C3'	1.2142 (4)	0.62256 (12)	0.19913 (13)	0.0310 (4)
O3'	1.2674 (3)	0.61527 (10)	0.28009 (9)	0.0395 (4)
O3	1.0856 (5)	0.56555 (11)	0.15411 (13)	0.0641 (6)

Table 2. *Selected geometric parameters (Å, °)*

N1—C1A	1.485 (2)	C2'—N3	1.333 (2)
C1A—C1B	1.513 (3)	C2'—O2'	1.234 (3)
C1A—C1'	1.521 (2)	N3—C3A	1.455 (2)
C1'—O1'	1.229 (2)	C3A—C3B	1.516 (3)
C1'—N2	1.325 (2)	C3A—C3'	1.541 (2)
N2—C2A	1.442 (2)	C3'—O3	1.231 (2)
C2A—C2'	1.518 (2)	C3'—O3'	1.260 (2)
N1—C1A—C1B	110.3 (2)	O2'—C2'—C2A	121.5 (2)
N1—C1A—C1'	108.76 (14)	N3—C2'—C2A	115.9 (2)
C1B—C1A—C1'	113.6 (2)	C2'—N3—C3A	120.3 (2)
O1'—C1'—N2	124.3 (2)	N3—C3A—C3B	110.6 (2)
O1'—C1'—C1A	121.07 (15)	N3—C3A—C3'	113.25 (14)
N2—C1'—C1A	114.6 (2)	C3B—C3A—C3'	111.2 (2)
C1'—N2—C2A	122.0 (2)	O3—C3'—O3'	125.9 (2)
N2—C2A—C2'	111.7 (2)	O3—C3'—C3A	114.9 (2)
O2'—C2'—N3	122.6 (2)	O3'—C3'—C3A	119.2 (2)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1N1⋯O1'i	0.89	2.12	2.916 (2)	150.8
N1—H2N1⋯O3'ii	0.89	1.89	2.716 (3)	154.3
N3—HN3⋯O2'iii	0.86	2.12	2.922 (4)	154.2
N1—H3N1⋯O3'iii	0.89	2.17	3.030 (4)	161.3
N2—HN2⋯O3iv	0.86	1.97	2.758 (3)	151.7

Symmetry codes: (i) $x - \frac{1}{2}, \frac{3}{2} - y, 1 - z$; (ii) $1+x, y, z$; (iii) $\frac{1}{2}+x, \frac{3}{2}-y, 1-z$; (iv) $2 - x, \frac{1}{2} + y, \frac{1}{2} - z$.

Data collection: *SDP* (Enraf–Nonius, 1985). Cell refinement: *SDP*. Data reduction: local program. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *INSIGHTII* (Biosym Technologies, 1995); *Xtal-GX* (Hall & du Boulay, 1995). Software used to prepare material for publication: local program.

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γ-Lactones: Methyl 11-Oxa-10-oxobicyclo-[6.3.0]undec-4-ene-9-carboxylate, Methyl 11-Oxa-10-oxobicyclo[6.3.0]undec-2-ene-9-carboxylate and Methyl 9-Oxa-10-oxobi-cyclo[6.3.0]undecane-11-carboxylate

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Abstract

The structures of three γ-lactone derivatives, methyl 11-oxa-10-oxobicyclo[6.3.0]undec-4-ene-9-carboxylate, $C_{12}H_{16}O_4$, methyl 11-oxa-10-oxobicyclo[6.3.0]undec-2-ene-9-carboxylate, $C_{12}H_{16}O_4$, and methyl 9-oxa-10-oxobicyclo[6.3.0]undecane-11-carboxylate, $C_{12}H_{18}O_4$, have been solved in order to determine the relative configuration of their three asymmetric stereocentres.

Comment

γ-Lactone rings are key structural features of many perfume oils (Mori, 1989). They also exhibit important biological activities such as cytotoxic, antitumoral and bactericidal properties (Hoffmann & Rabe, 1985).

Among butyrolactone derivatives, α-methylene lactones are particularly important and can be prepared by decarboxylation–methylation of α-carbomethoxy- or α-carboxy-substituted lactones (Petragnani, Ferraz & Silva, 1986). Such substrates can be conveniently prepared by Mn^{III}-mediated addition of acetic acid derivatives on saturated systems (de Klein, 1986; Melikyan, 1993).

The potassium salt of monomethylmalonate is of particular interest as it allows the direct synthesis of γ-lactones α-substituted by a carbomethoxy group which are the precursors of α-methylene lactones (Lamarque, Méou & Brun, 1994). When applied to 1,3-cyclooctadiene, such an approach leads to different stereoisomers as three stereocentres are formed during the reaction. While the geometry of two of them can be determined easily by NMR spectroscopy [*cis* or *trans* junction between the γ-lactone ring and the cyclooctene (Lamarque, Méou & Brun, 1994)], it is very difficult to assign the relative configuration of the remaining stereocentre (i.e. the C atom bearing the

carbomethoxy group) by such a technique. The relative configuration of the three asymmetric centres C8, C1 and C11 is *trans,trans* in the three molecules. It is interesting to note that there is a greater geometric analogy between methyl 11-oxa-10-oxobicyclo[6.3.0]-undec-2-ene-9-carboxylate, (II), and methyl 9-oxa-10-oxobicyclo[6.3.0]undecane-11-carboxylate, (III), in spite of the presence of a double bond between C4 and C5 in compound (II), than exists between methyl 11-oxa-10-oxobicyclo[6.3.0]undec-4-ene-9-carboxylate, (I), and (II) which both contain a double bond in the eight-membered ring. This similarity can be characterized by the torsion angles C10—C11—C13—O15 [−72.2 (1) and −71.8 (1)° in compounds (II) and (III), respectively] and C2—C1—C8—C7 [101.0 (2) and 97.9 (1)° in compounds (II) and (III), respectively]. For compound (I), the corresponding torsion-angle values are −69.8 (2) and 81.2 (2)°, respectively.

(I)

(II)

(III)
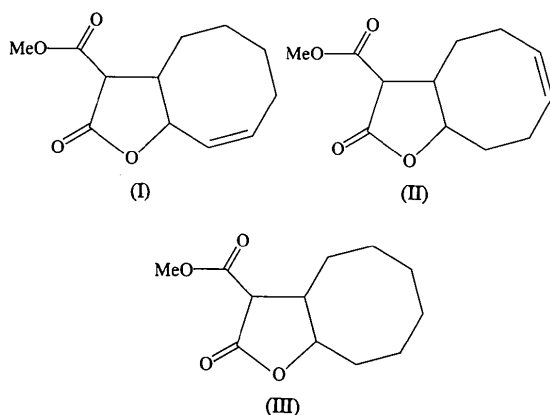
The same analogy also appears at the crystal-packing level, where the cells of compounds (II) and (III) are very similar with equivalent atomic positions. This is
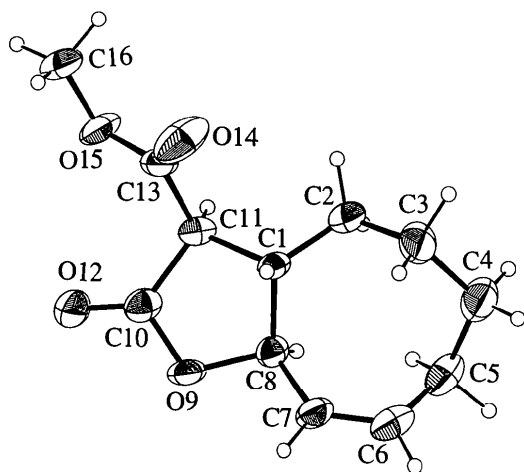
also expressed through the crystal melting points, 356 and 351 K for compounds (II) and (III), respectively, which differ significantly from the 382 K for compound (I).
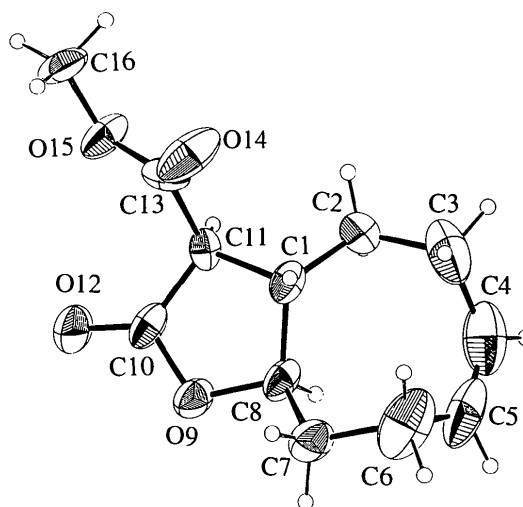

Fig. 2. Molecular structure of (II) with displacement ellipsoids at the 50% probability level.
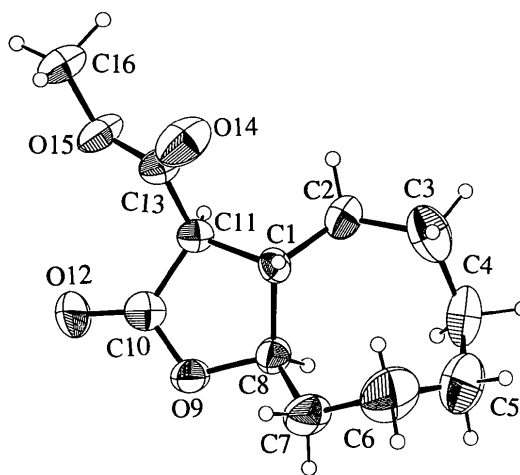
Fig. 3. Molecular structure of (III) with displacement ellipsoids at the 50% probability level.

Experimental

For all three compounds, the crystals used for X-ray analysis were grown from ethanol/hexane solutions.

Compound (I)

Crystal data

$C_{12}H_{16}O_4$	Cu $K\alpha$ radiation
$M_r = 224.3$	$\lambda = 1.5418$ Å

Fig. 1. Molecular structure of (I) with displacement ellipsoids at the 50% probability level.

Monoclinic
$P2_1/a$
$a = 6.933\,(2)$ Å
$b = 22.519\,(4)$ Å
$c = 7.408\,(2)$ Å
$\beta = 94.60\,(3)°$
$V = 1152.8\,(4)$ Å^3
$Z = 4$
$D_x = 1.29$ Mg m^{-3}
$D_m = 1.27\,(2)$ Mg m^{-3}
D_m measured by flotation

Cell parameters from 25
reflections
$\theta = 15–45°$
$\mu = 0.80$ mm^{-1}
$T = 293$ K
Square platelet
$0.4 \times 0.4 \times 0.2$ mm
Colourless

Data collection

Enraf–Nonius CAD-4
diffractometer
θ scans
Absorption correction:
none
2578 measured reflections
2333 independent reflections
1783 observed reflections
$[I > 3.0\sigma(I)]$

$R_{int} = 0.035$
$\theta_{max} = 45°$
$h = -7 \rightarrow 7$
$k = 0 \rightarrow 23$
$l = 0 \rightarrow 8$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F
$R = 0.0479$
$wR = 0.0479$
$S = 0.71$
1783 reflections
145 parameters
H atoms placed in theo-
retical positions with
bond lengths 1 Å and
$U = 0.05$Å^2

Unit weights applied
$(\Delta/\sigma)_{max} = 0.12$
$\Delta\rho_{max} = +0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: none
Atomic scattering factors
from *International Tables*
for X-ray Crystallography
(1974, Vol. IV, Table
2.3.1)

Table 1. *Fractional atomic coordinates and equivalent*
isotropic displacement parameters (Å^2) for (I)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C1	0.4595 (3)	0.3796 (1)	0.6530 (3)	0.03 (1)
C2	0.6293 (4)	0.3942 (1)	0.5465 (3)	0.04 (1)
C3	0.7220 (4)	0.4554 (1)	0.5904 (4)	0.05 (1)
C4	0.9048 (4)	0.4564 (1)	0.7217 (4)	0.05 (1)
C5	0.8898 (4)	0.4221 (1)	0.8987 (4)	0.05 (1)
C6	0.7203 (4)	0.4387 (1)	0.9995 (3)	0.05 (1)
C7	0.5483 (4)	0.4117 (1)	0.9796 (3)	0.04 (1)
C8	0.5100 (3)	0.3622 (1)	0.8517 (3)	0.03 (1)
O9	0.3359 (3)	0.3303 (1)	0.9012 (2)	0.04 (1)
C10	0.2394 (4)	0.3079 (1)	0.7525 (3)	0.04 (1)
C11	0.3372 (3)	0.3266 (1)	0.5855 (3)	0.03 (1)
O12	0.0946 (3)	0.2789 (1)	0.7630 (2)	0.06 (1)
C13	0.1946 (4)	0.3379 (1)	0.4249 (3)	0.04 (1)
O14	0.1595 (4)	0.3856 (1)	0.3627 (3)	0.07 (1)
O15	0.1120 (3)	0.2882 (1)	0.3609 (2)	0.05 (1)
C16	-0.0260 (5)	0.2941 (1)	0.2072 (3)	0.06 (2)

Table 2. *Selected geometric parameters (Å, °) for (I)*

C1—C2	1.505 (3)	C8—O9	1.476 (3)
C1—C8	1.536 (3)	O9—C10	1.341 (3)
C1—C11	1.525 (3)	C10—C11	1.517 (3)
C2—C3	1.544 (3)	C10—O12	1.205 (3)
C3—C4	1.535 (4)	C11—C13	1.506 (3)
C4—C5	1.533 (4)	C13—O14	1.186 (3)
C5—C6	1.490 (3)	C13—O15	1.327 (3)
C6—C7	1.336 (3)	O15—C16	1.433 (3)
C7—C8	1.473 (3)		

C2—C1—C8	115.5 (2)	C8—O9—C10	110.1 (2)
C8—C1—C11	101.1 (2)	O9—C10—C11	109.8 (2)
C1—C2—C3	114.5 (2)	C11—C10—O12	129.2 (2)
C2—C3—C4	117.1 (2)	O9—C10—O12	120.9 (2)
C3—C4—C5	115.2 (2)	C1—C11—C10	102.9 (2)
C4—C5—C6	114.4 (2)	C1—C11—C13	116.1 (2)
C5—C6—C7	124.3 (2)	C10—C11—C13	112.5 (2)
C6—C7—C8	121.9 (2)	C11—C13—O14	124.1 (2)
C1—C8—C7	116.0 (2)	C11—C13—O15	112.1 (2)
C1—C8—O9	103.5 (2)	O14—C13—O15	123.7 (2)
C7—C8—O9	108.5 (2)	C13—O15—C16	116.5 (2)

Compound (II)

Crystal data

$C_{12}H_{16}O_4$
$M_r = 224.3$
Monoclinic
$P2_1/a$
$a = 7.116\,(2)$ Å
$b = 23.074\,(4)$ Å
$c = 7.266\,(2)$ Å
$\beta = 100.45\,(3)°$
$V = 1173.3\,(4)$ Å^3
$Z = 4$
$D_x = 1.27$ Mg m^{-3}
$D_m = 1.28\,(2)$ Mg m^{-3}
D_m measured by flotation

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25
reflections
$\theta = 15–45°$
$\mu = 0.79$ mm^{-1}
$T = 293$ K
Square prism
$0.3 \times 0.2 \times 0.2$ mm
Colourless

Data collection

Enraf–Nonius CAD-4
diffractometer
θ scans
Absorption correction:
none
2760 measured reflections
2488 independent reflections
1756 observed reflections
$[I > 3.0\sigma(I)]$

$R_{int} = 0.028$
$\theta_{max} = 45°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 24$
$l = 0 \rightarrow 8$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F
$R = 0.0424$
$wR = 0.0424$
$S = 0.64$
1756 reflections
145 parameters
H atoms placed in theo-
retical positions with
bond lengths 1 Å and
$U = 0.05$Å^2

Unit weights applied
$(\Delta/\sigma)_{max} = 0.11$
$\Delta\rho_{max} = +0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Extinction correction: none
Atomic scattering factors
from *International Tables*
for X-ray Crystallography
(1974, Vol. IV, Table
2.3.1)

Table 3. *Fractional atomic coordinates and equivalent*
isotropic displacement parameters (Å^2) for (II)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C1	0.5645 (3)	0.3740 (1)	0.9586 (3)	0.04 (1)
C2	0.7313 (4)	0.3744 (1)	0.8564 (4)	0.06 (1)
C3	0.8612 (4)	0.4289 (2)	0.8865 (5)	0.09 (2)
C4	0.9893 (4)	0.4291 (2)	1.0760 (6)	0.09 (2)
C5	0.9370 (5)	0.4464 (1)	1.2307 (6)	0.08 (2)
C6	0.7482 (5)	0.4692 (1)	1.2421 (6)	0.09 (2)
C7	0.6038 (4)	0.4209 (1)	1.2891 (4)	0.06 (1)
C8	0.6070 (3)	0.3667 (1)	1.1720 (3)	0.04 (1)
O9	0.4509 (2)	0.3313 (1)	1.2182 (2)	0.05 (1)
C10	0.3529 (3)	0.3056 (1)	1.0663 (3)	0.04 (1)

C11	0.4318 (3)	0.3230 (1)	0.8969 (3)	0.03 (1)
O12	0.2218 (3)	0.2740 (1)	1.0780 (3)	0.06 (1)
C13	0.2766 (3)	0.3336 (1)	0.7261 (3)	0.04 (1)
O14	0.2322 (3)	0.3817 (1)	0.6690 (3)	0.08 (1)
O15	0.1942 (2)	0.2862 (1)	0.6541 (2)	0.05 (1)
C16	0.0418 (4)	0.2933 (1)	0.4936 (3)	0.06 (1)

Table 4. Selected geometric parameters ($\mathring{A}$, °) for (II)

C1—C2	1.510 (3)	C8—O9	1.466 (3)
C1—C8	1.534 (3)	C10—O9	1.333 (3)
C1—C11	1.524 (3)	C10—C11	1.497 (3)
C2—C3	1.553 (4)	C10—O12	1.199 (3)
C3—C4	1.507 (5)	C11—C13	1.524 (3)
C4—C5	1.309 (5)	C13—O14	1.207 (3)
C5—C6	1.459 (4)	C13—O15	1.305 (3)
C6—C7	1.594 (4)	C16—O15	1.451 (3)
C7—C8	1.515 (3)		
C2—C1—C8	118.0 (2)	C8—O9—C10	111.3 (2)
C8—C1—C11	102.2 (2)	O9—C10—C11	110.1 (2)
C1—C2—C3	115.9 (2)	C11—C10—O12	129.3 (2)
C2—C3—C4	112.2 (3)	O9—C10—O12	120.6 (2)
C3—C4—C5	124.5 (3)	C1—C11—C10	105.7 (2)
C4—C5—C6	124.5 (3)	C1—C11—C13	115.7 (2)
C5—C6—C7	113.3 (2)	C10—C11—C13	112.8 (2)
C6—C7—C8	112.4 (2)	C11—C13—O14	122.2 (2)
C1—C8—C7	117.2 (2)	C11—C13—O15	113.5 (2)
C1—C8—O9	105.9 (2)	O14—C13—O15	124.2 (2)
C7—C8—O9	104.0 (2)	C13—O15—C16	116.3 (2)

Compound (III)

Crystal data

$C_{12}H_{18}O_4$
$M_r = 226.3$
Monoclinic
$P2_1/a$
$a = 7.147\ (2)$ $\mathring{A}$
$b = 23.199\ (4)$ $\mathring{A}$
$c = 7.357\ (2)$ $\mathring{A}$
$\beta = 100.50\ (3)°$
$V = 1199.4\ (4)$ $\mathring{A}^3$
$Z = 4$
$D_x = 1.25$ Mg m^{-3}
$D_m = 1.26\ (2)$ Mg m^{-3}
D_m measured by flotation

Cu $K\alpha$ radiation
$\lambda = 1.5418$ $\mathring{A}$
Cell parameters from 25
  reflections
$\theta = 15-45°$
$\mu = 0.77$ mm^{-1}
$T = 293$ K
Square prism
$0.4 \times 0.2 \times 0.2$ mm
Colourless

Data collection

Enraf–Nonius CAD-4
  diffractometer
θ scans
Absorption correction:
  none
2799 measured reflections
2706 independent reflections
1923 observed reflections
  $[I > 3.0\sigma(I)]$

$R_{int} = 0.033$
$\theta_{max} = 45°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 23$
$l = 0 \rightarrow 8$
3 standard reflections
  frequency: 60 min
  intensity decay: none

Refinement

Refinement on F
$R = 0.0442$
$wR = 0.0442$
$S = 0.66$
1923 reflections
145 parameters
H atoms placed in theo-
  retical positions with
  bond lengths 1 $\mathring{A}$ and
  $U = 0.05\mathring{A}^2$

Unit weights applied
$(\Delta/\sigma)_{max} = 0.11$
$\Delta\rho_{max} = +0.18$ e $\mathring{A}^{-3}$
$\Delta\rho_{min} = -0.20$ e $\mathring{A}^{-3}$
Extinction correction: none
Atomic scattering factors
  from *International Tables
  for X-ray Crystallography*
  (1974, Vol. IV, Table
  2.3.1)

Table 5. *Fractional atomic coordinates and equivalent isotropic displacement parameters ($\mathring{A}^2$) for (III)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C1	0.4894 (1)	0.3727 (1)	1.0150 (1)	0.03 (1)
C2	0.6526 (2)	0.3730 (1)	0.9105 (2)	0.06 (1)
C3	0.7935 (3)	0.4231 (1)	0.9379 (3)	0.07 (1)
C4	0.9229 (2)	0.4237 (1)	1.1296 (3)	0.07 (1)
C5	0.8702 (3)	0.4639 (1)	1.2712 (4)	0.09 (1)
C6	0.6706 (4)	0.4682 (1)	1.2899 (3)	0.10 (2)
C7	0.5568 (2)	0.4181 (1)	1.3446 (2)	0.06 (1)
C8	0.5418 (2)	0.3645 (1)	1.2242 (2)	0.04 (1)
O9	0.3878 (1)	0.3303 (1)	1.2757 (1)	0.05 (1)
C10	0.2797 (2)	0.3059 (1)	1.1303 (2)	0.04 (1)
C11	0.3556 (1)	0.3214 (1)	0.9565 (1)	0.03 (1)
O12	0.1449 (2)	0.2755 (1)	1.1426 (1)	0.06 (1)
C13	0.2023 (2)	0.3322 (1)	0.7898 (2)	0.04 (1)
O14	0.1673 (2)	0.3788 (1)	0.7208 (2)	0.08 (1)
O15	0.1172 (1)	0.2847 (1)	0.7246 (1)	0.05 (1)
C16	−0.0278 (2)	0.2898 (1)	0.5621 (2)	0.06 (1)

Table 6. Selected geometric parameters ($\mathring{A}$, °) for (III)

C1—C2	1.509 (2)	C7—C8	1.519 (2)
C1—C8	1.528 (2)	C8—O9	1.462 (2)
C1—C11	1.538 (1)	O9—C10	1.327 (1)
C2—C3	1.527 (3)	C10—C11	1.520 (1)
C3—C4	1.539 (3)	C10—O12	1.211 (1)
C4—C5	1.497 (3)	C13—O14	1.201 (1)
C5—C6	1.461 (3)	C13—O15	1.307 (1)
C6—C7	1.514 (3)	O15—C16	1.437 (2)
C2—C1—C8	116.3 (1)	C1—C8—O9	105.7 (1)
C8—C1—C11	102.2 (1)	C7—C8—O9	105.6 (1)
C1—C2—C3	119.2 (1)	C8—O9—C10	112.2 (1)
C2—C3—C4	113.5 (2)	O9—C10—C11	109.4 (1)
C3—C4—C5	117.3 (2)	C11—C10—O12	127.7 (1)
C4—C5—C6	118.8 (2)	O9—C10—O12	122.9 (1)
C5—C6—C7	123.6 (2)	C1—C11—C10	104.3 (1)
C6—C7—C8	117.2 (2)	O14—C13—O15	123.8 (1)
C1—C8—C7	117.6 (1)	C13—O15—C16	116.9 (1)

For all compounds, data collection: *CAD-4 Software* (Enraf–Nonius, 1977); cell refinement: *CAD-4 Software*; data reduction: *DATARED* (Pèpe, 1979); program(s) used to solve structures: *MULTAN*80 (Main *et al.*, 1980); program(s) used to refine structures: *SHELX*76 (Sheldrick, 1976); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *AME* (Software Systems, 1986).

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2,5-Dithiahexane-1,6-diyl-4,4'-bis(1,3-dioxolan-2-one)

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Abstract

Molecules of the title compound, $C_{10}H_{14}O_6S_2$, possess crystallographically imposed inversion symmetry, with the central S—C—C—S moiety assuming an ideal *trans* conformation. Molecular-geometry parameters within the 1,3-dioxolan-2-one substituent are compared with those found in the small number of related compounds whose structures have been determined.

Comment

The title compound, (V), is a product of the reaction shown below (Pett *et al.*, 1988).
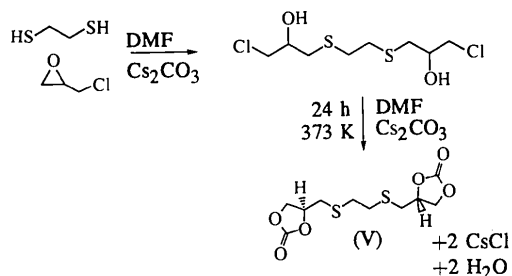
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The molecule occupies a crystallographic inversion centre and therefore possesses C_i molecular symmetry. A small number of structures containing 1,3-dioxolan-2-one units have been reported and some relevant molecular-geometry parameters are given in Table 3. Compound (I) has no reported e.s.d.'s and those for (IV) are high, presumably due in part to the presence of the I atom. The useful comparisons are therefore with compounds (II) and (III), and the title compound shows a very similar geometry to these compounds.
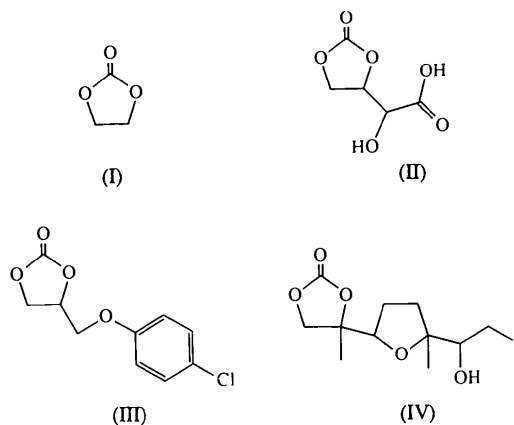
The 1,3-dioxolan-2-one ring is not planar; an excellent least-squares plane may be defined by atoms O1, C2, O2 and O3 [mean deviation 0.005 (3) Å], but atoms C4 and C5 lie 0.122 (5) below and 0.158 (5) Å above this plane, respectively. This imparts a twist conformation to the ring which is distinct from the conformation seen in compound (II) where atoms C4 and C5 are displaced unequally (by 0.04 and 0.13 Å, respectively), but in the same direction, from the carbonate plane (Moen, 1982). In compound (III), the ring conformation is essentially planar (Katzhendler, Ringel, Goldblum, Gibson
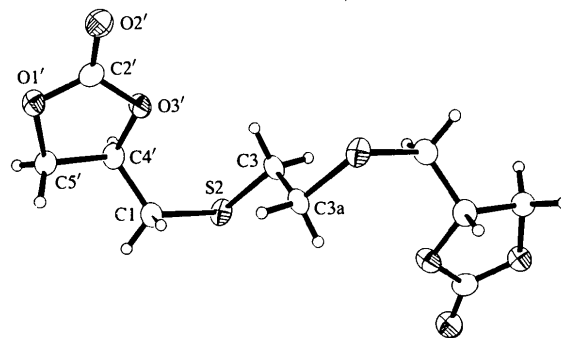

Fig. 1. A view of the title molecule with the atom-numbering scheme; atom C3a is related to atom C3 by inversion through ($\frac{1}{2}$,0,0). Displacement ellipsoids enclose 50% probability surfaces and H atoms are represented by small spheres of arbitrary radii.