

Fig. 1. ORTEP (Johnson, 1976) drawing showing the conformation of a molecule of (*E,E*)-bis(2-acetoxy-1-iodo-2-phenylvinyl) disulfide and the atom-numbering scheme.

Vol. IV). The scattering factor for H atoms is that of Stewart, Davidson & Simpson (1965). Atomic coordinates and the equivalent isotropic thermal parameters are given in Table 1.\* Bond lengths and angles are in Table 2. The structure is shown in Fig. 1. No hydrogen bonding or close approaches to S atoms are found.

\* 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 53588 (15 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 Calliterpenone Hemihydrate

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**Abstract.** 16 $\alpha$ ,17-Dihydroxy-13 $\beta$ -kauran-3-one, C<sub>20</sub>H<sub>32</sub>O<sub>3</sub>· $\frac{1}{2}$ H<sub>2</sub>O, *M*<sub>r</sub> = 329.48, monoclinic, *C*2, *a* = 13.475 (6), *b* = 6.338 (1), *c* = 22.904 (7) Å,  $\beta$  = 114.75 (2)°, *V* = 1776 (1) Å<sup>3</sup>, *Z* = 4, *D*<sub>x</sub> = 1.232 g cm<sup>-3</sup>, graphite-monochromatized Mo *K* $\alpha$  radiation ( $\lambda$  = 0.71069 Å),  $\mu$  = 0.77 cm<sup>-1</sup>, *F*(000) = 723.92, *T* = 295 K, *R* = 0.055 for 1353 observed reflections. The CH<sub>2</sub>OH side chain is found to be in the  $\beta$ -position rather than in the sterically more

**Related literature.** The present compound is quite different from that resulting from an analogous reaction of a chlorinating agent with *S*-(2-phenylvinyl) thioacetate (Pedersen, Hazell & Senning, 1989). Hordvik (1966) gives a correlation between the S—S distance and the dihedral angle around it; the present values of 2.039 (3) Å and 74.1 (3)° agree with this. Distances and angles in the central part of the molecule agree with values found by Jones & Power (1976) in a similar compound.

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favourable  $\alpha$ -position of the diterpenoid molecule. The hydroxyl groups are involved in intermolecular hydrogen bonding with the water molecule of crystallization and also with the neighbouring diterpenoid molecules. No intramolecular hydrogen bonding was observed between O(2) and O(3) of the two hydroxy groups which could form a sterically favoured five-membered ring.

**Experimental.** Calliterpenone was isolated from petroleum ether (60–80°C) extract of *Callicarpa cana*

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Table 1. Data collection and processing parameters

Diffractometer	Nicolet R3m
Crystal size	0.42 × 0.24 × 0.08 mm
Scan type and speed	$\omega$ -2 $\theta$ ; 2.02–8.37° min <sup>-1</sup>
Scan range	1° below $K\alpha_1$ to 1° above $K\alpha_2$
Background counting	Stationary counts for one-half of scan time at each end of scan
Collection range	$h, k, \pm l; h = 0 \rightarrow 16, k = 0 \rightarrow 8, l = -27 \rightarrow 27; 2\theta_{\max} = 48^\circ$
Unique data measured	1481
Observed data	1353, $F_o > 2\sigma(F_o)$
Number of variables	141
$R_f$	0.055
Weighting scheme	$w = [\sigma^2(F_o) + 0.0015 F_o ^2]^{-1}$
$wR$	0.073
$S$	1.382
Residual extrema in final difference map	0.22 to -0.20 e Å <sup>-3</sup>
Least-squares shift/e.s.d.	0.002

Table 2. Atomic coordinates ( $\times 10^4$ ) and temperature factors ( $\text{\AA}^2 \times 10^3$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}/U_{\text{eq}}$
O(1)	2994 (3)	1174 (8)	5048 (1)	59 (1)*
O(2)	-1151 (3)	1179 (6)	359 (1)	43 (1)*
HO(2)	-688	2304	389	60
O(3)	-3194 (2)	2606 (7)	376 (2)	52 (1)*
HO(3)	-3542	3525	7	60
O(4)	0	4608 (9)	0	58 (2)*
HO(4)	539	5719	256	60
C(1)	361 (4)	577 (8)	3669 (2)	40 (1)
C(2)	1143 (3)	1440 (10)	4325 (2)	46 (1)
C(3)	2336 (3)	1212 (8)	4491 (2)	40 (1)
C(4)	2736 (4)	1245 (9)	3953 (2)	42 (1)
C(5)	1841 (3)	506 (8)	3287 (2)	36 (1)
C(6)	2158 (4)	847 (8)	2728 (2)	39 (1)
C(7)	1429 (3)	-468 (8)	2152 (2)	40 (1)
C(8)	209 (3)	0	1919 (2)	31 (1)
C(9)	-119 (3)	5 (8)	2492 (2)	33 (1)
C(10)	648 (3)	1249 (8)	3112 (2)	30 (1)
C(11)	-1339 (3)	594 (8)	2253 (2)	38 (1)
C(12)	-2078 (4)	-526 (9)	1627 (2)	42 (1)
C(13)	-1602 (3)	-620 (8)	1124 (2)	36 (1)
C(14)	-485 (4)	-1673 (9)	1439 (2)	42 (1)
C(15)	-165 (3)	2045 (8)	1505 (2)	34 (1)
C(16)	-1299 (3)	1538 (8)	941 (2)	33 (1)
C(17)	-2132 (4)	3292 (8)	819 (2)	43 (2)*
C(18)	3119 (5)	3520 (13)	3944 (3)	68 (3)*
C(19)	3716 (4)	-233 (15)	4153 (3)	79 (3)*
C(20)	525 (4)	3655 (9)	3030 (2)	40 (2)*

\* Equivalent isotropic  $U_{\text{eq}}$  defined as one-third of the trace of the orthogonized  $U_{ij}$  tensor. The exponent of the isotropic temperature factor takes the form  $-8\pi^2 U \sin^2 \theta / \lambda^2$ .

L. Subsequent column chromatography over silica gel (70–230 mesh) eluting with hexane followed by benzene yielded a white crystalline solid which was recrystallized from aqueous ethanol to yield a solid with m.p. 428–429 K,  $[\alpha]_D^{20} = +29.7$  (in  $\text{CHCl}_3$ ). Crystals suitable for X-ray diffraction were obtained by further recrystallization from ethanol. Intensity data collection and processing followed established procedures in our laboratory (Kumar Das, Chen, Yap & Mak, 1986) and details are summarized in Table 1. A least-squares fit of 18 reflections was used for measuring lattice parameters. Lorentz-polarization effects were corrected. 2 standard

Table 3. Interatomic bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ )

O(1)—C(3)	1.211 (4)	O(2)—C(16)	1.447 (6)
O(3)—C(17)	1.432 (5)	C(1)—C(2)	1.531 (6)
C(1)—C(10)	1.541 (7)	C(2)—C(3)	1.496 (6)
C(3)—C(4)	1.538 (8)	C(4)—C(5)	1.568 (5)
C(4)—C(18)	1.534 (10)	C(4)—C(19)	1.525 (9)
C(5)—C(6)	1.525 (7)	C(5)—C(10)	1.557 (6)
C(6)—C(7)	1.522 (6)	C(7)—C(8)	1.530 (6)
C(8)—C(9)	1.551 (7)	C(8)—C(14)	1.532 (5)
C(8)—C(15)	1.560 (5)	C(9)—C(10)	1.573 (5)
C(9)—C(11)	1.545 (6)	C(10)—C(20)	1.537 (7)
C(11)—C(12)	1.534 (6)	C(12)—C(13)	1.537 (8)
C(13)—C(14)	1.525 (6)	C(13)—C(16)	1.534 (7)
C(15)—C(16)	1.566 (5)	C(16)—C(17)	1.520 (7)
C(2)—C(1)—C(10)	114.0 (4)	C(1)—C(2)—C(3)	116.0 (4)
O(1)—C(3)—C(2)	120.2 (5)	O(1)—C(3)—C(4)	119.8 (4)
C(2)—C(3)—C(4)	119.7 (3)	C(3)—C(4)—C(5)	112.9 (4)
C(3)—C(4)—C(18)	104.9 (5)	C(3)—C(4)—C(19)	113.2 (4)
C(3)—C(4)—C(19)	107.4 (4)	C(5)—C(4)—C(19)	109.0 (5)
C(18)—C(4)—C(19)	109.1 (5)	C(4)—C(5)—C(6)	113.7 (4)
C(4)—C(5)—C(10)	116.8 (4)	C(6)—C(5)—C(10)	111.2 (3)
C(5)—C(6)—C(7)	110.1 (4)	C(6)—C(7)—C(8)	113.9 (4)
C(7)—C(8)—C(9)	110.5 (3)	C(7)—C(8)—C(14)	111.1 (3)
C(9)—C(8)—C(14)	107.7 (4)	C(7)—C(8)—C(15)	113.6 (4)
C(9)—C(8)—C(15)	112.3 (3)	C(14)—C(8)—C(15)	101.2 (3)
C(8)—C(9)—C(10)	116.9 (4)	C(8)—C(9)—C(11)	109.8 (3)
C(10)—C(9)—C(11)	113.5 (4)	C(1)—C(10)—C(5)	107.0 (3)
C(1)—C(10)—C(9)	108.1 (4)	C(5)—C(10)—C(9)	107.4 (4)
C(1)—C(10)—C(20)	108.9 (4)	C(5)—C(10)—C(20)	112.3 (4)
C(9)—C(10)—C(20)	113.0 (3)	C(9)—C(11)—C(12)	113.4 (4)
C(11)—C(12)—C(13)	114.0 (4)	C(12)—C(13)—C(14)	107.5 (4)
C(12)—C(13)—C(16)	114.3 (4)	C(14)—C(13)—C(16)	101.3 (4)
C(8)—C(14)—C(13)	102.8 (4)	C(8)—C(15)—C(16)	106.6 (3)
O(2)—C(16)—C(13)	105.4 (4)	O(2)—C(16)—C(15)	109.2 (4)
C(13)—C(16)—C(15)	103.7 (3)	O(2)—C(16)—C(17)	108.8 (3)
C(13)—C(16)—C(17)	116.2 (4)	C(15)—C(16)—C(17)	113.1 (4)
O(3)—C(17)—C(16)	110.5 (4)		

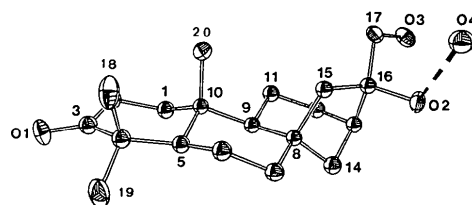


Fig. 1. A perspective view of the title compound.

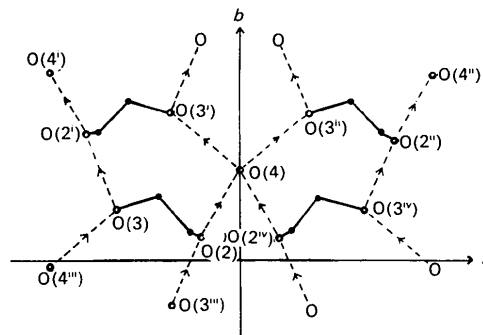


Fig. 2. The hydrogen-bonding scheme.

Table 4. *Hydrogen-bonding scheme*

Standard deviations: 0.1 Å and 0.8°.

For each hydrogen bond, the first O atom listed is the donor atom.

O(2)···O(4)	2.98 Å	O(4)···O(2)···O(3 <sup>iii</sup> )	120.3°
O(3)···O(2)	2.74	O(2)···O(3)···O(4 <sup>iii</sup> )	110.1
O(4)···O(3 <sup>ii</sup> )	2.92	O(2)···O(4)···O(2 <sup>ii</sup> )	86.3
		O(3 <sup>i</sup> )···O(4)···O(3 <sup>i</sup> )	98.7
		O(2)···O(4)···O(3 <sup>i</sup> )	95.2
		O(2)···O(4)···O(3 <sup>ii</sup> )	149.3

Symmetry code: (i)  $-\frac{1}{2} - x, \frac{1}{2} + y, -z$ ; (ii)  $\frac{1}{2} + x, \frac{1}{2} + y, z$ ; (iii)  $-\frac{1}{2} - x, -\frac{1}{2} + y, -z$ ; (iv)  $-x, y, -z$ .

reflections, no intensity variation. Direct phase determination revealed all non-H atoms and an O atom belonging to a water molecule. All O atoms and the four non-ring C atoms were assigned anisotropic thermal parameters while the ring C atoms were refined isotropically. The H atoms attached to C atoms were generated geometrically (C—H = 0.96 Å; the methyl groups were handled as rigid groups), assigned fixed isotropic thermal factors and allowed to ride on their respective parent C atoms. The H atoms attached to O atoms were located from a difference Fourier map; their positional parameters were not refined and they were given a fixed isotropic thermal parameter of 0.06 Å<sup>2</sup>.

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## Structure of ( $\pm$ )-7(S)-16-Deformyl-21-oxogeissoschizine–Oxindole

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**Abstract.** Methyl (3*S*\*,7*S*\*,8*a*'*S*\*)-2',3',6',7',8*a*'-hexahydro-6-(*E*)-ethylidene-2,5'-dioxospiro[3*H*-indole-3,1'(5'*H*)-indolizine]-7'-yl acetate, C<sub>20</sub>H<sub>22</sub>N<sub>2</sub>O<sub>4</sub>, *M<sub>r</sub>* = 354.50, monoclinic, *P*2<sub>1</sub>/*c*, *a* = 10.5052 (7), *b* = 16.442 (2), *c* = 10.3415 (8) Å,  $\beta$  = 95.358 (6)°, *V* = 1778.4 (3) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.32 g cm<sup>-3</sup> (198 K),  $\mu$  = 0.8677 cm<sup>-1</sup>, Mo *K* $\alpha$  radiation,  $\lambda$  = 0.7107 Å, *F*(000) = 752, *T* = 198 K, *R* = 0.0351 for 2635 reflections with *F<sub>o</sub>*  $\geq$  4[ $\sigma$ (*F<sub>o</sub>*)]. The indole NH group is hydrogen bonded to a carbonyl oxygen, O8 (related by  $-x, 1-y, 1-z$ ), with relevant parameters: N1···O8 2.857 (2) Å, H1···O8 1.94 (2) Å, N1—H1···O8 170 (2)°. The rings of the indolizine moiety are quasi-*trans* fused with ring junction torsion angles for C3—C8*a*—N4'—C3' of  $-12.08$  (15)° and C8'—C8*a*—N4'—C5' of 43.9 (2)°.

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All computations were performed on a Data General Corporation Nova 3/12 minicomputer with the *SHELXTL* program package (Sheldrick, 1982). Analytical expressions of neutral-atom scattering factors were employed (Cromer, 1974). Blocked-cascade least-squares refinement on *F* (Schilling, 1970) yielded the *R* indices and other parameters listed in Table 1.\* Final atomic parameters are given in Table 2; Table 3 gives bond lengths and angles. Fig. 1 shows a molecule with the atom numbering, while Fig. 2 shows the hydrogen-bonding scheme (see also Table 4).

\* 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 53567 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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**Experimental.** (1) was synthesized by an oxidative rearrangement of 16-deformyl-21-oxogeissoschizine. Treatment of 16-deformyl-21-oxogeissoschizine with *tert*-butylhypochlorite and triethylamine was followed by stirring at ambient temperature in acidic methanol in the presence of AgClO<sub>4</sub>. Full details of the synthetic procedure will appear elsewhere (Martin, Benage, Hunter, Geraci & Mortimore,

