

compressibilities are large and the melting temperatures are low, although bomb design considerations limited the pressure and hence temperature range for work on the heavier solidified gases.¹⁵ The present experiments on solid neon extend to melting temperatures and pressures of up to 53.5 K and 2.5 kbar, and cover a range of molar volumes from 13.39 cm³/mole to 12.39 cm³/mole. The precision of these measurements does not appear to be limited by the heat capacity of the bomb which was used.

The separated neon isotopes have a relatively large (10%) mass difference, and hence should exhibit appreciably different thermodynamic properties. The lattice parameters and thermal expansions of these isotopes have been studied by Batchelder et al.,¹⁶ while Clusius et al.¹⁷ and more recently Somoza and Fenichel¹⁸ have reported C_p measurements for ²⁰Ne and ²²Ne.

2. EXPERIMENTAL DETAILS

These experiments utilize the conventional heat-pulse method of isothermal calorimetry, and the details (as well as the raw data) will be found elsewhere.¹⁹ A completely metal system is used, in part because of potential hazards due to the high pressure in the bomb calorimeter. The bomb, which contains the solid neon and hence is the most important part of the system, is connected to the outside of the cryostat by a fine stainless-steel capillary. The capillary and the bomb are isolated from their surroundings by vacuum spaces (and are kept warm by heaters) while the bomb is filled with solid neon at the melting line. The latent heat of fusion is removed by means of a direct connection to a liquid-helium bath through a mechanical heat switch, so a liquid–solid interface moves slowly upwards in the bomb after freezing has started until the capillary is blocked. The upper part of the capillary is kept filled with solid neon at 4 K throughout the specific heat measurements until all of the data have been taken. The melting temperature which corresponds to the molar volume of the neon is determined at least twice by measuring the rapid increase of the heat capacity of the solid neon in the bomb as the sample is warmed through the melting temperature. This temperature is used to calculate the pressure of the neon in the bomb (and hence the volume of the bomb)²⁰ at this temperature. The number of moles of neon in the sample is determined by expanding the gas into calibrated, temperature-controlled standard volumes at the end of the measurements with the cryostat at room temperature. The measurements of electrical quantities (for calculating the heat input to the sample and the resistance of the germanium thermometer) are sufficiently precise so that they introduce no appreciable error into the results, and the final precision at intermediate

temperatures (2–30 K) is estimated at 0.2%. The errors increase slightly at lower temperatures where the heat capacity of the bomb is relatively more important, and at higher temperatures where the small thermal diffusivity of the neon causes relatively long relaxation times.

2.1. Cryostat and Calorimetric Details

The bomb (which will be described below) is suspended inside the sample chamber by a stainless-steel capillary (0.018 in. o.d. and 0.010 in. i.d. containing a 0.009-in.-stainless-steel wire) which passes from the sample chamber to room temperature through a separate vacuum jacket. The copper walls of the sample chamber form an isothermal shield. The temperature of this shield can be monitored with either a germanium or a platinum resistance thermometer and can be kept constant anywhere between 0.9 and 77 K with either pumped liquid helium in a 100-cm³ pot or a heater. This shield is surrounded by a second vacuum jacket which is attached to a 2-liter liquid-helium reservoir at 4 K. The main cryostat vacuum is common to both the helium reservoir and a liquid-nitrogen container–77 K radiation shield. Thus, there are four distinct vacuum systems: one for the main cryostat (which is sealed-off during an experiment), a second to isolate the isothermal shield from the 4 K shield (this vacuum can be maintained with an oil diffusion pump), a third (which is pumped continuously with an ion pump) to isolate the calorimeter from the isothermal shield, and a fourth (which again is pumped by an ion diffusion pump) to isolate the fill capillary from the liquid-helium bath as the sample is being formed.

The design of the bomb is shown to scale in Fig. 1. The bomb is constructed from hardened beryllium copper (Berlyco 25, half-hard initially) primarily because this material contains no transition metals and hence has a small electronic heat capacity. In addition, these alloys can be machined easily as received, after which they can be heat treated to have a yield strength of approximately 2×10^5 psi at 77 K. Early attempts to construct a bomb by silver soldering this material were unsuccessful, since we were not able to carry out this operation and at the same time retain this yield strength. The final design (Fig. 1) uses a Bridgman-type unsupported area indium seal at the top end for the main seal, and indium solder for supplementary pressure seals. Extrusion of the indium is prevented by the triangular extrusion rings. The capillary is silver soldered to a beryllium copper disc which in turn is indium soldered to the top seal. This design unfortunately leaves a dead space above the bottom of the capillary, and we never were able to fill the bomb completely with solid neon at the melting line. This problem could be remedied easily by a slight modification. The internal copper fins (four at 90°) which are relied upon to conduct heat to the sample are silver soldered