

needle valve E, F. The pressure of gas in the pipette was adjusted to the desired value by means of the compressor and the pipette sealed by closing the needle valve B. The manifold was then closed to the high-pressure system and opened to the gas burette and pumping system to evacuate the connecting lines. After this had been done the burette was sealed from the pumping system by mercury and the gas enclosed in the pipette expanded into the burette which was maintained at constant temperature in an oil bath. Here its pressure was measured, using a mercury manometer. From volume and pressure measurements with the burette the number of moles of gas initially enclosed in the pipette was found and hence its density at the high pressure calculated. The burette had been calibrated previously⁵ but a correction was applied in these measurements for the fact that 1 ml of the gas was at the cryostat temperature.

The cryostat consisted of a Dewar flask containing liquid nitrogen and fitted with a rubber bung through which passed the steel connecting line to the pipette. Temperatures were measured by a platinum resistance thermometer in a pocket immersed in the liquid nitrogen adjacent to the pipette. A connection to a vacuum pump was also provided.

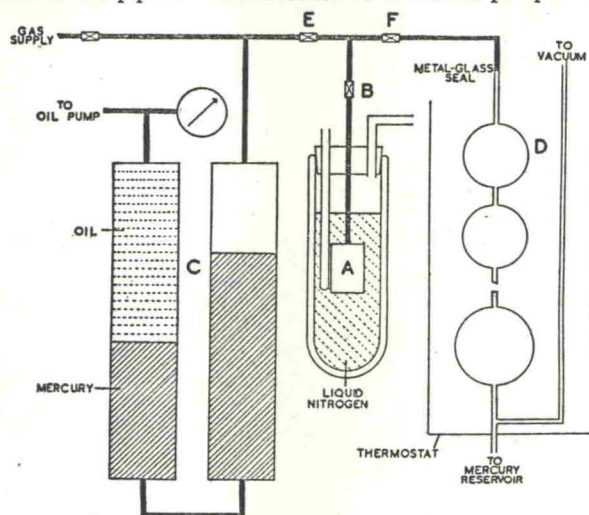


FIG. 1.—Schematic diagram of apparatus.

to reduce the pressure on the liquid nitrogen and so obtain temperatures below the normal boiling point.

It was necessary that the valve B used for sealing the gas pipette be outside the cryostat and it was therefore inevitable that a small proportion of the enclosed gas was approximately at the ambient temperature instead of that of the cryostat. Care was taken to ensure that this amount should be as small as possible; it was about 10% of the total. The temperature of the needle valve B was noted at each density determination and the number of moles of gas at this temperature calculated from the compressibility data of Michels and Goudekot⁷ and subtracted from the total to find the number of moles at the cryostat temperature.

The total volume of the pipette and the fraction of this volume which was immersed in the cryostat were calculated from the dimensions of the apparatus. These volumes were checked by comparing the results of some measurements of the compressibility of hydrogen at 293° K and 79° K with the data of Michels and Goudekot⁷ and of Johnson and White⁸ respectively.

High pressures were measured by Budenberg standard test gauges of the Bourdon tube type, the accuracy of such gauges being sufficient for the purpose of the investigation. Commercial (electrolytic) hydrogen was purified by the method of Hainsworth and MacInnes.⁹ Gas from a cylinder was passed at a pressure of 100-150 atm through a tube containing platinized asbestos heated to 110° C and then through a steel trap immersed in Dry Ice and alcohol to the gas compressor.

Deuterium was prepared from 99.7% D₂O by a method similar to that used by Schiff and Steacie.¹⁰ A 500 ml steel reaction vessel was immersed in Dry Ice and alcohol, evacuated and then filled with dry nitrogen. It was next opened and 50 ml of D₂

introduced. The reaction vessel was then cooled to deep freezing temperatures and the reaction allowed to proceed. The vessel was then removed. After a short wait the pressure rose to 100 atm in about

| pressure (atm) | T = 64° |
|----------------|---------|
| 350 | |
| 500 | |
| 790 | |
| 1000 | |
| 1250 | |

† measurements at

| pressure (atm) | T = 64° |
|----------------|---------|
| 150 | |
| 200 | |
| 300 | |
| 350 | |
| 400 | |
| 500 | |
| 700 | |
| 900 | |

PRESSURE (ATM)

1000

500

0.002

FIG. 2.—Theoretical and experimental

through a liquid nitrogen trap. The reaction vessel was then cooled while the reaction proceeded. After a short wait the pressure rose to 100 atm in about