A Structural Study of Cu-Zn Ferrites by Infrared Spectra

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ABSTRACT

Infrared (IR) spectra of Zn$^{2+}$ ions substituted Cu spinel ferrite, having the general formula $Cu_{1-s}ZnFe_2O_4$, (where $s$ stepped by 0.2 such that $0.0 \leq s \leq 1.0$), have been analyzed in the frequency

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range \((200-1000) \text{cm}^{-1}\). The above mixed ferrites were prepared by the conventional standard double sintering ceramic method. Four absorption bands were observed, their position and intensity were found to be strongly dependent on \(s\)-value. Mainly, two prominent bands were observed, a high frequency band \(\nu_T\) at around \((536-568) \text{cm}^{-1}\) and a low frequency band \(\nu_O\) at around \((394) \text{cm}^{-1}\) were assigned to tetrahedral \(T_d\) and octahedral \(O_h\) sites, respectively, of spinel lattice. Upon introducing \(Zn^{2+}\) ions, IR spectra indicated new shoulders or splitting in the absorption bands, such the first primary band \(\nu_T\) which consists of two shoulders at about \((700\text{ and } 600) \text{cm}^{-1}\). A small absorption band \(\nu_3\) was observed at about \((300-325) \text{cm}^{-1}\). This may indicate the presence of the divalent metal ion-oxygen complex on \(O_h\) sites. Other small weak absorption band \(\nu_4\) was, also, observed at about \((265-270) \text{cm}^{-1}\). Its intensity increased with the addition of \(Zn^{2+}\) ions. Force constant \(F_C\) was calculated for \(T_d\) and \(O_h\) sites, and was found to decrease with increasing \(Zn^{2+}\) ions. Half bandwidths of \(T_d\) sites \((\Gamma_T)\) and \(O_h\) sites \((\Gamma_o)\) changed linearly with increasing \(Zn^{2+}\) ions. Threshold frequency \(\nu_{th}\) for the electronic transition was determined and found to increase with increasing \(Zn^{2+}\) ions. On the basis of the analysis of IR absorption bands, we concluded the cations distribution for the given mixed ferrite. The ionic radii for each site were correlated to the cations distribution of the given ferrite.

**Keywords:** Ferrites, Infrared, copper zinc ferrites.
INTRODUCTION:

Magnetic and electric properties of the ferrite materials strongly depend on their chemical and physical structure, in the determination of the precise configuration of the atoms and the ions in the ferromagnetic semiconductor [1].

In particular, the IR spectra of the ferrite materials are an important tool to describe the various ordering problems, also, in the investigation of the structural properties of the mixed ferrites. They give information not only about the positions of the ions in the spinel lattice crystal, but, also, about their vibration mode. The vibration spectra can be indicated to the valance state of the ions and their occupation in the spinel lattice crystal. Application of the IR spectroscopy to the ferrite materials is to detect the completion of the solid-state reaction, the cations distribution and the deformation of spinel structure [2].

The IR spectral absorption bands mainly appear due to the vibrations of the oxygen ions with the cations producing various frequencies in the unit cell. In a certain mixed spinel ferrite materials, as the concentration of the divalent metal ions increasing, it gives rise to a structural change or cation distribution in the spinel crystal lattice without affecting the spinel ferrite structure [1]. The structural changes brought about by the metal ions that are either lighter or heavier than divalent ions in the ferrites strongly influence the lattice vibrations. The vibration frequency depends on the cation mass, the cation oxygen distance and the bonding–force [3].

Many researchers studied the IR spectra of several ferrites [4-8]. However, no reports were found in the literature regarding IR spectra of $Zn^{2+}$ ions substituted $Cu$ spinel ferrite. Therefore, in the present communication we analyzed the IR spectra of mixed $Cu-Zn$ spinel ferrite.

EXPERIMENTAL TECHNIQUE:

Polycrystalline mixed ferrite of the general formula $Cu_{1-s}Zn_sFe_2O_4$, (where $s$ stepped by 0.2 according to $0.0 \leq s \leq 1.0$), was prepared by the conventional standard double sintering ceramic method. Weighted high purity metal oxides were mixed, and then ground to a very fine powder for 5 hours. Then the mixed powders were presintered at $(750\,^\circ\,C)$ for 3 hours soaking time. After that, the prefired powder was well ground for 3 hours and by using a small quantity of butyl alcohol as a binding material. Samples pressed with hydraulic press under constant pressure of
(3×10⁸ Kg.cm⁻²) in the form of a disc shape with diameter (11mm). All samples sintered for 5 hours soaking time at (1100°C). For each sintering of the samples were cooled down gradually to room temperature with a rate of 1°C/min. For recording IR spectra, the powder of the samples were mixed with (KBr) in the ratio (1:200) by weight to ensure uniform dispersion in KBr pellet. The mixed powder of samples were then placed in a cylindrical disc and pressed at (3×10¹⁰ Kg.cm⁻²) by a hydraulic press. Clean discs of (1mm) thickness were obtained. IR spectra measurements were carried out at room temperature in the range from (200-1000) cm⁻¹ using Berkin Elmer Model 883 infrared Spectrophotometer.

RESULTS AND DISCUSSION:

The values of the absorption bands frequency are given in Table (1), while Figure (1) indicates the IR spectra of the mentioned compounds. It illustrates the presence of two prominent absorption bands, along with some side bands in the range from 200 cm⁻¹ up to 800 cm⁻¹ regions. It shows that, the higher absorption bands νₜ are in the range from 536 cm⁻¹ up to 568 cm⁻¹ and the lower absorption bands are νₒ located around 394 cm⁻¹. This is in a good agreement with the observation of many researchers for various ferrite materials [4-6]. They found that, νₜ accrues in the frequency range from 550 cm⁻¹ up to 600 cm⁻¹, attributed to the stretching intrinsic vibrations of the Tₜ group complex corresponding to the highest restoring force. However, νₒ was observed in the frequency range from 300 cm⁻¹ up to 450 cm⁻¹, which can be correlated with the stretching intrinsic vibrations of the Oₜ group complex (bond-bending vibrations). Several scientific research [1,2,7] have reported that νₜ greater than νₒ, and the bands νₜ and νₒ are characteristic of the lattice vibration of the metallic oxides, arise from the vibration of the oxygen ions against the metal ions in the lattice Tₜ and Oₜ complex.
Table (1): Absorption bands frequency for the $T_d$ and the $O_h$ sites of the mixed Cu-Zn spinel ferrite.

<table>
<thead>
<tr>
<th>s</th>
<th>$T_d$ sites</th>
<th>$O_h$ sites</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\nu_{1sh.} cm^{-1}$</td>
<td>$\nu_{2sh.} cm^{-1}$</td>
</tr>
<tr>
<td>0.0</td>
<td>-</td>
<td>600</td>
</tr>
<tr>
<td>0.2</td>
<td>-</td>
<td>600</td>
</tr>
<tr>
<td>0.4</td>
<td>700</td>
<td>600</td>
</tr>
<tr>
<td>0.6</td>
<td>700</td>
<td>600</td>
</tr>
<tr>
<td>0.8</td>
<td>700</td>
<td>600</td>
</tr>
<tr>
<td>1.0</td>
<td>700</td>
<td>600</td>
</tr>
</tbody>
</table>

It is clear that, $\nu_T$ are caused by the stretching of the $T_d$ metal ions and the oxygen bonding, while $\nu_O$ are caused by the vibrations of the oxygen ions in the direction perpendicular to the axis joining the $T_d$ ions and the oxygen ions [2]. The IR spectra in Figure (1) indicate the existence of new shoulders or splitting in the absorption bands, i.e. the first primary band $\nu_T$ consists of two shoulders at $\nu_{1sh.} = 700 \text{ cm}^{-1}$ and $\nu_{2sh.} = 600 \text{ cm}^{-1}$. Similar results have been observed in various ferrite systems [2,8]. It has been shown by Potakova [9] that, the presence of the $Fe^{2+}$ ions in spinel ferrite can produce splitting or shoulders on the absorption bands. This is Jahn–Teller distortion produced by the $Fe^{2+}$ ions. This distortion causes local deformation in the spinel lattice owing to the noncubic component of the crystal field potential and, hence, leads to splitting of the band $\nu_T$ [10].

A third absorption band $\nu_3$ appears in the range from $300 \text{ cm}^{-1}$ up to $325 \text{ cm}^{-1}$, because a small amount of $Fe^{2+}$ ions are produced in the mentioned compounds. This band is attributed to the $Fe^{2+}-O^{2-}$ bond of the $O_h$ sites [8,10]. It appears relatively narrow and weak in intensity, and the intensity increases with the increasing $Zn^{2+}$ ions. This tends to the $\nu_O$ band becomes narrow and the $\nu_3$ more intense, therefore, $\nu_3$ could be seen very clearly at high concentration of the $Zn^{2+}$ ions.

Another absorption band $\nu_4$ appears in the range from $265 \text{ cm}^{-1}$ up to $270 \text{ cm}^{-1}$. It is attributed to some types of lattice vibration involving displacement of
the $T_d$ cations in the samples.

Fig.(1): IR absorption spectra of the mixed Cu-Zn spinel ferrite
Its frequency depends on the mass of the foreign atom occupying the \( T_d \) sites in the ferrite samples and the mass of the divalent cations \([1,11]\). Its intensity is small compared to any other absorption bands, which increases with increasing of the \( Zn^{2+} \) ions. The \( IR \) spectrum of \( CuFe_2O_4 \) was studied by Mazen \([12]\). His study indicated that, there were two mainly absorption bands where \( \nu_T = 570 \text{ cm}^{-1} \) and \( \nu_O = 395 \text{ cm}^{-1} \). However, \( ZnFe_2O_4 \) was studied by several investigators \([4,10,13,14]\). They reported that, there were four bands given by \( (\nu_T = 555 \text{ cm}^{-1}, \nu_O = 393 \text{ cm}^{-1}, \nu_3 = 325 \text{ cm}^{-1} \) and \( \nu_4 = 169 \text{ cm}^{-1} \).

Referring to Table (1), it is noticed that, \( \nu_T \) shifts to the lower frequency with increasing the \( Zn^{2+} \) ions. The \( \nu_T \) and the \( \nu_O \) behavior with increasing of \( Zn^{2+} \) ions is illustrated in Figure (2). The same behavior was seen in previous works of different ferrite systems \([8,15]\).

A difference in the band position of \( \nu_T \) in the range from 536 \text{ cm}^{-1} \) up to 568 \text{ cm}^{-1} \) and of \( \nu_O \) (which has a constant value) is expected because of the difference in the \( Fe^{2+} - O^{2-} \) bond length for the \( T_d \) and the \( O_h \) complex \([2,4,8,10]\). It was found that, the \( Fe^{2+} - O^{2-} \) bond length of the \( T_d \) sites (0.189 \text{ nm}) is shorter than that of the \( O_h \) sites (0.199 \text{ nm}) \([16]\). This has been interpreted on the base of covalent bonding of the \( Fe^{3+} \) ions at the \( T_d \) sites. Since the vibration frequency is proportional to the force constant \( F_C \), so the \( Fe^{2+} - O^{2-} \) bond vibration (\( \nu_T \)) is shifted to a lower frequency with increasing the \( Zn^{2+} \) ions. This indicates that, \( F_C \) of the \( Fe^{2+} - O^{2-} \) bond is decreasing in the mixed \( Cu-Zn \) spinel ferrite. The calculated values of the force constant \( F_{CT} \) and \( F_{CO} \) for the \( T_d \) and the \( O_h \) sites, respectively, are listed in Table (2) using the following relation \([8]\)

\[
F_C = 4\pi^2 c^2 \nu^2 m
\]

where:

- \( c \) is the light velocity \( \approx 2.99 \times 10^{10} \text{ cm} \cdot \text{sec}^{-1} \).
- \( \nu \) is the vibration frequency of the \( T_d \) and the \( O_h \) sites.
- \( m \) is the reduced mass for the \( Fe^{2+} \) ions and the \( O^{2-} \) ions, it is found equal \( \approx 2.061 \times 10^{-23} \text{ gm} \).
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Fig. (2): Behavior of the absorption bands $v_T$ and $v_O$ with the composition $s$.

Table (2): Calculated values of the force constant $F_{CT}$ and $F_{CO}$.

<table>
<thead>
<tr>
<th>s</th>
<th>$(F_{CT} \times 10^5)$ dyne.cm$^{-1}$</th>
<th>$(F_{CO} \times 10^5)$ dyne.cm$^{-1}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>2.361</td>
<td>1.142</td>
</tr>
<tr>
<td>0.2</td>
<td>2.312</td>
<td>1.136</td>
</tr>
<tr>
<td>0.4</td>
<td>2.247</td>
<td>1.136</td>
</tr>
<tr>
<td>0.6</td>
<td>2.182</td>
<td>1.136</td>
</tr>
<tr>
<td>0.8</td>
<td>2.125</td>
<td>1.136</td>
</tr>
<tr>
<td>1.0</td>
<td>2.103</td>
<td>1.136</td>
</tr>
</tbody>
</table>

The IR spectra of the mentioned compositions show a change in the absorption bands by introducing the $Zn^{2+}$ ions, of larger ionic radius and higher atomic weight, on the $T_d$ sites. This tends to increase the ionic radius of the $T_d$ and the $O_h$ sites with addition of the $Zn^{2+}$ ions [2]. This can be ascribed to the method of preparation, the grain size and the porosity that influence the band position [1,4,10]. Consequently this affects the $Fe^{2+} - O^{2-}$ bond stretching vibration [17]. Therefore, there are slightly shifts towards low frequency in the $v_T$ and the $v_O$ bands. Clearly, for the
normal spinel ferrites both the two prominent bands depend on the nature of the $O_h$ ions and fewer on the $T_d$ ions [18], such as Zn spinel ferrite. Referring to the IR spectra, it is noticed that, the half bandwidths of the $T_d$ sites, $\Gamma_T$, and of the $O_h$ sites, $\Gamma_O$, change linearly with the increasing the Zn$^{2+}$ ions.

Table (3) shows the values of $\Gamma_T$, $\Gamma_O$, and $\Gamma_T/\Gamma_O$. The behavior of $\Gamma_T$ and $\Gamma_O$ with the composition $s$ is illustrated in Figure (3), while the ratio $\Gamma_T/\Gamma_O$ is represented in Figure (4). The ratio $\Gamma_T/\Gamma_O$ seems to increase linearly with the composition $s$. $\Gamma_T$ and $\Gamma_O$ depend on the statistical distribution of the various cations over the $T_d$ and the $O_h$ sites [19]. This distribution depends on the replacement process of the smaller ionic radii of the $Fe^{3+}$ ions with (0.067nm) and the $Cu^{2+}$ ions with (0.076nm), by the larger ionic radius of the Zn$^{2+}$ ions with (0.084nm). Thus, there is a good correlation of $\Gamma_T$ and $\Gamma_O$ with the ionic radii $R_T$ and $R_O$ of the $T_d$ and the $O_h$ sites, respectively, as will explained literally.

![Graph showing variation of $\Gamma_T$ and $\Gamma_O$ with the composition $s$.](image)

**Fig. (3): Variation of $\Gamma_T$ and $\Gamma_O$ with the composition $s$.**
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![Graph](image)

**Fig. (4):** Change of $\Gamma_T/\Gamma_O$ with the composition $s$.

**Table (3):** Values of $\nu_{th}$, $\Gamma_T$, $\Gamma_O$ and $\Gamma_T/\Gamma_O$ for the mixed Cu-Zn spinel ferrite.

<table>
<thead>
<tr>
<th>$s$</th>
<th>$(\nu_{th})\text{cm}^{-1}$</th>
<th>$(\Gamma_T)\text{cm}^{-1}$</th>
<th>$(\Gamma_O)\text{cm}^{-1}$</th>
<th>$\Gamma_T/\Gamma_O$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>760</td>
<td>140</td>
<td>90</td>
<td>1.56</td>
</tr>
<tr>
<td>0.2</td>
<td>770</td>
<td>145</td>
<td>85</td>
<td>1.71</td>
</tr>
<tr>
<td>0.4</td>
<td>780</td>
<td>150</td>
<td>81</td>
<td>1.85</td>
</tr>
<tr>
<td>0.6</td>
<td>790</td>
<td>155</td>
<td>78</td>
<td>1.98</td>
</tr>
<tr>
<td>0.8</td>
<td>800</td>
<td>160</td>
<td>76</td>
<td>2.1</td>
</tr>
<tr>
<td>1.0</td>
<td>810</td>
<td>165</td>
<td>75</td>
<td>2.2</td>
</tr>
</tbody>
</table>

According to Waldron [4], the threshold frequency $\nu_{th}$ for the electronic transition can be determined from the maximum point of absorption spectra where it reaches a limiting value. Table (3) shows these threshold values, which are illustrated in figure (5). It is found that, the threshold frequency $\nu_{th}$ increases with increasing of the Zn content.

This increment in $\nu_{th}$ is not reflected on the corresponding value of the activation energy $E_a$. The threshold energy $E_{th}$ was calculated from the relation $E_{th} = h \nu_{th}$ that has a constant average value about 0.1$eV$, where $h$ is Planck’s constant.
It is necessary to know the cations distribution, which will be suggested as the following considerations. When the non-magnetic $\text{Zn}^{2+}$ ions occupy the $T_d$ sites, which are favored by the polarization affect, this will occur by replacing the $\text{Fe}^{3+}$ ions in the $T_d$ sites. However, the $T_d$ sites preference of the cations depends upon their electronic configuration [20]. The $\text{Zn}^{2+}$ ions show a marked stronger preference for the $T_d$ sites than the $\text{Fe}^{3+}$ ions, where their (4s 3d) electrons form a covalent bond with 2p electrons of the oxygen ion [21,22].

In the light of the above considerations, the cations distribution for the mixed $\text{Cu-Zn}$ spinel ferrite can be written as follows:

$$\text{(Zn}^{2+}\text{Fe}^{3+}_{1-x})_T\{\text{Cu}^{2+}\text{Fe}^{3+}_{1+x}\}_O\text{O}_{4}^{-}$$

The above formula has a good agreement with the cations distribution, which was suggested by several authors [17,23-25].

To calculate the ionic radius for the $T_d$ and the $O_h$ sites, we used the following equations [9,26]

![Fig. (5): Change of $\nu_{10}$ with the composition $s$.](image)
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\[ R_T = sR_{Zn^{2+}} + (1-s)R_{Fe^{3+}} \quad (2) \]
\[ R_O = \frac{1}{2}[(1-s)R_{Cu^{2+}} + (1+s)R_{Fe^{3+}}] \quad (3) \]

where:
\( R_T \) and \( R_O \) are the mean ionic radii per molecule for the \( T_d \) and the \( O_h \) sites, respectively.
\( R_{Zn^{2+}} \) is the ionic radius of the \( Zn^{2+} \) ion
\( R_{Fe^{3+}} \) is the ionic radius of the \( Fe^{3+} \) ion.
\( R_{Cu^{2+}} \) is the ionic radius of the \( Cu^{2+} \) ion.

In Figure (6), the ionic radii \( R_T \) and \( R_O \) are plotted versus the composition \( s \), while \( R_T/R_O \) is, also, plotted versus the composition \( s \) in Figure (7). From these figures, it is clear that, \( R_T \) increases while \( R_O \) decreases with increasing of the \( Zn^{2+} \) ions. This behavior is attributed to the replacement of the \( Fe^{3+} \) ions to the larger ionic radius of the \( Zn^{2+} \) ions on the \( T_d \) sites and the \( Cu^{2+} \) ions to the smaller ionic radius of the \( Fe^{3+} \) ions on the \( O_h \) sites. Therefore, increase the \( Zn^{2+} \) ions leads to the change of \( \Gamma_T \) and \( \Gamma_O \) as shown in Figure (3).

From the \( IR \) spectra of the mixed Cu-Zn spinel ferrite in Figure (1), it is observed that, when the \( Zn^{2+} \) ions increase, a change in the intensity of \( T_d \) and \( O_h \) absorption band occurs. Other researchers found this behavior [2,8,12], for different ferrite materials.

The intensity of the \( IR \) absorption bands is function of the concentration and thickness of the samples in solid solutions [27]. This intensity of the \( T_d \) and the \( O_h \) bands depends on the ionic replacement and consequently on the magnetic dipole moment. Changing the magnetic dipole moment with the inter-nuclear distance during \( d\mu/dr \) depends on the intensity \( E \) of the absorption band. It is defined by [28]

\[ E = 10^{-3} N_o \frac{N_o}{2mc^2} \left( \frac{d\mu}{dr} \right)^2 \quad (4) \]

where \( N_o \) is the Avogadro's number.
It is clear that, the intensity values give an induction of the $d\mu/d\tau$ values. The latter value represents the contribution of the ionic character of the $Fe^{2+} - O^{2-}$ bond in the spinel lattice [8,29].

The change of the intensity with increasing the $Zn^{2+}$ ions is explained on the basis of the cations distribution of the mixed Cu-Zn spinel ferrite, which is given before. From this form, it will be seen that, as the $Zn^{2+}$ ions increase the $Cu^{2+}$ ions decrease in the $O_h$ sites and the $Fe^{3+}$ ions leave the
$T_d$ sites to the $O_h$ sites. This may disturb the $Fe^{2+} - O^{2-}$ bond of $T_d$ and $O_h$ sites in the spinel lattice [8].

The $Fe^{2+} - O^{2-}$ bond exerted by the $Fe^{2+}$ ions is formed in the samples during the sintering process. It is to be noted that, the sintering process affects the number of the $Fe^{2+}$ ions in each site. On the other hand, the electronic distribution of the $Fe^{2+} - O^{2-}$ bond is greatly affected when the $Zn^{2+}$ ions are introduced, which consequently affects the value of $d\mu / dr$ of the $Fe^{2+} - O^{2-}$ bond, as measured by the absorbance of the IR absorption bands.

From the previous discussion, one can conclude that the IR spectra can give information about changes in the molecular structure of the ferrites, due to the perturbation occurring in the $Fe^{2+} - O^{2-}$ bond upon introducing of the $Zn^{2+}$ ions.

**CONCLUSION:**

Infrared spectra of the diamagnetic $Zn^{2+}$ ions substituted on the $Cu$ spinel ferrite have been analyzed in the frequency range $(200-1000)cm^{-1}$. Four bands were observed, their positions and intensities were found to depend strongly on the composition $s$.

1. The IR spectra indicated that two main prominent bands are detected, one of a high frequency band $\nu_T$ at around $(532-568)cm^{-1}$ and the other of a low frequency band $\nu_O$ at $395 cm^{-1}$ and are assigned to the $T_d$ and the $O_h$ sites, respectively.

2. Upon introducing the $Zn^{2+}$ ions, the IR spectra indicated new shoulders or splitting in the absorption bands, the primary bands $\nu_T$ are consisted of two shoulders at $700 cm^{-1}$ and $600 cm^{-1}$.

3. Other subsidiary absorption band $\nu_3$ was observed in the range 300-325$cm^{-1}$. It is related to the presence of the divalent metal band ion-oxygen complex on the $O_h$ sites.

4. Other small weak absorption band $\nu_4$ was observed at $266 cm^{-1}$, its intensity increased with increasing the $Zn^{2+}$ ions.
5. Force constant $F_C$ of the $T_d$ and the $O_h$ sites were calculated and found to decrease with increasing the $Zn^{2+}$ ions.
6. The half bandwidth of the $T_d$ sites was assigned to $\Gamma_T$ and of $O_h$ sites was assigned to $\Gamma_O$, they change linearly with increasing the $Zn^{2+}$ ions.
7. The threshold frequency $v_{th}$ of the electronic transition was determined and found to increase with increasing the $Zn^{2+}$ ions.
8. On the basis of analyzing the IR absorption bands, we deduced the cations distribution for the given mixed ferrite as the following form $(Zn^{2+}_{2s}Fe^{3+}_{2s})Cu^{2+}_{2s}O^{2-}_{2s}$. This illustrated that the non-magnetic $Zn^{2+}$ ions has a preference to the $T_d$ sites, where the $Cu^{2+}$ ions occupy the $O_h$ sites.
9. The ionic radii of the $T_d$ and the $O_h$ sites were calculated and found to change linearly with increasing the $Zn^{2+}$ ions.

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