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Crystal structure of 10-oxysophoridine, $C_{15}H_{22}N_2O_2$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.12 × 0.11 × 0.10 mm
Wavelength:	Cu K α radiation (1.54184 Å)
μ :	0.72 mm ⁻¹
Diffractometer, scan mode:	SuperNova, Atlas S2, ω
$\theta_{\max}$, completeness:	73.3°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5493, 2410, 0.025
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2312
$N(\text{param})_{\text{refined}}$:	172
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3, 4]

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Abstract

$C_{15}H_{22}N_2O_2$, orthorhombic, $P2_12_12_1$ $a = 5.27590(10)$ Å, $b = 10.3482(3)$ Å, $c = 23.5372(6)$ Å, $V = 1285.04(6)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0338$, $wR_{\text{ref}}(F^2) = 0.0852$, $T = 100(1)$ K.

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The molecular structure is shown in the Figure. Table 1 contains crystallographic data, and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The dry roots of *Sophora flavescens* were purchased from Xi'an, Shanxi Province, China. Dried Powder (25.0 kg) was extracted three times with 95% ethanol at room temperature. The solution was concentrated in vacuum to get a residue (1.6 kg). The crude extract was suspended in water and adjusted pH to 4–5 with HCl, and then partitioned with chloroform. The aqueous layer was adjusted to pH 9–10 with ammonium hydroxide and extracted by chloroform. After evaporation of solvent, the chloroform soluble fraction (463 g) was obtained. This fraction was chromatographed on a silica gel column, eluted with $CHCl_3$ – CH_3OH solvent system, which afforded six fractions (Fr. 1–7). The title compound was obtained from Fr.4 and crystallized in methanol.

Experimental details

Using Olex2 [2], the structure was solved with the ShelXT [3].

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} [*] /U _{eq}
O1	0.1299 (3)	0.03547 (13)	0.25683 (6)	0.0217 (3)
O2	0.5907 (3)	0.54518 (14)	0.50883 (6)	0.0244 (4)
N1	0.3033 (3)	0.13754 (15)	0.33289 (7)	0.0157 (4)
N2	0.5268 (3)	0.47198 (15)	0.41984 (7)	0.0161 (4)
C2	0.3546 (4)	0.01317 (18)	0.36118 (8)	0.0186 (4)
H2A	0.315285	−0.057380	0.335516	0.022 [*]
H2B	0.247748	0.004782	0.394508	0.022 [*]
C3	0.6319 (4)	0.00597 (19)	0.37860 (8)	0.0195 (4)
H3A	0.738808	0.008776	0.345084	0.023 [*]
H3B	0.663963	−0.074743	0.398282	0.023 [*]
C4	0.6949 (4)	0.11958 (19)	0.41752 (8)	0.0180 (4)
H4A	0.873567	0.117031	0.427186	0.022 [*]
H4B	0.598487	0.111879	0.452448	0.022 [*]
C5	0.6338 (4)	0.24871 (18)	0.38918 (8)	0.0148 (4)
H5	0.748597	0.261349	0.356921	0.018 [*]
C6	0.3592 (4)	0.25302 (18)	0.36772 (8)	0.0144 (4)
H6	0.248232	0.249985	0.401083	0.017 [*]
C7	0.3044 (4)	0.38125 (18)	0.33628 (8)	0.0141 (4)
H7	0.126186	0.403857	0.342587	0.017 [*]
C8	0.3457 (4)	0.36573 (19)	0.27261 (8)	0.0166 (4)
H8A	0.316483	0.447395	0.253469	0.020 [*]
H8B	0.518678	0.338867	0.265177	0.020 [*]
C9	0.1622 (4)	0.26470 (18)	0.25079 (8)	0.0173 (4)
H9A	−0.009920	0.295034	0.256408	0.021 [*]
H9B	0.188306	0.252973	0.210319	0.021 [*]
C10	0.1951 (4)	0.13650 (19)	0.28051 (8)	0.0162 (4)
C11	0.4691 (4)	0.49179 (18)	0.35926 (8)	0.0154 (4)
H11	0.629931	0.490301	0.338454	0.018 [*]
C12	0.3466 (4)	0.62430 (19)	0.34931 (8)	0.0193 (4)
H12A	0.477402	0.690163	0.348377	0.023 [*]
H12B	0.261254	0.624440	0.312813	0.023 [*]
C13	0.1571 (5)	0.65614 (19)	0.39581 (9)	0.0228 (5)
H13A	0.029372	0.588942	0.398191	0.027 [*]
H13B	0.073222	0.737393	0.387522	0.027 [*]
C14	0.3006 (5)	0.6659 (2)	0.45170 (9)	0.0264 (5)
H14A	0.391867	0.747259	0.452378	0.032 [*]
H14B	0.178798	0.667487	0.482559	0.032 [*]
C15	0.4862 (4)	0.55779 (18)	0.46229 (8)	0.0184 (4)
C17	0.6777 (4)	0.35723 (18)	0.43260 (8)	0.0180 (4)
H17A	0.634221	0.326093	0.470227	0.022 [*]
H17B	0.855915	0.380307	0.432767	0.022 [*]

Comment

The plant *S. flavescens* belongs to Leguminosae family. It is widely distributed in western and central Asia, especially in northwest China [5]. Previous chemical investigations of *S. flavescens* have revealed that matrine-type alkaloids are the main chemical constituents of this plant [6, 7]. Traditionally, the seeds and roots of this plant have been used as traditional Chinese medicine for the treatment of rheumatism, fever, bacterial infection, and heart disease [8]. Because of their

complex stereostructure and diverse biological effects, matrine-type alkaloids have become increasingly attractive targets for both pharmacologists and synthetic chemists.

The title compound was determined structurally as 10-oxyphosphoridine [9], which belongs to a class of quinolizidine alkaloids with unique 6/6/6/6 tetracyclic skeletons. It has the basic skeleton of two valerolactam and two piperidine groups. The C=O bond lengths 1.233(2)–1.234(2) Å, C–N and C–C bond lengths are within normal ranges [10].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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