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endo-2,4-Dibromo-3a,4,7,7a-tetrahydro-4,7-methanoindene-1,8-dione 8-Ethylene Acetal

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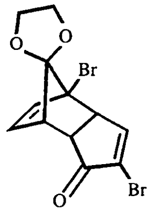
Abstract

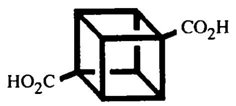
In the title compound, $C_{12}H_{10}Br_2O_3$, the C_{sp^2}—Br bond is found to be shorter than the C_{sp^3}—Br bond, while the bonds involving the apical C atom (the methano bridge) have distances between those observed for the corresponding bonds in the chloro-substituted compound 1,2,4,5,6,7,8,9-octachloro-10,10-dimethoxytricyclo-[5.2.1.0^{2,6}]deca-4,8-diene-3-one [Galešić *et al.* (1985). *Acta Cryst.* C**41**, 1662–1664] and those observed for the corresponding bonds in similar compounds without halogen substituents.

Comment

The title compound, (1), is a key precursor in the synthesis of cubane-1,4-dicarboxylic acid, (2). The preparation of (1) was first described by Eaton & Cole (1964) and later modified by Chapman *et al.* (1970). We report herein the X-ray structure determination of (1).



(1)



(2)

The C4—Br2 distance is significantly shorter than the distance found for the other C—Br bond; a similar situation is observed in 1,2,4,5,6,7,8,9-octachloro-10,10-dimethoxytricyclo[5.2.1.0^{2,6}]deca-4,8-diene-3-one (Galešić *et al.*, 1985), where the C_{sp^2}—Cl bonds were found to be shorter than the C_{sp^3}—Cl bonds. The atoms of the cyclopentene ring are coplanar, with the maximum deviation from the plane being 0.024 (2) Å (C6), while the cyclopentane and oxolane rings have envelope conformations (Altona *et al.*, 1968). The dihedral angle between the cyclopentene ring and the plane defined by atoms C1, C9, C8 and C7 is 59.07 (14)°. The bonds involving the apical C atom, C7—C10 and C1—C10, have lengths between those observed for the corresponding bonds in the above-mentioned Cl-substituted compound (1.573–1.576 Å; Galešić *et al.*, 1985) and those observed for the corresponding bonds in similar compounds without halogen substitutents (1.519–1.547 Å), such as diethyl 4,7-dimethyl-5,10-dioxo-*endo*-tricyclo[5.2.1.0^{2,6}]deca-3,8-diene-3,8-dicarboxylate (Smits *et al.*, 1988) and 1-methyl-3-oxotricyclo[5.2.1.0^{2,6}]deca-4,8-diene-6-carboxylic acid (Watson *et al.*, 1990). In contrast, the C1—C2 and C6—C7 bond lengths are at the lower end of the range observed for such compounds (1.562–1.622 Å).

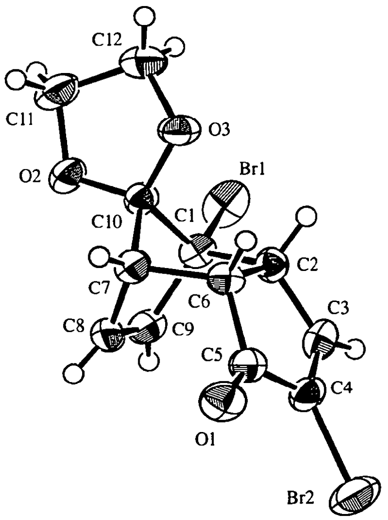


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of (1). Displacement ellipsoids are drawn at the 50% probability level. H atoms have been given arbitrary radii (0.1 Å).

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Experimental

Compound (1) was prepared as described by Chapman *et al.* (1970) and crystals were obtained by slow evaporation from toluene.

Crystal data

$C_{12}H_{10}Br_2O_3$
$M_r = 362.02$
Triclinic
$P\bar{1}$
$a = 6.2510\,(11)$ Å
$b = 8.187\,(2)$ Å
$c = 11.784\,(2)$ Å
$\alpha = 91.43\,(2)^\circ$
$\beta = 94.74\,(2)^\circ$
$\gamma = 90.56\,(2)^\circ$
$V = 600.8\,(2)$ Å^3
$Z = 2$
$D_x = 2.001$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10.6$–18.4°
$\mu = 6.739$ mm^{-1}
$T = 293\,(1)$ K
Needle
0.67 × 0.28 × 0.22 mm
Colourless

Data collection

Enraf–Nonius CAD-4-MachS diffractometer
$\omega/2\theta$ scans
Absorption correction: Gaussian integration (Sheldrick, 1976)
$T_{\min} = 0.139$, $T_{\max} = 0.299$
3518 measured reflections
2761 independent reflections

2232 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.014$
$\theta_{\max} = 27.48^\circ$
$h = -8 \rightarrow 1$
$k = -10 \rightarrow 10$
$l = -15 \rightarrow 15$
3 standard reflections frequency: 160 min intensity decay: 8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.088$
$S = 1.073$
2760 reflections
194 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2 + 0.1744P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.601$ e Å^{-3}
$\Delta\rho_{\min} = -0.446$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Br1—C1	1.925 (3)	C3—C4	1.320 (4)
Br2—C4	1.872 (3)	C6—C7	1.563 (4)
C1—C9	1.499 (4)	C7—C8	1.489 (4)
C1—C2	1.547 (4)	C7—C10	1.541 (4)
C1—C10	1.552 (4)	C8—C9	1.313 (4)
C9—C1—C2	108.9 (2)	C3—C4—C5	112.0 (3)
C9—C1—C10	100.2 (2)	C3—C4—Br2	127.0 (2)
C2—C1—C10	100.2 (2)	C5—C4—Br2	121.0 (2)
C9—C1—Br1	115.2 (2)	C8—C7—C10	100.1 (2)
C2—C1—Br1	114.9 (2)	C8—C7—C6	106.9 (2)
C10—C1—Br1	115.5 (2)	C10—C7—C6	99.5 (2)
C4—C3—C2	112.2 (3)	C7—C10—C1	92.4 (2)

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1186). Services for accessing these data are described at the back of the journal.

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