O2—C1—O1	126.6 (3)	O4—C3—C4	112.0 (3)
O2—C1—C2	117.4 (3)	C4—C3—C2	110.0 (3)
O1—C1—C2	116.0 (3)	O5—C4—O6	125.3 (3)
O3—C2—C1	113.2 (3)	O5—C4—C3	121.7 (3)
O3—C2—C3	107.4 (3)	O6—C4—C3	113.0 (3)
C1—C2—C3	108.4 (3)	N1—C5—C6	110.7 (3)
O4—C3—C2	111.6 (3)	N2—C6—C5	110.6 (3)
O4—C3—C4—O5	−23.5 (4)	O1—C1—C2—C3	105.8 (4)
C2—C3—C4—O5	101.1 (4)	O4—C3—C2—O3	58.0 (3)
O4—C3—C4—O6	156.5 (3)	C4—C3—C2—O3	−67.0 (3)
C2—C3—C4—O6	−78.8 (3)	O4—C3—C2—C1	−64.7 (3)
O2—C1—C2—O3	167.4 (3)	C4—C3—C2—C1	170.4 (2)
O1—C1—C2—O3	−13.3 (5)	N1—C5—C6—N2	−60.6 (5)
O2—C1—C2—C3	−73.5 (4)		

Table 3. *Least-squares-planes data*

Plane 1: C1, C2, C3, C4; equation: $-0.375\,(30)x + 2.477\,(11)y + 14.815\,(6)z = 9.121\,(18)$. Plane 2: C1, C2, O1, O2, O3; equation: $6.589\,(4)x - 1.360\,(15)y + 6.723\,(22)z = 4.785\,(20)$. Plane 3: C3, C4, O4, O5, O6; equation: $-6.851\,(3)x - 2.909\,(9)y - 1.343\,(32)z = 2.066\,(15)$.

Dihedral angles (°)

Plane 1/Plane 2	72.37 (24)	Plane 2/Plane 3	32.16 (12)
Plane 1/Plane 3	75.49 (25)		

Deviations of atoms (Å) from their least-squares planes

	Plane 1	Plane 2	Plane 3
C1	0.060 (2)	−0.012 (3)	
C2	−0.058 (2)	−0.109 (2)	
C3	−0.063 (2)		−0.199 (2)
C4	0.061 (2)		−0.032 (3)
O1		−0.035 (1)	
O2		0.068 (2)	
O3		0.089 (2)	
O4			0.158 (2)
O5			−0.049 (1)
O6			0.123 (2)

H atoms, except H1, H2*A* and H2*B*, were obtained by difference Fourier syntheses and were refined isotropically. Atoms H1, H2*A* and H2*B* were located geometrically.

Data collection: *CAD*-4-*PC* (Enraf–Nonius, 1993). Cell refinement: *CAD*-4-*PC*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1121). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-Acetoxymethylpyrrole-2-carbaldehyde

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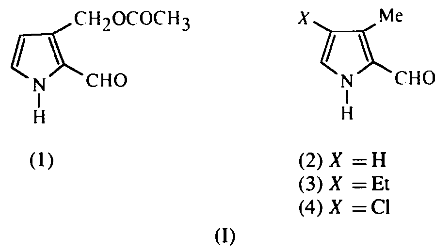
Abstract

In the title compound (the ester 2-formyl-3-pyrrolylmethyl acetate, $C_8H_9NO_3$), hydrogen bonding between the N—H function in one molecule and the ester carbonyl O atom in a molecule related to the first by the 2_1 screw axis leads to the formation of zigzag chains.

Comment

3-Acetoxymethylpyrrole-2-carbaldehyde, (1), was prepared as an intermediate in a new synthetic route to 3,8-didehydroheliotridin-5-one (McNab & Thornley, 1993). Because of interest in the crystal structures of 3-substituted pyrrole-2-carbaldehyde derivatives [(2)–(4)] (Smith, Bobe, Minnetian, Hope & Yanuck, 1985), we undertook a determination of its structure at 150 K.



(1)

(2) X = H
(3) X = Et
(4) X = Cl

(I)

Ring bond lengths in (1) follow the same pattern as seen in compounds (2)–(4) (Smith *et al.*, 1985). Thus, the substitution at the 2 and 3 positions of the pyrrole ring in all four compounds is reflected in the C2—C3 bond being consistently longer than the C4—C5 bond. Similarly, the N1—C2 bond in (1) is significantly longer than that between N1 and C5, a trend also observed in (2)–(4); this can be rationalized by conjugative electron donation from the lone pair of the N atom to the aldehyde function.

The ring angles in (1) are very similar to those in the methyl analogue (2), being widest at N1 and C5. The angles to each substituent are widened in the direction of the other substituent, particularly at C2 where the C3—C2—C21 angle is 130.6 (3)°.

The O atom (O21) of the aldehyde function is twisted so as to lie 0.167 (5) Å from the plane of the pyrrole ring. The acetoxymethyl substituent (C6–C9) is essentially planar and its normal forms an angle of 76.42 (11)° with the normal to the pyrrole ring.

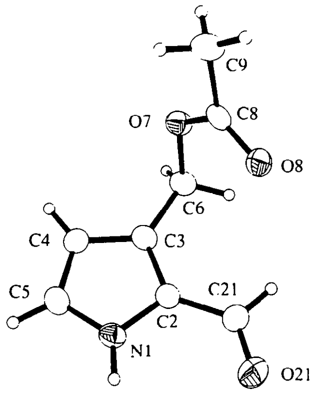


Fig. 1. A view of the molecule with atom-numbering scheme. Displacement ellipsoids enclose 50% probability surfaces.

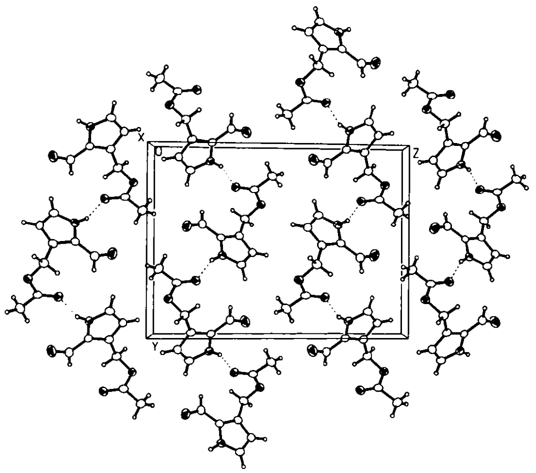


Fig. 2. A packing diagram of the structure showing intermolecular hydrogen bonding between the N—H group and the carbonyl O atom of the ester function.

As can be seen from Fig. 2, the structure exhibits intermolecular hydrogen bonding between the N—H function and the carbonyl O atom of the ester function. These interactions link molecules related by the operation of the 2_1 screw axis into zigzag chains.

Experimental

The title compound was prepared by photolysis of 4-acetoxymethylpyridine *N*-oxide in aqueous copper(II) sulfate solution. Crystals could be obtained by slow evaporation from either ethanol or toluene.

Crystal data

$C_8H_9NO_3$
$M_r = 167.16$
Monoclinic
$P2_1/n$
$a = 4.463\ (3)$ Å
$b = 11.546\ (5)$ Å
$c = 15.697\ (5)$ Å
$\beta = 95.75\ (5)°$
$V = 804.8\ (7)$ Å^3
$Z = 4$
$D_x = 1.380$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 24 reflections
$\theta = 13–16°$
$\mu = 0.107$ mm^{-1}
$T = 150.0\ (2)$ K
Irregular block
0.43 × 0.27 × 0.19 mm
Colourless

Data collection

Stoe Stadi-4 four-circle diffractometer
$\omega–2\theta$ scans using on-line profile fitting (Clegg, 1981)
Absorption correction: none
1492 measured reflections
1404 independent reflections
1015 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.0402$
$\theta_{\rm max} = 25.01°$
$h = -5 \rightarrow 5$
$k = -2 \rightarrow 13$
$l = -11 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0538$
$wR(F^2) = 0.1703$
$S = 0.983$
1392 reflections
146 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0611P)^2 + 1.37P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.03$

$\Delta\rho_{\rm max} = 0.23$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.27$ e Å^{-3}
Extinction correction: *SHELXL93* (Sheldrick, 1993)
Extinction coefficient: 0.021 (6)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
N1	0.6335 (6)	−0.0829 (2)	0.7734 (2)	0.0267 (6)
C2	0.4518 (7)	0.0135 (3)	0.7640 (2)	0.0244 (7)
C3	0.3078 (6)	0.0242 (3)	0.8381 (2)	0.0238 (7)

C4	0.4070 (7)	−0.0675 (3)	0.8923 (2)	0.0284 (7)
C5	0.6045 (7)	−0.1319 (3)	0.8503 (2)	0.0295 (7)
C6	0.0956 (7)	0.1186 (3)	0.8591 (2)	0.0272 (7)
O7	0.2367 (5)	0.1971 (2)	0.92455 (13)	0.0284 (6)
C8	0.4232 (7)	0.2779 (2)	0.8981 (2)	0.0254 (7)
O8	0.4685 (5)	0.2888 (2)	0.82381 (13)	0.0305 (6)
C9	0.5695 (10)	0.3479 (3)	0.9704 (2)	0.0393 (9)
C21	0.4430 (8)	0.0862 (3)	0.6898 (2)	0.0318 (8)
O21	0.6045 (6)	0.0738 (2)	0.63177 (15)	0.0456 (7)

Table 2. *Selected geometric parameters* (Å, °)

N1—C5	1.351 (4)	C4—C5	1.370 (5)
N1—C2	1.377 (4)	C6—O7	1.464 (4)
C2—C3	1.389 (4)	O7—C8	1.344 (4)
C2—C21	1.433 (4)	C8—O8	1.209 (4)
C3—C4	1.402 (4)	C8—C9	1.490 (5)
C3—C6	1.502 (4)	C21—O21	1.225 (4)
C5—N1—C2	109.0 (3)	N1—C5—C4	109.1 (3)
N1—C2—C3	107.4 (3)	O7—C6—C3	111.5 (3)
N1—C2—C21	121.9 (3)	C8—O7—C6	116.7 (2)
C3—C2—C21	130.6 (3)	O8—C8—O7	122.6 (3)
C2—C3—C4	107.2 (3)	O8—C8—C9	125.2 (3)
C2—C3—C6	127.1 (3)	O7—C8—C9	112.2 (3)
C4—C3—C6	125.7 (3)	O21—C21—C2	124.5 (3)
C5—C4—C3	107.3 (3)		
C2—C3—C6—O7	−107.1 (3)	C6—O7—C8—C9	−176.3 (3)
C4—C3—C6—O7	70.0 (4)	N1—C2—C21—O21	−4.4 (5)
C3—C6—O7—C8	79.4 (3)	C3—C2—C21—O21	172.3 (3)
C6—O7—C8—O8	2.1 (4)		

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1992).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1024). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dimethyl [α-(Benzylamino)-*p*-chlorobenzyl]phosphonate

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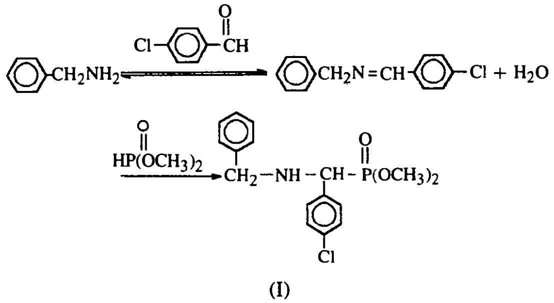
Abstract

The title compound, $C_{16}H_{19}ClNO_3P$, is one of a series of α-substituted aminomethyl phosphonates. There are two independent molecules in the asymmetric unit and in each the P atom adopts a tetrahedral configuration. The P═O double-bond length and the mean P—O single-bond length are 1.425 (5) and 1.560 (5) Å for molecule (1) and 1.417 (6) and 1.562 (5) Å for molecule (2), respectively. The P═O double-bond lengths in both molecules are shorter than those in 1,3,2-oxazaphospholidine derivatives and caged bicyclic phosphorus compounds.

Comment

α-Substituted aminomethyl phosphonates have been investigated (Li, Wang, Zhang, Miao, Liu, Cao & Guo, 1988; Li, Wang, Han, Liu, Cao, Jiang, Miao & Liu, 1988) because of their potential biological activity. The title compound, (I), was synthesized and its crystal structure is presented here.



(I)

The asymmetric unit contains two independent molecules. The P atom adopts a distorted tetrahedral configuration; the bond angles around the P atom are in the range 100.9 (2)–116.1 (3)° for molecule (1) and 97.8 (3)–119.3 (3)° for molecule (2). The mean P—O

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