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Di- and tetra-nuclear nickel(II) complexes of an unsymmetric compartmental ligand bearing a terdentate linear arm and a terdentate dipodal arm

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Abstract

The unsymmetric compartmental proligand HL^{O} reacts with $Ni(OAc)_2$ and NaSCN, to give the homodinuclear complex $[Ni_2(L^{O})(AcO)(SCN)_2]$ (**3**), whereas the unsymmetric compartmental proligand HL^{N} reacts with $Ni(OAc)_2$ in the presence of either $[BF_4]^-$ or $[PF_6]^-$ counteranions to give the tetranuclear complexes $[Ni_4(L^{1})_2(OAc)_4][X]_2$, (**4a**, $X = PF_6$; **4b**, $X = BF_4$), in which ligand modification has occurred such that the iminic pendant arm of the proligand has been converted into an amidic pendant arm. The crystal structures of **3** and $(\mathbf{4b})_2 \cdot 3CH_3OH \cdot 2H_2O$ are reported.

Keywords: Crystal structure; Tetranuclear nickel(II) complexes; Dinickel(II) complexes; Compartmental ligands

1. Introduction

We have recently reported the synthesis and crystal structures of two dinuclear nickel(II) complexes of donor unsymmetric compartmental ligands derived from cresol and bearing a terdentate linear arm and a terdentate dipodal arm—the aminic site was derived from bis-pyridin-2-ylmethylamine and the iminic sites were derived from N^1-(2-aminoethyl)ethane-1,2-diamine (diethylenetriamine) in HL^{N}, and from 2-(2-aminoethylamino)-ethanol in HL^{O} (Fig. 1) [1]. The binding of the terminal donor atom of the terdentate linear arm was dependent on the nature of the accompanying counteranion and so in $[Ni_2(L^{O})(AcO)_2]BF_4$ (**1**) in the presence of the non-coordinating tetrafluoroborate anion a terminal alcohol is coordinated whereas in $[Ni_2(L^{N})(AcO)(SCN)_2]$ (**2**), in the presence of added isothiocyanate ion, the terminal amine is not coordinated but the anion is—this appears to reflect the comparative bond strengths of the putative donor groups.

At the time of our initial report concerning **1** and **2** we had not been able to crystallise the corresponding complexes in which the pendant alcohol is accompanied by an isothiocyanate anion and the pendant amine by a tetrafluoroborate anion. This has now been accomplished and in this paper, we report the results of the reaction of HL^{O} with $Ni(OAc)_2$ and NaSCN, and of HL^{N} with $Ni(OAc)_2$ in the presence of either $[BF_4]^-$ or $[PF_6]^-$ counteranions together with the crystal structures of the products.

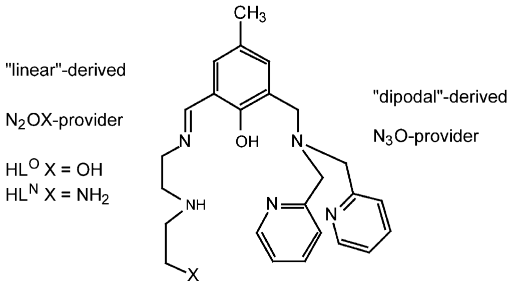

Fig. 1. Unsymmetric compartmental ligands bearing a terdentate linear arm and a terdentate dipodal arm.

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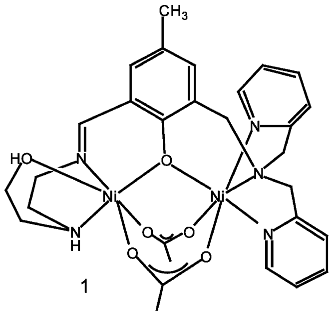

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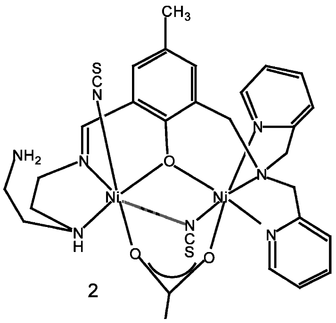

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2. Experimental

The proligands HL^N and HL^O were prepared following the methods of Ref. 2 and 3-[(bispyridin-2-ylmethylamino)methyl]-2-2-hydroxy-5-methylbenzaldehyde was prepared by the method of Ref. [3].

2.1. Complexation reactions

2.1.1. [Ni$_2$(***L***$^{\mathbf{O}}$)(AcO)(SCN)$_2$] (**3**)

2-(2-Aminoethylamino)ethanol 45 mg (0.43 mmol) and 3-[(bispyridin-2-ylmethylamino)methyl]-2-2-hydroxy-5-methylbenzaldehyde 150 mg (0.43 mmol) in 10 cm^3 of methanol were refluxed for 40 min and then cooled to room temperature (r.t.). Nickel acetate tetrahydrate (215 mg, 0.864 mmol) was added and the resulting solution refluxed for 1 h. On cooling, sodium thiocyanate (70 mg, 0.864 mmol) was added, and slow evaporation of the methanolic solution resulted in the formation of green crystals. MS (FAB$^+$) $m/z = 666$ $[M-\mathrm{NCS}]^+$. Analysis of bulk sample, Found: C, 46.23; H, 5.16: N, 11.94. Calc. for $[C_{29}H_{33}N_7O_4S_2Ni_2]\cdot 2CH_3OH\cdot H_2O$, C, 46.13; H, 5.33: N, 12.15%.

2.1.2. [Ni$_4$(***L***$^{\mathbf{1}}$)$_2$(OAc)$_4$][PF$_6$]$_2$ (**4a**)

Nickel acetate tetrahydrate (173 mg, 0.694 mmol) was added to a solution of $\mathbf{HL^N}$ (150 mg, 0.347 mmol) in methanol (10 cm^3) and heated at reflux for 1 h. On cooling, sodium hexafluorophosphate (117 mg, 0.694 mmol addition of 2 equiv.) was added to the solution.

2.1.3. [Ni$_4$(***L***$^{\mathbf{1}}$)$_2$(OAc)$_4$][BF$_4$]$_2$ (**4b**)

N^1-(2-aminoethyl)ethane-1,2-diamine (45 mg; 0.43 mmol) was added to a stirred solution of 3-[(bispyridin-2-ylmethyl-amino-)methyl]-2-2-hydroxy-5-methylbenzaldehyde (150 mg; 0.43 mmol) in methanol (10 cm^3). The solution was refluxed for 40 min and then cooled. Nickel acetate tetrahydrate (215 mg, 0.864 mmol) was added and the resulting solution refluxed for 1 h. On cooling, sodium tetrafluoroborate (95 mg, 0.864 mmol) was added to the solution.

In both of the above preparations green solids were recovered as bulk samples from the reaction solution. Crystals suitable for X-ray analysis were grown by diffusion of diethylether into methanolic solutions of the bulk samples. Bulk sample analyses for **4a** and **4b** were inconsistent and evidenced the presence of solvent molecules. Consequently several crystals of **4a** and **4b** were screened to confirm the homogeneity of each crystalline batch. A representative bulk sample analysis of **4b** gave: Found: C, 41.83; H, 5.36; N, 10.18. Calc. for $C_{58}H_{78}B_2F_8N_{12}O_{12}Ni_4\cdot CH_3OH\cdot 6H_2O$: C, 42.07; H, 5.59; N, 9.98%.

2.2. X-ray crystallography

Complex **3** crystallised from $CH_3CN-CH_2Cl_2$ as green plates and complexes $\mathbf{4a}\cdot 2H_2O$ and $(\mathbf{4b})_2\cdot 3CH_3OH\cdot 2H_2O$ crystallised as green plates on diffusion of diethylether into methanol solutions. For all complexes, data collected were measured on a Bruker SMART CCD area detector with an Oxford Cryosystems low temperature system at 150 K. Crystallographic data is summarised in the Table 1.

For complex **3** cell parameters were refined from the setting angles of 38 reflections (θ range 1.84°–28.28°) and for complex $(\mathbf{4b})_2\cdot 3CH_3OH\cdot 2H_2O$ cell parameters were refined from the setting angles of 63 reflections (θ range 0.79°–28.34°). Reflections were measured from a hemisphere of data collected of frames each covering 0.3° in omega. For **3**, of the 19 715 reflections measured, all of which were corrected for Lorentz and polarisation effects and for absorption by semi-empirical methods based on symmetry-equivalent and repeated reflections (minimum and maximum transmission coefficients 0.6944 and 0.9230), 4876 independent reflections exceeded the significance level $|F|/\sigma(|F|) > 4.0$. For $(\mathbf{4b})_2\cdot 3CH_3OH\cdot 2H_2O$, of the 45 806 reflections measured, all of which were corrected for Lorentz and polarisation effects and for absorption by semi empirical methods based on symmetry-equivalent and repeated reflections (minimum and maximum transmission coefficients

Table 1
Summary of crystallographic data for $[Ni_2(\mathbf{L^O})(AcO)(NCS)_2]$ (**3**); and $[Ni_4(\mathbf{L^1})_2(AcO)_4][BF_4]_2\cdot 3MeOH\cdot 2H_2O$ ($(\mathbf{4b})_2\cdot 3CH_3OH\cdot 2H_2O$)

	3	$(\mathbf{4b})_2\cdot 3CH_3OH\cdot 2H_2O$
Empirical formula	$C_{29}H_{33}N_7Ni_2O_4S_2$	$C_{119}H_{164}B_4F_{16}N_{24}Ni_8O_{29}$
Formula weight	725.16.14	3211.66
Space group	$Pna2_1$($C_2{}^9$, No. 15)	$P\bar{1}$($C_i{}^1$, No. 2)
a (Å)	14.893(2)	10.923(3)
b (Å)	16.566(3)	26.221(8)
c (Å)	12.9394(19)	26.234(8)
α (°)	90	87.499(9)
β (°)	90	78.122(7)
γ (°)	90	78.036(7)
V (Å^3)	3192.3(9)	7193.1(4)
ρ_{calc} (Mg m^{-3})	1.509	1.483
Z	4	2
μ (mm^{-1})	1.356	1.120
λ (Å)	0.71073	0.71073
F(000)	1500	3332
R	0.0392	0.0738
wR_2	0.0851	0.2117

0.6098 and 0.8773), 11 732 independent reflections exceeded the significance level $|F|/\sigma(|F|) > 4.0$. The structures were solved by direct methods and refined by full matrix least-squares methods on F^2. Hydrogen atoms were placed geometrically and refined with a riding model (including torsional freedom for methyl groups) and with U_{iso} constrained to be 1.2 (1.5 for methyl groups) times U_{eq} of the carrier atom with the exception of O1W from $(\mathbf{4b})_2\cdot 3CH_3OH\cdot 2H_2O$ for which H atoms were attempted but found to be indeterminate. For **3** refinement converged at a final $R_1 = 0.0392$ ($wR_2 = 0.0851$, for all 6665 data, 409 parameters, mean and maximum δ/σ 0.000, 0.000), the minimum and maximum final electron density were −0.418 and 0.386 e Å^{-3}. The atom O4 was found to be disordered and refined to a percentage occupancy of 57.8: 42.2%. A weighting scheme $w = 1/[\sigma^2(F_o{}^2) + (0.0337P)^2 + 0.00P]$ where $P = (F_o{}^2 + 2F_c{}^2)/3$ was used in the latter stages of refinement. For $(\mathbf{4b})_2\cdot 3CH_3OH\cdot 2H_2O$ refinement converged at a final $R = 0.0738$ ($wR_2 = 0.2117$, for all 32 510 data, 1788 parameters, mean and maximum δ/σ 0.000, 0.001) with allowance for the thermal anisotropy of all non-hydrogen atoms (with the exceptions of O1W and O2W which were refined isotropically, O1S and C1S which were found to be disordered and refined isotropically to the occupancy of 45.9/54.1%, O2S and C2S which were found to be disordered and refined isotropically to the occupancy of 48.2/51.8% and O3S and C3S which were found to be disordered and refined isotropically to the occupancy of 53.9/46.1%). Minimum and maximum final electron density −0.857 and 1.349 e Å^{-3} (the latter being 1.09 Å away from O2W). A weighting scheme $w = 1/[\sigma^2(F_o{}^2) + (0.0856P)^2 + 0.00P]$ where $P = (F_o{}^2 + 2F_c{}^{2)}/3$ was used in the latter stages of refinement.

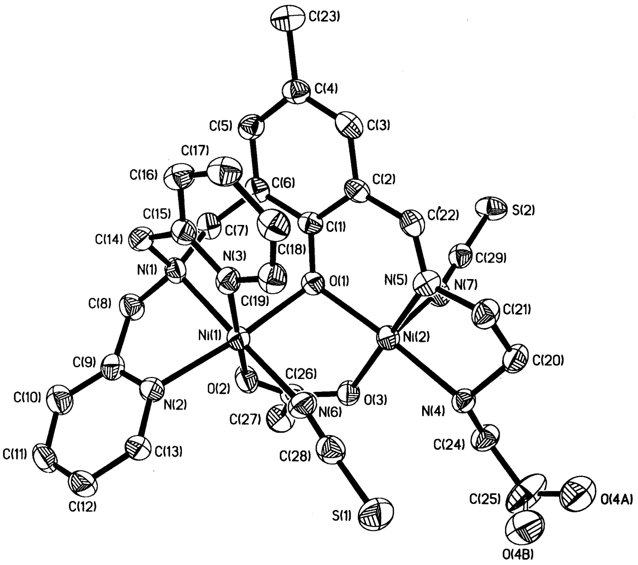
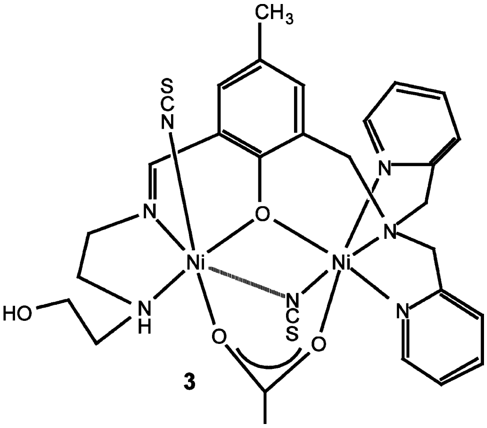


Fig. 2. ORTEP drawing of the molecular structure of $[Ni_2(\mathbf{L^O})(AcO)(SCN)_2]$ (**3**), showing the crystallographic labelling. Thermal ellipsoids for the non-hydrogen atoms are drawn at the 50% probability level. Selected bonds (Å) and angles (°) at the metal atoms: Ni(1)–O(2), 2.039(3); Ni(1)–O(1), 2.060(2); Ni(1)–N(6), 2.073(4); Ni(1)–N(2), 2.088(4); Ni(1)–N(3), 2.094(3); Ni(1)–N(1), 2.111(3); Ni(2)–O(1), 1.966(2); Ni(2)–N(7), 2.013(4); Ni(2)–N(5), 2.025(3); Ni(2)–O(3), 2.041(3); Ni(2)–N(4), 2.102(3); Ni(1)–Ni(2), 3.2136(8) Å. N(6)–Ni(1)–N(1), 175.03(14); O(2)–Ni(1)–N(3), 172.26(13); O(1)–Ni(1)–N(2), 162.93(13); O(1)–Ni(1)–N(6), 91.49(13); N(2)–Ni(1)–N(6), 101.21(14); N(2)–Ni(1)–N(1), 79.11(13); N(1)–Ni(1)–O(1), 89.22(11); N(5)–Ni(2)–O(3), 175.29(13); O(1)–Ni(2)–N(4), 163.82(13); O(1)–Ni(2)–N(7), 99.66(12); O(3)–Ni(2)–N(7), 93.66(13); N(4)–Ni(2)–N(7), 93.83(14); N(5)–Ni(2)–N(7), 91.03(14); Ni(1)–O(1)–Ni(2), 105.87(10).

The programs used in the determination and refinement of the structures were Siemens SMART and SAINT for control and integration software [4] and SHELXTL as implemented on the Viglen Pentium computer [5].

3. Results and discussion

The structure of the complex $[Ni_2(\mathbf{L^O})(AcO)(SCN)_2]$ (**3**), derived from $H\mathbf{L^O}$, $Ni(OAc)_2$ and NaSCN, has been found to closely parallel that of **2**. Two six-coordinate nickel(II) atoms are bridged by the cresolic oxygen atom of $[\mathbf{L^N}]^-$, and a *syn–syn* bidentate ethanoate anion (Fig. 2). The six coordination of the nickel(II) atom in the aminic compartment [Ni(1)] is completed by interaction with the three nitrogen atoms of the dipodal arm and the N atom of an isothiocyanate anion. The phenolic oxygen atom O(1) is *trans* to a pyridinyl nitrogen atom N(2) [O(1)–Ni(1)–N(2), 162.9°]; the second pyridinyl nitrogen atom N(3) is *trans* to oxygen atom O(2) from the bidentate acetate bridge [O(2)–Ni(1)–N(3), 172.3°] and the articular amino nitrogen atom N(1) is *trans* to the nitrogen atom N(6) from the thiocyanate anion [N(1)–Ni(1)–N(6), 175.1°]. At first sight, the coordination geometry at the nickel(II) in the iminic compartment, Ni(2), appears to be close to square pyramidal, $\tau = 0.30$, [6] with an NCS^- in an axial position. However, the two isothiocyanate anions in the molecule are *trans* to each other and the NCS^- coordinated at Ni(1) is disposed towards the open face of the square pyramidal Ni(2). The Ni(2)–N(6) distance is 2.738 Å and so there is a weak interaction leading to an unsymmetrical isothiocyanato bridge between Ni(1) and Ni(2) [Ni(1)–N(6), 2.072(4) Å]. A distorted octahedral coordination geometry results with the remaining sites arising through interaction with two N donor atoms from the linear iminic arm and the N atom from a monodentate isothiocyanato anion. The phenolic oxygen atom O(1) is *trans* to a aminic nitrogen atom N(4) [O(1)–Ni(2)–N(4), 163.5°]; the articular aminic nitrogen atom N(5) is *trans* to oxygen atom O(3) from the bidentate acetate bridge [O(3)–Ni(2)–N(5), 175.3°] and the nitrogen atom N(7) from the monodentate thiocyanate anion N(1) is *trans* to the nitrogen atom N(6) from the bridging thiocyanate anion [N(7)–Ni(2)–N(6), 175.1°]. The terminal hydroxyl function is not coordinated and the Ni–Ni separation of 3.214 Å in **3**, is shorter than that found in **2**, 3.332 Å. The terminal O atom of the pendant aminoethanol is not cooordinated to the nickel(II) atom and is disordered with a percentage occupancy of 57.8: 42.2%.



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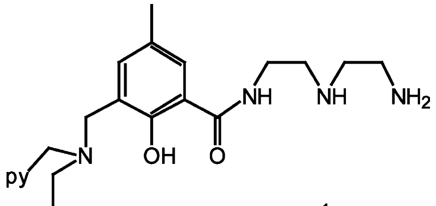
In contrast the reaction of HL^N with $Ni(AcO)_2$ in the presence BF_4^- or PF_6^- anions gave an unexpected and intriguing result. The crystal structures of the reaction products, $[Ni_4(\mathbf{L}^1)_2(OAc)_4][X]_2$ (**4a**, X = PF_6: **4b**, X = BF_4), revealed that in each case the cationic species is tetranuclear; the structure of [(**4b**)$_2$·$3CH_3OH·2H_2O$] is discussed herein with that of **4a**·$2H_2O$ submitted electronically. Both structures show that ligand modification has occurred such that the iminic pendant arm of the proligand has been converted into an amidic pendant arm ($\mathbf{HL}^1$). At the amidic centre the bond angles at the amidic C–O atom are close to 120° indicating an sp^2 hybridised carbon centre and there are short C–O distances of approximately 1.26 Å indicating a double bond. The C–N distances are approximately 1.33 Å. Varying the reaction procedure, by starting from the preformed pro-ligand $\mathbf{HL}^N$ (as for **4a**·$2H_2O$) or from $\mathbf{HL}^N$ prepared in situ (as for (**4b**)$_2$·$3CH_3OH·2H_2O$) led only to the products containing $\mathbf{HL}^1$.



HL¹

There are two closely similar molecules in the structure of complex (**4b**)$_2$·$3CH_3OH·2H_2O$ one of which is detailed here (Fig. 3). Each nickel atom is in a pseudo-octahedral geometry with those in the aminic compartments [Ni(1A)] coordinated by N_3O_2 donor sets and with those in the amidic compartments [Ni(2A)] coordinated by N_2O_4 donor sets. The octahedra share an apex, O(1A), and the pairs of nickel atoms are bridged by the cresolato-O atoms and by two *syn–syn* acetates. As in complex **3**, the six coordination at the aminic nickel site is completed by the three N donor atoms of the dipodal arm. The cresolic oxygen atom O(1A) is *trans* to a pyridinyl nitrogen atom N(2A) [O(1A)–Ni(1A)–N(2A), 168.3°]; the second pyridinyl nitrogen atom N(3A) is *trans* to oxygen atom O(2A) from a bidentate acetate bridge [O(2A)–Ni(1A)–N(3A), 172.1°]. The articular amino nitrogen atom N(1A) is *trans* to oxygen atom O(4A) from the second bidentate acetate bridge [N(1A)–Ni(1A)–O(4A), 169.3°]. The six coordination at the amidic nickel site is completed by the amido-O atom and chelation to two N donor atoms of the amidic arm from an adjacent dinuclear unit so forming the tetranuclear array. The phenolic oxygen atom O(1A) is now *trans* to the aminic nitrogen atom N(5AA) [O(1A)–Ni(2A)–N(5AA), 168.6°]; the terminal amino nitrogen atom N(6AA) is *trans* to oxygen atom O(5A) from a bidentate acetate bridge [N(6AA)–Ni(2A)–O(5A), 176.9°] and the amidic oxygen atom O(6A) is *trans* to oxygen atom O(3A) from the second bidentate acetate bridge [O(6A)–Ni(2A)–O(3A), 177.5°]. The Ni$\cdots$Ni separations are averaged at 3.415 Å and may be compared with the Ni$\cdots$Ni separations in the related dinuclear complexes bearing two *syn–syn* bridging acetates **1** and ($[Ni_2\mathbf{L}^2(AcO)_2(MeOH)]PF_6$) (**5**), in which the separations are, 3.332 and 3.396 Å, respectively [3].

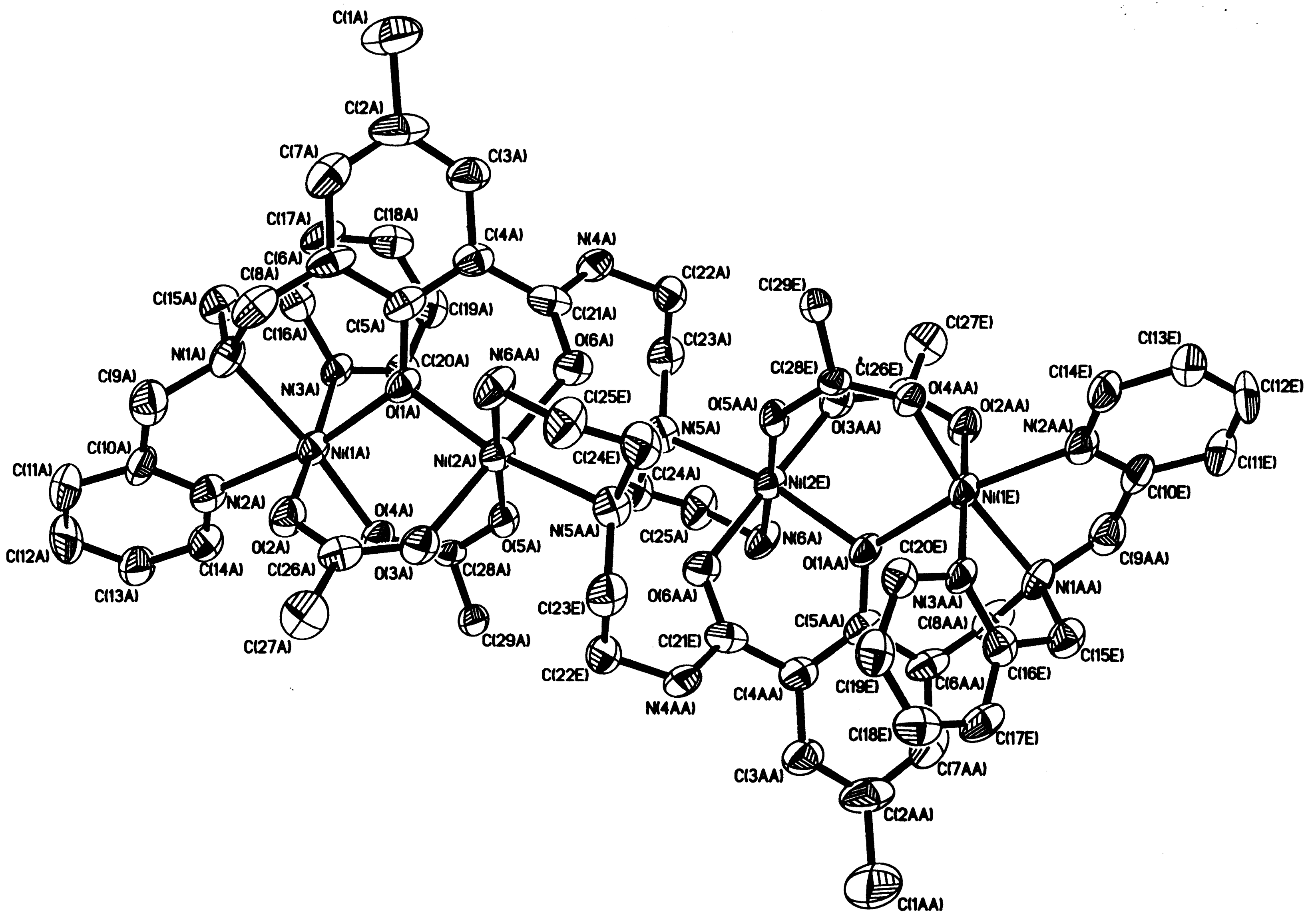
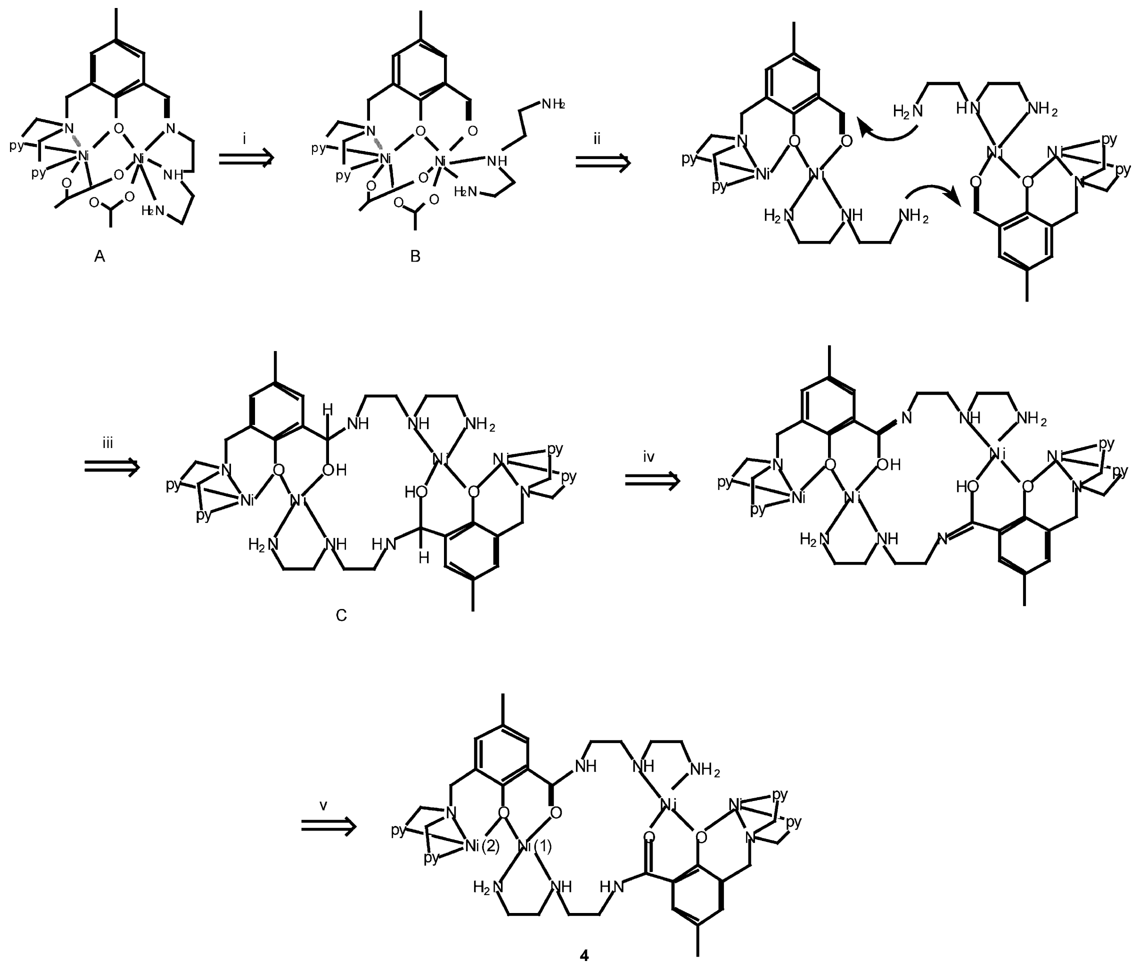
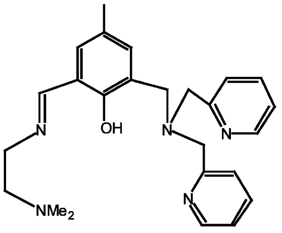


Fig. 3. ORTEP drawing of the molecular structure of the cation $[Ni_4(\mathbf{L^1})_2(OAc)_4]^{2+}$ from one molecule of $(\mathbf{4b})_2 \cdot 3CH_3OH \cdot 2H_2O)$, showing the crystallographic labelling. Thermal ellipsoids for the non-hydrogen atoms are drawn at the 50% probability level. Selected bonds (Å) and angles (°) at the metal atoms of one dinuclear set: Ni(1A)–O(4A), 2.003(5); Ni(1A)–O(1A), 2.034(5); Ni(1A)–O(2A), 2.042(5); Ni(1A)–N(3A), 2.077(6); Ni(1A)–N(2A), 2.094(6); Ni(1A)–N(1A), 2.116(7); Ni(2A)–O(1A), 2.006(5); Ni(2A)–O(3A), 2.059(5); Ni(2A)–O(5A), 2.060(5); Ni(2A)–O(6A), 2.098(5); Ni(2A)–N(5AA), 2.147(6); Ni(1A)–Ni(2A), 3.4179(15) Å; Ni(1B)–Ni(2B), 3.4117(14) Å. O(2A)–Ni(1A)–N(3A), 172.1(2); O(1A)–Ni(1)–(N2A), 168.3(3); O(4A)–Ni(1)–N(1A), 169.3(2); O(4A)–Ni(1A)–O(2A), 91.6(2); O(4A)–Ni(1A)–N(3A), 94.9(2); O(2A)–Ni(1A)–N(1A), 92.6(2); N(3A)–Ni(1A)–N(1A), 81.75(2); O(3A)–Ni(2A)–O(6A), 177.50(19); O(5A)–Ni(2A)–N(6AA), 176.9(2); O(1A)–Ni(2A)–N(5AA), 168.6(2); O(1A)–Ni(2A)–O(3A) 92.8(2), O(1A)–Ni(2A)–O(6A) 86.6(2), O(3A)–Ni(2A)–N(5AA), 94.9(2); N(5AA)–Ni(2A)–O(6A), 86.1(2); Ni(1A)–O(1)–Ni(2A), 115.6(2).



Scheme 1. Formation of complex **4**. (i) Hydrolysis; (ii) tetranuclear assembly; (iii) carbinolamine formation; (iv) oxidation of carbinolamine C–N bond; (v) tautomerisation. The *syn–syn* bridging acetates are removed in steps (ii)–(v) for clarity. The structure of **4** is given in Fig. 3.



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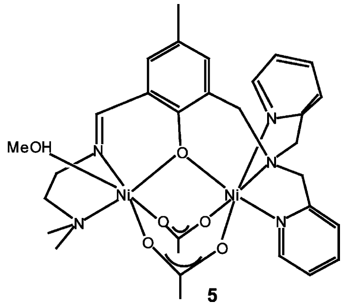

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A possible pathway for the generation of **4** is given in Scheme 1. It is proposed that a dinuclear complex of the proligand is formed (A) and that this hydrolyses at the imine to give a dinuclear complex (B) of the precursor aldehyde, 3-[(bis-pyridin-2-ylmethyl-amino-)methyl]-2-hydroxy-5-methylbenzaldehyde, as has been previously noted for related dinuclear nickel(II) complexes [2]. Two molecules of B then self assemble to produce the carbinolamine complex (C), which undergoes oxidation of the carbinolamine C–N bond followed by tautomerisation to produce the amidic product **4**. The overall reaction resembles that involving the oxidation of the C–N bond of a carbinolamine formed from L-tryptophan and formaldehyde by the flavoenzyme *N*-methyltryptophan oxidase, followed by tautomerisation to *N*-formyl-L-tryptophan [7].

4. Conclusion

The unsymmetric compartmental proligand $\mathbf{HL^O}$ bearing a terdentate linear arm with a terminal hydroxo-group reacts with $Ni(OAc)_2$ and NaSCN to give a homodinuclear complex $[Ni_2(\mathbf{L^O})(AcO)(SCN)_2]$ (**3**) in which the hydroxo-group is non-coordinating. In contrast the unsymmetric compartmental proligand $\mathbf{HL^N}$ in which the terdentate linear arm has a terminal amino-group reacts with $Ni(OAc)_2$ in the presence of either $[BF_4]^-$ or $[PF_6]^-$ counteranions to give the tetranuclear complexes $[Ni_4(\mathbf{L^1})_2(OAc)_4][X]_2$, (**4a**, $X = PF_6$; **4b**, $X = BF_4$), in which ligand oxidationation has occurred such that the iminic pendant arm of the proligand has been converted into an amidic pendant arm.

5. Supplementary material

Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publications nos. CCDC 188984 (complex **3**), CCDC 188983 (complex (**4b**)$_2 \cdot 3CH_3OH \cdot 2H_2O$) and CCDC 188985 (complex **4a**$\cdot 2H_2O$). Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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