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Complexes of Nickel(II) with 2,3-Dimethyl-4-formyl(benzhydrazide)-1-phenyl-3-pyrazolin-5-one

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ABSTRACT

Nickel(II) complexes of the Schiff base 2,3-dimethyl-4-formyl-(benz-hydrazide)-1-phenyl-3-pyrazolin-5-one (L) with the formulae $[Ni(L)_2 (CIO_4)]CIO_4$, $[Ni(L)_2(NO_3)](NO_3)$, and $[Ni(L)_2X_2]$ (X = Cl, Br, or I) have been synthesized and characterized by elemental analyses, electrical conductance in nonaqueous solvents, infrared and electronic spectra as well as magnetic susceptibility measurements. In these complexes, L acts as a neutral bidentate ligand coordinating through the azomethine nitrogen and the carbonyl oxygen of the pyrazolone ring. In the perchlorate and nitrate complexes one of the anions is coordinated to the metal ion in a bidentate fashion while in the halide complexes both the anions are coordinated. An octahedral geometry is assigned around the nickel(II) ion in all these complexes.

Key Words: Nickel(II); Schiff base; Hydrazines; Metal complex.

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INTRODUCTION

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The interesting and varied ligand behavior of hydrazines and hydrazones towards transition metal ions is well known.^[1] The pharmacological applications of antipyrine-based ligands and their metal complexes have drawn the attention of many investigators.^[2-4] In view of these and as part of our continuing interest on metal complexes^[5-10] with Schiff bases derived from antipyrine, we report here the synthesis and structural investigation on nickel(II) complexes of the Schiff base 2,3-dimethyl-4-formyl(benzhydrazide)-1-phenyl-3-pyrazolin-5-one (L). (Fig. 1)

RESULTS AND DISCUSSION

The formation of the complexes can be represented by the general equation shown below:

$$NiX_2 + 2L \rightarrow Ni(L)_2X_2$$
 (X = ClO₄, NO₃, Cl, Br, or I)

All the complexes are light yellow in color and are nonhygroscopic. They are soluble in acetonitrile, DMF, ethanol, methanol, and nitrobenzene, but insoluble in benzene, chloroform, ethyl acetate, and toluene. The analytical data (Table 1) indicate that the complexes have the general formula:

 $Ni(L)_2X_2$ (X = ClO₄, NO₃, Cl, Br, or I)

The molar conductance values of the complexes (Table 2) in acetonitrile, DMF, and nitrobenzene $(10^{-3} \text{ M solution})$ are in the range expected for 1 : 1 electrolyte for perchlorate and nitrate complexes while the corresponding chloride, bromide, and iodide complexes behave as nonelectrolytes.^[11]



Figure 1. 2,3-Dimethyl-4-formyl(benzhydrazide)-1-phenyl-3-pyrazolin-5-one (L).

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	Table 1.	Analytical d	lata ^a of the	nickel(II) co	omplexes of	L.			
Compound	Molecular formula	Formula weight	Metal (%)	Anion (%)	Carbon (%)	Hydrogen (%)	Nitrogen (%)	Yield (%)	$\underset{({}^{\circ}C)}{M.p.}$
L	$C_{19}H_{18}N_4O_2$	334.39	I	I	67.98 (68.25)	5.48 (5.43)	16.70 (16.76)	85	198
$[Ni(L)_2(CIO_4)](CIO_4)$	$C_{38}H_{36}Cl_2N_8NiO_{12}$	926.39	6.31 (6.34)	21.39 (21.47)	49.20 (49.26)	3.90 (3.92)	12.04 (12.10)	87	202
$[Ni(L)_2(NO_3)](NO_3)$	$C_{38}H_{36}N_{10}NiO_{10}$	851.50	6.82 (6.89)		53.52 (53.60)	4.20 (4.26)	16.41 (16.45)	91	262
$[Ni(L)_2Cl_2]$	$C_{38}H_{36}Cl_2N_8NiO_4$	798.40	7.30 (7.35)	8.82 (8.88)	57.09 (57.16)	4.58 (4.54)	14.00 (14.04)	88	292
$[Ni(L)_2Br_2]$	$\mathrm{C}_{38}\mathrm{H}_{36}\mathrm{Br}_{2}\mathrm{N}_{8}\mathrm{NiO}_{4}$	887.31	6.59 (6.62)	18.00 (18.01)	51.40 (51.43)	4.04 (4.09)	12.58 (12.63)	86	288
$[Ni(L)_2I_2]$	$C_{38}H_{36}I_2N_8NiO_4$	981.30	5.92 (5.98)	25.80 (25.86)	46.46 (46.50)	3.68 (3.70)	11.39 (11.42)	89	238
^a Calculated values in p	arentheses.								

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		Molar conductan	ce
Compound	Acetonitrile	DMF	Nitrobenzene
$[Ni(L)_2(ClO_4)](ClO_4)$	153.72	85.64	27.45
$[Ni(L)_2(NO_3)](NO_3)$	148.23	82.35	25.25
$[Ni(L)_2Cl_2]$	71.37	34.04	7.68
$[Ni(L)_2Br_2]$	93.33	35.14	10.98
$[Ni(L)_2I_2]$	94.42	41.72	12.08

Table 2. Molar conductance^a data of the nickel(II) complexes of L ($ohm^{-1} cm^{2} mol^{-1}$).

 $^{a}10^{-3}$ M solution.

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Thus, the complexes may be formulated as $[Ni(L)_2(ClO_4)](ClO_4)$, $[Ni(L)_2(NO_3)](NO_3)$, and $[Ni(L)_2X_2]$ (X = Cl, Br, or I).

Infrared Spectra

The important infrared spectral data of the ligand and its complexes are presented in Table 3. The infrared spectral bands at 1674 and 1630 cm⁻¹ of the ligand are characteristic of ν (C=O) of the pyrazolone ring and benzoyl moiety, respectively.^[7] In complexes, the band at 1674 cm⁻¹ is shifted to the region 1640–1657 cm⁻¹ indicating that the carbonyl oxygen is involved in coordination.^[7] The band corresponding to ν (C=O) of the benzoyl moiety remains practically unaltered at about 1625 cm⁻¹ indicating the noncoordination of benzoyl carbonyl in all these complexes.^[12] The strong band observed at 1571 cm⁻¹ in the ligand spectrum attributed to C=N stretching vibration is shifted to 1551–1558 cm⁻¹ indicating the coordination of azomethine nitrogen in the complexes.^[7] The ν (N–N) band at 998 cm⁻¹ in the ligand spectrum shifted to higher frequency (1006–1008 cm⁻¹) in the complexes which further supports the bonding through azomethine nitrogen.^[13]

In the perchlorate complex, the triply split band maxima observed at 1142, 1110, and 1020 cm⁻¹ are due to ν_8 , ν_6 , and ν_1 , vibrations, respectively, of the perchlorate group of $C_{2\nu}$ symmetry, which is indicative of bidentate coordination,^[14] but the band around 1089 cm⁻¹ is assigned to the ν_3 vibration of the ionic perchlorate of T_d symmetry. Thus, the presence of both ionic and bidentately coordinated perchlorate groups is indicated in these complexes.^[14] The vibrational frequencies observed at 940 and 636 cm⁻¹ corresponding to ν_2 and ν_3 vibrations, respectively, of the perchlorate ($C_{2\nu}$ group) group and the band at 625 cm⁻¹ due to the ν_4 vibration of the ionic perchlorate, which are not present in the spectrum of the free ligand, also supports the coexistence

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	Table 3.	Important IR spectral ba	inds (cm^{-1}) of L	and its nickel con	nplexes.	
L	$[\mathrm{Ni}(\mathrm{L})_2(\mathrm{CIO}_4)](\mathrm{CIO}_4)$	$[Ni(L)_2(NO_3)](NO_3)$	$[Ni(L)_2Cl_2]$	$[Ni(L)_2Br_2]$	$[Ni(L)_2I_2]$	Assignment
1,674 s	1,641 s	1,657 s	1,643 s	1,643 s	1,640 s	ν (C=0) ring
1,630 s	1,625 s	1,624 s	1,626 s	1,625 s	1,626 s	$\nu(C=0)$
1,571 s	1,558 s	1,552 s	1,552 s	1,554 s	1,551 s	$\nu(C=N)$
998 w	1,006 w	1,008 w	1,010 w	1,008 w	1,008 w	$\nu(N-N)$
	1,142 m	I				v ₈ -Coordinated ClO ₄
	1,110 s					v ₆ -Coordinated ClO ₄
	1,020 s	I				v ₁ -Coordinated ClO ₄
	940 w					ν_2 -Coordinated ClO ₄
	636 w	I				ν_3 -Coordinated CIO ₄
	1089 s	I				ν_3 -Ionic ClO ₄
	625 m	I				ν_4 -Ionic ClO ₄
		$1,440 \mathrm{m}$				v ₄ -Coordinated NO ₃
		1,247 m				ν_1 -Coordinated NO ₃
		1,052 m				ν_2 -Coordinated NO ₃
		1,384 s				ν_3 -Ionic NO ₃
		830 s				ν_2 -Ionic NO ₃
			240 w			$\nu(Ni-CI)$
				165 w		$\nu(Ni-Br)$
					125 w	$\nu(Ni-I)$
	450 w	452 w	452 w	450 w	452 w	$\nu(Ni-N)$
	548 w	550 w	552 w	550 w	548 w	$\nu(\rm Ni-O)$
Notes: $s = s$	trong, $m = medium$, $w = v$	weak.				



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of both ionic and bidentately coordinated perchlorate groups in these complexes.

In the nitrate complex, characteristic vibrational frequencies of both ionic and coordinated nitrate groups are observed. The presence of a coordinated nitrate group is indicated by the bands observed at 1,440 and 1,247 cm⁻¹ due to the v_4 and v_1 vibrations, respectively, of the nitrate group of C_{2V} symmetry.^[16] A medium band observed at 1052 cm⁻¹ due to the v_2 vibration of the nitrate group (C_{2V}), stands as additional evidence for the presence of coordinated nitrate ion. The difference in wave numbers between the two highest frequency bands ($v_4 - v_1$) of the C_{2V} nitrate is about 193 cm⁻¹, which indicates that the coordinated nitrate ion is bidentate.^[15,16] A very strong band at 1384 cm⁻¹ indicates the presence of an ionic nitrate group and it is due to the v_3 vibration of the nitrate group of D_{3h} symmetry. This is supported by another band of medium intensity observed around 830 cm⁻¹ due to the v_2 vibration of the ionic nitrate.^[15]

In the far infrared spectrum of chloride, bromide, and iodide complexes, Ni–Cl, Ni–Br, and Ni–I stretching vibrations occur at 240, 165, and 125 cm^{-1} , respectively, which are not present in the spectrum of the free ligand.

The above results, along with the conductance data, confirm that in these complexes one of the perchlorate and nitrate ions and two of the chloride, bromide, and iodide ions are coordinated to the metal ion. Furthermore, $\nu(Ni-N)$ and $\nu(Ni-O)$ stretching vibrations are observed at about 550 and 450 cm⁻¹, respectively, in all these complexes.^[17]

Electronic Spectra

Electronic spectral data of the ligand and complexes in the solid state (using Nujol) with their tentative assignments are given in Table 4. The electronic spectrum of the ligand shows two maxima at 23,866 and 41,153 cm⁻¹ corresponding to $n \to \pi^*$ and $\pi \to \pi^*$ transitions, respectively. In the complexes $n \to \pi^*$ bands are found to be blue-shifted to the region 24,510–25,839 cm⁻¹ while $\pi \to \pi^*$ bands are red-shifted to the region 40,160–41,052 cm⁻¹, respectively, compared to that of the ligand. The bands observed in the regions 21,551–21,978 cm⁻¹ and 15,527– 16,891 cm⁻¹ are attributed to ${}^{3}A_{2g}(F) \to {}^{3}T_{1g}(P)$ and ${}^{3}A_{2g}(F) \to {}^{3}T_{1g}(F)$ consistent with octahedral geometry around nickel(II) in these complexes.^[18,19] In addition to this, a low energy band in the region 9191– 9541 cm⁻¹ attributable to ${}^{3}A_{2g}(F) \to {}^{3}T_{2g}(F)$ supports the octahedral geometry around nickel(II) in these complexes.^[18,19] An intense absorption band in the region 32,573–33,989 cm⁻¹ may be due to a charge transfer process.



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Table 4. Electronic spectral data and magnetic moments of the nickel(II) complexes of L.

Compound	Abs. max. (cm^{-1})	Tentative assignment	$\mu_{\rm eff}$ (B.M.)
L	23,866 41,153	$egin{array}{ccc} n \longrightarrow \pi^* \ \pi \longrightarrow \pi^* \end{array}$	
[Ni(L) ₂ (ClO ₄)](ClO ₄)	25,839 40,650 32,573 21,929 15,576 9,505	$\begin{array}{c} n \longrightarrow \pi^{*} \\ \pi \longrightarrow \pi^{*} \\ \text{Charge transfer} \\ {}^{3}A_{2g}(F) \longrightarrow {}^{3}T_{1g}\left(P\right) \\ {}^{3}A_{2g}(F) \longrightarrow {}^{3}T_{2g}\left(F\right) \\ {}^{3}A_{2g}(F) \longrightarrow {}^{3}T_{2g}\left(F\right) \end{array}$	3.02
[Ni(L) ₂ (NO ₃)](NO ₃₎	25,125 40,160 33,364 21,739 15,600 9,191	$\begin{array}{c} n \longrightarrow \pi^{*} \\ \pi \longrightarrow \pi^{*} \\ \text{Charge transfer} \\ {}^{3}A_{2g}(F) \longrightarrow {}^{3}T_{1g}\left(P\right) \\ {}^{3}A_{2g}(F) \longrightarrow {}^{3}T_{2g}\left(F\right) \\ {}^{3}A_{2g}(F) \longrightarrow {}^{3}T_{2g}\left(F\right) \end{array}$	3.30
[Ni(L) ₂ Cl ₂]	24,630 40,485 32,573 21,551 15,576 9,310	$n \rightarrow \pi^{*}$ $\pi \rightarrow \pi^{*}$ Charge transfer ${}^{3}A_{2g}(F) \rightarrow {}^{3}T_{1g}(P)$ ${}^{3}A_{2g}(F) \rightarrow {}^{3}T_{1g}(F)$ ${}^{3}A_{2g}(F) \rightarrow {}^{3}T_{2g}(F)$	3.27
[Ni(L) ₂ Br ₂]	25,510 40,650 33,898 21,978 15,527 9,354	$\begin{array}{c} n \longrightarrow \pi^{*} \\ \pi \longrightarrow \pi^{*} \\ \text{Charge transfer} \\ ^{3}A_{2g}(F) \longrightarrow {}^{3}T_{1g}\left(P\right) \\ ^{3}A_{2g}(F) \longrightarrow {}^{3}T_{1g}\left(F\right) \\ ^{3}A_{2g}(F) \longrightarrow {}^{3}T_{2g}\left(F\right) \end{array}$	2.98
[Ni(L) ₂ I ₂]	24,813 41,052 33,444 21,929 16,891 9,541	$\begin{array}{c} n \longrightarrow \pi^{*} \\ \pi \longrightarrow \pi^{*} \\ \text{Charge transfer} \\ {}^{3}A_{2g}(F) \longrightarrow {}^{3}T_{1g}\left(P\right) \\ {}^{3}A_{2g}(F) \longrightarrow {}^{3}T_{1g}\left(F\right) \\ {}^{3}A_{2g}(F) \longrightarrow {}^{3}T_{2g}\left(F\right) \end{array}$	3.12

Magnetic Moment Data

The magnetic moments of the nickel(II) complexes are in the range 2.98 to 3.30 B.M. Typical octahedral nickel(II) complexes have magnetic moments between 2.90 and 3.40 B.M. depending on the magnitude of the



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orbital contribution.^[20] The results indicate that these complexes probably have octahedral geometry.^[21,22]

Based on the results of the present study, the structure shown in Fig. 2 may be assigned to the complexes.

EXPERIMENTAL

Materials

The metal salts were prepared from Analar BDH nickel carbonate and the respective 50% acids and the salts were crystallized out by evaporating the solution on a steam bath. For example NiCl₂·6H₂O was prepared as follows. Nickel carbonate was dissolved in hot 50% hydrochloric acid. A slight excess of carbonate was used. The undissolved carbonate was removed by filtration. The filtrate was concentrated by evaporation when crystals of NiCl₂·6H₂O separated.

Preparation of the Ligand

The ligand 2,3-dimethyl-4-formyl(benzhydrazide)-1-phenyl-3-pyrazolin-5-one was prepared and characterized as described earlier.^[7]

Preparation of Complexes

Perchlorate and Iodide Complexes

A quantity of 0.3657 g (1 mmol) of Ni(ClO₄)₂ · 6H₂O or 0.4206 g (1 mmol) of NiI₂·6H₂O in ethyl acetate (10 mL) was added to a boiling suspension of 0.7357 g (2.2 mmol) of the ligand in ethyl acetate (100 mL). The resulting mixture was refluxed for about 4 hr on a steam bath. The complex precipitated on cooling and was filtered and washed several times with hot benzene to remove the excess ligand. The complexes were dried under vacuum over phosphorus(V)oxide.

Chloride Complex

A quantity of 0.2377 g (1 mmol) of NiCl₂·6H₂O in methanol (10 mL) was added to a boiling suspension of 0.7357 g (2.2 mmol) of the ligand in ethyl acetate (100 mL). The resulting mixture was refluxed for about 3 hr on a



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steam bath and concentrated. The complex precipitated on cooling and was filtered and washed several times with hot benzene to remove the excess ligand. The complex was dried under vacuum over phosphorus(V)oxide.

Bromide and Nitrate Complex

A quantity of 0.3266 g (1 mmol) of NiBr₂ \cdot 6H₂O or 0.2908 g (1 mmol) of Ni(NO₃)₂ \cdot 6H₂O in acetone (10 mL) was added to a boiling suspension of 0.7257 g (2.2 mmol) of the ligand in acetone (100 mL). The mixture was refluxed for about 3 hr on a steam bath. The resulting solution was concentrated. The complexes precipitated on cooling and were filtered and washed several times with hot benzene to remove the excess ligand. The complexes were dried under vacuum over phosphorus(V)oxide.

ANALYSES ON PHYSICAL METHODS

All the complexes were analyzed for metal and halide contents by the standard methods^[23] and the perchlorate content by the Kurz method.^[24] The molar conductances of the complexes in acetonitrile, DMF, and nitrobenzene (10^{-3} M solution) were measured at room temperature using a Toshniwal conductivity bridge with a dip-type conductance cell (cell constant = 1.094). The infrared spectra of the ligand and complexes were recorded in the range 4000–400 cm⁻¹ on a Shimadzu IR–470 spectrometer. Electronic spectra of the ligand and the complexes in the solid state were measured in the range 200–1100 nm on a Shimadzu UV 160A spectrophotometer and in the range 1100–1500 nm on a Cary 2390 UV-VIS-NIR spectrophotometer. The elemental analyses (C, H, and N) were carried out in a Heraeus CHNO rapid analyzer. The magnetic moments were measured at room temperature by the Guoy method using mercury(II)tetrathiocyanatocobaltate(II) as the calibrant.

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REFERENCES

- Pelizzi, C.; Pelizzi, G.; Predieri, G.; Resolva, S. Investigation into aroylhydrazones as chelating agents. J. Chem. Soc. (Dalton Trans.) 1982, 1349.
- Singh, L.; Mohan, G.; Parashar, R.K.; Tripathi, S.P.; Sharma, R.C. Studies on antinflammatory activity of some lanthanone complexes of bioactive organic molecules. Curr. Sci. **1986**, *55*, 846–848.
- Alice, C.J.; Prabhakaran, C.P. Synthesis and characterization and biological studies of Pd(II) complexes of some ON and OS donors. Indian. J. Chem. 1990, 29A, 491–493.
- Alaudeen, M.; Abraham, A.; Radhakrishnan, P.K. Synthesis and antibacterial activity of rare-earth-perchlorate complexes of 4-(2'-hydroxynaphthylazo)-antipyrine. Proc. Indian. Acad. Sci. (Chem. Sci.) 1995, 107, 123–126.
- Joseph, S.; Radhakrishnan, P.K. Yttrium and lanthanide perchlorate complexes of *N*,*N'-bis*(4-antipyrylmethylidene)ethylenediamine. Synth. React. Inorg. Met.-Org. Chem. **1998**, 28 (3), 423–435.
- Madhu, N.T.; Radhakrishnan, P.K. Nickel(II) complexes of 1,2-di(imino-4'-antipyrinyl)ethane and 4-N-(4'-antipyrylmethylidene)aminoantipyrine. Synth. React. Inorg. Met.-Org. Chem. 2000, 30 (8), 1561–1579.
- Ajithkumar, G.; Radhakrishnan, P.K. Yttrium and lanthanide nitrate complexes of 2,3-dimethyl-4-formyl(benzhydrazide)-1-phenyl-3-pyrazolin-5-one. Synth. React. Inorg. Met.-Org. Chem. 2002, 32 (4), 831–842.
- Raju, K.C.; Radhakrishnan, P.K. Complexes of copper(II) with *N*,*N'-bis*(4-antipyrylmethylidene)ethylenediamine. Synth. React. Inorg. Met.-Org. Chem. 2002, *32* (6), 1117–1128.
- Raju, K.C.; Radhkrishnan, P.K. Complexes of cobalt(II) with N,N'-bis(4antipyrylmethylidene)ethylenediamine. Synth. React. Inorg. Met.-Org. Chem. 2002, 32 (10), 1719–1728.
- Raju, K.C.; Radhakrishnan, P.K. Complexes of iron(III) with *N*,*N'-bis*(4-antipyrylmethylidene)ethylenediamine. Synth. React. Inorg. Met.-Org. Chem. 2003, *33* (1), 23–33.
- Geary, W.J. The use of conductivity measurements in organic solvents for the characterization of coordination compounds. Coord. Chem. Rev. 1971, 7, 81–115.
- Kumar, P.A. Synthesis and spectral studies of some seven-coordinated rare earth metal complexes. Synth. React. Inorg. Met.-Org. Chem. 1997, 27 (4), 577–588.
- Singh, B.; Narang, K.K.; Srivastava, R. Studies on complexes of cobalt(II), nickel(II), copper(II) and cadmium(II) with acetophenone and 4-hydroxyacetophenone oxalolyl dihydrazones. Synth. React. Inorg. Met.-Org. Chem. 2001, 31 (8), 1387–1401.



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- 14. Scholar, R.P.; Merbach, A.E. Raman and infrared study of hexamethyl phosphoramide complexes of lanthanide perchlorates. Inorg. Chim. Acta. **1975**, *15*, 15–20.
- Gatehouse, B.M.; Livingstone, S.E.; Nijholm, R.S. Infrared spectra of some nitrates and other oxy-anion coordination complexes. J. Inorg. Nucl. Chem. 1958, 8, 75–78.
- Nakamato, K. Coordination compounds. In *Infrared and Raman Spectra* of *Inorganic and Coordination Compounds*, 4th Ed.; John Wiley and Sons, Inc.: New York, 1986; 254–257.
- Donia, A.M.; El-Saied, F.A. Complexes of manganese(II), cobalt(II), and nickel(II) with the keto form of some antipyrine Schiff base derivatives. Polyhedron **1988**, 7 (21), 2149–2153.
- Lever, A.B.P. Electronic spectra of dⁿ ions. In *Inorganic Electronic Spectroscopy*, 2nd Ed.; Elsevier: Amsterdam, 1984; 513–518.
- Rao, S.; Reddy, H.K. Complexes of chromium(III), cobalt(II), nickel(II), copper(II), and molybdenum(VI) with indane1,3-dione-2-isonicotinoylhydrazone. Indian J. Chem. **1992**, *31* (A), 58–60.
- Cotton, F.A.; Wilkinson, G. Advanced Inorganic Chemistry, 5th Ed.; Wiley-Interscience Publication: New York, 1988; 631–635.
- Patel, K.S.; Ikekwere, P.O. Complexes of nickel(II) sulphate with some cyclocarboxylicacidhydrazides. Synth. React. Inorg. Met.-Org. Chem. 1990, 20 (1), 39–53.
- Agarwal, R.K.; Garg, P.; Agarwal, H.; Chandra, S. Synthesis, magnetospectral and thermal studies of cobalt(II) and nickel(II) complexes of 4-[N-(4-dimethylaminobenzylidene)amino]antipyrine. Synth. React. Inorg. Met.-Org. Chem. **1997**, *27* (2), 251–268.
- Basett, J.; Denny, R.C.; Jeffery, G.H.; Mendhem, J. Titrimetric analysis. In Vogels Text Book of Quantitative Inorganic Analysis, 5th Ed.; ELBS: London, 1961; 341–379.
- 24. Kurz, E.; Kober, G.; Berl, M. Determination of perchlorates by fusion with nitrite. Anal. Chem. **1958**, *30* (12), 1983–1986.

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