



## **( $p, \rho, T$ ) of liquid $n$ -octane obtained with a spherical pycnometer at temperatures of 298.03 K and 313.15 K and pressures in the range 0.7 MPa to 32 MPa**

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The density  $\rho$  of  $n$ -octane at temperatures of 298.03 K and 313.15 K and at pressures between 0.7 MPa and 32 MPa have been obtained with a pycnometer. The results, which we estimate have a best-case precision of  $\pm 7 \cdot 10^{-5} \cdot \rho$ , are compared with values reported in the literature where the differences are less than  $\pm 4 \cdot 10^{-4} \cdot \rho$ . At each temperature and pressure the cell volume  $V_c$  was obtained from the mass of mercury required to fill it at  $T = 293.12$  K and  $p = 0.813$  MPa and from the literature values of the mechanical properties of Inconel 625 from which the cell was formed. The volume was also obtained with a mercury-filled positive displacement pump at three temperatures between 298.15 K and 333.15 K and pressure below 30 MPa. The volumes so determined differed by less than  $6 \cdot 10^{-5} \cdot V_c$  from the calculated values. © 1996 Academic Press Limited

### **1. Introduction**

Recently,<sup>(1,2)</sup> we published densities of five complex and compositionally characterized fluids obtained with a “cylindrical” pycnometer. Here we present measurements of mass density for  $n$ -octane obtained with a spherical pycnometer. This geometry was chosen because we can, with linear thermal expansion and elastic constants of the cell wall material taken from the literature, more easily calculate the internal cell volume  $V_c$  at a given temperature and pressure from one gravimetrically determined value of  $V_c(T, p)$ . In our previous work,<sup>(1,2)</sup> the mercury was expelled from a positive-displacement pump situated at ambient temperature into a cell located in a thermostat; the pycnometer and pump were separated by a valve. During these previous experiments the mass of mercury injected into the cell was compared with that determined gravimetrically and, usually, the value obtained by weighing was greater than that calculated from pump displacements. In particular, significant differences in mass were observed when the laboratory temperature increased during the course of the measurements, and we suspect that mercury migrated into the cell in the time required to measure the pressure.<sup>(1,2)</sup> Along a single isotherm these mass

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differences never introduced an error in mass density of more than  $6 \cdot 10^{-4} \cdot \rho^{(2)}$  and was typically  $2 \cdot 10^{-4} \cdot \rho$ . To reduce the effect of this systematic error, the cell was weighed after each isotherm. For the measurements reported here we inserted the positive-displacement pump into the stirred fluid bath. This modification should reduce the effect of undesirable mercury migration and eliminate the need to weigh the cell between isotherms.

The measurements have been analyzed to determine mass density  $\rho$  in terms of the equation:

$$\rho(T,p) = m/V_s(T,p) = m/\{V_c(T,p) - V_{\text{Hg}}(T,p)\}, \quad (1)$$

where  $V_c$  is the volume of the cell,  $V_s$  the volume occupied by the fluid,  $m$  the mass of fluid, and  $V_{\text{Hg}}$  the cell volume occupied by mercury, which was determined from a positive-displacement pump.

## 2. Apparatus and experimental

The apparatus is shown in figure 2. The experimental procedures have been described in detail elsewhere,<sup>(1)</sup> and only the important differences from our earlier work are given here. The spherical pycnometer was constructed, by the National Engineering Laboratory, U.K., as two hemispheres from a single cylindrical bar of Inconel 625. Before turning commenced, a port was machined into the pole of each hemisphere, each with a conical seat to accept a high-pressure fitting. Tooling marks on the inner surface were reduced by mechanical polishing. The completed hemispheres were joined by a weld that completely penetrated the wall leaving no gap at the intersection of the two hemispheres for fluid entrainment. Visual inspection of the interior surface showed that the procedure had been successful and that the visible weld was smooth and pit free. The external surface was machined to produce a uniform wall thickness of 15.49 mm. Mercury dilatometry measurement at  $T = 293$  K returned a volume of  $(222.608 \pm 0.005) \text{ cm}^3$ .<sup>(3)</sup>

The valve arrangement, shown in figure 1, ensured that mercury injected into the cell was always at the same temperature as the bath. Figure 1 also shows the  $100 \text{ cm}^3$  positive-displacement pump mounted inside the stirred fluid bath; we did this to reduce the effects of mercury migration discussed above and elsewhere.<sup>(1,2)</sup> The effective area of the positive displacement pump piston was determined by auxiliary experiments at a pressure of 13.789 MPa. The linear displacement  $l$  of the piston was obtained with a precision of  $0.001 \cdot l$ , from a scale mounted on the pump. For these measurements we used only the middle third of the pump displacement.

The cell was suspended in a stirred-fluid thermostat, controlled to better than  $3 \cdot 10^{-3} \text{ K}$ . The temperature of the thermostat was measured with a long-stem platinum resistance thermometer calibrated on ITS-90. The sample temperature was inferred from the thermostat value. For hydrocarbon density measurements, where the expansivity  $\alpha \approx 1 \cdot 10^{-3} \text{ K}^{-1}$ , to obtain a fractional precision in  $\rho$  of  $1 \cdot 10^{-5}$  implies a precision of 0.01 K in temperature; for cell volume calibration with mercury, for which  $\alpha \approx 2 \cdot 10^{-4} \text{ K}^{-1}$ , only 0.2 K precision in temperature is required. In view of the excellent thermostat stability and the accuracy of our thermometry, we conclude that

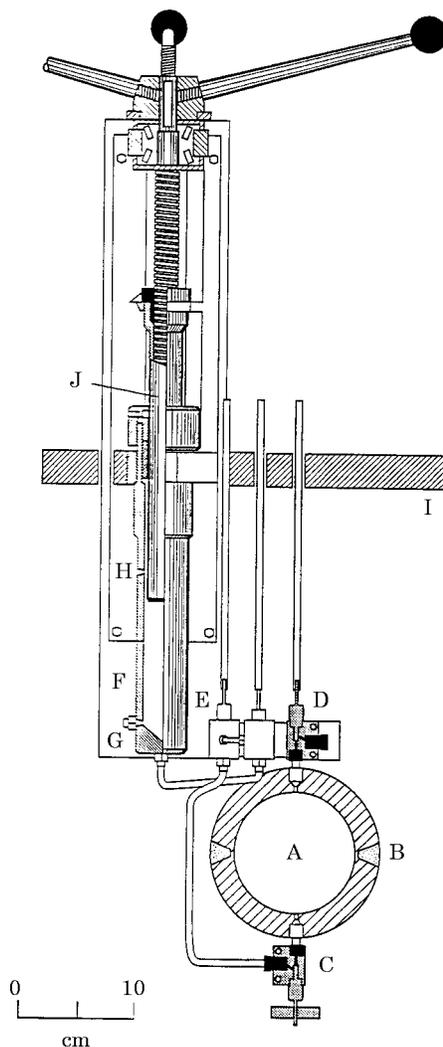


FIGURE 1. Cross-section through pycnometer apparatus. Spherical cell **A** with equatorial weld **B** fitted with valves **C** and **D**. **E** is the mercury control valve. The positive-displacement pump **H** with piston **J** was supported vertically on a plate **F**. The mercury entered the pump at port **G** from a reservoir at room temperature. The apparatus below **I** was suspended in a stirred-fluid thermostat. A long-stem resistance thermometer, with its platinum sensing element located at **B**, was inserted into the bath fluid.

inferring sample temperature from thermostat fluid value should not have introduced a significant systematic error into our measurements.

Pressures were measured with a precision of 3.5 kPa using a strain-gauge transducer which, when calibrated against an oil-lubricated piston gauge, was found to be linear to 4.5 kPa; for this calibration, as for the measurements, the gauge was filled with mercury, which in turn was separated from the piston assembly by a differential capacitance transducer, operated as a null detector. This transducer was

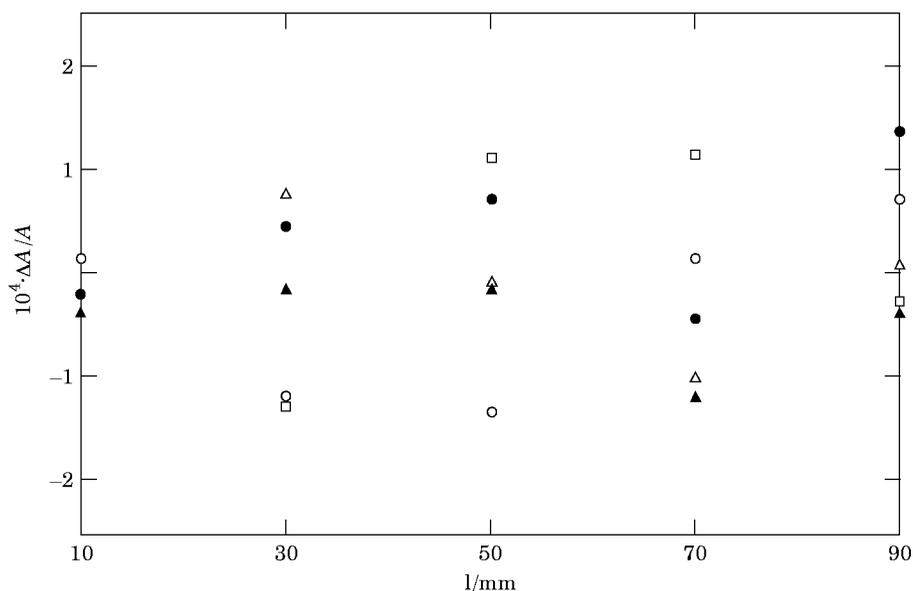


FIGURE 2. Fractional deviations  $\Delta A/A = \{A - A(\text{calc.})\}/A$  for the effective pump area  $A$  at a pressure of 13.978 MPa from equation (2). ●, 293.15 K; ○, 298.027 K; △, 313.15 K; ▲, 313.15 K; □, 333.15 K.

located above the plate (I, shown in figure 1). Hydrostatic-head corrections were applied so that the quoted pressures refer to the plane that passed through the mid-point of the sample within the cell. The maximum head correction was 54 kPa for the mercury column between the pressure gauge and cell. Since for hydrocarbons the isothermal compressibility  $\kappa_T \approx 1 \cdot 10^{-3} \text{ MPa}^{-1}$ , to achieve a fractional imprecision in  $\rho$  of  $1 \cdot 10^{-5}$  implies a precision in pressure of 10 kPa. Clearly, the gauge was adequate for our purpose.

Although a nominal volume scale was provided with the pump, it was necessary to determine the effective piston area. The procedures used for this calibration were similar to those described elsewhere.<sup>(1)</sup> Here we arbitrarily chose a reference pressure of 13.789 MPa for all displacement measurements, thereby eliminating the dilation of the pump cylinder from the calibration. Because the pump was mounted within the thermostat we calibrated the positive-displacement pump at each temperature before and after the density measurements.

The *n*-octane was *puriss* grade material supplied by Fluka Ltd. with a stated mole fraction purity of not less than 0.995. Information provided by the manufacturer indicated that the major impurity was water with mole fraction of about 0.0002. Before use, the sample was degassed and dried over grade 0.4 nm molecular sieves that had been baked previously at  $T = 500 \text{ K}$  under vacuum. No analysis or further purification was attempted.

The mercury was an aliquot of the sample Moldover *et al.*<sup>(4)</sup> used in their acoustic re-determination of the gas constant and is traceable to Cook;<sup>(5)</sup> it came from the NIST stock described in reference 6.

For all our weighings we used a double-pan swing balance with a resolution and accuracy of 1 mg. All apparent masses were corrected to mass.

### 3. Results and analysis

The temperature dependence of the density of pure mercury, required in the calculations, was obtained from Ambrose.<sup>(7)</sup> Since the compressibility of mercury is small ( $\kappa_T \approx 10^{-11} \text{ Pa}^{-1}$ ), we do not place severe demands on our limited knowledge of  $\kappa_T$ , and we assumed that the compressibility was linear.<sup>(4)</sup> In our experimental regime, densities of liquid mercury calculated from the chosen expression agreed to better than  $0.00007 \cdot \rho$  with correlations reported by others<sup>(8)</sup> and do not introduce a significant systematic error.

The effective area  $A$  of the pump was determined at each of five temperatures at a pressure of 13.789 MPa. Not surprisingly,  $A$  was found to depend on the piston position  $l$ . We chose a purely empirical expression to represent  $A(T, l)$  and, with an adaptive regression algorithm, found:

$$\begin{aligned} A(T, l)/\text{cm}^2 = & 0.9993_3 + 5.50_5 \cdot 10^{-5} \{ (T/\text{K}) - 273.15 \} - \\ & 9.84_2 \cdot 10^{-9} (l/\text{mm}) \{ (T/\text{K}) - 273.15 \}^2 + \\ & 7.88_4 \cdot 10^{-11} [(l/\text{mm}) \{ (T/\text{K}) - 273.15 \}]^2, \end{aligned} \quad (2)$$

with a standard deviation of  $9.7 \cdot 10^{-5}$  for 24 measurements. The volume of mercury displaced from the pump was then determined with:

$$V_{\text{Hg}}(T) = \int_{l_1}^{l_2} A(T, l) \cdot dl. \quad (3)$$

The effective area of the piston is shown in figure 2 as deviations from equation (2). These differences are always less than  $\pm 1 \cdot 10^{-4}$  and limit our ultimate precision to  $2.2 \cdot 10^{-5} \cdot \rho$  for *n*-octane. Each density determination requires two displacement pump measurements, each with a resolution of  $\approx 0.001 \text{ cm}^3$  contributing about  $2.5 \cdot 10^{-5} \cdot \rho$  to the uncertainty.

The cell volume was determined gravimetrically at  $T = 293.12 \text{ K}$  (equivalent to  $T = 298.15 \text{ K}$  on IPTS-48) and a pressure of 0.813 MPa. Assuming the mercury solely occupied the internal volume, we obtain  $V_c = 222.748 \text{ cm}^3$ . This value is  $0.14 \text{ cm}^3$  above the value determined previously.<sup>(3)</sup> We suspect that this difference arose due to the change in valves and connections between these two measurements. Changes in  $V_c$  arising from variations in temperature and pressure from the reference state, defined above, were calculated with expressions for a perfectly spherical shell from reference 9, where the wall compliance was determined with a pressure of 0.1 MPa exerted on the outer wall. For this calculation, we required values of Young's modulus  $E$ , Poisson's ratio  $\sigma$ , and the linear thermal expansion  $\alpha$ . The fractional change in volume arising from a pressure difference of 27 MPa across the wall was about  $-5 \cdot 10^{-4}$ . The value of  $E$  was obtained, at each temperature, from a literature correlation for Inconel 625,<sup>(10)</sup> while  $\sigma$  was assumed to be 0.278 and independent of

temperature.<sup>(11)</sup> The value of  $\alpha$  for Inconel 625 was obtained from reference 12. To check the validity of these calculations of  $V_c(T,p)$ , we also determined the cell volume with the positive-displacement pump along three isotherms at temperatures between 293.15 K and 333.15 K at pressures in the range 0.77 MPa to 27.8 MPa. The differences between the calculated and measured volumes, shown in figure 3, were never more than  $0.00015 \cdot V_c$ , and were less than  $0.00006 \cdot V_c$  for  $T < 313$  K. During the calibration experiments with the positive-displacement pump we obtained a volume  $V_c(293.12 \text{ K}, 0.813 \text{ MPa}) = 222.762 \text{ cm}^3$  which is  $6 \cdot 10^{-5} \cdot V_c$  above the gravimetrically determined value. After completing these volume calibration measurements, the mass of mercury injected into the cell from pump displacements was found to be 0.170 g below that determined by weighing; this difference corresponds to an uncertainty in volume of  $0.013 \text{ cm}^3$ . The cell was then cleaned, evacuated, and reweighed. Comparison of this cell mass with that determined prior to calibration revealed a 0.8095 g increase which would amount to an uncertainty in volume of  $0.06 \text{ cm}^3$ . We assumed that this mass was solely attributable to mercury which, we suspected, had been trapped in the valve seats and threads, and this value was then added to the mercury inventory for the *n*-octane measurements; had we not taken this mass into account, it would have introduced a systematic error of about  $3 \cdot 10^{-4} \cdot \rho$  in our *n*-octane density measurements. At the conclusion of the experiments, we removed the valves and interconnecting parts from the cell and found mercury beads deposited in the threads with a combined mass of 0.8 g, which supports our earlier assumption. The final discrepancy in the  $m(\text{Hg})$  inventory of 0.2 g corresponds to an uncertainty in volume of  $0.015 \text{ cm}^3$  at about  $6 \cdot 10^{-5} \cdot \rho$ .

Values of  $\rho$  for *n*-octane, listed in table 1, were obtained from the volume that a

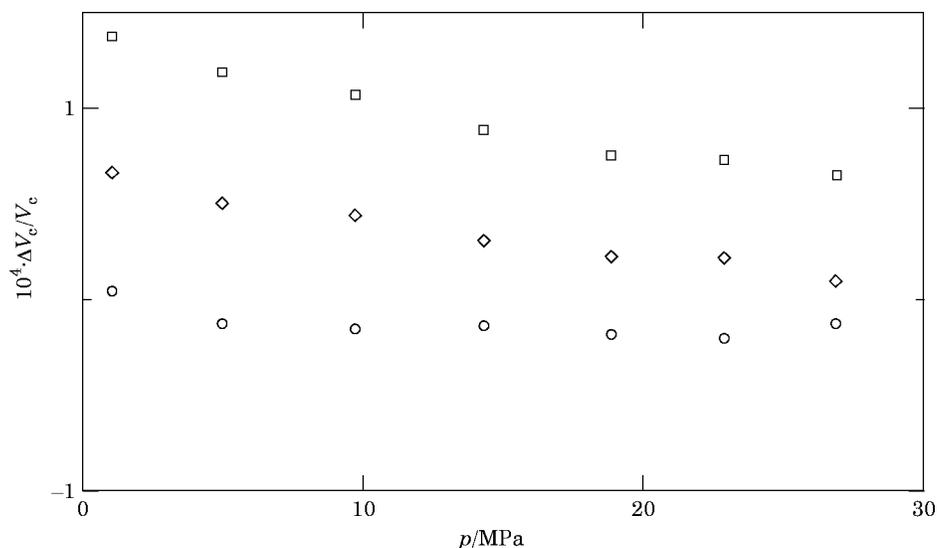


FIGURE 3. Fractional deviations  $\Delta V_c/V_c = \{V_c - V_c(\text{calc.})\}/V_c$  of the internal volume determined by the displacement pump, from values calculated with  $V_c = 222.748 \text{ cm}^3$  combined with the mechanical properties of Inconel 625.  $\circ$ ,  $T = 298.15 \text{ K}$ ;  $\diamond$ ,  $T = 313.15 \text{ K}$ ;  $\square$ ,  $T = 333.15 \text{ K}$ .

TABLE 1. Density  $\rho$  of *n*-octane at temperatures  $T$  and pressures  $p$  with an uncertainty that varies between  $7.4 \cdot 10^{-5} \rho$ , at 303.15 K and 0.675 MPa, and  $3.6 \cdot 10^{-4} \rho$ , at 298.028 K and 32.606 MPa

$T/\text{K}$	$p/\text{MPa}$	$\rho/(\text{kg}\cdot\text{m}^{-3})$	$p/\text{MPa}$	$\rho/(\text{kg}\cdot\text{m}^{-3})$	$p/\text{MPa}$	$\rho/(\text{kg}\cdot\text{m}^{-3})$
298.028	32.60 <sub>6</sub>	723.3 <sub>7</sub>	14.56 <sub>4</sub>	710.6 <sub>7</sub>	4.84 <sub>3</sub>	702.7 <sub>7</sub>
	27.58 <sub>5</sub>	720.0 <sub>4</sub>	9.67 <sub>3</sub>	706.8 <sub>1</sub>	0.74 <sub>4</sub>	699.1 <sub>6</sub>
	20.69 <sub>5</sub>	715.2 <sub>0</sub>				
303.15	27.66 <sub>7</sub>	709.7 <sub>7</sub>	14.41 <sub>2</sub>	699.4 <sub>0</sub>	4.86 <sub>4</sub>	690.9 <sub>2</sub>
	23.49 <sub>2</sub>	706.6 <sub>3</sub>	9.67 <sub>9</sub>	695.2 <sub>9</sub>	0.67 <sub>5</sub>	686.8 <sub>4</sub>
	19.36 <sub>6</sub>	703.4 <sub>6</sub>				

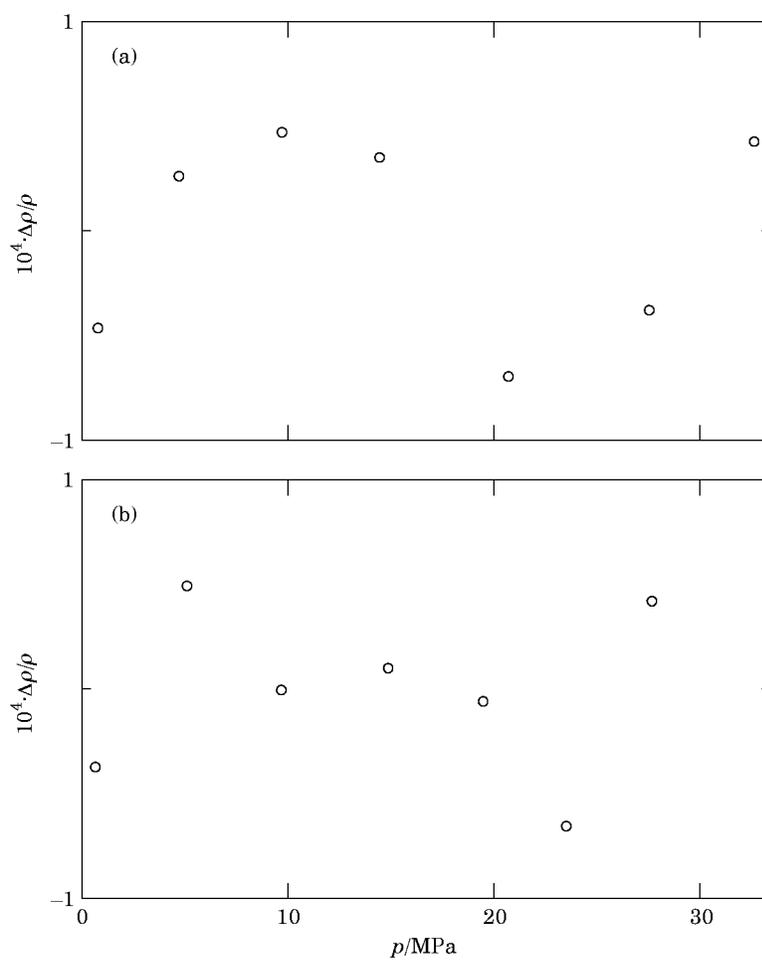


FIGURE 4. Fractional deviations  $\Delta\rho/\rho = \{\rho - \rho(\text{calc.})\}/\rho$  of the density of *n*-octane: (a)  $T=298.03\text{ K}$ , where  $\rho(\text{calc.})$  was given by equation (4); (b)  $T=313.15\text{ K}$ , where  $\rho(\text{calc.})$  was given by equation (5).

known mass of sample occupied at a given state with equation (1). Had we chosen the volume we had measured with the positive-displacement pump, our values of  $\rho$  would differ fractionally from those reported in table 1 by less than  $5 \cdot 10^{-5}$ .

We estimate, assuming that the mercury inventory is exact and the sample is pure, that each density measurement has a best-case precision of  $\pm 7 \cdot 10^{-5} \cdot \rho$ , obtained by combining, in quadrature, the uncertainties arising from the following sources: piston effective area ( $\delta A/A = 2.2 \cdot 10^{-5}$ ), displacement pump resolution of  $\approx 0.001 \text{ cm}^3$  (which corresponds to  $\delta \rho/\rho = 2.5 \cdot 10^{-5}$ ), weighing ( $\delta m/m = 1 \cdot 10^{-5}$ ), cell volume ( $\delta V_c/V_c = 5 \cdot 10^{-5}$ ), pressure ( $\delta p/p = 3 \cdot 10^{-5}$ ), and temperature ( $\delta T/T = 0.1 \cdot 10^{-5}$ ).

The only impurity identified in our sample was water. Prior to drying over molecular sieves the water mole fraction was, as indicated by the manufacturer, about 0.0002 and, although there is no evidence that the actual impurity level approached this value, it is a plausible upper bound for this substance. The effect of this quantity of water on the density of *n*-octane would be to introduce a systematic error of less than  $1 \cdot 10^{-4} \cdot \rho$ . The other likely impurities in this sample are hydrocarbons which would not introduce a significant systematic error.

The mass of mercury injected into the cell was compared with that determined by weighing after the *n*-octane measurements and it differed by only 0.202 g; this difference is about a factor of 10 less than observed previously.<sup>(1,2)</sup> The 0.202 g of mercury corresponds to  $\delta V_{\text{Hg}} \approx 0.014 \text{ cm}^3$  in a volume of the cell occupied by mercury of about  $40 \text{ cm}^3$ . This would lead to an upper bound on the uncertainty of  $3.6 \cdot 10^{-4} \cdot \rho$ . However, the measurements were conducted with a repetitive procedure and it is reasonable to assume that the total observed  $\delta V_{\text{Hg}}$  accumulated over the 14 density measurements. If we assume a linear distribution of the discrepancy in the mercury inventory over these measurements then the error arising from this source varies between  $7.4 \cdot 10^{-5} \cdot \rho$ , at 303.15 K and 0.675 MPa, and  $3.6 \cdot 10^{-4} \cdot \rho$ , at 298.028 K and 32.606 MPa.

#### 4. Discussion

The densities shown in table 1 are illustrated in figure 3 as deviations from the smoothing equations:

$$\rho(298.03 \text{ K}, p)/(\text{kg} \cdot \text{m}^{-3}) = 698.53_5 + 0.889_8 \cdot (p/\text{MPa}) - 3.9_6 \cdot 10^{-3} \cdot (p/\text{MPa})^2, \quad (4)$$

which represented the results with a standard deviation of  $0.043 \text{ kg} \cdot \text{m}^{-3}$  (or about  $6 \cdot 10^{-5} \cdot \rho$ ), and:

$$\rho(313.15 \text{ K}, p)/(\text{kg} \cdot \text{m}^{-3}) = 686.19_8 + 0.986_3 \cdot (p/\text{MPa}) - 4.8_9 \cdot 10^{-3} \cdot (p/\text{MPa})^2, \quad (5)$$

with a standard deviation of  $0.035 \text{ kg} \cdot \text{m}^{-3}$  (or about  $5 \cdot 10^{-5} \cdot \rho$ ). It is clear that the results are a smooth function of pressure. The differences of  $\pm 7 \cdot 10^{-5} \cdot \rho$ , shown in figure 4, are equivalent to the most optimistic estimate of  $\delta \rho/\rho$  for each determination, and are remarkably small considering the resolution of the displacement pump of  $\approx 0.001 \text{ cm}^3$ , which represents up to  $2.5 \cdot 10^{-5} \cdot \rho$ .

The only experimental values of  $\rho(p)$  in the literature with which we can directly

compare our results are those of Dymond *et al.*<sup>(13)</sup> with a cited accuracy of  $0.002 \cdot \rho$ . At  $p = 32.6$  MPa and  $T = 298.03$  K their result is  $1.0_3 \text{ kg} \cdot \text{m}^{-3}$  (about  $0.0014 \cdot \rho$ ) above our value, while at  $p = 0.1$  MPa their result is  $0.076 \text{ kg} \cdot \text{m}^{-3}$  ( $1.1 \cdot 10^{-4} \cdot \rho$ ) above the extrapolation of our results. We consider this agreement to be excellent. There are numerous values of  $\rho(p = 0.1 \text{ MPa})$  reported by other workers<sup>(14-22)</sup> and they are shown in figure 5, as deviations from the smoothing equation:

$$\rho(T)/(\text{kg} \cdot \text{m}^{-3}) = 718.77_9 - 0.7911_3 \cdot \{(T/\text{K}) - 273.15\} - 3.2_6 \cdot 10^{-4} \cdot \{(T/\text{K}) - 273.15\}^2 - 1.2_4 \cdot 10^{-6} \cdot \{(T/\text{K}) - 273.15\}^3, \quad (6)$$

based on the precise ( $\Delta\rho/\rho \approx 2 \cdot 10^{-6}$ ) buoyometric measurements at temperatures between 245.5 K and 379.3 K of Chappelow *et al.*,<sup>(14)</sup> the standard deviation of the fit was  $0.03_4 \text{ kg} \cdot \text{m}^{-3}$  (about  $4.7 \cdot 10^{-5} \cdot \rho$ ). Our results lie  $3.7 \cdot 10^{-4} \cdot \rho$  at 298.03 K and  $3.4 \cdot 10^{-4} \cdot \rho$  at 313.15 K below the value calculated using equation (6). The vibrating wire measurements of Dix *et al.*<sup>(17)</sup> at 289.82 K gave a value for the density of  $4.8 \cdot 10^{-4} \cdot \rho$  below that of equation (6), while at 323.14 K their result deviates by  $2.03 \cdot 10^{-4} \cdot \rho$  from that of equation (6). Thermodynamic Research Center tables<sup>(18)</sup> provide values at 293.15 K and 298.15 K that differ by  $-2.08 \cdot 10^{-4} \cdot \rho$  and  $-2.26 \cdot 10^{-4} \cdot \rho$  respectively from equation (6).

We gratefully thank BP Research and Engineering for permission to publish this work. We are pleased to acknowledge useful discussions with Dr Allan C. Scott, of the National Engineering Laboratory, U.K., who also designed the spherical cell. Finally, we thank Trevor Hesketh and Steven Randle, of BP Research and Engineering, for their design and engineering input to this project.

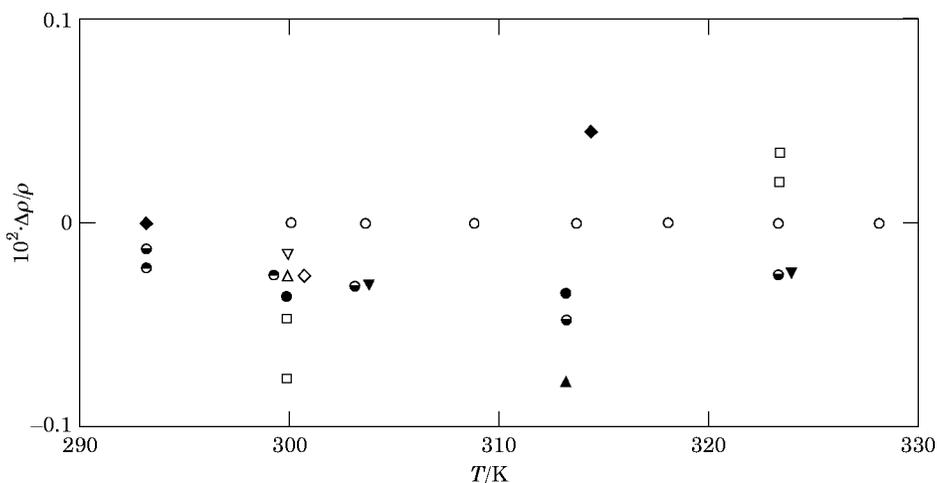


FIGURE 5. Fractional deviations  $\Delta\rho/\rho = \{\rho - \rho(\text{calc.})\}/\rho$  of the density of *n*-octane at a pressure of 0.1 MPa at temperature  $T$  from equation (6). ●, This work; ◇, reference 13; ○, reference 14; ▽, reference 15; △, reference 16; □, reference 17; ⊙, reference 18; ⊖, reference 19; ▲, reference 20; ▼, reference 21; ◆, reference 22.

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(Received 8 November 1995; in final form 2 February 1996)

WA-080