Crystal structure of (2*SR*,1'*RS*)-3-(1-naphthyl)-2-(2-oxocyclopentyl)-propionic acid methyl ester, C₁₉H₂₀O₃

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Abstract

C₁₉H₂₀O₃, monoclinic, *P*12₁/*n*1 (no. 14), *a* = 9.214(2) Å, *b* = 18.470(4) Å, *c* = 10.194(2) Å, β = 113.451(4)°, *V* = 1591.6 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.042, *wR*_{ref}(*F*²) = 0.123, *T* = 173 K.

Source of material

The title compound was prepared by alkylation of the lithium enolate of 1-trimethylsilyloxy-bicyclo[3.1.0]hexane-6-carboxylic acid methyl ester with 1-naphthyl iodomethane, followed by ring cleavage with fluoride and HPLC separation of the diastereomers [1–4]. The diastereoisomer described here was crystallized from *n*-hexane/ethyl acetate; m.p. 336–337 K.

Discussion

The crystal structure proves the constitution and configuration of the title compound. It also shows a staggered conformation with respect to C12 and C15 placing the cyclopentanone carbonyl group and the naphthyl unit in fairly close proximity which is important for subsequent cyclization reactions [5].

Table 1. Data collection and handling.

Crystal:	colorless plate, size 0.13 × 0.46 × 0.78 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	0.83 cm ^{−1}
Diffractionmeter, scan mode:	Bruker SMART CCD, φ/ω with Δω = 0.3°
2θ _{max} :	61.14°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	19463, 4873
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 3268
<i>N</i> (<i>param</i>) _{refined} :	200
Programs:	SHELXS-97 [6], SHELXL-97 [7], PLATON [8]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(2)	4e	0.6979	0.0034	0.0027	0.047
H(3)	4e	0.6062	−0.1139	−0.0650	0.058
H(4)	4e	0.7748	−0.2096	0.0101	0.055
H(6)	4e	1.0456	−0.2582	0.1437	0.053
H(7)	4e	1.3058	−0.2399	0.2867	0.055
H(8)	4e	1.3993	−0.1235	0.3631	0.050
H(9)	4e	1.2325	−0.0262	0.2934	0.039
H(11A)	4e	0.9092	0.0886	0.1018	0.034
H(11B)	4e	1.0836	0.0614	0.1835	0.034
H(12)	4e	1.0199	0.0328	0.3864	0.029
H(14A)	4e	0.5562	0.0669	0.3607	0.071
H(14B)	4e	0.5804	−0.0114	0.4249	0.071
H(14C)	4e	0.5299	−0.0004	0.2601	0.071
H(15)	4e	0.9946	0.1797	0.2991	0.035
H(17A)	4e	1.3353	0.2341	0.5683	0.061
H(17B)	4e	1.3127	0.1703	0.6614	0.061
H(18A)	4e	1.0873	0.2766	0.5116	0.065
H(18B)	4e	1.1172	0.2437	0.6633	0.065
H(19A)	4e	0.8804	0.1951	0.4575	0.059
H(19B)	4e	0.9899	0.1366	0.5642	0.059

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1)	4e	1.30010(9)	0.11556(5)	0.40524(9)	0.0280(4)	0.0418(5)	0.0500(5)	−0.0019(3)	0.0172(4)	−0.0061(4)
O(2)	4e	0.75696(8)	0.02123(4)	0.37399(8)	0.0229(4)	0.0395(4)	0.0348(4)	0.0001(3)	0.0120(3)	0.0108(3)
O(3)	4e	0.6917(1)	0.10817(5)	0.2086(1)	0.0283(4)	0.0520(5)	0.0732(7)	0.0133(4)	0.0178(4)	0.0354(5)
C(1)	4e	0.9231(1)	−0.02176(6)	0.1236(1)	0.0283(5)	0.0353(5)	0.0222(5)	−0.0058(4)	0.0097(4)	−0.0007(4)
C(2)	4e	0.7679(1)	−0.03526(7)	0.0354(1)	0.0314(6)	0.0511(7)	0.0295(6)	−0.0056(5)	0.0057(5)	0.0015(5)
C(3)	4e	0.7121(2)	−0.10619(8)	−0.0065(1)	0.0370(7)	0.0618(9)	0.0366(7)	−0.0212(6)	0.0056(6)	−0.0080(6)
C(4)	4e	0.8127(2)	−0.16300(7)	0.0381(1)	0.0513(8)	0.0478(7)	0.0376(7)	−0.0255(6)	0.0175(6)	−0.0129(6)
C(5)	4e	0.9743(2)	−0.15273(6)	0.1267(1)	0.0453(7)	0.0339(6)	0.0318(6)	−0.0125(5)	0.0210(5)	−0.0074(5)
C(6)	4e	1.0818(2)	−0.21144(6)	0.1726(2)	0.0631(9)	0.0289(6)	0.0515(8)	−0.0072(6)	0.0345(7)	−0.0083(5)
C(7)	4e	1.2369(2)	−0.20072(7)	0.2583(2)	0.0546(8)	0.0360(6)	0.0569(8)	0.0090(6)	0.0325(7)	0.0022(6)
C(8)	4e	1.2933(2)	−0.13043(7)	0.3040(1)	0.0351(6)	0.0441(7)	0.0473(7)	0.0043(5)	0.0179(6)	−0.0004(6)
C(9)	4e	1.1935(1)	−0.07228(6)	0.2621(1)	0.0302(6)	0.0329(5)	0.0355(6)	−0.0032(4)	0.0142(5)	−0.0034(5)
C(10)	4e	1.0308(1)	−0.08106(6)	0.1713(1)	0.0314(5)	0.0306(5)	0.0244(5)	−0.0061(4)	0.0143(4)	−0.0037(4)
C(11)	4e	0.9752(1)	0.05467(6)	0.1732(1)	0.0280(5)	0.0308(5)	0.0271(5)	−0.0010(4)	0.0112(4)	0.0044(4)
C(12)	4e	0.9636(1)	0.07052(5)	0.3173(1)	0.0213(5)	0.0230(4)	0.0286(5)	0.0025(4)	0.0097(4)	0.0053(4)
C(13)	4e	0.7901(1)	0.06949(5)	0.2926(1)	0.0241(5)	0.0266(5)	0.0342(6)	0.0023(4)	0.0123(4)	0.0053(4)
C(14)	4e	0.5919(1)	0.01889(8)	0.3532(2)	0.0253(6)	0.0667(9)	0.0525(8)	−0.0030(6)	0.0173(6)	0.0178(7)
C(15)	4e	1.0315(1)	0.14489(5)	0.3782(1)	0.0300(5)	0.0229(5)	0.0372(6)	0.0015(4)	0.0169(5)	0.0020(4)
C(16)	4e	1.2113(1)	0.14820(6)	0.4453(1)	0.0311(6)	0.0283(5)	0.0336(6)	−0.0049(4)	0.0136(5)	0.0010(4)
C(17)	4e	1.2620(2)	0.19764(8)	0.5736(2)	0.0529(8)	0.0550(8)	0.0448(8)	−0.0169(7)	0.0205(7)	−0.0170(6)
C(18)	4e	1.1105(2)	0.23253(7)	0.5680(2)	0.072(1)	0.0387(7)	0.068(1)	−0.0155(7)	0.0446(8)	−0.0221(6)
C(19)	4e	0.9858(2)	0.17439(7)	0.4968(2)	0.0538(8)	0.0391(6)	0.071(1)	−0.0086(6)	0.0432(8)	−0.0192(6)

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