Crystal structure of 2-dimethylamino-1-methyl-1*H*-quino[4,3-*e*]-1,2,4-thiadiazine 4,4-dioxide, $C_{13}H_{14}N_4O_2S$

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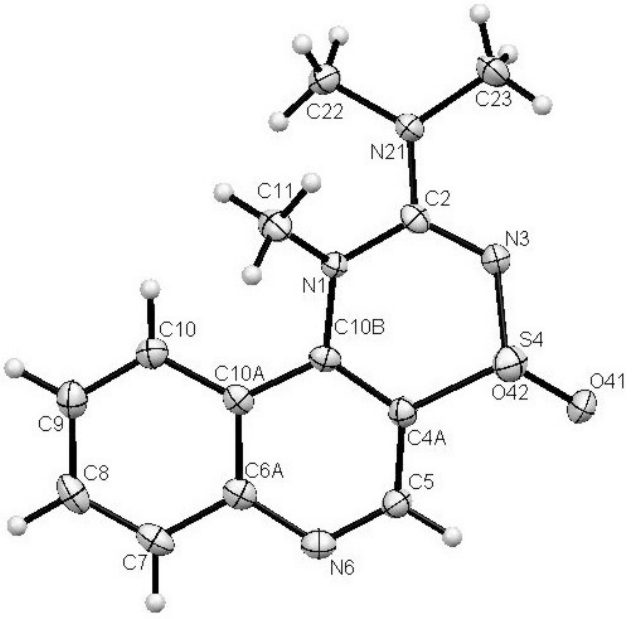

Abstract

$C_{13}H_{14}N_4O_2S$, monoclinic, $P12_1/c1$ (no. 14), a = 7.6239(1) Å, b = 20.4013(2) Å, c = 8.4919(1) Å, β = 101.908(1)°, V = 1292.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.026, $wR_{ref}(F^2)$ = 0.074, T = 100 K.

Source of material

The title compound was prepared in the reaction of 4-chloro-3-quinolinesulfonyl chloride with *N*,*N*,*N*′-trimethylguanidine sulfate [1]. Crystals of 2-dimethylamino-1-methyl-1*H*-quino[4,3-*e*]-1,2,4-thiadiazine 4,4-dioxide were obtained by slow evaporation of acetone solution at room temperature.

Experimental details

The aromatic hydrogen atoms were treated as riding on their parent carbon atoms with d(C—H) = 0.95 Å and U_{iso}(H) = 1.2 U_{eq}(C). Methyl H atoms were constrained as riding atoms, fixed to the parent atoms with a distance of 0.98 Å and U_{iso}(H) = 1.5 U_{eq}(C) and torsion angle were refined. Hydrogen atoms involved in hydrogen bonding were refined freely with isotropic atomic displacement parameters.

Discussion

Significant biological activity of benzo- and pyrido-1,2,4-thiadiazine *S*,*S*-dioxides as potassium channel openers [2-7] has turned the medicinal chemists' attention to other areno- and heteroareno-fused 1,2,4-thiadiazine *S*,*S*-dioxides as possible candidates for therapeutic application. The pharmacological activity of this class of compounds may be affected by the shape of the molecule.

The thiadiazine ring of 2-dimethylamino-1-methyl-1*H*-quino[4,3-*e*]-1,2,4-thiadiazine 4,4-dioxide in crystalline state adopts the boat conformation as could be deduced from the Cremer & Pople [8] parameters [Q = 0.530(1) Å, θ = 74.46° and φ = 4.12°]. The deviation from the thiadiazine ring mean plane is largest for the S4 (–0.3280(3) Å) and N1 (–0.26(1) Å) atoms. The values of the bond lengths d(N1—C2) = 1.394(2) Å and d(N3—C2) = 1.326(2) Å ranging between the typical data for single (1.47 Å) and double (1.27 Å) C—N bond lengths [9] as well as the short bond length d(C2—N21) = 1.337(2) Å indicate the diffuse electronic nature of the guanidine part of thiadiazine ring. These results are close to those for 3-isopropylamino-4*H*-pyrido[4,3-*e*]-1,2,4-thiadiazine 1,1-dioxide [2], 4-methyl-3-methylamino-4*H*-pyrido[4,3-*e*]-1,2,4-thiadiazine 1,1-dioxide, 3-dimethylamino-4-methyl-4*H*-pyrido[4,3-*e*]-1,2,4-thiadiazine 1,1-dioxide [10] and 3-amino-4*H*-pyrido[4,3-*e*]-1,2,4-thiadiazine 1,1-dioxide [11]. The three-dimensional arrangement of 2-dimethylamino-1-methyl-1*H*-quino[4,3-*e*]-1,2,4-thiadiazine 4,4-dioxide in the crystalline state is achieved by a non-covalent π–π stacking interaction between high electron density benzene and low electron density pyridine ring systems (symmetry codes: x,½–y,–½+z and x,½–y,½+z). The two aromatic moieties are in *face-to-face* alignment. The molecules are approximately parallel, separated by interplanar distance of 3.600(1) Å. The dihedral angle between pyridine and benzene mean planes equals 2.23(7)°. The displacement measured by the angle formed between the ring-centroid vector and the vector normal to the benzene ring mean plane is 4.10(5)°.

Table 1. Data collection and handling.

Crystal:	colorless plate, size 0.18 × 0.28 × 0.48 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.58 cm^{-1}
Diffractometer, scan mode:	Xcalibur with Sapphire3 detector, ω
$2\theta_{max}$:	50.1°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11752, 2283
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2143
$N(param)_{refined}$:	203
Programs:	SHELXS-97, SHELXL-97 [12], PLATON [13]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(5)	4*e*	0.3270	0.2408	0.7511	0.017
H(7)	4*e*	0.7209	0.1119	0.5833	0.019
H(8)	4*e*	0.9792	0.1250	0.4844	0.021
H(9)	4*e*	1.100(2)	0.2297(9)	0.465(2)	0.021
H(10)	4*e*	0.9530	0.3221	0.5317	0.018
H(11A)	4*e*	0.6929	0.3883	0.3809	0.025
H(11B)	4*e*	0.8726	0.4178	0.4890	0.025
H(11C)	4*e*	0.6956	0.4620	0.4463	0.025
H(22A)	4*e*	0.991(2)	0.5149(9)	0.845(2)	0.025
H(22B)	4*e*	0.945(2)	0.5208(9)	0.660(2)	0.025
H(22C)	4*e*	0.971(2)	0.451(1)	0.739(2)	0.025
H(23A)	4*e*	0.715(3)	0.589(1)	0.782(2)	0.030
H(23B)	4*e*	0.723(3)	0.552(1)	0.944(3)	0.030
H(23C)	4*e*	0.546(3)	0.5477(9)	0.815(2)	0.030

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(4)	4*e*	0.34290(4)	0.38049(2)	0.70703(4)	0.0108(2)	0.0114(2)	0.0164(2)	0.0006(1)	0.0051(1)	–0.0001(1)
O(41)	4*e*	0.2350(1)	0.36395(5)	0.8214(1)	0.0171(5)	0.0174(5)	0.0242(6)	–0.0014(4)	0.0119(4)	–0.0011(4)
O(42)	4*e*	0.2466(1)	0.40164(5)	0.5505(1)	0.0151(5)	0.0174(5)	0.0210(6)	0.0021(4)	0.0020(4)	0.0023(4)
N(1)	4*e*	0.6855(2)	0.39366(6)	0.6184(1)	0.0135(6)	0.0092(6)	0.0142(6)	–0.0004(4)	0.0058(5)	–0.0004(5)
N(3)	4*e*	0.4900(2)	0.43366(6)	0.7859(2)	0.0142(6)	0.0129(6)	0.0172(6)	–0.0003(5)	0.0059(5)	–0.0019(5)
N(6)	4*e*	0.5209(2)	0.19762(6)	0.6732(2)	0.0168(6)	0.0131(6)	0.0161(6)	–0.0011(5)	0.0030(5)	0.0012(5)
N(21)	4*e*	0.7369(2)	0.49266(6)	0.7560(1)	0.0135(6)	0.0101(6)	0.0186(6)	0.0001(5)	0.0049(5)	–0.0020(5)
C(2)	4*e*	0.6329(2)	0.43974(7)	0.7206(2)	0.0141(7)	0.0102(7)	0.0120(7)	0.0025(5)	0.0015(5)	0.0015(5)
C(4A)	4*e*	0.4771(2)	0.31371(7)	0.6805(2)	0.0117(7)	0.0132(7)	0.0121(7)	0.0007(5)	0.0021(5)	–0.0008(5)
C(5)	4*e*	0.4299(2)	0.24840(7)	0.7068(2)	0.0126(7)	0.0142(7)	0.0156(7)	–0.0016(5)	0.0035(5)	0.0005(6)
C(6A)	4*e*	0.6703(2)	0.20997(7)	0.6113(2)	0.0142(7)	0.0134(7)	0.0108(6)	0.0005(5)	–0.0005(5)	0.0002(5)
C(7)	4*e*	0.7651(2)	0.15474(7)	0.5709(2)	0.0219(7)	0.0106(7)	0.0147(7)	0.0011(6)	0.0010(6)	–0.0005(6)
C(8)	4*e*	0.9192(2)	0.16237(7)	0.5143(2)	0.0206(8)	0.0154(7)	0.0155(7)	0.0069(6)	0.0020(6)	–0.0022(6)
C(9)	4*e*	0.9893(2)	0.22553(8)	0.5002(2)	0.0143(7)	0.0206(8)	0.0170(7)	0.0026(6)	0.0036(6)	–0.0015(6)
C(10)	4*e*	0.9024(2)	0.27995(7)	0.5392(2)	0.0142(7)	0.0143(7)	0.0166(7)	–0.0008(6)	0.0030(6)	–0.0011(6)
C(10A)	4*e*	0.7371(2)	0.27408(7)	0.5909(2)	0.0128(7)	0.0123(7)	0.0101(7)	0.0011(5)	0.0002(5)	–0.0008(5)
C(10B)	4*e*	0.6328(2)	0.32777(7)	0.6283(2)	0.0124(7)	0.0110(7)	0.0100(6)	–0.0007(5)	0.0000(5)	–0.0005(5)
C(11)	4*e*	0.7414(2)	0.41746(7)	0.4712(2)	0.0246(8)	0.0134(7)	0.0147(7)	–0.0005(6)	0.0090(6)	0.0015(6)
C(22)	4*e*	0.9264(2)	0.49449(8)	0.7473(2)	0.0136(7)	0.0145(7)	0.0216(8)	–0.0017(6)	0.0044(6)	–0.0002(6)
C(23)	4*e*	0.6727(2)	0.55004(8)	0.8313(2)	0.0193(8)	0.0144(8)	0.0265(9)	0.0000(6)	0.0055(7)	–0.0079(7)

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