

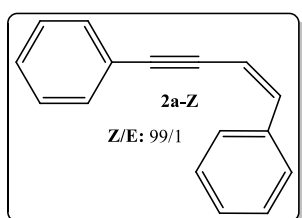
One-pot Synthesis of α,β -Unsaturated Ketones through Sequential Alkyne Dimerization/Hydration Reactions using Hoveyda-Grubbs Catalyst

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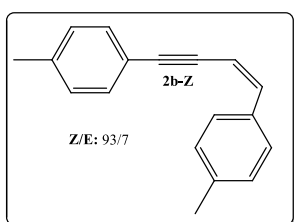
SUPPORTING INFORMATION

Spectral data for (Z)-but-1-en-3-yne-1,4-diylidibenzene (2a)



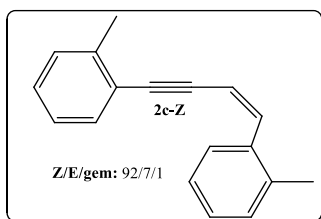
¹H NMR (400 MHz, CDCl₃) δ 7.94 (d, J = 7.5 Hz, 2H), 7.50 (d, J = 4.7 Hz, 2H), 7.39 – 7.25 (m, 6H + CHCl₃), 6.70 (d, J = 11.9 Hz, 1H), 5.93 (d, J = 11.9 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 138.58, 136.33, 131.50, 128.81, 123.17, 107.43, 95.90, 87.93; MS (EI): m/z 204 [M]⁺

Spectral data for (Z)-4,4'-(but-1-en-3-yne-1,4-diyl)bis(methylbenzene) (2b)



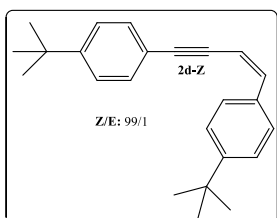
¹H NMR (400 MHz, CDCl₃) δ 7.84 (d, J = 7.6 Hz, 2H), 7.38-7.30 (m, 2H), 7.15-7.00 (m, 4H), 6.32 (d, J = 11.6 Hz, 1H), 5.85 (d, J = 11.6 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 140.94, 138.53, 134.02, 131.38, 129.62, 128.77, 127.19, 120.58, 106.89, 106.39, 95.84, 91.92, 88.50, 88.00, 21.49, 21.01; MS (EI): m/z 232 [M]⁺

Spectral data for (Z)-2,2'-(but-1-en-3-yne-1,4-diyl)bis(methylbenzene) (2c)



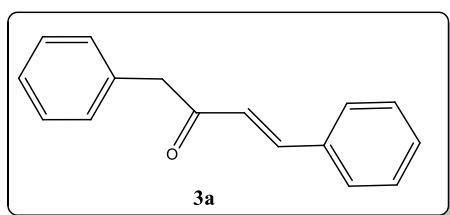
^1H NMR (400 MHz, CDCl_3) δ 8.57 (m, 1H), 7.75-7.66 (m, 2H), 7.41-7.15 (m, 5H + CHCl_3), 7.14 (d, $J=11.6$ Hz, 1H, Z-isomer), 6.63 (d, $J=16.2$ Hz, 1H, E-isomer), 6.26 (d, $J=11.6$ Hz, 1H, Z-isomer), 2.76 (s), 2.67 (s), 2.62 (s), 2.58 (s); ^{13}C NMR (100 MHz, CDCl_3) δ (Z-isomer) 140.22, 136.55, 132.13, 130.15, 129.51, 128.34, 125.58, 108.58, 94.02, 91.68, 20.88, 19.88; MS (EI): m/z 232 $[\text{M}]^+$

Spectral data for (Z)-4,4'-(but-1-en-3-yne-1,4-diyl)bis(tert-butylbenzene) (2d)



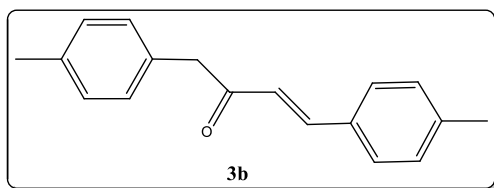
^1H NMR (400 MHz, CDCl_3) δ 7.77 (d, $J=8.3$ Hz, 2H), 7.38-7.20 (m, 6H + CHCl_3), 6.51 (d, $J=11.6$ Hz, 1H), 5.72 (d, $J=11.6$ Hz, 1H), 1.21 (s, 18H); ^{13}C NMR (100 MHz, CDCl_3) δ 131.38, 129.62, 128.77, 127.19, 120.58, 106.89, 106.39, 95.84, 91.92, 88.50, 88.00, 21.49, 21.01; MS (EI): m/z 316 $[\text{M}]^+$

Spectral data for (E)-1,4-diphenylbut-3-en-2-one



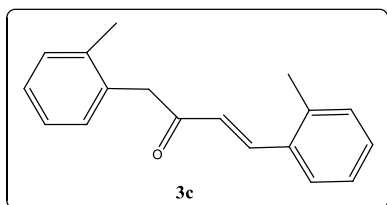
^1H NMR (400 MHz, CDCl_3) δ 7.52 (d, $J=16.2$ Hz, 1H), 7.38 (m, 2H), 7.25-7.15 (m, 8H), 6.67 (d, $J=16.2$ Hz, 1H), 3.83 (s, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 197.61, 143.64, 134.56, 130.70, 129.72, 129.12, 127.92, 127.21, 125.72, 48.52; MS (EI): m/z 222 $[\text{M}]^+$

Spectral data for (E)-1,4-di-p-tolylbut-3-en-2-one



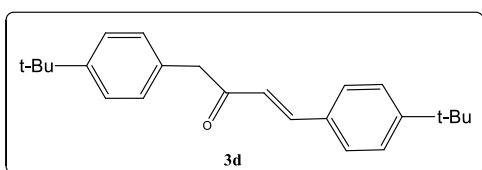
^1H NMR (400 MHz, CDCl_3) δ 7.52 (d, $J=16.2$ Hz, 1H), 7.33 (d, $J=7.8$ Hz, 2H), 7.25-7.06 (m, 6H), 6.64 (d, $J=16.2$ Hz, 1H), 3.80 (s, 2H), 2.27 (s, 3H) 2.24 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 196.92, 142.61, 140.31, 135.58, 128.68, 138.30, 123.17, 47.09, 20.25; MS (EI): m/z 250 $[\text{M}]^+$

Spectral data for (E)-1,4-di-o-tolylbut-3-en-2-one (3c)



^1H NMR (400 MHz, CDCl_3) δ 7.86 (d, $J=15.9$ Hz, 1H), 7.43 (d, $J=7.8$ Hz, 1H), 7.34-6.98 (m, 7H + CHCl_3), 6.63 (d, $J=16.0$ Hz, 1H), 3.87 (s, 2H), 2.33 (s, 3H), 2.22 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 197.32, 140.60, 130.82, 130.00, 128.87, 126.88, 126.02, 125.12, 47.22, 19.74; MS (EI): m/z 250 $[\text{M}]^+$

Spectral data for (E)-1,4-bis(4-(tert-butyl)phenyl)but-3-en-2-one



^1H NMR (400 MHz, CDCl_3) δ 7.58 (d, $J=16.1$ Hz, 1H), 7.42 (d, $J=7.8$ Hz, 2H), 7.25-7.15 (m, 6H), 6.73 (d, $J=16.1$ Hz, 1H), 3.89 (s, 2H), 1.26 (s, 18 H); ^{13}C NMR (100 MHz, CDCl_3) δ 197.98, 143.27, 141.19, 136.70, 129.67, 129.43, 128.41, 124.28, 47.76, 36.82, 29.73, MS (EI): m/z 334 $[\text{M}]^+$

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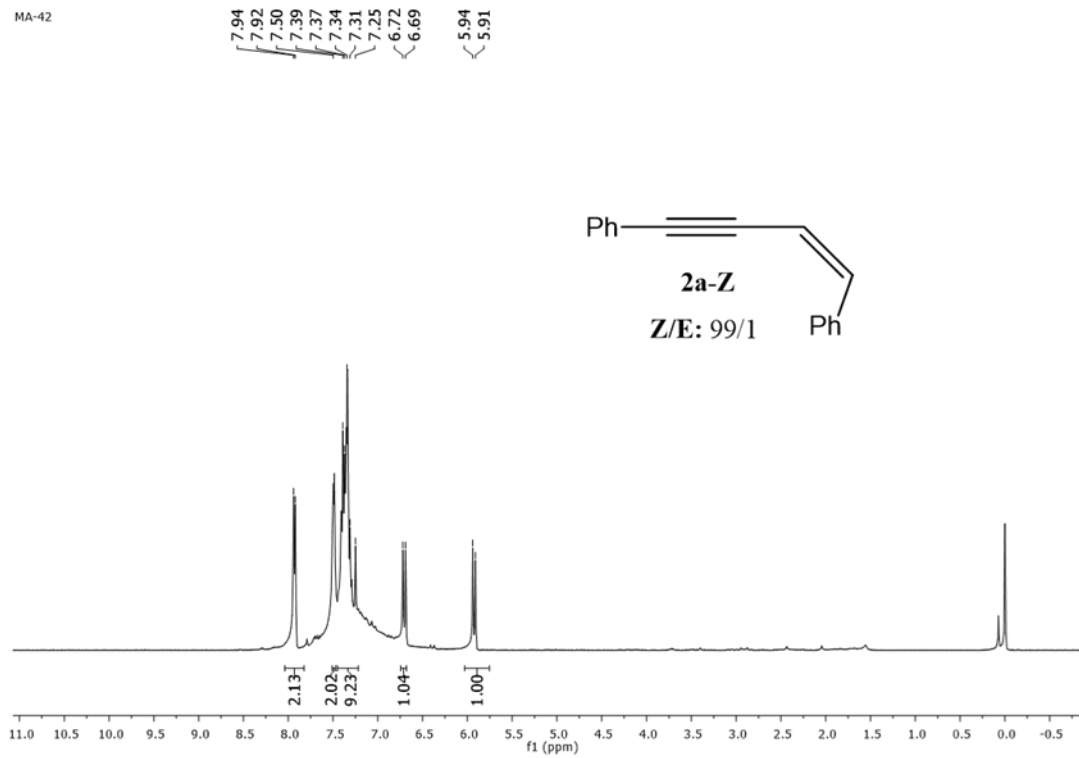


Figure S1. ^1H NMR spectrum of **2a** (400 MHz, CDCl_3) (Table 3, Entry 4)

MA-42

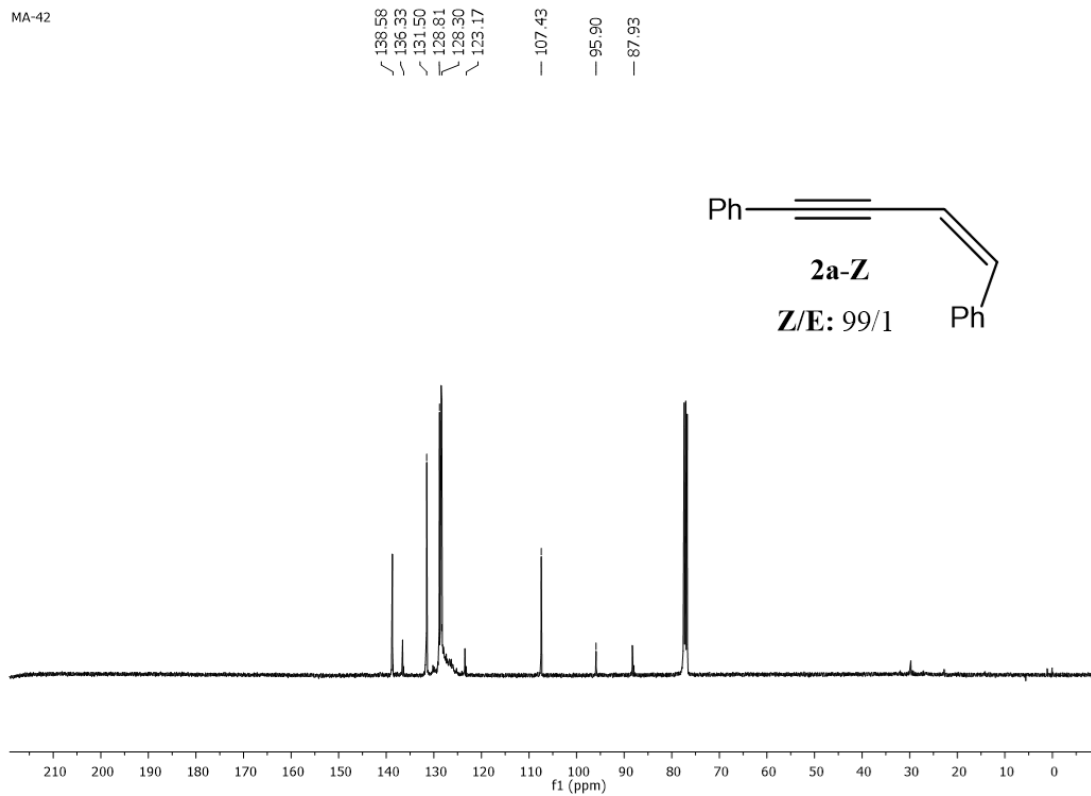


Figure S2. ^{13}C NMR spectrum of **2a** (100 MHz, CDCl_3) (Table 3, Entry 4)

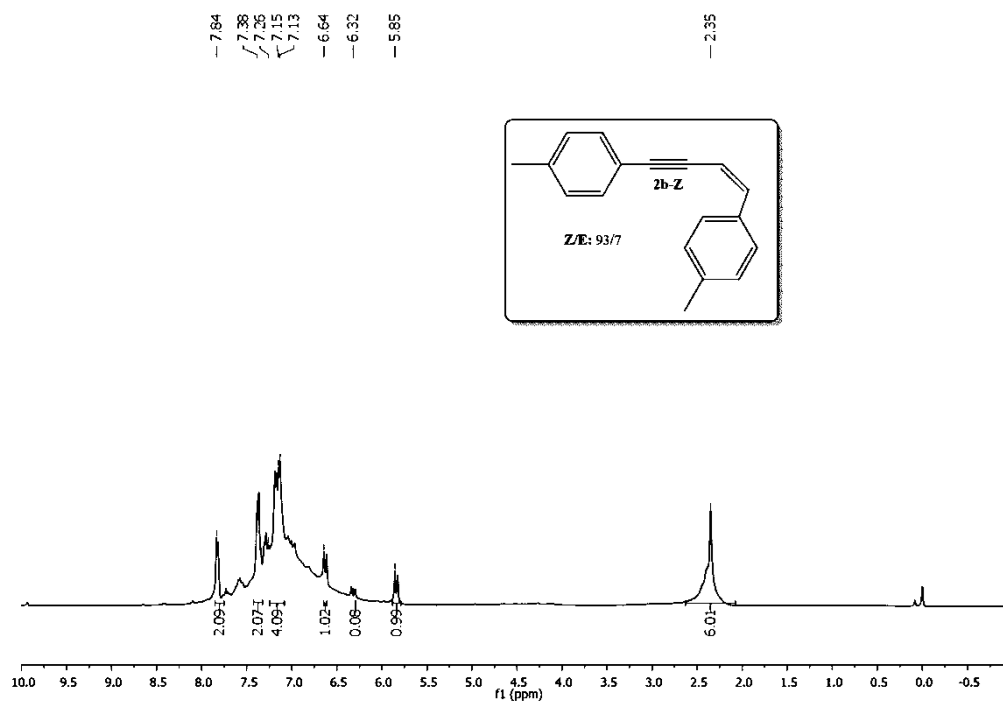


Figure S3. $^1\text{H NMR}$ spectrum of **2b** (400 MHz, CDCl_3) (Table 3, Entry 8)

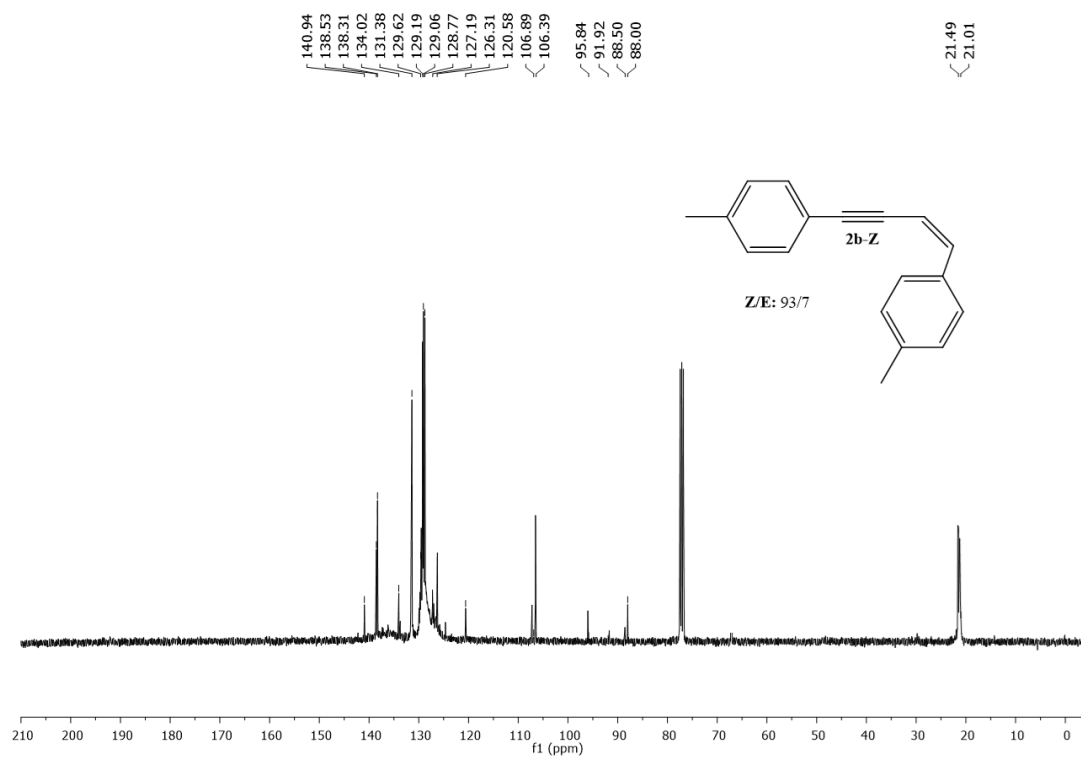


Figure S4. $^{13}\text{C NMR}$ spectrum of **2b** (100 MHz, CDCl_3) (Table 3, Entry 8)

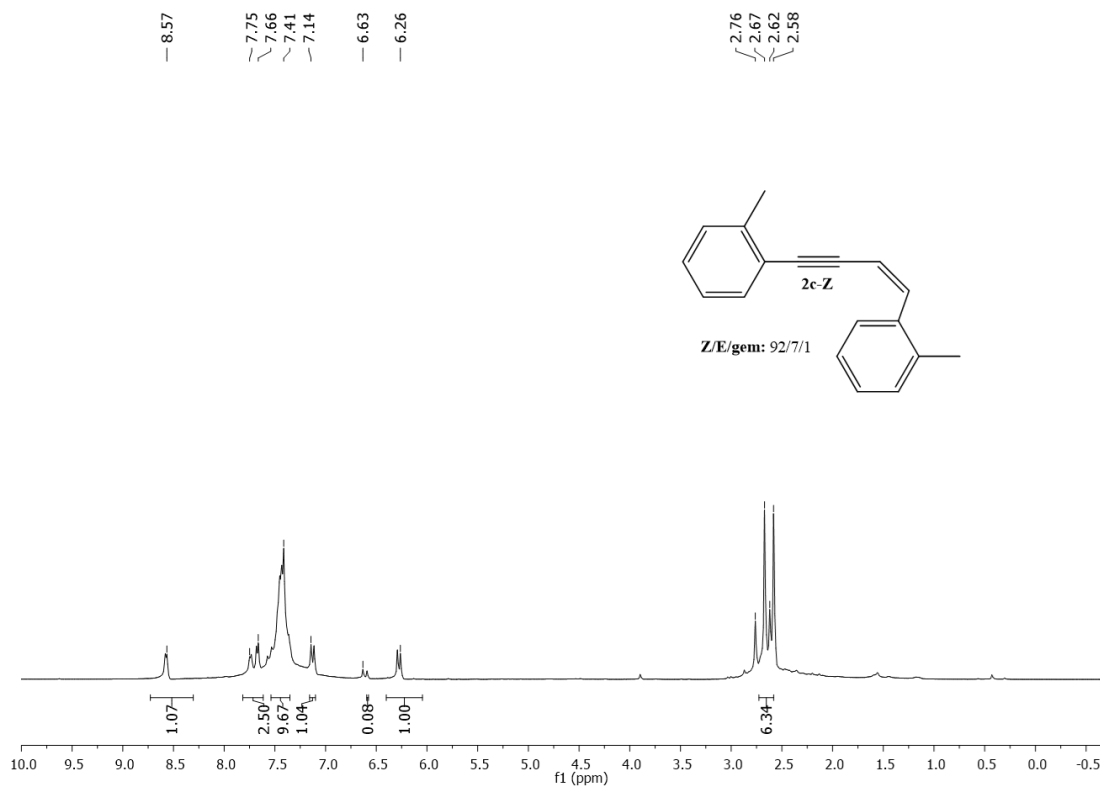


Figure S5. ^1H NMR spectrum of **2c** (400 MHz, CDCl_3) (Table 3, Entry 11)

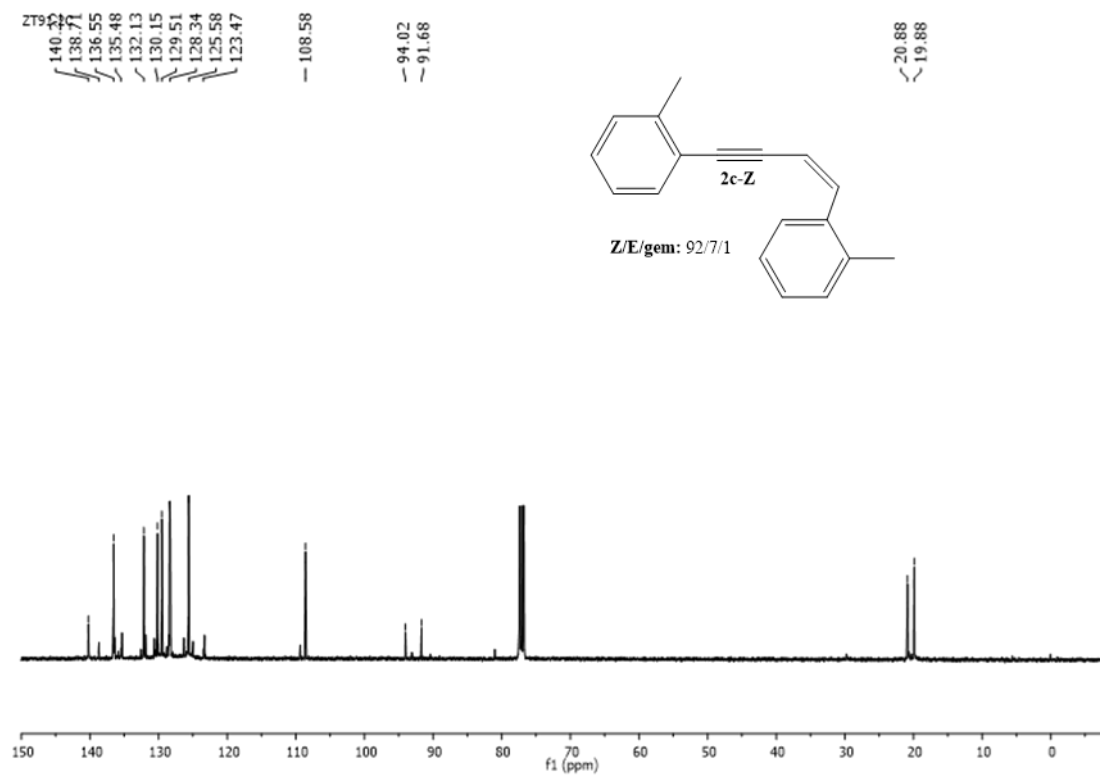


Figure S6. ^{13}C NMR spectrum of **2c** (100 MHz, CDCl_3) (Table 3, Entry 11)

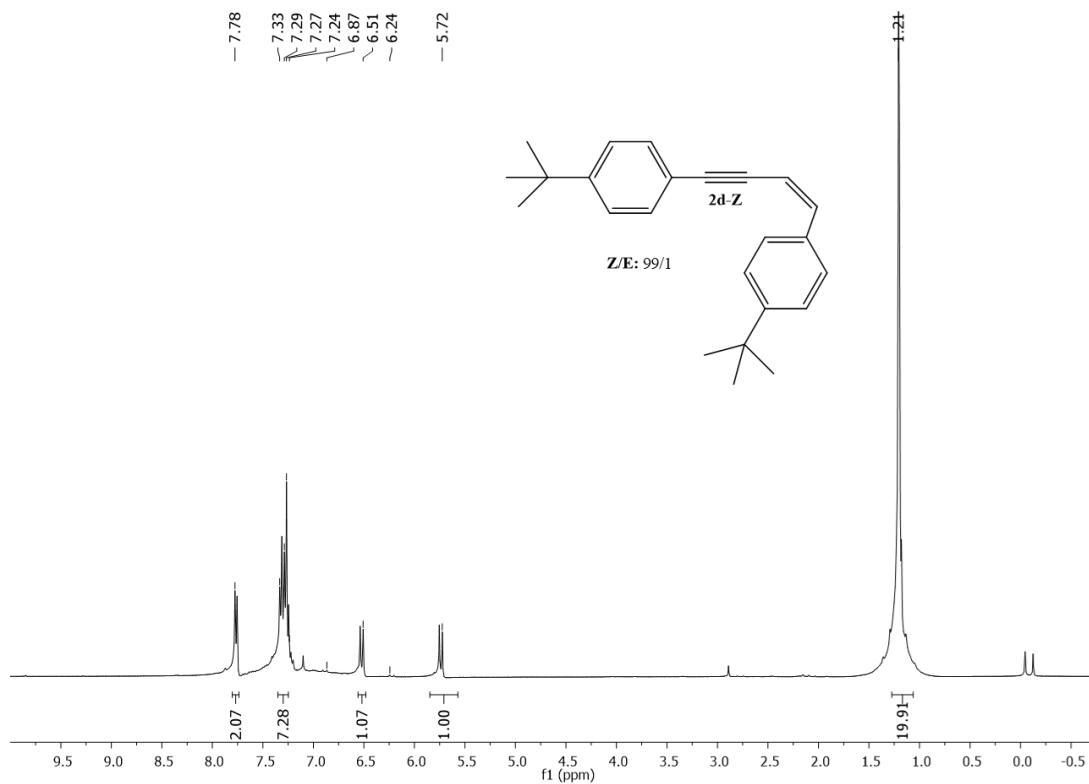


Figure S7. ^1H NMR spectrum of 2d (400 MHz, CDCl_3) (Table 3, Entry 14)

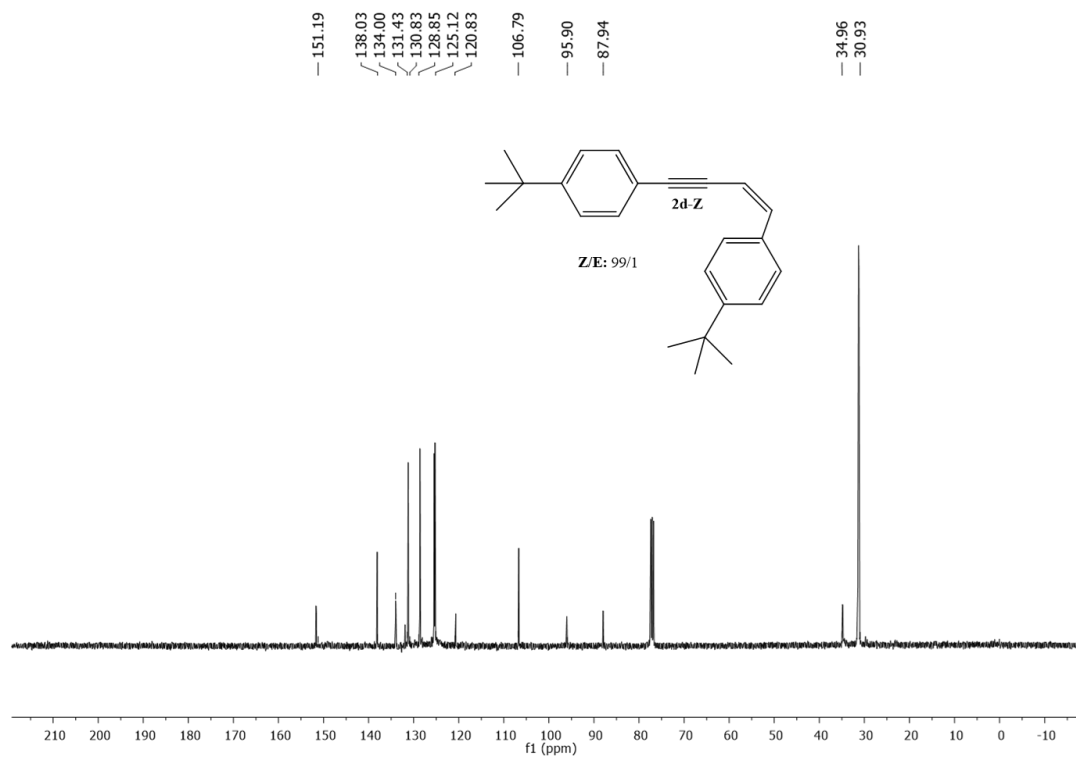


Figure S8. ^{13}C NMR spectrum of 2d (100 MHz, CDCl_3) (Table 3, Entry 14)

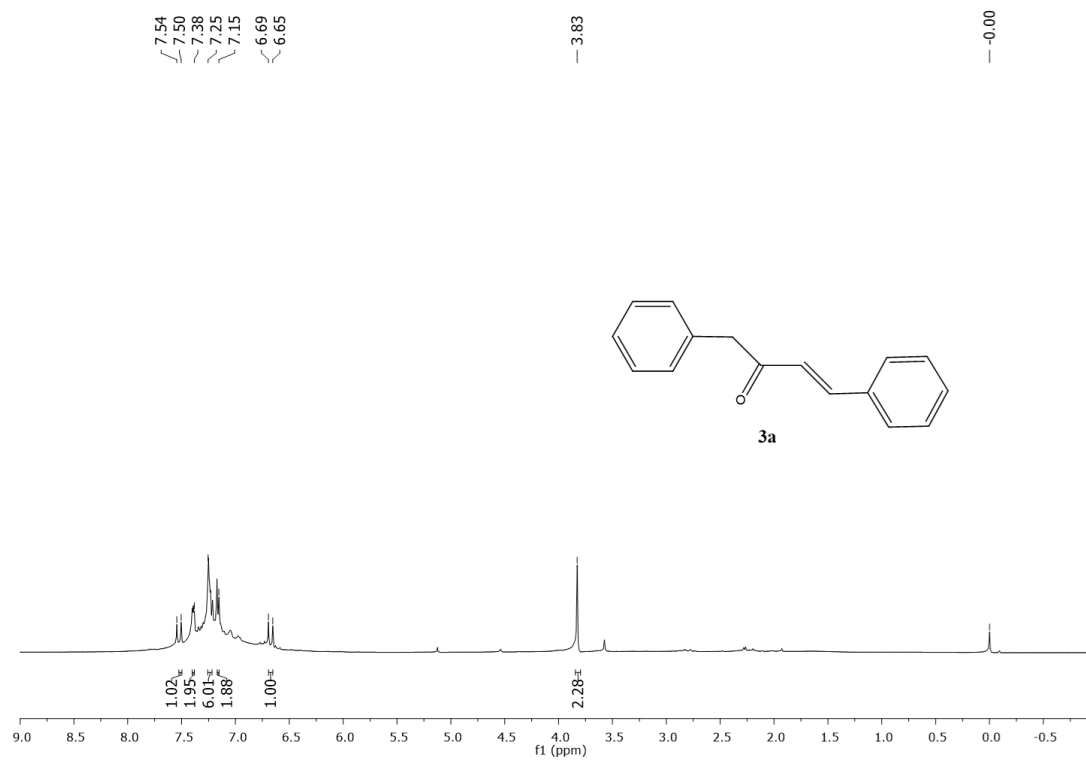


Figure S9. ¹H NMR spectrum of **3a** (400 MHz, CDCl₃)

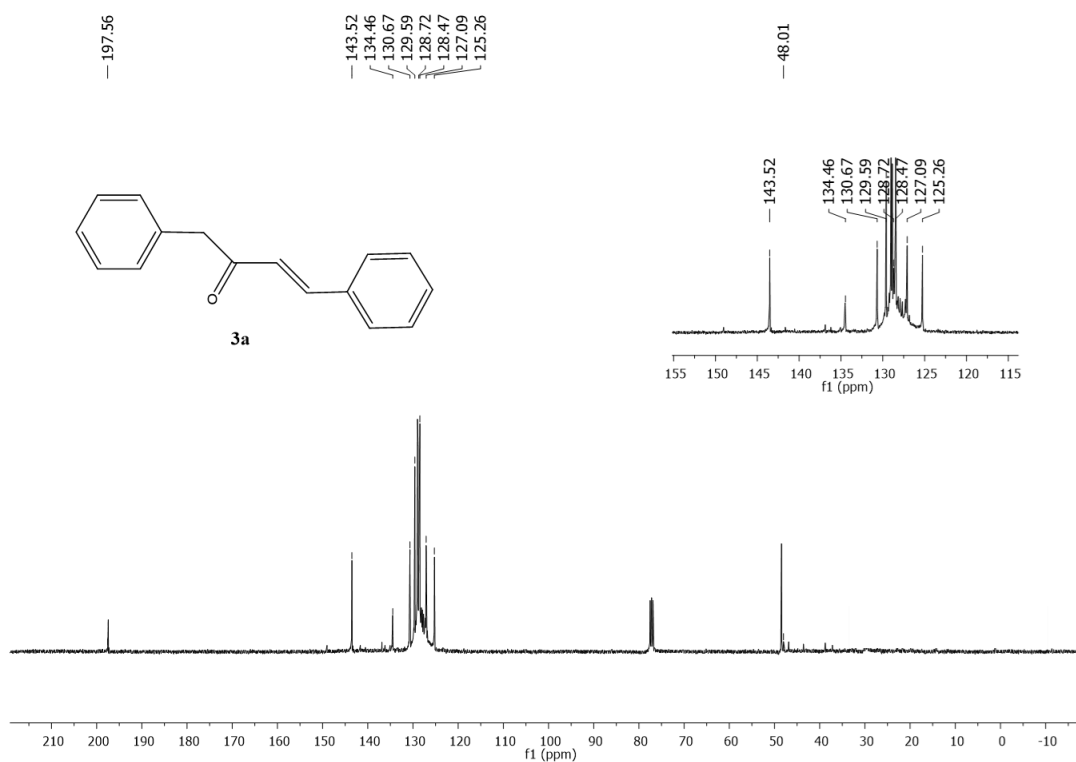


Figure S10. ¹³C NMR spectrum of **3a** (100 MHz, CDCl₃)

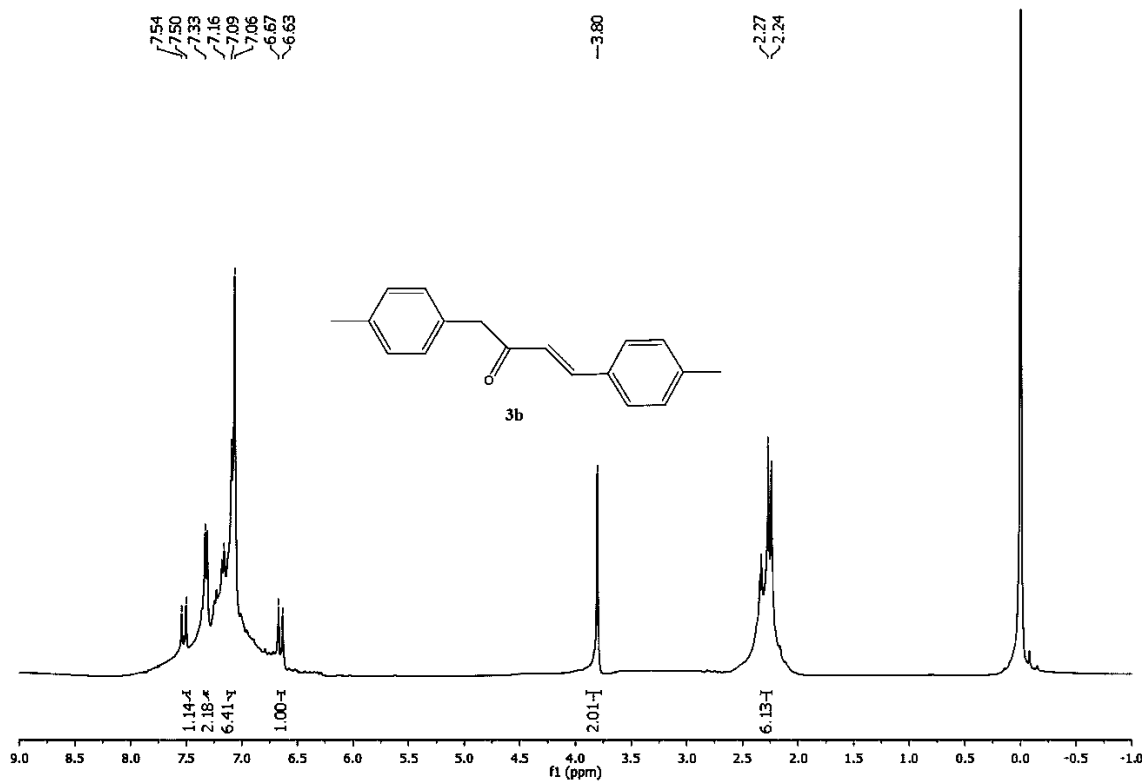


Figure S11. ¹H NMR spectrum of 3b (400 MHz, CDCl₃)

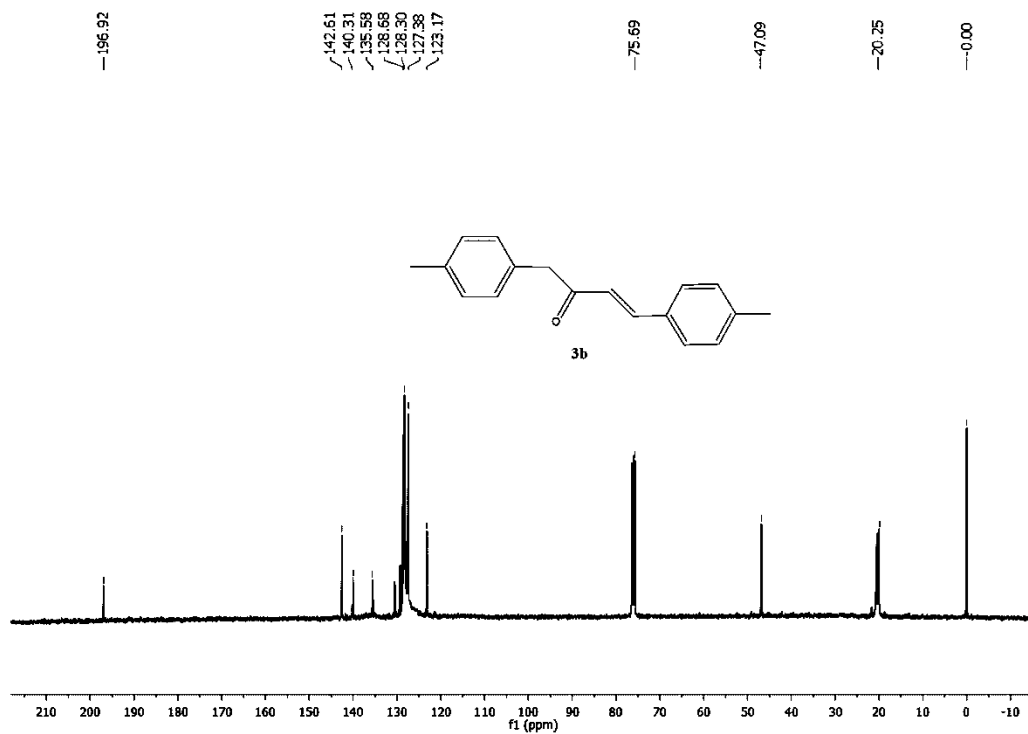


Figure S12. ¹³C NMR spectrum of 3b (400 MHz, CDCl₃)

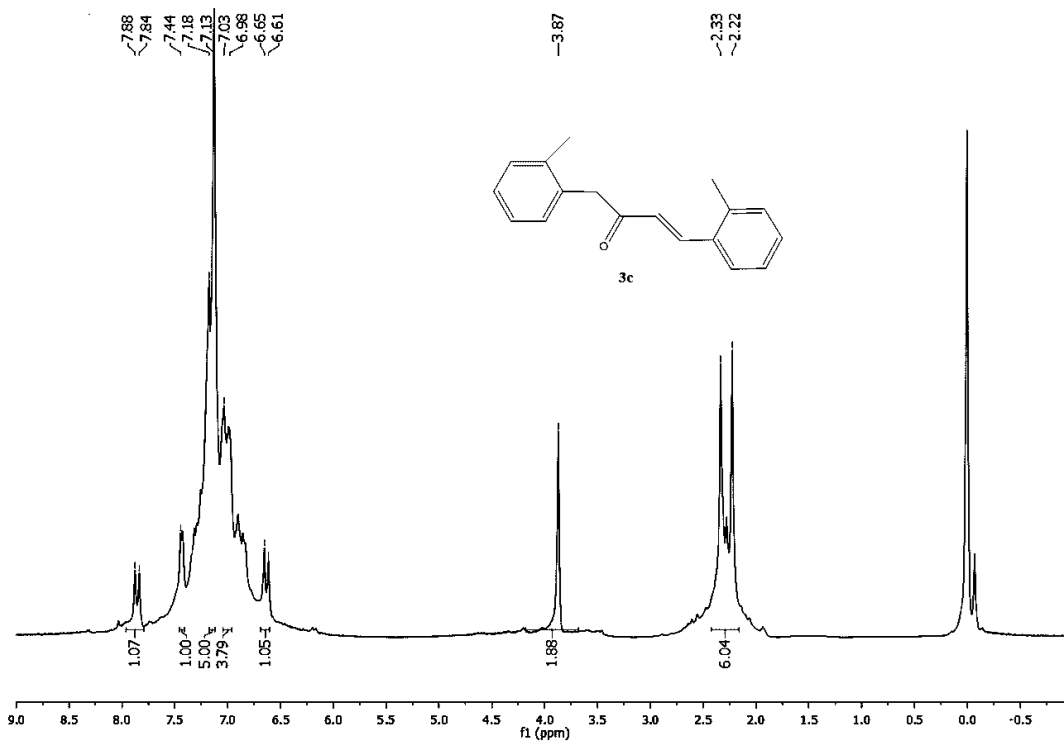


Figure S13. ¹H NMR spectrum of 3c (400 MHz, CDCl₃)

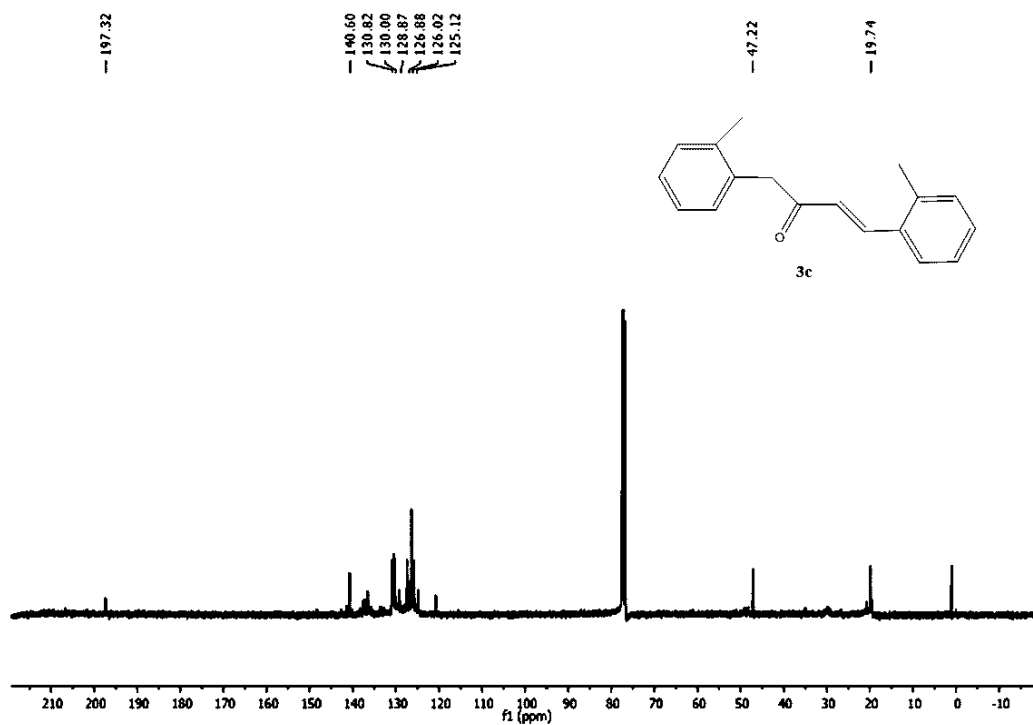


Figure S14. ¹³C NMR spectrum of 3c (400 MHz, CDCl₃)

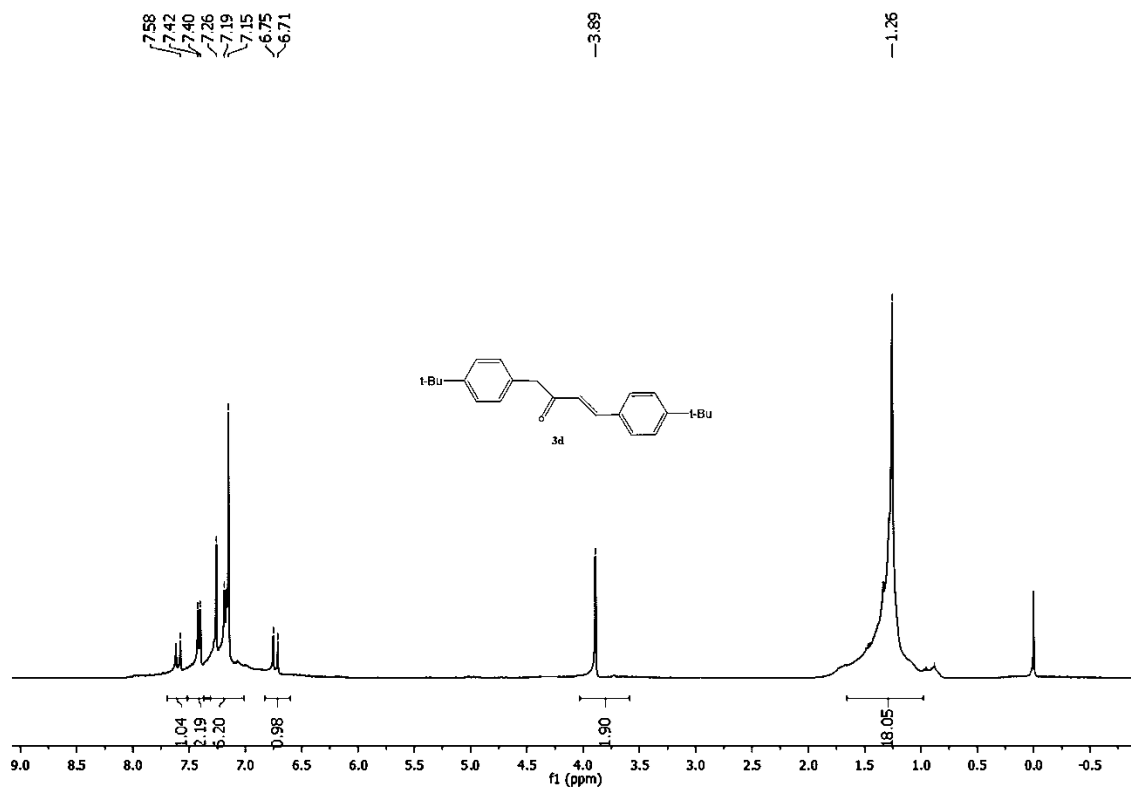


Figure S15. ¹H NMR spectrum of 3d (400 MHz, CDCl₃)

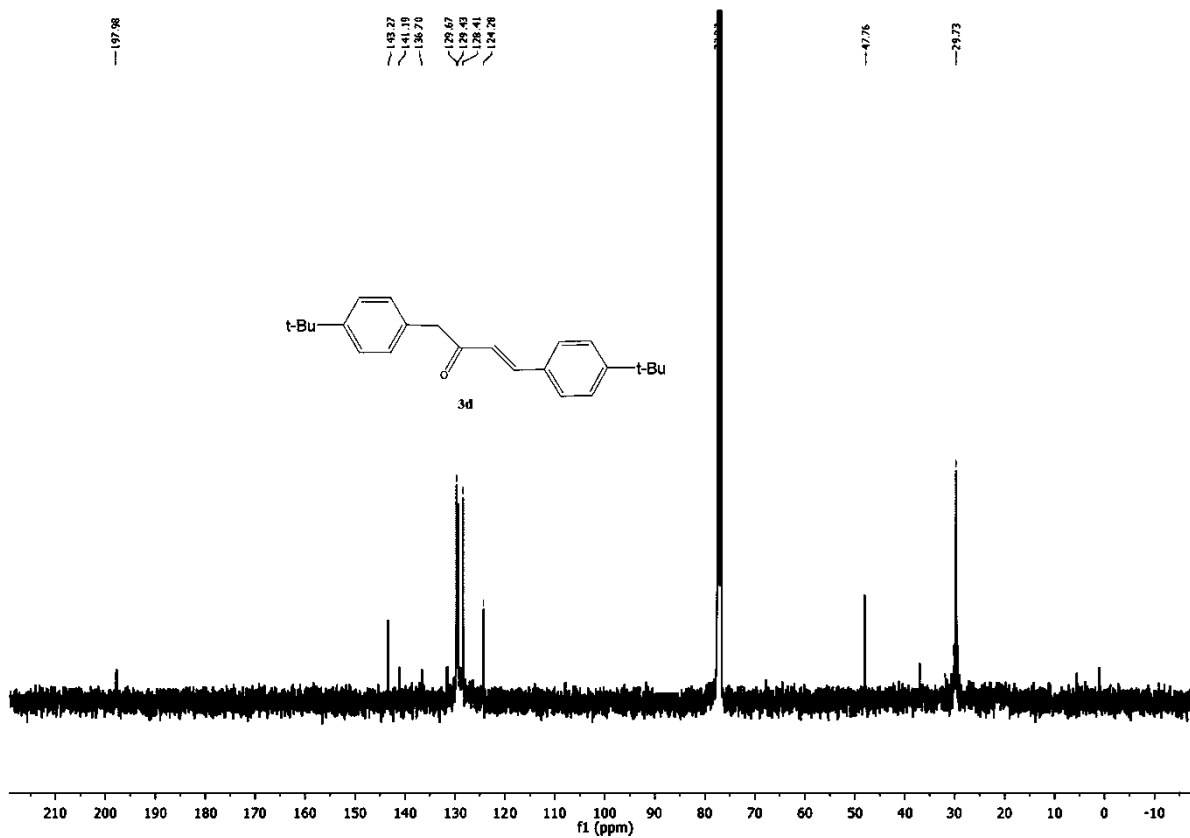


Figure S16. ¹³C NMR spectrum of 3d (400 MHz, CDCl₃)