Crystal structure of 3-[(3-oxo-1,3-dihydro-isobenzofuran-1-yl)-phenethyl-amino]-2-phenethyl-2,3-dihydro-isoindol-1-one, C₃₂H₂₈N₂O₃

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Abstract

C₃₂H₂₈N₂O₃, triclinic, $P\bar{1}$ (No. 2), $a = 8.648(8)$ Å, $b = 10.27(1)$ Å, $c = 15.31(2)$ Å, $\alpha = 80.69(3)^\circ$, $\beta = 73.71(2)^\circ$, $\gamma = 84.13(2)^\circ$, $V = 1286.1$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.119$, $wR_{\text{ref}}(F^2) = 0.379$, $T = 293$ K.

Source of material

A solution of phthalaldehydic acid (25 g, 166 mmol) in toluene (100 mL) was treated with phenethylamine (20.1 g, 166 mmol) and refluxed with stirring under a Dean-Stark trap for 2 hours. After concentrating to dryness, the title product was isolated by Si gel chromatography (4:1 CH₂Cl₂-MeOH, v/v) and crystallized as colourless needles in cyclohexane (yield 32%).

Experimental details

The reason for the high R -values is the low quality of the crystal due to its slow decomposition, as reflected on the high R_{int} value (0.087).

Discussion

The crystal is triclinic, space group $P\bar{1}$. The molecular structure and bond lengths and angles of the compound are given in the figure and fall within the normal ranges. No hydrogen bond interactions were found. The benzene rings lie parallel to each heterocyclic ring, forming an N1–C9–C–10 angle of 112.73° between the isoindole nitrogen and the ethylbenzene moiety. The isobenzofuranone ring leads to a linkage of N2 to the isoindolone

ring and benzene ring forming angles of 114.97° for N2–C32–C8 and 118.03° for N2–C32–C17. The bonds of N2 to carbons C32 and C8 measure 1.426 Å and 1.486 Å, respectively, in agreement with the different electronegativity of the heteroatoms linked to these saturated carbon atoms.

Table 1. Data collection and handling.

Crystal:	colourless prism, size $0.17 \times 0.17 \times 0.37$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	0.81 cm^{-1}
Diffractometer, scan mode:	Siemens SMART CCD, ϕ/ω
$2\theta_{\text{max}}$:	56.3°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	7125, 5066
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1773
$N(\text{param})_{\text{refined}}$:	335
Programs:	SHELXS-97 [1], SHELXL-97 [2], ORTEP-3 [3], WinGX [4]

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

Atom	Site	x	y	z	U_{iso}
H(3)	2i	0.5610	0.4255	0.1119	0.071
H(4)	2i	0.6462	0.3003	0.2345	0.090
H(5)	2i	0.5674	0.3637	0.3773	0.076
H(6)	2i	0.4017	0.5548	0.4061	0.071
H(8)	2i	0.3230	0.7994	0.2828	0.048
H(9A)	2i	0.0707	0.8812	0.1897	0.057
H(9B)	2i	0.1334	0.8366	0.0920	0.057
H(10A)	2i	0.3285	0.9812	0.1553	0.075
H(10B)	2i	0.3503	0.9610	0.0534	0.075
H(12)	2i	−0.0297	1.0384	0.0912	0.063
H(13)	2i	−0.1701	1.2410	0.0761	0.101
H(14)	2i	−0.0417	1.4356	0.0706	0.117
H(15)	2i	0.2128	1.4207	0.0766	0.113
H(16)	2i	0.3589	1.2222	0.0867	0.088
H(17A)	2i	0.0556	0.5421	0.4125	0.073
H(17B)	2i	0.0620	0.5519	0.3084	0.073
H(18A)	2i	−0.1972	0.6512	0.4442	0.080
H(18B)	2i	−0.2036	0.5318	0.3935	0.080
H(20)	2i	−0.1493	0.5894	0.2159	0.095
H(21)	2i	−0.2288	0.7348	0.1013	0.112
H(22)	2i	−0.3523	0.9372	0.1301	0.119
H(23)	2i	−0.3955	1.0031	0.2729	0.117
H(24)	2i	−0.3170	0.8536	0.3909	0.088
H(27)	2i	0.3638	1.1213	0.4334	0.074
H(28)	2i	0.2408	1.2810	0.3449	0.081
H(29)	2i	0.0324	1.2288	0.2924	0.079
H(30)	2i	−0.0436	1.0150	0.3156	0.074
H(32)	2i	−0.0255	0.7854	0.4565	0.064

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(1)	2i	0.3413(8)	0.6487(7)	0.1287(4)	0.060(4)	0.063(4)	0.049(4)	0.019(4)	−0.022(3)	−0.024(3)
C(2)	2i	0.4194(7)	0.5574(5)	0.1904(4)	0.051(4)	0.029(3)	0.052(4)	−0.006(3)	−0.007(3)	0.004(3)
C(3)	2i	0.5260(8)	0.4489(6)	0.1708(5)	0.067(5)	0.040(4)	0.063(4)	−0.003(3)	−0.008(4)	−0.004(3)
C(4)	2i	0.5785(9)	0.3759(8)	0.2442(6)	0.068(5)	0.067(5)	0.086(6)	0.025(4)	−0.022(4)	−0.014(4)
C(5)	2i	0.5315(9)	0.4145(7)	0.3299(5)	0.063(5)	0.052(4)	0.079(5)	−0.006(4)	−0.032(4)	0.009(4)
C(6)	2i	0.4310(8)	0.5284(6)	0.3481(4)	0.066(5)	0.064(4)	0.045(4)	0.013(4)	−0.017(3)	−0.004(3)
C(7)	2i	0.3764(7)	0.6005(6)	0.2761(4)	0.048(4)	0.044(4)	0.036(3)	0.006(3)	−0.008(3)	0.001(3)
C(8)	2i	0.2671(7)	0.7254(5)	0.2753(4)	0.044(4)	0.037(3)	0.038(3)	−0.007(3)	−0.010(3)	−0.003(3)
C(9)	2i	0.1670(8)	0.8579(6)	0.1431(4)	0.054(4)	0.052(4)	0.033(3)	0.011(3)	−0.013(3)	−0.002(3)
C(10)	2i	0.2707(8)	0.9760(7)	0.1102(5)	0.049(4)	0.066(5)	0.061(4)	0.008(4)	−0.010(3)	0.007(3)
C(11)	2i	0.1811(9)	1.1066(6)	0.0940(4)	0.075(5)	0.046(4)	0.040(4)	0.007(4)	−0.019(3)	0.007(3)
C(12)	2i	0.0205(8)	1.1153(6)	0.0886(4)	0.052(4)	0.044(4)	0.053(4)	−0.001(3)	−0.009(3)	0.005(3)
C(13)	2i	−0.064(1)	1.2363(9)	0.0796(5)	0.093(6)	0.088(7)	0.059(5)	0.041(6)	−0.020(4)	−0.002(4)
C(14)	2i	0.014(2)	1.353(1)	0.0757(6)	0.15(1)	0.069(7)	0.060(5)	0.040(7)	−0.023(6)	0.003(4)
C(15)	2i	0.163(2)	1.3437(8)	0.0792(6)	0.16(1)	0.048(5)	0.073(6)	−0.009(6)	−0.037(6)	0.005(4)
C(16)	2i	0.251(1)	1.2235(7)	0.0867(5)	0.104(6)	0.063(5)	0.053(4)	−0.009(5)	−0.028(4)	0.009(4)
C(17)	2i	0.0250(8)	0.5978(7)	0.3618(5)	0.062(5)	0.073(5)	0.048(4)	−0.001(4)	−0.015(3)	−0.008(3)
C(18)	2i	−0.1588(8)	0.6170(8)	0.3859(5)	0.063(5)	0.085(5)	0.050(4)	−0.024(4)	−0.010(3)	−0.003(4)
C(19)	2i	−0.2212(8)	0.7114(8)	0.3131(5)	0.046(4)	0.102(6)	0.045(4)	−0.015(4)	−0.011(3)	−0.006(4)
C(20)	2i	−0.1973(9)	0.6723(9)	0.2276(5)	0.063(5)	0.122(7)	0.053(5)	−0.003(5)	−0.017(4)	−0.013(5)
C(21)	2i	−0.246(1)	0.759(1)	0.1594(6)	0.063(6)	0.152(9)	0.064(6)	−0.030(6)	−0.011(4)	−0.007(6)
C(22)	2i	−0.320(1)	0.881(1)	0.1765(7)	0.045(5)	0.17(1)	0.076(7)	−0.025(6)	−0.025(5)	0.026(7)
C(23)	2i	−0.3467(9)	0.920(1)	0.2615(8)	0.046(5)	0.119(8)	0.122(9)	0.009(5)	−0.024(5)	−0.010(7)
C(24)	2i	−0.2973(9)	0.8306(9)	0.3322(5)	0.045(4)	0.105(7)	0.067(5)	−0.014(4)	−0.003(4)	−0.020(5)
C(25)	2i	0.2824(9)	0.8528(7)	0.4802(4)	0.062(5)	0.074(5)	0.042(4)	0.006(4)	−0.018(3)	−0.019(3)
C(26)	2i	0.2311(8)	0.9727(7)	0.4246(4)	0.050(4)	0.071(5)	0.035(3)	0.018(4)	−0.011(3)	−0.015(3)
C(27)	2i	0.2829(9)	1.1007(8)	0.4096(4)	0.061(5)	0.078(5)	0.050(4)	0.006(4)	−0.016(3)	−0.029(4)
C(28)	2i	0.208(1)	1.1949(7)	0.3580(5)	0.088(6)	0.061(5)	0.048(4)	0.008(4)	−0.010(4)	−0.017(4)
C(29)	2i	0.0843(9)	1.1626(8)	0.3250(4)	0.079(5)	0.068(5)	0.046(4)	0.031(4)	−0.019(4)	−0.017(4)
C(30)	2i	0.0371(9)	1.0363(7)	0.3393(4)	0.076(5)	0.071(5)	0.043(4)	0.026(4)	−0.024(3)	−0.024(3)
C(31)	2i	0.1135(8)	0.9409(6)	0.3902(4)	0.051(4)	0.057(4)	0.031(3)	0.012(3)	−0.004(3)	−0.010(3)
C(32)	2i	0.0843(8)	0.7949(7)	0.4164(4)	0.044(4)	0.079(5)	0.037(3)	0.017(3)	−0.013(3)	−0.018(3)
N(1)	2i	0.2511(7)	0.7415(5)	0.1811(3)	0.075(4)	0.052(3)	0.029(3)	0.027(3)	−0.018(2)	−0.008(2)
N(2)	2i	0.1057(6)	0.7199(5)	0.3428(3)	0.038(3)	0.048(3)	0.040(3)	−0.007(2)	−0.004(2)	−0.003(2)
O(1)	2i	0.3599(6)	0.6523(5)	0.0459(3)	0.089(4)	0.081(3)	0.041(3)	0.028(3)	−0.021(2)	−0.024(2)
O(2)	2i	0.2029(6)	0.7492(5)	0.4734(3)	0.079(3)	0.074(3)	0.035(2)	0.020(3)	−0.023(2)	−0.007(2)
O(3)	2i	0.3787(6)	0.8402(6)	0.5247(3)	0.084(4)	0.115(5)	0.066(3)	0.013(3)	−0.048(3)	−0.006(3)

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