Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(1*RS*,2*SR*,6*SR*,7*SR*)-4,10-Dioxatricyclo[5.2.1.$0^{2,6}$]-dec-8-en-3-one

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.006 Å
R factor = 0.085
wR factor = 0.232
Data-to-parameter ratio = 13.6

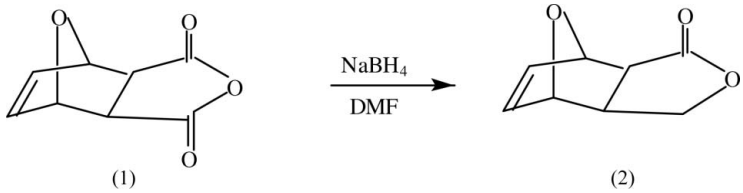
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 23 August 2006
Accepted 25 August 2006

The asymmetric unit of the title compound, $C_8H_8O_3$, contains two molecules. Each molecule involves one planar and two non-planar five-membered rings.

Comment

Since lactone (2) can be prepared optically pure, it becomes a potential precursor for the stereoselective synthesis of natural products (Bloch *et al.*, 1985). Lactone (2) is also used as an intermediate in the synthesis of a cardiovascular agent (Patel *et al.*, 1992). In addition, 7-oxanorbornene derivatives have been screened for their ability to inhibit protein phosphatase 2A (McCluskey *et al.*, 2000). Lactone (2) was prepared by reduction of the *exo* maleic anhydride adduct (1) (Lee & Herndon, 1978). Knowledge of the molecular structure of compound (2) is therefore important in order to understand its formation and reactivity.



(1) (2)

The asymmetric unit of (2) contains two molecules (Fig. 1). The molecules involve five-membered planar *A* (O1*A*/C1*A*/C2*A*/C7*A*/C8*A*) and *A*′ (O1*B*/C1*B*/C2*B*/C7*B*/C8*B*) and non-planar *B* (O3*A*/C2*A*/C3*A*/C6*A*/C*A*), *C* (O3*A*/C3*A*–C6*A*) and *B*′ (O3*B*/C2*B*/C3*B*/C6*B*/C7*B*), *C*′ (O3*B*/C3*B*–C6*B*) rings. The bond lengths and angles (Table 1) are within normal ranges (Allen *et al.*, 1987).

The dihedral angles between the planes *D* (C2*A*/C3*A*/C6*A*/C7*A*), *E* (O3*A*/C3*A*/C6*A*), *F* (C3*A*–C6*A*) and *D*′ (C2*B*/C3*B*/C6*B*/C7*B*), *E*′ (O3*B*/C3*B*/C6*B*), *F*′ (C3*B*–C6*B*) are *D*/*E* = 59.75 (22)°, *F*/*E* = 51.39 (23)° and *D*′/*E*′ = 59.51 (25)° and *F*′/*E*′ = 51.06 (27)°.

Experimental

Compound (2) was prepared according to a literature method (Eggelte *et al.*, 1973). Compound (1) (5.0 g, 0.03 mol) in DMF (25 ml) was added to $NaBH_4$ (1.14 g, 0.03 mol) in DMF (15 ml) over a period of 30 min and stirred for 3 h at 273 K. After evaporation of the solvent, a solid residue of the borate complex was obtained, which was hydrolysed with 1 *M* H_2SO_4 (75 ml). The hydrolysate was extracted with chloroform (4 × 50 ml). The organic extracts were washed with brine (50 ml), dried ($MgSO_4$) and evaporated under reduced pressure. Recrystallization of compound (2) from dichloromethane/pentane (1:3) gave colorless crystals (yield 3.26 g, 71%; m.p. 365–366 K).

Crystal data

$C_8H_8O_3$
M_r = 152.14
Monoclinic, $P2_1/c$
a = 17.110 (4) Å
b = 5.4621 (12) Å
c = 17.129 (4) Å
β = 119.711 (4)°
V = 1390.4 (6) Å^3
Z = 8
D_x = 1.454 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 294 (2) K
Irregular fragment, colorless
0.45 × 0.30 × 0.24 mm

Data collection

Bruker CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
6961 measured reflections
2720 independent reflections
2039 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 27.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.085
$wR(F^2)$ = 0.232
S = 0.97
2720 reflections
200 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1175P)^2 + 2.0024P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O3*A*—C3*A*	1.433 (4)	O3*B*—C3*B*	1.434 (5)
O3*A*—C6*A*	1.436 (4)	O3*B*—C6*B*	1.431 (5)
O1*A*—C1*A*	1.336 (5)	O1*B*—C1*B*	1.328 (5)
O1*A*—C8*A*	1.457 (4)	O1*B*—C8*B*	1.445 (5)
O2*A*—C1*A*	1.215 (5)	O2*B*—C1*B*	1.216 (5)
C3*A*—O3*A*—C6*A*	96.5 (3)	C3*B*—O3*B*—C6*B*	96.6 (3)
C1*A*—O1*A*—C8*A*	111.3 (3)	C1*B*—O1*B*—C8*B*	111.3 (3)
O2*A*—C1*A*—C2*A*	128.0 (4)	O2*B*—C1*B*—C2*B*	127.9 (4)

H atoms were positioned geometrically, with C—H = 0.93, 0.98 and 0.97 Å for aromatic, methine and methylene H atoms, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia,1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

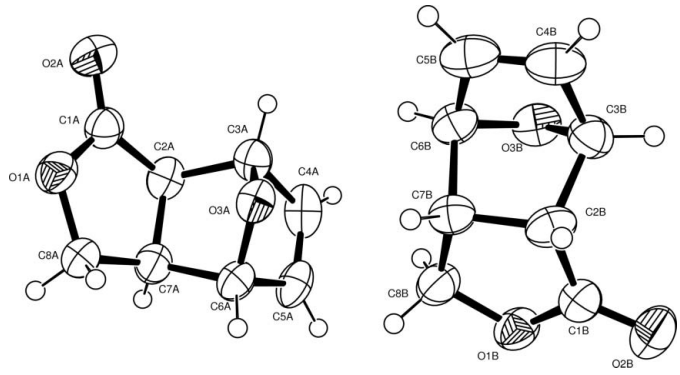


Figure 1
The asymmetric unit of (2), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 70% probability level.

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