

Samples were originally prepared in three ways:

- a. By binding with a solution of cellulose acetate in amyl acetate
- b. By pressing such a compact at about 8000 psi
- c. By pressing dry powders at the same pressure.

Without lateral support of the sample, however, plastic flow during the application of pressure results in such extensive extrusion that little sample thickness is left between the diamond faces. This typically is of the order of a fraction of a mil. Only after very long exposures then an the Debye lines of X-ray photograph clearly visible. Lateral support of the sample was, however, achieved by the use of copper discs approximately 2.3 mm in diameter with a 0.8-mm hole. Such discs are prepared by electrodeposition for use in electron-microscopy sample preparation and are commercially available. Similar discs with a copper grid across the center hole were tested, but were not as effective.

In addition to the loss of sample, the extrusion which occurred in the absence of any lateral support allowed the sample material outside the face of the smaller diamond to take part of the load. This reduced the load on the diamond and hence the pressure achieved in the indicated volume of the sample.

3. Results

a. Rubidium Chloride

Rubidium chloride was mixed 50 percent by volume with MgO. The sample preparation technique was the same as described above for KCl-NaCl. An initial run with a gas pressure of 100 psi was made to check the completeness of the conversion to the high pressure form at the load represented by that gas pressure and to determine exposure time. Run 60 was then made at two values of the gas pressure, 150 psig and 100 psig. Two values of the compressibility product were obtained from the splitting of the (110) and (200) lines of RbCl-II. These were 0.126 and 0.150 with a mean of 0.138. The MgO with a much lower scattering cross section gave no observable diffraction lines and hence no internal pressure calibration was obtained. After observing the completeness of the reconversion to RbCl-I in run 61, run 62 was made with gas pressures of 60 and 0 psig. Both RbCl-I and RbCl-II lines were observed with no splitting of either indicating that at the higher load only the high-pressure form was present and at the lower load only the low-pressure form.

Run 63 at gas pressures of 40 and 0 psig showed only RbCl-I. Four values of the compressibility product with a mean of 0.124 were obtained. Assuming that the pressure is proportional to the load, the compressibility of the high-pressure form is $5.6 \times 10^{-3} \text{ kbar}^{-1}$.

b. Rubidium Bromide

Internal pressure calibration in the rubidium bromides system was difficult. The use of rubidium chloride which had previously been studied was not practicable because the two rubidium salts form a solid solution. Sodium iodide and rubidium bromide metathesize to give sodium bromide and rubidium iodide with the most intense X-ray diffraction from the latter because of its high average Z. Silver gives interferences with both the low- and high-pressure forms. Platinum has a much lower compressibility and splitting could not be observed.

Since internal calibration was difficult, the pressure ratio was calculated from geometry. This indicated an average pressure over the anvils equal to 0.071 kilobar per psi gas pressure. However, a number of runs indicated that the transition previously determined to occur at 6.0 kilobars was observed at a pressure of approximately 10 psi giving a pressure ratio of 0.6 kilobar per psi gas pressure. This gives a ratio of peak-to-average pressure of about 8.5 which is higher than previously observed. Using this calibration, however, and the observed splitting runs 138 and 139 an average value of β equal to 7.1×10^{-3} kilobar⁻¹ or $7.1 \times 10^{-12} \text{ cm}^2 \text{ dyne}^{-1}$ was obtained for the high-pressure phase (CsCl structure) of rubidium bromide.