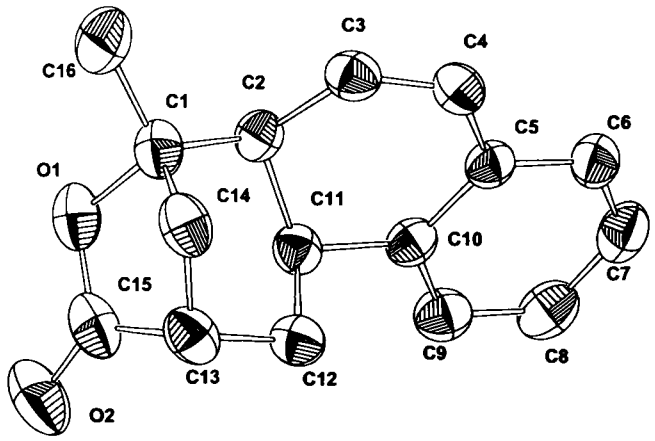
Crystal structure of 2*SR*,5*SR*,5a*SR*,11b*SR*-5-methyl-1,5,5a,11b-tetrahydro-2,5-methanonaphtho[2,1-*c*]oxepin-3(2*H*)-one, $C_{16}H_{16}O_2$

O. Rademacher*[I], R. Zimmer[II], C. U. Dinesh[II] and H.-U. Reißig[II]

[I] Technische Universität Dresden, Institut für Anorganische Chemie, D-01062 Dresden, Germany
[II] Freie Universität Berlin, Institut für Chemie - Organische Chemie, Takustr. 3, D-14195 Berlin, Germany

Received June 6, 2002, accepted and available on-line August 13, 2002; CCDC-No. 1267/875



Abstract

$C_{16}H_{16}O_2$, monoclinic, $P12_1/n1$(No. 14), a = 12.475(4) Å, b = 7.515(5) Å, c = 13.259(4) Å, β = 95.55(3)°, V = 1237.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.045, $wR_{ref}(F^2)$ = 0.119, T = 293 K.

Source of material

The title compound was obtained by samarium diiodide induced reaction of methyl 2*RS*-2-(1-naphthylmethyl)-4-oxopentanoate in 34% yield (purified by chromatography on silica gel, colourless crystals, mp 453 K – 455 K, recrystallized from hexane/ethyl acetate) [1].

Discussion

The crystal structure proves the constitution and configuration of the title compound. Due to the *cis*-arrangement of the 1-hydroxy and the 15-carboxy group formation of a γ-lactone was possible. This γ-lactone ring and the bridge-head hydrogens at C2 and C11 are in *cis*-arrangement. Bonding distances and bond angles show normal values.

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.25 × 0.40 × 0.60 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.84 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	51.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2518, 2415
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1855
$N(param)_{refined}$:	163
Programs:	SHELXS-97 [2], SHELXL-97 [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	0.3667	0.3495	0.6396	0.046
H(3)	4*e*	0.4871	0.1532	0.5368	0.050
H(4)	4*e*	0.4857	0.3151	0.3956	0.050
H(6)	4*e*	0.4150	0.5843	0.2985	0.054
H(7)	4*e*	0.2779	0.7820	0.2541	0.060
H(8)	4*e*	0.1199	0.7669	0.3313	0.063
H(9)	4*e*	0.1019	0.5578	0.4564	0.056
H(11)	4*e*	0.1913	0.3549	0.5846	0.045
H(12A)	4*e*	0.1013	0.1731	0.4671	0.055
H(12B)	4*e*	0.2082	0.1057	0.4266	0.055
H(13)	4*e*	0.1330	–0.1147	0.5366	0.058
H(14A)	4*e*	0.3340	–0.0815	0.5287	0.058
H(14B)	4*e*	0.2999	–0.1846	0.6248	0.058
H(16A)	4*e*	0.4509	0.1528	0.7684	0.096
H(16B)	4*e*	0.4917	0.0114	0.6940	0.096
H(16C)	4*e*	0.4158	–0.0468	0.7756	0.096

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*e*	0.3349(1)	0.0828(2)	0.6559(1)	0.0495(9)	0.0460(9)	0.0404(8)	0.0059(7)	0.0107(7)	0.0039(7)
C(2)	4*e*	0.3452(1)	0.2525(2)	0.5925(1)	0.0425(8)	0.0358(8)	0.0352(7)	0.0013(6)	0.0021(6)	–0.0014(6)
C(3)	4*e*	0.4314(1)	0.2337(2)	0.5212(1)	0.0363(7)	0.0406(8)	0.0491(9)	0.0026(6)	0.0038(6)	0.0017(7)
C(4)	4*e*	0.4300(1)	0.3286(2)	0.4368(1)	0.0375(8)	0.0448(9)	0.0438(8)	0.0011(6)	0.0093(6)	–0.0007(7)
C(5)	4*e*	0.3432(1)	0.4539(2)	0.4070(1)	0.0392(7)	0.0381(8)	0.0324(7)	–0.0033(6)	0.0000(6)	–0.0041(6)
C(6)	4*e*	0.3523(1)	0.5797(2)	0.3311(1)	0.0493(9)	0.0476(9)	0.0370(8)	–0.0055(7)	0.0031(6)	0.0006(7)
C(7)	4*e*	0.2701(1)	0.6971(2)	0.3039(1)	0.065(1)	0.0458(9)	0.0390(8)	–0.0028(8)	–0.0038(7)	0.0069(7)

* Correspondence author
(e-mail: otto.rademacher@chemie.tu-dresden.de)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(8)	4*e*	0.1761(1)	0.6888(2)	0.3503(1)	0.056(1)	0.0470(9)	0.0517(9)	0.0105(8)	–0.0077(8)	0.0034(8)
C(9)	4*e*	0.1656(1)	0.5634(2)	0.4255(1)	0.0423(8)	0.0473(9)	0.0494(9)	0.0042(7)	0.0024(7)	–0.0018(7)
C(10)	4*e*	0.2484(1)	0.4466(2)	0.4555(1)	0.0388(8)	0.0364(8)	0.0339(7)	0.0014(6)	–0.0006(6)	–0.0049(6)
C(11)	4*e*	0.2360(1)	0.3045(2)	0.5349(1)	0.0367(8)	0.0396(8)	0.0361(7)	0.0031(6)	0.0083(6)	–0.0023(6)
C(12)	4*e*	0.1757(1)	0.1413(2)	0.4870(1)	0.0421(8)	0.0507(9)	0.0459(8)	–0.0075(7)	0.0045(7)	–0.0015(7)
C(13)	4*e*	0.1793(1)	–0.0157(2)	0.5619(1)	0.0506(9)	0.0419(9)	0.055(1)	–0.0089(7)	0.0146(7)	–0.0036(7)
C(14)	4*e*	0.2951(1)	–0.0726(2)	0.5884(1)	0.057(1)	0.0382(8)	0.0537(9)	0.0021(7)	0.0195(8)	0.0025(7)
C(15)	4*e*	0.1512(1)	0.0525(2)	0.6631(1)	0.057(1)	0.0455(9)	0.058(1)	0.0015(8)	0.0226(8)	0.0112(8)
C(16)	4*e*	0.4322(2)	0.0468(3)	0.7302(1)	0.069(1)	0.066(1)	0.055(1)	0.008(1)	–0.0012(9)	0.0218(9)
O(1)	4*e*	0.2427(1)	0.1095(2)	0.71678(8)	0.0642(7)	0.0542(7)	0.0409(6)	0.0041(6)	0.0188(5)	0.0027(5)
O(2)	4*e*	0.0656(1)	0.0636(2)	0.6960(1)	0.0633(8)	0.080(1)	0.086(1)	0.0087(7)	0.0408(7)	0.0158(8)

Acknowledgments. This work was supported by the Volkswagen-Stiftung and the Fonds der Chemischen Industrie.

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