Inorganic Chemistry
including bioinorganic chemistry
Inorg. Chem., 1996, 35(17), 4989-4994, DOI:10.1021/ic9601044

Summary of Structure Determination and Solution Refinement. A blue crystal of [5]I was mounted on a glass fiber with epoxy cement, at room temperature and cooled to 193 K in a N_2 cold stream. Cell parameters were calculated from the least-squares fitting of the setting angles for 25 reflections ($2\theta_{avg}$=23.3). Lorentz and polarization corrections were applied to 3726 reflections. A semi-empirical absorption correction was applied. All crystallographic calculations were performed on a Nicolet R3m single-crystal X-ray diffractometer using graphite-monochromated MoKa radiation and P3VAX3.42 software supplied by Nicolet Analytical X-ray Instruments, Madison, WI. A total of 2013 unique reflections were used in further calculations. The molecular structure was solved by Direct Methods and refined anisotropically for all non-hydrogen atoms with full-matrix least-squares method. Direct methods program used was SHELXS, SHEXTL-PLUS. Hydrogen atoms were placed in idealized positions with isotropic thermal parameters fixed at 0.08 $Å^2$. Neutral atom scattering factors and anomalous scattering correction terms were taken from a standard source (Ibers, J.A.; Hamilton, W.C. *International Tables for X-Ray Crystallography* Kynoch Press, Birmingham, England, 1974, Vol. 4, pp 99, 149).

Table IS. Summary of Crystallographic Data for $[(tbtp\text{-}daco)Ni^{II}][I]\cdot H_2O$

Complex	$[(tbtp\text{-}daco)Ni^{II}][I]\cdot H_2O$
Molecular formula	$C_{13}H_{27}N_2O_3S_2NiI$
Formula weight (g/mol)	509.1
Crystal size (mm)	0.1x0.22x0.23
Space group	orthorhombic
	Pbca(No.61)
a, (Å)	10.898(3)
b, (Å)	18.103(5)
c, (Å)	19.020(5)
V, (Å^3)	3752(2)
Z	8
ρ (calcd), (g cm^{-3})	1.802
Temp. (K)	193
Radiation	Mo-Kα (λ=0.71073Å)
Min./Max. transmission coeff.	0.7727/0.9660
No. reflections collected	3726
Observed reflections	2010
	$\lvert F\rvert \geq 4.0\sigma F$
Unique reflections[a]	1728 (R_{int} = 4.41%)
μ, (mm^{-1})	2.892
Index ranges	$0 \leq h \leq 12$
	$-21 \leq k \leq 0$
	$0 \leq l \leq 22$
Data to parameter ratio	10.0:1
R (%)[a]	5.3
wR (%)[a]	5.5
S[a]	1.47
Largest, mean Δ/σ final	0.0023, 0.0004
Largest pos., neg. peak (e$^-$Å^3)	0.68, -0.84
g ($w^{-1} = (\sigma F)^2 + gF^2$)	0.0003

[a] Residuals: $R_{int} = [\Sigma F^2-(F_{mean})^2] / [\Sigma F^2]$; $R = \Sigma|F_o-F_c| / \Sigma F_o$; $wR = \{[\Sigma w(F_o-F_c)^2] / [\Sigma w(F_o)^2]\}^{1/2}$; $S = \{[\Sigma w(F_o-F_c)^2] / [N_{data}-N_{parameters}]\}^{1/2}$

Table IIS. Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2x10^3$) for [(tbtp-daco)Ni^{II}][I]•H_2O, [5]I.

	x	y	z	U(eq)[a,b]
I(1)	1409(1)	313(1)	1649(1)	55(1)
Ni(1)	3176(1)	-2474(1)	4512(1)	20(1)
S(1)	2700(2)	-2569(2)	5742(1)	28(1)
S(2)	2648(3)	-1170(1)	4418(1)	31(1)
O(1)	4945(5)	-2298(3)	4858(3)	23(2)
O(2)	6306(5)	-2433(4)	5731(3)	28(2)
O(3)	4533(8)	-514(4)	1912(5)	68(4)
N(1)	3539(7)	-3618(4)	4473(4)	29(3)
N(2)	3549(7)	-2480(5)	3429(4)	33(3)
C(1)	5235(7)	-2387(5)	5485(5)	18(3)
C(2)	4257(8)	-2454(6)	6065(5)	28(3)
C(3)	2539(11)	-3566(5)	5685(6)	40(4)
C(4)	3503(10)	-3917(5)	5211(5)	35(4)
C(5)	4766(10)	-3813(6)	4183(6)	41(4)
C(6)	5041(10)	-3576(6)	3423(6)	45(4)
C(7)	4832(10)	-2759(6)	3277(6)	40(4)
C(8)	2543(11)	-3973(6)	4067(6)	44(4)
C(9)	2339(10)	-3723(6)	3303(6)	46(4)
C(10)	2596(10)	-2923(7)	3042(5)	44(4)
C(11)	3542(11)	-1705(6)	3153(5)	45(4)
C(12)	2565(12)	-1228(6)	3461(5)	47(4)
C(13)	4059(10)	-662(6)	4556(7)	46(4)

[a] U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

[b] Estimated standard deviations are given in parenthesis.

Table IIIS. Complete list of bond lengths (Å)[a] for [(tbtp-daco)Ni^{II}][I]•H_2O, [5]I.

Ni(1)-S(1)	2.402(3)	Ni(1)-S(2)	2.438(3)
Ni(1)-O(1)	2.062(6)	Ni(1)-N(1)	2.108(8)
Ni(1)-N(2)	2.100(8)	Ni(1)-O(2A)	2.096(6)
S(1)-C(2)	1.817(9)	S(1)-C(3)	1.82 (1)
S(2)-C(12)	1.83 (1)	S(2)-C(13)	1.81 (1)
O(1)-C(1)	1.24 (1)	O(2)-C(1)	1.26 (1)
O(2)-Ni(1A)	2.096(6)	N(1)-C(4)	1.50 (1)
N(1)-C(5)	1.49 (1)	N(1)-C(8)	1.48 (1)
N(2)-C(7)	1.51 (1)	N(2)-C(10)	1.50 (1)
N(2)-C(11)	1.50 (1)	C(1)-C(2)	1.54 (1)
C(3)-C(4)	1.52 (1)	C(5)-C(6)	1.54 (2)
C(6)-C(7)	1.52 (2)	C(8)-C(9)	1.54 (2)
C(9)-C(10)	1.55 (2)	C(11)-C(12)	1.49 (2)

[a] Estimated standard deviations are given in parenthesis.

Table IVS. Complete list of bond angles (deg.)[a] for [(tbtp-daco)Ni^{II}][I]•H_2O, [5]I.

S(1)-Ni(1)-S(2)	95.1(1)	S(1)-Ni(1)-O(1)	84.4(2)
S(2)-Ni(1)-O(1)	95.4(2)	S(1)-Ni(1)-N(1)	90.3(2)
S(2)-Ni(1)-N(1)	173.2(2)	O(1)-Ni(1)-N(1)	89.3(3)
S(1)-Ni(1)-N(2)	175.5(3)	S(2)-Ni(1)-N(2)	88.8(2)
O(1)-Ni(1)-N(2)	97.7(3)	N(1)-Ni(1)-N(2)	85.7(3)
S(1)-Ni(1)-O(2A)	89.9(2)	S(2)-Ni(1)-O(2A)	80.3(2)
O(1)-Ni(1)-O(2A)	172.5(2)	N(1)-Ni(1)-O(2A)	95.5(3)
N(2)-Ni(1)-O(2A)	88.4(3)	Ni(1)-S(1)-C(2)	96.9(3)
Ni(1)-S(1)-C(3)	91.9(3)	C(2)-S(1)-C(3)	102.9(5)
Ni(1)-S(2)-C(12)	91.6(4)	Ni(1)-S(2)-C(13)	106.3(4)
C(12)-S(2)-C(13)	102.4(6)	Ni(1)-O(1)-C(1)	121.6(5)
C(1)-O(2)-Ni(1A)	145.5(6)	Ni(1)-N(1)-C(5)	108.5(6)
Ni(1)-N(1)-C(4)	114.5(6)	C(4)-N(1)-C(5)	106.5(8)
Ni(1)-N(1)-C(8)	107.9(6)	C(5)-N(1)-C(8)	108.1(8)
C(5)-N(1)-C(8)	111.3(8)	Ni(1)-N(2)-C(7)	111.5(6)
Ni(1)-N(2)-C(10)	110.3(6)	C(7)-N(2)-C(10)	111.5(8)
Ni(1)-N(2)-C(11)	109.7(6)	C(7)-N(2)-C(11)	104.6(8)
C(10)-N(2)-C(11)	109.0(8)	O(1)-C(1)-O(2)	126.9(8)
O(1)-C(1)-C(2)	121.4(7)	O(2)-C(1)-C(2)	111.7(8)
S(1)-C(2)-C(1)	114.4(6)	S(1)-C(3)-C(4)	112.6(7)
N(1)-C(4)-C(3)	114.8(8)	N(1)-C(5)-C(6)	117.2(9)
C(5)-C(6)-C(7)	114.4(9)	N(2)-C(7)-C(6)	115.4(9)
N(1)-C(8)-C(9)	118.1(9)	C(8)-C(9)-C(10)	123.2(9)
N(2)-C(10)-C(9)	117.8(9)	N(2)-C(11)-C(12)	114.1(9)
S(2)-C(12)-C(11)	113.0(8)		

[a] Estimated standard deviations are given in parenthesis.

Table VS. Anisotropic displacement parameters ($Å^2x10^3$)[a,b] for [(tbtp-daco)Ni^{II}][I]•H_2O, [5]I.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
I(1)	54(1)	56(1)	56(1)	-28(1)	-11(1)	7(1)
Ni(1)	16(1)	22(1)	23(1)	1(1)	0(1)	-1(1)
S(1)	19(1)	38(2)	27(1)	5(1)	2(1)	0(1)
S(2)	23(1)	27(1)	44(2)	9(1)	4(1)	1(1)
O(1)	13(3)	33(4)	22(4)	1(3)	1(3)	-1(3)
O(2)	16(3)	35(4)	34(4)	-1(3)	-10(3)	0(4)
O(3)	61(6)	46(5)	97(7)	11(5)	5(5)	2(5)
N(1)	21(5)	26(4)	42(5)	-7(4)	2(5)	2(4)
N(2)	25(5)	47(5)	27(4)	1(4)	0(4)	-2(5)
C(1)	8(5)	19(5)	28(5)	-4(5)	9(4)	-3(4)
C(2)	18(5)	44(6)	22(5)	8(5)	3(4)	5(6)
C(3)	42(7)	35(6)	43(6)	10(5)	4(6)	-12(6)
C(4)	27(6)	25(6)	53(7)	2(5)	-11(6)	-1(5)
C(5)	29(7)	28(6)	66(8)	-16(6)	-2(6)	13(5)
C(6)	22(6)	62(8)	50(8)	-19(7)	-11(6)	9(6)
C(7)	33(7)	53(8)	34(6)	2(6)	12(6)	3(6)
C(8)	31(7)	39(7)	61(8)	-14(6)	-2(6)	-5(6)
C(9)	28(6)	57(8)	52(7)	-22(7)	-3(7)	-3(6)
C(10)	23(6)	76(9)	35(6)	-20(6)	5(6)	-2(6)
C(11)	49(8)	61(8)	25(6)	21(5)	6(6)	4(7)
C(12)	61(8)	44(7)	35(7)	21(5)	-4(7)	15(7)
C(13)	30(6)	26(6)	82(9)	15(6)	4(7)	-2(5)

[a]The anisotropic displacement exponent takes the form:

$-2\pi^2(h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12})$

[b]Estimated standard deviations are given in parenthesis.

Table VIS. H-atom coordinates ($x10^4$) and isotropic displacement parameters ($Å^2x10^3$) for $[(tbtp\text{-}daco)Ni^{II}][I]\cdot H_2O$, [5]I.

	x	y	z	U_{iso}
H(1O3)	3835	-392	1749	80
H(2O3)	4588	-981	1948	80
H(2A)	4443	-2873	6355	80
H(2B)	4279	-2022	6357	80
H(3A)	1734	-3681	5512	80
H(3B)	2609	-3772	6148	80
H(4A)	4298	-3865	-5422	80
H(4B)	3324	-4436	5181	80
H(5A)	4837	-4341	4204	80
H(5B)	5389	-3611	4485	80
H(6A)	4533	-3853	3107	80
H(6B)	5880	-3695	3319	80
H(7A)	5399	-2485	2794	80
H(7B)	5025	-2658	2794	80
H(8A)	1787	-3909	4320	80
H(8B)	2717	-4493	4059	80
H(9A)	1493	-3823	3198	80
H(9B)	2815	-4046	3010	80
H(10A)	2831	-2954	2557	80
H(10B)	1836	-2655	3060	80
H(11A)	3448	-1715	2652	80
H(11B)	4325	-1486	3254	80
H(12A)	2644	-739	3271	80
H(12B)	1777	-1417	3326	80
H(13A)	3887	-143	4523	80
H(13B)	4390	-771	5012	80
H(13C)	4644	-798	4201	80

Table VIIS. Torsion angles (°)[a] less those to Hydrogen atoms for [(tbtp-daco)Ni^{II}][I]•H_2O, [5]I.

S(2)-Ni(1)-S(1)-C(2)	-97.2(4)	S(2)-Ni(1)-S(1)-C(3)	159.5(4)
O(1)-Ni(1)-S(1)-C(2)	-2.3(4)	O(1)-Ni(1)-S(1)-C(3)	-105.5(4)
N(1)-Ni(1)-S(1)-C(2)	87.0(4)	N(1)-Ni(1)-S(1)-C(3)	-16.3(5)
N(2)-Ni(1)-S(1)-C(2)	115(3)	N(2)-Ni(1)-S(1)-C(3)	11(3)
O(2A)-Ni(1)-S(1)C(2)	-177.5(4)	O(2A)-Ni(1)-S(1)-C(3)	79.2(4)
S(1)-Ni(1)-S(2)-C(12)	-163.3(4)	S(1)-Ni(1)-S(2)-C(13)	93.2(4)
O(1)-Ni(1)-S(2)-C(12)	111.9(4)	O(1)-Ni(1)-S(2)-C(13)	8.4(5)
N(1)-Ni(1)-S(2)-C(12)	-22(2)	N(1)-Ni(1)-S(2)-C(13)	-125(2)
N(2)-Ni(1)-S(2)-C(12)	14.3(5)	N(2)-Ni(1)-S(2)-C(13)	-89.2(5)
O(2A)-Ni(1)-S(2)-C(12)	-74.3(4)	O(2A)-Ni(1)-S(2)-C(13)	-177.7(5)
S(1)-Ni(1)-O(1)-C(1)	9.5(6)	S(2)-Ni(1)-O(1)-C(1)	104.1(6)
N(1)-Ni(1)-O(1)-C(1)	-80.8(7)	N(2)-Ni(1)-O(1)-C(1)	-166.4(7)
O(2A)-Ni(1)-O(1)-C(1)	50(2)	S(1)-Ni(1)-N(1)-C(5)	-7.4(6)
S(1)-Ni(1)-N(1)-C(5)	-126.1(6)	S(1)-Ni(1)-N(1)-C(8)	109.5(6)
S(2)-Ni(1)-N(1)-C(5)	-149.3(17)	S(2)-Ni(1)-N(1)-C(5)	92(2)
S(2)-Ni(1)-N(1)-C(8)	-33(2)	O(1)-Ni(1)-N(1)-C(5)	77.0(6)
O(1)-Ni(1)-N(1)-C(5)	-41.7(7)	O(1)-Ni(1)-N(1)-C(8)	-166.2(6)
N(2)-Ni(1)-N(1)-C(5)	174.7(6)	N(2)-Ni(1)-N(1)-C(5)	56.0(7)
N(2)-Ni(1)-N(1)-C(8)	-68.4(6)	O(2A)-Ni(1)-N(1)-C(5)	-97.3(6)
O(2A)-Ni(1)-N(1)-C(5)	144.0(7)	O(2A)-Ni(1)-N(1)-C(8)	19.5(6)
S(1)-Ni(1)-N(2)-C(7)	-86(3)	S(1)-Ni(1)-N(2)-C(10)	38(3)
S(1)-Ni(1)-N(2)-C(11)	158(3)	S(2)-Ni(1)-N(2)-C(7)	125.2(6)
S(2)-Ni(1)-N(2)-C(10)	-110.4(6)	S(2)-Ni(1)-N(2)-C(11)	9.8(6)
O(1)-Ni(1)-N(2)-C(7)	29.9(7)	O(1)-Ni(1)-N(2)-C(10)	154.4(6)
O(1)-Ni(1)-N(2)-C(11)	-85.5(6)	N(1)-Ni(1)-N(2)-C(7)	-58.8(7)
N(1)-Ni(1)-N(2)-C(10)	65.6(7)	O(2A)-Ni(1)-N(2)-C(7)	-154.4(6)
N(1)-Ni(1)-N(2)-C(11)	-174.2(7)	O(2A)-Ni(1)-N(2)-C(11)	90.2(6)
O(2A)-Ni(1)-N(2)-C(10)	-30.0(7)	C(3)-S(1)-C(2)-C(1)	90.6(8)
Ni(1)-S(1)-C(2)-C(1)	-3.0(7)	C(2)-S(1)-C(3)-C(5)	-57.6(8)
Ni(1)-S(1)-C(3)-C(5)	39.9(7)	C(13)-S(2)-C(12)-C(11)	67.8(9)
Ni(1)-S(2)-C(12)-C(11)	-39.3(8)	NI(1)-O(1)-C(1)-C(2)	-14.7(11)
Ni(1)-O(1)-C(1)-O(2)	165.6(7)	Ni(1A)-O(2)-C(1)-C(2)	171.0(8)

Ni(1A)-O(2)-C(1)-O(1)	-9.3(17)	C(5)-N(1)-C(5)-C(3)	160.1(8)
Ni(1)-N(1)-C(5)-C(3)	36.4(10)	Ni(1)-N(1)-C(5)-C(6)	-60.8(10)
C(8)-N(1)-C(5)-C(3)	-80.3(10)	C(8)-N(1)-C(5)-C(6)	61.8(11)
C(5)-N(1)-C(5)-C(6)	179.3(8)	C(5)-N(1)-C(8)-C(9)	176.5(9)
Ni(1)-N(1)-C(8)-C(9)	59.4(10)	Ni(1)-N(2)-C(7)-C(6)	68.9(10)
C(5)-N(1)-C(8)-C(9)	-67.0(11)	C(11)-N(2)-C(7)-C(6)	-172.6(8)
C(10)-N(2)-C(7)-C(6)	-55.0(11)	C(7)-N(2)-C(10)-C(9)	72.0(11)
Ni(1)-N(2)-C(10)-C(9)	-52.5(10)	Ni(1)-N(2)-C(11)-C(12)	-39.3(10)
C(11)-N(2)-C(10)-C(9)	-173.1(9)	C(10)-N(2)-C(11)-C(12)	81.7(10)
C(7)-N(2)-C(11)-C(12)	-159.0(9)	O(2)-C(1)-C(2)-S(1)	-169.0(7)
O(1)-C(1)-C(2)-S(1)	11.3(12)	N(1)-C(5)-C(6)-C(7)	53.0(12)
S(1)-C(3)-C(5)-N(1)	-56.2(10)	N(1)-C(8)-C(9)-C(10)	-31.5(15)
C(5)-C(6)-C(7)-N(2)	-57.4(12)	N(2)-C(11)-C(12)-S(2)	57.3(11)
C(8)-C(9)-C(10)-N(2)	27.0(15)		

[a]Estimated standard deviations are given in parenthesis.

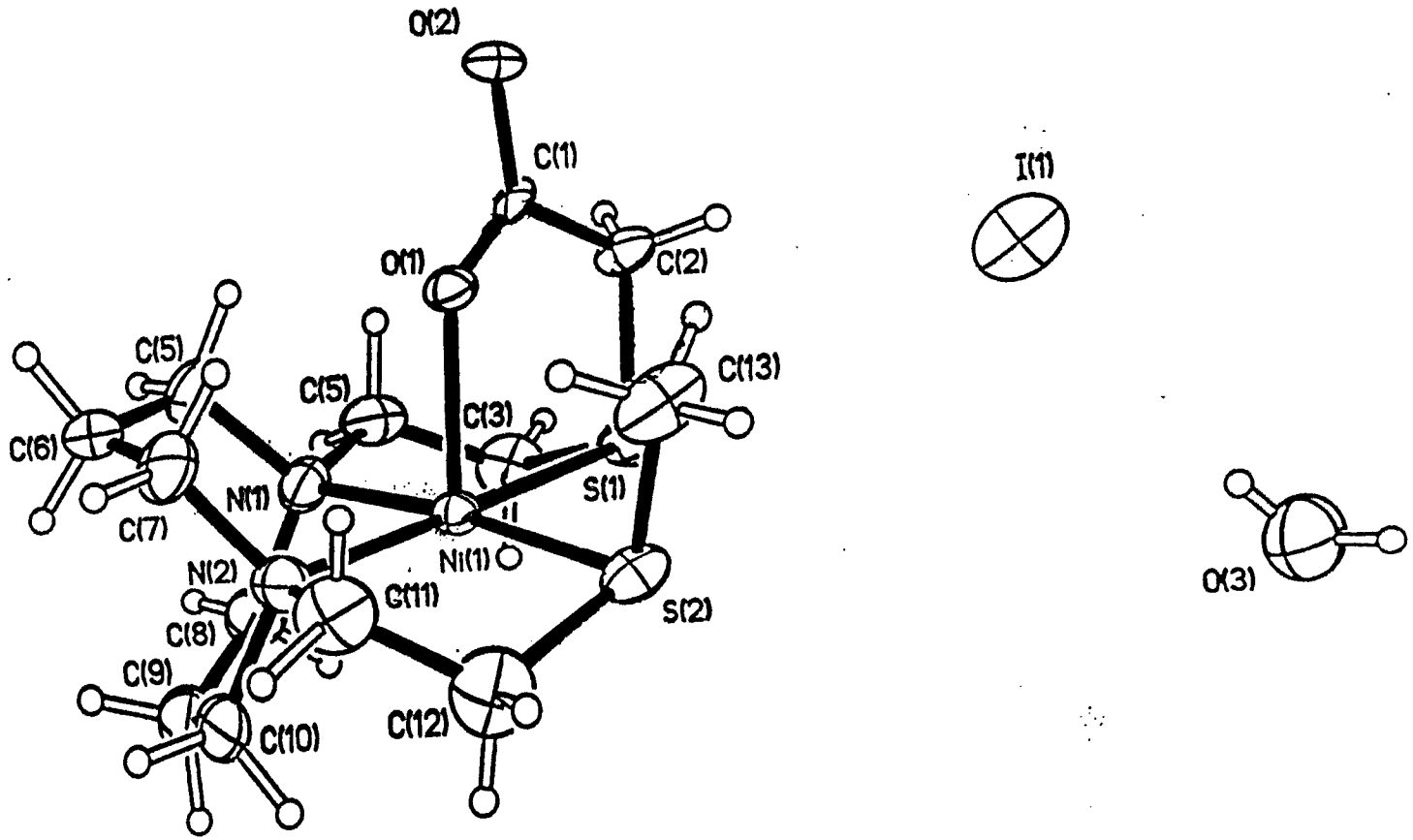

Figure IS. Crystal structure of [(tbtp-daco)Ni^{II}][I]•H_2O, [5]I (thermal ellipsoids at 50% probability)

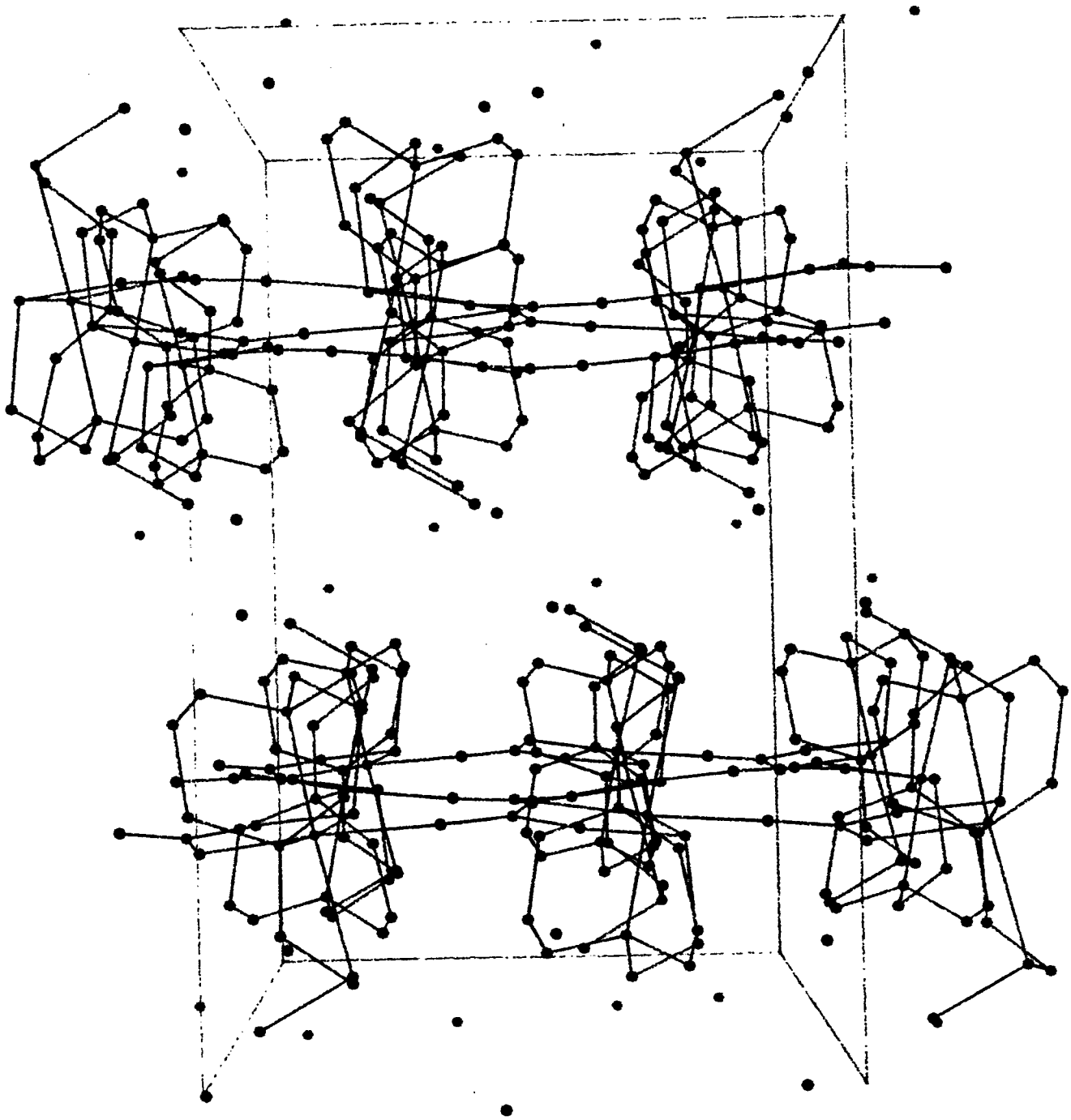

Figure IIS. Packing diagram of [(tbtp-daco)Ni^{II}][I]•H_2O, [5]I, viewed down the *b* axis.