

and was hence forced through a constriction. This geometry might have been unfavorable because of the internal friction in the solidified gas. It might have been responsible for some of the pressure hysteresis observed by McCormick. In the present arrangement, the coil is supported only by a base, as shown in Fig. 2. In this construction, the compressed solid can flow outside as well as inside the coil. The coil assembly can be readily inserted and taken out of the high-pressure cavity. The coil is a single layer of #30 Nyclad copper wire with a 1- $\mu$ H inductance. It is held together and connected to a brass ring with a small amount of Armstrong adhesive A-12. One end of the coil is soldered to the ring which in turn is in electrical contact with the surface of the cavity. The other coil lead is soldered to a metal rod that passes through the middle of a Teflon insulator and is attached to the top of a sealing cone. This cone is insulated from the cavity by a conical seal of pipestone. The sealing cone narrows to a bolt which extends beyond the bottom of the cavity and is held there by a Teflon washer and a nut. The end of the bolt is soldered to a rigid coaxial transmission line which is connected to the oscillator on top of the cryostat. A Speer carbon resistor, thermally grounded to the high-pressure cavity, is used to monitor the temperature.

Before each new experiment, the piston is kept outside the cavity. The cavity is sealed by a thin foil of copper pressed over the bore with a hollow plug. An indium O-ring insures leak tightness. After the apparatus has been cooled to 77°K, it is cooled very slowly to 4.2°K. Electrolytic H<sub>2</sub> or D<sub>2</sub> with a purity of about 99.6% is condensed slowly into the cavity via a stainless capillary which is heated over its whole length so as to prevent blocking during this process. We assumed that the slow cooling along the thin capillary would get rid of the remaining small O<sub>2</sub> impurity in the gas by adsorption on the walls. Once the cavity is filled with solid, the foil is pierced by the piston and the potassium gasket is made tight by a quick compression to about 1000 atm. During the various pressure cycles, the coil is subjected to repeated strains and usually only lasts for about three experiments.

### B. Electronics

The nuclear resonance signal is detected by a low-level Robinson oscillator<sup>37</sup> followed by a phase sensitive detector. The operating frequency is about 14 Mc/sec for H<sub>2</sub> and 5 Mc/sec for D<sub>2</sub> and is measured by a Hewlett-Packard electronic counter. The magnetic field is modulated by an audio field of about 0.5 G for H<sub>2</sub> and 0.2 G for D<sub>2</sub>. The applied field from a 12-in. Varian magnet is kept constant and the frequency of the oscillator is swept slowly. The derivative of the absorption line is traced on a chart recorder and frequency markers are inserted at regular intervals. The use of low rf levels is

- A HOLLOW PLUG
- B PISTON
- C POTASSIUM
- D COPPER FOIL
- E CAPILLARY
- F COIL
- G BRASS RING
- H TEFLON SPACER
- I PIPESTONE CONE
- J SEALING CONE
- K NUT AND SPACER

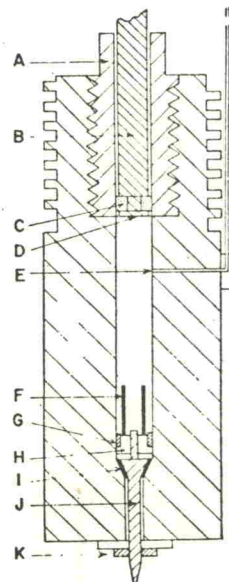


FIG. 2. The high-pressure cavity with piston and rf coil.

necessary to avoid saturation and the resulting distortion of the line. Sugawara's extensive study<sup>5</sup> indicated that an undistorted line is observed with an  $H_{rf}$  of less than  $10^{-2}$  G. In the present experiment, the level is kept at about  $6 \times 10^{-3}$  G, corresponding to an rf level of about 12 mV rms across the coil.

### C. Procedure During an Experiment

A crucial part of the experiment was the determination of the density. As it was not possible to completely fill the sample chamber with the solidified gas, it was necessary to cycle the pressure up and down from zero to the maximum pressure to be reached. After about three such cycles, the solid was packed uniformly and the piston-displacement reading became reproducible. The determination of the density was then made from the piston position  $x$ . For this it was necessary to find the piston position  $x_0$  for zero pressure. However, due to friction in the solid and between the piston, the washer and the inner wall of the sample chamber, the pressure  $P$  inside the solid never returned to zero after a cycle. This friction caused the  $x$  versus the exterior applied pressure  $P_{app}$  to form a hysteresis loop. But it was empirically found that a plot of the slope of the displacement  $\Delta x/\Delta P$  as a function of the increasing applied pressure gave a straight line above 500 atm. By extrapolating this relation to lower pressures, one could obtain an approximate value for  $x_0$ . The error in relative density was estimated to be about 0.01. This is the combined error in the  $x$  versus  $P_{app}$  extrapolation and the uncertainty in the measurement of the piston-tip position relative to the bottom of the cavity. Cycles of  $P_{app}$  versus the volume  $V$  of H<sub>2</sub> in the cavity gave a  $P$ - $V$  relation in agreement with that of Stewart and Swenson.<sup>18,19</sup>

<sup>37</sup> F. N. H. Robinson, J. Sci. Instr. 36, 481 (1959).

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