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# Synthesis and Reactivity in Inorganic, Metal-Organic, and Nano-Metal Chemistry

Publication details, including instructions for authors and subscription information: <u>http://www.tandfonline.com/loi/lsrt20</u>

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To cite this article: K. Deepa, N. T. Madhu & P. K. Radhakrishnan (2005): Cadmium(II) Complexes of 1,2-Di(Imino- 4'-Antipyrinyl)Ethane, Synthesis and Reactivity in Inorganic, Metal-Organic, and Nano-Metal Chemistry, 35:10, 883-888

To link to this article: <u>http://dx.doi.org/10.1080/15533170500358135</u>

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#### Synthesis and Reactivity in Inorganic, Metal-Organic and Nano-Metal Chemistry, 35:883–888, 2005 Copyright © 2005 Taylor & Francis, Inc. ISSN: 1553-3174 print/1532-2440 online DOI: 10.1080/15533170500358135

# Cadmium(II) Complexes of 1,2-Di(Imino-4'-Antipyrinyl)Ethane

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Cadmium(II) complexes of the Schiff base 1,2-di(imino-4'antipyrinyl)ethane (GA) having general formulae  $[Cd(GA)]X_2$ ;  $(X = NO_3^-, ClO_4^-)$  and  $[Cd(GA)X_2]$ ;  $(X = Cl^-, Br^- \text{ or } I^-)$  have been synthesized and characterized by elemental analysis, electrical conductance in non-aqueous solvents, infrared and electronic spectra as well as thermogravimetric analysis. In all these complexes, GA acts as a neutral tetradentate ligand coordinating through both the carbonyl oxygens and both the azomethine nitrogens. Both the anions are coordinated in the halide complexes while these remain as counter ions in the perchlorate and nitrate complexes. Thermal decomposition behavior of the nitrate complex indicates that it is stable up to 234 °C and undergoes a three-stage decomposition pattern yielding the anhydrous Cadmium(II) oxide as the final residue.

Keywords Cadmium(II) complexes, 1,2-di(imino-4'-antipyrinyl) ethane

#### **INTRODUCTION**

A number of Schiff base complexes, especially derived from pyrazolone ligands such as antipyrine and 4-aminoantipyrine have been widely investigated in view of their novel structural features, unusual magnetic properties and relevance to biological processes and also they exhibit antibacterial and antiinflammatory properties (Alaudeen et al., 1995; Singh et al., 1986). Relatively little is known about the Cadmium complexes of 4-aminoantipyrine Schiff base (Agarwal et al., 1992; Dholakiya and Patel, 2002). In view of this and as a part of our continuing interest on transition metal complexes of pyrazolone derivatives (Madhu and Radhakrishnan, 2000a, 2001a; Raju et al., 2002), we report here the synthesis and characterization of cadmium(II) complexes of Schiff base ligand derived from pyrazolone (GA) (Figure 1).

#### **RESULTS AND DISCUSSION**

The formation of the complexes may be formulated by the general equation shown next.

$$CdX_2 + GA \rightarrow Cd(GA)X_2;$$
  
(X = ClO<sub>4</sub><sup>-</sup>, NO<sub>3</sub><sup>-</sup>, Cl<sup>-</sup>, Br<sup>-</sup>or I<sup>-</sup>)

The perchlorate, bromide and iodide complexes are soluble in DMF, ethanol, methanol, nitrobenzene and benzene, but insoluble in chloroform and ethyl acetate. The chloride and nitrate complexes are soluble in acetonitrile, benzene, DMF, ethanol and methanol but insoluble in nitrobenzene.

The elemental analyses data of the complexes (Table 1) suggest that the complexes can be formulated as  $Cd(GA)X_2$ ; (X =  $ClO_4^-$ ,  $NO_3^-$ ,  $Cl^-$ ,  $Br^-$  or  $I^-$ ).

## **Electrical Conductance**

The molar conductance values of the complexes (Table 2) in non-aqueous solvents such as DMF, methanol and nitrobenzene or acetonitrile ( $10^{-3}$  M Solutions) indicate a 1 : 2 electrolytic nature for perchlorate and nitrate complexes (Nair and Radhakrishnan, 1995; Geary, 1971) and non-electrolytic behavior for the chloride, bromide and iodide complexes (Raju et al., 2002). The slightly higher values observed for chlorocomplex in methanol and acetonitrile and nitrate complex in acetonitrile may be due to the partial displacement of coordinated anions by solvent molecules. Hence the complexes may be formulated as  $[Cd(GA)]X_2$  (where  $X = CIO_4^-$  or  $NO_3^-$ ) and  $[Cd(GA)X_2]$ (where  $X = CI^-$ , Br<sup>-</sup>, or I<sup>-</sup>).

#### Infrared Spectra

The pertinent spectral bands of GA and its complexes together with tentative assignments are presented in Table 3. GA shows a very strong band at  $1650 \text{ cm}^{-1}$ , which may be attributed to C==O stretching vibration (Madhu and Radhakrishnan, 2001b). In complexes this band is shifted to the region  $1613-1639 \text{ cm}^{-1}$ 



Received 9 May 2005; accepted 9 September 2005.

The authors are indebted to Mahatma Gandhi University, Kerala, India for providing the laboratory facilities. We also thank to the Heads of Regional Sophisticated Instrumentation Center, IIT, Chennai and Regional Research Laboratory, Trivandrum, Kerala for providing some instrumental facilities. Also, we wish to express our heartfelt thanks to Mr. K. C. Raju, for some helpful discussions.

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FIG. 1. 1,2-Di(imino-4'-antipyrinyl)ethane.

showing the coordination of both carbonyl oxygens (Radhakrishnan et al., 1984). An intense band of GA at 1576 cm<sup>-1</sup> attributable to the C=N stretch in GA (Madhu and Radhakrishnan, 2001b), is shifted to the region  $1593-1605 \text{ cm}^{-1}$  in complexes indicating the coordination of both azomethine nitrogens (Marykutty et al., 2001). The perchlorate complex shows an intense, broad and unsplit band at  $1094 \,\mathrm{cm}^{-1}$  assignable to the  $v_3$  vibration of uncoordinated perchlorate ion having T<sub>d</sub> symmetry. This is supported by another sharp band of medium intensity at  $622 \text{ cm}^{-1}$  due to the  $v_4$  vibration of perchlorate ion having T<sub>d</sub> symmetry (Scholar et al., 1975). For the nitrate complex, a very strong band observed at  $1370 \,\mathrm{cm}^{-1}$  is attributable to the  $v_3$  vibration of uncoordinated nitrate ion having D<sub>3h</sub> symmetry (Gatehouse et al., 1958). This is supported by another band of medium intensity at  $820 \,\mathrm{cm}^{-1}$ , which is assignable to the  $\nu_2$  vibration of nitrate (D<sub>3h</sub>) ion (Ajithkumar and Radhakrishnan, 2002). In Far IR spectra of the halide complexes, the Cd-Cl, Cd-Br and Cd-I stretching vibrations occur at 235, 209 and 167 cm<sup>-1</sup> respectively, which are absent in the spectrum of the free ligand.

The above results along with conductance data indicate that perchlorate and nitrate anions remain as counter ions, while the halide ions are coordinated to the metal ion in these complexes.

#### **Electronic Spectra**

The electronic spectral data of GA and the complexes in solid state (using Nujol) with tentative assignments are presented in Table 4. The electronic spectra of GA shows two band maxima at 26,180 and 39,220 cm<sup>-1</sup> corresponding to  $n \rightarrow \pi^* \cdot and \pi \rightarrow \pi^*$  transitions respectively (Madhu and Radhakrishnan, 2001b). In complexes the  $n \rightarrow \pi^*$  bands are found to be red shifted to the region 21,881–24,271 cm<sup>-1</sup> while the  $\pi \rightarrow \pi^* \cdot band$  is found to be blue shifted to the region 39,370–40,322 cm<sup>-1</sup> compared to that of GA. The electronic spectra of all the complexes exhibit an intense absorption band in the region 32,362–33,444 cm<sup>-1</sup> that might be due to a charge transfer process.

#### **Thermal Behavior and Kinetic Aspects**

The TG data for cadmium(II) nitrate complex is presented in Table 5. The thermal decomposition studies were carried out in the temperature range  $30-800^{\circ}$ C. The three-stage decomposition pattern leads to the following conclusions. The

complex is stable up to  $234^{\circ}$ C revealing the absence of any water or solvent molecules in the complex. In the first stage ( $234-288^{\circ}$ C) a mass loss of 42.2% is attributed to the pyrolytic removal of one of the nitrate ions and half of the ligand. The IR spectrum of the residue after first stage also confirms the presence of nitrate ions and ligand portions. In the second stage ( $288-468^{\circ}$ C), a mass loss of 32% was observed that is due to the removal of rest of the ligand portion. The IR spectrum of the residue after this stage confirms the absence of any ligand portions. In the third stage ( $468-518^{\circ}$ C), the mass loss of 8.8% is assigned to the removal of the remaining nitrate ion. The final residue is quantitatively proved to be anhydrous cadmium oxide.

The non-isothermal kinetic aspects of these pyroltic decompositions were carried out by the application of Coats–Redfern method (Coats and Redfern, 1964). The kinetic parameters, namely, the activation energy (E), the pre-exponential factor (A) and the entropy of activation ( $\Delta$ S) were calculated (given in Table 5). There is no particular trend for E, A or  $\Delta$ S among the different stages of degradations. Based on the results of present physicochemical investigations, the structures shown in Figure 2 and Figure 3 may be assigned to the reported complexes.

#### **EXPERIMENTAL**

#### Materials

The cadmium salts were prepared from Analar cadmium carbonate by dissolving it in respective 50% acids (Merck) and then crystallizing out salts by evaporating the solution on a steam bath. The 4-aminoantipyrine and glyoxal were purchased from Aldrich Chemical Co., USA. The Schiff base 1,2-di(imino-4'-antipyrinyl)ethane (GA) was prepared and characterized as reported earlier (Madhu and Radhakrishnan, 2000b). Solvents used were either GPR or AR grade (Merck, India; BDH, India or SRL, India).

#### Preparation of Complexes

#### Perchlorate Complex

First, 1 mmol of  $Cd(ClO_4)_2^- \cdot 6H_2O$  in ethyl acetate (10 mL) was added drop wise to the boiling suspension of the Schiff base (1.2 mmol) in ethyl acetate (100 mL). This mixture was refluxed for about 2 h on a steam bath. The precipitated complex was filtered and washed with hot ethyl acetate to remove any excess ligand. The complexes were then recrystallized from methanol, dried over phosphorous(V) oxide and kept under vacuum. (*Warning! Perchlorate salts with organic compounds are potentially explosive. They should be handled in small quantities and with caution.*)

#### Nitrate, Chloride, Bromide and Iodide Complexes

Here, 1 mmol of  $Cd(NO_3)_2^- \cdot 4H_2O$ ,  $CdCl_2^- \cdot 2H_2O$ ,  $CdBr_2$  or  $CdI_2$  in methanol (10 mL) was added drop-wise to the boiling suspension of the Schiff base (1.2 mmol) in ethyl acetate

Empirical Formula M.P. Hydrogen Yield formulae weight Metal (%) (%) Nitrogen (%) (%) (°C) Complex Anion (%) Carbon (%) 588  $[Cd(GA)](ClO_4)_2$  $CdC_{24}H_{24}Cl_2N_6O_{10}$ 739.80 26.82 (26.88) 38.91 (38.96) 3.22 (3.26) 85 238 15.18 (15.19) 11.31 (11.36)  $[Cd(GA)](NO_3)_2$ CdC24H24N8O8 664.91 43.31 (43.35) 3.69 (3.63) 16.90 (16.85) 230 16.94 (16.90) 90 \_\_\_\_  $CdC_{24}H_{24}Cl_2N_6O_2$ 611.81 11.60 (11.58) 3.90 (3.95) 87  $[Cd(GA)Cl_2]$ 18.34 (18.37) 47.07 (47.11) 13.69 (13.73) 227  $[Cd(GA)Br_2]$  $CdC_{24}H_{24}Br_2N_6O_2$ 700.71 16.05 (16.04) 22.79 (22.80) 41.11 (41.13) 3.39 (3.45) 11.95 (11.99) 91 233  $[Cd(GA)I_2]$  $CdC_{24}H_{24}I_2N_6O_2$ 794.71 241 14.09 (14.14) 31.90 (31.93) 36.23 (36.27) 3.02 (3.04) 10.52 (10.57) 88

 TABLE 1

 Analytical data<sup>a</sup> of the cadmium(II) complexes of GA

<sup>a</sup>Calculated values in parenthesis.

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TABLE 2	
Molar conductance <sup><math>a</math></sup> data of the cadmium(II) complexes <sup><math>b</math></sup> v	vith GA

		Molar conductance				
Complex	DMF	Methanol	Nitrobenzene	Acetonitrile		
$[Cd(GA)](ClO_4)_2$	140.87	166.49	53.78			
$[Cd(GA)](NO_3)_2$	140.87	160.08	_	290.04		
$[Cd(GA)Cl_2]$	24.33	71.71	_	104.67		
$[Cd(GA)Br_2]$	25.61	2.82	0.51	_		
[Cd(GA)I <sub>2</sub> ]	28.17	26.89	0.89	_		

<sup>*a*</sup>Ohm<sup>-1</sup> cm<sup>2</sup> mol<sup>-1</sup>. <sup>*b*</sup>10<sup>-3</sup> M solutions.

TABLE 3

Important infrared spectral bands (cm<sup>-1</sup>) of GA and its cadmium(II) complexes<sup>a</sup>

GA	$[Cd(GA)](ClO_4)_2$	$[Cd(GA)](NO_3)_2$	[Cd(GA)Cl <sub>2</sub> ]	[Cd(GA)Br <sub>2</sub> ]	$[Cd(GA)I_2]$	Assignments
1650 s	1633 s	1613 s	1613 s	1613 s	1639 s	ν(C==O)
1576 s	1605 s	1598 s	1598 s	1598 s	1593 s	$\nu(C=N)$
	1094 s	_	_	_	_	$\nu_3$ -ionic ClO <sub>4</sub>
_	622 m	_	_	_	_	$v_4$ -ionic ClO <sub>4</sub>
_	_	1370 s	_	_	_	$v_3$ -ionic NO <sub>3</sub>
_	_	820 m	_	_	_	$v_2$ ionic NO <sub>3</sub>
_	_	—	235 m	_	_	$\nu$ (Cd–Cl)
_	_	—	_	209 m	_	$\nu$ (Cd-Br)
_	_	—	_	_	167 m	$\nu$ (Cd–I)
_	418 w	415 w	420 w	420 w	410 w	$\nu$ (Cd–O)
—	345 w	345 w	348 w	348 w	340 w	$\nu$ (Cd-N)

<sup>*a*</sup>s—strong; m—medium; w—weak.

Sample	Abs. max $(cm^{-1})$	Tentative assignments
GA	26,180	$\mathrm{n}  ightarrow \pi^{*}$
	39,220	$\pi  ightarrow \pi^{*}$
$[Cd(GA)](ClO_4)_2$	23,529	$ m n  ightarrow \pi^{*}$
	40,000	$\pi  ightarrow \pi^{*}$
	33,444	Charge transfer
$[Cd(GA)](NO_3)_2$	21,881	$ m n  ightarrow \pi^{*}$
	40,000	$\pi ightarrow \pi^{*}$
	33,333	Charge transfer
[Cd(GA)Cl <sub>2</sub> ]	21,978	$ m n  ightarrow \pi^*$
	40,322	$\pi ightarrow\pi^{*}$
	33,444	Charge transfer
$[Cd(GA)Br_2]$	23,529	$ m n  ightarrow \pi^{*}$
	39,370	$\pi ightarrow\pi^{*}$
	32,362	Charge transfer
$[Cd(GA)I_2]$	24,271	$ m n  ightarrow \pi^*$
	40,000	$\pi ightarrow \pi^{*}$
	33,444	Charge transfer

TABLE 4 Electronic spectral data of cadmium(II) complexes of GA

Phenome	nological data and kinetion	e paramet	ers for the	rmal deco	mposition of cadmiur	n(II) nitrate comp	olex with GA
	Stage of	Thermal data <sup>a</sup>					
Complex	decomposition	T <sub>i</sub>	T <sub>p</sub>	$T_{\rm f}$	$E (kJ/mol^{-1})$	$A(S^{-1})$	$\Delta S (J/mol^{-1})$

288

468

518

104.40

82.27

580.90

TABLE 5

 ${}^{a}T_{i}$  = initial temperature;  $T_{p}$  = peak temperature;  $T_{f}$  = final temperature.

234

288

468

280

461

507



Ι

Π

III

FIG. 2. Tentative structure of  $[Cd(GA)]X_2$ ;  $(X = ClO_4, NO_3, Ph = -C_6H_5)$ .



FIG. 3. Tentative structure of  $[Cd(GA)X_2]$ ;  $(X = Cl^-, Br^- \text{ or } I^-; Ph = -C_6H_5)$ .

(100 mL). This mixture was then refluxed on a steam bath for about 2 h. The precipitated complex was filtered and washed repeatedly with hot ethyl acetate to remove any excess ligand. The complexes were then recrystallized from methanol and dried over phosphorous(V) oxide and kept under vacuum.

### ANALYTICAL METHODS

 $[Cd(GA)](NO_3)_2$ 

All the complexes were analyzed for metal and halide contents by standard methods (Bassett et al., 1961) and the perchlorate content by the Kurz method (Kurz et al., 1958). The electrical conductance of the complexes in DMF, methanol and nitrobenzene or acetonitrile  $(10^{-3} \text{ M solutions})$  were measured at room temperature using Toshniwal conductivity bridge with a dip-type conductance cell (cell constant  $1.2807 \,\mathrm{cm}^{-1}$ ). The infrared spectra of the ligand and the

complexes were recorded in the range  $4000-400 \text{ cm}^{-1}$  on a Shimadzu 1R 470 spectrophotometer in KBr disc and in the range 400-200 cm<sup>-1</sup> on a Perkin Elmer 983 IR Spectrophotometer in polyethylene powders. The electronic spectra of the ligand and the complexes in solid state (paste with Nujol) were measured in the range 200-1100 nm on a Shimadzu UV 160A Spectrophotometer. The elemental analyses (C, H, and N) were carried out in a Heraeus CHNO rapid analyzer. Thermogravimetric analysis was conducted on a Delta series TGA7 thermal analyzer in nitrogen atmosphere (sample mass: 2 mg, heating rate: 10°C/min) from room temperature to about 800°C. The kinetic parameters were calculated using a computer program.

113.60

 $6.8 \times 10^{-2}$ 

 $4.3\times10^{13}$ 

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-210.60

-274.60

-8.27

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