quite similar. In both these structures, as well as that of peptide III, the packing can be described as alternating layers formed of hydrophilic peptide rings and hydrophobic side chains. The long axis of the molecule is roughly perpendicular to the layers. By coincidence, this direction is approximately along the a axis in both peptides I and II. The hydrogen-bond networks of the hydrophilic layers include both intermolecular hydrogen bonds and bonds to bridging water molecules, while the packing in the hydrophobic layers is dominated by van der Waals contacts between the leucyl and phenylalanyl side chains.

Further structural studies of cyclic peptides such as peptides I and II whose conformations are not constrained by the presence of proline residues or transannular linkages will prove valuable in assessing the roles of packing forces and solvent interactions in the determination of their molecular conformations.

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X-ray Powder Diffraction Investigation of Naphthalene up to 0.5 GPa

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Abstract

The pressure-induced changes of structural parameters of $C_{10}H_8$, space group $P2_1/a$, $Z = 2$, have been investigated by X-ray powder diffraction at room temperature up to 0.51 GPa. Structure refinement based on measured Bragg intensities yields the reorientation of the molecules, which are assumed to be rigid. $R = 0.031$ and $R_w = 0.014$ for nine intensities and three parameters. At 0.5 GPa the angles between the long axis of the molecule at the origin and the

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monoclinic b and c axes remain nearly unchanged, whereas the angle between the molecular plane and the bc plane shows a decrease of about $1\cdot4\,(1)°$. The experimental results are compared with theoretical packing analyses using intermolecular potentials of the form $U(r) = -Ar^{-6} + B\exp(-\alpha r)$. Agreement is found in the 'quasi-harmonic' approximation.

I. Introduction

The crystalline structure of naphthalene results for the most part from weak van der Waals forces, whereas the binding of the atoms in the molecule is provided by strong covalent bonds. Therefore, the shape of the molecule remains essentially unchanged when it is incorporated in the crystal lattice.

The structure model of naphthalene at room temperature and pressure is that of Cruickshank (1957). At high pressures (up to $0\cdot57$ GPa) only measurements of the lattice constants exist (El Hamamsy, Elnahwy, Damask, Taub & Daniels, 1977). The aim of this work was to determine the molecular reorientation under pressure from Bragg intensities and to analyze the packing on the basis of the potential parameter sets of Kitaigorodsky (1966), Williams (1967), Mirskaya, Kozlova & Bereznitskaya (1974) and Taddei, Righini & Manzelli (1977).

II. Experimental

The experimental set-up consisted of a conventional $\omega/2\theta$ powder arrangement using Mo $K\alpha_1$ radiation (Si monochromator), scintillation counters and digital counting electronics. The high-pressure cell which was newly developed by us is shown in Fig. 1. It was designed especially for a two-circle diffractometer with linear-beam cross sections. Powdered samples can be investigated under hydrostatic helium-gas pressure up to $0\cdot5$ GPa. The X-ray windows of the cell are 10 mm high and permit a scattering angle of $-90° \le 2\theta \le 90°$. Because of its cylindrical symmetry around the axis of the diffractometer and the large volume ($5\text{mm} \oslash \times 10$ mm) available for the sample the device is suitable for recording high-quality diffraction patterns even from weakly scattering compounds.

The sample used was commercial 'naphthalene for scintillation measurements' with a purity higher than 99% (Merck). The raw material was ground and sieved to a particle size smaller than 63 μm. Finer sieving was not possible because of the stickiness of the powder. The sample was placed in a Mark tube ($2\cdot5$ mm in diameter and 20 mm in length) and weakly compressed. A special holder kept this tube in the pressure volume of the cell such that its cylindrical axis coincided with the axis of the diffractometer.

Control of the diffractometer, data collection and processing were performed by a microcomputer in combination with a host computer. To achieve reproducibility of the measurements it was necessary to stabilize the temperature of the whole diffractometer to $\pm0\cdot3$ K.

In the pressure range up to $0\cdot51$ GPa two series of measurements were performed using two different samples. Representative diffraction patterns at $0\cdot01$ and $0\cdot51$ GPa are shown in Fig. 2. The scattered intensity was always recorded on both sides of the primary beam to obtain all the information contained. Fine collimation of the exit beam by two slits cut off all reflections of the high-pressure cell resulting in a nearly constant background intensity with distinct reflections
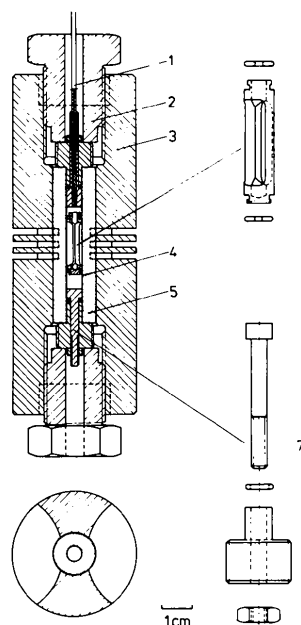

Fig. 1. The high-pressure cell. (1) High-pressure tubing. (2) Retaining screw. (3) Steel body. (4) Aluminium sleeve. (5) Boron carbide cylinder. (6) Sample holder. (7) Sealing arrangement.
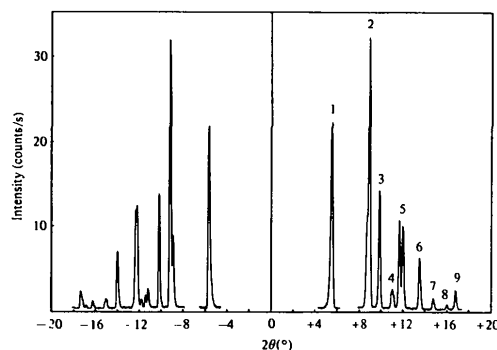

Fig. 2. Diffraction patterns of naphthalene powder at $0\cdot01$ GPa (right side) and $0\cdot51$ GPa (left side). The numbered reflection groups were used for the refinement.

of the naphthalene powder. During the measurements a minimum helium-gas pressure of 0·01 GPa was applied to keep the sublimation rate of the naphthalene low. At the beginning and end of each series, and sometimes in between, we made reference measurements at 0·01–0·02 GPa to look for any changes in the sample. In most cases intensity fluctuations of the reflections did not exceed one statistical standard deviation σ; in all cases they were below 3σ.

As the structure of naphthalene at normal pressure is known (Cruickshank, 1957), it is sufficient to measure the Bragg angles and the ratios of the intensities with and without pressure to obtain complete information on the structural changes. In contrast to absolute intensities these values can be measured very accurately, since scale factors and corrections, which depend only weakly on the scattering angle, are largely eliminated.

In most cases several Bragg reflections are superimposed on reflection groups (Fig. 2). The almost constant background could be subtracted in a reliable way by a fit of first- or second-order polynomials to regions to the left and right of the groups. Thereupon the integrated intensity of such groups was evaluated by a summation of the single counting rates.

The systematic errors of the powder diffractometer were not corrected by the usual formulae (Wilson, 1965), because their accuracy was not believed to be sufficient, taking into account the small Bragg angles (Fig. 2) and the special geometry of the arrangement caused by the high-pressure cell. We took the necessary corrections from a computer simulation of the line profiles. For this purpose the primary beam was divided into discrete rays, which were individually diffracted by the sample. The summing of the appropriately weighted parts of the diffraction cones penetrating the detector slit generated the line profile of the reflection. The geometry of the horizontal and vertical slits used, the dimensions of the X-ray window of the high-pressure cell, and the shape of the specimen as well as the absorption in it were taken into account. From the results of the computer simulation the
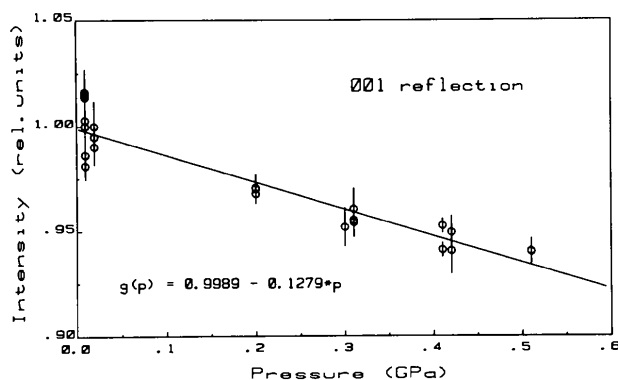
systematic correction of the intensity ratios could be ascertained to better than 0·6%.

For each series of measurements and for both sides of the diffraction pattern separately, the mean values of the integrated intensities at 0·01–0·02 GPa were calculated. All data were divided by these mean values. Fig. 3 shows as an example the resulting intensity ratios of the 001 reflection. The most reliable information on the pressure dependence of the intensities can be obtained from least-squares fits of straight lines $g(p)$ to the data, as random fluctuations of single measuring points are equalized.

III. Results

III.1. Determination of the pressure-induced molecular reorientation

At room temperature the naphthalene crystal makes no structural phase transition, at least up to 3 GPa (Nicol, Vernon & Woo, 1975). One molecule is situated with its center at the origin – hereafter called the reference molecule – whereas the second is related to the first by a screw diad operation (Cruickshank, 1957).

The following procedure is based on the assumption that the molecule in the crystal represents a rigid unit, which under pressure can move only as a whole. Possible deviations from this behavior will be discussed later (§ IV). Therefore, the Euler angles of the reference molecule are sufficient to characterize the orientations of the molecules in the unit cell. The Euler angles φ, θ and ψ are defined by (Goldstein, 1963):

$$\mathbf{x}_0 = R(\varphi,\theta,\psi) \cdot \mathbf{x}_M.$$

We assume that the x_M axis is perpendicular to the molecular plane and the y_M and z_M axes lie parallel to the long and short axes of the molecule, respectively. (x_0,y_0,z_0) defines the crystal coordinate system with the x_0 axis parallel to $\mathbf{a}$, the y_0 axis parallel to $\mathbf{b}$ and the z_0 axis perpendicular.

The determination of the Euler angles from the measurements was made by minimization of:

$$\chi^2 = \sum_{m=1}^{m_0} \frac{1}{w_m} \left\{ \left[\frac{\sum\limits_{hkl} I_{hkl}(p)}{\sum\limits_{hkl} I_{hkl}(0)} \right]_m - g_m(p) \right\}^2. \quad (1)$$

The m_0 measured intensity ratios are contained in the functions $g_m(p)$. The weights w_m result from the variance matrices for the parameters of these functions. The fitted intensity ratios

$$\left[\frac{\sum\limits_{hkl} I_{hkl}(p)}{\sum\limits_{hkl} I_{hkl}(0)} \right]_m$$

Fig. 3. Relative intensity variations of the 001 reflection with pressure.

(the summation includes all hkl triples of the measured reflection group with index m) were calculated from the structure factor, the polarization factor, the Lorentz factor and the volume of the unit cell at pressures p and 0 respectively.

Besides the structure factor, these magnitudes depend only on the lattice constants. These were obtained from a fit to the measured Bragg angles in good accordance with the results of El Hamamsy *et al.* (1977). The structure factor at pressure 0 was calculated from the structure parameters given by Cruickshank (1957). The X-ray scattering factors for the C and H atoms were taken from McWeeny (1951) and from Stewart, Davidson & Simpson (1965), respectively.

The thermal parameters, which are expressed by a translation tensor **R** and a libration tensor **L** (Cruickshank, 1956), could not be deduced from the measurements because of the small number (nine) of independent observations. Therefore, these parameters were extrapolated approximately from the normal-pressure values with the aid of the Grüneisen-mode parameters:

$$\gamma(\mathbf{q},s) = -\frac{V}{\omega(\mathbf{q},s)}\frac{\partial\omega(\mathbf{q},s)}{\partial V},$$

with the crystal volume V, phonon wavevector $\mathbf{q}$ and branch index s. Experimental values of γ vary between 1·7 and 7 (Dows *et al.*, 1973; Nicol *et al.*, 1975; Schmelzer, 1981) with an increasing tendency for smaller ω and weak dispersion (Schmelzer, 1981). This systematic behavior was used for a pressure scaling of the translation and libration tensors with an average Grüneisen parameter $\bar{\gamma} = 5$ (Pawley, 1967):

$$T_{ij}(p) = T_{ij}(0)\left[1 - \bar{\gamma}\frac{V(p) - V(0)}{V(0)}\right]^{-2}, \qquad (2)$$

and a similar expression for L_{ij}. The components $T_{ij}(0)$ and $L_{ij}(0)$ were taken from the structure refinement of Cruickshank (1957).

As a result of the refinement at $p = 0\cdot5$ GPa, the Euler angles of the reference molecule are:*

$$\varphi = -42\cdot18\ (51)° \qquad [-39\cdot79°]$$

$$\theta = -29\cdot95\ (9)° \qquad [-28\cdot61°]$$

$$\psi = 65\cdot00\ (36)° \qquad [64\cdot07°].$$

In square brackets the Euler angles at 0·0 GPa calculated from the data of Cruickshank (1957) are given.

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* Lists of lattice constants at different pressures, anisotropic linear compressibilities and observed and calculated intensity ratios have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36907 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The relatively large standard deviations of φ and ψ are caused by the small value of θ resulting in a strong correlation, which becomes evident in the correlation matrix:

	φ	θ	ψ
φ	1	−0·64	−0·96
θ		1	0·72
ψ			1.

The new fractional coordinates of the molecule are listed in Table 1. The two-dimensional projection in the ac plane (Fig. 4) shows that the long axis of the molecule follows the enlargement of the monoclinic β angle for about 1°. At the same time the molecule performs a realignment of about 1·4° parallel to the bc plane.

Table 1. *Fractional coordinates of the reference molecule at 0·5 GPa and at normal pressure after Cruickshank (1957)*

The 0·5 GPa values are listed above Cruickshank's values. The standard deviations were calculated from the variance matrix of the Euler angles. Cruickshank gives as an estimate for the standard deviations of the C-atom coordinates a range between 0·0032 and 0·0044 Å.

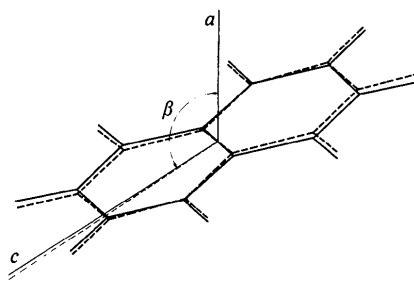|	x	y	z
C(1)	0·0840 (4)	0·0194 (16)	0·3295 (2)
	0·0856	0·0186	0·3251
C(2)	0·1137 (3)	0·1634 (12)	0·2239 (6)
	0·1148	0·1588	0·2200
C(3)	0·0470 (1)	0·1054 (3)	0·0362 (4)
	0·0472	0·1025	0·0351
C(4)	0·0752 (3)	0·2539 (4)	−0·0780 (8)
	0·0749	0·2471	−0·0784
C(5)	0·0129 (4)	0·1919 (11)	−0·2563 (6)
	0·0116	0·1869	−0·2541
H(1)	0·1351 (6)	0·0679 (23)	0·4728 (4)
	0·1375	0·0657	0·4663
H(2)	0·1874 (5)	0·3266 (16)	0·2807 (12)
	0·1888	0·3176	0·2752
H(3)	0·1489 (5)	0·4168 (5)	−0·0212 (14)
	0·1490	0·4056	−0·0233
H(4)	0·0362 (5)	0·3080 (15)	−0·3420 (10)
	0·0345	0·2999	−0·3394

Fig. 4. Projection of the naphthalene molecule at the origin of the unit cell on the crystalline ac plane. Solid lines: 0·0 GPa orientation. Dotted lines: 0·5 GPa orientation.

III.2. *Model calculations*

We compared our experimental results with the predictions of theoretical packing analyses. Frequently the nonbonding van der Waals interaction between two atoms in organic molecular crystals is represented by atom–atom potentials of the '6-exp' type:

$$U(r) = -\frac{A}{r^6} + B \exp(-\alpha r).$$

r is the distance between the two atoms of different molecules. In the case of naphthalene there are nine constants: A, B, α for each C—C, C—H and H—H pairing. We used the values proposed by Kitaigorodsky (1966), Williams (1967) (set IV), Mirskaya, Kozlova & Bereznitskaya (1974) and Taddei, Righini & Manzelli (1977).

For the purpose of packing analyses the lattice energy of the crystal

$$W(p) = \tfrac{1}{2}\sum_{ij} U(r_{ij}) + p\,\Delta V \qquad (3)$$

is minimized with respect to the structural parameters. The summation includes all nonbonding atomic distances $r_{ij} = |\mathbf{r}_i - \mathbf{r}_j|$ in the crystal. The work $p\,\Delta V$ done by pressure p is added, ΔV being the volume change of the crystal due to pressure. If the calculations are carried out with rigid molecules, expression (3) depends only on the lattice constants a,b,c,β and the Euler angles φ,θ,ψ.

Evidently, this model neglects the lattice vibrations and the zero-temperature motion. In order to consider the influence of the lattice vibrations on the equilibrium structure – at least approximately – the lattice constants were fixed at the measured values at 0·0 and 0·5 GPa respectively and only the Euler angles were varied. This is called 'quasiharmonic' approximation.

The minimizations were performed with a computer program developed in its original version by Pawley & Mika (1974). In all cases the direct summation extended to an interatomic distance of 7 Å; more distant interactions were included by an integration. The internal coordinates of the naphthalene molecule

were taken from the structure refinement of Cruickshank (1957). In Table 2 the results of the calculations are listed.

IV. Discussion

The most prominent feature of the procedure used for the determination of the Euler angles under pressure is the use of the relative intensity ratios only. The advantage of this method has been emphasized in § III. However, it is based on the validity of two assumptions.

(1) The unchanged shape of the molecule under pressure (rigidity).

(2) The correction of the Debye–Waller factor by an average Grüneisen parameter.

The bond lengths and bond angles of the free molecule in the gas phase (*mmm* symmetry) are known from electron diffraction experiments of Almenningen, Bastiansen & Dyvik (1961). Within the standard deviations the values agree with the X-ray determination of Cruickshank (1957). However, Cruickshank found deviations of the C atoms from the molecular plane of up to 0·007 Å, in contrast to the structure refinement of deuterated naphthalene (Pawley & Yeats, 1969), indicating no significant violation of the *mmm* symmetry. Therefore, a distortion of the molecule when it enters into the crystal is not yet assured, but in any case is very small.

On pressurizing the crystal, the molecules move closer together resulting in an increase in the intermolecular forces. In the model calculations (§ III.2) we computed forces and moments which act on the atoms of the molecules in equilibrium. The possible changes of the bond lengths and angles could be estimated by the data of Whitmer, Cyvin & Cyvin (1978) for the intramolecular force constants (C—C stretching: 47 nN Å⁻¹; C—H stretching: 50 nN Å⁻¹; C—C—C bending: 7 nN Å⁻¹). The resulting maximum corrections of bond lengths and angles are 0·0015 Å and 0·2° respectively. These values do not exceed the standard deviations of 0·005 Å and 0·2° given by Cruickshank

Table 2. *Results of the packing analyses for different* '6-exp' *parameter sets at* 0·0 *and* 0·5 *GPa*

For comparison the measured structure is given in the last column.

Structural parameter	Kitaigorodsky (1966)	Williams (1967)	Mirskaya *et al.* (1974)	Taddei *et al.* (1977)	Experiment
φ (0·0 GPa) (°)	−43·5	−43·0	−43·3	−43·3	−39·8
φ (0·5 GPa) (°)	−45·6	−45·2	−45·4	−45·3	−42·2
$\Delta\varphi$ (°)	−2·1	−2·2	−2·1	−2·0	−2·4
θ (0·0 GPa) (°)	−28·2	−28·3	−28·5	−28·3	−28·6
θ (0·5 GPa) (°)	−29·3	−29·2	−29·4	−29·2	−29·9
$\Delta\theta$ (°)	−1·1	−0·9	−0·9	−0·9	−1·3
ψ (0·0 GPa) (°)	64·9	65·2	64·7	64·6	64·1
ψ (0·5 GPa) (°)	66·2	66·5	65·9	65·8	65·0
$\Delta\psi$ (°)	1·3	1·3	1·2	1·2	0·9

(1957). Therefore, it is assumed that molecular deformations under pressure do not influence the experimental results.

The scaling of the translation and libration tensors by an average Grüneisen parameter cannot describe the changes of the thermal motion completely. If the average Grüneisen parameter is introduced as an additional adjustment parameter, the value of χ^2 drops from 54 to 15 [equation (1)] with $\bar{\gamma} = 6.63$. Therefore, it is supposed that the residual discrepancies between refinement and experimental data are caused mainly by insufficient correction of the thermal motion.

With a parameterized $\bar{\gamma}$ the angle between the long axis of the reference molecule and the vertical shows a decrease of about $0.1 (1)°$ at 0.5 GPa. The resulting coordinates of the C and H atoms deviate at most by $0.012 (16)$ and $0.017 (23)$ Å, respectively, from the values cited in Table 1. However, the striking reorientation of the molecule with pressure as shown in Fig. 4 is not influenced significantly due to these deviations.

The packing analyses give values of θ and ψ which agree with measurements up to 0.7 and $1.5°$ respectively, whereas φ deviates about $4°$. All parameter sets yield almost the same molecular orientation. The variations of φ and ψ with pressure are within the experimental errors, whereas the decrease of θ is slightly too small. We interpret the results of the model calculations as follows.

The main part of the intermolecular interactions is described correctly by all parameter sets used. In order to achieve complete agreement with the measured structure without any constraint on the lattice constants, the temperature must be included which gives rise to the thermal expansion of the lattice. In addition to the '6-exp' potential, representing essentially the van der Waals terms of the molecular interactions, further contributions – for example, electrostatic multipole forces (Neto, Righini, Califano & Walmsley, 1978) – are to be considered.

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Structures of the Triclinic and Monoclinic Modifications of (2-Oxo-1-pyrrolidinyl)acetamide

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Abstract

$C_6H_{10}N_2O_2$ crystallizes in a triclinic (T) and a monoclinic (M) modification. T has space group $P\bar{1}$ with $a = 6.403 (3)$, $b = 6.618 (4)$, $c = 8.556 (6)$ Å, $\alpha = 79.85 (3)$, $\beta = 102.39 (3)$, $\gamma = 91.09 (3)°$, $Z = 2$. M has space group $P2_1/n$ with $a = 6.525 (2)$, $b = 6.440 (2)$, $c = 16.463 (5)$ Å, $\beta = 92.19 (3)°$, $Z = 4$. Least-squares refinement gave $R(T) = 5.8\%$ for 2499 and $R(M) = 6.4\%$ for 2435 independent reflections