

The sensing elements for the controllers were small platinum resistance thermometers bound firmly to the outsides of A and C.

Inside vessel A was a stainless steel cup D (EN 58B) of capacity slightly greater than vessel C, connected by a drive rod to a d.c. motor E (with reduction gearing). This was in the cold part of the vessel, and caused the cup to rotate at 100 rev/min. The motor and cup

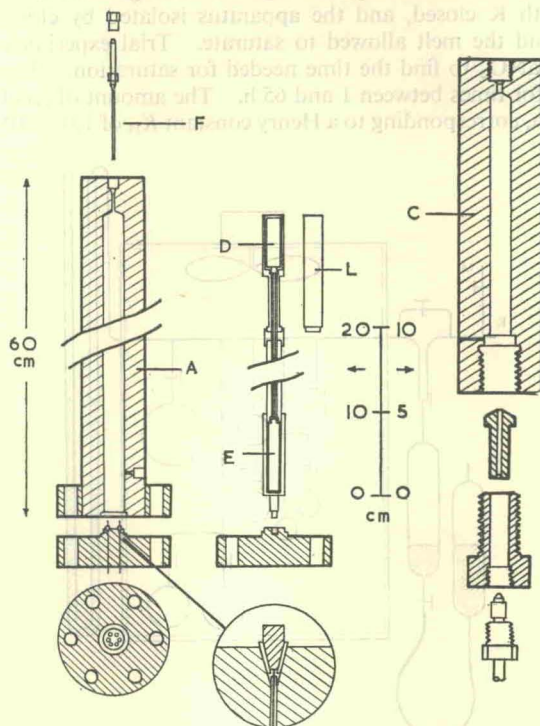


FIG. 2.—Detail of components of the high pressure apparatus. The letters are referred to in the text.

were mounted in housing made from EN 58B, the function of which was to support the components and also to fill the dead space inside the vessel. The junctions of two stainless steel clad chromel-alumel thermocouples (Spembley Technical Products Ltd.) were positioned inside the cup D, one at the top and one at the bottom, to indicate the temperature and check for the absence of a gradient. During runs, the temperature difference between the two junctions was not greater than 1 deg. The thermocouple connections through the lower flange of the vessel were made via cones of chromel and alumel, insulated from the flange by nylon sleeves. Chromel and alumel wires passed from the lower ends of the cones to the cold junctions, which were kept in ice. The pressure tubing connecting A to B had brazed to its lower end a length of stainless steel capillary tubing, which extended to within a few mm of the bottom of D. The working gas from the compressor G could be admitted to the apparatus through valve H, and the pressure could be read to ± 10 bar by the Bourdon gauge J (Budenberg Gauge Co. Ltd.). The gauge calibration was checked periodically against a dead weight tester. The apparatus could be evacuated, or vented to the atmosphere through valve K. The valves, tubing, and high pressure fittings were purchased from Pressure Products (U.K.) Ltd. The apparatus was mounted in a steel supporting frame, which was surrounded by a safety barrier made from $\frac{3}{8}$ in. mild steel plate.

PROCEDURE

The vessels were sealed, and were evacuated through K with B open. B was then closed, and air admitted to the rest of the apparatus. The lower flange and the mechanisms attached

to it were withdrawn from A. A sample of salt was placed in cup D, and melted. A piece of stainless steel gauze was slid over the two thermocouple junctions, which were then positioned in the melt. The gauze caused the melt to be stirred when the cup was rotated by the motor. The assembly was quickly inserted into A, which was at the working temperature, and the closure bolts were fastened. Air was flushed out of A, by alternately admitting the working gas through H and venting through K. The gas was then pumped in to the working pressure, with K closed, and the apparatus isolated by closing H. The stirrer motor was started, and the melt allowed to saturate. Trial experiments were performed on the system $\text{Ar} + \text{NaNO}_3$ to find the time needed for saturation. The melt was exposed to the gas at 200 bar for times between 1 and 65 h. The amount of gas taken up reached a limiting value after 5 h, corresponding to a Henry constant K_H of $1.07 \times 10^{-7} \text{ mol ml}^{-1} \text{ bar}^{-1}$.

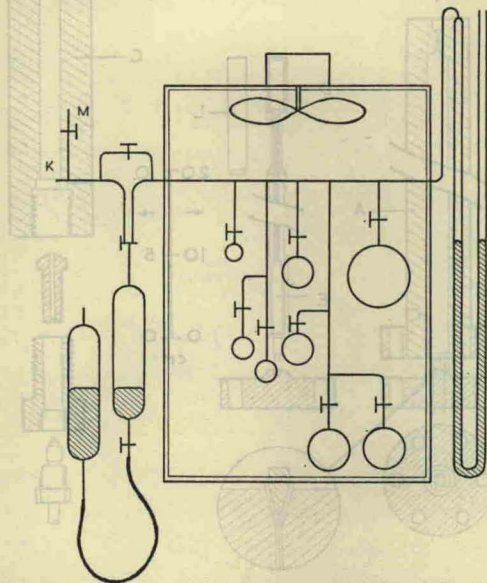


FIG. 3.—Apparatus used to measure the amount of gas recovered from the melt.

In a second experiment, the melt was exposed to the gas at 640 bar for 8 h. The pressure was then reduced to 200 bar, and stirring continued for a further 12 h. The Henry constant at the end of this time was $1.05 \times 10^{-7} \text{ mol ml}^{-1} \text{ bar}^{-1}$. This confirms that equilibrium had been reached in the first experiment. In all subsequent runs, 12 h was chosen as a sufficient and convenient time for saturation. At the end of this time, the motor was turned off and the pressure and temperature noted. Valve B was then opened slowly. The gas pressure in A forced melt up the delivery tube F into vessel C, which was completely filled in a few seconds. B was then closed again, isolating a sample of saturated melt in C. The pressure was released from A, the bolts removed and the inner assembly withdrawn. The cup D, the drive shaft and the upper part of the support housing were removed, and were replaced by the stainless steel cup L (see fig. 2). The modified assembly was replaced in A. The glass apparatus shown in fig. 3 was then attached to the low pressure side of valve K. The volumes of the glass bulbs and the glass tubing had previously been determined (the latter as a function of mercury height in the manometer). K was opened, and vessel A, the Toepler pump, the glass bulbs, and the manometer were evacuated through M. All taps were then closed. Valve B was opened, and evolution of gas caused the melt in C to be ejected down tube F into the cup L. (L was made bigger than the original cup D, because some frothing appeared to occur at this stage. Re-use of D sometimes resulted in spillage.) The evolved gas was transferred to the system of bulbs by means of the Toepler pump, a sufficient number of bulbs being used to give a final pressure just below atmospheric. The