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# 1-Allyl-4-(1,3-benzothiazol-2-yl)-5-methyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

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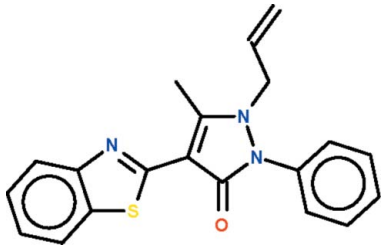
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.004 Å; disorder in main residue; $R$ factor = 0.050; $wR$ factor = 0.172; data-to-parameter ratio = 16.2.

The title compound, $C_{20}H_{17}N_3OS$, is a 1*H*-pyrazol-3(2*H*)-one having aromatic 4-(1,3-benzothiazol-2-yl)- and 2-phenyl substituents. The five-membered ring and fused ring system are planar, the r.m.s. deviations being 0.021 and 0.005 Å, respectively. The five-membered ring is aligned at 7.9 (2)° with respect to the fused-ring system. The allyl and phenyl parts of the molecule are both disordered over two positions in a 1:1 ratio. Weak intermolecular C—H···O hydrogen bonding is present in the crystal structure.

## Related literature

For the structure of a related compound (*E*)-4-(2,3-dihydro-1,3-benzothiazol-2-ylidene)-3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one, see: Chakibe *et al.* (2010).




## Experimental

### Crystal data

$C_{20}H_{17}N_3OS$
$M_r$ = 347.43
Orthorhombic, *Pbca*
$a$ = 17.8734 (5) Å
$b$ = 10.4297 (2) Å
$c$ = 18.9578 (4) Å
$V$ = 3534.00 (14) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.20 mm$^{-1}$
$T$ = 293 K
0.30 × 0.30 × 0.25 mm

### Data collection

Bruker X8 APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.944, $T_{max}$ = 0.953
18349 measured reflections
3678 independent reflections
2341 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.172
$S$ = 1.00
3678 reflections
227 parameters
17 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.25 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C2—H2···O1$^{i}$ | 0.93 | 2.59 | 3.318 (3) | 135 |
| C12—H12$A$···O1$^{ii}$ | 0.97 | 2.51 | 3.404 (4) | 152 |
| C12—H12$C$···O1$^{ii}$ | 0.97 | 2.48 | 3.404 (4) | 159 |

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank Université Mohammed V-Agdal and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5047).

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# supporting information

*Acta Cryst.* (2010). E**66**, o2842 [https://doi.org/10.1107/S1600536810040316]

## 1-Allyl-4-(1,3-benzothiazol-2-yl)-5-methyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

**Imane Chakib, Abdelfettah Zerzouf, Hafid Zouihri, El Mokhtar Essassi and Seik Weng Ng**

### S1. Comment

(*E*)-4-(2,3-Dihydro-1,3-benzothiazol-2-ylidene)-3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one is an amine that can under a nucleophilic substitution with organo bromides to form 2-substituted derivatives if tetra-*n*-butyl ammonium bromide is used as catalyst. In this study, the compound is reacted with allyl bromide to yield the title compound (Scheme I, Fig. 1).

### S2. Experimental

To a solution of (*E*)-4-(2,3-dihydro-1,3-benzothiazol-2-ylidene)-3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one (1 g, 3.25 mmol) in DMF (50 ml), was added sodium carbonate (2.5 g, 23 mmol), tetra-*n*-butyl ammonium bromide (0.15 g, 1 mmol) and allyl bromide (5.6 g, 46 mmol). The mixture was stirred for 24 h at room temperature. The solid material was removed by filtration and the solution was evaporated under reduced. The residue was washed with dichloromethane and hexane, and the recrystallized from ethanol to afford the title compound as colorless crystals.

### S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93–0.97 Å) and were included in the refinement in the riding model approximation, with $U$(H) set to 1.2–1.5$U_{eq}$(C).

The allyl and phenyl units are disordered over two positions; the disorder could be refined, and was assumed to be a 1:1 type of disorder. For the allyl unit, the single-bond distances were restrained to 1.50±0.01 Å and the double-bond distances to 1.35±0.01 Å; the anisotropic temperature factors were restrained to be nearly isotropic. The phenyl rings were refined as rigid hexagons of 1.39 Å sides; the N–$C_{phenyl}$ pair of distances were restrained to within 0.01 Å of each other. Additionally, the temperature factors of the primed atoms were restrained to those of the unprimed ones.

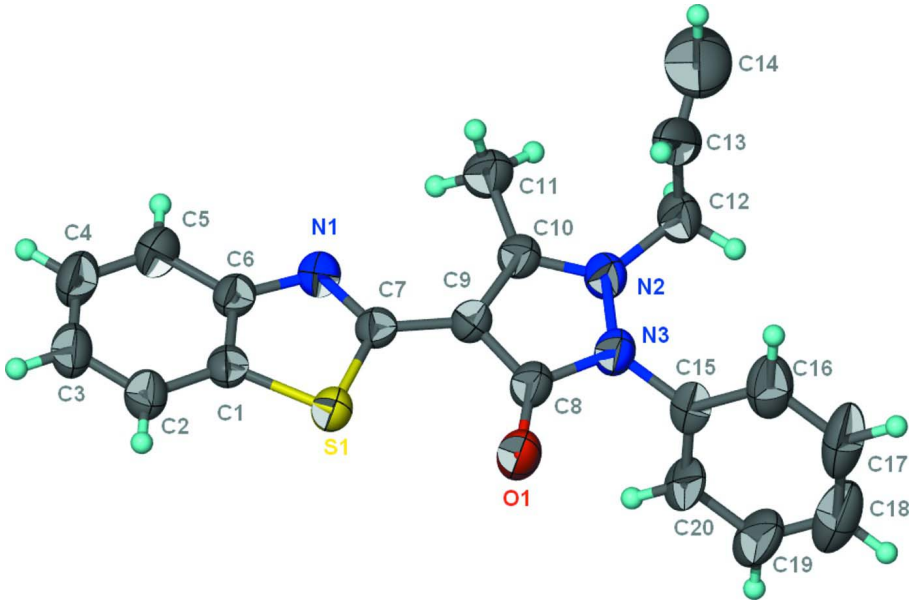


**Figure 1**

Thermal ellipsoid plot (Barbour, 2001) of $C_{20}H_{17}N_3OS$ at the 50% probability level; hydrogen atoms are drawn as arbitrary radius. The disorder is not shown

**1-Allyl-4-(1,3-benzothiazol-2-yl)-5-methyl-2-phenyl- 1*H*-pyrazol-3(2*H*)-one**

*Crystal data*

$C_{20}H_{17}N_3OS$
$M_r = 347.43$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 17.8734\ (5)$ Å
$b = 10.4297\ (2)$ Å
$c = 18.9578\ (4)$ Å
$V = 3534.00\ (14)$ Å$^3$
$Z = 8$
$F(000) = 1456$
$D_x = 1.306$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3745 reflections
$\theta = 2.3–22.5°$
$\mu = 0.20$ mm$^{-1}$
$T = 293$ K
Prism, colorless
0.30 × 0.30 × 0.25 mm

*Data collection*

Bruker X8 APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.944$, $T_{max} = 0.953$
18349 measured reflections
3678 independent reflections
2341 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 26.6°$, $\theta_{min} = 2.4°$
$h = -22\rightarrow21$
$k = -13\rightarrow12$
$l = -23\rightarrow23$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.172$
$S = 1.00$
3678 reflections
227 parameters
17 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.096P)^2 + 0.8646P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.32$ e Å$^{-3}$
$\Delta\rho_{min} = -0.25$ e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| S1 | 0.54022 (4) | 0.59623 (7) | 0.57529 (3) | 0.0590 (2) | |
| O1 | 0.63189 (12) | 0.5096 (2) | 0.45761 (10) | 0.0808 (6) | |
| N1 | 0.61613 (12) | 0.7853 (2) | 0.62953 (11) | 0.0590 (6) | |
| N2 | 0.79529 (13) | 0.6762 (2) | 0.48741 (11) | 0.0593 (6) | |
| N3 | 0.75432 (13) | 0.5809 (2) | 0.45335 (11) | 0.0627 (6) | |
| C1 | 0.50073 (15) | 0.6812 (2) | 0.64448 (12) | 0.0531 (6) | |
| C2 | 0.43115 (17) | 0.6640 (3) | 0.67650 (14) | 0.0669 (7) | |
| H2 | 0.3989 | 0.5995 | 0.6617 | 0.080* | |
| C3 | 0.41164 (19) | 0.7448 (3) | 0.73035 (15) | 0.0762 (9) | |
| H3 | 0.3654 | 0.7349 | 0.7522 | 0.091* | |
| C4 | 0.4595 (2) | 0.8412 (3) | 0.75287 (16) | 0.0798 (9) | |
| H4 | 0.4454 | 0.8943 | 0.7900 | 0.096* | |
| C5 | 0.52717 (19) | 0.8589 (3) | 0.72110 (15) | 0.0743 (8) | |
| H5 | 0.5587 | 0.9242 | 0.7362 | 0.089* | |
| C6 | 0.54904 (16) | 0.7785 (2) | 0.66569 (12) | 0.0566 (6) | |
| C7 | 0.61898 (14) | 0.6956 (2) | 0.58142 (11) | 0.0495 (6) | |
| C8 | 0.68208 (17) | 0.5800 (3) | 0.48056 (13) | 0.0603 (7) | |
| C9 | 0.68293 (15) | 0.6721 (2) | 0.53608 (12) | 0.0518 (6) | |
| C10 | 0.75276 (15) | 0.7255 (2) | 0.53898 (12) | 0.0548 (6) | |
| C11 | 0.78327 (19) | 0.8222 (3) | 0.58934 (16) | 0.0760 (8) | |
| H11A | 0.8269 | 0.8617 | 0.5693 | 0.114* | |
| H11B | 0.7965 | 0.7806 | 0.6327 | 0.114* | |
| H11C | 0.7461 | 0.8865 | 0.5984 | 0.114* | |
| C12 | 0.87637 (17) | 0.6862 (3) | 0.48056 (16) | 0.0746 (8) | |
| H12A | 0.8914 | 0.7755 | 0.4806 | 0.090* | 0.50 |
| H12B | 0.8926 | 0.6475 | 0.4366 | 0.090* | 0.50 |
| H12C | 0.8825 | 0.7782 | 0.4852 | 0.090* | 0.50 |
| H12D | 0.8812 | 0.6708 | 0.4303 | 0.090* | 0.50 |
| C13 | 0.9121 (11) | 0.6151 (15) | 0.5440 (7) | 0.101 (3) | 0.50 |
| H13 | 0.8915 | 0.5398 | 0.5616 | 0.121* | 0.50 |
| C14 | 0.9722 (11) | 0.6652 (18) | 0.5718 (9) | 0.139 (4) | 0.50 |
| H14A | 0.9920 | 0.7407 | 0.5535 | 0.166* | 0.50 |
| H14B | 0.9953 | 0.6254 | 0.6099 | 0.166* | 0.50 |
| C13′ | 0.9175 (11) | 0.5938 (15) | 0.5294 (7) | 0.101 (3) | 0.50 |
| H13′ | 0.9148 | 0.5076 | 0.5171 | 0.121* | 0.50 |
| C14′ | 0.9555 (11) | 0.6196 (18) | 0.5854 (8) | 0.139 (4) | 0.50 |
| H14C | 0.9603 | 0.7041 | 0.6004 | 0.166* | 0.50 |
| H14D | 0.9781 | 0.5538 | 0.6107 | 0.166* | 0.50 |
| C15 | 0.7691 (10) | 0.5140 (10) | 0.3885 (4) | 0.0510 (18) | 0.50 |

| | | | | | |
|---|---|---|---|---|---|
| C16 | 0.8277 (10) | 0.4304 (14) | 0.3741 (5) | 0.0863 (18) | 0.50 |
| H16 | 0.8561 | 0.3969 | 0.4108 | 0.104* | 0.50 |
| C17 | 0.8437 (10) | 0.3967 (13) | 0.3048 (5) | 0.105 (3) | 0.50 |
| H17 | 0.8829 | 0.3407 | 0.2951 | 0.126* | 0.50 |
| C18 | 0.8012 (11) | 0.4466 (10) | 0.2499 (4) | 0.110 (4) | 0.50 |
| H18 | 0.8119 | 0.4241 | 0.2035 | 0.132* | 0.50 |
| C19 | 0.7426 (10) | 0.5303 (12) | 0.2643 (6) | 0.102 (3) | 0.50 |
| H19 | 0.7142 | 0.5637 | 0.2275 | 0.123* | 0.50 |
| C20 | 0.7265 (9) | 0.5640 (12) | 0.3336 (7) | 0.0714 (19) | 0.50 |
| H20 | 0.6874 | 0.6199 | 0.3432 | 0.086* | 0.50 |
| C15′ | 0.7791 (10) | 0.5424 (10) | 0.3848 (5) | 0.0510 (18) | 0.50 |
| C16′ | 0.8326 (10) | 0.4458 (14) | 0.3884 (4) | 0.0863 (18) | 0.50 |
| H16′ | 0.8571 | 0.4294 | 0.4308 | 0.104* | 0.50 |
| C17′ | 0.8494 (11) | 0.3738 (12) | 0.3288 (5) | 0.105 (3) | 0.50 |
| H17′ | 0.8852 | 0.3092 | 0.3312 | 0.126* | 0.50 |
| C18′ | 0.8127 (11) | 0.3984 (9) | 0.2656 (4) | 0.110 (4) | 0.50 |
| H18′ | 0.8240 | 0.3502 | 0.2257 | 0.132* | 0.50 |
| C19′ | 0.7593 (10) | 0.4949 (13) | 0.2620 (5) | 0.102 (3) | 0.50 |
| H19′ | 0.7347 | 0.5113 | 0.2197 | 0.123* | 0.50 |
| C20′ | 0.7425 (9) | 0.5669 (11) | 0.3216 (7) | 0.0714 (19) | 0.50 |
| H20′ | 0.7067 | 0.6315 | 0.3192 | 0.086* | 0.50 |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0542 (4) | 0.0698 (4) | 0.0528 (4) | −0.0033 (3) | 0.0062 (3) | −0.0106 (3) |
| O1 | 0.0647 (13) | 0.1020 (15) | 0.0756 (13) | −0.0228 (12) | 0.0166 (10) | −0.0337 (12) |
| N1 | 0.0559 (14) | 0.0630 (12) | 0.0580 (12) | 0.0042 (10) | 0.0020 (10) | −0.0066 (10) |
| N2 | 0.0529 (14) | 0.0655 (12) | 0.0594 (12) | −0.0072 (11) | 0.0080 (10) | −0.0040 (10) |
| N3 | 0.0568 (14) | 0.0740 (13) | 0.0573 (12) | −0.0097 (11) | 0.0118 (11) | −0.0143 (10) |
| C1 | 0.0521 (16) | 0.0634 (14) | 0.0438 (12) | 0.0102 (12) | −0.0007 (11) | 0.0048 (10) |
| C2 | 0.0615 (18) | 0.0812 (18) | 0.0581 (15) | 0.0026 (15) | 0.0106 (13) | 0.0007 (13) |
| C3 | 0.067 (2) | 0.100 (2) | 0.0611 (16) | 0.0151 (18) | 0.0163 (15) | 0.0006 (15) |
| C4 | 0.084 (2) | 0.090 (2) | 0.0657 (17) | 0.0216 (19) | 0.0131 (17) | −0.0169 (16) |
| C5 | 0.075 (2) | 0.0803 (18) | 0.0677 (17) | 0.0077 (16) | 0.0047 (16) | −0.0213 (15) |
| C6 | 0.0589 (17) | 0.0610 (14) | 0.0500 (13) | 0.0111 (13) | −0.0020 (12) | −0.0043 (11) |
| C7 | 0.0514 (15) | 0.0534 (12) | 0.0438 (11) | 0.0041 (11) | −0.0018 (10) | 0.0018 (10) |
| C8 | 0.0599 (17) | 0.0689 (15) | 0.0521 (13) | −0.0055 (14) | 0.0088 (13) | −0.0060 (12) |
| C9 | 0.0538 (16) | 0.0566 (13) | 0.0450 (12) | 0.0004 (12) | 0.0022 (11) | −0.0006 (10) |
| C10 | 0.0558 (16) | 0.0580 (13) | 0.0507 (13) | −0.0014 (12) | 0.0024 (12) | −0.0003 (11) |
| C11 | 0.070 (2) | 0.0852 (19) | 0.0725 (17) | −0.0151 (16) | 0.0033 (15) | −0.0177 (15) |
| C12 | 0.0580 (19) | 0.090 (2) | 0.0763 (19) | −0.0105 (16) | 0.0148 (15) | −0.0134 (16) |
| C13 | 0.072 (3) | 0.124 (5) | 0.108 (6) | 0.039 (3) | −0.021 (4) | −0.065 (4) |
| C14 | 0.136 (8) | 0.165 (9) | 0.115 (6) | 0.033 (7) | 0.001 (5) | 0.023 (6) |
| C13′ | 0.072 (3) | 0.124 (5) | 0.108 (6) | 0.039 (3) | −0.021 (4) | −0.065 (4) |
| C14′ | 0.136 (8) | 0.165 (9) | 0.115 (6) | 0.033 (7) | 0.001 (5) | 0.023 (6) |
| C15 | 0.059 (4) | 0.031 (4) | 0.0631 (17) | −0.024 (4) | 0.0240 (16) | −0.001 (2) |
| C16 | 0.087 (3) | 0.067 (3) | 0.105 (4) | 0.001 (3) | 0.026 (4) | −0.022 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C17 | 0.122 (5) | 0.085 (5) | 0.108 (8) | 0.000 (4) | 0.058 (7) | −0.028 (5) |
| C18 | 0.133 (8) | 0.098 (8) | 0.098 (5) | −0.045 (8) | 0.057 (6) | −0.038 (5) |
| C19 | 0.100 (7) | 0.149 (8) | 0.0579 (19) | −0.034 (6) | 0.029 (3) | −0.017 (3) |
| C20 | 0.069 (6) | 0.102 (2) | 0.043 (4) | −0.020 (3) | 0.028 (3) | 0.007 (2) |
| C15′ | 0.059 (4) | 0.031 (4) | 0.0631 (17) | −0.024 (4) | 0.0240 (16) | −0.001 (2) |
| C16′ | 0.087 (3) | 0.067 (3) | 0.105 (4) | 0.001 (3) | 0.026 (4) | −0.022 (3) |
| C17′ | 0.122 (5) | 0.085 (5) | 0.108 (8) | 0.000 (4) | 0.058 (7) | −0.028 (5) |
| C18′ | 0.133 (8) | 0.098 (8) | 0.098 (5) | −0.045 (8) | 0.057 (6) | −0.038 (5) |
| C19′ | 0.100 (7) | 0.149 (8) | 0.0579 (19) | −0.034 (6) | 0.029 (3) | −0.017 (3) |
| C20′ | 0.069 (6) | 0.102 (2) | 0.043 (4) | −0.020 (3) | 0.028 (3) | 0.007 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—C1 | 1.733 (2) | C12—H12C | 0.9701 |
| S1—C7 | 1.752 (3) | C12—H12D | 0.9701 |
| O1—C8 | 1.238 (3) | C13—C14 | 1.305 (10) |
| N1—C7 | 1.307 (3) | C13—H13 | 0.9300 |
| N1—C6 | 1.383 (3) | C14—H14A | 0.9300 |
| N2—C10 | 1.341 (3) | C14—H14B | 0.9300 |
| N2—N3 | 1.393 (3) | C13′—C14′ | 1.289 (9) |
| N2—C12 | 1.459 (3) | C13′—H13′ | 0.9300 |
| N3—C8 | 1.391 (4) | C14′—H14C | 0.9300 |
| N3—C15′ | 1.430 (6) | C14′—H14D | 0.9300 |
| N3—C15 | 1.438 (6) | C15—C16 | 1.3900 |
| C1—C6 | 1.392 (4) | C15—C20 | 1.3900 |
| C1—C2 | 1.395 (4) | C16—C17 | 1.3900 |
| C2—C3 | 1.369 (4) | C16—H16 | 0.9300 |
| C2—H2 | 0.9300 | C17—C18 | 1.3900 |
| C3—C4 | 1.388 (5) | C17—H17 | 0.9300 |
| C3—H3 | 0.9300 | C18—C19 | 1.3900 |
| C4—C5 | 1.363 (4) | C18—H18 | 0.9300 |
| C4—H4 | 0.9300 | C19—C20 | 1.3900 |
| C5—C6 | 1.399 (4) | C19—H19 | 0.9300 |
| C5—H5 | 0.9300 | C20—H20 | 0.9300 |
| C7—C9 | 1.451 (3) | C15′—C16′ | 1.3900 |
| C8—C9 | 1.425 (3) | C15′—C20′ | 1.3900 |
| C9—C10 | 1.368 (4) | C16′—C17′ | 1.3900 |
| C10—C11 | 1.492 (4) | C16′—H16′ | 0.9300 |
| C11—H11A | 0.9600 | C17′—C18′ | 1.3900 |
| C11—H11B | 0.9600 | C17′—H17′ | 0.9300 |
| C11—H11C | 0.9600 | C18′—C19′ | 1.3900 |
| C12—C13′ | 1.525 (9) | C18′—H18′ | 0.9300 |
| C12—C13 | 1.551 (8) | C19′—C20′ | 1.3900 |
| C12—H12A | 0.9700 | C19′—H19′ | 0.9300 |
| C12—H12B | 0.9700 | C20′—H20′ | 0.9300 |
| | | | |
| C1—S1—C7 | 88.53 (12) | H12A—C12—H12B | 108.5 |
| C7—N1—C6 | 110.1 (2) | C13′—C12—H12C | 121.0 |

| | | | |
|---|---|---|---|
| C10—N2—N3 | 108.3 (2) | C13—C12—H12C | 110.8 |
| C10—N2—C12 | 126.9 (2) | C13′—C12—H12D | 116.7 |
| N3—N2—C12 | 122.1 (2) | H12C—C12—H12D | 104.1 |
| C8—N3—N2 | 108.7 (2) | C14—C13—C12 | 117.5 (15) |
| C8—N3—C15′ | 128.5 (7) | C14—C13—H13 | 121.3 |
| N2—N3—C15′ | 117.3 (6) | C12—C13—H13 | 121.3 |
| C8—N3—C15 | 118.9 (7) | C13—C14—H14A | 120.0 |
| N2—N3—C15 | 130.3 (6) | C13—C14—H14B | 120.0 |
| C6—C1—C2 | 121.4 (2) | H14A—C14—H14B | 120.0 |
| C6—C1—S1 | 109.81 (19) | C14′—C13′—C12 | 128.4 (15) |
| C2—C1—S1 | 128.8 (2) | C14′—C13′—H13′ | 115.8 |
| C3—C2—C1 | 118.2 (3) | C12—C13′—H13′ | 115.8 |
| C3—C2—H2 | 120.9 | C13′—C14′—H14C | 120.0 |
| C1—C2—H2 | 120.9 | C13′—C14′—H14D | 120.0 |
| C2—C3—C4 | 121.2 (3) | H14C—C14′—H14D | 120.0 |
| C2—C3—H3 | 119.4 | C16—C15—C20 | 120.0 |
| C4—C3—H3 | 119.4 | C16—C15—N3 | 127.6 (11) |
| C5—C4—C3 | 120.6 (3) | C20—C15—N3 | 111.0 (10) |
| C5—C4—H4 | 119.7 | C15—C16—C17 | 120.0 |
| C3—C4—H4 | 119.7 | C15—C16—H16 | 120.0 |
| C4—C5—C6 | 119.9 (3) | C17—C16—H16 | 120.0 |
| C4—C5—H5 | 120.0 | C18—C17—C16 | 120.0 |
| C6—C5—H5 | 120.0 | C18—C17—H17 | 120.0 |
| N1—C6—C1 | 115.6 (2) | C16—C17—H17 | 120.0 |
| N1—C6—C5 | 125.7 (3) | C17—C18—C19 | 120.0 |
| C1—C6—C5 | 118.7 (3) | C17—C18—H18 | 120.0 |
| N1—C7—C9 | 124.4 (2) | C19—C18—H18 | 120.0 |
| N1—C7—S1 | 115.99 (19) | C20—C19—C18 | 120.0 |
| C9—C7—S1 | 119.57 (17) | C20—C19—H19 | 120.0 |
| O1—C8—N3 | 123.1 (2) | C18—C19—H19 | 120.0 |
| O1—C8—C9 | 131.8 (3) | C19—C20—C15 | 120.0 |
| N3—C8—C9 | 105.0 (2) | C19—C20—H20 | 120.0 |
| C10—C9—C8 | 108.3 (2) | C15—C20—H20 | 120.0 |
| C10—C9—C7 | 128.8 (2) | C16′—C15′—C20′ | 120.0 |
| C8—C9—C7 | 122.9 (2) | C16′—C15′—N3 | 111.8 (10) |
| N2—C10—C9 | 109.4 (2) | C20′—C15′—N3 | 125.8 (11) |
| N2—C10—C11 | 121.2 (3) | C15′—C16′—C17′ | 120.0 |
| C9—C10—C11 | 129.4 (2) | C15′—C16′—H16′ | 120.0 |
| C10—C11—H11A | 109.5 | C17′—C16′—H16′ | 120.0 |
| C10—C11—H11B | 109.5 | C18′—C17′—C16′ | 120.0 |
| H11A—C11—H11B | 109.5 | C18′—C17′—H17′ | 120.0 |
| C10—C11—H11C | 109.5 | C16′—C17′—H17′ | 120.0 |
| H11A—C11—H11C | 109.5 | C19′—C18′—C17′ | 120.0 |
| H11B—C11—H11C | 109.5 | C19′—C18′—H18′ | 120.0 |
| N2—C12—C13′ | 112.3 (9) | C17′—C18′—H18′ | 120.0 |
| N2—C12—C13 | 107.8 (9) | C18′—C19′—C20′ | 120.0 |
| N2—C12—H12A | 110.1 | C18′—C19′—H19′ | 120.0 |
| C13′—C12—H12A | 118.2 | C20′—C19′—H19′ | 120.0 |

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| C13—C12—H12A | 110.1 | C19′—C20′—C15′ | 120.0 |
| N2—C12—H12B | 110.1 | C19′—C20′—H20′ | 120.0 |
| C13—C12—H12B | 110.1 | C15′—C20′—H20′ | 120.0 |
| | | | |
| C10—N2—N3—C8 | 5.6 (3) | C12—N2—C10—C11 | 13.0 (4) |
| C12—N2—N3—C8 | 168.3 (2) | C8—C9—C10—N2 | 2.0 (3) |
| C10—N2—N3—C15′ | 161.9 (8) | C7—C9—C10—N2 | 179.5 (2) |
| C12—N2—N3—C15′ | −35.5 (8) | C8—C9—C10—C11 | −177.2 (3) |
| C10—N2—N3—C15 | 168.7 (9) | C7—C9—C10—C11 | 0.3 (5) |
| C12—N2—N3—C15 | −28.7 (9) | C10—N2—C12—C13′ | 77.9 (7) |
| C7—S1—C1—C6 | 1.10 (18) | N3—N2—C12—C13′ | −81.3 (7) |
| C7—S1—C1—C2 | 179.7 (2) | C10—N2—C12—C13 | 64.1 (7) |
| C6—C1—C2—C3 | −0.8 (4) | N3—N2—C12—C13 | −95.2 (7) |
| S1—C1—C2—C3 | −179.3 (2) | N2—C12—C13—C14 | −142.4 (14) |
| C1—C2—C3—C4 | −0.1 (4) | C13′—C12—C13—C14 | 106 (7) |
| C2—C3—C4—C5 | 0.8 (5) | N2—C12—C13′—C14′ | −108 (2) |
| C3—C4—C5—C6 | −0.6 (5) | C13—C12—C13′—C14′ | −35 (6) |
| C7—N1—C6—C1 | 0.3 (3) | C8—N3—C15—C16 | −133.0 (8) |
| C7—N1—C6—C5 | 179.4 (3) | N2—N3—C15—C16 | 65.4 (10) |
| C2—C1—C6—N1 | −179.8 (2) | C15′—N3—C15—C16 | 91 (6) |
| S1—C1—C6—N1 | −1.1 (3) | C8—N3—C15—C20 | 60.6 (8) |
| C2—C1—C6—C5 | 1.0 (4) | N2—N3—C15—C20 | −101.0 (10) |
| S1—C1—C6—C5 | 179.8 (2) | C15′—N3—C15—C20 | −75 (6) |
| C4—C5—C6—N1 | −179.4 (3) | C20—C15—C16—C17 | 0.0 |
| C4—C5—C6—C1 | −0.3 (4) | N3—C15—C16—C17 | −165.3 (9) |
| C6—N1—C7—C9 | −177.2 (2) | C15—C16—C17—C18 | 0.0 |
| C6—N1—C7—S1 | 0.6 (3) | C16—C17—C18—C19 | 0.0 |
| C1—S1—C7—N1 | −1.01 (19) | C17—C18—C19—C20 | 0.0 |
| C1—S1—C7—C9 | 176.89 (19) | C18—C19—C20—C15 | 0.0 |
| N2—N3—C8—O1 | 176.1 (3) | C16—C15—C20—C19 | 0.0 |
| C15′—N3—C8—O1 | 23.3 (8) | N3—C15—C20—C19 | 167.6 (9) |
| C15—N3—C8—O1 | 10.8 (7) | C8—N3—C15′—C16′ | −121.9 (9) |
| N2—N3—C8—C9 | −4.3 (3) | N2—N3—C15′—C16′ | 87.4 (7) |
| C15′—N3—C8—C9 | −157.0 (7) | C15—N3—C15′—C16′ | −71 (6) |
| C15—N3—C8—C9 | −169.5 (7) | C8—N3—C15′—C20′ | 40.7 (11) |
| O1—C8—C9—C10 | −178.9 (3) | N2—N3—C15′—C20′ | −110.1 (9) |
| N3—C8—C9—C10 | 1.4 (3) | C15—N3—C15′—C20′ | 92 (6) |
| O1—C8—C9—C7 | 3.4 (5) | C20′—C15′—C16′—C17′ | 0.0 |
| N3—C8—C9—C7 | −176.2 (2) | N3—C15′—C16′—C17′ | 163.7 (10) |
| N1—C7—C9—C10 | 7.8 (4) | C15′—C16′—C17′—C18′ | 0.0 |
| S1—C7—C9—C10 | −169.9 (2) | C16′—C17′—C18′—C19′ | 0.0 |
| N1—C7—C9—C8 | −175.0 (2) | C17′—C18′—C19′—C20′ | 0.0 |
| S1—C7—C9—C8 | 7.2 (3) | C18′—C19′—C20′—C15′ | 0.0 |
| N3—N2—C10—C9 | −4.7 (3) | C16′—C15′—C20′—C19′ | 0.0 |
| C12—N2—C10—C9 | −166.3 (3) | N3—C15′—C20′—C19′ | −161.3 (9) |
| N3—N2—C10—C11 | 174.6 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C2—H2···O1$^{i}$ | 0.93 | 2.59 | 3.318 (3) | 135 |
| C12—H12*A*···O1$^{ii}$ | 0.97 | 2.51 | 3.404 (4) | 152 |
| C12—H12*C*···O1$^{ii}$ | 0.97 | 2.48 | 3.404 (4) | 159 |

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