3-[(Diphenylmethylene)amino]phthalonitrile

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Key indicators

Single-crystal X-ray study

$T = 183$ K

Mean σ(C–C) = 0.003 Å

R factor = 0.058

wR factor = 0.133

Data-to-parameter ratio = 14.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The crystal structure of the title compound, $C_{21}H_{13}N_3$, is stabilized by C—H···N, C—H···π and π···π interactions.

Comment

Nitroaromatic compounds have been found to undergo photo-induced reactions with 1,2-diphenylacetylene to give, among others, benzenamine derivatives (Tian & Xu, 2002). The crystal structures of the benzenamine products having 4-chloro and 4-acetyl substituents have been reported elsewhere (Usman *et al.*, 2002). As an extension of such photoreaction of nitroaromatic compounds, we have prepared the title compound, (I), which was obtained from the photo-induced reaction of 2,3-dicyanonitrobenzene with 1,2-diphenylacetylene. An X-ray crystal structure determination of the title compound, (I), was undertaken to elucidate its molecular conformation.

(I)
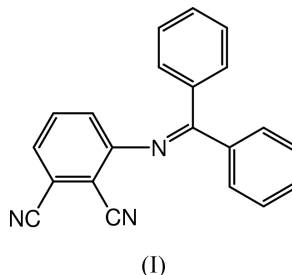
The bond lengths and angles in (I) have normal values, except for the bond lengths C1—C20 and C2—C21 [1.441 (3) Å], which are slightly elongated compared with a typical Csp^2—Csp distance (Allen *et al.*, 1987). Such elongations have been observed in other benzenecarbonitrile derivatives [C_{ar}—C_{CN} 1.444 (7) (Toupet *et al.*, 1989) and 1.445 (4) Å (Zhang *et al.*, 2002)].

The nitroaromatic moiety is nearly planar, with atom N1 lying 0.107 (1) Å from the aromatic plane. The C1–C6 phenyl ring makes dihedral angles of 64.1 (1) and 70.8 (1)°, respectively, with the C8–C13 and C14–C19 phenyl rings, while the dihedral angle between these two phenyl rings is 68.8 (1) Å. As a result of their sp^2 character, atoms C6, N1, C7, C8 and C14 linking the three phenyl rings are almost coplanar, with C6—N1—C7—C8 and C6—N1—C7—C14 torsion angles of 9.2 (3) and −167.9 (2)°, respectively.

In the solid state, molecules of (I) are linked by inter-molecular C—H···N-type hydrogen bonds into infinite zigzag molecular chains along the *b* axis (Fig. 2). Intermolecular C—H···π interactions involving C3 and the C8–C13 aromatic ring are observed in this structure [Table 2; Cg(*A*) denotes the
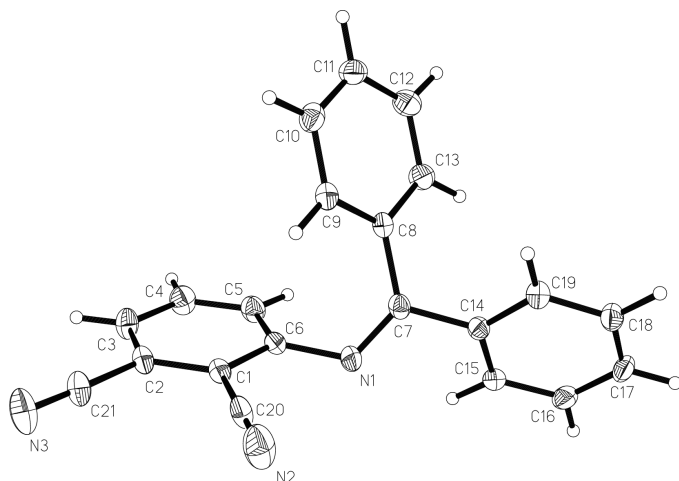
Figure 1
The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

centroid of the ring C8–C13]. The molecular packing is further stabilized by π–π-stacking interactions (perpendicular distance 3.540 Å) involving phenyl rings C1–C6 and C1–C6 of the symmetry-related molecule at $(1/2-x, 3/2-y, -z)$.

Experimental

The title compound was prepared by the photo-induced reaction of an acetonitrile solution of 2,3-dicyanonitrobenzene with an excess of 1,2-diphenylacetylene. The title compound, one of the products in the reaction, was isolated by column chromatography on silica gel. Single crystals suitable for X-ray diffraction study were grown by slow evaporation from petroleum ether–ethyl acetate (5:1) solution.

Crystal data

$C_{21}H_{13}N_3$	$D_x = 1.240$ Mg m^{-3}
$M_r = 307.34$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 4600
$a = 15.3572 (2)$ Å	reflections
$b = 8.0212 (2)$ Å	$\theta = 2.7$–$28.3°$
$c = 26.7363 (2)$ Å	$\mu = 0.08$ mm^{-1}
$\beta = 91.648 (1)°$	$T = 183 (2)$ K
$V = 3292.1 (1)$ Å^3	Slab, yellow
$Z = 8$	$0.44 \times 0.28 \times 0.12$ mm

Data collection

Siemens SMART CCD area-detector diffractometer	3941 independent reflections
ω scans	2251 reflections with $I > 2\sigma(I)$
Absorption correction: multiscan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.072$
	$\theta_{max} = 28.3°$
$T_{min} = 0.968$, $T_{max} = 0.991$	$h = -20 \rightarrow 20$
9550 measured reflections	$k = -10 \rightarrow 10$
	$l = -25 \rightarrow 35$

Refinement

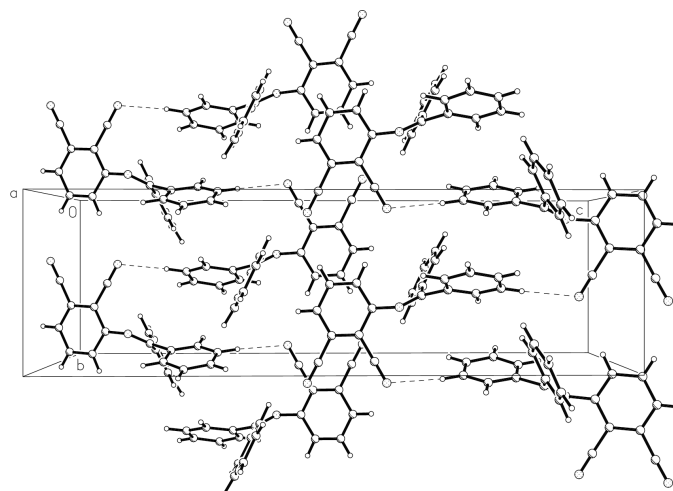|	
Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0427P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.058$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.133$	$(\Delta/\sigma)_{max} < 0.001$
$S = 0.86$	$\Delta\rho_{max} = 0.32$ e Å^{-3}
3941 reflections	$\Delta\rho_{min} = -0.28$ e Å^{-3}
270 parameters	Extinction correction: *SHELXTL*
All H-atom parameters refined	Extinction coefficient: 0.0092 (7)

Figure 2
Packing of the title molecules viewed down the *a* axis, showing the molecular chain formation along the *b* axis.

Table 1
Selected interatomic distances (Å).

N1—C7	1.286 (2)	C7—C14	1.493 (2)
N1—C6	1.410 (2)	C7—C8	1.498 (2)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C18—H18$\cdots$N2^i	1.02 (2)	2.59 (2)	3.526 (3)	152 (1)
C3—H3$\cdots$Cg(A^{ii})	0.97 (2)	3.03 (2)	3.901 (2)	150 (1)

Symmetry codes: (i) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $-x, 2 - y, -z$.

H atoms were located from a difference map and both positional and isotropic displacement parameters were refined. The C—H range is 0.94 (2)-1.02 (2) Å.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

Tian, J. Z. & Xu, J.-H. (2002). Unpublished work.

Toupet, L., Miniewicz, A. & Ecolivet, C. (1989). *Acta Cryst.* C**45**, 1044–1047.

Usman, A., Razak, I. A., Fun, H.-K., Chantrapromma, S., Tian, J. Z., Zhang, Y. & Xu, J.-H. (2002). *Acta Cryst.* C**58**. Submitted.

Zhang, Y., Usman, A., I. A. Razak, Fun, H.-K., Chantrapromma, S. & Xu, J.-H. (2002). *Acta Cryst.* E**58**, o132–o133.