

# Compressed-Liquid Density Measurements of Methyl Oleate and Methyl Linoleate

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**ABSTRACT:** Compressed-liquid densities of the fatty acid methyl esters (FAMES) methyl linoleate and methyl oleate have been measured with a vibrating-tube densimeter. The temperature and pressure ranges of the measured data are from (270 to 470) K and (0.5 to 50) MPa. The data at 10 MPa and below have been extrapolated to 0.083 MPa (the approximate ambient pressure in Boulder, CO) for comparison with existing literature data. A correlation is given that represents the temperature and pressure dependence of the reported experimental data within their estimated uncertainties.

## 1. INTRODUCTION

Fatty acid methyl esters (FAMES) include compounds that have a wide scope of applications from the perfume and food industries to being some of the major components of biodiesel fuels. In the production of biodiesel fuel, oil produced from the feedstock is chemically modified through a process called transesterification, which substitutes a simple alcohol, such as methanol, for the complex alcohol, glycerol. The products of this process are primarily FAMES, and the process results in a material with a lower viscosity that is more suitable for fuel than the oil derived from the feedstock. Some of the advantages of biodiesel fuels include the following: it is renewable, it is energy-efficient, it displaces petroleum-derived diesel fuel, it can be used as a 20 % blend in most diesel equipment with no or only minor modifications, and it can reduce global warming gas emissions.<sup>1</sup>

The rising price and unreliable supply of petroleum are galvanizing the search for renewable fuels that can be used within existing infrastructure with few modifications. Biodiesel fuel is a viable candidate, but more research is necessary to understand the full potential of a variety of feedstocks and the characteristics of the resulting fuels. Five fatty acid methyl esters have been found to make up the majority of the components present in biodiesel fuel derived from the majority of current biodiesel fuel feedstocks. Methyl linoleate (CAS Registry No. 112-63-0) and methyl oleate (CAS Registry No. 112-62-9) are the two most important of those five FAMES.<sup>2</sup> Measured compressed-liquid densities of methyl linoleate and methyl oleate from (270 to 470) K and (0.5 to 50) MPa are reported here. The major goal of the measurements reported here is to provide data that are lacking in the literature and thus enable the formulation of more accurate equations of state. Pure fluid equations can then be used in combination to build surrogate mixture models for such complex fluids as the biodiesel fuels.<sup>3</sup> These models can then serve as tools to aid in the design of new equipment and new fuel formulations to increase efficiency.

## 2. SAMPLE LIQUID

The samples measured in this work were purchased from Sigma-Aldrich Corporation (To describe materials and experimental

procedures adequately, it is occasionally necessary to identify commercial products by manufacturers' names or labels. In no instance does such identification imply endorsement by the National Institute of Standards and Technology, nor does it imply that the particular product or equipment is necessarily the best available for the purpose). Both the methyl linoleate and the methyl oleate were specified by the manufacturer as being greater than 99 % pure. Analyses of the samples were performed in our laboratory by gas chromatography–flame ionization detection (GC-FID) on a 30 m capillary column with a 0.1 mm coating of 50 % cyanopropyl/50 % dimethylpolysiloxane as the stationary phase. This phase provides separations based upon polarity and is specifically intended for the analysis of the FAMES that compose biodiesel fuel. Details of the analysis technique are given in Smith et al.<sup>4</sup> and Bruno and Svoronos.<sup>5</sup> Both samples were found to be within manufacturer specifications and showed no detectable impurities.

A small amount of inhibitor (*tert*-butylhydroquinone, TBHQ) was added to both samples to avoid the possibility of chemical decomposition at higher temperatures. The amount of TBHQ added to the methyl linoleate sample was 0.16 % by weight and 0.09 % for the methyl oleate. TBHQ has a higher density than that of either methyl linoleate or methyl oleate. As such, the effect of the addition of TBHQ to our samples would have increased the values of our measured densities. The density of TBHQ at 298.15 K is approximately 1050 kg·m<sup>-3</sup>, while methyl linoleate and methyl oleate have densities of approximately 881 kg·m<sup>-3</sup> and 870 kg·m<sup>-3</sup> at that temperature. As such, the addition of the TBHQ could have potentially raised the densities of our measured values of methyl linoleate by up to 0.3 kg·m<sup>-3</sup> and those of methyl oleate by as much as 0.2 kg·m<sup>-3</sup>.

## 3. EXPERIMENTAL SECTION

The densities of the compressed test liquids were measured with the automated densimeter of Outcalt and McLinden.<sup>6</sup> Central to the apparatus is a commercial vibrating-tube densimeter. Several

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physical and procedural improvements have been implemented beyond that of the commercial instrument operated in a stand-alone mode to minimize the uncertainty in the measurements. Some of these improvements include more accurate measurements of temperature and pressure, better temperature control, and complete automation of the instrument control and data acquisition. The temperature range of the instrument is (270 to 470) K with pressures up to 50 MPa. In this work, we measured 11 isotherms over the range (0.5 to 50) MPa for each of the samples. The instrument was calibrated with propane and toluene over the entire temperature and pressure range. Further details of the calibration procedure can be found in Outcalt and McLinden.<sup>6</sup> The overall combined uncertainty ( $k = 2$ ) in density is at most  $0.81 \text{ kg} \cdot \text{m}^{-3}$ , corresponding to a relative uncertainty in density of 0.09 % to 0.11 %. The addition of the TBHQ to the methyl linoleate and methyl oleate samples is not expected to have changed the overall uncertainty of the measurements, but rather to have added a positive bias to the measurements of approximately  $0.3 \text{ kg} \cdot \text{m}^{-3}$ .

#### 4. RESULTS AND CORRELATIONS

Tables 1 and 2 list the measured density values of the compressed-liquids methyl linoleate and methyl oleate from (270 to 470) K to pressures of 50 MPa, respectively. The tables include density values extrapolated to the local ambient pressure of 0.083 MPa at each temperature. The extrapolated data were obtained by fitting second-order polynomials to the isothermal densities at pressures less than or equal to 10 MPa and then calculating the densities at 0.083 MPa from the polynomials. To compare our data to the ambient pressure data found in the literature, the extrapolated data were correlated with a Rackett-type equation:

$$\rho = \beta_1 \cdot \beta_2^{-(1 + (1 - T/\beta_3)^{\beta_4})} \quad (1)$$

The parameters for eq 1 are listed in Table 3. The critical temperatures of methyl linoleate (767.4 K) and methyl oleate (764.0 K) as found in the DIPPR database<sup>7</sup> were used as the value of the  $\beta_3$  parameter. The values were taken from Lydersen<sup>8</sup> and were estimated by Lydersen's method, based on a predicted boiling point. The average absolute deviation (AAD) of our extrapolated data from the correlation is 0.02 % for methyl linoleate and 0.01 % for methyl oleate.

A survey of the literature for ambient pressure density data found twelve sources of data on methyl linoleate<sup>2,9–19</sup> and nine sources for methyl oleate.<sup>2,19–26</sup> The data sets of Ott et al.<sup>2</sup> and Pratas et al.<sup>19</sup> represent the majority of the available data, while many of the sources represent only one or two data points. This is especially true for methyl linoleate. Figure 1a,b shows deviations of our extrapolated data and the literature data from our correlations for methyl linoleate and methyl oleate, respectively. Also shown in the figures are the deviations of the correlations for saturated liquid densities given in the NIST Standard Reference Database 23 (REFPROP)<sup>27</sup> and the DIPPR<sup>7</sup> database. Both parts of Figure 1, a and b, illustrate that our extrapolated densities are slightly larger than the majority of the literature data and generally agree within 0.2 %. Error bars showing the uncertainty bounds of our data ( $\pm 0.1$  %) and the data of Ott et al. ( $\pm 0.1$  %) are shown in both figures at points around 310 K to help indicate agreement between the two data sets within their combined uncertainties. As mentioned previously, the addition of the TBHQ to both the methyl linoleate and methyl oleate samples would have increased

Table 1. Compressed-Liquid Densities of Methyl Linoleate Measured in the High Pressure Vibrating-Tube Densimeter along Isotherms from (270 to 470) K<sup>a</sup>

270 K	290 K		310 K		330 K		350 K		370 K		390 K		410 K		430 K		450 K		470 K		
	pressure MPa	density kg·m <sup>-3</sup>	pressure MPa	density kg·m <sup>-3</sup>	pressure MPa	density kg·m <sup>-3</sup>	pressure MPa	density kg·m <sup>-3</sup>	pressure MPa	density kg·m <sup>-3</sup>	pressure MPa	density kg·m <sup>-3</sup>	pressure MPa	density kg·m <sup>-3</sup>	pressure MPa	density kg·m <sup>-3</sup>	pressure MPa	density kg·m <sup>-3</sup>	pressure MPa	density kg·m <sup>-3</sup>	
49.97	928.2	49.97	915.3	49.99	902.8	50.00	890.4	49.98	878.4	49.98	866.4	49.99	854.7	49.97	843.3	50.01	831.8	49.98	820.6	49.98	809.7
39.99	923.8	39.99	910.7	39.99	897.8	39.99	885.1	40.00	872.7	39.99	860.4	40.00	848.3	40.00	836.5	40.00	824.5	39.99	812.8	39.99	801.5
29.99	919.3	30.00	905.8	30.00	892.5	29.98	879.5	29.99	866.7	29.99	854.0	29.99	841.4	29.99	829.1	29.99	816.6	30.00	804.3	29.99	792.4
20.00	914.6	19.99	900.7	20.00	887.0	19.99	873.5	19.99	860.3	19.99	847.0	20.00	834.0	19.99	821.0	19.99	807.9	19.99	794.9	19.99	782.2
10.00	909.6	9.99	895.2	9.99	881.1	9.99	867.1	9.99	853.4	10.00	839.5	9.99	825.8	9.99	812.1	9.99	798.1	10.00	784.3	9.99	770.6
5.00	907.0	5.00	892.4	4.99	878.0	4.99	863.8	5.00	849.7	5.00	835.5	4.99	821.3	4.99	807.2	5.00	792.7	4.99	778.3	4.99	764.0
3.99	906.5	3.99	891.8	3.99	877.4	3.99	863.1	3.99	848.9	4.00	834.6	3.99	820.4	3.99	806.2	3.99	791.6	4.00	777.1	3.99	762.6
3.00	905.9	2.99	891.2	2.99	876.7	2.99	862.4	2.99	848.2	2.99	833.8	3.00	819.5	2.99	805.1	2.99	790.5	2.99	775.8	3.00	761.2
1.99	905.4	1.99	890.6	1.99	876.1	1.99	861.6	1.99	847.4	1.99	832.9	1.99	818.5	2.00	804.1	1.99	789.3	2.00	774.5	1.99	759.7
0.99	904.9	0.99	890.0	1.00	875.4	0.99	860.9	1.00	846.6	0.99	832.1	0.99	817.6	1.00	803.0	1.00	788.1	0.99	773.2	0.99	758.3
0.49	904.6	0.50	889.7	0.50	875.1	0.49	860.6	0.50	846.2	0.49	831.6	0.49	817.1	0.49	802.5	0.49	787.5	0.49	772.5	0.50	757.5
0.083	904.4	0.083	889.5	0.083	874.8	0.083	860.3	0.083	845.9	0.083	831.3	0.083	816.7	0.083	802.0	0.083	787.0	0.083	771.9	0.083	756.9

<sup>a</sup> Values extrapolated to 0.083 MPa are indicated in *italics*.

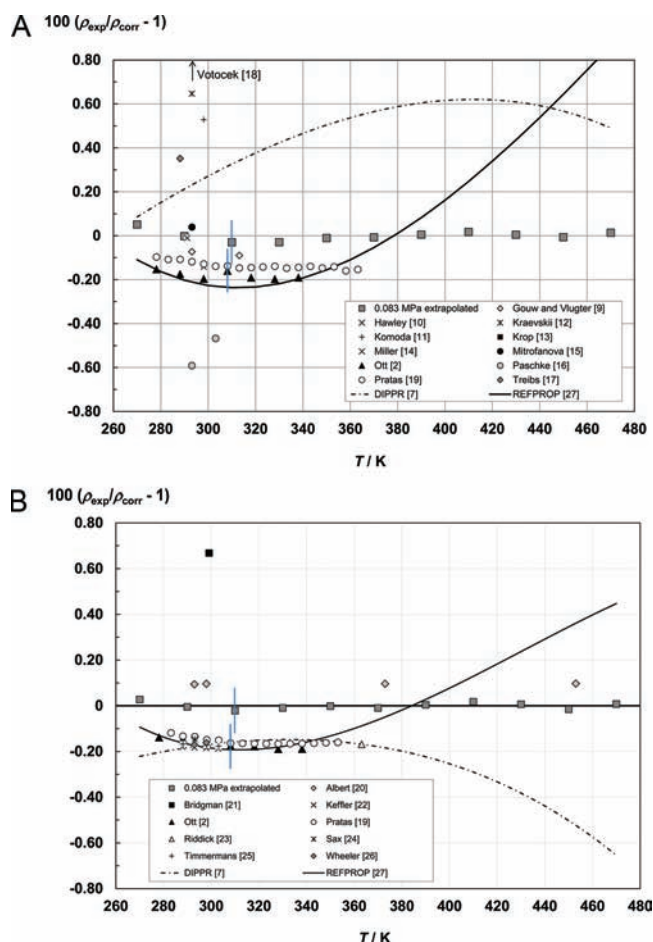
Table 2. Compressed-Liquid Densities of Methyl Oleate Measured in the High-Pressure Vibrating-Tube Densimeter along Isotherms from (270 to 470) K<sup>a</sup>

270 K		290 K		310 K		330 K		350 K		370 K		390 K		410 K		430 K		450 K		470 K	
pressure	density	pressure	density	pressure	density	pressure	density	pressure	density	pressure	density	pressure	density	pressure	density	pressure	density	pressure	density	pressure	density
<i>p</i>	$\rho$	<i>p</i>	$\rho$	<i>p</i>	$\rho$	<i>p</i>	$\rho$	<i>p</i>	$\rho$	<i>p</i>	$\rho$	<i>p</i>	$\rho$	<i>p</i>	$\rho$	<i>p</i>	$\rho$	<i>p</i>	$\rho$	<i>p</i>	$\rho$
MPa	kg·m <sup>-3</sup>	MPa	kg·m <sup>-3</sup>	MPa	kg·m <sup>-3</sup>	MPa	kg·m <sup>-3</sup>	MPa	kg·m <sup>-3</sup>	MPa	kg·m <sup>-3</sup>	MPa	kg·m <sup>-3</sup>	MPa	kg·m <sup>-3</sup>	MPa	kg·m <sup>-3</sup>	MPa	kg·m <sup>-3</sup>	MPa	kg·m <sup>-3</sup>
49.83	915.9	49.96	903.4	49.99	891.1	49.99	878.9	49.98	867.0	49.97	855.1	49.99	843.6	50.00	832.2	49.99	820.8	50.01	809.7	49.97	798.8
39.99	911.7	39.99	898.7	39.99	886.1	39.99	873.6	40.00	861.3	39.99	849.1	40.00	837.2	39.99	825.3	39.99	813.5	39.99	801.9	39.99	790.6
29.99	907.1	29.99	893.8	29.99	880.8	29.99	868.0	30.00	855.3	29.99	842.6	29.98	830.2	30.00	817.9	29.99	805.5	29.99	793.3	29.99	781.5
19.99	902.4	19.99	888.7	20.00	875.3	19.99	862.0	20.00	848.8	20.01	835.7	19.99	822.7	20.00	809.8	19.99	796.7	20.00	783.9	19.99	771.2
9.99	897.4	10.00	883.3	9.99	869.3	10.00	855.6	9.99	841.8	10.00	828.1	10.00	814.5	10.00	800.8	9.99	786.9	10.00	773.1	10.00	759.5
4.99	894.8	4.99	880.4	5.00	866.2	4.99	852.2	5.00	838.1	5.01	824.0	5.00	810.0	5.00	795.8	4.99	781.5	5.00	767.1	5.01	752.9
3.99	894.3	3.99	879.8	4.00	865.6	3.99	851.5	3.99	837.3	3.99	823.2	3.99	809.0	3.99	794.8	3.99	780.3	3.99	765.8	3.99	751.5
2.99	893.7	3.00	879.2	3.00	865.0	3.00	850.8	2.99	836.6	2.99	822.3	3.00	808.1	3.00	793.7	3.00	779.2	2.99	764.5	2.99	750.0
1.99	893.2	2.00	878.6	2.00	864.3	2.00	850.1	2.00	835.8	1.99	821.4	2.00	807.1	2.00	792.7	1.99	778.0	1.99	763.2	2.00	748.6
0.99	892.6	1.00	878.0	0.99	863.6	1.00	849.3	0.99	835.0	0.99	820.6	0.99	806.2	0.99	791.6	1.00	776.8	1.00	761.9	0.99	747.0
0.49	892.3	0.49	877.7	0.49	863.3	0.50	849.0	0.50	834.6	0.50	820.1	0.49	805.7	0.49	791.0	0.50	776.2	0.49	761.2	0.49	746.3
0.083	892.1	0.083	877.5	0.083	863.0	0.083	848.7	0.083	834.3	0.083	819.7	0.083	805.2	0.083	790.6	0.083	775.7	0.083	760.6	0.083	745.7

<sup>a</sup> Values extrapolated to 0.083 MPa are indicated in *italics*.

Table 3. Parameters of the Correlation (eq 1), for the Densities Methyl Linoleate and Methyl Oleate at Ambient Pressure of 0.083 MPa and Temperatures from (270 to 470) K

parameter	methyl linoleate		methyl oleate	
	value	standard deviation	value	standard deviation
$\beta_1/\text{kg}\cdot\text{m}^{-3}$	174.41	0.05	170.56	0.05
$\beta_2$	0.39868	0.00004	0.39673	0.00004
$\beta_3/\text{K}$	767.40	0.03	764.00	0.03
$\beta_4$	0.54592	0.00007	0.54249	0.00007

Figure 1. Deviations of the extrapolated density data of the methyl linoleate of this work and literature data from the correlation (eq 1). Also shown are deviations of the DIPPR<sup>7</sup> and REFPROP<sup>27</sup> correlations.

the density values by up to 0.3 kg·m<sup>-3</sup>. If the reported densities were reduced to reflect this bias, agreement with the majority of the literature data would be even better.

In Figure 1a the DIPPR<sup>7</sup> correlation was derived from all of the data sources shown with the exceptions of Ott et al.,<sup>2</sup> Paschke et al.,<sup>16</sup> and Pratas et al.<sup>19</sup> In the temperature range from (270 to 370) K the DIPPR<sup>7</sup> correlation shows positive deviations from our correlation, while the deviations of the REFPROP<sup>27</sup> correlation (which included the data of Ott et al.<sup>2</sup>) are negative and agree closely with the data of Pratas et al.<sup>19</sup> Much of the data included in the DIPPR<sup>7</sup> correlation have values larger than those



**Table 4.** Parameters of the Correlation (eqs 2 and 3), for the Compressed-Liquid Densities of Methyl Linoleate and Methyl Oleate at Temperatures from (270 to 470) K and Pressures up to 50 MPa

parameter	methyl linoleate		methyl oleate	
	value	standard deviation	value	standard deviation
C	0.08133	0.00009	0.08181	0.00009
D <sub>1</sub>	377.6	0.5	379.7	0.5
D <sub>2</sub>	-316.4	0.5	-323.1	0.5
D <sub>3</sub>	70.01	0.16	72.95	0.16

of Ott et al.,<sup>2</sup> Paschke et al.,<sup>16</sup> and Pratas et al.<sup>19</sup> The deviation of the single data point of Votocek et al.<sup>18</sup> (1.07 %) being so much greater that it is not shown in the figure. The DIPPR<sup>7</sup> and the REFPROP<sup>27</sup> correlations being based on somewhat conflicting data sets thus predict considerably different values for density in the temperature range (278 to 363) K. The larger deviations of both the REFPROP<sup>27</sup> and DIPPR<sup>7</sup> correlations from our data at temperatures above 340 K are expected, as prior to this report, there were no available data to constrain the fits.

The DIPPR<sup>7</sup> correlation in Figure 1b shows a negative deviation from our correlation below approximately 370 K, as does the REFPROP<sup>27</sup> correlation, and there is good agreement between the two database correlations in this temperature range. Our extrapolated densities for methyl oleate are higher in comparison to the majority of the ambient pressure data in the literature, but as mentioned above, are within the combined uncertainty bounds of our data ( $\pm 0.1$  %) and the data of Ott et al.<sup>2</sup> ( $\pm 0.1$  %) which agrees well with much of the literature data.

The compressed-liquid density data were correlated with a Tait equation similar to that of Dymond and Malhotra<sup>28</sup> of the form

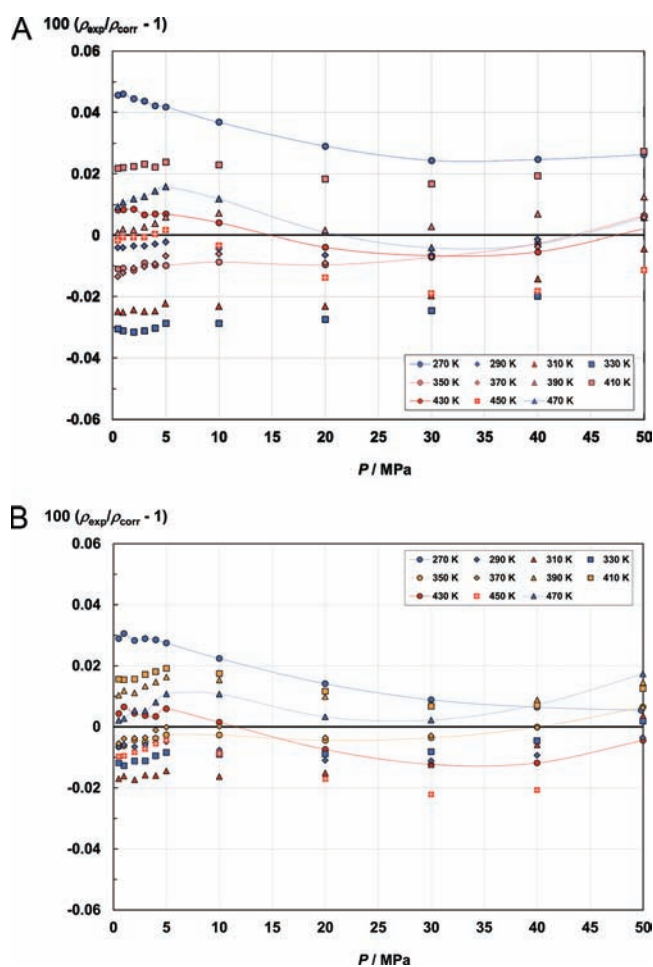
$$\rho(T, p) = \frac{\rho_{\text{ref}}(T, p_{\text{ref}})}{1 - C \ln \left( \frac{p + D(T)}{p_{\text{ref}} + D(T)} \right)} \quad (2)$$

where  $\rho_{\text{ref}}(T)$  is the temperature-dependent density at the reference pressure  $p_{\text{ref}} = 0.083$  MPa from eq 1. The temperature dependence of the parameter  $C$  was omitted because it was not needed to fit the data within their experimental uncertainty. The temperature dependence of the Tait parameter  $D(T)$  was expressed by a quadratic polynomial,

$$D(T) = D_1 + D_2 T_r + D_3 T_r^2 \quad (3)$$

where  $T_r$  is the absolute temperature  $T$  divided by 273.15 K. Parameters for eqs 2 and 3 are given in Table 4. Figure 2a,b shows deviations of the measured compressed-liquid density data relative to the adjusted Tait equation of state. These correlations represent both our methyl linoleate and methyl oleate compressed-liquid density data well within their estimated uncertainty of 0.1 %. One advantage of the Tait equation of state is that it can be reliably extrapolated to pressures considerably higher than those to which it was adjusted.<sup>29</sup> In the case of the samples measured in this work, we expect that extrapolations to pressures of 100 MPa will yield densities within the estimated experimental uncertainty of 0.1 %.

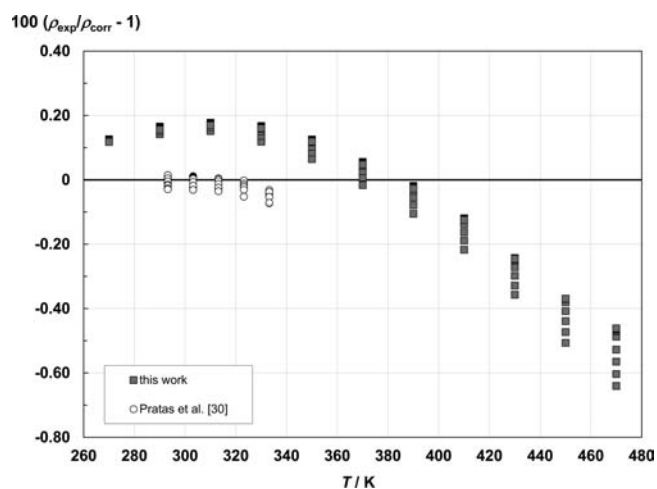
A search of the literature revealed no compressed-liquid density data for methyl linoleate, and only two sets of data for methyl oleate: Bridgman<sup>21</sup> and Pratas et al.<sup>30</sup> The Bridgman<sup>21</sup>



**Figure 2.** Deviations of density data of compressed-liquid methyl linoleate from the correlation (eqs 2 and 3), as a function of pressure.

data, however, are from 1932, and the sample purity may be questionable. There is no statement of purity in the paper, only that the sample “was obtained from Eastman Kodak Co. and used without further purification.” The single ambient pressure value from this reference had by far the largest deviation from our correlation (eq 1) of any of the literature data and as such was not in agreement with the majority of the data. Additionally, all but 2 of the 22 compressed-liquid data points of Bridgman<sup>21</sup> were taken at pressures above 50 MPa, and 17 of them were taken at pressures greater than 100 MPa (the highest pressure at which we would expect our correlation to extrapolate within the experimental uncertainty of 0.1 %). For the above reasons, no comparison of our data to the Bridgman<sup>21</sup> data is provided.

Figure 3 shows deviations of our measured compressed-liquid density data for methyl oleate and that of Pratas et al.<sup>30</sup> to values predicted by REFPROP<sup>27</sup> as a function of temperature. The Pratas et al.<sup>30</sup> data range was in temperature from (293 to 333) K and in pressure from (0.1 to 45) MPa. The agreement between the data reported in this work and that of Pratas et al.<sup>30</sup> is relatively good, but not within the combined uncertainties of the two data sets as our uncertainty is 0.1 % and that of Pratas et al.<sup>30</sup> is  $0.1 \text{ kg} \cdot \text{m}^{-3}$  or approximately 0.01 %. The large deviations of our data from that of the REFPROP<sup>27</sup> predictions at higher temperatures are probably due in large part to the fact that the REFPROP<sup>27</sup> correlation



**Figure 3.** Deviations of density data of compressed-liquid methyl oleate from the REFPROP<sup>27</sup> database as a function of temperature.

was formulated without any compressed-liquid density data and no density data above 338 K.

## 5. CONCLUSIONS

Densities of compressed-liquid methyl linoleate and methyl oleate samples have been measured over a temperature range of (270 to 470) K with pressures to 50 MPa. Densities of the compressed-liquids measured in this work were extrapolated to ambient pressure for comparison with existing literature data and show good agreement. The reported compressed-liquid densities for methyl linoleate provide data in a temperature and pressure range not otherwise available in the literature and those for methyl oleate extend the available data from (333 to 470) K. Correlations formulated for the reported compressed-liquid densities represent the data well within their experimental uncertainty of 0.1 % and provide a predictive tool for engineering applications.

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