

Pressure-Volume-Temperature Property Determination for Fluids at Extreme Pressures

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Abstract

Pressure-volume-temperature (*PVT*) properties, more commonly known as density data play an important role in various chemical processes and provide a fundamental database for the development and examination of thermodynamic models. For example, ultradeep oil exploration process requires density data at extreme pressure conditions, which are crucial for the design and optimization of drilling and production equipment. This review discusses the current techniques for high-pressure determination. Three most common techniques, vibrating-body, bellows, and floating-piston, are described in detail with presentation of some typical results and advantages and drawbacks are discussed for each method. Other techniques are also briefly introduced at the end of this review.

Introduction

Pressure-volume-temperature (*PVT*) properties, also named as densities or volumetric properties, are indispensable for the design and optimization related to all kinds of chemical processes such as distillation and extraction in the production and purification of oils, polymers, pharmaceuticals, and other natural materials. In some processes, *PVT* data at extreme pressures and/or temperatures are required. For example, a recent report from National Energy Technology Laboratory (NETL), Department of Energy (DOE) [1] states the significance of high-pressure density data in the exploration of ultradeep oil reservoirs in sedimentary basins approximately 6100 m or more underground, at which the temperature and pressure can reach 260°C (500°F) and 240 MPa (35,000 psi), respectively. A part of the content in the NETL report is given below.

"Global increase in oil demand and depleting reserves has derived a need to find new oil resources. To find these untapped reservoirs, oil companies are exploring various remote and harsh locations such as deep waters in Gulf of Mexico, remote arctic regions, unexplored deep deserts, etc. Further, the depth of new oil/gas wells being drilled has increased considerably to

tap these new resources. With the increase in the well depth, the bottomhole temperature and pressure are also increasing to extreme values (i.e. up to 500°F and 35,000 psi).

The density and viscosity of natural gas and crude oil at reservoir conditions are critical fundamental properties required for accurate assessment of the amount of recoverable petroleum within a reservoir and the modeling of the flow of these fluids within the porous media. These properties are also used to design appropriate drilling and production equipment such as blow out preventers, risers, etc. With the present state of art, there is no accurate database for these fluid properties at extreme conditions..."

Further, *PVT* data are critical fundamental data for the determination of other properties. For example, *PVT* data are required for the determination and prediction of high-pressure viscosity [2,3], which is another critical property related to various chemical processes. In modeling studies, *PVT* data at extreme pressures provide a database for testing contemporary equations of state (EoS) [4-7] and improving or establishing a specific EoS suitable for use at high temperatures and pressures [8,9]. The

established EoS can then be used to predict important properties such as specific heat, enthalpy, and miscibility [10-12]. These data becomes increasingly demanded in oil industry. Presently oil companies are using EoS based on lower temperature and pressure database that exhibits an unsatisfactory predictive capability. For example, the percent deviation between actual values and predicted values can reach $\pm 50\%$ at extreme conditions from NETL's report [1].

This review intends to explore the experimental techniques for the *PVT* data (density data) determination at high pressures. Three techniques, including vibrating-body, bellows, and floating-piston method, are most commonly used and therefore will be described in detail. The working mechanisms along with the schematic diagram of typical instruments will be given and some representative results for each technique will be presented. Last, the challenges for these techniques will be put forward. In addition to these three techniques, several other methods reported in literature will be also briefly introduced for the high-pressure density determination.

Vibrating-Body Instrument

Vibrating-body instrument has been widely used for the high-pressure density determination due to its high accuracy [13-24]. It employs a vibrating body to simultaneously measure the high-pressure density and viscosity data of the fluid of interest. Detailed mechanism of this method involves a theoretical model with complicated physical equations on the mechanics of vibration and fluid mechanics, which can be found elsewhere [25]. Briefly, a body surrounded by the fluid of interest is set into a vibratory motion, which can be characterized by two motional parameters, the resonant period and the width of the resonance curve. The two parameters are both affected by the hydrodynamic drag exerted on the body by the fluid of interest and can be correlated to the fluid density and viscosity with two equations. Given that the two parameters can be directly

measured, the fluid density and viscosity data at different temperatures and pressures can be obtained, in principle, simultaneously solving the two correlation equations. However, these equations are typically complicated and each of the equations includes both density and viscosity along with the two motional parameters, making it difficult to solve the two equations. Therefore, for a traditional vibrating-body instrument, the density data are also obtained elsewhere, for example, from other literature data or from the calculation with an EoS or a correlation equation such as the Tait equation. Hence, only viscosity is unknown and can be solved with the correlation equations on the resonant period and the width of the resonance curve [26,27]. From this point of view, the vibrating-body instrument is usually called vibrating-body viscometer.

Krall *et al.* [28] used an oscillating-disk viscometer, one kind of vibrating-body instrument, in which a body is set into an oscillatory motion and then allowed to decay freely, to simultaneously measure the density and viscosity of toluene at temperatures to 150°C and the pressures to 30 MPa. An uncertainty of 0.3% was obtained for both density and viscosity at the temperatures below 125°C . When the temperature goes higher, the uncertainty increased to 1.5%. The loss of accuracy is probably due to a larger variation of the elastic properties of the torsion wire connected to the oscillating disk at higher temperatures. In their later paper [27], Krall *et al.* employed the same apparatus for the same system, pure toluene at the same temperature and pressure range. However, instead of simultaneously determining the density and viscosity data, Krall *et al.* calculated the density data with the modified Tait equation and only measured the viscosities at each temperature and pressure level. Interestingly, the uncertainty of 0.5% was obtained over the entire temperature range up to 150°C , indicating that it may still lead to a larger error even though the two aforementioned correlation equations with motional parameters can be successfully solved

to obtain densities and viscosities simultaneously.

To circumvent the difficulties in solving correlation equations, Wakeham's research group [13,14,21-24] designed a vibrating-wire instrument for the simultaneous determination of density and viscosity data. The working equations appear similar to other vibrating-body instruments. The vibrating-wire is connected to a sinker immersed in the fluid and detected the buoyancy force exerted on the sinker in terms of the wire tension, which is then related to its resonant period with a complete theoretical analysis. This approach brings about an equation dealing with the buoyancy of the sinker, which only depends on density, in addition to the aforementioned correlation that depends on both density and viscosity. Therefore, the working equations can be set to solve densities independently (or at least partly independently) and then viscosities, leading to an increase in the precision of the density determination using the vibrating-body instrument.

Figure 1 shows a schematic diagram of a typical vibrating-wire system [15]. A vibrating-wire is assembled in a high-pressure vessel with two permanent magnets mounted to generate magnetic field in the region of the wire. A suspended sinker is connected to one end of the wire and immersed into the fluid of interest in the vessel. The other end of the wire is placed outside the high-pressure vessel and connected to multiple electronic devices, which is used for signal generation, amplification, and processing, and for the determination of the amplitude and frequency of the wire vibration.

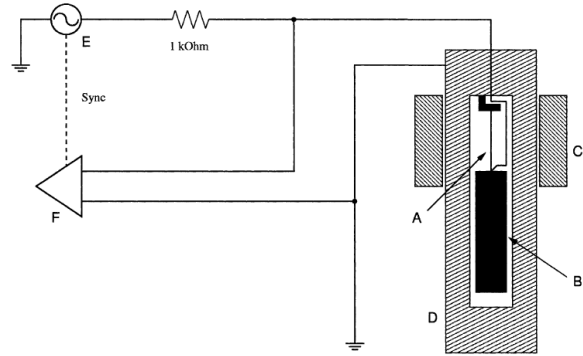


Figure 1. Schematic diagram of a vibrating-wire equipment: A, vibrating wire; B, suspended sinker; C, permanent magnet; D, high-pressure vessel; E, function generator that provides the driving signal; F, lock-in amplifier. Reproduced from reference [15].

With the continuous improvement of the apparatus, the vibrating-wire instrument can be used to determine density and viscosity data for compressed liquid systems at temperatures to 200°C and pressures to 200 MPa. For example, Caudwell *et al.* [13,14] determined the density and viscosity data for a series of hydrocarbons including *n*-octane, *n*-decane, *n*-dodecane, *n*-octadecane, 1,3-dimethylbenzene, 1,2,3,4-tetrahydronaphthalene, and 1-methylnaphthalene over the temperature range of 25°C to 200°C and pressure range of 0.1 MPa to 200 MPa. The estimated uncertainty was 0.2% in density over the entire temperature and pressure range. The percent deviations between the measured density data and the literature data were generally within $\pm 0.5\%$, although the percent deviations in the range of -0.9% to 0.5% were obtained for *n*-octane at the temperatures exceeding 50°C.

As mentioned before, one of the most important benefits for the vibrating-body instrument is its capability of simultaneously determining both density and viscosity data over a wide range of temperatures and pressures. Given that most viscosity measurement techniques require the fluid's density data that have to be determined separately with a densimeter, the vibrating-body method eliminates errors of producing identical temperature and pressure conditions in separate

instruments [29]. The accuracy of this apparatus is also valued considering that the uncertainty within 0.2% can be obtained even at temperatures to 200°C and pressures to 200 MPa. Further, by simply modifying the wire radius and/or wire material, the instrument can be used for the determination for a variety of fluids with different viscosity ranges [25].

Nevertheless, the torsion wire limits the use of the apparatus at extreme temperatures. To date, no density data have been reported with a vibrating-body instrument at temperatures above 200°C, since the elastic properties of the wire begin to vary greatly at extreme temperatures during the measurement. This argument is also strengthened by the fact of an increase in the uncertainty in toluene densities at temperatures above 125°C observed by Krall *et al.* [28] and an increase in the percent deviation between the experimental density data and the literature data for *n*-octane at temperatures above 50°C observed by Caudwell *et al.* [14]. Another disadvantage is that the fluid of interest in the high-pressure vessel cannot be observed from outside. This is usually not so crucial until the fluid of interest experiences a phase change, for example a liquid long-chain *n*-alkane may transform to a solid phase when increasing the pressure.

Bellow Volumometer

It has been a long history for the bellow volumometer to be used for the high-pressure density determination [30-47] since Bridgman first employed this equipment for the density determination at pressures to 5000 MPa [30-33], which guided him to be a Nobel laureate. It operates by the principle that the length of the bellows changes with the applied pressure. At a given pressure, the change of volume is obtained by measuring the change of length of the bellows by some instrument, such as a linear variable differential transformer (LVDT) and, hence, the density of the fluid inside the bellows can be calculated with a known mass of the fluid loaded into the volumometer.

Figure 2 gives an example of an experimental set-up of the bellow volumometer [37]. A given amount of sample is loaded into the bellows in a pressure vessel. The bellows stay within a stainless steel tube and can travel longitudinally within the tube when compressing the samples. The plug in the lower end of the bellows is threaded to carry a stainless steel rod with a magnetic core at the end. The samples in the bellows are compressed by contracting the bellows with some hydraulic fluid. The contraction of the bellows makes the core at the end of the rod accordingly travel through a LVDT in the lower section of the pressure vessel, which then generates the information related to the change in volume, ΔV . The initial volume V_0 can be obtained from the sample mass, m , and the density at atmosphere pressure, and therefore the density at a given pressure, ρ , can be calculated with the equation

$$\rho = m / (V_0 - \Delta V) \quad (1)$$

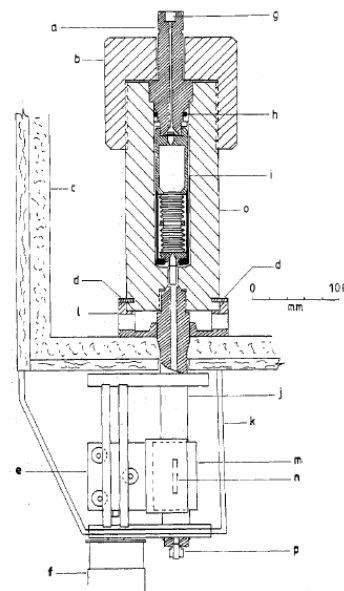


Figure 2. Schematic diagram of a bellow volumometer: a, closure plug; b, cap nut; c, thermostat bath; d, clamp ring; e, LVDT support; f, micrometer head; g, hydraulic oil inlet; h, o-ring seal; i, volumometer cell; j, lower pressure vessel; k, air bath; l, support base; m, LVDT windings; n, core; o, main enclosure vessel; p, drain plug. Reproduced from reference [37].

One of the most important advantages for the bellow volumometer is its capability of measuring densities at ultra-high pressures within a fair temperature range. Bridgman [30-33], the pioneer for the thermophysical properties determination at extreme conditions, reported the densities at the temperature to 175°C and pressures to 5000 MPa for a large number of compounds including *n*-alkanes, cyclic hydrocarbons, aromatic hydrocarbons, alcohols, halocarbons, and so forth. However, the uncertainty for the density measurement was not given. Woolf's research group [37-47] determined density data for a number of organic compounds at temperatures to 100°C and pressures to 500 MPa with a bellow system. The uncertainty in density is within 0.2% over the entire temperature and pressure range.

There are several disadvantages for the bellow volumometer. First, the bellows may cause a permanent strain when stretching the bellows too much. This drawback may be exacerbated for the density determination of light gases, which possess a large compressibility and therefore need a longer distance to be pressurized to a desired pressure. Secondly, the loss of some mechanical properties may occur for the bellows at high temperatures, leading to a decrease in accuracy of density determination. In fact, most of the density determinations from literature were performed no greater than 100°C [34-47] except densities at 175°C for some compounds reported by Bridgman [30-33] who did not report the uncertainty in density. Thirdly, since the fluid of interest is trapped in the bellows, so any phase change that might happen during the pressurizing process cannot be observed. Further, care must be taken when operating the bellow apparatus since it is possible to break or damage the bellows if the differential pressure exceeds around 0.2 MPa.

Floating-piston Densimeter

Same in principles of density determination with bellow volumometer, floating-piston densimeter also uses volume displacement information to

measure densities, either by fixing the temperature and continuously changing the volume and pressure [4-7,48-54], or by keeping the volume fixed and changing the pressure and temperature [55-58]. The former method that fixes the temperature and changes the volume and pressure is more commonly used, giving waiting longer time needed for a change in temperatures. Unlike bellow systems, the floating-piston technique uses a piston with a special o-ring to generate high-pressure conditions and separate the testing fluid from the hydraulic fluid.

Figure 3 shows the schematic diagram of a typical floating-piston view cell apparatus [4]. A hole is drilled through a cylindrical view cell. A sapphire window is fitted to one end of the cell, which is sealed by an o-ring and a backup ring. The piston is placed in the cell with an o-ring used to separate the fluid of interest from a hydraulic fluid. The fluid of interest is then loaded into the cell through one of the side ports and compressed to the desired operating pressure by displacing the piston in the cell using a hydraulic fluid pressurized with a high-pressure generator. The pressure is measured on the hydraulic fluid side of the piston and a rod with a magnetic core is also connected to this side. The core travels through an LVDT to measure the change of the internal cell volume due to the displacement of the piston. The whole cell can be wrapped with heating bands to obtain high operating temperatures and a thermocouple is connected to one of the side ports for the temperature measurement of the fluid in the view cell. Knowing the mass of the fluid of interest loaded into the cell from an external weighing scale before loading and the internal cell volume from LVDT, densities can be calculated at a given temperature and pressure.

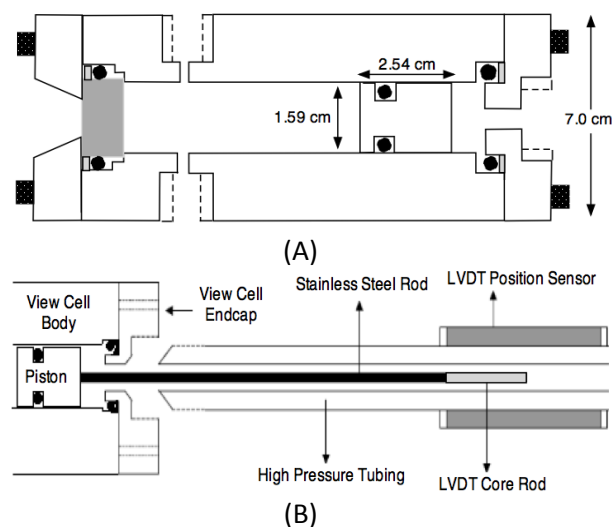
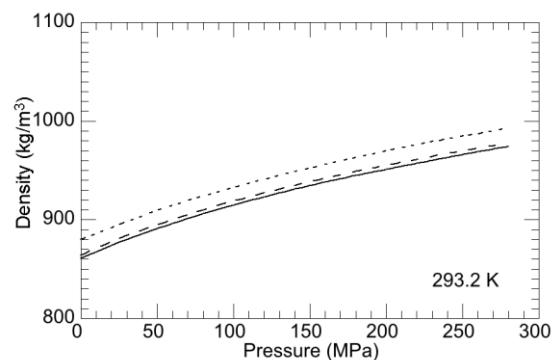


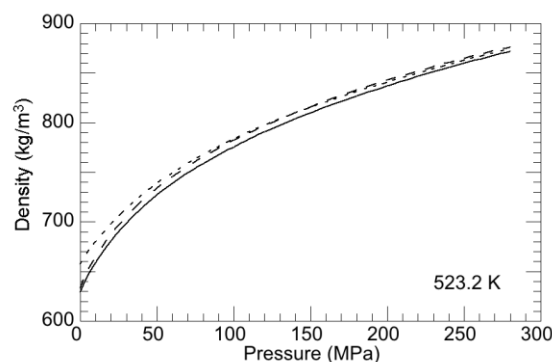
Figure 3. Schematic diagram of (A) a floating-piston apparatus and (B) the LVDT used for volume measurements. Reproduced from reference [4].

With the floating-piston apparatus, Wu *et al.* [6,7] determined the densities of cyclic isomers (ethylcyclohexane, *cis*-1,2-dimethylcyclohexane, *cis*-1,4-dimethylcyclohexane, and *trans*-1,4-dimethylcyclohexane) and aromatic isomers (*o*-xylene, *m*-xylene, and *p*-xylene) at the temperatures to 250°C and pressures to 275 MPa. The density data at each temperature isotherm were obtained by continuous changing the volume and pressure followed by the same procedure at another temperature. The density data agreed with available literature data within $\pm 0.4\%$. The ordering of density isotherms for both cyclic and aromatic isomers were compared at the lowest temperature, 20°C, and the highest temperature, 250°C over the entire pressure range, which agreed with the density ordering at the atmosphere pressure reported in the literature. Wu *et al.* [6,7] also observed that the density differences among the isomers were smaller at 250°C than at 20°C. For example, the density isotherms for *o*-xylene, *m*-xylene, and *p*-xylene became almost indistinguishable at 250°C, as is shown in Figure 4 [7]. Densities of toluene were reported at temperatures to 400°C and pressures to 300 MPa by Frank *et al.* [58], who used the same apparatus but a different method

with Wu *et al.* by holding the volume constant and changing the temperature and pressure. The measured density data agreed with literature data within 0.2% above 100 MPa and 1.2% below 100 MPa.



(A)



(B)

Figure 4. Densities of cis-1,2 (---), ethylc6 (—), cis-1,4 (•••), and trans-1,4 (- •• - ••) at (A) 20°C and (B) 250°C. Reproduced from reference [7].

The floating-piston densimeter is able to determine densities at extreme temperatures with a good choice of o-rings [58]. Unlike vibrating-body and bellows instrument, it also allows the observation of inside fluid through a sapphire window. Hence, it is possible for the floating-piston densimeter to determine the density and phase behavior with the same equipment simultaneously [6,7].

However, care must be taken when choosing the o-rings when assembling the instrument. The material of o-rings will affect whether or not one experiment run can be performed successfully

and how accurate the density data are. The properties of the testing fluid and the operating temperatures should be taken into account when choosing o-rings. For example, fluorocarbon o-rings (e.g. Viton® o-rings) are usually used for the density measurement of hydrocarbons while ethylene propylene o-rings are more suitable for the determination of some polar organic compounds such as acetone and dimethylether that might greatly swell the fluorocarbon o-rings. O-ring-fluid compatibility information can be found on webpages of most o-ring companies [59], which is used as a guide for choosing appropriate o-rings for different testing fluids. For the measurement at extreme temperatures, special o-rings are needed such as polytetrafluoroethylene (PTFE) and Viton® Extreme o-rings, which are designed for high-temperature use. Further, during the density determination for light gases, a moderate amount of testing fluids may penetrate into o-rings at elevated temperatures, leading to some errors in the determined densities.

Other Techniques

In addition to the aforementioned commonly used techniques, there are a number of other methods for the high-pressure density determination. However, it is hard to evaluate if these methods are applicable to a wider temperature and pressure range as well as to a variety of fluids due to limited studies for each method reported in the literature. The following part will give a brief introduction of these methods.

Glen and Johns [60] reported the use of a hydrostatic balance densimeter for the determination of toluene densities at the temperature to 100°C and pressures to 30 MPa. The main part of the densimeter includes a balance with high accuracy and a sinker immersed in the fluid of interest. The buoyancy forces of the fluid exerted on the sinker are determined by the balance and the density can be calculated as

$$\rho = \frac{m_s - m_s^*}{V_s} \quad (2)$$

where m_s and m_s^* are the mass of the sink weighed in the vacuum and weighed in the testing fluid, respectively. V_s is the volume of the sinker, which is calibrated at 20°C and atmosphere pressure. The temperature and pressure dependence of the sinker volume is also taken into account with a correlation equation

$$V_s = V_0 [1 + 3\varepsilon_t (T - T_0)] [1 - \kappa_t (P - P_0)] \quad (3)$$

where V_0 , T_0 , and P_0 are volume, temperature, and pressure at a specified reference condition, respectively. ε_t and κ_t are the thermal expansion and isothermal compressibility, respectively, which are functions of temperature and can be obtained from other literature [60]. The estimated uncertainty in the toluene density and the percent deviation between the determined data and literature data are both within $\pm 0.01\%$ over the studied temperature and pressure range.

Glaser *et al.* [61] reported density data of n-hexadecane and methane-eicosane mixture at temperatures to 90°C and pressure to 18 MPa with a Cailletet tube. Figure 5 shows the schematic diagram of a Cailletet apparatus [62], which uses mercury as a sealing and pressure-transmitting fluid. Same in principle with bellows and floating-piston apparatus, the Cailletet tube calculates the density by determining the volume, $V(P, T)$, at each temperature and pressure level. To obtain $V(P, T)$, the height, h , is measured from the top of the tube to the mercury meniscus and the following calibration equation is used.

$$V(P, T) = (ah + b) [1 + \alpha(T - 293.15)] \cdot [1 + 1.07 \times 10^{-11} (P - 101325)] \quad (4)$$

where P and T are pressures in Pa and temperatures in K. a and b are coefficients

obtained from calibration experiments, which are independent of temperatures and pressures. α is the isobaric expansivity of glass. Same with the floating-piston apparatus, the Cailletet tube is able to simultaneously determine high-pressure density and phase transition data in a single experiment. However, the operation of this system needs to handle hazardous mercury and the glass tube limits the use of the apparatus at ultra-high pressures [63].

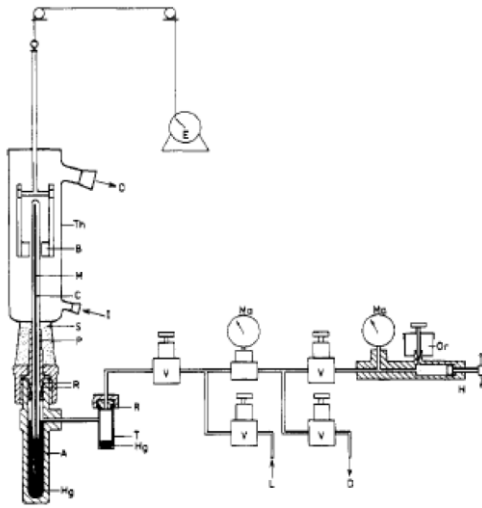


Figure 5. Schematic diagram of Cailletet apparatus: A, autoclave; B, button magnets; C, capillary glass tube; D, drain; H, rotating hand pump; Hg, mercury; I, thermostat liquid in; L, line to dead weight pressure gauges; M, mixture being investigated; Ma, manometers; O, thermostat liquid out; Or, hydraulic oil reservoir; P, closing plug; R, Viton o-rings; S, silicone rubber stopper; T, mercury trap; Th, glass thermostat; V, valve. Reproduced from reference [62].

Tanaka *et al.* [64,65] used a high-pressure burette apparatus made of Pyrex glass for the determination of densities of hydrocarbon mixtures and ethanol-water mixtures at temperatures to 75°C and pressures to 150 MPa. The bottom part of the burette was immersed in mercury. When subjected to a given pressure, the mercury was pushed into the apparatus through a tube. By measuring the volume of the mercury that went into the apparatus at a given pressure, the volume of the testing fluid can be calculated and thus the density can be obtained

with a known mass of the fluid loaded into the apparatus. The uncertainty in density was estimated to be within 0.1%. Same with the Cailletet tube, the high-pressure burette apparatus also suffers from the issue of handling mercury and limitation of glass-made equipment for the use at ultra-high pressures.

Daridon *et al.* [66-69] measured the speeds of sound at different temperatures and pressures and then correlated to the densities with the equation

$$\rho(P, T) = \rho(P_0, T) + \int_{P_0}^P c^{-2} dP + T \int_{P_0}^P \frac{\alpha_p^2}{C_p} dP \quad (5)$$

where $\rho(P_0, T)$ designates the density at the temperature T at the atmosphere pressure P_0 , and c is the speed of sound. α and C_p are the isobaric coefficient of thermal expansion and isobaric heat capacity, respectively. The first term on the right hand side of the equation, $\rho(P_0, T)$, can be obtained from experimental measurements with a regular densimeter. The second term is calculated with a correlation equation of c with P and T [66,68], and the third term is computed iteratively using a predictor-corrector procedure [66]. Besides, other thermophysical properties such as isentropic and isothermal compressibility were also calculated with the speed of sound. The data agreed with literature data within 0.5%.

Conclusion

Vibrating-body, bellows, and floating piston techniques are most commonly used techniques for determination of PVT properties over a wide range of temperatures and pressures. Vibrating-body apparatus is known for its high accuracy; whereas the use is limited at high temperatures due to the great variation of the elastic properties of the wire. Bellows system can be used to measure densities at extremely high pressures, but care must be taken to avoid the permanent strain of the bellows due to the stretching and the easy breaking due to the high-pressure difference between the two sides of the

bellows. Floating-piston system is valued for high-temperature density determination and direct observation of the testing fluid inside the apparatus, although it needs careful choice of o-rings before an experiment. There are also several other techniques for the high-pressure density measurement. However, limited data make it hard to evaluate their accuracy for a wide temperature and pressure range as well as for different fluids of interest.

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