Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Methoxybenzaldehyde (phthalazin-1-ylidene)hydrazone

Ray J. Butcher,[a] Jerry P. Jasinski,[b]* H. S. Yathirajan,[c] A. M. Vijesh[d] and B. Narayana[d]

[a]Department of Chemistry, Howard University, 525 College Street NW, Washington, DC 20059, USA, [b]Department of Chemistry, Keene State College, 229 Main Street, Keene, NH 03435-2001, USA, [c]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, and [d]Department of Studies in Chemistry, Mangalore University, Mangalagangotri 574 199, India
Correspondence e-mail: jjasinski@keene.edu

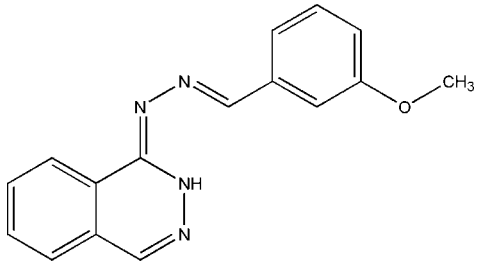
Received 20 July 2007; accepted 28 July 2007

Key indicators: single-crystal X-ray study; T = 203 K; mean σ(C–C) = 0.002 Å; R factor = 0.057; wR factor = 0.180; data-to-parameter ratio = 22.9.

The title compound, $C_{16}H_{14}N_4O$, crystallizes with two independent molecules in the asymmetric unit. The phthalazin-1-yl and 4-methoxybenzaldehyde groups are coplanar with each other in each of the independent molecules with angles between their mean planes of 15.4 (4) and 13.3 (9)°. The crystal packing is stabilized by intermolecular N—H···N hydrogen bonding between inverted phthalazin-1-yl groups allowing the independent molecules to be stacked in groups oblique to the ab plane in a zigzag pattern.

Related literature

For related structures, see: Lynch & McClenaghan (2002*a*,*b*). For related literature, see: El-Masry *et al.* (2000); Pandey *et al.* (1999); Singh & Dash, 1988; Hodnett & Dunn, 1970; Desai *et al.* (2001); Aydogan *et al.* (2001); Taggi *et al.* (2002).



Experimental

Crystal data

$C_{16}H_{14}N_4O$
M_r = 278.31
Monoclinic, $P2_1/c$
a = 12.1351 (8) Å
b = 14.2016 (7) Å
c = 16.0716 (8) Å
β = 90.012 (6)°
V = 2769.7 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 203 K
0.55 × 0.41 × 0.37 mm

Data collection

Oxford Diffraction Gemini R diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
T_{min} = 0.833, T_{max} = 1.000
(expected range = 0.806–0.968)
19111 measured reflections
8720 independent reflections
3864 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
2 standard reflections
every 50 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.180
S = 0.99
8720 reflections
381 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2A—H2AA···N1B	0.87	2.36	3.0451 (19)	136
N2B—H2BA···N1A	0.87	2.36	3.0466 (19)	136

Data collection: *CrysAlisPro* (Oxford Diffraction, 2007); cell refinement: *CrysAlisPro*; data reduction: *CrysAlisPro*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *WinGX* (Farrugia, 1999); software used to prepare material for publication: *WinGX*.

AMV thanks SeQuent Scientific Ltd, Mangalore, India, for the sample of the starting material, 1-hydrazinophthalazine. RJB acknowledges the NSF MRI program (grant No. CHE-0619278) for funds to purchase the X-ray diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2185).

References

Aydogan, F., Ocal, N., Turgut, Z. & Yolacan, C. (2001). *Bull. Korean Chem. Soc.* **22**, 476–480.
Desai, S. B., Desai, P. B. & Desai, K. R. (2001). *Heterocycl. Commun.* **7**, 83–90.
El-Masry, A. H., Fahmy, H. H. & Abdelwahed, S. H. A. (2000). *Molecules*, **5**, 1429–1438.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Hodnett, E. M. & Dunn, W. J. (1970). *J. Med. Chem.* **13**, 768–770.
Lynch, D. E. & McClenaghan, I. (2002*a*). *Acta Cryst.* C**58**, o642–o644.
Lynch, D. E. & McClenaghan, I. (2002*b*). *Acta Cryst.* E**58**, o1051–o1052.
Oxford Diffraction (2007). *CrysAlisPro* (Version 171.31.8) and *CrysAlis RED* (Version 1.171.31.8). Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
Pandey, S. N., Sriram, D., Nath, G. & De Clercq, E. (1999). *Il Farmaco*, **54**, 624–628.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Singh, W. M. & Dash, B. C. (1988). *Pesticides*, **22**, 33–37.
Taggi, A. E., Hafez, A. M., Wack, H., Young, B., Ferraris, D. & Lectka, T. (2002). *J. Am. Chem. Soc.* **124**, 6626–6635.

  doi:10.1107/S1600536807037051

supplementary materials

Acta Cryst. (2007). E**63**, o3674 [**doi:10.1107/S1600536807037051**]

4-Methoxybenzaldehyde (phthalazin-1-ylidene)hydrazone

R. J. Butcher, J. P. Jasinski, H. S. Yathirajan, A. M. Vijesh and B. Narayana

Comment

Schiff bases are used as substrates in the preparation of number of industrial and biologically active compounds *via* ring closure, cycloaddition and replacement reactions. Moreover, Schiff bases are also known to have biological activities such as antimicrobial, antifungal, and antitumor, as well they can be herbicides. Schiff bases have also been employed as ligands for complexation of metal ions. On the industrial scale, they have wide range of applications such as dyes and pigments. A new Schiff base, $C_{16}H_{14}N_4O$, (I), has been synthesized and its crystal structure is reported herein.

Two independent molecules of (I), molecules A and B, Fig. 1, comprise the crystallographic asymmetric unit. The phthalazin-1-yl and 4-methoxybenzaldehyde groups are coplanar with each other in each of the independent molecules with an angle between their mean planes of 15.4 (4) and 13.3 (9) Å in A and B, respectively. These groups are also planar with the hydrazone group, forming torsion angles of N4—N3—C8—N2 [1.8 (2)°] and N4—C9—C10—C11 [14.1 (2)°] for molecule A, and N4—N3—C8—N2 [−6.1 (2)°] and N4—C9—C10—C11 [−2.7 (2)°] for B.

Molecules A and B are linked by intermolecular N—H···N hydrogen bonding interactions (Table 1) between inverted phthalazin-1-yl groups. These stack diagonal to the *a* axis in a zigzag pattern (Fig. 2).

Experimental

A mixture of 1-hydrazinophthalazine (0.32 g, 0.002 mol) and 4-methoxybenzaldehyde (0.272 g, 0.002 mol) in absolute ethanol (15 ml) containing 2 drops of 4 *M* sulfuric acid was refluxed for about 3 h. On cooling, the solid separated was filtered and recrystallized from acetone (m.p. 419–23 K). Analysis found: C 68.93, H 5.01, N 20.04%; $C_{16}H_{14}N_4O$ requires: C 69.05, H 5.07, N 20.13%.

Refinement

The H atoms were included in the riding model approximation with C—H = 0.94 Å and N—H = 0.87 Å, and with U_{iso}(H) = 1.18–1.49 U_{eq}(C,*N*).

Figures

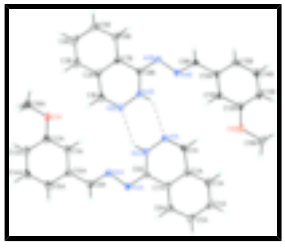


Fig. 1. Molecular structure of the two independent molecules in (I), showing atom labelling, 50% probability displacement ellipsoids and N—H···N hydrogen bonds as dashed lines.

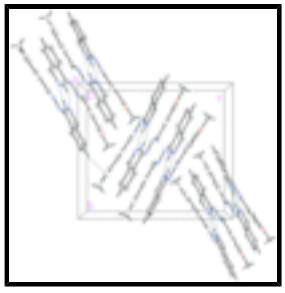


Fig. 2. Packing diagram of (I), viewed down the *a* axis. Dashed lines indicate N—H···N hydrogen bonds.

4-Methoxybenzaldehyde (phthalazin-1-ylidene)hydrazone

Crystal data

$C_{16}H_{14}N_4O$	F_{000} = 1168
M_r = 278.31	D_x = 1.335 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5610 reflections
a = 12.1351 (8) Å	θ = 4.6–32.4º
b = 14.2016 (7) Å	μ = 0.09 mm^{-1}
c = 16.0716 (8) Å	T = 203 K
β = 90.012 (6)º	Chunk, yellow
V = 2769.7 (3) Å^3	0.55 × 0.41 × 0.37 mm
Z = 8	

Data collection

Oxford Diffraction Gemini R diffractometer	R_{int} = 0.035
Radiation source: fine-focus sealed tube	θ_{max} = 32.5º
Monochromator: graphite	θ_{min} = 4.6º
T = 203 K	h = −18→15
φ and ω scans	k = −20→20
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2007)	l = −22→24
T_{min} = 0.833, T_{max} = 1.000	2 standard reflections
19111 measured reflections	every 50 reflections
8720 independent reflections	intensity decay: none
3864 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.057	H-atom parameters constrained
$wR(F^2)$ = 0.180	$w = 1/[\sigma^2(F_o^2) + (0.0884P)^2]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{max} = 0.002$
8720 reflections	$\Delta\rho_{max} = 0.27$ e Å^{-3}
381 parameters	$\Delta\rho_{min} = -0.23$ 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}
O1A	0.15752 (11)	0.62767 (9)	−0.14043 (8)	0.0594 (4)
O1B	−0.24183 (10)	1.16684 (8)	0.30313 (7)	0.0461 (3)
N1A	−0.03360 (11)	0.99989 (10)	0.12664 (9)	0.0438 (4)
N2A	0.06600 (11)	0.97845 (9)	0.09289 (8)	0.0387 (3)
H2AA	0.0711	0.9246	0.0674	0.046*
N3A	0.25209 (11)	1.00864 (9)	0.06025 (8)	0.0380 (3)
N4A	0.24386 (11)	0.92220 (9)	0.01902 (8)	0.0371 (3)
N1B	−0.04553 (11)	0.79403 (9)	0.04541 (9)	0.0420 (3)
N2B	−0.14890 (11)	0.82042 (9)	0.07002 (8)	0.0364 (3)
H2BA	−0.1556	0.8766	0.0910	0.044*
N3B	−0.33832 (11)	0.79568 (9)	0.09271 (8)	0.0375 (3)
N4B	−0.33017 (11)	0.88110 (9)	0.13504 (8)	0.0359 (3)
C1A	−0.04070 (14)	1.07925 (12)	0.16550 (11)	0.0455 (4)
H1AA	−0.1090	1.0952	0.1893	0.055*
C2A	0.04879 (14)	1.14498 (11)	0.17491 (10)	0.0388 (4)
C3A	0.03655 (16)	1.23042 (12)	0.21789 (11)	0.0462 (4)
H3AA	−0.0317	1.2465	0.2416	0.055*
C4A	0.12422 (17)	1.29002 (12)	0.22514 (10)	0.0481 (5)
H4AA	0.1160	1.3472	0.2540	0.058*
C5A	0.22507 (16)	1.26718 (12)	0.19052 (11)	0.0479 (5)
H5AA	0.2849	1.3087	0.1961	0.057*
C6A	0.23812 (14)	1.18376 (11)	0.14790 (10)	0.0402 (4)
H6AA	0.3068	1.1688	0.1243	0.048*
C7A	0.15041 (13)	1.12177 (10)	0.13965 (9)	0.0321 (4)
C8A	0.15901 (13)	1.03289 (10)	0.09489 (9)	0.0318 (3)
C9A	0.33402 (14)	0.89556 (11)	−0.01453 (9)	0.0375 (4)

H9AA	0.3968	0.9341	−0.0106	0.045*
C10A	0.34102 (14)	0.80607 (11)	−0.05895 (9)	0.0362 (4)
C11A	0.24709 (14)	0.75681 (11)	−0.08197 (9)	0.0378 (4)
H11A	0.1771	0.7822	−0.0707	0.045*
C12A	0.25592 (15)	0.67071 (12)	−0.12138 (10)	0.0418 (4)
C13A	0.35773 (16)	0.63304 (13)	−0.13927 (11)	0.0483 (5)
H13A	0.3635	0.5745	−0.1662	0.058*
C14A	0.45109 (16)	0.68217 (14)	−0.11715 (12)	0.0544 (5)
H14A	0.5208	0.6567	−0.1293	0.065*
C15A	0.44415 (15)	0.76813 (13)	−0.07745 (10)	0.0483 (5)
H15A	0.5086	0.8009	−0.0629	0.058*
C16A	0.16018 (19)	0.54019 (14)	−0.18424 (13)	0.0643 (6)
H16A	0.0855	0.5176	−0.1924	0.096*
H16B	0.2016	0.4944	−0.1522	0.096*
H16C	0.1952	0.5492	−0.2379	0.096*
C1B	−0.03699 (15)	0.71162 (12)	0.01132 (11)	0.0455 (4)
H1BA	0.0334	0.6919	−0.0057	0.055*
C2B	−0.12730 (14)	0.64846 (11)	−0.00231 (10)	0.0388 (4)
C3B	−0.11454 (17)	0.56187 (13)	−0.04278 (11)	0.0511 (5)
H3BA	−0.0445	0.5427	−0.0611	0.061*
C4B	−0.20408 (19)	0.50515 (13)	−0.05565 (12)	0.0596 (5)
H4BA	−0.1955	0.4475	−0.0836	0.072*
C5B	−0.30724 (18)	0.53223 (13)	−0.02775 (12)	0.0564 (5)
H5BA	−0.3679	0.4923	−0.0364	0.068*
C6B	−0.32239 (15)	0.61678 (12)	0.01251 (11)	0.0447 (4)
H6BA	−0.3927	0.6346	0.0313	0.054*
C7B	−0.23160 (14)	0.67581 (10)	0.02510 (9)	0.0349 (4)
C8B	−0.24254 (14)	0.76758 (10)	0.06503 (9)	0.0324 (3)
C9B	−0.42284 (13)	0.91673 (11)	0.15517 (10)	0.0368 (4)
H9BA	−0.4881	0.8870	0.1378	0.044*
C10B	−0.42954 (13)	1.00313 (11)	0.20488 (9)	0.0353 (4)
C11B	−0.33484 (13)	1.04619 (10)	0.23426 (9)	0.0348 (4)
H11B	−0.2656	1.0198	0.2223	0.042*
C12B	−0.34132 (14)	1.12789 (11)	0.28118 (9)	0.0371 (4)
C13B	−0.44355 (15)	1.16444 (12)	0.30276 (10)	0.0440 (4)
H13B	−0.4486	1.2178	0.3371	0.053*
C14B	−0.53719 (15)	1.12146 (12)	0.27317 (11)	0.0473 (4)
H14B	−0.6065	1.1466	0.2869	0.057*
C15B	−0.53156 (14)	1.04202 (11)	0.22357 (10)	0.0413 (4)
H15B	−0.5964	1.0145	0.2027	0.050*
C16B	−0.24432 (17)	1.25121 (12)	0.35107 (12)	0.0554 (5)
H16D	−0.1695	1.2716	0.3625	0.083*
H16E	−0.2824	1.2396	0.4031	0.083*
H16F	−0.2827	1.2999	0.3202	0.083*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

O1A	0.0544 (8)	0.0535 (8)	0.0702 (9)	0.0068 (6)	−0.0055 (7)	−0.0259 (7)
O1B	0.0552 (8)	0.0388 (7)	0.0444 (7)	0.0024 (5)	−0.0011 (6)	−0.0124 (5)
N1A	0.0339 (8)	0.0406 (8)	0.0568 (9)	0.0036 (6)	0.0016 (7)	−0.0031 (7)
N2A	0.0384 (8)	0.0336 (7)	0.0442 (8)	0.0006 (6)	−0.0021 (6)	−0.0063 (6)
N3A	0.0444 (8)	0.0318 (7)	0.0377 (7)	−0.0023 (6)	0.0053 (7)	−0.0001 (6)
N4A	0.0457 (8)	0.0304 (7)	0.0352 (7)	0.0014 (6)	0.0018 (7)	−0.0002 (6)
N1B	0.0406 (8)	0.0409 (8)	0.0444 (8)	0.0013 (6)	0.0010 (7)	−0.0027 (7)
N2B	0.0416 (8)	0.0291 (7)	0.0384 (7)	−0.0002 (6)	0.0007 (6)	−0.0060 (6)
N3B	0.0449 (8)	0.0278 (7)	0.0397 (7)	−0.0052 (6)	−0.0018 (7)	−0.0052 (6)
N4B	0.0420 (8)	0.0293 (7)	0.0364 (7)	−0.0016 (6)	0.0020 (6)	−0.0021 (6)
C1A	0.0375 (10)	0.0399 (10)	0.0590 (11)	0.0028 (7)	0.0069 (9)	−0.0013 (9)
C2A	0.0446 (10)	0.0361 (9)	0.0358 (8)	0.0013 (7)	0.0003 (8)	0.0011 (7)
C3A	0.0577 (12)	0.0382 (10)	0.0427 (9)	0.0060 (8)	0.0066 (9)	−0.0028 (8)
C4A	0.0706 (13)	0.0343 (9)	0.0395 (9)	0.0018 (9)	−0.0022 (9)	−0.0064 (8)
C5A	0.0605 (12)	0.0364 (10)	0.0468 (10)	−0.0089 (8)	−0.0087 (9)	−0.0029 (8)
C6A	0.0433 (10)	0.0377 (9)	0.0397 (9)	−0.0034 (7)	−0.0033 (8)	0.0006 (7)
C7A	0.0381 (9)	0.0296 (8)	0.0286 (7)	−0.0001 (6)	−0.0034 (7)	0.0025 (6)
C8A	0.0379 (9)	0.0288 (8)	0.0286 (7)	0.0004 (6)	−0.0043 (7)	0.0033 (6)
C9A	0.0447 (10)	0.0349 (9)	0.0330 (8)	−0.0030 (7)	0.0078 (8)	0.0055 (7)
C10A	0.0431 (10)	0.0369 (9)	0.0287 (8)	0.0024 (7)	0.0046 (7)	0.0062 (7)
C11A	0.0407 (10)	0.0390 (9)	0.0336 (8)	0.0096 (7)	0.0016 (7)	−0.0034 (7)
C12A	0.0488 (11)	0.0432 (10)	0.0334 (8)	0.0079 (8)	−0.0026 (8)	−0.0028 (7)
C13A	0.0573 (12)	0.0465 (10)	0.0411 (9)	0.0161 (9)	0.0011 (9)	−0.0050 (8)
C14A	0.0453 (11)	0.0659 (13)	0.0519 (11)	0.0192 (10)	0.0091 (9)	−0.0098 (10)
C15A	0.0428 (11)	0.0563 (11)	0.0460 (10)	0.0064 (8)	0.0063 (9)	0.0006 (9)
C16A	0.0768 (15)	0.0559 (13)	0.0602 (12)	0.0015 (10)	−0.0052 (12)	−0.0202 (10)
C1B	0.0467 (11)	0.0406 (10)	0.0492 (10)	0.0066 (8)	0.0007 (9)	−0.0011 (8)
C2B	0.0523 (11)	0.0289 (8)	0.0351 (8)	0.0064 (7)	−0.0035 (8)	−0.0014 (7)
C3B	0.0660 (13)	0.0399 (10)	0.0474 (10)	0.0152 (9)	−0.0080 (10)	−0.0077 (8)
C4B	0.0843 (15)	0.0346 (10)	0.0599 (12)	0.0097 (10)	−0.0186 (11)	−0.0145 (9)
C5B	0.0717 (14)	0.0362 (10)	0.0611 (12)	−0.0089 (9)	−0.0153 (11)	−0.0062 (9)
C6B	0.0523 (11)	0.0340 (9)	0.0477 (10)	−0.0044 (8)	−0.0062 (9)	−0.0058 (8)
C7B	0.0476 (10)	0.0285 (8)	0.0285 (7)	−0.0003 (7)	−0.0057 (7)	−0.0005 (6)
C8B	0.0436 (10)	0.0275 (8)	0.0261 (7)	−0.0018 (7)	−0.0038 (7)	0.0003 (6)
C9B	0.0376 (9)	0.0318 (8)	0.0411 (9)	−0.0063 (7)	0.0022 (8)	0.0041 (7)
C10B	0.0429 (9)	0.0297 (8)	0.0332 (8)	0.0031 (7)	0.0087 (7)	0.0049 (6)
C11B	0.0371 (9)	0.0319 (8)	0.0355 (8)	0.0040 (7)	0.0053 (7)	−0.0022 (7)
C12B	0.0501 (10)	0.0306 (8)	0.0306 (8)	0.0040 (7)	0.0019 (8)	0.0009 (6)
C13B	0.0617 (12)	0.0336 (9)	0.0367 (9)	0.0147 (8)	0.0069 (9)	−0.0012 (7)
C14B	0.0466 (11)	0.0460 (10)	0.0492 (10)	0.0136 (8)	0.0087 (9)	0.0037 (9)
C15B	0.0360 (9)	0.0434 (10)	0.0446 (9)	−0.0003 (7)	0.0037 (8)	0.0068 (8)
C16B	0.0767 (14)	0.0413 (10)	0.0481 (10)	0.0024 (9)	−0.0030 (10)	−0.0179 (8)

Geometric parameters (Å, °)

O1A—C12A	1.376 (2)	C12A—C13A	1.377 (2)
O1A—C16A	1.428 (2)	C13A—C14A	1.377 (3)
O1B—C12B	1.3740 (19)	C13A—H13A	0.9400
O1B—C16B	1.4248 (19)	C14A—C15A	1.380 (2)

N1A—C1A	1.291 (2)	C14A—H14A	0.9400
N1A—N2A	1.3594 (17)	C15A—H15A	0.9400
N2A—C8A	1.369 (2)	C16A—H16A	0.9700
N2A—H2AA	0.8700	C16A—H16B	0.9700
N3A—C8A	1.3056 (19)	C16A—H16C	0.9700
N3A—N4A	1.3985 (17)	C1B—C2B	1.433 (2)
N4A—C9A	1.2771 (19)	C1B—H1BA	0.9400
N1B—C1B	1.296 (2)	C2B—C7B	1.395 (2)
N1B—N2B	1.3678 (18)	C2B—C3B	1.400 (2)
N2B—C8B	1.3640 (19)	C3B—C4B	1.368 (3)
N2B—H2BA	0.8700	C3B—H3BA	0.9400
N3B—C8B	1.307 (2)	C4B—C5B	1.384 (3)
N3B—N4B	1.3943 (17)	C4B—H4BA	0.9400
N4B—C9B	1.2750 (19)	C5B—C6B	1.376 (2)
C1A—C2A	1.440 (2)	C5B—H5BA	0.9400
C1A—H1AA	0.9400	C6B—C7B	1.399 (2)
C2A—C7A	1.397 (2)	C6B—H6BA	0.9400
C2A—C3A	1.404 (2)	C7B—C8B	1.459 (2)
C3A—C4A	1.364 (3)	C9B—C10B	1.466 (2)
C3A—H3AA	0.9400	C9B—H9BA	0.9400
C4A—C5A	1.383 (3)	C10B—C11B	1.385 (2)
C4A—H4AA	0.9400	C10B—C15B	1.388 (2)
C5A—C6A	1.378 (2)	C11B—C12B	1.386 (2)
C5A—H5AA	0.9400	C11B—H11B	0.9400
C6A—C7A	1.388 (2)	C12B—C13B	1.389 (2)
C6A—H6AA	0.9400	C13B—C14B	1.375 (2)
C7A—C8A	1.457 (2)	C13B—H13B	0.9400
C9A—C10A	1.460 (2)	C14B—C15B	1.383 (2)
C9A—H9AA	0.9400	C14B—H14B	0.9400
C10A—C11A	1.387 (2)	C15B—H15B	0.9400
C10A—C15A	1.395 (2)	C16B—H16D	0.9700
C11A—C12A	1.381 (2)	C16B—H16E	0.9700
C11A—H11A	0.9400	C16B—H16F	0.9700
C12A—O1A—C16A	118.45 (15)	C10A—C15A—H15A	120.2
C12B—O1B—C16B	117.30 (13)	O1A—C16A—H16A	109.5
C1A—N1A—N2A	116.61 (14)	O1A—C16A—H16B	109.5
N1A—N2A—C8A	126.68 (13)	H16A—C16A—H16B	109.5
N1A—N2A—H2AA	116.7	O1A—C16A—H16C	109.5
C8A—N2A—H2AA	116.7	H16A—C16A—H16C	109.5
C8A—N3A—N4A	111.83 (13)	H16B—C16A—H16C	109.5
C9A—N4A—N3A	113.51 (14)	N1B—C1B—C2B	124.64 (17)
C1B—N1B—N2B	116.30 (14)	N1B—C1B—H1BA	117.7
C8B—N2B—N1B	126.61 (13)	C2B—C1B—H1BA	117.7
C8B—N2B—H2BA	116.7	C7B—C2B—C3B	119.45 (16)
N1B—N2B—H2BA	116.7	C7B—C2B—C1B	118.11 (14)
C8B—N3B—N4B	111.63 (13)	C3B—C2B—C1B	122.43 (17)
C9B—N4B—N3B	113.99 (13)	C4B—C3B—C2B	119.97 (18)
N1A—C1A—C2A	124.48 (16)	C4B—C3B—H3BA	120.0
N1A—C1A—H1AA	117.8	C2B—C3B—H3BA	120.0

C2A—C1A—H1AA	117.8	C3B—C4B—C5B	120.37 (17)
C7A—C2A—C3A	119.81 (16)	C3B—C4B—H4BA	119.8
C7A—C2A—C1A	118.05 (15)	C5B—C4B—H4BA	119.8
C3A—C2A—C1A	122.14 (16)	C6B—C5B—C4B	121.03 (18)
C4A—C3A—C2A	119.70 (17)	C6B—C5B—H5BA	119.5
C4A—C3A—H3AA	120.1	C4B—C5B—H5BA	119.5
C2A—C3A—H3AA	120.1	C5B—C6B—C7B	119.05 (18)
C3A—C4A—C5A	120.68 (16)	C5B—C6B—H6BA	120.5
C3A—C4A—H4AA	119.7	C7B—C6B—H6BA	120.5
C5A—C4A—H4AA	119.7	C2B—C7B—C6B	120.11 (15)
C6A—C5A—C4A	120.23 (17)	C2B—C7B—C8B	118.05 (14)
C6A—C5A—H5AA	119.9	C6B—C7B—C8B	121.82 (16)
C4A—C5A—H5AA	119.9	N3B—C8B—N2B	123.57 (14)
C5A—C6A—C7A	120.32 (17)	N3B—C8B—C7B	120.23 (14)
C5A—C6A—H6AA	119.8	N2B—C8B—C7B	116.20 (14)
C7A—C6A—H6AA	119.8	N4B—C9B—C10B	121.29 (15)
C6A—C7A—C2A	119.26 (15)	N4B—C9B—H9BA	119.4
C6A—C7A—C8A	122.82 (15)	C10B—C9B—H9BA	119.4
C2A—C7A—C8A	117.92 (14)	C11B—C10B—C15B	119.38 (15)
N3A—C8A—N2A	123.68 (14)	C11B—C10B—C9B	120.61 (14)
N3A—C8A—C7A	120.08 (14)	C15B—C10B—C9B	120.01 (15)
N2A—C8A—C7A	116.23 (14)	C10B—C11B—C12B	120.53 (15)
N4A—C9A—C10A	120.94 (15)	C10B—C11B—H11B	119.7
N4A—C9A—H9AA	119.5	C12B—C11B—H11B	119.7
C10A—C9A—H9AA	119.5	O1B—C12B—C11B	115.26 (14)
C11A—C10A—C15A	119.05 (15)	O1B—C12B—C13B	124.77 (15)
C11A—C10A—C9A	121.42 (15)	C11B—C12B—C13B	119.96 (16)
C15A—C10A—C9A	119.52 (16)	C14B—C13B—C12B	119.09 (16)
C12A—C11A—C10A	120.31 (16)	C14B—C13B—H13B	120.5
C12A—C11A—H11A	119.8	C12B—C13B—H13B	120.5
C10A—C11A—H11A	119.8	C13B—C14B—C15B	121.37 (16)
O1A—C12A—C13A	124.04 (16)	C13B—C14B—H14B	119.3
O1A—C12A—C11A	115.33 (15)	C15B—C14B—H14B	119.3
C13A—C12A—C11A	120.63 (17)	C14B—C15B—C10B	119.56 (16)
C12A—C13A—C14A	119.18 (17)	C14B—C15B—H15B	120.2
C12A—C13A—H13A	120.4	C10B—C15B—H15B	120.2
C14A—C13A—H13A	120.4	O1B—C16B—H16D	109.5
C13A—C14A—C15A	121.15 (17)	O1B—C16B—H16E	109.5
C13A—C14A—H14A	119.4	H16D—C16B—H16E	109.5
C15A—C14A—H14A	119.4	O1B—C16B—H16F	109.5
C14A—C15A—C10A	119.68 (18)	H16D—C16B—H16F	109.5
C14A—C15A—H15A	120.2	H16E—C16B—H16F	109.5
C1A—N1A—N2A—C8A	−1.0 (2)	C11A—C10A—C15A—C14A	−0.8 (2)
C8A—N3A—N4A—C9A	−178.96 (13)	C9A—C10A—C15A—C14A	178.01 (15)
C1B—N1B—N2B—C8B	2.6 (2)	N2B—N1B—C1B—C2B	0.5 (2)
C8B—N3B—N4B—C9B	172.58 (13)	N1B—C1B—C2B—C7B	−2.0 (3)
N2A—N1A—C1A—C2A	0.1 (3)	N1B—C1B—C2B—C3B	177.08 (15)
N1A—C1A—C2A—C7A	0.1 (3)	C7B—C2B—C3B—C4B	0.4 (3)
N1A—C1A—C2A—C3A	−179.98 (16)	C1B—C2B—C3B—C4B	−178.65 (17)

C7A—C2A—C3A—C4A	−0.2 (2)	C2B—C3B—C4B—C5B	−1.0 (3)
C1A—C2A—C3A—C4A	179.81 (16)	C3B—C4B—C5B—C6B	0.7 (3)
C2A—C3A—C4A—C5A	0.0 (3)	C4B—C5B—C6B—C7B	0.0 (3)
C3A—C4A—C5A—C6A	0.2 (3)	C3B—C2B—C7B—C6B	0.3 (2)
C4A—C5A—C6A—C7A	−0.3 (2)	C1B—C2B—C7B—C6B	179.45 (15)
C5A—C6A—C7A—C2A	0.1 (2)	C3B—C2B—C7B—C8B	−178.37 (13)
C5A—C6A—C7A—C8A	179.61 (14)	C1B—C2B—C7B—C8B	0.7 (2)
C3A—C2A—C7A—C6A	0.2 (2)	C5B—C6B—C7B—C2B	−0.6 (2)
C1A—C2A—C7A—C6A	−179.89 (15)	C5B—C6B—C7B—C8B	178.09 (14)
C3A—C2A—C7A—C8A	−179.37 (14)	N4B—N3B—C8B—N2B	−6.1 (2)
C1A—C2A—C7A—C8A	0.6 (2)	N4B—N3B—C8B—C7B	174.81 (12)
N4A—N3A—C8A—N2A	1.8 (2)	N1B—N2B—C8B—N3B	177.24 (14)
N4A—N3A—C8A—C7A	−178.92 (12)	N1B—N2B—C8B—C7B	−3.7 (2)
N1A—N2A—C8A—N3A	−179.13 (14)	C2B—C7B—C8B—N3B	−179.08 (14)
N1A—N2A—C8A—C7A	1.6 (2)	C6B—C7B—C8B—N3B	2.2 (2)
C6A—C7A—C8A—N3A	−0.1 (2)	C2B—C7B—C8B—N2B	1.8 (2)
C2A—C7A—C8A—N3A	179.37 (13)	C6B—C7B—C8B—N2B	−176.90 (14)
C6A—C7A—C8A—N2A	179.17 (14)	N3B—N4B—C9B—C10B	175.84 (12)
C2A—C7A—C8A—N2A	−1.3 (2)	N4B—C9B—C10B—C11B	−2.7 (2)
N3A—N4A—C9A—C10A	178.92 (12)	N4B—C9B—C10B—C15B	177.89 (14)
N4A—C9A—C10A—C11A	14.1 (2)	C15B—C10B—C11B—C12B	−0.7 (2)
N4A—C9A—C10A—C15A	−164.71 (15)	C9B—C10B—C11B—C12B	179.95 (13)
C15A—C10A—C11A—C12A	1.1 (2)	C16B—O1B—C12B—C11B	179.70 (14)
C9A—C10A—C11A—C12A	−177.74 (13)	C16B—O1B—C12B—C13B	−0.4 (2)
C16A—O1A—C12A—C13A	−2.5 (3)	C10B—C11B—C12B—O1B	−176.73 (13)
C16A—O1A—C12A—C11A	177.52 (15)	C10B—C11B—C12B—C13B	3.4 (2)
C10A—C11A—C12A—O1A	179.25 (14)	O1B—C12B—C13B—C14B	176.61 (14)
C10A—C11A—C12A—C13A	−0.8 (2)	C11B—C12B—C13B—C14B	−3.5 (2)
O1A—C12A—C13A—C14A	−179.82 (16)	C12B—C13B—C14B—C15B	1.0 (3)
C11A—C12A—C13A—C14A	0.2 (3)	C13B—C14B—C15B—C10B	1.7 (2)
C12A—C13A—C14A—C15A	0.1 (3)	C11B—C10B—C15B—C14B	−1.9 (2)
C13A—C14A—C15A—C10A	0.3 (3)	C9B—C10B—C15B—C14B	177.52 (14)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2A—H2AA···N1B	0.87	2.36	3.0451 (19)	136
N2B—H2BA···N1A	0.87	2.36	3.0466 (19)	136

Fig. 1

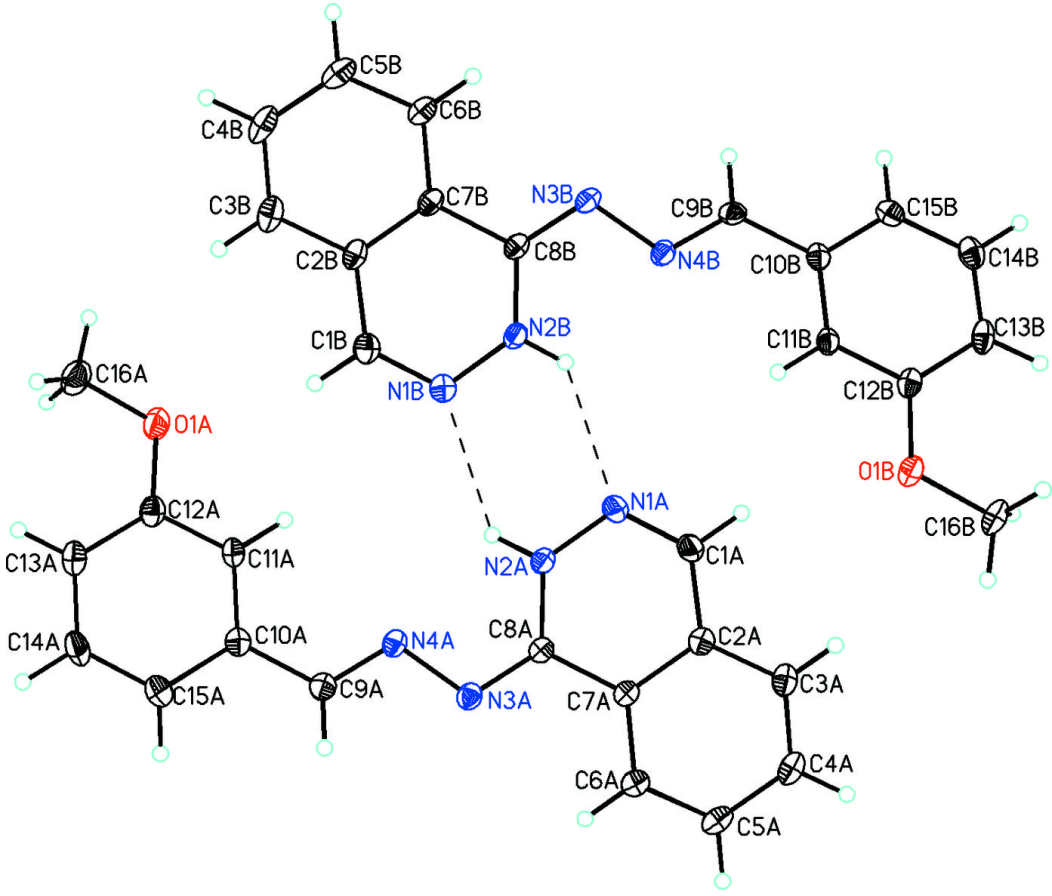

C5B
C6B
C4B
C3B
C7B
N3B
C9B
C15B
C2B
C8B
N4B
C10B
C14B
C1B
N2B
C11B
C13B
C16A
N1B
C12B
O1A
O1B
N1A
C12A
C16B
C1A
C13A
C11A
N2A
C14A
C10A
N4A
C8A
C2A
C3A
C15A
C9A
N3A
C7A
C4A
C6A
C5A

Fig. 2