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Synthesis and investigation and microbial activity of open chain of organo– selenium compounds

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Abstract - The target organo –selenium compounds [3-7] were synthesized from the reaction between diethyl malonate and 4-amino benzoyl chloride to produce compound [1], which is react with sodium hydrogen selenide to give corresponding sodium aroylselenide [2], which is react with one of aroyl derivatives as shown in scheme (1) to produce compounds [3-7]. All the synthesized compounds have been investigated using different chemical techniques, such as (H.NMR–spectra, (C.H.N)-analysis, FT.IR–spectra), melting points and biological study.

Key words - Organo selenium, Sulphur, Selenium

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The structures of organo- selenium compounds are mentioned in the literature as being similar to those of organo-sulphur compounds, but their properties present significant differences, because of their toxicity and their extremely unpleasant odour organo-selenium compounds have been relatively little explored.

Organo-selenium compounds have been tested as antifungal, antibacterial, antiparasitic, ant-inflammatory, antihistamine, anticancer agents[1-3], industrial, pharmaceutical applications[4-9], antidandruff hair shampoos, small quantities of selenium compounds are used as human dietary supplements, dyes applications[3] or as aligands and other uses[10].

EXPERIMENTAL METHODOLOGY

– All chemical used were supplied from Fluka and BDH-chemical company.

– All measurements were carried out by :

- melting points : electro thermal 9300, melting point engineering LTD, U.K.
- FT-IR spectra : fourrier transform infrared shimadzu (8300) (FT-IR), KBr-disc was performed by CO.S.Q. Iraq.
- H-NMR spectra: in DMSO as solvent.
- Elemental analysis (C.H.N) : EA-017 Mth.
- Uv–Visible spectra: shimadzu–1700, double beam with computerized, Japan.

Synthesis Bis (4-amide benzoyl chloride)-methane [1]:

The preparation starts with the reaction between (0.05 mole, 8g) of diethyl malonate and (0.1 mole, 15.5 g) of 4-amino benzoyl chloride with reflux for (3 hrs),after cooling, the precipitate was filtered off and recrystallized from ethanol to produce 87% of compound [1].

Synthesis Bis (4-amide-sodium benzoyl selenide)-methane [2]:

(0.04 mole,15g) of compound [1] was reacted with (0.08 mole, 8.23g) of sodium hydrogen selenide and magnetically stirred for (1 hrs), the precipitate was filtered off to produce % 85 of compound [2].

Synthesis of Bis (4-(4-substitute benzoyl)–amide–benzoyl selenide)-methane [3-7] :

A mixture of (0.01 mole,5.11g) of compound [2] with one of aroyl chloride (0.02 mole, 3.11g) of 4-amino benzoyl chloride, (0.02mole, 3.45g) of 4-mercap to benzoyl chloride, (0.02 mole, 2.2g) of mercapto aceto chloride, (0.02 mole, 3.6g) of 4-N,N-dimethyl amino benzoyl chloride, (0.02 mole, 3.8g) of toluene sulphonyl chloride) respectively, were refluxed for (3 hrs), after cooling, the precipitate was filtered off and recrystallized from ethanol to produce (%86-%82) of compounds [3-7], respectively :

Compound [3] : Bis (4-(4-amino benzoyl)-amide-benzoyl selenide)-methane.

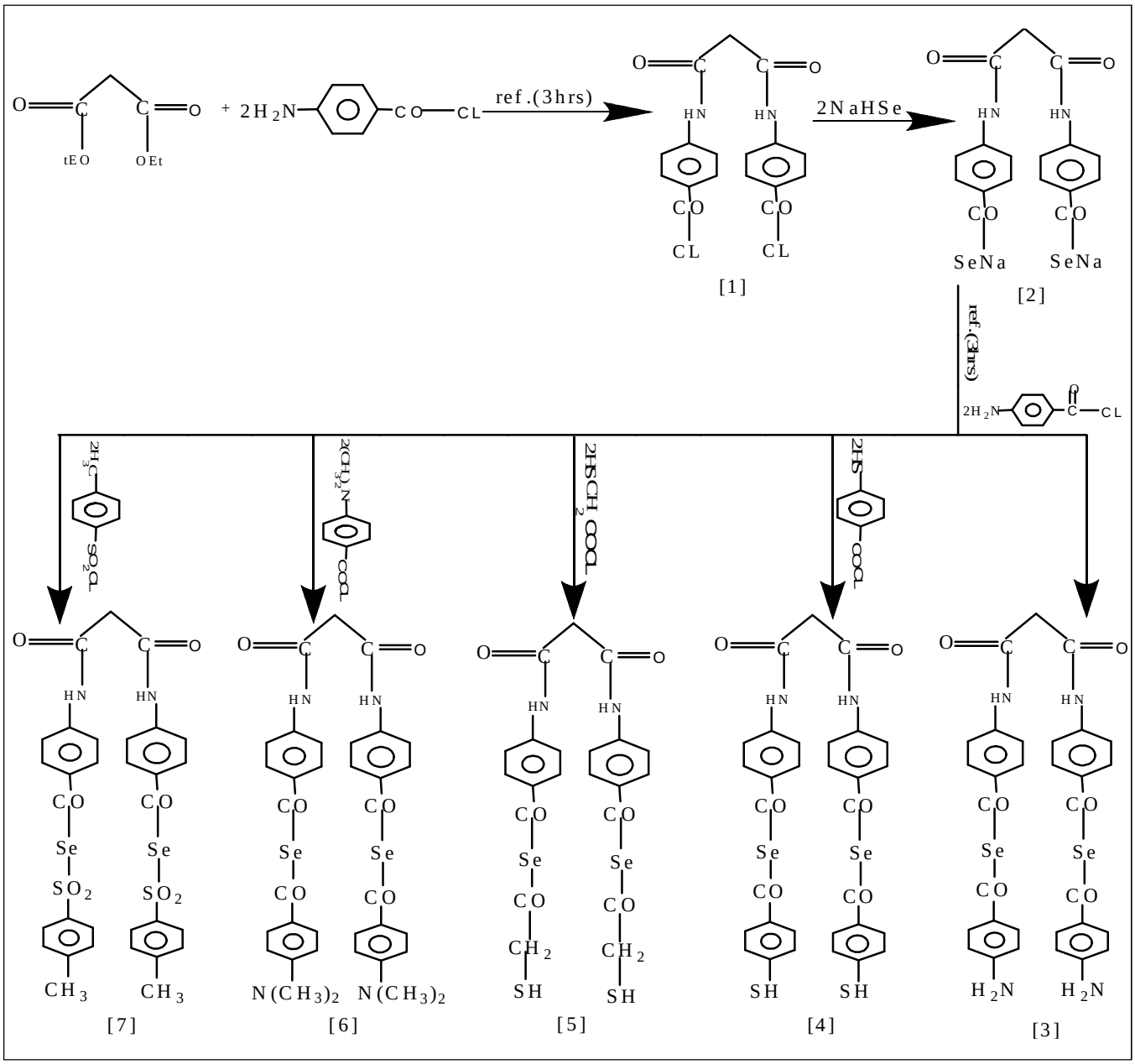
Compound [4] : Bis(4-(4-mercapto benzoyl)-amide-benzoyl selenide)-methane.

Compound [5] : Bis(4-(acetothiol) -amide-benzoyl selenide)-methane.

Compound [6] : Bis(4-(4-N,N-dimethyl amino benzoyl)-amide-benzoyl selenide)-methane.

Compound [7] : Bis (4-(4-toluine sulphonyl)-amide-benzoylselenide)-methane.

Reaction scheme:



EXPERIMENTAL FINDINGS AND ANALYSIS

Synthesized compounds [1-7] have been characterized by their melting points and spectronic techniques(FT.IR, H.NMR-spectrum, (C.H.N)-analysis) :

FT.IR–Spectra :

FT.IR–spectra showed :appearance band at (740)cm^{-1} due to (C-Cl) in compound [1],while this band is disappear in compounds [2-7] and other bands are appear :

- at (1685-1700)cm^{-1} due to(CO) of amide[3, 15], (3200-3455) cm^{-1} due to (-NH) of amide [11,15], (1655-1685) cm^{-1} due to [3, 15] (CO-Se) carbonyle of selenide, these bands in compounds[3-7].
- at (3425)cm^{-1} due to ($-NH_2$) [11-13] in compound [3].
- at (2611)cm^{-1} due to (-SH)[12] in compound [4].
- at (2610)cm^{-1} due to (-SH)[12], (2920)cm^{-1}due to (C-H) aliphatic of ($-CH_2$), (1411) cm^{-1} due to (CH_2-S)[3, 15] in compound [5].
- at (1379) cm^{-1} due to ($-N(CH_3)_2$) in compound [6].
- at (1253,1342) cm^1 due to ($-SO_2$) sulphone group[15], (2920) cm^{-1} due to (C-H) aliphatic of ($-CH_3$) group in compound [7].

Appearance of these bands are strong evidence to synthesised compounds [1-7], other data of functional groups shown in the following Table 1, Fig. 1-5.

H.NMR-Spectrum :

H.NMR-Spectrum of compounds [1-7] showed : singlet signal at ?9.9 for proton)[15] of amide (CO-NH), singlet signal at ∫ (3.08-3.61) for two protons of methane group (CO-CH_2-CO) malonate after reaction, doublete of doublet signal at ∫ (7.26-7.82) for protons of venyl group (benzoyl amide), all these bands in compounds[1-7]., while other beaks appear:

- Signal at ∫ 8.5 for protons of ($-NH_2$)[11] in compound [3].
- Signal at ∫ 10.98 for protons of (-SH) aromatic[12] in compound[4].
- Signal at ∫ 4.27 for protons (-SH) aliphatic [11,12] in compound [5].
- Signal at ∫ 3.93 for six protons of ($-N(CH_3)_2$) in

Table 1 : FT.IR data (cm^{-1}) of compounds [1-7]

Comp. No.	υ(C=O)of amide	(NH)of amide	(—C(=O)—Se) carbonyl of selenide	Other bands
[1]	1695 S	3320 m	-----	(C-Cl): 740 S
[2]	1690 S	3300 m	1660 S	
[3]	1700 S	3200 m	1685 S	($-NH_2$) :3425 m
[4]	1700 S	3338 m	1665 S	(-SH): 2611 w
[5]	1690 S	3455 m	1665 S	(-SH): 2610 w, (C-H)aliphatic:2920w (CH_2-S): 1411 S
[6]	1685 S	3304 m	1661 S	(4-$N(CH_3)_2$): 1379 S
[7]	1690 S	3450 m	1655 S	($-SO_2$)sulphone:1253S, (C-SO_2) : 1342 S (C-H)aliphatic:2920 m

Table 2 : Melting points, M.F. and elemental analysis of compounds [1-7]

Comp. No.	M.F	M.P. (C°)	Calc. / Found (C%)	H (%)	N (%)
[1]	$C_{17}H_{12}N_2O_4Cl_2$	139	54.40	3.20	7.466
			54.31	3.14	7.348
[2]	$C_{17}H_{12}N_2O_4Se_2Na_2$	147	39.853	2.344	5.470
			39.788	2.286	5.410
[3]	$C_{31}H_{24}N_4O_6Se_2$	181	52.697	3.399	7.932
			52.600	3.265	7.878
[4]	$C_{31}H_{22}N_2O_6Se_2S_2$	194	50.275	2.973	3.784
			50.189	2.906	3.679
[5]	$C_{21}H_{18}N_2O_6Se_2S_2$	162	40.914	2.922	4.546
			40.875	2.865	4.469
[6]	$C_{35}H_{32}N_4O_6Se_2$	187	55.123	4.199	7.349
			55.095	4.107	7.268
[7]	$C_{31}H_{26}N_2O_8Se_2S_2$	197	47.943	3.350	3.608
			47.855	3.307	3.517

compound[6].

- Signal at ∫ 2.97 for three protons of (-CH_3) in compound[7].

and other peaks in Fig. (6-9).

(C.H.N)-Analysis :

It was found from (C.H.N)-analysis, from compared the

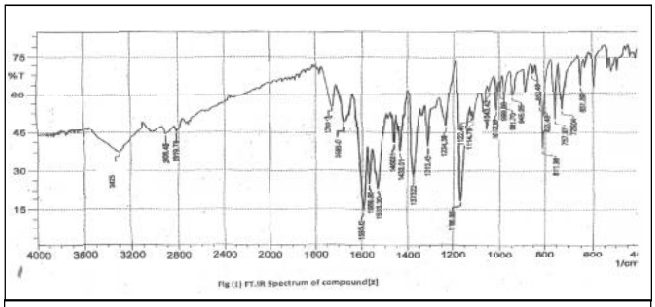


Fig. 1 : FT.IR spectrum of compound (3)

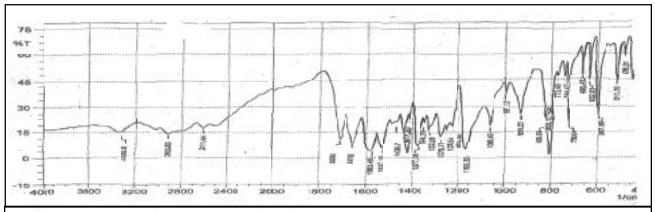


Fig. 2 : FT.IR spectrum of compound (4)

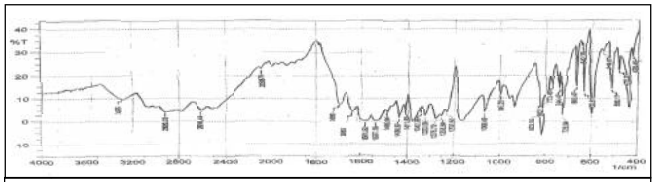


Fig. 3 : FT.IR spectrum of compound (5)

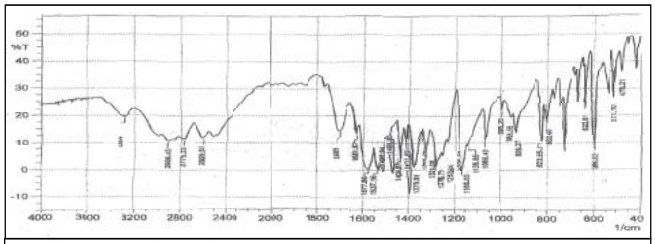


Fig. 4 : FT.IR spectrum of compound (6)

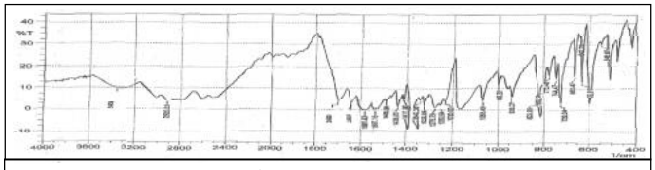


Fig. 5 : FT.IR spectrum of compound (7)

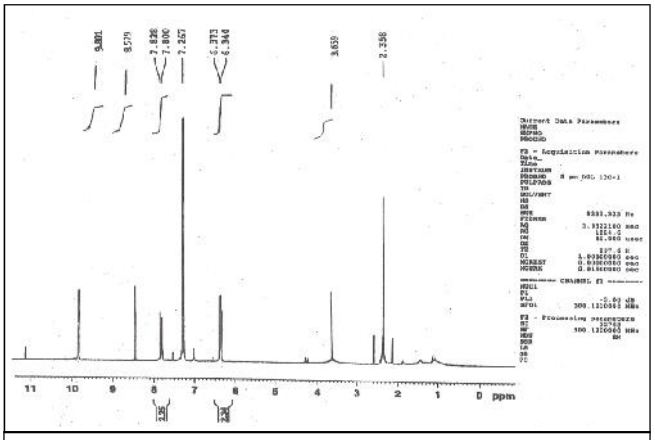


Fig. 6 : HNMR-spectrum of compound (3)

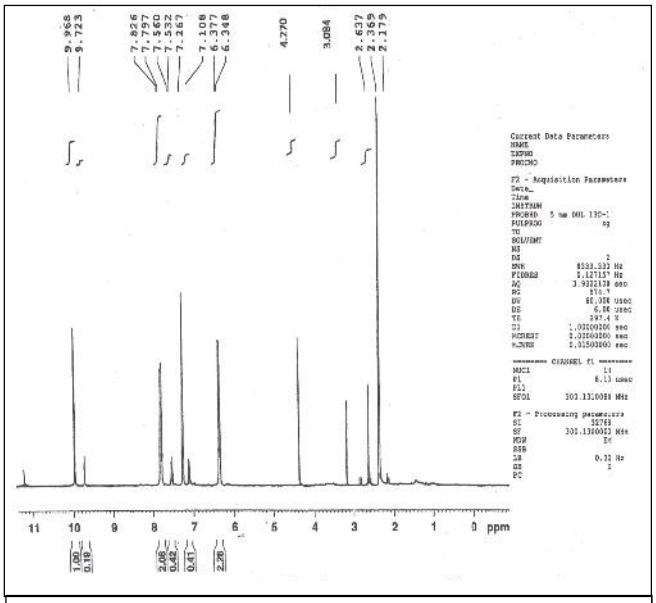


Fig. 7 : HNMR-spectrum of compound (5)

Table 3 : Antibacterial activity of the compounds[1-7] {diameter of zone (mm)}

Compounds[1-7] *	Diameter of zone(mm)	
	G+: Staphylococcus. aureus	*G-: Pseudomonas. aeruginosa*
Compounds[1]	14	9
Compounds[2]	19	12
Compounds[3]	23	17
Compounds[4]	32	26
Compounds[5]	31	24
Compounds[6]	29	20
Compounds[7]	34	28
Amoxyline**	36	30

*Minimum Inhibitory concentration (MIC)of compounds[1] (5mg/ml).
**Amoxyline (0.1mg/ml)

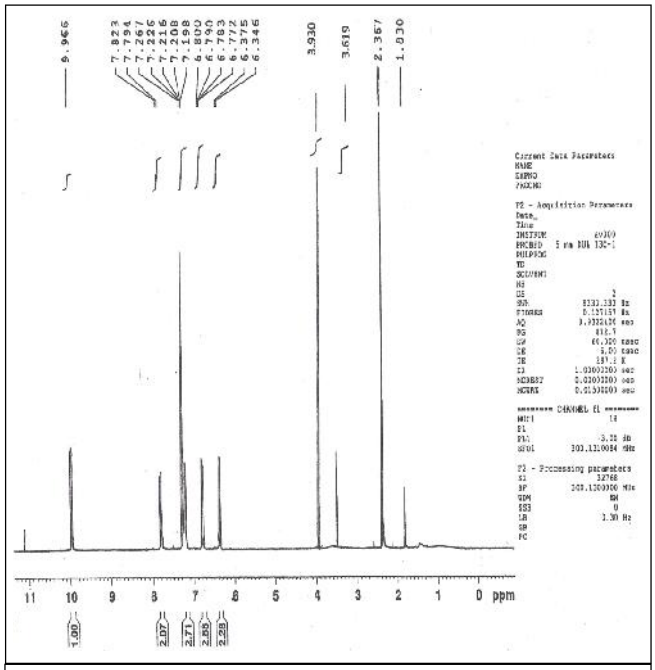


Fig. 8 : HNMR-spectrum of compound (6)

calculated data for compounds[1-7] are in a good agreement with experimentally, the results were compactable, the data of analysis and meltng points are listed in Table 2.

Assay of antimicrobial activity [16]:

Antimicrobial activity was tested by the filter paper disc diffusion method against gram positive bacteria (*Staphylococcus aureus*) and gram negative bacteria (*Pseudomonas aeruginosa*), 0.1 ml of the bacterial suspensions was seeded on agar.

To determine minimum inhibitory concentration(MIC) for each compounds [1-7] were ranged between (5-10)mg/ml by dissolved in (DMSO) and preparation 0.1mg/ml standard antibiotic amoxyline as positive standardand reference.

The positive results or sensitivity were established by the presence of clear zone of inhibition around active compounds which were measured with a meter rule and diameters were recorded based on (mm), the assays were performed with two replicates.

Generally, The results showed that the compounds[1-7] have great inhibitory effect against tested bacteria as compared with Synthetic antibiotic Amoxyline.

Table 3 showed the zone of inhibition of the compounds[1-7] in this study ranged (from 34 to 9) mm. From results, we noted that the compounds [4,5,7] have higher antibacterial activity against *S.aureus and P. aeruginosa* is due to the presence of selenium and sulphone with sulfur atomes(Se, SO_2, S) in their structures.

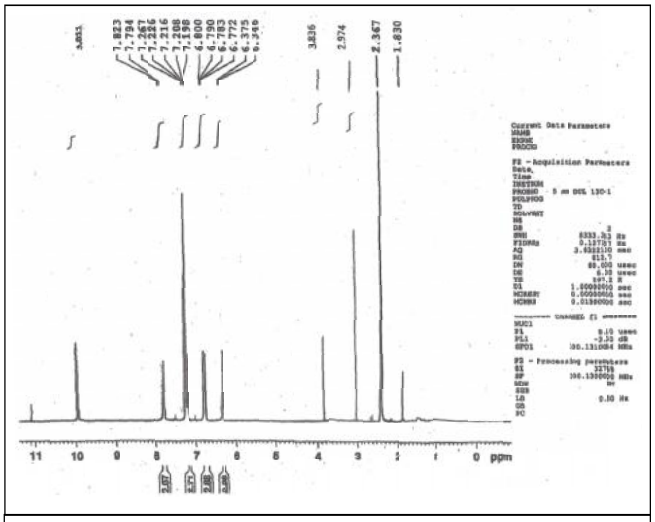


Fig. 9 : HNMR-spectrum of compound (7)

Consequently,these compounds become more effective in precipitating proteins on bacteria cell walls.These atoms form hydrogen bonds with cell wall protein and hence, destroying the cell membranes, these compounds had abroad antibacterial activity.

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