New Multi-1,2,3-Selenadiazole Aromatic Derivatives

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Abstract: The aromatic polyketones 3a-d are versatile compounds for the synthesis of the multi-1,2,3-selenadiazole aromatic derivatives 1a-d. The preparation starts with the reaction between the multi-bromomethylene benzene derivatives 2a-d and 4-hydroxyacetophenone to give compounds 3a-d which are transformed through the reaction with semicarbazide hydrochloride or ethyl hydrazine carboxylate into the corresponding semicarbazones derivatives 4a-d or hydrazones 5a-d. The reaction with selenium dioxide leads to regiospecific ring closure of semicarbazones or hydrazones to give the multi-1,2,3-selenadiazole aromatic derivatives in high yield.

Keywords: 1,2,3-Selenadiazoles; semicarbazones; aromatic derivatives

Introduction

Heterocyclic systems with multi-arm 1,2,3-thiadiazoles were recently prepared by Meier et. al [1-2] and heterocyclic systems containing two 1,2,3-selenadiazole rings were also recently prepared by Reddy et. al [3-4], but multi-arm 1,2,3-selenadiazoles are still unknown. Therefore depending on the previous experience of the principal investigator in synthesizing multi-arm 1,2,3-thiadiazoles, the analogous multi-arm selenadiazoles were prepared following the method that was first reported by Lalezari et. al [5-7], through reaction in the presence of acetic acid of selenium dioxide with α-ketomethylene semicarbazones or hydrazones which contain aminocarbonyl or ethoxycarbonyl groups as good leaving groups. NOE measurements showed that the (E)-configuration largely predominated around the CN double bond.
Selenium containing heterocycles are of increasing interest because of their interesting chemical properties [8-13] and varied biological activities [14-16]. Remarkable differences are known to exist between Se- and S-containing compounds. Due to the larger size of the Se-atom, selenium compounds show an increased polarizability and therefore they are, in general, less stable than the corresponding S-analogues [17-20]. We report herein on our efforts to generate the multi-branched benzene derivatives 1a, 1b, 1c and 1d, in which the 1,2,3-selenadiazole rings are linked to the central benzene core via phenoxyethylene spacers.

**Scheme 1**

Our synthetic procedure (see Scheme 1) started from the commercially available bromomethyl-benzene derivatives 2a-2d. Multiple substitution with 4-hydroxyacetophenone gave the corresponding polyketones 3a-3d, which were transformed into the target compounds 1a-1d by the reaction of the

| Series | n  |
|--------|----|
| a      | 6  |
| b      | 4  |
| c      | 3  |
| d      | 2  |

**Results and Discussion**

Our synthetic procedure (see Scheme 1) started from the commercially available bromomethyl-benzene derivatives 2a-2d. Multiple substitution with 4-hydroxyacetophenone gave the corresponding polyketones 3a-3d, which were transformed into the target compounds 1a-1d by the reaction of the
corresponding semicarbazones 4a-4d or ethoxycarbonyl hydrazones 5a-5d, essentially as described by Lalezari et. al. [5-7]. The yields of all three steps were optimized, so that the total overall yields for the sequences 2a → 1a, 2b → 1b, 2c → 1c and 2d → 1d amounted to 90%, 85%, 89% and 85%, respectively (Table 1).

### Table 1

| Ring system | Cpd. | Yield | Cpd. | Yield | Cpd. | Yield | Cpd. | Yield |
|-------------|------|-------|------|-------|------|-------|------|-------|
| R           |      |       |      |       |      |       |      |       |
| Br          | 2a   | —     | 2b   | —     | 2c   | —     | 2d   | —     |
| O           | 3a   | 87    | 3b   | 90    | 3c   | 95    | 3d   | 89    |
| N           | 4a   | 91    | 4b   | 81    | 4c   | 96    | 4d   | 98    |
| Se          | 5a   | 92    | 5b   | 87    | 5c   | 85    | 5d   | 91    |
| OCH2CH3     | 1a   | 96    | 1b   | 92    | 1c   | 87    | 1d   | 60    |

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**Experimental**

**General**

The solvents were purified by standard procedures. The melting points (m.p.) were determined on an Electrothermal digital melting point apparatus and are uncorrected. Infrared (IR) spectra of pure substances were recorded as KBr-pellets using a Nicolet 410 FT-IR spectrometer (ν in cm⁻¹). The ¹H- and ¹³C-NMR spectra were recorded on Bruker AM400 and AC200 spectrometers in CDCl₃ or DMSO-d₆ using TMS as internal standard. The spectral data are reported in delta (δ) units relative to
the TMS reference peak. The mass spectra were recorded using a Finnigan MAT95 field desorption (FD, 5 kV ionizing energy) instrument. The signals are given as m/z with the relative intensity between brackets. Elemental analyses were performed in the Analytical Laboratory of the Institute of Organic Chemistry of University of Mainz, Germany. Bromo compounds 2a-2d (1,2,3,4,5,6-hexakis-, 1,2,4,5-tetrakis-, 1,3,5-tri- and 1,4-dibromomethylbenzene, respectively), ethyl hydrazine carboxylate, semicarbazide hydrochloride and sodium acetate were obtained from Aldrich.

**General Procedure for the Preparation of Multi-Ketones 3a-d [1]**

A mixture of 4-hydroxyacetophenone (1 equivalent) and 2a (0.14 equivalents), 2b (0.21 equivalents), 2c (0.3 equivalents) or 2d (0.45 equivalents), potassium carbonate (1 equivalent) and potassium iodide (in the same equivalent amount as the bromo compound used) plus a few drops of Aliquat 336 were refluxed in dry acetone (100 mL) for 48 hours. The reaction was followed by TLC (eluent: chloroform) till completion. After cooling, the reaction mixture was diluted with water (50 mL) and extracted with dichloromethane (3 × 40 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.

**1-{4-[2,3,4,5,6-Penta(4-acetylphenoxymethyl)benzyloxy]phenyl}-1-ethanone (3a).**

Colorless powder (87% yield), m.p. 234-235°C; IR: v 1675, 1604, 1508, 1239, 1002, 832 cm⁻¹; ¹H-NMR (CDCl₃): 2.50 (s, 3H, CH₃), 5.26 (s, 2H, CH₂O), 6.89, 7.83 (d, d, 2H, 2H, AA'BB''); ¹³C-NMR (CDCl₃): 26.20 (CH₃), 63.60 (CH₂O), 137.70 (central benzene ring Cq), 114.21 and 130.70 (side chain benzene CH), 131.30, 161.80 (side chain benzene Cq), 196.40 (C=O); MS: 967 (M⁺, 100); Anal. % Calcd. for C₆₀H₅₄O₁₂: C, 74.52; H, 5.63. Found: C, 74.27; H, 5.54.

**1-{4-[2,4,5-Tri(4-acetylphenoxymethyl)benzyloxy]phenyl}-1-ethanone (3b).**

Colorless powder (90% yield); m.p. 224-226°C; IR: v 1665, 1604, 1502, 1250, 1170, 830 cm⁻¹; ¹H-NMR (CDCl₃): 2.53 (s, 3H, CH₃), 5.23 (s, 2H, CH₂O), 6.94, 7.89 (d, d, 2H, 2H, AA'BB''), 7.65 (s, benzene ring central CH); ¹³C-NMR (CDCl₃): 26.30 (CH₃), 67.70 (CH₂O), 129.80 (central benzene ring CH), 135.10 (central benzene ring Cq), 114.41 and 130.60 (side chain benzene CH), 131.10, 162.10 (side chain benzene Cq), 196.40 (C=O); MS: 670 (M⁺, 100); Anal. % Calcd. for C₄₂H₃₈O₈: C, 75.21; H, 5.71. Found: C, 75.13; H, 5.59.

**1-{4-[3,5-Di(4-acetylphenoxymethyl)benzyloxy]phenyl}-1-ethanone (3c).**

Pale yellow powder (100% yield); m.p. 82-83°C; IR: v 2910, 1670, 1591, 1500, 1250, 1172, 836 cm⁻¹. ¹H-NMR (DMSO-d₆): 2.49 (s, 3H, CH₃), 5.22 (s, 2H, CH₂O), 7.09, 7.90 (d, d, 2H, 2H, AA'BB''), 7.53 (s, benzene ring central CH); ¹³C-NMR (DMSO-d₆): 26.30 (CH₃), 69.20 (CH₂O), 126.60 (central benzene ring CH), 137.20 (central benzene ring Cq), 114.61 and 130.50 (side chain benzene CH), 130.10, 162.10 (side chain benzene Cq), 196.50 (C=O); MS: 523 (M⁺, 100); Anal. % Calcd. for C₃₃H₃₀O₆: C, 75.84; H, 5.79. Found: C, 75.81; H, 5.68.
1-\{4-\text{Mono-(4-acetylphenoxymethyl) benzyl oxy phenyl}\}-1-ethanone (3d).

Colorless crystals (89% yield); m.p. 181-182\(^\circ\)C; IR: v 1668, 1591, 1239, 996, 823 cm\(^{-1}\); \(^1\)H-NMR (CDCl\(_3\)): 2.53 (s, 3H, CH\(_3\)), 5.12 (s, 2H, CH\(_2\)O), 7.00, 7.90 (d, d, 2H, 2H, AA'BB'), 7.44 (d, benzene ring central CH); \(^13\)C-NMR (CDCl\(_3\)): 26.40 (CH\(_3\)), 69.70 (CH\(_2\)O), 127.80 (central benzene ring CH), 136.20 (central benzene ring Cq), 114.50 and 130.60 (side chain benzene CH), 130.60, 162.40 (side chain benzene Cq), 196.70 (C=O); MS: 374 (M\(^+\), 100); Anal. % Calcd. for C\(_{24}\)H\(_{22}\)O\(_4\): C, 76.99; H, 5.92. Found: C, 76.78; H, 5.81.

General procedure for the preparation of multiple semicarbazones 4a-d

A mixture of semicarbazide hydrochloride (1 equivalent) and sodium acetate (1 equivalent) was dissolved in absolute ethanol (40 mL). The mixture was heated for 15 min under reflux, then filtered while hot to remove precipitated sodium chloride. The filtrate was mixed with ketone 3a (0.14 equivalents), ketone 3b (0.21 equivalents), ketone 3c (0.30 equivalents) or ketone 3d (0.45 equivalents), respectively. The reaction mixture was heated to reflux then two drops of concentrated hydrochloric acid were added. The mixture was heated under reflux for overnight with continuously removal of generated water. After that the solvent was removed under vacuum and the residue was washed with diethyl ether.

1-\{4,2,3,4,5,6-Penta(4-acetylphenoxymethyl benzyl oxy)phenyl\}-1-ethanone-N-aminocarbonyl semicarbazone (4a).

White powder (91% yield), m.p. 300\(^\circ\)C (dec.); IR: v 3417, 3212, 1687, 1597, 1431, 1239, 1014, 829 cm\(^{-1}\); \(^1\)H-NMR (DMSO-d\(_6\)): 2.08 (s, 3H, CH\(_3\)-C=N)), 5.37 (s, 2H, CH\(_2\)O), 6.41 (s, 2H, NH\(_2\)), 6.93, 7.70 (d, d, 2H, 2H, AA'BB'), 9.18 (s, 1H, N-H); \(^13\)C-NMR (DMSO-d\(_6\)): 13.29 (CH\(_3\)-C=N), 63.90 (CH\(_2\)O), 137.60 (central benzene ring Cq), 114.30 and 127.50 (side chain benzene CH), 130.40, 159.60 (side chain benzene Cq), 143.90 (C=N), 157.40 (C=O); MS: 1309.4 (M\(^+\), 100); Anal. % Calcd. for C\(_{66}\)H\(_{72}\)N\(_{18}\)O\(_{12}\): C, 60.54; H, 5.54; N, 19.25. Found: C, 60.35; H, 5.54; N, 19.30.

1-\{4,2,4,5-Tri(4-acetylphenoxymethyl benzyl oxy)phenyl\}-1-ethanone-N-aminocarbonyl semicarbazone (4b).

White powder (81% yield); m.p. 250\(^\circ\)C (dec.); IR: v 3429, 3180, 1670, 1599, 1418, 1258, 836 cm\(^{-1}\); \(^1\)H-NMR (DMSO-d\(_6\)): 2.12 (s, 3H, CH\(_3\)-C=N)), 5.34 (s, 2H, CH\(_2\)O), 5.99 (s, 2H, NH\(_2\)), 7.06, 7.86 (d, d, 2H, 2H, AA'BB'), 7.71 (s, central benzene ring CH), 9.19 (s, 1H, N-H); \(^13\)C-NMR (DMSO-d\(_6\)): 13.38 (CH\(_3\)-C=N), 67.08 (CH\(_2\)O), 129.10 (central benzene ring CH), 134.80 (central benzene ring Cq), 114.70 and 127.50 (side chain benzene CH), 130.40, 159.60 (side chain benzene Cq), 144.00 (C=N), 157.50 (C=O); MS: 899 (M\(^+\), 100); Anal. % Calcd. for C\(_{46}\)H\(_{50}\)N\(_{12}\)O\(_{8}\): C, 62.33; H, 5.67; N, 18.17. Found: C, 62.29; H, 5.61; N, 18.05.
1-4-{3,5-Di(4-acetylphenoxymethyl)benzoyl}phenyl)-1-ethanone-N-aminocarbonyl semicarbazone (4c).

Pale yellow powder (100% yield); m.p. 240°C (dec.); IR: ν 3417, 3250, 1681, 1579, 1508, 1470, 1226, 829 cm⁻¹; ¹H-NMR (DMSO-d₆): 2.13 (s, 3H, CH₃-C=N), 5.15 (s, 2H, CH₂O), 6.55 (s, 2H, NH₂), 6.98, 7.76 (d, d, 2H, 2H, AA′BB′), 7.49 (s, central benzene ring CH), 9.25 (s, 1H, N-H); ¹³C-NMR (DMSO-d₆): 13.38 (CH₃-C=N), 69.15 (CH₂O), 126.44 (central benzene ring CH), 137.76 (central benzene ring Cq), 114.54 and 127.50 (side chain benzene CH), 131.20, 158.73 (side chain benzene Cq), 144.11 (C=O), 157.90 (C=O); MS: 693.77 (M⁺, 100); Anal. % Calcd. for C₃₆H₃₉N₉O₆: C, 61.46; H, 5.61; N, 18.70; Found: C, 63.32; H, 5.51; N, 18.63.

1-4-{4-(4-acetylphenoxymethyl)benzoyl}phenyl)-1-ethanone-N-aminocarbonyl semicarbazone (4d).

White powder (100% yield); m.p. 300°C (dec.); IR: ν 3417, 3212, 1681, 1604, 1502, 1418, 1239, 1014, 829 cm⁻¹; ¹H-NMR (DMSO-d₆): 2.12 (s, 3H, CH₃-C=N), 5.20 (s, 2H, CH₂O), 5.90 (s, 2H, NH₂), 7.08, 7.89 (d, d, 2H, 2H, AA′BB′), 7.45 (d, central benzene ring CH), 9.21 (s, 1H, N-H); ¹³C-NMR (DMSO-d₆): 13.30 (CH₃-C=N), 69.30 (CH₂O), 127.80 (central benzene ring CH), 136.41 (central benzene ring Cq), 114.50 and 128.00 (side chain benzene CH), 131.17, 159.70 (side chain benzene Cq), 143.00 (C=N), 158.70 (C=O); MS: 488.5 (M⁺, 100); Anal. % Calcd. for C₂₆H₂₈N₆O₄: C, 63.92; H, 5.78; N, 17.20; Found: C, 63.71; H, 5.72; N, 17.15.

General procedure for the preparation of multiple hydrazones 5a-d [1].

A solution of di- or tri- or tetra- or hexaketo 3a-d (1 equivalent), a few drops of concentrated hydrochloric acid and 6, 9, 12 or 18 equivalents of ethyl hydrazinecarboxylate in dry chloroform (50 mL) was heated under reflux overnight with continuously removal of generated water. The solution was concentrated and the residue was washed with diethyl ether and chloroform.

1-4-{2,3,4,5,6-Penta(4-acetylphenoxymethyl)benzoyl}phenyl)-1-ethanone-N-ethoxycarbonyl hydrazone (5a).

White solid powder (92% yield); m.p. 293°C (dec.); IR: ν 3231, 1719, 1610, 1502, 1233, 1047, 829 cm⁻¹; ¹H-NMR (DMSO-d₆): 1.23 (t, 3H, CH₃CH₂), 2.12 (s, 3H, CH₃-C=N), 4.13 (q, 2H, CH₃CH₂), 5.29 (s, 2H, CH₂O), 6.95, 7.58 (d, d, 2H, 2H, AA′BB′), 9.96 (s, 1H, N-H); ¹³C-NMR (DMSO-d₆): 14.23 (CH₃CH₂), 15.01 (CH₃-C=N), 60.95 (CH₂CH₂O), 64.36 (CH₂O-ph), 138.09 (central benzene ring Cq), 114.86 and 127.94 (side chain benzene CH), 131.93, 159.41 (side chain benzene Cq), 149.25 (C=N), 154.76 (CO₂); MS: 1484 (M⁺, 100); Anal. % Calcd. for C₇₈H₇₀N₁₂O₁₈: C, 63.15; H, 6.11; N, 11.33; Found: C, 62.79; H, 6.27; N, 11.44.
1-{4-[2,4,5-Tri-(4-acetylphenoxymethyl)benzyloxy]phenyl}-1-ethanone-N-ethoxycarbonyl hydrazone (5b).

White powder (87% yield), m.p. 211-213°C; IR: ν 3200, 3045, 1700, 1596, 1492, 1235, 1040, 828 cm⁻¹; ¹H-NMR (DMSO-d₆): 1.24 (t, 3H, CH₃CH₂), 2.16 (s, 3H, CH₃-C=N)), 4.16 (q, 2H, CH₂CH₂), 5.27 (s, 2H, CH₂O), 7.03, 7.65 (d, d, 2H, 2H, AA'BB'), 7.71 (s, central benzene ring CH), 9.98 (s, 1H, N-H); ¹³C-NMR (DMSO-d₆): 14.28 (CH₃CH₂), 15.19 (CH₃-C=N), 60.98 (CH₂CH₂O), 67.40 (CH₂O-ph), 129.37 (central benzene ring CH), 135.43 (central benzene ring Cq), 115.08 and 128.00 (side chain benzene CH), 131.76, 159.33 (side chain benzene Cq), 148.70 (C=O), 154.80 (C=O); MS: 1015 (M⁺, 100); Anal. % Calcd. for C₅₄H₆₂N₈O₁₂: C, 63.89; H, 6.16; N, 11.04, Found: C, 63.58; H, 6.09; N, 10.97.

1-{4-[3,5-Di(4-acetylphenoxymethyl)benzyloxy]phenyl}-1-ethanone-N-ethoxycarbonyl hydrazone (5c).

Pale yellow powder (85% yield), m.p. 202-204°C; IR: ν 3200, 3035, 1705, 1600, 1503, 1238, 1040, 830 cm⁻¹; ¹H-NMR (DMSO-d₆): δ 1.24 (t, 3H, CH₃CH₂), 2.17 (s, 3H, CH₃-C=N)), 4.15 (q, 2H, CH₃CH₂), 5.18 (s, 2H, CH₂O), 7.03, 7.67 (d, d, 2H, 2H, AA'BB'), 7.52 (s, central benzene ring CH), 9.99 (s, 1H, N-H); ¹³C-NMR (DMSO-d₆): δ 14.30 (CH₃CH₂), 15.20 (CH₃-C=N), 60.90 (CH₂CH₂O), 69.60 (CH₂O-ph), 126.90 (central benzene ring CH), 138.06 (central benzene ring Cq), 115.10 and 128.00 (side chain benzene CH), 131.60, 159.50 (side chain benzene Cq), 148.70 (C=O), 154.20 (C=O); MS: 781 (M⁺, 100); Anal. % Calcd. for C₄₂H₄₈N₆O₉: C, 64.60; H, 6.20; N, 10.76, Found: C, 64.48; H, 6.13; N, 10.50.

1-{4-[4-Mono(4-acetylphenoxymethyl)benzyloxy]phenyl}-1-ethanone-N-ethoxycarbonyl hydrazone (5d).

White powder (91% yield); m.p. 259-260°C; IR: ν 3205, 3035, 1705, 1600, 1500, 1230, 822 cm⁻¹; ¹H-NMR (DMSO-d₆): δ 1.22 (t, 3H, CH₃CH₂), 2.15 (s, 3H, CH₃-C=N)), 4.16 (q, 2H, CH₂CH₂), 5.10 (s, 2H, CH₂O), 6.99, 7.87 (d, d, 2H, 2H, AA'BB'), 7.43 (d, central benzene ring CH), 9.99 (s, 1H, N-H); ¹³C-NMR (DMSO-d₆): δ 13.60 (CH₃CH₂), 14.70 (CH₃-C=N), 60.80 (CH₂CH₂O), 67.80 (CH₂O-Ph), 127.60 (central benzene ring CH), 136.30 (central benzene ring Cq), 114.50 and 127.60 (side chain benzene CH), 130.50, 160.10 (side chain benzene Cq), 148.81 (C=O), 154.20 (C=O); MS: 547 (M⁺, 100); Anal. % Calcd. for C₃₀H₃₄N₄O₆: C, 65.92; H, 6.27; N, 10.25, Found: C, 65.99; H, 6.18; N, 10.15.

General procedure for preparation of multiple 1,2,3-selenadiazoles 1a-d

Hydrazone 5a (0.47 mmol) or 5b (0.59 mmol) or 5c (0.9 mmol) or 5d (0.27 mmol) was dissolved in glacial acetic acid (30 mL) with vigorous stirring and gentle heating to 40-45°C. The solution was treated with selenium dioxide powder (8.46 mmol, 7.08 mmol, 8.1 mmol or 0.81 mmol, respectively) and the mixture was kept under gentle heating with vigorous stirring. After 2 min, the color of the mixture becomes red. Monitoring of the reaction by TLC showed that the reaction was complete in two days. The mixture was filtered and the filtrate was poured into ice water and extracted with chloroform (3 × 50 mL). The combined organic layers were washed with saturated sodium hydroxide.
carbonate solution and dried using magnesium sulphate. The solvent was removed under vacuum. The crude product was purified by chromatography using methanol or ethyl acetate as eluents, followed by recrystallization from chloroform/hexane.

4-(4-{2,3,4,5,6-Penta[4-(1,2,3-selenadiazole-4-yl)phenoxy)methyl]benzyloxy}phenyl)-1,2,3-selenadiazole (1a).

Yellow-orange solid (100%); m.p. 77-78°C; IR: v 3096, 2975, 1739, 1611, 1527, 1469, 1220, 989, 841 cm⁻¹; ¹H-NMR (CDCl₃): 5.27 (s, 2H, CH₂O), 7.06, 7.17 (d, d, 2H, 2H, AA'BB'), 9.68 (s, 1H, CHSe); ¹³C-NMR (CDCl₃): 63.62 (C=CH₂O), 128.50 (central benzene ring Cq), 115.36 and 130.20 (side chain benzene CH), 119.20, 152.40 (side chain benzene Cq), 159.61 (C=NC), 137.70 (CHSe); MS: 1500 (M⁺, 100); Anal. % Calcd. For C₆₀H₄₂N₁₂O₆Se₆: C, 48.01; H, 2.82; N, 11.20; Se, 31.57, Found: C, 47.91; H, 2.74; N, 11.11.

4-(4-{2,4,5-Tri[4-(1,2,3-selenadiazole-4-yl)phenoxy)methyl]benzyloxy}phenyl)-1,2,3-selenadiazole (1b).

Yellow-orange solid (92% yield); m.p. 87-88°C; IR: v 3096, 2930, 1725, 1611, 1527, 1469, 1245, 1040, 822 cm⁻¹; ¹H-NMR (CDCl₃): 5.25 (s, 2H, CH₂O), 7.12, 7.22 (d, d, 2H, 2H, AA'BB'), 7.70 (s, benzene central ring CH), 9.71 (s, 1H, CHSe); ¹³C-NMR (CDCl₃): 67.70 (C=CH₂O), 124.00 (central benzene ring CH), 129.80 (central benzene ring Cq), 115.89 and 130.12 (side chain benzene CH), 118.80, 152.60 (side chain benzene Cq), 159.92 (C=NC), 136.40 (CHSe); MS: 1026 (M⁺, 100); Anal. % Calcd. For C₄₂H₃₀N₈O₄Se₄: C, 49.13; H, 2.95; N, 10.91; Se, 30.77, Found: C, 49.10; H, 2.75; N, 10.71.

4-(4-{3,5-Di[4-(1,2,3-selenadiazole-4-yl)phenoxy)methyl]benzyloxy}phenyl)-1,2,3-selenadiazole (1c).

Yellow-orange solid (87% yield); m.p. 100-102°C; IR: v 3096, 2917, 1738, 1610, 1534, 1450, 1239, 1046, 835 cm⁻¹; ¹H-NMR (CDCl₃): 5.15 (s, 2H, CH₂O), 7.11, 7.20 (d, d, 2H, 2H, AA'BB'), 7.49 (s, central benzene ring CH), 9.72 (s, 1H, CHSe); ¹³C-NMR (CDCl₃): 69.69 (C=CH₂O), 126.00 (central benzene ring CH), 129.70 (central benzene ring Cq), 115.99 and 130.03 (side chain benzene CH), 118.80, 149.15 (side chain benzene Cq), 160.15 (C=NC), 137.60 (CHSe); MS: 789 (M⁺, 100); Anal. % Calcd. For C₃₃H₂₄N₆O₃Se₃: C, 50.20; H, 3.06; N, 10.64; Se, 30.01, Found: C, 50.10; H, 3.00; N, 10.59.

1,4-Bis[4-(1,2,3-selenadiazol-4-yl)phenoxy)methyl]benzene (1d).

Yellow-orange solid (60% yield); m.p. 160-162°C; IR: v 3077, 2911, 1732, 1610, 1533, 1456, 1239, 1008, 822 cm⁻¹; ¹H-NMR (DMSO-d₆): 5.15 (s, 2H, CH₂O), 7.12, 7.21 (d, d, 2H, 2H, AA'BB'), 7.47 (d, benzene central ring CH), 9.72 (s, 1H, CHSe); ¹³C-NMR (DMSO-d₆): 70.00 (CH₂O), 127.89 (central benzene ring CH), 129.18 (central benzene ring Cq), 116.10 and 130.00 (side chain benzene CH), 121.00, 152.00 (side chain benzene Cq), 160.00 (C=NC), 138.00 (CHSe); MS: 552 (M⁺, 100); Anal. % Calcd. For C₄₂H₁₈N₄O₂Se₂: C, 52.18; H, 3.28; N, 10.14; Se, 28.60, Found: C, 52.03; H, 3.25; N, 10.20.
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