Crystal structure of 5-(1-pentyl)-2,3,3aα,4,5,7aα-hexahydroindene-4α-carboxanilide, $C_{21}H_{29}NO$

S. Kaltia[I], J. Matikainen[I], T. Hase[I] and I. Mutikainen*,[II]

[I] University of Helsinki, Department of Chemistry, Laboratory of Organic Chemistry, P. O. Box 55 (A. I. Virtasen aukio 1), FIN-00014 University of Helsinki, Finland

[II] University of Helsinki, Department of Chemistry, Laboratory of Inorganic Chemistry, P. O. Box 55 (A. I. Virtasen aukio 1), FIN-00014 University of Helsinki, Finland

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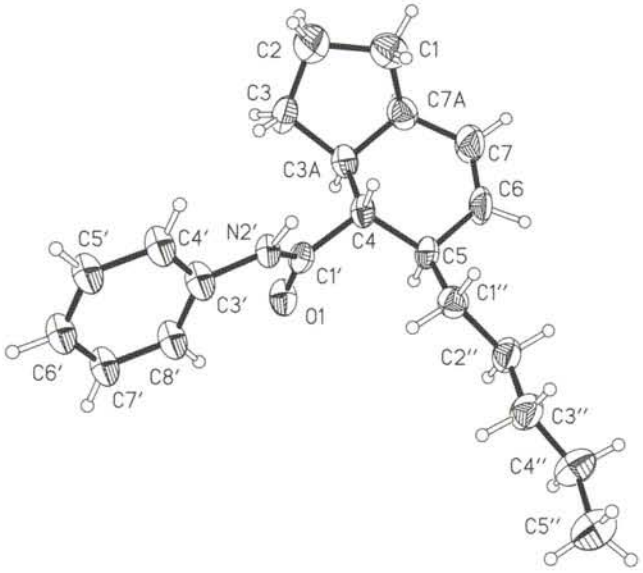

Abstract

$C_{21}H_{29}NO$, orthorhombic, *Pbca* (No. 61), $a = 16.775(3)$ Å, $b = 9.539(2)$ Å, $c = 23.524(5)$ Å, $V = 3764.2$ Å^3, $Z = 8$, $R_{gt}(F) = 0.092$, $wR_{obs}(F^2) = 0.203$, $T = 293$ K.

Source of material

The title compound was prepared by IMDA reaction from ethyl 5-*cis*-10-trans-12-*trans*-octadecatrienoate [1]. The *trans*- and *cis*-fused isomers were then separated through argentation chromatography. The free acid was obtained by refluxing thea trans-fused ester in 1:2 aqueous 0.5 M NaOH:ethanol for 2 h. Removal of the ethanol, acidification (dil. HCl), extraction (ether), and washing and drying of the extracts resulted the free acid. On prolonged standing at 253 K, small crystals were obtained and recrystallized from pentane.

Discussion

The intramolecular Diels-Alder (IMDA) reaction is a highly useful tool in synthetic organic chemistry and has become increasingly important in recent years [2-6]. Stereochemical aspects play a central role in these synthetic applications, as well as in many theoretical studies dealing with the IMDA reaction. Together with the previously reported carboxanilide derivate [7] the title compound can be used as reference compounds for *cis*-fused and *trans*-fused systems synthesized by IMDA reaction [1, 8, 9]. The conformations of the five-membered cyclopentane rings are twisted with puckering parameters $Q = 0.371$ Å and $\varphi(M) = 158.0°$ [10, 11]. These values compare well with those, $Q(M) = 0.462$ and $\varphi(M) = 349.1°$ of related carboxanilide compound [6]. The puckering parameters of the six-membered ring are $Q = 0.392$ Å, $T = 53.4$ and $\varphi(M) = 43.6°$. The resulting canonical conformation for *cis*-fused title compound can be described as $^1H_2$ half-chair slightly distorted towards E_2. The pentyl and the carboxyanilide substituents are oriented in *trans*-position. The puckering of is very similar to the earlier reported carboxanilide compound [7] even though the ring-fusion of the present compound is *cis*.

Table 1. Data collection and handling.

Crystal:	colorless, prismatic, size 0.04 × 0.07 × 0.12 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.66 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC-7S, ω/2θ
$2\theta_{max}$:	48.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3758, 2953
Criterion for F^2_{obs}, $N(hkl)_{gt}$:	$F^2_{obs} > 2\,\sigma(F^2_{obs})$, 850
$N(param)_{refined}$:	181
Programs:	SHELXTL-PC [12], SHELXL-97 [13], TEXSAN [14]

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

Atom	Site	x	y	z	U_{iso}
H(1B)	8*c*	0.0780(7)	–0.054(1)	0.8266(5)	0.133
H(1A)	8*c*	–0.0060(7)	0.008(1)	0.8438(5)	0.133
H(2B)	8*c*	0.0258(8)	–0.032(2)	0.7382(5)	0.193
H(2A)	8*c*	–0.0286(8)	0.096(2)	0.7559(5)	0.193
H(3B)	8*c*	0.0625(6)	0.234(1)	0.7204(4)	0.117
H(3A)	8*c*	0.1225(6)	0.109(1)	0.7130(4)	0.117
H(3AA)	8*c*	0.1387(5)	0.3084(9)	0.7915(4)	0.091
H(4A)	8*c*	0.2126(5)	0.0392(8)	0.7972(3)	0.076
H(5A)	8*c*	0.2709(5)	0.288(1)	0.8489(3)	0.085
H(6A)	8*c*	0.2274(7)	0.164(1)	0.9374(4)	0.120
H(7A)	8*c*	0.0989(7)	0.132(1)	0.9303(4)	0.148
H(7AA)	8*c*	0.0411(6)	0.233(1)	0.8477(4)	0.107
H(2')	8*c*	0.273(1)	–0.014(8)	0.723(1)	0.072
H(4')	8*c*	0.2748(6)	–0.105(1)	0.6309(4)	0.108
H(5')	8*c*	0.3212(6)	–0.094(1)	0.5376(4)	0.108

* Correspondence author (e-mail: ilpo.mutikainen@helsinki.fi)

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(6')	8c	0.3933(6)	0.092(1)	0.5068(4)	0.108
H(7')	8c	0.4186(6)	0.277(1)	0.5667(4)	0.108
H(8')	8c	0.3705(5)	0.272(1)	0.6587(4)	0.108
H(1AA)	8c	0.3309(5)	0.0125(9)	0.8676(4)	0.092
H(1AB)	8c	0.3701(5)	0.1148(9)	0.8237(4)	0.092
H(2AB)	8c	0.3996(6)	0.270(1)	0.9007(4)	0.108
H(2AA)	8c	0.3687(6)	0.154(1)	0.9426(4)	0.108

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(3AB)	8c	0.4709(6)	0.004(1)	0.9148(4)	0.122
H(3AC)	8c	0.5000(6)	0.113(1)	0.8693(4)	0.122
H(4AB)	8c	0.5349(6)	0.268(1)	0.9432(5)	0.139
H(4AA)	8c	0.5091(6)	0.155(1)	0.9878(5)	0.139
H(5BB)	8c	0.6099(7)	0.006(1)	0.950(4)	0.164
H(5AB)	8c	0.644(2)	0.140(7)	0.980(2)	0.164
H(5AA)	8c	0.639(2)	0.133(7)	0.914(2)	0.164

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)	8c	0.0424(7)	0.026(1)	0.8225(5)	0.088(8)	0.083(8)	0.096(8)	–0.002(7)	0.005(7)	0.009(7)
C(2)	8c	0.0240(8)	0.055(2)	0.7600(5)	0.16(1)	0.14(1)	0.09(1)	–0.06(1)	–0.011(9)	0.003(9)
C(3)	8c	0.0870(6)	0.155(1)	0.7396(4)	0.083(7)	0.102(9)	0.049(6)	0.012(7)	–0.014(6)	–0.002(6)
C(3A)	8c	0.1344(5)	0.2060(9)	0.7924(4)	0.077(7)	0.054(6)	0.051(6)	0.007(5)	0.003(5)	0.006(5)
C(4)	8c	0.2179(5)	0.1415(8)	0.7968(3)	0.076(7)	0.039(5)	0.037(5)	0.005(5)	0.001(5)	–0.007(4)
C(5)	8c	0.2599(5)	0.188(1)	0.8519(3)	0.073(6)	0.063(6)	0.035(5)	0.002(6)	–0.002(5)	–0.008(5)
C(6)	8c	0.2050(7)	0.167(1)	0.9013(4)	0.093(8)	0.115(9)	0.033(5)	0.004(8)	0.000(6)	–0.001(6)
C(7)	8c	0.1275(7)	0.151(1)	0.8973(4)	0.079(8)	0.16(1)	0.053(7)	0.006(9)	0.011(6)	0.002(7)
C(7A)	8c	0.0828(6)	0.162(1)	0.8428(4)	0.068(7)	0.082(8)	0.064(7)	0.008(6)	0.009(6)	0.006(6)
C(1')	8c	0.2651(5)	0.183(1)	0.7446(3)	0.063(6)	0.054(6)	0.041(5)	0.003(5)	–0.004(5)	–0.010(5)
O(1)	8c	0.2853(4)	0.3054(7)	0.7360(2)	0.101(5)	0.047(3)	0.051(4)	0.000(4)	0.000(3)	0.000(3)
N(2')	8c	0.2830(4)	0.0775(7)	0.7090(2)	0.074(5)	0.034(4)	0.037(4)	0.002(4)	0.001(4)	–0.001(3)
C(3')	8c	0.3159(6)	0.087(1)	0.6533(4)	0.097(3)	0.067(3)	0.053(2)	0.000(3)	0.013(2)	0.007(2)
C(4')	8c	0.3025(6)	–0.026(1)	0.6180(4)	0.097(3)	0.067(3)	0.053(2)	0.000(3)	0.013(2)	0.007(2)
C(5')	8c	0.3315(6)	–0.020(1)	0.5623(4)	0.097(3)	0.067(3)	0.053(2)	0.000(3)	0.013(2)	0.007(2)
C(6')	8c	0.3741(6)	0.090(1)	0.5439(4)	0.097(3)	0.067(3)	0.053(2)	0.000(3)	0.013(2)	0.007(2)
C(7')	8c	0.3890(6)	0.200(1)	0.5794(4)	0.097(3)	0.067(3)	0.053(2)	0.000(3)	0.013(2)	0.007(2)
C(8')	8c	0.3600(5)	0.198(1)	0.6344(4)	0.097(3)	0.067(3)	0.053(2)	0.000(3)	0.013(2)	0.007(2)
C(1'')	8c	0.3413(5)	0.1105(9)	0.8594(4)	0.061(6)	0.066(6)	0.056(6)	0.005(6)	0.003(5)	–0.001(5)
C(2'')	8c	0.3941(6)	0.170(1)	0.9061(4)	0.088(8)	0.074(7)	0.054(6)	–0.012(6)	–0.015(6)	0.002(5)
C(3'')	8c	0.4763(6)	0.103(1)	0.9068(4)	0.074(8)	0.114(9)	0.056(6)	0.002(7)	–0.005(5)	–0.012(6)
C(4'')	8c	0.5314(6)	0.168(1)	0.9501(5)	0.071(7)	0.102(9)	0.105(9)	0.014(7)	–0.018(7)	–0.020(8)
C(5'')	8c	0.6134(7)	0.106(1)	0.9484(5)	0.088(9)	0.11(1)	0.13(1)	0.008(8)	–0.022(8)	–0.007(9)

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