Synthesis S. Majnooni et al. Paper

Straightforward and Expeditious One-Pot Tandem Synthesis of 3,5-Diaryl-1,2,4-Selenadiazoles from Aryl Nitriles

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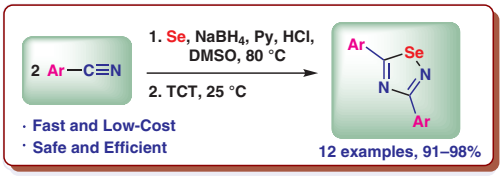

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Abstract A one-pot process for the efficient synthesis of 3,5-diaryl-1,2,4-selenadiazoles from aryl nitriles has been developed. This tandem transformation was performed in excellent yields (91–98%) via in situ selenoamidation and a subsequent oxidative dimerization reaction. This method features relatively mild reaction conditions, operational simplicity, straightforward separation of the products, and the utilization of inexpensive reagents.

Key words selenadiazoles, selenoamides, cyanuric chloride, dimethyl sulfoxide, oxidative dimerization

During the last few decades, interest in organoselenium motifs has increased due to their broad applications in many areas, such as semiconductor preparation,[1] biochemistry investigations,[2] catalyst synthesis[3] and, particularly, their beneficial effects in human health, which makes these compounds robust in biological and medicinal chemistry.[2,4] Among them, selenadiazole scaffolds are a significant class of five-membered heterocyclic compounds, containing selenium and nitrogen heteroatoms, which have revealed unique properties, such as antibacterial,[5] anti-HIV,[6] antimicrobial,[7] anticancer,[8] antifungal[9] and antitumor[10] activities (Figure 1).

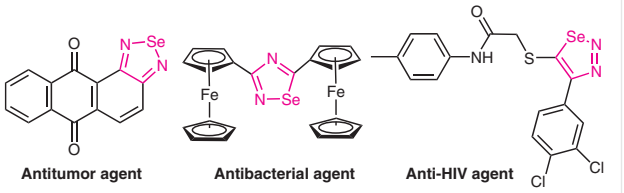
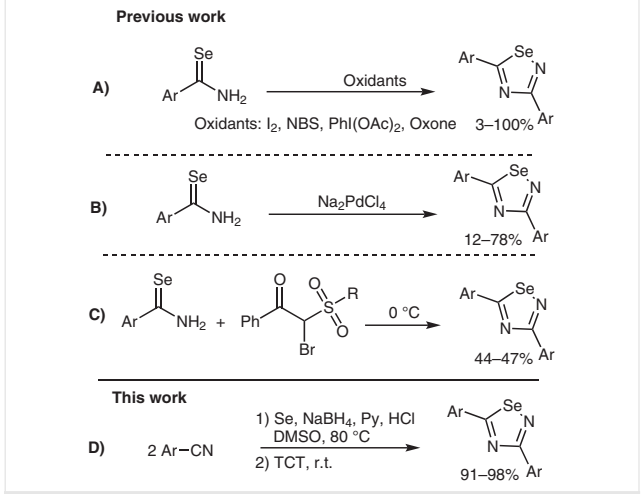


Figure 1 Representative bioactive selenadiazoles

Despite the extensive biological activities and synthetic applications of 1,2,4-selenadiazoles, the practical procedures for the synthesis of these compounds is still limited. The most common routes for the synthesis of 3,5-disubstituted 1,2,4-selenadiazoles are concentrated on the dimerization of primary selenoamides through the utilization of iodine,[11] NBS,[12] $PhI(OAc)_2$[13] and Oxone (Scheme 1, A).[14] These compounds can also be prepared through palladium(II) salts[15] or by utilizing α-bromo ketones (Scheme 1, B and C).[16]



Scheme 1 Synthetic routes to 1,2,4-selenadiazoles

Nonetheless, various limitations are associated with these protocols, such as harsh manipulations, the use of metal catalysts, low yields and prolonged reaction times.[11–16] However, the major drawback of these methods is the effort required for the synthesis of selenoamides as key substrate in a separate step. Usually selenoamides are prepared using highly toxic and unstable reagents, such as H_2Se or NaSeH

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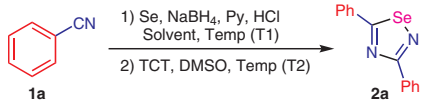
(generated from $NaBH_4$/Se,[17] LiAlHSeH[18]), Se/CO,[19] tris(trimethylsilyl) monoselenophosphate,[20] P_2Se_5/H_2O,[21] Al_2Se_3 in the presence of Et_3N/pyridine/H_2O_2,[22] and P_2Se_5/EtOH/H_2O.[21a] Considering the risks in using these reagents and the purification problems, developing a novel one-pot tandem strategy in order to minimize the purification process and increase chemical efficiency is highly demanded.

Due to the ease of nucleophilic substitution reactions of cyanuric chloride (TCT), it has emerged as a fascinating reagent in various synthetic transformations with broad application in organic synthesis.[23] However, to the best of our knowledge, TCT has not been utilized for the formation of an N–Se bond so far.

In continuation of our efforts to develop new and practical methods for the synthesis of heterocycles,[24] we herein present the first one-pot tandem procedure for the preparation of symmetrical 3,5-diaryl-1,2,4-selenadiazoles. In this protocol, aryl nitrile is applied directly as the starting material in dimethyl sulfoxide (DMSO) for the access to the desired products by a one-pot two-step sequential reaction (Scheme 1, D).

At the outset, the optimal reaction conditions were devised utilizing benzonitrile (**1a**) for the synthesis of 3,5-diphenyl-1,2,4-selenadiazole (**2a**) as a template reaction. We investigated the effects of solvent, temperature and the amount of TCT, as shown in Table 1.

Table 1 Optimization of the Reaction Conditions for the Synthesis of **2a**[a]



1a **2a**

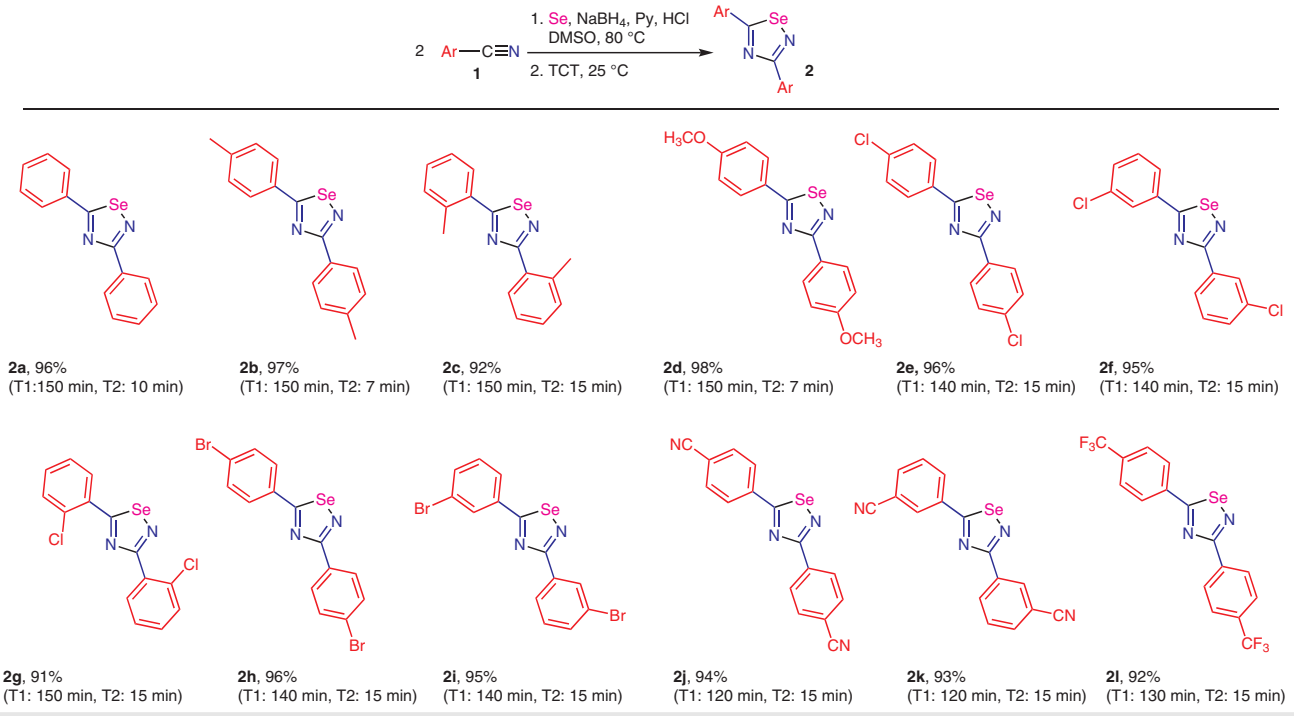
Entry	Solvent	Molar ratio (TCT/DMSO; step 2)	Temp (°C) T1	Temp (°C) T2	Yield (%)[b]
1	EtOH	0.34:1	80	r.t.	85
2	MeOH	0.34:1	80	r.t.	64
3	toluene	0.34:1	80	r.t.	trace
4	CH_3CN	0.34:1	80	r.t.	trace
5	DMSO	0.34:0	80	r.t.	96
6	DMSO	0.34:0	100	r.t.	96
7	DMSO	0.34:0	60	r.t.	83
8	DMSO	0.34:0	80	80	96
9	DMSO	0.2:0	80	r.t.	65
10	DMSO	0.4:0	80	r.t.	96

[a] Reaction conditions: (1) Se (8 mmol), $NaBH_4$ (8.8 mmol), solvent (4 mL), N_2 atmosphere, 20 min; then, addition of **1a** (2 mmol), pyridine (1.29 mL, 16 mmol), HCl (2 N, 4 mL), 2 h; (2) TCT/DMSO, r.t., 10 min.
[b] Isolated yields.

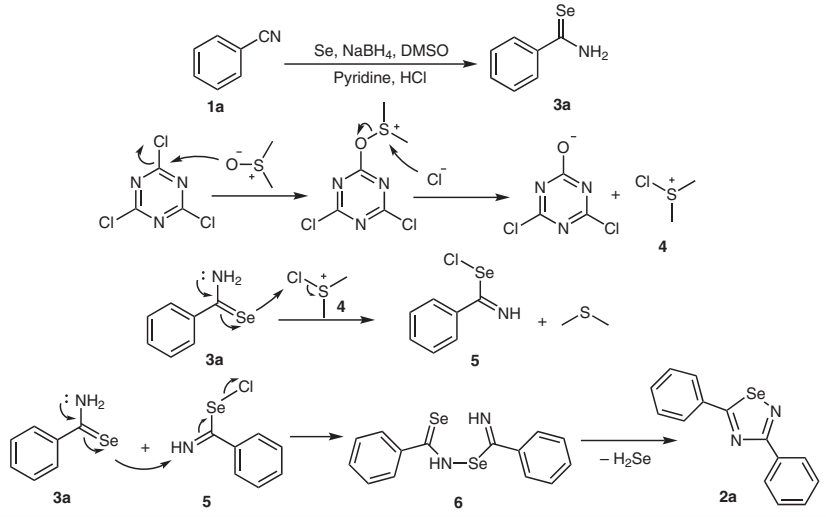
At first, the reaction of benzonitrile (**1a**) with Se/$NaBH_4$ was carried out in EtOH under N_2 atmosphere in the presence of pyridine and dilute hydrochloric acid for 150 minutes. A mixture of TCT in DMSO was subsequently added to the reaction medium and the mixture was stirred at room temperature for about 10 minutes, affording the corresponding 1,2,4-selenadiazole **2a** in 85% yield (Table 1, entry 1). According to numerous literature reports of the suitable activity of $NaBH_4$ in DMSO,[25] we performed a practical modification of this method by replacing EtOH by DMSO which performs the dual function of solvent and reagent along with TCT. It was found that the efficiency of the reaction was improved significantly and the best yield (96%) was obtained (entry 5). Therefore, DMSO was chosen as the optimal solvent. The employment of other solvents was not satisfactory in this reaction (entries 2–4). Next, we investigated the effect of temperature on the reaction progress and found that the best yield was obtained when the conversion of benzonitrile into the corresponding benzoselenamide was performed at 80 °C, with subsequent TCT addition at ambient temperature. Increasing the temperature did not effect improvement of the yield (entries 6 and 8). On the other hand, the TCT amount was also evaluated under the reaction conditions (entries 5, 9 and 10). As illustrated in Table 1, 0.34 mmol of TCT was sufficient to give an excellent yield of the desired product (entry 5).

Under the optimized conditions, the substrate scope and limitations of this protocol were examined utilizing diverse aromatic and aliphatic nitriles (Scheme 2). The results revealed that aromatic nitriles bearing an electron-donating group, such as methyl (**2b** and **2c**) and methoxy (**2d**), as well as ones with an electron-withdrawing group [e.g., Cl (**2e–g**), Br (**2h** and **2i**), CN (**2j** and **2k**), CF_3 (**2l**)] are compatible with this method and the desired products were obtained in excellent yields (91–98%). It is noteworthy to mention that nitriles possessing a group at the *ortho*-position provided the products in slightly lower yields (91–92%) due to steric hindrance (entries **2c** and **2g**). Furthermore, efforts to extend this methodology to aliphatic nitriles were not successful and an unidentified mixture was obtained.

Inspired by our experimental results and previous reports,[24] a possible mechanism for this transformation is depicted in Scheme 3. Initially, benzoselenoamide (**3a**) is easily prepared by the reaction of benzonitrile (**1a**) with selenium powder and $NaBH_4$ in DMSO in the presence of pyridine and dilute hydrochloric acid. Meanwhile, chlorodimethylsulfonium ion (**4**) as an active intermediate is prepared via the reaction of TCT and DMSO. Subsequently, Se–Cl bond formation of benzoselenoamide in the presence of the reactive intermediate leads to the formation of **5** which, after a dimerization reaction with another benzoselenoamide, provides *N*-substituted benzoselenoamide **6**. Eventually, intramolecular cyclization of **6** and then aromatization via release of H_2Se affords the corresponding 3,5-diphenyl-1,2,4-selenadiazole (**2a**).



Scheme 2 Substrate scope of the reaction



Scheme 3 Plausible route

In summary, we have developed a novel tandem one-pot multistep protocol for the efficient conversion of benzonitrile derivatives into the corresponding 1,2,4-selenadiazoles. The main feature of this transformation is the in situ formation of selenoamide/oxidative dimerization reaction, reducing the number of purification processes, in excellent yields.

All chemical reagents and solvents used in this work were obtained from Merck and Sigma-Aldrich. The progress of reactions was monitored by TLC (silica gel 60 F254). Melting points were determined with a Stuart Scientific SMP-2 apparatus. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance 400 MHz Fourier transform spectrometer. Coupling constants are reported in hertz (Hz). Fourier transform infrared (FTIR) spectra were recorded on a Nicolet Impact 400D spectrophotometer (KBr pellets) in the range of 400–4000 cm^{-1}.

3,5-Disubstituted 1,2,4-Selenadiazoles 2a–l; General Procedure

To a solution of selenium powder (8 mmol) in DMSO (4 mL) was added $NaBH_4$ (8.8 mmol) under nitrogen atmosphere. The reaction mixture was stirred at room temperature for about 20 min and then the aryl nitrile **1** (2 mmol) and pyridine (1.29 mL, 16 mmol) were added. Subsequently, the mixture was heated at 80 °C, while HCl (2 N, 4 mL) was added dropwise over 30 min, and then stirring was continued. The progress of the reaction was monitored by TLC. When the reaction was completed, it was cooled to ambient temperature and TCT (0.34 mmol) was added; the mixture was stirred at room temperature. After completion of the reaction, as monitored by TLC (petroleum ether/ethyl acetate, 8:2), the mixture was washed with water (4.0 mL) and extracted with chloroform (2 × 5 mL). The extract was dried over anhydrous Na_2SO_4, the solvent was removed under vacuum, MeOH/water (1:1, 5 mL) was added, and the solid phase was collected by filtration and dried under vacuum to give the pure product.

3,5-Diphenyl-1,2,4-selenadiazole (2a)

Yield: 0.274 g (96%); white crystals; mp 85–87 °C (Lit.[13] 85 °C).

IR (KBr): 3099, 3049, 2955, 2925, 2857, 1631, 1587, 1514, 1482, 1443, 1319, 1277, 1091, 963, 757, 679 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 8.34–8.32 (m, 2 H), 7.95–7.92 (m, 2 H), 7.49–7.41 (m, 6 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 193.88, 174.46, 134.39, 134.15, 132.14, 130.11, 129.37, 128.81, 128.70, 128.26.

3,5-Bis(4-methylphenyl)-1,2,4-selenadiazole (2b)

Yield: 0.304 g (97%); white crystals; mp 113–115 °C (Lit.[13] 122 °C).

IR (KBr): 3011, 2915, 2855, 1606, 1522, 1486, 1402, 1315, 1268, 1091, 1019, 961, 815 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 8.31 (d, *J* = 8.4 Hz, 2 H), 7.91 (d, *J* = 8.4 Hz, 2 H), 7.33–7.30 (dd, *J* = 8.4, 2.4 Hz, 4 H), 2.44 (s, 6 H).

3,5-Bis(2-methylphenyl)-1,2,4-selenadiazole (2c)

Yield: 0.288 g (92%); white crystals; mp 55–57 °C.

IR (KBr): 3047, 2923, 1591, 1518, 1475, 1440, 1415, 1366, 1328, 1274, 1068, 759, 680 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 8.00 (d, *J* = 8 Hz, 1 H), 7.97 (d, *J* = 8 Hz, 1 H), 7.31–7.22 (m, 6 H), 2.60 (s, 6 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 188.13, 173.83, 138.10, 137.25, 132.45, 131.84, 131.25, 131.04, 130.93, 130.01, 129.87, 129.66, 129.37, 126.02, 22.88, 22.14.

Anal. Calcd for $C_{16}H_{14}N_2Se$: C, 61.35; H, 4.50; N, 8.94. Found: C, 61.22; H, 4.63; N, 8.85.

3,5-Bis(4-methoxyphenyl)-1,2,4-selenadiazole (2d)

Yield: 0.338 g (98%); white crystals; mp 133–135 °C (Lit.[13] 137 °C).

IR (KBr): 3056, 3004, 2936, 2833, 1605, 1581, 1487, 1414, 1305, 1275, 1247, 1166, 1089, 1030, 834 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 8.36 (d, *J* = 8.8 Hz, 2 H), 7.96 (d, *J* = 8.8 Hz, 2 H), 7.01 (d, *J* = 8.8 Hz, 4 H), 3.92 (s, 3 H), 3.90 (s, 3 H).

3,5-Bis(4-chlorophenyl)-1,2,4-selenadiazole (2e)

Yield: 0.340 g (96%); white crystals; mp 159–161 °C (Lit.[13] 167 °C).

IR (KBr): 3047, 2921, 1588, 1509, 1473, 1396, 1310, 1281, 1230, 1090, 1012, 965, 832, 732 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 8.24 (d, *J* = 8.8 Hz, 2 H), 7.84 (d, *J* = 8.4 Hz, 2 H), 7.41–7.36 (m, 4 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.69, 173.38, 138.39, 136.29, 132.65, 132.46, 130.12, 129.68, 129.40, 128.96.

3,5-Bis(3-chlorophenyl)-1,2,4-selenadiazole (2f)

Yield: 0.336 g (95%); white crystals; mp 123–125 °C.

IR (KBr): 3070, 2921, 1568, 1504, 1467, 1422, 1292, 1227, 1073, 979, 893, 786, 723 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 8.42 (s, 1 H), 8.29 (d, *J* = 7.6 Hz, 1 H), 8.05 (s, 1 H), 7.86 (d, *J* = 7.6 Hz, 1 H), 7.56 (d, *J* = 8 Hz, 1 H), 7.50–7.43 (m, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.68, 173.06, 135.68, 135.54, 135.44, 134.75, 132.13, 130.68, 130.26, 130.02, 128.97, 127.76, 126.85, 126.65.

Anal. Calcd for $C_{14}H_8Cl_2N_2Se$: C, 47.49; H, 2.28; N, 7.91. Found: C, 47.63; H, 2.34; N, 7.84.

3,5-Bis(2-chlorophenyl)-1,2,4-selenadiazole (2g)

Yield: 0.322 g (91%); white crystals; mp 87–89 °C.

IR (KBr): 3057, 2951, 2918, 2849, 1588, 1490, 1448, 1308, 1267, 1109, 1051, 960, 781, 751 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 8.73–8.71 (m, 1 H), 8.01–7.99 (m, 1 H), 7.65–7.63 (m, 1 H), 7.56–7.54 (m, 1 H), 7.52–7.49 (m, 2 H), 7.43–7.41 (m, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 183.67, 173.19, 133.62, 133.32, 132.39, 132.24, 131.36, 130.75, 130.54, 130.36, 129.93, 128.78, 127.64, 126.77.

Anal. Calcd for $C_{14}H_8Cl_2N_2Se$: C, 47.49; H, 2.28; N, 7.91. Found: C, 47.36; H, 2.36; N, 7.80.

3,5-Bis(4-bromophenyl)-1,2,4-selenadiazole (2h)

Yield: 0.425 g (96%); white crystals; mp 161–163 °C (Lit.[13] 162 °C).

IR (KBr): 3043, 2919, 2860, 1583, 1506, 1471, 1392, 1309, 1282, 1231, 1068, 830 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 8.18 (d, *J* = 8.4 Hz, 2 H), 7.79 (d, *J* = 8.4 Hz, 2 H), 7.59–7.54 (m, 4 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.87, 173.50, 133.06, 132.86, 132.66, 131.92, 130.36, 129.55, 126.86, 124.77.

3,5-Bis(3-bromophenyl)-1,2,4-selenadiazole (2i)

Yield: 0.421 g (95%); white crystals; mp 133–135 °C.

IR (KBr): 3065, 2921, 2860, 1563, 1500, 1466, 1416, 1293, 1228, 1068, 977, 790, 725, 678 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 8.57 (t, *J* = 2 Hz, 1 H), 8.35–8.33 (dt, *J* = 7.6, 1.2 Hz, 1 H), 8.20 (t, *J* = 2 Hz, 1 H), 7.92–7.89 (dt, *J* = 8, 1.2 Hz, 1 H), 7.73–7.70 (m, 1 H), 7.64–7.61 (m, 1 H), 7.43–7.37 (m, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.57, 172.91, 135.90, 135.64, 135.06, 133.18, 131.86, 130.90, 130.63, 130.28, 127.31, 127.10, 123.51, 122.86.

Anal. Calcd for $C_{14}H_8Br_2N_2Se$: C, 37.96; H, 1.82; N, 6.32. Found: C, 37.83; H, 1.90; N, 6.25.

4,4′-(1,2,4-Selenadiazole-3,5-diyl)dibenzonitrile (2j)

Yield: 0.315 g (94%); white crystals; mp 194–196 °C.

IR (KBr): 3066, 2956, 2923, 2857, 2227, 1730, 1605, 1478, 1402, 1317, 1185, 1081, 967, 841 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 8.44 (d, *J* = 8.4 Hz, 2 H), 8.05 (d, *J* = 8.4 Hz, 2 H), 7.78–7.72 (m, 4 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.97, 172.10, 137.50, 137.21, 133.27, 132.67, 129.34, 128.69, 124.49, 118.62, 117.93, 113.76.

Anal. Calcd for $C_{16}H_8N_4Se$: C, 57.33; H, 2.41; N, 16.71. Found: C, 57.45; H, 2.49; N, 16.62.

3,3′-(1,2,4-Selenadiazole-3,5-diyl)dibenzonitrile (2k)

Yield: 0.312 g (93%); white crystals; mp 179–181 °C.

IR (KBr): 3071, 2955, 2921, 2855, 2227, 1581, 1496, 1476, 1405, 1320, 1167, 1085, 997, 801 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 8.64 (t, *J* = 2 Hz, 1 H), 8.57–8.54 (dt, *J* = 8, 1.6 Hz, 1 H), 8.26 (t, *J* = 1.6 Hz, 1 H), 8.15–8.13 (dt, *J* = 8, 1.6 Hz, 1 H), 7.81–7.78 (dt, *J* = 7.6, 1.2 Hz, 1 H), 7.71–7.69 (dt, *J* = 7.6, 1.6 Hz, 1 H), 7.63–7.54 (m, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.26, 172.32, 135.24, 134.89, 134.86, 133.52, 132.79, 132.59, 132.26, 131.37, 130.49, 129.72, 118.43, 117.54, 114.12, 113.17.

Anal. Calcd for $C_{16}H_8N_4Se$: C, 57.33; H, 2.41; N, 16.71. Found: C, 57.20; H, 2.49; N, 16.80.

3,5-Bis(4-(trifluoromethyl)phenyl)-1,2,4-selenadiazole (2l)

Yield: 0.388 g (92%); white crystals; mp 134–136 °C.

IR (KBr): 2930, 1617, 1521, 1491, 1408, 1323, 1240, 1162, 1112, 1062, 1017, 836 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 8.43 (d, *J* = 8 Hz, 2 H), 8.04 (d, *J* = 8 Hz, 2 H), 7.72–7.67 (m, 4 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.90, 173.27, 137.03, 136.82, 133.75 (q, *J* = 33 Hz), 131.91 (q, *J* = 32 Hz), 129.12, 128.53, 126.49 (q, *J* = 4 Hz), 125.75 (q, *J* = 4 Hz), 124.95, 122.67.

Anal. Calcd for $C_{16}H_8F_6N_2Se$: C, 45.62; H, 1.91; N, 6.65. Found: C, 45.48; H, 1.99; N, 6.73.

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Supporting Information

Supporting information for this article is available online at https://doi.org/10.1055/s-0039-1690126.

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