Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-(4-Bromophenylhydrazono)-1-(5-bromopyrimidin-2-yl)-3-methyl-2-pyrazolin-5-one

M. Subramanyam,[a] B. Lingappa,[b] A. Thiruvalluvar,[a]* R. J. Butcher[c] and Balakrishna Kalluraya[b]

[a]PG Research Department of Physics, Rajah Serfoji Government College (Autonomous), Thanjavur 613 005, Tamil Nadu, India, [b]Department of Studies in Chemistry, Mangalore University, Mangalagangothri 574 199, Karnataka, India, and [c]Department of Chemistry, Howard University, 525 College Street NW, Washington, DC 20059, USA
Correspondence e-mail: athiru@vsnl.net

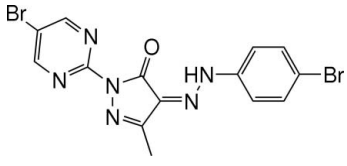
Received 16 November 2007; accepted 20 December 2007

Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.008 Å; R factor = 0.083; wR factor = 0.139; data-to-parameter ratio = 23.0.

The asymmetric unit of the title compound, $C_{14}H_{10}Br_2N_6O$, contains two crystallographically independent molecules. The pyrazole ring of one molecule makes dihedral angles of 22.0 (3) and 3.5 (3)° with the pyrimidine and benzene rings, respectively; the corresponding values in the other molecule are 9.2 (3) and 2.1 (3)°, respectively. The molecules are linked by N—H···O, C—H···N and C—H···Br hydrogen bonds.

Related literature

For related literature, see: Baraldi *et al.* (1996, 2003); Kalluraya & Rahiman (1997); Kalluraya *et al.* (2001); Lingappa *et al.* (2006, 2007). For related crystal structures, see: Thiruvalluvar, Subramanyam, Kalluraya *et al.* (2007*a*,*b*); Thiruvalluvar, Subramanyam, Lingappa *et al.* (2007).



Experimental

Crystal data

$C_{14}H_{10}Br_2N_6O$
M_r = 438.08
Triclinic, $P\overline{1}$
a = 9.1647 (3) Å
b = 12.2833 (5) Å
c = 14.0288 (5) Å
α = 86.532 (3)°
β = 84.218 (3)°
γ = 78.747 (3)°
V = 1539.67 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 5.28 mm^{-1}
T = 200 (2) K
0.44 × 0.39 × 0.28 mm

Data collection

Oxford Diffraction Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
T_{min} = 0.205, T_{max} = 0.320 (expected range = 0.146–0.228)
24958 measured reflections
9542 independent reflections
6336 reflections with $I > 2\sigma(I)$
R_{int} = 0.057

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.082
$wR(F^2)$ = 0.139
S = 1.25
9542 reflections
415 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.83 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N6*A*—H6*A*···O5*A*	0.88	2.14	2.821 (6)	134
N6*B*—H6*B*···O5*B*	0.88	2.10	2.782 (6)	134
C13*A*—H13*A*···N12*B*^{i}	0.95	2.61	3.193 (7)	120
C15*A*—H15*A*···Br4^{ii}	0.95	2.92	3.761 (6)	148
C15*B*—H15*B*···Br2^{ii}	0.95	2.87	3.746 (6)	153
C31*A*—H31*A*···Br3^{i}	0.98	2.88	3.858 (6)	173
C31*B*—H31*D*···Br1^{i}	0.98	2.92	3.862 (6)	160
C45*A*—H45*A*···N16*B*^{ii}	0.95	2.62	3.539 (7)	162

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

RJB acknowledges the NSF-MRI program for funding to purchase the X-ray CCD diffractometer. AT thanks the UGC, India, for the award of a Minor Research Project [file No. MRP-2355/06(UGC-SERO), link No. 2355, 10/01/2007].

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2116).

References

Baraldi, P. G., Cacciari, B. & Spalluto, G. (1996). *J. Med. Chem.* **39**, 1164–1169.
Baraldi, P. G., Fruttarolo, F., Tabrizi, M. A., Preti, D., Romagnoli, H., El-Kashef, H., Moorman, A., Varani, K., Gessi, S., Merighi, S. & Borea, P. A. (2003). *J. Med. Chem.* **46**, 1229–1233.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Kalluraya, B. & Rahiman, A. M. (1997). *Pol. J. Chem.* **1049**, 71–75.
Kalluraya, B., Rahiman, A. M. & Banji, D. (2001). *Arch. Pharm. Med. Chem.* **263**, 334–341.
Lingappa, B., Kalluraya, B. & Satheesha Rai, N. (2006). *Indian J. Org. Chem.* **2**, 5–6.
Lingappa, B., Kalluraya, B. & Satheesha Rai, N. (2007). *Phosphorus, Sulphur Silicon Relat. Elem.* **182**, 1393–1401.
Oxford Diffraction (2007). *CrysAlis CCD* and *CrysAlis RED*. Versions 1.171.32. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Thiruvalluvar, A., Subramanyam, M., Kalluraya, B. & Lingappa, B. (2007*a*). *Acta Cryst.* E**63**, o2911.
Thiruvalluvar, A., Subramanyam, M., Kalluraya, B. & Lingappa, B. (2007*b*). *Acta Cryst.* E**63**, o3362.
Thiruvalluvar, A., Subramanyam, M., Lingappa, B. & Kalluraya, B. (2007). *Acta Cryst.* E**63**, o3425.

  doi:10.1107/S1600536807067943

supplementary materials

Acta Cryst. (2008). E**64**, o362 [**doi:10.1107/S1600536807067943**]

4-(4-Bromophenylhydrazono)-1-(5-bromopyrimidin-2-yl)-3-methyl-2-pyrazolin-5-one

M. Subramanyam, B. Lingappa, A. Thiruvalluvar, R. J. Butcher and B. Kalluraya

Comment

The chemistry of pyrimidine and its derivatives has been studied due to their diverse biological activities (Baraldi *et al.*, 1996; Lingappa *et al.*, 2006, 2007). Pyrazoles and their derivatives are found to be associated with diverse pharmacological activities. Pyrazoles constitute an important class of heterocycles, which display interesting biological properties such as fungicidal, anti-inflammatory, anti-arthritic, anti-depressant and antiviral activity (Baraldi *et al.*, 2003; Kalluraya *et al.*, 2001; Kalluraya & Rahiman, 1997). The title compound, has been analysed as part of our crystallographic studies on pyrimidine derivatives (Thiruvalluvar *et al.*, 2007*a*,b; Thiruvalluvar, Subramanyam, Lingappa *et al.*, 2007). The asymmetric unit of the title compound, $C_{14}H_{10}Br_2N_6O$, Fig. 1, contains two crystallographically independent molecules, A and B. The pyrazole ring of A makes dihedral angles of 22.0 (3)° and 3.5 (3)° with the pyrimidine ring and phenyl ring respectively; the corresponding values in molecule B are 9.2 (3)° and 2.1 (3)°. The molecules are linked by N—H···O, C—H···N and C—H···Br hydrogen bonds; see Fig. 2 and hydrogen bond table.

Experimental

Ethyl-2-(4-bromohydrazono-3-oxobutanoate (3.2 g, 0.01 mol) was dissolved in glacial acetic acid (15 ml). To this a solution of 2-hydrazino-5-bromo-pyrimidine (1.9 g, 0.01 mol) in glacial acetic acid (20 ml) was added and the mixture was refluxed for 4 h in an oil bath. It was cooled and allowed to stand overnight. The separated solid was filtered, dried and then recrystallized from ethanol to give reddish flakes. Further recrystallization from ethyl acetate gave the yellowish red crystals suitable for X-ray analysis (3.0 g, 70%).

Refinement

H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.88–0.98 Å and U_{iso}=1.2 or 1.5 times U_{eq}(C).

Figures

Fig. 1. The molecular structure of the title compound with the atomic numbering and 50% probability displacement ellipsoids. H atoms are shown as small spheres of arbitrary radius.

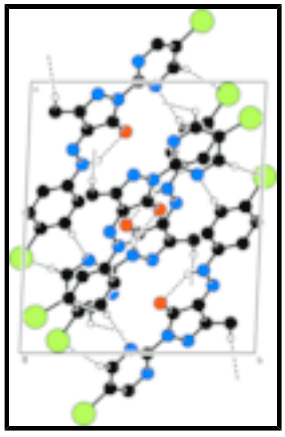


Fig. 2. The packing of the title compound, viewed down the *a* axis. Dashed lines indicate hydrogen bonds.

4-(4-Bromophenylhydrazono)-1-(5-bromopyrimidin-2-yl)-3-methyl-2-pyrazolin-5-one

Crystal data

$C_{14}H_{10}Br_2N_6O$	$Z = 4$
$M_r = 438.08$	$F_{000} = 856$
Triclinic, $P\overline{1}$	$D_x = 1.890$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 534(1) K
$a = 9.1647$ (3) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 12.2833$ (5) Å	Cell parameters from 5753 reflections
$c = 14.0288$ (5) Å	$\theta = 4.6$–$32.4°$
$\alpha = 86.532$ (3)°	$\mu = 5.28$ mm^{-1}
$\beta = 84.218$ (3)°	$T = 200$ (2) K
$\gamma = 78.747$ (3)°	Prism, pale-yellow
$V = 1539.67$ (10) Å^3	0.44 × 0.39 × 0.28 mm

Data collection

Oxford Diffraction Gemini diffractometer	9542 independent reflections
Radiation source: fine-focus sealed tube	6336 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.057$
$T = 200$(2) K	$\theta_{max} = 32.5°$
φ and ω scans	$\theta_{min} = 4.6°$
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2007)	$h = -13 \rightarrow 13$
$T_{min} = 0.205$, $T_{max} = 0.320$	$k = -18 \rightarrow 18$
24958 measured reflections	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.082$	H-atom parameters constrained
$wR(F^2) = 0.139$	$w = 1/[\sigma^2(F_o^2) + (0.0029P)^2 + 9.1374P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.25$	$(\Delta/\sigma)_{max} < 0.001$
9542 reflections	$\Delta\rho_{max} = 0.83$ e Å^{-3}
415 parameters	$\Delta\rho_{min} = -0.64$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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}
Br1	0.25785 (9)	0.06030 (5)	0.13400 (5)	0.0442 (2)
Br2	0.35450 (8)	0.83940 (5)	0.95346 (4)	0.0376 (2)
O5A	0.3522 (5)	0.4304 (3)	0.4711 (3)	0.0337 (12)
N1A	0.1660 (5)	0.4635 (4)	0.3613 (3)	0.0230 (12)
N2A	0.0409 (5)	0.5473 (4)	0.3440 (3)	0.0233 (12)
N5A	0.1545 (5)	0.6383 (4)	0.5533 (3)	0.0236 (12)
N6A	0.2645 (5)	0.5980 (4)	0.6055 (3)	0.0272 (14)
N12A	0.1363 (5)	0.3829 (4)	0.2204 (3)	0.0269 (14)
N16A	0.2742 (5)	0.2773 (4)	0.3423 (3)	0.0283 (14)
C3A	0.0276 (6)	0.6191 (4)	0.4112 (4)	0.0241 (16)
C4A	0.1428 (6)	0.5855 (4)	0.4762 (4)	0.0249 (17)
C5A	0.2370 (6)	0.4835 (4)	0.4405 (4)	0.0251 (17)
C11A	0.1945 (6)	0.3696 (4)	0.3045 (4)	0.0233 (17)
C13A	0.1549 (6)	0.2918 (4)	0.1690 (4)	0.0256 (17)
C14A	0.2345 (6)	0.1912 (5)	0.2024 (4)	0.0267 (17)
C15A	0.2941 (7)	0.1879 (4)	0.2893 (4)	0.0294 (16)
C31A	−0.0912 (7)	0.7199 (5)	0.4167 (4)	0.0310 (17)
C41A	0.2811 (6)	0.6529 (4)	0.6887 (4)	0.0218 (14)
C42A	0.1781 (6)	0.7470 (5)	0.7170 (4)	0.0269 (17)
C43A	0.1993 (7)	0.7997 (5)	0.7976 (4)	0.0289 (17)
C44A	0.3238 (6)	0.7582 (4)	0.8478 (4)	0.0240 (16)
C45A	0.4250 (6)	0.6644 (5)	0.8210 (4)	0.0274 (17)
C46A	0.4032 (6)	0.6110 (5)	0.7402 (4)	0.0268 (17)
Br3	0.35984 (7)	0.28407 (6)	−0.22267 (5)	0.0439 (2)
Br4	0.35687 (7)	1.01017 (5)	0.64977 (4)	0.0352 (2)

O5B	0.3253 (4)	0.5890 (3)	0.1841 (3)	0.0289 (11)
N1B	0.1528 (5)	0.6272 (4)	0.0654 (3)	0.0242 (12)
N2B	0.0319 (5)	0.7132 (4)	0.0455 (3)	0.0257 (12)
N5B	0.1476 (5)	0.8095 (4)	0.2504 (3)	0.0243 (12)
N6B	0.2564 (5)	0.7670 (4)	0.3048 (3)	0.0255 (12)
N12B	0.1135 (5)	0.5468 (4)	−0.0740 (3)	0.0246 (12)
N16B	0.3245 (5)	0.4721 (4)	0.0142 (3)	0.0297 (16)
C3B	0.0199 (6)	0.7867 (5)	0.1108 (4)	0.0257 (17)
C4B	0.1306 (6)	0.7519 (5)	0.1779 (4)	0.0243 (16)
C5B	0.2192 (6)	0.6457 (5)	0.1473 (4)	0.0225 (16)
C11B	0.1992 (6)	0.5427 (4)	−0.0014 (4)	0.0221 (14)
C13B	0.1610 (6)	0.4687 (4)	−0.1378 (4)	0.0253 (17)
C14B	0.2899 (6)	0.3913 (5)	−0.1295 (4)	0.0280 (17)
C15B	0.3703 (7)	0.3965 (5)	−0.0520 (4)	0.0337 (17)
C31B	−0.0924 (7)	0.8914 (5)	0.1110 (4)	0.0378 (19)
C41B	0.2806 (6)	0.8256 (5)	0.3837 (4)	0.0248 (17)
C42B	0.1926 (7)	0.9294 (5)	0.4041 (4)	0.0326 (17)
C43B	0.2166 (7)	0.9838 (5)	0.4834 (4)	0.0327 (17)
C44B	0.3283 (6)	0.9332 (5)	0.5407 (4)	0.0261 (16)
C45B	0.4170 (7)	0.8313 (5)	0.5212 (4)	0.0281 (17)
C46B	0.3920 (6)	0.7777 (4)	0.4417 (4)	0.0258 (16)
H6A	0.32796	0.53710	0.58914	0.0325*
H13A	0.11317	0.29614	0.10926	0.0305*
H15A	0.35103	0.11993	0.31218	0.0351*
H31A	−0.15409	0.72242	0.36379	0.0462*
H31B	−0.15261	0.71796	0.47794	0.0462*
H31C	−0.04557	0.78606	0.41202	0.0462*
H42A	0.09463	0.77462	0.68162	0.0322*
H43A	0.12971	0.86348	0.81856	0.0344*
H45A	0.50800	0.63667	0.85679	0.0324*
H46A	0.47163	0.54612	0.72037	0.0320*
H6B	0.31353	0.70229	0.29195	0.0304*
H13B	0.10427	0.46644	−0.19044	0.0301*
H15B	0.46075	0.34463	−0.04581	0.0402*
H31D	−0.15362	0.89370	0.05723	0.0567*
H31E	−0.15657	0.89487	0.17142	0.0567*
H31F	−0.04134	0.95486	0.10445	0.0567*
H42B	0.11701	0.96245	0.36400	0.0391*
H43B	0.15775	1.05438	0.49828	0.0397*
H45B	0.49313	0.79874	0.56111	0.0337*
H46B	0.45184	0.70742	0.42684	0.0303*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0592 (4)	0.0278 (3)	0.0472 (4)	−0.0018 (3)	−0.0158 (3)	−0.0168 (3)
Br2	0.0477 (4)	0.0328 (3)	0.0341 (3)	−0.0035 (3)	−0.0141 (3)	−0.0127 (3)
O5A	0.036 (2)	0.031 (2)	0.034 (2)	0.0029 (19)	−0.0159 (19)	−0.0093 (18)

N1A	0.026 (2)	0.020 (2)	0.023 (2)	0.0008 (19)	−0.0097 (18)	−0.0067 (18)
N2A	0.026 (2)	0.018 (2)	0.025 (2)	−0.0002 (19)	−0.0041 (18)	−0.0041 (18)
N5A	0.028 (2)	0.023 (2)	0.020 (2)	−0.003 (2)	−0.0056 (18)	−0.0030 (18)
N6A	0.033 (3)	0.022 (2)	0.025 (2)	0.001 (2)	−0.005 (2)	−0.0042 (19)
N12A	0.032 (3)	0.023 (2)	0.026 (2)	−0.001 (2)	−0.010 (2)	−0.0059 (19)
N16A	0.035 (3)	0.023 (2)	0.025 (2)	0.004 (2)	−0.011 (2)	−0.0035 (19)
C3A	0.028 (3)	0.020 (2)	0.024 (3)	−0.003 (2)	−0.005 (2)	0.001 (2)
C4A	0.029 (3)	0.021 (3)	0.026 (3)	−0.007 (2)	−0.002 (2)	−0.004 (2)
C5A	0.031 (3)	0.022 (3)	0.022 (3)	−0.003 (2)	−0.005 (2)	0.000 (2)
C11A	0.025 (3)	0.022 (3)	0.024 (3)	−0.005 (2)	−0.004 (2)	−0.005 (2)
C13A	0.029 (3)	0.025 (3)	0.023 (3)	−0.002 (2)	−0.008 (2)	−0.004 (2)
C14A	0.029 (3)	0.029 (3)	0.026 (3)	−0.012 (2)	−0.004 (2)	−0.008 (2)
C15A	0.037 (3)	0.016 (2)	0.034 (3)	0.002 (2)	−0.012 (3)	0.000 (2)
C31A	0.032 (3)	0.029 (3)	0.032 (3)	−0.002 (3)	−0.007 (2)	−0.007 (2)
C41A	0.026 (3)	0.021 (2)	0.019 (2)	−0.005 (2)	−0.003 (2)	−0.002 (2)
C42A	0.029 (3)	0.025 (3)	0.026 (3)	−0.001 (2)	−0.008 (2)	−0.001 (2)
C43A	0.034 (3)	0.021 (3)	0.030 (3)	0.002 (2)	−0.005 (2)	−0.008 (2)
C44A	0.031 (3)	0.023 (3)	0.019 (2)	−0.006 (2)	−0.004 (2)	−0.003 (2)
C45A	0.026 (3)	0.033 (3)	0.024 (3)	−0.005 (2)	−0.005 (2)	−0.005 (2)
C46A	0.025 (3)	0.029 (3)	0.027 (3)	−0.004 (2)	−0.004 (2)	−0.006 (2)
Br3	0.0343 (3)	0.0493 (4)	0.0480 (4)	0.0003 (3)	−0.0035 (3)	−0.0294 (3)
Br4	0.0448 (4)	0.0345 (3)	0.0278 (3)	−0.0061 (3)	−0.0067 (3)	−0.0123 (2)
O5B	0.033 (2)	0.029 (2)	0.0251 (19)	−0.0004 (18)	−0.0116 (17)	−0.0067 (16)
N1B	0.023 (2)	0.023 (2)	0.027 (2)	−0.0024 (19)	−0.0071 (18)	−0.0026 (19)
N2B	0.024 (2)	0.024 (2)	0.027 (2)	0.003 (2)	−0.0061 (19)	−0.0031 (19)
N5B	0.026 (2)	0.024 (2)	0.023 (2)	−0.003 (2)	−0.0044 (18)	−0.0050 (18)
N6B	0.028 (2)	0.025 (2)	0.023 (2)	−0.001 (2)	−0.0056 (19)	−0.0060 (18)
N12B	0.026 (2)	0.023 (2)	0.025 (2)	−0.003 (2)	−0.0058 (18)	−0.0033 (18)
N16B	0.030 (3)	0.034 (3)	0.023 (2)	0.003 (2)	−0.0068 (19)	−0.006 (2)
C3B	0.023 (3)	0.026 (3)	0.027 (3)	0.000 (2)	−0.005 (2)	−0.004 (2)
C4B	0.027 (3)	0.027 (3)	0.020 (2)	−0.006 (2)	−0.003 (2)	−0.006 (2)
C5B	0.024 (3)	0.027 (3)	0.018 (2)	−0.007 (2)	−0.005 (2)	−0.001 (2)
C11B	0.024 (3)	0.021 (2)	0.020 (2)	0.000 (2)	−0.003 (2)	−0.002 (2)
C13B	0.028 (3)	0.024 (3)	0.025 (3)	−0.005 (2)	−0.006 (2)	−0.004 (2)
C14B	0.025 (3)	0.029 (3)	0.028 (3)	0.001 (2)	0.001 (2)	−0.010 (2)
C15B	0.026 (3)	0.038 (3)	0.033 (3)	0.009 (3)	−0.009 (2)	−0.007 (3)
C31B	0.041 (4)	0.039 (3)	0.029 (3)	0.009 (3)	−0.009 (3)	−0.011 (3)
C41B	0.025 (3)	0.027 (3)	0.022 (3)	−0.003 (2)	0.000 (2)	−0.007 (2)
C42B	0.038 (3)	0.031 (3)	0.028 (3)	0.000 (3)	−0.011 (3)	−0.003 (2)
C43B	0.037 (3)	0.028 (3)	0.031 (3)	0.002 (3)	−0.004 (3)	−0.009 (2)
C44B	0.032 (3)	0.029 (3)	0.018 (2)	−0.008 (3)	−0.001 (2)	−0.002 (2)
C45B	0.029 (3)	0.032 (3)	0.023 (3)	−0.003 (3)	−0.006 (2)	−0.002 (2)
C46B	0.031 (3)	0.021 (2)	0.025 (3)	−0.004 (2)	−0.002 (2)	−0.003 (2)

Geometric parameters (Å, °)

Br1—C14A	1.888 (6)	C41A—C46A	1.390 (8)
Br2—C44A	1.909 (5)	C41A—C42A	1.392 (8)
Br3—C14B	1.888 (6)	C42A—C43A	1.385 (8)

Br4—C44B	1.912 (6)	C43A—C44A	1.396 (8)
O5A—C5A	1.230 (7)	C44A—C45A	1.376 (8)
O5B—C5B	1.221 (7)	C45A—C46A	1.394 (8)
N1A—N2A	1.414 (7)	C13A—H13A	0.9500
N1A—C11A	1.409 (7)	C15A—H15A	0.9500
N1A—C5A	1.400 (7)	C31A—H31A	0.9800
N2A—C3A	1.310 (7)	C31A—H31C	0.9800
N5A—N6A	1.307 (6)	C31A—H31B	0.9800
N5A—C4A	1.318 (7)	C42A—H42A	0.9500
N6A—C41A	1.418 (7)	C43A—H43A	0.9500
N12A—C11A	1.331 (7)	C45A—H45A	0.9500
N12A—C13A	1.342 (7)	C46A—H46A	0.9500
N16A—C11A	1.335 (7)	C3B—C4B	1.440 (8)
N16A—C15A	1.336 (7)	C3B—C31B	1.482 (9)
N6A—H6A	0.8800	C4B—C5B	1.459 (8)
N1B—N2B	1.411 (7)	C13B—C14B	1.374 (8)
N1B—C11B	1.415 (7)	C14B—C15B	1.385 (8)
N1B—C5B	1.403 (7)	C41B—C46B	1.388 (8)
N2B—C3B	1.307 (7)	C41B—C42B	1.398 (9)
N5B—C4B	1.312 (7)	C42B—C43B	1.389 (8)
N5B—N6B	1.323 (6)	C43B—C44B	1.392 (8)
N6B—C41B	1.414 (7)	C44B—C45B	1.379 (9)
N12B—C13B	1.332 (7)	C45B—C46B	1.389 (8)
N12B—C11B	1.340 (7)	C13B—H13B	0.9500
N16B—C15B	1.332 (7)	C15B—H15B	0.9500
N16B—C11B	1.326 (7)	C31B—H31D	0.9800
N6B—H6B	0.8800	C31B—H31E	0.9800
C3A—C31A	1.481 (8)	C31B—H31F	0.9800
C3A—C4A	1.447 (8)	C42B—H42B	0.9500
C4A—C5A	1.460 (7)	C43B—H43B	0.9500
C13A—C14A	1.388 (8)	C45B—H45B	0.9500
C14A—C15A	1.381 (8)	C46B—H46B	0.9500
Br1···Br2^{i}	3.7421 (9)	C5B···N2A	3.347 (7)
Br2···C15B^{ii}	3.746 (6)	C11B···C45A^{x}	3.527 (8)
Br2···Br1^{iii}	3.7421 (9)	C11B···C44A^{x}	3.578 (7)
Br2···C3B^{iv}	3.733 (6)	C11B···N12B^{v}	3.319 (7)
Br3···C31B^{v}	3.740 (6)	C13A···C42A^{ix}	3.405 (8)
Br3···N6B^{vi}	3.536 (5)	C13A···N12B^{v}	3.193 (7)
Br3···C46B^{vi}	3.659 (6)	C13B···C41A^{x}	3.467 (8)
Br4···C45B^{vii}	3.721 (6)	C13B···N2B^{v}	3.244 (7)
Br4···C43A	3.644 (6)	C13B···N1B^{v}	3.358 (7)
Br1···H31D^{v}	2.9200	C13B···C46A^{x}	3.361 (8)
Br2···H15B^{ii}	2.8700	C13B···N12A^{v}	3.245 (7)
Br3···H6B^{vi}	3.0900	C14B···C46A^{x}	3.438 (8)
Br3···H46B^{vi}	3.2000	C15B···O5B^{vi}	3.219 (7)
Br3···H31E^{v}	3.1600	C15B···Br2^{ii}	3.746 (6)

Br3···H31A^{v}	2.8800	C31A···C5A^{ix}	3.493 (8)
Br4···H31E^{viii}	3.1000	C31A···N5B	3.323 (8)
Br4···H15A^{ii}	2.9200	C31B···Br3^{v}	3.740 (6)
O5A···N5A	3.044 (6)	C41A···C45B	3.425 (8)
O5A···C46B^{ii}	3.375 (7)	C41A···C13B^{iv}	3.467 (8)
O5A···N6A	2.821 (6)	C41B···C4A	3.555 (8)
O5A···N16A	2.917 (6)	C42A···C13A^{ix}	3.405 (8)
O5B···N16B	2.857 (6)	C42A···C45B	3.569 (8)
O5B···N5B	3.022 (6)	C43A···Br4	3.644 (6)
O5B···N6B	2.782 (6)	C44A···C11B^{iv}	3.578 (7)
O5B···C15B^{vi}	3.219 (7)	C45A···C11B^{iv}	3.527 (8)
O5B···N1A	3.220 (6)	C45B···Br4^{vii}	3.721 (6)
O5B···C46A^{ii}	3.344 (7)	C45B···C41A	3.425 (8)
O5A···H46B^{ii}	2.6800	C45B···C42A	3.569 (8)
O5A···H6A	2.1400	C46A···C13B^{iv}	3.361 (8)
O5A···H31B^{ix}	2.8400	C46A···C14B^{iv}	3.438 (8)
O5B···H6B	2.1000	C46A···O5B^{ii}	3.344 (7)
O5B···H46A^{ii}	2.6500	C46B···N5A	3.250 (7)
O5B···H15B^{vi}	2.8200	C46B···O5A^{ii}	3.375 (7)
N1A···O5B	3.220 (6)	C46B···N6A	3.372 (7)
N1B···C13B^{v}	3.358 (7)	C46B···C4A	3.573 (7)
N2A···N12A	2.698 (6)	C46B···Br3^{vi}	3.659 (6)
N2A···C5B	3.347 (7)	C5A···H6A	2.4900
N2A···C4A^{ix}	3.402 (7)	C5A···H31B^{ix}	2.8700
N2B···N12B	2.667 (6)	C5B···H6B	2.4700
N2B···C13B^{v}	3.244 (7)	C15A···H45B^{ii}	3.0400
N5A···C46B	3.250 (7)	C15B···H45A^{ii}	3.0400
N5A···O5A	3.044 (6)	C31A···H43B^{viii}	3.0100
N5B···O5B	3.022 (6)	C42B···H31C	3.0500
N5B···C31A	3.323 (8)	H6A···C5A	2.4900
N5B···C3A	3.411 (7)	H6A···H46A	2.3900
N6A···C46B	3.372 (7)	H6A···O5A	2.1400
N6A···O5A	2.821 (6)	H6B···H46B	2.3900
N6B···C4A	3.404 (7)	H6B···O5B	2.1000
N6B···Br3^{vi}	3.536 (5)	H6B···C5B	2.4700
N6B···C3A	3.233 (7)	H6B···Br3^{vi}	3.0900
N6B···O5B	2.782 (6)	H13A···N2B^{v}	2.6800
N12A···C13B^{v}	3.245 (7)	H13A···N12B^{v}	2.6100
N12A···N12B^{v}	3.188 (6)	H13B···N12A^{v}	2.6400
N12A···N2A	2.698 (6)	H13B···N2A^{v}	2.6700
N12B···N2B	2.667 (6)	H15A···Br4^{ii}	2.9200
N12B···C13A^{v}	3.193 (7)	H15B···O5B^{vi}	2.8200
N12B···C11B^{v}	3.319 (7)	H15B···Br2^{ii}	2.8700

N12B···N12A^{v}	3.188 (6)	H31A···Br3^{v}	2.8800
N12B···N12B^{v}	3.120 (6)	H31B···N16A^{ix}	2.6500
N16A···O5A	2.917 (6)	H31B···C5A^{ix}	2.8700
N16B···O5B	2.857 (6)	H31B···O5A^{ix}	2.8400
N2A···H13B^{v}	2.6700	H31C···N5B	2.7700
N2B···H13A^{v}	2.6800	H31C···C42B	3.0500
N5A···H42A	2.4800	H31C···H43B^{viii}	2.3900
N5B···H42B	2.4900	H31D···Br1^{v}	2.9200
N5B···H31C	2.7700	H31E···Br4^{viii}	3.1000
N12A···H13B^{v}	2.6400	H31E···Br3^{v}	3.1600
N12B···H13A^{v}	2.6100	H31F···H43A^{viii}	2.4900
N16A···H45B^{ii}	2.6300	H42A···N5A	2.4800
N16A···H31B^{ix}	2.6500	H42B···N5B	2.4900
N16B···H45A^{ii}	2.6200	H43A···H31F^{viii}	2.4900
C3A···N5B	3.411 (7)	H43B···H31C^{viii}	2.3900
C3A···C4A^{ix}	3.436 (7)	H43B···C31A^{viii}	3.0100
C3A···C5A^{ix}	3.414 (8)	H45A···C15B^{ii}	3.0400
C3A···N6B	3.233 (7)	H45A···N16B^{ii}	2.6200
C3B···Br2^{x}	3.733 (6)	H45B···C15A^{ii}	3.0400
C4A···N6B	3.404 (7)	H45B···N16A^{ii}	2.6300
C4A···N2A^{ix}	3.402 (7)	H46A···O5B^{ii}	2.6500
C4A···C41B	3.555 (8)	H46A···H6A	2.3900
C4A···C46B	3.573 (7)	H46B···H6B	2.3900
C4A···C3A^{ix}	3.436 (7)	H46B···Br3^{vi}	3.2000
C5A···C31A^{ix}	3.493 (8)	H46B···O5A^{ii}	2.6800
C5A···C3A^{ix}	3.414 (8)		
N2A—N1A—C5A	112.8 (4)	H31B—C31A—H31C	109.00
N2A—N1A—C11A	117.8 (4)	H31A—C31A—H31C	109.00
C5A—N1A—C11A	129.2 (5)	C43A—C42A—H42A	120.00
N1A—N2A—C3A	106.9 (4)	C41A—C42A—H42A	121.00
N6A—N5A—C4A	118.1 (5)	C42A—C43A—H43A	120.00
N5A—N6A—C41A	119.7 (5)	C44A—C43A—H43A	120.00
C11A—N12A—C13A	116.3 (5)	C44A—C45A—H45A	121.00
C11A—N16A—C15A	115.1 (5)	C46A—C45A—H45A	121.00
C41A—N6A—H6A	120.00	C41A—C46A—H46A	120.00
N5A—N6A—H6A	120.00	C45A—C46A—H46A	120.00
N2B—N1B—C5B	112.4 (4)	N2B—C3B—C31B	122.6 (5)
C5B—N1B—C11B	129.1 (5)	N2B—C3B—C4B	110.7 (5)
N2B—N1B—C11B	118.2 (4)	C4B—C3B—C31B	126.7 (5)
N1B—N2B—C3B	107.2 (4)	N5B—C4B—C5B	128.2 (5)
N6B—N5B—C4B	117.4 (5)	N5B—C4B—C3B	124.6 (5)
N5B—N6B—C41B	119.9 (5)	C3B—C4B—C5B	107.2 (5)
C11B—N12B—C13B	115.0 (5)	O5B—C5B—C4B	128.0 (5)
C11B—N16B—C15B	114.9 (5)	N1B—C5B—C4B	102.5 (5)

C41B—N6B—H6B	120.00	O5B—C5B—N1B	129.5 (5)
N5B—N6B—H6B	120.00	N12B—C11B—N16B	128.5 (5)
N2A—C3A—C31A	122.8 (5)	N1B—C11B—N12B	115.7 (5)
C4A—C3A—C31A	126.4 (5)	N1B—C11B—N16B	115.8 (5)
N2A—C3A—C4A	110.8 (5)	N12B—C13B—C14B	121.8 (5)
C3A—C4A—C5A	106.9 (5)	C13B—C14B—C15B	117.9 (5)
N5A—C4A—C5A	128.3 (5)	Br3—C14B—C15B	120.7 (4)
N5A—C4A—C3A	124.9 (5)	Br3—C14B—C13B	121.4 (4)
O5A—C5A—C4A	128.5 (5)	N16B—C15B—C14B	121.9 (6)
N1A—C5A—C4A	102.7 (4)	C42B—C41B—C46B	120.3 (5)
O5A—C5A—N1A	128.9 (5)	N6B—C41B—C42B	120.8 (5)
N1A—C11A—N16A	116.1 (5)	N6B—C41B—C46B	118.9 (5)
N12A—C11A—N16A	127.8 (5)	C41B—C42B—C43B	119.5 (6)
N1A—C11A—N12A	116.2 (5)	C42B—C43B—C44B	118.9 (6)
N12A—C13A—C14A	120.5 (5)	Br4—C44B—C45B	120.0 (4)
Br1—C14A—C15A	120.0 (4)	Br4—C44B—C43B	117.7 (4)
C13A—C14A—C15A	118.2 (5)	C43B—C44B—C45B	122.4 (5)
Br1—C14A—C13A	121.8 (4)	C44B—C45B—C46B	118.2 (5)
N16A—C15A—C14A	122.0 (5)	C41B—C46B—C45B	120.7 (5)
C42A—C41A—C46A	121.2 (5)	N12B—C13B—H13B	119.00
N6A—C41A—C46A	118.3 (5)	C14B—C13B—H13B	119.00
N6A—C41A—C42A	120.5 (5)	N16B—C15B—H15B	119.00
C41A—C42A—C43A	119.0 (5)	C14B—C15B—H15B	119.00
C42A—C43A—C44A	119.4 (5)	C3B—C31B—H31D	109.00
Br2—C44A—C43A	117.7 (4)	C3B—C31B—H31E	109.00
Br2—C44A—C45A	120.3 (4)	C3B—C31B—H31F	109.00
C43A—C44A—C45A	122.0 (5)	H31D—C31B—H31E	109.00
C44A—C45A—C46A	118.6 (5)	H31D—C31B—H31F	109.00
C41A—C46A—C45A	119.8 (5)	H31E—C31B—H31F	109.00
N12A—C13A—H13A	120.00	C41B—C42B—H42B	120.00
C14A—C13A—H13A	120.00	C43B—C42B—H42B	120.00
N16A—C15A—H15A	119.00	C42B—C43B—H43B	121.00
C14A—C15A—H15A	119.00	C44B—C43B—H43B	121.00
C3A—C31A—H31A	109.00	C44B—C45B—H45B	121.00
H31A—C31A—H31B	109.00	C46B—C45B—H45B	121.00
C3A—C31A—H31B	109.00	C41B—C46B—H46B	120.00
C3A—C31A—H31C	109.00	C45B—C46B—H46B	120.00
C5A—N1A—N2A—C3A	−1.5 (6)	C15B—N16B—C11B—N1B	−176.9 (5)
C11A—N1A—N2A—C3A	173.3 (5)	N2A—C3A—C4A—N5A	−176.9 (5)
N2A—N1A—C5A—O5A	−175.7 (5)	N2A—C3A—C4A—C5A	1.9 (6)
N2A—N1A—C5A—C4A	2.5 (6)	C31A—C3A—C4A—N5A	3.2 (9)
C11A—N1A—C5A—O5A	10.3 (9)	C31A—C3A—C4A—C5A	−178.0 (5)
C11A—N1A—C5A—C4A	−171.5 (5)	N5A—C4A—C5A—O5A	−5.6 (10)
N2A—N1A—C11A—N12A	22.2 (7)	N5A—C4A—C5A—N1A	176.2 (5)
N2A—N1A—C11A—N16A	−156.5 (5)	C3A—C4A—C5A—O5A	175.7 (6)
C5A—N1A—C11A—N12A	−164.1 (5)	C3A—C4A—C5A—N1A	−2.6 (6)
C5A—N1A—C11A—N16A	17.2 (8)	N12A—C13A—C14A—Br1	178.0 (4)
N1A—N2A—C3A—C4A	−0.4 (6)	N12A—C13A—C14A—C15A	−0.3 (8)
N1A—N2A—C3A—C31A	179.6 (5)	C13A—C14A—C15A—N16A	1.6 (9)

C4A—N5A—N6A—C41A	179.7 (5)	Br1—C14A—C15A—N16A	−176.7 (4)
N6A—N5A—C4A—C3A	179.9 (5)	N6A—C41A—C46A—C45A	178.5 (5)
N6A—N5A—C4A—C5A	1.3 (8)	N6A—C41A—C42A—C43A	−178.8 (5)
N5A—N6A—C41A—C42A	2.3 (8)	C42A—C41A—C46A—C45A	−0.9 (9)
N5A—N6A—C41A—C46A	−177.0 (5)	C46A—C41A—C42A—C43A	0.5 (9)
C13A—N12A—C11A—N1A	−175.8 (5)	C41A—C42A—C43A—C44A	0.7 (9)
C13A—N12A—C11A—N16A	2.8 (8)	C42A—C43A—C44A—Br2	176.1 (4)
C11A—N12A—C13A—C14A	−1.7 (8)	C42A—C43A—C44A—C45A	−1.6 (9)
C15A—N16A—C11A—N1A	177.0 (5)	C43A—C44A—C45A—C46A	1.2 (9)
C15A—N16A—C11A—N12A	−1.5 (9)	Br2—C44A—C45A—C46A	−176.4 (4)
C11A—N16A—C15A—C14A	−0.8 (8)	C44A—C45A—C46A—C41A	0.0 (8)
C5B—N1B—C11B—N16B	−1.4 (8)	N2B—C3B—C4B—N5B	178.8 (5)
C11B—N1B—N2B—C3B	−173.3 (5)	N2B—C3B—C4B—C5B	0.8 (7)
N2B—N1B—C5B—O5B	−179.6 (6)	C31B—C3B—C4B—N5B	0.1 (9)
N2B—N1B—C5B—C4B	−0.4 (6)	C31B—C3B—C4B—C5B	−178.0 (5)
C11B—N1B—C5B—O5B	−6.2 (10)	N5B—C4B—C5B—O5B	1.1 (10)
C11B—N1B—C5B—C4B	173.0 (5)	N5B—C4B—C5B—N1B	−178.2 (6)
N2B—N1B—C11B—N12B	−6.1 (7)	C3B—C4B—C5B—O5B	179.1 (6)
C5B—N1B—N2B—C3B	0.9 (6)	C3B—C4B—C5B—N1B	−0.2 (6)
C5B—N1B—C11B—N12B	−179.2 (5)	N12B—C13B—C14B—Br3	−177.8 (4)
N2B—N1B—C11B—N16B	171.7 (5)	N12B—C13B—C14B—C15B	0.1 (8)
N1B—N2B—C3B—C31B	177.8 (5)	Br3—C14B—C15B—N16B	179.2 (4)
N1B—N2B—C3B—C4B	−1.0 (6)	C13B—C14B—C15B—N16B	1.3 (9)
C4B—N5B—N6B—C41B	179.7 (5)	N6B—C41B—C42B—C43B	178.5 (5)
N6B—N5B—C4B—C3B	−178.9 (5)	C46B—C41B—C42B—C43B	−0.6 (9)
N6B—N5B—C4B—C5B	−1.3 (9)	N6B—C41B—C46B—C45B	−178.5 (5)
N5B—N6B—C41B—C42B	−1.3 (8)	C42B—C41B—C46B—C45B	0.6 (9)
N5B—N6B—C41B—C46B	177.8 (5)	C41B—C42B—C43B—C44B	0.0 (9)
C13B—N12B—C11B—N16B	0.7 (8)	C42B—C43B—C44B—Br4	−179.5 (5)
C11B—N12B—C13B—C14B	−1.0 (8)	C42B—C43B—C44B—C45B	0.5 (9)
C13B—N12B—C11B—N1B	178.1 (5)	Br4—C44B—C45B—C46B	179.5 (4)
C15B—N16B—C11B—N12B	0.6 (8)	C43B—C44B—C45B—C46B	−0.5 (9)
C11B—N16B—C15B—C14B	−1.6 (8)	C44B—C45B—C46B—C41B	−0.1 (9)

Symmetry codes: (i) *x*, *y*−1, *z*−1; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) *x*, *y*+1, *z*+1; (iv) *x*, *y*, *z*+1; (v) −*x*, −*y*+1, −*z*; (vi) −*x*+1, −*y*+1, −*z*; (vii) −*x*+1, −*y*+2, −*z*+1; (viii) −*x*, −*y*+2, −*z*+1; (ix) −*x*, −*y*+1, −*z*+1; (x) *x*, *y*, *z*−1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6A—H6A···O5A	0.88	2.14	2.821 (6)	134
N6B—H6B···O5B	0.88	2.10	2.782 (6)	134
C13A—H13A···N12B^{v}	0.95	2.61	3.193 (7)	120
C15A—H15A···Br4^{ii}	0.95	2.92	3.761 (6)	148
C15B—H15B···Br2^{ii}	0.95	2.87	3.746 (6)	153
C31A—H31A···Br3^{v}	0.98	2.88	3.858 (6)	173
C31B—H31D···Br1^{v}	0.98	2.92	3.862 (6)	160
C45A—H45A···N16B^{ii}	0.95	2.62	3.539 (7)	162

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

Fig. 1

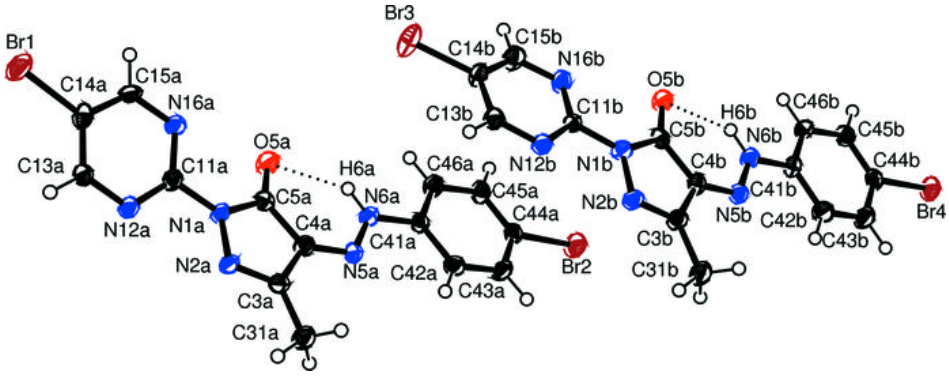

Br1
C14a
C15a
N16a
C13a
C11a
O5a
H6a
C5a
N6a
C4a
N12a
N1a
N2a
C41a
N5a
C3a
C31a
C42a
C43a
C46a
C45a
C44a
Br2
Br3
C14b
C15b
N16b
C13b
C11b
N12b
N1b
O5b
C5b
H6b
N6b
C4b
N2b
C3b
C31b
C41b
N5b
C42b
C43b
C46b
C45b
C44b
Br4

Fig. 2