



Research Article

ISSN : 0975-7384
CODEN(USA) : JCPRC5

Metal complexes of ammonium phenyl dithiocarbamate: Preparation, characterization, and biological activity

Archana Dinkar Ingle, Harshal Devghare and Kiran Parase

Department of Chemistry, R.D.I.K. College, Badnera (M.S), India

ABSTRACT

Metal complexes of Ammonium Phenyl di-thiocarbamate from Ammonium phenyl dithiocarbamate (L^1) and Copper Chloride (M^1), Nickel Chloride (M^2), Cobalt Nitrate (M^3), Cadmium Chloride (M^4), Mercurus Chloride (M^5) are reported and characterized based on IR, NMR. In the present investigation compounds have been tested for their antibacterial activity against Escherichia coli, Staphalococcus aureus, Proteus vulgaris and Psudomonas aeruginosa.

Key words : Ammonium phenyl di-thiocarbamate, copper chloride, *Escherichia coli*, *Psudomonas aeruginosa*, *Staphalococcus aureus*, IR, NMR

INTRODUCTION

Organic dithiocarbamates have attracted a great deal of importance due to their interesting chemistry and wide utility.[1-7] Organic dithiocarbamates are valuable synthetic intermediates,[8] which are ubiquitously found in a variety of biologically active compounds. Functionalization of the carbamate moiety offers an attractive method for the generation of derivatives, which may constitute interesting medicinal and biological properties.[9] Dithiocarbamates are also widely used in medicinal chemistry and have found application in the treatment of cancer[10] and have been tested in clinical trials for various indications including HIV[11-14]. Organic dithiocarbamates are valuable synthetic intermediates,[15] which are ubiquitously found in a variety of biologically active compounds. Functionalization of the carbamate moiety offers an attractive method for the generation of derivatives, which may constitute interesting medicinal and biological properties[16].

EXPERIMENT SECTION

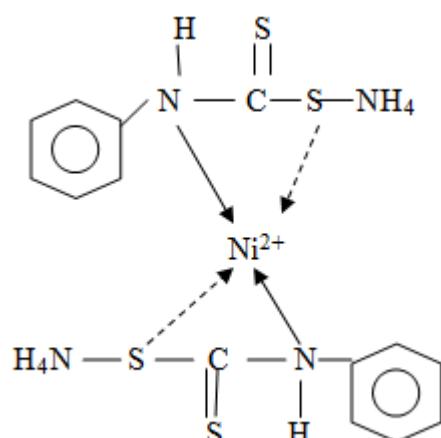
A] Preparation of ammonium phenyldithiocarbamate:

Preparation of complexes :

1] nickel complex :

1M ammonium phenyldithiocarbamate and 1M nickel chloride solution are mixed with each other, complex is formed.

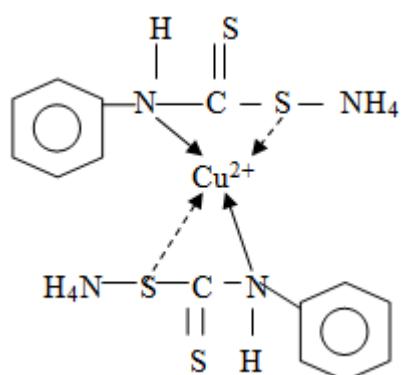
Structure :



2] copper complex :

1M ammonium phenyl dithiocarbamate and 1M copper chloride solution are mixed with each other, complex is formed.

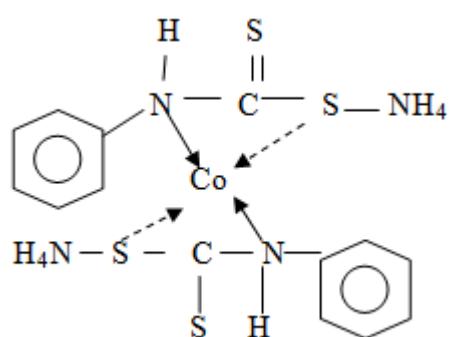
Structure :



3] Cobalt Complex :

1M ammonium phenyl dithiocarbamate and 1M cobalt nitrate solution mixed with each other, complex is formed.

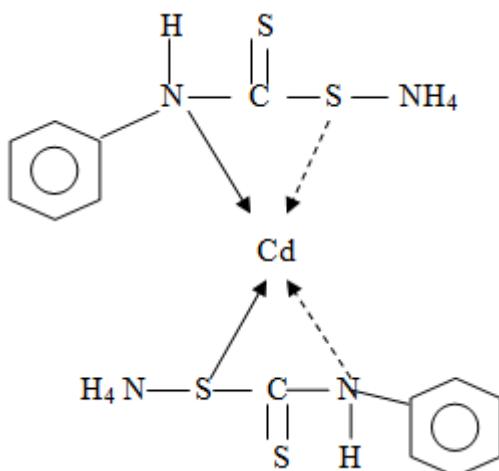
Structure :



4] Cadmium Complex :

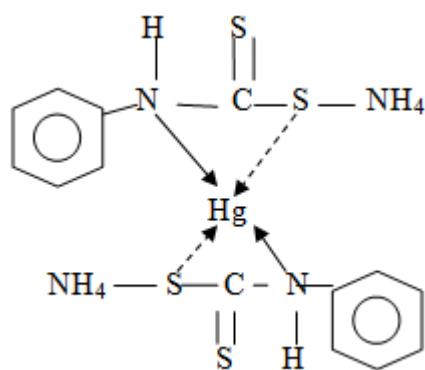
1M ammonium phenyl dithiocarbamate and 1M Cadmium chloride solution are mixed with each other, complex is formed.

Structure :



5] Mercuruscomplex :1M ammonium phenyl dithiocarbamateand 1M Mercurus chloride solution are mixed with each other, complex is formed.

Structure :



RESULTS AND DISCUSSION

The reaction of ammonium phenyl thiocarbamate solution and metal like Copper Chloride(M¹), Nickel Chloride(M²), Cobalt Nitrate(M³), Cadmium Chloride(M⁴), Mercurus Chloride(M⁵) gives complexes The IR, 1H NMR elemental analysis (Table-1)

Spectral data:

Nickel Complex:

¹H-NMR spectrum analysis of Complex of Ni²⁺ showed the presence of following peaks. The chemical shift can be correlated as shown below in table (1)

IR VALUE:

n3398 cm⁻¹ (N-H), 1633cm⁻¹ (C=S) , 1450 cm⁻¹ (C-N), 757 cm⁻¹ (C-S), 697 cm⁻¹ (Ni-N) and NMR data as bellow :

Table No.1

Sr. No.	Signal Position (δ -ppm)	Relative No. of H- atom	Multiplicity	Assignment
1	7.2	10	s	Ar-H
2	3.8	2	d	-NH
3	9.8	8	s	-NH ₄

Spectral data:**Copper complex:**

¹H-NMR spectrum analysis of Complex of Cu²⁺ showed the presence of following peaks. The chemical shift can be correlated as shown below in table No. 2

IR DATA:

3204 cm⁻¹ (N-H), 1593cm⁻¹ (C=S) , 1450 cm⁻¹ (C-N), 756 cm⁻¹ (C-S), 691 cm ⁻¹ (Cu-N) and NMR data as bellow in table 2.

Table No. 2

Sr. No.	Signal Position (δ -ppm)	Relative No. of H- atom	Multiplicity	Assignment
1	7.4	10	S	Ar-H
2	4.2	2	d	N-H
3	9.87	8	S	NH ₄

Spectral data:**Mercury complex:**

¹H-NMR spectrum analysis of Complex of Hg²⁺ showed the presence of following peaks.

IR VALUE:

3201 cm⁻¹ (N-H), 1546cm⁻¹ (C=S) , 1403 cm⁻¹ (C-N), 756 cm⁻¹ (C-S), 696 cm ⁻¹ (Hg-N) and NMR data as bellow in The chemical shift can be correlated as shown below intable No.3

Table No. 3

Sr. No.	Signal Position (δ -ppm)	Relative No. of H- atom	Multiplicity	Assignment
1	7.4	1	S	Ar-H
2	4.4	1	d	-NH
3	10.3	4	S	-NH ₄

Cadmium complex:

¹H-NMR spectrum analysis of Complex of Cd²⁺ showed the presence of following peaks. The chemical shift can be correlated as shown below. Table No .4

IR VALUE:

3245 cm⁻¹ (N-H), 1598cm⁻¹ (C=S) , 1498 cm⁻¹ (C-N), 758cm⁻¹ (C-S), 689 cm ⁻¹ (Cd-N) and NMR data as bellow in table No.4

Table No. 4

Sr. No.	Signal Position (δ -ppm)	Relative No. of H- atom	Multiplicity	Assignment
1	7.6	1	S	Ar-H
3	3.9	1	d	N-H
4	9.87	4	S	NH ₄

Spectral data :**Cobalt complex:**

¹H-NMR spectrum analysis of Complex of Co²⁺ showed the presence of following peaks. The chemical shift can be correlated as shown below.

IR VALUE:

3190 cm⁻¹ (N-H), 1589cm⁻¹ (C=S) , 1495 cm⁻¹ (C-N), 756cm⁻¹ (C-S), 694 cm ⁻¹ (Co-N) and NMR data as bellow in table No.5

Table:5

Sr. No.	Signal Position (δ -ppm)	Relative No. of H- atom	Multiplicity	Assignment
1	7.7	1	S	Ar-H
3	4.1	1	d	N-H
4	10.5	4	S	NH ₄

On the basis of elemental analysis and spectral data the molecular formula of Ni²⁺, Cu²⁺, Hg²⁺, Cd²⁺was established as , C₁₄ H₂₀ N₄ S₄ M. The M.P.recorded in the table 6 :

Table:6

A)Metal Complex (Ammonium phenyl dithiocarbamate)	Melting Point
Copper complex	112° C
Nickel chloride	108° C
Cadmium complex	110° C
Mercurus complex	105° C
Cobalt complex	115° C

Microbial activity:

All the compounds have been screened for both antimicrobial using cupplate agar diffusion method¹⁷⁻¹⁸ by measuring the inhibition zone in mm. The compounds were taken at a concentration of 1 mg/ml using DMF. as solvent. The compounds were screen for antibacterial activity against *Escherichia coli*, *Staphylococcus aureus*, *Proteus vulgaris* and *Pseudomonas aeruginosa* in nutrient agar medium. The results are presented in Table-7

From the observation ,Nickel complex and copper complex show significant activity against *P. aeruginosa* and *E.Coli*. Cobalt complex show significant activity against *S.aureus*

Table:7Antimicrobial activities of Metal Complexes of Ammonium phenyl dithiocarbamate.

Compound	Antibacterial			
	<i>E.Coli</i>	<i>S.aureus</i>	<i>P.vulgaris</i>	<i>P.aeruginosa</i>
Nickel complex	19	15	10	25
Copper complex	16	16	12	21
Cobalt complex	16	20	18	18
Mercury complex	12	14	20	17
Cadmium complex	10	15	17	20

CONCLUSION

The new series complex of ammonium phenyl di thiocarbamates were synthesized by utilizing a simple and efficient method in good yields. The structures assigned have been supported by adequate spectral data. The results of antimicrobial activity revealed that of the compounds exhibited prominent activity against the bacteria *E.coli* and *S.aureus*, *P.vulgaris*, *P.aeruginosa*.

Acknowledgements

I am indebted to Dr. Dikshit, Deputy Directors and Head, SAIF, lucknow for recording IR and NMR spectra of my samples and also for C, H and N analyses.

REFERENCES

- [1] (a) Han, C.; Porco Jr, J. A. *Org. Lett.* **2007**, 9(2), 1517; (b) Ranise, A.; Spallarossa, A.; Schenone, S.; Burno, O.; Bondavalli, F.; Vargiu, L.; Marceddu, T.; Mura, M.; Colla, P. L.; Pani, A. *J. Med. Chem.* **2003**, 46(3), 768; (c) Cao, S. L.; Feng, Y. P.; Jiang, Y. Y.; Liu, S. Y.; Ding, G. Y.; Li, R. T. *Bioorg. Med. Chem. Lett.* **2005**, 15(2), 1915; (d) Salvatore, R. N.; Sahab, S.; Jung, K. W. *Tetrahedron Lett.* **2001**, 42(4), 2055; (e) Adams, P.; Baron, F. A. *Chem. Rev.* **1965**, 65(3), 567.
- [2](a) Rafin, C.; Veignie, E; Sancholle, M.; Postal, D.; Len, C.; Villa, P.; Ronco, G. *J. Agric. Food. Chem.* **2000**, 48(1), 5283; (b) Jager, P.; Rentzea, C. N.; Kieczka, H. in *Ullmann's Encyclopedia of Industrial Chemistry*, 5th edn. (VCH, Weinheim) **1986**, 51.
- [3] (a) Tsuboi, S.; Takeda, S.; Yamasaki, Y.; Sakai, T.; Utsuka, M.; Ishida, S.; Yamada, E.; Hirano, J. *Chem. Lett.* **1992**, 8(2), 1417; (b) Katritzky, A. R.; Singh, S.; Mahapatra, P. P.; Clemense, N.; Kirichenko, K. *Arkivoc* **2005**, 9(2), 63.
- [4.] Greene, T. W.; Wuts, P. G. M. *Protecting Group in Organic Synthesis*, 3rd edn. (Wiley Interscience, New York) **1999**, 484.
- [5](a) Garin, J.; Melandz, E.; Merchain, F. L.; Tejero, T.; Urid, S.; Ayestaron, J. *Synthesis* **1991**, 147; (b) Chaturvedi, D.; Ray, S.; *Tetrahedron Lett.* **2006**, 47(4), 1307.
- [6] (a) Crich, D.; Quintero, L. *Chem. Rev.* **1989**, 8(2), 1413; (b) Barton, D. H. R.; *Tetrahedron* **1992**, 48(2), 2529; (c) Zard, S. Z. *Angew Chem. Int. Ed. (Engl.)* **1997**, 36(3), 672. 75
- [7] .Zhang, D.; Chen, J.; Liang, Y.; Zhou, H. *Synth. Commun.* **2005**, 35, 521.
- [8]. (a) Boas, U.; Jakobsen, M. H. *J. Chem. Soc., Chem. Commun.* **1995**; (b) Elgemeie, G. H.; Sayed, S. H. *Synthesis* **2001**, 1747; (c) Mukerjee, A. K.; Ashare, R. *Chem. Rev.* **1991**, 91, 1; (d) Boas, U.; Gertz, H.; Christensen, J. B.; Heegaard, P. M. H. *Tetrahedron Lett.* **2004**, 45, 269. 77

- [9] (a) Dhooghe, M.; De Kime, N. *Tetrahedron* **2006**, 62, 513; (b) Fernandez, J. M. G.; Mellet, C. O.; Blanco, J. L.; Mota, J. F.; Gadelle, A.; Coste-Sarguet, A.; Defaye, J. *Carbohydr. Res.* **1995**, 268(4), 57.
- [10] (a) Ronconi, L.; Marzano, C.; Zanello, P.; Corsini, M.; Miolo, G.; Macca, C.; Trevisan, A.; Fregoni, D. *J. Med. Chem.* **2006**, 49, 1648; (b) Walter, W.; Bode, K. D. *Angew. Chem. Int. Ed. Engl.* **1967**, 6, 281; (c) Elgemeie, G. H.; Sayed, S. *H. Synthesis* **2001**, 1747.
- [11] *AIDS Res. Hum. Retrov.* **1993**, 9, 83.
- [12] Hersh, E. M.; Brewton, G.; Abrams, D.; Bartlett, J.; Galpin, J.; Gill, P.; Gorter, R.; Gottlieb, M.; Jonikas, J. J.; Landesman, S. *JAMA* **1991**, 265(2), 1538.
- [13] Kaplan, C. S.; Petersen, E. A.; Yocom, D.; Hersh, E. M. *Life Sci.* **1989**, 45, iii.
- [14] Lang, J. M.; Touraine, J. L.; Trepo, C.; Choutet, P.; Kirstetter, M.; Falkenrodt, A.; Herviou, L.; Livrozet, J. M.; Retornaz, G.; Touraine, F. *Lancet* **1988**, 2(3), 702.
- [15] (a) Boas, U.; Jakobsen, M. H. *J. Chem. Soc., Chem. Commun.* **1995**; (b) Elgemeie, G. H.; Sayed, S. *H. Synthesis* **2001**, 1747; (c) Mukerjee, A. K.; Ashare, R. *Chem. Rev.* **1991**, 91, 1; (d) Boas, U.; Gertz, H.; Christensen, J. B.; Heegaard, P. *M. H. Tetrahedron Lett.* **2004**, 45, 269. 77
- [16] (a) Dhooghe, M.; De Kime, N. *Tetrahedron* **2006**, 62, 513; (b) Fernandez, J. M. G.; Mellet, C. O.; Blanco, J. L. J.; Mota, J. F.; Gadelle, A.; Coste-Sarguet, A.; Defaye, J. *Carbohydr. Res.* **1995**, 268(2), 57.
- [17] Kawangh, F., *Analytical Microbiology*, Academic press, New York (**1963**).
- [18] British Pharmacopaeia- II, Biological assay and Tests, The Stationary Office Ltd., London, **A-205** (1998).
- [19] AbhishekMathur, Satish K. Verma, RakshandaBhat,Santosh K. Singh, ArchanaPrakash, GBKS Prasad and VK Dua, *J. Chem. Pharm. Res.*, **2010**, 2(4):364-370
- [20] A. J. Odola and J. A. O. Woods, *J. Chem. Pharm. Res.*, **2011**, 3(6):865-871
- [21] Priya M. Madalageri and OblennavarKotresh, *J. Chem. Pharm. Res.*, **2012**, 4(5):2697-2703
- [22] M. R. Hemaa, M. Ramaiah, V. P. Vaidyab, B. S. Shivakumara and G. S. Suresha, *J. Chem. Pharm. Res.*, **2013**, 5(4):47-51
- [23] G K Vanita, M Ramaiah, K Shashikala Devi , K Veena, V P Vaidya, *J. Chem. Pharm. Res.*, **2010**, 2(6), 258-264.
- [24] K ShashikalaDevi , M Ramaiah ,G K Vanita , K Veena , V P Vaidya, *J. Chem. Pharm. Res.*, **2011**, 3(1), 445-451.
- [25] G K Vanita, M Ramaiah, K Shashikala Devi , K Veena, V P Vaidya, *J. Chem. Pharm. Res.*, **2010**, 2(6), 258-264.
- [26] KShashikala Devi , M Ramaiah ,G K Vanita , K Veena , V P Vaidya, *J. Chem. Pharm. Res.*, **2011**, 3(1), 445-451.