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## Structure Reports

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## (R)-3,4,5-Trideoxy-5,6-didehydro-1,2-O-(2,2,2-trichloroethylidene)- $\alpha$ -D-glucofuranose-6,3-carbolactone: a new derivative of $\alpha$ -chloralose

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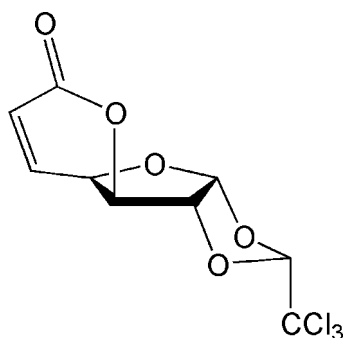
Received 29 July 2008; accepted 13 August 2008

Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å;  $R$  factor = 0.045;  $wR$  factor = 0.111; data-to-parameter ratio = 15.4.

The title compound [systematic name: (*R*)-2-trichloromethyl-3a,3b,7a,8a-tetrahydro-5*H*-pyrano[2',3':4,5]furan[2,3-*d*][1,3]-dioxol-5-one],  $\text{C}_9\text{H}_7\text{Cl}_3\text{O}_5$ , a tricyclic system that contains a central  $\alpha$ -D-furanose ring *cis*-fused with a dioxolane ring as well as a  $\delta$ -lactone ring, exhibits a twisted conformation. The  $\text{CCl}_3$  group has an axial orientation. The furanose ring approximates an envelope conformation due to the  $\alpha,\beta$ -unsaturated lactone functionality. The asymmetric unit contains two independent molecules with almost identical geometries.

### Related literature

For background regarding  $\alpha$ -chloralose and  $\delta$ -lactones, see: Collins *et al.* (1983); Zosimo-Landolfo & Tronchet (1999); Wu *et al.* (1992).



### Experimental

#### Crystal data

$\text{C}_9\text{H}_7\text{Cl}_3\text{O}_5$   
 $M_r = 301.50$   
Orthorhombic,  $P2_12_12_1$   
 $a = 9.129$  (4) Å  
 $b = 11.264$  (4) Å  
 $c = 23.156$  (7) Å

$V = 2381.1$  (15) Å<sup>3</sup>  
 $Z = 8$   
Mo  $K\alpha$  radiation  
 $\mu = 0.77$  mm<sup>-1</sup>  
 $T = 298$  (1) K  
0.60 × 0.40 × 0.10 mm

#### Data collection

Siemens P4 diffractometer  
Absorption correction:  $\psi$  scan  
(*XSCANS*; Siemens, 1996)  
 $T_{\min} = 0.803$ ,  $T_{\max} = 0.926$   
6901 measured reflections  
4735 independent reflections

3838 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.044$   
3 standard reflections  
every 97 reflections  
intensity decay: 2%

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$   
 $wR(F^2) = 0.111$   
 $S = 1.06$   
4735 reflections  
307 parameters  
H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.34$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.35$  e Å<sup>-3</sup>  
Absolute structure: Flack (1983),  
2010 Friedel pairs  
Flack parameter: 0.04 (8)

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

This work was supported by SEP-PROMEP (Mexico) through grant PROMEP/103.5/06/0959.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2095).

### References

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**supplementary materials**

*Acta Cryst.* (2008). E64, o1784 [ doi:10.1107/S1600536808026196 ]

**(*R*)-3,4,5-Trideoxy-5,6-didehydro-1,2-*O*-(2,2,2-trichloroethylidene)- $\alpha$ -D-glucofuranose-6,3-carbolactone: a new derivative of  $\alpha$ -chloralose**

**V. Aburto-Luna, R.-L. Meza-León and S. Bernès**

**Comment**

$\alpha$ -Chloralose [1,2-*O*-(2,2,2-trichloroethylidene)- $\alpha$ -D-glucofuranose, (1) in Scheme 2] is an easily available carbohydrate derivative, bearing well studied biological properties. It is a mild hypnotic drug, which is currently used as an anesthetic in veterinary medicine, as a rodenticide, *etc.* It has been characterized as a molecule possessing potent *CNS* activity, and has been evaluated in human and animal models, for its therapeutic properties (Collins *et al.*, 1983). A number of  $\alpha$ -chloralose derivatives have been prepared (*e.g.*, Zosimo-Landolfo & Tronchet, 1999), since it is known that trichloroethylidene acetals are potential biologically active compounds.

On the other hand,  $\delta$ -lactones are important flavor and aroma constituents found in many natural products. In some instances,  $\delta$ -lactone derivatives have been shown to have anti-cancer and apoptosis inducing properties against various human tumors and animal cell lines (Wu *et al.*, 1992).

We have synthesized a compound combining both functionalities, (I), with the hope that this compound will also cumulate properties corresponding to each functionality. The starting material was  $\alpha$ -chloralose, (1, scheme 2), which was first oxidized into an aldehyde, (2), and then transformed to the corresponding acrylic acid (3) *via* a Wittig reaction affording a pure *Z* isomer. Cyclization furnished the lactone (I).

The asymmetric unit of (I) contains two molecules (Figs. 1 and 2), with almost identical geometry. A fit between two independent molecules (non-H atoms) gives a r.m.s. deviation of 0.103 Å. The tricyclic system includes a central  $\alpha$ -D-furanose ring approximating an envelope conformation, with C7a as flap atom (C17a in the other molecule). This ring is *cis*-fused with a dioxolane ring, which may be considered as twisted on O3 and C8 (O13 and C18, resp.). The CCl<sub>3</sub> substituent has an axial orientation, as in  $\alpha$ -chloralose. Finally, the  $\alpha,\beta$ -unsaturated  $\delta$ -lactone ring is *cis*-fused with the furanose, and displays a rigid envelope conformation, with a total puckering amplitude of 0.347 (4) Å [0.361 (4) Å for the second molecule]. Molecules are well separated in the crystal, and no significant intermolecular contacts are detected.

**Experimental**

The synthesis of (I) is depicted in scheme 2. A solution of  $\alpha$ -chloralose (5 g, 16.23 mmol) in ethanol (60 ml) was mixed with a solution of NaIO<sub>4</sub> (3.47 g, 16.23 mmol) in H<sub>2</sub>O (6 ml). After stirring this solution at 298 K for 1 h., a white precipitate appeared, which was washed with ethanol. The filtrates were combined and concentrated under reduced pressure to give a solid product, (2). Ethyl-triphenylphosphoronylidene (6.64 g, 19.06 mmol) was added to a solution of (2), and stirred for 2 h. at 298 K. The mixture was then extracted with CH<sub>2</sub>Cl<sub>2</sub>, in order to eliminate oxide triphenylphosphine, acidified until pH 3, and extracted again with ethyl acetate. The organic phase was dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated, to give (3) as a very thick syrup. By adding *N,N*-dicyclohexylcarbodiimide (DCC) to a dry-CH<sub>2</sub>Cl<sub>2</sub> solution of (3), under Ar, the lactone (I) was formed over 2 h. The solution was filtered in order to eliminate urea, and the filtrate concentrated, to give (I) as a

## supplementary materials

white solid (3.58 g, 11.93 mmol; 73% yield). NMR and mass spectra are in agreement with the X-ray structure (see archived CIF). Single crystals were obtained by evaporation of an AcOEt solution of (I), at 298 K.

### Refinement

The absolute configuration was assigned after refining the Flack parameter (Flack, 1983), using 2010 measured Friedel pairs. All H atoms were placed in idealized positions, and refined as riding to their carrier atoms. C—H bond lengths were fixed to 0.93 (Csp<sup>2</sup>—H bonds) or 0.98 Å (methine CH groups), and isotropic displacement parameters calculated as  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier C})$ .

### Figures

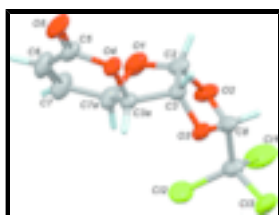


Fig. 1. Structure of the first independent molecule. Displacement ellipsoids are shown at the 30% probability level.

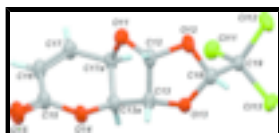


Fig. 2. Structure of the second independent molecule. Displacement ellipsoids are shown at the 30% probability level.

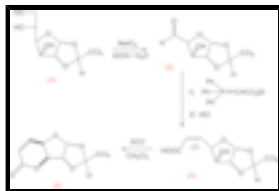


Fig. 3. The synthesis of (I)

### (R)-2-Trichloromethyl-3a,3b,7a,8a-tetrahydro-5H-pyrano[2',3':4,5]furano[2,3-d][1,3]dioxol-5-one

#### Crystal data

C<sub>9</sub>H<sub>7</sub>Cl<sub>3</sub>O<sub>5</sub>

$M_r = 301.50$

Orthorhombic,  $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 9.129(4) \text{ \AA}$

$b = 11.264(4) \text{ \AA}$

$c = 23.156(7) \text{ \AA}$

$V = 2381.1(15) \text{ \AA}^3$

$Z = 8$

$F_{000} = 1216$

$D_x = 1.682 \text{ Mg m}^{-3}$

Melting point = 416–418 K

Mo  $K\alpha$  radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 78 reflections

$\theta = 4.6\text{--}12.4^\circ$

$\mu = 0.77 \text{ mm}^{-1}$

$T = 298(1) \text{ K}$

Cell measurement pressure: 101(2) kPa

Prism, colourless

$0.60 \times 0.40 \times 0.10 \text{ mm}$

*Data collection*

Siemens P4 diffractometer	$R_{\text{int}} = 0.044$
Radiation source: fine-focus sealed tube	$\theta_{\text{max}} = 26.2^\circ$
Monochromator: graphite	$\theta_{\text{min}} = 1.8^\circ$
$T = 298(1)$ K	$h = -11 \rightarrow 11$
$\omega$ scans	$k = -14 \rightarrow 14$
Absorption correction: $\psi$ scan (XSCANS; Siemens, 1996)	$l = -28 \rightarrow 28$
$T_{\text{min}} = 0.803$ , $T_{\text{max}} = 0.926$	3 standard reflections
6901 measured reflections	every 97 reflections
4735 independent reflections	intensity decay: 2%
3838 reflections with $I > 2\sigma(I)$	

*Refinement*

Refinement on $F^2$	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.044$	$w = 1/[\sigma^2(F_o^2) + (0.0319P)^2 + 1.7306P]$
$wR(F^2) = 0.111$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{\text{max}} < 0.001$
4735 reflections	$\Delta\rho_{\text{max}} = 0.34 \text{ e } \text{\AA}^{-3}$
307 parameters	$\Delta\rho_{\text{min}} = -0.35 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none
Secondary atom site location: difference Fourier map	Absolute structure: Flack (1983), 2010 Friedel pairs
	Flack parameter: 0.04 (8)

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	1.0538 (2)	0.40323 (18)	0.81819 (5)	0.1242 (7)
C12	0.80275 (18)	0.39332 (15)	0.74470 (6)	0.1046 (5)
C13	0.87777 (16)	0.60974 (15)	0.80223 (5)	0.0921 (4)
O1	1.0741 (4)	0.3469 (3)	0.60422 (14)	0.0810 (10)
C2	1.1264 (5)	0.4432 (4)	0.63415 (15)	0.0612 (10)
H2A	1.2277	0.4611	0.6232	0.073*
O2	1.1138 (3)	0.4251 (3)	0.69476 (11)	0.0730 (9)
C3	1.0260 (4)	0.5478 (3)	0.62175 (13)	0.0510 (8)
H3A	1.0796	0.6165	0.6067	0.061*
O3	0.9574 (3)	0.5723 (2)	0.67532 (9)	0.0533 (6)
C3A	0.9171 (4)	0.5005 (3)	0.57924 (14)	0.0486 (8)
H3AA	0.8186	0.5317	0.5865	0.058*
O4	0.9700 (3)	0.5335 (2)	0.52305 (9)	0.0540 (6)
C5	0.9475 (5)	0.4644 (4)	0.47699 (16)	0.0626 (11)

## supplementary materials

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O5	0.9798 (5)	0.5044 (3)	0.43064 (11)	0.0863 (11)
C6	0.8919 (7)	0.3451 (4)	0.4862 (2)	0.0852 (16)
H6A	0.8621	0.3004	0.4546	0.102*
C7	0.8828 (7)	0.2992 (4)	0.5377 (2)	0.0914 (18)
H7A	0.8493	0.2218	0.5421	0.110*
C7A	0.9250 (6)	0.3685 (4)	0.58927 (18)	0.0684 (12)
H7AA	0.8616	0.3469	0.6218	0.082*
C8	1.0435 (5)	0.5219 (4)	0.71861 (14)	0.0561 (10)
H8A	1.1158	0.5798	0.7323	0.067*
C9	0.9472 (5)	0.4813 (4)	0.76875 (16)	0.0675 (12)
Cl11	0.48873 (14)	0.45925 (9)	0.55424 (4)	0.0678 (3)
Cl12	0.32408 (13)	0.58949 (15)	0.47031 (5)	0.0852 (4)
Cl13	0.63514 (12)	0.59974 (13)	0.46866 (4)	0.0733 (3)
O11	0.3340 (3)	0.6649 (2)	0.68090 (10)	0.0568 (7)
C12	0.3800 (5)	0.7463 (3)	0.63918 (14)	0.0527 (9)
H12A	0.3338	0.8239	0.6452	0.063*
O12	0.3488 (3)	0.7016 (3)	0.58318 (10)	0.0583 (7)
C13	0.5434 (5)	0.7557 (3)	0.64229 (13)	0.0503 (9)
H13A	0.5779	0.8380	0.6443	0.060*
O13	0.5930 (3)	0.6951 (2)	0.59195 (10)	0.0506 (6)
C13A	0.5833 (4)	0.6837 (3)	0.69550 (14)	0.0476 (8)
H13B	0.6778	0.6433	0.6912	0.057*
O14	0.5842 (3)	0.7680 (2)	0.74220 (10)	0.0552 (7)
C15	0.5407 (5)	0.7344 (3)	0.79577 (14)	0.0547 (9)
O15	0.5549 (4)	0.8058 (3)	0.83396 (11)	0.0814 (10)
C16	0.4746 (5)	0.6189 (3)	0.80334 (14)	0.0598 (10)
H16A	0.4626	0.5891	0.8405	0.072*
C17	0.4312 (5)	0.5554 (3)	0.75938 (15)	0.0542 (9)
H17A	0.3833	0.4836	0.7655	0.065*
C17A	0.4586 (4)	0.5981 (3)	0.69935 (13)	0.0460 (8)
H17B	0.4750	0.5307	0.6734	0.055*
C18	0.4790 (4)	0.7002 (3)	0.55198 (14)	0.0498 (8)
H18A	0.4872	0.7724	0.5286	0.060*
C19	0.4815 (4)	0.5908 (3)	0.51320 (13)	0.0500 (8)

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cl1	0.1478 (14)	0.1669 (16)	0.0579 (6)	0.0583 (13)	0.0186 (8)	0.0493 (9)
Cl2	0.1170 (11)	0.1059 (11)	0.0909 (9)	-0.0468 (9)	0.0423 (8)	-0.0014 (8)
Cl3	0.0981 (9)	0.1150 (11)	0.0632 (6)	0.0175 (8)	0.0090 (6)	-0.0219 (7)
O1	0.119 (3)	0.0595 (19)	0.0646 (18)	0.0256 (19)	0.0019 (19)	0.0040 (15)
C2	0.068 (2)	0.074 (3)	0.0413 (18)	0.009 (2)	0.0125 (18)	0.0106 (19)
O2	0.082 (2)	0.094 (2)	0.0429 (13)	0.0327 (18)	0.0134 (13)	0.0182 (15)
C3	0.059 (2)	0.0539 (19)	0.0401 (16)	-0.0027 (19)	0.0064 (16)	0.0059 (15)
O3	0.0669 (16)	0.0559 (14)	0.0371 (11)	0.0089 (13)	0.0027 (11)	0.0023 (10)
C3A	0.060 (2)	0.0480 (19)	0.0382 (16)	-0.0023 (17)	0.0064 (16)	0.0007 (15)
O4	0.0784 (17)	0.0454 (13)	0.0380 (11)	-0.0015 (13)	0.0041 (13)	0.0050 (10)

C5	0.089 (3)	0.055 (2)	0.0439 (19)	0.008 (2)	0.010 (2)	-0.0023 (17)
O5	0.141 (3)	0.0767 (19)	0.0410 (13)	0.003 (2)	0.0180 (18)	0.0018 (13)
C6	0.137 (5)	0.059 (3)	0.060 (3)	-0.015 (3)	0.002 (3)	-0.015 (2)
C7	0.153 (5)	0.054 (3)	0.067 (3)	-0.021 (3)	0.008 (3)	-0.004 (2)
C7A	0.106 (4)	0.051 (2)	0.048 (2)	-0.016 (2)	0.009 (2)	0.0023 (18)
C8	0.056 (2)	0.071 (3)	0.0408 (17)	0.005 (2)	0.0053 (17)	0.0085 (17)
C9	0.075 (3)	0.082 (3)	0.0449 (19)	0.009 (2)	0.0119 (19)	0.0139 (19)
Cl11	0.0934 (8)	0.0501 (5)	0.0599 (5)	-0.0081 (5)	0.0111 (6)	-0.0027 (4)
Cl12	0.0661 (6)	0.1375 (12)	0.0518 (5)	0.0059 (7)	-0.0119 (5)	-0.0211 (7)
Cl13	0.0659 (6)	0.1098 (9)	0.0443 (5)	-0.0028 (6)	0.0161 (4)	0.0017 (6)
O11	0.0608 (16)	0.0652 (17)	0.0443 (13)	0.0013 (13)	-0.0010 (12)	0.0127 (12)
C12	0.077 (3)	0.047 (2)	0.0342 (16)	0.0121 (19)	-0.0010 (17)	-0.0003 (15)
O12	0.0589 (16)	0.080 (2)	0.0364 (12)	0.0177 (14)	-0.0038 (11)	-0.0060 (12)
C13	0.075 (3)	0.0403 (17)	0.0355 (15)	-0.0042 (18)	0.0029 (16)	0.0013 (14)
O13	0.0557 (14)	0.0606 (16)	0.0356 (11)	-0.0098 (12)	0.0012 (10)	-0.0033 (11)
C13A	0.063 (2)	0.0419 (18)	0.0377 (16)	0.0000 (16)	-0.0030 (15)	-0.0069 (15)
O14	0.0838 (18)	0.0433 (13)	0.0386 (11)	-0.0123 (13)	-0.0030 (12)	-0.0040 (10)
C15	0.080 (3)	0.0457 (18)	0.0378 (16)	-0.0019 (19)	-0.0027 (18)	-0.0038 (15)
O15	0.137 (3)	0.0614 (17)	0.0461 (14)	-0.0131 (19)	-0.0012 (17)	-0.0150 (14)
C16	0.093 (3)	0.050 (2)	0.0359 (16)	0.001 (2)	-0.0011 (19)	0.0065 (15)
C17	0.080 (3)	0.0399 (18)	0.0430 (17)	-0.0034 (18)	-0.0025 (18)	0.0076 (15)
C17A	0.065 (2)	0.0376 (16)	0.0356 (14)	0.0000 (17)	-0.0054 (15)	-0.0001 (13)
C18	0.062 (2)	0.0500 (19)	0.0376 (15)	0.0018 (17)	0.0035 (17)	0.0075 (15)
C19	0.053 (2)	0.064 (2)	0.0326 (14)	-0.0013 (19)	0.0015 (14)	-0.0001 (15)

*Geometric parameters (Å, °)*

Cl1—C9	1.741 (4)	Cl11—C19	1.761 (4)
Cl2—C9	1.741 (5)	Cl12—C19	1.747 (4)
Cl3—C9	1.760 (5)	Cl13—C19	1.744 (4)
O1—C2	1.373 (6)	O11—C12	1.397 (4)
O1—C7A	1.426 (6)	O11—C17A	1.429 (4)
C2—O2	1.423 (4)	C12—O12	1.420 (4)
C2—C3	1.520 (6)	C12—C13	1.497 (6)
C2—H2A	0.9800	C12—H12A	0.9800
O2—C8	1.381 (5)	O12—C18	1.390 (4)
C3—O3	1.417 (4)	C13—O13	1.424 (4)
C3—C3A	1.497 (5)	C13—C13A	1.520 (5)
C3—H3A	0.9800	C13—H13A	0.9800
O3—C8	1.395 (4)	O13—C18	1.394 (4)
C3A—O4	1.436 (4)	C13A—O14	1.439 (4)
C3A—C7A	1.507 (6)	C13A—C17A	1.495 (5)
C3A—H3AA	0.9800	C13A—H13B	0.9800
O4—C5	1.337 (5)	O14—C15	1.356 (4)
C5—O5	1.201 (5)	C15—O15	1.202 (4)
C5—C6	1.452 (7)	C15—C16	1.445 (5)
C6—C7	1.302 (6)	C16—C17	1.306 (5)
C6—H6A	0.9300	C16—H16A	0.9300
C7—C7A	1.478 (6)	C17—C17A	1.492 (5)

## supplementary materials

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C7—H7A	0.9300	C17—H17A	0.9300
C7A—H7AA	0.9800	C17A—H17B	0.9800
C8—C9	1.527 (5)	C18—C19	1.526 (5)
C8—H8A	0.9800	C18—H18A	0.9800
C2—O1—C7A	108.6 (4)	C12—O11—C17A	108.3 (3)
O1—C2—O2	110.9 (4)	O11—C12—O12	109.8 (3)
O1—C2—C3	107.9 (3)	O11—C12—C13	108.2 (3)
O2—C2—C3	104.4 (3)	O12—C12—C13	105.6 (3)
O1—C2—H2A	111.1	O11—C12—H12A	111.0
O2—C2—H2A	111.1	O12—C12—H12A	111.0
C3—C2—H2A	111.1	C13—C12—H12A	111.0
C8—O2—C2	108.6 (3)	C18—O12—C12	107.9 (3)
O3—C3—C3A	110.6 (3)	O13—C13—C12	104.1 (3)
O3—C3—C2	104.6 (3)	O13—C13—C13A	109.4 (3)
C3A—C3—C2	104.4 (3)	C12—C13—C13A	103.9 (3)
O3—C3—H3A	112.3	O13—C13—H13A	112.9
C3A—C3—H3A	112.3	C12—C13—H13A	112.9
C2—C3—H3A	112.3	C13A—C13—H13A	112.9
C8—O3—C3	107.5 (3)	C18—O13—C13	106.6 (3)
O4—C3A—C3	106.3 (3)	O14—C13A—C17A	112.7 (3)
O4—C3A—C7A	112.3 (3)	O14—C13A—C13	105.0 (3)
C3—C3A—C7A	102.6 (3)	C17A—C13A—C13	102.1 (3)
O4—C3A—H3AA	111.7	O14—C13A—H13B	112.1
C3—C3A—H3AA	111.7	C17A—C13A—H13B	112.1
C7A—C3A—H3AA	111.7	C13—C13A—H13B	112.1
C5—O4—C3A	121.4 (3)	C15—O14—C13A	120.1 (3)
O5—C5—O4	117.2 (4)	O15—C15—O14	117.0 (3)
O5—C5—C6	124.4 (4)	O15—C15—C16	123.9 (3)
O4—C5—C6	118.4 (3)	O14—C15—C16	119.0 (3)
C7—C6—C5	121.6 (4)	C17—C16—C15	121.7 (3)
C7—C6—H6A	119.2	C17—C16—H16A	119.1
C5—C6—H6A	119.2	C15—C16—H16A	119.1
C6—C7—C7A	120.9 (4)	C16—C17—C17A	119.9 (3)
C6—C7—H7A	119.5	C16—C17—H17A	120.0
C7A—C7—H7A	119.5	C17A—C17—H17A	120.0
O1—C7A—C7	110.8 (5)	O11—C17A—C17	108.4 (3)
O1—C7A—C3A	104.6 (4)	O11—C17A—C13A	104.4 (3)
C7—C7A—C3A	112.6 (4)	C17—C17A—C13A	113.0 (3)
O1—C7A—H7AA	109.6	O11—C17A—H17B	110.3
C7—C7A—H7AA	109.6	C17—C17A—H17B	110.3
C3A—C7A—H7AA	109.6	C13A—C17A—H17B	110.3
O2—C8—O3	107.2 (3)	O12—C18—O13	107.1 (2)
O2—C8—C9	109.6 (3)	O12—C18—C19	109.1 (3)
O3—C8—C9	110.1 (3)	O13—C18—C19	110.3 (3)
O2—C8—H8A	110.0	O12—C18—H18A	110.1
O3—C8—H8A	110.0	O13—C18—H18A	110.1
C9—C8—H8A	110.0	C19—C18—H18A	110.1
C8—C9—C11	109.3 (3)	C18—C19—C113	108.3 (3)
C8—C9—C12	111.3 (3)	C18—C19—C112	109.2 (3)

C11—C9—C12	110.3 (3)	C113—C19—C112	109.01 (16)
C8—C9—C13	107.2 (3)	C18—C19—C111	111.3 (2)
C11—C9—C13	109.1 (2)	C113—C19—C111	109.7 (2)
C12—C9—C13	109.6 (3)	C112—C19—C111	109.3 (2)
C7A—O1—C2—O2	94.8 (4)	C17A—O11—C12—O12	99.0 (3)
C7A—O1—C2—C3	-19.0 (4)	C17A—O11—C12—C13	-15.8 (4)
O1—C2—O2—C8	-128.5 (4)	O11—C12—O12—C18	-124.3 (3)
C3—C2—O2—C8	-12.5 (5)	C13—C12—O12—C18	-7.8 (4)
O1—C2—C3—O3	113.3 (3)	O11—C12—C13—O13	107.1 (3)
O2—C2—C3—O3	-4.7 (4)	O12—C12—C13—O13	-10.4 (4)
O1—C2—C3—C3A	-2.9 (4)	O11—C12—C13—C13A	-7.4 (4)
O2—C2—C3—C3A	-120.9 (3)	O12—C12—C13—C13A	-124.9 (3)
C3A—C3—O3—C8	132.0 (3)	C12—C13—O13—C18	25.0 (4)
C2—C3—O3—C8	20.1 (4)	C13A—C13—O13—C18	135.5 (3)
O3—C3—C3A—O4	151.9 (3)	O13—C13—C13A—O14	157.7 (3)
C2—C3—C3A—O4	-96.1 (3)	C12—C13—C13A—O14	-91.6 (3)
O3—C3—C3A—C7A	-90.1 (4)	O13—C13—C13A—C17A	-84.5 (3)
C2—C3—C3A—C7A	21.9 (4)	C12—C13—C13A—C17A	26.1 (3)
C3—C3A—O4—C5	147.0 (4)	C17A—C13A—O14—C15	35.4 (5)
C7A—C3A—O4—C5	35.6 (5)	C13—C13A—O14—C15	145.7 (3)
C3A—O4—C5—O5	171.5 (4)	C13A—O14—C15—O15	174.2 (4)
C3A—O4—C5—C6	-10.9 (6)	C13A—O14—C15—C16	-8.6 (6)
O5—C5—C6—C7	167.8 (6)	O15—C15—C16—C17	164.2 (5)
O4—C5—C6—C7	-9.5 (8)	O14—C15—C16—C17	-12.7 (7)
C5—C6—C7—C7A	1.8 (10)	C15—C16—C17—C17A	4.1 (7)
C2—O1—C7A—C7	154.9 (4)	C12—O11—C17A—C17	153.7 (3)
C2—O1—C7A—C3A	33.4 (4)	C12—O11—C17A—C13A	33.0 (3)
C6—C7—C7A—O1	-93.4 (7)	C16—C17—C17A—O11	-92.0 (5)
C6—C7—C7A—C3A	23.4 (8)	C16—C17—C17A—C13A	23.2 (5)
O4—C3A—C7A—O1	80.2 (4)	O14—C13A—C17A—O11	76.1 (3)
C3—C3A—C7A—O1	-33.5 (4)	C13—C13A—C17A—O11	-35.9 (3)
O4—C3A—C7A—C7	-40.1 (6)	O14—C13A—C17A—C17	-41.4 (4)
C3—C3A—C7A—C7	-153.8 (4)	C13—C13A—C17A—C17	-153.5 (3)
C2—O2—C8—O3	25.7 (4)	C12—O12—C18—O13	23.9 (4)
C2—O2—C8—C9	145.1 (4)	C12—O12—C18—C19	143.2 (3)
C3—O3—C8—O2	-28.8 (4)	C13—O13—C18—O12	-30.9 (3)
C3—O3—C8—C9	-147.9 (3)	C13—O13—C18—C19	-149.5 (3)
O2—C8—C9—C11	55.3 (4)	O12—C18—C19—C113	173.3 (2)
O3—C8—C9—C11	173.0 (3)	O13—C18—C19—C113	-69.4 (3)
O2—C8—C9—C12	-66.7 (4)	O12—C18—C19—C112	54.7 (3)
O3—C8—C9—C12	51.0 (4)	O13—C18—C19—C112	172.1 (2)
O2—C8—C9—C13	173.4 (3)	O12—C18—C19—C111	-66.0 (3)
O3—C8—C9—C13	-68.9 (4)	O13—C18—C19—C111	51.3 (4)

Fig. 1

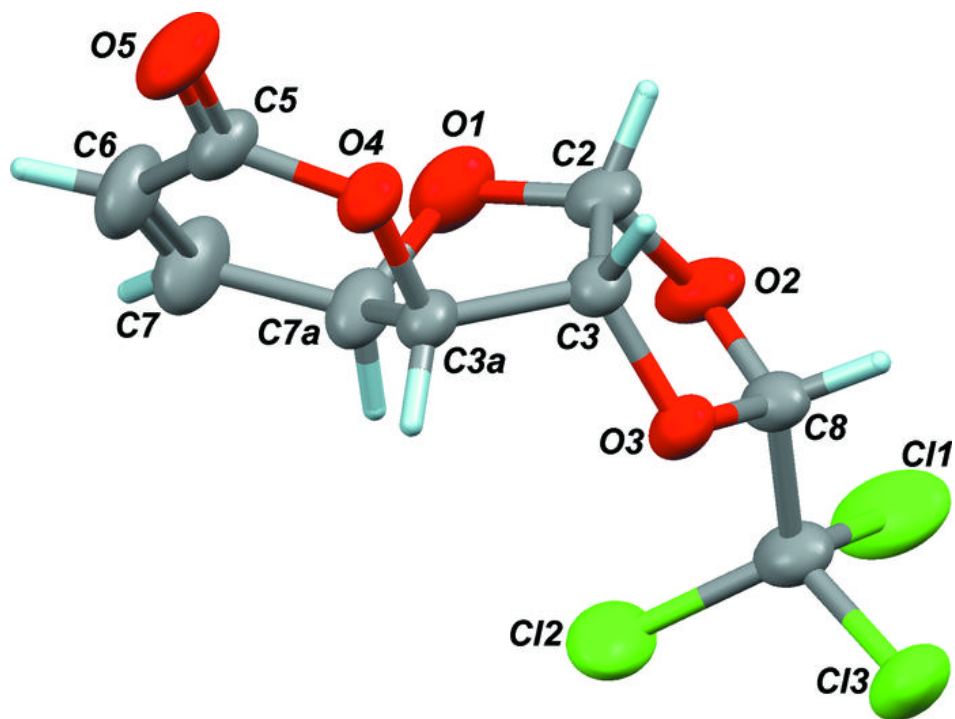


Fig. 2

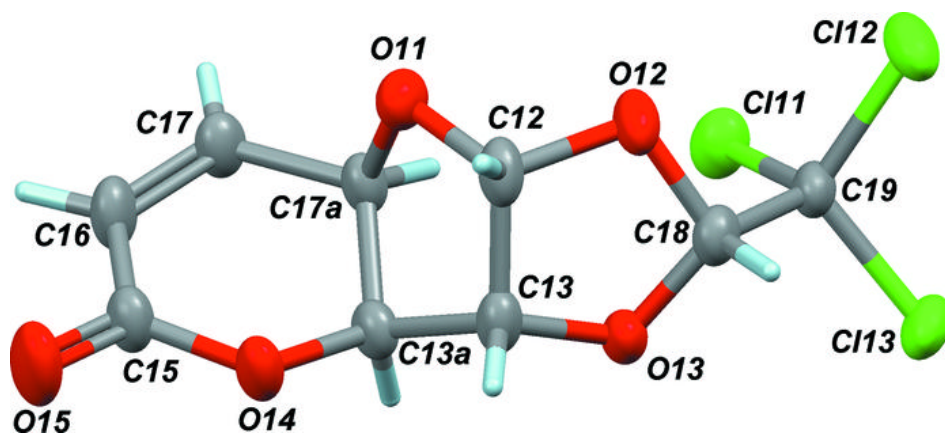


Fig. 3

