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Solid-Phase Organic Synthesis of Vinyl-Substituted 1,3,4-Oxadiazoles Using Polymer-Bound α-Selenopropionic Acid

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Abstract: Vinyl-substituted 1,3,4-oxadiazoles can be efficiently synthesized through acylation, cyclocondensation, and oxidation–elimination reaction from polystyrene-supported α-selenopropionic acid and acid hydrazides. This new solid-phase organic synthesis method could provide the target compounds in good yield and purity, with advantages of decreased volatility and simplification of workup procedure.

Keywords: Polystyrene-supported α-selenopropionic acid, solid-phase organic synthesis, vinyl-substituted 1,3,4-oxadiazole

Heterocycles are important elements in many pharmacologically active compounds. Combinatorial synthesis of the heterocycle libraries is an effective way for the development of drug candidates, and solid-phase organic synthesis (SPOS) is arguably the most important method for the high-speed synthesis of heterocycles in combinatorial synthesis.[1] 1,3,4-Oxadiazoles constitute a class of heterocycles that has attracted significant interest in medicinal and pesticide chemistry[2,3] and polymer

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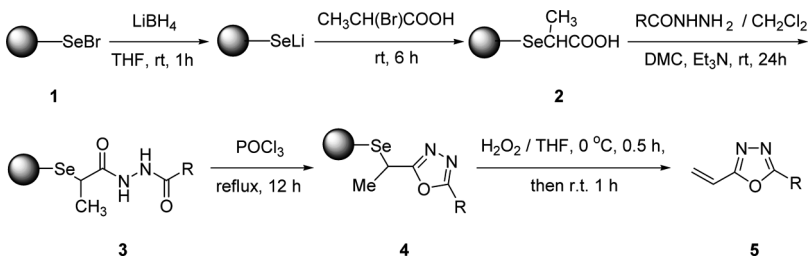
and material science.[4,5] Although solution-phase[6] or solid-phase[7] synthetic methods for 1,3,4-oxadiazoles are well documented, efforts are continuing for the development of more efficient methods with experimental simplicity. Among the 1,3,4-oxadiazoles, vinyl-substituted derivatives have now attracted considerable attention because of their biological properties,[8] such as antiallergic, antibacterial, and anti-HIV activity. In addition, vinyl-substituted 1,3,4-oxadiazoles are versatile intermediates for the preparation of complex natural products and play an important role as useful additives in material chemistry.[9] However, in view of the fact that vinyl-substituted heterocycles are easily polymerized because of their highly reactive terminal double bond connected directly with an electron-withdrawing heterocyclic group,[10] the incorporation of vinyl substituents into heterocycles is seldom reported.[11] It is well known that phenylseleno group is readily converted to a leaving group, giving access to carbon–carbon double bond via oxidation followed by β-elimination under extremely mild conditions.[12] Moreover, the polymeric selenium reagents[13] have been now developed for SPOS with a combined advantage of decrease volatility and simplification of product workup. In continuation of our interest in solid-phase organoselenium chemistry,[14] we sought to develop a convenient and efficient preparation of vinyl-substituted 1,3,4-oxadiazoles from polystyrene-supported α-selenopropionic acid (Scheme 1).

Polymer-supported α-selenopropionic acid **2** was prepared by treatment of a THF-swollen suspension of cross-linked (1%) polystyrene-bound selenium bromide **1**[13a] with $LiBH_4$, followed by treatment with 2-bromopropionic acid according to our previous method.[14a] The minimum loading of COOH of resin **2** verified by their Fourier transform infrared (FT-IR) spectra showing a strong carbonyl absorption at 1726 cm^{-1} was determined by acid–base titration[15] 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



Scheme 1. Solid-phase synthesis of the route of vinyl-substituted 1,3,4-oxadiazoles.

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 this 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}. It has been reported that in the presence of DMC (2-chloro-1,3-dimethylimidazolinium chloride), 1,3,4-oxadiazoles can be obtained in good yields from carboxylic acids and acid hydrazides in CH_2Cl_2 at ambient temperature.[16] Interestingly, when DMC 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 (Fourier transform infrared) 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 cyclodehydration of diacyl hydrazide resin **3a** treated with a variety of dehydrating agents, such as thionyl chloride, phosphorous oxychloride, phosphorous pentoxide, triphenylphosphine, and trifluoromethane-sulfonic anhydride, was performed in our hands. After a series of experiments, the cyclocondensation reaction of resin **3** in the presence of phosphorus oxychloride was carried out efficiently according to the reported method[11] to furnish resin **4**, followed by selenoxide syn elimination with excess of 30% hydrogen peroxide to afford the corresponding vinyl-substituted 1,3,4-oxadiazoles **5** in moderate to good yields (74–81%) and with good purities (90–95%) of crude materials in all cases (Table 1). The residual resin, polystyrene-supported phenylseleninic acid, was obtained as a by-product whose

Table 1. Yields and purities of vinyl-substituted 1,3,4-oxadiazoles **5**

Entry	R	Products	Yield (%)[a]	Purity (%)[b]
1	C_6H_5	**5a**	80	95
2	*p*-$CH_3OC_6H_4$	**5b**	81	94
3	*p*-ClC_6H_4	**5c**	78	93
4	*p*-$NO_2C_6H_4$	**5d**	76	91
5	$C_6H_5CH_2$	**5e**	77	92
6	*n*-C_6H_{13}	**5f**	75	92
7	*i*-C_3H_7	**5g**	74	90

[a]Overall yields based on polystyrene-supported α-selenopropionic acid **2** (1.20 mmol COOH/g).

[b]Determined by HPLC (high-performance liquid chromatography) of crude cleavage product ($\lambda = 254$ nm).

infrared data was identical to the previously reported data[17] and showed no residual C=N and O–N group absorptions, indicating the oxidation–elimination was complete.

In summary, a novel and efficient procedure for the solid-phase synthesis of vinyl-substituted 1,3,4-oxadiazoles in moderate to good yields and with good purities using polymer-supported α-selenopropionic acid with advantages of decreased volatility and simplification of product workup has been developed.

EXPERIMENTAL

Melting points were determined on X_4 melting-point apparatus and are uncorrected. ^{1}H NMR (400-MHz) and ^{13}C NMR (100-MHz) spectra were recorded on a Bruker Avance (400-MHz) spectrometer using $CDCl_3$ 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, 70 eV) were recorded on a HP5989B mass spectrometer. HPLC analysis was carried out on Agilent 1100 (250×4.6 mm C_{18} column, gradient elution 50/20/30 THF/CH_3OH/H_2O (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 bromide[13a] and other starting materials were purchased from commercial suppliers and used without further purification. CH_2Cl_2 was distilled from phosphorous pentoxide, and THF was distilled from sodium-benzophenone immediately prior to use.

General Procedure for the Preparation of 5-Vinyl-1,3,4-oxadiazoles (5a–5g)

Resin **2** (0.84 g, 1.0 mmol) was swelled in anhydrous CH_2Cl_2 (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) were 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 H_2O, THF, MeOH, and CH_2Cl_2 (2×5 mL of each) and then dried under vacuum to afford resin **3**. Then the resin **3** and phosphorus oxychloride (25 mL) were refluxed for 12 h under a nitrogen atmosphere. After completion of the reaction, the mixture was cooled to room temperature, and the resin **4** was collected by filtration and washed successively with DMF (1×10 mL), DMSO (1×10 mL),

THF (1×10 mL), THF/H_2O (1:1) (2×10 mL), H_2O (2×10 mL), and CH_2Cl_2 (2×10 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 CH_2Cl_2 (3×10 mL). The filtrate was washed with H_2O (2×20 mL), dried over $MgSO_4$, and evaporated to dryness under vacuum to obtain the crude products **5**. Further purification was via flash chromatography with EtOAc/*n*-hexane (1/2-1/1 v/v) as the eluent for ^{1}H NMR, ^{13}C NMR, and microanalysis.

Data

5-Phenyl-2-vinyl-1,3,4-oxadiazole (**5a**)

Colorless oil (lit.[11] oil); ^{1}H NMR: $\delta = 8.11$–8.08 (m, 2 H), 7.56–7.50 (m, 3 H), 6.82–6.78 (m, 1 H), 6.40–5.95 (m, 2 H); ^{13}C NMR: $\delta = 164.5$, 163.4, 131.9, 128.9, 127.1, 124.8, 123.8, 120.2; IR (film): $\nu = 3069$, 2925, 1626, 1550, 1535, 1460, 980, 730, 705 cm^{-1}; EIMS: m/z (%) = 172 (M^+). Anal. calcd. for $C_{10}H_8N_2O$: C, 69.76; H, 4.68; N, 16.27. Found: C, 69.70; H, 4.73; N, 16.31.

5-(p-Methoxyphenyl)-2-vinyl-1,3,4-oxadiazole (**5b**)

White solid, mp 80–81 °C (lit.[11] mp 80–82 °C); ^{1}H NMR: $\delta = 7.85$ (d, $J = 8.8$ Hz, 2 H), 6.95 (d, $J = 8.8$ Hz, 2 H), 6.65–6.60 (m, 1 H), 6.20–5.71 (m, 2 H), 3.74 (s, 3 H); ^{13}C NMR: $\delta = 163.5$, 162.5, 161.7, 128.3, 124.1, 119.4, 115.5, 114.2, 55.1; IR (KBr): $\nu = 3065$, 2986, 2845, 1645, 1530, 1368, 1270, 1086, 1020, 940, 695 cm^{-1}; EIMS: m/z (%) = 202 (M^+). Anal. calcd. for $C_{11}H_{10}N_2O_2$: C, 65.34; H, 4.98; N, 13.85. Found: C, 65.29; H, 5.05; N, 13.80.

5-(p-Chlorophenyl)-2-vinyl-1,3,4-oxadiazole (**5c**)

White solid, mp 88–90 °C; ^{1}H NMR: $\delta = 7.89$ (d, $J = 8.5$ Hz, 2H), 7.02 (d, $J = 8.5$ Hz, 2H), 6.73–6.68 (m, 1H), 6.21–6.04 (m, 2H); ^{13}C NMR: $\delta = 164.6$, 163.9, 137.3, 129.0, 128.7, 128.3, 124.8, 120.2; IR (KBr): $\nu = 3060$, 2925, 1620, 1550, 1470, 1412, 1093, 980, 828, 790, 500 cm^{-1}; EIMS: m/z (%) = 206 (M^+). Anal. calcd. for $C_{10}H_7ClN_2O$: C, 58.13; H, 3.41; N, 13.56. Found: C, 58.07; H, 3.46; N, 13.61.

5-(p-Nitrophenyl)-2-vinyl-1,3,4-oxadiazole (**5d**)

Light yellow solid, mp 135–137 °C; ^{1}H NMR: $\delta = 8.05$ (d, $J = 8.7$ Hz, 2 H), 7.12 (d, $J = 8.7$ Hz, 2 H), 6.78–6.71 (m, 1 H), 6.23–6.11 (m, 2 H); ^{13}C NMR: $\delta = 165.2$, 164.3, 145.5, 135.8, 128.7, 125.1, 123.3, 120.5; IR (KBr): $\nu = 3060$, 2961, 1618, 1595, 1528, 1470, 1412, 1350, 1225, 1098, 1028, 860, 836, 727, 610 cm^{-1}; EIMS: m/z (%) = 217 (M^+). Anal. calcd. for $C_{10}H_7N_3O_3$: C, 55.30; H, 3.25; N, 19.35. Found: C, 55.34; H, 3.30; N, 19.41.

5-Benzyl-2-vinyl-1,3,4-oxadiazole (**5e**)

Colorless oil; ^{1}H NMR: $\delta = 7.37$–7.32 (m, 2 H), 7.27–7.24 (m, 3 H), 6.68–6.61 (m, 1 H), 6.05–5.87 (m, 2 H), 4.20 (s, 2 H); ^{13}C NMR: $\delta = 164.5$, 163.8, 133.2, 128.8, 128.6, 127.1, 124.5, 120.1, 31.6; IR (film): $\nu = 3033$, 2925, 1567, 1528, 1496, 1031, 982, 945, 731, 696 cm^{-1}; EIMS: m/z (%) = 186 (M^+). Anal. calcd. for $C_{11}H_{10}N_2O$: C, 70.95; H, 5.41; N, 15.04. Found: C, 70.91; H, 5.46; N, 15.08.

5-(n-Hexyl)-2-vinyl-1,3,4-oxadiazole (**5f**)

Colorless oil (lit.[11] oil); ^{1}H NMR: $\delta = 6.65$–6.58 (m, 1H), 6.12–5.88 (m, 2 H), 2.79–2.74 (m, 2 H), 1.76–1.70 (m, 2 H), 1.35–1.20 (m, 6 H), 0.83 (t, $J = 7.1$ Hz, 3 H); ^{13}C NMR: $\delta = 166.5$, 163.8, 124.5, 120.2, 31.2, 28.9, 26.5, 25.3, 22.6, 13.9; IR (film): $\nu = 2958$, 2930, 2872, 1572, 1530, 1380, 1030, 981, 942, 765 cm^{-1}; EIMS: m/z (%) = 180 (M^+). Anal. calcd. for $C_{10}H_{16}N_2O$: C, 66.63; H, 8.95; N, 15.54. Found: C, 66.68; H, 9.01; N, 15.58.

5-(i-Propyl)-2-vinyl-1,3,4-oxadiazole (**5g**)

Colorless oil (lit.[11] oil); ^{1}H NMR: $\delta = 6.65$–6.58 (m, 1 H), 6.22–6.08 (m, 1 H), 5.79–5.70 (m, 1 H), 3.15–3.08 (m, 1 H), 1.33 (s, 3 H), 1.31 (s, 3 H); ^{13}C NMR: $\delta = 170.1$, 163.5, 124.0, 120.1, 26.2, 19.9; IR (film): $\nu = 2978$, 2938, 2879, 1565, 1387, 1370, 1012, 980, 942 cm^{-1}; EIMS: m/z (%) = 138 (M^+). Anal. calcd. for $C_7H_{10}N_2O$: C, 60.85; H, 7.30; N, 20.28. Found: C, 60.90; H, 7.36; N, 20.32.

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