

## Synthesis and Characterization of a New Organoselenium and Organotellurium Compounds Depending on 9-Chloro-10-Nitro-9,10-Dihydroanthracene

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### Abstract

The present work refers to the preparation of novel organoselenocyanate and organotellurocyanate that occurs in two reaction steps: the first one is the synthesis of potassium selenocyanate and potassium tellurocyanate obtained by the reaction of selenium and tellurium metal with potassium cyanide in dimethyl sulphoxide as a solvent under nitrogen gas of interest. The second one is the synthesis of selenocyanate and tellurocyanate derivatives which is from the reaction between the synthesized potassium selenocyanate and potassium tellurocyanate and 9-chloro-10-nitro-9,10-dihydroanthracene. all the compounds were characterized by 1-H NMR, infrared (IR), and mass spectroscopy (MS).

**Keywords:** *organoselenium, organotellurium, selenium metal, tellurium metal, anthracene and sodium cyanide*

### Introduction

Organic synthesis, materials science, food chemistry, medical chemistry, and biology all benefit from organoselenium and organotellurium compounds<sup>1</sup>. Since Sharpless' seminal research on the ring opening reaction of epoxides with selenium nucleophiles in 1973<sup>2</sup>, the use of chalcogens in nucleophilic ring opening reactions (NRORs) has piqued chemists' interest<sup>3</sup>. A variety of procedures for transforming strained heterocycles into functionalized valuable chalcogen-containing molecules, such as b-hydroxy-, b-aminoselenides, and tellurides, have been developed based on this foundation<sup>4</sup>. A number of chalcogen-mediated functional-group transformations have also been identified and used in organic synthesis and organocatalysis. For the production of b-hydroxy and b-amino chalcogenides, NRORs of epoxides and aziridines are the preferred process. These important compounds are versatile reagents with a wide range of applications in organic synthesis due to their ability to selectively functionalize the carbon-chalcogen bond. Complete synthesis of lycorine,<sup>5</sup> (-)-galanthamine,<sup>6</sup> (+)-7-deoxypancratistatin<sup>7</sup>, and plumisclerin<sup>8</sup>, for example, all have a selenium-mediated ring opening reaction as a main phase. Furthermore, functionalized organoselenides and organotellurides are effective drug candidates with potential uses in medicinal chemistry due to their biological properties<sup>9</sup>. The current range of ring-opening reactions for the synthesis of functionalised

organoselenium and organotellurium compounds is reviewed in this Perspective.

## 2. EXPERIMENTAL

### 2.1 Chemicals and Apparatus

Chemicals acquired from Sigma-Aldrich, Fluka and BDH utilized without filtration. Liquefying point was dictated by utilizing open hairlike tube dissolving point mechanical assembly. <sup>1</sup>H NMR spectra was recorded on Bruker DRX System AL (500 MHz) with TMS as an inner reference utilizing DMSO-d<sub>6</sub> dissolvable. Infra-red spectra were recorded with KBr circles utilizing a FT-IR spectrophotometer Shimadzu model 8400 S in reach 4000- 250 cm<sup>-1</sup>. Dissolving purposes of every single strong compound were resolved utilizing a MPS10 electrically warmed liquefying point mechanical assembly.

### 2.2 [9- nitro-10-selenocyanato-9-10-dihydroanthracene]:

Put in a round with two neck (0.005 mol, 0.44 g) of finely ground and dried selenium with (0.006 mol, 0.33 g) of finely ground and dried potassium cyanide, then add 15 mL of freshly distilled dimethyl sulfoxide and heat the mixture for one hour at a temperature of 100 ° C. In an inert atmosphere of nitrogen, then 15 ml of distilled dimethyl sulfoxide was added to the hot converter and then left to cool at room temperature where a pale yellow solution was added. Then (0.0056 mol, 1.33 g) of the compound (9-chloro-10-nitro-9, 10-dihydroanthracene) was added and repelled back under nitrogen for two hours at a temperature of 100 ° C. The solution was filtered while it was hot and left until it cooled to laboratory temperature. Then the filtrate was poured into a baker containing 250 ml of cold distilled water, and reddish-brown crystals were observed<sup>10</sup>. The solution was filtered to obtain crystals, then washed with distilled water and ethanol and recrystallized with ethanol. (The resulting weight was 0.8 g, the reaction yield was 47%, its melting point: 110-112 d)

### 2.3 [9- nitro-10-tellurocyanato-9-10-dihydroanthracene]:

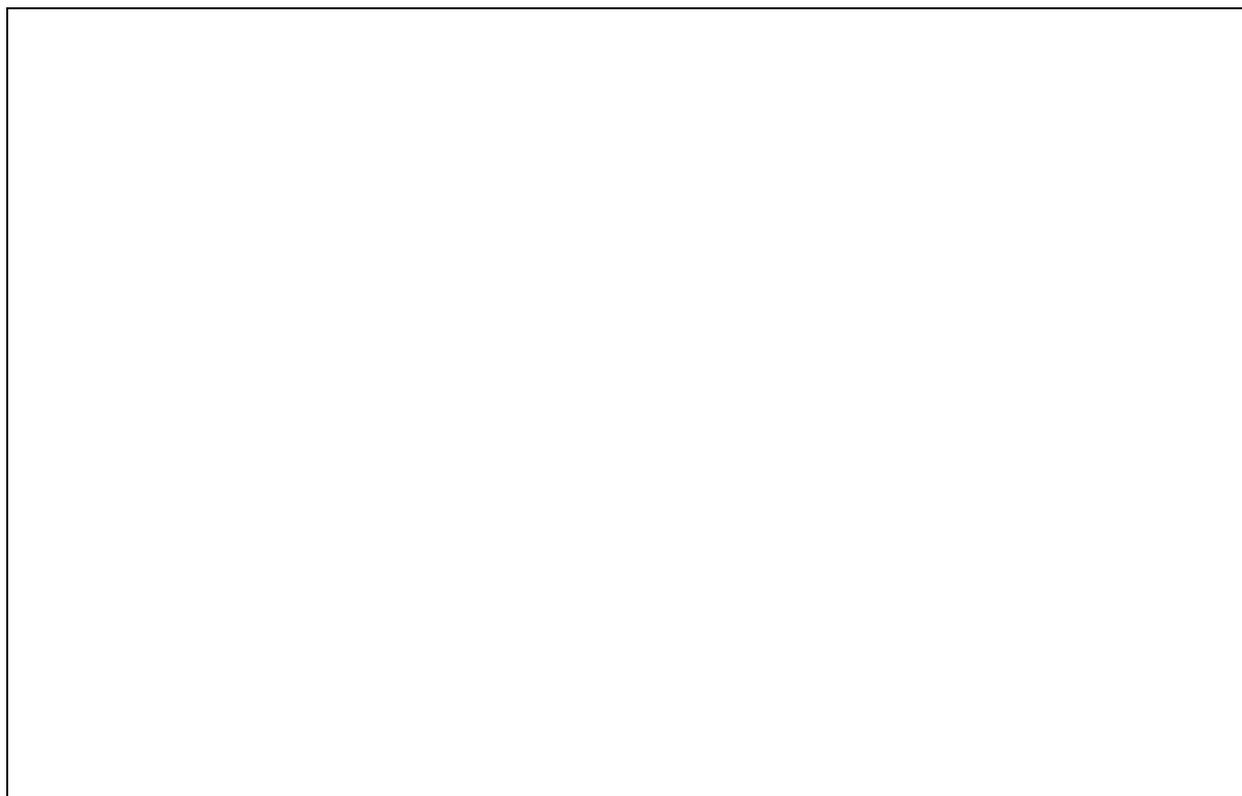
Put in a round with two neck (0.005 mol, 0.63 g) of finely ground and dried tellurium with (0.006 mol, 0.33 g) of well ground and dried potassium cyanide, then add 15 mL of freshly distilled dimethyl sulfoxide and heat the mixture for one hour at 100 ° C in An inert atmosphere of nitrogen, then 15 ml of distilled dimethyl sulfoxide was added to the hot transformer and then left to cool at room temperature where a pale yellow solution was added. Then (0.0056 mol, 1.33 g) of the compound (9-chloro-10-nitro-9,10-dihydroanthracene) was added to it and rose back under nitrogen for two hours at a temperature of 100 ° C. The solution was filtered while it was hot and left until it cooled to the laboratory temperature. Then the filtrate was poured into a baker containing 250 ml of cold distilled water and the appearance of greenish-yellow crystals was observed<sup>11</sup>. The solution was filtered to obtain crystals, then washed with distilled water and ethanol and recrystallized with ethanol. (Yield weight 0.67 g, reaction yield 35%, melting point: 107-105 d)

**Table (1) Physical data for organoselenium and organotellurium compounds**

No	Molecular formula	M.Wt	Color	m.p. C°	Yield %
1	C <sub>15</sub> H <sub>10</sub> N <sub>2</sub> O <sub>2</sub> Se	329.21	Reddish brown	110-112D	47%
2	C <sub>15</sub> H <sub>10</sub> N <sub>2</sub> O <sub>2</sub> Te	377.85	Greenish yellow	105-107D	35%

### 3. RESULTS AND DISCUSSION

The compound 9-chloro-10-nitro-9,10-dihydroanthracene is formed by the reaction between anthracene and nitric acid, acetic acid then with hydrochloric acid<sup>12</sup>, these step reaction gave a great yield of starting material. [9- nitro-10-selenocyanato-9-10-dihydroanthracene] and [9- nitro-10-tellurocyanato-9-10-dihydroanthracene] have been obtained by reacting between 9-chloro-10-nitro-9,10-dihydroanthracene and potassium selenocyanate and potassium tellurocyanate respectively in DMSO under N<sub>2</sub> gas, as shown in Scheme (1)



#### **Scheme (1) preparation of compounds [9- nitro-10-selenocyanato-9-10-dihydroanthracene] and [9- nitro-10-tellurocyanato-9-10-dihydroanthracene]**

The spectral measurements of <sup>1</sup>HNMR for [9- nitro-10-selenocyanato-9-10-dihydroanthracene] as shown in Figure (1) and Table (2), showed Different bundles appear in the aromatic region extending between ( 7.68 – 9.01 ppm). Where there appeared multiple beams extending between (7.68 – 7.9 ppm) the integration of 8 protons belonging to the symmetric rings at the ends of the intermediate ring. He

also showed beams extending between ( 8.21 – 8.3 ppm) and the integration of 2 protons belonging to the protons of the intermediate ring<sup>(13-14)</sup>.

The spectral measurements of <sup>1</sup>H NMR for [9- nitro-10-tellurocyanato-9-10-dihydroanthracene] as shown in Figure (2) and Table (2), showed Different bundles appear in the aromatic region extending between ( 7.68 – 9.01 ppm ). Where there appeared multiple beams extending between ( 7.68 – 7.95 ppm ) and the integration of 8 protons belonging to the symmetric rings at the ends of the intermediate ring. He also showed beams extending between ( 8.21 – 9.01 ppm ) and the integration of 2 protons belonging to the protons of the intermediate ring<sup>(13-14)</sup>.

Table (2) <sup>1</sup>H NMR Spectral Data for Selected Compounds

	Structure for comp.	<sup>1</sup> H NMR (DMSO-d <sub>6</sub> ); TMS = 0 ppm
1		7.68 - 7.95 (m, 8H, Ar-H); 8.21 – 9.01 (m, 2H,Alph-H)
2		7.68 – 7.95 (m, 8H, Ar-H); 8.21 – 9.01 (m, 2H,Alph - H);

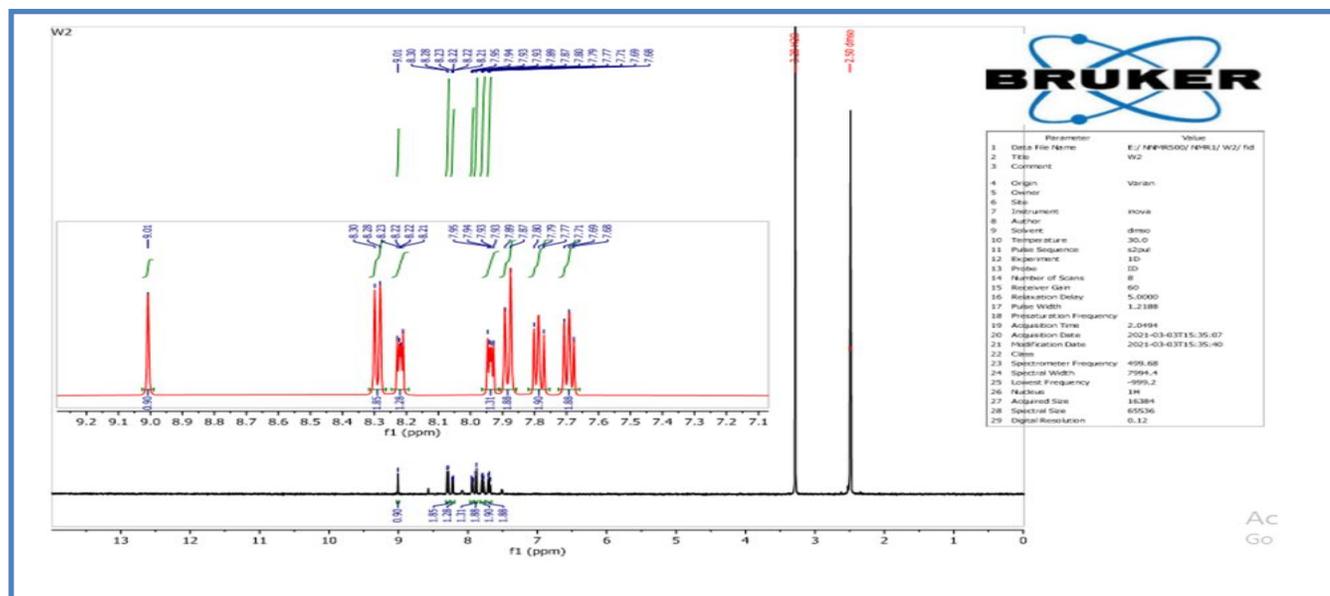
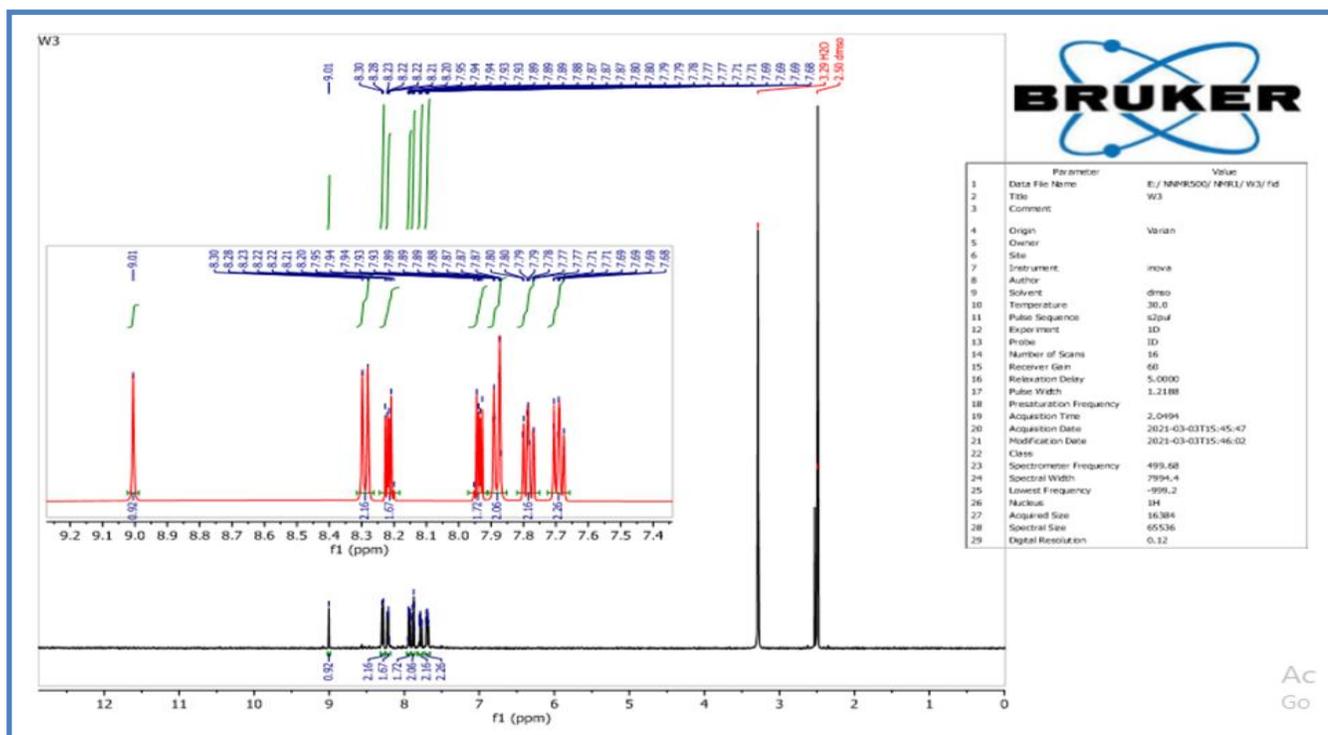


Figure (1): <sup>1</sup>H NMR spectrum for [9- nitro-10-selenocyanato-9-10-dihydroanthracene]



IR spectra for all compounds displayed common features in certain regions and characteristic bands in the fingerprint and other regions. IR spectra for all compounds displayed common features in certain regions and characteristic bands in the fingerprint and other regions. The IR spectra confirm the suggested structure for organoselenium compound<sup>(13-14)</sup>. As shown in Figure (3), Table (3). In the IR spectrum, we observe a clear band at (1944 cm<sup>-1</sup>) respectively refer to C≡N<sup>(13-14)</sup>, the appearance of a band at (1674 cm<sup>-1</sup>) respectively indicated to C=C bond for, while the NO<sub>2</sub> group shows a very clear band at (1513 cm<sup>-1</sup>), and the C-N group shows a very clear band at (1162 cm<sup>-1</sup>), band appeared at (515 cm<sup>-1</sup>)<sup>(13-14)</sup>. respectively respectively for C - Se.

The IR spectra confirm the suggested structure for organotellurium compounds.<sup>(13-14)</sup>. As shown in Figure (4), Table (3). In the IR spectrum, we observe a clear band at (2208 cm<sup>-1</sup>) respectively refer to C≡N<sup>(13-14)</sup>, the appearance of a band at (1584 cm<sup>-1</sup>) respectively indicated to C=C bond for, while the NO<sub>2</sub> group shows a very clear band at (1565 cm<sup>-1</sup>), and the C-N group shows a very clear band at (1166 cm<sup>-1</sup>) band appeared at (520 cm<sup>-1</sup>)<sup>(13-14)</sup>. respectively respectively for C - Te.

**Table (3) FT - IR Spectral Data for Selected Compounds**

C≡N	Aromatic C=C	NO <sub>2</sub>	C-N	C-Se	C-Te
1944	1674	1513	1162	515	—
2208	1584	1565	1166	—	520

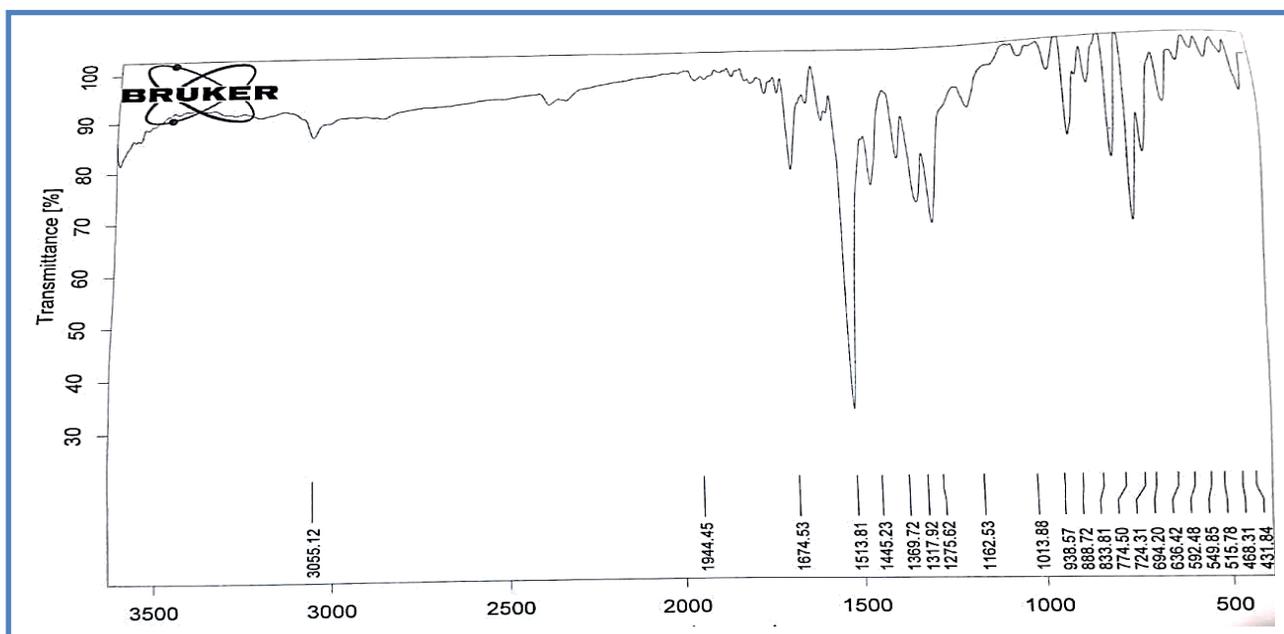
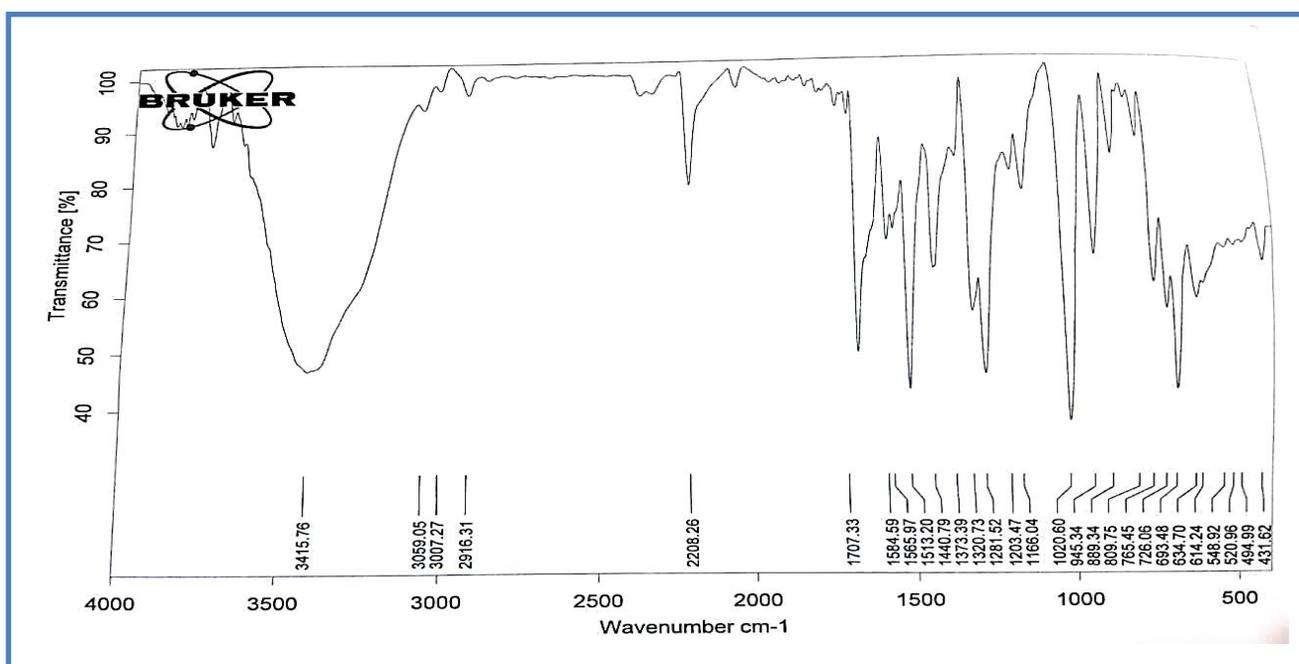


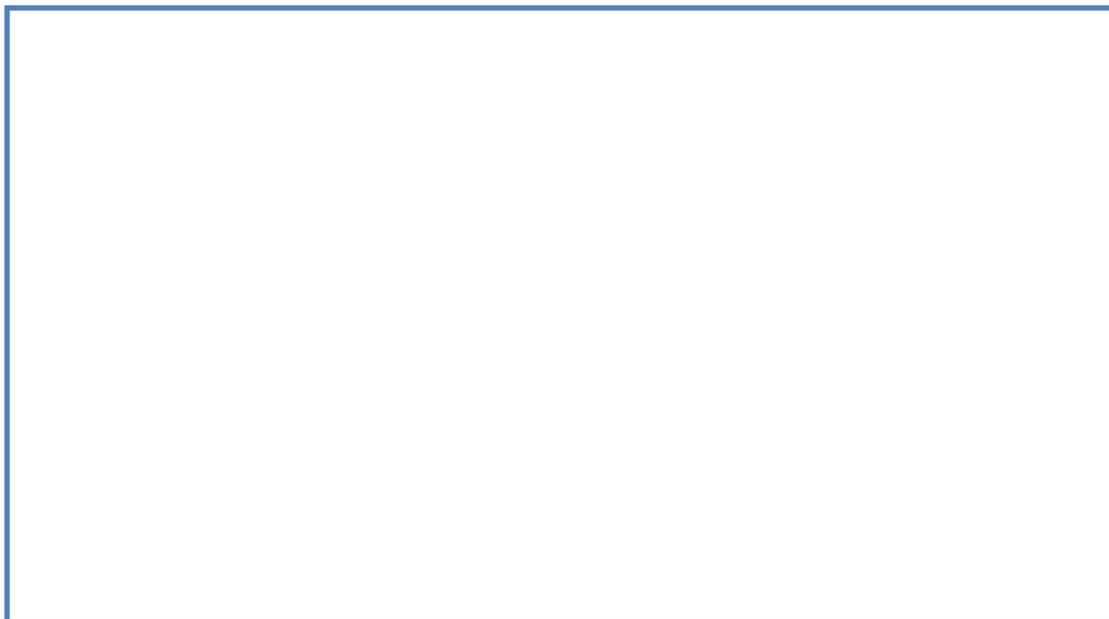
Figure (3): IR spectrum for [9-nitro-10-selenocyanato-9-10-dihydroanthracene]



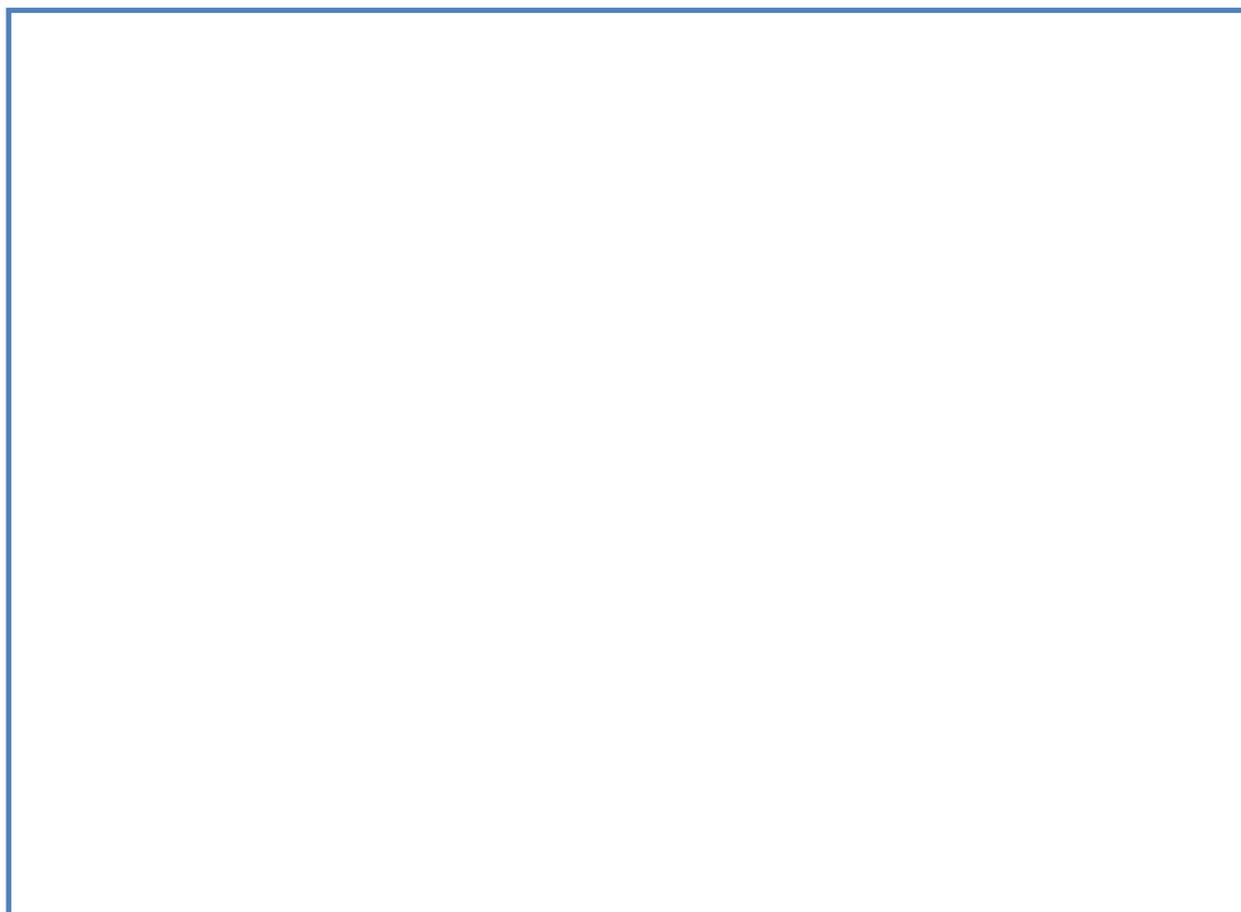
The mass spectrum of compound **9-nitro-10-selenocyanato-9-10-dihydroanthracene** showed us the parent ion beam at 327 m/z, which represents the molecular weight of the compound, and the appearance of a relatively high abundance beam at 176 m/z belonging to group  $C_{10}H_{11}NO_2^{+}$  in addition to other packages.

The mass spectrum of compound **9-nitro-10-tellurocyanato-9-10-dihydroanthracene** showed us the





**Scheme (2) mechanism for fragmentation for [9- nitro-10-selenocyanato-9-10-dihydroanthracene**



**Scheme (3) mechanism for fragmentation for [9- nitro-10-tellurocyanato-9-10-dihydroanthracene]**

**References :**

1. Lenardão, E. J., Santi, C. & Sancineto, L. *New frontiers in organoselenium compounds*. (Springer, 2018).
2. Sharpless, K. B. & Lauer, R. F. Mild procedure for the conversion of epoxides to allylic alcohols. First organoselenium reagent. *J. Am. Chem. Soc.* **95**, 2697–2699 (1973).
3. Wirth, T. *Organoselenium chemistry: synthesis and reactions*. (John Wiley & Sons, 2012).
4. Tanini, D. *et al.* Direct and straightforward access to substituted alkyl selenols as novel carbonic anhydrase inhibitors. *Eur. J. Med. Chem.* **185**, 111811 (2020).
5. Shin, H.-S., Jung, Y.-G., Cho, H.-K., Park, Y.-G. & Cho, C.-G. Total synthesis of (±)-lycorine from the endo-cycloadduct of 3, 5-dibromo-2-pyrone and (E)-β-borylstyrene. *Org. Lett.* **16**, 5718–5720 (2014).
6. Trost, B. M., Tang, W. & Toste, F. D. Divergent enantioselective synthesis of (–)-galanthamine and (–)-morphine. *J. Am. Chem. Soc.* **127**, 14785–14803 (2005).
7. Pandey, G., Fernandes, R., Dey, D. & Majumder, B. Total synthesis of (+)-trans-dihydronarciclasine from (+)-7-azabicyclo [2.2. 1] heptanone. *Tetrahedron* **74**, 5752–5757 (2018).
8. Gao, Y., Wei, Y. & Ma, D. Synthetic studies toward plumisclerin A. *Org. Lett.* **21**, 1384–1387 (2019).
9. Hodage, A. S., Phadnis, P. P., Wadawale, A., Priyadarsini, K. I. & Jain, V. K. Synthesis, characterization and structures of 2-(3, 5-dimethylpyrazol-1-yl) ethylseleno derivatives and their probable glutathione peroxidase (GPx) like activity. *Org. Biomol. Chem.* **9**, 2992–2998 (2011).
10. Chary, N. S. & Fernandez-Alba, A. R. Determination of volatile organic compounds in drinking and environmental waters. *TrAC Trends Anal. Chem.* **32**, 60–75 (2012).
11. 4-(Chloromethyl)-2-Hydroxybenzaldehyde, S. and I. of N. O. C. D. from. No Title. *Dep. Chem. Coll. Sci. Univ. Thi-Qar, Iraq*. Mohammed I. Abowd1\* and Nuha H. Al-Saadawy2 (2019).
12. Kadhim, M. A. & Al-Saadawy, N. H. Synthesis and Characterization for Some New Organoselenium Compounds Depending on 8-Hydroxyquinoline. *Ann. Rom. Soc. Cell Biol.* 2162–2172 (2021).
13. Roth, A. E. *Game-theoretic models of bargaining*. (Cambridge University Press, 1985).
14. Banert, K. & Toth, C. Synthesis and reactions of vinyl isoselenocyanates. *Angew. Chemie Int. Ed. English* **34**, 1627–1629 (1995).