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Crystal structure of 2,4-dibenzoyl-*N,N*-dimethylbenzenamine, C₂₂H₁₉NO₂

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Abstract

C₂₂H₁₉NO₂, monoclinic, *P*₂/c (no. 14), *a* = 12.7728(19) Å, *b* = 7.9765(12) Å, *c* = 20.451(3) Å, β = 123.946(7)°, *V* = 1728.5(5) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.049, *wR*_{ref}(*F*²) = 0.150, *T* = 296(2) K.

CCDC no.: 1433714

The crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was prepared by a literature method [1]. In a 250 mL three-neck flask equipped with a mechanical

Table 1: Data collection and handling.

Crystal:	Colourless blocks
Wavelength:	Size 0.25 × 0.20 × 0.16 mm
μ:	Mo Kα radiation (0.71073 Å)
Diffractometer, scan mode:	0.8 cm ⁻¹
2θ _{max} , completeness:	Nonius CAD4, ω and 2θ
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	52°, >99%
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	10046, 3383, 0.173
<i>N</i> (<i>param</i>) _{refined} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2697
Programs:	229
	CAD-4 [3, 4], SHELX [5]

Table 2: Fractional atomic coordinates and isotropic or equivalent displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.33289(11)	0.8899(2)	0.07217(7)	0.0781(4)
O2	0.24768(10)	0.76442(13)	0.36947(7)	0.0579(3)
N1	0.52798(11)	0.83648(16)	0.43202(7)	0.0519(4)
C14	0.27577(11)	0.67266(16)	0.33420(7)	0.0382(3)
C11	0.46157(11)	0.84040(17)	0.35182(8)	0.0414(3)
C12	0.34993(11)	0.74243(16)	0.30369(7)	0.0381(3)
C9	0.43824(12)	0.94241(19)	0.23249(9)	0.0474(4)
H9	0.4671	1.0113	0.2091	0.057*
C15	0.23266(10)	0.49418(16)	0.31823(7)	0.0363(3)
C8	0.33100(12)	0.84220(17)	0.18439(8)	0.0423(3)
C7	0.27137(13)	0.8450(2)	0.09814(9)	0.0501(4)
C13	0.29031(11)	0.74308(16)	0.22229(8)	0.0397(3)
H13	0.2203	0.6745	0.1918	0.048*
C10	0.50114(11)	0.94120(19)	0.31270(9)	0.0477(4)
H10	0.5721	1.0087	0.3424	0.057*
C20	0.13796(12)	0.44626(19)	0.32860(9)	0.0480(4)
H20	0.0999	0.5257	0.3421	0.058*
C1	0.13673(13)	0.7931(2)	0.04205(8)	0.0489(4)
C17	0.25130(13)	0.2083(2)	0.28954(9)	0.0545(4)
H17	0.2895	0.1280	0.2765	0.065*
C16	0.28844(12)	0.37398(18)	0.29823(8)	0.0449(4)
H16	0.3511	0.4049	0.2906	0.054*
C18	0.15772(14)	0.16143(19)	0.30015(9)	0.0565(4)
H18	0.1334	0.0497	0.2947	0.068*
C19	0.10063(14)	0.2803(2)	0.31871(10)	0.0583(4)
H19	0.0363	0.2489	0.3247	0.070*
C2	0.04414(13)	0.8227(2)	0.05660(9)	0.0525(4)
H2	0.0661	0.8700	0.1042	0.063*
C3	−0.08077(15)	0.7820(2)	0.00031(11)	0.0681(5)

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H3	−0.1428	0.8035	0.0097	0.082*
C6	0.10247(16)	0.7236(2)	−0.02963(10)	0.0623(5)
H6	0.1633	0.7065	−0.0405	0.075*
C4	−0.11231(17)	0.7095(3)	−0.06947(11)	0.0790(6)
H4	−0.1957	0.6800	−0.1068	0.095*
C22	0.62596(14)	0.9609(2)	0.47885(10)	0.0657(5)
H22A	0.7030	0.9269	0.4849	0.099*
H22B	0.6394	0.9703	0.5298	0.099*
H22C	0.5999	1.0675	0.4527	0.099*
C5	−0.02127(19)	0.6801(3)	−0.08460(10)	0.0746(6)
H5	−0.0435	0.6309	−0.1319	0.090*
C21	0.52899(14)	0.6914(2)	0.47520(9)	0.0587(4)
H21A	0.4728	0.7104	0.4917	0.088*
H21B	0.6130	0.6735	0.5207	0.088*
H21C	0.5018	0.5943	0.4418	0.088*

stirrer, dry dichloromethane (80 mL) and anhydrous aluminum chloride powder (22.0 g, 165 mmol) were added successively. Then *N,N*-dimethylaniline (10.0 g, 825 mmol) was added under ice-cooling condition. At the same condition, benzoyl chloride (25.51 g, 181.5 mmol) in dry methylene chloride (60 mL), was slowly added. After the dropping, it was heated under reflux for 4 h. After completion of the reaction, the reaction solution was poured into a mixture of ice-water and hydrochloric acid (7:1, v/v) and stirred for another 2 h. The organic layer was washed with water and caustic sodium bicarbonate separately for two times. The organic phases were combined and dried over anhydrous sodium sulfate. Removal of organic solvent under reduced pressure left a pale yellow solid (16.31 g, 60% crude yield). Crystallization from acetone gave prism-shaped crystals of pure product by evaporation within 10 days at room temperature.

Experimental details

Carbon-bound H atoms were placed in calculated positions (C–H 0.93 Å) and were included in the refinement in the riding model approximation, with *U*_{iso}(H) set to 1.2*U*_{eq}(C). The H atoms of the methyl group were allowed to rotate with a fixed angle around the C–C bond to best fit the

experimental electron density (HFIX 137 in the SHELX program suite [5]), with *U*_{iso}(H) set to 1.5*U*_{eq}(O).

Discussion

The title compound is an important intermediate in the production of naphthopyran compounds. As one of the most important photochromic compounds, naphthopyrans are widely used in many areas, such as decoration, anti-forgery technology and national defense. For the advantages of the property of good fatigue resistance and proper fading speed, the application of naphthopyrans in optical lens has gotten a heck of attention and research. However, even the product, which has been commercialized, still has some defects in such properties.

There is one molecule in the asymmetric unit. The bond lengths and angles are within normal ranges [2]. The dihedral angles between the benzen rings, A(C1–C2–C3–C4–C5–C6), B(C8–C9–C10–C11–C12–C13), C(C15–C16–C17–C18–C19–C20), are between A and B = 49.2(2)°, between B and C = 107.0(3)°, between A and C = 121.8(2)°. There is at least one C–H···O intramolecular hydrogen bond, which may support the stabilization of the crystal structure.

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