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The Dielectric Permittivity of Saturated Liquid Carbon Dioxide and Propane Measured Using Cross Capacitors

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Relative dielectric permittivities ε_r of saturated liquid carbon dioxide (CO_2) and propane (C_3H_8) were measured using well-characterized cross capacitors in the range $260\,K < T < 300\,K$. The molar polarizability $\wp_{sat} \equiv (\varepsilon_r - 1)/[(\varepsilon_r + 2)\rho_{sat}]$ of CO_2 was calculated using the equation of state of Span and Wagner to convert our values of the saturation vapor pressure p_{sat} to values of the saturated liquid density ρ_{sat}. In the range $260\,K < T < 300\,K$, $\wp_{sat} = (7.659 \pm 0.001)$ $cm^3 \cdot mol^{-1}$. The systematic difference between measurements made with two different capacitors was $0.001\,cm^3 \cdot mol^{-1}$; this difference is equivalent to a shift of 12 mK in the liquid temperature. The uncertainty of $\wp_{sat}$ from the equation of state of CO_2 is approximately $0.002\,cm^3 \cdot mol^{-1}$. Our values of $\wp_{sat}$ for CO_2 are consistent with the results of Moriyoshi et al. and of May et al.; however, our values are 0.5% larger than the values determined by Haynes. For saturated liquid propane, our values of ε_r agree with the values of Haynes and Younglove within the combined uncertainty of 0.0003.

KEY WORDS: carbon dioxide; cross capacitors; dielectric constant; permittivity; polarizability; propane; saturated liquid.

1. INTRODUCTION

Measurements of the dielectric properties of natural gas and of gas condensate fluids are becoming increasingly important. For example, Jaeschke et al. [1] have proposed to deduce the heating value of natural gas as it

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is transported in pipelines by measuring several thermophysical properties including the relative dielectric permittivity ε_r. Recently, Harvey and Lemmon [2] developed an improved method of predicting ε_r for natural gas mixtures. In support of these activities, we report new dielectric permittivity measurements of saturated liquid carbon dioxide (CO_2) and propane (C_3H_8) in the temperature range $260\,K < T < 300\,K$. Our CO_2 measurements help resolve the inconsistency between the CO_2 results of Haynes [3] and those of Moriyoshi et al. [4] and of May et al. [5] in favor of the latter groups of authors. For saturated C_3H_8, our values of ε_r differ from the values of Haynes and Younglove [6] by 0.0003 or less, which is within the combined uncertainty of the measurements. Thus, we suspect that Haynes' apparatus (or CO_2 samples) had a particular problem that did not recur when the apparatus was used to study other fluids. Our CO_2 results should also help improve the calibration of the microwave pVT cells developed by May et al. [5] to study the phase behavior of natural gas condensates.

The present measurements use three cross capacitors: (1) the toroidal cross capacitor used by Moldover and Buckley [7] to accurately measure ε_r for He, Ar, N_2, CH_4, and CO_2 (vapor); (2) the 16-rod cross capacitor used by Schmidt and Moldover [8] to extend the Moldover–Buckley measurements to additional isotherms and additional fluids (O_2, C_2H_6, and C_3H_8 vapor) and (3) a multi-ring toroidal cross capacitor that will be described in a future publication. Schmidt and Moldover showed that cross capacitors (1) and (2) yield values of ε_r for argon that differ by no more than 1×10^{-6} on three different isotherms and at pressures up to 6 MPa. Their work established cross capacitors as accurate tools for measuring ε_r in commercially important gases. Here, we demonstrate that cross capacitors can also be used to accurately measure ε_r in liquids.

2. CARBON DIOXIDE

2.1. Equipment and Procedures

The saturated liquid CO_2 ε_r measurements were made using the same ring and rod cross capacitors used by Schmidt and Moldover [8]; details of the capacitance measurements are not repeated here. Initially, the ring cross capacitor was placed in a stirred oil bath, as shown in Fig. 1. The stirred oil bath had a homogeneity and stability (several weeks) better than 0.01 K. The bath temperature was measured with an estimated uncertainty of 0.02 K using a commercially manufactured platinum resistance thermometer. The pressure was measured using a quartz crystal pressure gauge with a full scale of 21 MPa and an estimated uncertainty of 0.3 kPa

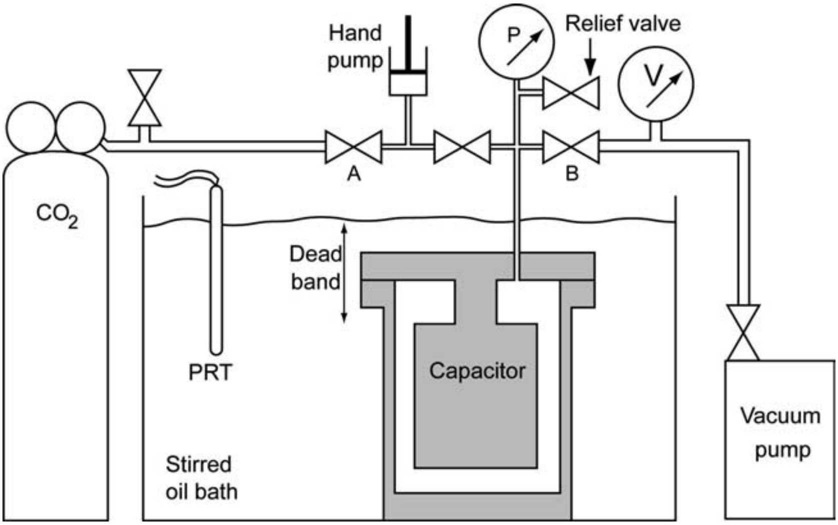

Fig. 1. The experimental system used to measure ε_r in saturated liquid CO_2. Essentially the same system was used for the 16-rod capacitor (shown) and the ring capacitor. When the bath temperature was above room temperature, the lines between valves A and B were maintained above 305 K. Pressure and Vacuum meters are indicated by "P" and "V".

over the limited range 0–7 MPa. Under steady-state conditions, the temperature of the bath, the capacitor, and the pressure vessel housing the capacitor were all assumed to be equal. The CO_2 used in this work came from the same cylinder used by Schmidt and Moldover. The manufacturer claimed its purity was 99.9999% by volume and its water content was $<0.25 \times 10^{-6}$, by mole fraction.

After three liquid CO_2 measurements, an electrical short developed in the ring capacitor; we replaced it with the rod capacitor. Measurements of helium's dielectric permittivity were conducted before and, in the case of the rod capacitor, after the liquid CO_2 measurements. The differences between these helium measurements and those reported by Schmidt and Moldover were smaller than the noise floor of the present system. (The temperature and pressure uncertainties of the current system are larger than those uncertainties in the system used by Schmidt and Moldover.) Halfway through the rod capacitor measurements of liquid CO_2, another electrical short developed. The rod capacitor was disassembled, and the electrical short was repaired. Before continuing with the CO_2 measurements, the rod capacitor was re-calibrated with helium. The rod capacitor was found to have a new effective compressibility of

$(5.9 \pm 0.1) \times 10^{-12}\,\mathrm{Pa}^{-1}$, which contrasts with the value $6.9 \times 10^{-12}\,\mathrm{Pa}^{-1}$ reported by Schmidt and Moldover. We attribute this difference to a slight change in the location of, and stresses on, the shield enclosing the rod capacitor.

The pressure vessel containing the capacitor was filled with saturated liquid CO_2 on three separate occasions: once for the ring capacitor and twice for the rod capacitor. In all three cases, the system was cooled to below room temperature and the pressure vessel was evacuated. The measured vacuum capacitance at this temperature was recorded and, after flushing several times, the system was pressurized with CO_2 vapor to just below p_{sat}. Once thermal equilibrium was re-established, all the valves between the CO_2 cylinder and the pressure vessel were opened and this resulted in a rapid increase in the measured capacitance. After several minutes the measured capacitance would stop increasing significantly and the valves were then closed. The system was then allowed to thermally equilibrate.

Usually, the filling procedure resulted in a liquid CO_2 level slightly above the oil level in the stirred bath. The liquid level in the pressure vessel was inferred from the measured pressure and capacitance; the pressure was used to determine if the liquid level was too high and the capacitance was used to determine if it was too low. (Capacitance measurements could also be used to infer that the liquid level was too high, but the pressure gauge was the primary indicator. Further detail is provided in the following paragraphs.) As indicated in Fig. 1, an 8 cm high "dead band" existed near the top of the pressure vessel. When the liquid–vapor interface was within the dead band, changes in the liquid level did not cause detectable changes in either the pressure or capacitance at steady-state. Furthermore, when the liquid–vapor interface was within the dead band, the measured values of T_{sat} and p_{sat} were consistent with each other and with the CO_2 equation of state (EOS) of Span and Wagner [9]. The values of $(p_{\mathrm{sat}}, \varepsilon_{\mathrm{r}})$ for saturated liquid CO_2 measured with the two cross capacitors are listed in Table I; each $(p_{\mathrm{sat}}, \varepsilon_{\mathrm{r}})$ is an average of at least three measurements with liquid levels within the system's dead band. We report our measured p_{sat} rather than T_{sat} because it has a similar relative uncertainty, and because the measured pressure was that of the CO_2 sample, whereas the temperature was that of one point within the stirred oil bath.

The volume of the pressure vessel was about $1000\,\mathrm{cm}^3$. The quantity of the CO_2 in the pressure vessel could be changed using a hand pump with a $60\,\mathrm{cm}^3$ stroke. When the pump reached the end of its stroke, it was isolated from the pressure vessel and either vented or re-pressurized. In this way the overall sample density, and thus the CO_2 liquid level, could be varied in a controlled fashion.

Table I. Relative Dielectric Permittivities of Saturated Liquid CO_2

Capacitor	p_{sat}(MPa)	ε_r
Rod	3.0468	1.59874
Rod	3.9704	1.55419
Ring	4.4312	1.53222
Ring	4.5502	1.52652
Rod	5.4101	1.48379
Ring	5.4165	1.48338
Rod	5.4509	1.48166
Rod	6.0733	1.44670
Rod	6.5755	1.41306

The estimated uncertainty in p_{sat} is $\pm 3 \times 10^{-4}$ MPa. The reproducibility of each ε_r measurement was $\pm 1 \times 10^{-5}$, except for the highest pressure measurement for which it was $\pm 4 \times 10^{-5}$.

Figure 2 displays the time dependence of the pressure and of the rod capacitance. In this case, the liquid level was initially too high; then, it was lowered into the system's dead band. For this data, the bath temperature was 290.72 K, for which the EOS gives $p_{sat} = 5410$ kPa. The initial pressure was 5430 kPa, which corresponds to a value of T_{sat} approximately 0.15 K higher than the bath temperature. Presumably a thermal gradient existed along the fill-lines between the temperature of the room and that of the bath. Thus, the top of the system's dead band corresponds approximately to the height of the oil level in the stirred bath.

Figure 2 shows that when CO_2 was removed from the pressure vessel, the pressure decreased to 5410 kPa, a value that is consistent with the bath temperature. At the same time, the capacitance dropped because the dielectric permittivity of the liquid decreased from that of a slightly compressed liquid at 290.72 K to that of the saturated liquid. Subsequently, (See Fig. 2.) two smaller aliquots of CO_2 were removed from the pressure vessel; in both cases the pressure and the capacitance returned to the equilibrium values. Thus, the final three plateaus in the data shown in Fig. 2 correspond to CO_2 liquid levels within the system's dead band.

Figure 3 displays the time dependence of the pressure and of the ring capacitance as three aliquots of CO_2 were withdrawn from the pressure vessel. Initially, the liquid level was within the system's dead band. Following the first two withdrawals, the pressure returned to 4431 kPa; this pressure was consistent with the measured bath temperature of 282.51 K. The

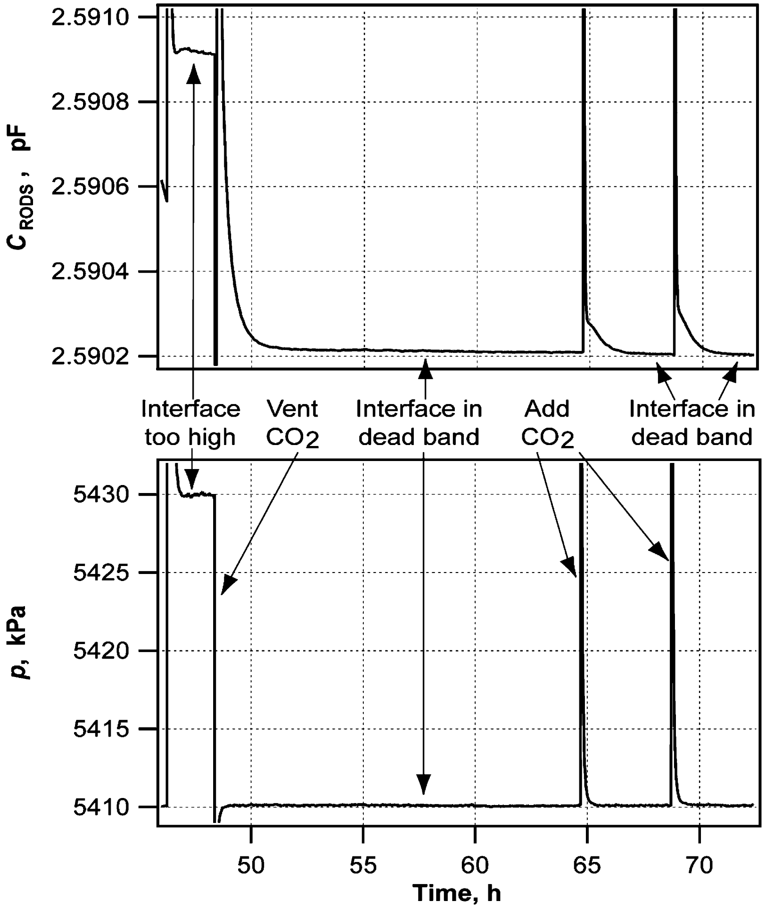

Fig. 2. Time dependence of the pressure and capacitance (rods) near 5.410 MPa. The pressure vessel had been overfilled with CO_2 so that the liquid–vapor interface was in the fill-lines above the surface of the oil bath, as indicated by the pressure plateau at 5.430 MPa. At 48 h, CO_2 was vented from the pressure vessel lowering the interface below the surface of the oil bath. The pressure settled to a plateau at 5.410 MPa that is consistent with the bath temperature (290.72 K). Small additions of CO_2 at 65 and 69 h did not change the steady-state capacitance or the pressure; this indicates that the interface remained in the dead band.

third withdrawal significantly reduced the measured capacitance because the liquid level fell below the capacitor's top electrode, the height that defines the bottom of the dead band. Once the liquid level fell below the capacitor's top electrode, the capacitance exhibited a long time constant.

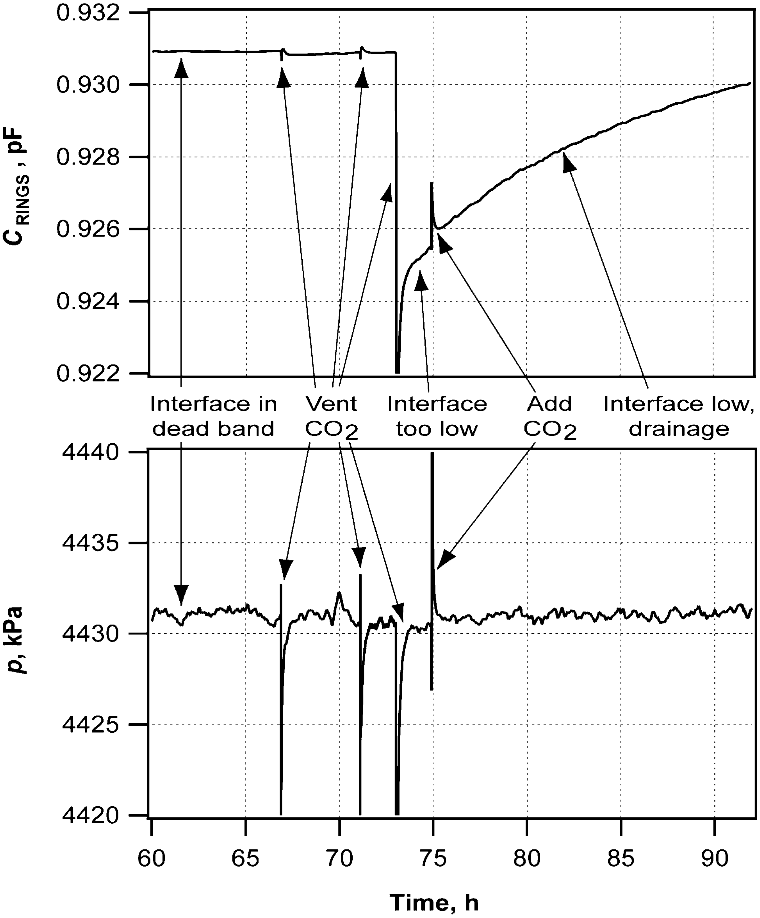

Fig. 3. Time dependence of pressure and capacitance (ring) near 4.431 MPa. The insensitivity of the measured capacitance and pressure to the venting of small amounts of CO_2 at 67 and 71 h indicates that the liquid–vapor interface remained in the dead band. Following the venting at 73 h, the level dropped below the capacitor's top electrode as indicated by the decrease of the capacitance.

This may be the result of thin CO_2 films draining from the walls of the pressure vessel and slowly increasing the liquid level in the capacitor.

Once the measurements at a given temperature were completed, the bath temperature was changed and the system was given at least 12 h to equilibrate. When the new temperature was below the previous one, the liquid level fell and, if necessary, more CO_2 was transferred into the pressure vessel. Conversely, when the new temperature was higher than the previous one, the liquid level increased and, if necessary, CO_2 was vented. A spring-loaded pressure relief valve was connected to the pressure vessel in case thermal control of the bath was accidentally lost. For the two measurements made at temperatures above ambient, the fill-lines between valves A and B in Fig. 1 were maintained above 305 K. (The critical temperature of CO_2 is 304.13 K [10])

2.2. Discussion

We discuss our CO_2results in terms of the molar polarizability $\wp$ that is determined from the molar density ρ and ε_r through

$$\wp = \left(\frac{\varepsilon_r - 1}{\varepsilon_r + 2}\right)\frac{1}{\rho}. \tag{1}$$

For carbon dioxide, the EOS of Span and Wagner [9], as implemented in the software package REFPROP 7.0 [11], was used to compute ρ_{sat} from our measurements of p_{sat}. This EOS has a claimed density uncertainty of 0.03%. When we compared our results to those of Moriyoshi et al. [4] and of May et al. [5], we used this same EOS to determine the saturated liquid density from either the reported saturated temperature T_{sat} or pressure p_{sat}. Consequently, the uncertainty of the EOS does not enter into these comparisons.

Figure 4 compares our results for the polarizability of saturated liquid CO_2 ($\wp_{sat}$) with results from the literature. Moriyoshi et al. [4] report values of $\varepsilon_r(T_{sat}, p_{sat})$; however their values of T_{sat} and p_{sat} are inconsistent, within the claimed uncertainties of their temperature and pressure measurements and the claimed uncertainty of the EOS. We therefore deduced two sets of $\wp_{sat}$ from their data. One set (plotted as upright triangles) are values of $\wp_{sat}(T_{sat})$; the second set (plotted as inverted triangles) are values of $\wp_{sat}(p_{sat})$. Below 300 K, the differences between the two sets of $\wp_{sat}$ are of order $0.01\,cm^3 \cdot mol^{-1}$ and these sets overlap our data within this uncertainty. Near the critical temperature of CO_2, the inconsistency in the values of (T_{sat}, p_{sat}) reported by Moriyoshi et al. becomes very large, corresponding to more than $0.1\,cm^3 \cdot mol^{-1}$ in $\wp_{sat}$.

May et al. [5] used a microwave pVT cell to measure permittivities, dew points, and liquid volume fractions in hydrocarbon mixtures; they conducted a calibration of their cell with saturated liquid CO_2. During this calibration, a temperature difference of 0.2 K was intentionally maintained between the top and bottom of their cell. Figure 4 displays their single result for saturated liquid CO_2 as a diamond with an error bar indicating the uncertainty resulting from the temperature gradient.

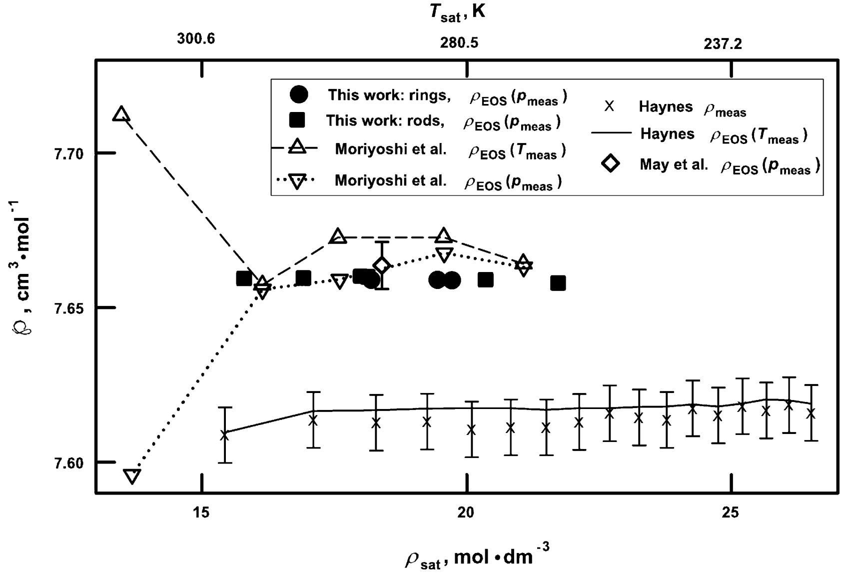

Fig. 4. The molar polarizability $\wp_{\rm sat}$ of saturated liquid carbon dioxide. The data shown as crosses with error bars are calculated from Haynes' [3] simultaneous measurements of $\rho_{\rm sat}$ and $\varepsilon_{\rm r}$. The other values of $\wp_{\rm sat}$ were calculated from the reported values of $\varepsilon_{\rm r}$ and the values of $\rho_{\rm sat}$ determined using the EOS from Ref. [9] and the reported values of either $T_{\rm sat}$ or $p_{\rm sat}$. The lower horizontal axis is the saturated liquid density $\rho_{\rm sat}$; the upper horizontal axis is the corresponding saturation temperature $T_{\rm sat}$. For clarity, the data of Moriyoshi et al. [4] are connected by broken lines.

Remarkably, the measurement by May et al. made at 460 MHz agrees with the present results made at 2 kHz, within their uncertainty.

In Fig. 4, the polarizability determined from Haynes' [3] simultaneous measurements of $\rho_{\rm sat}$ and $\varepsilon_{\rm r}$ are plotted as crosses with error bars that are based on the stated uncertainty of the two measurements. The solid line in Fig. 4 shows the polarizabilities determined from Haynes' values of $T_{\rm sat}$ and $\varepsilon_{\rm r}$. (Haynes does not report saturation pressures.) The two sets of polarizabilities determined from Haynes' $\varepsilon_{\rm r}$ measurements are consistent with each other; the EOS densities smooth the scatter in the polarizabilities derived from the measured densities. However, Haynes results for $\wp_{\rm sat}$ are consistently 0.5% smaller than the other results.

Figure 5 expands the polarizability scale of Fig. 4 by a factor of 30 in order to display the small difference between the $\wp_{\rm sat}$ values that we determined using the ring and rod capacitors. The error bars indicate the standard deviation of the three or more measurements made at each $p_{\rm sat}$ with the liquid level within the system's dead band. The differences between the

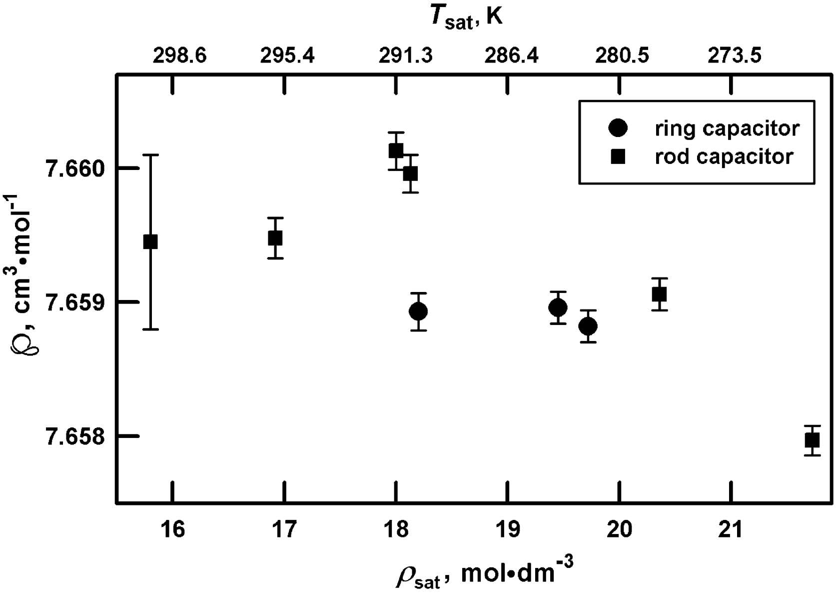

Fig. 5. The molar polarizability of saturated liquid CO_2 determined in this work using the ring cross capacitor and the rod cross capacitor. These capacitors are described in Refs. 7 and 8.

measurements made at a fixed pressure were caused by small changes in the bath temperature; these differences are much larger than the precision with which a capacitance ratio could be measured. The reproducibility of the ε_r measurements was better than $\pm 1 \times 10^{-5}$ for all but the highest temperature data point, for which it was $\pm 4 \times 10^{-5}$.

The largest difference between the two data sets occurs at 290.75 K (5.41 MPa) and is the result of a difference of approximately 0.0001 in the values of ε_r measured with the two capacitors. We believe that the uncertainties resulting from the imperfections of the instruments and any impurities introduced in handling the CO_2 are too small to account for this difference. This difference in ε_r corresponds to a fractional difference in density of 2×10^{-4}. Along the saturation curve, such a density difference would correspond to a pressure change of at least 2000 Pa, which is seven times larger than the estimated pressure uncertainty. However, the density difference can be accounted for by small changes (non-uniformity) in temperature and pressure that move the fluid's state off the saturation curve and into the compressed liquid region. For example, the volumetric thermal expansion coefficient of the saturated liquid is 0.0165 K^{-1} at 290.75 K [11]. Then, if the temperature at the interface were higher than the temperature of the bulk of the CO_2 in the capacitor by only 12 mK, the density of the CO_2 would be, fractionally, 2×10^{-4} higher than the density deduced from the measured vapor pressure. The inhomogeneity of the bath was known to be approximately 10 mK; thus, such a temperature difference is conceivable, particularly if the liquid level were near the top of the system's dead band.

Furthermore, if the effects of the pressure head in the rod capacitor due to the liquid height are considered, the required temperature change is even smaller. The rod capacitor was approximately 10 cm high, and the top of the dead band was about 8 cm above the top of the rod capacitor; thus, the pressure at the bottom of the rod capacitor could have been as much as 1400 Pa greater than the measured vapor pressure. The isothermal compressibility of saturated liquid CO_2 at 290.75 K is $3.6 \times 10^{-8}\,\mathrm{Pa}^{-1}$ [11] and, therefore, the fractional density increase due to the pressure head could be as much as 5×10^{-5}.

The ring capacitor was approximately 2.5 cm high and was housed in a slightly shorter pressure vessel than the vessel used for the rods. Thus, the ring capacitor was less susceptible than the rod capacitor to the systematic effects described above, and the ε_r values measured with the ring capacitor are likely to be the more accurate. Both of the cross capacitors used in this work were optimized for measurements of gases, and not for measurements of saturated liquids. Nevertheless, the accuracy of the liquid CO_2 ε_r data reported here is equivalent to or better than liquid ε_r data measured by other workers with capacitances 50–100 times larger. The results of these cross-capacitor measurements have reduced the uncertainty in the polarizability of saturated liquid CO_2 by an order of magnitude, to a level that is more than adequate for practical purposes.

3. PROPANE

The saturated liquid propane measurements were made eight months after the final CO_2 measurement. During that interval, the temperature and pressure measurement systems were improved: the stirred oil bath was replaced with a stirred ethanol bath with a long-term stability better than 1 mK and a homogeneity better than 3 mK, and the bath temperature was measured with two long-stemmed standard platinum resistance thermo meters. These improvements reduced the uncertainty of the sample temperature to 0.005 K. The pressure gauge was re-calibrated between 0 and 7 MPa with a standard uncertainty of 200 Pa.

The rod cross capacitor was replaced with a new cross capacitor composed of three ring capacitors, stacked vertically and connected electrically in parallel. This new capacitor, which will be described in a future publi-

Table II. Measurements of (T, p, ε_r) for Vapor-Phase and Saturated-Liquid Propane

T (K)	p (MPa)	ε_r	Phase
283.359	0.1989	1.004219	Vapor
283.359	0.4096	1.009128	Vapor
283.359	0.6399	1.015186	Vapor
260.357	0.3145	1.74180	Sat. Liquid
266.419	0.3843	1.72879	Sat. Liquid
272.895	0.4709	1.71466	Sat. Liquid
278.265	0.5531	1.70257	Sat. Liquid
283.360	0.6405	1.69090	Sat. Liquid
288.316	0.7350	1.67949	Sat. Liquid
288.317	0.7349	1.67933	Sat. Liquid
293.209	0.8378	1.66773	Sat. Liquid

The estimated uncertainties in T and p are ± 0.005 K and $\pm 2 \times 10^{-4}$ MPa, respectively. The estimated uncertainty in ε_r is $\pm 4 \times 10^{-6}$ for the vapor measurements, and $\pm 5 \times 10^{-5}$ for the liquid measurements. We suspect that the ε_r value at 288.316 K is an outlier.

cation, has a height similar to that of the 16-rod capacitor and a vacuum capacitance of about 1.5 pF. With the improvements in temperature and pressure measurement, the accuracy of vapor phase ε_r measurements was comparable to those of Schmidt and Moldover [8]; the estimated uncertainty in vapor ε_r determinations is 4×10^{-6}, which is due almost entirely to the uncertainty in the measured pressure. However, when filled with liquid, this larger and more intricate cross capacitor exhibited a long time constant and a small drift over several days, which limited the reproducibility of the liquid phase ε_r measurements to 5×10^{-5}. This observation, together with the relative performance of the two capacitors used for the CO_2 measurements, suggests that smaller, simpler cross capacitors are better suited for liquid ε_r measurements.

The values of (T, p, ε_r) measured in this work for both vapor and saturated liquid propane are listed in Table II. We used propane from the same cylinder used by Schmidt and Moldover. The three propane vapor measurements of ε_r agree with the values predicted using the correlation of Harvey and Lemmon [2], within the estimated uncertainty of the measurements; of the three vapor data, only the lowest pressure ε_r measurement deviates by more than 1.5×10^{-6} from the correlation. This is not surprising because Harvey and Lemmon's correlation for propane vapor is based primarily on the ε_r data of Schmidt and Moldover.

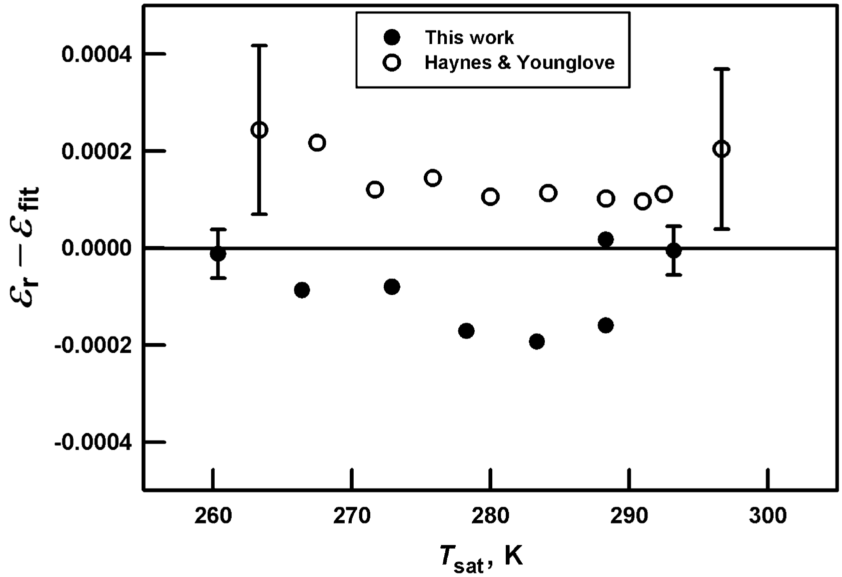

Fig. 6. Deviations ($\varepsilon_r - \varepsilon_{fit}$) of the saturated liquid propane ε_r measurements made in this work, and by Haynes and Younglove [6], from an empirical dielectric permittivity ε_{fit} calculated using Eqs. (2) and (3). We suspect that the larger of the two ε_r measurements at 288.32 K made in this work is an outlier. The present values of ε_r differ from those of Haynes and Younglove by an amount comparable to their estimated uncertainty.

The values of T_{sat} and p_{sat} for propane listed in Table II are consistent with each other and with the C_3H_8 equation of state of Bücker and Wagner [12]. To compare our saturated liquid propane ε_r measurements with those of Haynes and Younglove [6], we first converted the temperatures they reported in the range 260–300 K from IPTS-68 to ITS-90. Then, at each of these temperatures and each of the temperatures listed in Table II, the quantity ε_{fit} was calculated using

$$\left(\frac{\varepsilon_{fit} - 1}{\varepsilon_{fit} + 2}\right) = \rho_{sat}\,\wp_{sat}, \tag{2}$$

where ρ_{sat} was calculated from T_{sat} using the EOS of Bücker and Wagner [12], and $\wp_{sat}$ was calculated from the empirical expression

$$\frac{\wp_{sat}}{\mathrm{cm}^3 \cdot \mathrm{mol}^{-1}} = 16.2788 - 65.5\left(\frac{T_{sat}}{\mathrm{K}}\right)^{-1}. \tag{3}$$

The deviations ($\varepsilon_r - \varepsilon_{fit}$) for our measurements and for those of Haynes and Younglove [6] are shown in Fig. 6. Haynes and Younglove estimated that the fractional uncertainty of their data was $0.0001\varepsilon_r$, which for this range of ε_r values is about 0.0002. The error bars on our data denote the reproducibility of the liquid ε_r measurements as the temperature of the cross capacitor was cycled between 260 and 293 K. With the exception of the data at 288.32 K, the ε_r values at each temperature were all reproduced within $\pm 5 \times 10^{-5}$. We do not have a reason why the two ε_r data at 288.32 K differ by 1.7×10^{-4}; however, based on the deviations $(\varepsilon_r - \varepsilon_{\text{fit}})$ shown in Fig. 6, it appears that the larger of the two values $(\varepsilon_r = 1.67949$ at 288.316 K) is an outlier.

The present values of ε_r for saturated liquid propane agree with those of Haynes and Younglove within the combined uncertainty. This agreement implies that the apparatus (or CO_2 samples) used by Haynes had a particular problem that was not present when the apparatus was used to study propane and other fluids.

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