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KI mediated one-pot cascade reaction for synthesis of 1,3,4-selenadiazoles†

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An efficient catalytic system consisting of KI and $K_2S_2O_8$ for a one-pot pseudo three-component cascade reaction in the preparation of a diverse array of 1,3,4-selenadiazole derivatives from easily accessible precursors aldehydes, hydrazine and elemental selenium is demonstrated in this paper. Here, KI is used as the surrogate of iodine and $K_2S_2O_8$ as the oxidant. The key advantages of this protocol include an easy reaction set up, operational simplicity, high functional group tolerance and utilisation of low toxicity chemicals. Further, a radical quenching reaction was also performed to confirm the mechanistic pathway.

Introduction

Berzelius, a Swedish chemist, was unprecedentedly able to separate selenium, one of the non-metals, from the chalcogenide family, in 1817. It exists in the soil either in its inorganic forms, such as elemental Se (Se^0), selenite (SeO_3^{2-}), selenide (Se^{2-}) or selenate (SeO_4^{2-}) and is also obtained in its organic forms, such as selenomethionine and selenocysteine.[1–3] Elemental selenium has been known to play a pivotal role in various industries, some of which include medicinal, catalysis, metallurgical, chemical and electrical industries.[4–6] In addition, it is a critical component found in electrical devices such as fuel cells, solar cells, UV light detectors, and radiation and laser printers. Due to some of the advantages associated with selenium, such as non-toxicity, low melting point, easy availability, simple composition, and direct fundamental band gap, it has gained wide attention.[7,8] Further, the biological effects of Se have also been reported. Some of those are that it improves the antiviral activity in animals and helps to stimulate organogenesis and, thus, the growth of root systems.[9,10]

The organoselenium compounds have been found to exhibit various properties such as antitumor, antiviral, antidepressant, anti-inflammatory, anticonvulsant and antioxidant.[11] Therefore, there have been continuous efforts to develop strategies for the synthesis of organic selenium compounds, some of which include selenadiazole, selenazoles, diselenides, selenophenes, selenoamides, selenium-incorporated heterocycles and many more.[12–15] 1,3,4-Selenadiazoles and their derivatives fall under important selenium-containing compounds as they find wide applications in agricultural, pharmaceutical and material chemistry. Due to the "N–C–Se" linkage, 1,3,4-selenadiazoles can form a chelate with metal ions *in vivo*, can work as an active centre and show good tissue permeability. 1,3,4-Selenadiazoles have been reported to have biological activities such as analgesic, antibacterial, anticonvulsant, antitumor and have also been incorporated in anti-inflammatory medicines, fungicides and pesticides.[16–18] 1,3,4-Selenadiazoles have also been used as corrosion inhibitors, thermotropic liquid crystals, dyes and metal ion complex-forming reagents.[19–24] Glutaminase inhibitor having 1,3,4-selenadiazole has been shown to have antitumor activities.[25]

A number of synthetic routes have been demonstrated so far for the synthesis of 1,3,4-selenathiazoles. After going through the literature few methods for the preparation of these bioactive derivatives have been found which includes reaction of dimethylformamide azine with hydrogen selenide,[26] reaction of isoselenocyanates with selenosemicarbazides.[27] Furthermore, selenathiazoles have been synthesized by ring-closure reaction of selenobenzamides with hydrazine hydrate,[28] reaction of carboxylic acid with selenosemicarbazide and phosphoryl chloride[29] treatment of 1,2-diacetylhydrazine with phosphorus pentaselenide.[30] In 2021, S. K. Bowroju and co-workers developed a simple one-pot three-component cascade method for the synthesis of 1,3,4-selenathiazoles derivatives.[31] To best of our knowledge, only one articles reported in literature regarding this one-pot three-component cascade reaction. So, there has been plenty of opportunity to investigate over this methodology. But some of these procedures have numerous drawbacks such as low yield, high cost of starting materials as well as catalyst, harsh reaction condition, longer reaction time. So, there is ample scope to improve the synthetic method of these biologically active molecules.

Potassium iodide (KI) is a readily available, cheaper, non-toxic reagent and environmentally safe chemical as it can use as a medication for hyperthyroidism. So, use of KI is safe for human health and our mother earth also. KI has been reported

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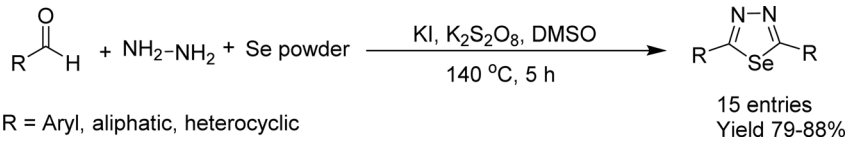


Scheme 1 One-pot three-component cascade reaction for synthesis of 1,3,4-selenathiazoles.

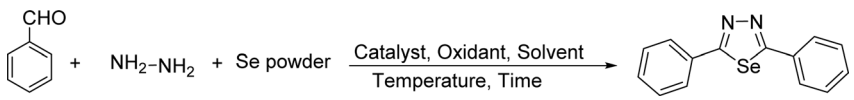
as an excellent catalyst in many organic reactions[32] as well as oxidant, $K_2S_2O_8$ has also been explored in fewer reactions.[33]

So, keeping this in mind, herein we demonstrate a synthetic protocol through one-pot three-component cascade reaction (Scheme 1) for the design of 1,3,4-selenathiazoles from readily accessible aldehydes, selenium powder and hydrazine using catalytic system KI and $K_2S_2O_8$.

Result and discussion

For the optimisation of reaction condition, benzaldehyde (2 mmol), hydrazine (1 mmol), and selenium powder (1 mmol) were taken as a model reaction and the reactions were scanned under various circumstances. To obtain the intended result, it was found that the solvent, reaction temperature, catalyst quantity, amount of oxidant and reaction duration had a significant impact. An optimization study was started using various oxidants maintaining a constant 70 mg of catalysts under reflux conditions for 9 h (Table 1, entries 1–5). It was discovered that 2 equivalent of oxone, *m*-chloroperoxybenzoic acid (MCPBA), hydrogen peroxide produced low yields in dimethyl sulfoxide (DMSO) (Table 1, entries 1, 2, 3). Trace amount of the product was obtained when the reaction was occurred under neat conditions for 9 h at 140 °C in presence of 2 equivalent of potassium persulphate catalyst (Table 1, entry 4). The yield of the product was found to increase unexpectedly to 87% when we changed the solvent to DMSO at 140 °C for 9 h (Table 1, entry 5). When the reaction was carried out under various solvents such as water, methanol, ethanol, *N*,*N*-dimethylformamide (DMF) under same conditions, it was observed that the yield of the product decreased (Table 1, entries 6–9). So, solvent has a great influence in this reaction. Simultaneously, we also optimized the amount of oxidant and it was found that 1 equivalent oxidant was enough to give good yield of the products (Table 1, entry 11). Following that, we adjusted the catalyst amount and discovered that 87% of product was obtained in presence of only 30 mg of catalyst (Table 1, entry 11). We also observed that, in the absence of catalyst, no product was

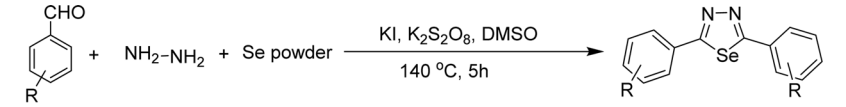
Table 1 Optimization of reaction conditions[a]

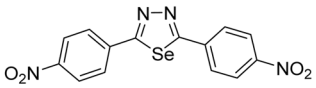
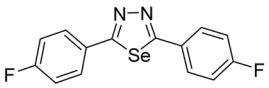
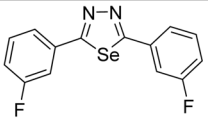
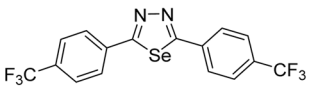
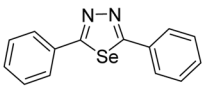
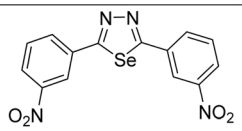
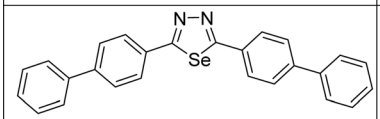
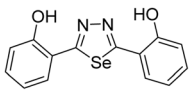
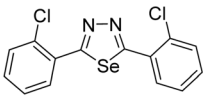
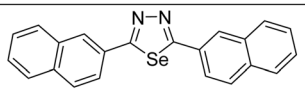
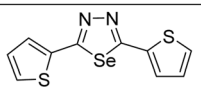
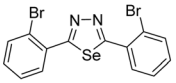
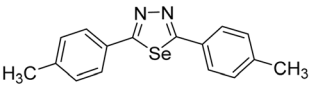
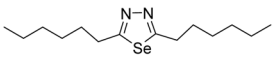
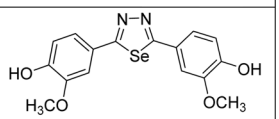


Entry	Solvent	Catalyst loading (mg)	Oxidant (equivalent)	Temperature (°C)	Time (h)	Yield[b] (%)
1	DMSO	70	MCPBA (2)	Reflux	9	48
2	DMSO	70	Hydrogen peroxide (2)	Reflux	9	49
3	DMSO	70	Oxone (2)	Reflux	9	50
4	Neat	70	$K_2S_2O_8$ (2)	140	9	Trace
5	DMSO	70	$K_2S_2O_8$ (2)	140	9	87
6	Water	70	$K_2S_2O_8$ (2)	140	9	60
7	Methanol	70	$K_2S_2O_8$ (2)	140	9	62
8	Ethanol	70	$K_2S_2O_8$ (2)	140	9	64
9	DMF	70	$K_2S_2O_8$ (2)	140	9	63
10	DMSO	50	$K_2S_2O_8$ (1)	140	9	87
11	DMSO	30	$K_2S_2O_8$ (1)	140	9	86
12	DMSO	30	$K_2S_2O_8$ (0.5)	140	9	73
13	DMSO	20	$K_2S_2O_8$ (1)	140	9	71
14	DMSO	Nil	$K_2S_2O_8$ (2)	140	9	—
15	DMSO	30	$K_2S_2O_8$ (1)	90	9	78
16	DMSO	30	$K_2S_2O_8$ (1)	60	9	61
17	DMSO	30	$K_2S_2O_8$ (1)	Rt	9	Trace
18[c]	**DMSO**	**30**	**$K_2S_2O_8$ (1)**	**140**	**5**	**88**
19	DMSO	30	$K_2S_2O_8$ (1)	140	3	72
20	DMSO	I_2 (40 mg)	Nil	140	5	25%

[a] Reaction of benzaldehyde (2 mmol), hydrazine (1 mmol) selenium powder (1 mmol). [b] Isolated yield after column chromatography. [c] The bold sign signifies the best reaction conditions.

Table 2 Synthesis of functionalized 2,5-diphenyl-1,3,4-selenadiazole derivatives[a] from functionalized aldehydes



 Entry 1: 2a, 88%	 Entry 2: 2b, 87%	 Entry 3: 2c, 85%
 Entry 4: 2d, 86%	 Entry 5: 2e, 84%	 Entry 6: 2f, 87%
 Entry 7: 2g, 86%	 Entry 8: 2h, 80%	 Entry 9: 2i, 81%
 Entry 10: 2j, 87%	 Entry 11: 2k, 83%	 Entry 12: 2l, 82%
 Entry 13: 2m, 79%	 Entry 14: 2n, trace	 Entry 15: 2o, trace

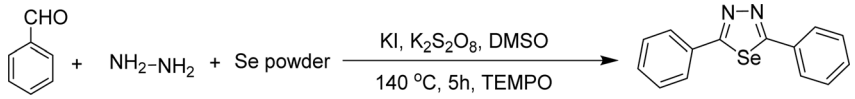
[a] Reaction of aldehydes (2 mmol), hydrazine (1 mmol), selenium powder (1 mmol), potassium persulphate (1 equiv.) and KI (30 mg) at 140 °C under in DMSO for 5 h. Isolated yield after column chromatography.

generated (Table 1, entry 14). Thus, it may be inferred from this finding that KI plays the catalytic role required for this process. Subsequently, we optimised the reaction time and temperature, and it was observed that only 5 h in 140 °C was sufficient to provide the maximum yield of the product (Table 1, entry 18). If we further reduced the time, yield of the product was also decreased (Table 1, entry 19). We found that only trace amount of the product was obtained at room temperature (Table 1, entry 17). At last we tried the reaction with elemental iodine (40 mg) in absence of any oxidising agent in DMSO but only 25% yield of product was obtained (Table 1, entry 20).

After getting the optimised reaction condition, diverse array of functionalized aldehydes were treated with hydrazine and Se powder in the presence of potassium iodide and potassium persulphate in DMSO as part of a circumstantial study carried out in our scheme (Table 2). It was observed that when electron withdrawing groups were present in the benzaldehyde, specially $-NO_2$, –F, $-CF_3$, –Cl, then yield of the product was high (Table 2, entries 1, 2, 4, 9). But when electron donating group was attached with the benzaldehyde especially –OH, $-CH_3$ then the yield of the product was low (Table 2, entries 8, 13). Nitrobenzaldehyde gave highest yield of the product **2a** was 88% (Table 2, entry 1). Also, in case of 4-triflurobenzaldehyde, the yield of the product **2d** was 86% (Table 2, entry 4). We also incorporated heterocyclic aldehydes like thiophene-2-aldehyde which produce the target molecule with good yield (Table 2, entry 11). Moreover, biphenyl aldehyde and 2-napthyl aldehyde also gave excellent yield of the product **2g** and **2j** (Table 2, entries 7, 10). But 2-chlorobenzaldehyde and 2-bromobenzaldehyde gave quite low yield which may be due to steric hindrance presence in the substrate molecule (Table 2, entries 9, 12). It was also observed that heptaldehyde and vanilne give trace amount of yield (Table 2, entries 14, 15).

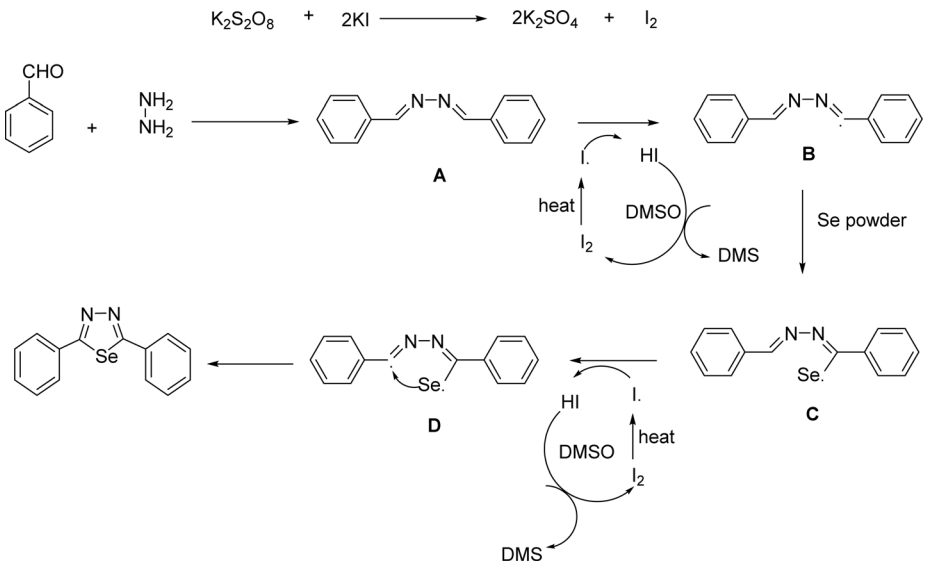
After a careful literature study and to validate the proposed radical pathway for this organic transformation (G. Brahmachari, *et al.*, *J. Org. Chem.*, 2021),[36] a sets of control experiments (Table 3) was performed under the optimized reaction condition in presence of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) as the radical-trapping reagents. The formation of the desired product was restricted in the presence of radical-

Table 3 Result of control experiment[a]



Entry	Radical-trapping reagent	Amount (equivalent)	Temperature (°C)	Time (h)	Yield[b] (%)
1	TEMPO	0.25	140	5	44
2	TEMPO	0.50	140	5	18
3	TEMPO	1.0	140	5	0

[a] Reaction of aldehydes (2 mmol), hydrazine (1 mmol), selenium powder (1 mmol) in DMSO under the optimized reaction conditions in the presence of radical-trapping reagents TEMPO. [b] Isolated yield.



Scheme 2 Possible mechanism for 2,5-diphenyl-1,3,4-selenadiazole derivatives.

trapping reagents TEMPO (Table 3, entry 3). It was found that when 0.25 equiv. TEMPO was used, the yield of the product was 44% (Table 3, entry 1) but with 0.5 equiv. TEMPO under same reaction condition yield of the product was very less (Table 3, entry 2). Finally, when 1 equiv. TEMPO was used under same condition the reaction was quenched giving no products. From this observation it can be concluded that the proposed reaction protocol proceed through a radical mechanistic pathway.

Mechanism for the synthesis of 2,5-diphenyl-1,3,4-selenadiazole derivative

Based on the above preliminary findings and literature survey, a probable mechanism was drawn in Scheme 2. During the reaction of potassium persulphate ($K_2S_2O_8$) and potassium iodide (KI), iodine molecule may be generated *in situ*. Iodine radicals may be formed when iodine molecules gets heated in DMSO. The *in situ* generated iodine radicals thus abstracts a proton from A to generate the intermediate B and itself gets converted into HI during the process. HI then combines with DMSO[34] to regenerate iodine and the cycle continues (W. Li, *et al.*, *Front. Chem.*, 2020; S. K. Bowroju, *et al.*, *RSC Adv.*, 2021).[31,34] After that, the radical intermediate B may combine with elemental selenium (Y. Murata, *et al.*, *RSC Adv.*, 2022; P. Sun, *et al.*, *J. Org. Chem.*, 2017).[35] By the reaction with selenium free-radical, intermediate C may be produced. Once again, a proton is taken from C by iodine radicals to produce the radical intermediates D and HI. Eventually, cyclization of intermediate D yields the desired product. Although we have drawn here a probable mechanism here but to verify the proposed radical pathway, we have performed control experiment using radical quencher (Table 3) (G. Brahmachari, *et al.*, *J. Org. Chem.*, 2021).[36] From the results obtained from control experiment, it can be concluded that the reaction surely follow the radical mechanistic pathway.

Conclusion

This work represents the preparation of a wide variety of 1,3,4-selenadiazole derivatives through one-pot three-component cascade reaction from readily available and non-toxic starting materials such as elemental selenium, hydrazine, and

aldehydes by the use of an effective catalytic system KI and $K_2S_2O_8$. Use of low toxic chemicals, high functional group tolerance with good yield, ease of reaction setup, and operational simplicity are the foremost superiority of the reported protocols.

Experimental section

General procedure for the synthesis of functionalized 2,5-diphenyl-1,3,4-selenadiazole

A mixture of benzaldehyde (2 mmol), hydrazine (1 mmol) and selenium powder (1 mmol), potassium persulphate (1 equiv.) were stirred at 140 °C for 5 h in DMSO in open air in a round-bottom flask (50 ml) using a magnetic stirring bar. The reaction progress was monitored using TLC with a mixture of ethyl acetate and *n*-hexane as the eluent system. After completion of the reaction, the mixture was quenched to room temperature and ethyl acetate (5 ml) was added to the reaction mixture and the catalyst was separated by simple filtration and was extracted with ethyl acetate twice (2 × 20 ml). Combined extracts were washed with distilled water, dried over anhydrous Na_2SO_4 and concentrated. The crude product was purified by passing through a column packed with silica gel (eluent: ethyl acetate–petroleum ether). The products obtained were known compounds and were identified by NMR spectroscopy.

Spectroscopy data of functionalized 2,5-diphenyl-1,3,4-selenadiazole

2,5-Bis(4-nitrophenyl)-1,3,4-selenadiazole. Pale yellow solid; mp 212–214 °C.

^{1}H NMR (400 MHz, DMSO) δ (ppm): 8.15 (d, J = 8.52 Hz, 1H), 8.31 (d, J = 8.72 Hz, 1H).

^{13}C NMR (100 MHz, DMSO) δ (ppm): 124.17, 131.15, 136.86, 150.46, 166.31.

2,5-Di(naphthalen-2-yl)-1,3,4-selenadiazole. White solid; mp 135–138 °C.

^{1}H NMR (400 MHz, DMSO) δ (ppm): 7.39–7.45 (m, 2H), 7.57–7.65 (m, 3H), 7.77–8.21 (m, 1H), 8.62 (s, 1H).

^{13}C NMR (100 MHz, DMSO) δ (ppm): 125.63, 127.25, 128.10, 128.61, 128.76, 129.73, 130.99, 132.60, 135.38, 167.93.

2,5-Diphenyl-1,3,4-selenadiazol. White solid; mp 116–118 °C.

^{1}H NMR (400 MHz, DMSO) δ (ppm): 7.30 (t, J = 7.16 Hz, 2H), 7.50 (t, J = 7.56 Hz, 1H), 8.13 (d, J = 7.92 Hz, 2H).

^{13}C NMR (100 MHz, DMSO) δ (ppm): 129.03, 129.72, 131.23, 133.32, 167.79.

2,5-Bis(4-(trifluoromethyl)phenyl)-1,3,4-selenadiazole. White solid; mp 154–156 °C.

^{1}H NMR (400 MHz, DMSO) δ (ppm): 7.88 (d, J = 8.32 Hz, 2H), 8.12 (t, J = 8.16 Hz, 2H).

2,5-Bis(3-fluorophenyl)-1,3,4-selenadiazole. White solid; mp 142–145 °C.

^{1}H NMR (400 MHz, DMSO) δ (ppm): 7.46–7.50 (m, 2H), 7.65 (t, J = 7.84 Hz, 1H), 7.79 (d, J = 7.6 Hz, 1H).

^{13}C NMR (100 MHz, DMSO) δ (ppm): 116.08, 116.30, 120.22, 120.43, 125.89, 131.22, 131.30, 133.65, 133.72, 161.20, 163.63, 166.65.

2,5-Bis(3-nitrophenyl)-1,3,4-selenadiazole. Yellow solid; mp 201–203 °C.

^{1}H NMR (400 MHz, DMSO) δ (ppm): 7.80 (t, J = 7.96 Hz, 1H), 8.34 (d, J = 7.72 Hz, 1H), 8.44–8.47 (m, 1H), 8.60 (t, J = 1.64 Hz, 1H).

^{13}C NMR (100 MHz, DMSO) δ (ppm): 124.13, 127.79, 130.99, 132.90, 135.82, 148.32, 165.97.

2,5-Bis(4-fluorophenyl)-1,3,4-selenadiazole. White solid; mp 151–154 °C.

^{1}H NMR (400 MHz, DMSO) δ (ppm): 7.33 (q, J = 8.76 Hz, 2H), 7.98–8.21 (m, 2H).

^{13}C NMR (100 MHz, DMSO) δ (ppm): 115.98, 116.20, 127.84, 132.52, 132.61, 164.12, 166.61, 166.83.

2,5-Di([1,1′-biphenyl]-4-y1)-1,3,4-selenadiazole. Brown solid; mp 145–147 °C.

^{1}H NMR (400 MHz, DMSO) δ (ppm): 7.36 (t, J = 7.28 Hz, 1H), 7.42–7.73 (m, 2H), 8.02 (d, J = 8.28 Hz, 1H).

^{13}C NMR (100 MHz, DMSO) δ (ppm): 127.20, 127.34, 128.72, 129.51, 129.95, 130.47, 139.38, 144.78, 167.77.

2,5-Di(thiophen-3-yl)-1,3,4-selenadiazole. Off white solid; mp 232–237 °C.

^{1}H NMR (400 MHz, DMSO) δ (ppm): 7.17 (t, J = 3.8 Hz, 1H), 7.71 (t, J = 2.48 Hz, 1H), 8.84 (d, J = 4.72 Hz, 1H).

^{13}C NMR (100 MHz, DMSO) δ (ppm): 128.78, 133.68, 133.73, 163.52.

2,5-Bis(2-chlorophenyl)-1,3,4-selenadiazole. White solid; mp 128–131 °C.

^{1}H NMR (400 MHz, DMSO) δ (ppm): 7.40–7.44 (m, 1H), 7.51–7.53 (m, 1H), 7.75 (d, J = 6.84 Hz, 1H).

^{13}C NMR (100 MHz, DMSO) δ (ppm): 127.72, 131.03, 131.15, 131.89, 133.06, 167.34.

2,5-Bis(2-bromophenyl)-1,3,4-selenadiazole. Light brown solid; mp 125–127 °C.

^{1}H NMR (400 MHz, DMSO) δ (ppm): 7.41–7.49 (m, 1H), 7.70–7.55 (m, 1H).

^{13}C NMR (100 MHz, DMSO) δ (ppm): 120.38, 128.18, 131.03, 132.99, 134.21, 167.85.

2,5-Di-*p*-tolyl-1,3,4-selenadiazole. Off white solid; mp 143–146 °C.

^{1}H NMR (400 MHz, DMSO) δ (ppm): 7.27 (d, J = 7.92 Hz, 2H), 7.83 (t, J = 8.08 Hz, 2H).

^{13}C NMR (100 MHz, DMSO) δ (ppm): 128.45, 129.54, 129.78, 143.47, 167.81.

2,2′-(1,3,4-Selenadiazole-2,5-diyl)diphenol. White solid; mp 162–165 °C.

^{1}H NMR (400 MHz, DMSO) δ (ppm): 6.97 (t, J = 7.6 Hz, 1H), 7.05 (d, J = 8.4 Hz, 1H), 7.37 (t, J = 7.6 Hz, 1H), 7.97 (t, J = 7.4 Hz, 1H).

^{13}C NMR (100 MHz, DMSO) δ (ppm): 111.07, 116.90, 119.95, 120.25, 129.57, 133.15, 155.85.

4,4′-(1,3,4-Selenadiazole-2,5-diyl)bis(2-methoxyphenol). Off white solid; mp 187–189 °C.

^{1}H NMR (400 MHz, DMSO) δ (ppm): 6.89–6.95 (m, 2H), 7.08 (d, J = 1.6 Hz, 1H).

Author contributions

K. Datta has completed the whole experimental work, preparation of manuscript. Dr B. Mitra and Dr G. C. Pariyar helped him

in the selection of work and reviewing and editing of the whole procedure under the supervision of Prof. P. Ghosh.

Conflicts of interest

The authors declare no conflicts of interest.

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