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Key indicators

Single-crystal X-ray study

T = 97 K

Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$

R factor = 0.044

wR factor = 0.122

Data-to-parameter ratio = 17.1

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

2-Methyl-*N,N'*-bis[2-(methylsulfanyl)ethyl]-2-(2-pyridyl)-*N,N'*-ditosylpropane-1,3-diamine

The crystal structure of the title compound, $\text{C}_{29}\text{H}_{39}\text{N}_3\text{O}_4\text{S}_4$, was determined at 97 K and possesses normal geometric parameters.

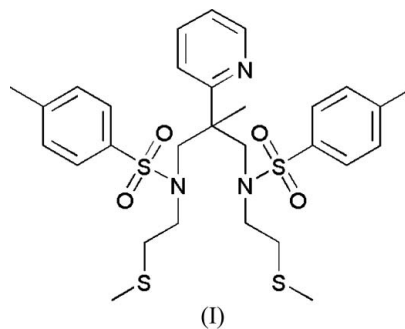
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Comment

Organic ligands containing pyridyl and thioether functional groups have been used to study the copper coordination chemistry relevant to bio-inorganic systems (Klein *et al.*, 2004; Nonoyama *et al.*, 1975; Nonoyama, 1975; Karlin *et al.*, 1979; Champloy *et al.*, 1998). Recent efforts in our laboratory have focused on generating a family of ligands that contain a pyridyl group with two thioether arms (Klein *et al.*, 2005). We report here the crystal structure of the tosylated thioether amine ligand 2-methyl-*N,N'*-bis[2-(methylsulfanyl)ethyl]-2-(2-pyridyl)-*N,N'*-ditosylpropane-1,3-diamine, (I).



The title compound was synthesized by the deprotonation of *N*-(2-methylsulfanylethyl)-*p*-toluenesulfonamide with sodium hydride and subsequent addition of $\text{CH}_3\text{C}(\text{C}_5\text{H}_4\text{N})(\text{CH}_2\text{OTs})_2$ to form the tosylated amine product.

The molecular structure of (I) possesses normal geometric parameters. Intermolecular interactions between molecules are limited to polar and van der Waals interactions.

Experimental

N-(2-methylsulfanylethyl)-*p*-toluenesulfonamide (Mizukami & Kono, 1970) (6.618 g, 27.01 mmol) was dissolved in dry DMF under N_2 and heated to 378 K. Sodium hydride (2.72 g, 113.4 mmol) was added to this solution with stirring at 378 K. Stirring at 378 K was continued for 1 h following the cessation of hydrogen gas evolution. The reaction mixture was cooled to room temperature and the DMF solution was filtered from excess sodium hydride. $\text{CH}_3\text{C}(\text{C}_5\text{H}_4\text{N})(\text{CH}_2\text{OTs})_2$ (Friedrich *et al.*, 1997) (6.416 g, 13.50 mmol), dissolved in DMF, was added to this solution over a period of 2 h and stirred at 378 K for an additional 2 h. The reaction mixture was cooled to room temperature and poured into water. The aqueous solution was extracted with chloroform. The organic extracts were

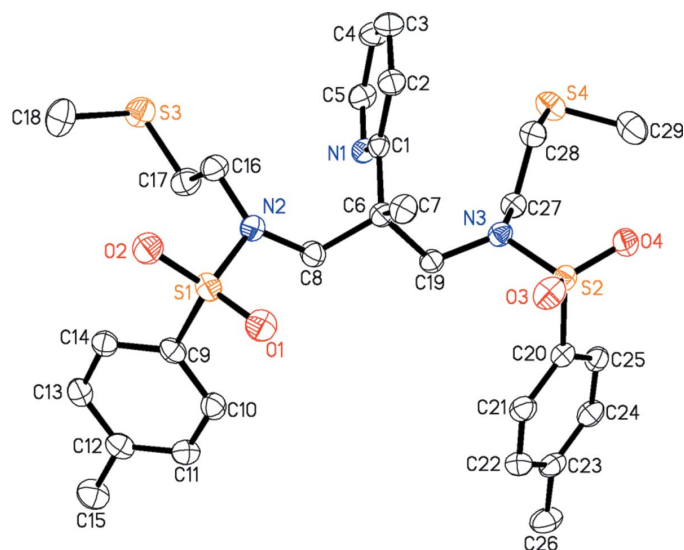


Figure 1
View of the title complex, showing the atom-labeling scheme. H atoms have been omitted for clarity. Displacement ellipsoids are drawn at the 50% probability level.

combined and the chloroform removed *in vacuo* to reveal a red-brown oil. The title compound was obtained from this crude product as a white powder following recrystallization from ethanol (2.46 g, 29.3% yield). Crystals suitable for X-ray structural analysis were obtained by slow evaporation of ethanol solutions of the title compound. (m.p. 406–409 K). ^1H NMR (300 MHz, CDCl_3): δ 8.45–8.49 (*m*, 1H), 7.59–7.66 (*m*, 5H), 7.36–7.41 (*m*, 1H), 7.22–7.26 (*m*, 4H), 7.10–7.15 (*m*, 1H), 3.70 (*d*, $J = 14$ Hz, 2H), 3.39 (*d*, $J = 14$ Hz, 2H), 2.43–2.70 (*m*, 8H), 2.36 (*s*, 6H), 1.74 (*s*, 6H), 1.55 (*s*, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (75 MHz, CDCl_3): δ 163.13, 149.34, 143.82, 137.25, 136.29, 130.04, 127.68, 122.51, 122.35, 57.82, 50.51, 46.90, 31.75, 21.77, 19.80, 15.52. Analysis calculated for $\text{C}_{29}\text{H}_{39}\text{N}_3\text{O}_4\text{S}_4$: C 56.01, H 6.32, N 6.76%; found: C 55.99, H 6.30, N 6.59%.

Crystal data

$\text{C}_{29}\text{H}_{39}\text{N}_3\text{O}_4\text{S}_4$	$Z = 2$
$M_r = 621.87$	$D_x = 1.306 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 11.446$ (2) Å	Cell parameters from 8357 reflections
$b = 12.166$ (2) Å	$\theta = 2.3$ – 28.2°
$c = 12.584$ (2) Å	$\mu = 0.34 \text{ mm}^{-1}$
$\alpha = 92.881$ (3) $^\circ$	$T = 97$ (2) K
$\beta = 112.681$ (3) $^\circ$	Block, colorless
$\gamma = 99.595$ (3) $^\circ$	$0.47 \times 0.33 \times 0.31 \text{ mm}$
$V = 1581.5$ (5) Å 3	

Data collection

Bruker APEX CCD area-detector diffractometer	6174 independent reflections
ω scans	5852 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 2002)	$R_{\text{int}} = 0.023$
$T_{\text{min}} = 0.857$, $T_{\text{max}} = 0.902$	$\theta_{\text{max}} = 26.0^\circ$
17215 measured reflections	$h = -14 \rightarrow 14$
	$k = -14 \rightarrow 14$
	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 1.42P]$
$R[F^2 > 2\sigma(F^2)] = 0.044$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.122$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.00$	$\Delta\rho_{\text{max}} = 0.87 \text{ e \AA}^{-3}$
6174 reflections	$\Delta\rho_{\text{min}} = -0.49 \text{ e \AA}^{-3}$
361 parameters	
H-atom parameters constrained	

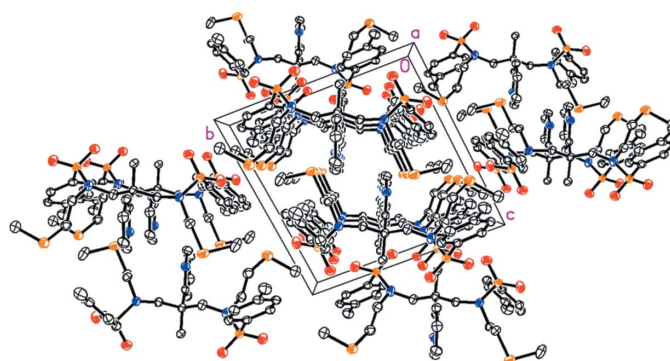


Figure 2
The molecular packing of the title complex, viewed along the a axis. H atoms have been omitted for clarity. Displacement ellipsoids are drawn at the 50% probability level.

Table 1

Selected geometric parameters (Å, $^\circ$).

S3–C18	1.802 (2)	N2–C8	1.470 (2)
S3–C17	1.808 (2)	N2–C16	1.479 (3)
S4–C29	1.799 (3)	N3–C19	1.472 (2)
S4–C28	1.806 (2)	N3–C27	1.492 (2)
C18–S3–C17	100.42 (11)	C7–C6–C19	109.87 (15)
C29–S4–C28	98.76 (11)	C19–C6–C8	101.91 (14)
C8–N2–C16	118.57 (16)	N2–C8–C6	115.19 (15)
C8–N2–S1	116.49 (13)	N2–C16–C17	114.24 (16)
C16–N2–S1	118.99 (13)	C16–C17–S3	111.55 (14)
C19–N3–C27	116.72 (15)	N3–C19–C6	115.02 (15)
C19–N3–S2	116.48 (12)	N3–C27–C28	111.47 (15)
C27–N3–S2	115.53 (12)	C27–C28–S4	111.05 (13)
C9–S1–N2–C8	−64.34 (16)	C27–N3–C19–C6	−101.63 (18)
C9–S1–N2–C16	88.16 (16)	C1–C6–C19–N3	57.0 (2)
C20–S2–N3–C19	64.97 (14)	C7–C6–C19–N3	−67.7 (2)
C20–S2–N3–C27	−77.65 (14)	C8–C6–C19–N3	174.47 (15)
C8–N2–C16–C17	61.6 (2)	C19–N3–C27–C28	119.73 (17)
N2–C16–C17–S3	176.98 (14)	N3–C27–C28–S4	177.34 (13)
C18–S3–C17–C16	−75.92 (18)	C29–S4–C28–C27	−86.41 (16)

H atoms were positioned geometrically ($\text{C–H} = 0.95$ – 0.99 Å) and refined using a riding model, with $U_{\text{iso}} = 1.2$ (1.5 for methyl) times $U_{\text{eq}}(\text{C})$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1998); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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