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New Dinuclear Schiff Base Complexes of Mn(II) Derived from Amino Acids

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ABSTRACT

In this study we describe the synthesis and characterization of four and six coordinated compounds of Mn(II)with Schiff bases derived from vanillin with amino acids (glycine, L-serine, L-tyrosine, and L-phenylalanine).These complexes were characterized by many physicochemical methods such as elemental analysis (CHN),magnetic susceptibility, molar conductance as well as spectral studies such as IR and UV-Visible .The analytical data showed that the complexes having the formulas $[Mn(VA)(H_2O)]_2$,$[Mn_2(VA)(CH_3COO)(H_2O)_2]$ ***,where*** A^-***=serine or tyrosine*** $[Mn_2(Vg)(CH_3COO)(H_2O)_3]$, $[Mn_2(VA^*H_2)_2(H_2O)_2(NO_3)_2](NO_3)_2$ ***and*** $Na_2[Mn_2(VA^*)_2(H_2O)_2(NO_3)_2]$ ***, A* = all the amino acids .The ligands behave as Tetradentate coordinating through the atoms NOOO, or tridentate NOO; where V= vanillin, A=amino acids (glycine and phenylalanine) that suggested tetrahedral and octahedral geometry.***

Keywords: *Schiff base complexes, Mn(II) complexes, Amino Acid complexes.*

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معقدات قواعد شيف ثنائية النوى للمنغنيز الثنائي المشتقة من الاحماض الامينية

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الملخص

تم تحضير معقدات جديدة للمنغنيز الثنائي ذات تناسق رباعي وسداسي لقواعد شيف المشتقة من الفانلين والاحماض الامينية (كلايسين ، ل- سيرين ، ل- تايروسين ، ل- فينل الانين) شخصت المعقدات بالتحليل الدقيق للعناصر والقياسات المغناطيسية ، التوصيلية الكهربائية المولارية والطرق الطيفية منها الأشعة تحت الحمراء والاطياف الالكترونية، لقد كانت الصيغ العامة لها سيرين او تايروسين

$[Mn(VA)(H_2O)]_2$,$[Mn_2(VA^-)(CH_3COO)(H_2O)_2]$,$[Mn_2(Vg)(CH_3COO)(H_2O)_3]$,

A^-= $[Mn_2(V{*}AH_2)_2(H_2O)_2(NO_3)_2](NO_3)_2$,$Na_2[Mn_2(V{*}A)_2(H_2O)_2(NO_3)_2]$, A=glycine or

.phenylalanine , A*=all amino acids

وباستخدام هذه الطرائق وجد بان اليكاندات تتناسق بشكل رباعي السن عن طريق ذرة النتروجين وثلاث ذرات اوكسجين وثلاثي السن عن طريق النتروجين وذرتي الاوكسجين لتعطي تناسق رباعي وسداسي ذات بنية رباعي السطوح وثماني السطوح الاكثر احتمالا

الكلمات الدالة : معقدات قواعد شيف ، معقدات المنغنيز الثنائية، معقدات الاحماض الامينية.

1.INTRODUCTION

Schiff bases constitute an interesting class of chelating agents capable of coordinating metal ions giving complexes which serve as models for biological systems [1-3].Recently , considerable attention has been paid to the chemistry of metal complexes of amino acids of Schiff bases containing oxygen , nitrogen and other donors for physiological reasons [4-6] , since amino acids are absorbed well from intestinal lumen by specific active transport mechanisms [7] , amino acids containing imines display significant biological [8-10] , they easily form stable complexes with most transition metal ions [11-13] . According we report herein , synthesis and characterization of some new complexes of Mn(II) with N,O donor Schiff base derived from vanillin with some amino acids , and they characterized by different chemical, physical and spectral methods .

2.EXPERIMENTAL

2.1.Chemicals

All chemicals and solvents used were of analytical grade .The metal salts were commercially available pure samples. They included manganese (II) chloride, (Aldrich), manganese (II) acetate (Fluka), manganese (II) nitrate (Bisolve), vanillin (B.D.H).

2.2.Physical Methods

1. Melting point and decomposition temperature were determined using SMP30 melting point apparatus.

2. IR spectra measurements were recorded using FTIR-Tensor 27-Burker Co. Germany 2003 as KBr pellets in the range (400-4000 cm^{-1}).

3. UV-visible spectral measurements were done on Shimadzu 1800 spectrophotometry for 10^{-3} M complexes in DMF solvent at room temp.using 1cm quarts cell in range (190-1100) nm.

4. Microanalysis (C, H, N) were performed using Costech Instrumental Elemental Combustion.

5. Molar conductance of complexes were measured at room temp. for 10^{-3}M in DMF using Multiline F-SET-2WTW Wissenschaf Technics Werktattem 82362 Weicheim.

6. Magnetic susceptibility of the complexes were measured by Bruker–BM6.Manganese contents were determined by applying

7. Atomic absorption using Sens AAGB Scientific Equipment, after the decomposition of the complexes with concentrated nitric acid.

2.3.Synthetic Methods

1.Preparation of the Schiff base salts(ligands)

Sodium vanillin amino acids imine: Equal amounts of amino acids 0.01mol (1.05g of L-serine or 1.65g of L-phenylalanine or 0.77g of glycine or 1.81g of L-tyrosine)in 20ml(25% distilled water + 75% ethanol) was mixed with vanillin (1.5g, 0.01mol) in 20ml ethanol in presence of sodium acetate (0.82g ,0.01mol).The mixture was heated at 50 c in water bath for an hour , the mixture was cooled and measured the pH .Then the solution was evaporated about its half volume and left for overnight to complete precipitation, the precipitate was collected by filtration, washed with 1:1 ethanol –water mixture and diethyl ether and it was dried over anhydrous over anhydrous $CaCl_2$ .The analytical data for C.H.N and % yield Table.(1&2).

2.Preparation of the complexes

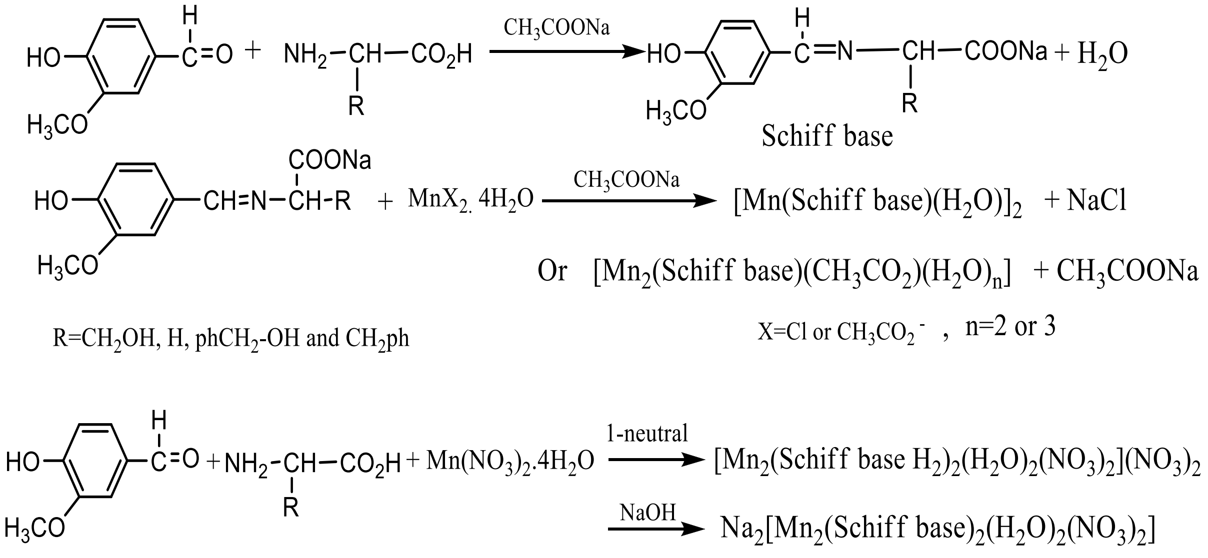
A- Preparation of manganese (II) chloride complexes 0.01mol of Schiff base salt in 20 ml ethanol has been added to 0.01mol manganese (II) chloride in 10ml of hot ethanolic solution ,followed by slow addition of aqueous solution of sodium acetate (0.02 mol). The mixture has been refluxed for half an hour at 50c with stirring, followed by cooling, and measuring the pH, then evaporated to half its volume, cooled, filtered, washed with ether and dried over $CaCl_{2.}$

B- Preparation of manganese (II) acetate By following above procedure in A expect without addition of sodium acetate and refluxing time 2 hours.

C-preparation of manganese (II) nitrate A general procedure has been adopted for the preparation of complexes in neutral and basic medium. In neutral medium, a solution of 0.01mol of each of amino acids and vanillin in20 ml(25% distilled water +75% ethanol) has been added to the solution of manganese (II) nitrate(0.01mol), the mixture has been refluxed for an half hour at 50c with stirring ,followed by cooling and measuring the pH, The product has been filtered off, washed with ether and dried over $CaCl_2$. In basic medium, complexes have been prepared by applying the same amount used in neutral medium, and after mixing the manganese nitrate with ligands and heating on a water bath, sodium hydroxide solution (1M) has been added until pH (9-12), then followed above steps as in neutral medium.

3.RESULTS AND DISCUSSION

The reaction of manganese salts with Schiff bases can be represented by the following reactions:



The solid complexes are coloured, insoluble in water, methanol and ethanol but soluble in DMF at 10^{-3}M Table.(3) revealed that complexes (1-5) are non electrolytic indicating neutral complexes, while complexes (6-13) are1:1 electrolyte in nature for nitrate complexes in neutral medium and in basic medium .The molar conductance values and the metal contents are in a good agreement with given formulations.

4.IR SPECTRA

The coordination sites of the ligands involved in the bonding with metal ions had been determined by careful comparison of the Table.(4&5) infrared spectra of those compounds with that of the parent ligands. The ligands basically composed of different groups of potent ability to coordinate with the metal ions. The IR spectra of the Schiff bases showed a strong band in the region (1672-1650) cm^{-1}, which is characteristic of the azomethine (stretching frequency C=N) group. In all complexes, this band is slightly shifted to lower frequency indicating coordination of the Schiff bases through azomethine nitrogen atom [14]. The IR spectrum of the ligands exhibit a broad band of stretching frequency of phenolic(OH) group at (3369-3443) cm^{-1} (the broadness due to the presence of hydrogen bonding) and a second band at(1371-1300) cm^{-1}due to bending phenolic OH group, these two bands are absent in the complexes due to de protonation on coordination to the metal ion, and shift toward a lower frequency on coordination for complexes(6-9) prepared in neutral medium,.. In addition, the stretching vibration of C-O single band split into two peaks support the above suggested coordination of the hydroxyl group without de protonation[15].

Where as in basic medium, these bands will disappear due to de protonation. Another important strong band observed in the spectrum of the free ligand ascribed to phenolic stretching (C-O)group at (1245-1267)$cm^{-1}$is shifted to lower frequency in all complexes. This is usually indicates that the (C-O)group of the ligand involved incoordination with the metal ion through the deprotonated oxygen of phenolic group[16].The ligands exhibit other two intense bands at (1411-1334) ,(1590-1585)cm^{-1}corresponding to symmetric and asymmetric stretching frequencies of (COOH)group, respectively of the organic ligand and of the acetato group.On complexation symmetric bands were shifted to a higher frequencies or remained unaltered in the position of the ligands, while asymmetric bands were shifted toward a lower frequencies respectively [16]. The difference between the symmetry and asymmetry stretching vibration of COO$^-$group ($\triangle\nu$ which is equal to 150-160cm^{-1}) gave indication about the manner of coordination of carboxylic group, this value showed that amino acid Schiff bases coordinated through COO$^-$group which was acted as monodentate [17]. The acetato complexes exhibited bands differences as bidentate chelating acetato group and confirmed by electronic spectra of the complexes [16].The presence of (COO$^-$) group makes the coordination phenomenon is more complicated due to presence of acetato group belongs to metal. The C-O stretching vibration of the free acetate ion was observed at ca 1600 cm^{-1} and shifted to a lower frequency that is conformity with many authors [17].The IR spectra of nitrato complexes display three (N-O) stretching bands .The infrared data indicated the occurrence of two strong absorption bands in1470-1424 cm^{-1}, 1290-1234 $cm^{-1}$and 950 cm^{-1}regions, which were attributed to(v_5,$v_1$andv_2)modes of vibrations of the covalently bonded nitrate groups, respectively[15].The (v_5-v_1) is taken as an approximate measure of the covalence of nitrate group[15], a value of ~220 $cm^{-1}$for the complexes suggested strong covalence for the metal-nitrate bonding. Authors have shown that the number and relative energies measure of the covalence of nitrate combination frequencies (v_5-v_1) in the infrared spectrum and may be used as an aid to distinguish the various coordination modes of the nitra to group and have suggested that bidentate coordination of the nitrato group involves a greater distortion from D_{3h} symmetry than unidentate coordination, therefore, bidentate nitrate groups should show a larger separation of (v_5-v_1). After an investigation of the spectra of a number of compounds showed that the separation for monodentate nitrate groups appeared to be 115 cm^{-1} and that for bidentate groups 220 cm^{-1}. In the present complexes, a separation of 190-180 cm^{-1}, and the nitro groups seem to be bidentate .On the other hand, the spectra of some complexes showed the presence of a band at (1380-1385) cm^{-1}as due to ionic nature of nitrato group [15]. The aqua complexes contain weak to medium a broad band at (3451-3205) cm^{-1}due to stretching vibration OH of water [18]and a sharp shoulder at (1513-1540) cm^{-1}may be assigned to bending vibration of water. Water molecules are coordinated, confirmed by occurrence of additional strong and sharp band at 813-876cm^{-1}due to OH rocking vibrations [18]. For all complexes new bands were observed at (410-518) and (524-590) cm^{-1}, these bands assigned to the stretching modes of M-N and M-O, respectively [16].The presence of these bands support the formation of the complexes under investigation figures.

5.ELECTRONIC SPECTRA AND MAGNETIC MOMENT

The spectrum of the ligands exhibited two bands in the UV intervals at (40650 -40535)cm^{-1}and(30864 -30756)cm^{-1} , assigned toπ π^* and n π^* transitions respectively.

The electronic spectra of manganese (II) complexes the d-d transitions, doubly forbidden (due to spin and laporte-forbidden) from the term^6 A_{1g} towards, the quartet terms $^4T_{1g}$ (G),$^4A_{2g}$(G), ^4E_g (G) exhibit a very small intensity and are concealed by the intra ligand transitions. The absorption bands appeared due to charge transfer of ligands to metal [19].For complexes (1-5) exhibited bands in region (29411-35714) cm^{-1}.These complexes show magnetic moments in the range (5.03-5.90)B.M. Thus five unpaired electrons are present in the complex molecule. Obviously, the hybridization of Mn(II) ion in the complex is either sp^3 or dsp^3. However, sp^3hybridisation and tetrahedral structures of these complexes are preferred over octahedral structures because all bands in the visible region are above 20000cm^{-1}.It has been observed by several workers [20], that tetrahedral Mn(II) complexes exhibit absorption bands above 20000cm^{-1}and octahedral Mn(II) complexes exhibit at least one band below 20000cm^{-1}. While complexes (6-13) show one a strong band at(13333-14992)cm^{-1}besides above 20000cm^{-1},and have magnetic moments in the range (4.19-4.51) B.M. Bashing on this discussion a low spin octahedral arrangement may be proposed around Mn(II) ion[20].The data are given in Table.(6).

6.CONCLUSION

From the above discussion of various physicochemical and spectral studies, we conclude that the Schiff base ligands of glycine and phenyl alanine coordinated as tridenate through phenoxy oxygen,carboxy oxygen and azomethine nitrogen atoms, while the Schiff base ligands of serine and tyrosine coordinated as tetradenate through phenoxy oxygen,carboxy oxygen,azomethine nitrogen and alcoholic or phenoxy oxygen atoms. The ligands are used as stabilizer for dinuclear metal complexes and according to the measurements and theoretical calculations the Mn(II)chloride or acetate complexes have tetrahedral geometries, while Mn(II)nitrate complexes have octahedral geometries around central metal ion, Figure.(1&2).

Table.(1): Names, Structures and Abbreviations of Schiff Base Ligands

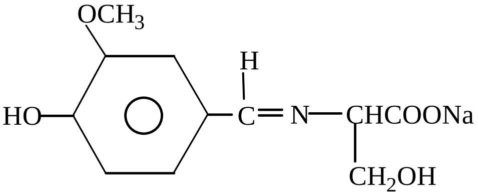
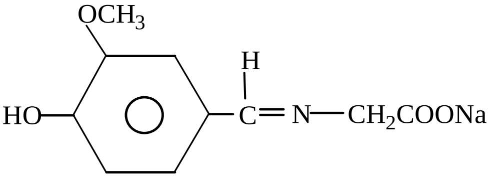
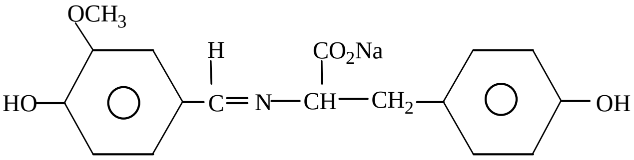
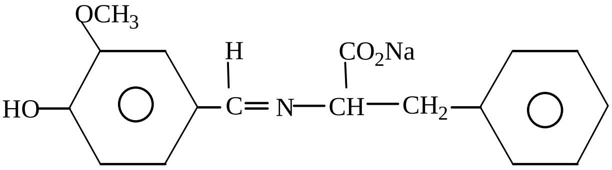
Schiff base compound	Structure	Abbreviation
Sodiumvanillinserine imine		**NaVs**
Sodiumvanillinglycine imine		**NaVg**
Sodiumvanillintyrosine imine		**NaVt**
Sodiumvanillinphenylalanineimine		**NaVphe**

Table.(2): Some physical properties of Schiff Base Ligands

No.	Abbrev.	Chemical formula	Color	m. p c °	Yield (%)	% Analysis ,Calc.(Observed.)		
						C	H	N
1	NaVs	$C_{11}H_{12}$ NO_5Na	Yellow	83	80	50.57 (50.41)	4.59 (4.45)	5.36 (5.22)
2	NaVg	$C_{10}H_{10}$ NO_4Na	Yellow	75	85	51.94 (51.82)	4.32 (4.19)	6.06 (5.90)
3	NaVt	$C_{17}H_{16}$ NO_5Na	Pale Yellow	197	75	60.53 (60.47)	4.74 (4.80)	4..15 (4.02)
4	NaVphe	$C_{17}H_{16}$ NO_4Na	Dark Yellow	112	69	63.35 (63.20)	4.98 (4.80)	4.36 (4.25)

Table.(3): Analytical data and physical properties of the complexes

No.	Complexes	m.p c °	Color	Λ_M	$\mu_{eff.}$	% Analysis Calc. (Observed.)			
						C	H	N	M
1.	$[Mn(Vg)(H_2O)]_2$	251	yellow	27	4.19	42.70 (4.84)	4.27 (4.30)	4.98 (5.10)	19.57 (20.07)
2.	$[Mn(Vphe)(H_2O)]_2$	90	brown	20	4.51	54.98 (55.02)	4.85 (4.96)	3.77 (3.65)	14.82 (14.99)
3.	$[Mn_2(VS)(CH_3CO_2)(H_2O_{)2}]$	105	yellow	12	4.19	35.21 (35.45)	4.28 (4.56)	3.16 (3.32)	24.83 (25.00)
4.	$[Mn_2(VT)(CH_3CO_2)(H_2O)_2]$	271	brown	15	4.23	43.93 (44.11)	4.43 (4.51)	2.69 (2.84)	21.19 (22.12)
5.	$[Mn_2(Vg)(CH_3CO_2)(H_2O)_3]$	75	brown	13	4.43	33.41 (33.25)	4.40 (4.32)	3.24 (3.50)	25.52 (25.78)
6.	$[Mn_2(VgH_2)_2(H_2O)_2(NO_3)_2](NO_3)_2$	133	yellow	123	4.5	29.48 (29.65)	3.43 (3.53)	10.31 (10.11	13.51 (13.66)
7.	$[Mn_{2(V}pheH_2)_2(H_2O)_2(NO_3)_2](NO_3)_2$	123	yellow	125	4.18	41.04 (40.96)	3.62 (3.81)	8.45 (8.63)	11.06 (10.97)
8.	$[Mn_2(VSH_2)_2(H_2O)_2(NO_3)_2](NO_3)_2$	135	brown	115	4.5	30.20 (30.44)	3.20 (3.47)	9.61 (9.82)	12.58 (12.71)
9.	$[Mn_2(VTH_2)_2(H_2O)_2(NO_3)_2](NO_3)_2$	210d	yellow	120	4.5	22.23 (22.42)	3.50 (3.69)	8.18 (8.23)	10.72 (10.88)
10.	$Na_2[Mn_2(Vg)_2(H_2O)_2(NO_3)_2]$	317d	brown	110	4.19	32.78 (32.92)	3.27 (3.34)	7.65 (7.87)	15.02 (14 95)
11.	$Na_2[Mn_2(Vphe)_2(H_2O)_2(NO_3)_2]$	198d	brown	100	4.23	55.73 (55.84)	4.91 (5.15)	7.65 (7.78)	15.02 (15.26)
12	$Na_2[Mn_2(VS)_2(H_2O)_2(NO_3)_2]$	237d	brown	95	4.42	33.34 (33.50)	3.53 (3.63)	7.07 (6.94)	13.88 (14.11)
13	$Na_2[Mn_2(VT)_2(H_2O)_2(NO_3)_2]$	245	brown	85	4.51	43.23 (43.55)	3.81 (4.04)	5.93 (6.12)	11.65 (11.83)

d= decomposition point **A_M= molar conductivities in Ω^{-1} cm^{2}mol^{-1}**

Table.(4): Important IR spectra bands $cm^{-1}$of the ligands

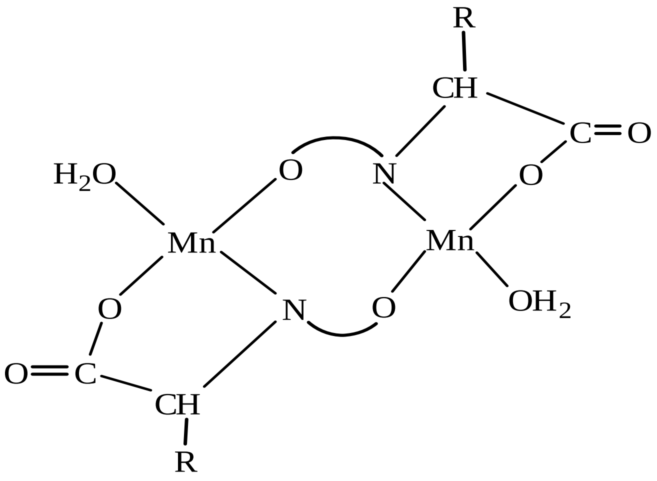
Ligand	νC=N	ν C-O	δO-H	$\nu s(COO)^-$	$\nu as(COO)^-$	νO-H
NaVg	1665	1266	1371	1430	1588	3377
NaVphe	1672	1246	1339	1411	1579	3443
NaVS	1650	1245	1300	1434	1590	3369
NaVT	1665	1267	1338	1429	1585	3440

Table.(5): Important IR spectral bands (cm^{-1}) of the complexes

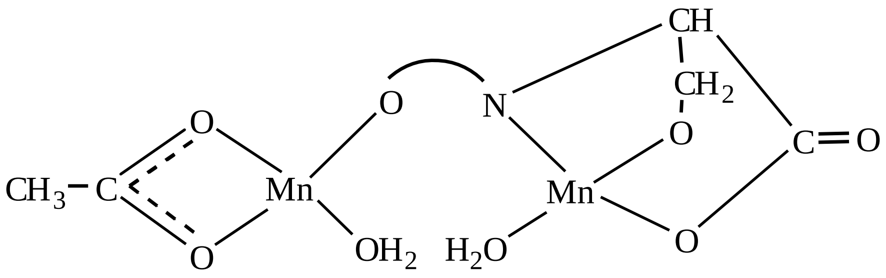
Comp. No	$\nu_{C=N}$	ν C-O	νsCOO	νasCOO	νM-N	νM-O	νOH_2 $R(H_2O)$	Others
1	1578	1229	1481	1543	410	521	3267, 820	-
2	1604	1200	1410	1562	518	589	3269, 858	-
3	1600	1200	1434	1585	514	588	3280, 813	-
4	1609	1214	1436	1585	432	575	3205, 841	-
5	1595	1206	1430	1584	451	535	3425, 865	-
6	1578	1218	1415	1565	502	586	3451, 830	1470, 1290, 950,1380
7	1578	1219	1431	1589	501	571	3238, 866	1424,1234,950,1385
8	1600	1217	1434	1586	490	542	3425, 835	1463,1270,950,1380
9	1578	1216	1431	1581	502	587	3426, 830	1455,1245,950,1385
10	1590	1214	1417	1573	433	529	3306, 841	$\nu_{ionic}NO_3$,1385
11	1561	1225	1410	1582	469	524	3441, 848	$\nu_{ionic}NO_3$1380
12	1607	1220	1434	1580	439	552	3417, 876	$\nu_{ionic}NO_3$,1380
13	1580	1242	1429	1580	416	543	3425, 866	$\nu_{ionic}NO_3$,1385

Table.(6): Electronic Spectral data and geometries of complexes

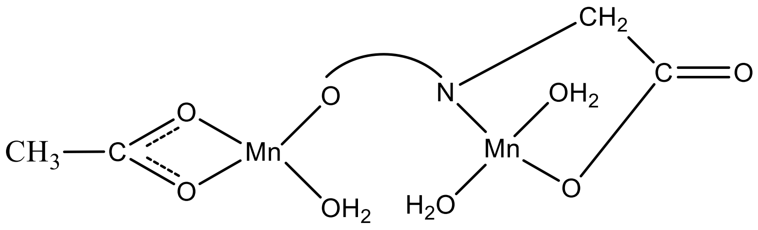
Complex No	Electronic Spectra cm^{-1}	Geometry
1	29587, 31748	Tetrahedral
2	29411, 32679	Tetrahedral
3	326793	Tetrahedral
4	34602	Tetrahedral
5	35714	Tetrahedral
6	13351, 38910	Octahedral
7	13333 , 35087	Octahedral
8	14925 , 32051	Octahedral
9	14880 , 34602	Octahedral
10	13386, 40485	Octahedral
11	13351 , 33333	Octahedral
12	14705 , 42492	Octahedral
13	14992, 40100	Octahedral



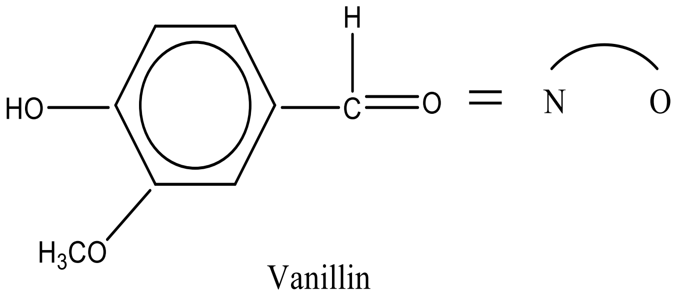
$[Mn(VL)(H_2O)]_2$, L=glycine or tyrosine



$[Mn_2(VE)(CH_3CO_2)(H_2O)_2]$, E= serine or Tyrosine

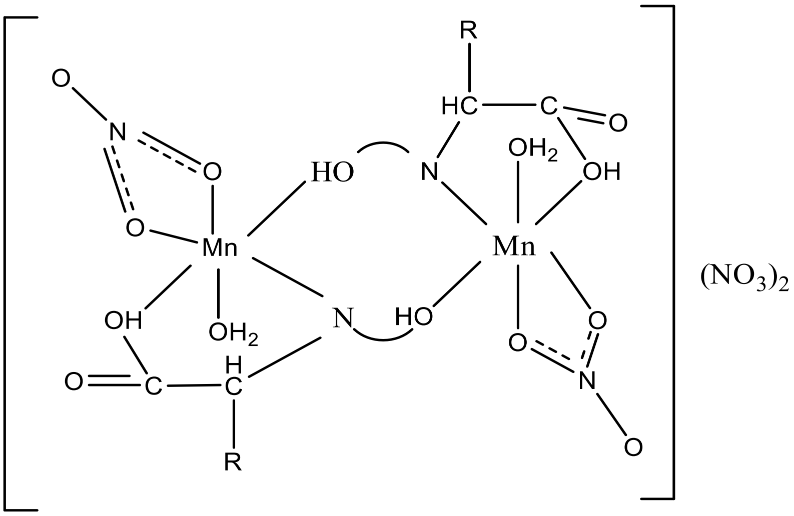


$[Mn_2(Vg)(CH_3CO_2)(H_2O)_3]$

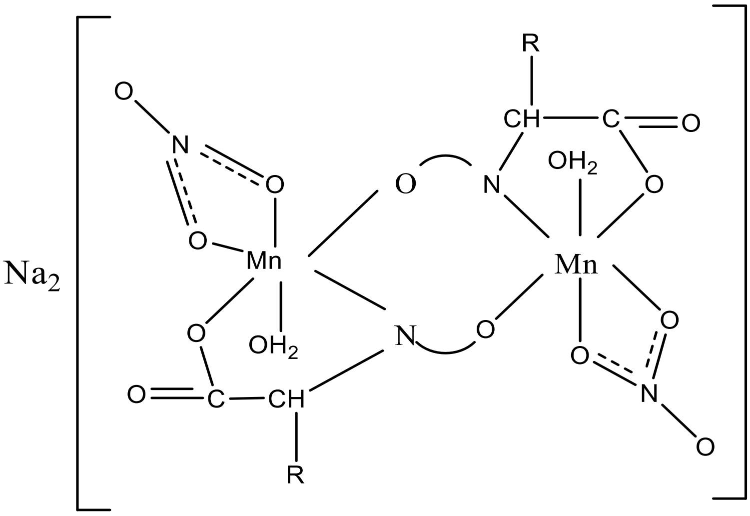


Vanillin

Figure.(1)



$[Mn_2(\text{Schiff base }H_2)(H_2O)_2(NO_3)_2](NO_3)_2$



$Na_2[Mn_2(\text{Schiff base})_2(H_2O)_2(NO_3)_2]$

Figure.(2)

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