Crystal Structure of β -Phenylcinnamaldehyde-4-bromoaniline

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The title compound, *N*- β -phenylcinnamaldehyd-4-bromoaniline, C₂₁H₁₆BrN, possesses normal geometrical parameters. The observed bond lengths and angles imply that there is little electronic conjugation between three benzene ring systems. In the crystal, molecules interact by way of weak C-H...N and C-H... π interactions, leading to chains propagating in [010].

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It has been shown that the reaction of 1:1 stiochiometric ratios of aldehydes or ketones and amines yields monodentate Schiff base ligands, such as *N*-(2-benzylidenepropylidene)-4-bromoaniline¹ or *N*-(4-bromobenzylidene)-2-hydroxyaniline.²

Fig. 1 Chemical structure.

In our ongoing studies on the synthesis, structure and spectra of copper(I) complexes with bidentate Schiff-base ligands, such as *Phca*₂-*dab*³ and *Phca*₂-*en*,⁴ arising from β -phenylcinnamaldehyde and suitable amines, we report here on the crystal structure of the title compound *N*- β -phenylcinnamaldehyd-4-bromoaniline, **1** (Fig. 1).

To a stirring solution of β -phenylcinnamaldehyde (208 mg, 0.2 mmol) in methanol (5 ml), cooled in an ice bath, a solution of 4-bromoaniline (344 mg, 0.2 mmol) in methanol (5 ml) was added drop-wise. The mixture was then stirred for an additional 30 min. *N*- β -Phenylcinnamaldehyd-4-bromoaniline, **1**, was obtained as a yellow microcrystalline precipitate. Yellow crystals of **1** were obtained by slow evaporation of acetone-chloroform (1:1 v/v) solution at 273 K. Intensity data for a yellow single crystal (dimensions of 0.40 × 0.40 × 0.25 mm) were collected at 120 K on a Nonius Kappa CCD diffractometer (Mo *K*_α radiation). The structure was solved by direct methods using the program SHELXS-97. The refinement and all further calculations were carried out using SHELXL-97.

Table 1 Crystallographic and experimental data

Formula	C ₂₁ H ₁₆ BrN
Formula weight	362.26
Crystal system	triclinic
Space group	<i>P</i> $\bar{1}$
<i>a</i>	5.8424(2) Å
<i>b</i>	11.4712(5) Å
<i>c</i>	12.2178(5) Å
α	89.574(2)°
β	87.245(3)°
γ	89.481(3)°
<i>V</i>	817.83(6) Å ³
<i>D</i> _x	1.471 g cm ⁻³
No. of reflections measured	3764
2 θ _{max}	55.4°
<i>R</i> (<i>F</i>), <i>wF</i> (<i>F</i> ²)	0.048, 0.113
($\Delta\rho$) _{max} , ($\Delta\rho$) _{min}	+0.38, -0.60 e Å ⁻³
Crystallographic data (excluding structure factors) for the structure <i>Phca</i> - <i>Bran</i> has been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 634999. Copies of the data can be obtained free of charge on application to CCDS, 12 Union Road, Cambridge CB2 1EZ, UK.	

Table 2 Final atomic coordinates and equivalent isotropic thermal parameters

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} (Å ²)
C1	0.7262 (6)	0.1982 (3)	0.6656 (3)	0.0302 (8)
C2	0.5734 (6)	0.1212 (3)	0.7161 (3)	0.0327 (9)
C3	0.3911 (6)	0.0837 (3)	0.6588 (3)	0.0277 (8)
C4	0.3611 (6)	0.1201 (3)	0.5530 (3)	0.0279 (8)
C5	0.5176 (5)	0.1953 (3)	0.5026 (3)	0.0241 (8)
C6	0.7027 (5)	0.2353 (3)	0.5590 (3)	0.0210 (7)
C7	0.9409 (5)	0.3101 (3)	0.4171 (3)	0.0216 (7)
C8	1.1093 (5)	0.3927 (3)	0.3769 (3)	0.0222 (7)
C9	1.2143 (5)	0.3938 (3)	0.2769 (3)	0.0199 (7)
C10	1.3974 (5)	0.4790 (3)	0.2492 (3)	0.0194 (7)
C11	1.5503 (5)	0.5125 (3)	0.3266 (3)	0.0212 (7)
C12	1.7213 (5)	0.5914 (3)	0.3012 (3)	0.0236 (8)
C13	1.7397 (6)	0.6405 (3)	0.1974 (3)	0.0269 (8)
C14	1.5886 (6)	0.6089 (3)	0.1196 (3)	0.0260 (8)
C15	1.4191 (5)	0.5286 (3)	0.1451 (3)	0.0241 (8)
C16	1.1570 (5)	0.3107 (3)	0.1895 (3)	0.0211 (7)
C17	0.9566 (5)	0.3221 (3)	0.1340 (3)	0.0268 (8)
C18	0.9117 (6)	0.2465 (3)	0.0502 (3)	0.0324 (9)
C19	1.0639 (6)	0.1591 (3)	0.0203 (3)	0.0309 (9)
C20	1.2612 (7)	0.1459 (3)	0.0770 (3)	0.0320 (9)
C21	1.3097 (6)	0.2213 (3)	0.1595 (3)	0.0273 (8)
N1	0.8636 (4)	0.3164 (2)	0.5164 (2)	0.0236 (6)
Br1	0.17663 (6)	-0.01722 (3)	0.73107 (3)	0.03657 (17)

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Table 3 Selected bond distances (Å) and angles (°)

Bond distances (Å)			
C3–Br1	1.896 (3)	C7–C8	1.439 (4)
C6–N1	1.407 (4)	C8–C9	1.341 (4)
C7–N1	1.277 (4)		
Bond angles (°)			
C4–C3–Br1	120.5 (3)	C9–C8–C7	126.2 (3)
C2–C3–Br1	118.1 (3)	C8–C9–C10	120.4 (3)
C1–C6–N1	117.0 (3)	C8–C9–C16	122.7 (3)
C5–C6–N1	124.3 (3)	C10–C9–C16	116.8 (3)
N1–C7–C8	119.3 (3)		
Torsion angles (°)			
C1–C2–C3–Br1	–177.6 (3)	C7–C8–C9–C10	–175.4 (3)
Br1–C3–C4–C5	178.9 (3)	C7–C8–C9–C16	4.0 (5)
C2–C1–C6–N1	178.2 (3)	C8–C7–N1–C6	–178.6 (3)
C4–C5–C6–N1	–176.6 (3)	C1–C6–N1–C7	141.6 (3)
N1–C7–C8–C9	176.5 (3)	C5–C6–N1–C7	–41.6 (5)

Fig. 2 Molecular structure of **1**, showing 50% displacement ellipsoids (arbitrary spheres for the H atoms).

The H-atoms were placed in idealized locations and refined as riding atoms. Data collection and refinement parameters are listed in Table 1. The atomic coordinates for non-hydrogen atoms are listed in Table 2 and selected inter-atomic parameters are collected in Table 3.

The molecular structure is shown in Fig. 2. Its geometrical parameters are normal. The dihedral angles between the three aromatic rings containing atoms C(1), C(10) and C(16) are as follows: C(1)/C(10) = 5.02(16)°, C(1)/C(16) = 89.07(17)°, C(10)/C(16) = 89.59(16)°. The N(1), C(7), C(8) and C(9) grouping is essentially flat (r.m.s. deviation from the mean plane = 0.017 Å) and the C(7)–C(8) bond length of 1.439(4) Å suggests a small amount of double-bond character. The

Fig. 3 Details of **1**, showing the C–H...N and C–H... π interactions. Symmetry codes as in Table 4.

Table 4 Hydrogen bond geometry (Å, °)

D–H...A	D...H	H...A	D...A	D–H...A
C12–H12...N1 ⁱ	0.95	2.60	3.545 (4)	173
C17–H17...C _g ⁱⁱ	0.95	2.72	3.668 (4)	175

The four columns refer to the C–H separation, H...A separation, C...A separation and C–H...A angle. C_g = centroid of C10–C16 benzene ring. Symmetry codes: (i) 3–x, 1–y, 1–z; (ii) x–1, y, z.

C(6)–N(1)–C(7) bond angle of 120.7(3)° is consistent with the *sp*² hybrid character for the N atom.⁵ In the crystal, molecules of **1** form inversion generated pairs *via* weak C–H...N interactions. Then, a near linear C–H... π interaction helps to link the dimeric pairs into chains propagating in [010] (Fig. 3 and Table 4).

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