

NOTE

Rare Earth Chelates with O, N Donor Ligands: Complexes of N-Hydroxy-N-Phenyl-N'-p-Anisyl Benzamidine with Nd(III), Sm(III), La(III), Pr(III) and Y(III)

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The rare earth (La, Pr, Nd, Sm, Gd, Y) with N-hydroxy-N-phenyl-N'-p-anisyl benzamidine (HPAB) has been synthesized and characterised on the basis of elemental analysis, IR spectra, electrolytic conductance and electronic spectral data. On the basis of the characterisation the composition of the complex seems to be $\text{Ln}(\text{HPAB})_3 \cdot 2\text{H}_2\text{O}$. All the studied complexes show that the rare-earth ions have six coordination number.

N-Hydroxy benzamidines have extensively been used as chelating agents.¹⁻⁴ N-Hydroxy-N-phenyl-N'-p-anisyl benzamidine (HPAB) acts as a nonselective ligand towards transition metal ions. The binary chelates of the transition metal have also been studied with this reagent. The present communication forms a part of our continued investigation on the N-hydroxy-N-phenyl-N'-p-anisyl chelates with Nd(III), Sm(III), La(III), Pr(III) and Y(III). The complexation between this reagent (HPAB) and

rare earth ions is attributed to the presence of $-\text{N}(\text{OH})-\overset{\text{I}}{\text{C}}=\text{N}-$.

The preparation of N-hydroxy-N-phenyl-N'-p-anisyl benzamidine consists in the condensation of equimolar quantities of N-phenyl hydroxylamine and N-p-anisyl benzimidoyl chloride in absolute ether⁵ at 0°-5°C. The purity of the reagent was checked by elemental analysis, TLC and IR spectra.

All the chemicals used were of AR grade and the solvents were purified by standard methods.

In the process of preparation of complex, the first step consists in the preparation of the hydrated lanthanide trichloroacetate. It is prepared by treatment of lanthanide oxide with aqueous solution of trichloroacetic acid. The solution was evaporated to dryness and solid mass was washed with chloroform for removal of excess of the acid.

Reagent (HPAB) was added to a solution of lanthanide trichloroacetate in methyl alcohol. The solution was refluxed for nearly one hour

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and insoluble complex filtered. The complex was washed with chloroform for removal of trichloroacetic acid formed in the reaction. Further the excess of lanthanide trichloroacetate was removed by washing with acetone.

Metal content was estimated by EDTA titration. IR spectra was recorded on Beckmann IR-12 spectrophotometer in KBr disc and electronic spectra of complexes were recorded in DMF in the region 350–700 nm using Beckmann spectrophotometer model-25. Toshniwal conductivity bridge was used to determine the electrical conductance on 10^{-3} M solutions in DMF.

Electrolytic conductance measurements on 10^{-3} M solutions in DMF show that these lanthanide complexes are nonelectrolytes. The complexes are soluble in ethanol, methanol and DMF but insoluble in chloroform.

The microanalytical results show the composition of these complexes to be $\text{Ln}(\text{HPAB})_3 \cdot 2\text{H}_2\text{O}$ where $\text{Ln} = \text{Nd}, \text{Sm}, \text{La}, \text{Pr}$ and Y . The presence of lattice water in the complexes may be ascertained by recording the loss in weight up to 150°C and no loss upto 200°C .

A comparison of the infrared spectral data of the ligand and complexes show the chelation of the cyano and oxime groups of the ligand. The $\nu_{\text{C-N}}$ and $\nu_{\text{O-H}}$ frequencies of the ligand observed at 1585 and 3080 cm^{-1} respectively shift to lower values in the complex. The N—O stretching vibration appearing at 945 cm^{-1} in the ligand are also observed to shift to lower frequency in the complex. A band in the region $3410\text{--}3395\text{ cm}^{-1}$ again confirms the presence of lattice water.

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