

# Ethyl 5-cyano-4-[2-(2,4-dichlorophenoxy)acetamido]-1-phenyl-1H-pyrrole-3-carboxylate

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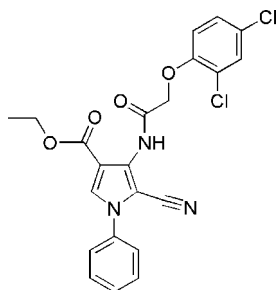
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Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.066;  $wR$  factor = 0.154; data-to-parameter ratio = 15.0.

In the title compound,  $\text{C}_{22}\text{H}_{17}\text{Cl}_2\text{N}_3\text{O}_4$ , the pyrrole ring and the 2,4-dichlorophenyl group form a dihedral angle of  $8.14(13)^\circ$ ; the phenyl ring is twisted with respect to the pyrrole ring, forming a dihedral angle of  $60.77(14)^\circ$ . The  $\text{C}=\text{O}$  bond length is  $1.213(3)$  Å, indicating that the molecule is in the keto form, associated with a  $-\text{CONH}-$  group, and the amide group adopts the usual *trans* conformation. The molecule is stabilized by an intramolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen-bonding interaction. In the crystal, the stacked molecules exhibit intermolecular  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{N}$  hydrogen-bonding interactions.

## Related literature

For the preparation and biological activity of acid amides, see: Xue *et al.* (2007); Li *et al.* (1995). For related structures, see: He *et al.* (2007a,b).



## Experimental

### Crystal data

$\text{C}_{22}\text{H}_{17}\text{Cl}_2\text{N}_3\text{O}_4$   
 $M_r = 458.29$   
Monoclinic,  $C2/c$   
 $a = 32.8846(18)$  Å  
 $b = 7.6224(4)$  Å  
 $c = 17.2834(9)$  Å  
 $\gamma = 89.773(1)^\circ$   
 $V = 4332.2(4)$  Å<sup>3</sup>  
 $Z = 8$   
Mo  $K\alpha$  radiation  
 $\mu = 0.33$  mm<sup>-1</sup>  
 $T = 298$  K  
 $0.10 \times 0.10 \times 0.10$  mm

### Data collection

Bruker SMART 4K CCD area-detector diffractometer  
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)  
 $T_{\min} = 0.968$ ,  $T_{\max} = 0.968$   
16521 measured reflections  
4279 independent reflections  
3034 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.081$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.066$   
 $wR(F^2) = 0.154$   
 $S = 0.99$   
4279 reflections  
285 parameters  
H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\max} = 0.32$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.31$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{O3}$	0.82 (2)	2.15 (3)	2.813 (3)	137 (2)
$\text{C7}-\text{H7A}\cdots\text{O2}^i$	0.97	2.57	3.341 (3)	137
$\text{C3}-\text{H3}\cdots\text{N3}^i$	0.93	2.61	3.312 (3)	133

Symmetry code: (i)  $-x + 2, y, -z + \frac{1}{2}$ .

Data collection: SMART (Bruker, 2001); cell refinement: SAINT-Plus (Bruker, 2001); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: SHELXTL (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2840).

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**supplementary materials**

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## Ethyl 5-cyano-4-[2-(2,4-dichlorophenoxy)acetamido]-1-phenyl-1*H*-pyrrole-3-carboxylate

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### Comment

The chemical and pharmacological properties of acid amides have been investigated extensively, owing to their potentially beneficial chemical and biological activities (Xue *et al.*, 2007 and Li *et al.*, 1995). As part of our studies on the synthesis and characterization of related compounds (He *et al.*, 2007a,b), we report here the synthesis and crystal structure of ethyl 4-(2-(2,4-dichlorophenoxy)acetamido)-5-cyano-1-phenyl-1*H*-pyrrole-3-carboxylate, (I).

Within the molecule of (I), the bond lengths and angles present no unusual features. The pyrrole ring and the 2,4-dichlorophenyl group form a dihedral angle of 8.14 (13)°, the C17—C22 phenyl ring is twisted with respect to pyrrole ring, with a dihedral angle of 60.77 (14)°. The C=O bond length is 1.213 (3) Å, indicating that the molecule is in the keto form (Fig. 1), associated with —CONH— moiety, and the amide group adopts the usual *trans* conformation. The crystal structure is stabilized by intramolecular N—H···O hydrogen bonds interactions. In addition, the stacked molecules exhibit intermolecular C—H···O and C—H···N hydrogen bonds interactions (Fig 2. Table 1).

### Experimental

To a solution of the ethyl 4-amino-5-cyano-1-phenyl-1*H*-pyrrole-3-carboxylate (3 mmol) in dry dichloromethane (15 ml) was added 2-(2,4-dichlorophenoxy)acetyl chloride at 273–278 K. After stirring the reaction mixture for 4 h from 273–278 K to room temperature. The solution was concentrated under reduced pressure and the residue was recrystallized from ethanol to give the title compound in a yield of 45%. Crystals suitable for single-crystal X-ray diffraction were obtained by recrystallization from a mixed solvent of ethanol and dichloromethane (1:3 v/v) at room temperature.

### Refinement

The carbon-bound hydrogen atoms were positioned geometrically and refined using a riding model with C—H = 0.93 Å,  $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$  for  $\text{Csp}^2$ , C—H = 0.97 Å,  $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$  for  $\text{CH}_2$  and C—H = 0.96 Å,  $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{C})$  for  $\text{CH}_3$ . The H atom of the NH group was found from a difference Fourier map and refined with a fixed  $U_{\text{iso}}$  of 0.05.

### Figures

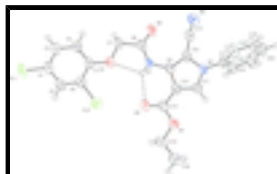


Fig. 1. A view of the molecule of (I) showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. Intramolecular hydrogen bonds are drawn as a dashed line.

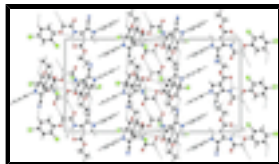


Fig. 2. Part of the crystal structure of (I), showing hydrogen bonds stacking interactions.

## Ethyl 5-cyano-4-[2-(2,4-dichlorophenoxy)acetamido]-1-phenyl-1H-pyrrole-3-carboxylate

### Crystal data

$C_{22}H_{17}Cl_2N_3O_4$	$F_{000} = 1888$
$M_r = 458.29$	$D_x = 1.401 \text{ Mg m}^{-3}$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: $-C 2yc$	Cell parameters from 4400 reflections
$a = 32.8846 (18) \text{ \AA}$	$\theta = 2.5\text{--}26.3^\circ$
$b = 7.6224 (4) \text{ \AA}$	$\mu = 0.33 \text{ mm}^{-1}$
$c = 17.2834 (9) \text{ \AA}$	$T = 298 \text{ K}$
$\beta = 90^\circ$	Block, yellow
$V = 4332.2 (4) \text{ \AA}^3$	$0.10 \times 0.10 \times 0.10 \text{ mm}$
$Z = 8$	

### Data collection

Bruker SMART 4K CCD area-detector diffractometer	4279 independent reflections
Radiation source: fine-focus sealed tube	3034 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.081$
$T = 298 \text{ K}$	$\theta_{\text{max}} = 26.0^\circ$
$\varphi$ and $\omega$ scans	$\theta_{\text{min}} = 2.4^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$h = -40 \rightarrow 39$
$T_{\text{min}} = 0.968$ , $T_{\text{max}} = 0.968$	$k = -9 \rightarrow 9$
16521 measured reflections	$l = -21 \rightarrow 21$

### Refinement

Refinement on $F^2$	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.066$	$w = 1/[\sigma^2(F_o^2) + (0.055P)^2]$
$wR(F^2) = 0.154$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{\text{max}} = 0.001$
4279 reflections	$\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$
285 parameters	$\Delta\rho_{\text{min}} = -0.31 \text{ e \AA}^{-3}$
	Extinction correction: SHELXL97 (Sheldrick, 2008),
	$F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Primary atom site location: structure-invariant direct methods Extinction coefficient: 0.0042 (4)

Secondary atom site location: difference Fourier map

*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>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	1.06329 (6)	0.1879 (3)	0.05715 (13)	0.0426 (6)
C2	1.09068 (7)	0.1312 (3)	0.11185 (14)	0.0466 (6)
H2	1.0813	0.0801	0.1573	0.056*
C3	1.13204 (7)	0.1495 (3)	0.09995 (14)	0.0472 (6)
H3	1.1505	0.1107	0.1369	0.057*
C4	1.14550 (7)	0.2257 (3)	0.03284 (15)	0.0463 (6)
C5	1.11863 (7)	0.2840 (3)	-0.02255 (14)	0.0470 (6)
H5	1.1282	0.3351	-0.0679	0.056*
C6	1.07783 (7)	0.2659 (3)	-0.01021 (14)	0.0469 (6)
C7	1.00695 (6)	0.1112 (4)	0.13490 (13)	0.0558 (7)
H7A	1.0184	0.1792	0.1770	0.067*
H7B	1.0147	-0.0103	0.1420	0.067*
C8	0.96114 (7)	0.1270 (4)	0.13558 (14)	0.0511 (7)
C9	0.90125 (6)	0.1809 (3)	0.05703 (13)	0.0368 (5)
C10	0.88509 (6)	0.2629 (3)	-0.00962 (13)	0.0399 (6)
C11	0.84392 (7)	0.2760 (3)	0.00079 (14)	0.0440 (6)
H11	0.8259	0.3266	-0.0340	0.053*
C12	0.86902 (6)	0.1425 (3)	0.10524 (12)	0.0387 (5)
C13	0.86531 (6)	0.0454 (3)	0.17456 (14)	0.0446 (6)
C14	0.90849 (7)	0.3115 (3)	-0.07786 (13)	0.0431 (6)
C15	0.90645 (8)	0.4263 (4)	-0.20453 (14)	0.0589 (7)
H15A	0.9189	0.3226	-0.2267	0.071*
H15B	0.9277	0.5115	-0.1943	0.071*
C16	0.87619 (9)	0.5007 (4)	-0.25914 (15)	0.0693 (8)
H16A	0.8568	0.4119	-0.2729	0.104*
H16B	0.8898	0.5412	-0.3048	0.104*
H16C	0.8624	0.5970	-0.2350	0.104*
C17	0.79429 (7)	0.2161 (4)	0.10469 (13)	0.0448 (6)
C18	0.77391 (8)	0.0684 (4)	0.12715 (16)	0.0647 (8)
H18	0.7852	-0.0419	0.1193	0.078*

## supplementary materials

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C19	0.73596 (8)	0.0858 (5)	0.16203 (18)	0.0770 (10)
H19	0.7217	-0.0133	0.1775	0.092*
C20	0.71982 (8)	0.2485 (5)	0.17348 (18)	0.0729 (10)
H20	0.6945	0.2596	0.1968	0.088*
C21	0.74032 (9)	0.3942 (5)	0.15128 (18)	0.0774 (10)
H21	0.7291	0.5045	0.1596	0.093*
C22	0.77795 (8)	0.3792 (4)	0.11624 (16)	0.0650 (8)
H22	0.7920	0.4789	0.1007	0.078*
Cl1	1.197500 (19)	0.25261 (10)	0.01853 (5)	0.0708 (3)
Cl2	1.04355 (2)	0.33930 (11)	-0.07912 (4)	0.0718 (3)
N1	0.94269 (5)	0.1515 (3)	0.06772 (11)	0.0413 (5)
H1	0.9550 (7)	0.163 (3)	0.0265 (14)	0.050*
N2	0.83373 (5)	0.2042 (3)	0.06907 (11)	0.0428 (5)
N3	0.85896 (7)	-0.0374 (3)	0.22814 (13)	0.0672 (7)
O1	1.02216 (4)	0.1736 (3)	0.06353 (9)	0.0548 (5)
O2	0.94335 (5)	0.1152 (3)	0.19676 (10)	0.0876 (8)
O3	0.94465 (5)	0.2865 (3)	-0.08352 (10)	0.0602 (5)
O4	0.88571 (5)	0.3817 (2)	-0.13360 (9)	0.0512 (5)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0333 (11)	0.0469 (14)	0.0477 (14)	-0.0048 (11)	0.0088 (9)	-0.0098 (11)
C2	0.0392 (12)	0.0525 (16)	0.0481 (14)	0.0005 (11)	0.0085 (10)	-0.0034 (11)
C3	0.0365 (12)	0.0482 (15)	0.0568 (15)	-0.0009 (11)	0.0050 (10)	-0.0051 (12)
C4	0.0351 (12)	0.0420 (15)	0.0619 (17)	-0.0056 (10)	0.0123 (11)	-0.0102 (12)
C5	0.0456 (14)	0.0446 (15)	0.0509 (15)	-0.0098 (12)	0.0122 (11)	-0.0045 (12)
C6	0.0453 (13)	0.0466 (15)	0.0488 (15)	-0.0050 (11)	0.0058 (11)	-0.0047 (11)
C7	0.0332 (12)	0.088 (2)	0.0467 (15)	-0.0018 (13)	0.0080 (10)	0.0048 (14)
C8	0.0367 (12)	0.0704 (19)	0.0462 (15)	-0.0007 (12)	0.0077 (10)	0.0003 (13)
C9	0.0346 (11)	0.0349 (12)	0.0408 (13)	0.0005 (9)	0.0069 (9)	-0.0042 (10)
C10	0.0379 (12)	0.0401 (14)	0.0417 (13)	-0.0004 (10)	0.0061 (9)	-0.0062 (10)
C11	0.0402 (12)	0.0495 (15)	0.0422 (14)	0.0065 (11)	0.0030 (10)	0.0003 (11)
C12	0.0335 (11)	0.0396 (14)	0.0429 (13)	0.0006 (10)	0.0037 (9)	-0.0053 (10)
C13	0.0387 (12)	0.0448 (15)	0.0503 (15)	-0.0017 (11)	0.0095 (10)	-0.0015 (12)
C14	0.0451 (13)	0.0406 (14)	0.0438 (14)	-0.0024 (11)	0.0071 (10)	-0.0033 (11)
C15	0.0665 (16)	0.0642 (19)	0.0462 (15)	-0.0044 (14)	0.0142 (12)	0.0064 (13)
C16	0.095 (2)	0.0593 (19)	0.0532 (17)	0.0005 (16)	0.0097 (15)	0.0086 (14)
C17	0.0321 (11)	0.0607 (17)	0.0417 (14)	0.0026 (11)	0.0075 (9)	-0.0056 (12)
C18	0.0486 (15)	0.062 (2)	0.083 (2)	-0.0056 (14)	0.0190 (13)	-0.0104 (16)
C19	0.0512 (16)	0.090 (3)	0.089 (2)	-0.0172 (17)	0.0248 (15)	-0.0068 (19)
C20	0.0404 (15)	0.113 (3)	0.065 (2)	0.0102 (17)	0.0119 (13)	-0.0050 (18)
C21	0.0597 (18)	0.076 (2)	0.097 (2)	0.0263 (17)	0.0216 (16)	0.0000 (19)
C22	0.0525 (15)	0.062 (2)	0.081 (2)	0.0180 (14)	0.0167 (14)	0.0102 (15)
Cl1	0.0377 (4)	0.0833 (7)	0.0914 (6)	-0.0088 (3)	0.0170 (3)	0.0030 (4)
Cl2	0.0563 (4)	0.0931 (7)	0.0661 (5)	-0.0070 (4)	-0.0030 (3)	0.0237 (4)
N1	0.0296 (9)	0.0548 (13)	0.0395 (11)	0.0012 (9)	0.0095 (8)	-0.0021 (9)
N2	0.0327 (10)	0.0500 (12)	0.0457 (12)	0.0018 (9)	0.0088 (8)	-0.0022 (9)

N3	0.0656 (14)	0.0708 (17)	0.0651 (15)	-0.0111 (13)	0.0067 (11)	0.0144 (14)
O1	0.0330 (8)	0.0832 (14)	0.0482 (10)	-0.0031 (8)	0.0087 (7)	0.0094 (9)
O2	0.0396 (10)	0.177 (3)	0.0465 (11)	0.0017 (12)	0.0101 (8)	0.0107 (13)
O3	0.0381 (9)	0.0839 (15)	0.0584 (12)	0.0009 (9)	0.0113 (8)	0.0078 (10)
O4	0.0494 (9)	0.0605 (12)	0.0436 (9)	0.0053 (8)	0.0091 (7)	0.0083 (8)

*Geometric parameters (Å, °)*

C1—O1	1.363 (2)	C12—N2	1.401 (3)
C1—C2	1.377 (3)	C12—C13	1.415 (3)
C1—C6	1.393 (3)	C13—N3	1.141 (3)
C2—C3	1.384 (3)	C14—O3	1.210 (3)
C2—H2	0.9300	C14—O4	1.334 (3)
C3—C4	1.372 (3)	C15—O4	1.445 (3)
C3—H3	0.9300	C15—C16	1.486 (4)
C4—C5	1.378 (4)	C15—H15A	0.9700
C4—C11	1.742 (2)	C15—H15B	0.9700
C5—C6	1.367 (3)	C16—H16A	0.9600
C5—H5	0.9300	C16—H16B	0.9600
C6—C12	1.734 (3)	C16—H16C	0.9600
C7—O1	1.415 (2)	C17—C18	1.368 (4)
C7—C8	1.513 (3)	C17—C22	1.370 (3)
C7—H7A	0.9700	C17—N2	1.440 (3)
C7—H7B	0.9700	C18—C19	1.394 (3)
C8—O2	1.213 (3)	C18—H18	0.9300
C8—N1	1.335 (3)	C19—C20	1.365 (4)
C9—C12	1.381 (3)	C19—H19	0.9300
C9—N1	1.395 (3)	C20—C21	1.356 (4)
C9—C10	1.416 (3)	C20—H20	0.9300
C10—C11	1.371 (3)	C21—C22	1.384 (3)
C10—C14	1.458 (3)	C21—H21	0.9300
C11—N2	1.345 (3)	C22—H22	0.9300
C11—H11	0.9300	N1—H1	0.82 (2)
O1—C1—C2	124.7 (2)	O3—C14—C10	123.1 (2)
O1—C1—C6	116.3 (2)	O4—C14—C10	112.99 (19)
C2—C1—C6	119.0 (2)	O4—C15—C16	108.3 (2)
C1—C2—C3	120.7 (2)	O4—C15—H15A	110.0
C1—C2—H2	119.7	C16—C15—H15A	110.0
C3—C2—H2	119.7	O4—C15—H15B	110.0
C4—C3—C2	119.1 (2)	C16—C15—H15B	110.0
C4—C3—H3	120.5	H15A—C15—H15B	108.4
C2—C3—H3	120.5	C15—C16—H16A	109.5
C3—C4—C5	121.2 (2)	C15—C16—H16B	109.5
C3—C4—C11	119.2 (2)	H16A—C16—H16B	109.5
C5—C4—C11	119.60 (19)	C15—C16—H16C	109.5
C6—C5—C4	119.3 (2)	H16A—C16—H16C	109.5
C6—C5—H5	120.3	H16B—C16—H16C	109.5
C4—C5—H5	120.3	C18—C17—C22	120.9 (2)
C5—C6—C1	120.7 (2)	C18—C17—N2	120.8 (2)

## supplementary materials

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C5—C6—C12	119.96 (19)	C22—C17—N2	118.3 (2)
C1—C6—C12	119.30 (18)	C17—C18—C19	118.9 (3)
O1—C7—C8	109.44 (19)	C17—C18—H18	120.5
O1—C7—H7A	109.8	C19—C18—H18	120.5
C8—C7—H7A	109.8	C20—C19—C18	119.9 (3)
O1—C7—H7B	109.8	C20—C19—H19	120.0
C8—C7—H7B	109.8	C18—C19—H19	120.0
H7A—C7—H7B	108.2	C21—C20—C19	120.7 (3)
O2—C8—N1	123.9 (2)	C21—C20—H20	119.6
O2—C8—C7	118.8 (2)	C19—C20—H20	119.6
N1—C8—C7	117.25 (19)	C20—C21—C22	120.1 (3)
C12—C9—N1	129.6 (2)	C20—C21—H21	119.9
C12—C9—C10	107.26 (19)	C22—C21—H21	119.9
N1—C9—C10	123.14 (18)	C17—C22—C21	119.4 (3)
C11—C10—C9	107.27 (19)	C17—C22—H22	120.3
C11—C10—C14	127.6 (2)	C21—C22—H22	120.3
C9—C10—C14	124.99 (19)	C8—N1—C9	125.75 (19)
N2—C11—C10	109.4 (2)	C8—N1—H1	123.4 (17)
N2—C11—H11	125.3	C9—N1—H1	110.5 (18)
C10—C11—H11	125.3	C11—N2—C12	108.81 (17)
C9—C12—N2	107.22 (19)	C11—N2—C17	125.1 (2)
C9—C12—C13	133.6 (2)	C12—N2—C17	125.30 (19)
N2—C12—C13	118.87 (18)	C1—O1—C7	116.70 (18)
N3—C13—C12	173.9 (2)	C14—O4—C15	116.29 (18)
O3—C14—O4	123.9 (2)		
O1—C1—C2—C3	179.2 (2)	C22—C17—C18—C19	0.2 (4)
C6—C1—C2—C3	-0.7 (4)	N2—C17—C18—C19	179.4 (2)
C1—C2—C3—C4	0.2 (4)	C17—C18—C19—C20	-0.2 (5)
C2—C3—C4—C5	0.1 (4)	C18—C19—C20—C21	0.0 (5)
C2—C3—C4—C11	178.79 (19)	C19—C20—C21—C22	0.3 (5)
C3—C4—C5—C6	0.2 (4)	C18—C17—C22—C21	0.1 (4)
C11—C4—C5—C6	-178.57 (18)	N2—C17—C22—C21	-179.2 (3)
C4—C5—C6—C1	-0.6 (4)	C20—C21—C22—C17	-0.4 (5)
C4—C5—C6—C12	179.78 (19)	O2—C8—N1—C9	5.3 (4)
O1—C1—C6—C5	-179.0 (2)	C7—C8—N1—C9	-175.4 (2)
C2—C1—C6—C5	0.9 (4)	C12—C9—N1—C8	-21.0 (4)
O1—C1—C6—C12	0.6 (3)	C10—C9—N1—C8	158.0 (2)
C2—C1—C6—C12	-179.51 (19)	C10—C11—N2—C12	0.3 (3)
O1—C7—C8—O2	-164.7 (3)	C10—C11—N2—C17	170.5 (2)
O1—C7—C8—N1	15.9 (3)	C9—C12—N2—C11	0.7 (3)
C12—C9—C10—C11	1.5 (3)	C13—C12—N2—C11	-173.6 (2)
N1—C9—C10—C11	-177.7 (2)	C9—C12—N2—C17	-169.5 (2)
C12—C9—C10—C14	-174.7 (2)	C13—C12—N2—C17	16.2 (3)
N1—C9—C10—C14	6.1 (4)	C18—C17—N2—C11	126.0 (3)
C9—C10—C11—N2	-1.1 (3)	C22—C17—N2—C11	-54.7 (3)
C14—C10—C11—N2	174.9 (2)	C18—C17—N2—C12	-65.4 (3)
N1—C9—C12—N2	177.8 (2)	C22—C17—N2—C12	113.9 (3)
C10—C9—C12—N2	-1.3 (2)	C2—C1—O1—C7	6.4 (3)
N1—C9—C12—C13	-9.1 (4)	C6—C1—O1—C7	-173.7 (2)



C10—C9—C12—C13	171.7 (3)	C8—C7—O1—C1	172.4 (2)
C11—C10—C14—O3	-175.9 (2)	O3—C14—O4—C15	0.9 (4)
C9—C10—C14—O3	-0.5 (4)	C10—C14—O4—C15	-177.4 (2)
C11—C10—C14—O4	2.5 (4)	C16—C15—O4—C14	179.6 (2)
C9—C10—C14—O4	177.9 (2)		

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O3	0.82 (2)	2.15 (3)	2.813 (3)	137 (2)
C7—H7A...O2 <sup>i</sup>	0.97	2.57	3.341 (3)	137
C3—H3...N3 <sup>i</sup>	0.93	2.61	3.312 (3)	133

Symmetry codes: (i)  $-x+2, y, -z+1/2$ .

Fig. 1

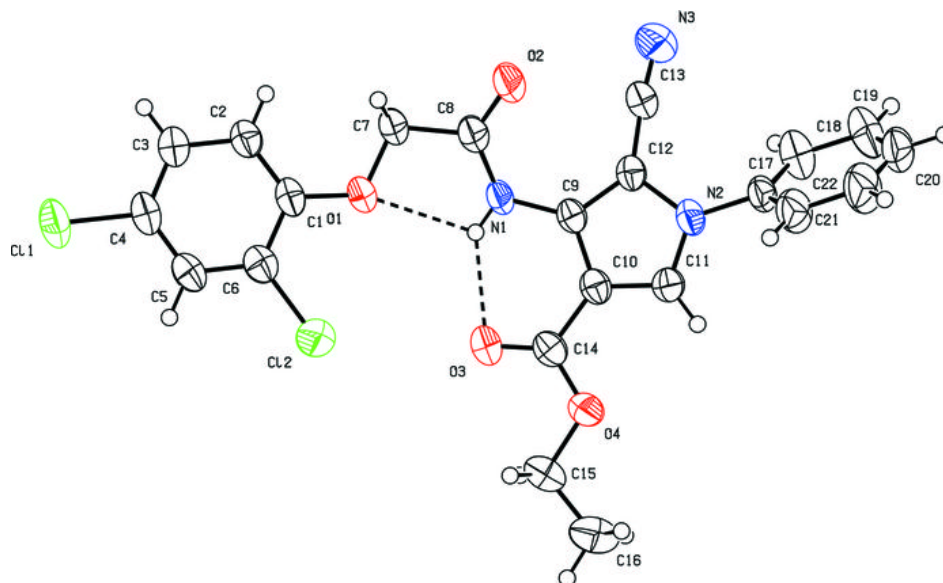


Fig. 2

