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Conductometric Studies

Macrocyclic Schiff Bases Ligands

Highlights

Stable Adducts of TRIS

Manganese Soap Solutions

Discovering Thoughts, Inventing Future

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Conductometric Studies on Manganese Soap Solutions

By Vasu Mitra & S.K. Upadhyay
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Abstract- The conductometric studies of the solution of manganese butyrate and caprylate in a mixture of 50% Benzene and 50% Methanol (v/v) were employed to determine the specific conductance, limiting molar conductance at infinite dilution, degree of dissociation, dissociation constant and CMC. The values of critical micelles concentration decreases with increasing chain length of fatty acid component. The result show that the soaps behaves as a weak electrolytes below the CMC. The thermodynamic parameters for both dissociation and association processes are evaluated.

Keywords: *manganese soaps, CMC, weak electrolytes, conductivity.*

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Conductometric Studies on Manganese Soap Solutions

Vasu Mitra [α] & S.K. Upadhyay [σ]

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I. Introduction

The transition metal soaps as lubricants catalysts, stabilizers and corrosion inhibiting agents has fascinated research workers in the past. The physico-chemical behavior of transition metal soaps has been studied by them. The characteristics and structure of these soaps depend on the method and conditions of preparation. The conductometric investigations are not extended to these metal soaps of still lower fatty acids [11]. Previous communications on interfacial tension, viscosity and conductivity confirmed that aqueous solution of transition metal butyrate could form micelles. The excellent solubility of transition metal butyrate in a 50% methanol and 50% benzene mixture (v/v) has necessitated this study on the conductance and micellar behaviour of these compounds in this mixture and different temperatures.

II. Experimental

All the chemicals used were BDH/AR grade. Chromium soaps (butyrate and caprylate) were prepared by direct metathesis of the corresponding potassium soap with slight-excess of the solution of manganese chloride at 50-55°c under vigorous stirring [8-10]. The precipitated soaps were washed several times with water and acetone. The metal soaps thus obtained were first dried in an air oven at 50-60°c and the final drying of the soaps was carried out under reduced pressure. The soaps were purified by recrystallization with Benzene-methanol mixture. The purity of the soaps was confirmed by the determination of melting points.

A digital conductivity meter (Toshniwal CL01.10A) and a dipping type conductivity cell with Platinized electrodes (cell constant =0.90) were used for measuring the conductance of the soap solutions. All measurements were made at 35-50°c (±0.05°c). The specific and molar conductance were expressed in Ω^{-1} cm^{-1} and $\Omega^{-1}cm^2$ g^{-1} mol^{-1} respectively.

Table 1 : Conductivity of Manganese Butyrate in Mixture of 50% Benzene and 50% Methanol at 40±0.05°C.

S.No.	Concentration $C\times10^2$	Specific conductance $\kappa\times10^6$	Molar conductance 'μ'	1/μ	$\mu^2c^2\times10^4$ 'α'	Degree of dissociation	Dissociation constant $K\times10^4$
1.	1.00	17.0	1.70	0.58	2.89	0.620	2.509
2.	1.18	17.6	1.49	0.67	3.09	0.544	2.000
3.	1.43	18.9	1.32	0.75	3.56	0.482	1.768
4.	1.54	19.8	1.28	0.78	3.88	0.467	1.810
5.	1.65	20.7	1.25	0.80	4.25	0.456	1.898
6.	1.91	22.3	1.16	0.86	4.90	0.423	1.914
7.	2.07	23.6	1.14	0.87	5.56	0.416	2.112
8.	2.26	24.8	1.09	0.91	6.06	0.398	2.139
9.	2.49	26.2	1.05	0.95	6.83	0.383	2.258
10.	2.77	28.0	1.01	0.99	7.82	0.369	2.444
11.	3.12	30.9	0.99	1.01	9.54	0.361	2.866

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12.	3.57	33.8	0.94	1.06	11.26	0.343	3.131
13.	4.16	37.3	0.89	1.12	13.70	0.325	3.520
14.	4.54	39.7	0.87	1.51	15.60	0.318	3.887
15.	4.99	42.3	0.84	1.19	17.56	0.306	4.112
16.	5.55	45.2	0.81	1.23	20.20	0.296	4.538
17.	6.24	46.1	0.73	1.36	20.74	0.266	3.993
18.	7.14	48.4	0.67	1.49	22.88	0.244	3.918
19.	8.33	53.2	0.63	1.58	27.54	0.230	4.385
20.	10.00	60.6	0.60	1.66	36.00	0.219	5.379

Table 2 : Conductivity of Manganese Caprylate in Mixture of 50% Benzene and 50% Methanol at 40±0.05°C.

S.No.	Concentration $C\times10^{2}$	Specific conductance	Molar conductance	1/μ	$\mu^{2}c^{2}\times10^{4}$	Degree of dissociation	Dissociation constant
	$\kappa\times10^{6}$	'μ'			'α'		$K\times10^{4}$
1.	1.00	13.5	1.35	0.74	1.82	0.600	2.160
2.	1.18	14.6	1.23	0.81	2.10	0.546	1.996
3.	1.43	15.8	1.10	0.90	2.47	0.493	1.933
4.	1.54	16.5	1.07	0.93	2.71	0.475	1.936
5.	1.65	17.2	1.04	0.96	3.94	0.462	1.996
6.	1.91	18.7	0.97	1.03	3.43	0.431	2.053
7.	2.07	19.6	0.94	1.06	3.78	0.417	2.349
8.	2.26	20.8	0.92	1.08	4.36	0.409	2.398
9.	2.49	22.0	0.88	1.13	4.80	0.391	2.434
10.	2.77	23.6	0.85	1.17	5.41	0.378	2.665
11.	3.12	25.4	0.81	1.23	6.38	0.360	2.838
12.	3.57	28.0	0.78	1.28	7.75	0.347	3.262
13.	4.16	31.1	0.74	1.35	9.47	0.329	3.673
14.	4.54	32.3	0.71	1.40	10.39	0.316	3.800
15.	4.99	34.4	0.68	1.47	11.51	0.302	3.930
16.	5.55	37.0	0.66	1.51	13.41	0.293	4.383
17.	6.24	39.9	0.63	1.58	15.45	0.280	4.748
18.	7.14	43.2	0.60	1.66	18.35	0.267	5.295
19.	8.33	48.2	0.57	1.75	22.54	0.253	6.017
20.	10.00	53.3	0.53	1.88	28.09	0.236	6.881

Table 3 : Values of CMC, μ_0 and K of Manganese Soaps

S.N.	Soap (g mole l^{-1})	CMC	μ_0	$K \times 10^{5}$
1.	Manganese butyrate	0.043	2.74	17.20
2.	Manganese caprylate	0.041	2.25	16.50

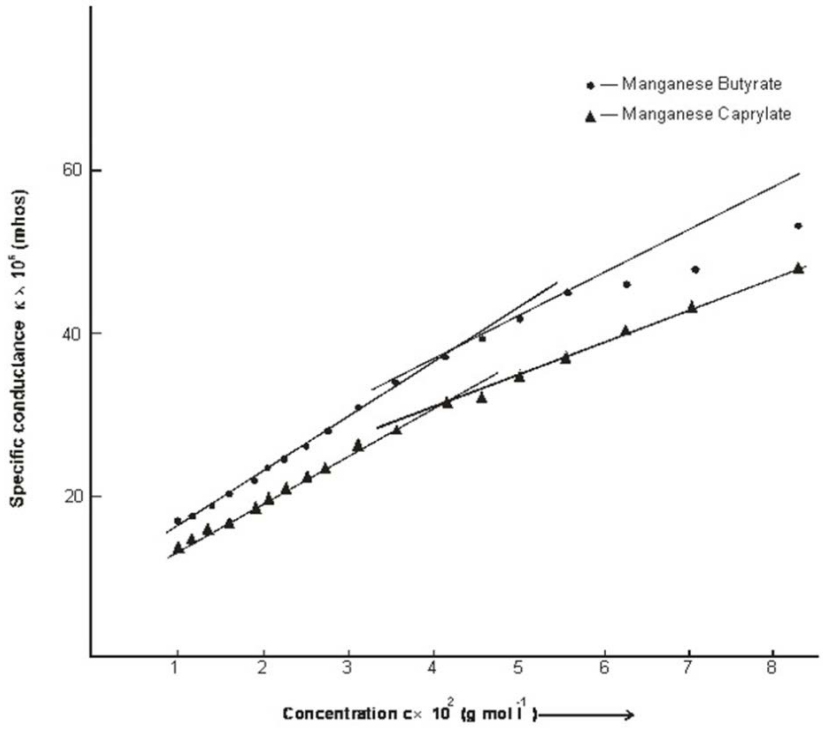

Figure 1 : Specific Conductance Vs Concentration

III. Results and Discussion

a) Specific Conductance

The specific conductance, k, of the solutions of manganese soaps (butyrate and caprylate) in 50% benzene and 50% Methanol increases with increasing soap concentration and decreasing chain length of fatty acid constituent of the soap molecule (Table 1 and 2). The increase in the specific conductance may be due to the ionization of manganese soaps into simple metal cations, Mn^{+2} and fatty acid anions $RC00^{-}$ (where R is C_3H_7 and C_7H_{15} for butyrate and caprylate respectively) in dilute solutions and due to the formation of micelles of higher soap concentrations. The decrease in specific conductance with increasing chain length of soap may be due to the increasing size and decreasing mobility of anions with increasing chain length of soap. The plots of specific conductance Vs. soap concentrations (Fig.1) are characterized by an intersection of two straight lines at a concentration which corresponds to the CMC of manganese soaps (butyrate: 0.043M and caprylate .041M, respectively). It is suggested that these soaps are considerably ionized in dilute solutions and the anions begin to aggregate to form ionic micelles at CMC.

b) Molar Conductance and Ionization Constant

The molar conductance, μ of the solutions of chromium soaps in 50% methanol and 50% benzene (v/v) decreases with increasing soap concentration as well as chain-length of the soap. The decrease is attributed to the combined effects of ionic atmosphere, solvation of ions and decrease of mobility and the formation of micelles. The plots of molar conductance μ vs. Square root of soap concentration, $C^{1/2}$, are not linear which indicate that the soaps behave as weak electrolyte in dilute solutions. The limiting molar conductance, μ_0, of these soap solutions cannot be obtained by usual extrapolation method and the Debye-Huckel-Onsager equation is not applicable to these soap solutions [12-13].

The molar conductance results show that the dilute solutions of manganese soaps behave as weak electrolyte. Since the number of ions for a weak electrolyte is relatively small in dilute solutions and the interionic effects are negligible and so the activities of ions may be taken as almost equal to the concentrations and conductance ratio, μ / μ_0 is a reasonably good measure for the degree of ionization α, (where μ is the molar conductance at finite concentration and μ_0 is the molar conductance at infinite dilution). On substituting the value of α in the equation of ionization constant for 1:3 electrolyte one gets

The values of the ionization constant K and μ_0 can be obtained from the slope and intercept of the line as plots of μ^3c^3 vs. $1/\mu$ for dilute soap solution. The values of limiting molar conductance, μ_0, are 2.74 and 2.25 where as the ionisation constants are 17.20 and 16.50 for manganese butyrate and caprylate, respectively.

The values of degree of ionization, α, at different concentrations have been calculated by assuming that

they are equal to the conductance ratio, μ/μ_0 (Tables 1 and 2). The values of degree of ionization, α, show that the solutions of manganese soaps behave as weak electrolyte. The plots of the degree of ionization vs. soap concentration show that the degree of ionization of manganese soaps decreases rapidly with the soap concentration in dilute solutions where as it decreases slowly in concentrated solutions.

The values of ionization constant, K (Table 1 and 2) again confirm that these soaps behave as a weak electrolyte in solutions. The ionization constant exhibits a drift with increasing soap concentration which may be due to the fact that the conductance ratio, μ/μ_0 is not exactly equal to the degree of ionization α and the activity coefficients of ions are not exactly equal to unity and the failure of the simple Debye-Huckel activity equation under these conditions.

The results show that the soap behaves as a weak electrolyte in dilute solutions below the CMC.

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Preparation, Characterisation and Reactions of Stable Adducts of Tris (Pentafluorophenyl) Antimony (V) Diisothiocyanates

By Ram Nath Prasad Yadav

Tribhuvan Uiniversity, Nepal

Abstract- A series of new stable adducts of tris (pentafluo-rophenyl)antimony(V) diisothiocyante of the type $(C_6H_5)_3$ $Sb(NCS)_2$.L. Where, L = dipropyl formamide (DPF), 3-methylpyridine(3-Picoline), dimethyl formamide (DMF), tryphenylphosphine oxide(Ph_3PO), triphenylarsine oxide (Ph_3AsO), dimethyl sulfoxide (DMSO), thiourea (TU), pyridine(C_5H_5N) have been synthesized by the reaction of tris (pentafluorophenyl)antimony(V) diisothiocyanate with desired ligand in anhydrous methanol. Tris(pentafluorophenyl) antimony diisothiocyanate was obtained by the metathetical reaction of tris(pentafluorophenyl) antimony (V) dichloride and potassium thiocyanate in anhydrous methanol. The molecular weight measurement and molar conductance data of the complexes revealed them to be monomeric and non-conducting in nature. Elemental analysis and IR data indicates that the complexes have hexacoordinated octahedral structure.

Keywords: tris(pentafluorophenyl) antimony, stable adduct, diisothiocyanate, elemental analysis, IR spectra, hexacoordinate, octahedral.

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Ram Nath Prasad Yadav

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Keywords: tris(pentafluorophenyl) antimony, stable adduct, diisothiocyanate, elemental analysis, IR spectra, hexacoordinate, octahedral.

I. Introduction

The Lewis acidity of pentavalent organoantimony compounds, R_nSbX_{5-n} has extensively been investigated in the last two decades by various groups of workers. The studies are not confined to R_2SbCl_3 and $RSbCl_4$ having more chlorine content but has been extended to R_3SbCl_2 derivatives as well. The latter class of compounds, based on hydrocarbon ligands are not good acceptors, but the introduction of CF_3 and C_6F_5 groups on to the metal atom (Sb) considerably enhances the Lewis acidity as evident by the formation of hexacoordinate complexes of the type $(CF_3)_3SbCl_2.L$ and $(C_6F_5)_3SbCl_2.L$. The synthesis and stereochemistry of tris(pentafluorophenyl)antimony(V) dichloride with a number of ligands viz. Dimethyl formamide, diphenyl formamide, triphenylphosphine oxide, arsine oxide, pyridine, picolines, thiourea. etc. has been reported(Singhal et al. 2002). An octahedral environment around antimony has tentatively been proposed for such complexes. On the basis of analytical, and spectroscopic data, it may be noted that except for a single reference on the formation and characterisation of $(C_6F_5)_3SbCl_2.L$, no other study related to the synthesis of molecular adducts has been reported to date (Agarwal 1990).

In view of our interest in the chemistry of pentafluorophenylantimony (III and V) derivatives (Yadav 2012[a], Yadav 2013[a], Yadav 2013[b] and Yadav 2014) and various other aspects of fluorocarbon based organoantimony compounds including their antimicrobial and antitumour activity (Yadav 2012[b] and Yadav 2013[c]), coupled with the paucity of published data in the field, we have synthesised a series of neutral adducts of tris (pentafluorophenyl) antimony (V) diisothiocynate, $(C_6F_5)_3Sb(NCS)_2$, with oxygen, nitrogen and sulphur donor Lewis bases. A few complexes of $(C_6F_5)_3SbCl_2$ have also been synthesised for the sake of comparison. The results of this investigation are reported in this paper.

II. Result and Discussion

Tris (pentafluorophenyl) antimony (v) diisothiocyanate obtained by the metathetical reaction of $(C_6F_3)_3SbCl_2$ and potassium thiocyanate, recrystallized and dried before use, was treated with the desired ligand in equivalent molar ratio in anhydrous methanol. The reactions were carried out under anhydrous oxygen free conditions.

$$(C_6F_5)_3SbCl_2 + 2KNCS \xrightarrow{\text{Methanol}} (C_6F_5)_3Sb(NCS)_2 \ldots\ldots\ldots \quad (1)$$

$$(C_6F_5)_3Sb(NCS)_2 + L \xrightarrow{\text{Methanol}} (C_6F_5)_3Sb(NCS)_2.L \ldots\ldots\ldots\ldots \quad (2)$$

Where, L = DPF, 3-Picoline, DMF, Ph_3PO, Ph_3AsO, DMSO, TU, C_5H_5N

All the reactions were found to proceed smoothly under mild conditions. The completion of the reaction takes place within 3 hrs. In most of the cases products were obtained as solid after evaporating the solvent which were crystallized with petroleum ether (40-60°C) or the mixture of diethyl ether and petroleum ether (60-80°). The complexes are soluble in common organic solvents such as chloroform, acetonitrile etc. They show monomeric constitution in freezing benzene. The complexes are

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stable, non-susceptible to oxygen and can be stored for several weeks without decomposition. The constancy in melting point after repeated crystallization as well as TLC run in polar solvent with a single spot excluded the presence of mixture of reactants. Elemental analysis, conductance and molecular weight data are given in table (2 & 3) and correspond well to the proposed formulation of the complexes. The observed values of molecular weight indicate their monomeric constitution while the values of molar conductance of 10^{-3}M solution in acetonitrile ranges between 20-30 Ohm^{-1} cm^2 mol^{-1} at room temperature (30°C) which shows the absence of ionic species in solution.

a) Infrared Spectra

All the complexes, listed in Table 1, were characterised in the solid state by their infrared spectra in the region 4000-200 cm^{-1}. Important IR frequencies for the complexes together with their assignments are listed in Table 4. These assignments have been made by comparing the spectra in 4000-200 cm^{-1} region in the solid state of the complexes with those of the free ligands. The infrared absorptions due to pentafluorophenyl groups bonded to antimony are almost identical and do not differ significantly from those observed for other pentafluorophenyl antimony compounds reported earlier from this laboratory (Premraj et al. 1989).

b) Infrared Spectra of the Adducts with Oxygen Donors

The ν(C=O) modes in various amides bases appearing at 1650 ± 15 cm^{-1} undergo negative shift and are identified at 1608 ± 10 cm^{-1} in the spectra of the adducts suggesting weakening of the C=O bond and coordination through the oxygen atom of the base. On the basis of - Δν(CO), the DPF was found better donors as compared to DMF (Premraj & Mishra 1991).

An absorption of strong intensity for ν(S=O), ν'(AS=O) and ν(P=O) lying at 1045, 880 and 1195 cm^{-1} respectively, in the spectra of the free ligands undergoes a distinct negative shift on complexation. The corresponding absorption in the spectra of the adducts appears at 940, 835 and 1162 cm^{-1} suggesting coordination from oxygen atom of the base. The relative donor abilities of the ligand as apparent from the value of - Δν(C=O), follow the sequence DMSO > Ph_3AsO > Ph_3PO. On the basis of present and some previous studies a medium strong band in the region 380-410 cm^{-1} is assigned to ν(Sb-O) stretching frequency (Premraj & Mishra 1991).

c) Infrared Spectra of the Adducts with Nitrogen Donors

The ν(CN) frequency in $(C_6F_5)_3SbCl_2Py$ and $(C_6F_5)SbCl_2$.3-Pic is seen to decrease significantly to 1610 ± 5 cm^{-1}. In addition to this a band at 3310±10 cm^{-1} assignable to ν(NH) mode in free ligand is shifted to slightly lower frequency 3010 ± 20 cm^{-1} (Premraj & Mishra 1991). In the IR spectra of the ligand the assignment of the Sb-N bond is tentatively assigned at about 385 ± 5 cm^{-1}.

d) Infrared Spectra of the Adducts with Sulphur Donor

In sulphur donor ligand (TU) an absorption at 1069 cm^{-1} reported to posses equal contribution from ν (CN) and ν(CS). This remains unaffected on adduct formation and appears at 1075 cm^{-1}. When coordination occurs through sulphur atom, the ν(CN) suffers a positive shift while the ν(CS) suffers an almost equal negative shift. As a consequence to this the resulting absorption remains apparently unchanged (Premraj & Mishra 1991). The positive shift of ν(NH) from 3360 cm^{-1} and 3300 cm^{-1} in free ligand to 3410 and 3370 cm^{-1} in its adduct indicates absence of coordination through N-atom of the ligand and indirectly suggest Sb ← S bonding. However, on the basis of some previous observation and present studies, the (Sb-S) bond is assigned at 380 cm^{-1} (Premraj & Mishra 1991).

The diagnostic frequencies due to NCS group bound to antimony appear around at 2080, 840 and 475 cm^{-1} which could be attributed to asymmetric (NCS), symmetric (C-S) and bonding mode δ NCS, respectively. The pattern and intensity does not show any significant change reported earlier for $(C_6F_5)_3Sb(NCS)_2$ compounds (Premraj et al. 1985). Sb–C bond appears in the range 445-465 cm^{-1} (Hall & Sowerby 1988 and Nunn et al. 1996).

e) Stereochemistry of the Neutral Molecular Adducts $(Rf)_3Sb(NCS)_2.L$

It has been assumed that the addition of a Lewis base. L, to the central atom in a trigonal bipyramidal molecule takes place in a trigonal plane and steric and electrostatic factor play an important role in determining the position of entry of L. It is well established that the more electronegative group goes to the axial position and less electronegative on equatorial positions. Therefore, base L should settle in the equatorial position. It is also supported by a tentative assignment of Sb-N band at 326 cm^{-1} appearing in all the spectra and attributed to the NCS present in the axial positions.

In view of the above idea the nucleophilic attack at the position between the two fluoro groups to produce structure (Meinema & Noltes 1976) appears to be most favourable, since Rf is less electronegative than any halogen atom directly bonded to metal.

Thus analytical, conductance measurement, molecular weight determination and IR data clearly indicates that the newly synthesized complexes have hexacoordination environment around antimony with octahedral configuration as has been suggested for $R_2SbCl_3.L$ complexes (Premraj & Mishra 1991). It is generally accepted that the tris(pentafluorophenyl)-antimony(V) diisothiocyanate have a geometry of a

trigonal bipyramidal with two halogen atoms occupying apical positions. In adduct formation as indicated, antimony atom increases its coordination number to six, for our hexacoordinate complexes. A tentative assignments of octahedral structure may be represented as below.

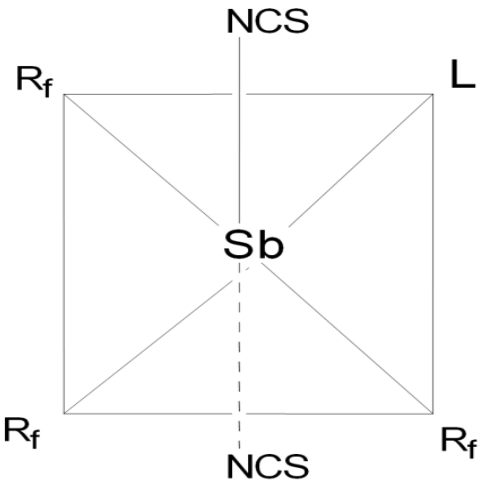

Figure : Suggested structure of $(R_f)_3Sb(NCS)_2L$

R_f = C_6F_5 and L = DPF, 3-Picoline, DMF, Ph_3PO, Ph_3AsO, DMSO, TU, C_5H_5N

III. Experimental

Tris (pentafluorophenyl) antimony (v) dichloride was prepared by passing chlorine gas into the solution of tris(pentafluorophenyl) antimony in pet-ether and tris (pentafluorophenyl) antimony(V) diisothiocyanate was prepared by the metathesis of tris(pentafluorophenyl)-antimony(V) dichloride and potassium thiocyanate. All the ligands were of reagent grade and used without further purification. All solvents were purified and dried by standard procedures (Vogel 1971).

The molar conductance of 10 — 3M solutions was determined at 25°C with a PR-9500 Philips conductivity assembly. Molecular weights were determined cryoscopically in benzene using a Beckmann thermometer of ±0.01°C accuracy. The stoichiometry of the compounds was established by elemental analysis. Percentage of C, H and N of the compounds was obtained on a semi-microscale (using elemental analysiser Carlo Eaba 1106, Thomas CH and Coleman analyser.

Details of the typical representative experiments are described below, the analytical data, molar conductance and IR assignments are summarised in Table 1- 4.

a) Reaction of $(C_6F_5)_3Sb(NCS)_2$ with DMF Ligand (5)

In an oxygen free atomsphere a solution of tris(pentafluorophenyl) antimony(V) diisothiocyanate (0.7388 g, 0.5 mmol) in methanol (25 ml) and DMF (0.0731 g, 0.5 mmol) in the same solvent (25 ml) were stirred together at 80°C for 3 h. After that it was filtered off. The filtrate on concentration in vacuo yielded a white crystalline solid and was recrystallised from petroleum ether (40-60°C) to afford tris(pentafluorophenyl) antimony diisothiocyanate dimethyl formamide adduct $(C_6F_5)_3Sb(NCS)_2$.DMF. M.P.: 197°C, Yield: 0.536 g, (66%).

b) Reaction of $(C_6F_5)_3Sb(NCS)_2$ with Ph_3PO Ligand (6)

A solution of tris(pentafluorophenyl)antimony(V) diisothiocyanate (0.7388 g, 0.5 mmol) in methanol (25 ml) and a solution of the same solvent (25 ml) of triphenyl phosphine oxide (0.278 g, 0.5 mmol) were stirred together at 80°C for 3 h under nitrogen. It was filtered off and the filtrate on concentration in vacuo afforded a light brown solid and was recrystallised from solvent ether to give tris(pentafluorophenyl)- antimony-diisothiocyanate triphenylphosphine oxide adduct. M.P.: 200°C Yield: 0.569g, (56%).

Table 1 : Preparation and Properties of Stable Adducts of Tris (Pentafluorophenyl)Antimony(V) Diisothiocyanates

S. No.	Complex $(R_f)_3Sb(NCS)_2$.L.	Molar Ratio	Ligand (g) Solvent (ml)	$(R_f)_3Sb(NCS)_2$(g) Solvent (ml)	M.P. (°C)	Colour	Recrystallisation solvents
	R_f=C_6F_5						
1.	$(R_f)_3Sb(NCS)_2$.DPF	1:1	DPF (0.129) MeOH (25)	0.7388 MeOH (25)	195	White	Petroleum ether (40-60°C)
2.	$(R_f)_3Sb(NCS)_2$.α-Pic	1:1	α-Pic (0.073) MeOH (25)	0.7388 MeOH (25)	204	Light brown	Petroleum ether (40-60°C)
3.	$(R_f)_3Sb(NCS)_2$.β-Pic	1:1	β-Pic (0.073) MeOH (25)	0.7388 MeOH (25)	170	Light brown	Petroleum ether (40-60°C)
4.	$(R_f)_3Sb(NCS)_2$.γ-Pic	1:1	γ-Pic (0.073) MeOH (25)	0.7388 MeOH (25)	188	Light brown	Petroleum ether (40-60°C)
5.	$(R_f)_3Sb(NCS)_2$.DMF	1:1	DMF (0.073) MeOH (25)	0.7388 MeOH (25)	197	White	Petroleum ether (40-60°C)
6.	$(R_f)_3Sb(NCS)_2.Ph_3PO$	1:1	Ph_3PO (0.278) MeOH (25)	0.7388 MeOH (25)	200	Light brown	Petroleum ether (40-60°C)
7.	$(R_f)_3Sb(NCS)_2.Ph_3AsO$	1:1	Ph_3AsO (0.322) MeOH (25)	0.7388 MeOH (25)	165	White	Petroleum ether (40-60°C)

8.	$(R_f)_3Sb(NCS)_2$.DMSO	1:1	DMSO (0.078) MeOH (25)	0.7388 MeOH (25)	180	White	Petroleum ether (40-60°C)
9.	$(R_f)_3Sb(NCS)_2$.TU	1:1	TU (0.076) MeOH (25)	0.7388 MeOH (25)	183	Off white	Petroleum ether (40-60°C)
10.	$(R_f)_3Sb(NCS)_2$.Py.	1:1	Py. (0.079) Methanol (25)	0.7388 MeOH (25)	207	Light brown	Petroleum ether (40-60°C)

Table 2 : Elemental Analysis of Stable Adducts of Tris (Pentafluorophenyl) Antimony (V) Diisothiocyanates

S.No.	Complex	Empirical formula	Found (Calcd) (%)		
			C	H	N
1.	$(C_6F_5)_3Sb(NCS)_2.(C_3H_7)_2HCON$	$C_{27}H_{15}F_{15}N_3OS_2Sb$	37.34 (37.39)	1.73 (1.79)	4.84 (4.88)
2.	$(C_6F_5)_3Sb(NCS)_2.C_6H_7N$	$C_{26}H_7F_{15}N_3S_2Sb$	37.51 (37.60)	0.84 (0.90)	5.05 (5.08)
3.	$(C_6F_5)_3Sb(NCS)_2.C_6H_7N$	$C_{26}H_7F_{15}N_3S_2Sb$	37.51 (37.60)	0.84 (0.90)	5.05 (5.08)
4.	$(C_6F_5)_3Sb(NCS)_2C_6H_7N$	$C_{26}H_7F_{15}N_3S_2Sb$	37.51 (37.60)	0.84 (0.90)	5.05 (5.08)
5.	$(C_6F_5)_3Sb(NCS)_2.HCON(CH_3)_2$	$C_{23}H_7F_{15}N_3OS_2Sb$	34.00 (34.15)	0.86 (0.88)	5.17 (5.20)
6.	$(C_6F_5)_3Sb(NCS)_2.(C_6H_5)_3PO$	$C_{38}H_{15}F_{15}N_2OS_2Sb$	44.85 (44.92)	1.48 (1.52)	2.75 (2.78)
7.	$(C_6F_5)_3sSb(NCS)_2.(C_6H_5)AsO$	$C_{38}H_{15}F_{15}N_2OS_2AsSb$	42.99 (43.02)	1.41 (1.45)	2.64 (2.70)
8.	$(C_6F_5)_3Sb(NCS)_2.(CH_3)_2SO$	$C_{22}H_6F_{15}N_2OS_3Sb$	32.32 (32.37)	0.73 (0.78)	3.43 (3.46)
9.	$(C_6F_5)_3Sb(NCS)_2.NH_2CSNH_2$	$C_{21}H_4F_{15}N_4S_3Sb$	30.93 (30.98)	0.49 (0.54)	6.87 (6.89)
10.	$(C_6F_5)_3Sb$ $(NCS)_2.C_5H_5N$	$C_{25}H_5F_{15}N_3S_2Sb$	36.69 (36.72)	0.61 (0.68)	5.14 (5.22)

Table 3 : Molecular weight, conductance measurement and yield of stable adducts of Tris(pentafluorophenyl)antimony(V) diisothiocyanates

S. No.	Complex	Molar conductance (Ohm^{-1} cm^2 mol^{-1}) acetonitrile	Molecular weight in nitrobenzene Found (calcd).	Yield	
				g	%
1.	$(C_6F_5)_3Sb(NCS)_2.HCON(C_3H_7)_2$	20.6	865.70 (867.75)	0.555	64
2.	$(C_6F_5)_3Sb(NCS)_2.\alpha$-$C_6H_7N$	22.2	825.75 (831.75)	0.574	69
3.	$(C_6F_5)_3Sb(NCS)_2.\beta$-$C_6H_7N$	22.3	825.75 (831.75)	0.582	70
4.	$(C_6F_5)_3Sb(NCS)_2.\gamma$-$C_6H_7N$	22.7	825.75 (831.75)	0.590	71
5.	$(C_6F_5)_3Sb(NCS)_2.HCON(CH_3)_2$	28.9	805.10 (811.75)	0.536	66
6.	$(C_6F_5)_3Sb(NCS)_2.(C_6H_5)_3PO$	25.2	1014.75 (1016.75)	0.569	56
7.	$(C_6F_5)_3Sb(NCS)_2.(C_6H_5)_3AsO$	24.4	1069.60 (1060.67)	0.605	57
8.	$(C_6F_5)_3Sb(NCS)_2.(CH_3)_2SO$	27.6	812.75 (816.75)	0.564	69
9.	$(C_6F_5)_3Sb(NCS)_2.NH_2CSNH_2$	30.8	813.75 (814.75)	0.578	71
10.	(C_6F_5) Sb $(NCS)_2.C_5H_5N$	29.9	815 (817.75)	0.589	72

Table 4 : IR Spectra for $(R_f)_3Sb(NCS)_2L$ (Cm^{-1})

Compd. No. (Adduct)	ν(Sb-C)	ν(Sb-S)/(Sb-O)/ (Sb-N)	ν(C=N)/ (S=O)/ ν(P-O)/(N-H)/(As-O) ligand (complex)
1	458 ms	385ms	1660 (1612)
2	461 ms	382	1615
3	458 ms	384 w	1612
4	445 ms	320 w	1610
5	447 ms	395 ms	1660 (1615)
6	465 ms	405 ms	1195 (1162)
7	459 ms	390 ms	880 (835)
8	450 ms	380 ms	1045 (940)
9	455 ms	382 w	3300 (3368)
10	459 ms	381 w	1612

ms = medium strong, w = weak

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Synthesis Characterization and Antibacterial Activity of New Open and Macrocyclic Schiff Bases Ligands

By Hamid Hussein Eissa

University of Hajah, Yemen

Abstract- In this work we focused on the synthesis of two new macrocyclic Schiff bases: (III) , (IV), (VI) containing nitrogen – oxygen donor atoms were synthesized by condensation of intermediate compounds: 1,6- bis (2- formylphenel) hexane (I)and α,α'-bis(2-carboxyaldehyde phenoxy) xylene(II) with 4,4'-Diamino-diphenylmethane and 4-Aminophenyl sulfone. Also new open Schiff bases (V) which were prepared by condensation of benzylhydrazidewith 1,6- bis (2- formylphenel) hexane (I). Identification of these macrocyclic Schiff bases: 1,16-di aza-3,4,13,14-tri phenyl-17,25 -di phenyl methane-5,12-di oxacyclo penta-icozane-1,15-diene(III), 1,16-di aza-3,4,7,10,13,14-tri phenyl-17,25 -di phenyl methan-5,8-di oxacyclo penta-icozane-1,15-diene. (IV).1,16-di aza-3,4,13,14-tri phenyl-17,25 -di phenyl sulphide-5,12-di oxacyclo penta-icozane-1,15-diene(V),and N',N'-(2,2'-(hexane-1,6-bis(oxy)) bis(2,1-phenylene))bis (methanylidene) dibenzhydrazide. (VI).The Schiff bases were checked by different spectral technique (LC-MS, ^{1}H-NMR, IR, elemental analyses). The new Schiff Bases were studied for antibacterial activities against (Bacillus subtilis and Staphylococcus aureus) are Gram positive and (Salmonella typhi and Escherichia coli) are Gram negative. The ligands were exhibited a variable activity of inhibition on the growth of the bacteria.

Keywords: *macrocyclic schiff bases, open schiff bases spectral technique, antibacterial activity.*

GJSFR-B Classification : *FOR Code: 030503*

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I. Introduction

Polyazamacrocycles with large cavities have received recent interests as inorganic cation receptors [Kopolow et al,1973]. The cyclic arrangement of a large number of donor atoms and the flexibility of these ligands make them good hosts for ions [Lindoy et al.,1976]. They were also used as spectrophotometric analytical reagents [Lisowski et al.,1999]. Metal ions have enormous importance in many biological processes, especially heavy metal ions which are effective enzyme inhibitors exerting toxic effects on living system [Esteban et al.,1037]. Therefore, separation and determination of toxic metal ions such as mercury, lead, and cadmium in environmental sources play an important role for healthy life [Vance et al.,1997]. Although new paragraph macrocyclic compounds containing oxygen, sulfur, and nitrogen are knowing. Macrocyclic compounds used in solvent extraction were mostly the oxygen donor type [Ekstrom et al.,1980]. A number of methods for the preparation of the large polyazamacrocyclics have been reported. The most common synthetic procedure requires the use of N-tosyl groups to protect and activate the nitrogen atoms in the cyclization step [Ekstrom et al.,1979]. Ring closure occurs by a condensation reaction of *N*-tosylated polyamines with the appropriate ditosylate ester or dihalide in DMF in the presence of base [Anderegg et al.,1980]. These reactions allow the production of polyazamacrocycles in moderate yields [Adam et al.,1994], but removing the *N*- tosyl groups requires drastic conditions and is not always straightforward. Another cyclization process uses the template ring closure for formation of cyclic di- or tetra dentate Schiff bases. This is a simple process, but it is often difficult to choose the correct template metal ion or to predict certain ring contraction reactions were the template cation dose not coordinate with all of the ring nitrogen atoms [Fenton et al.,1981]. In some cases, reduction of the cyclic Schiff base and removal of the template ion have been difficult [Adam et al.,1981]. A non template method for the formation of macrocyclic poly Schiff bases has also been studied. This procedure often gave a polymeric material beside cyclization reaction, while there is no need to remove a metal ion [Henrick et al.,1984]. In the present work we used a non template method for the formation of four novel macrocyclic Schiff bases: (III), (IV), (V) and (V) then were used for removed of various metal ions from the aqueous phase into the organic phase in liquid – liquid extraction system.

II. Experimental

a) Materials and Method

Chloroform, dichloromethane, dimethylformamide, acetonitrile, methanol, K_2CO_3, were analytical grade reagents and were purchased from Merck. 4,4'-Diaminodiphenylmethane,4-Aminophenyl sulfone, benzylhydrazide, salicylaldehyde, 1,6- dibromohexane, α,α'-Dichlor-p-xylene were obtained from sigma- Aldrich. IR spectra were recorded on Jusco 300 FT-IR Spectrometer using KBr discs. Mass spectra of the ligands were carried out using a micro mass QuattroLC-

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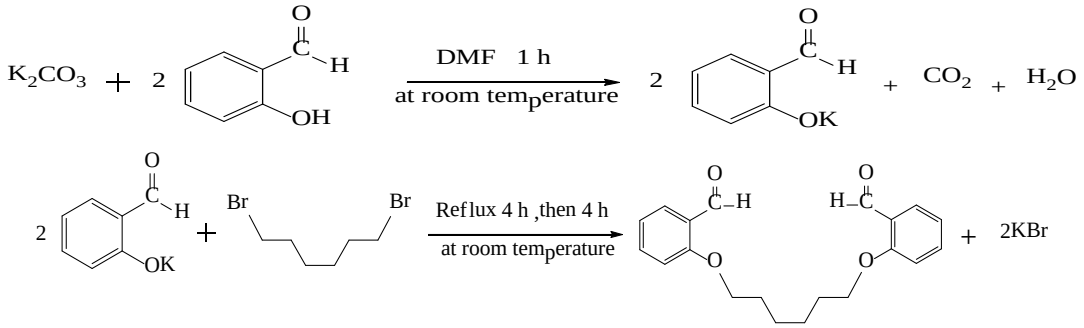
MS/MS Spectrometer. NMR spectra were recorded at ambient BRUKERRAVANCE PX-400 Spectrometer.

i. *Synthesis of 1,6- Bis (2- Formylphenel) Hexane (I)*

To a stirred solution of salicylaldehyde (2.44g, 0.02mol) and K_2CO_3 (1.38g, 0.01mol) in DMF (50ml) 1,6-dibromohexane (2.24 g, 0.01mol) in DMF (10ml) was added dropwise. The reaction was heated for 4h at 150-155 ^{0}C and then stirred at room temperature for 4h [Kenneth et al.,1995]. After the reaction was completed, 100 ml distilled water was added, left in a refrigerator for 1h, the precipitate was filtered, washed by 50 ml water, dried in air and recrystallized from ethanol. (scheme No.1)- (I)

Yield: 80% , colour: White, m.p: 75 0C.

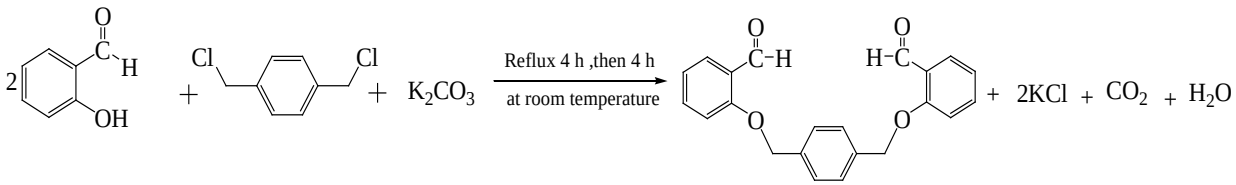


(Scheme No.1)- (I)

ii. *Synthesis of A,A'-Bis(2-Carboxyaldehyde Phenoxy) Xylene (II)*

To a stirred solution of salicylaldehyde (2.44g , 0.02mol) and K_2CO_3(1.38 g, 0.01mol) in DMF (50ml) α,α'-Dichlor-p-xylene (1.39g, 0.01 mol) in DMF(10ml) was added dropwise. The reaction was heated for 4h at 150-155^{0}C and then stirred at room temperature for 4h, after the reaction was completed, 100 ml distilled water was added, left in a refrigerator for 1h, the precipitate was filtered [Lindoy et al.,1976, Kenneth et al.,1995], washed by 50 ml water, dried in air and recrystallized from ethanol.(scheme No.2).

Yield: 85%, colour: White, m.p: 107 0C.



(Scheme No.2)- (II)

iii. *Synthesis of 1,16-Di Aza-3,4,13,14-Tri Phenyl-17,25 -Di Phenyl Methane-5,12-Di Oxacyclo Penta-Icozane-1,15-Diene(III)*

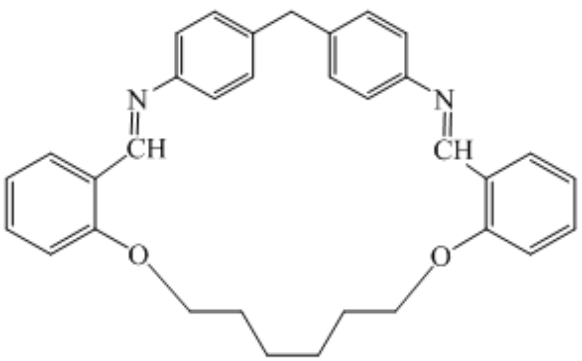
The macrocyclic compound (III) was prepared by dropwise addition of a solution of 4,4'-Diaminodiphenylmethane (0.40g, 0.002 mol) in methanol (40 ml) to a stirred solution of compound (I) (0.652g, 0.002 mol) in methanol (60 ml). The stirring was continued for 12h, a white powder[Salih et al.,2007] was precipitated which was filtered and washed with cold diethyl ether, and recrystallized from mixed (DMF , ethanol9:1). (scheme No.3).

Yield: 80%, colour: Yellow, m.p= 284 ^{0}C. formula: ($C_{33}H_{32}N_2O_2$), M.Wt:(488g).

IR (KBr disk): 3041.8 - 3083.3 cm^{-1} ((C-H), aromatic), 2870.6-2946.7 cm^{-1} (C-H), aliphatic), 1660.4cm^{-1} (C=N), 1573.7-1593.3 cm^{-1} (C=C, aromatic), 1243.5cm^{-1} (C-O).

^{1}H-NMR ($CDCl_3$-400MHz) δ=8.512 (s, 2H, CH=N), 6.954 - 7.766 (m, 16 H, Ar), 4.087-4.119 (s,4H ,O-CH_2-), 2.649 (s,2H, Ph-CH2-Ph), 1.625 – 1.927 (m, 8H,-CH_2-).

Elemental analysis found % C : 81.07 , H: 6.72 , N: 5.69 , O: 6.52 calculated for ($C_{33}H_{32}N_2O_2$) % C: 81.12, H: 6.60 , N: 5.73 ,O:6.55.



(Scheme No.3) (III)

iv. *Synthesis of 1,16-Di Aza-3,4,7,10,13,14-Tri Phenyl-17,25 -Di Phenyl Methan-5,8-Di Oxacyclo Penta-Icozane-1,15-Diene. (IV).*

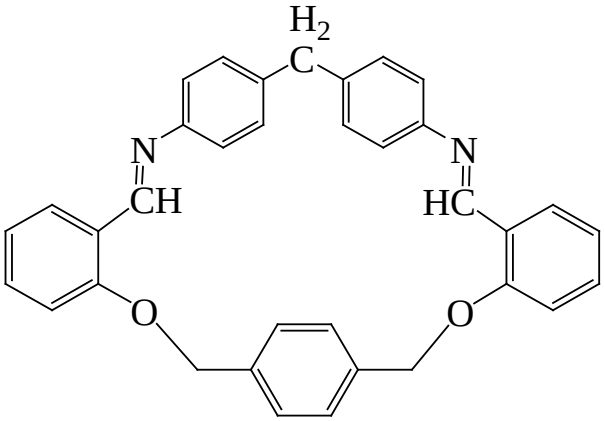
The macrocyclic compound (IV) was prepared by dropwise addition of a solution of 4,4'-Diaminodiphenylmethane (0.40g, 0.002 mol) in methanol (40 ml) to a stirred solution of compound (II) (0.692g, 0.002 mol) in methanol (60 ml). The stirring was continued for 12h, a white powder [Salih et al.,2007] was precipitated which was filtered and washed with cold diethyl ether, and recrystallized from mixed (DMF , ethanol.9:1).(scheme No.4).

Yield: 80%, colour: White, m.p> 300 0C dec. formula: ($C_{35}H_{28}N_2O_2$), M.Wt: (508g).

IR (KBr disk): 3056.6 cm^{-1} ((C-H), aromatic), 2870.7-2946.6 cm^{-1} ((C-H), aliphatic), 1660.8 cm^{-1} (C=N), 1575.2-1595.0 cm^{-1} (C=C, aromatic), 1243.2 cm^{-1} ((C-O).

^{1}H-NMR ($CDCl_3$-400MHz) δ=8.661(s,2H,CH=N), 6.965-8.160 (m,20 H, Ar), 5.073 (s, 4H- O-CH_2-Ph-), 3.994 (s,2H,-Ph-CH_2-Ph).

Elemental analysis found % C: 82.71, H: 5.48, N: 6.42, O: 5.39 calculated for ($C_{35}H_{28}N_2O_2$) % C: 82.65, H: 5.55, N: 5.51, O: 6.29.



(Scheme No.4)- (IV)

v. *Synthesis of 1,16-Di Aza-3,4,13,14-Tri Phenyl-17,25 -Di Phenyl Sulphide-5,12-Di Oxacyclo Penta-Icozane-1,15-Diene(V)*

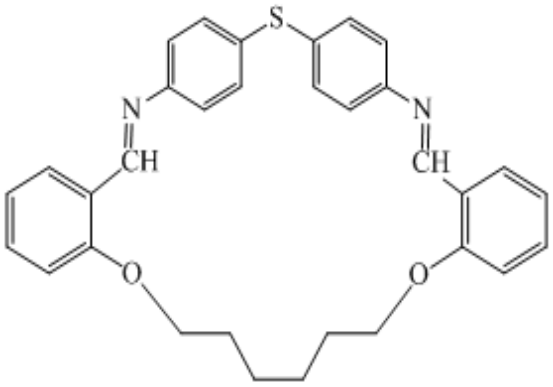
The macrocyclic compound (V) was prepared by dropwise addition of a solution of 4-Aminophenyl sulfone (0.432 g, 0.002 mol) in methanol (40 ml) to a stirred solution of compound (II) (0.692g, 0.002 mol) in methanol (60 ml). The stirring was continued for 12h, a white powder [Salih et al.,2007]was precipitated which was filtered and washed with cold diethyl ether, and recrystallized from mixed (DMF , ethanol. 9:1). (scheme No.5).

Yield: 65 %, colour: Yellow, m.p> 300 0C dec. formula: ($C_{32}H_{30}N_2O_2S$), M.Wt: (508g).

IR (KBr disk): 3047.6 cm^{-1} ((C-H), aromatic), 2864.7-2942.4 cm^{-1} (C-H), aliphatic), 1675.2cm^{-1} (C=N), 1594.2 cm^{-1} (C=C, aromatic), 1245.8cm^{-1} ((C-O).

^{1}H-NMR (CDCl3-400MHz) δ = 8.140 (s,2H,CH=N), 7.117-7.854 (m,16 H, Ar) , 3.729 (s,4H ,O-CH_2-), 1.164 - 1.642 (m, 8H,-CH_2-).

Elemental analysis found % C: 75.98, H: 5.86, N: 5.49, O: 6.44, S: 6.23 calculated for ($C_{32}H_{30}N_2O_2S$) % C: 75.86, H: 5.97, N: 5.53, O:6.32, S: 6.33.



(Scheme No.5)- (V)

vi. *Synthesis of N',N'-(2,2'-(Hexane-1,6-Bis(Oxy))Bis(2,1-Phenylene))Bis(Methanylidene) Dibenzhydrazide.(VI)*

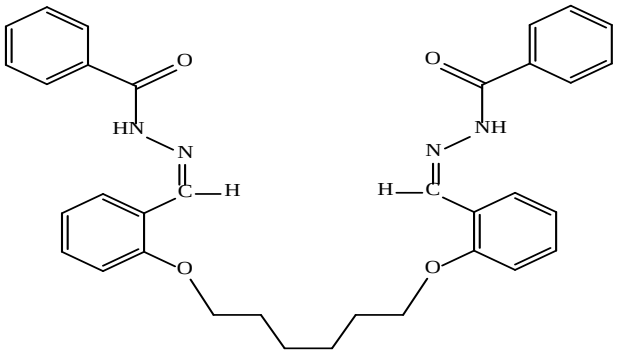
The open Schiff base (VI) was prepared by dropwise addition of a solution of the benzhylhyrazide (2.72 g, 0.02 mol) in DMF (40 mL) to a stirred solution of 1,6-bis(2- formyl phenyl)-hexane (I) (3.26 g, 0.01mol) in DMF (60 mL) containing a few drops of concentrated HCl. The reaction mixture was heated to reflux for 5 hrs, where white precipitate was formed after cooling. On cooling, 200 ml distilled water was added and the mixture was kept in a refrigerator. After 2 hrs, the precipitate [Sultan et al.,2011] was filtered and washed with 200 mL water. The solid obtained was collected and recrystallized from mixture (DMF EtOH9:1) as yellow crystals. A white colored precipitate was washed with water, ethanol, CHCl3 and diethyl ether, respectively. Then dried in air. (scheme No.6).

Yield: 84%, colour: White, m.p= 263 ^{0}C dec. formula:($C_{35}H_{28}N_2O_2$), M.Wt: (562 g).

IR (KBr disk): 3217.27cm^{-1} (N-H), 3035.96 – 3062.96 cm^{-1}((C-H), aromatic, 2870.08 – 2939.52((C-H), aliphatic), 1647.21cm^{-1} (C=O), 1642.0 cm^{-1} (C=N).

^{1}H-NMR ($CDCl_3$-400MHz) δ =11.891 (s,2H, CO-NH-), 8.824 (s,2H,CH=N), 7.007 - 8.479 (m,18H, Ar), 1.065 – 1.820(m,8H,-CH_2-CH_2-CH_2-CH_2-), 2.512 - 3.353 (DMSO,H_2O).

Elemental analysis found % C: 72.81, H: 5.98, N: 10.04, O: 11.17 calculated for ($C_{34}H_{34}N_4O_4$) % C: 72.58; H: 6.09; N: 9.96; O: 11.37.



(Scheme No.6) (VI)

b) Biological Activity

The prepared compounds were tested for their antimicrobial activity against four speices of bacteria (Bacillus subtilis, Escherichia coli, Staphylococcus aureus, Salmonella typhi) using filter paper disc method [Ibrahim et al.,2006]The screened compounds were dissolved individually in DMSO (dimethyl sulfoxide) in order to make up a solution of 50, 100, and 200 µg/ml concentration for each of these compounds. Filter paper discs (Whitman No.1 filter paper, 5mm diameter) were saturated with the solution of these compounds. The discs were placed on the surface of solidified Nutrient agar dishes seeded by the tested bacteria. The diameters of inhibition zones (mm) were measured at the end of an incubation period, which was 24 h at 37C for bacteria. Discs saturated with DMSO are used as solvent control. Ciprofloxacin 100 µg/ml was used as reference substance for bacteria.[Ibrahim et al.,2006]

III. Result and Discussion

a) Synthesis

The prepared macrocyclic (III, IV, V, VI) were synthesized by the condensation of by condensation of intermediate compounds 1,6- bis (2- formylphenel) hexane (I) and α,α'-bis(2-carboxyaldehyde phenoxy) xylene (II) with both 4,4'-Diaminodiphenylmethane and 4-Aminophenyl sulfonein the molar ratio 1:1 in absolute methanol. Also new open Schiff bases (VI) which was prepared by condensation of benzylhydrazide with 1,6-bis (2- formylphenel) hexane in the molar ratio 2:1 in DMF. The reactions proceeded smoothly, producing the corresponding Schiff bases ligands in good yield. The ligands are soluble in common organic solvent but insoluble in water. The structures of the ligands were elucidated by elemental analyses, MS, FTIR, electronic absorption, and ^{1}H- NMR spectra, which help in elucidating their empirical formula Table 1.

Table 1 : Physical and chemical properties of the synthesized compounds[III]-[VI]

Schiff base	Color	M.Wt	Melting point ^{0}C	Yield %	Crystallization Solvent
III	White	448	248	80	DMF, EtOH (9:1)
IV	White	508	> 300	85	DMF, EtOH (9:1)
V	Yellow	506	> 300	65	DMF, EtOH (9:1)
VI	White	562	263 – 264	84	DMF, EtOH (9:1)

b) Elemental analyses of macrocyclic and open (III, IV, V, VI)

The results of elemental analyses macrocyclic and open (III, IV, V, VI), as shown in Table 2, are in good agreement with those required by the proposed formulae.

Table 2 : Elemental analysis data of the synthesized compounds[III]-[VI]

Schiff base	Elemental analysis Calculated (Found %)				
	C	H	N	S	O
III	81.07 (81.12)	6.72 (6.60)	5.69 (5.73)	--------	6.52 (6.55)
IV	82.71 (82.65)	5.48 (5.55)	6.42 (6.51)	--------	5.39 (5.29)
V	75.98 (75.86)	5.86 (5.97)	5.49 (5.53)	6.23 (6.33)	6.44 (6.32)
VI	72.81 (72.58)	5.98 (6.09)	10.04 (9.96)	--------	11.17 (11.37)

c) IR spectra analysis of macrocyclic Schiff bases (III, IV, V, VI)

i. *Compound (III)*

A strong band at 1660.4cm^{-1} in the IR spectrum of the macrocyclic Schiff base (Figure (1)) are assigned to υ(C=N) of azomethine vibrations. The band in the spectra at 1593.3 - 1573.7cm^{-1} is due to (C=C) of aromatic rings. The band in the spectra at 1243.5 cm^{-1} is due to (C-O). while the band at 2946.7 - 2870.6cm^{-1} is attributed to (C-H aliph). Also, the band at 3083.3 - 3041.8cm^{-1} is attributed to (C-H ar). [Salih et al.,2007, Sultan et al.,2011].

ii. *Compound (IV)*

A strong band at 1660.8 cm^{-1} in the IR spectrum of the macrocyclic Schiff base (Figure (2)) are assigned to υ(C=N) of azomethine vibrations. The band in the

spectra at 1595.0 - 1575.2 cm^{-1} is due to (C=C) of aromatic rings. The band in the spectra at 1243.2 cm^{-1} is due to (C-O). while the band at 2946.6 - 2870.7 cm^{-1} is attributed to (C-H aliph). Also, the band at 3056.6 cm^{-1} is attributed to (C-H ar). [Salih et al.,2007, Sultan et al.,2011].

iii. *Compound (V)*

A strong band at 1675.2 cm^{-1} in the IR spectrum of the macrocyclic Schiff base (Figure (3)) are assigned to υ(C=N) of azomethine vibrations. The band in the spectra at 1594.2 cm^{-1} is due to (C=C) of aromatic rings. The band in the spectra at 1245.8 cm^{-1} is due to (C-O). while the band at 2942.4 - 2864.7 cm^{-1} is attributed to (C-H aliph). Also, the band at 3047.6 cm^{-1} is attributed to (C-H ar). [Salih et al.,2007, Sultan et al.,2011].

iv. *Compound (VI)*

A strong band at 1600.92 and 1647.21cm^{-1} in the IR spectrum of the Schiff base (Figure (4)) are assigned to υ(C=N) of azomethine and carbonyl υ(C=O) vibrations, respectively. An intense band at 3217.27 cm^{-1} is due to the -NH- vibrations of the hydrazine group. The band in the spectra at 1554.83 cm-1 is due to (C=C) of aromatic rings. The band in the spectra at 1249.86 cm^{-1} is due to (C-O). While the band at 2870.08 – 2939.52 cm-1 is attributed to (C-H aliph). Also, the band at 3035.96 – 3062.96 cm^{-1} is attributed to (C-H ar). [Salih et al.,2007, Sultan et al.,2011].

However, in the IR spectra of Schiff bases this band (C=O) disappears and a new vibration band for azo methane (-HC=N-), indicating that complete condensation takes place. [17-18].

Table 3 : IR spectral data of the synthesized compounds[III]-[VI]

Schiff base	v(C-O)	v(C=C)	v(HC=N)	v(C=O)	v(C-H) aliph	v(C-H) arom	v(N-H)
III	1243.5	1593.3 - 1573.7	1660.4	---------	2946.7 - 2870.6	3083.3 - 3041.8	---------
IV	1243.2	1595.0 - 1575.2	1660.8	---------	2946.6 - 2870.7	3056.6	---------
V	1245.8	1594.2	1675.2		2942.4 - 2864.7	3047.6	
VI	1249.86	1554.83	1600.92	1647.21	2870.08 – 2939.52	3035.96 – 3062.96	3217.27

d) ^{1}-H-NMR Spectra of macrocyclic Schiff bases (III, IV, V, VI).

i. *Compound (III)*

The ^{1}H NMR spectrum (Figure (5)) of the Schiff base (III), showed that in the signals at 8.512 ppm were assigned to the protons of imine -CH=N groups, The multiple signals in the region 1.927 - 1.625 ppm were assigned to protons of methylene groups in two different environments [Salih et al.,2007, Sultan et al.,2011]. The multiple signals in the region 7.766 - 6.954 ppm were assigned to the aromatic protons. While The signals at 4.119 - 4.087 ppm were assigned to the protons of (-O-CH_2-) group.

ii. *Compound (IV)*

The ^{1}H NMR spectrum (Figure (6)) of the Schiff base (IV), showed that in the signals at 8.661 ppm were assigned to the protons of imine -CH=N groups [Salih et al.,2007, Sultan et al.,2011].The multiple signals in the region 8.160 - 6.965 ppm were assigned to the aromatic protons. While the signals at 5.073 ppm were assigned to the protons of (-O-CH_2-) group.

iii. *Compound (V)*

The ^{1}H NMR spectrum (Figure (7)) of the Schiff base (V), showed that in the signals at 8.140 ppm were assigned to the protons of imine -CH=N groups, The multiple signals in the region 1.642 - 1.164 ppm were assigned to protons of methylene groups in two different environments [Salih et al.,2007, Sultan et al.,2011].The multiple signals in the region 7.854 - 7.117 ppm were assigned to the aromatic protons. While the signals at 3.729 ppm were assigned to the protons of (-O-CH2-) group. Also the signal at 11.891 ppm were assigned to the protons of amide (-CO-NH-) groups.

iv. *Compound (VI)*

The ^{1}H NMR spectrum (Figure (8)) of the Schiff base (VI), showed that in the signals at 8.824 ppm were assigned to the protons of imine -CH=N groups, The multiple signals in the region 1.065 – 1.820 ppm were assigned to protons of methylene groups in two different environments [Salih et al.,2007, Sultan et al.,2011].The multiple signals in the region 8.479 - 7.007 ppm were assigned to the aromatic protons. While the signals at 4.087 ppm were assigned to the protons of (-O-CH_2-) group. Also the signal at 11.891 ppm were assigned to the protons of amide (-CO-NH-) groups.

The other obtained values for ^{1}-H-NMR chemical shifts of the compounds are given in the experimental section. [Pathak et al.,2000, Rajaa.,2008].

Table 4 : ^{1}H-NMR Spectra of the synthesized compounds[III]-[VI]

Schiff base	Chemical Shifts δ ppm				
	C-H aromatic	CH=N	-CO-NH-	-O-CH_2-	(-CH_2-CH_2-)$_n$
III	7.766 - 6.954 (m,16 H)	8.512 (s,2H)	----------	4.119 - 4.087 (s,4H)	1.927 - 1.625 (m,8H)
IV	8.160 - 6.965 (m,20 H)	8.661 (s,2H)	----------	5.073	----------
V	7.854 - 7.117 (m,16 H)	8.140 (s,2H)	11.891 (s,2H)	3.729 (m,8H)	1.642 - 1.164 (m,8H)
VI	8.479 - 7.007 (m,18H)	8.824 (s,2H)	11.891 (s,2H)	4.087 (s,2H)	1.065 – 1.820 (m,8H)

e) Biological Activity

During the last two or three decades, attention has been increasingly paid to the synthesis of macrocyclic and open (III, IV, V, VI) which exhibits various biological activities including antibacterial, fungicidal, tuberculostatic and plant growth regulative properties [19]. It was judicious to investigate the synthesis of various new types of Schiff base and studied their antibacterial activity against four strains of bacteria (Bacillus subtilis, Escherichia coli, Staphylococcus aureus, Salmonella typhi). The concentrations used for the screened compounds are 50, 100, and 200 µg/ml. Ciprofloxacin was used as reference standard while DMSO as control and inhibition zones are measured in mm. The new compounds were tested against one strain each of a gram positive and two gram negative. The test results presence in Table (3.11), a new compound was active against tested and another compounds are no active.

All compounds are no active where used 50, 100 µg/ml but active in the concentrations 200 µg/ml see table (3.4).

Table 5 : Antibacterial activity of the synthesized compounds[III]-[VI]

Shiff base	Bacteria			
	Gram negative		Gram positive	
	B. subtilis	S. aureus	E.coli	S. typhi
III	17 mm	18 mm	15 mm	18 mm
IV	18 mm	16 mm	16 mm	19 mm
V	20 mm	18 mm	17 mm	18 mm
VI	20 mm	18 mm	17 mm	18 mm
Control	00 mm	00 mm	00 mm	00 mm
Ciprofloxacin	20 mm	20 mm	20 mm	20 mm

(-)No zones of inhibition were observed.
Moderately sensitive, (+) Inhibition zones of 7-10mm.
Sensitive, (++) Inhibition zones of 11-14mm.
High sensitive, (+++) Inhibition zones of 15-20mm.

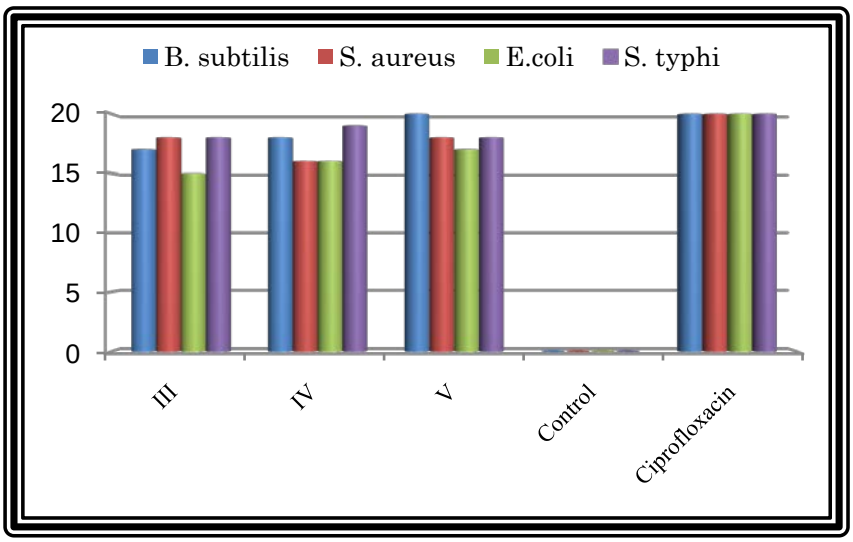


Figure 9 : Antibacterial activity of synthesized compounds[III]-[VI].

IV. Conclusion

1. The compounds are new and were prepared for the first time.
2. The new compounds were identified by melting point, elemental analyses 1HNMR, IR, LC-MS, spectral methods.
3. The prepared compounds have been biologically screened i.e. studying their effects against two gram-positive, two gram-negative bacteria. The results show that their activities were found to vary from moderate to very strong.

V. Acknowledgment

The authors are grateful to the Department of Chemistry, Faculty of Science, University of Damascus, Damascus, Syria.

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Figures

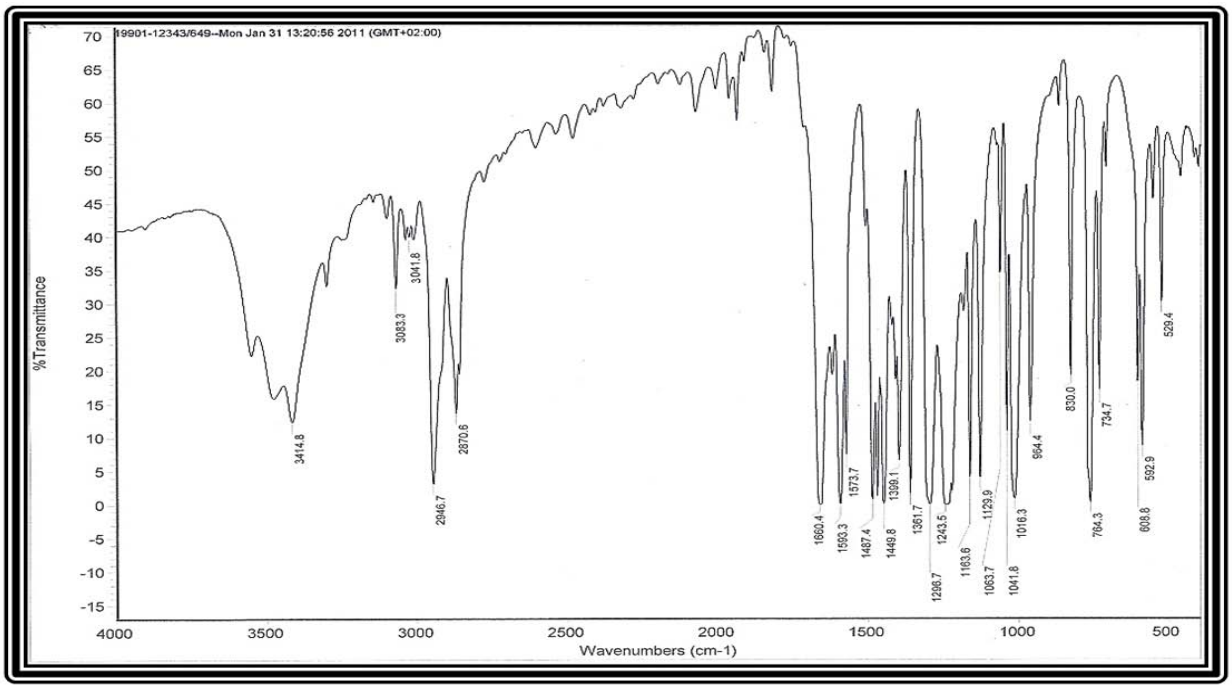

Figure 1 : IR spectrum of Macrocyclic Schiff base(III).

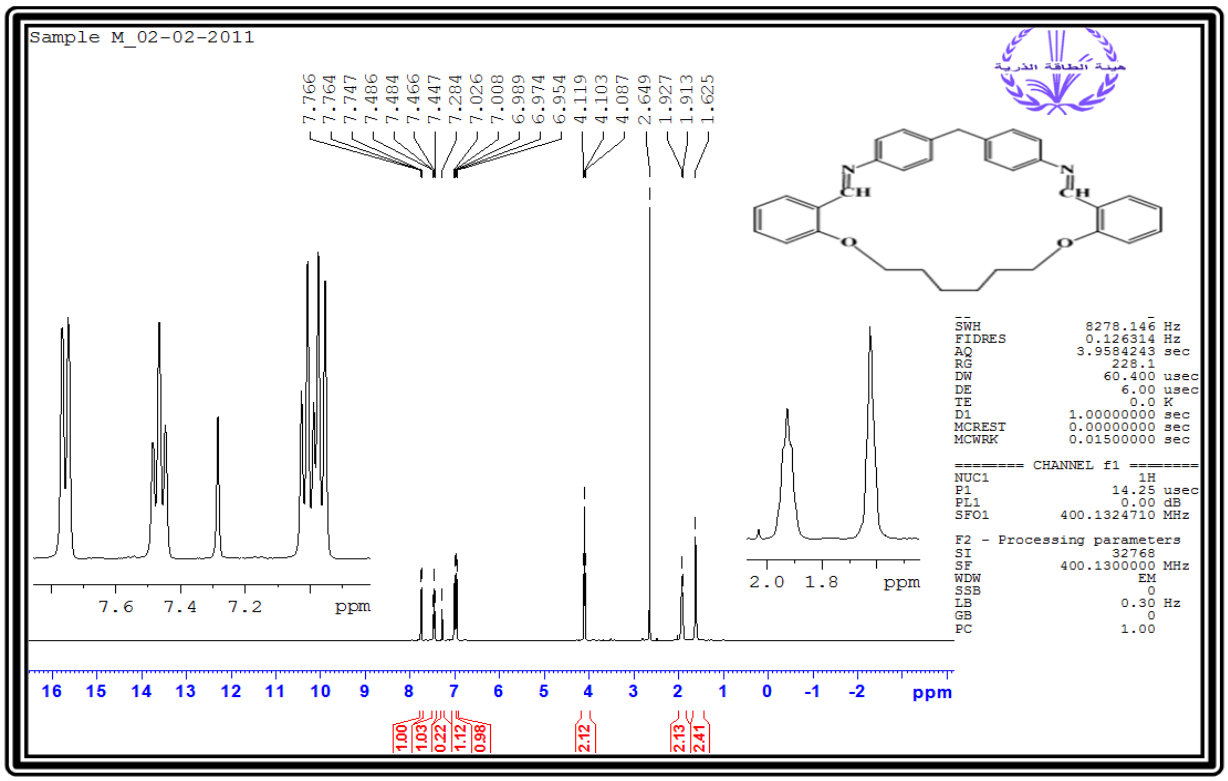

Figure 5 : ^{1}HNMR spectrum of Macrocyclic Schiff base(III)

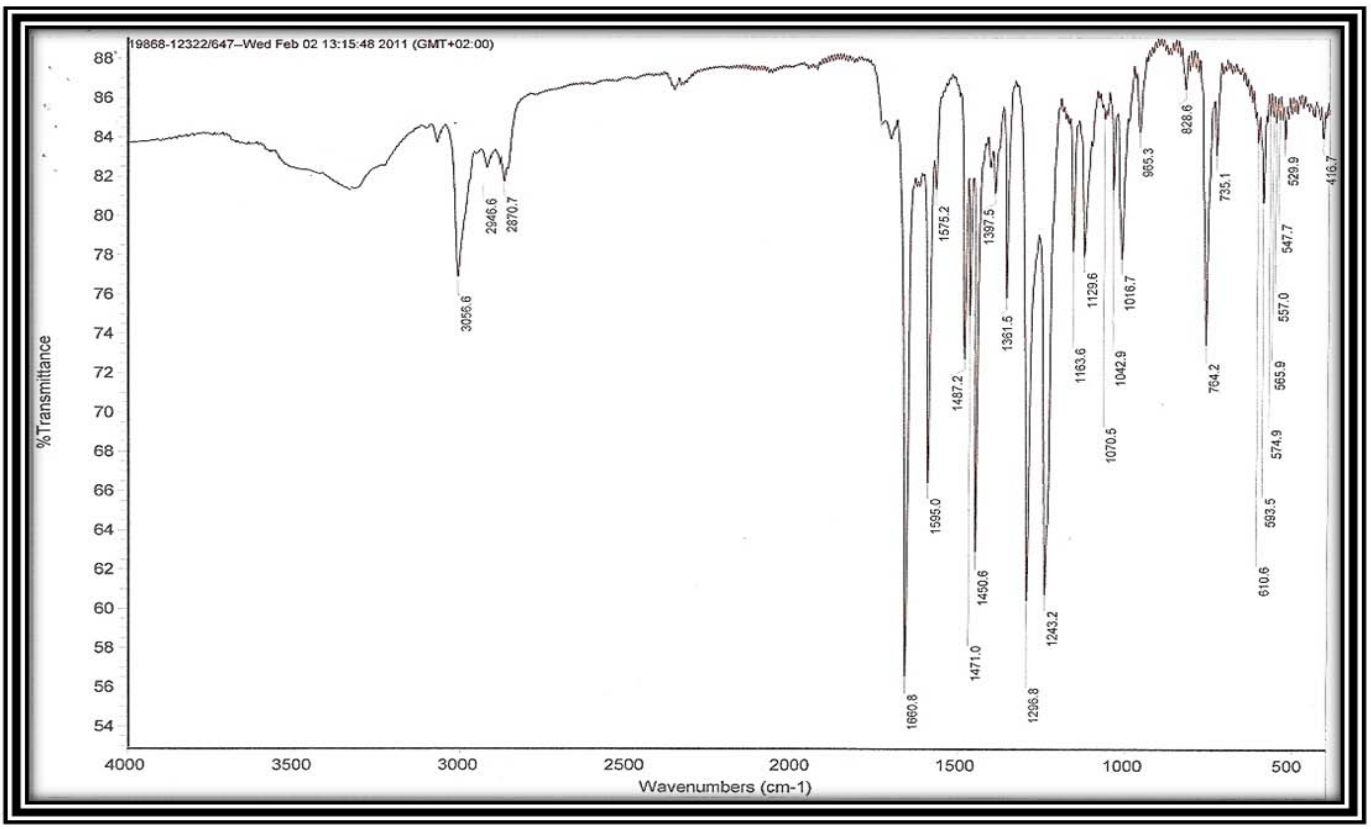

Figure 2 : IR of Macrocyclic Schiff base(IV)

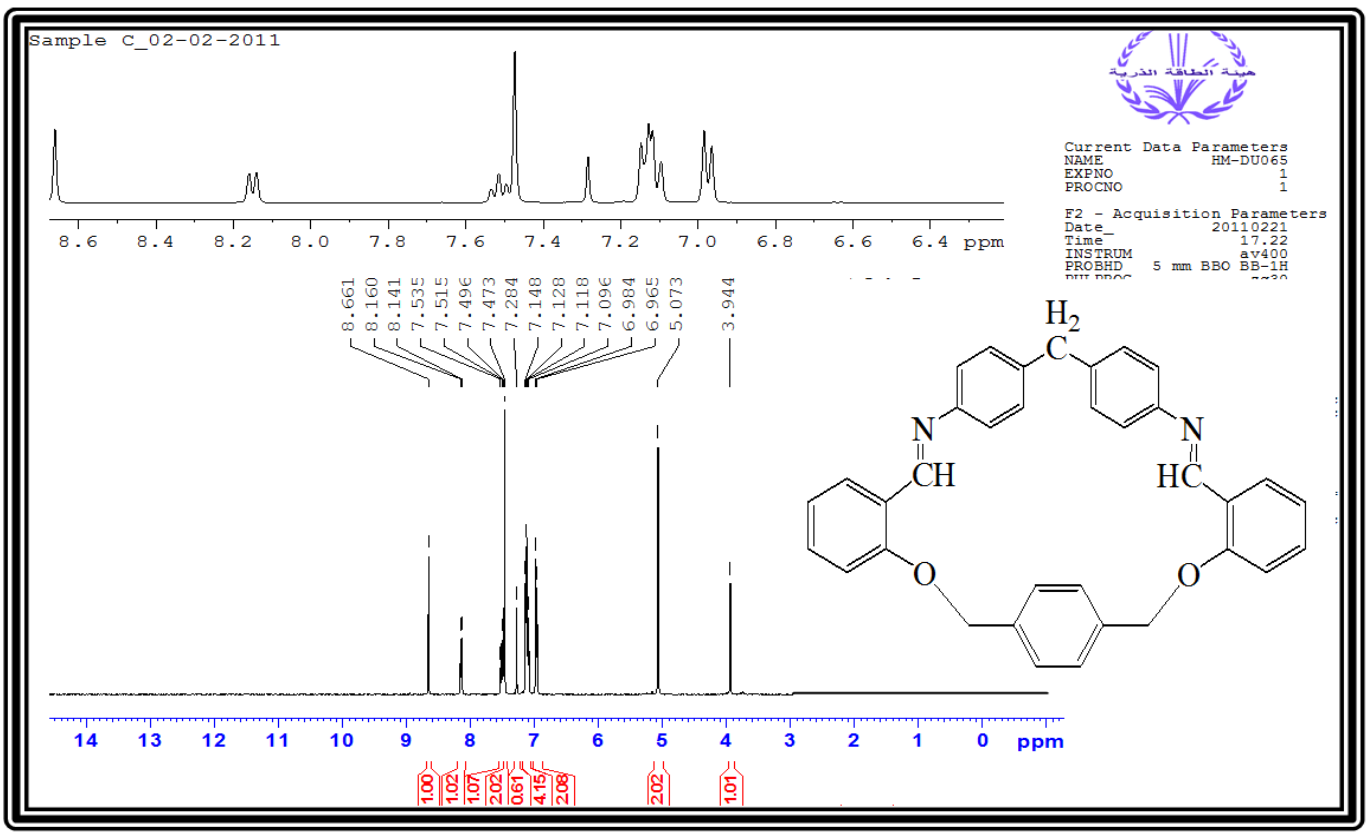

Figure 6 : [1-]HNMR spectrum of Macrocyclic- Schiff base(IV).

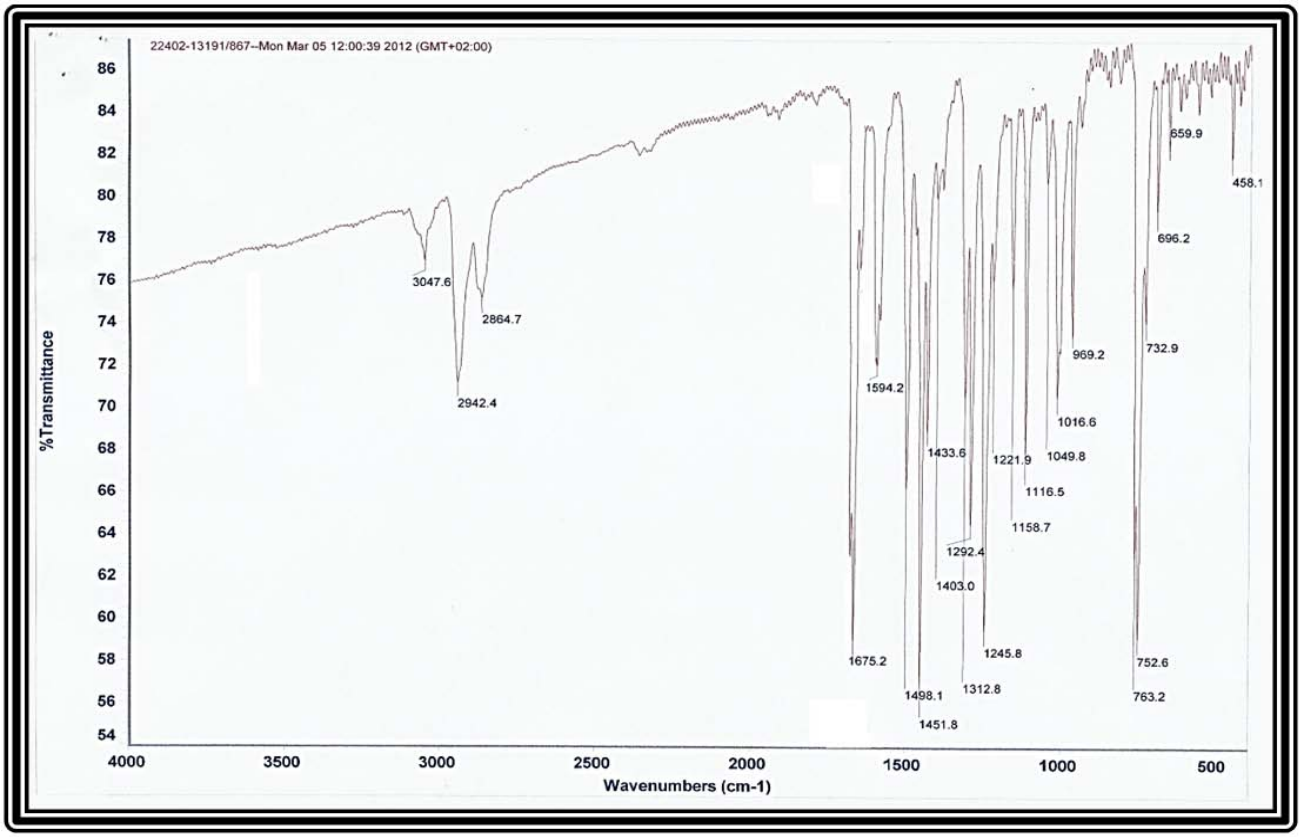

Figure 3 : IR of Macrocyclic Schiff base(IV)

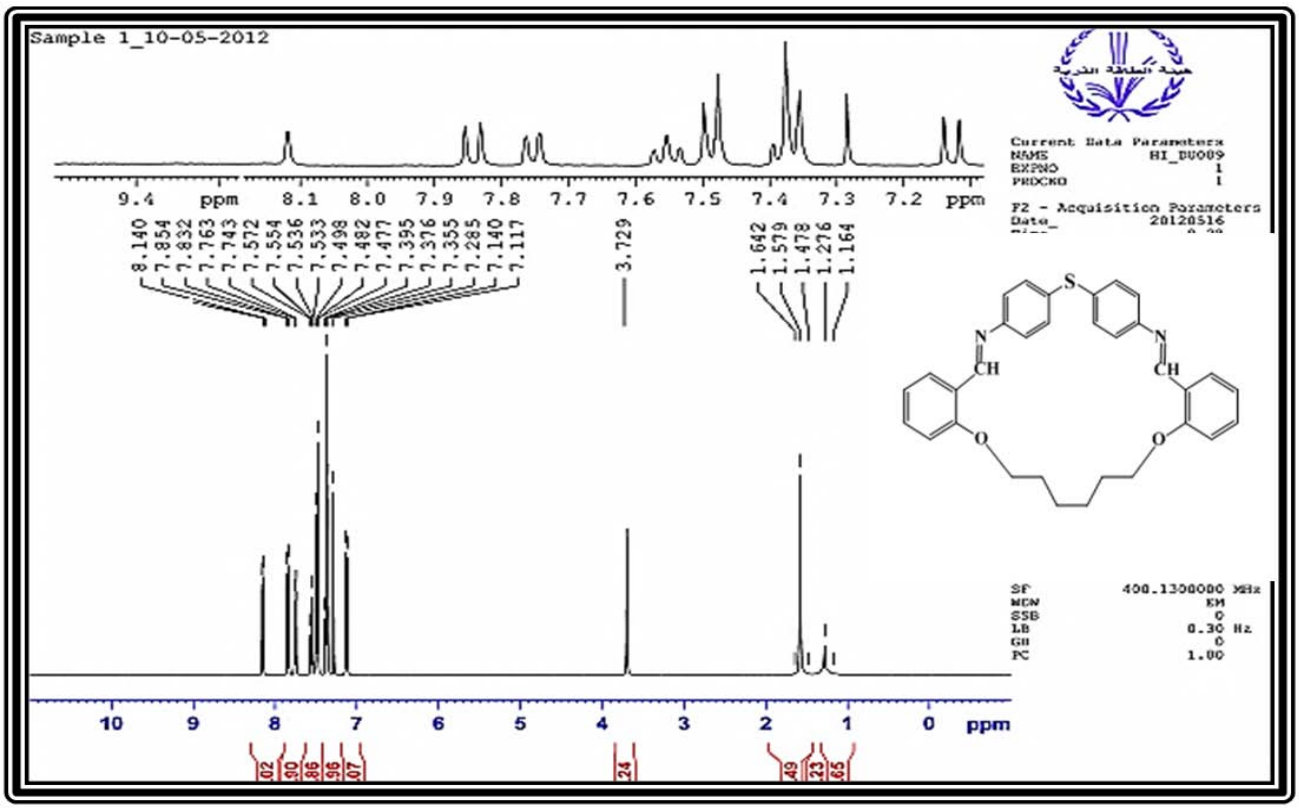

Figure 7 : [1-]HNMR spectrum of Macrocyclic- Schiff base(IV).

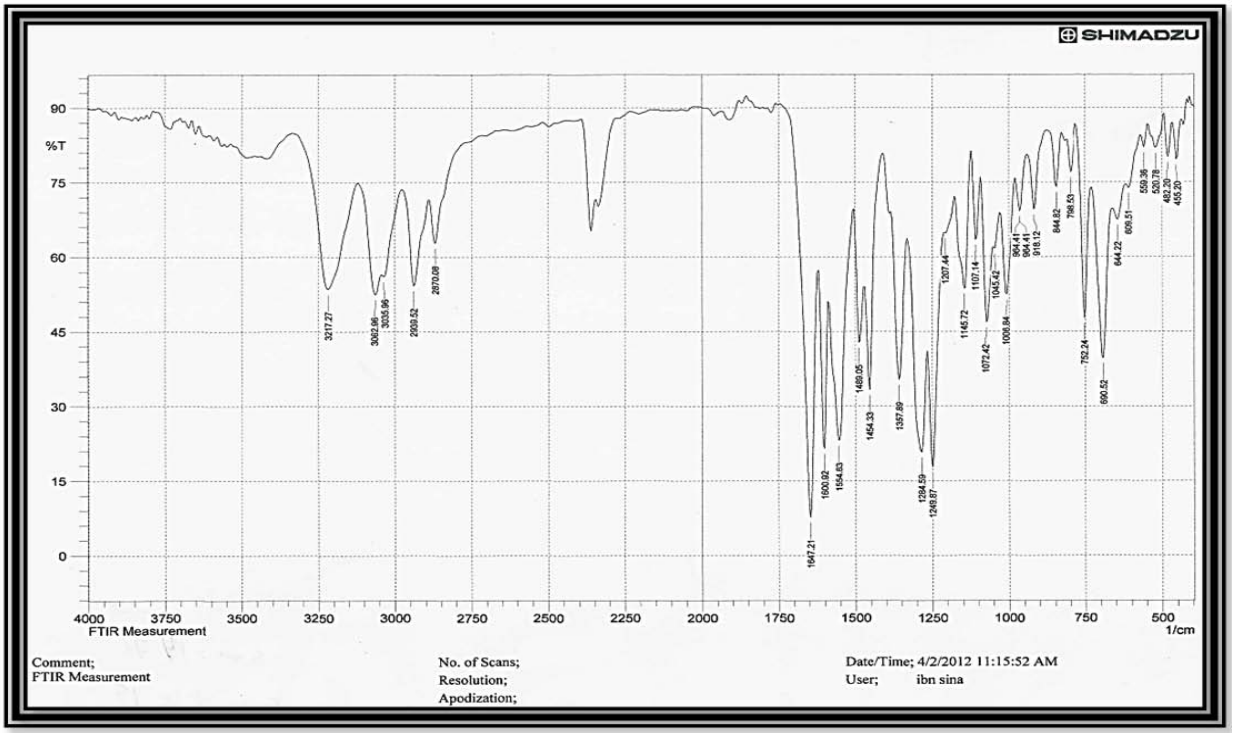

Figure 4 : IR spectrum of Schiff base(V).

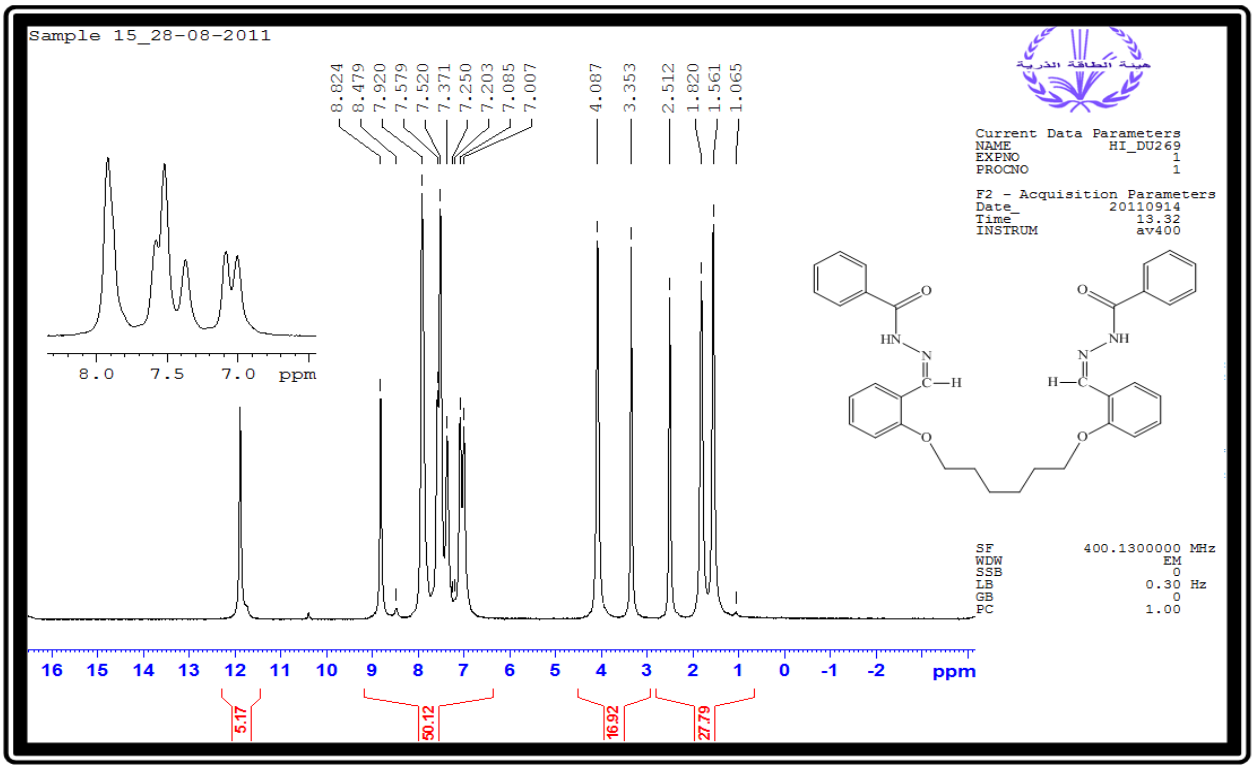

Figure 8 : ^{1}-HNMR spectrum of Schiff base(V).

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Qualitative and Quantitative Analysis for Determination of Glue Sniffer's Urine

By Abdul Rahim Yacob & Mohamad Raizul Zinalibdin
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Abstract- Inhalant abuse is defined as an intentional inhalation of solvents or volatile substances present in materials such as glues and paints. The most commonly abused inhalant today is glue due to low price and easy access. Glue sniffing produces fast and pleasurable sensory experience to the abuser. Quantitative analysis of hippuric acid using UV-Vis at wavelength 417 nm was determined successfully. The results gave a regression coefficient of 0.994. The recovery, accuracy and coefficient variance of hippuric acid were 96.57%, 2.94% and 0.50% respectively. Reversed-phase high performance liquid chromatography using a simple method for the simultaneous determination of hippuric acid and benzoic acid was also described. The chromatography was performed on a Nova-Pak C18 (3.9 x 150 mm) column with a mobile phase of methanol: water: acetic acid (20:80:0.2) using UV detection at 254 nm. The calibration of standards was linear within concentration range of 0.125 to 6.0 mg/mL of hippuric acid and benzoic acid respectively.

Keywords: *glue, hippuric acid, color test, ultra violet-visible.*

GJSFR-B Classification : *FOR Code: 039999*

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Abdul Rahim Yacob [α] & Mohamad Raizul Zinalibdin [σ]

Abstract- Inhalant abuse is defined as an intentional inhalation of solvents or volatile substances present in materials such as glues and paints. The most commonly abused inhalant today is glue due to low price and easy access. Glue sniffing produces fast and pleasurable sensory experience to the abuser. Quantitative analysis of hippuric acid using UV-Vis at wavelength 417 nm was determined successfully. The results gave a regression coefficient of 0.994. The recovery, accuracy and coefficient variance of hippuric acid were 96.57%, 2.94% and 0.50% respectively. Reversed-phase high performance liquid chromatography using a simple method for the simultaneous determination of hippuric acid and benzoic acid was also described. The chromatography was performed on a Nova-Pak C18 (3.9 x 150 mm) column with a mobile phase of methanol: water: acetic acid (20:80:0.2) using UV detection at 254 nm. The calibration of standards was linear within concentration range of 0.125 to 6.0 mg/mL of hippuric acid and benzoic acid respectively. The recovery, accuracy and coefficient variance of hippuric acid were 104.54%, 0.2% and 0.2% and for benzoic acid were 98.48%, 1.25% and 0.60% respectively. A mobile G.S. Kit was developed which employed a mixture of pyridine, benzenesulphonyl chloride and distilled water use as quantitative analysis. Urine samples containing hippuric acid the metabolite of toluene were analyzed using the G.S. Kit. The results show that the mixture would change its color from yellow to red. This method was successful in screening urine samples of suspect toluene abusers or glue sniffers among secondary school children at Johor Bahru and suspect abuser from Hospital Sultanah Aminah with the collaboration of the National Anti Drug Agency.

Keywords: glue, hippuric acid, color test, ultra violet-visible.

I. Introduction

Toluene is also known as methyl benzene or phenyl methane. It is a clear and water insoluble liquid with typical smell and redolent of sweet smell of existed compound of benzene [1]. This chemical is widely used organic solvent in the printing, painting, automotive, shoemaking, adhesive material and the pharmaceutical industries [2]. Normally, toluene is found in many products including paint and contact adhesives as a solvent. Besides that, some grades of toluene contain traces of xylene and benzene [1]. Toluene has a lower boiling point, flammable chemical and easy evaporated. It also the common substance in glue and thinner sniffed by drug abusers.

In Malaysia, the abuse of organic volatile solvents has been observed since the early 1980s. The problem of solvent abuse is predominant in East Malaysia (i.e., Sabah and Sarawak) and in Johore the southern part of West Malaysia bordering Singapore as reported by Navaratnam et, al. (1988) [3]. The trend of inhalant abuse in Malaysia has remained stable during the last 5 years, in contrast to other countries in the region, especially Thailand and Singapore. The types of substances abused include paint thinner, nail polish remover, gasoline, and glue. The most commonly abused inhalant is glue. Preventive education on the various aspects of inhalant abuse and its harmful effects has been carried out as part of preventive education on drug abuse in Malaysia. In the affected states, pamphlets on inhalant use have been produced by the departments of education for use in schools. Resource guides on inhalant abuse have been produced by the Government for health and welfare professionals and teachers.

Detected inhalant abusers of school age are provided counseling by teachers and subjected to disciplinary action. Police report nonschool children to parents for supervision. Presently, no legislation in Malaysia relates specifically to the abuse of inhalants. Nonetheless, existing legislation (i.e., the Juvenile Courts Act of 1947) could be used to a limited extent to help children or adolescents younger than 16 years old who are inhalant abusers. They can be placed in an approved home by the juvenile court. The police also can charge inhalant abusers under the Minor Offense Ordinance of 1955 if they also disturb the peace in the process of abusing.

Recently in next decade this activities rise back by newspaper because of three teenagers aged 18 to 20 were found dead near a school in Cheras, Kuala Lumpur and Police found three can of glue nearby [5]. With the info from these newspapers and previous data it is feared that this problem lead to "time bomb" disasters for future generation in Malaysia.

Toluene can be absorbed into the blood flow from the lung and the gastrointestinal tract and through the skin and mucosa. Brain and liver serve as reservoir for toluene [6]. Following inhalation or oral exposure to toluene, approximately 60 – 75% of absorbed toluene is metabolite to benzoic acid [7, 8]. Please refer to figure

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1. The initial step involves side chain oxidation to benzyl alcohol by cytochrome P450 enzymes. Benzyl alcohol is then further oxidized to benzoic acid by alcohol dehydrogenase and aldehyde dehydrogenase. Benzoic acid is subsequently conjugated with glycine to form hippuric acid [7] the reaction in a figure 2. Benzoic acid may also be conjugated with glucuronic acid to form benzoyl glucuronide in the urine. Less than 1% of absorbed toluene undergoes ring hydroxylation to form o-, and p-creosol, which are excreted in the urine as glucuronide or sulphate conjugates [9, 10,11].

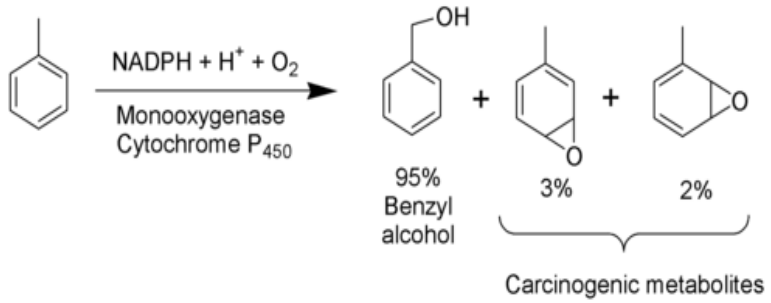

Figure 1 : First step of toluene metabolite

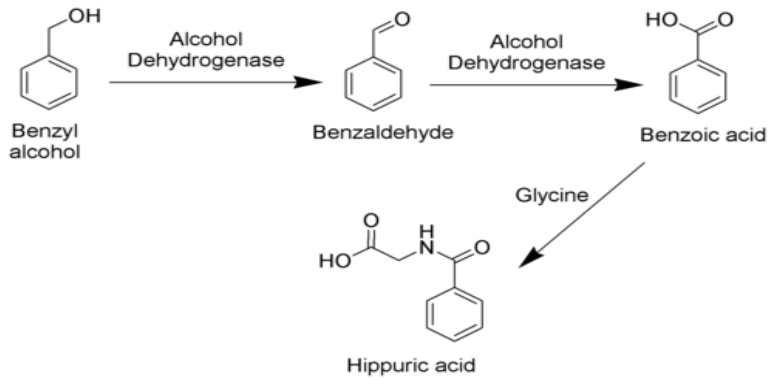

Figure 2 : Second step of toluene metabolite

Novel color reaction for hippuric acid has been developing since 1950 and keep continue in early 1980. The first color reaction developing by Gaffney et al (1954) use based upon azlactone formation resulting from the reaction of an aldehyde with hippuric acid. In this instance, hippuric acid was converted to a deep orange colored azlactone, 2-phenyl-4-(p-dimethylamino) benzal-5-oxazolone by treatment of hippuric acid with acetic anhydride and p-dimethylaminobenzaldehyde [12]. Later, in 1960 Charles J.U found out new method using pyridine and benzenesulfonyl chloride to develop deep red color in present of hippuric acid [13].

The novel color reaction will be applied in this research to determine quantitatively metabolite of toluene which is hippuric acid using Ultra Violet Visible (UV-Vis). The method of color test are using pyridine and benzenesulphonyl chloride was yellow and became reddish by addition of distilled water has been reported by Manabu Yoshida et al (2005). Actually, this novel color reaction has been developing by Charles J.U. However, this color test has been modifying by Manabu Yoshida with adding distillated water to see the color change using naked eye. This novel color reaction will be use as a screening test for glue abuser among the secondary school in Malaysia and might be help the National Anti Drug Agency and Royal Police of Malaysia to prevent the glue abuser among student and teenagers.

II. Experimental

a) Reagents

Hippuric Acid 98%, Benzenesulphonyl chloride (BSC) 98%, Toluene 99.3%, and pyridine 98% purchased from Sigma Aldrich.

b) Instrument

Ultra Violet Visible (UV-Vis) Perkin Elmer.

c) Determination of Hippuric Acid using UV-Vis.

i. *Sample Selection*

Sample urine received from screening using colour test at secondary school around Johore Bahru, Malaysia.

ii. *Sample Preparation*

a. *Urine Sample*

Urine sample of 0.1 ml will be added to 0.25 pyridine and 0.1 ml BSC with 1.0 ml distilled water

b. *Preparation of Standard Hippuric Acid*

12.5 mg, 25.0 mg, 50.0 mg, 100 mg, 150 mg and 200 mg of hippuric acid will be weighed into 100 ml

volumetric flask. The different volumetric flask will be label as standard HA 0.125, HA 0.25, HA 0.5, HA 1.0, HA 1.5 and HA 2.0. The solution will be mark up with distilled water. All standard will be sonicated in a sonicator waterbath. All standard will be run with the UV-Vis Perkin Elmer at wavelength 243 nm. Calibration curve of hippuric acid will develop to get the significance of concentration hippuric acid with absorbance.

III. Results and Discussion

a) Result for Metabolite of Toluene

i. *Colour test for Hippuric Acid*

Table 3.1.1 : Concentration using color test method of hippuric acid at 417 nm wavelength

Concentration of Hippuric Acid	Absorbance
0.000	0.000
0.125	0.149
0.250	0.284
0.500	0.337
1.000	0.654
1.500	0.797
2.000	0.993
3.000	1.500
6.000	2.667

The UV-Vis of the mixtures were recorded 300 to 700 nm using Perkin Elmer Ultra Violet Visible (UV-Vis). A color chart for the semiquantitation of hippuric acid was prepared using hippuric acid standards containing 0.125 mg/ml to 6.00 mg/ml hippuric acid. Tables 3.1.1 show the concentration using color test method of hippuric acid at wavelength 417 nm. Based on the table, this color test method has successfully determined the presence of hippuric acid at different concentration. These results proved that this method might be applied to detect hippuric acid.

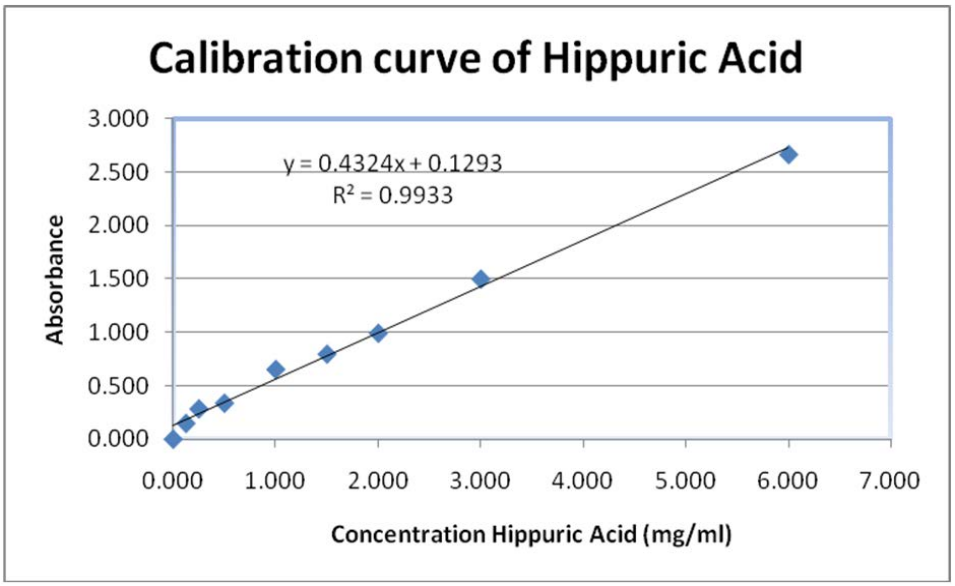

Figure 3.1.1 : Calibration curve of hippuric acid using colour test method

From the calibration curve of hippuric acid using the colour test method, the R2=0.9933 and the amount hippuric acid will be calculate for the equation y=0.4324x + 0.1293. Based on the graph the amount of hippuric acid in the urine can be calculated and shown in the Table 3.1.4.

ii. *Screening Urine Sample Using Colour Test Method*

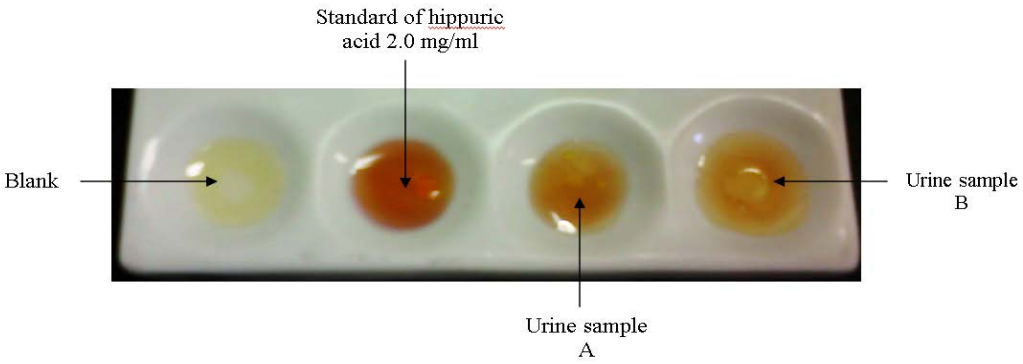

Figure 3.1.1.1 : Colour test for screening urine sample

The colour test for screening urine sample has been shown at the Figure 3.1.1.1. When urine sample contained no hippuric acid, the mixture became colourless and transparent. The urine sample A and B illustrated light red colour similar to the colour of the hippuric acid standard. Thus proving that hippuric acid was present in this urine samples. As comparison, a standard hippuric acid of 2.0 mg/ml also shows the same colour development.

The reaction of the colour test method gave the red colour development. Figure 3.1.1.2 shows chemical equation between benzenesulphonychloride, pyridine and hippuric acid will take part when the chemicals mixed together. In that equation, the chloride will attacked the amine group or hydroxyl group to form ester and ether group. The chloride atom will be form hydrochloric acid. Pyridine is one of the indicators in this reaction similar to phenolphthalein. When the acidic reacted with the pyridine, the red colour developed in this reaction. The equations show that hydrochloric acid react with pyridine to form red colour. This is indicates the presence of hippuric acid in urine glue abuser.

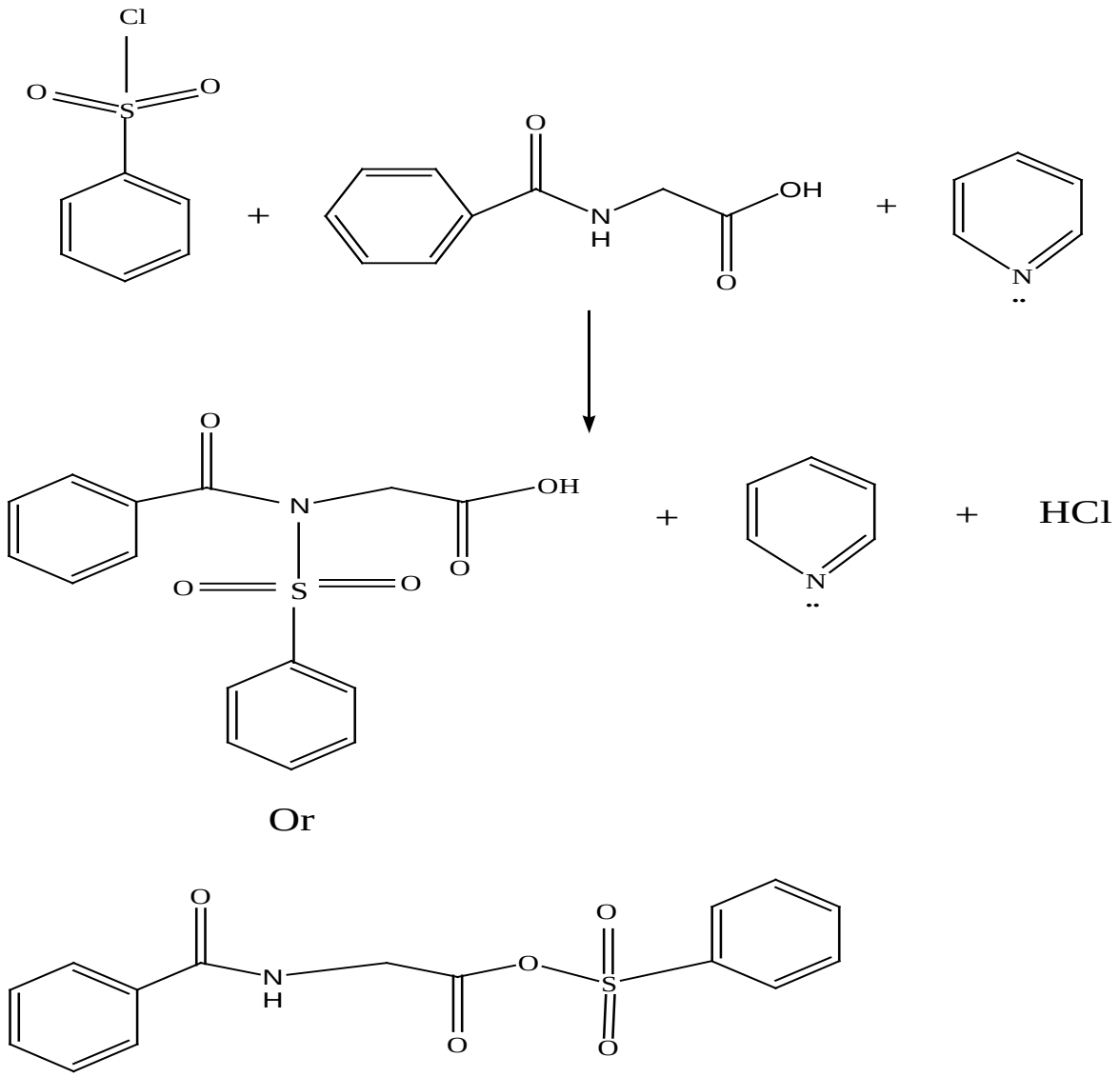

Figure 3.1.1.2 : Chemical equation of colour test method

iii. *Result of Screening Sample at Secondary School*

22 students were involved in the screening urine test at Seri Rahmat Secondary School, Johore Bahru. A number of 4 students gave positive results for hippuric acid in urine sample while the others gave negative results. At the same time, the National Anti Drug Agency was screening 5 drugs using dip strip kit to the same samples and none of the students are tested positive. The positive hippuric acid sample will be further analysing quantitatively using Ultra Violet Visible (UV-Vis) available in the lab. The colour reaction is shown in Figure 3.1.3. Even though, the colour test method is quite useful for qualitative analysis, the reddish colour of the reaction mixture faded gradually, so that semi quantification should be performed as soon as possible.

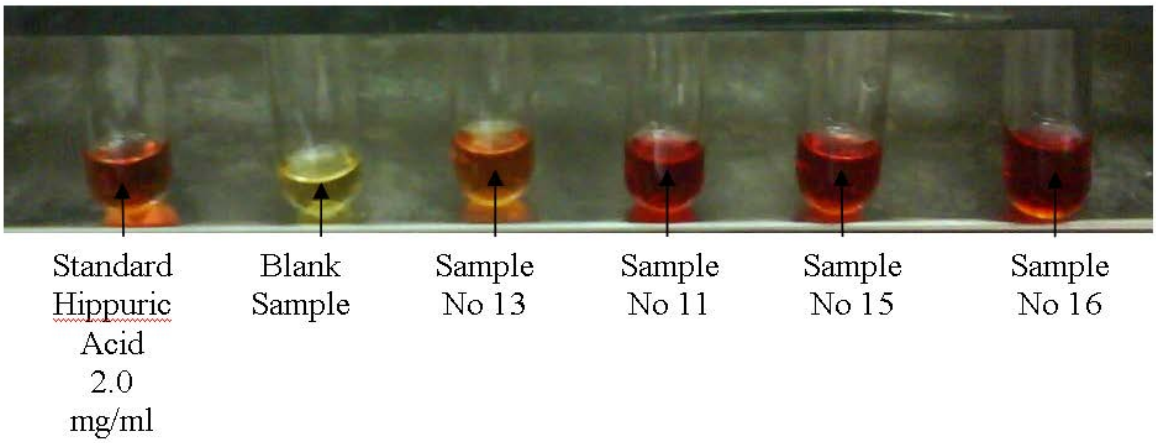

Figure 3.1.3 : Colour test for screening urine sample at secondary school

iv. *Result of Quantitatively of Hippuric Acid using Colour Test by Ultra Violet Visible*

Based from the screening urine test using color test method the result show four students gave the positive of existence of hippuric acid. From the calibration curve of hippuric acid using color test method the equation of y=0.4324x + 0.1293 the amount hippuric acid will be calculate with the R2=0.9933. Calculation amount of hippuric acid in urine has been show at Table 3.1.4.

Table 3.1.6 : Result of amount of hippuric acid using novel color reaction screening urine sample at secondary school.

Sample number	Amount of hippuric acid (mg/ml)
11	3.35
13	1.40
15	2.54
16	3.00

Based from the Manabu et al., (2005) stated that the amount of hippuric acid level more than 2.0 mg/ml indicates the sniffering of toluene with high probability and that a level from 1.0 to 2.0 mg/ml suggests the possibility of toluene abuse. Based from article, the normal human body will produce at least 0.10 mg/ml hippuric acid per day [14]. The result shows in the Table 3.1.6 three of the students have high probability of toluene abuse while other has possibility of toluene abuse.

IV. Conclusion

For screening urine of glue abuser color test method has been used to detect quantitatively hippuric acid with Ultra Violet Visible (UV-Vis) at wavelength 417 nm. It us calculate the intensity of red color. The coefficient value (R2) of determined by this method is 0.9933.

The method of color test are using pyridine and benzenesulphonyl chloride was yellow and became reddish by addition of distilled water has been reported by Manabu et al., (2005). It has been used in this research and has successfully resulted to detect glue sniffer easily using color reaction method with naked eye. In addition, this color test method has been used to screen urine sample among students at the secondary school in Johor Bahru.

Result from the screening at the secondary school show that from 22 students screening urine test, four of them give the positive existence of hippuric acid. Three of the students have high probability of toluene abuse while one of them gave possibility of toluene abuse.

In this case, the spectrophotometer method is thus useful for rapid screening quantitatively for hippuric acid in urine to glue sniffing and toluene abuser. However, the novel color reaction method would be quite useful for the screening of glue sniffing and toluene abuser qualitatively in way to help National Anti Drug Agency, Department of Education and Royal Police of Malaysia to prevent this problem become bigger.

V. Acknowledgment

Special thanks to National Anti Drugs Agency (AADK) for collaboration in this research, financial support from Ministry of Science Technology and Innovation (MOSTI) and Department of Chemistry, Johore branch.

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The Color Pollution Removal (Acid Red 88) of Industrial Waste Waters by Electrocoagulation Method

By Hassan Zhian, Bahrooz Khezry & Chalak Azimi
Islamic Azad University, Iran

Abstract- The aim of this study was to investigate the decolorization of Acid Red 88 (AR88) using electrocoagulation and photo electrocoagulation techniques. Chemical dyes have the most practical usage in different industries, having an essential role in most water industries, they have a high solubility in water, thus, water wastes contain a high level of these dyes. An effective way of eliminating these dyes is electrocoagulation The effect of operational parameters such as initial dye concentration, electrolysis time and electrolyte concentration was studied. The electrochemical cell comprised Al as cathode and Fe as anode which were placed inside a container having a low pressure mercury lamp at the top. 40 mg.L-1 of dye solution was withdrawn for each experiment and after adjusting the electrode distance to 15 mm, current density to 41.8 A m-2, different electrolytes at varying concentrations were added and the absorbance was measured at several reaction times. The results showed that electrocoagulation was more effective than photo electro-coagulation using NaBr, KBr, NaCl and KClaselectrolytes and decolorization rate increased with increasing electrolyte concentration. Also the coupled system with NaF electrolyte showed better decolorization rate than electrocoagulation, but the decolorization rate was higher at low concentrations of electrolyte. Therefore, it is concluded that electrocoagulation was more effective than coupled system in most cases and type of electrolyte plays an important role since employed NaCl/KCl would enhance the decolorization rate considerably.

Keywords: red acid 88, electrocoagulation, industrial water wastes containing dyes, and elimination of industrial dyes, and dyes solved in water.

GJSFR-B Classification : FOR Code: 259999p, 039903

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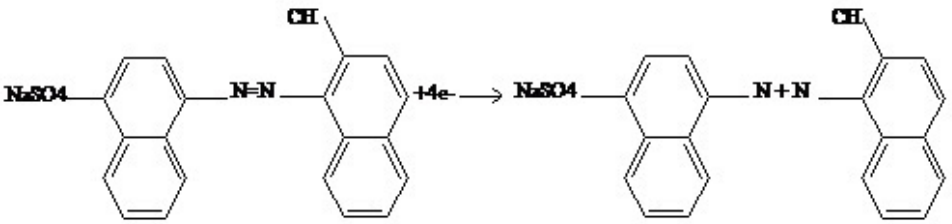
Keywords: red acid 88, electrocoagulation, industrial water wastes containing dyes, and elimination of industrial dyes, and dyes solved in water.

I. Introduction

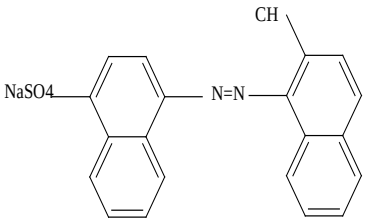
On group of water pollutants are synthesized compounds such as solvents, detergents, dyes, pesticides, food additives, and drugs.[1-2] Since the chemical compounds are increasingly being used, their effects on environment, the risk of their presence in the environment and the efficient methods of their eradication should be studied.[3]

In textile industry where a lot of water is used in the process, water waste purification is a considerably important issue therefore, the plans are made to provide the chance for the industries to increasingly enjoy The science and researches regarding water purification solution, worthy to mention in Azonic.[4-5]

Dyes which contain Azo (-N=N-) if in any condition this factor breaks down, its dye is eliminated[6], as an example:



Red acid 88(AR88)is a mono azo dye and member of acidosis dyes in water solution with molecular formula of $C_2O\ H_{13}\ N_2\ O_4$ SNA and molecular mass of 400 g.mol^{-1}. This acid (C.I.No.15620) has the following formula structure:



Coagulation in which the colloidal particles are destabilized and ready to deposit, start sedimentation by complete coagulation of large particles produced by Vander Waals forces, coagulation is the best method to

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eliminate water waste colloidal particles which would be possible by using some specific chemicals.[7-8] The reduction of colloidal particles is due to the decrease of repulsive potential of electrical doubled layer and the electrical solution effect of iron or aluminum electrodes where releases iron or aluminum irons in anode, and hydrogen gas in cathode. Hydrogen helps flock particles float on the surface of water, a process called electrical congregation.[9] Coagulation is based on the addition of a coagulant to water to construct the nucleus of coagulation and deposit the impurities. Dye sediment process is essentially due to electro static attraction, for motion of unsolder dye complexes, and superficial physical absorption on the sediments produced by adding the coagulant.[10-11]

II. Ion Elimination Mechanism by Electrical Coagulation Process

In electrical coagulation, the sacrificial electrodes are mostly iron or aluminum and the reaction mechanism is summarized as follow,

$$CrO^{2-}{}_{4(aq)} + 3Fe^{2+}{}_{(aq)} + 4H2O_{(l)} + 4OH^{-}{}_{(aq)} \rightarrow 3Fe(OH)_{3(s)} + Cr(OH)_{3(s)}$$

$$CrO_4{}^{2-}{}_{(aq)} + 3Fe^{2+}{}_{(aq)} + 4H_2O_{(l)} \rightarrow 3Fe^{3+}{}_{(aq)} + Cr^{3+}{}_{(aq)} + 8OH_{(aq)}$$

III. Dye Elimination Mechanism in Electro Coagulation

Depending on pH of the environments, and the type of ions existing in the solution there can be different mechanisms to explain bilateral effects between dye molecules and the dye produced by iron and aluminum ion water treatment for example $Fe(OH)_n$ gelatin suspensions which are produced due to electrochemical process can separate the pollutants from the water wastes by complexitizing and surface absorption as well as electrostatic attraction caused by coagulation and floatation.[14-15] In complexitizing it is assumed that the pollutant (ex.dye molecule) is attached to the metal Ion as a ling. Surface absorption mechanism in high ph environment and sediment mechanism in ph environment lower than 6.5 are considered.

a) Sedimentation Process

$$\text{Dye} + \text{mono meric Fe} \rightarrow [\text{Dye-monomeric Fe}]_S$$

$$\text{Dye} + \text{poly meri Fe} \rightarrow [\text{Dye-poiymeric Fe}]_S$$

b) Surface Absorption Process

$$DyE + Fe(OH)_{n(s)} \rightarrow\rightarrow [\text{sludge}]$$

$$[\text{DyE-polymeric Fe}]_{(s)} + Fe(OH)_{n(s)} \rightarrow\rightarrow\rightarrow [\text{sludge}]$$

IV. Methods

All experiments have been accomplished in none continues reactor at 297^0K in 500ml solution using iron and aluminum and graphite electrodes. Considering low conductivity of the sample ordinary salt was used to increase electrical conductivity of red acid 88 water solutions. In order to control the current and apply wattage a rectifier was used. In the experiment the reactors content was transferred to a scaled cylinder for the flocks to deposit. The produced clots were small at first and stayed at the top of the column, but after a while these clots joined each other and erected massive coagulations and started to deposit and the dying substance observed by coagulation was eliminated, then the solution in the upper part of cylinder was filtered and measured, in order to evaluate the efficiency of electro coagulation in dye elimination. Using before and after electrolyze absorption measure meant by UV/Vis photometer spectrum and remaining dye density calibration chart, cells have been observed in different forms.

$$Fe^{3+}{}_{(aq)} + H_2O_{(l)} \rightarrow Fe(OH)^{2+}{}_{(aq)} + H^{+}{}_{(aq)}$$

$$Fe^{3+}{}_{(aq)} + 2H_2O_{(l)} \rightarrow Fe(OH)^{+2} + 2H^{+}{}_{(aq)}$$

$$F^{3+}{}_{(aq)} + 3H_2O_{(l)} \rightarrow Fe(OH)_{3(s)} + 3H^{+}{}_{(aq)}$$

Ferric Ions, in alkali environment can also create $Fe_2(H_2O)_6{}'Fe(H_2O)_8(OH)_2{}^{4+}$, $Fe(H_2O)4(OH)^{+2}$, $Fe(H_2O)_5OH^{2+}$, $Fe(OH)^{-4}$، $Fe(OH)_2{}^{4+}$.

By hydroxide ions where consequently all convert to $Fe(OH)_3$.[12] ($Cro_4{}^{-2}$) Cr^{+6} Ions existing in water wastes can also be eliminated by electro coagulation using iron as sacrificial anode.[13] Fe^{2+}ion effected by electro oxidizations of iron anode in alkali environment would reduce Cr^{6+} to $Cr^{3+}$and would itself convert to Fe^{3+} as shown in the following reaction:

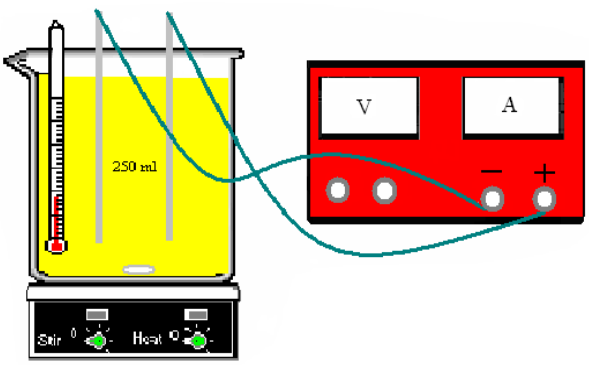

Figure 1 : Electrocoagulation apparatus

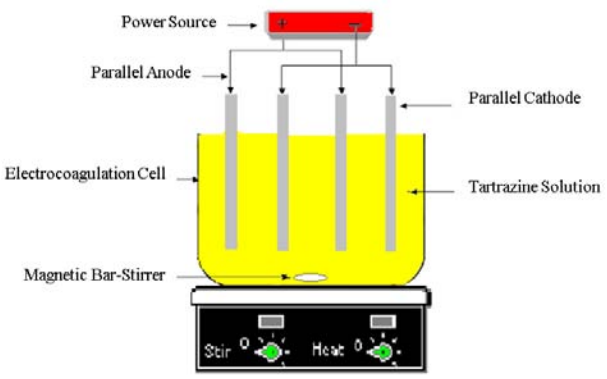

Figure 2 : Bench-scale EC reactor with monopolar electrodes in parallel connections

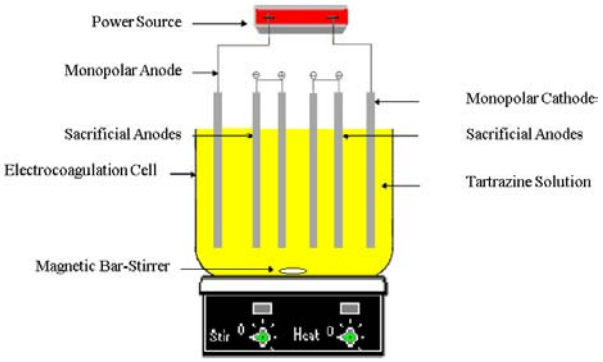

Figure 3 : Bench-scale EC reactor with monopolar electrodes in series connections

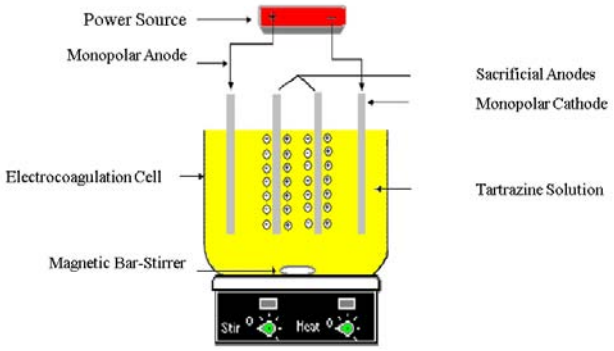

Figure 4 : Bench-scale EC reactor with bipolar electrodes in parallel connections

V. Method of Providing the Results

In all experiments, in order to measure the elimination percentage the following formula has been used:

$$CR\% = \frac{C_0 - C}{C_0} * 100$$

In which C_o is primary density C is the dye solution at the end of each experiment.

Study of the results would suggest the most efficient electro coagulation is achieved where in a cell containing mono polar electrodes in series formation with (sacrificial aluminum and iron anode and 304 steel cathode) iron is used as anode and aluminum as cathode or iron as anode and graphite as cathode the preference of iron anode over aluminum anode can be due following two reasons.

A: elimination process using aluminum anode is basically accomplished by electro coagulation, but with iron anode both electro coagulation and electro oxidization are involved.

B: the absorption capacity of pollutant on Aluminum hydroxide clots are less than iron hydroxide clots the result of experiment showing the effect of current density on AR88 elimination in an electrochemical cell in both series and parallel formations were compared and suggests that the series formation of monopolar electrodes is much more efficient in dye elimination and it can be because the series connected electrodes produce more resistance, thus, needs more potential difference to create current. More potential difference induces stronger field which consequently applies more power on the ions inside the field, as a result their velocity in reduction of oxidization and enhancement of AR88 ions will increase dye elimination efficiency.

In this formation of electrodes, more flocks are produced and aluminum hydroxide flocks are relatively large with less density and easily are floating and separated.

Table 1 : The effect of sodium chloride density on elimination efficiency

Time Density	0		2			4			6			8		
NaCl	A	C_{Dye}	A	C_{Dye}	CR%	A	C_{Dye}	CR%	A	C_{Dye}	CR%	A	C_{Dye}	CR%
100	1.296	42	0.795	20.78	52.75	0.452	9.40	78.09	0.045	0	100	0.02	0	100
200	1.274	41.33	0.515	12.30	72.98	0.428	8.70	79.45	0.052	0	100	0.015	0	100
300	1.281	41.54	0.741	19.15	56.22	0.621	14.52	65.20	0.15	0.25	99.50	0.019	0	100
400	1.251	40.63	0.849	42.42	46.95	0.427	8.64	79.22	0.052	0	100	0.008	0	100
500	1.249	40.60	0.799	20.90	50.74	0.539	12.11	70.62	0.047	0	100	0.016	0	100
600	1.280	41.51	0.766	19.90	54.34	0.466	9.92	76.78	0.063	0.36	99.20	0.013	0	100

Table 2 : the effect of current density on elimination efficiency

Density	A	C_{Dye}	CR%
40	0.754	22.51	47.54

60	0.381	7.34	84
80	0.075	0.98	98.58
100	0.015	0	100
120	0.03	0	100
140	No transition		

Table 3 : The effect of solution temperature on elimination efficiency

T(K)	288		293		298		303		308	
Time Electrolyze	A	CR%	A	CR%	A	CR%	A	CR%	A	CR%
2	0.89	39.07	0.947	41.51	0.784	53.56	0.858	57.08	0.858	57.08
4	0.562	69.95	0.528	72.47	0.440	78.72	0.495	82.61	0.045	100
6	0.016	100	0.521	100	0.015	100	0.028	100	0.025	100
8	0.011	100	0.012	100	0.009	100	0.008	100	0.013	100

Table 4 : The effect of electrodes distance on elimination efficiency

Distance(cm)	A	C_{dye}	CR%
0.5	0.115	1.8	96.25
0.75	0.079	0.92	98.97
1	0.037	0	100
1.25	0.042	0	100
1.5	0.052	0	100
1.75	0.055	0.08	100

Table 5 : The effect of electrolyses time on elimination efficiency

Time Electrolyze (min)	A	$C_{day}(mgL^{-1})$	CR%
0	1.473	41.32	0
2	0.515	12.30	81.98
4	0.466	9.91	85.65
6	0.052	0	100
8	0.014	0	100
10	0.008	0	100

Table 6 : The effect of AR88 primary density on the efficiency of elimination

Time electrode (min)	2			4			6			8		
Concentration mgL^{-1}	A	C_{Day}	CR%	A	C_{Day}	CR%	A	C_{Day}	CR%	A	C_{Day}	CR%
20	0.535	15.90	41.50	0.255	5.34	74.80	0.016	0	100	0.012	0	100
40	0.515	15.30	75.75	0.466	9.9	76.47	0.037	0	100	0.015	0	100
60	1.363	38	38.5	0.898	22.00	62.38	0.044	0	100	0.018	0	100
80	1.642	49.18	40.36	1.668	47.24	43.40	0.198	5.63	95.21	0.072	0.08	100

Table 7 : The effect of primary phone limitation efficiency

Electrolyze time(4 min)			6 min			8 min		
pH	A	CR%	pH	A	CR%	pH	A	CR%
2.4	0.038	12	2.35	0.005	100	2.64	0.005	100
2.41	0.193	89.65	3.4	0.084	96.98	3.68	0.011	100
4.55	0.288	84.43	4.85	0.208	79.97	5.69	0.023	100
5.65	0.496	68.75	5.85	0.329	78.28	6.69	0.015	100

Table 8 : The effect of electrodes on elimination efficiency

λ_{max}= 254		λ_{max}= 508			Cathode material	Anode material
A	A_0	CR%	$C_{Dye}(mg\ L^{-1})$	A		
1.223	0.841	69.17	13.33	0.449	Fe	Fe
0.398	0.778	95.02	3.39	0.122	Al	Al
0.448	0.848	98.8	0.89	0.072	Al	Fe
0.382	0.820	96.07	1.98	0.108	Fe	Al

0.46	0.853	99.68	0.19	0.049	C	Fe
0.430	0.866	94.32	2.34	0.129	C	Al

Table 9 : The effect of electrolyze time on elimination efficiency regarding wave length 254 nm using iron a node and Aluminum cathode.

$$(C_o[Dye] = 40\ mg\ L^{-1}, C_{NaCl} = 200\ mg\ L^{-1},$$

$$[i] = 100\ Am^{-2}, d = 1.5\ cm, Fe/Al\ Anode/Cathode)$$

A	Electrolyze time(min)
0.873	0
0.307	5
0.303	10
0.303	15
0.319	30
0.339	45
0.430	60
0.314	75
0.333	90
0.348	105
0.398	120
0.391	135
0.381	150

VI. Conclusion

Considering the results obtained from the experiments of AR88 electro coagulation. The following outcomes can be achieved:

1. Electro coagulation, in comparison with other water treatment facilities is cheaper and more efficient in dye elimination of solutions containing dye eliminator AR88.
2. Electrocoagulation does not need much chemicals.
3. Dye elimination rate depends on factors such as current density, time of electrolyze, solution primary density, solution primary pH, experimented solution conductivity, time connection, distance between electrodes, solution temperature, stiring, type of electrodes and their formation.
4. In this method where 500ml solution containing.

Nacl 40mg AR88+200mg in electro coagulation by an electrochemical cell with iron anode and aluminum cathode in pH of about 6.7 and sediment time of 5 minutes and current density of 100 Am^{-2}, temperature of 298^0k (24^0C) and electrodes 1.5 centimeters apart and 6 minutes electrolyses time 100 dye is eliminated, looks to be an appropriate method of treatment of water wastes containing AR88.

1. In AR88 solution coagulation ordinary salt is to be the best electrolyte.
2. In AR88 electro coagulation maximum absorption reduction is at wave lengths of 245nm and 508nm,in an electro coagulation cell with iron anode and Aluminum cathode, and iron anode and graphite cathode.

   In an electrocoagulation cell with iron anode and cathode, and aluminum sacrificial electrodes, in series formation and an electro coagulation containing iron electrodes and aluminum sacrificial electrodes in parallel formation shows maximum absorption reduction too.
3. Considering high effects of different factors such as temperature, sediment stay time on AR88 elimination efficiency, it seems dye elimination is mostly done by physical surface absorption and complex formation is less involved.

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- Paper Title should be of Font Size 24 with one Column section.
- Author Name in Font Size of 11 with one column as of Title.
- Abstract Font size of 9 Bold, "Abstract" word in Italic Bold.
- Main Text: Font size 10 with justified two columns section
- Two Column with Equal Column with of 3.38 and Gaping of .2
- First Character must be three lines Drop capped.
- Paragraph before Spacing of 1 pt and After of 0 pt.
- Line Spacing of 1 pt
- Large Images must be in One Column
- Numbering of First Main Headings (Heading 1) must be in Roman Letters, Capital Letter, and Font Size of 10.
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You can use your own standard format also.
Author Guidelines:

1. General,

2. Ethical Guidelines,

3. Submission of Manuscripts,

4. Manuscript's Category,

5. Structure and Format of Manuscript,

6. After Acceptance.

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Research letters: The letters are small and concise comments on previously published matters.

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(b) A brief Summary, "Abstract" (less than 150 words) containing the major results and conclusions.

(c) Up to ten keywords, that precisely identifies the paper's subject, purpose, and focus.

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(e) Resources and techniques with sufficient complete experimental details (wherever possible by reference) to permit repetition; sources of information must be given and numerical methods must be specified by reference, unless non-standard.

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(h) Brief Acknowledgements.

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References

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30. Think and then print: When you will go to print your paper, notice that tables are not be split, headings are not detached from their descriptions, and page sequence is maintained.

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Key points to remember:

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- Please note the criterion for grading the final paper by peer-reviewers.

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- To the point depiction of the research
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- Present a justification. Status your particular theory (es) or aim(s), and describe the logic that led you to choose them.
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- Describe the method entirely
- To be succinct, present methods under headings dedicated to specific dealings or groups of measures
- Simplify - details how procedures were completed not how they were exclusively performed on a particular day.
- If well known procedures were used, account the procedure by name, possibly with reference, and that's all.

Approach:

- It is embarrassed or not possible to use vigorous voice when documenting methods with no using first person, which would focus the reviewer's interest on the researcher rather than the job. As a result when script up the methods most authors use third person passive voice.
- Use standard style in this and in every other part of the paper - avoid familiar lists, and use full sentences.

What to keep away from

- Resources and methods are not a set of information.
- Skip all descriptive information and surroundings - save it for the argument.
- Leave out information that is immaterial to a third party.

Results:

The principle of a results segment is to present and demonstrate your conclusion. Create this part a entirely objective details of the outcome, and save all understanding for the discussion.

The page length of this segment is set by the sum and types of data to be reported. Carry on to be to the point, by means of statistics and tables, if suitable, to present consequences most efficiently.You must obviously differentiate material that would usually be incorporated in a study editorial from any unprocessed data or additional appendix matter that would not be available. In fact, such matter should not be submitted at all except requested by the instructor.

Content

- Sum up your conclusion in text and demonstrate them, if suitable, with figures and tables.
- In manuscript, explain each of your consequences, point the reader to remarks that are most appropriate.
- Present a background, such as by describing the question that was addressed by creation an exacting study.
- Explain results of control experiments and comprise remarks that are not accessible in a prescribed figure or table, if appropriate.
- Examine your data, then prepare the analyzed (transformed) data in the form of a figure (graph), table, or in manuscript form.

What to stay away from

- Do not discuss or infer your outcome, report surroundings information, or try to explain anything.
- Not at all, take in raw data or intermediate calculations in a research manuscript.
- Do not present the similar data more than once.
- Manuscript should complement any figures or tables, not duplicate the identical information.
- Never confuse figures with tables - there is a difference.

Approach

- As forever, use past tense when you submit to your results, and put the whole thing in a reasonable order.
- Put figures and tables, appropriately numbered, in order at the end of the report
- If you desire, you may place your figures and tables properly within the text of your results part.

Figures and tables

- If you put figures and tables at the end of the details, make certain that they are visibly distinguished from any attach appendix materials, such as raw facts
- Despite of position, each figure must be numbered one after the other and complete with subtitle
- In spite of position, each table must be titled, numbered one after the other and complete with heading
- All figure and table must be adequately complete that it could situate on its own, divide from text

Discussion:

The Discussion is expected the trickiest segment to write and describe. A lot of papers submitted for journal are discarded based on problems with the Discussion. There is no head of state for how long a argument should be. Position your understanding of the outcome visibly to lead the reviewer through your conclusions, and then finish the paper with a summing up of the implication of the study. The purpose here is to offer an understanding of your results and hold up for all of your conclusions, using facts from your research and generally accepted information, if suitable. The implication of result should be visibly described. Infer your data in the conversation in suitable depth. This means that when you clarify an observable fact you must explain mechanisms that may account for the observation. If your results vary from your prospect, make clear why that may have happened. If your results agree, then explain the theory that the proof supported. It is never suitable to just state that the data approved with prospect, and let it drop at that.

- Make a decision if each premise is supported, discarded, or if you cannot make a conclusion with assurance. Do not just dismiss a study or part of a study as "uncertain."
- Research papers are not acknowledged if the work is imperfect. Draw what conclusions you can based upon the results that you have, and take care of the study as a finished work
- You may propose future guidelines, such as how the experiment might be personalized to accomplish a new idea.
- Give details all of your remarks as much as possible, focus on mechanisms.
- Make a decision if the tentative design sufficiently addressed the theory, and whether or not it was correctly restricted.
- Try to present substitute explanations if sensible alternatives be present.
- One research will not counter an overall question, so maintain the large picture in mind, where do you go next? The best studies unlock new avenues of study. What questions remain?
- Recommendations for detailed papers will offer supplementary suggestions.

Approach:

- When you refer to information, differentiate data generated by your own studies from available information
- Submit to work done by specific persons (including you) in past tense.
- Submit to generally acknowledged facts and main beliefs in present tense.

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Introduction	Containing all background details with clear goal and appropriate details, flow specification, no grammar and spelling mistake, well organized sentence and paragraph, reference cited	Unclear and confusing data, appropriate format, grammar and spelling errors with unorganized matter	Out of place depth and content, hazy format
Methods and Procedures	Clear and to the point with well arranged paragraph, precision and accuracy of facts and figures, well organized subheads	Difficult to comprehend with embarrassed text, too much explanation but completed	Incorrect and unorganized structure with hazy meaning
Result	Well organized, Clear and specific, Correct units with precision, correct data, well structuring of paragraph, no grammar and spelling mistake	Complete and embarrassed text, difficult to comprehend	Irregular format with wrong facts and figures
Discussion	Well organized, meaningful specification, sound conclusion, logical and concise explanation, highly structured paragraph reference cited	Wordy, unclear conclusion, spurious	Conclusion is not cited, unorganized, difficult to comprehend
References	Complete and correct format, well organized	Beside the point, Incomplete	Wrong format and structuring

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