Crystal structure of *S-p*-nitrophenyl-*S*-phenyl-*S*-fluorothiazyne, $(C_6H_5)NFS(C_6H_4NO_2)$

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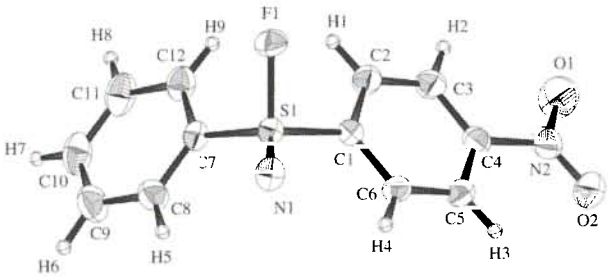

Abstract

$C_{12}H_9FN_2O_2S$, monoclinic, $P12_1/n1$ (No. 14), $a = 8.638(4)$ Å, $b = 14.070(3)$ Å, $c = 10.735(3)$ Å, $\beta = 111.82(2)°$, $V = 1211.2$ Å^3, $Z = 4$, $R_{gt}(F) = 0.042$, $wR_{gt}(F) = 0.039$, $T = 296$ K.

Source of material

The title compound was prepared by reacting *S-p*-nitrophenyl-*S*-phenyl-*N*-bromosulfilimine with tetrabutylammonium fluoride in THF at 273 K [1-5]. Suitable crystals were obtained by slow evaporation of a benzene-hexane mixture.

Experimental details

All non-hydrogen atoms were modelled, the hydrogen atoms were added at calculated positions (d(C—H) = 1.08 Å) and subsequently refined.

Discussion

Thiazynes are compounds with S—N triple bonds and are of interest because their structures and reactivity [6, 7]. In general, because these compounds are difficult to purify and crystallize as suitable single crystals for X-ray analysis, their structure have remained elusive. The ORTEP plot (50% probability ellipsoids) shows the distorted tetrahedral bonding arrangement about the sulfur center and the location of the S—N triple bond. The bond distances S(1)—N(1), S(1)—F(1), S(1)—C(1) and S(1)—C(7) are equal to 1.444(2) Å, 1.638(2) Å, 1.792(2) Å and 1.778(2) Å, respectively.

Table 1. Data collection and handling.

Crystal:	colorless, prismatic, size 0.30 × 0.60 × 0.70 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	2.74 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC7R, ω/2θ
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2891, 2758
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\ \sigma(I_{obs})$, 2071
$N(param)_{refined}$:	199
Programs:	teXsan [8], DIFABS [9], ORTEP [10]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*e*	0.089(2)	0.202(1)	0.642(2)	0.086(6)
H(2)	4*e*	0.094(2)	0.121(1)	0.448(2)	0.078(5)
H(3)	4*e*	0.436(2)	−0.066(1)	0.690(1)	0.075(5)
H(4)	4*e*	0.424(2)	0.014(1)	0.877(1)	0.059(4)
H(5)	4*e*	0.162(2)	0.094(1)	1.114(2)	0.072(5)
H(6)	4*e*	−0.089(2)	0.083(1)	1.140(2)	0.085(6)
H(7)	4*e*	−0.328(2)	0.158(1)	0.979(2)	0.092(6)
H(8)	4*e*	−0.311(2)	0.238(1)	0.794(2)	0.112(7)
H(9)	4*e*	−0.058(2)	0.246(1)	0.765(2)	0.077(5)

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*	0.27513(4)	0.17375(2)	0.92865(3)	0.0385(1)	0.0434(2)	0.0441(2)	−0.0014(1)	0.0149(1)	−0.0034(1)
F(1)	4*e*	0.2626(1)	0.28071(5)	0.86540(9)	0.0709(5)	0.0387(4)	0.0783(6)	−0.0022(4)	0.0329(5)	0.0020(4)
O(1)	4*e*	0.1790(2)	0.0014(1)	0.3266(1)	0.173(2)	0.120(1)	0.0460(7)	0.040(1)	0.0297(8)	−0.0014(7)
O(2)	4*e*	0.3714(2)	−0.09151(9)	0.4526(1)	0.124(1)	0.0683(8)	0.0771(8)	0.0132(7)	0.0526(8)	−0.0109(6)
N(1)	4*e*	0.4177(1)	0.15229(9)	1.0484(1)	0.0400(6)	0.0677(8)	0.0471(6)	0.0016(5)	0.0086(5)	−0.0070(6)
N(2)	4*e*	0.2717(2)	−0.0268(1)	0.4361(1)	0.096(1)	0.0586(8)	0.0533(7)	−0.0068(7)	0.0361(7)	−0.0066(6)

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Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*e*	0.2601(1)	0.11523(9)	0.7763(1)	0.0405(6)	0.0409(6)	0.0396(6)	–0.0005(5)	0.0154(5)	0.0009(5)
C(2)	4*e*	0.1603(2)	0.1476(1)	0.6500(1)	0.0649(9)	0.0616(9)	0.0462(7)	0.0194(8)	0.0201(7)	0.0081(6)
C(3)	4*e*	0.1623(2)	0.0995(1)	0.5383(1)	0.072(1)	0.0620(9)	0.0432(8)	0.0137(8)	0.0175(7)	0.0101(7)
C(4)	4*e*	0.2655(2)	0.0219(1)	0.5563(1)	0.0629(8)	0.0451(7)	0.0459(7)	–0.0061(6)	0.0258(6)	–0.0002(6)
C(5)	4*e*	0.3640(2)	–0.0114(1)	0.6810(1)	0.0599(8)	0.0420(8)	0.0515(7)	0.0035(6)	0.0215(7)	–0.0012(6)
C(6)	4*e*	0.3595(2)	0.0357(1)	0.7922(1)	0.0502(7)	0.0427(7)	0.0424(7)	0.0065(6)	0.0117(6)	0.0044(6)
C(7)	4*e*	0.0736(1)	0.1721(1)	0.9373(1)	0.0409(6)	0.0454(7)	0.0474(7)	–0.0017(6)	0.0180(5)	–0.0080(6)
C(8)	4*e*	0.0652(2)	0.1239(1)	1.0459(2)	0.0567(8)	0.064(1)	0.0619(9)	–0.0007(8)	0.0273(8)	0.0055(7)
C(9)	4*e*	–0.0860(2)	0.1193(1)	1.0625(2)	0.072(1)	0.086(1)	0.084(1)	–0.0087(9)	0.0478(9)	0.002(1)
C(10)	4*e*	–0.2246(2)	0.1609(1)	0.9695(2)	0.0525(8)	0.090(1)	0.100(1)	–0.0108(8)	0.0429(9)	–0.0245(9)
C(11)	4*e*	–0.2143(2)	0.2083(2)	0.8614(2)	0.0445(8)	0.105(2)	0.078(1)	0.0110(9)	0.0172(8)	–0.017(1)
C(12)	4*e*	–0.0646(2)	0.2160(1)	0.8425(2)	0.0473(7)	0.077(1)	0.0539(9)	0.0115(7)	0.0155(7)	–0.0018(8)

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