Crystal structure of bis(dipentyldithiocarbamato)nickel(II), $Ni(C_{11}H_{22}NS_2)_2$

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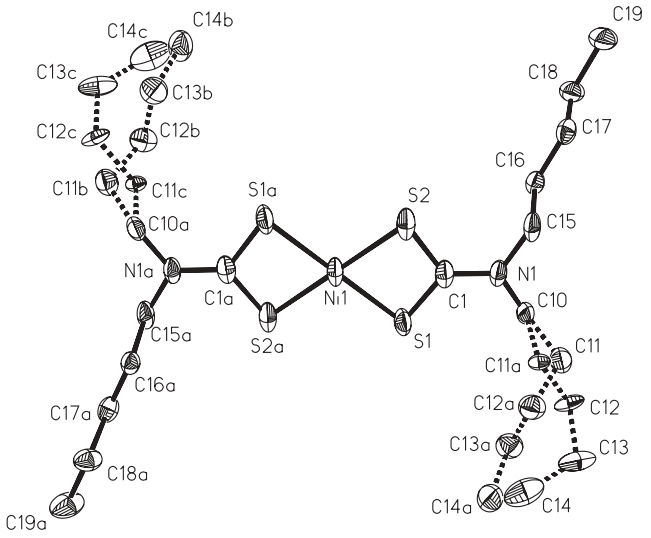

Abstract

$C_{22}H_{44}N_2NiS_4$, monoclinic, $P12_1/n1$ (No. 14), a = 10.402(2) Å, b = 13.261(3) Å, c = 10.701(2) Å, β = 114.79(3)°, V = 1340.1 Å^3, Z = 2, $R_{gt(F)}$ = 0.049, $wR_{ref(F^2)}$ = 0.118, T = 120 K.

Source of material

The title compound, a known species [1], was isolated as a green blocks by the slow evaporation of an *n*-hexane solution.

Discussion

From the figure, it is apparent that the central atom is four-coordinated. The NiS_4 chromophore is essentially planar and deviation of carbon atom from dithiocarbamate group plane is about 0.07 Å [2]. The Ni—S distances are 2.190(1) Å and 2.179(1) Å; the bond lengths of S1—C1 and S2—C1 are 1.700(4) Å and 1.705(4) Å, respectively. The N1—C1 distance is very short (1.315 Å) which is well known for the dithiocarbamate complexes [3]. The best convergence was reached for two positions for C11 to C14 atoms with occupating factors 0.5.

Table 1. Data collection and handling.

Crystal:	dark green prism, size 0.35 × 0.35 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	10.47 cm^{-1}
Diffractometer, scan mode:	KUMA KM-4, ω
$2\theta_{max}$:	57.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10565, 3120
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2199
$N(param)_{refined}$:	169
Programs:	PARST 95 [2], SHELXS-97 [4], SHELXL-97 [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(10B)	4*e*		0.6094	0.1738	0.4050	0.050
H(10A)	4*e*		0.7829	0.2365	0.4811	0.050
H(11C)	4*e*	0.50	0.6703	0.3770	0.3287	0.050
H(11B)	4*e*	0.50	0.7167	0.3650	0.3861	0.050
H(11A)	4*e*	0.50	0.5175	0.3065	0.2384	0.050
H(11D)	4*e*	0.50	0.6242	0.3668	0.2799	0.050
H(12D)	4*e*	0.50	0.6478	0.3632	0.5155	0.050
H(12A)	4*e*	0.50	0.5665	0.3223	0.4991	0.050
H(12C)	4*e*	0.50	0.5141	0.4403	0.2858	0.050
H(12B)	4*e*	0.50	0.4719	0.2861	0.3113	0.050
H(13D)	4*e*	0.50	0.2979	0.3851	0.3261	0.050
H(13C)	4*e*	0.50	0.3741	0.2443	0.3331	0.050
H(13A)	4*e*	0.50	0.4960	0.4937	0.3491	0.050
H(13B)	4*e*	0.50	0.4422	0.3343	0.4513	0.050
H(14A)	4*e*	0.50	0.4170	0.4094	0.3281	0.050
H(14B)	4*e*	0.50	0.2623	0.4124	0.1988	0.050
H(14E)	4*e*	0.50	0.1987	0.3593	0.1328	0.050
H(14C)	4*e*	0.50	0.2559	0.4710	0.2732	0.050
H(14D)	4*e*	0.50	0.3393	0.3666	0.2359	0.050
H(14F)	4*e*	0.50	0.1449	0.3475	0.2825	0.050
H(15B)	4*e*		0.9060	0.2762	0.3570	0.046
H(15A)	4*e*		0.8764	0.2098	0.2257	0.044
H(16B)	4*e*		0.9874	0.1374	0.5052	0.040
H(16A)	4*e*		0.9492	0.0684	0.3614	0.049
H(17B)	4*e*		1.1616	0.2272	0.4583	0.047
H(17A)	4*e*		1.1241	0.1512	0.3230	0.060
H(18B)	4*e*		1.2428	0.0926	0.6104	0.046
H(18A)	4*e*		1.1964	0.0032	0.4640	0.090
H(19C)	4*e*		1.4249	0.1640	0.5707	0.080
H(19B)	4*e*		1.3832	0.0966	0.4182	0.090
H(19A)	4*e*		1.4595	0.0454	0.5808	0.130

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni(1)	2*b*		1/2	0	0	0.0531(4)	0.0207(3)	0.0302(3)	–0.0019(2)	–0.0101(2)	–0.0002(2)
S(1)	4*e*		0.48692(9)	0.09220(6)	0.16405(9)	0.0483(5)	0.0439(5)	0.0435(5)	0.0003(4)	–0.0115(4)	–0.0137(4)
S(2)	4*e*		0.7045(1)	0.07592(6)	0.07825(8)	0.0608(5)	0.0303(4)	0.0334(4)	–0.0071(3)	–0.0032(4)	–0.0021(3)
N(1)	4*e*		0.7343(3)	0.1876(2)	0.2979(3)	0.048(2)	0.024(1)	0.039(1)	0.006(1)	–0.009(1)	–0.006(1)
C(1)	4*e*		0.6544(3)	0.1286(2)	0.1962(3)	0.051(2)	0.021(1)	0.038(2)	0.003(1)	–0.009(1)	0.002(1)
C(10)	4*e*		0.6883(3)	0.2242(3)	0.4006(4)	0.034(2)	0.048(2)	0.064(2)	0.010(2)	–0.013(2)	–0.028(2)
C(11)	4*e*	0.50	0.6396(8)	0.3263(5)	0.4172(9)	0.045(4)	0.044(4)	0.045(4)	–0.004(3)	0.010(4)	–0.001(4)
C(11A)	4*e*	0.50	0.6066(6)	0.3240(4)	0.3317(7)	0.028(3)	0.028(3)	0.026(3)	0.008(2)	0.017(3)	0.008(2)
C(12)	4*e*	0.50	0.5597(7)	0.3669(5)	0.4406(6)	0.055(4)	0.045(3)	0.039(3)	0.027(3)	0.035(3)	0.010(3)
C(12A)	4*e*	0.50	0.5016(7)	0.3562(6)	0.3078(7)	0.047(4)	0.049(4)	0.062(5)	0.001(3)	0.025(4)	0.005(3)
C(13)	4*e*	0.50	0.3772(8)	0.3265(6)	0.3434(7)	0.054(4)	0.053(4)	0.049(4)	0.007(3)	0.016(3)	0.007(3)
C(13A)	4*e*	0.50	0.4517(9)	0.4486(6)	0.3915(9)	0.069(5)	0.047(4)	0.103(7)	0.028(4)	0.062(5)	0.030(4)
C(14)	4*e*	0.50	0.245(1)	0.3735(7)	0.248(1)	0.064(6)	0.061(6)	0.049(5)	0.013(4)	0.005(4)	–0.006(4)
C(14A)	4*e*	0.50	0.310(1)	0.4165(8)	0.289(1)	0.11(1)	0.053(6)	0.095(9)	0.027(6)	0.061(8)	0.022(6)
C(15)	4*e*		0.8785(3)	0.2106(2)	0.3182(3)	0.054(2)	0.022(1)	0.039(2)	–0.005(1)	–0.003(1)	–0.003(1)
C(16)	4*e*		0.9830(3)	0.1346(2)	0.4104(3)	0.041(2)	0.027(1)	0.030(1)	0.000(1)	0.009(1)	–0.001(1)
C(17)	4*e*		1.1298(3)	0.1534(2)	0.4214(3)	0.053(2)	0.030(2)	0.042(2)	–0.010(1)	0.024(2)	–0.006(1)
C(18)	4*e*		1.2381(3)	0.0829(2)	0.5199(4)	0.041(2)	0.042(2)	0.071(2)	–0.001(1)	0.031(2)	–0.001(2)
C(19)	4*e*		1.3848(4)	0.1014(3)	0.5291(5)	0.056(2)	0.062(2)	0.127(4)	–0.010(2)	0.060(3)	–0.020(3)

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