

# 5,6,7-Trichloro-2-methoxy-8-hydroxyquinoline

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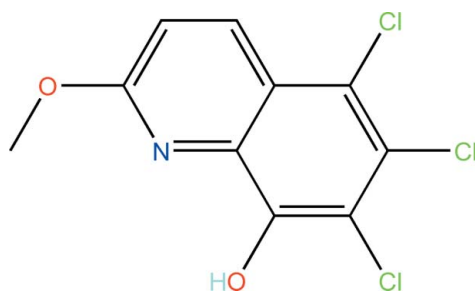
Received 22 February 2011; accepted 23 March 2011

Key indicators: single-crystal X-ray study;  $T = 150$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.039;  $wR$  factor = 0.110; data-to-parameter ratio = 13.8.

In the title compound,  $\text{C}_{10}\text{H}_6\text{Cl}_3\text{NO}_2$ , a mean plane fitted through all non-H atoms has an r.m.s. deviation of 0.035 Å. In the crystal, adjacent molecules are connected by  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds and  $\pi-\pi$  stacking interactions [centroid-centroid distance = 3.650 (1) Å], resulting in an infinite chain which propagates in the  $b$ -axis direction.

## Related literature

The title compound was obtained as an unexpected product from an attempt to synthesize a Top1 (DNA topoisomerase IB) inhibitor. For general background to Top1, see: Pommier (2006). For the synthesis, see: Shen *et al.* (2008); Cheng *et al.* (2008).



## Experimental

### Crystal data

$\text{C}_{10}\text{H}_6\text{Cl}_3\text{NO}_2$   
 $M_r = 278.51$   
Monoclinic,  $P2_1/c$   
 $a = 10.0782$  (3) Å  
 $b = 4.9979$  (1) Å  
 $c = 21.5827$  (6) Å  
 $\beta = 99.287$  (2)°  
 $V = 1072.87$  (5) Å<sup>3</sup>  
 $Z = 4$   
Cu  $K\alpha$  radiation  
 $\mu = 7.61$  mm<sup>-1</sup>  
 $T = 150$  K  
 $0.40 \times 0.21 \times 0.20$  mm

### Data collection

Oxford Diffraction Xcalibur Onyx  
Nova diffractometer  
Absorption correction: multi-scan  
(*CrysAlis PRO*; Oxford Diffraction, 2006)  
 $T_{\min} = 0.151$ ,  $T_{\max} = 0.312$   
4752 measured reflections  
2035 independent reflections  
1812 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.024$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$   
 $wR(F^2) = 0.110$   
 $S = 1.04$   
2035 reflections  
147 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.98$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.26$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                           | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------------|-------|-------------|-------------|---------------|
| $\text{O2}-\text{H2A}\cdots\text{O2}^i$ | 0.84  | 2.25        | 2.9844 (16) | 146           |

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2006); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NK2089).

## References

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Shen, D. Q., Cheng, Y., An, L. K., Bu, X. Z., Huang, Z. S. & Gu, L. Q. (2008). *Chin. Chem. Lett.* **19**, 533–536.  
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

## supporting information

*Acta Cryst.* (2011). E67, o1108 [https://doi.org/10.1107/S1600536811010853]

**5,6,7-Trichloro-2-methoxy-8-hydroxyquinoline****Qiu-Mao Chen, Guo-Bin Yi, Lin-Kun An and Xiao-Long Feng****S1. Comment**

DNA topoisomerase I (Top1) is an essential nuclear enzyme, and can be used as a target to discovery anticancer agents (Pommier, 2006). In our previous effort to find novel Top1 inhibitor, the title compound was obtained as a unexpected product from an attempt to synthesize 6,7-dichloroquinoline-5,8-dione (Cheng *et al.*, 2008 and Shen *et al.*, 2008).

The asymmetric unit of the title compound is shown in Fig. 1. All non-H atoms of the molecule adopt an approximately planar conformation (r.m.s. deviation = 0.035 Å). In the crystal, adjacent molecules are connected by O—H $\cdots$ O hydrogen bonds and  $\pi$ - $\pi$  stacking interactions [centroid-centroid distance = 3.650 (1) Å], resulting in supramolecular chains along the *b*-axis (Fig. 2).

**S2. Experimental**

According to our previously published procedure (Shen *et al.*, 2008), the oxidation of 8-Hydroxyquinoline in concentrated hydrochloric acid with sodium chlorate can give a light yellow solid. The recrystallization of the solid from methanol would give the light yellow crystal.

**S3. Refinement**

All H atoms were positioned geometrically and refined using a riding model refined using riding mode. The C—H distances of methyl and benzene ring were 0.98 Å and 0.95 Å, with  $U_{\text{iso}}(\text{H})=1.5U_{\text{eq}}(\text{C})$  and  $1.2U_{\text{eq}}(\text{C})$ . The O—H distance was 0.84 Å, with  $U_{\text{iso}}(\text{H})=1.5U_{\text{eq}}(\text{O})$ .

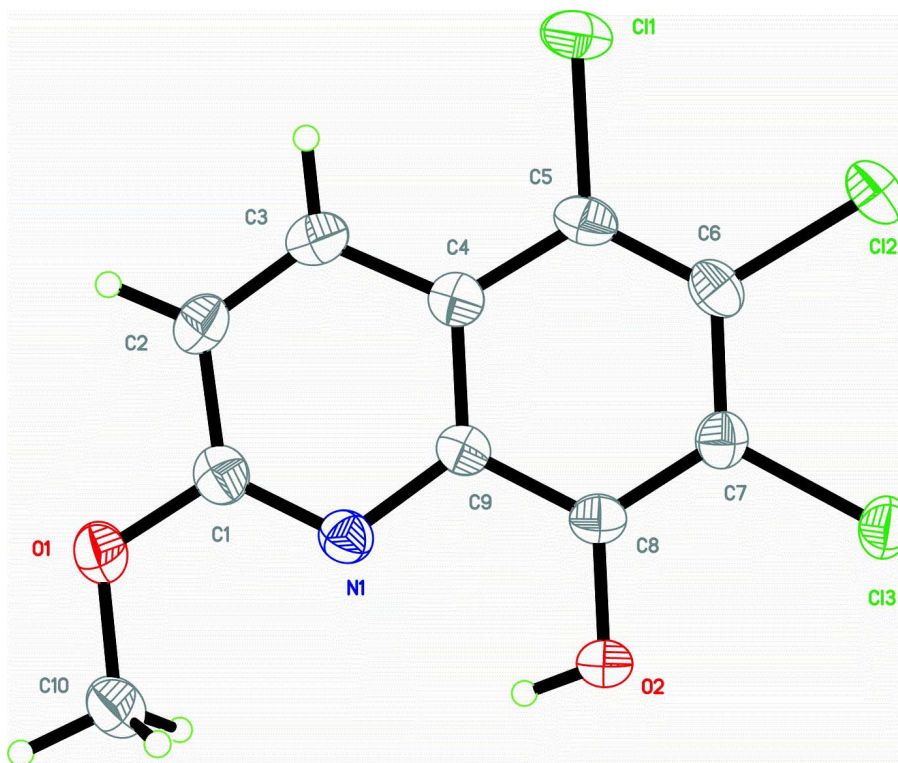


Figure 1

The structure of the title compound, with atom labels and 50% probability displacement ellipsoids for non-H atoms.

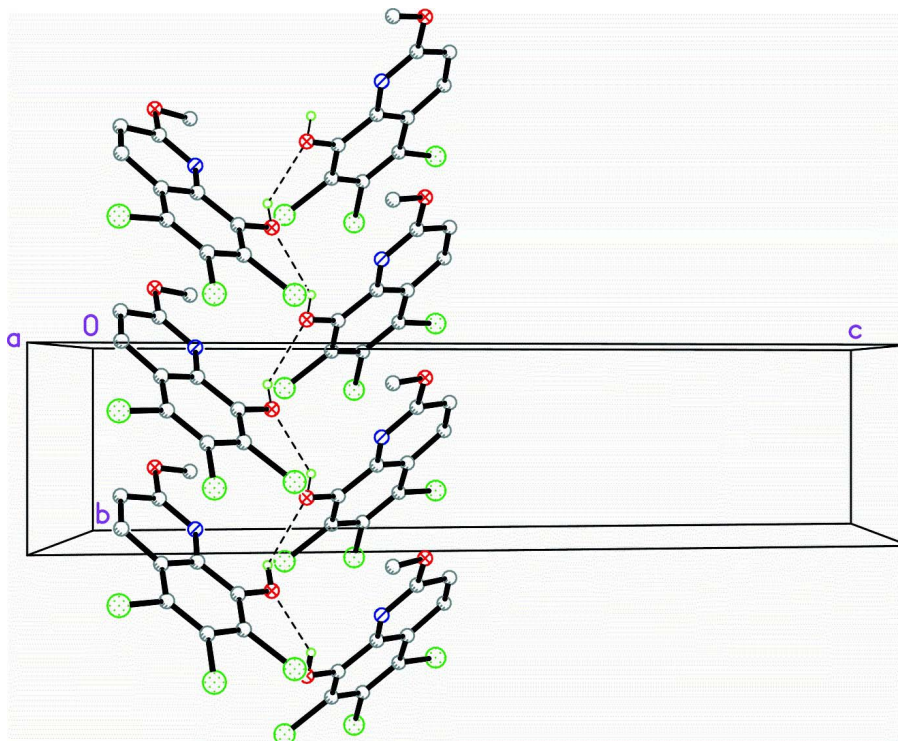


Figure 2

Infinite one-dimensional hydrogen bond in the *b*-axis direction. C-bound H atoms are omitted.

### 5,6,7-Trichloro-2-methoxy-8-hydroxyquinoline

#### Crystal data

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$M_r = 278.51$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 10.0782$  (3) Å

$b = 4.9979$  (1) Å

$c = 21.5827$  (6) Å

$\beta = 99.287$  (2)°

$V = 1072.87$  (5) Å<sup>3</sup>

$Z = 4$

$F(000) = 560$

$D_x = 1.724$  Mg m<sup>-3</sup>

Cu  $K\alpha$  radiation,  $\lambda = 1.54178$  Å

Cell parameters from 3109 reflections

$\theta = 2.1\text{--}71.2^\circ$

$\mu = 7.61$  mm<sup>-1</sup>

$T = 150$  K

Block, light yellow

$0.40 \times 0.21 \times 0.20$  mm

#### Data collection

Oxford Diffraction Xcalibur Onyx Nova diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.2417 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: multi-scan

(*CrysAlis PRO*; Oxford Diffraction, 2006)

$T_{\min} = 0.151$ ,  $T_{\max} = 0.312$

4752 measured reflections

2035 independent reflections

1812 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\max} = 71.4^\circ$ ,  $\theta_{\min} = 4.2^\circ$

$h = -11 \rightarrow 12$

$k = -6 \rightarrow 5$

$l = -17 \rightarrow 25$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.110$

$S = 1.04$

2035 reflections

147 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-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0765P)^2 + 0.4225P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.98$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.26$  e Å<sup>-3</sup>

#### 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<sup>2</sup>)

|     | <i>x</i>    | <i>y</i>     | <i>z</i>    | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|--------------|-------------|----------------------------------|
| Cl1 | 0.47126 (6) | 0.33323 (15) | 0.06402 (3) | 0.0429 (2)                       |

|      |               |              |              |              |
|------|---------------|--------------|--------------|--------------|
| Cl2  | 0.49339 (5)   | 0.73083 (11) | 0.17798 (3)  | 0.03287 (19) |
| Cl3  | 0.27565 (6)   | 0.71889 (11) | 0.26624 (3)  | 0.03218 (19) |
| C1   | 0.0208 (2)    | −0.1743 (5)  | 0.08460 (10) | 0.0290 (5)   |
| C2   | 0.1158 (2)    | −0.1986 (5)  | 0.04240 (11) | 0.0313 (5)   |
| H2   | 0.1030        | −0.3266      | 0.0094       | 0.038*       |
| C3   | 0.2240 (2)    | −0.0355 (5)  | 0.05045 (10) | 0.0307 (5)   |
| H3   | 0.2880        | −0.0468      | 0.0227       | 0.037*       |
| C4   | 0.2422 (2)    | 0.1530 (4)   | 0.10032 (10) | 0.0263 (5)   |
| C5   | 0.3521 (2)    | 0.3325 (5)   | 0.11348 (10) | 0.0286 (5)   |
| C6   | 0.3623 (2)    | 0.5061 (4)   | 0.16321 (10) | 0.0273 (5)   |
| C7   | 0.2632 (2)    | 0.5050 (4)   | 0.20283 (10) | 0.0262 (4)   |
| C8   | 0.1556 (2)    | 0.3337 (4)   | 0.19120 (10) | 0.0253 (4)   |
| C9   | 0.1429 (2)    | 0.1572 (4)   | 0.13944 (9)  | 0.0239 (4)   |
| C10  | −0.1871 (3)   | −0.3090 (7)  | 0.11264 (13) | 0.0458 (7)   |
| H10A | −0.1477       | −0.3474      | 0.1563       | 0.069*       |
| H10B | −0.2610       | −0.4339      | 0.0990       | 0.069*       |
| H10C | −0.2214       | −0.1252      | 0.1095       | 0.069*       |
| N1   | 0.03253 (18)  | −0.0055 (4)  | 0.13107 (8)  | 0.0268 (4)   |
| O1   | −0.08538 (18) | −0.3391 (4)  | 0.07291 (8)  | 0.0368 (4)   |
| O2   | 0.06144 (16)  | 0.3332 (3)   | 0.22959 (7)  | 0.0311 (4)   |
| H2A  | 0.0161        | 0.1917       | 0.2241       | 0.047*       |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| Cl1 | 0.0362 (3)  | 0.0575 (4)  | 0.0393 (4)  | −0.0108 (3)  | 0.0186 (3)  | −0.0010 (3)  |
| Cl2 | 0.0250 (3)  | 0.0306 (3)  | 0.0418 (3)  | −0.0062 (2)  | 0.0017 (2)  | 0.0043 (2)   |
| Cl3 | 0.0324 (3)  | 0.0297 (3)  | 0.0335 (3)  | −0.0017 (2)  | 0.0023 (2)  | −0.0073 (2)  |
| C1  | 0.0298 (11) | 0.0299 (12) | 0.0261 (11) | −0.0015 (9)  | 0.0009 (9)  | 0.0037 (9)   |
| C2  | 0.0351 (13) | 0.0340 (12) | 0.0242 (10) | 0.0016 (10)  | 0.0029 (9)  | −0.0032 (9)  |
| C3  | 0.0308 (11) | 0.0359 (13) | 0.0264 (10) | 0.0044 (10)  | 0.0075 (8)  | 0.0013 (9)   |
| C4  | 0.0257 (11) | 0.0272 (11) | 0.0256 (10) | 0.0014 (9)   | 0.0030 (8)  | 0.0027 (9)   |
| C5  | 0.0248 (11) | 0.0335 (12) | 0.0284 (11) | −0.0005 (9)  | 0.0068 (9)  | 0.0081 (9)   |
| C6  | 0.0247 (10) | 0.0241 (11) | 0.0319 (11) | −0.0018 (9)  | 0.0013 (8)  | 0.0057 (9)   |
| C7  | 0.0277 (10) | 0.0220 (10) | 0.0284 (10) | 0.0023 (8)   | 0.0027 (8)  | 0.0007 (8)   |
| C8  | 0.0245 (10) | 0.0262 (11) | 0.0257 (10) | 0.0024 (9)   | 0.0057 (8)  | 0.0030 (8)   |
| C9  | 0.0229 (10) | 0.0244 (11) | 0.0242 (10) | 0.0007 (8)   | 0.0035 (8)  | 0.0036 (8)   |
| C10 | 0.0373 (14) | 0.0620 (18) | 0.0399 (14) | −0.0201 (13) | 0.0110 (11) | −0.0090 (13) |
| N1  | 0.0261 (9)  | 0.0280 (10) | 0.0264 (9)  | −0.0019 (8)  | 0.0047 (7)  | 0.0016 (7)   |
| O1  | 0.0368 (9)  | 0.0393 (10) | 0.0346 (9)  | −0.0125 (8)  | 0.0067 (7)  | −0.0060 (7)  |
| O2  | 0.0283 (8)  | 0.0348 (9)  | 0.0331 (8)  | −0.0051 (7)  | 0.0132 (7)  | −0.0062 (7)  |

*Geometric parameters (Å, °)*

|        |           |       |           |
|--------|-----------|-------|-----------|
| Cl1—C5 | 1.730 (2) | C5—C6 | 1.371 (3) |
| Cl2—C6 | 1.724 (2) | C6—C7 | 1.416 (3) |
| Cl3—C7 | 1.725 (2) | C7—C8 | 1.372 (3) |
| C1—N1  | 1.302 (3) | C8—O2 | 1.357 (3) |

|               |              |               |              |
|---------------|--------------|---------------|--------------|
| C1—O1         | 1.342 (3)    | C8—C9         | 1.413 (3)    |
| C1—C2         | 1.429 (3)    | C9—N1         | 1.366 (3)    |
| C2—C3         | 1.350 (3)    | C10—O1        | 1.446 (3)    |
| C2—H2         | 0.9500       | C10—H10A      | 0.9800       |
| C3—C4         | 1.420 (3)    | C10—H10B      | 0.9800       |
| C3—H3         | 0.9500       | C10—H10C      | 0.9800       |
| C4—C9         | 1.410 (3)    | O2—H2A        | 0.8400       |
| C4—C5         | 1.418 (3)    |               |              |
| N1—C1—O1      | 120.9 (2)    | C8—C7—C6      | 120.35 (19)  |
| N1—C1—C2      | 124.0 (2)    | C8—C7—Cl3     | 119.08 (17)  |
| O1—C1—C2      | 115.1 (2)    | C6—C7—Cl3     | 120.57 (16)  |
| C3—C2—C1      | 118.5 (2)    | O2—C8—C7      | 119.9 (2)    |
| C3—C2—H2      | 120.8        | O2—C8—C9      | 119.90 (19)  |
| C1—C2—H2      | 120.8        | C7—C8—C9      | 120.15 (19)  |
| C2—C3—C4      | 120.1 (2)    | N1—C9—C4      | 123.60 (19)  |
| C2—C3—H3      | 120.0        | N1—C9—C8      | 116.37 (18)  |
| C4—C3—H3      | 120.0        | C4—C9—C8      | 120.0 (2)    |
| C9—C4—C5      | 118.5 (2)    | O1—C10—H10A   | 109.5        |
| C9—C4—C3      | 116.5 (2)    | O1—C10—H10B   | 109.5        |
| C5—C4—C3      | 125.0 (2)    | H10A—C10—H10B | 109.5        |
| C6—C5—C4      | 120.9 (2)    | O1—C10—H10C   | 109.5        |
| C6—C5—Cl1     | 120.67 (18)  | H10A—C10—H10C | 109.5        |
| C4—C5—Cl1     | 118.37 (18)  | H10B—C10—H10C | 109.5        |
| C5—C6—C7      | 120.0 (2)    | C1—N1—C9      | 117.30 (18)  |
| C5—C6—Cl2     | 121.00 (17)  | C1—O1—C10     | 116.41 (19)  |
| C7—C6—Cl2     | 119.02 (16)  | C8—O2—H2A     | 109.5        |
| N1—C1—C2—C3   | −0.7 (4)     | Cl3—C7—C8—O2  | −0.4 (3)     |
| O1—C1—C2—C3   | 178.7 (2)    | C6—C7—C8—C9   | −0.1 (3)     |
| C1—C2—C3—C4   | 0.6 (3)      | Cl3—C7—C8—C9  | 179.78 (16)  |
| C2—C3—C4—C9   | 0.1 (3)      | C5—C4—C9—N1   | 179.7 (2)    |
| C2—C3—C4—C5   | 179.5 (2)    | C3—C4—C9—N1   | −0.8 (3)     |
| C9—C4—C5—C6   | 0.2 (3)      | C5—C4—C9—C8   | −1.3 (3)     |
| C3—C4—C5—C6   | −179.3 (2)   | C3—C4—C9—C8   | 178.2 (2)    |
| C9—C4—C5—Cl1  | −178.18 (16) | O2—C8—C9—N1   | 0.5 (3)      |
| C3—C4—C5—Cl1  | 2.4 (3)      | C7—C8—C9—N1   | −179.7 (2)   |
| C4—C5—C6—C7   | 1.0 (3)      | O2—C8—C9—C4   | −178.55 (19) |
| Cl1—C5—C6—C7  | 179.29 (16)  | C7—C8—C9—C4   | 1.3 (3)      |
| C4—C5—C6—Cl2  | −178.17 (17) | O1—C1—N1—C9   | −179.3 (2)   |
| Cl1—C5—C6—Cl2 | 0.2 (3)      | C2—C1—N1—C9   | 0.0 (3)      |
| C5—C6—C7—C8   | −1.0 (3)     | C4—C9—N1—C1   | 0.8 (3)      |
| Cl2—C6—C7—C8  | 178.16 (17)  | C8—C9—N1—C1   | −178.2 (2)   |
| C5—C6—C7—Cl3  | 179.09 (17)  | N1—C1—O1—C10  | 2.7 (3)      |
| Cl2—C6—C7—Cl3 | −1.8 (2)     | C2—C1—O1—C10  | −176.6 (2)   |
| C6—C7—C8—O2   | 179.70 (19)  |               |              |

*Hydrogen-bond geometry (Å, °)*

| <i>D</i> —H $\cdots$ <i>A</i>           | <i>D</i> —H | H $\cdots$ <i>A</i> | <i>D</i> $\cdots$ <i>A</i> | <i>D</i> —H $\cdots$ <i>A</i> |
|-----------------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O2—H2 <i>A</i> $\cdots$ O2 <sup>i</sup> | 0.84        | 2.25                | 2.9844 (16)                | 146                           |

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