**Transport Properties of Ce Doped Cd Ferrites (*CdFe2-xCexO4* ) ()**

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**ABSTRACT**

Cadmium ferrites belong to normal spinel ferrites and they exhibit interesting electrical, magnetic, and optical properties. The pure and cerium doped cadmium ferrites (x = 0.0, 0.125, 0.250, 0.375, 0.5) were synthesized by a chemical co-precipitating technique using sodium hydroxide as a co-precipitating agent. The structures and phase purity of fabricated nanomaterials were analyzed by X-ray diffraction (XRD). The crystallite size for all the prepared nanomaterials was in the range of 28-46 nm. The lattice constant and unit cell volume were found to decrease with the increasing concentration of Cerium, which was confirmed by the peak shift in the XRD pattern. The X-ray density for all Nano ferrites increased with the enhancement of Cerium composition. The resistivity of the nanomaterials has random behavior with the enhancement of cerium composition for a temperature, but the value of resistivity at x = 0.125 has the lowest value and at x = 0.375 has the highest value for almost all temperatures. For specific concentrations, a decreasing trend of resistivity of fabricated materials was found with an increment of temperature. The activation energies were also calculated, and it increased for x = 0.125 and then decreased for all the nanomaterials. For the confirmation of the M-O bonds, FTIR analysis of all the nano ferrites was also performed. The analysis shows a higher frequency absorption band in the range of 531.24-534.84 cm-1. This absorption band confirms that metal oxides are formed in all the synthesized nanoparticles.

**Keywords:** spinel; ferrites; absorption band; resistivity; activation energies.

**1. Introduction**

A hefty category of oxides are the ferrites which possess extraordinary magnetic characteristics. The researchers showed special interest in the investigation and application of these ferrites during the last fifty years. The applications of ferrites are included in the handling of power, electronic technology, and in the field of biotechnology. Spinel ferrites and natural spinel MgAl2O4 which was first found by Bragg possess identical crystal structures. Magnetite (Fe3O4) is an important ferrite, perhaps the oldest which has many applications. Research on ferrites is a very active research field nowadays. It has mesmerizing properties coupled close to the presence of and cations together. Soft ferrites have many applications in microwave devices, thermo-junctions, high-frequency cores, humidity sensors, antennas, multilayer chip indicators, high-frequency transformers, ferrite wave absorbers, electronics, and magnetic storage devices [[1](#_ENREF_1), [2](#_ENREF_2)].

The structure of ferrite may be tailored, although fundamental configuration continues to exist identical. Nanocrystalline magnetic materials can be obtained by different methods such as co-precipitation [[1](#_ENREF_1), [3-6](#_ENREF_3)], hydrothermal [[7](#_ENREF_7)], sonochemical [[8](#_ENREF_8)], citrate precursor [[9](#_ENREF_9)], sol-gel auto combustion [[10-12](#_ENREF_10)], etc. Our present study is on soft ferrites which have many applications such as microwave devices, thermo-junctions, high-frequency cores, humidity sensors, antennas, multilayer chip inductor (MLCI), high-frequency transformers, ferrite wave absorber, electronics, magnetic storage devices [[13](#_ENREF_13)].

The physical properties of ferrites are powerfully dependent on many parameters, like particle size, crystallite dimensions, cationic distribution, and their purity is extensively interrelated with the method of synthesis of the ferrites [[14](#_ENREF_14)].

Ferrites display interesting and fruitful variations in electrical properties by the inclusion of various cations within ferrite materials [[15](#_ENREF_15)]. Structural and electrical characteristics of ferrites were upgraded by many researchers by the inclusion of rare-earth ions [[16-19](#_ENREF_16)].

In our present study Ce-Cd system was synthesized, with general formula , where 0 (Ce3+ doped CF). The rare earth metal cerium was doped in the cadmium soft ferrites which have a normal spinel structure. Five samples were prepared with compositions x = 0.0, 0.125, 0.25, 0.375, 0.5 by chemical Co-precipitation approach to observe their electrical, dielectric and structural characteristics. The content of rare earth metal cerium was increased in regular steps and the nanoparticles were fabricated for the said compositions. At last, the prepared samples were characterized using XRD, FTIR, LCR, and I-V.

**2. Materials and method**

To prepare , metal solutions of required molarities were prepared by dissolving cadmium nitrate, cerium chloride, and iron chloride in deionized water. Cerium doped cadmium ferrites CdFe2-xCexO4 were synthesized by using the highly pure cadmium nitrate tetrahydrate Cd (NO3)2.4H2O, ferric chloride hexahydrate FeCl3.6H2O, and cerium chloride heptahydrate CeCl3.7H2O. Five samples were prepared with the composition x=0.0, 0.125, 0.25, 0.375, 0.5. There are several methods to dope cerium into cadmium soft ferrites, but the chemical co-precipitation method was used in this study. Materials were measured according to the stoichiometry calculation. The measured quantities of these materials were mixed and dissolved into 100 ml of de-ionized water in the beakers separately. These solutions of the samples were stirred on the stirrer and a solution of NaOH was poured dropwise into the solution until the pH of the solution became 11 and the stirring process was stopped. After this solution was put into a water bath maintained at a temperature of 80 for 24 hours. The pH of the solution was made 7 by washing precipitates with deionized water. The substance that was left after washing was dried in an oven for 36 hours. After that, each sample was ground in a pestle and mortar for 1hour to make it in powder form. After grinding, samples were placed in a furnace for sintering at 900 for 8 hours. The purpose of sintering is to compact the material. After sintering the substance was ground further for 1 hour in the pestle and mortar to make it powder. This powder was characterized using XRD, I-V, LCR, and FTIR.

**3. Results and their Discussion**

**3.1 Structural analysis**

The XRD patterns of Ce3+ doped CF samples are shown in Fig 1(a). The diffraction peaks were labeled with planes (220), (311), (222), (400), (422), and (511), and well-matched with the standard JCPDS Card Nos. 00-086-2267 and 00-08-0234, also confirmed the formation of cubic spinel Ce3+ doped CF ferrites. [[20](#_ENREF_20)]. The additional peaks of CeO2 [[21](#_ENREF_21), [22](#_ENREF_22)] well-matched with JCPDS card No. 81–0792 represented by a symbol (\*), as shown in Fig 1(a). Secondary phases of cerium oxide CeO2 appeared when cerium concentration was enhanced from x=0 and intensity of peak continues to increase with the rise of cerium concentration which may be due to (a) less solubility of cerium contrary to that of iron and (b) greater bond energy of Ce3+­­O2 as compared to Fe3+­­O2-. Hence the immense amount of energy is needed to incorporate Ce3+ ions for Fe3+ ions and this creates secondary phases [[23](#_ENREF_23), [24](#_ENREF_24)]. It was also observed from Fig 1(b) that the peak with the plane (311) shifts toward a greater angle and the intensity of peaks decreases as increasing Ce3+ cations, which may be due to oxygen vacancies and strain production [[21](#_ENREF_21)].



**Fig. 1(a)** XRD patterns for Ce3+ doped CF samples **(b)** Peaks (311) shift towards right

The prominent peaks (220), (311), and (511) were used to determine the crystallite size (*D*) of the as-prepared ferrites using Scherrer’s relation [[25](#_ENREF_25)];

(1)

where 0.9 is Scherrer constant. , , and represent full width at half maximum height of diffraction peak, the wavelength of the X-ray used and Bragg’s angle, respectively. The crystallite size (*D*) was increased with Ce3+ doping for the (220), (311), and (511) peaks (Table 1) and graphically represented in Fig 2(a). The lattice constant (*a*), unit cell volume and X-ray density were determined using relation (2), (3), and (4), respectively [[26](#_ENREF_26), [27](#_ENREF_27)] and reported in Table 1.

(2)

where indicates the wavelength of the X-ray beam used and ‘’ stands for Bragg’s angle. (hkl) are the corresponding miller indices to each peak in the pattern [[28](#_ENREF_28), [29](#_ENREF_29)].

(3)

(4)

where Z = 8 indicates the number of formula units present in a unit cell for spinel ferrite. Mw represents the molecular weight of synthesized nano ferrites and is Avogadro’s number. The graphical representation of Ce3+ concentration (*x*) *versus* lattice constant (*a*), unit cell volume and X-ray density for the (220), (311), and (511) peaks are depicted in Fig 2(b-d). The change in the lattice parameters may be due to the insertion of a large ionic radius of Ce3+ (1.143 Å) as compared to Fe3+ (0.645 Å) [[30](#_ENREF_30)].

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| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Table 1.** Crystallite size, lattice constant, unit cell volume, and X-ray density for (220), (311) and (511) plans of the as-prepared samples | | | | | | | | | | | | | | |
| Samples | Miller indices (hkl) | | | | | | | | | | | | | |
| (220) | (311) | (511) | (220) | (311) | (511) | (220) | (311) | (511) | | (220) | (311) | (511) |
| Crystallite size  *D* (nm) | | | Lattice parameter  *a* () | | | Unit cell volume  *V*cell () | | | | X-ray density  (g/cm3) | | | |
|  | 27 | 28 | 24 | 8.7336 | 8.7293 | 8.7254 | 665.98 | 664.43 | | 665.56 | 5.75 | 5.76 | 5.75 |
|  | 29 | 32 | 32 | 8.7156 | 8.7041 | 8.7016 | 664.60 | 662.00 | | 661.44 | 5.97 | 5.99 | 5.99 |
|  | 33 | 33 | 34 | 8.7048 | 8.6891 | 8.6956 | 657.80 | 658.54 | | 660.08 | 6.24 | 6.24 | 6.22 |
|  | 36 | 35 | 37 | 8.6948 | 8.6841 | 8.6719 | 657.80 | 657.43 | | 654.70 | 6.46 | 6.46 | 6.49 |
|  | 48 | 46 | 47 | 8.6909 | 8.6891 | 8.6926 | 657.80 | 658.57 | | 659.41 | 6.67 | 6.67 | 6.65 |



**Fig. 2** Plots of Ce concentration (x) *versus* **(a)** crystallite size **(b)** lattice constant **(c)** unit cell volume **(d)** X-ray density for (220), (311), and (511) planes

Cd2+ is a divalent metal ion whereas Ce3+ has a trivalent metal ion, in which Cd2+ occupies the tetrahedral site and both Ce+3 and Fe+3 occupy the octahedral site. Hence cation distribution for Ce3+ doped CF samples are reported in Table 2. The radii at the tetrahedral (*A*) and octahedral (*B*) sites were calculated using the following relation;

(5)

(6)

The ionic radii: = 0.78Ao, = 1.143Ao, = 0.645Ao [[31](#_ENREF_31)]. *C*Cd, *C*Fe, *C*Ce represent the concentration of Cd2+, Ce3+and Fe3+ ions on different sites. The result is summarized in Table 2. The theoretical lattice constant () was calculated by equation (7) and is given in Table 2.

= 8/3[(+) +(rB + )] (7)

where 1.32Å represents ionic radii of Oxygen. The comparison of the experimental and theoretical lattices constant represented in Fig 3(a). The theoretical value of the lattice constant () was found to increase with the addition of cerium as shown in Fig 3(a). This increasing trend could be associated with the increasing cerium content where the ionic radius of Ce3+ (1.143 Å) is larger than that of Fe3+ ion (0.645 Å) replacing iron ions on octahedral *B*-site which causes asymmetry in the structure. Hence, the lattice constant should be increased with the increasing content of the Ce3+ during the substitution process [[23](#_ENREF_23)]. Besides this, a decreasing trend was observed in the experimental value of the lattice parameter with the inclusion of dopant ions which could be attributed to the compression of spinel lattice induced by the secondary phases. The secondary phases are formed on the surface of grains during the calcination process. The deformity in the internal grain region due to the induced secondary phases corresponds to the contraction of spinel lattice and as a result, the lattice parameter diminished. The doping ions, calcination temperature, and time are responsible for the contraction of the spinel lattice. Sintering conditions are very important for the diffusion of ions into the spinel lattice [[23](#_ENREF_23), [32](#_ENREF_32), [33](#_ENREF_33)]. The oxygen ion parameter (*u*) depends upon chemical composition, preparation condition, and sintering procedure and can be calculated by using equation (8);

*u* = [(*r*A+*r*O)1/*a*+1/4] (8)

The oxygen ion parameter and lattice parameter increase by increasing Ce doping and are listed in Table 2.The hoping length of *A* site, as well as B site, were determined by relation:

*L*A = *a* (( (9)

*L*B = *a* (32 - ) (10)

where the smallest space of *A*-site cation and *B*-site cation from oxygen ion is represented by *L*A and *L*B respectively. = (u – 0.375) is deviance from the ideal “*u*” parameter. The hopping length variation *versus* cerium concentration is represented in Fig 3(b). The bond lengths at *A*- and *B*- sites (,), shared tetrahedral edge length (), shared and unshared octahedral edge lengths (,) were calculated and are given in Table 2.

|  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Table 2**. The theoretical value of the lattice parameter (), radii of octahedral and tetrahedral (and ), Oxygen ion parameter (*u*), Hoping lengths (, ), Bond lengths at A- and B sites (,), shared tetrahedral edge length (), shared and unshared octahedral edge lengths (,). | | | | | | | | | | | |
| **Cation distribution** | **(Å)** | **(Å)** | **(Å)** | ***u***  **(Å)** | **(Å)** | **(Å)** | **(Å)** | **(Å)** | **(Å)** | **(Å)** | **(Å)** |
| (Cd)[Fe2] O4 | 8.47 | 0.78 | 0.64 | 0.3888 | 3.7799 | 3.0862 | 2.1 | 2.0681 | 3.429 | 2.7432 | 3.0957 |
| (Cd)[Ce0.125Fe1.875] O4 | 8.55 | 0.78 | 0.67 | 0.3892 | 3.7689 | 3.0773 | 2.1 | 2.0591 | 3.429 | 2.7254 | 3.0874 |
| (Cd)[Ce0.25Fe1.75] O4 | 8.63 | 0.78 | 0.7 | 0.3895 | 3.7624 | 3.072 | 2.1 | 2.0537 | 3.429 | 2.7148 | 3.0824 |
| (Cd)[Ce0.375Fe1.625] O4 | 8.72 | 0.78 | 0.73 | 0.3896 | 3.7603 | 3.0702 | 2.1 | 2.0519 | 3.429 | 2.7113 | 3.0807 |
| (Cd)[Ce0.5Fe1.5] O4 | 8.80 | 0.78 | 0.76 | 0.3895 | 3.7624 | 3.0720 | 2.1 | 2.0537 | 3.429 | 2.7148 | 3.0824 |



**Fig. 3** Plots of **(a)** Ceconcentration (x) *versus* experimental and theoretical lattice constants **(b)** Ceconcentration (x) *versus* hopping length at A- and B- sites

**3.2 FTIR analysis**

Functional group analysis and absorption bands of Ce3+ doped CF samples were found by performing FTIR analysis. Two main absorption bands are observed in the range of 1000 to 400 cm-1 as clear from Fig 4. The absorption band around ~534 cm-1 corresponds to the tetrahedral vibrations and ~416 cm-1 corresponds to the octahedral vibrations. These bands may be due to stretching vibrations of metal and oxygen ions at tetrahedral and octahedral sites. From the FTIR spectra band is shifted to the lower wavelength which may be attributed to the increase of Ce3+ ions in the ferrite. The Cd2+ ions preferably occupy the tetrahedral (*A*) site in the spinel ferrites [[31](#_ENREF_31)]. The absorption band around ~3532 cm-1 corresponds to the O-H peak and indicates the presence of adsorbed water [[34](#_ENREF_34)]. The peaks around ~2062 cm-1 is attributed to the stretching. Bands around 1443 cm-1 and 859.11 cm-1 correspond to the C-N ****stretching. The absorption band around 1206 cm-1 corresponds to the C-F stretching.

**Fig 4:** FTIR spectra of the Ce3+ doped CF samples

**3.3 I-V measurement analysis**

The electrical resistivity of Ce3+ doped CF samples sintered at 900 were computed employing two probes approach in a temperature span of 40 – 450. Resistances of manufactured samples were evaluated using the slope of the I-V graph, where the voltage was along the x-axis and current was plotted along the y-axis. The formula is as follows;

(11)

The electrical resistivity of manufactured samples was determined using the mathematical expression;

(12)

where “*h*” represents the thickness of pallets. “*A*” gives the area of cross-section of pellets prepared from all the samples.

Fig 5 reveals the way electrical resistivity changes with the enhancement of temperature of all the samples of cerium doped cadmium soft ferrites. Values of resistivity found for different concentrations of cerium doped cadmium ferrites at certain temperatures 313 K, 423 K, 523 K, 673 K, 723 K are given in Table 3. Table 3 reveals that the resistivity of pure CdFe2O4 at 313 K (room temperature) is very high (63.21 ×108 Ω cm) and its value decreases with the increasing temperature up to 623 K (63.21 ×108 Ω cm – 0.0054 ×108 Ω cm) and increases when the temperature is further increased from 623K (0.011 ×108 Ω cm). The same trend is seen for all the samples as is clear from Fig 5. Change in behavior of resistivity indicates that the nature of magnetic materials changes from one form to another [[35](#_ENREF_35)]. Region-I has the ferromagnetic nature of the samples as Fig 5 declares a decrease in resistivity with the rise of temperature and region-II indicates the paramagnetic nature of the materials. To evaluate the activation energy of the samples Arrhenius plots are drawn (Fig 6). The conduction mechanism in ferrites is the hopping mechanism. According to the hopping mechanism, when the temperature of ferrites increases, mobility of charge carriers also increases, and a current flow by hopping from one iron atom to the other. So, ferrite conduction is imputed to the mechanism of hopping of electrons between ferrous (Fe2+) and ferric (Fe3+) at octahedral sites. The resistivity of samples with composition has random behavior as is clear from Fig 7, but the resistivity at x = 0.125 has a minimum value and at x = 0.375 has a maximum value at almost all temperatures as shown in Table 3. Fig 7 exhibits that the resistivity increased for all the samples except for the samples with concentrations x = 0.125 and x = 0.5. This increase in resistivity may be due to the insertion of cerium (75 µΩ cm) at the place of iron (9.71 µΩ cm) [[36](#_ENREF_36)]. The decrease in the value of resistivity at x = 0.5 may be attributed to the secondary phase (CeO2) that reside at the grain boundaries and hinder the further migration of iron (Fe3+) from octahedral site to tetrahedral site, so hopping of electrons between Fe3+ and Fe2+ increases resulting in a decrease in resistivity [[37](#_ENREF_37)]. Fig 6 shows graphs of ln *vs*. 1000/T for Ce3+ doped CF samples. Activation energies (*E*a) were determined employing formula [[35](#_ENREF_35), [36](#_ENREF_36)].

(13)

where the value of Boltzmann constant . Table 4 shows that the activation energy of fabricated nano-ferrites increased with an increase of dopant concentration and also as cleared from Fig 8, Same results reported in papers [[36](#_ENREF_36)].

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **Table 3:** Electrical resistivity of at different temperatures | | | | | |
| **Composition** | **Resistivity × (Ω-cm)** | | | | |
| **313K** | **423K** | **523K** | **623K** | **723K** |
| CdFe2O4  CdFe1.875Ce0.125O4  CdFe1.75Ce0.25O4  CdFe1.625Ce0.375O4  CdFe1.5Ce0.5O4 | 63.21  42.00  59.068  121.49  44.05 | 1.40  0.42  0.77  9.40  0.95 | 0.074  0.013  0.032  0.213  0.035 | 0.0054  0.0013  0.0013  0.0219  0.0014 | 0.011  0.0042  0.0053  0.1030  0.0085 |



**Fig 5:** Plot of temperature *versus* variation of ln of



**Fig 6:** Plot of 1000/T *versus* ln



**Fig 7:** Plot of Ce concentration *versus* resistivity



**Fig 8:** Plot of activation energy as a function of composition

The drift mobility () for fabricated samples was evaluated using equation [[38](#_ENREF_38), [39](#_ENREF_39)];

(14)

where ‘*e*’ represents the charge on the electron, “” shows DC electrical resistivity and “”is charge carriers concentration. The following relation was employed for the evaluation of is [[40](#_ENREF_40), [41](#_ENREF_41)];

(15)

where “*M*” represents molecular weight. ‘*N*a’ shows Avogadro's number. Sintered density is represented by ‘’ and the number of iron atoms in the chemical formula of ferrite is represented by ‘’. Fig 9 helps to comprehend the relationship between drift mobility and temperature. From the plot (Fig 9), drift mobility rises with the enhancement of temperature till 623K and after that drift mobility starts to reduce with a rise in temperature and resistivity increases correspondingly. This shows that the change in mobility with temperature is responsible for the disparity of resistivity with the temperature rise. Hence, with the enhancement of temperature charge carriers begin to hop from one site to another increasing the drift mobility and decreasing the resistivity of Nano ferrites. The values of carrier concentration and drift mobility are given in Table 4. It was seen from the Fig 10 that drift mobility increased from 5.68 to 27.74 at 673 K for the concentration of cerium from x = 0 to x = 0.50 except x = 0.375.

**Fig 9:** Plot of drift mobility of (x = 0, 0.125, 0.25, 0.375 and 0.50) with temperature.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **Table 4:** Drift mobility, carrier concentration and activation energy at 673K | | | | | |
| **Parameters** | **x = 0.0** | **x = 0.125** | **x = 0.25** | **x = 0.375** | **x = 0.5** |
| **at 673K** | 5.68  1.258  1.2356  -0.0642  1.8784 | 6.21  1.149  1.5109  -1.0211  2.5320 | 12.16  1.046  1.3903  -1.2986  2.6943 | 6.70  0.934  1.3403  -1.4164  2.7568 | 27.74  0.821  1.3647  -1.6069  2.9716 |



**Fig 10:** Plot of Cerium concentration vs drift mobility of (x=0, 0.125, 0.25, 0.375 and 0.50) at 673K

**3.4 Dielectric properties**

The graph of dielectric constant versus frequency for all the samples at ambient temperature is shown in Fig.11 which shows a reducing trend of dielectric constant with the rise in frequency. This exhibits that cerium doped cadmium ferrites depict normal dielectric behavior. Many researchers found the same dielectric behavior in many ferrites [[36](#_ENREF_36), [40](#_ENREF_40)]. When the frequency of applied field increases then the number of electrons that accumulate at grain boundary decrease and hence interchange of electrons also decreases as a result polarization decrease and dielectric constant decreases. Fig.12 shows a graph of dielectric loss tangent vs frequency at ambient temperature for all the samples. Dielectric loss reduces with the enhancement of frequency. Fig.13 shows the graph between impedance vs frequency. The impedance has an inverse relation with frequency. The decreasing trend of dielectric constant with an increase in frequency can be interpreted on account of the Maxwell-Wagner model [[42](#_ENREF_42), [43](#_ENREF_43)]. This model explains that grain boundary having greater resistance separates the conducting grains in every ferrite structure. At low frequency, the values of the dielectric constant are very large due to the presence of polarization which may be due to the accumulation of a large number of electrons on the grain boundary in nanomaterial ferrites. The polarization in ferrites is due to a mechanism same as that of the conduction process. The polarization occurs due to the local displacement of electrons in the direction of the applied field which may be attributed to the interchange of electrons between and ions on the octahedral site [[44](#_ENREF_44)].

**Fig 11**: Plot of the dielectric constant of (x = 0, 0.125, 0.25, 0.375 and 0.50) vs frequency

**Fig 12:** Plot of dielectric loss tangent of (x = 0, 0.125, 0.25, 0.375 and 0.50) vs frequency.

**Fig 13:** Plot of the impedance of (x = 0, 0.125, 0.25, 0.375 and 0.50) vs frequency.

**Conclusion**

The co-precipitation method was employed for the fabrication of nano ferrite powders. X-ray diffraction study shows cubic spinel ferrites with traces of secondary phases of CeO2 for all samples with cerium composition. The crystallite size of the synthesized material fluctuates from 28 nm to 46 nm. FTIR study confirmed metal oxide bands at (531.23 cm-1-534.84 cm-1) corresponding to the tetrahedral bond vibrations, verifying the formation of the cubic spinel structure of . The resistivity of synthesized material was found to reduce with the enhancement of temperature, declaring semiconducting behavior of synthesized Ce-Cd samples. The activation energy increased with an increase in concentration for samples.

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