CRYSTAL STRUCTURE STUDIES OF METAL-PLANT AUXIN COMPLEXES BY X-RAY DIFFRACTION ANALYSIS

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ABSTRACT

The present investigation deals with crystal-structure studies of metal complexes of plant auxins using X-ray diffraction techniques. Complexes of Gd (III) with indole-3-butyric acid (IBA), Gd (III) with naphthalene acetic acid (NAA), and Dy (III) with naphthalene acetic acid were examined to determine their crystalline nature and lattice characteristics. Powder X-ray diffraction studies revealed well-defined crystalline structures. Analysis of diffraction peaks, interplanar spacing values, and Bragg reflections indicated primitive cubic lattice characteristics in the synthesized complexes. The calculated and observed d-spacing values showed close agreement, confirming the reliability of the structural assignments. The study demonstrates the usefulness of X-ray diffraction methods for determining lattice parameters and crystal symmetry of rare-earth metal complexes of plant growth regulators.

INTRODUCTION

Before the discovery of X-ray diffraction techniques, there was very little direct evidence regarding the systematic arrangement of atoms in crystalline solids. The development of crystallography and X-ray diffraction methods provided a powerful tool for investigating molecular and atomic structures in solids. X-ray diffraction soon became one of the most reliable methods for studying the crystal structures of inorganic and coordination compounds.

Crystallographic investigations are important in chemistry, metallurgy, materials science, and coordination chemistry. The arrangement of atoms and ions within a crystal lattice determines many physical and chemical properties of the material. Powder X-ray diffraction has therefore become an indispensable technique for identifying crystal systems, determining lattice parameters, and studying structural symmetry.

Plant auxins such as indole-3-butyric acid (IBA) and naphthalene acetic acid (NAA) are biologically important growth regulators capable of coordinating with metal ions through donor atoms present in their functional groups. Rare-earth metals such as gadolinium (III) and dysprosium (III) readily form stable complexes with these ligands. Structural investigation of such complexes is essential for understanding their coordination behavior and crystalline characteristics.

The present study deals with the crystal-structure determination of metal-plant auxin complexes using powder X-ray diffraction methods. The work focuses mainly on Gd(III)-IBA, Gd(III)-NAA, and Dy(III)-NAA complexes.

THEORETICAL BACKGROUND

The diffraction of X-rays by crystal planes follows Bragg's law:

$$n\lambda = 2d \sin\vartheta$$

where n is the order of reflection, λ is the wavelength of X-ray radiation, d is the interplanar spacing, and θ is the Bragg angle.

For a cubic lattice, the interplanar spacing relation is given by:

$1/d^2 = (h^2 + k^2 + l^2)/a^2$

where a is the lattice parameter and h, k, and l are the Miller indices. The quantity N is defined as:

$N = h^2 + k^2 + l^2$

Substitution into Bragg's equation gives:

$\sin^2\vartheta = (\lambda^2 / 4a^2)\, N$

Thus, for cubic systems, the common factor (C.F.) is obtained from the ratio of $\sin^2\theta$ to N:

$N = \sin^2\vartheta / C.F.$

The lattice parameter can be calculated from:

$a = d\sqrt{N}$

The density equation for cubic crystals is:

$\rho = nM / (N_a\, a^3)$

where ρ is the density of the crystal, n is the number of formula units per unit cell, M is the molar mass, N_a is Avogadro’s number, and a is the lattice parameter.

For face-centered cubic (fcc) lattices, reflections are permitted only when h, k, and l are all even or all odd. In body-centered cubic (bcc) lattices, reflections occur only when h + k + l is even. The presence or absence of these systematic reflection conditions helps in identifying the crystal system.

EXPERIMENTAL

Materials

All chemicals used were of analytical-grade purity. The metal complexes were synthesized according to standard procedures used for the preparation of rare-earth metal auxin complexes. The isolated complexes were recrystallized from acetone before recording their X-ray diffractograms.

X-Ray Diffraction Measurements

Powder X-ray diffraction patterns were recorded using a Philips PW-1710 X-ray diffractometer equipped with a PM-8210 single-pan recorder.

Instrumental conditions:

Radiation source: CuKα

Recorder speed: 1 cm min^{-1}

Goniometer speed: 2° 2θ min^{-1}

Operating current: 30 mA

Operating voltage: 40 kV

RESULTS AND DISCUSSION

The powder X-ray diffractograms of the Gd(III)-IBA, Gd(III)-NAA, and Dy(III)-NAA complexes showed sharp and well-defined diffraction peaks, indicating their crystalline nature.

The first few reflections obtained from the diffractograms were used to determine the common factor and identify the lattice type. From the observed 2θ values, the corresponding $\sin^2\theta$ values were calculated. Division of these values by the common factor yielded N values corresponding to $h^2 + k^2 + l^2$.

The observed sequence of N values was consistent with a primitive cubic lattice. Numbers such as 7, 15, 23, and 31 are absent because they cannot be represented as the sum of three squares for cubic indexing.

The diffraction reflections corresponded to possible sets of crystal planes for a primitive cubic system. Since the characteristic suppression conditions for fcc and bcc lattices were not observed, the possibility of face-centered cubic and body-centered cubic structures was ruled out.

Agreement between calculated and experimental d-spacing values further confirmed the validity of the indexing procedure and crystal assignment.

Table 1. Experimental Data from X-Ray Powder Diffractogram of Gd(III)-IBA Complex

2θ	θ	$10^3 \times \sin\theta$	$10^3 \times \sin^2\theta$	$\sin^2\theta$ / C.F.	h k l	a (Å)	d Found	d Calc.
6.40	3.20	55.8210	3.11598	1.016 (1)	1 0 0	13.79	13.79	13.94
9.00	4.50	78.4900	6.15582	2.007 (2)	1 1 0	13.87	9.81	9.86
12.60	6.30	109.7340	12.04160	3.927 (4)	2 0 0	14.03	7.02	6.97
18.00	9.00	156.4340	24.47100	7.98 (8)	2 2 0	13.92	4.92	4.93
22.20	11.10	192.5219	37.06400	12.08 (12)	2 2 2	13.85	4.00	4.03
23.20	11.60	201.0770	40.43200	13.18 (13)	3 2 0	13.81	3.83	3.87
25.40	12.70	219.8460	48.33200	15.76 (16)	4 0 0	14.01	3.50	3.50
26.00	13.00	224.4950	50.60000	16.55 (17)	4 1 0	14.14	3.43	3.38
26.80	13.40	231.7470	53.70700	17.57 (18)	3 3 0	14.10	3.32	3.29
28.00	14.00	241.9290	58.52600	19.09 (19)	3 3 1	13.87	3.18	3.20
30.00	15.00	258.8190	66.98700	21.85 (22)	3 3 2	13.95	2.98	2.97
31.20	15.60	268.9190	72.31900	23.58 (24)	4 2 2	14.03	2.87	2.85

Common Factor (C.F.) = 0.00306576

The calculated lattice parameter values remained nearly constant throughout the diffraction sequence. The average lattice parameter obtained for the Gd(III)-IBA complex was approximately 13.95 Å.

The close agreement between experimental and calculated d-values confirms the reliability of the primitive cubic indexing. The diffraction patterns therefore establish that the investigated metal-auxin complexes possess primitive cubic crystal structures.

CONCLUSION

Powder X-ray diffraction studies of rare-earth metal complexes of plant auxins revealed that the synthesized complexes are crystalline in nature. Analysis of diffraction peaks, Bragg reflections, and interplanar spacing values confirmed the presence of cubic lattice systems.

The absence of the characteristic reflection conditions of fcc and bcc lattices further supported the assignment of primitive cubic geometry. Agreement between observed and calculated d-spacing values validated the crystallographic calculations.

The study demonstrates that X-ray diffraction is a reliable and effective technique for determining crystal structures and lattice parameters of biologically important metal-auxin complexes.

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