ture hole. This preserves the packings so that they usually last for about two dozen runs.

To seal the window plugs to their seats, a modified Poulter seal is used.[9–11] The beryllium plugs and the beryllium-copper seats were lapped until matching surfaces were smooth and flat enough to adhere and support their own weight. In principle, the matching flat surfaces provide the gas-tight seal to prevent leakage out of the center hole. In practice, however, it is easier to apply a thin coat of adhesive mixture made of equal parts of G. E. No. 7031 varnish and toluene. The adhesive bond serves to hold the window securely in place during handling and also makes it possible to use parts whose faces no longer adhere because they have become lightly scratched or dented with use.

In preliminary tests, sapphire windows cracked along hexagonal cleavage planes when pressure was released too abruptly. Therefore, the pressure is always changed slowly, usually a few hundred bars per minute, and no difficulty with window fracture has been experienced.

This "unsupported area" design, using the varnish adhesive and soft indium for an initial seal, has proved quite satisfactory for withstanding the shock of cooling to low temperature and subsequent varying of pressure. Its principal shortcoming is the fact that the seal is unstable between 0 and 2000 bars at low temperatures. If the pressure is allowed to drop to this level, leaks develop through the adhesive coating.

C. Electrical Leads

When necessary, electrical leads can be introduced to the sample space in two ways. Fine wires can be led down the pressure tubing from a conventional closure at room temperature or a frozen oil seal.[12] Alternatively, one of the window ports can be used for the purpose. The sapphire window is replaced by a shorter beryllium-copper disk with a lapped face and shallow tapped holes for terminals for a single lead. The varnish coating insulates the disk from the rest of the cell, which then serves as a terminal for the other lead.

D. Cryostat

A diagram of the cryostat used in the color center experiment is shown in Fig. 2. It is a special Pyrex double Dewar system built by H. S. Martin and Son.

The pressure cell is mounted in contact with the inner Dewar filled with the appropriate refrigerant. The outer Dewar terminates in a Kovar cup which gives good thermal contact to the copper radiation shield extending down into the tail of the cryostat. The outside diameter of this

[9] T. C. Poulter, Phys. Rev. **35**, 297 (1930).
[10] E. Fishman and H. G. Drickamer, Anal. Chem. **28**, 804 (1956).
[11] The sapphire windows were ordered with the usual specifications that the cylinder axis be parallel to the c axis within 5° and the faces be flat to within 0.0001 in.
[12] J. S. Dugdale and J. A. Hulbert, Can. J. Phys. **35**, 720 (1957).
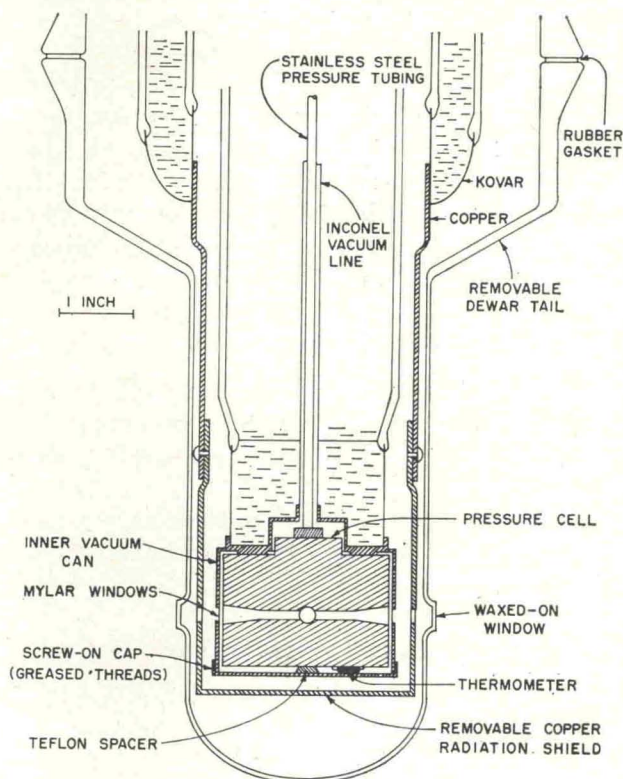

FIG. 2. Tail section of the cryostat.

tail is $3\frac{1}{2}$ in. so that it fits directly into the light proof housing on a spectrophotometer. This tail section and the lower part of the radiation shield are removable to allow easy access to the pressure cell between runs.

The Dewar volumes were chosen large enough to avoid frequent transfers and to maintain the cell at helium temperature overnight. Assuming that cooling results from the heat of vaporization alone, the 650-g cell requires $1\frac{1}{2}$ liters of liquid helium to cool from nitrogen to helium temperature. Once cooled, the helium loss rate is about 100 cc/h, so that the 2-liter inner Dewar is more than adequate. The nitrogen loss rate of the outer Dewar is such that its level drops only half as fast.

The cryostat employs a common vacuum for both Dewars, and any leakage of helium from the pressure cell is intolerable with the pressure ratio of 10^{12}. Therefore, the cell is surrounded by a close-fitting vacuum can of copper with a separate vacuum line. For runs where it is necessary to cross the region of instability of 0–2 kilobars at low temperature, the can is fitted with three 0.001-in. Mylar windows fastened to the inside surface with Epoxy resin, and the threads on the end closure of the can are coated with vacuum grease. In this way it is possible to isolate the cell and maintain a vacuum of 10^{-6} to 10^{-5} mm Hg in the main Dewar while the helium pressure in the can rises as high as one atmosphere. The disadvantage of this scheme is that the Mylar windows act as thin film interference

filters and thus complicate the background absorption correction, so they are left off whenever possible.

In cases where only a brief measurement at a single pressure is necessary, a small metal cryostat has been built in which the pressure cell itself serves as the bottom closure of the helium cold finger, and no separate vacuum jacket is provided. This is feasible because the leakage from the cell is zero when it is properly assembled and pressurized at room temperature before cooling down.

E. Gas Pressure System

Helium gas is supplied to the cell from a remote, intensifier-driven system, assembled from commercially available parts, which can compress gases to 10 kilobars. Contrary to usual practice, a needle valve is included in the high pressure gas line so that the intensifier can be recycled if necessary and the pressure cell sealed off from the system during a run.

F. Temperature and Pressure Determination

The temperature of the pressure cell is measured externally with a thermocouple or carbon resistor. It is assumed that this gives a good indication of the sample temperature, since the sample is surrounded by compressed helium and a large thermal ballast. With liquid helium in the Dewar, the temperature for a given run falls in the range 7 to 12°K, while with liquid nitrogen it is close to 78°K.

The determination of pressure at the sample is much more difficult. Above the freezing curve of helium the pressure is measured externally, using a coil of No. 40 B. & S. gauge manganin wire located in a separate pressure vessel at room temperature. The pressure is measured with an accuracy of about 0.5%, and can be held constant to within 10 bars. This gauge vessel also serves a secondary function as a pressure reservoir, its larger volume suppressing the variation of pressure with temperature in the cell.

Below the freezing curve, the manganin gauge no longer gives the sample pressure, since the pressure line is blocked with frozen helium. The final pressure in the cell at the lowest temperature can be estimated, however, using thermodynamic data available for solid helium.[13–15] Assuming cooling at constant volume from the freezing point determined by the gauge pressure, the calculated final pressure agrees within about 100 to 200 bars with that deduced indirectly from the pressure shift of the F-center absorption band. Part of this discrepancy is probably due to the uncertain correction for the change in volume on freezing, Δv_m, which affects the final pressure if the helium in the tubing freezes before that in the cell. This is apparently not the case for the cryostat described above. Several techniques for measuring the internal pressure directly and precisely at these temperatures are being investigated.

In order to change pressure during helium temperature runs, it is necessary to warm the cell and pressure tubing above the helium melting temperature, change pressure, and recool.

DISCUSSION

The performance of the apparatus described above has demonstrated the feasibility of a whole class of new measurements at high pressures and low temperatures. The cell requires little or no modification to serve in studies of the Mössbauer effect, γ- and x-ray radiation effects, photoconductivity, and fluorescence, for example. Certain limitations and difficulties have also been exposed. In particular, the problems of leakage at low pressure and accurate pressure calibration at helium temperature will require further development of techniques.

ACKNOWLEDGMENTS

[13] J. S. Dugdale and F. E. Simon, Proc. Roy. Soc. (London) **A218**, 291 (1953).
[14] F. A. Holland, J. A. W. Huggill, and G. O. Jones, Proc. Roy. Soc. (London) **A207**, 268 (1951).
[15] E. R. Grilly and R. L. Mills, Ann. Phys. **8**, 1 (1959).