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PYCNOMETER INSTALLATION, OPERATION AND CALIBRATION
Class # 2340

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Abstract

This paper describes pycnometers that are used to calibrate density meters (densitometers). Within this paper, the fluid stream is to be considered a Natural Gas Liquid, Refined Product or Supercritical Fluid. Important points for consideration are:

- Pycnometer Selection
- Pycnometer Certification, Field Validation and Recertification
- Proper Pycnometer Installation
- Densitometer Calibration

Safety Considerations

Pipeline systems frequently operate at high pressures. Natural Gas Liquids (NGL) and Refined Products are flammable and may be a health hazard.

- Review MSDS sheets for the product to be tested.
- Verify that the equipment used for densitometer proving and the associated pressure relief devices are rated for the system pressures.
- Wear proper PPE
- Perform appropriate test of the atmosphere surrounding the densitometer proving site for possible hazards.
- Natural Gas Liquids (NGL) that are contained in a 100% liquid full container can produce large pressure increases with a small increase in temperature. The pycnometer should be filled, weighed and emptied in as short a time period as possible.

Mass Measurement

During the 1970s the wider use of chemical feed stocks and fractionation of Natural Gas Liquids (NGL) created a need for more precise measurement. For these applications, volumetric measurement systems were replaced by mass measurement systems. In the late 1970s and early 1980s the mass measurement technique was expanded to include supercritical fluids such as Carbon Dioxide and Ethylene.

Many mass measurement systems measure the flowing fluid volume and the flowing fluid density. Mass is calculated by multiplying volume by density:

$$\text{Mass} = \text{Volume} \times \text{Density}$$
$$\text{Pounds} = \text{Gallons} \times \text{Pounds/ Gallon}$$

To ensure that the density component of this calculation is accurate, it is necessary to calibrate the density meter (densitometer). The densitometer is calibrated by comparing the displayed density to the actual density as determined by a pycnometer.

Pycnometer

A pycnometer is a certified volume that is capable of containing a representative sample of the test fluid. The mass of the fluid in the filled pycnometer is divided by the certified volume of the pycnometer to determine the density of the test fluid.

The certified volume can be as simple as a glass beaker. But Natural Gas Liquid (NGL) and Refined Liquid Products require that the pycnometer be of the flow-through type.

Flow-Through Pycnometer Types

There are 3 flow-through types of pycnometers:

- Single Cylinder

  The single-cylinder pycnometer consists most commonly of a stainless steel cylinder with a volume of approximately 500 cubic centimeters with a valve at each end and fitted with a rupture disk. The cylinder is oriented vertically and flow through the cylinder is from bottom to top.

- Single Sphere

  The single-sphere pycnometer consists most commonly of a stainless steel sphere with a volume of approximately 1,000 cubic centimeters. It has an inlet valve that admits the product into the sphere at about the vertical mid-point. A combination outlet valve and rupture disk are positioned at the top of the sphere. A siphon tube attached to the outlet to ensure that there is good mixing as the fluid flows through the sphere. A weep hole at the top of the siphon tube ensures that gasses are not trapped in the upper portions of the sphere.

- Double-Wall Vacuum Sphere

  The double-wall vacuum sphere is constructed exactly as the single sphere except that a second sphere surrounds the inner sphere. A vacuum is drawn between the two spheres to provide an insulating barrier between the two spheres. This insulating barrier helps to stabilize the temperature of the fluid in the pycnometer and reduces the formation of condensation on the outer wall.

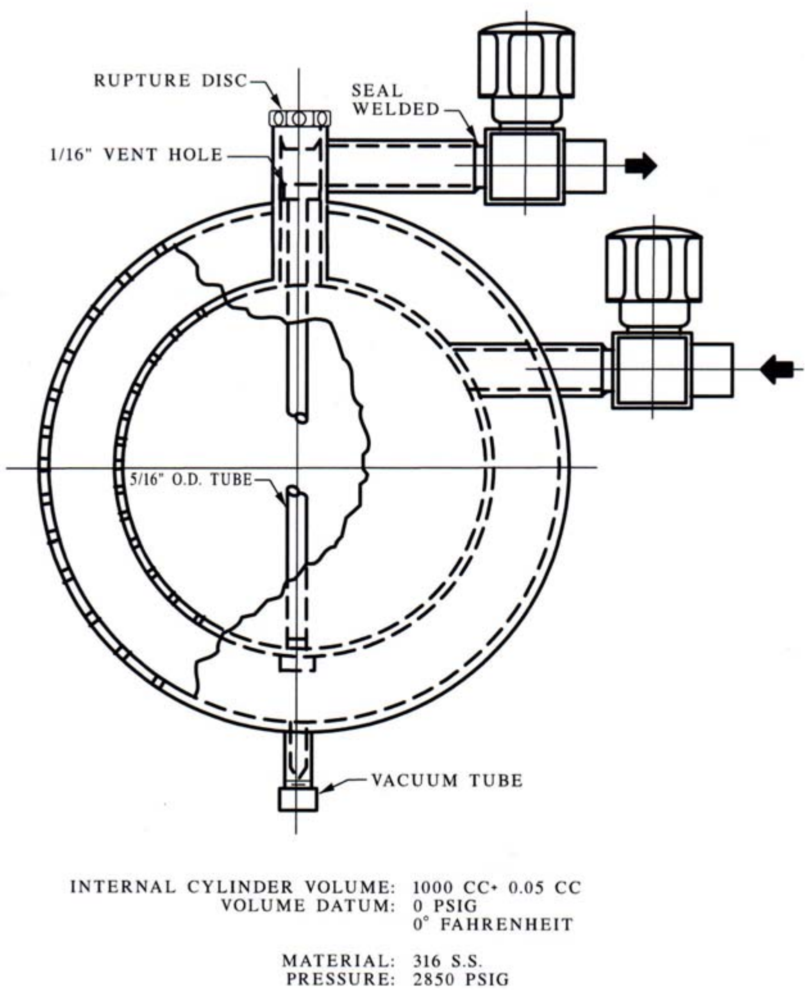

Figure 1. Double-Wall Sphere Pycnometer

Pycnometer Design Requirements

The flow-through pycnometer must have the following characteristics:

- Able to safely contain the product during the fill, transport and weighing process
- Be of sufficient volume to ensure that the weighing errors are insignificant.
  - Not less than 500 cubic centimeters
  - 1,000 cubic centimeters is most common.
- Be light enough and of small enough volume that it can be weighed on a conventional balance - Filled mass (weight) should not exceed 5,000 grams.
- Easy-to-clean, safe surface design and finish
- Internal flow pattern shall be designed to facilitate adequate purging and to prevent entrapment of heavy impurities or of gasses.
- Precision shutoff valves shall be fitted at each the flow inlet and outlet of the pycnometer. The valves bodies shall be in integral part of the pycnometer.
- A full-flow rupture disk shall be installed.
- A serial number shall be permanently affixed to the vessel.

Pycnometer Installation

The pycnometer installation design shall be in accordance with American Petroleum Institute (API) Manual of Petroleum Measurement (MPMS) Standard 14 - Natural Gas Fluids Measurement Section 6.10 (API MPMS 14.6.10), Density Sampling Systems.

In general, the pycnometer and densitometer should contain a representative sample of the fluid stream that has not significantly been altered in composition, temperature or pressure from the flowing pipeline conditions. Points of consideration include:

- During the densitometer calibration, the density difference between the flow meter, densitometer and pycnometer shall not deviate more than 0.05%.
- Slip-stream flow shall begin from a sample probe.
- The system shall ensure that the fluid is homogenous within the flow meter, densitometer and pycnometer. The design shall not permit entrapment of bubbles or particulate.
- Flow rate through the densitometer and pycnometer shall be sufficient to minimize lags in density variation between the flow meter, densitometer and pycnometer. A flow meter in the pycnometer piping can be helpful to verify sufficient flow.
- The system shall not cause cavitations or flashing of the fluid.
- The system shall minimize fluid pulsation and pressure surges.
- The system shall be thermally bonded to the flow meter piping
- The system shall include pressure and temperature instrumentation to ensure that conditions within the pycnometer, densitometer and flow meter are nearly identical.
- The system shall not hinder the ability of the flow meter to properly function. It shall not alter the velocity profile of the flow meter or bypass the flow meter.
- The overall design of the system shall facilitate convenient and safe connection of the pycnometer, filling of the pycnometer, blow down of the associated piping and removal of the pycnometer for weighing.

Laboratory Certification

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

- Certified air-filled 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 (Et)

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

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 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 a valve is repaired. (This is not necessary if the valve manufacturer can substantiate that there would be no change in pycnometer volume in excess of 0.02% from valve repair.)

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 0.02%, the pycnometer must be recertified.

Simply stated, the steps are as follows:

- 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.

More detailed steps for field certification are listed in API MPMS 14.6 part15.

Calibrating a Densitometer

The basic procedure for calibrating a densitometer is to fill a pycnometer with a fluid that is identical in composition, temperature and pressure with the fluid that is flowing through the densitometer. Valves on the pycnometer are closed and the densitometer reading is noted at the same time. The weight of the previously evacuated pycnometer (W_o) is subtracted from the weight of the filled pycnometer (W_f) to determine the weight of the fluid. The volume of the pycnometer is calculated using the laboratory certified values for pycnometer base volume (PBV), the correction of the base volume for the effect of the temperature and the correction of the base volume for the effect of pressure. The weight of the fluid in the pycnometer is divided by the volume of the pycnometer to determine the density of the fluid. The calculated density of the fluid in the pycnometer is compared to the densitometer reading to determine a densitometer correction factor.

The following steps are taken to perform the Densitometer calibration:

1. Verify the calibration of all test equipment
2. Verify that the pycnometer is clean by comparing the evacuated weight (Wo) to the pycnometer certification
3. Install the pycnometer and initiate flow through the pycnometer.
4. Verify that the temperature and pressure of the pycnometer and of the densitometer are equal to the temperature and pressure in the pipeline.
5. Remove the filled pycnometer, noting the densitometer reading.
6. Measure the pycnometer liquid filled weight.
7. Calculate a densitometer calibration factor
8. Repeat steps 3 through 7 to achieve two consecutive tests that agree within 0.02%
9. Evacuate the pycnometer and verify that the weight returns to Wo.

Average the two densitometer calibration factors to determine the new densitometer proving factor.

The densitometer calibration procedure is described in greater detail in API MPMS 14.6.13

References

API MPMS Chapter 14.6 “Natural Gas Fluids Measurement”, Continuous Density Measurement.