Crystal structure of (10b*SR*,11*RS*)-10b-hydroxy-11-phenyl-10b,11-dihydro-isoindolo[2,1*a*]indol-6-one, $C_{21}H_{15}NO_2$

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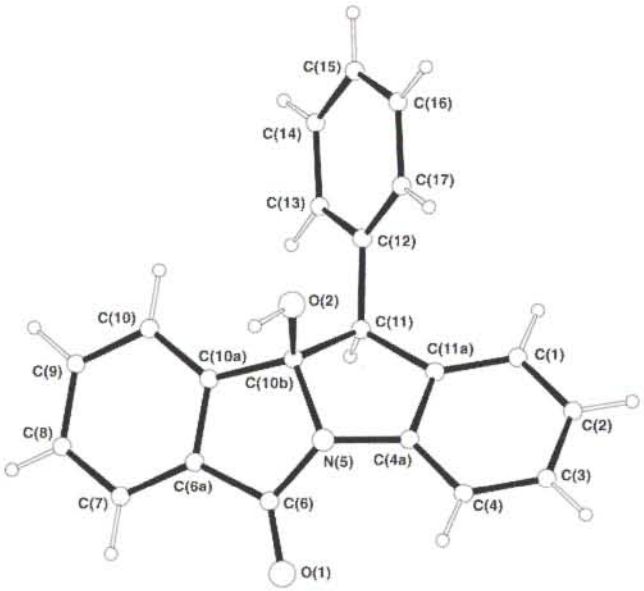

Abstract

$C_{21}H_{15}NO_2$, monoclinic, $P12_1/n1$ (No. 14), a = 11.496(1) Å, b = 7.208(1) Å, c = 19.394(1) Å, β = 97.16(1)°, V = 1594.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.037, $wR_{ref}(F^2)$ = 0.095, T = 293 K.

Source of material

The title compound was prepared by photocyclization of *N*-phthaloyl 2-amino diphenylmethane in acetone in 96% yield. This reaction is initiated by a homolytic 1,7-hydrogen abstraction [1, 2] generating a 1,5-triplet biradical which, after intersystem crossing, cyclizes to give the title compound with remarkably high *cis*-diastereoselectivity [3]. The compound was crystallized from acetone, mp = 467 K – 469 K.

Discussion

In the solid state two intermolecular hydrogen bonds between the amide carbonyl and the hydroxy goups are connecting two molecules with identical bond angles of O(1)–H(O2)–O(2) of 176° and bond lengths of O1···H of 1.84 Å.

Table 1. Data collection and handling.

Crystal:	pale yellow prism, size 0.20 x 0.25 x 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.84 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, φ/ω-scans
$2\theta_{max}$:	54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6563, 3473
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2947
$N(param)_{refined}$:	277
Programs:	SHELXS-97 [4], SHELXL-97 [5], Xtal [6]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.269(1)	0.407(2)	0.2310(7)	0.048(4)
H(2)	4*e*	0.074(1)	0.322(2)	0.2477(8)	0.056(4)
H(3)	4*e*	0.038(1)	0.168(2)	0.3510(8)	0.062(4)
H(4)	4*e*	0.196(1)	0.104(2)	0.4406(8)	0.053(4)
H(7)	4*e*	0.629(1)	0.287(2)	0.6134(8)	0.058(4)
H(8)	4*e*	0.824(2)	0.367(2)	0.6035(9)	0.075(5)
H(9)	4*e*	0.897(2)	0.376(3)	0.4940(9)	0.079(5)
H(10)	4*e*	0.770(1)	0.309(2)	0.3924(8)	0.054(4)
H(11)	4*e*	0.483(1)	0.475(2)	0.3553(6)	0.031(3)
H(13)	4*e*	0.647(1)	0.577(2)	0.3067(8)	0.057(4)
H(14)	4*e*	0.759(2)	0.580(3)	0.2104(8)	0.069(5)
H(15)	4*e*	0.711(2)	0.371(3)	0.1169(9)	0.077(5)
H(16)	4*e*	0.557(1)	0.152(2)	0.1204(9)	0.064(5)
H(17)	4*e*	0.454(1)	0.148(2)	0.2158(7)	0.048(4)
H(O2)	4*e*	0.573(1)	–0.045(2)	0.3870(8)	0.065(5)

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*	0.2546(1)	0.3439(2)	0.27406(6)	0.0362(6)	0.0359(6)	0.0428(6)	0.0032(5)	0.0013(5)	0.0029(5)
C(2)	4*e*	0.1406(1)	0.2929(2)	0.28419(7)	0.0333(6)	0.0413(7)	0.0552(7)	0.0033(5)	-0.0026(5)	–0.0041(6)
C(3)	4*e*	0.1184(1)	0.2057(2)	0.34481(7)	0.0295(6)	0.0448(7)	0.0642(8)	–0.0025(5)	0.0086(6)	–0.0061(6)
C(4)	4*e*	0.2088(1)	0.1633(2)	0.39692(7)	0.0372(6)	0.0423(7)	0.0496(7)	–0.0015(5)	0.0153(5)	0.0022(5)
C(4A)	4*e*	0.3212(1)	0.2132(2)	0.38576(6)	0.0318(5)	0.0345(6)	0.0376(6)	0.0027(4)	0.0066(4)	0.0004(4)
N(5)	4*e*	0.42786(8)	0.1836(1)	0.42942(5)	0.0332(5)	0.0426(5)	0.0316(5)	0.0030(4)	0.0088(4)	0.0028(4)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(6)	4*e*	0.4570(1)	0.2174(2)	0.49880(6)	0.0469(6)	0.0372(6)	0.0306(5)	0.0139(5)	0.0095(5)	0.0052(4)
C(6A)	4*e*	0.5846(1)	0.2597(2)	0.50801(6)	0.0467(7)	0.0370(6)	0.0322(6)	0.0114(5)	0.0016(5)	–0.0025(5)
C(7)	4*e*	0.6584(1)	0.2943(2)	0.56863(7)	0.0626(9)	0.0529(8)	0.0339(6)	0.0184(7)	–0.0045(6)	–0.0085(6)
C(8)	4*e*	0.7735(1)	0.3383(2)	0.56275(8)	0.0626(9)	0.067(1)	0.0494(8)	0.0070(8)	–0.0203(7)	–0.0159(7)
C(9)	4*e*	0.8142(1)	0.3439(2)	0.49846(8)	0.0445(8)	0.074(1)	0.0618(9)	–0.0057(7)	–0.0105(7)	–0.0072(8)
C(10)	4*e*	0.7409(1)	0.3052(2)	0.43763(7)	0.0379(6)	0.0627(9)	0.0439(7)	–0.0038(6)	–0.0004(5)	–0.0033(6)
C(10A)	4*e*	0.6248(1)	0.2651(2)	0.44338(6)	0.0367(6)	0.0406(6)	0.0323(5)	0.0038(5)	–0.0004(4)	–0.0025(5)
C(10B)	4*e*	0.52682(9)	0.2102(2)	0.38794(5)	0.0305(5)	0.0381(6)	0.0284(5)	0.0004(4)	0.0066(4)	–0.0014(4)
C(11)	4*e*	0.47550(9)	0.3535(2)	0.33258(5)	0.0298(5)	0.0327(6)	0.0331(5)	–0.0011(4)	0.0037(4)	–0.0003(4)
C(11A)	4*e*	0.34537(9)	0.3033(2)	0.32552(6)	0.0302(5)	0.0311(5)	0.0365(5)	0.0010(4)	0.0059(4)	0.0000(4)
C(12)	4*e*	0.53819(9)	0.3599(2)	0.26893(5)	0.0286(5)	0.0383(6)	0.0330(5)	0.0004(4)	0.0025(4)	0.0073(4)
C(13)	4*e*	0.6289(1)	0.4873(2)	0.26657(7)	0.0356(6)	0.0518(8)	0.0454(7)	–0.0085(5)	0.0029(5)	0.0075(6)
C(14)	4*e*	0.6932(1)	0.4901(2)	0.21089(8)	0.0373(7)	0.077(1)	0.0565(8)	–0.0123(7)	0.0098(6)	0.0161(8)
C(15)	4*e*	0.6670(1)	0.3687(3)	0.15643(7)	0.0396(7)	0.094(1)	0.0451(7)	0.0034(7)	0.0156(6)	0.0155(8)
C(16)	4*e*	0.5767(1)	0.2429(2)	0.15773(7)	0.0480(8)	0.075(1)	0.0356(6)	0.0037(7)	0.0069(6)	–0.0018(6)
C(17)	4*e*	0.5126(1)	0.2380(2)	0.21392(6)	0.0368(6)	0.0497(7)	0.0354(6)	–0.0040(5)	0.0041(5)	0.0025(5)
O(1)	4*e*	0.38917(8)	0.2146(1)	0.54284(4)	0.0574(6)	0.0627(6)	0.0368(4)	0.0229(5)	0.0199(4)	0.0119(4)
O(2)	4*e*	0.54977(7)	0.0463(1)	0.35291(4)	0.0405(4)	0.0380(4)	0.0297(4)	0.0058(3)	0.0051(3)	–0.0007(3)

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