were placed in geometrically calculated positions (C—H 0·96 Å); refinement on F with 2057 reflections and 281 refined parameters; $w = 1{\cdot}0/[\sigma^2(F) + (0{\cdot}001F^2)]$ which led to featureless analysis of variance in terms of $\sin\theta$ and F_o; $S = 2{\cdot}49$, $R = 0{\cdot}085$, $wR = 0{\cdot}091$ (for all data $R = 0{\cdot}162$, $wR = 0{\cdot}137$), $(\Delta/\sigma)_{\max} = 0{\cdot}004$, no extinction correction; largest peak in final ΔF map $\pm 0{\cdot}3\,(2)$ e Å^{-3}; atomic scattering factors for neutral atoms and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974); the weakly reflecting crystal is the reason for the high R value; programs: *PARST* (Nardelli, 1983), *SHELXTL-Plus* (Sheldrick, 1987), *PCK*83 (Williams, 1984), *PLATON* (Spek, 1982), *MISSYM* (Le Page, 1987). The molecule and the numbering scheme are shown in Fig. 1 and a stereoscopic view in Fig. 2. Positional parameters and the equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1.* Bond lengths, bond angles, torsion angles, least-squares planes and dihedral angles are given in Table 2.

Related literature. Cram & Trueblood (1981) and Maverick, Seiler, Schweizer & Dunitz (1980).

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* 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 52633 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 5-Hydroxy-2-methyl-3-[1,2,3,4-tetrahydro-2-(1,4-dihydro-5-hydroxy-2-methyl-1,4-dioxo-3-naphthyl)-5-hydroxy-2-methyl-1,4-dioxo-3-naphthylmethyl]-1,4-napthoquinone Toluene Solvate*

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Abstract. $C_{34}H_{24}O_9.C_7H_8$, $M_r = 668{\cdot}7$, triclinic, $P\bar{1}$, $a = 9{\cdot}489\,(6)$, $b = 13{\cdot}020\,(13)$, $c = 14{\cdot}773\,(22)$ Å, $\alpha = 96{\cdot}22\,(10)$, $\beta = 98{\cdot}60\,(9)$, $\gamma = 113{\cdot}15\,(6)°$, $V = 1631{\cdot}0$ Å^3, $Z = 2$, $D_x = 1{\cdot}36$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}51$ cm^{-1}, $F(000) = 700$, $T = 293$ K, $R = 0{\cdot}069$ for 4844 observed reflexions. The structure of the natural product consists of three plumbagin moieties plus an additional C atom. Two plumbagin units in the trimeric structure are inclined at 80° to each other about the C(9)—C(11) single bond. The third unit is linked in a *trans* arrangement to C(8) *via* a methylene group. The recrystallizing solvent, toluene, has been incorporated into the crystal.

Experimental. Red crystal, 1·10 × 1·10 × 0·64 mm. Nicolet *P*3 automated diffractometer, monochromated Mo $K\alpha$ radiation. Cell dimensions from setting angles of 12 independent reflexions with $2\theta \simeq 20°$. Data corrected for Lorentz and polarization effects, absorption ignored. 7930 unique intensities were measured with $2\theta \le 60°$ with ω–2θ scans; 4844 reflexions had $F > 5\sigma(F)$. Range of hkl:

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* Plumbazeylanone toluene solvate.

Table 1. *Fractional coordinates with e.s.d.'s and equivalent values of the anisotropic temperature factor coefficients*

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
O(1)	−0·2092 (3)	0·1277 (2)	−0·0090 (2)	0·071
O(2)	−0·2901 (4)	−0·1647 (2)	0·2813 (2)	0·096
O(3)	−0·3232 (4)	0·0235 (2)	0·3198 (2)	0·096
O(4)	−0·2000 (4)	0·5628 (2)	0·0673 (2)	0·093
O(5)	0·0425 (3)	0·2735 (2)	0·1473 (2)	0·058
O(6)	0·3226 (3)	0·4309 (3)	0·1882 (2)	0·083
O(7)	0·0213 (3)	0·4053 (2)	0·3382 (2)	0·070
O(8)	−0·1758 (5)	0·2767 (3)	0·6421 (2)	0·150
O(9)	0·3051 (4)	0·5209 (3)	0·4324 (3)	0·112
C(1)	−0·2084 (4)	0·0065 (3)	0·0982 (2)	0·048
C(2)	−0·1755 (4)	−0·0651 (3)	0·0371 (3)	0·059
C(3)	−0·1769 (5)	−0·1656 (3)	0·0598 (3)	0·071
C(4)	−0·2157 (5)	−0·1979 (3)	0·1413 (3)	0·073
C(5)	−0·2512 (4)	−0·1278 (3)	0·2029 (3)	0·065
C(6)	−0·2441 (4)	−0·0233 (3)	0·1834 (2)	0·052
C(7)	−0·2709 (4)	0·0545 (3)	0·2512 (3)	0·059
C(8)	−0·2229 (4)	0·1758 (3)	0·2382 (2)	0·047
C(9)	−0·2696 (4)	0·1831 (3)	0·1339 (2)	0·045
C(10)	−0·2198 (4)	0·1092 (3)	0·0692 (2)	0·049
C(11)	−0·1860 (4)	0·3067 (3)	0·1213 (2)	0·046
C(12)	−0·2476 (4)	0·3801 (3)	0·0993 (2)	0·055
C(13)	−0·1426 (5)	0·5001 (3)	0·0927 (2)	0·060
C(14)	0·0286 (4)	0·5399 (3)	0·1189 (2)	0·055
C(15)	0·1250 (5)	0·6523 (3)	0·1226 (3)	0·072
C(16)	0·2850 (6)	0·6882 (4)	0·1499 (3)	0·083
C(17)	0·3503 (5)	0·6154 (4)	0·1731 (3)	0·079
C(18)	0·2536 (4)	0·5005 (3)	0·1671 (3)	0·062
C(19)	0·0916 (4)	0·4629 (3)	0·1413 (2)	0·049
C(20)	−0·0123 (4)	0·3440 (3)	0·1368 (2)	0·047
C(21)	−0·2804 (4)	0·2436 (3)	0·3042 (2)	0·058
C(22)	−0·1806 (4)	0·2840 (3)	0·4016 (2)	0·058
C(23)	−0·2335 (5)	0·2539 (3)	0·4789 (3)	0·075
C(24)	−0·1259 (7)	0·2990 (4)	0·5715 (3)	0·095
C(25)	0·0414 (6)	0·3702 (4)	0·5776 (3)	0·089
C(26)	0·1475 (9)	0·4016 (5)	0·6606 (3)	0·127
C(27)	0·3040 (10)	0·4685 (6)	0·6650 (4)	0·152
C(28)	0·3549 (7)	0·5078 (5)	0·5892 (5)	0·131
C(29)	0·2477 (7)	0·4787 (4)	0·5047 (4)	0·097
C(30)	0·0924 (5)	0·4083 (3)	0·4981 (3)	0·072
C(31)	0·0194 (5)	0·3684 (3)	0·4087 (2)	0·061
C(32)	−0·4506 (4)	0·1260 (3)	0·1030 (3)	0·060
C(33)	−0·4186 (5)	0·3595 (4)	0·0789 (3)	0·087
C(34)	−0·3991 (6)	0·1771 (4)	0·4796 (3)	0·109
C(35)	0·1802 (9)	1·0016 (6)	0·3818 (5)	0·156
C(36)	0·1963 (9)	0·9738 (9)	0·2969 (6)	0·203
C(37)	0·2112 (7)	1·0099 (8)	0·2278 (4)	0·151
C(38)	0·2236 (9)	1·1298 (7)	0·2471 (6)	0·156
C(39)	0·2116 (8)	1·1798 (7)	0·3285 (6)	0·150
C(40)	0·1876 (10)	1·1138 (11)	0·3969 (5)	0·205
C(41)	0·1781 (12)	1·1490 (12)	0·4776 (7)	0·311

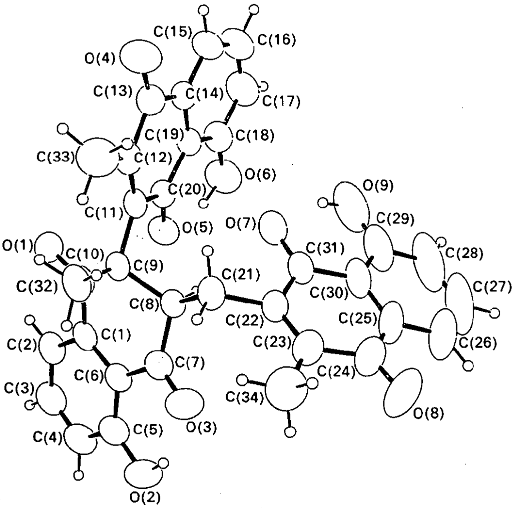

Fig. 1. The atomic arrangement in the molecule.

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

O(1)—C(10)	1·217 (5)	O(2)—C(5)	1·348 (6)
O(3)—C(7)	1·232 (5)	O(4)—C(13)	1·207 (5)
O(5)—C(20)	1·231 (4)	O(6)—C(18)	1·347 (5)
O(7)—C(31)	1·245 (5)	O(8)—C(24)	1·226 (7)
O(9)—C(29)	1·343 (8)	C(1)—C(2)	1·382 (5)
C(1)—C(6)	1·408 (5)	C(1)—C(10)	1·485 (5)
C(2)—C(3)	1·381 (6)	C(3)—C(4)	1·372 (7)
C(4)—C(5)	1·388 (6)	C(5)—C(6)	1·399 (5)
C(6)—C(7)	1·467 (5)	C(7)—C(8)	1·506 (5)
C(8)—C(9)	1·561 (5)	C(8)—C(21)	1·542 (5)
C(9)—C(10)	1·535 (5)	C(9)—C(11)	1·535 (5)
C(9)—C(32)	1·549 (5)	C(11)—C(12)	1·346 (5)
C(11)—C(20)	1·497 (5)	C(12)—C(13)	1·509 (5)
C(12)—C(33)	1·512 (6)	C(13)—C(14)	1·472 (6)
C(14)—C(15)	1·376 (6)	C(14)—C(19)	1·399 (5)
C(15)—C(16)	1·380 (7)	C(16)—C(17)	1·367 (7)
C(17)—C(18)	1·399 (6)	C(18)—C(19)	1·393 (6)
C(19)—C(20)	1·460 (5)	C(21)—C(22)	1·510 (5)
C(22)—C(23)	1·350 (6)	C(22)—C(31)	1·471 (6)
C(23)—C(24)	1·481 (6)	C(23)—C(34)	1·497 (7)
C(24)—C(25)	1·472 (8)	C(25)—C(26)	1·372 (8)
C(25)—C(30)	1·403 (6)	C(26)—C(27)	1·38 (2)
C(27)—C(28)	1·36 (1)	C(28)—C(29)	1·397 (9)
C(29)—C(30)	1·377 (8)	C(30)—C(31)	1·462 (6)
C(35)—C(36)	1·32 (2)	C(35)—C(40)	1·43 (2)
C(36)—C(37)	1·17 (2)	C(37)—C(38)	1·51 (2)
C(38)—C(39)	1·35 (2)	C(39)—C(40)	1·38 (2)
C(40)—C(41)	1·26 (2)		

C(2)—C(1)—C(6)	120·0 (3)	C(2)—C(1)—C(10)	119·3 (3)
C(6)—C(1)—C(10)	120·5 (3)	C(1)—C(2)—C(3)	120·1 (4)
C(2)—C(3)—C(4)	120·9 (4)	C(3)—C(4)—C(5)	119·8 (4)
O(2)—C(5)—C(4)	117·0 (4)	O(2)—C(5)—C(6)	122·5 (4)
C(4)—C(5)—C(6)	120·4 (4)	C(1)—C(6)—C(5)	118·7 (4)
C(1)—C(6)—C(7)	120·7 (3)	C(5)—C(6)—C(7)	120·6 (4)
O(3)—C(7)—C(6)	121·1 (4)	O(3)—C(7)—C(8)	120·6 (4)
C(6)—C(7)—C(8)	118·2 (3)	C(7)—C(8)—C(9)	111·5 (3)
C(7)—C(8)—C(21)	113·0 (3)	C(9)—C(8)—C(21)	112·7 (3)
C(8)—C(9)—C(10)	111·5 (3)	C(8)—C(9)—C(11)	109·0 (3)
C(8)—C(9)—C(32)	108·9 (3)	C(10)—C(9)—C(11)	107·8 (3)
C(10)—C(9)—C(32)	102·3 (3)	C(11)—C(9)—C(32)	117·1 (3)
O(1)—C(10)—C(1)	120·9 (3)	O(1)—C(10)—C(9)	119·3 (3)
C(1)—C(10)—C(9)	119·2 (3)	C(9)—C(11)—C(12)	129·2 (3)
C(9)—C(11)—C(20)	111·5 (3)	C(12)—C(11)—C(20)	119·4 (3)
C(11)—C(12)—C(13)	120·5 (4)	C(11)—C(12)—C(33)	128·0 (4)
C(13)—C(12)—C(33)	111·5 (4)	O(4)—C(13)—C(12)	119·7 (4)
O(4)—C(13)—C(14)	120·7 (4)	C(12)—C(13)—C(14)	119·6 (4)
C(13)—C(14)—C(15)	120·1 (4)	C(13)—C(14)—C(19)	119·2 (3)
C(15)—C(14)—C(19)	120·7 (4)	C(14)—C(15)—C(16)	119·0 (4)
C(15)—C(16)—C(17)	121·8 (4)	C(16)—C(17)—C(18)	119·6 (5)
O(6)—C(18)—C(17)	117·8 (4)	O(6)—C(18)—C(19)	122·8 (4)
C(17)—C(18)—C(19)	119·4 (4)	C(14)—C(19)—C(18)	119·4 (4)
C(14)—C(19)—C(20)	120·0 (4)	C(18)—C(19)—C(20)	120·6 (4)
O(5)—C(20)—C(11)	119·2 (3)	O(5)—C(20)—C(19)	120·3 (3)
C(11)—C(20)—C(19)	120·5 (3)	C(8)—C(21)—C(22)	113·3 (3)
C(21)—C(22)—C(23)	124·4 (4)	C(21)—C(22)—C(31)	114·8 (3)
C(23)—C(22)—C(31)	120·6 (4)	C(22)—C(23)—C(24)	120·2 (5)
C(22)—C(23)—C(34)	124·6 (4)	C(24)—C(23)—C(34)	115·2 (4)
O(8)—C(24)—C(23)	120·1 (5)	O(8)—C(24)—C(25)	120·5 (5)
C(23)—C(24)—C(25)	119·4 (4)	C(24)—C(25)—C(26)	120·8 (5)
C(24)—C(25)—C(30)	119·6 (4)	C(26)—C(25)—C(30)	119·6 (5)
C(25)—C(26)—C(27)	119·9 (6)	C(26)—C(27)—C(28)	121·2 (7)
C(27)—C(28)—C(29)	119·5 (7)	O(9)—C(29)—C(28)	116·7 (6)
O(9)—C(29)—C(30)	123·2 (5)	C(28)—C(29)—C(30)	120·1 (6)
C(25)—C(30)—C(29)	119·6 (5)	C(25)—C(30)—C(31)	119·2 (4)
C(29)—C(30)—C(31)	121·2 (4)	O(7)—C(31)—C(22)	119·6 (4)
O(7)—C(31)—C(30)	120·2 (4)	C(22)—C(31)—C(30)	120·1 (4)
C(36)—C(35)—C(40)	110·6 (8)	C(35)—C(36)—C(37)	140·9 (10)
C(36)—C(37)—C(38)	107·0 (8)	C(37)—C(38)—C(39)	124·3 (8)
C(38)—C(39)—C(40)	115·4 (8)	C(35)—C(40)—C(39)	121·6 (8)
C(35)—C(40)—C(41)	114·9 (11)	C(39)—C(40)—C(41)	123·4 (13)

$-13 < h < 12$, $-18 < k < 17$, $0 < l < 20$. Two reference reflexions monitored periodically showed no significant variation in intensity.

The structure was determined with *MITHRIL* (Gilmore, 1984) and the H atoms were located on difference Fourier maps. The positional parameters of the hydroxy H atoms were refined but other H atoms were only allowed to ride on their attached C atoms with C—H 1·00 (2) Å. Blocked full-matrix least-squares calculations with *SHELX76* (Sheldrick, 1976) on F with anisotropic thermal parameters for C and O atoms and common isotropic thermal parameters for the toluene, methyl and all other H atoms converged at R = 0·069, unit weights. The plumbazeylanone and toluene molecules were refined in alternate least-squares calculations such that the maximum number of parameters refined was 400. The high R value is due to some unresolved disorder, as indicated by high temperature factors and distorted geometries, in the toluene molecule. Atomic scattering factors from *SHELX76*. Final $\Delta/\sigma \leq$ 0·01, final $\Delta\rho$ max. = 0·3, $\Delta\rho$ min. = −0·3 e Å^{-3}. Molecular geometries were generated by the *GX* package (Mallinson & Muir, 1985).

Atomic coordinates are listed in Table 1, bond lengths and valency angles in Table 2.* The atomic arrangement is shown in Fig. 1.

Related literature. The isolation of plumbazeylanone (Gunaherath, Gunatilaka & Thomson, 1984) and a preliminary report of the plumbazeylanone toluene solvate (Gunaherath, Gunatilaka, Cox, Howie & Thomson, 1988) have been published. The crystal structure of plumbagin is also known (Vijayalakshmi, Rajan & Srinivasan, 1987) as are related structures such as 2,5-dihydroxy-3,8-dimethyl-1,4-naphthalenedione (Cowe, Cox, Cordell, Che, Fong, & Howie 1986), 5,8-dihydroxy-2-methyl-1,4-naphthoquinone (Cradwick, Hall & Wood, 1977) and 3-hydroxy-2-methyl-1,4-naphthoquinone (Gaultier & Hauw, 1965).

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* Lists of structure factors, anisotropic thermal parameters, H-atom positions and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52703 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1990). C**46**, 1351–1353

Structure of (±)-(1*R**,4*S**,6*S**)-1-Benzyloxy-4,8,11,11-tetramethyl-6-phenylthiobicyclo[5.3.1]undec-7-en-3-one

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Abstract. $C_{28}H_{34}O_2S$, M_r = 434·64, triclinic, $P\bar{1}$, a = 8·9868 (7), b = 11·2933 (11), c = 12·3497 (6) Å, α = 80·937 (6), β = 73·108 (5), γ = 87·405 (7)°, V = 1184·32 (15) Å^3, Z = 2, D_x = 1·22 g cm^{-3}, μ = 1·509 cm^{-1}, Mo $K\alpha$ radiation, λ = 0·7107 Å, F(000) = 468, T = 298 K, R = 0·0583 for 6924 reflections [$F_o \geq 4\sigma(F_o)$]. The cyclooctane portion of the [5.3.1] ring system is in the boat-chair conformation while the cyclohexene portion assumes the boat conformation. The carbonyl group and the alkene group are nearly parallel in the molecule with a dihedral angle of 14·9 (1)° between planes through the two groups. Ring strain appears to cause distortion in the alkene functionality. The bond length [C7—C8 1·343 (2) Å] is long for an isolated C—C double bond. The torsion angles deviate between 3·7 (2) and 18·39 (14)° (absolute values) from ideality. This twist in the alkene group is reflected in the non-planarity of the group [max. deviation −0·177 (2) Å for C9] and in the dihedral angle between the nearly planar portions (C6,C7,C8,C11 and C7,C8,C9,C16) which is 12·1 (1)°.

Experimental. The bicyclo[5.3.1] undecene (1), which possesses the key structural subunit that is present in the Taxane diterpenes (Martin, White & Wagner, 1982) was obtained by the alkylation of the enolate of (1) with methyl iodide (Martin, White, Wagner,

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