Crystal structure of *N*-[4-(9,10-dioxo-anthracene-1-ylamino-1,4-dienyl-amino)]-5-dimethylaminonaphthalene-sulfonamide, C₃₂H₂₅N₃O₄S

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Received April 18, 2005, accepted and available on-line April 27, 2005; CCDC no. 1267/1534

Abstract

C₃₂H₂₅N₃O₄S, monoclinic, *P*12₁/*c*1 (no. 14),
 $a = 19.895(4)$ Å, $b = 8.431(2)$ Å, $c = 16.394(3)$ Å,
 $\beta = 108.65(3)^\circ$, $V = 2605.5$ Å³, $Z = 4$,
 $R_g(F) = 0.045$, $wR_{\text{ref}}(F^2) = 0.105$, $T = 120$ K.

Source of material

The crystals were grown by vapor diffusion from petroleum ether onto commercial grade methanol/chloroform to produce thin brown plates.

Discussion

This molecule was synthesized within our research on multi-functional auto-quenchable organic fluorescence molecules [1]. Such molecules are composed of three distinct moieties, a redox unit, a spacer and a fluorophore, covalently bonded to each other.

The title compound is composed of an anthraquinone ring, which acts as a redox sub-unit of a *p*-phenylene diamine ring, which acts as a spacer and a 5-dimethylamino-naphthalene sulfonyl (dansyl) moiety, which acts as the fluorophore. These three units do not lie in the same molecular plane (figure, top). This molecule is composed of crystalline plates belonging to an achiral space group with one chiral asymmetric molecule in the asymmetric unit. One pair of molecules in the unit cell, which are related by an inversion center, have their anthraquinone rings anti-parallel one to the other. The distance between the two anthraquinone rings, as measured between corresponding pairs of atoms is 3.52(3) Å. The C14–C1...C15–C16 torsion angle between the anthraquinone ring and the *p*-phenylene diamine ring is 49.2° and is defined as the dihedral angle between the two aromatic planes. The torsion angles S1–N2–C18–C19 and N2–S1–C21–C30 between the *p*-phenylene diamine unit and the 5-dimethyl amino naphthalene rings are 64.4° and 55.1°, respectively. Each molecule has three hydrogen bonds. One is intramolecular between the N–H and the anthraquinone oxygen which lies on the same side of the molecule. The N(H)···O bond length is 1.80 Å. The other hydrogen bonds involve both the NH and SO atoms of the same NHSO₂ functionality and are intermolecular. The NH proton at this group is hydrogen-bonded to an oxygen atom in a 2₁ screw related NHSO₂ neighbor, while an NHSO₂ oxygen atom is hydrogen-bonded to the NH atom in a second 2₁ screw related molecule that is related by translation of the first neighbor. Thus, the NHSO₂ group is both an intermolecular H-donor and an H-acceptor. Both NH···OSO are the same length (2.07 Å), since both H-bonds involve two NHSO₂ moieties (figure, bottom).

Table 1. Data collection and handling.

Crystal:	brown plate, size 0.20 × 0.20 × 0.50 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.70 cm ⁻¹
Diffractionmeter, scan mode:	Nonius KappaCCD, ϕ/ω
$2\theta_{\text{max}}$:	49.42°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	4217, 4217
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3105
$N(\text{param})_{\text{refined}}$:	371
Programs:	SHELXS-97 [3], SHELXL-97 [4]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(5)	4e	0.5322	0.4162	0.2720	0.036
H(6)	4e	0.6463	0.3261	0.2875	0.041
H(7)	4e	0.7410	0.3987	0.4074	0.040

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Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(8)	4e	0.7232	0.5740	0.5083	0.036
H(12)	4e	0.5583	0.8696	0.6244	0.034
H(13)	4e	0.4435	0.9455	0.6156	0.033
H(14)	4e	0.3473	0.8710	0.5000	0.030
H(16)	4e	0.2771	0.9668	0.3559	0.029
H(17)	4e	0.1553	1.0070	0.3203	0.023
H(19)	4e	0.1225	0.5323	0.3048	0.021
H(20)	4e	0.2444	0.4912	0.3452	0.022
H(22)	4e	0.0504	0.5557	0.4794	0.020
H(23)	4e	0.1241	0.5971	0.6226	0.022
H(24)	4e	0.1768	0.8397	0.6624	0.023

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(27)	4e	0.1573	1.3400	0.5402	0.025
H(28)	4e	0.0666	1.3140	0.4085	0.025
H(29)	4e	0.0170	1.0697	0.3609	0.022
H(31A)	4e	0.2429	1.3476	0.6705	0.040
H(31B)	4e	0.2557	1.2565	0.7598	0.040
H(31C)	4e	0.1770	1.3055	0.7026	0.040
H(32A)	4e	0.2758	0.9521	0.6249	0.050
H(32B)	4e	0.3172	1.0515	0.7091	0.050
H(32C)	4e	0.3007	1.1298	0.6158	0.050
H(1)	4e	0.360(1)	0.627(3)	0.333(2)	0.043(8)
H(2)	4e	0.039(1)	0.886(3)	0.258(1)	0.022(7)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(1)	4e	0.4025(1)	0.7436(3)	0.4360(1)	0.018(1)	0.020(1)	0.022(1)	-0.003(1)	0.004(1)	0.004(1)
C(2)	4e	0.4727(1)	0.6970(3)	0.4402(1)	0.017(1)	0.022(1)	0.023(1)	-0.001(1)	0.003(1)	0.004(1)
C(3)	4e	0.4852(1)	0.5975(3)	0.3732(1)	0.016(1)	0.025(1)	0.027(1)	0.002(1)	0.005(1)	0.005(1)
O(1)	4e	0.43681(9)	0.5508(2)	0.3095(1)	0.018(1)	0.047(1)	0.0352(9)	0.0038(8)	0.0020(8)	-0.0129(9)
C(4)	4e	0.5589(1)	0.5478(3)	0.3824(1)	0.018(1)	0.024(1)	0.029(1)	0.000(1)	0.007(1)	0.007(1)
C(5)	4e	0.5708(1)	0.4476(3)	0.3206(2)	0.024(2)	0.033(2)	0.034(1)	0.004(1)	0.011(1)	0.003(1)
C(6)	4e	0.6385(1)	0.3939(3)	0.3299(2)	0.030(2)	0.035(2)	0.043(2)	0.007(1)	0.020(1)	0.006(1)
C(7)	4e	0.6948(1)	0.4384(3)	0.4004(2)	0.024(2)	0.035(2)	0.047(2)	0.007(1)	0.017(1)	0.016(1)
C(8)	4e	0.6841(1)	0.5406(3)	0.4609(2)	0.018(1)	0.036(2)	0.034(1)	0.001(1)	0.007(1)	0.016(1)
C(9)	4e	0.6163(1)	0.5950(3)	0.4530(1)	0.016(1)	0.023(1)	0.028(1)	0.001(1)	0.006(1)	0.010(1)
C(10)	4e	0.6047(1)	0.7004(3)	0.5195(2)	0.019(1)	0.031(2)	0.027(1)	-0.002(1)	0.001(1)	0.008(1)
O(2)	4e	0.65479(9)	0.7477(2)	0.5801(1)	0.020(1)	0.057(1)	0.038(1)	-0.0004(9)	-0.0021(8)	-0.0039(9)
C(11)	4e	0.5304(1)	0.7461(3)	0.5119(1)	0.017(1)	0.025(1)	0.026(1)	-0.000(1)	0.004(1)	0.005(1)
C(12)	4e	0.5193(1)	0.8376(3)	0.5765(1)	0.024(1)	0.031(2)	0.025(1)	-0.003(1)	0.002(1)	0.001(1)
C(13)	4e	0.4508(1)	0.8827(3)	0.5711(1)	0.030(2)	0.026(2)	0.025(1)	-0.001(1)	0.008(1)	-0.003(1)
C(14)	4e	0.3936(1)	0.8379(3)	0.5025(1)	0.021(1)	0.027(2)	0.027(1)	0.004(1)	0.009(1)	0.004(1)
N(1)	4e	0.3459(1)	0.6927(3)	0.3692(1)	0.015(1)	0.028(1)	0.026(1)	0.0026(9)	0.0043(9)	-0.0035(9)
C(15)	4e	0.2728(1)	0.7250(3)	0.3538(1)	0.015(1)	0.025(1)	0.017(1)	0.003(1)	0.0035(9)	0.002(1)
C(16)	4e	0.2458(1)	0.8785(3)	0.3461(1)	0.020(1)	0.023(1)	0.028(1)	-0.004(1)	0.006(1)	0.001(1)
C(17)	4e	0.1736(1)	0.9022(3)	0.3243(1)	0.019(1)	0.016(1)	0.023(1)	0.003(1)	0.007(1)	0.004(1)
C(18)	4e	0.1274(1)	0.7736(3)	0.3081(1)	0.012(1)	0.022(1)	0.011(1)	0.003(1)	0.0029(9)	0.0011(9)
C(19)	4e	0.1539(1)	0.6203(3)	0.3157(1)	0.019(1)	0.018(1)	0.015(1)	-0.003(1)	0.0046(9)	-0.0010(9)
C(20)	4e	0.2262(1)	0.5962(3)	0.3390(1)	0.021(1)	0.018(1)	0.014(1)	0.006(1)	0.002(1)	0.0005(9)
N(2)	4e	0.0525(1)	0.8008(2)	0.2791(1)	0.018(1)	0.016(1)	0.0153(9)	0.0030(9)	0.0032(8)	0.0037(8)
S(1)	4e	0.00160(3)	0.74232(7)	0.33389(3)	0.0147(3)	0.0167(3)	0.0148(3)	-0.0008(2)	0.0029(2)	-0.0010(2)
O(3)	4e	-0.06157(8)	0.8342(2)	0.30253(9)	0.0139(8)	0.027(1)	0.0211(8)	0.0027(7)	0.0023(6)	0.0020(7)
O(4)	4e	-0.00486(8)	0.5724(2)	0.32796(9)	0.0237(9)	0.0170(9)	0.0201(7)	-0.0048(7)	0.0046(7)	-0.0033(7)
C(21)	4e	0.0502(1)	0.7843(3)	0.4422(1)	0.012(1)	0.019(1)	0.018(1)	0.002(1)	0.0049(9)	-0.0010(9)
C(22)	4e	0.0685(1)	0.6585(3)	0.4979(1)	0.018(1)	0.013(1)	0.022(1)	0.000(1)	0.008(1)	-0.001(1)
C(23)	4e	0.1142(1)	0.6820(3)	0.5824(1)	0.023(1)	0.018(1)	0.015(1)	0.003(1)	0.006(1)	0.0042(9)
C(24)	4e	0.1440(1)	0.8266(3)	0.6062(1)	0.022(1)	0.022(1)	0.013(1)	0.001(1)	0.0052(9)	-0.0004(9)
C(25)	4e	0.1276(1)	0.9585(3)	0.5499(1)	0.014(1)	0.016(1)	0.016(1)	0.003(1)	0.0048(9)	0.0001(9)
C(26)	4e	0.1600(1)	1.1099(3)	0.5756(1)	0.018(1)	0.020(1)	0.015(1)	0.001(1)	0.0056(9)	-0.0014(9)
C(27)	4e	0.1367(1)	1.2389(3)	0.5227(1)	0.025(1)	0.016(1)	0.021(1)	-0.000(1)	0.008(1)	-0.002(1)
C(28)	4e	0.0828(1)	1.2229(3)	0.4433(1)	0.025(1)	0.017(1)	0.020(1)	0.004(1)	0.007(1)	0.001(1)
C(29)	4e	0.0530(1)	1.0784(3)	0.4152(1)	0.019(1)	0.018(1)	0.016(1)	0.003(1)	0.003(1)	0.0009(9)
C(30)	4e	0.0758(1)	0.9419(3)	0.4670(1)	0.015(1)	0.016(1)	0.016(1)	0.0025(9)	0.0070(9)	-0.0012(9)
N(3)	4e	0.2152(1)	1.1176(2)	0.6561(1)	0.020(1)	0.020(1)	0.0195(9)	-0.0030(9)	-0.0003(8)	-0.0012(8)
C(31)	4e	0.2234(1)	1.2692(3)	0.7009(1)	0.031(2)	0.024(1)	0.021(1)	-0.009(1)	0.002(1)	-0.002(1)
C(32)	4e	0.2828(1)	1.0578(3)	0.6511(2)	0.023(1)	0.033(2)	0.037(1)	0.001(1)	-0.001(1)	0.005(1)

Acknowledgment. The authors are grateful to Dr. L. J. W. Shimon for her contribution to the structure analysis.

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