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SYNTHESIS AND APPLICATION OF 1,2,3-TRIAZOLE ALLYL ACETATES: EXPEDIENT ACCESS TO PYRIDINE DERIVATIVES

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Abstract – A series of novel 1,2,3-triazole allyl acetates were prepared by CuTC-catalyzed azide-alkyne cycloaddition (CuAAC) from 3-acyloxy-1,4-enynes and sulfonyl azides. The 1,2,3-triazole allyl acetates could be further transformed to pyridine derivatives through 6π-azaelectrocyclization followed by elimination.

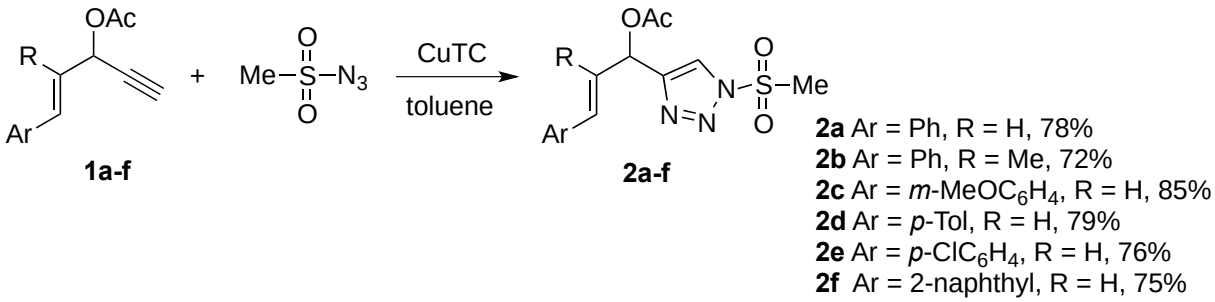
The reaction between azides and alkynes is belong to Huisgen 1,3-dipolar cycloaddition, which is one of the important methods to access 1,2,3-triazole.[1] The azide-alkyne cycloaddition (AAC) could be occurred without or with catalysts. In 2001, Medal group[2] and Sharpless group[3] reported copper-catalyzed AAC reaction independently. Since then copper-catalyzed azide-alkyne cycloaddition (CuAAC) was booming and focused by many chemists. The concept of "Click Chemistry" was proposed due to the excellent performance of CuAAC reaction in different fields.[4] Recently, ruthenium[5]- or iridium[6]-catalyzed AAC reactions were also accomplished, which further diversified the libraries of 1,2,3-triazoles.[7]

However, when sulfonyl azides were used instead of aryl or alkyl azides in CuAAC reaction, the 1-sulfonyl-1,2,3-triazoles were not stable because of the strong electron-withdrawing sulfonyl group. Treating the 1-sulfonyl-1,2,3-triazole with inorganic or organic base, *N*-sulfonyl ketenimine was formed after releasing the nitrogen derived from triazole.[8] Many strategies have emerged to prepare stable 1-sulfonyl-1,2,3-triazoles, such as 2,6-lutidine as base,[9] copper(I)-thiophene-2-carboxylate (CuTC) as copper source[10] or the combination of $Cu(OAc)_2 \cdot H_2O$ and 2-aminophenol as catalysts.[11]

Although the synthetic protocol of 1-sulfonyl-1,2,3-triazoles is well documented, the efficient and convenient preparation of allyl substituted 1-sulfonyl-1,2,3-triazoles, especially allylic acyloxy substituted compounds, remains unexplored. It shows a potential and elegant annulation pathway for the synthesis of pyridines. According to Davies, Murakami and Fokin's seminar work, rhodium (II) as catalyst could open the triazole ring of 1-sulfonyl-1,2,3-triazoles to generate Rh(II) carbene species.[12] The allylic acyloxy group could attack the Rh(II) carbene species to form dienimines by 1,2-acyloxy

migration.[13] Pyridines could be afforded from dienimines *via* 6π-azaelectrocyclization followed by elimination. Based on our previous study,[14] herein we reported a CuTC-catalyzed azide-alkyne cycloaddition from 3-acyloxy-1,4-enynes and sulfonyl azides to access various 1,2,3-triazole allyl acetates, which could be further converted to pyridine derivatives.

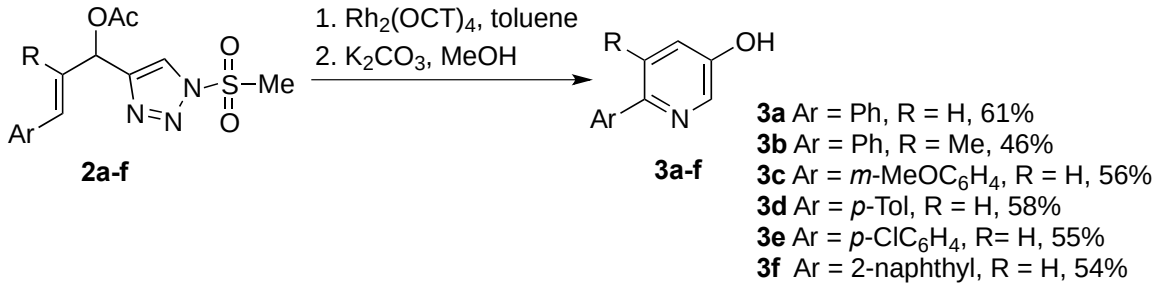
The synthetic route of 1,2,3-triazole allyl acetates was shown in Scheme 1. Treatment of 3-acyloxy-1,4-enynes (**1a-f**) with methanesulfonyl azide and CuTC was conducted in toluene at ambient temperature to give corresponding substituted 1,2,3-triazole allyl acetates (**2a-f**) in good yields. (*E*)-1-Phenylpent-1-en-4-yn-3-yl acetate (**1a**) was first selected as substrate for the examination. (*E*)-1-(1-(Methylsulfonyl)-1*H*-1,2,3-triazol-4-yl)-3-phenylallyl acetate (**2a)** was acquired in 78% yield. If the compound with trisubstituted alkene moiety (**1b**) was employed instead of **1a**, the yield of **2b** decreased slightly. We were pleased to find the reactions for **1c-e** bearing electron-donating or electron-withdrawing aryl groups occurred smoothly. The yields of *m*-methoxy (**2c**) or *p*-methyl group (**2d**) attached to the aromatic ring were higher than *p*-chlorophenyl one (**2e**). The substrate with *m*-methoxylphenyl group (**1c**) provided the best yield (up to 85%). The naphtyl group could also be tolerated. **2f** was obtained in a little lower yield than phenyl substituted one (**2a**).



Scheme 1. The synthesis of 1,2,3-triazole allyl acetates

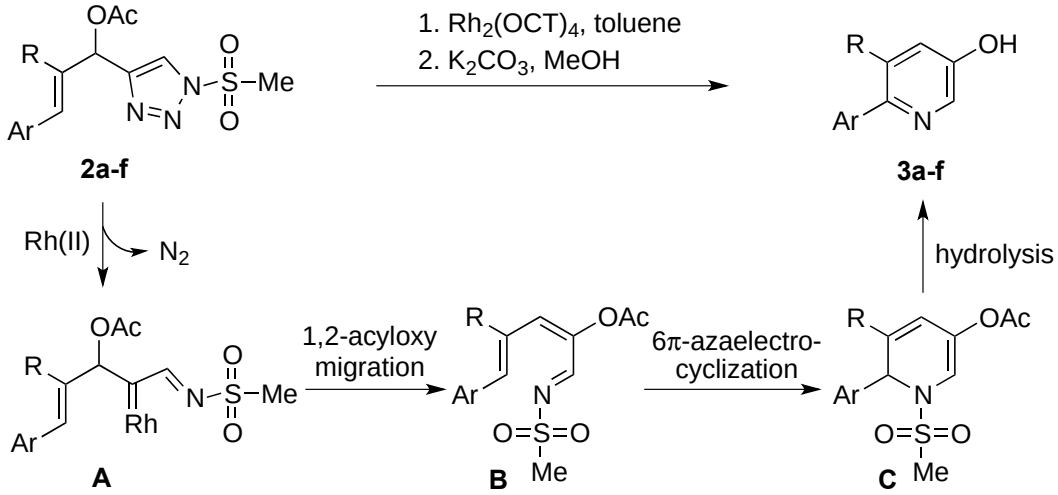
With 1,2,3-triazole allyl acetates (**2a-f**) in hand, the application of these compounds to access pyridine derivatives was outlined in Scheme 2. It required two steps to achieve this transformation. The first step was Rh(II)-catalyzed ring opening to reveal metallocarbene. The second step was hydrolysis. It inspired us to explore "one-pot" strategy. $Rh_2(OCT)_4$ (dirhodium(II) octanoate) was demonstrated as best catalyst. The reaction was proceeded with $Rh_2(OCT)_4$ in toluene at 80 ^oC. After 1-4 hours, the solvent was evaporated. The intermediate was dissolved in methanol again. Potassium carbonate was added to the system. The desired pyridines were obtained after separation. 6-Phenylpyridin-3-ol (**3a**) was acquired in highest yield (61%). Moderate yield could be acquired for other cases (**3b-f**). For the substrate with the trisubstituted alkene adjacent acetoxy group, the yield of **3b** dropped dramatically. No obvious electron

effect was observed for the products **3c-e**. When 2-naphthyl substrate was used, the yield of **3f** was slightly lower (54%) than phenyl one (**3a**).



Scheme 2. The synthesis of pyridines from 1,2,3-triazole allyl acetates

On the basis of the above results, the mechanism for the construction of pyridines is proposed in Scheme 3. The Rh(II) carbene species (**A**) are formed from *N*-sulfonyl-1,2,3-triazoles as precursors. The acetoxy group of **A** attacks the Rh(II) carbene species to undergo 1,2-acetoxy migration to give dienimines (**B**). A (1,3) strain and gauche strain may result in the selective formation of (*E*)-enol acetates.[16] Then 6π-azaelectrocyclization of **B** is realized to generate dihydropyridine acetates (**C**) at 80 °C. Finally, treating the dihydropyridine acetates (**C**) with potassium carbonate, the pyridine derivatives are afforded after hydrolysis.



Scheme 3. The proposed mechanism for the synthesis of pyridines

In conclusion, a facile and efficient synthesis of 1,2,3-triazole allyl acetates is developed from 3-acyloxy-1,4-enynes and sulfonyl azides by CuTC-catalyzed azide-alkyne cycloaddition (CuAAC). It offers an elaborately designed approach to access pyridine derivatives through 6π-azaelectrocyclization

and elimination. Efforts to further expand the substrate scope and the investigation of other modes of azacyclization are underway in our laboratories.

EXPERIMENTAL

Melting points was determined with a hot plate apparatus with uncorrected. All ^{1}H NMR (400 MHz or 500 MHz) and ^{13}C NMR (100 MHz or 126 MHz) were recorded on VARIAN INOVA-400M and AVANCE II 500 spectrometer at 25 °C. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as internal standard ($CDCl_3$: δ 7.26, for ^{1}H NMR and $CDCl_3$: δ 77.0 for ^{13}C NMR). For ^{1}H NMR, data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, dd = double doublet, m = multiplet), coupling constants (Hz) and integration. High resolution mass spectrometry data were obtained with UPLC/Q-Tof Mass Spectrometer and were determined by electrospray ionization (ESI).

Starting Materials. 3-Acyloxy-1,4-enynes were prepared from the respective cinnamaldehyde and ethynylmagnesium bromide with acetylation by the standard method.[15] All other chemicals used in this study were commercially available.

Typical Procedure for the Preparation of

(*E*)-1-(1-(Methylsulfonyl)-1*H*-1,2,3-triazol-4-yl)-3-phenylallyl acetate (2a): To a stirred solution of **1a** (0.4 mmol, 80 mg) in toluene (2 mL), was added methanesulfonyl azide (0.4 mmol, 48.4 mg) and CuTC (0.02 mmol, 3.8 mg) under nitrogen atmosphere. The mixture was stirred overnight at ambient temperature. Then the mixture was quenched by saturated aqueous NH_4Cl (2 mL). The organic materials were extracted with AcOEt (3×2 mL). The combined extracts were washed with brine (8 mL) and dried over anhydrous Na_2SO_4. Evaporation of the solvent gave a residue, which was purified by column chromatography on silica gel to give **2a** (100 mg, 78%). Yellow oil. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 2.16 (s, 3H), 3.56 (s, 3H), 6.52 (dd, *J* = 15.5, 1.5 Hz, 1H), 6.61 (d, *J* = 1.5 Hz, 1H), 6.80 (d, *J* = 15.5 Hz, 1H), 7.28-7.40 (m, 3H), 7.43-7.47 (m, 2H), 8.17 (s, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 21.4, 43.0, 68.6, 122.3, 124.0, 127.1, 128.8, 128.9, 135.2, 135.7, 146.4, 170.1. HRMS (ESI) Calcd for $C_{14}H_{16}N_3O_4S$ $[M+H]^+$: 322.0857. Found: 322.0862.

(*E*)-2-Methyl-1-(1-(methylsulfonyl)-1*H*-1,2,3-triazol-4-yl)-3-phenylallyl acetate (2b): Yellow oil. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 1.89 (s, 3H), 2.17 (s, 3H), 3.56 (s, 3H), 6.55 (s, 1H), 6.67 (s, 1H), 7.20-7.40 (m, 5H), 8.15 (s, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 14.7, 21.3, 42.9, 73.2, 122.3, 127.4, 128.5, 129.3, 129.8, 133.5, 136.6, 146.4, 169.9. HRMS (ESI) Calcd for $C_{15}H_{18}N_3O_4S$ $[M+H]^+$: 336.1014. Found: 336.1019.

(*E*)-3-(3-Methoxyphenyl)-1-(1-(methylsulfonyl)-1*H*-1,2,3-triazol-4-yl)allyl acetate (2c): Yellow oil. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 2.10 (s, 3H), 3.53 (s, 3H), 3.79 (s, 3H), 6.48 (dd, *J* = 15.8, 1.8 Hz, 1H),

6.60 (d, *J* = 1.8 Hz, 1H), 6.72 (d, *J* = 15.8 Hz, 1H), 6.81-6.83 (m, 1H), 6.89-6.91 (m, 1H), 6.98-7.01 (m, 1H), 7.20-7.30 (m, 1H), 8.15 (s, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 21.3, 42.9, 55.5, 68.5, 112.2, 114.6, 119.8, 122.5, 124.4, 129.9, 135.0, 137.2, 146.3, 160.1, 170.0. HRMS (ESI) Calcd for $C_{15}H_{18}N_3O_5S$ $[M+H]^+$: 352.0963. Found: 352.0969.

(*E*)-1-(1-(Methylsulfonyl)-1*H*-1,2,3-triazol-4-yl)-3-(*p*-tolyl)allyl acetate (2d): Yellow oil. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 2.15 (s, 3H), 2.36 (s, 3H), 3.54 (s, 3H), 6.48 (dd, *J* = 15.5, 1.5 Hz, 1H), 6.61 (d, *J* = 1.5 Hz, 1H), 6.80 (d, *J* = 15.5 Hz, 1H), 7.12 (d, *J* = 7.5 Hz, 2H), 7.35 (d, *J* = 7.5 Hz, 2H), 8.14 (s, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 21.4, 21.5, 42.9, 68.8, 122.3, 123.0, 127.0, 129.6, 132.9, 135.3, 138.8, 146.5, 170.1. HRMS (ESI) Calcd for $C_{15}H_{18}N_3O_4S$ $[M+H]^+$: 336.1014. Found: 336.1020.

(*E*)-3-(4-Chlorophenyl)-1-(1-(methylsulfonyl)-1*H*-1,2,3-triazol-4-yl)allyl acetate (2e): Yellow oil. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 2.15 (s, 3H), 3.55 (s, 3H), 6.48 (dd, *J* = 15.5, 1.5 Hz, 1H), 6.60 (d, *J* = 1.5 Hz, 1H), 6.76 (d, *J* = 15.5 Hz, 1H), 7.28-7.40 (m, 4H), 8.13 (s, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 21.3, 42.9, 68.5, 122.3, 124.7, 128.3, 129.1, 133.9, 134.2, 134.5, 146.1, 170.0. HRMS (ESI) Calcd for $C_{14}H_{15}ClN_3O_4S$ $[M+H]^+$: 356.0467. Found: 356.0475.

(*E*)-1-(1-(Methylsulfonyl)-1*H*-1,2,3-triazol-4-yl)-3-(naphthalen-2-yl)allyl acetate (2f): Yellow oil. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 2.14 (s, 3H), 3.54 (s, 3H), 6.51 (dd, *J* = 15.8, 1.8 Hz, 1H), 6.62 (d, *J* = 2.0 Hz, 1H), 6.78 (d, *J* = 15.8 Hz, 1H), 7.45-7.52 (m, 3H), 7.82-7.91 (m, 4H), 8.16 (s, 1H). ^{13}C-NMR (126 MHz, $CDCl_3$): δ 21.4, 43.0, 68.7, 122.3, 123.7, 124.3, 126.6, 126.7, 127.7, 127.9, 128.4, 128.6, 133.2, 133.6, 135.4, 146.4, 170.1. HRMS (ESI) Calcd for $C_{18}H_{18}N_3O_4S$ $[M+H]^+$: 372.1014. Found: 372.1019.

Typical Procedure for the Preparation of 6-Phenylpyridin-3-ol (3a): To a stirred solution of **2a** (0.2 mmol, 64.2 mg) in toluene (4 mL), was added $Rh_2(OCT)_4$ (0.002 mmol, 1.6 mg) under nitrogen atmosphere. The mixture was stirred for 2 h at 80 °C. The solvent was evaporated. The residue was dissolved in MeOH (2 mL). Potassium carbonate (0.4 mmol, 55.2 mg) was added to the mixture. The mixture was stirred for 4 h and purified by column chromatography on silica gel (1% water deactivation) to give **3a** (21 mg, 61%). Colorless solid. Mp 188-190 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 7.22 (d, *J* = 1.5 Hz, 1H), 7.36-7.40 (m, 1H), 7.42-7.46 (m, 2H), 7.61 (d, *J* = 7.5 Hz, 1H), 7.87 (d, *J* = 7.5 Hz, 2H), 8.32 (d, *J* = 1.5 Hz, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 121.8, 124.2, 126.7, 128.5, 129.0, 137.6, 139.1, 150.6, 151.9. HRMS (ESI) Calcd for $C_{11}H_{10}NO$ $[M+H]^+$: 172.0757. Found: 172.0759.

5-Methyl-6-phenylpyridin-3-ol (3b): Colorless solid. Mp 171-173 °C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 2.20 (s, 3H), 7.02 (s, 1H), 7.32-7.48 (m, 5H), 8.12 (s, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 21.0, 122.5, 124.6, 127.1, 129.4, 133.4, 137.1, 138.9, 150.3, 152.1. HRMS (ESI) Calcd for $C_{12}H_{12}NO$ $[M+H]^+$: 186.0914. Found: 186.0921.

6-(3-Methoxyphenyl)pyridin-3-ol (3c): Yellow solid. Mp 196-198 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 3.87 (s, 3H), 6.91 (d, *J* = 7.6 Hz, 1H), 7.20-7.24 (m, 1H), 7.32-7.44 (m, 3H), 7.60 (d, *J* = 7.6 Hz, 1H),

8.28 (s, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 55.5, 112.1, 114.7, 119.3, 123.0, 125.4, 130.0, 137.0, 140.1, 149.3, 153.8, 160.2. HRMS (ESI) Calcd for $C_{12}H_{12}NO_2$ $[M+H]^+$: 202.0864. Found: 202.0871.

6-(*p*-Tolyl)pyridin-3-ol (3d): Colorless solid. Mp 168-170 ºC. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 2.39 (s, 3H), 7.21-7.24 (m, 3H), 7.60 (d, *J* = 7.8 Hz, 1H), 7.78 (d, *J* = 7.8 Hz, 2H), 8.30 (s, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 21.4, 121.9, 124.5, 126.4, 126.7, 127.7, 129.6, 136.2, 137.5, 138.4, 148.9, 152.8. HRMS (ESI) Calcd for $C_{12}H_{12}NO$ $[M+H]^+$: 186.0914. Found: 186.0919.

6-(4-Chlorophenyl)pyridin-3-ol (3e): Yellow solid. Mp 187-189 ºC. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.22 (d, *J* = 1.5 Hz, 1H), 7.36-7.42 (m, 2H), 7.59 (d, *J* = 7.5 Hz, 1H), 7.82 (d, *J* = 7.5 Hz, 2H), 8.29 (s, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 121.6, 124.1, 128.0, 129.1, 129.3, 134.6, 137.8, 149.4, 151.9. HRMS (ESI) Calcd for $C_{11}H_9ClNO$ $[M+H]^+$: 206.0368. Found: 206.0375.

6-(Naphthalen-2-yl)pyridin-3-ol (3f): Colorless solid. Mp 211-213 ºC. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 7.28-7.32 (m, 1H), 7.45-7.50 (m, 2H), 7.76 (d, *J* = 7.6 Hz, 1H), 7.84-7.94 (m, 4H), 8.04 (d, *J* = 7.6 Hz, 1H), 8.35 (s, 1H), 8.38 (d, *J* = 1.6 Hz, 1H). ^{13}C-NMR (126 MHz, $CDCl_3$): δ 122.1, 124.3, 124.7, 125.8, 126.5, 126.5, 127.9, 128.7, 128.7, 133.5, 133.8, 136.4, 137.8, 150.4, 152.1. HRMS (ESI) Calcd for $C_{15}H_{12}NO$ $[M+H]^+$: 222.0914. Found: 222.0921.

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