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Crystal structure of the tetranickel complex with ^{i}propanethiolate

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ABSTRACT

The title complex $[Ni_4(SPr^i)_8]$ was confirmed by single crystal X-ray crystal diffraction analysis. The crystals are monoclinic, space group P2/n with a = *12.760(3)*, b = *10.059(2)*, c = *14.484(3)* Å, $\alpha = 90$, $\beta = 93.70(3)$, $\gamma = 90°$, $V = 1855.3(6)$ *Å*^{3}, $Z = 4$, $F(000) = 880$, $D_c = 1.496$ *g/cm*^{3}, $\mu = 2.463$ *mm*$^{-1}$, *the final R = 0.0352 and wR = 0.0580*. A total of 18,588 reflections were collected, of which 4404 were independent ($R_{int} = 0.0631$). In the crystal packing diagram, intermolecular C—H···Ni hydrogen bonds stabilize the solid state of the title complex.

KEYWORDS

nickel; thiolate; crystal structure

Introduction

Our previous studies revealed that a nickel dichloride complex with 1,2-bis(diphenylphosphino)ethane (DPPE) was synthesized and characterized by X-ray crystallography [1]. During our recent research, we unexpectedly obtained the crystal structure of the title complex $[Ni_4(SPr^i)_8]$ by the reaction of $Ni(dppe)Cl_2$ with ^{i}PrSH in the presence of Et_3N. And its structure was confirmed by single crystal X-ray diffraction analysis. In this paper, we describe the structural characterization of the tetranickel complex $[Ni_4(SPr^i)_8]$.

Experimental

Crystal structure determination: The crystal of the title complex with dimensions of 0.20 mm × 0.18 mm × 0.12 mm was mounted on a Rigaku Saturn CCD area detector diffractometer with a graphite-monochromated MoKα radiation ($\lambda = 0.71073$Å) by using a phi and scan modes at 113(2) K in the range of $2.02° \le \theta \le 27.92°$. The crystal belongs to monoclinic system with space group P2/*n* and crystal parameters of $a = 12.760(3)$ Å, $b = 10.059(2)$ Å, $c = 14.484(3)$ Å, $\alpha = 90°$, $\beta = 93.70(3)°$, $\gamma = 90°$, $V = 1855.3(6)$ A^3, $D_c = 1.496$ g/cm^3. The absorption coefficient $\mu = 2.463$ mm^{-1}, and $Z = 4$. A summary of crystal data is presented in Table 1.

The structure was solved by direct methods with SHELXS-97 [2] and refined by the full-matrix least squares method on F^2 data using SHELXL-97 [3]. The empirical absorption corrections were applied to all intensity data. H atom of N-H was initially located in a difference

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Table 1. Crystal data and structure refinement.

Empirical formula	$C_{12}H_{28}Ni_2S_4$
Formula weight	418.00
Crystal system	Monoclinic
Unit cell dimensions	
a (Å)	12.760(3)
b (Å)	10.059(2)
c (Å)	14.484(3)
Unit cell angles (ˆ)	
α	90
β	93.70(3)
γ	90
Volume (Å^3)	1855.3(6)
Z	4
Temperature (K)	113(2)
space group	P2/*n*
Wavelength (Å)	0.71073
Calculated density (g/cm^3)	1.496
Absorption coefficient (mm^{-1})	2.463
F(000)	880
Crystal size (mm)	0.20 × 0.18 × 0.12
Theta range for data collection (°)	2.02–27.92
Reflections collected	18588
Independent reflections	4404 [$R_{(int)} = 0.0631$]
Final *R* indices [I > 2σ (I)]	$R_1 = 0.0352$, w$R_2 = 0.0580$

Fourier map and were refined with the restraint Uiso(H) = 1.2Ueq(N). Other H atoms were positioned geometrically and refined using a riding model, with d(C–H) = 0.93–0.97 Å and Uiso(H) = 1.2Ueq(C) or 1.5Ueq(Cmethyl). The final full-matrix least squares refinement gave $R = 0.0352$ and $wR = 0.0580$.

Results and discussion

The title complex [$Ni_4(SPr^i)_8$] was confirmed by single crystal X-ray diffraction analysis. The selected bond lengths and bond angles are listed in Table 2. The structure was solved by direct methods. Anisotropic displacement parameters were applied to all nonhydrogen atoms in

Table 2. Selected bond lengths (Å) and bond angles (°).

Bond lengths			
Ni(1)-S(4)	2.2043(8)	Ni(1)-S(2)	2.2047(8)
Ni(1)-S(1)	2.2241(8)	Ni(1)-S(3)	2.2261(8)
Ni(1)-Ni(2)	2.6784(6)	Ni(1)-Ni(3)	2.6851(6)
Ni(2)-S(2)	2.2129(8)	Ni(2)-S(1)	2.2131(8)
Ni(3)-S(4)	2.2093(7)	Ni(3)-S(3)	2.2100(8)
Ni(3)-Ni(1)A	2.6851(6)	S(1)-C(1)	1.853(2)
S(2)-C(4)	1.837(2)	S(3)-C(7)	1.848(2)
S(4)-C(10)	1.833(2)	C(1)-C(2)	1.520(3)
C(1)-C(3)	1.521(3)	C(4)-C(5)	1.522(3)
Bond angles			
S(4)-Ni(1)-S(2)	174.67(3)	S(4)-Ni(1)-S(1)	98.23(3)
S(2)-Ni(1)-S(1)	80.68(3)	S(4)-Ni(1)-S(3)	80.64(3)
Ni(2)-Ni(1)-Ni(3)	89.20(2)	S(2)-Ni(2)-S(1)	80.75(4)
Ni(1)A-Ni(2)-Ni(1)	90.95(3)	S(4)-Ni(3)-S(3)	80.88(4)
Ni(1)-Ni(3)-Ni(1)A	90.66(3)	Ni(2)-S(1)-Ni(1)	74.26(3)
Ni(1)-S(2)-Ni(2)	74.64(2)	Ni(3)-S(3)-Ni(1)	74.50(3)
Ni(1)-S(4)-Ni(3)	74.94(3)	C(2)-C(1)-C(3)	111.7(2)
C(2)-C(1)-S(1)	108.58(18)	C(3)-C(1)-S(1)	109.77(18)

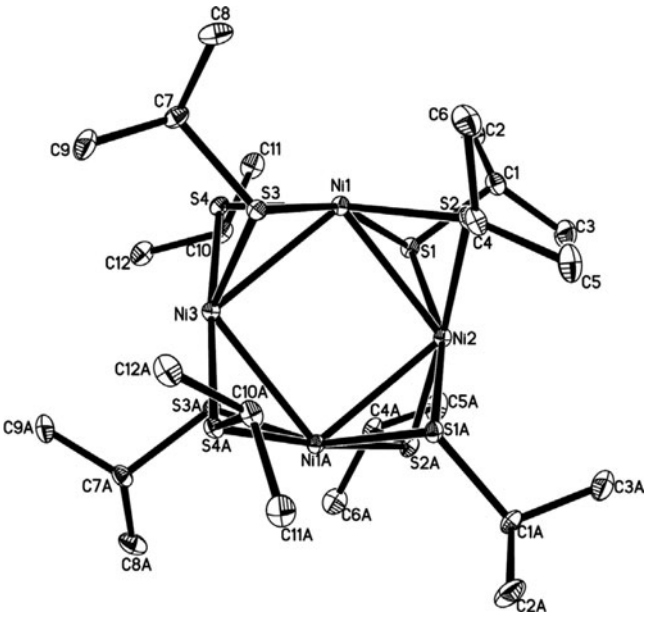

Figure 1. Molecular structure of the title complex.

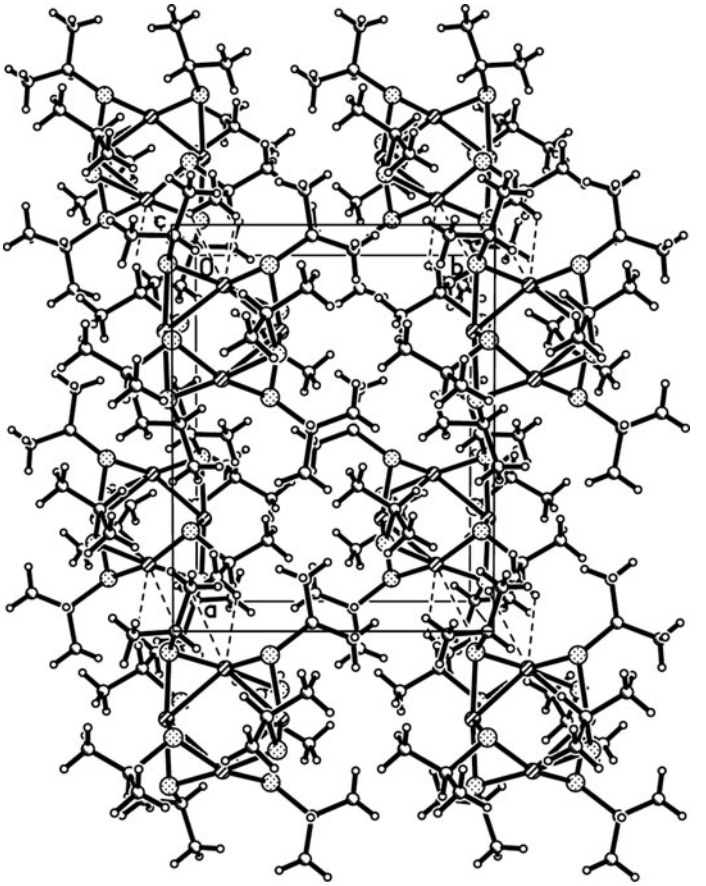

Figure 2. The crystal packing view of the title complex.

full-matrix least-square refinements based on F^2. The hydrogen atoms were set in calculated positions with a common fixed isotropic thermal parameter.

The molecular structure and packing views of the title complex are shown in Figures 1 and 2, respectively. The title complex crystallizes in monoclinic space group P2/*n* with four molecules in the unit cell and one molecule in the asymmetric unit. As shown in Figure 1, the molecular structure contains a square tetranickel backbone with eight ^{i}propanethiolate ligands. Each nickel coordinates with four sulfur of the ^{i}propanethiolate. The four-membered ring $Ni_1Ni_2Ni_1ANi_3$ is coplanar with the mean deviation of 0. The bond distances [Ni(1)-S(4) = 2.2043(8) Å, Ni(1)-S(2) = 2.2047(8) Å, Ni(1)-S(1) = 2.2241(8) Å, Ni(1)-S(3) = 2.2261(8) Å, Ni(1)-Ni(2) = 2.6784(6) Å, Ni(1)-Ni(3) = 2.6851(6) Å, Ni(2)-S(2) = 2.2129(8) Å, Ni(2)-S(1) = 2.2131(8) Å, Ni(3)-S(4) = 2.2093(7) Å, S(1)-C(1) = 1.853(2) Å and C(1)-C(2) = 1.520(3) Å] and bond angles [S(4)-Ni(1)-S(2) = 174.67(3)°, S(4)-Ni(1)-S(1) = 98.23(3)°, S(2)-Ni(1)-S(1) = 80.68(3)°, Ni(2)-Ni(1)-Ni(3) = 89.20(2)°, S(2)-Ni(2)-S(1) = 80.75(4)°, Ni(1)A-Ni(2)-Ni(1) = 90.95(3)°, S(4)-Ni(3)-S(3) = 80.88(4)° and Ni(1)-Ni(3)-Ni(1)A = 90.66(3)°] are similar to analogous complexes [4-29].

As shown in Figure 2, the crystal packing diagram of the title complex displays that intermolecular C—H···Ni hydrogen bonds existing to stabilize the solid state.

Conclusions

In summary, the title complex $[Ni_4(SPr^i)_8]$ has been characterized by X-ray diffraction analysis.

Acknowledgments

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Supplementary Information

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication number CCDC 1036539 for the title complex. Copies of the data can be obtained free of charge at http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk.

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