

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

1*H*-Benzimidazol-2-ylmethyl phenyl ether

Noor Doha Hassan, Hairul Anuar Tajuddin, Zanariah Abdullah and Seik Weng Ng*

 Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
 Correspondence e-mail: seikweng@um.edu.my

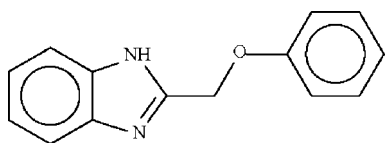
Received 2 March 2009; accepted 4 March 2009

 Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.009$ Å; R factor = 0.046; wR factor = 0.111; data-to-parameter ratio = 8.6.

There are two molecules in the asymmetric unit of the title compound, $\text{C}_{14}\text{H}_{10}\text{N}_2\text{O}$: the dihedral angles between their aromatic ring planes are 47.4 (4) and 46.8 (3)°. In the crystal structure, molecules are linked by $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds from the secondary nitrogen $\text{N}-\text{H}$ donor to the tertiary N -atom acceptor of a symmetry-related neighbour, resulting in hydrogen-bonded chains. The two independent chains both propagate in [100].

Related literature

For related phenoxy-substituted *N*-heterocycles, see: Abdullah & Ng (2008); Hassan *et al.* (2008); Idris *et al.* (2009); Shah Bakhtiar *et al.* (2009).



Experimental

Crystal data

 $\text{C}_{14}\text{H}_{10}\text{N}_2\text{O}$
 $M_r = 224.26$

 Orthorhombic, $Pca2_1$
 $a = 10.0299$ (5) Å

 $b = 8.5391$ (4) Å
 $c = 27.000$ (1) Å
 $V = 2312.5$ (2) Å³
 $Z = 8$

 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 120$ K
 $0.40 \times 0.10 \times 0.03$ mm

Data collection

 Bruker SMART APEX CCD
 diffractometer
 Absorption correction: none
 15022 measured reflections

 2699 independent reflections
 1783 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.091$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.111$
 $S = 1.00$
 2699 reflections
 313 parameters
 3 restraints

 H atoms treated by a mixture of
 independent and constrained
 refinement
 $\Delta\rho_{\text{max}} = 0.19$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.23$ e Å⁻³

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

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{N1}-\text{H1}\cdots\text{N2}^{\text{i}}$ | 0.88 (1) | 2.03 (2) | 2.879 (7) | 163 (6) |
| $\text{N3}-\text{H3}\cdots\text{N4}^{\text{ii}}$ | 0.88 (1) | 1.97 (2) | 2.845 (8) | 172 (5) |

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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: *pubCIF* (Westrip, 2009).

We thank the University of Malaya for supporting this study (FS358/2008 A).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2923).

References

- Abdullah, Z. & Ng, S. W. (2008). *Acta Cryst.* **E64**, o2165.
 Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
 Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Hassan, N. D., Tajuddin, H. A., Abdullah, Z. & Ng, S. W. (2008). *Acta Cryst.* **E64**, o1820.
 Idris, A., Afiffin, A., Abdullah, Z. & Ng, S. W. (2009). *Acta Cryst.* **E65**, o7.
 Shah Bakhtiar, N., Abdullah, Z. & Ng, S. W. (2009). *Acta Cryst.* **E65**, o114.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Westrip, S. P. (2009). *pubCIF*. In preparation.

supplementary materials

Acta Cryst. (2009). E65, o733 [doi:10.1107/S1600536809007922]

1*H*-Benzimidazol-2-ylmethyl phenyl ether

N. D. Hassan, H. A. Tajuddin, Z. Abdullah and S. W. Ng

Experimental

Phenol (1.88 g, 20 mmol) was mixed with sodium hydroxide (0.08 g, 20 mmol) in several drops of water. The water was then evaporated. The paste was heated with 2-(chloromethyl)benzimidazole 3.30 g, 20 mmol) at 423–433 K for 6 h. The product was dissolved in water and the solution extracted with chloroform. The chloroform phase was dried over sodium sulfate; the evaporation of the solvent a brown product; this was purified by column chromatography with an ethyl acetate/hexane mixture. Crystals were grown from this solvent system gave well shaped colorless crystals along with some unidentified brown material.

Refinement

Anomalous dispersion was negligible and Friedel pairs were merged before refinement.

The C-bound H-atoms were placed in calculated positions (C—H 0.95–98 Å) and refined as riding with $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The N-bound H atoms were located in a difference map, and were refined with a restraint of N–H 0.88±0.01 Å; their U_{iso} values were freely refined.

Figures

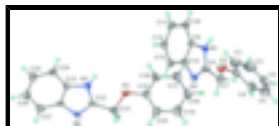


Fig. 1. The molecular structure of (I) with displacement ellipsoids shown at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

1*H*-Benzimidazol-2-ylmethyl phenyl ether

Crystal data

$\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}$

$M_r = 224.26$

Orthorhombic, $Pca2_1$

Hall symbol: P 2c -2ac

$a = 10.0299$ (5) Å

$b = 8.5391$ (4) Å

$c = 27.000$ (1) Å

$V = 2312.5$ (2) Å³

$Z = 8$

$F_{000} = 944$

$D_x = 1.288$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 1070 reflections

$\theta = 2.8$ – 21.3°

$\mu = 0.08$ mm⁻¹

$T = 120$ K

Prism, colorless

$0.40 \times 0.10 \times 0.03$ mm

supplementary materials

Data collection

| | |
|--|--|
| Bruker SMART APEX CCD diffractometer | 1783 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{\text{int}} = 0.091$ |
| Monochromator: graphite | $\theta_{\text{max}} = 27.5^\circ$ |
| $T = 120$ K | $\theta_{\text{min}} = 2.4^\circ$ |
| ω scans | $h = -12 \rightarrow 12$ |
| Absorption correction: None | $k = -11 \rightarrow 10$ |
| 15022 measured reflections | $l = -35 \rightarrow 34$ |
| 2699 independent reflections | |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.046$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.111$ | $w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.1971P]$ |
| $S = 1.00$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 2699 reflections | $(\Delta/\sigma)_{\text{max}} = 0.001$ |
| 313 parameters | $\Delta\rho_{\text{max}} = 0.19 \text{ e } \text{\AA}^{-3}$ |
| 3 restraints | $\Delta\rho_{\text{min}} = -0.23 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|-------------|--------------|----------------------------------|
| O1 | 0.0484 (3) | 1.2098 (3) | 0.50009 (13) | 0.0299 (7) |
| O2 | 0.2955 (3) | 0.7163 (3) | 0.31456 (13) | 0.0325 (7) |
| N1 | 0.0514 (5) | 0.9631 (5) | 0.5645 (2) | 0.0218 (10) |
| H1 | -0.030 (2) | 0.991 (4) | 0.557 (2) | 0.026* |
| N2 | 0.2729 (4) | 0.9563 (5) | 0.56067 (17) | 0.0248 (9) |
| N3 | 0.2988 (6) | 0.4652 (5) | 0.2512 (2) | 0.0232 (11) |
| H3 | 0.2154 (18) | 0.496 (4) | 0.254 (2) | 0.028* |
| N4 | 0.5228 (4) | 0.4592 (5) | 0.25497 (16) | 0.0228 (9) |
| C1 | 0.0158 (8) | 1.3554 (8) | 0.4819 (3) | 0.0241 (15) |
| C2 | -0.0889 (9) | 1.3564 (10) | 0.4484 (3) | 0.038 (2) |
| H2 | -0.1315 | 1.2614 | 0.4392 | 0.046* |
| C3 | -0.1303 (13) | 1.4968 (6) | 0.4286 (5) | 0.046 (3) |
| H3A | -0.2025 | 1.4980 | 0.4059 | 0.055* |
| C4 | -0.0687 (9) | 1.6364 (10) | 0.4412 (3) | 0.0363 (19) |
| H4 | -0.0977 | 1.7326 | 0.4271 | 0.044* |
| C5 | 0.0356 (9) | 1.6329 (9) | 0.4745 (3) | 0.036 (2) |

| | | | | |
|------|-------------|-------------|--------------|-------------|
| H5 | 0.0789 | 1.7276 | 0.4835 | 0.043* |
| C6 | 0.0775 (12) | 1.4931 (5) | 0.4947 (4) | 0.031 (3) |
| H6 | 0.1494 | 1.4920 | 0.5177 | 0.037* |
| C7 | 0.1627 (4) | 1.1991 (5) | 0.53097 (18) | 0.0251 (10) |
| H7A | 0.1569 | 1.2759 | 0.5584 | 0.030* |
| H7B | 0.2446 | 1.2206 | 0.5117 | 0.030* |
| C8 | 0.1653 (6) | 1.0354 (7) | 0.5510 (2) | 0.0227 (13) |
| C9 | 0.0880 (4) | 0.8244 (5) | 0.58670 (16) | 0.0207 (10) |
| C10 | 0.0147 (4) | 0.7057 (5) | 0.60944 (17) | 0.0241 (9) |
| H10 | -0.0799 | 0.7088 | 0.6108 | 0.029* |
| C11 | 0.0864 (4) | 0.5826 (6) | 0.63004 (18) | 0.0266 (11) |
| H11 | 0.0403 | 0.5013 | 0.6469 | 0.032* |
| C12 | 0.2252 (4) | 0.5768 (5) | 0.62635 (17) | 0.0241 (10) |
| H12 | 0.2713 | 0.4901 | 0.6403 | 0.029* |
| C13 | 0.2976 (4) | 0.6931 (5) | 0.60308 (15) | 0.0239 (9) |
| H13 | 0.3919 | 0.6867 | 0.6003 | 0.029* |
| C14 | 0.2276 (4) | 0.8200 (5) | 0.58383 (16) | 0.0219 (9) |
| C15 | 0.2627 (8) | 0.8623 (8) | 0.3334 (3) | 0.0249 (15) |
| C16 | 0.1578 (8) | 0.8609 (10) | 0.3667 (3) | 0.0348 (19) |
| H16 | 0.1140 | 0.7654 | 0.3744 | 0.042* |
| C17 | 0.1170 (12) | 0.9988 (5) | 0.3886 (4) | 0.035 (3) |
| H17 | 0.0473 | 0.9978 | 0.4124 | 0.042* |
| C18 | 0.1780 (9) | 1.1382 (10) | 0.3759 (3) | 0.0333 (18) |
| H18 | 0.1481 | 1.2340 | 0.3899 | 0.040* |
| C19 | 0.2824 (9) | 1.1375 (9) | 0.3427 (3) | 0.0334 (19) |
| H19 | 0.3246 | 1.2337 | 0.3346 | 0.040* |
| C20 | 0.3278 (12) | 0.9996 (4) | 0.3207 (4) | 0.030 (3) |
| H20 | 0.4002 | 0.9999 | 0.2980 | 0.036* |
| C21 | 0.4121 (4) | 0.7020 (6) | 0.28515 (19) | 0.0288 (11) |
| H21A | 0.4927 | 0.7198 | 0.3055 | 0.035* |
| H21B | 0.4110 | 0.7797 | 0.2579 | 0.035* |
| C22 | 0.4117 (5) | 0.5409 (7) | 0.2650 (2) | 0.0211 (12) |
| C23 | 0.3366 (4) | 0.3256 (5) | 0.22992 (16) | 0.0201 (10) |
| C24 | 0.2635 (4) | 0.2077 (5) | 0.20763 (16) | 0.0241 (9) |
| H24 | 0.1689 | 0.2108 | 0.2062 | 0.029* |
| C25 | 0.3346 (4) | 0.0861 (6) | 0.18773 (18) | 0.0262 (11) |
| H25 | 0.2881 | 0.0041 | 0.1714 | 0.031* |
| C26 | 0.4741 (4) | 0.0791 (5) | 0.19069 (18) | 0.0286 (10) |
| H26 | 0.5197 | -0.0077 | 0.1766 | 0.034* |
| C27 | 0.5464 (4) | 0.1960 (5) | 0.21372 (16) | 0.0260 (10) |
| H27 | 0.6408 | 0.1905 | 0.2162 | 0.031* |
| C28 | 0.4760 (4) | 0.3219 (5) | 0.23303 (16) | 0.0203 (9) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|-------------|-------------|-------------|--------------|--------------|--------------|
| O1 | 0.0255 (17) | 0.0259 (16) | 0.0382 (17) | 0.0004 (14) | -0.0086 (14) | 0.0034 (14) |
| O2 | 0.0274 (17) | 0.0236 (16) | 0.0465 (18) | -0.0025 (13) | 0.0146 (15) | -0.0054 (14) |

supplementary materials

| | | | | | | |
|-----|-----------|-------------|-----------|--------------|--------------|--------------|
| N1 | 0.007 (2) | 0.0248 (18) | 0.033 (3) | 0.005 (2) | -0.0012 (19) | -0.001 (3) |
| N2 | 0.017 (2) | 0.0212 (18) | 0.037 (2) | 0.000 (2) | 0.002 (2) | -0.002 (2) |
| N3 | 0.015 (3) | 0.0226 (18) | 0.032 (3) | -0.001 (2) | 0.002 (2) | -0.001 (3) |
| N4 | 0.014 (2) | 0.0246 (19) | 0.029 (2) | 0.004 (2) | 0.0014 (18) | -0.006 (2) |
| C1 | 0.023 (3) | 0.021 (3) | 0.027 (3) | 0.006 (2) | 0.003 (2) | 0.004 (2) |
| C2 | 0.029 (4) | 0.033 (4) | 0.052 (4) | 0.002 (3) | -0.006 (3) | 0.011 (3) |
| C3 | 0.026 (7) | 0.053 (8) | 0.059 (8) | 0.005 (2) | -0.011 (6) | 0.015 (3) |
| C4 | 0.038 (4) | 0.033 (4) | 0.039 (3) | 0.010 (3) | 0.011 (3) | 0.013 (3) |
| C5 | 0.045 (5) | 0.029 (4) | 0.032 (3) | -0.002 (3) | -0.002 (3) | 0.006 (3) |
| C6 | 0.042 (7) | 0.034 (5) | 0.016 (4) | 0.001 (2) | -0.010 (4) | 0.0036 (17) |
| C7 | 0.016 (2) | 0.024 (3) | 0.036 (3) | -0.0022 (16) | -0.0023 (17) | 0.004 (2) |
| C8 | 0.021 (3) | 0.021 (2) | 0.026 (3) | -0.006 (2) | 0.000 (2) | 0.003 (3) |
| C9 | 0.015 (2) | 0.023 (2) | 0.024 (2) | -0.0020 (17) | -0.0003 (17) | -0.0032 (19) |
| C10 | 0.016 (2) | 0.025 (2) | 0.031 (2) | -0.0027 (18) | 0.0027 (18) | -0.001 (2) |
| C11 | 0.032 (3) | 0.022 (3) | 0.026 (2) | -0.0074 (19) | 0.0008 (19) | -0.002 (2) |
| C12 | 0.025 (3) | 0.018 (2) | 0.030 (2) | 0.0031 (19) | -0.0003 (19) | -0.0006 (19) |
| C13 | 0.016 (2) | 0.026 (2) | 0.029 (2) | 0.0034 (17) | 0.0003 (18) | 0.0000 (18) |
| C14 | 0.017 (2) | 0.023 (2) | 0.025 (2) | 0.0029 (18) | 0.0011 (17) | -0.0022 (17) |
| C15 | 0.025 (3) | 0.024 (3) | 0.025 (3) | 0.000 (2) | 0.000 (2) | -0.004 (2) |
| C16 | 0.028 (4) | 0.036 (4) | 0.040 (4) | -0.007 (3) | 0.012 (3) | -0.010 (3) |
| C17 | 0.029 (6) | 0.040 (6) | 0.037 (6) | -0.0004 (19) | 0.003 (5) | -0.012 (2) |
| C18 | 0.033 (4) | 0.035 (4) | 0.032 (3) | 0.009 (3) | -0.002 (3) | -0.012 (3) |
| C19 | 0.044 (4) | 0.025 (3) | 0.031 (3) | 0.001 (3) | -0.004 (3) | 0.002 (3) |
| C20 | 0.029 (6) | 0.023 (5) | 0.038 (5) | 0.0021 (17) | -0.006 (4) | -0.0012 (18) |
| C21 | 0.018 (3) | 0.033 (3) | 0.036 (3) | -0.0038 (18) | 0.0055 (18) | -0.004 (2) |
| C22 | 0.010 (3) | 0.025 (2) | 0.029 (3) | 0.002 (2) | 0.0020 (19) | 0.005 (3) |
| C23 | 0.019 (2) | 0.020 (2) | 0.021 (2) | 0.0017 (17) | 0.0031 (17) | 0.0017 (19) |
| C24 | 0.019 (2) | 0.026 (2) | 0.028 (2) | -0.0038 (18) | -0.0001 (17) | 0.003 (2) |
| C25 | 0.028 (3) | 0.025 (3) | 0.026 (2) | -0.0021 (19) | -0.0023 (19) | -0.005 (2) |
| C26 | 0.029 (3) | 0.027 (3) | 0.030 (2) | 0.004 (2) | 0.001 (2) | -0.003 (2) |
| C27 | 0.019 (2) | 0.028 (2) | 0.032 (2) | 0.0029 (18) | 0.0016 (18) | 0.0038 (19) |
| C28 | 0.014 (2) | 0.020 (2) | 0.026 (2) | -0.0038 (17) | 0.0009 (17) | 0.0047 (17) |

Geometric parameters (Å, °)

| | | | |
|--------|------------|---------|------------|
| O1—C1 | 1.376 (8) | C10—H10 | 0.9500 |
| O1—C7 | 1.421 (5) | C11—C12 | 1.397 (6) |
| O2—C15 | 1.386 (8) | C11—H11 | 0.9500 |
| O2—C21 | 1.419 (5) | C12—C13 | 1.381 (6) |
| N1—C8 | 1.349 (8) | C12—H12 | 0.9500 |
| N1—C9 | 1.377 (6) | C13—C14 | 1.392 (5) |
| N1—H1 | 0.880 (10) | C13—H13 | 0.9500 |
| N2—C8 | 1.299 (7) | C15—C16 | 1.383 (11) |
| N2—C14 | 1.397 (6) | C15—C20 | 1.385 (11) |
| N3—C22 | 1.357 (8) | C16—C17 | 1.380 (11) |
| N3—C23 | 1.376 (6) | C16—H16 | 0.9500 |
| N3—H3 | 0.881 (10) | C17—C18 | 1.382 (11) |
| N4—C22 | 1.342 (7) | C17—H17 | 0.9500 |
| N4—C28 | 1.395 (6) | C18—C19 | 1.377 (13) |

| | | | |
|------------|------------|---------------|------------|
| C1—C6 | 1.373 (10) | C18—H18 | 0.9500 |
| C1—C2 | 1.386 (12) | C19—C20 | 1.396 (11) |
| C2—C3 | 1.377 (11) | C19—H19 | 0.9500 |
| C2—H2 | 0.9500 | C20—H20 | 0.9500 |
| C3—C4 | 1.384 (12) | C21—C22 | 1.480 (8) |
| C3—H3A | 0.9500 | C21—H21A | 0.9900 |
| C4—C5 | 1.381 (13) | C21—H21B | 0.9900 |
| C4—H4 | 0.9500 | C23—C24 | 1.383 (6) |
| C5—C6 | 1.379 (10) | C23—C28 | 1.402 (5) |
| C5—H5 | 0.9500 | C24—C25 | 1.369 (6) |
| C6—H6 | 0.9500 | C24—H24 | 0.9500 |
| C7—C8 | 1.500 (7) | C25—C26 | 1.402 (6) |
| C7—H7A | 0.9900 | C25—H25 | 0.9500 |
| C7—H7B | 0.9900 | C26—C27 | 1.382 (6) |
| C9—C10 | 1.394 (6) | C26—H26 | 0.9500 |
| C9—C14 | 1.403 (5) | C27—C28 | 1.387 (6) |
| C10—C11 | 1.389 (6) | C27—H27 | 0.9500 |
| C1—O1—C7 | 117.3 (4) | C12—C13—H13 | 121.2 |
| C15—O2—C21 | 118.5 (4) | C14—C13—H13 | 121.2 |
| C8—N1—C9 | 106.6 (5) | C13—C14—N2 | 130.7 (4) |
| C8—N1—H1 | 127 (3) | C13—C14—C9 | 120.2 (4) |
| C9—N1—H1 | 126 (3) | N2—C14—C9 | 109.1 (3) |
| C8—N2—C14 | 104.6 (5) | C16—C15—C20 | 121.8 (8) |
| C22—N3—C23 | 107.3 (5) | C16—C15—O2 | 114.3 (6) |
| C22—N3—H3 | 129 (3) | C20—C15—O2 | 124.0 (7) |
| C23—N3—H3 | 124 (3) | C17—C16—C15 | 119.8 (8) |
| C22—N4—C28 | 104.1 (4) | C17—C16—H16 | 120.1 |
| C6—C1—O1 | 125.2 (7) | C15—C16—H16 | 120.1 |
| C6—C1—C2 | 120.1 (8) | C16—C17—C18 | 119.7 (10) |
| O1—C1—C2 | 114.7 (7) | C16—C17—H17 | 120.1 |
| C3—C2—C1 | 119.1 (9) | C18—C17—H17 | 120.1 |
| C3—C2—H2 | 120.4 | C19—C18—C17 | 119.7 (8) |
| C1—C2—H2 | 120.4 | C19—C18—H18 | 120.2 |
| C2—C3—C4 | 121.4 (11) | C17—C18—H18 | 120.2 |
| C2—C3—H3A | 119.3 | C18—C19—C20 | 121.9 (9) |
| C4—C3—H3A | 119.3 | C18—C19—H19 | 119.0 |
| C5—C4—C3 | 118.6 (8) | C20—C19—H19 | 119.0 |
| C5—C4—H4 | 120.7 | C15—C20—C19 | 117.0 (11) |
| C3—C4—H4 | 120.7 | C15—C20—H20 | 121.5 |
| C6—C5—C4 | 120.6 (9) | C19—C20—H20 | 121.5 |
| C6—C5—H5 | 119.7 | O2—C21—C22 | 106.5 (4) |
| C4—C5—H5 | 119.7 | O2—C21—H21A | 110.4 |
| C1—C6—C5 | 120.2 (10) | C22—C21—H21A | 110.4 |
| C1—C6—H6 | 119.9 | O2—C21—H21B | 110.4 |
| C5—C6—H6 | 119.9 | C22—C21—H21B | 110.4 |
| O1—C7—C8 | 106.6 (4) | H21A—C21—H21B | 108.6 |
| O1—C7—H7A | 110.4 | N4—C22—N3 | 113.0 (5) |
| C8—C7—H7A | 110.4 | N4—C22—C21 | 123.7 (5) |
| O1—C7—H7B | 110.4 | N3—C22—C21 | 123.1 (5) |

supplementary materials

| | | | |
|-----------------|------------|-----------------|------------|
| C8—C7—H7B | 110.4 | N3—C23—C24 | 131.8 (4) |
| H7A—C7—H7B | 108.6 | N3—C23—C28 | 105.7 (4) |
| N2—C8—N1 | 114.3 (5) | C24—C23—C28 | 122.5 (4) |
| N2—C8—C7 | 124.8 (5) | C25—C24—C23 | 116.6 (4) |
| N1—C8—C7 | 120.6 (5) | C25—C24—H24 | 121.7 |
| N1—C9—C10 | 132.6 (4) | C23—C24—H24 | 121.7 |
| N1—C9—C14 | 105.3 (4) | C24—C25—C26 | 122.0 (4) |
| C10—C9—C14 | 122.1 (4) | C24—C25—H25 | 119.0 |
| C11—C10—C9 | 117.0 (4) | C26—C25—H25 | 119.0 |
| C11—C10—H10 | 121.5 | C27—C26—C25 | 121.2 (4) |
| C9—C10—H10 | 121.5 | C27—C26—H26 | 119.4 |
| C10—C11—C12 | 120.9 (4) | C25—C26—H26 | 119.4 |
| C10—C11—H11 | 119.5 | C26—C27—C28 | 117.5 (4) |
| C12—C11—H11 | 119.5 | C26—C27—H27 | 121.3 |
| C13—C12—C11 | 122.0 (4) | C28—C27—H27 | 121.3 |
| C13—C12—H12 | 119.0 | C27—C28—N4 | 129.8 (4) |
| C11—C12—H12 | 119.0 | C27—C28—C23 | 120.2 (4) |
| C12—C13—C14 | 117.7 (4) | N4—C28—C23 | 110.0 (3) |
| C7—O1—C1—C6 | -6.1 (11) | C21—O2—C15—C16 | -172.0 (6) |
| C7—O1—C1—C2 | 174.2 (6) | C21—O2—C15—C20 | 7.9 (10) |
| C6—C1—C2—C3 | -0.5 (15) | C20—C15—C16—C17 | -0.9 (14) |
| O1—C1—C2—C3 | 179.1 (8) | O2—C15—C16—C17 | 179.0 (8) |
| C1—C2—C3—C4 | 0.6 (18) | C15—C16—C17—C18 | 2.4 (16) |
| C2—C3—C4—C5 | -0.4 (18) | C16—C17—C18—C19 | -2.4 (16) |
| C3—C4—C5—C6 | 0.0 (15) | C17—C18—C19—C20 | 1.1 (14) |
| O1—C1—C6—C5 | -179.4 (6) | C16—C15—C20—C19 | -0.4 (15) |
| C2—C1—C6—C5 | 0.2 (16) | O2—C15—C20—C19 | 179.6 (6) |
| C4—C5—C6—C1 | 0.1 (16) | C18—C19—C20—C15 | 0.4 (15) |
| C1—O1—C7—C8 | 172.5 (5) | C15—O2—C21—C22 | -172.6 (5) |
| C14—N2—C8—N1 | -1.5 (7) | C28—N4—C22—N3 | 0.2 (7) |
| C14—N2—C8—C7 | 173.1 (5) | C28—N4—C22—C21 | -175.0 (5) |
| C9—N1—C8—N2 | 2.0 (8) | C23—N3—C22—N4 | -1.0 (7) |
| C9—N1—C8—C7 | -172.9 (5) | C23—N3—C22—C21 | 174.3 (5) |
| O1—C7—C8—N2 | 146.8 (6) | O2—C21—C22—N4 | -149.1 (5) |
| O1—C7—C8—N1 | -39.0 (7) | O2—C21—C22—N3 | 36.1 (7) |
| C8—N1—C9—C10 | 177.0 (5) | C22—N3—C23—C24 | -176.6 (5) |
| C8—N1—C9—C14 | -1.6 (6) | C22—N3—C23—C28 | 1.3 (6) |
| N1—C9—C10—C11 | -177.4 (5) | N3—C23—C24—C25 | 176.7 (5) |
| C14—C9—C10—C11 | 1.0 (6) | C28—C23—C24—C25 | -1.0 (6) |
| C9—C10—C11—C12 | -2.2 (6) | C23—C24—C25—C26 | 1.5 (7) |
| C10—C11—C12—C13 | 1.2 (6) | C24—C25—C26—C27 | -0.5 (7) |
| C11—C12—C13—C14 | 1.1 (6) | C25—C26—C27—C28 | -1.1 (6) |
| C12—C13—C14—N2 | 176.8 (4) | C26—C27—C28—N4 | -175.4 (4) |
| C12—C13—C14—C9 | -2.3 (5) | C26—C27—C28—C23 | 1.5 (6) |
| C8—N2—C14—C13 | -178.8 (5) | C22—N4—C28—C27 | 177.8 (5) |
| C8—N2—C14—C9 | 0.4 (6) | C22—N4—C28—C23 | 0.7 (5) |
| N1—C9—C14—C13 | -179.9 (4) | N3—C23—C28—C27 | -178.7 (4) |
| C10—C9—C14—C13 | 1.3 (6) | C24—C23—C28—C27 | -0.5 (6) |
| N1—C9—C14—N2 | 0.7 (5) | N3—C23—C28—N4 | -1.3 (5) |

C10—C9—C14—N2

-178.0 (4)

C24—C23—C28—N4

176.9 (4)

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H··· <i>A</i> | <i>D</i> —H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> —H··· <i>A</i> |
|--------------------------|-------------|---------------|-----------------------|-------------------------|
| N1—H1···N2 ⁱ | 0.88 (1) | 2.03 (2) | 2.879 (7) | 163 (6) |
| N3—H3···N4 ⁱⁱ | 0.88 (1) | 1.97 (2) | 2.845 (8) | 172 (5) |

Symmetry codes: (i) $x-1/2, -y+2, z$; (ii) $x-1/2, -y+1, z$.

Fig. 1

