$C_{32}H_{29}NO_4$

in that it is disubstituted at the atom α to the carbonyl C atom.

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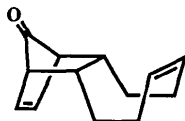
Tricyclo[8.2.1.0²,⁹]trideca-5,11-dien-13-one

By J. Gabriel Garcia, Frank R. Fronczek and Mark L. McLaughlin*

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Abstract. $C_{13}H_{16}O$, $M_r = 188\cdot3$, orthorhombic, $P2_12_12_1$, $a = 8\cdot6255\,(12)$, $b = 8\cdot8372\,(12)$, $c = 13\cdot6741\,(7)$ Å, $V = 1042\cdot3\,(3)$ Å³, $Z = 4$, $D_x = 1\cdot200\,\mathrm{g\,cm^{-3}}$, $\lambda(\mathrm{Cu}\,K\alpha) = 1\cdot54184$ Å, $\mu = 5\cdot36\,\mathrm{cm^{-1}}$, $F(000) = 408$, $T = 297$ K, $R = 0\cdot031$ for 1785 observations with $I > 3\sigma(I)$ (of 2138 unique data). The cyclooctene ring adopts the twist–boat conformation. There is a near-zero torsion angle of the boat at the ring-fusion bond, with magnitude of $-2\cdot8\,(2)°$. The torsion angles about the bonds comprising the sides of the boat are $33\cdot8\,(2)$ and $18\cdot5\,(2)°$. The cyclooctene and norbornenone C=C bonds have lengths of $1\cdot297\,(3)$ and $1\cdot318\,(2)$ Å, respectively, and the C=O bond length is $1\cdot205\,(2)$ Å.

Experimental. The title compound was prepared by allowing one equivalent of 1,1-dimethoxy-2,3,4,5-tetrachlorocyclopentadiene to react with eight equivalents of 1,5-cyclooctadiene neat at 406–413 K (Akhtar, Fray & Yarrow, 1968) followed by reduction in sodium–ethanol and hydrolysis with sulfuric acid–water–ether (Eaton, Sidhu, Langford, Cullison & Pietruszewski, 1987).

Crystals that deposited from the reaction flask, m.p. 338–340 K, were suitable; a clear colorless crystal with dimensions $0\cdot15 \times 0\cdot18 \times 0\cdot32$ mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer with Cu $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined from setting angles of 25 reflections having $30 > \theta > 25°$. The $\omega-2\theta$ scans were designed for $I = 20\sigma(I)$, subject to max. scan time = 60 s, scan rates varied from $1\cdot0-4\cdot1°\,\mathrm{min^{-1}}$. A hemisphere of data having $2 < \theta < 75°$, $0 \leq h \leq 10$, $-11 \leq k \leq 11$, $-17 \leq l \leq 17$ was measured and corrected for background, Lorentz, polarization and decay. ψ scans of four reflections exhibited no decrease in intensity with rotation about the diffraction vector, thus no absorption correction was applied. Three standard reflections (400, 031, 006) decreased in intensity by $5\cdot0\%$ apparently due to sublimation and a linear correction was applied. 4637 data were measured, equivalent data were averaged, $R_{int} = 0\cdot014$, yielding 2138 unique data. Systematic absences $h00$ with h odd, $0k0$ with k odd and $00l$ with l odd indicated space group $P2_12_12_1$. The structure was solved by direct methods using *RANTAN* (Yao, 1981), refined by full-matrix least squares based upon F, using data for which $I > 3\sigma(I)$, weights $w = 4F_o^2[\sigma^2(I) + (0\cdot02F_o^2)^2]^{-1}$ using the *Enraf–Nonius Structure Determination Package* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), and anomalous coefficients of Cromer (1974). Heavy-atom coordinates were refined with anisotropic thermal parameters; H-atom coordinates were located by ΔF and were refined with

Table 1. *Coordinates and equivalent isotropic thermal parameters*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
O	0·6674 (1)	0·4059 (2)	0·89767 (9)	6·72 (3)
C1	0·3318 (1)	0·5348 (1)	0·81568 (9)	3·42 (2)
C2	0·2987 (1)	0·5000 (2)	0·92635 (9)	3·61 (2)
C3	0·1300 (2)	0·4894 (2)	0·9581 (1)	4·83 (3)
C4	0·0595 (2)	0·6448 (2)	0·9835 (2)	7·57 (5)
C5	0·1116 (2)	0·7797 (2)	0·9280 (1)	5·75 (4)
C6	0·1216 (2)	0·7908 (2)	0·8336 (2)	5·94 (4)
C7	0·0758 (2)	0·6693 (2)	0·7647 (1)	6·81 (4)
C8	0·1974 (2)	0·5496 (2)	0·7437 (1)	5·03 (3)
C9	0·4429 (2)	0·4033 (2)	0·7847 (1)	3·99 (3)
C10	0·3550 (2)	0·2571 (2)	0·7908 (1)	4·69 (3)
C11	0·3259 (2)	0·2279 (2)	0·8835 (1)	5·26 (3)
C12	0·3929 (2)	0·3518 (2)	0·9446 (1)	4·62 (3)
C13	0·5319 (2)	0·3885 (2)	0·8797 (1)	4·42 (3)

Table 2. *Bond distances* (Å), *angles* (°) *and selected torsion angles* (°)

O—C13	1·205 (2)	C5—C6	1·297 (3)
C1—C2	1·571 (2)	C6—C7	1·483 (3)
C1—C8	1·526 (2)	C7—C8	1·517 (2)
C1—C9	1·565 (2)	C9—C10	1·500 (2)
C2—C3	1·522 (2)	C9—C13	1·514 (2)
C2—C12	1·561 (2)	C10—C11	1·318 (2)
C3—C4	1·542 (2)	C11—C12	1·494 (2)
C4—C5	1·483 (3)	C12—C13	1·526 (2)
C2—C1—C8	120·0 (1)	C1—C9—C10	108·4 (1)
C2—C1—C9	103·1 (1)	C1—C9—C13	98·2 (1)
C8—C1—C9	110·8 (1)	C10—C9—C13	97·8 (1)
C1—C2—C3	117·5 (1)	C9—C10—C11	108·5 (1)
C1—C2—C12	102·9 (1)	C10—C11—C12	108·7 (1)
C3—C2—C12	113·6 (1)	C2—C12—C11	108·9 (1)
C2—C3—C4	112·7 (1)	C2—C12—C13	97·9 (1)
C3—C4—C5	118·8 (2)	C11—C12—C13	97·7 (1)
C4—C5—C6	126·2 (2)	O—C13—C9	131·0 (1)
C5—C6—C7	124·0 (2)	O—C13—C12	132·1 (2)
C6—C7—C8	116·1 (1)	C9—C13—C12	96·8 (1)
C1—C8—C7	117·6 (1)		
C8—C1—C2—C3	−2·8 (2)	C1—C2—C3—C4	−85·5 (2)
C2—C1—C8—C7	58·4 (2)	C2—C3—C4—C5	33·8 (2)
C6—C7—C8—C1	18·5 (2)	C4—C5—C6—C7	2·5 (3)
C3—C4—C5—C6	48·7 (3)	C5—C6—C7—C8	−85·0 (2)

isotropic thermal parameters. Final $R = 0.031$ for 1785 observed data (0·041 for all 2138 data), $wR = 0.037$, and $S = 1.456$ for 192 variables. Max. shift 0.09σ in the final cycle, max. residual density 0·18, min. -0.13 e Å^{-3}, and extinction coefficient $g = 9.34$ (12) × 10^{-6} where the factor $(1 + gI_c)^{-1}$ was applied to F_c. The fractional coordinates of the title compound are given in Table 1. Fig. 1 is a perspective drawing showing the atom numbering. Fig. 2 illustrates the unit cell. Bond distances, angles, and selected torsion angles are presented in Table 2.*

* Lists of structure factors, anisotropic thermal parameters, distances and angles involving H atoms, torsion angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54058 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. Bond lengths and bond angles C1—C2 1·571 (2), C10—C11 1·318 (2) Å and C8—C1—C9 110·8 (1)° of tricyclo[8.2.1.0^{2,9}]trideca-5,11-dien-13-one are similar to those of *endo,-endo,anti*-1,6,7,8,9,14,15,16-octachloropentacyclo-[12.2.1.1^{6,9}.0^{2,13}.0^{5,10}]octadeca-7,15-diene [C1—C2 1·587 (3), C10—C11 1·327 (3) Å and C8—C1—C9 114·1 (2)°] (Garcia, Fronczek & McLaughlin, 1991*a*) and to those of *endo,endo,anti*-1,6,7,8,9,14,15,-16-octachloro-17,17,18,18-tetramethoxypentacyclo-[12.2.1.1^{6,9}.0^{2,13}.0^{5,10}]octadeca-7,15-diene [C1—C2 1·579 (3), C10—C11 1·324 (3) Å and C8—C1—C9 113·2 (2)°] (Garcia, Fronczek & McLaughlin, 1991*b*). The C5—C6 bond distance of 1·297 (3) Å for the title compound is in agreement with that of *trans*-spiro[bicyclo[6.3.0]undec-4-ene-10,2'-[1,3]-dioxolane]-2,2,7,7-tetracarboxylate, 1·319 (4) Å (Bertram, Jansen, Peters, Meier & Winterfeldt, 1986). Torsion angle C8—C1—C2—C3 at the ring fusion bond is −2·8 (2)° in accordance with those of *endo,-endo,anti*-1,6,7,8,9,14,15,16-octachloropentacyclo-[12.2.1.1^{6,9}.0^{2,13}.0^{5,10}]octadeca-7,15-diene [−0·3 (3)°] (Garcia *et al.*, 1991*a*), and *endo,endo,anti*-1,6,7,8,9,14,15,16-octachloro-17,17,18,18-tetrameth-oxypentacyclo[12.2.1.1^{6,9}.0^{2,13}.0^{5,10}]octadeca-7,15-diene [1·8 (3)°] (Garcia *et al.*, 1991*b*).
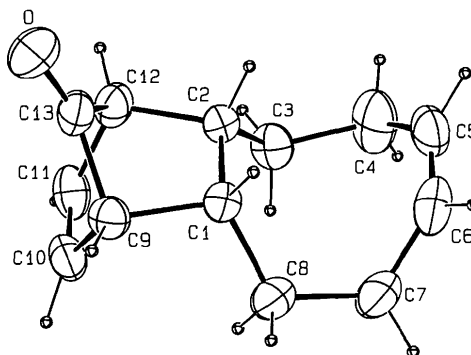

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule, representing C atoms as 30% probability ellipsoids and H atoms as circles of arbitrary radius.
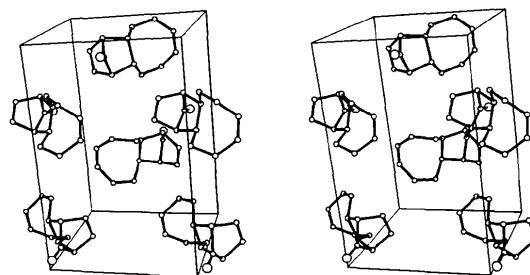

Fig. 2. Stereoview of the unit cell. **c** is vertical, **b** is horizontal and **a** is into the plane of the paper.

The cyclooctene ring has a twist–boat conformation, with torsion angles ω_1 C3—C2—C1—C8, C4—C5—C6—C7, ω_2 C2—C1—C8—C7, C1—C2—C3—C4, C3—C4—C5—C6, C5—C6—C7—C8 and ω_3 C2—C3—C4—C5, C1—C8—C7—C6 of $\omega_1 = -2\cdot8$ (2), $\omega_2 = 58\cdot4$ (2), $-85\cdot5$ (2) and $\omega_3 = 33\cdot8$ (2), $18\cdot5$ (2)° that are distorted from the ideal boat D_{2d} conformation (of cylooctane) having torsion angles of $\omega_1 = \pm\,0$, $\omega_2 = \pm\,76\cdot4$ and $\omega_3 = \pm\,0$° (Hendrickson, 1964), and $\omega_1 = \pm\,0$, $\omega_2 = \pm\,73\cdot4$, and $\omega_3 = \pm\,0$° (Ivanov & Osawa, 1984).

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5-Methyl-4-(1-piperidyl)-5-trifluoromethyl-1,2-oxathiole

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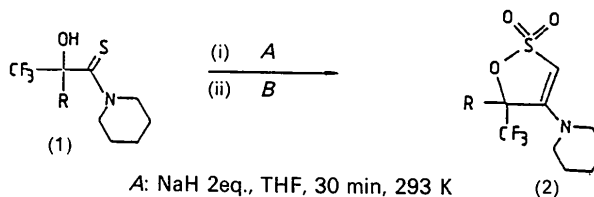
and C. Maliverney and H. G. Viehe

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Abstract. $C_{10}H_{14}F_3NO_3S$, $M_r = 285\cdot28$, orthorhombic, $Pca2_1$, $a = 9\cdot324$ (2), $b = 12\cdot038$ (3), $c = 11\cdot252$ (3) Å, $V = 1263\cdot0$ (5) Å^3, $Z = 4$, $D_x = 1\cdot50$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 2\cdot97$ cm^{-1}, $F(000) = 592$, $T = 291$ K, $R = 0\cdot032$ for 1124 observed reflections. The five-membered unsaturated sultone ring is slightly nonplanar adopting a flat C2 half-chair conformation with endocyclic torsion angles of $5\cdot2$ (8), $-1\cdot9$ (8), $-1\cdot8$ (8), $5\cdot1$ (8) and $-6\cdot6$ (8)°. The endocyclic S—O and S—C bond lengths are $1\cdot599$ (2) and $1\cdot689$ (4) Å, respectively, and the C—S—O angle is $95\cdot4$ (2)°.

Experimental. The reaction of methanesulfonyl chloride with the sodium alcoholate of the 2-hydroxythioamide (1), in the presence of an excess of sodium hydride, leads unexpectedly to the product (2) (Maliverney, Merenyi & Viehe, 1990). This compound was identified as being a sultone, *i.e.* a cyclic sulfonate and the X-ray analysis was undertaken in order to confirm this structure and to determine the geometry of this new unsaturated heterocycle.

A: NaH 2eq., THF, 30 min, 293 K   (2)
B: Cl—SO$_2$—CH$_3$ 1·2 eq., 24 h, 293 K

Parallelepiped crystal of approximate dimensions $0\cdot35 \times 0\cdot22 \times 0\cdot14$ mm. Lattice parameters refined