C(10)	0.571 (1)	0.0999 (4)	0.8924 (3)	0.0651
C(11)	0.884 (1)	0.1618 (4)	0.7123 (3)	0.0540
C(12)	0.548 (1)	0.1673 (4)	1.0241 (3)	0.0629
C(13)	0.879 (1)	0.0753 (4)	0.9941 (3)	0.0656
C(14)	0.839 (1)	0.1431 (5)	1.1203 (3)	0.0736
C(15)	0.885 (1)	0.1744 (5)	0.5705 (3)	0.0702
C(16)	1.056 (2)	0.1004 (5)	0.5955 (4)	0.0799
C(17)	0.967 (1)	0.0719 (4)	0.6738 (3)	0.0714
C(18)	0.965 (1)	0.0898 (5)	1.0687 (4)	0.0743
C(19)	0.633 (1)	0.1811 (4)	1.0985 (3)	0.0719

Table 2. *Selected geometric parameters* (Å, °)

S(1)—Cl(2)	2.046 (2)	C(8)—C(12)	1.368 (7)
S(1)—N(4)	1.581 (4)	C(8)—C(13)	1.378 (8)
S(1)—O(5)	1.409 (4)	C(9)—C(11)	1.514 (8)
S(1)—O(7)	1.415 (4)	C(11)—C(17)	1.529 (8)
O(3)—C(9)	1.318 (6)	C(12)—C(19)	1.383 (8)
O(3)—C(10)	1.462 (6)	C(13)—C(18)	1.389 (8)
N(4)—C(11)	1.497 (6)	C(14)—C(18)	1.373 (9)
N(4)—C(15)	1.488 (6)	C(14)—C(19)	1.35 (1)
O(6)—C(9)	1.206 (7)	C(15)—C(16)	1.51 (1)
C(8)—C(10)	1.489 (7)	C(16)—C(17)	1.498 (9)
N(4)—S(1)—Cl(2)	104.9 (2)	C(11)—C(9)—O(3)	109.6 (5)
O(5)—S(1)—Cl(2)	105.1 (2)	C(11)—C(9)—O(6)	125.3 (5)
O(5)—S(1)—N(4)	109.2 (2)	C(8)—C(10)—O(3)	107.9 (5)
O(7)—S(1)—Cl(2)	105.1 (2)	C(9)—C(11)—N(4)	110.4 (4)
O(7)—S(1)—N(4)	109.6 (2)	C(17)—C(11)—N(4)	102.7 (4)
O(7)—S(1)—O(5)	121.6 (3)	C(17)—C(11)—C(9)	111.7 (5)
C(10)—O(3)—C(9)	116.7 (5)	C(19)—C(12)—C(8)	121.0 (6)
C(11)—N(4)—S(1)	118.0 (3)	C(18)—C(13)—C(8)	120.3 (5)
C(15)—N(4)—S(1)	120.1 (4)	C(19)—C(14)—C(18)	119.9 (6)
C(15)—N(4)—C(11)	110.1 (4)	C(16)—C(15)—N(4)	104.3 (4)
C(12)—C(8)—C(10)	119.6 (5)	C(17)—C(16)—C(15)	103.3 (6)
C(13)—C(8)—C(10)	121.9 (5)	C(16)—C(17)—C(11)	105.3 (5)
C(13)—C(8)—C(12)	118.6 (5)	C(14)—C(18)—C(13)	119.9 (6)
O(6)—C(9)—O(3)	125.0 (5)	C(14)—C(19)—C(12)	120.3 (6)

Analysis of the intensity data revealed that the systematic absences were consistent with the space group $P2_12_12_1$. Corrections were applied for Lorentz and polarization effects. The structure was solved using the direct methods program *SIR*88 (Burla, Camalli, Cascarano, Giacovazzo, Polidori, Spagna & Viterbo, 1989), which revealed the positions of all the non-H atoms. All other computations were performed using the *CRYSTALS* program (Watkin, Carruthers & Betteridge, 1985). The H atoms were subsequently placed geometrically during the refinement process. Full-matrix least-squares refinement was used for the positions and isotropic temperature factors of all the non H-atoms; a parameter to allow for the effects of secondary extinction was included.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: HE1006). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,6-Bis(*p*-nitrophenylthiomethyl)pyridine

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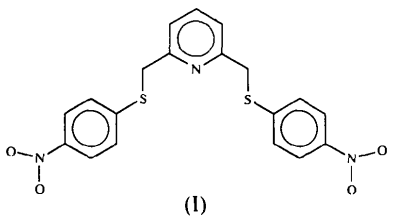
Abstract

In the title compound, $C_{19}H_{15}N_3O_4S_2$, the pyridine ring, one thiomethyl group and the adjoining nitrobenzene group are approximately in the same plane. The C—C—S—C torsion angle is −179.9 (3)°. The C atom of the second thiomethyl group is also in this plane, but the S(2) atom is *anti* to the plane resulting in a C—C—S—C torsion angle of 67.7 (4)°.

Comment

As a part of our study on metal complexes of *S,S'*-alkyl and *S,S'*-aryl substituted 2,6-bis(thiomethyl)pyridine derivatives (Teixidor, Sánchez-Castelló, Lucena, Escriche, Kivekäs, Sundberg & Casabó, 1991), we now report the crystal structure of 2,6-bis(*p*-nitrophenylthiomethyl)pyridine, (I). This type of ligand is a source of an NS_2-coordinating

moiety. A drawing of the molecule is shown in Fig. 1.



(I)

The solid-state conformation of the title ligand is not very suitable for tridentate coordination as a result of the axial orientation of the S(2) atom with respect to the pyridine ring. The nitro group N(2)O(1)O(2) is only slightly (3.5°) twisted with respect to the neighbouring benzene group, but the corresponding angle for the other nitro group is 9.1°.

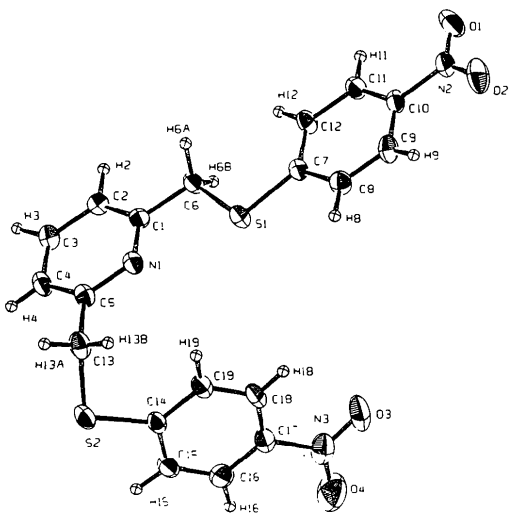


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the title compound. Displacement ellipsoids are shown at 30% probability levels, except for H atoms which are drawn with isotropic temperature factors of 1.0 Å^2.

Experimental

NaOH 98% (1.80 g, 45.0 mmol) in ethanol (200 cm^3) was stirred under reflux for 30 min with *p*-nitrothiophenol (7.00 g, 45.0 mmol). This solution was added to a solution of 2,6-bis(bromomethyl)pyridine (5.38 g, 20.0 mmol) in ethanol (100 cm^3) and was stirred for 1 h at 273 K. A deep yellow precipitate appeared which was filtered off and washed twice with water (30 cm^3). The resulting residue was dissolved in thf (100 cm^3), dried (Na_2SO_4) and evaporated under vacuum to obtain an orange solid. Yield: 56% (4.86 g). Analysis: calculated for $C_{19}H_{15}N_3O_4S_2$, C 55.2, H 3.6, N 10.2, S 15.5%; found, C 55.3, H 3.8, N 9.9, S 15.3%. ^{1}H NMR ($CDCl_3$): δ 4.52 (*s*, 4, py—CH_2—S), δ 7.77 (*m*, 11, aromatic). Slow evaporation of a methanolic solution of this compound afforded slightly yellow crystals suitable for X-ray diffraction analaysis. Dehydrated and deoxygenated ethanol was used in the synthesis. 2,6-Bis(bromomethyl)pyridine was prepared according to a reported procedure (Offerman & Vögtle, 1977). *p*-Nitrothiophenol is commercially available (Aldrich) and was used as received. Microanalyses were performed on a Perkin–Elmer 240-B instrument. Proton NMR spectra were recorded on a Bruker 400 MHz AC instrument.

Crystal data

$C_{19}H_{15}N_3O_4S_2$	Mo $K\alpha$ radiation
M_r = 413.47	λ = 0.71069 Å
Triclinic	Cell parameters from 25 reflections
$P\bar{1}$	θ = 16–20°
a = 10.779 (2) Å	μ = 0.305 mm^{-1}
b = 12.241 (2) Å	T = 296 K
c = 7.540 (2) Å	Needle
α = 98.50 (2)°	0.20 × 0.20 × 0.15 mm
β = 103.95 (2)°	Yellow
γ = 74.24 (1)°	
V = 925.6 (7) Å^3	
Z = 2	
D_x = 1.483 Mg m^{-3}	

Data collection

Rigaku AFC-5*S* diffractometer	R_{int} = 0.039
ω–2θ scans	θ_{max} = 25°
Absorption correction: none	h = 0 → 14
3443 measured reflections	k = −16 → 16
3254 independent reflections	l = −10 → 10
1963 observed reflections [$I > \sigma(I)$]	3 standard reflections monitored every 150 reflections
	intensity variation: none

Refinement

Refinement on F	$w = 1/\sigma^2(F)$
R = 0.051	$(\Delta/\sigma)_{max}$ = 0.03
wR = 0.046	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
S = 1.25	$\Delta\rho_{min}$ = −0.29 e Å^{-3}
1963 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
298 parameters	
Only coordinates of H atoms refined	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
S(1)	0.4396 (1)	0.42936 (9)	0.6825 (2)	3.69 (5)
S(2)	0.8119 (1)	0.02513 (9)	0.9996 (2)	4.69 (6)
O(1)	−0.0517 (3)	0.8816 (3)	0.4391 (6)	7.7 (2)
O(2)	−0.1363 (3)	0.7469 (3)	0.2914 (5)	6.5 (2)
O(3)	0.2253 (3)	0.2726 (3)	1.2039 (5)	6.8 (2)
O(4)	0.3084 (4)	0.1573 (4)	1.4140 (6)	9.4 (3)
N(1)	0.6938 (3)	0.3101 (3)	0.8209 (4)	3.3 (2)
N(2)	−0.0470 (3)	0.7810 (4)	0.3957 (6)	4.7 (2)
N(3)	0.3146 (4)	0.1988 (4)	1.2794 (7)	5.6 (2)
C(1)	0.6904 (4)	0.4204 (3)	0.8705 (5)	3.1 (2)
C(2)	0.7972 (4)	0.4568 (4)	0.9788 (6)	3.6 (2)
C(3)	0.9118 (4)	0.3770 (4)	1.0386 (6)	4.0 (2)
C(4)	0.9165 (4)	0.2625 (4)	0.9885 (6)	3.9 (2)
C(5)	0.8065 (4)	0.2326 (3)	0.8800 (5)	3.4 (2)

C(6)	0.5635 (4)	0.5047 (3)	0.7990 (6)	3.5 (2)
C(7)	0.3012 (3)	0.5409 (3)	0.6098 (5)	3.0 (2)
C(8)	0.1924 (4)	0.5060 (4)	0.5007 (6)	3.9 (2)
C(9)	0.0795 (4)	0.5840 (4)	0.4312 (6)	4.2 (2)
C(10)	0.0725 (4)	0.6980 (3)	0.4733 (5)	3.4 (2)
C(11)	0.1774 (4)	0.7345 (3)	0.5831 (6)	3.9 (2)
C(12)	0.2918 (4)	0.6561 (3)	0.6504 (6)	3.5 (2)
C(13)	0.8038 (4)	0.1100 (4)	0.8184 (7)	4.4 (2)
C(14)	0.6616 (4)	0.0815 (3)	1.0709 (6)	3.6 (2)
C(15)	0.6553 (4)	0.0481 (4)	1.2361 (7)	4.2 (2)
C(16)	0.5433 (5)	0.0860 (4)	1.3059 (7)	4.6 (3)
C(17)	0.4350 (4)	0.1575 (3)	1.2068 (7)	4.0 (2)
C(18)	0.4365 (4)	0.1907 (3)	1.0414 (7)	4.1 (2)
C(19)	0.5499 (4)	0.1530 (3)	0.9718 (6)	3.8 (2)

Table 2. *Selected geometric parameters* (Å, °)

S(1)—C(6)	1.809 (5)	C(3)—C(4)	1.386 (6)
S(1)—C(7)	1.764 (3)	C(4)—C(5)	1.374 (6)
S(2)—C(13)	1.808 (6)	C(5)—C(13)	1.510 (6)
S(2)—C(14)	1.759 (4)	C(7)—C(8)	1.392 (6)
O(1)—N(2)	1.218 (6)	C(7)—C(12)	1.379 (6)
O(2)—N(2)	1.215 (5)	C(8)—C(9)	1.370 (5)
O(3)—N(3)	1.217 (5)	C(9)—C(10)	1.370 (6)
O(4)—N(3)	1.223 (8)	C(10)—C(11)	1.370 (6)
N(1)—C(1)	1.340 (5)	C(11)—C(12)	1.378 (5)
N(1)—C(5)	1.346 (4)	C(14)—C(15)	1.389 (7)
N(2)—C(10)	1.462 (5)	C(14)—C(19)	1.401 (5)
N(3)—C(17)	1.464 (7)	C(15)—C(16)	1.371 (7)
C(1)—C(2)	1.381 (6)	C(16)—C(17)	1.380 (6)
C(1)—C(6)	1.507 (5)	C(17)—C(18)	1.373 (8)
C(2)—C(3)	1.375 (5)	C(18)—C(19)	1.384 (7)
C(6)—S(1)—C(7)	102.7 (2)	S(1)—C(7)—C(12)	126.6 (3)
C(13)—S(2)—C(14)	104.9 (2)	C(8)—C(7)—C(12)	118.5 (3)
C(1)—N(1)—C(5)	117.9 (3)	C(7)—C(8)—C(9)	121.0 (4)
O(1)—N(2)—O(2)	123.1 (4)	C(8)—C(9)—C(10)	119.5 (4)
O(1)—N(2)—C(10)	117.9 (4)	N(2)—C(10)—C(9)	119.3 (4)
O(2)—N(2)—C(10)	119.0 (4)	N(2)—C(10)—C(11)	120.0 (4)
O(3)—N(3)—O(4)	122.9 (5)	C(9)—C(10)—C(11)	120.7 (3)
O(3)—N(3)—C(17)	118.9 (5)	C(10)—C(11)—C(12)	119.9 (4)
O(4)—N(3)—C(17)	118.2 (4)	C(7)—C(12)—C(11)	120.5 (4)
N(1)—C(1)—C(2)	122.7 (3)	S(2)—C(13)—C(5)	114.8 (3)
N(1)—C(1)—C(6)	116.4 (3)	S(2)—C(14)—C(15)	115.9 (3)
C(2)—C(1)—C(6)	120.9 (4)	S(2)—C(14)—C(19)	125.0 (4)
C(1)—C(2)—C(3)	118.9 (4)	C(15)—C(14)—C(19)	119.1 (4)
C(2)—C(3)—C(4)	119.0 (4)	C(14)—C(15)—C(16)	121.4 (4)
C(3)—C(4)—C(5)	118.9 (3)	C(15)—C(16)—C(17)	118.5 (5)
N(1)—C(5)—C(4)	122.6 (4)	N(3)—C(17)—C(16)	119.2 (5)
N(1)—C(5)—C(13)	115.2 (3)	N(3)—C(17)—C(18)	118.9 (4)
C(4)—C(5)—C(13)	122.1 (3)	C(16)—C(17)—C(18)	121.9 (4)
S(1)—C(6)—C(1)	109.6 (3)	C(17)—C(18)—C(19)	119.6 (4)
S(1)—C(7)—C(8)	114.9 (3)	C(14)—C(19)—C(18)	119.6 (5)

S(1)—C(6)—C(1)—N(1)	6.8 (5)
S(1)—C(6)—C(1)—C(2)	−174.3 (3)
S(2)—C(13)—C(5)—N(1)	−112.0 (4)
S(2)—C(13)—C(5)—C(4)	68.1 (5)
O(1)—N(2)—C(10)—C(9)	178.2 (4)
O(1)—N(2)—C(10)—C(11)	−3.4 (6)
O(2)—N(2)—C(10)—C(9)	−2.0 (6)
O(2)—N(2)—C(10)—C(11)	176.4 (4)
O(3)—N(3)—C(17)—C(16)	170.9 (4)
O(3)—N(3)—C(17)—C(18)	−9.3 (6)
O(4)—N(3)—C(17)—C(16)	−9.0 (7)
O(4)—N(3)—C(17)—C(18)	170.7 (5)
C(1)—C(6)—S(1)—C(7)	−179.9 (3)
C(5)—C(13)—S(2)—C(14)	67.7 (4)
C(6)—S(1)—C(7)—C(8)	174.8 (3)
C(6)—S(1)—C(7)—C(12)	−5.2 (4)
C(13)—S(2)—C(14)—C(15)	−166.0 (3)
C(13)—S(2)—C(14)—C(19)	16.0 (4)

The structure was solved by direct methods (Gilmore, 1984) and successive Fourier syntheses. Refinement was performed with full-matrix least-squares methods, with non-H atoms anisotropic and H atoms with fixed displacement parameters (1.2 × B_{eq} of the carrying atom). The calculations were performed with *TEXSAN* (Molecular Structure Corporation, 1989) software using a VAXstation 3520 computer. The figures were drawn using *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances involving H atoms have been deposited with the IUCr (Reference: AB1100). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-(*o*-Methoxyphenoxy)-1-methylbenzimidazole, $C_{15}H_{14}N_2O_2$

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Abstract

The benzimidazole ring is planar and makes a dihedral angle of 79.02 (2)° with the aryloxy ring. The N—CH_3 and O—CH_3 groups are *anti* to each other. The molecules are held together in the crystal by van der Waals interactions.

Comment

Several 1-dialkylaminoalkyl-2-aryloxybenzimidazoles have been found to exhibit excellent muscle-relaxant