Crystal Structure of 2,5-Dibenzoyl-4-iodoaniline

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The title compound, 2,5-dibenzoyl-4-iodoaniline, crystallizes in the monoclinic space group $P2_1/n$ with the following unit-cell parameters: $a = 9.896(2)$, $b = 8.4430(17)$, $c = 20.980(4)$ Å, $\beta = 98.01(3)^\circ$ and $V = 1735.8(6)$ Å³. The crystal structure of this compound was solved by direct methods and refined to a final $R = 0.064$ for 3115 reflections with $I > 2\sigma(I)$. In the crystal packing, the molecules are linked *via* N-H...O and C-H...O hydrogen bonds.

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The title compound contains amino and halogen groups that can react with different groups to prepare various functional organic compounds. It is a kind of aromatic organic intermediate that can be used for many fields, such as aromatic conductive polymers and organometallic chemistry.¹⁻³ Herein we report on its synthesis and crystal structure.

The title compound (Fig. 1) was prepared according to a general iodination procedure *via* diazotization.⁴ 2,5-Dibenzoyl-1,4-diaminobenzene (1.0 g, 3.21 mmol) dissolved in ether (10 mL) was added to boron trifluoride etherate (1.6 mL, 12.85 mmol), followed by the addition of isoamyl nitrite (1.52 mL, 11.24 mmol). After the precipitate was isolated, it was added to a solution of sodium iodide (0.52 g, 3.53 mmol) and iodide (0.07 g, 0.32 mmol) in acetonitrile (20 mL). After 60 min, the mixture was poured into sodium thiosulfate aqution, extracted with dichloroethane, and dried over anhydrous magnesium sulfate. Finally, column chromatography (1:8, ethyl acetate/petroleum ether) gave the title compound (0.9 g, 66% yield). Its single crystal suitable for X-ray structural analysis was obtained by slowly evaporating from ethanol at room temperature for 10 days.

A colorless crystal having dimensions of $0.10 \times 0.10 \times 0.05$ mm³ was chosen for X-ray diffraction studies. The experimental crystallographic details are given in Table 1. Measurements were made on a Enraf-Nonius CAD4 diffractometer equipped with graphite crystal monochromatized Mo K_α radiation ($\lambda = 0.71073$ Å) at 293 K. The structure was solved by direct methods with the SHELXS-97 and refined by full-matrix least-squares procedures on F^2 , using the program SHELXL-97.⁵ All

non-hydrogen atoms were refined anisotropically and all hydrogen atoms were refined using appropriate riding models, with N-H = 0.86 Å, C-H = 0.93 Å for aromatic CH and with $U_{iso}(H) = 1.5 U_{eq}(N)$ or $1.2 U_{eq}(C)$.

The selected bond lengths and bond angles are listed in Table 2. The fractional atomic coordinates, along with their equivalent isotropic displacement parameters, are presented in Table S1 (Supporting Information). As can be seen from Fig. 2, the structure consists of a substituted phenyl ring A(C8-C13) and two benzoyl rings: B(C1-C6) and C(C15-C20). The dihedral angles between the phenyl ring plane (A) and the benzoyl ring

Table 1 Crystal and experimental data

Chemical formula: C ₂₀ H ₁₄ INO ₂	
Formula weight = 427.22	
$T = 293$ K	
Crystal system: monoclinic	
Space group: $P2_1/n$	$Z = 4$
$a = 9.896(2)$ Å	$\alpha = 90^\circ$
$b = 8.4430(17)$ Å	$\beta = 98.01(3)^\circ$
$c = 20.980(4)$ Å	$\gamma = 90^\circ$
$V = 1735.8(6)$ Å ³	
$D_x = 1.635$ mg/m ³	
Radiation: Mo K_α ($\lambda = 0.71073$ Å)	
$\mu(\text{Mo } K_\alpha) = 1.86$ mm ⁻¹	
$F(000) = 840$	
Crystal size = $0.10 \times 0.10 \times 0.05$ mm	
No. of reflections collected = 3306	
No. of independent reflections = 3115	
θ range for data collection: 2.0 to 25.2°	
Data/Restrains/Parameters = 3115/0/217	
Goodness-of-fit on $F^2 = 0.998$	
R indices [$I > 2\sigma(I)$]: $R1 = 0.064$, $wR2 = 0.192$	
R indices (all data): $R1 = 0.109$, $wR2 = 0.160$	
$(\Delta\rho)_{\max} < 0.001$	
$(\Delta\rho)_{\max} = 1.17$ e Å ⁻³	
$(\Delta\rho)_{\min} = -1.60$ e Å ⁻³	
Measurement: Enraf-Nonius CAD4	
Program system: CAD4 Express	
Structure determination: SHELXS-97, SHELXL-97	
Refinement: full matrix	
CCDC deposition number: 701161	

Fig. 1 Chemical diagram of 2,5-dibenzoyl-4-iodo-aniline.

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Table 2 Selected bond distances (Å) and angles (°)

I-C12	2.082 (9)
N-C9	1.350 (11)
O1-C7	1.250 (11)
O2-C14	1.223 (11)
C13-C12-I	120.8 (7)
C11-C12-I	119.8 (7)
O2-C14-C15	120.7 (9)
O2-C14-C11	117.8 (9)
O1-C7-C3	118.7 (9)
O1-C7-C8	121.0 (9)
N-C9-C8	124.0 (8)
N-C9-C10	118.6 (9)

Fig. 2 ORTEP structure of 2,5-dibenzoyl-4-iodo aniline, showing 50% probability ellipsoids.

Table 3 Hydrogen bond geometry (Å, °)

D-H...A	D-H	H...A	D...A	D-H...A
N-H0A...O1	0.86	2.06	2.685(12)	129
N-H0B...O2 ⁱ	0.86	2.18	3.021(12)	164
C2-H2A...O2 ⁱⁱ	0.93	2.50	3.387(14)	159
C17-H17A...O1 ⁱⁱⁱ	0.93	2.54	3.462(14)	169

Symmetry code: (i) $-x+2, -y+2, -z$; (ii) $-x+2, -y+1, -z$; (iii) $x-1, y+1, z$.

plane are 61.0° (B) and 75.8° (C), respectively. The intramolecular N-H...O hydrogen bonds result in the formations of a non-planar six-membered rings: D(N/C9/C8/C7/O1/H0A).

As can be seen from the packing diagram (Fig. 3), the molecules are stacked along the b axis. The intermolecular hydrogen bond between N-H0B...O2, C2-H2A...O2 and C17-H17A...O1 were observed (Table 3). There are not only intermolecular hydrogen bonds, but also I...I interaction (4.082 Å) (van der Waals radius of I is 2.15 Å), which are effective in

Fig. 3 Molecular packing diagram of 2,5-dibenzoyl-4-iodo-aniline.

stabilization of the structure.

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Supporting Information

Table S1 Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²); Fig. S1 Molecular packing diagram of 2,5-dibenzoyl-4-iodoaniline.

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