Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(Isopropylidenehydrazono)-3-methyl-2,3-dihydroquinazolin-4(1*H*)-one

Ahmad Kamali,[a] Iran Sheikhshoaie,[a]* Mahboubeh A. Sharif[b] and Hamid Reza Khavasi[c]

[a]Department of Chemistry, Faculty of Science, Shahid-Bahonar University of Kerman, Kerman, Iran, [b]Department of Chemistry, Islamic Azad University, Qom Branch, Qom, Iran, and [c]Department of Chemistry, Shahid Beheshti University, Tehran, Iran
Correspondence e-mail: sheikhshoaie_i@yahoo.com

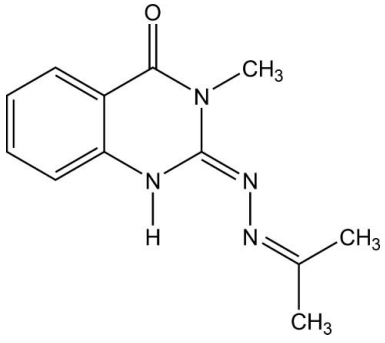
Received 16 June 2007; accepted 22 July 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.061; wR factor = 0.178; data-to-parameter ratio = 15.6.

In the title compound, $C_{12}H_{14}N_4O$, intermolecular N—H···N hydrogen bonds link the molecules into pairs around twofold rotation axes. C—H···O hydrogen bonds and π–π stacking interactions with a distance of 3.5555 (15) Å are observed between the pairs.

Related literature

For related literature, see: Djinović *et al.* (1990); Rewcastle *et al.* (1995); Seijas *et al.* (2000); Rajnikant *et al.* (2001); Tulyasheva *et al.* (2005); Naveen *et al.* (2006).



Experimental

Crystal data

$C_{12}H_{14}N_4O$
M_r = 230.27
Monoclinic, $C2/c$
a = 17.042 (5) Å
b = 7.6443 (19) Å
c = 18.228 (5) Å
β = 93.36 (2)°
V = 2370.5 (11) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.50 × 0.35 × 0.25 mm

Data collection

Stoe IPDSII diffractometer
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 2005)
T_{min} = 0.961, T_{max} = 0.985
5052 measured reflections
2474 independent reflections
1991 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.178
S = 1.07
2474 reflections
159 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···N1^{i}	0.89 (3)	2.11 (4)	2.968 (3)	164 (4)
C5—H5c···O1^{ii}	0.96	2.61	3.345 (3)	134

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

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-AREA*; 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).

We are grateful to Kerman University for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2203).

References

Djinović, K., Golič, L. & Leban, I. (1990). *Acta Cryst.* C**46**, 281–286.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Naveen, S., Bhattacharjee, R., Gayathri, V., Anandalwar, S. M. & Prasad, J. S. (2006). *Anal. Sci.* **22**, x243–x244.
Rajnikant, Gupta, V. K., Deshmukh, M. B., Varghese, B. & Dinesh (2001). *Cryst. Res. Technol.* **36**, 1451–1456.
Rewcastle, G. W., Denny, W. A., Bridges, A. J., Zhou, H. R., Cody, D. R., McMichael, A. & Fry, D. W. (1995). *J. Med. Chem.* **38**, 3482–3487.
Seijas, J. A., Vasquez-Tato, M. P. & Martinez, M. M. (2000). *Tetrahedron Lett.* **41**, 2215–2217.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Stoe & Cie (2005). *X-AREA* (Version 1.31) and *X-SHAPE* (Version 2.05). Stoe & Cie, Darmstadt, Germany.
Tulyasheva, M., Rasulev, B. F., Tojiboev, A. G., Turgunov, K. K., Tashkhodjaev, B., Abdullaev, N. D. & Shakhidoyatov, K. M. (2005). *Molecules* **10**, 1209–1217.

  doi:10.1107/S1600536807035714

supplementary materials

Acta Cryst. (2007). E**63**, o3612 [**doi:10.1107/S1600536807035714**]

2-(Isopropylidenehydrazono)-3-methyl-2,3-dihydroquinazolin-4(1*H*)-one

A. Kamali, I. Sheikhshoaie, M. A. Sharif and H. R. Khavasi

Comment

N-heterocycles are of considerable importance in view of their presence in several biological systems. Quinazoline and its derivatives have a wide range of biological activity, and are potential drugs for various disorders. They have been found to be anti-cancer, anti-inflammatory and anti-hypertensive agents (Seijas *et al.*, 2000; Rewcastle *et al.,* 1995). In this direction, many multidentate N-heterocycles and their complexes have been prepared and characterized.

The molecular structure and unit-cell contents of the title compound are shown in Figures 1 and 2, respectively. The bond lengths and angles agree well with values reported in the literature for some analogous structures containing quinazoline or pyrimidine rings (Djinović *et al.*, 1990). Both the benzene and pyrimidine rings of the quinazoline group adopt almost planar conformations. The average value of the endocyclic bond angles for the pyrimidine ring and benzene ring are very close to 120°.

Intermolecular N—H···N hydrogen bonds (Table 1) link the molecules into pairs around 2-fold rotation axes. Molecules in adjacent pairs adopt centrosymmetric orientations with π–π stacking interactions between symmetry-related benzene and pyrimidine rings (Figure 3). The centroid-centroid distance between the N3/C4/N4/C12/C7/C8 and C7/C8/C9/C10/C11/C12 rings is 3.5555 (15) Å [symmetry code: −*x*, −*y*, 1 − *z*]. C—H···O interactions are also observed between molecules (Table 1).

Experimental

2-Aminobenzoic acid (2.74 g, 20 mmol) and methyl iso-thiocyanomethane (1.46 g, 20 mmol) were refluxed for 2 h in 50 ml acetic acid, yielding a pale yellow precipitate, which was filtered and dried. The resulting powder (1.92 g) was dissolved in 50 ml butanol, and hydrazine (2.5 ml) was added. The mixture was refluxed for 4–5 h, and the title compound was obtained as a white powder. After filtration, the powder was dissolved in acetone (30 ml) and colourless crystals were obtained after a few days standing at room temperature.

Refinement

H atoms bound to C atoms were placed geometrically and allowed to ride during refinement with C—H = 0.93 or 0.96 Å and with U_{iso}(H) = 1.2 or 1.5U_{eq}(C). Atom H4 was located in a difference Fourier map and refined isotropically without restraint.

Figures

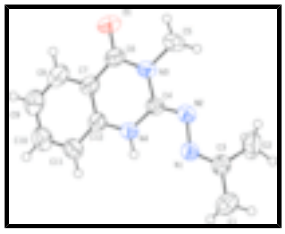


Fig. 1. The molecular structure of the title compound, showing displacement ellipsoids at the 50% probability level for non-H atoms.

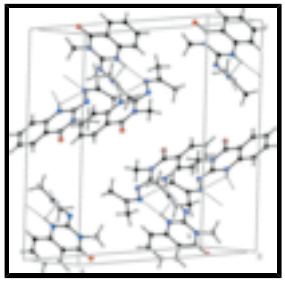


Fig. 2. Unit-cell contents of the title compound. Hydrogen bonds are shown by dashed lines.

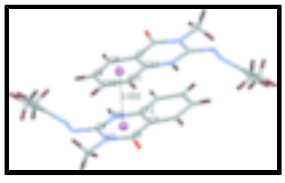


Fig. 3. A view of the π–π stacking interactions between two parallel rings. The centroid-centroid distance between the N3/C4/N4/C12/C7/C8 and C7/C8/C9/C10/C11/C12 rings is 3.5555 (15) Å [symmetry code: −*x*, −*y*, 1 − *z*).

2-(Isopropylidenehydrazono)-3-methyl-2,3-dihydroquinazoline-4(1*H*)-one

Crystal data

$C_{12}H_{14}N_4O$	F_{000} = 976
M_r = 230.27	D_x = 1.290 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 2000 reflections
a = 17.042 (5) Å	θ = 2.2–26.7º
b = 7.6443 (19) Å	μ = 0.09 mm^{-1}
c = 18.228 (5) Å	*T* = 293 (2) K
β = 93.36 (2)º	Block, colourless
V = 2370.5 (11) Å^3	0.50 × 0.35 × 0.25 mm
Z = 8	

Data collection

Stoe IPDSII diffractometer	2474 independent reflections
Radiation source: fine-focus sealed tube	1991 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.030
T = 293(2) K	θ_{max} = 26.7º
rotation method scans	θ_{min} = 2.2º

Absorption correction: numerical (X-SHAPE; Stoe & Cie, 2005)

T_{min} = 0.961, T_{max} = 0.985

5052 measured reflections

h = −21→18

k = −9→8

l = −23→23

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.061

$wR(F^2)$ = 0.178

S = 1.07

2474 reflections

159 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0919P)^2 + 1.3884P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.029

$\Delta\rho_{max}$ = 0.29 e Å^{-3}

$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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}
C1	0.57814 (17)	0.4861 (4)	0.18688 (15)	0.0808 (8)
H1A	0.5239	0.4999	0.1974	0.097*
H1B	0.5815	0.4272	0.1407	0.097*
H1C	0.6024	0.5991	0.1844	0.097*
C2	0.68686 (19)	0.4623 (4)	0.28983 (16)	0.0855 (9)
H2A	0.7334	0.3937	0.2847	0.103*
H2B	0.6752	0.4672	0.3407	0.103*
H2C	0.6952	0.5786	0.2720	0.103*
C3	0.61960 (12)	0.3805 (3)	0.24639 (11)	0.0533 (5)
C4	0.59540 (11)	0.0259 (2)	0.34711 (10)	0.0434 (4)
C5	0.71897 (12)	−0.0990 (3)	0.39858 (15)	0.0704 (7)
H5A	0.7395	0.0170	0.4059	0.084*
H5B	0.7362	−0.1454	0.3533	0.084*

H5C	0.7376	−0.1726	0.4386	0.084*
C6	0.59394 (13)	−0.2015 (3)	0.44262 (12)	0.0555 (5)
C7	0.50876 (12)	−0.1824 (2)	0.44147 (10)	0.0480 (5)
C8	0.46452 (15)	−0.2768 (3)	0.49027 (11)	0.0598 (6)
H8	0.4897	−0.3527	0.5239	0.072*
C9	0.38448 (16)	−0.2584 (3)	0.48906 (13)	0.0673 (6)
H9	0.3552	−0.3216	0.5214	0.081*
C10	0.34776 (14)	−0.1443 (4)	0.43892 (13)	0.0672 (6)
H10	0.2934	−0.1316	0.4381	0.081*
C11	0.38959 (12)	−0.0496 (3)	0.39044 (11)	0.0554 (5)
H11	0.3638	0.0260	0.3571	0.066*
C12	0.47093 (11)	−0.0679 (2)	0.39171 (9)	0.0443 (4)
N1	0.59441 (9)	0.2260 (2)	0.25684 (8)	0.0483 (4)
N2	0.63887 (9)	0.1261 (2)	0.30930 (9)	0.0488 (4)
N3	0.63284 (9)	−0.0931 (2)	0.39540 (9)	0.0514 (4)
N4	0.51535 (9)	0.0280 (2)	0.34470 (8)	0.0451 (4)
H4	0.4908 (15)	0.101 (3)	0.3133 (14)	0.063 (7)*
O1	0.63060 (11)	−0.3067 (2)	0.48180 (12)	0.0832 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0830 (17)	0.0804 (17)	0.0788 (16)	−0.0074 (14)	0.0020 (13)	0.0358 (14)
C2	0.0961 (19)	0.0788 (17)	0.0796 (16)	−0.0426 (15)	−0.0116 (14)	0.0101 (14)
C3	0.0534 (11)	0.0584 (12)	0.0481 (10)	−0.0081 (9)	0.0041 (8)	0.0063 (9)
C4	0.0438 (9)	0.0413 (9)	0.0440 (9)	0.0009 (7)	−0.0077 (7)	−0.0020 (7)
C5	0.0480 (11)	0.0715 (15)	0.0892 (17)	0.0045 (11)	−0.0162 (11)	0.0151 (13)
C6	0.0647 (13)	0.0433 (10)	0.0561 (11)	−0.0018 (9)	−0.0165 (9)	0.0062 (9)
C7	0.0616 (12)	0.0397 (9)	0.0418 (9)	−0.0042 (8)	−0.0056 (8)	−0.0021 (7)
C8	0.0887 (17)	0.0448 (11)	0.0452 (10)	−0.0083 (10)	−0.0018 (10)	0.0017 (8)
C9	0.0794 (16)	0.0671 (14)	0.0565 (12)	−0.0175 (12)	0.0148 (11)	0.0014 (11)
C10	0.0574 (12)	0.0794 (16)	0.0656 (13)	−0.0091 (12)	0.0103 (10)	0.0001 (12)
C11	0.0489 (11)	0.0629 (12)	0.0539 (11)	−0.0031 (9)	−0.0008 (9)	0.0018 (9)
C12	0.0516 (10)	0.0413 (9)	0.0394 (8)	−0.0058 (8)	−0.0035 (7)	−0.0038 (7)
N1	0.0455 (8)	0.0533 (9)	0.0452 (8)	−0.0054 (7)	−0.0049 (6)	0.0064 (7)
N2	0.0412 (8)	0.0532 (9)	0.0511 (9)	−0.0027 (7)	−0.0062 (6)	0.0053 (7)
N3	0.0453 (9)	0.0494 (9)	0.0576 (9)	0.0033 (7)	−0.0126 (7)	0.0072 (7)
N4	0.0403 (8)	0.0495 (9)	0.0445 (8)	−0.0010 (7)	−0.0073 (6)	0.0087 (7)
O1	0.0779 (11)	0.0676 (11)	0.1006 (13)	0.0041 (9)	−0.0245 (10)	0.0375 (10)

Geometric parameters (Å, °)

C1—C3	1.496 (3)	C6—O1	1.223 (2)
C1—H1A	0.960	C6—N3	1.391 (3)
C1—H1B	0.960	C6—C7	1.458 (3)
C1—H1C	0.960	C7—C12	1.391 (3)
C2—C3	1.492 (3)	C7—C8	1.399 (3)
C2—H2A	0.960	C8—C9	1.370 (4)
C2—H2B	0.960	C8—H8	0.930

C2—H2C	0.960	C9—C10	1.386 (4)
C3—N1	1.275 (3)	C9—H9	0.930
C4—N2	1.292 (2)	C10—C11	1.373 (3)
C4—N4	1.362 (2)	C10—H10	0.930
C4—N3	1.394 (2)	C11—C12	1.392 (3)
C5—N3	1.466 (3)	C11—H11	0.930
C5—H5A	0.960	C12—N4	1.386 (2)
C5—H5B	0.960	N1—N2	1.410 (2)
C5—H5C	0.960	N4—H4	0.89 (3)
C3—C1—H1A	109.5	C12—C7—C8	119.5 (2)
C3—C1—H1B	109.5	C12—C7—C6	119.69 (18)
H1A—C1—H1B	109.5	C8—C7—C6	120.83 (18)
C3—C1—H1C	109.5	C9—C8—C7	120.6 (2)
H1A—C1—H1C	109.5	C9—C8—H8	119.7
H1B—C1—H1C	109.5	C7—C8—H8	119.7
C3—C2—H2A	109.5	C8—C9—C10	119.1 (2)
C3—C2—H2B	109.5	C8—C9—H9	120.5
H2A—C2—H2B	109.5	C10—C9—H9	120.5
C3—C2—H2C	109.5	C11—C10—C9	121.6 (2)
H2A—C2—H2C	109.5	C11—C10—H10	119.2
H2B—C2—H2C	109.5	C9—C10—H10	119.2
N1—C3—C2	124.3 (2)	C10—C11—C12	119.3 (2)
N1—C3—C1	117.4 (2)	C10—C11—H11	120.3
C2—C3—C1	118.3 (2)	C12—C11—H11	120.3
N2—C4—N4	125.47 (17)	N4—C12—C7	119.13 (17)
N2—C4—N3	117.90 (16)	N4—C12—C11	121.06 (17)
N4—C4—N3	116.62 (16)	C7—C12—C11	119.81 (18)
N3—C5—H5A	109.5	C3—N1—N2	115.55 (16)
N3—C5—H5B	109.5	C4—N2—N1	112.38 (15)
H5A—C5—H5B	109.5	C6—N3—C4	124.23 (17)
N3—C5—H5C	109.5	C6—N3—C5	118.01 (17)
H5A—C5—H5C	109.5	C4—N3—C5	117.71 (17)
H5B—C5—H5C	109.5	C4—N4—C12	123.79 (16)
O1—C6—N3	120.5 (2)	C4—N4—H4	117.5 (16)
O1—C6—C7	123.3 (2)	C12—N4—H4	118.5 (16)
N3—C6—C7	116.22 (17)		
O1—C6—C7—C12	−175.7 (2)	C1—C3—N1—N2	175.12 (19)
N3—C6—C7—C12	3.7 (3)	N4—C4—N2—N1	−7.7 (3)
O1—C6—C7—C8	4.9 (3)	N3—C4—N2—N1	173.30 (15)
N3—C6—C7—C8	−175.71 (17)	C3—N1—N2—C4	143.76 (18)
C12—C7—C8—C9	0.5 (3)	O1—C6—N3—C4	177.7 (2)
C6—C7—C8—C9	179.9 (2)	C7—C6—N3—C4	−1.7 (3)
C7—C8—C9—C10	−0.2 (3)	O1—C6—N3—C5	−4.9 (3)
C8—C9—C10—C11	0.1 (4)	C7—C6—N3—C5	175.70 (19)
C9—C10—C11—C12	−0.2 (3)	N2—C4—N3—C6	176.04 (17)
C8—C7—C12—N4	178.54 (16)	N4—C4—N3—C6	−3.1 (3)
C6—C7—C12—N4	−0.9 (3)	N2—C4—N3—C5	−1.4 (3)
C8—C7—C12—C11	−0.6 (3)	N4—C4—N3—C5	179.48 (18)

C6—C7—C12—C11	179.92 (18)	N2—C4—N4—C12	−172.75 (17)
C10—C11—C12—N4	−178.67 (19)	N3—C4—N4—C12	6.3 (3)
C10—C11—C12—C7	0.5 (3)	C7—C12—N4—C4	−4.4 (3)
C2—C3—N1—N2	−5.4 (3)	C11—C12—N4—C4	174.78 (18)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···N1^{i}	0.89 (3)	2.11 (4)	2.968 (3)	164 (4)
C5—H5c···O1^{ii}	0.96	2.61	3.345 (3)	134

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

Fig. 1

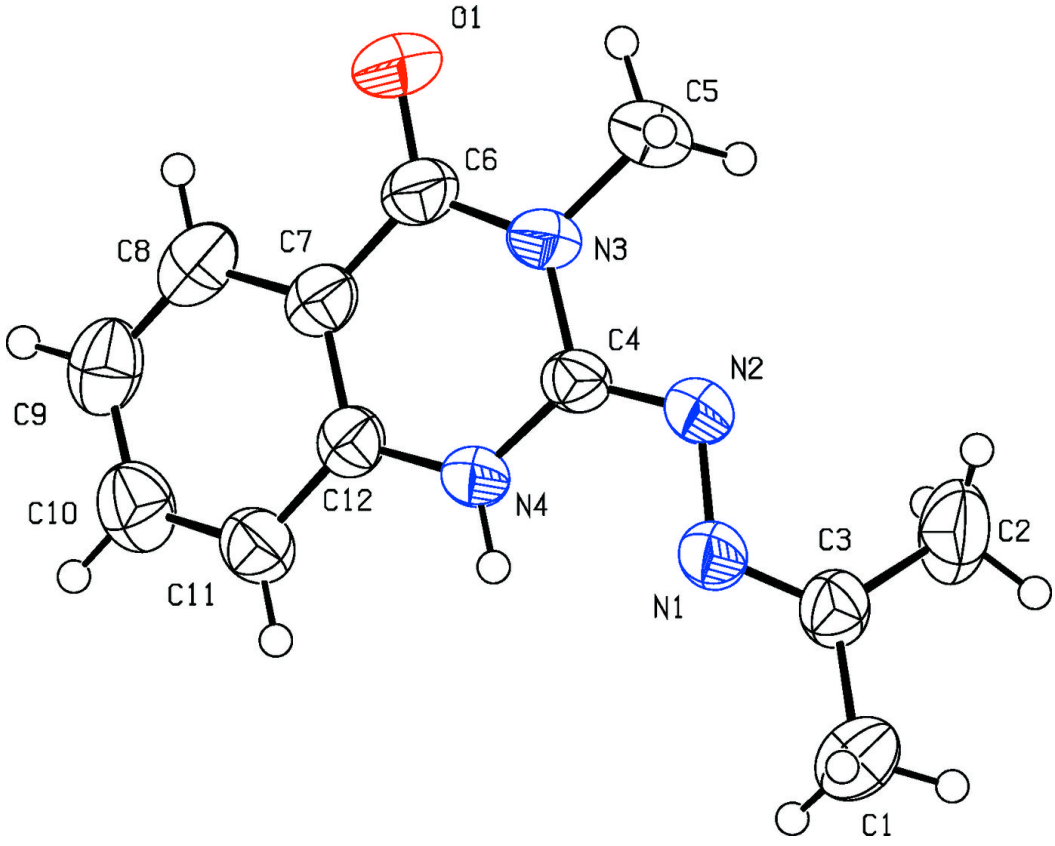


Fig. 2

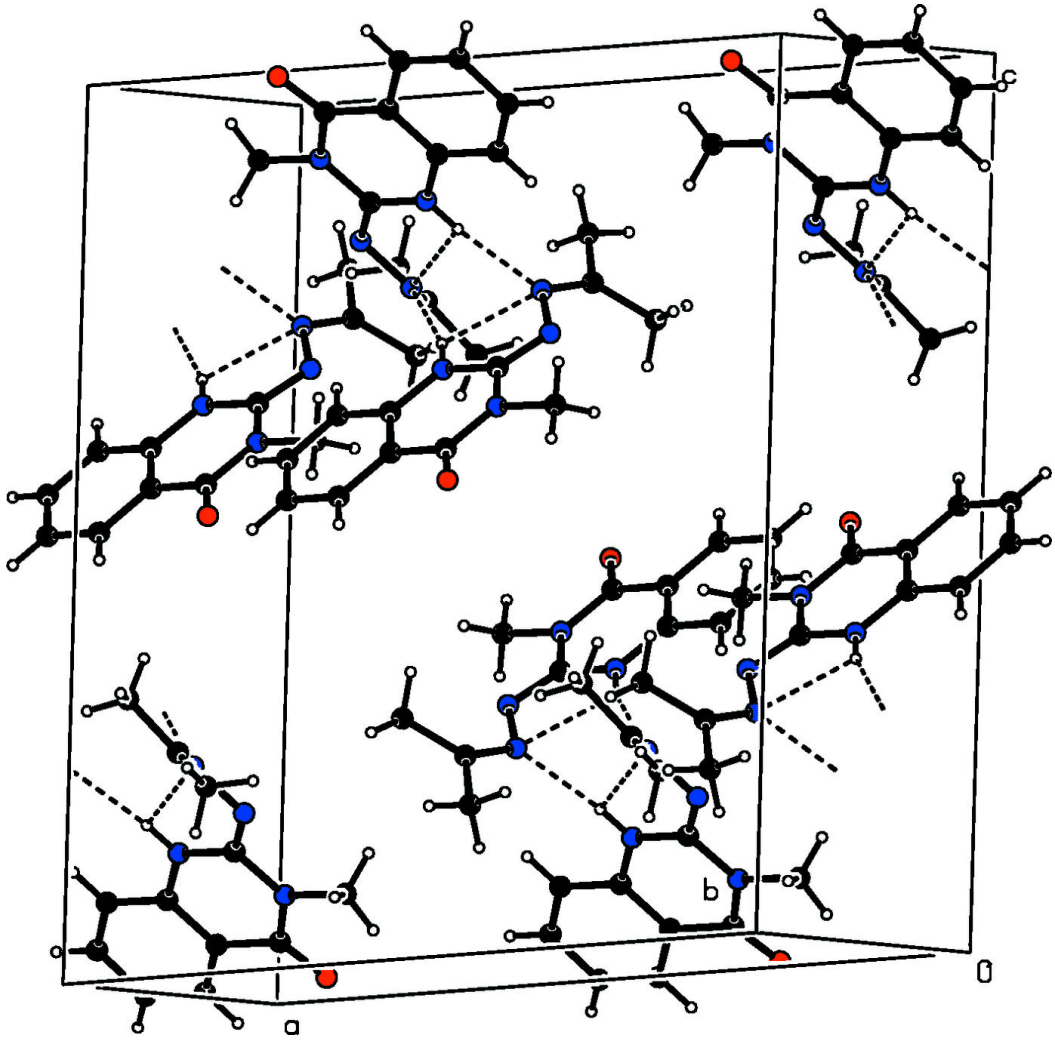


Fig. 3