

Solid-phase organic synthesis of vinyl-substituted 1,2,4-triazoles based on polymer-supported α -selenopropionic acid

Gui-Yun Fu, Lei Guo, Xue-Chun Mao, Shou-Ri Sheng,* Shi-Yang Fei,
and Ming-Zhong Cai

*College of Chemistry and Chemical Engineering, Jiangxi Normal University (Yaohu Campus),
Nanchang, 330022, P. R. China
E-mail: shengsr@jxnu.edu.cn*

Abstract

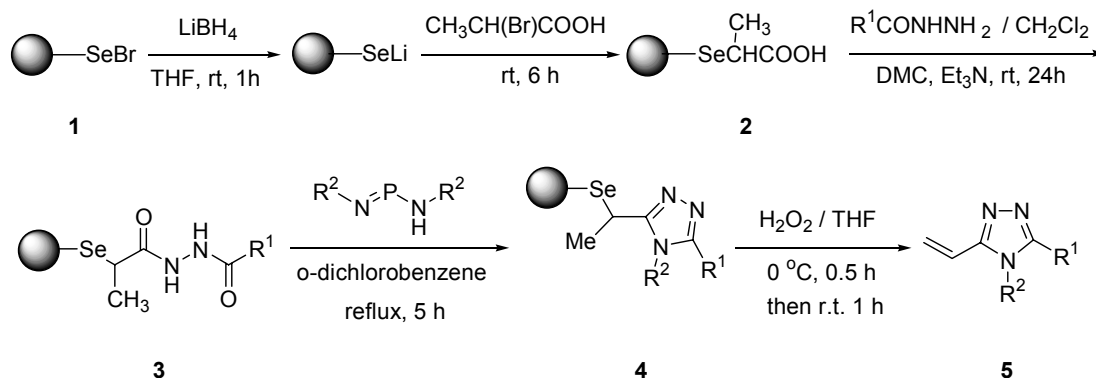
Acylation of polystyrene-supported α -selenopropionic acid with acid hydrazides, followed by cyclocondensation with arylphosphazoanilides, and oxidation-elimination with 30 % hydrogen peroxide efficiently afforded vinyl-substituted 1,2,4-triazoles in good yield and purity, with advantages of decreased volatility and simplification of work-up procedure.

Keywords: Solid-phase organic synthesis, polystyrene-supported α -selenopropionic acid, vinyl-substituted 1,2,4-triazole

Introduction

Polymer-supported reagents have attracted growing interest because they can provide attractive and practical methods for combinatorial chemistry and solid-phase organic synthesis (SPOS) in recent years.¹ SPOS using insoluble solid supports such as polystyrene resins takes advantage of the simple removal of excess or consumed reagents by a simple filtration workup operation. Now, the design and synthesis of pharmacologically relevant heterocyclic molecules using SPOS methodology is recognized as a valuable tool for acceleration of drug discovery. The 1,2,4-triazole system has been known to be an important heterocycle in biologically active molecules. This five-numbered ring was also found in potent agonist or antagonist receptor ligands.² Furthermore, 1,2,4-triazole derivatives have been used as mimics³ of the amide bond in order to increase bioavailability of the parent bioactive molecules. They have also been incorporated into peptides to surrogate *cis* amide bonds.⁴ Among the 1,2,4-triazoles, vinyl substituted derivatives have now attracted considerable attention because of their biological properties,⁵ such as antiallergic, antibacterial, and anti-HIV activity. However, to our knowledge, vinyl substituted 1,2,4-triazoles are seldom reported.⁶ Although SPOS of 1,2,4-triazoles are investigated,⁷ efforts are continuing for the development of more efficient methods with experimental simplicity.

Recently, we have reported vinyl-substituted 1,2,4-triazoles using polymer-supported ethyl β -phenylselenopropionate reagent.⁸ In continuation of our interest in SPOS of biologically relevant heterocyclic compounds based on polymeric selenium reagent, we here wish to describe another simple and efficient traceless solid-phase synthetic approach to vinyl-substituted 1,2,4-triazoles based on polymer-supported α -selenopropionic acid (Scheme 1).



Scheme 1. SPOS of vinyl-substituted 1,2,4-triazoles.

Results and Discussions

Polymer-supported α -selenopropionic acid **2** was prepared by treatment of a THF-swollen suspension of cross-linked (1%) polystyrene bound selenium bromide **1**⁹ with LiBH_4 , followed by treatment with 2-bromopropionic acid according to our previous method.¹⁰ The minimum loading of COOH of resin **2** verified by their FT-IR spectra showing a strong carbonyl absorption at 1726 cm^{-1} were determined by acid-base titration¹¹ to be 1.20 mmol/g . With resin **2** in hand, the acylation of the polymeric α -selenopropionic acid **2** with various acid hydrazides, the key for the success of this protocol was investigated. Here, the diacylhydrazination reaction was investigated starting from resin **2** and benzoic hydrazide. When DCC (dicyclohexyl carbodiimide) or HBTU (*O*-benzotriazol-1-yl-*N,N,N',N'*-tetramethyluronium hexafluorophosphate) was used as a coupling reagent to promote above reaction at room temperature or under reflux in CH_2Cl_2 or THF for 10 h or even for longer time, the diacylhydrazination on solid-phase was not complete as monitored by FT-IR study, which showed two strong peaks of carbonyl absorptions near 1726 and 1595 cm^{-1} . Interestingly, when DMC (2-chloro-1,3-dimethylimidazolium chloride)¹² was added to the suspension of resin **2** with benzoic hydrazide in CH_2Cl_2 at room temperature, the conversion of resin **2** to polymer-bound diacyl hydrazide **3a** was complete. The FT-IR spectrum of resin **3a** showed a single strong carbonyl peak at 1595 cm^{-1} , with disappearance of the band at 1726 cm^{-1} . Next, the cyclocondensation of diacyl hydrazide resin **3** with arylphosphazone (Ar-N=P(NH)-Ar)¹³ proceeded smoothly according our published method to furnish resin **4** (FT-IR spectra showed

C=N group absorptions at 1635-1641 cm^{-1} , with disappearance of the band at 1595 cm^{-1}), followed by selenoxide syn elimination with excess of 30 % hydrogen peroxide to afford the corresponding vinyl-substituted 1,2,4-triazoles **5** in moderate to good yields (74-83 %) and with good purities (88-94 %) of crude materials in all cases (Table 1). The residual resin, polystyrene-supported phenylseleninic acid, was obtained as a by-product, whose infrared data was identical to the previously reported data,¹⁴ and showed no residual C=N group absorption indicating the oxidation-elimination was complete.

Table 1. The yields and purities of vinyl-substituted 1,2,4-triazoles **5a-5l**

Entry	R ¹	R ²	Products	Yield (%) ^a	Purity (%) ^b
1	C ₆ H ₅	C ₆ H ₅	5a	82	94
2	C ₆ H ₅	4-CH ₃ C ₆ H ₄	5b	81	90
3	C ₆ H ₅	3-CH ₃ OC ₆ H ₄	5c	80	91
4	C ₆ H ₅	4-ClC ₆ H ₄	5d	78	89
5	C ₆ H ₅ CH ₂	2-CH ₃ C ₆ H ₅	5e	75	88
6	C ₆ H ₅ CH ₂	3-CH ₃ OC ₆ H ₄	5f	76	90
7	C ₆ H ₅ CH ₂	2,4-(CH ₃) ₂ C ₆ H ₃	5g	80	92
8	C ₆ H ₅ CH ₂	4-ClC ₆ H ₄	5h	82	91
9	CH ₃	C ₆ H ₅	5i	83	92
10	CH ₃	3-CH ₃ OC ₆ H ₄	5j	80	91
11	CH ₃	4-ClC ₆ H ₄	5k	78	90
12	CH ₃	1-naphthalenyl	5l	74	88

^aOverall yields based on polystyrene-supported α -selenopropionic acid **2** (1.20 mmol COOH/g).

^bDetermined by HPLC of crude cleavage product ($\lambda = 254 \text{ nm}$).

In summary, a novel and efficient procedure for the solid-phase synthesis of vinyl-substituted 1,2,4-triazoles in moderate to good yields and with good purities using polymer-supported α -selenopropionic acid. The advantages of this method include straightforward operation, lack of odor, stability, and high purity of the product.

Experimental Section

General Procedure. Melting points were determined on X₄ melting point apparatus and are uncorrected. ¹H NMR (400 MHz) and ¹³C NMR (100 MHz) spectra were recorded on a Bruker Avance (400 MHz) spectrometer using CDCl₃ as the solvent and TMS as an internal standard. FT-IR spectra were taken on a Perkin-Elmer SP One FT-IR spectrophotometer. Microanalyses were performed with a PE 2400 elemental analyzer. Mass spectra (EI, 70eV) were recorded on a HP5989B mass spectrometer. HPLC analysis was carried out on Agilent 1100 (250×4.6 mm C₁₈

Column, gradient elution 50/20/30 THF/CH₃OH/H₂O (v/v), 1 mL/min, UV detection at $\lambda = 254$ nm). Polystyrene (H 1000, 100-200 mesh, cross-linked with 1% divinylbenzene) for the preparation of polystyrene-supported selenium bromide⁹ and other starting materials were purchased from commercial suppliers and used without further purification. CH₂Cl₂ was distilled from phosphorous pentoxide and THF was stilled from sodium-benzophenone immediately prior to use. Arylphosphazoanilide (Ar-N=P-NH-Ar)⁸ and polystyrene-supported α -selenopropionic acid¹⁰ were prepared in our laboratory according to the literature procedure

General procedure for the preparation of vinyl-substituted 1,2,4-triazole derivatives (5a-5l)

Resin **2** (1.0 mmol) was swelled in anhydrous CH₂Cl₂ (20 mL) at room temperature for 30 min. Acid hydrazides (2.0 mmol), DMC (0.20 g, 1.2 mmol) and triethylamine (2.0 mmol) was added under nitrogen to the mixture. After 24 h with stirring at room temperature, the mixture was filtered and the resin was washed thoroughly successively with H₂O, THF, MeOH and CH₂Cl₂ (2×5 mL of each) and then dried under vacuum to afford resin **3**. Under a positive pressure of nitrogen, arylphosphazoanilide (3.0 mmol) was added to a suspension of the swollen resin **3** in 1,2-dichlorobenzene (15 mL). The mixture was stirred for 5 h at reflux. Resin **4** was collected by filtration and washed successively with DMF (3×10 mL), THF/H₂O (2:1) (2×10 mL), THF (2×10 mL), H₂O (2×10 mL), THF (2×10 mL), acetone (2×10 mL), THF (1×10 mL), CH₃OH (2×10 mL), and THF (1×10 mL). The washed resin **4** was then suspended in THF (15 mL), and 30 % hydrogen peroxide (0.5 mL, 5.8 mmol) was added; the mixture was stirred for 30 min at 0 °C, followed by 1 h at room temperature. The mixture was filtered, and the residual resin was washed with CH₂Cl₂ (3×10 mL). The filtrate was washed with H₂O (2×20 mL), dried over MgSO₄, and evaporated to dryness under vacuum to obtain the crude products **5**. Further purification was *via* flash chromatography with *n*-hexane/acetone (1:1, v/v) as the eluent for ¹H NMR, ¹³C NMR, and microanalysis.

4,5-Diphenyl-3-vinyl-1,2,4-triazole (5a). White solid. Mp 114-115 °C; ¹H NMR: $\delta = 7.52-7.50$ (m, 3H, Ar-*H*), 7.41-7.39 (m, 2H, Ar-*H*), 7.34-7.22 (m, 5H, Ar-*H*), 6.32-6.30 (m, 1H, =CH), 6.32-6.30 (m, 1H, =CH₂), 6.26-6.22 (m, 1H, =CH₂); ¹³C NMR: $\delta = 154.1$ (C3), 153.0 (C5), 134.5 (=CH), 130.0 (C), 129.7 (2 × CH), 129.5 (C), 128.2 (2 × CH), 128.1 (2 × CH), 127.5 (2 × CH), 126.8 (CH), 122.1 (CH), 120.4 (=CH₂); EIMS: m/z (%) = 247 (M⁺); IR (KBr): $\nu = 3060, 1596, 1498, 1468, 1445, 1428, 1080, 780, 696$ cm⁻¹; Anal. Calcd for C₁₆H₁₃N₃: C, 77.71; H, 5.30; N, 16.99. Found: C, 77.68; H, 5.36; N, 16.95.

4-(4-Methylphenyl)-5-phenyl-3-vinyl-1,2,4-triazole (5b). White solid. Mp 106-108 °C; ¹H NMR: $\delta = 7.45$ (d, $J = 8.8$ Hz, 2H, Ar-*H*), 7.34-7.26 (m, 5H, Ar-*H*), 7.09 (d, $J = 8.8$ Hz, 2H, Ar-*H*), 6.31-6.29 (m, 1H, =CH), 6.26-6.22 (m, 1H, =CH₂), 5.52-5.50 (m, 1H, =CH₂), 2.45 (s, 3H, CH₃); ¹³C NMR: $\delta = 154.0$ (C3), 153.4 (C5), 140.1 (C), 131.6 (=CH), 130.6 (2 × CH), 129.6 (2 × CH), 128.5 (2 × CH), 128.3 (2 × CH), 127.2 (C), 126.9 (CH), 122.3 (CH), 120.5 (=CH₂), 21.1 (CH₃); EIMS: m/z (%) = 261 (M⁺); IR (KBr): $\nu = 2920, 2851, 1632, 1515, 1470, 1449, 1430, 1395, 832, 760, 695$ cm⁻¹; Anal. Calcd for C₁₇H₁₅N₃: C, 78.13; H, 5.79; N, 16.08. Found: C, 78.16; H, 5.84; N, 16.05.

4-(3-Methoxyphenyl)-5-phenyl-3-vinyl-1,2,4-triazole (5c). White solid. Mp 82-83 °C; ^1H NMR: δ = 7.48-7.40 (m, 2H, Ar-*H*), 7.34-7.32 (m, 1H, Ar-*H*), 7.30-7.27 (m, 3H, Ar-*H*), 7.08-7.05 (m, 1H, Ar-*H*), 6.84-6.81 (m, 1H, Ar-*H*), 6.72-6.70 (m, 1H, Ar-*H*), 6.35-6.32 (m, 1H, =*CH*), 6.22-6.18 (m, 1H, =*CH*₂), 5.55-5.52 (m, 1H, =*CH*₂), 3.80 (s, 3H, OCH₃); ^{13}C NMR: δ = 160.8 (C), 153.8 (C3), 153.1 (C5), 135.5 (=CH), 130.7 (C), 129.5 (2 × CH), 128.4 (C), 128.2 (CH), 126.7 (CH), 122.5 (CH), 120.5 (=CH₂), 120.0 (2 × CH), 115.3 (CH), 113.2 (CH), 55.5 (OCH₃); EIMS: m/z (%) = 277 (M⁺); IR (KBr): ν = 3424, 3066, 2924, 2851, 1602, 1495, 1468, 1431, 1229, 1026, 695 cm⁻¹; Anal. Calcd for C₁₇H₁₅N₃O: C, 73.63; H, 5.45; N, 15.15. Found: C, 73.66; H, 5.48; N, 15.18.

4-(4-Chlorophenyl)-5-phenyl-3-vinyl-1,2,4-triazole (5d). White solid. Mp 159-161 °C; ^1H NMR: δ = 7.51 (d, J = 8.4 Hz, 2H, Ar-*H*), 7.39-7.26 (m, 5H, Ar-*H*), 7.18 (d, J = 8.4 Hz, 2H, Ar-*H*), 6.32-6.27 (m, 1H, =*CH*), 6.22-6.20 (m, 1H, =*CH*₂), 5.56-5.53 (m, 1H, =*CH*₂); ^{13}C NMR: δ = 153.6 (C3), 152.9 (C5), 135.7 (=CH), 132.5 (C), 130.2 (C), 129.5 (2 × CH), 128.8 (2 × CH), 128.5 (2 × CH), 128.1 (C), 126.3 (2 × CH), 122.6 (=CH₂), 120.0 (CH); EIMS: m/z (%) = 281 (M⁺); IR (KBr): ν = 3091, 3065, 1495, 1466, 1428, 1090, 1010, 935, 846, 758, 699 cm⁻¹; Anal. Calcd for C₁₆H₁₂ClN₃: C, 68.21; H, 4.29; N, 14.91. Found: C, 68.24; H, 4.32; N, 14.96.

4-(2-Methylphenyl)-5-benzyl-3-vinyl-1,2,4-triazole (5e). White solid. Mp 71-73 °C; ^1H NMR: δ = 7.39-7.36 (m, 1H, Ar-*H*), 7.28-7.20 (m, 2H, Ar-*H*), 7.13-7.05 (m, 3H, Ar-*H*), 6.95-6.91 (m, 1H, Ar-*H*), 6.84-6.81 (m, 2H, Ar-*H*), 6.20-6.12 (m, 1H, =*CH*), 5.95-5.91 (m, 1H, =*CH*₂), 5.36-5.34 (m, 1H, =*CH*₂), 4.04-3.91 (m, 2H, CH₂), 1.52 (s, 3H, CH₃); ^{13}C NMR: δ = 153.7 (C3), 152.3 (C5), 136.0 (=CH), 135.1 (C), 132.2 (CH), 131.2 (C), 130.3 (2 × CH), 128.7 (2 × CH), 128.4 (C), 127.8 (CH), 127.3 (CH), 126.5 (CH), 121.1 (=CH₂), 120.8 (CH), 31.5 (CH₂), 16.7 (CH₃); EIMS: m/z (%) = 275 (M⁺); IR (KBr): ν = 3036, 2925, 1498, 1460, 1441, 1018, 936, 770, 723, 699, 585 cm⁻¹; Anal. Calcd for C₁₈H₁₇N₃: C, 78.52; H, 6.22; N, 15.26. Found: C, 78.54; H, 6.26; N, 15.29.

4-(3-Methoxyphenyl)-5-benzyl-3-vinyl-1,2,4-triazole (5f). White solid. Mp 96-98 °C; ^1H NMR: δ = 7.36-7.28 (m, 1H, Ar-*H*), 7.18-7.15 (m, 3H, Ar-*H*), 7.03-6.97 (m, 3H, Ar-*H*), 6.61-6.58 (m, 1H, Ar-*H*), 6.34-6.32 (m, 1H, Ar-*H*), 6.26-6.20 (m, 1H, =*CH*), 6.15-6.11 (m, 1H, =*CH*₂), 5.46-5.43 (m, 1H, =*CH*₂), 4.02 (s, 2H, CH₂), 3.63 (s, 3H, OCH₃); ^{13}C NMR: δ = 160.2 (C), 153.8 (C3), 152.3 (C5), 135.7 (C), 134.2 (=CH), 130.3 (C), 128.5 (2 × CH), 128.3 (2 × CH), 126.6 (CH), 121.8 (=CH₂), 120.8 (CH), 119.5 (CH), 116.0 (CH), 112.7 (CH), 55.4 (OCH₃), 31.5 (CH₂); EIMS: m/z (%) = 291 (M⁺); IR (KBr): ν = 3029, 2905, 1496, 1441, 1090, 1030, 845, 746, 733 cm⁻¹; Anal. Calcd for C₁₈H₁₇N₃O: C, 74.20; H, 5.88; N, 14.42. Found: C, 74.24; H, 5.93; N, 14.39.

4-(2,4-Dimethylphenyl)-5-benzyl-3-vinyl-1,2,4-triazole (5g). White solid. Mp 111-113 °C; ^1H NMR: δ = 7.14-7.06 (m, 5H, Ar-*H*), 6.91-6.80 (m, 3H, Ar-*H*), 6.22-6.16 (m, 1H), 5.96-5.93 (m, 1H, Ar-*H*), 5.36-5.33 (m, 1H, =*CH*), 4.02-3.88 (m, 2H, =*CH*₂), 2.40 (s, 3H, CH₃), 1.51 (s, 3H, CH₃); ^{13}C NMR: δ = 153.9 (C3), 152.3 (C5), 140.0 (C), 135.6 (=CH), 135.1 (C), 131.7 (C), 129.5 (2 × CH), 128.7 (2 × CH), 128.0 (C), 127.8 (CH), 127.2 (CH), 126.7 (CH), 121.0 (CH), 120.8 (=CH₂), 31.2 (CH₂), 21.1 (CH₃), 16.5 (CH₃); EIMS: m/z (%) = 289 (M⁺); IR (KBr): ν =

3029, 2925, 1505, 1456, 1015, 940, 825, 728 cm^{-1} ; Anal. Calcd for $\text{C}_{19}\text{H}_{19}\text{N}_3$: C, 78.86; H, 6.62; N, 14.52. Found: C, 78.89; H, 6.66; N, 14.56.

4-(4-Chlorophenyl)-5-benzyl-3-vinyl-1,2,4-triazole (5h). White solid. Mp 115-116 $^{\circ}\text{C}$; ^1H NMR: δ = 7.40 (d, J = 8.4 Hz, 2H, Ar-*H*), 7.18-7.15 (m, 3H, Ar-*H*), 6.95-6.91 (m, 2H, Ar-*H*), 6.91 (d, J = 8.4 Hz, 2H, Ar-*H*), 6.23-6.18 (m, 1H, =CH), 6.12-6.07 (m, 1H, =CH₂), 5.46-5.43 (m, 1H, =CH₂), 4.03 (s, 2H, CH₂); ^{13}C NMR: δ = 153.8 (C3), 152.3 (C5), 135.7 (=CH), 135.2 (C), 131.8 (C), 129.7 (2 \times CH), 128.8 (2 \times CH), 128.4 (2 \times CH), 128.2 (C), 126.7 (2 \times CH), 122.1 (CH), 120.5 (=CH₂), 31.2 (CH₂); EIMS: m/z (%) = 295 (M^+); IR (KBr): ν = 3030, 1495, 1436, 1090, 946, 740, 567 cm^{-1} ; Anal. Calcd for $\text{C}_{17}\text{H}_{14}\text{ClN}_3$: C, 69.03; H, 4.77; N, 14.21. Found: C, 68.98; H, 4.81; N, 14.24.

5-Methyl-4-phenyl-3-vinyl-1,2,4-triazole (5i). White solid. Mp 156-158 $^{\circ}\text{C}$; ^1H NMR: δ = 7.61-7.55 (m, 3H, Ar-*H*), 7.25-7.22 (m, 2H, Ar-*H*), 6.31-6.23 (m, 1H, =CH), 6.13-6.09 (m, 1H, =CH₂), 5.47-5.44 (m, 1H, =CH₂), 2.30 (s, 3H, CH₃); ^{13}C NMR: δ = 152.2 (C3), 151.7 (C5), 133.8 (=CH), 130.1 (2 \times CH), 129.7 (C), 127.0 (2 \times CH), 121.4 (CH), 120.8 (=CH₂), 10.8 (CH₃); EIMS: m/z (%) = 185 (M^+); IR (KBr): ν = 3051, 1635, 1600, 1504, 1431, 942, 788, 699 cm^{-1} ; Anal. Calcd for $\text{C}_{11}\text{H}_{11}\text{N}_3$: C, 71.33; H, 5.99; N, 22.69. Found: C, 71.27; H, 6.03; N, 22.65.

5-Methyl-4-(3-methoxyphenyl)-3-vinyl-1,2,4-triazole (5j). White solid. Mp 146-148 $^{\circ}\text{C}$; ^1H NMR: δ = 7.49-7.45 (m, 1H, Ar-*H*), 7.09-7.05 (m, 1H, Ar-*H*), 6.81-6.80 (m, 1H, Ar-*H*), 6.75-6.73 (m, 1H, Ar-*H*), 6.30-6.25 (m, 1H, =CH), 6.15-6.09 (m, 1H, =CH₂), 5.45-5.43 (m, 1H, =CH₂), 3.85 (s, 3H, OCH₃), 2.32 (s, 3H, CH₃); ^{13}C NMR: δ = 160.6 (C), 152.0 (C3), 151.8 (C5), 134.8 (=CH), 130.8 (CH), 121.6 (CH), 121.0 (=CH₂), 119.3 (CH), 115.2 (C), 113.0 (CH), 55.5 (OCH₃), 10.9 (CH₃); EIMS: m/z (%) = 215 (M^+); IR (KBr): ν = 3055, 2931, 1605, 1495, 1460, 1430, 1280, 1221, 1023, 940, 841 cm^{-1} ; Anal. Calcd for $\text{C}_{12}\text{H}_{13}\text{N}_3\text{O}$: C, 66.96; H, 6.09; N, 19.52. Found: C, 66.91; H, 6.13; N, 19.55.

5-Methyl-4-(4-chlorophenyl)-3-vinyl-1,2,4-triazole (5k). White solid. Mp 166-168 $^{\circ}\text{C}$; ^1H NMR: δ = 7.52 (d, J = 8.1 Hz, 2H, Ar-*H*), 7.22 (d, J = 8.1 Hz, 2H, Ar-*H*), 6.24-6.19 (m, 1H, =CH), 6.09-6.05 (m, 1H, =CH₂), 6.44-6.41 (m, 1H, =CH₂), 2.27 (s, 3H, CH₃); ^{13}C NMR: δ = 152.0 (C3), 151.7 (C5), 136.0 (=CH), 132.1 (2 \times CH), 130.2 (C), 128.2 (2 \times CH), 121.7 (=CH₂), 120.4 (CH), 11.0 (CH₃); EIMS: m/z (%) = 219 (M^+); IR (KBr): ν = 3056, 1525, 1498, 1430, 1091, 1015, 936, 758 cm^{-1} ; Anal. Calcd for $\text{C}_{11}\text{H}_{10}\text{ClN}_3$: C, 60.14; H, 4.59; N, 19.13. Found: C, 60.18; H, 4.63; N, 19.16.

5-Methyl-4-(1-naphthalenyl)-3-vinyl-1,2,4-triazole (5l). Pale yellow solid. Mp 141-143 $^{\circ}\text{C}$; ^1H NMR: δ = 8.07-8.01 (m, 2H, Ar-*H*), 7.64-7.60 (m, 2H, Ar-*H*), 7.56-7.53 (m, 1H, Ar-*H*), 7.41-7.23 (m, 2H, Ar-*H*), 6.15-6.09 (m, 1H, =CH), 6.05-6.01 (m, 1H, =CH₂), 5.35-5.32 (m, 1H, =CH₂), 2.23 (s, 3H, CH₃); ^{13}C NMR: δ = 152.8 (C3), 152.6 (C5), 134.5 (=CH), 130.6 (C), 130.1 (C), 129.7 (C), 128.7 (CH), 128.4 (CH), 127.4 (CH), 125.8 (CH), 125.4 (CH), 121.6 (CH), 121.4 (CH), 120.8 (=CH₂), 10.9 (CH₃); EIMS: m/z (%) = 235 (M^+); IR (KBr): ν = 3420, 3060, 2925, 1600, 1520, 1470, 1071, 958, 812, 780 cm^{-1} ; Anal. Calcd for $\text{C}_{15}\text{H}_{13}\text{N}_3$: C, 76.57; H, 5.57; N, 17.86. Found: C, 76.60; H, 5.62; N, 17.82.

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