(4) The cell is mounted on a particularly stable STOE XYZ goniometer head with a height adjustment of 1omm and fits on a Philips single crystal diffractometer, which has a rather small χ-circle. So the whole equipment can easily be used on different types of four-circle diffractometers.

In our construction the gasket is the only strong absorber left in the paths of the X-rays. The use of beryllium reduces this shortcomings dramatically. Recently pressures up to 80kbar have been reached with these beryllium gaskets.

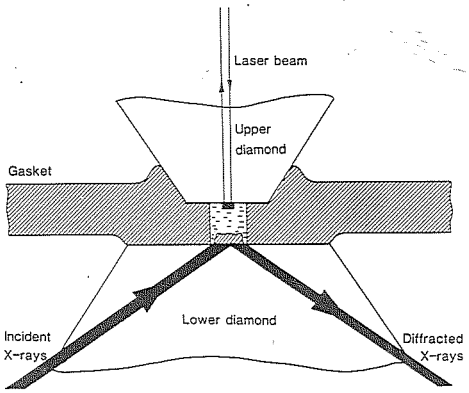


Fig. 2

16.6–5 HIGH PRESSURE PHASES OF SOME ORGANIC COMPOUNDS. By H. Ahsbahs and U. Ohms, Institute for Mineralogy, University of Marburg, Lahnberge, 3550 Marburg, FRG

We investigated the behaviour under pressure of a number of organic compounds whose electron densities have been determined in the Sonderforschungsbereich "Kristallstruktur und chemische Bindung" in Marburg.

A single crystal of the UREA high pressure phase (transition 4.6kbar) could be grown. Using our pressure cell for four-circle diffractometers (Ahsbahs, Rev. Sci. Instr. (1984) 55, 99) the structure has been determined. The crystal was of poor quality. Another measurement with a better crystal will be performed. Crystal data determined so far: a=858, b=827, c=364pm, space group $P2_12_12_1$, Z=4. Asymetric unit: one molecule in general position. R-value 0.087. In the normal pressure phase the molecules are connected by four hydrogen bonds of about 300pm length. In the high pressure phase there are only three hydrogen bonds, two of them shortend by ≈10pm, one is extended a little.

At ambient conditions HEXACYANOBENZENE is cubic, space group Pa3. At 4kbar a high pressure modification is formed. The X-ray powder diffraction lines can be indexed hexagonal with a=870 and c=2460pm.

4-METHYLPYRIDINE is liquid at room temperature and freezes under pressure (1.3kbar) in the same structure as at low temperature. With increasing pressure the structure deforms.

TETRACYANOETHYLENE is dimorph at ambient conditions. The cubic form changes to a high pressure modification with only slightly increased density in the order of 20kbar, the monoclinic one has two high pressure phases at 3kbar and at 20kbar. On release both return to their original cubic and monoclinic phase respectively.

16.6–6 IN SITU HETEROGENEOUS CATALYSIS STUDIES AT VARIABLE TEMPERATURES BY AUTOMATED X-RAY DIFFRACTION SPECTROMETRY. By Glover A. Jones, Central Research & Development Department, E. I. du Pont de Nemours & Company, Experimental Station, Wilmington, Delaware 19898, U.S.A.

A fixed-bed microreactor, capable of operating with mixtures of reducing or oxidizing liquids or gases at pressures between 100 millitorr and one atmosphere, and at temperatures between 80° and 1000° Kelvin, has been constructed especially for x-ray diffraction applications. In a model system study, the crystalline phase catalyst transformations during the oxidation of tetrahydrofuran (THF) to γ-butyrolactone over a $V_2O_5$-$SiO_2$ catalyst have been examined. The fixed-bed reactor, operated in isothermal mode and computer-controlled at temperatures between 443° and 488° Kelvin at atmospheric pressure, was used to collect x-ray diffraction data at each programmed setpoint. XRD measurements in situ show significant proportion of V(IV) on carrying out the oxidation of THF on the catalysts. Reduction in $H_2$ at temperatures above 443° Kelvin gives rise to $V_4O_9$, which is similar to the effect when the reduction is induced by THF.

16.6–7 HIGH-PRESSURE CELL FOR GUINIER X-RAY DIFFRACTION. By K. Knorr, Institut für Kristallographie der Universität Tübingen, West-Germany.

Diamond anvil cells for x-ray diffraction are available up to very high pressures. The disadvantages as very small samples, nonuniformity of the pressure, and inaccuracy of the pressure determination are well-known. For a great number of experiments e.g. investigations of plastic crystals or incommensurate structures pressures below 4 kbar are sufficient. For this range it is possible to construct small gas-pressure cells for hydrostatic pressure conditions and with large sample areas suitable for Guinier diffraction geometrie. We have designed and tested such a cell for applications in the temperature range from 100 to 400 K and for pressures up to 4.1 Kbar at all temperatures. This pressure cell with a diameter of 26 mm and a hight of 20 mm is machined from CuBe alloy, the windows from 2 mm thick Be plates. The slits for the diffracted and the primary beam, respectively, allow diffraction angles from $2\theta = 60°$ to - 30° and oscillation of the sample of ± 10° relative to the primary beam. As the experiments have shown this movement is necessary because many powder samples develop texture effects as a result of recrystallisation during pressure and temperature treatment. He gas is used as the pressure medium. The pressure of 4 kbar is generated by a small transportable (weight 35 kp) supply unit which is operated manually. The pressure cell has been tested successfully in a reinvestigation of the ammonium nitrate p-T-diagram.