# syn-5,10,15-Tris(dichloromethyl)-5,10,15-trihydroxy-5H-diindeno-[1,2-a:1′,2′-c]fluorene dichloromethane 0.82-solvate

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The title compound, $C_{30}H_{18}Cl_6O_3\cdot0.82CH_2Cl_2$, consists of a slightly cup-shaped seven-ring truxene nucleus with hydroxy and dichloromethyl substituents at stereocenters 5R/S, 10R/S and 15R/S. C—Cl distances are in the range 1.759 (4)–1.783 (3) Å. Solvent channels parallel to the $b$ axis appear to be partially occupied by highly disordered dichloromethane solvent molecules, the contribution of which were removed from the refinement with the SQUEEZE procedure in *PLATON* [Spek (2009). *Acta Cryst.* **D65**, 148–155]. Only one of the OH groups forms a hydrogen bond, which is intermolecular to another OH group, forming centrosymmetric dimers in the crystal.

## Related literature

For further details of the synthesis and information on the synthesis of buckybowls, see: Abdourazak *et al.* (1995). For applications of truxenes, see: Diring & Ziessel (2009). Similar structures have been reported by De Frutos *et al.* (1999); Amsharov & Jansen (2007) and Menard *et al.* (2011).
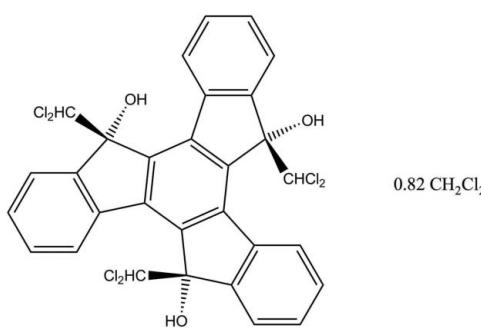


0.82 CH₂Cl₂

## Experimental

### Crystal data

| | |
|---|---|
| $C_{30}H_{18}Cl_6O_3\cdot0.82CH_2Cl_2$ | $\gamma$ = 68.798 (2)° |
| $M_r$ = 708.78 | $V$ = 1576.88 (9) Å³ |
| Triclinic, $P\bar{1}$ | $Z$ = 2 |
| $a$ = 10.9719 (4) Å | Mo $K\alpha$ radiation |
| $b$ = 11.6186 (3) Å | $\mu$ = 0.72 mm⁻¹ |
| $c$ = 14.0431 (5) Å | $T$ = 90 K |
| $\alpha$ = 71.009 (2)° | 0.38 × 0.13 × 0.05 mm |
| $\beta$ = 85.291 (2)° | |

### Data collection

| | |
|---|---|
| Nonius KappaCCD diffractometer with an Oxford Cryostream cooler | Minor, 1997) |
| | $T_{min}$ = 0.814, $T_{max}$ = 0.972 |
| Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & | 10851 measured reflections |
| | 5744 independent reflections |
| | 3608 reflections with $I > 2\sigma(I)$ |
| | $R_{int}$ = 0.046 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.053 | 356 parameters |
| $wR(F^2)$ = 0.156 | H-atom parameters constrained |
| $S$ = 0.97 | $\Delta\rho_{max}$ = 0.42 e Å⁻³ |
| 5744 reflections | $\Delta\rho_{min}$ = −0.32 e Å⁻³ |

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

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| O1—H91$\cdots$O3$^i$ | 0.84 | 2.04 | 2.834 (3) | 158 |

Symmetry code: (i) $-x+2, -y+1, -z+1$.

Data collection: *COLLECT* (Bruker, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KJ2201).

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# supplementary materials

## *syn*-5,10,15-Tris(dichloromethyl)-5,10,15-trihydroxy-5*H*-diindeno[1,2-*a*:1′,2′-*c*]fluorene dichloromethane 0.82-solvate

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### Comment

The nucleus of the title compound is truxene ($C_{27}H_{18}$, CAS: 548–35-6), a nearly planar seven-ring aromatic molecule. Compounds containing this ring system have been previously investigated for use in liquid crystal devices, chiral recognition systems, and fluorescent probes (Diring & Ziessel, 2009). The title compound was synthesized as an intermediate material in the formation of buckybowls (half-buckminsterfullerenes, Abdourazak *et al.*, 1995). Two isomers were separated by chromatography, and the yellow component is herein shown to be the *syn* isomer, with all three OH groups on the same side of the truxene nucleus. The molecule has a slightly cupped shape, with three hydroxy groups oriented toward the inside of the cup and three dichloromethyl groups on the outside of the cup. Relative to the mean plane of the central 6-ring (which is a slightly puckered crown, $\delta$(r.m.s.) = 0.01 (1) Å), the three pairs of carbon atoms on the outer rim of the molecule average 0.36 (1) (C4, C5), 0.15 (1) (C13, C14) and 0.07 (1) (C22, C23) Å above the plane. Of the three OH groups available for hydrogen bond formation, only O1 forms a hydrogen bond, to OH group O3 at 2 - *x*, 1 - *y*, 1 - *z*, thus there are centrosymmetric dimers about 1, 1/2, 1/2, as shown in Fig. 2. A solvent channel with a unit cell volume of 330 Å³, parallel to the **b** axis and centered at 1/2**a**, displays residual electron density which presumably represents remnants of disordered solvent molecules most of which have evaporated from the crystal since the original synthesis. Procedure SQUEEZE, as implemented in *PLATON* (Spek, 2009), subtracted 69 electrons from the observed structure amplitudes as an approximate solvent contribution.

### Experimental

A solution of lithium dicyclohexylamine was prepared by adding 93 mmol dicyclohexeylamine and 93 mmol n-butyl-lithium to 75 ml of dried tetrahydrofuran (THF). This solution was added dropwise over one hour to a suspension of 7.3 mmol truxenone in 100 ml of THF and 6 ml of dichloromethane (DCM) at 273 K. The solution was stirred for one hour and then quenched with aqueous ammonium chloride. The THF was removed under reduced pressure and the remaining mixture was extracted with DCM. The resulting organic layer was washed with aqueous citric acid, dried and evaporated. Flash chromatography (silica gel, DCM) was used to isolate 5,10,15-tris(dichloromethyl)-5,10,15-trihydroxy-5*H*-Di-indeno[1,2 - a:1′,2′-c] fluorene and further chromatography (silica gel, DCM-hexane 3:1) was used to separate the compound into two components. The *syn*- component reported here crystallized from DCM as yellow blades.

### Refinement

Observed structure amplitudes were modified by *PLATON* to eliminate diffuse electron density found in the solvent accessible channel. All H atoms were placed in calculated positions, guided by difference maps, with C—H bond distances 0.95 (aromatic-H) and 1.00 (alkyl-H) Å, O—H distances 0.84 Å, and displacement parameters $U_{iso}=1.2U_{eq}$ (aromatic and alkyl C) and $1.5U_{eq}$ (hydroxyl-O), and thereafter refined as riding. A torsional parameter was refined for

each OH group.

## Computing details

Data collection: *COLLECT* (Bruker, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).
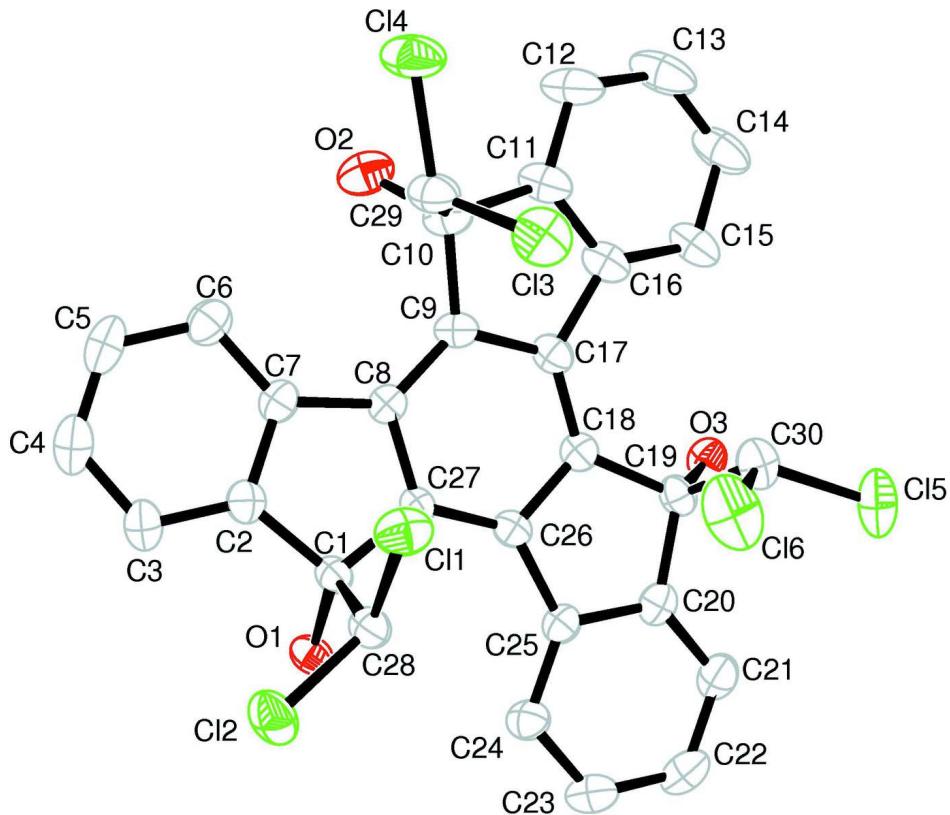


**Figure 1**

View of the title compound (50% probability displacement ellipsoids). H atoms are not shown.
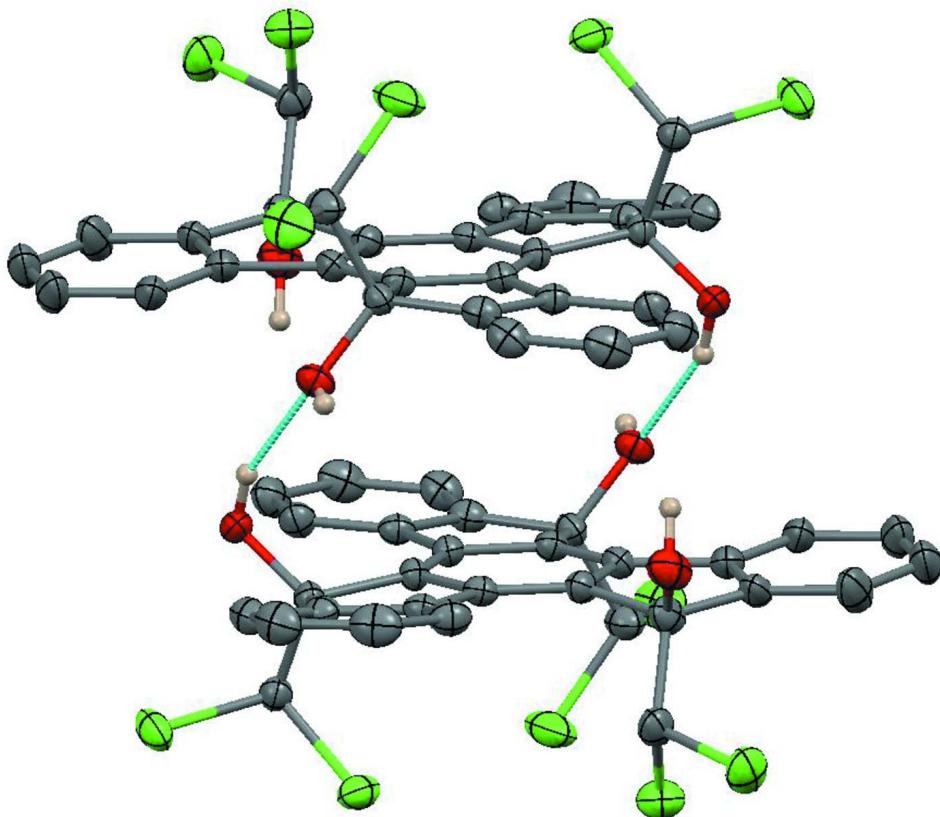
**Figure 2**

Hydrogen-bonded dimer with 40% probability ellipsoids. Only OH hydrogen atoms are illustrated.

**syn-5,10,15-Tris(dichloromethyl)-5,10,15-trihydroxy- 5H-diindeno[1,2-a:1′,2′-c]fluorene dichloromethane 0.82-solvate**

*Crystal data*
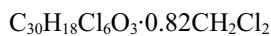
| | |
|---|---|
| $C_{30}H_{18}Cl_6O_3 \cdot 0.82CH_2Cl_2$ | $Z = 2$ |
| $M_r = 708.78$ | $F(000) = 717$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.493$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 10.9719$ (4) Å | Cell parameters from 5548 reflections |
| $b = 11.6186$ (3) Å | $\theta = 2.6–25.4°$ |
| $c = 14.0431$ (5) Å | $\mu = 0.72$ mm$^{-1}$ |
| $\alpha = 71.009$ (2)° | $T = 90$ K |
| $\beta = 85.291$ (2)° | Blade, yellow |
| $\gamma = 68.798$ (2)° | $0.38 \times 0.13 \times 0.05$ mm |
| $V = 1576.88$ (9) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Nonius KappaCCD diffractometer with an Oxford Cryostream cooler | Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997) |
| | $T_{min} = 0.814$, $T_{max} = 0.972$ |
| Radiation source: fine-focus sealed tube | 10851 measured reflections |
| Graphite monochromator | 5744 independent reflections |
| Detector resolution: 9 pixels mm$^{-1}$ | 3608 reflections with $I > 2\sigma(I)$ |
| CCD rotation images, thick slices scans | $R_{int} = 0.046$ |

$\theta_{max} = 25.4°$, $\theta_{min} = 2.6°$

$h = -13 \rightarrow 13$

$k = -13 \rightarrow 13$

$l = -16 \rightarrow 16$

*Refinement*

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.053$

$wR(F^2) = 0.156$

$S = 0.97$

5744 reflections

356 parameters

0 restraints

0 constraints

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

$w = 1/[\sigma^2(F_o^2) + (0.0932P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.42$ e Å$^{-3}$

$\Delta\rho_{min} = -0.32$ e Å$^{-3}$

Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc$^*$=kFc[1+0.001xFc$^2\lambda^3$/sin(2$\theta$)]$^{-1/4}$

Extinction coefficient: 0.0066 (17)

*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 $F^2 > 2\sigma(F^2)$ 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 | 0.9165 (3) | 0.2835 (3) | 0.4130 (2) | 0.0263 (8) |
| C2 | 1.0278 (3) | 0.2228 (3) | 0.3532 (2) | 0.0285 (8) |
| C3 | 1.0854 (4) | 0.0926 (3) | 0.3608 (3) | 0.0350 (9) |
| H3 | 1.0534 | 0.0294 | 0.405 | 0.042* |
| C4 | 1.1916 (4) | 0.0571 (3) | 0.3017 (3) | 0.0456 (10) |
| H4 | 1.2323 | −0.0319 | 0.3059 | 0.055* |
| C5 | 1.2389 (4) | 0.1469 (3) | 0.2375 (3) | 0.0457 (10) |
| H5 | 1.31 | 0.1198 | 0.1967 | 0.055* |
| C6 | 1.1834 (4) | 0.2786 (3) | 0.2315 (3) | 0.0364 (9) |
| H6 | 1.217 | 0.3409 | 0.1879 | 0.044* |
| C7 | 1.0783 (3) | 0.3157 (3) | 0.2909 (2) | 0.0290 (8) |
| C8 | 1.0065 (3) | 0.4414 (3) | 0.3096 (2) | 0.0256 (8) |
| C9 | 1.0110 (3) | 0.5655 (3) | 0.2661 (2) | 0.0287 (8) |
| C10 | 1.0801 (4) | 0.6173 (3) | 0.1721 (3) | 0.0354 (9) |
| C11 | 1.0489 (4) | 0.7564 (3) | 0.1689 (2) | 0.0373 (9) |
| C12 | 1.1007 (5) | 0.8464 (4) | 0.1053 (3) | 0.0513 (11) |
| H12 | 1.1611 | 0.8243 | 0.056 | 0.062* |
| C13 | 1.0610 (5) | 0.9699 (4) | 0.1163 (3) | 0.0573 (12) |
| H13 | 1.0946 | 1.0329 | 0.0739 | 0.069* |
| C14 | 0.9740 (5) | 1.0007 (3) | 0.1877 (3) | 0.0511 (11) |
| H14 | 0.9473 | 1.0856 | 0.1934 | 0.061* |

| | | | |
|---|---|---|---|
| C15 | 0.9236 (4) | 0.9111 (3) | 0.2522 (3) | 0.0393 (9) |
| H15 | 0.864 | 0.9338 | 0.3018 | 0.047* |
| C16 | 0.9621 (4) | 0.7877 (3) | 0.2427 (2) | 0.0341 (9) |
| C17 | 0.9350 (3) | 0.6691 (3) | 0.3023 (2) | 0.0290 (8) |
| C18 | 0.8517 (3) | 0.6459 (3) | 0.3803 (2) | 0.0268 (8) |
| C19 | 0.7502 (3) | 0.7421 (3) | 0.4248 (2) | 0.0286 (8) |
| C20 | 0.6942 (3) | 0.6552 (3) | 0.5068 (2) | 0.0306 (8) |
| C21 | 0.6064 (4) | 0.6882 (3) | 0.5771 (3) | 0.0379 (9) |
| H21 | 0.5725 | 0.7754 | 0.5784 | 0.046* |
| C22 | 0.5674 (4) | 0.5929 (4) | 0.6468 (3) | 0.0430 (10) |
| H22 | 0.5065 | 0.6146 | 0.6959 | 0.052* |
| C23 | 0.6178 (4) | 0.4662 (4) | 0.6442 (3) | 0.0409 (9) |
| H23 | 0.5888 | 0.402 | 0.6906 | 0.049* |
| C24 | 0.7099 (4) | 0.4311 (3) | 0.5749 (2) | 0.0328 (8) |
| H24 | 0.7458 | 0.3434 | 0.5751 | 0.039* |
| C25 | 0.7485 (3) | 0.5262 (3) | 0.5056 (2) | 0.0273 (8) |
| C26 | 0.8442 (3) | 0.5207 (3) | 0.4249 (2) | 0.0259 (7) |
| C27 | 0.9198 (3) | 0.4203 (3) | 0.3888 (2) | 0.0250 (7) |
| C28 | 0.7807 (3) | 0.2974 (3) | 0.3769 (2) | 0.0301 (8) |
| H28 | 0.7126 | 0.3475 | 0.4147 | 0.036* |
| C29 | 1.0181 (4) | 0.6046 (3) | 0.0826 (2) | 0.0381 (9) |
| H29 | 1.0298 | 0.5109 | 0.0982 | 0.046* |
| C30 | 0.6479 (4) | 0.8437 (3) | 0.3406 (3) | 0.0382 (9) |
| H30 | 0.6955 | 0.8851 | 0.2841 | 0.046* |
| Cl1 | 0.75059 (9) | 0.38564 (9) | 0.24653 (6) | 0.0407 (3) |
| Cl2 | 0.76348 (10) | 0.14417 (8) | 0.40087 (7) | 0.0441 (3) |
| Cl3 | 0.84936 (10) | 0.69516 (9) | 0.06527 (7) | 0.0513 (3) |
| Cl4 | 1.09646 (12) | 0.65136 (9) | −0.03237 (7) | 0.0540 (3) |
| Cl5 | 0.53505 (11) | 0.96814 (9) | 0.38451 (9) | 0.0596 (3) |
| Cl6 | 0.56204 (12) | 0.76978 (10) | 0.29285 (9) | 0.0644 (4) |
| O1 | 0.9273 (2) | 0.21078 (19) | 0.51741 (15) | 0.0296 (5) |
| H91 | 1.0026 | 0.1931 | 0.5403 | 0.044* |
| O2 | 1.2147 (3) | 0.5482 (3) | 0.17035 (19) | 0.0452 (7) |
| H92 | 1.2533 | 0.5455 | 0.2207 | 0.068* |
| O3 | 0.8099 (2) | 0.8111 (2) | 0.46100 (17) | 0.0318 (6) |
| H93 | 0.764 | 0.8384 | 0.5054 | 0.048* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.030 (2) | 0.0224 (14) | 0.0263 (17) | −0.0106 (14) | 0.0010 (15) | −0.0066 (13) |
| C2 | 0.026 (2) | 0.0287 (16) | 0.0279 (18) | −0.0063 (14) | −0.0052 (15) | −0.0080 (14) |
| C3 | 0.032 (2) | 0.0283 (16) | 0.043 (2) | −0.0056 (15) | −0.0052 (18) | −0.0143 (15) |
| C4 | 0.039 (2) | 0.0364 (19) | 0.059 (3) | −0.0018 (18) | −0.004 (2) | −0.0236 (19) |
| C5 | 0.033 (2) | 0.048 (2) | 0.053 (2) | 0.0002 (19) | 0.001 (2) | −0.029 (2) |
| C6 | 0.034 (2) | 0.0423 (19) | 0.0331 (19) | −0.0113 (17) | 0.0019 (17) | −0.0151 (16) |
| C7 | 0.027 (2) | 0.0331 (16) | 0.0288 (18) | −0.0097 (15) | 0.0005 (16) | −0.0140 (15) |
| C8 | 0.0245 (19) | 0.0295 (16) | 0.0249 (17) | −0.0098 (14) | −0.0005 (15) | −0.0107 (14) |
| C9 | 0.032 (2) | 0.0347 (17) | 0.0242 (17) | −0.0182 (15) | 0.0033 (15) | −0.0093 (14) |
| C10 | 0.038 (2) | 0.0448 (19) | 0.0316 (19) | −0.0234 (18) | 0.0078 (17) | −0.0142 (16) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C11 | 0.057 (3) | 0.0422 (19) | 0.0252 (18) | −0.0323 (18) | −0.0001 (18) | −0.0098 (16) |
| C12 | 0.079 (3) | 0.064 (2) | 0.032 (2) | −0.050 (2) | 0.018 (2) | −0.0173 (19) |
| C13 | 0.098 (4) | 0.054 (2) | 0.039 (2) | −0.056 (3) | 0.001 (3) | −0.0072 (19) |
| C14 | 0.092 (4) | 0.0381 (19) | 0.036 (2) | −0.039 (2) | −0.001 (2) | −0.0082 (17) |
| C15 | 0.061 (3) | 0.0322 (17) | 0.0283 (18) | −0.0247 (18) | −0.0066 (18) | −0.0034 (15) |
| C16 | 0.044 (2) | 0.0349 (17) | 0.0269 (18) | −0.0211 (16) | −0.0052 (17) | −0.0050 (15) |
| C17 | 0.035 (2) | 0.0275 (15) | 0.0279 (18) | −0.0148 (15) | −0.0020 (16) | −0.0088 (14) |
| C18 | 0.030 (2) | 0.0279 (15) | 0.0244 (17) | −0.0108 (14) | −0.0003 (15) | −0.0099 (14) |
| C19 | 0.031 (2) | 0.0244 (15) | 0.0323 (18) | −0.0115 (14) | −0.0024 (16) | −0.0094 (14) |
| C20 | 0.027 (2) | 0.0344 (17) | 0.0325 (19) | −0.0086 (15) | −0.0002 (16) | −0.0150 (15) |
| C21 | 0.030 (2) | 0.0397 (18) | 0.047 (2) | −0.0106 (16) | 0.0038 (19) | −0.0201 (17) |
| C22 | 0.033 (2) | 0.059 (2) | 0.045 (2) | −0.0178 (19) | 0.0121 (19) | −0.0277 (19) |
| C23 | 0.042 (2) | 0.054 (2) | 0.037 (2) | −0.0279 (19) | 0.0097 (19) | −0.0162 (18) |
| C24 | 0.039 (2) | 0.0364 (17) | 0.0286 (18) | −0.0171 (16) | 0.0061 (17) | −0.0143 (15) |
| C25 | 0.027 (2) | 0.0311 (16) | 0.0253 (17) | −0.0093 (15) | 0.0008 (15) | −0.0115 (14) |
| C26 | 0.028 (2) | 0.0259 (15) | 0.0241 (17) | −0.0112 (14) | −0.0016 (15) | −0.0061 (13) |
| C27 | 0.0277 (19) | 0.0213 (14) | 0.0256 (17) | −0.0089 (14) | 0.0002 (15) | −0.0066 (13) |
| C28 | 0.036 (2) | 0.0278 (15) | 0.0273 (17) | −0.0127 (15) | 0.0015 (16) | −0.0082 (14) |
| C29 | 0.052 (3) | 0.0415 (18) | 0.0271 (18) | −0.0252 (18) | 0.0081 (18) | −0.0115 (15) |
| C30 | 0.038 (2) | 0.0342 (17) | 0.042 (2) | −0.0103 (16) | −0.0083 (18) | −0.0120 (16) |
| Cl1 | 0.0387 (6) | 0.0539 (5) | 0.0282 (5) | −0.0186 (4) | −0.0025 (4) | −0.0078 (4) |
| Cl2 | 0.0459 (6) | 0.0337 (4) | 0.0604 (6) | −0.0205 (4) | −0.0039 (5) | −0.0163 (4) |
| Cl3 | 0.0539 (7) | 0.0569 (6) | 0.0443 (6) | −0.0206 (5) | −0.0086 (5) | −0.0141 (5) |
| Cl4 | 0.0835 (9) | 0.0613 (6) | 0.0301 (5) | −0.0419 (6) | 0.0162 (5) | −0.0163 (4) |
| Cl5 | 0.0493 (7) | 0.0383 (5) | 0.0795 (8) | 0.0047 (5) | −0.0130 (6) | −0.0230 (5) |
| Cl6 | 0.0675 (8) | 0.0548 (6) | 0.0770 (8) | −0.0218 (5) | −0.0331 (6) | −0.0202 (5) |
| O1 | 0.0374 (15) | 0.0285 (11) | 0.0235 (12) | −0.0147 (11) | −0.0026 (11) | −0.0046 (9) |
| O2 | 0.0409 (18) | 0.0636 (15) | 0.0411 (15) | −0.0277 (14) | 0.0087 (13) | −0.0209 (13) |
| O3 | 0.0332 (15) | 0.0343 (12) | 0.0341 (13) | −0.0123 (11) | 0.0019 (11) | −0.0185 (10) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—O1 | 1.424 (4) | C16—C17 | 1.481 (5) |
| C1—C2 | 1.514 (5) | C17—C18 | 1.388 (5) |
| C1—C27 | 1.526 (4) | C18—C26 | 1.415 (4) |
| C1—C28 | 1.550 (5) | C18—C19 | 1.523 (4) |
| C2—C3 | 1.383 (4) | C19—O3 | 1.424 (4) |
| C2—C7 | 1.406 (5) | C19—C20 | 1.516 (5) |
| C3—C4 | 1.390 (5) | C19—C30 | 1.550 (5) |
| C3—H3 | 0.95 | C20—C21 | 1.369 (5) |
| C4—C5 | 1.369 (6) | C20—C25 | 1.403 (4) |
| C4—H4 | 0.95 | C21—C22 | 1.392 (5) |
| C5—C6 | 1.403 (5) | C21—H21 | 0.95 |
| C5—H5 | 0.95 | C22—C23 | 1.384 (5) |
| C6—C7 | 1.389 (5) | C22—H22 | 0.95 |
| C6—H6 | 0.95 | C23—C24 | 1.391 (5) |
| C7—C8 | 1.485 (4) | C23—H23 | 0.95 |
| C8—C9 | 1.388 (4) | C24—C25 | 1.384 (4) |
| C8—C27 | 1.421 (4) | C24—H24 | 0.95 |
| C9—C17 | 1.416 (4) | C25—C26 | 1.481 (4) |

| | | | |
|---|---|---|---|
| C9—C10 | 1.525 (4) | C26—C27 | 1.387 (4) |
| C10—O2 | 1.406 (4) | C28—Cl1 | 1.773 (3) |
| C10—C11 | 1.512 (5) | C28—Cl2 | 1.778 (3) |
| C10—C29 | 1.549 (5) | C28—H28 | 1 |
| C11—C16 | 1.396 (5) | C29—Cl3 | 1.759 (4) |
| C11—C12 | 1.397 (5) | C29—Cl4 | 1.783 (3) |
| C12—C13 | 1.398 (5) | C29—H29 | 1 |
| C12—H12 | 0.95 | C30—Cl6 | 1.772 (4) |
| C13—C14 | 1.370 (6) | C30—Cl5 | 1.779 (4) |
| C13—H13 | 0.95 | C30—H30 | 1 |
| C14—C15 | 1.389 (5) | O1—H91 | 0.84 |
| C14—H14 | 0.95 | O2—H92 | 0.84 |
| C15—C16 | 1.389 (4) | O3—H93 | 0.84 |
| C15—H15 | 0.95 | | |
| | | | |
| O1—C1—C2 | 113.5 (2) | C9—C17—C16 | 108.6 (3) |
| O1—C1—C27 | 115.3 (3) | C17—C18—C26 | 121.1 (3) |
| C2—C1—C27 | 102.3 (3) | C17—C18—C19 | 129.2 (3) |
| O1—C1—C28 | 104.8 (3) | C26—C18—C19 | 109.5 (3) |
| C2—C1—C28 | 113.4 (3) | O3—C19—C20 | 113.9 (3) |
| C27—C1—C28 | 107.7 (2) | O3—C19—C18 | 110.5 (3) |
| C3—C2—C7 | 121.1 (3) | C20—C19—C18 | 102.7 (2) |
| C3—C2—C1 | 127.6 (3) | O3—C19—C30 | 107.4 (2) |
| C7—C2—C1 | 111.0 (3) | C20—C19—C30 | 113.4 (3) |
| C2—C3—C4 | 118.0 (3) | C18—C19—C30 | 108.8 (3) |
| C2—C3—H3 | 121 | C21—C20—C25 | 121.2 (3) |
| C4—C3—H3 | 121 | C21—C20—C19 | 128.3 (3) |
| C5—C4—C3 | 121.8 (3) | C25—C20—C19 | 110.5 (3) |
| C5—C4—H4 | 119.1 | C20—C21—C22 | 119.3 (3) |
| C3—C4—H4 | 119.1 | C20—C21—H21 | 120.3 |
| C4—C5—C6 | 120.6 (4) | C22—C21—H21 | 120.3 |
| C4—C5—H5 | 119.7 | C23—C22—C21 | 119.8 (3) |
| C6—C5—H5 | 119.7 | C23—C22—H22 | 120.1 |
| C7—C6—C5 | 118.5 (3) | C21—C22—H22 | 120.1 |
| C7—C6—H6 | 120.8 | C22—C23—C24 | 121.2 (3) |
| C5—C6—H6 | 120.8 | C22—C23—H23 | 119.4 |
| C6—C7—C2 | 120.0 (3) | C24—C23—H23 | 119.4 |
| C6—C7—C8 | 132.0 (3) | C25—C24—C23 | 118.9 (3) |
| C2—C7—C8 | 107.9 (3) | C25—C24—H24 | 120.5 |
| C9—C8—C27 | 119.1 (3) | C23—C24—H24 | 120.5 |
| C9—C8—C7 | 132.6 (3) | C24—C25—C20 | 119.6 (3) |
| C27—C8—C7 | 108.4 (3) | C24—C25—C26 | 132.1 (3) |
| C8—C9—C17 | 121.0 (3) | C20—C25—C26 | 108.3 (3) |
| C8—C9—C10 | 129.1 (3) | C27—C26—C18 | 119.2 (3) |
| C17—C9—C10 | 109.6 (3) | C27—C26—C25 | 132.1 (3) |
| O2—C10—C11 | 113.4 (3) | C18—C26—C25 | 108.6 (3) |
| O2—C10—C9 | 116.3 (3) | C26—C27—C8 | 120.7 (3) |
| C11—C10—C9 | 101.9 (3) | C26—C27—C1 | 129.5 (3) |
| O2—C10—C29 | 104.8 (3) | C8—C27—C1 | 109.4 (3) |

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| C11—C10—C29 | 113.9 (3) | C1—C28—Cl1 | 111.0 (2) |
| C9—C10—C29 | 106.7 (3) | C1—C28—Cl2 | 112.4 (2) |
| C16—C11—C12 | 121.1 (3) | Cl1—C28—Cl2 | 109.33 (18) |
| C16—C11—C10 | 111.7 (3) | C1—C28—H28 | 108 |
| C12—C11—C10 | 127.2 (3) | Cl1—C28—H28 | 108 |
| C11—C12—C13 | 118.1 (4) | Cl2—C28—H28 | 108 |
| C11—C12—H12 | 120.9 | C10—C29—Cl3 | 112.3 (2) |
| C13—C12—H12 | 120.9 | C10—C29—Cl4 | 112.2 (3) |
| C14—C13—C12 | 120.5 (4) | Cl3—C29—Cl4 | 109.22 (19) |
| C14—C13—H13 | 119.8 | C10—C29—H29 | 107.6 |
| C12—C13—H13 | 119.8 | Cl3—C29—H29 | 107.6 |
| C13—C14—C15 | 121.7 (3) | Cl4—C29—H29 | 107.6 |
| C13—C14—H14 | 119.2 | C19—C30—Cl6 | 111.6 (2) |
| C15—C14—H14 | 119.2 | C19—C30—Cl5 | 111.2 (2) |
| C16—C15—C14 | 118.7 (4) | Cl6—C30—Cl5 | 109.7 (2) |
| C16—C15—H15 | 120.6 | C19—C30—H30 | 108.1 |
| C14—C15—H15 | 120.6 | Cl6—C30—H30 | 108.1 |
| C15—C16—C11 | 119.9 (3) | Cl5—C30—H30 | 108.1 |
| C15—C16—C17 | 132.3 (3) | C1—O1—H91 | 109.5 |
| C11—C16—C17 | 107.7 (3) | C10—O2—H92 | 109.5 |
| C18—C17—C9 | 118.9 (3) | C19—O3—H93 | 109.5 |
| C18—C17—C16 | 132.5 (3) | | |
| | | | |
| O1—C1—C2—C3 | 42.4 (5) | C17—C18—C19—C20 | −179.8 (3) |
| C27—C1—C2—C3 | 167.2 (3) | C26—C18—C19—C20 | 5.5 (3) |
| C28—C1—C2—C3 | −77.1 (4) | C17—C18—C19—C30 | 59.8 (4) |
| O1—C1—C2—C7 | −132.3 (3) | C26—C18—C19—C30 | −114.9 (3) |
| C27—C1—C2—C7 | −7.4 (3) | O3—C19—C20—C21 | 54.4 (5) |
| C28—C1—C2—C7 | 108.3 (3) | C18—C19—C20—C21 | 174.0 (3) |
| C7—C2—C3—C4 | −2.6 (5) | C30—C19—C20—C21 | −68.8 (5) |
| C1—C2—C3—C4 | −176.8 (3) | O3—C19—C20—C25 | −123.5 (3) |
| C2—C3—C4—C5 | 0.0 (6) | C18—C19—C20—C25 | −3.9 (4) |
| C3—C4—C5—C6 | 1.8 (6) | C30—C19—C20—C25 | 113.3 (3) |
| C4—C5—C6—C7 | −1.0 (6) | C25—C20—C21—C22 | −2.0 (5) |
| C5—C6—C7—C2 | −1.5 (5) | C19—C20—C21—C22 | −179.7 (3) |
| C5—C6—C7—C8 | 173.5 (3) | C20—C21—C22—C23 | 0.1 (6) |
| C3—C2—C7—C6 | 3.4 (5) | C21—C22—C23—C24 | 1.8 (6) |
| C1—C2—C7—C6 | 178.4 (3) | C22—C23—C24—C25 | −1.9 (5) |
| C3—C2—C7—C8 | −172.7 (3) | C23—C24—C25—C20 | 0.0 (5) |
| C1—C2—C7—C8 | 2.4 (4) | C23—C24—C25—C26 | 178.7 (3) |
| C6—C7—C8—C9 | 8.3 (6) | C21—C20—C25—C24 | 2.0 (5) |
| C2—C7—C8—C9 | −176.2 (4) | C19—C20—C25—C24 | −179.9 (3) |
| C6—C7—C8—C27 | −171.2 (3) | C21—C20—C25—C26 | −177.1 (3) |
| C2—C7—C8—C27 | 4.3 (4) | C19—C20—C25—C26 | 1.0 (4) |
| C27—C8—C9—C17 | 2.7 (5) | C17—C18—C26—C27 | −1.1 (5) |
| C7—C8—C9—C17 | −176.7 (3) | C19—C18—C26—C27 | 174.1 (3) |
| C27—C8—C9—C10 | −170.7 (3) | C17—C18—C26—C25 | 179.6 (3) |
| C7—C8—C9—C10 | 9.8 (6) | C19—C18—C26—C25 | −5.2 (4) |
| C8—C9—C10—O2 | −54.7 (5) | C24—C25—C26—C27 | 4.6 (6) |

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| C17—C9—C10—O2 | 131.2 (3) | C20—C25—C26—C27 | −176.5 (3) |
| C8—C9—C10—C11 | −178.6 (3) | C24—C25—C26—C18 | −176.3 (4) |
| C17—C9—C10—C11 | 7.4 (4) | C20—C25—C26—C18 | 2.6 (4) |
| C8—C9—C10—C29 | 61.8 (4) | C18—C26—C27—C8 | 1.6 (5) |
| C17—C9—C10—C29 | −112.2 (3) | C25—C26—C27—C8 | −179.4 (3) |
| O2—C10—C11—C16 | −131.7 (3) | C18—C26—C27—C1 | −170.7 (3) |
| C9—C10—C11—C16 | −6.0 (4) | C25—C26—C27—C1 | 8.4 (6) |
| C29—C10—C11—C16 | 108.5 (3) | C9—C8—C27—C26 | −2.4 (5) |
| O2—C10—C11—C12 | 45.7 (5) | C7—C8—C27—C26 | 177.2 (3) |
| C9—C10—C11—C12 | 171.4 (4) | C9—C8—C27—C1 | 171.3 (3) |
| C29—C10—C11—C12 | −74.1 (5) | C7—C8—C27—C1 | −9.1 (4) |
| C16—C11—C12—C13 | −1.3 (6) | O1—C1—C27—C26 | −53.4 (5) |
| C10—C11—C12—C13 | −178.4 (4) | C2—C1—C27—C26 | −177.1 (3) |
| C11—C12—C13—C14 | 0.1 (6) | C28—C1—C27—C26 | 63.2 (4) |
| C12—C13—C14—C15 | 0.9 (7) | O1—C1—C27—C8 | 133.7 (3) |
| C13—C14—C15—C16 | −0.7 (6) | C2—C1—C27—C8 | 10.0 (3) |
| C14—C15—C16—C11 | −0.5 (5) | C28—C1—C27—C8 | −109.8 (3) |
| C14—C15—C16—C17 | 175.1 (4) | O1—C1—C28—Cl1 | −179.55 (18) |
| C12—C11—C16—C15 | 1.5 (6) | C2—C1—C28—Cl1 | −55.2 (3) |
| C10—C11—C16—C15 | 179.0 (3) | C27—C1—C28—Cl1 | 57.2 (3) |
| C12—C11—C16—C17 | −175.1 (3) | O1—C1—C28—Cl2 | −56.7 (3) |
| C10—C11—C16—C17 | 2.4 (4) | C2—C1—C28—Cl2 | 67.6 (3) |
| C8—C9—C17—C18 | −2.2 (5) | C27—C1—C28—Cl2 | −179.9 (2) |
| C10—C9—C17—C18 | 172.3 (3) | O2—C10—C29—Cl3 | −175.6 (2) |
| C8—C9—C17—C16 | 179.0 (3) | C11—C10—C29—Cl3 | −51.1 (4) |
| C10—C9—C17—C16 | −6.4 (4) | C9—C10—C29—Cl3 | 60.5 (3) |
| C15—C16—C17—C18 | 8.0 (7) | O2—C10—C29—Cl4 | −52.1 (3) |
| C11—C16—C17—C18 | −176.0 (4) | C11—C10—C29—Cl4 | 72.4 (4) |
| C15—C16—C17—C9 | −173.5 (4) | C9—C10—C29—Cl4 | −176.0 (2) |
| C11—C16—C17—C9 | 2.5 (4) | O3—C19—C30—Cl6 | −177.7 (2) |
| C9—C17—C18—C26 | 1.4 (5) | C20—C19—C30—Cl6 | −50.9 (3) |
| C16—C17—C18—C26 | 179.8 (3) | C18—C19—C30—Cl6 | 62.7 (3) |
| C9—C17—C18—C19 | −172.7 (3) | O3—C19—C30—Cl5 | −54.8 (3) |
| C16—C17—C18—C19 | 5.7 (6) | C20—C19—C30—Cl5 | 71.9 (3) |
| C17—C18—C19—O3 | −57.9 (4) | C18—C19—C30—Cl5 | −174.5 (2) |
| C26—C18—C19—O3 | 127.4 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| O1—H91···O3[i] | 0.84 | 2.04 | 2.834 (3) | 158 |

Symmetry code: (i) −x+2, −y+1, −z+1.