Crystal structure of bis(1-pyridin-2-yl-ethylidene)-*p*-tolylamine)nickel dithiocyanat, [Ni(C₁₄H₁₄N₂)₂](NCS)₂

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Abstract

C₃₀H₂₈N₆NiS₂, triclinic, $P\bar{1}$ (no. 2), $a = 9.9405(8)$ Å, $b = 11.641(1)$ Å, $c = 14.648(2)$ Å, $\alpha = 73.846(1)^\circ$, $\beta = 75.172(1)^\circ$, $\gamma = 77.102(2)^\circ$, $V = 1552.6$ Å³, $Z = 2$, $R_{\text{int}}(F) = 0.049$, $wR_{\text{ref}}(F^2) = 0.113$, $T = 298$ K.

Source of material

1-Pyridin-2-yl-ethanone (0.1 mmol, 12.1 mg), NH₄SCN (0.1 mmol, 7.6 mg), Ni(CH₃COO)₂ (0.1 mmol, 17.7 mg) and *p*-tolylamine (0.1 mmol, 10.7 mg) were dissolved in methanol (10 ml). The mixture was stirred for 30 min at room temperature to give a clear brown solution. After keeping this solution in air for 7 d, brown block-shaped crystals of the title compound were obtained on slow evaporation of the solvent. The crystals were collected, washed with methanol and dried in a vacuum desiccator using anhydrous CaCl₂ (yield 54 %).

Discussion

Schiff base complexes have been extensively investigated. Up to now it is still a hot research field, which could be attributed to their strong coordination ability and versatile coordination modes, antiseptic and antitumor activities [1,2] together with the special catalysis behavior and magnetism properties [3]. Schiff bases of pyridone and its complexes continue to be a subject of current interest. However, only limited Schiff bases of ketone have been reported up to now [4,5].

The crystal structure of the title compound is composed of a cation Ni²⁺, two Schiff base ligands and two SCN⁻ anion. The nickel atom is octahedrally coordinated by N1 and N2 from the Schiff base ligand, N3 and N4 from another Schiff base ligand and N5, N6 from two SCN⁻, which is in agreement with the rule of the op-

timal coordination number [6]. The bond length and bond angles of Schiff base ligands are in the normal ranges and in agreement with that of ketone Schiff base [7]. The C2=N2 and C16=N4 bond lengths of 1.281(4) and 1.268(4) Å are conform to a double bonds. The pyridine rings and phenyl rings are nearly planar and the mean deviations of the atoms from the least-squares planes A (N1/C3–C7), B (C8–C13), C (N3/C17–C21), D (C22–C27) are 0.006, 0.002, 0.011 and 0.006 Å, respectively. The methyl groups are slightly twisted out of benzene mean plane, atom C14 deviates from ring B by 0.049 Å, whereas C28 by 0.071 Å. The Schiff base ligands adopt a *trans* configuration about the C=N bond. To avoid steric conflicts, the two rigid ring systems of Schiff base ligand, pyridine ring and phenyl ring are rotated with respect to each other. Rings A, B and C, D are oriented at dihedral angles of $\angle A/B = 88.43(1)^\circ$ and $\angle C/D = 77.66(1)^\circ$.

Table 1. Data collection and handling.

Crystal:	brown block, size 0.40 × 0.41 × 0.45 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	7.88 cm ⁻¹
Diffractometer, scan mode:	Bruker SMART CCD, ϕ/ω
$2\theta_{\text{max}}$:	50.04°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	8083, 5381
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 3369
$N(\text{param})_{\text{refined}}$:	364
Programs:	SHELXS-97 [8], SHELXL-97 [9], SHELXTL [10]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(1A)	2i	0.2158	0.6869	0.4634	0.114
H(1B)	2i	0.3163	0.6160	0.5347	0.114
H(1C)	2i	0.1640	0.5859	0.5539	0.114
H(4)	2i	0.2781	0.4012	0.6329	0.076
H(5)	2i	0.3495	0.1982	0.6973	0.088
H(6)	2i	0.4702	0.0742	0.5957	0.083
H(7)	2i	0.5170	0.1541	0.4307	0.073
H(9)	2i	0.4117	0.7366	0.2862	0.090
H(10)	2i	0.3313	0.9154	0.1863	0.106
H(12)	2i	-0.0072	0.7861	0.1916	0.098
H(13)	2i	0.0688	0.6068	0.2932	0.079
H(14A)	2i	0.0791	0.9677	0.0566	0.198
H(14B)	2i	0.1777	1.0338	0.0854	0.198
H(14C)	2i	0.0219	1.0311	0.1436	0.198
H(15A)	2i	0.1394	0.2654	0.1281	0.143
H(15B)	2i	0.0167	0.3550	0.1753	0.143
H(15C)	2i	0.0720	0.2294	0.2391	0.143
H(18)	2i	0.1390	0.4850	0.0368	0.083
H(19)	2i	0.2312	0.6523	-0.0657	0.087
H(20)	2i	0.4097	0.7155	-0.0282	0.080

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Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(21)	2i	0.4958	0.6093	0.1063	0.068
H(23)	2i	0.0947	0.3231	0.4591	0.067
H(24)	2i	0.0527	0.1658	0.5916	0.079
H(26)	2i	0.2546	−0.0703	0.4311	0.083

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(27)	2i	0.3013	0.0879	0.2980	0.073
H(28A)	2i	0.1090	−0.1402	0.5955	0.141
H(28B)	2i	0.0436	−0.0469	0.6607	0.141
H(28C)	2i	0.2060	−0.0961	0.6435	0.141

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	Occ	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Ni(1)	2i		0.42345(5)	0.39670(4)	0.28585(3)	0.0562(3)	0.0515(3)	0.0390(3)	−0.0165(2)	−0.0070(2)	−0.0090(2)
S(1)	2i	0.68(1)	0.8243(6)	0.6027(8)	0.1623(4)	0.090(2)	0.124(4)	0.080(2)	−0.064(3)	0.014(2)	−0.037(3)
S(2)	2i	0.30(1)	0.705(1)	0.0564(8)	0.1730(7)	0.166(4)	0.058(1)	0.080(1)	−0.015(2)	0.016(2)	−0.0179(9)
S(1')	2i	0.32	0.850(1)	0.548(1)	0.1853(8)	0.090(2)	0.124(4)	0.080(2)	−0.064(3)	0.014(2)	−0.037(3)
S(2')	2i	0.70	0.6367(7)	0.0373(3)	0.1717(3)	0.166(4)	0.058(1)	0.080(1)	−0.015(2)	0.016(2)	−0.0179(9)
N(1)	2i		0.4235(3)	0.3205(3)	0.4321(2)	0.053(2)	0.053(2)	0.045(2)	−0.011(1)	−0.013(1)	−0.011(2)
N(2)	2i		0.3033(3)	0.5368(2)	0.3540(2)	0.055(2)	0.053(2)	0.049(2)	−0.013(1)	−0.018(1)	−0.011(1)
N(3)	2i		0.3698(3)	0.4884(2)	0.1535(2)	0.059(2)	0.051(2)	0.036(2)	−0.021(1)	−0.004(1)	−0.010(1)
N(4)	2i		0.2471(3)	0.3222(2)	0.2875(2)	0.057(2)	0.047(2)	0.035(2)	−0.017(1)	−0.003(1)	−0.006(1)
N(5)	2i		0.5987(3)	0.4755(3)	0.2587(2)	0.063(2)	0.078(2)	0.062(2)	−0.023(2)	−0.012(2)	−0.017(2)
N(6)	2i		0.5415(3)	0.2473(3)	0.2419(2)	0.067(2)	0.065(2)	0.054(2)	−0.015(2)	−0.006(2)	−0.013(2)
C(1)	2i		0.2438(4)	0.6097(3)	0.5046(3)	0.088(3)	0.084(3)	0.064(3)	−0.001(2)	−0.017(2)	−0.039(2)
C(2)	2i		0.2992(3)	0.5168(3)	0.4451(3)	0.052(2)	0.063(3)	0.051(2)	−0.012(2)	−0.015(2)	−0.019(2)
C(3)	2i		0.3535(3)	0.3917(3)	0.4929(2)	0.049(2)	0.064(2)	0.045(2)	−0.015(2)	−0.014(2)	−0.015(2)
C(4)	2i		0.3254(4)	0.3494(4)	0.5925(3)	0.076(3)	0.076(3)	0.044(2)	−0.017(2)	−0.016(2)	−0.016(2)
C(5)	2i		0.3688(4)	0.2293(4)	0.6304(3)	0.086(3)	0.089(3)	0.047(2)	−0.021(2)	−0.018(2)	−0.007(2)
C(6)	2i		0.4399(4)	0.1559(4)	0.5706(3)	0.081(3)	0.061(3)	0.060(3)	−0.016(2)	−0.026(2)	0.007(2)
C(7)	2i		0.4667(4)	0.2045(3)	0.4716(3)	0.063(2)	0.060(3)	0.059(2)	−0.009(2)	−0.014(2)	−0.011(2)
C(8)	2i		0.2486(4)	0.6533(3)	0.3000(3)	0.069(3)	0.057(3)	0.053(2)	−0.009(2)	−0.017(2)	−0.021(2)
C(9)	2i		0.3264(5)	0.7461(4)	0.2676(3)	0.091(3)	0.069(3)	0.079(3)	−0.023(2)	−0.037(3)	−0.011(2)
C(10)	2i		0.2782(5)	0.8529(4)	0.2078(3)	0.124(4)	0.058(3)	0.091(3)	−0.025(3)	−0.032(3)	−0.012(3)
C(11)	2i		0.1518(6)	0.8688(4)	0.1792(3)	0.121(4)	0.068(3)	0.070(3)	0.004(3)	−0.039(3)	−0.019(2)
C(12)	2i		0.0775(5)	0.7766(4)	0.2111(3)	0.089(3)	0.083(3)	0.076(3)	0.003(3)	−0.035(3)	−0.021(3)
C(13)	2i		0.1228(4)	0.6687(4)	0.2717(3)	0.071(3)	0.069(3)	0.066(3)	−0.012(2)	−0.020(2)	−0.021(2)
C(14)	2i		0.1031(6)	0.9862(4)	0.1098(4)	0.195(6)	0.082(4)	0.107(4)	0.017(4)	−0.066(4)	−0.004(3)
C(15)	2i		0.0986(5)	0.2952(4)	0.1856(3)	0.120(4)	0.107(4)	0.079(3)	−0.069(3)	−0.042(3)	0.007(3)
C(16)	2i		0.2048(4)	0.3516(3)	0.2082(2)	0.059(2)	0.058(2)	0.048(2)	−0.022(2)	−0.010(2)	−0.013(2)
C(17)	2i		0.2625(4)	0.4542(3)	0.1337(2)	0.066(2)	0.054(2)	0.035(2)	−0.016(2)	−0.006(2)	−0.011(2)
C(18)	2i		0.2106(4)	0.5123(4)	0.0510(3)	0.078(3)	0.085(3)	0.049(2)	−0.027(2)	−0.020(2)	−0.008(2)
C(19)	2i		0.2659(4)	0.6111(4)	−0.0101(3)	0.087(3)	0.083(3)	0.041(2)	−0.019(2)	−0.018(2)	0.004(2)
C(20)	2i		0.3719(4)	0.6479(3)	0.0117(3)	0.090(3)	0.064(3)	0.039(2)	−0.023(2)	−0.007(2)	0.002(2)
C(21)	2i		0.4218(4)	0.5845(3)	0.0927(2)	0.069(2)	0.056(2)	0.045(2)	−0.025(2)	−0.003(2)	−0.009(2)
C(22)	2i		0.2052(3)	0.2223(3)	0.3651(2)	0.051(2)	0.055(2)	0.048(2)	−0.022(2)	−0.005(2)	−0.010(2)
C(23)	2i		0.1289(3)	0.2438(3)	0.4532(2)	0.053(2)	0.060(2)	0.053(2)	−0.019(2)	−0.003(2)	−0.012(2)
C(24)	2i		0.1028(4)	0.1493(4)	0.5324(3)	0.062(2)	0.083(3)	0.050(2)	−0.029(2)	−0.001(2)	−0.008(2)
C(25)	2i		0.1490(4)	0.0309(4)	0.5261(3)	0.060(3)	0.069(3)	0.064(3)	−0.029(2)	−0.015(2)	0.003(2)
C(26)	2i		0.2231(4)	0.0092(3)	0.4373(3)	0.071(3)	0.054(2)	0.080(3)	−0.021(2)	−0.014(2)	−0.006(2)
C(27)	2i		0.2512(4)	0.1041(3)	0.3572(3)	0.065(2)	0.060(3)	0.059(2)	−0.021(2)	−0.002(2)	−0.016(2)
C(28)	2i		0.1247(4)	−0.0725(4)	0.6145(3)	0.089(3)	0.101(3)	0.077(3)	−0.041(3)	−0.021(3)	0.028(3)
C(29)	2i		0.6955(4)	0.5215(4)	0.2205(3)	0.067(3)	0.097(3)	0.056(3)	−0.030(2)	−0.007(2)	−0.038(2)
C(30)	2i		0.5891(4)	0.1620(4)	0.2140(3)	0.095(3)	0.055(3)	0.040(2)	−0.008(2)	0.000(2)	0.000(2)

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