Crystal structure of 8*b*-hydroxy-2,3-bis(2-pyridyl)spiro[3,3*a*-dihydro-2*H*-cyclopenta[*a*]indene-1,2'-indane]-1',3',4-trione ethanol solvate, C₃₂H₂₆N₂O₅

Pauls Janis Pastors^{*,I}, Sergey Belyakov^{I,II} and Valdis Kampars^I

^I Department of Material Science and Applied Chemistry, Riga Technical University, Azenes Str. 14/24, Riga, LV 1048, Latvia

^{II} Latvian Institute of Organic Synthesis, 21 Aizkraukles Street, Riga, LV 1006, Latvia

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Abstract

C₃₂H₂₆N₂O₅, monoclinic, *P*2₁/*n* (no. 14), *a* = 13.4384(2) Å, *b* = 11.1393(2) Å, *c* = 17.0663(3) Å, β = 94.6270(9)°, *V* = 2546.4 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0634, *wR*_{ref}(*F*²) = 0.1812, *T* = 193 K.

Table 1. Data collection and handling.

Crystal:	pink prisms, size 0.22×0.29×0.37 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	0.92 cm ^{−1}
Diffractometer, scan mode:	Bruker-Nonius Kappa CCD, φ and ω
2θ _{max} :	55.76°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	11599, 6050
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 4262
<i>N</i> (<i>param</i>) _{refined} :	341
Programs:	SHELX [2], ORTEP [3], SCALEPACK [4]

Source of material

5 mmol indan-1,3-dione, 5 mmol pyridine-2-carbaldehyde, 13 ml of methanol in the presence of a catalytic amount of piperidine, were stirred for 30 min. The reaction mixture is left for one month. A mixture of two products was obtained: a pink powdery substance and blue-black crystals. Using the difference of solubility in chloroform blue-black crystals separated by filtration, washed on the filter with hexane, and dried. The compound was recrystallized from an ethanol solution.

Experimental details

The hydrogen atoms were refined using riding model with *U*_{iso}(H) = 1.2*U*_{eq}.

Discussion

The present work is the continuation of the systematic investigations of structure of indan-1,3-dione derivatives. These compounds represent a potential substances for creation of new non-linear optical materials [1]. The molecular structure of the title compound with atomic numbering scheme and thermal ellipsoids is presented in the figure. The five-membered cycle of C1', C2', C7B, C7A, C2 is characterized by an envelope form (the C2 atom deviates from the plane of C1', C2', C7B, C7A by 0.558(3) Å). All other rings in the molecule are almost planar. In the crystal structure there are ethanol molecules with disordered hydroxy groups which take part in intermolecular hydrogen bonds of O–H⋯O and O–H⋯N types. Parameters of these bonds are following: O1'⋯O1E = 2.718(3) Å (H1'⋯O1E = 1.78 Å, O1'–H1'⋯O1E = 164°); O1E⋯N1B = 2.841(3) Å (H1E⋯N1B = 1.96 Å, O1E–H1E⋯N1B = 150°).

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

Atom	Site	Occ.	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1')	4e		0.2837	0.4533	0.5649	0.042
H(4')	4e		−0.1545	0.3071	0.4457	0.043
H(4)	4e		0.0724	−0.0800	0.5741	0.040
H(2')	4e		0.1596	0.4814	0.4378	0.035
H(7)	4e		0.3395	0.0845	0.7453	0.047
H(7')	4e		0.0902	0.2994	0.6699	0.044
H(6')	4e		−0.0854	0.2546	0.6842	0.051
H(5')	4e		−0.1987	0.2581	0.5740	0.049
H(5)	4e		0.1141	−0.1603	0.6983	0.051
H(6)	4e		0.2425	−0.0835	0.7799	0.053
H(7B)	4e		0.1996	0.2719	0.3510	0.034
H(7A)	4e		0.3529	0.3242	0.4738	0.035
H(3B)	4e		0.2218	0.3477	0.2319	0.050
H(3A)	4e		0.4023	0.2205	0.3147	0.055
H(5A)	4e		0.4717	−0.1311	0.3562	0.057
H(6A)	4e		0.3975	−0.1212	0.4790	0.064
H(4B)	4e		0.3119	0.4834	0.1562	0.064
H(5B)	4e		0.4280	0.6238	0.2187	0.070
H(4A)	4e		0.4778	0.0338	0.2760	0.063

* Correspondence author (e-mail: pauls@ktf.rtu.lv)

Table 2. continued.

Atom	Site	Occ.	x	y	z	U _{iso}
H(6B)	4e		0.4464	0.6255	0.3575	0.074
C(1E)	4e		0.4013(3)	0.6468(3)	0.5568(2)	0.0693(9)
H(1EA)	4e	0.6	0.4685	0.6708	0.5493	0.083
H(1EB)	4e	0.6	0.3559	0.6885	0.5200	0.083
H(1EC)	4e	0.4	0.4550	0.5897	0.5585	0.083
H(1ED)	4e	0.4	0.4208	0.7184	0.5307	0.083
C(2E)	4e		0.3783(3)	0.6775(4)	0.6371(2)	0.075(1)

Table 2. continued.

Atom	Site	Occ.	x	y	z	U _{iso}
H(2EA)	4e		0.3984	0.6122	0.6716	0.090
H(2EB)	4e		0.4137	0.7489	0.6542	0.090
H(2EC)	4e		0.3078	0.6907	0.6381	0.090
O(1E)	4e	0.6	0.3945(2)	0.5260(2)	0.5388(2)	0.0357(6)
H(1E)	4e	0.6	0.3789	0.5447	0.4835	0.043
O(2E)	4e	0.4	0.3330(4)	0.6031(5)	0.5146(3)	0.065(1)
H(2E)	4e	0.4	0.3489	0.5782	0.4670	0.078

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(3)	4e	0.1204(1)	0.0989(1)	0.45177(8)	0.0316(8)	0.0323(8)	0.0227(7)	−0.0009(6)	−0.0009(6)	−0.0041(6)
O(1')	4e	0.2171(1)	0.4368(1)	0.57839(8)	0.0425(9)	0.0348(8)	0.0278(7)	−0.0095(7)	0.0030(6)	−0.0108(6)
C(9')	4e	−0.0018(2)	0.3305(2)	0.4853(1)	0.034(1)	0.026(1)	0.028(1)	0.0040(8)	0.0050(8)	−0.0028(8)
O(1)	4e	0.3771(1)	0.2619(2)	0.62305(9)	0.0340(8)	0.050(1)	0.0369(9)	−0.0074(7)	−0.0052(7)	−0.0060(7)
C(9)	4e	0.1804(2)	0.0563(2)	0.5864(1)	0.031(1)	0.029(1)	0.0238(9)	0.0039(8)	0.0041(8)	0.0006(8)
C(3')	4e	0.0485(2)	0.3590(2)	0.4134(1)	0.037(1)	0.031(1)	0.027(1)	0.0056(9)	0.0033(9)	0.0002(9)
C(3)	4e	0.1730(1)	0.1235(2)	0.5108(1)	0.026(1)	0.028(1)	0.0214(9)	0.0019(8)	0.0037(7)	−0.0018(8)
C(8')	4e	0.0687(2)	0.3267(2)	0.5494(1)	0.035(1)	0.024(1)	0.026(1)	−0.0001(8)	0.0085(8)	−0.0035(8)
O(3')	4e	0.0121(1)	0.3562(2)	0.34596(9)	0.0433(9)	0.066(1)	0.0276(8)	0.0048(9)	−0.0008(7)	0.0009(8)
C(2)	4e	0.2407(2)	0.2341(2)	0.5213(1)	0.027(1)	0.031(1)	0.0208(9)	−0.0030(8)	0.0024(8)	−0.0008(8)
C(1')	4e	0.1733(2)	0.3504(2)	0.5261(1)	0.033(1)	0.028(1)	0.0216(9)	−0.0054(8)	0.0042(8)	−0.0039(8)
C(4')	4e	−0.1019(2)	0.3055(2)	0.4928(1)	0.034(1)	0.034(1)	0.041(1)	0.005(1)	0.0045(9)	−0.005(1)
C(1)	4e	0.3032(2)	0.2074(2)	0.5988(1)	0.029(1)	0.038(1)	0.0233(9)	0.0007(9)	0.0006(8)	−0.0049(9)
C(4)	4e	0.1249(2)	−0.0412(2)	0.6077(1)	0.036(1)	0.031(1)	0.035(1)	0.0004(9)	0.0053(9)	0.0021(9)
C(2')	4e	0.1572(2)	0.3907(2)	0.4389(1)	0.039(1)	0.026(1)	0.0235(9)	−0.0019(9)	0.0062(8)	−0.0006(8)
C(7)	4e	0.2823(2)	0.0529(2)	0.7102(1)	0.040(1)	0.051(2)	0.026(1)	0.007(1)	0.0004(9)	0.002(1)
C(8)	4e	0.2573(2)	0.1040(2)	0.6368(1)	0.031(1)	0.038(1)	0.0228(9)	0.0031(9)	0.0033(8)	0.0004(9)
C(7')	4e	0.0398(2)	0.3002(2)	0.6239(1)	0.042(1)	0.042(1)	0.028(1)	−0.000(1)	0.0089(9)	−0.000(1)
C(6')	4e	−0.0600(2)	0.2752(2)	0.6314(1)	0.047(1)	0.047(2)	0.037(1)	−0.001(1)	0.018(1)	0.002(1)
C(5')	4e	−0.1303(2)	0.2768(2)	0.5667(2)	0.034(1)	0.042(1)	0.048(1)	−0.001(1)	0.014(1)	−0.003(1)
C(5)	4e	0.1503(2)	−0.0920(2)	0.6813(1)	0.048(1)	0.039(1)	0.041(1)	0.003(1)	0.013(1)	0.013(1)
C(6)	4e	0.2273(2)	−0.0454(2)	0.7312(1)	0.054(2)	0.052(2)	0.028(1)	0.011(1)	0.008(1)	0.014(1)
C(7B)	4e	0.2341(2)	0.3276(2)	0.3902(1)	0.033(1)	0.032(1)	0.0219(9)	−0.0031(9)	0.0065(8)	−0.0015(8)
N(1A)	4e	0.3550(2)	0.0534(2)	0.4679(1)	0.046(1)	0.047(1)	0.040(1)	0.013(1)	0.0068(9)	0.0091(9)
C(7A)	4e	0.3038(2)	0.2605(2)	0.4523(1)	0.029(1)	0.034(1)	0.024(1)	−0.0053(9)	0.0058(8)	−0.0003(8)
C(2A)	4e	0.3548(2)	0.1504(2)	0.4222(1)	0.026(1)	0.038(1)	0.030(1)	−0.0034(9)	0.0031(8)	−0.0033(9)
N(1B)	4e	0.3526(2)	0.4904(2)	0.3746(1)	0.064(1)	0.050(1)	0.043(1)	−0.023(1)	0.016(1)	0.003(1)
C(2B)	4e	0.2880(2)	0.4126(2)	0.3385(1)	0.037(1)	0.035(1)	0.031(1)	−0.0003(9)	0.0101(9)	0.0048(9)
C(3B)	4e	0.2716(2)	0.4077(2)	0.2575(1)	0.047(1)	0.049(2)	0.030(1)	0.006(1)	0.009(1)	0.009(1)
C(3A)	4e	0.3998(2)	0.1497(2)	0.3520(2)	0.055(2)	0.043(1)	0.043(1)	−0.000(1)	0.021(1)	−0.002(1)
C(5A)	4e	0.4460(2)	−0.0535(3)	0.3749(2)	0.038(1)	0.046(2)	0.059(2)	0.009(1)	0.002(1)	−0.010(1)
C(6A)	4e	0.4003(2)	−0.0456(3)	0.4436(2)	0.052(2)	0.051(2)	0.057(2)	0.018(1)	0.006(1)	0.010(1)
C(4B)	4e	0.3224(2)	0.4852(3)	0.2116(2)	0.056(2)	0.066(2)	0.039(1)	0.011(2)	0.017(1)	0.024(1)
C(5B)	4e	0.3882(2)	0.5657(3)	0.2481(2)	0.053(2)	0.062(2)	0.065(2)	0.006(1)	0.027(1)	0.034(2)
C(4A)	4e	0.4453(2)	0.0461(3)	0.3281(2)	0.054(2)	0.055(2)	0.051(2)	0.004(1)	0.020(1)	−0.011(1)
C(6B)	4e	0.4019(2)	0.5653(3)	0.3292(2)	0.063(2)	0.059(2)	0.065(2)	−0.019(2)	0.018(2)	0.012(2)

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