trans-Dipyridinebis(sulfamerazinato)-nickel(II)–pyridine (1/4)

G. M. Golzar Hossain* and A. J. Amoroso

School of Chemistry, Cardiff University, Cardiff CF10 3AT, Wales

Correspondence e-mail: acsbd@yahoo.com

The title compound, $[\text{Ni}(\text{C}_{11}\text{H}_{11}\text{N}_4\text{O}_2\text{S})_2(\text{C}_5\text{H}_5\text{N})_2]\cdot4\text{C}_5\text{H}_5\text{N}$, contains the centrosymmetric octahedral complex *trans*-[Ni(smr)$_2$(py)$_2$] (where smr is the sulfamerazinate anion and py is pyridine) linked to four pyridine molecules *via* N—H···N hydrogen bonds. This is the first crystal structure of a metal complex of sulfamerazine.

Comment

The sulfamerazine molecule was introduced into medical therapy because, like many sulfonamide derivatives, it exhibits antibacterial activity. The presence of several potential donor sites, namely the amino, pyrimidine and sulfonamide N atoms and the sulfonyl O atoms, make this ligand a versatile complexing agent. Here, we report the structure of the title nickel complex, (I), of the sulfamerazinate anion (Fig. 1). This is the first crystal structure of a metal complex of sulfamerazine.

(I)
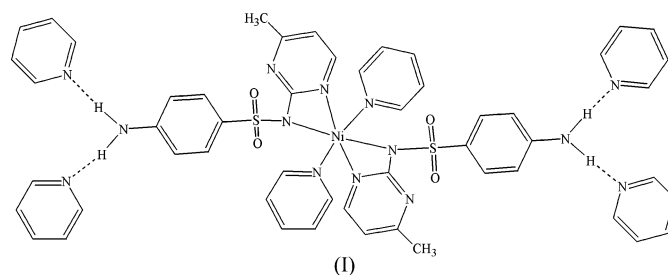
In complex (I), the Ni atom lies on a centre of inversion, and the complex contains two bidentate N-coordinated sulfamerazinate anions and two pyridine molecules occupying the *trans* sites. In addition, four pyridine molecules are linked *via* N—H···N hydrogen bonds to the terminal amino groups of the sulfamerazinate ligands (Table 1, Fig. 1).

The relative orientations of the sulfamerazinate ligands and the coordinated pyridine molecules are such that they are nearly perpendicular to each other. Such an orientation of the ligands around the Ni atom appears to be dictated more by steric considerations than any other factors.

The Ni—N bond distances involving the sulfonamide atom N11, the pyrimido atom N12 and the pyrimidine atom N1 are very similar, at 2.139 (2), 2.100 (2) and 2.080 (2) Å, respectively. The tetrahedral coordination at S is distorted, as also found in the neutral sulfamerazine molecule. The endocyclic angle at C11 in complex (I) is 125.2 (2)°, which is somewhat smaller than the corresponding values found in the various polymorphs of the free sulfamerazine molecule [127.5 (2) (Hossain, 2006), 127.1 (7) (Acharya *et al.*, 1982), and 127.1 (4)
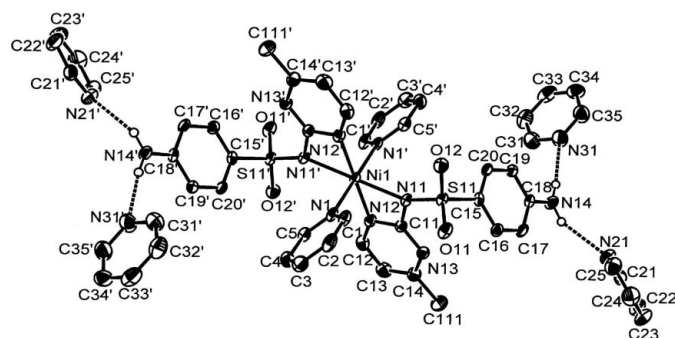
Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms bonded to C atoms have been omitted for clarity. Hydrogen bonds are shown as dashed lines. Atoms marked with a prime are at the symmetry position $(1 - x, 1 - y, 1 - z)$.

and 128.2 (4)° (Caria & Mohamed, 1992)], due to the coordination to the Ni centre.

The S—O bond distances of 1.4460 (15) and 1.4435 (17) Å in (I) are longer than the corresponding bonds in pure sulfamerazine, where the values obtained are 1.4398 (16) and 1.4293 (17) Å (Hossain, 2006), 1.430 (6) and 1.441 (6) Å (Acharya *et al.*, 1982), and 1.424 (4) and 1.435 (3), and 1.414 (4) and 1.431 (3) Å (Caria & Mohamed, 1992).

The H atoms of the amino groups form intermolecular hydrogen bonds with the N atoms of four pyridine molecules (Table 1, Fig. 1).

Experimental

Solid sulfamerazine (Hsmr) (0.529 g, 2 mmol) was dissolved in hot methanol (50 ml) and a methanolic solution (10 ml) of $NiCl_2 \cdot 6H_2O$ (0.238 g, 1 mmol) was added slowly with constant stirring on a hot plate at 333 K. A pink precipitate formed and stirring of the mixture was continued for 6 h. The precipitate was then filtered off and dried over silica gel. The precipitate was dissolved in a mixture of pyridine and water (10 ml, 1:10 *v/v*) and stirred for 30 min. The solution was then filtered and left for crystallization, and a week later pale-violet block-shaped crystals of (I) were obtained. These were removed by filtration and dried over silica gel.

Crystal data

$[Ni(C_{11}H_{11}N_4O_2S)_2(C_5H_5N)_2] \cdot 4C_5H_5N$	$\gamma = 84.144\ (1)°$
$M_r = 1059.91$	$V = 1311.85\ (5)$ Å^3
Triclinic, $P\bar{1}$	$Z = 1$
$a = 9.9781\ (2)$ Å	$D_x = 1.342$ Mg m^{-3}
$b = 10.0610\ (2)$ Å	Mo $K\alpha$ radiation
$c = 13.2582\ (4)$ Å	$\mu = 0.51$ mm^{-1}
$\alpha = 89.744\ (1)°$	$T = 150\ (2)$ K
$\beta = 82.238\ (1)°$	Block, pale violet
	$0.18 \times 0.16 \times 0.06$ mm

Data collection

Nonius KappaCCD area-detector diffractometer	24079 measured reflections
ω scans	5955 independent reflections
Absorption correction: multi-scan (Blessing, 1995)	4409 reflections with $I > 2\sigma(I)$
$T_{min} = 0.914$, $T_{max} = 0.970$	$R_{int} = 0.159$
	$\theta_{max} = 27.5°$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.053$	$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2]$
$wR(F^2) = 0.143$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.001$
5955 reflections	$\Delta\rho_{max} = 0.50$ e Å^{-3}
332 parameters	$\Delta\rho_{min} = -1.13$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N14—H14B$\cdots$N31	0.88	2.15	3.029 (3)	173
N14—H14A$\cdots$N21	0.88	2.33	3.067 (3)	141

All H atoms were treated as riding atoms, with C—H = 0.95 Å and N—H = 0.88 Å, and with $U_{iso}(H) = 1.2U_{eq}(C,N)$, or $1.5U_{eq}(C)$ for the methyl groups. The mosaicity of the compound was high, so R_{int} is 0.159. The deepest hole is located 0.90 Å from atom Ni1.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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