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Thermal, Solid Conductivity and Antioxidant Study of New Organo selenium Compounds Based on 1,4-Bis(chloromethyl)-2,5 Dimethylbenzene

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Abstract

Of new selenium organ derivatives, two types of derivatives were prepared in two or three steps. In the first one, P-xylene reacted with p-formaldehyde and hydrochloric acid gave1,4-bis(chloromethyl) -2,5-dimethylbenzene which is transformed to a selencyanide compound by reacting with KSeCN under N2 atmosphere to give the corresponding new compounds. In the second type 1,4- bis(chloromethyl) -2,5-dimethylbenzene reacted with hydroxybenzaldehyde to give (Z)-3-(4-chloro-2,5-dimethylstyryl) phenol which reacted with KSeCN under N2 atmosphere to give the corresponding new compounds. All compounds were characterized by melting point, FTIR, elemental analysis, UV- Visible, 1H-NMR and13C-NMR and Mass spectra. Meanwhile, TG/DTA analysis of some of these ligands was conducted to evaluate the thermal stability, kinetic, and characteristic thermodynamic parameters. Antioxidant study showed that the prepared compounds under study have good efficacy for capturing the free radical DPPH, close to the activity of the standard antioxidant Vit. C, which was used in different concentrations.

Introduction

Chloromethylation of aromatic compounds are promising intermediate chemicals because of the easy transformation to many chemicals, such as Pharmaceutical, corrosion inhibitors and conductive compounds [1-3]. Friedel-Crafts halo methylation method and HCl/paraformaldehyde reaction are the most common methods used for the preparation of such intermediates [4]. Selenium is a crucial trace element that is naturally occurring with both nutritional and toxicological properties. Selenium is non-toxic to humans in low

concentrations, but it plays a significant role in a human's health, in addition to regulating many critical functions involving cells. Therefore, an adduct that releases selenium to the biological system at a steady rate acts as an effective pharmaceutical agent [5,6][4]. This is mediated by incorporating it into selenoproteins. Apparently, the effectiveness of selenium adducts as an anti-infective or antioxidant agents depends on the bioavailability of a selenium atom at the site of action [7]. A previous study showed that the compound [2-NC5H3(3- CONH2) Se]2 exhibited highest GPx activity and free radical scavenging ability [8].
In this study we will examine the thermal stability, solid conductivity and the antioxidant properties of several new organoselenium compounds based on 1,4-bis(chloromethyl)-2,5-dimethylbenzene

Experimental procedure

P-Xylene, selenium powder, hydrochloric acid, paraformaldehyde, potassium cyanide, hydroxyl benzaldehyde and all other chemicals and reagents were of analytical grade and used as received. Melting points were determined on digital melting point apparatus and uncorrected. 1H and 13NMR spectra were recorded on a BRUKER-500MHz spectrometer using DMSO-d6 as the solvent with tetramethylsilane (TMS) as an internal standard. GC-MS spectra were recorded on HP6890 Gas Chromatograph with a HP5973 Mass Spectrometric Detector. The measurement of conductivity has been done using four probe control unit method. The prepared compounds were dissolved in DMSO then evaporated on a plate of aluminum. The antioxidant activity of the prepared compounds under study is expressed by the percentage of free radical capture compared to the reference solution of Ascorbic acid (Vit.C) according to the following equation.

$$\%\, Scavenging = \frac{\text{A of control} - \text{A of sample 1}}{A\ of\ Control} * 100$$

Where A is the absorbance

Syntheses of 1,4-bis(chloromethyl)-2,5-dimethylbenzene

All the three-necked flasks were loaded with p-Xylene (10.0 g), formalin solution (75 ml), 16 mL of concentrated hydrochloric acid (105 ml). The reaction mixture was refluxed after a five-hour stirring. A yellow oily layer is formed on the surface, the mixture is then poured in a 500ml beaker which is left for 24 hours in a refrigerator to cool down. The organic phase was combined and rinsed with saturated NaHCO3 solution (2 × 20 mL) and water (2 × 20 mL). The organic phase was dried with sodium sulfate, filtered and washed several time with methanol. A white precipitate is formed with m.p.(131-132) °C and a yield of 70%.[8]

Synthesis of 1,4-dimethyl-2,5-bis (selenocyanatomethyl) benzene (1)

A mixture of (0.133g; 0.04mol) (95%) KCN and (0.42g; 0.04mol) selenium powder in 25ml of distilled DMSO (as a solvent) was refluxed with stirring under argon atmosphere for about three hours. During this time, the

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solution turns deep purple then became pale yellow. A solution of 1,4-bis(chloromethyl) -2,5-dimethylbenzene (0.70g; 0.04mol) in 15 ml of DMSO was added gradually. A yellow solution is formed after about 15 minutes. The solution is filtered and poured in 300 ml of distilled water, left at room temperature for 24 hours, then extracted with three portions of 15 ml diethyl ether, The solvent was evaporated by a rotary evaporator, a yellow solid product formed, recrystallized from ethanol. Precipitate is formed with m.p. (110-112) °C and a yield of 60.8 %.[9]

C.H.N. C 42.2 0(42.12) ; H 3.64(3.54) ; N 7.70(8.19) ; Uv.V.(ύ1 = 38462cm-1 ; ύ1 = 40241cm-1) ; IR ύ$C \equiv N$ = 2144 cm-1 ; ύ$C - H$ ale = 2924., 2862 cm-1 ;ύ$C - H$ aro = 3009 cm-1 ; ύ$C = C$ =1500. 1450 cm-1 ύ$Se - C$ = 451cm-1H1NMR 7.23(s), 7.03 (s))ph(;4.27-4.73 (s,s) H9, H10; 2.37 (s) H7, H8 13C NMR 136.51(2,5) ; 135.24 (3,6) ; 132.53 (1,4) ; 45.16 (9, 10) ; 18.14 (7,8)

((2,5 - dimethyl-1,4-phenylene) bis(methylene)) bis (di (l2-iodanylidene) iodo-l6-selane) (2)

A solution of (0.32g; 0.001mol) iodine in 7.5 ml of chloroform was added gradually to a solution of two hours under argon atmosphere. A deep brown precipitate formed, filtered and recrystallized from ethanol/chloroform (1: 1). Reddish-brown crystals are isolated with m.p. (80-82) oC as a yield of 98 %.

C.H.N.C11.42(12.3) ; H1.15(2.11) ; Uv.V.(ύ1 38759cm-1 ; ύ1 = 35651cm-1 ;ύ1=293100cm-1); IR 1 ; ύ$C - H$ ale = 2924., 2858 cm-1 ;ύ$C - H$ aro = 3009 cm-1 ; ύ$C = C$ =1500. 1450 cm-1 ύ$Se - C$ = 455 cm-1 Se-I=574cm-1. 1H NMR (υ ppm) 7.23(s) , 7.10 (s))ph(;3.86-4.73 (s,s) H9, H10; 2.30 (s) H7, H8 13C NMR 136.60(2,5) ; 135.26 (3,6) ; 132.78 (1,4) ; 45.17 (9, 10) ; 18.14 (7,8).

2-bis(4-(chloromethyl)-2,5-dimethylphenyl) diselane (3)

In a two neck round bottom flask, place (0.355gm,4.5mmole) of selenium powder and (0.25gm,6.5mmole) of NaBH4 and 12.5 ml of ethanol, the mixture was refluxed for six hours, cooled to room temperature then (0.25gm,6.5mmol) of 1,4- bis(chloromethyl) -2,5-dimethylbenzene was added with a continuous stirring for 15 min. The solution poured in a 100 ml of distilled water and stirred for 15 min., then extracted with (3×50ml). The organic layer has been extracted, solvent evaporated, red crystals are formed, washed with water m.p. (172-174) °C and a yield of 83.5 %.

C.H.N.48.70(47.91) ;H4.91(4.96); UV/Vis. .(ύ1 38986cm-1 ; ύ1 = 27397cm-1 ; IR 1 ; ύ$C - H$ ale = 2924., 2858 cm-1 ;ύ$C - H$ aro =2997 cm-1 ; ύ$C = C$ =1500. 1438 cm-1 ύ$Se - C$ = 451.36 cm-1 C-Cl=675cm-1. 1H NMR (υ ppm) 6.80 (s) ,6.60 (s))ph , ph' (;3.60 (s) H9, 9' , H10, 10'; 2.05 (s) H7,7' , H8,8'

(E)-2-(2,5-dimethyl-4-(selenocyanatomethyl) styryl) phenol (4)

The First step included the preparation of 2-(4-(chloromethyl)-2,5-dimethylstyryl) phenol according to literature. In the second step a mixture of (0.66g; 0.01mol) (95%) KCN and (1.27g; 0.01mol) selenium powder in 25ml of distilled DMSO (as a solvent) was refluxed with stirring under argon atmosphere for about three hours, during this time the solution turns deep purple then becomes pale yellow. A solution of 2-(4-(chloromethyl)-2,5-

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dimethylstyryl) phenol (0.84gm,0.0024mmole) in 15 ml of DMSO was added gradually, a yellow solution is formed after about 15 minutes. The solution is then filtered and poured in 300 ml of distilled water, left at room temperature for 24 hours, extracted with three portions of 15 ml chloroform, evaporated reddish-brown solid is formed, and recrystallized from ethanol. Precipitate is formed with m.p. (152-154) ºC and a yield of 79% is realized.

C.H.N. C 63.61(62.64); H 5.01(5.35) N 4.09(3.22)) UV/Vis. (ύ1 40161cm-1; ύ2 38911cm-1; ύ3 = 27894cm-1) IR 1; ύ$C - H\ ale$ = 2924., 2858 cm-1; ύ$C - H$ aro =3063 cm-1; ύ$C = C$ =1593. 1442 cm-1 ύ$Se - C$ = 536 cm-1 OH 3163 cm-1; 1H NMR (υ ppm) 9.84 (s) OH; 6.5 - 7.93 (m) 2ph (groupes); 3.37 (s) H10, H11; 1.2 - 2.62 (s) H7, H8 13C NMR 155.46 (17) ; 131.91(16) ; 131.91(13,15) ; 130.66 (9, 10); 129.40 (3,6).

(E)-2(2,5-dimethyl-((triiodo-ƛ4-selenyl)methyl)styryl) phenol (5).

A solution of (0.64gm,5 mmole) of iodine (I_2) in 15 ml of chloroform was added to (0.32gm,1.00mmole) of compound (4) and dissolved in 20 ml of chloroform. The mixture is stirred at R.T. for two hours under argon atmosphere. A deep orange solid is formed, filtered, dissolved in ethanol/chloroform (1:1), evaporated of solvents, given a non-crystalized solid that yields 46%.

C.H.N. C 29.30(62.64) ; H 2.46(2.48)) ; Uv.V.(ύ1 33841cm-1 ; ύ1 = 27510cm-1) ; IR; ύ$C - H\ ale$ 2858, cm-1 ;ύ$C - H$ aro = 2916.47 cm-1 ; ύ$C = C$ =1593- 1435cm-1 VOH=3433.4cm-1ύSe-I=536cm-1 ύSe-C=466-435cm-1 1H NMR (υ ppm) 7.23(s) - 7.03 (s)) ph(;4.27-4.73 (s,s) H9, H10; 2.37 (s) H7, H8

(E)-2-(2,5-dimethyl-4-((tribromo-ƛ4-selanyl)methyl)styryl) phenol (6)

A solution of (0.79gm,0.01 mole) of bromine (Br_2) in 15 ml of chloroform was added to (0.64gm,0.0018 mole) of compound (4) and dissolved in 20 ml of chloroform. The mixture is stirred at R.T. for two hours under argon atmosphere. A pale-yellow solid is formed, filtered and recrystallized from ethanol/dichloromethane (1:1) with m.p.(240-242)ºC and a yield of 70 %.

C.H.N. C36.73(35.63); H3.08(3.25); Uv.V.(ύ1 39139cm-1 ; ύ1 33003cm1-;ύ1=31696cm-1 ; IR; ύ$C - H\ ale$ 2916, cm-1; ύ$C - H$ aro 3448 cm-1 ; ύ$C = C$ =1504- 1458cm-1 VOH=3506.6cm-1ύSeBr=543.9cm-1 ύSe-C=489-455cm-1 1H NMR (υ ppm) 7.23(s) - 7.03 (s))ph(;4.27-4.73 (s,s) H9, H10; 2.37 (s) H7, H8

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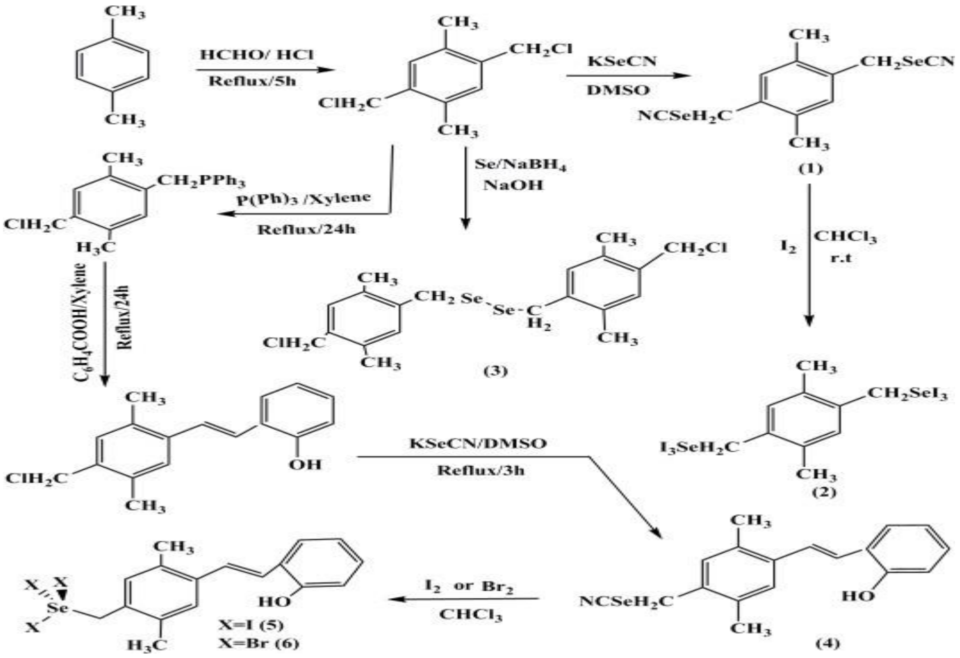

Scheme (1) Preparation pathway for organoselenium compound

Results and discussion:

Two different series of new compounds are reported in this paper. The first series are based on ortho-substituted p-xylene, while the second series contain phenolic group, in addition to p-xylene group moiety. The first series were prepared by the reaction of 1,4-bis(chloromethyl)-2,5-dimethylbenzene with KSeCN. The second series were prepared from reaction of 2- (4-(chloromethyl)-2,5-dimethylstyryl) phenol with KSeCN, Scheme 1. Both series were converted to trihalo by reacting compounds 1 and 2 with iodine and bromine, respectively, Scheme 1. Reaction of 1,4-bis(chloromethyl)-2,5- dimethylbenzene with Na2Se gave the corresponding diselenide (3), Scheme 1. In general, all prepared compounds were obtained in good yields and given satisfactory CHN analysis, see Experimental Section. Uv-Visible Spectra for prepared compounds were recorded for a DMSO solutions with 1×10-5 - 1×10-4 M concentrations in a range of 200-800nm. All compounds showed two types of bands. The first one was shown at wave number 38462- 40161cm-1. These bands may appear due to π-π* in phenyl or phenol groups, while the other bands appeared at 27510- 33003 cm-1 that may be attributed to n-π* in cyanide group or charge transfer from filled p orbitals in bromide or iodide to empty d orbitals in selenium atom. Infrared Spectra of new compounds gave the first evidence about expected structures.

Strong bands appeared in the range (3136-3506 cm-1) for compounds 4,5 and 6 refers for OH group in phenolic compounds, while selenocyanate compounds showed stretching vibration at 2144 cm-1 for triple bond in cyanide group. The disappearing of this band in compounds 2,3 and 4 gave the most characteristic evidence for conversion of this group to bromide and iodide selenium salts. On the the other hand, the bands appeared at

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(1450-1500 cm-1) for C=C, (2924.18-2858.6 cm-1) due to aliphatic C-H vibrations and 3009- 3080 cm-1 for aromatic C-H stretching vibrations.

^{1}H and ^{13}CNMR Spectra

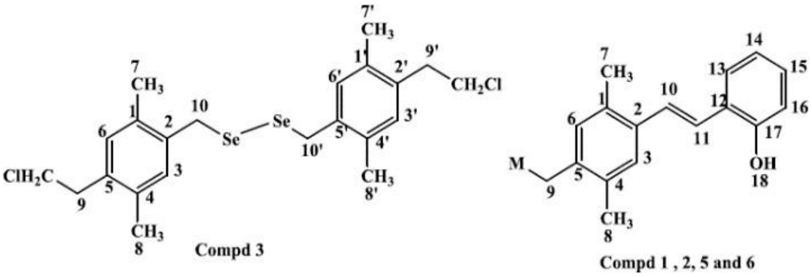

Figure (1) Position of 1H and 13CNMR in compound

1H NMR study of new compounds show expected bands for aromatic systems in p-xylene and phenolic compounds at chemical shift range (6.60 -7.93) ppm, while the phenolic proton appeared as a single band at 9.84 ppm. Aliphatic protons gave bands at expected regions 2.30 and 4.27-4.73 ppm depending on the electronegativity of neighboring groups. 13CNMR Spectra also gave good evidence for proposed structures. Thus, aromatic carbons gave bands at 132-136.60 ppm, while aliphatic carbons gave bands at 45.17 and 18.14.

The spectrum of compound 2 showed a signal at m/z (513.86) with intensity of 25% due to the formation of (C8H6Se2I2) and another signal at m/z (134.22) that refers to C10H14 after the loosing of Se2I2 group, while the band with intensity of 72% appeared at (105.17) due to aromatic fragment with C8H10 formula. Compound 3 spectrum showed a signal at m/z (339) due to the loss of SeI group and the formation of (C10H13SeI), and another signal at m/z (325) refers to C9H11SeI, while the band with the high intensity appeared at (592.89) due to the fragment with C10H13SeI3 formula. Compound 4 spectrum showed a signal at m/z (324.29) due to the formation of (C18H15NSe) and another signal at m/z (250.20) refer to C13H11NSe formula. Compound 5 spectrum showed a signal at m/z (1014.29) that may appear due to coupling of two molecules with the loosing of iodide ion and the formation of (C34H35I3OSe) and another signal at m/z (578.86) refer to C9H11SeI3, while the band with the high intensity appeared at (571.19) due to the fragment with C17H18SeOI2 formula.

The spectrum of compound 6 also showed a signal at m/z (952.18) that may be due to coupling of two molecules and the formation of (C34H34Br4O2Se2) and another signal at m/z (499.51) refer to C30H28O2Se, while the band with the high intensity appeared at (199.16) due to the fragment with C9H12Se formula.

Determination of the compound’s efficacy as antioxidants:

The antioxidant activity of compounds containing selenium and tellurium has led researchers to focus heavily on developing these compounds as a treatment or disease prevention. The 1,1-diphenyl-2-picrylhydrazyl (DPPH) free radical capture method is a widely applied method for evaluating the activity of antioxidant compounds (2,3). According to figure (2), a number of selenium and tellurium compounds have been prepared that have the ability to capture the free radical DPPH as shown in table (1).

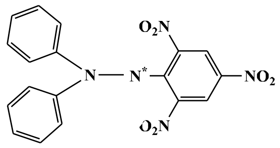

Figure (2): shows the chemical structure of the free radical DPPH Diphenylpicrylhydrazyl.

The antioxidant activity of the compounds prepared under study is expressed by the percentage of free radical capture compared to the reference solution of Ascorbic acid (Vit. C) according to the following equation:

$$\%\, Scavenging = \frac{\text{A of control} - \text{A of sample 1}}{A\ of\ Control} * 100$$

Where A is the absorbance

Table (1) The ability of the prepared compounds to capture the free radical DPPH.

Conc.	Vit. C	1	2	4	7
25mg/ml	93.57%	94.6%	67.24%	59.3%	45%
12.5mg/ml	93.47%	91.68%	59.3%	57.6%	43%
6.25mg/ml	93.4%	90.1%	54.4%	57.2%	38%

The effectiveness of DPPH free radical capture depends on the difference in the composition and kinetics of the prepared compounds that have the ability to reduce DPPH and according to the following equations:

$$\text{DPPH} + \text{AH} \longrightarrow \text{DPPH-H} + \text{A}^-$$

$$\text{DPPH}^- + \text{R}^- \longrightarrow \text{DPPH-R}$$

The course of the reaction is monitored by measuring the absorbance, as the change in the dark violet color to the yellow color of the DPPH free radical can be measured at a wavelength of 517 nm. As the results have shown that compound no. (1) Possesses the highest capacity of 94.06% as an antioxidant among the compounds, then it is followed by compound no. (7) have less efficacy. From table (1) we conclude that the antioxidant compounds prepared under study have good efficacy for capturing the free radical DPPH, close to the activity of the standard antioxidant Vit. C, which was used in different concentrations (6,7) .

Electrical conductivity study

The electrical conductivity of some of the compounds, 3 and 5, were studied for their resistivity value of a layer of an electronic material by thin film method. Thus, four-point probe measure (FPP) was used as a commonly tool to measure resistivity value 9. From current-voltage characteristics relationship, the samples was evaluated by using casting method to prepare studied compounds, the study have been done using four probe point measuring technique according to following equation:

$$\delta dc = \frac{DI}{AV}$$

Where δ_{dc} resistivity, D is thickness of film, I is the current on the circuit, A is the area of electrode and V is the applied voltage on the film.

Figure (3) shows the intrinsic structure-property relationships between current versus voltage that leads to both linear and nonlinear behavior. The ohmic behavior noticed at 60 volt at all the sample can be explained by the fact that there negligible injection of charge carriers from outer probes supply current to the sample film, because the thermally generated charge carriers are greater than the injected charge from electrodes. The deviation from ohmic behavior could be shown at greater voltage than 40 V with another suggested mechanism that clarifies charge transfer in sample film the same behavior that was detected for another system.

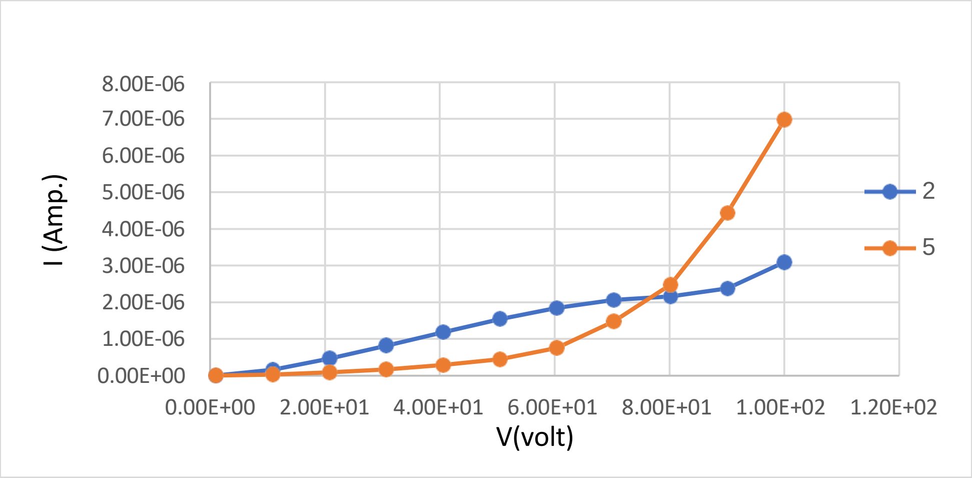

Figure (3) Relationships between current versus voltage that lead to both linear and nonlinear behaviour in 25^{o}C.

Figure (4) shows the relationship between current-voltage of sample at the temperature range (303-323) K. According to formed shapes, two different regions were detected. At low voltage of less than 40 v ohmic behaviors were observed for all temperature. The region up to temperature 303K shows a drastic voltage which may be explained by the relative disorder level present in the material, which in turn determines the amount of localized states. The current increase with increasing temperatures and the applied voltage is identical to the characteristic semiconductor material. When the temperatures increase, more localized electrons have the energy to bridge the gap, and the system becomes more mobile of charge carriers [6]. On the other hand, the molecular structure of the compound greatly influences the charge carriers mobility according to the (I-V) characteristics.

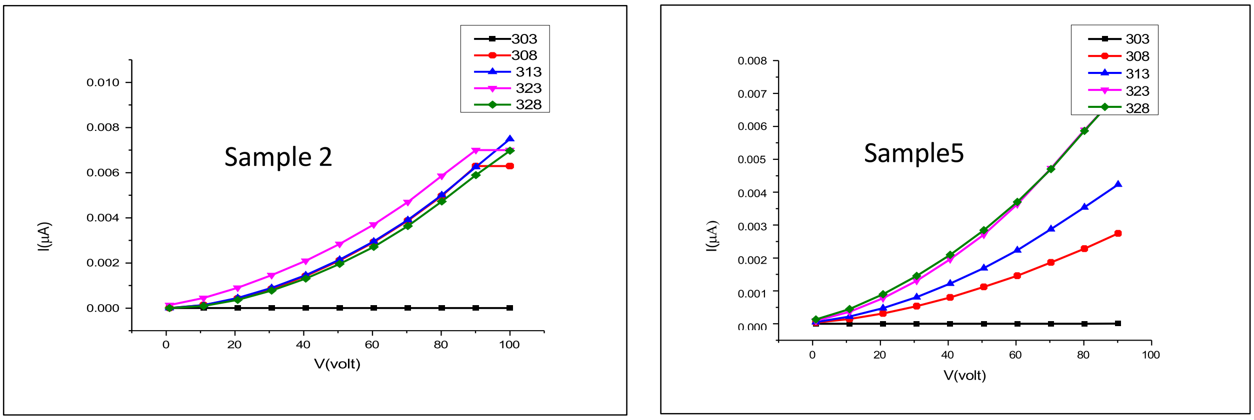

Figure (4) relationships between current versus voltage at different temperatures for 2 and 5sample

In semiconductors, the conductivity is thermally activated and it typically follows an arrhenius relationship:

$$\sigma = \sigma_o \ e^{\left(\frac{-E_a}{K_\beta T}\right)}$$

Where σ_o is perfector, E_a is the activation energy K_β is the Boltzmann constant and T is the absolute temperature. The sharp increase of conductivity can be attributed to large heat energy absorbed by the sample and thus induced mobility of electrons. However, in figure (5) show the electrical conductivity increases linearly [7]

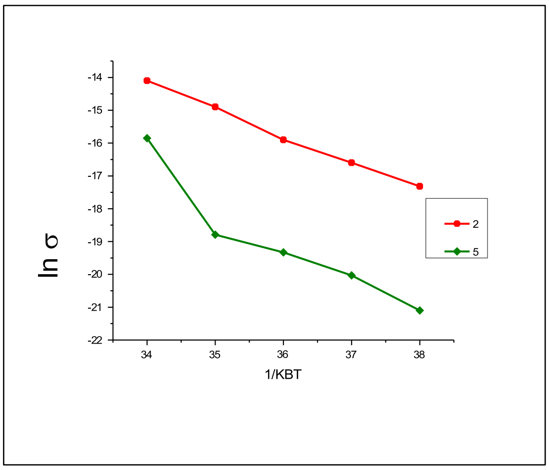

Figure (5) relationship between conductivity with different temperatures

Thermogravimetric analyses

It is clear from the graphics obtained from the thermos gravimetric analysis data that the phase transitions are subject to a first-order equation, where the data followed a straight line for each phase, and the slope of the straight line gave a constant rate of reaction rate, and the half-life was determined, from which the activation energy and thermodynamic parameters were calculated (ΔH,ΔS and ΔG) in table (2).

Table (2) kinetic and thermodynamic parameters

SAMPLE	K min-1	t1/2 min	Ea (J.mol-1) x103	ΔH (J.mol-1)x103	ΔS (J.mol-1.K-1)	ΔG (J.mol-1)x104
BH1	0.10972	6.317	21.59698	12.671	- 1016.2	110.3622
BH2	0.08423	8.229	16.14237	7.2168	-258.4	28.46093
BH3	0.10204	6.792	19.90069	10.976	- 253.6	28.32346
BH7	0.11569	5.991	15.12371	6.1990	-1023.2	109.8392

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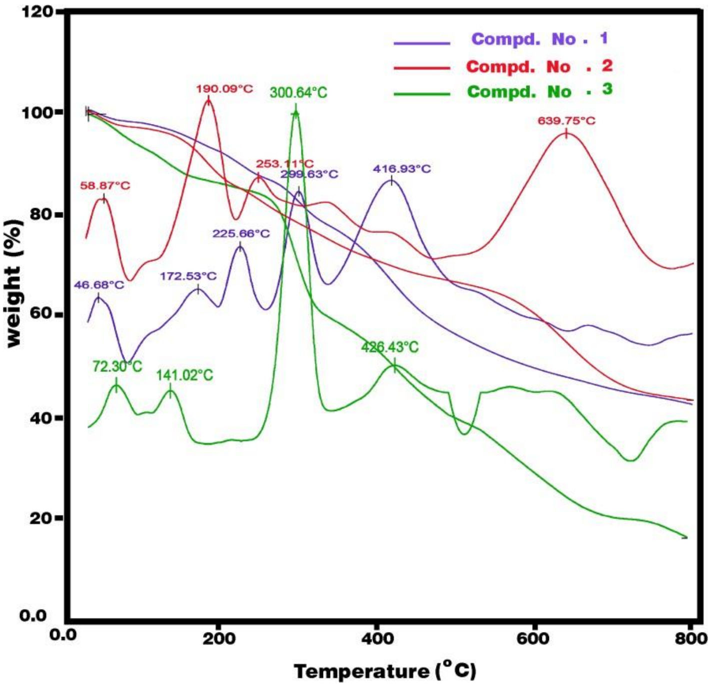

Figure(6) Thermogravimetric analyses of compounds

Conclusion:

By studying the electrical conductivity of sample 2 and 5 at room temperature, and in a range of temperatures, the results show an increase in conductivity with increasing temperature to 323K after which the linear direction deviates from it, i.e., it behaves as a non-ohmic, which proves its behavior as semiconductors.

On the other hand, it was observed that the activation energy of sample 2 (0.814 ev) is less than the activation energy of sample 5 (1.174 ev). We conclude that the energy barrier between the valance band and conductance band changes according to the composition of the organic compound which Se is linked. Hence, these compounds can be used in electronic, light-emit, diodes and solar cells.

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