

# Synthesis and Characterization of a New Coordination Compounds of Cr(III), Fe(III) and Cu(II) with Ligand Derived from N,N'-bis(Salicylidene)-Methinmethyldiamine

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*Several new coordination compounds of Cr(III), Fe(III) and Cu(II) with ligand derived from N,N'-bis(salicylidene)-methinmethyldiamine were synthesized in the molar ratio of ligand to central atom of 1:1. These compounds were also characterized using chemical analysis, derivatographic and thermogravimetric analyses, IR absorption spectroscopy, ESR spectroscopy, Mössbauer spectroscopy and X-ray diffraction. The new compounds crystallize in triclinic system with Cr(III), Fe(III) or Cu(II) hexacoordinated as central atoms in octahedral structures. The obtaining reactions of these complexes can be used in gravimetric determination of Cr(III), Fe(III) or Cu(II) with an error of  $\pm 0.21\%$ .*

*Keywords: Chromium(III), Iron(III), Copper(II), precipitating reagent*

Schiff bases, where is also classified N,N'-bis(salicylidene)-methinmethyldiamine ligand, have been employed for a long time in the synthesis of coordinative compounds, some of them having a special importance [1-10]. These compounds have properties of catalysts for enantioselective epoxidation or organic redox reaction [2, 3, 5]. Various Schiff bases are used as chelating analytical reagents [1, 8-13].

In this paper is presented the synthesis and the study of the complex compounds obtained from the reactions of N,N'-bis(salicylidene)-methinmethyldiamine with Cr(III), Fe(III) and Cu(II) ions in the combination ratio of the reactants of 2:1. The obtained reaction products were investigated by chemical analysis, derivatographic thermogravimetry, IR absorption spectroscopy, ESR spectroscopy, Mössbauer spectroscopy and X-ray diffraction. The central atoms Cr(III), Fe(III) and Cu(II) are hexacoordinated in octahedral structures with donor atoms of oxygen and nitrogen provided by N,N'-bis(salicylidene)-methinmethyldiamine. Based on the X-ray diffractograms, it was determined that the novel compounds crystallize in triclinic system. It is of great importance that the obtaining reactions of these compounds can be applied to determine quantitatively Cr(III), Fe(III) and Cu(II). In addition, these complexing reactions present take place under normal conditions, and the formed precipitates can be easily separated by filtration.

## Experimental part

All chemicals were of analytical reagent grade. As reactants were employed  $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$ ,  $\text{FeCl}_3$ ,  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  (purchased from Merck), whilst N,N'-bis(salicylidene)-methinmethyldiamine was obtained according to the direction given in literature [11-13]. The syntheses of studied coordinative compounds were achieved according to the methods described in literature [11]. At first were prepared 0.2 M solutions of reactants using water as solvent

for Cr(III), Fe(III) and Cu(II) salts, and respectively a 1:1 (in volumes) mixture of ethylic alcohol (98%) and water for N,N'-bis(salicylidene)-methinmethyldiamine ( $\text{H}_2\text{L}$ ).

The syntheses of the studied compounds were carried out by mixing and stirring of  $\text{H}_2\text{L}$  ligand solution (200 mL in each synthesis) with each one of Cr(III), Fe(III) and Cu(II) solutions (200 mL), at room temperature for 60 min. Reaction products are precipitates and can be separated by filtration, then washed on the funnel vacuum nozzle and dried at 100°C until a constant weight was reached. Under the mentioned conditions, 100% efficiencies were achieved.

The content in C, H, N and Cr(III), Fe(III) or Cu(II) was determined for each obtained compound by using the appropriate chemical method [11, 13].

The thermal analysis and parameter determination of thermal decomposition reactions were achieved by means of derivatograms recorded with a Q1500D (MOM Budapest) derivatograph. An amount of 50 mg from each investigated compound was introduced into a ceramic crucible and heated up to a temperature of 1000°C. The heating rate was 10°C/min. The recorder was set at the following values: TG - 500  $\mu\text{V}$ , DTG-2.5 mV and T-500  $\mu\text{V}$ .  $\text{Al}_2\text{O}_3$  calcinated at 1200°C was used as reference material.

The absorption spectra in the IR range of the ligand and synthesized compounds were recorded between 4000 - 200  $\text{cm}^{-1}$  on a Perkin Elmer FT-IR 100 spectrometer with KBr pellet technique.

ESR spectra of the studied coordinated compounds were recorded on an IFA Bucharest spectrometer using diphenyl picryl hydrazyl (DPPH) under a magnetic field of 3216.9 Gauss that corresponds to sample centre at a frequency of 9030 MHz. Based on the methods described in [17], g factor and the number of electrons corresponding to one gram of each central atom using DPPH as reference were calculated. For this purpose, it was employed the graphical method of double integration and the

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relationship  $N_x = N_e \cdot A_x / A_e$  where:  $N_x$  and  $N_e$  ( $= 2.81 \cdot 10^{-19}$  odd electrons/mL) represent the number of odd electrons of the investigated and reference samples;  $A_x$  and  $A_e$  – areas of ESR spectra of the analyzed sample and reference respectively. The spectroscopic splitting factor was calculated with the relationship  $g = g_e \cdot H_x / H_e$  where:  $g$  and  $g_e = 2.0055$  are spectroscopic splitting factor of the investigated and reference samples;  $H_x$  and  $H_e = 3216.9$  Gauss - magnetic field of the spectrum centre of the investigated and reference samples. These two relationships can be applied only when both spectral curves (investigated and reference samples) are of the same type (Gauss or Lorentz). ESR spectral curves of all the investigated compounds and reference employed are of the Lorentz type.

Mössbauer spectra of the compound containing Fe(III) were recorded with an electrodynamic device with an uniform accelerated motion of the source. As source,  $^{57}\text{Co}$  isotope in Cr matrix was used. Values of isomer shift,  $\delta$ , were measured as against the reference of sodium nitroprusside that present a doublet with values of  $\Delta E_0$  of 0.82 and 60 mm/s respectively. The experiments were carried out at room temperature (300 K) and at liquid nitrogen temperature (80 K). Mössbauer spectra were processed by means of an EC-1033 computer provided with specialized software.

X-ray diffractogram recordings of the studied compounds were achieved for solid samples by means of a Siemens D-500 diffractometer provided with data acquisition system in the angular range of  $2\theta$  between 5 and  $60^\circ$ , a step of  $0.1^\circ/\text{s}$ , at room temperature with Cu ( $K_\alpha$ ) radiation filtered by Ni. The anode voltage was 40 KV, while the anodic current was 100 mA. Diffractograms and data processing were performed with TREOR, DIFRAC-AT, CRYSTAL softwares according to the direction in literature.

## Results and discussions

Based on the experimental data and combination ratio of Cr(III), Fe(III) and Cu(II) ions with N,N'-bis(salicylidene)-methinmethyldiamine, chemical analysis of synthesized compounds (table 1) was performed with an error of  $\pm 0.22\%$  in order to determine the content in C, H, N, Cr(III), Fe(III) and Cu(II) respectively. These analyses emphasized that one anion derived from ligand and two molecules of water correspond to each central atom. In case of the compounds containing Cr(III) and Fe(III), it was emphasized an OH group bound to the central atom. Therefore, it can be concluded that the formulae of the synthesized compounds are as follows:

$\text{Cr}(\text{C}_{16}\text{H}_{14}\text{N}_2\text{O}_2)(\text{OH})\times\text{H}_2\text{O}$ ,  $\text{Fe}(\text{C}_{16}\text{H}_{14}\text{N}_2\text{O}_2)(\text{OH})\times\text{H}_2\text{O}$  and  $\text{Cu}(\text{C}_{16}\text{H}_{14}\text{N}_2\text{O}_2)\times 2\text{H}_2\text{O}$ . Further in, these compounds will be noted as CrL(OH) $\times\text{H}_2\text{O}$ , FeL(OH) $\times\text{H}_2\text{O}$  and CuL $\times 2\text{H}_2\text{O}$  respectively.

Calculated according to Freeman-Carroll method [14], temperatures corresponding to thermal decomposing stages together with the reaction order and activation energy values are shown in table 2.

It is worthy to be mentioned that thermal decomposing stages listed in table 2 refer to thermal decomposing that follows the elimination of coordinated water. Water in the investigated compounds is removed at the following temperatures: for CrL(OH) $\times\text{H}_2\text{O}$  at  $165\text{-}180^\circ\text{C}$ , for FeL(OH) $\times\text{H}_2\text{O}$  at  $160\text{-}185^\circ\text{C}$  and for CuL $\times 2\text{H}_2\text{O}$  at  $150\text{-}180^\circ\text{C}$ . The coordination water is removed at higher temperature values than crystallization and zeolitic water [19].

Heat alone is a decomposing agent, yet in practice it commonly accelerates the action of some degrading agents such as oxygen, humidity, etc. [15, 16]

Processes taking place under the action of temperature and oxygen from air are sometimes very complex and difficult to explain. Often are emphasized temperatures at the beginning and ending stages of thermal decomposing. Also, reaction order and activation energy are calculated [14-18]. For this reason, thermal stability of the synthesized compounds was investigated. Elimination of the coordinated water is followed by two stages of thermal decomposing. In table 2 there are shown the corresponding values of reaction order and activation energy. Final solid products of thermal decomposing reactions are metal oxides such as  $\text{Cr}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$  and CuO. Molecular structure of gaseous products yielded by thermal decomposing of investigated compounds could not be identified.

Thermal stability decreases with the increase of central atom basicity:  $\text{CuL} > \text{FeL}(\text{OH}) > \text{CrL}(\text{OH})$ , which is in agreement with other works reported so far [11-13, 17, 18]. It is worthy to note that the reaction order increases from the first stage to the second one as the layer through which is eliminated the volatile component becomes thinner. If the compound layer had the thickness of a molecule, the elimination of gaseous products would not have withstood any resistance and the reaction order should be equal to 1. In reality, the layer of substance to be thermally decomposed might be of a few millimetres. As thermal decomposing takes place, this layer is thinner and thinner from a stage to another, and dispersion degree increases due to chemical reaction. Consequently, gaseous products withstand lower resistance to evaporate

**Table 1**  
ELEMENTAL COMPOSITION OF STUDIED COMPOUNDS (%).

Compound	C		H		N		M	
	Exp.	Calc.	Exp.	Calc.	Exp.	Calc.	Exp.	Calc.
$\text{CrL}(\text{OH})\cdot\text{H}_2\text{O}$	54.39	54.46	4.81	4.95	7.93	7.98	14.73	14.58
$\text{CdL}_2\text{FeL}(\text{OH})\cdot\text{H}_2\text{O}$	53.80	53.68	4.76	4.62	7.84	7.76	15.65	15.52
$\text{CuL}\cdot 2\text{H}_2\text{O}$	52.52	52.63	4.92	4.83	7.66	7.58	17.38	17.45

**Table 2**  
CHARACTERISTIC TEMPERATURES OF THERMAL DECOMPOSING STAGES, REACTION ORDERS AND ACTIVATION ENERGIES (KJ/mol) OF THE STUDIED COMPOUNDS

Compound	Stage I				Stage II			
	Ti °C	Tf °C	n	E (kJ/mol)	Ti °C	Tf °C	n	E (kJ/mol)
$\text{CrL}(\text{OH})$	190	295	0.75	189.3	295	475	0.85	270.3
$\text{FeL}(\text{OH})$	185	280	0.80	179.4	280	410	0.88	195.7
$\text{CuL}$	150	270	0.78	205.6	270	500	0.90	315.2

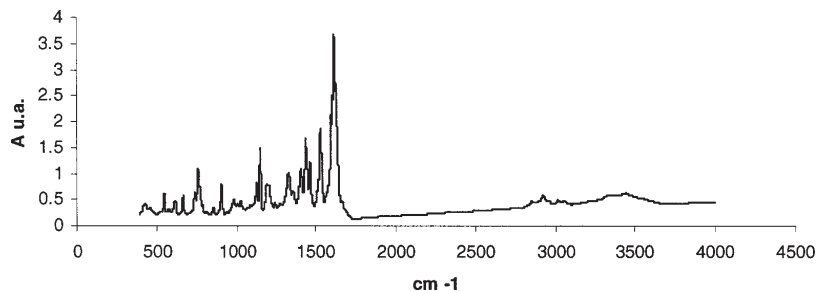


Fig. 1. IR absorption spectrum of  $\text{CuL}\cdot 2\text{H}_2\text{O}$  complex.

**Table 3**  
IR SPECTRAL BANDS CORRESPONDING TO  $\text{H}_2\text{L}$  LIGAND AND  
 $\text{CrL}(\text{OH})\cdot\text{H}_2\text{O}$ ,  $\text{FeL}(\text{OH})\cdot\text{H}_2\text{O}$ ,  $\text{CuL}\cdot 2\text{H}_2\text{O}$  COMPOUNDS ( $\text{cm}^{-1}$ ).

Ligand and complexes	$\nu_{\text{H}_2\text{O}}$	$\nu_{\text{C}=\text{N}}$	$\delta_{\text{OH}}$	$\nu_{\phi}$	$\nu_{\text{M}-\text{O}}$	$\nu_{\text{M}-\text{N}}$
$\text{H}_2\text{L}$	-	1625	1280	1560	-	-
$\text{CrL}(\text{OH})\cdot\text{H}_2\text{O}$	3420-3275	1615	-	1540	480	695
$\text{FeL}(\text{OH})\cdot\text{H}_2\text{O}$	3400-3250	1605	-	1550	434	686
$\text{CuL}\cdot 2\text{H}_2\text{O}$	3380-3230	1595	-	1530	450	663

and reaction order tends to 1, which is in good agreement with data presented in this paper and also in literature [11, 15, 18].

IR absorption spectra of ligand and synthesized compounds point out important information on chemical bonds of central atoms Cr(III), Fe(III) and Cu(II) with some atoms in these compounds. The main purpose of spectra interpretation was to note whether there are bands corresponding to bonds like M-O and M-N= (M represents chromium, iron and copper ions) originated from the interaction of functional groups  $>\text{COH}$ ,  $-\text{N}=\text{CH}-$  and with  $\text{OH}_2$  molecule (fig. 1). Thus, it was emphasized the presence of some very important bonds between central atoms Cr(III), Fe(III) and Cu(II) and donor ligand atoms, oxygen and nitrogen corresponding to stretch frequencies:  $\nu_{\text{M}-\text{O}}$  and  $\nu_{\text{M}-\text{N}}$  (table 3).

IR absorption spectra of  $\text{H}_2\text{L}$  ligand correspond to Schiff bases [22-24]. All Schiff bases, including  $\text{H}_2\text{L}$  present wide bands partially superposed in the range  $32450-3220\text{cm}^{-1}$ , that can be ascribed to phenolic OH group in *orto* position. In case of  $\text{H}_2\text{L}$ , the absorption of this group lies between  $3290$  and  $3275\text{cm}^{-1}$  which might suggest intra or inter molecular hydrogen bond formation ( $-\text{O}-\text{H}\dots\text{N}$  respectively  $-\text{O}-\text{H}\dots\text{O}$ ).

In agreement with data reported by other authors [6, 7, 11], it can be observed at  $3145-3120\text{cm}^{-1}$  a high and sharp frequency corresponding to stretch vibration of CH group in  $\text{H}_2\text{L}$  molecule. Azomethinic group occurs at a stretch vibration as split bend pattern with two maximums ranged between  $1620-1600$  respectively  $1605-1575\text{cm}^{-1}$ , which is also in agreement with literature [6, 11].

Absorption bands within the range  $1285-1220\text{cm}^{-1}$  are ascribed to Schiff base stretch vibrations of C-O (phenolic) [23, 24, 27]. Substitution of hydrogen atom in OH group (phenolic) with metal atoms determines the disappearance of IR absorption spectra of  $\nu_{\phi\text{OH}}$ . High frequency band due to the absorption of azomethinic group is shifted after complexation due to the coordinative bond formed between nitrogen atom of this group and atoms of the respective metals,  $>\text{C}=\text{N}\rightarrow\text{Mn}^{n+}$ . Noticed in the range  $1285-1225\text{cm}^{-1}$  on  $\text{H}_2\text{L}$  spectra, bands of vibration frequencies belonging to C-O group become less intense and are shifted towards higher wavelengths in spectra of complex compounds.

The presence of coordinated water molecules in all synthesized compounds is pointed out through a wide band in the range  $3510-3250\text{cm}^{-1}$  and two relatively weak bands around  $850$  and  $700\text{cm}^{-1}$ , that can be assimilated to OH

group that corresponds to stretch vibrations according to literature [5-7, 22]. Therefore, it can be inferred that chelate structures with 6 respectively 4 atoms are formed.

In references [21, 24] is stated based on experimental data that IR absorption spectra give information on the type of water contained in various substances, such as adsorption, insertion, crystallization, coordination or constitution water. According to these authors, constitution water is OH group bond to a metallic ion having the absorption at  $1660\text{cm}^{-1}$ . This was also noticed in case of  $\text{CrL}(\text{OH})\cdot\text{H}_2\text{O}$  and  $\text{FeL}(\text{OH})\cdot\text{H}_2\text{O}$  compounds that present absorption maximums at  $1670$  and  $1665\text{cm}^{-1}$  respectively. These OH groups bond to central atoms Cr(III) and Fe(III) pass in water molecules and an oxygen atom bond to metallic atoms [11, 15, 21].

Processing ESR spectra [10, 19, 27], calculating g factor and the number of odd electrons corresponding to a central atom (table 4) can be drawn the following conclusions:

**Table 4**  
VALUES OF G FACTOR, MAGNETIC FIELD INTENSITY  $H_x$  (GAUSS)  
AND ODD ELECTRON NUMBER OF THE CENTRAL ATOM Cr(III),  
Fe(III) AND Cu(II).

Compound	g	$H_x$	N
$\text{CrL}(\text{OH})\cdot\text{H}_2\text{O}$	2.0136	3230	2.88
$\text{FeL}(\text{OH})\cdot\text{H}_2\text{O}$	2.0200	3240	4.91
$\text{CuL}\cdot 2\text{H}_2\text{O}$	2.0295	3255	0.93

- all central atoms of synthesized compounds contain in the external layer a maximum number of odd electrons: 3 in Cr(III), 5 in Fe(III) and 1 in Cu(II);

- spectroscopic splitting factor of odd electrons in the structure of central atoms is higher than that of free electron due to the contribution of orbital momentum and covalence degree of central atom bonds;

- g value decreases with the increase of the compound stability.

Mössbauer spectrum recorded for  $\text{FeL}(\text{OH})\cdot\text{H}_2\text{O}$  at a temperature of  $300\text{K}$  indicates a III<sup>rd</sup> oxidation state with high spin ( $S=5/2$ ) of central atom Fe(III), which is in agreement with the relatively ionic character of O-Fe-N bonds (fig.2).

Values of  $E_o$  parameter (table 5) indicates an octahedral encompassment of Fe(III) central atom with a disposal of the electrons in sublevels:  $t_{2g}^3$  and  $e_g^2$ .

In case of Fe(III) compounds with various ligands, Mössbauer spectrum presents a slight shift in the symmetry of the central atom encompassment. This is also the case of  $\text{FeL}(\text{OH})\cdot\text{H}_2\text{O}$  compound when in the

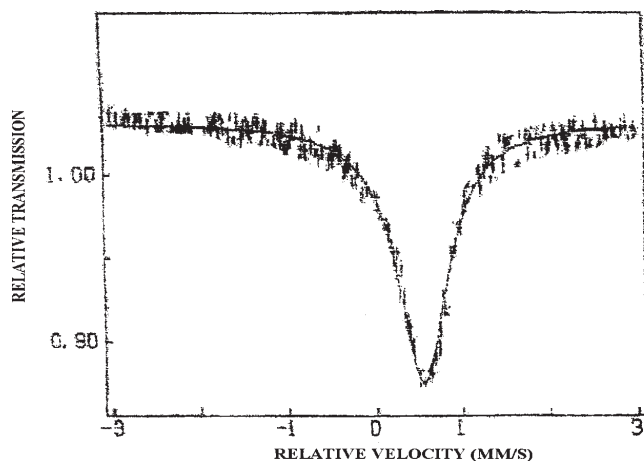


Fig.2. Mössbauer spectrogram of  $\text{FeL(OH)}\times\text{H}_2\text{O}$  complex recorded at a temperature of 300 K

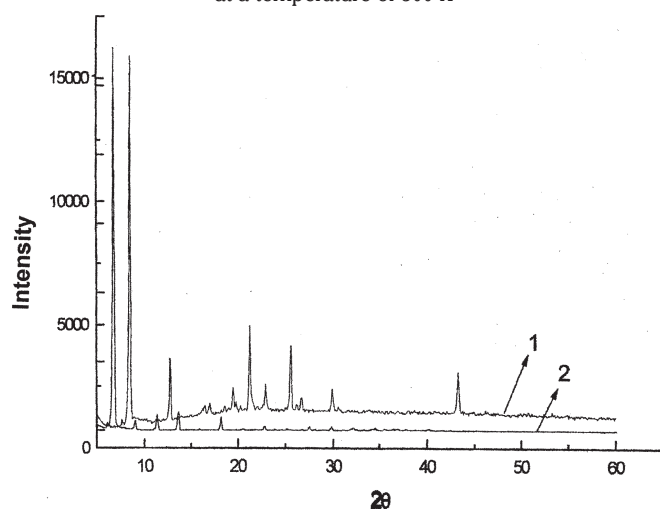


Fig. 3. . Diffractograms of  $\text{H}_2\text{L}$  (1) and  $\text{CuL}\times 2\text{H}_2\text{O}$  (2) complexes.

coordination participates two different donor atoms (O and N). Quadrupol splitting is hardly noticeable due to the participation into coordination of O and N donor atoms presenting a non-equivalent distribution on the molecular orbitals on the directions of rectangular axes. Ligand participate at Fe(III) coordination with two atoms of nitrogen and other two atoms of oxygen derived from OH group and  $\text{H}_2\text{O}$ . In this situation, the distance between Fe-O will be a little shorter than that of Fe-N. This interpretation of Mössbauer spectrum of  $\text{FeL(OH)}\times\text{H}_2\text{O}$  is in agreement with literature [10, 11, 19, 27].

Processing the X-ray diffractogram of  $\text{H}_2\text{L}$  ligand, it results that it has a high crystallinity degree which allows the obtaining of triclinic symmetry with parameters of crystalline cells presented in table 6.

Table 5  
VALUES OF MÖSSBAUER PARAMETERS OF  $\text{FeL(OH)}\times\text{H}_2\text{O}$  COMPLEX

T (K)	$\delta$ (mm/s)	$\Delta E_Q$ (mm/s)	$\Gamma$ (mm/s)
300	0.60	0.82	0.35

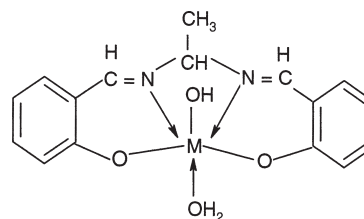


Fig. 4. . Structure of complexes  $\text{CrL(OH)}\times\text{H}_2\text{O}$  and  $\text{FeL(OH)}\times\text{H}_2\text{O}$ , where  $\text{M}=\text{Cr(III), Fe(III)}$ .

Among investigated coordination compounds it was established that only  $\text{CuL}\times 2\text{H}_2\text{O}$  presents a high crystallinity degree, which makes possible to process its diffractogram and classify it in the system of triclinic symmetry as in the case of  $\text{H}_2\text{L}$  ligand.

As regards the structures of the investigated compounds,  $\text{CrL(OH)}\times\text{H}_2\text{O}$  and  $\text{FeL(OH)}\times\text{H}_2\text{O}$  have biphasic character. Besides their local ordering states corresponding to amorphous state, these two compounds present diffraction lines characteristic to crystalline state. However these lines were not sufficient to index the diffractogram and establish the crystalline system. These compounds in structure of which the central atom is bond to OH group are like numerous compounds of Cr(III) and Fe(III) that present also local ordering which is characteristic to the amorphous state [9, 11, 28].

$\text{CuL}\times 2\text{H}_2\text{O}$  compound presents a very high degree of crystallinity that allows one to index its diffractogram. Thus, it was classified in the triclinic system having parameter values of crystalline network as listed in table 6.

Interpreting diffractograms of the ligand and  $\text{CuL}\times 2\text{H}_2\text{O}$  it gives that the coordination process of central atom Cu(II) is determined by the crystallographic characteristics of the ligand. This is possible by achieving a similarity between two from elemental cell parameters of the compound reported to those of the ligand. This similarity was achieved through very close values of b and c parameters which show that central atom Cu(II) coordinates with two atoms of nitrogen and oxygen of the ligand in the ab plane on c crystallographic direction accompanied by the volume compression of the compound elemental cell. Thus, the central atom Cu(II) coordinates with two molecules of water, one above and the other beneath the plan where is placed the ligand. However, it is preserved the octahedral symmetry of the central atom.

Table 6  
PARAMETERS VALUES OF ELEMENTAL CELLS OF A  $\text{H}_2\text{L}$  LIGAND AND  $\text{CuL}\times 2\text{H}_2\text{O}$  COMPLEX

Elemental cell parameters	Values obtained by indexation	
	$\text{H}_2\text{L}$	$\text{CuL}\cdot 2\text{H}_2\text{O}$
a (Å)	8.114	9.199
b (Å)	21.135	18.806
c (Å)	17.504	16.168
$\alpha$	55°	80°
$\beta$	87°	75°
$\gamma$	57°	60°
Elemental cell volume (Å <sup>3</sup> )	3001.75	2797.00

Based on chemical analysis, IR absorption spectra, Mössbauer spectra, ESR spectra, it was pointed out that the structures of synthesized compounds are classified in the octahedral configuration with hexacoordinated central atoms. In cases of  $\text{CrL}(\text{OH})\times\text{H}_2\text{O}$  and  $\text{FeL}(\text{OH})\times\text{H}_2\text{O}$  compounds, the coordination of central atom is established with two oxygen atoms and two nitrogen atoms of the ligand, an oxygen atom derived from OH group and another oxygen atom from water molecule. For  $\text{CuL}\times 2\text{H}_2\text{O}$ , beside two atoms of nitrogen and two of oxygen from ligand, two atoms from water molecules participate to the coordination of central atom of Cu(II).

### Conclusions

Three new compounds were synthesized through the interaction of  $N,N'$ -bis(salicylidene)-methinmethyldiamine with Cr(III), Fe(III) and Cu (II) in the molar ratio of 1:1 in weak acid medium.

These compounds were characterized employing modern methods, such as chemical analysis, derivatographic analysis, IR absorption spectroscopy, ESR spectroscopy, Mössbauer spectroscopy and X-ray diffraction.

It was established that to each central atom of Cr(III) and Fe(III) respectively correspond an divalent anion derived from  $N,N'$ -bis(salicylidene)-methinmethyldiamine ligand, an OH group and a water molecule. To Cu(II) central atom corresponds an anion of the same type of ligand and two water molecules.

The new complexes crystallize in triclinic system with the central atom hexacoordinated in octahedral structures.

The obtaining reaction of these new complexes can be employed to dose Cr(III), Fe(III) and Cu(II) by gravimetric method with an error of  $\pm 0.21\%$ .

### References

1. EL-KASSEM, M., MOHAMED MOSSOUNA, Folosirea bazelor Schiff ca reactivi analitici cheletizanti, Teză de doctorat, Institutul Politehnic Iași, 1980
2. SIMANDI, L.I., Dioxigen Activation and Homogeneous Catalytic Oxidation, Elsevier Amsterdam, 1991
3. ROCKDIFFE, A.D., MARTELL, E.A., J. Molecular Catalysis A., Chemical, **106**, 1996, p.211
4. PUI, A., BERDAN I., CASCAVAL AL., Rev. Roum. Chim., **45(4)**, 2000, p.331
5. PUI, A., Rev. Roum. Chim., **46**, 2001, p. 67
6. PUI, A., VIZITIU, M., Rev. Chim.(Bucuresti), **58**, no. 1, 2007, p. 25

7. ROȘU, T., GULEA, A., PAHONȚU E., COTOVAIA, A., Rev. Chim.(Bucuresti), **58**, no. 5, 2007, p. 475
8. FLONDOR, M., ROȘCA, I., SIBIESCU, D., Rev. Chim.(Bucuresti), **59**, no. 12, 2008, p. 1330
9. FLONDOR, M., SIBIESCU, D., ROȘCA, I., SUTIMAN, D., CĂILEAN, A., CREȚESCU, I., Rev. Chim.(Bucuresti), **60**, no. 2, 2009, p. 119
10. SUNDARAVEL, K., SURESH, E., PALANIANDAVAR, M., Inorganica Chimica Acta, **362**, 2009, p. 199
11. FLONDOR, M., Compuși coordinativi ai cationilor unor metale tranzitionale, Teză de doctorat, Facultatea de Inginerie chimică și Protecția mediului, Univ. „Gh. Asachi” Iași, 2007
12. POPA, GR., MOLDOVEANU, S., Reactivi organici în chimia analitică, Ed. Acad. Române, București, 1976
13. POPA, GR., CROITORU, V., Chimie analitică cantitativă, Ed. Didactică și Pedagogică, București, 1971
14. FREEMAN, S.E., CARROLL, B., J. PHY. Chem., **62**, 1958, p. 314
15. BROWN M., E., Introduction to Thermal Analysis, Ed. Springer, 2001
16. SEGAL, E., FĂTU, D., Introducere în cinetica reacțiilor neizoterme, Ed. Acad. Române, București, 1983
17. ROȘCA, I., FOCA, N., SUTIMAN, D., CĂILEAN, A., SIBIESCU, D., VIZITIU, M., J. Thermal Analysis and Calorimetry, **56**, 1999, p. 401
18. ROȘCA, I., SUTIMAN, D., SIBIESCU, D., VIZITIU, M., CĂILEAN, A., OPREA, L., J. Serbia Chem. Soc., **67**, 2002, p. 617
19. POLIVAN, C.G., Studii de rezonanță paramagnetică electronică a structurii unor combinații complexe de interes biologic, Ed. Acad. Române, București, 1995
20. EBSWORTH, V.A.E., ROUKIN, H.W., CRADOCK, S., Structural methods of inorganic chemistry, Blackwell Sci., Oxford, 1998.
21. ROCCHICCIOLI, C., La spectrographie d'absorbtion infrarouge, moyen d'étude de l'eau dans les substances minerales, Chimie Analytique, nr. **9**, p. 452, 1964
22. MAHON, M., GINLEY, J., Mc., ROONEY, A., WALSH, M., D., Inorganica Chimica Acta, **362**, 2009, p. 2353.
23. LECONTE, J., Spectroscopie dans l'infrarouge. Ed. Dunod, Paris, 1995
24. BALABAN, T. A., BANCUI, M., POGANY, I., Aplicații ale metodelor fizice în chimia organică, Ed. Științifică și Enciclopedică București, 1983
25. GOLDONCHI, I., HREBER, R., H., Chemical Application of Mossbauer Spectroscopy, New York, London Acad. Press., 1978
26. SIBIESCU, D., Chimia compușilor coordinativi, Ed. Tehnopress, Iași, 2005
27. FOCA, N., SIBIESCU, D., OANCEA, S., Metode fizico-chimice aplicate în studiul combinațiilor complexe, Ed. Tehnopress, Iasi, 2006
28. WELLES, F.A., Structural inorganic chemistry, Clarendon Press Oxford, 1999

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