

Electronic Supplementary Material (ESI) for New Journal of Chemistry.

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Supplementary Information

Nickel-catalyzed electrocarboxylation of allylic halides with CO₂

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1. Materials and Instrument

Galvanostatic electrosynthesis was performed using a digital direct current-regulated power supply (HY3005MT, HYelec® China). Voltammetric measurements were conducted using electrochemical station (CHI660E, Chenhua) in a conventional three-electrode cell. The product yield was determined by high-performance liquid chromatography (HPLC) instrument (DIONEX Ultimate 3000 pump) (Thermo Scientific, Germering, Germany) equipped with a UV (RS Variable Wavelength) (Thermo Scientific, Germering, Germany) detector. ^1H NMR spectra were recorded on an AVANCE 500 (500 MHz, Bruker, Germany) spectrometer in CDCl_3 with Me_4Si as an internal standard. All substrates, vinylacetic acid (**2d**) were used as received (Energy chemical, China). MeCN were kept over 4 Å molecular sieves.

2. Infrared characterization of Nickel(II) Complexes

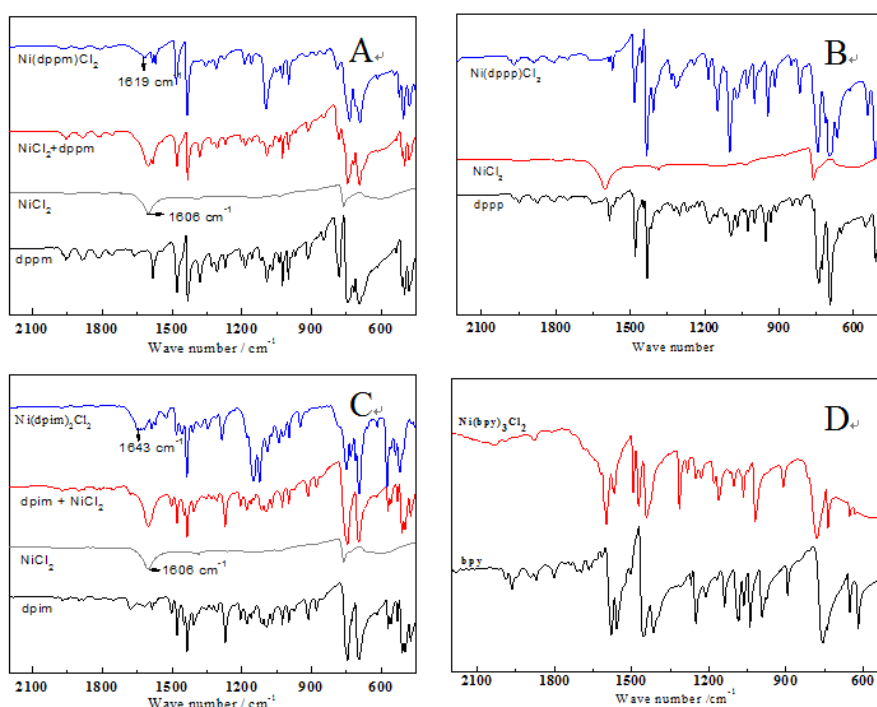


Fig.S1 IR of $\text{Ni}(\text{dppm})\text{Cl}_2$ (A); $\text{Ni}(\text{dppp})\text{Cl}_2$ (B); $\text{Ni}(\text{dpim})_2\text{Cl}_2$ (C); $\text{Ni}(\text{bpy})_3\text{Cl}_2$ (D).

3. Electrochemical behavior of $\text{Ni}(\text{dppm})\text{Cl}_2$.

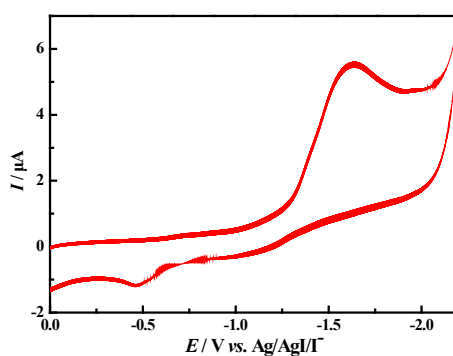
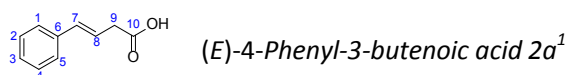
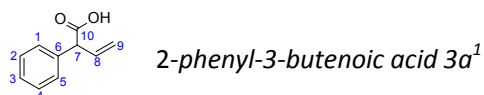


Fig. S2 CV of $\text{Ni}(\text{dppm})\text{Cl}_2$ in DMF-TEAI solution at 10 °C saturated with N_2 on GC electrode at a sweep rate of 100 $\text{mV} \cdot \text{s}^{-1}$.

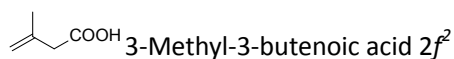
4. NMR characterization



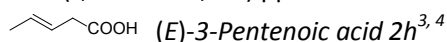
^1H NMR (500 MHz, CDCl_3) δ 7.43-7.38 (m, 2H), 7.34 (t, $J = 7.6$ Hz, 2H), 7.28-7.24 (m, 1H), 6.55 (d, $J = 15.9$ Hz, 1H), 6.31 (dt, $J = 15.9, 7.1$ Hz, 1H), 3.33 (dd, $J = 7.1, 1.4$ Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 178.24 (C-10), 136.66 (C-6), 134.05 (C-7), 128.60 (C-2, C-4), 127.74 (C-3), 126.37 (C-1, C-5), 120.80 (C-8), 38.08 (C-9) ppm.



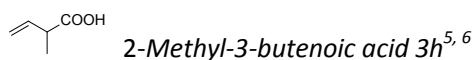
^1H NMR (500 MHz, CDCl_3) δ 7.44 -7.29 (m, 5H), 6.24 (ddd, $J = 17.7, 10.2, 8.0$ Hz, 1H), 5.40-5.05 (m, 2H), 4.36 (d, $J = 8.0$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 178.71 (C-10), 137.40 (C-6), 135.00 (C-8), 128.86 (C-2, C-4), 128.15 (C-1, C-5), 127.67 (C-3), 118.15 (C-9), 55.58 (C-7) ppm.



^1H NMR (500 MHz, CDCl_3) δ 4.98 (p, $J = 1.6$ Hz, 1H), 4.91 (t, $J = 1.5$ Hz, 1H), 3.11 (d, $J = 1.2$ Hz, 2H), 1.86 (s, $J = 1.1$ Hz, 3H) ppm.



^1H NMR (400 MHz, CDCl_3) δ 9.55 (s, 1H), 5.82-5.40 (m, 2H), 3.12 (dd, $J = 6.9, 6.6$ Hz, 2H), 1.79-1.56 (m, 3H) ppm.



^1H NMR (400 MHz, CDCl_3) δ 10.69 (s, 1H), 5.94 (ddd, $J = 17.4, 10.3, 7.4$ Hz, 1H), 5.27-5.09 (m, 2H), 3.19 (pt, $J = 7.1, 1.2$ Hz, 1H), 1.31 (d, $J = 7.0$ Hz, 3H) ppm.

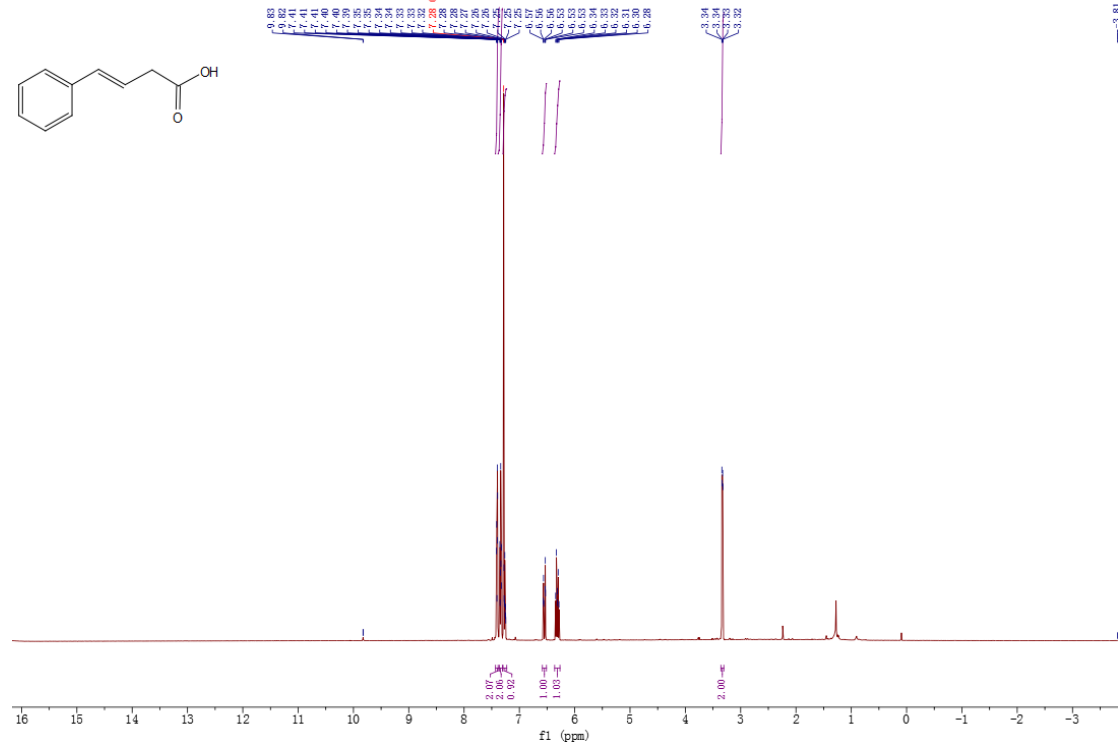


^1H NMR (500 MHz, CDCl_3) δ 6.07 (dd, $J = 10.0, 10.0$ Hz, 1H), 5.19-5.12 (m, 2H), 1.35 (s, 6H) ppm.



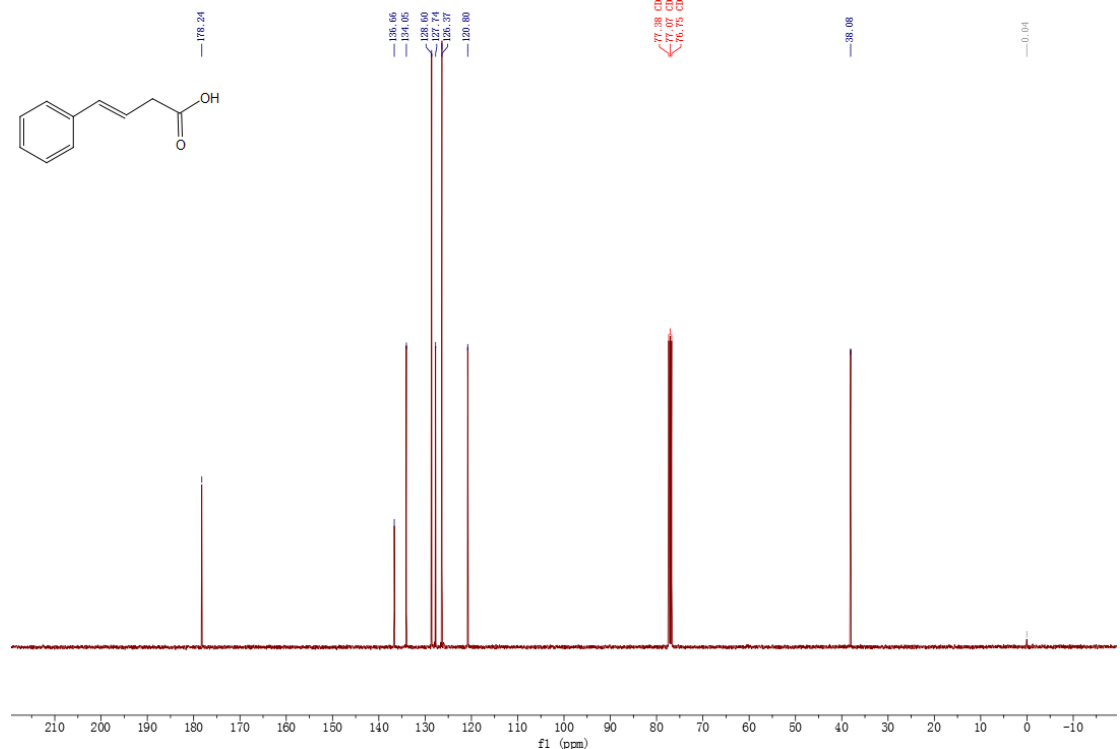
^1H NMR (500 MHz, CDCl_3) δ 5.33-5.29 (m, 1H), 3.10 (d, $J = 10.0$ Hz, 2H), 1.77 (d, $J = 5.0$ Hz, 3H), 1.66 (d, $J = 5.0$ Hz, 3H).

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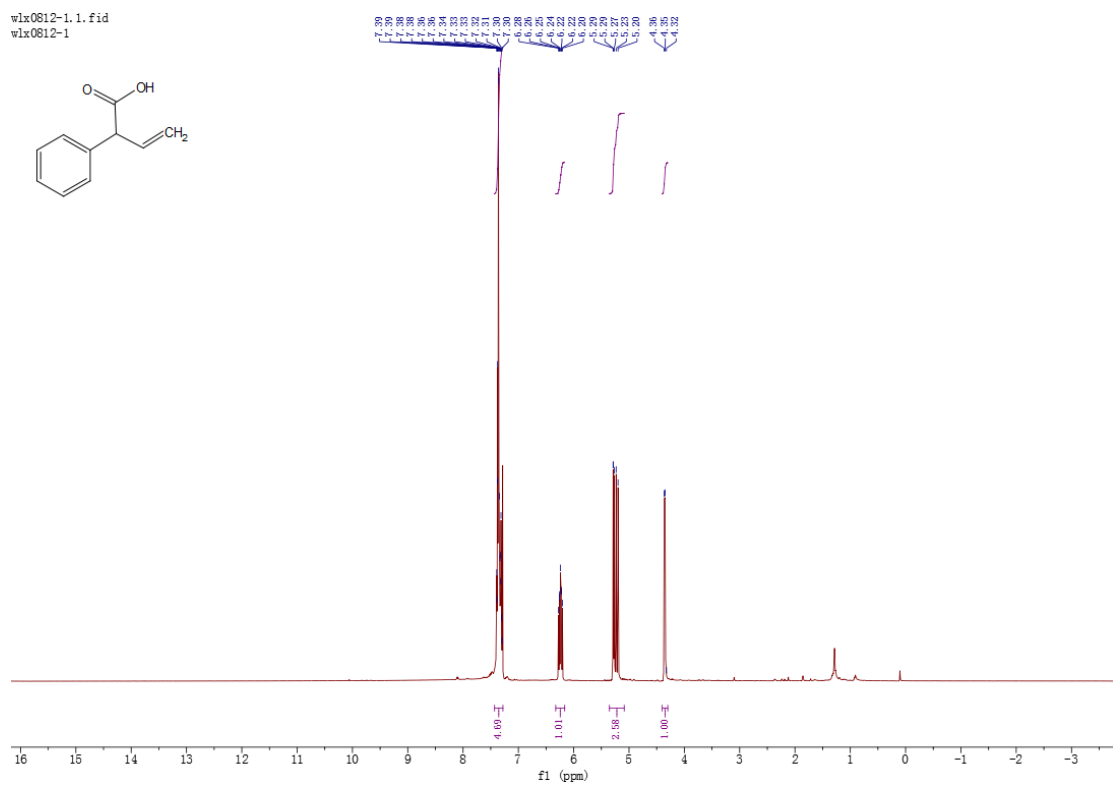
¹H NMR (500 MHz, CDCl₃) of (E)-phenyl-3-butenic acid **2a**

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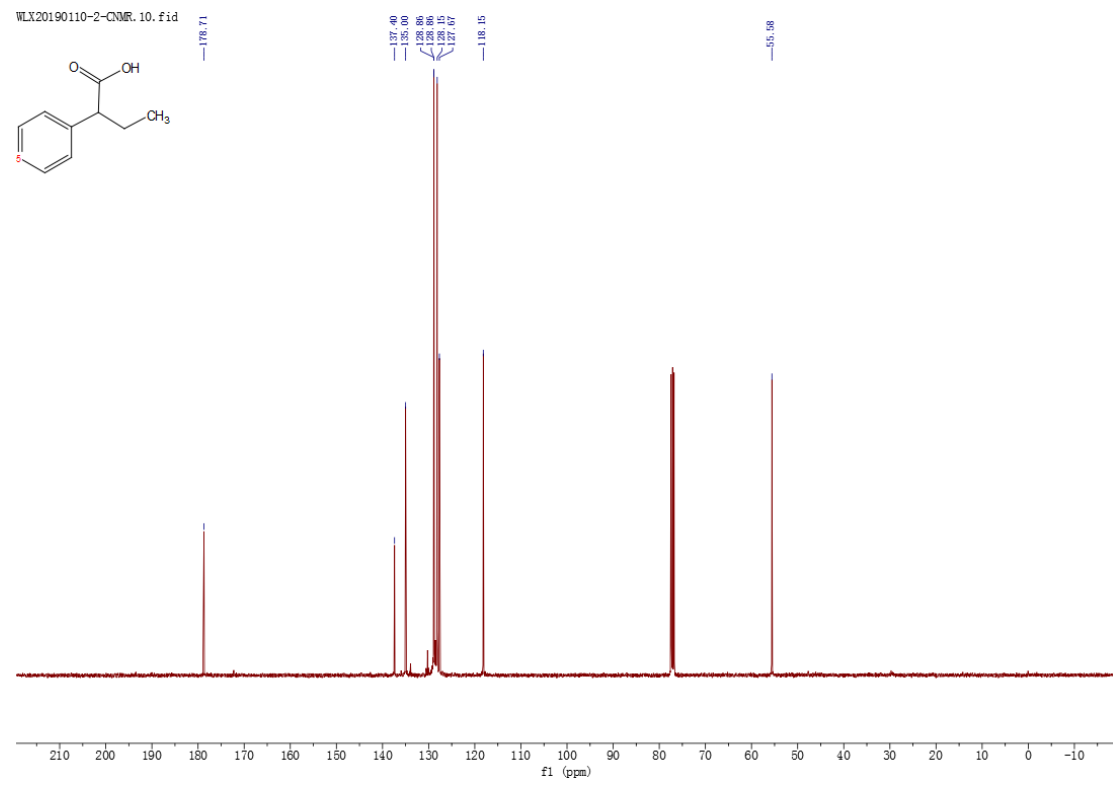
¹³C NMR (101 MHz, CDCl₃) of (E)-phenyl-3-butenic acid **2a**

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wlx0812-1



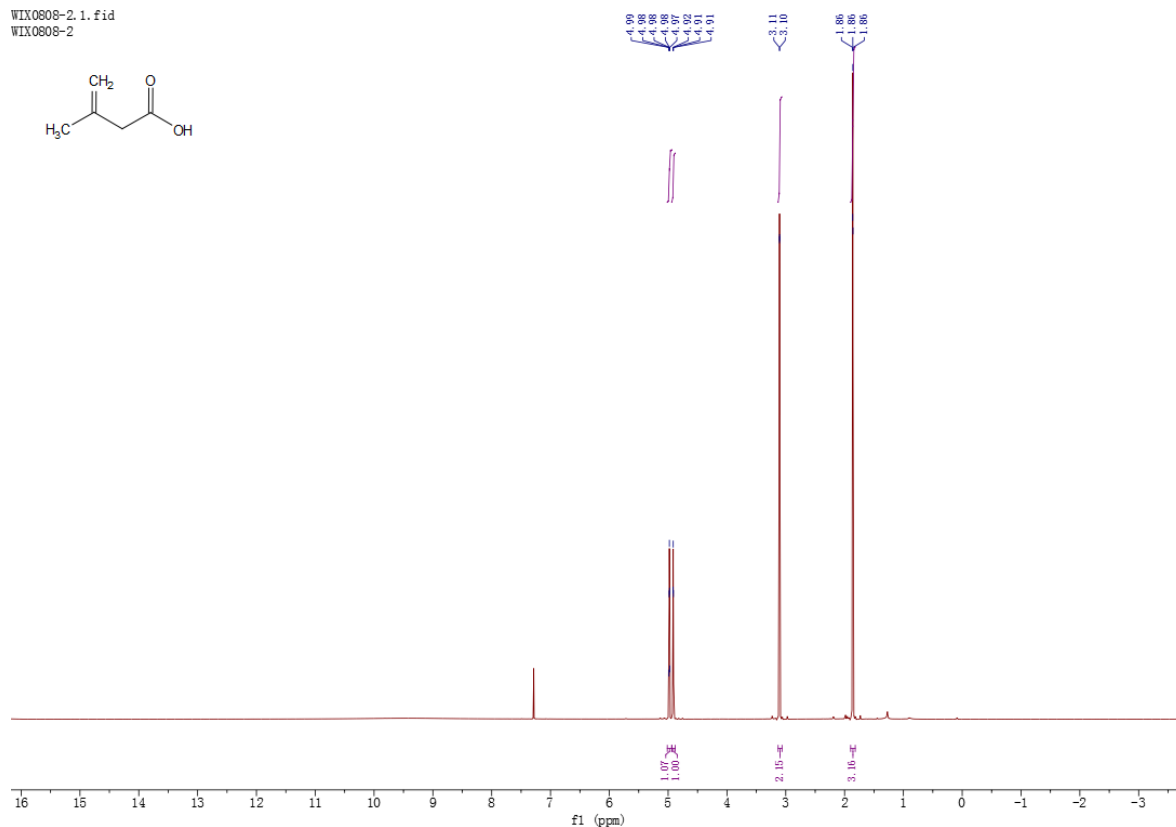
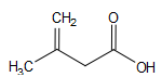
¹H NMR (500 MHz, CDCl₃) of 2-phenyl-3-butenoic acid **3a**

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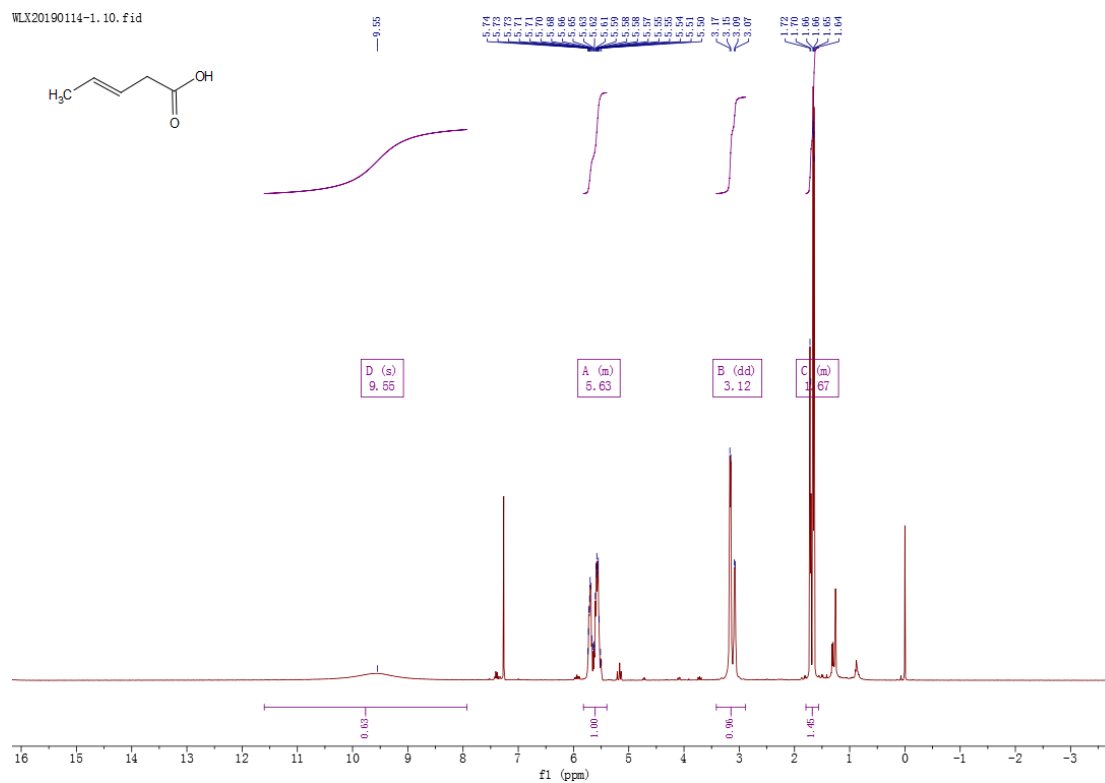
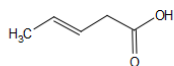
¹³C NMR (101 MHz, CDCl₃) of 2-phenyl-3-butenoic acid **3a**

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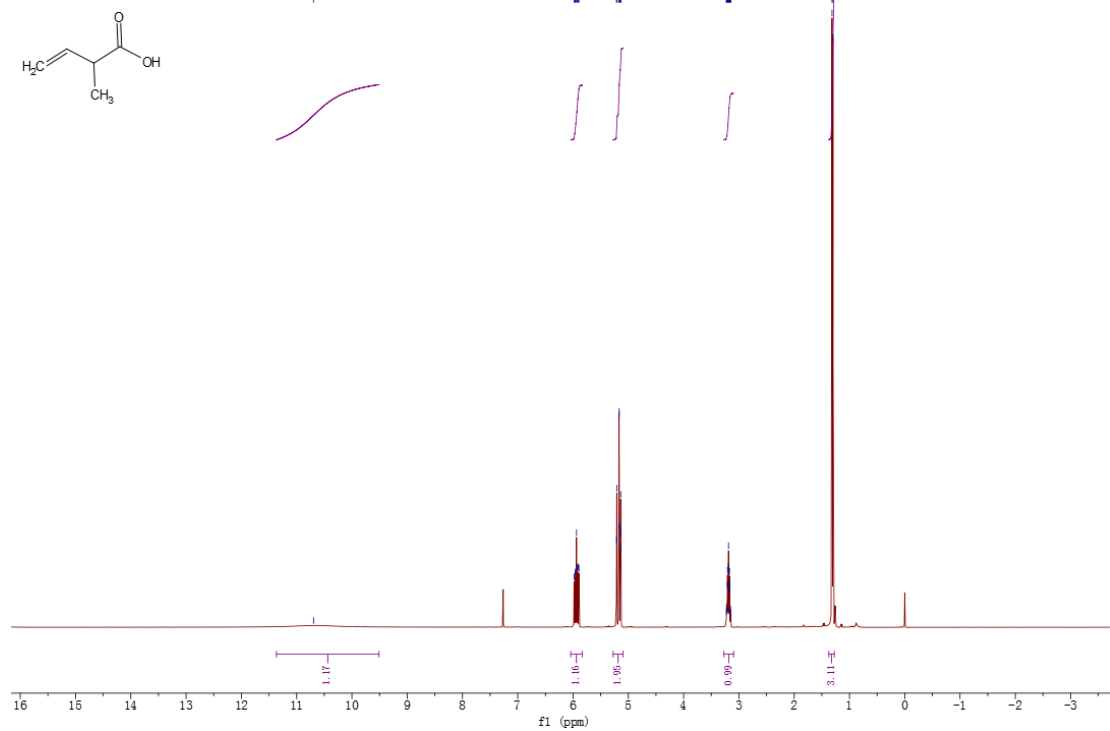
¹H NMR (500 MHz, CDCl₃) of 3-Methyl-3-butenoic acid **2f**

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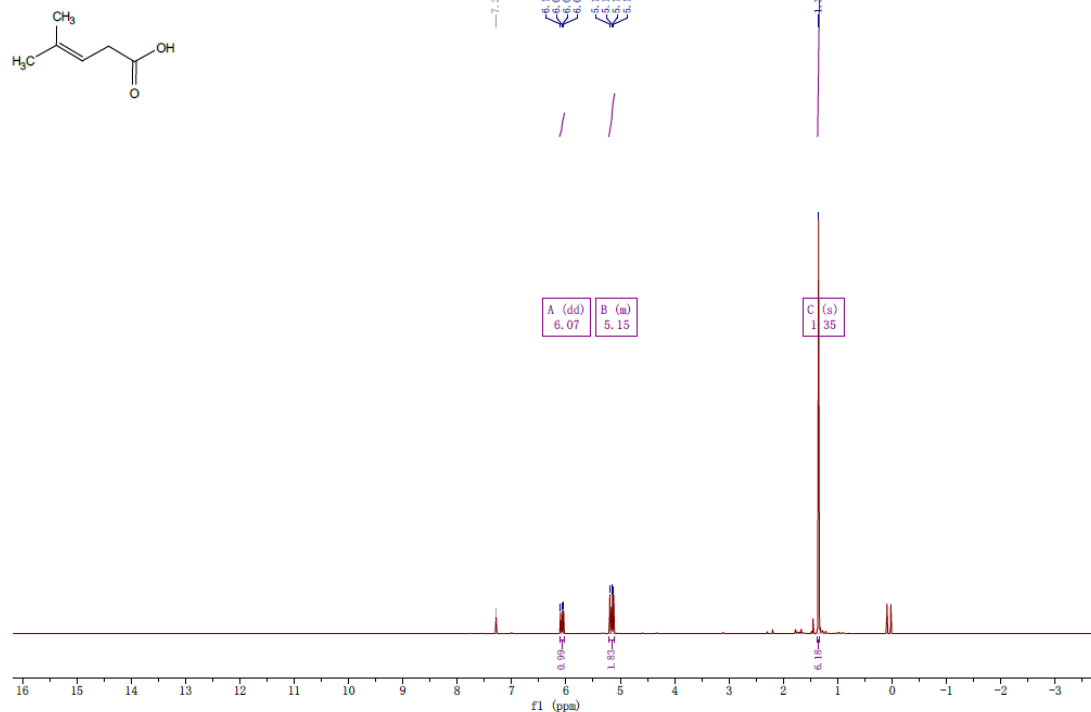
¹H NMR (400 MHz, CDCl₃) of (*E*)-3-pentenoic acid **2h**

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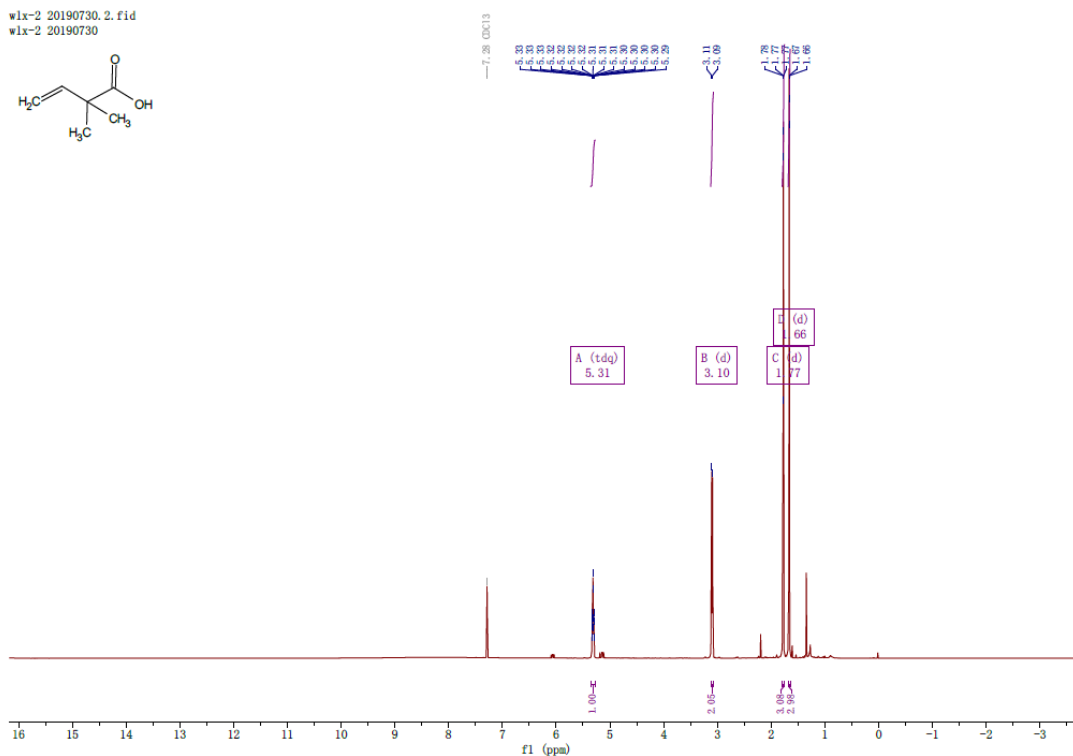
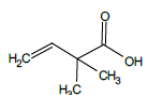
¹HNMR (400 MHz, CDCl₃) of 2-Methyl-3-butenoic acid **3h**

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wlx-1 20190730



¹HNMR (500 MHz, CDCl₃) of 4-methylpent-3-enoic acid **2j**

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wlx-2 20190730



¹HNMR (500 MHz, CDCl₃) of 2,2-dimethylbut-3-enoic acid 3j

Reference

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