metal-organic papers

Aqua{2-[(*E*)-(3,5-dibromo-2-oxidophenyl)-methyleneamino]ethanesulfonato-κ^3O,N,O'}-(1,10-phenanthroline-κ^2N,N')nickel(II) ethanol solvate

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Key indicators

Single-crystal X-ray study
$T = 296$ K
Mean σ(C–C) = 0.009 Å
R factor = 0.044
wR factor = 0.093
Data-to-parameter ratio = 14.0

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

The title compound, [Ni(C$_9$H$_9$Br$_2$NO$_4$S)(C$_8$H$_8$N$_2$)(H$_2$O)]·C$_2$H$_5$OH, was synthesized in a water–ethanol solution. In the structure, the Ni atom is six-coordinated by three ligands to form the neutral complex, in which the Ni atom has a distorted octahedral coordination geometry. Molecules form dimers through O—H···O hydrogen bonds.

Comment

Sulfur- and amino-acid-containing Schiff base complexes have been studied for many years (Casella & Gullotti, 1981, 1986; Wang *et al.*, 1994; Jiang *et al.*, 2003; Zhang *et al.*, 2003) and have aroused increasing interest because of their antiviral, anti-cancer and antibacterial activities. Taurine, an amino acid containing sulfur, is indispensable to humans and has important physiological functions. Recently, Schiff base complexes derived from taurine have been reported (Zhang *et al.*, 2003, 2004; Jiang *et al.*, 2004; Xu *et al.*, 2004). We report here the synthesis and crystal structure of a new nickel(II) complex, (I), prepared by the reaction of Ni(OAc)$_2$·4H$_2$O, and the sodium salt of the Schiff base ligand 2-[(*E*)-(3,5-dibromo-2-hydroxyphenyl)methyleneamino]ethanesulfonic acid, which was derived in turn by the reaction of taurine with 3,5-dibromosalicylaldehyde and 1,10-phenanthroline (phen).

(I)
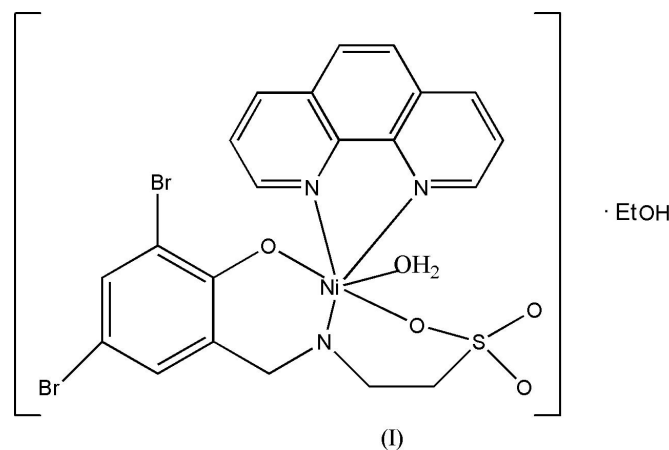
The title complex is isostructural with [Zn(C$_9$H$_9$Br$_2$NO$_4$S)-(C$_8$H$_8$N$_2$)(H$_2$O)]·C$_2$H$_5$OH, for which we have already determined the structure (Zhang *et al.*, 2005). As shown in Fig. 1, the molecule of (I) is mononuclear. The Ni atom is coordinated by one N and two O atoms of the tridentate ligand (*L*), two N atoms of the bidentate ligand (phen) and one O atom of the coordinated water, forming a slightly distorted octahedral geometry, with atoms O1 and O2 of *L* in axial positions. The sum of the equatorial coordination angles (see Table 1) is close

to 360° (359.58°), and the bond angles involving the Ni atom are similar to values observed in nickel complexes of a salicylaldehyde Schiff base and are in good agreement with those in other taurine-containing complexes. The Ni1—O1 bond length is shorter than Ni1—N1, indicating that the hydroxyl O atom has a stronger coordination ability than the imine N atom, while sulfonate atom O2 has stronger coordination ability than water atom O5, indicating that the coordination ability of the sulfonate has been reinforced by chemical modification (Cai *et al.*, 2001).

Molecules related by twofold axes form dimers through O—H···O hydrogen bonding (see Table 2 and Fig. 2). These dimers are, in turn, linked by weaker C—H···O interactions (which are not discussed in detail here) into a three-dimensional network.

Experimental

The sodium salt of the Schiff base ligand 2-[(*E*)-(3,5-dibromo-2-hydroxyphenyl)methyleneamino]ethanesulfonic acid was synthesized as reported previously (Xu *et al.*, 2004). The ligand (2.0 mmol) was dissolved in aqueous ethanol (25 ml). To this solution, Ni(OAc)$_2$·4H$_2$O (2.0 mmol) was added and the mixture was stirred under reflux at 333 K for 6 h. Phen (2.0 mmol) was added and the reaction continued for a further 2 h. After cooling to room temperature and filtration, the filtrate was left to stand at room temperature. Blue crystals suitable for X-ray diffraction were obtained in a yield of 61.5%. Analysis found (%): C 40.18, H 3.34, N 6.07, S 4.63, Ni 8.29; C$_{23}$H$_{23}$Br$_2$NiN$_3$O$_6$S requires (%): C 40.15, H 3.37, N 6.10, S 4.66, Ni 8.27; IR (KBr, ν cm^{-1}): 1033.1, 1038.4, 1149.5, 1185.4 (—SO$_3$), 1624.5 (ν C=N), 1604.5, 1521.6 (ν C=N + C=C), 3419.2 (ν O—H).

Crystal data

[Ni(C$_9$H$_9$Br$_2$NO$_4$S)(C$_8$H$_8$N$_2$)-(H$_2$O)]·C$_2$H$_6$O	D_x = 1.738 Mg m^{-3}
M_r = 688.03	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 33 reflections
a = 18.725 (4) Å	θ = 2.8–14.1°
b = 19.623 (4) Å	μ = 3.90 mm^{-1}
c = 15.647 (3) Å	T = 296 (2) K
β = 113.82 (1)°	Block, blue
V = 5259.6 (19) Å^3	0.54 × 0.38 × 0.36 mm
Z = 8	

Data collection

Siemens *P*4 diffractometer	R_{int} = 0.032
ω scans	θ_{max} = 25.3°
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	h = 0 → 22
	k = 0 → 23
T_{min} = 0.165, T_{max} = 0.246	l = −18 → 17
5341 measured reflections	3 standard reflections
4776 independent reflections	every 97 reflections
2204 reflections with $I > 2\sigma(I)$	intensity decay: 1.1%

Refinement

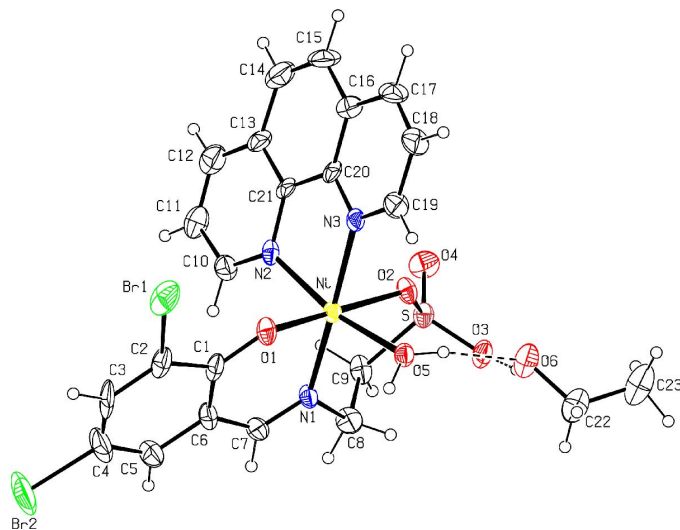|	
Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0406P)^2]$
$R[F^2 > 2\sigma(F^2)]$ = 0.044	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.093	$(\Delta/\sigma)_{max}$ = 0.001
S = 0.80	$\Delta\rho_{max}$ = 0.49 e Å^{-3}
4776 reflections	$\Delta\rho_{min}$ = −0.62 e Å^{-3}
340 parameters	Extinction correction: *SHELXL97*
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.00032 (4)

Figure 1
View of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Dashed lines indicate hydrogen bonds.
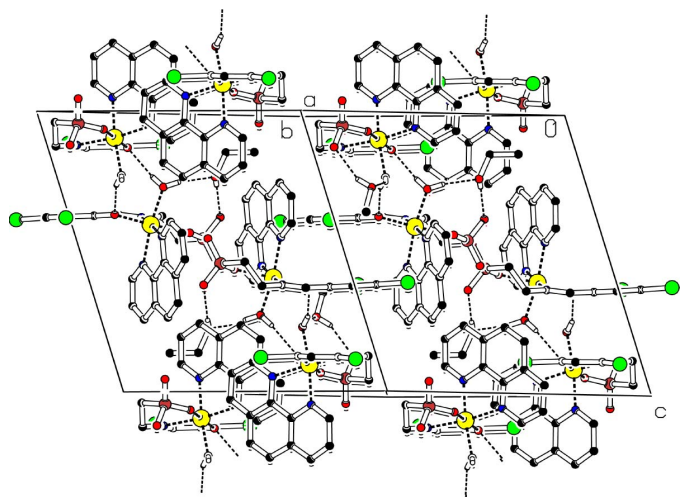

Figure 2
Packing diagram (Spek, 2003) of the title complex, showing the hydrogen bonds as dashed lines. H atoms not involved in hydrogen bonds have been omitted.

Table 1
Selected geometric parameters (Å, °).

Ni—O1	2.007 (3)	S—O2	1.473 (3)
Ni—N1	2.058 (4)	N1—C7	1.271 (6)
Ni—O5	2.070 (4)	N1—C8	1.465 (6)
Ni—O2	2.096 (3)	N2—C10	1.319 (6)
Ni—N3	2.104 (4)	N2—C21	1.366 (6)
Ni—N2	2.109 (4)	N3—C19	1.327 (6)
S—O4	1.438 (4)	N3—C20	1.356 (6)
S—O3	1.447 (4)		
O1—Ni—N1	90.79 (16)	O5—Ni—N3	90.99 (18)
O1—Ni—O5	90.08 (16)	O2—Ni—N3	83.35 (14)
N1—Ni—O5	94.54 (17)	O1—Ni—N2	95.51 (15)
O1—Ni—O2	175.06 (14)	N1—Ni—N2	94.85 (17)
N1—Ni—O2	92.40 (15)	O5—Ni—N2	168.99 (17)
O5—Ni—O2	85.90 (16)	O2—Ni—N2	87.98 (14)
O1—Ni—N3	93.85 (15)	N3—Ni—N2	79.20 (18)
N1—Ni—N3	172.77 (18)		

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Table 2
Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O5—H5A···O6	0.82 (7)	1.99 (6)	2.759 (7)	156 (6)
O5—H5B···O1^i	0.81 (4)	1.98 (4)	2.767 (5)	162 (5)
O6—H6O···O3	0.82 (7)	2.05 (6)	2.828 (7)	157 (6)

Symmetry code: (i) $1-x, y, \frac{3}{2}-z$.

The H atoms on O5 and O6 were located in a difference Fourier map and their positions and isotropic displacement parameters were refined, with the O—H distances constrained in the range 0.816–0.820 Å. All other H atoms were positioned geometrically and were treated as riding atoms and refined isotropically, with C—H distances of 0.93–0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Siemens, 1994); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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