Crystal structure of *N*1-(4,4-dimethoxyhexahydro-1 λ ⁶-thiopyran-1-yliden)-4-methyl-1-benzenesulfonamide, C₁₄H₂₁NO₄S₂

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Received March 17, 2001, CCDC-No. 1267/639

Abstract

C₁₄H₂₁NO₄S₂, monoclinic, C12/c1 (No. 15), *a* = 34.05(1) Å, *b* = 7.492(4) Å, *c* = 12.870(8) Å, β = 107.24(3)°, *V* = 3135.7 Å³, *Z* = 8, *R*_{gt}(*F*) = 0.046, *wR*_{ref}(*F*) = 0.057, *T* = 293 K.

Source of material

Prepared by treatment of 4,4-dimethoxytetrahydro-2*H*-thiopyran with chloramine T in 82 % yield [1]. Colourless crystals were obtained from the slow evaporation of an ethanol solution of the compound; mp. 417 K – 419 K.

Discussion

The key feature of the structure is the axial disposition of the imine group at the ring sulfur atom. Key parameters are distances: *d*(S1–N1) 1.639(3) Å, *d*(S2–N1) 1.609(3) Å and angle S1–N1–S2 of 111.5(1)°.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.26 × 0.32 × 0.32 mm
Wavelength:	Mo <i>K</i> _α radiation (0.7107 Å)
μ :	3.54 cm ⁻¹
Diffraction, scan mode:	Rigaku AFC6R, $\omega/2\theta$
2 θ _{max} :	55.12°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	3973, 3908
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 3 σ (<i>I</i> _{obs}), 2551
<i>N</i> (<i>param</i>) _{refined} :	190
Programs:	SIR97 [2], teXsan [3], ORTEPII [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(1)	8 <i>f</i>	0.3699	−0.1558	−0.3703	0.039
H(2)	8 <i>f</i>	0.3636	−0.0912	−0.2617	0.039
H(3)	8 <i>f</i>	0.3006	−0.1550	−0.3841	0.038
H(4)	8 <i>f</i>	0.3030	0.0472	−0.3568	0.038
H(5)	8 <i>f</i>	0.3132	0.2029	−0.6064	0.039
H(6)	8 <i>f</i>	0.3106	0.2681	−0.4942	0.039
H(7)	8 <i>f</i>	0.3772	0.2970	−0.5023	0.040
H(8)	8 <i>f</i>	0.3786	0.0941	−0.5251	0.040
H(9)	8 <i>f</i>	0.4770	0.4006	−0.2078	0.043
H(10)	8 <i>f</i>	0.5314	0.2863	−0.0686	0.048
H(11)	8 <i>f</i>	0.5274	0.1667	0.1944	0.061
H(12)	8 <i>f</i>	0.5379	0.0337	0.1140	0.061
H(13)	8 <i>f</i>	0.5580	0.2214	0.1316	0.061
H(14)	8 <i>f</i>	0.4546	0.1996	0.1124	0.047
H(15)	8 <i>f</i>	0.4002	0.3153	−0.0254	0.042
H(16)	8 <i>f</i>	0.2420	0.1650	−0.6721	0.065
H(17)	8 <i>f</i>	0.2136	−0.0005	−0.6845	0.065
H(18)	8 <i>f</i>	0.2562	−0.0181	−0.7043	0.065
H(19)	8 <i>f</i>	0.2816	−0.3165	−0.5609	0.054
H(20)	8 <i>f</i>	0.3118	−0.3732	−0.6245	0.054
H(21)	8 <i>f</i>	0.3269	−0.3607	−0.4985	0.054

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
S(1)	8 <i>f</i>	0.38695(1)	0.14013(8)	−0.34394(4)	0.0281(3)	0.0306(3)	0.0282(3)	0.0002(2)	0.0065(2)	−0.0017(2)
S(2)	8 <i>f</i>	0.39166(1)	0.45099(7)	−0.23706(4)	0.0310(3)	0.0279(3)	0.0242(2)	−0.0014(2)	0.0086(2)	−0.0002(2)
O(2)	8 <i>f</i>	0.36712(4)	0.5657(2)	−0.1910(1)	0.0405(9)	0.0320(9)	0.0346(8)	0.0057(7)	0.0123(7)	−0.0025(7)
O(3)	8 <i>f</i>	0.40936(5)	0.5311(2)	−0.3159(1)	0.0440(9)	0.039(1)	0.0301(8)	−0.0091(8)	0.0137(7)	0.0048(7)

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Table 3. Continued.

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(4)	8f	0.25961(4)	-0.0024(2)	-0.5495(1)	0.0241(7)	0.055(1)	0.0386(9)	0.0022(7)	0.0079(6)	-0.0005(8)
O(5)	8f	0.32019(4)	-0.1260(2)	-0.5705(1)	0.0304(8)	0.0355(9)	0.0370(8)	-0.0009(7)	0.0120(6)	-0.0089(7)
N(1)	8f	0.36308(5)	0.2821(2)	-0.2868(1)	0.0260(9)	0.034(1)	0.0294(9)	-0.0035(8)	0.0084(7)	-0.0051(8)
C(2)	8f	0.35937(7)	-0.0624(3)	-0.3362(2)	0.041(1)	0.024(1)	0.029(1)	0.001(1)	0.0077(9)	0.0050(9)
C(3)	8f	0.31346(6)	-0.0452(3)	-0.3916(2)	0.035(1)	0.031(1)	0.032(1)	-0.003(1)	0.0135(9)	0.000(1)
C(4)	8f	0.30323(6)	-0.0010(3)	-0.5125(2)	0.025(1)	0.035(1)	0.031(1)	0.0015(9)	0.0070(8)	-0.0014(9)
C(5)	8f	0.32123(7)	0.1785(3)	-0.5305(2)	0.037(1)	0.037(1)	0.024(1)	0.002(1)	0.0081(9)	0.0038(9)
C(6)	8f	0.36795(6)	0.1826(3)	-0.4879(2)	0.037(1)	0.038(1)	0.027(1)	-0.005(1)	0.0130(9)	-0.000(1)
C(7)	8f	0.43345(6)	0.3689(3)	-0.1297(2)	0.026(1)	0.029(1)	0.025(1)	-0.0035(9)	0.0049(8)	-0.0028(9)
C(8)	8f	0.47247(6)	0.3598(3)	-0.1424(2)	0.034(1)	0.044(1)	0.033(1)	-0.004(1)	0.0150(9)	-0.002(1)
C(9)	8f	0.50470(7)	0.2911(3)	-0.0597(2)	0.029(1)	0.044(1)	0.048(1)	-0.001(1)	0.012(1)	-0.008(1)
C(10)	8f	0.49871(7)	0.2286(3)	0.0366(2)	0.034(1)	0.034(1)	0.036(1)	0.001(1)	0.0012(9)	-0.011(1)
C(10')	8f	0.53373(7)	0.1559(4)	0.1275(2)	0.044(1)	0.049(2)	0.047(1)	0.006(1)	-0.006(1)	-0.010(1)
C(11)	8f	0.45933(7)	0.2402(3)	0.0471(2)	0.041(1)	0.049(1)	0.027(1)	0.003(1)	0.008(1)	0.001(1)
C(12)	8f	0.42689(6)	0.3090(3)	-0.0345(2)	0.031(1)	0.046(1)	0.030(1)	0.001(1)	0.0122(9)	0.004(1)
C(41)	8f	0.24132(7)	0.0395(4)	-0.6621(2)	0.034(1)	0.071(2)	0.047(1)	0.006(1)	-0.005(1)	0.000(1)
C(42)	8f	0.30918(7)	-0.3097(3)	-0.5630(2)	0.040(1)	0.038(1)	0.058(2)	-0.004(1)	0.017(1)	-0.017(1)

Acknowledgment. The Australian Research Council is thanked for support.

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