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Preparation of polystyrene-supported vinyl sulfone and its application in the solid-phase organic synthesis of 1-monosubstituted 1,2,3-triazoles

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ABSTRACT

A polystyrene-supported vinyl sulfone reagent was prepared and used for the solid-phase organic synthesis of 1-monosubstituted 1,2,3-triazoles via a 1,3-dipolar cycloaddition reaction with azides and subsequent cleavage from the polymer support through an elimination reaction in the presence of potassium *tert*-butoxide. The advantages of this method include straightforward operation, good yield and high purity of the crude products.

1. Introduction

Solid-phase organic synthesis (SPOS) is a method in which molecules are bound to an insoluble solid support, such as polystyrene resin, and are synthesized step-by-step in a reactant solution. Compared with normal synthesis performed in a liquid state, the removal of excess reactant or byproduct from the product is easier, which allows rapid product purification and allows a given reaction to be driven to completion through the use of an excess of reagents [1–3]. SPOS has become a powerful technique for the rapid generation of a large number of structurally diverse compounds and for the discovery of new active molecules [4–6]. Heterocyclic compounds have received special attention because of their broad range of biological activities. As a result, an increasing number of pharmaceutically useful heterocyclic compounds have been prepared using solid-phase methodologies [7–9]. Triazole moieties are found in various biologically active compounds because they are readily transformed into various biodynamic agents, including those with antithrombotic, PAF antagonist, and hypolipidemic properties [10–12]. Because of the importance of triazole, many practical synthetic methods have been utilized for their preparation [13–21]. However, the literature contains only a few reports concerning synthetic methods for 1-monosubstituted 1,2,3-triazoles [22–30]. Sulfinate-functionalized resins have been efficiently prepared and utilized in SPOS, and the resulting sulfone linker has been found to be both robust and versatile [9]. Previous and recent reports from Kurth and co-workers [31,32], Lam's research group [33,34], other laboratories [35–40] and ours [41,42] have detailed sulfone-linking strategies for SPOS methods to explore sulfone-based chemical transformations. As a part of our ongoing research program focused on the use of a versatile traceless sulfone linker in SPOS, we herein have explored an efficient solid-phase synthetic methodology for the preparation of 1-monosubstituted 1,2,3-triazoles. To our knowledge, this method has not been previously reported.

2. Experimental

2.1. General

Melting points were determined on an X_4 melting point apparatus and are uncorrected. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AVANCE 400 NMR spectrometer, using $CDCl_3$ as the solvent and tetramethylsilane (TMS) as an internal standard. FTIR spectra were collected on a Perkin–Elmer SP One FTIR spectrophotometer. Microanalyses were performed with a Carlo Erba 1106 Elemental Analyzer. High-performance liquid chromatography (HPLC) analysis was performed on an Agilent 1100 automated system equipped with a photodiode array (PDA) detector (λ_{max} = 254 - nm used for this study); a gradient of acetonitrile–H_2O was used on a RP-18e column (150 × 4.6 mm^2). Three kinds of polystyrene/1% divinylbenzene sodium sulfinates (**1**) (loading: 2.10, 1.80 and 1.25 mmol – SO_2Na/g, respectively) were purchased from Tianjin Nankai Hecheng Science and Technology Co. (100–200 mesh).

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Azides [43] were prepared according to procedures reported in the literature. The other chemicals were obtained from commercial suppliers (Aldrich, USA and Shanghai Chemical Company, China) and used without purification. All organic solvents were dried using standard methods.

2.2. Preparation of polystyrene-supported 2-phenylsulfonylethanol (**2**)

Resin **1** (2.0 g, 3.6 mmol) was swollen in a mixture of DMF (10 mL) and THF (20 mL) by gently shaking the resin–DMF–THF mixture at room temperature for 30 min. 2-Chloroethanol (2.4 mL), tetrabutylammonium iodide (0.3 g), and potassium iodide (1.8 g) were added to this mixture and the resulting reaction mixture was shaken at 80 °C under a nitrogen atmosphere for 24 h. After filtration, the resin was washed successively with DMF, H_2O, CH_3OH and ether (2 × 10 mL of each); and then dried under reduced pressure over phosphorous pentoxide at 50–60 °C to afford resin **2** as yellow beads.

For analytical purposes, the 3,5-dinitrobenzoate of resin **2** was prepared according to the published method [44] by the reaction of resin **2** (0.5 g) with 3,5-dinitrobenzoyl chloride (0.5 g) in dry pyridine (10 mL). After being stirred at 80 °C for 1.5 h, the resin was collected on a filter, washed successively with THF, H_2O, CH_3OH and ether (2 × 10 mL of each), and dried as previously described to yield the corresponding 3,5-dinitrobenzoate resin (0.64 g) that exhibited a strong carbonyl absorption at 1732 cm^{-1} in its FTIR spectrum.

2.3. Preparation of polystyrene-supported vinyl sulfone (**3**)

Resin **2** (2.0 g) was swollen in THF (30 mL) at 0 °C, and acetic anhydride (1.3 g, 13.0 mmol) and DBU (3.0 g, 20.0 mmol) were added. The reaction was shaken at room temperature for 12 h, and the resin was subsequently collected by filtration, washed with H_2O (2 × 15 mL), THF/H_2O (1:1, 2 × 10 mL), THF (2 × 10 mL), and ether (2 × 10 mL), and dried in a vacuum overnight to afford polymer **3** as yellow beads: IR (KBr): ν = 2924, 2855, 1600, 1490, 1450, 1377, 1315, 1147 cm^{-1}.

Polymeric **3** was reacted with 5-ethoxy-l,2,3,4-terthydroisoquinoline in accordance with the reported method [45] was subsequently treated with MeI and diisopropylcthylamine successively to release *N*-methyl-5-ethoxy-l,2,3,4-terthydroisoquinoline; the vinyl functional loading of polymeric **3** based on the amount of *N*-methyl-5-ethoxy-l,2,3,4-terthydroisoquinoline was calculated to be 1.67 mmol/g.

2.4. General procedure for the preparation of 1-monosubstituted 1,2,3-triazoles (**5a**–**5j**)

Azide (3.0 mmol), $CuSO_4{\cdot}5H_2O$ (18.5 mg, 0.05 mmol) and sodium ascorbate (50 mg, 0.25 mmol) were added to the suspension of resin **3** (1.67 mmol, 1.0 g) pre-swollen in CH_2Cl_2/H_2O (1:1, 20 mL), and then shaken at room temperature for 12 h. After which the resin was collected by filtration, washed with H_2O (2 × 15 mL), THF/H_2O (1:1, 2 × 10 mL), THF (2 × 10 mL), CH_2Cl_2 (2 × 10 mL) and ether (2 × 10 mL), and dried in a vacuum overnight, affording yellow resin **4**. Subsequently, resin **4** was swollen in THF (15 mL) for 1 h. Then potassium *tert*-butoxide (0.4 g, 3.5 mmol) was added to the suspension at 0 °C. The reaction mixture was stirred at the same temperature for 1 h, and then was warmed to room temperature and shaken for 12 h. After which the resin was filtered and washed with THF (2 × 10 mL), THF/H_2O (1/1, 2 × 10 mL), and H_2O (2 × 10 mL). The combined filtrate and washings were concentrated to remove organic solvent, and the residue was extracted with EtOAc, dried over anhydrous magnesium sulfate, and then concentrated under reduced pressure to give crude products **5** with 93–97% purity determined by HPLC, which were further purified by flash silica gel column chromatography eluted with ethyl acetate/hexane to provide pure products **5a**–**5j** for their structure analyses.

2.4.1. 1-Phenyl-1H-1,2,3-triazole (**5a**)

Light yellow solid, mp 51–52 °C (52–53 °C [25]); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.01 (d, J = 1.0 Hz, 1H), 7.85 (d, J = 1.0 Hz, 1H), 7.77–7.74 (m, 2H), 7.56–7.53 (m, 2H), 7.46–7.44 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 137.6, 135.5, 130.1, 128.9, 122.7, 121.1.

2.4.2. 1-(4-Methylphenyl)-1H-1,2,3-triazole (**5b**)

White solid, mp 84–85 °C (86 °C [25]); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.98 (d, J = 0.9 Hz, 1H), 7.84 (d, J = 0.9 Hz, 1H), 7.62 (d, J = 8.0 Hz, 2H), 7.30 (d, J = 8.0 Hz, 2H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 138.5, 134.4, 133.9, 130.1, 121.5, 120.3, 20.9.

2.4.3. 1-(4-Methoxyphenyl)-1H-1,2,3-triazole (**5c**)

White solid, mp 77–79 °C (78–80 °C [25]); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.92 (d, J = 0.9 Hz, 1H), 7.84 (d, J = 0.9 Hz, 1H), 7.65 (d, J = 6.8 Hz, 2H), 7.04 (d, J = 6.8 Hz, 2H), 3.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 160.2, 135.6, 131.0, 122.9, 122.5, 115.0, 55.8.

2.4.4. 1-(4-Chlorophenyl)-1H-1,2,3-triazole (**5d**)

Light yellow solid, mp 110–112 °C (111–113 °C [25]); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.99 (s, 1H), 7.86 (s, 1H), 7.71 (d, J = 8.8 Hz, 2H), 7.51 (d, J = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 138.7, 134.6, 133.9, 130.0, 121.5, 120.2.

2.4.5. 1-(2-Chlorophenyl)-1H-1,2,3-triazole (**5e**)

Yellow oil (Yellow oil [26]); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.05 (s, 1H), 7.86 (s, 1H), 7.62–7.57 (m, 2H), 7.47–7.45 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 134.6, 133.1, 130.5, 130.3, 128.5, 127.6, 127.3, 125.2.

2.4.6. 1-(4-Fluorophenyl)-1H-1,2,3-triazole (**5f**)

White solid, mp 63–65 °C ([26]); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.94 (d, J = 1.0 Hz, 1H), 7.84 (d, J = 1.0 Hz, 1H), 7.72 (t, $^4J_{HF}$ = 6.8 - Hz, 2H), 7.24 (t, $^3J_{HF}$ = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 163.2 ($^1J_{CF}$ = 986 Hz), 135.4, 128.5 ($^4J_{CF}$ = 13.2 Hz), 124.7 ($^3J_{CF}$ = 33.2 Hz), 121.0, 117.2 ($^2J_{CF}$ = 86.4 Hz).

2.4.7. 1-(4-Nitrophenyl)-1H-1,2,3-triazole (**5g**)

Yellow solid, mp 202–204 °C (202–205 °C [25]); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.44 (d, J = 8.8 Hz, 2H), 8.14 (s, 1H), 8.01 (d, J = 8.8 Hz, 2H), 7.93 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 147.5, 141.1, 135.4, 125.6, 121.6, 120.5.

2.4.8. 1-(3-Nitrophenyl)-1H-1,2,3-triazole (**5h**)

Light yellow solid, mp 114–115 °C (114–116 °C [25]); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.63 (t, J = 2.0 Hz, 1H), 8.34–8.31 (m, 1H), 8.22–8.20 (m, 1H), 8.12 (d, J = 1.0 Hz, 1 H), 7.92 (d, J = 1.0 Hz, 1H), 7.78 (t, J = 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 148.7, 137.8, 135.0, 130.9, 126.2, 123.4, 120.1, 115.5.

2.4.9. (E)-1-Cinnamyl-1H-1,2,3-triazole (**5i**)

Light yellow solid, mp 45–47 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.92 (d, J = 1.0 Hz, 1H), 7.85 (t, J = 14.8 Hz, 1H), 7.43–7.25 (m, 5H), 6.66 (d, J = 15.8 Hz, 1H), 6.34 (dt, J = 15.8, 6.6 Hz, 1H), 5.11 (dd, J = 6.6, 1.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 146.9, 135.5, 134.8, 128.7, 128.5, 126.6, 122.0, 119.2, 52.0. IR (KBr): ν = 3060, 2922, 2854, 1595, 1489, 1445, 1137, 1214, 1052, 961, 773, 747, 690 cm^{-1}. Anal. Calcd. for $C_{11}H_{11}N_3$: C, 71.33; H, 5.98; N, 22.69. Found: C, 71.26; H, 5.90; N, 22.61.

*2.4.10. 1-Benzyl-1H-1,2,3-triazole (**5j**)*

White solid, mp 51–52 °C (51–53 °C [25]); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.72 (s, 1H), 7.47 (s, 1H), 7.40–7.38 (m, 3H), 7.28–7.26 (m, 2H), 5.57 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 134.7, 134.2, 129.0, 128.6, 127.7, 123.5, 53.8.

3. Results and discussion

Initially, the procedure for the preparation of polystyrene-bound vinyl sulfone **3** was investigated. As shown in Scheme 1, in our preliminary experiments, polystyrene/1% divinylbenzene sodium sulfinate **1** (1.80 mmol – SO_2Na/g) was selected to react with 2-chloroethanol in DMF–THF (1/2) in the presence of tetrabutylammonium iodide (TBAI) and potassium iodide to afford polystyrene-bound 2-phenylsulfonylethanol **2**, which was amenable to FTIR monitoring for the appearance of the expected a large hydroxyl stretch at 3500 cm^{-1}, as well as the characteristic sulfone absorption bands at 1317 and 1119 cm^{-1}. Encouraged by this positive result, the other two sodium sulfinate resins with loading of 2.10 and 1.25 mmol–SO_2Na/g were further evaluated, respectively. After several experiments, it was found that quite similar result was obtained under the same reaction conditions with higher loading resin **1**, but it is expensive relatively comparing with the resin **1** with 1.80 mmol/g loading. When using lower loading resin **1**, its lower activity was observed, the similar reaction generated the desired product in a lower yield with longer reaction time. The hydroxyl group attachment on the resin **2** was calculated to be 1.65 mmol–CH_2CH_2OH/g from the nitrogen elemental analysis of its corresponding 3,5-dinitrobenzoate ester intermediate prepared by treatment of **2** with 3,5-dinitrobenzoyl chloride according to the method described in literature [44]. Polystyrene-bound vinyl sulfone **3** was then obtained in near quantitative yield from 2-hydroxy sulfone resin **2** by acetylation followed by in situ elimination in the presence of 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU). It should be noted that reaction **2** → **3** was not amenable to FTIR analysis, nevertheless, the complete conversion of the hydroxy group of resin **2**–**3** was further confirmed by treating **3** with acetyl chloride/triethylamine: no acetoxy absorption band was observed in the FTIR spectrum of the product. On the other hand, the loading of vinyl functional group of resin **3** was determined to be 1.67 mmol/g following the published protocol [45].

Secondly, the construction of the triazolyl heterocycles using polystyrene-bound vinyl sulfone **3** was further examined. As illustrated in Scheme 2, azides were also found to undergo 1,3-dipolar cycloadditions with resin **3** smoothly in the presence of $CuSO_4{\cdot}5H_2O$ and sodium ascorbate in CH_2Cl_2/H_2O (1/1) with the solution-phase method established [46] at room temperature to form resin **4**. Since this transformation exhibited no reliably diagnostic absorption peaks in the FTIR spectra, the elimination release step was undertaken. After a number of basic (Et_3N, DBU and *t*-BuOK) elimination conditions were evaluated, and optimal results were obtained by treating resin **4** with potassium *tert*-butoxide in THF at room temperature for 12 h to afford 1-monosubstituted 1,2,3-triazoles **5a**–**5j**. The results are summarized in Table 1. As can be observed in Table 1, aryl azides carrying either an electron donating substituent, such as methyl and methoxy (Table 1, entries 2 and 3) or an electron with drawing group including halogens (Table 1, entries 4–6) and nitro (Table 1, entries 7 and 8) could perform efficiently with good to excellent yields. However, aryl azides bearing an *ortho*-substituent (e.g., the chloro group) (Table 1, entry 5) showed a slight decrease in yield (81%) due to the steric hindrance. For other substrates such as (*E*)-cinnamyl azide and benzyl azide, higher yields were obtained (Table 1, entries 9 and 10). It should be noted that, besides of good isolated yields of the target compounds **5a**–**5j** (Table 1), HPLC analysis of the crude products cleaved from the resin directly indicated their high purities in all cases (93–97%). The impurities included in the cleavage products may be generated from the possible side reactions or the residual trace reactants embedded in the resins in the reactions.

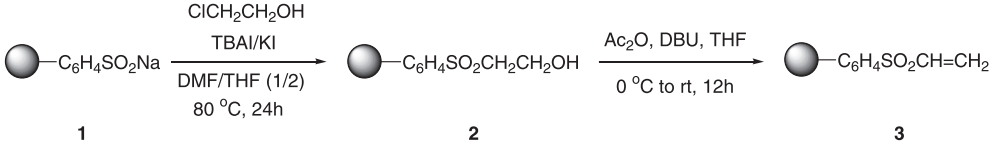
Table 1
Yields and purities of 1-monosubstituted 1,2,3-triazoles (**5a–5j**).

Entry	R	Product	Yield[a] (%)	Purity[b] (%)
1	C_6H_5	**5a**	93	97
2	4-$CH_3C_6H_4$	**5b**	93	96
3	4-$CH_3OC_6H_4$	**5c**	95	97
4	4-ClC_6H_4	**5d**	87	95
5	2-ClC_6H_4	**5e**	81	93
6	4-FC_6H_4	**5f**	90	97
7	4-$NO_2C_6H_4$	**5g**	85	94
8	3-$NO_2C_6H_4$	**5h**	88	95
9	(*E*)-$C_6H_5CH{=}CHCH_2$	**5i**	89	96
10	$C_6H_5CH_2$	**5j**	92	96

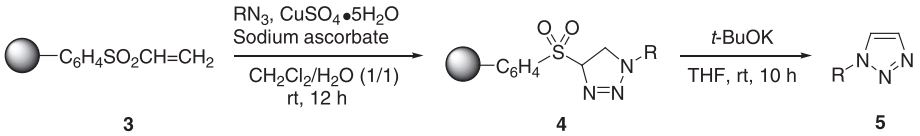
[a] Overall isolated yields based on polystyrene-supported vinyl sulfone **3**.
[b] Determined by HPLC of crude cleavage product.

4. Conclusions

We have developed a facile protocol for the traceless solid-phase synthesis of 1-monosubstituted 1,2,3-triazoles by 1,3-dipolar cycloaddition reaction of polystyrene-bound vinyl sulfone with azides, and subsequent elimination reaction. This method provides a novel access to 1-monosubstituted aryl-, alkyl- and vinyl-1,2,3-triazoles respectively in good yields and purities with simplification of product work-up, which are important heterocycle compounds in medicinal chemistry and materials science. Further studies on the application of the present methodology to the synthesis of biological active compounds are proceeding.



Scheme 1. Preparation of polystyrene-bound vinyl sulfone **3.**



Scheme 2. SOPS of 1-monosubstituted 1,2,3-triazoles **5a–5j**.

Acknowledgments

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