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Cobalt(II) and nickel(II) complexes of thiomethylated anilines: Characterization and *in-vitro* antimicrobial studies

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Abstract

The cobalt(II) (1A-6A) and nickel(II) (1B-6B) complexes of the thiomethylated anilines substituted with -H (1), $-CH_3$ (2), $-OCH_3$ (3), -Cl (4), -Br (5) and $–NO_2$ (6) were synthesized and characterized by elemental analyses, FTIR, UV-Vis spectra and conductance measurements. The experimental elemental analyses of the compounds were in agreement with calculated values, resulting to $[ML_2Cl_2]$ (M = Co, Ni) stoichiometries of the complexes. Infrared spectra showed the bidentate ligands were coordinated to the metal ions through the nitrogen and sulphur atoms. The electronic spectra of the ligands exhibited $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions in the ranges 262–271 nm and 298–315 nm, which were shifted to the longer wavelengths of 265–278 nm and 302–328 nm in the metal complexes respectively. The $d \rightarrow d$ bands in the visible region of the spectra suggested octahedral geometries for these complexes. The conductance values of the metal complexes in DMF in the ranges 28.6–35.8 and 68.1–74.3 Ω^{-1} cm^2 mol^{-1} indicated their behavior as nonelectrolytes (cobalt(II)) and 1:1 electrolytes (nickel(II)). The compounds were evaluated for *in-vitro* antimicrobial activity against *S. aureus, B. subtilis, E. coli* and *C. albicans* using the agar disc diffusion technique. The Gram-positive bacteria were more susceptible to the test compounds than the Gram-negative bacterium and the fungus. The cobalt(II) complexes demonstrated better inhibitory activity (8–14 mm) against the microbial organisms compared to the parent ligands (7–13 mm) and the nickel(II) complexes (8–10 mm).

Keywords: Cobalt(II); nickel(II); thiomethylated anilines; characterization; *in-vitro* antimicrobial

Introduction

Anilines substituted with the thiomethyl group at the *ortho* position are potential bidentate ligands with hard-borderline nitrogen and soft sulphur donor atoms with a tendency to form strong chelates with the borderline cobalt(II) and nickel(II) [1]. These ligands are useful intermediates in production of many organic compounds, which are themselves precursors of certain reactions. By reduction with Raney nickel [2], they can be used to generate *ortho*-methylated anilines, which are useful intermediates in production of dyes, rubber, herbicides [3], as well as in electro-optical and many other industrial processes. The biological relevance of cobalt and nickel ions in living organisms necessitated their choice for complexation with the thiomethylated aniline ligands. Though a few reports are available on the metal(II) complexes of some of these ligands, the spectral and antimicrobial properties have not been documented [4]. In this study, the cobalt(II) and nickel(II) complexes of thiomethylated anilines substituted with the methyl, methoxy, chloro, bromo and nitro groups have been prepared, and the spectral data and *in-vitro* antimicrobial activity of these metal(II) complexes are reported.

Materials and Methods

Materials and physical measurements

The reagents and solvents of analytical grade were purchased from commercial suppliers and were used as received. CHNS analyses were determined on ElementarAnalysensystemevarioMICRO V1.6.2 GmbH. The melting points (uncorrected) were determined on Galenkemp melting point apparatus. The mid-infrared spectra of the compounds were obtained on PerkinElmer Spectrum

100 ATR-FTIR spectrometer (4000–650 cm^{-1} region). Far-infrared spectra of the metal complexes were obtained as mulls on Perkin Elmer Spectrum 400 FTIR/FIR spectrometer (700–30 cm^{-1}). The electronic spectra were obtained in DMF (10^{-3} M) on a PerkinElmer Lambda 25 UV/VIS spectrometer (250–1100 nm). Molar conductance measurements were carried out in DMF (10^{-3} M) on AZ® 86555 pH/mV/Cond./TDS/Temp instrument. Room temperature magnetic susceptibility measurements were obtained from Sherwood magnetic susceptibility balance. The ligands 1–6 were synthesised and characterised as previously reported [5, 6].

Synthesis of Co(II) and Ni(II) complexes

Cobalt(II) complex, 1A, was prepared by adding an ethanolic solution of 1 (0.06 g, 0.38 mmol) to $CoCl_2.6H_2O$ (0.04 g, 0.15 mmol) in 2mL ethanol, the mixture was stirred at room temperature for 4 h to ensure complete reaction. The light pink precipitate was obtained by filtration, washed with ethanol and ethylacetate, dried under vacuum and weighed as 0.13 g. The cobalt(II) complexes, 2A-6A, were similarly prepared.

Nickel(II) complex, 2A, was prepared by adding an ethanolic solution of 2 (0.06 g, 0.38 mmol) to $NiCl_2.6H_2O$ (0.04 g, 0.15 mmol) in 2 mL ethanol, the mixture was stirred at room temperature for 4 h during which a green precipitate formed. The compound was obtained by filtration, washed with ethanol and ethyl acetate, dried under vacuum and weighed as 0.14 g. The nickel(II) complexes, 2B-6B, were similarly prepared. The analytical data of the cobalt(II) and nickel(II) complexes were recorded in Table 1.

Table 1. Analytical data for Co(II) and Ni(II) complexes.

Complexes/	Mol.	Colour	M. pt	Yield C	H	N	S	
Formula	Wt		(°C)	%	Found (Calculated)			%
[$C_{16}H_{22}Cl_2CoN_2S_2$] 1A	436.33	Pink	130-132	79	43.92 (44.04)	5.42 (5.08)	6.08 (6.42)	14.61 (14.70)
[$C_{16}H_{22}Cl_2N_2NiS_2$] 1B	436.09	Green	200-202	85	44.02 (44.07)	5.31 (5.08)	6.45 (6.42)	14.15 (14.71)
[$C_{18}H_{26}Cl_2CoN_2S_2$] 2A	464.38	Pink	155-157	95	46.99 (46.55)	6.05 (5.64)	5.98 (6.03)	13.67 (13.81)
[$C_{18}H_{26}Cl_2N_2NiS_2$] 2B	464.14	Green	212-214	86	46.82 (46.58)	5.85 (5.65)	5.95 (6.04)	13.53 (13.82)
[$C_{18}H_{28}Cl_2CoN_2O_3S_2$] 3A	496.38	Purple	130-133	50	42.34 (42.03)	4.79 (5.49)	5.50 (5.45)	11.32 (12.47)
[$C_{18}H_{30}Cl_2N_2NiO_4S_2$] 3B	496.14	Green	220-222	45	41.50 (40.62)	5.82 (5.68)	5.11 (5.26)	10.30 (12.05)
[$C_{16}H_{20}Cl_4CoN_2S_2$] 4A	505.22	Pink	178-180	46	38.33 (38.04)	4.02 (3.99)	5.51 (5.54)	12.36 (12.69)
[$C_{16}H_{20}Cl_4N_2NiS_2$] 4B	504.98	Green	220-222	80	37.64 (38.06)	4.01 (3.99)	5.36 (5.55)	12.09 (12.70)
[$C_{16}H_{20}Br_2Cl_2CoN_2S_2$] 5A	594.12	Pink	176-178	55	32.97 (32.35)	3.37 (3.39)	4.78 (4.72)	10.38 (10.79)
[$C_{16}H_{20}Br_2Cl_2N_2NiS_2$] 5B	593.88	Green	230-232	89	31.71 (32.36)	3.21 (3.39)	4.58 (4.72)	9.11 (10.80)
[$C_{16}H_{20}Cl_2CoN_4O_4S_2$] 6A	526.32	Yellow	> 200	55	36.64 (36.51)	3.82 (3.83)	10.65 (10.64)	12.18 (12.18)
[$C_{16}H_{20}Cl_2N_4NiO_4S_2$] 6B	526.08	Yellow	204-206	87	36.56 (36.53)	3.95 (3.83)	10.49 (10.65)	11.66 (12.19)

Antimicrobial Susceptibility Testing

The ligands, cobalt(II) and nickel(II) complexes were screened in vitro for their antibacterial activity against Staphylococcus aureus ATCC 6538, Bacillus subtilis (subsp. spizizenii) ATCC 6633, Escherichia coli ATCC 8739 and for antifungal activity against Candida albicans ATCC 2091. Ampicillin (AMP) and ketoconazole (KTZ) were used as positive controls for the antibacterial and antifungal tests respectively. All the growth media (Mueller Hinton agar (MHA), agar bacteriological, potato dextrose agar (PDA) and nutrient broth) were prepared according to standard procedure [7]. Agar disc diffusion method was employed to determine the susceptibility of the microorganisms to the test compounds [8, 9]. The preparation of the agar plates, culturing of the microbial strains, the inoculation of the plates and preparation of drugs stock solutions followed described procedure [10, 11]. Each microbial inoculum was standardized by reference to 0.5McFarland turbidity standard [7].

Results and discussion

General chemistry

The synthesis and characterisation of ligands 1-6 have been previously reported [6]. The cobalt(II) and nickel(II) complexes were obtained as pink (or purple) and green solids respectively. The elemental analyses of the metal(II) complexes were in close agreement with their predicted values, with stoichiometry [ML_2Cl_2] (M = Co, Ni). Yields of the compounds were in the range 45-95%. The metal complexes are soluble in common organic solvents such as ethanol, methanol, acetonitrile, tetrahydrofuran,*N*,*N'*-dimethylformamide and *N*,*N'*-dimethylsulphoxide. The uncorrected melting points of the complexes are in the range 130–230°C with the exception of 6A (>200°C). The analytical data for the metal complexes were recorded in Table 1.

Infrared spectra

The important infrared vibrational frequencies of the ligands and metal compounds are compared in Table 2. In the spectra of the ligands, two intense bands in the ranges 3450–3398 cm^{-1} and 3352–3307 cm^{-1} were attributed to v(N–H) asymmetric and symmetric modes respectively. The frequencies of these bands were lowered in the metal complexes to 3308–3256 cm^{-1} and 3229–3198 cm^{-1} respectively [12]. Reduction in frequencies could be a result of the electron density of the nitrogen being directed to the metal ion, leaving the amino protons less tightly bound to the nitrogen [13]. In the cobalt and nickel complexes substituted with H (1A, 1B), CH_3 (2A, 2B), OCH_3 (3A, 3B), these N–H bands underwent a decreased and an increased intensity respectively. In the complexes containing the electron withdrawing groups (5A, 5B, 6A, 6B) the intensity of the bands generally was seen to decrease. The bend mode, δNH_2, and the v(C–N) stretch of the amine group had absorptions in the ranges 1639–1618 cm^{-1} and 1293–1272 cm^{-1} respectively in the ligands. These bands were shifted to lower frequencies in the metal complexes in the ranges 1624–1596 cm^{-1} (δNH_2) and 1273–1250 cm^{-1} (vC–N). The reduction in v(C–N) on complexation is expected as a result of decrease in the C–N double bond character, indicating the binding of the ligands through the nitrogen atom. Bands arising from v(C–S) in the ligands appeared with weak to medium intensities between 696 cm^{-1} and 674 cm^{-1}. They were shifted to lower frequencies in the metal(II) complexes in the range 682–670 cm^{-1}. The reduced frequency of the C–S stretch was a consequence of the binding of the ligands through the sulphur atom. In the far infrared regions, new bands of medium intensities, which were absent in the spectra of the ligands have been observed in the spectra of the metal complexes. These were assigned as v(M–N) (446–424 cm^{-1}), v(M–Cl) (338–321 cm^{-1}) and v(M–S) (236–221 cm^{-1}) [14-16].

Table 2. IR frequencies of important bands (cm^{-1})

Entry	$v_{asy, sym}$(N–H)	δNH_2, v(C–N)	v(C–S)	v(M–N)	v(M–S)	v(M–Cl)	
1	3424, 3352	1618, 1272	691	----		---	----
1A	3281, 3218	1610, 1251	674	426	321	221	
1B	3278, 3221	1611, 1253	675	428	323	231	
2	3420, 3346	1625, 1275	688	----	----	----	
2A	3273, 3214	1600, 1269	678	444		327	224
2B	3268, 3212	1600, 1264	680	446		328	231
3	3409, 3341	1626, 1293	689	----		----	----
3A	3308, 3229	1596, 1273	680	438		338	231
3B	3290, 3223	1606, 1272	682	430		324	231
4	3399, 3307	1625, 1275	685	----		----	----
4A	3268, 3212	1615, 1250	678	446		336	227
4B	3272, 3218	1610, 1252	681	446		328	231
5	3398, 3317	1624, 1275	687	----		----	----
5A	3269, 3211	1606, 1250	678	442		332	222
5B	3269, 3215	1607, 1251	678	444		322	230
6	3450, 3347	1639, 1278	696	----		----	----
6A	3271, 3227	1624, 1255	670	424		337	231
6B	3256, 3198	1624, 1255	676	424		338	236

Electronic spectra and molar conductance

The electronic spectral data of the ligands and the metal complexes were recorded in Table 3. The ligands 1–5 exhibited a weak band at 262–271 nm and a more intense band in the range 298–324 nm. These bands were respectively assigned to $\pi \rightarrow \pi^*$ transition within the aryl ring and $n \rightarrow \pi^*$ transition of the lone electrons to the aryl ring. The ligand transitions were also displayed by the metal(II) complexes in the ranges 265–278 nm and 302–328 nm respectively. Co(II) complexes comprised of two distinct moderate bands in the visible region with the lower energy transition occurring in the range 604–612 nm, and was designated as $^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$ (v_3) transition. Absorptions between 672 and 679 nm were assigned to the $^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$ (v_2) transition, and the weak bands in the near infrared region occurring in the range 910–925 nm were attributed to $^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$ (v_1) transition. These bands corresponded to those assigned in Co(II) octahedral systems [17]. The electronic spectra of Ni(II) complexes consisted of an intense band in the range 410–430 nm, assigned to the transition $^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$ (v_3). The transitions that appeared as moderately intense bands in the regions 622–627 nm and 688–701 nm were as a result of $^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$ (v_2) and $^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$ (v_1) respectively. Other weak transitions were observed as shoulders in the ranges 578–588 nm and 766–786 nm and were assigned as the $^3A_{2g}(F) \rightarrow {}^1T_{2g}(P)$ and $^3A_{2g}(F) \rightarrow {}^1A_{1g}(G)$ transitions respectively. The v_1, v_2, v_3 transitions observed in these complexes are indicative of nickel(II) ion in an octahedral environment[17].

The nitro-substituted metal complexes also had additional bands at 382 and 440 nm (6A) and at 380 nm (6B), which have been assigned as ligand to metal transitions. The molar conductance values of the cobalt(II) complexes in DMF were found in the range 28.6–35.8 Ω^{-1} cm^2 mol^{-1}, indicating their non-electrolytic behavior and suggesting the chlorine species were covalently bonded to the metal ions within the coordination sphere. Though various ranges have been quoted for 1:1 electrolytes [18, 19], an average and reasonable value was compiled as approximately 65–90 Ω^{-1} cm^2 mol^{-1} [20]. Nickel(II) complexes showed conductance values in the range 68.1–74.3 Ω^{-1} cm^2 mol^{-1}, demonstrating their 1:1 electrolytic property in DMF [20]. This could be as a result of the displacement of a chloride anion outside the coordination sphere.

Table 3. Electronic spectra of compounds and conductance of metal complexes in DMF

Entry	Wavelength [λ_{max}, nm](ε, mol^{-1} dm^3 cm^{-1})		Λ_M ^{a}
	$\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$	$d \rightarrow d$	
1	265, 298	----	-----
1A	273 (682), 307 (3210)	612 (180), 678 (300), 924 (1)	29.9
1B	274 (101),302 (517)	419 (11), 587 (1), 627 (2), 700 (4), 766 (4)	69.2
2	264, 305	----	-----
2A	273 (485), 312 (3190)	611 (360), 679 (585), 923 (1.5)	35.8
2B	273 (370), 321 (1920)	418 (19), 585 (7), 625 (9), 689 (6), 769 (6)	69.5
3	265, 315	----	-----
3A	265 (8800), 315 (9540)	604 (900), 672 (550)	33.2
3B	275 (428), 328 (4615)	422 (40), 578 (18), 624 (18), 690 (16), 786 (9)	69.4
4	262, 311	----	-----
4A	274 (680), 315 (3060)	612(215), 678 (355), 923 (3.3)	28.6
4B	273 (176), 314 (940)	417 (11), 588 (0.3), 625 (2), 701 (4), 774 (3)	69.5
5	263, 311	----	-----
5A	276 (985), 316 (3230)	611 (300), 678(490), 925 (1.3)	30.8
5B	278 (620), 328 (2370)	410 (24),587 (1),625 (8), 699 (8), 773 (4)	74.3
6	271, 315, 382	----	-----
6A	275 (2570), 315 (4080) 382 (31000)	440 (1890), 612(317), 678 (520), 910 (2.5)	28.8
6B	275 (3520), 315 (7460) 380 (53765)	430 (485), 585 (7), 622 (9), 688 (8), 775 (6)	68.1

^{a} units = Ω^{-1} cm^2 mol^{-1} ^{b} units = B.M.

Antimicrobial Susceptibility Tests

The antimicrobial susceptibility test results of the synthesized compounds were recorded in Table 4. The inhibitory activity of the tested compounds was compared with that of standard antibacterial drug, ampicillin (23–40 mm), and antifungal drug, ketoconazole (23 mm). The Gram-positive bacteria, Staphylococcus aureus and Bacillus subtilis, were more susceptible to the synthesized compounds than the Gram-negative bacterium, Escherichia coli, and the fungus, Candida albicans. The cobalt(II) complexes showed inhibitory zones at 8–14 mm against the microbial organisms compared to the parent ligands (7–13 mm) and the nickel(II) complexes (8–10 mm). The methoxy-substituted ligand (3) and its cobalt(II) complex (3A) demonstrated the highest activity among this series against the Gram-positive bacteria, in the range 12-14 mm.

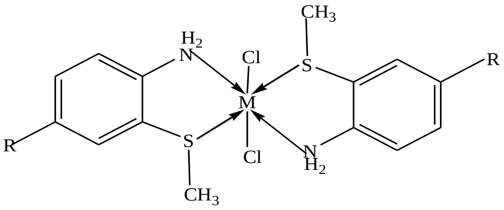
Table 4. Agar disc inhibition zones (mm) at 250 µg/disc

Compounds	***S. aureus***	***B. subtilis***	***E. coli***	***C. albicans***
1	8	7	6	6
1A	11	11	6	6
1B	8	8	7	7
2	8	8	7	6
2A	9	8	6	6
2B	8	10	6	6
3	13	12	7	6
3A	14	13	7	6
3B	9	10	6	6
4	8	8	7	6
4A	10	12	7	9
4B	8	8	6	7
5	8	9	7	6
5A	10	10	7	9
5B	9	8	6	6
6	7	9	7	6
6A	10	13	7	9
6B	8	7	6	6
DMF	6	6	6	6
AMP^a	40	38	23	--
KTZ^a	--	--	--	23

^{a}125 µg/disc

Conclusion

The cobalt(II) and nickel(II) complexes of the thiomethylated anilines containing the methyl, methoxy, chloro, bromo and nitro substituents were synthesized and obtained as $[ML_2Cl_2]$ based on elemental analyses. The infrared spectra revealed the ligands were coordinated to the metal ions as bidentate neutral ligands through their nitrogen and sulphur atoms. The electronic spectra of the metal complexes consisted of $\pi \rightarrow \pi^*$and $n \rightarrow \pi^*$ transitions. The observed $d \rightarrow d$ bands were consistent with octahedral Co(II) and Ni(II) geometries. The cobalt complexes behaved as neutral non-electrolytic compounds in DMF, indicating the chloride ions were bound to the metal ions within the coordination sphere. The nickel complexes demonstrated the 1:1 electrolyte behaviour in DMF, suggesting the presence of one chloride anion outside the coordination sphere. The suggested structures of the metal complexes in the solid state are presented in Figure 1. The evaluation of the compounds as potential antimicrobial agents, by agar disc diffusion, revealed that *B. subtilis* and *S. aureus* were more susceptible to the test compounds in comparison to *E. coli* and *C. albicans*. The cobalt(II) complexes, and especially, the methoxy-substituted compound, demonstrated the best inhibitory activity among the series of compounds tested.



M = Co, Ni
R = H, CH_3, OCH_3, Cl, Br, NO_2

Figure 1. The structures of the metal(II) complexes

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