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# Diethyl 6,13-dioxo-5,7,12,13b,13c,14-hexahydro-6*H*,13*H*-5a,6a,12a,13a-tetraazabenz[5,6]azuleno[2,1,8-*ija*]benz[*f*]azulene-13b,13c-dicarboxylate 1,2-dichloroethane solvate

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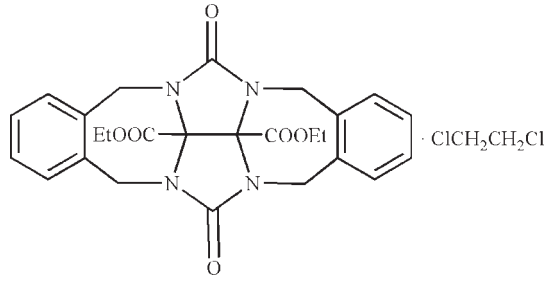
Key indicators: single-crystal X-ray study; $T$ = 292 K; mean $\sigma$(C–C) = 0.005 Å; disorder in main residue; $R$ factor = 0.063; $wR$ factor = 0.188; data-to-parameter ratio = 12.9.

In the title inclusion compound, $C_{26}H_{26}N_4O_6\cdot C_2H_4Cl_2$, the solvent molecule occupies a cavity inside the clip-type molecule which is based on the glycoluril skeleton with two ethyl acetate substituents on the convex face of the glycoluril system. The dihedral angle between the aromatic rings of the host is 43.59 (4)° and the centroid–centroid distance is 6.741 (5) Å. The 1,2-dichloroetane molecule adopts a *gauche* conformation enabling it to participate in C—H···$\pi$ interactions with the host. The packing motif in the title compound differs from that observed in the crystal structures of the host and in the benzene solvate. The host molecules are linked into tapes by $\pi$–$\pi$ stacking interactions (centroid–centroid distance = 3.733 Å) and are further assembled into layers *via* C—H···O interactions. One of the ethyl groups is disorded over two positions with site-occupancy factors of 0.702 (14) and 0.298 (14).

## Related literature

For the related structures, see: Chen *et al.* (2007); Hof *et al.*,(2002); Hu *et al.* (2007); Isaacs & Fettinger (1999); Wang *et al.* (2006).




## Experimental

### Crystal data

$C_{26}H_{26}N_4O_6\cdot C_2H_4Cl_2$
$M_r$ = 589.46
Triclinic, $P\overline{1}$
$a$ = 8.9468 (10) Å
$b$ = 11.1544 (13) Å
$c$ = 15.6260 (18) Å
$\alpha$ = 69.257 (2)°
$\beta$ = 82.688 (2)°
$\gamma$ = 76.892 (2)°
$V$ = 1418.5 (3) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.28 mm$^{-1}$
$T$ = 292 K
0.30 × 0.20 × 0.10 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
10171 measured reflections
4947 independent reflections
4056 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.020

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.188
$S$ = 1.04
4947 reflections
383 parameters
28 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.69 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.64 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C28—H28$B$···$Cg2$ | 0.97 | 2.55 | 3.476 (3) | 160 |
| C5—H5···O5$^{i}$ | 0.93 | 2.59 | 3.393 (4) | 145 |
| C7—H7$B$···O5$^{i}$ | 0.97 | 2.50 | 3.359 (3) | 148 |
| C14—H14$A$···O2$^{ii}$ | 0.96 | 2.57 | 3.514 (9) | 168 |
| C14′—H14$F$···O2$^{ii}$ | 0.96 | 2.47 | 3.351 (6) | 153 |

Symmetry codes: (i) $x+1, y, z$; (ii) $-x, -y+1, -z+1$. $Cg2$ is the centroid of the C21–C26 ring.

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: GK2232).

## References

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Hof, F., Craig, S. L., Nuckolls, C. & Rebek, J. Jr (2002). *Angew. Chem. Int. Ed.* **41**, 1488–1508.
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# supporting information

*Acta Cryst.* (2009). E**65**, o2746–o2747 [https://doi.org/10.1107/S1600536809041373]

# Diethyl 6,13-dioxo-5,7,12,13b,13c,14-hexahydro-6*H*,13*H*-5a,6a,12a,13a-tetraazabenz[5,6]azuleno[2,1,8-*ija*]benz[*f*]azulene-13b,13c-dicarboxylate 1,2-dichloroethane solvate

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## S1. Comment

Molecular clips based on the glycoluril skeleton, which have a well defined geometry due to the rigidity of the fused rings, have been prepared for a wide variety of supramolecular applications, including molecular recognition (Hu *et al.*, 2007), molecular assemblies (Hof *et al.*, 2002), crystal engineering (Wang *et al.*, 2006; Chen *et al.*, 2007),

The title host compound is a kind of molecular receptor, possessing a well defined U-shaped cavity formed by the glycoluril framework with two aromatic side walls. The crystal structures of monoclinic apohost obtained from dichloroethane solution (C2/c, Z′ = 1/2; Wang *et al.*, 2006) and monoclinic benzene solvate, $C_{26}H_{26}N_4O_6.0.25C_6H_6$, ($P2_1/c$, $Z' = 2$; Isaacs *et al.*, 1999) have been already reported. Here, we report the structure of a new solvate (P-1, Z′ = 1), $C_{26}H_{26}N_4O_6.C_2H_4Cl_2$, crystallized from 1,2-dichoroethane/methanol (3:1 vol.) solution.

In the triclinic pseudopolymorph, the asymmetric unit contains one host molecule and one solvent molecule of 1,2-dichloroethane (Fig.1). There is a C-H···$\pi$ interaction betweeen the host and the guest. The distance between atom H28B and *Cg*2 (the centroid of the C21—C26 ring) is 2.67 Å (Table 1).

As solvent molecule occupies the cavity, the title compound exhibits different packing motif than the previously known two monoclinic forms. In Isaacs structure, the molecule was kined into three-dimensional network structure by $\pi$-$\pi$ stacking, C—H···$\pi$ and C—H···O hydrogen-bonds. In our previously reported structure of the apohost, the ethyl group occupied the cavity with C—H···$\pi$ interactions and the molecules were linked into layers to the *ab* plane by four pairs of C—H···O hydrogen-bonds parallel .

The title molecules are linked into tapes by $\pi$-$\pi$ stacking interactions. The benzene rings *Cg*1 (C1—C6) in the molecules at (*x*, *y*, *z*) and (1 - *x*, 1 - *y*, 1 - *z*) are strictly parallel, with an interplanar spacing of 3.589 Å, a ring centroid separation of 3.733 Å and a centroid offset of 1.027 Å. In addition, intermolecular C—H···O (Table 1) hydrogen-bonds link molecules into two-dimensional layer structure.

## S2. Experimental

The host compound was synthesized as reported previously (Wang *et al.*, 2006). Crystals of the title compound were obtained by slow evaporation at 293 K of 1,2-dichoroethane/methanol (*vol.* 3:1) solution .

## S3. Refinement

The H atoms from methyl groups were placed in calculated positions, with C—H=0.96 Å, and refined to fit the electron density [$U_{iso}$(H)=1.5$U_{eq}$(C)]. Other H atoms were placed in calculated positions, with C—H = 0.93Å (aromatic) and 0.97Å (methylene), and refined in riding mode [$U_{iso}$(H)=1.2$U_{eq}$(C)]. One of the ester ethyl groups (C13,C14) is disordered over two positions. Restraints were imposed on the geometry of the disordered ethyl group and anisotropic displacement

parameters. The occupanicies of the disordered atoms C13/C13′ and C14/C14′ refined at 0.702 (14)/0.298 (14).

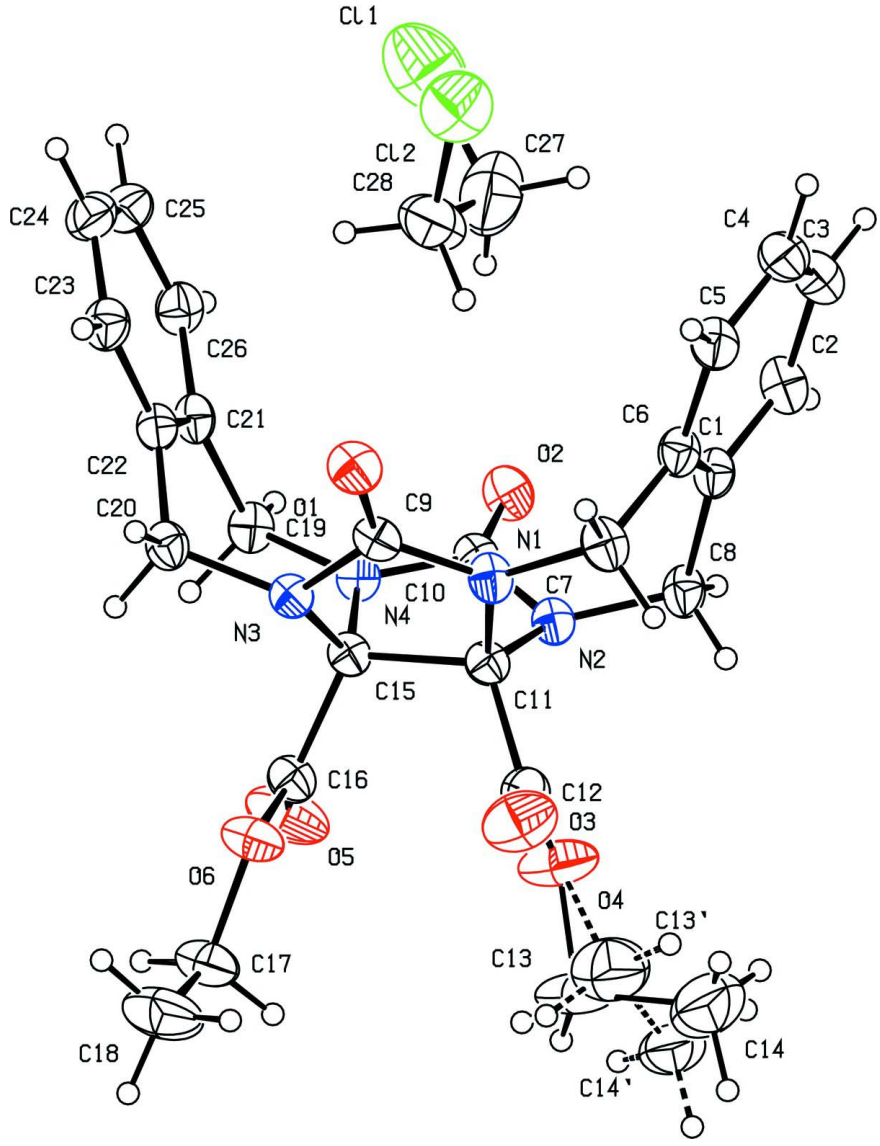


**Figure 1**

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

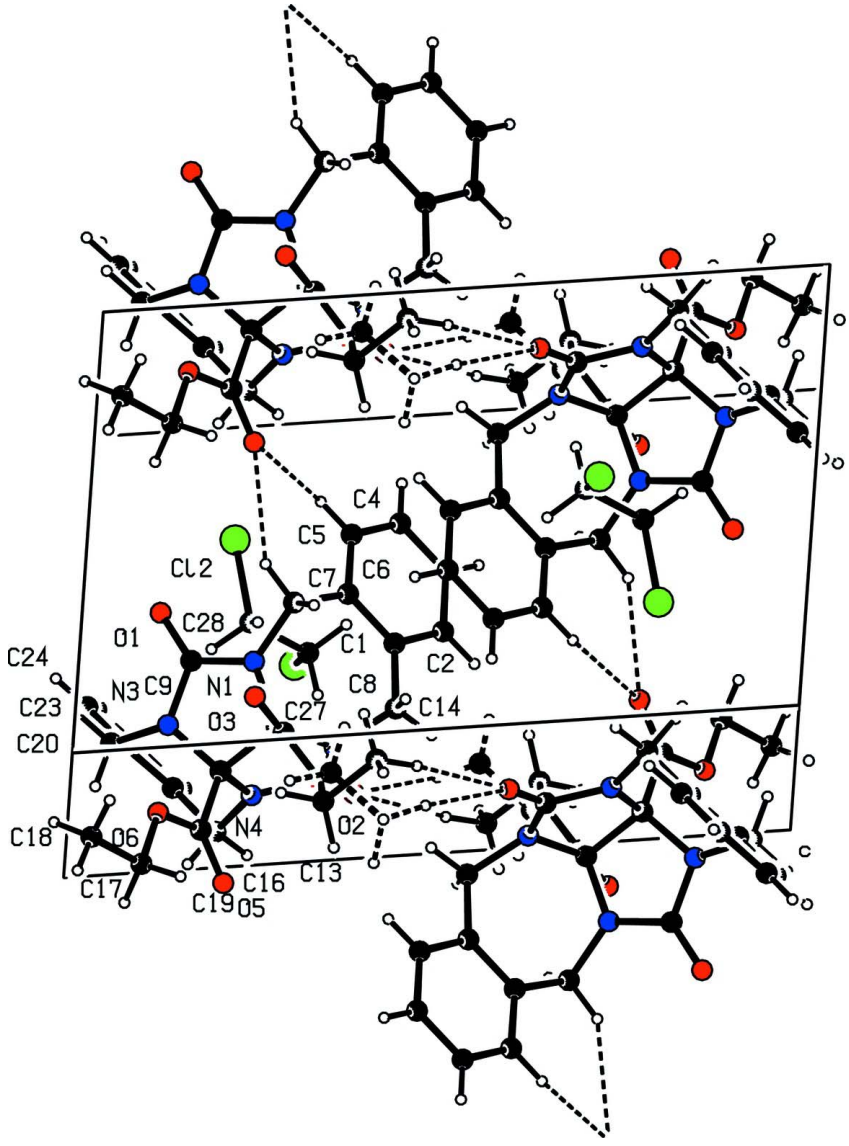


**Figure 2**

Crystal packing of the title compound

## Diethyl 6,13-dioxo-5,7,12,13b,13c,14-hexahydro-6*H*,13*H*-5a,6a,12a,13a- tetraazabenz[5,6]azuleno[2,1,8-*ija*]benz[*f*]azulene-13b,13c- dicarboxylate 1,2-dichloroethane solvate

*Crystal data*

$C_{26}H_{26}N_4O_6{\cdot}C_2H_4Cl_2$
$M_r$ = 589.46
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a$ = 8.9468 (10) Å
$b$ = 11.1544 (13) Å
$c$ = 15.6260 (18) Å
$\alpha$ = 69.257 (2)°
$\beta$ = 82.688 (2)°
$\gamma$ = 76.892 (2)°
$V$ = 1418.5 (3) Å$^3$
$Z$ = 2
$F$(000) = 616
$D_x$ = 1.380 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 5260 reflections
$\theta$ = 2.3–26.1°
$\mu$ = 0.28 mm$^{-1}$

$T$ = 292 K
Block, colorless

0.30 × 0.20 × 0.10 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
10171 measured reflections
4947 independent reflections

4056 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.020
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 2.0°
$h$ = −10→10
$k$ = −13→13
$l$ = −18→18

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.188
$S$ = 1.04
4947 reflections
383 parameters
28 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
$w = 1/[\sigma^2(F_o^2) + (0.1032P)^2 + 0.8923P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.69 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.64 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 (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.3563 (3) | 0.4517 (3) | 0.43467 (17) | 0.0419 (6) | |
| C2 | 0.4101 (3) | 0.3329 (3) | 0.5006 (2) | 0.0527 (7) | |
| H2 | 0.3424 | 0.2944 | 0.5472 | 0.063* | |
| C3 | 0.5627 (4) | 0.2702 (3) | 0.4988 (2) | 0.0585 (8) | |
| H3 | 0.5962 | 0.1900 | 0.5434 | 0.070* | |
| C4 | 0.6644 (3) | 0.3268 (3) | 0.4308 (2) | 0.0558 (8) | |
| H4 | 0.7670 | 0.2856 | 0.4294 | 0.067* | |
| C5 | 0.6127 (3) | 0.4452 (3) | 0.3650 (2) | 0.0489 (7) | |
| H5 | 0.6816 | 0.4833 | 0.3191 | 0.059* | |
| C6 | 0.4600 (3) | 0.5090 (3) | 0.36552 (18) | 0.0418 (6) | |
| C7 | 0.4116 (3) | 0.6394 (3) | 0.29185 (19) | 0.0477 (7) | |
| H7A | 0.3833 | 0.7071 | 0.3197 | 0.057* | |
| H7B | 0.4982 | 0.6589 | 0.2487 | 0.057* | |
| C8 | 0.1895 (3) | 0.5177 (3) | 0.44266 (18) | 0.0453 (6) | |
| H8A | 0.1394 | 0.4605 | 0.4949 | 0.054* | |

| | | | | | |
|---|---|---|---|---|---|
| H8B | 0.1866 | 0.5974 | 0.4552 | 0.054* | |
| C9 | 0.2946 (3) | 0.6387 (2) | 0.15541 (18) | 0.0398 (6) | |
| C10 | 0.0566 (3) | 0.4531 (2) | 0.34105 (17) | 0.0372 (6) | |
| C11 | 0.1265 (3) | 0.6567 (2) | 0.27998 (17) | 0.0378 (6) | |
| C12 | 0.0736 (3) | 0.7930 (3) | 0.2899 (2) | 0.0508 (7) | |
| C14 | −0.0459 (15) | 0.9472 (8) | 0.4212 (6) | 0.128 (4) | 0.702 (14) |
| H14A | −0.0458 | 0.8751 | 0.4776 | 0.192* | 0.702 (14) |
| H14B | −0.0985 | 1.0265 | 0.4314 | 0.192* | 0.702 (14) |
| H14C | 0.0581 | 0.9543 | 0.3994 | 0.192* | 0.702 (14) |
| C13 | −0.1260 (11) | 0.9249 (6) | 0.3514 (6) | 0.095 (3) | 0.702 (14) |
| H13A | −0.2332 | 0.9230 | 0.3705 | 0.114* | 0.702 (14) |
| H13B | −0.1203 | 0.9933 | 0.2925 | 0.114* | 0.702 (14) |
| C15 | 0.0392 (3) | 0.6362 (2) | 0.20652 (16) | 0.0368 (5) | |
| C16 | −0.1181 (3) | 0.7301 (3) | 0.18476 (19) | 0.0444 (6) | |
| C17 | −0.2468 (4) | 0.9420 (3) | 0.1062 (3) | 0.0680 (9) | |
| H17A | −0.3236 | 0.9100 | 0.0863 | 0.082* | |
| H17B | −0.2870 | 0.9616 | 0.1614 | 0.082* | |
| C18 | −0.2110 (6) | 1.0594 (4) | 0.0345 (3) | 0.0979 (15) | |
| H18A | −0.1399 | 1.0935 | 0.0562 | 0.147* | |
| H18B | −0.3036 | 1.1239 | 0.0187 | 0.147* | |
| H18C | −0.1658 | 1.0379 | −0.0187 | 0.147* | |
| C19 | −0.0402 (3) | 0.4324 (3) | 0.20547 (18) | 0.0429 (6) | |
| H19A | −0.1262 | 0.4892 | 0.1699 | 0.052* | |
| H19B | −0.0778 | 0.3591 | 0.2512 | 0.052* | |
| C20 | 0.1259 (3) | 0.6119 (3) | 0.05267 (17) | 0.0428 (6) | |
| H20A | 0.1919 | 0.6513 | 0.0009 | 0.051* | |
| H20B | 0.0205 | 0.6467 | 0.0350 | 0.051* | |
| C21 | 0.0802 (3) | 0.3813 (3) | 0.14224 (17) | 0.0419 (6) | |
| C22 | 0.1590 (3) | 0.4658 (3) | 0.07154 (17) | 0.0406 (6) | |
| C23 | 0.2683 (3) | 0.4133 (3) | 0.01628 (19) | 0.0493 (7) | |
| H23 | 0.3224 | 0.4681 | −0.0301 | 0.059* | |
| C24 | 0.2981 (4) | 0.2818 (3) | 0.0286 (2) | 0.0550 (7) | |
| H24 | 0.3710 | 0.2487 | −0.0095 | 0.066* | |
| C25 | 0.2200 (4) | 0.1994 (3) | 0.0975 (2) | 0.0572 (8) | |
| H25 | 0.2394 | 0.1106 | 0.1059 | 0.069* | |
| C26 | 0.1127 (3) | 0.2490 (3) | 0.1541 (2) | 0.0497 (7) | |
| H26 | 0.0611 | 0.1927 | 0.2011 | 0.060* | |
| C27 | 0.4248 (6) | 0.1793 (8) | 0.3161 (4) | 0.130 (2) | |
| H27A | 0.3189 | 0.2096 | 0.3335 | 0.156* | |
| H27B | 0.4875 | 0.1658 | 0.3663 | 0.156* | |
| C28 | 0.4742 (5) | 0.2807 (4) | 0.2334 (4) | 0.0969 (16) | |
| H28A | 0.4497 | 0.3640 | 0.2436 | 0.116* | |
| H28B | 0.4169 | 0.2891 | 0.1822 | 0.116* | |
| Cl1 | 0.4405 (2) | 0.03136 (15) | 0.29818 (12) | 0.1337 (6) | |
| Cl2 | 0.67477 (15) | 0.24467 (12) | 0.20429 (9) | 0.0987 (4) | |
| N1 | 0.2825 (2) | 0.6424 (2) | 0.24224 (15) | 0.0421 (5) | |
| N2 | 0.1009 (2) | 0.5506 (2) | 0.36207 (14) | 0.0387 (5) | |
| N3 | 0.1477 (2) | 0.6518 (2) | 0.12931 (13) | 0.0373 (5) | |

| | | | | | |
|---|---|---|---|---|---|
| N4 | 0.0168 (2) | 0.5040 (2) | 0.25176 (14) | 0.0392 (5) | |
| O1 | 0.4116 (2) | 0.6274 (2) | 0.10852 (13) | 0.0527 (5) | |
| O2 | 0.0510 (2) | 0.34424 (19) | 0.39298 (13) | 0.0516 (5) | |
| O3 | 0.1344 (3) | 0.8818 (2) | 0.2469 (2) | 0.0853 (8) | |
| O4 | −0.0449 (3) | 0.7988 (2) | 0.3460 (2) | 0.0856 (8) | |
| O5 | −0.2343 (3) | 0.7010 (3) | 0.2211 (2) | 0.0865 (9) | |
| O6 | −0.1047 (2) | 0.84312 (19) | 0.12437 (15) | 0.0582 (6) | |
| C14′ | −0.178 (2) | 0.9307 (14) | 0.4359 (10) | 0.083 (6) | 0.298 (14) |
| H14D | −0.2692 | 0.9053 | 0.4272 | 0.125* | 0.298 (14) |
| H14E | −0.2036 | 1.0188 | 0.4364 | 0.125* | 0.298 (14) |
| H14F | −0.1368 | 0.8732 | 0.4933 | 0.125* | 0.298 (14) |
| C13′ | −0.062 (3) | 0.9224 (17) | 0.3593 (19) | 0.109 (9) | 0.298 (14) |
| H13C | −0.0937 | 0.9929 | 0.3033 | 0.131* | 0.298 (14) |
| H13D | 0.0366 | 0.9318 | 0.3737 | 0.131* | 0.298 (14) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0371 (13) | 0.0563 (16) | 0.0361 (13) | −0.0142 (11) | −0.0003 (10) | −0.0176 (12) |
| C2 | 0.0476 (16) | 0.0627 (18) | 0.0445 (15) | −0.0184 (13) | −0.0048 (12) | −0.0086 (13) |
| C3 | 0.0564 (19) | 0.0537 (17) | 0.0596 (18) | −0.0080 (14) | −0.0156 (15) | −0.0096 (14) |
| C4 | 0.0385 (15) | 0.0658 (19) | 0.068 (2) | −0.0054 (13) | −0.0085 (14) | −0.0292 (16) |
| C5 | 0.0338 (14) | 0.0666 (18) | 0.0494 (16) | −0.0145 (12) | 0.0019 (12) | −0.0218 (14) |
| C6 | 0.0354 (13) | 0.0540 (15) | 0.0411 (14) | −0.0148 (11) | −0.0013 (11) | −0.0187 (12) |
| C7 | 0.0353 (14) | 0.0592 (17) | 0.0485 (15) | −0.0192 (12) | −0.0022 (11) | −0.0118 (13) |
| C8 | 0.0372 (14) | 0.0653 (17) | 0.0349 (13) | −0.0139 (12) | 0.0024 (11) | −0.0176 (12) |
| C9 | 0.0352 (13) | 0.0367 (13) | 0.0410 (14) | −0.0095 (10) | 0.0016 (11) | −0.0049 (10) |
| C10 | 0.0270 (12) | 0.0450 (14) | 0.0365 (13) | −0.0094 (10) | 0.0044 (10) | −0.0105 (11) |
| C11 | 0.0305 (12) | 0.0421 (13) | 0.0402 (13) | −0.0094 (10) | 0.0005 (10) | −0.0123 (11) |
| C12 | 0.0506 (17) | 0.0498 (16) | 0.0552 (17) | −0.0114 (13) | −0.0061 (14) | −0.0194 (14) |
| C14 | 0.196 (11) | 0.081 (5) | 0.103 (6) | 0.005 (6) | −0.015 (6) | −0.044 (4) |
| C13 | 0.089 (5) | 0.063 (4) | 0.115 (5) | 0.019 (3) | 0.020 (4) | −0.036 (3) |
| C15 | 0.0323 (12) | 0.0401 (13) | 0.0359 (13) | −0.0097 (10) | 0.0002 (10) | −0.0094 (10) |
| C16 | 0.0347 (14) | 0.0491 (15) | 0.0466 (15) | −0.0094 (11) | −0.0008 (11) | −0.0125 (12) |
| C17 | 0.0547 (19) | 0.0559 (19) | 0.076 (2) | 0.0096 (15) | −0.0052 (16) | −0.0134 (16) |
| C18 | 0.099 (3) | 0.054 (2) | 0.103 (3) | 0.013 (2) | 0.000 (3) | 0.000 (2) |
| C19 | 0.0405 (14) | 0.0486 (15) | 0.0423 (14) | −0.0183 (11) | 0.0012 (11) | −0.0136 (12) |
| C20 | 0.0441 (14) | 0.0478 (15) | 0.0330 (13) | −0.0109 (11) | −0.0008 (11) | −0.0085 (11) |
| C21 | 0.0398 (14) | 0.0502 (15) | 0.0383 (13) | −0.0132 (11) | −0.0062 (11) | −0.0140 (11) |
| C22 | 0.0378 (13) | 0.0494 (15) | 0.0353 (13) | −0.0096 (11) | −0.0032 (10) | −0.0140 (11) |
| C23 | 0.0454 (15) | 0.0612 (18) | 0.0432 (15) | −0.0137 (13) | 0.0013 (12) | −0.0189 (13) |
| C24 | 0.0495 (17) | 0.0634 (19) | 0.0596 (18) | −0.0066 (14) | 0.0005 (14) | −0.0337 (15) |
| C25 | 0.0621 (19) | 0.0514 (17) | 0.0642 (19) | −0.0075 (14) | −0.0086 (15) | −0.0272 (15) |
| C26 | 0.0543 (17) | 0.0487 (16) | 0.0494 (16) | −0.0181 (13) | −0.0049 (13) | −0.0146 (13) |
| C27 | 0.073 (3) | 0.229 (8) | 0.118 (4) | −0.017 (4) | 0.000 (3) | −0.105 (5) |
| C28 | 0.089 (3) | 0.066 (2) | 0.145 (4) | 0.007 (2) | −0.055 (3) | −0.044 (3) |
| Cl1 | 0.1401 (13) | 0.1025 (10) | 0.1312 (12) | −0.0443 (9) | −0.0254 (10) | 0.0126 (8) |
| Cl2 | 0.1068 (9) | 0.0891 (8) | 0.1020 (8) | −0.0310 (6) | 0.0009 (7) | −0.0292 (6) |

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| N1 | 0.0317 (11) | 0.0554 (13) | 0.0394 (12) | −0.0153 (9) | 0.0016 (9) | −0.0134 (10) |
| N2 | 0.0312 (11) | 0.0488 (12) | 0.0351 (11) | −0.0116 (9) | 0.0020 (8) | −0.0119 (9) |
| N3 | 0.0331 (11) | 0.0408 (11) | 0.0344 (11) | −0.0087 (8) | 0.0027 (8) | −0.0090 (9) |
| N4 | 0.0401 (11) | 0.0413 (11) | 0.0369 (11) | −0.0146 (9) | 0.0001 (9) | −0.0107 (9) |
| O1 | 0.0362 (10) | 0.0659 (13) | 0.0494 (11) | −0.0124 (9) | 0.0105 (9) | −0.0143 (9) |
| O2 | 0.0540 (12) | 0.0500 (11) | 0.0436 (11) | −0.0175 (9) | −0.0005 (9) | −0.0034 (9) |
| O3 | 0.106 (2) | 0.0513 (14) | 0.101 (2) | −0.0313 (14) | 0.0131 (17) | −0.0258 (14) |
| O4 | 0.0864 (18) | 0.0585 (14) | 0.101 (2) | −0.0014 (13) | 0.0328 (16) | −0.0346 (14) |
| O5 | 0.0343 (12) | 0.0731 (16) | 0.119 (2) | −0.0072 (10) | 0.0087 (13) | 0.0011 (15) |
| O6 | 0.0463 (11) | 0.0451 (11) | 0.0637 (13) | 0.0029 (9) | 0.0031 (9) | −0.0041 (10) |
| C14′ | 0.103 (11) | 0.060 (7) | 0.082 (9) | 0.005 (7) | −0.001 (8) | −0.031 (6) |
| C13′ | 0.102 (12) | 0.102 (11) | 0.117 (12) | −0.003 (8) | 0.006 (9) | −0.044 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.385 (4) | C16—O6 | 1.300 (3) |
| C1—C6 | 1.401 (4) | C17—C18 | 1.460 (5) |
| C1—C8 | 1.518 (4) | C17—O6 | 1.465 (4) |
| C2—C3 | 1.388 (4) | C17—H17A | 0.9700 |
| C2—H2 | 0.9300 | C17—H17B | 0.9700 |
| C3—C4 | 1.377 (5) | C18—H18A | 0.9600 |
| C3—H3 | 0.9300 | C18—H18B | 0.9600 |
| C4—C5 | 1.377 (4) | C18—H18C | 0.9600 |
| C4—H4 | 0.9300 | C19—N4 | 1.453 (3) |
| C5—C6 | 1.391 (4) | C19—C21 | 1.520 (4) |
| C5—H5 | 0.9300 | C19—H19A | 0.9700 |
| C6—C7 | 1.511 (4) | C19—H19B | 0.9700 |
| C7—N1 | 1.459 (3) | C20—N3 | 1.466 (3) |
| C7—H7A | 0.9700 | C20—C22 | 1.515 (4) |
| C7—H7B | 0.9700 | C20—H20A | 0.9700 |
| C8—N2 | 1.464 (3) | C20—H20B | 0.9700 |
| C8—H8A | 0.9700 | C21—C26 | 1.387 (4) |
| C8—H8B | 0.9700 | C21—C22 | 1.403 (4) |
| C9—O1 | 1.208 (3) | C22—C23 | 1.389 (4) |
| C9—N1 | 1.362 (3) | C23—C24 | 1.376 (4) |
| C9—N3 | 1.386 (3) | C23—H23 | 0.9300 |
| C10—O2 | 1.206 (3) | C24—C25 | 1.375 (5) |
| C10—N4 | 1.367 (3) | C24—H24 | 0.9300 |
| C10—N2 | 1.389 (3) | C25—C26 | 1.378 (4) |
| C11—N2 | 1.439 (3) | C25—H25 | 0.9300 |
| C11—N1 | 1.441 (3) | C26—H26 | 0.9300 |
| C11—C12 | 1.543 (4) | C27—C28 | 1.480 (8) |
| C11—C15 | 1.573 (3) | C27—Cl1 | 1.742 (7) |
| C12—O3 | 1.186 (4) | C27—H27A | 0.9700 |
| C12—O4 | 1.292 (4) | C27—H27B | 0.9700 |
| C14—C13 | 1.496 (8) | C28—Cl2 | 1.784 (5) |
| C14—H14A | 0.9600 | C28—H28A | 0.9700 |
| C14—H14B | 0.9600 | C28—H28B | 0.9700 |

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| C14—H14C | 0.9600 | O4—C13′ | 1.438 (9) |
| C13—O4 | 1.455 (5) | C14′—C13′ | 1.500 (9) |
| C13—H13A | 0.9700 | C14′—H14D | 0.9600 |
| C13—H13B | 0.9700 | C14′—H14E | 0.9600 |
| C15—N3 | 1.435 (3) | C14′—H14F | 0.9600 |
| C15—N4 | 1.440 (3) | C13′—H13C | 0.9700 |
| C15—C16 | 1.547 (4) | C13′—H13D | 0.9700 |
| C16—O5 | 1.175 (3) | | |

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|---|---|---|---|
| C2—C1—C6 | 118.6 (3) | H18B—C18—H18C | 109.5 |
| C2—C1—C8 | 118.8 (2) | N4—C19—C21 | 113.5 (2) |
| C6—C1—C8 | 122.5 (2) | N4—C19—H19A | 108.9 |
| C1—C2—C3 | 121.4 (3) | C21—C19—H19A | 108.9 |
| C1—C2—H2 | 119.3 | N4—C19—H19B | 108.9 |
| C3—C2—H2 | 119.3 | C21—C19—H19B | 108.9 |
| C4—C3—C2 | 119.8 (3) | H19A—C19—H19B | 107.7 |
| C4—C3—H3 | 120.1 | N3—C20—C22 | 115.3 (2) |
| C2—C3—H3 | 120.1 | N3—C20—H20A | 108.4 |
| C3—C4—C5 | 119.3 (3) | C22—C20—H20A | 108.4 |
| C3—C4—H4 | 120.3 | N3—C20—H20B | 108.4 |
| C5—C4—H4 | 120.3 | C22—C20—H20B | 108.4 |
| C4—C5—C6 | 121.6 (3) | H20A—C20—H20B | 107.5 |
| C4—C5—H5 | 119.2 | C26—C21—C22 | 119.2 (3) |
| C6—C5—H5 | 119.2 | C26—C21—C19 | 119.6 (2) |
| C5—C6—C1 | 119.2 (3) | C22—C21—C19 | 121.3 (2) |
| C5—C6—C7 | 118.8 (2) | C23—C22—C21 | 118.6 (3) |
| C1—C6—C7 | 122.0 (2) | C23—C22—C20 | 119.3 (2) |
| N1—C7—C6 | 112.8 (2) | C21—C22—C20 | 122.1 (2) |
| N1—C7—H7A | 109.0 | C24—C23—C22 | 121.4 (3) |
| C6—C7—H7A | 109.0 | C24—C23—H23 | 119.3 |
| N1—C7—H7B | 109.0 | C22—C23—H23 | 119.3 |
| C6—C7—H7B | 109.0 | C25—C24—C23 | 119.9 (3) |
| H7A—C7—H7B | 107.8 | C25—C24—H24 | 120.0 |
| N2—C8—C1 | 115.4 (2) | C23—C24—H24 | 120.0 |
| N2—C8—H8A | 108.4 | C24—C25—C26 | 119.7 (3) |
| C1—C8—H8A | 108.4 | C24—C25—H25 | 120.1 |
| N2—C8—H8B | 108.4 | C26—C25—H25 | 120.1 |
| C1—C8—H8B | 108.4 | C25—C26—C21 | 121.2 (3) |
| H8A—C8—H8B | 107.5 | C25—C26—H26 | 119.4 |
| O1—C9—N1 | 126.6 (2) | C21—C26—H26 | 119.4 |
| O1—C9—N3 | 125.6 (2) | C28—C27—Cl1 | 112.0 (4) |
| N1—C9—N3 | 107.8 (2) | C28—C27—H27A | 109.2 |
| O2—C10—N4 | 126.3 (2) | Cl1—C27—H27A | 109.2 |
| O2—C10—N2 | 126.0 (2) | C28—C27—H27B | 109.2 |
| N4—C10—N2 | 107.6 (2) | Cl1—C27—H27B | 109.2 |
| N2—C11—N1 | 113.7 (2) | H27A—C27—H27B | 107.9 |
| N2—C11—C12 | 113.9 (2) | C27—C28—Cl2 | 112.9 (3) |
| N1—C11—C12 | 110.0 (2) | C27—C28—H28A | 109.0 |

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|---|---|---|---|
| N2—C11—C15 | 103.24 (19) | Cl2—C28—H28A | 109.0 |
| N1—C11—C15 | 101.53 (19) | C27—C28—H28B | 109.0 |
| C12—C11—C15 | 113.7 (2) | Cl2—C28—H28B | 109.0 |
| O3—C12—O4 | 125.4 (3) | H28A—C28—H28B | 107.8 |
| O3—C12—C11 | 122.0 (3) | C9—N1—C11 | 113.3 (2) |
| O4—C12—C11 | 112.6 (2) | C9—N1—C7 | 124.3 (2) |
| O4—C13—C14 | 105.1 (6) | C11—N1—C7 | 122.2 (2) |
| O4—C13—H13A | 110.7 | C10—N2—C11 | 110.7 (2) |
| C14—C13—H13A | 110.7 | C10—N2—C8 | 120.4 (2) |
| O4—C13—H13B | 110.7 | C11—N2—C8 | 120.6 (2) |
| C14—C13—H13B | 110.7 | C9—N3—C15 | 111.0 (2) |
| H13A—C13—H13B | 108.8 | C9—N3—C20 | 120.1 (2) |
| N3—C15—N4 | 114.1 (2) | C15—N3—C20 | 120.6 (2) |
| N3—C15—C16 | 113.7 (2) | C10—N4—C15 | 113.5 (2) |
| N4—C15—C16 | 109.9 (2) | C10—N4—C19 | 123.9 (2) |
| N3—C15—C11 | 103.08 (18) | C15—N4—C19 | 122.6 (2) |
| N4—C15—C11 | 101.56 (19) | C12—O4—C13′ | 105.1 (7) |
| C16—C15—C11 | 113.7 (2) | C12—O4—C13 | 119.7 (5) |
| O5—C16—O6 | 125.1 (3) | C13′—O4—C13 | 23.8 (11) |
| O5—C16—C15 | 122.6 (3) | C16—O6—C17 | 115.7 (2) |
| O6—C16—C15 | 112.2 (2) | C13′—C14′—H14D | 109.5 |
| C18—C17—O6 | 107.9 (3) | C13′—C14′—H14E | 109.5 |
| C18—C17—H17A | 110.1 | H14D—C14′—H14E | 109.5 |
| O6—C17—H17A | 110.1 | C13′—C14′—H14F | 109.5 |
| C18—C17—H17B | 110.1 | H14D—C14′—H14F | 109.5 |
| O6—C17—H17B | 110.1 | H14E—C14′—H14F | 109.5 |
| H17A—C17—H17B | 108.4 | O4—C13′—C14′ | 110.2 (11) |
| C17—C18—H18A | 109.5 | O4—C13′—H13C | 109.6 |
| C17—C18—H18B | 109.5 | C14′—C13′—H13C | 109.6 |
| H18A—C18—H18B | 109.5 | O4—C13′—H13D | 109.6 |
| C17—C18—H18C | 109.5 | C14′—C13′—H13D | 109.6 |
| H18A—C18—H18C | 109.5 | H13C—C13′—H13D | 108.1 |

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| C6—C1—C2—C3 | −0.6 (4) | N2—C11—N1—C9 | 120.0 (2) |
| C8—C1—C2—C3 | −177.7 (3) | C12—C11—N1—C9 | −110.9 (2) |
| C1—C2—C3—C4 | 0.7 (5) | C15—C11—N1—C9 | 9.8 (3) |
| C2—C3—C4—C5 | −0.4 (5) | N2—C11—N1—C7 | −63.1 (3) |
| C3—C4—C5—C6 | 0.1 (5) | C12—C11—N1—C7 | 66.1 (3) |
| C4—C5—C6—C1 | 0.0 (4) | C15—C11—N1—C7 | −173.2 (2) |
| C4—C5—C6—C7 | 179.3 (3) | C6—C7—N1—C9 | −105.6 (3) |
| C2—C1—C6—C5 | 0.3 (4) | C6—C7—N1—C11 | 77.8 (3) |
| C8—C1—C6—C5 | 177.2 (2) | O2—C10—N2—C11 | 167.3 (2) |
| C2—C1—C6—C7 | −179.0 (3) | N4—C10—N2—C11 | −14.3 (3) |
| C8—C1—C6—C7 | −2.0 (4) | O2—C10—N2—C8 | 18.7 (4) |
| C5—C6—C7—N1 | 125.7 (3) | N4—C10—N2—C8 | −162.9 (2) |
| C1—C6—C7—N1 | −55.0 (4) | N1—C11—N2—C10 | −90.4 (2) |
| C2—C1—C8—N2 | −125.1 (3) | C12—C11—N2—C10 | 142.5 (2) |
| C6—C1—C8—N2 | 57.9 (4) | C15—C11—N2—C10 | 18.7 (2) |

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| N2—C11—C12—O3 | 150.2 (3) | N1—C11—N2—C8 | 58.1 (3) |
| N1—C11—C12—O3 | 21.2 (4) | C12—C11—N2—C8 | −69.0 (3) |
| C15—C11—C12—O3 | −91.9 (3) | C15—C11—N2—C8 | 167.2 (2) |
| N2—C11—C12—O4 | −32.4 (3) | C1—C8—N2—C10 | 72.8 (3) |
| N1—C11—C12—O4 | −161.4 (3) | C1—C8—N2—C11 | −72.7 (3) |
| C15—C11—C12—O4 | 85.6 (3) | O1—C9—N3—C15 | 168.2 (2) |
| N2—C11—C15—N3 | −134.01 (19) | N1—C9—N3—C15 | −12.5 (3) |
| N1—C11—C15—N3 | −16.0 (2) | O1—C9—N3—C20 | 19.6 (4) |
| C12—C11—C15—N3 | 102.1 (2) | N1—C9—N3—C20 | −161.1 (2) |
| N2—C11—C15—N4 | −15.6 (2) | N4—C15—N3—C9 | −91.4 (2) |
| N1—C11—C15—N4 | 102.4 (2) | C16—C15—N3—C9 | 141.5 (2) |
| C12—C11—C15—N4 | −139.5 (2) | C11—C15—N3—C9 | 17.9 (3) |
| N2—C11—C15—C16 | 102.4 (2) | N4—C15—N3—C20 | 57.1 (3) |
| N1—C11—C15—C16 | −139.6 (2) | C16—C15—N3—C20 | −70.1 (3) |
| C12—C11—C15—C16 | −21.5 (3) | C11—C15—N3—C20 | 166.3 (2) |
| N3—C15—C16—O5 | 147.3 (3) | C22—C20—N3—C9 | 71.6 (3) |
| N4—C15—C16—O5 | 18.0 (4) | C22—C20—N3—C15 | −74.0 (3) |
| C11—C15—C16—O5 | −95.1 (3) | O2—C10—N4—C15 | −178.7 (2) |
| N3—C15—C16—O6 | −34.2 (3) | N2—C10—N4—C15 | 2.9 (3) |
| N4—C15—C16—O6 | −163.5 (2) | O2—C10—N4—C19 | 0.3 (4) |
| C11—C15—C16—O6 | 83.4 (3) | N2—C10—N4—C19 | −178.1 (2) |
| N4—C19—C21—C26 | 124.3 (3) | N3—C15—N4—C10 | 118.4 (2) |
| N4—C19—C21—C22 | −56.0 (3) | C16—C15—N4—C10 | −112.5 (2) |
| C26—C21—C22—C23 | −0.7 (4) | C11—C15—N4—C10 | 8.3 (3) |
| C19—C21—C22—C23 | 179.7 (2) | N3—C15—N4—C19 | −60.6 (3) |
| C26—C21—C22—C20 | 178.0 (2) | C16—C15—N4—C19 | 68.5 (3) |
| C19—C21—C22—C20 | −1.6 (4) | C11—C15—N4—C19 | −170.8 (2) |
| N3—C20—C22—C23 | −122.5 (3) | C21—C19—N4—C10 | −102.0 (3) |
| N3—C20—C22—C21 | 58.8 (3) | C21—C19—N4—C15 | 76.9 (3) |
| C21—C22—C23—C24 | 1.2 (4) | O3—C12—O4—C13′ | −12.5 (15) |
| C20—C22—C23—C24 | −177.6 (3) | C11—C12—O4—C13′ | 170.2 (14) |
| C22—C23—C24—C25 | −0.7 (4) | O3—C12—O4—C13 | 8.0 (7) |
| C23—C24—C25—C26 | −0.4 (5) | C11—C12—O4—C13 | −169.3 (5) |
| C24—C25—C26—C21 | 0.8 (4) | C14—C13—O4—C12 | −88.2 (9) |
| C22—C21—C26—C25 | −0.3 (4) | C14—C13—O4—C13′ | −32 (3) |
| C19—C21—C26—C25 | 179.3 (3) | O5—C16—O6—C17 | 4.0 (5) |
| Cl1—C27—C28—Cl2 | −66.8 (4) | C15—C16—O6—C17 | −174.5 (3) |
| O1—C9—N1—C11 | −180.0 (2) | C18—C17—O6—C16 | −177.6 (3) |
| N3—C9—N1—C11 | 0.7 (3) | C12—O4—C13′—C14′ | −170.1 (18) |
| O1—C9—N1—C7 | 3.2 (4) | C13—O4—C13′—C14′ | 58.7 (18) |
| N3—C9—N1—C7 | −176.2 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C28—H28*B*···*Cg*2 | 0.97 | 2.55 | 3.476 (3) | 160 |
| C5—H5···O5$^{i}$ | 0.93 | 2.59 | 3.393 (4) | 145 |
| C7—H7*B*···O5$^{i}$ | 0.97 | 2.50 | 3.359 (3) | 148 |

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|---|---|---|---|---|
| C14—H14*A*···O2$^{ii}$ | 0.96 | 2.57 | 3.514 (9) | 168 |
| C14′—H14*F*···O2$^{ii}$ | 0.96 | 2.47 | 3.351 (6) | 153 |

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