O(126)—C(127)	1.394 (5)	O(226)—C(227)	1.432 (6)
C(128)—O(129)	1.217 (4)	C(228)—O(229)	1.232 (4)
C(128)—C(130)	1.494 (5)	C(228)—C(230)	1.491 (5)
C(130)—C(131)	1.380 (4)	C(230)—C(231)	1.395 (5)
C(130)—C(135)	1.393 (5)	C(230)—C(235)	1.375 (6)
C(131)—C(132)	1.397 (6)	C(231)—C(232)	1.370 (6)
C(132)—C(133)	1.359 (6)	C(232)—C(233)	1.353 (8)
C(133)—C(134)	1.369 (5)	C(233)—C(234)	1.362 (6)
C(134)—C(135)	1.384 (6)	C(234)—C(235)	1.417 (6)

N(102)—S(101)—C(113)	102.5 (1)	N(202)—S(201)—C(213)	100.5 (2)
N(102)—S(101)—O(136)	107.0 (1)	N(202)—S(201)—O(236)	109.0 (2)
N(102)—S(101)—O(137)	107.9 (2)	N(202)—S(201)—O(237)	106.9 (2)
C(113)—S(101)—O(136)	107.2 (2)	C(213)—S(201)—O(236)	107.7 (2)
C(113)—S(101)—O(137)	111.9 (2)	C(213)—S(201)—O(237)	111.6 (2)
O(136)—S(101)—O(137)	119.0 (2)	O(236)—S(201)—O(237)	119.5 (2)
S(101)—N(102)—C(103)	114.1 (2)	S(201)—N(202)—C(203)	115.2 (3)
S(101)—N(102)—C(138)	121.6 (2)	S(201)—N(202)—C(238)	121.7 (2)
C(103)—N(102)—C(138)	121.9 (3)	C(203)—N(202)—C(238)	122.5 (3)
N(102)—C(103)—C(104)	124.0 (3)	N(202)—C(203)—C(204)	124.3 (3)
N(102)—C(103)—C(107)	123.2 (3)	N(202)—C(203)—C(207)	123.4 (3)
C(104)—C(103)—C(107)	112.7 (3)	C(203)—C(204)—C(207)	112.1 (3)
C(103)—C(104)—N(105)	98.5 (2)	C(203)—C(204)—N(205)	98.4 (3)
C(103)—C(104)—C(120)	118.1 (2)	C(203)—C(204)—C(220)	117.5 (2)
N(105)—C(104)—C(120)	114.0 (2)	N(205)—C(204)—C(220)	115.3 (2)
C(104)—N(105)—N(106)	111.4 (2)	C(204)—N(205)—N(206)	111.8 (2)
C(104)—N(105)—C(128)	118.7 (3)	C(204)—N(205)—C(228)	119.6 (3)
N(106)—N(105)—C(128)	120.7 (3)	N(206)—N(205)—C(228)	120.3 (3)
N(105)—N(106)—C(107)	103.1 (2)	N(205)—N(206)—C(207)	102.8 (2)
N(105)—N(106)—C(114)	114.0 (2)	N(205)—N(206)—C(214)	113.0 (2)
C(107)—N(106)—C(114)	114.8 (2)	C(207)—N(206)—C(214)	116.0 (2)
C(103)—C(107)—N(106)	111.4 (2)	C(203)—C(207)—N(206)	111.5 (3)
C(103)—C(107)—C(108)	125.4 (3)	C(203)—C(207)—C(208)	124.7 (3)
N(106)—C(107)—C(108)	122.6 (3)	N(206)—C(207)—C(208)	123.2 (3)
C(107)—C(108)—C(109)	123.7 (3)	C(207)—C(208)—C(209)	123.6 (3)
C(107)—C(108)—C(113)	117.3 (3)	C(207)—C(208)—C(213)	115.9 (4)
C(109)—C(108)—C(113)	118.5 (3)	C(209)—C(208)—C(213)	120.1 (3)
C(108)—C(109)—C(110)	119.2 (3)	C(208)—C(209)—C(210)	119.5 (3)
C(109)—C(110)—C(111)	121.1 (4)	C(209)—C(210)—C(211)	120.9 (4)
C(110)—C(111)—C(112)	120.8 (3)	C(210)—C(211)—C(212)	121.2 (4)
C(111)—C(112)—C(113)	118.1 (3)	C(211)—C(212)—C(213)	119.3 (3)
S(101)—C(113)—C(108)	117.0 (2)	S(201)—C(213)—C(208)	120.3 (3)
S(101)—C(113)—C(112)	120.7 (3)	S(201)—C(213)—C(212)	120.8 (3)
C(108)—C(113)—C(112)	122.2 (4)	C(208)—C(213)—C(212)	118.8 (4)
N(106)—C(114)—C(115)	121.6 (3)	N(206)—C(214)—C(215)	121.4 (3)
N(106)—C(114)—C(119)	119.4 (3)	N(206)—C(214)—C(219)	119.2 (2)
C(115)—C(114)—C(119)	118.9 (3)	C(215)—C(214)—C(219)	119.2 (3)
C(114)—C(115)—C(116)	120.1 (3)	C(214)—C(215)—C(216)	119.7 (3)
C(115)—C(116)—C(117)	120.1 (3)	C(215)—C(216)—C(217)	120.7 (3)
C(116)—C(117)—C(118)	120.0 (4)	C(216)—C(217)—C(218)	119.7 (3)
C(117)—C(118)—C(119)	120.1 (4)	C(217)—C(218)—C(219)	120.7 (3)
C(114)—C(119)—C(118)	120.7 (3)	C(214)—C(219)—C(218)	119.9 (3)
C(104)—C(120)—C(121)	122.1 (3)	C(204)—C(220)—C(221)	123.8 (3)
C(104)—C(120)—C(125)	119.6 (3)	C(204)—C(220)—C(225)	117.7 (3)
C(121)—C(120)—C(125)	118.2 (3)	C(221)—C(220)—C(225)	118.5 (3)
C(120)—C(121)—C(122)	121.2 (3)	C(220)—C(221)—C(222)	121.0 (4)
C(121)—C(122)—C(123)	119.5 (3)	C(221)—C(222)—C(223)	120.0 (3)
C(122)—C(123)—C(124)	120.1 (4)	C(222)—C(223)—C(224)	120.0 (3)
C(122)—C(123)—O(126)	124.4 (3)	C(222)—C(223)—O(226)	116.0 (3)
C(124)—C(123)—O(126)	115.5 (3)	C(224)—C(223)—O(226)	124.0 (4)
C(123)—C(124)—C(125)	120.1 (4)	C(223)—C(224)—C(225)	119.0 (4)
C(120)—C(125)—C(124)	120.8 (3)	C(220)—C(225)—C(224)	121.5 (3)
C(123)—O(126)—C(127)	119.1 (3)	C(223)—O(226)—C(227)	116.7 (3)
N(105)—C(128)—O(129)	120.0 (3)	N(205)—C(228)—O(229)	119.2 (3)
N(105)—C(128)—C(130)	119.1 (3)	N(205)—C(228)—C(230)	120.2 (3)
O(129)—C(128)—C(130)	120.7 (3)	O(229)—C(228)—C(230)	120.6 (2)
C(128)—C(130)—C(131)	117.9 (3)	C(228)—C(230)—C(231)	118.6 (3)
C(128)—C(130)—C(135)	122.0 (3)	C(228)—C(230)—C(235)	120.7 (3)
C(131)—C(130)—C(135)	119.9 (3)	C(231)—C(230)—C(235)	120.4 (3)
C(130)—C(131)—C(132)	119.8 (3)	C(230)—C(231)—C(232)	119.9 (4)
C(131)—C(132)—C(133)	119.7 (3)	C(231)—C(232)—C(233)	119.9 (4)
C(132)—C(133)—C(134)	120.9 (4)	C(232)—C(233)—C(234)	122.0 (4)
C(133)—C(134)—C(135)	120.5 (4)	C(233)—C(234)—C(235)	119.3 (5)
C(130)—C(135)—C(134)	119.1 (3)	C(230)—C(235)—C(234)	118.5 (3)

The structure was solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Other programs used were *ORTEP* (Johnson, 1976) and *SDP* (B. A. Frenz & Associates Inc., 1982). For H atoms, B was chosen as equal to 1.30 × B_{eq} of the neighbouring heavy atom.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and geometry involving H atoms, least-squares-planes data and equations, dihedral angles between planes and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71671 (47 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1044]

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A New Quassinoid Isolated from *Picrolemma pseudocoffea*

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Abstract

The rings in 15β-(acetyloxy)-13,20-epoxy-11β,12α-dihydroxy-16,2′-dioxo-2,2′-olide-picras-1(1′),2-dien-21-oic acid methyl ester, $C_{25}H_{28}O_{11}.H_2O$ (systematic name: methyl 4β-acetyloxy-14β,15α-dihydroxy-8α,12β-dimethyl-5,11-dioxo-3,3a,4,5,6a,7,7a,8,12b,13-decahydro-3,13-ethano-1*H*,11*H*-furo[3,4-*c*]furo-

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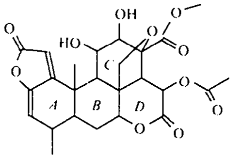
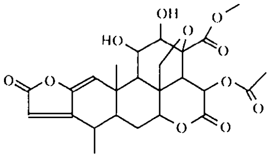
[3′,2′:5,6]naphtho[2,3-*b*]pyran-3-carboxylate monohydrate) have all-*trans* ring fusion. The carbomethoxy and the acetyloxy moieties are each in a pseudo-equatorial position and the hydroxyl groups are *trans* to each other. In the picrasane skeleton the methyl group at C(4) is in an α-configuration and the γ-lactone ring is *cis* with respect to the methyl group at C(10).

Comment

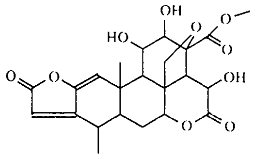
Picrolemma pseudocoffea Ducke is a tropical Simaroubaceae found in the Amazon forest. In an earlier study of specimens of this plant collected in French Guyana, two new potent cytotoxic quassinoids, namely sergeolide (II) and 15-deacetylsergeolide (III), were identified (Moretti, Polonsky, Vuilhorgne & Prange, 1982; Polonsky, Bhatnagar & Moretti, 1984). From the dichloromethane extract of the stem of *P. pseudocoffea* collected in the Brazilian Amazon forest, a new substance (I), named neosergeolide, was isolated. This compound was barely soluble in $CDCl_3$ and for that reason the NMR spectra were measured in a pyridine-d_5 solution; this prevented the determination of the stereochemical formula and therefore a three-dimensional crystal structure study was undertaken.



(I)



(II)



(III)

The ring conformations are as follows: *A* sofa, *B* chair, *C* distorted chair and *D* half-boat, as indicated by the puckering parameters (Cremer & Pople, 1975) shown in Table 2. As in other quassinoids (Polonsky, 1985) the hydroxyl groups are *trans* to each other. The water molecule is involved in several hydrogen bonds: $O(2^{i})\cdots Ow = 2.853\ (4)$, $O(2^{i})\cdots H(Ow) = 1.936\ (3)$ Å, $Ow—H(Ow)\cdots O(2^{i}) = 160.3\ (2)^{\circ}$ [symmetry code: (i) $1-x,\ -\frac{1}{2}+y,\ 1-z$]; $O(5^{ii})\cdots Ow = 2.915\ (4)$, $O(5^{ii})\cdots H'(Ow) = 2.091\ (2)$ Å, $Ow—H'(Ow)\cdots O(5^{ii}) = 149.4\ (2)^{\circ}$ [symmetry code: (ii) $-x,\ \frac{1}{2}+y,\ 1-z$]; $O(8)\cdots Ow^{iii} = 2.841\ (4)$, $Ow^{iii}\cdots H(O8) = 2.102\ (3)$ Å, $O(8)—H(O8)\cdots Ow^{iii} = 174.6\ (2)^{\circ}$ [symmetry code: (iii) $-x,\ \frac{1}{2}+y,\ 1-z$].

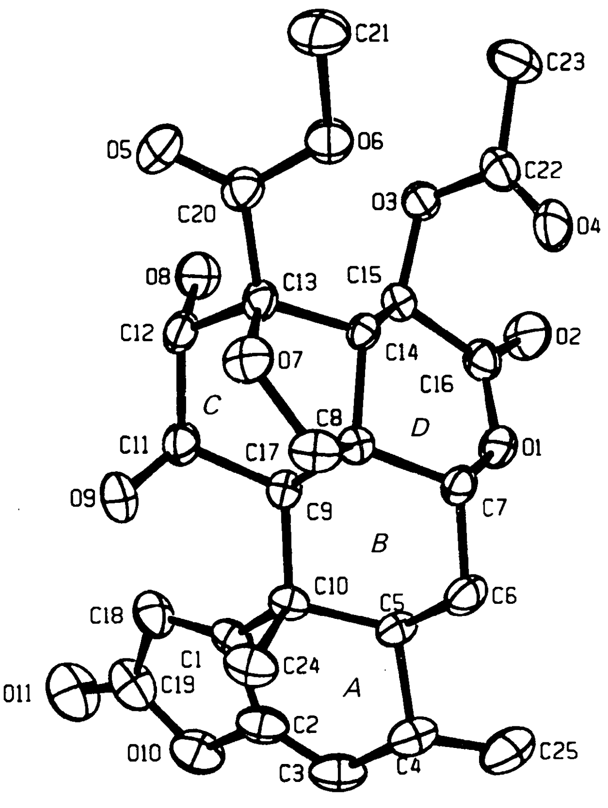


Fig. 1. *ORTEP* (Johnson, 1965) view of the structure of $C_{25}H_{28}O_{11}.H_2O$ showing the atom labelling. Displacement ellipsoids are shown at the 50% probability level.

Experimental

Crystal data

$C_{25}H_{28}O_{11}.H_2O$
$M_r = 522.50$
Monoclinic
$P2_1$
$a = 8.372\ (1)$ Å
$b = 6.257\ (2)$ Å
$c = 22.299\ (2)$ Å
$\beta = 99.44\ (1)^{\circ}$
$V = 1152.2\ (6)$ Å^3
$Z = 2$
$D_x = 1.50$ Mg m^{-3}

Mo *K*α radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9\text{–}21^{\circ}$
$\mu = 0.113$ mm^{-1}
$T = 292$ K
Irregular
0.45 × 0.25 × 0.18 mm
Colourless
Crystal source: from dichloromethane/methanol

Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{min} = 0.80$, $T_{max} = 1.13$
3476 measured reflections
3398 independent reflections

2657 observed reflections [$I > 3\sigma(I)$]
$R_{int} = 0.011$
$\theta_{max} = 30^{\circ}$
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 31$
1 standard reflection
frequency: 30 min
intensity variation: ±0.8%

Refinement

Refinement on F
$R = 0.045$
$wR = 0.049$
$S = 1.80$
2657 reflections
335 parameters
$w = 1/[\sigma^2(|F_o|) + 0.0003|F_o|^2]$
$(\Delta/\sigma)_{\max} = 0.007$
$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *SHELX76* (Sheldrick, 1976)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
O(1)	0.1203 (2)	0.3782 (5)	0.2242 (1)	2.75 (6)
O(2)	0.2415 (3)	0.5885 (5)	0.2954 (1)	3.83 (8)
O(3)	0.1588 (2)	0.3245 (4)	0.3864 (1)	2.31 (6)
O(4)	0.3431 (2)	0.1466 (5)	0.3442 (1)	3.48 (7)
O(5)	−0.1911 (3)	−0.1317 (6)	0.4299 (1)	3.94 (8)
O(6)	0.0615 (3)	−0.1282 (5)	0.4113 (1)	3.35 (7)
O(7)	−0.1826 (2)	−0.1660 (4)	0.2955 (1)	2.64 (6)
O(8)	−0.2414 (2)	0.3298 (5)	0.3732 (1)	3.15 (7)
O(9)	−0.4763 (2)	0.0763 (6)	0.2412 (1)	3.97 (8)
O(10)	−0.5903 (3)	0.7045 (5)	0.0771 (1)	3.63 (7)
O(11)	−0.7726 (3)	0.7952 (6)	0.1356 (1)	4.77 (9)
C(1)	−0.4326 (3)	0.4776 (5)	0.1447 (1)	2.30 (8)
C(2)	−0.4506 (4)	0.5799 (6)	0.0853 (1)	2.95 (9)
C(3)	−0.3559 (4)	0.5586 (7)	0.0442 (1)	3.6 (1)
C(4)	−0.2041 (4)	0.4271 (7)	0.0571 (1)	3.03 (9)
C(5)	−0.1575 (3)	0.3794 (6)	0.1261 (1)	2.22 (7)
C(6)	−0.0210 (4)	0.2146 (6)	0.1366 (1)	2.61 (8)
C(7)	0.0392 (3)	0.1752 (6)	0.2028 (1)	2.14 (8)
C(8)	−0.0917 (3)	0.1203 (5)	0.2399 (1)	1.82 (7)
C(9)	−0.2378 (3)	0.2770 (5)	0.2269 (1)	1.78 (7)
C(10)	−0.3034 (3)	0.3063 (5)	0.1574 (1)	1.98 (7)
C(11)	−0.3640 (3)	0.2296 (6)	0.2686 (1)	2.51 (8)
C(12)	−0.2897 (3)	0.1569 (6)	0.3339 (1)	2.48 (8)
C(13)	−0.1419 (3)	0.0150 (5)	0.3352 (1)	2.01 (7)
C(14)	−0.0100 (3)	0.1204 (5)	0.3065 (1)	1.68 (6)
C(15)	0.0584 (3)	0.3368 (5)	0.3274 (1)	1.95 (7)
C(16)	0.1513 (3)	0.4397 (6)	0.2819 (1)	2.44 (8)
C(17)	−0.1427 (3)	−0.1146 (6)	0.2364 (1)	2.48 (8)
C(18)	−0.5529 (4)	0.5476 (7)	0.1719 (2)	3.07 (9)
C(19)	−0.6535 (4)	0.6943 (6)	0.1307 (2)	3.4 (1)
C(20)	−0.0954 (3)	−0.0863 (6)	0.3978 (1)	2.42 (8)
C(21)	0.1134 (5)	−0.2480 (8)	0.4667 (2)	4.1 (1)
C(22)	0.2988 (3)	0.2181 (6)	0.3889 (1)	2.53 (8)
C(23)	0.3889 (4)	0.2023 (8)	0.4518 (2)	4.1 (1)
C(24)	−0.3817 (4)	0.1074 (6)	0.1239 (1)	2.91 (9)
C(25)	−0.0692 (5)	0.5480 (9)	0.0331 (2)	4.4 (1)
Ow	0.4871 (3)	0.1258 (5)	0.6094 (1)	4.77 (9)

Table 2. *Selected geometric and puckering parameters* (Å, °)

O(1)—C(7)	1.482 (4)	O(1)—C(16)	1.327 (4)
O(2)—C(16)	1.205 (4)	O(3)—C(15)	1.442 (3)
O(3)—C(22)	1.341 (4)	O(4)—C(22)	1.205 (4)
O(5)—C(20)	1.193 (4)	O(6)—C(20)	1.325 (4)
O(6)—C(21)	1.449 (5)	O(7)—C(13)	1.443 (4)
O(7)—C(17)	1.448 (3)	O(8)—C(12)	1.409 (4)
O(9)—C(11)	1.410 (4)	O(10)—C(2)	1.392 (4)
O(10)—C(19)	1.386 (4)	O(11)—C(19)	1.200 (5)
C(1)—C(2)	1.456 (4)	C(1)—C(10)	1.516 (4)
C(1)—C(18)	1.332 (4)	C(2)—C(3)	1.313 (5)
C(3)—C(4)	1.502 (5)	C(4)—C(5)	1.553 (4)
C(4)—C(25)	1.527 (6)	C(5)—C(6)	1.528 (5)
C(5)—C(10)	1.571 (4)	C(6)—C(7)	1.500 (4)
C(7)—C(8)	1.517 (4)	C(8)—C(9)	1.557 (4)
C(8)—C(14)	1.530 (3)	C(8)—C(17)	1.529 (5)
C(9)—C(10)	1.568 (4)	C(9)—C(11)	1.546 (4)
C(10)—C(24)	1.541 (5)	C(11)—C(12)	1.555 (4)
C(12)—C(13)	1.519 (4)	C(13)—C(14)	1.515 (4)
C(13)—C(20)	1.524 (4)	C(14)—C(15)	1.514 (4)
C(15)—C(16)	1.519 (4)	C(18)—C(19)	1.464 (5)
C(22)—C(23)	1.483 (5)		
C(7)—O(1)—C(16)	124.6 (2)	C(1)—C(10)—C(5)	107.1 (2)
C(20)—O(6)—C(21)	116.3 (3)	C(1)—C(10)—C(24)	104.3 (2)
C(2)—O(10)—C(19)	107.0 (3)	C(5)—C(10)—C(24)	108.6 (2)
C(2)—C(1)—C(18)	107.2 (3)	O(9)—C(11)—C(9)	110.0 (2)
O(10)—C(2)—C(1)	109.0 (3)	C(9)—C(11)—C(12)	114.3 (2)
C(1)—C(2)—C(3)	127.3 (3)	O(8)—C(12)—C(11)	112.8 (3)
C(3)—C(4)—C(5)	111.2 (3)	C(11)—C(12)—C(13)	112.8 (3)
C(5)—C(4)—C(25)	111.2 (3)	O(7)—C(13)—C(14)	101.4 (2)
C(4)—C(5)—C(10)	114.1 (2)	C(12)—C(13)—C(14)	112.7 (2)
C(5)—C(6)—C(7)	112.6 (3)	C(14)—C(13)—C(20)	118.3 (2)
O(1)—C(7)—C(8)	110.6 (2)	C(8)—C(14)—C(15)	112.7 (2)
C(7)—C(8)—C(9)	111.8 (2)	O(3)—C(15)—C(14)	111.6 (2)
C(7)—C(8)—C(17)	114.2 (2)	C(14)—C(15)—C(16)	112.5 (2)
C(9)—C(8)—C(17)	113.0 (2)	O(1)—C(16)—C(15)	119.0 (3)
C(8)—C(9)—C(10)	113.2 (2)	O(7)—C(17)—C(8)	105.7 (2)
C(10)—C(9)—C(11)	117.0 (2)	O(10)—C(19)—O(11)	119.7 (3)
C(1)—C(10)—C(9)	113.0 (2)	O(11)—C(19)—C(18)	132.4 (4)
C(5)—C(10)—C(9)	107.5 (2)	O(5)—C(20)—C(13)	123.6 (3)
C(9)—C(10)—C(24)	115.9 (2)	O(3)—C(22)—O(4)	122.4 (3)
O(9)—C(11)—C(12)	110.0 (3)	O(4)—C(22)—C(23)	125.4 (3)
C(15)—O(3)—C(22)	116.0 (2)	O(8)—C(12)—C(13)	106.7 (2)
C(13)—O(7)—C(17)	108.5 (2)	O(7)—C(13)—C(12)	109.7 (2)
C(2)—C(1)—C(10)	116.4 (3)	O(7)—C(13)—C(20)	103.6 (2)
C(10)—C(1)—C(18)	135.8 (3)	C(12)—C(13)—C(20)	110.2 (2)
O(10)—C(2)—C(3)	123.7 (3)	C(8)—C(14)—C(13)	99.7 (2)
C(2)—C(3)—C(4)	120.7 (3)	C(13)—C(14)—C(15)	121.6 (2)
C(3)—C(4)—C(25)	108.2 (3)	O(3)—C(15)—C(16)	110.3 (2)
C(4)—C(5)—C(6)	110.0 (3)	O(1)—C(16)—O(2)	118.7 (3)
C(6)—C(5)—C(10)	110.6 (2)	O(2)—C(16)—C(15)	121.9 (3)
O(1)—C(7)—C(6)	103.8 (2)	C(1)—C(18)—C(19)	108.7 (3)
C(6)—C(7)—C(8)	114.7 (2)	O(10)—C(19)—C(18)	108.0 (3)
C(7)—C(8)—C(14)	106.3 (2)	O(5)—C(20)—O(6)	123.7 (3)
C(9)—C(8)—C(14)	113.1 (2)	O(6)—C(20)—C(13)	112.6 (3)
C(14)—C(8)—C(17)	97.3 (2)	O(3)—C(22)—C(23)	112.2 (3)
C(8)—C(9)—C(11)	111.1 (2)		

Puckering parameters

Ring	q_2	q_3	Q	θ	φ
A	0.099 (3)	0.076 (3)	0.129 (3)	53 (1)	262 (2)
B	0.027 (3)	0.166 (3)	0.168 (3)	9.2 (9)	329 (6)
C	0.073 (3)	0.163 (3)	0.179 (3)	24.1 (8)	300 (2)
D	0.238 (3)	−0.317 (3)	0.396 (3)	143.1 (4)	312.8 (6)

Data were corrected for Lp effects. The structure was solved by direct methods. H atoms were found by difference Fourier synthesis and were included as fixed contributors with an overall isotropic temperature factor that refined to $U_{iso} = 0.062$ (2) Å^2. Programs used were: *SHELXS86* (Sheldrick, 1985), *SHELX76* (Sheldrick, 1976) and *ORTEP* (Johnson, 1965). The refinement was carried out using a blocked-matrix least-squares method. Most of the calculations were performed on a VAX 6420 computer at the Instituto de Física e Química de São Carlos.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71757 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1066]

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2-Acetoxy-7-methoxynaphthalene

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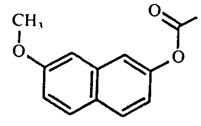
Abstract

The naphthalene ring system of the title compound (7-methoxy-2-naphthyl acetate, $C_{13}H_{12}O_3$) is slightly non-planar, the average deviation being 0.025 (1) Å, with a maximum deviation of 0.041 (1) Å for the C atom carrying the methoxy group. The methoxy group has the methyl *syn* to the neighboring α-C atom, and is nearly coplanar with the ring, with a C—C—O—C torsion angle of −5.2 (2)°. The dihedral angle between the naphthalene ring system and the acetoxyl group is 115.8 (1)°, with a C—C—O—C torsion angle of 67.2 (2)°.

Comment

The title compound, (1), was prepared by acetylation of 7-methoxy-2-naphthol in acetic anhydride (Gorelic, Reznichenko, Andronova & Luk'yanets, 1983), as an intermediate in the synthesis of new binaphthylacetylenes. Crystals of (1), m.p. 397.5–398.5 K, were isolated by slow evaporation of methanol.



(1)

The methoxy O atom is closer to C2 than C10 as indicated by the angle difference of about 5° from the theoretical 120° for O1—C1—C2 and O1—C1—C10 [114.4 (1) and 125.2 (1)°, respectively]. This is explained by steric interaction between the methyl group and H10. This is observed in similar 2-methoxynaphthalene structures (Prince, Fronczek & Gandour, 1989, 1991*a*, 1991*b*). The carbonyl group is displaced from the plane of the naphthalene system to avoid steric interaction with the H atoms on the ring.

A search of the January 1992 version of the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) revealed no compound with an acetoxy substituent on position 2 or position 7 of naphthalene. Structures containing the 2,7-dioxynaphthyl fragment were found (see Prince, Fronczek & Gandour, 1989, 1991*a*, 1991*b*, and references therein; Watson, Nagl & Eduok, 1989).

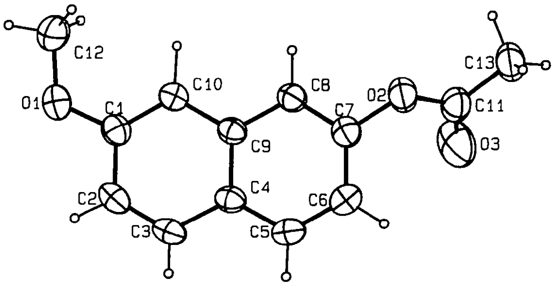


Fig. 1. View of the title compound showing the numbering scheme with displacement ellipsoids drawn at the 40% probability level. H atoms are drawn as circles of arbitrary radii.

Experimental

Crystal data

$C_{13}H_{12}O_3$
$M_r = 216.2$
Orthorhombic
$P2_12_12_1$
$a = 5.8414$ (5) Å
$b = 7.9263$ (10) Å
$c = 23.776$ (4) Å
$V = 1100.8$ (4) Å^3
$Z = 4$
$D_x = 1.305$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 25$–$30°$
$\mu = 0.72$ mm^{-1}
$T = 295$ K
Rectangular prism
0.70 × 0.47 × 0.28 mm
Colorless

Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
$T_{\min} = 0.9499$, $T_{\max} = 0.9983$
2657 measured reflections
2269 independent reflections

2219 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int} = 0.014$
$\theta_{\max} = 75°$ (2 octants)
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 9$
$l = -29 \rightarrow 29$
3 standard reflections
frequency: 166.6 min
intensity variation: <2%

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