

NOTE

Fluoroboro- and Alkoxyboro-capped Encapsulated Compounds of Iron(II) Derived from Oxalodihydroxamic Acid

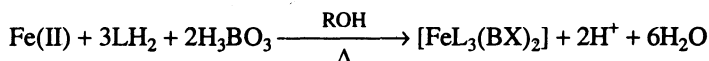
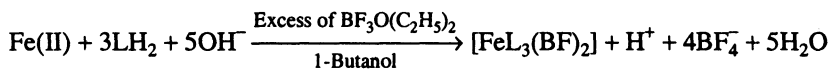
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A fluoroboro-capped iron(II) clathrochelate was synthesized in one step reaction by combining iron(II), dihydroxamic acid and boron trifluoride in butanol medium followed by the addition of KOH dissolved in butanol. A series of alkoxyboro-capped iron(II) clathrochelates were also synthesized in one step reaction by combining iron(II), dihydroxamic acid and boric acid. The alkoxy groups were provided by the corresponding alcohol. Elemental analyses correspond to the stoichiometry, $[\text{FeL}_3(\text{BX})_2]$ L = Oxalodihydroximate ion, X = F, OC_2H_5 , *i*- OC_3H_7 and *n*- OC_4H_9). The complexes have red to deep violet colour.

Earlier reported¹ fluoroboro-, alkylboro- and alkoxyboro-capped clathrochelates have been derived from dioxime ligands containing α -diimine moieties bonded to the metal ions. Our investigations²⁻⁴ on the various macrocyclic complexes with wide ranging structures inspired us to look for the systems that would provide structurally novel encapsulated complexes. The oxalodihydroxamic acid (Fig. 1) appeared promising. As a matter of fact, a series of fluoroboro- and alkoxyboro-capped iron(II) clathrochelates have been synthesized in one step reaction by combining iron(II), dihydroxamic acid and boric acid. The alkoxy groups were provided by the corresponding alcohols namely methyl, ethyl, isopropyl and *n*-butyl alcohols. The reactions occurring during the synthesis may be represented as:



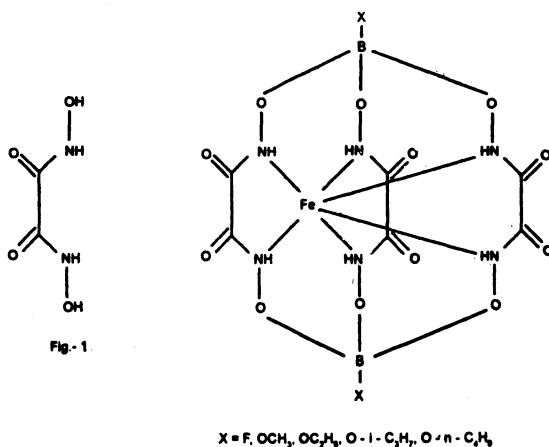
LH_2 stands for oxalodihydroxamic acid and X for OCH_3 , OC_2H_5 , *i*- OC_3H_7 or *n*- OC_4H_9 .

Infrared spectra of the free ligand are found to be rather simple and are well resolved. The bands corresponding to $\nu(\text{O}-\text{H})$, amide-I and amide-II are observed near 3210, 1616 and 1234 cm^{-1} respectively and for $\nu(\text{N}-\text{O})$ at 1030 and 830 cm^{-1} . On the other hand, the vibrational spectra of the fluoroboro- and alkoxyboro-capped complexes show complex patterns.

For the alkoxyboro-capped complexes additional bands appear near 960 and

800 cm^{-1} for $\nu(\text{B}-\text{O})$ and a number of vibrational bands in the region 1450–1280 cm^{-1} for deformation modes of vibration of the alkyl moieties. The fluoroboro complex shows $\nu(\text{B}-\text{F})$ near 1080 cm^{-1} apart from $\nu(\text{B}-\text{O})$. It is remarkable that the band due to $\nu(\text{O}-\text{H})$ of the free ligand has disappeared from the spectra of all the complexes implying that the capping occurs involving oxygen atoms of the oxime groups with the loss of protons.

The encapsulated complexes are all diamagnetic. Electronic spectra of the complexes show two to three intense bands which are located in the vicinity of 22,000 cm^{-1} and in the region 40,000–30,000 cm^{-1} . The earlier known¹⁻⁴ encapsulated complexes derived from dioximes show similar features and charge transfer bands of comparable energies. It gives the impression that the complexes possess similar geometries where the iron(II) ion is bonded to six nitrogen atoms in an antiprismatic geometry of the encapsulating ligands. The clathrochelates represent a novel class of complexes containing a sextet of exocyclic oxo groups as shown in Fig 1.



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