

FeCl₃-catalyzed propargylation of aromatic compounds with propargylic acetates

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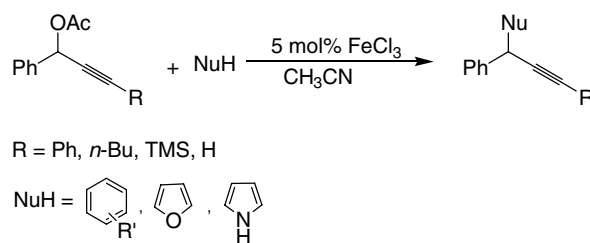
Abstract—A new method for the synthesis of propargylated aromatic compounds is developed. The reaction was carried out at room temperature in the presence of a catalytic amount of FeCl₃ in acetonitrile, high product yields were obtained with excellent regioselectivity and the reaction proceeded smoothly without exclusion of moisture or air.

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Friedel–Crafts alkylation is one of the most important C–C bond forming reactions, which provides an efficient synthetic route to numerous functionalized aromatic compounds possessing special properties. Thus, the research of this reaction has got much attention, a series of aromatic substances with various electrophilic substrates such as active carbonyl compounds, epoxides, and electron-deficient olefins have been extensively studied.¹ However, little attention has been paid to the reaction of propargylic alcohol derivatives with aromatic compounds, despite such reactions playing an important role in the synthesis of some natural products such as *O*-methyldeitol, mimosifoliol, and β-apopicropodophyllin and so on.^{2,4} The Nicholas reaction has been known to be effective for propargylation of aromatic compounds by using a stoichiometric amount of Co₂(CO)₈, where several steps are necessary to obtain propargylated products from propargylic alcohols via cationic propargyl complexes [Co₂(CO)₆(propargyl)]⁺.³ Recently, Toste and co-workers⁴ and Uemura and co-workers⁵ have described efficient rhenium [(dppm) ReOCl₃] and diruthenium [[Cp*⁺RuCl(SR)₂]] catalyzed propargylation of aromatic compounds with propargylic alcohols, respectively. In addition, Campagne and co-workers⁶ and Dyker and co-workers⁷ introduced the gold [NaAuCl₄·2H₂O; AuCl₃] as an alternative catalyst for the prop-

argylation. However, the peculiarity and high cost of such catalysts are a barrier to their large-scale use. Therefore, development of a general, efficient, cheap, and readily available catalyst for propargylation of aromatic compounds is highly desirable.

Herein, we wish to report an efficient FeCl₃-catalyzed propargylation of aromatic compounds with propargylic acetates bearing not only a terminal alkyne group but also internal alkyne group, sp³-C–C bonds were formed after the propargylation event (Scheme 1). To the best of our knowledge, there was no report in the literature on the reaction of propargylic esters with aromatic compounds. The reaction was carried out at room temperature in the presence of a catalytic amount of FeCl₃ in acetonitrile. High product yields were obtained with excellent reaction regioselectivity and the reaction proceeded smoothly without exclusion of moisture or air from the reaction mixture.⁸



Scheme 1.

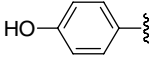
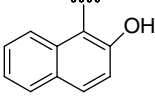
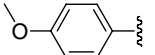
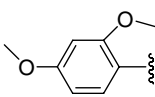
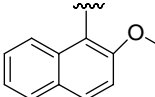


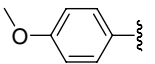
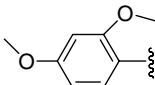

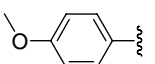
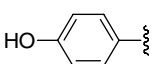

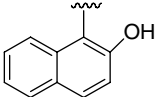

Keywords: Propargylation of aromatic compounds; Propargylic acetates; Iron(III) chloride.

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In order to determine the scope and limitation of this reaction, a series of propargylic acetates with various aromatic compounds were investigated. First, reactions of 1,3-diphenylprop-2-ynyl acetate (**1a**) with various aromatic compounds were carried out. Typical results

were shown in Table 1. The corresponding propargyl adducts were obtained in high yields with a complete regioselectivity (Table 1, entries 1–7). In the cases of electron-rich arenes such as phenol, β -naphthol, methoxybenzene, 1,3-dimethoxybenzene, and β -methoxy-

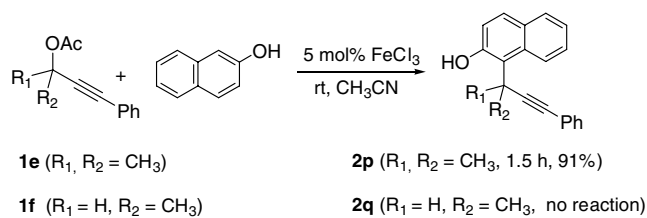
Table 1. Propargylation of aromatic compounds with propargylic acetates via Scheme 1^a

Entry	R	Nu	Time (h)	Product	Yields of 2 ^c (%)
1	1a , Ph		0.5	2a	91
2	1a , Ph		0.5	2b	93
3	1a , Ph		0.5	2c	83
4	1a , Ph		3.0	2d	80
5	1a , Ph		0.5	2e	92
6	1a , Ph		0.5	2f	81
7	1a , Ph		4.0	2g	60 ^b
8	1b , TMS		2.0	2h	80
9	1b , TMS		3.0	2i	78
10	1b , TMS		5.0	2j	80
11	1c , <i>n</i> -Bu		0.5	2k	85
12	1c , <i>n</i> -Bu		0.5	2l	93
13	1c , <i>n</i> -Bu		1.0	2m	86
14	1d , H		3.0	2n	68
15	1d , H		6.0	2o	55

^a The reactions of **1** (1 mmol) with NuH (3 mmol) were carried out in the presence of FeCl₃ (0.05 mmol) in CH₃CN (2 mL) at room temperature.

^b The reaction was carried out at 60 °C.

^c Isolated yields.



Scheme 2.

naphthalene, the corresponding Friedel–Crafts propargylated products (**2a–e**) were obtained in 91%, 93%, 83%, 80%, and 92% isolated yields, respectively (entries 1–5). Heterocyclic aromatic compound furan can also be propargylated with **1a** at room temperature, the propargylation occurred selectively at the α -position of furan, giving the propargylic adduct **2f** in 81% isolated yield (entry 6). Nevertheless, in the case of pyrrole, longer reaction time and higher reaction temperature were required to obtain **2g** in 60% isolated yield (entry 7). In all cases, propargylation occurred selectively at the electron-rich position of the aromatic compounds. These results indicated that the reaction proceeded electrophilically.

On the other hand, the reaction could also be extended to propargyl acetates bearing a variety of alkyne substituents. Variation in the alkyne substituent from an aryl to an alkyl or trimethylsilyl (**1a–c**) was well tolerated, without noticeable difference observed in the reaction temperature, time, and product yield. Gratifyingly, 1-phenylprop-2-ynyl acetate (**1d**) bearing a terminal alkyne substituent was successfully arylated in moderate yields and no polymerization was detected, although compared to internal alkyne substituent, longer reaction time was required (entries 14 and 15). Nonbenzylic propargyl acetates also participated in the aromatic propargylation. Iron(III)-catalyzed coupling of tertiary aliphatic acetate (**1e**) with β -naphthol allowed for the construction of a quaternary carbon (Scheme 2, **2p**); however, secondary aliphatic acetate (**1f**) failed to get the propargylated aromatic compound after 24 h (Scheme 2, **2q**). The experimental results suggested a mechanism through the formation of propargylic cation intermediate. Unstability of the propargylic cation intermediate clearly made the substitution reaction less favorable.

In conclusion, we have developed a general and efficient FeCl_3 -catalyzed propargylation of aromatic compounds, leading to the construction of C–C bonds. The corresponding propargylic adducts were obtained in high yields with complete regioselectivity. In comparison with rhenium, ruthenium, and gold complexes, which were used to catalyze the propargylation of aromatic compounds, FeCl_3 as the catalyst offer several relevant advantages including cheapness, commercial availability, and mild reaction conditions of this transformation. Further investigations for the elucidation of the detailed reaction mechanism and broadening the scope of this methodology are currently going on in our laboratory.

Acknowledgements

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- Typical experimental procedure:**
 (a) *Preparation of propargylic acetates.* Bartels, A.; Mahrwald, R.; Müller, K. *Adv. Synth. Catal.* **2004**, *346*, 483.
 (b) *Preparation of propargylated aromatic compounds.* A typical experimental procedure for the reaction of propargylic acetates with aromatic compounds catalyzed by 5 mol % FeCl_3 is described below: to a 5 mL flask, 1,3-diphenylprop-2-ynyl acetate (**1a**) (250 mg, 1 mmol), phenol (282 mg, 3.0 mmol), CH_3CN (2 mL), and anhydrous FeCl_3 (8 mg, 0.05 mmol) were successively added, and then the mixture was magnetically stirred at room temperature for 0.5 h. The solvent was concentrated under reduced pressure by an aspirator, and then the residue was purified by silica gel column chromatography (EtOAc/hexane) to afford 4-(1,3-diphenylprop-2-ynyl)phenol (**2a**) as a yellow solid (258 mg, 91% yield).
 As for the known compounds, ^1H NMR and ^{13}C NMR are provided, data are in accordance with previously reported results. For the new compounds, ^1H NMR, ^{13}C NMR, IR, and elemental analysis data are provided.

4-(1,3-Diphenylprop-2-ynyl)phenol (2a): Yield 91%. A yellow solid (mp 82–86 °C). ¹H NMR (CDCl₃, 400 MHz): δ 4.91 (br s, 1H), 5.14 (s, 1H), 6.76 (apparent d, 2H, *J* = 8.5 Hz), 7.20–7.34 (m, 8H), 7.39–7.49 (m, 4H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ 42.8, 84.7, 90.4, 115.4, 123.4, 126.8, 127.7, 127.9, 128.2, 128.6, 129.1, 131.6, 134.0, 141.9, 154.2 ppm; IR (film) *v*_{max}: 3353, 3058, 3027, 2216, 1618, 1602, 1509, 1490 cm⁻¹.

1-(1,3-Diphenylprop-2-ynyl)naphthalen-2-ol (2b): Yield 93%. A pale yellow oil. ¹H NMR (CDCl₃, 400 MHz): δ 6.27 (s, 1H), 6.38 (s, 1H), 7.16 (d, 1H, *J* = 8.9 Hz), 7.20–7.50 (m, 12H), 7.75 (d, 1H, *J* = 8.9 Hz), 7.80 (d, 1H, *J* = 8.0 Hz), 8.03 (d, 1H, *J* = 8.6 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ 33.7, 86.0, 88.3, 117.6, 119.1, 122.5, 123.0, 123.3, 126.8, 127.0, 127.2, 128.3, 128.5, 128.7, 128.8, 129.6, 129.8, 131.8, 132.3, 139.5, 152.3 ppm; IR (film) *v*_{max}: 3420, 3054, 3027, 2217, 1631, 1595, 1520, 1490 cm⁻¹.

1-(3-(4-Methoxyphenyl)-3-phenylprop-1-ynyl)benzene (2c): Yield 83%. A clear oil. ¹H NMR (CDCl₃, 400 MHz): δ 3.78 (s, 3H), 5.16 (s, 1H), 6.85 (d, 2H, *J* = 8.8 Hz), 7.19–7.48 (m, 12H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ 43.1, 55.4, 84.6, 90.4, 113.7, 123.2, 126.4, 127.4, 127.5, 127.8, 128.2, 128.5, 131.2, 133.5, 141.5, 157.8 ppm; IR (film) *v*_{max}: 3060, 3029, 3002, 1608, 1584, 1509, 1251 cm⁻¹.

1-(3-(2,4-Dimethoxyphenyl)-3-phenylprop-1-ynyl)benzene (2d): Yield 80%. A white solid (mp 101–102 °C). ¹H NMR (CDCl₃, 400 MHz): δ 3.78 (s, 3H), 3.80 (s, 3H), 5.58 (s, 1H), 6.44 (d, 1H, *J* = 2.4 Hz), 6.49 (dd, 1H, *J* = 8.4 Hz and 2.4 Hz), 7.16–7.31 (m, 6H), 7.43–7.51 (m, 5H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ 36.1, 55.3, 55.5, 83.4, 91.1, 98.6, 104.5, 122.9, 123.8, 126.4, 127.7, 128.1, 128.3, 129.5, 131.7, 142.1, 157.1, 159.9 ppm; IR (film) *v*_{max}: 3059, 3027, 3001, 2224, 1610, 1588, 1504, 1453, 1264 cm⁻¹.

1-(1,3-Diphenylprop-2-ynyl)-2-methoxynaphthalene (2e): Yield 92%. A white solid (mp 110–111 °C). ¹H NMR (CDCl₃, 400 MHz): δ 3.88 (s, 3H), 6.52 (s, 1H), 7.10–7.29 (m, 9H), 7.39–7.43 (m, 2H), 7.48–7.51 (m, 2H), 7.71 (apparent d, 1H, *J* = 8.8 Hz), 7.75 (d, 1H, *J* = 8.8 Hz), 8.15 (apparent d, 1H, *J* = 8.7 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ 32.9, 57.1, 84.3, 90.7, 113.6, 122.1, 123.6, 124.1, 125.7, 126.2, 126.4, 127.3, 128.1, 128.4, 128.5, 128.6, 129.9, 130.2, 131.9, 132.2, 141.1, 154.3 ppm; IR (film) *v*_{max}: 3059, 3029, 1623, 1596, 1512, 1490, 1250 cm⁻¹.

2-(1,3-Diphenylprop-2-ynyl)furan (2f): Yield 81%. A yellow oil. ¹H NMR (CDCl₃, 400 MHz): δ 5.26 (s, 1H), 6.28 (apparent d, 1H, *J* = 3.0 Hz), 6.30–6.33 (m, 1H), 7.23–7.39 (m, 7H), 7.45–7.51 (m, 4H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ 37.8, 83.9, 87.4, 106.6, 110.3, 123.1, 127.3, 127.8, 128.1, 128.2, 128.6, 131.7, 138.8, 142.2, 153.7 ppm; IR (film) *v*_{max}: 3117, 3061, 3030, 1598, 1501, 1491, 1453, 1071 cm⁻¹.

2-(1,3-Diphenylprop-2-ynyl)-1H-pyrrole (2g): Yield 60%. A brown solid (mp 68–71 °C). ¹H NMR (CDCl₃, 400 MHz): δ 5.28 (s, 1H), 6.02–6.06 (m, 1H), 6.15 (dd, 1H, *J* = 6.0 and 2.8 Hz), 6.68–6.71 (m, 1H), 7.23–7.37 (m, 6H), 7.42–7.49 (m, 4H), 8.16 (br s, 1H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ 37.3, 84.2, 88.6, 106.6, 108.7, 117.4, 123.2, 127.3, 127.8, 128.2, 128.3, 128.8, 130.6, 131.8, 140.2 ppm; IR (film) *v*_{max}: 3431, 1597, 1489, 1452 cm⁻¹.

(3-(4-Methoxyphenyl)-3-phenylprop-1-ynyl)trimethylsilane (2h): Yield 80%. A clear oil. ¹H NMR (CDCl₃, 400 MHz): δ 0.27 (s, 9H), 3.82 (s, 3H), 5.05 (s, 1H), 6.90 (d, 2H, *J* = 8.1 Hz), 7.26–7.45 (m, 7H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ 0.2, 43.3, 55.3, 88.9, 107.1, 114.0, 126.8, 127.8, 128.6, 128.9, 133.8, 141.9, 158.5 ppm; IR (film) *v*_{max}: 3062, 3028, 2171, 1603, 1510, 1453, 1249, 840 cm⁻¹.

(3-(2,4-Dimethoxyphenyl)-3-phenylprop-1-ynyl)trimethylsilane (2i): Yield 78%. A colorless solid (mp 82–84 °C). ¹H NMR (CDCl₃, 400 MHz): δ 0.19 (s, 9H), 3.77 (s, 3H), 3.78

(s, 3H), 5.40 (s, 1H), 6.41 (d, 1H, *J* = 2.4 Hz), 6.48 (dd, 1H, *J* = 8.4 and 2.4 Hz), 7.13–7.19 (m, 1H), 7.22–7.28 (m, 2H), 7.35–7.40 (m, 2H), 7.42 (d, 1H, *J* = 8.4 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ 0.19, 36.5, 55.4, 55.5, 87.4, 98.6, 104.5, 107.8, 122.7, 126.4, 127.7, 128.2, 129.4, 141.9, 157.0, 159.9 ppm; IR (film) *v*_{max}: 3027, 2169, 1612, 1588, 1503, 1452, 1249, 1036 cm⁻¹.

(3-(Furan-2-yl)-3-phenylprop-1-ynyl)trimethylsilane (2j): Yield 80%. A clear oil. ¹H NMR (CDCl₃, 400 MHz): δ 0.02 (s, 9H), 4.85 (s, 1H), 6.01 (d, 1H, *J* = 3.1 Hz), 6.08 (dd, 1H, *J* = 3.1 Hz and 1.7 Hz), 7.02–7.22 (m, 6H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ 0.0, 38.2, 88.4, 103.7, 106.5, 110.2, 127.3, 127.8, 128.6, 138.6, 142.2, 153.7 ppm; IR (film) *v*_{max}: 3063, 3029, 2177, 1603, 1495, 1453, 1076, 843 cm⁻¹. Anal. Calcd for C₁₆H₁₈OSi: C, 75.54; H, 7.13. Found: C, 75.45; H, 7.42.

1-Methoxy-4-(1-phenylhept-2-ynyl)benzene (2k): Yield 85%. A clear oil. ¹H NMR (CDCl₃, 400 MHz): δ 0.92 (t, 3H, *J* = 7.2 Hz), 1.39–1.48 (m, 2H), 1.50–1.58 (m, 2H), 2.27 (td, 2H, *J* = 7.2 Hz and 2.3 Hz), 3.76 (s, 3H), 4.92 (s, 1H), 6.80–6.84 (m, 2H), 7.16–7.36 (m, 7H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ 13.7, 18.7, 22.1, 31.1, 42.5, 55.3, 80.8, 85.0, 113.9, 126.6, 127.8, 128.5, 128.8, 134.8, 142.8, 158.3 ppm; IR (film) *v*_{max}: 1600, 1509, 1452, 1248 cm⁻¹. Anal. Calcd for C₂₀H₂₂O: C, 86.29; H, 7.97. Found: C, 86.15; H, 8.08.

4-(1-Phenylhept-2-ynyl)phenol (2l): Yield 93%. A yellow oil. ¹H NMR (CDCl₃, 400 MHz): δ 0.92 (t, 3H, *J* = 7.2 Hz), 1.38–1.59 (m, 4H), 2.27 (td, 2H, *J* = 7.2 and 2.0 Hz), 4.66–4.76 (br s, 1H), 4.91 (s, 1H), 6.72–6.77 (m, 2H), 7.16–7.25 (m, 3H), 7.29 (t, 2H, *J* = 7.6 Hz), 7.32–7.37 (m, 2H); ¹³C NMR (CDCl₃, 100 MHz): δ 13.7, 18.7, 22.1, 31.1, 42.5, 80.8, 85.0, 115.3, 126.6, 127.8, 128.5, 129.1, 135.0, 142.8, 154.2; IR (film) *v*_{max}: 3387, 1598, 1510, 1452 cm⁻¹. Anal. Calcd for C₁₉H₂₀O: C, 86.32; H, 7.63. Found: C, 86.51; H, 7.35.

2-(1-Phenylhept-2-ynyl)furan (2m): Yield 86%. A pale yellow oil. ¹H NMR (CDCl₃, 400 MHz): δ 0.91 (t, 3H, *J* = 7.3 Hz), 1.38–1.48 (m, 2H), 1.49–1.57 (m, 2H), 2.26 (td, 2H, *J* = 6.8 Hz and 2.3 Hz), 5.01 (s, 1H), 6.18 (d, 1H, *J* = 3.2 Hz), 6.27 (dd, 1H, *J* = 3.2 Hz and 1.8 Hz), 7.21–7.42 (m, 6H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ 13.6, 18.5, 22.0, 30.9, 37.3, 77.8, 84.3, 106.2, 110.2, 127.1, 127.7, 128.5, 139.5, 142.0, 154.6 ppm; IR (film) *v*_{max}: 3063, 3029, 1599, 1502, 1453, 1073 cm⁻¹. Anal. Calcd for C₁₇H₁₈O: C, 85.67; H, 7.61. Found: C, 85.75; H, 7.42.

1-(1-Phenylprop-2-ynyl)naphthalen-2-ol (2n): Yield 68%. A colorless oil. ¹H NMR (CDCl₃, 400 MHz): δ 2.60 (d, 1H, *J* = 3.9 Hz), 6.08 (s, 1H), 6.14 (d, 1H, *J* = 3.9 Hz), 7.13 (d, 1H, *J* = 8.8 Hz), 7.18–7.45 (m, 7H), 7.74 (d, 1H, *J* = 8.8 Hz), 7.78 (d, 1H, *J* = 8.0 Hz), 7.94 (d, 1H, *J* = 8.6 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ 32.9, 73.9, 83.4, 117.3, 119.1, 123.1, 123.4, 126.9, 127.1, 128.7, 128.9, 129.7, 130.0, 132.3, 138.9, 152.1 ppm; IR (film) *v*_{max}: 3455, 3284, 3058, 3027, 2112, 1630, 1599, 1520, 1490 cm⁻¹. Anal. Calcd for C₁₉H₁₄O: C, 88.34; H, 5.46. Found: C, 88.51; H, 5.40.

2-(1-Phenylprop-2-ynyl)furan (2o): Yield 55%. A pale yellow oil. ¹H NMR (CDCl₃, 400 MHz): δ 2.44 (d, 1H, *J* = 2.5 Hz), 5.06 (d, 1H, *J* = 2.5 Hz), 6.21–6.24 (m, 1H), 6.29–6.32 (m, 1H), 7.24–7.44 (m, 6H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ 37.0, 72.0, 81.9, 106.7, 110.3, 127.5, 127.7, 128.6, 138.2, 142.3, 153.1 ppm; IR (film) *v*_{max}: 3293, 3063, 3030, 2122, 1599, 1503, 1453 cm⁻¹.

1-(2-Methyl-4-phenylbut-3-yn-2-yl)naphthalen-2-ol (2p): Yield 91%. A pale yellow oil. ¹H NMR (CDCl₃, 400 MHz): δ 1.39 (s, 6H), 5.59 (s, 1H), 6.88–7.19 (m, 9H), 7.59 (d, 2H, *J* = 8.8 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ 26.3, 75.3, 116.0, 118.8, 122.9, 125.0, 126.3, 127.2, 127.8, 128.3, 129.8, 130.1, 130.3, 130.4, 135.4, 141.6, 153.0 ppm; IR (film) *v*_{max}: 3473, 3054, 2248, 1623, 1590, 1505, 1453 cm⁻¹.