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# (2*Z*)-1-(5-Hydroxy-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)-3-(4-methylanilino)-but-2-en-1-one

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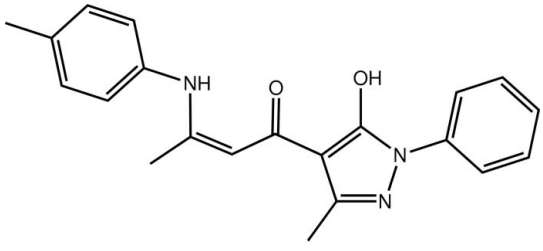
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.060; $wR$ factor = 0.164; data-to-parameter ratio = 16.5.

A twist is evident in the title compound, $C_{21}H_{21}N_3O_2$, the dihedral angle between the terminal six-membered rings being 29.46 (10)°; the linked five- and six-membered rings are coplanar [1.30 (11)°]. The carbonyl O atom accepts intramolecular hydrogen bonds from the adjacent hydroxy and amine groups. The three-dimensional crystal packing is achieved through C—H$\cdots\pi$ interactions.

## Related literature

For background to the synthesis, see: Gelin *et al.* (1983); Bendaas *et al.* (1999). For the structures of the 4-chloro and 4-methoxy analogues, see: Asiri, Al-Youbi, Alamry *et al.* (2011); Asiri, Al-Youbi, Faidallah *et al.* (2011).




## Experimental

### *Crystal data*

$C_{21}H_{21}N_3O_2$
$M_r$ = 347.41
Monoclinic, $P2_1/c$
$a$ = 14.9041 (8) Å
$b$ = 6.9222 (4) Å
$c$ = 17.1921 (8) Å
$\beta$ = 96.190 (5)°
$V$ = 1763.35 (16) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 100 K
0.40 × 0.02 × 0.02 mm

### *Data collection*

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{min}$ = 0.967, $T_{max}$ = 0.998
12780 measured reflections
4055 independent reflections
2455 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.076

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.164
$S$ = 1.02
4055 reflections
246 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.29 e Å$^{-3}$

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

*Cg*1 and *Cg*2 are the centroids of the C1–C6 and C15–C20 rings, respectively.

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| O1—H1$\cdots$O2 | 0.86 (1) | 1.71 (2) | 2.509 (2) | 155 (4) |
| N3—H2$\cdots$O2 | 0.89 (1) | 1.91 (2) | 2.671 (3) | 143 (2) |
| C14—H14*A*$\cdots$*Cg*1$^{i}$ | 0.98 | 2.69 | 3.475 (2) | 138 |
| C14—H14*C*$\cdots$*Cg*1$^{ii}$ | 0.98 | 2.66 | 3.563 (2) | 153 |
| C17—H17$\cdots$*Cg*2$^{iii}$ | 0.95 | 2.58 | 3.424 (2) | 148 |

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

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5179).

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# supplementary materials

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## (2*Z*)-1-(5-Hydroxy-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)-3-(4-methylanilino)but-2-en-1-one

**Abdullah M. Asiri, Abdulrahman O. Al-Youbi, Seik Weng Ng and Edward R. T. Tiekink**

### Comment

In connection with recent structural studies (Asiri, Al-Youbi, Alamry *et al.*, 2011; (Asiri, Al-Youbi, Faidallah *et al.*, 2011) of compounds prepared by reactions between pyrazoles and aniline derivatives following literature procedures (Gelin *et al.*, 1983; Bendaas *et al.*, 1999), the title compound, 3-(4-toluidino)-1-(5-hydroxy-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)but-2-en-1-one (I) was investigated.

While in (I), Fig. 1, the linked five- and six-membered rings are co-planar, forming a dihedral angle of 1.30 (11)°, there is a twist about the N3—C15 bond as seen in the value of the C13—N3—C15—C16 torsion angle of 146.9 (2)°; the dihedral angle between the terminal six-membered rings is 29.46 (10)°. The carbonyl-O2 atom accepts hydrogen bonds from the adjacent hydroxyl- and amine-groups, Table 1. These groups do not participate in intermolecular interactions. Rather, molecules are consolidated in the three-dimensional crystal packing by C—H···$\pi$ interactions, Fig. 2 and Table 1.

### Experimental

A solution of 4-acetoacetyl-5-hydroxy-3-methyl-1-phenylpyrazole (0.005 mol) and *p*-toluidine (0.005 mol) in ethanol (25 ml) was refluxed for 2 h. The precipitate, obtained from the hot solution, was collected, washed with methanol and recrystallized from its ethanol-benzene solution as yellow needles; *M*.pt: 419–421 K.

### Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H = 0.95 to 0.98 Å, $U_{iso}$(H) = 1.2 to 1.5$U_{eq}$(C)] and were included in the refinement in the riding model approximation. The N—H and O—H-atoms were located in a difference Fourier map, and were refined with distance restraints of N—H = 0.88±0.01 and O—H = 0.84±0.01 Å, respectively; their $U_{iso}$ values were refined. Owing to poor agreement, the ($\bar{3}$ 6 3) reflection was omitted from the final cycles of refinement.

### Computing details

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO* (Agilent, 2011); data reduction: *CrysAlis PRO* (Agilent, 2011); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

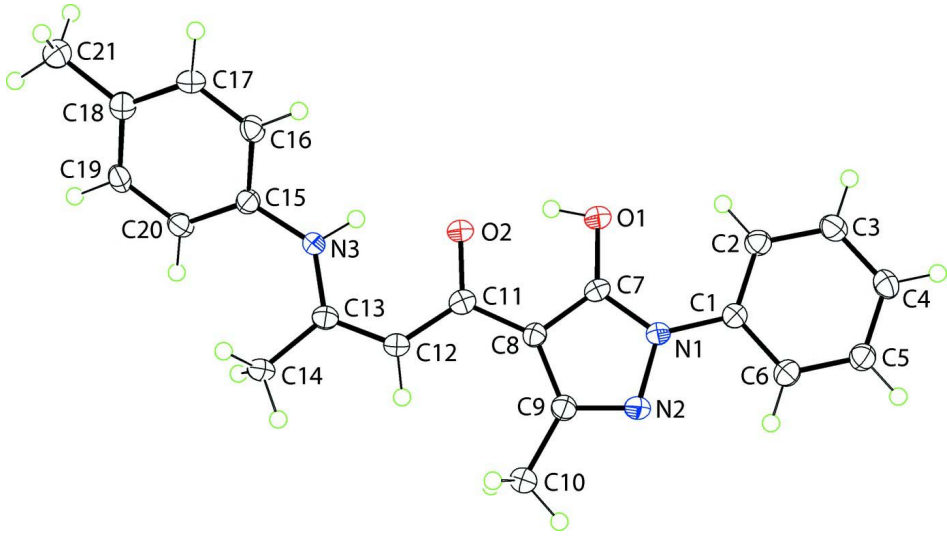


**Figure 1**

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

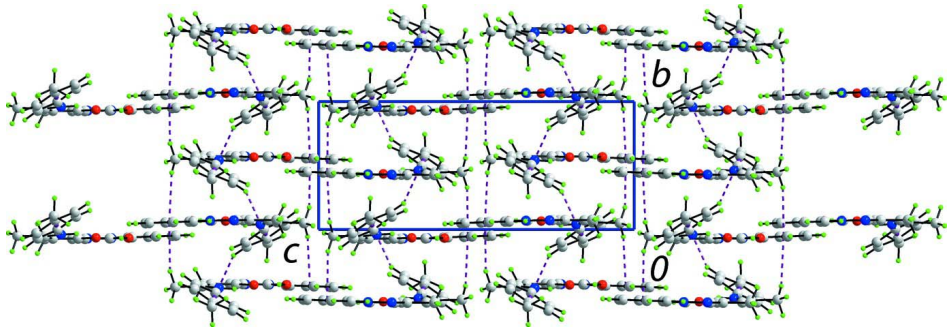


**Figure 2**

A view in projection down the *a* axis of the unit-cell contents of (I). The C—H···$\pi$ interactions are shown as purple dashed lines.

**(2*Z*)-1-(5-Hydroxy-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)- 3-(4-methylanilino)but-2-en-1-one**

*Crystal data*

$C_{21}H_{21}N_3O_2$
$M_r$ = 347.41
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a$ = 14.9041 (8) Å
$b$ = 6.9222 (4) Å
$c$ = 17.1921 (8) Å
$\beta$ = 96.190 (5)°
$V$ = 1763.35 (16) Å$^3$
$Z$ = 4
$F(000)$ = 736
$D_x$ = 1.309 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 2466 reflections
$\theta$ = 2.4–27.5°
$\mu$ = 0.09 mm$^{-1}$
$T$ = 100 K
Needle, yellow
0.40 × 0.02 × 0.02 mm

*Data collection*

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm$^{-1}$
$\omega$ scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{min}$ = 0.967, $T_{max}$ = 0.998
12780 measured reflections
4055 independent reflections
2455 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.076
$\theta_{max}$ = 27.6°, $\theta_{min}$ = 2.4°
$h$ = −18→19
$k$ = −8→8
$l$ = −22→18

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.164
$S$ = 1.02
4055 reflections
246 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.065P)^2 + 0.099P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.29 e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.50304 (12) | 0.0677 (2) | 0.39888 (10) | 0.0275 (4) |
| O2 | 0.37132 (11) | 0.0655 (2) | 0.29485 (9) | 0.0244 (4) |
| N1 | 0.64285 (13) | 0.0686 (2) | 0.34357 (10) | 0.0192 (4) |
| N2 | 0.67114 (13) | 0.0711 (2) | 0.26853 (10) | 0.0229 (4) |
| N3 | 0.23145 (13) | 0.0409 (3) | 0.18426 (11) | 0.0215 (4) |
| C1 | 0.70881 (15) | 0.0615 (3) | 0.40934 (12) | 0.0186 (5) |
| C2 | 0.68417 (16) | 0.0546 (3) | 0.48530 (13) | 0.0231 (5) |
| H2A | 0.6223 | 0.0541 | 0.4941 | 0.028* |
| C3 | 0.75125 (17) | 0.0486 (3) | 0.54797 (13) | 0.0256 (5) |
| H3 | 0.7347 | 0.0431 | 0.5998 | 0.031* |
| C4 | 0.84175 (17) | 0.0503 (3) | 0.53633 (13) | 0.0257 (5) |
| H4 | 0.8870 | 0.0465 | 0.5797 | 0.031* |
| C5 | 0.86567 (17) | 0.0577 (3) | 0.46044 (13) | 0.0245 (5) |
| H5 | 0.9276 | 0.0600 | 0.4519 | 0.029* |
| C6 | 0.79969 (15) | 0.0618 (3) | 0.39707 (13) | 0.0227 (5) |
| H6 | 0.8165 | 0.0649 | 0.3453 | 0.027* |
| C7 | 0.55156 (15) | 0.0701 (3) | 0.33854 (13) | 0.0193 (5) |
| C8 | 0.51783 (15) | 0.0733 (3) | 0.25960 (12) | 0.0187 (5) |
| C9 | 0.59647 (16) | 0.0733 (3) | 0.21952 (13) | 0.0215 (5) |
| C10 | 0.60368 (17) | 0.0740 (3) | 0.13342 (13) | 0.0302 (6) |
| H10A | 0.6674 | 0.0793 | 0.1243 | 0.045* |
| H10B | 0.5721 | 0.1871 | 0.1096 | 0.045* |
| H10C | 0.5763 | −0.0439 | 0.1100 | 0.045* |
| C11 | 0.42170 (16) | 0.0712 (3) | 0.23786 (13) | 0.0207 (5) |
| C12 | 0.38378 (16) | 0.0712 (3) | 0.15824 (12) | 0.0205 (5) |

| | | | | |
|---|---|---|---|---|
| H12 | 0.4245 | 0.0831 | 0.1196 | 0.025* |
| C13 | 0.29317 (16) | 0.0556 (3) | 0.13212 (12) | 0.0200 (5) |
| C14 | 0.26394 (16) | 0.0493 (3) | 0.04612 (12) | 0.0229 (5) |
| H14A | 0.2209 | −0.0566 | 0.0348 | 0.034* |
| H14B | 0.3167 | 0.0283 | 0.0177 | 0.034* |
| H14C | 0.2351 | 0.1720 | 0.0296 | 0.034* |
| C15 | 0.13787 (16) | 0.0037 (3) | 0.17314 (13) | 0.0209 (5) |
| C16 | 0.10125 (15) | −0.1006 (3) | 0.23183 (12) | 0.0225 (5) |
| H16 | 0.1400 | −0.1484 | 0.2751 | 0.027* |
| C17 | 0.00953 (16) | −0.1349 (3) | 0.22785 (13) | 0.0231 (5) |
| H17 | −0.0138 | −0.2033 | 0.2692 | 0.028* |
| C18 | −0.04959 (16) | −0.0719 (3) | 0.16470 (13) | 0.0227 (5) |
| C19 | −0.01234 (16) | 0.0307 (3) | 0.10594 (13) | 0.0229 (5) |
| H19 | −0.0509 | 0.0746 | 0.0618 | 0.028* |
| C20 | 0.07922 (16) | 0.0702 (3) | 0.11024 (12) | 0.0227 (5) |
| H20 | 0.1022 | 0.1432 | 0.0700 | 0.027* |
| C21 | −0.14922 (16) | −0.1052 (3) | 0.16189 (14) | 0.0295 (6) |
| H21 | −0.1795 | −0.0515 | 0.1132 | 0.044* |
| H21B | −0.1721 | −0.0416 | 0.2067 | 0.044* |
| H21C | −0.1612 | −0.2442 | 0.1639 | 0.044* |
| H1 | 0.4494 (12) | 0.069 (5) | 0.375 (2) | 0.102 (15)* |
| H2 | 0.2582 (15) | 0.034 (3) | 0.2329 (7) | 0.031 (7)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0212 (10) | 0.0405 (10) | 0.0217 (9) | −0.0003 (8) | 0.0060 (8) | −0.0002 (7) |
| O2 | 0.0207 (9) | 0.0336 (9) | 0.0199 (8) | −0.0008 (7) | 0.0060 (7) | 0.0005 (6) |
| N1 | 0.0186 (10) | 0.0209 (9) | 0.0184 (9) | 0.0007 (7) | 0.0039 (8) | −0.0005 (7) |
| N2 | 0.0219 (11) | 0.0283 (10) | 0.0190 (10) | −0.0012 (8) | 0.0048 (8) | −0.0007 (7) |
| N3 | 0.0180 (11) | 0.0307 (11) | 0.0159 (10) | −0.0003 (8) | 0.0016 (8) | 0.0020 (8) |
| C1 | 0.0204 (12) | 0.0138 (10) | 0.0216 (11) | 0.0004 (9) | 0.0024 (9) | 0.0017 (8) |
| C2 | 0.0235 (13) | 0.0237 (11) | 0.0224 (12) | 0.0006 (10) | 0.0039 (10) | −0.0017 (9) |
| C3 | 0.0288 (14) | 0.0278 (12) | 0.0203 (12) | 0.0009 (10) | 0.0028 (10) | −0.0003 (9) |
| C4 | 0.0277 (14) | 0.0269 (12) | 0.0213 (12) | −0.0019 (10) | −0.0024 (10) | −0.0005 (9) |
| C5 | 0.0224 (13) | 0.0253 (12) | 0.0256 (12) | −0.0007 (10) | 0.0022 (10) | 0.0008 (9) |
| C6 | 0.0218 (13) | 0.0208 (11) | 0.0254 (12) | −0.0010 (9) | 0.0029 (10) | −0.0005 (9) |
| C7 | 0.0182 (12) | 0.0175 (10) | 0.0230 (12) | −0.0001 (9) | 0.0058 (9) | −0.0005 (8) |
| C8 | 0.0184 (12) | 0.0172 (10) | 0.0203 (11) | −0.0008 (9) | 0.0018 (9) | 0.0009 (8) |
| C9 | 0.0218 (13) | 0.0209 (11) | 0.0218 (12) | −0.0001 (9) | 0.0022 (10) | 0.0009 (8) |
| C10 | 0.0248 (14) | 0.0447 (15) | 0.0214 (12) | 0.0012 (11) | 0.0038 (11) | 0.0016 (10) |
| C11 | 0.0219 (13) | 0.0164 (10) | 0.0244 (12) | 0.0002 (9) | 0.0046 (10) | 0.0007 (8) |
| C12 | 0.0216 (12) | 0.0208 (11) | 0.0196 (11) | −0.0004 (9) | 0.0047 (9) | 0.0009 (8) |
| C13 | 0.0230 (13) | 0.0171 (10) | 0.0202 (11) | 0.0002 (9) | 0.0043 (10) | 0.0013 (8) |
| C14 | 0.0249 (13) | 0.0261 (12) | 0.0187 (11) | 0.0002 (10) | 0.0067 (10) | 0.0002 (9) |
| C15 | 0.0195 (13) | 0.0227 (11) | 0.0205 (11) | −0.0008 (9) | 0.0026 (9) | −0.0036 (8) |
| C16 | 0.0218 (13) | 0.0237 (12) | 0.0214 (12) | 0.0037 (9) | 0.0000 (10) | 0.0018 (8) |
| C17 | 0.0250 (13) | 0.0234 (11) | 0.0219 (12) | −0.0010 (10) | 0.0070 (10) | 0.0026 (9) |
| C18 | 0.0226 (13) | 0.0217 (11) | 0.0239 (12) | 0.0032 (9) | 0.0026 (10) | −0.0041 (9) |
| C19 | 0.0232 (13) | 0.0270 (12) | 0.0176 (11) | 0.0038 (9) | −0.0022 (10) | −0.0030 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C20 | 0.0247 (13) | 0.0249 (11) | 0.0185 (11) | 0.0027 (10) | 0.0029 (10) | 0.0019 (9) |
| C21 | 0.0260 (14) | 0.0347 (13) | 0.0279 (13) | −0.0015 (11) | 0.0038 (11) | −0.0017 (10) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C7 | 1.327 (3) | C10—H10A | 0.9800 |
| O1—H1 | 0.858 (10) | C10—H10B | 0.9800 |
| O2—C11 | 1.298 (3) | C10—H10C | 0.9800 |
| N1—C7 | 1.354 (3) | C11—C12 | 1.423 (3) |
| N1—N2 | 1.400 (2) | C12—C13 | 1.381 (3) |
| N1—C1 | 1.417 (3) | C12—H12 | 0.9500 |
| N2—C9 | 1.322 (3) | C13—C14 | 1.496 (3) |
| N3—C13 | 1.356 (3) | C14—H14A | 0.9800 |
| N3—C15 | 1.411 (3) | C14—H14B | 0.9800 |
| N3—H2 | 0.888 (10) | C14—H14C | 0.9800 |
| C1—C6 | 1.393 (3) | C15—C20 | 1.393 (3) |
| C1—C2 | 1.395 (3) | C15—C16 | 1.398 (3) |
| C2—C3 | 1.389 (3) | C16—C17 | 1.382 (3) |
| C2—H2A | 0.9500 | C16—H16 | 0.9500 |
| C3—C4 | 1.385 (3) | C17—C18 | 1.393 (3) |
| C3—H3 | 0.9500 | C17—H17 | 0.9500 |
| C4—C5 | 1.390 (3) | C18—C19 | 1.398 (3) |
| C4—H4 | 0.9500 | C18—C21 | 1.498 (3) |
| C5—C6 | 1.387 (3) | C19—C20 | 1.386 (3) |
| C5—H5 | 0.9500 | C19—H19 | 0.9500 |
| C6—H6 | 0.9500 | C20—H20 | 0.9500 |
| C7—C8 | 1.396 (3) | C21—H21 | 0.9800 |
| C8—C9 | 1.422 (3) | C21—H21B | 0.9800 |
| C8—C11 | 1.441 (3) | C21—H21C | 0.9800 |
| C9—C10 | 1.496 (3) | | |
| | | | |
| C7—O1—H1 | 101 (3) | H10B—C10—H10C | 109.5 |
| C7—N1—N2 | 109.97 (18) | O2—C11—C12 | 121.6 (2) |
| C7—N1—C1 | 131.08 (18) | O2—C11—C8 | 116.39 (19) |
| N2—N1—C1 | 118.94 (18) | C12—C11—C8 | 122.0 (2) |
| C9—N2—N1 | 105.72 (18) | C13—C12—C11 | 125.9 (2) |
| C13—N3—C15 | 130.94 (19) | C13—C12—H12 | 117.1 |
| C13—N3—H2 | 111.0 (16) | C11—C12—H12 | 117.1 |
| C15—N3—H2 | 117.1 (16) | N3—C13—C12 | 120.0 (2) |
| C6—C1—C2 | 120.0 (2) | N3—C13—C14 | 120.3 (2) |
| C6—C1—N1 | 118.77 (19) | C12—C13—C14 | 119.61 (19) |
| C2—C1—N1 | 121.2 (2) | C13—C14—H14A | 109.5 |
| C3—C2—C1 | 119.1 (2) | C13—C14—H14B | 109.5 |
| C3—C2—H2A | 120.4 | H14A—C14—H14B | 109.5 |
| C1—C2—H2A | 120.4 | C13—C14—H14C | 109.5 |
| C4—C3—C2 | 121.2 (2) | H14A—C14—H14C | 109.5 |
| C4—C3—H3 | 119.4 | H14B—C14—H14C | 109.5 |
| C2—C3—H3 | 119.4 | C20—C15—C16 | 118.0 (2) |
| C3—C4—C5 | 119.2 (2) | C20—C15—N3 | 125.0 (2) |
| C3—C4—H4 | 120.4 | C16—C15—N3 | 117.0 (2) |

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| C5—C4—H4 | 120.4 | C17—C16—C15 | 120.9 (2) |
| C6—C5—C4 | 120.4 (2) | C17—C16—H16 | 119.5 |
| C6—C5—H5 | 119.8 | C15—C16—H16 | 119.5 |
| C4—C5—H5 | 119.8 | C16—C17—C18 | 121.6 (2) |
| C5—C6—C1 | 120.0 (2) | C16—C17—H17 | 119.2 |
| C5—C6—H6 | 120.0 | C18—C17—H17 | 119.2 |
| C1—C6—H6 | 120.0 | C17—C18—C19 | 117.1 (2) |
| O1—C7—N1 | 125.3 (2) | C17—C18—C21 | 121.2 (2) |
| O1—C7—C8 | 126.2 (2) | C19—C18—C21 | 121.6 (2) |
| N1—C7—C8 | 108.45 (19) | C20—C19—C18 | 121.8 (2) |
| C7—C8—C9 | 104.0 (2) | C20—C19—H19 | 119.1 |
| C7—C8—C11 | 119.7 (2) | C18—C19—H19 | 119.1 |
| C9—C8—C11 | 136.3 (2) | C19—C20—C15 | 120.5 (2) |
| N2—C9—C8 | 111.87 (19) | C19—C20—H20 | 119.7 |
| N2—C9—C10 | 119.0 (2) | C15—C20—H20 | 119.7 |
| C8—C9—C10 | 129.1 (2) | C18—C21—H21 | 109.5 |
| C9—C10—H10A | 109.5 | C18—C21—H21B | 109.5 |
| C9—C10—H10B | 109.5 | H21—C21—H21B | 109.5 |
| H10A—C10—H10B | 109.5 | C18—C21—H21C | 109.5 |
| C9—C10—H10C | 109.5 | H21—C21—H21C | 109.5 |
| H10A—C10—H10C | 109.5 | H21B—C21—H21C | 109.5 |

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|---|---|---|---|
| C7—N1—N2—C9 | −0.2 (2) | C11—C8—C9—N2 | −178.4 (2) |
| C1—N1—N2—C9 | 178.60 (16) | C7—C8—C9—C10 | 179.3 (2) |
| C7—N1—C1—C6 | −180.0 (2) | C11—C8—C9—C10 | 1.1 (4) |
| N2—N1—C1—C6 | 1.5 (3) | C7—C8—C11—O2 | −0.3 (3) |
| C7—N1—C1—C2 | −0.1 (3) | C9—C8—C11—O2 | 177.8 (2) |
| N2—N1—C1—C2 | −178.62 (17) | C7—C8—C11—C12 | −179.00 (18) |
| C6—C1—C2—C3 | 0.1 (3) | C9—C8—C11—C12 | −1.0 (4) |
| N1—C1—C2—C3 | −179.74 (17) | O2—C11—C12—C13 | −3.5 (3) |
| C1—C2—C3—C4 | 0.4 (3) | C8—C11—C12—C13 | 175.14 (19) |
| C2—C3—C4—C5 | −0.2 (3) | C15—N3—C13—C12 | −172.5 (2) |
| C3—C4—C5—C6 | −0.5 (3) | C15—N3—C13—C14 | 5.7 (3) |
| C4—C5—C6—C1 | 1.0 (3) | C11—C12—C13—N3 | 0.9 (3) |
| C2—C1—C6—C5 | −0.8 (3) | C11—C12—C13—C14 | −177.35 (18) |
| N1—C1—C6—C5 | 179.08 (17) | C13—N3—C15—C20 | −36.1 (3) |
| N2—N1—C7—O1 | 179.73 (18) | C13—N3—C15—C16 | 146.9 (2) |
| C1—N1—C7—O1 | 1.1 (3) | C20—C15—C16—C17 | −0.6 (3) |
| N2—N1—C7—C8 | 0.1 (2) | N3—C15—C16—C17 | 176.58 (19) |
| C1—N1—C7—C8 | −178.55 (18) | C15—C16—C17—C18 | 1.6 (3) |
| O1—C7—C8—C9 | −179.57 (19) | C16—C17—C18—C19 | −1.0 (3) |
| N1—C7—C8—C9 | 0.1 (2) | C16—C17—C18—C21 | −178.35 (19) |
| O1—C7—C8—C11 | −1.0 (3) | C17—C18—C19—C20 | −0.6 (3) |
| N1—C7—C8—C11 | 178.65 (17) | C21—C18—C19—C20 | 176.7 (2) |
| N1—N2—C9—C8 | 0.3 (2) | C18—C19—C20—C15 | 1.7 (3) |
| N1—N2—C9—C10 | −179.30 (17) | C16—C15—C20—C19 | −1.0 (3) |
| C7—C8—C9—N2 | −0.2 (2) | N3—C15—C20—C19 | −177.94 (19) |

*Hydrogen-bond geometry (Å, º)*

Cg1 and Cg2 are the centroids of the C1–C6 and C15–C20 rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···O2 | 0.86 (1) | 1.71 (2) | 2.509 (2) | 155 (4) |
| N3—H2···O2 | 0.89 (1) | 1.91 (2) | 2.671 (3) | 143 (2) |
| C14—H14*A*···*Cg*2$^{i}$ | 0.98 | 2.69 | 3.475 (2) | 138 |
| C14—H14*C*···*Cg*2$^{ii}$ | 0.98 | 2.66 | 3.563 (2) | 153 |
| C17—H17···*Cg*3$^{iii}$ | 0.95 | 2.58 | 3.424 (2) | 148 |

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