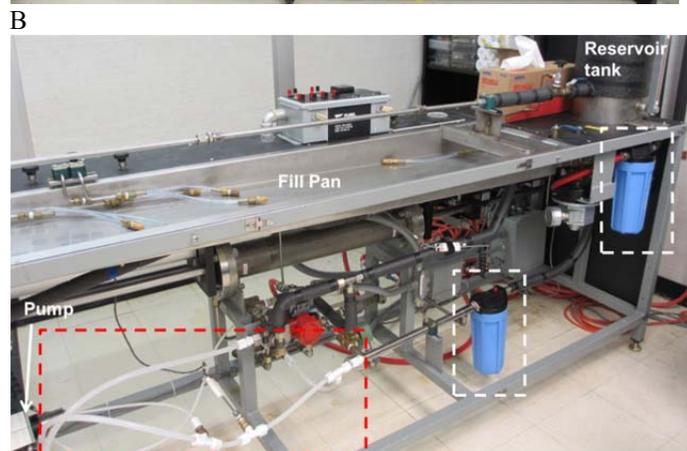


Figure 2. A) The 2.5 L/s LFS. B) The 0.1 L/s LFS. The plumbing that was replaced is outlined in red. The filters that were added are outlined in white.

²Two types of piston arrangements are generally used in these systems. An *active* piston can both drive and measure a volumetric flow out of the cylinder (like a syringe), while a *passive* piston is pushed through the cylinder by pressure from a separate pump. The 0.1 L/s LFS employs a *passive* piston and the 2.5 L/s LFS employs an *active* piston.

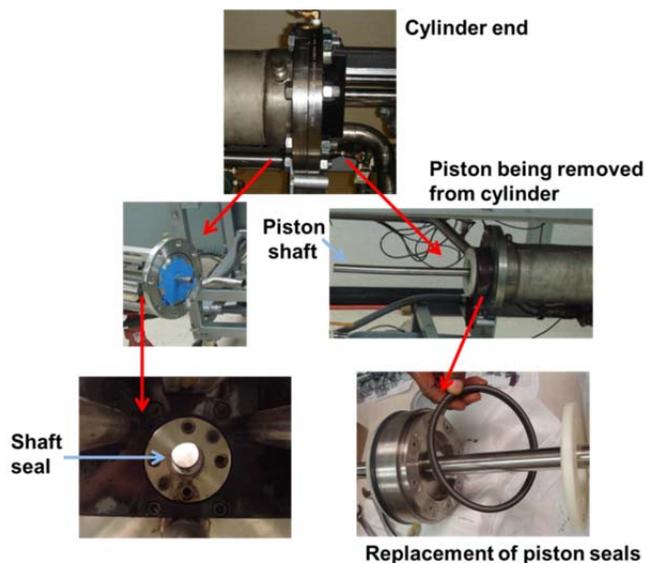


Figure 3. The cylinder of the 2.5 L/s LFS disassembled for cleaning and seal replacement.

The battle with rust is ongoing. We anticipate the multiple filters will remove any remaining rust. The 2.5 L/s LFS has been using water or 5 % PG + W for nearly two years. There is no visible rust present in the PG + W solution when the LFS is drained. The 0.1 L/s LFS has been using a 5 % PG + W mixture for nearly six months. We check the filters every four months to check for rust. Welds that depended on flux for shielding are the main source of new rust. When feasible, rusty welds will be replaced with welds utilizing tungsten inert gas shielding that resist rusting.

We tested three different materials for the piston and shaft seals to determine which lasts longer in contact with water. The three materials are: 1) virgin polytetrafluoroethylene (PTFE), 2) carbon-filled PTFE and 3) polymer-filled PTFE. Thus far, we have not noticed differences in the wear rates of the different seal materials.

Water Compatibility of Meters and Validation of PG + W Calibrations

When a PG + W mixture is used in a primary flow standard, to calibrate a meter for measuring the flow of liquid hydrocarbons, the meter calibrated with the standard must be water compatible and the calibration must be independent of the properties of the calibrating liquid.

Turbine Flowmeters

Turbine flowmeters consist of a bladed turbine rotor supported by one or more bearings, which is mounted inside a flow tube. Each rotation of the rotor generates an electrical signal which is converted to a flow rate [9]. NIST uses multiple turbine meters of varying size and consisting of single and dual rotors as check standards on the LFSs.

Rusting of turbine meter bearings is a concern. Turbine meters can be purchased with ceramic bearings that are resistant to rust. However, many turbine meters have bearings that are made from 440C stainless steel, which may

not be corrosion resistant. We were concerned that these bearings would rust even during a short (typically 8 hours) calibration period. Therefore, we tested two 2.5 cm dual rotor turbine meters with 440C bearings to measure the effects of water on their performance. The test lasted nine months. One meter was calibrated on the 2.5 L/s LFS in the 5 % PG + W mixture, immediately removed from the test section, rinsed with ethanol and dried using a stream of nitrogen. This meter was stored in a clean dry place in between calibrations. The second meter was also calibrated on the 2.5 L/s LFS in the 5 % PG + W mixture; however, following calibration, this meter was neither cleaned nor dried. Instead, it was stored with water inside it between calibrations. (The water had been purified by reverse osmosis; however, it had not been degassed.)

Figure 4 shows the ratio of the upstream rotor frequency to the downstream rotor frequency for eight calibrations starting in July of 2012 and ending in March of 2013. The calibrations of these turbine meters were not affected by exposure to water. Generalizing from this experience, we concluded that turbine meters with 440C bearings can be calibrated in PG + W mixtures without adverse consequences.

The calibration of turbine meters is independent of the kinematic viscosity of the flowing liquid if the meter is used in its low-uncertainty range (usually a 10:1 turndown ratio). Below this range, the kinematic viscosity affects the calibration regardless of the liquid used, particularly because the kinematic viscosity is temperature dependent. NIST has demonstrated this 2.5 cm-diameter single and dual rotor turbine meters and in a 1.25 cm-diameter dual rotor turbine meter [3,4,8]. The 2.5 L/s LFS was used to validate the calibration of these meters. Further validation has been made using the 0.1 L/s LFS.

Figure³ 5A shows 5 years of the calibration history of a 1.25 cm-diameter single rotor turbine meter. The meter was calibrated in Stoddard solvent on the NIST “Cox¹” hydrocarbon liquid flow standard (a retired gravimetric flow standard with uncertainty of 0.12 %, $k = 2$) and in a 5 % PG + W mixture on the 0.1 L/s LFS. To compare results, a curve was fit to the Stoddard solvent calibration data and the difference between the best fit and the calibration results for the PG + W calibration points were calculated. The PG + W and Stoddard solvent calibrations agree within 0.33 %. This is consistent with our expectation (0.4 %) based on uncertainty of the kinematic viscosity of the calibration liquid and long term reproducibility of the flow meter and that of the Cox standard.

Figures 5B and 5C show the calibration history spanning 6 years for a 0.6 cm-diameter, dual-rotor turbine meter. The meter was calibrated using Stoddard solvent on

³ The data presented in Figure 5 uses the dimensionless numbers Roshko, $Ro = fD^2/\nu$, and Strouhal, $St = f\pi D^3/(4\dot{V})$ for the x and y axis respectively where f is the rotor frequency for the MUT, D is the diameter of the MUT, \dot{V} is the actual volumetric flow at the MUT, and ν is the kinematic viscosity of the calibration fluid.

the Cox standard and the 0.1 L/s LFS and also in a 5 % PG + W mixture on the 0.1 L/s LFS. For the upstream rotor, the PG + W calibration results were compared to a best-fit curve to the Stoddard solvent calibration data, as we did with the turbine meter in Figure 5A. The PG + W and Stoddard solvent calibrations agree within 0.09 %, well within the reproducibility of multiple calibrations (0.48 %). The downstream rotor of this meter has poor reproducibility of 1.7 %. The PG + W calibration data is well within this scatter.

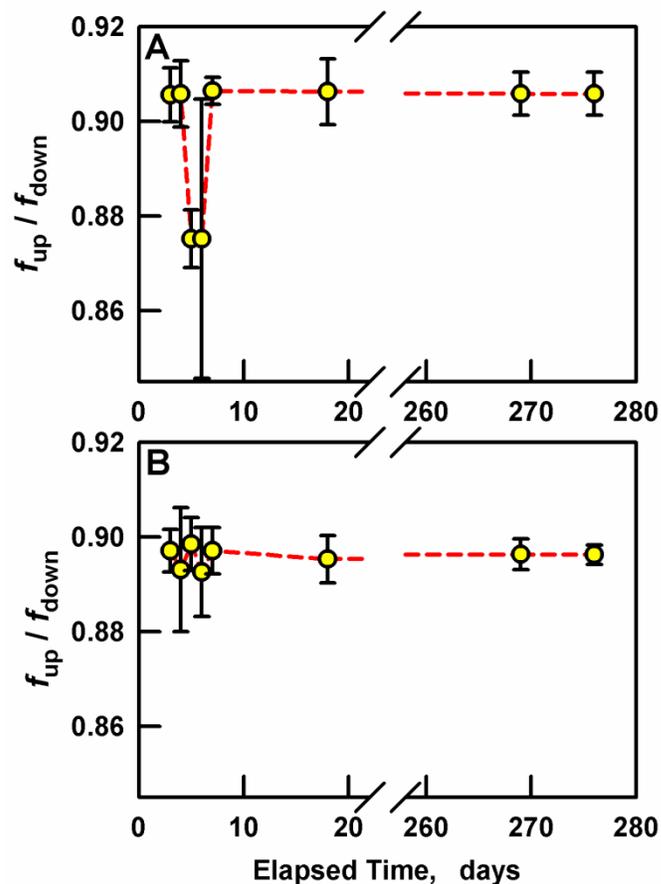


Figure 4. The ratio of the upstream rotor frequency to the downstream rotor frequency during 8 calibrations spanning almost a year. A) Turbine meter that was rinsed and dried in between each calibration. B) Turbine meter that was left continuously in reverse osmosis water. The error bars shown are the standard deviation of 7 measurements.

Positive Displacement Flowmeters

Positive displacement (PD) flow meters come in a variety of mechanical arrangements. PD meters measure the volume rate of liquid flow by repeatedly filling a container [9]. The total volume of liquid flowing through the meter in a given time is the product of the volume of the container and the number of fillings. The number of fillings is registered by a counter in a similar fashion to turbine meters. The PD meter that NIST uses as one of the LFS check standards is a piston type PD meter. Four pistons and cylinders are arranged in a

radial fashion around a central crankshaft. The nominal diameter of the inlet and outlet plumbing is 1.25 cm.

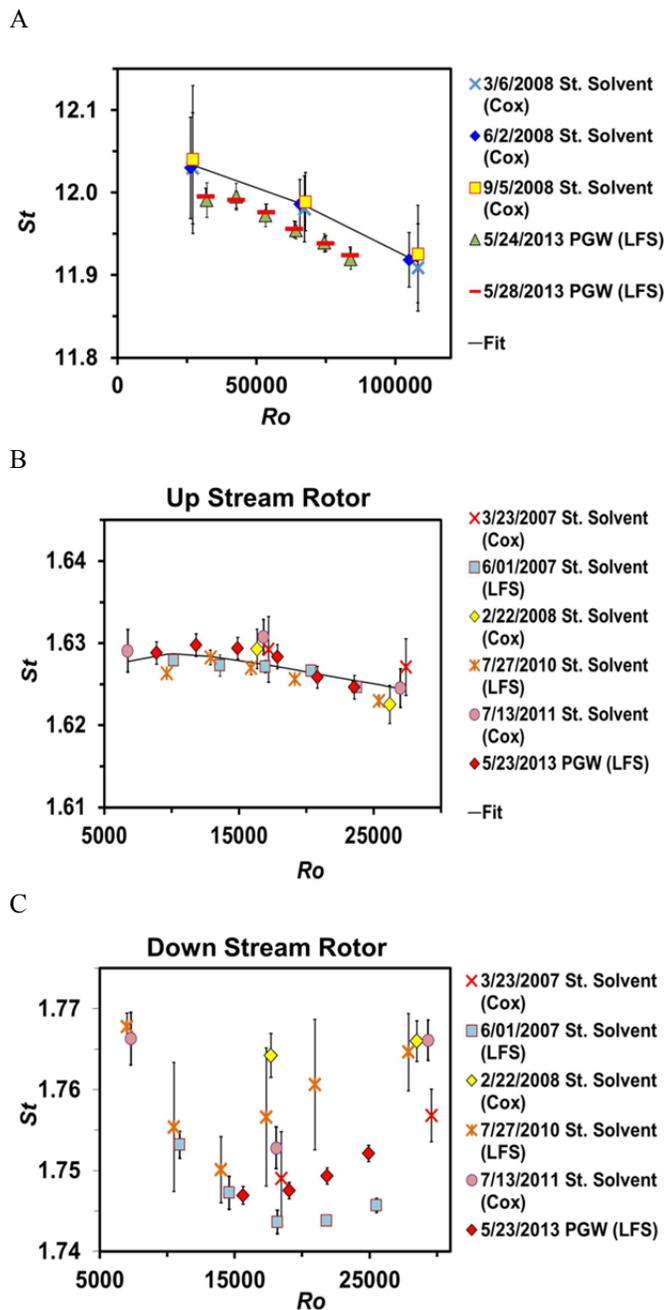


Figure 5. Validation of the calibration of a A) 1.25 cm-diameter single rotor turbine meter and B & C) a 0.6 cm-diameter dual rotor turbine meter in Stoddard solvent and 5 % PG + W mixture. The data for these turbine meters was taken from two of NIST’s primary LFSs. “Cox” refers to a retired gravimetric hydrocarbon liquid flow standard and the name “LFS” refers to the 0.1 L/s LFS. The error bars shown are the root-sum-square (RSS) of the LFS uncertainty and the standard deviation of the turbine meter measurement at a confidence level of 95 %.

According to the manufacturer, NIST’s PD meter will not rust in water. However, pure water causes galling in these meters because of insufficient lubrication between the pistons and the

cylinders’ walls. Furthermore, water breaks down the nitride hardening⁴ on the pistons. The 5% PG + W mixture provides enough lubrication to prevent galling, even if the meter is continuously exposed to it. We tested the effect of leaving a 5 % PG + W mixture in NIST’s PD meter on its calibration. The meter was calibrated in the 0.1 L/s LFS using a PG + W mixture. Then, it was left installed in the LFS for 5 days and then calibrated again. Figure 6 shows that the calibration did not change. Based on this finding, PD meters from this manufacturer can be calibrated in 5 % PG + W mixture without degradation of the meter.

PD meters are similar to turbine meters insofar as their calibration is independent of the calibration liquid’s density and viscosity if used in the low-uncertainty, usable range (50:1 for NIST’s PD meter). For the same reasons that are applicable to turbine meters, below this range, density and viscosity effect the calibration regardless of the fluid used. Therefore, the switch to 5 % PG + W from Stoddard solvent will not change the calibration results. To validate this, NIST’s PD meter was calibrated in Stoddard solvent and 5 % PG + W mixture. Figure 6 shows the calibration history spanning over 5 years. The meter was calibrated in Stoddard solvent on the Cox and the 0.1 L/s LFS and in a 5 % PG + W mixture on the 0.1 L/s LFS. The PG + W calibration results were compared to a best fit line to the Stoddard solvent calibration data. The PG + W and Stoddard solvent calibrations agree within 0.23 %, within the reproducibility of multiple calibrations (0.24 %).

Coriolis Flowmeters

Coriolis flowmeters are inertial flowmeters. They consist of one or two flow tubes in various geometries. The flow tube(s) are excited at their natural resonant frequency with a closed-loop, controlled-amplitude drive signal. Under conditions of zero flow, there is no phase difference between the two pickoffs. With flow present, the tubes twist apart due to the Coriolis force acting on the fluid mass. This adds to, or subtracts from, the tubes’ resonant motion, giving a relative phase displacement of the sine waves generated by the pickoffs that is directly proportional to the mass flow rate [10].

Coriolis meters are made of stainless steel or other nickel alloys [11]. Therefore, they are not very susceptible to rust. Coriolis meters measure the mass flow rate and have negligible sensitivity to changes in fluid properties due to temperature, density, absolute viscosity and composition.

To verify that calibration of a Coriolis meter in 5 % PG + W agrees with the calibration in Stoddard solvent, we calibrated a 0.6 cm-diameter coriolis meter in both fluids. Figure 7 shows the calibration history spanning over 5 years. The meter was calibrated in Stoddard solvent on the Cox standard and in a 5 % PG + W mixture using the 2.5 L/s LFS and the 0.1 L/s LFS. The PG + W calibration results were compared to a best fit line to the Stoddard solvent calibration

⁴ Nitride hardening (*a.k.a.* nitriding) is a process that reduces wear of steel parts that rub against each other as in the case of a piston moving in a cylinder with tight tolerances.

data. The PG + W and Stoddard solvent calibrations agree within 0.097 %, within the repeatability of the Stoddard solvent calibration (0.13 %).

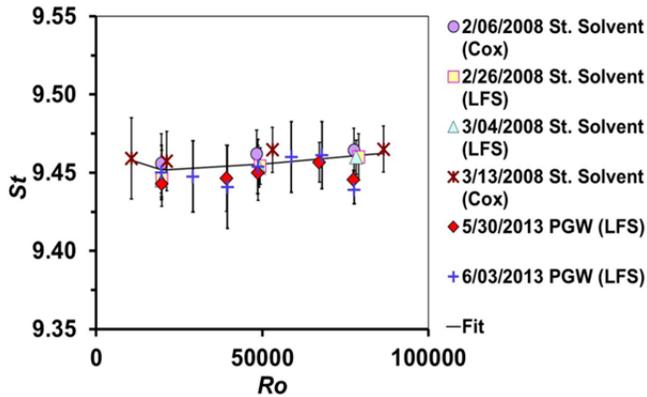


Figure 6. Validation of the calibration of a 1.25 cm PD meter in Stoddard solvent and 5 % PG + W mixture. The error bars shown are the RSS of the LFS uncertainty and the standard deviation of the PD meter measurement at a confidence level of approximately 95 %.

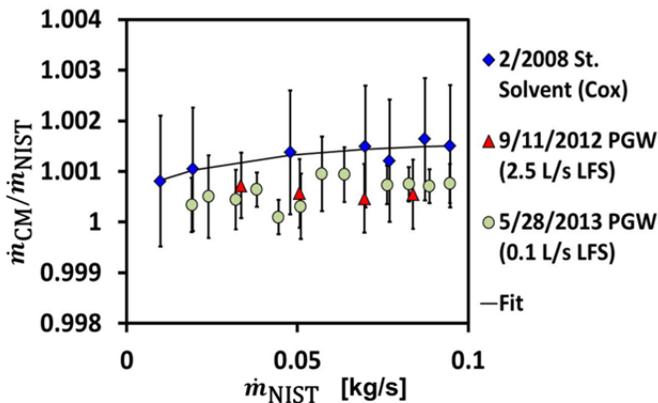


Figure 7. Comparison of the calibration of a 0.6 cm-diameter Coriolis meter in Stoddard solvent and 5 % PG + W mixture. The error bars are the expanded uncertainty ($k = 2$ corresponding to a 95 % confidence level) that accounts for uncertainties from: (1) the LFS, (2) the measured density and, (3) the reproducibility of the coriolis meter.

Cleaning of Meters Following Calibration

Our testing shows that 440C turbine meter bearings are unaffected by calibration in aqueous solution and that NIST's PD meter does not gall in a 5 % PG + W mixture. However, NIST cleans customer's meters after calibration. This reduces the chances of rusting and galling and also prevents contamination of the liquid that customers will meter.

The cleaning procedure following water exposure will depend on the type of meter. Most meters (*i.e.* turbine and coriolis) can be simply rinsed with alcohol using a hand held squirt bottle, or capped, filled with alcohol, inverted several times to fill and mix trapped volumes, and drained. PD meters may have crevices that will retain water if not rinsed more aggressively. NIST places them in a recirculating flow of ethanol at approximately 15 % of the

maximum flow for at least five minutes. We recommend that the meter should be with its RF or magnetic pickoff located in the downward position to assure all water is removed from the relatively small cavity there.

There are many acceptable ways to dry flow meters: 1) application of vacuum, 2) application of a stream of dry gas and 3) hanging the meter and waiting for draining and evaporation. For turbine and PD meters, one end of the meter is capped while it is evacuated from the other end for at least one hour. This avoids the risk of over-spinning the turbine or PD meter from blowing a strong gas stream through it. Over-spinning is not a concern for coriolis meters; therefore, they are quickly dried using a stream of dry nitrogen. In the event that no vacuum or inert gas is available, meters are hung to dry at various orientations for a minimum of three hours at each orientation to be sure all void volumes in the meter have drained dry. If the meter is to be stored, a lubricant can be applied to moving parts.

Summary and Future Directions

NIST has successfully changed calibration fluids in its piston LFSs. The LFS have been modified to be water compatible. We have investigated the compatibility of turbine, PD and coriolis meters with water and found no problems when reasonable precautions were taken. Furthermore, we have shown that the calibration of these meters in the 5 % PG + W mixture is equivalent to their calibration with Stoddard solvent.

Turbine meter bearings made of 440C are not affected by the PG + W mixture even when exposed to it continuously for 9 months. NIST's PD meter did not experience galling or rust formation when left exposed to the PG + W mixture for 5 days. The coriolis meter was unaffected by the PG + W mixture.

To reflect the change away from a hydrocarbon liquid calibration service, NIST has combined the liquid flow calibration services of water and the PG + W mixture into a single liquid flow calibration service. During this process, NIST decommissioned the Cox hydrocarbon liquid flow standard (a gravimetric flow standard with uncertainty of 0.12 %, $k = 2$).

NIST has developed a new dynamic gravimetric standard using the principles described in [12]. NIST is validating the dynamic standard by making extensive comparisons with a traditional 65 L/s LFS [13] and with the 2.5 L/s LFS. The dynamic standard spans the flow range 0.015 L/s to 15 L/s and will replace the upper flow range NIST has lost by decommissioning the Cox standard, overlap the 2.5 L/s LFS (0.020 L/s to 2.5 L/s) and overlap into the lower flow range of the 0.1 L/s LFS (0.003 L/s to 0.1 L/s). The 15 L/s LFS will utilize 5 % PG + W as its working fluid. This will allow for proficiency testing across all of NIST's LFS.

The switch to PG + W in NIST's LFS marks the beginning of a pollution free calibration service. The comparisons presented here and in other studies [3,4,8] show that there is no difference between the calibration results

obtained using Stoddard solvent and those obtained using a 5 % PG + W mixture.

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