Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Ethyl 2-[4-(dimethylamino)phenyl]-1-phenyl-1*H*-benzimidazole-5-carboxylate

**Keng Yoon Yeong,[a] Mohamed Ashraf Ali,[a] Tan Soo Choon,[a] Mohd Mustaqim Rosli[b] and Ibrahim Abdul Razak[b]*‡**

[a]Institute for Research in Molecular Medicine, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: arazaki@usm.my

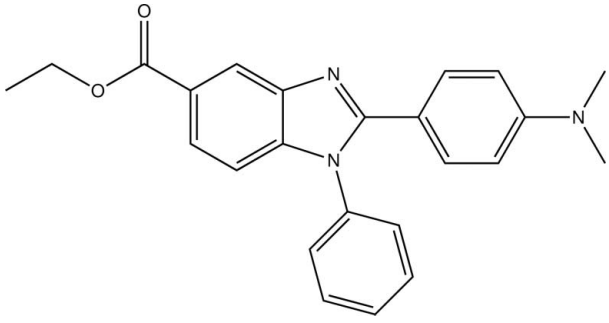
Received 26 April 2013; accepted 7 May 2013

Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.050; $wR$ factor = 0.125; data-to-parameter ratio = 21.8.

In the title compound, $C_{24}H_{23}N_3O_2$, the benzimidazole ring system makes dihedral angles of 7.28 (5) and 67.17 (5)°, respectively, with the planes of the benzene and phenyl rings, which in turn make a dihedral angle of 69.77 (6)°. In the crystal, molecules are connected by C—H···N and C—H···O interactions, forming a layer parallel to the *bc* plane. A $\pi$–$\pi$ interaction, with a centroid–centroid distance of 3.656 (1) Å, is observed in the layer.

## Related literature

For applications of benzimidazole compounds, see: Rao *et al.* (2002); Thakurdesai *et al.* (2007); McKellar & Scott (1990). For a related structure, see: Yoon *et al.* (2012). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



## Experimental

### Crystal data

$C_{24}H_{23}N_3O_2$
$M_r$ = 385.45
Triclinic, $P\overline{1}$
$a$ = 8.7196 (1) Å
$b$ = 10.4133 (2) Å
$c$ = 11.3658 (2) Å
$\alpha$ = 79.312 (1)°
$\beta$ = 74.393 (1)°
$\gamma$ = 89.781 (1)°
$V$ = 975.56 (3) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 100 K
0.36 × 0.25 × 0.25 mm

### Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.970, $T_{max}$ = 0.980
21016 measured reflections
5777 independent reflections
4602 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.029

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.125
$S$ = 1.03
5777 reflections
265 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 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···$A$ |
|---|---|---|---|---|
| C15—H15$A$···N2$^{i}$ | 0.95 | 2.57 | 3.4868 (16) | 164 |
| C18—H18$A$···O2$^{ii}$ | 0.95 | 2.50 | 3.2217 (17) | 133 |
| C19—H19$A$···O2$^{iii}$ | 0.95 | 2.38 | 3.3209 (16) | 170 |

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

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

The authors would like to express their gratitude to Pharmacogenetic and Novel Therapeutic Research, Institute for Research in Molecular Medicine and Department of Pharmacology, School of Pharmaceutical Sciences, Universiti Sains Malaysia. This work was funded through Research Grant No. RUC (1001/PSK/8620012) and HiCoE Research Grant No (311/CIPPM/4401005). IAR also thanks USM for the Short Term Grant, No. 304/PFIZIK/6312078.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5269).

## References

Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
McKellar, Q. A. & Scott, E. W. (1990). *J. Vet. Pharmacol. Ther.* **13**, 223–247.
Rao, A., Chimirri, A., Clercq, E. D., Monforte, A. M., Monforte, P., Pannecouque, C. & Zappala, M. (2002). *Il Farmaco*, **57**, 819–823.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Thakurdesai, P. A., Wadodkar, S. G. & Chopade, C. T. (2007). *Pharmacology Online*, **1**, 314–329.
Yoon, Y. K., Ali, M. A., Choon, T. S., Asik, S. I. J. & Razak, I. A. (2012). *Acta Cryst.* E**68**, o59.

‡ Thomson Reuters ResearcherID: A-5599-2009.

# supporting information

*Acta Cryst.* (2013). E**69**, o886 [doi:10.1107/S1600536813012440]

## Ethyl 2-[4-(dimethylamino)phenyl]-1-phenyl-1*H*-benzimidazole-5-carboxylate

**Keng Yoon Yeong, Mohamed Ashraf Ali, Tan Soo Choon, Mohd Mustaqim Rosli and Ibrahim Abdul Razak**

### S1. Comment

The synthesis of benzimidazole derivatives is an active area of research in medicinal chemistry. Benzimidazoles are a class of bioactive heterocyclic compounds which exhibit a wide range of activities such as anti-HIV (Rao *et al.*, 2002), anti-inflammatory (Thakurdesai *et al.*, 2007) and anthelmintics (McKellar & Scott, 1990). As part of our ongoing structural studies on benzimidazole derivatives (Yoon *et al.*, 2012), we now report the structure of the title compound.

In the title compound (Fig. 1), the benzimidazole ring (N1/N2/C1–C7) is planar with a maximum deviation of 0.025 (1) Å for atom N1. It makes dihedral angles of 7.28 (5) and 67.17 (5)°, respectively, with the benzene (C8–C13) and phenyl (C14–C19) rings, and these two rings make a dihedral angle of 69.77 (6)° with each other.

In the crystal (Fig. 2), the molecules are connected by intermolecular C15—H15A···N2$^{i}$, C18—H18A···O2$^{ii}$ and C19—H19A···O2$^{iii}$ interactions (Table 1) to form two-dimensional layers parallel to the *bc*-plane. A $\pi$–$\pi$ interaction between the benzene rings of C1–C6 and C8–C13 also contributes in stabilizing the crystal structure with their centroid distances of 3.656 (1) Å (2 - *x*, 1 - *y*, 1 - *z*).

### S2. Experimental

Ethyl 3-amino-4-(penylamino)benzoate (0.84 mmol) and sodium metabisulfite adduct of 4-dimethylamino benzaldehyde (1.68 mmol) were dissolved in DMF. The reaction mixture was reflux at 130 °C for 2 hrs. After completion, the reaction mixture was diluted in Ethyl acetate (20 ml) and washed with water (20 ml). The organic layer was collected, dried over $Na_2SO_4$ and the evaporated *in vacuo* to yield the product. The product was recrystallized from Ethyl acetate.

### S3. Refinement

All the H atoms were positioned geometrically and refined using a riding model with with C—H = 0.95–0.99 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C) or 1.5$U_{eq}$(C) for methyl H atoms. A rotating group model was applied to the methyl group.

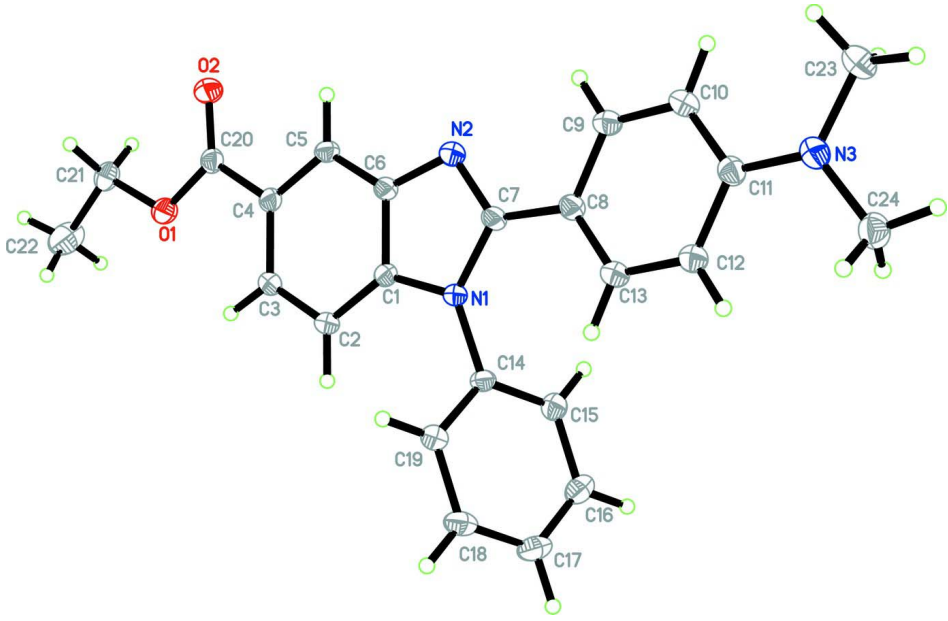



**Figure 1**

The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

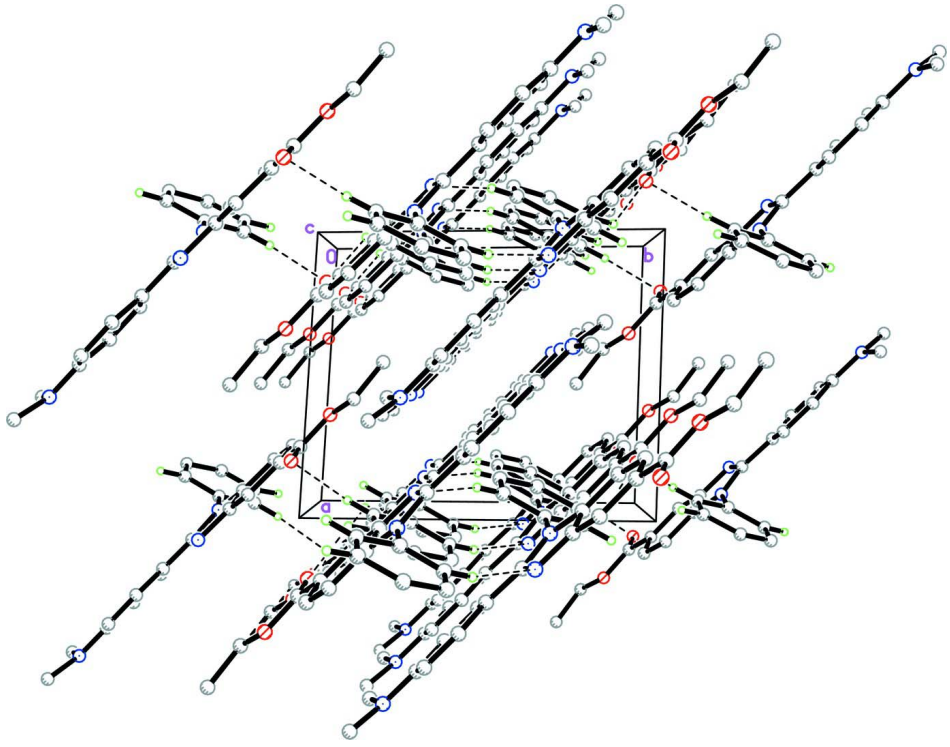



**Figure 2**

A crystal packing view of the title compound. Dashed lines indicate hydrogen bonds. H atoms not involved in the hydrogen bonds have been omitted for clarity.

**Ethyl 2-[4-(dimethylamino)phenyl]-1-phenyl-1*H*-benzimidazole-5-carboxylate**

*Crystal data*

$C_{24}H_{23}N_3O_2$
$M_r = 385.45$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.7196$ (1) Å
$b = 10.4133$ (2) Å
$c = 11.3658$ (2) Å
$\alpha = 79.312$ (1)°
$\beta = 74.393$ (1)°
$\gamma = 89.781$ (1)°
$V = 975.56$ (3) Å$^3$
$Z = 2$
$F(000) = 408$
$D_x = 1.312$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7512 reflections
$\theta = 2.5$–$30.2°$
$\mu = 0.09$ mm$^{-1}$
$T = 100$ K
Block, yellow
0.36 × 0.25 × 0.25 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, 2009)
$T_{min} = 0.970$, $T_{max} = 0.980$
21016 measured reflections
5777 independent reflections
4602 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 30.2°$, $\theta_{min} = 1.9°$
$h = -12 \rightarrow 12$
$k = -14 \rightarrow 14$
$l = -15 \rightarrow 16$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.125$
$S = 1.03$
5777 reflections
265 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
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2 + 0.401P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.38$ 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 Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 1.34962 (11) | −0.05308 (9) | 0.29082 (8) | 0.02242 (19) |
| O2 | 1.18718 (12) | 0.04740 (9) | 0.18312 (9) | 0.0266 (2) |

| | | | | |
|---|---|---|---|---|
| N1 | 0.98249 (12) | 0.30045 (9) | 0.65882 (9) | 0.0171 (2) |
| N2 | 0.88283 (12) | 0.33970 (10) | 0.49238 (9) | 0.0181 (2) |
| N3 | 0.41760 (14) | 0.72466 (11) | 0.79245 (11) | 0.0251 (2) |
| C1 | 1.05977 (14) | 0.22039 (11) | 0.57924 (11) | 0.0166 (2) |
| C2 | 1.17590 (15) | 0.12968 (11) | 0.58860 (11) | 0.0191 (2) |
| H2A | 1.2151 | 0.1116 | 0.6600 | 0.023* |
| C3 | 1.23128 (15) | 0.06738 (11) | 0.48914 (11) | 0.0189 (2) |
| H3A | 1.3098 | 0.0043 | 0.4923 | 0.023* |
| C4 | 1.17363 (14) | 0.09557 (11) | 0.38285 (11) | 0.0171 (2) |
| C5 | 1.05674 (14) | 0.18556 (11) | 0.37516 (11) | 0.0173 (2) |
| H5A | 1.0180 | 0.2040 | 0.3036 | 0.021* |
| C6 | 0.99820 (14) | 0.24776 (11) | 0.47532 (11) | 0.0167 (2) |
| C7 | 0.87548 (14) | 0.37009 (11) | 0.60175 (11) | 0.0171 (2) |
| C8 | 0.76152 (14) | 0.46126 (11) | 0.65582 (11) | 0.0178 (2) |
| C9 | 0.67109 (15) | 0.52985 (12) | 0.58132 (12) | 0.0205 (2) |
| H9A | 0.6880 | 0.5167 | 0.4984 | 0.025* |
| C10 | 0.55857 (15) | 0.61573 (12) | 0.62502 (12) | 0.0216 (2) |
| H10A | 0.5000 | 0.6602 | 0.5718 | 0.026* |
| C11 | 0.52922 (14) | 0.63840 (11) | 0.74735 (12) | 0.0192 (2) |
| C12 | 0.61759 (14) | 0.56812 (12) | 0.82302 (12) | 0.0197 (2) |
| H12A | 0.5998 | 0.5801 | 0.9064 | 0.024* |
| C13 | 0.72998 (14) | 0.48187 (12) | 0.77812 (11) | 0.0193 (2) |
| H13A | 0.7870 | 0.4356 | 0.8317 | 0.023* |
| C14 | 1.02675 (14) | 0.31301 (11) | 0.76961 (11) | 0.0169 (2) |
| C15 | 1.10245 (14) | 0.42822 (12) | 0.77495 (12) | 0.0199 (2) |
| H15A | 1.1247 | 0.4994 | 0.7062 | 0.024* |
| C16 | 1.14503 (15) | 0.43744 (13) | 0.88256 (12) | 0.0234 (3) |
| H16A | 1.1955 | 0.5160 | 0.8879 | 0.028* |
| C17 | 1.11435 (16) | 0.33248 (14) | 0.98248 (12) | 0.0251 (3) |
| H17A | 1.1432 | 0.3398 | 1.0559 | 0.030* |
| C18 | 1.04162 (15) | 0.21708 (13) | 0.97494 (12) | 0.0234 (3) |
| H18A | 1.0224 | 0.1450 | 1.0427 | 0.028* |
| C19 | 0.99673 (15) | 0.20678 (12) | 0.86827 (11) | 0.0195 (2) |
| H19A | 0.9462 | 0.1282 | 0.8630 | 0.023* |
| C20 | 1.23411 (14) | 0.02962 (11) | 0.27581 (11) | 0.0186 (2) |
| C21 | 1.40780 (16) | −0.12580 (13) | 0.19169 (12) | 0.0237 (3) |
| H21A | 1.4474 | −0.0649 | 0.1109 | 0.028* |
| H21B | 1.3210 | −0.1834 | 0.1862 | 0.028* |
| C22 | 1.53955 (19) | −0.20550 (18) | 0.22155 (17) | 0.0406 (4) |
| H22A | 1.5714 | −0.2651 | 0.1628 | 0.061* |
| H22B | 1.5030 | −0.2564 | 0.3065 | 0.061* |
| H22C | 1.6309 | −0.1474 | 0.2150 | 0.061* |
| C23 | 0.34150 (17) | 0.80634 (14) | 0.70882 (14) | 0.0289 (3) |
| H23A | 0.2964 | 0.7515 | 0.6638 | 0.043* |
| H23B | 0.4205 | 0.8701 | 0.6492 | 0.043* |
| H23C | 0.2562 | 0.8526 | 0.7569 | 0.043* |
| C24 | 0.41620 (17) | 0.76536 (13) | 0.90791 (13) | 0.0260 (3) |
| H24A | 0.3841 | 0.6904 | 0.9770 | 0.039* |

| | | | | |
|---|---|---|---|---|
| H24B | 0.3404 | 0.8344 | 0.9219 | 0.039* |
| H24C | 0.5231 | 0.7988 | 0.9029 | 0.039* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0267 (5) | 0.0233 (4) | 0.0198 (4) | 0.0066 (4) | −0.0075 (4) | −0.0088 (3) |
| O2 | 0.0379 (5) | 0.0266 (5) | 0.0194 (5) | 0.0062 (4) | −0.0131 (4) | −0.0072 (4) |
| N1 | 0.0224 (5) | 0.0161 (5) | 0.0155 (5) | 0.0039 (4) | −0.0093 (4) | −0.0038 (4) |
| N2 | 0.0211 (5) | 0.0175 (5) | 0.0164 (5) | 0.0018 (4) | −0.0070 (4) | −0.0023 (4) |
| N3 | 0.0284 (6) | 0.0242 (5) | 0.0256 (6) | 0.0092 (4) | −0.0107 (5) | −0.0074 (4) |
| C1 | 0.0210 (5) | 0.0147 (5) | 0.0148 (5) | 0.0002 (4) | −0.0062 (4) | −0.0031 (4) |
| C2 | 0.0247 (6) | 0.0177 (5) | 0.0174 (5) | 0.0034 (4) | −0.0105 (5) | −0.0028 (4) |
| C3 | 0.0237 (6) | 0.0164 (5) | 0.0183 (6) | 0.0035 (4) | −0.0085 (5) | −0.0033 (4) |
| C4 | 0.0214 (5) | 0.0144 (5) | 0.0156 (5) | −0.0008 (4) | −0.0056 (4) | −0.0022 (4) |
| C5 | 0.0216 (5) | 0.0173 (5) | 0.0143 (5) | −0.0005 (4) | −0.0076 (4) | −0.0019 (4) |
| C6 | 0.0194 (5) | 0.0146 (5) | 0.0166 (5) | −0.0001 (4) | −0.0073 (4) | −0.0011 (4) |
| C7 | 0.0200 (5) | 0.0145 (5) | 0.0169 (5) | 0.0005 (4) | −0.0072 (4) | −0.0001 (4) |
| C8 | 0.0197 (5) | 0.0159 (5) | 0.0179 (5) | 0.0007 (4) | −0.0067 (4) | −0.0012 (4) |
| C9 | 0.0240 (6) | 0.0217 (6) | 0.0173 (6) | 0.0036 (5) | −0.0087 (5) | −0.0034 (4) |
| C10 | 0.0232 (6) | 0.0214 (6) | 0.0219 (6) | 0.0045 (5) | −0.0102 (5) | −0.0020 (5) |
| C11 | 0.0190 (5) | 0.0167 (5) | 0.0218 (6) | 0.0010 (4) | −0.0060 (5) | −0.0026 (4) |
| C12 | 0.0215 (6) | 0.0199 (6) | 0.0180 (6) | 0.0018 (4) | −0.0064 (4) | −0.0028 (4) |
| C13 | 0.0217 (6) | 0.0190 (5) | 0.0174 (5) | 0.0021 (4) | −0.0072 (4) | −0.0013 (4) |
| C14 | 0.0195 (5) | 0.0194 (5) | 0.0141 (5) | 0.0046 (4) | −0.0074 (4) | −0.0049 (4) |
| C15 | 0.0213 (6) | 0.0193 (6) | 0.0195 (6) | 0.0023 (4) | −0.0053 (4) | −0.0050 (4) |
| C16 | 0.0223 (6) | 0.0270 (6) | 0.0240 (6) | 0.0006 (5) | −0.0076 (5) | −0.0108 (5) |
| C17 | 0.0244 (6) | 0.0356 (7) | 0.0193 (6) | 0.0040 (5) | −0.0101 (5) | −0.0095 (5) |
| C18 | 0.0254 (6) | 0.0286 (6) | 0.0166 (6) | 0.0041 (5) | −0.0086 (5) | −0.0010 (5) |
| C19 | 0.0220 (6) | 0.0187 (5) | 0.0191 (6) | 0.0020 (4) | −0.0084 (5) | −0.0026 (4) |
| C20 | 0.0224 (6) | 0.0154 (5) | 0.0178 (6) | −0.0008 (4) | −0.0053 (4) | −0.0029 (4) |
| C21 | 0.0261 (6) | 0.0239 (6) | 0.0218 (6) | 0.0023 (5) | −0.0035 (5) | −0.0107 (5) |
| C22 | 0.0298 (7) | 0.0525 (10) | 0.0517 (10) | 0.0160 (7) | −0.0183 (7) | −0.0301 (8) |
| C23 | 0.0271 (7) | 0.0285 (7) | 0.0343 (7) | 0.0102 (5) | −0.0137 (6) | −0.0062 (6) |
| C24 | 0.0272 (6) | 0.0243 (6) | 0.0260 (7) | 0.0052 (5) | −0.0054 (5) | −0.0065 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C20 | 1.3478 (14) | C11—C12 | 1.4099 (16) |
| O1—C21 | 1.4513 (15) | C12—C13 | 1.3862 (17) |
| O2—C20 | 1.2127 (15) | C12—H12A | 0.9500 |
| N1—C1 | 1.3837 (15) | C13—H13A | 0.9500 |
| N1—C7 | 1.4011 (14) | C14—C15 | 1.3904 (17) |
| N1—C14 | 1.4404 (14) | C14—C19 | 1.3907 (16) |
| N2—C7 | 1.3238 (15) | C15—C16 | 1.3897 (17) |
| N2—C6 | 1.3866 (15) | C15—H15A | 0.9500 |
| N3—C11 | 1.3829 (16) | C16—C17 | 1.3908 (19) |
| N3—C24 | 1.4489 (17) | C16—H16A | 0.9500 |

| | | | |
|---|---|---|---|
| N3—C23 | 1.4500 (17) | C17—C18 | 1.3882 (19) |
| C1—C2 | 1.3937 (16) | C17—H17A | 0.9500 |
| C1—C6 | 1.4070 (16) | C18—C19 | 1.3923 (17) |
| C2—C3 | 1.3813 (17) | C18—H18A | 0.9500 |
| C2—H2A | 0.9500 | C19—H19A | 0.9500 |
| C3—C4 | 1.4115 (16) | C21—C22 | 1.491 (2) |
| C3—H3A | 0.9500 | C21—H21A | 0.9900 |
| C4—C5 | 1.3909 (16) | C21—H21B | 0.9900 |
| C4—C20 | 1.4805 (17) | C22—H22A | 0.9800 |
| C5—C6 | 1.3908 (17) | C22—H22B | 0.9800 |
| C5—H5A | 0.9500 | C22—H22C | 0.9800 |
| C7—C8 | 1.4653 (16) | C23—H23A | 0.9800 |
| C8—C13 | 1.3995 (17) | C23—H23B | 0.9800 |
| C8—C9 | 1.4069 (16) | C23—H23C | 0.9800 |
| C9—C10 | 1.3802 (17) | C24—H24A | 0.9800 |
| C9—H9A | 0.9500 | C24—H24B | 0.9800 |
| C10—C11 | 1.4087 (18) | C24—H24C | 0.9800 |
| C10—H10A | 0.9500 | | |

| | | | |
|---|---|---|---|
| C20—O1—C21 | 115.44 (10) | C8—C13—H13A | 119.2 |
| C1—N1—C7 | 106.59 (9) | C15—C14—C19 | 121.28 (11) |
| C1—N1—C14 | 122.51 (9) | C15—C14—N1 | 120.01 (10) |
| C7—N1—C14 | 130.52 (10) | C19—C14—N1 | 118.68 (11) |
| C7—N2—C6 | 105.79 (10) | C16—C15—C14 | 118.87 (11) |
| C11—N3—C24 | 119.19 (11) | C16—C15—H15A | 120.6 |
| C11—N3—C23 | 119.82 (11) | C14—C15—H15A | 120.6 |
| C24—N3—C23 | 117.76 (11) | C15—C16—C17 | 120.50 (12) |
| N1—C1—C2 | 131.70 (11) | C15—C16—H16A | 119.8 |
| N1—C1—C6 | 105.58 (10) | C17—C16—H16A | 119.8 |
| C2—C1—C6 | 122.72 (11) | C18—C17—C16 | 120.03 (12) |
| C3—C2—C1 | 116.81 (11) | C18—C17—H17A | 120.0 |
| C3—C2—H2A | 121.6 | C16—C17—H17A | 120.0 |
| C1—C2—H2A | 121.6 | C17—C18—C19 | 120.16 (12) |
| C2—C3—C4 | 121.33 (11) | C17—C18—H18A | 119.9 |
| C2—C3—H3A | 119.3 | C19—C18—H18A | 119.9 |
| C4—C3—H3A | 119.3 | C14—C19—C18 | 119.14 (12) |
| C5—C4—C3 | 121.25 (11) | C14—C19—H19A | 120.4 |
| C5—C4—C20 | 117.45 (10) | C18—C19—H19A | 120.4 |
| C3—C4—C20 | 121.30 (11) | O2—C20—O1 | 122.55 (11) |
| C6—C5—C4 | 118.08 (10) | O2—C20—C4 | 124.51 (11) |
| C6—C5—H5A | 121.0 | O1—C20—C4 | 112.95 (10) |
| C4—C5—H5A | 121.0 | O1—C21—C22 | 107.42 (11) |
| N2—C6—C5 | 130.04 (11) | O1—C21—H21A | 110.2 |
| N2—C6—C1 | 110.18 (10) | C22—C21—H21A | 110.2 |
| C5—C6—C1 | 119.77 (11) | O1—C21—H21B | 110.2 |
| N2—C7—N1 | 111.84 (10) | C22—C21—H21B | 110.2 |
| N2—C7—C8 | 122.37 (10) | H21A—C21—H21B | 108.5 |
| N1—C7—C8 | 125.72 (11) | C21—C22—H22A | 109.5 |

| | | | |
|---|---|---|---|
| C13—C8—C9 | 116.88 (11) | C21—C22—H22B | 109.5 |
| C13—C8—C7 | 125.18 (11) | H22A—C22—H22B | 109.5 |
| C9—C8—C7 | 117.88 (11) | C21—C22—H22C | 109.5 |
| C10—C9—C8 | 122.03 (12) | H22A—C22—H22C | 109.5 |
| C10—C9—H9A | 119.0 | H22B—C22—H22C | 109.5 |
| C8—C9—H9A | 119.0 | N3—C23—H23A | 109.5 |
| C9—C10—C11 | 121.03 (11) | N3—C23—H23B | 109.5 |
| C9—C10—H10A | 119.5 | H23A—C23—H23B | 109.5 |
| C11—C10—H10A | 119.5 | N3—C23—H23C | 109.5 |
| N3—C11—C10 | 121.81 (11) | H23A—C23—H23C | 109.5 |
| N3—C11—C12 | 121.07 (11) | H23B—C23—H23C | 109.5 |
| C10—C11—C12 | 117.11 (11) | N3—C24—H24A | 109.5 |
| C13—C12—C11 | 121.30 (11) | N3—C24—H24B | 109.5 |
| C13—C12—H12A | 119.4 | H24A—C24—H24B | 109.5 |
| C11—C12—H12A | 119.4 | N3—C24—H24C | 109.5 |
| C12—C13—C8 | 121.63 (11) | H24A—C24—H24C | 109.5 |
| C12—C13—H13A | 119.2 | H24B—C24—H24C | 109.5 |

| | | | |
|---|---|---|---|
| C7—N1—C1—C2 | −179.19 (12) | C8—C9—C10—C11 | −0.05 (19) |
| C14—N1—C1—C2 | 7.2 (2) | C24—N3—C11—C10 | −166.84 (12) |
| C7—N1—C1—C6 | 1.38 (12) | C23—N3—C11—C10 | −7.59 (19) |
| C14—N1—C1—C6 | −172.24 (10) | C24—N3—C11—C12 | 13.84 (18) |
| N1—C1—C2—C3 | −178.49 (12) | C23—N3—C11—C12 | 173.09 (12) |
| C6—C1—C2—C3 | 0.86 (18) | C9—C10—C11—N3 | 179.62 (12) |
| C1—C2—C3—C4 | 0.58 (18) | C9—C10—C11—C12 | −1.04 (18) |
| C2—C3—C4—C5 | −1.15 (18) | N3—C11—C12—C13 | −179.78 (12) |
| C2—C3—C4—C20 | 179.44 (11) | C10—C11—C12—C13 | 0.87 (18) |
| C3—C4—C5—C6 | 0.24 (17) | C11—C12—C13—C8 | 0.40 (19) |
| C20—C4—C5—C6 | 179.67 (10) | C9—C8—C13—C12 | −1.47 (18) |
| C7—N2—C6—C5 | −178.08 (12) | C7—C8—C13—C12 | −178.60 (11) |
| C7—N2—C6—C1 | 1.17 (13) | C1—N1—C14—C15 | 108.78 (13) |
| C4—C5—C6—N2 | −179.65 (11) | C7—N1—C14—C15 | −63.17 (17) |
| C4—C5—C6—C1 | 1.16 (17) | C1—N1—C14—C19 | −69.23 (15) |
| N1—C1—C6—N2 | −1.61 (13) | C7—N1—C14—C19 | 118.82 (13) |
| C2—C1—C6—N2 | 178.89 (11) | C19—C14—C15—C16 | −1.54 (18) |
| N1—C1—C6—C5 | 177.73 (10) | N1—C14—C15—C16 | −179.49 (11) |
| C2—C1—C6—C5 | −1.76 (18) | C14—C15—C16—C17 | 0.87 (19) |
| C6—N2—C7—N1 | −0.27 (13) | C15—C16—C17—C18 | 0.43 (19) |
| C6—N2—C7—C8 | −177.42 (10) | C16—C17—C18—C19 | −1.10 (19) |
| C1—N1—C7—N2 | −0.73 (13) | C15—C14—C19—C18 | 0.88 (18) |
| C14—N1—C7—N2 | 172.19 (11) | N1—C14—C19—C18 | 178.86 (11) |
| C1—N1—C7—C8 | 176.31 (11) | C17—C18—C19—C14 | 0.45 (19) |
| C14—N1—C7—C8 | −10.77 (19) | C21—O1—C20—O2 | −3.58 (17) |
| N2—C7—C8—C13 | 169.33 (11) | C21—O1—C20—C4 | 176.91 (10) |
| N1—C7—C8—C13 | −7.41 (19) | C5—C4—C20—O2 | −1.07 (18) |
| N2—C7—C8—C9 | −7.78 (17) | C3—C4—C20—O2 | 178.36 (12) |
| N1—C7—C8—C9 | 175.48 (11) | C5—C4—C20—O1 | 178.43 (10) |
| C13—C8—C9—C10 | 1.30 (18) | C3—C4—C20—O1 | −2.14 (16) |

| | | | |
|---|---|---|---|
| C7—C8—C9—C10 | 178.65 (11) | C20—O1—C21—C22 | 177.00 (12) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C15—H15*A*···N2$^{i}$ | 0.95 | 2.57 | 3.4868 (16) | 164 |
| C18—H18*A*···O2$^{ii}$ | 0.95 | 2.50 | 3.2217 (17) | 133 |
| C19—H19*A*···O2$^{iii}$ | 0.95 | 2.38 | 3.3209 (16) | 170 |

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