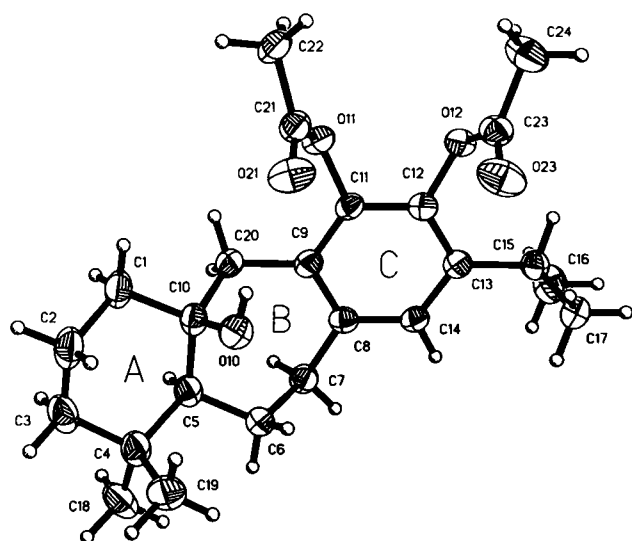


Crystal structure of diacetate of demethylsalvicanol, C₂₄H₃₄O₅

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C₂₄H₃₄O₅, orthorhombic, *P*2₁2₁2₁ (No. 19), *a* = 8.621(2) Å, *b* = 11.227(2) Å, *c* = 23.402(5) Å, *V* = 2265.0 Å³, *Z* = 4, *R*(*F*) = 0.058, *R*_w(*F*²) = 0.164.

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless prism, size 0.12 x 0.34 x 0.54 mm
Wavelength:	Cu K α radiation (1.54178 Å)
μ :	6.53 cm ⁻¹
Diffractometer:	Nicolet P3/F
Scan mode:	$\theta/2\theta$
<i>T</i> _{measurement} :	293 K
$2\theta_{\max}$:	100°
<i>N</i> (<i>hkl</i>) _{unique} :	1371
Criterion for <i>I</i> _o :	<i>I</i> _o > 2 σ (<i>I</i> _o)
<i>N</i> (<i>param</i>) _{refined} :	266
Programs:	SIR92, PARST, SHELXL-93

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(10)	4a	0.817(7)	0.350(6)	0.330(3)	0.06
H(1A)	4a	0.7716	0.5186	0.3943	0.06
H(1B)	4a	0.6005	0.5653	0.3991	0.06
H(2A)	4a	0.7093	0.3512	0.4507	0.06
H(2B)	4a	0.6673	0.4674	0.4847	0.06
H(3A)	4a	0.4106	0.4485	0.4586	0.06
H(3B)	4a	0.4653	0.3343	0.4912	0.06
H(5)	4a	0.3846	0.4314	0.3466	0.06
H(6A)	4a	0.3225	0.2441	0.3055	0.06
H(6B)	4a	0.4993	0.2260	0.2935	0.06
H(7A)	4a	0.3375	0.3012	0.2144	0.06
H(7B)	4a	0.3455	0.4243	0.2456	0.06
H(14)	4a	0.5073	0.2761	0.1409	0.06
H(15)	4a	0.8324	0.3379	0.0506	0.06
H(16A)	4a	0.6318	0.2755	-0.0093	0.06
H(16B)	4a	0.5884	0.3955	0.0210	0.06
H(16C)	4a	0.5170	0.2753	0.0426	0.06
H(17A)	4a	0.8066	0.1331	0.0364	0.06
H(17B)	4a	0.7058	0.1238	0.0919	0.06
H(17C)	4a	0.8810	0.1604	0.0961	0.06
H(18A)	4a	0.2103	0.2378	0.3818	0.06
H(18B)	4a	0.1985	0.3625	0.4116	0.06
H(18C)	4a	0.2263	0.2478	0.4484	0.06
H(19A)	4a	0.4642	0.1313	0.3787	0.06
H(19B)	4a	0.4694	0.1422	0.4455	0.06
H(19C)	4a	0.6093	0.1877	0.4087	0.06
H(20A)	4a	0.5312	0.5537	0.2909	0.06
H(20B)	4a	0.7101	0.5664	0.2999	0.06
H(22A)	4a	1.0901	0.7008	0.2393	0.06
H(22B)	4a	1.2200	0.6085	0.2546	0.06
H(22C)	4a	1.1199	0.6659	0.3033	0.06
H(24A)	4a	1.1742	0.5411	0.0798	0.06
H(24B)	4a	1.2492	0.4178	0.0648	0.06
H(24C)	4a	1.2889	0.4804	0.1228	0.06

Source of material: The title compound was extracted from the roots of *Salvia aspera* and crystallized from a mixture hexane/ethyl acetate by slowly evaporation. Refinement of absolute configuration by Flack's method was not conclusive, so the atom coordinates and the figure show the stereochemistry relative to C10(R) as found in salvicanol 11-*p*-bromobenzoate. The H-atom attached to O10 was located on an electron difference map and its coordinates refined. A fixed isotropic thermal parameter *U* = 0.06 Å² was used for all the hydrogen atoms.

The title compound is the diacetyl derivative of demethylsalvicanol, an icetexane-type diterpenoid previously isolated from *Salvia mellifera*, whose structure was established by comparison with salvicanol. The A/B junction in salvicanol was firmly established to be trans, by X-ray analysis (see ref. 1). Based on possible biogenetic correlations with salvicanol, the demethylsalvicanol was proposed as A/B trans fused, however, spectroscopic data were not conclusive. The six and seven-membered trans [$\tau_{4,5,10,1} = -45.0(6)^\circ$ and $\tau_{6,5,10,20} = 64.9(5)^\circ$] fused rings A and B adopt chair conformations, while the aromatic ring C is essentially planar. The hydroxy and acetoxy substituents are located on the β -face of the tricyclic skeleton. The acetoxy groups are nearly perpendicular to the aromatic ring, and that attached to C11 makes an intramolecular hydrogen bond [O10–H10, 0.82(6) Å; H10...O21, 2.22(7) Å; O10...O21, 2.993(6) Å; O10–H10...O21, 156(6)°]. The orientation of the isopropyl substituent is such that the plane of the aromatic ring bisects the angle CH₃–CH–CH₃, with methyl groups directed away from the acetoxy attached to C12. No unusual features were observed for bond lengths and angles. Crystal packing is entirely due to van der Waals forces.

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(10)	4a	0.7284(5)	0.3276(3)	0.3356(2)	0.062(2)	0.048(2)	0.072(2)	0.014(2)	0.004(2)	0.002(2)
O(11)	4a	0.8946(5)	0.5689(3)	0.2209(2)	0.062(2)	0.047(2)	0.057(2)	-0.009(2)	-0.010(2)	0.001(2)
O(12)	4a	0.9471(5)	0.4716(3)	0.1196(2)	0.057(2)	0.057(2)	0.057(2)	-0.009(2)	0.005(2)	0.003(2)
O(21)	4a	1.0061(5)	0.4474(4)	0.2847(2)	0.069(3)	0.067(3)	0.100(3)	-0.007(2)	-0.025(2)	0.015(3)
O(23)	4a	1.0830(5)	0.3115(5)	0.1451(2)	0.060(3)	0.104(4)	0.115(4)	0.004(3)	0.005(3)	0.032(3)
C(1)	4a	0.6639(8)	0.4941(6)	0.3970(2)	0.093(5)	0.063(3)	0.051(3)	0.006(4)	0.003(3)	-0.012(3)
C(2)	4a	0.641(1)	0.4197(6)	0.4516(2)	0.123(6)	0.073(4)	0.046(3)	0.006(5)	-0.006(4)	-0.009(3)
C(3)	4a	0.477(1)	0.3792(6)	0.4561(3)	0.125(7)	0.061(4)	0.049(4)	0.017(4)	0.016(4)	0.011(3)
C(4)	4a	0.4235(8)	0.3024(5)	0.4064(2)	0.086(5)	0.054(3)	0.058(4)	0.015(3)	0.011(3)	0.001(3)
C(5)	4a	0.4580(7)	0.3650(5)	0.3486(2)	0.065(4)	0.047(3)	0.053(3)	0.013(3)	0.007(3)	0.002(3)
C(6)	4a	0.4186(7)	0.2856(5)	0.2973(2)	0.062(4)	0.058(3)	0.058(3)	-0.007(3)	0.011(3)	-0.005(3)
C(7)	4a	0.4007(7)	0.3502(5)	0.2393(2)	0.056(4)	0.057(3)	0.054(3)	-0.001(3)	0.000(3)	-0.005(3)
C(8)	4a	0.5508(6)	0.3773(4)	0.2096(2)	0.049(3)	0.046(3)	0.049(3)	-0.002(3)	-0.004(3)	0.007(3)
C(9)	4a	0.6560(6)	0.4551(4)	0.2344(2)	0.054(4)	0.042(3)	0.052(3)	0.000(3)	-0.002(3)	-0.001(3)
C(10)	4a	0.6195(7)	0.4223(5)	0.3431(2)	0.068(4)	0.047(3)	0.044(3)	0.009(3)	0.001(3)	-0.001(3)
C(11)	4a	0.7890(6)	0.4807(4)	0.2032(2)	0.052(3)	0.043(3)	0.045(3)	-0.005(3)	-0.008(3)	-0.001(3)
C(12)	4a	0.8151(6)	0.4317(5)	0.1496(2)	0.047(3)	0.050(3)	0.048(3)	-0.001(3)	-0.004(3)	0.005(3)
C(13)	4a	0.7139(7)	0.3517(5)	0.1249(2)	0.055(3)	0.048(3)	0.043(3)	0.000(3)	-0.006(3)	0.003(3)
C(14)	4a	0.5804(6)	0.3279(5)	0.1564(2)	0.048(3)	0.045(3)	0.050(3)	-0.006(3)	-0.005(3)	0.000(3)
C(15)	4a	0.7434(7)	0.2964(5)	0.0674(2)	0.064(4)	0.066(3)	0.044(3)	-0.009(3)	0.001(3)	-0.004(3)
C(16)	4a	0.6077(9)	0.3121(7)	0.0267(3)	0.098(5)	0.110(5)	0.052(4)	0.000(5)	-0.014(4)	-0.004(4)
C(17)	4a	0.7883(9)	0.1665(5)	0.0735(3)	0.085(4)	0.064(4)	0.069(4)	-0.012(4)	0.013(4)	-0.019(3)
C(18)	4a	0.2484(9)	0.2861(7)	0.4126(3)	0.101(6)	0.090(5)	0.067(4)	0.005(4)	0.026(4)	0.023(4)
C(19)	4a	0.499(1)	0.1795(5)	0.4102(3)	0.112(6)	0.048(3)	0.084(4)	0.011(4)	0.009(4)	0.017(3)
C(20)	4a	0.6279(7)	0.5096(5)	0.2924(2)	0.058(3)	0.043(3)	0.053(3)	0.001(3)	0.002(3)	-0.003(3)
C(21)	4a	1.0060(7)	0.5401(6)	0.2595(3)	0.053(4)	0.057(4)	0.060(4)	-0.002(3)	0.000(3)	-0.010(3)
C(22)	4a	1.1187(8)	0.6372(5)	0.2646(3)	0.073(4)	0.064(4)	0.079(4)	-0.013(4)	-0.003(4)	-0.026(3)
C(23)	4a	1.0784(7)	0.4052(6)	0.1224(3)	0.051(4)	0.071(4)	0.058(3)	-0.010(4)	-0.004(3)	0.000(3)
C(24)	4a	1.2089(8)	0.4664(8)	0.0951(3)	0.063(4)	0.122(6)	0.082(4)	-0.015(5)	0.008(4)	0.013(4)

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