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Structure of *ent*-9,15-Dioxo-8,9-seco-14,16-kauradiene-3 α ,7 β -diol Diacetate (Shikoccin Monoacetate)

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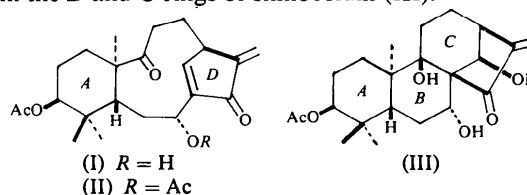
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Abstract. C₂₄H₃₂O₆, orthorhombic, *P*2₁2₁2₁, *a* = 13.218 (2), *b* = 19.053 (3), *c* = 9.205 (1) Å, *V* = 2318.2 (5) Å³, *Z* = 4, *D*_x = 1.193 Mg m⁻³, λ(Cu *K*α) = 1.54178 Å, μ(Cu *K*α) = 0.700 mm⁻¹. The structure was solved by a direct method, and refined by full-matrix least squares to *R*(*F*) = 0.082 for 1679 reflexions collected with a four-circle diffractometer. The *A* ring has a distorted chair form, and the ten-membered *B/C* ring has approximate *C*₂ symmetry. Intramolecular nonbonding contacts of 3.010 (12) and 3.074 (12) Å were observed for C(9)⋯C(14) and O(9)⋯C(14), respectively. The conformation of the ten-membered ring is stabilized by the interactions between the C(9) carbonyl group and the conjugated groups of the planar *D* ring.

Introduction. Several new kaurene-type diterpenes were isolated from *Rabdosia shikokiana* (Makino) Hara var. *occidentalis* (Murata) Hara (Labiatae) (Node, Ito, Fuji & Fujita, 1982). The major diterpenoid, shikoccin (I),

was shown to possess marginal *in vivo* antitumor activity (Nagao, Ito, Kohno, Kuroda & Fujita, 1982). The original aim of the present X-ray study of shikoccin monoacetate (II) was to establish the structure of shikoccin (I). The monoacetate (II) was obtained from shikoccin (I) on treatment with acetic anhydride in pyridine. It was also obtained by acetylation of another minor diterpenoid, shikoccidin (III), whose stereochemical structure has been determined by the X-ray method (Fujita, Ito, Uchida, Fuji, Taga & Osaki, 1979). A structural point of interest in the present monoacetate (II) is the conformation of the ten-membered ring which was converted from the *B* and *C* rings of shikoccidin (III).



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A single crystal with dimensions $0.10 \times 0.13 \times 0.25$ mm was obtained by recrystallization from methanol, and was mounted on a Rigaku AFC-5 diffractometer. A set of 1679 unique reflexions was measured to $2\theta = 120^\circ$ in the ω - 2θ scanning mode with a scan speed of 4° min^{-1} . The scan width was $\Delta(2\theta) = 2.0^\circ + 0.3^\circ \times \tan \theta_c$, where θ_c was the calculated Bragg angle for Cu K α radiation. Three standard reflexions were measured every 50 reflexions. No absorption correction was applied.

The structure analysis was carried out on a FACOM M-200 computer at the Data Processing Center, Kyoto University. The structure was solved with *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1977), using 200 reflexions for phase generation, and refined by full-matrix least squares with *ORFLS* (Busing, Martin & Levy, 1962) minimizing $\sum w(F_o - kF_c)^2$ with $w^{-1} = \sigma^2(F_o) + (0.023F_o)^2$, where $\sigma(F_o)$ was the standard deviation of F_o from counting statistics. All H atoms were located from difference Fourier maps, and were refined isotropically. The absolute structure was determined from that of shikoccidin (III). The final discrepancy indexes were 0.082 for $R = \sum |F_o - kF_c| / \sum |F_o|$ and 0.105 for $R_w = (\sum w|F_o - kF_c|) / \sum w|F_o|$.

Table 1. Atomic parameters with e.s.d.'s in parentheses for non-hydrogen atoms in shikoccin monoacetate

B_{eq} is the isotropic equivalent of the anisotropic thermal parameters (Hamilton, 1959).

	x	y	z	B_{eq} (\AA^2)
C(1)	0.2745 (7)	0.2393 (5)	0.7923 (12)	5.81
C(2)	0.3192 (8)	0.1669 (5)	0.7549 (12)	6.13
C(3)	0.4276 (8)	0.1636 (5)	0.7909 (9)	5.58
C(4)	0.4936 (7)	0.2175 (4)	0.7114 (8)	5.26
C(5)	0.4475 (6)	0.2930 (4)	0.7386 (8)	4.47
C(6)	0.5069 (7)	0.3518 (5)	0.6608 (9)	5.07
C(7)	0.5780 (7)	0.3922 (4)	0.7613 (9)	5.37
C(8)	0.5224 (6)	0.4256 (4)	0.8813 (9)	5.15
C(9)	0.2948 (6)	0.3696 (4)	0.7798 (10)	5.49
C(10)	0.3288 (6)	0.2985 (4)	0.7098 (9)	5.42
C(11)	0.2863 (7)	0.3751 (5)	0.9444 (11)	5.73
C(12)	0.2864 (8)	0.4530 (6)	1.0003 (16)	7.30
C(13)	0.3925 (8)	0.4822 (5)	1.0167 (11)	6.71
C(14)	0.4503 (7)	0.4746 (5)	0.8790 (12)	5.96
C(15)	0.5325 (8)	0.4005 (6)	1.0335 (9)	6.72
C(16)	0.4571 (7)	0.4420 (5)	1.1216 (10)	6.78
C(17)	0.4538 (14)	0.4392 (7)	1.2669 (14)	10.28
C(18)	0.5997 (7)	0.2161 (5)	0.7696 (10)	5.64
C(19)	0.4965 (10)	0.1964 (6)	0.5494 (10)	6.26
C(20)	0.3049 (9)	0.2993 (7)	0.5476 (12)	7.23
C(21)	0.4863 (8)	0.1271 (6)	1.0291 (10)	7.41
C(22)	0.4885 (16)	0.1482 (12)	1.1841 (16)	10.51
C(23)	0.7085 (9)	0.4310 (5)	0.6020 (11)	7.26
C(24)	0.7415 (15)	0.4892 (8)	0.5054 (21)	11.39
O(3)	0.4372 (5)	0.1744 (3)	0.9491 (5)	5.70
O(7)	0.6251 (4)	0.4484 (3)	0.6748 (7)	6.63
O(9)	0.2694 (5)	0.4181 (3)	0.7047 (9)	7.93
O(15)	0.5906 (6)	0.3562 (4)	1.0776 (7)	8.35
O(21)	0.5275 (7)	0.0769 (4)	0.9836 (9)	10.49
O(23)	0.7485 (6)	0.3762 (4)	0.6102 (11)	9.77

Table 2. Fractional coordinates and isotropic thermal parameters (\AA^2) for the hydrogen atoms in shikoccin monoacetate

	x	y	z	B_{iso}
H(C1)A	0.289 (6)	0.245 (4)	0.896 (10)	5.8 (18)
H(C1)B	0.203 (6)	0.238 (4)	0.753 (8)	5.8 (18)
H(C2)A	0.303 (6)	0.160 (4)	0.658 (10)	5.6 (19)
H(C2)B	0.287 (6)	0.143 (4)	0.813 (9)	4.3 (19)
H(C3)	0.467 (7)	0.124 (4)	0.772 (9)	6.6 (22)
H(C5)	0.446 (5)	0.295 (3)	0.846 (7)	3.3 (14)
H(C6)A	0.547 (6)	0.334 (4)	0.578 (9)	6.3 (19)
H(C6)B	0.456 (7)	0.379 (4)	0.619 (10)	7.3 (23)
H(C7)	0.622 (6)	0.361 (4)	0.814 (9)	6.0 (18)
H(C11)A	0.225 (7)	0.356 (4)	0.964 (10)	6.4 (21)
H(C11)B	0.343 (7)	0.345 (4)	0.986 (10)	7.9 (23)
H(C12)A	0.247 (7)	0.443 (5)	1.084 (11)	7.6 (22)
H(C12)B	0.237 (7)	0.479 (4)	0.914 (11)	7.2 (24)
H(C13)	0.396 (6)	0.535 (5)	1.050 (10)	7.7 (21)
H(C14)	0.416 (7)	0.487 (4)	0.792 (10)	5.9 (21)
H(C17)A	0.401 (7)	0.467 (5)	1.309 (10)	7.1 (22)
H(C17)B	0.521 (7)	0.410 (5)	1.275 (10)	6.8 (24)
H(C18)A	0.648 (6)	0.246 (4)	0.729 (9)	5.2 (18)
H(C18)B	0.608 (5)	0.233 (4)	0.880 (9)	5.2 (16)
H(C18)C	0.641 (6)	0.168 (4)	0.776 (9)	6.6 (20)
H(C19)A	0.433 (7)	0.171 (4)	0.542 (8)	5.4 (19)
H(C19)B	0.568 (7)	0.156 (5)	0.564 (10)	8.5 (23)
H(C19)C	0.514 (7)	0.228 (4)	0.499 (11)	6.6 (25)
H(C20)A	0.297 (8)	0.254 (6)	0.515 (14)	8.1 (26)
H(C20)B	0.225 (8)	0.312 (4)	0.531 (11)	7.6 (23)
H(C20)C	0.335 (8)	0.337 (6)	0.514 (13)	11.5 (30)
H(C22)A	0.504 (8)	0.127 (5)	1.252 (13)	9.1 (28)
H(C22)B	0.429 (8)	0.166 (6)	1.201 (12)	7.6 (29)
H(C22)C	0.542 (10)	0.195 (6)	1.221 (12)	11.1 (33)
H(C24)A	0.772 (9)	0.524 (6)	0.533 (14)	13.0 (32)
H(C24)B	0.798 (9)	0.479 (6)	0.447 (14)	10.1 (31)
H(C24)C	0.670 (9)	0.520 (6)	0.460 (15)	12.7 (37)

$kF_c |^2 / \sum |F_o|^2)^{1/2}$. The atomic scattering factors for all atoms were obtained from *International Tables for X-ray Crystallography* (1974).

The final atomic parameters are given in Tables 1 and 2.*

Discussion. A perspective molecular drawing of shikoccin monoacetate is shown in Fig. 1. The bond lengths and the valence angles for non-H atoms are listed in Table 3. The atomic numbering, the torsion angles about the covalent bonds, and the short intramolecular nonbonding distances are given in Fig. 2. The A ring is distorted from the ideal chair form because of the axial methyl-methyl interactions as usually observed in other kaurene derivatives; the C(19)···C(20) nonbonding distance is 3.203 (18) Å. The ten-membered B/C ring has an approximate C_2 conformation in which the conventional twofold axis passes through the

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36986 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

C(5)–C(6) and C(12)–C(13) bonds. The C_2 conformation corresponds to one of the eleven conformations which were classified for cyclodecane by Hendrickson (1964). According to Beagley (1977), however, the favorable conformation for cyclodecane or its related compounds is a twisted boat–chair–boat form or a twisted chair–chair–chair form. In the study of 5,10-seco-steroid derivatives by Mez, Rist, Ermer, Lorenc, Kalvoda & Michailovic (1976), the chair–chair–chair form was observed from the X-ray and NMR analyses, while the boat–chair–boat form was predicted from the conformational force-field calculations. The boat–chair–boat form was actually found in another 5,10-seco-steroid derivative by Fuhrer, Lorenc, Pavlovic, Rihs, Rist, Kalvoda & Michailovic (1979). The present 8,9-seco-diterpenoid has the same cyclodecenone ring as the 5,10-seco-steroids. The ten-membered ring fusion with the accompanying rings, however, differs from those of 5,10-seco-steroids. The *A* ring is fused at C(5)–C(10) and the *D* ring is fused at C(13)–C(14)–C(8). Hence, the internal rotations about these bonds are largely restricted by the ring fusion. Model building by the use of the Dreiding stereomodel allows two possible conformations for the shikoccin molecule as shown schematically in Fig. 3(*a*) and (*b*). The observed structure corresponds to the (*a*) form. The *D* ring is planar within 0.09 Å, and C(14)

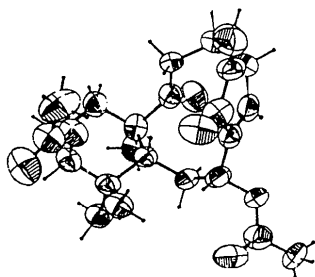


Fig. 1. A perspective view of a shikoccin monoacetate molecule. The thermal ellipsoids are at 50% probability except for those of the H atoms.

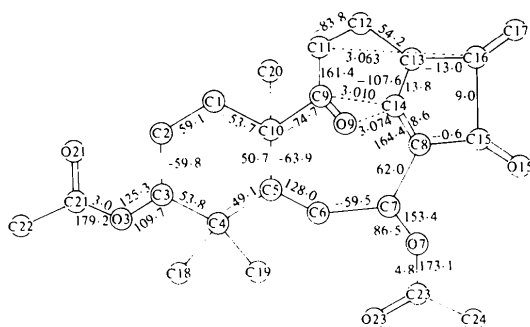


Fig. 2. The atomic numbering and the torsion angles ($^{\circ}$) in shikoccin monoacetate. E.s.d.'s for the torsion angles are 0.5–1.6 $^{\circ}$. The dashed lines indicate short intramolecular contacts with the distances given in Å (e.s.d.'s are 0.012 Å).

Table 3. Bond lengths (Å) and valence angles ($^{\circ}$) in shikoccin monoacetate

C(1)–C(2)	1.540 (14)	C(1)–C(10)	1.538 (13)
C(2)–C(3)	1.472 (15)	C(3)–C(4)	1.533 (13)
C(3)–O(3)	1.476 (10)	C(4)–C(5)	1.582 (11)
C(4)–C(18)	1.502 (13)	C(4)–C(19)	1.545 (12)
C(5)–C(6)	1.544 (12)	C(5)–C(10)	1.595 (11)
C(6)–C(7)	1.527 (12)	C(7)–C(8)	1.471 (12)
C(7)–O(7)	1.472 (10)	C(8)–C(14)	1.334 (12)
C(8)–C(15)	1.486 (12)	C(9)–C(10)	1.566 (11)
C(9)–C(11)	1.523 (14)	C(9)–O(9)	1.202 (11)
C(10)–C(20)	1.526 (14)	C(11)–C(12)	1.571 (15)
C(12)–C(13)	1.516 (15)	C(13)–C(14)	1.487 (15)
C(13)–C(16)	1.499 (14)	C(15)–C(16)	1.509 (14)
C(15)–O(15)	1.211 (13)	C(16)–C(17)	1.339 (16)
C(21)–C(22)	1.483 (18)	C(21)–O(3)	1.333 (12)
C(21)–O(21)	1.178 (14)	C(23)–C(24)	1.487 (20)
C(23)–O(7)	1.332 (13)	C(23)–O(23)	1.173 (13)
C(2)–C(1) C(10)	111.6 (8)	C(1)–C(2)–C(3)	111.2 (8)
C(2)–C(3)–C(4)	114.7 (8)	C(2)–C(3)–O(3)	107.4 (8)
C(4)–C(3)–O(3)	109.2 (7)	C(3)–C(4)–C(5)	108.3 (7)
C(3)–C(4)–C(18)	110.4 (7)	C(3)–C(4)–C(19)	107.5 (7)
C(5)–C(4)–C(18)	108.6 (7)	C(5)–C(4)–C(19)	113.5 (7)
C(18)–C(4)–C(19)	108.5 (8)	C(4)–C(5)–C(6)	113.0 (7)
C(4)–C(5)–C(10)	114.4 (6)	C(6)–C(5)–C(10)	112.1 (6)
C(5)–C(6)–C(7)	113.4 (7)	C(6)–C(7)–C(8)	111.4 (7)
C(6)–C(7)–O(7)	107.4 (7)	C(8)–C(7)–O(7)	107.6 (6)
C(7)–C(8)–C(14)	130.3 (8)	C(7)–C(8)–C(15)	121.6 (7)
C(14)–C(8)–C(15)	107.7 (8)	C(10)–C(9)–C(11)	119.3 (7)
C(10)–C(9)–O(9)	120.6 (8)	C(11)–C(9)–O(9)	119.9 (8)
C(1)–C(10)–C(5)	109.2 (6)	C(1)–C(10)–C(9)	107.3 (7)
C(1)–C(10)–C(20)	113.2 (8)	C(5)–C(10)–C(9)	105.7 (6)
C(5)–C(10)–C(20)	111.5 (7)	C(9)–C(10)–C(20)	109.5 (8)
C(9)–C(11)–C(12)	113.0 (8)	C(11)–C(12)–C(13)	112.3 (8)
C(12)–C(13)–C(14)	110.8 (9)	C(12)–C(13)–C(16)	113.8 (9)
C(14)–C(13)–C(16)	101.9 (8)	C(8)–C(14)–C(13)	114.9 (9)
C(8)–C(15)–C(16)	106.2 (8)	C(8)–C(15)–O(15)	126.7 (9)
C(16)–C(15)–O(15)	127.2 (8)	C(13)–C(16)–C(15)	107.3 (8)
C(13)–C(16)–C(17)	130.2 (11)	C(11)–C(16)–C(17)	122.5 (10)
C(22)–C(21)–O(3)	111.0 (11)	C(22)–C(21)–O(21)	123.6 (12)
O(3)–C(21)–O(21)	125.3 (9)	C(24)–C(23)–O(7)	111.0 (10)
C(24)–C(23)–O(23)	124.8 (12)	O(7)–C(23)–O(23)	124.2 (10)
C(3)–O(3)–C(21)	119.5 (7)	C(7)–O(7)–C(23)	116.2 (7)

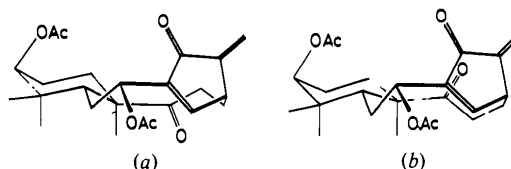


Fig. 3. Two possible structures for shikoccin monoacetate.

has short nonbonding contacts of 3.010 (12) Å to C(9) and of 3.074 (12) Å to O(9). The C(9)–O(9) carbonyl bond in the (*a*) form has a parallel arrangement with the C(8)–C(14) double bond and has an antiparallel arrangement with the C(15)–O(15) carbonyl bond, while the C(9)–O(9) carbonyl bond in the (*b*) form is almost perpendicular to the C(8)–C(14) and C(15)–O(15) bonds. The (*a*) form is, therefore, considered to be preferable to the (*b*) form, and the C_2 conformation of the ten-membered ring is stabilized by the interactions between the C(9)–O(9) carbonyl group and the conjugated groups of the *D* ring. Deviations of each atom from the five-atom least-squares plane for

acetoxy groups are less than 0.02 Å, and both acetoxy groups are attached to the C-atom rings in the usual eclipse form. There are no special intermolecular approaches to influence the molecular conformation significantly.

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Structure of 2,2-Dimethyl-1-phenyl- λ^3 -phosphetane 1-Oxide

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Abstract. $C_{11}H_{15}OP$, $M_r = 194.2$, monoclinic, space group $C2/c$, $Z = 8$, $a = 29.749$ (5), $b = 6.745$ (1), $c = 11.143$ (8) Å, $\beta = 107.33$ (2)°, $U = 2134.3$ Å³, $D_x = 1.21$ Mg m⁻³, $\mu(\text{Mo } K\alpha, \lambda = 0.71073 \text{ Å}) = 0.175$ mm⁻¹. The structure was solved by direct methods from diffractometer data and refined to an R value of 0.043 for 1374 observed reflections. The phosphetane ring is puckered with a dihedral angle of 25.8 (3)°.

Introduction. A number of previous X-ray structure studies on phosphetanes (Swank & Caughlin, 1968; Moret & Trefonas, 1969; Mazhar-ul-Haque, 1970, 1971, 1979; Fitzgerald, Campbell, Smith, Caughlin & Cremer, 1978) have indicated that these four-membered heterocyclic ring systems are significantly non-planar, with dihedral angles in the range 17–30° [exceptionally, one rigid and highly strained tetracyclic phosphetane (Mazhar-ul-Haque, Rashid & Cremer, 1978) did exhibit a dihedral angle of almost 47°].

The conformers which result from this ring non-planarity have provided an opportunity to investigate the stereochemical preferences of these systems. In

particular, Fitzgerald *et al.* (1978) correlated the preferred solid-state conformations with the extent and type of interactions between ring substituents. Additionally, these authors detected a qualitative correlation between the extent of ring substitution and the magnitude of the ring dihedral angle.

The present investigation is concerned with a structural study of a phosphetane with relatively little ring substitution.

Due to the hygroscopic nature of the crystals, no density measurement was attempted.

A crystal, *ca* 0.25 × 0.38 × 1.0 mm, mounted in a sealed glass capillary, was used for intensity measurements on an Enraf–Nonius CAD-4 diffractometer controlled by a PDP8/a computer at a take-off angle of 5° with graphite-monochromated Mo $K\alpha$ radiation.

Accurate cell dimensions and their standard deviations were obtained by least-squares refinement of the setting angles of 25 accurately centered reflections.

1874 independent reflections (to $\theta \leq 25^\circ$) were measured by ω - 2θ scans using the ZIGZAG routine (Enraf–Nonius, 1977); background counts were taken on each side of the peak. The intensities of three standard reflections were measured every 90 reflections and showed no significant change in intensities

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