Crystal structure of *N,N'*-bis(4-nitrophenyl)-1,4-naphthalenedicarboxamide bis(dimethylsulfoxide) solvate, $C_{24}H_{16}N_4O_6 \cdot 2C_2H_6OS$

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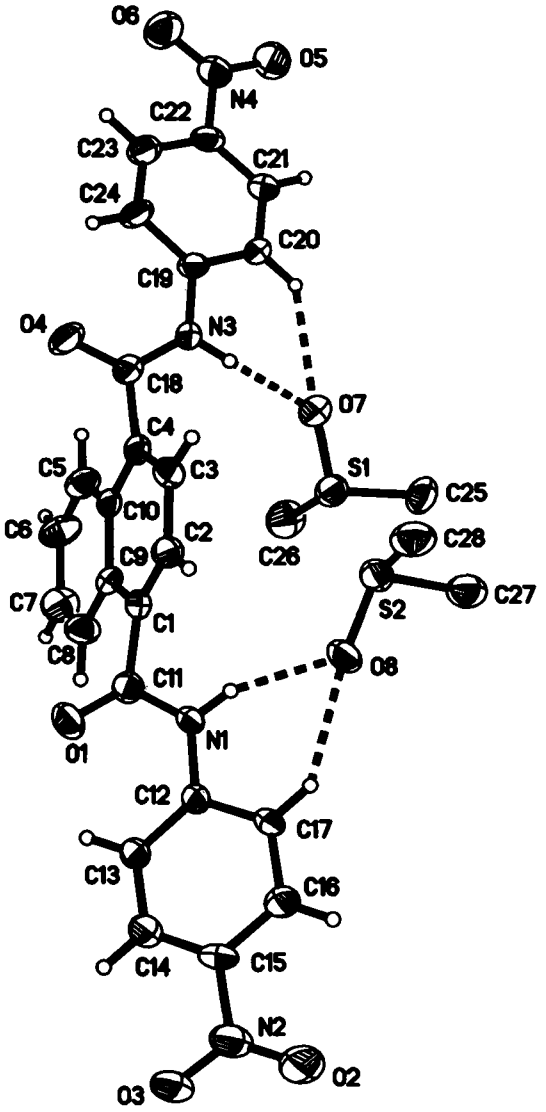
Received April 21, 2006, accepted and available on-line May 15, 2006; CCDC no. 1267/1768



Abstract

$C_{28}H_{28}N_4O_8S_2$, monoclinic, *I*12/*a*1 (no. 15), $a = 23.820(5)$ Å, $b = 9.166(3)$ Å, $c = 26.778(6)$ Å, $\beta = 95.58(2)°$, $V = 5818.9$ Å^3, $Z = 8$, $R_{gt}(F) = 0.069$, $wR_{ref}(F^2) = 0.176$, $T = 290$ K.

Source of material

Naphthalene-1,4-dicarboxylic acid (2 mmol) and an excess of thionyl chloride in dioxane (20 ml) was boiled under reflux for 6 hours; the solution was distilled by reduced pressure and the yellow solid was acquired. *p*-Nitroaniline (4 mmol) in 20 ml tetrahydrofuran was added to the yellow solid and boiled under reflux for 1 day; the solution was cooled to ambient temperature and filtered to remove the tetrahydrofuran. The precipitate was dissolved in the dimethylsulfoxide, kept for one month at ambient temperature, and brown single crystals suitable for X-ray diffraction were obtained.

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Experimental details

The low N_{gt}/N_{param} ratio is probably caused by the poor quality of the crystals.

Discussion

1,4-naphthalic dicarboxylic acid derivatives are a class of intermediates important for applications as monomers in the preparation of polymers or printing receptor [1-3]. To extend this research, we have synthesized the title compound and determined its crystal structure.

In the crystal structure, the significant shorter bonds of N1—C11 (1.358(7) Å), N1—C12 (1.412(6) Å), N3—C18 (1.348(8) Å) and N3—C19 (1.403(7) Å) indicate the electron delocalization. Both N atoms (N1 and N3) have effectively planar coordination. The dihedral angle between the C15/N2/O2/O3 and C12 – C17 planes is 8.4(4)° and that between C22/N4/O5/O6 and C19 – C24 planes is 4.5(2)°. The dihedral angle between the C1/C11/O1/N1 and C12 – C17 planes is 2.7(4)° and that between C4/C18/N3/O4 and C19 – C24 planes is 10.5(2)°. The dihedral angle between the C1/C11/O1/N1 and C1 – C4/C9/C10 planes is 43.9(3)° and that between C4/C18/N3/O4 and C1 – C4/C9/C10 planes is 80.2(2)°. The dihedral angle between the two aromatic rings (C1 – C4/C9/C10 and C5 – C10) of 2.8(2)° indicates that the naphthalene ring system is slightly deviated from planarity. In the molecule, there are two types of intermolecular hydrogen bonds. The first type are N–H···O bonds (N1–H1N···O8, *d*(H1N···O8) = 2.03 Å, and N3–H3N···O7, *d*(H3N···O7) = 1.88 Å). The other type are C–H···O bonds (C17–H17···O8, *d*(H17···O8) = 2.39 Å, and C20–H20···O7, *d*(H20···O7) = 2.45 Å).

Table 1. Data collection and handling.

Crystal:	brown block, size 0.22 × 0.22 × 0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.39 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5157, 5086
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1524
$N(param)_{refined}$:	381
Programs:	SHELXS-97 [4], SHELXL-97 [5]

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

Atom	Site	x	y	z	U_{iso}
H(1N)	8*f*	0.7546	0.3685	0.1768	0.078
H(3N)	8*f*	0.4876	0.3943	0.1149	0.078
H(2)	8*f*	0.6739	0.2715	0.1996	0.078
H(3)	8*f*	0.5759	0.2748	0.1939	0.078
H(5)	8*f*	0.5236	0.1462	0.0287	0.078
H(6)	8*f*	0.5685	0.1110	–0.0407	0.078
H(7)	8*f*	0.6671	0.1148	–0.0343	0.078
H(8)	8*f*	0.7182	0.1527	0.0406	0.078
H(13)	8*f*	0.8585	0.1205	0.1463	0.078
H(14)	8*f*	0.9533	0.1482	0.1679	0.078
H(16)	8*f*	0.9253	0.5282	0.2347	0.078
H(17)	8*f*	0.8286	0.4970	0.2150	0.078
H(20)	8*f*	0.4141	0.5415	0.0889	0.078
H(21)	8*f*	0.3185	0.5721	0.0678	0.078

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(23)	8*f*	0.2934	0.1485	0.0932	0.078
H(24)	8*f*	0.3884	0.1163	0.1149	0.078
H(25A)	8*f*	0.5685	0.8594	0.1126	0.078
H(25B)	8*f*	0.5392	0.8374	0.0582	0.078
H(25C)	8*f*	0.6051	0.8466	0.0672	0.078
H(26A)	8*f*	0.5825	0.4679	0.0244	0.078
H(26B)	8*f*	0.6134	0.6153	0.0153	0.078
H(26C)	8*f*	0.5474	0.6061	0.0063	0.078
H(27A)	8*f*	0.6802	0.8251	0.1547	0.078
H(27B)	8*f*	0.7081	0.8558	0.2092	0.078
H(27C)	8*f*	0.6423	0.8610	0.1978	0.078
H(28A)	8*f*	0.6617	0.5470	0.2836	0.078
H(28B)	8*f*	0.6304	0.6961	0.2734	0.078
H(28C)	8*f*	0.6961	0.6931	0.2855	0.078

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}
S(1)	8*f*	0.57506(7)	0.6219(2)	0.08908(7)	0.054(1)	0.055(1)	0.070(1)	–0.003(1)	0.0002(9)	0.005(1)
S(2)	8*f*	0.67234(7)	0.6254(2)	0.20404(7)	0.042(1)	0.067(1)	0.087(2)	0.004(1)	–0.011(1)	–0.027(1)
O(1)	8*f*	0.7686(2)	0.0925(5)	0.1194(2)	0.052(3)	0.065(4)	0.134(5)	0.019(3)	–0.023(3)	–0.041(3)
O(2)	8*f*	1.0252(2)	0.4744(7)	0.2349(2)	0.060(3)	0.124(5)	0.153(6)	–0.003(4)	–0.020(3)	–0.071(5)
O(3)	8*f*	1.0391(2)	0.2559(6)	0.2088(2)	0.043(3)	0.083(4)	0.118(4)	0.012(3)	0.000(3)	0.008(4)
O(4)	8*f*	0.4816(2)	0.0680(6)	0.1205(2)	0.044(3)	0.052(4)	0.216(7)	–0.008(3)	0.011(3)	0.031(4)
O(5)	8*f*	0.2193(2)	0.5065(6)	0.0565(2)	0.057(3)	0.074(4)	0.150(5)	0.016(3)	–0.008(3)	0.004(4)
O(6)	8*f*	0.2064(2)	0.2755(6)	0.0643(2)	0.056(4)	0.081(5)	0.186(6)	–0.007(3)	–0.001(4)	0.004(4)
O(7)	8*f*	0.5167(2)	0.5786(5)	0.1000(2)	0.063(3)	0.055(3)	0.139(5)	–0.010(3)	0.029(3)	–0.003(3)
O(8)	8*f*	0.7317(2)	0.5674(5)	0.2011(2)	0.044(3)	0.075(4)	0.124(4)	0.016(3)	–0.005(3)	–0.042(3)
N(1)	8*f*	0.7744(2)	0.3007(5)	0.1650(2)	0.033(3)	0.043(3)	0.061(4)	0.006(3)	0.002(3)	–0.012(3)
N(2)	8*f*	1.0080(2)	0.3563(9)	0.2171(2)	0.047(4)	0.096(6)	0.074(5)	0.008(4)	0.002(3)	–0.018(4)
N(3)	8*f*	0.4694(2)	0.3130(5)	0.1127(2)	0.042(3)	0.033(3)	0.075(4)	0.000(3)	0.007(3)	–0.003(3)
N(4)	8*f*	0.2368(2)	0.3821(8)	0.0655(2)	0.049(4)	0.067(5)	0.086(5)	0.009(4)	0.003(3)	–0.004(5)
C(1)	8*f*	0.6826(3)	0.2166(7)	0.1299(2)	0.044(4)	0.032(4)	0.060(5)	0.000(3)	0.009(4)	0.000(4)
C(2)	8*f*	0.6537(3)	0.2485(7)	0.1690(3)	0.047(4)	0.046(5)	0.063(5)	0.002(4)	0.004(4)	0.007(4)
C(3)	8*f*	0.5942(3)	0.2490(7)	0.1661(3)	0.057(4)	0.052(5)	0.065(5)	0.000(4)	0.023(4)	–0.008(4)
C(4)	8*f*	0.5636(3)	0.2112(7)	0.1220(3)	0.044(4)	0.032(4)	0.072(5)	0.000(3)	0.001(4)	0.004(4)
C(5)	8*f*	0.5628(3)	0.1509(7)	0.0313(3)	0.048(4)	0.064(5)	0.071(5)	–0.010(4)	–0.007(4)	0.010(5)
C(6)	8*f*	0.5891(3)	0.1281(9)	–0.0099(3)	0.056(5)	0.102(7)	0.061(5)	–0.024(5)	–0.006(4)	0.003(5)
C(7)	8*f*	0.6485(3)	0.1304(8)	–0.0059(3)	0.080(6)	0.093(6)	0.060(5)	–0.013(5)	0.027(4)	–0.025(5)
C(8)	8*f*	0.6790(3)	0.1550(7)	0.0389(3)	0.051(4)	0.076(6)	0.062(5)	–0.002(4)	0.013(4)	–0.017(4)
C(9)	8*f*	0.6526(2)	0.1839(6)	0.0826(2)	0.041(4)	0.038(4)	0.055(4)	–0.009(3)	0.004(3)	–0.007(3)
C(10)	8*f*	0.5919(3)	0.1817(6)	0.0784(2)	0.040(4)	0.028(4)	0.061(5)	0.001(3)	0.001(4)	0.006(3)
C(11)	8*f*	0.7460(3)	0.1967(8)	0.1366(2)	0.046(4)	0.046(5)	0.058(5)	0.002(4)	–0.004(4)	0.001(4)
C(12)	8*f*	0.8334(2)	0.3079(7)	0.1769(2)	0.039(4)	0.037(4)	0.061(5)	0.005(3)	–0.003(3)	–0.002(4)
C(13)	8*f*	0.8716(2)	0.2029(7)	0.1640(2)	0.040(4)	0.038(4)	0.088(5)	0.007(4)	–0.006(4)	0.007(4)
C(14)	8*f*	0.9283(3)	0.2192(7)	0.1770(2)	0.051(5)	0.053(5)	0.068(5)	0.006(4)	0.010(4)	0.002(4)
C(15)	8*f*	0.9485(2)	0.3390(8)	0.2032(2)	0.029(4)	0.074(6)	0.056(5)	0.006(4)	0.004(3)	0.008(4)
C(16)	8*f*	0.9115(3)	0.4459(8)	0.2172(2)	0.043(4)	0.064(5)	0.070(5)	–0.003(4)	–0.005(4)	–0.015(4)
C(17)	8*f*	0.8537(2)	0.4281(7)	0.2047(2)	0.036(4)	0.050(5)	0.069(5)	0.008(3)	0.008(3)	–0.016(4)
C(18)	8*f*	0.5012(3)	0.1915(8)	0.1191(3)	0.046(4)	0.042(5)	0.098(6)	–0.002(4)	0.013(4)	0.009(5)
C(19)	8*f*	0.4106(2)	0.3244(7)	0.1031(2)	0.033(4)	0.049(5)	0.057(4)	0.001(3)	0.007(3)	0.009(4)
C(20)	8*f*	0.3897(2)	0.4625(7)	0.0895(2)	0.042(4)	0.039(4)	0.060(4)	–0.006(3)	0.010(3)	0.000(4)
C(21)	8*f*	0.3330(2)	0.4810(8)	0.0772(2)	0.038(4)	0.058(5)	0.071(5)	0.003(4)	0.009(4)	–0.005(4)
C(22)	8*f*	0.2983(2)	0.3631(9)	0.0791(2)	0.033(4)	0.068(5)	0.058(4)	–0.001(4)	0.006(3)	–0.008(4)
C(23)	8*f*	0.3181(3)	0.2269(8)	0.0926(3)	0.048(5)	0.057(6)	0.096(6)	–0.001(4)	0.012(4)	0.017(5)
C(24)	8*f*	0.3744(3)	0.2077(7)	0.1051(2)	0.040(4)	0.052(5)	0.101(6)	–0.009(4)	0.008(4)	0.024(5)
C(25)	8*f*	0.5716(3)	0.8132(7)	0.0808(3)	0.076(5)	0.047(5)	0.112(6)	–0.011(4)	0.002(5)	0.006(5)
C(26)	8*f*	0.5802(3)	0.5722(8)	0.0267(2)	0.085(6)	0.097(7)	0.071(5)	–0.011(5)	0.004(4)	–0.014(5)
C(27)	8*f*	0.6762(3)	0.8133(7)	0.1898(2)	0.053(4)	0.088(6)	0.078(5)	0.001(4)	0.003(4)	–0.005(5)
C(28)	8*f*	0.6642(3)	0.6423(8)	0.2692(2)	0.050(4)	0.090(6)	0.114(7)	0.003(5)	0.001(4)	0.018(5)

Acknowledgment. The authors thank the Center for Test and Analysis, Sichuan University, for financial support.

References

1. Fukuzumi, T.; Tajiri, T.; Tsukada, H.; Yoshida, J.: Preparation of polyester stretched films with excellent stretchability, adhesion, heat resistance, and transparency. Japan Patent no. JP 06298919 (1994).
2. Tsukada, H.; Tajiri, T.; Fukuzumi, T.; Yoshida, J.: Preparation of flexible and transparent polyesters with excellent moldability and mechanical characteristics. Japan Patent no. JP 06298918 (1994).
3. Kuromya, Y.; Hirano, E.; Samu, F.: Sublimation-type thermal-transfer printing receptor containing naphthalene compound. Japan Patent no. JP 09048177 (1997).
4. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
5. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.