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[5-Bromo-2-(2-(dimethylamino) ethyliminomethyl)phenolato- $\kappa^3 N, N', O$]-isothiocyanato-nickel(II), $C_{12}H_{14}BrN_3NiOS$

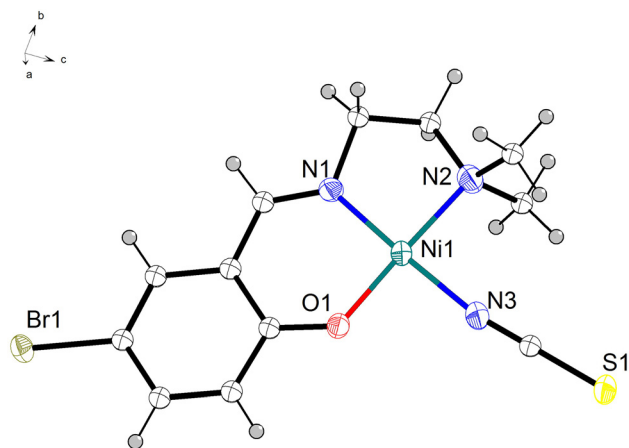


Table 1: Data collection and handling.

Crystal:	Tan block
Size:	0.12 × 0.14 × 0.16 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	4.26 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART APEX-II,
$\theta_{\max}$, completeness:	26.4°, 99%
$N(hkl)_{\text{unique}}$	2914
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 2265
$N(\text{param})_{\text{refined}}$	179
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

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Abstract

$C_{12}H_{14}BrN_3NiOS$, monoclinic, $P2_1/c$ (no. 14), $a = 7.0191(5)$ Å, $b = 10.8476(7)$ Å, $c = 19.0237(12)$ Å, $\beta = 95.564(2)^\circ$, $V = 1441.65(17)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0416$, $wR_{\text{ref}}(F^2) = 0.1007$, $T = 148$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

5-Bromosalicylaldehyde (0.201 g, 1.0 mmol) and N,N -dimethylethylenediamine (0.088 g, 1 mmol) were dissolved in

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30 mL ethanol, the Schiff base solution was stirred at room temperature for 24 h. After this, $Ni(NO_3)_2 \cdot 6H_2O$ (0.029 g, 1.0 mmol) was added and the obtained brown mixture was further stirred for another 30 min. Then, NaSCN (0.080 g, 1.0 mmol) was added and stirred for 2 h and filtered. The acquired brown filtrate was sealed in a beaker with parafilm and kept undisturbed at room temperature. Pale brown crystals of the title compound were afforded after one month by slow evaporation.

Experimental details

X-ray single crystal diffraction data was collected on a Bruker SMART APEX II diffractometer with rotating anode source (MoK α radiation, $\lambda = 0.71073$ Å) and equipped with a gas-flow apparatus at 148 K. Data reduction was performed using the program SAINT [1]. The structure was solved with the Olex2 program [2] as an interface together with the SHELXT and SHELXL programs [3, 4]. All H atoms were placed in geometrically idealized positions and refined using a riding model, with C–H = 0.98 (methylene), 0.95 Å (benzene) and 0.97 Å (methyl) ($U_{\text{iso}}(\text{H})$ values were set 1.2 times for H atoms on methylene and benzene, and 1.5 times for methyl.) WINABS-2012/1 (Bruker, 2012) was used for absorption correction.

Thus the final HKLF 4 output contains 17786 reflections with $R_{\text{int}} = 0.0667$ (8614 with $I > 3\sigma(I)$, $R_{\text{int}} = 0.0515$).

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} [*] /U _{eq}
Br1	0.79555 (7)	0.32320 (4)	0.35604 (2)	0.02832 (17)
Ni1	0.72669 (8)	0.66364 (5)	0.68527 (3)	0.02211 (18)
S1	0.83064 (19)	0.54942 (12)	0.92133 (6)	0.0337 (3)
O1	0.7272 (4)	0.5040 (3)	0.65137 (15)	0.0254 (7)
N1	0.6960 (5)	0.7339 (3)	0.59691 (19)	0.0250 (8)
N2	0.7332 (6)	0.8317 (3)	0.7230 (2)	0.0328 (9)
N3	0.7594 (6)	0.5990 (3)	0.7776 (2)	0.0300 (9)
C1	0.7327 (5)	0.5523 (4)	0.5281 (2)	0.0195 (9)
C2	0.7402 (6)	0.4689 (4)	0.5864 (2)	0.0212 (9)
C3	0.7626 (6)	0.3431 (4)	0.5721 (2)	0.0260 (10)
H3	0.766580	0.285999	0.610077	0.031*
C4	0.7790 (6)	0.2999 (4)	0.5054 (3)	0.0294 (11)
H4	0.794446	0.214222	0.497460	0.035*
C5	0.7730 (6)	0.3829 (4)	0.4491 (2)	0.0199 (9)
C6	0.7493 (6)	0.5063 (4)	0.4593 (2)	0.0211 (9)
H6	0.744070	0.561228	0.420272	0.025*
C7	0.7045 (6)	0.6804 (4)	0.5367 (2)	0.0225 (9)
H7	0.690948	0.730284	0.495493	0.027*
C8 ^a	0.6552 (10)	0.8667 (4)	0.5991 (3)	0.0452 (14)
H8A ^a	0.516326	0.880792	0.600546	0.054*
H8B ^a	0.696421	0.907992	0.556726	0.054*
C8A ^b	0.6552 (10)	0.8667 (4)	0.5991 (3)	0.0452 (14)
H8A1 ^b	0.766691	0.912014	0.584008	0.054*
H8A2 ^b	0.544331	0.884700	0.564437	0.054*
C9 ^a	0.7626 (11)	0.9157 (6)	0.6635 (4)	0.0329 (15)
H9A ^a	0.716419	0.999463	0.673590	0.040*
H9B ^a	0.900525	0.920881	0.656821	0.040*
C9A ^b	0.618 (3)	0.9090 (15)	0.6609 (9)	0.0329 (15)
H9A1 ^b	0.479167	0.903094	0.665195	0.040*
H9A2 ^b	0.655577	0.996890	0.664988	0.040*
C10	0.5717 (7)	0.8570 (5)	0.7643 (3)	0.0463 (15)
H10A	0.451329	0.846290	0.734245	0.069*
H10B	0.580435	0.941903	0.781950	0.069*
H10C	0.575326	0.799781	0.804255	0.069*
C11	0.9111 (8)	0.8581 (6)	0.7693 (4)	0.061 (2)
H11A	0.909442	0.812333	0.813678	0.092*
H11B	0.918916	0.946682	0.779435	0.092*
H11C	1.022378	0.832835	0.745468	0.092*
C12	0.7881 (7)	0.5785 (4)	0.8374 (2)	0.0257 (10)

^aOccupancy: 0.710 (9), ^bOccupancy: 0.290 (9).

Comment

Nickel represents an essential element for life and widely exists in a series of enzymes in organisms. It also plays an important role in maintaining the stability of membrane as well as the ultrastructure of cells [5]. Given the unique biological activity of nickel, recently nickel containing compounds have been subject of increasing interest in recent years [6, 7]. As a part of our current research interest on the exploration of the regulating effects of Schiff base ligands on transition metal complexes [8–10], herein we

report a new Ni(II) complex based on halogenated Schiff base ligand.

The molecular structure is shown in Figure. The complex is a mononuclear nickel complex constructed by a half-salen type Schiff base and the thiocyanate ligand. The nickel atom exhibits a square plane coordination configuration, which is completed by one phenolic oxygen atom and two N atoms from the half-salen type Schiff base ligand, along with a terminal N atom from thiocyanate. The bond lengths and angles are related to the distortion of the coordination. It is worth noting that the asymmetry of the diamines yields a difference between the bond length of Ni–N that come from the two N atoms of the half salen type Schiff base ligand. The bond length and bond angles range from 1.839(4) to 1.957(4) Å and 86.90(16) to 178.27(16)°, respectively. The bond lengths of Ni–N and Ni–O are consistent with those previously found in similar nickel (II) complexes. The linear thiocyanate ligand shows a tilted coordination mode with metal. The coordination modes of the N atom from the linear thiocyanate ligand is essentially identical to that seen in the half-salen ligand, and the bond length as well as bond angle are at the average level and shows no significant difference to the Schiff base ligand.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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