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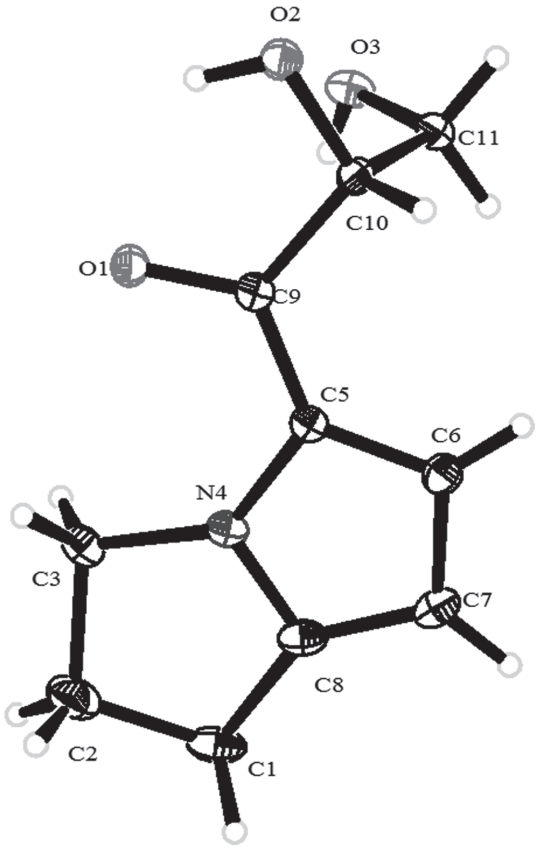
Jun-Jie Tan†, Xiao-Yu Liu†, Jie Zhang, Xi Chen, Yi-Ming Li* and Bing-Hua Jiao*

Crystal structure of (*R*)-1-(2,3-dihydro-1*H*-pyrrolizin-5-yl)-2,3-dihydroxypropan-1-one, $C_{10}H_{13}NO_3$



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Abstract

$C_{10}H_{13}NO_3$, monoclinic, $P2_1$ (no. 4), $a = 11.8118(5)$ Å, $b = 4.9299(2)$ Å, $c = 14.5436(7)$ Å, $\beta = 89.011(3)°$, $V = 945.28(7)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0302$, $wR_{ref}(F^2) = 0.0774$, $T = 170$ K.

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One of two crystallographically independant molecules of the title structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.20 × 0.08 × 0.03 mm
Wavelength:	Cu *Kα* radiation (1.54178 Å)
μ:	8.4 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	130°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10070, 2946, 0.041
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2898
$N(param)_{refined}$:	216
Programs:	SHELX [3], Bruker programs [4]

Source of material

The title compound was isolated from the fungus *Eutypella* sp. D-1, which was harvested from the soil of London Island of Kongsfjorden of Nylesund District (altitude of 100 m) in the Arctic [1]. The culture (150 L) was centrifuged to give the broth and mycelia. The broth was exhaustively extracted with EtOAc three times, then the EtOAc layers were combined and evaporated under reduced pressure at a temperature not exceeding 40 °C to yield a dark brown gum (200 g), which was subjected to column chromatography (CC) on silica gel and eluted with EtOH in petroleum ether (PE) (0 to 100%, stepwise) to yield 5 fractions (Fr.1 to Fr.5). Fr.4 (25 g) was applied to a MCI gel

† These authors contributed equally to this work.

***Corresponding authors: Yi-Ming Li,** School of Pharmacy, Shanghai University of Traditional Chinese Medicine, Shanghai 201203, P.R. China, e-mail: ymli@shutcm.edu.cn; and **Bing-Hua Jiao,** Department of Biochemistry and Molecular Biology, Second Military Medical University, Shanghai 200433, P.R. China, e-mail: bhjiao@smmu.edu.cn
Jun-Jie Tan: School of Pharmacy, Shanghai University of Traditional Chinese Medicine, Shanghai 201203, P.R. China; and Department of Natural Medicinal Chemistry, Shanghai Institute of Materia Medica, Chinese Academy of Sciences, Shanghai 201203, P.R. China
Xiao-Yu Liu: Department of Biochemistry and Molecular Biology, Second Military Medical University, Shanghai. 200433, P.R. China
Jie Zhang and Xi Chen: School of Pharmacy, Shanghai University of Traditional Chinese Medicine, Shanghai 201203, P.R. China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O6	0.61299(10)	0.9928(3)	0.11529(8)	0.0363(3)
H6	0.5536	1.0560	0.0878	0.055*
O5	0.59988(10)	0.6984(3)	−0.03748(7)	0.0399(3)
H5	0.6367	0.8158	−0.0562	0.060*
O4	0.80900(10)	0.8687(3)	−0.00537(7)	0.0408(3)
O3	0.37342(9)	0.1183(3)	0.30346(8)	0.0349(3)
H3	0.3995	−0.0281	0.3219	0.052*
O2	0.47257(10)	0.6339(2)	0.36607(7)	0.0304(3)
O1	0.57102(10)	0.2859(2)	0.47526(7)	0.0292(3)
N15	0.98851(11)	0.6759(3)	0.12807(8)	0.0290(3)
N4	0.74433(11)	−0.0991(3)	0.45470(8)	0.0266(3)
C22	0.61006(14)	0.7074(4)	0.10931(11)	0.0335(4)
H22A	0.5305	0.6467	0.1009	0.040*
H22B	0.6507	0.6303	0.1607	0.040*
C21	0.66478(13)	0.6034(4)	0.03881(10)	0.0310(4)
H21	0.6639	0.4046	0.0390	0.037*
C20	0.78949(13)	0.7019(4)	0.04525(10)	0.0293(4)
C16	0.87513(13)	0.5894(4)	0.10990(10)	0.0289(4)
C14	1.05908(13)	0.8670(4)	0.09102(11)	0.0350(4)
H14A	1.0282	1.0496	0.0896	0.042*
H14B	1.0647	0.8123	0.0356	0.042*
C13	1.17633(16)	0.8466(6)	0.15000(13)	0.0505(5)
H13A	1.2367	0.8111	0.1191	0.061*
H13B	1.1937	1.0160	0.1796	0.061*
C19	1.04638(14)	0.5346(4)	0.19192(11)	0.0348(4)
C18	0.97262(15)	0.3489(5)	0.21740(11)	0.0375(4)
H18	0.9909	0.2259	0.2606	0.045*
C17	0.86532(14)	0.3827(4)	0.16557(10)	0.0318(4)
H17	0.7988	0.2843	0.1678	0.038*
C12	1.17049(16)	0.6190(6)	0.20972(15)	0.0563(6)
H12A	1.2201	0.4700	0.2006	0.068*
H12B	1.1933	0.6811	0.2661	0.068*
C11	0.45855(14)	0.2603(4)	0.27099(10)	0.0314(4)
H11A	0.4210	0.3789	0.2271	0.038*
H11B	0.5050	0.1313	0.2474	0.038*
C10	0.53723(13)	0.4293(3)	0.33602(10)	0.0261(4)
H10	0.5957	0.5166	0.3100	0.031*
C9	0.59878(12)	0.2567(3)	0.40769(9)	0.0236(3)
C5	0.68662(12)	0.0693(3)	0.39377(9)	0.0240(3)
C3	0.73494(14)	−0.1636(4)	0.53988(10)	0.0335(4)
H3A	0.6661	−0.2688	0.5419	0.040*
H3B	0.7347	−0.0006	0.5727	0.040*
C2	0.84450(17)	−0.3304(5)	0.56835(13)	0.0520(5)
H2A	0.8290	−0.4811	0.6023	0.062*
H2B	0.9044	−0.2185	0.6004	0.062*
C1	0.88310(14)	−0.4353(4)	0.49058(13)	0.0413(5)
H1A	0.9663	−0.4268	0.4963	0.050*
H1B	0.8577	−0.6205	0.4783	0.050*
C8	0.82480(13)	−0.2445(4)	0.42592(11)	0.0322(4)
C7	0.82227(14)	−0.1706(4)	0.34472(11)	0.0365(4)
H7	0.8691	−0.2386	0.3102	0.044*
C6	0.73589(13)	0.0257(4)	0.32457(11)	0.0312(4)
H6A	0.7149	0.1122	0.2739	0.037*
H2	0.474(2)	0.594(6)	0.4151(17)	0.051(7)*

CC ($MeOH/H_2O$, 50 to 100%) to obtain 12 fractions (Fr.4A-Fr.4L). Fr.4C (1.99 g) was subjected to a Sephadex LH-20 CC that was eluted with MeOH to obtain 8 subfractions (Fr.4C1-Fr.4C8). Fr.4C6 was further separated using the ODS-A—HG reversed-phase silica gel CC ($MeOH/H_2O$, 20 to 30%), silica gel CC ($CH_2Cl_2/MeOH = 50/1$) and semi-preparative HPLC ($CH_3CN/H_2O = 15/85$, 4 mL/min) to give the title compound (14 mg, Rt = 18.62 min). Colorless crystals were obtained by slow evaporation at room temperature from PE and acetone in a 5:1 ratio.

Experimental details

Hydrogen atoms of O—H were found from a Fourier difference map, and refined with a fixed distance of 0.86 Å and isotropic displacement parameters of 1.5 U_{eq} of the parent atoms. The remaining hydrogen atoms were placed in calculated positions and refined with a riding model with isotropic displacement parameters set to 1.2 (sp^2) and 1.5 (sp^3) times U_{eq} of the parent atom. The absolute structure was confirmed by the Flack parameter $x = 0.03(15)$.

Discussion

The title compound is a new substituted pyrrolizine alkaloid and its planar structure has been elucidated by spectroscopic methods (including NMR, MS and IR). The absolute stereochemistry of the chiral centres at C10 were determined as *R* by X-ray diffraction using CuKα radiation [2]. In both independent molecules there is one intramolecular hydrogen bond (*cf*. the figure) and there are some other intermolecular ones connecting adjacent molecules.

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