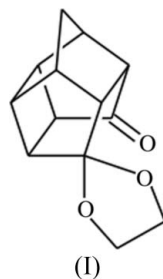


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rademeyerm@ukzn.ac.za**Key indicators**Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.062
 wR factor = 0.172
Data-to-parameter ratio = 16.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**Pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane-8,11-dione ethylene acetal**

The title molecule, $\text{C}_{13}\text{H}_{14}\text{O}_3$, is a chiral molecule which exhibits C—C single-bond lengths that deviate from the expected value. Both enantiomers are present in the crystal structure.

Comment

The structure of the title compound, (I), was determined as part of an ongoing investigation of the chemical reactivity and crystal structures of polycyclic pentacycloundecane (PCU) cage derivatives. Previous studies indicated that PCU cages and derivatives often display C—C bond lengths that deviate from the expected value of 1.54 Å (Flippen-Anderson *et al.*, 1991; Linden *et al.*, 2005; Kruger *et al.*, 2005). It is typically found that bonds involving atoms C4, C8 and C11 are shorter than normal, and that the C9—C10 bond is longer than expected.



The title compound is unsymmetrically substituted at the mouth of the cage, with a ketone substituent at atom C8 and a ketal substituent at atom C11 (Fig. 1). The asymmetric unit consists of one chiral molecule, with eight molecules (four of each enantiomer) in the unit cell (Fig. 2). This compound exhibits the shortening and elongation of certain C—C single bonds, as reported for related PCU derivatives (Table 1). The longest C—C single bond is the C9—C10 bond [1.581 (3) Å], with the C3—C10, C6—C7, C5—C9, C2—C6, C5—C6 and C1—C7 bonds also exceeding the expected value of 1.54 Å. The bonds involving the bridgehead atom C4 are shorter than expected; C3—C4 and C4—C5 exhibit values of 1.525 (3) and 1.521 (3) Å, respectively. The shortest bond is the C7—C8 bond [1.509 (3) Å], with the C10—C11, C8—C9 and C12—C13 bonds also exhibiting short C—C single-bond lengths. The unsymmetrical substitution at the mouth of the cage results in corresponding bonds in the molecule displaying different bond lengths, for example, the C1—C11 bond [1.536 (3) Å] is longer than the corresponding C7—C8 bond.

The five-membered rings, C2—C6 and C3—C5/C9/C10, are each in an envelope conformation (flap at C4), and the acetal ring, O1/C11/O2/C13/C12, also adopts an envelope confor-

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mation (flap atom C1). The rings, C1–C3/C10/C11 and C5–C9, are twisted on C10–C11 and C8–C9, respectively.

Experimental

The synthetic procedure reported by Dekker & Oliver (1979) was followed to prepare the title compound, (I). In this procedure, a mixture of pentacycloundecanedione (Cookson *et al.*, 1964) (183 g, 1.05 mol), ethylene glycol (81.2 ml, 1.45 mol) and *p*-toluenesulfonic acid (6.11 g, 3.21×10^{-2} mol) in benzene (600 ml) was refluxed (Dean–Stark trap) with stirring for 4 d. The reaction mixture was cooled to room temperature and slowly poured into ice-cold 10% (v/v) aqueous Na_2CO_3 (1000 ml), extracted with dichloromethane (3×500 ml) and the combined extracts were dried over anhydrous Na_2SO_4 . Evaporation of the solvent followed by recrystallization of the crude residue yielded the monoacetal as colourless crystals (170.04 g, 74%). IR (KBr) max 1747 cm^{-1} (C=O); ^1H NMR (CDCl_3 , 300 MHz): δ 1.51–1.84 (2H, methylene bridge protons), 2.35–2.92 (8H, methine protons), 3.78–3.91 (4H, ketal protons); ^{13}C NMR (CDCl_3 , 75 MHz): δ 36.35 (*d*), 38.74 (*t*), 41.33–53.02 (*d*), 64.52–65.71 (*t*, ketal carbons), 113.92 (*s*), 215.14 (*s*). The MS spectrum was identical to that of an authentic sample.

Crystal data

$\text{C}_{13}\text{H}_{14}\text{O}_3$	$D_x = 1.455 \text{ Mg m}^{-3}$
$M_r = 218.24$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 3784 reflections
$a = 24.7387$ (10) Å	$\theta = 3.2$ – 28.2°
$b = 6.6340$ (3) Å	$\mu = 0.10 \text{ mm}^{-1}$
$c = 12.7631$ (5) Å	$T = 173$ (2) K
$\beta = 107.914$ (2)°	Plate, colourless
$V = 1993.09$ (14) Å ³	$0.37 \times 0.22 \times 0.11 \text{ mm}$
$Z = 8$	

Data collection

Bruker SMART CCD area-detector diffractometer	1984 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.026$
Absorption correction: none	$\theta_{\text{max}} = 28.0^\circ$
10114 measured reflections	$h = -32 \rightarrow 32$
2394 independent reflections	$k = -8 \rightarrow 8$
	$l = -16 \rightarrow 11$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0769P)^2 + 5.0276P]$
$R[F^2 > 2\sigma(F^2)] = 0.062$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.172$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.58 \text{ e \AA}^{-3}$
2394 reflections	$\Delta\rho_{\text{min}} = -0.27 \text{ e \AA}^{-3}$
145 parameters	
H-atom parameters constrained	

Table 1

Selected bond lengths (Å).

C11–C10	1.510 (3)	C5–C6	1.560 (3)
C11–C1	1.536 (3)	C9–C8	1.514 (3)
C10–C3	1.556 (3)	C8–C7	1.509 (3)
C10–C9	1.581 (3)	C7–C6	1.557 (3)
C3–C4	1.525 (3)	C7–C1	1.569 (3)
C3–C2	1.537 (3)	C6–C2	1.558 (3)
C4–C5	1.521 (3)	C2–C1	1.550 (3)
C5–C9	1.557 (3)	C12–C13	1.524 (3)

All H atoms were placed in calculated positions, with methylene C–H distances of 0.99 Å and methine C–H distances of 1.00 Å, and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$.

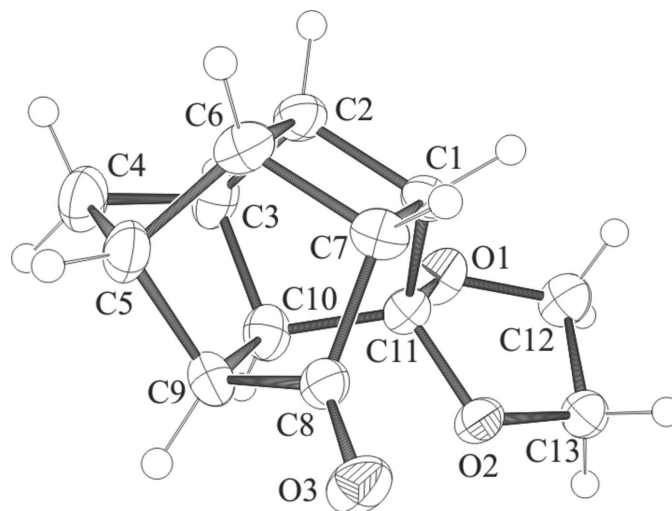


Figure 1

The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level (ORTEP3; Farrugia, 1997).

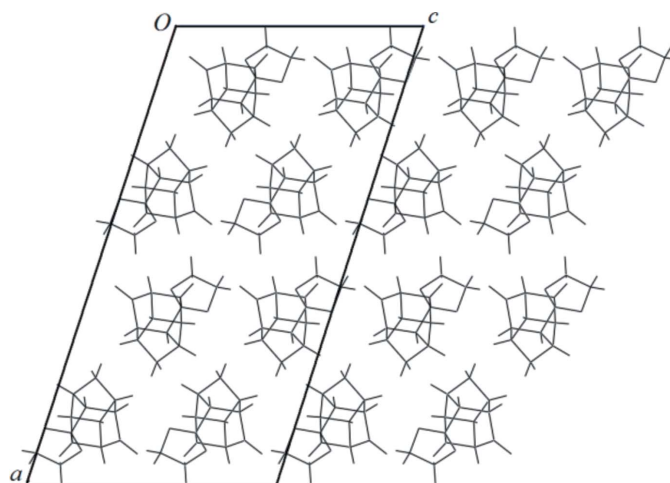


Figure 2

Packing diagram (MERCURY; Bruno *et al.*, 2002) of (I), viewed along the *b* axis.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP3 (Farrugia, 1997) and MERCURY (Bruno *et al.*, 2002); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON (Spek, 2003).

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