Synthesis of New Heterocycles containing more than one 1,2,3-Thia or Selenadiazole Rings

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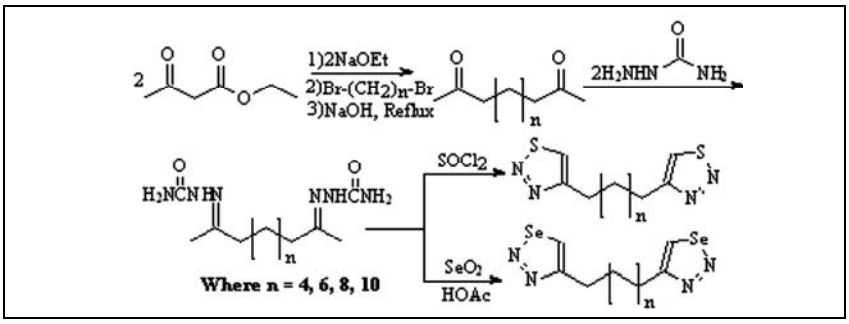

The diketones **2a-d** with different alkyl chain length are used for the synthesis of di-1,2,3-thia or selenadiazole derivatives **4a-d** and **5a-d**. The diketones **2a-d** where prepared by a unique method through the reaction between the corresponding dibromoalkanes **1a-d** and ethyl acetoacetate, which are transformed into the corresponding semicarbazone derivatives **3a-d**. The di-1,2,3-thia or selenadiazole derivatives **4a-d** and **5a-d** were prepared from the semicarbazones derivatives **3a-d** on oxidative cyclization with thionyl chloride and selenium dioxide respectively in high yield.

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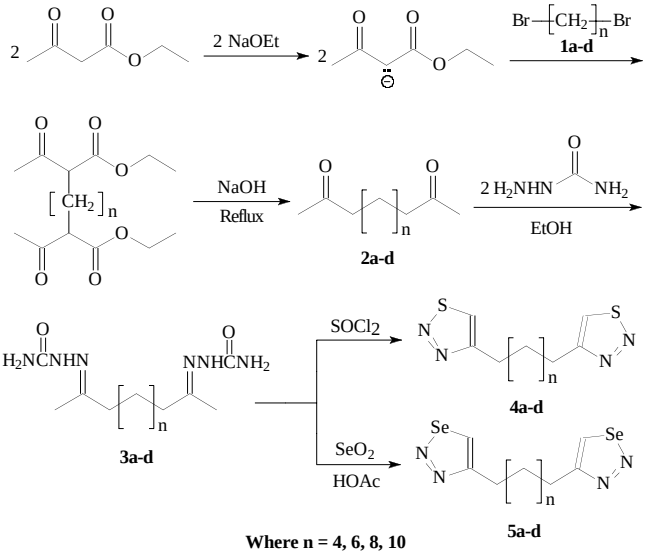
INTRODUCTION

Sulfur or selenium containing heterocyclic systems are of increasing interest because of their chemical properties [1] especially their wide application in synthesizing other heterocyclic compounds [2-7] and biological activities as antibacterial and antifangi [8-11]. Both sulfur and selenium compounds show wide similarities according to their applications in the syntheses of pharmaceuticals, dyes, and fine chemicals. However, remarkable differences between S- and Se-compounds are found. Due to the larger size of the Se-atom, selenium compounds show an increased polarizability, which leads to less stability compared to the S-analogues. Also, the characteristic physical properties of selena-heterocycles make them attractive materials in the development of organo-electric or organo-optic materials.

Multi-arm 1,2,3-thia and selenadiazole aromatic derivatives were recently prepared from the corresponding multi-arm ketones using both of the Hurd and Mori and the Lalezari and Shafiee methods respectively by Al-Smadi and Meier *et. al* [12-14], however heterocyclic systems containing two 1,2,3-selenadiazole rings were also recently prepared through reacting the corresponding multiple diazoketones with Lawesson reagent by Reddy *et. al* [15-16]. But di-1,2,3-selenadiazole derivatives with different alkyl spacers are still unknown. Therefore depending on a previous knowledge of the principal investigator in synthesizing multi-arm 1,2,3-thia and selenadiazoles, the analogous target compounds are prepared.

The preparation was carried out following the methods that are first reported by Lalezari *et. al.* [17-19] and Hurd and Mori [20], through reacting the corresponding semicarbazones or hydrazones of α-ketomethylene functionality, which contain aminocarbonyl or ethoxycarbonyl groups as good leaving groups with selenium dioxide in the presence of acetic acid or with thionyl chloride.

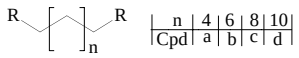
Scheme 1: Synthesis of compounds **4a-d** and **5a-d**

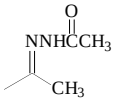
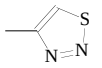
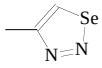


Our synthetic procedure of the heterocyclic compounds **4a-d** and **5a-d** started from the dibromoalkanes **1a-d**. The diketones **2a-d** were prepared by reacting the ethyl acetoacetate with equivalent amount of sodium ethanoate to generate the corresponding thermodynamically stable enolate derivatives which react with the dibromoalkanes **1a-d**, then hydrolysis with sodium hydroxide under reflux lead to the formation of the diketones **2a-d**. Condensation of **2a-d** with semicarbazide in absolute ethanol forms the corresponding semicarbazones **3a-d**. Treatment of **3a-d** with Thionyl chloride using the Hurd and Mori procedure provided compounds **4a-d**, whereas with selenium dioxide in acetic acid using Lalezari procedure provided the compounds **5a-d** in high chemical yield.

Table 1

Chemical percentage yield of the compounds **1-**, **2-**, **3-**, **4-** and **5a-d**.



R	Cpd*	Yield[%]	Cpd	Yield[%]	Cpd	Yield[%]	Cpd	Yield[%]
Br	**1a**		**1b**		**1c**		**1d**	
	**2a**	99	**2b**	97	**2c**	94	**2d**	93
	**3a**	95	**3b**	98	**3c**	86	**3d**	89
	**4a**	88	**4b**	81	**4c**	83	**4d**	86
	**5a**	75	**5b**	78	**5c**	68	**5d**	66

[Cpd*] is compound

RESULTS AND DISCUSSION

New five-membered heterocyclic compounds **4a-d** and **5a-d** containing two 1,2,3-thiadiazole or selenadiazole rings that are separated by different alkyl spacers were synthesized and characterized. The synthesis of compounds **4a-d** and **5a-d** was carried out in a multi-step procedure starting from the corresponding diketones **2a-d** by applying the Hurd-Mori or the Lalezari methods respectively. The diketones **2a-d** have all been prepared in high chemical yield using a unique method starting from the corresponding dibromoalkanes **1a-d**. All the compounds **4a-d** and **5a-d** were obtained in a high chemical yield. The compounds **4a-d** and **5a-d** exhibit aromatic character and have the ability to eliminate molecules of nitrogen with ring opening if heated above 140 °C to form new heterocycles. Therefore, these compounds can be used for crosslinking of polymers to prepare negative photoresist material and they can be used as nucleus for synthesis of new dendrimers. Since the compounds **4a-d** and **5a-d** are solid, it was found that the mp of the compound increases as the number of carbon atoms in the molecule increases. Also, it was found that the mp of the 1,2,3-thiadiazole derivatives is higher than that for the corresponding 1,2,3-selenadiazole derivatives. Due to structural similarities, the compounds **4a-d** all showed similar modes of stretching in their ir-spectra with small differences in the position and shape of the peaks. The same behavior was observed in case of compounds **5a-d**.

EXPERIMENTAL

Melting points (mp) were determined on an electrothermal digital melting point apparatus. Infrared (ir) spectra were recorded using a NICOLET 410 FT-IR spectrometer (ν in cm^{-1}). The ir spectra of pure substances were measured as KBr-pellets. The ^{1}H and ^{13}C nmr spectra were recorded on Bruker AM400 and AC200 spectrometers in deuteriochloroform or DMSO-d_6 with TMS as internal standard. The spectral data were reported in delta (δ) units relative to TMS reference line. The mass spectra were carried out by using the instrument MAT95 of the Finnigan Company (FD: 5 kV Ionizing energy, field desorption). Elemental analyses were performed in the analytical laboratory of the institute of organic chemistry of university of Mainz, Germany. Dibromoalkanes, semicarbazide hydrochloride and ethyl acetoacetate were obtained from Aldrich.

General Procedure for the Preparation of Di-Ketones (2a-d). Sodium (6.10 g, 265.10 mmol) were added in small portions to 150 mL absolute ethanol, then the whole mixture was refluxed to dissolve the remaining sodium pieces. Then an equivalent amount of ethyl acetoacetate (34.50 g, 265.10 mmol) was added drop wise over a 15 min. period untill a clear solution formed. After that (132.50 mmol) of the dibromoalkanes **1a-d** were added to the reaction mixture and left refluxing over night. A saturated sodium hydroxide solution was added to the reaction mixture and left refluxing such that a white solid was formed. The reaction was monitored using TLC in chloroform until completion. After cooling, the reaction mixture was diluted with water (50 mL) and extracted with diethyl ether (3 × 50 mL). The combined organic layers were dried over magnesium sulphate. The solvent was evaporated under vacuum and the residual solid was washed with diethyl ether. When necessary, a recrystallization from acetone or chloroform was performed.

2,9-Decandione (2a). This compound was obtained as colorless crystals (acetone), mp 53-54°; ir (potassium bromide): 2931, 2845 (C-H), 1696 (C=O), 1457, 1393, 1346, 1155 cm^{-1}; ^{1}H nmr (deuteriochloroform): δ 1.25 (s, 4H, CH_2), 1.53-1.57 (m, 4H, CH_2), 2.21 (s, 6H, CH_3), 2.43 (t, 4H, -CH_2-CO-); ^{13}C nmr (deuteriochloroform): δ 23.91 (CH_3), 29.16-29.81 (CH_2), 43.75 (-CH_2-CO-), 209.33 (C=O); ms: (5 kV, fd) m/z (%) 170 (100). *Anal.* Calcd. for $C_{10}H_{18}O_2$: C, 70.55; H, 10.66. Found: C, 70.28; H, 10.81.

2,11-Dodecandione (2b). This compound was obtained as pale yellow powder, mp 62-63°; ir (potassium bromide): 2933, 2847 (C-H), 1697 (C=O), 1456, 1394, 1348, 1155 cm^{-1}. ^{1}H nmr (deuteriochloroform): δ 1.24 (s, 8H, CH_2), 1.51-1.55 (m, 4H, CH_2), 2.18 (s, 6H, CH_3), 2.41 (t, 4H, -CH_2-CO-); ^{13}C nmr (deuteriochloroform): δ 23.89 (CH_3), 29.13-29.78 (CH_2), 43.76 (-CH_2-CO-), 209.35 (C=O); ms: (5 kV, fd) m/z (%) 198 (100).

Anal. Calcd. for $C_{12}H_{22}O_2$: C, 72.68; H, 11.18. Found: C, 72.36; H, 11.48.

2,13-Tetradecandione (**2c**)**.** This compound was obtained as colorless powder, m.p 71-72°; ir (potassium bromide): 2927, 2843 (C-H), 1695 (C=O), 1453, 1393, 1349, 1157 cm^{-1}. ^1H nmr (deuteriochloroform): δ 1.23 (s, 12H, CH_2), 1.52-1.57 (m, 4H, CH_2), 2.17 (s, 6H, CH_3), 2.39 (t, 4H, -CH_2-CO-); ^{13}C nmr (deuteriochloroform): δ 23.88 (CH_3), 29.19-29.84 (CH_2), 43.77 (-*C*H_2-CO-), 209.36 (C=O); ms: (5kV, fd) m/z (%) 226 (100). *Anal*. Calcd. for $C_{14}H_{26}O_2$: C, 74.29; H, 11.58. Found: C, 74.07; H, 11.29.

2,15-Hexadecandione (**2d**)**.** This compound was obtained as colorless powder, m.p 78-79; ir (potassium bromide): 2928, 2841 (C-H), 1692 (C=O), 1450, 1396, 1348, 1156 cm^{-1}. ^1H nmr (deuteriochloroform): δ 1.22 (s, 16H, CH_2), 1.51-1.55 (m, 4H, CH_2), 2.15 (s, 6H, CH_3), 2.38 (t, 4H, -CH_2-CO-); ^{13}C nmr (deuteriochloroform): δ 23.86 (CH_3), 29.16-29.81 (CH_2), 43.79 (-*C*H_2-CO-), 209.38 (C=O); ms: (5kV, fd) m/z (%) 254 (100). *Anal*. Calcd. for $C_{16}H_{30}O_2$: C, 75.54; H, 11.88. Found: C, 75.33; H, 11.59.

General procedure for the preparation of Disemicarbazones (**3a-d**)**.** Semicarbazide hydrochloride (4.44 g, 40.00 mmol) and sodium acetate (3.33 g, 40.00 mmol) were dissolved in absolute ethanol (50 mL). The obtained mixture was heated for 20 min under reflux. The resulting mixture was filtered while hot to remove precipitated sodium chloride. The filtrate was mixed with diketones **2a, 2b, 2c, 2d** (18.50 mmol), respectively. The reaction mixture was heated till refluxing then two drops of concentrated hydrochloric acid were added. The mixture was heated under reflux overnight with continuous removal of the water generated. Finally, the solvent was removed under vacuum and the residue was washed with diethyl ether.

2,9-Decandione disemicarbazone (**3a**)**.** This compound was obtained as white powder, mp 211-212°; ir (potassium bromide): 3408 (NH_2), 3243 (NH), 2944 (C-H), 1688 (C=O), 1574, 1418, 1279 cm^{-1}; ^1H nmr (dimethyl sulfoxide-d_6): δ 1.23 (s, 4H, CH_2), 1.53-1.57 (m, 4H, CH_2), 2.16 (s, 6H, CH_3), 2.39 (t, 4H, -CH_2-CO-), 6.50 (s, 4H, NH_2), 9.15 (s, 2H, NH); ^{13}C nmr (dimethyl sulfoxide-d_6): δ 23.91 (CH_3), 29.16-29.81 (CH_2), 43.75 (-*C*H_2-CO-), 144.00 (C=N), 158.70 (C=O); ms: (5 kV, fd) m/z (%) 284 (100). *Anal*. Calcd. for $C_{12}H_{24}N_6O_2$: C, 50.69; H, 8.51; N, 29.55. Found: C, 50.38; H, 8.29; N, 29.41.

2,11-Dodecandione disemicarbazone (**3b**)**.** This compound was obtained as white powder, mp 215-216°; ir (potassium bromide): 3410 (NH_2), 3249 (NH), 2946 (C-H), 1693 (C=O), 1579, 1425, 1283 cm^{-1}; ^1H nmr (dimethyl sulfoxide-d_6): δ 1.26 (s, 8H, CH_2), 1.52-1.55 (m, 4H, CH_2), 2.18 (s, 6H, CH_3), 2.40 (t, 4H, -CH_2-CO-), 6.53 (s, 4H, NH_2), 9.91 (s, 2H, N-H); ^{13}C nmr (dimethyl sulfoxide-d_6): δ 23.89 (CH_3), 29.13-29.78 (CH_2), 43.76 (-*C*H_2-CO-), 144.11 (C=N), 158.90 (C=O); ms: (5 kV, fd) m/z (%) 312 (100). *Anal*. Calcd. for $C_{14}H_{28}N_6O_2$: C, 53.82; H, 9.03; N, 26.90. Found: C, 53.63; H, 8.89; N, 26.75.

2,13-Tetradecandione disemicarbazone (**3c**)**.** This compound was obtained as white powder, mp 219-220°; ir (potassium bromide): 3413 (NH_2), 3251 (NH), 2947 (C-H), 1696 (C=O), 1577, 1421, 1282 cm^{-1}; ^1H nmr (dimethyl sulfoxide-d_6): δ 1.27 (s, 12H, CH_2), 1.55-1.59 (m, 4H, CH_2), 2.19 (s, 6H, CH_3), 2.38 (t, 4H, -CH_2-CO-), 6.54 (s, 4H, NH_2), 9.17 (s, 2H, NH); ^{13}C nmr (dimethyl sulfoxide-d_6): δ 23.87 (CH_3), 29.15-29.79 (CH_2), 43.78 (-*C*H_2-CO-), 144.13 (C=N), 158.93 (C=O); ms: (5 kV, fd) m/z (%) 340 (100). *Anal*. Calcd. for $C_{16}H_{32}N_6O_2$: C, 56.44; H, 9.47; N, 24.68. Found: C, 56.17; H, 9.27; N, 24.34.

2,15-Hexadecandione disemicarbazone (**3d**)**.** This compound was obtained as white powder, mp 223-224°; ir (potassium bromide): 3417 (NH_2), 3262 (NH), 2948 (C-H), 1698 (C=O), 1579, 1429, 1291 cm^{-1}; ^1H nmr (dimethyl sulfoxide-d_6): δ 1.29 (s, 16H, CH_2), 1.57-1.69 (m, 4H, CH_2), 2.21 (s, 6H, CH_3), 2.43 (t, 4H, -CH_2-CO-), 6.51 (s, 4H, NH_2), 9.85 (s, 2H, NH); ^{13}C nmr (dimethyl sulfoxide-d_6): δ 23.91 (CH_3), 29.16-29.81 (CH_2), 43.79 (-*C*H_2-CO-), 144.30 (C=N), 158.98 (C=O); ms: (5 kV, fd) m/z (%) 368 (100). *Anal*. Calcd. for $C_{18}H_{36}N_6O_2$: C, 58.67; H, 9.84; N, 22.80. Found: C, 58.46; H, 9.67; N, 22.71.

General procedure for preparation of Di-1,2,3-Thiadiazoles (4a-d). Semicarbazones **3a** or **3b** or **3c** or **3d** respectively (0.36 mmol) was dissolved in thionyl chloride (25 mL) with vigorous stirring and cooling 0 °C. The mixture was allowed to warm up to room temperature with continuous stirring. Monitoring of reaction progress with TLC showed that the reaction was completed in 10 hours at which time the solvent was removed under vacuum. The crude product was washed with diethyl ether and recrystallized from chloroform and petroleum ether when necessary.

1,6-Bis(1,2,3-thiadiazole-4-yl)hexane (**4a**)**.** This compound was obtained as pale-yellow powder, mp 85-86°; ir (potassium bromide): 2958, 2908, 1576, 1468, 1433, 1223, 982 cm^{-1}; ^1H nmr (deuteriochloroform): δ 1.38 (s, 4H, CH_2), 2.74-2.77 (m, 4H, CH_2), 3.21 (t, 4H, CH_2), 8.26 (s, 2H, CHS); ^{13}C nmr (deuteriochloroform): δ 28.31-29.64 (CH_2), 37.45 (CH_2), 131.29 (C5), 164.32 (C4); ms: (5 kV, fd) m/z (%) 254 (100). *Anal*. Calcd. For $C_{10}H_{14}N_4S_2$: C, 47.22; H, 5.54; N, 22.02; S, 25.21. Found: C, 47.13; H, 5.25; N, 22.20; S, 25.36.

1,8-Bis(1,2,3-thiadiazole-4-yl)octane (**4b**)**.** This compound was obtained as yellow-orange solid, mp 88-89°; ir (potassium bromide): 2959, 2879, 1580, 1477, 1424, 1229, 971 cm^{-1}; ^1H nmr (deuteriochloroform): δ 1.37 (s, 8H, CH_2), 2.74-2.78 (m, 4H, CH_2), 3.18 (t, 4H, CH_2), 8.21 (s, 2H, CHS); ^{13}C nmr (deuteriochloroform): δ 28.29-29.66 (CH_2), 37.43 (CH_2), 131.27 (C5), 164.30 (C4); ms: (5 kV, fd) m/z (%) 282 (100). *Anal*. Calcd. For $C_{12}H_{18}N_4S_2$: C, 51.03; H, 6.42; N, 19.84; S, 22.71. Found: C, 51.18; H, 6.38; N, 19.59; S, 22.83.

1,10-Bis(1,2,3-thiadiazole-4-yl)decane (**4c**)**.** This compound was obtained as pale yellow solid, mp 91-92°; ir (potassium bromide): 2966, 2877, 1583, 1475, 1433, 1224, 976 cm^{-1}; ^1H nmr (deuteriochloroform): δ 1.32 (s, 12H, CH_2), 2.70-2.75 (m, 4H, CH_2), 3.15 (t, 4H, CH_2), 8.17 (s, 2H, CHS); ^{13}C nmr (deuteriochloroform): δ 28.26-29.63 (CH_2), 37.39 (CH_2), 131.24 (C5), 164.27 (C4); ms: (5kV, fd) m/z (%) 310 (100). *Anal*. Calcd. For $C_{14}H_{22}N_4S_2$: C, 54.16; H, 7.14; N, 18.04; S, 20.66. Found: C, 54.04; H, 7.35; N, 18.31; S, 20.48.

1,12-Bis(1,2,3-thiadiazole-4-yl)dodecane (**4d**)**.** This compound was obtained as beige powder, mp 93-94°; ir (potassium bromide): 2962, 2875, 1580, 1472, 1435, 1220, 972 cm^{-1}; ^1H nmr (deuteriochloroform): δ 1.30 (s, 16H, CH_2), 2.71-2.74 (m, 4H, CH_2), 3.12 (t, 4H, CH_2), 8.13 (s, 2H, CHS); ^{13}C nmr (deuteriochloroform): δ 28.27-29.64 (CH_2), 37.38 (CH_2), 131.21(C5), 164.25(C4); ms: (5kV, fd) m/z (%) 338 (100). *Anal*. Calcd. For $C_{16}H_{26}N_4S_2$: C, 56.77; H, 7.74; N, 16.55; S, 18.94. Found: C, 56.71; H, 7.63; N, 16.41; S, 18.83.

General procedure for preparation of Di-1,2,3-Selenadiazoles (5a-d). Semicarbazones **3a** or **3b** or **3c** or **3d** respectively (0.36 mmol) was dissolved in glacial acetic acid (35 mL) with vigorous stirring and gentle heating 40-44 °C. The solution was treated with selenium dioxide powder (0.84 mmol) and the mixture was kept under gentle heating with vigorous stirring. After 2 min, the color of the mixture becomes red.

Monitoring reaction progress using TLC showed that the reaction was completed in 30 hours at which time the mixture was filtered and the filtrate was poured over ice water and extracted with chloroform (3 × 60 mL). The combined organic layers were washed with saturated sodium hydrogen carbonate solution and dried using magnesium sulphate. The solvent was removed under vacuum. The crude product was chromatographed using ethyl acetate as eleuent. The recrystalization was followed from chloroform/hexane.

1,6-Bis(1,2,3-selenadiazole-4-yl)hexane (5a). This compound was obtained as pale yellow-brown solid, mp 70-71°; ir (potassium bromide): 2938, 2858, 1686, 1428, 1233 cm^{-1}; ^1H nmr (deuteriochloroform): δ 1.28 (s, 4H, CH_2), 2.55-2.59 (m, 4H, CH_2), 3.12 (t, 4H, CH_2), 8.79 (s, 2H, CHSe); ^{13}C nmr (deuteriochloroform): δ 27.31-28.64 (CH_2), 36.45 (CH_2), 133.29 (C5), 162.31 (C4); ms: (5 kV, fd) m/z (%) 348 (100). *Anal.* Calcd. For $C_{10}H_{14}N_4Se_2$: C, 34.50; H, 4.05; N, 16.09; Se, 45.36. Found: C, 34.33; H, 4.15; N, 16.20.

1,8-Bis(1,2,3-selenadiazole-4-yl)octane (5b). This compound was obtained as yellow-brown solid, mp 72-73°; ir (potassium bromide): 2939, 2859, 1690, 1427, 1234 cm^{-1}; ^1H nmr (deuteriochloroform): δ 1.29 (s, 8H, CH_2), 2.56-2.60 (m, 4H, CH_2), 3.13 (t, 4H, CH_2), 8.79 (s, 2H, CHSe); ^{13}C nmr (deuteriochloroform): δ 27.29-28.56 (CH_2), 36.44 (CH_2), 133.26 (C5), 162.30 (C4); ms: (5 kV, fd) m/z (%) 376 (100). *Anal.* Calcd. For $C_{12}H_{18}N_4Se_2$: C, 38.31; H, 4.82; N, 14.89; Se, 41.97. Found: C, 38.11; H, 4.62; N, 14.59.

1,10-Bis(1,2,3-selenadiazole-4-yl)decane (5c). This compound was obtained as light brown solid, mp 74-75°; ir (potassium bromide): 2936, 2857, 1693, 1425, 1233 cm^{-1}; ^1H nmr (deuteriochloroform): δ 1.25 (s, 12H, CH_2), 2.54-2.58 (m, 4H, CH_2), 3.09 (t, 4H, CH_2), 8.78 (s, 2H, CHSe); ^{13}C nmr (deuteriochloroform): δ 27.26-28.53 (CH_2), 36.43 (CH_2), 133.24 (C5), 162.27 (C4); ms: (5kV, fd) m/z (%) 404 (100). *Anal.* Calcd. For $C_{14}H_{22}N_4Se_2$: C, 41.59; H, 5.49; N, 13.86; Se, 39.06. Found: C, 41.26; H, 5.25; N, 13.61.

1,12-Bis(1,2,3-selenadiazole-4-yl)dodecane (5d). This compound was obtained as brown solid, mp 76-77°; ir (potassium bromide): 2923, 2846, 1696, 1423, 1230 cm^{-1}; ^1H nmr (deuteriochloroform): δ 1.21 (s, 16H, CH_2), 2.51-2.56 (m, 4H, CH_2), 3.04 (t, 4H, CH_2), 8.76 (s, 2H, CHSe); ^{13}C nmr (deuteriochloroform): δ 27.23-28.54 (CH_2), 36.41 (CH_2), 133.21(C5), 162.25 (C4); ms: (5kV, fd) m/z (%) 432 (100). *Anal.* Calcd. For $C_{16}H_{26}N_4Se_2$: C, 44.45; H, 6.06; N, 12.96; Se, 36.53. Found: C, 44.71; H, 6.14; N, 12.69.

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