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(1*Z*,2*E*)-1-(3,5-Dimethyl-1*H*-pyrazol-1-yl)ethane-1,2-dione dioxime

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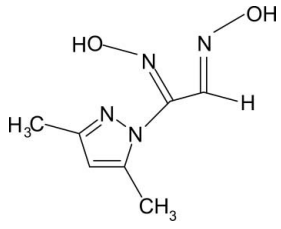
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.060; wR factor = 0.164; data-to-parameter ratio = 21.1.

In the molecule of the title compound, $C_7H_{10}N_4O_2$, the glyoxime group has an *E* configuration. In this configuration, both oxime groups are involved as donors in O—H···N intermolecular hydrogen bonding, linking the molecules to form a supramolecular structure.

Related literature

For general background, see: Sevagapandian *et al.* (2000); Marsman *et al.* (1999); Karle *et al.* (1996); Etter *et al.* (1990); Bertolasi *et al.* (1982); Chertanova *et al.* (1994). For related literature, see: Hökelek, Batı *et al.* (2001); Hökelek, Zülfikaroğlu *et al.* (2001); Büyükgüngör *et al.* (2003); Hökelek *et al.* (2004*a*,*b*,*c*); Özel Güven *et al.* (2007). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_7H_{10}N_4O_2$
M_r = 182.19
Monoclinic, $P2_1/c$
a = 9.7598 (5) Å
b = 9.8572 (6) Å
c = 9.8140 (7) Å
β = 104.254 (3)°
V = 915.08 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (2) K
0.30 × 0.20 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID-S diffractometer
Absorption correction: none
26614 measured reflections
2785 independent reflections
1879 reflections with $I > 2\sigma(I)$
R_{int} = 0.065

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.164
S = 1.02
2785 reflections
132 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1^{i}	0.97 (4)	2.51 (4)	3.209 (3)	129 (3)
O1—H1···N3^{i}	0.97 (4)	2.10 (4)	2.966 (3)	149 (3)
O2—H2···N1	0.95 (3)	1.78 (3)	2.720 (2)	172 (3)

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

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: *ORTEP-3 for Windows* (Farrugia, 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: XU2297).

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supplementary materials

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(1*Z*,2*E*)-1-(3,5-Dimethyl-1*H*-pyrazol-1-yl)ethane-1,2-dione dioxime

N. Sarikavakli, E. Sahin and T. Hökelek

Comment

Oxime and dioxime derivatives are very important compounds in the chemical industry and medicine (Sevagapandian *et al.*, 2000). The oxime (–C=N—OH) moiety is potentially ambidentate, with possibilities of coordination through nitrogen and/or oxygen atoms. It is a functional group that has not been extensively explored in crystal engineering. In the solid state, oximes are usually associated *via* O—H···N hydrogen bonds of length 2.8 Å.

Oxime groups possess stronger hydrogen-bonding capabilities than alcohols, phenols, and carboxylic acids (Marsman *et al.*, 1999), in which intermolecular hydrogen bonding combines moderate strength and directionality (Karle *et al.*, 1996) in linking molecules to form supramolecular structures; this has received considerable attention with respect to directional noncovalent intermolecular interactions (Etter *et al.*, 1990). The hydrogen-bond systems in the crystals of oximes have been analysed and a correlation between a pattern of hydrogen bonding and N—O bond lengths has been suggested (Bertolasi *et al.*, 1982). The configurational and/or conformational isomers of glyoxime derivatives (dioximes) have also been analysed (Chertanova *et al.*, 1994).

The structures of oxime and dioxime derivatives have been the subject of much interest in our laboratory; examples are 2,3-dimethylquinoxaline-dimethyl- glyoxime (1/1), [(II) Hökelek, Batı *et al.*, 2001], 1-(2,6-dimethylphenylamino)propane-1,2-dione dioxime, [(III) (Hökelek, Zülfikaroğlu *et al.*, 2001), *N*-hydroxy-2-oxo-2,*N*'-diphenylacetamidine, [(IV) (Büyükgüngör *et al.*, 2003], *N*-(3,4-dichlorophenyl)-*N*'-hydroxy-2-oxo-2-phenylacetamidine, [(V) Hökelek *et al.*, 2004*a*], *N*-hydroxy-*N*'-(1-naphthyl)-2-phenylacetamidin-2-one [(VI) Hökelek *et al.*, 2004*b*], *N*-(3-chloro-4-methylphenyl)-*N*'-hydroxy-2-oxo-2 -phenylacetamidine [(VII) Hökelek *et al.*, 2004*c*] and 2-(1*H*-benzimidazol-1-yl) −1-phenylethanone oxime [(VIII) Özel Güven *et al.*, 2007]. The structure determination of the title molecule was carried out in order to investigate the strength of the hydrogen bonding capability of the oxime groups and to compare the geometry of the oxime moieties with the previously reported ones.

In the molecule of the title compound (Fig. 1), the bond lengths and angles are generally within normal ranges (Allen *et al.*, 1987). It contains glyoxime and 3,5-dimethylpyrazole moieties. The dihedral angles between the glyoxime planes A (O1/N3/C7), B (O2/N4/C6) and pyrazole ring C (N1/N2/C2—C4) are A/B = 0.71 (15)°, A/C = 68.12 (8)° and B/C = 68.47 (9)°. In the glyoxime moiety, the N3—O1 [1.390 (2) Å] bond is slightly longer than N4—O2 [1.374 (2) Å], while C6—N4—O2 [113.0 (1)°] angle is larger than C7—N3—O1 [112.2 (2)°], reflecting the types and electron-withdrawing or -donating properties of the substituents bonded to C atoms of the glyoxime moiety.

Some significant changes in the geometry of the oxime moieties are evident when the bond lengths and angles are compared with the corresponding values in compounds (II)-(VIII) (Table 2). The glyoxime moiety has an E configuration [C6—C7—N3—O1 179.58 (15)° and C7—C6—N4—O2 − 179.53 (14)°; Chertanova *et al.*, 1994]. In this configuration, both oxime groups are involved as donors in O—H···N intermolecular hydrogen bondings (Table 1).

In the crystal structure, the intermolecular O—H···N hydrogen bonds (Table 1) link the molecules to form a supramolecular structure (Fig. 2), in which they seem to be highly effective in the stabilization of the structure.

Experimental

For the preparation of the title compound, 3,5-dimethyl-1*H*-pyrazole (0,9613 g, 10 mmol) was dissolved in ethanol (5 ml), and CH_3COONa (1.36 g, 10 mmol) in water (3 ml) was added to this solution, and then solid anti-chloroglyoxime (1.225 g, 10 mmol) was added slowly with stirring. When almost half of the anti-chloroglyoxime was added, the ligand started to precipitate. When the addition was completed, stirring was continued for 2 h at room temperature. The precipitate was filtered, washed with water and dried at room temperature in a vacuum oven and recrystallized from an ethanol-water solution (yield 1.35 g, 68%).

Refinement

Atoms H1, H2 and H7 were located in difference syntheses and refined isotropically [O1—H1 = 0.97 (4) Å, U_{iso}(H) = 0.169 (15) Å^2; O2—H2 = 0.95 (3) Å, U_{iso}(H) = 0.097 (8) Å^2 and C7—H7 = 1.00 (2) Å, U_{iso}(H) = 0.073 (6) Å^2]. The remaining H atoms were positioned geometrically, with C—H = 0.93 and 0.96 Å for aromatic and methyl H, respectively, and constrained to ride on their parent atom, with U_{iso}(H) = xU_{eq}(C), where x = 1.5 for methyl H, and x = 1.2 for aromatic H atoms.

Figures

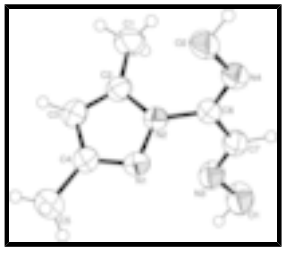

Fig. 1. The molecular structure of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

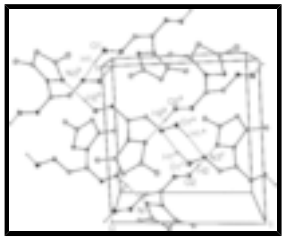

Fig. 2. A packing diagram of the title compound. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity [symmetry codes: (*a*) −*x*, *y* + 1/2, −*z* + 1/2; (*b*) −*x*, −*y*, −*z*; (*c*) *x*, −*y* + 1/2, *z* + 1/2].

(1*Z*,2E)-1-(3,5-Dimethyl-1*H*-pyrazol-1-yl)-1,2-ethanedione dioxime

Crystal data

$C_7H_{10}N_4O_2$	F_{000} = 384
M_r = 182.19	D_x = 1.322 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 3673 reflections
a = 9.7598 (5) Å	θ = 2.0–30.5º

b = 9.8572 (6) Å	μ = 0.10 mm^{-1}
c = 9.8140 (7) Å	T = 298 (2) K
β = 104.254 (3)º	Block, colourless
V = 915.08 (10) Å^3	0.30 × 0.20 × 0.15 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID-S diffractometer	1879 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.065
Monochromator: graphite	θ_{max} = 30.6º
T = 298(2) K	θ_{min} = 2.9º
ω scans	h = −13→13
Absorption correction: none	k = −14→14
26614 measured reflections	l = −13→13
2785 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.060	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.164	$w = 1/[\sigma^2(F_o^2) + (0.072P)^2 + 0.1814P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max} < 0.001$
2785 reflections	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
132 parameters	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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}

O1	−0.12971 (15)	0.10253 (16)	0.03495 (17)	0.0774 (5)
H1	−0.124 (4)	0.034 (4)	−0.034 (4)	0.169 (15)*
O2	0.29576 (13)	0.24903 (13)	0.53291 (13)	0.0596 (4)
H2	0.293 (3)	0.317 (3)	0.601 (3)	0.097 (8)*
N1	0.31551 (15)	0.05600 (13)	0.23301 (13)	0.0479 (3)
N2	0.24604 (14)	0.06268 (13)	0.33862 (13)	0.0449 (3)
N3	−0.00111 (16)	0.08524 (15)	0.13275 (16)	0.0586 (4)
N4	0.16951 (15)	0.26301 (15)	0.43425 (14)	0.0534 (4)
C1	0.2119 (3)	−0.0631 (2)	0.5483 (2)	0.0714 (6)
H1A	0.2215	0.0189	0.6026	0.107*
H1B	0.2587	−0.1360	0.6064	0.107*
H1C	0.1134	−0.0845	0.5139	0.107*
C2	0.27711 (19)	−0.04401 (17)	0.42716 (17)	0.0513 (4)
C3	0.3714 (2)	−0.12097 (18)	0.37819 (19)	0.0598 (5)
H3	0.4135	−0.2013	0.4172	0.072*
C4	0.39228 (18)	−0.05630 (17)	0.25889 (17)	0.0508 (4)
C5	0.4841 (2)	−0.0981 (2)	0.1644 (2)	0.0705 (6)
H5A	0.4299	−0.0965	0.0684	0.106*
H5B	0.5191	−0.1883	0.1884	0.106*
H5C	0.5623	−0.0365	0.1757	0.106*
C6	0.14943 (18)	0.17055 (16)	0.33936 (16)	0.0473 (4)
C7	0.01985 (19)	0.17524 (18)	0.22847 (18)	0.0544 (4)
H7	−0.048 (2)	0.251 (2)	0.233 (2)	0.073 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0613 (8)	0.0801 (10)	0.0744 (10)	0.0127 (7)	−0.0144 (7)	−0.0193 (8)
O2	0.0622 (8)	0.0612 (8)	0.0500 (7)	0.0063 (6)	0.0033 (6)	−0.0149 (6)
N1	0.0562 (8)	0.0498 (7)	0.0380 (6)	0.0046 (6)	0.0122 (6)	0.0029 (5)
N2	0.0538 (8)	0.0430 (7)	0.0376 (6)	0.0048 (5)	0.0108 (5)	0.0000 (5)
N3	0.0535 (8)	0.0575 (8)	0.0561 (8)	0.0039 (6)	−0.0033 (6)	−0.0062 (7)
N4	0.0575 (8)	0.0543 (8)	0.0463 (7)	0.0060 (6)	0.0088 (6)	−0.0067 (6)
C1	0.0967 (16)	0.0668 (12)	0.0581 (11)	0.0024 (10)	0.0331 (11)	0.0104 (9)
C2	0.0627 (10)	0.0477 (9)	0.0435 (8)	0.0029 (7)	0.0131 (7)	0.0044 (6)
C3	0.0736 (12)	0.0511 (10)	0.0548 (10)	0.0162 (8)	0.0159 (8)	0.0101 (8)
C4	0.0540 (9)	0.0531 (9)	0.0435 (8)	0.0082 (7)	0.0086 (7)	0.0001 (7)
C5	0.0738 (13)	0.0821 (14)	0.0588 (11)	0.0203 (10)	0.0223 (9)	−0.0025 (10)
C6	0.0546 (9)	0.0445 (8)	0.0422 (8)	0.0052 (7)	0.0108 (6)	−0.0017 (6)
C7	0.0563 (10)	0.0535 (9)	0.0500 (9)	0.0082 (8)	0.0067 (7)	−0.0036 (7)

Geometric parameters (Å, °)

O1—N3	1.3901 (19)	C1—H1C	0.9600
O1—H1	0.97 (4)	C2—C3	1.368 (2)
O2—N4	1.3738 (18)	C2—C1	1.492 (3)
O2—H2	0.95 (3)	C3—H3	0.9300
N1—C4	1.326 (2)	C4—C3	1.392 (2)
N2—C2	1.350 (2)	C4—C5	1.498 (2)

N2—N1	1.3727 (18)	C5—H5A	0.9600
N2—C6	1.422 (2)	C5—H5B	0.9600
N3—C7	1.271 (2)	C5—H5C	0.9600
N4—C6	1.283 (2)	C6—C7	1.452 (2)
C1—H1A	0.9600	C7—H7	1.00 (2)
C1—H1B	0.9600		
N3—O1—H1	102 (2)	C2—C3—H3	126.5
N4—O2—H2	104.5 (15)	C4—C3—H3	126.5
C4—N1—N2	104.85 (13)	N1—C4—C3	110.52 (15)
C2—N2—N1	111.97 (13)	N1—C4—C5	120.50 (16)
C2—N2—C6	128.54 (14)	C3—C4—C5	128.99 (16)
N1—N2—C6	119.41 (12)	C4—C5—H5A	109.5
C7—N3—O1	112.23 (15)	C4—C5—H5B	109.5
C6—N4—O2	112.99 (13)	H5A—C5—H5B	109.5
C2—C1—H1A	109.5	C4—C5—H5C	109.5
C2—C1—H1B	109.5	H5A—C5—H5C	109.5
H1A—C1—H1B	109.5	H5B—C5—H5C	109.5
C2—C1—H1C	109.5	N4—C6—N2	123.49 (15)
H1A—C1—H1C	109.5	N4—C6—C7	118.07 (15)
H1B—C1—H1C	109.5	N2—C6—C7	118.43 (14)
N2—C2—C3	105.65 (15)	N3—C7—C6	118.92 (16)
N2—C2—C1	122.60 (16)	N3—C7—H7	124.3 (12)
C3—C2—C1	131.74 (16)	C6—C7—H7	116.8 (12)
C2—C3—C4	107.00 (15)		
N2—N1—C4—C3	0.52 (19)	N1—N2—C6—C7	67.2 (2)
N2—N1—C4—C5	−179.96 (16)	O1—N3—C7—C6	179.58 (15)
C2—N2—N1—C4	−0.89 (18)	O2—N4—C6—N2	1.6 (2)
C6—N2—N1—C4	−177.87 (14)	O2—N4—C6—C7	−179.53 (14)
N1—N2—C2—C3	0.90 (19)	N2—C2—C3—C4	−0.5 (2)
C6—N2—C2—C3	177.53 (16)	C1—C2—C3—C4	178.3 (2)
N1—N2—C2—C1	−178.03 (16)	N1—C4—C3—C2	0.0 (2)
C6—N2—C2—C1	−1.4 (3)	C5—C4—C3—C2	−179.47 (19)
C2—N2—C6—N4	69.6 (2)	N4—C6—C7—N3	179.27 (17)
N1—N2—C6—N4	−114.00 (18)	N2—C6—C7—N3	−1.8 (2)
C2—N2—C6—C7	−109.24 (19)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1^{i}	0.97 (4)	2.51 (4)	3.209 (3)	129 (3)
O1—H1···N3^{i}	0.97 (4)	2.10 (4)	2.966 (3)	149 (3)
O2—H2···N1	0.95 (3)	1.78 (3)	2.720 (2)	172 (3)

Symmetry codes: (i) −*x*, −*y*, −*z*.

Table 2. Comparison of the bond lengths and angles (Å, °) in the oxime moieties of the title compound, (I), with the corresponding values in the related compounds (II)–(VIII).

Bond/angle	(I)	(II)	(III)	(IV)	(V)	(VI)	(VII)	(VIII)

N3—O1	1.390 (2)	1.403 (2)	1.423 (3)	1.417 (1)	1.429 (4)	1.424 (2)	1.416 (3)	1.383 (7)
N4—O2	1.374 (2)	1.396 (2)	1.396 (3)				1.397 (3)	
N3—C7	1.271 (2)	1.281 (2)	1.290 (3)	1.290 (1)	1.241 (6)	1.289 (2)	1.282 (3)	1.300 (7)
N4—C6	1.283 (2)	1.281 (2)	1.282 (3)				1.289 (3)	
C6—C7	1.452 (2)	1.477 (3)	1.489 (3)	1.510 (1)	1.551 (7)	1.513 (2)	1.501 (4)	1.491 (8)
		1.473 (3)					1.502 (4)	
C6—C7—N3	118.9 (2)	115.2 (2)	116.6 (2)	114.3 (1)	118.3 (5)	113.2 (1)	114.4 (2)	115.3 (5)
C7—C6—N4	118.1 (2)	115.0 (2)	115.0 (2)				113.4 (2)	
C7—N3—O1	112.2 (2)	112.4 (1)	109.4 (2)	110.7 (1)	112.2 (4)	110.6 (1)	110.7 (2)	111.4 (5)
C6—N4—O2	113.0 (1)	112.2 (1)	111.5 (2)				111.1 (2)	

Notes: (II): 2,3-dimethylquinoxaline dimethylglyoxime (1/1) (Hökelek, Batı *et al.*, 2001), (III): 1-(2,6-dimethylphenylamino)propane-1,2-dione dioxime (Hökelek, Zülfikaroğlu & Batı, 2001), (IV): *N*-hydroxy-2-oxo-2,*N*'-di- phenylacetamidine (Büyükgüngör *et al.*, 2003), (V): *N*-(3,4-dichloro- phenyl)-*N*'-hydroxy-2-oxo-2-phenylacetamidine (Hökelek *et al.*, 2004*a*), (VI): *N*-hydroxy-*N*'-(1-naphthyl)-2-phenylacetamidin-2-one (Hökelek *et al.*, 2004*b*), (VII): *N*-(3-chloro-4-methylphenyl)-*N*'-hydroxy-2-oxo-2-phenylacetamidine-2,3- dimethylquinoxaline dimethylglyoxime (1/1) (Hökelek *et al.*, 2004*c*) and (VIII): 2-(1*H*-benzimidazol-1-yl)-1-phenylethanone oxime (Özel Güven *et al.*, 2007).

Fig. 1

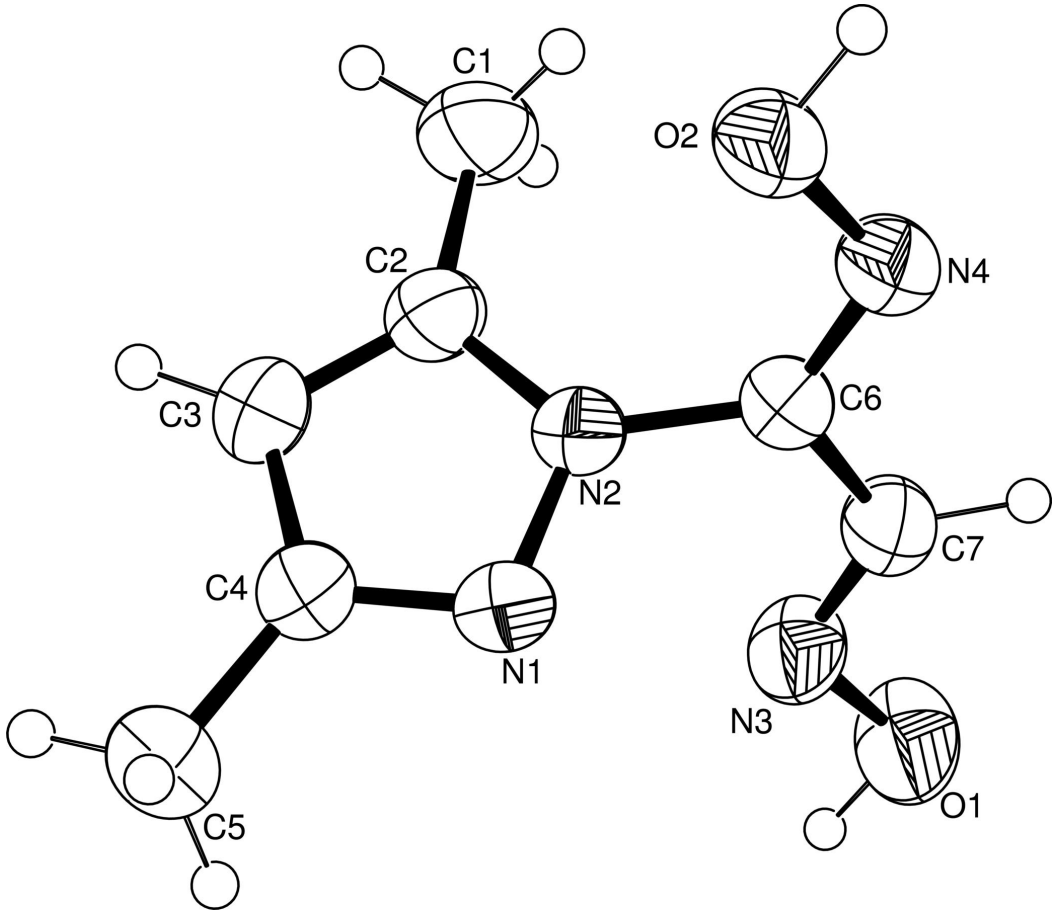

Fig. 2