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3-Hydroxy-3-methyl-4-(3-methylquinoxalin-2-yl)butan-2-one

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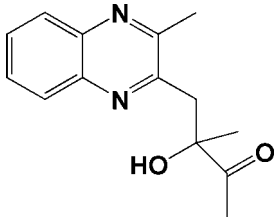
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.035; wR factor = 0.098; data-to-parameter ratio = 9.0.

In the title compound, $C_{14}H_{16}N_2O_2$, the 2-functionalized side-arm adopts a cisoid conformation. The molecule is stabilized by an intramolecular O—H···N hydrogen bond. In the crystal structure, adjacent molecules are connected *via* O—H···O hydrogen bonds to form chains running parallel to the *a* axis. These chains are further linked by C—H···N hydrogen bonds to form double-stranded layers stacking back-to-back along the *c*-axis direction.

Related literature

For the structure of an anthraquinone analogue, see: Baron *et al.* (1984). For related references, see: Allen (2002); Anaflous *et al.* (2004); Benchat *et al.* (2003); Bouabdallah *et al.* (2006); El-Bendary *et al.* (1996); Keeble *et al.* (2001); Lin (1996); Milkevitch *et al.* (1996); Molnar *et al.* (1994); Nallas & Brewer (1996); Ramalho *et al.* (2004); Waring *et al.* (2002).



Experimental

Crystal data

$C_{14}H_{16}N_2O_2$
M_r = 244.29
Triclinic, $P\overline{1}$
a = 5.3212 (6) Å
b = 7.3946 (9) Å
c = 16.327 (2) Å
α = 80.487 (14)°
β = 81.612 (14)°
γ = 80.917 (14)°
V = 620.91 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 173 (2) K
0.50 × 0.46 × 0.46 mm

Data collection

Stoe IPDS diffractometer
Absorption correction: none
4479 measured reflections
2048 independent reflections
1624 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.098
S = 1.03
2048 reflections
227 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H10···O2^{i}	0.878 (15)	2.440 (16)	3.0967 (14)	132.0 (13)
O1—H10···N1	0.878 (15)	2.170 (16)	2.8082 (14)	129.2 (14)
C2—H2···O2^{i}	0.993 (16)	2.436 (15)	3.2898 (18)	143.7 (12)
C5—H5···N2^{ii}	0.991 (17)	2.619 (17)	3.5676 (18)	160.3 (12)
C12—H12B···O1^{iii}	0.963 (16)	2.513 (16)	3.4373 (19)	160.8 (12)

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

Data collection: *EXPOSE* (Stoe & Cie, 2000); cell refinement: *CELL* (Stoe & Cie, 2000); data reduction: *INTEGRATE* (Stoe & Cie, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG2059).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Anaflous, A., Benchat, N., Mimouni, M., Abouricha, S., Ben Hadda, T., El Bali, B., Hakkou, A. & Hacht, B. (2004). *Lett. Drug. Des. Disc.* **1**, 35–44.
Baron, M., Giorgi-Renault, S., Renault, J., Mailliet, P., Carré, D. & Etienne, J. (1984). *Can. J. Chem.* **62**, 526–530.
Benchat, N., El Bali, B., Abouricha, S., Moueqqit, M., Mimouni, M. & Ben Hadda, T. (2003). *Chem. Prep. Server, Med./Pharm. Chem.* **2**, 1–18.
Bouabdallah, I., Zidane, I., Touzani, R., Hacht, B. & Ramdani, A. (2006). *ARKIVOC*, pp. 77–81.
El-Bendary, E. R., El-Ashmawy, M. B., Barghash, A. M., Shehata, I. A. & El-Kerdawy, M. M. (1996). *Boll. Chim. Farm.* **135**, 617–620.
Keeble, J., Al-Swayeh, O. A. & Moore, P. K. (2001). *Br. J. Pharmacol.* **133**, 1023–1028.
Lin, S.-K. (1996). *Molecules*, **1**, 37–40.
Milkevitch, M., Brauns, E. & Brewer, K. J. (1996). *Inorg. Chem.* **35**, 1737–1739.
Molnar, S. M., Nallas, G., Bridgewater, J. S. & Brewer, K. J. (1994). *J. Am. Chem. Soc.* **116**, 5206–5210.
Nallas, G. N. A. & Brewer, K. J. (1996). *Inorg. Chim. Acta*, **253**, 7–13.
Ramalho, T. C., Da Cunha, E. F. & De Alencastro, R. B. (2004). *J. Mol. Struct. (THEOCHEM)*, **676**, 149–153.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (2000). *EXPOSE*, *CELL* and *INTEGRATE*. Stoe & Cie GmbH, Darmstadt, Germany.
Waring, M. J., Ben Hadda, T., Kotchevar, A. T., Ramdani, A., Touzani, R., Elkadiri, S., Hakkou, A., Bouakka, M. & Ellis, T. (2002). *Molecules*, **7**, 641–656.

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supplementary materials

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3-Hydroxy-3-methyl-4-(3-methylquinoxalin-2-yl)butan-2-one

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Comment

Quinoxaline derivatives containing one or two functionalized side-arms at various positions have emerged as potentially interesting drugs owing to their DNA interactive behaviour (Nallas *et al.*, 1996; 253, Milkevitch *et al.*, 1996; Molnar *et al.*, 1994), as antidiabetic agents (El-Bendary *et al.*, 1996), anti-HIV agents (Keeble *et al.*, 2001), or NMDA receptor antagonists (Lin *et al.*, 1996). We recently investigated the DNA interactions and evaluation of anticancer, anti-tuberculosis and antifungal activity of some 2,3-bifunctionalized quinoxalines: (Waring *et al.*, 2002). In addition some anti-mycobacterial screening of imidazo[1,2-*a*]-pyrimidine (-pyridine) derivatives containing the (O═N—C—N) or (O═C—C—N) pharmacophore have been undertaken. These studies showed that compounds bearing a formyl, hydroxy or nitroso side chain in the 3-position are highly active as anti-tubercular and anti-bacterial agents (Benchat *et al.*, 2003; Anaflous *et al.*, 2004). From the general structure-activity relationship, it appears that functionalized side chain(*s*) characterized by pendant sites such as N—C—Y—*Z* [where Y—*Z* is N=O, C=O or CH_2—OH] are crucial for bioactivity. The two terminal hetero-atom centres, N and *Z*, seem to have critical interactions with the bacterial cell receptors or as ligand donors in metal complexation (Ramalho *et al.*, 2004). In a continuation of our investigations of the structure-activity relationship off functionalized-quinoxalines, we describe here the synthesis and crystal structure of the title compound, (labelled as 4 in the scheme).

The molecular structure of compound (4), is illustrated in Fig. 1. The bond lengths and angles are normal (Allen *et al.*, 2002), and similar to those observed in an anthraquinone analogue (Baron *et al.*, 1984). The pyrazine ring and the benzene ring of the quinoxaline unit are almost coplanar, making a dihedral angle of 1.66 (7)°. The fuctionalized arm at atom C8 adopts a cisoidal-conformation, with an intramolecular hydrogen bond, O2—H2O—N1 distance of 2.243 (6) Å. Bonds C7—C9 and C8—C10 lie in the plane of quinoxaline system, while atoms in the bond C10—C11 deviate from this plane by 5.63 (8)°. In the crystal structure adjacent molecules are connected *via* O—H···O hydrogen bonds to form chains running parallel to the *a* axis (Fig. 2 and Table 1). These chains are further linked by C—H···N hydrogen bonds to form double stranded layers stacking back-to-back along the *c* axis (Fig. 3).

These preliminary results prompt several pertinent observations: (i) this new synthetic method of dissymmetric quinoxalines can furnish many interesting models for studying the interaction of quinoxaline derivative antibiotics with DNA, because hydrogen bonding of a proton to the negatively charged centers of DNA is generally favoured; (ii) The non-geometric symmetry enables us to prepare armed quinoxalines as bis-intercalators for cancer therapy and (iii) bis-functionalized quinoxalines can easily be prepared from 2,3-dimethyl quinoxaline and other dimethyl benzoquinoxaline precursors.

Experimental

Benzene-1,2-diamine (1) (10.8 g, 100 mmol) and biacetyl (2) (8.8 g, 100 mmol) were stirred in refluxing ethanol (100 ml) for 4–5 h. After cooling to room temperature, the volume of solvent was reduced using a rota-evaporator. The desired compound (4), was separated from the white major-product (3) as analytically pure yellow crystals, by using chromatography on silica and hexane as eluant [compound (3): 3 g, 72% and compound (4): 1.2 g, 25%]. 2,3-dimethylquinoxaline (3): R_f = 0. 49

(2/3 ether/hexane). *M*.p. = 105–107°C; ^{1}H-NMR (60 MHz, $CDCl_3$) δ p.p.m.: 7.6–7.9 (m, 4H, Ph); 2.75 (s, 6H, 2CH3). ^{13}C NMR (300 MHz, D_2O) δ p.p.m.: 153.48, 143.08, 128.85, 128.66, 128.31, 23.19. MS (EI) m/*z*: $[MH]^+$ = 159 (100%), 160. The structure of compound (3) can be compared to 2,3-dimethylquinoxaline, previously described by (Bouabdallah *et al.*, 2006). 3-hydroxy-3-methyl-4-(3-methyl-2-quinoxalinyl)-2-butanone (4): *M*.p. = 92–95°C. R_f = 0.1 (2/3 ether/hexane). IR (KBr, ν cm^{-}1): 3462 (OH), 3051, 2977, 2936, 1702 (C=O), 1567 (C=N), 1490, 1452. ^{1}H NMR (300 MHz, $CDCl_3$) δ p.p.m.: 8.2–7.8 (m, 4H, Ph), 5.9 (s, 1H, OH), 3.8–3.2 (HA—B, J = 17 Hz, 2H, CH2), 2.8 (s, 3H, CH3), 2.5 (s, 3H, CH3), 1.5 (s, 3H, CH3). ^{13}C NMR (300 MHz, D_2O) δ p.p.m.: 214.5, 153.89, 153.8, 149.95, 139.38, 129.46, 129.23, 128.57, 127.7, 79.6, 42.44, 25.82, 25.0, 22.7. MS (EI) m/*z*: $[M—H]^+$ = 245 (100%).

Refinement

The H atoms could all be located from difference Fourier maps and were freely refined with isotropic parameters: C—H = 0.95 (2) − 1.018 (18) Å.

Figures

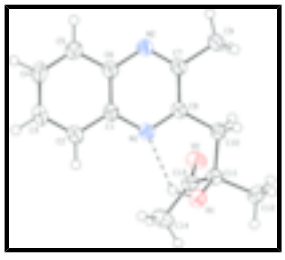


Fig. 1. Molecular structure of compound (4), showing the atomic numbering scheme and displacement ellipsoids drawn at the 50% probability level. The intramolecular O2—H2···N1 interaction is shown as a dashed line.

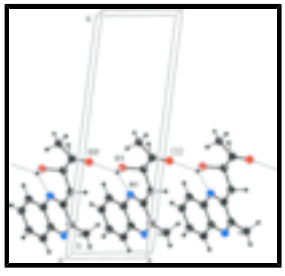


Fig. 2. The crystal packing of compound (4) viewed down the *b* axis. The intramolecular O—H···N hydrogen bond and the intermolecular O—H···O hydrogen bonds are shown as dashed lines [symmetry operation (i) = *x* − 1,*y*,*z*]

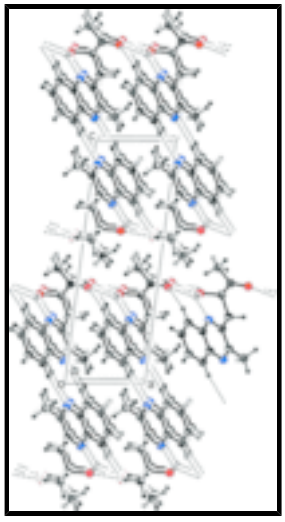


Fig. 3. The crystal packing of compound (4) viewed down the *b* axis, showing both the O—H···O, C—H···O, and C—H···N hydrogen bonds as dashed lines.

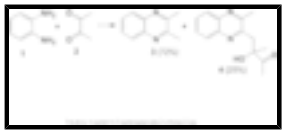


Fig. 4. The formation of the title compound.

3-Hydroxy-3-methyl-4-(3-methylquinoxalin-2-yl)butan-2-one

Crystal data

$C_{14}H_{16}N_2O_2$	$Z = 2$
$M_r = 244.29$	$F_{000} = 260$
Triclinic, $P\overline{1}$	$D_x = 1.307$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 368 K
$a = 5.3212$ (6) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 7.3946$ (9) Å	Cell parameters from 5263 reflections
$c = 16.327$ (2) Å	$\theta = 2.6$–26.0°
$\alpha = 80.487$ (14)°	$\mu = 0.09$ mm^{-1}
$\beta = 81.612$ (14)°	$T = 173$ (2) K
$\gamma = 80.917$ (14)°	Block, pale yellow
$V = 620.91$ (13) Å^3	0.50 × 0.46 × 0.46 mm

Data collection

Stoe IPDS diffractometer	2048 independent reflections
Radiation source: fine-focus sealed tube	1624 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.042$
Detector resolution: 0.81Å pixels mm^{-1}	$\theta_{max} = 25.0^\circ$
$T = 173(2)$ K	$\theta_{min} = 2.8^\circ$
φ rotation scans	$h = -6 \rightarrow 5$
Absorption correction: none	$k = -8 \rightarrow 8$
4479 measured reflections	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.035$	All H-atom parameters refined
$wR(F^2) = 0.098$	$w = 1/[\sigma^2(F_o^2) + (0.0643P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = <0.001$
2048 reflections	$\Delta\rho_{max} = 0.23$ e Å^{-3}
227 parameters	$\Delta\rho_{min} = -0.14$ 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 $F^2 > 2$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	0.52890 (17)	0.50679 (13)	0.36113 (6)	0.0316 (3)
H10	0.483 (3)	0.412 (2)	0.3438 (10)	0.045 (5)*
O2	1.12728 (17)	0.23419 (13)	0.39326 (6)	0.0356 (3)
N1	0.6921 (2)	0.23771 (14)	0.25476 (6)	0.0280 (3)
N2	0.9341 (2)	0.20339 (15)	0.09209 (6)	0.0307 (3)
C1	0.6328 (3)	0.10928 (17)	0.21190 (8)	0.0273 (3)
C2	0.4461 (3)	−0.00391 (19)	0.24855 (9)	0.0332 (3)
H2	0.359 (3)	0.010 (2)	0.3057 (10)	0.040 (4)*
C3	0.3836 (3)	−0.12887 (19)	0.20466 (10)	0.0388 (4)
H3	0.254 (4)	−0.204 (2)	0.2292 (11)	0.052 (5)*
C4	0.5069 (3)	−0.1458 (2)	0.12373 (10)	0.0405 (4)
H4	0.457 (3)	−0.237 (2)	0.0960 (11)	0.052 (5)*
C5	0.6899 (3)	−0.03915 (19)	0.08700 (9)	0.0371 (4)
H5	0.785 (3)	−0.052 (2)	0.0309 (11)	0.044 (4)*
C6	0.7557 (3)	0.09258 (17)	0.13013 (8)	0.0287 (3)
C7	0.9864 (3)	0.32600 (17)	0.13386 (8)	0.0279 (3)
C8	0.8618 (2)	0.34390 (16)	0.21727 (7)	0.0252 (3)
C9	1.1810 (3)	0.4483 (2)	0.09244 (9)	0.0359 (3)
H9A	1.317 (3)	0.442 (2)	0.1278 (10)	0.042 (4)*

H9B	1.253 (4)	0.411 (2)	0.0387 (12)	0.054 (5)*
H9C	1.103 (4)	0.574 (3)	0.0839 (11)	0.058 (5)*
C10	0.9241 (3)	0.48740 (18)	0.26329 (8)	0.0276 (3)
H10A	0.875 (3)	0.613 (2)	0.2328 (9)	0.032 (4)*
H10B	1.111 (3)	0.476 (2)	0.2620 (10)	0.040 (4)*
C11	0.8009 (2)	0.47863 (17)	0.35441 (8)	0.0274 (3)
C12	0.8763 (3)	0.6337 (2)	0.39282 (9)	0.0335 (3)
H12A	0.806 (3)	0.752 (2)	0.3623 (10)	0.040 (4)*
H12B	1.060 (3)	0.627 (2)	0.3879 (10)	0.041 (4)*
H12C	0.810 (3)	0.623 (2)	0.4549 (12)	0.050 (5)*
C13	0.9016 (2)	0.29549 (18)	0.40545 (8)	0.0284 (3)
C14	0.7209 (3)	0.2059 (2)	0.47249 (10)	0.0400 (4)
H14A	0.584 (4)	0.172 (2)	0.4482 (11)	0.046 (4)*
H14B	0.643 (4)	0.296 (3)	0.5095 (12)	0.059 (5)*
H14C	0.806 (4)	0.094 (3)	0.5036 (12)	0.058 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0201 (5)	0.0371 (5)	0.0395 (5)	−0.0025 (4)	−0.0070 (4)	−0.0098 (4)
O2	0.0223 (6)	0.0455 (6)	0.0360 (5)	0.0016 (4)	−0.0034 (4)	−0.0031 (4)
N1	0.0269 (7)	0.0313 (6)	0.0267 (6)	−0.0073 (5)	−0.0045 (4)	−0.0022 (4)
N2	0.0348 (7)	0.0342 (6)	0.0245 (5)	−0.0108 (5)	−0.0037 (5)	−0.0026 (4)
C1	0.0274 (7)	0.0276 (6)	0.0282 (7)	−0.0060 (5)	−0.0075 (5)	−0.0016 (5)
C2	0.0314 (8)	0.0342 (7)	0.0340 (7)	−0.0098 (6)	−0.0024 (6)	−0.0017 (6)
C3	0.0374 (10)	0.0344 (7)	0.0469 (9)	−0.0164 (6)	−0.0062 (7)	−0.0005 (6)
C4	0.0484 (10)	0.0344 (7)	0.0446 (8)	−0.0155 (6)	−0.0116 (7)	−0.0080 (6)
C5	0.0445 (9)	0.0376 (7)	0.0327 (7)	−0.0120 (6)	−0.0060 (6)	−0.0083 (6)
C6	0.0298 (8)	0.0300 (6)	0.0270 (7)	−0.0061 (5)	−0.0070 (5)	−0.0018 (5)
C7	0.0292 (8)	0.0302 (6)	0.0249 (6)	−0.0070 (5)	−0.0065 (5)	−0.0001 (5)
C8	0.0232 (7)	0.0279 (6)	0.0250 (6)	−0.0050 (5)	−0.0063 (5)	−0.0008 (5)
C9	0.0396 (9)	0.0417 (8)	0.0283 (7)	−0.0175 (7)	−0.0010 (6)	−0.0017 (6)
C10	0.0253 (8)	0.0321 (7)	0.0274 (7)	−0.0087 (5)	−0.0054 (5)	−0.0040 (5)
C11	0.0188 (7)	0.0352 (7)	0.0297 (7)	−0.0040 (5)	−0.0045 (5)	−0.0072 (5)
C12	0.0250 (8)	0.0413 (8)	0.0380 (8)	−0.0046 (6)	−0.0057 (6)	−0.0153 (6)
C13	0.0231 (7)	0.0390 (7)	0.0250 (6)	−0.0031 (5)	−0.0048 (5)	−0.0096 (5)
C14	0.0283 (8)	0.0502 (9)	0.0364 (8)	−0.0016 (7)	−0.0024 (6)	0.0033 (7)

Geometric parameters (Å, °)

O1—C11	1.4201 (16)	C7—C9	1.4958 (19)
O1—H10	0.875 (19)	C8—C10	1.4993 (17)
O2—C13	1.2148 (15)	C9—H9A	0.980 (18)
N1—C8	1.3078 (17)	C9—H9B	0.97 (2)
N1—C1	1.3673 (17)	C9—H9C	0.95 (2)
N2—C7	1.3054 (17)	C10—C11	1.5311 (18)
N2—C6	1.3652 (18)	C10—H10A	0.994 (15)
C1—C2	1.404 (2)	C10—H10B	0.980 (18)
C1—C6	1.4139 (19)	C11—C12	1.5245 (18)

C2—C3	1.365 (2)	C11—C13	1.5308 (18)
C2—H2	0.992 (16)	C12—H12A	0.980 (17)
C3—C4	1.403 (2)	C12—H12B	0.963 (18)
C3—H3	0.956 (19)	C12—H12C	1.018 (18)
C4—C5	1.359 (2)	C13—C14	1.489 (2)
C4—H4	0.959 (19)	C14—H14A	0.958 (19)
C5—C6	1.4050 (19)	C14—H14B	0.98 (2)
C5—H5	0.991 (17)	C14—H14C	0.98 (2)
C7—C8	1.4423 (18)		
C11—O1—H10	106.8 (11)	C7—C9—H9C	110.3 (11)
C8—N1—C1	117.85 (11)	H9A—C9—H9C	106.9 (15)
C7—N2—C6	117.53 (11)	H9B—C9—H9C	108.9 (15)
N1—C1—C2	119.91 (12)	C8—C10—C11	115.04 (12)
N1—C1—C6	120.18 (12)	C8—C10—H10A	109.9 (8)
C2—C1—C6	119.91 (12)	C11—C10—H10A	109.2 (8)
C3—C2—C1	119.60 (14)	C8—C10—H10B	109.1 (9)
C3—C2—H2	121.2 (9)	C11—C10—H10B	108.7 (9)
C1—C2—H2	119.2 (9)	H10A—C10—H10B	104.5 (12)
C2—C3—C4	120.37 (15)	O1—C11—C12	106.24 (10)
C2—C3—H3	119.6 (10)	O1—C11—C13	111.65 (11)
C4—C3—H3	120.1 (10)	C12—C11—C13	107.16 (10)
C5—C4—C3	121.28 (14)	O1—C11—C10	112.08 (10)
C5—C4—H4	121.9 (11)	C12—C11—C10	109.40 (12)
C3—C4—H4	116.9 (11)	C13—C11—C10	110.10 (10)
C4—C5—C6	119.68 (14)	C11—C12—H12A	108.0 (9)
C4—C5—H5	122.8 (10)	C11—C12—H12B	110.9 (9)
C6—C5—H5	117.5 (10)	H12A—C12—H12B	108.8 (13)
N2—C6—C5	119.54 (12)	C11—C12—H12C	110.4 (10)
N2—C6—C1	121.30 (11)	H12A—C12—H12C	111.5 (13)
C5—C6—C1	119.16 (13)	H12B—C12—H12C	107.3 (14)
N2—C7—C8	121.49 (12)	O2—C13—C14	122.42 (12)
N2—C7—C9	117.81 (12)	O2—C13—C11	119.26 (12)
C8—C7—C9	120.70 (11)	C14—C13—C11	118.19 (11)
N1—C8—C7	121.65 (11)	C13—C14—H14A	109.6 (10)
N1—C8—C10	118.14 (11)	C13—C14—H14B	108.5 (11)
C7—C8—C10	120.21 (12)	H14A—C14—H14B	106.9 (15)
C7—C9—H9A	110.9 (9)	C13—C14—H14C	111.8 (11)
C7—C9—H9B	109.4 (11)	H14A—C14—H14C	108.4 (14)
H9A—C9—H9B	110.4 (15)	H14B—C14—H14C	111.4 (15)
C8—N1—C1—C2	−178.03 (11)	C1—N1—C8—C7	−0.80 (17)
C8—N1—C1—C6	0.77 (18)	C1—N1—C8—C10	178.89 (11)
N1—C1—C2—C3	178.49 (12)	N2—C7—C8—N1	0.30 (19)
C6—C1—C2—C3	−0.3 (2)	C9—C7—C8—N1	−179.65 (11)
C1—C2—C3—C4	0.7 (2)	N2—C7—C8—C10	−179.39 (11)
C2—C3—C4—C5	−0.1 (2)	C9—C7—C8—C10	0.66 (18)
C3—C4—C5—C6	−0.8 (2)	N1—C8—C10—C11	6.54 (17)
C7—N2—C6—C5	178.86 (12)	C7—C8—C10—C11	−173.76 (11)
C7—N2—C6—C1	−0.28 (18)	C8—C10—C11—O1	−61.42 (14)

C4—C5—C6—N2	−178.03 (12)	C8—C10—C11—C12	−178.98 (10)
C4—C5—C6—C1	1.1 (2)	C8—C10—C11—C13	63.50 (14)
N1—C1—C6—N2	−0.23 (19)	O1—C11—C13—O2	165.51 (11)
C2—C1—C6—N2	178.57 (12)	C12—C11—C13—O2	−78.55 (15)
N1—C1—C6—C5	−179.38 (12)	C10—C11—C13—O2	40.34 (16)
C2—C1—C6—C5	−0.58 (19)	O1—C11—C13—C14	−18.62 (16)
C6—N2—C7—C8	0.26 (18)	C12—C11—C13—C14	97.32 (14)
C6—N2—C7—C9	−179.79 (11)	C10—C11—C13—C14	−143.78 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H10···O2^{i}	0.878 (15)	2.440 (16)	3.0967 (14)	132.0 (13)
O1—H10···N1	0.878 (15)	2.170 (16)	2.8082 (14)	129.2 (14)
C2—H2···O2^{i}	0.993 (16)	2.436 (15)	3.2898 (18)	143.7 (12)
C5—H5···N2^{ii}	0.991 (17)	2.619 (17)	3.5676 (18)	160.3 (12)
C12—H12B···O1^{iii}	0.963 (16)	2.513 (16)	3.4373 (19)	160.8 (12)

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

Fig. 1

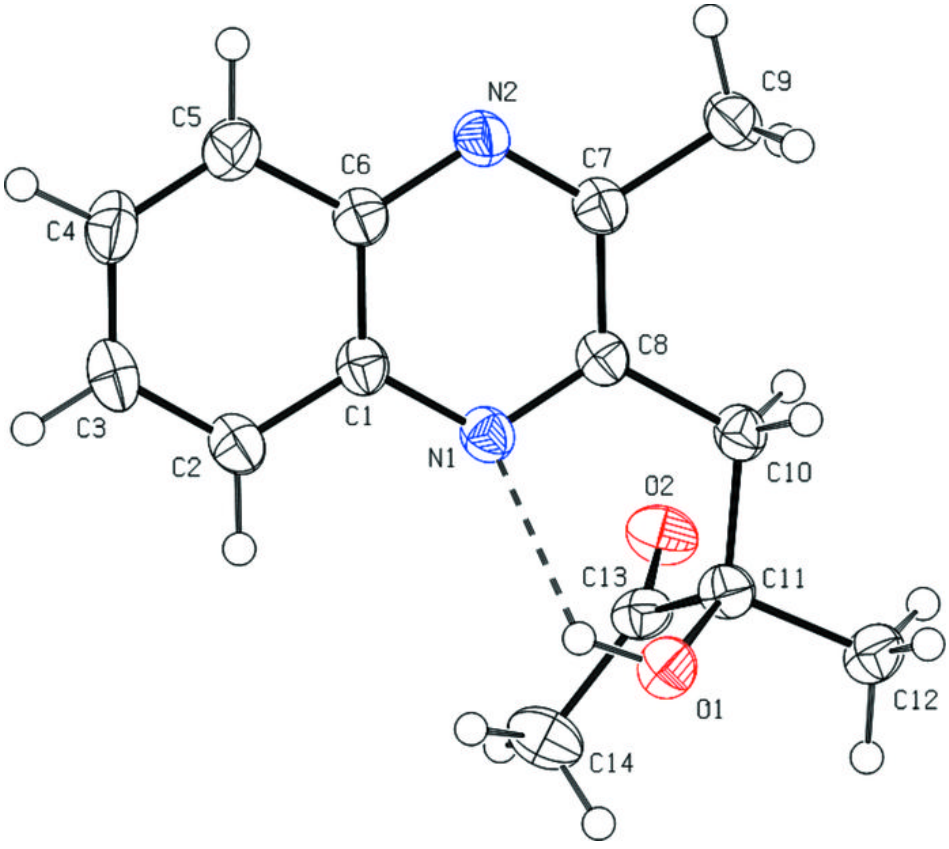


Fig. 2

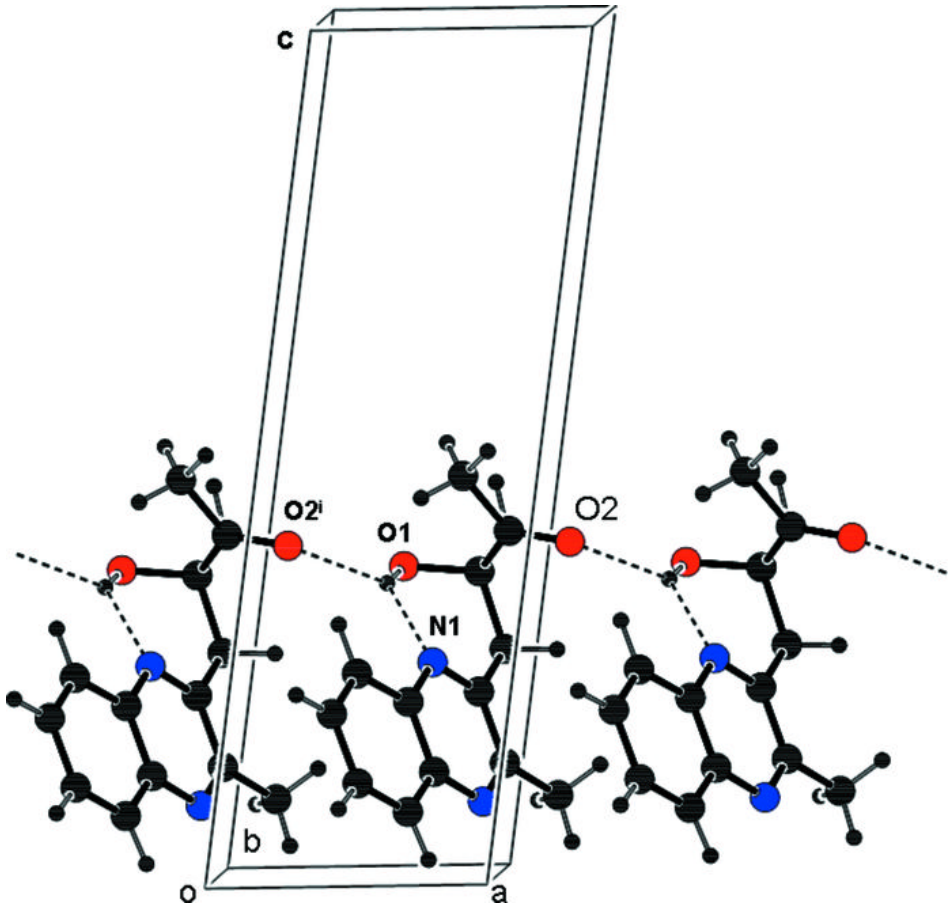


Fig. 3

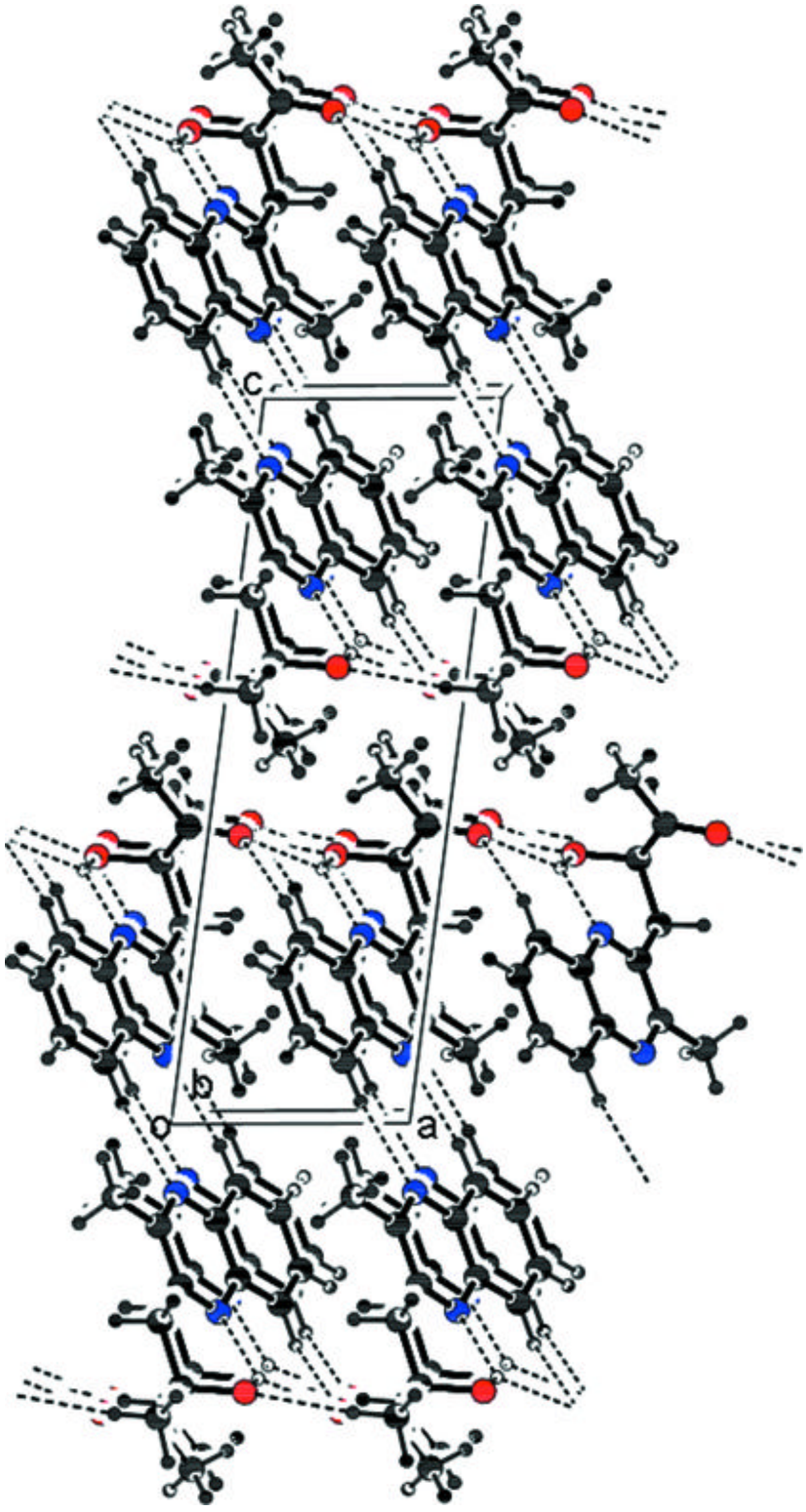
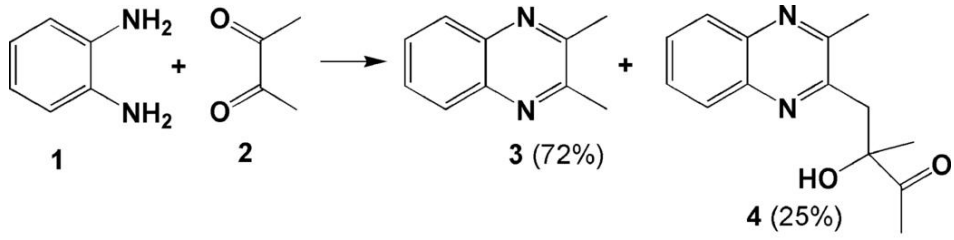


Fig. 4



1 **2** **3** (72%) **4** (25%)

3-hydroxy-3-methyl-4-(3-methylquinoxalin-2-yl)butan-2-one