



Fig. 1. The beryllium high pressure vessel 24 and part of the channels in the steel supports 9 and 14 are filled with lithium. The test sample 23 surrounded by lithium 19 is placed in a cylindrical opening in the beryllium vessel 24. The x-ray cassette (diameter 68.4 mm) 10 has the usual collimator 22 and catheter 11 for the Debye chamber. The x-ray film 13 is pressed onto the body of the cassette by the ring 20. The pressure is produced in the apparatus by means of the upper screw 15 and the lower nuts on the columns, not shown in the diagram.

Secondary rays were filtered out by putting a 0.016 mm thick aluminum foil in front of the film on the cassette ring. A nickel filter 0.01 mm thick was placed on the collimator cap to filter the copper radiation and reduce the background on the diffraction picture. Exposures were from 1.5 to 3 hours at 30 kv and 26 ma. A demountable tube [4] was used as the x-ray source.

Some pictures were taken in a 143.25 mm diameter VRS-3 chamber at atmospheric pressure to index the diagrams and calculate the lattice parameters of pentaerythritol. A diagram had already been taken in this chamber on a 0.23 mm diameter aluminum sample with asymmetric location of the film. The calculated effective diameter of the chamber was 143.44 mm. The pentaerythritol was used in the form of a 0.5 mm diameter cylinder in a zapon mantle. The results gave the following values for the lattice parameters of pentaerythritol:  $a_0 = 6.10 \pm 0.01$  A and  $c_0 = 8.73 \pm 0.015$  A.

The pictures were taken under pressure in our apparatus using the asymmetric method, calculating the ef-

fective radius of the chamber for each picture, and using the corresponding x-ray diagram to check the displacement of the sample in the beryllium vessel. Within the limits of experimental error, no displacement of the sample was observed from raising and lowering the pressure.

It was found experimentally that the spread in the value of the parameters  $a$  and  $c$  became less as the length of time the sample was kept under pressure was increased, varying from 12 hours to 18 days. With samples kept under pressure for 18 days, the mean-square error in the parameters  $a$  and  $c$  (calculated from measurements on different lines in the x-ray diagram) was less than 0.01 A. All the pictures were taken at room temperature,  $18^\circ \pm 1^\circ$  C. The films were measured independently on the comparator by two observers to avoid subjective errors.

Pictures were taken with both increasing and decreasing pressure, as well as with repeated increase in pressure. The x-ray patterns taken with increase in pressure (in both the first and in repeated cycles) gave very nearly the same results. However, the diagrams taken with decreasing pressure showed hysteresis. The reason for the hysteresis is apparently that there is friction in the system, and the pressure measurements are not right when the pressure is being lowered.

Two to four diagrams were taken at each pressure, giving 46 pressure diagrams in all. The results of the calculations made on the x-ray data are given in the table.

3. A least-squares treatment of the results up to cubic terms in the pressure gave the following expressions for the change in the crystal lattice parameters of pentaerythritol along the  $a$  and  $c$  axes, respectively:

1) For pressures up to  $4200 \text{ kg/cm}^2$

$$-\frac{\Delta a}{a_0} = 0.563 \cdot 10^{-5} P$$

$$- 1.984 \cdot 10^{-9} P^2 + 2.204 \cdot 10^{-13} P^3,$$

$$-\frac{\Delta c}{c_0} = 0.457 \cdot 10^{-5} P$$

$$+ 1.587 \cdot 10^{-9} P^2 - 4.078 \cdot 10^{-13} P^3;$$

2) For the pressure range  $5600\text{--}9000 \text{ kg/cm}^2$

$$-\frac{\Delta a}{a_0} = 0.804 \cdot 10^{-5} P$$

$$- 1.431 \cdot 10^{-9} P^2 + 0.845 \cdot 10^{-13} P^3,$$

$$-\frac{\Delta c}{c_0} = 1.060 \cdot 10^{-5} P$$

$$- 1.690 \cdot 10^{-9} P^2 + 0.994 \cdot 10^{-13} P^3.$$

The calculated values of  $\Delta a/a_0$  and  $\Delta c/c_0$  agree with the measured values to the fourth decimal place. Figures