Crystal structure of (3a*R*,8a*S*,9a*S*)-8a-methyl-3,5-dimethylene-decahydronaphtho[2,3-*b*]furan-2(3*H*)-one, $C_{15}H_{20}O_2$

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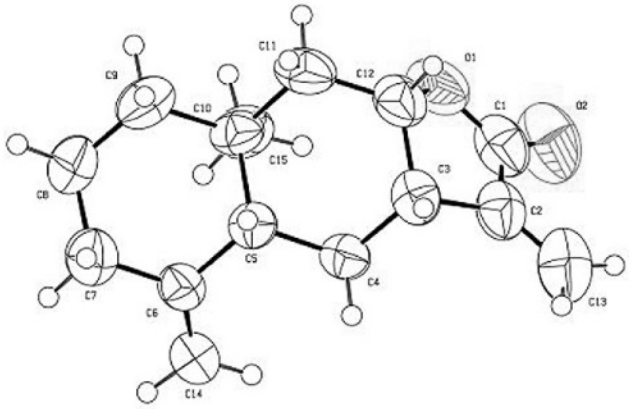

Abstract

$C_{15}H_{20}O_2$, orthorhombic, $P2_12_12_1$ (no. 19), a = 6.5445(5) Å, b = 8.7394(6) Å, c = 22.794(2) Å, V = 1303.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.0748, $wR_{ref}(F^2)$ = 0.1419, T = 298 K.

Table 1. Data collection and handling.

Crystal:	colourless irregular, size 0.06×0.1×0.3 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.77 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6511, 2278
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1055
$N(param)_{refined}$:	155
Programs:	SHELX [6]

Source of material

1 Kg of dried roots of *Inular racemosa* was grinded into powder and heated in 500 ml ethanol under reflux for 2 h, and the solution was braise to obtain 112 g of ointment. After the ointment was dispersed in 200 ml water, and extracted three times with 50 ml petroleum ether, braised to 18 g of another ointment. 5 g of the tittle product was obtained by silica gel column (60-100 mesh) and hexane:ethyl acetate (50:1,*v*/*v*) as mobile phase. Crystals suitable for single crystal X-ray diffraction were obtained by recrystallization from a mixed solvent of methanol and dichloromethane (1:3 = *v*/*v*) at room temperature. **m.p.** 111.2-112.9 °C; **^{1}H-NMR** ($CDCl_3$, 500 MHz) δ ppm: 6.13 (1H, brs, H-13a), 5.59 (1H, brs, H-13b), 4.77 (1H, brs, H-15a), 4.44 (1H, brs, H-15b), 4.50 (1H, brs, H-8), 2.99 (1H, m, H-7), 2.34 (1H, d, J = 12.0 Hz, H-3b), 2.20 (1H, d, J = 16.0 Hz, H-9a), 2.01 (1H, m, H-3a), 1.85 (1H, J = 12.5 Hz, H-5), 1.75 (1H, m, H-6a), 1.71 (1H, m, H-9b), 1.60 (2H, m, H-2), 1.54 (1H, m, H-1a), 1.403 (1H, m, H-6b), 1.252 (1H, m, H-1b), 0.829 (3H, s, 14-CH_3); **^{13}C-NMR** ($CDCl_3$, 100 MHz) δ ppm: 24.2, 25.3, 29.4, 30.5, 38.8, 40.3, 45.8, 57.3, 72.5, 111.4, 125.2, 138.6, 149.5, 169.8; **MS**: m/z = 233 $[M+H]^+$.

Discussion

Inula racemosa is one of traditional Chinese drugs which had various biological and pharmaceutical activities such anti-inflammatory, hepatoprotective activity, antidematophytic and antifungal activity [1, 2]. The roots of Inular racemosa in our study were collected in Tibet. The title compound had been isolated from the extracts of roots of Inula racemosa. Although (3a*R*,8a*S*,9a*S*)-8a-methyl-3,5-dimethylene-decahydronaphtho[2,3-*b*]furan-2(3*H*)-one was not a new compund, its X-ray crystal structure has not been reported in the literature [3]. This structure determination is part of our interest on the development of heterocyclic compounds [4, 5].

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

Atom	Site	x	y	z	U_{iso}
H(3)	4*a*	0.8342	0.6186	0.1036	0.073
H(4A)	4*a*	0.5605	0.3797	0.0730	0.069
H(4B)	4*a*	0.7490	0.4348	0.0363	0.069
H(5)	4*a*	0.9679	0.3269	0.1051	0.062
H(7A)	4*a*	1.1218	0.0671	0.0886	0.098
H(7B)	4*a*	0.9778	–0.0627	0.0651	0.098
H(8A)	4*a*	0.7974	–0.0684	0.1521	0.108
H(8B)	4*a*	1.0301	–0.0797	0.1684	0.108
H(9A)	4*a*	1.0408	0.1751	0.1929	0.102
H(9B)	4*a*	0.8615	0.0993	0.2279	0.102
H(11A)	4*a*	0.7065	0.3712	0.2388	0.090
H(11B)	4*a*	0.9145	0.4127	0.2092	0.090
H(12)	4*a*	0.7322	0.6233	0.1977	0.087
H(13A)	4*a*	0.3695	0.8111	0.0556	0.121
H(13B)	4*a*	0.5979	0.7846	0.0313	0.121
H(14A)	4*a*	0.7493	0.0308	–0.0032	0.085
H(14B)	4*a*	0.6511	0.1991	0.0027	0.085
H(15A)	4*a*	0.5087	0.1506	0.2040	0.116
H(15B)	4*a*	0.5348	0.0966	0.1388	0.116
H(15C)	4*a*	0.4483	0.2602	0.1524	0.116

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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)	4*a*	0.4557(6)	0.5319(4)	0.1843(2)	0.068(2)	0.116(3)	0.082(3)	0.019(2)	0.009(2)	–0.022(2)
O(2)	4*a*	0.1888(6)	0.6408(5)	0.1409(2)	0.061(2)	0.126(3)	0.162(4)	0.020(2)	–0.002(2)	–0.022(3)
C(1)	4*a*	0.3689(9)	0.6145(7)	0.1413(3)	0.060(4)	0.081(4)	0.098(4)	0.009(3)	–0.012(4)	–0.028(4)
C(2)	4*a*	0.5251(8)	0.6575(6)	0.0981(3)	0.066(3)	0.057(3)	0.087(4)	0.000(3)	–0.017(3)	–0.014(3)
C(3)	4*a*	0.7076(7)	0.5619(5)	0.1112(2)	0.047(3)	0.061(3)	0.076(3)	0.001(3)	–0.008(2)	–0.009(3)
C(4)	4*a*	0.7012(7)	0.4140(5)	0.0758(2)	0.058(3)	0.064(3)	0.051(3)	0.001(3)	0.003(2)	–0.004(3)
C(5)	4*a*	0.8285(6)	0.2869(5)	0.1017(2)	0.038(2)	0.064(3)	0.053(3)	0.002(2)	–0.003(2)	0.000(3)
C(6)	4*a*	0.8422(7)	0.1461(5)	0.0647(2)	0.057(3)	0.061(3)	0.055(3)	0.008(3)	0.003(3)	–0.004(3)
C(7)	4*a*	0.9829(8)	0.0284(5)	0.0894(2)	0.083(4)	0.082(4)	0.081(4)	0.019(3)	–0.008(3)	–0.005(3)
C(8)	4*a*	0.9261(9)	–0.0133(6)	0.1519(2)	0.111(4)	0.079(4)	0.081(4)	0.028(4)	–0.018(4)	0.007(3)
C(9)	4*a*	0.9070(8)	0.1285(6)	0.1890(2)	0.093(4)	0.098(4)	0.064(3)	0.011(4)	–0.019(3)	0.011(4)
C(10)	4*a*	0.7588(7)	0.2471(6)	0.1642(2)	0.057(3)	0.076(3)	0.047(3)	0.001(3)	–0.009(2)	0.001(3)
C(11)	4*a*	0.7714(7)	0.3920(6)	0.2014(2)	0.070(3)	0.100(4)	0.054(3)	0.002(3)	–0.009(3)	–0.017(3)
C(12)	4*a*	0.6774(8)	0.5350(6)	0.1763(2)	0.073(4)	0.076(4)	0.069(4)	0.009(3)	–0.015(3)	–0.021(3)
C(13)	4*a*	0.4946(9)	0.7610(6)	0.0578(3)	0.087(4)	0.078(4)	0.138(5)	0.011(4)	–0.010(4)	–0.006(4)
C(14)	4*a*	0.7375(7)	0.1232(5)	0.0168(2)	0.081(3)	0.064(3)	0.067(3)	0.011(3)	0.003(3)	–0.007(3)
C(15)	4*a*	0.5427(7)	0.1827(5)	0.1649(2)	0.072(4)	0.093(4)	0.067(3)	–0.004(3)	0.015(3)	0.015(3)

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