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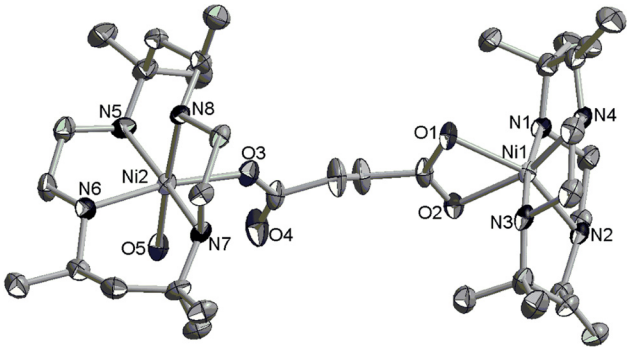
Crystal structure of [(μ_2-succinato $\kappa^3O,O':O''$)-bis-(5,5,7,12,12,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane)]dinickel(II)] diperchlorate, dihydrate $C_{36}H_{82}Cl_2N_8Ni_2O_{15}$



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Abstract

$C_{36}H_{82}Cl_2N_8Ni_2O_{15}$, monoclinic, $P2_1/n$ (no. 14), a = 17.657(3) Å, b = 16.047(3) Å, c = 18.346(3) Å, β = 109.062(4)°, V = 4913.2(16) Å^3, Z = 4, $R_{gt}(F)$ = 0.0561, $wR_{ref}(F^2)$ = 0.1664, T = 173(2) K.

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The molecular structure is shown in the figure (couter anions and the water molecules are omitted for clarity). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

An acetonitrile solution (20 mL) of [NiL]$(ClO_4)_2$ (0.54 g, 1 mmol) (L = 5,5,7,12,12,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane) was added to a mixed solution of succinic acid (0.06 g, 0.5 mmol) and NaOH (0.04 g, 1 mmol) in water (10 mL). The resulting blue solution was evaporated slowly at room temperature to give blue prism-shaped crystals for several days.

Table 1: Data collection and handling.

Crystal:	Blue block
Size:	0.41 × 0.40 × 0.21 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.95 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	27.0°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	21,783, 10,493, 0.033
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 6805
$N(param)_{refined}$:	598
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

Experimental details

The structure was solved using direct methods, which yielded the positions of all non-hydrogen atoms. All hydrogen atoms of the ligands were placed in calculated positions with fixed isotropic thermal parameters and included in the structure factor calculations in the final stage of refinement. The U_{iso} values of the hydrogen atoms of methyl groups were set to $1.5U_{eq}$(C) and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{eq}$(C, N).

Comment

The design and synthesis of extended bridged polymetallic complexes have attracted much interest in the development of materials chemistry. Some complexes bridged by dicarboxylato ligands have been reported [5–6].

X-ray crystal structural analysis reveals that the asymmetric unit of the title structure contains one

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7542 (2)	1.1454 (3)	0.2110 (2)	0.0322 (9)
H1A	0.7322	1.2012	0.1923	0.039*
H1B	0.8044	1.1377	0.1989	0.039*
C2	0.7709 (2)	1.1393 (3)	0.2969 (2)	0.0293 (9)
H2A	0.7933	1.0837	0.3155	0.035*
H2B	0.8108	1.1820	0.3238	0.035*
C3	0.7042 (2)	1.1324 (2)	0.3961 (2)	0.0290 (9)
H3	0.7133	1.0711	0.4039	0.035*
C4	0.7750 (2)	1.1776 (3)	0.4520 (2)	0.0387 (10)
H4A	0.8245	1.1599	0.4435	0.058*
H4B	0.7780	1.1643	0.5050	0.058*
H4C	0.7681	1.2379	0.4437	0.058*
C5	0.6273 (2)	1.1550 (3)	0.4129 (2)	0.0319 (9)
H5A	0.6108	1.2114	0.3917	0.038*
H5B	0.6406	1.1588	0.4695	0.038*
C6	0.5542 (2)	1.0967 (3)	0.3819 (3)	0.0336 (10)
C7	0.5747 (3)	1.0084 (3)	0.4115 (3)	0.0408 (11)
H7A	0.5264	0.9736	0.3938	0.061*
H7B	0.5951	1.0088	0.4680	0.061*
H7C	0.6157	0.9856	0.3917	0.061*
C8	0.4884 (3)	1.1289 (3)	0.4131 (3)	0.0435 (11)
H8A	0.4778	1.1878	0.3995	0.065*
H8B	0.5063	1.1228	0.4693	0.065*
H8C	0.4392	1.0965	0.3902	0.065*
C9	0.4784 (2)	1.1679 (3)	0.2579 (3)	0.0365 (10)
H9A	0.5082	1.2195	0.2788	0.044*
H9B	0.4274	1.1687	0.2694	0.044*
C10	0.4612 (2)	1.1645 (3)	0.1724 (3)	0.0378 (10)
H10A	0.4307	1.1133	0.1512	0.045*
H10B	0.4283	1.2132	0.1477	0.045*
C11	0.5265 (2)	1.1478 (3)	0.0725 (2)	0.0361 (10)
H11	0.5069	1.0892	0.0612	0.043*
C12	0.4648 (3)	1.2055 (3)	0.0180 (3)	0.0551 (14)
H12A	0.4127	1.1976	0.0254	0.083*
H12B	0.4601	1.1923	−0.0354	0.083*
H12C	0.4819	1.2635	0.0292	0.083*
C13	0.6062 (3)	1.1550 (3)	0.0572 (3)	0.0381 (10)
H13A	0.6321	1.2075	0.0810	0.046*
H13B	0.5942	1.1606	0.0008	0.046*
C14	0.6680 (3)	1.0845 (3)	0.0852 (2)	0.0347 (10)
C15	0.7382 (3)	1.1017 (3)	0.0558 (3)	0.0454 (11)
H15A	0.7576	1.1587	0.0695	0.068*
H15B	0.7204	1.0952	−0.0003	0.068*
H15C	0.7816	1.0622	0.0795	0.068*
C16	0.6318 (3)	1.0004 (3)	0.0543 (3)	0.0400 (10)
H16A	0.6735	0.9574	0.0694	0.060*
H16B	0.6094	1.0028	−0.0020	0.060*
H16C	0.5892	0.9867	0.0757	0.060*
C17	0.6036 (2)	0.9251 (2)	0.2402 (3)	0.0325 (10)
C18	0.5965 (3)	0.8326 (3)	0.2496 (3)	0.0448 (12)
H18A	0.6068	0.8036	0.2061	0.054*
H18B	0.6379	0.8145	0.2978	0.054*
C19	0.5172 (3)	0.8081 (3)	0.2521 (3)	0.0522 (13)
H19A	0.4780	0.8142	0.1996	0.063*
H19B	0.5015	0.8476	0.2861	0.063*
C20	0.5105 (2)	0.7197 (2)	0.2801 (3)	0.0328 (9)
C21	0.2564 (2)	0.6489 (3)	0.2818 (2)	0.0315 (9)
H21A	0.2289	0.6942	0.3000	0.038*
H21B	0.2267	0.5965	0.2812	0.038*
C22	0.2570 (2)	0.6684 (2)	0.2017 (2)	0.0311 (9)
H22A	0.2014	0.6751	0.1664	0.037*
H22B	0.2862	0.7211	0.2021	0.037*
C23	0.3116 (3)	0.6204 (3)	0.1012 (2)	0.0341 (9)
H23	0.3518	0.6666	0.1120	0.041*
C24	0.2354 (3)	0.6497 (3)	0.0382 (3)	0.0475 (12)
H24A	0.1934	0.6075	0.0305	0.071*
H24B	0.2469	0.6580	−0.0101	0.071*
H24C	0.2172	0.7024	0.0539	0.071*
C25	0.3469 (2)	0.5444 (3)	0.0727 (2)	0.0343 (9)
H25A	0.3121	0.4961	0.0726	0.041*
H25B	0.3428	0.5551	0.0184	0.041*
C26	0.4335 (2)	0.5184 (3)	0.1160 (2)	0.0341 (10)
C27	0.4555 (3)	0.4461 (3)	0.0716 (3)	0.0441 (11)
H27A	0.5100	0.4269	0.0996	0.066*
H27B	0.4528	0.4651	0.0201	0.066*
H27C	0.4177	0.4000	0.0670	0.066*
C28	0.4921 (3)	0.5903 (3)	0.1197 (3)	0.0460 (12)
H28A	0.4834	0.6346	0.1528	0.069*
H28B	0.4830	0.6122	0.0677	0.069*
H28C	0.5472	0.5698	0.1409	0.069*
C29	0.4076 (3)	0.4100 (2)	0.2033 (3)	0.0358 (10)
H29A	0.4385	0.3661	0.1875	0.043*
H29B	0.3518	0.4081	0.1675	0.043*
C30	0.4078 (3)	0.3924 (2)	0.2834 (3)	0.0351 (10)
H30A	0.3813	0.3382	0.2844	0.042*
H30B	0.4637	0.3888	0.3187	0.042*
C31	0.3774 (2)	0.4557 (3)	0.3943 (2)	0.0327 (9)
H31	0.4348	0.4692	0.4226	0.039*
C32	0.3596 (3)	0.3684 (3)	0.4197 (3)	0.0422 (11)
H32A	0.3035	0.3540	0.3925	0.063*
H32B	0.3693	0.3683	0.4754	0.063*
H32C	0.3947	0.3275	0.4073	0.063*
C33	0.3253 (2)	0.5203 (3)	0.4166 (2)	0.0346 (10)
H33A	0.3258	0.5067	0.4694	0.042*
H33B	0.2696	0.5123	0.3819	0.042*
C34	0.3450 (2)	0.6132 (3)	0.4153 (2)	0.0310 (9)
C35	0.4294 (2)	0.6342 (3)	0.4677 (2)	0.0369 (10)
H35A	0.4687	0.6066	0.4485	0.055*
H35B	0.4364	0.6150	0.5202	0.055*
H35C	0.4375	0.6947	0.4681	0.055*
C36	0.2845 (3)	0.6628 (3)	0.4430 (3)	0.0417 (11)
H36A	0.2899	0.7224	0.4343	0.063*
H36B	0.2952	0.6528	0.4982	0.063*
H36C	0.2299	0.6447	0.4142	0.063*
Cl1	0.66033 (7)	0.10390 (7)	0.66170 (7)	0.0425 (3)
Cl2	0.85128 (6)	0.88737 (6)	0.20413 (7)	0.0358 (2)
N1	0.69523 (18)	1.07966 (19)	0.17206 (18)	0.0267 (7)

Table 2: (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H1C	0.7250	1.0258	0.1855	0.032*
N2	0.69522 (17)	1.15238 (19)	0.31433 (18)	0.0256 (7)
H2C	0.6791	1.2122	0.3045	0.031*
N3	0.52669 (18)	1.09387 (19)	0.2952 (2)	0.0295 (8)
H3A	0.4896	1.0451	0.2802	0.035*
N4	0.53820 (18)	1.16500 (19)	0.15537 (19)	0.0290 (7)
H4D	0.5628	1.2215	0.1682	0.035*
N5	0.4429 (2)	0.4926 (2)	0.19741 (19)	0.0318 (8)
H5C	0.5018	0.4864	0.2239	0.038*
N6	0.36456 (19)	0.4598 (2)	0.30986 (19)	0.0286 (7)
H6A	0.3059	0.4527	0.2821	0.034*
N7	0.33957 (18)	0.6403 (2)	0.33500 (18)	0.0264 (7)
H7D	0.3627	0.6978	0.3409	0.032*
N8	0.29694 (18)	0.59898 (19)	0.17466 (18)	0.0256 (7)
H8D	0.2609	0.5493	0.1650	0.031*
Ni1	0.61140 (3)	1.07906 (3)	0.23348 (3)	0.02325 (14)
Ni2	0.40119 (3)	0.57213 (3)	0.27082 (3)	0.02559 (14)
O1	0.54938 (16)	0.96458 (16)	0.18808 (17)	0.0322 (6)
O1W	0.8077 (3)	0.8876 (4)	0.3809 (3)	0.0969 (17)
H1WA	0.767 (3)	0.920 (4)	0.362 (4)	0.116*
H1WB	0.837 (3)	0.893 (4)	0.351 (3)	0.116*
O2	0.66254 (16)	0.96475 (16)	0.28406 (16)	0.0315 (6)
O2W	0.3829 (3)	0.9645 (4)	0.1474 (4)	0.134 (2)
H2WA	0.4340 (13)	0.955 (6)	0.160 (5)	0.161*
H2WB	0.367 (4)	0.943 (3)	0.1828 (17)	0.161*
O3	0.44240 (16)	0.68719 (17)	0.25149 (16)	0.0336 (7)
O4	0.57152 (19)	0.6872 (2)	0.3263 (2)	0.0556 (9)
O5	0.51771 (17)	0.55019 (17)	0.35857 (18)	0.0351 (7)
H5D	0.532 (2)	0.5985 (11)	0.346 (2)	0.042*
H5E	0.550 (2)	0.5133 (15)	0.353 (3)	0.042*
O6	0.5923 (2)	0.1546 (2)	0.6249 (2)	0.0557 (9)
O7	0.7168 (3)	0.1465 (3)	0.7165 (4)	0.148 (3)
O8	0.6371 (4)	0.0375 (3)	0.7043 (4)	0.127 (2)
O9	0.6868 (3)	0.0595 (3)	0.6106 (3)	0.1096 (19)
O10	0.7704 (2)	0.8593 (2)	0.1762 (2)	0.0635 (10)
O11	0.8563 (2)	0.9680 (2)	0.2372 (2)	0.0547 (9)
O12	0.8969 (2)	0.8313 (2)	0.2616 (2)	0.0766 (13)
O13	0.8842 (3)	0.8919 (3)	0.1425 (2)	0.0791 (13)

$[(NiL)_2(C_4H_4O_4)(H_2O)]^{2+}$ cation, two $[ClO_4]^-$ anions and two water molecules.

In contrast to similar structures [5–6], each Ni(II) atom displays a six-coordinate octahedral coordination geometry by coordination with four nitrogen atoms from macrocyclic ligand in a folded conformation, plus three oxygen atoms from the succinato ligand (see the figure). The Ni–N bond lengths [2.084(3)–2.164(3) Å] are close to the Ni–O bond lengths [2.057(3)–2.187(3) Å].

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