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# 2-{(*E*)-[(2-Hydroxy-1-phenylethyl)imino]methyl}-4-[(*E*)-(4-methylphenyl)diazenyl]phenol

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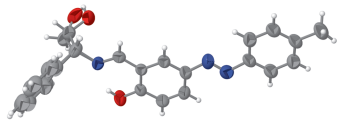
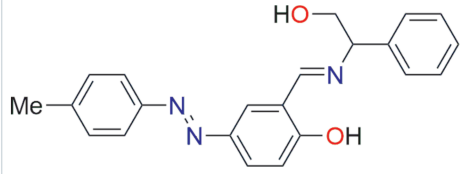
**Structural data:** full structural data are available from iucrdata.iucr.org

In the title compound, $C_{22}H_{21}N_3O_2$, an intramolecular O—H···N hydrogen bond is observed between the phenol and methanimine groups of the molecule. The 2-phenylethan-1-ol part is disordered over two orientations [occupancies 0.566 (17) and 0.434 (17)]. In the crystal, disordered intermolecular O—H···O hydrogen bonds between adjacent molecules form chains parallel to the *b* axis.

3D view



Chemical scheme




## Structure description

Primary amines react with azo compounds, along with aldehydes or ketones, to yield azo-Schiff bases (Su *et al.*, 2015). Azo-Schiff bases serve as chelating ligands in coordination chemistry, where they form complexes with different metal ions that can be applied in catalysis and materials science (Kargar *et al.*, 2022). Studies in the pharmaceutical sector have shown that azo-Schiff bases possess significant biological activities, including antimicrobial (da Silva *et al.*, 2011), antioxidant (Hameed *et al.*, 2017), and anticancer (El-Sonbati *et al.*, 2015) effects. Their potential for interaction with biological targets like enzymes and DNA has paved the way for new drug development approaches (Kaswan, 2023). Moreover, the ability of the azo group to function as a free radical scavenger boosts its potential in addressing oxidative stress-related disorders (Su *et al.*, 2015). This work details the synthesis and crystal structure of an azo-Schiff base.

The asymmetric unit of the crystal structure comprises one molecule of the title compound (Fig. 1). The molecule consists of a (tolyldiazenyl)phenol segment (C1-C12/C22/N1/N2/O1) linked to a 2-phenylethan-1-ol segment (C14–C21/O2) by a methanimine group (C13, N3). The (tolyldiazenyl)phenol and methanimine segments are almost coplanar [torsion angle C8—C9—C13—N3 is 178.9 (3)°]. The 2-phenylethan-1-ol part is disordered over two orientations [occupancies 0.566 (17) for component containing O2,

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2$A$···O1$^{i}$ | 0.82 | 2.00 | 2.745 (10) | 151 |
| O1—H1···N3 | 0.82 | 1.88 | 2.605 (4) | 147 |
| O2$A$—H2$B$···O2$^{ii}$ | 0.82 | 2.14 | 2.88 (2) | 149 |

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

and 0.434 (17) for component containing O2*A*]. The interplanar angle between the rings C1–C6 and C7–C12 is 1.39 (18)°, between rings C1–C6 and C16–C21 62.0 (5)°, and between rings C1–C6 and C16*A*–C21*A* 62.4 (5)°.

An intramolecular O1—H1···N3 hydrogen bond occurs between the phenol and methanimine groups (Table 1). The two positions of the 2-phenylethan-1-ol moiety allow two alternative O—H···O hydrogen bonds to be formed with adjacent molecules in the crystal packing (Table 1), one of which forms chains of molecules propagating parallel to the *b* axis. The other hydrogen bond links pairs of molecules within the chain perpendicular to the direction of chain propagation.

## Synthesis and crystallization

A solution of 4-toluidine (0.214 g, 2 mmol) in concentrated HCl (2 ml) and $H_2O$ (5 ml) was cooled to 273–278 K. Sodium nitrite (0.14 g, 2 mmol) in $H_2O$ (0.5 ml) was added dropwise over 10 minutes. The mixture was stirred for 30 minutes at 273–278 K, followed by the addition of salicylaldehyde (0.244 g, 2 mmol), $H_2O$ (4 ml), NaOH (0.08 g, 2.0 mmol), and $Na_2CO_3$ (0.74 g, 7.0 mmol). The mixture was then stirred for 1 h at 273–278 K. The crude azo product was filtered, washed with $H_2O$, and dried at 298 K under vacuum. A solution of D-phenylglycinol (0.137 g, 1.0 mmol) in MeOH (20 ml) was added to a solution of the azo product (0.240 g, 1.0 mmol) in MeOH (20 ml). The mixture was refluxed for 3 h, and the solid obtained was removed by filtration, dried, and crystallized from ethanol solution to give yellow needles of the title compound in 54.1% yield, m.p. 463–465 K. (KBr) $\nu$ (cm$^{-1}$): 3178, 2979, 1615, 1495, 1372. $^1$H NMR (400 MHz, DMSO; $\delta$ p.p.m.): 14.44 (*s*, 1H), 8.80 (*s*, 1H), 8.11 (*d*, $J$ = 2.6 Hz, 1H), 7.92 (*dd*, $J$ = 8.9, 2.6 Hz, 1H), 7.74 (*d*, $J$ = 8.1 Hz, 2H), 7.45–7.32 (*m*, 7H), 7.01 (*d*, $J$ = 8.9 Hz, 1H), 5.22 (*s*, 1H), 4.57 (*dd*, $J$ = 8.3, 4.4 Hz, 1H), 3.73 (*m*, 2H), 2.39 (*s*, 3H). $^{13}$C NMR (100 MHz, DMSO; $\delta$ p.p.m.): 166.6, 165.9, 150.5, 144.2, 141.2, 140.2, 130.4, 129.7, 129.1, 128.1, 127.5, 126.5, 121.7, 119.2, 118.5, 74.0, 66.3, 21.5. The absolute configuration of C15 is *R*.

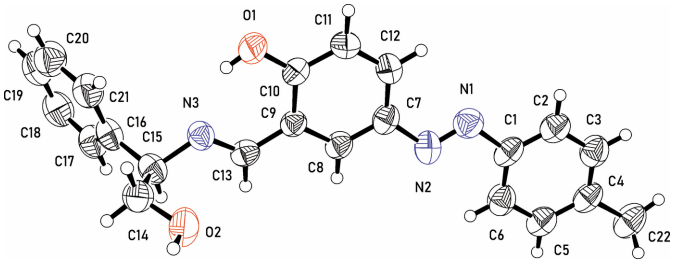



**Figure 1**
An *ORTEP* representation of the title compound showing 50% probability ellipsoids. Only the major component of the disordered 2-phenylethan-1-ol segment is shown.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{22}H_{21}N_3O_2$ |
| $M_r$ | 359.42 |
| Crystal system, space group | Monoclinic, *I*2 |
| Temperature (K) | 293 |
| *a*, *b*, *c* (Å) | 14.7749 (8), 6.0051 (3), 22.3625 (12) |
| $\beta$ (°) | 108.080 (6) |
| $V$ (Å$^3$) | 1886.14 (18) |
| $Z$ | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.08 |
| Crystal size (mm) | 0.28 × 0.25 × 0.20 |
| Data collection | |
| Diffractometer | SuperNova, Dual, Cu at home/near, Atlas |
| Absorption correction | Gaussian (*CrysAlis PRO*; Rigaku OD, 2024) |
| $T_{min}$, $T_{max}$ | 0.514, 1.000 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 15526, 4696, 3546 |
| $R_{int}$ | 0.024 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.698 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.054, 0.148, 1.07 |
| No. of reflections | 4696 |
| No. of parameters | 318 |
| No. of restraints | 450 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.28, −0.17 |
| Absolute structure | Flack *x* determined using 1258 quotients [$(I^+)-(I^-)$]/[$(I^+)+(I^-)$] (Parsons et al., 2013) |
| Absolute structure parameter | 0.1 (5) |

Computer programs: *CrysAlis PRO CCD* (Rigaku OD,2024)), *CrysAlis PRO* (Rigaku OD, 2023), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2019/2* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The 2-phenylethan-1-ol part of the molecule is disordered and was modeled as two components with occupancies refining to 0.566 (17) and 0.434 (17).

## Acknowledgements

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## References


da Silva, C. M., da Silva, D. L., Modolo, L. V., Alves, R. B., de Resende, M. A. & Martins, C. V. B. de Fátima (2011). *J. Adv. Res.* **2**, 1–8.

El-Sonbati, A. Z., El-Bindary, A. A., Diab, M. A., Mohamed, G. G. & Morgan, S. M. (2015). *Spectrochim. Acta A Mol. Biomol. Spectrosc.* **137**, 1126–1135.

Hameed, A., al-Rashida, M., Uroos, M., Abid Ali, S. & Khan, K. M. (2017). *Expert Opin. Ther. Pat.* **27**, 63–79.

Kargar, H., Fallah-Mehrjardi, M., Behjatmanesh-Ardakani, R., Bahadori, M., Moghadam, M., Ashfaq, M., Munawar, K. S. & Tahir, M. N. (2022). *Polyhedron* **213**, 115622.

Kaswan, P. (2023). *Inorg. Chim. Acta* **556**, 121610.

Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.

Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.

Rigaku OD (2023). *CrysAlis PRO*. Rigaku Oxford Diffraction, Yarnton, England.

Rigaku OD (2024). *CrysAlis PRO* CCD. Rigaku Oxford Diffraction, Yarnton, England.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Su, R., Lü, L., Zheng, S., Jin, Y. & An, S. (2015). *Chem. Res. Chin. Univ.* **31**, 60–64.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

# full crystallographic data

*IUCrData* (2025). **10**, x250599 [https://doi.org/10.1107/S2414314625005991]

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**2-{(*E*)-[(2-Hydroxy-1-phenylethyl)imino]methyl}-4-[(*E*)-(4-methylphenyl)diazenyl]phenol**

*Crystal data*

$C_{22}H_{21}N_3O_2$
$M_r$ = 359.42
Monoclinic, *I*2
*a* = 14.7749 (8) Å
*b* = 6.0051 (3) Å
*c* = 22.3625 (12) Å
*β* = 108.080 (6)°
*V* = 1886.14 (18) Å$^3$
*Z* = 4
*F*(000) = 760
$D_x$ = 1.266 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 6820 reflections
*θ* = 2.9–27.5°
*μ* = 0.08 mm$^{-1}$
*T* = 293 K
Block, orange
0.28 × 0.25 × 0.20 mm

*Data collection*

SuperNova, Dual, Cu at home/near, Atlas diffractometer
Detector resolution: 10.5082 pixels mm$^{-1}$
*ω* scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2024)
$T_{min}$ = 0.514, $T_{max}$ = 1.000
15526 measured reflections
4696 independent reflections
3546 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.024
$\theta_{max}$ = 29.7°, $\theta_{min}$ = 2.8°
*h* = −20→20
*k* = −8→7
*l* = −30→30

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.148
*S* = 1.07
4696 reflections
318 parameters
450 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 1.3125P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.17 e Å$^{-3}$
Absolute structure: Flack *x* determined using 1258 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al., 2013)
Absolute structure parameter: 0.1 (5)

*Special details*

**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**. Non-hydrogen atoms were refined with anisotropic displacement parameters. In the final cycles of refinement, hydrogen atom geometry was idealized, and a riding model was used with $U_{iso}$ set at 1.2 or 1.5 times the value of $U_{eq}$ for the atom to which the hydrogen atoms are bonded.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.6053 (2) | 0.3897 (6) | 0.62137 (17) | 0.0589 (9) | |
| C2 | 0.6836 (3) | 0.4712 (7) | 0.66603 (18) | 0.0658 (10) | |
| H2 | 0.681939 | 0.612038 | 0.682988 | 0.079* | |
| C3 | 0.7648 (3) | 0.3448 (7) | 0.68589 (19) | 0.0678 (10) | |
| H3 | 0.817801 | 0.401195 | 0.716574 | 0.081* | |
| C4 | 0.7697 (2) | 0.1358 (7) | 0.66138 (17) | 0.0613 (9) | |
| C5 | 0.6900 (3) | 0.0521 (7) | 0.61597 (18) | 0.0647 (10) | |
| H5 | 0.692188 | −0.088654 | 0.599117 | 0.078* | |
| C6 | 0.6068 (3) | 0.1773 (7) | 0.59551 (18) | 0.0654 (10) | |
| H6 | 0.553136 | 0.121343 | 0.565295 | 0.078* | |
| C7 | 0.3730 (2) | 0.5987 (6) | 0.54591 (16) | 0.0541 (8) | |
| C8 | 0.2943 (2) | 0.5117 (6) | 0.50225 (15) | 0.0516 (7) | |
| H8 | 0.298040 | 0.370315 | 0.486231 | 0.062* | |
| C9 | 0.2087 (2) | 0.6284 (5) | 0.48102 (14) | 0.0459 (6) | |
| C10 | 0.2030 (2) | 0.8452 (6) | 0.50522 (16) | 0.0553 (8) | |
| C11 | 0.2834 (3) | 0.9315 (6) | 0.55060 (18) | 0.0654 (10) | |
| H11 | 0.280349 | 1.071206 | 0.567794 | 0.078* | |
| C12 | 0.3664 (2) | 0.8122 (6) | 0.56994 (17) | 0.0611 (9) | |
| H12 | 0.419441 | 0.873684 | 0.599499 | 0.073* | |
| C13 | 0.1282 (2) | 0.5278 (6) | 0.43591 (15) | 0.0502 (7) | |
| H13 | 0.134933 | 0.386246 | 0.420945 | 0.060* | |
| C14 | −0.0997 (7) | 0.4207 (18) | 0.4027 (6) | 0.064 (2) | 0.566 (17) |
| H14A | −0.128781 | 0.544663 | 0.417697 | 0.077* | 0.566 (17) |
| H14B | −0.149973 | 0.335661 | 0.373353 | 0.077* | 0.566 (17) |
| C15 | −0.0319 (7) | 0.510 (2) | 0.3689 (4) | 0.057 (2) | 0.566 (17) |
| H15 | −0.005627 | 0.382549 | 0.352299 | 0.069* | 0.566 (17) |
| C16 | −0.0758 (10) | 0.669 (2) | 0.3152 (4) | 0.0572 (19) | 0.566 (17) |
| C17 | −0.0684 (10) | 0.637 (2) | 0.2553 (5) | 0.066 (2) | 0.566 (17) |
| H17 | −0.039330 | 0.508395 | 0.247036 | 0.079* | 0.566 (17) |
| C18 | −0.1031 (9) | 0.791 (2) | 0.2075 (5) | 0.074 (2) | 0.566 (17) |
| H18 | −0.096249 | 0.766965 | 0.168079 | 0.089* | 0.566 (17) |
| C19 | −0.1473 (8) | 0.9778 (19) | 0.2189 (5) | 0.070 (2) | 0.566 (17) |
| H19 | −0.170095 | 1.082938 | 0.187252 | 0.084* | 0.566 (17) |
| C20 | −0.1585 (9) | 1.011 (2) | 0.2771 (6) | 0.078 (2) | 0.566 (17) |
| H20 | −0.192024 | 1.134716 | 0.283963 | 0.093* | 0.566 (17) |
| C21 | −0.1202 (11) | 0.863 (2) | 0.3253 (5) | 0.070 (2) | 0.566 (17) |

| | | | | | |
|---|---|---|---|---|---|
| H21 | −0.124083 | 0.893000 | 0.365174 | 0.084* | 0.566 (17) |
| N3 | 0.04729 (18) | 0.6253 (5) | 0.41539 (12) | 0.0517 (6) | 0.566 (17) |
| O2 | −0.0510 (6) | 0.2833 (14) | 0.4543 (5) | 0.087 (3) | 0.566 (17) |
| H2A | −0.089839 | 0.214465 | 0.466496 | 0.131* | 0.566 (17) |
| C14A | −0.1148 (9) | 0.483 (3) | 0.3978 (8) | 0.068 (3) | 0.434 (17) |
| H14C | −0.114683 | 0.596685 | 0.428563 | 0.082* | 0.434 (17) |
| H14D | −0.176288 | 0.484100 | 0.365402 | 0.082* | 0.434 (17) |
| C15A | −0.0353 (9) | 0.525 (3) | 0.3690 (5) | 0.058 (3) | 0.434 (17) |
| H15A | −0.015932 | 0.382088 | 0.355769 | 0.070* | 0.434 (17) |
| C16A | −0.0695 (12) | 0.674 (3) | 0.3122 (5) | 0.059 (3) | 0.434 (17) |
| C17A | −0.0522 (11) | 0.607 (2) | 0.2573 (6) | 0.066 (3) | 0.434 (17) |
| H17A | −0.020482 | 0.473357 | 0.256443 | 0.080* | 0.434 (17) |
| C18A | −0.0823 (10) | 0.738 (2) | 0.2036 (4) | 0.070 (3) | 0.434 (17) |
| H18A | −0.070788 | 0.693030 | 0.166851 | 0.084* | 0.434 (17) |
| C19A | −0.1298 (9) | 0.937 (2) | 0.2048 (5) | 0.066 (3) | 0.434 (17) |
| H19A | −0.149919 | 1.025228 | 0.168929 | 0.080* | 0.434 (17) |
| C20A | −0.1470 (10) | 1.005 (2) | 0.2598 (6) | 0.072 (3) | 0.434 (17) |
| H20A | −0.178745 | 1.137755 | 0.260600 | 0.086* | 0.434 (17) |
| C21A | −0.1169 (12) | 0.873 (3) | 0.3134 (5) | 0.069 (3) | 0.434 (17) |
| H21A | −0.128439 | 0.918086 | 0.350193 | 0.083* | 0.434 (17) |
| N3A | 0.04729 (18) | 0.6253 (5) | 0.41539 (12) | 0.0517 (6) | 0.434 (17) |
| O2A | −0.0972 (11) | 0.2732 (18) | 0.4266 (4) | 0.093 (3) | 0.434 (17) |
| H2B | −0.072975 | 0.288871 | 0.464641 | 0.140* | 0.434 (17) |
| N1 | 0.5240 (2) | 0.5375 (5) | 0.60296 (15) | 0.0645 (8) | |
| N2 | 0.4553 (2) | 0.4536 (6) | 0.56411 (14) | 0.0628 (8) | |
| O1 | 0.12420 (19) | 0.9622 (5) | 0.48643 (15) | 0.0800 (9) | |
| H1 | 0.084660 | 0.895236 | 0.458203 | 0.120* | |
| C22 | 0.8598 (3) | 0.0019 (8) | 0.6838 (2) | 0.0820 (13) | |
| H22A | 0.906649 | 0.083753 | 0.715575 | 0.123* | |
| H22B | 0.883322 | −0.026996 | 0.649024 | 0.123* | |
| H22C | 0.846987 | −0.136700 | 0.700952 | 0.123* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0540 (18) | 0.059 (2) | 0.069 (2) | 0.0148 (16) | 0.0266 (17) | 0.0177 (17) |
| C2 | 0.064 (2) | 0.061 (2) | 0.072 (2) | 0.0097 (18) | 0.0208 (18) | 0.0055 (18) |
| C3 | 0.056 (2) | 0.074 (3) | 0.071 (2) | 0.0041 (18) | 0.0172 (18) | 0.009 (2) |
| C4 | 0.0563 (19) | 0.064 (2) | 0.070 (2) | 0.0158 (18) | 0.0288 (17) | 0.0193 (19) |
| C5 | 0.063 (2) | 0.063 (2) | 0.072 (2) | 0.0086 (18) | 0.0259 (18) | 0.0100 (19) |
| C6 | 0.0529 (19) | 0.075 (3) | 0.068 (2) | 0.0034 (18) | 0.0181 (17) | 0.009 (2) |
| C7 | 0.0480 (16) | 0.061 (2) | 0.0542 (17) | 0.0068 (14) | 0.0177 (14) | 0.0086 (15) |
| C8 | 0.0478 (16) | 0.0506 (18) | 0.0579 (18) | 0.0077 (14) | 0.0186 (14) | 0.0008 (15) |
| C9 | 0.0434 (15) | 0.0446 (16) | 0.0511 (15) | 0.0034 (13) | 0.0168 (12) | 0.0031 (13) |
| C10 | 0.0503 (18) | 0.0476 (19) | 0.0631 (19) | 0.0072 (14) | 0.0107 (15) | 0.0028 (16) |
| C11 | 0.066 (2) | 0.048 (2) | 0.073 (2) | 0.0039 (17) | 0.0084 (18) | −0.0097 (18) |
| C12 | 0.0508 (18) | 0.063 (2) | 0.061 (2) | −0.0018 (16) | 0.0057 (15) | −0.0031 (17) |
| C13 | 0.0519 (17) | 0.0467 (17) | 0.0565 (17) | 0.0032 (14) | 0.0232 (14) | 0.0023 (14) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C14 | 0.052 (4) | 0.062 (5) | 0.080 (4) | 0.002 (3) | 0.021 (3) | −0.002 (4) |
| C15 | 0.048 (3) | 0.064 (4) | 0.061 (3) | −0.007 (3) | 0.019 (3) | −0.008 (3) |
| C16 | 0.044 (3) | 0.073 (4) | 0.055 (3) | −0.009 (3) | 0.015 (3) | −0.010 (3) |
| C17 | 0.056 (4) | 0.078 (4) | 0.064 (4) | −0.001 (3) | 0.018 (3) | −0.010 (3) |
| C18 | 0.070 (4) | 0.091 (5) | 0.061 (3) | −0.009 (4) | 0.019 (3) | −0.012 (4) |
| C19 | 0.066 (4) | 0.084 (5) | 0.058 (4) | −0.008 (4) | 0.015 (4) | −0.006 (4) |
| C20 | 0.074 (4) | 0.083 (4) | 0.071 (5) | 0.002 (4) | 0.016 (4) | −0.002 (4) |
| C21 | 0.066 (4) | 0.077 (4) | 0.063 (4) | −0.001 (3) | 0.014 (3) | −0.003 (4) |
| N3 | 0.0447 (12) | 0.0554 (15) | 0.0544 (13) | 0.0008 (12) | 0.0144 (11) | −0.0056 (13) |
| O2 | 0.056 (4) | 0.102 (4) | 0.093 (5) | −0.015 (3) | 0.006 (4) | 0.047 (4) |
| C14A | 0.051 (4) | 0.081 (6) | 0.076 (5) | −0.007 (5) | 0.024 (4) | 0.010 (5) |
| C15A | 0.049 (4) | 0.062 (4) | 0.062 (4) | −0.004 (4) | 0.015 (4) | −0.011 (4) |
| C16A | 0.045 (4) | 0.073 (4) | 0.059 (4) | −0.008 (4) | 0.013 (4) | −0.011 (4) |
| C17A | 0.058 (5) | 0.083 (5) | 0.058 (4) | −0.003 (4) | 0.018 (4) | −0.013 (4) |
| C18A | 0.069 (5) | 0.082 (5) | 0.059 (4) | −0.003 (4) | 0.020 (4) | −0.019 (4) |
| C19A | 0.066 (5) | 0.080 (5) | 0.054 (4) | 0.000 (4) | 0.020 (4) | −0.014 (4) |
| C20A | 0.069 (4) | 0.084 (4) | 0.063 (5) | 0.002 (4) | 0.023 (4) | −0.008 (5) |
| C21A | 0.065 (4) | 0.080 (5) | 0.060 (5) | 0.004 (4) | 0.016 (4) | −0.010 (4) |
| N3A | 0.0447 (12) | 0.0554 (15) | 0.0544 (13) | 0.0008 (12) | 0.0144 (11) | −0.0056 (13) |
| O2A | 0.093 (7) | 0.115 (6) | 0.054 (5) | −0.053 (5) | −0.005 (4) | 0.018 (4) |
| N1 | 0.0606 (18) | 0.0615 (19) | 0.0714 (19) | 0.0025 (15) | 0.0206 (15) | 0.0031 (16) |
| N2 | 0.0498 (15) | 0.077 (2) | 0.0577 (16) | 0.0032 (14) | 0.0118 (13) | 0.0020 (16) |
| O1 | 0.0621 (16) | 0.0603 (16) | 0.098 (2) | 0.0228 (14) | −0.0036 (14) | −0.0209 (15) |
| C22 | 0.061 (2) | 0.086 (3) | 0.101 (3) | 0.027 (2) | 0.029 (2) | 0.021 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.363 (5) | C16—C17 | 1.391 (8) |
| C1—C6 | 1.403 (5) | C17—C18 | 1.384 (9) |
| C1—N1 | 1.448 (4) | C17—H17 | 0.9300 |
| C2—C3 | 1.373 (5) | C18—C19 | 1.361 (9) |
| C2—H2 | 0.9300 | C18—H18 | 0.9300 |
| C3—C4 | 1.380 (6) | C19—C20 | 1.377 (9) |
| C3—H3 | 0.9300 | C19—H19 | 0.9300 |
| C4—C5 | 1.389 (5) | C20—C21 | 1.376 (9) |
| C4—C22 | 1.502 (5) | C20—H20 | 0.9300 |
| C5—C6 | 1.392 (5) | C21—H21 | 0.9300 |
| C5—H5 | 0.9300 | O2—H2A | 0.8200 |
| C6—H6 | 0.9300 | C14A—O2A | 1.400 (11) |
| C7—C8 | 1.369 (5) | C14A—C15A | 1.526 (10) |
| C7—C12 | 1.405 (5) | C14A—H14C | 0.9700 |
| C7—N2 | 1.448 (4) | C14A—H14D | 0.9700 |
| C8—C9 | 1.394 (4) | C15A—N3A | 1.463 (9) |
| C8—H8 | 0.9300 | C15A—C16A | 1.507 (8) |
| C9—C10 | 1.422 (5) | C15A—H15A | 0.9800 |
| C9—C13 | 1.432 (4) | C16A—C17A | 1.3900 |
| C10—O1 | 1.312 (4) | C16A—C21A | 1.3900 |
| C10—C11 | 1.400 (5) | C17A—C18A | 1.3900 |

| | | | |
|---|---|---|---|
| C11—C12 | 1.370 (5) | C17A—H17A | 0.9300 |
| C11—H11 | 0.9300 | C18A—C19A | 1.3900 |
| C12—H12 | 0.9300 | C18A—H18A | 0.9300 |
| C13—N3A | 1.281 (4) | C19A—C20A | 1.3900 |
| C13—N3 | 1.281 (4) | C19A—H19A | 0.9300 |
| C13—H13 | 0.9300 | C20A—C21A | 1.3900 |
| C14—O2 | 1.419 (10) | C20A—H20A | 0.9300 |
| C14—C15 | 1.526 (8) | C21A—H21A | 0.9300 |
| C14—H14A | 0.9700 | O2A—H2B | 0.8200 |
| C14—H14B | 0.9700 | N1—N2 | 1.221 (4) |
| C15—N3 | 1.475 (7) | O1—H1 | 0.8200 |
| C15—C16 | 1.517 (8) | C22—H22A | 0.9600 |
| C15—H15 | 0.9800 | C22—H22B | 0.9600 |
| C16—C21 | 1.387 (8) | C22—H22C | 0.9600 |
| | | | |
| C2—C1—C6 | 120.7 (3) | C16—C17—H17 | 119.0 |
| C2—C1—N1 | 115.4 (4) | C19—C18—C17 | 119.4 (8) |
| C6—C1—N1 | 123.9 (4) | C19—C18—H18 | 120.3 |
| C1—C2—C3 | 119.7 (4) | C17—C18—H18 | 120.3 |
| C1—C2—H2 | 120.1 | C18—C19—C20 | 120.2 (8) |
| C3—C2—H2 | 120.1 | C18—C19—H19 | 119.9 |
| C2—C3—C4 | 121.5 (4) | C20—C19—H19 | 119.9 |
| C2—C3—H3 | 119.2 | C21—C20—C19 | 120.2 (8) |
| C4—C3—H3 | 119.2 | C21—C20—H20 | 119.9 |
| C3—C4—C5 | 118.9 (3) | C19—C20—H20 | 119.9 |
| C3—C4—C22 | 120.3 (4) | C20—C21—C16 | 121.1 (8) |
| C5—C4—C22 | 120.8 (4) | C20—C21—H21 | 119.5 |
| C4—C5—C6 | 120.4 (4) | C16—C21—H21 | 119.5 |
| C4—C5—H5 | 119.8 | C13—N3—C15 | 119.0 (6) |
| C6—C5—H5 | 119.8 | C14—O2—H2A | 109.5 |
| C5—C6—C1 | 118.7 (4) | O2A—C14A—C15A | 106.6 (10) |
| C5—C6—H6 | 120.6 | O2A—C14A—H14C | 110.4 |
| C1—C6—H6 | 120.6 | C15A—C14A—H14C | 110.4 |
| C8—C7—C12 | 118.5 (3) | O2A—C14A—H14D | 110.4 |
| C8—C7—N2 | 115.1 (3) | C15A—C14A—H14D | 110.4 |
| C12—C7—N2 | 126.4 (3) | H14C—C14A—H14D | 108.6 |
| C7—C8—C9 | 122.1 (3) | N3A—C15A—C16A | 109.9 (9) |
| C7—C8—H8 | 119.0 | N3A—C15A—C14A | 110.6 (10) |
| C9—C8—H8 | 119.0 | C16A—C15A—C14A | 110.7 (10) |
| C8—C9—C10 | 119.0 (3) | N3A—C15A—H15A | 108.5 |
| C8—C9—C13 | 119.7 (3) | C16A—C15A—H15A | 108.5 |
| C10—C9—C13 | 121.3 (3) | C14A—C15A—H15A | 108.5 |
| O1—C10—C11 | 120.2 (3) | C17A—C16A—C21A | 120.0 |
| O1—C10—C9 | 121.3 (3) | C17A—C16A—C15A | 117.9 (8) |
| C11—C10—C9 | 118.5 (3) | C21A—C16A—C15A | 122.1 (8) |
| C12—C11—C10 | 120.7 (3) | C18A—C17A—C16A | 120.0 |
| C12—C11—H11 | 119.6 | C18A—C17A—H17A | 120.0 |
| C10—C11—H11 | 119.6 | C16A—C17A—H17A | 120.0 |

| | | | |
|---|---|---|---|
| C11—C12—C7 | 121.2 (3) | C17A—C18A—C19A | 120.0 |
| C11—C12—H12 | 119.4 | C17A—C18A—H18A | 120.0 |
| C7—C12—H12 | 119.4 | C19A—C18A—H18A | 120.0 |
| N3A—C13—C9 | 122.5 (3) | C20A—C19A—C18A | 120.0 |
| N3—C13—C9 | 122.5 (3) | C20A—C19A—H19A | 120.0 |
| N3—C13—H13 | 118.7 | C18A—C19A—H19A | 120.0 |
| C9—C13—H13 | 118.7 | C21A—C20A—C19A | 120.0 |
| O2—C14—C15 | 111.1 (7) | C21A—C20A—H20A | 120.0 |
| O2—C14—H14A | 109.4 | C19A—C20A—H20A | 120.0 |
| C15—C14—H14A | 109.4 | C20A—C21A—C16A | 120.0 |
| O2—C14—H14B | 109.4 | C20A—C21A—H21A | 120.0 |
| C15—C14—H14B | 109.4 | C16A—C21A—H21A | 120.0 |
| H14A—C14—H14B | 108.0 | C13—N3A—C15A | 123.1 (8) |
| N3—C15—C16 | 108.1 (7) | C14A—O2A—H2B | 109.5 |
| N3—C15—C14 | 108.4 (7) | N2—N1—C1 | 112.5 (3) |
| C16—C15—C14 | 115.3 (8) | N1—N2—C7 | 113.1 (3) |
| N3—C15—H15 | 108.3 | C10—O1—H1 | 109.5 |
| C16—C15—H15 | 108.3 | C4—C22—H22A | 109.5 |
| C14—C15—H15 | 108.3 | C4—C22—H22B | 109.5 |
| C21—C16—C17 | 117.1 (7) | H22A—C22—H22B | 109.5 |
| C21—C16—C15 | 120.6 (7) | C4—C22—H22C | 109.5 |
| C17—C16—C15 | 122.2 (8) | H22A—C22—H22C | 109.5 |
| C18—C17—C16 | 121.9 (8) | H22B—C22—H22C | 109.5 |
| C18—C17—H17 | 119.0 | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2*A*···O1$^{i}$ | 0.82 | 2.00 | 2.745 (10) | 151 |
| O1—H1···N3 | 0.82 | 1.88 | 2.605 (4) | 147 |
| O2*A*—H2*B*···O2$^{ii}$ | 0.82 | 2.14 | 2.88 (2) | 149 |

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