Polyhedron 44 (2012) 72–76

Contents lists available at SciVerse ScienceDirect

Polyhedron

journal homepage: www.elsevier.com/locate/poly

New cobalt(II) and nickel(II) complexes of 2-hydroxy-benzyl derivatives of 4-aminoantipyrine

Perizad A. Fatullayeva [a], Ajdar A. Medjidov [a,*], Abel M. Maharramov [b], Atash V. Gurbanov [b], Rizvan K. Askerov [b], Karim Q. Rahimov [a], Maximilian N. Kopylovich [c], Kamran T. Mahmudov [b,c,*], Armando J.L. Pombeiro [c]

[a] Institute of Chemical Problems of Azerbaijan Academy of Sciences, H.Cavid Str. 29, Az 1143 Baku, Azerbaijan
[b] Department of Chemistry, Baku State University, Z. Xalilov Str. 23, Az 1148 Baku, Azerbaijan
[c] Centro de Química Estrutural, Complexo I, Instituto Superior Técnico, Technical University of Lisbon, Av. Rovisco Pais, 1049-001 Lisbon, Portugal

ARTICLE INFO

Article history:
Received 9 April 2012
Accepted 11 June 2012
Available online 3 July 2012

Keywords:
4-Aminoantipyrine
Schiff condensation with reduction
Cobalt(II) and nickel(II) complexes
X-ray structural analysis

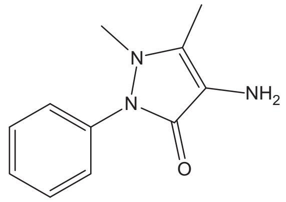
ABSTRACT

The complexes $[Co(HL^2)_2]\cdot H_2O\cdot 3CH_3OH$ (**3**), $[Ni(HL^1)_2]\cdot 4H_2O\cdot CH_3OH$ (**4**) and $[Ni(HL^2)_2]\cdot 4H_2O\cdot CH_3OH$ (**5**) with 4-((2-hydroxybenzyl)amino)-1,5-dimethyl-2-phenyl-1H-pyrazol-3(2H)-one (H_2L^1, **1**) and 4-((5-bromo-2-hydroxybenzyl)amino)-1,5-dimethyl-2-phenyl-1H-pyrazol-3(2H)-one (H_2L^2, **2**) ligands were synthesized and characterized by elemental analysis, ESI-MS, IR spectroscopy and X-ray single-crystal analysis. Compounds **1** and **2** were prepared by the Schiff condensation of 4-aminoantipyrine and salicylaldehyde or its 5-bromo-derivative with subsequent reduction. **1** and **2** were characterized by elemental analysis, ESI-MS, IR, ^1H and ^{13}C NMR spectroscopies. All the complexes **3**–**5** have a metal-to-ligand ratio of 1:2, the ligands being coordinated in the ONO mode, while the Co(II) and Ni(II) ions display a distorted octahedral geometry. In the solid state, **3**–**5** are arranged as 1D supramolecular chains through H-bonded water–methanol clusters placed in the voids of the metal-organic units.

1. Introduction

Schiff bases of 4-aminoantipyrine (hereafter denoted as SBA, Scheme 1) and their complexes have attracted attention over the past decades, not only due to their relatively easy synthesis, but also in view of their potential biological, pharmacological and analytical applications [1–14]. In spite of the high interest, only few of the SBA coordination compounds have been characterized structurally; the reported examples include Cu^{II} [15–17], Zn^{II} [18], Fe^{II} [15] and Co^{II} [18] complexes of different nuclearities and topologies, with the SBA ligands usually coordinating through the imine nitrogen and carbonyl oxygen. Moreover, the flexibility, stability and, as a consequence, the coordination ability of the SBA ligands can be significantly enhanced by reduction of the C=N imine bond [19] and introduction of additional functionalities, e.g. hydroxybenzo- ones. As result, upon complexation the ligands can form stable fused five- and six-membered metallocycles, leading to predictable coordination (see below).

On the other hand, the engineering of networks by divergent polydentate ligand–metal blocks united through solvent molecules has been performed towards the creation of new supramolecular architectures [20,21] which are of particular interest as models for various biological assemblies [22]. For example, several copper complexes with ligands formed by the Schiff condensation of salicylaldehyde and amino acids with subsequent reduction were explored as models of catalytic centres for enzymatic racemization and transamination [23].



Scheme 1. 4-Aminoantipyrine.

Thus, we believe that the synthesis of new complexes with derivatives of 4-aminoantipyrine as ligands and their structural characterization is interesting at least for some of the above mentioned applications, and hence we focused this work on the following aims: (*i*) to synthesize 4-((2-hydroxybenzyl)amino)-1,5-dimethyl-2-phenyl-1H-pyrazol-3(2H)-one (H_2L^1, **1**) and 4-((5-bromo-2-hydroxybenzyl)amino)-1,5-dimethyl-2-phenyl-1H-pyrazol-3(2H)-one (H_2L^2,

---

* Corresponding authors.
E-mail addresses: ajdarmen@yahoo.com (A.A. Medjidov), kamran_chem@mail.ru, kamran_chem@yahoo.com (K.T. Mahmudov).

http://dx.doi.org/10.1016/j.poly.2012.06.061

2); (*ii*) to prepare Co^{II} and Ni^{II} complexes with the $H_2L^{1,2}$ ligands and shed light on their structural details.

2. Experimental

2.1. Materials and instrumentation

All the chemicals were obtained from commercial sources (Aldrich) and were used as received. Infrared spectra (4000–400 cm^{-1}) were recorded on a BIO-RAD FTS 3000MX instrument in KBr pellets. The ^1H and ^{13}C NMR spectra were performed at room temperature on a Bruker Avance II + 300 (UltraShield™ Magnet) spectrometer operating at 300.130 and 75.468 MHz for proton and carbon-13, respectively. The chemical shifts are reported in ppm using tetramethylsilane as the internal reference. The acidity of the solutions was measured using a CG825 pH-meter with an ESL-43-07 glass electrode adjusted by standard buffer solutions and an EVL-1M3.1 silver-silver chloride reference electrode. Electrospray mass spectra were run with an ion-trap instrument (Varian 500-MS LC Ion Trap Mass Spectrometer) equipped with an electrospray (ESI) ion source. For electrospray ionization, the drying gas and flow rate were optimized according to the particular sample with 35 p.s.i. nebulizer pressure. Scanning was performed from *m/z* 100 to 1200 in methanol solution. The compounds were observed in the positive mode (capillary voltage = 80–105 V).

2.2. Synthesis of $H_2L^{1,2}$

A 1:1 equimolar methanolic solution of 4-aminoantipyrine (0.406 g, 0.002 mol) and 2-hydroxybenzaldehyde (0.244 g, 0.002 mol) or 5-bromo-2-hydroxybenzaldehyde (0.402 g, 0.002 mol) was mixed and slightly heated (80 °C) for 2 h with stirring. The characteristic yellow precipitate obtained by the Schiff condensation was filtered off and recrystallized from methanol, yields are around 85% for both Schiff bases. The Schiff base intermediate (0.002 mol) was dissolved in 20 mL of ethanol, then sodium borohydride ($NaBH_4$, 0.003 mol) was added in portions under vigorous stirring until the disappearance of the yellow color. Then reaction mixture was diluted with 100 mL of water and the basic solution was neutralized with HCl until it was neutral (pH 7). Colorless microcrystals of H_2L started to form immediately after the acidification. After *ca.* 1 h $H_2L^{1,2}$ was separated by filtration and recrystallized from methanol.

H_2L^1 (**1**): yield, 84% (based on 4-aminoantipyrine), soluble in methanol, ethanol and acetone. *Anal.* Calc. for $C_{18}H_{19}N_3O_2$ (M = 309.15): C, 69.88; H, 6.19; N, 13.58. Found: C, 69.92; H, 6.30; N, 13.62%. IR, cm^{-1}: 3360 ν(OH), 3341 ν(NH), 1630 ν(C=O). ESI-MS: *m/z*: 310 $[M+H]^+$. ^1H NMR in DMSO-d_6, δ (ppm): 2.42 (s, 3H, CCH_3), 3.15 (s, 3H, NCH_3), 4.23 (m, 2H, CH_2), 6.68–7.76 (9H, Ar–H), 13.11 (s, 1H, O–H), 11.08 (s, 1H, N–H). ^{13}C-$\{^1H\}$ NMR in DMSO-d_6, δ (ppm): 16.3 (CH_3), 31.2 (CH_3), 45.7 (CH_2), 115.1, 121.4, 123.9, 122.8, 128.4, 128.7, 129.8 (Ar–H), 130.8 (Ar–CH_2), 132.5 ($C_{py}NH$), 133.5 ($C_{py}CH_3$), 135.9 ($C_{Ar}N_{py}$), 146.5 (Ar–OH), 160.7 (C=O).

H_2L^2 (**2**): yield, 80% (based on 4-aminoantipyrine), soluble in methanol, ethanol and acetone. *Anal.* Calc. for $C_{18}H_{18}BrN_3O_2$ (M = 387.06): C, 55.68; H, 4.67; N, 10.82. Found: C, 55.75; H, 4.57; N, 10.46%. IR, cm^{-1}: 3480 ν(OH), 3184 ν(NH), 1638 ν(C=O). ESI-MS: *m/z*: 388 $[M+H]^+$. ^1H NMR in DMSO-d_6, δ (ppm): 2.46 (s, 3H, CCH_3), 3.18 (s, 3H, NCH_3), 4.31 (m, 2H, CH_2), 6.55–7.47 (8H, Ar–H), 12.94 (s, 1H, O–H), 11.42 (s, 1H, N–H). ^{13}C-$\{^1H\}$ NMR in DMSO-d_6, δ (ppm): 16.5 (CH_3), 31.5 (CH_3), 45.8 (CH_2), 112.3 (Ar–Br), 119.2, 120.1, 122.7, 127.5, 127.8, 129.1 (Ar–H), 131.0 (Ar–CH_2), 133.1 ($C_{py}NH$), 133.4 ($C_{py}CH_3$), 134.8 ($C_{Ar}N_{py}$), 146.2 (Ar–OH), 161.0 (C=O).

2.3. Preparation of the complexes $[Co(HL^2)_2]\cdot H_2O\cdot 3CH_3OH$ (**3**), $[Ni(HL^1)_2]\cdot 4H_2O\cdot CH_3OH$ (**4**) and $[Ni(HL^2)_2]\cdot 4H_2O\cdot CH_3OH$ (**5**)

To 50 mL of an ethanol–water (1:1 *v*/*v*) solution of $H_2L^{1,2}$ (0.2 mmol), 0.2 mmol NaOH was added under continuous stirring. After 5 min, 0.1 mmol $CoSO_4\cdot 7H_2O$ (or $NiSO_4\cdot 7H_2O$ in the case of **4** and **5**) was added. The mixture was stirred under solvent refluxing for 5 min and then left for slow solvent evaporation. Light brown (**3**), dark brown (**4**) or light blue (**5**) crystals of the complexes started to form after ca 2 days, and after 5 d they were filtered off, washed with a small amount of methanol and dried in air. Crystals suitable for X-ray diffraction analysis were obtained upon recrystallization from methanol.

$[Co(HL^2)_2]\cdot H_2O\cdot 3CH_3OH$ (**3**): yield, 88% (based on Co). *Anal.* Calc. for $C_{39}H_{48}Br_2CoN_6O_8$ (M = 947.57): C, 49.43; H, 5.11; N, 8.87. Found: C, 49.18; H, 5.04; N, 8.75%. IR, cm^{-1}: 3196 ν(NH), 1650 ν(C=O). ESI-MS: *m/z*: 833.12 $[M–H_2O–3CH_3OH+H]^+$.

Table 1
Crystal data and structure refinement details for **3**–**5**.

	3	**4**	**5**
Formula unit	$C_{39}H_{48}Br_2CoN_6O_8$	$C_{37}H_{48}N_6NiO_9$	$C_{37}H_{46}Br_2N_6NiO_9$
Formula weight (g mol^{-1})	947.58	779.52	937.33
Crystal system	monoclinic	monoclinic	monoclinic
Space group	*P*2(1)/n	*P* 21/c	*C* 2/c
a (Å)	10.2212(9)	13.383(2)	29.2416(10)
b (Å)	23.9559(18)	10.6236(17)	10.6365(4)
c (Å)	17.2575(13)	13.363(2)	13.6850(5)
α (°)	90	90	90
β (°)	98.703(2)	105.768(3)	105.3570(10)
γ (°)	90	90	90
Z	4	2	4
V ($Å^3$)	4177.0(6)	1828.4(5)	4104.4(3)
D_{calc} (g cm^{-3})	1.507	1.416	1.517
μ(Mo Kα) (mm^{-1})	2.381	0.595	2.479
Reflections collected	47972	14561	23211
Independent reflections	10377	3930	5073
R_{int}	0.0467	0.0290	0.0214
Final $R1$[a], $wR2$[b] ($I \geqslant 2\sigma$)	0.0319, 0.0680	0.0519, 0.1348	0.0418, 0.1166
GOF on F^2	1.002	1.004	1.000

[a] $R1 = \sum||F_o| - |F_c||/\sum|F_o|$.

[b] $wR2 = [\sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]]^{1/2}$.

$[Ni(HL^1)_2]·4H_2O·CH_3OH$ (**4**): yield, 82% (based on Ni). *Anal.* Calc. for $C_{37}H_{48}N_6NiO_9$ ($M = 779.28$): C, 57.01; H, 6.21; N, 10.78. Found: C, 56.76; H, 6.03; N, 10.33%. IR, cm^{-1}: 3362 ν(NH), 1652 *ν*(C=O). ESI-MS: *m/z*: 675.19 $[M–4H_2O–CH_3OH+H]^+$.

$[Ni(HL^2)_2]·4H_2O·CH_3OH$ (**5**): yield, 85% (based on Ni). *Anal.* Calc. for $C_{37}H_{46}Br_2N_6NiO_9$ ($M = 937.29$): C, 47.41; H, 4.95; N, 8.97. Found: C, 47.12; H, 4.63; N, 8.72%. IR, cm^{-1}: 3213 ν(NH), 1648 *ν*(C=O). ESI-MS: *m/z*: 832.21 $[M–4H_2O–CH_3OH+H]^+$.

2.4. X-ray structure determinations

X-ray quality single crystals of complexes **3–5** were immersed in cryo-oil, mounted in a Nylon loop and measured at a temperature of 100(2) (**3** and **4**) or 296(2) K (**5**) (Table 1). The diffraction experiments were carried out on a Bruker APEX-II CCD diffractometer. The program SHELXTL [24] was used for collecting frames of data, indexing reflections and for the determination of the lattice parameters, SAINTP [24] for integration of the intensity of reflections and scaling, SADABS [25] for absorption correction, and SHELXTL [26] for space group and structure determination, least-squares refinements on F_2. Positional and thermal parameters of the non-hydrogen atoms were refined in the least-squares cycles. All hydrogens were inserted in calculated positions. Least square refinements with anisotropic thermal motion parameters for all the non-hydrogen atoms and isotropic for the remaining atoms were employed.

3. Results and discussion

3.1. Synthesis and characterization of compounds 1–5

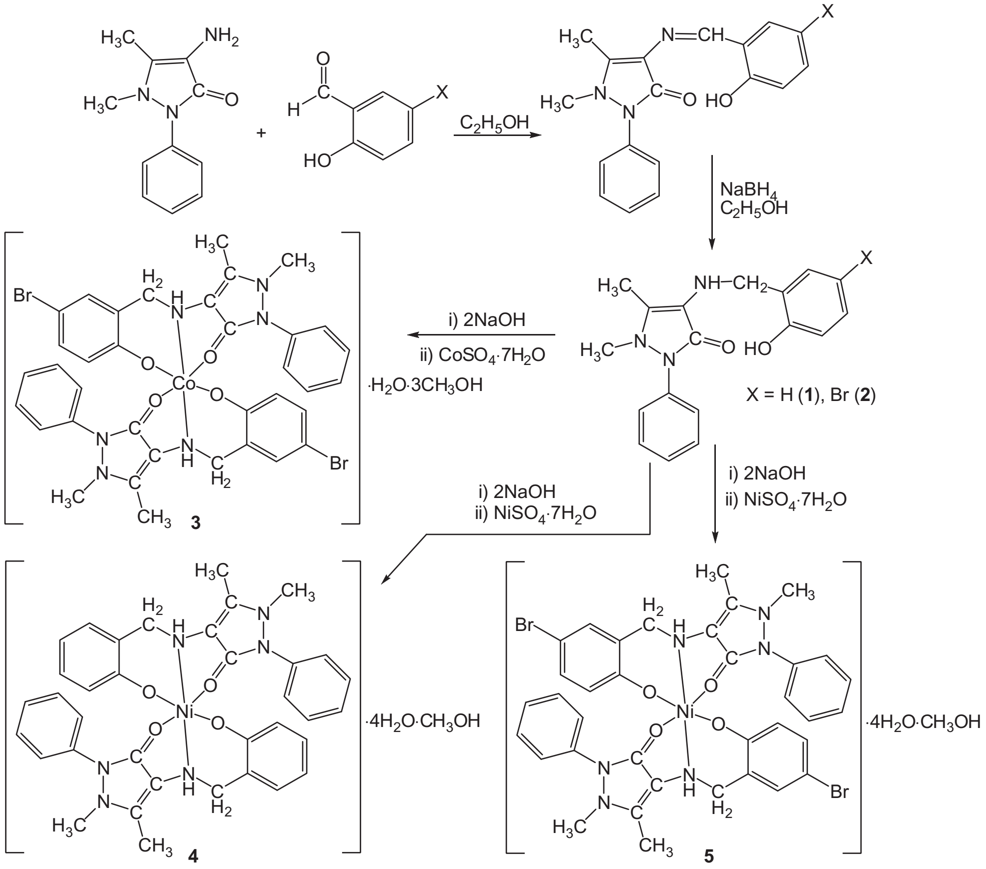
2-Hydroxy-benzyl derivatives of 4-aminoantipyrine, **1** and **2**, were prepared by the Schiff condensation of 4-aminoantipyrine and salicylaldehyde or its 5-bromo-derivative with subsequent reduction with $NaBH_4$ in ethanol. Dissolution of the ligands **1** and **2** followed by deprotonation and introduction of nickel(II) or cobalt(II) sulfates allowed complexes **3**–**5** to be isolated upon slow solvent evaporation as their crystalline solvates (Scheme 2).

Compounds **1** and **2** are colourless, while complexes $[Co(HL^2)_2]·H_2O·3CH_3OH$ (**3**), $[Ni(HL^1)_2]·4H_2O·CH_3OH$ (**4**) and $[Ni(HL^2)_2]·4H_2O·CH_3OH$ (**5**) are light brown, dark brown and light blue, respectively. In compounds **1** and **2**, the broad IR signals centred at 3360, 3480 and 3341, 3184 cm^{-1} can be ascribed to the OH and NH functional groups, respectively, while sharp and intense peaks at 1630 and 1638 cm^{-1} can be attributed to ν(C=O) stretching modes. Upon reduction, the characteristic IR signal for the azomethine group ν(C=N) at 1653 cm^{-1} [17] disappears; the disappearance of the characteristic C*H*=N peaks at *δ* 9.60–9.75 in the ^1H NMR spectra also confirms the reduction. The ^1H NMR peaks at *δ* 13.11 (for **1**) and 12.94 (for **2**) are attributable to the intramolecularly H-bonded phenolic OH group of the salicylaldehyde moiety. Moreover, $-CH_2-$ and N–H protons are observed at *δ* 4.23 and 11.08 (for **1**) and 4.32 and 11.42 (for **2**).

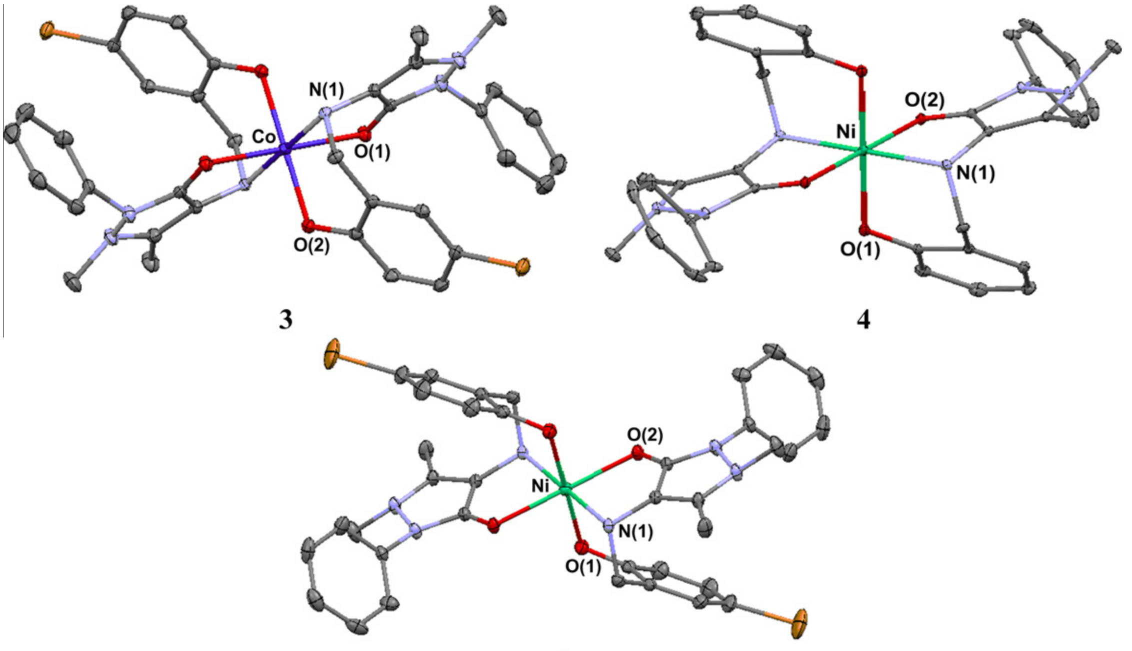
In the IR spectra of the complexes, the frequencies of the ν(C=O) bond (1650, 1652 and 1648 cm^{-1} for **3**, **4** and **5** respectively) are moved towards higher wavenumbers by approx. 14–20 cm^{-1} in comparison to the spectra of ligands (1630 and 1638 cm^{-1} for **1** and **2,** respectively), confirming coordination through the oxygen atom. The data of elemental analysis and ESI-mass spectroscopy are also in accordance with the proposed formulation.

3.2. Description of the X-ray crystal structures of compounds 3–5

Crystals of $[Co(HL^2)_2]·H_2O·3CH_3OH$ (**3**), $[Ni(HL^1)_2]·4H_2O·CH_3OH$ (**4**) and $[Ni(HL^2)_2]·4H_2O·CH_3OH$ (**5**) suitable for X-ray diffraction



Scheme 2. Synthesis of derivatives of 4-aminoantipyrine and their complexes.



3 **4** **5**

Fig. 1. Ellipsoid plots with the atom labelling scheme (ellipsoids are drawn at the 50% probability level) for complexes **3**–**5**. H atoms and crystallization solvent molecules in **3**–**5** are omitted for clarity.

Table 2
Selected distances (Å) and angles (°) of **3**–**5**.

3[i]		**4**[ii]		**5**[iii]	
Co(1)–O(1)	2.2032(14)	Ni(1)–O(1)	2.030(2)	Ni(1)–O(1)	2.0560(15)
Co(1)–N(1)	2.1774(16)	Ni(1)–N(1)	2.120(2)	Ni(1)–N(1)	2.1159(16)
Co(1)–O(2)	2.0206(14)	Ni(1)–O(2)	2.1701(18)	Ni(1)–O(2)	2.1434(14)
O(2)–Co(1)–O(2)#1	180.000(1)	O(1)–Ni(1)–O(1)#1	180.00(8)	O(1)–Ni(1)–O(1)#1	180.00(7)
O(2)–Co(1)–N(1)#1	89.10(6)	O(1)–Ni(1)–N(1)	91.69(8)	O(1)–Ni(1)–N(1)#1	88.32(6)
O(2)–Co(1)–N(1)	90.90(6)	O(1)–Ni(1)–N(1)#1	88.31(8)	O(1)–Ni(1)–N(1)	91.68(6)
N(1)–Co(1)–N(1)#1	180.000(1)	N(1)–Ni(1)–N(1)#1	180.00(10)	N(1)–Ni(1)–N(1)#1	180.000(1)
O(2)–Co(1)–O(1)	94.48(6)	O(1)–Ni(1)–O(2)	90.05(7)	O(1)–Ni(1)–O(2)	90.59(6)
N(1)–Co(1)–O(1)	82.32(6)	N(1)–Ni(1)–O(2)	83.63(7)	N(1)–Ni(1)–O(2)	84.40(6)
O(2)–Co(1)–O(1)#1	85.52(6)	O(1)–Ni(1)–O(2)#1	89.95(7)	O(1)–Ni(1)–O(2)#1	89.41(6)
N(1)–Co(1)–O(1)#1	97.68(6)	N(1)–Ni(1)–O(2)#1	96.37(7)	N(1)–Ni(1)–O(2)#1	95.60(6)
O(1)–Co(1)–O(1)#1	180.00(5)	O(2)–Ni(1)–O(2)#1	180.0	O(2)–Ni(1)–O(2)#1	180.000(1)

Symmetry transformations used to generate equivalent atoms: [i]#1 −x,−y + 1,−z + 2; [ii] #1 −x,−y + 1,−z; [iii] #1 −x + 3/2,−y + 1/2,−z + 1.

analysis were obtained upon recrystallization from methanol solution; schematic representations of **3**–**5** are shown in Scheme 2, while the ellipsoid plots are depicted in Fig. 1. In all the complexes, the positive charges of the metal ions are neutralized by the deprotonated phenoxy oxygens of $H_2L^{1,2}$. The metal atoms exhibit similar distorted octahedral environments, with two coordinated oxygen and one nitrogen atom from each of the $H_2L^{1,2}$ ligands, the axial positions being occupied by the oxygen atoms (Fig. 1).

In compound **3**, the O(1), N(1) and Co(1) atoms form the basal plane with the Co(1)–O(1) and Co(1)–N(1) distances being 2.2032(14) and 2.1774(16) Å, respectively (Table 2). The apical positions are occupied by two O(2) atoms with a Co(1)–O(2) distance of 2.0206(14) Å. The cobalt atom belongs to a fused five-membered Co1–N1–C1–C2–O1 and six-membered Co1–N1–C18–C13–C12–O2 metallacycle. A regular geometric configuration was found with O(2)–Co(1)–N(1)#1, O(2)–Co(1)–N(1), O(2)–Co(1)–O(1), N(1)–Co(1)–O(1), O(2)–Co(1)–O(1)#1 and N(1)–Co(1)–O(1)#1 angles of 89.10(6), 90.90(6), 94.48(6), 82.32(6), 85.52(6) and 97.68(6)°, respectively.

Complexes **4** and **5** consist of neutral $[Ni(HL^1)_2]$ and $[Ni(HL^2)_2]$ units, co-crystallized with four water and one methanol molecule. The nickel atoms in both complexes **4** and **5** also have a distorted octahedral geometry with two oxygen and one nitrogen atom of each ligand being coordinated. In both complexes, two N(1) and two O(2) and the Ni(1) atom form the basal plane, with Ni(1)–N(1) 2.120(2) and Ni(1)–O(2) 2.1701(18), and Ni(1)–N(1) 2.1159(16) and Ni(1)–O(2) 2.1434(14) Å for **4** and **5**, respectively. The apical positions are occupied by O(1) with a Ni(1)–O(1) distance of 2.030(2) and 2.0560(15) Å in **4** and **5**, respectively.

The bonding parameters in **3**–**5** are comparable to those of other metal complexes with SBA ligands [18]. Furthermore, owing to the presence of solvent (methanol and water) molecules, the overall crystal structure is stabilized by multiple O–H···O hydrogen bonds, which give rise to 1D chains (for example, see Fig. 2 for **4**). The non-coordinated water and methanol molecules form hydrogen bonds with coordinated carbonyl and phenoxy oxygens, and with the imino nitrogen atom. The average $H_2O\cdots O$, $H_2O\cdots N$, $CH_3HO\cdots O$ and $CH_3HO\cdots N$ distances of the hydrogen bonds in **3**–**5**

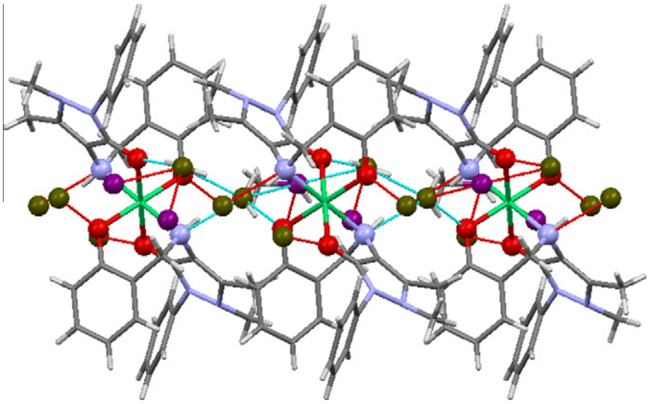


Fig. 2. 1D chain of **4** viewed along the *b* axis, with crystallization solvent (water, methanol) molecules represented by a "ball and stick" model. Intramolecular H-bonds are shown as blue and red dashed lines. Color codes: N (imino nitrogen, light blue), O (carbonyl and phenoxy oxygens, red), O (methanol, purple), O (water, olive green). (Color online.)

fall within the 2.602(4)–3.432(3) Å range, consistent with strong hydrogen bonding [27,28]. In accord with the geometry of the complexes, the channels incorporating the solvent molecules have an elongated profile. The crystal structures of **3**–**5** are stabilized by interactions between the coordinated carbonyl oxygen, phenoxy oxygen, imino nitrogen atoms and the solvent molecules, giving 1D networks in which the molecules are nested in such a way that close packing results. Thus, the supramolecular crystal packing of the complexes can be represented as chains extending parallel to the crystallographic c-axis (for **3**) and b-axis (for **4** and **5**). Because all the strong electron acceptors are involved in intrachain formation, only weak π-interactions support interchain agglomeration.

4. Conclusions

Three new Co^{II} and Ni^{II} complexes with 2-hydroxy-benzyl derivatives of 4-aminoantipyrine (synthesized by the Schiff condensation of salicylaldehyde and 4-aminoantipyrine with subsequent reduction) as ligands have been prepared and characterized by convenient methods, while their structures in the solid state have been determined by X-ray crystallography. The metal centres in the complexes have a distorted octahedral geometry, with the equatorial positions occupied by amino nitrogens and carbonyl oxygens of the 4-aminoantipyrine moieties, while the phenoxy oxygen atoms are seated at the apical positions. The number of crystallization solvent molecules and their arrangements in complexes **3**–**5** are related, leading to 1D hydrogen-bonding 1D networks involving O(w)–H and imine N, phenoxy and carbonyl O atoms.

Appendix A. Supplementary data

CCDC 872243–872245 contain the supplementary crystallographic data for **3**–**5**, respectively. These data can be obtained free of charge via 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; or e-mail: deposit@ccdc.cam.ac.uk.

References

[1] G. Rajendran, G.S. Sreeletha, Asian J. Chem. 13 (2001) 1142.
[2] G. Rajendran, G.S. Sreeletha, Asian J. Chem. 14 (2002) 1639.
[3] Y. Zheng, J. Lin, Y. Liang, Q. Lin, Y. Yu, C. Guo, S. Wang, H. Zhang, Mater. Lett. 54 (2002) 424.
[4] N. Raman, A. Kulandaisamy, A. Shunmugasundaram, K. Jeyasubramanian, Transition Met. Chem. 26 (2001) 131.
[5] R. Montalvo-Gonzalez, A. Ariza-Castolo, J. Mol. Struc. 655 (2003) 375.
[6] R.K. Agarwal, Transition Met. Chem. 32 (2007) 143.
[7] K.Z. Ismail, Transition Met. Chem. 25 (2000) 522.
[8] N. Raman, A. Kulandaisamy, C. Thangaraja, Transition Met. Chem. 29 (2004) 129.
[9] N. Raman, J.D. Raja, A. Sakthivel, J. Chem. Sci. 119 (2007) 303.
[10] G. Turan-Zitouni, M. Sıvacı, F.S. Kılıc, K. Erol, Eur. J. Med. Chem. 36 (2001) 685.
[11] T. Rosu, M. Negoiu, S. Pasculescu, E. Pahontu, D. Poirier, A. Gulea, Eur. J. Med. Chem. 45 (2010) 774.
[12] D. Burdulene, A. Palaima, Z. Stumbryavichyute, Z. Talaikite, Pharm. Chem. J. 33 (1999) 191.
[13] S. Chandra, D. Jain, A.K. Sharma, P. Sharma, Molecules 14 (2009) 174.
[14] T. Rosu, S. Pasculescu, V. Lazar, C. Chifiriuc, R. Cernat, Molecules 11 (2006) 904.
[15] X. Wang, Y. Zheng, Inorg. Chem. Commun. 10 (2007) 709.
[16] P. Mosae Selvakumar, E. Suresh, P.S. Subramanian, Polyhedron 26 (2007) 749.
[17] T. Rosu, E. Pahontu, C. Maxima, R. Georgescu, N. Stanica, A. Gulea, Polyhedron 30 (2011) 154.
[18] H. Liu, X. Shi, M. Xu, Z. Li, L. Huang, D. Bai, Z. Zeng, Eur. J. Med. Chem. 46 (2011) 1638.
[19] B. Sreenivasulu, M. Vetrichelvan, F. Zhao, S. Gao, J.J. Vittal, Eur. J. Inorg. Chem. (2005) 4635.
[20] C.B. Aakeröy, N.R. Champness, C. Janiak, CrystEngComm 12 (2010) 22.
[21] S.R. Batten, J. Solid State Chem. 178 (2005) 2475.
[22] Z.-T. Li, J.-L. Hou, C. Li, Acc. Chem. Res. 41 (2008) 1343.
[23] L.L. Koh, J.D. Ranford, W.T. Robinson, J.O. Stevenson, A.L.C. Tan, D. Wu, Inorg. Chem. 35 (1996) 6466.
[24] SMART & SAINT Software Reference Manuals, Version 6.22, Bruker AXS Analytic X-ray Systems, Inc., Madison, WI, 2000.
[25] G.M. Sheldrick, SADABS Software for Empirical Absorption Correction, University of Göttingen, Germany, 2000.
[26] SHELXTL Reference Manual, Version 5.1, Bruker AXS, Analytic X-ray Systems, Inc., Madison, WI, 1997.
[27] P. Gilli, V. Bertolasi, L. Pretto, A. Lycka, G. Gilli, J. Am. Chem. Soc. 124 (2002) 13554.
[28] P. Gilli, L. Pretto, V. Bertolasi, G. Gilli, Acc. Chem. Res. 42 (2009) 33.