Yi-Xia Gong* and Yi-Ding Geng*

The crystal structure of 2-[(S)-1-(naphthalen-1-yl)ethyl]-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one, C₁₉H₂₀NO₂

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.24 × 0.22 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	Bruker, φ and ω
$\theta_{\max}$, completeness:	28.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6593, 3365, 0.028
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2368
$N(\text{param})_{\text{refined}}$:	222
Programs:	Bruker [1], SHELX [2]

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Abstract

C₁₉H₂₀NO₂, monoclinic, $P2_1$ (no. 4), $a = 6.685(5)$ Å, $b = 8.878(7)$ Å, $c = 13.239(9)$ Å, $\beta = 97.720(2)^\circ$, $V = 778.75(10)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0423$, $wR_{\text{ref}}(F^2) = 0.0970$, $T = 296(2)$ 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

All chemicals, reagents and solvents are of analytical grade and are commercially available. Preparation of 2-[(S)-1-(naphthalen-1-yl)ethyl]-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one: (S)-3-chloro-*N*-(furan-2-ylmethyl)-*N*-(1-

(naphthalen-1-yl)ethyl)propanamide (5 g, 14.63 mmol) was dissolved in DMF (100 mL) at room temperature, followed by potassium carbonate (6.07 g, 43.89 mmol) and tetrabutylammonium iodide (0.54 g, 1.463 mmol) in order. Then the mixture reacted under stirring at 110 °C for 2 h. Subsequently, the reaction mixture was poured into water and extracted with dichloromethane. The organic phase was dehydrated with anhydrous sodium sulfate and concentrated at reduced pressure to afford a yellow liquid. The crude mixture was purified by silica gel column chromatography, eluted with petroleum ether and ethyl acetate (5:1) to give a white solid (1.48 g, yield 82.65%). ¹H NMR (400 MHz, CDCl₃) δ 8.07 (d, $J = 8.3$ Hz, 1H), 7.93–7.66 (m, 2H), 7.47 (ddd, $J = 19.9, 15.3, 7.3$ Hz, 4H), 6.48–6.07 (m, 3H), 4.88 (d, $J = 4.3$ Hz, 1H), 3.82 (d, $J = 11.6$ Hz, 1H), 3.09 (d, $J = 11.6$ Hz, 1H), 2.47 (dd, $J = 8.8, 3.5$ Hz, 1H), 2.19 (dd, $J = 7.9, 3.8$ Hz, 1H), 1.71 (d, $J = 6.9$ Hz, 3H), 1.61–1.50 (m, 1H). Crystals were grown in hexane and ethyl acetate (3:1) at room temperature.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

The class of chemical compounds the title compound belongs to has members that are important intermediates for

*Corresponding authors: Yi-Xia Gong and Yi-Ding Geng, College of Pharmacy, Jiamusi University, Jiamusi, 154007, P. R. China, E-mail: gongyixia_2006@163.com (Y.-X. Gong), gengyiding86@163.cn (Y.-D. Geng). <https://orcid.org/0000-0001-8629-2494> (Y.-X. Gong). <https://orcid.org/0000-0002-0719-9476> (Y.-D. Geng)

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

Atom	x	y	z	U _{iso} */U _{eq}
C1	0.9230 (4)	0.3512 (4)	0.34228 (19)	0.0492 (7)
C2	0.7237 (4)	0.3731 (4)	0.2925 (2)	0.0556 (8)
H2	0.6826	0.4691	0.2706	0.067*
C3	0.5911 (5)	0.2560 (4)	0.2761 (2)	0.0678 (9)
H3	0.4613	0.2738	0.2434	0.081*
C4	0.6459 (6)	0.1112 (5)	0.3072 (3)	0.0781 (10)
H4	0.5557	0.0319	0.2928	0.094*
C5	0.8309 (6)	0.0860 (4)	0.3584 (3)	0.0748 (10)
H5	0.8654	−0.0110	0.3809	0.090*
C6	0.9756 (5)	0.2043 (4)	0.3791 (2)	0.0579 (8)
C7	1.1658 (6)	0.1796 (5)	0.4347 (2)	0.0737 (10)
H7	1.1995	0.0842	0.4605	0.088*
C8	1.2999 (6)	0.2917 (5)	0.4514 (2)	0.0740 (11)
H8	1.4245	0.2735	0.4898	0.089*
C9	1.2547 (5)	0.4389 (5)	0.4111 (2)	0.0639 (9)
H9	1.3516	0.5145	0.4215	0.077*
C10	1.0691 (4)	0.4696 (4)	0.35725 (18)	0.0486 (7)
C11	1.0170 (4)	0.6283 (4)	0.31796 (18)	0.0464 (6)
H11	0.895 (4)	0.660 (3)	0.3462 (17)	0.040 (6)*
C12	1.1810 (5)	0.7455 (4)	0.3465 (2)	0.0655 (9)
H12A	1.1379	0.8407	0.3167	0.098*
H12B	1.3028	0.7144	0.3215	0.098*
H12C	1.2058	0.7555	0.4193	0.098*
C13	0.7786 (4)	0.6938 (3)	0.16427 (18)	0.0431 (6)
C14	0.7778 (4)	0.6966 (3)	0.05050 (18)	0.0424 (6)
H14	0.804 (4)	0.794 (4)	0.032 (2)	0.053 (8)*
C15	0.9452 (3)	0.5826 (3)	0.03481 (18)	0.0419 (6)
C16	1.0856 (3)	0.5801 (4)	0.13347 (17)	0.0504 (7)
H16A	1.1370	0.4795	0.1493	0.061*
H16B	1.1983	0.6485	0.1314	0.061*
C17	0.9991 (4)	0.6064 (4)	−0.07113 (19)	0.0497 (7)
H17	1.1173	0.6476	−0.0891	0.060*
C18	0.8429 (4)	0.5564 (4)	−0.1322 (2)	0.0539 (8)
H18	0.8268	0.5571	−0.2031	0.065*
C19	0.6949 (5)	0.4976 (4)	−0.0660 (2)	0.0563 (8)
H19	0.599 (4)	0.417 (4)	−0.088 (2)	0.059 (8)*
C20	0.5937 (4)	0.6306 (4)	−0.01717 (19)	0.0579 (8)
H20A	0.5351	0.7024	−0.0680	0.069*
H20B	0.4910	0.5965	0.0228	0.069*
N1	0.9546 (3)	0.6306 (3)	0.20728 (14)	0.0433 (5)
O1	0.6447 (3)	0.7393 (3)	0.21102 (14)	0.0581 (6)
O2	0.8294 (3)	0.4453 (2)	0.02196 (14)	0.0536 (5)

the synthesis of isoindolinone compounds and can be used to prepare fluoresce and biologically active molecules [3, 4]. They can participate in the synthesis of alkaloids Aspernidine A and Aspernidine B for the preparation of anticancer drugs and antiviral drugs [5, 6]. At the same time, some of them are materials for the preparation of oxanorbornene analogs useful in rubber industry [7].

The structure of the title compound was elucidated by spectroscopic methods (NMR) and X-ray diffraction. It

contained one epoxyisoindol ring and one naphthyl moiety. All bond lengths and angles in the crystal structure are within the normal range [8]. The compound exhibits weak intramolecular hydrogen bonding of the C–H...O type. The packing of crystal structure is dominated by weak intermolecular interactions, and the intermolecular packing is stabilized by the van der Waals forces.

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References

1. Bruker. *APEX2, SAINT SADABS*: Madison, USA, 2008.
2. Sheldrick G. M. A short history of SHELX. *Acta Crystallogr.* 2008, *A64*, 112–122.
3. Benson N., Suleiman O., Odoh S. O., Woydziak Z. R. Pyrazole, imidazole, and isoindolone dipyrinone analogues: pH-dependent fluorophores that red-shift emission frequencies in a basic solution. *J. Org. Chem.* 2019, *84*, 11856–11862.
4. Yao C., Bao Y. S., Lu T., Zhou Q. F. Stereoselective synthesis of functionalized benzooxazepino[5,4a]isoindolone derivatives via cesium carbonate catalyzed formal [5 + 2] annulation of 2(2-hydroxyphenyl) isoindoline-1,3-dione with allenates. *Org. Lett.* 2018, *20*, 2152–2155.
5. Scherlach K., Schuermann J., Dahse H. M., Hertweck C. Aspernidine A and B, prenylated isoindolinone alkaloids from the model fungus *Aspergillus nidulans*. *J. Antibiot.* 2010, *63*, 375–377.
6. Zhang G. J., Sun S. W., Zhu T. J., Lin Z. J., Gu J. Y., Li D. H., Gu Q. Q. Antiviral isoindolone derivatives from an endophytic fungus *Emerella* sp. associated with *Aegiceras corniculatum*. *Phytochemistry* 2011, *72*, 1436–1442.
7. Minoru I., Masato O., Makoto S. Synthesis and domino metathesis of functionalized 7-oxanorbornene analogs toward *cis*-fused heterocycles. *Tetrahedron* 2008, *64*, 2740–2749.
8. Bai Y. F., Yuan L., Chen Y., Wang L. J., Wang C., Sun T. M. Synthesis, crystal and calculated structure, and biological activity of 2-((6-bromo-2-methoxyquinolin-3-yl) (phenyl)methyl)-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one. *J. Chem. Crystallogr.* 2012, *42*, 318–322.