Crystal structure of (13*S**,13*aR**)-2,3-dimethoxy-13-hydroxy-13*a*-methyl-8-oxo-5,6,13,13*a*-tetrahydro-8*H*-dibenzo[*a,g*]quinolizine, C₂₀H₂₁NO₄

L. Liu^I, R. N. Warren^I, R. A. Russell^{II} and E. R. T. Tiekink^{*,III,I}^I Central Queensland University, Centre for Molecular Architecture, Rockhampton, Queensland, 4702, Australia^{II} Deakin University, Centre for Chiral and Molecular Technologies, Geelong, Victoria, 3217, Australia^{III} The University of Adelaide, Department of Chemistry, Australia 5005

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Abstract

C₂₀H₂₁NO₄, monoclinic, *P*2₁/*c*1 (No. 14), *a* = 7.521(2) Å, *b* = 23.257(2) Å, *c* = 9.784(2) Å, β = 95.57(2)°, *V* = 1703.3 Å³, *Z* = 4, *R*_g(*F*) = 0.060, *wR*_{ref}(*F*²) = 0.183, *T* = 293 K.

Source of material

To a cooled solution (195 K) of diisopropylamine (0.7 ml, 5 mmol) in dry tetrahydrofuran (THF, 90 ml) under dry nitrogen was added *n*-butyllithium (5 mmol) dropwise using a syringe. A solution of phthalide (0.670 g, 5 mmol) in dry THF (20 ml) was then added dropwise, using a syringe, to generate the bright yellow phthalide anion. After stirring for 5 min a solution of 6,7-dimethoxy-3,4-dihydroisoquinoline (1.03 g, 5 mmol) in dry THF (20 ml) was added dropwise, from a syringe, and the resulting mixture maintained at 195 K with stirring for a further 30 min. The solution was allowed to slowly warm to 293 K, diluted with hydrochloric acid (2 M, 120 ml) and extracted with chloroform (4 × 40 ml). The combined extracts were washed with brine, dried over MgSO₄, filtered and evaporated to dryness. The resultant gum was purified by column chromatography on silica gel using a gradient of hexane in ethyl acetate. The product was isolated in 71% yield and recrystallised from ethyl acetate as colourless rods, mp 490 K – 491 K.

Experimental details

The H atoms were placed in their geometrically calculated positions and included in the final refinement in the riding model approximation.

Discussion

The molecules associate in the crystal structure via conventional H-bonding interactions operating between the hydroxyl and carbonyl groups. Thus, *d*(O13–H...O8ⁱ) = 1.75 Å, *d*(O13...O8ⁱ) = 2.814(4) Å and the angle subtended at H is 171°; symmetry operation *i*: –1 + *x*, +*y*, +*z*. The result of such interactions is the formation of chain running along the crystallographic *a*-axis.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.15 × 0.29 × 0.45 mm
Wavelength:	Mo <i>K</i> _α radiation (0.7107 Å)
μ:	0.92 cm ^{−1}
Diffractometer, scan mode:	Rigaku AFC6R, ω/2θ
2θ _{max} :	50.2°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	3353, 3017
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 1710
<i>N</i> (<i>param</i>) _{refined} :	227
Programs:	teXsan [1], SIR92 [2], SHELXL-97 [3]

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(13)	4e	0.2338	0.8808	0.3077	0.073(4)
H(1)	4e	0.3528	0.8129	0.5505	0.058(2)
H(2a)	4e	0.2351	0.7539	0.6829	0.087(4)
H(2b)	4e	0.0580	0.7720	0.5959	0.087
H(2c)	4e	0.0511	0.7466	0.7437	0.087
H(3a)	4e	0.3538	0.9774	0.9719	0.087
H(3b)	4e	0.1871	0.9638	1.0509	0.087
H(3c)	4e	0.1669	1.0035	0.9208	0.087
H(4)	4e	0.4596	0.9802	0.7972	0.058
H(5a)	4e	0.7527	0.9870	0.7117	0.073(3)
H(5b)	4e	0.6846	0.9965	0.5562	0.073
H(6a)	4e	0.9648	0.9502	0.5717	0.073
H(6b)	4e	0.8883	0.8997	0.6556	0.073
H(9)	4e	1.0398	0.8725	0.1237	0.058(2)
H(10)	4e	0.9103	0.8448	−0.0892	0.058
H(11)	4e	0.6045	0.8339	−0.1258	0.058
H(12)	4e	0.4268	0.8495	0.0521	0.058
H(13)	4e	0.4953	0.9255	0.3166	0.073
H(13a)	4e	0.7407	0.7923	0.5350	0.087
H(13b)	4e	0.7274	0.7914	0.3741	0.087
H(13c)	4e	0.557	0.7797	0.4504	0.087

* Correspondence author (e-mail: chmtert@nus.edu.sg)

^I Current address: National University of Singapore, Department of Chemistry, Singapore 117543

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(2)	4e	0.1349(3)	0.8258(1)	0.7422(3)	0.051(2)	0.052(2)	0.067(2)	-0.011(1)	0.030(1)	-0.004(1)
O(3)	4e	0.1889(3)	0.9213(1)	0.8770(3)	0.055(2)	0.060(2)	0.055(2)	-0.004(1)	0.028(1)	-0.005(1)
O(8)	4e	1.0508(3)	0.9176(1)	0.3624(3)	0.033(2)	0.075(2)	0.073(2)	-0.014(1)	0.016(1)	-0.018(2)
O(13)	4e	0.3497(3)	0.8557(1)	0.2911(3)	0.031(1)	0.073(2)	0.054(2)	-0.007(1)	0.008(1)	-0.015(1)
N(7)	4e	0.7931(4)	0.8997(1)	0.4566(3)	0.027(2)	0.054(2)	0.046(2)	-0.004(1)	0.011(1)	-0.004(2)
C(1)	4e	0.3754(5)	0.8465(2)	0.6007(4)	0.041(2)	0.042(2)	0.044(2)	-0.004(2)	0.007(2)	-0.002(2)
C(1a)	4e	0.5162(5)	0.8823(2)	0.5697(4)	0.036(2)	0.042(2)	0.037(2)	0.000(2)	0.007(2)	0.001(2)
C(2)	4e	0.2702(5)	0.8598(2)	0.7036(4)	0.036(2)	0.046(2)	0.045(2)	-0.003(2)	0.008(2)	0.005(2)
C(2a)	4e	0.1185(6)	0.7702(2)	0.6868(6)	0.066(3)	0.064(3)	0.105(4)	-0.019(2)	0.034(3)	-0.009(3)
C(3)	4e	0.2994(5)	0.9113(2)	0.7758(3)	0.040(2)	0.047(2)	0.036(2)	0.003(2)	0.011(2)	0.004(2)
C(3a)	4e	0.2273(7)	0.9705(2)	0.9621(5)	0.091(4)	0.059(3)	0.078(3)	-0.008(3)	0.053(3)	-0.019(2)
C(4)	4e	0.4386(5)	0.9464(2)	0.7475(4)	0.045(2)	0.041(2)	0.044(2)	-0.002(2)	0.009(2)	-0.002(2)
C(4a)	4e	0.5501(5)	0.9321(2)	0.6447(3)	0.038(2)	0.041(2)	0.038(2)	-0.001(2)	0.009(2)	0.001(2)
C(5)	4e	0.7127(5)	0.9676(2)	0.6264(4)	0.046(2)	0.049(2)	0.048(2)	-0.010(2)	0.012(2)	-0.004(2)
C(6)	4e	0.8583(5)	0.9281(2)	0.5845(4)	0.035(2)	0.062(3)	0.049(2)	-0.008(2)	0.005(2)	-0.007(2)
C(8)	4e	0.8971(5)	0.8995(2)	0.3501(4)	0.032(2)	0.045(2)	0.053(2)	-0.001(2)	0.014(2)	-0.003(2)
C(8a)	4e	0.8109(5)	0.8783(1)	0.2160(4)	0.038(2)	0.038(2)	0.044(2)	0.001(2)	0.014(2)	0.003(2)
C(9)	4e	0.9165(5)	0.8681(2)	0.1090(4)	0.040(2)	0.053(2)	0.055(3)	-0.000(2)	0.019(2)	0.003(2)
C(10)	4e	0.8394(5)	0.8515(2)	-0.0180(4)	0.054(2)	0.054(2)	0.044(2)	0.003(2)	0.024(2)	0.001(2)
C(11)	4e	0.6568(5)	0.8448(2)	-0.0397(4)	0.060(3)	0.057(3)	0.037(2)	0.005(2)	0.009(2)	-0.001(2)
C(12)	4e	0.5499(5)	0.8544(2)	0.0671(4)	0.042(2)	0.055(2)	0.045(2)	0.004(2)	0.010(2)	-0.001(2)
C(12a)	4e	0.6266(5)	0.8711(1)	0.1947(4)	0.038(2)	0.036(2)	0.041(2)	0.002(2)	0.011(2)	0.003(2)
C(13)	4e	0.5166(4)	0.8839(2)	0.3139(3)	0.031(2)	0.045(2)	0.042(2)	-0.000(2)	0.010(2)	-0.003(2)
C(13a)	4e	0.6235(4)	0.8659(1)	0.4495(4)	0.030(2)	0.036(2)	0.048(2)	-0.003(2)	0.013(2)	-0.001(2)
C(13b)	4e	0.6662(5)	0.8013(2)	0.4525(4)	0.054(2)	0.050(2)	0.058(2)	0.008(2)	0.018(2)	0.008(2)

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