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3-Phenyl-1*H*-pyrazole-5-carboxylic acid

Xiao-Yuan Zhang,[a]* Wei Liu,[b] Wei Tang[c] and Yue-Bo Lai[d]

[a]College of Chemistry and Life Sciences, Gannan Normal University, Ganzhou 341000, People's Republic of China, [b]China Tobacco Jiang Xi Industrial Company R&D Center, Nanchang 330047, People's Republic of China, [c]The College Rear Service Company, Jiangsu Food Science College, Huai'an 223001, People's Republic of China, and [d]College of Sport, Gannan Normal University, Ganzhou 341000, People's Republic of China
Correspondence e-mail: julia021326@163.com

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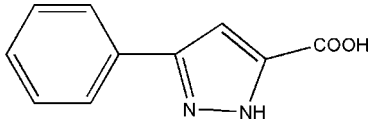
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.003 Å; R factor = 0.054; wR factor = 0.167; data-to-parameter ratio = 13.5.

In the title compound, $C_{10}H_8N_2O_2$, the dihedral angle between the phenyl and pyrazole ring planes is 21.27 (6)°. Intermolecular O—H···O and N—H···N hydrogen bonds link the molecules into chains along c. These are further connected by intermolecular C—H···O hydrogen bonds, resulting in a two-dimensional framework.

Related literature

For information on the pharmaceutical properties of the title compound, see: Shin *et al.* (2005).



Experimental

Crystal data

$C_{10}H_8N_2O_2$
M_r = 188.18
Monoclinic, $P2_1/c$
a = 10.2391 (12) Å
b = 5.3647 (6) Å
c = 17.5907 (17) Å
β = 106.581 (8)°
V = 926.07 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 291 (3) K
0.32 × 0.30 × 0.18 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.970, T_{max} = 0.983
9603 measured reflections
1817 independent reflections
1377 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.167
S = 1.07
1817 reflections
135 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···N1^{i}	0.91 (2)	2.10 (2)	2.961 (2)	158.0 (17)
O1—H1···O2^{ii}	0.876 (10)	1.771 (10)	2.6462 (19)	178 (2)
C7—H7···O2^{iii}	0.93	2.70	3.531 (3)	150

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, -y+2, -z+1$; (iii) $-x, -y+1, -z+1$.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg & Berndt, 2005); software used to prepare material for publication: *CrystalStructure* (Rigaku/MSC, 2005).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2324).

References

Brandenburg, K. & Berndt, M. (2005). *DIAMOND*. Release 3.0c. Crystal Impact GbR, Bonn, Germany.
Rigaku/MSC (2005). *CrystalClear* and *CrystalStructure*. Versions 3.70. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Shin, Y. J., Semple, G., Gharbaoui, T., Decaire, M., Skinner, P. J. & Averbuj, C. (2005). Canadian Patent No. CAP2528834.

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supplementary materials

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3-Phenyl-1*H*-pyrazole-5-carboxylic acid

X.-Y. Zhang, W. Liu, W. Tang and Y.-B. Lai

Comment

Pyrazole carboxylic acid and ester derivatives exhibit useful pharmaceutical properties, for example as agonists for the nicotinic acid receptor, refered to as RUP25 herein (Shin *et al.*, 2005).

The molecular structure of (I) is shown in Fig. 1. The dihedral angle between the phenyl plane and the pyrazole plane is 21.27 (6) Å. The carboxylate C4—O1—O2 plane, is twisted slightly with respect to the pyrazole ring plane, the dihedral angle between them is 9.0 (1) °.

In the crystal structure, intermolecular O1—H1···O2^{i} [symmetry code: (i) 1 − *x*, 2 − *y*, 1 − *z*] and N2—H2···N1^{ii} [symmetry code: (ii) 1 − *x*, *y* − 1/2, −*z* + 3/2] hydrogen bonds link the molecules into chains, which are further connected by intermolecular C7—H7···O2^{iii} [symmetry code: (iii) −*x*, *y* + 3/2, −*z* + 3/2] hydrogen bonds (C—O =3.531 (3) Å), resulting in a two-dimensional-dimensional framework.

Experimental

To a stirred solution of NaOH (300 ml, 20%) at 350 K was added 5- phenyl-2*H*-pyrazole-3-carboxylic acid ethyl ester (21.6 g, 0.1 mol), and the solution was stirred for 8 h. The reaction mixture was cooled to 293 K, filtered. A white flocculent precipitate appeared when the filtrate was adjusted to pH = 3–5 using dilute HCl. The title compound (yield 16.90 g, 89.90%) was obtained by filtering this precipitate and crystals appropriate for data collection were obtained by recrystallization from acetone/methanol (1:1).

Refinement

All H atoms bound to C were included in calculated positions and refined as riding with C—H= 0.93 Å, and U_{iso}(H)= 1.2 U_{eq}(C). Other hydrogen atoms were located in a difference Fourier map and refined freely with U_{iso}(H) = 1.5 U_{eq}(O) and U_{iso}(H) = 1.2 U_{eq}(N) respectively.

Figures

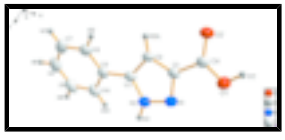
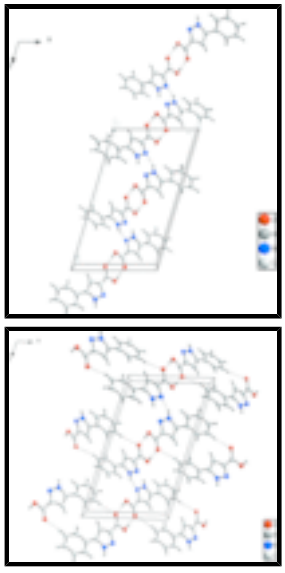


Fig. 1: The molecular structure of (I), showing 30% probability displacement ellipsoids., Fig. 2: The crystal packing of (I), view down the *b* axis, showing the one dimensional chain structure extending along the *c* axis. Hydrogen bonds are shown as dashed lines., Fig. 3: The crystal packing of (I), viewed down the *b* axis, showing the two-dimensional-dimensional hydrogen-bonded framework. Hydrogen bonds are shown as dashed lines.



3-Phenyl-1*H*-pyrazole-5-carboxylic acid

Crystal data

$C_{10}H_8N_2O_2$	F_{000} = 392
M_r = 188.18	D_x = 1.350 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71070 Å
Hall symbol: -P 2ybc	Cell parameters from 1971 reflections
a = 10.2391 (12) Å	θ = 2.7–25.0º
b = 5.3647 (6) Å	μ = 0.10 mm^{-1}
c = 17.5907 (17) Å	T = 291 (3) K
β = 106.581 (8)º	Block, colorless
V = 926.07 (17) Å^3	0.32 × 0.30 × 0.18 mm
Z = 4	

Data collection

Rigaku Saturn diffractometer	1817 independent reflections
Radiation source: rotating anode	1377 reflections with $I > 2\sigma(I)$
Monochromator: confocal	R_{int} = 0.046
T = 291(3) K	θ_{max} = 26.0º
ω scans	θ_{min} = 2.1º
Absorption correction: multi-scan (CrystalClear; Rigaku/MSC, 2005)	h = −12→12
T_{min} = 0.970, T_{max} = 0.983	k = −6→6
9603 measured reflections	l = −21→21

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.054$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.167$	$w = 1/[\sigma^2(F_o^2) + (0.1024P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
1817 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
135 parameters	$\Delta\rho_{min} = -0.16$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2$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}
O1	0.54576 (15)	0.8690 (3)	0.59886 (8)	0.0705 (5)
H1	0.571 (2)	0.983 (4)	0.5702 (13)	0.106*
O2	0.38316 (15)	0.7806 (3)	0.48748 (7)	0.0705 (5)
N1	0.45958 (15)	0.4857 (3)	0.67529 (8)	0.0535 (5)
N2	0.38121 (16)	0.3093 (3)	0.69483 (9)	0.0529 (5)
H2	0.4141 (19)	0.226 (4)	0.7415 (12)	0.063*
C1	0.39024 (17)	0.5507 (3)	0.60188 (9)	0.0486 (5)
C2	0.26909 (17)	0.4153 (3)	0.57510 (10)	0.0510 (5)
H2A	0.2045	0.4275	0.5259	0.061*
C3	0.26539 (18)	0.2603 (3)	0.63673 (10)	0.0476 (5)
C4	0.44173 (19)	0.7448 (3)	0.55878 (11)	0.0526 (5)
C5	0.16279 (18)	0.0802 (3)	0.64533 (10)	0.0494 (5)
C6	0.0304 (2)	0.0999 (4)	0.59687 (12)	0.0617 (6)
H6	0.0071	0.2280	0.5599	0.074*
C7	−0.0674 (2)	−0.0702 (5)	0.60325 (14)	0.0744 (7)
H7	−0.1560	−0.0552	0.5704	0.089*

C8	−0.0356 (3)	−0.2601 (4)	0.65715 (15)	0.0775 (7)
H8	−0.1018	−0.3747	0.6607	0.093*
C9	0.0940 (3)	−0.2798 (4)	0.70570 (14)	0.0748 (7)
H9	0.1157	−0.4083	0.7427	0.090*
C10	0.1939 (2)	−0.1121 (3)	0.70098 (12)	0.0606 (6)
H10	0.2818	−0.1275	0.7349	0.073*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0759 (10)	0.0725 (10)	0.0537 (9)	−0.0270 (7)	0.0033 (7)	0.0068 (7)
O2	0.0755 (10)	0.0812 (10)	0.0451 (8)	−0.0211 (8)	0.0018 (7)	0.0123 (7)
N1	0.0577 (9)	0.0554 (9)	0.0418 (9)	−0.0068 (7)	0.0053 (7)	0.0030 (7)
N2	0.0602 (10)	0.0535 (9)	0.0394 (9)	−0.0042 (7)	0.0053 (7)	0.0062 (7)
C1	0.0568 (11)	0.0479 (10)	0.0386 (9)	−0.0023 (8)	0.0096 (8)	0.0000 (7)
C2	0.0549 (11)	0.0528 (10)	0.0381 (10)	−0.0035 (8)	0.0016 (8)	0.0014 (7)
C3	0.0552 (11)	0.0441 (10)	0.0402 (9)	0.0009 (7)	0.0083 (8)	0.0003 (7)
C4	0.0560 (11)	0.0533 (11)	0.0448 (11)	−0.0049 (8)	0.0083 (9)	−0.0010 (8)
C5	0.0588 (11)	0.0454 (10)	0.0461 (10)	−0.0029 (8)	0.0181 (9)	−0.0055 (8)
C6	0.0646 (12)	0.0622 (12)	0.0561 (12)	−0.0084 (9)	0.0136 (10)	−0.0043 (9)
C7	0.0669 (14)	0.0816 (16)	0.0772 (16)	−0.0174 (11)	0.0244 (11)	−0.0203 (12)
C8	0.0873 (17)	0.0698 (15)	0.0907 (18)	−0.0229 (13)	0.0502 (15)	−0.0199 (13)
C9	0.106 (2)	0.0524 (12)	0.0796 (16)	−0.0020 (12)	0.0487 (16)	0.0045 (11)
C10	0.0739 (13)	0.0519 (11)	0.0594 (13)	0.0036 (9)	0.0246 (10)	0.0050 (9)

Geometric parameters (Å, °)

O1—C4	1.283 (2)	C5—C6	1.384 (3)
O1—H1	0.876 (10)	C5—C10	1.395 (3)
O2—C4	1.241 (2)	C6—C7	1.383 (3)
N1—C1	1.330 (2)	C6—H6	0.9300
N1—N2	1.347 (2)	C7—C8	1.366 (3)
N2—C3	1.352 (2)	C7—H7	0.9300
N2—H2	0.91 (2)	C8—C9	1.362 (3)
C1—C2	1.398 (2)	C8—H8	0.9300
C1—C4	1.472 (3)	C9—C10	1.382 (3)
C2—C3	1.375 (2)	C9—H9	0.9300
C2—H2A	0.9300	C10—H10	0.9300
C3—C5	1.467 (2)		
C4—O1—H1	112.4 (16)	C6—C5—C3	119.40 (16)
C1—N1—N2	103.88 (14)	C10—C5—C3	122.18 (17)
N1—N2—C3	113.67 (14)	C7—C6—C5	120.3 (2)
N1—N2—H2	118.8 (12)	C7—C6—H6	119.9
C3—N2—H2	127.2 (13)	C5—C6—H6	119.9
N1—C1—C2	111.56 (16)	C8—C7—C6	120.9 (2)
N1—C1—C4	121.00 (16)	C8—C7—H7	119.5
C2—C1—C4	127.44 (15)	C6—C7—H7	119.5
C3—C2—C1	105.65 (15)	C9—C8—C7	119.3 (2)

C3—C2—H2A	127.2	C9—C8—H8	120.4
C1—C2—H2A	127.2	C7—C8—H8	120.4
N2—C3—C2	105.24 (15)	C8—C9—C10	121.2 (2)
N2—C3—C5	123.34 (15)	C8—C9—H9	119.4
C2—C3—C5	131.40 (17)	C10—C9—H9	119.4
O2—C4—O1	124.12 (18)	C9—C10—C5	119.9 (2)
O2—C4—C1	119.29 (16)	C9—C10—H10	120.0
O1—C4—C1	116.59 (16)	C5—C10—H10	120.0
C6—C5—C10	118.42 (17)		
C1—N1—N2—C3	0.0 (2)	N2—C3—C5—C6	157.93 (18)
N2—N1—C1—C2	−0.3 (2)	C2—C3—C5—C6	−20.1 (3)
N2—N1—C1—C4	179.23 (16)	N2—C3—C5—C10	−22.1 (3)
N1—C1—C2—C3	0.4 (2)	C2—C3—C5—C10	159.78 (19)
C4—C1—C2—C3	−179.07 (18)	C10—C5—C6—C7	−0.9 (3)
N1—N2—C3—C2	0.2 (2)	C3—C5—C6—C7	179.05 (18)
N1—N2—C3—C5	−178.32 (16)	C5—C6—C7—C8	0.0 (3)
C1—C2—C3—N2	−0.33 (19)	C6—C7—C8—C9	0.7 (3)
C1—C2—C3—C5	178.01 (17)	C7—C8—C9—C10	−0.4 (3)
N1—C1—C4—O2	171.64 (19)	C8—C9—C10—C5	−0.5 (3)
C2—C1—C4—O2	−9.0 (3)	C6—C5—C10—C9	1.1 (3)
N1—C1—C4—O1	−8.9 (3)	C3—C5—C10—C9	−178.80 (18)
C2—C1—C4—O1	170.48 (18)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···N1^{i}	0.91 (2)	2.10 (2)	2.961 (2)	158.0 (17)
O1—H1···O2^{ii}	0.876 (10)	1.771 (10)	2.6462 (19)	178 (2)
C7—H7···O2^{iii}	0.93	2.70	3.5311 (25)	150

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+3/2; (ii) −*x*+1, −*y*+2, −*z*+1; (iii) −*x*, −*y*+1, −*z*+1.

Fig. 1

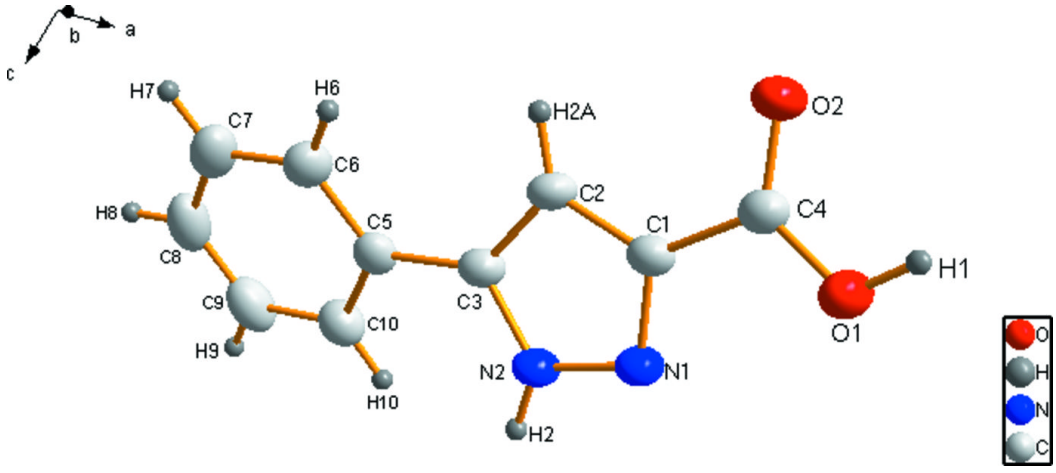

a
b
c
H7
C7
H6
C6
H8
C8
C5
C9
C10
H9
H10
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Fig. 2

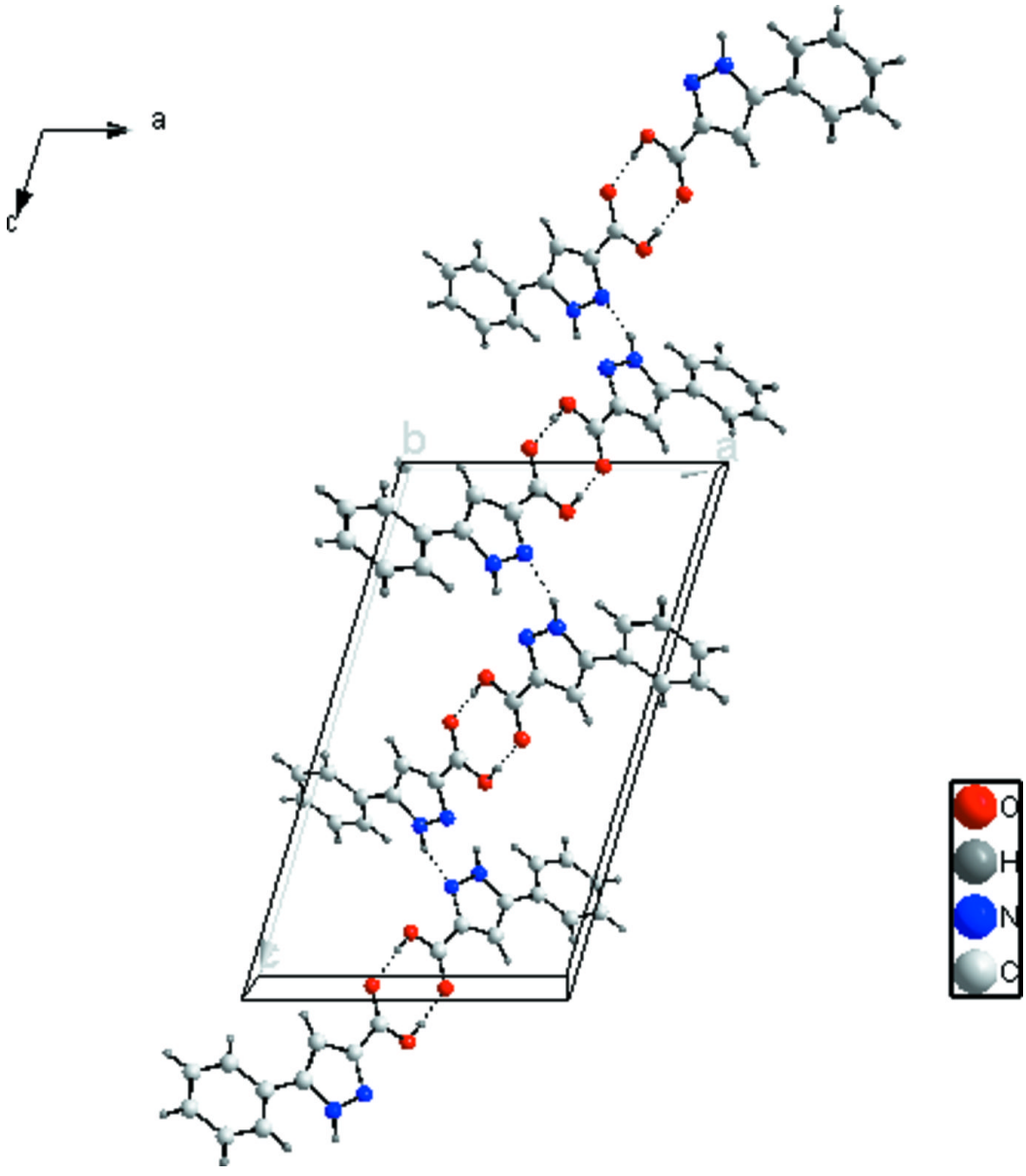

Fig. 3