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ORIGINAL PAPER

Solid-State Intermolecular Contacts Involving the Nitrile Group in *p*-Cyano-*N*-(*p*-cyanobenzylidene)aniline and 4,4′-(azinodimethylidyne)*bis*-benzonitrile

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Abstract As part of an investigation of the solid-state intermolecular contacts in which the nitrile group participates, the crystal structures of *p*-cyano-*N*-(*p*-cyanobenzylideneaniline), $C_{15}H_9N_3$ (CN/CN) and 4,4′-(azinodimethylidyne) *bis*-benzonitrile, $C_{16}H_{10}N_4$ (CN//CN) have been determined at −100 °C. Cell parameters for CN/CN: $a = 4.7270(9)$ Å, $b = 10.443(2)$ Å, $c = 11.943(2)$ Å; $\alpha = 90°$, $\beta = 98.70(3)°$, $\gamma = 90°$; monoclinic, space group $P2_1/c$. Cell parameters for CN//CN: $a = 3.8008(8)$ Å, $b = 7.9627(16)$ Å, $c = 11.181(2)$ Å; $\alpha = 70.23(3)°$, $\beta = 84.66(3)°$, $\gamma = 81.93(3)°$; triclinic, space group $P\bar{1}$. Both molecules occupy crystallographic inversion centers, which requires that (CN/CN) be disordered. Both molecules assume nearly planar conformations in the solid state. Neither (CN/CN) nor (CN//CN) is isostructural with its halogen-nitrile substituted analogues, although (CN/CN) is found to be isostructural with the corresponding stilbene. Both (CN/CN) and (CN//CN) engage in centrosymmetric H-bonding interactions that define an $R_2^2(10)$ motif. This motif is also observed in many related structures, although noteworthy exceptions can be found.

Keywords Benzylideneaniline hydrazone · Nitrile · Disorder · Weak H-bonding

Introduction

Solid-state intermolecular interactions involving the nitrile group play a significant role in determining molecular packing and are therefore of particular interest from the standpoint of crystal engineering. How the C≡N group exerts its influence has been examined in several different contexts in solid-state investigations, reflecting the variety of packing motifs in which it appears. The nitrile group can serve as an H-bond acceptor in crystals, frequently participating in relatively weak and unconventional hydrogen bonding and forming part of a commonly occurring H-bonded $R_2^2(10)$ supramolecular synthon (Fig. 1a) [13]. As a polar functional group, it engages in dipole-dipole interactions that help determine molecular packing and that have been the focus of recent research (Fig. 1b) [35]; exclusively non-centrosymmetric interactions have been examined by [20]. Yet another role played by this group in the packing of crystalline solids is as a Lewis base capable of interacting with Lewis acids, particularly with halogen atoms on neighboring molecules (Fig. 1c) [11, 12, 22, 29].

It is primarily the role of the nitrile group as a Lewis base in the solid state that we have examined in previous work, reporting the packing arrangements of isomeric pairs of *p*-cyano-*N*-(*p*-halobenzylidene)anilines and *p*-halo-*N*-(*p*-cyanobenzylidene)anilines, where the halogen atoms are chlorine, bromine, and iodine [26, 27]. A substantial number of benzylideneaniline crystal structures show disorder of the molecules: end-for-end or about the long molecular axis or both [2, 15–17, 26, 37]. By examining benzylideneanilines bearing both a nitrile group and a

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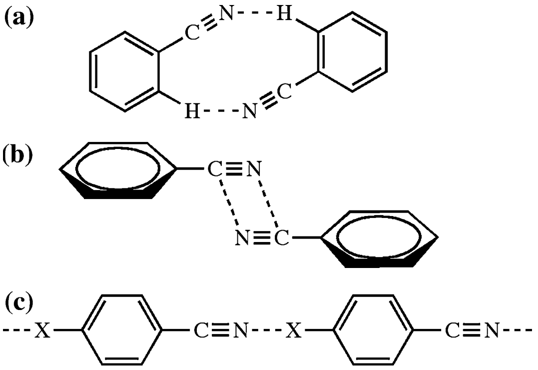

Fig. 1 Solid-state packing motifs involving the nitrile group: **a** centrosymmetric interaction with approaches to neighboring H atoms; **b** dipole–dipole interaction; **c** Lewis base-Lewis acid interaction with halogen atoms

halogen substituent, we sought to determine whether or not interactions of the Lewis acid-Lewis base type between nitrile groups and halogen atoms on neighboring molecules could reduce or eliminate the disorder. In several of these structures, CN···X interactions were in fact found to be important to the packing. In the iodine-nitrile benzylideneanilines LALMEQ and LALNUH [27], (Fig. 2), close iodine-nitrile interactions were observed; both structures are ordered. In the bromine-substituted cyanobenzylidene structure MUTZIK and its isostructural chlorine-substituted analogue MUTZOQ [26], (Fig. 2) disorder was found even though halogen-nitrile contacts were found as well. That these contacts are insufficient to eliminate the disorder is consistent with the relative weakness of bromine and chlorine compared to iodine with respect to strength as Lewis acids. Similarly, it has been reported by other workers that no evidence of bromine-nitrile interactions is found in the crystal structure of (*E*)-4-(4-bromostyryl) benzonitrile [21]. In our determination of the bromine- and chlorine-substituted cyanoaniline structures MUTZUW and MUVBAG [26], (Fig. 2), which are isostructural with each other but not with any of the other four, we found neither disorder nor halogen-nitrile contacts, the disorder being removed by halogen-halogen interactions. In further studies, we reported the crystal structures of the corresponding 4-cyano-4′-halo-*bis*-benzaldehyde hydrazones [25], (Fig. 2). In DIWGIA (X = I), DIWGEW (X = Br), and one polymorph of DIWGAS (X = Cl), halogen-nitrile contacts are present, while a second polymorph of DIWGAS lacks them; regardless, all four structures show partial disorder between the two ends of the molecule.

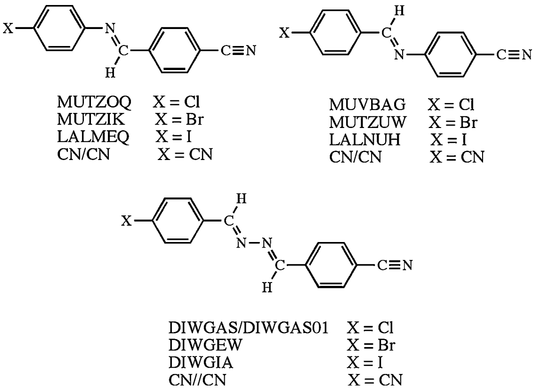

Fig. 2 Substituted benzylideneanilines and *bis*-benzaldehyde hydrazones examined in previous work and in this report

In the disordered structures noted above, differences in size and shape between the halogen atom and the nitrile group are sufficiently small so that each of these substituents can replace the other in the disordered packing arrangement. On the other hand, replacing the halogen atom with another nitrile group would eliminate any differences between the two substituents entirely. Would the resulting highly symmetrical dicyanobenzylideneaniline or dicyano-*bis*-benzaldehyde hydrazone necessarily be disordered, or would intermolecular interactions such as CN···H–C contacts acting in the absence of the nitrile-halogen interaction be sufficient to fix the molecules into an ordered crystal structure? To answer this question, we have synthesized the two relevant compounds, *p*-cyano-*N*-(*p*-cyanobenzylideneaniline) (CN/CN) and 4,4′-(azinodimethylidyne)*bis*-benzonitrile (CN//CN) (Fig. 2), and have determined their crystal structures.

Experimental

The preparation of the benzylideneaniline (CN/CN) was accomplished using the common method, refluxing the substituted benzaldehyde and substituted aniline in ethanol for approximately 15 min and allowing the solution to cool slowly. Recrystallization from dimethylformamide (DMF) of the solid thus obtained yielded high-melting yellow needles, mp = 230 °C.

The preparation of the *bis*-cyanobenzaldehyde hydrazone (CN//CN) was accomplished in two steps, the preparation of 4-cyanobenzaldehyde hydrazone followed by its condensation with 4-cyanobenzaldehyde. For the preparation of the hydrazone, a solution of 4-cyanobenzaldehyde (0.5 g, 3.8 mmol) dissolved in approximately 10 mL of ethanol was added dropwise with stirring to an aqueous 8% hydrazine solution (14.25 g, 36 mmol hydrazine). The milky reaction mixture was stirred for approximately 30 min after completion of the addition and then was refrigerated overnight (at 4 °C). The solid hydrazone (mp = 63 °C) was removed by filtration and used without recrystallization. A portion of it (0.06 g, 0.40 mmol) was added to a solution of 4-cyanobenzaldehyde (0.06 g, 0.45 mmol) dissolved in 10 mL of absolute ethanol. The mixture was heated (lower than 50 °C) with stirring for approximately 1 h, cooled, and then refrigerated overnight. The crude *bis*-cyanobenzaldehyde hydrazone was recrystallized from DMF, yielding yellow needles. Like (CN/CN), (CN//CN) proved to be a high-melting solid, mp = 319 °C.

Data sets for (CN/CN) and (CN//CN) were collected at −100 °C on a Bruker-AXS *SMART* CCD diffractometer using Mo Kα radiation (λ = 0.71073 Å). For the benzylideneaniline (CN/CN), the data were integrated with *SAINT* [6] and corrected for absorption with *SADABS* [4, 31]. Structure solution and refinement were accomplished using *SHELXTL* [33]. The structure was found to be disordered, the molecules being located on crystallographic inversion centers in $P2_1/c$ with $Z = 2$ ($Z' = 0.5$). In the refinement cycles, C7 and N1 were constrained to have the same position and the same anisotropic displacement parameters.

The structure determination for the *bis*-cyanobenzaldehyde hydrazone (CN//CN) was complicated by twinning. Most of the crystals in fact appeared to be twinned, and several that did not appear on visual inspection to be twinned proved to be twinned nonetheless during attempts at unit cell determination. Data for (CN//CN) were ultimately collected on a non-merohedrally twinned crystal grown from DMF. The crystal used was indexed using *CELLNOW* [32]. The major twin component fitted 192 reflections; the minor twin component fitted 164 of the overall 270 reflections used. The twin law was found to be [100/−0.613,−1,0/0,0,−1], which corresponds to a 180° rotation about the *a* axis. The data were integrated with *SAINT* [6] and corrected for absorption and scaling with *TWINABS* [4, 31]. The ratio of the major and minor twin components was 0.672(3):0.328(3) based on refinement using *SHELXTL* [33]. As in (CN/CN), molecules of (CN//CN) were found to be located on crystallographic inversion centers, giving $Z = 1$ ($Z' = 0.5$) in space group $P\bar{1}$.

Hydrogen atoms, with the exception of H7 in (CN/CN), were included in both structures at geometrically idealized positions and constrained to ride on their parent atoms with C–H distances of 0.95 Å and with $U_{\mathrm{iso}}(\mathrm{H}) = 1.2\ U_{\mathrm{eq}}(\mathrm{C})$. H7 was allowed to refine to avoid problems arising from the disorder.

Table 1 Crystal data, data collection, and refinement parameters

Compound	(CN/CN)	(CN//CN)
Molecular formula	$C_{15}H_9N_3$	$C_{16}H_{10}N_4$
CCDC deposition number	CCDC763127	CCDC763128
M_r	231.25	258.28
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/c$	$P\bar{1}$
a (Å)	4.7270(9)	3.8008(8)
b (Å)	10.443(2)	7.9627(16)
c (Å)	11.943(2)	11.181(2)
α (°)	90	70.23(3)
β (°)	98.70(3)	84.66(3)
γ (°)	90	81.93(3)
V (Å^3)	582.8(2)	314.93(11)
Z	2	1
T (°C)	−100(2)	−100(2)
Radiation	Mo $K\alpha$, λ = 0.71073 Å	Mo $K\alpha$, λ = 0.71073 Å
D_{calc} (mg/m^3)	1.318	1.362
F_{000}	240	134
μ (mm^{-1})	0.08	0.09
Crystal dimensions (mm)	0.50 × 0.15 × 0.15	0.30 × 0.20 × 0.15
Abs. corr.	*SADABS* [4, 31]	*TWINABS* [4, 31]
$T_{\min}$, $T_{\max}$	0.94, 0.99	0.96, 0.99
Meas/indep/obs($I > 2\sigma(I)$)	6558/1322/1137	5504/2521/1966
R_{int}	0.028	0.000
R [$F^2 > 2\sigma(F^2)$]	0.039	0.052
$wR(F^2)$	0.119	0.157
S	1.07	1.00
Reflections/parameters/restraints	1322/86/0	2521/92/0
H-atom treatment	Mixed independent and constrained	Constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å^{-3})	0.23, −0.18	0.26, −0.27

Results and Discussion

Unit cell parameters and structure determination data are listed in Table 1. The atom labeling and the anisotropic displacement parameters for both (CN/CN) and (CN//CN) are shown in Fig. 3. As noted previously, both (CN/CN) and (CN//CN) lie on centers of symmetry, which for (CN/CN) requires disorder between C7 and N1. No evidence of disorder is observed in (CN//CN). The bond lengths and angles in both molecules are normal. In both molecules the benzene rings are tilted only slightly out of the plane of the central bridging atoms: by 1.4(1)° in (CN/CN) and 1.1(1)° in (CN//CN). Planarity (or near-planarity) in benzylideneanilines is rare but not unprecedented [1, 10, 23, 36].

The C≡N···H–C contact geometry defining the $R_2^2(10)$ motif shown in Fig. 1a and found in both (CN/CN) and (CN//CN) is given in Table 2. In Fig. 4 are shown two molecules of (CN/CN) at xyz and $-1-x$, $2-y$, $1-z$ assembled into a ribbon parallel to the [2,−1,0] direction through this interaction, as well as two molecules (at $-1-x$, $-1/2+y$, $1/2-z$ and x, $3/2-y$, $1/2+z$) from

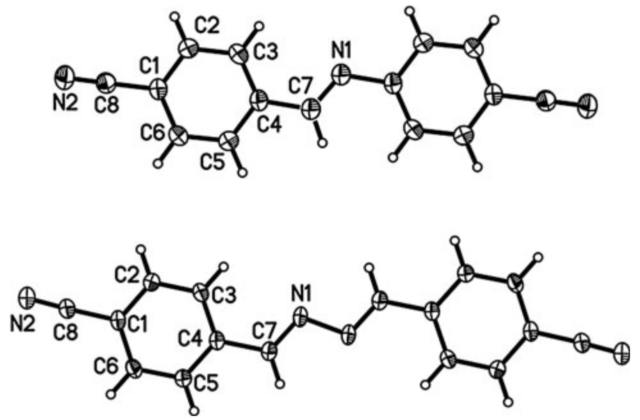

Fig. 3 *Above*: (CN/CN). *Below*: (CN//CN). Only the crystallographically independent atoms are *numbered*. Both molecules lie on a center of symmetry, which means that in (CN/CN) atoms *C7* and *N1* are disordered with respect to each other. Displacement ellipsoids are shown at the 50% probability level and H atoms are shown as small *spheres* of arbitrary radius

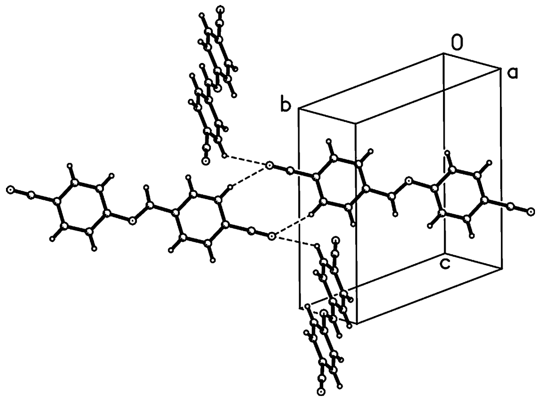

Fig. 4 Two molecules of (CN/CN) assembled into a ribbon parallel to the [2,−1,0] direction (*middle* of figure, molecules at xyz and $-1-x$, $2-y$, $1-z$), and molecules from two adjacent ribbons (*upper left*: $-1-x$, $-1/2+y$, $1/2-z$; *lower right*: x, $3/2-y$, $1/2+z$). The *dashed lines* show the centrosymmetric H6···N2 approaches of 2.56 Å within the ribbon and the H2···N2 approaches of 2.79 Å between neighboring ribbons

adjacent ribbons. Nonlinear interactions of this type between nitrile groups and H-bond donors are commonly observed [14]. The antiparallel C≡N···C≡N interactions (as measured from the midpoints of the C≡N bonds of the centrosymmetrically related molecules at xyz and $-1-x$, $2-y$, $1-z$) are at a distance of 3.765(2) Å, considerably larger than the 3.57 Å distance considered by Lee et al. [20] to be the dipole–dipole distance limit for C≡N···C≡N interactions that are not part of an $R_2^2(10)$ motif. The second molecule is displaced from the mean plane of the first by 0.767(14) Å. The ribbons assemble into layers parallel to the (001) plane; the perpendicular distance between nearest benzene rings in this layer is 3.490(4) Å, but the centroid–centroid separation of 4.8124(11) Å precludes a significant interaction of the π–π type. The ribbons in the next adjacent layer lie along the [210] direction and are rotated 84.3(1)° with respect to the ribbons in the first layer. The same packing motifs are found in the crystal structure of the corresponding stilbene derivative, 1,2-bis(4-cyanophenyl)ethylene [24], with which (CN/CN) is isostructural. (CN/CN) is not isostructural with any of the halogen-nitrile benzylideneanilines we described previously (Fig. 2).

In Fig. 5 are shown four molecules of (CN//CN) assembled into two ribbons parallel to the [2,−1,−1] direction, molecules within each ribbon being connected through the $R_2^2(10)$ motif shown in Fig. 1a. The antiparallel C≡N···C≡N interaction (as measured from the midpoints of the C≡N bonds of the centrosymmetrically related molecules at xyz and $3-x$, $-y$, $-z$) is at a distance of 3.560(2) Å, which is comparable to the dipole–dipole limit considered by Lee et al. [20]. The second molecule is displaced from the mean plan of the first by 0.62(4) Å. The ribbons are stacked into layers parallel to the (0,1,−1) plane through π–π contacts; the perpendicular distance between nearest benzene rings in this layer is 3.420(2) Å with a centroid-to-centroid distance of 3.8008(8) Å (the a-axis repeat distance). The ribbons in the next adjacent layer also lie along the [2,−1,−1] direction and are parallel to the ribbons in the first layer, being related to these by translation. This packing involves a second H···N contact, C≡N···H5; together these contacts define a centrosymmetric $R_4^2(10)$ motif also shown in Fig. 5 (and shown schematically in Fig. 6; see Table 2 for contact parameters) that occurs frequently in the crystal structures of nitriles [28]. Here it occurs in (CN//CN) but not in (CN/CN),

Table 2 Intermolecular C≡N···H–C contact geometry

Contacts	N···H (Å)	N···C (Å)	C≡N···H (°)	N···H–C (°)
(CN/CN)				
C8≡N2(xyz)···H6–C6($-1-x$, $2-y$, $1-z$)	2.56	3.4420 (19)	133	155
C8≡N2(xyz)···H2–C2($-1-x$, $-1/2+y$, $1/2-z$)	2.79	3.398 (2)	155	123
(CN//CN)				
C8≡N2(xyz)···H6–C6($3-x$, $-y$, $-z$)	2.60	3.4662 (16)	141	152
C8≡N2(xyz)···H5–C5(x, $-1+y$, z)	2.70	3.591 (2)	131	157

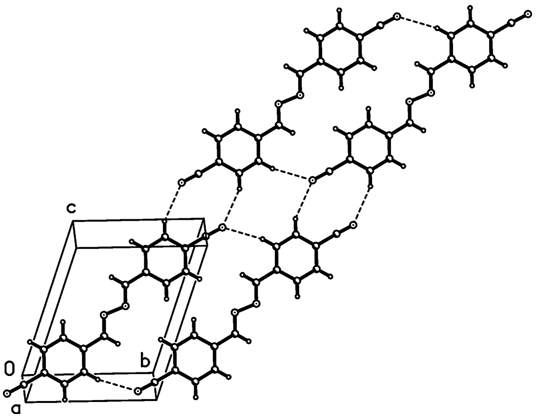

Fig. 5 Four molecules of (CN//CN) at *xyz*; x, $1 + y$, z; $-2 + x$, $1 + y$, $1 + z$; and $-2 + x$, $2 + y$, $1 + z$ assembled into two ribbons parallel to the [2,−1,−1] direction; molecules within each ribbon are connected via the $R^2_2(10)$ motif, and adjacent ribbons are connected via the $R^2_4(10)$ motif (center of figure). The dashed lines show the centrosymmetric H6···N2 approaches of 2.60 Å within the ribbons and the centrosymmetric H5···N2 approaches of 2.70 Å between neighboring ribbons

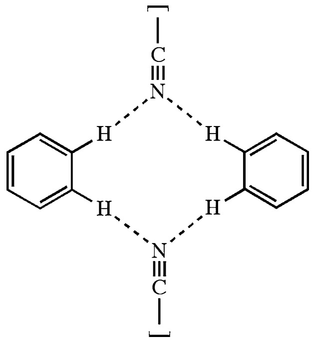

Fig. 6 Centrosymmetric $R^2_4(10)$ packing motif observed in (CN//CN) but not in (CN/CN)

where the rotation of each layer of ribbons with respect to its neighboring layers disrupts this motif. As (CN/CN) is not isostructural with the halogen-nitrile benzylideneanilines described previously, neither is (CN//CN) isostructural with the halogen-nitrile *bis*-benzaldehyde hydrazones described previously (Fig. 2).

Formation of centrosymmetric dimers connected by the $R^2_2(10)$ motif shown in Fig. 1a is common in cyano-substituted compounds. Two examples relevant to (CN//CN) are 4,4′-[ethylenebis(nitrilomethylidyne)]dibenzonitrile [19], related to (CN//CN) by the addition of an ethyl spacer in the center of the molecule; and (*E,E*)-*p*-cyanoacetophenone azine [9], related to (CN//CN) by the replacement of the bridge hydrogen atoms by methyl groups. In the former structure, molecules are connected by means of the $R^2_2(10)$ contact, which is located about an inversion center in space group $P\bar{1}$. In the latter structure, the molecule is planar in spite of the presence of the bulky methyl groups on the bridge, and the $R^2_2(10)$ motif is again present and located around a crystallographic inversion center in space group $P2_1/n$. In contrast, and especially striking in light of the results presented here for (CN/CN), is the case of 4,4′-dicyanobiphenyl [5]. No conformation of the (CN/CN) molecule can be centrosymmetric, yet the molecule occupies a crystallographic inversion center by means of disorder. On the other hand, the 4,4′-dicyanobiphenyl molecule, which in a planar conformation would be centrosymmetric and capable of occupying an inversion center (as does the unsubstituted biphenyl molecule: [3, 7, 8, 18, 30, 34], instead assumes a non-centrosymmetric, twisted conformation and occupies a general position in space group $P2_1$. In the biphenyl structure the two $C{\equiv}N\cdots C{\equiv}N$ approaches are crystallographically independent (3.514 and 3.610 Å) rather than related by inversion symmetry. The associated $C{\equiv}N\cdots H$ interactions, rather than defining centrosymmetric dimers as in (CN/CN), instead define chains of molecules extending through the crystal, and inversion symmetry is absent from the packing arrangement.

Supplementary Material

CCDC763127 and CCDC763128 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/data_request/cif, or by e-mailing data_request@ccdc.cam.ac.uk, or by contacting the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44(0)1223-33603.

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