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The Characterization of a Newly Layered Bimetallic Hydrogen Selenite Copper-Selenium: Synthesis and Structure

Mohamed Loukil

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Abstract

Single-crystal X-ray diffraction data were used to solve the structure of a newly layered copper-selenium hydrogen selenite and further refined to a final reliability factor, $R_1 = 0.038$. This structure was found to have an orthorhombic space group $PBn2_1$, with $a = 7.1753(4) \text{ \AA}$, $b = 9.0743(4) \text{ \AA}$, $c = 17.725(9) \text{ \AA}$, $V = 1154.06(10) \text{ \AA}^3$, and $Z = 4$. Although this structure may be described to exhibit a bidimensional structure, it is actually three-dimensional in shape. The bidimensional structure is made up of layers, parallel to the (010) plane, which contain copper atoms and $(\text{HSeO}_3)^-$ anions with sheets interconnected by $[\text{CuCl}_3(\text{H}_2\text{O})_3]$ groups. Bond valence sum calculations were used to evaluate the Se and Cu oxidation states. Both the infrared (IR) and Raman spectra were obtained and employed to confirm the presence of hydrogen selenites ($\text{Se}-\text{O}-\text{H}$). Also, the dielectric constant at different frequencies and temperatures revealed a phase transition at 383 K.

Keywords: infrared, structural study, dielectric properties, bimetallic hydrogen selenites

1. Introduction

During the past years, lot of interest has been shown in hydrogen selenite chemistry motivating research focused on expanding the knowledge of the structural and bonding principles of this ligand. There is an important number of divalent metal hydrogen selenite crystal structures reported in the literature. For example, $\text{M}(\text{HSeO}_3)_2$ (where M: Cu, Mg, Sr, Ba) [1, 2]; $\text{M}(\text{HSeO}_3)_2 \cdot \text{H}_2\text{O}$ (where M: Ca, Cu) [3–6]; $\text{M}(\text{HSeO}_3)_2 \cdot \text{NH}_4\text{Cl}$ (where M: Cu) [7]; $[\text{Cu}(\text{HSeO}_3)_2\text{Cu}_x\text{M}_{1-x}\text{Cl}_2(\text{H}_2\text{O})_4]$ with M = Cu, Co, Mn, Ni, and Zn [8]. This family has a

three-dimensional structure, but it may be considered as being derived from the $[\text{Cu}(\text{HSeO}_3)_2]$ -type structure.

Compounds exhibiting mixed valences (like Se^{4+} and Cu^{2+}) are at the center of many studies owing to their potential applications in relation to the electronic exchange. For almost all of these compounds, except $[\text{Cu}(\text{HSeO}_3)_2]$, magnetic measurements have revealed the occurrence of weak ferromagnetism at low temperature ($T \sim 10\text{--}20\text{ K}$) for which a tentative explanation is offered for this peculiar property in agreement with other authors [8, 9]. In this study, the crystal structure of the compound $[\text{Cu}_{0.335}\text{Se}_{0.582}(\text{HSeO}_3)_2\text{CuCl}_3(\text{H}_2\text{O})_3]$, herein presented, was obtained using an X-ray single structure and various spectroscopic (IR and Raman) characterization, as well as dielectric measurements.

2. Materials and methods

The experiments were carried out using a single crystal of $[\text{Cu}_{0.335}\text{Se}_{0.582}(\text{HSeO}_3)_2\text{CuCl}_3(\text{H}_2\text{O})_3]$ grown by slow evaporation from a mixture of hydrochloric acid containing stoichiometric $\text{CuCl}_2\text{--SeO}_2$ at room temperature in the ratio 1/2. Blue thin rectangular parallelepiped crystals were grown after vaporizing in air for 15 days approximately. The determination of $[\text{Cu}_{0.335}\text{Se}_{0.582}(\text{HSeO}_3)_2\text{CuCl}_3(\text{H}_2\text{O})_3]$ formula was achieved by the crystal structure refinement approach at room temperature.

For electrical impedance measurements (in the range 1–10 KHz), a Hewlett-Packard 4192 ALF automatic bridge monitored by a HP Vectra microcomputer was used. Prepared dense translucent pellets with approximately a diameter of 8 mm and a thickness of 1–1.2 mm, covered with graphite electrodes were utilized for the measurements.

The measurement of electrical impedances were equally carried out in the range, 1–10 KHz, using a Hewlett-Packard 4192 ALF automatic bridge monitored by a HP Vectra microcomputer. For these types of experiments, dense translucent pellet samples were prepared, with a diameter of 8 mm and thickness between 1 and 1.2 mm. All the pellets were then covered with graphite electrodes prior to measurements.

A modern nondispersive Fourier Transform (FT-IR) spectrometer (Perkin-Elmer 1750 spectrophotometer IR-470) was employed for the characterization of the crystalline powders after mixing with KBr, with very notable IR-active functional groups found in the samples investigated. Scans of IR spectra were recorded in the range $400\text{--}4000\text{ cm}^{-1}$ without apodization. To record Raman spectra of the solid samples for the study of the various phases, a conventional scanning Raman instrument (Horiba Jobin Yvan HR800 microcomputer system), equipped with a Spex 1403 double monochromator (with a pair of 600 grooves/mm gratings) and a Hamamatsu 928 photomultiplier detector was used. Solid materials were sampled at different temperatures for this analysis. During the recording of prominent Raman peaks, excitation radiation from the instrument was fulfilled by a coherent radiation emitted by a He-Neon laser operating at a wavelength of 633 nm, with an output laser power of 50 mW. In order to acquire high-resolution Raman spectra, the spectral resolution of the slit width varied from 3 to 1 cm^{-1} .

To study the crystal structure of $[\text{Cu}_{0.335}\text{Se}_{0.582}(\text{HSeO}_3)_2\text{CuCl}_3(\text{H}_2\text{O})_3]$, an APEX II diffractometer (powder XRD) fitted with graphite-crystal monochromated Mo $K\alpha$ radiation (0.71073 Å) was employed. In this study, a total of 3093 reflections were collected, among which only 2803 reflections, namely those for which $I > 2\sigma(I)$, were actually used in the determination and refinement of the structure. Corrections were made for Lorentz-Polarization and absorption effects. **Table 1** presents the data collection procedure and structure refinement at room temperature.

A three-dimensional Patterson synthesis approach was used to determine the selenium atoms positions in the compound. On the one hand, the Fourier function allowed for the localization of the chlorine (Cl), copper (Cu), and oxygen (O) atoms. On the other hand, the hydrogen atoms were localized from a difference Fourier synthesis and introduced as fixed contributors. Conversely, all the non-hydrogen atoms were typically assigned anisotropic thermal displacements. The structure solution and refinement were carried out using SHELX programs [10, 11]. The bond lengths and angles are given in **Table 2**.

Crystallographic data	T = 296(2) K
Formula	$[\text{Cu}_{0.335}\text{Se}_{0.582}(\text{HSeO}_3)_2\text{CuCl}_3(\text{H}_2\text{O})_3]$
Formula weight	547.15
Space group	Pbn2 ₁
a (Å)	7.1753(4)
b (Å)	9.0743(4)
c (Å)	17.7246(9)
V (Å ³)	1154.06(10)
Z	4
ρ_{calc} (g/cm ³)	3.149
μ (mm ⁻¹)	11.359
Crystal size (mm ³)	0.05 × 0.04 × 0.03
Crystal shape	Octahedral
F(000)	1030
Data collection instrument	Kappa-APEX II
Radiation, graphite	$\lambda\text{Mo } K\alpha$ (0.71073 Å)
θ range for data collection (°)	6.80–30.57
Index ranges	$-10 \leq h \leq 9$; $-12 \leq k \leq 12$; $-23 \leq l \leq 25$
Total reflections	8605
Reflection with ($F > 4\sigma(F)$)	3093
R(F) (%)	3.38
W _{R2} (%)	1.74

Table 1. Crystal structure data and experimental conditions for the structure determination of $[\text{Cu}_{0.335}\text{Se}_{0.582}(\text{HSeO}_3)_2\text{CuCl}_3(\text{H}_2\text{O})_3]$.

a: SeO₃ polyhedron

Se1—O4 = 1.70(5)
 Se1—O5 = 1.70(5)
 Se1—O7 = 1.71(5)
 Se3—O3 = 1.67(4)
 Se3—O6 = 1.66(6)
 Se3—O8 = 1.77(5)

O5—Se1—O7 = 102(2)
 O5—Se1—O4 = 102(2)
 O7—Se1—O4 = 98(2)
 O3—Se3—O6 = 98(3)
 O3—Se3—O8 = 101(3)
 O6—Se3—O8 = 100(2)

b: Cu(Se)O₄Cl₂

Cu2/Se2—O3 = 1.98(4)
 Cu2(c)/Se2(c)—O4(b) = 1.91(4)
 Cu2/Se2—O5 = 1.94(4)
 Cu2(d)/Se2(d)—O6(a) = 2.01(5)
 Cu2/Se2—Cl2 = 2.77(2)
 Cu2/Se2—Cl3(e) = 2.80(2)

O4(b)—Cu2/Se2—O6(a) = 91(2)
 O4(b)—Cu2/Se2—O3 = 179(3)
 O6(a)—Cu2/Se2—O3 = 90(2)
 O4(b)—Cu2/Se2—O5 = 89(2)
 O6(a)—Cu2/Se2—O5 = 180(4)
 O3—Cu2/Se2—O5 = 90.1(2)
 Cl2—Cu2/Se2—O6(a) = 89.52(8)
 O3—Cu2/Se2—Cl3(e) = 89.11(5)
 Cl2—Cu2/Se2—O3 = 91.1(4)
 Cl2—Cu2/Se2—O4(b) = 89.83(3)
 O5—Cu2/Se2—Cl2 = 94.18(7)

c: CuCl₃(H₂O) octahedron

Cu1—Cl1 = 2.66 (8)
 Cu1—Cl2 = 2.30 (2)
 Cu1—Cl3 = 2.30(2)
 Cu1—O1 = 1.99(7)
 Cu1—O2 = 2.02(6)
 Cu1—O9 = 2.21(3)

O1—Cu1—O2 = 88.1(3)
 O1—Cu1—Cl3 = 176(2)
 O1—Cu1—Cl2 = 90.9(2)
 O2—Cu1—Cl2 = 176.1(5)
 O2—Cu1—Cl1 = 83(2)
 Cl3—Cu1—Cl2 = 92.2(3)
 Cl3—Cu1—Cl1 = 94.1(3)

Symmetry code: a: $-x + 1/2, y + 1/2, z$; b: $-x + 3/2, y + 1/2, z$; c: $-x + 3/2, y - 1/2, z$; d: $-x + 1/2, y - 1/2, z$; e: $-x + 1, -y + 2, z + 1/2$.

Table 2. Interatomic distances for [Cu_{0.335}Se_{0.582}(HSeO₃)₂CuCl₃(H₂O)₃] samples (this study).

3. Results and discussion

3.1. Structure description

From the charge balance in [Cu_{0.335}Se_{0.582}(HSeO₃)₂CuCl₃(H₂O)₃], it can be suggested that the average oxidation state of Cu(2)/Se(2) is equal to 3, which would fit to 33.5% of Cu²⁺ and 58.2% of Se⁴⁺. This outcome was confirmed by performing a calculation of bond valence sums around the centers of the cation sites. The steps and expressions used in the calculation of the bond valence are published in [12]. More specifically, the bond valence (S_{ij}) is expressed as $S_{ij} = \exp[(R_0 - R_{ij})/B]$, where R_0 and B are the experimentally determined parameters and R_{ij} is the bond length of the cation-anion pair [12]. The sum of the bond valence ($\sum_j S_{ij}$) around an ion calculated must be equal to the formal valence (V_i) of this ion, based on the valence sum rule.

In this work, our calculation shows that, for the pyramidal sites (Se1 and Se3), the sum of bond valence is ~4, which is equal to selenium formal valence. Thus, the bond valence sums around

the octahedral site of Cu(2) are typically consistent with the value +2.7, confirming the presence of selenium Se^{4+} and cuprite Cu^{2+} in the same site. It is also observed that the blue single crystal $[\text{Cu}_{0.335}\text{Se}_{0.582}(\text{HSeO}_3)_2\text{CuCl}_3(\text{H}_2\text{O})_3]$ crystallizes in the orthorhombic system, space group $\text{Pbn}2_1$. Structurally, the crystal structure of $[\text{Cu}_{0.332}\text{Se}_{0.582}(\text{HSeO}_3)_2\text{CuCl}_3(\text{H}_2\text{O})_3]$ represents a new type of structure for complexes of hydrogen selenites (**Figure 1**). The building blocks $[\text{Cu}_{0.335}\text{Se}_{0.582}(\text{HSeO}_3)_2]$ and $[\text{CuCl}_3(\text{H}_2\text{O})_3]$, hereunder drawn, are arranged to form layers in the structure parallel to the (001) plane between which the lone pairs E are located (**Figure 2**). Due to the stereochemical activity of the lone pairs E, Se has very asymmetric coordination polyhedral SeO_3 pyramids.

Spatially, the high anisotropic distribution of anions observed around each cation is characteristically of a strong stereochemical activity of their electron lone pair E for the Se1 and Se3 atoms. The consequence for the coordination polyhedral is the description of a distorted SeO_3 triangular pyramid, in which the Se—O(7) and Se—O(8) are marginally longer than the other Se—O bonds (**Table 2**). Thereof, the lone pair E so directed to constitute the fourth vertex of an SeO_3E tetrahedron (**Figure 3**).

The O—Se—O with angles of values 98(2) and 102(2) formed from Se—O chemical bonds are situated on one side of the Se atom, whereas the other side is hitherto a “dead” zone around

Figure 1. Perspective view of the $[\text{Cu}_{0.335}\text{Se}_{0.582}(\text{HSeO}_3)_2\text{CuCl}_3(\text{H}_2\text{O})_3]$ unit cell content.

Figure 2. A projected along a-axis view of the $[\text{Cu}_{0.335}\text{Se}_{0.582}(\text{HSeO}_3)_2\text{CuCl}_3(\text{H}_2\text{O})_3]$ unit cell content.

Figure 3. Environment of selenium Se(1), Se(3) cations.

the lone pair E of the Se atom. As such, the SeO_3 polyhedral has strong dipole moments, due to such a sharp asymmetry of the atomic arrangement of the first coordination spheres, which provide considerable dipole-dipole contribution to the inter-anion potentials. Taking a closer look at the structure in **Figure 4**, one can easily see that each Cu(1) atom is surrounded by three oxygen (O) atoms and three chlorine (Cl) atoms. This basically forms a slightly distorted octahedron with distances ranging from 1.99(7) to 2.21(3) Å for Cu—O and 2.30 (2) Å for Cu—Cl.

A remarkable deviation from full occupancy was exhibited in the occupancy of the Cu(2) site during refinement. This is an indication of a substitution with Se, resulting in final occupancies constrained in sum to 1.0, and refined to 0.335(4) and 0.582(2), respectively. It should be noted that the deviation from 1.0 is due to the mixed valence between Cu and Se, for Cu(2) and Se(2), respectively. As shown in **Figure 5**, the Cu(2)/Se(2) atoms are surrounded by four oxygen atoms and two chlorine atoms to form an irregular octahedron.

From the earlier arguments, the structure depicted in **Figure 2** can be ascribed to being formed by Cu(1), Se(2)/Cu(2) polyhedral that structurally shares chlorine (Cl) corners in infinite chains along the direction [001]. Therein, the sequential metal atoms in the chain trend schematically following Cu(1)—Se(2)/Cu(2)—Cu(1)'—Se(2)/Cu(2)—Cu(1), whereby the prime refers to the polyhedral generated by the symmetry operation: $1 - x, 2 - y, -0.5 + z$, in the [010] direction. As a result, the three-dimensional network is thus formed. It can be argued that the lattice cohesion may be strengthened by the hydrogen bonds within the layer (O(8)—H(7)—O(4)) and (O(7)—H(8)—O(3)) or outside the layer.

Figure 4. Structural environment of copper cations Cu(1).

Figure 5. Environment of copper/selenium cations Cu(2)/Se(2).

3.2. Spectroscopic studies

In order to confirm the crystallographic results of the following compound: [Cu_{0.335}Se_{0.582}(HSeO₃)₂CuCl₃(H₂O)₃], IR, and Raman spectroscopy were used. **Figure 6** shows that the IR spectrum is restricted to the mid-infrared frequency range: 400–4000 cm⁻¹.

Figure 6. Infrared spectrum of $[\text{Cu}_{0.332}\text{Se}_{0.582}(\text{HSeO}_3)_2\text{CuCl}_3(\text{H}_2\text{O})_3]$ at room temperature.

In this chapter, the band corresponding to the symmetric stretching vibrations of SeO_2 groups was observed at around 825 cm^{-1} in the Raman spectrum (**Figure 7**). Similarly, a strong intense broad band is observed in the infrared (IR) spectrum for this mode. These findings are in agreement with those reported by Cody and al. and Micka et al. for vibrational analysis on a series of alkali hydrogen selenites. From the work of these authors, the symmetric stretching vibrations are around 850 cm^{-1} [13–15].

From **Figure 7**, a band of very weak intensity is observed at 710 cm^{-1} , accompanied by a shoulder at 738 cm^{-1} , which is ascribed to asymmetric stretching vibrations of SeO_2 groups. In the $686\text{--}740\text{ cm}^{-1}$ region, a corresponding IR spectrum with an intense (broad) frequency absorption is present. In the literature [16, 17], these modes have been observed at a very much lower wave numbers than those in alkali hydrogen selenites.

Another observation is that copper (selenium) atoms are located at the center of CuO_4Cl_2 coordination octahedra. The axial Cu—Cl bonds are longer than the others, and they are coordinated to water molecules. It can be stated that the high spin d-configuration of Cu leads to Jahn-Teller distortions by Jahn and Teller [18] hitherto producing planar Cu—O bonds in the $1.91\text{--}1.98\text{ Å}$ range and an axial Cu—Cl distance of $2.77\text{--}2.80\text{ Å}$. It can be ascertained that the planar oxygen (O) atoms are shared by Cu and Se atoms which can lead to the observed reduction in the stretching frequencies of the SeO_2 groups.

Typically, the stretching vibrations of the HSeO_3^- ion (vSe—OH), which often characteristically appear in the $600\text{--}650\text{ cm}^{-1}$ region [16, 17], are also appearing at lower wave numbers ($\sim 627\text{ cm}^{-1}$). In the IR spectrum, this mode is observed as an intense broad band at 532 cm^{-1} .

Figure 7. Raman spectrum of the compound at room temperature.

The O—O distance involving the Se—OH system with one of the equatorial oxygen atoms of the neighboring $\text{Cu(2)/Se(2)O}_4\text{Cl}_2$ group is 2.662 Å. The observed lowering of the Se—O(H) vibrations from the free-state values is a confirmation of the corresponding strong hydrogen bonds determined in the X-ray diffraction data. As presented in Table 3, it is seen that symmetric deformation vibrations of the HSeO_3^- ion have given only weak bands of the Raman spectrum, while a medium intense broad band is obtained in the IR. In the corresponding asymmetric bending vibration, Raman spectrum shows medium intense bands with a weak band in the IR. A reduction in the symmetry of the HSeO_3 ion may be causing the changes in the activity of these modes. From the observed strong hydrogen bonding and the distortion of $\text{Cu(Se)O}_4\text{Cl}_2$ octahedra, we can deduce that the Jahn-Teller distortion affects the HSeO_3 vibrations.

Fundamentally, the hydrogen-bonded OH groups may lead to three vibrations, namely: $\nu(\text{OH})$ stretching, the in-plane (OH), and the out-of-plane (OH) deformation vibrations. In fact, the stretching bands of strongly H-bonded systems are intense and usually built up of a number of unresolved components owing to strong interaction between the proton vibration and the $\nu(\text{O}\cdots\text{O})$ vibrations [19, 20]. It is also clearly elucidated from the literature [21, 22] that the broad $\nu(\text{OH})$ band in Fermi resonance with the overtones of the $\nu(\text{OH})$ modes splits into three bands A, B, and C. The A mode is typically observed as a strong broad band at 2850 cm^{-1} in the IR spectrum, while the B mode is obtained as a medium intense band at 2360 cm^{-1} . A medium intense broad band at 1900 cm^{-1} and a weak one at 1730 cm^{-1} are generally being assigned to C bands. From a practical viewpoint, the appearance of these bands confirms the existence of strong hydrogen bonds in the crystal. The in-plane bending $\nu(\text{OH})$ vibrations are

Raman	IR	Assignments
	3553 mbr 3170 wbr	$\nu_1\text{H}_2\text{O}$
	2920 w 2850 w 2694 wbr	$\nu\text{OH(A)}$
	2360 mbr	$\nu\text{OH(B)}$
	1900 vwbr 1730 vw	$\nu\text{OH(C)}$
	1606 mbr	$\nu_2\text{H}_2\text{O}$
	1222 m	δOH
	1043–920	γOH
	896 m	
825 vs	825 s	νSeO_2
738 wsh 710wbr 627 m	740 s 686 s	
515 m 501 sh	532 s	$\nu\text{Se—OH}$
425 vw	442 w	δSeO_2
380 vw		$\delta\text{asO—Se—OH}$
350 sh 337 m		$\delta\text{sO—Se—OH}$
288 w		Cu—O stretching
225 vw		stretching modes of Cu—Cl
201 mbr 143wbr 129 m 120w 83 m		External modes

Relative intensities: sh, shoulder; m, medium; w, weak; vw; very weak; mbr, medium broad; s, strong; vs., very strong.

Table 3. Assignment and frequencies (cm^{-1}) observed for IR and Raman spectra of $[\text{Cu}_{0.335}\text{Se}_{0.582}(\text{HSeO}_3)_2\text{CuCl}_3(\text{H}_2\text{O})_3]$ at room temperature.

less sensitive to the hydrogen bond strength than the $\nu(\text{OH})$ mode [15]. A medium band observed at 1280 cm^{-1} in the IR spectrum is attributed to the in-plane $\nu(\text{OH})$ bending mode and a medium intense broad band in the $870\text{--}940\text{ cm}^{-1}$ region to the $\nu(\text{OH})$ mode.

Two prominent broad bands were observed in the stretching region of the typical water in the Raman spectra of the main (title) compound. Similarly, in the IR spectrum, a corresponding strong broad band with two distinct peaks exhibited at 3553 and 3170 cm^{-1} are noticeable for this mode. The bending mode of H_2O that appears at around 1606 cm^{-1} in the IR is noteworthy. The considerable shifting of stretching and bending frequencies from those of a free water

molecule (H_2O) [23] may be an indication of the presence of strong hydrogen bonding in the new crystal. The external modes of the HSeO_3 ion, lattice modes of water, and metal-oxygen stretching modes appear approximately below 200 cm^{-1} [1].

3.3. Dielectric studies

Figure 8 shows an illustration of the temperature dependence of the dielectric constant (ϵ') in the frequency range [1–10] KHz, and in the temperature region of 300–500 K obtained for $[\text{Cu}_{0.335}\text{Se}_{0.582}(\text{HSeO}_3)_2\text{CuCl}_3(\text{H}_2\text{O})_3]$. These curves (**Figure 8**) exhibit the following characteristics:

(1) There is one anomaly in the dielectric constant ϵ' observed at about 383 K, (2) there is a maxima in the permittivity curves, displaced to higher temperatures with increasing frequency, and (3) apparently, this is a transition which can be attributed to the “order-disorder” phase transition, probably characterizing the motion of H^+ diffusion related to the motion of HSeO_3 groups, as reported in the literature [16, 17].

Figure 8. Temperature dependence of ϵ' as a function of frequency.

4. Conclusion

In conclusion, the author ascertains that a novel substituted hydrogen selenites $[\text{Cu}_{0.335}\text{Se}_{0.582}(\text{HSeO}_3)_2\text{CuCl}_3(\text{H}_2\text{O})_3]$, have been successfully prepared via slow evaporation method. The crystal structure of the novel compound is characterized by the presence of structural blocs with structures as such $[\text{Cu}_{0.335}\text{Se}_{0.582}(\text{HSeO}_3)_2]$ and $[\text{CuCl}_3(\text{H}_2\text{O})_3]$. The principal compound is arranged to form layers in the structure parallel to the (001) plane between which the lone pairs E are located. So, the main feature of the structure of this compound is based on different coordination polyhedral, SeO_3 pyramids, and $[\text{CuCl}_3(\text{H}_2\text{O})_3]$ groups. The presence of hydrogen selenites (Se—O—H) was confirmed by IR and Raman spectra. The particularity of $[\text{Cu}_{0.335}\text{Se}_{0.582}(\text{HSeO}_3)_2\text{CuCl}_3(\text{H}_2\text{O})_3]$ is that it undergoes a phase transition on heating at 383 K. High temperature structure investigation of the new compound is in our future plans in order to confirm the nature of this transformation.

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Author details

Mohamed Loukil

Address all correspondence to: m.loukil@yahoo.fr

Sfax Faculty of Sciences - Sfax University, Laboratory Material Sciences and Environment, Tunisia

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