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# 2-Hydroxy-2,3,5,10,11,11a-hexahydro-1*H*-pyrrolo[2,1-*c*][1,4]benzodiazepine-5,11-dione

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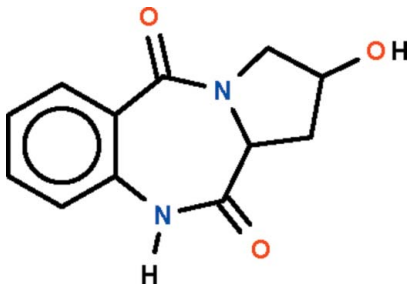
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.044; $wR$ factor = 0.110; data-to-parameter ratio = 7.9.

The seven-membered ring of the title compound, $C_{12}H_{12}N_2O_3$, which is fused with the phenylene ring, adopts a boat-shaped conformation (with the methine C atom as the prow and the phenylene C atoms as the stern); the H atom on the methine linkage exists in an axial position. The five-membered ring that is fused with the seven-membered ring adopts an envelope conformation (with the C atom bearing the hydroxy substituent representing the flap) [the deviation from the plane defined by the other four atoms is 0.200 (7) Å in one molecule and 0.627 (5) Å in the other]. The two independent molecules are disposed about a pseudo center of inversion and are connected by a pair of N—H···O hydrogen bonds. Adjacent dimers are linked by a pair of O—H···O hydrogen bonds, generating a chain running along the *b* axis.

## Related literature

For the structure of *cyclo*-(anthranoyl-prolyl), the compound without the hydroxy substituent, see: Feigel *et al.* (1990).




## Experimental

### Crystal data

$C_{12}H_{12}N_2O_3$
$M_r$ = 232.24
Monoclinic, $P2_1$
$a$ = 4.8366 (2) Å
$b$ = 25.7449 (11) Å
$c$ = 8.5420 (4) Å
$\beta$ = 96.509 (2)°
$V$ = 1056.77 (8) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 293 K
0.20 × 0.15 × 0.10 mm

### Data collection

Bruker APEXII CCD-detector diffractometer
7493 measured reflections
2435 independent reflections
2027 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.110
$S$ = 1.03
2435 reflections
309 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.34 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3o···O5$^{i}$ | 0.84 | 2.04 | 2.750 (4) | 142 |
| O6—H6o···O2$^{ii}$ | 0.82 | 1.94 | 2.746 (4) | 168 |
| N1—H1n···O4 | 0.88 | 2.08 | 2.908 (4) | 156 |
| N3—H3n···O1 | 0.88 | 2.10 | 2.922 (4) | 155 |

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); 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: *publCIF* (Westrip, 2010).

We thank Université Mohammed V-Agdal and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2124).

## References

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# supporting information

*Acta Cryst.* (2011). E**67**, o1906 [doi:10.1107/S1600536811025244]

# 2-Hydroxy-2,3,5,10,11,11a-hexahydro-1*H*-pyrrolo[2,1-*c*][1,4]benzodiazepine-5,11-dione

**Sarah Ourahou, Hafid Zouihri, Mohamed Massoui, El Mokhtar Essassi and Seik Weng Ng**

## S1. Comment

An early study reported the structure of pyrrolo[2,1-*c*][1,4]benzodiazepine-5,11-dione (Feigel *et al.*, 1990). The present structure (Scheme I) is the hydroxy-substituted analog. The seven-membered ring that is fused with the phenylene ring adopts a boat-shaped conformation. The five-membered ring adopts an envelope conformation. The two independent molecules are disposed about a false center-of-inversion and are connected by a pair of N—H···O hydrogen bonds (Fig. 1). Adjacent dimers are linked by a pair of O—H···O hydrogen bonds (Table 1) to generate a chain running along the *b*-axis of the monoclinic unit cell (Fig. 2).

## S2. Experimental

*N*-Carboxyanthranilic anhydride (isatoic anhydride) (3 g,18.4 mmol) and *allo*-4-hydroxy-*L*-proline (2.41 g, 18.4 mmol) were heated in DFM (60 ml). The solvent was evaporated under reduced pressure and the residue was washed with water (60 ml). The product isolated was recrystallized from ethanol to furnish colorless crystals.

## S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H = 0.93–0.98 Å) and were included in the refinement in the riding model approximation, with $U_{iso}$(H) set to 1.2$U_{eq}$(C). The oxygen- and nitrogen-bound H atoms were similarly treated (N—H = 0.88, O—H = 0.84 Å). Some 1461 Friedel pairs were merged. The absolute configuration was assumed to be that of the *allo*-*L*-proline reactant.

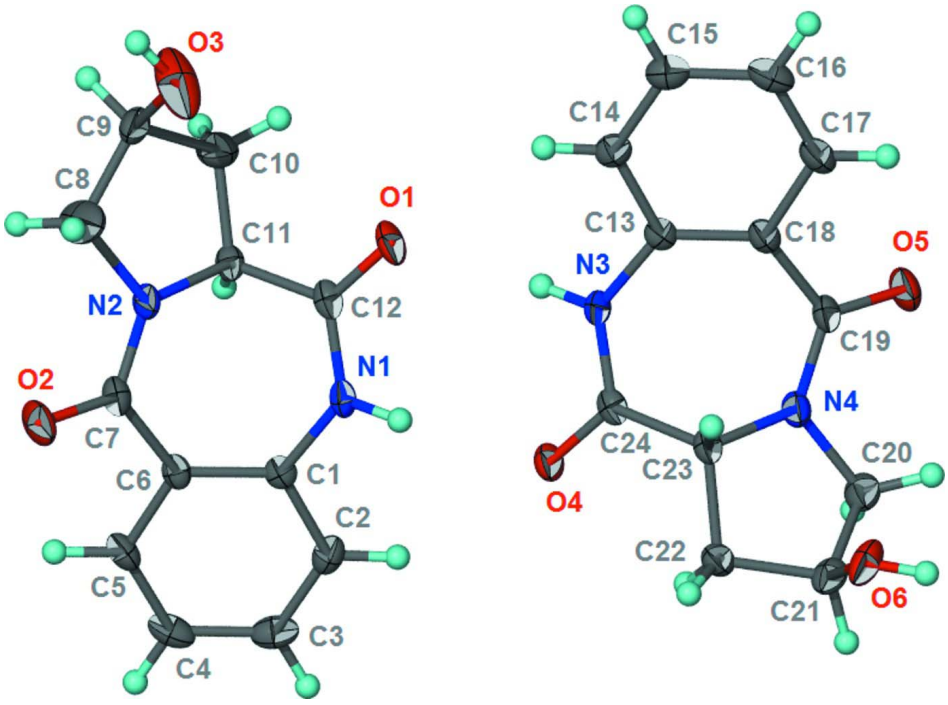


**Figure 1**

Thermal ellipsoid plot (Barbour, 2001) of the two independent molecules of $C_{12}H_{12}N_2O_3$ at the 50% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

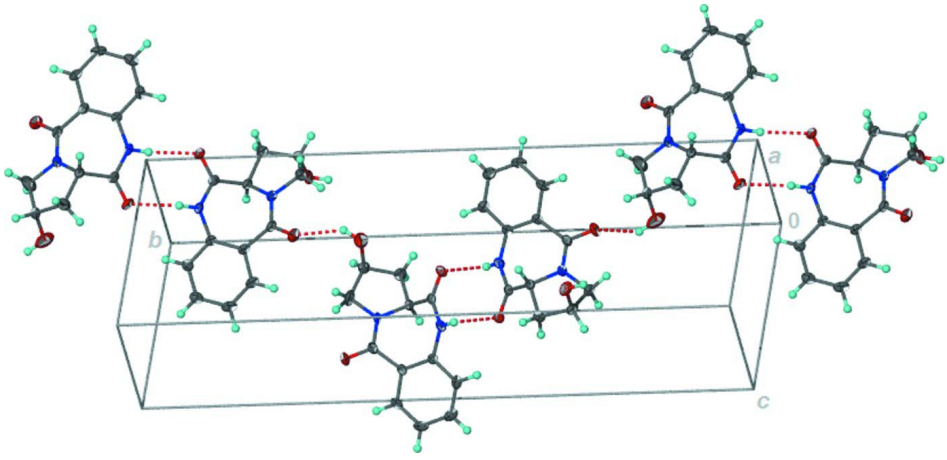


**Figure 2**

The hydrogen-bonded chain structure.

### 2-Hydroxy-2,3,5,10,11,11a-hexahydro-1*H*-pyrrolo[2,1- *c*][1,4]benzodiazepine-5,11-dione

*Crystal data*

$C_{12}H_{12}N_2O_3$
$M_r = 232.24$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 4.8366$ (2) Å
$b = 25.7449$ (11) Å
$c = 8.5420$ (4) Å
$\beta = 96.509$ (2)°
$V = 1056.77$ (8) Å$^3$
$Z = 4$
$F(000) = 488$
$D_x = 1.460$ Mg m$^{-3}$

Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 2147 reflections
$\theta$ = 2.5–27.3°
$\mu$ = 0.11 mm$^{-1}$
$T$ = 293 K
Prism, colorless
0.20 × 0.15 × 0.10 mm

*Data collection*

Bruker APEXII CCD-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
7493 measured reflections
2435 independent reflections
2027 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 2.4°
$h$ = −6→6
$k$ = −19→33
$l$ = −11→9

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.110
$S$ = 1.03
2435 reflections
309 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 0.2538P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.40 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.34 e Å$^{-3}$

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

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.7223 (5) | 0.49980 (10) | 0.5932 (3) | 0.0327 (6) |
| O2 | 0.4033 (6) | 0.65398 (10) | 0.9439 (3) | 0.0395 (7) |
| O3 | 0.8361 (7) | 0.63368 (14) | 0.4475 (6) | 0.0754 (13) |
| H3O | 0.8909 | 0.6615 | 0.4102 | 0.113* |
| O4 | 0.6970 (4) | 0.39402 (9) | 0.8727 (3) | 0.0275 (5) |
| O5 | 1.0501 (6) | 0.23833 (10) | 0.5383 (3) | 0.0386 (7) |
| O6 | 1.3550 (5) | 0.25061 (10) | 1.0454 (3) | 0.0363 (6) |
| H6O | 1.4044 | 0.2201 | 1.0495 | 0.054* |
| N1 | 0.4993 (6) | 0.49948 (11) | 0.8102 (3) | 0.0241 (6) |
| H1N | 0.6015 | 0.4725 | 0.8434 | 0.029* |
| N2 | 0.4375 (6) | 0.60987 (11) | 0.7194 (3) | 0.0257 (6) |
| N3 | 0.9248 (6) | 0.39419 (11) | 0.6586 (3) | 0.0239 (6) |
| H3N | 0.8207 | 0.4208 | 0.6241 | 0.029* |
| N4 | 1.0092 (6) | 0.28335 (11) | 0.7599 (3) | 0.0265 (6) |
| C1 | 0.3057 (6) | 0.51590 (13) | 0.9102 (4) | 0.0211 (7) |
| C2 | 0.1926 (7) | 0.47711 (14) | 0.9988 (4) | 0.0281 (8) |
| H2 | 0.2441 | 0.4427 | 0.9865 | 0.034* |
| C3 | 0.0073 (8) | 0.48915 (16) | 1.1031 (4) | 0.0336 (9) |
| H3 | −0.0655 | 0.4629 | 1.1609 | 0.040* |
| C4 | −0.0728 (7) | 0.54019 (17) | 1.1234 (4) | 0.0335 (9) |
| H4 | −0.2035 | 0.5481 | 1.1916 | 0.040* |
| C5 | 0.0436 (7) | 0.57897 (14) | 1.0412 (4) | 0.0280 (8) |

| | | | | |
|---|---|---|---|---|
| H5 | −0.0053 | 0.6133 | 1.0573 | 0.034* |
| C6 | 0.2341 (6) | 0.56785 (13) | 0.9338 (4) | 0.0215 (7) |
| C7 | 0.3665 (7) | 0.61335 (14) | 0.8647 (4) | 0.0266 (8) |
| C8 | 0.5274 (12) | 0.65602 (18) | 0.6375 (6) | 0.0571 (14) |
| H8A | 0.7034 | 0.6689 | 0.6888 | 0.069* |
| H8B | 0.3898 | 0.6834 | 0.6367 | 0.069* |
| C9 | 0.5579 (7) | 0.63853 (14) | 0.4722 (4) | 0.0302 (8) |
| H9 | 0.4641 | 0.6631 | 0.3962 | 0.036* |
| C10 | 0.4224 (9) | 0.58547 (16) | 0.4508 (4) | 0.0379 (9) |
| H10A | 0.5437 | 0.5618 | 0.4027 | 0.045* |
| H10B | 0.2481 | 0.5881 | 0.3829 | 0.045* |
| C11 | 0.3702 (6) | 0.56560 (13) | 0.6127 (4) | 0.0226 (7) |
| H11 | 0.1733 | 0.5565 | 0.6120 | 0.027* |
| C12 | 0.5502 (6) | 0.51928 (13) | 0.6704 (4) | 0.0229 (7) |
| C13 | 1.1233 (6) | 0.37765 (13) | 0.5599 (4) | 0.0217 (7) |
| C14 | 1.2309 (7) | 0.41619 (14) | 0.4691 (4) | 0.0262 (7) |
| H14 | 1.1749 | 0.4505 | 0.4791 | 0.031* |
| C15 | 1.4186 (7) | 0.40417 (16) | 0.3649 (4) | 0.0324 (9) |
| H15 | 1.4902 | 0.4303 | 0.3060 | 0.039* |
| C16 | 1.5008 (7) | 0.35323 (17) | 0.3478 (4) | 0.0341 (9) |
| H16 | 1.6315 | 0.3450 | 0.2798 | 0.041* |
| C17 | 1.3881 (7) | 0.31511 (16) | 0.4319 (4) | 0.0300 (8) |
| H17 | 1.4390 | 0.2808 | 0.4173 | 0.036* |
| C18 | 1.1984 (7) | 0.32618 (13) | 0.5392 (4) | 0.0247 (7) |
| C19 | 1.0782 (7) | 0.27964 (13) | 0.6126 (4) | 0.0256 (7) |
| C20 | 0.9495 (7) | 0.23740 (14) | 0.8527 (4) | 0.0319 (8) |
| H20A | 1.0455 | 0.2070 | 0.8193 | 0.038* |
| H20B | 0.7513 | 0.2304 | 0.8439 | 0.038* |
| C21 | 1.0603 (7) | 0.25316 (14) | 1.0199 (4) | 0.0297 (8) |
| H21 | 0.9728 | 0.2334 | 1.0990 | 0.036* |
| C22 | 0.9835 (7) | 0.30997 (14) | 1.0207 (4) | 0.0257 (7) |
| H22A | 1.0910 | 0.3281 | 1.1067 | 0.031* |
| H22B | 0.7870 | 0.3144 | 1.0303 | 0.031* |
| C23 | 1.0551 (6) | 0.32964 (12) | 0.8612 (4) | 0.0224 (7) |
| H23 | 1.2516 | 0.3398 | 0.8696 | 0.027* |
| C24 | 0.8747 (6) | 0.37459 (13) | 0.7980 (4) | 0.0221 (7) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0281 (12) | 0.0261 (14) | 0.0466 (15) | 0.0027 (10) | 0.0160 (11) | −0.0110 (12) |
| O2 | 0.0514 (16) | 0.0236 (15) | 0.0462 (17) | −0.0087 (12) | 0.0166 (13) | −0.0161 (12) |
| O3 | 0.053 (2) | 0.048 (2) | 0.135 (4) | −0.0167 (17) | 0.057 (2) | −0.031 (2) |
| O4 | 0.0263 (12) | 0.0226 (13) | 0.0352 (14) | 0.0035 (10) | 0.0101 (10) | −0.0022 (11) |
| O5 | 0.0516 (16) | 0.0244 (15) | 0.0418 (17) | 0.0011 (12) | 0.0137 (13) | −0.0114 (12) |
| O6 | 0.0276 (12) | 0.0255 (14) | 0.0564 (17) | 0.0064 (10) | 0.0072 (11) | 0.0143 (13) |
| N1 | 0.0227 (13) | 0.0198 (15) | 0.0295 (15) | 0.0064 (11) | 0.0014 (11) | −0.0034 (12) |
| N2 | 0.0334 (15) | 0.0139 (15) | 0.0308 (16) | −0.0040 (11) | 0.0084 (13) | −0.0038 (12) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N3 | 0.0249 (13) | 0.0200 (15) | 0.0269 (14) | 0.0083 (11) | 0.0030 (11) | 0.0014 (12) |
| N4 | 0.0299 (15) | 0.0195 (16) | 0.0313 (15) | 0.0002 (11) | 0.0090 (12) | −0.0029 (13) |
| C1 | 0.0179 (14) | 0.0233 (17) | 0.0212 (16) | −0.0014 (13) | −0.0012 (12) | −0.0023 (13) |
| C2 | 0.0306 (18) | 0.0210 (18) | 0.0313 (18) | −0.0009 (14) | −0.0031 (15) | 0.0015 (15) |
| C3 | 0.0305 (18) | 0.044 (2) | 0.0262 (17) | −0.0118 (16) | 0.0016 (15) | 0.0053 (16) |
| C4 | 0.0247 (17) | 0.052 (3) | 0.0248 (18) | −0.0047 (17) | 0.0069 (14) | −0.0074 (17) |
| C5 | 0.0266 (16) | 0.031 (2) | 0.0259 (18) | 0.0038 (14) | 0.0028 (14) | −0.0107 (15) |
| C6 | 0.0194 (14) | 0.0204 (17) | 0.0240 (16) | −0.0003 (13) | −0.0004 (12) | −0.0053 (13) |
| C7 | 0.0252 (16) | 0.0212 (19) | 0.0336 (19) | 0.0018 (13) | 0.0045 (14) | −0.0059 (15) |
| C8 | 0.094 (4) | 0.029 (3) | 0.052 (3) | −0.028 (2) | 0.026 (3) | −0.005 (2) |
| C9 | 0.0321 (18) | 0.0222 (18) | 0.038 (2) | 0.0071 (14) | 0.0096 (15) | 0.0070 (15) |
| C10 | 0.049 (2) | 0.034 (2) | 0.032 (2) | −0.0083 (18) | 0.0119 (17) | −0.0012 (17) |
| C11 | 0.0207 (15) | 0.0187 (17) | 0.0287 (17) | −0.0016 (12) | 0.0037 (12) | −0.0033 (14) |
| C12 | 0.0155 (14) | 0.0201 (17) | 0.0329 (18) | −0.0039 (12) | 0.0022 (13) | −0.0083 (14) |
| C13 | 0.0186 (15) | 0.0252 (18) | 0.0207 (16) | 0.0009 (13) | 0.0001 (12) | −0.0009 (14) |
| C14 | 0.0265 (16) | 0.0284 (19) | 0.0230 (17) | −0.0016 (14) | −0.0009 (13) | 0.0010 (14) |
| C15 | 0.0309 (18) | 0.039 (2) | 0.0270 (18) | −0.0058 (16) | 0.0024 (15) | 0.0028 (16) |
| C16 | 0.0264 (17) | 0.052 (3) | 0.0243 (18) | 0.0002 (17) | 0.0055 (14) | −0.0045 (17) |
| C17 | 0.0283 (17) | 0.038 (2) | 0.0245 (17) | 0.0081 (15) | 0.0038 (14) | −0.0052 (15) |
| C18 | 0.0236 (16) | 0.0271 (19) | 0.0235 (17) | 0.0034 (13) | 0.0028 (13) | −0.0032 (14) |
| C19 | 0.0255 (16) | 0.0206 (18) | 0.0310 (18) | 0.0047 (13) | 0.0054 (14) | −0.0035 (14) |
| C20 | 0.0338 (17) | 0.025 (2) | 0.038 (2) | −0.0038 (15) | 0.0113 (15) | 0.0012 (16) |
| C21 | 0.0269 (16) | 0.0259 (19) | 0.038 (2) | −0.0012 (14) | 0.0107 (14) | 0.0073 (15) |
| C22 | 0.0259 (16) | 0.028 (2) | 0.0230 (17) | 0.0043 (14) | 0.0034 (13) | −0.0016 (14) |
| C23 | 0.0197 (15) | 0.0201 (18) | 0.0280 (17) | 0.0001 (12) | 0.0046 (13) | −0.0038 (13) |
| C24 | 0.0180 (14) | 0.0209 (17) | 0.0274 (17) | −0.0046 (12) | 0.0018 (13) | −0.0078 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C12 | 1.226 (4) | C8—C9 | 1.505 (6) |
| O2—C7 | 1.247 (4) | C8—H8A | 0.9700 |
| O3—C9 | 1.391 (4) | C8—H8B | 0.9700 |
| O3—H3O | 0.8400 | C9—C10 | 1.517 (5) |
| O4—C24 | 1.233 (4) | C9—H9 | 0.9800 |
| O5—C19 | 1.238 (4) | C10—C11 | 1.522 (5) |
| O6—C21 | 1.419 (4) | C10—H10A | 0.9700 |
| O6—H6O | 0.8200 | C10—H10B | 0.9700 |
| N1—C12 | 1.347 (4) | C11—C12 | 1.525 (5) |
| N1—C1 | 1.402 (4) | C11—H11 | 0.9800 |
| N1—H1N | 0.8800 | C13—C18 | 1.391 (5) |
| N2—C7 | 1.328 (4) | C13—C14 | 1.396 (5) |
| N2—C8 | 1.470 (5) | C14—C15 | 1.377 (5) |
| N2—C11 | 1.472 (4) | C14—H14 | 0.9300 |
| N3—C24 | 1.340 (4) | C15—C16 | 1.383 (6) |
| N3—C13 | 1.413 (4) | C15—H15 | 0.9300 |
| N3—H3N | 0.8800 | C16—C17 | 1.365 (6) |
| N4—C19 | 1.341 (4) | C16—H16 | 0.9300 |
| N4—C20 | 1.471 (5) | C17—C18 | 1.398 (5) |

| | | | |
|---|---|---|---|
| N4—C23 | 1.475 (4) | C17—H17 | 0.9300 |
| C1—C2 | 1.401 (5) | C18—C19 | 1.500 (5) |
| C1—C6 | 1.402 (5) | C20—C21 | 1.522 (5) |
| C2—C3 | 1.369 (5) | C20—H20A | 0.9700 |
| C2—H2 | 0.9300 | C20—H20B | 0.9700 |
| C3—C4 | 1.386 (6) | C21—C22 | 1.509 (5) |
| C3—H3 | 0.9300 | C21—H21 | 0.9800 |
| C4—C5 | 1.377 (5) | C22—C23 | 1.530 (5) |
| C4—H4 | 0.9300 | C22—H22A | 0.9700 |
| C5—C6 | 1.401 (4) | C22—H22B | 0.9700 |
| C5—H5 | 0.9300 | C23—C24 | 1.512 (5) |
| C6—C7 | 1.489 (5) | C23—H23 | 0.9800 |
| | | | |
| C9—O3—H3O | 109.5 | C10—C11—C12 | 114.1 (3) |
| C21—O6—H6O | 109.5 | N2—C11—H11 | 109.6 |
| C12—N1—C1 | 128.9 (3) | C10—C11—H11 | 109.6 |
| C12—N1—H1N | 115.6 | C12—C11—H11 | 109.6 |
| C1—N1—H1N | 115.6 | O1—C12—N1 | 121.7 (3) |
| C7—N2—C8 | 120.8 (3) | O1—C12—C11 | 122.9 (3) |
| C7—N2—C11 | 124.6 (3) | N1—C12—C11 | 115.3 (3) |
| C8—N2—C11 | 112.8 (3) | C18—C13—C14 | 119.2 (3) |
| C24—N3—C13 | 128.3 (3) | C18—C13—N3 | 124.6 (3) |
| C24—N3—H3N | 115.8 | C14—C13—N3 | 116.0 (3) |
| C13—N3—H3N | 115.8 | C15—C14—C13 | 120.9 (3) |
| C19—N4—C20 | 122.2 (3) | C15—C14—H14 | 119.5 |
| C19—N4—C23 | 124.8 (3) | C13—C14—H14 | 119.5 |
| C20—N4—C23 | 111.1 (3) | C14—C15—C16 | 120.0 (3) |
| C2—C1—C6 | 119.0 (3) | C14—C15—H15 | 120.0 |
| C2—C1—N1 | 116.3 (3) | C16—C15—H15 | 120.0 |
| C6—C1—N1 | 124.6 (3) | C17—C16—C15 | 119.3 (3) |
| C3—C2—C1 | 121.0 (3) | C17—C16—H16 | 120.3 |
| C3—C2—H2 | 119.5 | C15—C16—H16 | 120.3 |
| C1—C2—H2 | 119.5 | C16—C17—C18 | 121.9 (4) |
| C2—C3—C4 | 120.6 (4) | C16—C17—H17 | 119.0 |
| C2—C3—H3 | 119.7 | C18—C17—H17 | 119.0 |
| C4—C3—H3 | 119.7 | C13—C18—C17 | 118.6 (3) |
| C5—C4—C3 | 119.2 (3) | C13—C18—C19 | 126.1 (3) |
| C5—C4—H4 | 120.4 | C17—C18—C19 | 115.2 (3) |
| C3—C4—H4 | 120.4 | O5—C19—N4 | 121.2 (3) |
| C4—C5—C6 | 121.5 (3) | O5—C19—C18 | 119.8 (3) |
| C4—C5—H5 | 119.2 | N4—C19—C18 | 119.0 (3) |
| C6—C5—H5 | 119.2 | N4—C20—C21 | 102.8 (3) |
| C5—C6—C1 | 118.7 (3) | N4—C20—H20A | 111.2 |
| C5—C6—C7 | 116.3 (3) | C21—C20—H20A | 111.2 |
| C1—C6—C7 | 124.7 (3) | N4—C20—H20B | 111.2 |
| O2—C7—N2 | 121.8 (3) | C21—C20—H20B | 111.2 |
| O2—C7—C6 | 119.1 (3) | H20A—C20—H20B | 109.1 |
| N2—C7—C6 | 119.1 (3) | O6—C21—C22 | 106.7 (3) |

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| N2—C8—C9 | 105.7 (3) | O6—C21—C20 | 111.9 (3) |
| N2—C8—H8A | 110.6 | C22—C21—C20 | 101.7 (3) |
| C9—C8—H8A | 110.6 | O6—C21—H21 | 112.0 |
| N2—C8—H8B | 110.6 | C22—C21—H21 | 112.0 |
| C9—C8—H8B | 110.6 | C20—C21—H21 | 112.0 |
| H8A—C8—H8B | 108.7 | C21—C22—C23 | 103.7 (3) |
| O3—C9—C8 | 111.6 (4) | C21—C22—H22A | 111.0 |
| O3—C9—C10 | 108.2 (3) | C23—C22—H22A | 111.0 |
| C8—C9—C10 | 107.2 (3) | C21—C22—H22B | 111.0 |
| O3—C9—H9 | 109.9 | C23—C22—H22B | 111.0 |
| C8—C9—H9 | 109.9 | H22A—C22—H22B | 109.0 |
| C10—C9—H9 | 109.9 | N4—C23—C24 | 111.5 (3) |
| C9—C10—C11 | 107.9 (3) | N4—C23—C22 | 102.8 (3) |
| C9—C10—H10A | 110.1 | C24—C23—C22 | 113.1 (3) |
| C11—C10—H10A | 110.1 | N4—C23—H23 | 109.8 |
| C9—C10—H10B | 110.1 | C24—C23—H23 | 109.8 |
| C11—C10—H10B | 110.1 | C22—C23—H23 | 109.8 |
| H10A—C10—H10B | 108.4 | O4—C24—N3 | 120.9 (3) |
| N2—C11—C10 | 104.7 (3) | O4—C24—C23 | 122.2 (3) |
| N2—C11—C12 | 109.1 (2) | N3—C24—C23 | 116.8 (3) |

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| C12—N1—C1—C2 | 148.1 (3) | C24—N3—C13—C18 | 36.1 (5) |
| C12—N1—C1—C6 | −36.1 (5) | C24—N3—C13—C14 | −149.2 (3) |
| C6—C1—C2—C3 | 2.3 (5) | C18—C13—C14—C15 | −2.8 (4) |
| N1—C1—C2—C3 | 178.3 (3) | N3—C13—C14—C15 | −177.8 (3) |
| C1—C2—C3—C4 | 0.0 (5) | C13—C14—C15—C16 | 0.7 (5) |
| C2—C3—C4—C5 | −2.2 (5) | C14—C15—C16—C17 | 1.8 (5) |
| C3—C4—C5—C6 | 2.1 (5) | C15—C16—C17—C18 | −2.2 (5) |
| C4—C5—C6—C1 | 0.2 (5) | C14—C13—C18—C17 | 2.4 (4) |
| C4—C5—C6—C7 | −174.0 (3) | N3—C13—C18—C17 | 176.9 (3) |
| C2—C1—C6—C5 | −2.3 (4) | C14—C13—C18—C19 | −172.5 (3) |
| N1—C1—C6—C5 | −178.0 (3) | N3—C13—C18—C19 | 2.0 (5) |
| C2—C1—C6—C7 | 171.3 (3) | C16—C17—C18—C13 | 0.1 (5) |
| N1—C1—C6—C7 | −4.3 (5) | C16—C17—C18—C19 | 175.6 (3) |
| C8—N2—C7—O2 | −8.4 (6) | C20—N4—C19—O5 | 12.2 (5) |
| C11—N2—C7—O2 | −172.4 (3) | C23—N4—C19—O5 | 175.1 (3) |
| C8—N2—C7—C6 | 170.3 (4) | C20—N4—C19—C18 | −166.2 (3) |
| C11—N2—C7—C6 | 6.4 (5) | C23—N4—C19—C18 | −3.2 (5) |
| C5—C6—C7—O2 | 30.1 (5) | C13—C18—C19—O5 | 145.2 (3) |
| C1—C6—C7—O2 | −143.7 (3) | C17—C18—C19—O5 | −29.8 (4) |
| C5—C6—C7—N2 | −148.7 (3) | C13—C18—C19—N4 | −36.4 (5) |
| C1—C6—C7—N2 | 37.5 (5) | C17—C18—C19—N4 | 148.5 (3) |
| C7—N2—C8—C9 | −173.7 (3) | C19—N4—C20—C21 | 145.8 (3) |
| C11—N2—C8—C9 | −8.0 (5) | C23—N4—C20—C21 | −19.3 (3) |
| N2—C8—C9—O3 | −105.8 (4) | N4—C20—C21—O6 | −76.1 (3) |
| N2—C8—C9—C10 | 12.6 (5) | N4—C20—C21—C22 | 37.4 (3) |
| O3—C9—C10—C11 | 107.7 (4) | O6—C21—C22—C23 | 75.0 (3) |
| C8—C9—C10—C11 | −12.9 (5) | C20—C21—C22—C23 | −42.3 (3) |

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| C7—N2—C11—C10 | 165.1 (3) | C19—N4—C23—C24 | 67.3 (4) |
| C8—N2—C11—C10 | 0.1 (4) | C20—N4—C23—C24 | −128.1 (3) |
| C7—N2—C11—C12 | −72.3 (4) | C19—N4—C23—C22 | −171.3 (3) |
| C8—N2—C11—C12 | 122.7 (4) | C20—N4—C23—C22 | −6.7 (3) |
| C9—C10—C11—N2 | 7.9 (4) | C21—C22—C23—N4 | 30.4 (3) |
| C9—C10—C11—C12 | −111.4 (3) | C21—C22—C23—C24 | 150.7 (3) |
| C1—N1—C12—O1 | −177.9 (3) | C13—N3—C24—O4 | 178.4 (3) |
| C1—N1—C12—C11 | −0.6 (5) | C13—N3—C24—C23 | 1.2 (5) |
| N2—C11—C12—O1 | −116.6 (3) | N4—C23—C24—O4 | 117.9 (3) |
| C10—C11—C12—O1 | 0.1 (4) | C22—C23—C24—O4 | 2.7 (4) |
| N2—C11—C12—N1 | 66.1 (4) | N4—C23—C24—N3 | −64.9 (4) |
| C10—C11—C12—N1 | −177.1 (3) | C22—C23—C24—N3 | 179.9 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3o···O5$^{i}$ | 0.84 | 2.04 | 2.750 (4) | 142 |
| O6—H6o···O2$^{ii}$ | 0.82 | 1.94 | 2.746 (4) | 168 |
| N1—H1n···O4 | 0.88 | 2.08 | 2.908 (4) | 156 |
| N3—H3n···O1 | 0.88 | 2.10 | 2.922 (4) | 155 |

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