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# 13c-(2-Chloroethoxy)-1,13c-dihydro-2,3-epoxydibenzo[*a*,*kl*]xanthan-1-one

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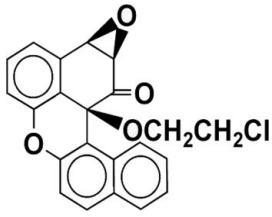
Key indicators: single-crystal X-ray study; $T$ = 193 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.047; $wR$ factor = 0.136; data-to-parameter ratio = 14.0.

The title compound, $C_{22}H_{15}ClO_4$, containing three chiral C atoms, is an intermediate in the design of chiral alcohols. In the crystal structure, a chain structure is generated through C—H···O contacts and an intramolecular C—H···O interaction also occurs. The dihedral angle between the benzene ring and the naphthalene system is 16.5°.

## Related literature

For related literature, see: Aronne *et al.* (2008); Sasidharan *et al.* (2002); Tan *et al.* (2001); Wang *et al.* (2003); Yamazaki (2008).




## Experimental

### *Crystal data*

$C_{22}H_{15}ClO_4$
$M_r$ = 378.79
Orthorhombic, $P2_12_12_1$
$a$ = 7.7966 (13) Å
$b$ = 10.4468 (18) Å
$c$ = 21.349 (4) Å
$V$ = 1738.9 (5) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.25 mm$^{-1}$
$T$ = 193 (2) K
0.30 × 0.20 × 0.10 mm

### *Data collection*

Rigaku Mercury diffractometer
Absorption correction: multi-scan (Jacobson, 1998)
$T_{min}$ = 0.930, $T_{max}$ = 0.966
10185 measured reflections
3416 independent reflections
2809 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.029

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.136
$S$ = 1.06
3416 reflections
244 parameters
19 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.59 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.35 e Å$^{-3}$
Absolute structure: Flack (1983), 1440 Friedel pairs
Flack parameter: 0.00 (11)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C14—H14$A$···O2$^{i}$ | 0.95 | 2.55 | 3.392 (4) | 147 |
| C19—H19$A$···O4 | 0.95 | 2.54 | 3.084 (3) | 117 |

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

Data collection: *CrystalClear* (Rigaku/MSC, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2302).

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# supporting information

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## 13c-(2-Chloroethoxy)-1,13c-dihydro-2,3-epoxydibenzo[*a*,*kl*]xanthan-1-one

**Jin-Xiang Chen, Yu-Qin Wang, Shu-Guang Wu, Zhi-Hong Jiang and Zhi-Peng Chen**

### S1. Comment

Epoxides are well known as one of the most valuable building blocks used as intermediates and precursors for pharmaceuticals (Yamazaki, 2008; Aronne, *et al.*, 2008). The title compound, (I), is a key intermediate in the preparation of chiral alcohols, which we are designing for potential use as antiviral agents. The structure of (I), Fig. 1, provides information on the potential stereoselectivity of its ring-opening reactions (Sasidharan *et al.*, 2002; Wang *et al.*, 2003). The molecule of (I) contains six fused rings with the three aromatic rings almost coplanar. The six-membered carbocyclic ring adopts a slightly twisted boat conformation and the pyran ring is nearly planar. The epoxy group points in the same direction as the $OCH_2CH_2Cl$ group, having a *syn* relationship. In the crystal structure, molecules of (I) associate in a head-to-tail manner, parallel to the *b* axis, *via* O-H···O hydrogen bonds to form a 1D structure, Fig. 2 and Table 1.

### S2. Experimental

Compound (I) was obtained by epoxidation of 13*c*-(2-chloroethyloxy)-1-oxo-1,13*c*-dihydrodibenzo[a,kl]xanthene in methanol with aqueous hydrogen peroxide (30%) under mild reaction conditions (Tan *et al.*, 2001). Compound (I) was the main product, isolated in a yield of 92%. Crystals suitable for X-ray analysis were obtained from the slow evaporation of an acetone solution (m.p. 527–529 K). IR (KBr disk): 3409, 2905, 2359, 1720 (s, C=O), 1454, 1267, 1097, 776, 755, 508 $cm^{-1}$. $^1H$ NMR (300 MHz in $CDCl_3$/TMS): 3.15–3.25 (m, 2H), 3.27–3.38 (m, 2H), 4.04 (d, J = 3.9 Hz, 1H), 4.34 (d, J = 3.9 Hz, 1H), 7.24–7.26 (m, 1H), 7.27 (d, J = 9.0 Hz, 1H), 7.34–7.49 (m, 4H), 7.78–7.79 (m, 1H), 7.86 (d, J = 9.0 Hz, 1H), 7.96–8.02 (m, 1H) p.p.m. FAB-MS (m/*z*): 299($M^+$ –$OCH_2CH_2Cl$).

### S3. Refinement

The hydrogen atoms were placed in geometrically idealized positions with C—H = 0.95 - 1.00 Å and constrained to ride on their parent atoms with $U_{iso}$(H) = 1.2$U_{eq}$(C).

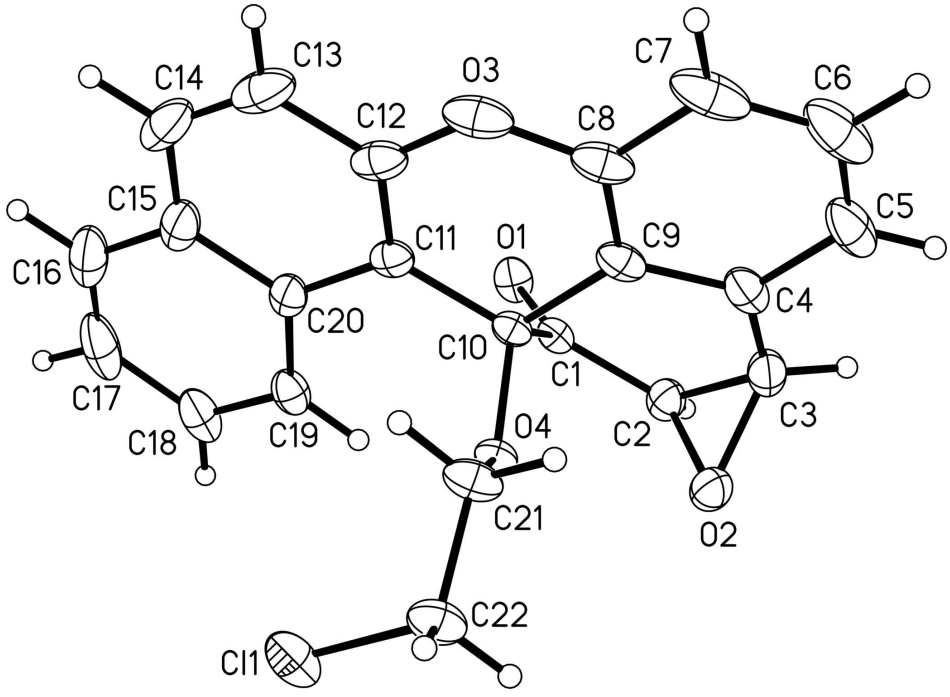


**Figure 1**

Molecular structure of (I) showing atom labelling and 30% probability ellipsoids. Hydrogen atoms are drawn as spheres of arbitrary radii.

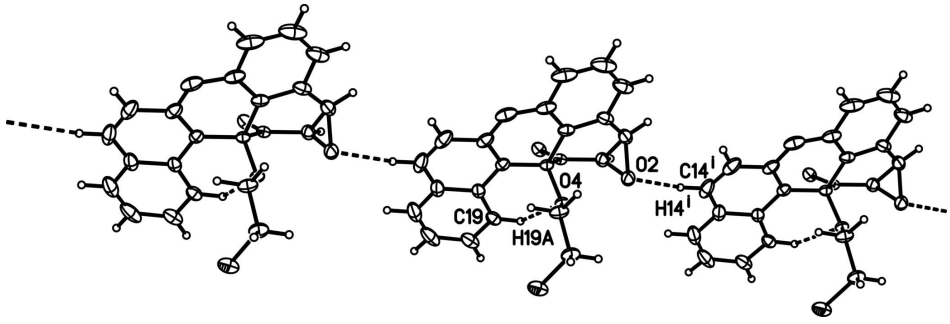


**Figure 2**

One-dimensional chain aligned along the *b* axis in (I) consolidated by C-H···O contacts, shown as dashed lines.

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*Crystal data*

$C_{22}H_{15}ClO_4$
$M_r$ = 378.79
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a$ = 7.7966 (13) Å
$b$ = 10.4468 (18) Å
$c$ = 21.349 (4) Å
$V$ = 1738.9 (5) Å$^3$
$Z$ = 4
$F(000)$ = 784
$D_x$ = 1.447 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 3462 reflections
$\theta$ = 3.0–26.0°
$\mu$ = 0.25 mm$^{-1}$
$T$ = 193 K
Block, white
0.30 × 0.20 × 0.10 mm

*Data collection*

Rigaku Mercury diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan Jacobson (1998)
$T_{min}$ = 0.930, $T_{max}$ = 0.966
10185 measured reflections
3416 independent reflections
2809 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.029
$\theta_{max}$ = 26.0°, $\theta_{min}$ = 1.9°
$h$ = −9→9
$k$ = −12→12
$l$ = −19→26

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.136
$S$ = 1.06
3416 reflections
244 parameters
19 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
$w = 1/[\sigma^2(F_o^2) + (0.074P)^2 + 0.4256P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.59 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.35 e Å$^{-3}$
Absolute structure: (Flack, 1983)
Absolute structure parameter: 0.00 (11)

*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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.46182 (15) | 0.38912 (11) | 0.01032 (4) | 0.0875 (3) |
| O1 | −0.1176 (2) | 0.3230 (2) | −0.16123 (9) | 0.0532 (5) |
| O2 | 0.2117 (3) | 0.0809 (2) | −0.15674 (11) | 0.0644 (6) |
| O3 | 0.3149 (3) | 0.5535 (3) | −0.24976 (11) | 0.0751 (7) |
| O4 | 0.2976 (2) | 0.31516 (16) | −0.11496 (8) | 0.0407 (4) |
| C1 | 0.0243 (3) | 0.2781 (2) | −0.16079 (11) | 0.0405 (5) |
| C2 | 0.0484 (4) | 0.1375 (3) | −0.16874 (14) | 0.0539 (7) |
| H2A | −0.0548 | 0.0821 | −0.1626 | 0.065* |
| C3 | 0.1656 (4) | 0.1034 (3) | −0.22079 (15) | 0.0639 (9) |
| H3A | 0.1337 | 0.0264 | −0.2460 | 0.077* |
| C4 | 0.2524 (4) | 0.2067 (4) | −0.25451 (14) | 0.0615 (8) |
| C5 | 0.3171 (5) | 0.1828 (5) | −0.31363 (17) | 0.0876 (14) |
| H5A | 0.3128 | 0.0983 | −0.3300 | 0.105* |
| C6 | 0.3871 (5) | 0.2784 (7) | −0.34885 (19) | 0.1044 (19) |
| H6A | 0.4346 | 0.2595 | −0.3888 | 0.125* |

| | | | | |
|---|---|---|---|---|
| C7 | 0.3890 (5) | 0.4024 (6) | −0.32672 (18) | 0.0925 (15) |
| H7A | 0.4359 | 0.4696 | −0.3513 | 0.111* |
| C8 | 0.3208 (4) | 0.4275 (4) | −0.26739 (15) | 0.0637 (8) |
| C9 | 0.2571 (3) | 0.3325 (3) | −0.22965 (13) | 0.0503 (7) |
| C10 | 0.1881 (3) | 0.3613 (2) | −0.16413 (11) | 0.0398 (5) |
| C11 | 0.1543 (3) | 0.5025 (3) | −0.15546 (13) | 0.0467 (6) |
| C12 | 0.2211 (4) | 0.5882 (3) | −0.19788 (16) | 0.0621 (9) |
| C13 | 0.1977 (6) | 0.7205 (4) | −0.1912 (2) | 0.0874 (14) |
| H13A | 0.2446 | 0.7775 | −0.2213 | 0.105* |
| C14 | 0.1084 (6) | 0.7670 (4) | −0.1419 (3) | 0.0954 (17) |
| H14A | 0.0913 | 0.8567 | −0.1382 | 0.114* |
| C15 | 0.0399 (5) | 0.6845 (3) | −0.09575 (19) | 0.0747 (11) |
| C16 | −0.0529 (6) | 0.7314 (5) | −0.0437 (3) | 0.0975 (16) |
| H16A | −0.0749 | 0.8206 | −0.0407 | 0.117* |
| C17 | −0.1110 (5) | 0.6531 (6) | 0.0017 (3) | 0.1038 (18) |
| H17A | −0.1753 | 0.6875 | 0.0355 | 0.125* |
| C18 | −0.0773 (4) | 0.5204 (4) | −0.00048 (18) | 0.0763 (11) |
| H18A | −0.1155 | 0.4656 | 0.0322 | 0.092* |
| C19 | 0.0119 (4) | 0.4717 (3) | −0.05086 (14) | 0.0554 (7) |
| H19A | 0.0361 | 0.3827 | −0.0523 | 0.067* |
| C20 | 0.0683 (4) | 0.5505 (3) | −0.10016 (15) | 0.0512 (7) |
| C21 | 0.4673 (3) | 0.3628 (3) | −0.11653 (14) | 0.0581 (8) |
| H21A | 0.4656 | 0.4573 | −0.1196 | 0.070* |
| H21B | 0.5282 | 0.3285 | −0.1536 | 0.070* |
| C22 | 0.5569 (4) | 0.3234 (4) | −0.05865 (15) | 0.0674 (9) |
| H22A | 0.6781 | 0.3511 | −0.0610 | 0.081* |
| H22B | 0.5555 | 0.2288 | −0.0557 | 0.081* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0914 (7) | 0.1130 (8) | 0.0581 (5) | −0.0041 (6) | −0.0129 (5) | −0.0133 (5) |
| O1 | 0.0330 (9) | 0.0667 (12) | 0.0600 (12) | −0.0019 (9) | −0.0022 (8) | −0.0140 (10) |
| O2 | 0.0653 (14) | 0.0560 (11) | 0.0719 (14) | 0.0091 (10) | −0.0164 (11) | −0.0079 (10) |
| O3 | 0.0592 (12) | 0.1059 (17) | 0.0602 (13) | −0.0229 (13) | −0.0064 (11) | 0.0374 (12) |
| O4 | 0.0337 (8) | 0.0517 (9) | 0.0366 (9) | −0.0029 (7) | −0.0054 (7) | 0.0054 (8) |
| C1 | 0.0370 (13) | 0.0516 (13) | 0.0329 (12) | −0.0024 (11) | −0.0016 (10) | −0.0019 (10) |
| C2 | 0.0519 (15) | 0.0492 (15) | 0.0606 (17) | −0.0034 (13) | −0.0072 (14) | −0.0061 (13) |
| C3 | 0.0591 (19) | 0.0673 (19) | 0.065 (2) | 0.0146 (16) | −0.0136 (15) | −0.0207 (17) |
| C4 | 0.0418 (14) | 0.098 (2) | 0.0451 (15) | 0.0212 (16) | −0.0067 (12) | −0.0141 (16) |
| C5 | 0.057 (2) | 0.154 (4) | 0.052 (2) | 0.038 (2) | −0.0018 (17) | −0.028 (2) |
| C6 | 0.056 (2) | 0.207 (6) | 0.050 (2) | 0.024 (3) | 0.0092 (17) | −0.007 (3) |
| C7 | 0.0469 (19) | 0.177 (5) | 0.053 (2) | −0.004 (2) | 0.0046 (15) | 0.037 (3) |
| C8 | 0.0410 (14) | 0.101 (2) | 0.0493 (15) | −0.0078 (16) | −0.0034 (13) | 0.0233 (16) |
| C9 | 0.0310 (12) | 0.0801 (19) | 0.0399 (13) | 0.0032 (13) | −0.0049 (10) | 0.0050 (14) |
| C10 | 0.0349 (12) | 0.0495 (13) | 0.0349 (12) | −0.0024 (10) | −0.0053 (10) | 0.0018 (10) |
| C11 | 0.0391 (13) | 0.0501 (14) | 0.0509 (15) | −0.0063 (11) | −0.0139 (11) | 0.0050 (12) |
| C12 | 0.0526 (18) | 0.0618 (18) | 0.072 (2) | −0.0124 (15) | −0.0249 (16) | 0.0204 (16) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C13 | 0.088 (3) | 0.057 (2) | 0.117 (3) | −0.023 (2) | −0.052 (3) | 0.032 (2) |
| C14 | 0.099 (3) | 0.0414 (17) | 0.146 (4) | −0.004 (2) | −0.069 (3) | 0.004 (2) |
| C15 | 0.067 (2) | 0.0560 (18) | 0.101 (3) | 0.0121 (17) | −0.045 (2) | −0.0239 (19) |
| C16 | 0.082 (3) | 0.080 (3) | 0.131 (4) | 0.031 (2) | −0.044 (3) | −0.053 (3) |
| C17 | 0.063 (2) | 0.142 (4) | 0.107 (4) | 0.032 (3) | −0.021 (2) | −0.079 (3) |
| C18 | 0.0557 (18) | 0.109 (3) | 0.064 (2) | 0.0057 (19) | −0.0051 (15) | −0.034 (2) |
| C19 | 0.0439 (14) | 0.0697 (18) | 0.0527 (17) | −0.0014 (13) | −0.0030 (12) | −0.0148 (14) |
| C20 | 0.0399 (14) | 0.0498 (14) | 0.0638 (18) | 0.0016 (12) | −0.0183 (13) | −0.0121 (13) |
| C21 | 0.0337 (13) | 0.092 (2) | 0.0481 (15) | −0.0082 (14) | −0.0058 (12) | 0.0153 (15) |
| C22 | 0.0472 (16) | 0.094 (2) | 0.0613 (19) | 0.0008 (17) | −0.0121 (15) | 0.0086 (17) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C22 | 1.786 (4) | C10—C11 | 1.509 (4) |
| O1—C1 | 1.201 (3) | C11—C12 | 1.376 (4) |
| O2—C2 | 1.427 (4) | C11—C20 | 1.448 (4) |
| O2—C3 | 1.433 (4) | C12—C13 | 1.401 (5) |
| O3—C8 | 1.370 (5) | C13—C14 | 1.353 (7) |
| O3—C12 | 1.376 (4) | C13—H13A | 0.9500 |
| O4—C21 | 1.414 (3) | C14—C15 | 1.413 (6) |
| O4—C10 | 1.437 (3) | C14—H14A | 0.9500 |
| C1—C2 | 1.490 (4) | C15—C16 | 1.413 (7) |
| C1—C10 | 1.546 (3) | C15—C20 | 1.421 (4) |
| C2—C3 | 1.482 (4) | C16—C17 | 1.347 (7) |
| C2—H2A | 1.0000 | C16—H16A | 0.9500 |
| C3—C4 | 1.463 (5) | C17—C18 | 1.411 (7) |
| C3—H3A | 1.0000 | C17—H17A | 0.9500 |
| C4—C5 | 1.382 (5) | C18—C19 | 1.378 (5) |
| C4—C9 | 1.418 (5) | C18—H18A | 0.9500 |
| C5—C6 | 1.364 (7) | C19—C20 | 1.406 (5) |
| C5—H5A | 0.9500 | C19—H19A | 0.9500 |
| C6—C7 | 1.379 (8) | C21—C22 | 1.478 (4) |
| C6—H6A | 0.9500 | C21—H21A | 0.9900 |
| C7—C8 | 1.399 (5) | C21—H21B | 0.9900 |
| C7—H7A | 0.9500 | C22—H22A | 0.9900 |
| C8—C9 | 1.371 (4) | C22—H22B | 0.9900 |
| C9—C10 | 1.529 (4) | | |
| | | | |
| C2—O2—C3 | 62.4 (2) | C12—C11—C20 | 119.1 (3) |
| C8—O3—C12 | 119.5 (2) | C12—C11—C10 | 119.3 (3) |
| C21—O4—C10 | 114.88 (19) | C20—C11—C10 | 121.3 (2) |
| O1—C1—C2 | 120.0 (3) | C11—C12—O3 | 124.0 (3) |
| O1—C1—C10 | 122.7 (2) | C11—C12—C13 | 121.7 (4) |
| C2—C1—C10 | 116.4 (2) | O3—C12—C13 | 114.3 (3) |
| O2—C2—C3 | 59.0 (2) | C14—C13—C12 | 120.1 (4) |
| O2—C2—C1 | 120.0 (2) | C14—C13—H13A | 120.0 |
| C3—C2—C1 | 113.6 (3) | C12—C13—H13A | 120.0 |
| O2—C2—H2A | 117.0 | C13—C14—C15 | 121.1 (3) |

| | | | |
|---|---|---|---|
| C3—C2—H2A | 117.0 | C13—C14—H14A | 119.4 |
| C1—C2—H2A | 117.0 | C15—C14—H14A | 119.4 |
| O2—C3—C4 | 118.3 (3) | C14—C15—C16 | 122.0 (4) |
| O2—C3—C2 | 58.6 (2) | C14—C15—C20 | 119.8 (4) |
| C4—C3—C2 | 118.5 (3) | C16—C15—C20 | 118.2 (4) |
| O2—C3—H3A | 116.4 | C17—C16—C15 | 121.8 (4) |
| C4—C3—H3A | 116.4 | C17—C16—H16A | 119.1 |
| C2—C3—H3A | 116.4 | C15—C16—H16A | 119.1 |
| C5—C4—C9 | 120.0 (4) | C16—C17—C18 | 120.7 (4) |
| C5—C4—C3 | 119.0 (4) | C16—C17—H17A | 119.7 |
| C9—C4—C3 | 120.7 (3) | C18—C17—H17A | 119.7 |
| C6—C5—C4 | 121.1 (5) | C19—C18—C17 | 118.8 (4) |
| C6—C5—H5A | 119.4 | C19—C18—H18A | 120.6 |
| C4—C5—H5A | 119.4 | C17—C18—H18A | 120.6 |
| C5—C6—C7 | 120.2 (4) | C18—C19—C20 | 121.8 (3) |
| C5—C6—H6A | 119.9 | C18—C19—H19A | 119.1 |
| C7—C6—H6A | 119.9 | C20—C19—H19A | 119.1 |
| C6—C7—C8 | 118.8 (4) | C19—C20—C15 | 118.6 (3) |
| C6—C7—H7A | 120.6 | C19—C20—C11 | 123.6 (2) |
| C8—C7—H7A | 120.6 | C15—C20—C11 | 117.9 (3) |
| O3—C8—C9 | 121.5 (3) | O4—C21—C22 | 108.9 (2) |
| O3—C8—C7 | 116.2 (4) | O4—C21—H21A | 109.9 |
| C9—C8—C7 | 122.3 (4) | C22—C21—H21A | 109.9 |
| C8—C9—C4 | 117.4 (3) | O4—C21—H21B | 109.9 |
| C8—C9—C10 | 121.5 (3) | C22—C21—H21B | 109.9 |
| C4—C9—C10 | 121.1 (3) | H21A—C21—H21B | 108.3 |
| O4—C10—C11 | 110.00 (19) | C21—C22—Cl1 | 112.7 (2) |
| O4—C10—C9 | 113.2 (2) | C21—C22—H22A | 109.0 |
| C11—C10—C9 | 111.5 (2) | Cl1—C22—H22A | 109.0 |
| O4—C10—C1 | 105.56 (18) | C21—C22—H22B | 109.0 |
| C11—C10—C1 | 113.6 (2) | Cl1—C22—H22B | 109.0 |
| C9—C10—C1 | 102.83 (19) | H22A—C22—H22B | 107.8 |

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| C3—O2—C2—C1 | −101.1 (3) | C2—C1—C10—O4 | −59.2 (3) |
| O1—C1—C2—O2 | −168.3 (3) | O1—C1—C10—C11 | 11.1 (3) |
| C10—C1—C2—O2 | 22.2 (4) | C2—C1—C10—C11 | −179.8 (2) |
| O1—C1—C2—C3 | 125.0 (3) | O1—C1—C10—C9 | −109.5 (3) |
| C10—C1—C2—C3 | −44.4 (3) | C2—C1—C10—C9 | 59.6 (3) |
| C2—O2—C3—C4 | 107.8 (3) | O4—C10—C11—C12 | 112.0 (3) |
| C1—C2—C3—O2 | 112.0 (3) | C9—C10—C11—C12 | −14.3 (3) |
| O2—C2—C3—C4 | −107.5 (3) | C1—C10—C11—C12 | −129.9 (3) |
| C1—C2—C3—C4 | 4.5 (4) | O4—C10—C11—C20 | −61.6 (3) |
| O2—C3—C4—C5 | 132.5 (3) | C9—C10—C11—C20 | 172.0 (2) |
| C2—C3—C4—C5 | −159.9 (3) | C1—C10—C11—C20 | 56.4 (3) |
| O2—C3—C4—C9 | −53.1 (4) | C20—C11—C12—O3 | 175.6 (2) |
| C2—C3—C4—C9 | 14.5 (4) | C10—C11—C12—O3 | 1.8 (4) |
| C9—C4—C5—C6 | 0.4 (5) | C20—C11—C12—C13 | −4.5 (4) |
| C3—C4—C5—C6 | 174.8 (3) | C10—C11—C12—C13 | −178.2 (3) |

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| C4—C5—C6—C7 | −2.2 (6) | C8—O3—C12—C11 | 11.4 (4) |
| C5—C6—C7—C8 | 1.0 (6) | C8—O3—C12—C13 | −168.5 (3) |
| C12—O3—C8—C9 | −9.9 (4) | C11—C12—C13—C14 | 0.3 (5) |
| C12—O3—C8—C7 | 167.8 (3) | O3—C12—C13—C14 | −179.7 (3) |
| C6—C7—C8—O3 | −175.5 (3) | C12—C13—C14—C15 | 1.2 (6) |
| C6—C7—C8—C9 | 2.2 (5) | C13—C14—C15—C16 | 179.6 (3) |
| O3—C8—C9—C4 | 173.6 (3) | C13—C14—C15—C20 | 1.5 (5) |
| C7—C8—C9—C4 | −3.9 (4) | C14—C15—C16—C17 | −176.8 (4) |
| O3—C8—C9—C10 | −4.4 (4) | C20—C15—C16—C17 | 1.4 (6) |
| C7—C8—C9—C10 | 178.1 (3) | C15—C16—C17—C18 | 1.5 (6) |
| C5—C4—C9—C8 | 2.7 (4) | C16—C17—C18—C19 | −1.8 (6) |
| C3—C4—C9—C8 | −171.7 (3) | C17—C18—C19—C20 | −0.7 (5) |
| C5—C4—C9—C10 | −179.4 (3) | C18—C19—C20—C15 | 3.5 (4) |
| C3—C4—C9—C10 | 6.3 (4) | C18—C19—C20—C11 | −176.6 (3) |
| C21—O4—C10—C11 | −67.8 (3) | C14—C15—C20—C19 | 174.5 (3) |
| C21—O4—C10—C9 | 57.6 (3) | C16—C15—C20—C19 | −3.8 (4) |
| C21—O4—C10—C1 | 169.3 (2) | C14—C15—C20—C11 | −5.4 (4) |
| C8—C9—C10—O4 | −108.7 (3) | C16—C15—C20—C11 | 176.3 (3) |
| C4—C9—C10—O4 | 73.4 (3) | C12—C11—C20—C19 | −173.0 (3) |
| C8—C9—C10—C11 | 15.9 (3) | C10—C11—C20—C19 | 0.7 (4) |
| C4—C9—C10—C11 | −162.0 (2) | C12—C11—C20—C15 | 6.9 (4) |
| C8—C9—C10—C1 | 137.9 (2) | C10—C11—C20—C15 | −179.4 (2) |
| C4—C9—C10—C1 | −40.0 (3) | C10—O4—C21—C22 | 171.6 (2) |
| O1—C1—C10—O4 | 131.7 (2) | O4—C21—C22—Cl1 | −62.9 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C14—H14*A*···O2$^{i}$ | 0.95 | 2.55 | 3.392 (4) | 147 |
| C19—H19*A*···O4 | 0.95 | 2.54 | 3.084 (3) | 117 |

Symmetry code: (i) *x*, *y*+1, *z*.