

Pycnometer Installation, Operation and Calibration

To be presented at the 2013 International School of Hydrocarbon Measurement (ISHM), Oklahoma City, Oklahoma USA, May 2013.

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This paper will discuss pycnometers used to calibrate densitometers. In using a pycnometer, you must have the correct scales, pressure gauges and thermometers. You will learn the steps necessary to install, operate and calibrate the instrument.

Safety Considerations

MSDS sheets are available for review prior to testing any product. Verification of the equipment used for densitometer proving and the associated pressure relief devices rated for the system pressures is required. Make sure the proper PPE is worn. Appropriate testing of the atmosphere surrounding the densitometer proving site must be performed for possible hazards. The pycnometer should be filled, weighed and emptied in as short a time as possible. Natural Gas Liquids (NGL) that are contained in a 100% liquid full container can produce large pressure increases with a small increase in temperature.

Laboratory Certification

The newly constructed pycnometer shall be laboratory certified prior to use. The certification shall provide the following information:

- Certified air-filtered weight (W_a)
- Certified evacuated weight (W_o)
- Pycnometer base volume (PBV)
- Certified Coefficient of Expansion due to internal pressure of the pycnometer (E_p)
- Certified Coefficient of Expansion due to temperature of the pycnometer (E_t)

These values are to be used in the calculation associated with using pycnometer to calibrate the densitometer.

Field Verification of Pycnometer Certification

The purpose of the field verification is to ensure that there has been no shift in the certified volume of the pycnometer (PBV) or of the evacuated weight (W_o). The field test should be performed at any time there is concern about the continued accuracy of the certified values. If the field test does not agree with the certified values within .02%, the pycnometer must be recertified.

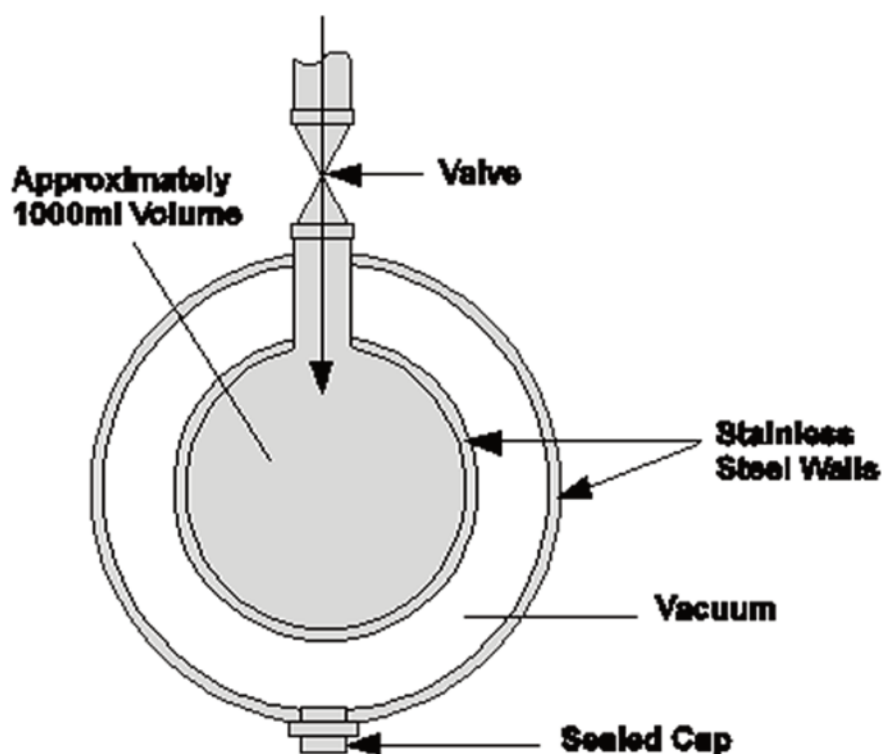


Figure 1: Double-Wall Sphere Pycnometer

The following steps should be followed:

- Thoroughly clean the pycnometer inside and out.
- Draw a vacuum on the pycnometer and weigh it.
- Open a valve to admit air into the pycnometer and weigh it again.
- Two consecutive field tests should agree within 0.02%.
- The field tests should agree with the certified values within 0.02%.

Note that a significant difference in elevation between the certifying laboratory and the field location will cause differences in the evacuated weight. To determine conformity with certified values, adjust the observed evacuated weight for variation in altitude.

Calibrating Pycnometer

Before a pycnometer can be installed there are calibrations that need to be completed. The accuracy of the proving is important in obtaining a successful pycnometer test. A key piece of equipment is the weigh scale. There are five pieces of equipment needed to perform an accurate pycnometer calibration of a density meter

- Pycnometer
- Weigh scale
- Certified test weights
- Certified Thermometer
- Certified Pressure Gauge

The weigh scale should be calibrated at the time of proving with certified apparent mass standards (test weights) to assure accuracy. Scales should be mounted on a stable and level surface. Air shields around the scale can stop the effects of current on the scale. The following shall be performed to verify pycnometers evacuated weight:



- Place evacuated pycnometer on the scale and record the scales readings.
- Verify that it does not differ more than 0.02% from the certificate.
- Once it is determined that the pycnometer is at the certified weight the pycnometer can be installed.

Installing Pycnometer

When installing the pycnometer there are a series of steps that must accurately be followed. The pycnometer may be installed in a series or parallel with the density meter. You should always allow product to enter the upstream pycnometer valve first. Gases or vapors must be vented/flared from the pycnometer and piping through the vent valve. Now that the pycnometer is fluid filled, close bypass valve to initiate flow through the pycnometer. Next, insulate the pycnometer to speed temperature stabilisation and prevent water condensation. The use of double-walled vacuum sphere pycnometer may not require this step.

Operations

After flow is initialised through the pycnometer, the following steps must be performed to verify density, deviation and stability:

- Observe the output of the density meter for a period to verify steady conditions.
- When temperature or pressure differentiate between density meter and pycnometer are within criteria given in API MPMS Chapter 14.6.7 (generally 0.2°F and 2 lbs.), record the following field data:
 - 1.) Observed density (density meter output)
 - 2.) Density meter temperature
 - 3.) Pycnometer temperature
 - 4.) Density meter pressure
 - 5.) Pycnometer pressure
- Partially open pycnometer bypass valve, immediately close the inlet and outlet valves of pycnometer. Close outlet valve first.
- Remove pycnometer from sample system, check of leakage. Any leakage from pycnometer shall void test.
- If pycnometer was covered with insulation blanket, remove.
- Make sure both valve opening and outside pycnometer are washed. Blow dry with nitrogen or air. Repeat as necessary. This is done to make sure all moisture that may have accumulated is free from instrument.
- Repeat the above steps until you have two (2) successful consecutive provings.
- At this time calculate an average Density Meter Factor (DMF).
- For applications in which the density meter's output is adjusted, perform an additional proving run to confirm that the adjusted density meter's output does not differ from the new test's result by more than 0.05%.

Pycnometer Recertification

The pycnometer should be recertified any time that the validity of the original certification is in question. The pycnometer shall be recertified:

- Every two (2) years
- If the pycnometer has been damaged
- If the pycnometer has been disassembled
- If the welded-on valve parts have been replaced
- If the rupture disk is replaced
- If the valve is repaired. (This is not necessary if the valve manufacturer can substantiate that there would be no change in the pycnometer volume in excess of 0.02% from valve repair.)

Master Density Meter

American Petroleum Institute (API) Chapter 9.4 working group is testing the use of a master meter densitometer to verify and calibrate on-line densitometers. This method hopes to eliminate

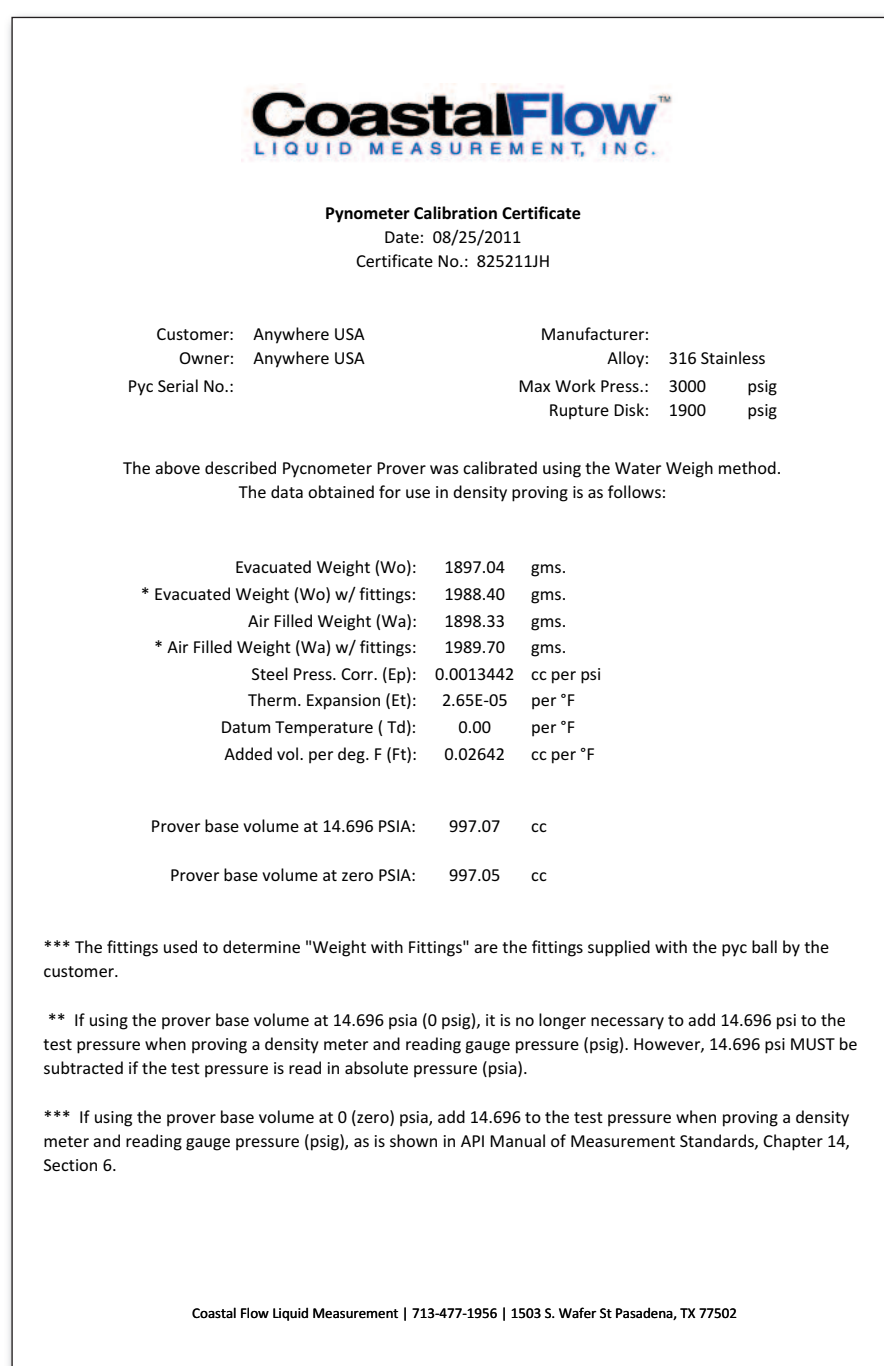


Figure 2: Sample Pycnometer Calibration Certification

inaccuracies associated with the methods of handling and calibration of densitometers using pycnometers. API realises the challenges of calibrating an on-line densitometer using a pycnometer. Studies are ongoing with respect to the calibration performed by the manufacture and the possibility of requiring a densitometer certification similar to the requirements of certifying a meter prover.

A master meter densitometer proving method coupled with densitometer certification requirements are expected to provide an alternative to the pycnometer method. Reducing the complexity and time required of the proving at a level of performance that will greatly reduce the uncertainty.

The International School of Hydrocarbon Measurement (ISHM), founded in 1924, will mark its 88th annual event in affiliation with the University of Oklahoma with the 2013 School & Exhibition, May 14-16, at the Cox Communications Center in Oklahoma City.

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