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# 5-Bromo-2-[5-(4-nitrophenyl)-3-phenyl-4,5-dihydro-1*H*-pyrazol-1-yl]pyrimidine

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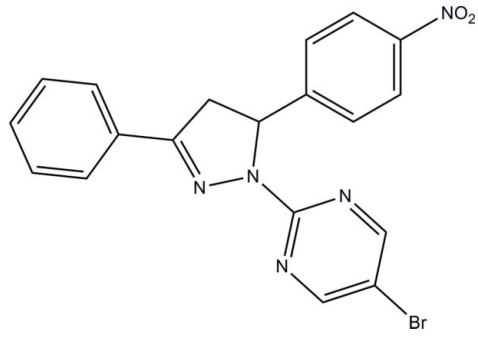
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.024; $wR$ factor = 0.060; data-to-parameter ratio = 12.7.

In the title pyrazoline compound, $C_{19}H_{14}BrN_5O_2$, the essentially planar pyrazoline and pyrimidine rings [maximum deviations = 0.013 (1) and 0.009 (1) Å, respectively] are inclined slightly to one another, making a dihedral angle of 10.81 (10)°. The nitrobenzene unit is almost perpendicular to the attached pyrazoline ring, as indicated by the dihedral angle of 84.61 (8)°. In the crystal structure, intermolecular C—H···N contacts link the molecules into dimers in an antiparallel manner. These dimers are further linked into one-dimensional chains along the *b* axis *via* C—H···O contacts. The crystal structure is consolidated by three different intermolecular $\pi$–$\pi$ interactions [range of centroid–centroid distances = 3.5160 (11)–3.6912 (11) Å].

## Related literature

For general background to and applications of the title compound, see: Hegde *et al.* (2006); Kalluraya & Chimbalkar (2001); Kalluraya *et al.* (2001); Rai *et al.* (2008); Rathish *et al.* (2009); Tawab *et al.* (1960). For closely related structures, see: Goh *et al.* (2009*a*,*b*). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).




## Experimental

### Crystal data

$C_{19}H_{14}BrN_5O_2$
$M_r$ = 424.26
Triclinic, $P\overline{1}$
$a$ = 6.9709 (1) Å
$b$ = 11.6500 (2) Å
$c$ = 12.4365 (2) Å
$\alpha$ = 114.969 (1)°
$\beta$ = 103.303 (1)°
$\gamma$ = 91.560 (1)°
$V$ = 882.12 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 2.36 mm$^{-1}$
$T$ = 100 K
0.33 × 0.22 × 0.12 mm

### Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.510, $T_{max}$ = 0.760
28197 measured reflections
3804 independent reflections
3431 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.026

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.060
$S$ = 1.05
3804 reflections
300 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.49 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.30 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C8—H8*B*···O2$^{i}$ | 0.95 (2) | 2.41 (2) | 3.352 (2) | 176.0 (17) |
| C11—H11*A*···N4$^{ii}$ | 0.92 (2) | 2.56 (2) | 3.431 (2) | 160.5 (18) |
| C19—H19*A*···O2$^{iii}$ | 0.98 (2) | 2.58 (2) | 3.412 (3) | 143.3 (17) |

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2577).

‡ Thomson Reuters ResearcherID: C-7576-2009.
§ Thomson Reuters ResearcherID: A-3561-2009.

## References


Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

Goh, J. H., Fun, H.-K., Nithinchandra, & Kalluraya, B. (2009*a*). *Acta Cryst.* E**65**, o3088–o3089.

Goh, J. H., Fun, H.-K., Nithinchandra, Rai, N. S. & Kalluraya, B. (2009*b*). *Acta Cryst.* E**65**, o3099–o3100.

Hegde, J. C., Rai, G., Puranic, V. G. & Kalluraya, B. (2006). *Synth. Commun.* **36**, 1285–1290.

Kalluraya, B. & Chimbalkar, R. M. (2001). *Indian J. Heterocycl. Chem.* **11**, 171–174.

Kalluraya, B., Chimbalkar, R. M., Rai, G., Gururaja, R. & Shenoy, S. (2001). *J. Indian Council Chemists*, **18**, 1–5.

Rai, N. S., Kalluraya, B., Lingappa, B., Shenoy, S. & Puranic, V. G. (2008). *Eur. J. Med. Chem.* **43**, 1715–1720.

Rathish, I. G., Kalim, J., Shamim, A., Sameena, B., Alam, M. S., Pillai, K. K., Surender, S. & Bagchi, V. (2009). *Bioorg. Med. Chem. Lett.* **19**, 255–258.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Tawab, S. A., Mustafa, A. & Kira, M. (1960). *Nature (London)*, **186**, 165–166.

# supporting information

*Acta Cryst.* (2009). E**65**, o3134–o3135 [doi:10.1107/S1600536809048600]

## 5-Bromo-2-[5-(4-nitrophenyl)-3-phenyl-4,5-dihydro-1*H*-pyrazol-1-yl]pyrimidine

**Jia Hao Goh, Hoong-Kun Fun, Adithya Adhikari and B. Kalluraya**

### S1. Comment

Pyrazoles and pyrazoline derivatives are an important class of heterocyclic compounds (Rai *et al.*, 2008; Hegde *et al.*, 2006). The addition of aliphatic diazo compounds to olefins leads to pyrazolines. Also, the addition of hydrazine or its derivatives to *α*, *β*-unsaturated aldehydes or ketones yields pyrazoline (Kalluraya & Chimbalkar, 2001). Pyrazoline derivatives have been found to possess potential anti-pyretic, analgesic (Tawab *et al.*, 1960), anti-inflammatory (Rathish *et al.*, 2009), and anti-microbial (Kalluraya *et al.*, 2001) properties. In the present work, an X-ray crystal structure analysis has been undertaken in order to determine the 3D chemical structure and also the crystal packing of the molecules. We herein report the synthesis and crystal structure of the title compound, (I).

In (I), Fig. 1, the pyrazoline (C7-C9/N1/N2) and pyrimidine (C16-C19/N3/N4) rings are essentially planar, with maximum deviations of 0.013 (1) and 0.009 (1) Å, respectively, for atoms N1 and C16. These two rings are slightly inclined to one another, making a dihedral angle of 10.81 (10)°. The nitrobenzene moiety is almost perpendicular to the attached pyrazoline ring, as indicated by the dihedral angle formed between the mean plane through C10-C15/N5/O1/O2 and the pyrazoline ring of 84.61 (8)°. The bond lengths and angles are consistent with those closely related structures (Goh *et al.*, 2009*a,b*).

In the crystal structure (Fig. 2), intermolecular C11—H11A···N4 contacts (Table 1) link the molecules into dimers in an anti-parallel manner. These dimers are further linked into a 1-D chain along the *b* axis by intermolecular C8—H8B···O2 and C19—H19A···O2 contacts (Table 1). The crystal structure is consolidated by three different weak *π*–*π* interactions involving the pyrazoline (*Cg*1), pyrimidine (*Cg*2) and C1-C6 benzene (*Cg*3) rings [$Cg1\cdots Cg1^{iv}$ = 3.5160 (11) Å, $Cg2\cdots Cg3^{ii}$ = 3.6912 (11) Å and $Cg2\cdots Cg3^{iv}$ = 3.5779 (11) Å, respectively; (ii) -x, 1-y, 1-z and (iv) 1-x, 1-y, 1-z].

### S2. Experimental

A mixture of 5-bromo-2-hydrazinopyrimidine (0.01 mol) and 3-(4-nitrophenyl)-1-phenyl-prop-2-en-1-one (0.01 mol) was taken in acetic acid (20 ml), and two drops of concentrated $H_2SO_4$ added. The mixture was refluxed for 4 h. The precipitated solids were filtered, dried and recrystallized from ethanol. The single crystals were obtained from a mixture of ethanol and DMF by slow evaporation.

### S3. Refinement

All the H atoms were located from difference Fourier map [range of C—H = 0.91 (2) – 0.995 (19) Å] and allowed to refine freely. The reflections (001) and ($0\bar{1}1$) were omitted as the intensities were affected by the beam-stop.

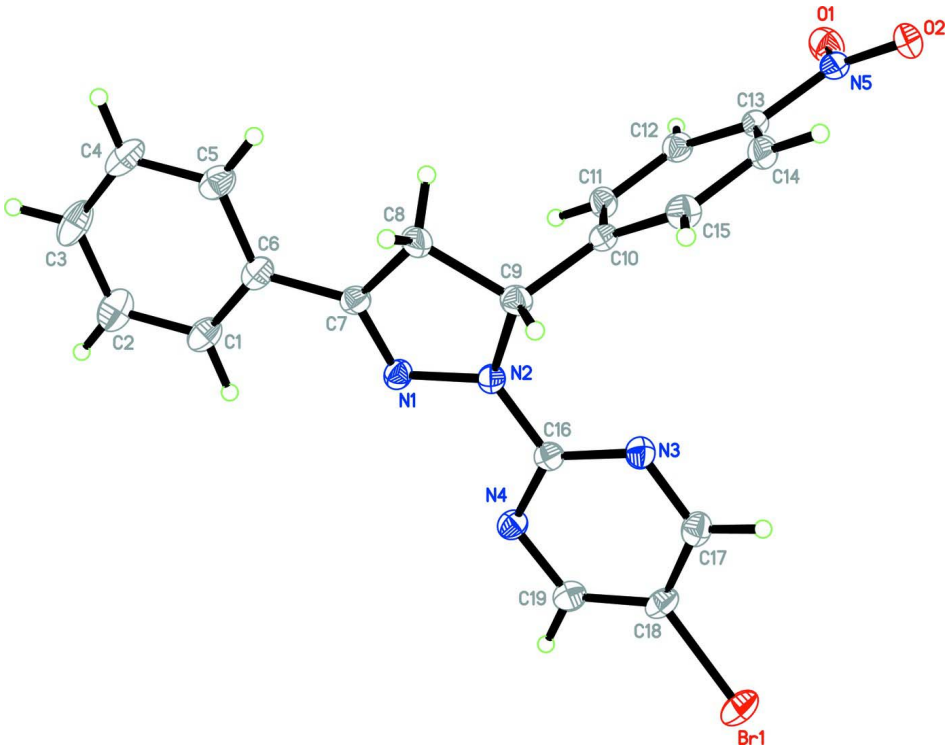


**Figure 1**

The molecular structure of (I), showing 50% probability displacement ellipsoids for non-H atoms and the atom-numbering scheme.

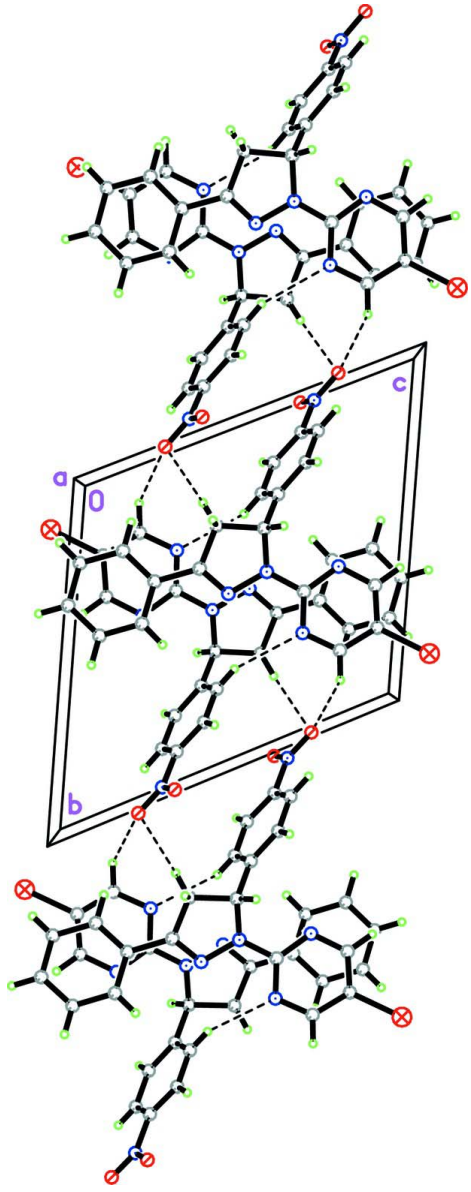


**Figure 2**

Part of the crystal structure of (I), viewed along the *a* axis, showing the dimers being linked into a 1-D chain along the *b* axis. Intermolecular contacts are shown as dashed lines.

**5-Bromo-2-[5-(4-nitrophenyl)-3-phenyl-4,5-dihydro-1*H*- pyrazol-1-yl]pyrimidine**

*Crystal data*

$C_{19}H_{14}BrN_5O_2$
$M_r = 424.26$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.9709$ (1) Å
$b = 11.6500$ (2) Å
$c = 12.4365$ (2) Å
$\alpha = 114.969$ (1)°
$\beta = 103.303$ (1)°
$\gamma = 91.560$ (1)°
$V = 882.12$ (2) Å$^3$
$Z = 2$
$F(000) = 428$
$D_x = 1.597$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9873 reflections
$\theta = 3.0$–33.9°
$\mu = 2.36$ mm$^{-1}$

$T$ = 100 K
Block, green

0.33 × 0.22 × 0.12 mm

*Data collection*

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.510, $T_{max}$ = 0.760

28197 measured reflections
3804 independent reflections
3431 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.026
$\theta_{max}$ = 27.0°, $\theta_{min}$ = 2.0°
$h$ = −8→8
$k$ = −14→14
$l$ = −15→15

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.060
$S$ = 1.05
3804 reflections
300 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0288P)^2 + 0.5594P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.49 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.30 e Å$^{-3}$

*Special details*

**Experimental**. The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1)K.

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Br1 | 0.32778 (3) | 0.891599 (17) | 1.068122 (15) | 0.02770 (7) |
| O1 | −0.3115 (2) | 0.02211 (12) | 0.64236 (12) | 0.0274 (3) |
| O2 | −0.0404 (2) | −0.01117 (12) | 0.74511 (11) | 0.0258 (3) |
| N1 | 0.2287 (2) | 0.49488 (13) | 0.46957 (12) | 0.0175 (3) |
| N2 | 0.2681 (2) | 0.47661 (13) | 0.57546 (12) | 0.0177 (3) |
| N3 | 0.3203 (2) | 0.54232 (13) | 0.78176 (13) | 0.0191 (3) |
| N4 | 0.2364 (2) | 0.68890 (13) | 0.69321 (13) | 0.0182 (3) |
| N5 | −0.1294 (2) | 0.03714 (13) | 0.68034 (13) | 0.0205 (3) |
| C1 | 0.1859 (3) | 0.48220 (17) | 0.22862 (16) | 0.0224 (4) |
| C2 | 0.1604 (3) | 0.4683 (2) | 0.10980 (17) | 0.0282 (4) |
| C3 | 0.1682 (3) | 0.3501 (2) | 0.01564 (17) | 0.0300 (4) |
| C4 | 0.2012 (3) | 0.24641 (19) | 0.04084 (17) | 0.0275 (4) |

| | | | | |
|---|---|---|---|---|
| C5 | 0.2323 (3) | 0.26060 (18) | 0.16071 (16) | 0.0236 (4) |
| C6 | 0.2252 (2) | 0.37903 (16) | 0.25600 (15) | 0.0201 (3) |
| C7 | 0.2607 (2) | 0.39338 (15) | 0.38231 (15) | 0.0176 (3) |
| C8 | 0.3349 (3) | 0.29328 (16) | 0.42148 (17) | 0.0228 (4) |
| C9 | 0.3406 (3) | 0.35347 (16) | 0.55889 (15) | 0.0191 (3) |
| C10 | 0.2129 (3) | 0.27153 (15) | 0.59130 (14) | 0.0175 (3) |
| C11 | 0.0070 (3) | 0.24832 (16) | 0.54497 (15) | 0.0188 (3) |
| C12 | −0.1072 (3) | 0.17094 (16) | 0.57339 (15) | 0.0191 (3) |
| C13 | −0.0101 (3) | 0.11782 (15) | 0.64817 (15) | 0.0178 (3) |
| C14 | 0.1942 (3) | 0.13677 (16) | 0.69325 (16) | 0.0211 (3) |
| C15 | 0.3060 (3) | 0.21486 (16) | 0.66433 (16) | 0.0213 (3) |
| C16 | 0.2747 (2) | 0.57397 (15) | 0.68747 (15) | 0.0165 (3) |
| C17 | 0.3326 (3) | 0.63670 (17) | 0.89244 (16) | 0.0207 (3) |
| C18 | 0.2999 (3) | 0.75881 (16) | 0.90934 (15) | 0.0204 (3) |
| C19 | 0.2496 (3) | 0.78065 (16) | 0.80562 (16) | 0.0201 (3) |
| H1A | 0.177 (3) | 0.563 (2) | 0.2909 (19) | 0.025 (5)* |
| H2A | 0.138 (4) | 0.540 (2) | 0.092 (2) | 0.038 (6)* |
| H3A | 0.154 (3) | 0.340 (2) | −0.066 (2) | 0.037 (6)* |
| H4A | 0.206 (3) | 0.169 (2) | −0.020 (2) | 0.024 (5)* |
| H5A | 0.257 (3) | 0.190 (2) | 0.178 (2) | 0.028 (5)* |
| H8A | 0.467 (3) | 0.2771 (19) | 0.4110 (19) | 0.024 (5)* |
| H8B | 0.250 (3) | 0.214 (2) | 0.378 (2) | 0.031 (6)* |
| H9A | 0.478 (3) | 0.3691 (17) | 0.6123 (17) | 0.013 (4)* |
| H11A | −0.055 (3) | 0.2853 (18) | 0.4970 (18) | 0.019 (5)* |
| H12A | −0.245 (3) | 0.1549 (19) | 0.5429 (19) | 0.024 (5)* |
| H14A | 0.258 (3) | 0.0991 (19) | 0.7416 (19) | 0.025 (5)* |
| H15A | 0.446 (3) | 0.2291 (19) | 0.6952 (19) | 0.025 (5)* |
| H17A | 0.372 (3) | 0.6149 (19) | 0.9598 (19) | 0.022 (5)* |
| H19A | 0.224 (3) | 0.8658 (19) | 0.8141 (18) | 0.019 (5)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Br1 | 0.02884 (11) | 0.02882 (11) | 0.01659 (9) | 0.00509 (7) | 0.00679 (7) | 0.00134 (7) |
| O1 | 0.0263 (7) | 0.0275 (7) | 0.0323 (7) | 0.0020 (5) | 0.0112 (6) | 0.0154 (6) |
| O2 | 0.0386 (8) | 0.0213 (6) | 0.0223 (6) | 0.0044 (5) | 0.0086 (6) | 0.0139 (5) |
| N1 | 0.0171 (7) | 0.0197 (7) | 0.0145 (6) | 0.0012 (5) | 0.0029 (5) | 0.0070 (5) |
| N2 | 0.0231 (8) | 0.0155 (6) | 0.0160 (7) | 0.0037 (5) | 0.0070 (6) | 0.0073 (5) |
| N3 | 0.0199 (7) | 0.0202 (7) | 0.0180 (7) | 0.0023 (6) | 0.0060 (6) | 0.0087 (6) |
| N4 | 0.0185 (7) | 0.0179 (7) | 0.0174 (7) | 0.0043 (5) | 0.0047 (6) | 0.0068 (6) |
| N5 | 0.0302 (9) | 0.0147 (6) | 0.0173 (7) | 0.0036 (6) | 0.0107 (6) | 0.0055 (6) |
| C1 | 0.0190 (9) | 0.0260 (9) | 0.0173 (8) | 0.0058 (7) | 0.0023 (7) | 0.0061 (7) |
| C2 | 0.0244 (10) | 0.0390 (11) | 0.0209 (9) | 0.0122 (8) | 0.0031 (7) | 0.0140 (8) |
| C3 | 0.0235 (10) | 0.0459 (12) | 0.0152 (8) | 0.0112 (8) | 0.0028 (7) | 0.0091 (8) |
| C4 | 0.0202 (9) | 0.0321 (10) | 0.0173 (8) | 0.0036 (8) | 0.0049 (7) | −0.0012 (8) |
| C5 | 0.0185 (9) | 0.0247 (9) | 0.0219 (9) | 0.0023 (7) | 0.0063 (7) | 0.0045 (7) |
| C6 | 0.0137 (8) | 0.0250 (9) | 0.0169 (8) | 0.0010 (6) | 0.0040 (6) | 0.0049 (7) |
| C7 | 0.0149 (8) | 0.0172 (7) | 0.0179 (8) | −0.0004 (6) | 0.0050 (6) | 0.0051 (6) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C8 | 0.0315 (10) | 0.0164 (8) | 0.0246 (9) | 0.0043 (7) | 0.0156 (8) | 0.0086 (7) |
| C9 | 0.0212 (9) | 0.0161 (7) | 0.0209 (8) | 0.0036 (6) | 0.0081 (7) | 0.0078 (6) |
| C10 | 0.0236 (9) | 0.0140 (7) | 0.0149 (7) | 0.0033 (6) | 0.0077 (6) | 0.0050 (6) |
| C11 | 0.0225 (9) | 0.0184 (8) | 0.0179 (8) | 0.0055 (7) | 0.0056 (7) | 0.0099 (7) |
| C12 | 0.0204 (9) | 0.0187 (8) | 0.0183 (8) | 0.0046 (7) | 0.0060 (7) | 0.0075 (7) |
| C13 | 0.0252 (9) | 0.0137 (7) | 0.0153 (7) | 0.0028 (6) | 0.0085 (7) | 0.0057 (6) |
| C14 | 0.0278 (10) | 0.0197 (8) | 0.0179 (8) | 0.0066 (7) | 0.0054 (7) | 0.0103 (7) |
| C15 | 0.0194 (9) | 0.0216 (8) | 0.0212 (8) | 0.0032 (7) | 0.0030 (7) | 0.0092 (7) |
| C16 | 0.0136 (8) | 0.0174 (8) | 0.0173 (8) | 0.0013 (6) | 0.0048 (6) | 0.0064 (6) |
| C17 | 0.0191 (9) | 0.0255 (9) | 0.0180 (8) | 0.0028 (7) | 0.0059 (7) | 0.0095 (7) |
| C18 | 0.0176 (8) | 0.0222 (8) | 0.0152 (8) | 0.0020 (7) | 0.0049 (6) | 0.0023 (7) |
| C19 | 0.0187 (9) | 0.0192 (8) | 0.0204 (8) | 0.0046 (7) | 0.0051 (7) | 0.0067 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Br1—C18 | 1.8863 (16) | C5—H5A | 0.94 (2) |
| O1—N5 | 1.228 (2) | C6—C7 | 1.468 (2) |
| O2—N5 | 1.2337 (19) | C7—C8 | 1.504 (2) |
| N1—C7 | 1.292 (2) | C8—C9 | 1.539 (2) |
| N1—N2 | 1.3886 (19) | C8—H8A | 0.97 (2) |
| N2—C16 | 1.365 (2) | C8—H8B | 0.95 (2) |
| N2—C9 | 1.481 (2) | C9—C10 | 1.519 (2) |
| N3—C17 | 1.332 (2) | C9—H9A | 0.995 (19) |
| N3—C16 | 1.346 (2) | C10—C11 | 1.391 (2) |
| N4—C19 | 1.334 (2) | C10—C15 | 1.393 (2) |
| N4—C16 | 1.348 (2) | C11—C12 | 1.389 (2) |
| N5—C13 | 1.471 (2) | C11—H11A | 0.91 (2) |
| C1—C2 | 1.384 (3) | C12—C13 | 1.387 (2) |
| C1—C6 | 1.399 (3) | C12—H12A | 0.93 (2) |
| C1—H1A | 0.95 (2) | C13—C14 | 1.379 (3) |
| C2—C3 | 1.394 (3) | C14—C15 | 1.390 (2) |
| C2—H2A | 0.96 (2) | C14—H14A | 0.93 (2) |
| C3—C4 | 1.383 (3) | C15—H15A | 0.94 (2) |
| C3—H3A | 0.95 (2) | C17—C18 | 1.381 (3) |
| C4—C5 | 1.392 (3) | C17—H17A | 0.96 (2) |
| C4—H4A | 0.91 (2) | C18—C19 | 1.388 (2) |
| C5—C6 | 1.400 (2) | C19—H19A | 0.98 (2) |
| | | | |
| C7—N1—N2 | 107.71 (14) | N2—C9—C10 | 113.23 (14) |
| C16—N2—N1 | 121.50 (13) | N2—C9—C8 | 101.32 (13) |
| C16—N2—C9 | 123.73 (14) | C10—C9—C8 | 113.11 (14) |
| N1—N2—C9 | 113.65 (13) | N2—C9—H9A | 110.2 (10) |
| C17—N3—C16 | 115.56 (15) | C10—C9—H9A | 107.2 (10) |
| C19—N4—C16 | 115.45 (14) | C8—C9—H9A | 111.9 (10) |
| O1—N5—O2 | 123.49 (14) | C11—C10—C15 | 119.99 (15) |
| O1—N5—C13 | 118.57 (14) | C11—C10—C9 | 121.01 (15) |
| O2—N5—C13 | 117.94 (15) | C15—C10—C9 | 118.95 (15) |
| C2—C1—C6 | 120.44 (17) | C12—C11—C10 | 120.27 (16) |

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| C2—C1—H1A | 118.6 (12) | C12—C11—H11A | 119.2 (12) |
| C6—C1—H1A | 121.0 (12) | C10—C11—H11A | 120.5 (12) |
| C1—C2—C3 | 120.15 (19) | C13—C12—C11 | 118.28 (16) |
| C1—C2—H2A | 120.0 (14) | C13—C12—H12A | 120.8 (12) |
| C3—C2—H2A | 119.8 (14) | C11—C12—H12A | 121.0 (12) |
| C4—C3—C2 | 119.95 (17) | C14—C13—C12 | 122.77 (15) |
| C4—C3—H3A | 119.4 (14) | C14—C13—N5 | 118.41 (15) |
| C2—C3—H3A | 120.6 (14) | C12—C13—N5 | 118.82 (15) |
| C3—C4—C5 | 120.16 (17) | C13—C14—C15 | 118.17 (16) |
| C3—C4—H4A | 120.9 (13) | C13—C14—H14A | 122.0 (13) |
| C5—C4—H4A | 118.9 (13) | C15—C14—H14A | 119.8 (13) |
| C4—C5—C6 | 120.28 (18) | C14—C15—C10 | 120.49 (17) |
| C4—C5—H5A | 120.2 (13) | C14—C15—H15A | 119.0 (12) |
| C6—C5—H5A | 119.5 (13) | C10—C15—H15A | 120.5 (12) |
| C1—C6—C5 | 118.96 (16) | N3—C16—N4 | 127.09 (15) |
| C1—C6—C7 | 121.19 (15) | N3—C16—N2 | 114.49 (14) |
| C5—C6—C7 | 119.85 (16) | N4—C16—N2 | 118.43 (14) |
| N1—C7—C6 | 121.79 (16) | N3—C17—C18 | 122.24 (16) |
| N1—C7—C8 | 114.40 (15) | N3—C17—H17A | 115.3 (12) |
| C6—C7—C8 | 123.81 (15) | C18—C17—H17A | 122.4 (12) |
| C7—C8—C9 | 102.87 (13) | C17—C18—C19 | 117.58 (15) |
| C7—C8—H8A | 112.2 (12) | C17—C18—Br1 | 121.08 (13) |
| C9—C8—H8A | 110.4 (12) | C19—C18—Br1 | 121.33 (13) |
| C7—C8—H8B | 112.8 (13) | N4—C19—C18 | 122.05 (16) |
| C9—C8—H8B | 111.1 (13) | N4—C19—H19A | 118.1 (11) |
| H8A—C8—H8B | 107.5 (18) | C18—C19—H19A | 119.8 (11) |

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| C7—N1—N2—C16 | 170.72 (14) | C15—C10—C11—C12 | 1.4 (2) |
| C7—N1—N2—C9 | 2.42 (18) | C9—C10—C11—C12 | 178.62 (15) |
| C6—C1—C2—C3 | 1.8 (3) | C10—C11—C12—C13 | −0.2 (2) |
| C1—C2—C3—C4 | 0.1 (3) | C11—C12—C13—C14 | −1.2 (2) |
| C2—C3—C4—C5 | −1.8 (3) | C11—C12—C13—N5 | 179.18 (14) |
| C3—C4—C5—C6 | 1.6 (3) | O1—N5—C13—C14 | 178.59 (15) |
| C2—C1—C6—C5 | −1.9 (3) | O2—N5—C13—C14 | −0.9 (2) |
| C2—C1—C6—C7 | 177.49 (16) | O1—N5—C13—C12 | −1.8 (2) |
| C4—C5—C6—C1 | 0.2 (3) | O2—N5—C13—C12 | 178.73 (14) |
| C4—C5—C6—C7 | −179.20 (16) | C12—C13—C14—C15 | 1.5 (3) |
| N2—N1—C7—C6 | 177.49 (14) | N5—C13—C14—C15 | −178.92 (15) |
| N2—N1—C7—C8 | −2.15 (19) | C13—C14—C15—C10 | −0.3 (3) |
| C1—C6—C7—N1 | 10.4 (2) | C11—C10—C15—C14 | −1.1 (2) |
| C5—C6—C7—N1 | −170.23 (16) | C9—C10—C15—C14 | −178.41 (15) |
| C1—C6—C7—C8 | −170.01 (16) | C17—N3—C16—N4 | −1.6 (2) |
| C5—C6—C7—C8 | 9.4 (2) | C17—N3—C16—N2 | 178.34 (14) |
| N1—C7—C8—C9 | 1.11 (19) | C19—N4—C16—N3 | 1.4 (2) |
| C6—C7—C8—C9 | −178.52 (15) | C19—N4—C16—N2 | −178.54 (14) |
| C16—N2—C9—C10 | 68.9 (2) | N1—N2—C16—N3 | −178.10 (14) |
| N1—N2—C9—C10 | −123.07 (15) | C9—N2—C16—N3 | −11.0 (2) |
| C16—N2—C9—C8 | −169.64 (15) | N1—N2—C16—N4 | 1.8 (2) |

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|---|---|---|---|
| N1—N2—C9—C8 | −1.64 (17) | C9—N2—C16—N4 | 168.92 (14) |
| C7—C8—C9—N2 | 0.35 (16) | C16—N3—C17—C18 | 0.3 (2) |
| C7—C8—C9—C10 | 121.86 (15) | N3—C17—C18—C19 | 1.0 (3) |
| N2—C9—C10—C11 | 49.0 (2) | N3—C17—C18—Br1 | −177.75 (12) |
| C8—C9—C10—C11 | −65.5 (2) | C16—N4—C19—C18 | 0.1 (2) |
| N2—C9—C10—C15 | −133.70 (16) | C17—C18—C19—N4 | −1.2 (3) |
| C8—C9—C10—C15 | 111.76 (18) | Br1—C18—C19—N4 | 177.53 (13) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C8—H8*B*···O2$^{i}$ | 0.95 (2) | 2.41 (2) | 3.352 (2) | 176.0 (17) |
| C11—H11*A*···N4$^{ii}$ | 0.92 (2) | 2.56 (2) | 3.431 (2) | 160.5 (18) |
| C19—H19*A*···O2$^{iii}$ | 0.98 (2) | 2.58 (2) | 3.412 (3) | 143.3 (17) |

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