

## Dimethyl 4-ethoxy-1-(4-methyl-2-pyridyl)-5-oxo-2,5-dihydro-1H-pyrrole-2,3-dicarboxylate

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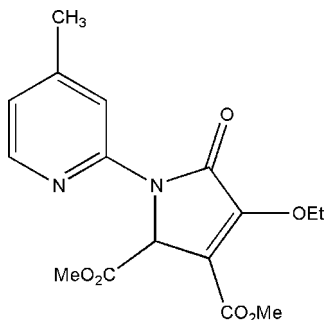
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Key indicators: single-crystal X-ray study;  $T = 120$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å; disorder in main residue;  $R$  factor = 0.043;  $wR$  factor = 0.119; data-to-parameter ratio = 18.8.

In the title compound,  $\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}_6$ , the dihedral angle between the aromatic ring planes is  $8.11(6)^\circ$ . One of the O atoms is disordered over two sites of equal occupancy. In the crystal structure, aromatic  $\pi$ - $\pi$  stacking [centroid-to-centroid separation =  $3.5503(8)$  Å] helps to consolidate the packing.

### Related literature

For background on 3-pyrrolines as synthetic intermediates, see: Tarnchompoo *et al.* (1987); Bienz *et al.* (1989). For further synthetic details, see: Anary-Abbasinejad *et al.* (2010).



### Experimental

#### Crystal data

$\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}_6$	$\gamma = 61.797(1)^\circ$
$M_r = 334.32$	$V = 781.73(9)$ Å <sup>3</sup>
Triclinic, $P\bar{1}$	$Z = 2$
$a = 9.2674(6)$ Å	Mo $K\alpha$ radiation
$b = 9.4219(6)$ Å	$\mu = 0.11$ mm <sup>-1</sup>
$c = 10.7650(7)$ Å	$T = 120$ K
$\alpha = 87.692(2)^\circ$	$0.22 \times 0.19 \times 0.15$ mm
$\beta = 72.037(1)^\circ$	

#### Data collection

Bruker SMART 1000 CCD diffractometer	14075 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 1998)	4340 independent reflections
$T_{\min} = 0.976$ , $T_{\max} = 0.984$	3811 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.020$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$	8 restraints
$wR(F^2) = 0.119$	H-atom parameters constrained
$S = 1.00$	$\Delta\rho_{\text{max}} = 0.37$ e Å <sup>-3</sup>
4340 reflections	$\Delta\rho_{\text{min}} = -0.29$ e Å <sup>-3</sup>
231 parameters	

Data collection: SMART (Bruker, 1998); cell refinement: SAINT-Plus (Bruker, 1998); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

The authors are grateful to Islamic Azad University, Yazd Branch, for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5395).

### References

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**supplementary materials**

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## Dimethyl 4-ethoxy-1-(4-methyl-2-pyridyl)-5-oxo-2,5-dihydro-1H-pyrrole-2,3-dicarboxylate

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### Comment

(*N*-Substituted 3-pyrrolines serve as useful synthetic intermediates (Tarnchompoo *et al.*, 1987; Bienz *et al.*, 1989). Recently we reported a one-pot procedure for the synthesis of some dialkyl *N*-(3-methyl-2-pyridyl)-4-ethoxy-5-oxo-2,5-dihydro-1H-pyrrole-2,3-dicarboxylate derivatives (Anary-Abbasinejad *et al.*, 2010).

Here we report the synthesis and crystal structure of the title compound, (I). It is rational to assume that compound **1** (Fig. 1) is produced from the initial production of ylide intermediate produced from three-component reaction of DMAD, 4-methyl-2-aminopyridine and triphenylphosphine, which then reacted with ethyl chlorooxoacetate to produce an oxamate derivative that underwent intramolecular Wittig reaction to give the products. Crystal packing of **I** is shown in Fig. 2. A considerable feature of the compound (I) is the presence of  $\pi$ - $\pi$  stacking interactions between six and five membered rings (Fig. 3) with distance 3.5503 (8) Å for  $Cg2 \cdots Cg3$  ( $Cg2 = N1/C10-C17$  and  $Cg3 = N2/C9-C12$ ).

### Experimental

To a magnetically stirred solution of  $PPh_3$  (0.26 g, 1 mmol) and 4-methyl-2-aminopyridine (0.9 g, 1 mmol) in  $CH_2Cl_2$  (10 ml) was added drop-wise a mixture of dimethyl acetylenedicarboxylate DMAD (0.14 g, 1 mmol) in  $CH_2Cl_2$  (3 ml) at room temperature over 2 min. The reaction mixture was then stirred for one more minute, then triethylamine (1 mmol) and ethyl chlorooxoacetate (1 mmol) was added and the reaction mixture was stirred for more 24 h. Solvent was evaporated and the residue was purified by column chromatography on  $SiO_2$  using EtOAC-hexane (1:4) mixture as eluent. The solid formed was filtrated, recrystallised from dichloromethane/ethanol (2:1) to yield colourless prisms of (I).

### Refinement

The H(C) atoms were placed in calculated positions and refined as riding with  $U_{iso}(H) = 1.2U_{eq}(C)$ .

### Figures

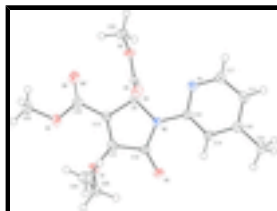


Fig. 1. The molecular structure of (I). Atoms are represented by 50% displacement ellipsoids. The occupancies of positions of disordered O6 atom are equal (0.5).

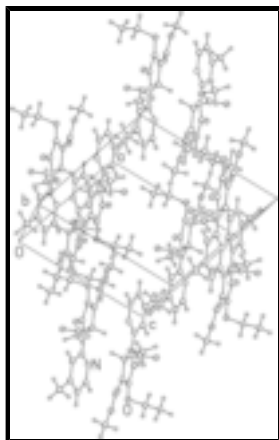


Fig. 2. The overview of I crystal packing. Atoms are represented by spheres.

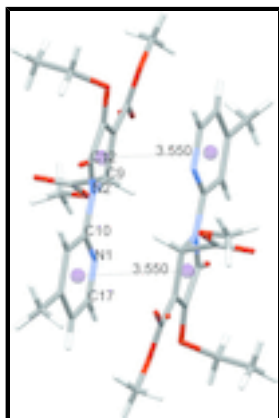


Fig. 3. Representation of  $\pi$ - $\pi$  stacking in I.

**Dimethyl 4-ethoxy-1-(4-methyl-2-pyridyl)-5-oxo-2,5-dihydro- 1H-pyrrole-2,3-dicarboxylate**

*Crystal data*

$C_{16}H_{18}N_2O_6$

$M_r = 334.32$

Triclinic,  $PT$

Hall symbol:  $-P 1$

$a = 9.2674 (6) \text{ \AA}$

$b = 9.4219 (6) \text{ \AA}$

$c = 10.7650 (7) \text{ \AA}$

$\alpha = 87.692 (2)^\circ$

$\beta = 72.037 (1)^\circ$

$\gamma = 61.797 (1)^\circ$

$V = 781.73 (9) \text{ \AA}^3$

$Z = 2$

$F(000) = 352$

$D_x = 1.420 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 259 reflections

$\theta = 3\text{--}29^\circ$

$\mu = 0.11 \text{ mm}^{-1}$

$T = 120 \text{ K}$

Prism, colourless

$0.22 \times 0.19 \times 0.15 \text{ mm}$

*Data collection*

Bruker SMART 1000 CCD  
diffractometer

Radiation source: fine-focus sealed tube  
graphite

4340 independent reflections

3811 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.020$

$\omega$  scans  $\theta_{\max} = 29.5^\circ$ ,  $\theta_{\min} = 2.0^\circ$   
 Absorption correction: multi-scan  $h = -12 \rightarrow 12$   
 (SADABS; Bruker, 1998)  
 $T_{\min} = 0.976$ ,  $T_{\max} = 0.984$   $k = -13 \rightarrow 13$   
 14075 measured reflections  $l = -14 \rightarrow 14$

### Refinement

Refinement on  $F^2$  Primary atom site location: structure-invariant direct methods  
 Least-squares matrix: full Secondary atom site location: difference Fourier map  
 $R[F^2 > 2\sigma(F^2)] = 0.043$  Hydrogen site location: inferred from neighbouring sites  
 $wR(F^2) = 0.119$  H-atom parameters constrained  
 $S = 1.00$   $w = 1/[\sigma^2(F_o^2) + (0.0685P)^2 + 0.3063P]$   
 4340 reflections where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 231 parameters  $\Delta\rho_{\max} = 0.37 \text{ e } \text{\AA}^{-3}$   
 8 restraints  $\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$

### Special details

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) etc. and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.17590 (10)	0.52219 (10)	0.39546 (8)	0.02303 (17)	
O2	0.40451 (10)	0.95161 (9)	0.14840 (8)	0.02268 (17)	
O3	0.51098 (11)	0.79492 (10)	0.29618 (8)	0.02475 (18)	
N1	0.71510 (11)	0.58162 (11)	-0.02268 (9)	0.01973 (18)	
O4	-0.06090 (10)	0.84294 (10)	0.37603 (9)	0.02895 (19)	
O5	0.54578 (10)	0.30748 (9)	0.25074 (8)	0.02478 (18)	
N2	0.52930 (11)	0.53670 (10)	0.15196 (8)	0.01807 (17)	
C8	0.44689 (12)	0.82101 (12)	0.21038 (10)	0.01806 (19)	
C9	0.39637 (12)	0.70530 (12)	0.16169 (10)	0.01788 (19)	
H9A	0.3761	0.7310	0.0756	0.021*	
C10	0.69758 (12)	0.47752 (12)	0.06007 (9)	0.01754 (19)	
C11	1.01376 (13)	0.37860 (13)	-0.12410 (10)	0.0217 (2)	
H11A	1.1218	0.3474	-0.1911	0.026*	

## supplementary materials

C12	0.46748 (13)	0.45026 (12)	0.23786 (10)	0.01882 (19)	
C13	0.23818 (13)	0.71222 (12)	0.26432 (10)	0.01865 (19)	
C14	0.83205 (13)	0.32265 (12)	0.05951 (10)	0.0201 (2)	
H14A	0.8133	0.2538	0.1220	0.024*	
C15	0.28055 (13)	0.56764 (12)	0.30958 (10)	0.0190 (2)	
C16	0.99386 (13)	0.27226 (13)	-0.03493 (10)	0.0209 (2)	
C17	0.87312 (13)	0.53027 (13)	-0.11305 (10)	0.0216 (2)	
H17A	0.8889	0.6027	-0.1729	0.026*	
C18	0.07123 (14)	0.86461 (13)	0.30661 (11)	0.0230 (2)	
C20	0.23239 (15)	0.44492 (14)	0.50530 (11)	0.0242 (2)	
H20A	0.3508	0.3514	0.4706	0.029*	
H20B	0.1538	0.4040	0.5559	0.029*	
C21	1.14368 (15)	0.10896 (14)	-0.03822 (13)	0.0295 (2)	
H21A	1.2431	0.1216	-0.0393	0.044*	
H21B	1.1749	0.0388	-0.1175	0.044*	
H21C	1.1108	0.0598	0.0400	0.044*	
C22	0.40700 (15)	1.08725 (13)	0.20518 (12)	0.0263 (2)	
H22A	0.4173	1.1584	0.1380	0.039*	
H22B	0.5061	1.0464	0.2372	0.039*	
H22C	0.2993	1.1485	0.2787	0.039*	
C23	-0.22672 (15)	0.98915 (16)	0.42475 (13)	0.0347 (3)	
H23A	-0.3177	0.9600	0.4659	0.052*	
H23B	-0.2518	1.0504	0.3514	0.052*	
H23C	-0.2230	1.0560	0.4899	0.052*	
C24	0.23145 (19)	0.56449 (17)	0.59437 (13)	0.0351 (3)	
H24A	0.2595	0.5142	0.6712	0.053*	
H24B	0.1163	0.6606	0.6235	0.053*	
H24C	0.3179	0.5966	0.5465	0.053*	
O6'	0.0517 (16)	0.9913 (13)	0.2663 (9)	0.0417 (17)	0.50
O6	0.0634 (17)	0.9971 (12)	0.2975 (10)	0.0393 (15)	0.50

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0207 (4)	0.0295 (4)	0.0238 (4)	-0.0162 (3)	-0.0077 (3)	0.0093 (3)
O2	0.0264 (4)	0.0174 (3)	0.0276 (4)	-0.0124 (3)	-0.0107 (3)	0.0065 (3)
O3	0.0280 (4)	0.0229 (4)	0.0262 (4)	-0.0124 (3)	-0.0126 (3)	0.0039 (3)
N1	0.0194 (4)	0.0210 (4)	0.0196 (4)	-0.0107 (3)	-0.0058 (3)	0.0030 (3)
O4	0.0168 (4)	0.0254 (4)	0.0354 (4)	-0.0078 (3)	-0.0006 (3)	0.0022 (3)
O5	0.0237 (4)	0.0182 (4)	0.0318 (4)	-0.0108 (3)	-0.0076 (3)	0.0060 (3)
N2	0.0161 (4)	0.0154 (4)	0.0202 (4)	-0.0072 (3)	-0.0033 (3)	0.0018 (3)
C8	0.0155 (4)	0.0157 (4)	0.0198 (4)	-0.0070 (3)	-0.0028 (3)	0.0017 (3)
C9	0.0168 (4)	0.0166 (4)	0.0192 (4)	-0.0078 (4)	-0.0049 (3)	0.0024 (3)
C10	0.0175 (4)	0.0184 (4)	0.0175 (4)	-0.0097 (4)	-0.0048 (3)	0.0001 (3)
C11	0.0188 (4)	0.0264 (5)	0.0197 (4)	-0.0118 (4)	-0.0044 (3)	0.0003 (4)
C12	0.0195 (4)	0.0188 (4)	0.0204 (4)	-0.0111 (4)	-0.0063 (3)	0.0024 (3)
C13	0.0168 (4)	0.0204 (5)	0.0190 (4)	-0.0096 (4)	-0.0051 (3)	0.0017 (3)
C14	0.0192 (4)	0.0181 (4)	0.0217 (5)	-0.0086 (4)	-0.0059 (4)	0.0015 (3)

C15	0.0181 (4)	0.0213 (5)	0.0202 (4)	-0.0118 (4)	-0.0059 (3)	0.0030 (3)
C16	0.0183 (4)	0.0208 (5)	0.0223 (5)	-0.0082 (4)	-0.0065 (4)	-0.0008 (4)
C17	0.0215 (5)	0.0250 (5)	0.0196 (4)	-0.0128 (4)	-0.0061 (4)	0.0037 (4)
C18	0.0197 (5)	0.0227 (5)	0.0223 (5)	-0.0085 (4)	-0.0044 (4)	0.0024 (4)
C20	0.0246 (5)	0.0249 (5)	0.0242 (5)	-0.0133 (4)	-0.0081 (4)	0.0090 (4)
C21	0.0199 (5)	0.0238 (5)	0.0350 (6)	-0.0051 (4)	-0.0052 (4)	0.0027 (4)
C22	0.0280 (5)	0.0168 (5)	0.0329 (6)	-0.0120 (4)	-0.0065 (4)	0.0016 (4)
C23	0.0176 (5)	0.0322 (6)	0.0358 (6)	-0.0034 (5)	0.0009 (4)	0.0014 (5)
C24	0.0472 (7)	0.0378 (7)	0.0276 (6)	-0.0245 (6)	-0.0155 (5)	0.0082 (5)
O6'	0.026 (2)	0.026 (2)	0.051 (4)	-0.0063 (13)	0.004 (2)	0.014 (2)
O6	0.0263 (16)	0.0202 (13)	0.058 (4)	-0.0087 (11)	-0.002 (3)	0.004 (2)

*Geometric parameters (Å, °)*

O1—C15	1.3364 (12)	C13—C18	1.4769 (14)
O1—C20	1.4679 (13)	C14—C16	1.3904 (14)
O2—C8	1.3238 (12)	C14—H14A	0.9500
O2—C22	1.4515 (13)	C16—C21	1.5004 (15)
O3—C8	1.2035 (13)	C17—H17A	0.9500
N1—C10	1.3315 (13)	C18—O6'	1.204 (10)
N1—C17	1.3471 (13)	C18—O6	1.218 (10)
O4—C18	1.3263 (13)	C20—C24	1.5037 (17)
O4—C23	1.4472 (14)	C20—H20A	0.9900
O5—C12	1.2165 (13)	C20—H20B	0.9900
N2—C12	1.3785 (12)	C21—H21A	0.9800
N2—C10	1.4115 (12)	C21—H21B	0.9800
N2—C9	1.4615 (12)	C21—H21C	0.9800
C8—C9	1.5369 (14)	C22—H22A	0.9800
C9—C13	1.5134 (13)	C22—H22B	0.9800
C9—H9A	1.0000	C22—H22C	0.9800
C10—C14	1.3999 (14)	C23—H23A	0.9800
C11—C17	1.3830 (15)	C23—H23B	0.9800
C11—C16	1.3954 (15)	C23—H23C	0.9800
C11—H11A	0.9500	C24—H24A	0.9800
C12—C15	1.5011 (14)	C24—H24B	0.9800
C13—C15	1.3440 (14)	C24—H24C	0.9800
C15—O1—C20	116.43 (8)	N1—C17—H17A	118.0
C8—O2—C22	116.22 (9)	C11—C17—H17A	118.0
C10—N1—C17	116.47 (9)	O6'—C18—O4	122.5 (6)
C18—O4—C23	115.57 (9)	O6—C18—O4	123.7 (6)
C12—N2—C10	127.04 (9)	O6'—C18—C13	123.4 (6)
C12—N2—C9	112.20 (8)	O6—C18—C13	121.9 (6)
C10—N2—C9	120.74 (8)	O4—C18—C13	113.18 (9)
O3—C8—O2	126.28 (10)	O1—C20—C24	110.36 (9)
O3—C8—C9	123.12 (9)	O1—C20—H20A	109.6
O2—C8—C9	110.56 (8)	C24—C20—H20A	109.6
N2—C9—C13	102.82 (8)	O1—C20—H20B	109.6
N2—C9—C8	110.34 (8)	C24—C20—H20B	109.6
C13—C9—C8	110.24 (8)	H20A—C20—H20B	108.1

## supplementary materials

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N2—C9—H9A	111.1	C16—C21—H21A	109.5
C13—C9—H9A	111.1	C16—C21—H21B	109.5
C8—C9—H9A	111.1	H21A—C21—H21B	109.5
N1—C10—C14	124.23 (9)	C16—C21—H21C	109.5
N1—C10—N2	114.20 (9)	H21A—C21—H21C	109.5
C14—C10—N2	121.57 (9)	H21B—C21—H21C	109.5
C17—C11—C16	118.70 (9)	O2—C22—H22A	109.5
C17—C11—H11A	120.7	O2—C22—H22B	109.5
C16—C11—H11A	120.7	H22A—C22—H22B	109.5
O5—C12—N2	127.50 (10)	O2—C22—H22C	109.5
O5—C12—C15	126.81 (9)	H22A—C22—H22C	109.5
N2—C12—C15	105.66 (8)	H22B—C22—H22C	109.5
C15—C13—C18	129.15 (9)	O4—C23—H23A	109.5
C15—C13—C9	109.65 (9)	O4—C23—H23B	109.5
C18—C13—C9	121.10 (9)	H23A—C23—H23B	109.5
C16—C14—C10	118.22 (10)	O4—C23—H23C	109.5
C16—C14—H14A	120.9	H23A—C23—H23C	109.5
C10—C14—H14A	120.9	H23B—C23—H23C	109.5
O1—C15—C13	127.97 (9)	C20—C24—H24A	109.5
O1—C15—C12	122.13 (9)	C20—C24—H24B	109.5
C13—C15—C12	109.62 (9)	H24A—C24—H24B	109.5
C14—C16—C11	118.34 (10)	C20—C24—H24C	109.5
C14—C16—C21	120.49 (10)	H24A—C24—H24C	109.5
C11—C16—C21	121.16 (10)	H24B—C24—H24C	109.5
N1—C17—C11	124.02 (10)		
C22—O2—C8—O3	-11.71 (15)	C20—O1—C15—C13	-130.46 (11)
C22—O2—C8—C9	166.06 (8)	C20—O1—C15—C12	56.26 (13)
C12—N2—C9—C13	-1.63 (11)	C18—C13—C15—O1	7.41 (18)
C10—N2—C9—C13	176.88 (8)	C9—C13—C15—O1	-176.23 (10)
C12—N2—C9—C8	115.93 (9)	C18—C13—C15—C12	-178.63 (10)
C10—N2—C9—C8	-65.56 (11)	C9—C13—C15—C12	-2.27 (11)
O3—C8—C9—N2	-40.90 (13)	O5—C12—C15—O1	-2.48 (16)
O2—C8—C9—N2	141.24 (8)	N2—C12—C15—O1	175.60 (9)
O3—C8—C9—C13	71.98 (12)	O5—C12—C15—C13	-176.85 (10)
O2—C8—C9—C13	-105.87 (9)	N2—C12—C15—C13	1.22 (11)
C17—N1—C10—C14	1.13 (15)	C10—C14—C16—C11	0.02 (15)
C17—N1—C10—N2	-179.48 (8)	C10—C14—C16—C21	178.63 (9)
C12—N2—C10—N1	171.31 (9)	C17—C11—C16—C14	1.30 (15)
C9—N2—C10—N1	-6.97 (13)	C17—C11—C16—C21	-177.30 (10)
C12—N2—C10—C14	-9.29 (16)	C10—N1—C17—C11	0.34 (15)
C9—N2—C10—C14	172.43 (9)	C16—C11—C17—N1	-1.56 (16)
C10—N2—C12—O5	0.04 (17)	C23—O4—C18—O6'	-12.9 (5)
C9—N2—C12—O5	178.45 (10)	C23—O4—C18—O6	9.6 (5)
C10—N2—C12—C15	-178.01 (9)	C23—O4—C18—C13	177.55 (10)
C9—N2—C12—C15	0.39 (11)	C15—C13—C18—O6'	173.7 (5)
N2—C9—C13—C15	2.39 (11)	C9—C13—C18—O6'	-2.3 (5)
C8—C9—C13—C15	-115.24 (9)	C15—C13—C18—O6	151.4 (5)
N2—C9—C13—C18	179.09 (9)	C9—C13—C18—O6	-24.6 (5)
C8—C9—C13—C18	61.45 (12)	C15—C13—C18—O4	-16.81 (16)



N1—C10—C14—C16	-1.31 (16)	C9—C13—C18—O4	167.21 (9)
N2—C10—C14—C16	179.34 (9)	C15—O1—C20—C24	66.34 (12)

Fig. 1

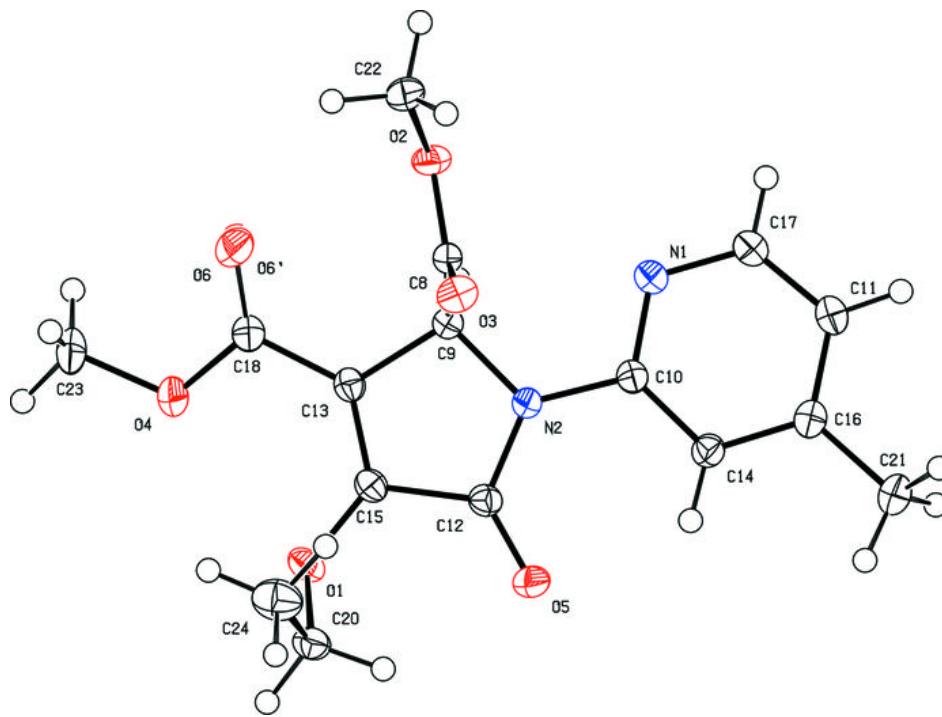


Fig. 2

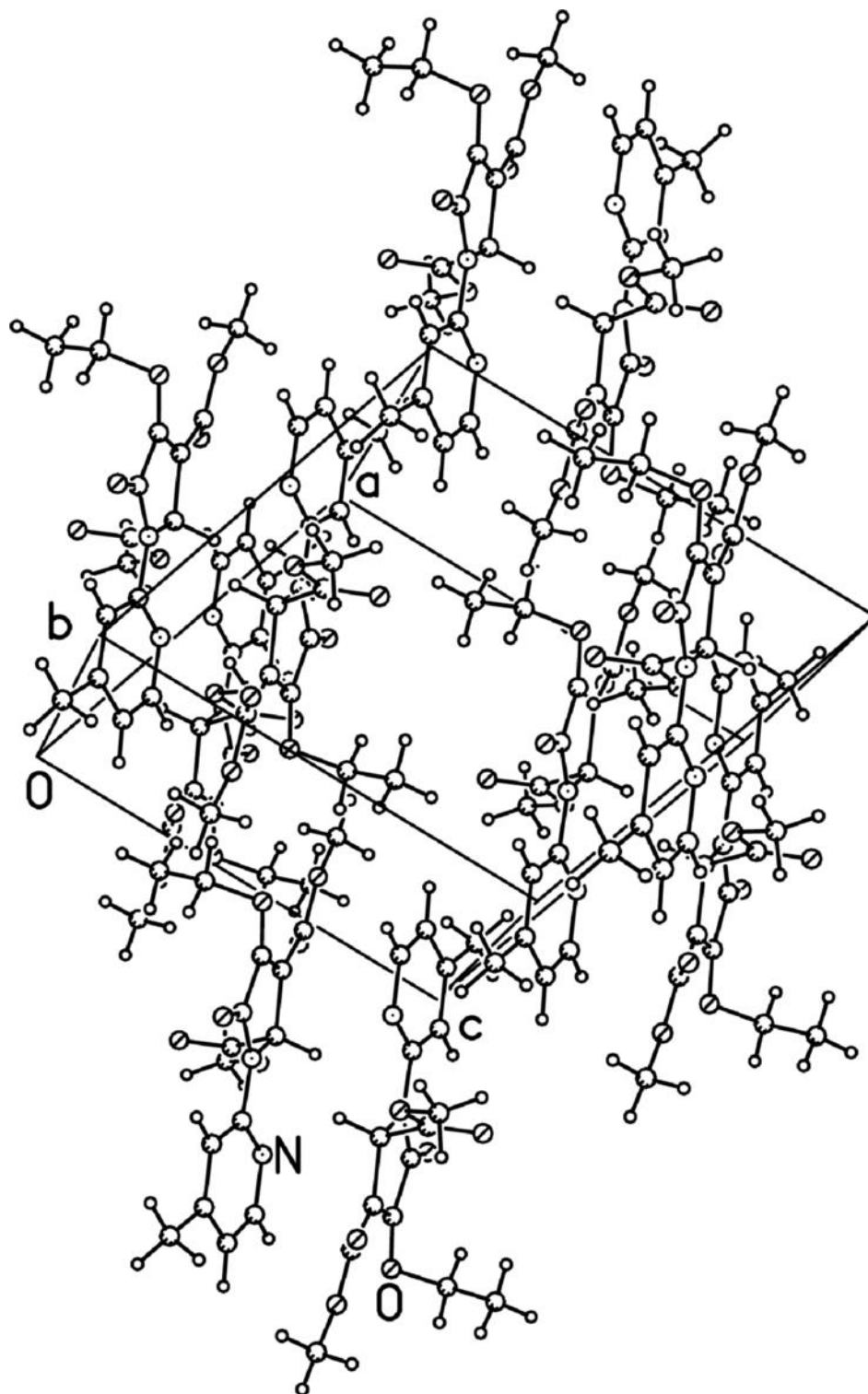


Fig. 3

