Synthesis, Characterization, And Antibacterial Activities Of Manganese (II), Cobalt(II), Iron (II), Nickel (II) , zinc (II) And Cadmium(II) Mixed-Ligand Complexes Containing Amino Acid(L-Valine) And Saccharin

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Abstract

A new six mixed ligand complexes of some transition metal ions Manganese (II), Cobalt(II), Iron (II), Nickel (II) , and non transition metal ion zinc (II) And Cadmium(II) with L-valine (Val H) as a primary ligand and Saccharin (HSac) as a secondary ligands have been prepared. All the prepared complexes have been characterized by molar conductance, magnetic susceptibility infrared, electronic spectral, Elemental microanalysis (C.H.N) and AA . The complexes with the formulas $[M(Val)_2(HSac)_2]$
M= Mn (II) , Fe (II) , Co(II) ,Ni(II), Cu (II),Zn(II) and Cd(II)
L- Val H= $(C_5H_{11}NO_2)$, $C_7H_5NO_3S$

The study shows that these complexes have octahedral geometry; The metal complexes have been screened for their in microbiological activities against bacteria. Based on the reported results, it may be concluded that the deprotonated ligand (L-valine) to (valinate ion (Val^-) by using (Na OH) coordinated to metal ions as bidentate ligand through the oxygen atom of the carboxylate group($-COO^-$), and the nitrogen atom of the amine group (NH_2), where the saccharin (H Sac) coordinated as a monodentate through the nitrogen atom.

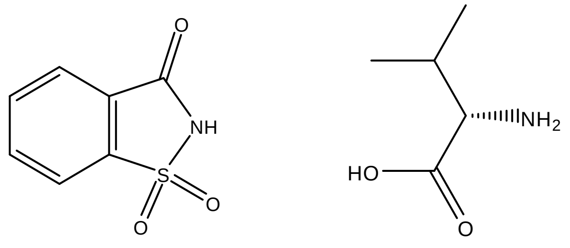
Keywords: L-Valine , mixed ligand complexes, Saccharin , spectral studies , Antibacterial activities.

1 – Introduction

The chemical properties and especially the physiological and biochemical activity of saccharin and its compounds have been intensively investigated mainly because of its suspected carcinogenic nature. In particular, since it has been shown that it causes cancer in rats, saccharin joined the list of human potential cancer-causing substances in USA .[1]

The structural data for metal saccharinates and metal complexes including saccharin and various coordinated mono- (imidazole, pyridine) or polycyclic N-donor ligands (2,2'-bipyridine, 1,10-phenantroline) were retrieved from the Cambridge Structural Database and analyzed. The influence of the nature of the metal ion and of the type of the metal-to-ligand bonding on the saccharinato geometry was examined. The structural data obtained by X-ray diffraction .[2-3]

Studies of mixed ligand complexes containing saccharinate and some other ligands have demonstrated that the coordination mode of saccharinate is highly adaptable to the steric requirements of the complex formed. [3-4] The coexistence of bonded and non-bonded saccharinate anions has also been demonstrated in a number of cases. Also the presence of non-deprotonated saccharin molecules in a number of complex structures has been established. .[4] Saccharin is a non nutritive sweetener, meaning that it is not metabolized by the body to produce energy. [5] Saccharin ($C_7H_5NO_3S$), also called o-sulfobenzoimide, is widely used as an artificial sweetening agent. Saccharin is a weak acid [1] .



L-Valine ($C_5H_{11}NO_2$) Saccharin ($C_7H_5NO_3S$)

Figure (1) formulae Saccharin and L-Valine

saccharinate moiety can act either as a single bidentate ligand for only one metal center or, more frequently, as a bidentate bridging ligand. A recent example for the first situation is the complex $[Pb(sac)_2ophen(H_2O)_2]$ (ophen = 1,10-phenanthroline) in which Pb(II) presents the unusual coordination number eight, with the two saccharinate anions acting as bidentate and the coordination sphere completed by the two N atoms of ophen and the two water O-atoms[6] . In the case of the simpler $[Pb(sac)_2].H_2O$ complex, the bidentate ligand originates a dimeric structure. [7]

Besides, the presence of free saccharin in the crystal lattices of certain complexes have been established, as mentioned above in the case of the $[Ln(sac)_2(H_2O)_6](sac)(Hsac).4H_2O$ complexes [8] Not withstanding, the first case in which this situation was found is, apparently, the VO^{2+} complex of composition $[VO(OH)(sac)(H_2O)_2(Hsac)]$ [4]

L-Valine is essential amino acid [8] widely distributed but rarely occurs in amount exceeding 10%. It is branched chain amino acid and can be derived from alanine by the introduction of two methyl group present on α-carbon atom. This is glycogenic. On domination, it forms methyl-malonyl-CoA which can be converted to succinyl -CoA in place of two H atoms of the methyl group. [9]

The present investigation deals with the preparation, spectroscopic studies of the complexes obtained during the reaction of saccharin and L-Valine with Manganese (II),Cobalt(II),Iron (II),Nickel (II),Zinc (II) and Cadmium(II) ions with the aim of investigating the coordination mode of saccharin and L-Valine in these complexes.

2- Experimental Section:

2-1. Materials:

All of the chemical used throughout this investigation were extra pure grade.

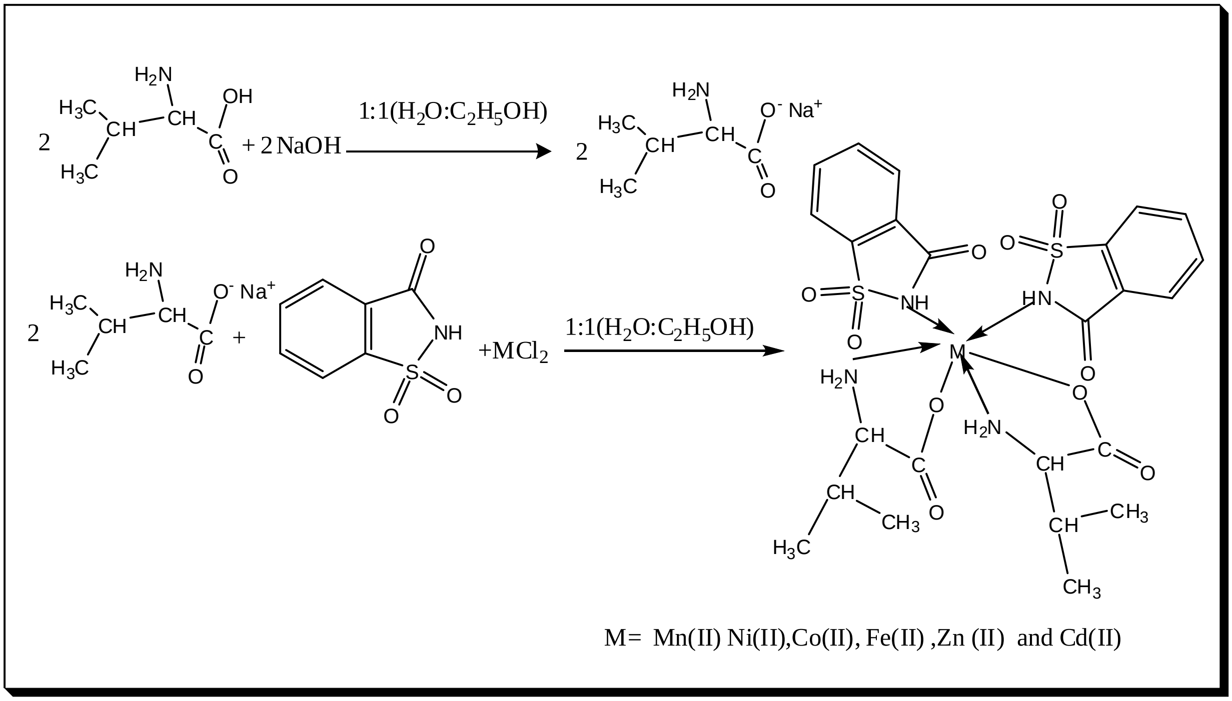
Chemicals Saccharin, (L-Valine), and salts were purchased from Merck and BDH were used without further purification.All the metal ions Mn(II),Co(II),Ni(II), Fe(II), Zn and Cd(II) were of Analar grade (BDH). They were used in the form of chlorides without further purification.

2-2. Instruments:

I.R spectra were recorded as KBr discs using Fourier Transform Infrared Spectrophotometer Shimadzu 24FT-I.R8300. Electronic spectra of the prepared complexes were measured in the region (200- 1100) nm for 10^{-3} M solutions in DMF at 25 ℃ using shimadzu-U.V-160 A Ultra Violet Visible- Spectrophotometer with 1.000 ± 0.001 cm matched quartz cell .Identify the metal percentage in the complexes by using Shimadzu flame atomic absorption Model;6809. Electrical conductivity measurements of the complexes were recorded at 25 ℃ for 10^{-3} M solutions of the samples in DMF using pw9527 Digital conductivity meter (Philips). Elemental microanalysis, was carried out using C.H.N elemental analyzer model 5500-Carlo Erba instrument. Melting points were recorded by using Stuart melting point apparatus. Chloride ion content were also evolution by (Mohr method), Magnetic susceptibility measurements were measured using Bruker magnet BM6.

2-3. General synthesis of the mixed ligand metal complexes[11]

A solution of Saccharin (0.366g, 2mmol) in aqueous ethanol (1:1, 10 ml) and solution of L-valine (0.234, 2 m mol) in aqueous ethanol (1:1,10 ml) containing sodium hydroxide (0.08, 2mmol) were added simultaneously to a solution of $MCl_2.nH_2O$ (1 m mol) in aqueous ethanol (1:1, 10 ml) in the stoichiometric ratio.[2Val:M: 2SacH].(Scheme 1) the above solution was stirred for 2o minute and allowed to stand for over night .the product formed was filtered off ,washed with aqueous ethanol (1:1) and dried in air ,and analyzed employing standard method .



M= Mn(II) Ni(II),Co(II), Fe(II) ,Zn (II) and Cd(II)

Scheme (1): Schematic representation Preparation of the Complexes [M(Val)$_2$(HSac)]

2-4. Antibacterial Activities: [12]

The antibacterial activity of the ligands and there complexs were tested on Gram positive bacteria, **Staphylococcus aureus** (+ve), and(**Escherichia coli , Salmonella typhi and Aeruginosa**) (-ve). The solvent used was dimethyl form amid(DMF) and sample from 1 to 200 μg/ml were used. Anti bactericidal activities of each compound were evaluated by the well-diffusion method.1 cm^3 of a 24 h broth culture containing 106 CFU/cm^3 was placed in sterile Petri-dishes. Molten nutrient agar(15 cm^3) kept at ca. 45^{o}C was then poured in the Petri-dishes and allowed to solidify. Then holes of 6 mm diameter were punched carefully using a sterile cork borer and these were completely filled with the test solutions. The plates were incubated for 24h at 37^{o}C.DMF was used as control. The results are shown in Scheme (2).

3- Results and Discussion

3-1. Characterization of Metal Complexes.

Generally, the complexes were prepared by reacting the respective metal chloride with the ligands using .[2Val:M: 2SacH]mole ratio, i.e. one mole of metal salt : one mole of saccharin and two moles of sodium valinate.The synthesis of mixed ligand Metal complexes may be represented as follows:

$2Val\,H + 2NaOH \rightarrow 2Val^- Na^+ + H_2O$

$2Val^- Na^+ + HSac + MCl_2 \rightarrow [M(Val)_2(HSac)] + 4H_2O + NaCl$

(where HSac is Sacarine and Val H is amino acid L-valine).

The formula weights and melting points , are given in (Table I).Based on the physicochemical characteristics (Table I), it was found that all the complexes were non-hygroscopic, stable at room temperature .The solubility of the complexes of ligands was studied in various solvents. The complexes are soluble in dimethyl sulfoxide (DMSO) , dimethyl formamide (DMF) ,ethanol, water while insoluble in common solvents such as benzene, ether, and chloroform, .The molar conductance values of the complexes in DMF solvent at 10^{-3} M concentration are very low (< 3) signifying their non-electrolytic nature [13]

3-2. Atomic Absorption and chloride ion content :

The atomic absorption measurements (Table-1) for all complexes gave approximated values for theoretical values. In conclusion, our investigation this suggest that the ligands acid L- valine and saccharin coordinate with M (II) forming octahedral geometry.

The analytical and physical data (Table 1) and spectral data (Tables 2 and 3) are compatible with the suggested structures Figure (2).

3-3. The UV-Visible Spectroscopy and Magnetic measurements:

The magnetic moments of the complexes shown in Table (2) were calculated from the measured magnetic susceptibilities after employing diamagnetic corrections. The electronic spectral data of the free ligands saccharin, L-valine and their complexes are summarized in Table -2 together with the proposed assignments and suggested geometries. The results obtained are in good agreement with other spectra and the literature. [14-16] The UV-Vis spectrum of the ligand (saccharin) shows peaks at 275 nm, 340 nm assigned to (π – π^*) and (n – π^*) electronic transitions. The spectrum of the free ligand L-valine), exhibits absorption peak at (280 nm)(35714 cm^{-1}) and an intense peak at 320 nm (3250cm^{-1}),which assigned to ($\pi \rightarrow \pi^*$), and ($n \rightarrow \pi^*$) transition respectively [14]

The Zn(II), and Cd(II) complexes did not display any peak in the visible region, no ligand field absorptions band was observed, therefore the bands appeared in the spectra of two complex could be attributed to charge transfer transition. in fact this result is a good agreement with previous work of octahedral geometry. The room temperature μ_{eff} value for the Co(II) complexes 3.81 B.M. suggest high spin octahedral geometry, which is further supported by the electronic spectral data .In the Ni(II) complexes. μ_{eff} value at room temperature are in 3.2 B.M. as expected for six coordinated spin free Ni(II) species. While magnetic susceptibility measurements for Cd(II), and Zn(II) (d^{10})(white complexes) showed diamagnetic as expected from their electronic configuration . [15,16].μ_{eff} for Mn^{2+} (d^5) complex was 4.22 B.M within the expected spin-only values. [17-19]. While magnetic susceptibility measurements for Cd(II), and Zn(II) (d^{10})(white complexes) showed diamagnetic as expected from their electronic configuration . [15,16]. The Pale Brown complex [Fe $(Val)_2$ $(HSac)_2$] spectrum showed three absorptions at wave number (36231)cm^{-1} related charge transfer. The other two absorption at (25706)cm^{-1} and (3696)cm^{-1} were caused by the electronic transitions $^6A_1g \rightarrow {}^4T_1g(p)$ and $^6A_1g \rightarrow {}^4A_1g, {}^4Eg(D)$ respectively. [20].

3-4. FT- Infra red spectra

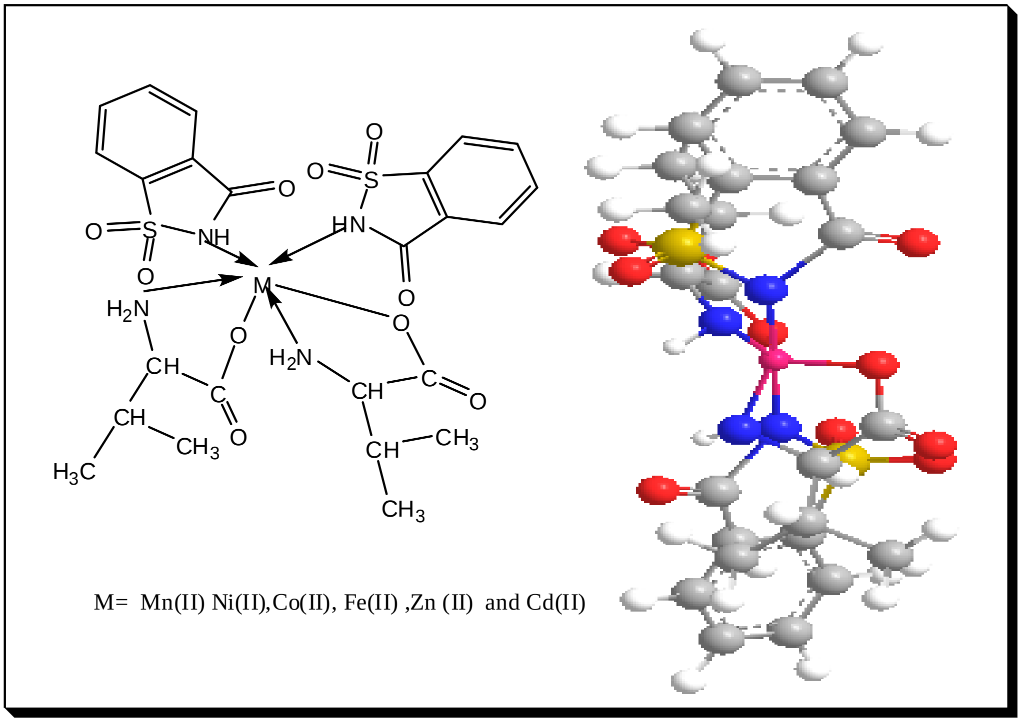
The assignment of some of the most characteristic FT-IR band of the complexes are summarized in (Table -3) together with those of (Sodium valinate ,and saccharin recorded for comparative purposes and facilitate the spectral analysis.

The FT- Infra red spectrum of free ligand (L- valin) exhibited a strong band around (3095) cm^1 that corresponds to the stretching vibration of υ (N-H) + υ (O-H), while another strong absorption band at (1612) cm^{-1} is appeared which could explained as υ $(-CO\bar{O})_{asy}$ where the υ $(-CO\bar{O})_{sym}$ was noticed at (1473) cm^{-1} .

The Fourier transform infrared spectrum of free ligand saccharin exhibits a weak band at 3095 cm^{-1} due to the υ (N-H) vibration [21-22]. The region involving the carbonyl stretching vibrations is relatively complex, as both the C=O vibrations of saccharin and of the Imine group lie closely together, and of the NH_2 moiety are expected in the same region [23]. The two SO_2-stretching vibrations appear at similar frequencies(1298 and 1163 cm^{-1} for $\upsilon_{as}(SO_2)$ and $\upsilon_s(SO_2)$, respectively. [21-24].Absorption bands in the (680-636)cm^{-1} region are considered to be due to metal-nitrogen vibrations [20-21] whilst those occurring around (536-581)cm^{-1} are thought to arise from metal-oxygen vibration . [20-22]

3-5. Proposed molecular structure

Studying complexes on bases of the above analysis , the existence of Hexa coordinated $[M(C_7H_5NO_3S)_2(C_5H_{11}NO_2)_2]$ abbreviated as $[M(HSac)_2(Val)_2]$ were, M(II)=Mn(II), Ni(II),Co(II), Fe(II),Zn(II) and Cd(II). proposed models of the species were built with chem. 3D shows in Figure(2).



M= Mn(II) Ni(II),Co(II), Fe(II) ,Zn (II) and Cd(II)

Figure (2) : The proposed structure and 3D-geometrical structure of the complexes

3-6. Antibacterial activity

The antibacterial activity of mixed ligand complexes 1-6 against Staphylococcus aureus (+ve), and Escherichia coli , Salmonella typhi and Aeruginosa) (-ve) were carried out by measuring the inhibition diameter .The test compounds were prepared at a concentration of 200 μg / mL. Solvent control that is, DMF was also maintained throughout the experiment simultaneously .The data are given in Table 4 Scheme (2),It is evident from the above data that the antibacterial activity significantly increased on coordination. It has been suggested that the ligands with nitrogen and oxygen donor systems inhibit enzyme activity. Coordination reduces the polarity of the metal ion mainly because of the partial sharing of its positive charge with the donor groups within the chelate ring system.(23,24). .Hence produce metal chelates can be employed as antibacterial.[24]

4- Conclusion:

In this paper new mixed ligand complexes containing L-valine and saccharin with the general formula [M(HSac)2(Val)2] where Synthesis and Characterization of Mixed Ligand Complexes of L-valine and saccharin with Mn(II), Ni(II), Co(II), Fe(II), Zn(II) and Cd(II) ions . The molar conductivity of the complexes in DMF solution were non electrolyte. The results showed that the deprotonated ligand (L-valine) to (valinate ion (Val^-) by using (Na OH) coordinated to metal ions as bidentate ligand through the oxygen atom of the carboxyl ate group($-COO^-$), and the nitrogen atom of the amine group (NH_2), where the saccharin coordinated as a monodentate through the nitrogen atom. Therefore, Conclusion Thus the evidences obtained from IR spectra, electronic spectra and magneto chemical measurements suggest an octahedral configuration Figure(2).

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Table 1-The physical properties of the CompoundS

					Analysis Calc.(found)				
Compounds	**M. wt**	**Color**	**Mp ℃ (de)**	**M% Calc. (found)**	**C%**	**H%**	**N%**	**Λ_M.C µS.cm^{-1} in DMF**	**Cl%**
saccharin (ligand) (SacH)	**183.19**	**Off White**	**183**	**-**	**-**	**-**	**-**	**1.34**	**-**
Valine	**117.15**	**White**	**244**	**-**	**-**	**-**	**-**	**1.15**	**-**
Mn(Val)$_2$ (SacH)$_2$	**653.58**	**Pale - yellow**	**234de**	**8.41 (9.1)**	**44.10) (44.90**	**4.63 (4 .88)**	**8.57 (9.1)**	**1.43**	**Nill**
Fe (Val)$_2$ (SacH)$_2$	**654.49**	**Pale Brown**	**222 de**	**8.53 (9.3)**	**44.04 (44.94)**	**4.62 (4.68)**	**8.56 (9.2)**	**1.28**	**Nill**
Co (Val)$_2$ (SacH)$_2$	**657.58**	**Pale red**	**128 de**	**8.96 (9.6)**	**43.84 (44.04)**	**4.60 (4.63)**	**8.52 (9.1)**	**1.38**	**Nill**
Ni (Val)$_2$ (SacH)$_2$	**657.34**	**Pale green**	**128 de**	**8.93 (9.6)**	**43.85 (44.04)**	**4.60 (4.63)**	**8.52 (9.1)**	**1.20**	**Nill**
Zn(Val)$_2$ (SacH)$_2$	**664.06**	**White**	**208 de**	**9.66 (10. 2)**	**43.41 (44.05)**	**4.55 (4.67)**	**8.44 (9.3)**	**1.36**	**Nill**
Cd(Val)$_2$ (SacH)$_2$	**711.06**	**White**	**230 de**	**15.81 (16.3)**	**40.54 (40.90)**	**4.25 (4.62)**	**7.88 (8.1)**	**1.39**	**Nill**

M.C = Molar Conductivity, Valinato anion = Val$^-$ = Valine($C_5H_{10}NO_2$)$^-$, de = decomposition ,, saccharin = $C_7H_5NO_3S$

Table 2- Electronic Spectral data, magnetic moment, of the studied compounds

compounds	λ_{nm}	υ^{-} $_{(\text{Cm})}^{-1}$	μ_{eff} (BM)	Assignment	Probable figure
saccharin (ligand) (SacH)	340 275	29411 36363	-	n→π* π→π*	-
L- Val H	320 280	35714 31250	-	n→π* π→π*	-
Mn(Val)$_2$ (Sac H)$_2$	818 278	12224 36231	5. 04	^{6}A1g →4T1g (G) CT	Octahedral
Fe (Val)$_2$ (Sac H)$_2$	422 389 276	23696 25706 36231	5.18	^{6}A1g→^{4}A1g,^{4}Eg(D) ^{6}A1g→^{4}T1g(p) CT	Octahedral
Co (Val)$_2$ (Sac H)$_2$	677 529 278	14771 18903 35971	3.81	^{4}T1g → ^{4}T1g (p) ^{4} T1g → ^{4} A2g CT	Distorted Octahedral
Ni (Val)$_2$ (Sac H)$_2$	276 409	36231 23866	233	CT ^{3}A2g (F) → ^{3}T1g (P)	Octahedral
Zn(Val)$_2$ (Sac H)$_2$	276	36231	0.0	CT	Octahedral
Cd (Val)$_2$ (SacH)$_2$	276	36231	0.0	CT	Octahedral

CT=charge transfer

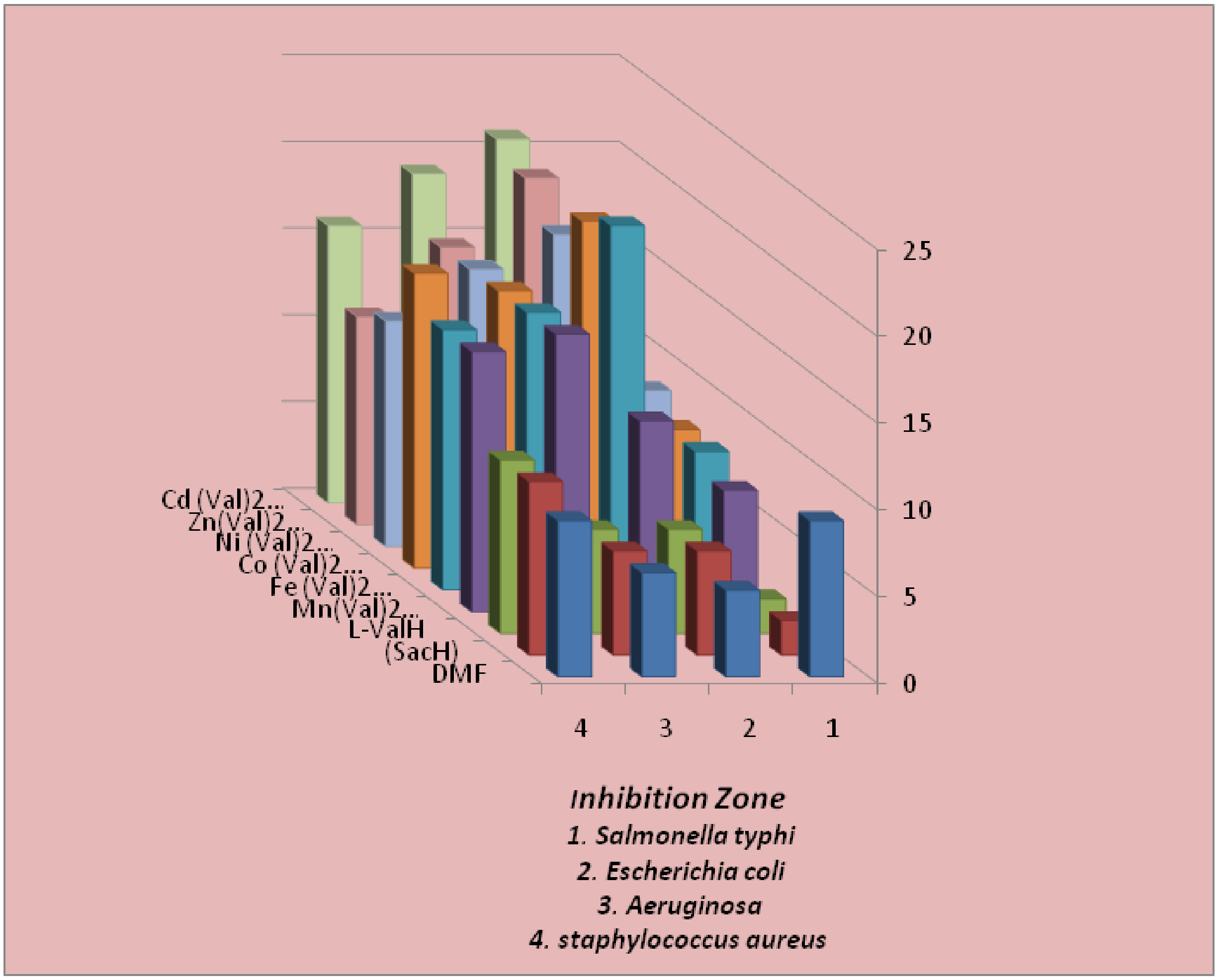
Table (3): FT-IR spectral data of mixed ligand complexes of composition [M(Val)$_2$ (SacH)$_2$

Compounds	**υ (N-H)asy** **υ (N-H)$_{sym}$**	**υ (C - H)alph** **υ (C - H)$_{cy}$**	**υ asy (SO$_2$)** **υ $_{sym}$ (SO$_2$)**	**υ (CNS) asy**	**υ (-COO$^-$)$_{asy}$**	**υ(-COO)$_{sym}$**	**M - N**	**M – O**
Val –H	3120	2978s	-	-	1612 vs	1473 s	-	-
Sac H	3095s	- 2976v s- sh	1298 vs 1178 vs	974m	-	-	-	-
Mn(Val)$_2$ (Sac H)$_2$	3140s	2943s	1256 m 1151 s	918m	1612vs 1570s	1473s 1396vs	667m	540m
Fe (Val)$_2$ (Sac H)$_2$	3394m	2776m	1298 m 1166m	918m	1612m- 1587s	1425s- 1396vs	667m	542m
Co (Val)$_2$ (Sac H)$_2$	3508-3101 vs	2949s	1290vs 1155 s	956s;	1622vs 1583vs	1356vs	673m	536m
Ni (Val)$_2$ (Sac H)$_2$	3336vs	3115m	1288 m 1155m	948m	1622vs 1581vs	1352s	680m	543m
Zn(Val)$_2$ (Sac H)$_2$	3441m 3290vs	2926vs- sh	1292 m 1147s	923 sh m	1616vs 1577vs	1338s	648m	581m
Cd (Val)$_2$ (Sac H)$_2$	3452 7300vs	2978 s sh	1271 m	956m	1627s 1587m	1330 m	636m	553s

S= sharp, v s= very sharp, w= weak, b= broad, vb = very broad

Table 4. Antimicrobial activity of the ligands and metal complexes Against (Staphylococcus aureus (+ve) and (Escherichia coli, Aeruginosa, Salmonella typhi and uginosa) (-ve)

Complexes	Inhibition Zone (mm)			
	Salmonella typhi (-ve)	**Escherichia coli (-ve)**	**Aeruginosa (-ve)**	**Staphylococcus aureus (+ve)**
DMF	9	5	6	9
(SacH)	2	6	6	10
L-Val H	2	6	6	10
Mn(Val)$_2$ (Sac H)$_2$	7	11	16	15
Fe (Val)$_2$ (Sac H)$_2$	8	21	16	15
Co (Val)$_2$ (Sac H)$_2$	8	20	16	17
Ni (Val)$_2$ (Sac H)$_2$	9	18	16	13
Zn(Val)$_2$(Sac H)$_2$	-	20	16	12
Cd (Val)$_2$(Sac H)$_2$	7	21	19	16



Scheme (2) Inhibitory activity of the ligands and metal complexes Against (Staphylococcus aureus (+ve) and Escherichia coli, Aeruginosa, Salmonella typhi and Staphylococcus aureus) (-ve)

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