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(1*Z*)-Phthalazin-1(2*H*)-one isopropylidenehydrazone

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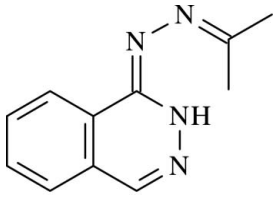
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.123; data-to-parameter ratio = 12.9.

The crystal structure of the title compound, $C_{11}H_{12}N_4$, is stabilized by an N—H···N and three C—H···N intermolecular hydrogen bonds. The N—H···N hydrogen bonds generate centrosymmetric $R_2^2(6)$ rings, while the three C—H···N hydrogen bonds forms edge-fused $R_2^2(7)R_2^2(7)R_2^2(10)$ ring motifs. Except for four H atoms of the methyl groups, all atoms are in the same plane and the dihedral angles between the aromatic and heterocyclic rings and the substituent group plane are 1.87 (5) and 1.53 (5)°, respectively.

Related literature

For related structures, see: Zheng *et al.* (2005*a*,*b*); Duan *et al.* (2005); Xu & Hu, (2007); Sarojini, Narayana *et al.* (2007); Sarojini, Yathirajan *et al.* (2007); Rollas *et al.* (2002); Küçükgüzel *et al.* (1999); Kundu *et al.* (2005); Kesslen & Euler (1999); Armstrong *et al.* (1998); Xu *et al.* (1997). For related literature, see: Etter (1990).



Experimental

Crystal data

$C_{11}H_{12}N_4$
M_r = 200.25
Triclinic, $P\overline{1}$
a = 7.176 (4) Å
b = 7.789 (4) Å
c = 9.800 (4) Å
α = 79.74 (4)°
β = 84.40 (4)°
γ = 83.53 (4)°
V = 533.9 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 296 K
0.78 × 0.55 × 0.19 mm

Data collection

Stoe IPDS 2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.346, T_{max} = 0.937
9440 measured reflections
2095 independent reflections
1679 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.123
S = 1.05
2095 reflections
163 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 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}	0.870 (17)	2.322 (17)	3.012 (2)	136.4 (13)
C11—H11B···N4^{ii}	0.96	2.80	3.585 (3)	139
C10—H10C···N3^{ii}	0.96	2.79	3.685 (3)	156
C11—H11C···N4^{iii}	0.96	2.88	3.711 (3)	145

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); 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: AT2397).

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supplementary materials

Acta Cryst. (2007). E**63**, o4084-o4085 [**doi:10.1107/S1600536807044807**]

(1*Z*)-Phthalazin-1(2*H*)-one isopropylidenehydrazone

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Comment

Hydrazones and related compounds are known to possess antimicrobial, anticonvulsant, analgesic, anti-inflammatory, anti-platelet, antitubercular and antitumoral activities. For example, isonicotinoyl hydrazones are antitubercular; 4-hydroxybenzoic acid [(5-nitro-2-furyl) methylene]-hydrazide (nifuroxazide) is an intestinal antiseptic; 4-fluorobenzoic acid [(5-nitro-2-furyl)methylene]-hydrazide (Rollas *et al.*, 2002) and 2,3,4-pentanetrione-3-[4-[[(5-nitro-2-furyl) methylene] hydrazino] carbonyl] phenyl]-hydrazone (Küçükgüzel *et al.*, 1999) have antibacterial activity. A number of azine compounds containing both a diimine linkage and N—N bond have been investigated in terms of their crystallography and coordination chemistry (Kundu *et al.*, 2005; Kesslen & Euler, 1999; Armstrong *et al.* 1998; Xu *et al.*, 1997). The crystal structures of *N*,*N*-bis(4-chlorobenzylidene) hydrazine (Zheng *et al.*, 2005*a*), *N*,*N*'-bis(3-nitrobenzylidene)hydrazine (Zheng *et al.* 2005*b*), *N*,*N*'-bis(3-hydroxy-4-methoxybenzylidene)hydrazine (Duan *et al.*, 2005), 1,2-bis[4-(trifluoromethyl)benzylidene]hydrazine (Xu & Hu, 2007), isopropylidene-6-methoxy-2-naphthohydrazide (Sarojini, Narayana *et al.*, 2007), 2-bromo-*N*'-isopropylidene-5- methoxybenzohydrazide (Sarojini, Yathirajan *et al.*, 2007) have been reported. A new hydrazone, $C_{11}H_{12}N_4$, (I) was synthesized and its crystal structure is reported.

The crystal packing is stabilized by an N—H···N and three C—H···N intermolecular hydrogen bonds. These hydrogen bonds generate centrosymmetric $R_2^2(6)$ and edge-fussed $R_2^2(7)R_2^2(7)R_2^2(10)$ ring motifs (Fig. 2) (Etter, 1990). Except the four protons of methyl groups, all atoms are in the same plane and the dihedral angles between the aromatic ring, heterocyclic ring and substituted group plane are 1.87 (5) ° and 1.53 (5) °, respectively. There are no C—H···π and π···π interactions in crystal packing.

Experimental

A mixture of (1*Z*)-phthalazin-1(2*H*)-one hydrazone (3.20 g, 0.02 mol) and acetone (1.3 ml, 0.02 mol) in 15 ml of absolute ethanol containing 2 drops of sulfuric acid was refluxed for about 3 h. On cooling, the solid separated was filtered and recrystallized from acetone by slow evaporation [m.p.: 387–389 K]. Analysis for $C_{11}H_{12}N_4$: Found (Calculated): C 65.89 (65.98), H 5.98 (6.04), N 27.91% (27.98%).

Refinement

All C-bound H atoms except the methyl group and N-bound H atom were located in Fourier difference map and refined freely. The methyl group H atoms were refined using the riding model approximation with d(C—H) = 0.96 and U_{iso}(H) = 1.5U_{eq}(parent atom).

Figures

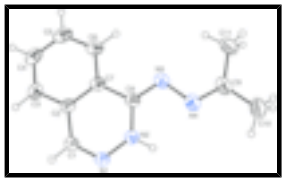

Fig. 1. A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

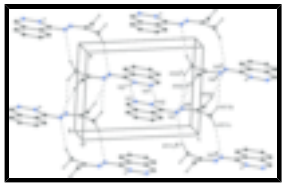

Fig. 2. Part of the crystal structure of (I), showing the formation of $R_2^2(6)$ and $R_2^2(7)R_2^2(7)R_2^2(10)$ motifs. [Symmetry codes: (i) $1-x, -y, 1-z$ (ii) $1-x, -y, z+1$ (iii) $-x, -y, z+1$].

Fig. 3. Reaction scheme.

(1*Z*)-Phthalazin-1(2*H*)-one isopropylidenehydrazone

Crystal data

$C_{11}H_{12}N_4$	$Z = 2$
$M_r = 200.25$	$F_{000} = 212$
Triclinic, $P\overline{1}$	$D_x = 1.246$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.176$ (4) Å	Cell parameters from 9440 reflections
$b = 7.789$ (4) Å	$\theta = 2.7$–27.8º
$c = 9.800$ (4) Å	$\mu = 0.08$ mm^{-1}
$\alpha = 79.74$ (4)º	$T = 296$ K
$\beta = 84.40$ (4)º	Prism, yellow
$\gamma = 83.53$ (4)º	0.78 × 0.55 × 0.19 mm
$V = 533.9$ (5) Å^3	

Data collection

Stoe IPDS 2 diffractometer	2095 independent reflections
Monochromator: plane graphite	1679 reflections with $I > 2\sigma(I)$
Detector resolution: 6.67 pixels mm^{-1}	$R_{int} = 0.061$
$T = 296$ K	$\theta_{max} = 26.0$º
ω scans	$\theta_{min} = 2.7$º
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	$h = -8 \rightarrow 8$
$T_{min} = 0.346$, $T_{max} = 0.937$	$k = -9 \rightarrow 9$
9440 measured reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.123$

$S = 1.05$

2095 reflections

163 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

$w = 1/[\sigma^2(F_o^2) + (0.0662P)^2 + 0.04P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.14$ e Å^{-3}

$\Delta\rho_{min} = -0.11$ e Å^{-3}

Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.046 (9)

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	0.3460 (2)	0.34934 (18)	0.39909 (14)	0.0546 (3)
C2	0.26486 (17)	0.47918 (16)	0.48054 (13)	0.0483 (3)
C3	0.2234 (2)	0.65485 (19)	0.42177 (17)	0.0605 (4)
C4	0.1516 (2)	0.77485 (19)	0.50384 (19)	0.0683 (4)
C5	0.1182 (2)	0.7230 (2)	0.6451 (2)	0.0703 (4)
C6	0.1561 (2)	0.55094 (19)	0.70560 (16)	0.0594 (4)
C7	0.23084 (16)	0.42685 (16)	0.62321 (13)	0.0466 (3)
C8	0.27783 (16)	0.24291 (16)	0.68173 (12)	0.0455 (3)
C9	0.2846 (2)	−0.0619 (2)	0.97379 (14)	0.0614 (4)
C10	0.3412 (3)	−0.2538 (2)	1.01029 (18)	0.0827 (5)
H10A	0.3839	−0.3003	0.9270	0.124*
H10B	0.2353	−0.3119	1.0559	0.124*
H10C	0.4410	−0.2727	1.0714	0.124*
C11	0.2077 (2)	0.0332 (3)	1.08959 (15)	0.0752 (5)
H11A	0.1719	0.1537	1.0525	0.113*

H11B	0.3021	0.0269	1.1538	0.113*
H11C	0.0996	−0.0199	1.1370	0.113*
N1	0.39192 (17)	0.18830 (14)	0.44897 (11)	0.0552 (3)
N2	0.35559 (16)	0.13888 (15)	0.58826 (11)	0.0532 (3)
N3	0.24853 (17)	0.18648 (15)	0.81368 (11)	0.0561 (3)
N4	0.30515 (17)	0.00592 (15)	0.84518 (11)	0.0589 (3)
H1	0.373 (2)	0.381 (2)	0.3020 (17)	0.068 (4)*
H2	0.393 (2)	0.031 (2)	0.6218 (16)	0.065 (4)*
H3	0.254 (3)	0.688 (3)	0.323 (2)	0.088 (6)*
H4	0.125 (3)	0.893 (2)	0.4637 (18)	0.081 (5)*
H5	0.076 (3)	0.808 (3)	0.701 (2)	0.090 (6)*
H6	0.131 (2)	0.509 (2)	0.8036 (17)	0.065 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0641 (8)	0.0528 (7)	0.0437 (7)	0.0047 (6)	0.0006 (5)	−0.0086 (5)
C2	0.0461 (6)	0.0462 (7)	0.0530 (7)	−0.0001 (5)	−0.0048 (5)	−0.0112 (5)
C3	0.0643 (8)	0.0508 (7)	0.0637 (9)	0.0011 (6)	−0.0082 (6)	−0.0053 (6)
C4	0.0710 (9)	0.0448 (8)	0.0892 (11)	0.0039 (6)	−0.0112 (8)	−0.0152 (7)
C5	0.0710 (10)	0.0559 (8)	0.0898 (11)	0.0039 (7)	−0.0035 (8)	−0.0362 (8)
C6	0.0618 (8)	0.0599 (8)	0.0601 (8)	−0.0025 (6)	0.0010 (6)	−0.0249 (6)
C7	0.0412 (6)	0.0487 (7)	0.0520 (7)	−0.0030 (5)	−0.0024 (5)	−0.0154 (5)
C8	0.0422 (6)	0.0500 (7)	0.0453 (6)	−0.0034 (5)	−0.0012 (5)	−0.0130 (5)
C9	0.0602 (8)	0.0741 (9)	0.0485 (7)	−0.0118 (7)	−0.0055 (6)	−0.0029 (6)
C10	0.1079 (14)	0.0743 (11)	0.0616 (9)	−0.0125 (9)	−0.0128 (8)	0.0057 (8)
C11	0.0716 (10)	0.1004 (13)	0.0491 (8)	−0.0029 (8)	0.0031 (7)	−0.0082 (8)
N1	0.0664 (7)	0.0509 (6)	0.0447 (6)	0.0070 (5)	0.0035 (4)	−0.0107 (4)
N2	0.0655 (7)	0.0448 (6)	0.0454 (6)	0.0046 (5)	0.0026 (5)	−0.0070 (5)
N3	0.0642 (7)	0.0584 (7)	0.0446 (6)	−0.0027 (5)	−0.0002 (4)	−0.0102 (5)
N4	0.0695 (7)	0.0584 (7)	0.0468 (6)	−0.0045 (5)	−0.0037 (5)	−0.0050 (5)

Geometric parameters (Å, °)

C1—N1	1.2809 (18)	C8—N3	1.2941 (17)
C1—C2	1.4364 (19)	C8—N2	1.3674 (17)
C1—H1	0.946 (16)	C9—N4	1.2780 (18)
C2—C7	1.3904 (19)	C9—C11	1.492 (2)
C2—C3	1.398 (2)	C9—C10	1.493 (2)
C3—C4	1.366 (2)	C10—H10A	0.9600
C3—H3	0.96 (2)	C10—H10B	0.9600
C4—C5	1.376 (3)	C10—H10C	0.9600
C4—H4	0.943 (18)	C11—H11A	0.9600
C5—C6	1.374 (2)	C11—H11B	0.9600
C5—H5	0.94 (2)	C11—H11C	0.9600
C6—C7	1.3966 (19)	N1—N2	1.3585 (16)
C6—H6	0.964 (16)	N2—H2	0.870 (17)
C7—C8	1.4600 (19)	N3—N4	1.4068 (18)

N1—C1—C2	124.53 (12)	N2—C8—C7	115.36 (11)
N1—C1—H1	115.2 (10)	N4—C9—C11	125.86 (15)
C2—C1—H1	120.2 (10)	N4—C9—C10	116.67 (14)
C7—C2—C3	119.55 (13)	C11—C9—C10	117.47 (13)
C7—C2—C1	118.09 (12)	C9—C10—H10A	109.5
C3—C2—C1	122.34 (13)	C9—C10—H10B	109.5
C4—C3—C2	120.34 (15)	H10A—C10—H10B	109.5
C4—C3—H3	121.8 (11)	C9—C10—H10C	109.5
C2—C3—H3	117.8 (11)	H10A—C10—H10C	109.5
C3—C4—C5	120.06 (14)	H10B—C10—H10C	109.5
C3—C4—H4	120.0 (11)	C9—C11—H11A	109.5
C5—C4—H4	120.0 (11)	C9—C11—H11B	109.5
C6—C5—C4	120.90 (14)	H11A—C11—H11B	109.5
C6—C5—H5	119.8 (11)	C9—C11—H11C	109.5
C4—C5—H5	119.2 (11)	H11A—C11—H11C	109.5
C5—C6—C7	119.73 (15)	H11B—C11—H11C	109.5
C5—C6—H6	123.1 (10)	C1—N1—N2	116.62 (11)
C7—C6—H6	117.1 (10)	N1—N2—C8	127.12 (11)
C2—C7—C6	119.41 (13)	N1—N2—H2	115.9 (10)
C2—C7—C8	118.27 (11)	C8—N2—H2	116.8 (10)
C6—C7—C8	122.31 (12)	C8—N3—N4	110.64 (11)
N3—C8—N2	123.62 (12)	C9—N4—N3	115.41 (13)
N3—C8—C7	121.02 (12)		
N1—C1—C2—C7	−1.0 (2)	C2—C7—C8—N3	−179.70 (11)
N1—C1—C2—C3	177.64 (13)	C6—C7—C8—N3	1.26 (19)
C7—C2—C3—C4	0.6 (2)	C2—C7—C8—N2	1.14 (16)
C1—C2—C3—C4	−178.02 (13)	C6—C7—C8—N2	−177.90 (11)
C2—C3—C4—C5	−0.5 (2)	C2—C1—N1—N2	1.8 (2)
C3—C4—C5—C6	0.0 (3)	C1—N1—N2—C8	−1.1 (2)
C4—C5—C6—C7	0.4 (2)	N3—C8—N2—N1	−179.50 (12)
C3—C2—C7—C6	−0.15 (19)	C7—C8—N2—N1	−0.36 (19)
C1—C2—C7—C6	178.50 (12)	N2—C8—N3—N4	−0.75 (18)
C3—C2—C7—C8	−179.23 (11)	C7—C8—N3—N4	−179.84 (10)
C1—C2—C7—C8	−0.57 (18)	C11—C9—N4—N3	−0.1 (2)
C5—C6—C7—C2	−0.4 (2)	C10—C9—N4—N3	179.27 (13)
C5—C6—C7—C8	178.68 (12)	C8—N3—N4—C9	178.92 (11)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···N1^{i}	0.870 (17)	2.322 (17)	3.012 (2)	136.4 (13)
C11—H11B···N4^{ii}	0.96	2.80	3.585 (3)	139
C10—H10C···N3^{ii}	0.96	2.79	3.685 (3)	156
C11—H11C···N4^{iii}	0.96	2.88	3.711 (3)	145

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

Fig. 1

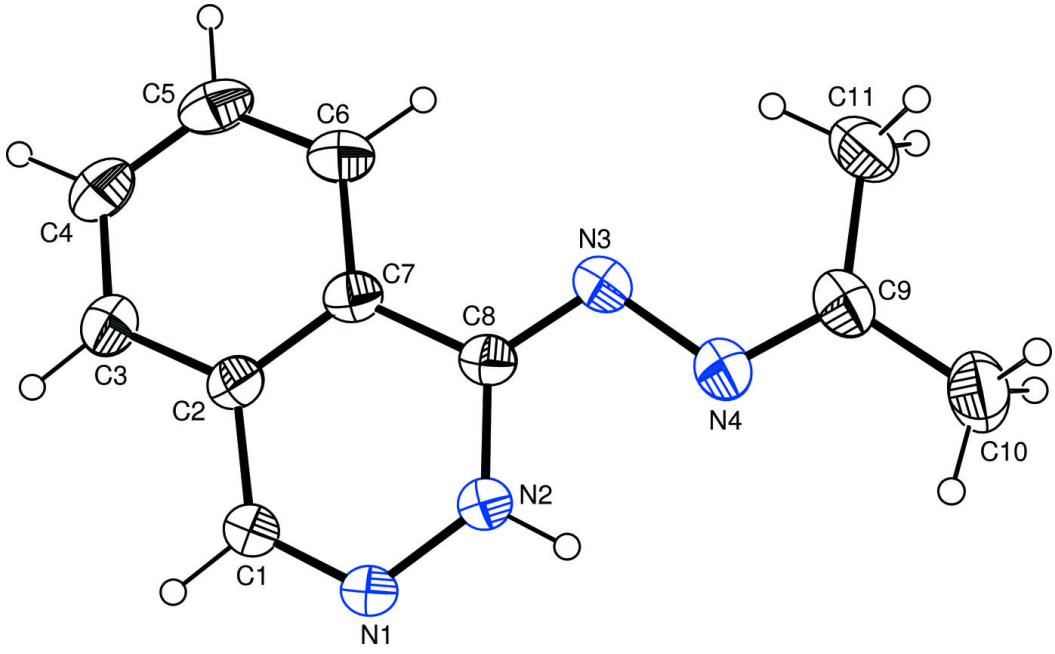

Fig. 2

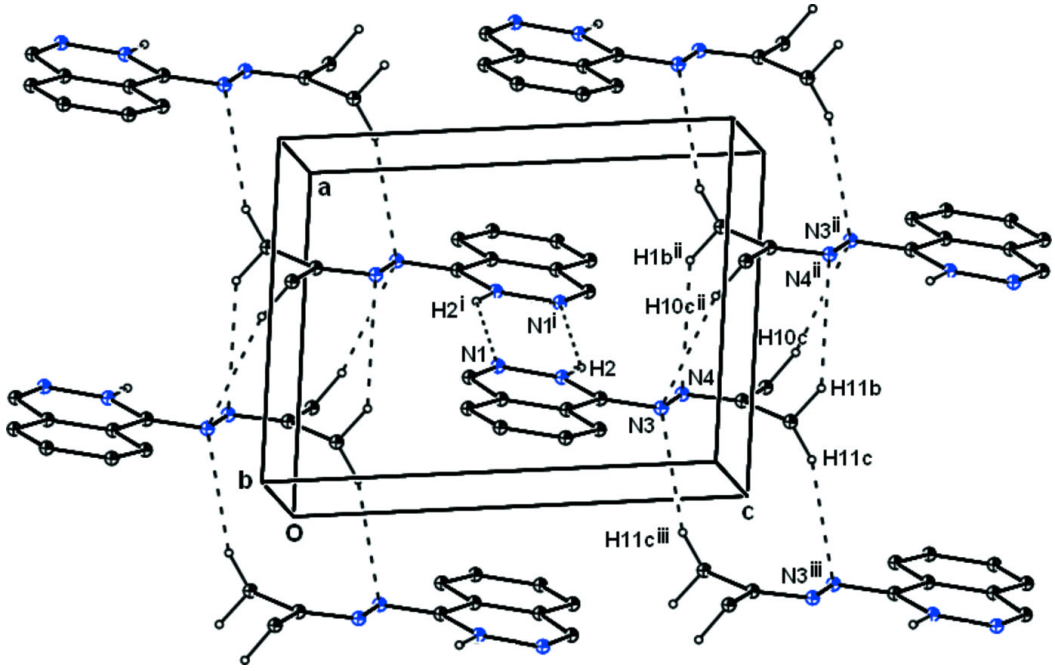
a
b
c
O
H2i
N1i
H1bii
H10cii
N3ii
N4ii
H10c
H11b
H11c
N1
H2
N4
N3
H11ciii
N3iii

Fig. 3