

- LEE, J. D. & GOODACRE, G. W. (1969). *Acta Cryst.* B25, 2127–2131.
- LEE, J. D. & GOODACRE, G. W. (1970). *Acta Cryst.* B26, 507–514.
- LEE, J. D. & GOODACRE, G. W. (1971). *Acta Cryst.* B27, 302–307.
- LEGROS, J. P., CHARBONNEL, Y., BARRANS, J. & GALY, J. (1978). *C. R. Acad. Sci. Sér. C*, 286, 319–320.
- MOSSET, A., BONNET, J. J. & GALY, J. (1977). *Acta Cryst.* B33, 2639–2644.
- PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd ed. Ithaca: Cornell Univ. Press.
- POHL, S. (1976). *Angew. Chem.* 88, 723.
- SEIDEL, W. & ISSLEIB, K. (1963). *Z. Anorg. Allg. Chem.* 325, 113–118.
- TROY, D. (1979). To be published.
- VAN DER HELM, D., LESSOR, A. E. JR & MERRITT, L. L. JR (1960). *Acta Cryst.* 13, 1050.
- WHEATLEY, P. J. (1960). *J. Chem. Soc.* pp. 523–526.
- WILSON, A. J. C. (1949). *Acta Cryst.* 2, 318–321.

Acta Cryst. (1980). B36, 402–406

The Structure and Absolute Configuration of PR Toxin

BY F. BAERT,* M. FOULON AND G. ODOU

Laboratoire de Physique des Solides, Equipe de Dynamique des Cristaux Moléculaires associée au CNRS (n° 465), Université des Sciences et Techniques de Lille I, 59650 Villeneuve d'Ascq, France

AND S. MOREAU

INSERM (U42), 369 rue Jules Guesde, 59650 Villeneuve d'Ascq, France and Laboratoire de Chimie Organique Physique, Université des Sciences et Techniques de Lille I, 59650 Villeneuve d'Ascq, France

(Received 13 June 1979; accepted 15 October 1979)

Abstract

$C_{17}H_{20}O_6$ is orthorhombic, space group $P2_12_12_1$, with $a = 13.980$ (11), $b = 12.341$ (12), $c = 9.609$ (9) Å, $Z = 4$. The structure was determined from 2622 independent intensities and refined to $R = 0.043$. The absolute configuration was solved by the Bijvoet method and corresponds to that predicted on chemical bases. The crystal structure is stabilized by van der Waals forces only.

Introduction

PR toxin has been isolated from cultures of *Penicillium Roqueforti* (Wei, Schnoes, Hart & Strong, 1975). The biological properties result particularly in the inhibition of protein and RNA synthesis (Moule, Jemmali & Rousseau, 1976). A study of the relationships between the chemical structure and the biological properties suggested the important role of an aldehyde group in position 12 associated with an epoxide group and an α,β -unsaturated ketone (Moule, Moreau & Bousquet, 1977). In order to discover the action mechanism, it was important to determine the absolute configuration,

since the octant rule of circular dichroism cannot be used on such a complicated ketone. The impossibility of obtaining a well crystallized Br derivative led us to attempt the determination of the absolute configuration from the anomalous dispersion of the O atoms.

Experimental

A well shaped crystal $0.30 \times 0.24 \times 0.21$ mm was chosen. The crystallographic data are reported in Table 1. Intensities for 2622 reflections were measured by the ω - 2θ scan on a four-circle Philips diffractometer. The scan width was 1.2° . The data were corrected for Lorentz and polarization factors, but not for absorption or extinction. 1284 reflections with $I \geq 3\sigma(I)$ were considered in the determination of the structure.

Table 1. *Crystal data*

$C_{17}H_{20}O_6$, orthorhombic, $P2_12_12_1$	
$M_r = 320.27$	$\lambda(\text{Mo } K\alpha) = 0.7107 \text{ \AA}$
$a = 13.980$ (11) Å	$\mu(\text{Mo } K\alpha) = 0.103 \text{ mm}^{-1}$
$b = 12.341$ (12)	$\lambda(\text{Cu } K\alpha) = 1.5418 \text{ \AA}$
$c = 9.609$ (9)	$\mu(\text{Cu } K\alpha) = 0.812 \text{ mm}^{-1}$
$V = 1657.8 \text{ \AA}^3$, $Z = 4$	$F(000) = 680$
$D_x = 1.28 \text{ Mg m}^{-3}$	Room temperature 294 (2) K

* To whom correspondence should be addressed.

Table 2. Fractional atomic parameters ($\times 10^4$, for H $\times 10^3$)

	x	y	z		x	y	z
O(5)	7135 (2)	6902 (2)	6594 (4)	H(C9)	633 (3)	1141 (3)	642 (4)
O(4)	5963 (2)	8570 (2)	7528 (4)	H(C13)	1041 (3)	1081 (4)	562 (5)
O(3)	9624 (2)	13657 (3)	5209 (4)	H'(C13)	1094 (3)	1179 (4)	593 (5)
O(2)	9256 (2)	11396 (2)	7462 (3)	H''(C13)	1032 (3)	1161 (4)	453 (5)
C(9)	6878 (2)	11087 (3)	6404 (4)	H(C5)	772 (3)	854 (3)	810 (5)
C(3)	6812 (3)	7684 (3)	5578 (4)	H'(C5)	879 (3)	851 (4)	754 (5)
O(1)	7652 (2)	12754 (2)	6484 (4)	H''(C5)	843 (3)	959 (3)	826 (5)
C(4)	7647 (3)	8424 (3)	5212 (4)	H(C4)	748 (3)	878 (3)	433 (5)
C(8)	7713 (3)	11779 (3)	6369 (4)	H(C14)	838 (3)	719 (4)	414 (5)
C(10)	6914 (2)	10011 (3)	6394 (4)	H'(C14)	877 (3)	741 (4)	566 (5)
C(7)	8665 (2)	11217 (3)	6235 (4)	H''(C14)	907 (3)	822 (3)	442 (5)
C(1)	5998 (3)	9414 (3)	6484 (5)	H(C3)	666 (3)	725 (3)	474 (5)
C(5)	7840 (2)	9371 (3)	6245 (4)	H(C2)	532 (3)	798 (3)	572 (5)
C(11)	9536 (3)	11899 (3)	6158 (4)	H(C1)	538 (3)	982 (4)	645 (5)
C(13)	10433 (3)	11502 (4)	5471 (6)	H(C6)	929 (3)	971 (3)	576 (4)
C(12)	9439 (3)	13117 (4)	6208 (5)	H'(C6)	844 (3)	1020 (3)	459 (4)
C(15)	8167 (3)	8968 (3)	7688 (4)	H(C17)	656 (3)	518 (4)	861 (5)
C(6)	8621 (3)	10104 (3)	5621 (4)	H'(C17)	705 (3)	461 (4)	736 (5)
C(16)	6565 (3)	6034 (3)	6791 (6)	H''(C17)	755 (3)	544 (4)	817 (5)
C(2)	5947 (3)	8269 (3)	6093 (5)	H(C12)	931 (3)	1339 (4)	728 (4)
C(14)	8542 (3)	7762 (3)	4856 (5)				
O(6)	5832 (3)	5916 (3)	6195 (5)				
C(17)	6968 (4)	5287 (4)	7857 (7)				

Structure determination and refinement

250 reflections with $|E| \geq 1.55$ were used in *MULTAN* (Germain, Main & Woolfson, 1971). A Fourier synthesis based on the solution with the best figure of merit revealed 18 of the 23 non-hydrogen atoms. A subsequent Fourier map led to the identification of the remaining atoms. Refinement was by full-matrix least squares. H atoms were located from difference maps, except for two H atoms of the methyl group C(17) which were fitted at their expected positions. In the final stages the positional parameters and isotropic temperature factors of the H atoms were allowed to vary. This procedure led to: $R = \sum |F_o| - |F_c| / \sum |F_o| = 0.046$ and $wR = \sum w(|F_o| - |F_c|)^2 / \sum w|F_o|^2 = 0.042$, with unit weights. The goodness of fit $\sum w(|F_o| - |F_c|)^2 / (N_o - N_p)$ was 4.63, where N_o = number of observations and N_p = number of parameters. During the final stages the unit weights were replaced by weights $w = 1/\sigma^2$ derived from counting statistics plus a term taking account of part of the systematic errors $\sigma^2(I) = \sigma^2(I)_{\text{counting}} + (CI)^2$ where C was 0.01. The values of R , wR , and goodness of fit became 0.043, 0.049, and 2.14 respectively.*

The atomic parameters are listed in Table 2. Scattering factors for the heavy atoms were those of Hanson, Herman, Lea & Skillman (1964), for H those

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34700 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

of Stewart, Davidson & Simpson (1965). Peaks and troughs in the final difference synthesis did not exceed $\pm 0.2 e \text{ \AA}^{-3}$.

Determination of the absolute configuration

The absolute configuration was determined on the basis of the O atoms, with the use of the Bijvoet method. Theoretical estimates of the Bijvoet ratio $[\Delta I/I = (I_{hkl} - I_{\bar{h}\bar{k}\bar{l}}) / 0.5 (I_{hkl} + I_{\bar{h}\bar{k}\bar{l}})]$ were calculated from the final coordinates given in Table 2 with the f' and f'' values of C and O. Since the anomalous-dispersion effect was expected to be very small, eighteen reflections with calculated ratios ≥ 0.02 were selected from the data. Measurements were made by a procedure which compensates for any short-term instability of the

Table 3. Comparison of observed and calculated Bijvoet ratios

hkl	100($\Delta I/I$) _c	100($\Delta I/I$) _o	hkl	100($\Delta I/I$) _c	100($\Delta I/I$) _o
10 3 1	-2.3	-0.7	8 2 3	+2.2	+5.0
16 3 1	-2.4	-7	11 7 3	-2.0	-7.8
12 6 1	+3.6	+2.6	2 2 4	-5.8	-2.4
7 8 1	-3.3	-8.6	7 4 4	-2.0	+0.5*
11 2 2	+2.3	+3.0	7 1 5	-4.3	-2.7
6 3 2	+3.8	+6.2	12 3 5	-2.2	-7.3
6 5 2	-4.3	-0.2	12 6 6	+2.0	+0.2
5 6 2	+2.0	+2.6	5 1 8	+2.1	-6.4*
10 2 3	-2.6	-1.6	1 2 8	-2.0	-8.0

* Denotes ratios of opposite signs (which are therefore in contradiction to the assigned configuration).

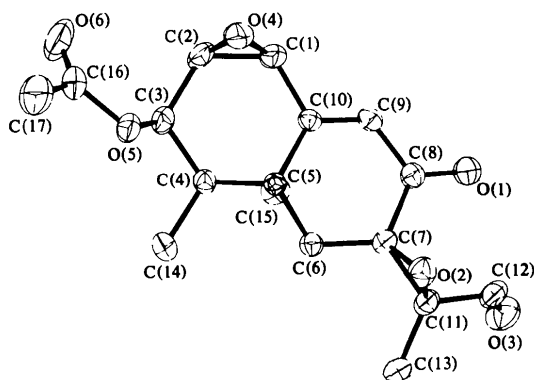


Fig. 1. A general view of the configuration of the molecule showing the atom numbering.

Table 4. Rigid-body-vibration parameters for the heavy atoms of the molecule, with *e.s.d.*'s in parentheses

T (\AA^2) ($\times 10^{-4}$)	326 (24)	33 (19)	-47 (24)
		393 (19)	-17 (20)
			299 (31)
L (rad^2) ($\times 10^{-4}$)	47 (3)	16 (2)	-4 (2)
		76 (8)	-6 (3)
			19 (2)
S (\AA rad) ($\times 10^{-5}$)	-30 (72)	38 (67)	51 (41)
	22 (90)	90 (111)	390 (65)
	27 (30)	40 (39)	59 (861)
r.m.s. ($U^o - U^c$) (\AA^2)	0.0080	e.s.d. U_{ij}^o (\AA^2)	0.0086

instrument: I_{hkl} and then $I_{\bar{h}\bar{k}\bar{l}}$ were measured successively for each reflection eight times with $\lambda(\text{Cu } K\alpha) = 1.5418 \text{ \AA}$. The ω - 2θ scan technique was used with a scan range of 1° and a scan speed of $0.003^\circ \text{ s}^{-1}$. The background was measured on either side of the peak for 84 s. The Bijvoet ratios were obtained only from I_{hkl} and $I_{\bar{h}\bar{k}\bar{l}}$, to avoid absorption effects which would hamper the measurements.

The intensities of each group of observations of the same chirality were averaged to yield 18 Bijvoet pairs. Table 3 lists the measured and calculated values. Comparison of the signs (except two indicated by an asterisk) shows that the absolute configuration is the same as that used for the calculated values, whose

coordinates are listed in Table 2. The absolute configuration corresponds to that predicted on chemical and correlative bases. Moreover the absolute configuration corresponds to the eremophilane type. Such a configuration has already been found for a fungi metabolite of this type (Riche, Pascard-Billy, Devys, Gaudemer, Barbier & Bousquet, 1974). Fig. 1 shows the shapes and orientations of the thermal-vibration ellipsoids.

Thermal-vibration analysis

The vibrational motions were analysed in terms of the rigid-body tensors T , L and S (Schomaker & Trueblood, 1968). The elements of the tensors T , L and S given in Table 4 are derived from a least-squares fit of the atomic B_{ij} 's. The r.m.s. difference between the observed U_{ij} and those calculated from TLS is 0.0080 \AA^2 .

This rather high value is mainly due to O(1), O(6), C(9), C(16) and C(13), whose $\Delta U^{ij} = (U_o^{ij} - U_c^{ij})$ are between 0.0015 and 0.0436 \AA^2 . The translations are almost isotropic with an average amplitude of 0.18 \AA . The off-diagonal elements of the libration tensor L referred to the inertial axes of the molecules are of the same size, Table 5, as those related to orthogonal axes, so it can be assumed that the molecules do not librate around their inertial axes. However, the angles between the eigenvectors of the tensor L and the inertial axes are not too large (5.67 , 7.52 and 15.39° respectively).

The largest amplitude of vibration is around the axis with the smallest moment of inertia.

The amplitudes of libration were used to correct for the effects of molecular vibration on the bond lengths (Busing & Levy, 1964); the values reported in Table 6 are in better agreement with the standard ones.

Results and discussion

Molecular geometry

The bond lengths (both uncorrected and corrected) and bond angles are reported in Table 6.

Table 5. T and L referred to the inertial axes of the molecule

Principal axes (\AA)		T (\AA^2) ($\times 10^{-4}$)		Principal axes ($^\circ$)		L (rad^2) ($\times 10^{-4}$)		Inertial moments			Direction cosines			
									x	y	z			
0.18	326	33	-46	3.92	40	-2	10	1662	0.874	-0.460	-0.154			
0.20		393	-16	4.97		83	13	305	0.457	0.886	-0.064			
0.16			300	2.49			21	1820	0.168	-0.014	-0.985			
0.20	409	-1	79	5.22		-2	13	305	0.457	0.886	-0.064			
0.18		323	126	3.63		40	10	1662	0.874	-0.460	-0.154			
0.16			315	2.40			21	1820	0.168	-0.014	-0.985			

Table 6. Bond lengths (Å) before and after libration correction for the heavy atoms, and bond angles ($^{\circ}$), with e.s.d.'s in parentheses

	Uncorrected	Corrected		Uncorrected	Corrected
O(5)—C(3)	1.445 (5)	1.452	C(4)—C(14)	1.533 (6)	1.541
O(5)—C(16)	1.349 (5)	1.353	C(8)—C(7)	1.506 (5)	1.514
O(4)—C(1)	1.447 (5)	1.453	C(10)—C(1)	1.479 (5)	1.485
O(4)—C(2)	1.429 (6)	1.437	C(10)—C(5)	1.523 (4)	1.530
O(3)—C(12)	1.196 (6)	1.202	C(7)—C(11)	1.482 (5)	1.487
O(2)—C(7)	1.456 (5)	1.465	C(7)—C(6)	1.497 (5)	1.503
O(2)—C(11)	1.452 (5)	1.460	C(1)—C(2)	1.464 (5)	1.470
C(9)—C(8)	1.446 (5)	1.451	C(5)—C(15)	1.543 (5)	1.552
C(9)—C(10)	1.329 (5)	1.333	C(5)—C(6)	1.539 (5)	1.545
C(3)—C(4)	1.524 (6)	1.529	C(11)—C(13)	1.499 (6)	1.507
C(3)—C(2)	1.492 (6)	1.500	C(11)—C(12)	1.510 (6)	1.515
O(1)—C(8)	1.211 (5)	1.215	C(16)—O(6)	1.184 (6)	1.190
C(4)—C(5)	1.557 (5)	1.564	C(16)—C(17)	1.489 (8)	1.496

C(10)—C(1)—C(2)	120.5 (4)	C(7)—O(2)—C(11)	61.3 (2)
C(4)—C(5)—C(10)	107.6 (3)	C(8)—C(9)—C(10)	124.0 (3)
C(4)—C(5)—C(15)	112.5 (3)	O(5)—C(3)—C(4)	108.5 (3)
C(4)—C(5)—C(6)	108.4 (3)	O(5)—C(3)—C(2)	110.7 (3)
C(10)—C(5)—C(15)	109.6 (3)	C(4)—C(3)—C(2)	114.0 (3)
C(10)—C(5)—C(6)	109.6 (3)	C(3)—C(4)—C(5)	115.8 (3)
C(15)—C(5)—C(6)	109.3 (3)	C(3)—C(4)—C(14)	110.9 (3)
O(2)—C(11)—C(7)	59.5 (2)	C(5)—C(4)—C(14)	113.7 (3)
O(2)—C(11)—C(13)	117.8 (3)	C(9)—C(8)—O(1)	121.9 (4)
O(2)—C(11)—C(12)	112.0 (3)	C(9)—C(8)—C(7)	116.3 (3)
C(7)—C(11)—C(13)	121.6 (4)	O(1)—C(8)—C(7)	121.8 (3)
C(7)—C(11)—C(12)	119.3 (3)	C(9)—C(10)—C(1)	117.7 (3)
C(13)—C(11)—C(12)	114.5 (4)	C(9)—C(10)—C(5)	123.4 (3)
O(3)—C(12)—C(11)	120.6 (4)	C(1)—C(10)—C(5)	118.9 (3)
C(7)—C(6)—C(5)	114.5 (3)	O(2)—C(7)—C(8)	111.3 (3)
O(5)—C(16)—O(6)	122.8 (4)	O(2)—C(7)—C(11)	59.2 (3)
O(5)—C(16)—C(17)	111.4 (4)	O(2)—C(7)—C(6)	118.8 (3)
O(2)—C(16)—C(17)	125.8 (5)	C(8)—C(7)—C(11)	118.0 (3)
O(4)—C(2)—C(3)	115.7 (3)	C(8)—C(7)—C(6)	114.8 (3)
O(4)—C(2)—C(1)	60.0 (3)	C(11)—C(7)—C(6)	122.4 (3)
C(3)—C(2)—C(1)	120.8 (4)	O(4)—C(1)—C(10)	115.4 (3)
C(3)—O(5)—C(16)	116.2 (3)	O(4)—C(1)—C(2)	58.8 (3)
C(1)—O(4)—C(2)	61.2 (3)		

C(9)—H(C9)	0.86	C(15)—H''(C15)	1.02
C(3)—H(C3)	0.99	C(6)—H(C6)	1.07
C(1)—H(C1)	1.00	C(6)—H'(C6)	1.02
C(4)—H(C4)	0.98	C(2)—H(C2)	1.00
C(13)—H(C13)	0.87	C(14)—H(C14)	1.01
C(13)—H'(C13)	0.92	C(14)—H'(C14)	0.94
C(13)—H''(C13)	0.92	C(14)—H''(C14)	1.03
C(12)—H(C12)	1.10	C(17)—H(C17)	0.92
C(15)—H(C15)	0.90	C(17)—H'(C17)	0.96
C(15)—H'(C15)	1.05	C(17)—H''(C17)	0.90

Table 7. Mean planes

Equations for the least-squares planes

$$P1: -0.2215x + 0.2665y - 0.9380z + 4.6366 = 0$$

$$P2: -0.0700x + 0.0217y - 0.9972z + 6.5601 = 0$$

	Deviation from plane P1 (with e.s.d.'s)		Deviation from plane P2 (with e.s.d.'s)
C(3)*	-0.0017 (38)	C(10)*	+0.0074 (38)
C(2)*	+0.0035 (47)	C(9)*	-0.0151 (38)
C(1)*	-0.0035 (47)	C(8)*	+0.0140 (38)
C(10)*	-0.0017 (37)	C(7)*	-0.0063 (38)
O(4)	+1.2092 (36)	O(5)	-0.0275 (38)
O(5)	+1.2761 (36)	C(6)	-0.5724 (38)
C(4)	-0.3185 (38)	C(15)	+1.4055 (38)
C(5)	+0.3650 (37)	O(2)	+1.2286 (29)
C(14)	-0.1455 (47)	O(1)	+0.0932 (38)
C(15)	+1.9071 (38)		

* Atoms included in the mean-plane calculations.

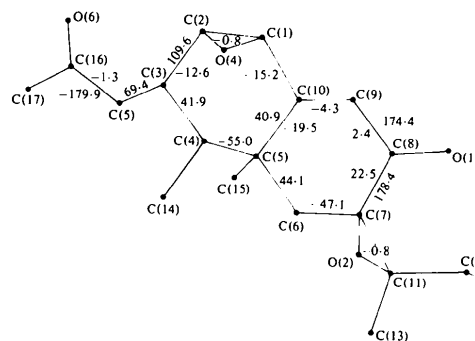


Fig. 2. Ring torsion angles ($^{\circ}$). E.s.d.'s are of the order of 3° .

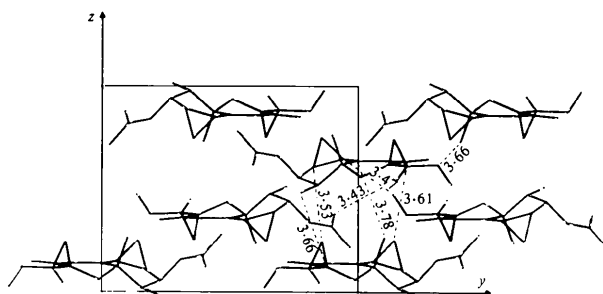


Fig. 3. Projection along *a* of the structure of PR toxin. The shortest distances (Å) between molecules are shown.

The bond lengths are close to the expected values except for the two $C_{sp^2}-C_{sp^2}$ type adjacent to the epoxide rings, which are 1.500 Å for C(3)—C(2) and 1.503 Å for C(7)—C(6). This shortness is linked to the special hybridization of the C atoms in the presence of the epoxide group. The extremely short C(1)—C(2), 1.470 Å, and C(7)—C(11), 1.487 Å, which are engaged in the epoxide rings, are in good agreement with those found by Brisse, Just & Blank (1978). The angles of the epoxide rings are all close to 60° and the average values of the C—C—O and C—O—C angles, 59.4 and 61.3° respectively, compare well with 59.2 and 61.4° (Brisse *et al.*, 1978).

The planes passing through C(3), C(2), C(1), C(10) = P1, and C(10), C(9), C(8), C(7) = P2, and the deviations of atoms from these planes are listed in Table 7.

As can be seen from their equations, these planes are nearly parallel to the *ab* plane. The plane of the $1\beta,2\beta$ -epoxide ring makes an angle of 103.4° with plane P1. The angle between P1 and P2 is 16.90° . The ring torsion angles are shown in Fig. 2. The epoxide group on C(1), C(2), the acetate group on C(3) and the methyl group on C(5) are mutually *syn*. This fact could explain the poor yield obtained during the esterification of the 3β -hydroxide by *p*-bromobenzoyl chloride to synthesize a bromo derivative of PR toxin.

Table 8. *Shortest intermolecular distances (Å) between non-hydrogen atoms*

O(4)—C(13)	2/011*	3.439 (6)	C(13)—O(6)	3/000	3.433 (6)
C(9)—C(15)	2/010	3.572 (5)	C(12)—C(15)	4/010	3.663 (6)
C(9)—C(14)	2/011	3.656 (6)	C(6)—O(4)	2/010	3.442 (5)
C(10)—C(15)	2/010	3.779 (5)	C(2)—O(2)	2/010	3.525 (5)
C(1)—O(3)	3/010	3.465 (5)	C(14)—C(9)	2/010	3.656 (6)
C(11)—O(4)	2/010	3.605 (5)	C(17)—O(3)	2/011	3.430 (7)

* Equivalent-position nomenclature: O(4)—C(13) 2/011 means O(4) at equivalent position (1) to C(13) at equivalent position (2), translated 0,1,1 unit cells in the *a*, *b* and *c* directions respectively. Code: (1) *x,y,z*; (2) $\frac{1}{2} - x, -y, \frac{1}{2} + z$; (3) $\frac{1}{2} + x, \frac{1}{2} - y, -z$; (4) $-x, \frac{1}{2} + y, \frac{1}{2} - z$.

Molecular packing

Fig. 3 illustrates the packing of the molecules in the plane (011). As shown in Table 8, no intermolecular distances are less than the sum of the van der Waals radii of the atoms involved, except that between C(9) and C(15).

The packing of the molecules can be described in terms of layers almost parallel to the *ab* plane (see equation of the mean plane of the rings). As shown in Fig. 3 and Table 8, each molecule is bound by van der Waals forces to six neighbouring molecules belonging to other layers.

Acta Cryst. (1980). **B36**, 406–415

An X-ray Determination of the Charge Deformation Density in 2-Cyanoguanidine

BY F. L. HIRSHFELD

Department of Structural Chemistry, Weizmann Institute of Science, Rehovot, Israel

AND H. HOPE

Department of Chemistry, University of California, Davis, California, USA

(Received 31 July 1979; accepted 31 October 1979)

Abstract

The static deformation density of 2-cyanoguanidine has been mapped by least-squares refinement against low-temperature X-ray data extending to 2.77 \AA^{-1} in $2 \sin \theta / \lambda$. Measured intensities were corrected empirically for extinction and scan truncation. The H atom vibration parameters were fixed with the aid of spectroscopic data. A multipole expansion of the deformation density imposed little more than minimum molecular symmetry and a nuclear cusp constraint. Qualitative and quantitative, *via* atomic partitioning, examination of the experimental deformation density

0567-7408/80/020406-10\$01.00

This work was supported in part by CNRS (ERA No. 465, Dynamique des Cristaux Moléculaires) and by INSERM (U42).

References

- BRISSE, F., JUST, G. & BLANK, F. (1978). *Acta Cryst.* **B34**, 557–562.
- BUSING, W. R. & LEVY, H. A. (1964). *Acta Cryst.* **17**, 142–146.
- GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.
- HANSON, M. P., HERMAN, F., LEA, J. D. & SKILLMAN, S. (1964). *Acta Cryst.* **17**, 1040–1044.
- MOULE, Y., JEMMALI, M. & ROUSSEAU, N. (1976). *Chem. Biol. Interactions*, **14**, 207–216.
- MOULE, Y., MOREAU, S. & BOUSQUET, J. F. (1977). *Chem. Biol. Interactions*, **17**, 185–192.
- RICHE, C., PASCARD-BILLY, C., DEVYS, M., GAUDEMER, A., BARBIER, M. & BOUSQUET, J. F. (1974). *Tetrahedron Lett.* pp. 2765–2766.
- SCHOMAKER, V. & TRUEBLOOD, K. N. (1968). *Acta Cryst.* **B24**, 63–76.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- WEI, R. D., SCHNOES, H. K., HART, P. A. & STRONG, F. H. (1975). *Tetrahedron*, **31**, 109–114.

supports the main predictions of simple resonance theory but shows better detailed agreement with SCF calculations on related molecules, including formamide and cyanogen azide. It also confirms the expected bond bending near the apex N atom.

Introduction

The structure of 2-cyanoguanidine, also called dicyandiamide, has long attracted the attention of crystallographers, who have applied both X-ray (Hughes, 1940; Zvonkova, Krivnov & Khvatkina, 1964) and

© 1980 International Union of Crystallography