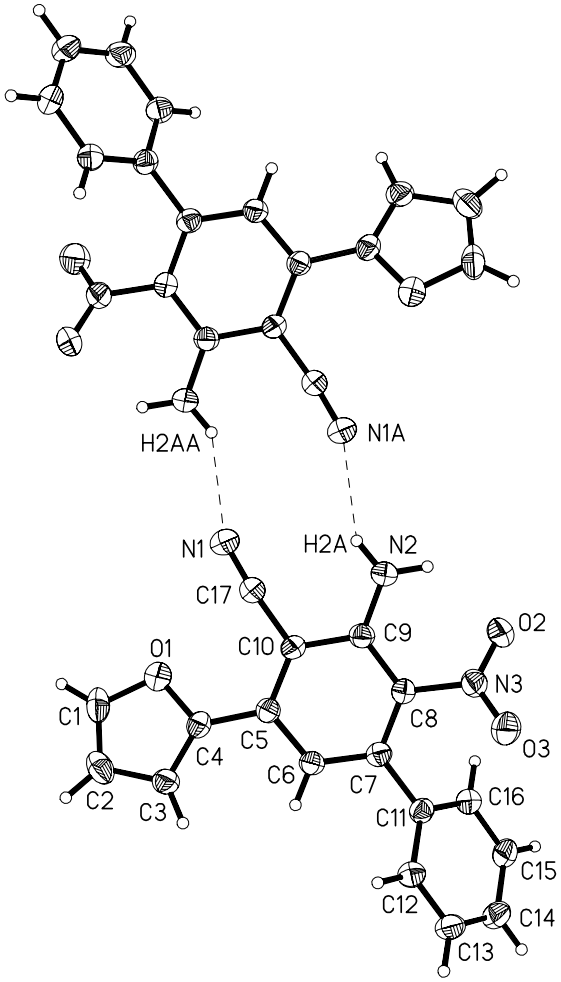
Crystal structure of 3-amino-5-(furan-2-yl)-2-nitrobiphenyl-4-carbonitrile, $C_{17}H_{11}N_3O_3$

Jie Li, Dong Xue and Zun-Ting Zhang*

Shaanxi Normal University, School of Chemistry and Materials Science, Xi'an, Shaanxi 710062, P. R. China

Received April 10, 2007, accepted and available on-line October 12, 2007; CCDC no. 1267/2053



Abstract

$C_{17}H_{11}N_3O_3$, trigonal, $R\bar{3}$ (no. 148), a = 36.612(2) Å, c = 5.5797(3) Å, V = 6477.5 Å^3, Z = 18, $R_{gt}(F)$ = 0.042, $wR_{ref}(F^2)$ = 0.121, T = 296 K.

Source of material

A 25 mL round bottom flask was charged with 158 mg 2-(1-(furan-2-yl)ethylidene)malononitrile, 149 mg (2-nitrovinyl)benzene and 68 mg EtONa. The mixture was refluxed with vigorous stirring at 343 K for 4 hours. The solvent was removed under reduced pressure. The product was purified by column chromatography (EtOAc/hexanes, 1:8) to yield 378 mg (76 %) light yellow solid. Single crystals suitable for X-ray diffraction were obtained by recrystallization from *n*-hexane and slow evaporation at room temperature.

* Correspondence author (e-mail: zhangzt@snnu.edu.cn)

Experimental details

The H atoms (H2A, H2B) at N2 were found from Fourier difference maps and refined freely. The N_{gt}/N_{param} ratio was relatively small due to the poor quality of the crystals.

Discussion

Polysubstituted benzenes and aromatic compounds are widely used in the chemical industry as well in the laboratory. Many liquid crystals belong to the group of polysubstituted benzenes and their derivatives. For pharmaceutical and agrochemical preparations, people recognize importance to aromatic or heteroaromatic components and their derivatives. In this paper, we report about the synthesis of 3-amino-5-(furan-2-yl)-2-nitrobiphenyl-4-carbonitrile and its structure.

The title compound is composed of two phenyl moieties, a furan ring moiety, a cyano group, an amino group and a nitro group. The cyano group at atom C10 is nearly coplanar with the furan ring (C5→C10; A), as indicated by the C6–C5–C10–C17 torsion angle of 176.7(2)°; the amino group at C9 atom is also coplanar with the ring A, the C7–C8–C9–N2 torsion angle being –179.5(2)°. Because of the large volume, the nitro group at atom C8 and the benzene ring A are twisted, the torsion angles are –36.4(2)° (C9–C8–N3–O2) and –34.2(3)° (C7–C8–N3–O3). To avoid steric conflicts, three rigid rings, substituted benzene ring A, furan ring (O1, C1→C4; B) and benzene ring (C11→C16; C) are not coplanar. They are rotated by 21.1(1)° (for A/B) and 48.84(7)° (for A/C). A dimer is formed by N–H···N^i(i: ⅔–x,⅓–y,–⅔–z) hydrogen bonding interactions. The distance of N···N^i is 3.099(3) Å. The paired N–H···N made a synthon $R^2_2(12)$. In the crystal structure, a 3_1 screw axis down generates $C(3)$ helical chains of molecules linked by C–H···π stacking interactions, with a C–H···Cg^{ii} (ii: ⅓–x+y,⅔–y,–⅓+z) separation of 2.847 Å and stacked along [001]. Crosslinks between these molecule are provided by N–H···N hydrogen bonds and C–H···π interactions, leading to a three-dimensional supermolecular structure.

Table 1. Data collection and handling.

Crystal:	yellow needle, size 0.14 × 0.20 × 0.41 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.00 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.2°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11180, 2568
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1626
$N(param)_{refined}$:	217
Programs:	SHELXS-97 [6], SHELXL-97 [7]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	18*f*	0.2902	0.3115	–0.3128	0.085
H(2)	18*f*	0.2618	0.3267	0.0352	0.083
H(3)	18*f*	0.2377	0.2652	0.3173	0.066
H(6)	18*f*	0.2061	0.1921	0.3808	0.056
H(12)	18*f*	0.1885	0.1662	0.7942	0.063
H(13)	18*f*	0.1297	0.1323	1.0361	0.076
H(14)	18*f*	0.0786	0.0644	0.9537	0.080
H(15)	18*f*	0.0856	0.0295	0.6307	0.076
H(16)	18*f*	0.1446	0.0624	0.3883	0.064
H(2A)	18*f*	0.3020(7)	0.1329(7)	–0.072(4)	0.066(7)
H(2B)	18*f*	0.2772(8)	0.0951(9)	0.092(4)	0.077(8)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(17)	18*f*	0.30307(7)	0.19711(7)	–0.1338(4)	0.063(2)	0.054(1)	0.061(1)	0.035(1)	0.014(1)	0.010(1)
N(2)	18*f*	0.28665(7)	0.12224(7)	0.0589(4)	0.061(1)	0.054(1)	0.069(1)	0.035(1)	0.019(1)	0.008(1)
N(3)	18*f*	0.22808(5)	0.08323(6)	0.4411(4)	0.052(1)	0.052(1)	0.070(1)	0.0285(9)	0.0073(9)	0.009(1)
O(1)	18*f*	0.27713(6)	0.25575(6)	–0.1539(3)	0.074(1)	0.060(1)	0.070(1)	0.034(1)	0.014(1)	0.011(1)
O(2)	18*f*	0.23102(6)	0.05680(5)	0.3212(3)	0.096(1)	0.049(1)	0.103(1)	0.041(1)	0.033(1)	0.0109(9)
O(3)	18*f*	0.22089(6)	0.07901(6)	0.6565(3)	0.101(1)	0.091(1)	0.064(1)	0.063(1)	0.011(1)	0.0233(9)
C(1)	18*f*	0.27922(8)	0.29408(7)	–0.1796(5)	0.074(2)	0.048(1)	0.085(2)	0.027(1)	0.001(1)	0.020(1)
C(2)	18*f*	0.26365(7)	0.30260(7)	0.0098(5)	0.050(1)	0.048(1)	0.114(2)	0.027(1)	–0.006(1)	–0.004(1)
C(3)	18*f*	0.25016(7)	0.26810(7)	0.1681(4)	0.052(1)	0.047(1)	0.061(1)	0.021(1)	0.008(1)	–0.007(1)
C(4)	18*f*	0.25874(5)	0.24044(5)	0.0627(3)	0.040(1)	0.047(2)	0.049(2)	0.018(1)	0.001(1)	0.005(1)
C(5)	18*f*	0.25150(6)	0.19968(6)	0.1459(3)	0.039(1)	0.041(1)	0.048(1)	0.0172(9)	0.0000(9)	–0.0012(9)
C(6)	18*f*	0.22045(6)	0.17907(6)	0.3214(3)	0.045(1)	0.044(1)	0.050(1)	0.022(1)	0.0040(9)	–0.0023(9)
C(7)	18*f*	0.21069(6)	0.13992(6)	0.4083(3)	0.044(1)	0.047(1)	0.043(1)	0.021(1)	0.0011(9)	–0.0020(9)
C(8)	18*f*	0.23387(6)	0.12134(6)	0.3256(3)	0.047(1)	0.042(1)	0.049(1)	0.021(1)	0.005(1)	0.0036(9)
C(9)	18*f*	0.26415(6)	0.13967(6)	0.1425(3)	0.043(1)	0.045(1)	0.050(1)	0.022(1)	0.0021(9)	–0.0021(9)
C(10)	18*f*	0.27240(6)	0.17950(6)	0.0550(3)	0.042(1)	0.046(1)	0.047(1)	0.020(1)	0.0060(9)	0.0025(9)
C(11)	18*f*	0.17325(6)	0.11774(6)	0.5687(3)	0.048(1)	0.050(1)	0.042(1)	0.026(1)	0.0017(9)	0.0054(9)
C(12)	18*f*	0.16806(7)	0.13845(7)	0.7613(3)	0.056(1)	0.055(1)	0.047(1)	0.028(1)	0.002(1)	–0.002(1)
C(13)	18*f*	0.13270(8)	0.11827(8)	0.9055(4)	0.074(2)	0.076(2)	0.051(1)	0.046(2)	0.014(1)	0.006(1)
C(14)	18*f*	0.10229(8)	0.07787(8)	0.8564(4)	0.062(2)	0.071(2)	0.067(2)	0.034(1)	0.022(1)	0.018(1)
C(15)	18*f*	0.10655(7)	0.05698(7)	0.6639(4)	0.059(2)	0.051(1)	0.073(2)	0.022(1)	0.016(1)	0.014(1)
C(16)	18*f*	0.14186(7)	0.07668(7)	0.5189(4)	0.057(1)	0.050(1)	0.053(1)	0.027(1)	0.006(1)	0.002(1)
N(1)	18*f*	0.32697(8)	0.20786(7)	–0.2847(4)	0.096(2)	0.083(2)	0.089(2)	0.058(1)	0.046(1)	0.030(1)

References

1. Rappoport, Z.: The Chemistry of Phenols. Wiley-VCH, New York 2003.
2. Dotz, K. H.; Tomuschat, P.: Annulation reactions of chromium carbene complexes: scope, selectivity and recent developments. Chem. Soc. Rev. **28** (1999) 187-198.
3. Wang, H.; Huang, J.; Wulff, W. D.; Rheingold, A. L.: The First Examples of a Meta-Benzannulation from the Reaction of Fischer Carbene Complexes with Alkynes. J. Am. Chem. Soc. **125** (2003) 8980-8981.
4. Wang, X.-B.; Zhang, Z.-T.; Wang, Q.-Y.: Hydrogen Bonding and Aromatic π-π Stacking in the Crystal Structures of Water-Soluble Daidzein Derivatives. Struct. Chem. **16** (2005) 461-468.
5. Zhang, Z.-T.; Wang, Q.-Y.: Syntheses and crystal structures of diethylated daidzein sulfonates. Heterocycles **65** (2005) 1947-1956.
6. Sheldrick, G. M.: Phase Annealing in SHELX90: Direct Methods for Larger Structures. Acta Crystallogr. A**46** (1990) 467-473.
7. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.