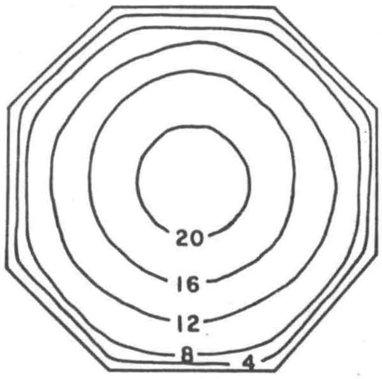

Fig. 4 Pressure contours - good dimond alignment, sample of nickel dimethylglyoxime diluted with 3 parts KBr at an applied pressure of 12 kbar

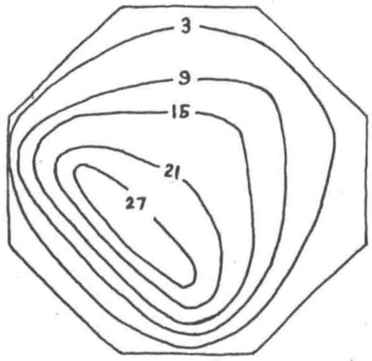

Fig. 5 Pressure contours - poor diamond alignment, same sample used in Fig. 4

microsection of a sample of nickel dimethylglyoxime in the diamond cell and know the peak position as a function of pressure, we can calculate the average pressure of each microsection. By using the microsectioning pattern of Fig.1, we can connect points of equal pressure and derive a pressure contour map such as that shown in Fig.4. A contour map from a similar sample with poorly aligned diamonds is given in Fig.5. If instead we use the microsectioning pattern shown in Fig.2, we get a pressure-distribution curve along the diameter of the cell.

Solid nickel dimethylglyoxime has an extinction coefficient of about $3 \times 10^3 cm^{-1}$ at 19,000 cm^{-1} so that it is difficult to get a good spectrum of the material unless great care is taken to get a particularly thin specimen. This substance was, therefore, in some experiments diluted with an alkali halide. A comparison of the spectra from diluted samples with that of the pure material indicated no spectral effects due to chemical interaction.

Aside from the spectrophotometric method, two other methods for the determination of pressure gradients have been used. A photographic method has been particularly applicable to the determination of the pressure distribution of materials with an absorption edge, such as mercurous compounds or thallous halides. In this method, the sample is viewed in the diamond cell under the microscope with monochromatic radiation. Those portions of the sample whose absorption edge is below the wavelength of radiation used will be transparent while those portions of the sample whose absorption edge is above will appear black. The boundary between the regions corresponds to the pressure at which the absorption edge occurs at the illumination wavelength. Thus, if the position of the absorption edge of a substance is known as a function of pressure, a series of photographs taken at different wavelengths can be used to develop pressure contour maps in much the same way as described above.

The method of following the position of a phase transition used by Roy et al (7) and Van Valkenburg (3) is simplified by the use of photographs. In this method, however, one can only follow the position of one contour (the pressure of phase transition) as a function of applied pressure and does not obtain pressure contours like those given with the other two methods.

EXPERIMENTAL PROCEDURE

The procedure for a typical pressure-gradient determination of the spectrophotometric method follows. Nickel dimethylglyoxime is ground in a mortar with 2, 3 or 5 parts by weight of KBr, NaCl or LiF until the sample is uniformly mixed. A milligram of the sample is placed on the larger diamond anvil and the smaller diamond anvil is placed in the cell and the diamonds are pressed together. The pressure is increased to about 5 kbars where the diamonds may be checked for proper alignment by noting the uniformity of the color variation across the diamond surface. The pressure is reduced to room pressure and then reapplied slowly to the desired pressure. Thus, the spectral measurements were made under conditions of increasing pressure.

A complete analysis of the friction of the lever-and-piston system has not been made but it was found that the hysteresis of the pressure data from the cell could be reduced substantially by liberally lubricating the screw of the pressure cell and by working the screw back and forth a