

## Ethyl 3,4-dimethyl-5-[(*E*)-(phenylimino)-methyl]-1*H*-pyrrole-2-carboxylate

Wei-Na Wu,<sup>a\*</sup> Lei Yang,<sup>a</sup> Xiao-Xia Li,<sup>b</sup> Bao-Feng Qin<sup>c</sup> and Qiu-Fen Wang<sup>a</sup>

<sup>a</sup>Department of Physics and Chemistry, Henan Polytechnic University, Jiaozuo 454000, People's Republic of China, <sup>b</sup>Institute of Functional Materials, Jiangxi University of Finance & Economics, Nanchang 330013, People's Republic of China, and <sup>c</sup>Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou 730000, People's Republic of China  
Correspondence e-mail: wuw08@hpu.edu.cn

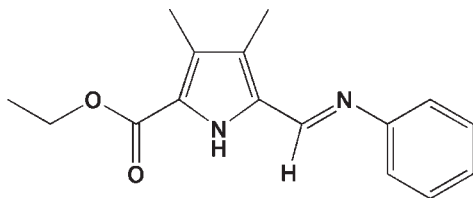
Received 12 April 2010; accepted 9 June 2010

Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.044;  $wR$  factor = 0.144; data-to-parameter ratio = 18.5.

In the title compound,  $\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}_2$ , the molecule adopts an *E* conformation about the  $\text{C}=\text{N}$  double bond. The dihedral angle between the pyrrole and phenyl rings is  $41.55(8)^\circ$ . In the crystal structure, pairs of intermolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds link the molecules into centrosymmetric dimers. In the dimer, the two pyrrole rings are almost coplanar and the two phenyl rings are parallel to each other.

### Related literature

For the structure of 5-formyl-3,4-dimethyl-1*H*-pyrrole-2-carboxylate, see Wu *et al.* (2009). For the similar structure of ethyl 5-[(2,3-dimethyl-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrazol-4-yl)iminomethyl]-3,4-dimethyl-1*H*-pyrrole-2-carboxylate, see Wang *et al.* (2009). For the coordination abilities for metal ions of pyrrol-2-ylmethyleneamine ligands, see: Wang *et al.* (2010); Yang *et al.* (2003).



### Experimental

#### Crystal data

$\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}_2$   
 $M_r = 270.32$   
Monoclinic,  $P2_1/c$   
 $a = 12.5463(7)$  Å  
 $b = 14.6525(9)$  Å  
 $c = 8.4490(5)$  Å  
 $\beta = 105.042(3)^\circ$   
 $V = 1500.00(15)$  Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.08$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.35 \times 0.26 \times 0.18$  mm

#### Data collection

Bruker SMART CCD diffractometer  
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.975$ ,  $T_{\max} = 0.986$   
12405 measured reflections  
3413 independent reflections  
2078 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.030$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$   
 $wR(F^2) = 0.144$   
 $S = 1.01$   
3413 reflections  
184 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.20$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.16$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2A}\cdots\text{O3}^i$	0.86	2.06	2.8883 (18)	162

Symmetry code: (i)  $-x, -y, -z$ .

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

The authors are grateful for financial support from the Doctoral Foundation of Henan Polytechnic University (B2009-65 648359 and B2009-70 648364).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2080).

### References

- Bruker (1997). SMART, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.  
Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
Wang, Y., Wu, W.-N. & Wang, Q.-F. (2009). *Acta Cryst.* **E65**, o1933.  
Wang, Y., Wu, W.-N., Wang, Q. & Yang, Z.-Y. (2010). *J. Coord. Chem.* **63**, 147–155.  
Wu, W.-N., Wang, Y. & Wang, Q.-F. (2009). *Acta Cryst.* **E65**, o1661.  
Yang, L. Y., Chen, Q. Q., Yang, G. Q. & Ma, J. S. (2003). *Tetrahedron*, **59**, 10037–10041.

**supplementary materials**

*Acta Cryst.* (2010). E66, o1655 [ doi:10.1107/S1600536810022051 ]

## Ethyl 3,4-dimethyl-5-[(*E*)-(phenylimino)methyl]-1*H*-pyrrole-2-carboxylate

W.-N. Wu, L. Yang, X.-X. Li, B.-F. Qin and Q.-F. Wang

### Comment

Pyrrol-2-ylmethyleneamine ligands have attracted much recent attention due to their excellent coordination abilities for metal ions (Yang *et al.*, 2006 & Wang *et al.*, 2010). As part of our ongoing search for a biologically active material, the title compound was synthesized and characterized by X-ray diffraction.

In the title compound, all the bond lengths are comparable with those observed in the other similar compound (Wang *et al.*, 2009). The molecule adopts an *E* configuration at the C=N double bond. The dihedral angle between pyrrole ring (N2/C8–C11, r.m.s. deviation 0.0035 Å) and phenyl ring (C1–C6, r.m.s. deviation 0.0036 Å) is 41.55 (8)°. In the crystal, the molecules are linked into a centrosymmetric dimer by two intermolecular N—H···O hydrogen bonds, forming a  $R_2^2(10)$  ring motif (Table 1, Fig. 2). In the dimer, the two pyrrole rings are almost coplanar (r.m.s. deviation 0.028 Å) and the two phenyl rings are parallel with each other. The crystal packing is further stabilized by the stacking between the C=N with the adjacent pyrrole ring, with centroid–centroid distances of 3.642 Å.

### Experimental

A quantity of aniline (0.186 g, 2 mmol) was dissolved in ethanol (10 ml), then an ethanol solution (10 ml) containing ethyl 5-formyl-3,4-dimethyl-1*H*-pyrrole-2-carboxylate (0.39 g, 2 mmol) was added dropwise at room temperature. After stirring for 4 h, the mixture was filtered and set aside to crystallize at room temperature for several days, giving yellow block crystals.

### Refinement

All H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and were thereafter treated as riding, with  $U_{\text{iso}}(\text{H})$  values of 1.5U<sub>eq</sub>(C) for methyl groups and 1.2U<sub>eq</sub>(C,N) for others.

### Figures

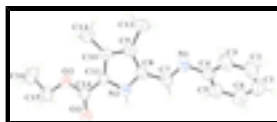


Fig. 1. The molecular structure shown with 50% probability displacement ellipsoids.

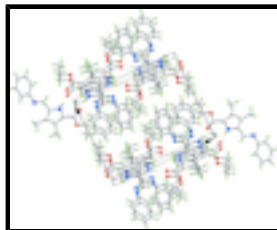


Fig. 2. Crystal packing of the title compound showing the dimers formed by hydrogen bonds (dashed lines).

## Ethyl 3,4-dimethyl-5-[(E)-(phenylimino)methyl]-1H-pyrrole-2-carboxylate

### Crystal data

$C_{16}H_{18}N_2O_2$	$F(000) = 576$
$M_r = 270.32$	$D_x = 1.197 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2ybc	Cell parameters from 5515 reflections
$a = 12.5463 (7) \text{ \AA}$	$\theta = 2.2\text{--}26.2^\circ$
$b = 14.6525 (9) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$c = 8.4490 (5) \text{ \AA}$	$T = 296 \text{ K}$
$\beta = 105.042 (3)^\circ$	Block, yellow
$V = 1500.00 (15) \text{ \AA}^3$	$0.35 \times 0.26 \times 0.18 \text{ mm}$
$Z = 4$	

### Data collection

Bruker SMART CCD diffractometer	3413 independent reflections
Radiation source: fine-focus sealed tube graphite	2078 reflections with $I > 2\sigma(I)$
phi and $\omega$ scans	$R_{\text{int}} = 0.030$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$\theta_{\text{max}} = 27.6^\circ$ , $\theta_{\text{min}} = 1.7^\circ$
$T_{\text{min}} = 0.975$ , $T_{\text{max}} = 0.986$	$h = -14 \rightarrow 16$
12405 measured reflections	$k = -14 \rightarrow 19$
	$l = -10 \rightarrow 8$

### Refinement

Refinement on $F^2$	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.044$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.144$	H-atom parameters constrained
$S = 1.01$	$w = 1/[\sigma^2(F_o^2) + (0.0645P)^2 + 0.3198P]$
3413 reflections	where $P = (F_o^2 + 2F_c^2)/3$
184 parameters	$(\Delta/\sigma)_{\text{max}} = 0.007$
0 restraints	$\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$

### Special details

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations

between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N2	0.02009 (11)	0.14614 (9)	0.03549 (16)	0.0474 (3)
H2A	-0.0130	0.0983	-0.0125	0.057*
O2	0.24807 (9)	0.06709 (8)	0.35169 (14)	0.0576 (3)
O3	0.12317 (10)	-0.01491 (9)	0.17093 (15)	0.0612 (4)
C14	0.15990 (13)	0.05888 (12)	0.22476 (19)	0.0462 (4)
C11	0.11351 (13)	0.14570 (11)	0.16129 (19)	0.0444 (4)
N1	-0.15004 (12)	0.33049 (10)	-0.16873 (18)	0.0545 (4)
C8	-0.01284 (13)	0.23366 (11)	-0.0027 (2)	0.0466 (4)
C10	0.14228 (13)	0.23604 (11)	0.20337 (19)	0.0458 (4)
C4	-0.25266 (15)	0.33933 (12)	-0.2860 (2)	0.0543 (5)
C7	-0.11212 (14)	0.25067 (12)	-0.1305 (2)	0.0514 (4)
H7	-0.1500	0.2012	-0.1874	0.062*
C9	0.06264 (13)	0.29161 (11)	0.1002 (2)	0.0465 (4)
C12	0.05849 (16)	0.39340 (12)	0.1001 (2)	0.0619 (5)
H12A	0.0308	0.4150	-0.0102	0.093*
H12B	0.1313	0.4172	0.1452	0.093*
H12C	0.0106	0.4135	0.1652	0.093*
C3	-0.26627 (17)	0.41010 (14)	-0.3976 (2)	0.0634 (5)
H3	-0.2072	0.4482	-0.3985	0.076*
C13	0.24063 (14)	0.26959 (13)	0.3311 (2)	0.0606 (5)
H13A	0.2367	0.3347	0.3399	0.091*
H13B	0.3067	0.2533	0.3006	0.091*
H13C	0.2416	0.2422	0.4347	0.091*
C2	-0.3674 (2)	0.42436 (17)	-0.5076 (3)	0.0782 (7)
H2	-0.3756	0.4714	-0.5838	0.094*
C15	0.29547 (16)	-0.01538 (14)	0.4354 (2)	0.0635 (5)
H15A	0.3295	-0.0515	0.3656	0.076*
H15B	0.2388	-0.0519	0.4644	0.076*
C16	0.37962 (17)	0.01386 (17)	0.5854 (3)	0.0793 (6)
H16A	0.4327	0.0528	0.5553	0.119*
H16B	0.4163	-0.0389	0.6416	0.119*
H16C	0.3442	0.0465	0.6562	0.119*
C5	-0.34233 (16)	0.28429 (15)	-0.2859 (3)	0.0727 (6)
H5	-0.3348	0.2364	-0.2116	0.087*
C1	-0.4555 (2)	0.37029 (19)	-0.5058 (3)	0.0869 (7)
H1	-0.5238	0.3809	-0.5789	0.104*
C6	-0.44248 (18)	0.30041 (18)	-0.3958 (3)	0.0893 (7)
H6	-0.5023	0.2631	-0.3950	0.107*

## supplementary materials

---

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N2	0.0494 (8)	0.0418 (8)	0.0461 (8)	0.0011 (6)	0.0038 (6)	-0.0006 (6)
O2	0.0557 (7)	0.0545 (8)	0.0523 (7)	0.0022 (6)	-0.0044 (6)	0.0051 (6)
O3	0.0666 (8)	0.0467 (8)	0.0594 (8)	0.0035 (6)	-0.0030 (6)	-0.0027 (6)
C14	0.0456 (9)	0.0511 (11)	0.0405 (9)	0.0002 (8)	0.0086 (7)	-0.0011 (7)
C11	0.0459 (9)	0.0457 (9)	0.0396 (9)	-0.0005 (7)	0.0075 (7)	0.0011 (7)
N1	0.0567 (9)	0.0494 (9)	0.0542 (9)	0.0074 (7)	0.0083 (7)	0.0051 (7)
C8	0.0506 (9)	0.0433 (9)	0.0460 (9)	0.0041 (7)	0.0128 (7)	0.0036 (7)
C10	0.0492 (9)	0.0479 (10)	0.0420 (9)	-0.0043 (7)	0.0149 (7)	-0.0020 (7)
C4	0.0579 (10)	0.0494 (10)	0.0528 (11)	0.0119 (8)	0.0092 (8)	0.0005 (8)
C7	0.0540 (10)	0.0481 (10)	0.0496 (10)	0.0036 (8)	0.0089 (8)	0.0022 (8)
C9	0.0512 (9)	0.0446 (9)	0.0465 (9)	-0.0008 (7)	0.0176 (8)	0.0013 (7)
C12	0.0680 (12)	0.0455 (10)	0.0714 (13)	-0.0028 (9)	0.0164 (10)	-0.0009 (9)
C3	0.0763 (13)	0.0590 (12)	0.0550 (11)	0.0166 (10)	0.0170 (10)	0.0081 (9)
C13	0.0593 (11)	0.0601 (12)	0.0583 (12)	-0.0110 (9)	0.0079 (9)	-0.0053 (9)
C2	0.0990 (17)	0.0783 (15)	0.0530 (12)	0.0344 (14)	0.0118 (12)	0.0095 (11)
C15	0.0636 (11)	0.0639 (12)	0.0568 (11)	0.0111 (9)	0.0046 (9)	0.0116 (9)
C16	0.0634 (12)	0.1025 (18)	0.0616 (13)	0.0050 (12)	-0.0025 (10)	0.0135 (12)
C5	0.0632 (12)	0.0629 (13)	0.0847 (15)	0.0045 (10)	0.0058 (11)	0.0142 (10)
C1	0.0742 (15)	0.0951 (19)	0.0755 (16)	0.0244 (14)	-0.0093 (12)	-0.0066 (13)
C6	0.0633 (13)	0.0848 (17)	0.106 (2)	0.0012 (12)	-0.0026 (13)	-0.0002 (15)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

N2—C8	1.360 (2)	C12—H12C	0.9600
N2—C11	1.363 (2)	C3—C2	1.381 (3)
N2—H2A	0.8600	C3—H3	0.9300
O2—C14	1.3313 (18)	C13—H13A	0.9600
O2—C15	1.448 (2)	C13—H13B	0.9600
O3—C14	1.216 (2)	C13—H13C	0.9600
C14—C11	1.443 (2)	C2—C1	1.363 (3)
C11—C10	1.394 (2)	C2—H2	0.9300
N1—C7	1.272 (2)	C15—C16	1.487 (3)
N1—C4	1.412 (2)	C15—H15A	0.9700
C8—C9	1.395 (2)	C15—H15B	0.9700
C8—C7	1.443 (2)	C16—H16A	0.9600
C10—C9	1.403 (2)	C16—H16B	0.9600
C10—C13	1.496 (2)	C16—H16C	0.9600
C4—C3	1.382 (2)	C5—C6	1.375 (3)
C4—C5	1.384 (3)	C5—H5	0.9300
C7—H7	0.9300	C1—C6	1.364 (3)
C9—C12	1.492 (2)	C1—H1	0.9300
C12—H12A	0.9600	C6—H6	0.9300
C12—H12B	0.9600		
C8—N2—C11	109.65 (13)	C4—C3—H3	119.9

C8—N2—H2A	125.2	C2—C3—H3	119.9
C11—N2—H2A	125.2	C10—C13—H13A	109.5
C14—O2—C15	117.92 (14)	C10—C13—H13B	109.5
O3—C14—O2	122.44 (15)	H13A—C13—H13B	109.5
O3—C14—C11	124.58 (15)	C10—C13—H13C	109.5
O2—C14—C11	112.98 (14)	H13A—C13—H13C	109.5
N2—C11—C10	107.94 (14)	H13B—C13—H13C	109.5
N2—C11—C14	118.44 (14)	C1—C2—C3	120.7 (2)
C10—C11—C14	133.60 (15)	C1—C2—H2	119.6
C7—N1—C4	118.36 (15)	C3—C2—H2	119.6
N2—C8—C9	108.10 (14)	O2—C15—C16	106.66 (17)
N2—C8—C7	119.37 (15)	O2—C15—H15A	110.4
C9—C8—C7	132.53 (16)	C16—C15—H15A	110.4
C11—C10—C9	107.29 (14)	O2—C15—H15B	110.4
C11—C10—C13	127.36 (15)	C16—C15—H15B	110.4
C9—C10—C13	125.34 (16)	H15A—C15—H15B	108.6
C3—C4—C5	118.75 (18)	C15—C16—H16A	109.5
C3—C4—N1	118.45 (17)	C15—C16—H16B	109.5
C5—C4—N1	122.62 (17)	H16A—C16—H16B	109.5
N1—C7—C8	122.82 (16)	C15—C16—H16C	109.5
N1—C7—H7	118.6	H16A—C16—H16C	109.5
C8—C7—H7	118.6	H16B—C16—H16C	109.5
C8—C9—C10	107.02 (14)	C6—C5—C4	120.0 (2)
C8—C9—C12	126.15 (15)	C6—C5—H5	120.0
C10—C9—C12	126.84 (15)	C4—C5—H5	120.0
C9—C12—H12A	109.5	C2—C1—C6	119.4 (2)
C9—C12—H12B	109.5	C2—C1—H1	120.3
H12A—C12—H12B	109.5	C6—C1—H1	120.3
C9—C12—H12C	109.5	C1—C6—C5	121.0 (2)
H12A—C12—H12C	109.5	C1—C6—H6	119.5
H12B—C12—H12C	109.5	C5—C6—H6	119.5
C4—C3—C2	120.1 (2)		
C15—O2—C14—O3	-5.1 (2)	C9—C8—C7—N1	-1.8 (3)
C15—O2—C14—C11	174.30 (15)	N2—C8—C9—C10	-0.44 (18)
C8—N2—C11—C10	-0.93 (18)	C7—C8—C9—C10	178.79 (17)
C8—N2—C11—C14	177.75 (15)	N2—C8—C9—C12	179.75 (15)
O3—C14—C11—N2	2.1 (3)	C7—C8—C9—C12	-1.0 (3)
O2—C14—C11—N2	-177.28 (13)	C11—C10—C9—C8	-0.12 (18)
O3—C14—C11—C10	-179.64 (17)	C13—C10—C9—C8	178.52 (15)
O2—C14—C11—C10	1.0 (3)	C11—C10—C9—C12	179.69 (16)
C11—N2—C8—C9	0.85 (18)	C13—C10—C9—C12	-1.7 (3)
C11—N2—C8—C7	-178.50 (14)	C5—C4—C3—C2	-0.8 (3)
N2—C11—C10—C9	0.63 (18)	N1—C4—C3—C2	-176.03 (17)
C14—C11—C10—C9	-177.76 (18)	C4—C3—C2—C1	1.3 (3)
N2—C11—C10—C13	-177.97 (15)	C14—O2—C15—C16	-171.10 (15)
C14—C11—C10—C13	3.6 (3)	C3—C4—C5—C6	0.2 (3)
C7—N1—C4—C3	-141.86 (17)	N1—C4—C5—C6	175.21 (19)
C7—N1—C4—C5	43.1 (3)	C3—C2—C1—C6	-1.2 (3)
C4—N1—C7—C8	-174.75 (16)	C2—C1—C6—C5	0.6 (4)

## supplementary materials

---

N2—C8—C7—N1

177.38 (16)

C4—C5—C6—C1

-0.1 (4)

### *Hydrogen-bond geometry (Å, °)*

*D*—H $\cdots$ *A*

*D*—H

H $\cdots$ *A*

*D* $\cdots$ *A*

*D*—H $\cdots$ *A*

N2—H2A $\cdots$ O3<sup>i</sup>

0.86

2.06

2.8883 (18)

162.

Symmetry codes: (i)  $-x, -y, -z$ .



Fig. 1

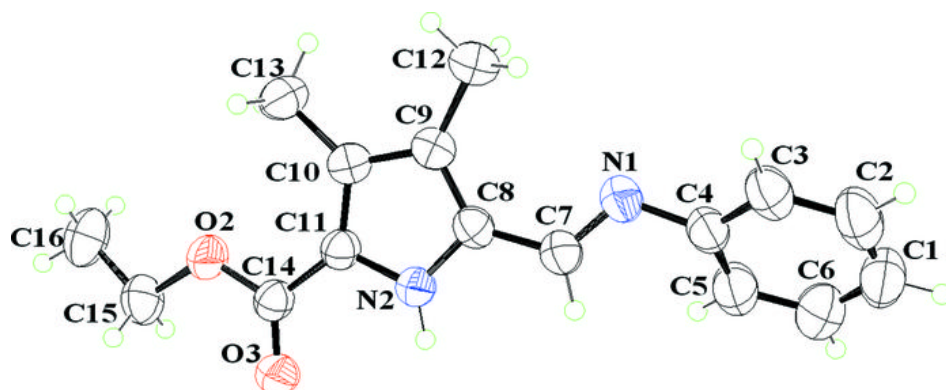


Fig. 2

