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

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Abstract

C₁₉H₂₀NO₂, monoclinic, *P*₂₁ (no. 4), *a* = 6.6936(4) Å, *b* = 8.8892(6) Å, *c* = 13.2577(8) Å, *β* = 97.740(1)°, *V* = 781.66(8) Å³, *Z* = 2, *R*_{gt}(*F*) = 0.0387, *wR*_{ref}(*F*²) = 0.0741, *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 and solvents are of analytical grade and are commercially available. Preparation: (*R*)-3-chloro-*N*-

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.24 × 0.22 × 0.20 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
<i>μ</i> :	0.08 mm ^{−1}
Diffractometer, scan mode:	<i>φ</i> and <i>ω</i>
<i>θ</i> _{max} , completeness:	29.1°, 99 %
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	6683, 3711, 0.016
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2σ(<i>I</i> _{obs}), 3176
<i>N</i> (<i>param</i>) _{refined} :	209
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], Olex2 [4]

(furan-2-ylmethyl)-*N*-(1-(naphthalen-1-yl)ethyl)prop-
anamide (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. 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 deep yellow liquid. The crude product
was purified by silica gel column chromatography, eluted
with petroleum ether and ethyl acetate (5:1) to give a white
solid (3.35 g, yield 75.3%). ¹H NMR (400 MHz, CDCl₃) *δ*
8.07 (d, *J* = 8.3 Hz, 1H), 7.85–7.22 (m, 2H), 7.46 (ddd,
J = 19.9, 15.3, 7.3 Hz, 4H), 6.45–6.04 (m, 3H), 4.87 (d,
J = 4.3 Hz, 1H), 3.81 (d, *J* = 11.6 Hz, 1H), 3.08 (d, *J* = 11.6 Hz,
1H), 2.46 (dd, *J* = 8.8, 3.5 Hz, 1H), 2.18 (dd, *J* = 7.9, 3.8 Hz,
1H), 1.73 (d, *J* = 6.9 Hz, 3H), 1.63–1.52 (m, 1H). Crystals were
grown in hexane and ethyl acetate (3:1) at room
temperature.

Experimental details

Hydrogen atoms were placed in their geometrically ideal-
ized positions and constrained to ride on their parent
atoms.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O1	0.6704 (2)	0.18875 (18)	0.47805 (11)	0.0523 (4)
N1	0.5458 (2)	0.3737 (2)	0.29279 (11)	0.0426 (4)
C1	0.7767 (4)	0.1156 (3)	0.20764 (17)	0.0544 (6)
H1	0.8179	0.2115	0.2295	0.065*
O2	0.8550 (2)	0.4824 (2)	0.28875 (11)	0.0579 (4)
C2	0.9094 (4)	−0.0013 (4)	0.2240 (2)	0.0662 (7)
H2	1.0392	0.0164	0.2566	0.079*
C3	0.8540 (5)	−0.1458 (4)	0.1929 (2)	0.0765 (8)
H3	0.9441	−0.2251	0.2071	0.092*
C4	0.6689 (5)	−0.1713 (3)	0.1418 (2)	0.0723 (8)
H4	0.6340	−0.2682	0.1195	0.087*
C5	0.5254 (4)	−0.0527 (3)	0.12113 (17)	0.0575 (6)
C6	0.5772 (3)	0.0941 (3)	0.15800 (15)	0.0476 (5)
C7	0.4307 (3)	0.2130 (3)	0.14280 (14)	0.0475 (5)
C8	0.2458 (4)	0.1817 (4)	0.08900 (17)	0.0623 (7)
H8	0.1490	0.2572	0.0787	0.075*
C9	0.1997 (5)	0.0349 (4)	0.0486 (2)	0.0739 (9)
H9	0.0751	0.0168	0.0103	0.089*
C10	0.3350 (5)	−0.0776 (4)	0.06551 (19)	0.0719 (8)
H10	0.3016	−0.1730	0.0399	0.086*
C11	0.4832 (3)	0.3711 (3)	0.18208 (14)	0.0452 (5)
H11	0.6001	0.4036	0.1504	0.054*
C12	0.3188 (4)	0.4885 (3)	0.15422 (19)	0.0634 (7)
H12A	0.2903	0.4965	0.0815	0.095*
H12B	0.3637	0.5841	0.1823	0.095*
H12C	0.1988	0.4587	0.1813	0.095*
C13	0.4139 (3)	0.3236 (3)	0.36625 (15)	0.0487 (5)
H13A	0.3621	0.2232	0.3503	0.058*
H13B	0.3017	0.3921	0.3683	0.058*
C14	0.5546 (3)	0.3257 (2)	0.46491 (15)	0.0408 (4)
C15	0.5007 (3)	0.3496 (3)	0.57106 (15)	0.0483 (5)
H15	0.3826	0.3908	0.5889	0.058*
C16	0.6571 (4)	0.2995 (3)	0.63246 (17)	0.0539 (6)
H16	0.6732	0.3004	0.7032	0.065*
C17	0.8057 (4)	0.2403 (3)	0.56569 (18)	0.0555 (6)
H17	0.8995	0.1641	0.5976	0.067*
C18	0.9061 (3)	0.3745 (3)	0.51712 (16)	0.0557 (6)
H18A	0.9639	0.4461	0.5681	0.067*
H18B	1.0092	0.3410	0.4773	0.067*
C19	0.7226 (3)	0.4403 (3)	0.44932 (14)	0.0411 (5)
H19	0.6897	0.5410	0.4723	0.049*
C20	0.7215 (3)	0.4364 (3)	0.33558 (15)	0.0420 (4)

Comment

2-[(R)-1-(naphthalen-1-yl)ethyl]-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one is an intermediate for the synthesis of some isoindolinone compounds, which can be used in the fields of medicine, organic synthesis and fine chemicals [5–7]. It can participate in the synthesis of

alkaloids Aspernidine A and Aspernidine B for the preparation of antidiabetic drugs [8]. In addition, it can be used to prepare potent histone deacetylase inhibitors with antiproliferative activity human liver microsome (HLM) [9]. 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 [10, 11].

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