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PYCNOMETER FOR VOLATILE LIQUIDS

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Fig. 2.

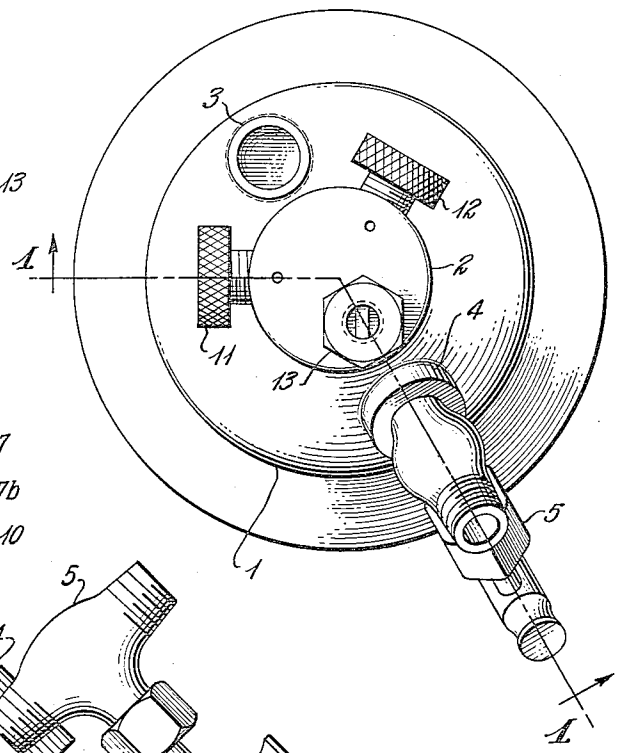
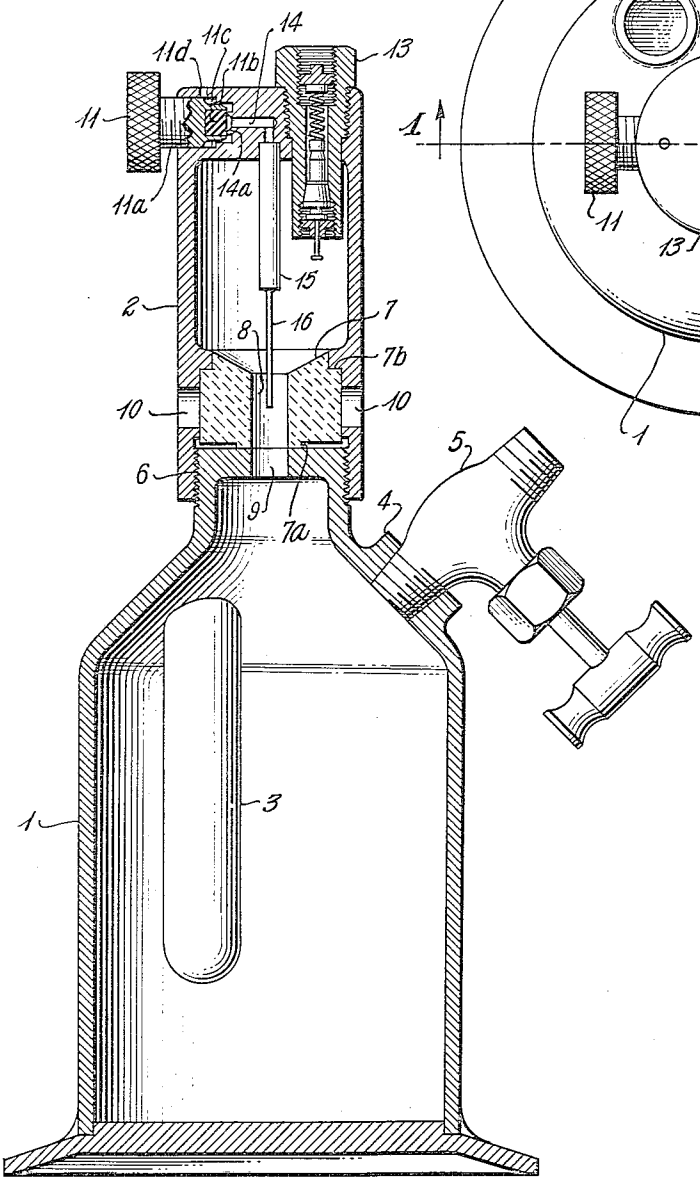


Fig. 1.



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PYCNOMETER FOR VOLATILE LIQUIDS

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9 Claims. (Cl. 73—32)

This invention relates to an apparatus for measuring a given volume of volatile liquid without changing the character or composition of the liquid during the volume determination process. In one of its aspects it relates to a pycnometer for determining the density of liquid petroleum gas (LPG).

In determining the density of volatile liquid mixtures great care must be taken to prevent loss of material due to vaporization since such losses generally result in a change of composition due to the great vaporization of the low boiling material in the mixture. There have been numerous methods devised to minimize or overcome this aforesaid loss of the lower boiling materials. These methods involve immersing the pycnometer in the liquid, by drawing the liquid in through a capillary tube, or by measuring the density by means of hydrometers while the liquid is kept under pressure. These methods involve the use of bulky or fragile equipment and generally give unsatisfactory results unless extreme care is taken to maintain the equipment within very narrow limits of pressures or temperatures or both.

One of the objects of this invention is to provide an improved pycnometer capable of handling liquids under pressure without changing the character or composition of the liquid during the volume determination process. The pycnometer is of particular value in determining the density or specific gravity of liquid petroleum gas (LPG). The pycnometer of our invention is sturdy, easily fillable, protects against changes of composition of the liquid during the volume measuring period, and is an extremely accurate apparatus and provides a relatively rapid means for the determination of volatile liquid densities. Other objects will be obvious to those skilled in the art upon reading the accompanying specification, claims, and drawings.

We will describe our invention by referring to attached drawings in which our invention is shown in one of its preferred embodiments.

Figure 1 is a vertical cross-sectional view of our pycnometer taken along the lines 1—1 of Figure 2.

Figure 2 is a top view of our pycnometer.

Referring now to the drawing, the pycnometer comprises two sections, a body or container 1 and a cylindrical neck 2. The body is narrowed in its upper portion and said narrowed portion is threaded to accommodate the cylindrical neck. Within the body and extending therein in a downward direction from the shoulder of said body is a thermowell 3. Also in the shoulder of the body is a connection 4 to which is connected a valve 5. The body and neck sections are both hollow and comprise the body chamber and the head chamber respectively.

The neck 2 is attached to the body 1 by means of a threaded connection 6 and is so constructed that in tightening this threaded joint a compression seal is formed at the top and bottom surface of a transparent plastic bushing 7 thus obviating the use of gaskets. Specifically, a

sealing action is thereby effected at a region 7*a* where a downwardly protruding portion of bushing 7 engages the top of container 1 and at a region 7*b* where a shoulder formed in the bushing 7 is engaged by complementary surfaces of the neck section 2. A hole 8 is drilled through said plastic bushing in such a manner as to coincide with a hole 9 in the body 1, said holes effecting communication between the interior of the body 1 and the interior of the neck 2. Four sight holes 10 are drilled in the lower portion of the neck. The holes are center spaced 90° apart around the neck and are so located that they expose the outer surface of the transparent bushing.

Within the head or neck section are manually controlled valves 11 and 12 and a spring loaded relief valve 13, said relief valve operating when the pressure exceeds a predetermined safe limit. Valve 11 is comprised of a threaded section 11*a* so that it can be quickly closed, said threaded section having a protruding annular section 11*b*, a resilient member 11*c* encompassed within the protruding section, and a relief conduit 11*d*. Valve 11 is so constructed and so located that when it is closed, the resilient member 11*c* closes upon a vertically disposed annular flared section 14*a* of a fine bore tube 14, the said flared portion of the bore tube being smaller in diameter than in said resilient member of valve 11. Extending downward from the internal terminal of said bore tube 14 is a draw off tube 15. The draw off tube 15 terminates in a needle or capillary tube 16, the said needle 16 extending downward into the bore of the bushing 7 to a position level with the centers of sight holes 10. The valve 11, fine bore tube 14, and the draw off tube 15 serves as a dip tube and provides for a means for leveling the liquid in the pycnometer at the bottom of the needle of said draw off tube. Valve 12 connecting the upper chamber with the atmosphere is provided to permit purging of the apparatus. Valve 13 is provided to relieve excess pressure say when the pycnometer is being raised to room temperature and operator fails to bleed the excess LPG off.

We prefer to construct our pycnometer of aluminum using brass valves and fittings in order to obtain a light weight and serviceable apparatus. We use a body chamber of 225+5 ml. and a neck chamber of approximately 20 ml. We prefer to weld the bottom on our body and to weld all seams. It will be obvious to those skilled in the art that many modifications of our pycnometer can be made without digressing from the scope of our invention. The material of construction of the pycnometer will be dependent upon the nature of the material to be tested therein. The valve 5 could be welded into the body. The bottom could be threaded. The body may be cast or formed. This size may be of any convenient dimensions. The number of sight holes can vary.

Operation of pycnometer

We will describe the operation of our pycnometer in connection with the determination of density of LPG. It should be understood that the use of the pycnometer is not limited to this material. Certain modifications can be made without departing from the scope of our invention.

Assuming that the pycnometer initially contains air, a small amount of LPG is admitted under pressure through valve 5 and the valve is then closed and disconnected from the LPG supply. The two manually controlled valves 11 and 12 are opened and the vapor is released thereby purging the pycnometer of air. The valves 11 and 12 are then closed, the pycnometer inverted and all liquid LPG is purged through the sampling valve 5.

After the pycnometer has been flushed free of air, the

LPG supply is connected to valve 5, the pycnometer is immersed in a Dry Ice bath, and valve 5 is opened. The Dry Ice will keep the LPG in a liquid condition and will condense the hydrocarbon (LPG) gas in the pycnometer thereby creating a vacuum. The combined effect of the pressure on the LPG supply and the vacuum developed in the pycnometer due to condensing the hydrocarbon will cause the pycnometer to fill to a level above the lower end of the needle 16. The valve 5 is closed and the LPG supply can be disconnected. The pycnometer is removed from the Dry Ice bath and a thermometer placed in the thermowell 3. The valve 11 is opened slightly and the level of the LPG is adjusted to the bottom of needle 16. Excess liquid must be released in small increments to avoid setting up temperature differences which promote bubble formation in the liquid in the body of the instrument. The pycnometer is allowed to stand until the temperature reaches a stable point somewhere around room temperature. While the temperature is increasing, it will be necessary to bleed liquid from the pycnometer at frequent intervals being careful to avoid bubbling. The holes 10 make it possible to observe the liquid level and to determine if there is bubbling.

After a stable condition of temperature is reached a final adjustment is made to bring the liquid level to the lower end of the needle 16, the pycnometer is wiped to remove any trace of moisture and is weighed. The density of the contents is calculated as follows:

EQ. I

$$\text{Density at test temperature} = \frac{\text{gross weight} - \text{empty weight}^2}{\text{volume}^1}$$

The density at a standard temperature can then be calculated if desired.

[1] The volume of the pycnometer can be determined by any desired method. We have determined the volume of a pycnometer by using a pure propane and a pure butane of standard density and proceeding according to the hereinbefore described procedure. The known density is substituted in the Equation I and the unknown volume determined. The summary of results taken on 17 determinations is shown below:

Determined volume of pycnometer, ml. max	226.4
Determined volume of pycnometer, ml. min	225.9
Average	226.2

[2] The empty weight determined when purged of gas and filled with air at room pressure.

The following precautions should be observed during the operation of the pycnometer:

During the final filling of the instrument, there must be no release of vapors from either of the two top valves else there might be a change in composition of the sample.

A periodic check of empty weight should be made. For example, this weight with one instrument tested varied from 361.88 grams to 362.35 grams over a two month period.

Should the pycnometer suffer a blow which would cause a change in volume, the volume should be redetermined.

EXAMPLE I

The following data are given to show the repeatability of results when using our pycnometer:

Material is pure butane

Test No.	Density as Determined at 68° F.	Deviation from Mean
1	0.5787	0.0003
2	0.5786	0.0002
3	0.5783	0.0001
4	0.5780	0.0004
5	0.5786	0.0002
6	0.5781	0.0003
7	0.5790	0.0006
8	0.5784	0.0000
9	0.5782	0.0002
average	0.5784	0.0003
NBS value [1]	0.5788	

[1] National Bureau of Standards: Liquid Densities of Eleven Hydrocarbons; Letter Circular LC-736.

EXAMPLE II

The following data are given to show how the pycnometer is used in determining the density on a blend.

Material is a blend [1] of propane and butane

Test No.	Determined Density at 68° F.	Deviation from Mean
1	0.5255	0.0002
2	0.5249	0.0004
3	0.5250	0.0003
4	0.5255	0.0002
5	0.5256	0.0003
6	0.5253	0.0000
average	0.5253	0.00023
Calculated Density	0.5247	
Average by hydrometer (3 determinations)	0.525	

[1] 69.15 wt. percent pure propane and 30.85 wt. percent pure butane.

We have described our invention in one of its preferred embodiments and have illustrated it with typical liquids to which it well adapted. The description and examples are for illustrative purposes only and are not to be considered as limiting in any sense. It will be obvious to those skilled in the art that many modifications can be made without departing from the scope of our invention as disclosed in the specification.

We claim:

1. In a pycnometer for measuring a predetermined volume of a volatile liquid, a head chamber, a body chamber, a flexible and transparent bushing disposed between said head chamber and said body chamber, means for attaching said head chamber to said body chamber, said means for attaching the head chamber and the body chamber being provided with openings for viewing the interior of said chambers through said transparent bushing, means for compressing said bushing thereby forming compression seals on the top and bottom surfaces of said bushing, said head chamber communicating through said bushing with said body chamber, means for withdrawing liquid material from said head chamber, means for purging vapor from said head chamber, and automatic means for releasing pressure from said head chamber when said pressure exceeds a predetermined value.

2. In a pycnometer for measuring a predetermined volume of a volatile liquid, a head section, a conduit within the walls of said head section connecting a draw off tube with the exterior of said head section, said conduit being in two sections, a valve operating between the two sections of said conduit, said draw off tube projecting downward from the internal terminus of the downstream section of said conduit to a predetermined position within the bore of the herinbelow transparent bushing, a body section, means for filling said body section, a transparent bushing disposed between said head section and said body section, means for attaching said head section to said body section, and means for compressing the transparent bushing when attaching said head section to said body section thereby creating compression seals at the top and bottom surfaces of said transparent bushing.

3. In the pycnometer of claim 2, the valve being comprised of a movable member, said movable member having a protruding cylindrical section encompassing a resilient member, the upstream end of the internal section of the valved conduit being flared, said flared portion being of smaller diameter than said resilient member, and said resilient member closing on said flared portion thereby blocking said conduit.

4. In a pycnometer for measuring a predetermined volume of a volatile liquid, a head chamber, a body chamber, a flexible and transparent bushing disposed between said head chamber, and said body chamber, means for attaching said head chamber to said body chamber, said means for attaching the head chamber and the body chamber being provided with openings for viewing the interior of said chambers through said transparent bush-

ing, means for compressing said bushing so as to form compression seals on the top and bottom surfaces of said bushing, said head chamber communicating through said bushing with said body chamber, means for withdrawing liquid material from said head chamber and means for purging vapor from said head chamber.

5. In a pycnometer for measuring a predetermined volume of a volatile liquid, a body chamber, a head chamber superposed on said body chamber and communicating therewith, a transparent bushing forming a seal between said head chamber and said body chamber, means in at least one of said chambers for viewing the interior thereof through said transparent bushing, a passage communicating from said head chamber at a position remote from said body chamber to the atmmosphere, a valve for controlling the opening of said passage, a draw-off tube operably connected to said passage and projecting to a position within said transparent bushing at a level of said means for viewing the interior of said chamber, and means for introducing a fluid to said body chamber.

6. The pycnometer of claim 5 wherein the means for filling said body chamber is a valved means and wherein said body chamber is equipped with a thermowell.

7. In a pycnometer comprising a head section and a body section, in said head section a manually controlled threaded screw valve comprising a resilient means terminating internally in a passageway, said passageway communicating with a draw-off tube, said draw-off tube projecting downwardly and terminating at the level of normal fill of said pycnometer, said body section disposed below and communicating with said head section, and a valved means for filling said body section.

8. In the pycnometer of claim 7, a transparent bushing between said head section and said body section at said level of normal fill, and means in one of said sections for observing the end of said draw-off tube terminating at level of normal fill of said pycnometer.

9. In a pycnometer for measuring a predetermined volume of a volatile liquid, a body chamber, a head chamber, a valved passageway communicating between said head chamber and the atmosphere, a draw-off tube communicating with and operably connected to the internal end of said passageway and projecting downward to a predetermined position within the hereinbelow transparent bushing, said head chamber having a projecting sleeve portion adapted to attach to said body chamber, a transparent bushing adapted to fit within said sleeve, means in said sleeve for observing the lower end of said draw-off tube through said transparent bushing, means for compressing said transparent bushing when attaching said head chamber to said body chamber so as to effect a compression seal at top and bottom of said transparent bushing, and means for filling said body chamber.

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