Infrared, Raman, thermal, kinetic modeling studies of mercury (II) ephedrine complex

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Abstract: The reaction between ephedrine HCl and $HgCl_2$ in 1:2 molar ratio in methanol was investigated at 70 ^oC. In normal conditions, the mercury(II) complex formulated as $[Hg(eph)_2](Cl)_2.2HCl$, was formed by chelating of mercuric chloride and ephedrine hydrochloride without pH adjustment. This complex was characterized by elemental analysis, infrared and Raman spectroscopy. IR spectrum of mercury complex show that ephedrine nucleus has two powerful donating sites –OH and –NH groups, so it is prefer to acts as bidentate ligand. The thermal stability mechanism of this complex was carried out by thermo gravimetric (TG) and differential thermo gravimetric (DTG) analysis which is facilitate to recognized the formation of this complex. The Coats-Redfern and Horowitz-Metzger relations were carried out to calculate the thermodynamic parameters. The bond angles, bond lengths, highest occupied molecular orbital (HOMO), the energy of the lowest unoccupied molecular orbital (LUMO) and energy gap of the studied complex were calculated using HyperChem software program.
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Key words: Ephedrine, Raman spectra, Thermal, Kinetics, Computational chemistry.

1. Introduction

Ephedra (Fig.1) is a Phanerogame-Gymnosperme from the family of Gnetaceen. There are 30 different types of this plant-species known which grow in Asia, Mediterranean countries and America. Specially Ephedra vulgaris, Ephedra equisetina and Ephedra sinica which contain ephedrin with its other isomers. A certain ephedra species has been used in ancient Chinese medicine since ages. Already in 5000 B.C. an ephedra plant was widely used in China under the name Ma-Huang. Ma-Huang has been mentioned as medicine of moderate therapeutic range in the first Chinese pharmacopoe, published under the government of Shen Lung in 1760 B.C. **A** detailed description of the plant and its pharmacological action is given in the Chinese pharmacopoe of modern times. The pharmacological studies have indicated that ephedrine is a sympathomimetic agonist at both α and β adrenergic receptors, which determine an increase of cardiac rate and contractility, peripheral vasoconstriction, bronchodilatation and central nervous system (CNS) stimulation [1]. Ephedrine is not the only alkaloid used in commercial products, since decongestant preparations usually contain pseudoephedrine. In recent years, the number of dietary supplements containing Ephedra, either as powdered botanical or, more frequently, as a standardized extract, had increased dramatically. Most of these products have been sold for the treatment of obesity or for increasing performance in body building. Often these dietary supplements also contained caffeine, either synthetic or from botanical extracts, in addition to other ingredients [2]. Weight loss and enhanced performance in body building may be due to the CNS stimulation and thermogenic properties of ephedrine [1]. However, severe contraindications have been given for individuals with hypertension or other cardiovascular diseases, glaucoma, diabetes and hyperthyroidism. Products containing E. sinica (or another botanical source of ephedrine) were among the most popular dietary supplements on the market, until their sale was banned by the U.S. Food and Drug Administration (FDA) in April 2004. After the ban of Ephedra products, “Ephedra-free” dietary supplements for weight loss were introduced. However, Ephedra-free is not necessarily danger-free [3]. Citrus aurantium is an ingredient in many of these Ephedra-free dietary supplements. The main active constituent of C. aurantium fruit extracts is (−)-synephrine [3], a phenethylamine alkaloid similar in structure to ephedrine. However, dietary supplements often contain C. aurantium in combination with concentrates of other herbs that are rich in caffeine and have the same potential to induce arrhythmia, hypertension, heart attacks and strokes as the combination of ephedrine and caffeine [3]. Depending on the oxidation stat of metal, the coordination number and the kind of coordinated ligand, there are many structures, which show

different biological and physico – chemical properties [4, 5]. The literature shows that there is a direct relationship between chemical structure and the antimicrobial properties of chemical compounds [6, 7]. Cui [6], for example, investigated a series of compounds containing NO groups and established that this group is exclusively responsible for the biological activity of some compounds. The author correlated the IR wave number of the valence vibration of the NO group with the antimicrobial activity of the compound. It was suggested that this parameter (the IR wave number) may be a good benchmark for determining the biological properties of compounds containing this group. Previously, the relationship between the chemical structure characterized by spectral parameters and antimicrobial activity was studied [5, 8, 9]. In this work, we prefer to throw a light on an essential compound as ephedrine to evaluate the probability of its interaction with mercury(II) ions upon the spectroscopic and thermal analysis tools. In addition, the bond lengths, bond angles and HOMO-LUMO energy gap were calculated with the employment of semi-empirical molecular orbital calculations. This paper is considered the initial point for the specialists in the medicinal field for condensed investigation.

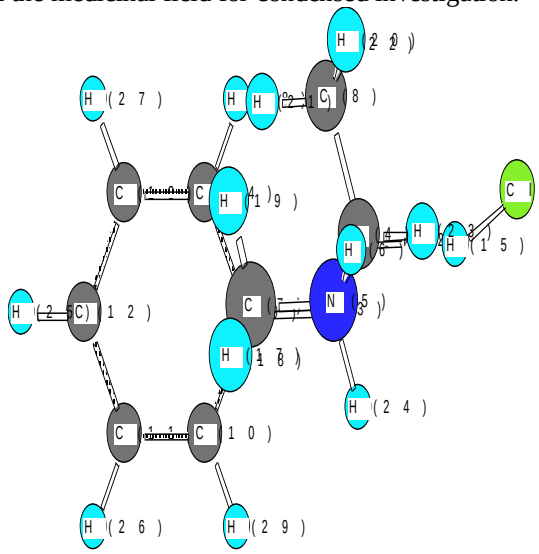


Fig. 1: 3D ephedrine (eph) structure with atom arrangement

2- Experimental

2-1- Reagents

Ephedrine (2-(methylamino)-1-phenylpropan-1-ol hydrochloride) used in this study was obtained from the Egyptian International Pharmaceutical Industrial Company (EIPICO). All other chemicals used in the preparations were of analytical reagent grade, commercially available from different sources (Fluka Co. and Aldrich Co.). Mercuric(II) chloride received from (Fluka Co.). All solvents are used as it is without further purification.

2-2- Synthesis of ephedrine mercuric (II) complex

The mercuric (II) complex was prepared by molar ratio 1: 2 (1 mmol of $HgCl_2$: 2 mmol eph) in methanol solvent. The resulted mixture was heated under reflux for 3 hr. The complex was separated from the reaction mixture and washed with boiling methanol and dried under vacuum over anhydrous $CaCl_2$.

2-3- Equipment and measurements

Carbon and Hydrogen content was determined at the Microanalytical Unit of Cairo University. The analysis of metal ions and their conjugated anion (chloride) were carried out according to standard methods [10]. IR spectra were recorded on a FT – IR 4100 Jasco, Japan spectrophotometer with (KBr – discs) in the υ = 4000 – 400 cm^{-1} range, while Raman laser spectra of samples were measured on the Bruker FT-Raman with laser 50 mW at Taif University. The molar conductivities of freshly prepared $1.0X10^{-3}$ mol/cm^3 DMF solutions were measured for the soluble mercuric complex as well as eph free ligand using Jenway 4010 conductivity meter. TG/DTG measurements are made in an N_2 atmosphere between room temperature and 800 ^oC using SCINCO DSC 1500 STA at Taif University. The initial geometry optimization was performed with HyperChem software (Version 7.5 Hypercube, Inc., Alberta, Canada), for free eph and its Hg(II) complex, the semi-empirical molecular orbital calculations have been employed. Two different methods are used for the calculation of kinetic parameters.

Horowitz and Metzger approximation method [11] derived the relation;

$$\ln\left[-\ln\left(1-\alpha\right)\right]=\frac{E}{RT_m}\Theta \qquad (1)$$

Where α, is the fraction of the sample decomposed at time t and $\Theta = T - T_m$. A plot of $\ln\left[-\ln\left(1-\alpha\right)\right]$ against Θ, was found to be linear, from the slope of which the energy of activation E, was calculated and pre-exponential factor Z can be deduced from the relation

$$Z=\frac{E\varphi}{RT_m^{\,2}}\exp\left(\frac{E}{RT_m}\right) \qquad (2)$$

Where φ is the linear heating rate, the entropy of activation ΔS, was calculated using equation (3).

$$\Delta S = R \ln\left(\frac{Zh}{K_B T_m}\right) \quad (3)$$

Where K_B, the Boltzmann's is constant, h is the Planck's constant and T_m, is the DTG peak temperature.

The order of reaction, n, can be calculated from the relation

$$n = 33.64758 - 182.295\alpha_m + 435.9073\alpha_m^2 - 551.157\alpha_m^3 + 357.3703_m^4 - 93.4828_m^5 \quad (4)$$

Where α_m is the fraction of the substance decomposed at T_m.

Coats and Redfern integral method, for first-order reactions, the Coats-Redfern [12] equation may be written in the form

$$\ln\left[\frac{-\ln(1-\alpha)}{T^2}\right] = \ln\left(\frac{ZR}{\varphi E}\right) - \frac{E}{RT} \quad (5)$$

A plot of $\ln\left[\frac{-\ln(1-\alpha)}{T^2}\right]$ versus $1/T$ was found to be linear that upon the slope of Coats-Redfern equation which E, was calculated and Z can be deduced from the intercept. The enthalpy of activation, ΔH, and the Gibbs free energy of activation, ΔG, can be calculated via the equations

$$\Delta H = E - RT_m \; ; \Delta G = \Delta H - T_m \Delta S \quad (6)$$

3- Results and Discussion

The carbon, hydrogen and nitrogen contents of white mercuric(II) ephedrine complex were performed and gave good agreement with calculated data. Yield: 73%; m.p. > 200 °C. calcd. Found; For $C_{20}H_{32}Cl_4HgN_2O_2$ (MW = 674.88 g/mol): calcd.: C, 35.59; H, 4.78; N, 4.15; Cl, 21.01 %. Found: C, 35.21; H, 4.54; N, 3.97; Cl, 20.85%. The molar conductance value of mercuric(II) ephedrine complex in DMF ($1.0X10^{-3}$ mol/cm^3) was measured at room temperature and the value is equal 240 ohm^{-1} cm^2 mol^{-1}. The comparison between the value of ephedrine free ligand and its mercuric(II) complex led us to concluded that Hg(II) complex has a electrolyte, this meaning that both of chlorine atom exhibited outside the coordination sphere.

A deliberate comparison between the significant band positions in between the ephedrine HCl free ligand with its relative mercuric(II) complex may give enough insight to elucidate the way of bonding of eph ligand towards the Hg(II) ion. Especially, with the absence of powerful technique such as X – ray crystallography. All spectra were carried in the range of 4000–400 cm^{-1} and the most significant bands are listed in Table 1. The spectrum of free ligand displays a series of significant bands as: 3330, 2972, 1591 and 1395 cm^{-1} which may assign to υ(OH), υ(NH), δ(NH) and δ(OH)- in plane bend. The lower appearance shinned on bands of OH and NH groups supports of the presence of intraligand H – bonding (Fig. 2) between the two neighboring groups. Ephedrine ligand bonded towards Hg(II) ion by mode of coordination through its two active (– OH and – NH) sites in neutral state (Fig. 3). This is expected due to the distribution of OH and NH groups, which primates this behavior as appeared from molecular modeling for the minimum internal energy structure (3.385 kcal/mol) by the use of MM$^+$ [13] force – field as implemented in hyperchem 7.5. The υ and δ OH in Hg(II) complex are more or less unshifted which may support its interaction after the decomposition of intraligand H – bonding. According to elemental analysis and thermogravimetric studies, the mercuric(II) complex resulted anhydrous not containing water molecules as crystallization. The new bands assigned for υ(M-N) and υ(M-O) are easily characterized in the low frequency field and υ(M-Cl) was detected in the scanning range of Raman spectrum. In Raman spectrum (Fig. 4), the 270 and 172 cm^{-1} bands are assigned to υ(Hg–Cl).

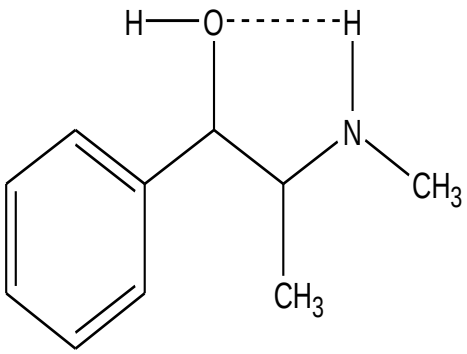


Fig. 2: The modeling structure of ephedrine hydrochloride

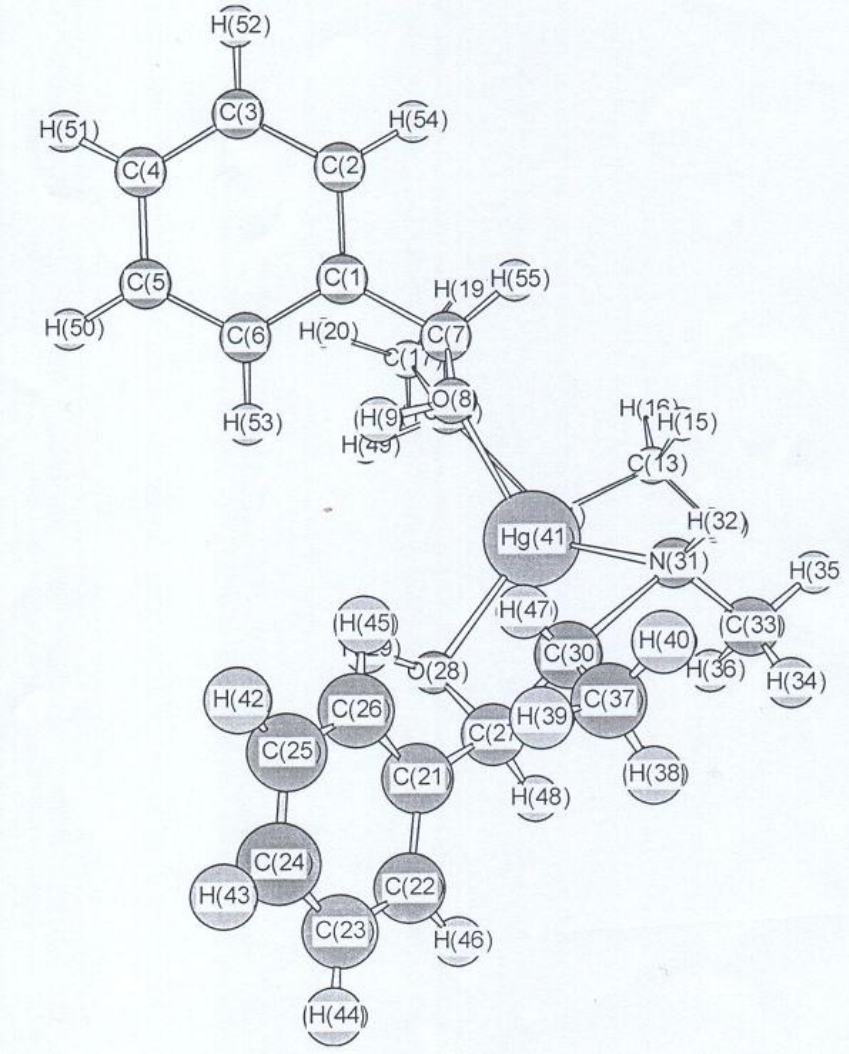


Fig. 3: Suggested structure of $[Hg(eph)_2](Cl)_2.2HCl$ complex

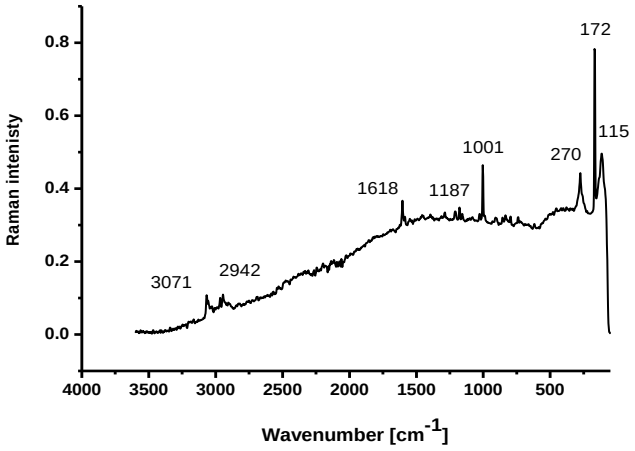


Fig. 4: Raman spectrum of mercuric(II) ephedrine complex

Simultaneous TG/DTG curves of $[Hg(eph)_2](Cl)_2.2HCl$ complex are shown in Fig. 5. The first mass loss (calcd. 59.78%; found 59.50%) between 25-325 ^oC corresponding to the endothermic peak at 300 ^oC is due to decomposition of both ephedrine HCl molecules. The second mass losses located in the wide range from 325-to-675 ^oC (calcd. 40.22%; found 40.50%) with three endothermic of T_{max}= 350, 500and 600 ^oC assigned to losses of one chlorine molecule and evaporated of mercury element which agreement with the physical behavior of mercury compounds. The thermal calculations based on the mass loss up to the final temperature are in agreement with the zero final residual. The kinetic parameters (Table 2) such as activation energy (ΔE*), enthalpy of activation (ΔH*), entropy of activation (ΔS*), free energy change of decomposition (ΔG*) were evaluated graphically by employing the Coats–Redfern relation [12] and Horowitz and Metzger integral method [11] (Fig. 6) for investigated $[Hg(eph)_2](Cl)_2.2HCl$ complex. The first decomposition step of mercuric (II) complex was discussed which negative entropy has indicated that this complex is formed spontaneously. The negative entropy also indicates a more ordered activated state that may be possible through the chemisorptions of oxygen and other decomposition products. The negative values of the entropies of activation are compensated by the values of the enthalpies of activation, leading to almost the same values for the free energies of activation [14].

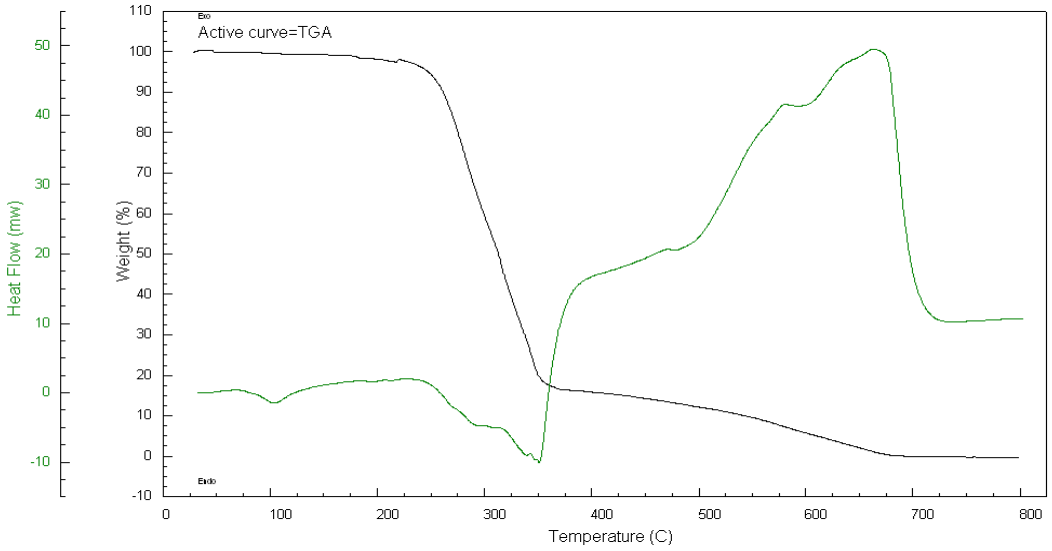


Fig. 5: TG/DTG curves of $[Hg(eph)_2](Cl)_2.2HCl$ complex

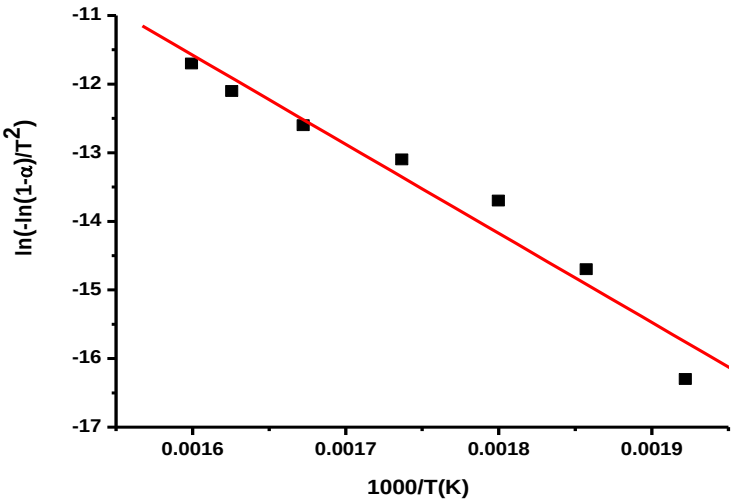


Fig. 6a: Coats–Redfern relation curve of $[Hg(eph)_2](Cl)_2.2HCl$ complex

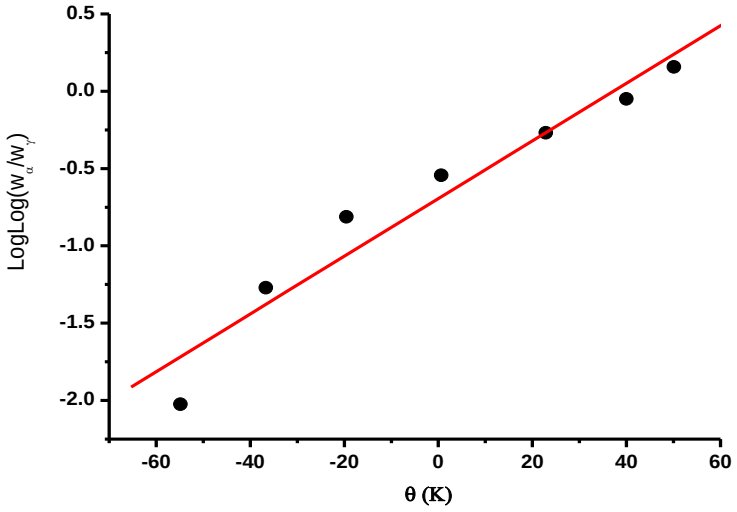
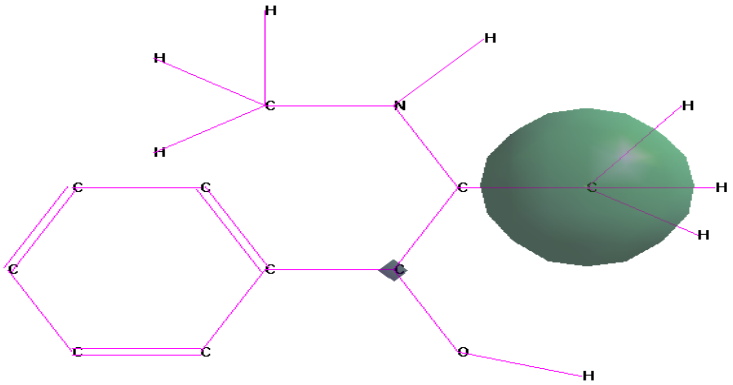
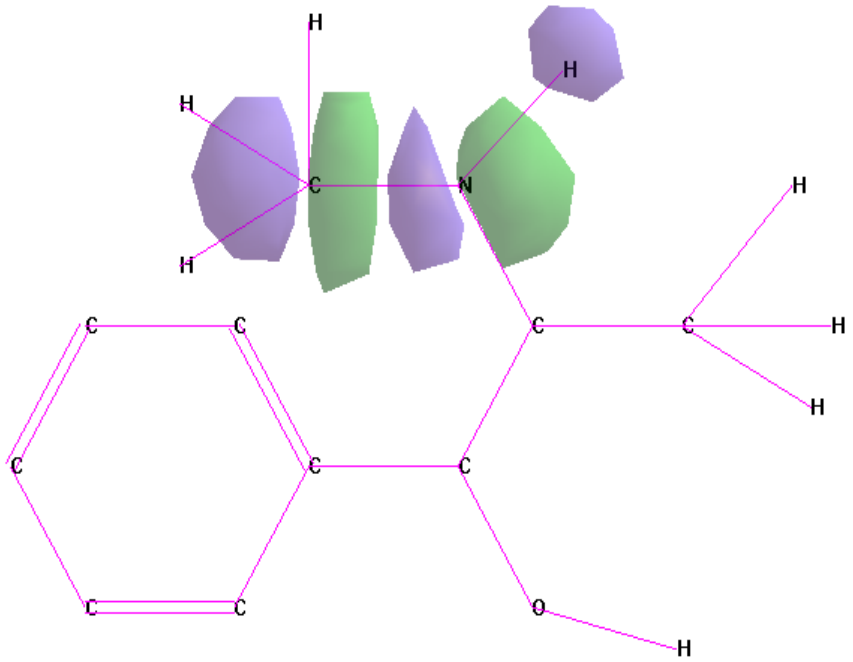


Fig. 6b: Horowitz and Metzger integral curve of $[Hg(eph)_2](Cl)_2.2HCl$ complex

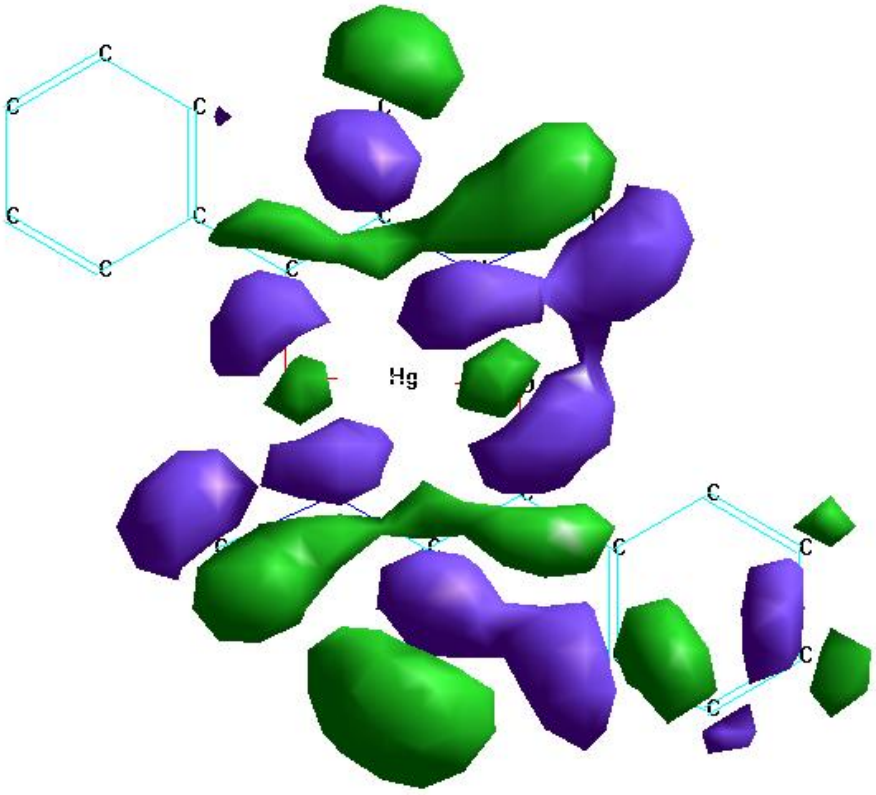
The geometric optimization is one of the theoretical tools help to recognize the place of chelation. The bond lengths and bond angles of the free eph ligand and studied Hg(II) complex were optimized (Tables 3 and 4). These Tables show the significant changes in bond lengths and bond angles for ephedrine free ligand comparable with Hg(II) complex, which supported the place of complexation. Formation of Hg-eph chelation cause displacement in the electron density of N-H and O-H bonds toward Hg(II) ion and makes these bonds weak and decreasing the bond lengths upon complexation state. In case of Hg(II) complex there are two new significant bond lengths exhibited at 2.13 and 2.166 which assigned to O-Hg and N-Hg bonds. Figure 7 refer to the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) for the free ephedrine ligand and its mercuric(II) complex. The energy gaps of eph ligand and Hg(II) complex are 12.51 and 16.07 eV, respectively, which reflect the chemical activity of the eph molecule. LUMO as an electron acceptor represents the ability to obtain an electron, while HOMO as an electron donor represents the ability to donate an electron. The smaller the LUMO and HOMO energy gaps, the easier it is for the HOMO electrons to be excited. The higher the HOMO energies, the easier it is for HOMO to donate electrons, the lower the LUMO energies, the easier it is for LUMO to accept electrons. A small gap means an unstable structure, unless no vibrational mode of the right symmetry exists for the molecule capable of changing its structure. A large energy gap between the HOMO and the LUMO means a stable molecular structure and interactions can occur, but only with high activation energy. This result proved the stability of Hg(II) complex rather than free ephedrine ligand.



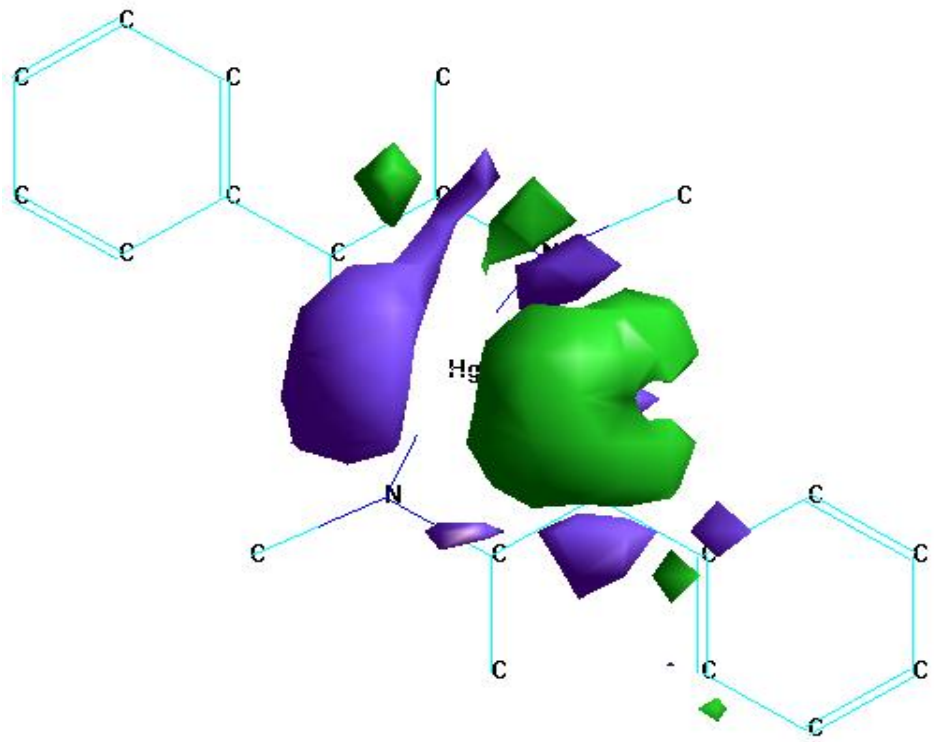
HOMO of eph free ligand = -5.693265 eV



LUMO of eph free ligand = 6.812272 eV



HOMO of eph-Hg(II) complex = -9.413819



LUMO of eph-Hg(II) complex = 6.658891

Fig. 7: HOMO-LUMO energies for ephedrine ligand and its mercury(II) complex

Table 1: Assignments of the IR essential spectral bands (cm^{-1}) of ephedrine and its $[Hg(eph)_2](Cl)_2.2HCl$ complex

Compound	ν_{OH}	ν_{NH}	δ_{OH} (outof plane)	ν_{C-O}	δ_{OH} (in plane)	δ_{NH}	ν_{M-Cl}	ν_{M-N}	ν_{M-O}
Ephedrine (eph)	3330	2972	751	1051	1395	1591	---	---	---
$[Hg(eph)_2](Cl)_2.2HCl$	3332	3031	745	1048	1455	1593	---	450	519

Table 2: Kinetic parameters using the Coats–Redfern (CR) and Horowitz–Metzger integral operated for the Hg(II) ephedrine complex at first decomposition step

Method	Kinetic Parameters					
	E ($Jmol^{-1}$)	A (S^{-1})	Δ S ($Jmol^{-1}K^{-1}$)	Δ H ($Jmol^{-1}$)	Δ G ($Jmol^{-1}$)	r
CR	1.18E+05	5.75E+08	-8.27E+01	1.13E+05	1.61E+05	0.9702
HM	1.08E+05	3.22E+07	-1.07E+02	1.03E+05	1.65E+05	0.9775

Table 3: Bond lengths and angles of ephedrine HCl free ligand

Atoms	Bond length (^oA)
O(1)-C(3)	1.421
O(1)-H(2)	0.942
C(3)-C(4)	1.514
C(3)-C(9)	1.497
C(3)-H(22)	1.111
C(4)-N(5)	1.453
C(4)-C(8)	1.523
C(4)-H(21)	1.113
N(5)-C(7)	1.453
N(5)-H(6)	1.02
C(7)-H(15)	1.113
C(7)-H(16)	1.113
C(7)-H(17)	1.113

C(8)-H(18)	1.113
C(8)-H(19)	1.113
C(8)-H(20)	1.113
C(9)-C(10)	1.42
C(9)-C(14)	1.42
C(10)-C(11)	1.42
C(10)-H(27)	1.1
C(11)-C(12)	1.42
C(11)-H(24)	1.1
C(12)-C(13)	1.42
C(12)-H(23)	1.1
C(13)-C(14)	1.42
C(13)-H(25)	1.1
C(14)-H(26)	1.1
Atoms	**Bond angle (deg)**
H(2)-O(1)-C(3)	106.898
O(1)-C(3)-C(4)	107.701
O(1)-C(3)-C(9)	109.5
O(1)-C(3)-H(22)	106.7
C(4)-C(3)-C(9)	109.51
C(4)-C(3)-H(22)	109.391
C(9)-C(3)-H(22)	113.838
C(3)-C(4)-N(5)	108.8
C(3)-C(4)-C(8)	109.509
C(3)-C(4)-H(21)	109.39
N(5)-C(4)-C(8)	108.8
N(5)-C(4)-H(21)	108.8
C(8)-C(4)-H(21)	111.493
C(4)-N(5)-C(7)	107.699
C(4)-N(5)-H(6)	109.47
H(6)-N(5)-C(7)	109.47
N(5)-C(7)-H(15)	108.799
N(5)-C(7)-H(16)	108.8
N(5)-C(7)-H(17)	108.8
H(15)-C(7)-H(16)	109.002
H(15)-C(7)-H(17)	109.002
H(16)-C(7)-H(17)	112.38
C(4)-C(8)-H(18)	110
C(4)-C(8)-H(19)	109.998
C(4)-C(8)-H(20)	109.998
H(18)-C(8)-H(19)	109.002
H(18)-C(8)-H(20)	109.002
H(19)-C(8)-H(20)	108.813
C(3)-C(9)-C(10)	121.399
C(3)-C(9)-C(14)	118.599
C(10)-C(9)-C(14)	120
C(9)-C(10)-C(11)	120
C(9)-C(10)-H(27)	119.998
C(11)-C(10)-H(27)	120
C(10)-C(11)-C(12)	120
C(10)-C(11)-H(24)	119.998
C(12)-C(11)-H(24)	119.998
C(11)-C(12)-C(13)	120.001
C(11)-C(12)-H(23)	119.998
C(13)-C(12)-H(23)	119.998
C(12)-C(13)-C(14)	120
C(12)-C(13)-H(25)	119.998
C(14)-C(13)-H(25)	119.998
C(9)-C(14)-C(13)	120
C(9)-C(14)-H(26)	119.998
C(13)-C(14)-H(26)	119.998

* Red color refer to the place of donation

Table 4: Bond lengths and angles of $[Hg(eph)_2](Cl)_2.2HCl$ complex

Atoms	**Bond length (^oA)**
C(1)-C(2)	1.42
C(1)-C(6)	1.42
C(1)-C(7)	1.497
C(2)-C(3)	1.42
C(2)-H(54)	1.1
C(3)-C(4)	1.42
C(3)-H(52)	1.1
C(4)-C(5)	1.42
C(4)-H(51)	1.1
C(5)-C(6)	1.42
C(5)-H(50)	1.1
C(6)-H(53)	1.1
C(7)-O(8)	1.41
C(7)-C(10)	1.523
C(7)-H(55)	1.113
O(8)-Hg(41)	2.13
O(8)-H(9)	0.992
C(10)-N(11)	1.906
C(10)-C(17)	1.523
C(10)-H(49)	1.113
N(11)-C(13)	1.446
N(11)-Hg(41)	2.166
N(11)-H(12)	1.028
C(13)-H(14)	1.113
C(13)-H(15)	1.113
C(13)-H(16)	1.113
C(17)-H(18)	1.113
C(17)-H(19)	1.113
C(17)-H(20)	1.113
C(21)-C(22)	1.42
C(21)-C(26)	1.42
C(21)-C(27)	1.497
C(22)-C(23)	1.42
C(22)-H(46)	1.1
C(23)-C(24)	1.42
C(23)-H(44)	1.1
C(24)-C(25)	1.42
C(24)-H(43)	1.1
C(25)-C(26)	1.42
C(25)-H(42)	1.1
C(26)-H(45)	1.1
C(27)-O(28)	1.41
C(27)-C(30)	1.523
C(27)-H(48)	1.113
O(28)-Hg(41)	2.13
O(28)-H(29)	0.992
C(30)-N(31)	1.899
C(30)-C(37)	1.523
C(30)-H(47)	1.113
N(31)-C(33)	1.446
N(31)-Hg(41)	2.166
N(31)-H(32)	1.028
C(33)-H(34)	1.113
C(33)-H(35)	1.113
C(33)-H(36)	1.113
C(37)-H(38)	1.113
C(37)-H(39)	1.113
C(37)-H(40)	1.113
Atoms	**Bond angle (deg)**
C(2)-C(1)-C(6)	119.998
C(2)-C(1)-C(7)	121.4
C(6)-C(1)-C(7)	118.599
C(1)-C(2)-C(3)	120
C(1)-C(2)-H(54)	120
C(3)-C(2)-H(54)	119.998
C(2)-C(3)-C(4)	120
C(2)-C(3)-H(52)	119.998
C(4)-C(3)-H(52)	119.998
C(3)-C(4)-C(5)	119.998
C(3)-C(4)-H(51)	119.998
C(5)-C(4)-H(51)	120
C(4)-C(5)-C(6)	120.001
C(4)-C(5)-H(50)	119.998
C(6)-C(5)-H(50)	119.998
C(1)-C(6)-C(5)	120
C(1)-C(6)-H(53)	119.998
C(5)-C(6)-H(53)	119.998
C(1)-C(7)-O(8)	109.47
C(1)-C(7)-C(10)	109.51
C(1)-C(7)-H(55)	109.388
O(8)-C(7)-C(10)	104.501
O(8)-C(7)-H(55)	109.472
C(10)-C(7)-H(55)	114.34

C(7)-O(8)-Hg(41)	104.501
C(7)-O(8)-H(9)	109.446
H(9)-O(8)-Hg(41)	109.516
C(7)-C(10)-N(11)	130.314
C(7)-C(10)-C(17)	109.51
C(7)-C(10)-H(49)	109.391
N(11)-C(10)-C(17)	109.472
N(11)-C(10)-H(49)	109.472
C(17)-C(10)-H(49)	75.07
C(10)-N(11)-C(13)	109.47
C(10)-N(11)-Hg(41)	76.486
C(10)-N(11)-H(12)	109.586
C(13)-N(11)-Hg(41)	109.47
H(12)-N(11)-C(13)	109.573
H(12)-N(11)-Hg(41)	135.266
N(11)-C(13)-H(14)	109.472
N(11)-C(13)-H(15)	109.47
N(11)-C(13)-H(16)	109.47
H(14)-C(13)-H(15)	109.002
H(14)-C(13)-H(16)	109.002
H(15)-C(13)-H(16)	110.409
C(10)-C(17)-H(18)	110
C(10)-C(17)-H(19)	110
C(10)-C(17)-H(20)	110
H(18)-C(17)-H(19)	109
H(18)-C(17)-H(20)	109.002
H(19)-C(17)-H(20)	108.811
C(22)-C(21)-C(26)	120
C(22)-C(21)-C(27)	120
C(26)-C(21)-C(27)	119.998
C(21)-C(22)-C(23)	120
C(21)-C(22)-H(46)	119.998
C(23)-C(22)-H(46)	119.998
C(22)-C(23)-C(24)	120
C(22)-C(23)-H(44)	119.998
C(24)-C(23)-H(44)	119.998
C(23)-C(24)-C(25)	120
C(23)-C(24)-H(43)	120
C(25)-C(24)-H(43)	119.998
C(24)-C(25)-C(26)	120.001
C(24)-C(25)-H(42)	120.001
C(26)-C(25)-H(42)	119.996
C(21)-C(26)-C(25)	120
C(21)-C(26)-H(45)	119.998
C(25)-C(26)-H(45)	119.998
C(21)-C(27)-O(28)	109.47
C(21)-C(27)-C(30)	109.51
C(21)-C(27)-H(48)	109.391
O(28)-C(27)-C(30)	104.499
O(28)-C(27)-H(48)	109.47
C(30)-C(27)-H(48)	114.34
C(27)-O(28)-Hg(41)	104.501
C(27)-O(28)-H(29)	109.498
H(29)-O(28)-Hg(41)	109.451
C(27)-C(30)-N(31)	130.554
C(27)-C(30)-C(37)	109.51
C(27)-C(30)-H(47)	109.391
N(31)-C(30)-C(37)	109.472
N(31)-C(30)-H(47)	109.47
C(37)-C(30)-H(47)	74.388
C(30)-N(31)-C(33)	109.47
C(30)-N(31)-Hg(41)	75.383
C(30)-N(31)-H(32)	109.363
C(33)-N(31)-Hg(41)	109.472
H(32)-N(31)-C(33)	109.349
H(32)-N(31)-Hg(41)	136.217
N(31)-C(33)-H(34)	109.47
N(31)-C(33)-H(35)	109.47
N(31)-C(33)-H(36)	109.47
H(34)-C(33)-H(35)	109.002
H(34)-C(33)-H(36)	109.002
H(35)-C(33)-H(36)	110.409
C(30)-C(37)-H(38)	109.998
C(30)-C(37)-H(39)	109.998
C(30)-C(37)-H(40)	109.998
H(38)-C(37)-H(39)	109.002
H(38)-C(37)-H(40)	109.002
H(39)-C(37)-H(40)	108.813
O(8)-Hg(41)-N(11)	104.499
O(8)-Hg(41)-O(28)	109.47
O(8)-Hg(41)-N(31)	109.47
N(11)-Hg(41)-O(28)	109.47
N(11)-Hg(41)-N(31)	119.274
O(28)-Hg(41)-N(31)	104.501

* Red color refer to the place of donation

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References

[1] Schaneberg B.T., S. Crockett, E. Badir and I.A. Khan, (2003). The role of chemical fingerprinting: application to Ephedra, Phytochemistry **62**, 911.

[2] Roman M.C., D. Gray, G. Luo and et al. (2004). Determination of Ephedrine Alkaloids in Botanicals and Dietary Supplements by HPLC-UV: Collaborative Study , J. AOAC Int. **87**, 1.

[3] Pellati F. and S. Benvenuti, (2007). Chromatographic and electrophoretic methods for the analysis of phenetylamine alkaloids in Citrus aurantium, J. Chromatogr. **A 1161**, 71.

[4] Lieberman R. L., A. Bino, N. Mirsky, D. A. Summers and R. C. Thompson, (2000). Synthesis, structure and magnetic properties of a chromium(III)nicotinamide complex $[Cr_3O(O_2CCH_3)_6(na)_3]^+$(na=nicotinamide)**,** Inorg. Chim. Acta **297**, 1.

[5] koczoń P., J. Piekut, M. Borawska and W. Lewandowski, (2003). Vibrational structure and antimicrobial activity of selected isonicotinates, potassium picolinate and nicotinate, J. Mol. Struct. **651**, 651 .

[6] Cui X., C. L. Joannou, M. N. Hughes and R. Cammack, (1992). The bacteriocidal effects of transition metal complexes containing the NO^+ group on the food -spoilage bacterium Clostridium sporogenes, FEMS Microbiol. Lett., **98** , 67 .

[7] Hueso- Urena F., M. Moreno – carretero, M. Romero – Molina, J. Salas – Peregrin, M. Sanchez – Sanchez, G. Alvarez de Cienfuegos – Lopez and R. Faure, (1993). Transition metal complexes with monodeprotonated isoorotic and 2-thioisoorotic acids: Crystal structure, spectral and magnetic study, and antimicrobial activity, J. Inorg. Biochem. **51** ,613.

[8] Koczoń P., J. Piekut, M. Borawska, R. Świslocka and W. Lewandowski, (2005). The relationship between chemical structure and antimicrobial activity of selected nicotinates, p-iodobenzoates, picolinates and isonicotinates, Spectrochim. Acta **A 61** , 1917 .

[9] koczoń P., J. Piekut, M. Borawska, R. Świslocka and W. Lewandowski, (2006). Vibrational and microbiological study on alkaline metal picolinates and o-iodobenzoates , Anal. and Bioanalytical chemistry **384**, 302.

[10] Vogel A. I., (1994). A Text Book of Quantitative Inorganic Analysis, Longman, London.

7/28/2012

[11] Coats A. W. and J. P. Redfern, (1964). Kinetic Parameters from Thermogravimetric Data 68, Nature **201** , 68.

[12] Horowitz H. H. and G. Metzger, (1963). A New Analysis of Thermogravimetric Traces, Anal. Chem. **35**, 1464.

[13] Allinger N. L., (1977). Conformational analysis. 130. MM2. A hydrocarbon force field utilizing V1 and V2 torsional terms, J. Am. Chem. Soc. **99,** 8127.

[14] Singh B. K., R. K. Sharma and B. S. Garg, (2006). Cobalt(II) complexes of biologically active glutathione: Spectroscopic and molecular modeling, Spectrochim. Acta **A 63**, 96.