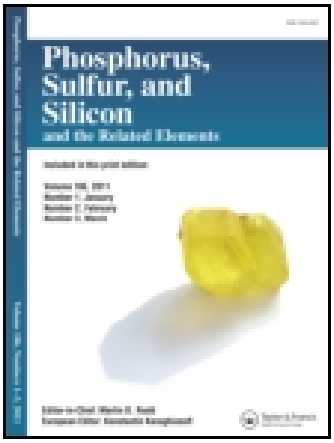

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gpss20

One-Pot Synthesis of 1-(E)-Phenylethenyl-1,2,3-Triazoles by Sequential Click-Elimination Reaction from 2-Azido-1-Phenyl-1-(Phenylseleno)Ethane

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Accepted author version posted online: 05 Apr 2013.Published online: 20 Sep 2013.

To cite this article: Hai-You Su , Shou-Ri Sheng , Xiao-Lan Zhang , Xiao-Ying Ding & Ming-Zhong Cai (2013) One-Pot Synthesis of 1-(E)-Phenylethenyl-1,2,3-Triazoles by Sequential Click-Elimination Reaction from 2-Azido-1-Phenyl-1-(Phenylseleno)Ethane, Phosphorus, Sulfur, and Silicon and the Related Elements, 188:11, 1591-1598, DOI: 10.1080/10426507.2013.769987

To link to this article: http://dx.doi.org/10.1080/10426507.2013.769987

Phosphorus, Sulfur, and Silicon, 188:1591–1598, 2013

ISSN: 1042-6507 print / 1563-5325 online
DOI: 10.1080/10426507.2013.769987

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ONE-POT SYNTHESIS OF 1-*(E)*-PHENYLETHENYL-1,2,3-TRIAZOLES BY SEQUENTIAL CLICK-ELIMINATION REACTION FROM 2-AZIDO-1-PHENYL-1-(PHENYLSELENO)ETHANE

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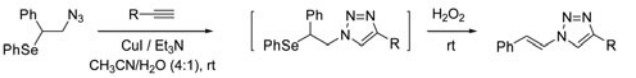
GRAPHICAL ABSTRACT



Abstract *A new convenient one-pot, two-step procedure involving sequential click chemistry and oxidation-elimination reaction for the preparation of 1,4-disubstituted 1,2,3-triazoles bearing 1-(E)-phenylethenyl group from 2-azido-1-phenyl-1-(phenylseleno)ethane is described. The prominent features of this protocol are mild reaction conditions, operational simplicity, and good to high yields of products, as well as avoidance of the isolation of the selenated intermediate.*

Supplementary materials are available for this article. Go to the publisher's online edition of Phosphorus, Sulfur, and Silicon and the Related Elements *for the following free supplemental files: Additional figures.*

Keywords 1-*(E)*-Phenylethenyl-1,2,3-triazoles; one-pot; 2-azido-1-phenyl-1-(phenylseleno) ethane; click chemistry; oxidation-elimination

Received 12 December 2012; accepted 22 January 2013.

Financial support from the National Natural Science Foundation of China (No. 21062007) and the Research Program of Jiangxi Province Department of Education (Nos. GJJ10385, GJJ10733, and GJJ11380) is gratefully acknowledged.

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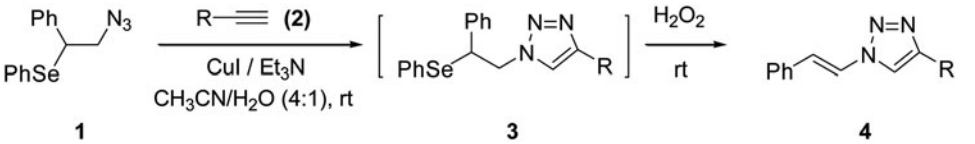
INTRODUCTION

Heterocyclic compounds are important structural units useful in medicinal chemistry and valuable as synthetic organic building blocks.[1] Among the various heterocycles, the 1,2,3-triazole class of heterocycles has attracted considerable interest because of its biological activities including antimicrobial,[2] antiallergic,[3] and anti-HIV[4] properties, and its wide applications in many fields such as organic synthesis,[5–7] material science,[8–10] and medicinal chemistry.[11–13] Since the discovery from the research groups of Sharpless[14] and Meldal,[15] the copper (I)-catalyzed 1,3-dipolar Huisgen cycloaddition reaction between azides and terminal alkynes (CuAAC),[16] "click-chemistry" has received growing interest in the regioselective synthesis of 1,4-disubstituted 1,2,3-triazoles efficiently.[17–23] Because of their importance, numerous structurally different 1,2,3-triazoles have been developed. But, less attention has been focused on the synthesis of styryl-substituted 1,2,3-triazoles.[24–26] Therefore, the synthesis of functionalized 1,2,3-triazoles containing a styryl group via a simple, step-economic, less hazardous methodology is still in demand.

It is well known that organoselenium reagents have been increasingly used as a powerful tool for the introducing new functional groups into organic substrates under extremely mild conditions.[27] For instance, the phenylseleno group is readily converted to a leaving group giving access to a carbon–carbon double bond via oxidation followed by β-elimination. On the other hand, one-pot sequential multicomponent reactions (MCRs), which combine two or more completely different reactions in a single transformation, provide significant advantage over conventional stepwise synthesis avoiding tedious chromatographic purifications. For this reason, more recent sequential one-pot processes have been utilized in rapid and convergent construction of 1,2,3-triazole molecules without the isolation of intermediates.[28–32] However, to our knowledge, there are few reported examples of the synthesis of 1,4-disubstituted 1,2,3-triazoles containing 1-phenylethyenyl group except 1-(*E*)-phenylethenyl-4-phenyl-1,2,3-triazole.[25,27] As part of an ongoing research program focused on the use organic selenium reagents in organic synthesis,[33] we report herein a one-pot, two-step route to the synthesis of 1-(*E*)-phenylethyenyl-1,2,3-triazoles using 2-azido-1-phenyl-1-(phenylseleno)ethane **(1)** as original material by sequential click and oxidation-elimination reactions, as depicted in Scheme 1.



Scheme 1

RESULTS AND DISCUSSION

In order to find the most suitable conditions for our purpose, copper (I) iodide as a catalyst in combination with different solvents at room temperature, were initially investigated using compound **1**[34] and phenylacetylene (**2a**) as model substrates for the synthesis of 1-(1-phenyl-1-phenylselenoethyl)-4-phenyl-1,2,3-triazole (**3a**), and the results are summarized in Table 1. First of all, dichloromethane was chosen as the solvent, but no product was obtained after the reaction proceed at room temperature even for 24 h in the presence

Table 1 Optimization conditions for the reaction of 1 and 2a[a]

Entry	Cu (I) catalyst	Solvent	Base	Time	Yield (%)[b]
1	CuI	CH_2Cl_2	—	24 h	—
2	CuI	CH_2Cl_2	Et_3N	24 h	48
3	CuI	THF	Et_3N	24 h	50
4	CuI	*t*-BuOH	Et_3N	24 h	42
5	CuI	DMSO	Et_3N	24 h	70
6	CuI	CH_3CN	Et_3N	15 h	88
7	CuI	CH_3CN/H_2O (4:1)	Et_3N	10 h	94
8	CuI	CH_3CN/H_2O (4:1)	Pyridine	15 h	75
9	CuI	CH_3CN/H_2O (4:1)	*i*-Pr_2NEt	15 h	70
10	$CuSO_4$ /Na ascorbate	CH_3CN/H_2O (4:1)	Et_3N	24 h	75
11	CuBr	CH_3CN/H_2O (4:1)	Et_3N	24 h	65
12	CuCl	CH_3CN/H_2O (4:1)	Et_3N	24 h	60
13	CuI	CH_3CN/H_2O (4:1)	Et_3N	6 h	62
14	CuI	CH_3CN/H_2O (4:1)	Et_3N	10 h	80[c], 94[d]
15	CuI	CH_3CN/H_2O (4:1)	Et_3N	10 h	95[e]
16	CuI	CH_3CN/H_2O (4:1)	Et_3N	24 h	70[f]
17	CuI	CH_3CN/H_2O (4:1)	Et_3N	10 h	94[g]
18	–	CH_3CN/H_2O (4:1)	Et_3N	24 h	0

[a]Reaction conditions: 1 (1.0 mmol), 2a (1.2 mmol), catalyst (10 mol%), base (1.2 mmol), solvent (5 mL), r.t.
[b]Isolated yield based on 1 after column chromatography.
[c]1.0 equivalent of triethylamine was used.
[d]1.5 equivalent of triethylamine was used.
[e]1.5 mmol of 2a was used.
[f]5 mol% of CuI was used.
[g]15 mol% of CuI was used.

of 10 mol% copper (I) iodide (Table 1, entry 1). It was reported that organic bases could promote the "click reaction,"[15] when 1.2 equivalent of triethylamine was added to the reaction system, the reaction gave a 48% yield of the corresponding compound **3a** (Table 1, entry 2), which is in accordance with the reported results that organic bases could promote the generation of the active copper acetylide complex, mediating the "click" reaction.[7] Encouraged by this result, other polar solvents such as tetrahydrofuran (THF), *t*-BuOH, CH_3CN, and dimethyl sulfoxide (DMSO) were further examined. After several experiments, it was found that CH_3CN seems to be the best solvent to furnish good yield (88%) (Table 1, entry 6). However, replacement of CH_3CN with CH_3CN/H_2O (4:1) co-solvent gave the desired product **3a** in better yield (94%) within 10 h of reaction (Table 1, entry 7). Thus, the CH_3CN/H_2O (4:1) was chosen as the typical co-solvent in subsequent optimizing experiments. Second, other organic bases such as diisopropylethylamine (DIPEA) and pyridine were introduced into the reaction and resulted in a drop of yields (Table 1, entries 8 and 9). Furthermore, reactions with other copper (I)-catalysts including $CuSO_4 \cdot 5H_2O$/Na ascorbate, CuBr, and CuCl generated the desired product **3a** in lower yield (Table 1, entries 10–12).

It is notable that the yield of **3a** dramatically dropped to 62% when the reaction time was decreased from 10 to 6 h (Table 1, entry 13). When 1.0 equivalent of triethylamine was utilized, the reaction gave rise to a reduction of the yield, while adding 1.5 equivalent of triethylamine to the reaction system, **3a** was obtained with quite similar results (Table 1, entry 14). Additionally, if the quantity of the **2a** was increased from 1.2 to 1.5 equivalents,

Table 2 Yields of 1-(*E*)-phenylethenyl-1,2,3-triazoles (4a–4i)

Entry	R	Product	Yield (%) [a]
1	C_6H_5	4a	90
2	4-$C_2H_5C_6H_4$	4b	91
3	2-$CH_3OC_6H_4OCH_2$	4c	89
4	2-$CH_3C_6H_4OCH_2$	4d	88
5	2-$ClC_6H_4OCH_2$	4e	86
6	2-$NO_2C_6H_4OCH_2$	4f	83
7	4-$NO_2C_6H_4OCH_2$	4g	84
8	2-Naphthoxymethyl	4h	85
9	n-C_4H_9	4i	82

[a]Isolated yield based on 1 after column chromatography.

the yield of **3a** was not improved significantly (Table 1, entries 7 and 15). Nevertheless, reducing the amount of CuI to 5 mol%, the **3a** yield dropped to 70% (Table 1, entry 16), whereas using 15 mol% CuI, the yield of the product **3a** was not increased (Table 1, entry 17). Clearly, the reaction did not proceed in the absence of CuI (Table 1, entry 18).

Subsequently, oxidation-elimination of **3a** with 30% hydrogen peroxide was performed in CH_3CN at room temperature to afford 1-(*E*)-phenylethenyl-4-phenyl-1,2,3-triazole (**4a**) in 90% yield. Indeed, although selenated intermediate **3a** can be isolated and purified by chromatography, we have found it most convenient to carry out the oxidation of the material in one-pot. Therefore, the one-pot preparation of **4a** was carried out again by treating **1** with phenylacetylene (**2a**) (1.2 equiv) in CH_3CN/H_2O (4:1) at room temperature for 10 h in the presence of triethylamine (1.2 equiv), followed by the addition excess of 30% hydrogen peroxide. After stirring for a further 1 h at room temperature (monitoring by TLC), **4a** was isolated in 90% yield as an exclusive product in one-pot, two-step (Table 2, entry 1).

In order to prove the applicability of the conditions for the preparation of different triazoles bearing 1-substituted phenylethenyl group at position 1, the methodology was extended to a series of terminal alkynes including aryl-, aryloxymethyl-, and n-butylacetylenes. As shown in Table 2, both aromatic and aliphatic alkynes underwent the reaction smoothly under the optimized reactions, and the corresponding 1,4-disubstituted triazoles (**4a–4i**) were obtained exclusively in good to excellent yields (82% to 91%).

It should be pointed out that the (*E*) configuration of the double bond of all products (**4a–4i**) was established from the coupling constants of the olefinic protons in their NMR spectra. For example, in the ^{1}H NMR spectrum of 1-*(E)*-phenylethenyl-4-(4-nitrophenoxymethyl)-1,2,3-triazole (**4g**), the coupling constants ($J = 14.8$ Hz) between the two olefinic protons at $\delta = 7.77$ and 7.22 ppm were observed, which indicated its exclusive production of *E*-isomer. In addition, the ^{1}H NMR spectrum of **4g** exhibited a typical singlet at $\delta = 7.99$ ppm for the triazolyl C_5–H proton, and its IR spectrum showed a weak C=C absorption at 1654 cm^{-1} and a strong unsaturated =C−H stretch at 951 cm^{-1}, corresponding its (*E*) configuration, and the characteristic band at 1510 cm^{-1} indicated the presence of a nitro group.

Further, it is worthy to mention that the phenylseleno moiety, introduced in the starting material, is eliminated as benzeneseleninic acid in the oxidation step. After neutralization

and acidification by addition of concd HCl to the alkaline aqueous extract, the water-soluble benzeneseleninate was converted into benzeneseleninic acid. After treatment with hydrazine monohydrate, diphenyl diselenide can be recovered [35,36] in 62% yield.

In summary, a novel one-pot tandem procedure for the synthesis of 1,4-disubstituted 1,2,3-triazoles containing 1-(*E*)-phenylethenyl group have been developed by the reaction of 2-azido-1-phenyl-1-(phenylseleno)ethane with terminal alkynes in the presence of copper (I) iodide and triethylamine in CH_3CN-H_2O co-solvent, followed by treatment of 30% H_2O_2. This convenient synthesis using sequential click chemistry and oxidation-elimination reaction gives the corresponding target compounds in good to excellent yields with simple work-up, mild reaction conditions, and without the isolation of the selenated intermediate. Further studies on this subject are currently in progress.

EXPERIMENTAL

Melting points were measured with an XT-4 melting point apparatus and were uncorrected. IR spectra were recorded on a Perkin-Elmer SP One FT-IR spectrophotometer. Mass spectra (EI, 70 eV) were recorded on a HP5989B mass spectrometer. Microanalyses were performed with a Carlo Erba 1106 Elemental Analyzer. The NMR spectra were taken on a Bruker 400 spectrometer operating at 400 MHz for ^{1}H and 100 MHz for ^{13}C, respectively, with TMS as an internal standard, and the chemical shifts are reported in δ units. The values of chemical shifts (δ) are given in ppm and coupling constants (J) in Hz. 2-Azido-1-phenyl-1-(phenylseleno)ethane (**1**) was prepared according to the literature procedures.[34] The other chemicals (AR grade) were obtained from commercial sources and used without further purification. All reactions were monitored by thin-layer chromatography (TLC), which was performed on silica gel 60 F254 (0.25 mm thickness), precoated aluminum sheets. Column chromatography was performed on silica gel (60–120 mesh) using ethyl acetate/hexane mixtures as the eluent. Selected ^{1}H and ^{13}C NMR spectra for the products are presented in the Supplemental Materials (Figures S1–S18).

Preparation of Triazoles (4a–4i): General Procedure

To a solution of 2-azido-1-phenyl-1-(phenylseleno)ethane (**1**) (302 mg, 1.0 mmol) in CH_3CN/H_2O (4:1, 5 mL) was added terminal alkyne (**2**) (1.2 mmol), Et_3N (1.2 mmol) and CuI (19 mg, 10 mol%), and the reaction mixture was stirred at room temperature. After completion of the reaction (10–12 h, as monitored by TLC), the mixture was cooled to 0°C, 30% hydrogen peroxide (1.0 mL, 11.6 mmol) was added, the reaction mixture was then warmed to room temperature and stirred for 1–1.5 h until the reaction was finished as determined by TLC. Finally, the resulting mixture was extracted with ether (3 ×10 mL), and the combined organic layers were washed with brine (3 mL), dried over anhydrous sodium sulfate, and concentrated under vacuum to give a crude product that was purified by column chromatography eluted with ethyl acetate/hexane to afford **4a–4i**.

1-*(E)*-Phenylethenyl-4-phenyl-1,2,3-triazole (4a). Pale yellow solid, mp 95–96°C (lit.[26] 96–98°C). ^{1}H NMR: δ = 7.79–7.77 (m, 2 H), 7.72 (s, 1 H), 7.42–7.32 (m, 8 H), 5.80 (s, 1 H), 5.48 (s, 1 H). ^{13}C NMR: δ = 146.6, 142.0, 133.7, 129.2, 128.9, 128.0, 127.8, 127.4, 126.4, 124.8, 118.8, 108.4. IR (film): ν = 3061, 2925, 2854, 1644, 1577, 1456, 1429, 1267, 1233, 1074, 1021, 898, 808, 763, 692 cm^{-1}.

1-*(E)*-Phenylethenyl-4-(4-ethylphenyl)-1,2,3-triazole (4b). Pale yellow viscous oil. ^{1}H NMR: δ = 7.90 (s, 1 H), 7.71–7.68 (m, 2 H), 7.41–7.29 (m, 5 H), 7.19–7.12 (m, 2 H),

5.78 (s, 1 H), 5.47 (s, 1 H), 2.60 (q, $J = 7.2$ Hz, 2 H), 1.20 (t, $J = 7.2$ Hz, 3 H). ^{13}C NMR: $\delta =$ 147.7, 144.6, 143.1, 134.7, 129.9, 128.9, 128.4, 127.7, 127.4, 125.8, 119.5, 109.3, 28.7, 15.5. IR (film): $\nu = 3028, 2964, 2928, 1642, 1496, 1445, 1265, 1230, 1017, 896, 839, 800,$ 772, 693 cm^{-1}. EIMS: m/z (%) = 275 [M$^+$]. Anal. Calcd for $C_{18}H_{17}N_3$: C, 78.52; H, 6.22; N, 15.26. Found: C, 78.39; H, 6.38; N, 15.15.

1-*(E)*-Phenylethenyl-4-(2-methoxyphenoxymethyl)-1,2,3-triazole (4c). White solid, mp 123–124°C. ^{1}H NMR: $\delta = 7.96$ (s, 1 H), 7.75 (d, $J = 14.8$ Hz, 1 H), 7.47 (d, J = 7.6 Hz, 2 H), 7.41–7.31 (m, 3 H), 7.15 (d, $J = 14.8$ Hz, 1 H), 7.07 (d, $J = 7.6$ Hz, 1 H), 6.97–6.88 (m, 3 H), 5.35 (s, 2 H), 3.88 (s, 3 H). ^{13}C NMR: $\delta = 149.6, 147.5, 144.8,$ 133.5, 129.0, 128.8, 126.8, 122.9, 122.0, 121.9, 120.9, 120.3, 114.3, 111.9, 63.1, 55.9. IR (KBr): $\nu = 3153, 3115, 2953, 1654, 1590, 1570, 1503, 1465, 1456, 1439, 1393, 1359,$ 1325, 1288, 1251, 1177, 1123, 1054, 1026, 1010, 956, 875, 750 cm^{-1}. EIMS: m/z (%) = 307 [M$^+$]. Anal. Calcd for $C_{18}H_{17}N_3O_2$: C, 70.34; H, 5.58; N, 13.67. Found: C, 70.20; H, 5.74; N, 13.55.

1-*(E)*-Phenylethenyl-4-(2-methylphenoxymethyl)-1,2,3-triazole (4d). Pale yellow solid, mp 129–130°C. ^{1}H NMR: $\delta = 7.90$ (s, 1 H), 7.77 (d, $J = 14.4$ Hz, 1 H), 7.48 (d, $J =$ 7.2 Hz, 2 H), 7.41–7.32 (m, 3 H), 7.19–7.13 (m, 3 H), 6.97 (d, $J = 8.4$ Hz, 1 H), 6.92 (d, $J = 14.4$ Hz, 1 H), 5.28 (s, 2 H), 2.04 (s, 3 H). ^{13}C NMR: $\delta = 156.4, 145.2, 133.5, 130.8,$ 129.0, 128.9, 127.0, 126.9, 126.8, 123.0, 122.1, 121.1, 120.0, 111.5, 62.2, 16.3. IR (KBr): $\nu = 3136, 3096, 2927, 1655, 1493, 1454, 1383, 1350, 1239, 1050, 1021, 950, 752$ cm^{-1}. EIMS: m/z (%) = 291 [M$^+$]. Anal. Calcd for $C_{18}H_{17}N_3O$: C, 74.20; H, 5.88; N, 14.42. Found: C, 74.03; H, 5.96; N, 14.32.

1-*(E)*-Phenylethenyl-4-(2-chlorophenoxymethyl)-1,2,3-triazole (4e). Pale yellow solid, mp 106–107°C. ^{1}H NMR: $\delta = 7.98$ (s, 1 H), 7.76 (d, $J = 14.8$ Hz, 1 H), 7.47 (d, $J = 7.2$ Hz, 2 H), 7.41–7.32 (m, 4 H), 7.26–7.19 (m, 2 H), 7.15 (d, $J = 15.6$ Hz, 1 H), 6.92–6.89 (m, 1 H), 5.34 (s, 2 H). ^{13}C NMR: $\delta = 153.7, 144.4, 133.5, 130.4, 129.0, 128.9,$ 127.9, 126.8, 123.2, 122.9, 122.2, 120.3, 116.2, 114.3, 63.4. IR (KBr): $\nu = 3104, 3086,$ 2931, 1654, 1587, 1485, 1388, 1350, 1242, 1161, 1133, 1049, 1018, 969, 856, 750 cm^{-1}. EIMS: m/z (%) = 311 [M$^+$]. Anal. Calcd for $C_{17}H_{14}ClN_3O$: C, 65.49; H, 4.53; N, 13.48. Found: C, 65.34; H, 4.68; N, 13.35.

1-*(E)*-Phenylethenyl-4-(2-nitrophenoxymethyl)-1,2,3-triazole (4f). Pale yellow solid, mp 134–135°C. ^{1}H NMR: $\delta = 8.04$ (s, 1 H), 7.87 (dd, $J = 8.0, 1.2$ Hz, 1 H), 7.76 (d, $J = 14.8$ Hz, 1 H), 7.58–7.54 (m, 1 H), 7.48 (d, $J = 7.2$ Hz, 2 H), 7.41–7.30 (m, 4 H), 7.22 (d, $J = 14.8$ Hz, 1 H), 7.10–7.06 (m, 1 H), 5.42 (s, 2 H). ^{13}C NMR: $\delta = 151.4, 143.6,$ 140.2, 134.3, 133.4, 129.0, 128.8, 126.8, 125.7, 122.8, 122.4, 121.2, 120.7, 115.4, 63.7. IR (KBr): $\nu = 3071, 2957, 1658, 1606, 1517, 1347, 1275, 1251, 1168, 1044, 993, 957, 862,$ 775, 750, 693 cm^{-1}. EIMS: m/z (%) = 322 [M$^+$]. Anal. Calcd for $C_{17}H_{14}N_4O_3$: C, 63.35; H, 4.38; N, 17.38. Found: C, 63.19; H, 4.56; N, 17.26.

1-*(E)*-Phenylethenyl-4-(4-nitrophenoxymethyl)-1,2,3-triazole (4g). Pale yellow solid, mp 149–150°C. ^{1}H NMR: $\delta = 8.22$–8.19 (m, 2 H), 7.99 (s, 1 H), 7.77 (d, $J =$ 14.8 Hz, 1 H), 7.48 (d, $J = 7.6$ Hz, 2 H), 7.42–7.33 (m, 3 H), 7.22 (d, $J = 14.8$ Hz, 1 H), 7.11–7.07 (m, 2 H), 5.36 (s, 2 H). ^{13}C NMR: $\delta = 163.0, 143.1, 141.9, 133.3, 129.0,$ 128.8, 126.8, 126.0, 122.7, 122.6, 120.6, 114.9, 62.3. IR (KBr): $\nu = 3080, 2930, 2875, 1654,$ 1607, 1591, 1510, 1495, 1459, 1348, 1297, 1249, 1176, 1153, 1113, 1048, 1018, 1006, 951, 868, 846, 751, 696 cm^{-1}. EIMS: m/z (%) = 322 [M$^+$]. Anal. Calcd for $C_{17}H_{14}N_4O_3$: C, 63.35; H, 4.38; N, 17.38. Found: C, 63.21; H, 4.53; N, 17.24.

1-*(E)*-Phenylethenyl-4-(2-naphthoxymethyl)-1,2,3-triazole (4h). White solid, mp 158–159°C. ^{1}H NMR: $\delta = 7.97$ (s, 1 H), 7.97–7.74 (m, 4 H), 7.48–7.44 (m, 3 H), 7.40–7.34

(m, 5 H), 7.23–7.20 (m, 1 H), 7.19 (d, $J = 14.8$ Hz, 1 H), 5.40 (s, 2 H). ^{13}C NMR: $\delta =$ 156.1, 144.6, 134.4, 133.5, 129.7, 129.2, 129.1, 128.9, 127.7, 126.9, 126.8, 126.6, 124.0, 122.9, 122.1, 120.1, 118.7, 107.3, 62.0. IR (KBr): $\nu = 3055$, 2917, 1658, 1627, 1599, 1511, 1461, 1390, 1260, 1218, 1183, 1119, 1043, 951, 837, 818, 749, 694 cm^{-1}. EIMS: m/z (%) = 327 [M$^+$]. Anal. Calcd for $C_{21}H_{17}N_3O$: C, 77.04; H, 5.23; N, 12.84. Found: C, 76.88; H, 5.38; N, 12.71.

1-*(E)*-Phenylethenyl-4-(n-butyl)-1,2,3-triazole (4i). Colorless oil. ^{1}H NMR: $\delta =$ 7.98 (s, 1 H), 7.75 (d, $J = 14.8$ Hz, 1 H), 7.42–7.38 (m, 2 H), 7.34–7.27 (m, 3 H), 7.15 (d, $J = 14.8$ Hz, 1 H), 2.70 (t, $J = 7.2$ Hz, 2 H), 1.68–1.60 (m, 2 H), 1.41–1.39 (m, 2 H), 0.98 (t, $J = 7.6$ Hz, 3 H). ^{13}C NMR: $\delta = 147.2$, 135.9, 133.9, 128.7, 128.1, 126.9, 125.7, 118.8, 32.3, 26.0, 22.6, 14.1. IR (film): $\nu = 3050$, 2923, 1655, 1625, 1600, 1464, 1390, 1263, 1222, 1180, 1119, 1040, 950, 835, 750, 695 cm^{-1}. EIMS: m/z (%) = 227 [M$^+$]. Anal. Calcd for $C_{14}H_{17}N_3$: C, 73.98; H, 7.54; N, 18.49. Found: C, 73.83; H, 7.69; N, 18.36.

Recovery of Diphenyl Diselenide

To the alkaline aqueous extract from the oxidation procedure (containing the benzeneseleninate anion) was added conc HCl, after which, the resulting suspension was evaporated and the residue was suspended in methanol (30 mL), and hydrazine monohydrate (3.5 mmol, 0.16 mL) was added gradually to the suspension and stirred until diphenyl diselenide was formed, as indicated by the yellow color. The mixture was then concentrated in vacuo, poured into water (30 mL) and extracted with ether (3 × 10 mL). The organic layer was dried over anhydrous sodium sulfate and evaporated. Diphenyl diselenide was recovered as a pure compound in 62% yield.

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