Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

[*N*,*N*′-Bis-(*o*-sulfidobenzylidene)-1,3-diaminopropane]nickel(II) 1,4-dioxane solvate

Michelle K. Taylor,* John Reglinski and Dawn Wallace

Department of Pure and Applied Chemistry, University of Strathclyde, 295 Cathedral St., Glasgow G1 1XL, Scotland

Correspondence e-mail: michelle.k.taylor@strath.ac.uk

Received 5 May 2004
Accepted 17 May 2004
Online 22 May 2004

Key indicators

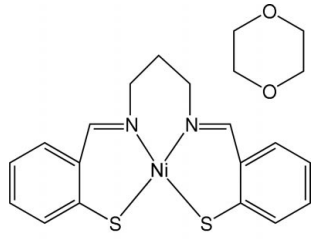
Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.003 Å
R factor = 0.029
wR factor = 0.057
Data-to-parameter ratio = 13.4

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

The title tetradentate Schiff base complex (systematic name: {2,2′-[propane-1,3-diylbis(nitrilomethylidyne)]benzenethiolato-κ^4S,N,N',S'}nickel(II) 1,4-dioxane solvate), [Ni($C_{17}H_{16}N_2S_2$)]·$C_4H_8O_2$, contains an Ni atom coordinated within a tetrahedrally distorted planar N_2S_2 environment, with average Ni—N and Ni—S bond lengths of 1.922 (1) and 2.167 (1) Å, respectively.

Comment

As part of our ongoing studies (Reglinski *et al.*, 2002*a*,*b*) on tetradentate Schiff base complexes with N_2X_2 donor sets and varying backbone lengths, the preparation of N_2S_2 complexes of this type was of interest. Eichorn & Goswami (1999) reported the use of a novel Schiff base semi-template for the formation of Ni^{II} complexes with mixed N/S-donating chelates. This method involves the reaction in ethanol of Ni^{II} complexes containing primary amine chelates and 2,2′-dithiodibenzaldehyde (DTDB). In order to assess the applicability of extending this method to the preparation of complexes with longer backbones, the title compound, (I), was prepared by this method. Crystals were obtained and the unit cell was found to be different from that of the previously reported structure of this compound (Gomes *et al.*, 1999), which has two independent nickel complex molecules in the asymmetric unit.



(I)

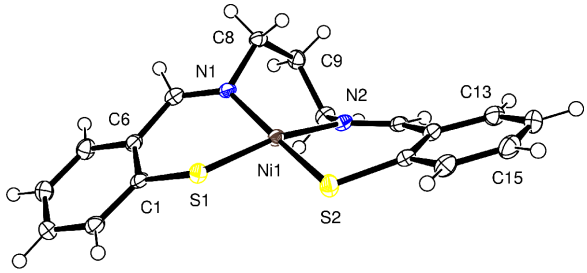


Figure 1
View of (I) (50% probability displacement ellipsoids). The solvent molecule has been omitted.

Experimental

The reaction of tris(propylenediamine)nickel(II)chloride and DTDB (Kasmai & Mischke, 1989) in ethanol produced a brown solid. Analysis found: C 54.28, H 4.58, N 7.04, S 16.62%; calculated for $C_{17}H_{16}N_2NiS_2$: C 55.01, H 4.34, N 7.55, S 17.28%; ^{1}H NMR (270 MHz; solvent $CDCl_3$): δ 7.83 (*s*, 2H, CH═N), 7.69 (*d*, 2H, aromatic), 7.22 (*d*, 2H, aromatic), 7.15 (*t*, 2H, aromatic), 7.00 (*t*, 2H, aromatic), 3.99 (*t*, 4H, ═NCH$_2$—), 2.09 (*p*, 2H, CCH$_2$C). Dark-brown crystals suitable for X-ray analysis were obtained by slow evaporation of a dioxane solution of the brown solid.

Crystal data

$[Ni(C_{17}H_{16}N_2S_2)]\cdot C_4H_8O_2$	$Z = 2$
$M_r = 459.25$	$D_x = 1.489$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 9.2099$ (3) Å	Cell parameters from 4660 reflections
$b = 9.3828$ (2) Å	$\theta = 1.6$–$27.5°$
$c = 13.2522$ (4) Å	$\mu = 1.17$ mm^{-1}
$\alpha = 77.392$ (2)°	$T = 123$ (2) K
$\beta = 88.719$ (2)°	Prism, brown
$\gamma = 66.761$ (2)°	0.25 × 0.25 × 0.20 mm
$V = 1024.30$ (5) Å^3	

Data collection

Nonius KappaCCD diffractometer	$R_{int} = 0.029$
φ and ω scans	$\theta_{max} = 27.5°$
Absorption correction: none	$h = -11 \rightarrow 11$
9186 measured reflections	$k = -11 \rightarrow 12$
4660 independent reflections	$l = -17 \rightarrow 17$
3758 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0146P)^2 + 0.5180P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.029$	
$wR(F^2) = 0.057$	
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.033$
4660 reflections	$\Delta\rho_{max} = 0.36$ e Å^{-3}
349 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
All H-atom parameters refined	

Table 1
Selected geometric parameters (Å, °).

N1—Ni1	1.9140 (14)	S1—Ni1	2.1760 (5)
N2—Ni1	1.9307 (15)	S2—Ni1	2.1574 (5)
N1—Ni1—S2	170.86 (4)	N2—Ni1—S1	169.35 (4)

All H atoms were found in a difference Fourier map and were refined isotropically [C—H = 0.90 (2)–1.02 (2) Å].

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO*; data reduction: *DENZO*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

JR wishes to thank the Cunningham Trust (Scotland) for financial assistance.

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