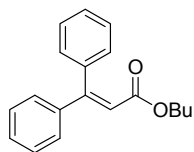


Supplementary Materials: Article A Reusable Palladium/Cationic 2,2'-bipyridyl System-Catalyzed Double Mizoroki-Heck Reaction in Water

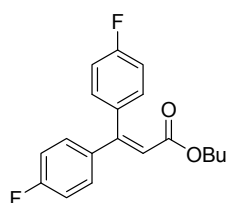
Yu-Chi Chen, Chien-Chi Wu, Wei-Ting Liao, Ling-Jun Liu and Fu-Yu Tsai

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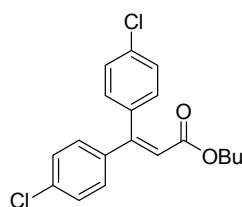
(1) ^1H and ^{13}C NMR spectral data of double Mizoroki-Heck products	S2
(2) References	S18
(3) Copies of ^1H and ^{13}C NMR spectra for unknown and unsymmetrical double Mizoroki-Heck compounds	S19

¹H and ¹³C NMR spectral data of double Mizoroki-Heck products

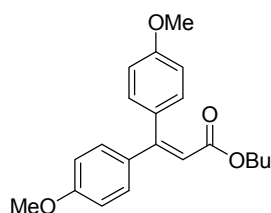
Butyl 3,3-diphenylacrylate (3a [1], Table 1, Entry 1). Pale yellow oil. ¹H NMR (300 MHz, CDCl₃): δ 0.86 (t, *J* = 7.3 Hz, 3H), 1.16–1.29 (m, 2H), 1.41–1.50 (m, 2H), 4.00 (t, *J* = 6.6 Hz, 2H), 6.37 (s, 1H), 7.19–7.40 (m, 10H); ¹³C NMR (75 MHz, CDCl₃): δ 13.7, 19.0, 30.4, 64.0, 117.5, 127.8, 128.0, 128.2, 128.3, 129.0, 129.3, 138.9, 140.8, 156.3, 166.2.



Butyl 3,3-bis(4-fluorophenyl)acrylate (3b [1], Table 1, Entry 2). Pale yellow oil. ¹H NMR (300 MHz, CDCl₃): δ 0.87 (t, *J* = 7.3 Hz, 3H), 1.25 (m, 2H), 1.39–1.57 (m, 2H), 4.01 (t, *J* = 6.6 Hz, 2H), 6.31 (s, 1H), 6.95–7.12 (m, 4H), 7.12–7.22 (m, 2H), 7.22–7.30 (m, 2H); ¹³C NMR (75 MHz, CDCl₃): δ 13.5, 19.0, 30.4, 64.1, 115.0 (*J*_{C-F} = 21.5 Hz), 115.4 (*J*_{C-F} = 21.5 Hz), 117.5, 130.1 (*J*_{C-F} = 9.0 Hz), 131.0 (*J*_{C-F} = 8.3 Hz), 134.5 (*J*_{C-F} = 3.8 Hz), 136.8 (*J*_{C-F} = 3.0 Hz), 154.2, 162.7 (*J*_{C-F} = 246.8 Hz), 163.5 (*J*_{C-F} = 249.0 Hz), 166.

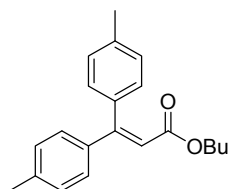


Butyl 3,3-bis(4-chlorophenyl)acrylate (3c, Table 1, Entry 3). Pale yellow oil. ¹H NMR (300 MHz, CDCl₃): δ 0.87 (t, *J* = 7.3 Hz, 3H), 1.17–1.29 (m, 2H), 1.42–1.52 (m, 2H), 4.00 (t, *J* = 6.6 Hz, 2H), 6.33 (s, 1H), 7.12 (d, *J* = 8.5 Hz, 2H), 7.19 (d, *J* = 8.7 Hz, 2H), 7.28 (d, *J* = 8.7 Hz, 2H), 7.35 (d, *J* = 8.5 Hz, 2H); ¹³C NMR (75 MHz, CDCl₃): δ 13.6, 19.0, 30.4, 64.2, 118.2, 128.3, 128.7, 129.4, 130.5, 134.4, 135.7, 136.8, 138.8, 153.8, 165.7. HRMS calcd for C₁₉H₁₈Cl₂O₂, 348.0684; found, 348.0682.

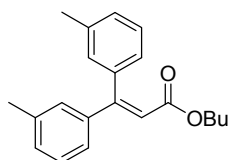


Butyl 3,3-bis(4-methoxyphenyl)acrylate (3d [1], Table 1, Entry 4). Pale yellow oil. ¹H NMR (300 MHz,

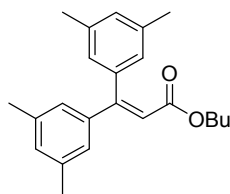
CDCl₃): δ 0.87 (t, J = 7.3 Hz, 3H), 1.19–1.31 (m, 2H), 1.44–1.54 (m, 2H), 3.79 (s, 3H), 3.82 (s, 3H), 4.01 (t, J = 6.6 Hz, 2H), 6.22 (s, 1H), 6.82 (d, J = 8.9 Hz, 2H), 6.89 (d, J = 8.9 Hz, 2H), 7.14 (d, J = 8.9 Hz, 2H), 7.23 (d, J = 8.9 Hz, 2H); ¹³C NMR (75 MHz, CDCl₃): δ 13.7, 19.1, 30.6, 55.2, 55.3, 63.8, 113.2, 113.6, 114.9, 129.9, 130.8, 131.3, 133.8, 156.2, 159.6, 160.7, 166.6.



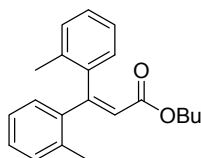
Butyl 3,3-di-*p*-tolylacrylate (3e [1], Table 1, Entry 5). Pale yellow oil. ¹H NMR (300 MHz, CDCl₃): δ 0.86 (t, J = 7.3 Hz, 3H), 1.17–1.29 (m, 2H), 1.42–1.52 (m, 2H), 2.34 (s, 3H), 2.38 (s, 3H), 4.00 (t, J = 6.6 Hz, 2H), 6.30 (s, 1H), 7.08–7.24 (m, 8H); ¹³C NMR (75 MHz, CDCl₃): δ 13.7, 19.1, 21.2, 21.4, 30.5, 63.9, 116.2, 128.3, 128.5, 129.0, 129.1, 136.1, 137.8, 138.2, 139.5, 156.7, 166.4.



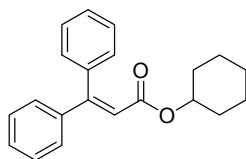
Butyl 3,3-di-*m*-tolylacrylate (3f, Table 1, Entry 6). Pale yellow oil. ¹H NMR (300 MHz, CDCl₃): δ 0.88 (t, J = 7.3 Hz, 3H), 1.18–1.30 (m, 2H), 1.43–1.52 (m, 2H), 2.33 (s, 3H), 2.37 (s, 3H), 4.02 (t, J = 6.6 Hz, 2H), 6.35 (s, 1H), 7.02–7.31 (m, 8H); ¹³C NMR (75 MHz, CDCl₃): δ 13.6, 19.0, 21.3, 21.4, 30.5, 63.9, 117.2, 125.5, 126.2, 127.6, 128.1, 128.7, 129.5, 130.0, 137.3, 137.8, 139.0, 140.9, 156.6, 166.3. HRMS calcd for C₂₁H₂₄O₂, 308.1776; found, 308.1776.



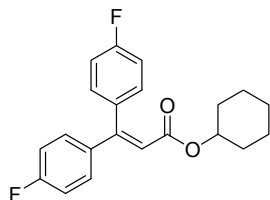
Butyl 3,3-bis(3,5-dimethylphenyl)acrylate (3g, Table 1, Entry 7). Pale yellow oil. ¹H NMR (300 MHz, CDCl₃): δ 0.88 (t, J = 7.3 Hz, 3H), 1.17–1.30 (m, 2H), 1.42–1.51 (m, 2H), 2.30 (s, 6H), 2.33 (s, 6H), 4.01 (t, J = 6.5 Hz, 2H), 6.30 (s, 1H), 6.83 (s, 2H), 6.94 (s, 2H), 7.01 (s, 2H); ¹³C NMR (75 MHz, CDCl₃): δ 13.7, 19.0, 21.3, 21.2, 30.5, 63.9, 117.1, 126.1, 126.7, 129.6, 130.9, 137.0, 137.7, 139.0, 141.1, 156.9, 166.4. HRMS calcd for C₂₃H₂₈O₂, 336.2089; found, 336.2084.



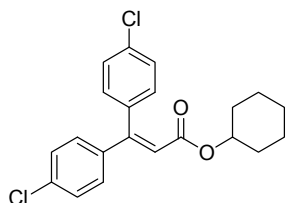
Butyl 3,3-di-*o*-tolylacrylate (3h, Table 1, Entry 8). Pale yellow oil. ^1H NMR (300 MHz, CDCl_3): δ 0.87 (t, $J = 7.3$ Hz, 3H), 1.18–1.29 (m, 2H), 1.40–1.49 (m, 2H), 2.17 (s, 3H), 2.35 (s, 3H), 4.01 (t, $J = 6.6$ Hz, 2H), 6.15 (s, 1H), 7.09–7.23 (m, 8H); ^{13}C NMR (75 MHz, CDCl_3): δ 13.6, 19.0, 19.8, 20.8, 30.4, 64.0, 122.2, 125.1, 125.6, 127.8, 128.2, 129.0, 129.7, 129.9, 131.1, 135.4, 135.6, 139.5, 140.6, 155.9, 166.0. HRMS calcd for $\text{C}_{21}\text{H}_{24}\text{O}_2$, 308.1776; found, 308.1781.



Cyclohexyl 3,3-diphenylacrylate (4a [2], Table 2, Entry 1). Pale yellow oil. ^1H NMR (300 MHz, CDCl_3): δ 1.13–1.33 (m, 5H), 1.45–1.46 (m, 1H), 1.49–1.61 (m, 2H), 1.63–1.76 (m, 2H), 4.69 (m, 1H), 6.36 (s, 1H), 7.19–7.38 (m, 10H); ^{13}C NMR (75 MHz, CDCl_3): δ 23.7, 25.3, 31.4, 72.5, 118.1, 127.8, 128.0, 128.2, 128.3, 129.1, 129.2, 139.1, 140.8, 155.8, 165.6.

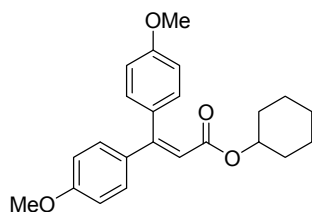


Cyclohexyl 3,3-bis(4-fluorophenyl)acrylate (4b, Table 2, Entry 2). Pale yellow solid (m.p. = 83–85 °C). ^1H NMR (300 MHz, CDCl_3): δ 1.15–1.30 (m, 5H), 1.46–1.50 (m, 1H), 1.61 (m, 2H), 1.72–1.78 (m, 2H), 4.66–4.72 (m, 1H), 6.29 (s, 1H), 6.96–7.09 (m, 4H), 7.14–7.27 (m, 4H); ^{13}C NMR (75 MHz, CDCl_3): δ 23.7, 25.2, 31.4, 72.6, 115.0 ($J_{\text{C-F}} = 21.5$ Hz), 115.4 ($J_{\text{C-F}} = 21.5$ Hz), 118.1, 130.0 ($J_{\text{C-F}} = 8.4$ Hz), 130.9 ($J_{\text{C-F}} = 8.1$ Hz), 134.6 ($J_{\text{C-F}} = 3.4$ Hz), 136.8 ($J_{\text{C-F}} = 3.0$ Hz), 153.7, 162.6 ($J_{\text{C-F}} = 246.1$ Hz), 163.4 ($J_{\text{C-F}} = 248.8$ Hz), 165.3. HRMS calcd for $\text{C}_{21}\text{H}_{20}\text{F}_2\text{O}_2$, 342.1431; found, 342.1433.

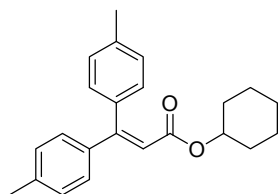


Cyclohexyl 3,3-bis(4-chlorophenyl)acrylate (4c, Table 2, Entry 3). Pale yellow solid (m.p. = 102–104 °C). ^1H NMR (300 MHz, CDCl_3): δ 1.24–1.27 (m, 5H), 1.47–1.50 (m, 1H), 1.61 (m, 2H), 1.71–1.74 (m,

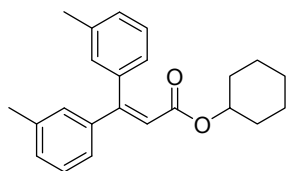
2H), 4.68–4.70 (m, 1H), 6.32 (s, 1H), 7.10–7.20 (m, 4H), 7.26–7.36 (m, 4H); ^{13}C NMR (75 MHz, CDCl_3): δ 23.7, 25.3, 31.5, 72.8, 118.8, 128.3, 128.7, 129.4, 130.5, 134.3, 135.6, 136.9, 138.9, 153.4, 165.2. HRMS calcd for $\text{C}_{21}\text{H}_{20}\text{Cl}_2\text{O}_2$, 374.0840; found, 374.0848.



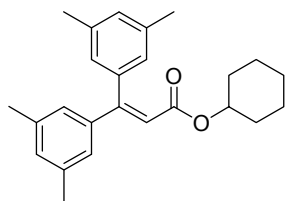
Cyclohexyl 3,3-bis(4-methoxyphenyl)acrylate (4d), Table 2, Entry 4). Pale yellow solid (m.p. = 82–84 °C). ^1H NMR (300 MHz, CDCl_3): δ 1.23–1.33 (m, 5H), 1.48–1.51 (m, 1H), 1.64–1.65 (m, 2H), 1.76–1.82 (m, 2H), 3.77 (s, 3H), 3.81 (s, 3H), 4.69–4.73 (m, 1H), 6.24 (s, 1H), 6.81 (d, J = 8.8 Hz, 2H), 6.90 (d, J = 8.7 Hz, 2H), 7.15 (d, J = 8.7 Hz, 2H), 7.23 (d, J = 8.8 Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3): δ 23.5, 25.1, 31.3, 54.9, 55.0, 71.9, 112.9, 113.4, 115.2, 129.6, 130.6, 131.1, 133.5, 155.5, 159.3, 160.4, 165.6. HRMS calcd for $\text{C}_{23}\text{H}_{26}\text{O}_4$, 336.1831; found, 336.1830.



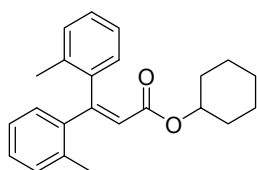
Cyclohexyl 3,3-di-*p*-tolylacrylate (4e), Table 2, Entry 5). Pale yellow solid (m.p. = 94–96 °C). ^1H NMR (300 MHz, CDCl_3): δ 1.29–1.35 (m, 5H), 1.53–1.55 (m, 1H), 1.69 (m, 2H), 1.83 (m, 2H), 2.39 (s, 3H), 2.44 (s, 3H), 4.78–4.79 (m, 1H), 6.37–6.39 (m, 1H), 7.14–7.18 (m, 4H), 7.22–7.27 (m, 4H); ^{13}C NMR (75 MHz, CDCl_3): δ 21.0, 21.2, 23.6, 25.2, 31.4, 72.0, 116.6, 128.1, 128.3, 128.8, 129.0, 136.1, 137.5, 138.1, 139.2, 156.1, 165.5. HRMS calcd for $\text{C}_{23}\text{H}_{26}\text{O}_2$, 334.1933; found, 334.1929.



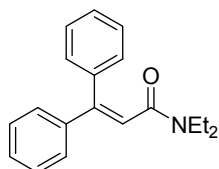
Cyclohexyl 3,3-di-*m*-tolylacrylate (4f), Table 2, Entry 6). Pale yellow oil. ^1H NMR (300 MHz, CDCl_3): δ 1.18–1.31 (m, 5H), 1.45–1.49 (m, 1H), 1.59–1.63 (m, 2H), 1.70–1.77 (m, 2H), 2.32 (s, 3H), 2.35 (s, 3H), 4.66–4.72 (m, 1H), 6.32 (s, 1H), 7.00–7.29 (m, 8H); ^{13}C NMR (75 MHz, CDCl_3): δ 21.4, 21.4, 23.6, 25.3, 31.4, 72.3, 117.9, 125.5, 126.2, 127.7, 128.1, 128.65, 128.70, 129.6, 130.0, 137.3, 137.9, 139.1, 140.9, 156.1, 165.8. HRMS calcd for $\text{C}_{23}\text{H}_{26}\text{O}_2$, 334.1933; found, 334.1937.



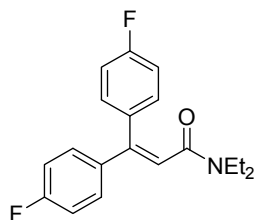
Cyclohexyl 3,3-bis(3,5-dimethylphenyl)acrylate (4g, Table 2, Entry 7). Pale yellow solid (m.p. = 79–81 °C). ^1H NMR (300 MHz, CDCl_3): δ 1.25–1.29 (m, 5H), 1.48 (m, 1H), 1.58 (m, 2H), 1.71–1.73 (m, 2H), 2.27 (s, 6H), 2.30 (s, 6H), 4.67–4.70 (m, 1H), 6.26 (s, 1H), 6.80 (s, 2H), 6.91 (s, 2H), 6.97 (s, 2H); ^{13}C NMR (75 MHz, CDCl_3): δ 21.2, 21.3, 23.6, 25.4, 31.4, 72.2, 117.8, 126.1, 126.7, 129.5, 130.9, 137.0, 137.7, 139.1, 141.1, 156.4, 165.9. HRMS calcd for $\text{C}_{25}\text{H}_{30}\text{O}_2$, 362.2246; found, 362.2254.



Cyclohexyl 3,3-di-*o*-tolylacrylate (4h, Table 2, Entry 8). Pale yellow oil. ^1H NMR (300 MHz, CDCl_3): δ 1.16–1.26 (m, 5H), 1.45 (m, 1H), 1.53–1.60 (m, 2H), 1.67–1.70 (m, 2H), 2.14 (s, 3H), 2.32 (s, 3H), 4.62–4.68 (m, 1H), 6.09 (s, 1H), 7.05–7.22 (m, 8H); ^{13}C NMR (75 MHz, CDCl_3): δ 19.8, 20.8, 23.6, 25.3, 31.3, 72.3, 122.8, 125.2, 125.6, 127.7, 128.1, 129.0, 129.7, 129.9, 131.1, 135.4, 135.6, 139.7, 140.6, 155.3, 165.5. HRMS calcd for $\text{C}_{23}\text{H}_{26}\text{O}_2$, 334.1933; found, 334.1938.

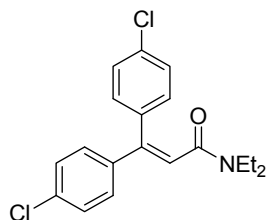


N,N-diethyl-3,3-diphenylacrylamide (5a [3], Table 3, Entry 1). Pale yellow oil. ^1H NMR (300 MHz, CDCl_3): δ 0.95 (t, J = 7.2 Hz, 3H), 0.97 (t, J = 7.2 Hz, 3H), 3.23 (q, J = 7.2 Hz, 2H), 3.31 (q, J = 7.2 Hz, 2H), 6.35 (s, 1H), 7.24–7.31 (m, 10H); ^{13}C NMR (75 MHz, CDCl_3): δ 12.1, 13.9, 38.6, 42.3, 121.6, 127.9, 128.0, 128.1, 128.15, 128.21, 129.3, 138.7, 141.2, 146.7, 167.5.

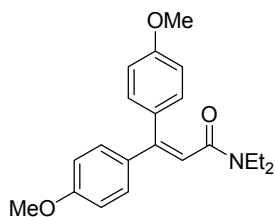


N,N-diethyl-3,3-bis(4-fluorophenyl)acrylamide (5b, Table 3, Entry 4). Yellow solid (m.p. = 86–88 °C). ^1H NMR (300 MHz, CDCl_3): δ 0.97 (t, J = 7.0 Hz, 3H), 0.99 (t, J = 7.0 Hz, 3H), 3.24 (q, J = 7.0 Hz, 2H),

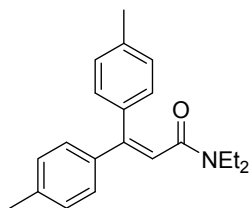
3.32 (q, $J = 7.0$ Hz, 2H), 6.29 (s, 1H), 6.96–7.01 (m, 4H), 7.19–7.24 (m, 4H); ^{13}C NMR (75 MHz, CDCl_3): δ 12.3, 14.0, 38.9, 42.4, 115.1 ($J_{\text{C-F}} = 21.4$ Hz), 115.3 ($J_{\text{C-F}} = 21.4$ Hz), 121.6, 129.7 ($J_{\text{C-F}} = 8.1$ Hz), 131.1 ($J_{\text{C-F}} = 8.1$ Hz), 134.6 ($J_{\text{C-F}} = 3.3$ Hz), 137.2 ($J_{\text{C-F}} = 3.2$ Hz), 145.0, 162.6 ($J_{\text{C-F}} = 246.5$ Hz), 162.8 ($J_{\text{C-F}} = 246.9$ Hz), 167.1. HRMS calcd for $\text{C}_{19}\text{H}_{19}\text{F}_2\text{NO}$, 315.1435; found, 315.1436.



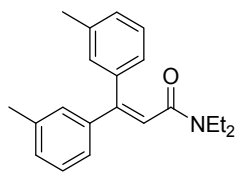
N,N-diethyl-3,3-bis(4-chlorophenyl)acrylamide (5c [4], Table 3, Entry 5). Brown solid. (m.p. = 82–84 °C, lit. 84–85 °C) ^1H NMR (300 MHz, CDCl_3): δ 0.98 (t, $J = 6.9$ Hz, 3H), 1.00 (t, $J = 6.9$ Hz, 3H), 3.24 (q, $J = 6.9$ Hz, 2H), 3.31 (q, $J = 6.9$ Hz, 2H), 6.34 (s, 1H), 7.14–7.17 (m, 4H), 7.25–7.28 (m, 4H); ^{13}C NMR (75 MHz, CDCl_3): δ 12.3, 14.1, 38.9, 42.4, 122.4, 128.4, 128.6, 129.3, 130.6, 134.4, 134.5, 136.8, 139.3, 144.9, 166.8.



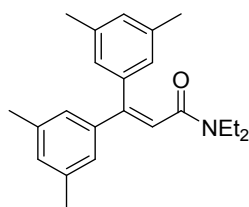
N,N-diethyl-3,3-bis(4-methoxyphenyl)acrylamide (5d, Table 3, Entry 6). Pale yellow oil. ^1H NMR (300 MHz, CDCl_3): δ 0.93 (t, $J = 7.0$ Hz, 3H), 0.95 (t, $J = 7.0$ Hz, 3H), 3.20 (q, $J = 7.0$ Hz, 2H), 3.28 (q, $J = 7.0$ Hz, 2H), 3.72 (s, 6H), 6.15 (s, 1H), 6.76–6.79 (m, 4H), 7.12–7.18 (m, 4H); ^{13}C NMR (75 MHz, CDCl_3): δ 12.2, 13.8, 38.6, 42.1, 54.9, 55.0, 113.1, 113.3, 118.9, 129.2, 130.5, 131.1, 133.8, 146.1, 159.3, 159.5, 167.7. HRMS calcd for $\text{C}_{21}\text{H}_{25}\text{NO}_3$, 339.1834; found, 339.1834.



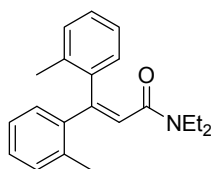
N,N-diethyl-3,3-di-*p*-tolylacrylamide (5e, Table 3, Entry 7). Pale yellow solid (m.p. = 83–85 °C). ^1H NMR (300 MHz, CDCl_3): δ 0.98 (t, $J = 7.1$ Hz, 6H), 2.33 (s, 3H), 2.34 (s, 3H), 3.25 (q, $J = 7.1$ Hz, 2H), 3.33 (q, $J = 7.1$ Hz, 2H), 6.28 (s, 1H), 7.07–7.18 (m, 8H); ^{13}C NMR (75 MHz, CDCl_3): δ 11.9, 13.5, 20.6, 20.8, 38.3, 41.9, 120.0, 127.6, 128.2, 128.4, 128.8, 135.6, 137.4, 137.6, 138.2, 146.4, 167.2. HRMS calcd for $\text{C}_{21}\text{H}_{25}\text{NO}$, 307.1936; found, 307.1939.



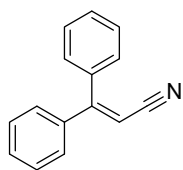
N,N-diethyl-3,3-di-*m*-tolylacrylamide (5f, Table 3, Entry 8). Pale yellow oil. ^1H NMR (300 MHz, CDCl_3): δ 0.94 (t, $J = 7.1$, 3H), 0.96 (t, $J = 7.1$, 3H), 2.27 (s, 3H), 2.28 (s, 3H), 3.22 (q, $J = 7.1$, 2H), 3.31 (q, $J = 7.1$, 2H), 6.30 (s, 1H), 7.00–7.19 (m, 8H); ^{13}C NMR (75 MHz, CDCl_3): δ 12.0, 13.7, 21.1, 38.4, 42.1, 121.2, 125.0, 126.2, 127.6, 127.8, 128.4, 128.6, 128.8, 129.7, 137.2, 137.5, 138.6, 141.1, 146.7, 167.4. HRMS calcd for $\text{C}_{21}\text{H}_{25}\text{NO}$, 307.1936; found, 307.1933.



3,3-Bis(3,5-dimethylphenyl)-N,N-diethylacrylamide (5g, Table 3, Entry 9). Pale yellow oil. ^1H NMR (300 MHz, CDCl_3): δ 0.97 (t, $J = 7.0$ Hz, 6H), 2.24 (s, 6H), 2.27 (s, 6H), 3.24 (q, $J = 7.0$, 2H), 3.32 (q, $J = 7.0$, 2H), 6.23 (s, 1H), 6.83 (s, 2H), 6.88 (s, 2H), 6.92 (s, 1H), 6.95 (s, 1H); ^{13}C NMR (75 MHz, CDCl_3): δ 12.0, 13.7, 21.0, 38.4, 42.2, 121.0, 125.7, 126.8, 129.5, 129.6, 137.0, 137.3, 138.7, 141.3, 147.0, 167.5. HRMS calcd for $\text{C}_{23}\text{H}_{29}\text{NO}$, 335.2249; found, 335.2243.

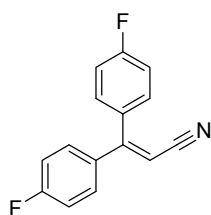


N,N-diethyl-3,3-di-*o*-tolylacrylamide (5h, Table 3, Entry 10). Pale yellow oil. ^1H NMR (300 MHz, CDCl_3): δ 0.94 (t, $J = 7.1$ Hz, 3H), 1.01 (t, $J = 7.1$ Hz, 3H), 2.00 (s, 3H), 2.22 (s, 3H), 3.30 (q, $J = 7.1$ Hz, 4H), 6.23 (s, 1H), 7.07–7.23 (m, 8H); ^{13}C NMR (75 MHz, CDCl_3): δ 12.4, 14.1, 20.2, 20.9, 38.8, 42.2, 125.4, 125.7, 125.8, 127.7, 127.9, 129.6, 129.9, 130.3, 130.9, 135.6, 135.8, 139.1, 141.0, 148.0, 166.9. HRMS calcd for $\text{C}_{21}\text{H}_{25}\text{NO}$, 307.1936; found, 307.1937.

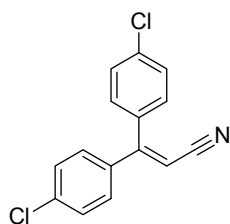


3,3-Diphenylacrylonitrile (6a [5], Table 4, Entry 1). Yellow oil. ^1H NMR (300 MHz, CDCl_3): δ 5.73 (s,

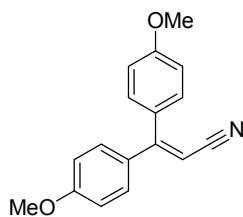
1H), 7.27–7.33 (m, 2H), 7.34–7.46 (m, 8H); ^{13}C NMR (75 MHz, CDCl_3): δ 94.6, 117.7, 128.25, 128.32, 128.4, 129.3, 129.8, 130.2, 136.8, 138.6, 162.8.



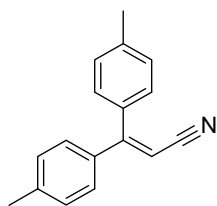
3,3-Bis(4-fluorophenyl)acrylonitrile (6b), Table 4, Entry 2). Yellow solid (m.p. = 77–79 °C). ^1H NMR (300 MHz, CDCl_3): δ 5.66 (s, 1H), 7.03–7.15 (m, 4H), 7.26–7.29 (m, 2H), 7.38–7.43 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3): δ 94.8, 115.77 (d, J = 21.7 Hz), 115.83 (d, J = 21.7 Hz), 117.6, 130.4 (d, J = 8.5 Hz), 131.6 (d, J = 8.5 Hz), 132.8 (d, J = 3.3 Hz), 134.8 (d, J = 3.2 Hz), 160.8, 163.6 (d, J = 249.6 Hz), 164.0 (d, J = 250.6 Hz). HRMS calcd for $\text{C}_{15}\text{H}_9\text{F}_2\text{N}$, 241.0703; found, 241.0702.



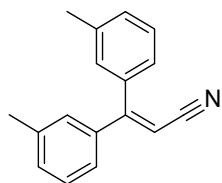
3,3-Bis(4-chlorophenyl)acrylonitrile (6c), Table 4, Entry 3). Yellow solid (m.p. = 92–94 °C). ^1H NMR (300 MHz, CDCl_3): δ 5.71 (s, 1H), 7.19–7.22 (m, 2H), 7.32–7.37 (m, 4H), 7.40–7.44 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3): δ 95.6, 117.4, 129.00, 129.04, 129.6, 130.8, 134.9, 136.4, 136.8, 136.9, 160.6. HRMS calcd for $\text{C}_{15}\text{H}_9\text{Cl}_2\text{N}$, 273.0112; found, 273.0107.



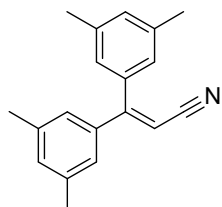
3,3-Bis(4-methoxyphenyl)acrylonitrile (6d) [6], Table 4, Entry 4). Yellow oil. ^1H NMR (300 MHz, CDCl_3): δ 3.81 (s, 3H), 3.83 (s, 3H), 5.52 (s, 1H), 6.85–6.88 (m, 2H), 6.92–6.95 (m, 2H), 7.21–7.24 (m, 2H), 7.36–7.39 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3): δ 55.2, 55.3, 91.3, 113.7, 113.8, 118.7, 129.4, 130.1, 131.2, 131.5, 160.8, 161.3, 162.2.



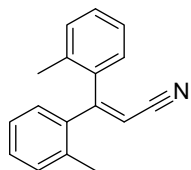
3,3-Di-*p*-tolylacrylonitrile (6e, Table 4, Entry 5). Yellow solid (m.p. = 94–96 °C). ^1H NMR (300 MHz, CDCl_3): δ 2.39 (s, 3H), 2.42 (s, 3H), 5.66 (s, 1H), 7.16–7.20 (m, 4H), 7.22–7.27 (m, 2H), 7.34–7.36 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3): δ 21.2, 21.3, 93.1, 118.2, 128.3, 129.0, 129.2, 129.4, 134.1, 136.1, 140.0, 140.6, 162.9. HRMS calcd for $\text{C}_{17}\text{H}_{15}\text{N}$, 223.1204; found, 233.1205.



3,3-Di-*m*-tolylacrylonitrile (6f, Table 4, Entry 6). Yellow oil. ^1H NMR (300 MHz, CDCl_3): δ 2.34 (s, 3H), 2.38 (s, 3H), 5.69 (s, 1H), 7.06–7.11 (m, 2H), 7.22–7.36 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3): δ 21.21, 21.24, 94.4, 117.9, 125.6, 126.5, 128.2, 128.4, 128.8, 129.9, 130.6, 131.0, 137.0, 138.0, 138.2, 138.9, 163.3. HRMS calcd for $\text{C}_{17}\text{H}_{15}\text{N}$, 223.1204; found, 233.1205.

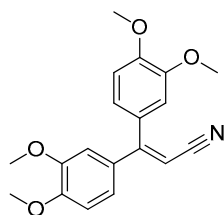


3,3-Bis(3,5-dimethylphenyl)acrylonitrile (6g, Table 4, Entry 7). Yellow solid (m.p. = 148–150 °C). ^1H NMR (300 MHz, CDCl_3): δ 2.299 (s, 3H), 2.300 (s, 3H), 2.341 (s, 3H), 2.342 (s, 3H), 5.65 (s, 1H), 6.895 (s, 1H), 6.897 (s, 1H), 7.014 (s, 1H), 7.015 (s, 1H), 7.07 (s, 1H), 7.09 (s, 1H); ^{13}C NMR (75 MHz, CDCl_3): δ 21.17, 21.21, 94.1, 118.1, 126.2, 127.1, 131.5, 132.0, 137.2, 137.9, 138.1, 139.1, 163.9. HRMS calcd for $\text{C}_{19}\text{H}_{19}\text{N}$, 261.1517; found, 261.1520.

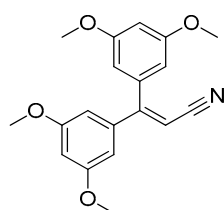


3,3-Di-*o*-tolylacrylonitrile (6h, Table 4, Entry 8). Yellow solid (m.p. = 73–75 °C). ^1H NMR (300 MHz, CDCl_3): δ 2.16 (s, 3H), 2.18 (s, 3H), 5.65 (s, 1H), 7.12–7.35 (m, 8H); ^{13}C NMR (75 MHz, CDCl_3): δ 20.0, 20.7, 100.1, 117.2, 125.8, 126.0, 129.3, 129.5, 129.6, 130.8, 131.2, 135.7, 135.8, 137.4, 138.7, 164.0. HRMS

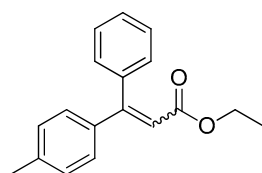
calcd for $C_{17}H_{15}N$, 223.1204; found, 233.1201.



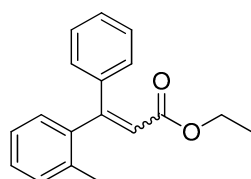
3,3-Bis(3,4-dimethoxyphenyl)acrylonitrile (6i) [7], Table 4, Entry 9). Light brown solid (m.p. = 117–119 °C). 1H NMR (300 MHz, $CDCl_3$): δ 3.78 (s, 3H), 3.82 (s, 3H), 3.87 (s, 3H), 3.89 (s, 3H), 5.52 (s, 1H), 6.77–6.89 (m, 4H), 6.94–6.95 (m, 1H), 6.99–7.03 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 55.80, 55.84, 55.9, 91.8, 110.5, 110.6, 111.2, 112.6, 118.6, 122.1, 123.1, 129.4, 131.7, 148.4, 148.6, 150.4, 150.9, 162.4.



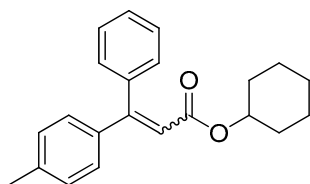
3,3-Bis(3,5-dimethoxyphenyl)acrylonitrile (6j) [8], Table 4, Entry 10). Light brown solid (m.p. = 153–155 °C, lit. 149–151 °C). 1H NMR (300 MHz, $CDCl_3$): δ 3.75 (s, 6H), 3.77 (s, 6H), 5.69 (s, 1H), 6.41 (s, 1H), 6.42 (s, 1H), 6.50 (t, J = 2.3 Hz, 1H), 6.52–6.53 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 55.5, 95.3, 102.0, 102.2, 106.7, 107.6, 117.6, 138.5, 140.6, 160.6, 160.7, 162.9.



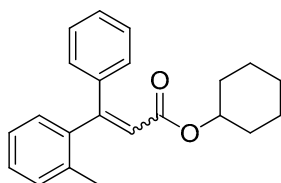
Ethyl 3-phenyl-3-(p-tolyl)acrylate (8ae) [9], Table 5, Entry 1). Yellow oil. 2.3:1 (*E/Z*) mixture. 1H NMR (300 MHz, $CDCl_3$): δ 1.10 (t, J = 7.1 Hz, maj.; 3H), 1.15 (t, J = 7.1 Hz, min.), 2.35 (s, maj.; 3H), 2.39 (s, min.), 4.03 (q, J = 7.1 Hz, maj.; 2H), 4.08 (q, J = 7.1 Hz, min.), 6.32 (s, min.), 6.35 (s, maj.; 1H), 7.09–7.13 (m, 2H), 7.18–7.20 (m, 3H), 7.31–7.38 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 13.95, 14.02, 21.2, 21.3, 59.9, 116.5, 117.1, 127.8, 128.0, 128.2, 128.26, 128.34, 128.5, 129.05, 129.14, 129.3, 135.9, 137.9, 138.0, 139.1, 139.6, 141.1, 156.5, 156.8, 166.2.



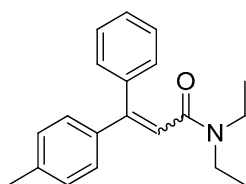
Ethyl 3-phenyl-3-(*o*-tolyl)acrylate (8ah [9], Table 5, Entry 2). Yellow oil. 1.0:1 (*E/Z*) mixture. ^1H NMR (300 MHz, CDCl_3): δ 1.07 (t, $J = 7.1$ Hz, 3H; *Z*), 1.17 (t, $J = 7.2$ Hz, 3H; *E*), 2.07 (s, 3H; *E*), 2.08 (s, 3H; *Z*), 4.01 (q, $J = 7.1$ Hz, 2H; *Z*), 4.12 (q, $J = 7.2$ Hz, 2H; *E*), 6.00 (s, 1H; *E*), 6.52 (s, 1H; *Z*), 7.19–7.26 (m, 9H), 7.28–7.32 (m, 9H); ^{13}C NMR (75 MHz, CDCl_3): δ 13.9, 14.0, 19.5, 20.3, 59.9, 60.1, 117.6, 120.1, 125.4, 125.6, 127.3, 127.6, 127.7, 128.4, 128.5, 129.1, 129.4, 129.5, 129.8, 130.6, 135.3, 135.9, 138.6, 138.7, 139.2, 141.9, 155.8, 156.7, 165.8, 166.3.



Cyclohexyl 3-phenyl-3-(*p*-tolyl)acrylate (8be, Table 5, Entry 3). Light brown oil. 2.4:1 (*E/Z*) mixture. ^1H NMR (300 MHz, CDCl_3): δ 1.16–1.31 (m, 5H), 1.47–1.49 (m, 1H), 1.59–1.61 (m, 2H), 1.71–1.74 (m, 2H), 2.34 (s, maj.; 3H), 2.38 (s, min.), 4.67–4.69 (m, 1H), 6.32 (s, min.), 6.34 (s, maj.; 1H), 7.09–7.12 (m, 2H), 7.17–7.21 (m, 4H), 7.29–7.30 (m, 1H), 7.35–7.37 (m, 2H). ^{13}C NMR (75 MHz, CDCl_3): δ 20.9, 21.2, 21.3, 23.5, 23.7, 25.3, 31.2, 31.4, 46.0, 72.3, 73.3, 76.4, 117.1, 117.8, 127.8, 127.9, 128.1, 128.2, 128.3, 128.5, 129.02, 129.04, 129.1, 155.8, 156.2, 165.70, 165.74, 172.3. HRMS calcd for $\text{C}_{22}\text{H}_{24}\text{O}_2$, 320.1776; found, 320.1780.

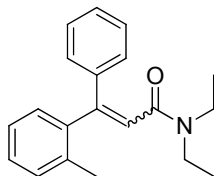


Cyclohexyl 3-phenyl-3-(*o*-tolyl)acrylate (8bh, Table 5, Entry 4). Colorless oil. 1.0:1 (*E/Z*) mixture. ^1H NMR (300 MHz, CDCl_3): δ 1.21–1.33 (m, 10H), 1.48–1.80 (m, 10H), 2.05 (s, 3H; *E*), 2.08 (s, 3H; *Z*), 4.65 (br, 1H), 4.75 (br, 1H), 6.00 (s, 1H; *Z*), 6.51 (s, 1H; *E*), 7.18–7.31 (m, 18H). ^{13}C NMR (75 MHz, CDCl_3): δ 19.6, 20.3, 23.6, 23.7, 25.27, 25.32, 31.35, 31.44, 72.3, 72.7, 118.4, 120.7, 125.4, 125.6, 127.3, 127.6, 128.2, 128.3, 128.4, 128.5, 129.1, 129.3, 129.5, 129.8, 130.6, 135.4, 136.0, 138.8, 141.9, 155.0, 156.1, 165.5, 165.8. HRMS calcd for $\text{C}_{22}\text{H}_{24}\text{O}_2$, 320.1776; found, 320.1778.

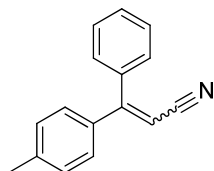


***N,N*-diethyl-3-phenyl-3-(*p*-tolyl)acrylamide (8ce, Table 5, Entry 5).** Light brown oil. 12.4:1 (*E/Z*) mixture. (*E*): ^1H NMR (300 MHz, CDCl_3): δ 0.93 (t, $J = 7.1$ Hz, 6H), 2.30 (s, 3H), 3.21 (q, $J = 7.1$ Hz, 2H),

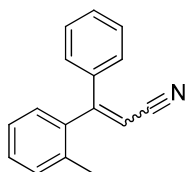
3.30 (q, $J = 7.1$ Hz, 2H), 6.32 (s, 1H), 7.06–7.17 (m, 4H), 7.22–7.28 (m, 5H). ^{13}C NMR (75 MHz, CDCl_3): δ 12.3, 14.0, 21.2, 38.7, 42.3, 120.9, 128.0, 129.0, 129.4, 138.3, 138.4, 139.0, 146.8, 167.7. HRMS calcd for $\text{C}_{20}\text{H}_{23}\text{NO}$, 293.1780; found, 293.1775.



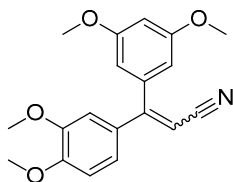
***N,N*-diethyl-3-phenyl-3-(*o*-tolyl)acrylamide (8ch**, Table 5, Entry 6). Light brown oil. 11.1:1 (*E/Z*) mixture. (*E*): ^1H NMR (300 MHz, CDCl_3): δ 0.93 (t, $J = 7.2$ Hz, 3H), 1.05 (t, $J = 7.2$ Hz, 3H), 2.02 (s, 3H), 3.24 (q, $J = 7.2$ Hz, 2H), 3.38 (q, $J = 7.2$ Hz, 2H), 6.05 (s, 1H), 7.12–7.27 (m, 9H). ^{13}C NMR (75 MHz, CDCl_3): δ 12.2, 13.9, 20.3, 38.9, 42.4, 123.7, 125.6, 127.9, 128.1, 128.4, 129.6, 130.4, 136.2, 138.8, 141.6, 146.1, 167.7. HRMS calcd for $\text{C}_{20}\text{H}_{23}\text{NO}$, 293.1780; found, 293.1782.



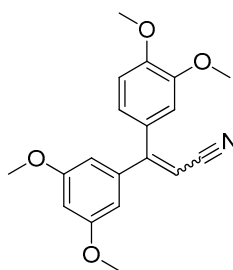
3-Phenyl-3-(*p*-tolyl)acrylonitrile (8de [10], Table 5, Entry 7). Yellow oil. 6.5:1 (*E/Z*) mixture. ^1H NMR (300 MHz, CDCl_3): δ 2.37 (s, maj.; 3H), 2.40 (s, min.), 5.66 (s, min.), 5.70 (s, maj.; 1H), 7.17–7.25 (m, 4H), 7.30–7.45 (m, 5H); ^{13}C NMR (75 MHz, CDCl_3): δ 21.3, 21.4, 93.9, 94.2, 118.0, 128.3, 128.4, 128.47, 128.52, 129.2, 129.3, 129.5, 129.9, 130.3, 134.1, 136.0, 137.1, 139.1, 140.3, 140.8, 163.0, 163.1.



3-Phenyl-3-(*o*-tolyl)acrylonitrile (8dh [11], Table 5, Entry 8). 4.7:1 (*E/Z*) mixture. (*E*): White solid (m.p. = 111–113 $^{\circ}\text{C}$, lit. 113–115 $^{\circ}\text{C}$). ^1H NMR (300 MHz, CDCl_3): δ 1.97 (s, 3H), 5.47 (s, 1H), 7.16–7.21 (m, 2H), 7.26–7.34 (m, 2H), 7.36–7.41 (m, 3H), 7.45–7.49 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3): δ 20.2, 96.7, 117.6, 125.9, 128.6, 129.3, 129.4, 130.2, 130.8, 136.1, 137.1, 139.6, 163.5.; (*Z*): Yellow oil. ^1H NMR (300 MHz, CDCl_3) δ 2.08 (s, 3H), 5.96 (s, 1H), 7.16–7.28 (m, 5H), 7.31–7.42 (m, 4H); ^{13}C NMR (75 MHz, CDCl_3) δ 19.6, 96.3, 117.3, 126.1, 127.1, 128.9, 129.2, 129.3, 130.5, 130.7, 135.8, 136.6, 137.4, 163.0.



3-(3,4-Dimethoxyphenyl)-3-(3,5-dimethoxyphenyl)acrylonitrile (8ei [12], Table 5, Entry 9). Light brown solid. 1:5.2 (*E/Z*) mixture. ^1H NMR (300 MHz, CDCl_3): δ 3.73 (s, min.), 3.76 (s, maj.; 6H), 3.81 (s, maj.; 3H), 3.84 (s, min.), 3.88 (s, maj.; 3H), 3.89 (s, min.), 5.58 (s, min.), 5.63 (s, maj.; 1H), 6.41 (d, J = 2.2 Hz, min), 6.52 (s, maj.; 3H), 6.79–6.92 (m, maj.; 3H), 6.99–7.09 (m, min.); ^{13}C NMR (75 MHz, CDCl_3): δ 55.4, 55.9, 93.1, 102.0, 106.9, 107.6, 110.7, 110.8, 118.0, 122.1, 131.0, 138.9, 148.8, 151.1, 160.5, 162.5.

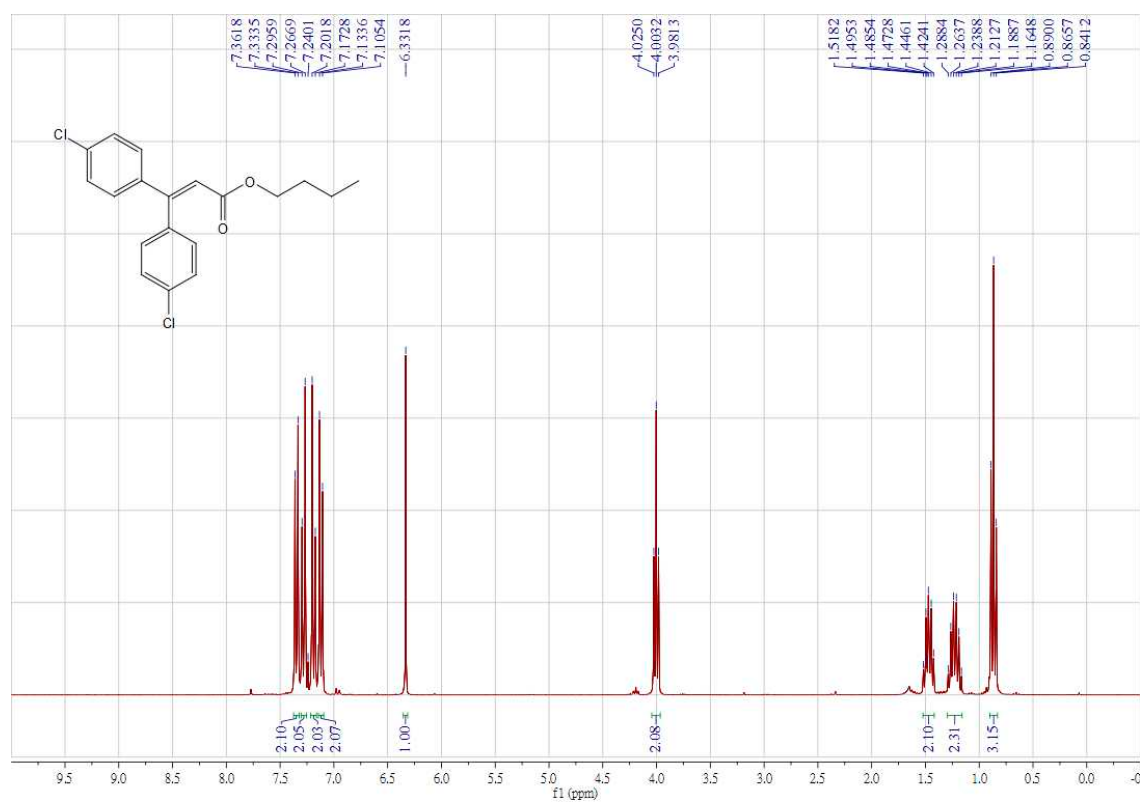
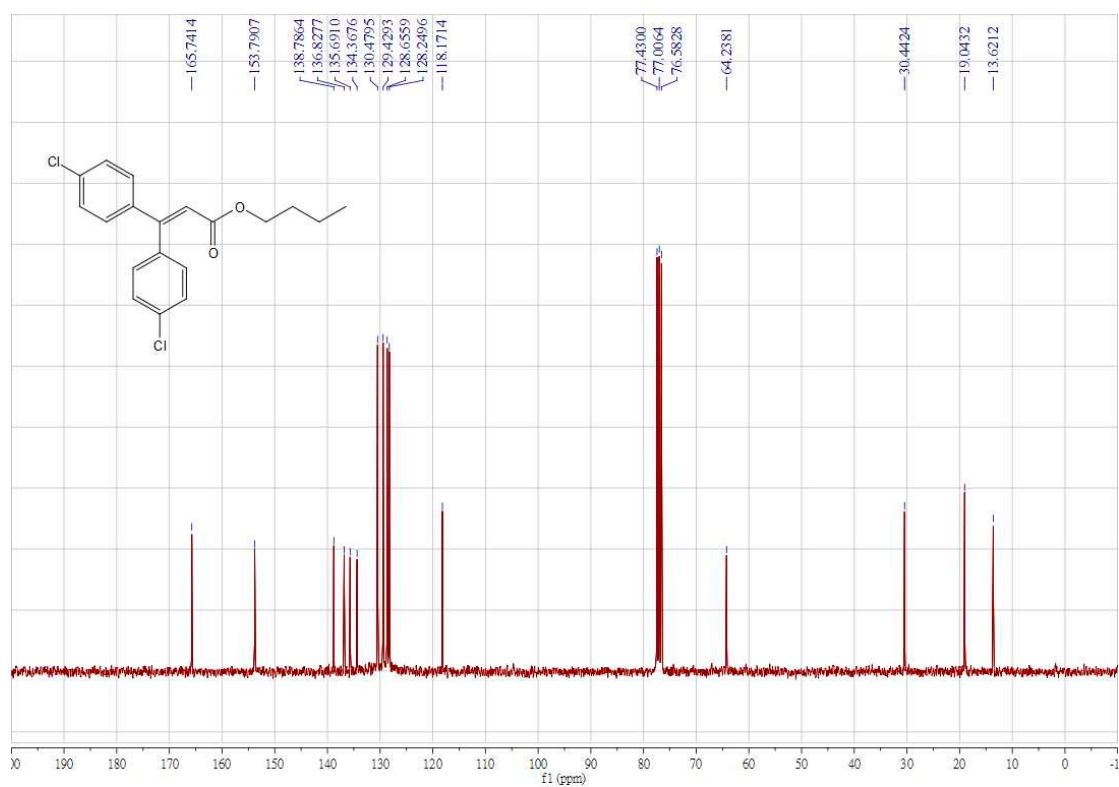


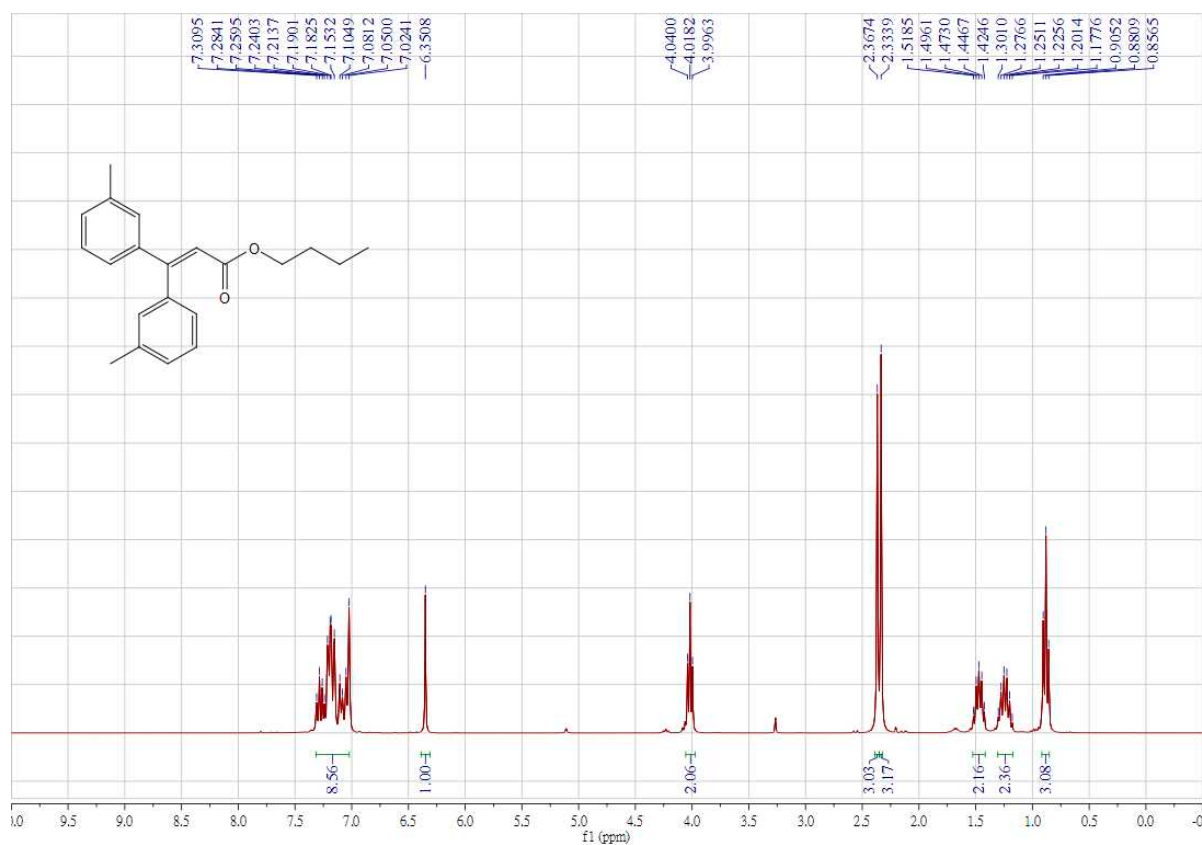
3-(3,4-Dimethoxyphenyl)-3-(3,5-dimethoxyphenyl)acrylonitrile (8ei [12], Table 5, Entry 10). Light brown solid. 4.3:1 (*E/Z*) mixture. ^1H NMR (300 MHz, CDCl_3): δ 3.74 (s, maj.; 6H), 3.77 (s, min.), 3.81 (s, min.), 3.84 (s, maj.; 3H), 3.88 (s, min.), 3.90 (s, maj.; 3H), 5.58 (s, maj.; 1H), 5.63 (s, min.), 6.41 (d, J = 2.3 Hz, maj.; 3H), 6.50–6.52 (m, min.), 6.79–7.03 (m, 3H); ^{13}C NMR (75 MHz, CDCl_3): δ 55.4, 55.85, 55.92, 56.0, 93.7, 102.0, 106.9, 107.6, 110.6, 112.5, 118.2, 123.2, 129.1, 141.3, 148.5, 150.6, 160.6, 162.7.

References

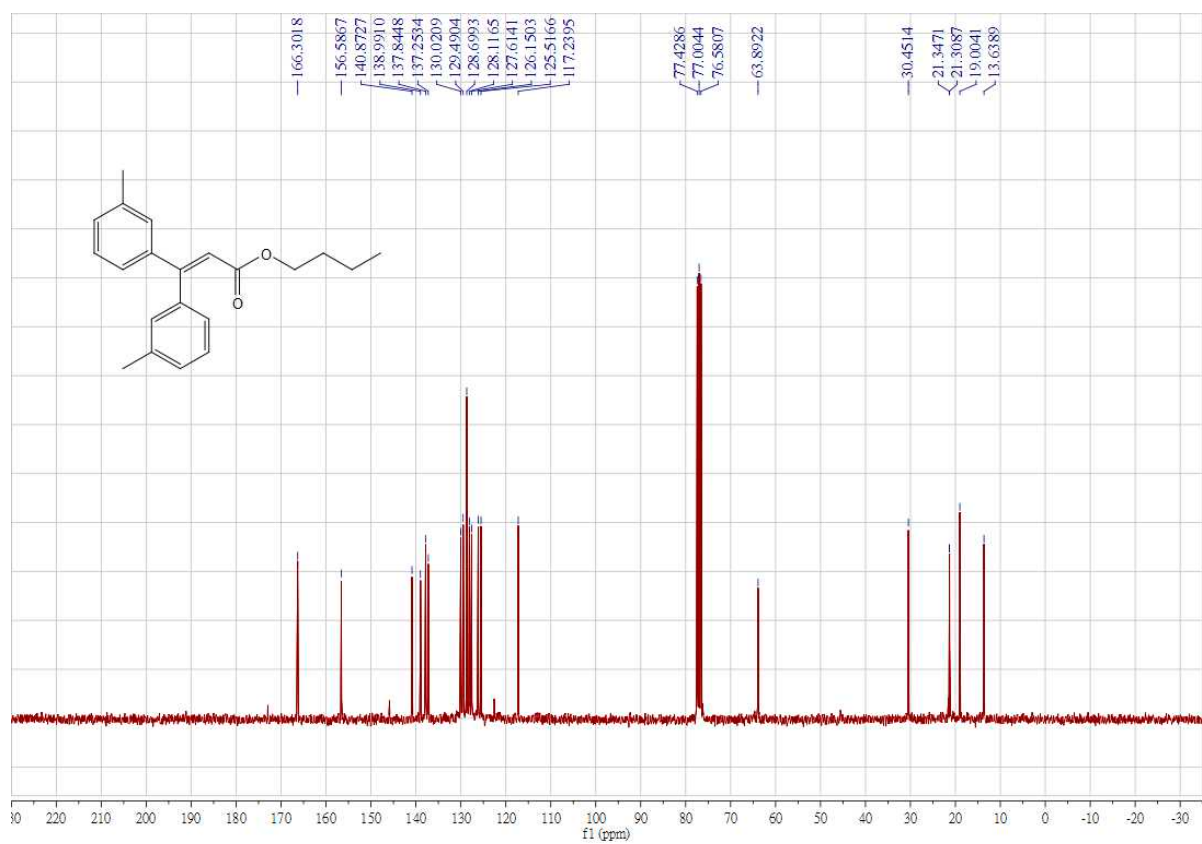
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Copies of ^1H and ^{13}C NMR spectra for unknown and unsymmetrical double Mizoroki-Heck products

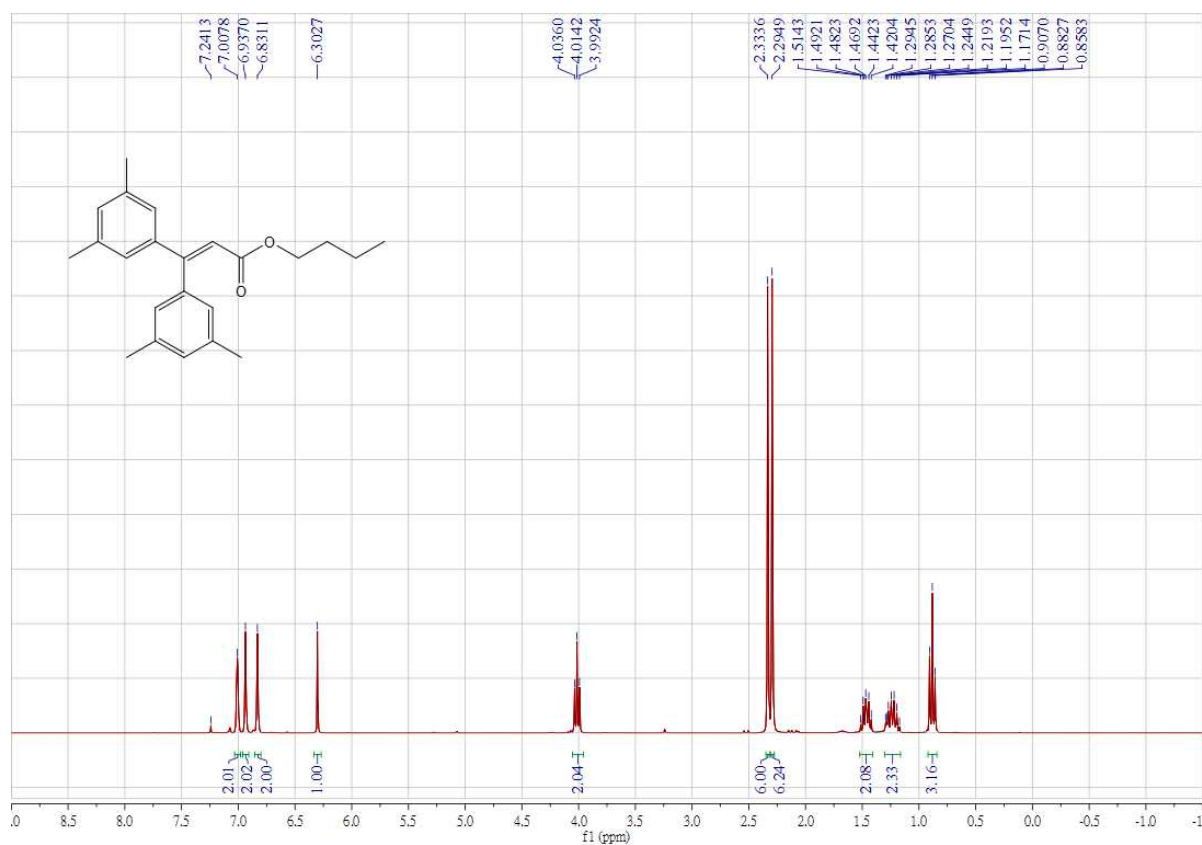
 ^1H NMR spectrum of compound 3c

 ^{13}C NMR spectrum of compound 3c

 ^1H NMR spectrum of compound 3f



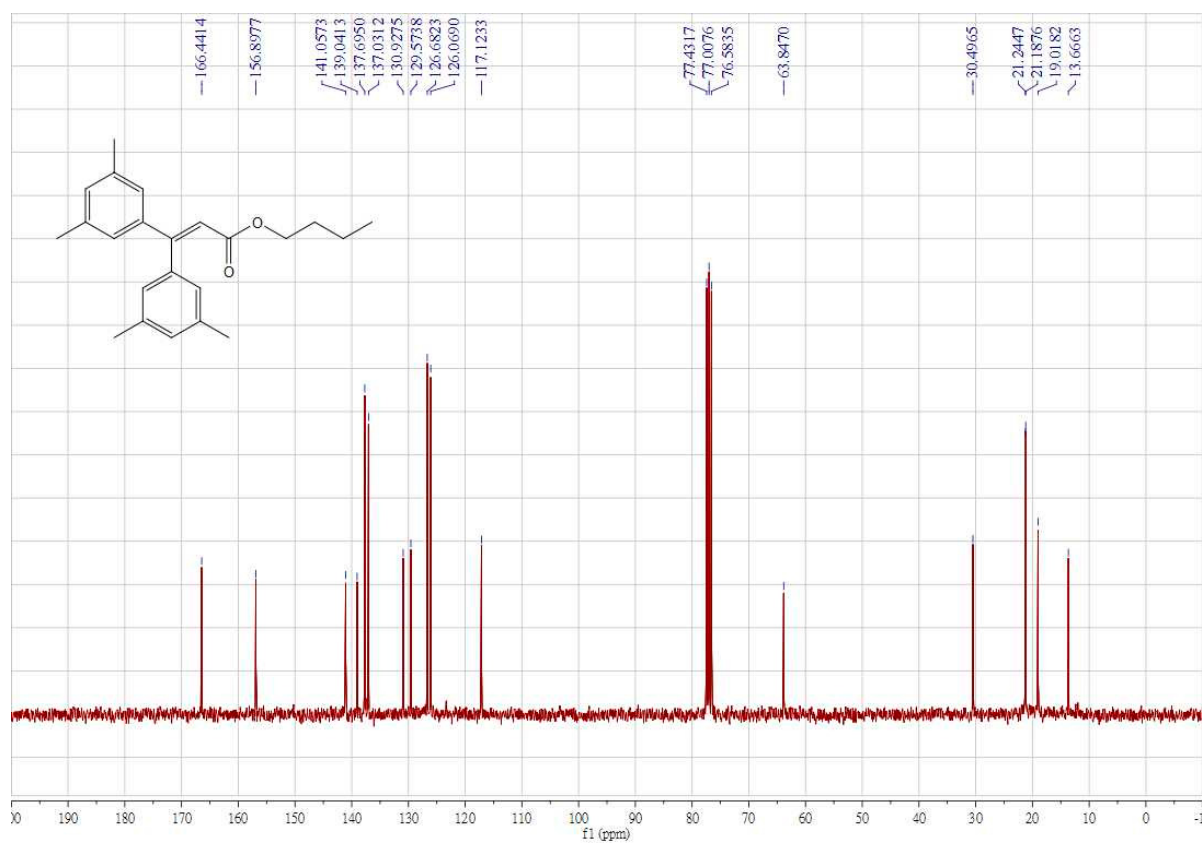
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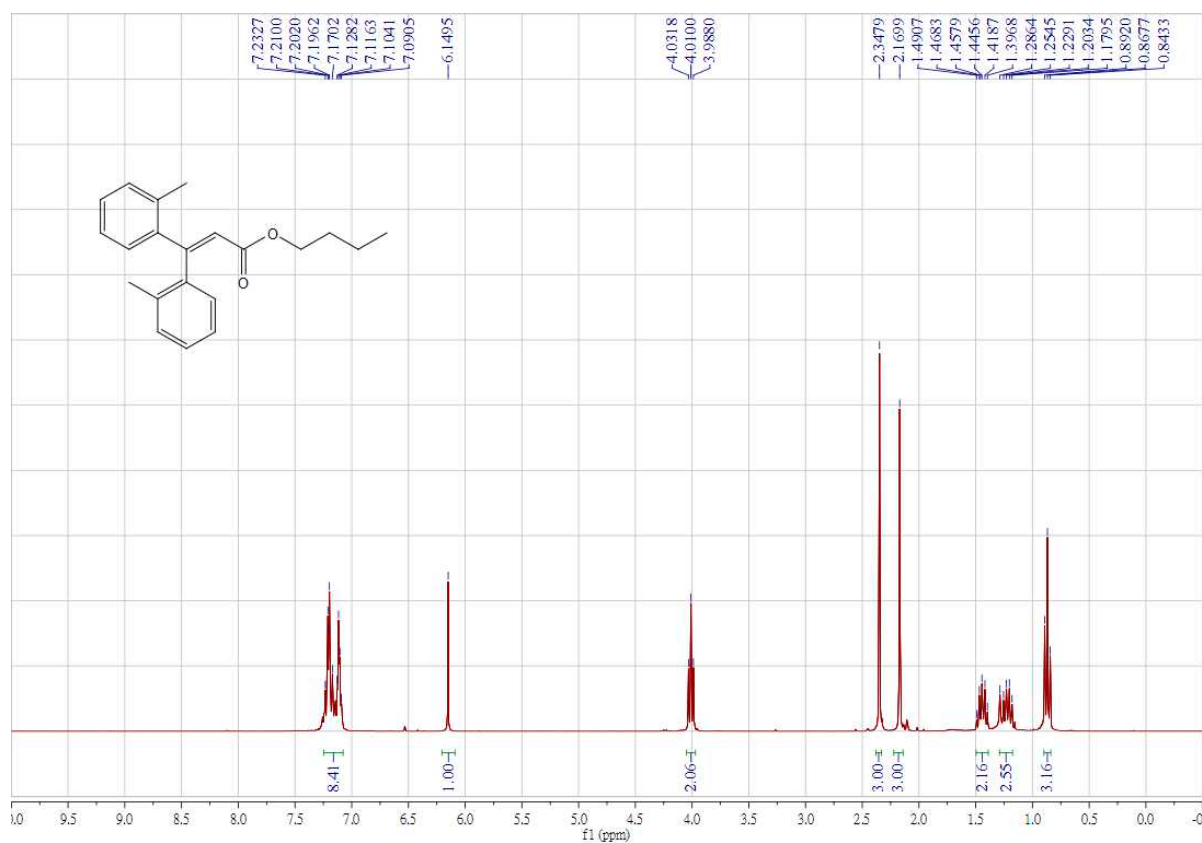
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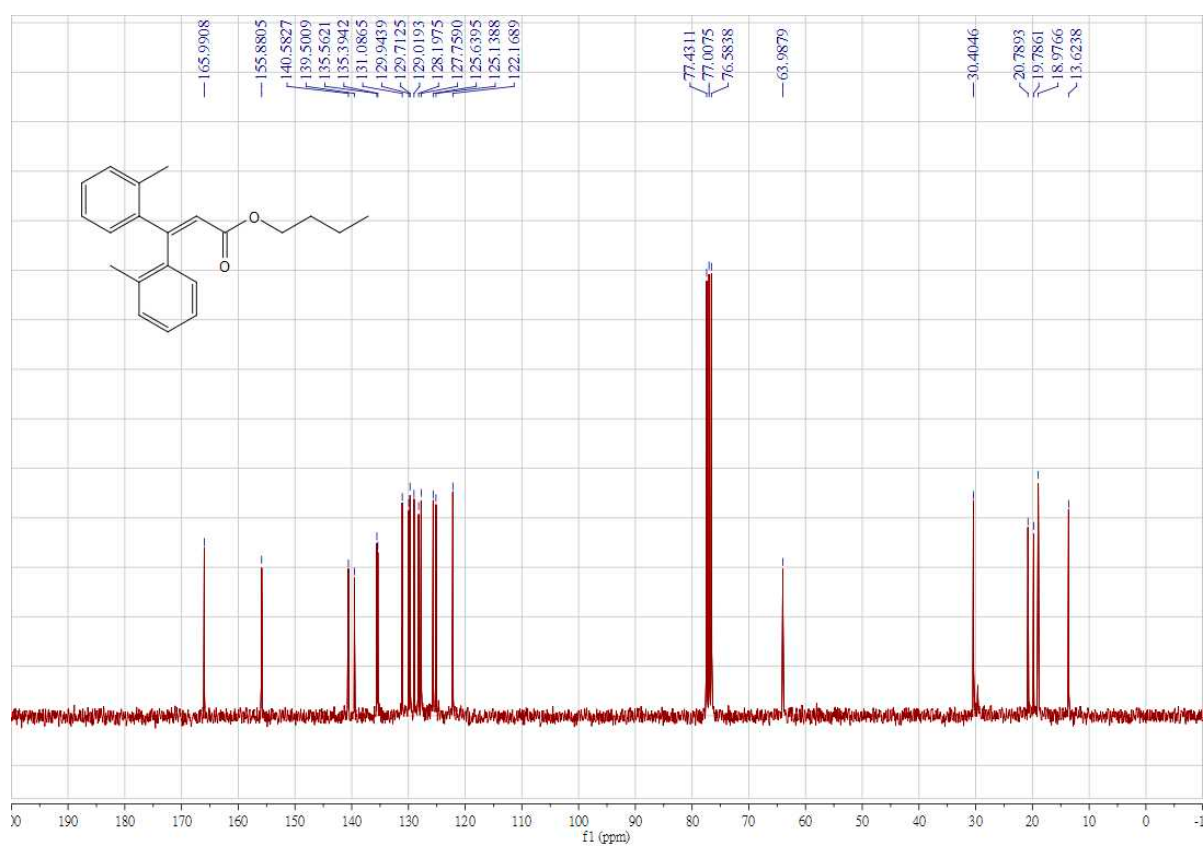
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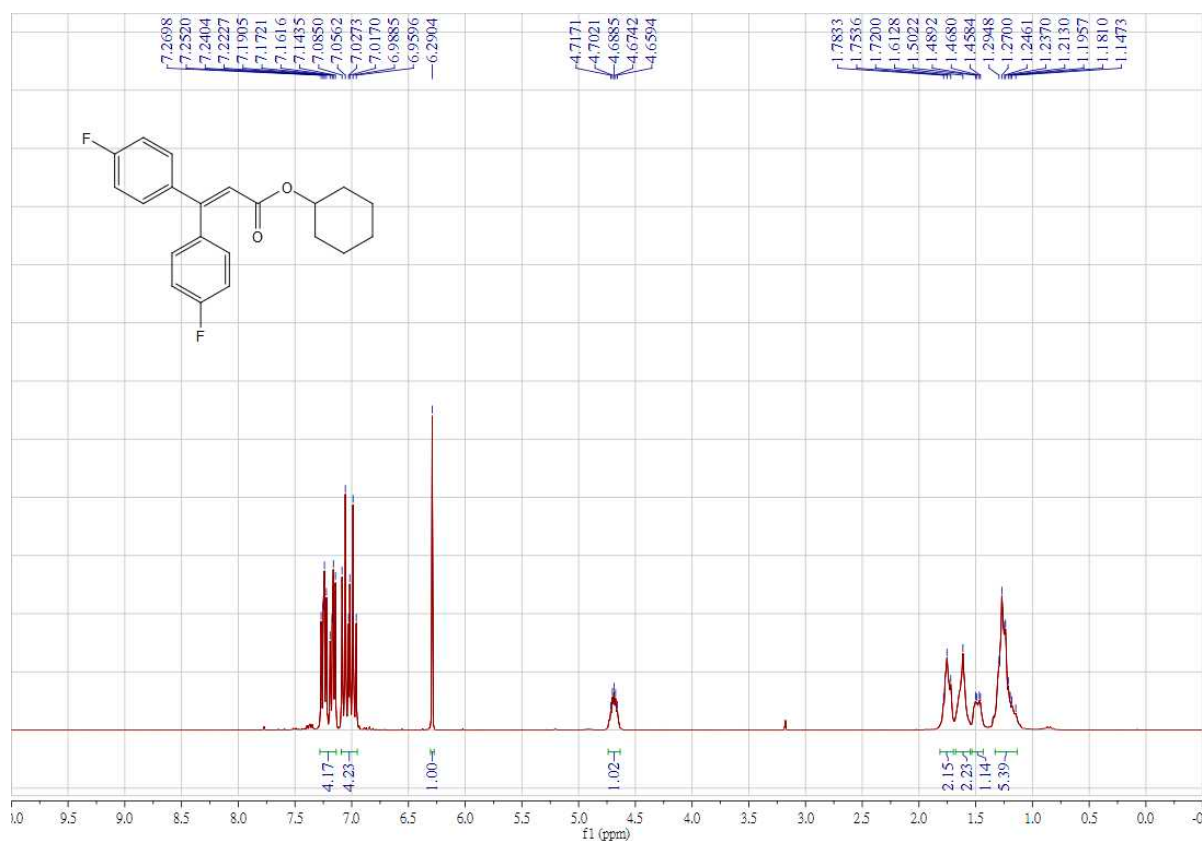
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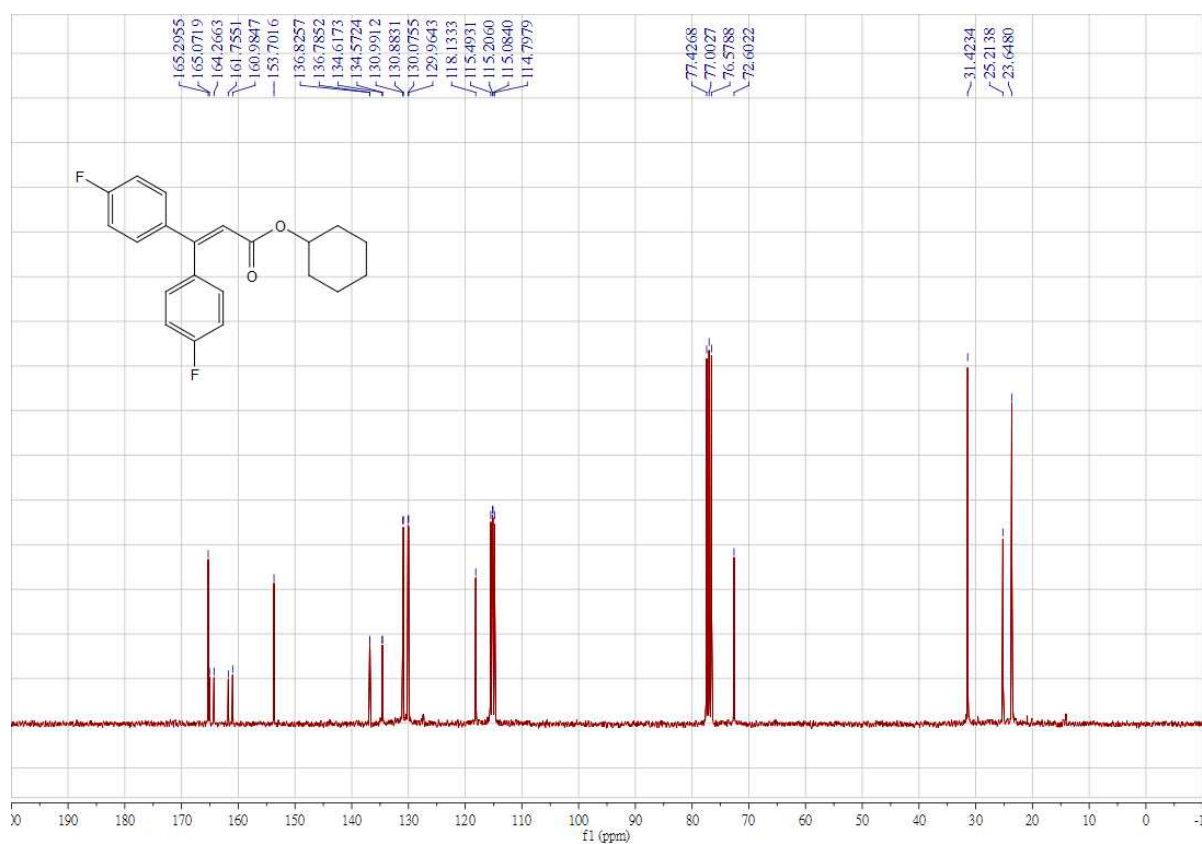
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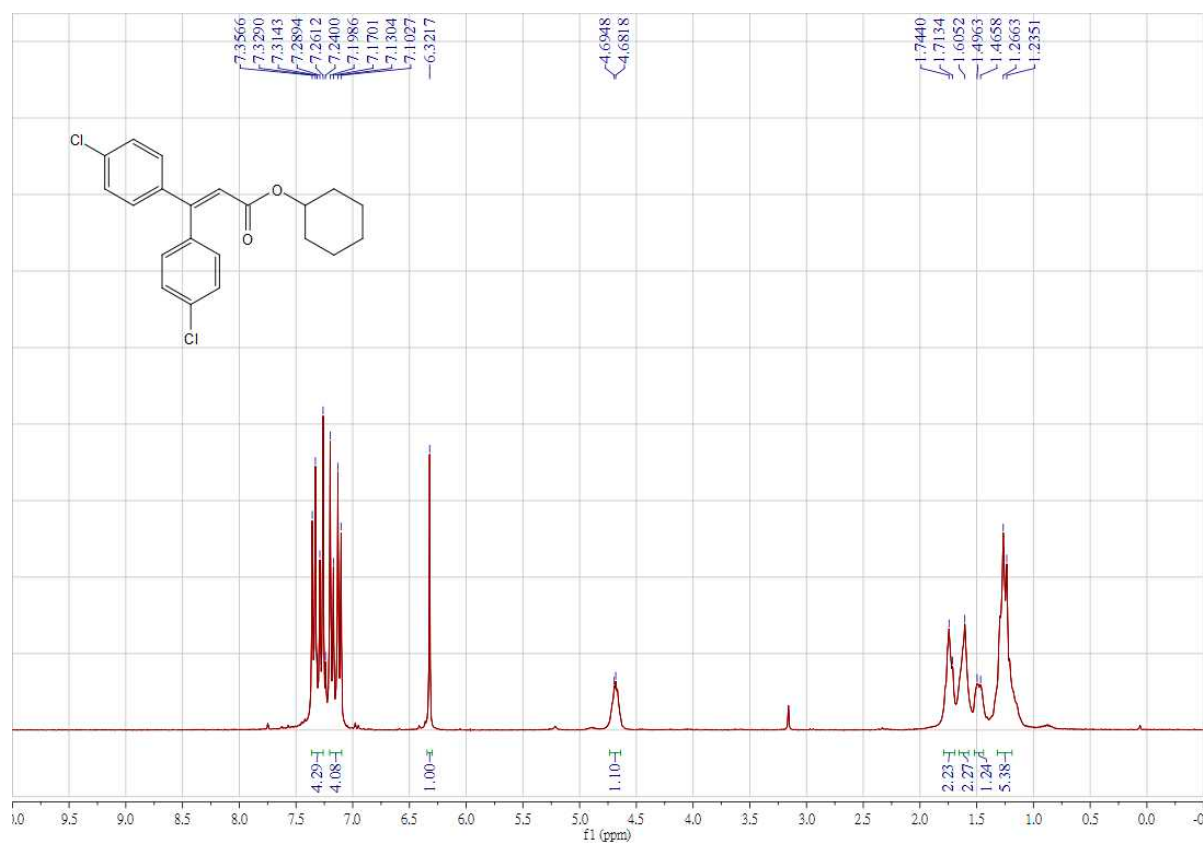
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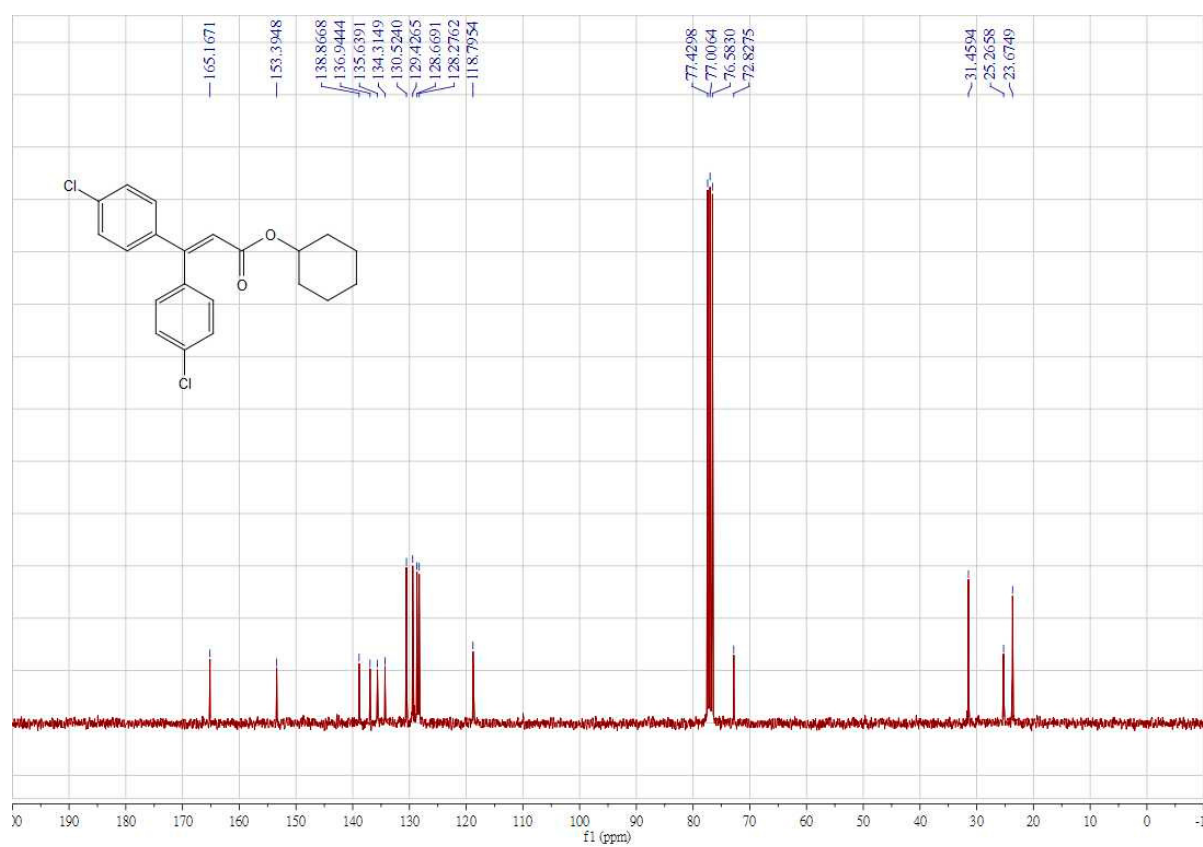
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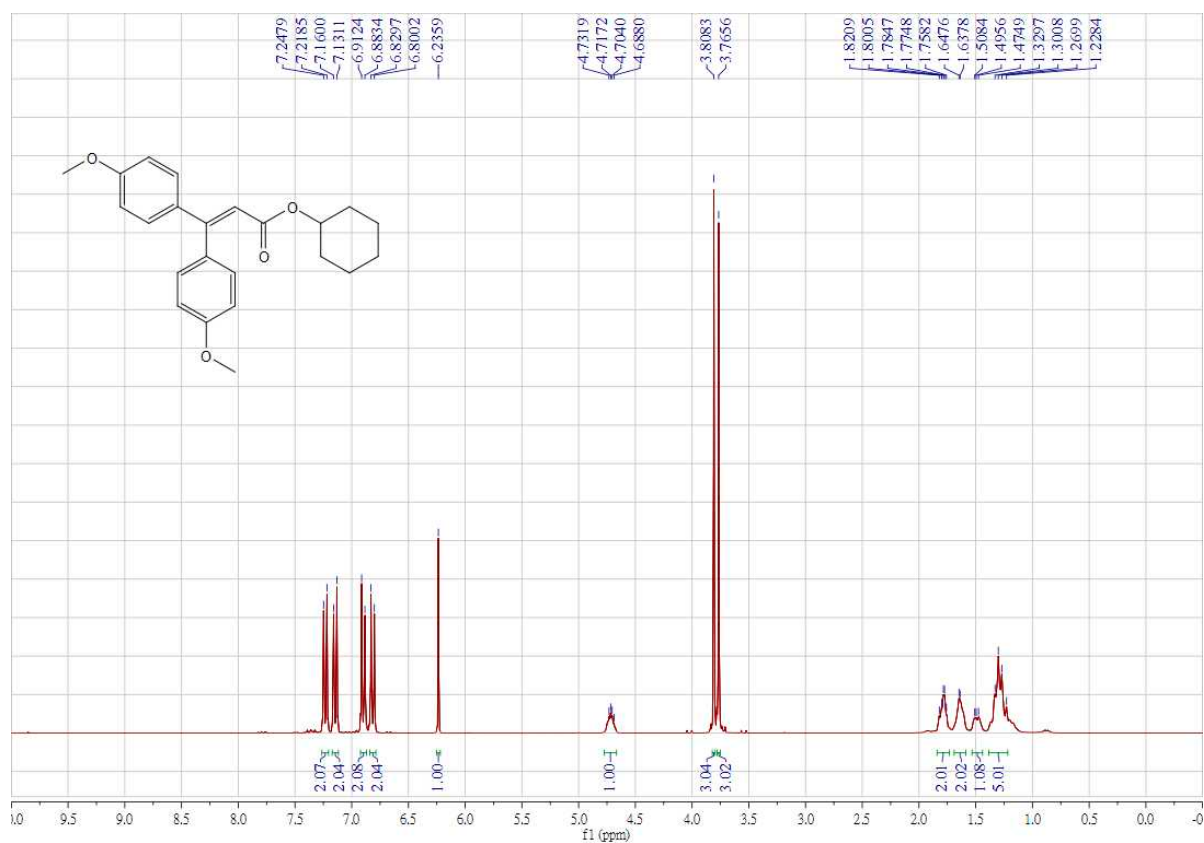
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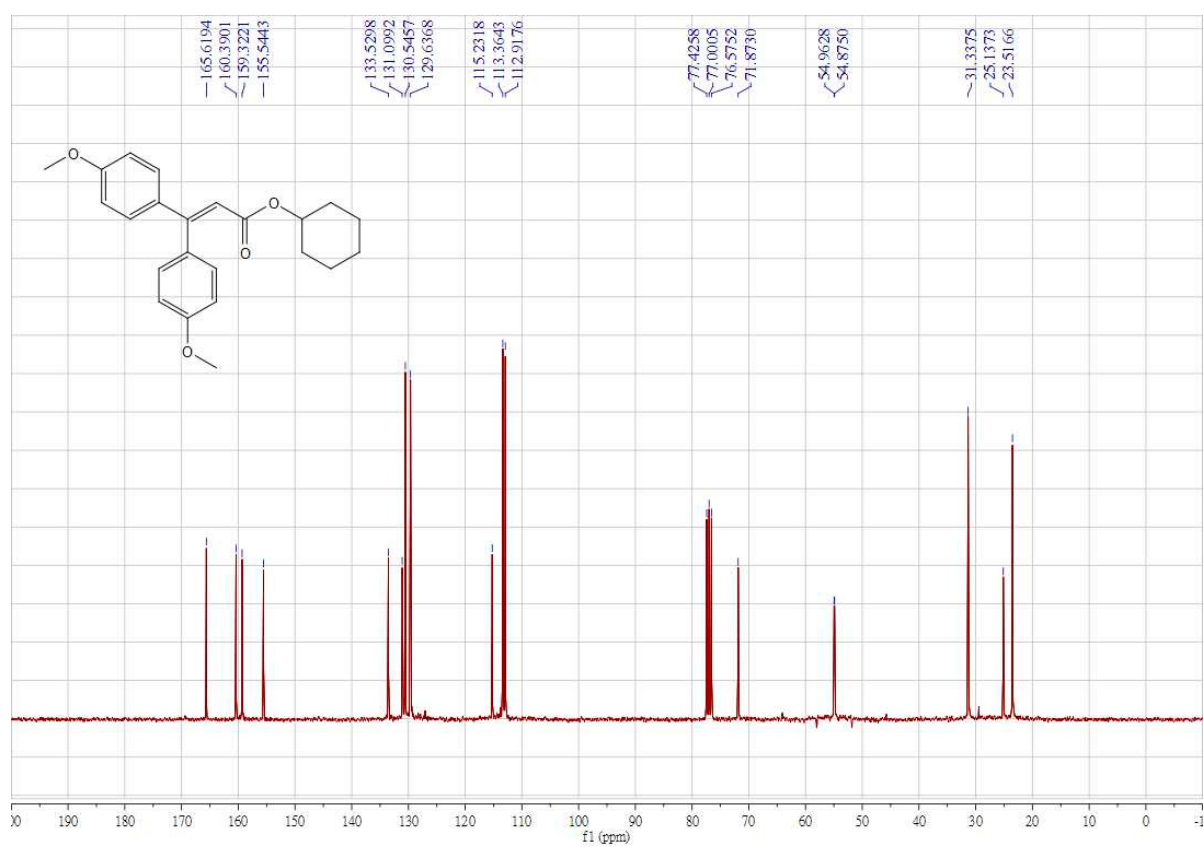
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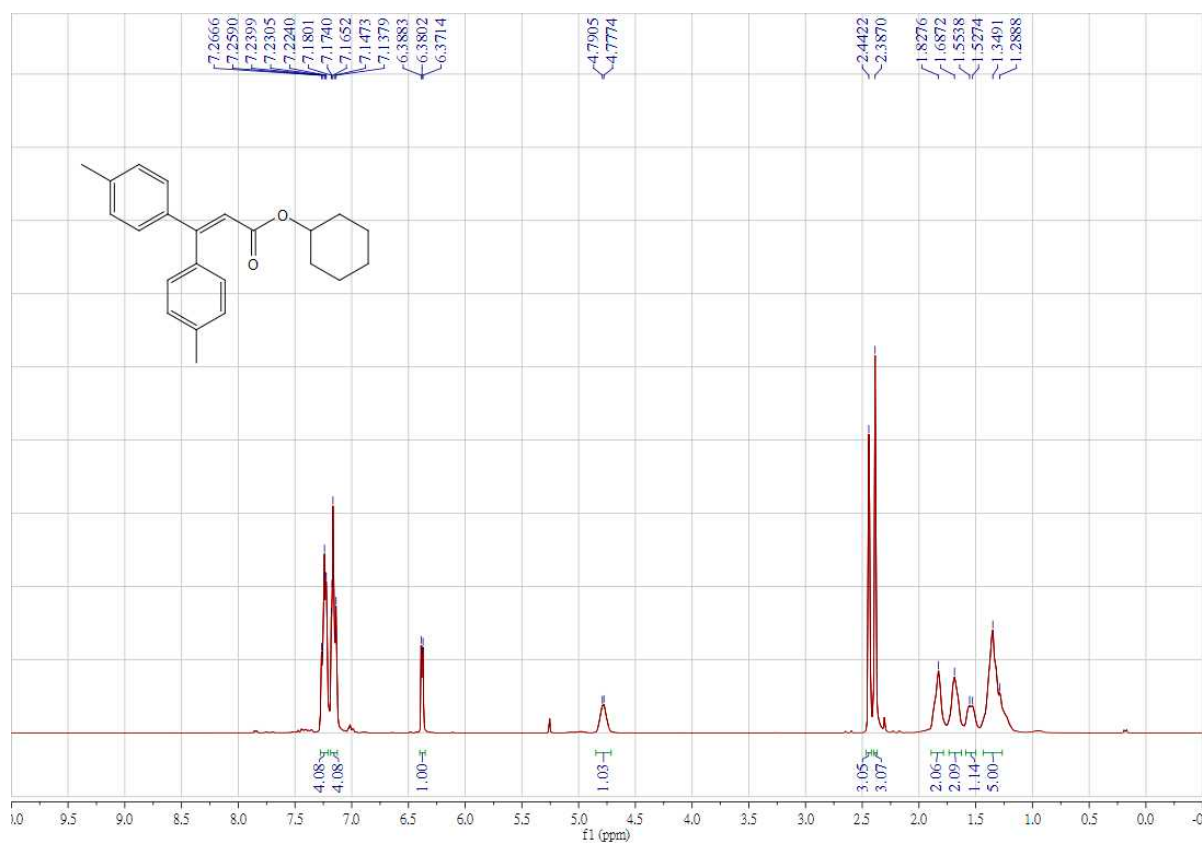
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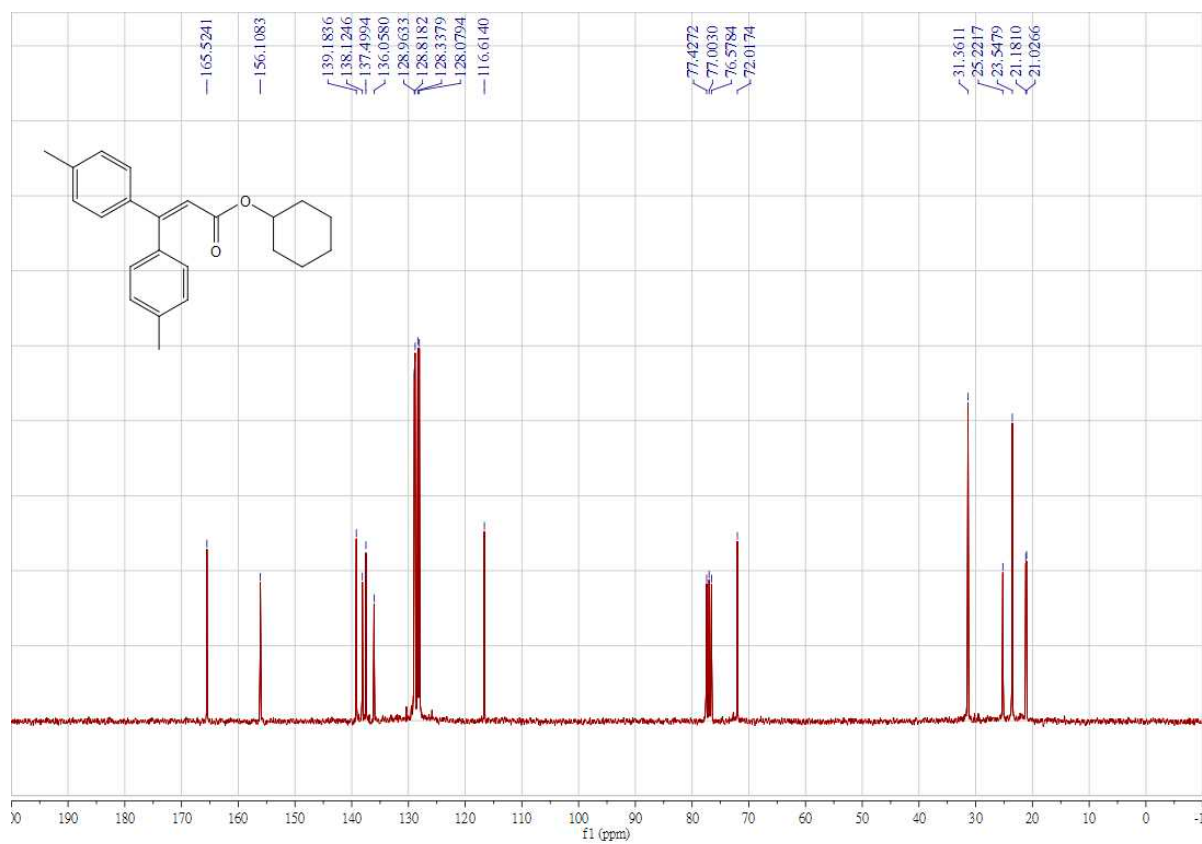
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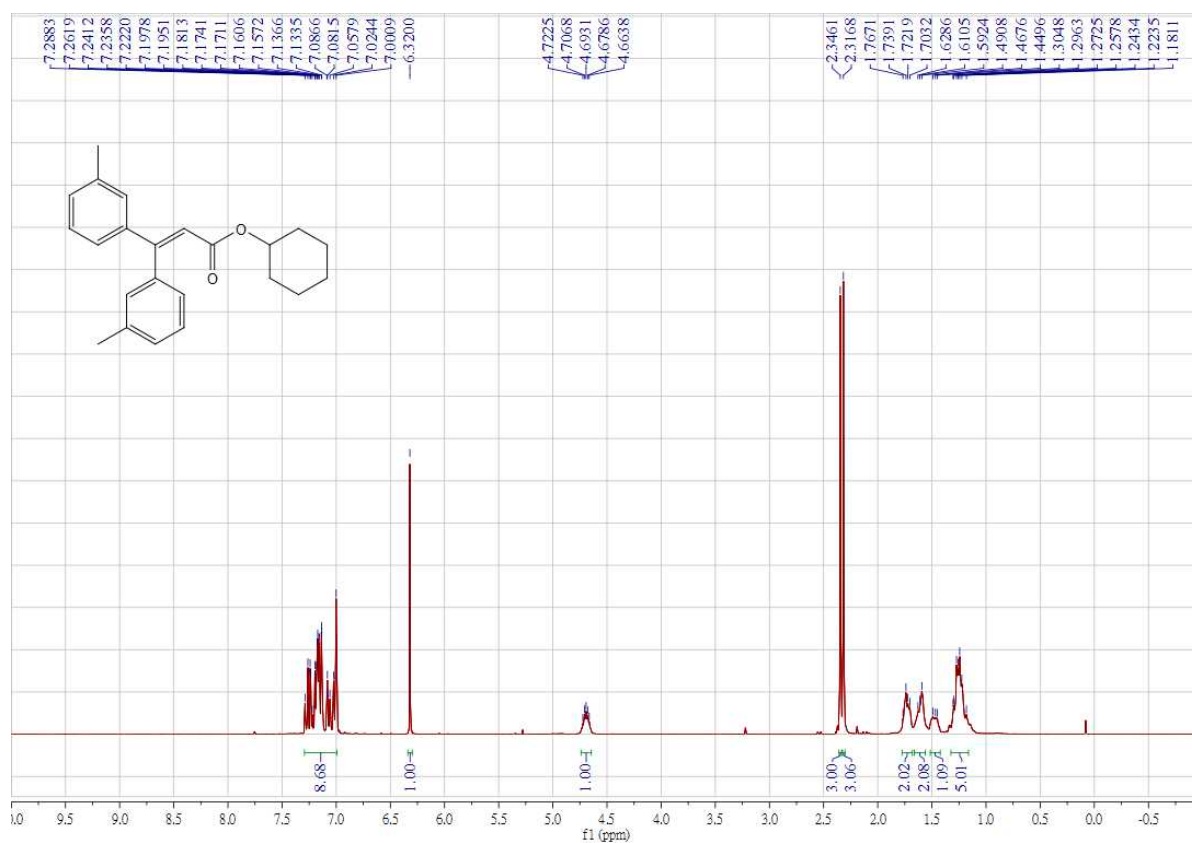
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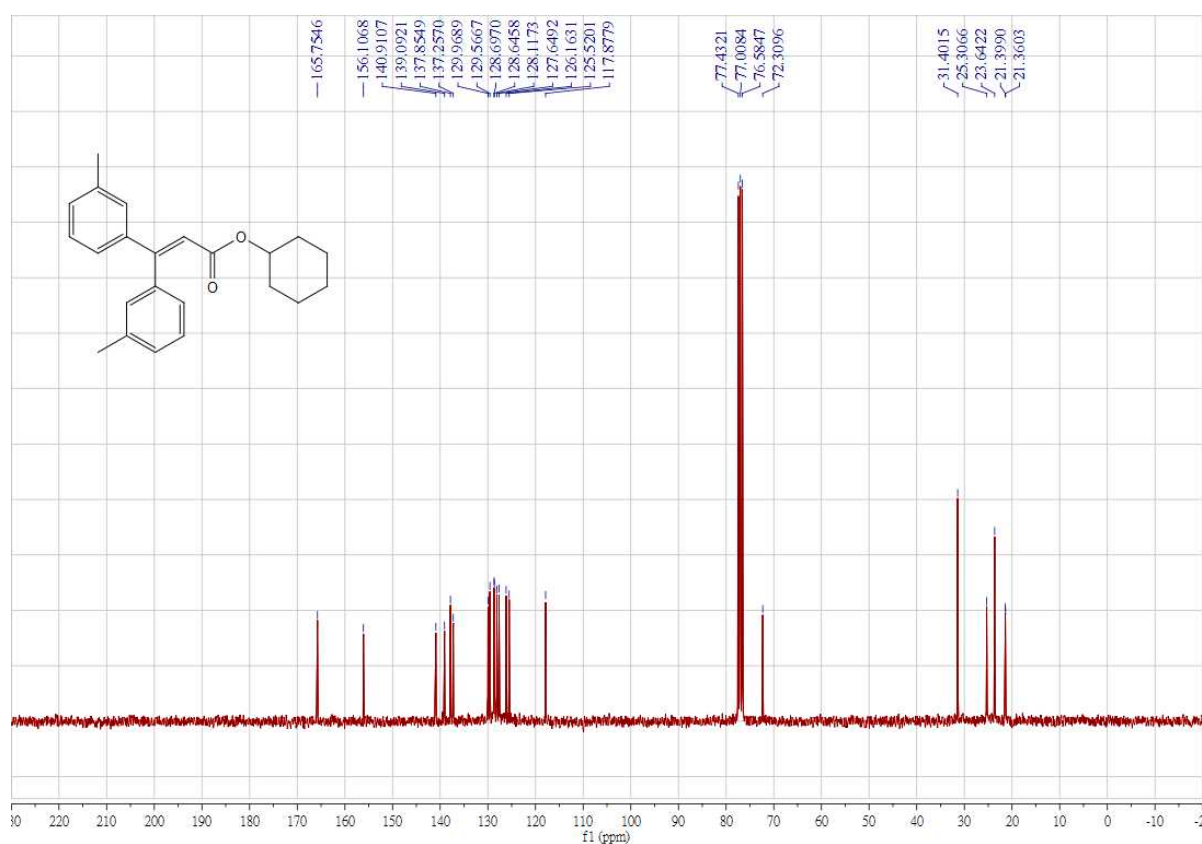
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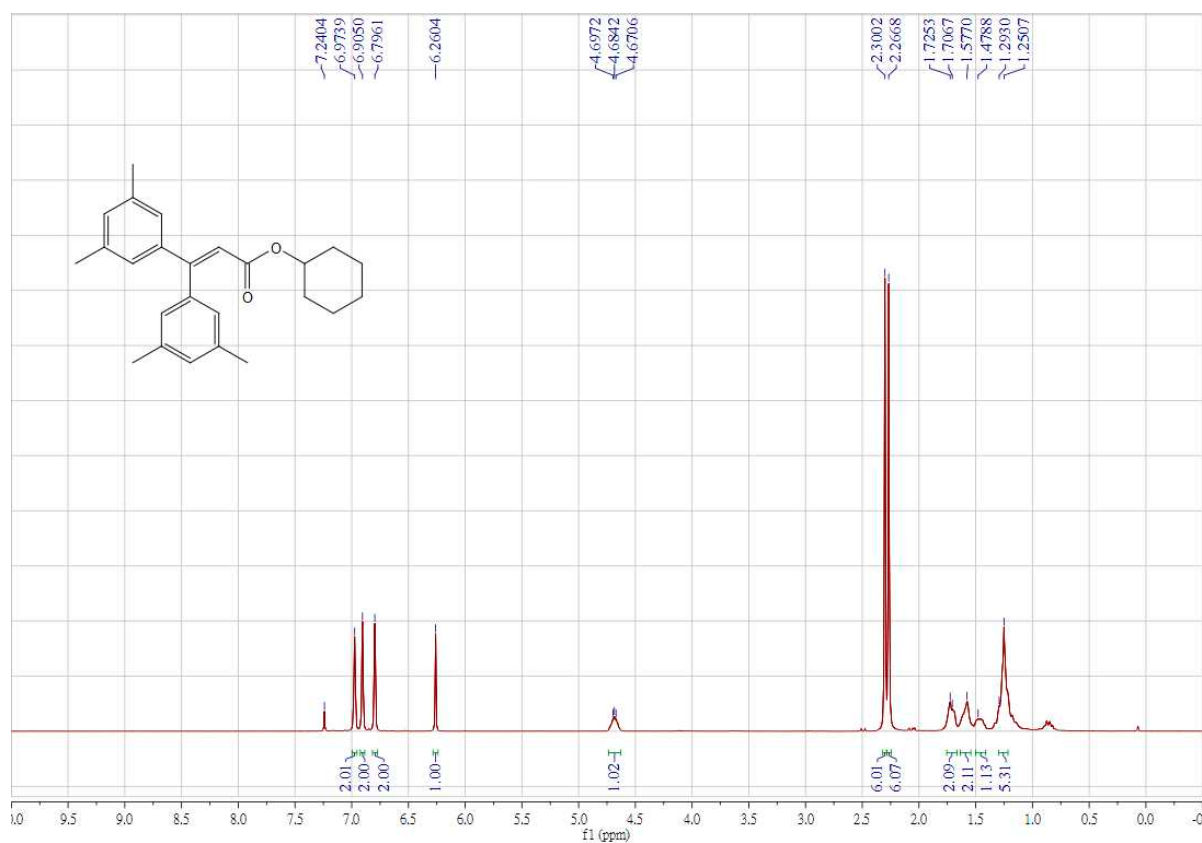
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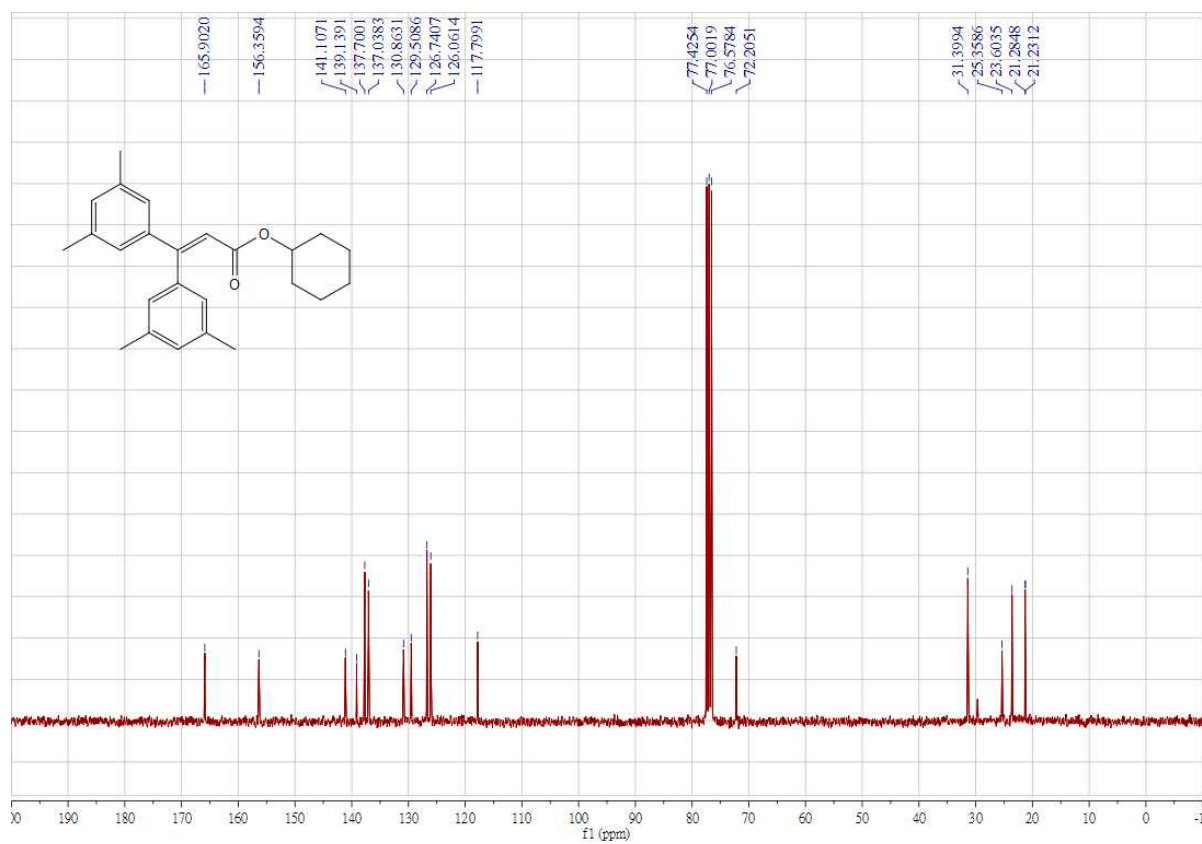
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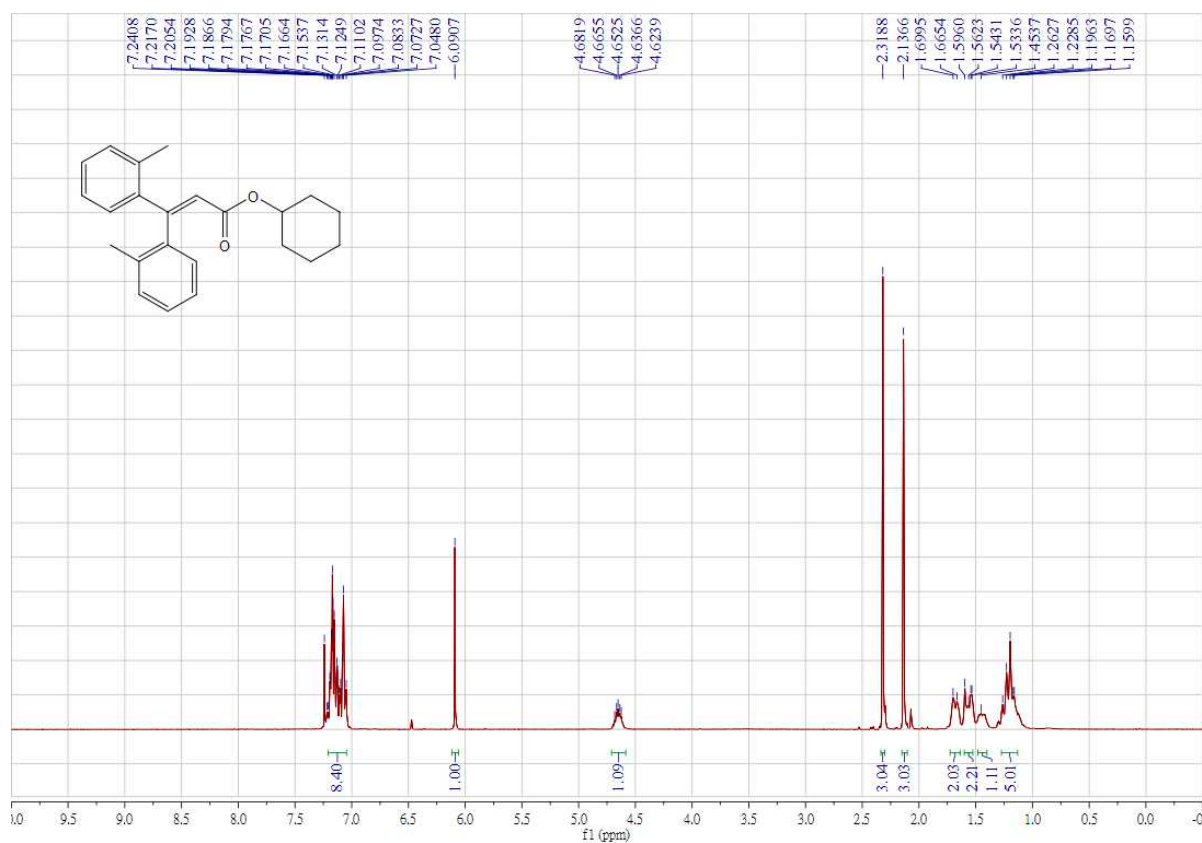
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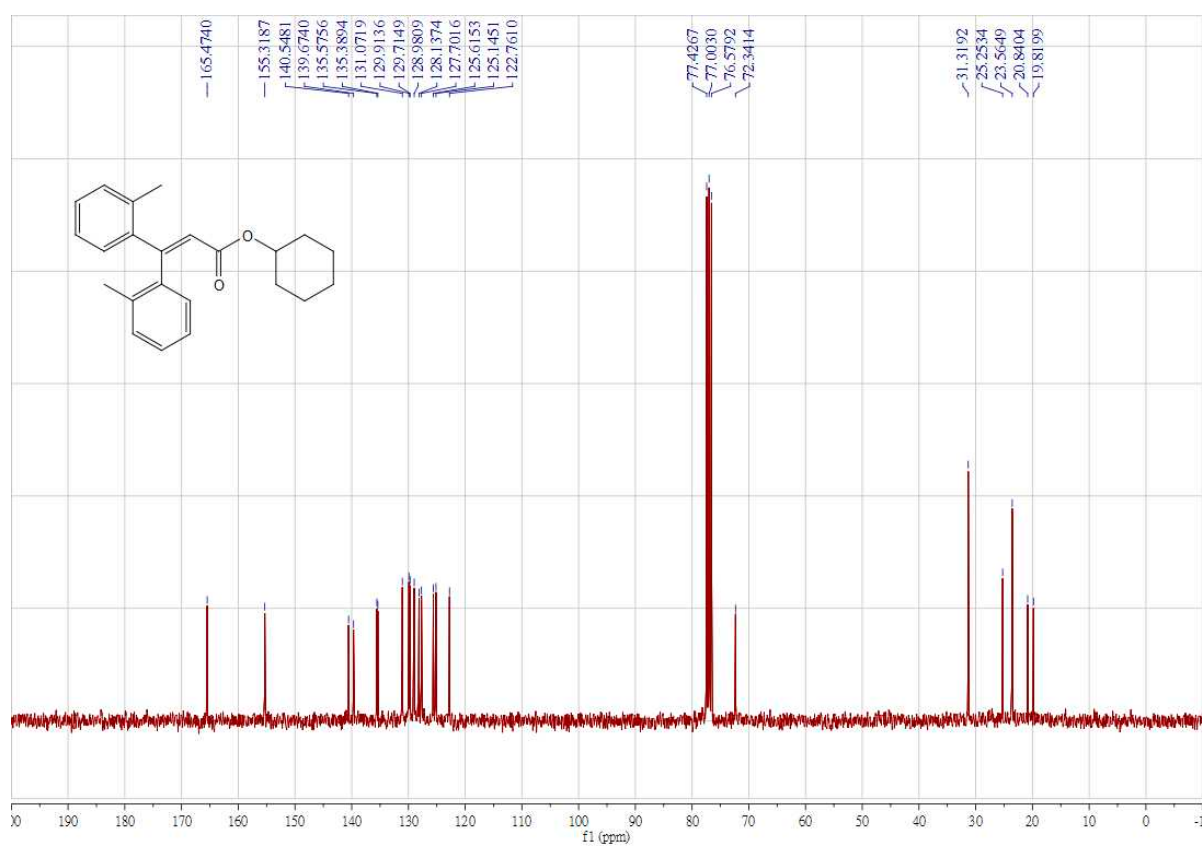
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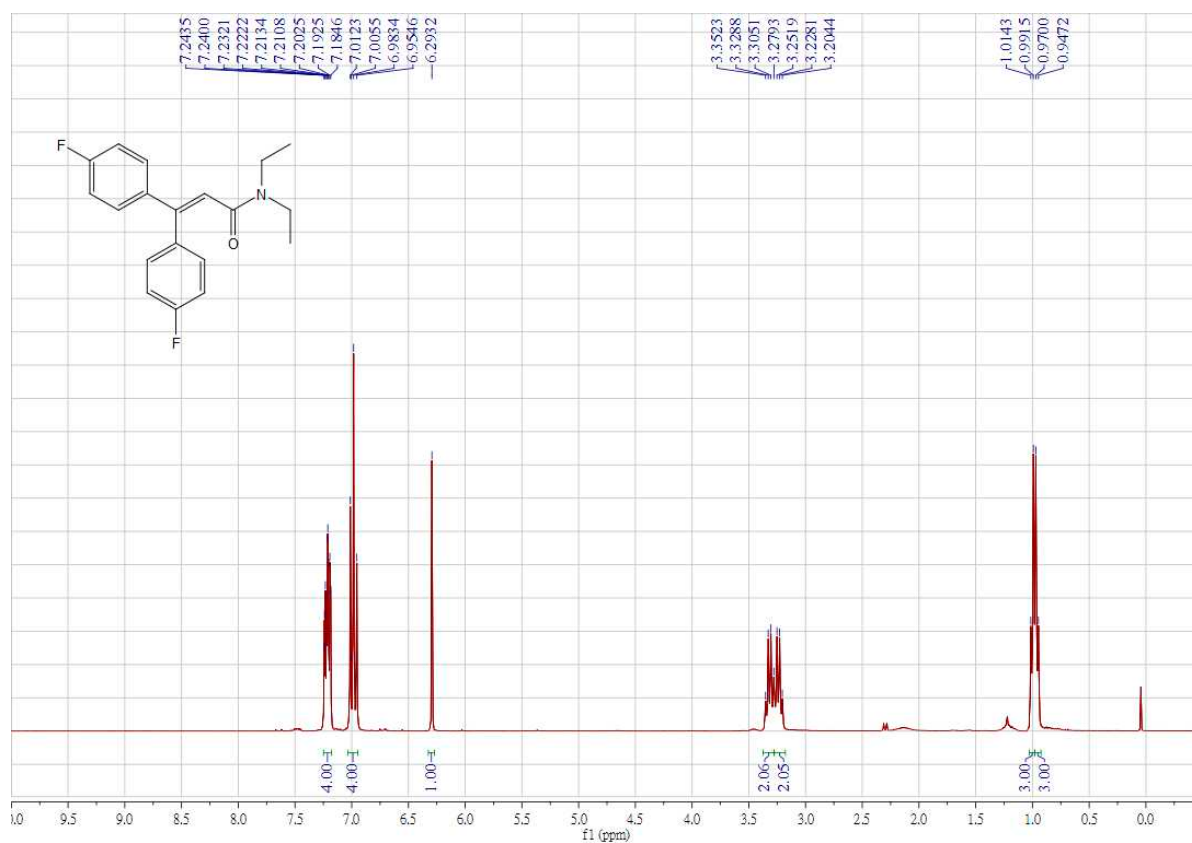
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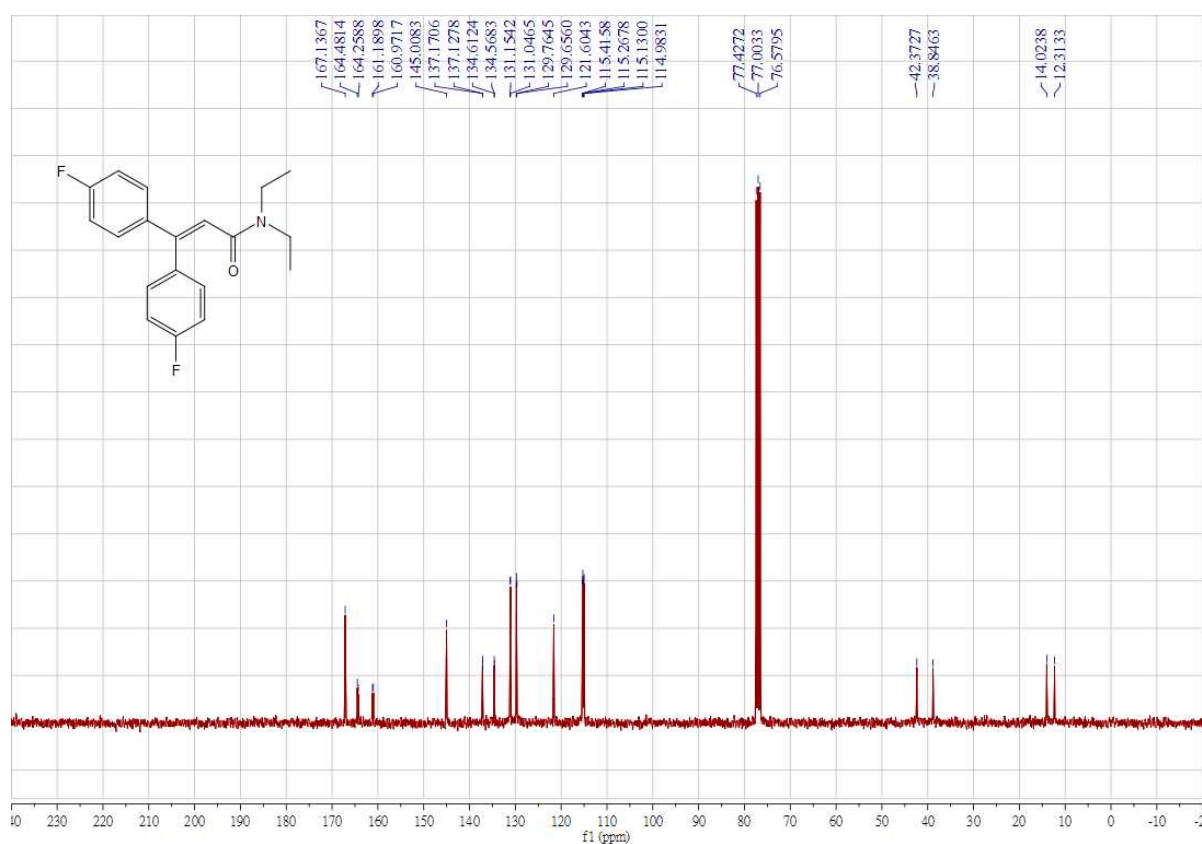
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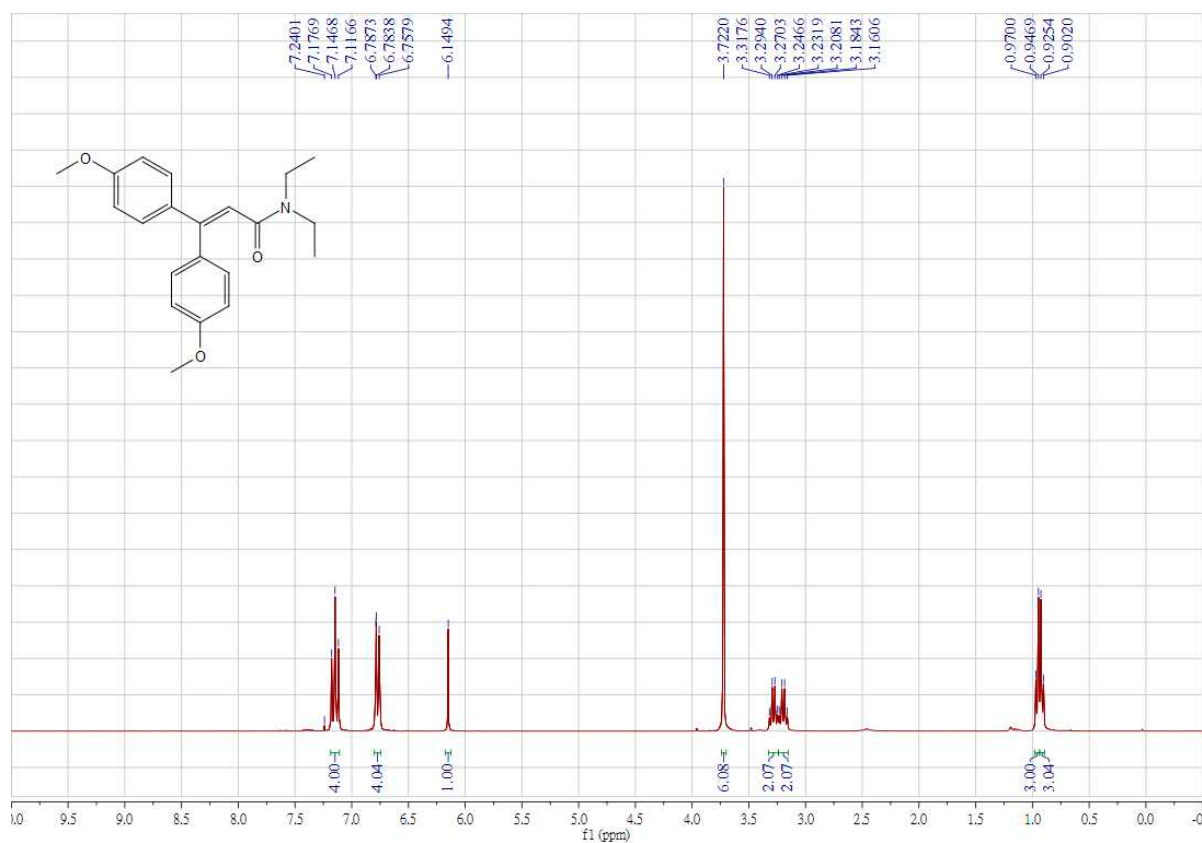
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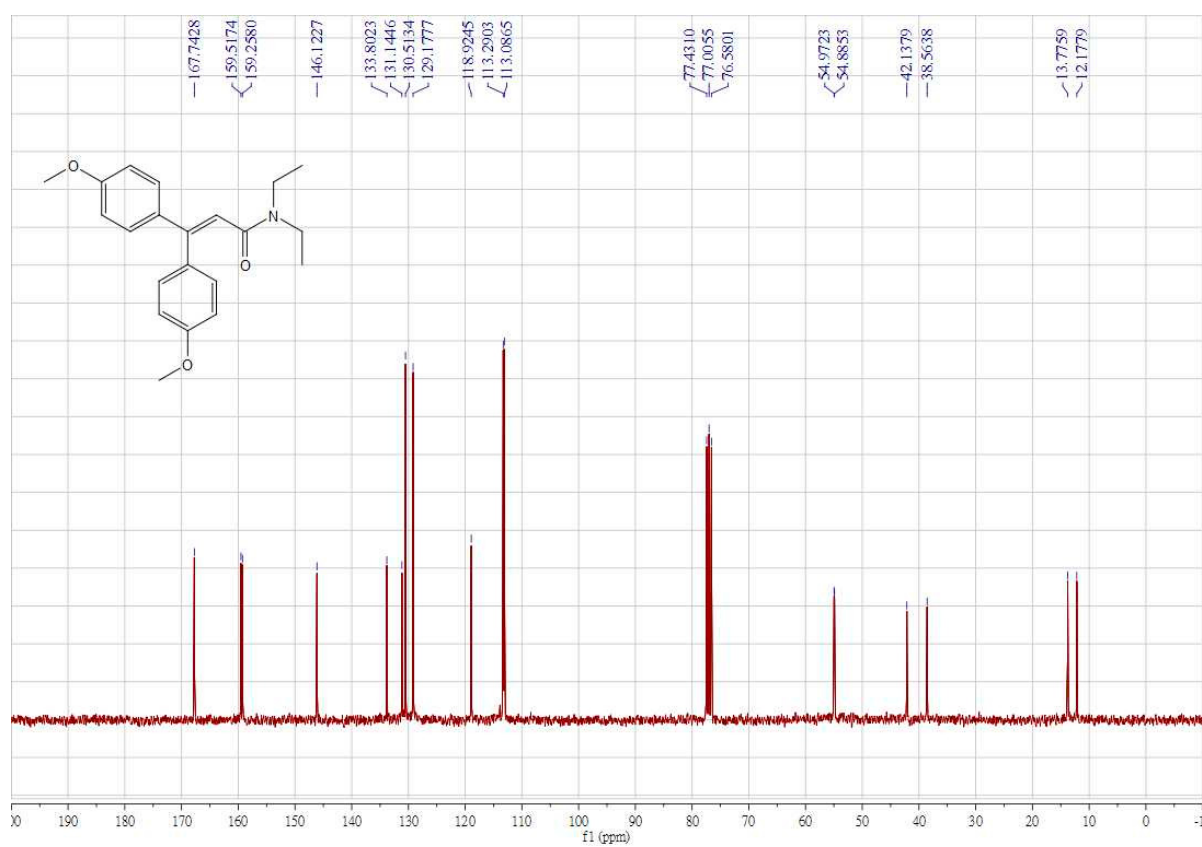
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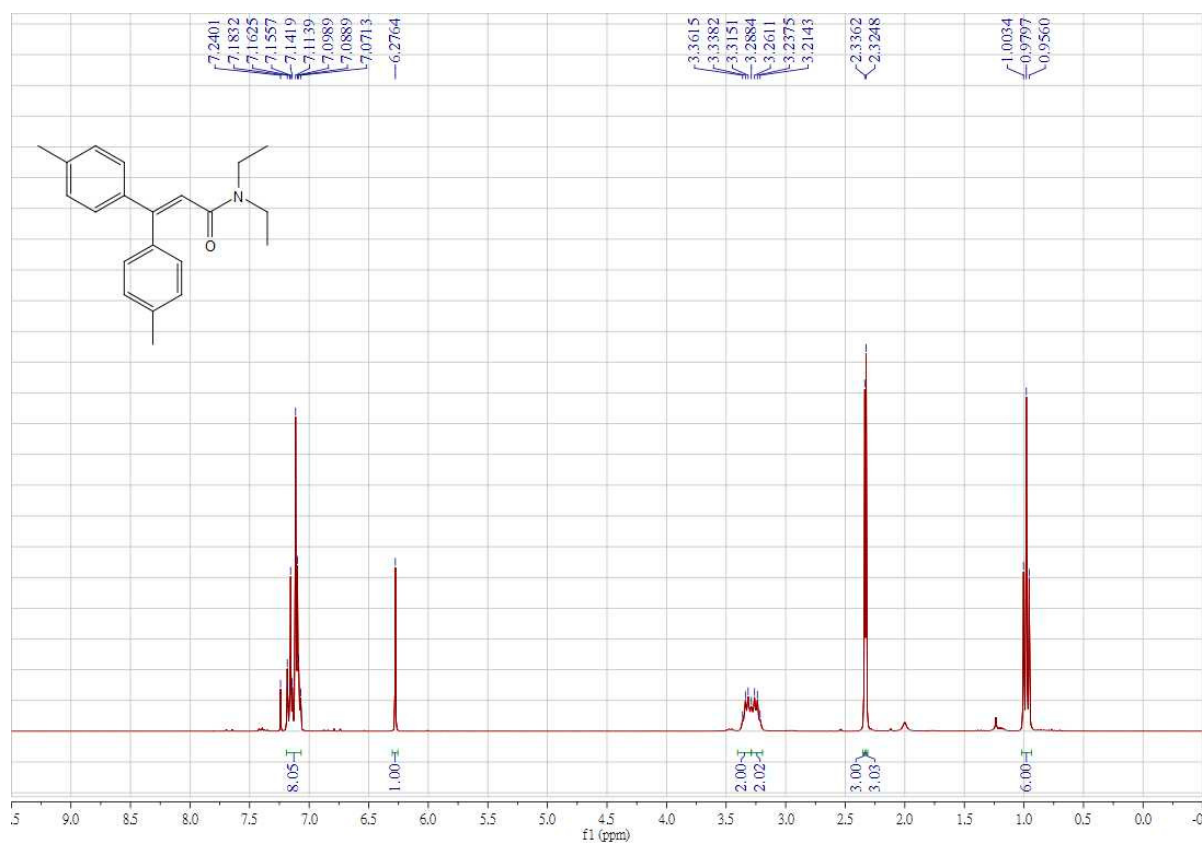
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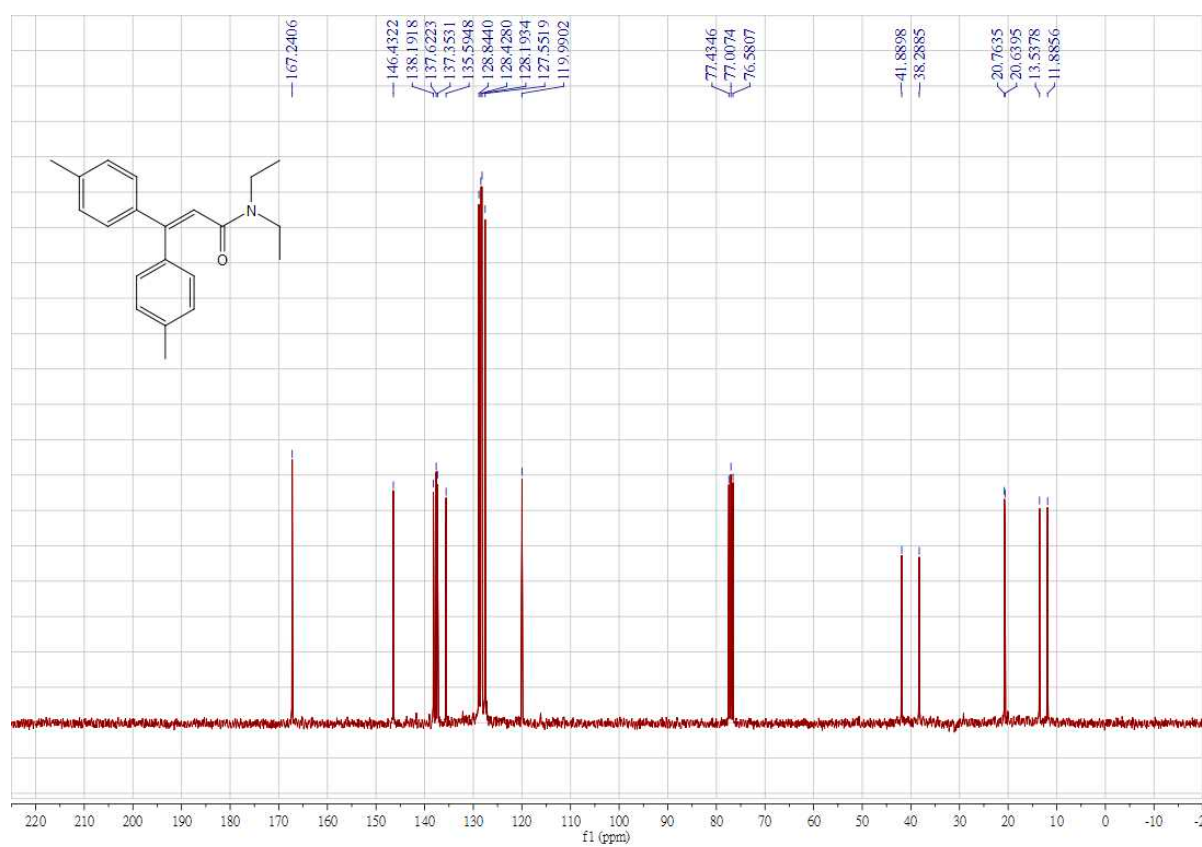
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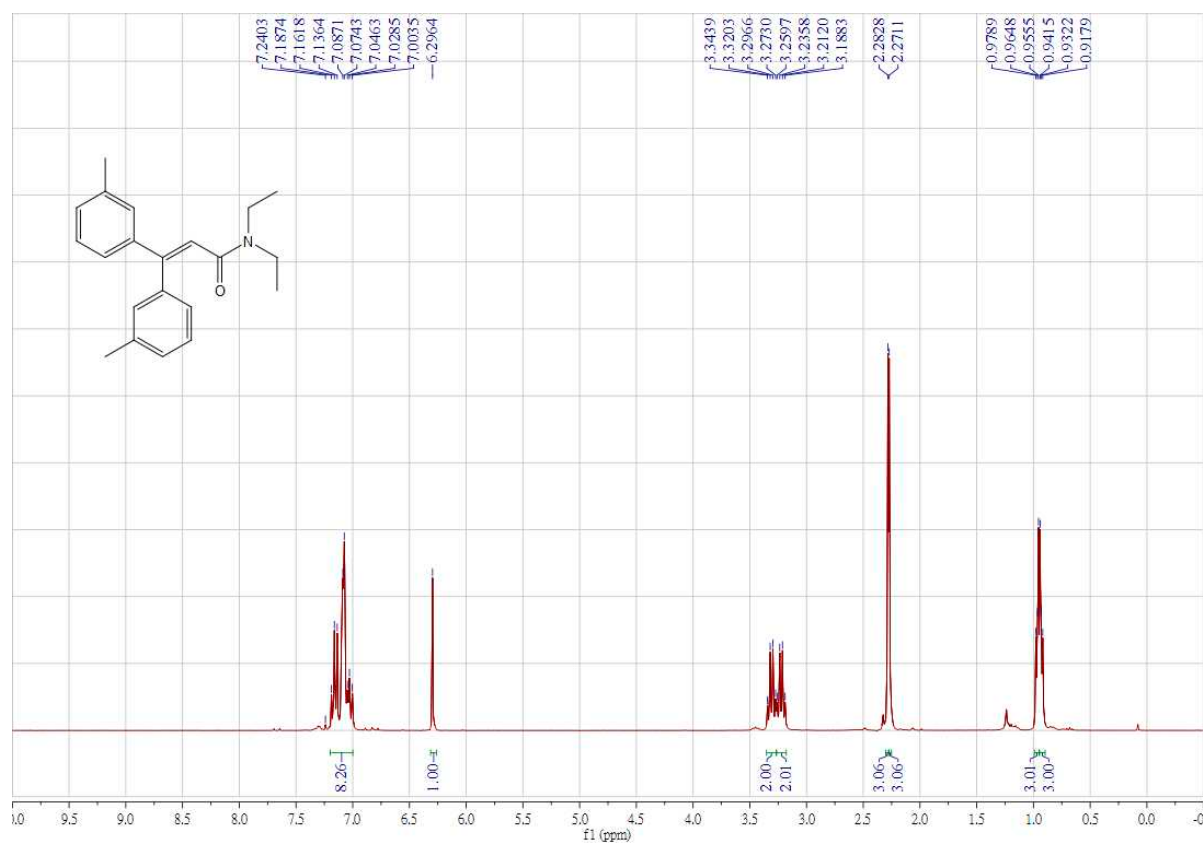
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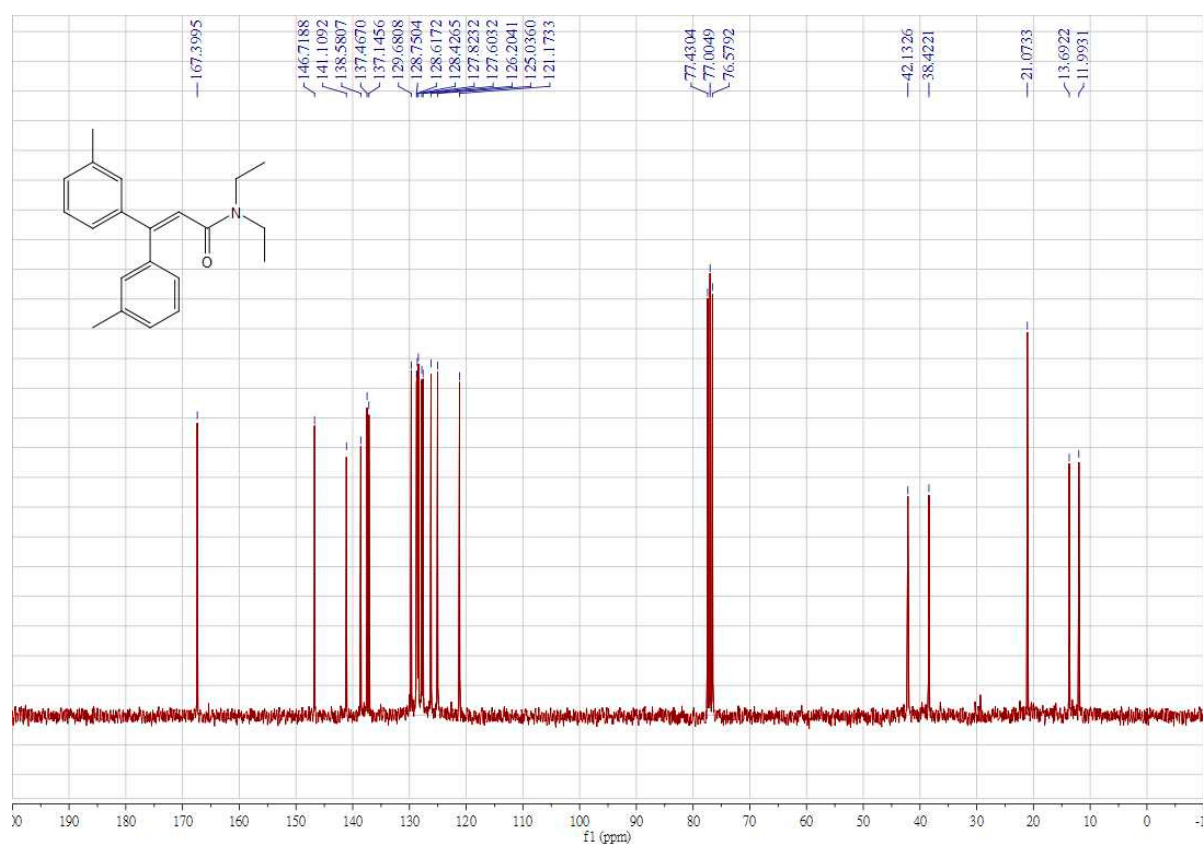
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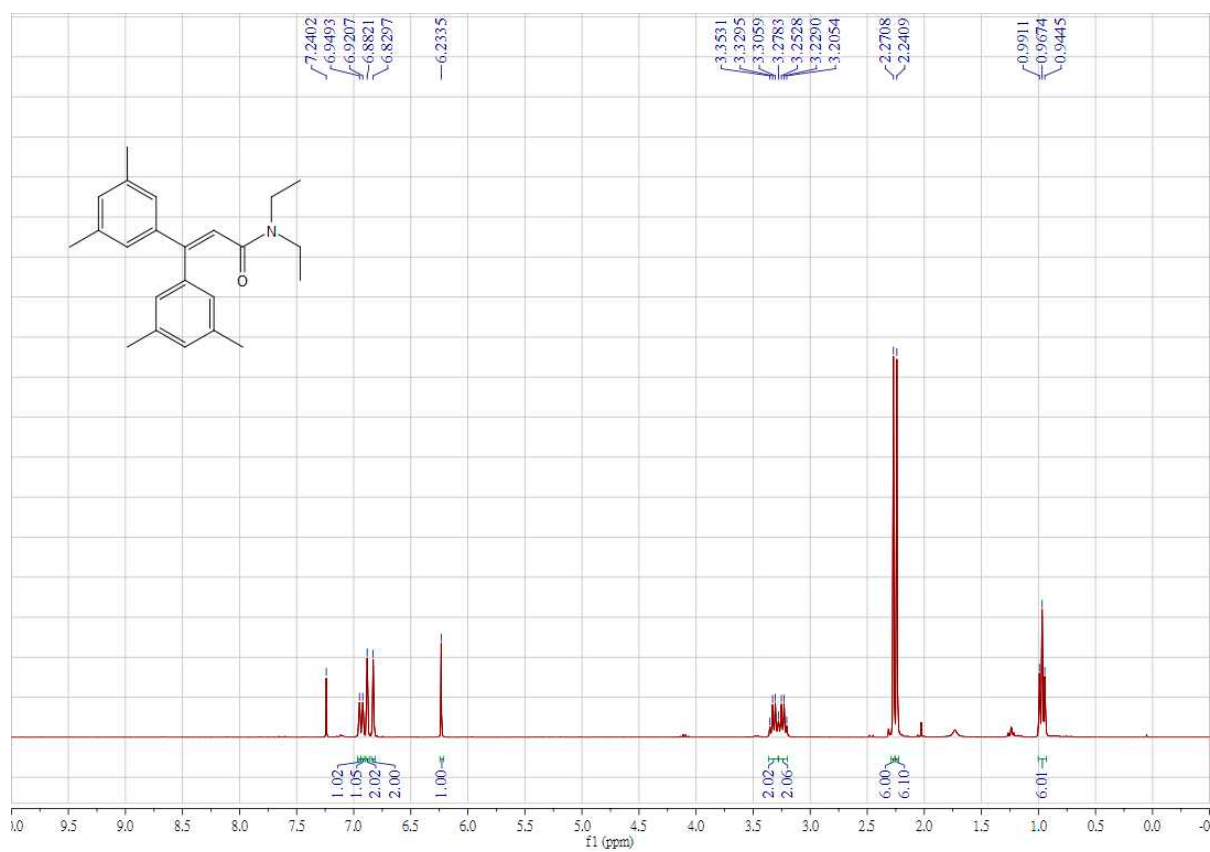
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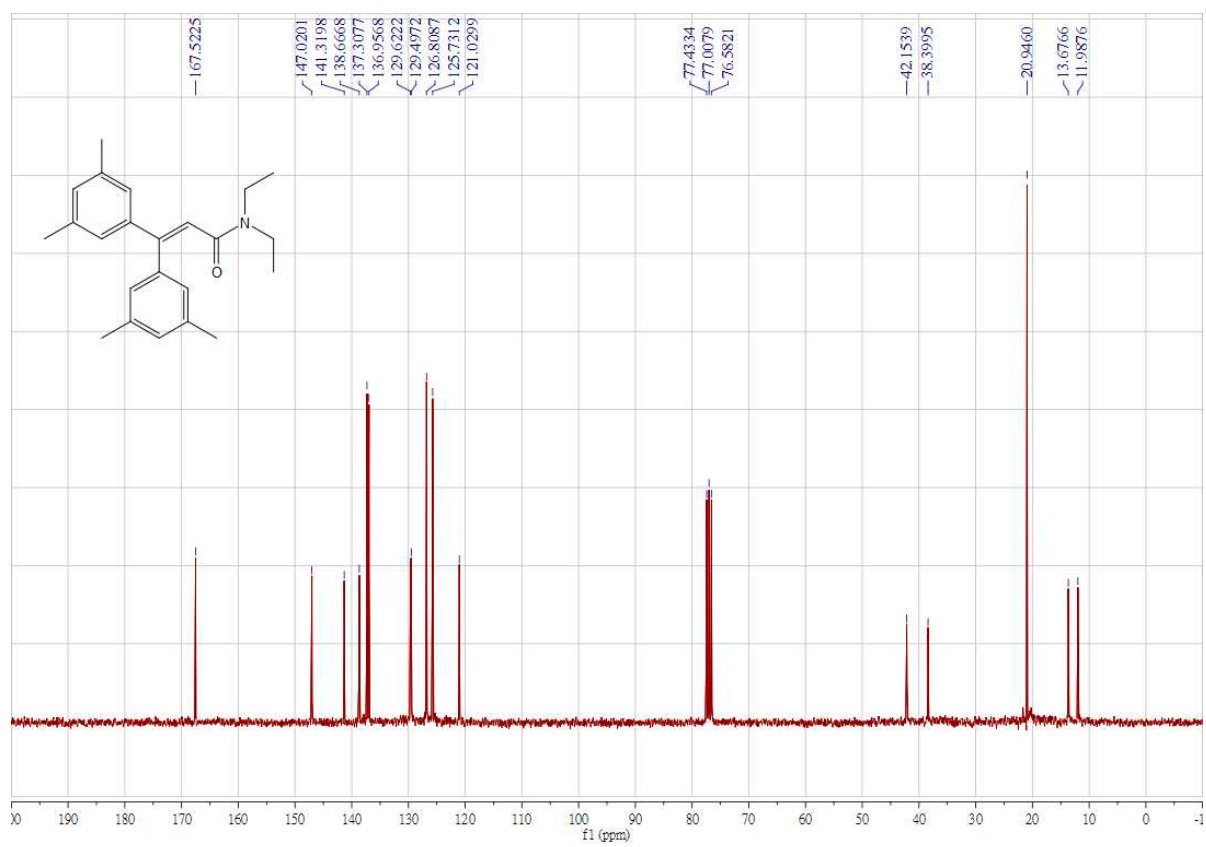
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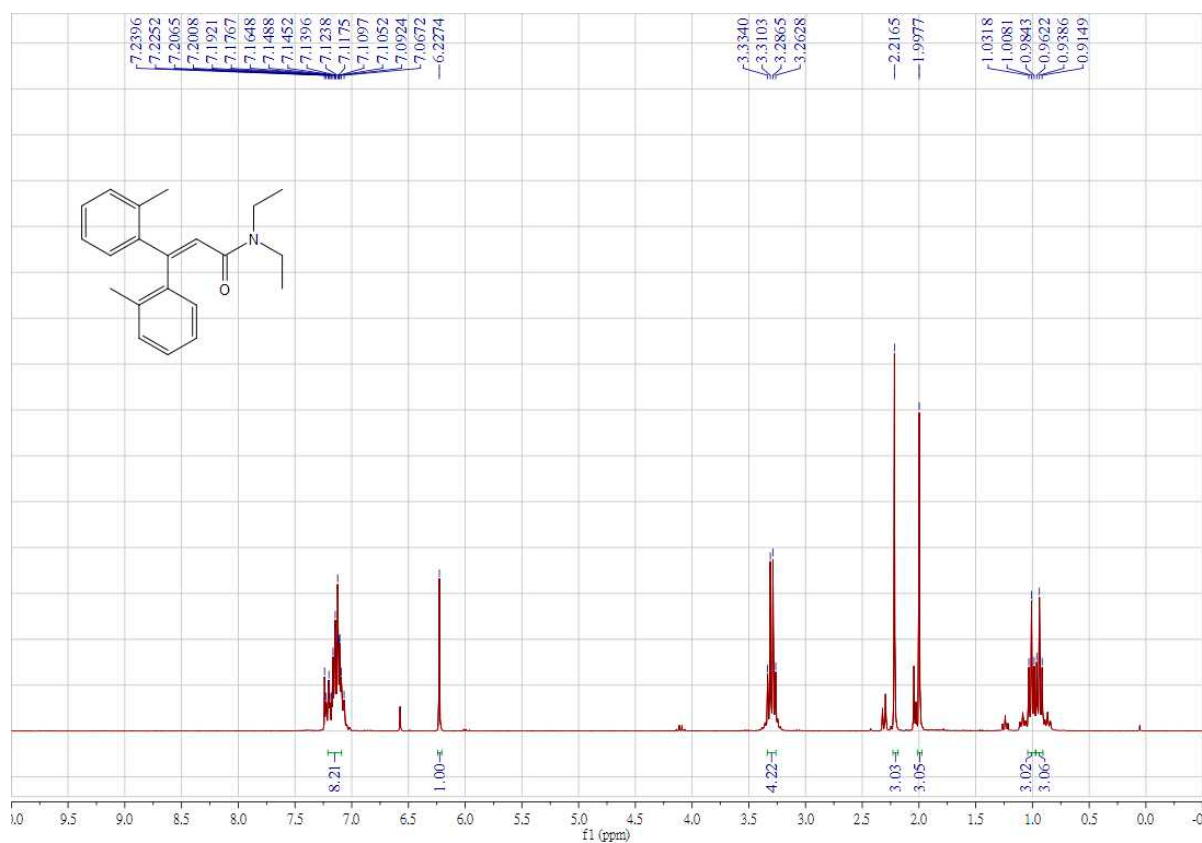
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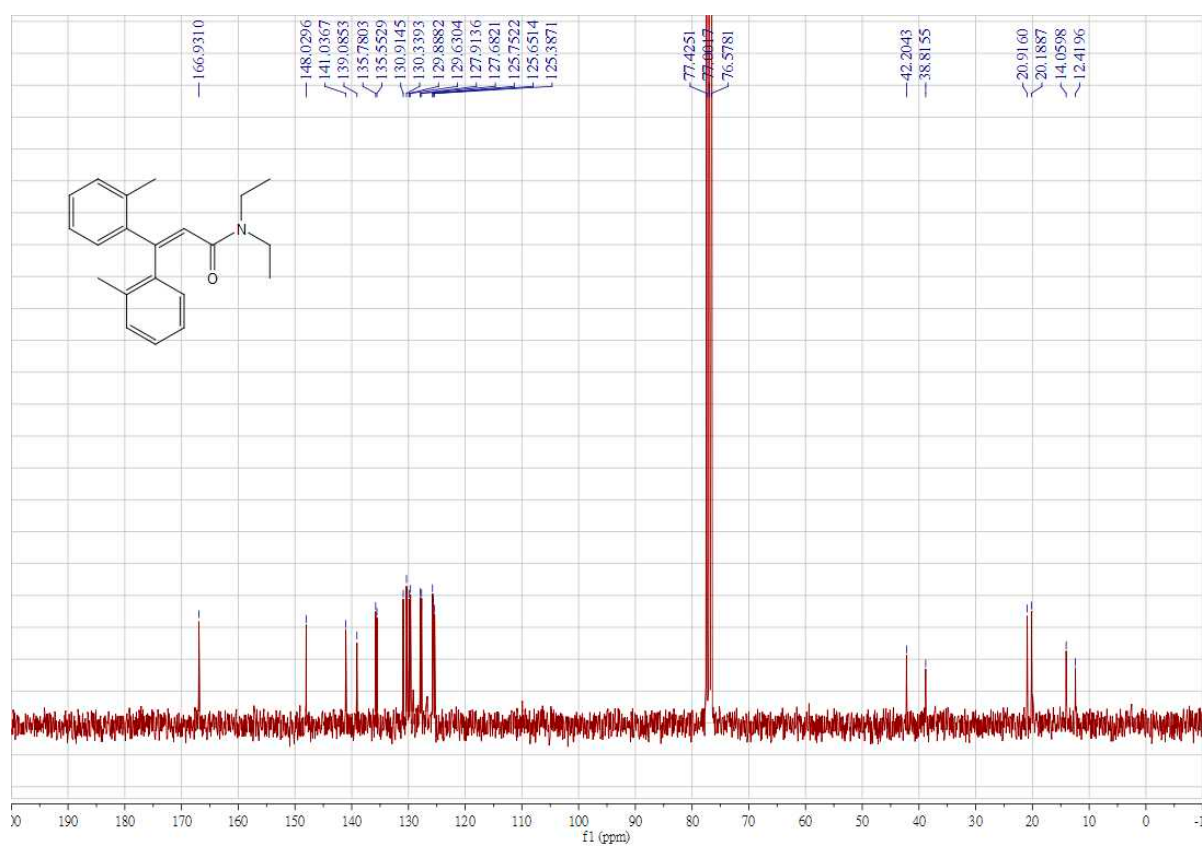
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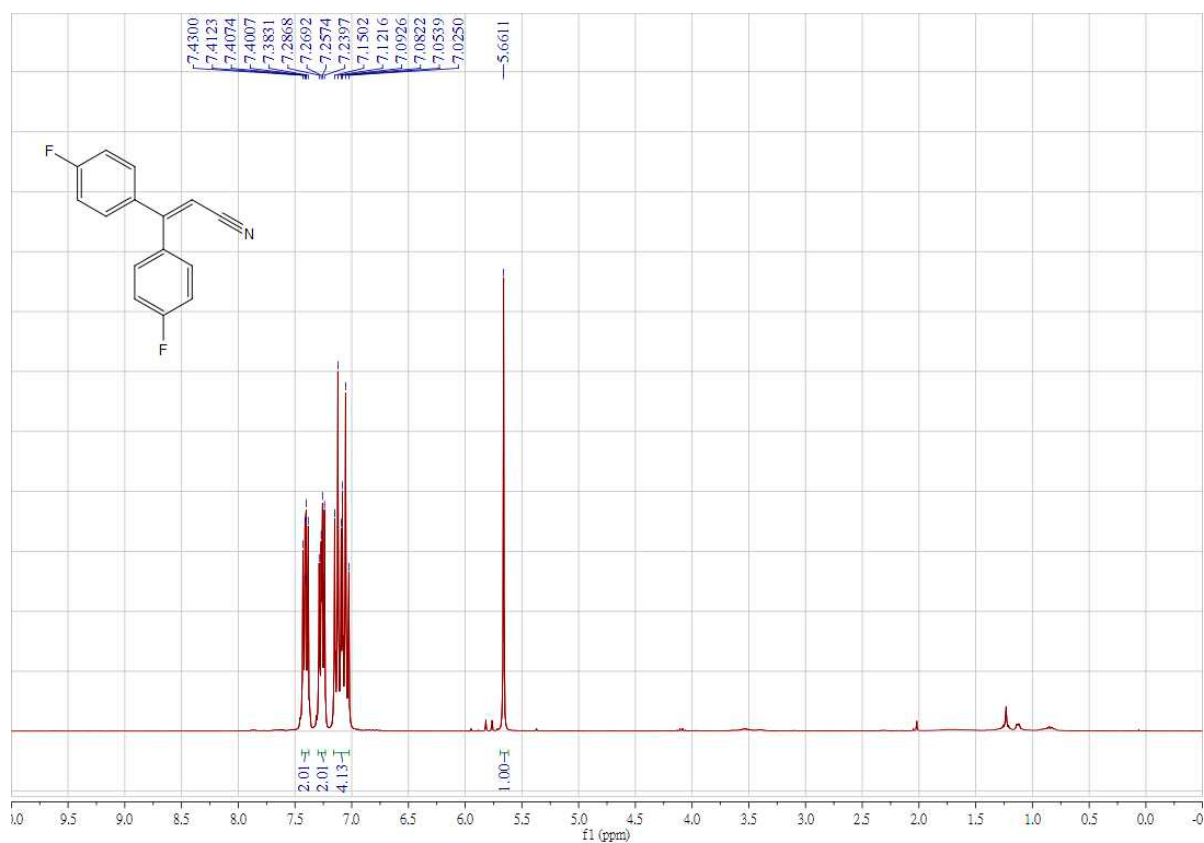
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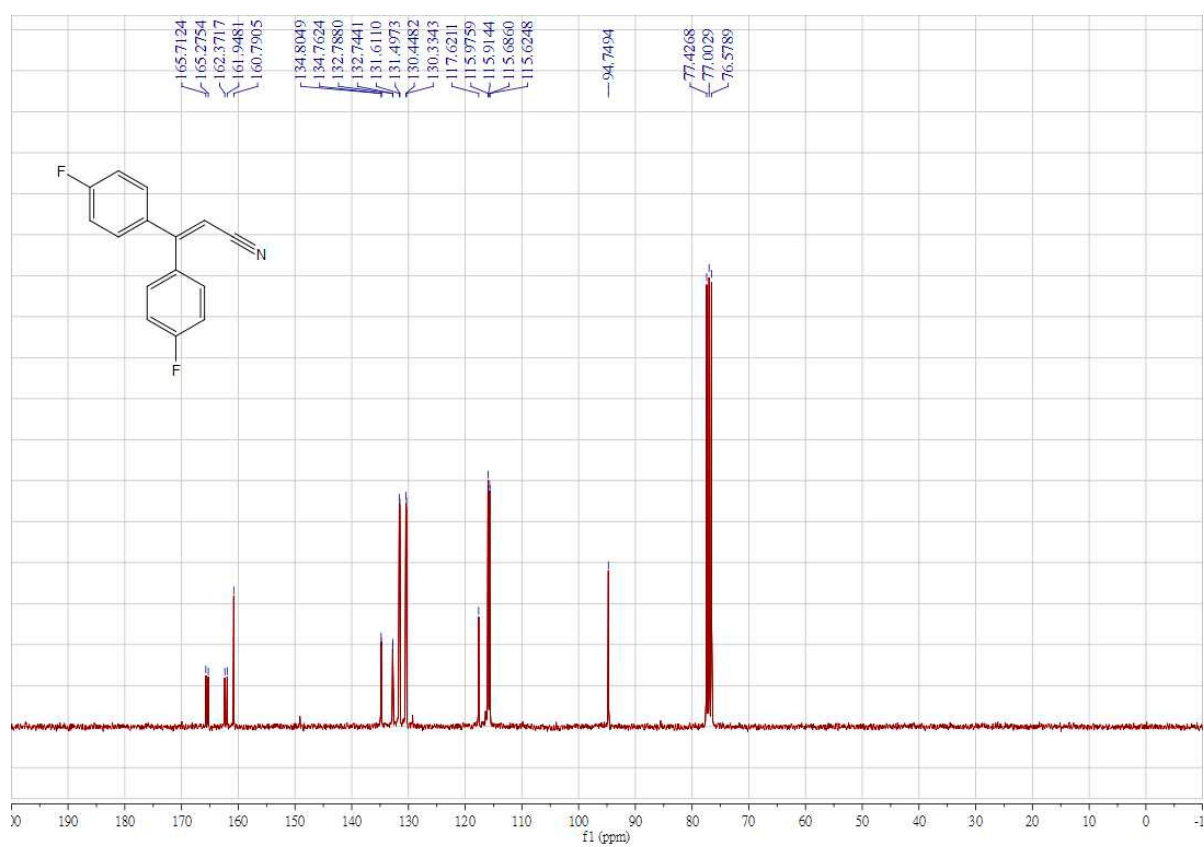
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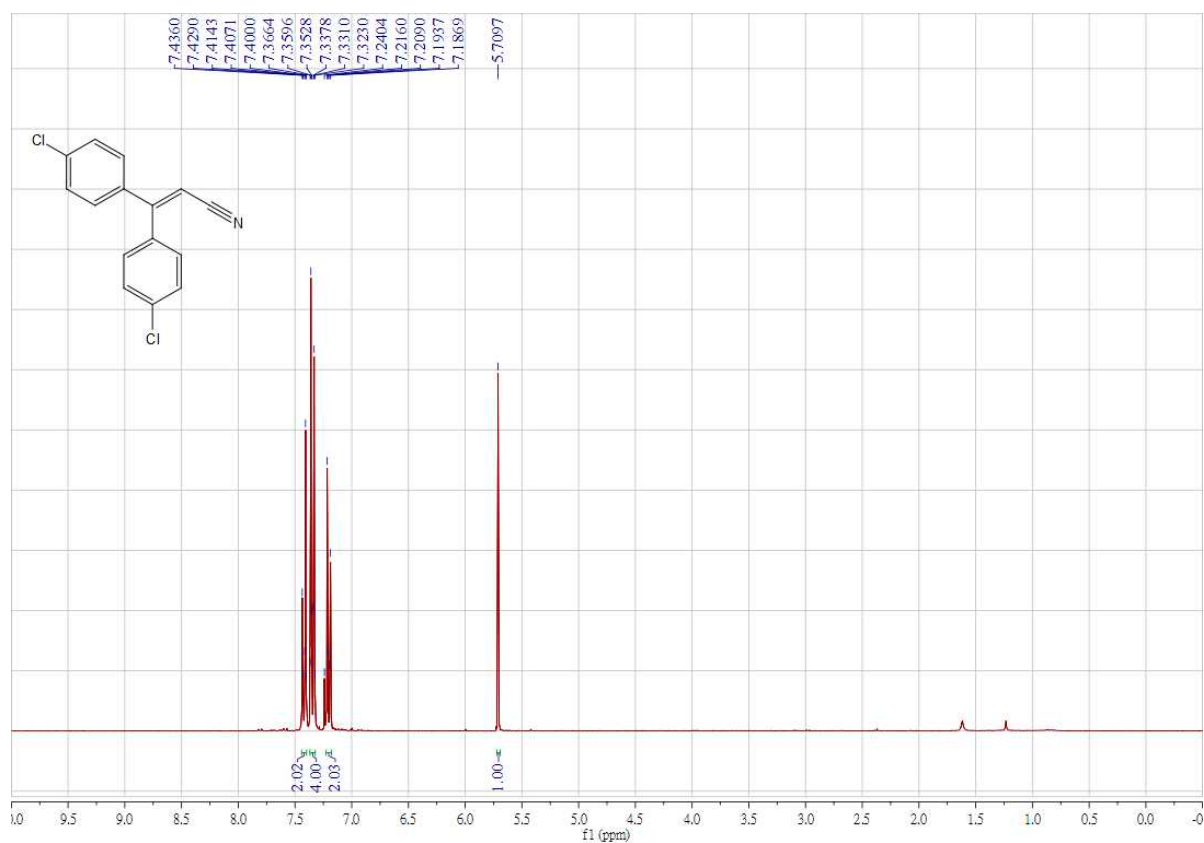
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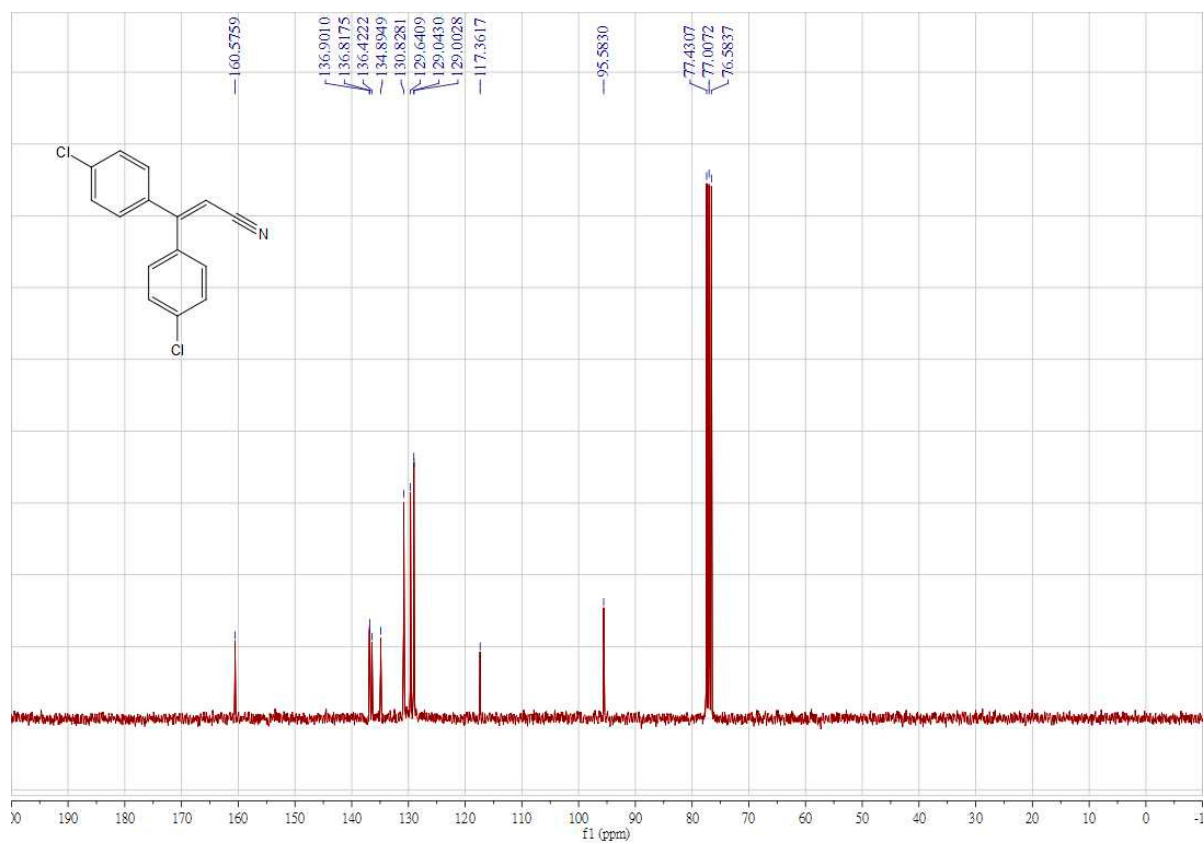
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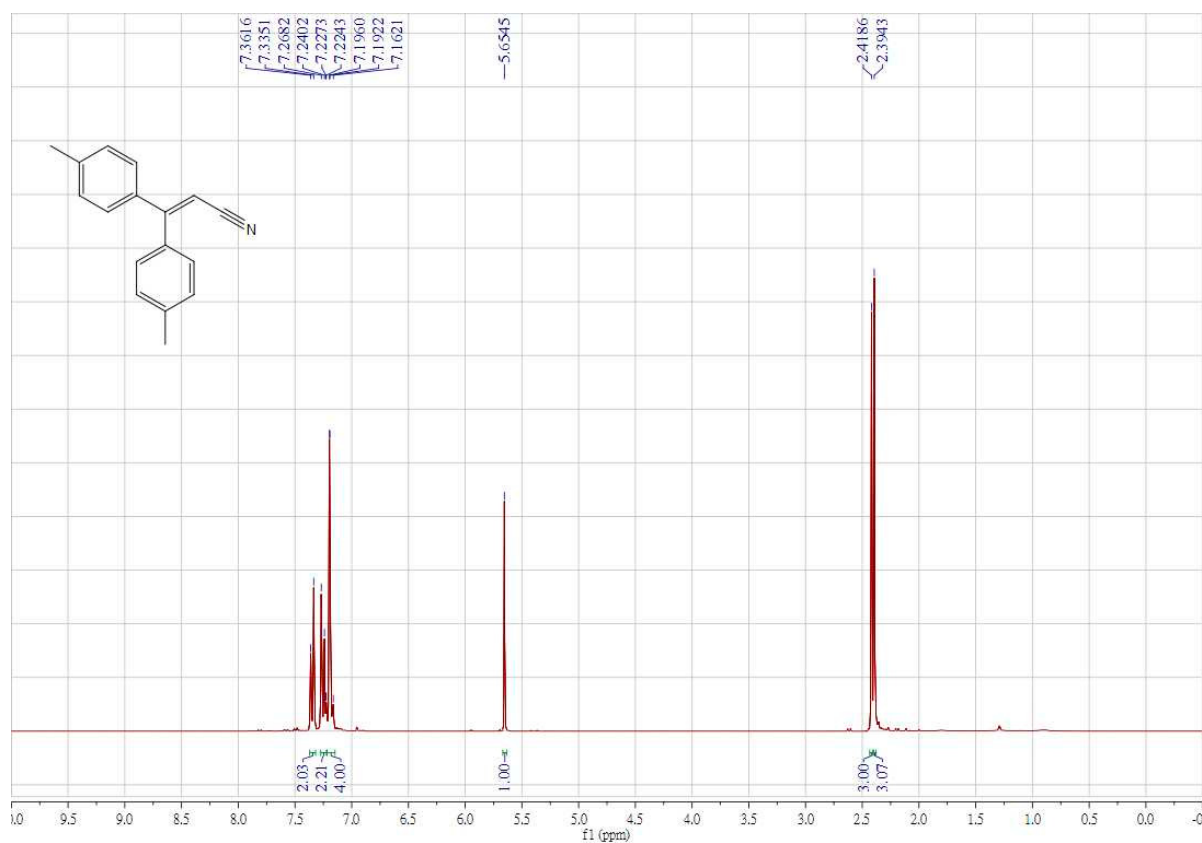
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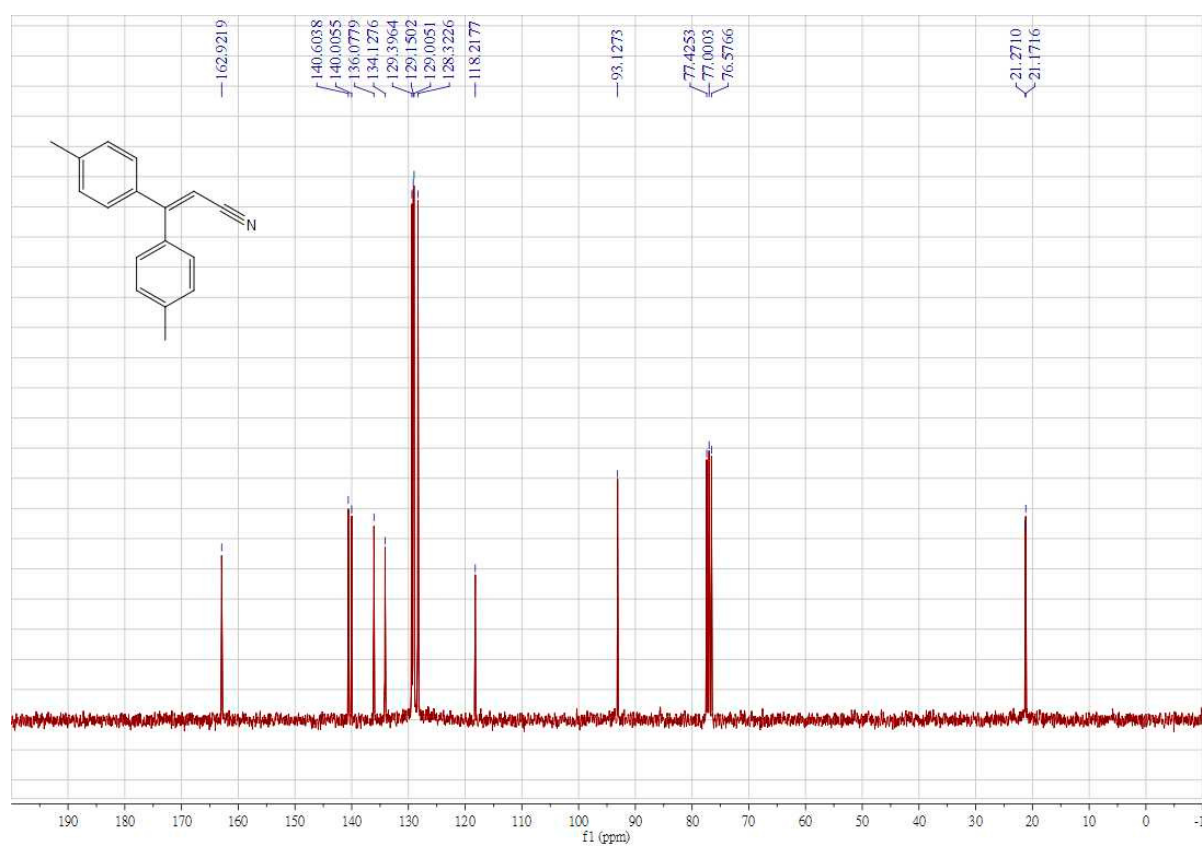
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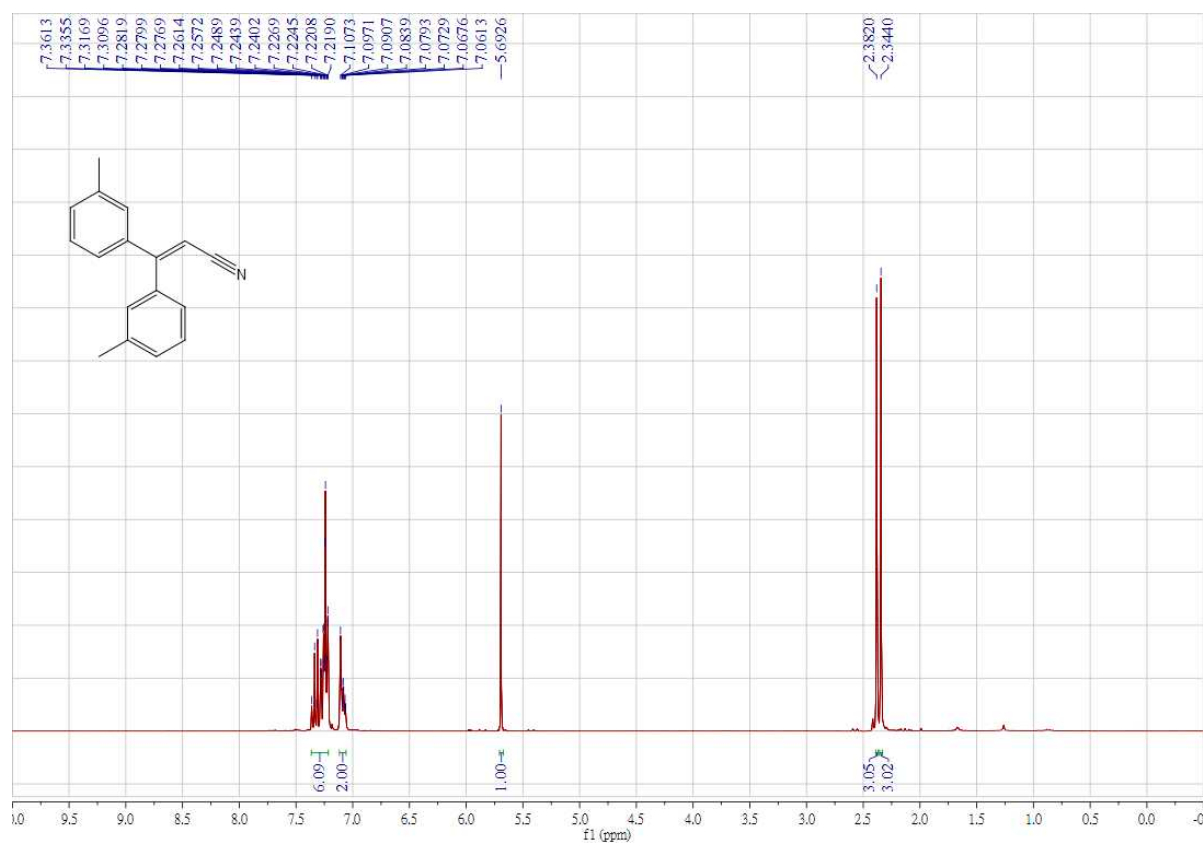
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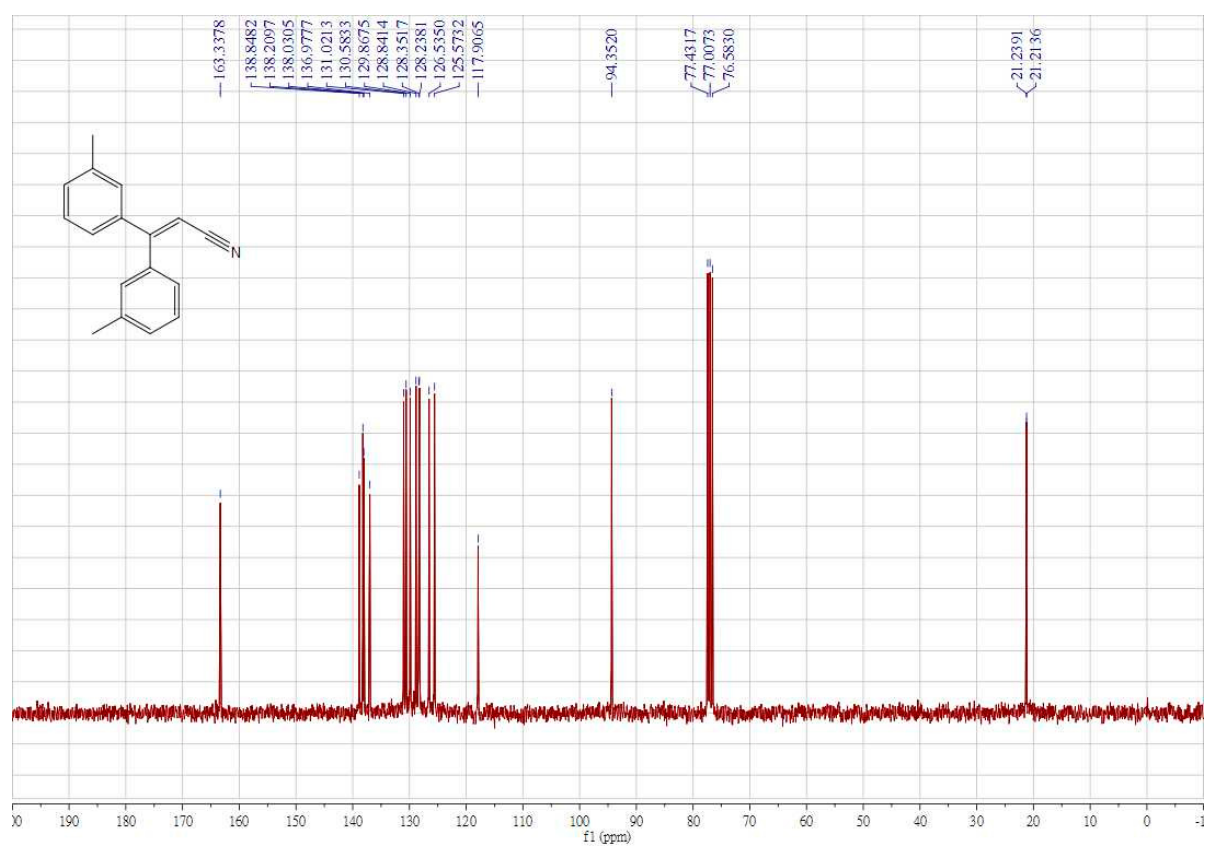
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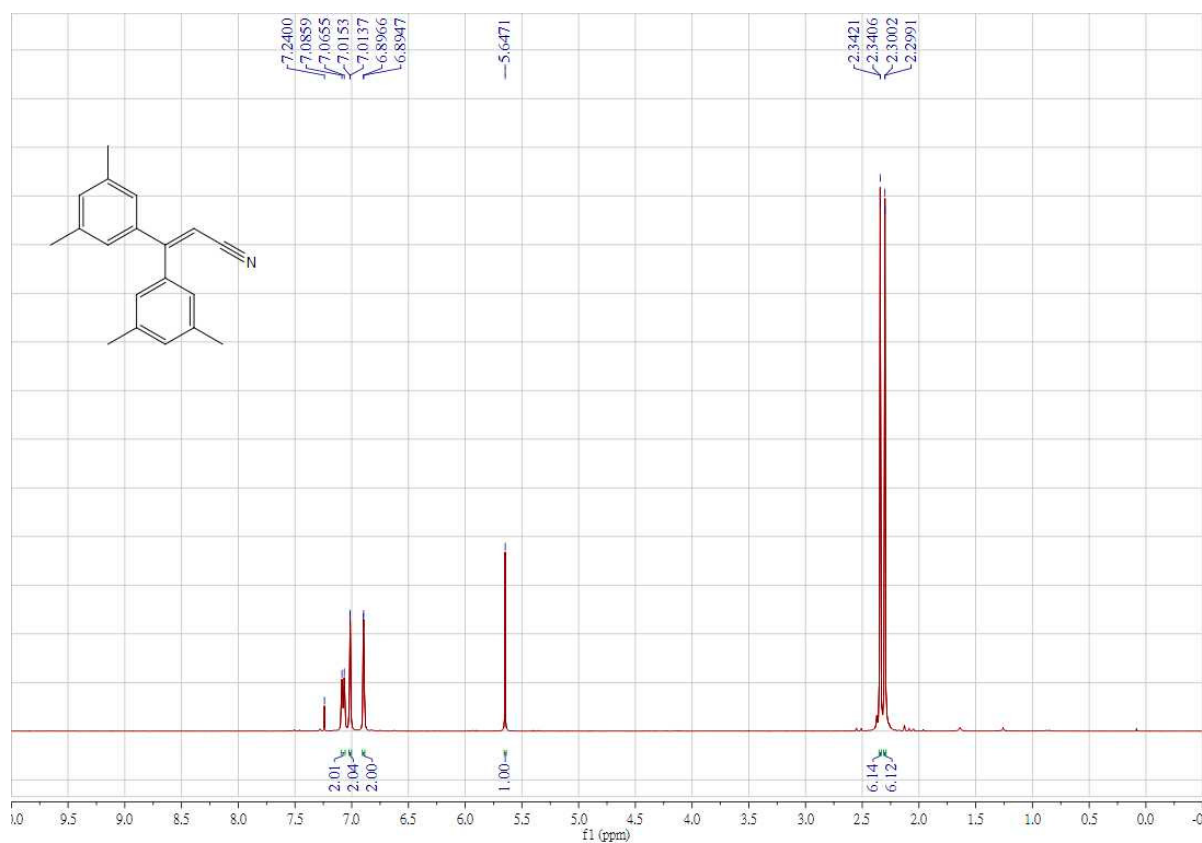
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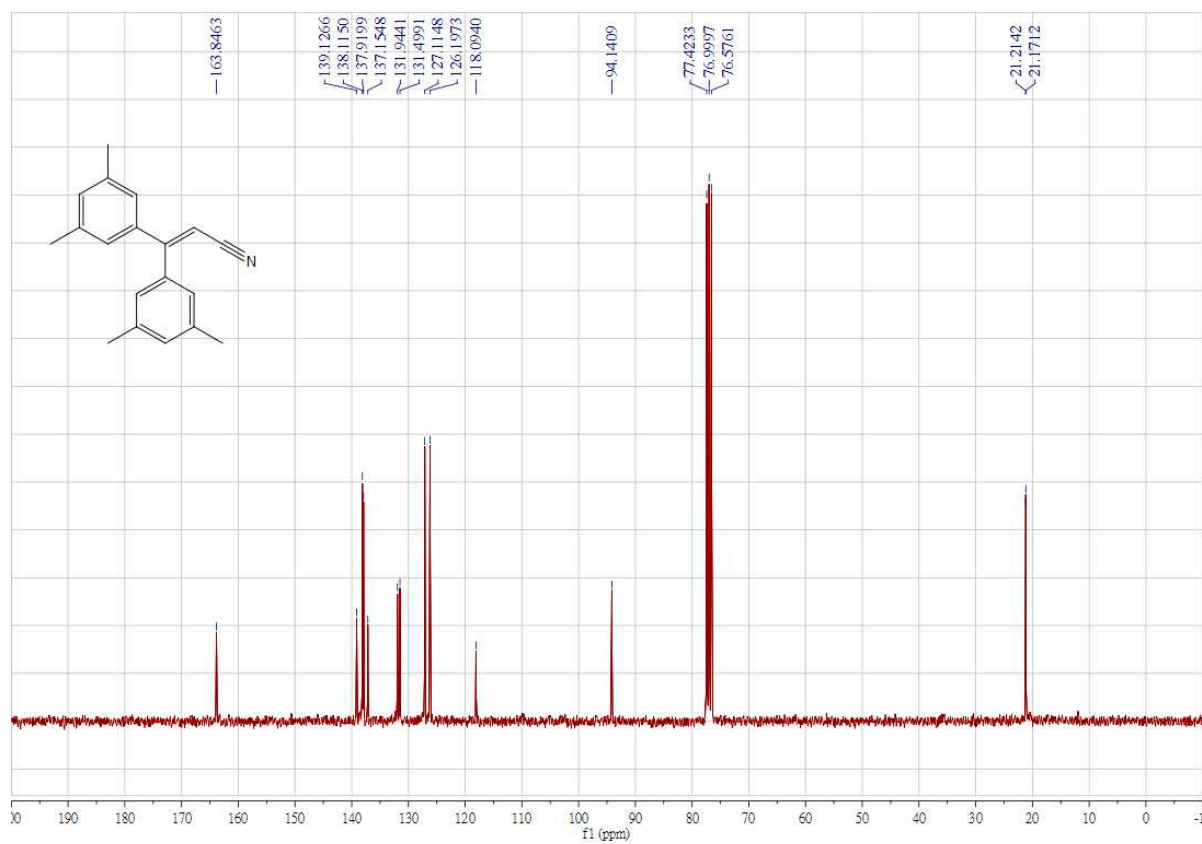
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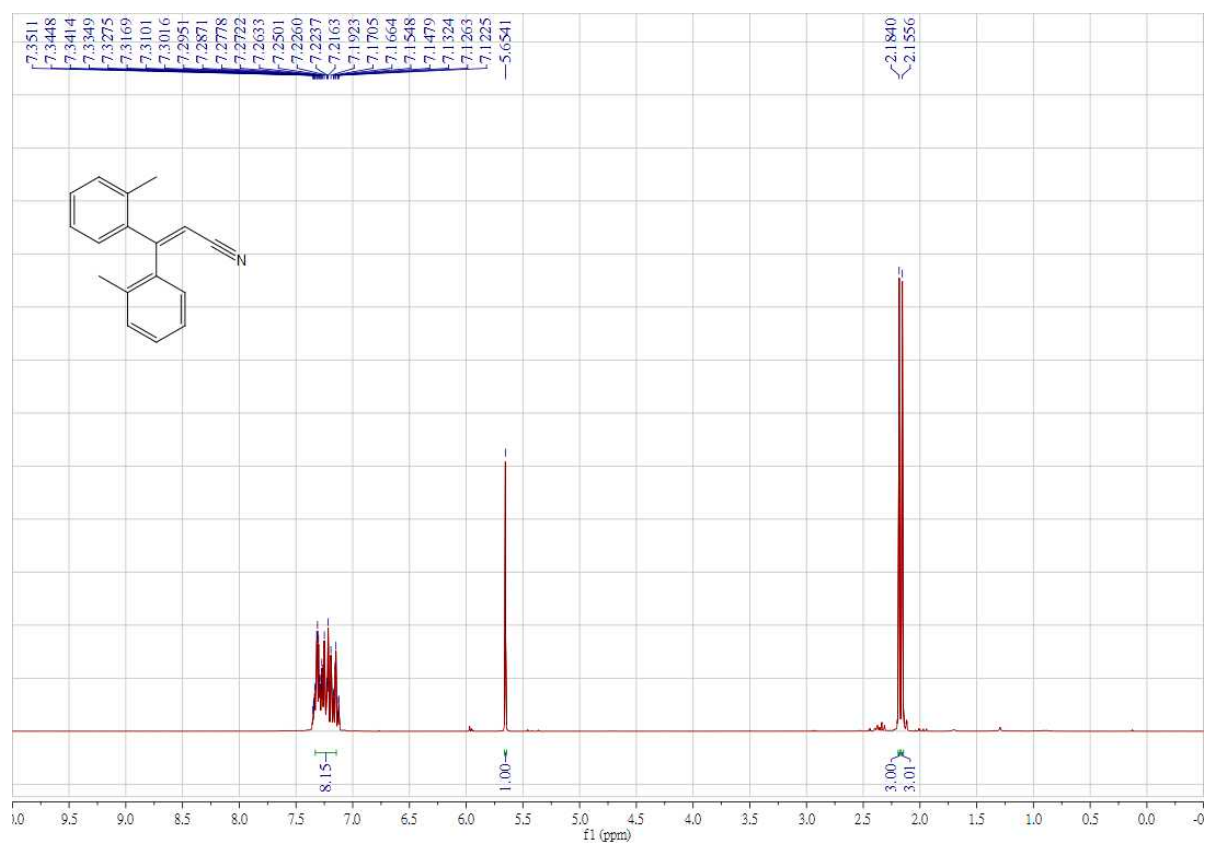
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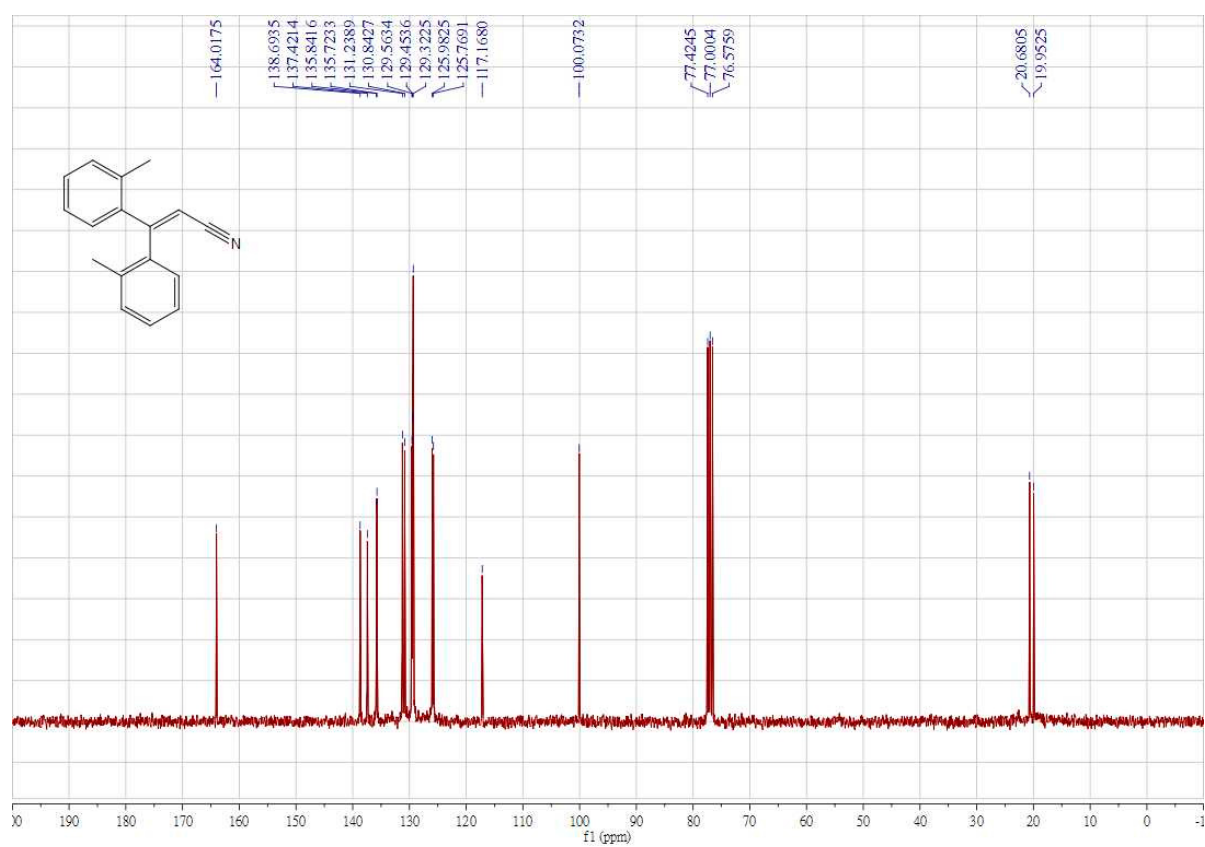
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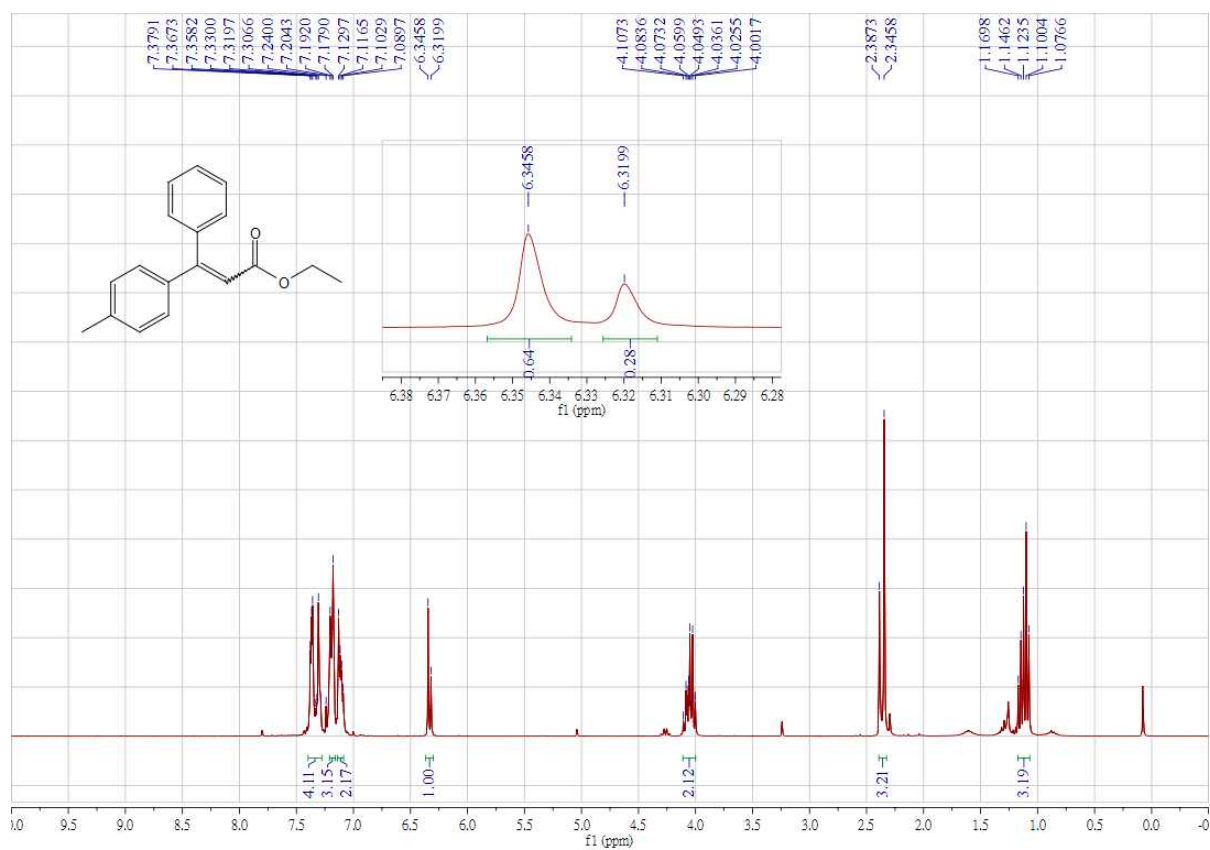
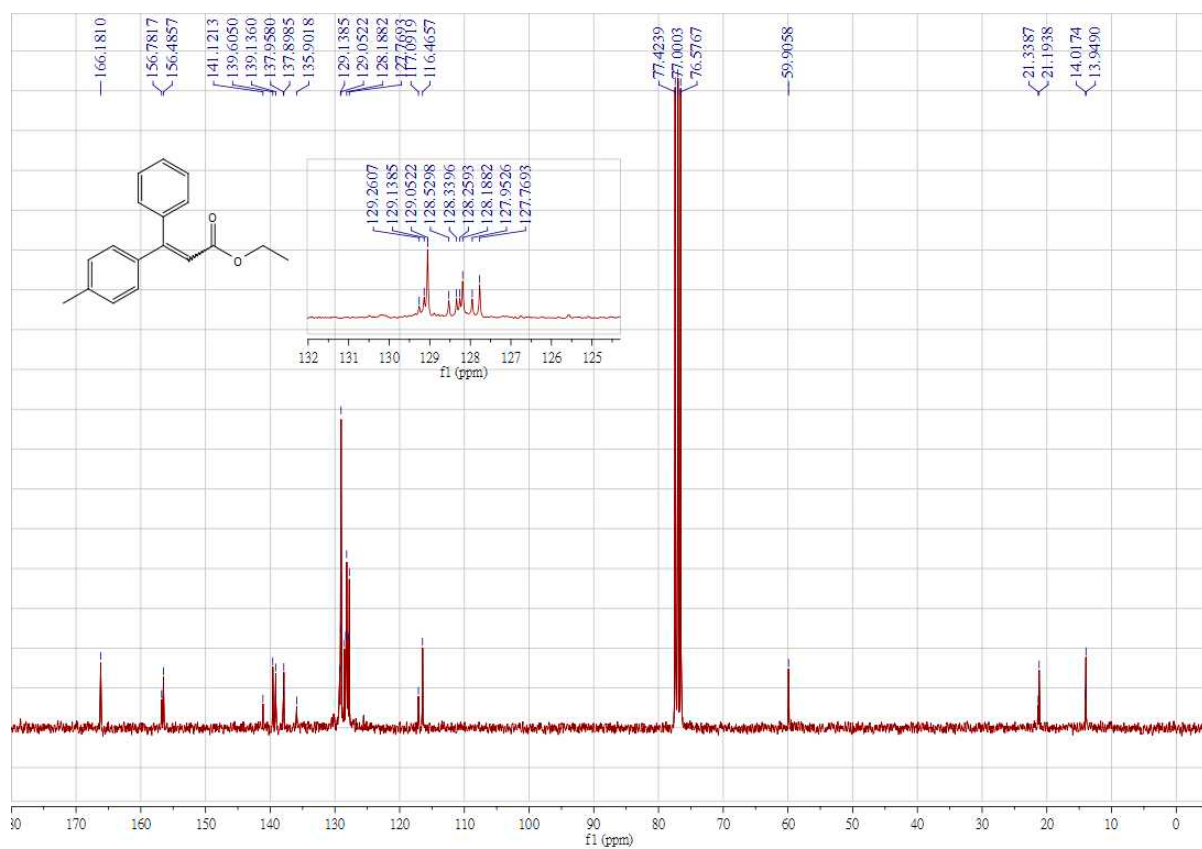
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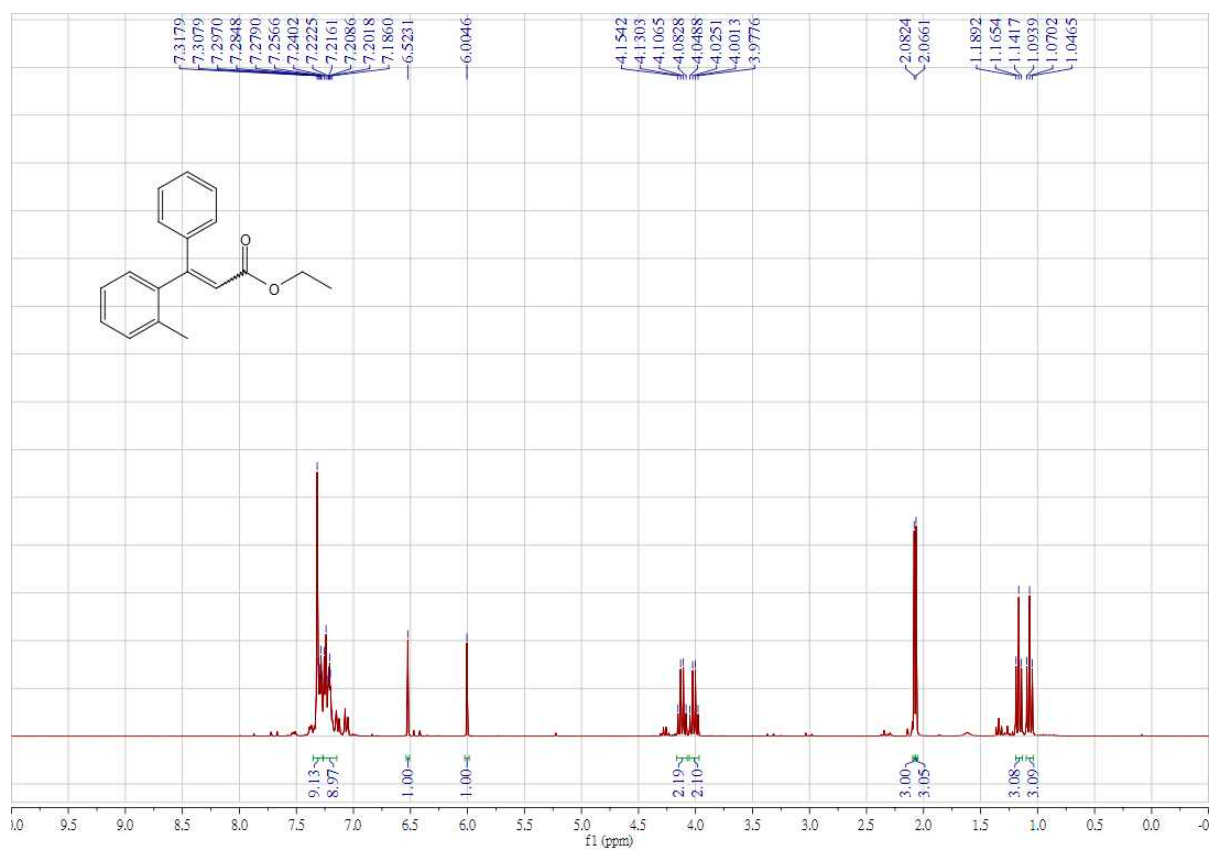


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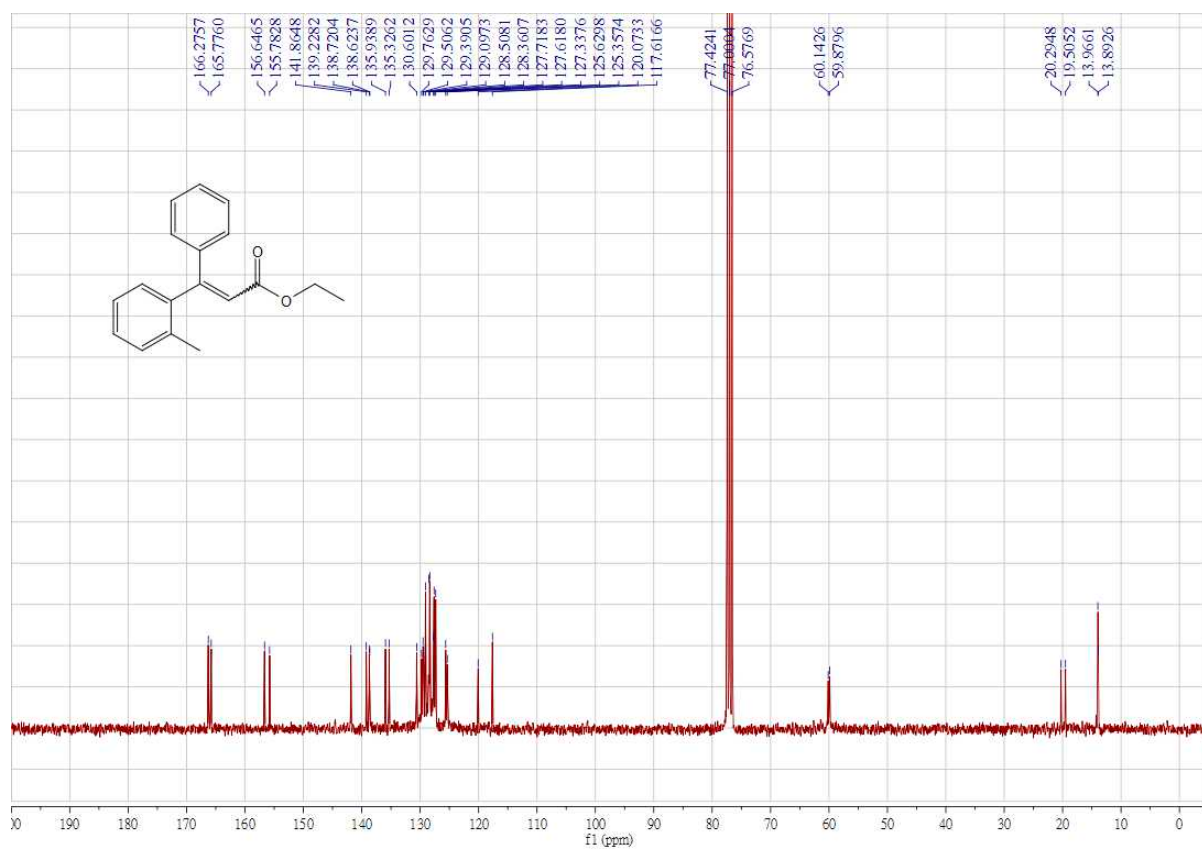


¹H NMR spectrum of compound **8ae**

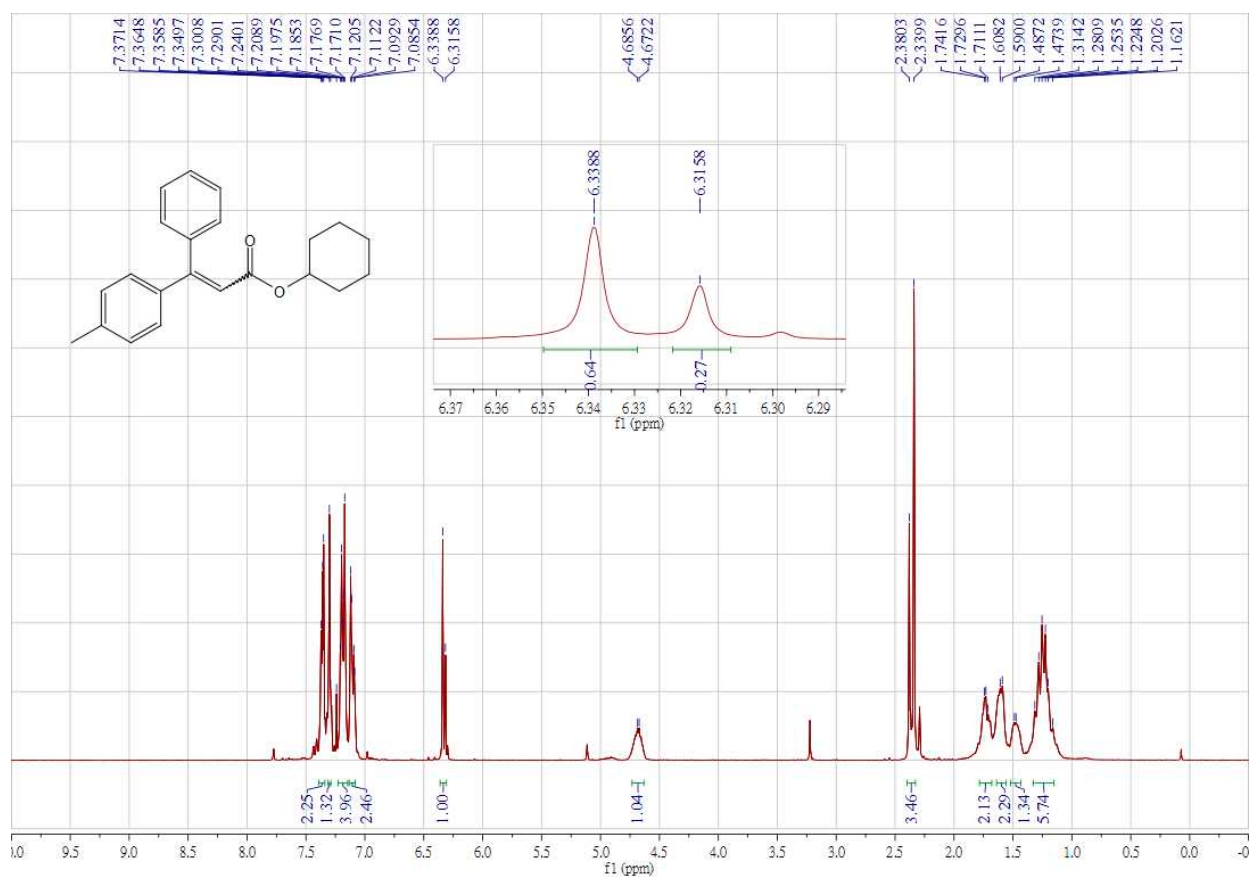
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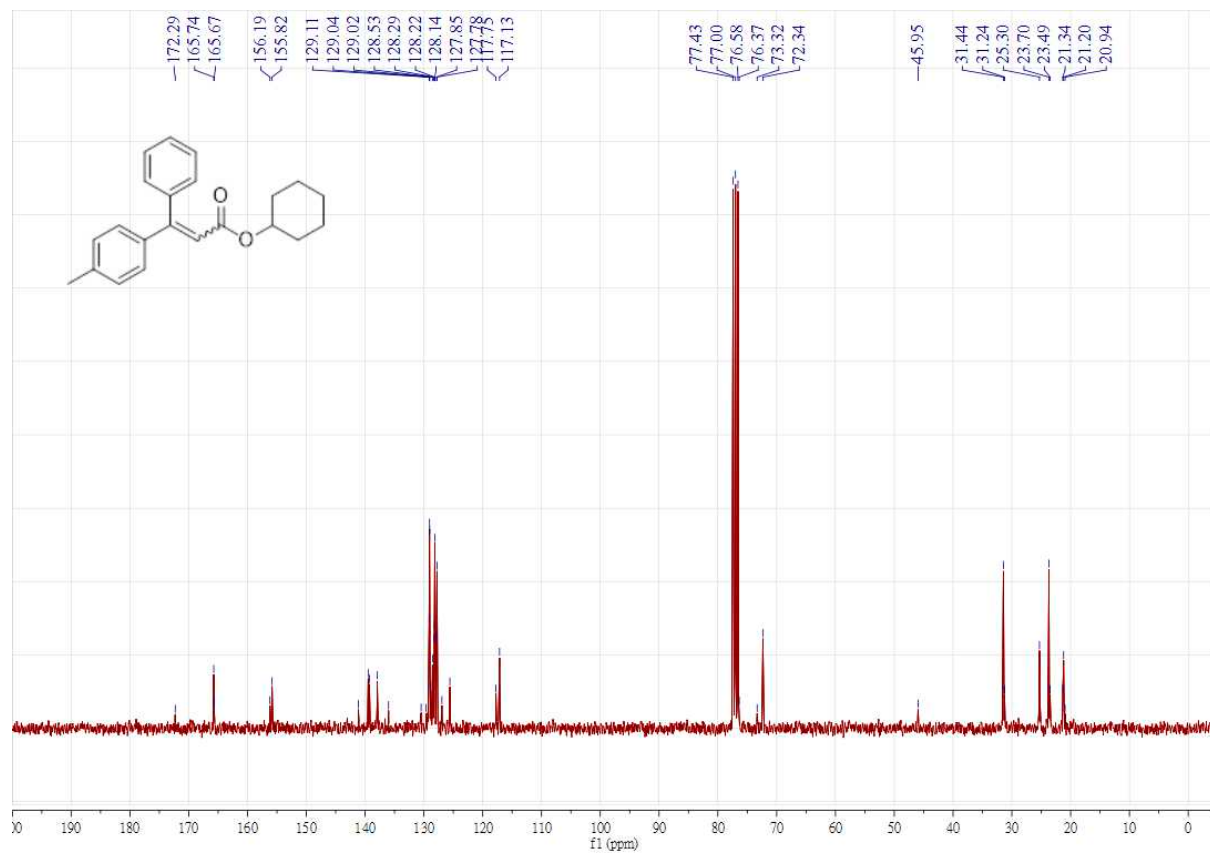
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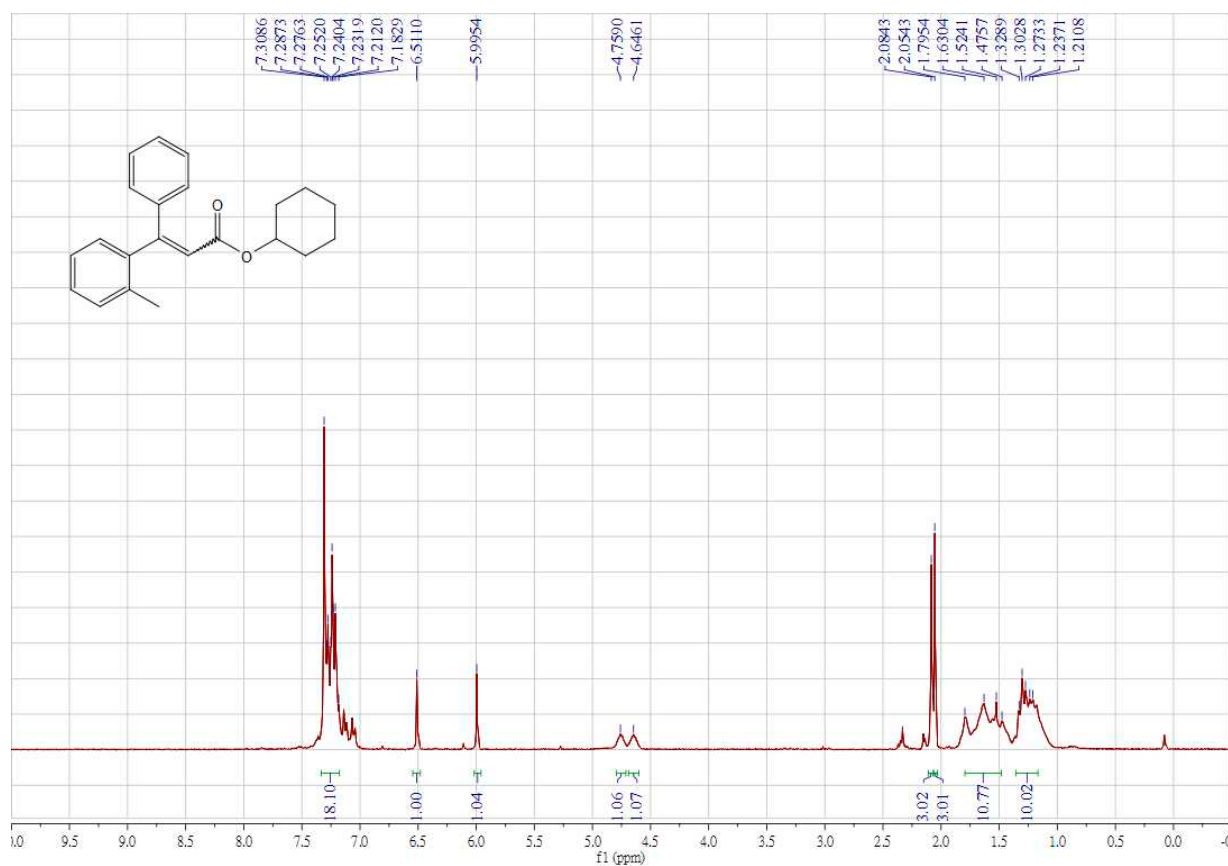
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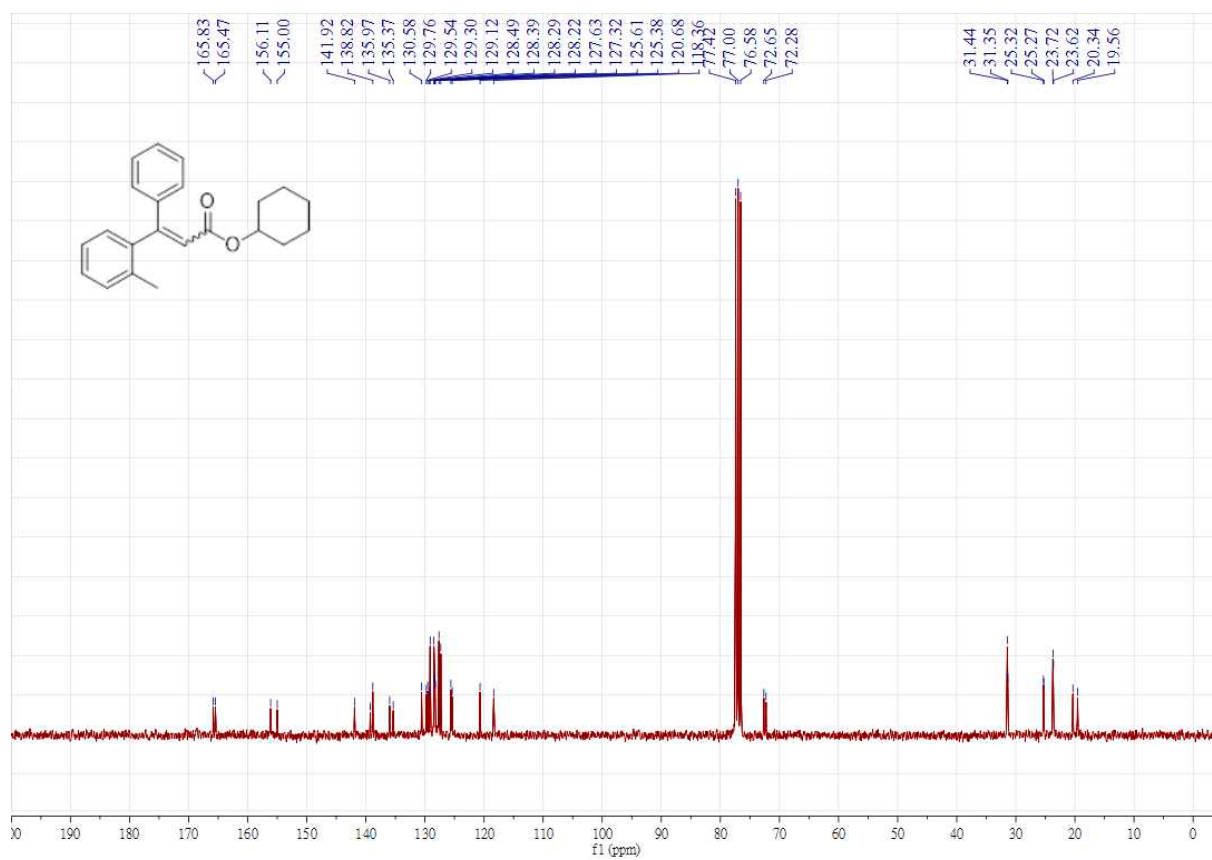
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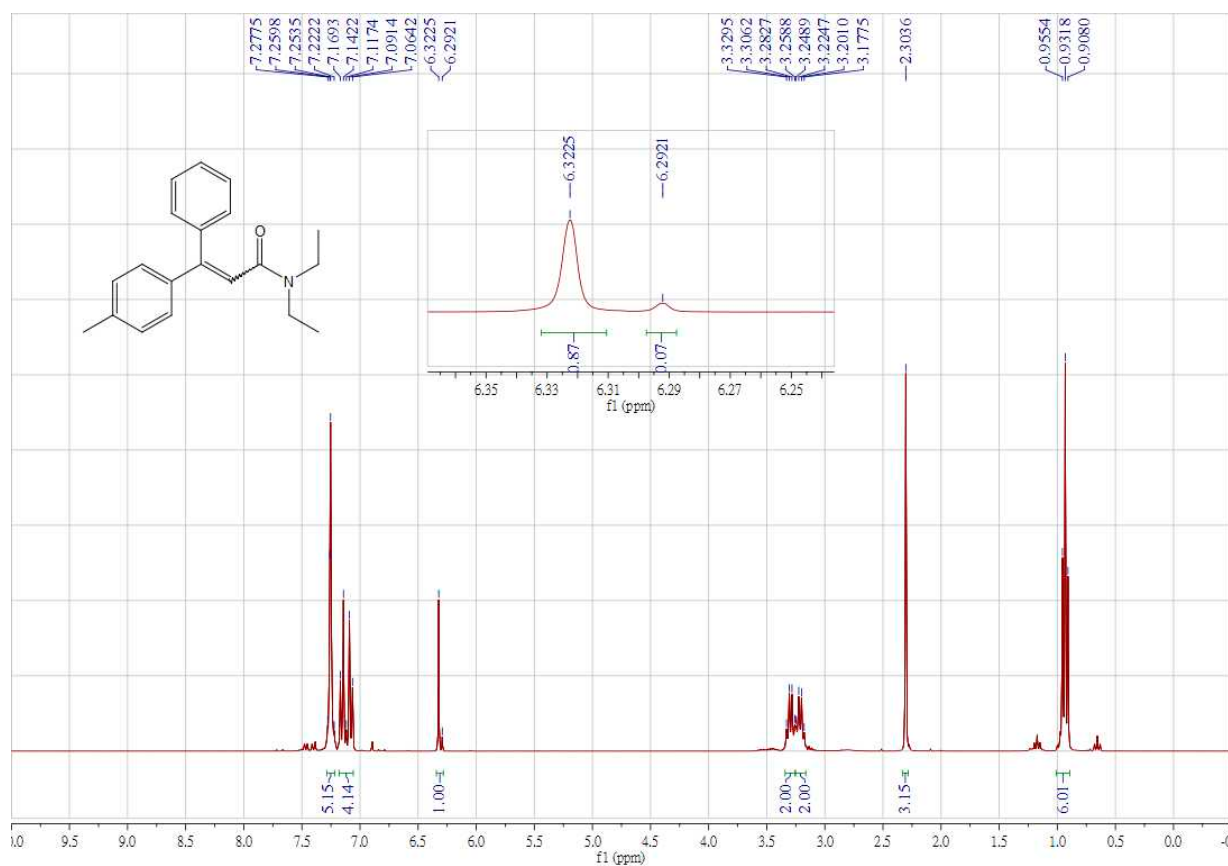
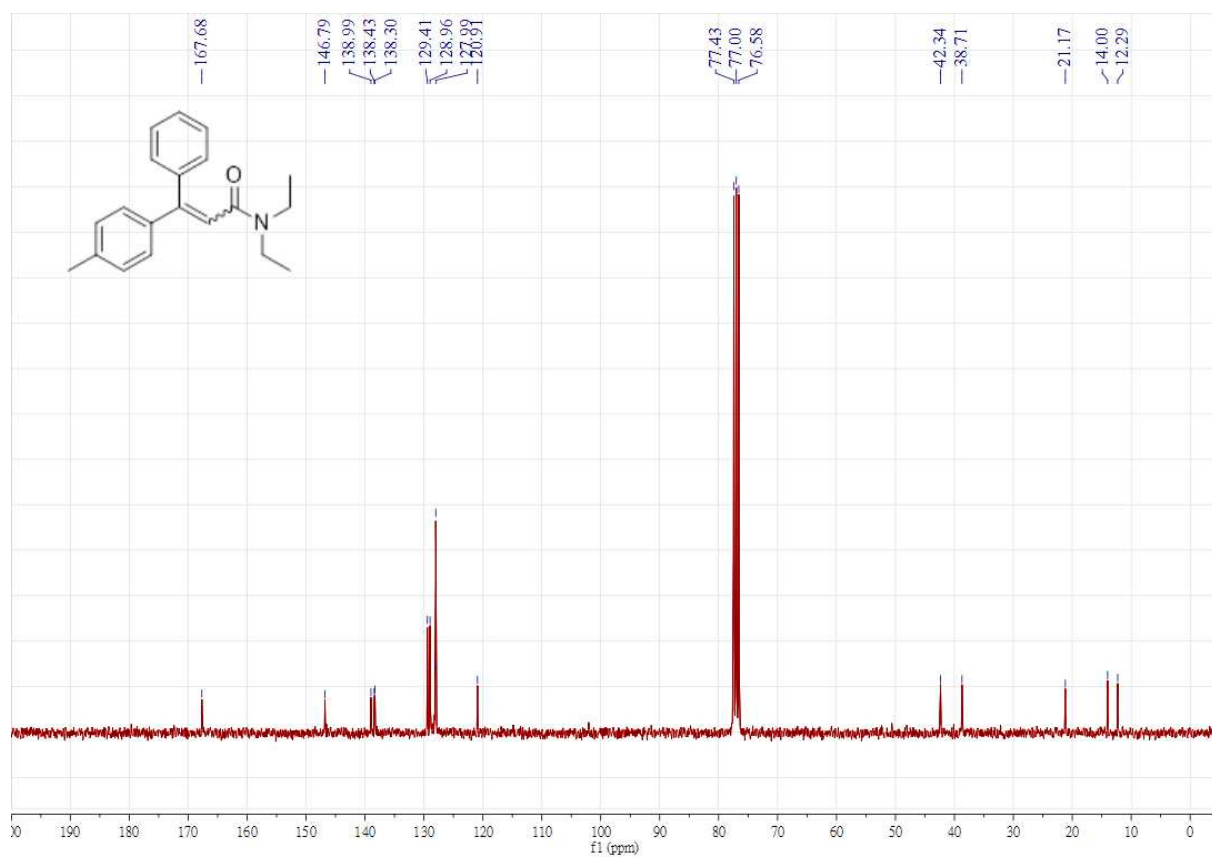
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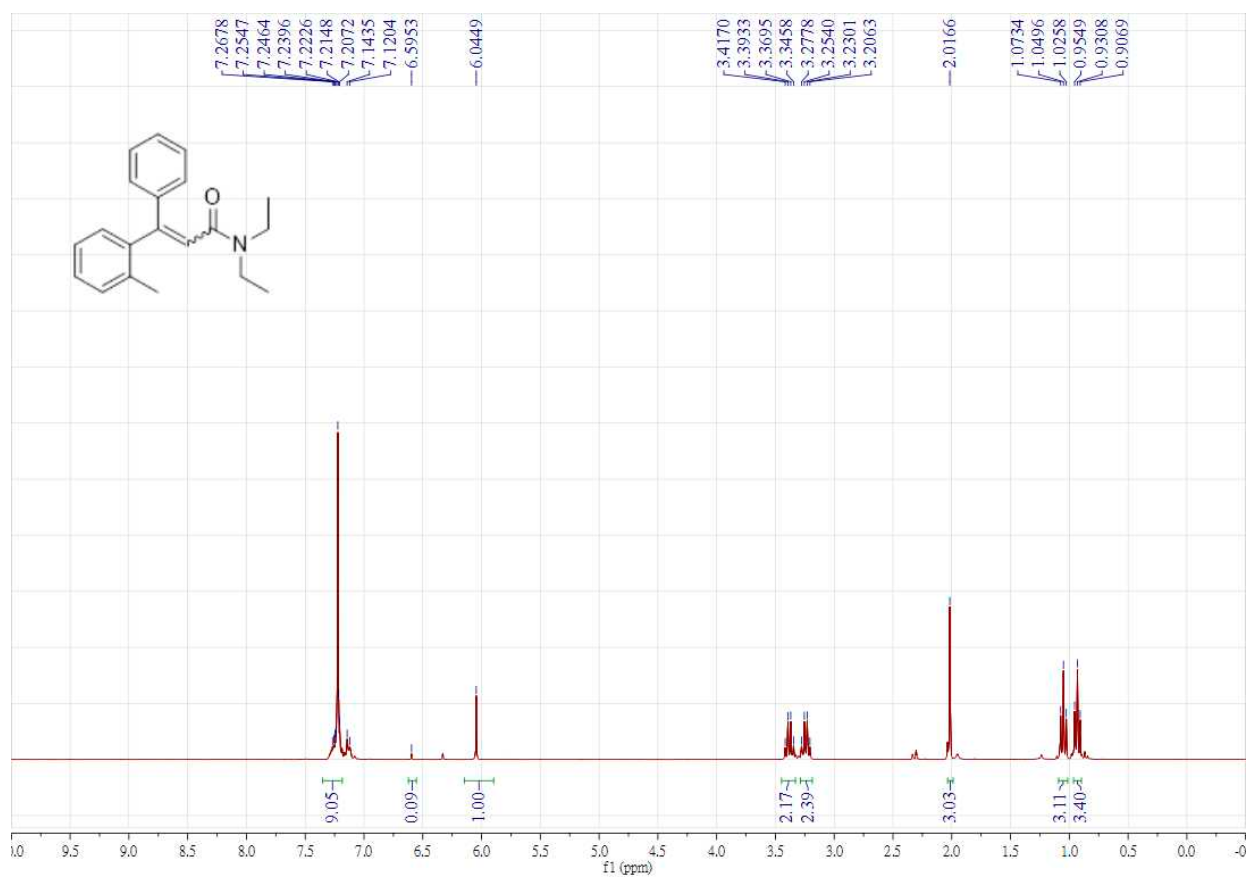


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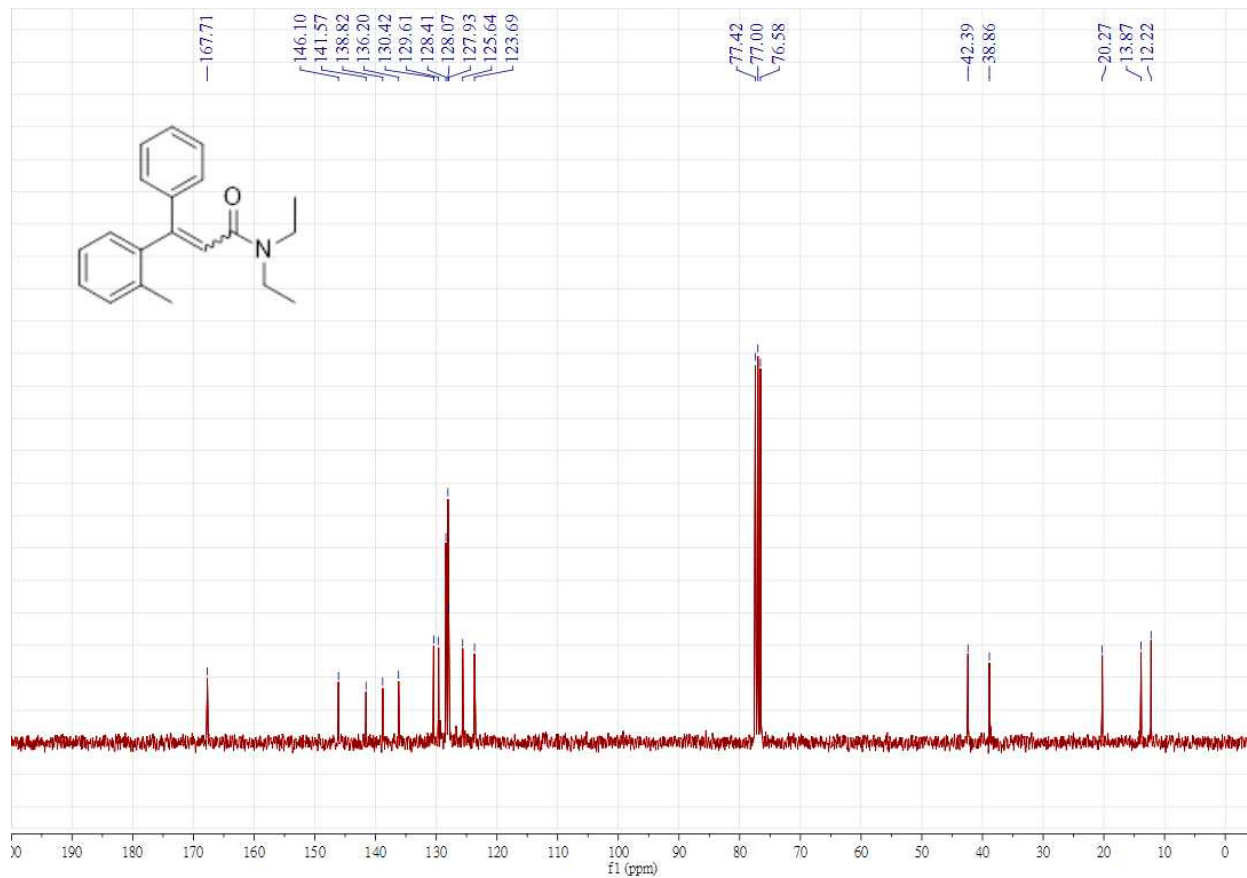


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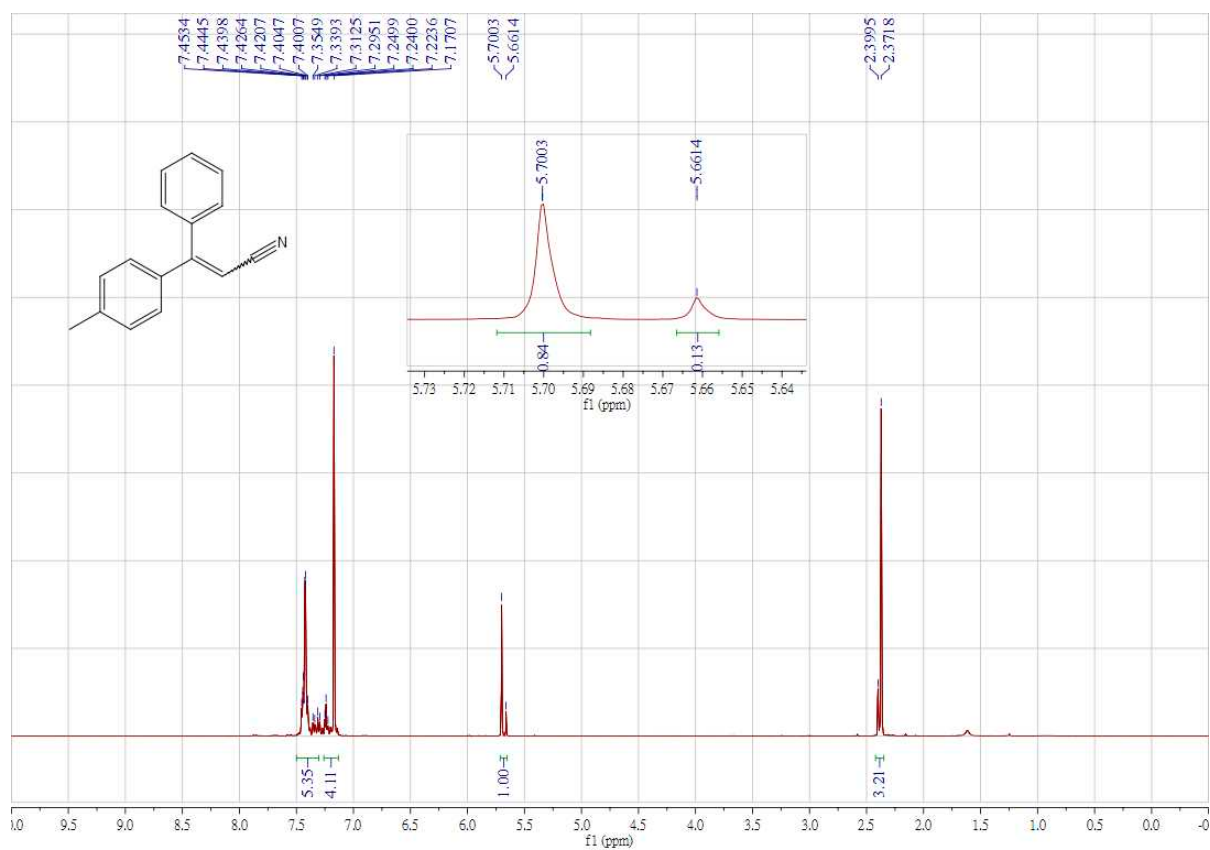
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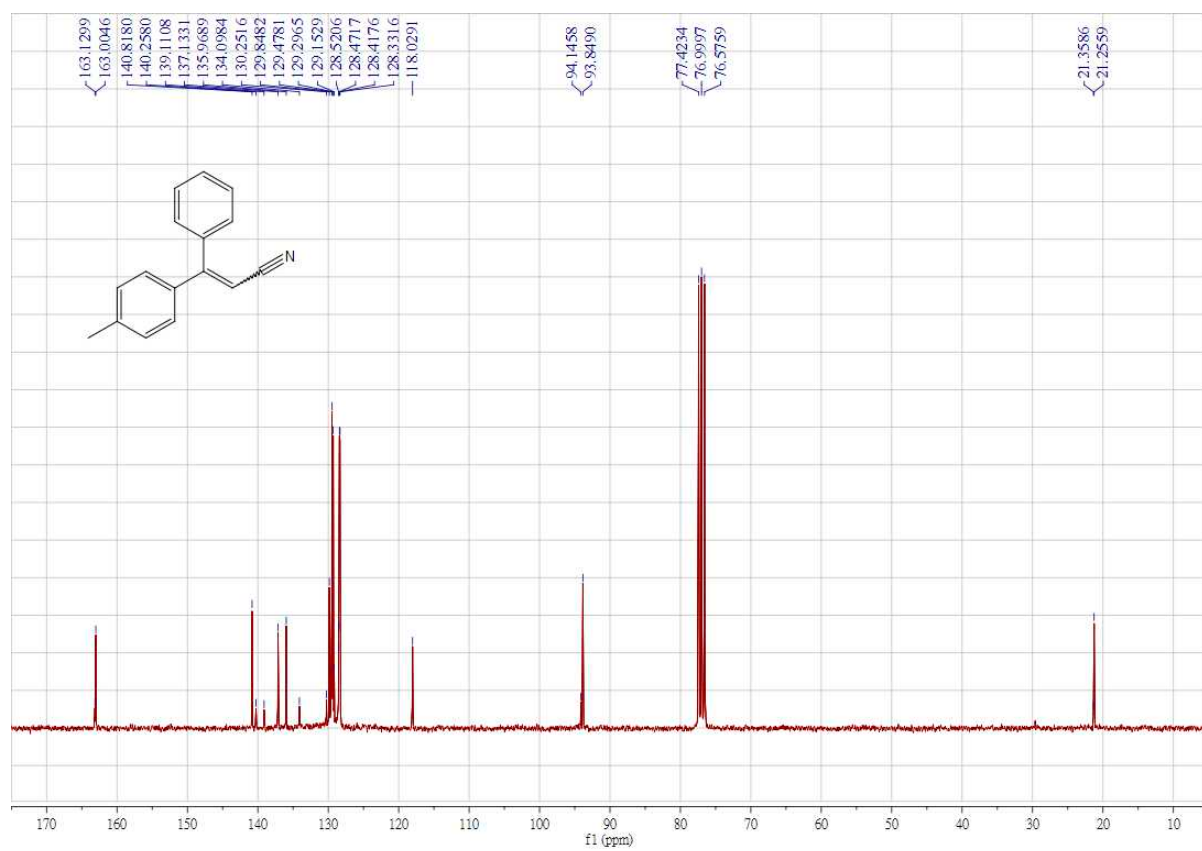
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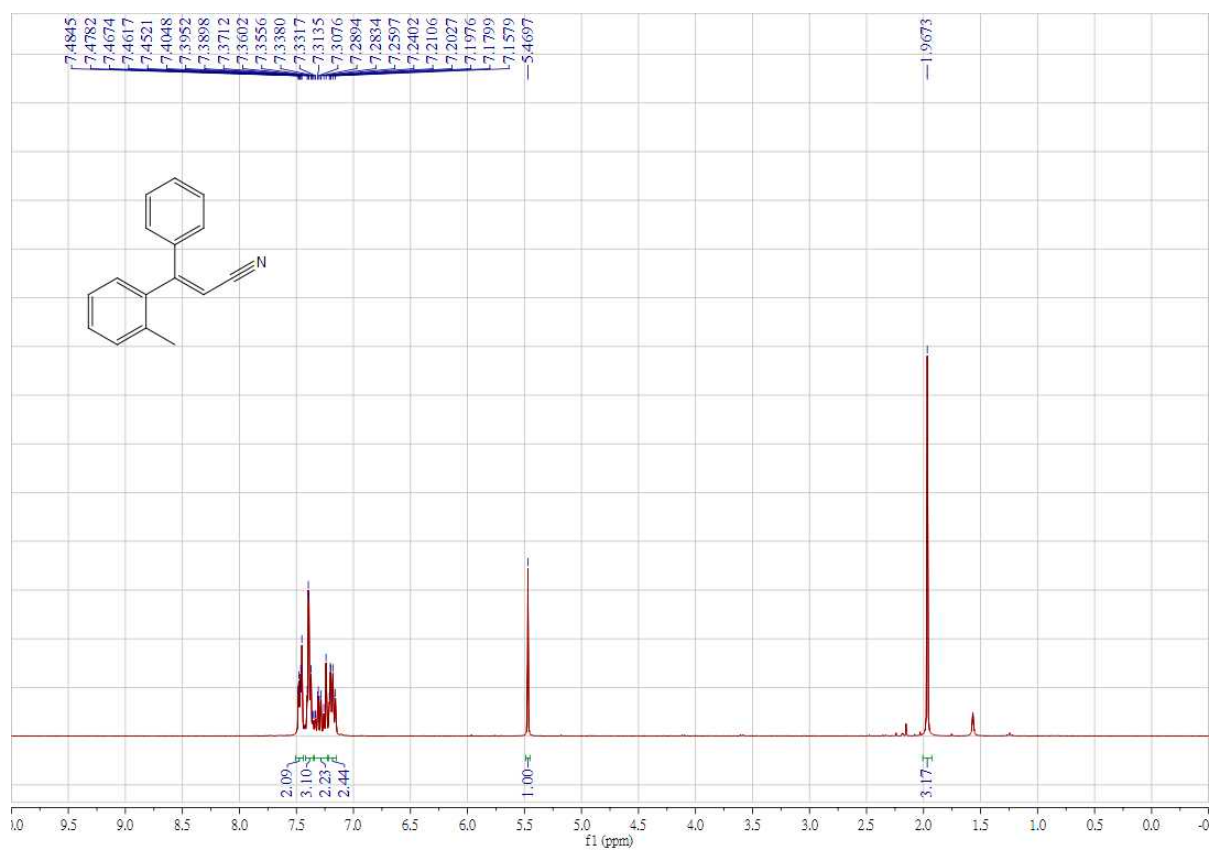
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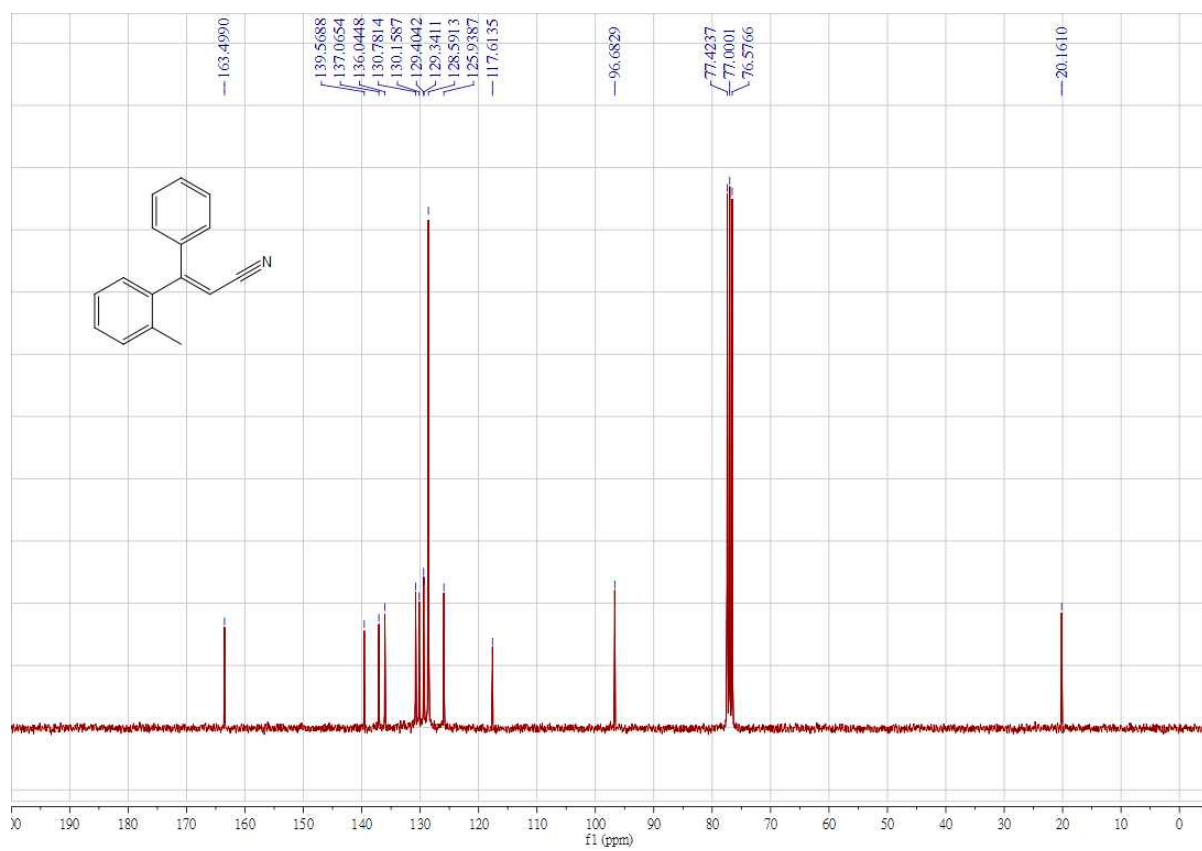
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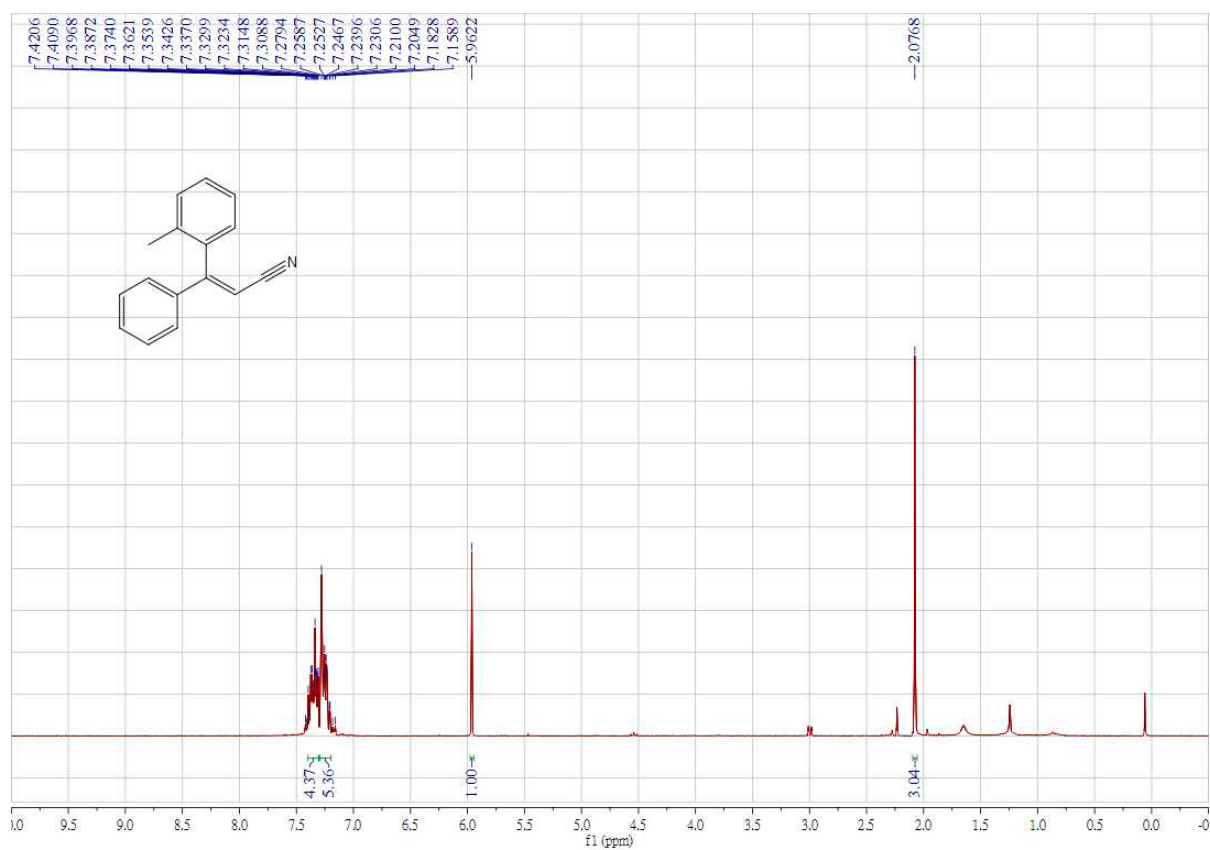
¹H NMR spectrum of compound **8dh** (*E*)



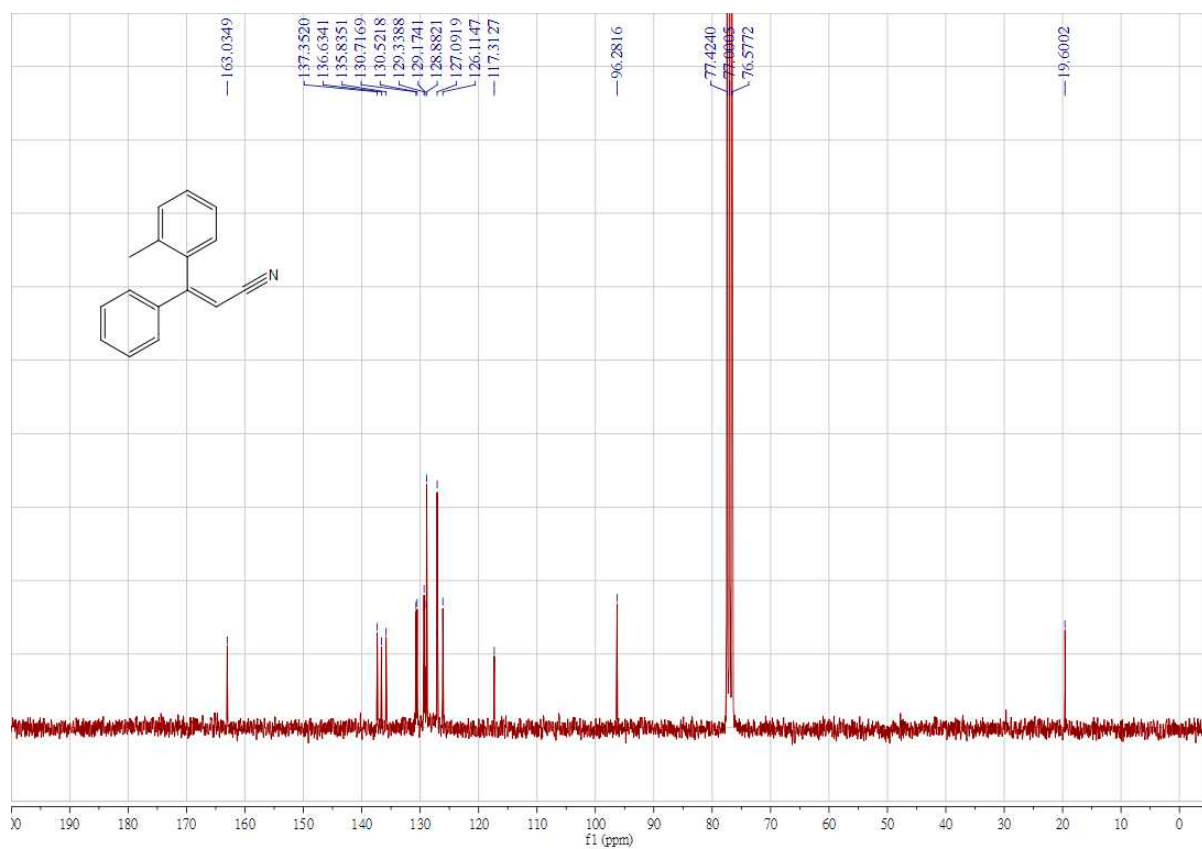
¹³C NMR spectrum of compound **8dh (E)**



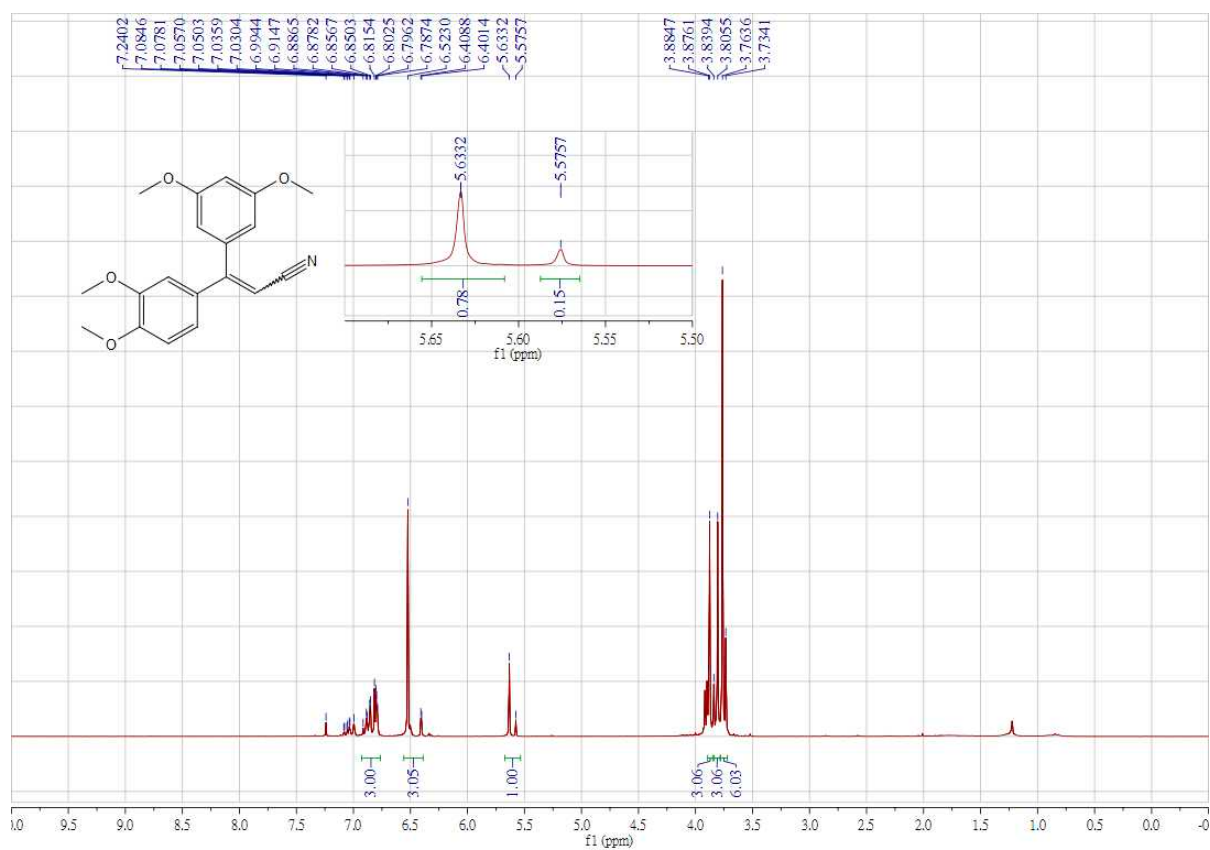
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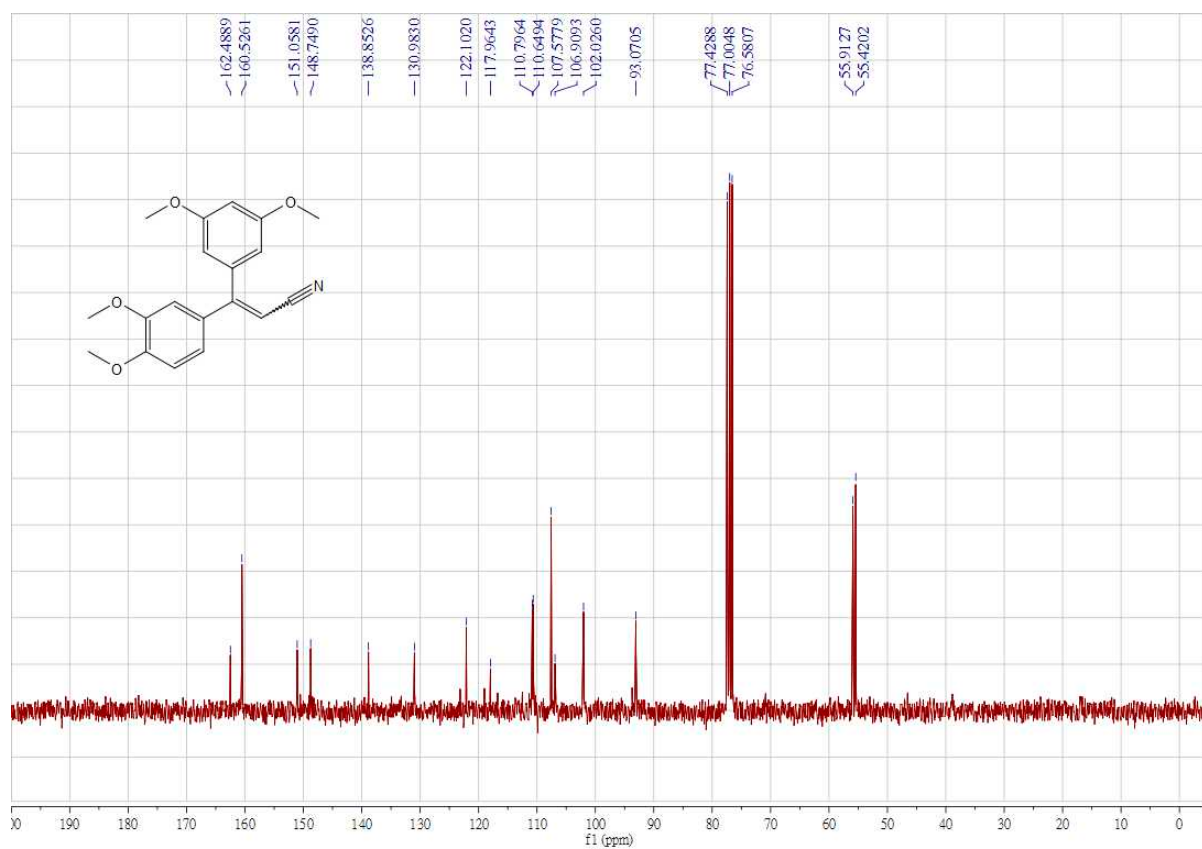
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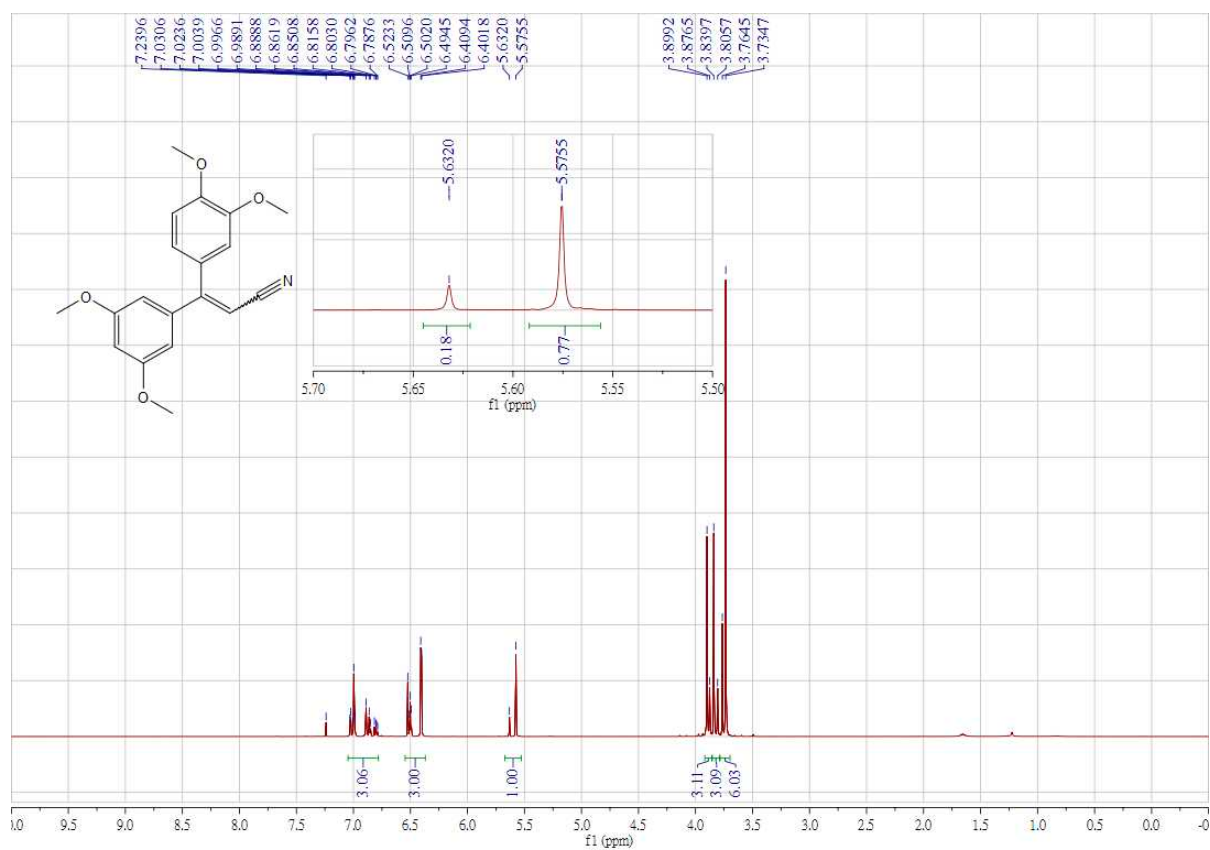
¹H NMR spectrum of compound **8ei**



¹³C NMR spectrum of compound 8ei



¹H NMR spectrum of compound 8ei



¹³C NMR spectrum of compound 8ei

