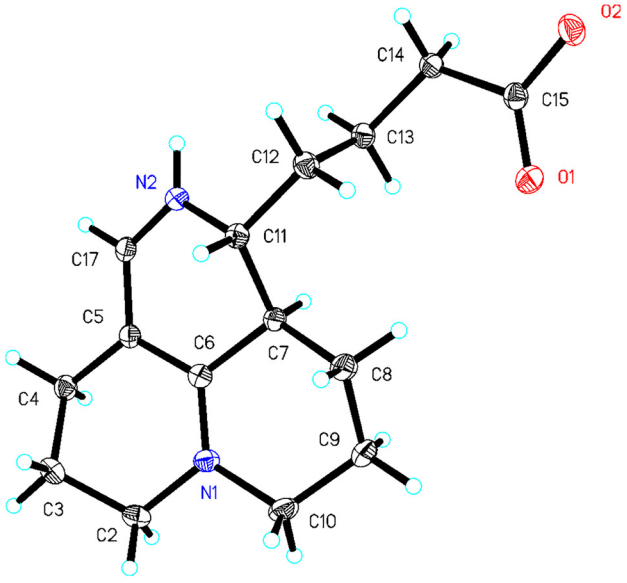
Kai Peng, Ding Luo* and Guang-cun Wang*

Crystal structure of (−)-flavesine H, $C_{15}H_{22}N_2O_2$



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Abstract

$C_{15}H_{22}N_2O_2$, monoclinic, $P2_1$ (no. 4), a = 8.4853(2) Å, b = 19.5759(4) Å, c = 9.0640(2) Å, β = 106.845(2)°, V = 1441.00(6) Å^3, Z = 4, $R_{gt}(F)$ = 0.00374, w$R_{ref}(F^2)$ = 0.0991, T = 150 K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of material

The dried roots of *Sophora flavescens* (25.0 kg) were sourced from Xi'an, Shanxi Province, China. After three extractions with 95 % ethanol, the combined extracts were concentrated under vacuum to yield 1.6 kg of residue. This was suspended in water, adjusted to pH 4 with HCl, and partitioned with chloroform. The aqueous phase was then adjusted to pH 9 with ammonium hydroxide and re-extracted with chloroform, yielding 464 g of chloroform-soluble fraction. Chromatography on macroporous resin produced several fractions, with the title compound isolated from the EtOH/H_2O (10:90) fraction and crystallized from methanol.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.12 × 0.11 × 0.10 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	0.73 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	74.0°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13135, 5610, 0.034
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 5461
$N(param)_{refined}$:	367
Programs:	Olex2,[1] SHELX[2,3]

2 Experimental details

Using Olex2,[1] the structure was solved with the ShelXT[2] structure solution program and refined with the ShelXL[3] refinement package.

3 Comment

Matrine-type alkaloids are a class of naturally occurring compounds predominantly found in plants of the *Sophora* genus. These alkaloids are characterized by their tetracyclic quinolizidine core structure.[4] Recent advancements in pharmacological research have highlighted the diverse biological activities.[5,6] Due to their intricate stereostructure and diverse biological activities, matrine-type alkaloids have garnered significant interest as promising targets for both pharmacological studies and synthetic chemistry endeavors.

The title compound was identified as (−)-flavesine H, the enantiomer of the previously reported (+)-flavesine H.[6] It belongs to the matrine-type alkaloids, distinguished by an open-loop ring D. Notably, the single-crystal structure of (+-)-flavesine H has not yet been reported. The C=O bond lengths 1.236(3)–1.277(3) Å, C–N bond lengths

***Corresponding authors: Ding Luo** and **Guang-cun Wang**, Department of Anesthesiology, The Fifth Affiliated Hospital of Jinan University, Heyuan 517000, P.R. China, E-mail: luoding@jnu.edu.cn (D. Luo), 258796520@qq.com (G.-c. Wang). https://orcid.org/0009-0009-3428-819X (D. Luo)
Kai Peng, Department of Anesthesiology, The Fifth Affiliated Hospital of Jinan University, Heyuan 517000, 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}
O1	0.0677 (2)	0.64406 (9)	0.4676 (2)	0.0312 (4)
O2	−0.0398 (2)	0.54071 (10)	0.3946 (2)	0.0325 (4)
N1	0.2342 (2)	0.88293 (10)	1.0100 (2)	0.0250 (4)
N2	0.0937 (3)	0.68675 (10)	1.0753 (2)	0.0253 (4)
H2	0.080730	0.642775	1.088276	0.030*
C2	0.2620 (3)	0.93510 (12)	1.1325 (3)	0.0284 (5)
H2A	0.201969	0.977264	1.088893	0.034*
H2B	0.380828	0.946217	1.168477	0.034*
C3	0.2061 (3)	0.91228 (13)	1.2692 (3)	0.0302 (5)
H3A	0.084373	0.913547	1.241358	0.036*
H3B	0.249871	0.944057	1.356499	0.036*
C4	0.2662 (3)	0.84011 (12)	1.3181 (3)	0.0273 (5)
H4A	0.387929	0.839547	1.357058	0.033*
H4B	0.221542	0.824367	1.401708	0.033*
C5	0.2091 (3)	0.79350 (12)	1.1809 (3)	0.0227 (4)
C6	0.2021 (3)	0.81839 (12)	1.0334 (3)	0.0221 (4)
C7	0.1687 (3)	0.76709 (12)	0.9019 (3)	0.0228 (4)
H7	0.272091	0.740381	0.913726	0.027*
C8	0.1234 (3)	0.80092 (13)	0.7436 (3)	0.0312 (5)
H8A	0.124652	0.766616	0.663669	0.037*
H8B	0.011184	0.820487	0.719391	0.037*
C9	0.2471 (4)	0.85701 (14)	0.7445 (3)	0.0357 (6)
H9A	0.359563	0.837566	0.771992	0.043*
H9B	0.223177	0.877524	0.640453	0.043*
C10	0.2374 (4)	0.91131 (13)	0.8601 (3)	0.0349 (6)
H10A	0.333400	0.942130	0.876549	0.042*
H10B	0.136731	0.938913	0.817096	0.042*
C11	0.0367 (3)	0.71732 (12)	0.9208 (3)	0.0238 (5)
H11	−0.064506	0.744516	0.915694	0.029*
C12	−0.0116 (3)	0.66129 (13)	0.7994 (3)	0.0268 (5)
H12A	−0.060624	0.683013	0.697835	0.032*
H12B	−0.097998	0.632781	0.822312	0.032*
C13	0.1278 (3)	0.61467 (12)	0.7872 (3)	0.0261 (5)
H13A	0.176239	0.591787	0.887508	0.031*
H13B	0.215004	0.642584	0.764062	0.031*
C14	0.0691 (3)	0.56065 (12)	0.6621 (3)	0.0273 (5)
H14A	−0.030474	0.538626	0.676404	0.033*
H14B	0.155336	0.525083	0.677879	0.033*
C15	0.0285 (3)	0.58544 (12)	0.4953 (3)	0.0246 (5)
C17	0.1647 (3)	0.72619 (12)	1.1945 (3)	0.0240 (5)
H17	0.186370	0.707345	1.294986	0.029*
O1A	0.2960 (3)	0.33844 (11)	0.1245 (2)	0.0472 (5)
O2A	0.2134 (4)	0.44442 (12)	0.1513 (3)	0.0557 (7)
N1A	0.4754 (3)	0.10655 (10)	−0.3462 (3)	0.0285 (4)
N2A	0.2809 (2)	0.29788 (10)	−0.4760 (2)	0.0249 (4)
H2AA	0.254774	0.341081	−0.496205	0.030*
C2A	0.5439 (4)	0.06458 (14)	−0.4479 (3)	0.0368 (6)
H2AB	0.516018	0.016033	−0.437788	0.044*
H2AC	0.665415	0.068760	−0.414701	0.044*
C3A	0.4784 (4)	0.08591 (14)	−0.6156 (3)	0.0370 (6)
H3AA	0.359780	0.074700	−0.654741	0.044*
H3AB	0.537190	0.060645	−0.678211	0.044*
C4A	0.5031 (3)	0.16262 (15)	−0.6300 (3)	0.0331 (5)
H4AA	0.622136	0.173228	−0.602999	0.040*
H4AB	0.450926	0.177527	−0.737429	0.040*
C5A	0.4265 (3)	0.19960 (12)	−0.5227 (3)	0.0248 (5)
C6A	0.4203 (3)	0.16933 (12)	−0.3843 (3)	0.0230 (4)
C7A	0.3672 (3)	0.21316 (12)	−0.2689 (3)	0.0234 (5)
H7A	0.464891	0.240971	−0.212318	0.028*
C8A	0.3144 (3)	0.17164 (14)	−0.1485 (3)	0.0313 (5)
H8AA	0.303139	0.202139	−0.064982	0.038*
H8AB	0.206226	0.150008	−0.196675	0.038*
C9A	0.4423 (4)	0.11699 (15)	−0.0821 (3)	0.0368 (6)
H9AA	0.549619	0.138795	−0.031290	0.044*
H9AB	0.408731	0.090203	−0.003620	0.044*
C10A	0.4600 (4)	0.06985 (14)	−0.2091 (3)	0.0366 (6)
H10C	0.558543	0.040806	−0.168267	0.044*
H10D	0.362777	0.039426	−0.240138	0.044*
C11A	0.2329 (3)	0.26319 (12)	−0.3523 (3)	0.0231 (4)
H11A	0.130395	0.236452	−0.400269	0.028*
C12A	0.1919 (3)	0.31801 (13)	−0.2476 (3)	0.0278 (5)
H12C	0.101107	0.346790	−0.310269	0.033*
H12D	0.152132	0.295109	−0.167830	0.033*
C13A	0.3365 (3)	0.36402 (14)	−0.1682 (3)	0.0324 (6)
H13C	0.380657	0.385045	−0.247381	0.039*
H13D	0.424956	0.335786	−0.100272	0.039*
C14A	0.2900 (3)	0.42046 (13)	−0.0726 (3)	0.0288 (5)
H14C	0.377674	0.455573	−0.050405	0.035*
H14D	0.187392	0.442392	−0.135672	0.035*
C15A	0.2640 (3)	0.39783 (13)	0.0797 (3)	0.0305 (5)
C17A	0.3626 (3)	0.26502 (12)	−0.5568 (3)	0.0247 (5)
H17A	0.379115	0.287390	−0.644186	0.030*
O3	0.0057 (3)	0.55455 (9)	0.1139 (2)	0.0358 (4)
H3D	0.070130	0.521348	0.113187	0.054*
H3C	−0.005520	0.554018	0.204122	0.054*
O4	0.1809 (3)	0.42944 (10)	0.4334 (2)	0.0386 (5)
H4C	0.190543	0.433391	0.343010	0.058*
H4D	0.110787	0.459768	0.438147	0.058*

1.314(3)–1.476(3) Å, C=C bond lengths 1.386(3)–1.408(3) Å and C–C bond lengths 1.507(3)–1.547(2) Å, which were derived from the title structure are within normal ranges.[7]

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

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

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