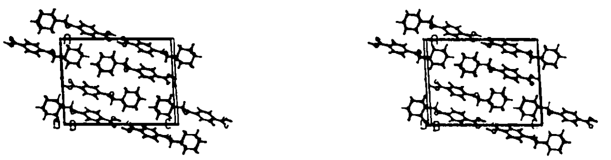

Fig. 2. Packing arrangement of (4).

Table 5 also indicates that H_β [H(9) in (1)–(5)] is in a position favorable for abstraction through a five-membered transition state and in fact is more nearly coplanar with the abstracting oxygen *n* orbital ($\tau = 7$–$13°$) than is H_e; the tertiary nature of H_β should also facilitate its abstraction (Wagner, 1976). Nevertheless, no products corresponding to abstraction of H_β could be detected either in solution or in the solid state (Ariel, Ramamurthy, Scheffer & Trotter, 1983). Reversible β-H abstraction is a possibility.

Intermolecular distances in (1)–(3) and (5) correspond to van der Waals interactions. Crystals of (4) contain hydrogen-bonded centrosymmetric carboxylic acid dimers (Fig. 2) with an O(2)···O(3) distance of 2·634 (4) Å, and an O(2)—H(O)···O(3) angle of 172·9°. The C(15)—O(2) and C(15)—O(3) bond lengths are quite close, 1·277 (5) and 1·245 (5) Å, respectively [Table 3, column (4)], as are the corresponding bond angles C(3)—C(15)—O(2) [117·0 (4)°] and C(3)—C(15)—O(3) [119·8 (4)°]. Although the doubly bonded O(3) and the hydroxyl oxygen, O(2), are not very distinguishable, on the basis of their dimensions, the difference Fourier map (with a cut-off of $\sin\theta/\lambda = 0{\cdot}3$ Å^{-1}) revealed only one carboxyl hydrogen H(O) 1·3 Å away from O(2) [and 1·4 Å from O(3)]. The carboxyl group may be somewhat disordered, but the X-ray room-temperature data could not define this disorder more precisely. The angle between the carboxyl group and the aromatic ring is 2·3 (1)°.

We thank Professor J. R. Scheffer, Dr V. Ramamurthy and Mr N. Omkaram for their collaborative studies on the solid-state photochemistry, the Natural Sciences and Engineering Research Council of Canada for financial support, and the University of British Columbia Computing Centre for assistance.

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Effect of Metal Ions in Organic Synthesis. XXVI. Structure of Methyl 1-(3-Chlorobenzoylamino)-4-diethylaminocarbonyl-2,5-dimethyl-1*H*-pyrrole-3-carboxylate, $C_{20}H_{24}ClN_3O_4$

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Abstract. $M_r = 405{\cdot}9$, monoclinic, $C2/c$, $a = 11{\cdot}050$ (1), $b = 26{\cdot}041$ (2), $c = 14{\cdot}413$ (1) Å, $\beta = 90{\cdot}84$ (1)°, $V = 4146{\cdot}9$ (6) Å^3, $Z = 8$, $D_x = 1{\cdot}30$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}2104$ mm^{-1}, $F(000) = 1712$, $T = 296$ K, $R = 0{\cdot}032$ for 1477 observed reflections. The X-ray crystal structure of the title compound clearly shows a pyrrole ring instead of the theoretically possible dihydropyridazine ring. The crystal packing exhibits two bimolecular hydrogen bonds, providing the formation of an unusual fourteen-membered pseudo ring.

Introduction. In previous papers, some of us (Attanasi, Bonifazi, Foresti & Pradella, 1982; Attanasi, Bonifazi & Buiani, 1983; Attanasi & Santeusanio, 1983; Attanasi, Filippone, Mei & Santeusanio, 1984*a*,*b*;

Attanasi & Perrulli, 1984) reported the synthesis of new and interesting 1-aminopyrrole derivatives by the reaction of azoalkenes with compounds containing an activated methylene group. These one-flask reactions occur at room temperature in the presence of catalytic amounts of copper(II) chloride dihydrate, affording the above-mentioned five-membered aromatic heterocycles, often in good yields.

The biological activities and spectroscopic properties (Attanasi, Santeusanio, Barbarella & Tugnoli, 1984) of some 1-aminopyrrole derivatives have been studied or are currently under investigation.

However, since the usual spectroscopic and physicochemical techniques did not permit an unambiguous assignment of the structure of these products, the present X-ray diffraction study was undertaken in order to confirm that the molecular structure of the title compound contains a pyrrole ring instead of a dihydropyridazine ring, as erroneously reported for analogous products (Brodka & Simon, 1971) and previously shown by some of us (Attanasi, Bonifazi, Foresti & Pradella, 1982). Moreover, this crystallographic study was carried out to determine exactly the geometry of the title compound, in an attempt to obtain some structural relationships between the solid and solution states of this molecule, especially with respect to ^{1}H and ^{13}C NMR spectroscopic characteristics.

Experimental. Single crystal obtained by suction of the precipitate formed in the reaction medium and recrystallized from methanol, as reported in detail by Attanasi, Grossi & Serra-Zanetti (1984). Prismatic crystal 0·77 × 0·48 × 0·24 mm. Philips PW 1100 four-circle diffractometer, graphite monochromator. 28 reflections ($2 < \theta < 30°$) used for measuring lattice parameters with Philips *LAT* routine. ω-scan mode, scan speed 0·12° s^{-1}, scan width 2·0°, θ range 2–20° (intensities were negligible at $\theta > 20°$). Three standard reflections every 180 min, mean intensity variation 2·0%, max. intensity variation 2·7%. 1945 independent data ($-10 \leq h \leq 10$, $-24 \leq k \leq 24$, $l \leq 13$, max. $\sin\theta/\lambda = 0{\cdot}48$ Å^{-1}), 1477 observed with $I > 3\sigma(I)$. Corrections for Lorentz–polarization and experimental absorption, between 1·0005 and 1·0275, following North, Phillips & Mathews (1968). Structure solved by direct methods (*MULTAN*78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). F magnitudes used in least-squares refinement. Coordinates of H atoms calculated by geometrical considerations (*XANADU*, Roberts & Sheldrick, 1975), confirmed by ΔF synthesis showing random fluctuations between 0·07 and −0·06 e Å^{-3}. Parameters refined: coordinates and anisotropic thermal parameters for non-hydrogen atoms, H atoms with isotropic temperature factors, scale factor and secondary-extinction value [final $g = 4{\cdot}3\ (2) \times 10^{-6}$] calculated from the anisotropic coefficients following Coppens & Hamilton (1970). Final value of $R_{\rm obs} = 3{\cdot}24\%$, $R_{\rm all} = 4{\cdot}79\%$, unit weights, $S = 1{\cdot}507$. Max. $\Delta/\sigma = 0{\cdot}002$ in the last cycle of refinement. Atomic scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974). A locally modified version of *ORFLS* (Busing, Martin & Levy, 1962) and the program *PARST* (Nardelli, 1983) were used. Fig. 1 was drawn with *ORTEP*II (Johnson, 1976).

Discussion. The stereochemistry of the title molecule is shown in Fig. 1, which also gives the atom numbering system used in the crystallographic analysis. The crystal packing is illustrated in Fig. 2. The fractional atomic coordinates for non-hydrogen atoms, together with the equivalent isotropic temperature factors, are listed in Table 1.* The intramolecular bond distances and angles are given in Table 2.

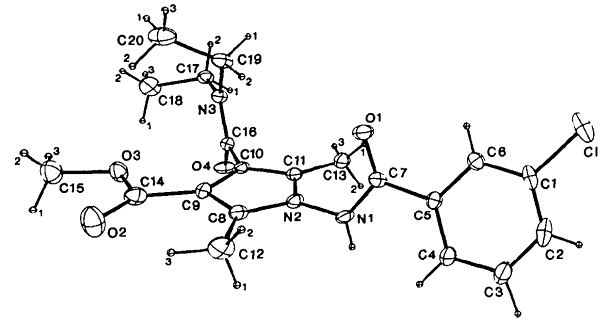

Fig. 1. Perspective view of the molecule.

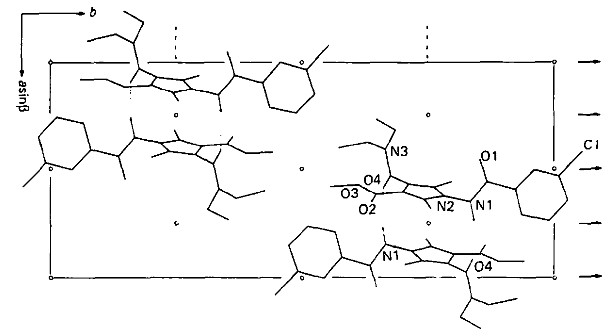

Fig. 2. Projection along the [001] axis of four equivalent molecules of the eight in the unit cell included in a slab with $20 < z < 60$. Intermolecular hydrogen bonds: N(1)—H(N1)···O(4) 2·788 (3) Å. N(1)—H(N1) 0·79 (2), H(N1)···O(4) 2·02 (2) Å, N(1)—H(N1)···O(4) 163 (2)°. The symmetry-related positions are: N(1) and H(N1): x, y, z; O(4): $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$.

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* Lists of structure factors, anisotropic temperature factors for non-hydrogen atoms, torsion angles, least-squares planes with atomic deviations, and fractional atomic coordinates for H atoms and their bond lengths and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39838 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å^2), *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Cl	4166 (1)	10547·4 (4)	6219 (1)	7·98 (4)
O(1)	4528 (2)	8520 (1)	6202 (1)	4·85 (7)
O(2)	6503 (3)	6371 (1)	7205 (2)	7·81 (10)
O(3)	5770 (2)	6121 (1)	5843 (1)	4·89 (7)
O(4)	6008 (2)	6582 (1)	3789 (1)	4·19 (6)
N(1)	6488 (2)	8333 (1)	5994 (2)	4·03 (8)
N(2)	6233 (2)	7817 (1)	5836 (2)	3·41 (8)
N(3)	4096 (2)	6655 (1)	4268 (1)	2·99 (7)
C(1)	5306 (4)	10087 (1)	6316 (2)	4·70 (12)
C(2)	6466 (4)	10244 (1)	6477 (3)	6·21 (14)
C(3)	7377 (3)	9887 (1)	6546 (3)	6·07 (13)
C(4)	7108 (3)	9370 (1)	6454 (2)	4·68 (11)
C(5)	5930 (3)	9212 (1)	6299 (2)	3·26 (9)
C(6)	5023 (3)	9580 (1)	6236 (2)	4·02 (10)
C(7)	5577 (3)	8663 (1)	6175 (2)	3·40 (10)
C(8)	6381 (2)	7436 (1)	6475 (2)	3·50 (9)
C(9)	6053 (2)	6988 (1)	6030 (2)	3·20 (9)
C(10)	5692 (2)	7114 (1)	5101 (2)	2·82 (9)
C(11)	5816 (2)	7627 (1)	4991 (2)	3·27 (9)
C(12)	6824 (3)	7543 (1)	7436 (2)	5·24 (11)
C(13)	5579 (3)	7965 (1)	4184 (2)	4·54 (10)
C(14)	6138 (3)	6482 (1)	6442 (3)	4·23 (11)
C(15)	5787 (3)	5598 (1)	6148 (3)	6·91 (14)
C(16)	5280 (3)	6762 (1)	4345 (2)	3·07 (9)
C(17)	3684 (3)	6297 (1)	3540 (2)	3·55 (9)
C(18)	3986 (4)	5748 (1)	3758 (3)	5·73 (12)
C(19)	3172 (3)	6858 (1)	4896 (2)	4·22 (10)
C(20)	2688 (3)	6460 (2)	5544 (2)	5·91 (12)

Table 2. *Bond distances* (Å) *and angles* (°), *with e.s.d.'s in parentheses*

Cl–C(1)	1·743 (3)	C(11)–N(2)	1·388 (3)
C(1)–C(2)	1·361 (5)	C(12)–C(8)	1·488 (4)
C(2)–C(3)	1·372 (5)	C(13)–C(11)	1·480 (4)
C(3)–C(4)	1·385 (4)	C(14)–C(9)	1·449 (4)
C(4)–C(5)	1·380 (4)	C(14)–O(2)	1·201 (4)
C(5)–C(6)	1·387 (4)	C(14)–O(3)	1·336 (3)
C(6)–C(1)	1·362 (4)	C(15)–O(3)	1·432 (4)
C(7)–C(5)	1·493 (4)	C(16)–C(10)	1·491 (3)
C(7)–O(1)	1·218 (3)	C(16)–O(4)	1·236 (3)
C(7)–N(1)	1·354 (3)	C(16)–N(3)	1·340 (3)
N(1)–N(2)	1·389 (3)	C(17)–N(3)	1·471 (3)
C(8)–N(2)	1·361 (3)	C(17)–C(18)	1·502 (4)
C(8)–C(9)	1·377 (4)	C(19)–N(3)	1·473 (4)
C(9)–C(10)	1·429 (4)	C(19)–C(20)	1·502 (5)
C(10)–C(11)	1·351 (3)		

C(14)–O(3)–C(15)	118·0 (3)	N(2)–C(8)–C(12)	121·9 (3)
N(2)–N(1)–C(7)	119·7 (2)	N(2)–C(8)–C(9)	105·8 (2)
N(1)–N(2)–C(11)	123·6 (2)	C(9)–C(8)–C(12)	132·3 (3)
N(1)–N(2)–C(8)	124·6 (2)	C(8)–C(9)–C(14)	124·4 (3)
C(8)–N(2)–C(11)	111·6 (2)	C(8)–C(9)–C(10)	108·1 (2)
C(17)–N(3)–C(19)	117·3 (2)	C(10)–C(9)–C(14)	127·4 (2)
C(16)–N(3)–C(19)	123·8 (2)	C(9)–C(10)–C(16)	128·4 (2)
C(16)–N(3)–C(17)	118·8 (2)	C(9)–C(10)–C(11)	108·0 (2)
Cl–C(1)–C(6)	119·7 (3)	C(11)–C(10)–C(16)	123·6 (2)
Cl–C(1)–C(2)	119·0 (2)	N(2)–C(11)–C(10)	106·5 (2)
C(2)–C(1)–C(6)	121·3 (3)	C(10)–C(11)–C(13)	131·5 (3)
C(1)–C(2)–C(3)	119·8 (3)	N(2)–C(11)–C(13)	122·0 (2)
C(2)–C(3)–C(4)	119·7 (3)	O(3)–C(14)–C(9)	111·1 (2)
C(3)–C(4)–C(5)	120·4 (3)	O(2)–C(14)–C(9)	127·8 (3)
C(4)–C(5)–C(7)	123·4 (3)	O(2)–C(14)–O(3)	121·1 (3)
C(4)–C(5)–C(6)	118·9 (3)	N(3)–C(16)–C(10)	118·4 (3)
C(6)–C(5)–C(7)	117·7 (3)	O(4)–C(16)–C(10)	120·8 (2)
C(1)–C(6)–C(5)	120·0 (3)	O(4)–C(16)–N(3)	120·8 (2)
N(1)–C(7)–C(5)	115·9 (2)	N(3)–C(17)–C(18)	112·8 (3)
O(1)–C(7)–C(5)	122·4 (2)	N(3)–C(19)–C(20)	113·1 (3)
O(1)–C(7)–N(1)	121·6 (2)		

The sum of the bond angles around the heteroatom N(2) is close to 360°, supporting sp^2 hybridization for this atom. Moreover, the sum of the bond angles involving the atoms of the five-membered aromatic heterocycle is exactly 540°, indicating that the heterocycle is a nearly regular and perfectly planar pentagon with very similar bond distances and angles. In fact, only the C(9)—C(10) bond length [1·429 (4) Å] is longer (by 0·048 Å) than the average value [1·382 (16) Å, $\Delta/\sigma = 2{\cdot}85$] and only the C(8)—N(2)—C(11) bond angle [111·6 (2)°] is significantly larger (by 3·6°) than 108°. For these reasons, the deviations from the least-squares plane for the atoms of the pyrrole ring are very small, ranging from −0·004 (2) Å for C(10) to 0·004 (3) Å for C(9); these values are in good agreement with those found previously by some of us (Attanasi, Bonifazi, Foresti & Pradella, 1982).

The geometries of the methyl groups at C(8) and C(11) are normal, the atoms C(12) and C(13) showing very small displacements [−0·002 (4) and 0·001 (4) Å, respectively] from the mean plane of the heterocycle.

Also the geometry of the methoxycarbonyl group at C(9) seems to be normal, the atom C(14) clearly being above the pyrrole mean plane [0·080 (3) Å]. The geometries of the groups at C(10) and N(2) reveal their usual amidic character, with small deviations of the bond distances and angles from the normal values. Both atoms C(16) and N(1) are slightly above the heterocyclic mean plane, 0·020 (3) and 0·023 (2) Å, respectively.

The benzo ring is almost planar, maximum deviation 0·006 (3) Å for C(1), with the Cl and carbonyl substituents displaced from the benzene mean plane by 0·024 (1) and 0·034 (3) Å, respectively.

The dihedral angle between the mean planes of the benzo ring and the pyrrole ring is 64·7 (1)°.

The crystal packing shows two homologous hydrogen bonds N(1)—H(N1)···O(4) between two molecules, leading to the formation of an unusual 'eight-atom' pseudo ring system with fourteen atoms. The total bond length N(1)—H(N1)···O(4) is 2·788 (3) Å. However, the bond distance 0·79 (2) Å for N(1)—H(N1) appears to be almost normal for a bond of this type, while the bond distance 2·02 (2) Å for the hydrogen bond H(N1)···O(4) may testify to good packing forces between the two linked molecules (Giuseppetti, Tadini, Bettinetti, Giordano & La Manna, 1984). Moreover, an additional longer hydrogen bond C—H···O (not drawn in Fig. 2) is present between the phenyl and pyrrole groups: C(4)—H(4)···O(4) 3·259 (4), C(4)—H(4) 0·97 (2), H(4)···O(4) 2·31 (2) Å, C(4)—H(4)···O(4) 165 (2)°. The N—H···O, as well as the C—H···O, interactions are certainly very important in determining the conformation and the molecular packing of the title compound, according to the detailed discussion by Berkovitch-Yellin & Leiserowitz (1984).

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Structure of 2-Amino-3-phosphonopropionic Acid, $C_3H_8NO_5P$

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Abstract. $M_r = 169{\cdot}1$, monoclinic, $C2/c$, $a = 19{\cdot}976\,(7)$, $b = 7{\cdot}028\,(3)$, $c = 10{\cdot}786\,(4)$ Å, $\beta = 121{\cdot}42\,(4)^\circ$, $V = 1292{\cdot}2\,(10)$ Å^3, $Z = 8$, $D_m = 1{\cdot}74\,(1)$, $D_x = 1{\cdot}74$ Mg m^{-3}, $\bar{\lambda}(\text{Cu }K\alpha) = 1{\cdot}5418$ Å, $\mu = 3{\cdot}59$ mm^{-1}, $F(000) = 704$, $T = 293$ K, final $R = 0{\cdot}039$ for 786 observed reflexions. The molecule exists as a zwitterion, $HOOC{-}CH(NH_3^+)CH_2{-}PO_3H^-$. The conformation about the $C^\beta{-}C^\alpha$ bond is *gauche–gauche*, angles φ[P—C—C—C] and φ'[P—C—C—N] 60·1 (4) and −62·1 (4)° respectively. The NMR results show, however, that in aqueous solution the most stable rotamer is that with the phosphonic and carboxyl groups *trans* relative to each other. The crystal structure is stabilized by five intermolecular and one intramolecular hydrogen bond.

Introduction. Replacement of the β-carboxyl group of aspartate by phosphonate (PO_3^{2-}) yields 2-amino-3-phosphonopropionic acid (β-PAsp).

β-PAsp has been found in living organisms (*Zoanthus sociatus, Tetrahymena pyriformis*) and plays an important role not only as a building unit in proteins but also as a precursor in the biosynthesis of 2-aminoethylphosphonic acid (2-AEP) (Hilderbrand, Curley-Joseph, Lubansky & Henderson, 1982).

Furthermore, Roberts, Foster, Sharif & Collins (1982) reported that the phosphonate analogues of the amino acids aspartate and glutamate interact with excitatory amino-acid receptors as excitatory transmitters in the brain in a manner similar to that of aspartate.

The present structural investigation was undertaken as part of our study of the conformation and hydrogen bonding of this biologically important molecule.

Experimental. Synthesis described by Soroka & Mastalerz (1976). Clear, colourless crystals from water at room temperature, dimensions 0·15 × 0·2 × 0·3 mm; density by flotation in carbon tetrachloride/ethylene bromide; monoclinic Cc or $C2/c$ from Weissenberg photographs, $C2/c$ confirmed by refinement; Syntex $P2_1$ computer-controlled four-circle diffractometer, Cu $K\alpha$ radiation, scintillation counter, graphite monochromator; cell parameters by least squares from setting angles of 15 reflexions with $25 \le 2\theta \le 36^\circ$ measured on the diffractometer; 803 independent reflexions; $2\theta_{\max} = 114^\circ$; variable θ–2θ scans, scan rate

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