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Structure of the Product from the Reaction Between Methyl 1-Methyl-1,2,3,4,9,10-hexahydrophenanthren-1-yl Ketone and Ethyl Orthoformate

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Abstract. (4bα,7aβ,10a*S**,13*S**)-5,6,7,7a,9,10,11,12-Octahydro-7a,13-dimethyl-8*H*-4b,9-(epoxymethano)-cyclopenta[*j*]phenanthren-8-one, $C_{20}H_{24}O_2$, M_r = 296·43, monoclinic, $P2_1$, a = 7·285 (1), b = 14·052 (2), c = 7·699 (2) Å, β = 94·54 (2)°, V = 786 (1) Å^3, Z = 2, D_x = 1·25 Mg m^{-3}, Cu $K\alpha$, λ = 1·5418 Å, μ = 0·54 mm^{-1}, $F(000)$ = 320, T = 293 K, R = 0·036 for 1577 reflections with $I > 2{\cdot}5\sigma(I)$. The X-ray analysis confirms the structure proposed on spectroscopic grounds for the compound obtained from the reaction between methyl 1-methyl-1,2,3,4,9,10-hexahydrophenanthren-1-yl ketone and ethyl orthoformate, involving attack on C(4a) and C(10a) of the former. The aromatic C–C bond lengths are 1·382 (4)–1·399 (4) Å, mean 1·391 Å. The C(sp^3)–C(sp^3) bond lengths range from 1·521 (3) to 1·559 (2) Å, mean 1·534 Å, the longest bonds being associated with the fully substituted C atoms [C(1), C(4a), C(10a)].

Table 1. *Fractional atomic coordinates* (×10^4) *and equivalent isotropic thermal parameters* (Å^2 ×10^3)

$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$.

	x	y	z	U_{eq}
O(1)	6703 (2)	8954	12898 (2)	60 (1)
O(2)	8880 (2)	7512 (2)	9990 (2)	37 (1)
C(1)	5556 (2)	8713 (2)	9856 (3)	40 (1)
C(2)	6620 (3)	9532 (2)	9109 (3)	48 (1)
C(3)	8513 (3)	9284 (2)	8523 (3)	48 (1)
C(4)	8449 (3)	9374 (2)	7435 (3)	41 (1)
C(4a)	7636 (2)	7547 (2)	8427 (2)	33 (1)
C(5)	9424 (3)	6134 (2)	7475 (3)	44 (1)
C(5a)	7746 (2)	6619 (2)	7418 (2)	36 (1)
C(6)	9626 (3)	5309 (2)	6528 (3)	55 (1)
C(7)	8126 (4)	4954 (2)	5507 (3)	59 (1)
C(8)	6451 (3)	5418 (2)	5449 (3)	53 (1)
C(8a)	6236 (3)	6254 (2)	6400 (2)	42 (1)
C(9)	4370 (3)	6700 (3)	6374 (3)	52 (1)
C(10)	4367 (3)	7701 (2)	7167 (3)	47 (1)
C(10a)	5623 (2)	7753 (2)	8851 (2)	36 (1)
C(11)	6298 (3)	8415 (2)	11704 (3)	42 (1)
C(12)	6365 (3)	7330 (2)	11810 (3)	44 (1)
C(13)	5104 (3)	7032 (2)	10227 (3)	43 (1)
C(14)	8309 (3)	7009 (2)	11461 (2)	43 (1)
C(15)	9772 (4)	7209 (3)	12936 (3)	62 (1)
C(16)	3566 (3)	9062 (2)	10062 (4)	58 (1)

Experimental. Colourless crystal of title compound (I) from pentane, dimensions 0·2 × 0·2 × 0·4 mm. Enraf–Nonius diffractometer, graphite monochromator, Cu $K\alpha$ radiation, generator settings 43 kV, 26 mA. Cell dimensions from setting angles of 25 independent reflections with θ 14–20°; 1760 intensities surveyed in the range θ 2–72°; h −8–8, k 0–17, l 0–9; $\omega/2\theta$ scan, scan width (0·80 + 0·14 tanθ)°; scan rate 1·20–6·67° min^{-1}; max. scan time 120 s; 1577 independent reflections with $I > 2{\cdot}5\sigma(I)$. Two reference reflections

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

O(1)–C(11)	1·209 (3)	C(5)–C(6)	1·384 (3)
O(2)–C(4a)	1·449 (2)	C(5a)–C(8a)	1·397 (3)
O(2)–C(14)	1·425 (2)	C(6)–C(7)	1·387 (4)
C(1)–C(2)	1·526 (3)	C(7)–C(8)	1·382 (4)
C(1)–C(10a)	1·559 (2)	C(8)–C(8a)	1·399 (3)
C(1)–C(11)	1·540 (3)	C(8a)–C(9)	1·496 (3)
C(1)–C(16)	1·551 (3)	C(9)–C(10)	1·532 (3)
C(2)–C(3)	1·525 (3)	C(10)–C(10a)	1·528 (2)
C(3)–C(4)	1·526 (3)	C(10a)–C(13)	1·534 (2)
C(4)–C(4a)	1·535 (2)	C(11)–C(12)	1·527 (3)
C(4a)–C(5a)	1·523 (2)	C(12)–C(13)	1·526 (3)
C(4a)–C(10a)	1·554 (2)	C(12)–C(14)	1·530 (3)
C(5)–C(5a)	1·397 (3)	C(14)–C(15)	1·521 (3)

C(1)–C(11)–O(1)	125·4 (2)	C(12)–C(11)–O(1)	125·5 (2)
C(14)–O(2)–C(4a)	118·3 (1)	C(4)–C(4a)–O(2)	101·4 (1)
C(5a)–C(4a)–O(2)	109·9 (1)	C(10a)–C(4a)–O(2)	111·7 (1)
C(12)–C(14)–O(2)	109·0 (1)	C(15)–C(14)–O(2)	105·7 (2)
C(10a)–C(1)–C(2)	115·4 (2)	C(11)–C(1)–C(2)	113·7 (2)
C(16)–C(1)–C(2)	107·9 (2)	C(3)–C(2)–C(1)	115·9 (2)
C(11)–C(1)–C(10a)	101·6 (1)	C(16)–C(1)–C(10a)	113·0 (2)
C(4a)–C(10a)–C(1)	109·4 (1)	C(10)–C(10a)–C(1)	115·0 (2)
C(13)–C(10a)–C(1)	102·1 (1)	C(16)–C(1)–C(11)	104·7 (2)
C(12)–C(11)–C(1)	109·1 (2)	C(4)–C(3)–C(2)	111·4 (2)
C(4a)–C(4)–C(3)	111·0 (2)	C(5a)–C(4a)–C(4)	110·9 (1)
C(10a)–C(4a)–C(4)	111·8 (1)	C(10a)–C(4a)–C(5a)	110·8 (1)
C(5)–C(5a)–C(4a)	118·9 (2)	C(8a)–C(5a)–C(4a)	122·0 (2)
C(10)–C(10a)–C(4a)	108·9 (1)	C(13)–C(10a)–C(4a)	108·1 (1)
C(6)–C(5)–C(5a)	121·4 (2)	C(8a)–C(5a)–C(5)	119·1 (2)
C(7)–C(6)–C(5)	119·2 (2)	C(8)–C(8a)–C(5a)	119·1 (2)
C(9)–C(8a)–C(5a)	121·9 (2)	C(8)–C(7)–C(6)	120·2 (2)
C(8a)–C(8)–C(7)	121·0 (2)	C(9)–C(8a)–C(8)	119·0 (2)
C(10)–C(9)–C(8a)	114·1 (2)	C(10a)–C(10)–C(9)	111·2 (2)
C(13)–C(10a)–C(10)	113·0 (2)	C(12)–C(13)–C(10a)	101·8 (1)
C(13)–C(12)–C(11)	102·6 (2)	C(14)–C(12)–C(11)	108·1 (2)
C(14)–C(12)–C(13)	106·6 (2)	C(15)–C(14)–C(12)	114·8 (2)

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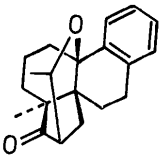
(122 and $21\bar{2}$) monitored periodically showed no significant variation in intensity. No absorption correction. Lorentz–polarization correction. Structure determined by direct phasing using *MITHRIL* (Gilmore, 1984). H atoms located in a difference Fourier synthesis. Full-matrix least-squares calculations on F with *SHELX*76 (Sheldrick, 1976); anisotropic thermal parameters for C and O atoms and isotropic for H. Convergence at $R = 0{\cdot}036$, $wR = 0{\cdot}038$, $\Delta/\sigma < 0{\cdot}1$, with $w = 1/\sigma^2(|F_o|)$. Final $\Delta\rho$ max. 0·15, min. $-0{\cdot}18$ e Å^{-3}. Scattering factors for C, H and O atoms were those incorporated in *SHELX*76.



(I)

Atomic coordinates are listed in Table 1 and molecular dimensions in Table 2.* Fig. 1, drawn with *ORTEP* (Johnson, 1965), illustrates the molecular structure.

* Lists of structure factors, torsional angles, anisotropic thermal parameters of the C and O atoms, positional and thermal parameters of the H atoms, and bond lengths involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43908 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

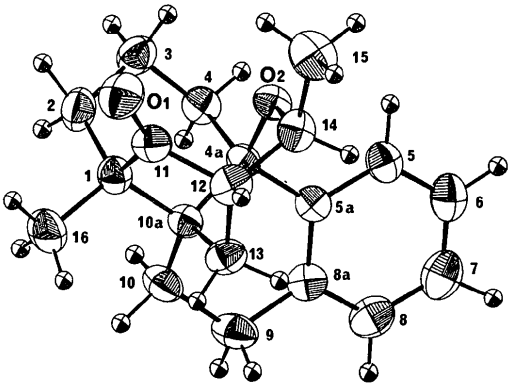


Fig. 1. Molecular structure and atomic numbering (Ghatak *et al.*, 1980). The thermal ellipsoids of the C and O atoms are drawn at the 50% probability level and the H atoms are represented by spheres of radius 0·1 Å.

Related literature. For the preparation of the compound see Ghatak, Sanyal, Ghosh, Sarkar, Raju & Wenkert (1980).

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Structure of 3,3,5,5,7,7-Hexaphenyl-1,2,4,6,3,5,7-tetraazatriphosphepine Hydrochloride Monohydrate

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Abstract. $C_{36}H_{32}N_4P_3^+.Cl^-.H_2O$, $NHNHPPh_2NPPh_2$-$NPPh_2^+.Cl^-.H_2O$, $M_r = 667{\cdot}1$, monoclinic, $P2_1/n$, $a = 8{\cdot}646$ (4), $b = 20{\cdot}969$ (4), $c = 19{\cdot}276$ (3) Å, $\beta = 96{\cdot}14$ (3)°, $V = 3475$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}27$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 2{\cdot}77$ cm^{-1}, $F(000) = 1392$, $T = 293$ K, $R = 0{\cdot}047$ for 3805 unique observed reflections. The highly puckered P_3N_4 ring has P–N distances of 1·568 (2) to 1·659 (2) Å, N–N of

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