CHAPTER - I

Introductory aspects of ferrites

1.1 Introduction:

Ferrites are ferromagnetic materials with good dielectric properties and with large number of technological applications like antenna rods, inductors, magnetic cores, memory chips, filter components etc. [1]

These materials have attracted scientists and technologist because of their numerous applications. Due to their high electrical resistivity, low eddy current and dielectric loss ferrites are extremely used in microwave devices, computers, magnetic recording medium.

Ferrites are ferromagnetic oxides consisting of iron oxide as their main component and metal oxides. According to their crystal structure they can be classified as spinel ferrite, garnet and hexaferrite. Spinel ferrites are described by chemical formula $\text{MFe}_2\text{O}_4$ where M stands for divalent metal ions like Cu, Ni, Mg, Co, Mn, Cd, Zn, etc.
The method of preparation plays an important role in governing the structural, electrical and magnetic properties of ferrites. Various methods are used to prepare ferrite material. Usually ferrites are prepared by conventional ceramic technology. Recently some novel chemical methods have been used to prepare ferrite. The advantage of chemical methods is that they can produce ultra fine particles of nano size dimensions.

The physical properties of ferrites depend on the method of preparation and type and amount of dopant [2]. The physical properties of ferrites also depend on the distribution of cations among the tetrahedral (A) and octahedral [B] sites.

The basic properties of ferrites are known to be sensitive to the processing technique [3, 4] and processing parameters. Large numbers of researchers are using wet chemical methods to prepare nano size spinel ferrites. Recently, research on nano size ferrite has been enhanced because they exhibit excellent properties compared to their bulk material. The study of properties of magnetic nano particles is of great importance both from scientific value and technological value.

1.2 Composition and crystal structure:

Ferrites crystallize into the spinel structure, which is named after the mineral spinel, MgAl$_2$O$_4$. The well known soft ferrites, which are useful as high frequency core material and the square loop ferrites used in magnetic memory belong to the spinel group.
\( \gamma \)-Fe\(_2\)O\(_3\) which is possibly the most widely used magnetic recording material also has closely related structure.

Spinel ferrites are ceramic ferromagnetic materials having the general chemical composition MO-Fe\(_2\)O\(_3\), where M is a divalent metal ion such as copper, zinc, cadmium, manganese etc. or a mixture of these.

The spinel crystal structure is determined primarily by the oxygen and oxygen ion lattice. The radii of oxygen ions are several times larger than the radii of metallic ions in the compound as can be seen from Table 1.1.

Consequently, the crystal structure can be thought of as being made up of the closest possible packing of layers of oxygen ions, with the metallic ions fit in at the interstices.

The unit cell is cubic, each unit cell contains eight MeFe\(_2\)\(^{3+}\)O\(_4\) molecules. The space group is \( \text{Fd} \overline{3} \text{m} \). The oxygen ions form a nearly closely packed cubic structure and the metal ions are distributed over the tetrahedral and octahedral holes. In a unit cell, eight tetrahedral and sixteen octahedral sites are occupied by the metal ions. The tetrahedral and octahedral sites are conventionally called A and B sites respectively. The oxygen ion positions are defined by a crystallographic parameter ‘\( u \)’, which generally lies between 0.375 A.U. and 400 A.U.
The ion packing situation is indicated in Fig. 1.1 (a). Where, the solid lines represent the top layer of oxygen spheres while the dotted lines represent the bottom layer of oxygen spheres. Upon close examination, it can be seen that there are two kinds of interstices, denoted by letters A and B. A metallic ion located at the A site has four nearest oxygen ion neighbours, three oxygen ions in the bottom layers and one oxygen ion in the top layer (Fig. 1.1. b). In other words it is in a site of tetrahedral co-ordination. If the metallic ion is located at B site, it has six nearest oxygen ion neighbours. These oxygen ions are situated at the corners of an octahedron (Fig. 1.1. C). Thus, the metallic ion is in a site of octahedral co-ordination. For zinc and cadmium ferrites, the
divalent metallic ions Zn$^{++}$ or Cd$^{++}$ are at the A site, while the two trivalent ferric ions 2Fe$^{+++}$ are at the B site. This structure is same as that of normal spinel.

![Fig. 2. Crystal structure of spinel ferrite](image)

A diagram of the unit cell of the spinel crystal structure is shown in Fig. 1.2 where the Mg$^{++}$, Al$^{+++}$, and O$^{2-}$ ions are represented by spheres of different sizes and shapes. Actually most of the simple ferrites, e.g. NiFe$_2$O$_4$ are of the inverse spinel structure in which one trivalent ferric ion Fe$^{+++}$ is at the A site while the remaining trivalent ferric ion Fe$^{+++}$ and the divalent metallic ion M$^{++}$ are at the B site.

If M$^{++}$ is a ferrous ion, then the ferrite Fe$_3$O$_4$ can be represented in the ionic form Fe$_3$O$_4$ = FeO. Fe$_2$O$_3$ = Fe$^{+++}$ (Fe$^{+++}$ Fe$^{++}$) O$_4^{-}$.

It is interesting to note here that if electrons were to transfer from the divalent ferrous ion (Fe$^{++}$) at the B site to the trivalent ferric ion (Fe$^{+++}$) at the B site, the compound would remain
completely unchanged. Thus, it might be expected that electron flow would be facilitated, giving rise to high conductivity or low resistivity. The resistivity of iron ferrite Fe₃O₄ is about 10⁻⁴ ohm meter which is three orders of magnitude higher than that of iron, is till too low for high frequency application. However, if the ferrous ion Fe⁺⁺ is replaced by the nickel ion Ni⁺⁺, for example, the chemical formula would be:

$$\text{NiO.Fe}_2\text{O}_3 = \text{Fe}^{+++} (\text{Fe}^{+++} \text{Ni}^{++}) \text{O}_4^-$$

It is now no longer possible for electrons to be interchanged between the Fe⁺⁺⁺ and Ni⁺⁺ ions at the B sites without having the final state different from initial one. Indeed, by substituting a metallic ion such as Ni⁺⁺ for the ferrous ion in Fe₃O₄ (Magnetic), the resistivity can be increased to as high a value as 10⁷ ohm meter.

There are notable exceptions to the MO.Fe₂O₃ formula for ferrites. For e.g., Ferroxdure, a ferrite used for permanent magnets, has the chemical formula BaO.6Fe₂O₃. It has a hexagonal structure rather than the cubic crystal structure of the spinel, giving rise to high uni-axial anisotropy. Because of its high anisotropy field, itself resonates with no externally applied field at about 50 Kilo mega cycles/sec. Consequently, this material can be utilized to build light and compact resonance isolators at these high frequencies. Rare earth garnets are hard ferrites and also have large applications in many fields. They have the general chemical formula 5Fe₂O₃.3M₂O₃ where M represents yttrium or same other rare earth ions from
samarium to lutetium. This structure differs from the spinel lattice of the conventional ferrites in several respects.

1.3 **Types of ferrite:**

The distribution of the metal ions over the two sites is governed by the relative site preference energies of the various cations present in spinel lattice. On the basis of the cation distribution, ferrites may be classified into three classes.

Ferrites are composed of iron oxide as their main constituent and metal oxides. Depending upon the crystal structures ferrites are of following three types.

1) Spinel Ferrite,
2) Garnet and
3) Hexaferrite.

1) **Spinel Ferrite:**

Spinel ferrites are described by the chemical formula $\text{MFe}_2\text{O}_4$ where M stands for divalent metal ions. The crystal structure of spinel ferrite possess two interstitial sites namely tetrahedral A and octahedral B. A variety of cations can accommodate at tetrahedral (A) site and octahedral [B] site enabling wide variation in the properties of ferrites.

M can be replaced by other divalent metal ions and we can have number of spinel ferrites. Fe$^{3+}$ ions can be replaced by other trivalent ions like Al$^{3+}$, Cr$^{3+}$, Ga$^{3+}$ etc. Fe$^{3+}$ ions can also be replaced
by combination of divalent and tetravalent ions. The basic electric and magnetic properties of some spinel ferrites are summarized in Table 1.2.

2) Garnet:

The chemical formula for ferrimagnetic garnet is \( \text{Me}_3\text{Fe}_5\text{O}_{12} \) where Me is a trivalent ion such as rare earth or yttrium. The unit cell is cubic and contains eight molecules of \( \text{Me}_3\text{Fe}_5\text{O}_{12} \) i.e. (160 atoms). The metal ions are distributed over three types of sites. The Me ions occupy the dodecahedral sites (called c sites), where they are surrounded by eight oxygen ions, the \( \text{Fe}^{3+} \) ions distributed over the tetrahedral (d) and octahedral (a) sites in the ratio 3 : 2. Thus, the cation distribution of \( \text{Me}_3\text{Fe}_5\text{O}_{12} \) can be written as \( \text{Me}^3_3\text{Fe}^{2+}_2\text{Fe}^{4+}_3\text{O}_{12} \).

As in the case of spinels, the magnetic alignment results from super exchange interaction via the intervening oxygen ions, and the interaction is expected to be greater for the shorter the Me-O distance and the closer the Me-O-Me angle is to 180°. On this basis it is concluded that the interaction between the d and a cations are relatively strong (both negative). These interactions central the magnetic alignments in the solid, resulting in the following spin arrangement.

\[
\begin{align*}
\text{Me}^3_3\text{Fe}^{2+}_2\text{Fe}^{4+}_3\text{O}_{12} & \quad \text{for magnetic Me ion} \\
\text{Me}^3_3\text{Fe}^{2+}_2\text{Fe}^{4+}_3\text{O}_{12} & \quad \text{for non-magnetic Me ion}
\end{align*}
\]
The total magnetic moments on the a and d ions are aligned antiparallel and the moments on the c-ions are antiparallel to those on the d-ion. Thus for the formula \((3\text{M}_2\text{O}_3)^c(2\text{Fe}_2\text{O}_3)^a(2\text{Fe}_2\text{O}_3)^d\) the arrangement is \(6\text{Fe}^d, 4\text{Fe}^d, 6\text{M}^c\). The net magnetic moment \(m\) (in Bohr magneton per unit formula) is

\[
m = 6m^c-(6m^d-4m^a)=6m^c-10\mu_B
\]

assuming a magnetic moment of \(5\mu_B\) per Fe ion. In terms of the unit formula \(\text{M}_3\text{Fe}_5\text{O}_{12}\) equation 1.1 becomes

\[
m= (3m^c-5\mu_B)
\]

where, \(m^c\) is the magnetic moment per Me ion.

3) **Ortho-ferrites:**

Ortho-ferrites have the general formula \(\text{MeFeO}_3\), where Me is a large trivalent metal ion, such as rare-earth ion or Y. They crystallize in a distorted pervoskite structure with an orthorhombic unit cell. These ortho-ferrites show a weak ferromagnetism, which has been attributed to the small canting in the alignment of two anti-ferromagnetically coupled lattices. The canting angle is of the order of \(10^{-2}\) rad but is sufficient to introduce a small net ferromagnetic moment perpendicular to the anti-ferromagnetic axis. The direction of spin orientation of the Fe ion in \(\text{HOFeO}_3\) and \(\text{ErFeO}_3\) has been experimentally determined (9) at room temperature and found to be parallel to the (100) axis on lowering the temperature the spin axis rotates, and at 1.25K the direction is (001) for \(\text{HOFeO}_3\) and (110) for \(\text{ErFeO}_3\). The spin
moment on the rare earth ion gets ordered at a much lower Neel temperature [6.5 K for HoFeO$_3$ and 4.3 K for ErFeO$_3$].

4) **Hexagonal ferrites:**

There are a number of ferrites that crystallize in hexagonal structure, and some of them have gained considerable technological importance in recent years. These ferrites are further sub-classified into M, W, Y, Z and U compounds. All these have different, though related, crystal structures. The M compounds have the simplest structure. Barium ferrite, the well known hard ferrites, belongs to this class. These compounds have the general formula MeFe$_{12}$O$_{19}$ where Me is a divalent ion of a large ionic radius, such as Ba$^{2+}$, Sr$^{2+}$, or Pb$^{2+}$. Some compounds with trivalent Me (e.g. La$^{3+}$, Al, Ga, Cr, Fe) are also known. In these, one iron per formula unit is present as Fe$^{2+}$ to allow for the charge compensation.

The crystal structure of barium ferrite is hexagonal with the unit cell made up of two unit formulae. The structure is related to the spinel structure in which the oxygen lattice, fcc, consist of a series of hexagonal layers of oxygen lying perpendicular to the (111) direction.

1.4 **Classification of spinel ferrite:**

The distribution of metal ions over tetrahedral (A) and octahedral [B] site in spinel ferrite leads to the classification of
spinel ferrite as under normal spinel, inverse spinel and random spinel.

1. **Normal Spinel:**

   In normal spinel all divalent metal ions are distributed at tetrahedral (A) sites whereas all trivalent Fe$^{3+}$ ions are distributed at octahedral [B] sites. This can be expressed as, $\text{M}[\text{Fe}_2\text{O}_4]$. The typical example of normal spinel ferrites is ZnFe$_2$O$_4$ and Cd Fe$_2$O$_4$ [5, 6].

2. **Inverse Spinel:**

   In this type of spinel ferrite, half of the trivalent Fe$^{3+}$ ions are distributed at tetrahedral (A) sites and remaining half of the trivalent Fe$^{3+}$ ions are distributed at octahedral (B) sites together with all divalent ions. The distribution of cations is expressed as,

   $$(\text{Fe})^A[\text{MFe}]^B\text{O}_4^{2-}.$$ 

   The typical example of inverse ferrite is nickel ferrite (NiFe$_2$O$_4$) and cobalt ferrite (CoFe$_2$O$_4$) [7, 8].

3. **Random Spinel:**

   In random spinel ferrite, all divalent ions and trivalent Fe$^{3+}$ ions are randomly distributed over a tetrahedral (A) sites and octahedral (B) sites. In general, this is expressed as,

   $$\left(\text{M}^2\text{Fe}_{1-x}^{3+}\right)^A\left[\text{M}^{2+}_{1-x}\text{Fe}_{1-x}^{3+}\right]^B\text{O}_4^2.$$
CuFe$_2$O$_4$ and MgFe$_2$O$_4$ are the examples of partially inverted ferrites. The classification of spinel ferrite can be expressed by general formula:

$$(M_{1-x}Fe_x)^A[M_xFe_{2-x}]^BO_4$$

where, $x$ is distribution parameter.

If, $x = 0 \Rightarrow (M)^A[Fe]^BO_4 \Rightarrow$ normal spinel

$x = 1 \Rightarrow (Fe)^A[MFe]^BO_4 \Rightarrow$ inverse spinel

$0 < x < 1 \Rightarrow$ random spinel

1.5 **Applications of Ferrite:**

Ferrite has been recognized, as one of the most important electro-ceramics in modern industries and its processing and application technology has been improved incessantly in the last few years. From the 1950s, as radio and television spreads, ferrites established a significant position in industries and now ferrites are most essential material in electronic industries.

There are several types of ferrite materials being used in microwave components for obtaining non-reciprocal device action like isolator and phase shifting. The properties of the ferrites for use in devices at different frequency range is mostly designed by substituting diamagnetic metal ions in place of Fe$^{3+}$ in the chemical formula unit of the ferrite.

Ferrite belonging to magnesium ferrite family having inherent hysteresis loop characteristics was first to be identified for use in memory cores. Amongst the ferrites, hot pressed and
single crystal Mn-Zn ferrites are found quite suitable for this purpose. Mn-Zn ferrites are used as a core material in computer, microprocessor and VCR system. Ni-Zn and Mg-Zn ferrites core are found to be increasing importance in the transformer and telecommunication field as they offer inexpensive alternatives to many other soft magnetic materials. Ni-Zn ferrites are adequate for use in the high frequency band because of their high resistivity with sufficient low losses for microwave applications [9, 10].

The low loss polycrystalline ferrites should be used in a high frequency range for the good performance in applications and classified by the initial permeability. For the low and high frequency applications, the most important technological properties are saturation magnetization (\(M_s\)), coercive force (\(H_c\)), initial permeability (\(m_0\)) and losses. It is generally not possible to obtain the best combination of these properties for any specific application. By varying the compositions or adding additives or by varying the preparation technique, one can, to a large extent control most parameters for any particular application. Some of the other low frequency applications of soft ferrites include magnetic recording heads, inductor, transformer cores, filter cores etc. accordingly the material has to be developed and tailor-made.

For magnetic recording head, besides low \(H_0\), thermal stability and corrosion resistant also requires low magnetostriction and high frequency response.
1.6 Literature survey and aim of the present work:

Magnetic nano-particles are of great technological importance because of their applications in many fields viz. magnetic fluid, information storage system, medical diagnostics etc. various synthesis techniques have been employed for the synthesis of fine particles of ferrites, which exhibit superior property when compared to their bulk counter part.

Chemical co-precipitation, thermal decomposition, sol-gel, hydrothermal methods etc. non conventional methods have been largely used for the synthesis of magnetic nano particles. Ultra fine ferrite particles can be prepared by the simple chemical co-precipitation method.

Magnetic nanoparticles continue to be of fascinating subject both from fundamental and application point of view [11]. This is because of the difference in the physical and chemical properties of fine particles and bulk material.

Extensive work has been carried out on the synthesis and characterization of ultrafine particles for understanding the basic structural, electrical and magnetic properties of ferrite.

It has been reported that the method of preparation plays an important role with regard to chemical, structural and magnetic properties of a spinel ferrite [12].

Among the ferrite the copper ferrite CuFe\(_2\)O\(_4\) is supposed to be unique spinel ferrite because;
i) The presence of Cu$^{+2}$ ions leads to favour Jahn-Teller type distortion of the interstitial sites and

ii) The cation distribution over the non equivalent sites is variable and depends on the synthesis methods and synthesis conditions. [13].

Copper ferrite is a partially inverse ferrite. According to the literature survey, the copper ferrite has been studied for its magnetic and structural properties [14-15].

The structural and magnetic properties of Zn substituted manganese ferrite, cobalt ferrite, nickel ferrites are reported in the literature [16-18].

To our knowledge, the investigations of structural, electrical and magnetic properties of zinc substituted copper ferrite and chromium substituted copper ferrite prepared by wet chemical co-precipitation technique is not systematically studied.

Since zinc is non-magnetic and chromium is magnetic, it will be interesting to study the structural, electrical and magnetic properties of Cu$_{1-x}$Zn$_x$Fe$_2$O$_4$ and CuFe$_{2-x}$Cr$_x$O$_4$. Therefore, in the present work we have investigated the structural, electrical and magnetic proportion of Cu$_{1-x}$Zn$_x$Fe$_2$O$_4$ and CuFe$_{2-x}$Cr$_x$O$_4$ spinel ferrite system prepared by wet chemical co-precipitation method.

The structural properties were investigated by means of X-ray diffraction techniques (XRD), scanning electron microscopy (SEM), infrared spectroscopy (IR).
The magnetic properties were investigated by means of pulse field magnetization technique and a.c. susceptibility technique.

The electrical and dielectric properties were investigated by two-probe technique as a function of composition, frequency and temperature. The d.c. electrical resistivity of all the samples was measured in the temperature range 300-800K as a function of composition. The dielectric properties were studied using LCR-Q meter and in the frequency range 20 Hz-1MHz.
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### Table 1.1

Ionic Radii of Several Elements\(^a\)

<table>
<thead>
<tr>
<th>Ion</th>
<th>Radius $\text{\text{\AA}} = 10^{-8}\text{ cm}$</th>
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<tbody>
<tr>
<td>O$^-${}</td>
<td>1.32(^b)</td>
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<tr>
<td>Fe$^{+++}$</td>
<td>0.67</td>
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<td>Fe$^{++}$</td>
<td>0.83</td>
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<td>Mn$^{++}$</td>
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<td>Mg$^{++}$</td>
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<tr>
<td>Ni$^{++}$</td>
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<tr>
<td>Zn$^{++}$</td>
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<tr>
<td>Cd$^{++}$</td>
<td>1.03</td>
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<td>Co$^{++}$</td>
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<td>Cu$^{++}$</td>
<td>0.70</td>
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<tr>
<td>Al$^{+++}$</td>
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\(^b\) Computed by Goldschmidt on the basis of empirical assumptions.

\(^c\) Computed by Pauling on the basis of wave mechanics.
Table 1.2
Magnetic and electrical data of some spine ferrites

<table>
<thead>
<tr>
<th>Compound</th>
<th>Cation distribution</th>
<th>$a_0$ (Å)</th>
<th>Resistivity, Ohm cm</th>
<th>$n_B$ Cal.</th>
<th>$n_B$ Obs</th>
<th>$\theta_C$ °C</th>
<th>$K_1 \times 10^{-3}$</th>
<th>$K_1 \times 10^{-3}$</th>
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<tr>
<td>ZnFe$_2$O$_4$</td>
<td>Zn$^{2+}$ [Fe$_{2+}$]$^3_4$</td>
<td>8.44</td>
<td>$10^2$</td>
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<tr>
<td>MnFe$_2$O$_4$</td>
<td>Mn$^{2+}$ [Fe$_{2+}$]$^3_4$</td>
<td>8.51</td>
<td>$10^4$</td>
<td>5</td>
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<tr>
<td>FeFe$_2$O$_4$</td>
<td>Fe$^{3+}$ [Fe$<em>{2+}$ Fe$</em>{3+}$]$^3_4$</td>
<td>8.39</td>
<td>$4 \times 10^{-3}$</td>
<td>4</td>
<td>4.1</td>
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<td>(Fe$_3$O$_4$)</td>
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<td>CoFe$_2$O$_4$</td>
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<td>8.38</td>
<td>$10^7$</td>
<td>3</td>
<td>3.94</td>
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<tr>
<td>NiFe$_2$O$_4$</td>
<td>Fe$^{3+}$ [Ni$<em>{2+}$ Fe$</em>{3+}$]$^3_4$</td>
<td>8.34</td>
<td>$10^3$-$10^4$</td>
<td>2</td>
<td>2.3</td>
<td>585</td>
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<td>-17</td>
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<td>(quenched)</td>
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<tr>
<td>CuFe$_2$O$_4$</td>
<td>Fe$^{3+}$ [Cu$<em>{2+}$ Fe$</em>{3+}$]$^3_4$</td>
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<td>$10^5$</td>
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<td>2.3</td>
<td>455</td>
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<td>-10</td>
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<td>MgFe$_2$O$_4$</td>
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<td>$10^7$</td>
<td>0</td>
<td>1.1</td>
<td>440</td>
<td>-40</td>
<td>-6</td>
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<tr>
<td>Li$<em>{0.5}$Fe$</em>{2.5}$O$_4$</td>
<td>Fe$^{3+}$ [Li$<em>{0.5}$Fe$</em>{2.5}$]$^3_4$</td>
<td>8.33</td>
<td>$10^2$</td>
<td>2.5</td>
<td>2.6</td>
<td>670</td>
<td>-83</td>
<td>-8</td>
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<tr>
<td>$\gamma$ Fe$_2$O$_3$</td>
<td>Fe$^{3+}$ [Fe$<em>{5/3}$ Fe$</em>{1/3}$]$^3_4$</td>
<td>8.34</td>
<td>---</td>
<td>2.5</td>
<td>2.3</td>
<td>575</td>
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