

Microwave-promoted Heck coupling using ultra-low metal catalyst concentrations

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Supporting Information

General experimental S2

^1H and ^{13}C NMR for the compounds prepared S3-S27

General experimental

Unless noted otherwise, all materials were obtained from commercial suppliers and used without further purification. The palladium stock solution used was elemental Pd in 20 % HCl. Concentration 1000 ug/ml. J.T. Baker cat. no. 5772-04. All reactions were carried out in air. Reactions were conducted using a focused microwave unit. The machine consists of a continuous focused microwave power delivery system with operator selectable power output from 0-300 W. Reactions were performed in glass vessels (capacity 10 mL) sealed with a septum. The pressure is controlled by a load cell connected to the vessel either via a 14-gauge needle which penetrates just below the septum surface or directly from the vessel. The temperature of the contents of the vessel was monitored using a calibrated infrared temperature control mounted under the reaction vessel. ^1H - and ^{13}C -NMR spectra were recorded at 293 K on a 400 MHz spectrometer. All yields reported are determined by integration of NMR signals relative to an internal standard.

Spectroscopic data

4-Methoxy-*trans*-stilbene (Compound 1): ^1H NMR (CDCl_3) δ 7.46-7.53 (m, 4H), 7.37 (t, J = 7.6 Hz, 2H), 7.26 (t, J = 7.3 Hz, 1H), 7.10 (d, J = 16.3 Hz, 1H), 7.00 (d, J = 16.3 Hz, 1H), 6.93 (dd, J = 8.8 Hz, J = 1.9 Hz, 2H), 3.85 (s, 3H); ^{13}C NMR (CDCl_3) δ 159.4, 137.7, 130.2, 128.7, 128.3, 127.8, 127.2, 126.7, 126.3, 114.2, 55.3.

4-Acetyl-*trans*-stilbene (Compound 2): ^1H NMR (CDCl_3) δ 7.95 (d, J = 8.4 Hz, 2H), 7.57 (d, J = 8.3 Hz, 2H), 7.53 (d, J = 7.4 Hz, 2H), 7.38 (t, J = 7.5 Hz, 2H), 7.30 (t, J = 7.3 Hz, 1H), 7.22 (d, J = 16.3 Hz, 1H), 7.12 (d, J = 16.3 Hz, 1H), 2.59 (s, 3H); ^{13}C NMR (CDCl_3) δ 197.4, 142.0, 136.7, 136.0, 131.5, 128.9, 128.8, 128.3, 127.5, 126.9, 126.5, 26.6.

Trans-stilbene (Compound 3): ^1H NMR (CDCl_3) δ 7.60 (dd, J = 8.3 Hz, J = 1.2 Hz, 4H), 7.44 (tt, J = 7.5 Hz, J = 1.6 Hz, 4H), 7.34 (tt, J = 7.3 Hz, J = 1.5 Hz, 2H), 7.20 (s, 2H); ^{13}C NMR (CDCl_3) δ 137.4, 128.8, 128.8, 127.7, 126.6.

4-Methyl-*trans*-stilbene (Compound 4): ^1H NMR (CDCl_3) δ 7.52 (d, J = 7.3 Hz, 2H), 7.43 (d, J = 8.1 Hz, 2H), 7.37 (t, J = 7.6 Hz, 2H), 7.26 (t, J = 7.3 Hz, 1H), 7.19 (d, J = 8.0 Hz, 2H), 7.09 (s, 2H), 2.38 (s, 3H); ^{13}C NMR (CDCl_3) δ 137.6, 137.5, 134.6, 129.4, 128.7, 127.7, 127.4, 126.5, 126.4, 21.3.

2-Methyl-*trans*-stilbene (Compound 5): ^1H NMR (CDCl_3) δ 7.65 (d, $J = 7.0$ Hz, 1H), 7.58 (d, $J = 7.6$ Hz, 2H), 7.37-7.43 (m, 3H), 7.23-7.33 (m, 4H), 7.05 (d, $J = 16.2$ Hz, 1H), 2.48 (s, 3H); ^{13}C NMR (CDCl_3) δ 137.8, 136.5, 135.9, 130.5, 130.1, 128.7, 127.6, 127.6, 126.6, 126.3, 125.4, 20.0.

4-Chloro-*trans*-stilbene (Compound 6): ^1H NMR (CDCl_3) δ 7.51 (d, $J = 7.3$ Hz, 2H), 7.44 (d, $J = 8.5$ Hz, 2H), 7.26-7.39 (m, 5H), 7.07 (s, 2H); ^{13}C NMR (CDCl_3) δ 137.0, 135.9, 133.2, 129.3, 128.8, 128.7, 127.9, 127.7, 127.4, 126.6.

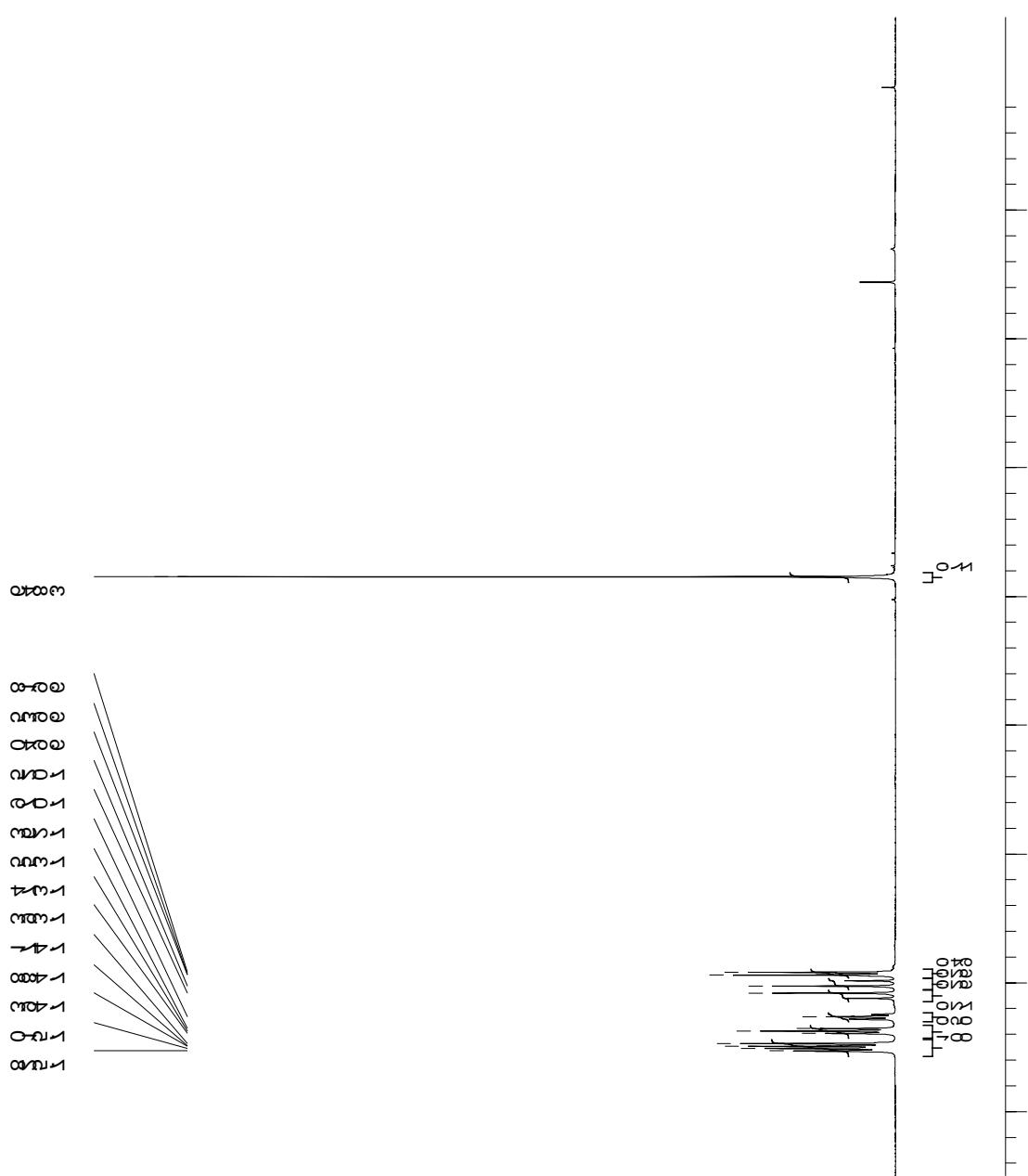
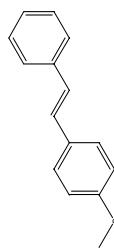
4-Fluoro-*trans*-stilbene (Compound 7): ^1H NMR (CDCl_3) δ 7.47-7.52 (m, 4H), 7.37 (t, $J = 7.6$ Hz, 2H), 7.25-7.32 (m, 1H), 7.00-7.10 (m, 4H); ^{13}C NMR (CDCl_3) δ 162.4 (d), 137.2, 133.5 (d), 128.7, 128.5, 128.0 (d), 127.7, 127.5, 126.5, 115.6 (d).

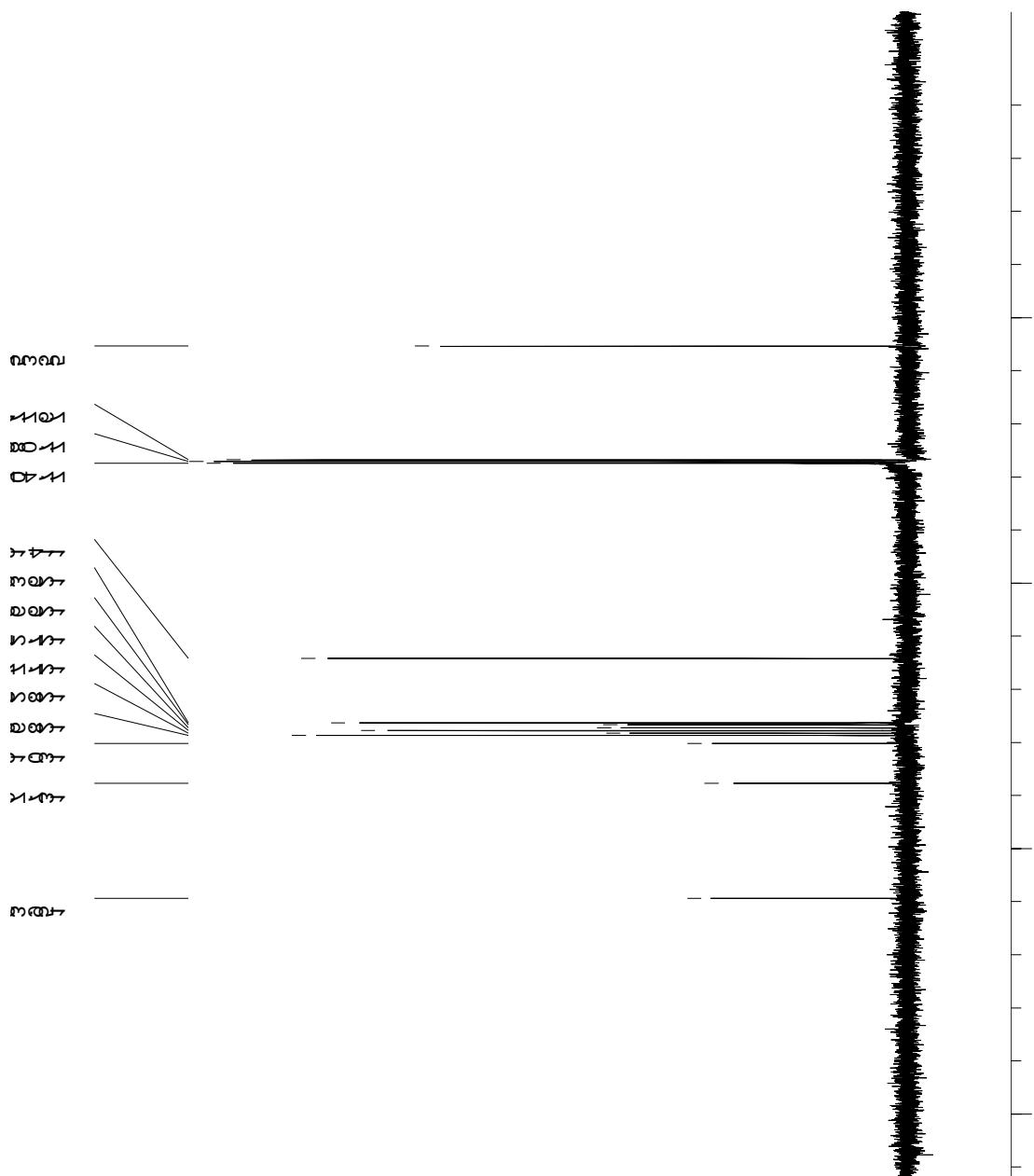
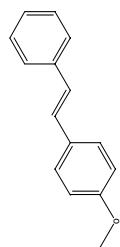
2-Methoxy-*trans*-stilbene (Compound 8): ^1H NMR (CDCl_3) δ 7.66 (dd, $J = 7.7$ Hz, $J = 1.5$ Hz), 7.55-7.62 (m, 3H), 7.26-7.43 (m, 5H), 7.19 (d, $J = 16.8$ Hz, 1H), 7.03 (t, $J = 7.6$ Hz), 6.95 (d, $J = 8.2$ Hz, 1H), 3.93 (s, 3H); ^{13}C NMR (CDCl_3) δ 157.0, 138.0, 129.1, 128.7, 128.6, 127.4, 126.6, 126.5, 126.4, 123.6, 120.8, 111.0, 55.6.

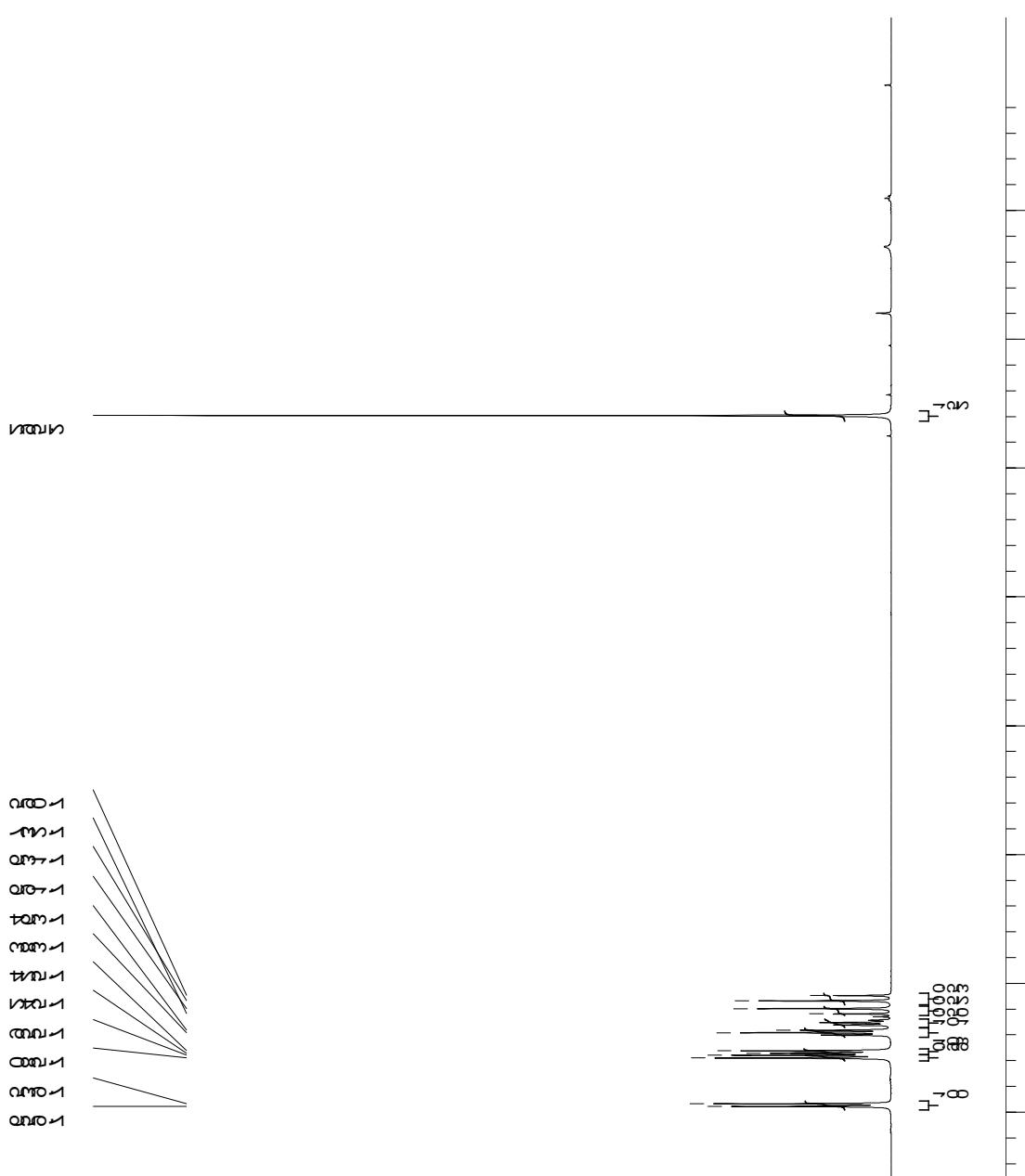
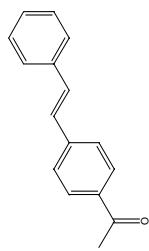
4-Methoxy-*trans*-cinnamic acid (Compound 9): ^1H NMR (CDCl_3) δ 7.75 (d, $J = 15.9$ Hz, 1H), 7.51 (d, $J = 8.7$ Hz, 2H), 6.92 (d, $J = 8.8$ Hz, 2H), 6.32 (d, $J = 15.9$ Hz, 1H), 3.85 (s, 3H); ^{13}C NMR (CDCl_3) δ 172.3, 161.8, 146.7, 130.1, 126.8, 114.6, 114.4, 55.4.

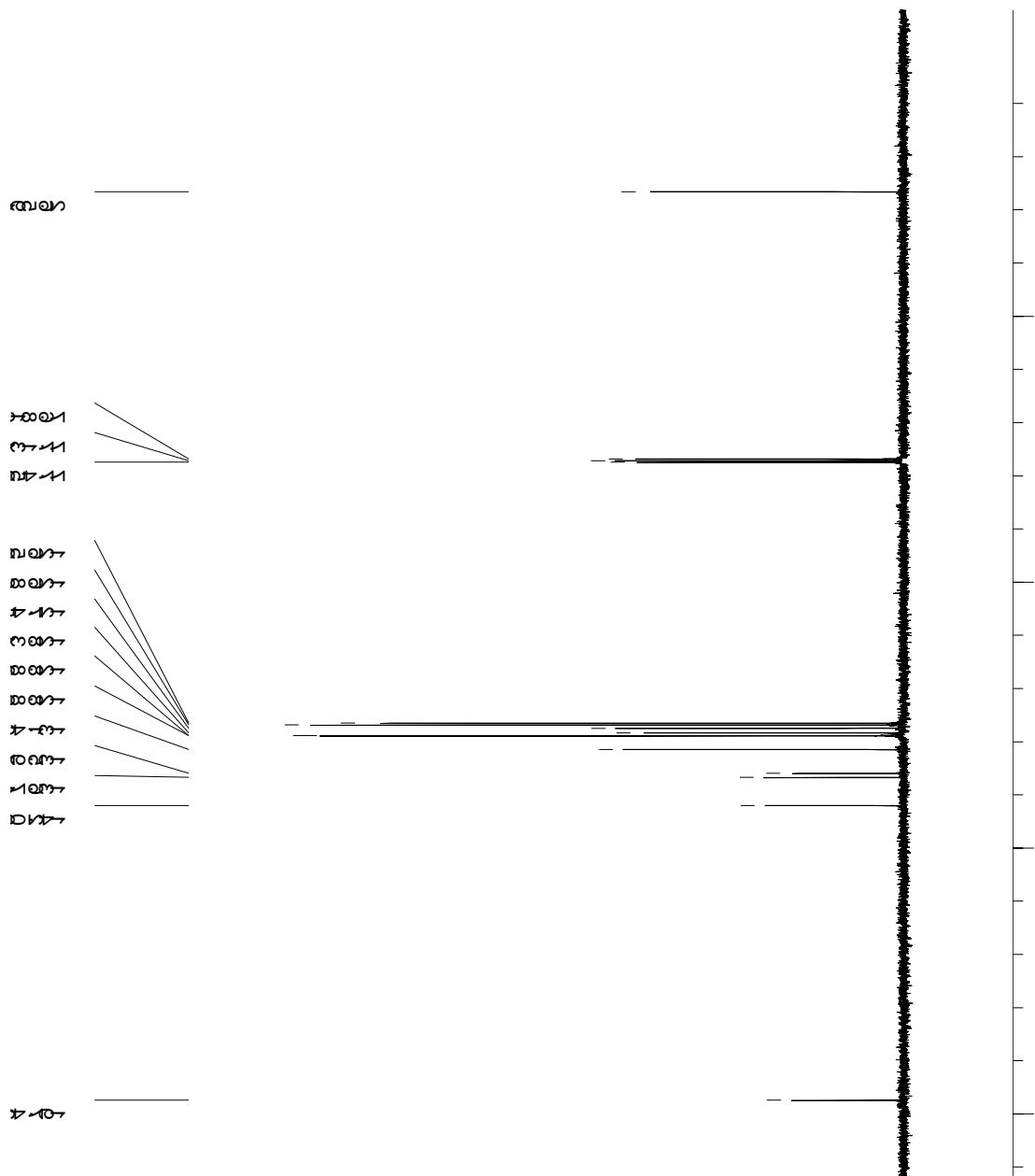
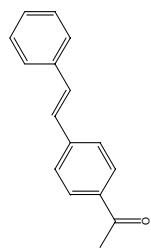
4-Acetyl-*trans*-cinnamic acid (Compound 10): ^1H NMR ($d_6\text{-DMSO}$) δ 7.93 (d, $J = 8.3$ Hz, 2H), 7.79 (d, $J = 8.3$ Hz, 2H), 7.61 (d, $J = 16.1$ Hz, 1H), 6.63 (d, $J = 16.1$ Hz, 1H), 2.55 (s, 3H); ^{13}C NMR ($d_6\text{-DMSO}$) δ 197.9, 167.7, 143.0, 139.0, 138.0, 129.1, 128.8, 122.2, 27.3.

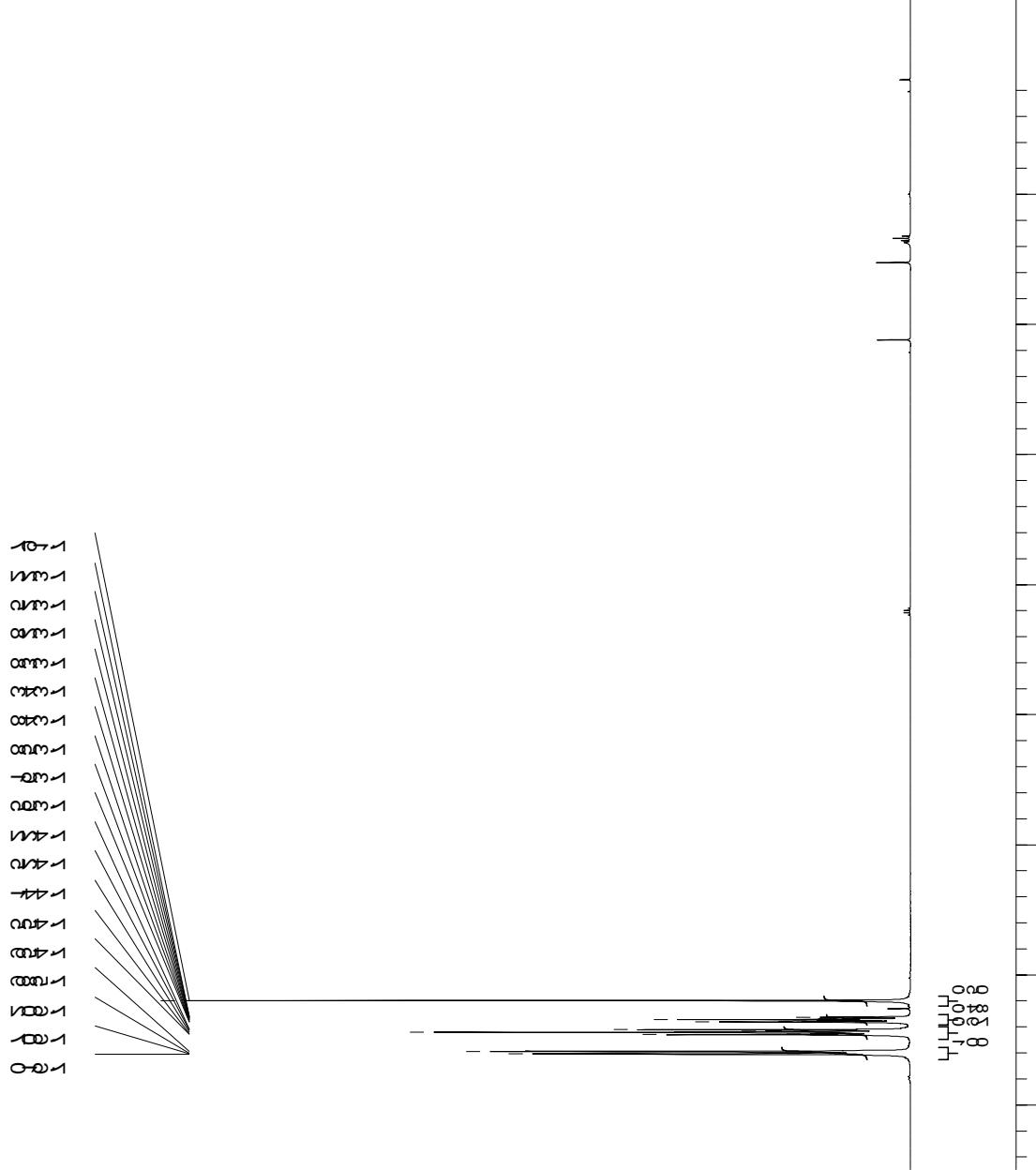
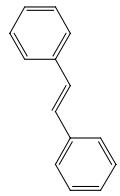
4-Methylcinnamic acid (Compound 11): E-isomer: ^1H NMR (CDCl_3) δ 7.78 (d, $J = 16.0$ Hz, 1H), 7.46 (d, $J = 8.1$ Hz, 2H), 7.22 (d, $J = 8.0$ Hz, 2H), 6.41 (d, $J = 15.9$ Hz, 1H), 2.39 (s, 3H); ^{13}C NMR (CDCl_3) δ 172.6, 147.1, 141.3, 131.3, 129.7, 128.4, 116.2, 21.5. Z-isomer: ^1H NMR (CDCl_3) δ 7.56 (d, $J = 8.1$ Hz, 2H), 7.35 (d, $J = 8.1$ Hz, 2H), 7.03 (d, $J = 12.3$ Hz, 1H), 5.93 (d, $J = 12.7$ Hz, 1H), 2.37 (s, 3H)

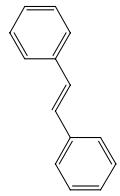






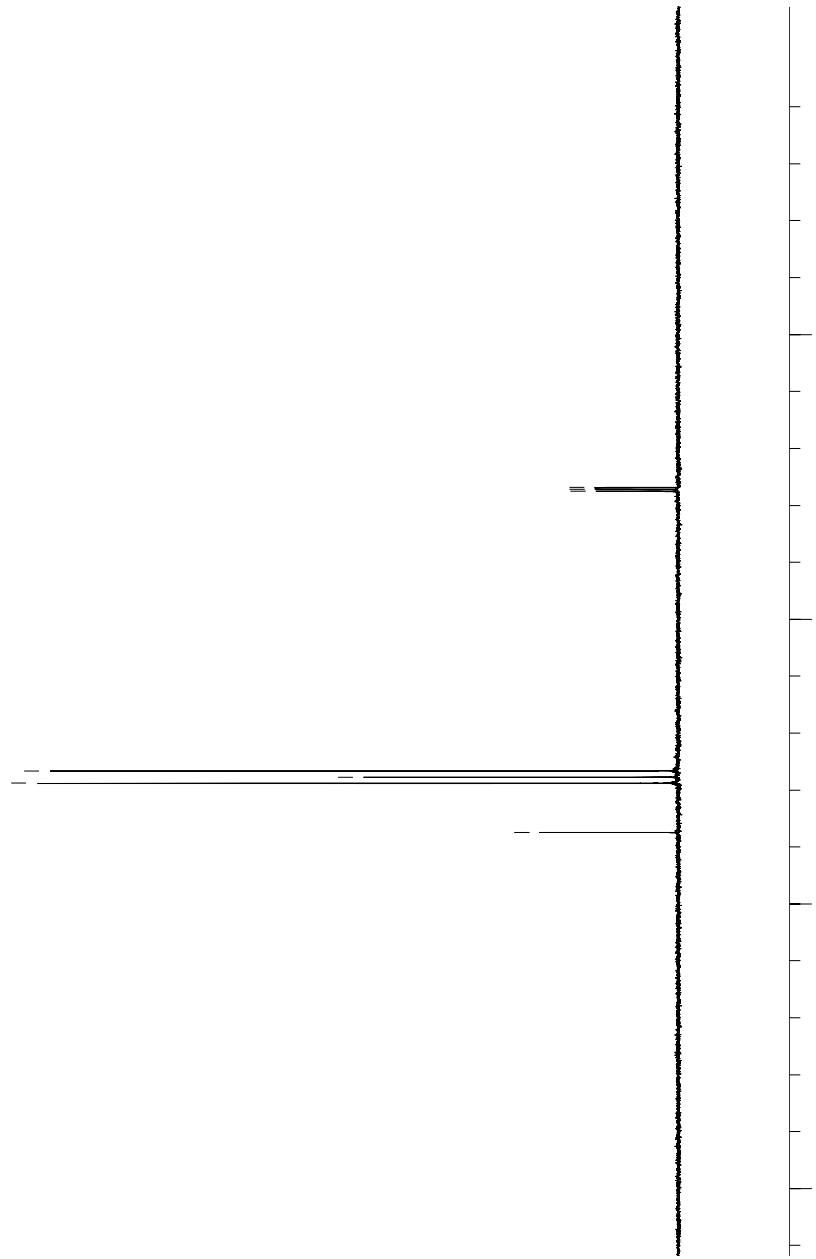


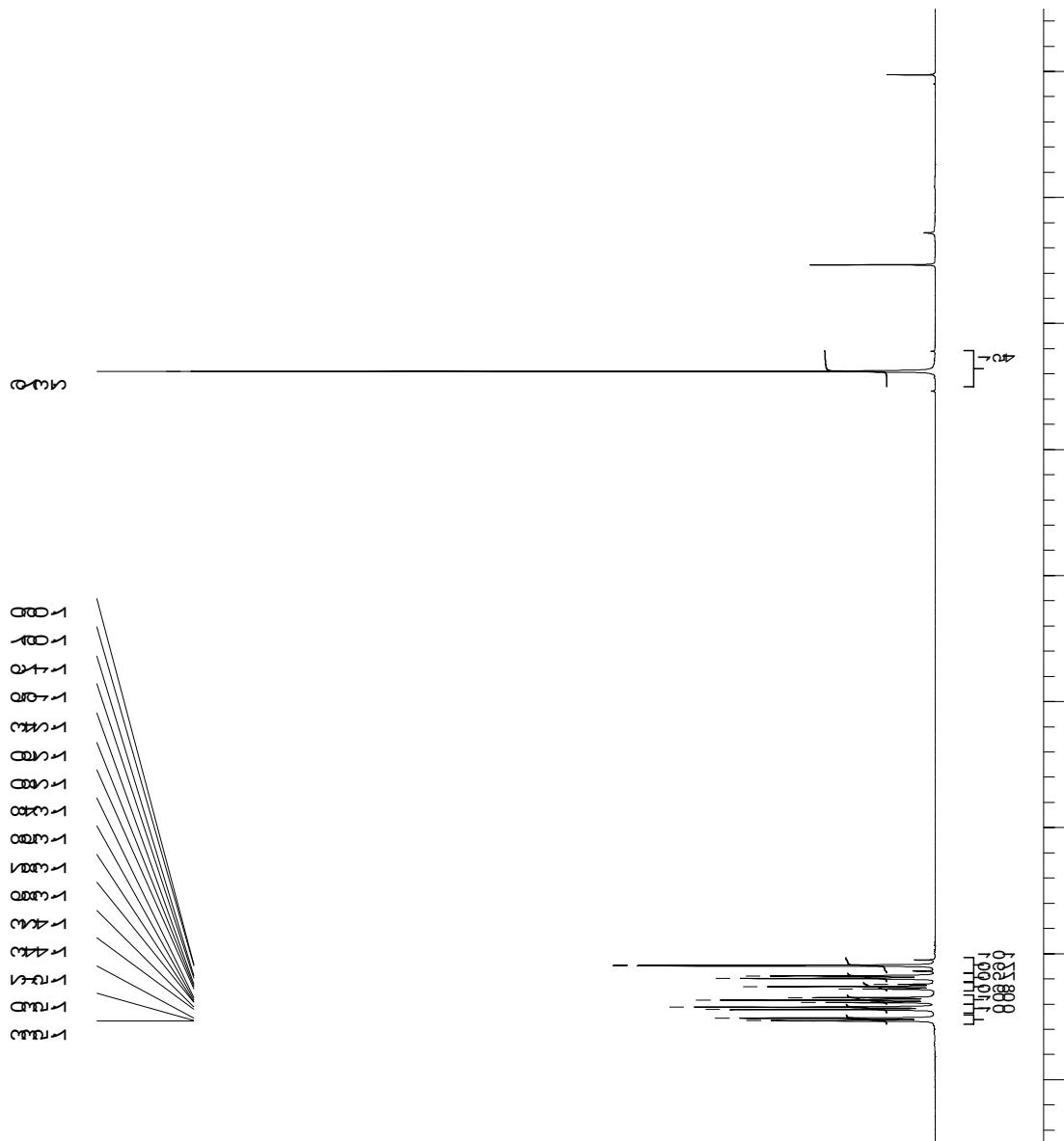
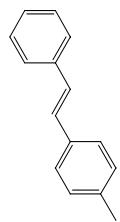




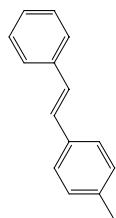
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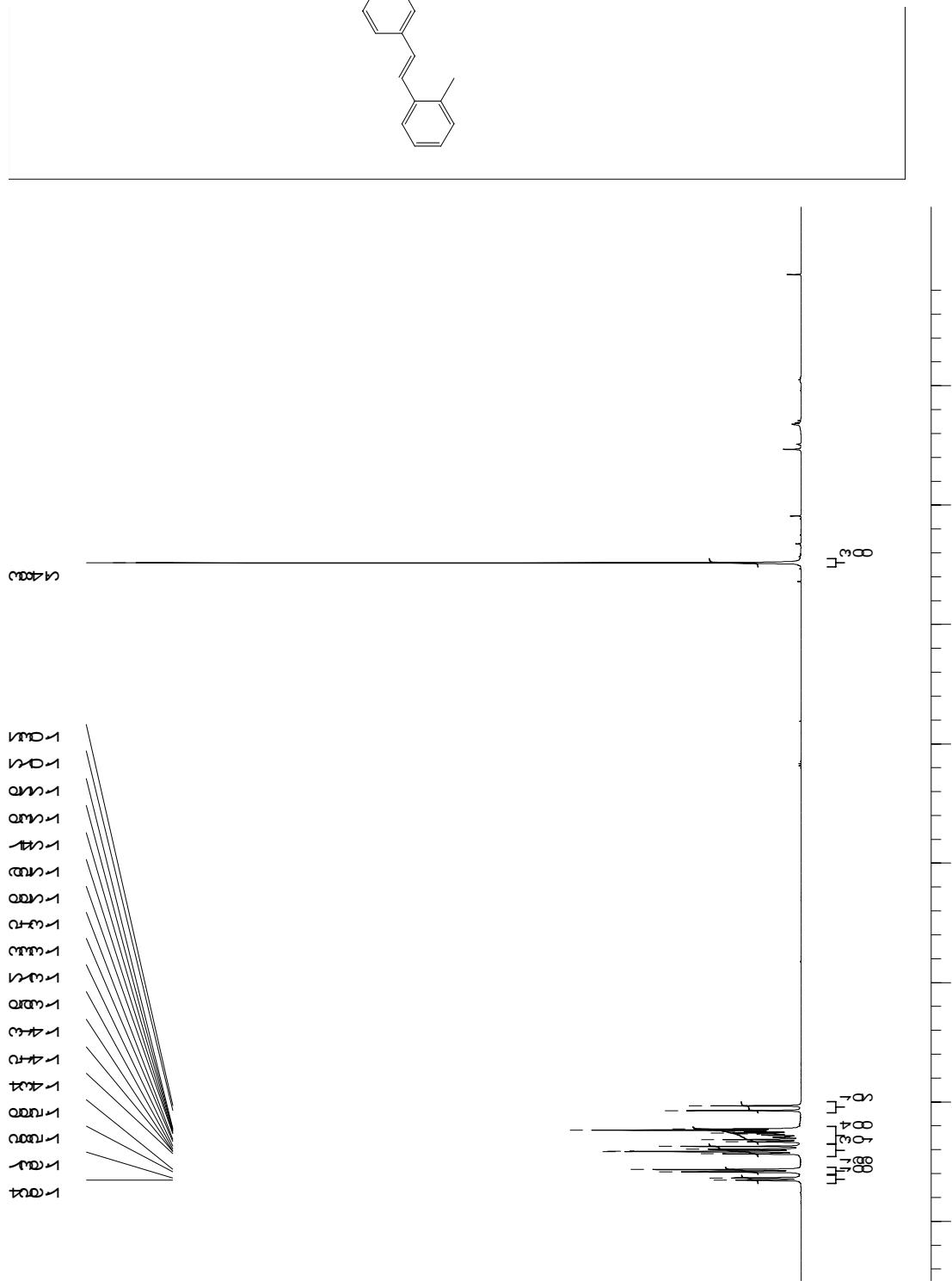
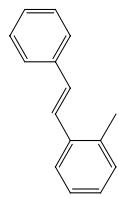
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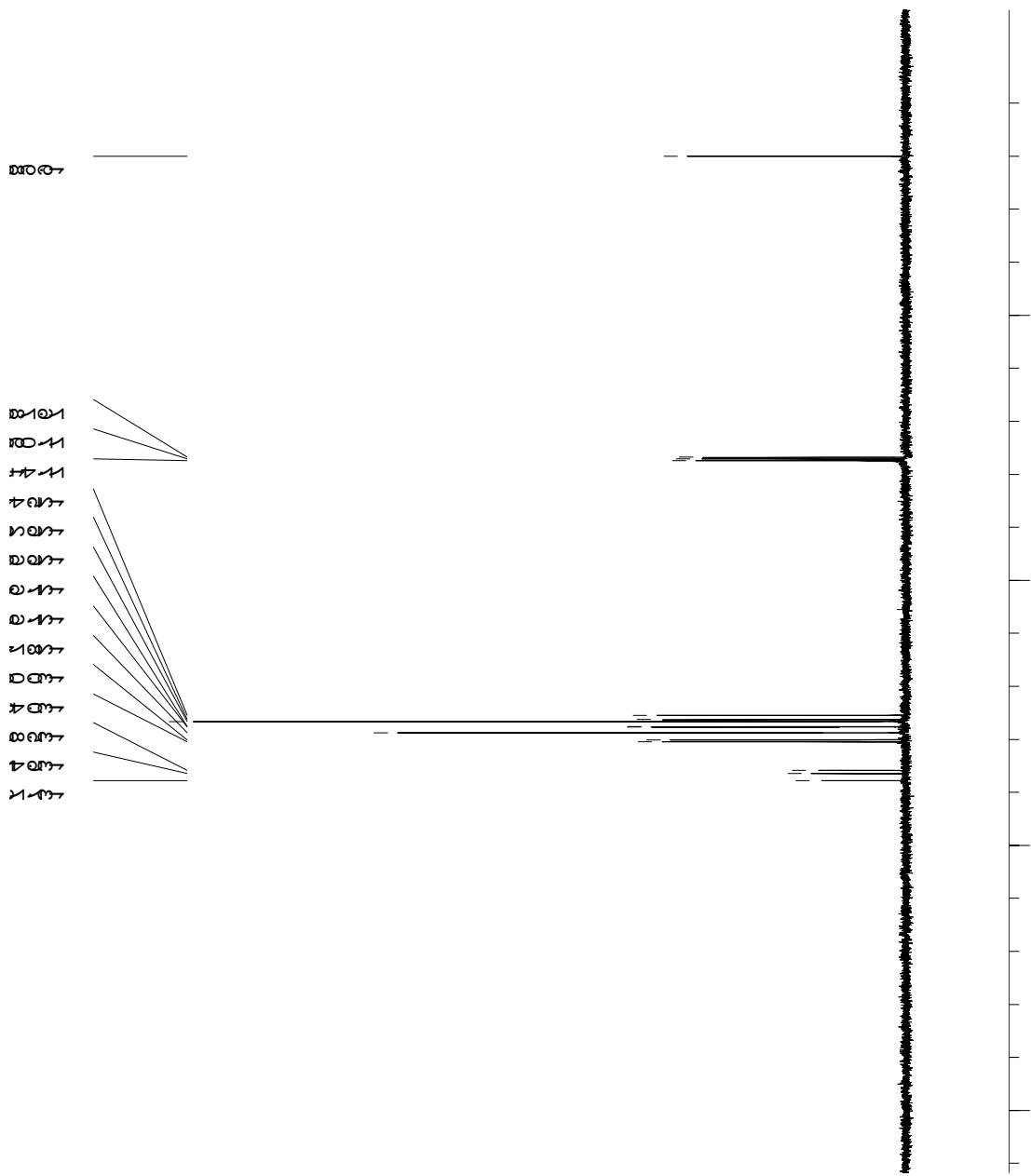
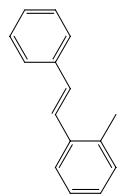


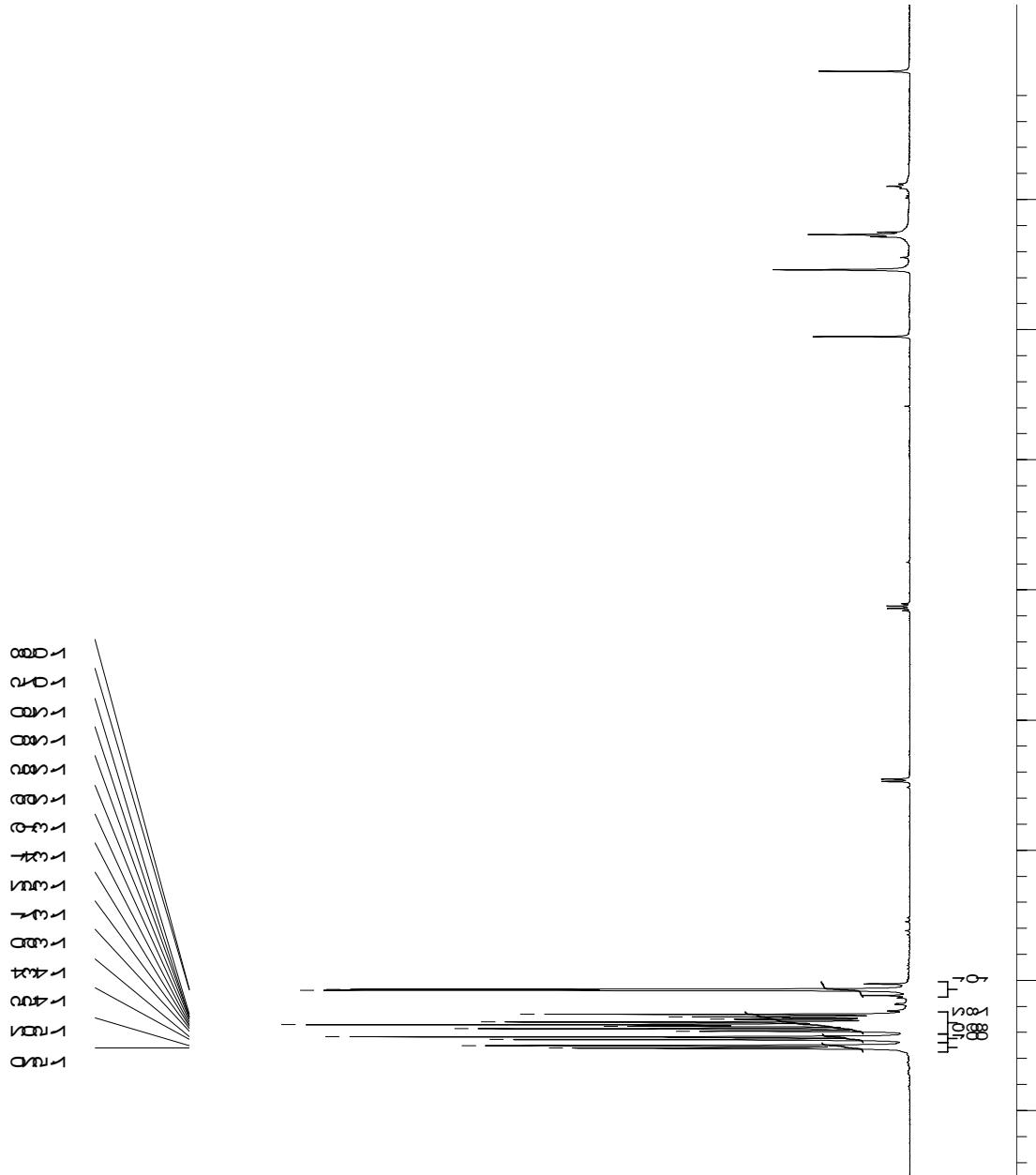
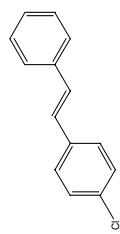
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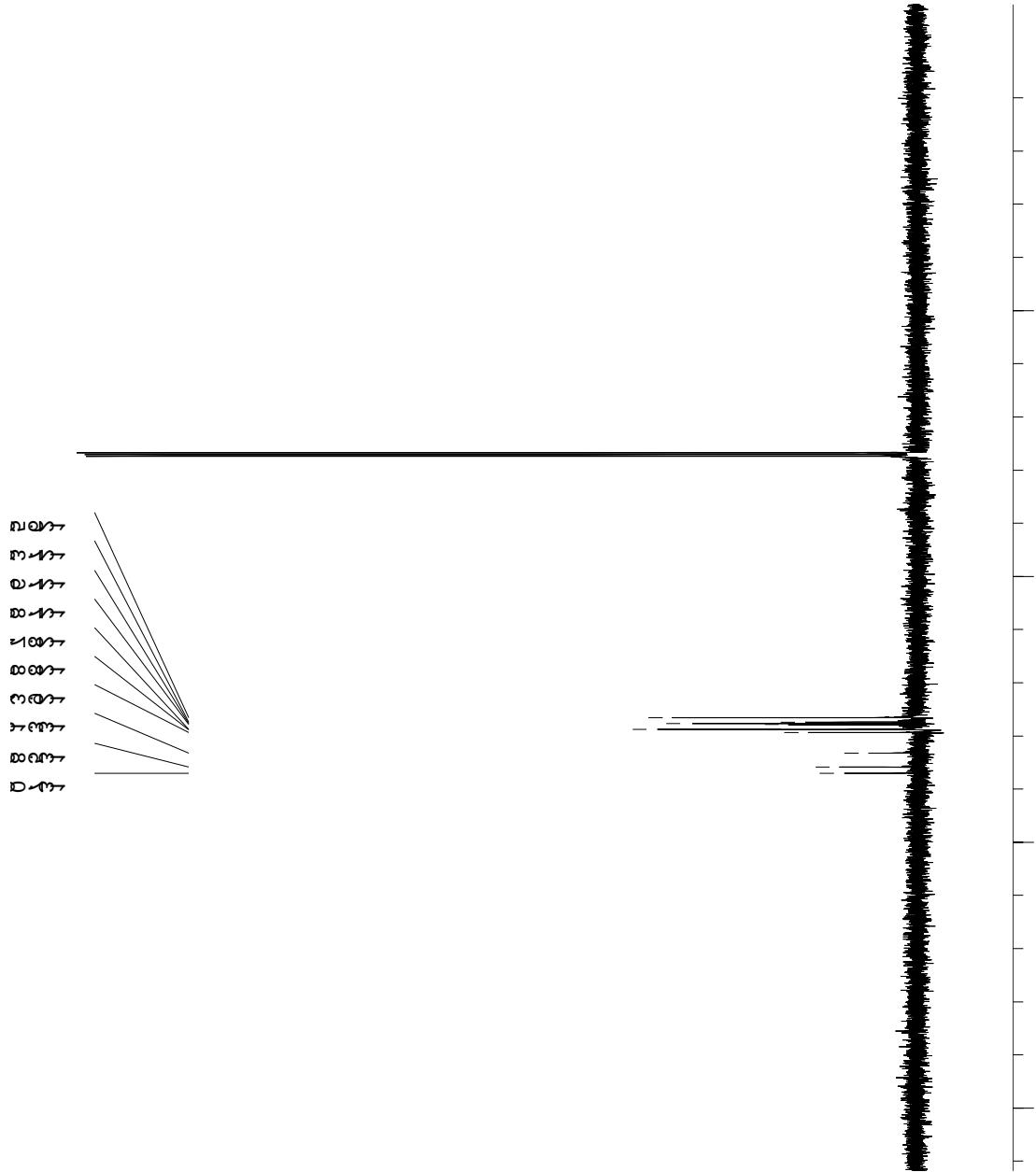
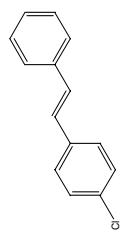
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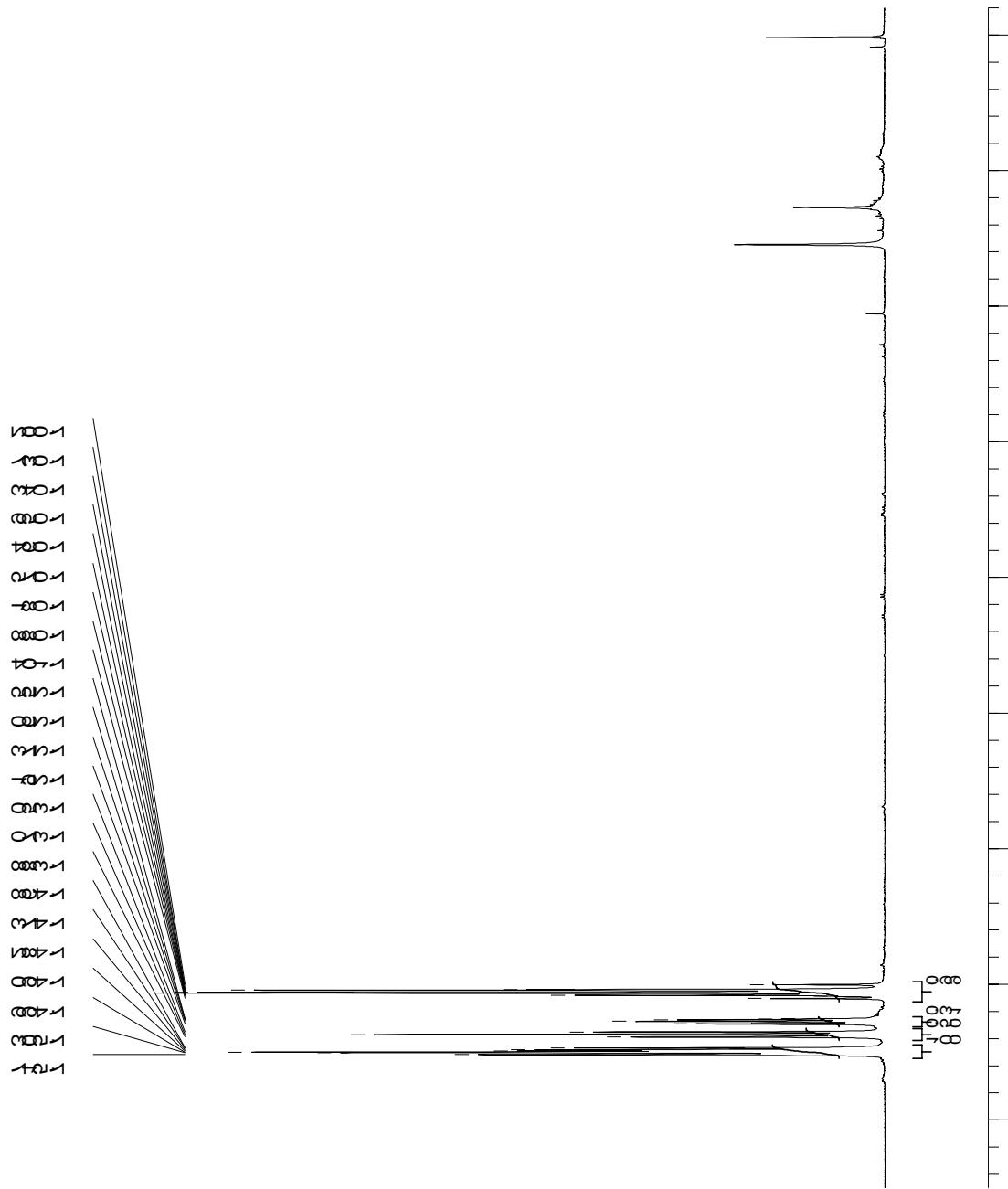
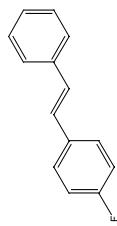


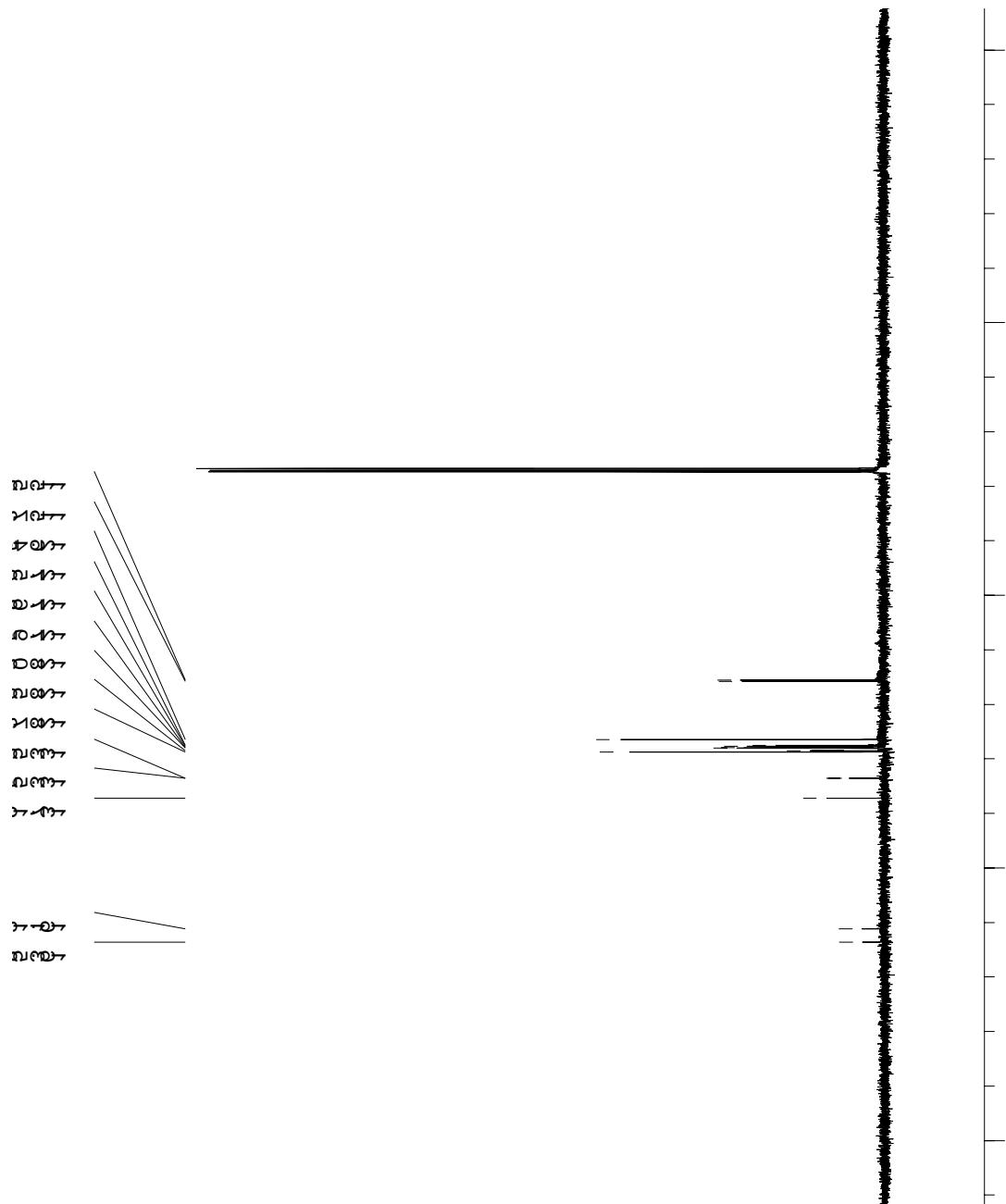
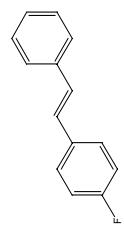


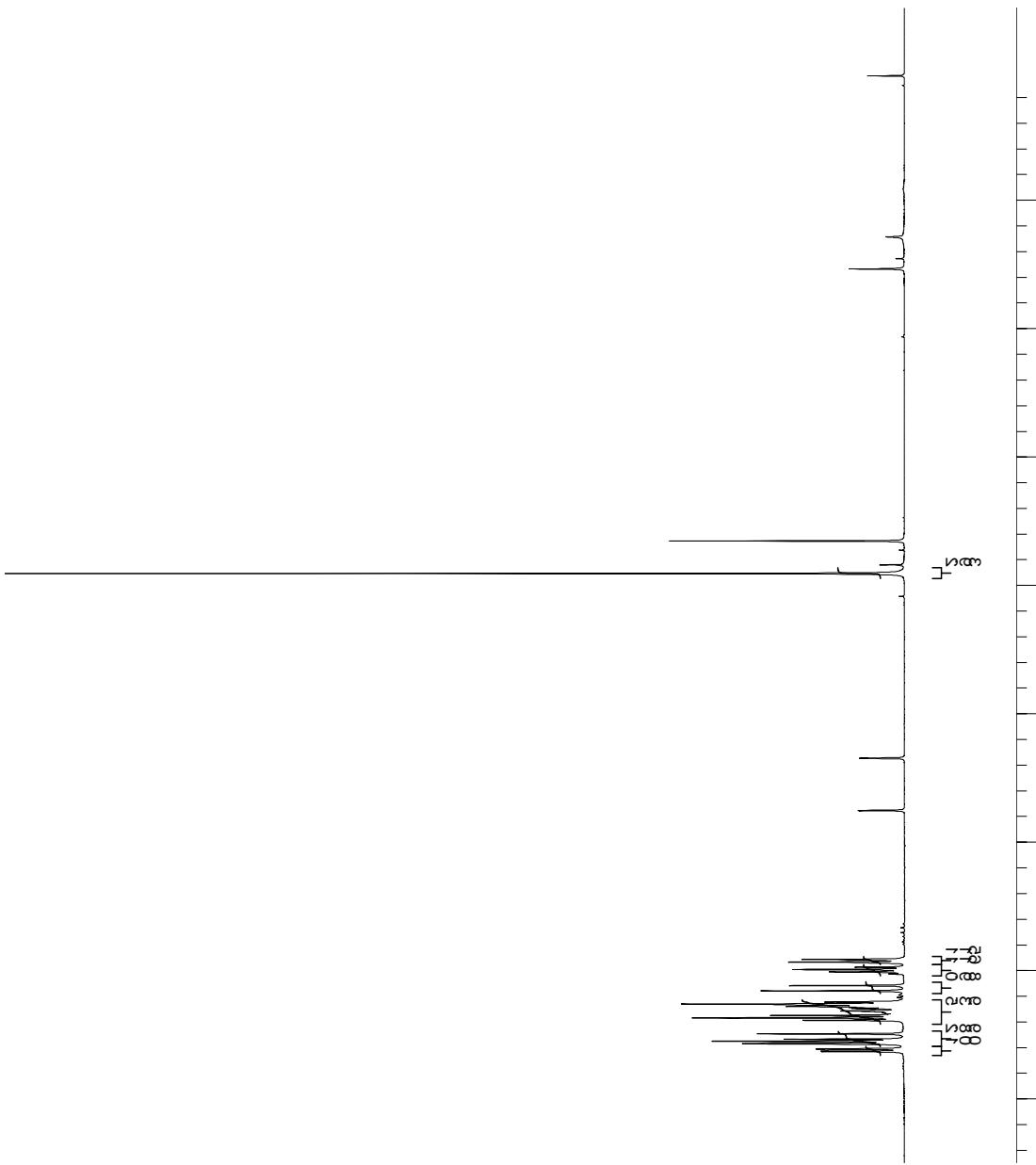
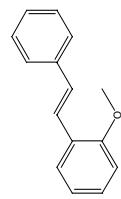


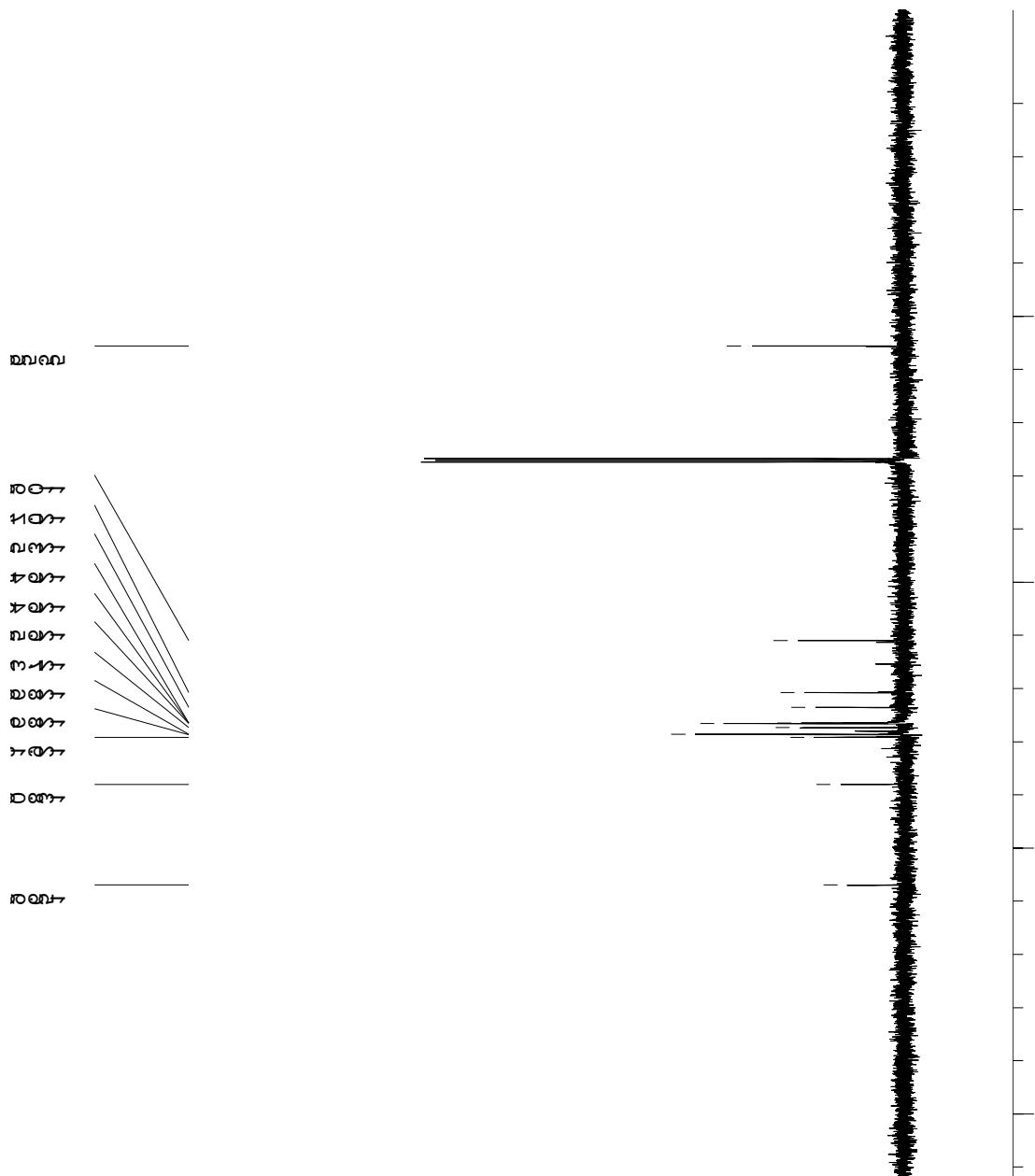
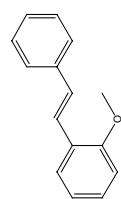
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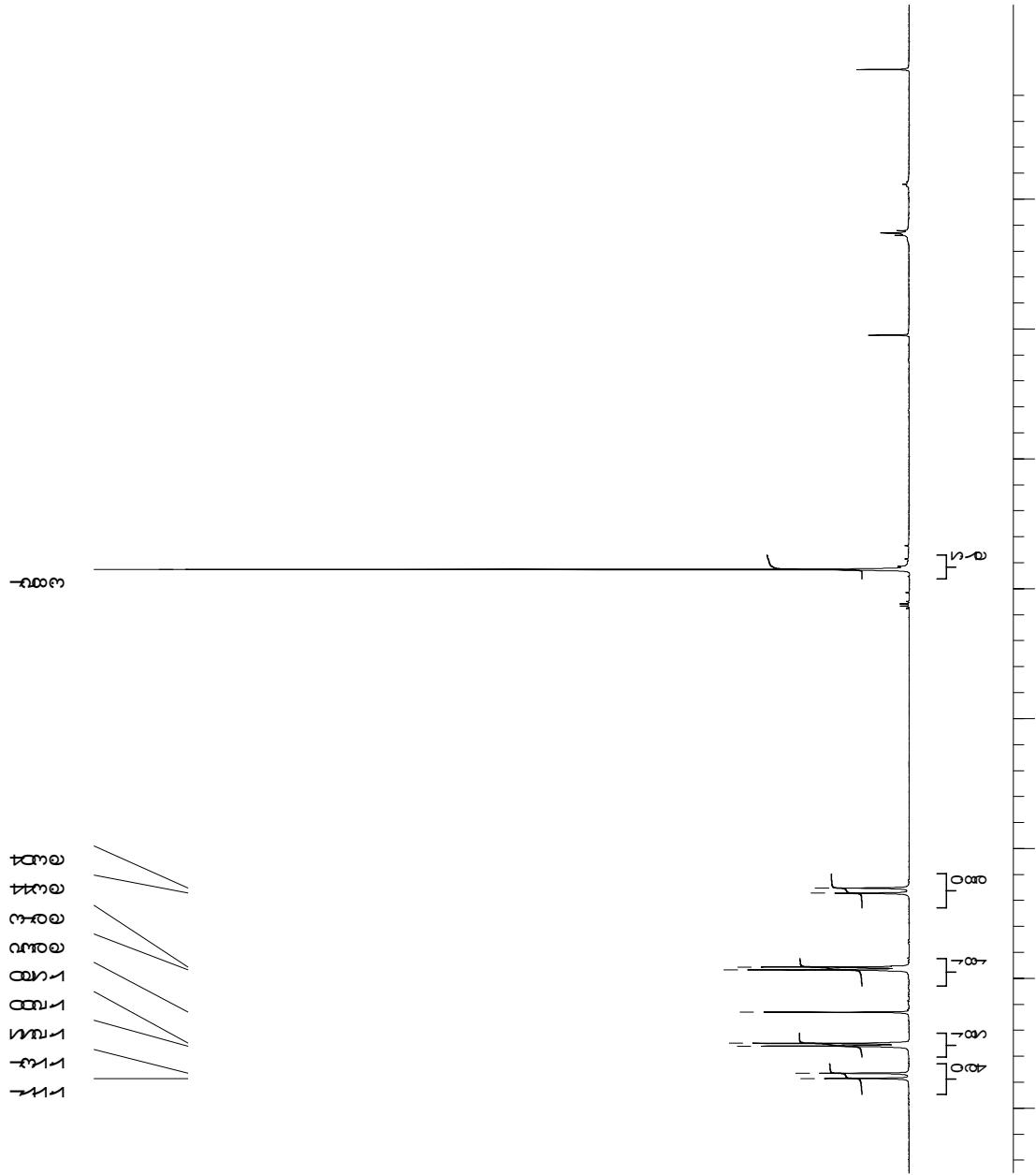
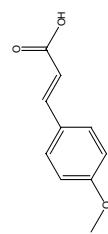


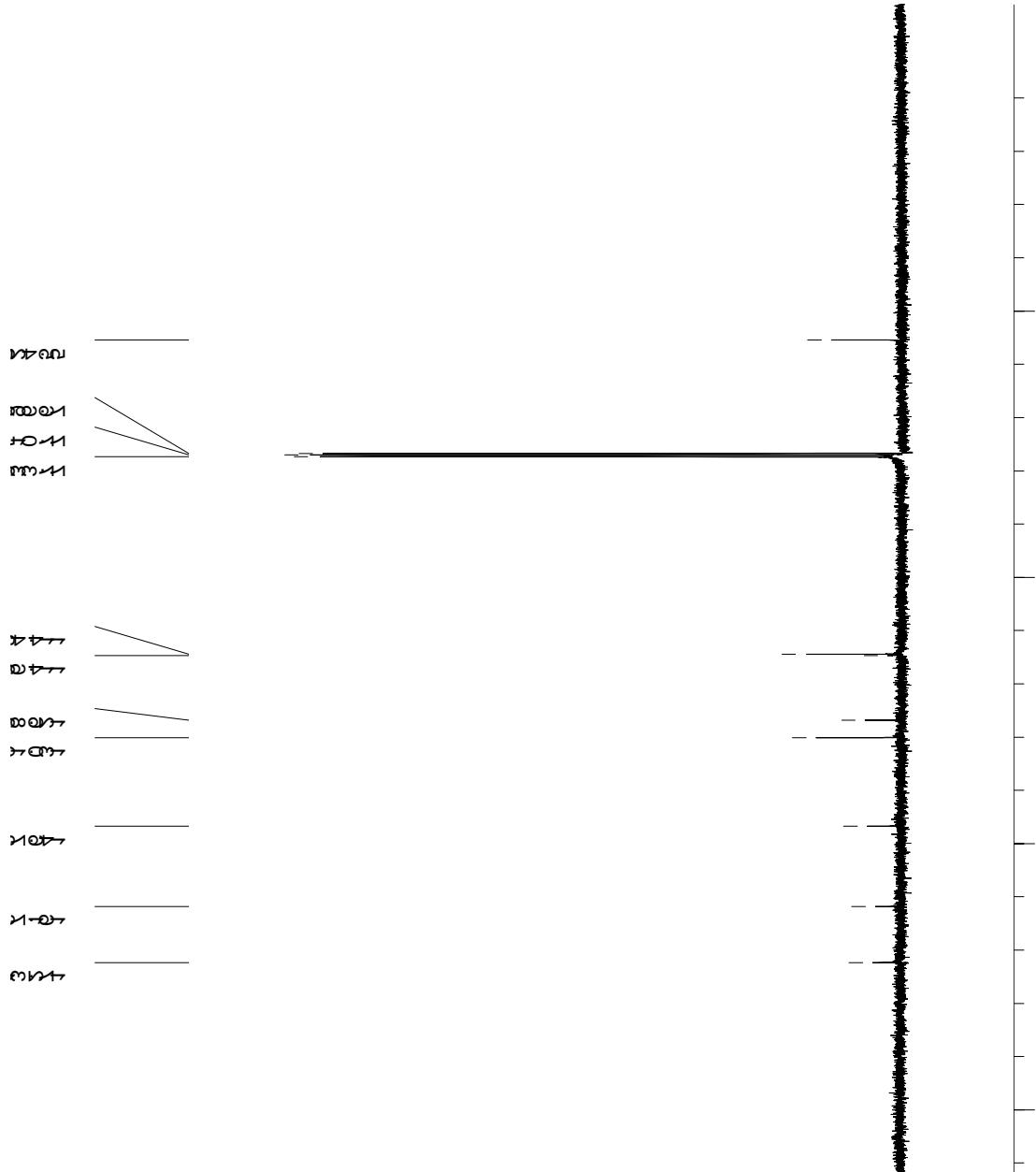
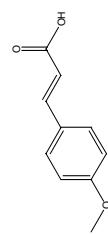


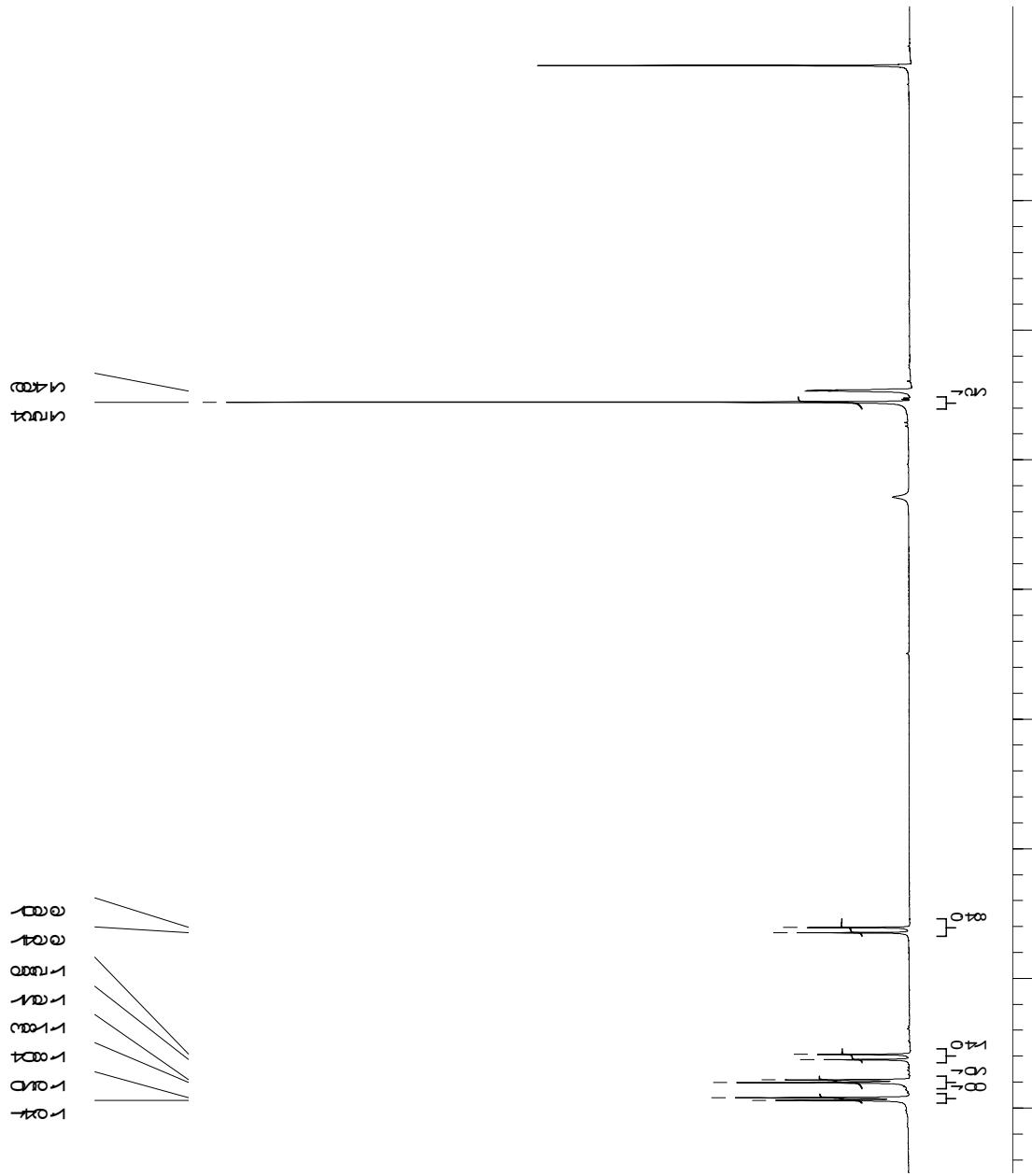
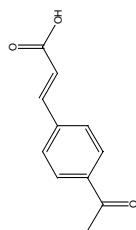


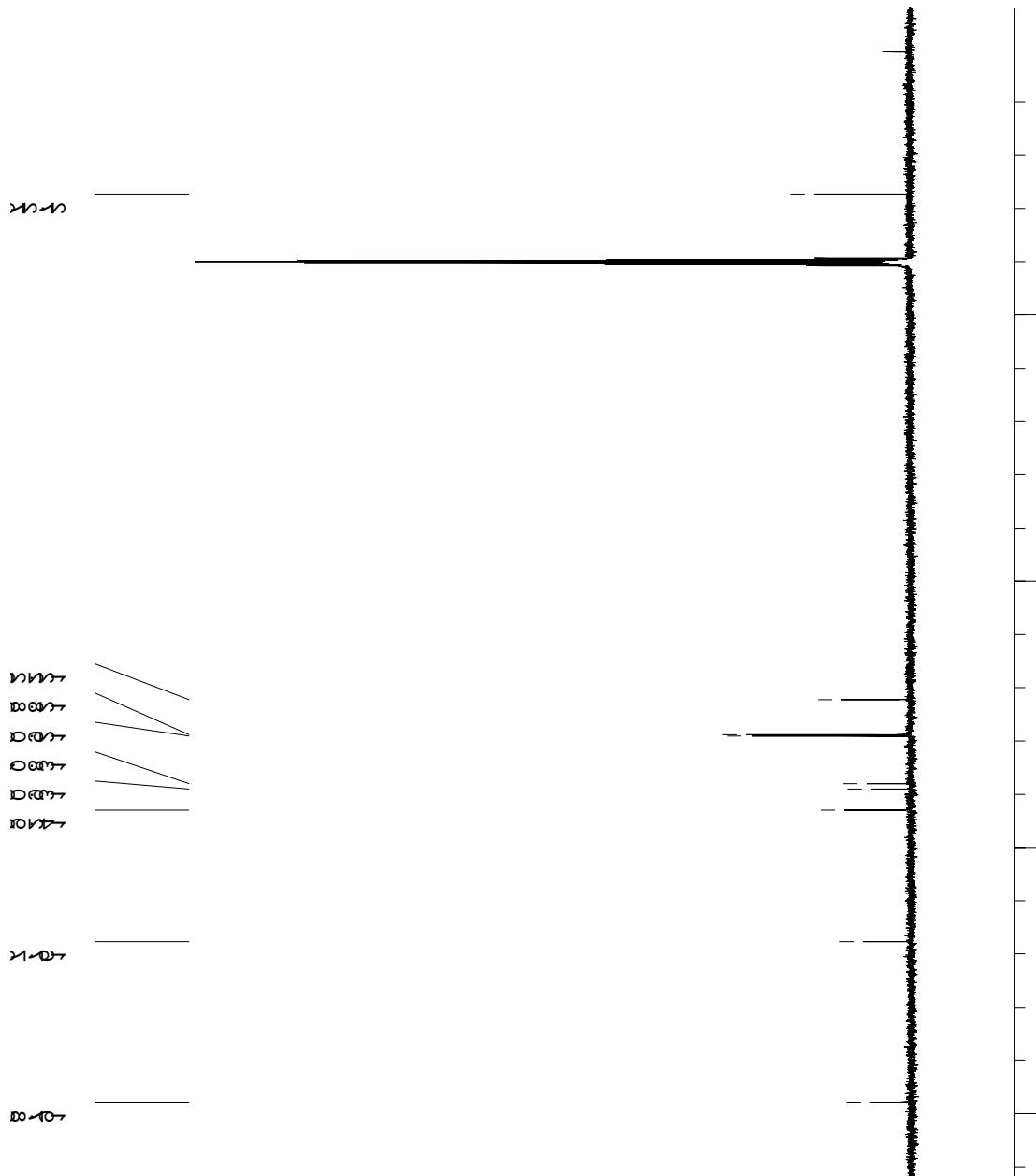
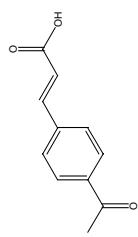


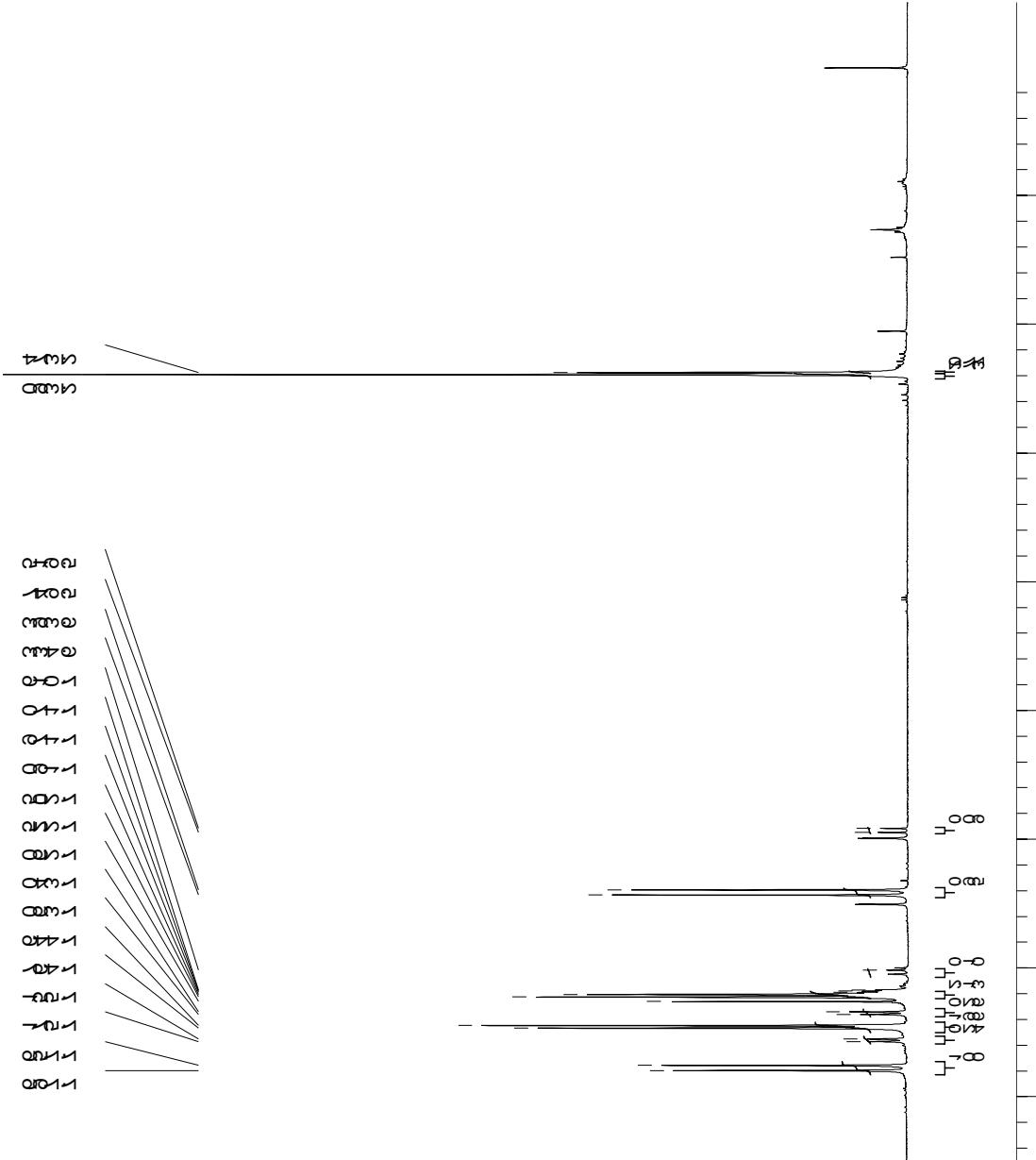


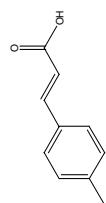












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