upon mixing with a moist ether solution of picric acid gave only imidazole picrate. In contrast with these properties the ketone (II) was resistant to hydrolysis by 0.5 N sodium hydroxide at reflux temperature and gave a picrate derivative, m.p.  $204^{\circ}$ .<sup>1</sup>

Attempts to acetylate benzimidazole, 2-methylbenzimidazole and lophine (2,4,5-triphenylimidazole) using isopropenyl acetate were unsuccessful.

#### Experimental<sup>3</sup>

1-Acetylimidazole (I).—A inixture of 2.0 g. (0.03 mole) of imidazole, m.p.  $89-90^{\circ}$ , and 20 ml. of isopropenyl acetate containing 10 drops of concentrated sulfuric acid in a 50-ml. round-bottomed flask equipped with a six-bulb Snyder column with condenser attached was heated sufficiently to keep the gas temperature at the top of the column at 60° for one hour. During this time acetone with traces of isopropenyl acetate was collected from the condenser. The reaction mixture was treated with anhydrous sodium carbonate to neutralize the sulfuric acid. The liquid portion was decanted from the solid and evaporated to dryness in a dry air stream at room temperature. A residue of colorless solid product remained, wt. 3.10 g. (94% yield), m.p. 93-96°. After three recrystallizations from isopropenyl acetate, analytically pure N-acetylimidazole was obtained with a constant m.p. 101.5-102.5° (bath preheated to 96°). Anal. Calcd. for CsHsON2: C, 54.53; H, 5.49; N,

Anal. Calcd. for  $C_{b}H_{8}ON_{2}$ : C, 54.53; H, 5.49; N, 25.45. Found: C, 54.45; H, 5.38; N, 25.25.

The preparation of a picrate derivative which was attempted in moist ether gave a compound identical with the picrate of imidazole, m.p. and mixed m.p. 212.5–213.5°.4

Anal. Calcd. for  $C_9H_7O_7N_8$ : C, 36.37; H, 2.37; N, 23.57. Found: C, 36.53; H, 2.34; N, 23.50.

(3) Microanalyses by Mr. Goji Kodama, Chemistry Department, University of Michigan.

(4) Oddo and Ingraffia<sup>1</sup> report a m.p. 212° but no analytical data. R. G. Fargher and F. L. Pyman, J. Chem. Soc., **115**, 217 (1919), report a m.p. 212° and a nitrogen content of 23.3%.

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### Densities of Mixtures. Water and Methanol

#### By G. Chapas

#### **RECEIVED AUGUST 25, 1952**

Writing on the densities of mixtures: water +methanol, Clifford and Campbell stated that no simple equation (of third degree or less) can represent the results of their determinations.<sup>1</sup> However computation of the specific volumes or, better, of the molal volume V of these mixtures shows that additivity is almost satisfied on the whole scale of composition; more precisely the volume of a mixture is very nearly equal to the sum of the volumes of its constituents, the deviation being not larger than 3% for about N = 0.5.

The mean molal volumes calculated from the authors' data are given in the second column of Table I for each value of the mole fraction N of methanol.

By application of the method of least squares, we get the equation

$$V = 18.088_6 + 18.676_5 N + 3.988 N^2$$
 (A)

The coefficient of the third degree term would be very small and it is preferable to neglect it.

The values of  $\vec{V}$  drawn from this equation are written in the third column of the table.

(1) G. Clifford and J. A. Campbell, THIS JOURNAL, 73, 5449 (1951).

Molal volume V, ml. From authors' Mole fraction. N From equation (A) data 0.000 00 18.06918.069 .049 98 19.056 19.012.137 79 20.69320.718.238 20 22.74222.744.375 28 25.62425.639.519 84 28.85528.843.589 01 30.448 30.453 .789 02 35.30135.288 .866 74 37.26637.252 1.000 00 40.72140.721

TABLE I

We may also draw from equation (A) the expressions of the partial molal volumes<sup>2</sup>

Methanol 
$$V_2 = V + (1 - N) \frac{dV}{dN} = 36.745_1 + 7.976 N - 3.988 N^2$$

Water  $V_1 = V - N \frac{dV}{dN} = 18.068_6 - 3.988 N^2$ 

or by setting  $N = N_2$ ,  $1 - N = N_1$ 

 $V_1 = 14.081 + 7.976 N_1 - 3.988 N_1^2$ 

 $V_2 = 36.745 + 7.976 N_2 - 3.988 N_2^2$ 

Then the partial molal volume  $V_1$  of water is always decreasing and gets the minimum value for  $N_1 =$ 0,  $V_1^0 = 14.081$  ml. in almost pure methanol; again methanol finds its minimum partial molal volume for  $N_2 = 0$ , that is, in pure water,  $V_2^0 =$ 36.745 ml.

It is not surprising that no simple equation can represent the densities of the mixtures since the molal volumes are very near to being additive; then the densities must be hyperbolic functions of the mole fraction N so that computation of a linear, or parabolic, or higher degree equation is without object. Emphasis must be put on the role played by the molal volume in physical chemistry and we must regret to read many interesting communications in which synthesized compounds are characterized only by melting point, boiling point or refractive index, density being lacking or given without a fairly good approximation. We might rather hope that such a constant be measured at several temperatures and the corresponding molal volumes calculated.

(2) G. N. Lewis and M. Randall, "Thermodynamics," McGraw-Hill Book Co., Inc., New York, N. Y., 1923.

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## Chlorophenylsilanes

# By Charles A. Burkhard Received September 4, 1952

Organopolysiloxane oils which contain chlorophenyl groups have been prepared and their lubricating properties studied. The preparation of these chlorophenyl-group-containing organopolysiloxane oils required that several new silanes be prepared and characterized: methyl-*p*-chlorophenyldichlorosilane, methylbis-*p*-chlorophenylchlorosilane and methyl-*m*-chlorophenyldichlorosilane. 1,3,5-Tris-*p*-chlorophenyl-1,3,5-trimethyl-