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1-(6-Chloropyridin-3-ylmethyl)-3-phenyl-1*H*-pyrazole-5-carboxylic acid

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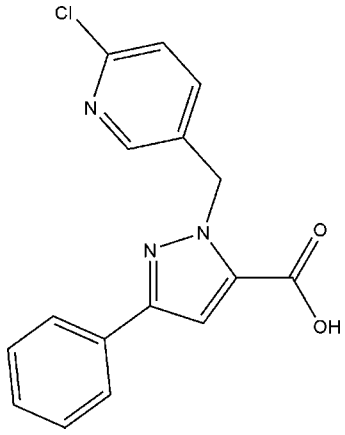
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.044; wR factor = 0.095; data-to-parameter ratio = 16.4.

In the title compound, $C_{16}H_{12}ClN_3O_2$, the pyrazole ring makes dihedral angles of 4.6 (1) and 67.1 (1)° with the phenyl and pyridine rings, respectively. The structure displays O—H···N hydrogen bonding.

Related literature

For related literature, see: Cottineau *et al.* (2002); Finn *et al.* (2003); Jia *et al.* (2004); Wei *et al.* (2006); Xia *et al.* (2007).



Experimental

Crystal data

$C_{16}H_{12}ClN_3O_2$
M_r = 313.74
Orthorhombic, *Fdd*2
a = 20.5430 (5) Å
b = 54.5902 (15) Å
c = 5.11340 (10) Å
V = 5734.4 (2) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 293 (2) K
0.26 × 0.14 × 0.04 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*APEX2*; Bruker, 2005)
T_{min} = 0.931, T_{max} = 0.989
11476 measured reflections
3276 independent reflections
2193 reflections with $I > 2\sigma(I)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.095
S = 1.01
3276 reflections
200 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983), 1442 Friedel pairs
Flack parameter: 0.05 (7)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N3^{i}	0.82	1.98	2.794 (3)	176

Symmetry code: (i) $x+\frac{1}{4}, -y+\frac{1}{4}, z+\frac{5}{4}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2243).

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supplementary materials

Acta Cryst. (2007). E**63**, o3348 [**doi:10.1107/S1600536807030954**]

1-(6-Chloropyridin-3-ylmethyl)-3-phenyl-1*H*-pyrazole-5-carboxylic acid

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Comment

Pyrazole framework plays an essential role in biologically active compounds. Many pyrazole derivatives are known to exhibit a wide range of biological properties such as anticoagulant (Jia *et al.*, 2004), antipyretic, antibacterial, hypoglycaemic, antihyperglycaemic, analgesic, anti-inflammatory, sedative-hypnotic (Cottineau *et al.*, 2002; Finn *et al.*, 2003), and antitumour (Wei *et al.*, 2006) activities. We report here the crystal structure of the title compound, (I).

Experimental

A mixture of ethyl 1-((6-chloropyridin-3-yl)methyl)-3-phenyl-1*H*-pyrazole-5-carboxylate (0.01 mol) that was synthesized according to the literature procedure (Xia *et al.*, 2007) and potassium hydroxide (0.02 mol) in ethanol (40 ml) was heated to reflux for 1 h. The solvent was removed under reduced pressure and the residue was dissolved in water and acidified with hydrochloric acid (10%). The precipitate was filtered and dried to give a white solid (yield 86%). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a solution of the solid in acetone at room temperature for 6 d.

Refinement

All H atoms were placed at geometrically calculated positions and allowed to ride with C—H = 0.97 Å (for CH_2 groups), and O—H = 0.82 Å; their isotropic displacement parameters were set to 1.2 times (CH_2 groups) or 1.5 times (O—H groups) the equivalent displacement parameter of their parent atoms.

Figures

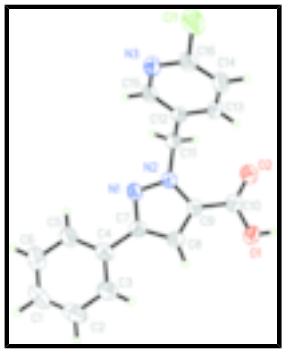

Fig. 1. The molecular strure of (I), showing displacement ellipsoids drawn at 50% probability level.

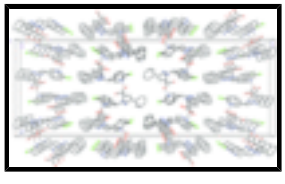

Fig. 2. Packing view of (I), shown down the *c* axis. Hydrogen bonds leading to columns units are drawn as dashed lines.

1-(6-Chloropyridin-3-ylmethyl)-3-phenyl-1*H*-pyrazole-5-carboxylic acid

Crystal data

$C_{16}H_{12}ClN_3O_2$	F_{000} = 2592
M_r = 313.74	D_x = 1.454 Mg m^{-3}
Orthorhombic, *Fdd*2	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: F 2 -2d	Cell parameters from 1585 reflections
a = 20.5430 (5) Å	θ = 2.5–21.4º
b = 54.5902 (15) Å	μ = 0.28 mm^{-1}
c = 5.11340 (10) Å	*T* = 293 (2) K
V = 5734.4 (2) Å^3	Plate, colourless
Z = 16	0.26 × 0.14 × 0.04 mm

Data collection

Bruker APEX2 CCD area-detector diffractometer	3276 independent reflections
Radiation source: fine-focus sealed tube	2193 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.058
T = 293(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 2.5º
Absorption correction: multi-scan (APEX2; Bruker, 2005)	*h* = −25→26
T_{min} = 0.931, T_{max} = 0.989	*k* = −67→70
11476 measured reflections	*l* = −6→6

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.044	$w = 1/[\sigma^2(F_o^2) + (0.0367P)^2 + 0.3657P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.095	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.02	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
3276 reflections	$\Delta\rho_{min}$ = −0.16 e Å^{-3}
200 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), with how many Friedel pairs?
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.05 (7)
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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.59781 (18)	0.25213 (5)	−0.3934 (7)	0.0630 (9)
H1	0.5909	0.2643	−0.5178	0.076*
C2	0.65305 (18)	0.25237 (6)	−0.2456 (7)	0.0635 (9)
H2	0.6840	0.2646	−0.2708	0.076*
C3	0.66326 (15)	0.23443 (5)	−0.0576 (6)	0.0544 (8)
H3	0.7009	0.2348	0.0437	0.065*
C4	0.61805 (12)	0.21602 (5)	−0.0195 (5)	0.0383 (6)
C5	0.56229 (13)	0.21617 (5)	−0.1724 (6)	0.0500 (7)
H5	0.5310	0.2040	−0.1489	0.060*
C6	0.55259 (15)	0.23404 (6)	−0.3588 (6)	0.0618 (9)
H6	0.5152	0.2338	−0.4612	0.074*
C7	0.62922 (12)	0.19686 (4)	0.1782 (5)	0.0348 (6)
C8	0.67929 (11)	0.19464 (4)	0.3623 (5)	0.0367 (6)
H8	0.7142	0.2052	0.3862	0.044*
C9	0.66637 (11)	0.17366 (4)	0.5004 (5)	0.0332 (6)
C10	0.70235 (12)	0.16293 (5)	0.7217 (5)	0.0359 (6)
C11	0.57428 (11)	0.14209 (4)	0.4677 (6)	0.0380 (6)
H11A	0.5281	0.1450	0.4422	0.046*
H11B	0.5811	0.1386	0.6517	0.046*
C12	0.59414 (11)	0.11997 (4)	0.3095 (5)	0.0350 (6)
C13	0.64818 (12)	0.10619 (4)	0.3792 (6)	0.0416 (7)
H13	0.6730	0.1106	0.5238	0.050*
C14	0.66494 (13)	0.08599 (5)	0.2343 (6)	0.0467 (7)
H14	0.7012	0.0766	0.2769	0.056*
C15	0.55921 (11)	0.11244 (4)	0.0930 (5)	0.0377 (6)
H15	0.5232	0.1216	0.0431	0.045*
C16	0.62635 (13)	0.08010 (5)	0.0246 (5)	0.0421 (7)
Cl1	0.64398 (4)	0.054295 (13)	−0.16079 (18)	0.0733 (3)
N1	0.58700 (9)	0.17805 (4)	0.2025 (4)	0.0373 (5)
N2	0.60997 (9)	0.16434 (3)	0.3997 (4)	0.0357 (5)
N3	0.57445 (10)	0.09264 (4)	−0.0486 (4)	0.0415 (6)
O1	0.75219 (8)	0.17705 (3)	0.7891 (4)	0.0445 (5)
H1A	0.7717	0.1708	0.9117	0.067*

O2	0.69003 (8)	0.14364 (3)	0.8283 (4)	0.0480 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.091 (2)	0.0468 (17)	0.051 (2)	0.0154 (17)	0.009 (2)	0.0181 (18)
C2	0.088 (2)	0.0406 (16)	0.062 (2)	−0.0096 (16)	0.008 (2)	0.0083 (16)
C3	0.0670 (19)	0.0435 (16)	0.053 (2)	−0.0087 (15)	−0.0061 (15)	0.0055 (15)
C4	0.0491 (16)	0.0328 (13)	0.0330 (15)	0.0067 (12)	0.0070 (13)	−0.0013 (12)
C5	0.0477 (16)	0.0501 (16)	0.0523 (19)	0.0053 (13)	−0.0031 (15)	0.0144 (16)
C6	0.0642 (19)	0.067 (2)	0.054 (2)	0.0120 (17)	−0.0065 (16)	0.0159 (19)
C7	0.0401 (14)	0.0307 (13)	0.0337 (16)	0.0017 (11)	0.0022 (12)	−0.0031 (11)
C8	0.0392 (14)	0.0322 (13)	0.0386 (17)	−0.0012 (10)	0.0008 (13)	−0.0034 (13)
C9	0.0344 (13)	0.0351 (13)	0.0300 (14)	0.0031 (11)	0.0007 (11)	−0.0010 (12)
C10	0.0338 (14)	0.0411 (15)	0.0327 (14)	0.0051 (12)	0.0011 (11)	−0.0048 (13)
C11	0.0324 (13)	0.0408 (15)	0.0409 (16)	−0.0010 (11)	0.0017 (12)	0.0065 (13)
C12	0.0323 (12)	0.0349 (13)	0.0379 (15)	−0.0066 (11)	−0.0018 (12)	0.0110 (13)
C13	0.0380 (14)	0.0449 (15)	0.0420 (18)	−0.0028 (12)	−0.0123 (13)	0.0027 (14)
C14	0.0486 (16)	0.0434 (15)	0.0481 (18)	0.0103 (13)	−0.0116 (15)	0.0010 (14)
C15	0.0334 (13)	0.0412 (14)	0.0386 (16)	0.0004 (11)	−0.0016 (13)	0.0139 (14)
C16	0.0533 (17)	0.0368 (14)	0.0361 (17)	0.0018 (13)	−0.0048 (13)	0.0031 (12)
Cl1	0.1066 (7)	0.0495 (4)	0.0638 (6)	0.0242 (4)	−0.0260 (5)	−0.0104 (4)
N1	0.0408 (12)	0.0361 (11)	0.0350 (12)	0.0030 (10)	−0.0042 (10)	0.0054 (10)
N2	0.0351 (11)	0.0342 (11)	0.0376 (14)	−0.0008 (9)	−0.0044 (10)	0.0044 (10)
N3	0.0405 (12)	0.0432 (12)	0.0408 (14)	−0.0010 (10)	−0.0076 (10)	0.0066 (12)
O1	0.0438 (10)	0.0461 (10)	0.0436 (13)	0.0002 (9)	−0.0131 (9)	−0.0006 (9)
O2	0.0544 (11)	0.0449 (10)	0.0446 (12)	−0.0011 (9)	−0.0081 (10)	0.0098 (10)

Geometric parameters (Å, °)

C1—C2	1.363 (5)	C10—O2	1.212 (3)
C1—C6	1.367 (4)	C10—O1	1.327 (3)
C1—H1	0.9300	C11—N2	1.460 (3)
C2—C3	1.388 (4)	C11—C12	1.510 (3)
C2—H2	0.9300	C11—H11A	0.9700
C3—C4	1.382 (3)	C11—H11B	0.9700
C3—H3	0.9300	C12—C15	1.382 (4)
C4—C5	1.387 (4)	C12—C13	1.388 (3)
C4—C7	1.473 (3)	C13—C14	1.373 (4)
C5—C6	1.378 (4)	C13—H13	0.9300
C5—H5	0.9300	C14—C16	1.372 (4)
C6—H6	0.9300	C14—H14	0.9300
C7—N1	1.350 (3)	C15—N3	1.338 (3)
C7—C8	1.400 (3)	C15—H15	0.9300
C8—C9	1.371 (3)	C16—N3	1.321 (3)
C8—H8	0.9300	C16—Cl1	1.736 (3)
C9—N2	1.366 (3)	N1—N2	1.342 (3)
C9—C10	1.473 (4)	O1—H1A	0.8200

C2—C1—C6	120.0 (3)	O1—C10—C9	110.8 (2)
C2—C1—H1	120.0	N2—C11—C12	113.7 (2)
C6—C1—H1	120.0	N2—C11—H11A	108.8
C1—C2—C3	120.2 (3)	C12—C11—H11A	108.8
C1—C2—H2	119.9	N2—C11—H11B	108.8
C3—C2—H2	119.9	C12—C11—H11B	108.8
C4—C3—C2	120.6 (3)	H11A—C11—H11B	107.7
C4—C3—H3	119.7	C15—C12—C13	117.4 (3)
C2—C3—H3	119.7	C15—C12—C11	121.8 (2)
C3—C4—C5	118.1 (3)	C13—C12—C11	120.8 (2)
C3—C4—C7	120.6 (2)	C14—C13—C12	119.8 (3)
C5—C4—C7	121.3 (2)	C14—C13—H13	120.1
C6—C5—C4	120.9 (3)	C12—C13—H13	120.1
C6—C5—H5	119.6	C16—C14—C13	117.7 (2)
C4—C5—H5	119.6	C16—C14—H14	121.1
C1—C6—C5	120.2 (3)	C13—C14—H14	121.1
C1—C6—H6	119.9	N3—C15—C12	123.5 (2)
C5—C6—H6	119.9	N3—C15—H15	118.2
N1—C7—C8	110.2 (2)	C12—C15—H15	118.2
N1—C7—C4	120.2 (2)	N3—C16—C14	124.5 (3)
C8—C7—C4	129.6 (2)	N3—C16—Cl1	115.7 (2)
C9—C8—C7	106.0 (2)	C14—C16—Cl1	119.8 (2)
C9—C8—H8	127.0	N2—N1—C7	105.52 (19)
C7—C8—H8	127.0	N1—N2—C9	111.93 (19)
N2—C9—C8	106.3 (2)	N1—N2—C11	117.81 (19)
N2—C9—C10	124.5 (2)	C9—N2—C11	130.2 (2)
C8—C9—C10	129.1 (2)	C16—N3—C15	117.0 (2)
O2—C10—O1	123.3 (2)	C10—O1—H1A	109.5
O2—C10—C9	125.9 (2)		
C6—C1—C2—C3	−0.7 (5)	N2—C11—C12—C13	83.1 (3)
C1—C2—C3—C4	0.6 (5)	C15—C12—C13—C14	0.1 (4)
C2—C3—C4—C5	−0.6 (4)	C11—C12—C13—C14	179.3 (2)
C2—C3—C4—C7	179.4 (3)	C12—C13—C14—C16	−0.6 (4)
C3—C4—C5—C6	0.6 (4)	C13—C12—C15—N3	0.7 (4)
C7—C4—C5—C6	−179.3 (2)	C11—C12—C15—N3	−178.5 (2)
C2—C1—C6—C5	0.8 (5)	C13—C14—C16—N3	0.5 (4)
C4—C5—C6—C1	−0.7 (5)	C13—C14—C16—Cl1	−178.4 (2)
C3—C4—C7—N1	−175.6 (2)	C8—C7—N1—N2	0.4 (3)
C5—C4—C7—N1	4.3 (4)	C4—C7—N1—N2	−178.8 (2)
C3—C4—C7—C8	5.4 (4)	C7—N1—N2—C9	−0.9 (3)
C5—C4—C7—C8	−174.7 (3)	C7—N1—N2—C11	−178.79 (19)
N1—C7—C8—C9	0.2 (3)	C8—C9—N2—N1	1.0 (3)
C4—C7—C8—C9	179.3 (2)	C10—C9—N2—N1	178.7 (2)
C7—C8—C9—N2	−0.7 (3)	C8—C9—N2—C11	178.6 (2)
C7—C8—C9—C10	−178.3 (2)	C10—C9—N2—C11	−3.7 (4)
N2—C9—C10—O2	6.5 (4)	C12—C11—N2—N1	86.5 (3)
C8—C9—C10—O2	−176.4 (3)	C12—C11—N2—C9	−90.9 (3)
N2—C9—C10—O1	−174.9 (2)	C14—C16—N3—C15	0.2 (4)
C8—C9—C10—O1	2.2 (3)	Cl1—C16—N3—C15	179.12 (18)

N2—C11—C12—C15	−97.8 (3)	C12—C15—N3—C16	−0.8 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1A···N3^{i}	0.82	1.98	2.794 (3)	176

Symmetry codes: (i) *x*+1/4, −*y*+1/4, *z*+5/4.

Fig. 1

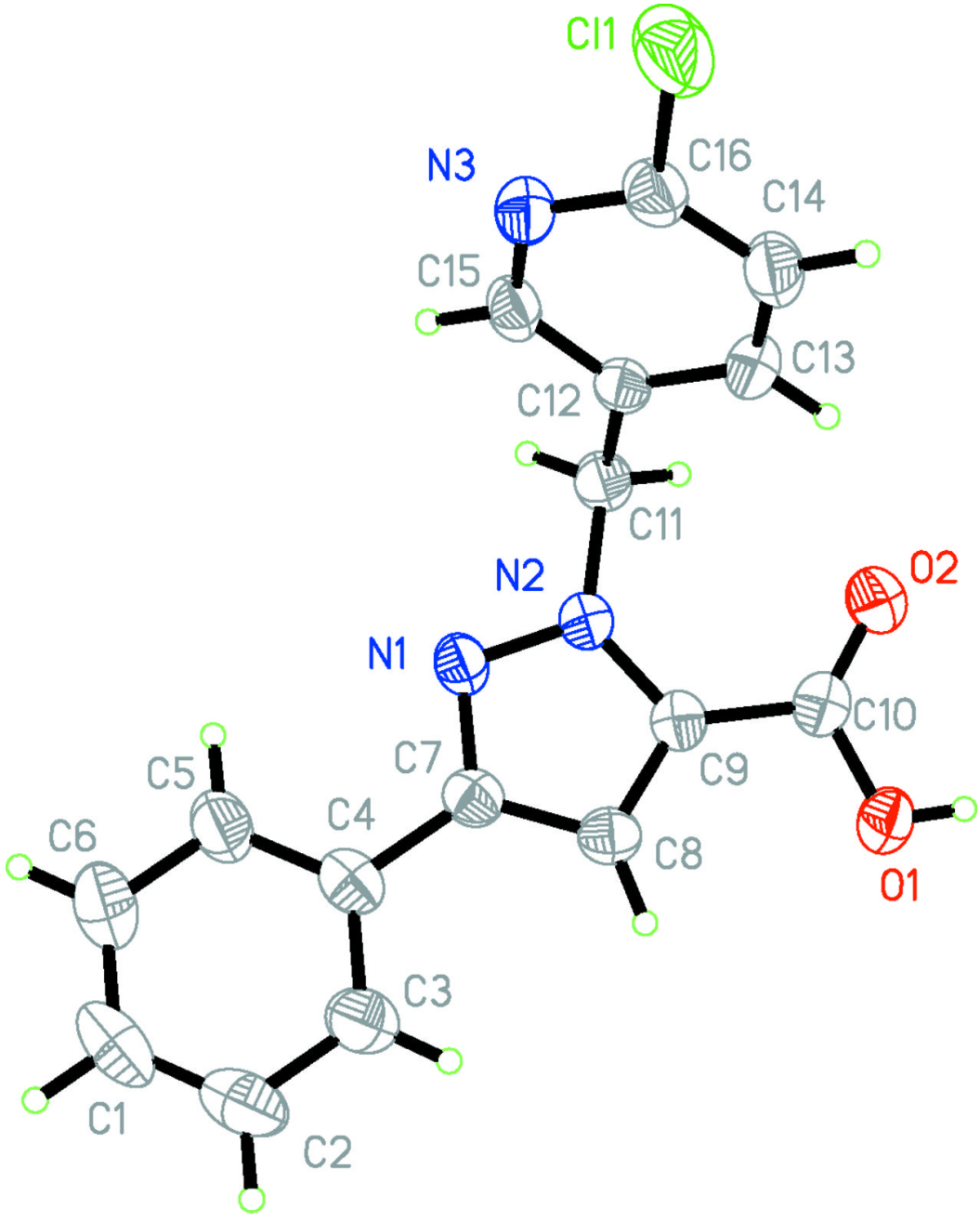
Cl1
N3
C16
C14
C15
C13
C12
C11
N2
O2
N1
C10
C7
C9
C5
C4
C6
C8
O1
C3
C1
C2

Fig. 2