

Synthesis and Characterization of Symmetrical and Unsymmetrical Analogues of 1,2-bis[bis(4-chlorophenyl)Methyl] Diselane

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Abstract The 'living' species selenide anion Se_2^{2-} , generated *in situ* by the reduction of 1,2-bis(bis(4-chlorophenyl)methyl)diselane with sodium borohydride, was treated with various organic halides to afford synthetically important organoselenium compounds including α,ω -diselenoethers and selenides bearing ester functionality, under mild and neutral conditions. These compounds have been characterized analytically with the help of various spectroscopic techniques *viz.*, IR, multinuclear NMR (^1H , ^{13}C , ^{77}Se) and mass spectrometry.

Keywords Selenide, Diselenide, Symmetrical Diselenide, Unsymmetrical Selenide, Selenoether

1. Introduction

In recent years, the synthesis of organoselenium compounds have become an increasingly popular area in the field of synthetic reagents and intermediates in organic synthesis [1,2]. Specifically, synthetic routes for the introduction of seleno-substituents into organic molecule which are the building blocks for the synthesis of modified selenocarbohydrates, selenoaminoacids and selenoproteins have attracted much attention due to their interesting structural and biological properties [3]. Literature is inundated with several synthetic methods for the synthesis of selenide anion, the more important of which include the use of reduction of diorganyl diselenide with sodium [4], samarium diiodide [5], Sm/TMSCl [6] and catalyzed system $\text{Cu}_2\text{O}/\text{Mg}/\text{bpy}$ [7]. Moreover, Grignard reagents [8,9] also react with selenium to give equivalent species of selenide anions.

A scrutiny of literature, pertinent to the chemistry of diaryl methyl selenium/tellurium compounds and their applications deserve a special mention here. Recently, it has been reported that diphenyl methyl selenocyanate (Ph_2CHSeCN) plays a protective role against carbon tetrachloride induced hepatotoxicity in mice [10]. Margolis and Pittman[11]

prepared bis(diphenylmethyl) diselenide by using two methodologies one involve the reduction of benzophenone using hydrogen selenide and another by treating diphenyl methyl bromide with sodium hydrogen selenide. Okama et al. [12] reported the synthesis of diphenyl methyl diselenide in satisfactory yields by the reduction of selenocarbonyl intermediate with NaBH_4 . Kamigata et al. [13] have shown the formation of methyl diphenylmethyl selenide and benzyl diphenylmethyl selenide in low yields by the reaction of 1,3-bis(alkylseleno)allenes and diphenyl diazomethane in refluxing benzene.

In the light of our experimental interests [14,15] on the aromatic selenium and tellurium compounds, we report in this communication the synthesis and characterization of 1,2-bis(bis(4-chlorophenyl)methyl)diselane and further a convenient method for the reductive cleavage of Se-Se bond of 1,2-bis(bis(4-chlorophenyl) methyl) diselane with sodium borohydride to generate selenide anion. Sodium borohydride, as a reducing agent capable of reducing many functionalities like nitrile, ester, carbonyl, epoxide and double bonds. Ethanolic sodium borohydride has been widely used to generate sodium selenide ions in a protic media. The complete reduction of diorganyl diselenides leads to the formation of organyl selenolates [16,17], which are colorless in solution. It has been found that selenide anion, a 'living' species, reacted smoothly with active organic halides to give the designed symmetrical/unsymmetrical selenium derivatives in good yields.

2. Materials and Methods

2.1. General Methods

All the reactions were carried out under dry and deoxygenated nitrogen atmosphere. Selenium/Tellurium was estimated by standard methods. Sodium borohydride (Loba), selenium (Hi-media) and tellurium (Aldrich) were stored in dessicator prior to use. 4,4'-Dichloro diphenylmethyl chloride was prepared by literature method. All other

chemicals were of analytical grade and used without further purification. Ethanol was freshly distilled and dehydrated under sodium metal. Separation and purification of the compounds was done by column chromatography performed on activated silica gel (100-120) using hexane as eluant.

^1H , $^{13}\text{C}\{^1\text{H}\}$ NMR was recorded in CDCl_3 using Me_4Si as an internal standard using Jeol AL 300 MHz spectrometer. $^{77}\text{Se}\{^1\text{H}\}$ NMR was recorded on the same instrument using Me_2Se as an external reference. Infrared spectra were obtained either between KBr pellets as neat liquids or as compressed KBr discs in case of solid compounds on Perkin-Elmer model-1430 spectrophotometer. Mass spectra were obtained on a micro Q-TOF mass spectrometer.

2.2. Synthesis of 1,2-bis[bis(4-chlorophenyl) methyl] Diselane

To a vigorously stirred mixture of powdered sodium hydroxide (3.0 g, 75 mmol), elemental selenium (4.0 g, 50 mmol) and dimethylformamide (30 ml), 100% hydrazine hydrate was added slowly. After stirring for nearly 6 h at room temperature, a solution of 4,4'-dichloro diphenylmethyl chloride (100 mmol) dissolved in 15 ml DMF was added dropwise. When the color of the solution became light yellow, it was diluted with about 250 ml of distilled water and extracted in dichloromethane (3×50 ml). The organic layer was washed with 6N HCl followed by distilled water and then dried over anhydrous sodium sulfate. Solvent was evaporated on a rotary-evaporator and the product was subjected to purification on a silica column using hexane as the eluant.

2.2.1. *1,2-bis(bis(4-chlorophenyl)methyl)diselane* [$(\text{ClC}_6\text{H}_4)_2\text{CHSe}]_2$ (**1**). Yellow crystalline solid (89% yield). m.p. 92-95 °C. IR ($\nu_{\text{max}}/\text{cm}^{-1}$, KBr): 3025, 2925, 2584, 2357, 1899, 1779, 1660, 1591, 1488, 1402, 1215, 1176, 1092, 1014, 925, 787, 681, 521, 499, 414. MS-EI, *m/e* (RI, assignment): 409 (15, $[\text{C}_{14}\text{H}_{10}\text{Se}_2\text{Cl}_2]^+$), 397 (6, $[\text{C}_{13}\text{H}_8\text{Se}_2\text{Cl}_2]^+2$), 234 (11, $[\text{C}_{13}\text{H}_9\text{Cl}_2]^+-1$), 204 (8, $[\text{C}_7\text{H}_5\text{SeCl}]^+-2$). NMR (^1H , 300 MHz, CDCl_3): δ 7.30-7.23 (m, 8H), 5.71 (s, 1H). NMR ($^{13}\text{C}\{^1\text{H}\}$, 75 MHz, CDCl_3): δ 139.71, 139.15, 138.94, 138.90, 138.48, 135.58, 133.91, 133.69, 133.60, 131.45, 131.17, 130.29, 129.99, 128.74, 128.24, 127.90, 49.71; NMR ($^{77}\text{Se}\{^1\text{H}\}$, 57 MHz, CDCl_3): δ 528.5.

2.3. Synthesis of Symmetrical 1,2-bis[bis(4-chloro phenyl) methyl] Alkanes

To a 3.15gm (5 mmol) solution of 1,2-bis(bis(4-chlorophenyl)methyl)diselane in 50 ml $\text{C}_2\text{H}_5\text{OH}-\text{DMF}$ (3:2) was added 0.456 gm (12 mmol) of NaBH_4 in three parts with continuous stirring at room temperature in 30 minutes. After 3 h of stirring, 10 mmol of alkylating agent, X (CH_2)_n X, diluted with equal volume of DMF was added dropwise at room temperature. Completion of reaction was monitored with the help of TLC. Extraction was done with dichloromethane after evaporating ethanol under vacuum. The organic layer was washed repeatedly with distilled water (3×40 ml), dried over anhydrous

Na_2SO_4 . Solvent is evaporated on a rotary-evaporator and the product is subjected to purification on a silica column using hexane as the eluant.

2.3.1. *Bis[bis(4-chlorophenyl) methylselanyl] methane*, [$(\text{ClC}_6\text{H}_4)_2\text{CHSe}]_2\text{CH}_2$ (**2a**). Yellow solid (74% yield). m.p. 58-60 °C. IR ($\nu_{\text{max}}/\text{cm}^{-1}$, KBr): 3435, 2954, 2853, 2360, 1639, 1488, 1092, 908, 787, 732, 525. NMR (^1H , 300 MHz, CDCl_3): δ 7.25-7.14 (m, 8H), 5.38 (s, 1H), 3.09 (s, 2H). NMR ($^{13}\text{C}\{^1\text{H}\}$, 75 MHz, CDCl_3): δ 138.77, 133.32, 131.02, 130.21, 129.91, 129.10, 128.48, 127.97, 48.50, 48.14.

2.3.2. *[2-{bis(4-chlorophenyl) methylselanyl} ethaneselanyl] bis(4-chlorophenyl)methane*, [$(\text{ClC}_6\text{H}_4)_2\text{CHSe}]_2(\text{CH}_2)_2$ (**2b**). Yellow solid (89% yield). m.p. 80-82 °C. IR ($\nu_{\text{max}}/\text{cm}^{-1}$, KBr): 3420, 2925, 2853, 2347, 1632, 1458, 1397, 906, 785, 758. MS-EI, *m/e* (RI, assignment): 646(5, $[\text{C}_{27}\text{H}_{20}\text{Se}_2\text{Cl}_4]^+1$), 518 (20, $[\text{C}_{28}\text{H}_{20}\text{Se}_2]^+2$), 513 (28, $[\text{C}_{22}\text{H}_{18}\text{Se}_2\text{Cl}_2]^+-2$), 330 (66, $[\text{C}_{14}\text{H}_{11}\text{SeCl}_2]^+$), 325 (7, $[\text{C}_{10}\text{H}_{10}\text{Se}_2\text{Cl}]^+$), 316 (6, $[\text{C}_{13}\text{H}_9\text{SeCl}_2]^+$), 236 (6, $[\text{C}_{13}\text{H}_9\text{SeCl}_2]^+$), 212 (9, $[\text{C}_4\text{H}_6\text{Se}_2]^+$). NMR (^1H , 300 MHz, CDCl_3): δ 7.21-7.16 (m, 8H), 5.15 (s, 1H), 2.51 (s, 4H). NMR ($^{13}\text{C}\{^1\text{H}\}$, 75 MHz, CDCl_3): δ 139.06, 135.61, 131.43, 131.09, 129.60, 128.85, 52.35, 51.92, 50.56.

2.3.3. *[3-{bis(4-chlorophenyl) methylselanyl} propylselanyl] bis(4-chlorophenyl)methane*, [$(\text{ClC}_6\text{H}_4)_2\text{CHSe}]_2(\text{CH}_2)_3$ (**2c**). Yellow semisolid (63% yield). IR ($\nu_{\text{max}}/\text{cm}^{-1}$, KBr): 3410, 3137, 2924, 2856, 2360, 1591, 1400, 1089, 1016, 909, 736. NMR (^1H , 300 MHz, CDCl_3): δ 7.27-7.21 (m, 8H), 5.25 (s, 1H), 3.43-3.23 (t, *J* = 6.3 Hz, 2H), 2.49-2.45 (t, *J* = 6.9 Hz, 2H), 2.04-1.96 (m, 2H). NMR ($^{13}\text{C}\{^1\text{H}\}$, 75 MHz, CDCl_3): δ 139.51, 133.24, 133.17, 130.81, 129.96, 129.89, 128.82, 128.38, 127.98, 46.99, 45.42, 32.66, 32.55, 24.06. NMR ($^{77}\text{Se}\{^1\text{H}\}$, 57 MHz, CDCl_3): δ 339.12.

2.4 Synthesis of Unsymmetrical 1,2-bis(bis(4-chloro phenyl) methyl)selenides

To a 3.15gm (5 mmol) solution of 1,2-bis(bis(4-chlorophenyl)methyl)diselane in 50 ml $\text{C}_2\text{H}_5\text{OH}-\text{DMF}$ (3:2) was added 0.456 gm (12 mmol) of NaBH_4 in three parts with continuous stirring at room temperature in 30 minutes. After 3 h of stirring, 10 mmol of alkylating agent (RX) diluted with equal volume of DMF was added dropwise at room temperature. Completion of reaction was monitored with TLC. Extraction is done with dichloromethane after evaporating ethanol under vacuum. The organic layer is washed repeatedly with distilled water (3×40 ml), dried over anhydrous Na_2SO_4 . Solvent is evaporated on a rotary-evaporator and the product is subjected to purification on a silica column using hexane as the eluant.

2.4.1. *Bis(4-chlorophenyl)methyl(methyl)selane*, [$(\text{ClC}_6\text{H}_4)_2\text{CHSeCH}_3$] (**3d**). Yellow oil (54% yield). IR ($\nu_{\text{max}}/\text{cm}^{-1}$, KBr): 3395, 2924, 2856, 2369, 2347, 1489, 1092, 909, 793. NMR (^1H , 300 MHz, CDCl_3): δ 7.22-7.12 (m, 8H), 5.12 (s, 1H), 1.76(s, 3H). NMR ($^{13}\text{C}\{^1\text{H}\}$, 75 MHz, CDCl_3): δ 139.68, 133.88, 130.89, 129.71, 128.92, 127.90, 46.25, 6.39. NMR ($^{77}\text{Se}\{^1\text{H}\}$, 57 MHz, CDCl_3): δ 266.8

2.4.2. *Bis(4-chlorophenyl)methyl(ethyl) selane*, $[(C_6H_4)_2CHSeCH_2CH_3]$ (**3e**). Light yellow solid (78% yield). m.p 50–53 °C. IR (ν_{max}/cm^{-1} , KBr): 3399, 2924, 1665, 1594, 1490, 1401, 1174, 1091, 908, 850, 736, 521. MS-EI, *m/e* (RI, assignment): 347 (4, $[C_{15}H_{14}SeCl_2]^+ + 2$), 331 (6, $[C_{14}H_{12}SeCl_2]^+$), 330 (24, $[C_{14}H_{11}SeCl_2]^+$), 318 (7, $[C_{13}H_9SeCl_2]^+ + 2$), 274 (47, $[C_{15}H_4Se]^+$), 246 (9, $[C_{13}H_{11}Se]^+ + 1$), 229 (6, $[C_{14}H_{12}SeCl_2]^+$), NMR (1H , 300 MHz, $CDCl_3$): δ 7.23–6.98 (m, 8H), 5.51 (s, 1H), 2.36–2.25 (q, $J = 7.5$ Hz, 2H), 1.17–1.24 (t, $J = 7.5$ Hz, 3H). NMR ($^{13}C\{^1H\}$, 75 MHz, $CDCl_3$): δ 141.90, 139.74, 133.52, 129.96, 128.64, 127.87, 126.96, 46.35, 19.58, 15.22. ($^{77}Se\{^1H\}$, 57 MHz, $CDCl_3$): δ 396.5

2.4.3. *Bis(4-chlorophenyl)methyl(allyl)selane*, $[(C_6H_4)_2CHSeCH_2CHCH_3]$ (**3f**). Colorless viscous oil (52% yield). IR (ν_{max}/cm^{-1} , KBr): 3432, 2924, 2347, 1897, 1489, 1399, 1259, 1092, 1016, 916, 786, 523. NMR (1H , 300 MHz, $CDCl_3$): δ 7.26–7.10 (m, 8H), 5.85–5.71 (m, 1H), 5.12 (s, 1H), 4.98–4.89 (d, $J = 9.9$ Hz, 1H), 4.77–4.71 (d, $J = 16.8$ Hz, 1H), 2.93–2.85 (d, $J = 7.5$ Hz, 2H). NMR ($^{13}C\{^1H\}$, 75 MHz, $CDCl_3$): δ 139.73, 133.76, 133.16, 130.20, 128.92, 128.85, 128.79, 126.96, 46.45, 44.84. NMR ($^{77}Se\{^1H\}$, 57 MHz, $CDCl_3$): δ 459.62.

2.4.4. *Bis(4-chlorophenyl)methyl(butyl) selane*, $[(C_6H_4)_2CHSeCH_2CH_2CH_2CH_3]$ (**3g**). Yellow oil liquid (63% yield). IR (ν_{max}/cm^{-1} , KBr): 3410, 2928, 2361, 1628, 1484, 1092, 793. NMR (1H , 300 MHz, $CDCl_3$): δ 7.28–7.17 (m, 8H), 5.19 (s, 1H), 2.36–2.27 (t, $J = 7.2$ Hz, 2H), 1.57–1.42 (m, 2H), 1.28–1.17 (m, 2H), 0.75–0.87 (t, $J = 7.2$ Hz, 3H). NMR ($^{13}C\{^1H\}$, 75 MHz, $CDCl_3$): δ 151.25, 141.01, 140.71, 139.91, 129.96, 128.83, 119.39, 45.01, 31.98, 25.88, 13.66.

2.4.5. *Ethyl2-[bis(4-chlorophenyl)methylselanyl]acetate*, $[(C_6H_4)_2CHSeCH_2COOCH_2CH_3]$ (**3h**). Yellow oil (78% yield). IR (ν_{max}/cm^{-1} , KBr): 3422, 2926, 1731, 1489, 1404, 1363, 1265, 1256, 1093, 1033, 793. MS-EI, *m/e* (RI, assignment): 359 (34, $[C_{16}H_{16}SeCl_2]^+$), 357 (6, $[C_{14}H_{12}SeCl_2]^+$), 330 (16, $[C_{14}H_{11}SeCl_2]^+$), 288 (6, $[C_{16}H_{16}Se]^+$), 279 (22, $[C_{16}H_{16}Cl_2]^+$), 273 (12, $[C_{15}H_{13}Se]^+ + 1$), 260 (7, $[C_{14}H_{10}Se]^+$). NMR (1H , 300 MHz, $CDCl_3$): δ 7.37–7.12 (m, 8H), 5.53 (s, 1H), 4.08–4.01 (q, $J = 7.2$ Hz, 2H), 2.82 (s, 2H), 1.23–1.16 (t, $J = 7.2$ Hz, 3H). NMR ($^{13}C\{^1H\}$, 75 MHz, $CDCl_3$): δ 170.74, 138.72, 138.68, 133.48, 130.14, 129.02, 128.84, 128.04, 61.11, 24.44, 14.32. NMR ($^{77}Se\{^1H\}$, 57 MHz, $CDCl_3$): δ 251.13.

2.4.6.

Ethyl3-[bis(4-chlorophenyl)methylselanyl]propanoate, $[(C_6H_4)_2CHSeCH_2CH_2COOCH_2CH_3]$ (**3i**). Yellow oil (65% yield). IR (ν_{max}/cm^{-1} , KBr): 3426, 2928, 2362, 1735, 1214, 1089, 793. NMR (1H , 300 MHz, $CDCl_3$): δ 7.27–7.17 (m, 8H), 5.31 (s, 1H), 4.09–4.02 (q, $J = 7.2$ Hz, 3H), 2.56–2.51 (m, 4H), 1.21–1.17 (t, $J = 7.2$ Hz, 3H). NMR ($^{13}C\{^1H\}$, 75 MHz, $CDCl_3$): δ 171.70, 141.01, 140.11, 133.01, 130.11, 128.79, 128.74, 128.72, 127.37, 60.55, 47.19, 35.14, 19.72, 14.42. NMR ($^{77}Se\{^1H\}$, 57 MHz, $CDCl_3$): δ 305.58.

2.4.7.

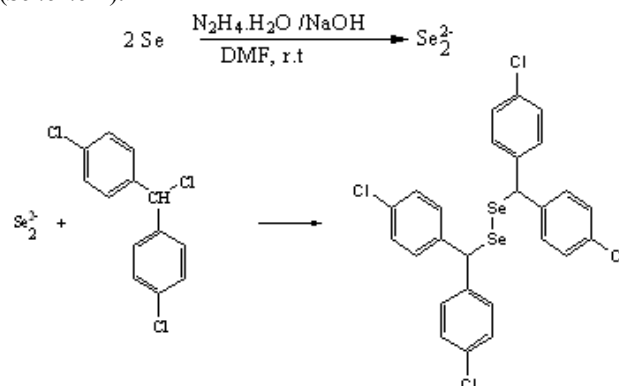
Ethyl4-[bis(4-chlorophenyl)methylselenyl]butanoate,

$[(C_6H_4)_2CHSeCH_2CH_2CH_2COOCH_2CH_3]$ (**3j**). Yellow oil (50% yield). IR (ν_{max}/cm^{-1} , KBr): 3432, 3028, 2925, 2250, 2076, 1720, 1599, 1490, 1451, 1408, 1203, 1090, 104, 909, 789, 735, 553, 502. NMR (1H , 300 MHz, $CDCl_3$): δ 7.25–7.118 (m, 8H), 5.22 (s, 1H), 4.08–3.96 (q, $J = 6.9$ Hz, 2H), 3.51–3.47 (t, $J = 6.9$ Hz, 2H), 2.92–2.22 (t, $J = 6.9$ Hz, 2H), 1.85–1.75 (m, 2H), 1.18–1.13 (t, $J = 6.9$ Hz, 3H). NMR ($^{13}C\{^1H\}$, 75 MHz, $CDCl_3$): δ 172.50, 139.66, 133.09, 129.92, 128.81, 127.90, 60.36, 34.05, 25.02, 14.32.

3. Results and Discussion

3.1. Synthesis

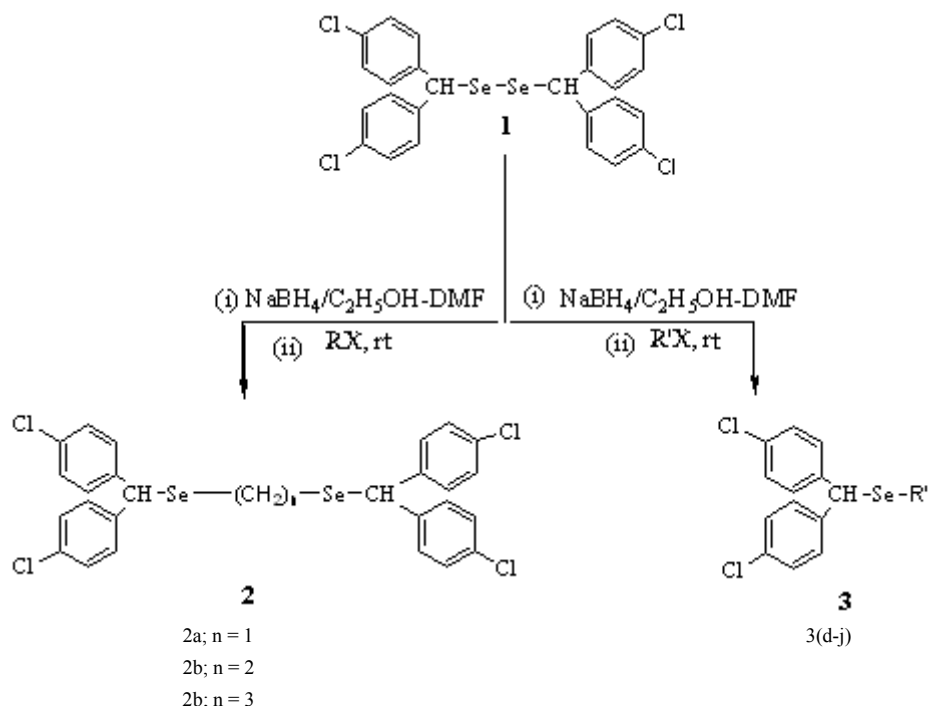
Herein, we are presenting an efficient procedure for the synthesis of 1,2-bis(bis(4-chlorophenyl) methyl) diselane (**1**) by the reduction of elemental selenium with hydrazine hydrate followed by alkylation using 4,4'-dichloro diphenylmethyl chloride. Studies indicate that out of all the reducing agent known for reduction of selenium *viz.*, alkali metals in ammonia, samarium iodide, rongalite and NaOH/PEG-400 etc., hydrazine hydrate was found to be most effective. [18] The diselenide ion Se_2^{2-} generated by the reduction of selenium using alkaline hydrazine hydrate in DMF upon *in situ* alkylation affords the 1,2-bis(bis(4-chlorophenyl)methyl) diselane in good yield (*Scheme 1*).



Scheme 1. Reaction Scheme for the synthesis of 1,2-bis[bis(4-chlorophenyl)methyl] diselane

Under similar reaction conditions, an attempt has been made to prepare the corresponding ditelluride. However, it could not be isolated as its rapid detelluration leads to the formation of 1,1,2,2-tetraphenylethane. The instability of 1,2-bis(bis(4-chlorophenyl)methyl)ditellane can be attributed to low bond dissociation energy of a highly sterically constrained aliphatic C-Te bond.

We have recently reported the facile synthesis of phenyl/benzylselenoalkanoates [19] and mono- and spiro β -lactam [20]. Taking a leaf out of this, we present here the synthesis of some novel selenoalkanoates (**3i-3j**). Starting substrate 1,2-bis(bis(4-chlorophenyl)methyl) diselane (**1**) was subjected to reductive cleavage with sodium borohydride for the synthesis of a variety of symmetrical/un-symmetrical dichloro diphenylmethyl selenides (*Scheme 2*).



Scheme 2. Reaction scheme for the synthesis of derivatives of 1,2-bis[bis(4-chlorophenyl) methyl] diselane

Table 1. Various symmetrical and unsymmetrical compounds synthesized by using variety of alkylating reagent (RX).

S. No.	Substrate	RX	Product	Yield (%)
1.	1	CH ₂ I ₂	2a	74
2.	1	Br(CH ₂) ₂ Br	2b	89
3.	1	Br(CH ₂) ₃ Br	2c	63
4.	1	CH ₃ I	3d	54
5.	1	CH ₃ CH ₂ Br	3e	78
6.	1	BrCH ₂ CH=CH ₂	3f	52
7.	1	CH ₃ CH ₂ CH ₂ CH ₂ Br	3g	63
8.	1	ClCH ₂ COOCH ₂ CH ₃	3h	78
9.	1	ClCH ₂ CH ₂ COOCH ₂ CH ₃	3i	65
10.	1	ClCH ₂ CH ₂ CH ₂ COOCH ₂ CH ₃	3j	50

The diselenide was made completely soluble using ethanol as a solvent. The *in situ* generation of selenolate ion forms a copious precipitates due to the poor solubility of borane complexed selenolate ion [(Ph₂CHSe)B(OC₂H₅)₃]⁻ [21]. However, use of dimethyl formamide (DMF) as a co-solvent, improved the yield of the unsymmetrical 1,2-bis(bis(4-chlorophenyl)methyl) selenides to two folds in most cases, mainly by solubilizing the 1,2-bis(bis(4-chlorophenyl)methyl) diselane ion [22] besides increasing their nucleophilicity. Dimethyl formamide being a highly polar solvent has been removed by simply washing with water.

It is interesting to note that most of these n-alkyl dichloro diphenyl methyl selenides (**3d-3j**) prepared undergo decomposition over a period of time to form symmetrical dialkyl diselenides and 1,1,2,2,4-tetraphenyl ethane with

traces the elemental selenium. This decomposition probably occurs by a radical mechanism that involves the cleavage of C-Se and Se-Se bond (*Scheme 3*). This is in agreement with the thermal and photochemical decomposition behaviour of dibenzyl diselenide and bis(4-diphenylmethyl) diselenide [23]. The primary process is presumed to involve the homolytic cleavage of C-Se bond towards dichloro diphenylmethyl carbon.

3.2. Spectroscopic Studies

All the compounds prepared were characterized by various spectroscopic techniques *viz.*, ¹H NMR, ¹³C{¹H} NMR, ⁷⁷Se{¹H} NMR and IR. spectroscopy. In the ¹H NMR spectra of these unsymmetrical dichloro diphenylmethyl selenides prepared, the methine protons resonate in the range of δ 5.12-5.51 ppm whereas, the aromatic protons display three different sets, two of which are multiplets appearing at δ 7.30-7.27 and δ 7.25-7.24 ppm while the third appears as a triplet in the region δ 7.21-7.20 ppm. However in symmetrical compounds, the methine proton shifts upward and the aromatic protons remain unaffected.

3.3. ⁷⁷Se-NMR Spectroscopic Analysis

Selenium atom is very sensitive to extremely small electronic and steric changes. Therefore, its chemical shift is a suitable tool to probe its chemical environment within the molecule. ⁷⁷Se{¹H} NMR was recorded in CDCl₃ employing dimethyl selenide as an external reference resonating at 0(δ, ppm). The selenium signal for compounds (1), (2c), (3d), (3f), and (3i) appears at δ 528.5, 339.1, 456.6, 251.1, and 305.5 ppm respectively

3.4. Mass Spectroscopic Analysis (EI-MS)

The mass spectra of all the compounds prepared are rich but complicated due to several isotopes of selenium and their different possible fragments. For most compounds analyzed, the molecular ion peak is very weak. The fragment corresponding to the mass of dichloro diphenylmethyl radical is most intense and appears invariably at 234, and exhibits the mixed dichloro pattern as well. The fragment ions containing selenium show a highly characteristic and definite pattern of signal intensities depending on the natural abundance of its various isotopes of selenium.

4. Conclusions

In this paper authors have used a convenient methodology for the synthesis of hitherto unknown 1,2-bis[bis(4-chlorophenyl)methyl]diselane and further a reductive cleavage of Se-Se bond of 1,2-bis[bis(4-chlorophenyl)methyl]diselane with sodium borohydride to generate selenide anion to synthesis variety of symmetrical and unsymmetrical analogues.

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