Crystal structure of taedol, $C_{10}H_{16}O_2$, *from Haplopappus taeda*

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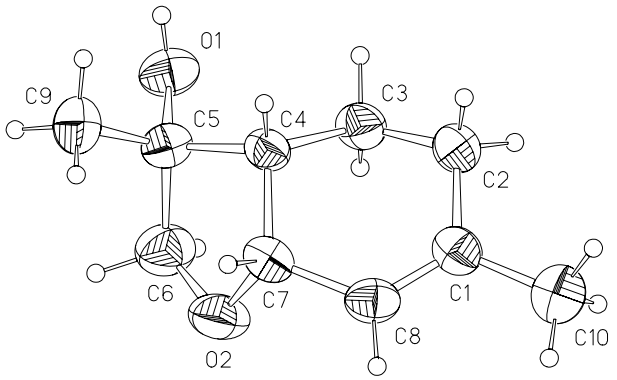

Abstract

$C_{10}H_{16}O_2$, monoclinic, $P12_11$ (no. 4), a = 8.974(2) Å, b = 5.933(1) Å, c = 8.994(2) Å, β = 96.29(3)°, V = 476.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.052, $wR_{obs}(F^2)$ = 0.148, T = 293 K.

Source of material

Taedol was isolated by column chromatography from the CH_2Cl_2 extract of the resinous exudates of leaves and stems of *Haplopappus taeda Reiche* and its structure was determinated by spectroscopical analysis. The monoterpene was crystallized in hexane to afford the material used in this study.

Experimental details

The N_{gt}/N_{param} ratio is small due to poor quality/decomposition of the crystals and connected with the use of a restricted *hkl* range according to the Laue group with respect that a higher accuracy of the structural parameters which could be reached with a complete data set. Therefore, all H atoms were placed in calculated positions, although they could be found from Fourier difference maps.

Discussion

Taedol was previously isolated from the an EtOAc extract of dried and ground stems of *Haplopappus taeda*, without any discussion about the stereochemistry [1]. The perspective view of tadeol shows a structure of a bicyclic derivative of α-terpineol, with an epoxy function between carbons C4 and C7. The ring of α-terpineol, assumes a sofa conformation (asymmetry parameter ΔC_2(C8–C1) = 0.004(6) Å) with atom C4 displaced by 0.600(2) Å from the C3–C2–C1–C8–C7 basal plane. The tetrahydrofuran ring adopts an envelope conformation with ΔC_s(C7) = 0.022(1) Å. The C1–C8–C7–O2 torsion angle of 115.4(3)° indicates the *cis* arrangement of the tetrahydrofuran-ring-junction H atoms. The bond distances and angles are in normal ranges.

Table 1. Data collection and handling.

Crystal:	colorless, prismatic, size 0.08 × 0.10 × 0.15 mm
Wavelength:	Cu K_α radiation (1.54180 Å)
μ:	6.38 cm^{-1}
Diffractometer, scan mode:	SEIFERT XRD 3000 SC, $\omega/2\theta$
$2\theta_{max}$:	119.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	826, 774
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 563
$N(param)_{refined}$:	113
Programs:	X-RAY80 [2], SHELXTL [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	2*a*	0.8255	–0.6810	0.1215	0.127
H(2A)	2*a*	0.3331	–0.2880	0.1481	0.097
H(2B)	2*a*	0.3966	–0.3902	0.3029	0.097
H(3A)	2*a*	0.5656	–0.2882	0.0651	0.089
H(3B)	2*a*	0.5425	–0.5292	0.1309	0.089
H(4)	2*a*	0.6904	–0.4361	0.3534	0.073
H(6A)	2*a*	0.9735	–0.1335	0.1233	0.117
H(6B)	2*a*	0.8218	–0.1654	0.0200	0.117
H(7)	2*a*	0.7752	–0.0888	0.4111	0.085
H(8)	2*a*	0.5773	0.1784	0.3573	0.089
H(9A)	2*a*	0.9466	–0.5912	0.3606	0.148
H(9B)	2*a*	0.9799	–0.3353	0.3922	0.148
H(9C)	2*a*	1.0615	–0.4632	0.2719	0.148
H(10A)	2*a*	0.3232	0.2142	0.3545	0.144
H(10B)	2*a*	0.2365	–0.0131	0.3675	0.144
H(10C)	2*a*	0.2429	0.0991	0.2105	0.144

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	2*a*	0.8263(5)	–0.5561(7)	0.0826(4)	0.113(3)	0.068(2)	0.076(2)	0.005(2)	0.018(2)	–0.004(2)
O(2)	2*a*	0.8006(5)	0.0072(7)	0.2030(5)	0.112(3)	0.066(2)	0.121(3)	–0.016(2)	0.047(2)	–0.004(2)
C(1)	2*a*	0.4376(6)	–0.056(1)	0.2879(5)	0.074(3)	0.075(3)	0.069(3)	–0.008(3)	0.007(2)	0.002(3)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(2)	2*a*	0.4184(5)	–0.2873(9)	0.2243(6)	0.069(3)	0.074(4)	0.095(4)	–0.006(3)	–0.009(3)	–0.009(3)
C(3)	2*a*	0.5551(5)	–0.371(1)	0.1563(5)	0.078(3)	0.068(3)	0.070(3)	–0.007(3)	–0.013(2)	–0.006(3)
C(4)	2*a*	0.6958(5)	–0.3403(9)	0.2654(5)	0.063(3)	0.057(3)	0.061(2)	–0.009(2)	–0.001(2)	0.001(2)
C(5)	2*a*	0.8434(6)	–0.3863(9)	0.1970(5)	0.082(3)	0.060(3)	0.073(3)	0.004(3)	0.010(2)	0.004(3)
C(6)	2*a*	0.8671(8)	–0.163(1)	0.1230(7)	0.112(4)	0.082(4)	0.104(4)	0.001(4)	0.034(3)	0.008(4)
C(7)	2*a*	0.7153(6)	–0.0945(9)	0.3134(5)	0.075(3)	0.069(3)	0.067(3)	–0.015(3)	0.008(2)	–0.003(3)
C(8)	2*a*	0.5714(6)	0.030(1)	0.3235(5)	0.094(4)	0.061(3)	0.068(3)	–0.004(3)	0.016(2)	–0.008(3)
C(9)	2*a*	0.9695(6)	–0.450(1)	0.3164(6)	0.072(3)	0.100(4)	0.120(4)	0.006(3)	–0.005(3)	0.001(4)
C(10)	2*a*	0.2975(6)	0.073(1)	0.3068(6)	0.093(4)	0.104(5)	0.090(3)	0.008(4)	0.010(3)	0.000(4)

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References

1. Marambio, O; Silva, M.: New Compounds from *Haplopappus taeda Reiche*. Bol. Soc. Chil. Quim. **34** (1989) 105-113.
2. Stewart, J. M.; Kundell, F. A.; Baldwin, J. C.: The X-RAY80 System. Computer Science Center, University of Maryland, Maryland, USA 1990.
3. Sheldrick, G. M.: SHELXTL. Structure Determination Software Suite. Version 5.0. Siemens Industrial Automation, Analytical Instrumentation, Madison, Wisconsin, USA 1995.