solvent molecule. H atoms were placed geometrically and refined with a riding model (including free rotation about C—C bonds for methyl groups) and with U_{iso} constrained to be 1.2 (1.5 for methyl groups) times U_{eq} of the carrier atom.

For all compounds, data collection: *SMART* (Siemens, 1994*b*); cell refinement: *SAINT* (Siemens, 1994*b*); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1994*a*); software used to prepare material for publication: *SHELXL*97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1212). Services for accessing these data are described at the back of the journal.

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Bis(tetraethylammonium) Bis[2-(trifluoroacetylamidato)benzenethiolato(2–)-*S*,*N*]-nickelate(II)

Norikazu Ueyama, Tomo-aki Tuji, Taka-aki Okamura and Akira Nakamura

Department of Macromolecular Science, Graduate School of Science, Osaka University, Toyonaka, Osaka 560-0043, Japan. E-mail: ueyama@chem.sci.osaka-u.ac.jp

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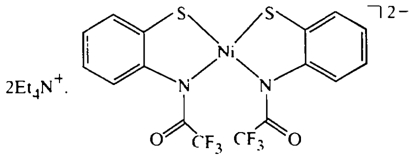
Abstract

The title compound, bis(tetraethylammonium) bis[*N*-(2-mercaptophenyl)trifluoroacetamidato(2–)-*N*,*S*]-nickelate(II), $(C_8H_{20}N)_2[Ni(C_8H_4F_3NOS)_2]$, consists of a distorted square-planar NiS_2N_2 core composed of two thiolate S and two deprotonated amide N atoms. The Ni—S bond distances are 2.177 (1) and 2.171 (1) Å, and the Ni—N bond distances are 1.954 (3) and 1.959 (3) Å.

Comment

Previously, we have reported deprotonation of the highly acidic CF_3CONH amide in the Co^{II}-thiolate complex, $(PPh_4)_2[Co\{S\text{-}2,6\text{-}(CF_3CONH)_2C_6H_3\}_4]$, resulting in *S*,*N*-chelate formation (Okamura *et al.*, 1998). The deprotonation was carried out by either air or amine *N*-oxide oxidation, producing a disulfide. On the other hand, numerous Ni^{II} complexes having an *S*(thiolato),*N*(amidate)-five-membered chelate ring have been synthesized using strong bases (Krüger *et al.*, 1991). Numerous related *S*,*N*-chelate Ni^{II} complexes with a thiolate-containing Schiff base have also been reported (Das & Livingstone, 1976; Ercan *et al.*, 1996). In this paper, we report the structure of the title Ni^{II} complex, (I).



(I)

The title complex exhibits a distorted square-planar Ni^{II} ion, with S1—Ni—N2 and S2—Ni—N1 angles of 169.04 (10) and 169.09 (9)°, respectively, as shown in Fig. 1. The S1—Ni—S2 angle of 87.84 (5)° indicates *cis* coordination of the two thiolate ligands. Atoms N1 and S1, or N1 and S2, are in the plane of their respective aromatic ring. These bond distances and angles are similar to those of the neutral Schiff base Ni^{II} complex, $[Ni^{II}(C_{13}H_9ClNS)_2]$ (Ercan *et al.*, 1996).

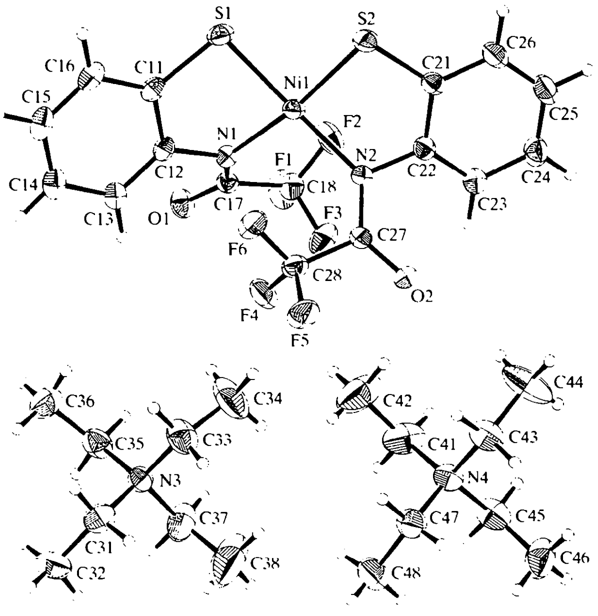


Fig. 1. A view of (I), with displacement ellipsoids drawn at the 25% probability level.

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Experimental

The synthesis of (I) was carried out by the reaction of (NEt_4)-$[Ni^{II}(SPh)_4]$ with $[S\text{-}o\text{-}(CF_3CONH)C_6H_4]_2$ in the presence of trimethylamine *N*-oxide in acetonitrile at room temperature (molar ratio 1:2:2). Dark-brown crystals were obtained from the solution.

Crystal data

$(C_8H_{20}N)_2[Ni(C_8H_4F_3NOS)_2]$
$M_r = 757.57$
Monoclinic
$P2_1/c$
$a = 11.951\,(4)$ Å
$b = 10.590\,(4)$ Å
$c = 28.852\,(2)$ Å
$\beta = 91.57\,(1)^\circ$
$V = 3650\,(1)$ Å^3
$Z = 4$
$D_x = 1.378$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 14.4$–14.9°
$\mu = 0.711$ mm^{-1}
$T = 296.2$ K
Prismatic
$0.6 \times 0.5 \times 0.5$ mm
Dark brown

Data collection

Rigaku AFC-5*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
9268 measured reflections
8854 independent reflections
4485 reflections with $I > 3\sigma(I)$

$R_{\rm int} = 0.036$
$\theta_{\rm max} = 27.5^\circ$
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 13$
$l = -37 \rightarrow 37$
3 standard reflections every 150 reflections intensity decay: none

Refinement

Refinement on *F*
$R = 0.045$
$wR = 0.047$
$S = 1.828$
4485 reflections
425 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o) + 0.00010|F_o|^2]$

$(\Delta/\sigma)_{\rm max} = 0.078$
$\Delta\rho_{\rm max} = 0.55$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.39$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Ni1—S1	2.177 (1)	S1—C11	1.762 (4)
Ni1—S2	2.171 (1)	S2—C21	1.749 (4)
Ni1—N1	1.954 (3)	N1—C12	1.434 (4)
Ni1—N2	1.959 (3)	N2—C22	1.428 (5)
S1—Ni1—S2	87.84 (5)	Ni1—S1—C11	95.6 (1)
S1—Ni1—N1	86.25 (9)	Ni1—S2—C21	96.0 (1)
S1—Ni1—N2	169.04 (10)	Ni1—N1—C12	111.7 (2)
S2—Ni1—N1	169.09 (9)	Ni1—N1—C17	131.2 (3)
S2—Ni1—N2	87.13 (9)	Ni1—N2—C22	112.3 (2)
N1—Ni1—N2	100.2 (1)	Ni1—N2—C27	130.7 (3)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *PATTY* in *DIRDIF*92 (Beurskens *et al.*, 1992). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1048). Services for accessing these data are described at the back of the journal.

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1,1,3,3,14,14,16,16,18,18,29,29-Dodecamethyl-3,14,18,29-tetrasila-1,16-distanna-[5.5]ferrocenophane

OLIVIER GAUSSET, GUYLAINE DELPON-LACAZE, MARKUS SCHÜRMANN AND KLAUS JURKSCHAT

Lehrstuhl für Anorganische Chemie II, Universität Dortmund, Otto-Hahn-Straße 6, 44227 Dortmund, Germany. E-mail: kjur@platon.chemie.uni-dortmund.de

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Abstract

The title compound, $[Fe_2\{Sn(C_9H_{15}Si)_2\}_2]$, is a new ferrocenophane containing two $(Me_2SiCH_2)_2SnMe_2$ bridges. The molecule lies about an inversion center. The intramolecular Si···Si distances are 7.827 (2) and 8.403 (2) Å, and the Sn···Sn intramolecular separation is 9.102 (1) Å.

Comment

In recent years, the host–guest chemistry of anion recognition has become a topic of increasing interest (Pierre & Baret, 1983; Dietrich, 1993; Schmidtchen & Berger, 1997). For some time we have been interested in the synthesis of tin- and silicon-containing multidentate Lewis acids for anion complexation (Altmann *et al.*, 1997). Some of our compounds were shown to function