

form layer was shaken twice with ice water, then dried with calcium chloride, filtered and evaporated under reduced pressure to a colorless sirup. The latter was dissolved in absolute ether, and the solution, after filtration, was evaporated *in vacuo* to a half-solid foamy mass. To the 25 cc. of absolute methyl alcohol solution of the residue, 3 cc. of absolute pyridine was added. Crystallization of the " $\gamma$ "-methylmaltoside heptaacetate soon started in the solution. After standing for an hour in the ice-box, the solution deposited 4 g. of long needles. The substance did not reduce Fehling's solution, and melted at 164°. It showed  $[\alpha]_D^{60}$  98.8°,  $[\alpha]_C^{20}$  79.0°,  $[\alpha]_{H_2}^{20}$  116.5°

(0.3967 g. of subst., 10 cc. of chloroform solution, 2-dm. semi-micro tube; rotations, 7.84, 6.27, 9.24° to the right, respectively). Recrystallization from methyl alcohol did not change the specific rotation. Freudenberg gives m. p. 163–164°, and  $[\alpha]_{578}$  101.6° in acetylene tetrachloride for pure " $\gamma$ "-heptaacetylmethylmaltoside. The analyses and the behavior of the substance toward alkali and very dilute acid were in accord with Freudenberg's data.

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## Viscosity of Deuterium<sup>1</sup>

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In conjunction with measurements on the rate of recombination of atomic deuterium it was necessary to evaluate the number of moles of deuterium circulated each second through the apparatus which consisted, essentially, of a reservoir bulb communicating through a capillary leak to a Wood discharge tube and a circulating pump which served to pump atomic-molecular deuterium mixtures along a recombination tube. The determination of the rate of flow of deuterium gave, in addition, data from which the viscosity of deuterium relative to hydrogen could be calculated.

About three liters of deuterium at 700 mm. and 30° prepared by decomposing 99.9% D<sub>2</sub>O with sodium as described by Lewis and Hanson<sup>2</sup> were used as a reservoir in the circulating system employing a Hypervac mechanical pump whose construction made it possible to render the higher pressure (exit) side gas tight and thus return the deuterium, through a liquid air trap, to the reservoir bulb. A capillary flowmeter using Apiezon "B" oil as a manometric fluid was thermostated with the reservoir bulb at 30° and served to check the constancy of flow of deuterium to the discharge tube.

The rate of flow of deuterium,  $dn/dt$ , was de-

(1) Presented at a Meeting of the Harvard-Technology Chemical Club, Massachusetts Institute of Technology, January 15, 1935. In the Bulletin of the American Physical Society, February 5, 1935, there is an abstract of a paper, "The Viscosity of Deuterium" to be presented by H. C. Torrey at the New York meeting of the American Physical Society and the Optical Society of America on February 23, 1935. The results contained therein are in accord with those of the present author.

(2) Lewis and Hanson, THIS JOURNAL, 56, 1687 (1934).

termined after all recombination rate measurements had been taken. Although the final measurements were completed in about two weeks, the deuterium had been circulated through the apparatus and Hypervac pump intermittently during a six-month period. All precautions were taken to minimize exchange as, for example, prolonged pumping to remove water from the phosphoric acid, which poisoned the recombination tube walls, as well as passage of discharges through the apparatus to heat the walls and further dry the phosphoric acid. Dr. Edward S. Gilfillan, of this Laboratory, very generously analyzed the gas at the conclusion of experiments and found a molecular weight of 3.80. The method employed was the comparison of the deuterium with ordinary hydrogen in a gas density balance. The gas, therefore, contained 90% deuterium and 10% hydrogen on a density basis or 81% D<sub>2</sub>, 18% DH and 1% H<sub>2</sub> assuming the usual equilibrium ratio for the molecular species.

Since the rapid pumping system maintained a pressure on the high vacuum side of the capillary leak leading to the Wood tube which was negligible (1 mm. or less) in comparison with that on the high pressure side (700 mm.), the rate of change of pressure in the reservoir bulb when the gas was sent to storage bulbs instead of being returned to the reservoir is

$$dP/dt = kP^2 \quad (1)$$

where  $k$  is evaluated from the integrated equation

$$(1/P_2 - 1/P_1) = -k(t_2 - t_1) \quad (2)$$

If  $V$  is the volume of the reservoir system, the flow of gas at a pressure  $P$  is given by

$$dn/dt = dP/dt \times V/RT \quad (3)$$

The table gives the results for hydrogen and deuterium where the rate of flow,  $dn/dt$ , has been computed at  $30^\circ$  for a pressure of 700 mm. which was maintained in the reservoir ( $V = 2199$  cc.) when circulating gas.

	$P_2$ , mm.	$P_1$ , mm.	$t_2 - t_1$ , sec.	$k$ , mm. <sup>-1</sup> sec. <sup>-1</sup>	$dn/dt$ , moles/sec.
H <sub>2</sub>	537.3	781.7	3800	$-1.616 \times 10^{-7}$	$-0.943 \times 10^{-5}$
D <sub>2</sub>	496.4	694.5	4740	$-1.212 \times 10^{-7}$	$-0.700 \times 10^{-5}$

To test the accuracy of the above method of determining the rate of flow, the value obtained for H<sub>2</sub> was compared with that obtained by electrolytically generating hydrogen at the rate at which it was being pumped through the capillary leak and calculating the flow from the electrolytic current. The two methods checked within the accuracy of the ammeter, about 0.5%, verifying the assumption contained in equation (1) that the flow of gas through the capillary leak is viscous. A further indication of the consistency of the data in the above table is found in the pressure drop across the capillary in the flow meter. In the case where a gas of viscosity  $\eta$  is flowing through a capillary of diameter  $d$  and length  $L$ , if the pressure drop across the capillary,  $\Delta p$ , is small compared with the mean pressure  $P$ , Poiseuille's equation may be written

$$dn/dt = \frac{d^4 P \Delta p}{128 L \eta R T} \quad (4)$$

For deuterium and hydrogen flowing through the same flowmeter the drop in pressure,  $\Delta p$ , should

be the same if the number of moles flowing per second is inversely proportional to the viscosity. Under the same conditions ( $P = 700$  mm.,  $T = 303.16^\circ$  A.) the values of  $\Delta p$  were 4.53 and 4.51 cm. of Apiezon "B" oil (2.86 and 2.85 mm. of Hg) for deuterium and hydrogen, respectively.

It is now possible to obtain a relative value for the viscosity of D<sub>2</sub> since the viscosity of the deuterium mixture relative to hydrogen is given by

$$\frac{dn/dt_{H_2}}{dn/dt_{D_2}} = \frac{0.934 \times 10^{-5}}{0.700 \times 10^{-5}} = 1.334_{\text{obs.}} \quad (5)$$

If we assume that the viscosities vary as the square root of the masses and that the viscosity of a mixture varies linearly with composition, the relative viscosity of the present mixture is given by

$$\frac{0.81 \sqrt{4} + 0.18 \sqrt{3} + 0.01 \sqrt{2}}{\sqrt{2}} = 1.375_{\text{calcd.}} \quad (6)$$

Equation (6) embodies the assumptions that the molecular forces in D<sub>2</sub>, DH and H<sub>2</sub> as well as the molecular diameters are the same. Since the experimental error in the analysis of the deuterium might be as high as 3% and since the linear variation of viscosity with composition may be slightly in error, it may be concluded that within the experimental error the viscosity of D<sub>2</sub> is 1.414 times that of H<sub>2</sub>.

F. G. Keyes (unpublished data) has treated the existing data for the viscosity of hydrogen by the method of least squares and finds that  $\eta = 5.882 \times 10^{-6} \sqrt{T}/(1 + 35.8/T)$ , from which  $\eta = 91.6$  c. g. s. units at  $30^\circ$ . For deuterium, therefore, the value may be taken as equal to  $\sqrt{2} \times 91.6$  or 129.5 c. g. s. units.

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