

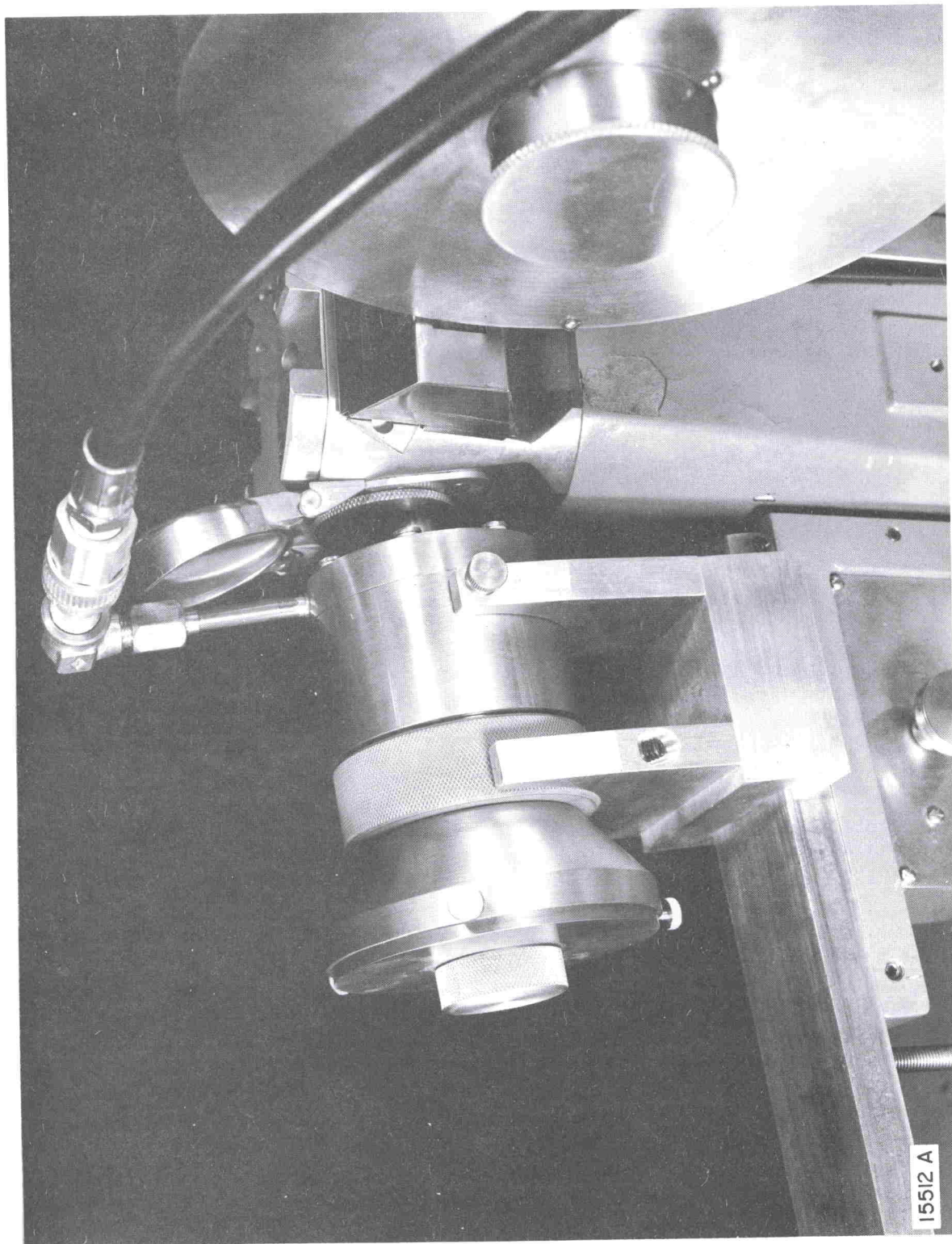


Figure 28 HIGH-PRESSURE CAMERA INSTALLED

Extrusion of the sample from between the faces of the diamond anvils results in a loss in intensity of the diffraction rings and, if extensive, in a reduction in pressure under the anvils by filling the space around the anvils, and absorbing some of the load. Powder samples have been prepared in several ways to avoid this difficulty. The best samples have been prepared by pre-pressing at about 1000 psi either dry powders or powders which have been infiltrated with parlodion.

Before pressing, a copper specimen support 2-1/2 mm in diameter with a 0.8-mm central hole is placed on the powder. During pressing the powder is forced into this hole. The excess powder is trimmed away and the sample is mounted between the diamond anvils. This copper ring gives lateral support to the sample.

Typical photographs are shown in Figures 29 and 30. This sample material is rubidium chloride. The first figure shows two exposures at 25 kilobars and at 1 atmosphere. Lines are observed of both the high-pressure form with the cesium chloride and the low-pressure form with the rock salt structure. Also visible are Laue spots from the two diamond crystals.

A complete list of the exposures made to data are given in Appendix B. The X-ray camera furnished by the manufacturer was supplied with two pistons. On one of these the punch diamond was rigidly fixed to the face of the piston. On the other the punch diamond was mounted on an insert which swiveled on a spherical bearing attached to the piston. In theory, this would facilitate the alignment of the diamond faces. However, in practice this mounting allowed the punch diamond to contact the anvil diamond at random positions. At the conclusion of a run, the punch diamond was found completely off the anvil diamond in at least one case. Although in each case initial contact between the diamonds was made with the axis vertical and a slight holding pressure was maintained after rotation to the horizontal, the weight of the self-aligning mount allowed the punch to drop out of position. Therefore, in all runs after No. 15, the fixed mount was used.

Diamond failures in this program have been infrequent although replacement deliveries have occasionally slowed the program. Only one failure of the punch diamond was observed. After preliminary runs, it was found that the height of the original punch diamond was less than the thickness of its mounting plate. On the first application of pressure, this diamond was pushed out of its mount. A new mounting plate was fabricated to allow 0.007-inch clearance. On the run subsequent to No. 17, a crack was heard as the pressure was being raised. Upon examination the punch diamond was found cracked on (111) planes with a small pyramidal piece broken out of the center. The replacement diamond was used as received until run 41. To allow more room around the diamonds and prevent transference of the load through the sample to the mounting plate, the diamond was remounted with the