

References

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Structure of 5,5,15,15-Tetramethyl-6,16-dioxapentacyclo[9.6.1.0^{1,13}.0^{2,10}.0^{3,8}]-octadeca-3(8),12-diene-7,17-dione

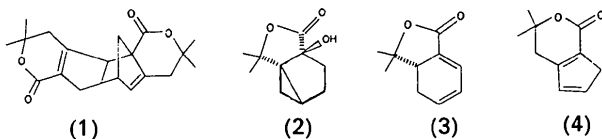
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Abstract. C₂₀H₂₄O₄, *M_r* = 328.41, monoclinic, *P*₂₁/*n*, *a* = 10.546 (5), *b* = 8.774 (2), *c* = 18.931 (9) Å, β = 101.88 (4)°, *V* = 1714 (1) Å³, *Z* = 4, *D_x* = 1.272 g cm⁻³, λ = 0.71073 Å, μ(Mo Kα) = 0.82 cm⁻¹, *F*(000) = 704, *T* = 298 K, *R* = 0.047 for 1889 observed reflections. The compound is obtained by a Diels–Alder cycloaddition between two 7,7-dimethyl-8-oxabicyclo[4.3.0]-nona-1,3-dien-9-one molecules. The folding along the common bond between the five-membered and the cyclohexene ring, plus the boat conformation of the latter, force the molecule to adopt an overall hemispherical conformation.

Experimental. The synthesis of compound (1) has been reported previously (Marsaioli, Nurnberg, Sarragiotto & Castellano, 1989).



The data collection and refinement parameters are summarized in Table 1. The structure was solved using standard direct-methods and difference-Fourier techniques. In the final cycles of full-matrix least-squares refinement all non-H atoms were treated anisotropically. H atoms were included as fixed contributors at positions found in difference syntheses, slightly modified when possible on stereochemical grounds, all with a common isotropic temperature factor that refined to *B* = 4.50 Å².

Scattering factors for non-H atoms were taken from Cromer & Mann (1968) with corrections for anomalous dispersion from Cromer & Liberman (1970), for the H atoms from Stewart, Davidson & Simpson (1965). Programs used were *SHELX76* (Sheldrick, 1976) and *ORTEP* (Johnson, 1965). All calculations were performed on a VAX 11/780 computer.

Atomic coordinates are listed in Table 2* and bond lengths and angles in Table 3. Fig. 1 is a stereoscopic projection of the molecule.

Related literature. The title compound is one of the two dimers formed in an attempt to transform (2) into lactone (3), treating (2) with perchloric acid (Marsaioli, Nurnberg, Sarragiotto & Castellano, 1989, and references therein). The structure deter-

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52969 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

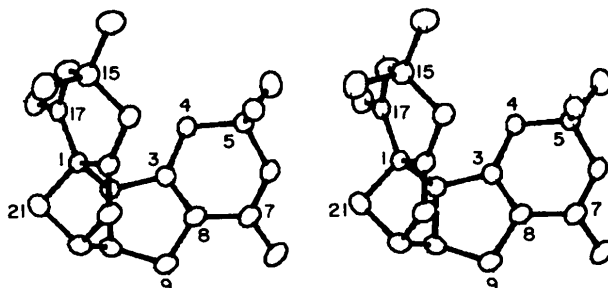


Fig. 1. Stereoscopic projection of compound (1).

Table 1. Crystallographic summary for (1)

Data collection*	
Mode	θ -2 θ
Scan rate ($^{\circ}$ min ⁻¹)	2.6-6.7
θ range ($^{\circ}$)	0-25
Range of hkl	$-12 \leq h \leq 12, 0 \leq k \leq 10, 0 \leq l \leq 22$
Total reflections measured	3341
Unique reflections	2705
R_{int}	0.030
Crystal dimensions (mm)	$\sim 0.5 \times 0.5 \times 0.4$
Structure refinement†	
Reflections used [$I > 3\sigma(I)$]	1889
No. of variables	218
R, wR	0.047, 0.057
Max. shift/e.s.d.	0.010
Max., min. density in final difference map ($e \text{ \AA}^{-3}$)	0.20, -0.25
S	0.93

* Unit-cell parameters by least-squares refinement of the setting angles of 25 reflections with $10 < \theta < 20^{\circ}$. Enraf-Nonius CAD-4 diffractometer with a graphite monochromator. One standard reflection (400) measured every half an hour of radiation time showed no significant variation. No correction for absorption.

† Function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w^{-1} = [\sigma^2(F_o) + 0.005F_o^2]$.

Table 2. Fractional atomic coordinates and isotropic temperature factors (\AA^2) for non-H atoms

	x	y	z	B_{iso}
C(1)	0.0974 (2)	-0.1714 (3)	-0.2066 (1)	2.59 (7)
C(2)	0.2157 (2)	-0.2028 (3)	-0.1424 (1)	2.61 (7)
C(3)	0.2561 (2)	-0.0813 (3)	-0.0862 (1)	2.59 (6)
C(4)	0.3221 (2)	0.0645 (3)	-0.0964 (1)	2.79 (7)
C(5)	0.3188 (3)	0.1764 (3)	-0.0341 (1)	3.16 (8)
C(7)	0.2926 (3)	-0.0398 (3)	0.0435 (1)	3.37 (8)
C(8)	0.2370 (2)	-0.1252 (3)	-0.0217 (1)	2.95 (7)
C(9)	0.1778 (3)	-0.2806 (3)	-0.0217 (1)	3.59 (8)
C(10)	0.1620 (3)	-0.3337 (3)	-0.0998 (1)	3.17 (7)
C(11)	0.0228 (3)	-0.3564 (3)	-0.1445 (1)	3.50 (8)
C(12)	-0.0559 (2)	-0.2133 (3)	-0.1387 (1)	3.42 (8)
C(13)	-0.0117 (2)	-0.1032 (3)	-0.1753 (1)	2.79 (7)
C(14)	-0.0455 (2)	0.0588 (3)	-0.1913 (1)	3.25 (8)
C(15)	-0.0536 (2)	0.0901 (3)	-0.2718 (1)	3.34 (8)
C(17)	0.1335 (2)	-0.0851 (3)	-0.2682 (1)	2.78 (7)
C(18)	0.4238 (3)	0.2968 (3)	-0.0272 (2)	4.25 (9)
C(19)	-0.1633 (3)	0.0045 (4)	-0.3190 (2)	4.8 (1)
C(20)	-0.0620 (3)	-0.2589 (4)	-0.2868 (2)	4.6 (1)
C(21)	0.0472 (3)	-0.3361 (3)	-0.2215 (1)	3.45 (8)
C(22)	0.1870 (3)	0.2496 (3)	-0.0402 (1)	3.62 (8)
O(1)	0.3478 (2)	0.0966 (2)	0.0356 (1)	3.89 (6)
O(2)	0.3000 (2)	-0.0885 (3)	0.1045 (1)	4.80 (7)
O(3)	0.0679 (2)	0.0415 (2)	-0.2926 (1)	3.53 (6)
O(4)	0.2224 (2)	-0.1238 (2)	-0.2957 (1)	3.74 (6)

mination led to the conclusion that a Diels-Alder cycloaddition occurred between two diene molecules possessing structure (4)

Table 3. Interatomic bond distances (\AA) and angles ($^{\circ}$) for non-H atoms

C(1)—C(2)	1.576 (3)	C(1)—C(17)	1.504 (3)
C(1)—C(13)	1.521 (3)	C(1)—C(21)	1.545 (4)
C(2)—C(3)	1.504 (3)	C(3)—C(4)	1.488 (3)
C(4)—C(5)	1.540 (3)	C(5)—C(18)	1.516 (4)
C(5)—C(22)	1.514 (4)	C(5)—O(1)	1.469 (3)
C(7)—C(8)	1.460 (4)	C(7)—O(1)	1.353 (3)
C(7)—O(2)	1.219 (3)	C(8)—C(3)	1.335 (3)
C(8)—C(9)	1.500 (4)	C(9)—C(10)	1.526 (4)
C(10)—C(2)	1.574 (4)	C(10)—C(11)	1.549 (4)
C(11)—C(12)	1.521 (4)	C(11)—C(21)	1.542 (4)
C(12)—C(13)	1.328 (4)	C(13)—C(14)	1.481 (4)
C(14)—C(15)	1.533 (4)	C(15)—C(19)	1.508 (4)
C(15)—C(20)	1.507 (4)	C(15)—O(3)	1.479 (3)
C(17)—O(3)	1.339 (3)	C(17)—O(4)	1.211 (3)
C(2)—C(1)—C(17)	113.5 (2)	C(2)—C(1)—C(13)	108.1 (2)
C(2)—C(1)—C(21)	99.5 (2)	C(13)—C(1)—C(17)	115.9 (2)
C(13)—C(1)—C(21)	100.4 (2)	C(17)—C(1)—C(21)	117.5 (2)
C(1)—C(2)—C(3)	119.0 (2)	C(1)—C(2)—C(10)	102.2 (2)
C(3)—C(2)—C(10)	103.5 (2)	C(2)—C(3)—C(4)	126.1 (2)
C(2)—C(3)—C(8)	111.6 (2)	C(4)—C(3)—C(8)	122.0 (2)
C(3)—C(4)—C(5)	111.3 (2)	C(4)—C(5)—C(18)	112.3 (2)
C(4)—C(5)—C(22)	112.2 (2)	C(18)—C(5)—C(22)	110.7 (2)
C(4)—C(5)—O(1)	110.5 (2)	C(18)—C(5)—O(1)	104.0 (2)
C(22)—C(5)—O(1)	106.8 (2)	C(8)—C(7)—O(1)	117.9 (2)
C(8)—C(7)—O(2)	123.9 (2)	O(1)—C(7)—O(2)	118.0 (2)
C(3)—C(8)—C(7)	121.0 (2)	C(3)—C(8)—C(9)	113.8 (2)
C(7)—C(8)—C(9)	124.2 (2)	C(8)—C(9)—C(10)	103.9 (2)
C(2)—C(10)—C(9)	107.1 (2)	C(2)—C(10)—C(11)	102.4 (2)
C(9)—C(10)—C(11)	118.1 (2)	C(10)—C(11)—C(12)	108.9 (2)
C(10)—C(11)—C(21)	100.2 (2)	C(12)—C(11)—C(21)	99.9 (2)
C(11)—C(12)—C(13)	107.9 (2)	C(1)—C(13)—C(12)	107.0 (2)
C(1)—C(13)—C(14)	117.9 (2)	C(12)—C(13)—C(14)	135.2 (2)
C(13)—C(14)—C(15)	109.7 (2)	C(14)—C(15)—C(19)	112.2 (2)
C(14)—C(15)—C(20)	110.6 (2)	C(19)—C(15)—C(20)	111.7 (2)
C(14)—C(15)—O(3)	109.8 (2)	C(19)—C(15)—O(3)	107.4 (2)
C(20)—C(15)—O(3)	104.7 (2)	C(1)—C(17)—O(3)	119.3 (2)
C(1)—C(17)—O(4)	122.3 (2)	O(3)—C(17)—O(4)	118.4 (2)
C(1)—C(21)—C(11)	93.0 (2)	C(7)—O(1)—C(5)	120.4 (2)
C(15)—O(3)—C(17)	124.2 (2)		

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