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X-Ray Power Diffraction Studies On Pr (III), Nd(III)p-Chlorobenzaldehydethiosemicarbazone System

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Abstract

Recent years work on the coordinate compounds of d-block and f-block metals ions with Schiff's base as ligands shows its wide applications in polymer industries [1], pharmaceutical industries [2] and agricultural industries [3]. Some of the complex compounds also show antibacterial [2], anti-viral [2] and anticancer [4] properties. The present study is focused on the analysis of powder X-rays diffraction data of Pr (III), Nd (III)-p-Chlorobenzaldehydethiosemicarbazone complex compounds. This compound also shows anticancer properties [4] and hence it requires detailed studies by researchers and scientists. Other physical properties like thermal stability, stability of compound in solution etc. of these compounds have already been discussed by several authors [5]-[7]. Pr (III), Nd(III)-p-Chlorobenzaldehydethiosemicarbazone is synthesized by direct combination with p -chloro benzaldehyde and thio semi carbazide solution [7]-[10] where it forms a complex with metal : ligand ratio of 1:3. Presently the data so obtained by powder X-ray diffraction gives intensity curves at different 20 & at different miller indices. When analyzed using gen formula of crystallography, the value of edge length calculated from all intensity peaks was almost equal suggesting cubic structure, and that of the other when compared with standard values shows that both the samples are cubic (P) type crystals.

Keywords: crystallography, cubic (P), indices, intensity, X-rays.

1. INTRODUCTION

The ligand Schiff's base exists in thi-ol and thio-enol tautomer & its enolic form shows mono-protonic and bi-dentate behavior, due to which it forms very stable chelates. X-ray is one of the analytical tools which is adaptable in structural applications. Countless X-ray powder files have been prepared and catalogued as ready references for the identification of unknown compounds, and the structural details of unknown samples. X-ray powder diffraction (XRD) is a rapid analytical technique used for phase identification of crystalline substances and by analyzing the data it provides information on the unit cell dimensions and the type of unit cell.

2. METHOD

Sample of Pr (III), Nd(III)-p-Chlorobenzaldehydethiosemicarbazone is finely powdered, powder diffraction method is useful for the crystal of simple structure, as sample is powdered so sample consists of many small crystals which are oriented in all directions, hence the incidence X-ray are scattered from all the possible set of planes (100, 110, 111....) & the scattered rays from different planes are detected by X-ray sensors which give the value of 2.

The finely powdered sample is kept in a glass tube and a beam of X-rays of appropriate wavelength is allowed to fall on the sample. The reflected rays of high intensities are observed and their corresponding angles (2 θ) are observed. Sin² θ can be calculated & when that's divided by the common factor, we obtain the miller indices of the different plans of crystals by comparing with reference table. Thus, the types of crystals may be determined. By using Bragg's equation edge length and d_{hkl} is determine. The formula used is:

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$$\begin{split} \lambda &= 2d \; Sin & \dots Eqn. \; 1 \\ d_{hkl} &= a/\sqrt{(h2+k2+l2)} & \dots Eqn. \; 2 \end{split}$$

*d = inter-planner distance, a = edge length, h,k,l are miller indices

3. EXPERIMENT

Equimolar aqueous solution of thiosemicarbazide was reacted with equimolar solution of p-chlorobenzaldehyde (acetone base) at room temperature with constant stirring. When the reaction was complete, a dirty white precipitate was obtained which was then filtered, washed and kept in an oven overnight at 350-40oC for drying [7]- [8]. GE X RD - 6 was used for X –ray power diffraction studies using curadiation of wavelength (λ) = 1.54 A°.

4. RESULTS AND DISCUSSION

X-ray power diffraction on tris-(p-Chlorobenzaldehydethiosemicarbazone) Nd(III).1¹/₂ H2O and tris-(p-Chlorobenzaldehydethiosemicarbazone) Pr(III).2H2O displayed 8 and 9 peaks respectively and their corresponding angles 2θ value were:

Compound	20 Values				
tris- (p-	12.00, 16.98, 20.98, 24.17, 27.07, 29.61, 34.47,				
Chlorobenzaldehydethiosemicarbazone) Nd(III) 1½ H2O	36,20				
tris- (p-	10.42, 15.26, 18.34, 21.70, 23.82, 26.02, 29.93, 31.92,				
Chlorobenzaldehydethiosemicarbazone)	37.08				
Pr(III) 2H2O					

TABLE 1: Observed 20 Values.

From above value of \Box , Sin2 \Box was calculated by dividing it with C.F (common factor) & the values so obtain was rounded off. The values were tallied with the values (N= h2+k2+l2) to indicate the hkl (Miller indices) (Table 2 and 3). For tris-(p-Chlorobenzaldehydethiosemicarbazone) Nd(III).11/2 H2O the round values were 1, 2, 3, 4, 5, 6,8, 9 (7 was missing) & these value correspond to Miller indices 100, 110, 111,... After comparing these values with standard values from reference tables, a cubic(P) type tris-(p-Chlorobenzaldehydethiosemicarbazone) crystal of Nd(III) 11/2 H2O was indicated. For tris-(p-Chlorobenzaldehydethiosemicarbazone) Pr(III) 2H2O the rounded off values were the same as Nd(III) complex (1,2,3,4,5,6,8,9,12) & again only 7 was missing. The possible Miller indices are 100,110,111222 respectively. Parameters of crystallographic study are given in table -2. By analyzing data of the table, the conclusion is that both the complexes are Cubic (P) type.

20	Sinθ	Sin²θ	Sin ² 0/C.F	Miller Ind.	d A°	a A°
12.00	0.09413	0.00886	1	100	8.18	8.18
16.98	0.13298	0.01768	1.99 (2)	110	8.79	8.20
20.95	0.1638	0.02683	3.028(3)	111	4.70	8.15
24.17	0.1887	0.03560	4.13 (4)	200	4.08	8.16
27.07	0,2110	0,04452	5,02 (5)	210	3.65	8.18
29.61	0.2305	0.05313	5.996(6)	211	3.34	8.20
34.47	0.2674	0.07150	8.069(8)	220	2.88	8.16
36.20	0.2805	0.07860	8.87 (9)	221/300	2.73	8.20
						Mean=8.18

TABLE 2: X-Ray Power Diffraction Study on Tris- (p-Chlorobenzaldehydethiosemicarbazone) Nd(III).11/2 H2O.

20	Sin0	Sin ² 0	Sin ² $\theta/C.F$	Miller Ind.	d A ^o	a A ^o
10.42	0.09080	0.00024	1	100	8.48	8.48
15.26	0.13276	0.0176	2.14(2)	110	5.80	8.20
18.34	0.15975	0.0255	3.09(3)	111	4.82	8.35
21.70	0.18826	0.03544	4.30(4)	200	4.09	8.18
23.82	0.2064	0.0426	5.17(5)	210	3.73	8.35
26.02	0.2251	0.0507	6.15(6)	211	3.42	8.38
29.93	0.2583	0.0667	8.09(8)	220	2.98	8.43
31.92	0.275	0.0756	9.18(9)	221	2.75	8.26
						Mean=8.34

TABLE 3: X-Ray Power Diffraction Study on Tris-(p-Chlorobenzaldehydethiosemicarbazone) Pr(III).2H2O.

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5. REFERENCES

[1] EI-Bindary. J thermodynamics and catalyst 2014, 5;2(research gate)

[2] Chang EL, Simmers C, Knight DA. Cobalt complexes as antiviral and antibacterial agents. Pharmaceuticals. 2010;3:1711-1728.DOI ;10.3390/PH 3061711

[3] Wang,Z; Gai,S; Cui, Y; Dong. J. Research on intelligent agriculture system based on digital twins and blockchain. J Hebei Acad, Sci.2021,38,66-73. [Google Scholor]

[4] Cepeda V,Fuertes MA. Castilla, J, Alonso C, Quevido C, Perez JM. Anti-Cancer agents Med Chem. 2007: 7; 3-18 [PubMed] [Google Scholar]

[5] G. Chettri . IJARESM ISSN: 2455-6211 2022 Vol. 10 issue 6. 2955 -2959

[6] G. Chhetri, IJCRT vol. 10 issue 6 2022, ISSN: 2320-2882 c737- c741

[7] G. Chhetri IJARESM vol. 10 issue 6 2022 ISSN: 2455-6211, p 2309- 2312

- [8] S. S. Sawhney and R. M. Sati, Thermochim. Acta, 66 (1983), 351-355.
- [9] S. S. Sawhney and Kamal Nain, Thermochim. Act, 99 (1986), 373-378.

[10] S. S. Sawhney and S. K. Chandel, Thermochim. Acta, 86 (1985), 379-381.

[11] S. S. Sawhney, Thermochim. Acta, 74 (1984), 361.

[12] S. S. Sawhney and R. M. Sati, Thermochim. Acta, 70 (1983), 373.

- [13] S. S. Sawhney and N. K. Arora, Thermochim. Acta, 126 (1988), 393.
- [14] H. Lipson, Acta Cryst., 2 (1949), 43.

[15] N. F. M. Henry, H. Lipson and W. A. Wooster, 'The Interpretation of X-ray Diffraction Photography, Macmillan and Co. Ltd., New York, St. Martin Press, 1961.

