

4-[4-(*n*-Decyloxy)benzylideneamino]benzonitrileRajnikant,^{a*} Dinesh,^a Rajinder K. Bamzai,^b Tej K. Razdan^b and D. Singh^a^aCondensed Matter Physics Group, Department of Physics, University of Jammu, Jammu Tawi 180 006, India, and ^bDepartment of Chemistry, University of Jammu, Jammu Tawi 180 006, IndiaCorrespondence e-mail:
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Key indicators

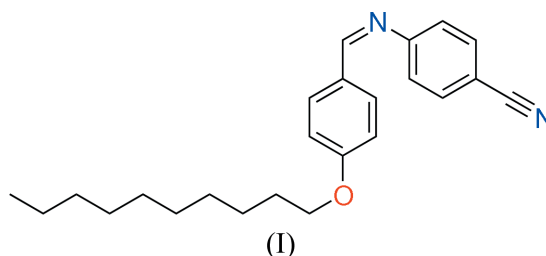
Single-crystal X-ray study
T = 295 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.042
wR factor = 0.132
Data-to-parameter ratio = 15.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $\text{C}_{24}\text{H}_{30}\text{N}_2\text{O}$, the configuration of the azomethine ($\text{C}=\text{N}$) bond is *E*. A $\text{C}-\text{H} \cdots \pi$ interaction also contributes to the stability of the structure. The molecules form infinite layers with an antiparallel arrangement of the CN groups.

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Comment

The title compound, (I), belongs to the group of aromatic compounds with azomethine bridges. This family of compounds is well utilized for their thermotropic liquid crystal behaviour (Davey *et al.*, 2005; Rajnikant *et al.*, 2000). Since these compounds readily order themselves to some degree in the liquid crystal phase, we were interested in probing structural mechanisms during the crystallization event. The present study is part of our ongoing work on the preparation of X-ray diffraction-quality single crystals of cyanobiphenyls and their structural studies (Rajnikant *et al.*, 1999, 2000, 2002).



The bond distances $\text{N}2-\text{C}7$ and $\text{C}1-\text{N}2$ (Table 1) are in good agreement with the analogous structure reported by Baumeister *et al.* (1987), while the bond distances $\text{C}11-\text{O}1$ and $\text{C}14-\text{O}1$ are quite close to those in the analogous structure reported by Davey *et al.* (2005). The bond angles $\text{N}2-\text{C}1-\text{C}2$ and $\text{N}2-\text{C}1-\text{C}6$ show that atom $\text{N}2$ is inclined more towards atom $\text{C}2$ than atom $\text{C}6$ of the phenyl ring $\text{C}1-\text{C}6$. Similarly, atom $\text{O}1$ deviates more towards atom $\text{C}10$ than $\text{C}11$.

The configuration about $\text{C}=\text{N}$ is *E*. The torsion angles $\text{C}2-\text{C}1-\text{N}2-\text{C}7$, $\text{C}6-\text{C}1-\text{N}2-\text{C}7$, $\text{C}1-\text{N}2-\text{C}7-\text{C}8$, $\text{N}2-\text{C}7-\text{C}8-\text{C}9$ and $\text{N}2-\text{C}7-\text{C}8-\text{C}13$ (Table 1) are in good agreement with values in the analogous structure reported by Baumeister *et al.* (1987). There is a dihedral angle of $30.8 (2)^\circ$ between the two benzene rings. Atom $\text{C}24$ of the cyano group deviates by -0.087 \AA , and atom $\text{O}1$ deviates by 0.175 \AA from the mean plane of the benzylideneaniline moiety.

The molecules of (I) form infinite layers with an antiparallel arrangement of the CN groups, parallel to the *bc* plane. A similar arrangement of CN groups was observed for two analogous structures (Mandal & Paul, 1985; Kuribayashi & Hori, 1998).

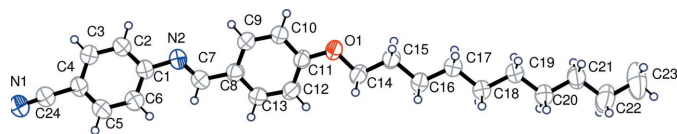


Figure 1
The molecular structure of (I), with displacement ellipsoids shown at the 50% probability level.

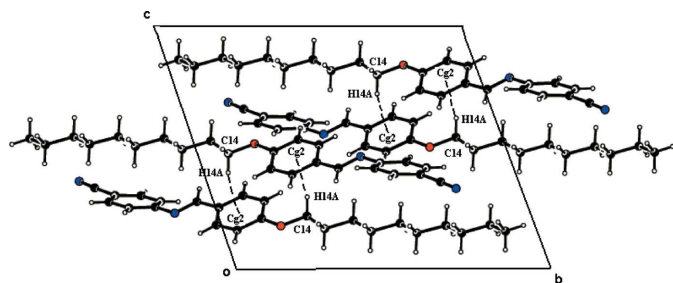


Figure 2
A packing diagram of (I), showing the formation of bilayers parallel to the *bc* plane. Dashed lines indicate C—H... π interactions.

There are C—H... π interactions between the ring (C8—C13)¹ and C14—H14A with a closest H...centroid separation of 2.84 Å [symmetry code: (i) $-x, 1 - y, 1 - z$] and an angle at H of 141°. The crystal packing is characterized by a perfectly antiparallel alignment of CN groups, forming an infinite layer extending along the *bc* face (Fig. 2), as observed in 4'-pentyloxy-4-cyanobiphenyl (Mandal & Paul, 1985) and 4-cyano-4'-octylbiphenyl (Kuribayashi & Hori, 1998).

Experimental

The title compound was synthesized by heating 4-decyloxybenzaldehyde (0.246 g, 1 mmol) with 4-cyanoaniline (0.118 g, 1 mmol) in dry methanol (20 ml) and pyridinium hydrochloride (0.0107 g, 0.1 mmol). 4-Decyloxybenzaldehyde was obtained by stirring phenol (2.82 g, 20 mmol) and *n*-decyl chloride (3.53 g, 20 mmol) in an aqueous solution of methylene chloride (40% NaOH; 10 ml CH₂Cl₂) containing benzyltributylammonium chloride (0.582 g, 10 mol) at room temperature for 6 h followed by a Reimer–Tiemann reaction to obtain *ortho*- and *para*-decyloxybenzaldehyde. The *para* product was separated by column chromatography. 4-Cyanoaniline was obtained by reducing the nitro group of *p*-nitrobenzoic acid to the corresponding amino derivative, followed by converting the carboxyl group into a cyano group. Crystals of (I) suitable for X-ray investigation were obtained by recrystallization from ethanol (yield: 0.172 g, 47%; m.p. 372 K).

Crystal data

C₂₄H₃₀N₂O
M_r = 362.50
 Triclinic, *P* $\bar{1}$
a = 8.564 (4) Å
b = 9.571 (2) Å
c = 14.114 (3) Å
 α = 101.56 (2)°
 β = 91.68 (3)°
 γ = 103.69 (2)°

V = 1097.6 (6) Å³
Z = 2
D_x = 1.097 Mg m⁻³
 Mo *K* α radiation
 μ = 0.07 mm⁻¹
T = 295 (2) K
 Plate, yellow
 0.4 × 0.3 × 0.1 mm

Data collection

Enraf–Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: none
 3886 measured reflections
 3859 independent reflections

1960 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.011$
 $\theta_{\text{max}} = 25.0^\circ$
 2 standard reflections
 every 100 reflections
 intensity decay: <2%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.132$
 $S = 1.01$
 3859 reflections
 244 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 0.0582P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.11 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.15 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

O1—C11	1.357 (2)	N2—C7	1.274 (2)
O1—C14	1.431 (2)	N2—C1	1.412 (2)
N1—C24	1.149 (2)		
C7—N2—C1	118.90 (17)	O1—C11—C10	115.50 (17)°
C2—C1—N2	118.67 (17)	O1—C11—C12	125.45 (17)°
C6—C1—N2	123.18 (19)		
C7—N2—C1—C2	-148.27 (19)	N2—C7—C8—C9	-4.4 (3)
C7—N2—C1—C6	35.7 (3)	N2—C7—C8—C13	4.1 (2)
C1—N2—C7—C8	2.9 (2)		

H atoms were positioned geometrically and treated as riding, with C—H distances in the range 0.93–0.97 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CAD-4 Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *PARST95* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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