

Automated Densimeter for the Rapid Characterization of Industrial Fluids[†]

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An apparatus for the automated measurement of the densities of compressed liquids over the temperature range of 270–470 K with pressures to 50 MPa is described. The heart of the apparatus is a commercial vibrating-tube densimeter. To minimize the uncertainty in our measurements, several physical and procedural improvements have been implemented beyond that of the commercial instrument operated in a stand-alone mode. The apparatus was designed, and software has been written, so that the operation and data acquisition are fully automated. The densimeter was calibrated using measurements under vacuum, as well as in propane and toluene, over the temperature and pressure ranges of the apparatus. Performance measurements on dimethyl ether are reported from 270 K to 470 K, up to pressures of 46 MPa, with an uncertainty of 0.64–0.81 kg/m³.

1. Introduction

Accurate measurement of the densities of compressed liquids is important to provide the reliable data that is necessary for the development of mathematical models (equations of state, EOS) used to predict the thermophysical properties of a fluid. These models then serve as a valuable tool for a wide range of applications, aiding in the design and optimization of industrial processes.

In this paper, we describe a new apparatus for the measurement of densities of compressed liquids. The instrument is based on a vibrating-tube densimeter. This method of measuring density is well-known and widely used.^{1–5} Because the measurement technique is not absolute, the accuracy of the data relies greatly on the accuracy of the calibration used to relate the response of the instrument to the measured density, and we describe calibration protocols. The apparatus described here was developed to measure a wide range of fluids in the compressed liquid state, and to do so in a highly automated manner such that the measurement of a complete liquid surface can be made with little operator intervention. This measurement capability nicely complements other apparatus within our research group at NIST.^{6,7}

Recently, there has been considerable interest in dimethyl ether (DME) as a fluid with a wide variety of potential commercial/industrial applications, some of which include application as a multipurpose clean fuel^{8,9} and as a chemical feedstock. DME is currently used as a refrigerant, a solvent, an extraction agent, and as a propellant in aerosols. The compressed liquid densities of DME were measured in this work to prove the capability of the new apparatus, and because there is little data available on this fluid in the literature.

2. Apparatus Description

Figure 1 illustrates a schematic of the apparatus that we have designed and constructed for the measurement of the densities

of compressed liquids. The apparatus consists of the following main components: an Anton Paar DMA-HPM vibrating-tube densimeter housed in a custom designed thermostat; a computer-controlled syringe pump; an oscillating quartz-crystal pressure transducer (PT); platinum resistance thermometers (PRTs); and a computer for instrument control and data acquisition. The programmable syringe pump and the two electronically actuated pneumatic valves (PV1 and PV2), together with the PT, with a range of 0–69 MPa, control the system pressure and fluid flow.

2.1. Densimeter and Thermostat. The basic principle of the measurement technique used in this work is that the period of the oscillation of the vibrating tube can be related to the density of the fluid inside the tube. In our apparatus, the vibrating U-tube is composed of Hastelloy C-276 and operates over a wide range of temperature (–20 °C to 200 °C) and pressure (up to 100 MPa). The densimeter is housed in a specially designed two-stage thermostat (illustrated in Figure 2), to achieve precise temperature control. The densimeter is the inner stage of the thermostat. The manufacturer-provided densimeter cover was replaced with copper plates that were machined to match the dimensions of the sides and top of the densimeter housing. This was done to increase the thermal mass and provide better thermal conduction, thus reducing possible temperature gradients along the length of the U-tube. The copper plates are screwed to the densimeter housing, and thin-film heaters are glued to each of the plates, to provide fine temperature control at the densimeter. One of the side plates has a thermowell, in which a standard platinum resistance thermometer (SPRT) is inserted to measure the densimeter temperature. In addition, a sample preheat loop on the inlet side of the system is brazed to one of the copper side plates to bring the fluid sample to temperature equilibrium more quickly.

The outer stage (shield) of the thermostat is a copper pipe 20.6 cm in diameter. It is maintained at a temperature 3 K lower than the densimeter, and thus it serves to minimize heat exchange between the densimeter and the ambient environment. The pipe is 47.6 cm in length and has had part of the circumference cut away longitudinally to produce an open-topped tube. End plates were machined and brazed in place on both ends of the pipe, and a top plate was fabricated to cover the open section, so that the shield would completely enclose the densimeter. The densimeter sits on standoffs inside the copper pipe, and glass wool insulation is packed around it. Insulation also surrounds the outside of the copper pipe, which

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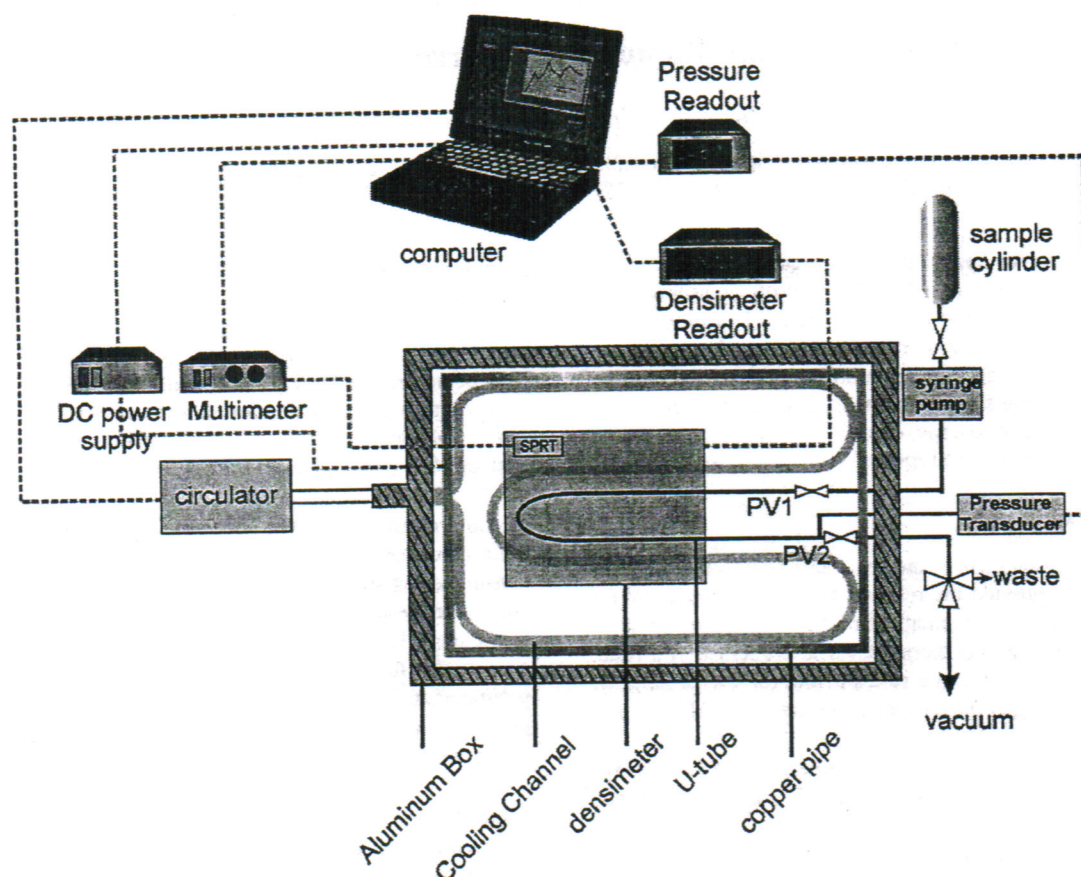


Figure 1. Schematic of the densimeter apparatus.

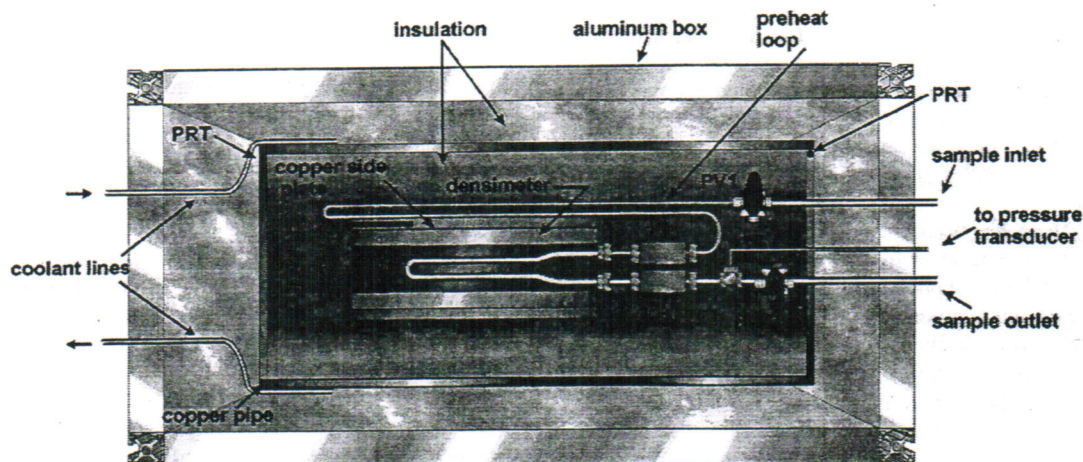


Figure 2. Top view of the thermostat.

is housed in an aluminum box to enclose the thermostated portion of the apparatus.

The apparatus temperature is controlled with electric heaters and a circulating bath. Thin-film heaters are glued to the densimeter and to the outside of the copper pipe. Copper tubing brazed onto the outside of the pipe in a serpentine pattern is connected to the circulator, to achieve subambient system temperatures. Two 100 Ω PRTs are located in the thermowells (one at each end of the copper pipe), to measure any temperature gradients along the length of the pipe. Readings from the SPRT and two PRTs are used by a proportional–integral–differential (PID) routine to calculate the necessary power output to the heaters. The shield (copper pipe) is controlled at 3 K below the

temperature setpoint, creating a small heat sink for the densimeter. This makes it possible to use very little power on the densimeter heaters to maintain the desired temperature with minimal thermal pulsing. In this manner, temperature stability (at the densimeter) of ± 5 mK is achieved.

2.2. Control and Data Acquisition. The apparatus is operated along isotherms with the programmable syringe pump controlling the system pressure. System pressure is measured by an oscillating quartz-crystal pressure transducer located outside of the aluminum box. The pressure transducer is housed in a thermostat and maintained at a constant temperature of 313 K to decrease the uncertainty in the pressure measurement.

Table 1. Values of Calibration Equation Parameters

A_i Parameters		B_i Parameters	
parameter	value	parameter	value
A_1	0.98469366×10^1	B_1	0.98462808×10^1
A_2	$-0.53751201 \times 10^{-5}$	B_2	$-0.25582589 \times 10^{-5}$
A_3	$-0.25400100 \times 10^{-5}$	B_3	$0.55902893 \times 10^{-8}$
A_4	$0.55912960 \times 10^{-8}$	B_4	$0.44041816 \times 10^{-6}$
A_5	$0.32030137 \times 10^{-6}$	B_5	$-0.76134993 \times 10^{-11}$
A_6	$-0.68853057 \times 10^{-11}$	B_6	$-0.49949869 \times 10^{-8}$
A_7	$-0.48533567 \times 10^{-8}$		

The experiment control and data acquisition for the liquid densimeter are achieved with software written in LabVIEW. A multimeter and power supplies are read and controlled via IEEE-488 communications; the rest of the equipment uses serial communications. In the main program, the operator is prompted to enter a temperature/pressure matrix that specifies what state points will be measured. After that information has been entered, the temperature control program is launched. The temperature control program runs independently but communicates with the main program via an internal server. By operating independent of the main program, the temperature control loop can run more frequently, and this facilitates better temperature control. Measurements of the temperature, pressure, and the period of oscillation of the vibrating tube are monitored every 30 s. The system is considered to be at equilibrium when, over 15 consecutive minutes, the standard deviation in the temperature is ≤ 3.5 mK, the standard deviation in the pressure is ≤ 5 kPa, and the standard deviation in the period of oscillation of the vibrating tube is ≤ 2 ns (which corresponds to ~ 0.01 kg/m³). After these convergence criteria are met, data are recorded and averaged for 5 more minutes. The control program then checks to see if there is another point to be measured, and, if so, it adjusts the temperature and/or pressure accordingly and again waits for convergence.

3. Calibration and Uncertainty

The measurement of density with a vibrating tube is not absolute; thus, the response of the instrument must be calibrated against fluids of known density. The calibration of the period of oscillation of the vibrating tube included measurements under vacuum from 270 K to 470 K at intervals of 20 K. Propane and toluene were measured at the same temperatures as vacuum: propane at pressures of 2–35 MPa, covering densities of 174–577 kg/m³, and toluene at pressures of 1.2–46 MPa, covering densities of 682–918 kg/m³. The propane had a stated purity of 99.999%, which was verified in-house, and the toluene was NIST Standard Reference Material 211d. Density values for propane were calculated with the equation of state (EOS) of Lemmon et al.,¹⁰ and the toluene densities were calculated with the equation of Lemmon and Span.¹¹ The NIST REFPROP database¹² was used to implement both of the EOSs. The vacuum data were fitted to the second-order polynomial

$$\tau_0 = c_0 + c_1T + c_2T^2 \quad (1)$$

where τ_0 is the period of oscillation (in microseconds) of the vibrating tube under vacuum, as a function of temperature. Density is calibrated in terms of the following 13-term equation:

$$\rho = (A_1 + A_2T + A_3T^2 + A_4T^3 + A_5P + A_6P^2 + A_7TP) \frac{\tau^2}{\tau_0^2} - (B_1 + B_2T^2 + B_3T^3 + B_4P + B_5P^2 + B_6TP) \quad (2)$$

where τ is the period of oscillation of the tube when filled with the compressed fluid. The values for A_i and B_i are given in Table 1. This equation closely resembles that which Ihmels et al.¹³ used to calibrate a similar vibrating-tube instrument. The exception is that one term was dropped, because it was determined to be statistically insignificant for our purposes.

The fitting software ODRPack¹⁴ was used to determine the values of A_i and B_i in eq 2. The propane data were fit with an absolute average deviation (AAD) of 0.10 kg/m³, and the toluene data with an AAD of 0.12 kg/m³. In the density range measured with propane, the equation of Lemmon et al.¹⁰ has an uncertainty of 0.005% ($k = 1$), which equates to ~ 0.04 kg/m³. The toluene equation has an uncertainty of 0.025% (or 0.15–0.25 kg/m³) over the densities used in the calibration.

The SPRT, the pressure transducer, and the period of oscillation of the vibrating tube are calibrated annually. The SPRT is calibrated against the triple points of water and mercury and the freezing points of tin and indium. The standard combined uncertainty in our temperature measurements including the uncertainties in the SPRT, the multimeter, and the calibration; and the possible temperature gradient between the U-tube and the SPRT is estimated to be 30 mK ($k = 1$). The two 100- Ω PRTs are calibrated against the triple point of water and the freezing point of indium; we have not assigned an uncertainty to these, because they are used only for temperature control of the shield, not to measure the temperature reported for a density measurement. The quartz-crystal pressure transducer is calibrated with a NIST-traceable dead-weight pressure gauge. The manufacturer's stated uncertainty of the PT is 0.01% of full range, which equates to ~ 7 kPa ($k = 2$); however, we have observed that, with regular calibration and by keeping the transmitter at a constant temperature, we can reduce the uncertainty to 0.005% of full range (or 3.5 kPa). The vacuum pressure at 310 K is checked periodically, and the reported pressures are adjusted to reflect any offset. Thus, we conservatively estimate our pressure uncertainty to be 10 kPa ($k = 2$).

The overall uncertainty in our reported densities is estimated to be in the range of 0.64–0.81 kg/m³, based on the parameters and their associated uncertainties, as shown in Table 2. An important component of the overall uncertainty comes from the uncertainty of the density of the calibration fluids. The overall

Table 2. Experimental Parameters Contributing to the Overall Measurement Uncertainty

property	uncertainty ($k = 1$)	equivalent in density [kg/m ³]
EOS for propane	0.005%	0.04
EOS for toluene	0.025%	0.15 to 0.25
temperature measurement	0.03 K	0.1
pressure measurement	5 kPa	0.02
calibration fluid impurity	0.05% heavy impurity	0.03
repeatability of vacuum	0.3 kg/m ³	0.3
combined standard uncertainty		0.32–0.40
expanded uncertainty ($k = 2$)		0.64–0.81

**Table 3. Compressed Liquid Densities of Dimethyl Ether:
270–370 K**

temperature [K]	pressure [MPa]	density [kg/m ³]
270.00	35.516	744.08
270.00	32.371	741.20
270.00	29.294	738.27
270.00	26.269	735.31
270.00	23.419	732.44
270.00	20.543	729.46
270.00	18.599	727.37
270.00	16.634	725.22
270.00	14.588	722.89
270.00	12.616	720.61
270.00	10.566	718.18
270.00	9.486	716.84
270.00	8.377	715.47
270.00	7.327	714.14
270.00	6.385	712.92
270.00	5.054	711.16
270.00	3.986	709.72
270.00	2.974	708.36
270.00	1.946	706.94
290.00	35.661	721.89
290.00	32.325	718.34
290.00	29.313	715.01
290.00	26.369	711.63
290.00	23.525	708.23
290.00	20.605	704.56
290.00	18.585	701.94
290.00	16.575	699.26
290.00	14.549	696.46
290.00	12.480	693.52
290.00	10.346	690.41
290.00	9.279	688.80
290.00	8.328	687.36
290.00	7.157	685.51
290.00	6.105	683.81
290.00	5.028	682.05
290.00	4.084	680.43
290.00	2.944	678.46
290.00	1.910	676.65
290.00	0.974	675.00
310.00	36.254	700.40
310.00	30.388	693.21
310.00	25.521	686.74
310.00	20.618	679.70
310.00	15.510	671.67
310.00	10.285	662.47
310.00	5.055	651.91
310.00	4.047	649.65
310.00	3.056	647.36
310.00	2.057	644.95
330.00	46.182	689.73
330.00	35.280	675.61
330.00	25.410	660.75
330.00	15.288	642.16
350.00	46.719	668.93
350.00	35.284	652.19
350.00	25.441	634.81
350.00	15.190	611.63
370.00	46.484	647.08
370.00	35.328	628.40
370.00	25.412	607.68
370.00	15.131	578.46

uncertainty in the reported density can be no smaller than the uncertainties in the EOSs for the fluids used in the calibration of the period of the vibrating tube. However, this is a point that is overlooked by many who use this method to determine density. The expanded uncertainty for our measurements was calculated by the root-sum-of-squares method¹⁵ with $k = 2$. The total uncertainty range is based on the density range of the fluid being measured. For instance, a fluid whose densities rely more

**Table 4. Compressed Liquid Densities of Dimethyl Ether:
390–470 K**

temperature [K]	pressure [MPa]	density [kg/m ³]
390.00	46.263	624.97
390.00	40.363	614.25
390.00	35.354	604.02
390.00	30.434	592.70
390.00	25.374	579.26
390.00	20.243	562.87
390.00	15.135	542.00
390.00	10.164	513.00
390.00	9.170	505.18
390.00	8.153	495.90
390.00	7.147	484.90
390.00	6.129	470.70
390.00	5.099	449.84
395.00	45.766	618.64
395.00	40.340	608.42
395.00	35.376	597.94
395.00	30.477	586.23
395.00	25.351	571.95
395.00	20.231	554.63
395.00	15.150	532.35
395.00	10.166	500.06
395.00	9.155	490.84
395.00	8.147	479.90
395.00	7.141	466.23
395.00	6.113	446.85
405.00	45.945	607.95
405.00	40.375	596.78
405.00	35.435	585.61
405.00	30.437	572.68
405.00	25.337	557.08
405.00	20.216	537.67
405.00	15.173	512.02
405.00	10.155	471.10
405.00	9.140	458.15
405.00	8.128	441.44
405.00	7.082	415.86
405.00	6.025	352.47
410.00	45.692	602.00
410.00	35.464	579.50
410.00	25.343	549.66
410.00	15.182	501.49
410.00	10.153	455.01
410.00	9.128	439.14
410.00	8.098	417.03
410.00	7.060	378.19
410.00	6.009	213.40
430.00	46.626	581.96
430.00	35.476	554.28
430.00	25.269	518.44
430.00	15.186	455.07
430.00	10.086	372.00
430.00	9.063	332.33
430.00	8.035	264.71
430.00	7.019	184.36
450.00	46.074	559.04
450.00	35.432	528.84
450.00	25.218	486.29
450.00	15.160	402.66
450.00	10.061	272.85
450.00	9.039	225.03
450.00	8.025	178.46
470.00	45.984	537.14
470.00	35.396	503.23
470.00	25.223	453.66
470.00	15.134	347.78
470.00	10.062	210.08
470.00	9.049	176.72
470.00	8.035	146.06

on the toluene region of the calibration will have a higher uncertainty than one whose densities are more similar to those of propane.

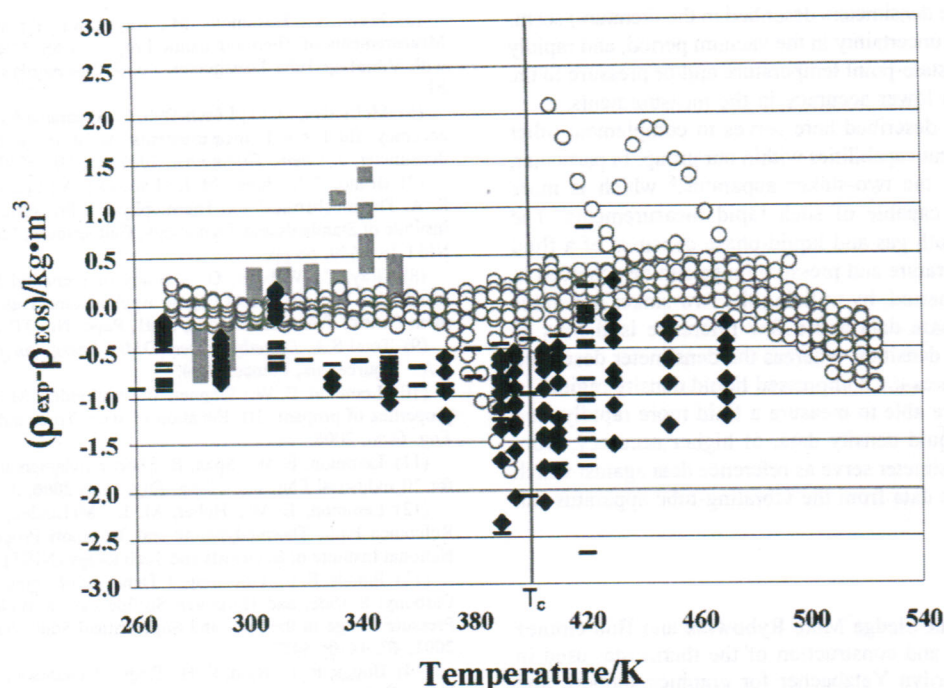


Figure 3. Deviations of available data from the equation of state (EOS) of Ihmels and Lemmon:¹⁶ (◆) this work; (○) Ihmels and Lemmon;¹⁶ (■), Bobbo et al.;³ (□), McLinden.¹⁷

4. Results for Dimethyl Ether

Compressed liquid densities of dimethyl ether (DME) were measured from 270 K to 470 K at pressures of 1–46 MPa over a density range of 146–744 kg/m³. A total of 129 points are reported in Tables 3 and 4. DME was chosen to demonstrate the capabilities of this apparatus for several reasons, two of which include (i) the compressed liquid densities of DME are within the range of our calibration, and (ii) data on this fluid had been measured with other similar instruments, and those data had been correlated with an EOS that could be used as a baseline against which we could compare our data to the other sources. Figure 3 shows deviations of measured densities from the EOS of Ihmels and Lemmon¹⁶ for our data, and the data of Bobbo et al.,³ Ihmels and Lemmon,¹⁶ and McLinden.¹⁷ Bobbo et al.³ measured densities at temperatures from 283 K to 353 K at pressures to 35 MPa with a vibrating-tube densimeter. Ihmels and Lemmon¹⁶ also used a vibrating-tube apparatus and measured densities at temperatures from 273 K to 523 K, up to pressures of 40 MPa, and McLinden¹⁷ measured densities at temperatures from 220 to 420 K, up to pressures of 34 MPa with a highly accurate dual-sinker densimeter (which is described in ref 6).

Figure 3 shows fairly good agreement between all four data sets at temperatures <330 K. At temperatures from 330 K to 370 K, a few of our data show negative deviations greater than our stated uncertainty, but they are in good agreement with the data of McLinden.¹⁷ At higher temperatures (particularly near the critical temperature of 400.3 K), our data agree very well with the data of McLinden,¹⁷ with slightly greater deviations than those of Ihmels and Lemmon.¹⁶ The deviations of our data from the baseline equation of Ihmels and Lemmon¹⁶ are due in part to a lack of agreement between our data and the data of Ihmels and Lemmon¹⁶ and Bobbo et al.³ The formulation of the baseline equation included the data of Ihmels and Lemmon¹⁶ and Bobbo et al.,³ but not our data or the data of McLinden;¹⁷ as a result, generally, our data and the data of McLinden¹⁷ have a tendency to have larger deviations from the baseline than those

of Ihmels and Lemmon¹⁶ and Bobbo et al.³ The stated uncertainty in the Bobbo et al.³ data is $\pm 0.1\%$ (or ~ 0.7 kg/m³), and this is also the value assigned to the Ihmels and Lemmon¹⁶ data, except in the critical region, where their stated uncertainty increases to 1%; in addition, note that, for both of these data sets, there are points that deviate from the baseline at values greater than their stated uncertainty. The large deviations of our data and Ihmels and Lemmon¹⁶ in the near-critical and critical regions illustrate the susceptibility of the vibrating-tube measurement method to small errors in temperature and pressure in this region, resulting in less-accurate measurements. In addition, there is greater uncertainty in the EOS in the critical and near-critical regions, so the data there are not fit as well.

5. Discussion and Conclusions

From Table 2, it becomes readily apparent that the greatest contributors to the uncertainty are the uncertainties of the EOS for the calibration fluids and the vacuum calibration. Because water has a more-accurate EOS, using it for the second calibration fluid in lieu of toluene would be one way to reduce the uncertainty associated with the EOSs; however, this is offset by the narrower density range of water and the better match of the propane and toluene densities, compared to those measured here for DME. The uncertainty in the vacuum calibration arises from the repeatability of the vacuum period as a function of temperature. Factors that affect the repeatability include (i) cycling in temperature, which may result in a change in the microstructure of the U-tube; and (ii) the greater pressure capability of this densimeter requires a heavier-than-normal U-tube (i.e., one with thicker walls), so that the sample mass, relative to the tube mass, is lower. In addition, the data that we have gathered to date indicate a hysteresis associated with the period of oscillation of the U-tube, such that several hours (or, in some cases, longer) are required for the vacuum period to reach a true equilibrium state. Thus, the greater temperature and pressure range of our apparatus (in comparison to the majority

of vibrating U-tube densimeters described in the literature) seems to result in greater uncertainty in the vacuum period, and rapidly moving from one state-point temperature and/or pressure to the next may result in lower accuracy in the measurements.

The densimeter described here serves to complement other density measurement capabilities within our group. In particular, it pairs well with the two-sinker apparatus,⁶ which is more accurate, but not capable of such rapid measurements. The measurement of both gas and liquid-phase densities of a fluid over a large temperature and pressure range can be achieved in a relatively short period, by utilizing the two-sinker apparatus to measure gas-phase data and a few reference isotherms of compressed liquid densities, whereas the densimeter described here rapidly measures the compressed liquid density region. In this manner, we are able to measure a fluid more rapidly, and the compressed liquid density data, of higher accuracy, from the two-sinker densimeter serve as reference data against which the accuracy of the data from the vibrating-tube apparatus can be calibrated.

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