Crystal structure of 2-[(3-oxo-1,3-dihydro-2-benzofuran-5-yl)methyl]-1*H*-isoindole-1,3(2*H*)-dione, $C_{17}H_{11}NO_4$

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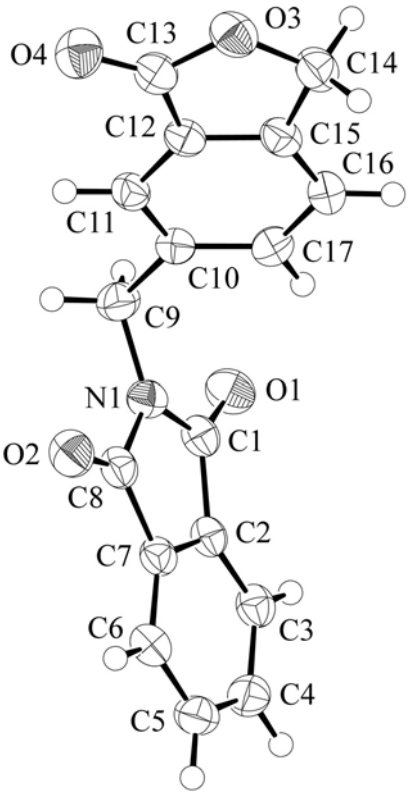

Abstract

$C_{17}H_{11}NO_4$, monoclinic, $P12_1/n1$ (no. 14), a = 11.324(2) Å, b = 8.587(2) Å, c = 13.942(3) Å, β = 102.08(3)°, V = 1325.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.056, $wR_{ref}(F^2)$ = 0.136, T = 173 K.

Source of material

The reagents are purchased as indicated and used without further purification: phthalide (98 %, Alfa Aesar), *N*-(hydroxymethyl)-phthalimide (97 %, Alfa Aesar), sulfuric acid (98 %, Mallinckrodt), acetic acid (99.7 %, Mallinckrodt), ethanol (Mallinckrodt). Phthalide (27.79 g, 0.21 mol) and concentrated H_2SO_4 (100 mL) were heated to 50 °C with stirring. Solid *N*-(hydroxymethyl)phthalimide (41.83 g, 0.24 mol) was added portionwise over 30 min. The solution was stirred for 1 h at 50 °C, and then heated to 105 °C overnight. After removal of heat, the reaction mixture was cooled to room temperature, and slowly poured into ethanol/water (1:3, 1200 mL) with vigorous stirring. The product was collected by vacuum filtration, repeatedly washed with water to neutrality and air-dried to give a light-peach solid (24.17 g) containing (49 % molar ratio by NMR) the title product, its isomer 2-[(1-oxo-1,3-dihydro-2-benzofuran-4-yl)-methyl]-1*H*-isoindole-1,3(2*H*)-dione (30 %), and residual phthalide (21 %). The isomer and phthalide were removed by extraction with acetic acid/ethanol (1:6). The peach-colored solid obtained was then recrystallized by acetic acid/isopropyl alcohol (1:5) give clear leaf-shaped light-peach colored crystals. ^{1}H NMR and IR data are available in the CIF.

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Discussion

The title compound was synthesized as an intermediate to the analogous 6-aminomethylphthalide. Each of the two fused rings is essentially planar. A twist in the molecule is indicated by the N1/C9/C10/C11 torsion angle of –131.5(2)°. The dihedral angle between the two fused ring systems is 73.35(6)°. The crystal structure comprises layers of molecules stacked along [100]. Layers are stabilized by $\pi\cdots\pi$ interactions between centrosymmetrically related N1 rings that are separated by 3.632(1) Å, symmetry operation: 1–*x*,–*y*,1–*z*. Additional stabilization is afforded by C–H$\cdots\pi$ contacts with the shortest of these occurring between centrosymmetric pairs: C14–H14a$\cdots$Cg(O3-ring) is 2.97 Å, C14$\cdots$Cg(O3-ring) is 3.860(3) Å with an angle of 149° at the H14a atom, symmetry operation: 1–*x*,1–*y*,2–*z*. Connections between layers are of the type C–H$\cdots$O and involve all three O atoms with the closest contact being C17–H17$\cdots$O4 of 2.40 Å, C17$\cdots$O4 of 3.268(3) Å with an angle at H17 of 151°, symmetry operation: –1/2+*x*,1/2–*y*,–1/2+*z*.

Table 1. Data collection and handling.

Crystal:	light-peach block, size 0.10 × 0.13 × 0.18 mm
Wavelength:	Mo K_α radiation (0.71070 Å)
μ:	1.06 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC12-κ & SATURN724, ω
$2\theta_{max}$:	53°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11451, 2690
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2379
$N(param)_{refined}$:	199
Programs:	teXsan [1], SIR92 [2], SHELXL-97 [3], ORTEP-II [4]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(3)	4*e*	0.2822	0.2463	0.3512	0.044
H(4)	4*e*	0.3776	0.4167	0.2593	0.049
H(5)	4*e*	0.5759	0.4943	0.3162	0.050
H(6)	4*e*	0.6840	0.4146	0.4717	0.045
H(9A)	4*e*	0.5841	–0.0192	0.7232	0.049
H(9B)	4*e*	0.4428	–0.0564	0.6931	0.049

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(11)	4*e*	0.6333	0.0425	0.8894	0.040
H(14A)	4*e*	0.4348	0.4512	1.0577	0.053
H(14B)	4*e*	0.3624	0.2988	1.0805	0.053
H(16)	4*e*	0.2866	0.3577	0.8626	0.051
H(17)	4*e*	0.3258	0.2243	0.7265	0.049

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}
O(1)	4*e*	0.3025(1)	0.0518(2)	0.5354(1)	0.0424(8)	0.0480(9)	0.0476(9)	–0.0106(7)	0.0124(6)	–0.0029(7)
O(2)	4*e*	0.6847(1)	0.2313(2)	0.6572(1)	0.0354(8)	0.061(1)	0.0386(8)	0.0020(7)	0.0014(6)	–0.0017(7)
O(3)	4*e*	0.5448(1)	0.2752(2)	1.1196(1)	0.0504(9)	0.0532(9)	0.0365(8)	–0.0005(7)	0.0113(7)	–0.0082(6)
O(4)	4*e*	0.6989(1)	0.1209(2)	1.1027(1)	0.0429(9)	0.056(1)	0.0454(9)	0.0006(7)	0.0002(7)	–0.0021(7)
N(1)	4*e*	0.4938(2)	0.1238(2)	0.6185(1)	0.0377(9)	0.0398(9)	0.0314(9)	0.0008(7)	0.0092(7)	0.0013(7)
C(1)	4*e*	0.3942(2)	0.1267(2)	0.5396(1)	0.035(1)	0.037(1)	0.034(1)	0.0006(8)	0.0101(8)	–0.0060(8)
C(2)	4*e*	0.4281(2)	0.2336(2)	0.4663(1)	0.031(1)	0.034(1)	0.033(1)	0.0018(8)	0.0084(8)	–0.0048(7)
C(3)	4*e*	0.3627(2)	0.2807(2)	0.3754(1)	0.035(1)	0.042(1)	0.034(1)	0.0027(9)	0.0069(8)	–0.0057(8)
C(4)	4*e*	0.4202(2)	0.3805(2)	0.3213(1)	0.047(1)	0.044(1)	0.030(1)	0.0052(9)	0.0063(8)	–0.0012(8)
C(5)	4*e*	0.5384(2)	0.4283(2)	0.3558(2)	0.051(1)	0.039(1)	0.038(1)	–0.000(1)	0.0174(9)	0.0008(9)
C(6)	4*e*	0.6033(2)	0.3812(2)	0.4475(1)	0.035(1)	0.042(1)	0.038(1)	–0.0019(8)	0.0115(8)	–0.0045(8)
C(7)	4*e*	0.5456(2)	0.2843(2)	0.5017(1)	0.033(1)	0.036(1)	0.030(1)	0.0024(8)	0.0089(8)	–0.0040(8)
C(8)	4*e*	0.5885(2)	0.2157(2)	0.6006(1)	0.033(1)	0.039(1)	0.034(1)	0.0043(8)	0.0090(8)	–0.0036(8)
C(9)	4*e*	0.5029(2)	0.0288(2)	0.7069(1)	0.052(1)	0.038(1)	0.033(1)	0.0046(9)	0.0116(9)	0.0031(8)
C(10)	4*e*	0.4822(2)	0.1201(2)	0.7948(1)	0.037(1)	0.034(1)	0.033(1)	0.0009(8)	0.0115(8)	0.0037(8)
C(11)	4*e*	0.5628(2)	0.1046(2)	0.8836(1)	0.032(1)	0.033(1)	0.039(1)	–0.0011(8)	0.0126(8)	0.0018(8)
C(12)	4*e*	0.5375(2)	0.1825(2)	0.9640(1)	0.033(1)	0.035(1)	0.035(1)	–0.0045(8)	0.0101(8)	0.0002(8)
C(13)	4*e*	0.6057(2)	0.1848(2)	1.0659(2)	0.037(1)	0.040(1)	0.040(1)	–0.0047(9)	0.0092(9)	–0.0021(8)
C(14)	4*e*	0.4346(2)	0.3359(3)	1.0576(2)	0.044(1)	0.049(1)	0.043(1)	0.003(1)	0.0149(9)	–0.0030(9)
C(15)	4*e*	0.4360(2)	0.2740(2)	0.9577(2)	0.037(1)	0.038(1)	0.039(1)	–0.0031(8)	0.0138(9)	–0.0004(8)
C(16)	4*e*	0.3558(2)	0.2929(3)	0.8684(2)	0.039(1)	0.046(1)	0.044(1)	0.0091(9)	0.0132(9)	0.0039(9)
C(17)	4*e*	0.3802(2)	0.2141(2)	0.7881(2)	0.042(1)	0.045(1)	0.034(1)	0.0058(9)	0.0068(9)	0.0064(9)

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