Crystal structure of *N*-(4-(3,6-dioxo-4-phenylsulfanylcyclo-1,4-dienylamino))-5-dimethylaminonaphtalene-sulfonamide, $C_{30}H_{25}N_3O_4S_2$

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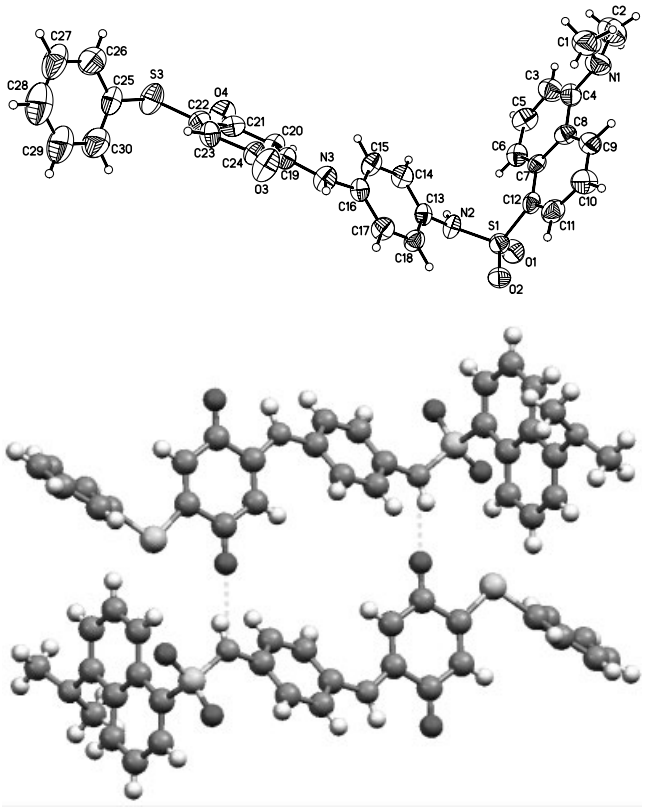

Abstract

$C_{30}H_{25}N_3O_4S_2$, monoclinic, $P12_1/c1$ (No. 14), a = 7.031(2) Å, b = 33.12(1) Å, c = 13.149(4) Å, β = 119.63(1)°, V = 2661.9 Å^3, Z = 4, $R_{gt}(F)$ = 0.058, $wR_{ref}(F^2)$ = 0.174, T = 293 K.

Source of material

The title compound was prepared in accordance with a literature procedure [1]. The crystals were grown by vapor diffusion of petroleum ether into commercial grade ethanol, to produce thin black plates.

Discussion

This organic fluorescence quenching molecule [2] possesses several distinct units in this highly functional molecule: a *S*-phenyl ring, a quinone ring, a *p*-phenylene diamine ring and a 5-dimethylamino-naphthalene ring. These units do not lie in the same molecular plane, as can clearly be seen in the ORTEP diagram in upper figure. The *S*-phenyl ring has a C25–S3–C22 angle of 103.8° with the quinone ring. The C19–N3–C16 angle between the *p*-phenylene diamine and quinone ring is 128.2°. The torsion angle C13–N2–S1–C12 between the *p*-phenylene diamine and 5-dimethylamino-naphthalene rings is –64.9°. The hydrogen bonds form between the NH bonded to the sulfonyl group and the quinonic oxygen on the same side of the molecule, in the second molecule. The N–H···O bond length is 1.97 Å. There are two intermolecular hydrogen bonds from each molecule in the asymmetric unit. These bonds occur between anti-parallel pairs of molecules and form centrosymmetric $R_2^2(22)$ hydrogen bond rings (lower figure). The two quinone rings of the pairs of molecules are related by an inversion center and as such are parallel to one another. The distance between the two quinone rings, as measured between corresponding pairs of atoms, is 3.91(3) Å.

Table 1. Data collection and handling.

Crystal:	black plate, size 0.18 × 0.32 × 0.49 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.42 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	41.62°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7773, 2768
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1554
$N(param)_{refined}$:	377
Programs:	SHELXS-97 [3], SHELXL-97 [4], SHELXTL-plus [5]

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

Atom	Site	x	y	z	U_{iso}
H(2X)	4*e*	0.7031	0.0803	0.6627	0.09
H(3X)	4*e*	0.1918	0.0055	0.1252	0.06
H(1A)	4*e*	0.7416	0.2673	0.3416	0.08
H(1B)	4*e*	0.5681	0.3002	0.3247	0.09
H(1C)	4*e*	0.5181	0.2544	0.3311	0.08
H(2A)	4*e*	0.9705	0.3097	0.6133	0.09
H(2B)	4*e*	0.8582	0.3318	0.4921	0.06
H(2C)	4*e*	1.0203	0.2960	0.5147	0.08
H(4)	4*e*	1.0855	0.2438	0.6447	0.12

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Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(5)	4*e*	1.1306	0.1850	0.7452	0.02
H(6)	4*e*	0.8418	0.1447	0.7122	0.02
H(9)	4*e*	0.3449	0.2591	0.4263	0.04
H(10)	4*e*	0.0422	0.2218	0.3887	0.05
H(11)	4*e*	0.0853	0.1581	0.4723	0.08
H(14)	4*e*	0.7844	0.0739	0.5085	0.03
H(15)	4*e*	0.7039	0.0437	0.3360	0.05
H(17)	4*e*	0.1052	0.0183	0.2781	0.04

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(18)	4*e*	0.1840	0.0483	0.4514	0.05
H(20)	4*e*	0.7569	–0.0156	0.3050	0.07
H(23)	4*e*	0.3910	–0.0741	–0.0729	0.03
H(26)	4*e*	0.8204	–0.1042	–0.1702	0.04
H(27)	4*e*	0.6127	–0.1402	–0.3428	0.10
H(28)	4*e*	0.3295	–0.1808	–0.3648	0.15
H(29)	4*e*	0.2664	–0.1893	–0.2110	0.27
H(30)	4*e*	0.4779	–0.1568	–0.0365	0.11

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)	4*e*	0.4180(3)	0.11368(5)	0.6401(2)	0.052(1)	0.045(1)	0.042(1)	0.001(1)	0.026(1)	0.000(1)
S(3)	4*e*	0.8488(3)	–0.10325(7)	0.0502(2)	0.055(2)	0.083(2)	0.057(1)	0.019(1)	0.021(1)	–0.009(1)
O(1)	4*e*	0.5435(8)	0.1203(1)	0.7643(4)	0.081(4)	0.059(3)	0.033(3)	–0.005(3)	0.028(3)	–0.006(3)
O(2)	4*e*	0.1965(7)	0.0997(1)	0.5888(4)	0.041(3)	0.051(3)	0.068(3)	–0.007(2)	0.028(3)	–0.005(3)
O(3)	4*e*	0.1554(8)	–0.0256(2)	–0.0309(4)	0.040(4)	0.093(4)	0.037(3)	0.012(3)	0.006(3)	–0.015(3)
O(4)	4*e*	0.9922(8)	–0.0611(2)	0.2545(4)	0.035(3)	0.082(4)	0.052(3)	0.011(3)	0.007(3)	–0.001(3)
N(1)	4*e*	0.726(1)	0.2778(2)	0.4867(6)	0.072(5)	0.057(5)	0.067(5)	–0.019(4)	0.038(4)	–0.007(4)
N(2)	4*e*	0.557(1)	0.0804(2)	0.6130(5)	0.035(4)	0.049(4)	0.037(4)	0.008(3)	0.013(3)	–0.009(3)
N(3)	4*e*	0.340(1)	0.0064(2)	0.1826(5)	0.039(4)	0.063(4)	0.030(4)	–0.001(3)	0.009(3)	–0.010(3)
C(1)	4*e*	0.630(2)	0.2747(3)	0.3602(8)	0.082(7)	0.079(8)	0.102(9)	0.016(7)	0.056(7)	0.032(6)
C(2)	4*e*	0.910(2)	0.3063(3)	0.531(1)	0.099(9)	0.071(8)	0.13(1)	–0.015(7)	0.058(8)	–0.001(7)
C(3)	4*e*	0.762(1)	0.2421(2)	0.5514(7)	0.070(7)	0.048(5)	0.037(5)	0.010(5)	0.028(5)	0.009(4)
C(4)	4*e*	0.963(2)	0.2287(3)	0.6307(7)	0.068(7)	0.061(6)	0.063(6)	–0.012(6)	0.032(6)	–0.003(5)
C(5)	4*e*	0.990(1)	0.1929(3)	0.6904(6)	0.030(6)	0.081(7)	0.046(5)	–0.007(5)	0.008(4)	–0.007(5)
C(6)	4*e*	0.818(1)	0.1691(2)	0.6727(6)	0.053(6)	0.052(6)	0.040(5)	0.003(5)	0.019(4)	–0.003(4)
C(7)	4*e*	0.604(1)	0.1819(2)	0.5924(5)	0.039(5)	0.044(5)	0.028(4)	0.003(4)	0.019(4)	–0.001(4)
C(8)	4*e*	0.570(1)	0.2188(2)	0.5325(6)	0.036(5)	0.051(5)	0.041(5)	–0.005(4)	0.018(4)	–0.004(4)
C(9)	4*e*	0.362(1)	0.2337(2)	0.4601(6)	0.060(7)	0.047(6)	0.067(6)	0.006(5)	0.038(5)	0.012(5)
C(10)	4*e*	0.181(1)	0.2116(2)	0.4383(7)	0.043(6)	0.068(6)	0.060(5)	0.015(5)	0.019(5)	0.025(5)
C(11)	4*e*	0.208(1)	0.1739(2)	0.4910(6)	0.045(6)	0.062(6)	0.060(6)	0.009(5)	0.027(5)	0.005(5)
C(12)	4*e*	0.409(1)	0.1597(2)	0.5683(6)	0.050(5)	0.040(5)	0.037(4)	0.015(4)	0.024(4)	0.008(4)
C(13)	4*e*	0.495(1)	0.0637(2)	0.5000(6)	0.040(5)	0.034(4)	0.030(4)	0.008(4)	0.013(4)	0.001(4)
C(14)	4*e*	0.647(1)	0.0624(2)	0.4626(6)	0.028(5)	0.055(5)	0.037(5)	–0.005(4)	0.006(4)	–0.009(4)
C(15)	4*e*	0.599(1)	0.0442(2)	0.3596(6)	0.050(5)	0.057(5)	0.041(5)	0.005(4)	0.025(4)	–0.007(4)
C(16)	4*e*	0.398(1)	0.0265(2)	0.2898(6)	0.039(5)	0.037(4)	0.031(4)	0.003(4)	0.010(4)	–0.003(4)
C(17)	4*e*	0.244(1)	0.0292(2)	0.3254(6)	0.041(5)	0.057(5)	0.046(5)	–0.003(4)	0.019(4)	–0.006(4)
C(18)	4*e*	0.290(1)	0.0471(2)	0.4289(6)	0.037(5)	0.050(5)	0.049(5)	–0.003(4)	0.022(4)	–0.010(4)
C(19)	4*e*	0.469(1)	–0.0169(2)	0.1563(6)	0.040(5)	0.041(4)	0.038(5)	0.005(4)	0.019(4)	0.000(4)
C(20)	4*e*	0.684(1)	–0.0263(2)	0.2298(6)	0.048(5)	0.050(5)	0.035(5)	0.010(4)	0.019(4)	0.006(4)
C(21)	4*e*	0.797(1)	–0.0525(2)	0.1918(6)	0.039(5)	0.058(5)	0.038(5)	0.002(4)	0.016(4)	0.008(4)
C(22)	4*e*	0.678(1)	–0.0716(2)	0.0733(6)	0.037(5)	0.049(5)	0.046(5)	0.004(4)	0.022(4)	0.001(4)
C(23)	4*e*	0.464(1)	–0.0623(2)	0.0008(6)	0.045(5)	0.045(5)	0.034(5)	–0.001(4)	0.011(4)	–0.007(4)
C(24)	4*e*	0.348(1)	–0.0347(2)	0.0358(6)	0.045(5)	0.050(5)	0.032(5)	0.001(4)	0.018(4)	–0.005(4)
C(25)	4*e*	0.672(1)	–0.1265(2)	–0.0862(6)	0.061(6)	0.057(6)	0.045(5)	0.015(4)	0.026(5)	–0.005(4)
C(26)	4*e*	0.710(2)	–0.1216(3)	–0.1776(8)	0.096(8)	0.064(6)	0.071(7)	0.027(6)	0.052(6)	0.024(6)
C(27)	4*e*	0.583(2)	–0.1428(3)	–0.2815(9)	0.14(1)	0.078(8)	0.053(7)	0.040(7)	0.058(8)	0.012(6)
C(28)	4*e*	0.416(2)	–0.1675(3)	–0.294(1)	0.11(1)	0.099(9)	0.071(9)	0.018(7)	0.031(8)	–0.020(7)
C(29)	4*e*	0.379(2)	–0.1724(3)	–0.202(1)	0.13(1)	0.104(8)	0.083(9)	–0.010(7)	0.062(8)	–0.037(7)
C(30)	4*e*	0.504(2)	–0.1527(3)	–0.0985(9)	0.094(8)	0.092(8)	0.069(7)	–0.010(6)	0.048(6)	–0.018(6)

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