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# Diethyl 1,4-dioxo-1,2,2a,3,4,10b-hexahydro-5*H*,10*H*-2,3,4a,10a-tetraazabenzo[*g*]cyclopenta[*cd*]azulene-2a,10b-dicarboxylate

**Jing Qin**

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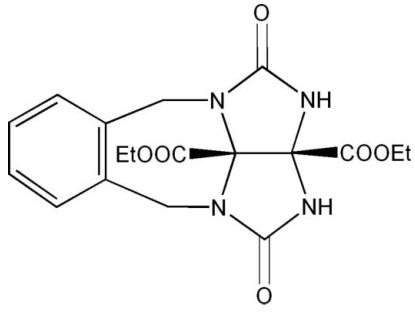
Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.049; $wR$ factor = 0.144; data-to-parameter ratio = 13.9.

In the title compound, $C_{18}H_{20}N_4O_6$, the dihedral angle between the two fused five-membered rings in the glycoluril unit is 64.42 (2)°. The crystal structure features intermolecular N—H···O and C—H···O interactions. An intramolecular C—H···O contact is also present.

## Related literature

For the preparation of the title compound, see: Wu *et al.* (2002*a*). For crystal engineering studies of glycoluril and its derivatives, see: Chen *et al.* (2007); Wang *et al.* (2006); Johnson *et al.* (2002); Wu *et al.* (2002*b*).




## Experimental

### Crystal data

$C_{18}H_{20}N_4O_6$
$M_r$ = 388.38
Triclinic, $P\overline{1}$
$a$ = 8.1394 (5) Å
$b$ = 9.4425 (5) Å
$c$ = 13.3576 (8) Å
$\alpha$ = 93.1550 (10)°
$\beta$ = 96.0560 (10)°
$\gamma$ = 112.3970 (10)°
$V$ = 938.80 (9) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 294 K
0.20 × 0.20 × 0.10 mm

### Data collection

Bruker SMART 4K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{min}$ = 0.976, $T_{max}$ = 0.989
7700 measured reflections
3624 independent reflections
3028 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.020

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.144
$S$ = 1.05
3624 reflections
261 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C7—H7$A$···O3 | 0.97 | 2.52 | 3.107 (2) | 119 |
| C17—H17$B$···O2$^{i}$ | 0.97 | 2.58 | 3.343 (3) | 136 |
| C7—H7$B$···O3$^{ii}$ | 0.97 | 2.59 | 3.478 (2) | 153 |
| N4—H4$A$···O2$^{iii}$ | 0.85 (3) | 2.05 (3) | 2.871 (2) | 165 (2) |
| N3—H3$A$···O5$^{iv}$ | 0.87 (3) | 2.09 (3) | 2.917 (2) | 159 (2) |

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2142).

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Wu, A., Fettinger, J. C. & Isaacs, L. (2002*b*). *Tetrahedron*, **58**, 9769–9777.

# supporting information

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## Diethyl 1,4-dioxo-1,2,2a,3,4,10b-hexahydro-5*H*,10*H*-2,3,4a,10a-tetraaza-benzo[*g*]cyclopenta[*cd*]azulene-2a,10b-dicarboxylate

**Jing Qin**

### S1. Comment

Glycoluril and its derivatives have been widely studied in supramolecular chemistry (Johnson *et al.*, 2002; Wu *et al.*, 2002b). As a continuation of our recent studies in this area (Wang *et al.*, 2006; Chen *et al.*, 2007), we herein report the crystal structure of the title compound (Fig. 1). The dihedral angle between the two fused five-membered rings in the glycoluril unit is 64.42 (2) °. In the crystal structure the molecules are connected via weak intermolecular N—H···O hydrogen bonding (Table 1).

### S2. Experimental

The title compound was synthesized according to a literature procedure (Wu *et al.*; 2002a). Crystals of (I) suitable for X-ray diffraction were grown by slow evaporation of a dichloromethane-methanol (1:2) solution of the title compound at room temperature.

### S3. Refinement

All H atoms were positioned with idealized geometry with C—H = 0.93–0.97 Å (methyl H atoms allowed to rotate but not to tip) and were refined isotropic ($U_{iso}$(H) = 1.2 $U_{eq}$(C) or 1.5 $U_{eq}$(C) (methyl C)) using a riding model.

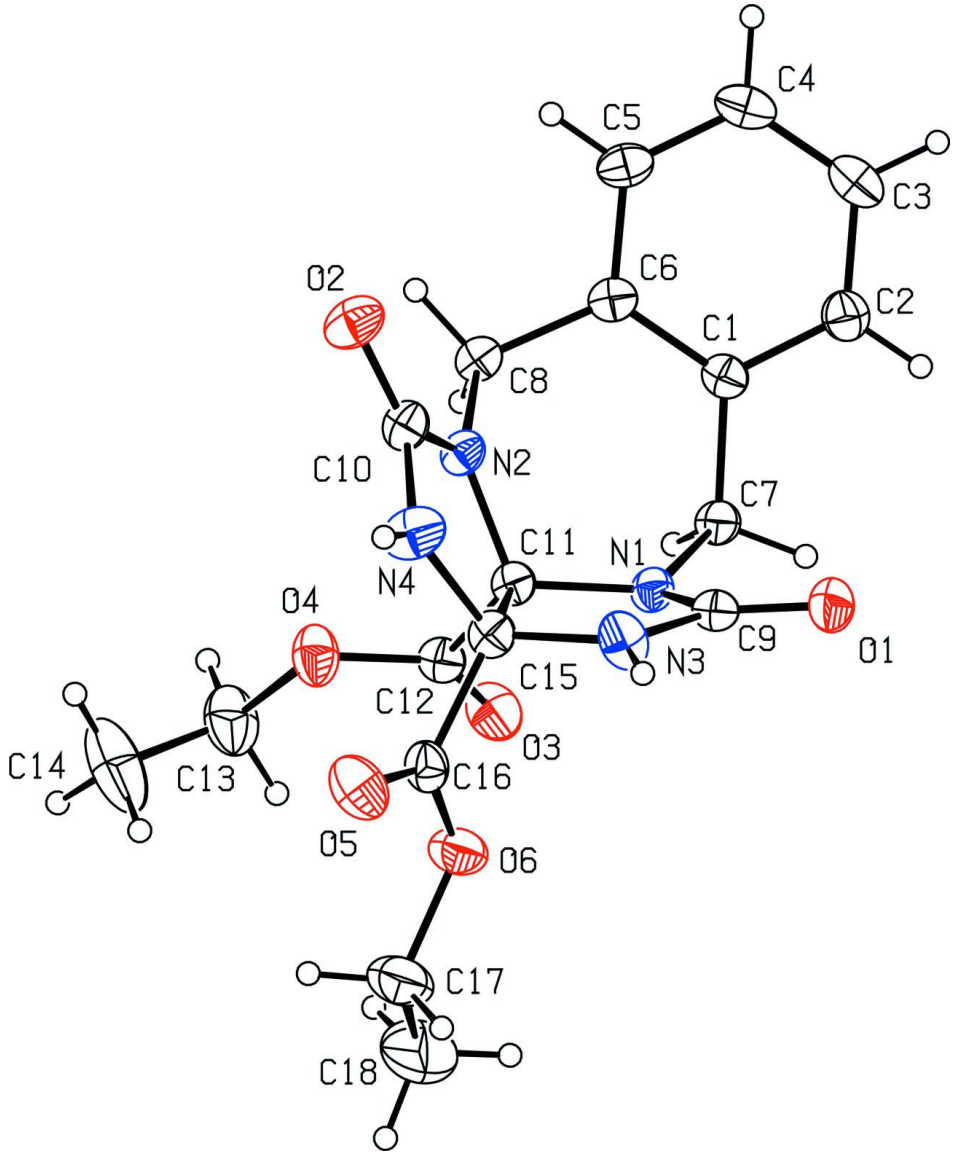


**Figure 1**

A view of (I), showing the atom-labelling scheme, with displacement ellipsoids drawn at the 30% probability level.

## Diethyl 1,4-dioxo-1,2,2a,3,4,10b-hexahydro-5*H*,10*H*-2,3,4a,10a-tetraazabenzo[g]cyclopenta[cd]azulene-2a,10b-dicarboxylate

*Crystal data*

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$a = 8.1394\ (5)$ Å
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$\alpha = 93.155\ (1)°$
$\beta = 96.056\ (1)°$
$\gamma = 112.397\ (1)°$
$V = 938.80\ (9)$ Å$^3$
$Z = 2$
$F(000) = 408$
$D_x = 1.374$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3262 reflections
$\theta = 2.4–27.5°$
$\mu = 0.11$ mm$^{-1}$

$T$ = 294 K
Block, colorless

0.20 × 0.20 × 0.10 mm

*Data collection*

Bruker SMART 4K CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{min}$ = 0.976, $T_{max}$ = 0.989

7700 measured reflections
3624 independent reflections
3028 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.020
$\theta_{max}$ = 26.0°, $\theta_{min}$ = 1.5°
$h$ = −10→8
$k$ = −11→11
$l$ = −16→16

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.144
$S$ = 1.05
3624 reflections
261 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
$w = 1/[\sigma^2(F_o^2) + (0.0741P)^2 + 0.3055P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.005
$\Delta\rho_{max}$ = 0.28 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$

*Special details*

**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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.7535 (3) | 0.3332 (2) | 0.06082 (12) | 0.0350 (4) |
| C2 | 0.6905 (3) | 0.1940 (2) | 0.00001 (15) | 0.0442 (5) |
| H2 | 0.5714 | 0.1512 | −0.0298 | 0.053* |
| C3 | 0.8025 (3) | 0.1180 (2) | −0.01692 (16) | 0.0516 (5) |
| H3 | 0.7589 | 0.0250 | −0.0579 | 0.062* |
| C4 | 0.9787 (3) | 0.1811 (2) | 0.02733 (16) | 0.0515 (5) |
| H4 | 1.0552 | 0.1318 | 0.0152 | 0.062* |
| C5 | 1.0421 (3) | 0.3177 (2) | 0.08982 (15) | 0.0437 (5) |
| H5 | 1.1607 | 0.3581 | 0.1207 | 0.052* |
| C6 | 0.9321 (2) | 0.3955 (2) | 0.10733 (12) | 0.0352 (4) |
| C7 | 0.6302 (3) | 0.4178 (2) | 0.07030 (12) | 0.0375 (4) |
| H7A | 0.6781 | 0.5130 | 0.0391 | 0.045* |
| H7B | 0.5141 | 0.3556 | 0.0321 | 0.045* |

| | | | | |
|---|---|---|---|---|
| C8 | 1.0095 (2) | 0.5470 (2) | 0.17322 (13) | 0.0359 (4) |
| H8A | 1.1323 | 0.5666 | 0.2006 | 0.043* |
| H8B | 1.0123 | 0.6287 | 0.1316 | 0.043* |
| C9 | 0.4886 (3) | 0.3441 (2) | 0.22434 (14) | 0.0392 (4) |
| C10 | 0.9584 (3) | 0.5307 (2) | 0.35329 (13) | 0.0395 (4) |
| C11 | 0.7426 (2) | 0.5735 (2) | 0.24235 (12) | 0.0330 (4) |
| C12 | 0.7751 (3) | 0.7358 (2) | 0.20982 (13) | 0.0372 (4) |
| C13 | 0.9500 (4) | 1.0015 (3) | 0.2570 (2) | 0.0691 (7) |
| H13A | 1.0481 | 1.0306 | 0.2168 | 0.083* |
| H13B | 0.8494 | 1.0152 | 0.2195 | 0.083* |
| C14 | 1.0045 (7) | 1.0966 (3) | 0.3520 (3) | 0.1181 (14) |
| H14A | 0.9041 | 1.0729 | 0.3891 | 0.177* |
| H14B | 1.0469 | 1.2031 | 0.3398 | 0.177* |
| H14C | 1.0988 | 1.0774 | 0.3906 | 0.177* |
| C15 | 0.6771 (3) | 0.5423 (2) | 0.34897 (13) | 0.0379 (4) |
| C16 | 0.6154 (3) | 0.6578 (2) | 0.40178 (14) | 0.0412 (4) |
| C17 | 0.4679 (4) | 0.8288 (3) | 0.3831 (2) | 0.0741 (8) |
| H17A | 0.5609 | 0.9012 | 0.4335 | 0.089* |
| H17B | 0.3639 | 0.7756 | 0.4160 | 0.089* |
| C18 | 0.4201 (6) | 0.9114 (4) | 0.3022 (3) | 0.1120 (13) |
| H18A | 0.5252 | 0.9687 | 0.2727 | 0.168* |
| H18B | 0.3718 | 0.9808 | 0.3300 | 0.168* |
| H18C | 0.3321 | 0.8386 | 0.2511 | 0.168* |
| N1 | 0.6035 (2) | 0.45511 (17) | 0.17377 (10) | 0.0356 (4) |
| N2 | 0.9101 (2) | 0.55255 (17) | 0.25637 (10) | 0.0345 (4) |
| N3 | 0.5268 (3) | 0.3968 (2) | 0.32473 (13) | 0.0491 (4) |
| H3A | 0.456 (3) | 0.355 (3) | 0.3683 (19) | 0.059* |
| N4 | 0.8307 (3) | 0.5358 (2) | 0.40898 (13) | 0.0532 (5) |
| H4A | 0.835 (3) | 0.525 (3) | 0.472 (2) | 0.064* |
| O1 | 0.37312 (19) | 0.22369 (16) | 0.18597 (12) | 0.0528 (4) |
| O2 | 1.0959 (2) | 0.51339 (19) | 0.38374 (10) | 0.0531 (4) |
| O3 | 0.7018 (2) | 0.75960 (17) | 0.13482 (10) | 0.0552 (4) |
| O4 | 0.8984 (2) | 0.84003 (15) | 0.27670 (11) | 0.0526 (4) |
| O5 | 0.6376 (3) | 0.68292 (19) | 0.49151 (11) | 0.0644 (5) |
| O6 | 0.5323 (2) | 0.71765 (18) | 0.33836 (11) | 0.0556 (4) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0430 (10) | 0.0384 (9) | 0.0271 (8) | 0.0191 (8) | 0.0058 (7) | 0.0050 (7) |
| C2 | 0.0461 (11) | 0.0437 (10) | 0.0410 (10) | 0.0161 (9) | 0.0054 (8) | 0.0024 (8) |
| C3 | 0.0708 (15) | 0.0391 (11) | 0.0507 (12) | 0.0266 (11) | 0.0138 (11) | 0.0024 (9) |
| C4 | 0.0657 (15) | 0.0492 (12) | 0.0557 (12) | 0.0368 (11) | 0.0181 (11) | 0.0113 (10) |
| C5 | 0.0426 (11) | 0.0528 (11) | 0.0450 (10) | 0.0261 (10) | 0.0106 (9) | 0.0166 (9) |
| C6 | 0.0391 (10) | 0.0427 (10) | 0.0286 (8) | 0.0197 (8) | 0.0072 (7) | 0.0108 (7) |
| C7 | 0.0406 (10) | 0.0476 (10) | 0.0266 (8) | 0.0221 (9) | −0.0021 (7) | −0.0009 (7) |
| C8 | 0.0340 (10) | 0.0443 (10) | 0.0305 (9) | 0.0155 (8) | 0.0059 (7) | 0.0078 (7) |
| C9 | 0.0373 (10) | 0.0384 (10) | 0.0451 (10) | 0.0186 (9) | 0.0059 (8) | 0.0022 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C10 | 0.0412 (11) | 0.0464 (10) | 0.0293 (9) | 0.0160 (9) | 0.0003 (8) | 0.0062 (8) |
| C11 | 0.0354 (10) | 0.0382 (9) | 0.0262 (8) | 0.0151 (8) | 0.0042 (7) | 0.0044 (7) |
| C12 | 0.0419 (10) | 0.0411 (10) | 0.0312 (9) | 0.0186 (9) | 0.0061 (8) | 0.0055 (7) |
| C13 | 0.0793 (18) | 0.0386 (12) | 0.0763 (17) | 0.0093 (12) | 0.0038 (14) | 0.0123 (11) |
| C14 | 0.170 (4) | 0.0489 (16) | 0.107 (3) | 0.019 (2) | 0.001 (3) | −0.0135 (16) |
| C15 | 0.0405 (10) | 0.0436 (10) | 0.0296 (9) | 0.0160 (9) | 0.0055 (7) | 0.0056 (7) |
| C16 | 0.0412 (11) | 0.0415 (10) | 0.0350 (10) | 0.0092 (9) | 0.0087 (8) | −0.0008 (8) |
| C17 | 0.0743 (18) | 0.0650 (16) | 0.096 (2) | 0.0388 (15) | 0.0268 (15) | −0.0014 (14) |
| C18 | 0.123 (3) | 0.088 (2) | 0.143 (3) | 0.070 (2) | −0.014 (3) | 0.001 (2) |
| N1 | 0.0346 (8) | 0.0415 (8) | 0.0296 (7) | 0.0153 (7) | 0.0002 (6) | 0.0005 (6) |
| N2 | 0.0339 (8) | 0.0433 (8) | 0.0263 (7) | 0.0155 (7) | 0.0013 (6) | 0.0051 (6) |
| N3 | 0.0571 (11) | 0.0412 (9) | 0.0423 (9) | 0.0088 (8) | 0.0185 (8) | 0.0041 (7) |
| N4 | 0.0579 (12) | 0.0867 (14) | 0.0265 (8) | 0.0393 (11) | 0.0068 (8) | 0.0152 (8) |
| O1 | 0.0427 (8) | 0.0437 (8) | 0.0624 (9) | 0.0079 (7) | 0.0054 (7) | −0.0045 (7) |
| O2 | 0.0493 (9) | 0.0791 (11) | 0.0373 (7) | 0.0323 (8) | 0.0003 (6) | 0.0148 (7) |
| O3 | 0.0773 (11) | 0.0517 (9) | 0.0407 (8) | 0.0328 (8) | −0.0054 (7) | 0.0069 (6) |
| O4 | 0.0588 (9) | 0.0372 (7) | 0.0505 (8) | 0.0091 (7) | −0.0048 (7) | 0.0069 (6) |
| O5 | 0.0891 (13) | 0.0677 (10) | 0.0367 (8) | 0.0307 (9) | 0.0136 (8) | −0.0042 (7) |
| O6 | 0.0633 (10) | 0.0630 (9) | 0.0520 (9) | 0.0378 (8) | 0.0095 (7) | 0.0002 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.390 (3) | C11—C12 | 1.548 (2) |
| C1—C6 | 1.401 (3) | C11—C15 | 1.577 (2) |
| C1—C7 | 1.513 (2) | C12—O3 | 1.188 (2) |
| C2—C3 | 1.386 (3) | C12—O4 | 1.317 (2) |
| C2—H2 | 0.9300 | C13—C14 | 1.437 (4) |
| C3—C4 | 1.375 (3) | C13—O4 | 1.466 (3) |
| C3—H3 | 0.9300 | C13—H13A | 0.9700 |
| C4—C5 | 1.382 (3) | C13—H13B | 0.9700 |
| C4—H4 | 0.9300 | C14—H14A | 0.9600 |
| C5—C6 | 1.387 (3) | C14—H14B | 0.9600 |
| C5—H5 | 0.9300 | C14—H14C | 0.9600 |
| C6—C8 | 1.508 (3) | C15—N4 | 1.437 (2) |
| C7—N1 | 1.466 (2) | C15—N3 | 1.441 (3) |
| C7—H7A | 0.9700 | C15—C16 | 1.531 (3) |
| C7—H7B | 0.9700 | C16—O5 | 1.190 (2) |
| C8—N2 | 1.451 (2) | C16—O6 | 1.311 (2) |
| C8—H8A | 0.9700 | C17—O6 | 1.466 (3) |
| C8—H8B | 0.9700 | C17—C18 | 1.471 (4) |
| C9—O1 | 1.208 (2) | C17—H17A | 0.9700 |
| C9—N3 | 1.365 (3) | C17—H17B | 0.9700 |
| C9—N1 | 1.376 (2) | C18—H18A | 0.9600 |
| C10—O2 | 1.223 (2) | C18—H18B | 0.9600 |
| C10—N4 | 1.354 (3) | C18—H18C | 0.9600 |
| C10—N2 | 1.363 (2) | N3—H3A | 0.87 (3) |
| C11—N1 | 1.440 (2) | N4—H4A | 0.85 (3) |
| C11—N2 | 1.445 (2) | | |

| | | | |
|---|---|---|---|
| C2—C1—C6 | 119.23 (17) | C14—C13—H13A | 109.9 |
| C2—C1—C7 | 119.00 (17) | O4—C13—H13A | 109.9 |
| C6—C1—C7 | 121.69 (16) | C14—C13—H13B | 109.9 |
| C3—C2—C1 | 121.0 (2) | O4—C13—H13B | 109.9 |
| C3—C2—H2 | 119.5 | H13A—C13—H13B | 108.3 |
| C1—C2—H2 | 119.5 | C13—C14—H14A | 109.5 |
| C4—C3—C2 | 119.5 (2) | C13—C14—H14B | 109.5 |
| C4—C3—H3 | 120.3 | H14A—C14—H14B | 109.5 |
| C2—C3—H3 | 120.3 | C13—C14—H14C | 109.5 |
| C3—C4—C5 | 120.1 (2) | H14A—C14—H14C | 109.5 |
| C3—C4—H4 | 120.0 | H14B—C14—H14C | 109.5 |
| C5—C4—H4 | 120.0 | N4—C15—N3 | 114.94 (17) |
| C4—C5—C6 | 121.16 (19) | N4—C15—C16 | 110.13 (16) |
| C4—C5—H5 | 119.4 | N3—C15—C16 | 109.09 (16) |
| C6—C5—H5 | 119.4 | N4—C15—C11 | 102.62 (15) |
| C5—C6—C1 | 118.94 (18) | N3—C15—C11 | 101.34 (14) |
| C5—C6—C8 | 119.12 (17) | C16—C15—C11 | 118.67 (15) |
| C1—C6—C8 | 121.91 (16) | O5—C16—O6 | 125.5 (2) |
| N1—C7—C1 | 115.68 (14) | O5—C16—C15 | 121.54 (18) |
| N1—C7—H7A | 108.4 | O6—C16—C15 | 112.92 (15) |
| C1—C7—H7A | 108.4 | O6—C17—C18 | 108.6 (3) |
| N1—C7—H7B | 108.4 | O6—C17—H17A | 110.0 |
| C1—C7—H7B | 108.4 | C18—C17—H17A | 110.0 |
| H7A—C7—H7B | 107.4 | O6—C17—H17B | 110.0 |
| N2—C8—C6 | 113.76 (15) | C18—C17—H17B | 110.0 |
| N2—C8—H8A | 108.8 | H17A—C17—H17B | 108.3 |
| C6—C8—H8A | 108.8 | C17—C18—H18A | 109.5 |
| N2—C8—H8B | 108.8 | C17—C18—H18B | 109.5 |
| C6—C8—H8B | 108.8 | H18A—C18—H18B | 109.5 |
| H8A—C8—H8B | 107.7 | C17—C18—H18C | 109.5 |
| O1—C9—N3 | 126.68 (18) | H18A—C18—H18C | 109.5 |
| O1—C9—N1 | 125.85 (18) | H18B—C18—H18C | 109.5 |
| N3—C9—N1 | 107.45 (16) | C9—N1—C11 | 111.93 (14) |
| O2—C10—N4 | 126.78 (16) | C9—N1—C7 | 120.52 (15) |
| O2—C10—N2 | 125.11 (18) | C11—N1—C7 | 121.64 (15) |
| N4—C10—N2 | 108.09 (16) | C10—N2—C11 | 113.25 (15) |
| N1—C11—N2 | 113.91 (14) | C10—N2—C8 | 124.21 (15) |
| N1—C11—C12 | 111.49 (13) | C11—N2—C8 | 122.46 (13) |
| N2—C11—C12 | 109.92 (14) | C9—N3—C15 | 114.32 (16) |
| N1—C11—C15 | 103.63 (14) | C9—N3—H3A | 122.6 (16) |
| N2—C11—C15 | 101.80 (13) | C15—N3—H3A | 122.3 (16) |
| C12—C11—C15 | 115.76 (14) | C10—N4—C15 | 113.12 (15) |
| O3—C12—O4 | 126.43 (17) | C10—N4—H4A | 123.5 (17) |
| O3—C12—C11 | 124.36 (17) | C15—N4—H4A | 123.0 (17) |
| O4—C12—C11 | 109.19 (14) | C12—O4—C13 | 116.92 (16) |
| C14—C13—O4 | 108.9 (2) | C16—O6—C17 | 116.28 (18) |

| | | | |
|---|---|---|---|
| C6—C1—C2—C3 | −1.4 (3) | O1—C9—N1—C7 | −17.8 (3) |
| C7—C1—C2—C3 | 175.22 (17) | N3—C9—N1—C7 | 163.56 (16) |
| C1—C2—C3—C4 | 0.2 (3) | N2—C11—N1—C9 | 97.72 (17) |
| C2—C3—C4—C5 | 1.3 (3) | C12—C11—N1—C9 | −137.17 (15) |
| C3—C4—C5—C6 | −1.5 (3) | C15—C11—N1—C9 | −12.01 (18) |
| C4—C5—C6—C1 | 0.3 (3) | N2—C11—N1—C7 | −55.3 (2) |
| C4—C5—C6—C8 | −177.86 (17) | C12—C11—N1—C7 | 69.8 (2) |
| C2—C1—C6—C5 | 1.2 (2) | C15—C11—N1—C7 | −165.00 (14) |
| C7—C1—C6—C5 | −175.36 (16) | C1—C7—N1—C9 | −78.3 (2) |
| C2—C1—C6—C8 | 179.26 (16) | C1—C7—N1—C11 | 72.4 (2) |
| C7—C1—C6—C8 | 2.7 (2) | O2—C10—N2—C11 | −178.59 (18) |
| C2—C1—C7—N1 | 124.69 (18) | N4—C10—N2—C11 | −0.2 (2) |
| C6—C1—C7—N1 | −58.8 (2) | O2—C10—N2—C8 | 4.8 (3) |
| C5—C6—C8—N2 | −126.25 (17) | N4—C10—N2—C8 | −176.82 (17) |
| C1—C6—C8—N2 | 55.7 (2) | N1—C11—N2—C10 | −116.92 (17) |
| N1—C11—C12—O3 | −3.8 (3) | C12—C11—N2—C10 | 117.15 (16) |
| N2—C11—C12—O3 | 123.5 (2) | C15—C11—N2—C10 | −6.07 (19) |
| C15—C11—C12—O3 | −121.9 (2) | N1—C11—N2—C8 | 59.8 (2) |
| N1—C11—C12—O4 | 177.71 (15) | C12—C11—N2—C8 | −66.1 (2) |
| N2—C11—C12—O4 | −55.00 (19) | C15—C11—N2—C8 | 170.64 (15) |
| C15—C11—C12—O4 | 59.6 (2) | C6—C8—N2—C10 | 99.2 (2) |
| N1—C11—C15—N4 | 127.94 (15) | C6—C8—N2—C11 | −77.2 (2) |
| N2—C11—C15—N4 | 9.48 (18) | O1—C9—N3—C15 | 177.73 (18) |
| C12—C11—C15—N4 | −109.69 (17) | N1—C9—N3—C15 | −3.7 (2) |
| N1—C11—C15—N3 | 8.88 (17) | N4—C15—N3—C9 | −113.33 (19) |
| N2—C11—C15—N3 | −109.58 (15) | C16—C15—N3—C9 | 122.44 (18) |
| C12—C11—C15—N3 | 131.26 (16) | C11—C15—N3—C9 | −3.5 (2) |
| N1—C11—C15—C16 | −110.44 (17) | O2—C10—N4—C15 | −174.2 (2) |
| N2—C11—C15—C16 | 131.09 (17) | N2—C10—N4—C15 | 7.4 (2) |
| C12—C11—C15—C16 | 11.9 (2) | N3—C15—N4—C10 | 98.4 (2) |
| N4—C15—C16—O5 | −29.5 (3) | C16—C15—N4—C10 | −137.96 (18) |
| N3—C15—C16—O5 | 97.5 (2) | C11—C15—N4—C10 | −10.7 (2) |
| C11—C15—C16—O5 | −147.25 (19) | O3—C12—O4—C13 | 0.2 (3) |
| N4—C15—C16—O6 | 153.07 (17) | C11—C12—O4—C13 | 178.62 (19) |
| N3—C15—C16—O6 | −79.91 (19) | C14—C13—O4—C12 | 150.2 (3) |
| C11—C15—C16—O6 | 35.3 (2) | O5—C16—O6—C17 | 2.3 (3) |
| O1—C9—N1—C11 | −171.16 (18) | C15—C16—O6—C17 | 179.63 (19) |
| N3—C9—N1—C11 | 10.2 (2) | C18—C17—O6—C16 | 166.1 (2) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C7—H7*A*···O3 | 0.97 | 2.52 | 3.107 (2) | 119 |
| C17—H17*B*···O2$^{i}$ | 0.97 | 2.58 | 3.343 (3) | 136 |
| C7—H7*B*···O3$^{ii}$ | 0.97 | 2.59 | 3.478 (2) | 153 |

| | | | | |
|---|---|---|---|---|
| N4—H4*A*···O2$^{iii}$ | 0.85 (3) | 2.05 (3) | 2.871 (2) | 165 (2) |
| N3—H3*A*···O5$^{iv}$ | 0.87 (3) | 2.09 (3) | 2.917 (2) | 159 (2) |

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