



## “SYNTHESIS AND CHARACTERIZATION OF Cr, Mo, W METAL CARBONYL COMPLEXES DERIVED FROM ISONICOTINOYLHYDRAZIDE SCHIFF BASES”

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### Abstract:

We have synthesized six complexes  $\{M(CO)_4[HMINH]\}$  1a-3a, (1a, M = Cr, 2a, M = Mo, 3a, M = W),  $\{M(CO)_4[HINH]\}$  1b-3b (1b, M = Cr, 2b, M = Mo, 3b, M = W) by photochemical displacement of two CO group from  $M(CO)_6$  by *N'*-(2-hydroxy-3-methoxybenzylidene)isonicotinohydrazide [HMINH] and *N'*-(2-hydroxy benzylidene)isonicotinohydrazide [HINH] derived from isonicotinohydrazide. The synthesized complexes exhibit variable degree of antibacterial activity. The complexes have been characterized by elemental analysis, IR, <sup>1</sup>H-NMR spectroscopy and magnetic studies. The spectroscopic studies suggest a bidentate behavior of the ligand via azomethine-N and phenolic-O donor atoms with the metal (0).

### KEYWORDS-

Schiff bases, coordination chemistry, isonicotinohydrazide, antibacterial activity.

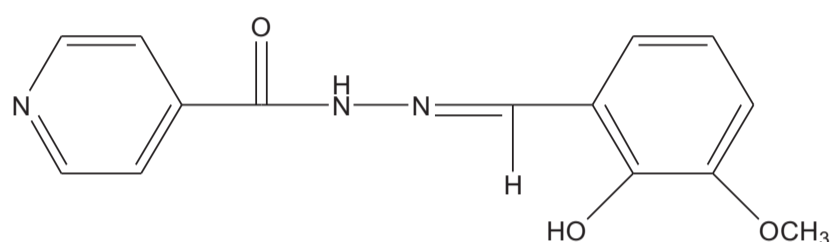
### INTRODUCTION

A Schiff base is a functional group that contains a carbon-nitrogen double bond with the nitrogen atom connected to an aryl or alkyl group but not hydrogen. Schiff bases are of the general formula  $R_1R_2C=N-R_3$ , where  $R_3$  is an aryl or alkyl group that makes the Schiff base a stable imine. Monodentate Schiff's bases are less known to form stable complexes, probably due to the insufficient basic strength of the imino nitrogen of the C=N group. Polydentate Schiff bases with phenolic OH or nitrogen or sulphur of the ring, suitably near to the imino nitrogen, may stabilize the metal-nitrogen bond through the formation of chelate rings. Schiff bases which are unaided by those donors but contain two azomethine nitrogen atoms, in spite of their facile ligating capabilities due to easy chances of chelation, have also been used in the CO displacement reactions of group VI metal carbonyls[1]. Isonicotinylhydrazine (INH), is an organic compound that is the first-line medication in prevention and treatment of tuberculosis. The compound was first synthesized in the early 20<sup>th</sup> century [2], but its activity against tuberculosis was first reported in the early 1950s, and three pharmaceutical companies attempted unsuccessfully to simultaneously patent the drug [3]. With the introduction of isonicotinylhydrazine, a cure for tuberculosis was first considered reasonable. Isonicotinylhydrazine is available in tablet, syrup, and injectable forms (given intramuscularly

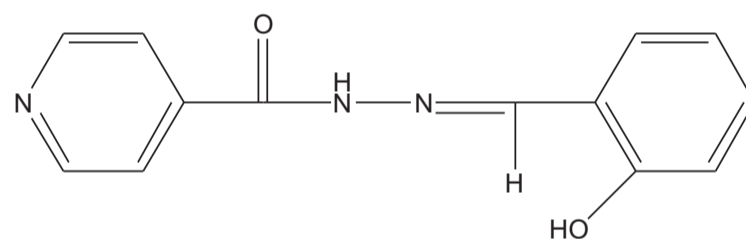
Title: “SYNTHESIS AND CHARACTERIZATION OF Cr, Mo, W METAL CARBONYL COMPLEXES DERIVED FROM ISONICOTINOYLHYDRAZIDE SCHIFF BASES” Source: Review of Research [2249-894X] AMIT SINGHAL<sup>1</sup>, MANISH KAUSHIK<sup>2</sup>, MUNESH KUMAR<sup>3</sup>, MANOJ AGARWAL<sup>4</sup> yr:2013 vol:2 iss:9

or intravenously). It is available worldwide, is inexpensive and is generally well tolerated. It is manufactured from isonicotinic acid, which is produced from 4-methylpyridine [4].

Metal-organic complexes containing bridged ligands are of current interest because of their continued interest due to their interesting molecular structure and their different functionalities [5-11]. We have synthesized complexes of the type  $\{M(CO)_4(L)\}$  1a-3a, 1b-3b (1a, M = Cr, L = HMINH; 2a, M = Mo, L = HMINH; 1c, M = W, L = HMINH; 1b, M = Cr, L = HINH; 2b, M = Mo, L = HINH; 3b, M = W, L = HINH) has been synthesized by photochemical displacement of two CO groups in group-6 metal carbonyls with by *N'*-(2-hydroxy-3-methoxybenzylidene)isonicotinohydrazide [HMINH] and *N'*-(2-hydroxybenzylidene)isonicotinohydrazide [HINH]. Both the ligands & their complexes were characterized on the basis of physical properties, elemental analysis data, magnetic studies, infrared and nuclear magnetic resonance spectroscopy. It was found that the ligands have bidentate nature coordinating through azomethine-N and phenolic-O donor atoms. Thus replacing two CO group to maintain charge density on metal centre.



*N'*-(2-hydroxy-3-methoxybenzylidene)isonicotinohydrazide [HMINH]



*N'*-(2-hydroxybenzylidene)isonicotinohydrazide [HINH]

## EXPERIMENTAL

Reactions were carried out under dry argon or in vacuo. All solvents were dried and degassed prior to use. Infrared spectra were recorded on a Perkin-Elmer spectrophotometer (Model-577) in KBr discs and  $CH_2Cl_2$ . All the melting points were determined in an open capillary and are uncorrected. All glassware was oven dried at 120°C, molecular weight of the complexes were determined cryoscopically in benzene, cyclooctadiene (COD), n-hexane, n-pentane, benzene were purchased from E. Merck, and  $M(CO)_6$  (M = Cr, Mo, W), dichloromethane were purchased from Aldrich and were used as supplied.

Magnetic susceptibility measurements of the complexes were carried out by Gouy method. UV irradiation were performed with a medium pressure 400W mercury lamp through a quartz bulb.

## SYNTHESIS OF THE LIGANDS-

Synthesis of ligand *N'*-(2-hydroxy-3-methoxybenzylidene)isonicotinohydrazide [HMINH]

The ligand *N'*-(2-hydroxy-3-methoxybenzylidene)isonicotinohydrazide [HMINH] was prepared by literature method [12] as followed. 0.15 g, (1.0 mmol) of 3-methoxysalicylaldehyde was refluxed for 30min with 0.14 g, (1.0 mmol) isonicotinozylhydrazide in 20ml methanol. The pale yellow solid separated by filtration, washed with methanol and dried. Yield 70.5%. Analysis Calc. for  $C_{14}H_{13}N_3O_3$  Mol. Wt. 271.27, C, 61.99; H, 4.83; N, 15.49 found Mol. Wt. 271.1, C, 61.07; H, 4.30; N, 15.19.

**Scheme**

Synthesis of ligand N'-(2-hydroxybenzylidene)isonicotinohydrazide [HINH]

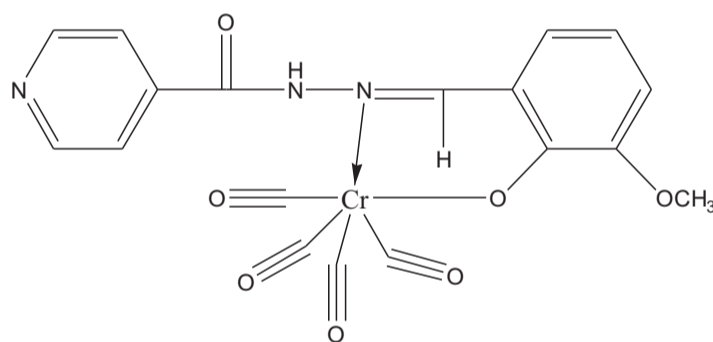
The ligand N'-(2-hydroxybenzylidene)isonicotinohydrazide [HINH] was prepared literature method [12] as followed. 0.11 g, (1.0 mmol) of salicylaldehyde was refluxed for 30min with 0.11 g, (1.0 mmol) isonicotinozylhydrazide in 20ml methanol. The light yellow solid separated by filtration, washed with methanol and dried. Yield 72.5%. Analysis Calc. for C<sub>13</sub>H<sub>11</sub>N<sub>3</sub>O<sub>3</sub> Mol. Wt. 241.25, C, 64.72; H, 4.60; N, 17.42 found Mol. Wt. 241.09, C, 64.12; H, 4.20; N, 17.09.

**Preparation of complexes**

Complexes {M(CO)<sub>4</sub>[L]} were prepared by the photochemical reactions of M(CO)<sub>6</sub> (M=Cr, Mo, W) with N'-(2-hydroxy-3-methoxybenzylidene)isonicotinohydrazide [HMINH] and N'-(2-hydroxybenzylidene)isonicotinohydrazide [HINH] and obtained in 75-80% yield by similar methods of which the following in typical.

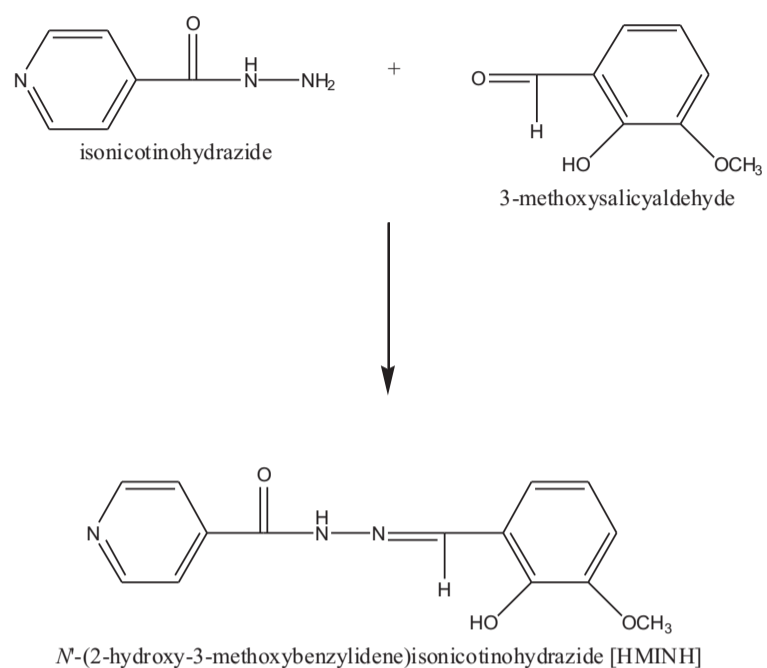
**Preparation of complex {Cr(CO)<sub>4</sub>[HMINH]}**

A quantity of 0.22 g (1.0 mmol) of Cr(CO)<sub>6</sub> was dissolved in 50ml of n-Heptane. Then, 0.27 g (1.0mmole) of N'-(2-hydroxy-3-methoxybenzylidene)isonicotinohydrazide [HMINH] was added to the above solution with constant stirring. The mixture was irradiated for 14 hrs. During the irradiation, the color of the reaction mixture changed from red to brown colour. After the irradiation, the reaction mixture was evaporated under vacuum, yielding a deep brown solid. The solid residue was then washed with n-hexane so as to remove any Cr (CO)<sub>6</sub>, ligand itself, and other n-hexane soluble impurities. The deep brown residue or remnant was dissolved in 1:1 CH<sub>2</sub>Cl<sub>2</sub>/n-hexane solution for recrystallization.



Complex (1a){Cr(CO)<sub>4</sub>[HMINH]}

Scheme 1 – Preparation of Ligand [HMINH]: Reaction of isonicotinohydrazide with 3-methoxysalicylaldehyde



Scheme 2– Preparation of Complexes: Reaction of *N*'-(2-hydroxy-3-methoxybenzylidene)isonicotinohydrazide [HMINH] with Cr(CO)<sub>6</sub>

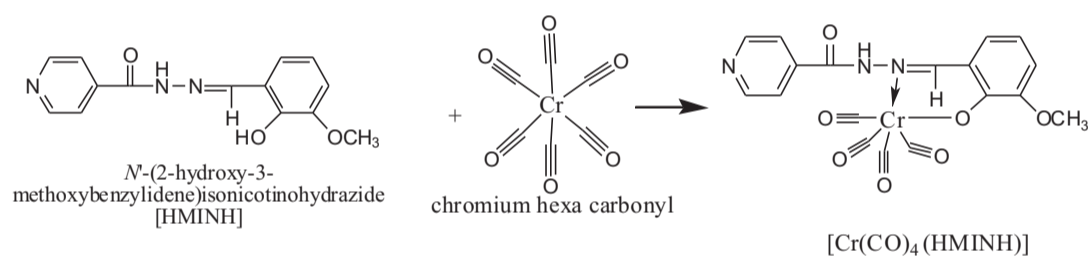


Table 1a – Physical and analytical data of Ligands

Ligands	Molecular Formula	Yield (%)	M.p. (°C)	Colour	Found (Calcd.) (%)			Mol. Wt.
					C	H	N	
a [HMINH]	C <sub>14</sub> H <sub>13</sub> N <sub>3</sub> O <sub>3</sub>	70.5	155	Pale Yellow	61.2 (62.0)	4.3 (4.8)	15.2 (15.5)	271.1 (271.3)
b [HINH]	C <sub>13</sub> H <sub>11</sub> N <sub>3</sub> O <sub>3</sub>	72.5	151	Light yellow	64.1 (64.7)	4.2 (4.6)	17.1 (17.4)	241.1 (241.3)

**Table 1 – Physical and analytical data of Complexes**

Complexes	Molecular Formula	Yield (%)	M.p. (°C)	Colour	Found (Calcd.) (%)			Mol. Wt.
					C	H	N	
1a	C <sub>18</sub> H <sub>12</sub> CrN <sub>3</sub> O <sub>7</sub>	75	165	Light Yellow	49.0 (49.3)	3.5 (3.7)	9.3 (9.6)	438.0 (438.3)
2a	C <sub>18</sub> H <sub>12</sub> MoN <sub>3</sub> O <sub>7</sub>	72	168	Yellow	44.3 (44.8)	3.0 (3.3)	8.3 (8.7)	482.3 (484.0)
3a	C <sub>18</sub> H <sub>12</sub> WN <sub>3</sub> O <sub>7</sub>	79	172	Yellow	37.5 (37.9)	2.3 (2.8)	7.1 (7.4)	570.0 (570.2)
1b	C <sub>17</sub> H <sub>10</sub> CrN <sub>3</sub> O <sub>6</sub>	76	167	Light Yellow	49.2 (50.0)	3.2 (3.5)	10.1 (10.3)	408.0 (408.3)
2b	C <sub>17</sub> H <sub>10</sub> MoN <sub>3</sub> O <sub>6</sub>	78	170	Yellow	44.8 (45.2)	2.8 (3.1)	9.0 (9.3)	452.3 (454.0)
3b	C <sub>17</sub> H <sub>10</sub> WN <sub>3</sub> O <sub>6</sub>	76	176	Cream	37.3 (38.1)	1.4 (1.9)	7.3 (7.8)	535.5 (536.1)

**Table 2a. Selected IR bands (cm<sup>-1</sup>) and (1H)-NMR (ppm) data of ligands**

Ligand	Selected IR bands (cm <sup>-1</sup> )				<sup>1</sup> H-NMR (in CDCl <sub>3</sub> ) δ ppm
	Imine ν (C=N)	amine ν (NH)	Hydrazine ν (N-N)	Phenolic ν (OH)	
a [HMINH]	1626	3201	998	3438	12.27 (s, 1H, OH), 10.74 (s, 1h, NH), 8.7 (s, 1H, azomethine C-H), 3.83 (s, 3H, O-CH <sub>3</sub> )
b [HINH]	1622	3206	993	3432	12.23 (s, 1H, OH), 10.73 (s, 1h, NH), 8.6 (s, 1H, azomethine CH)

Table 2b. Selected IR bands (cm<sup>-1</sup>) and (1H)-NMR (ppm) data of complexes

Complex	Selected IR bands (cm <sup>-1</sup> )				<sup>1</sup> H-NMR (in CDCl <sub>3</sub> ) δ ppm
	v (CO)	Imine v (C=N)	amine v (NH)	Hydrazine v (N-N)	
1a	2068, 2017, 1958, 1934	1597	3205	1022	10.5 (s, 1h, NH), 9.05 (s, 1H, azomethine C-H), 3.82 (s, 3H, O-CH <sub>3</sub> )
2a	2067, 2015, 1957, 1932	1599	3206	1025	10.45 (s, 1h, NH), 9.02 (s, 1H, azomethine C-H), 3.81 (s, 3H, O-CH <sub>3</sub> )
3a	2068, 2017, 1958, 1934	1607	3200	1022	10.47 (s, 1h, NH), 9.04 (s, 1H, azomethine C-H), 3.83 (s, 3H, O-CH <sub>3</sub> )
1b	2067, 2015, 1957, 1932	1604	3201	1033	10.48 (s, 1h, NH), 9.04 (s, 1H, azomethine C-H)
2b	2071, 2015, 1952, 1934	1598	3206	1031	10.47 (s, 1h, NH), 9.04 (s, 1H, azomethine C-H)
3b	2069, 2013, 1949, 1930	1594	3205	1027	10.47 (s, 1h, NH), 9.04 (s, 1H, azomethine C-H)

## RESULTS AND DISCUSSION

The ligands were prepared by condensation reaction of isonicotinozylhydrazide with corresponding methoxysalicylaldehyde or salicylaldehyde according to Scheme-1. Complexes (1a-3c) were prepared by photochemical reaction as shown in Scheme-2. Analytical data for {M(CO)<sub>4</sub>[HMINH1]} (1a-3a) and {M(CO)<sub>4</sub>[HINH]} (1b-3b) where (M= Cr, Mo & W); complexes are given in Table-1.

In this study, photochemical reactions of M(CO)<sub>6</sub> (M=Cr, Mo & W) with N'-(2-hydroxy-3-methoxybenzylidene) isonicotinohydrazide [HMINH] and N'-(2-hydroxy benzylidene) isonicotino hydrazide [HINH] ligands occurs in expected manner, and gave hither to a series of complexes (1a)-(3a) and (1b)-(3b) occur via the displacement of two CO from M(CO)<sub>6</sub> (M=Cr, Mo & W) and co-ordination of metal atom via deprotonated phenolic-O, and azomethine-N donor atoms yielding M(CO)<sub>4</sub>L complexes.

The i.r. spectra of ligands and the corresponding complexes provide information about the metal-ligand bonding. Important IR spectral bands M(CO)<sub>4</sub>L (L = N'-(2-hydroxy-3-methoxybenzylidene)isonicotinohydrazide [HMINH] and N'-(2-hydroxybenzylidene)isonicotinohydrazide [HINH]; M=Cr, Mo & W) are presented in Table-2b. The evidence about metal-imine nitrogen (M-N) bond formation is the shifting of C=N vibration found at 1590 cm<sup>-1</sup> in free ligand, shifts to lower wavelength in complexes 1a-3c, showing that the ligand coordinate to metal via the imine donor atom.[13]. The stretching vibrations found at 3020cm<sup>-1</sup> in free ligand, shifts to lower wavelength in complexes showing that the ligand is coordinating via amine donor atom.

A broad band centered at  $3201\text{ cm}^{-1}$  in the free ligand can be assigned to the  $\nu\text{N-H}$  stretching [14]. In the complexes this band remains unaltered, suggesting non-participation of NH group in bonding. The IR spectrum of the free ligand at  $3438\text{ cm}^{-1}$  and  $1353\text{ cm}^{-1}$  due to phenolic OH becomes absent in complexes, indicating deprotonation of phenolic OH on coordination with metal ion [15]. The weak band observed at  $500\text{ cm}^{-1}$  and  $430\text{ cm}^{-1}$  in complexes have been assigned to M-N and M-O bonds respectively [16].

The  $^1\text{H-NMR}$  spectrum of the free ligand shows signal due to OH at  $\delta$  (12.27) characteristic of phenolic OH proton [17, 18] which is absent in the complex, suggesting coordination through deprotonated phenolic oxygen. Signal due to NH at  $\delta$  (10.74) in free ligand appears at  $\delta$  (10.5) in the complex [19]. Presence of NH proton in the complex indicates that the ligand exists in the keto form. A singlet at  $\delta$  (8.7) observed in the spectrum for the free ligand shows downfield shift to  $\delta$  (9.05) indicates coordination of azomethine nitrogen in the complexes [17, 18], due to reduction of electron density at the azomethine C-H. Four bands in the range ( $2068\text{--}2072\text{ cm}^{-1}$ ), ( $2013\text{--}2018\text{ cm}^{-1}$ ), ( $1949\text{--}1958\text{ cm}^{-1}$ ) and ( $1932\text{--}1936\text{ cm}^{-1}$ ) arising from  $\nu$  (CO) vibrations are seen which presumably have local  $c_{2v}$  symmetry of  $\text{M}(\text{CO})_4$  unit in  $\{\text{M}(\text{CO})_4\text{SB}^1\}$  (1a-3a);  $\{\text{M}(\text{CO})_4\text{SB}_2\}$  (1b-3b) and  $\{\text{M}(\text{CO})_4\text{SB}^3\}$  (1c-3c);  $\{\text{M}(\text{CO})_4\text{SB}^4\}$  (1d-3d) and  $\{\text{M}(\text{CO})_4\text{SB}^5\}$  (1e-3e); where (M= Cr, Mo & W); complexes (Scheme-2). These values are in close resemblance to the values of  $\nu$  (CO) vibration for other nitrogen containing disubstituted group-6 metal carbonyls [20-23]. The presence of normal ligand bands indicated that these bands were intact in the complexes. The nature and number of CO bands resemble closely to the bands of other known trisubstituted metal carbonyls [24, 25].

In addition, magnetic susceptibility measurement shows that (1a-3e) complexes were diamagnetic. Since these complexes have  $\text{M}(0)$  [M=Cr, Mo, W] with a low spin  $d_6$  configuration. Such diamagnetism might arise from further splitting of the d-orbital in the low symmetry complexes i.e.  $d_{xy}^2$ ,  $d_{xz}^2$ ,  $d_{yz}^2$ ,  $d_{(x^2-y^2)}$ ,  $d_z^2$  [26, 27].

#### Antibacterial Activity

All the synthesized complexes were screened for their antibacterial activity by using agar diffusion method [28] against *S. aureus*, *B. subtilis* gram positive and *E. coli*, *S. paratyphi* gram negative bacteria in nutrient agar medium. Ciprofloxacin was used as standard drug for comparison.

These Schiff's Bases and their metal carbonyl complexes show antibacterial activity against *E. coli*, *S. aureus*, *B. subtilis* and *S. paratyphi*. It is found that 4-bromo-2-(2-methyl aminoethyl imino)methyl]phenol [ $L^1$ ] studied here follow such a decomposition scheme in the cell, and diazoalkane intermediates further could alkylate the nucleophilic centres of biologically important macromolecules and in particular DNA. 4-bromo-2-(2-dimethylaminoethylimino)methyl]phenol [ $L^2$ ], 4-bromo-2-(2-diethylaminoethylimino)methyl]phenol [ $L^3$ ] shows moderate activity. With the help of activity data it could be observed that complexes  $\text{M}(\text{CO})_3L^1$  (1a-1c) showed good activity against *E. coli* and *S. paratyphi*. Complexes  $\text{M}(\text{CO})_3L^2$  (2a-2c) and  $\text{M}(\text{CO})_3L^3$  (3a-3c) exhibited good activity against *S. paratyphi*.

#### CONCLUSION

The IR spectroscopic data are in well accord with a cis-chelating tridentate coordination of the ligand [29].  $L^1$ ,  $L^2$  &  $L^3$  ligands behave as a tridentate ligand via imine and amine N and phenolic O donor atom in 1a-3c. In view of above, we have now investigated the nine new complexes 1a-3c, which have been prepared for the first time, by the photochemical reaction of metal carbonyls  $\text{M}(\text{CO})_6$  (M=Cr, Mo & W), with 4-bromo-2-(2-alkylaminoethylimino)methyl]phenol ( $L^1, L^2, L^3$ ).

The results show that 4-bromo-2-(2-alkylaminoethylimino)methyl]phenol and its metalcarbonyl derivatives exert a moderate inhibitory effect on different bacterial activity, thus confirming our previous data about the cytotoxic activity of these compounds.

#### ACKNOWLEDGEMENTS

Sincere thanks are due to Head, Dept. of Chemistry, A. I. M. T., Greater Noida, Head, Dept. of Chemical Sciences, JJTU, Rajasthan and Gaurav Pharma Limited, Pitampura, New Delhi for providing research facilities. Authors are also thankful to Dr. Vivek Kumar, Professor, IIT Roorkee, (Saharanpur Campus) for his constant encouragement to carry out such research work. Special thanks are also due to CDRI, Lucknow, Intertek, Mumbai, I.I.T., Roorkee, Research Lab, Delhi University and TIFR, Mumbai for allocation of time for various analyses.

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