

6-(3,5-Dimethylbenzyl)-5-ethyl-1-[(2-phenylethoxy)methyl]pyrimidine-2,4(1H,3H)dione

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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.044; wR factor = 0.123; data-to-parameter ratio = 14.3.

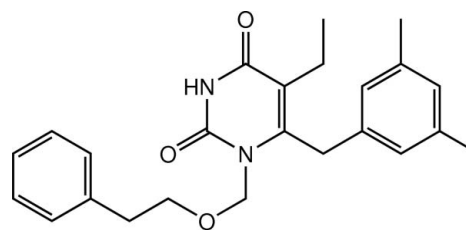
In the title pyrimidine derivative, $\text{C}_{24}\text{H}_{28}\text{N}_2\text{O}_3$, the uracil unit is essentially planar with an r.m.s. deviation of 0.0054 (1) Å for the eight non-H atoms. The pyrimidine ring is tilted by a dihedral angle of 77.08 (7)° with respect to the aromatic ring of the 3,5-dimethylbenzyl substituent, whereas it is nearly parallel to the benzene ring of the phenethoxymethyl unit, with a dihedral angle of 8.17 (8)°. An intramolecular C—H···O hydrogen bond generates an $S(6)$ ring motif. In the crystal, molecules are linked by a pair of amide–uracil N—H···O hydrogen bonds into an inversion $R_2^2(8)$ dimer. These dimers are stacked along the b axis through π – π interactions with a centroid–centroid distance of 3.9517 (8) Å. Weak C—H··· π interactions are also present.

Related literature

For bond-length data, see: Allen *et al.* (1987). For details of hydrogen-bond motifs, see: Bernstein *et al.* (1995). For background to anti-viral HIV therapies, see: El-Brollosy *et al.* (2007, 2008, 2009); Hopkins *et al.* (1996, 1999). For related structures, see: El-Brollosy *et al.* (2011, 2012).

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Experimental

Crystal data

$\text{C}_{24}\text{H}_{28}\text{N}_2\text{O}_3$
 $M_r = 392.48$
 Triclinic, $P\bar{1}$
 $a = 8.34310$ (1) Å
 $b = 8.47760$ (1) Å
 $c = 15.9821$ (2) Å
 $\alpha = 91.5660$ (1)°
 $\beta = 91.7820$ (1)°
 $\gamma = 109.8350$ (1)°
 $V = 1061.97$ (1) Å³
 $Z = 2$
 Cu $K\alpha$ radiation
 $\mu = 0.65$ mm⁻¹
 $T = 296$ K
 $0.58 \times 0.40 \times 0.31$ mm

Data collection

Bruker SMART APEXII CCD
 area-detector diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2009)
 $T_{\min} = 0.705$, $T_{\max} = 0.824$
 13575 measured reflections
 3863 independent reflections
 3542 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.123$
 $S = 1.05$
 3863 reflections
 270 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.27$ e Å⁻³
 $\Delta\rho_{\min} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg3 is the centroid of the C15–C20 ring.

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------------------|------------|-------------|-------------|---------------|
| N2—H1N2···O1 ⁱ | 0.889 (18) | 1.969 (18) | 2.8558 (15) | 175.7 (17) |
| C14—H14B···O3 | 0.97 | 2.36 | 3.0349 (18) | 126 |
| C21—H21B···Cg3 ⁱⁱ | 0.96 | 2.90 | 3.845 (3) | 170 |
| C22—H22C···Cg3 ⁱⁱⁱ | 0.96 | 2.92 | 3.861 (3) | 166 |

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

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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5086).

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supporting information

Acta Cryst. (2012). E68, o1031–o1032 [https://doi.org/10.1107/S1600536812009841]

6-(3,5-Dimethylbenzyl)-5-ethyl-1-[(2-phenylethoxy)methyl]-pyrimidine-2,4(1*H*,3*H*)dione

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S1. Comment

Non-nucleoside reverse transcriptase inhibitors (NNRTIs) are very promising therapies in the treatment of human immunodeficiency virus (HIV) (Hopkins *et al.*, 1996, 1999). In continuation to our interest in NNRTIs (El-Brollosy *et al.*, 2007, 2008, 2009), and as part of on-going structural studies of pyrimidine derivatives (El-Brollosy *et al.*, 2011), we synthesized the title compound (I) as a potential non-nucleoside reverse transcriptase inhibitor. It showed a good antiviral activity against HIV-1 (El-Brollosy *et al.*, 2009). Herein, the crystal structure of (I) was reported.

In the title pyrimidine derivative, C₂₄H₂₈N₂O₃, the uracil unit is essentially planar with an r.m.s. deviation of 0.0054 (1) Å for the eight non-H atoms (C1–C4/N1/N2/O1/O2). The 3,5-dimethylbenzyl unit is also planar with an r.m.s. deviation of 0.0049 (2) Å for the nine non-H atoms (C14–C22). The pyrimidine ring is tilted with the aromatic ring (C15–C20) of the 3,5-dimethylbenzyl substituent by a dihedral angle of 77.08 (7)°, whereas nearly parallel to the C8–C13 benzene ring of the phenylethoxymethyl unit with a dihedral angle of 8.17 (7)°. The dihedral angle between the two benzene rings (C8–C13 and C15–C20) is 82.14 (8)°. The orientation of the ethyl and phenethoxymethyl groups respected to the uracil can be indicated by the torsion angles C1–C2–C23–C24 = -85.87 (19)°, and C6–O3–C5–N1 = -67.31 (14)° and C5–O3–C6–C7 = 159.11 (12)°. Intramolecular weak C14—H14B···O3 interaction generates an S(6) ring motif (Fig. 1) (Bernstein *et al.*, 1995). The bond distances in (I) are within normal ranges (Allen *et al.*, 1987) and comparable to the related structures (El-Brollosy *et al.*, 2011, 2012).

In the crystal packing, (Fig. 2), the two molecules are linked by a pair of N2—H1N2···O1 hydrogen bonds (Table 1) into a centrosymmetric *R*₂²(8) dimer (Bernstein *et al.*, 1995). These dimers are stacked along the *b* axis (Fig. 2) through π – π interactions with a *Cg*1···*Cg*2^{iv,v} distance of 3.9517 (8) Å [symmetry codes: (iv) *x*, -1+*y*, *z*; (v) *x*, 1+*y*, *z*]. Weak C—H··· π interactions (Table 1) are also present.

S2. Experimental

5-Ethyl-6-(3,5-dimethylbenzyl)uracil (0.258 g, 1 mmol) was stirred in anhydrous CH₃CN (15 ml) under nitrogen and *N,O*-bis(trimethylsilyl)acetamide (0.87 ml, 3.5 mol) was added. After a clear solution was obtained (10 min), the reaction mixture was cooled to 223 K and trimethylsilyl trifluoromethanesulphonate (0.18 ml, 1 mmol) was added followed by dropwise addition of bis-(2-phenylethoxy)methane (0.512 g, 2 mmol). The mixture was stirred at room temperature for 4 hr. The reaction was quenched with saturated aqueous NaHCO₃ solution (5 ml) and evaporated under reduced pressure. The residue was extracted with ether (3 × 50 ml), the combined organic fractions were collected, dried (MgSO₄) and evaporated under reduced pressure. The residue was chromatographed on silica gel column with CHCl₃ to afford a white solid, which was recrystallized from ethanol to obtain the title compound as colorless crystals. Yield: 201 mg (51 %),

M.p.: 396–398 K.

S3. Refinement

The amide H atom was located in a difference map and refined isotropically. The remaining H atoms were placed in calculated positions with $d(\text{C—H}) = 0.93$ for aromatic, 0.97 for CH_2 and 0.96 Å for CH_3 atoms. The $U_{\text{iso}}(\text{H})$ values were constrained to be $1.5U_{\text{eq}}$ of the carrier atom for methyl H atoms and $1.2U_{\text{eq}}$ for the remaining H atoms. A rotating group model was used for the methyl groups.

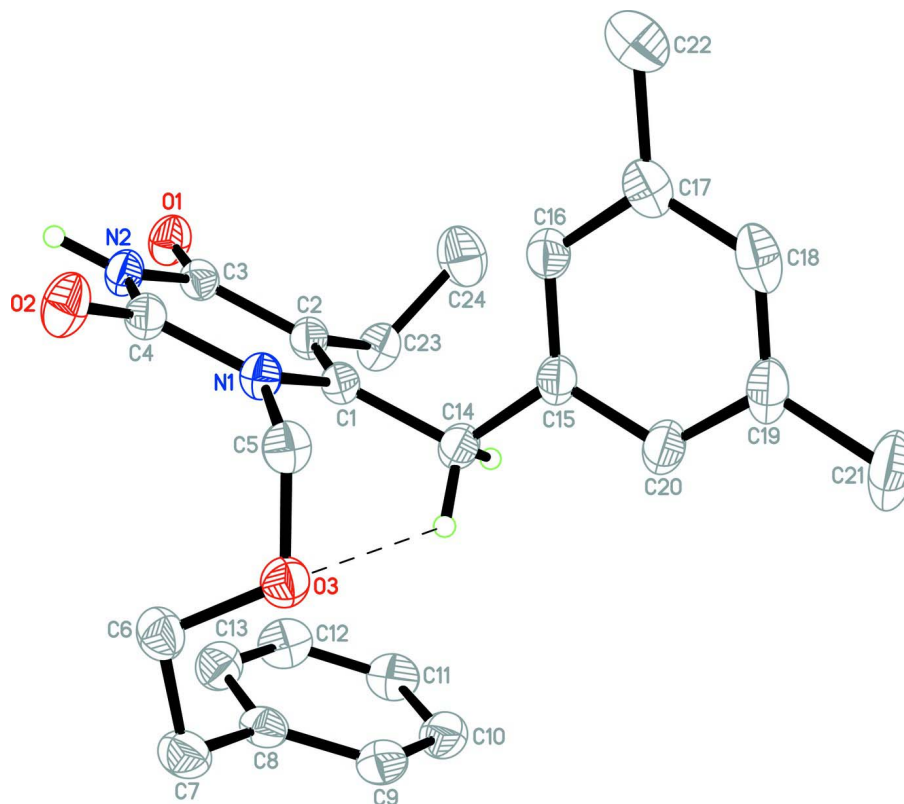


Figure 1

The structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. Only the non-aromatic benzyl H and amide H atoms were shown for clarity.

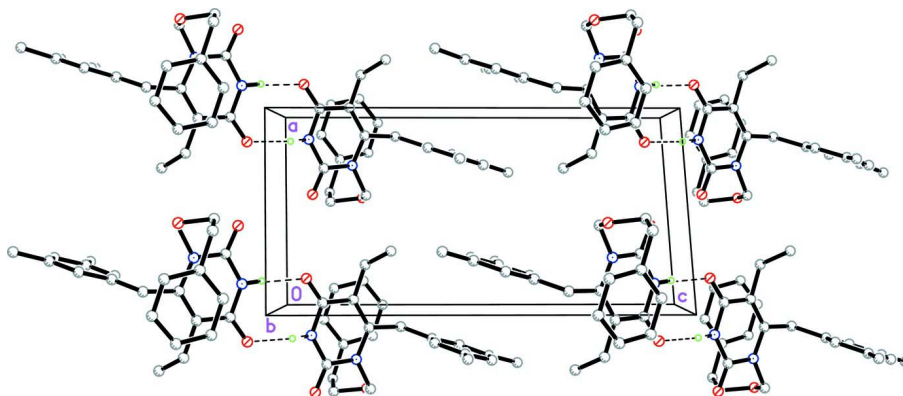


Figure 2

The crystal packing diagram of the title compound, viewed along the *b* axis. Only H atoms involving in intermolecular hydrogen bonds are drawn for clarify. N—H···O hydrogen bonds are shown as dashed lines.

6-(3,5-Dimethylbenzyl)-5-ethyl-1-[(2-phenylethoxy)methyl]pyrimidine- 2,4(1*H*,3*H*)dione

Crystal data

$C_{24}H_{28}N_2O_3$

$M_r = 392.48$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 8.34310(1) \text{ \AA}$

$b = 8.47760(1) \text{ \AA}$

$c = 15.9821(2) \text{ \AA}$

$\alpha = 91.5660(1)^\circ$

$\beta = 91.7820(1)^\circ$

$\gamma = 109.8350(1)^\circ$

$V = 1061.97(1) \text{ \AA}^3$

$Z = 2$

$F(000) = 420$

$D_x = 1.227 \text{ Mg m}^{-3}$

Melting point = 396–398 K

Cu $K\alpha$ radiation, $\lambda = 1.54178 \text{ \AA}$

Cell parameters from 3863 reflections

$\theta = 2.8\text{--}70.0^\circ$

$\mu = 0.65 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Block, colorless

$0.58 \times 0.40 \times 0.31 \text{ mm}$

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2009)

$T_{\min} = 0.705$, $T_{\max} = 0.824$

13575 measured reflections

3863 independent reflections

3542 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.022$

$\theta_{\max} = 70.0^\circ$, $\theta_{\min} = 2.8^\circ$

$h = -10 \rightarrow 10$

$k = -10 \rightarrow 10$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.123$

$S = 1.05$

3863 reflections

270 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 atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0599P)^2 + 0.2589P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.27 \text{ e \AA}^{-3}$

$$\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$$

Extinction correction: *SHELXTL* (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0158 (11)

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. 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 $F^2 > 2\text{sigma}(F^2)$ 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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|---------------|--------------|----------------------------------|
| N1 | 0.73374 (14) | 0.17782 (14) | 0.18049 (7) | 0.0389 (3) |
| N2 | 0.86631 (15) | 0.35816 (15) | 0.07678 (7) | 0.0416 (3) |
| H1N2 | 0.855 (2) | 0.413 (2) | 0.0316 (12) | 0.050 (4)* |
| O1 | 1.15299 (13) | 0.46537 (13) | 0.07074 (6) | 0.0516 (3) |
| O2 | 0.57921 (14) | 0.25880 (15) | 0.08011 (7) | 0.0586 (3) |
| O3 | 0.56324 (12) | -0.10907 (12) | 0.19600 (6) | 0.0465 (3) |
| C1 | 0.89379 (17) | 0.18893 (16) | 0.21512 (8) | 0.0372 (3) |
| C2 | 1.03943 (17) | 0.28209 (17) | 0.18009 (8) | 0.0398 (3) |
| C3 | 1.02860 (18) | 0.37579 (17) | 0.10647 (8) | 0.0400 (3) |
| C4 | 0.71675 (18) | 0.26506 (17) | 0.11021 (8) | 0.0408 (3) |
| C5 | 0.57628 (17) | 0.05835 (18) | 0.21083 (9) | 0.0439 (3) |
| H5A | 0.5718 | 0.0791 | 0.2706 | 0.053* |
| H5B | 0.4793 | 0.0771 | 0.1835 | 0.053* |
| C6 | 0.5432 (2) | -0.1640 (2) | 0.10923 (9) | 0.0496 (4) |
| H6A | 0.6137 | -0.0757 | 0.0755 | 0.060* |
| H6B | 0.4253 | -0.1918 | 0.0897 | 0.060* |
| C7 | 0.5968 (2) | -0.3174 (2) | 0.10163 (11) | 0.0545 (4) |
| H7A | 0.5223 | -0.4062 | 0.1337 | 0.065* |
| H7B | 0.5846 | -0.3567 | 0.0434 | 0.065* |
| C8 | 0.77867 (19) | -0.27984 (17) | 0.13271 (9) | 0.0457 (3) |
| C9 | 0.8179 (2) | -0.33678 (19) | 0.20831 (10) | 0.0511 (4) |
| H9A | 0.7302 | -0.4020 | 0.2402 | 0.061* |
| C10 | 0.9850 (2) | -0.2985 (2) | 0.23730 (11) | 0.0561 (4) |
| H10A | 1.0087 | -0.3380 | 0.2883 | 0.067* |
| C11 | 1.1160 (2) | -0.2024 (2) | 0.19109 (12) | 0.0579 (4) |
| H11A | 1.2285 | -0.1770 | 0.2105 | 0.070* |
| C12 | 1.0798 (2) | -0.1441 (2) | 0.11607 (12) | 0.0614 (4) |
| H12A | 1.1682 | -0.0782 | 0.0847 | 0.074* |
| C13 | 0.9130 (2) | -0.1827 (2) | 0.08704 (10) | 0.0545 (4) |
| H13A | 0.8901 | -0.1430 | 0.0360 | 0.065* |
| C14 | 0.89232 (19) | 0.09042 (18) | 0.29230 (8) | 0.0430 (3) |
| H14A | 1.0062 | 0.0871 | 0.3029 | 0.052* |

| | | | | |
|------|--------------|--------------|--------------|------------|
| H14B | 0.8170 | -0.0241 | 0.2806 | 0.052* |
| C15 | 0.83675 (18) | 0.15601 (18) | 0.37173 (8) | 0.0433 (3) |
| C16 | 0.8249 (2) | 0.31421 (19) | 0.38003 (9) | 0.0491 (4) |
| H16A | 0.8513 | 0.3851 | 0.3353 | 0.059* |
| C17 | 0.7738 (2) | 0.3697 (2) | 0.45442 (10) | 0.0573 (4) |
| C18 | 0.7354 (2) | 0.2618 (3) | 0.52015 (10) | 0.0642 (5) |
| H18A | 0.7011 | 0.2972 | 0.5699 | 0.077* |
| C19 | 0.7467 (2) | 0.1027 (2) | 0.51394 (9) | 0.0597 (4) |
| C20 | 0.7976 (2) | 0.0516 (2) | 0.43944 (9) | 0.0523 (4) |
| H20A | 0.8059 | -0.0549 | 0.4344 | 0.063* |
| C21 | 0.7013 (3) | -0.0151 (3) | 0.58581 (12) | 0.0859 (7) |
| H21A | 0.7585 | -0.0957 | 0.5808 | 0.129* |
| H21B | 0.5802 | -0.0724 | 0.5842 | 0.129* |
| H21C | 0.7364 | 0.0481 | 0.6380 | 0.129* |
| C22 | 0.7617 (3) | 0.5431 (3) | 0.46177 (14) | 0.0814 (6) |
| H22A | 0.6780 | 0.5439 | 0.5015 | 0.122* |
| H22B | 0.7287 | 0.5734 | 0.4081 | 0.122* |
| H22C | 0.8705 | 0.6222 | 0.4803 | 0.122* |
| C23 | 1.21678 (19) | 0.2957 (2) | 0.21143 (10) | 0.0509 (4) |
| H23A | 1.2136 | 0.1883 | 0.2322 | 0.061* |
| H23B | 1.2922 | 0.3205 | 0.1649 | 0.061* |
| C24 | 1.2888 (3) | 0.4295 (3) | 0.28019 (13) | 0.0758 (5) |
| H24A | 1.3994 | 0.4298 | 0.2989 | 0.114* |
| H24B | 1.2142 | 0.4065 | 0.3263 | 0.114* |
| H24C | 1.2987 | 0.5372 | 0.2591 | 0.114* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|------------|-------------|-------------|------------|-------------|-------------|
| N1 | 0.0382 (6) | 0.0428 (6) | 0.0351 (5) | 0.0130 (5) | 0.0022 (4) | 0.0040 (4) |
| N2 | 0.0461 (6) | 0.0448 (6) | 0.0340 (6) | 0.0152 (5) | 0.0002 (5) | 0.0083 (5) |
| O1 | 0.0464 (6) | 0.0569 (6) | 0.0462 (6) | 0.0096 (5) | 0.0055 (4) | 0.0146 (5) |
| O2 | 0.0461 (6) | 0.0739 (8) | 0.0567 (7) | 0.0212 (5) | -0.0057 (5) | 0.0165 (5) |
| O3 | 0.0450 (5) | 0.0460 (5) | 0.0447 (5) | 0.0105 (4) | 0.0018 (4) | 0.0049 (4) |
| C1 | 0.0413 (7) | 0.0390 (7) | 0.0321 (6) | 0.0148 (6) | -0.0005 (5) | -0.0003 (5) |
| C2 | 0.0419 (7) | 0.0420 (7) | 0.0349 (6) | 0.0133 (6) | 0.0006 (5) | 0.0021 (5) |
| C3 | 0.0442 (7) | 0.0393 (7) | 0.0346 (6) | 0.0120 (6) | 0.0025 (5) | 0.0006 (5) |
| C4 | 0.0435 (7) | 0.0438 (7) | 0.0364 (6) | 0.0165 (6) | -0.0012 (5) | 0.0019 (5) |
| C5 | 0.0387 (7) | 0.0503 (8) | 0.0418 (7) | 0.0135 (6) | 0.0060 (5) | 0.0033 (6) |
| C6 | 0.0455 (8) | 0.0550 (9) | 0.0478 (8) | 0.0177 (7) | -0.0073 (6) | -0.0035 (6) |
| C7 | 0.0501 (9) | 0.0463 (8) | 0.0630 (9) | 0.0129 (7) | -0.0099 (7) | -0.0063 (7) |
| C8 | 0.0472 (8) | 0.0377 (7) | 0.0527 (8) | 0.0161 (6) | -0.0016 (6) | -0.0064 (6) |
| C9 | 0.0487 (8) | 0.0435 (8) | 0.0588 (9) | 0.0122 (7) | 0.0035 (7) | 0.0046 (6) |
| C10 | 0.0566 (9) | 0.0526 (9) | 0.0604 (9) | 0.0208 (8) | -0.0064 (7) | 0.0038 (7) |
| C11 | 0.0437 (8) | 0.0571 (9) | 0.0740 (11) | 0.0190 (7) | -0.0016 (7) | -0.0018 (8) |
| C12 | 0.0506 (9) | 0.0626 (10) | 0.0719 (11) | 0.0190 (8) | 0.0167 (8) | 0.0082 (8) |
| C13 | 0.0589 (9) | 0.0562 (9) | 0.0520 (9) | 0.0240 (8) | 0.0054 (7) | 0.0045 (7) |
| C14 | 0.0465 (7) | 0.0439 (7) | 0.0388 (7) | 0.0153 (6) | 0.0008 (5) | 0.0076 (5) |

| | | | | | | |
|-----|-------------|-------------|-------------|-------------|-------------|--------------|
| C15 | 0.0442 (7) | 0.0470 (8) | 0.0339 (7) | 0.0093 (6) | -0.0027 (5) | 0.0054 (5) |
| C16 | 0.0574 (9) | 0.0488 (8) | 0.0370 (7) | 0.0127 (7) | 0.0015 (6) | 0.0039 (6) |
| C17 | 0.0604 (10) | 0.0636 (10) | 0.0446 (8) | 0.0177 (8) | -0.0007 (7) | -0.0067 (7) |
| C18 | 0.0635 (10) | 0.0863 (13) | 0.0354 (8) | 0.0165 (9) | 0.0033 (7) | -0.0063 (7) |
| C19 | 0.0591 (9) | 0.0743 (11) | 0.0346 (7) | 0.0082 (8) | -0.0019 (6) | 0.0100 (7) |
| C20 | 0.0575 (9) | 0.0543 (9) | 0.0394 (7) | 0.0113 (7) | -0.0019 (6) | 0.0115 (6) |
| C21 | 0.0950 (15) | 0.1073 (17) | 0.0425 (9) | 0.0158 (13) | 0.0066 (9) | 0.0258 (10) |
| C22 | 0.1037 (16) | 0.0772 (13) | 0.0676 (12) | 0.0374 (12) | 0.0066 (11) | -0.0150 (10) |
| C23 | 0.0451 (8) | 0.0563 (9) | 0.0510 (8) | 0.0163 (7) | 0.0024 (6) | 0.0109 (7) |
| C24 | 0.0590 (11) | 0.0903 (14) | 0.0677 (11) | 0.0142 (10) | -0.0150 (9) | -0.0076 (10) |

Geometric parameters (Å, °)

| | | | |
|----------|-------------|--------------|-------------|
| N1—C4 | 1.3913 (17) | C12—C13 | 1.379 (2) |
| N1—C1 | 1.4014 (17) | C12—H12A | 0.9300 |
| N1—C5 | 1.4664 (17) | C13—H13A | 0.9300 |
| N2—C4 | 1.3643 (18) | C14—C15 | 1.519 (2) |
| N2—C3 | 1.3783 (18) | C14—H14A | 0.9700 |
| N2—H1N2 | 0.887 (19) | C14—H14B | 0.9700 |
| O1—C3 | 1.2270 (17) | C15—C16 | 1.381 (2) |
| O2—C4 | 1.2141 (17) | C15—C20 | 1.3926 (19) |
| O3—C5 | 1.3992 (17) | C16—C17 | 1.398 (2) |
| O3—C6 | 1.4372 (18) | C16—H16A | 0.9300 |
| C1—C2 | 1.3524 (19) | C17—C18 | 1.385 (3) |
| C1—C14 | 1.5065 (17) | C17—C22 | 1.509 (3) |
| C2—C3 | 1.4548 (18) | C18—C19 | 1.385 (3) |
| C2—C23 | 1.5128 (19) | C18—H18A | 0.9300 |
| C5—H5A | 0.9700 | C19—C20 | 1.383 (2) |
| C5—H5B | 0.9700 | C19—C21 | 1.514 (2) |
| C6—C7 | 1.514 (2) | C20—H20A | 0.9300 |
| C6—H6A | 0.9700 | C21—H21A | 0.9600 |
| C6—H6B | 0.9700 | C21—H21B | 0.9600 |
| C7—C8 | 1.505 (2) | C21—H21C | 0.9600 |
| C7—H7A | 0.9700 | C22—H22A | 0.9600 |
| C7—H7B | 0.9700 | C22—H22B | 0.9600 |
| C8—C9 | 1.384 (2) | C22—H22C | 0.9600 |
| C8—C13 | 1.387 (2) | C23—C24 | 1.511 (3) |
| C9—C10 | 1.381 (2) | C23—H23A | 0.9700 |
| C9—H9A | 0.9300 | C23—H23B | 0.9700 |
| C10—C11 | 1.372 (2) | C24—H24A | 0.9600 |
| C10—H10A | 0.9300 | C24—H24B | 0.9600 |
| C11—C12 | 1.373 (3) | C24—H24C | 0.9600 |
| C11—H11A | 0.9300 | | |
| C4—N1—C1 | 121.92 (11) | C12—C13—C8 | 121.12 (15) |
| C4—N1—C5 | 116.53 (11) | C12—C13—H13A | 119.4 |
| C1—N1—C5 | 121.18 (11) | C8—C13—H13A | 119.4 |
| C4—N2—C3 | 126.71 (11) | C1—C14—C15 | 116.09 (11) |

| | | | |
|--------------|-------------|---------------|-------------|
| C4—N2—H1N2 | 115.1 (11) | C1—C14—H14A | 108.3 |
| C3—N2—H1N2 | 118.2 (11) | C15—C14—H14A | 108.3 |
| C5—O3—C6 | 114.94 (11) | C1—C14—H14B | 108.3 |
| C2—C1—N1 | 121.22 (12) | C15—C14—H14B | 108.3 |
| C2—C1—C14 | 122.80 (12) | H14A—C14—H14B | 107.4 |
| N1—C1—C14 | 115.98 (11) | C16—C15—C20 | 118.73 (14) |
| C1—C2—C3 | 118.98 (12) | C16—C15—C14 | 123.13 (12) |
| C1—C2—C23 | 124.62 (12) | C20—C15—C14 | 118.14 (13) |
| C3—C2—C23 | 116.39 (12) | C15—C16—C17 | 121.23 (14) |
| O1—C3—N2 | 120.07 (12) | C15—C16—H16A | 119.4 |
| O1—C3—C2 | 123.99 (13) | C17—C16—H16A | 119.4 |
| N2—C3—C2 | 115.93 (12) | C18—C17—C16 | 118.24 (16) |
| O2—C4—N2 | 122.00 (12) | C18—C17—C22 | 121.61 (16) |
| O2—C4—N1 | 122.80 (13) | C16—C17—C22 | 120.15 (16) |
| N2—C4—N1 | 115.20 (11) | C17—C18—C19 | 121.94 (15) |
| O3—C5—N1 | 112.94 (11) | C17—C18—H18A | 119.0 |
| O3—C5—H5A | 109.0 | C19—C18—H18A | 119.0 |
| N1—C5—H5A | 109.0 | C20—C19—C18 | 118.37 (15) |
| O3—C5—H5B | 109.0 | C20—C19—C21 | 120.28 (18) |
| N1—C5—H5B | 109.0 | C18—C19—C21 | 121.34 (17) |
| H5A—C5—H5B | 107.8 | C19—C20—C15 | 121.50 (16) |
| O3—C6—C7 | 107.77 (12) | C19—C20—H20A | 119.3 |
| O3—C6—H6A | 110.2 | C15—C20—H20A | 119.3 |
| C7—C6—H6A | 110.2 | C19—C21—H21A | 109.5 |
| O3—C6—H6B | 110.2 | C19—C21—H21B | 109.5 |
| C7—C6—H6B | 110.2 | H21A—C21—H21B | 109.5 |
| H6A—C6—H6B | 108.5 | C19—C21—H21C | 109.5 |
| C8—C7—C6 | 111.71 (12) | H21A—C21—H21C | 109.5 |
| C8—C7—H7A | 109.3 | H21B—C21—H21C | 109.5 |
| C6—C7—H7A | 109.3 | C17—C22—H22A | 109.5 |
| C8—C7—H7B | 109.3 | C17—C22—H22B | 109.5 |
| C6—C7—H7B | 109.3 | H22A—C22—H22B | 109.5 |
| H7A—C7—H7B | 107.9 | C17—C22—H22C | 109.5 |
| C9—C8—C13 | 117.69 (14) | H22A—C22—H22C | 109.5 |
| C9—C8—C7 | 121.57 (14) | H22B—C22—H22C | 109.5 |
| C13—C8—C7 | 120.71 (14) | C24—C23—C2 | 113.15 (14) |
| C10—C9—C8 | 121.20 (15) | C24—C23—H23A | 108.9 |
| C10—C9—H9A | 119.4 | C2—C23—H23A | 108.9 |
| C8—C9—H9A | 119.4 | C24—C23—H23B | 108.9 |
| C11—C10—C9 | 120.18 (15) | C2—C23—H23B | 108.9 |
| C11—C10—H10A | 119.9 | H23A—C23—H23B | 107.8 |
| C9—C10—H10A | 119.9 | C23—C24—H24A | 109.5 |
| C10—C11—C12 | 119.56 (15) | C23—C24—H24B | 109.5 |
| C10—C11—H11A | 120.2 | H24A—C24—H24B | 109.5 |
| C12—C11—H11A | 120.2 | C23—C24—H24C | 109.5 |
| C11—C12—C13 | 120.24 (16) | H24A—C24—H24C | 109.5 |
| C11—C12—H12A | 119.9 | H24B—C24—H24C | 109.5 |
| C13—C12—H12A | 119.9 | | |

| | | | |
|---------------|--------------|-----------------|--------------|
| C4—N1—C1—C2 | -1.78 (19) | C13—C8—C9—C10 | -0.1 (2) |
| C5—N1—C1—C2 | 170.99 (12) | C7—C8—C9—C10 | -178.47 (14) |
| C4—N1—C1—C14 | 178.71 (12) | C8—C9—C10—C11 | 0.0 (2) |
| C5—N1—C1—C14 | -8.52 (17) | C9—C10—C11—C12 | 0.3 (3) |
| N1—C1—C2—C3 | 1.72 (19) | C10—C11—C12—C13 | -0.4 (3) |
| C14—C1—C2—C3 | -178.80 (12) | C11—C12—C13—C8 | 0.4 (3) |
| N1—C1—C2—C23 | -177.60 (12) | C9—C8—C13—C12 | -0.1 (2) |
| C14—C1—C2—C23 | 1.9 (2) | C7—C8—C13—C12 | 178.30 (14) |
| C4—N2—C3—O1 | -179.20 (13) | C2—C1—C14—C15 | 109.79 (15) |
| C4—N2—C3—C2 | 1.31 (19) | N1—C1—C14—C15 | -70.71 (16) |
| C1—C2—C3—O1 | 179.10 (13) | C1—C14—C15—C16 | -14.3 (2) |
| C23—C2—C3—O1 | -1.5 (2) | C1—C14—C15—C20 | 166.30 (13) |
| C1—C2—C3—N2 | -1.44 (18) | C20—C15—C16—C17 | -0.3 (2) |
| C23—C2—C3—N2 | 177.93 (12) | C14—C15—C16—C17 | -179.73 (14) |
| C3—N2—C4—O2 | 179.05 (13) | C15—C16—C17—C18 | 0.1 (3) |
| C3—N2—C4—N1 | -1.3 (2) | C15—C16—C17—C22 | -179.92 (16) |
| C1—N1—C4—O2 | -178.91 (13) | C16—C17—C18—C19 | 0.2 (3) |
| C5—N1—C4—O2 | 8.0 (2) | C22—C17—C18—C19 | -179.82 (18) |
| C1—N1—C4—N2 | 1.45 (18) | C17—C18—C19—C20 | -0.2 (3) |
| C5—N1—C4—N2 | -171.63 (11) | C17—C18—C19—C21 | -178.89 (18) |
| C6—O3—C5—N1 | -67.31 (14) | C18—C19—C20—C15 | -0.1 (3) |
| C4—N1—C5—O3 | 108.67 (13) | C21—C19—C20—C15 | 178.66 (16) |
| C1—N1—C5—O3 | -64.47 (16) | C16—C15—C20—C19 | 0.3 (2) |
| C5—O3—C6—C7 | 159.11 (12) | C14—C15—C20—C19 | 179.75 (14) |
| O3—C6—C7—C8 | -58.84 (17) | C1—C2—C23—C24 | -85.87 (19) |
| C6—C7—C8—C9 | 106.27 (16) | C3—C2—C23—C24 | 94.80 (17) |
| C6—C7—C8—C13 | -72.06 (19) | | |

Hydrogen-bond geometry (\AA , $^\circ$)

Cg3 is the centroid of the C15–C20 ring.

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|--------------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N2—H1N2 \cdots O1 ⁱ | 0.889 (18) | 1.969 (18) | 2.8558 (15) | 175.7 (17) |
| C14—H14B \cdots O3 | 0.97 | 2.36 | 3.0349 (18) | 126 |
| C21—H21B \cdots Cg3 ⁱⁱ | 0.96 | 2.90 | 3.845 (3) | 170 |
| C22—H22C \cdots Cg3 ⁱⁱⁱ | 0.96 | 2.92 | 3.861 (3) | 166 |

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