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VISCOSITY AND DENSITY OF
2-METHYLTETRAHYDROFURAN
AS A FUNCTION OF TEMPERATURE

B. P. Engler and L. A. Harrah

Sandia Laboratories

VISCOSITY AND DENSITY OF β -METHYLBUTYRAMIDE AS A FUNCTION OF TEMPERATURE

R. L. Snyder and L. A. Hargreaves
Sandia Laboratories,* Albuquerque, New Mexico 87115

ABSTRACT

The viscosity and density of β -methylbutyramide have been determined from -60°C to 120°C. Viscosity measurements were made using a Ubbelohde viscometer and the density measurements were made using a pycnometer of our own design. The data are represented by the following formulas: $\ln \eta = -2.635 + 4.16T^{-1}$ and $d = 1.120 - 4.56 \times 10^{-4} T$, where η is the viscosity in centipoise, d , the density in grams per cubic centimeter, and T , the temperature in °K. Previous literature data, which extend the viscosity into the glassy state, are included.

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INTRODUCTION

The viscosity of solvents that are used in fluorescence spectroscopy is known to influence quantum efficiency, molecular motion, and excimer formation. In recent work, we have employed 2-methyltetrahydrofuran, a solvent frequently used for fluorescence measurements. A knowledge of the viscosity and density of this solvent over a wide temperature range would be quite useful. However, the only data found on 2-methyltetrahydrofuran were in the glass-forming range, approximately -182°C to -162°C .^{1,2} To complete the picture, data were taken from -60°C to 23°C to obtain formulae for the viscosity and density.

EXPERIMENTAL

An Ubbelohde viscometer was employed to determine the viscosity of 2-methyltetrahydrofuran from -60°C to 23°C . Temperature was maintained by placing the viscometer into an insulated ethanol bath. The temperature was lowered to -60°C by addition of liquid nitrogen. The solvent in the reservoir was charged into bulb D, Figure 1, and maintained here to insure that the solvent temperature was the same as that being read by a thermometer at this position, bulb C (Figure 1). Measurements of the elapsed time for flow of the solvent between two marks (A and B, Figure 1) were taken at 5° increments as the ethanol bath warmed to 23°C .

The viscosity was calculated using $\eta = Kd\Delta t$, where η is the viscosity in centipoise, d , the density in grams per cubic centimeter, Δt , the efflux time in seconds, and K , a calibration factor that equals 0.03008 centistokes per second for our viscometer. The precision of the calculated viscosity was 0.5%. The viscosity range, as calculated from

the efflux time and density data, ranged from 0.5 to 1.75 centipoise (see Tables I and II). A plot of the $\ln_e \eta$ of the viscosity versus the reciprocal of the absolute temperature results in a straight line (Figure 5) that is represented by $\ln_e \eta = -3.635 + 896.8T^{-1} \text{ } ^\circ\text{K.}$

A multiple level calibrated pycnometer of our own design was used to measure the density of 2-methyltetrahydrofuran from -60°C to 23°C . The pycnometer volume was calibrated to each of the level marks present on the tube (Table III). The height of each level and the height of the solvent at each temperature was measured using a cathetometer enabling calculation of the volume of liquid. This was done by using

$$\text{Volume} = (V_{i+1} - V_i) \left(\frac{H - H_i}{H_{i+1} - H_i} \right) + V_i$$

Where H is the height of the solvent and H_i and V_i are the height and volume respectively at the first level mark below the solvent (see Figure 2). The density was then calculated from the mass of solvent and its volume, and resulted in precision of 0.25%.

The pycnometer was clamped in an unsilvered dewar which contained ethanol and was cooled to -60°C with liquid nitrogen. The height of the solvent was determined at 5° increments with a cathetometer as the bath warmed to 23°C . The density, which ranged from 0.85 to 0.93 gm/cm^3 , is plotted against T in $^\circ\text{C}$ (Figure 3). The density data can be represented by the formula, $d = 1.138 - 9.56 \times 10^{-4} T^\circ\text{K.}$ The law of rectilinear diameters predicts that the mean density of liquid and vapor in equilibrium obeys the relationship $\frac{1}{2} (d_l + d_v) = A + BT$ from very low

temperatures to the critical point.³ At low temperatures where the vapor density is small compared with the liquid density, the liquid density itself should be a linear function of temperature. For the case considered here the vapor density reaches 0.17 of the liquid density at 22.6°C so that the linear temperature dependence is expected. The constants derived here neglect the contribution of vapor density and are twice the magnitude of the rectilinear diameter parameters.

CONCLUSIONS

From the data collected in Tables I-IV, which is shown in Figures 3 and 5, the relationship of density versus temperature in °K, and of ρ/ρ_c versus inverse temperature in °K are linear and, thus, allow extrapolation. These results combined with those of Reference 1 and 2 are shown in Figure 6 and give a more complete picture of the viscosity of 2-methyltetrahydrofuran over the range of -182°C to 23°C.

Table I

Efflux Time of 2-Methyltetrahydrofuran
Versus: Temperature

<u>Temperature</u>	<u>Efflux Time</u>
23°C	21.25 Sec.
0°C	26.70 Sec.
- 5°C	28.29 Sec.
-10°C	29.91 Sec.
-15°C	31.80 Sec.
-20°C	33.20 Sec.
-25°C	35.89 Sec.
-30°C	38.52 Sec.
-35°C	41.40 Sec.
-40°C	45.30 Sec.
-45°C	48.54 Sec.
-49°C	52.10 Sec.
-55°C	58.02 Sec.
-60°C	62.00 Sec.

Table II

Viscosity of 2-Methyltetrahydrofuran
Versus Temperature

<u>Temperature (°C)</u>	<u>Viscosity (centipoise)</u>
23°	0.5487
0°	0.7052
- 5°	0.7505
-10°	0.7930
-15°	0.8527
-20°	0.9137
-25°	0.9727
-30°	1.0418
-35°	1.1345
-40°	1.2468
-45°	1.3440
-49°	1.4406
-55°	1.6248
-60°	1.7456

Table III

Volume Determination of Pycnometer

<u>Height Level</u>	<u>Weight of Water (grms)</u>	<u>Volume (cm³)</u>
h_1	4.246	4.2565
h_2	4.547	4.5582
h_3	4.791	4.8028
h_4	4.990	5.0023

Table IV

<u>Density (gm/cm³)</u>	<u>Temperature °C</u>
0.8584	23°
0.864	14.5°
0.878	0°
0.882	- 5°
0.886	-10°
0.8914	-15°
0.896	-20°
0.901	-25°
0.906	-30°
0.911	-35°
0.915	-40°
0.925	-50°
0.931	-55°
0.934	-59°

FIGURE 1
VISCOMETER

FIGURE 2
PYCNOMETER

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