Supporting Information

Molecular Iodine Catalyzed Cross-Dehydrogenative Coupling Reaction between Two sp^3 C-H Bonds using Hydrogen Peroxide

Tomoya Nobuta, Norihiro Tada, Akitoshi Fujiya, Atsumasa Kariya, Tsuyoshi Miura, and Akichika Itoh*

Gifu Pharmaceutical University, 1-25-4 Daigaku-nishi, Gifu, 501-1196, Japan

E-mail: itoha@gifu-pu.ac.jp

1. General Information.

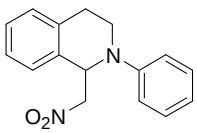
All dry solvents were obtained from Kanto Kagaku Co., Ltd. Other chemicals used were of reagent grade and were obtained from Tokyo Kasei Kogyo Co., Ltd., Wako Pure Chemical Industries, Ltd., Kishida Chemical Co., Ltd., and Nacalai Tesque. ^{1}H NMR and ^{13}C NMR spectra were obtained on a JEOL ECA 500 (500 MHz for ^{1}H NMR and 125 MHz for ^{13}C NMR). Chemical shifts (δ) are reported in parts per million (ppm) downfield from internal Me_4Si. Preparative thin-layer chromatography (TLC) was carried out on precoated plates of silica gel (MERCK, silica gel F-254).

2. General Procedure

Preparation of 2-aryl-1,2,3,4-tetrahydroisoquinolines[1]: Copper(I) iodide (200 mg, 1.0 mmol) and potassium phosphate (4.25 g, 20.0 mmol) were put into a two-neck flask. The two-neck flask was evacuated and back filled with nitrogen. 2-Propanol (10.0 mL), ethylene glycol (1.11 mL, 20.0 mmol), 1,2,3,4-tetrahydroisoquinoline (2.0 mL, 15.0 mmol) and iodobenzene (1.12 mL, 10.0 mmol) were added successively at room temperature. The reaction mixture was heated at 90 °C and kept for 24 h and then allowed to cool to room temperature. Diethyl ether (20 mL) and water (20 mL) were then added to the reaction mixture. The organic layer was extracted with diethyl ether (2 × 20 mL). The combined organic phases were washed with brine and dried over magnesium sulfate. The solvent was removed by rotary evaporation and purified by column chromatography on silica gel using hexane/ethyl acetate as eluent.

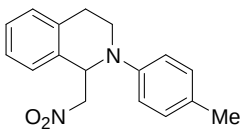
Synthesis of 1-(Nitromethyl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline (3aa) (Table 1, Entry 6): a solution of 2-phenyl-1,2,3,4-tetrahydroisoquinoline (**1a**, 0.3 mmol), I_2 (0.03 mmol) and 35% aq. H_2O_2 (0.6 mmol) in $MeNO_2$ (**2a**, 3 mL) was stirred at 40 °C for 12 h. The reaction mixture was washed with aq. $Na_2S_2O_3$, dried over magnesium sulfate, and concentrated *in vacuo*. Purification of the crude product by flash chromatography on silica gel (hexane : ethylacetate = 10 : 1) provided 1-(Nitromethyl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline (**3aa**) (74.6 mg, 93%).

1-(Nitromethyl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline (3aa)[2] (Table 2)



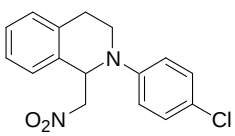
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.28-7.11 (m, 6H), 6.97 (d, *J* = 8.1 Hz, 2H), 6.84 (t, *J* = 7.4 Hz, 1 H), 5.54 (t, *J* = 7.2 Hz, 1 H), 4.85 (dd, *J* = 11.5, 7.5 Hz, 1 H), 4.54 (dd, *J* = 11.5, 6.3, 1H), 3.67-3.57 (m, 2H), 3.10-3.04 (m, 1H), 2.77 (dt, *J* = 16.1, 5.1 Hz, 1H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 148.5, 135.4, 133.0, 129.6, 129.3, 128.2, 127.1, 126.8, 119.5, 115.2, 78.9, 58.3, 42.2, 26.6.

1-(Nitromethyl)-2-*p*-tolyl-1,2,3,4-tetrahydroisoquinoline (3ba)[3] (Table 2)



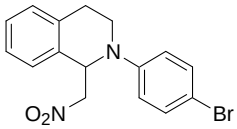
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.23-7.04 (m, 6H), 6.87 (d, *J* = 8.0 Hz, 2H), 5.47 (t, *J* = 7.2 Hz, 1 H), 4.81 (dd, *J* = 12.1, 8.0 Hz, 1 H), 4.52 (dd, *J* = 11.5, 6.3, 1H), 3.63-3.52 (m, 2H), 3.06-3.00 (m, 1H), 2.72 (dt, *J* = 16.6, 4.6 Hz, 1H), 2.24 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 146.5, 135.5, 133.1, 130.1, 129.4, 129.2, 128.1, 127.1, 126.7, 116.0, 78.9, 58.5, 42.4, 26.3, 20.5.

2-(4-Chlorophenyl)-1-(nitromethyl)-1,2,3,4-tetrahydroisoquinoline (3ca)[4] (Table 2)



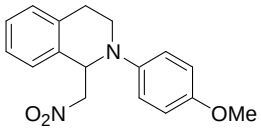
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.25-7.10 (m, 6H), 6.87 (d, *J* = 9.1 Hz, 2H), 5.47 (t, *J* = 7.2 Hz, 1 H), 4.81 (dd, *J* = 12.0, 8.6 Hz, 1 H), 4.54 (dd, *J* = 12.0, 6.3, 1H), 3.63-3.53 (m, 2H), 3.07-3.00 (m, 1H), 2.75 (dt, *J* = 16.6, 4.6 Hz, 1H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 147.2, 135.1, 132.6, 129.4, 128.4, 127.1, 126.9, 124.5, 116.6, 78.8, 58.3, 42.3, 26.2.

2-(4-Bromophenyl)-1-(nitromethyl)-1,2,3,4-tetrahydroisoquinoline (3da)[5] (Table 2)



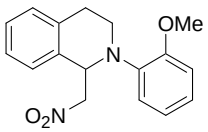
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.32-7.10 (m, 6H), 6.82 (dd, *J* = 12.1, 2.9 Hz, 2H), 5.46 (t, *J* = 7.5 Hz, 1 H), 4.80 (dd, *J* = 12.0, 8.0 Hz, 1 H), 4.54 (dd, *J* = 12.0, 6.3, 1H), 3.62-3.53 (m, 2H), 3.07-3.00 (m, 1H), 2.75 (dt, *J* = 16.0, 5.2 Hz, 1H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 147.6, 135.2, 132.6, 132.3, 129.4, 128.4, 127.1, 126.9, 116.9, 111.6, 78.7, 58.2, 42.2, 26.3.

2-(4-Methoxyphenyl)-1-(nitromethyl)-1,2,3,4-tetrahydroisoquinoline (3ea)[2] (Table 2)



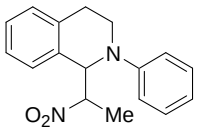
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.24-7.11 (m, 4H), 6.90 (dd, *J* = 12.6, 4.0 Hz, 2H), 6.80 (dd, *J* = 12.6, 3.4 Hz, 2H), 5.37 (dd, *J* = 8.5, 6.3 Hz, 1 H), 4.80 (dd, *J* = 12.0, 3.5 Hz, 1 H), 4.53 (dd, *J* = 12.0, 6.3, 1H), 3.72 (s, 3H), 3.58-3.49 (m, 2H), 3.02-3.00 (m, 1H), 2.67 (dt, *J* = 16.6, 4.0 Hz, 1H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 154.1, 143.2, 135.5, 133.0, 129.6, 128.0, 127.0, 126.7, 118.9, 114.8, 79.0, 59.0, 55.7, 43.2, 25.9.

2-(2-Methoxyphenyl)-1-(nitromethyl)-1,2,3,4-tetrahydroisoquinoline (3fa)[2] (Table 2)



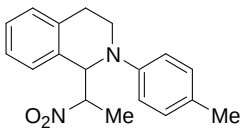
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.24-7.14 (m, 4H), 7.03-7.01 (m, 1), 7.00-6.81 (m, 3H), 5.50-5.48 (m, 1 H), 4.81 (dd, *J* = 12.0, 8.6 Hz, 1 H), 4.52 (dd, *J* = 12.0, 5.2, 1H), 3.81 (s, 3H), 3.59 (dd, *J* = 13.2, 5.2 Hz, 1H), 3.47 (ddd, *J* = 13.7, 11.4, 4.0, 1H), 3.01-2.94 (m, 1H), 2.70 (d, *J* = 16.6 Hz, 1H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 153.2, 139.0, 135.5, 133.8, 129.7, 127.7, 126.9, 126.5, 124.2, 122.1, 121.1, 112.6, 79.3, 58.3, 55.9, 43.1, 27.0.

1-(Nitroethyl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline (3ab)[2] (Table 2)



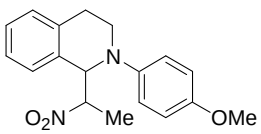
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.28-7.08 (m, 6H), 7.01-6.97 (m, 2H), 6.83-6.79 (m, 1H), 5.25-5.21 (m, 1H), 5.06-4.84 (m, 1H), 3.84-3.50 (m, 2H), 3.06-2.83 (m, 2H), [1.68 (d, *J* = 6.9 Hz), 1.52 (d, *J* = 6.9), 3H]. ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 149.3, 149.0, 135.7, 134.9, 133.9, 132.1, 129.6, 129.4, 129.2, 128.8, 128.5, 128.3, 127.4, 126.7, 126.2, 119.4, 118.9, 115.5, 114.6, 89.1, 85.6, 62.9, 61.3, 43.7, 42.8, 26.9, 26.5, 17.6, 16.5.

1-(Nitroethyl)-2-*p*-tolyl-1,2,3,4-tetrahydroisoquinoline (3bb)[3] (Table 2)



^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.24-6.99 (m, 6H), 6.89-6.87 (m, 2H), 5.18-5.14 (m, 1H), 5.04-4.83 (m, 1H), 3.82-3.48 (m, 2H), 3.03-2.79 (m, 2H), [2.25 (s), 2.23 (s), 3H], [1.67 (d, *J* = 6.9 Hz), 1.51 (d, *J* = 5.7), 3H]. ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 147.3, 146.9, 135.8, 135.0, 133.9, 132.2, 130.0, 129.9, 129.3, 129.0, 128.9, 128.5, 128.2, 127.4, 126.6, 126.2, 116.2, 115.3, 89.1, 85.6, 63.0, 61.6, 44.0, 43.1, 26.7, 26.4, 20.5, 20.4, 17.5, 16.5.

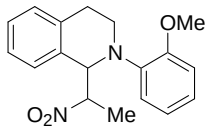
2-(4-Methoxyphenyl)-1-(nitroethyl)-1,2,3,4-tetrahydroisoquinoline (3eb)[2] (Table 2)



^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.25-7.01 (m, 4H), 6.92-6.90 (m, 2H), 6.83-6.76 (m, 2H), 5.07-4.84 (m, 2H), 3.80-3.45 (m, 5H), 3.00-2.74 (m, 2H), [1.67 (d, *J* = 6.3 Hz), 1.52 (d, *J* = 5.7), 3H]. ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 153.9, 153.7, 144.0, 143.6, 135.9, 135.1, 133.8, 132.2, 129.4, 129.1, 128.5, 128.1, 127.3, 126.6, 126.1, 119.0, 118.3, 114.8, 114.7, 88.9, 85.9, 63.6, 62.3,

55.7, 45.1, 44.1, 26.4, 26.1, 17.2, 16.7.

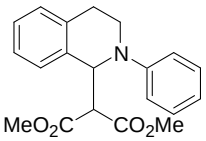
2-(2-Methoxyphenyl)-1-(nitroethyl)-1,2,3,4-tetrahydroisoquinoline (3fb)[2] (Table 2)



^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.26-7.11 (m, 4H), 7.04-6.98 (m, 1H), 6.89-6.78 (m, 3H), 5.02-4.98 (m, 1H), 4.93-4.80 (m, 1H), [3.82 (s), 3.76 (s), 3H], 3.66-3.38 (m, 2H), 2.88-2.72 (m, 2H), [1.64 (d, *J* = 6.9 Hz), 1.45 (d, *J* = 6.9), 3H]. ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 153.9, 153.7, 139.9, 139.7, 136.4, 135.5, 134.2, 133.0, 129.33, 129.29, 128.2, 127.8, 127.7, 127.1, 126.4, 126.0, 124.4, 124.3, 123.6, 123.3, 121.2, 121.1, 113.1, 112.1, 88.8, 86.2, 63.5, 63.0, 56.1, 55.6, 45.1, 44.6, 27.3, 27.0, 16.9, 16.8.

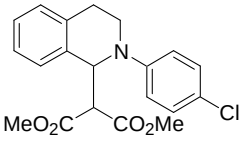
Synthesis of 2-(2-Phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)-malonic acid dimethyl ester (5aa) (Table 3): a solution of 2-phenyl-1,2,3,4-tetrahydroisoquinoline (**1a**, 0.3 mmol), I_2 (0.03 mmol) and 35% aq H_2O_2 (0.6 mmol) in dimethyl malonate (**6a**, 1 mL) was stirred at 40 °C for 12 h. The reaction mixture was washed with aq. $Na_2S_2O_3$, dried over sodium sulfate, and concentrated *in vacuo*. Purification of the crude product by flash chromatography on silica gel (hexane : ethylacetate : triethylamine = 100 : 1 : 5) provided 2-(2-Phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)-malonic acid dimethyl ester (**7aa**) (63.8 mg, 63%).

2-(2-Phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)-malonic acid dimethyl ester (5aa)[6] (Table 3)



^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.23-7.09 (m, 6H), 6.98 (d, *J* = 8.0 Hz, 2H), 6.75 (t, *J* = 6.9 Hz, 1 H), 5.70 (d, *J* = 9.2 Hz, 1 H), 3.95 (d, *J* = 9.1 Hz, 1 H), 3.74-3.60 (m, 5H), 3.54 (s, 3H), 3.09-3.03 (m, 1H), 2.89-2.84 (m, 1H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 168.4, 167.5, 148.9, 135.8, 134.9, 129.2, 129.1, 127.7, 127.2, 126.2, 118.7, 115.3, 59.2, 58.3, 52.7, 42.3, 26.1.

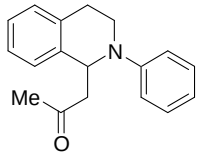
2-(2-(4-Chlorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)-malonic acid dimethyl ester (5ca)[7] (Table 3)



^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.21-7.10 (m, 6H), 6.89 (d, *J* = 9.1 Hz, 2H), 5.64 (d, *J* = 9.2 Hz, 1 H), 3.91 (d, *J* = 9.8 Hz, 1 H), 3.69-3.51 (m, 8H), 3.07-3.01 (m, 1H), 2.92-2.86 (m, 1H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 168.2, 167.4, 147.4, 135.5, 134.7, 129.1, 129.0, 127.9, 127.1, 126.3, 123.4, 116.2, 59.14, 59.11, 58.3, 52.7, 42.6, 26.1.

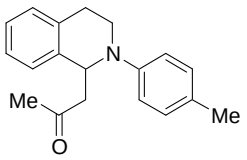
Synthesis of 1-(2-Phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)propan-2-one (5ab) (Table 3): a solution of 2-phenyl-1,2,3,4-tetrahydroisoquinoline (**1a**, 0.3 mmol), I_2 (0.03 mmol), AcOH (1.5 mmol) and 35% aq H_2O_2 (0.6 mmol) in acetone (**2a**, 3 mL) was stirred at 50 °C for 12 h. The reaction mixture was washed with aq. $NaHCO_3$, dried over magnesium sulfate, and concentrated *in vacuo*. Purification of the crude product by PTLC (hexane : ethylacetate = 5 : 1) provided 1-(2-Phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)propan-2-one (**5aa**) (Rf = 0.50, 55.9 mg, 70%).

1-(2-Phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)propan-2-one (5ab)[8] (Table 3)



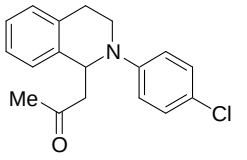
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.25-7.11 (m, 6H), 6.92 (d, *J* = 8.6 Hz, 2H), 6.76 (t, *J* = 7.5 Hz, 1 H), 5.39 (t, *J* = 6.3 Hz, 1H), 3.65-3.61 (m, 1H), 3.54-3.49 (m, 1H), 3.06-3.01 (m, 2H), 2.83-2.78 (m, 2H), 2.05 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 207.4, 149.0, 138.4, 134.6, 129.5, 128.8, 127.0, 126.9, 126.4, 118.4, 114.9, 54.9, 50.3, 42.2, 31.2, 27.3.

1-(2-p-Tolyl-1,2,3,4-tetrahydroisoquinolin-1-yl)propan-2-one (5bb)[8] (Table 3)



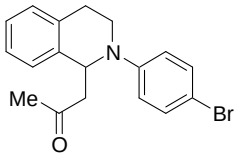
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.16-7.09 (m, 4H), 7.04 (d, *J* = 8.6 Hz, 2H), 6.85 (d, *J* = 8.6 Hz, 2H), 5.33 (t, *J* = 6.3 Hz, 1H), 3.63-3.59 (m, 1H), 3.50-3.44 (m, 1H), 3.05-2.99 (m, 2H), 2.80-2.73 (m, 2H), 2.23 (s, 3H), 2.05 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 207.5, 147.0, 138.4, 134.5, 123.0, 128.9, 128.1, 127.0, 126.8, 126.3, 115.8, 55.3, 50.2, 42.3, 31.1, 27.1, 20.5.

1-(2-(4-Chlorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)propan-2-one (5cb)[8] (Table 3)



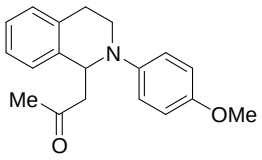
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.18-7.11 (m, 6H), 6.84 (d, *J* = 9.2 Hz, 2H), 5.33 (t, *J* = 6.9 Hz, 1H), 3.60-3.47 (m, 2H), 3.05-3.01 (m, 2H), 3.00-2.78 (m, 2H), 2.07 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 207.2, 147.6, 138.0, 134.3, 129.2, 128.8, 127.1, 126.9, 126.5, 123.0, 115.9, 54.9, 50.2, 42.3, 31.2, 27.1.

1-(2-(4-Bromophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)propan-2-one (5db)[8] (Table 3)



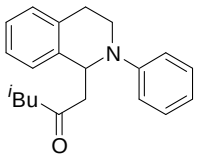
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.29 (d, *J* = 9.2, 2H), 7.17-7.11 (m, 4H), 6.79 (d, *J* = 9.1 Hz, 2H), 5.33 (t, *J* = 6.3 Hz, 1H), 3.59-3.46 (m, 2H), 3.05-2.98 (m, 2H), 2.83-2.79 (m, 2H), 2.06 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 207.2, 147.9, 138.0, 134.3, 132.1, 128.8, 127.1, 126.9, 126.5, 116.2, 110.1, 54.7, 50.2, 42.2, 31.3, 27.2.

1-(2-(4-Bromophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)propan-2-one (5eb)[8] (Table 3)



^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.16-7.09 (m, 4H), 6.91 (d, *J* = 9.1, 2H), 6.81 (d, *J* = 9.2 Hz, 2H), 5.23 (t, *J* = 6.3 Hz, 1H), 3.57-3.53 (m, 1H), 3.48-3.42 (m, 1H), 3.02-2.96 (m, 2H), 2.78-2.70 (m, 2H), 2.05 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 207.5, 153.4, 143.8, 138.3, 134.4, 129.1, 126.9, 126.7, 126.3, 118.5, 114.7, 56.1, 55.7, 50.1, 43.0, 31.0, 26.8.

4-Methyl-1-(2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)pentan-2-one (5ac)[9] (Table 3)



^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.23 (t, *J* = 7.4, 2H), 7.13 (d, *J* = 7.4, 4H), 6.93 (d, *J* = 8.0, 2H), 6.75 (t, *J* = 6.8, 1H), 5.43 (t, *J* = 6.3, 1H), 3.65-3.60 (m, 1H), 3.54-3.48 (m, 1H), 3.07-2.98 (m, 2H), 2.84-2.73 (m, 2H), 2.22-2.04 (m, 3H), 8.34 (t, *J* = 4.0, 6H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 209.3, 149.0, 138.6, 134.5, 129.5, 128.7, 127.0, 126.9, 126.4, 118.2, 114.7, 54.8, 53.2, 49.8, 42.1, 27.5, 24.5, 22.7, 22.6.

3. References

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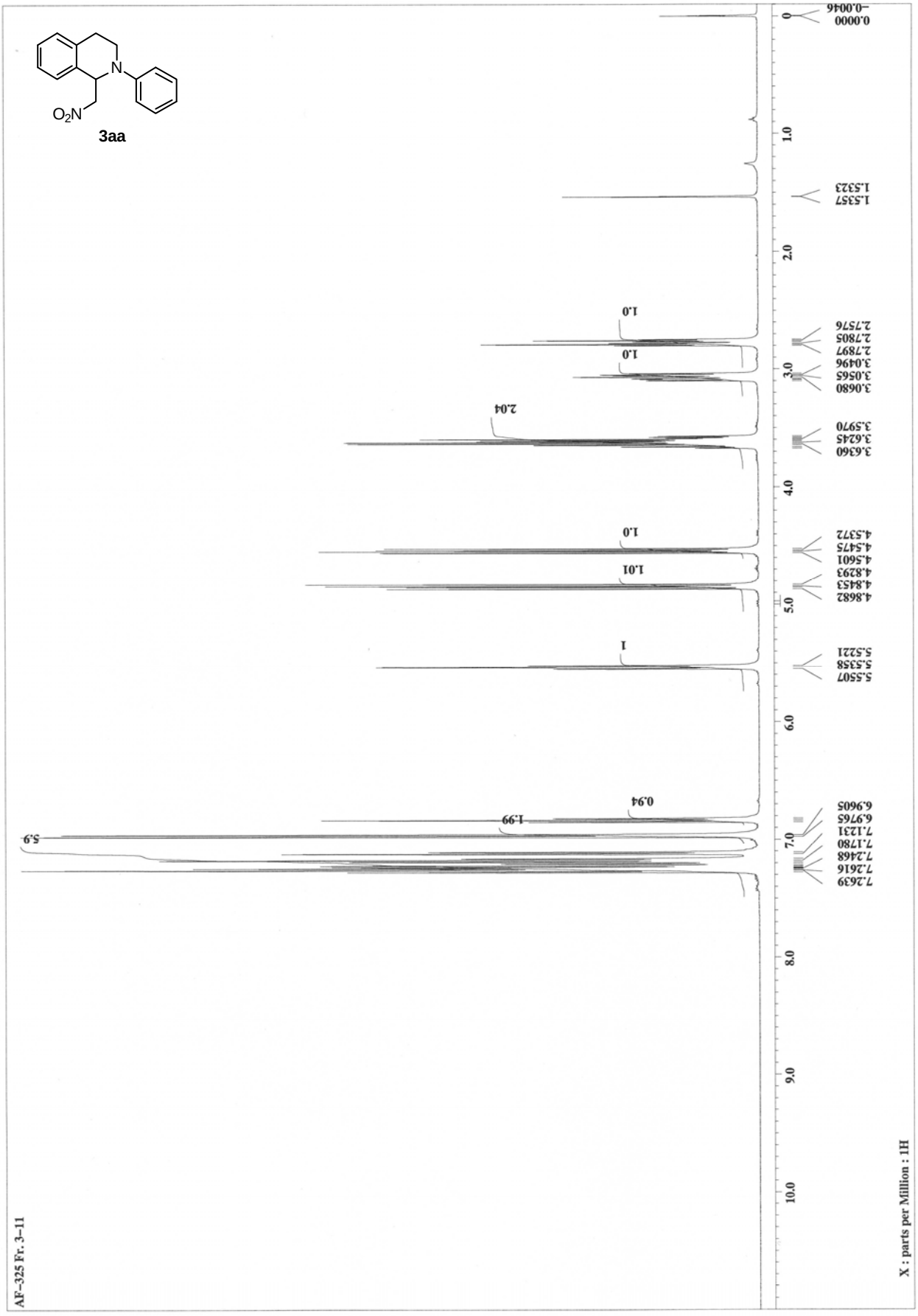
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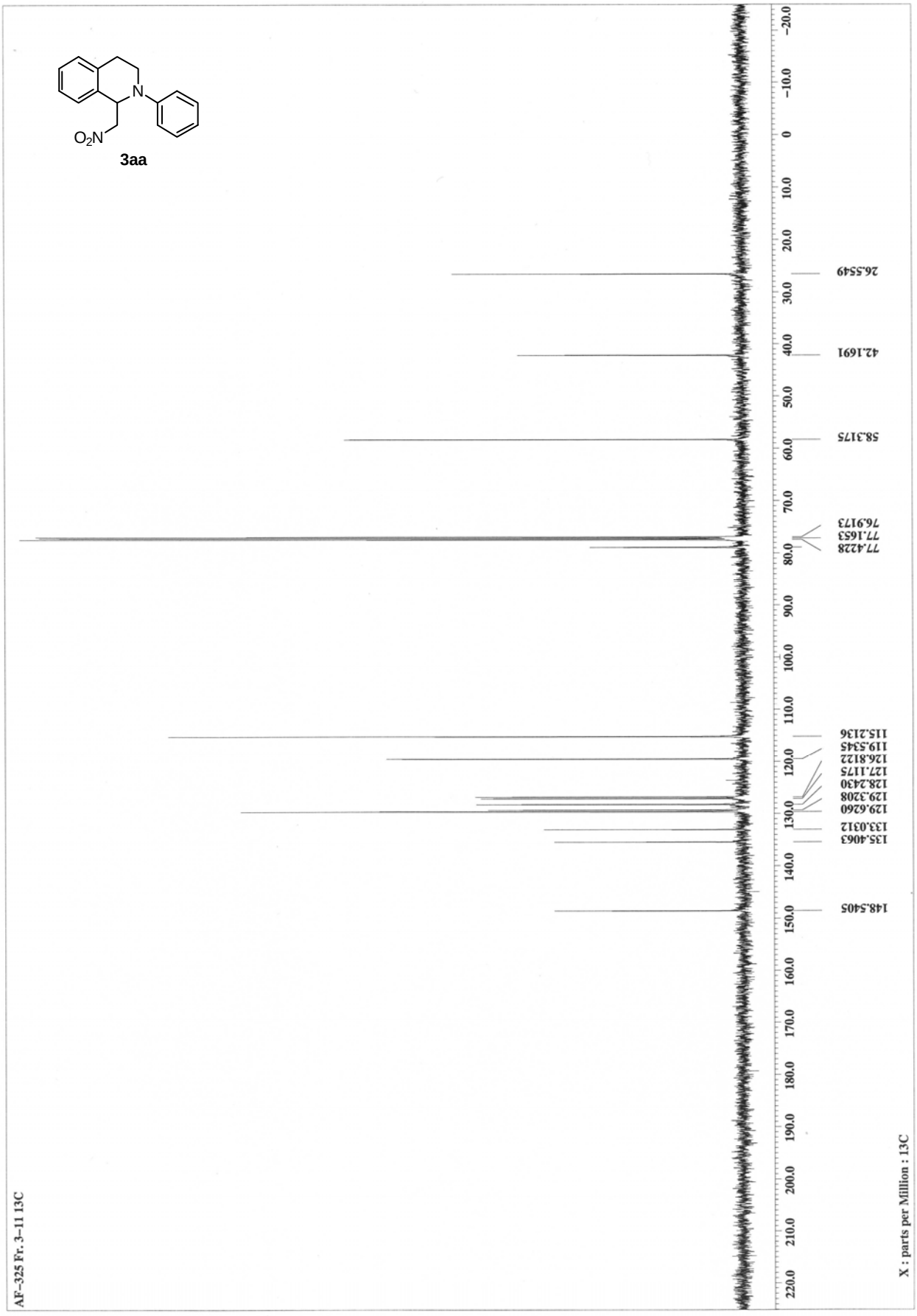
AF-325 Fr. 3-11
X : parts per Million : 1H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3aa
AF-325 Fr. 3-11 13C
X : parts per Million : 13C
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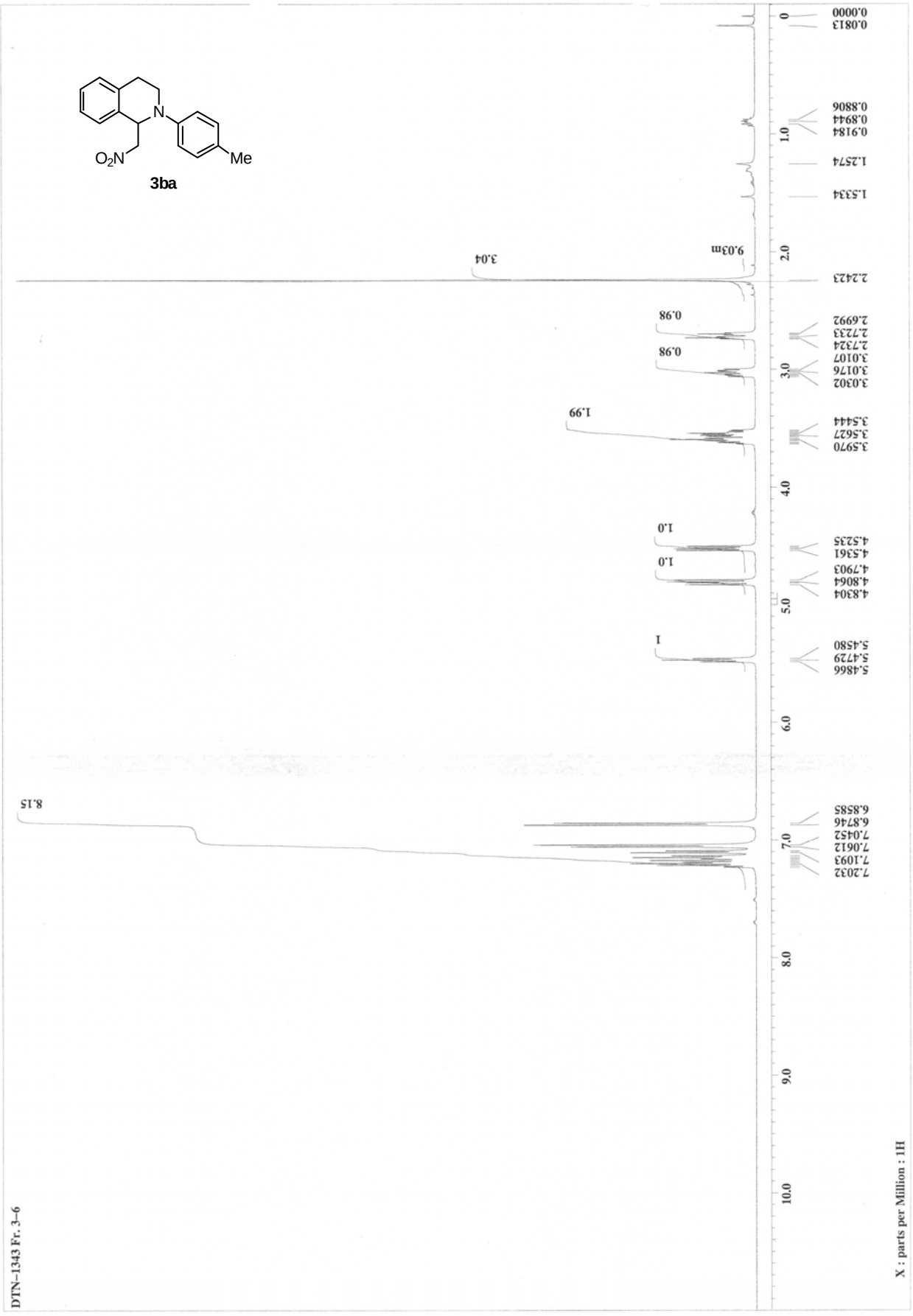
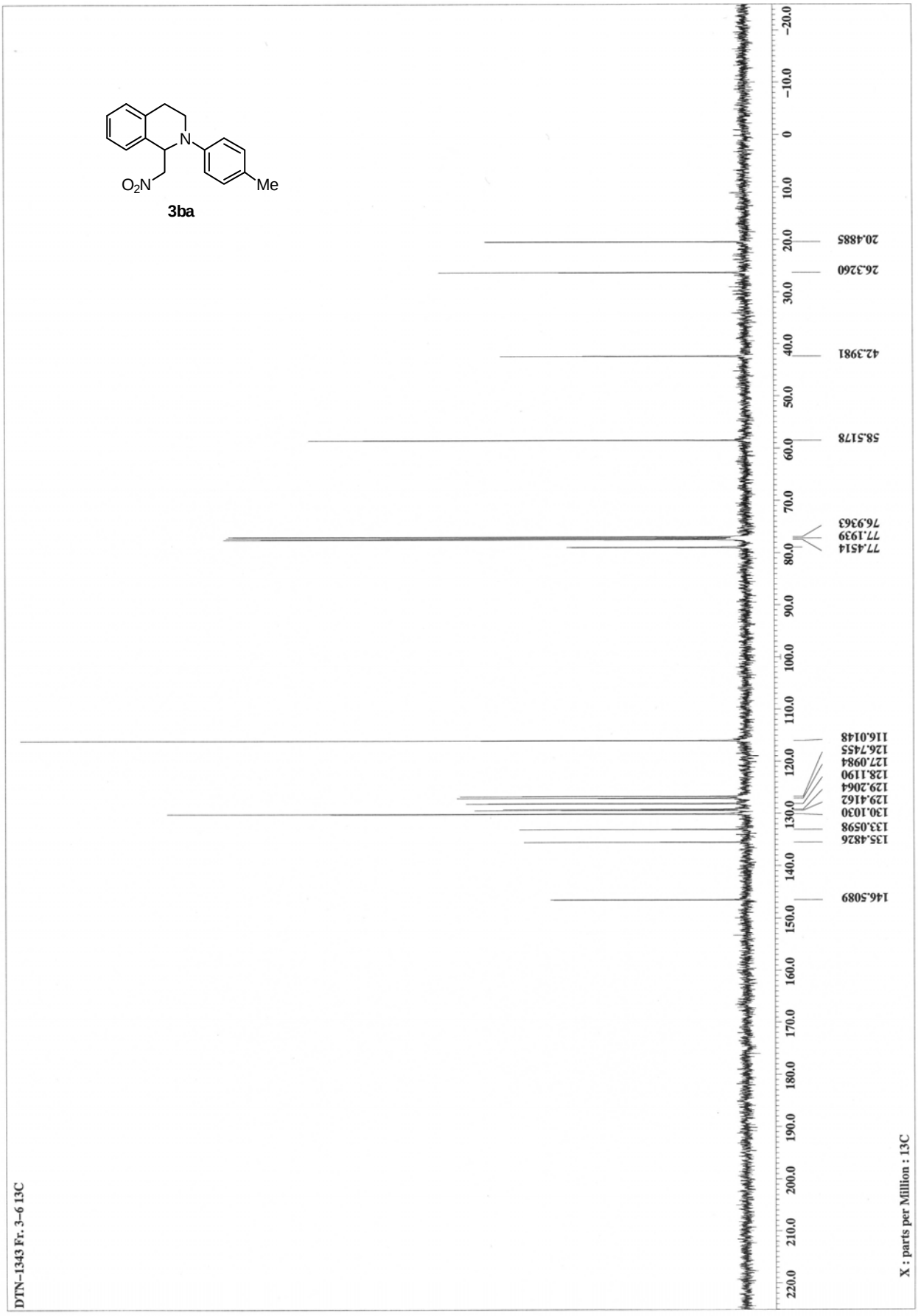
42.1691
58.3175
76.9173
77.1653
77.4228
115.2136
119.5345
126.8122
127.1175
128.2430
129.3208
129.6260
133.0312
135.4063
148.5405


O2N
Me
3ba
DTN-1343 Fr. 3-6
X : parts per Million : 1H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N
Me
3ba
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X : parts per Million : 13C
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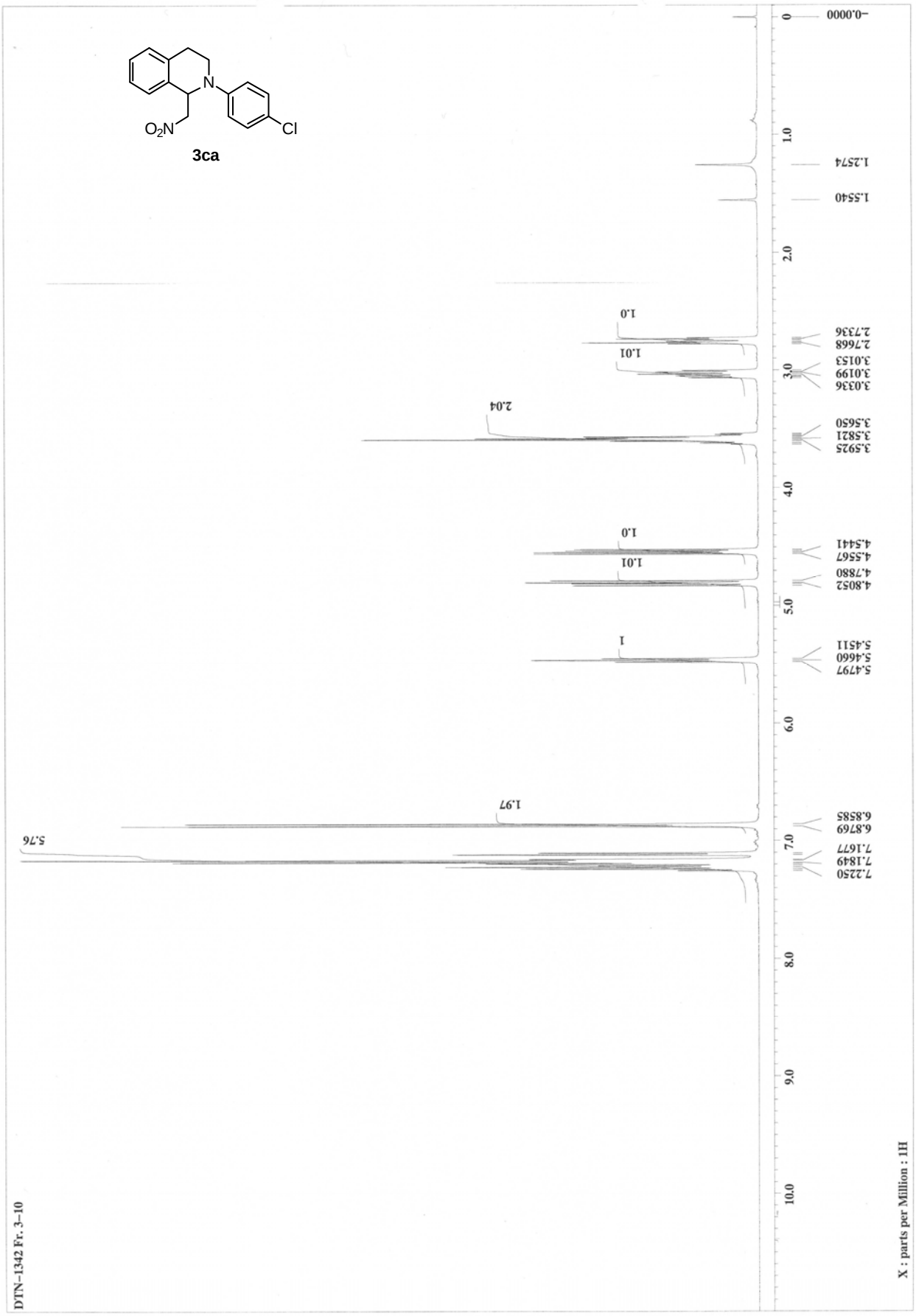
42.3981
58.5178
76.9363
77.1939
77.4514
116.0148
126.7455
127.0984
128.1190
129.2064
129.4162
130.1030
133.0598
135.4826
146.5089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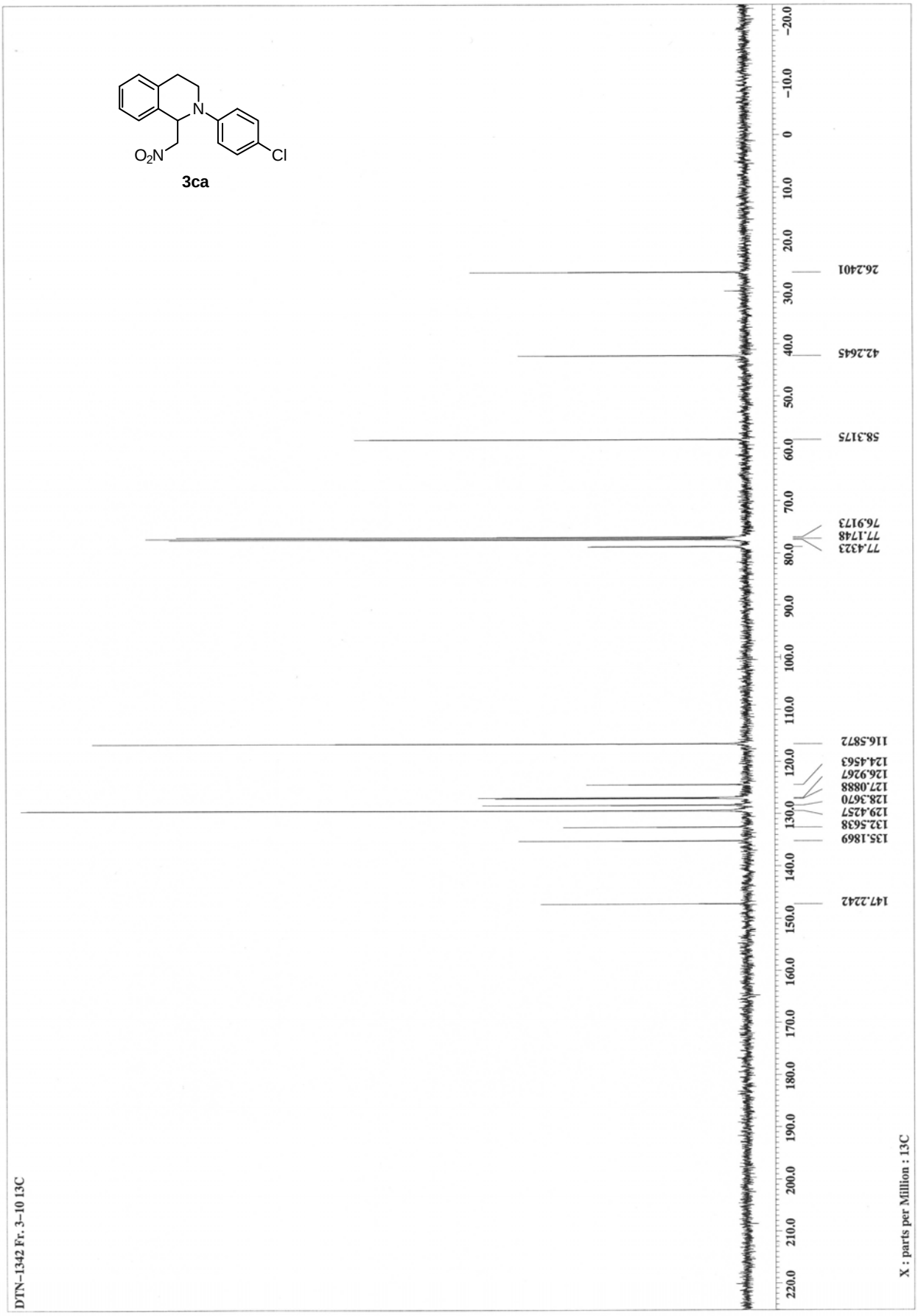
Cl
3ca
DTN-1342 Fr. 3-10
X : parts per Million : 1H


O2N
N
Cl
3ca
DTN-1342 Fr. 3-10 13C
X : parts per Million : 13C
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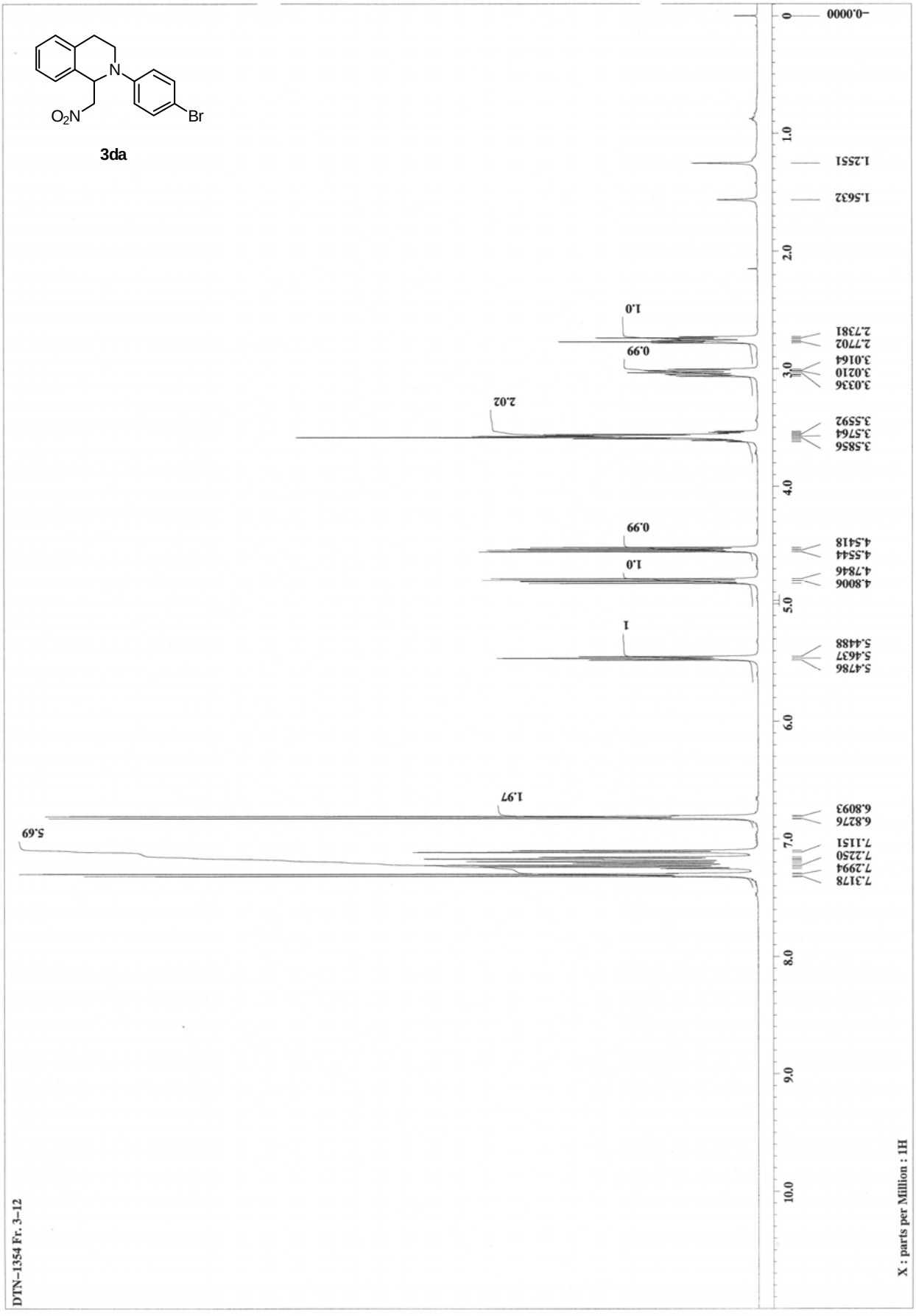
42.2645
58.3175
76.9173
77.1748
77.4323
116.5872
124.4563
126.9267
127.0888
128.3670
129.4257
132.5638
135.1869
147.2242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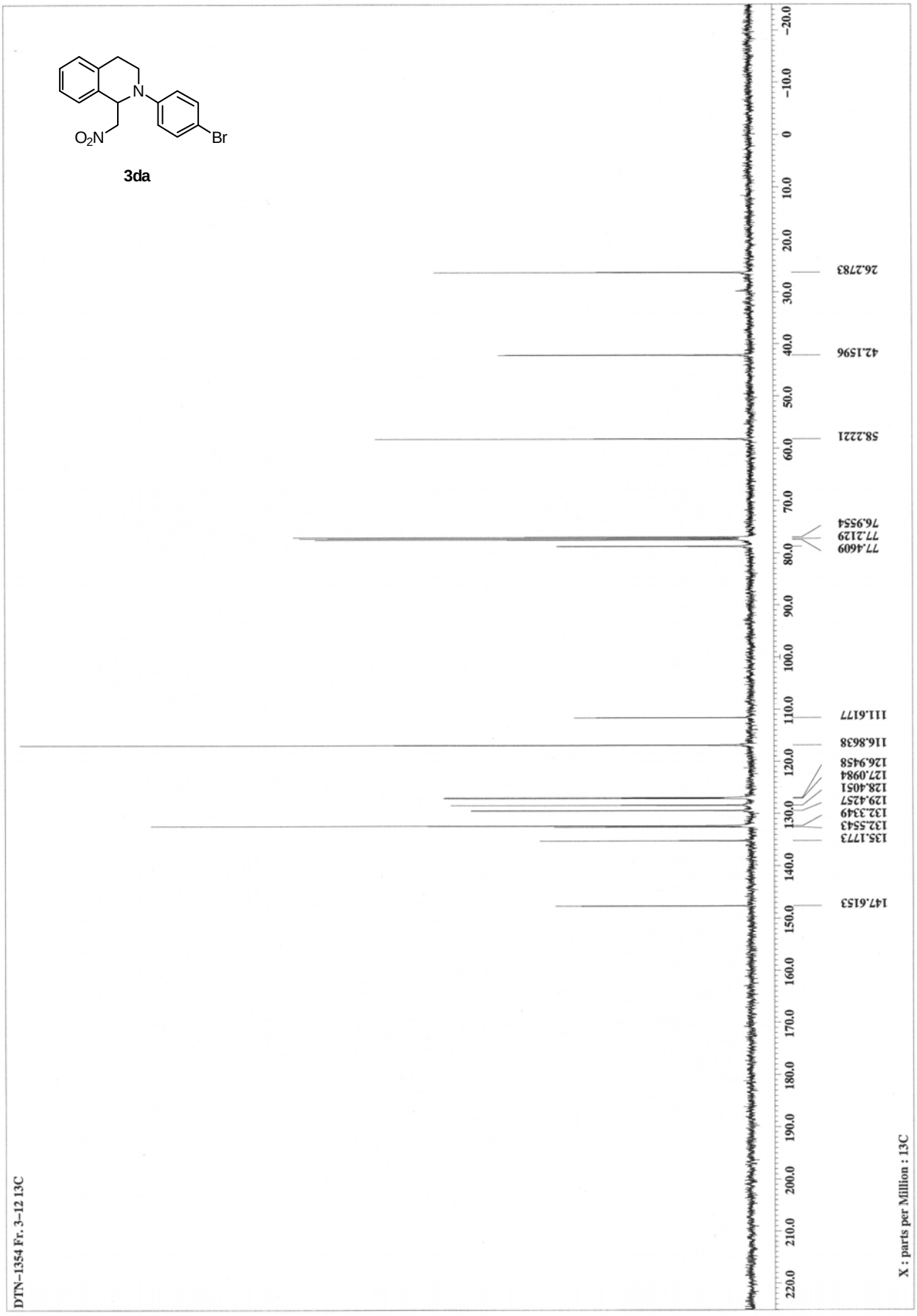
X : parts per Million : 1H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Br
3da
DTN-1354 Fr. 3-12 13C
X : parts per Million : 13C
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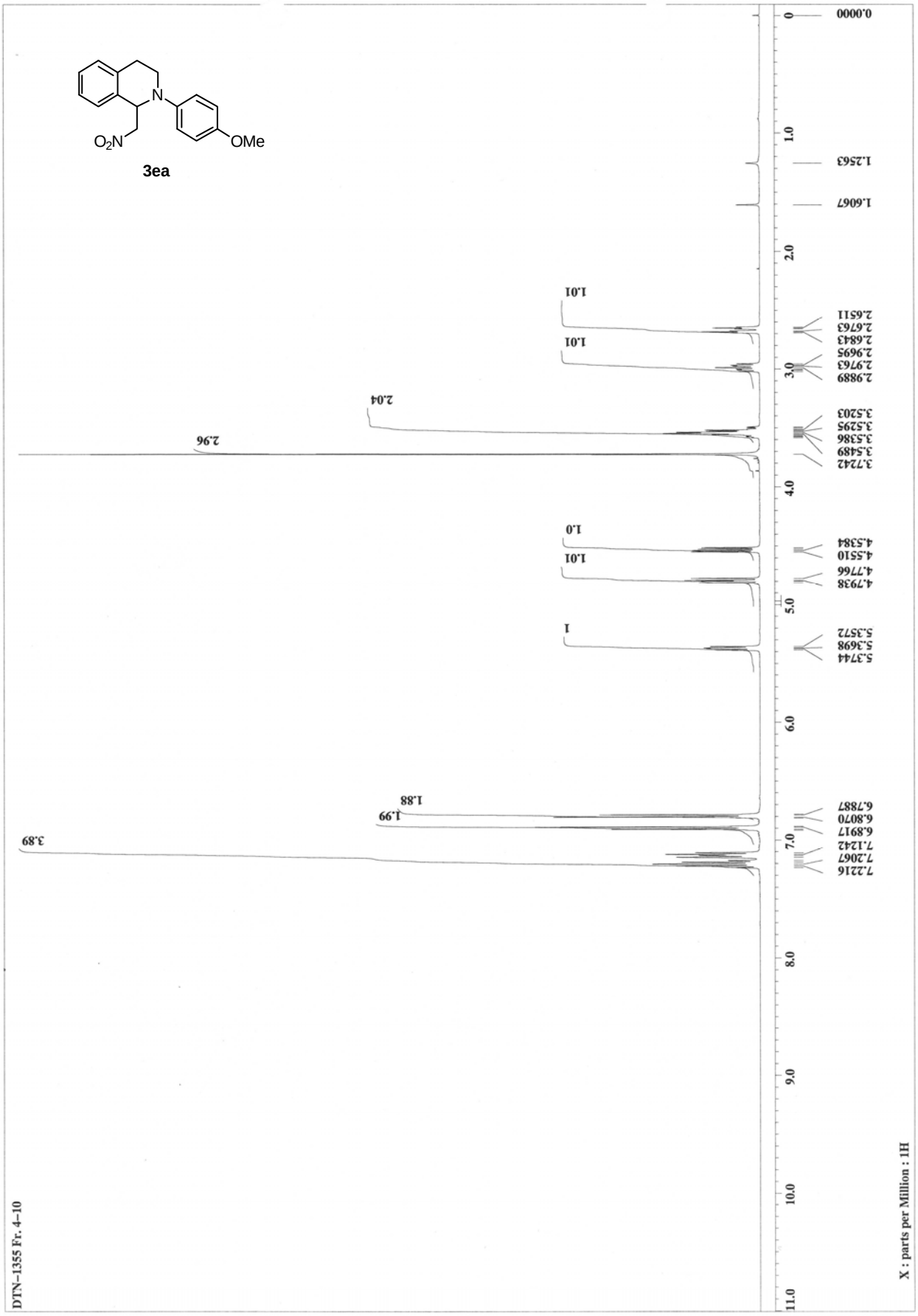
147.6153


O2N
N
OMe
3ea
DTN-1355 Fr. 4-10
X : parts per Million : 1H
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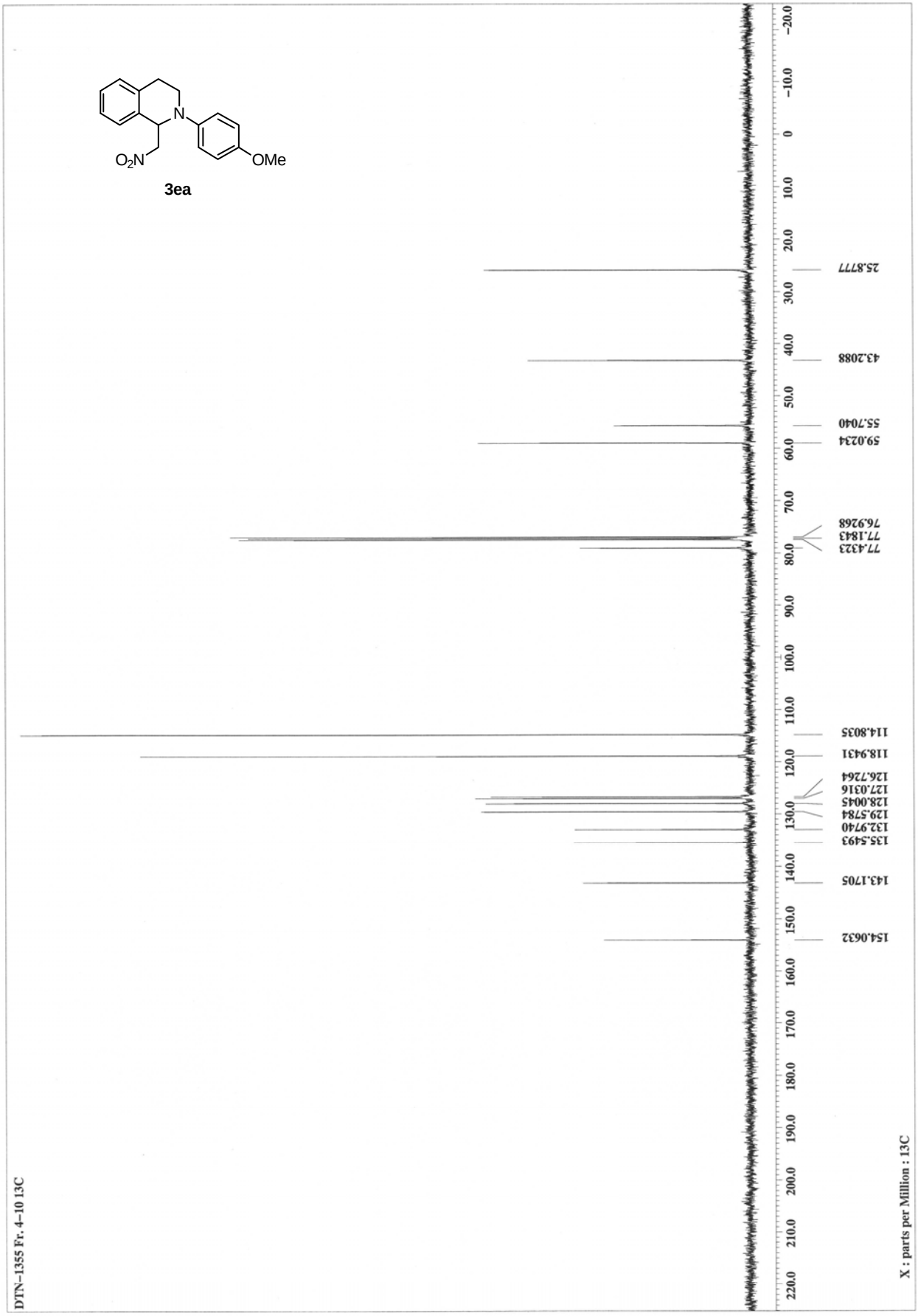
1.6067
2.6511
2.6763
2.6843
2.9695
2.9763
2.9889
3.5203
3.5295
3.5386
3.5489
3.7242
4.5384
4.5510
4.7766
4.7938
5.3572
5.3698
5.3744
6.7887
6.8070
6.8917
7.1242
7.2067
7.2216
1.01
1.01
2.04
2.96
1.0
1.01
1
1.99
1.88
3.89


O2N
OMe
3ea
DTN-1355 Fr. 4-10 13C
X : parts per Million : 13C
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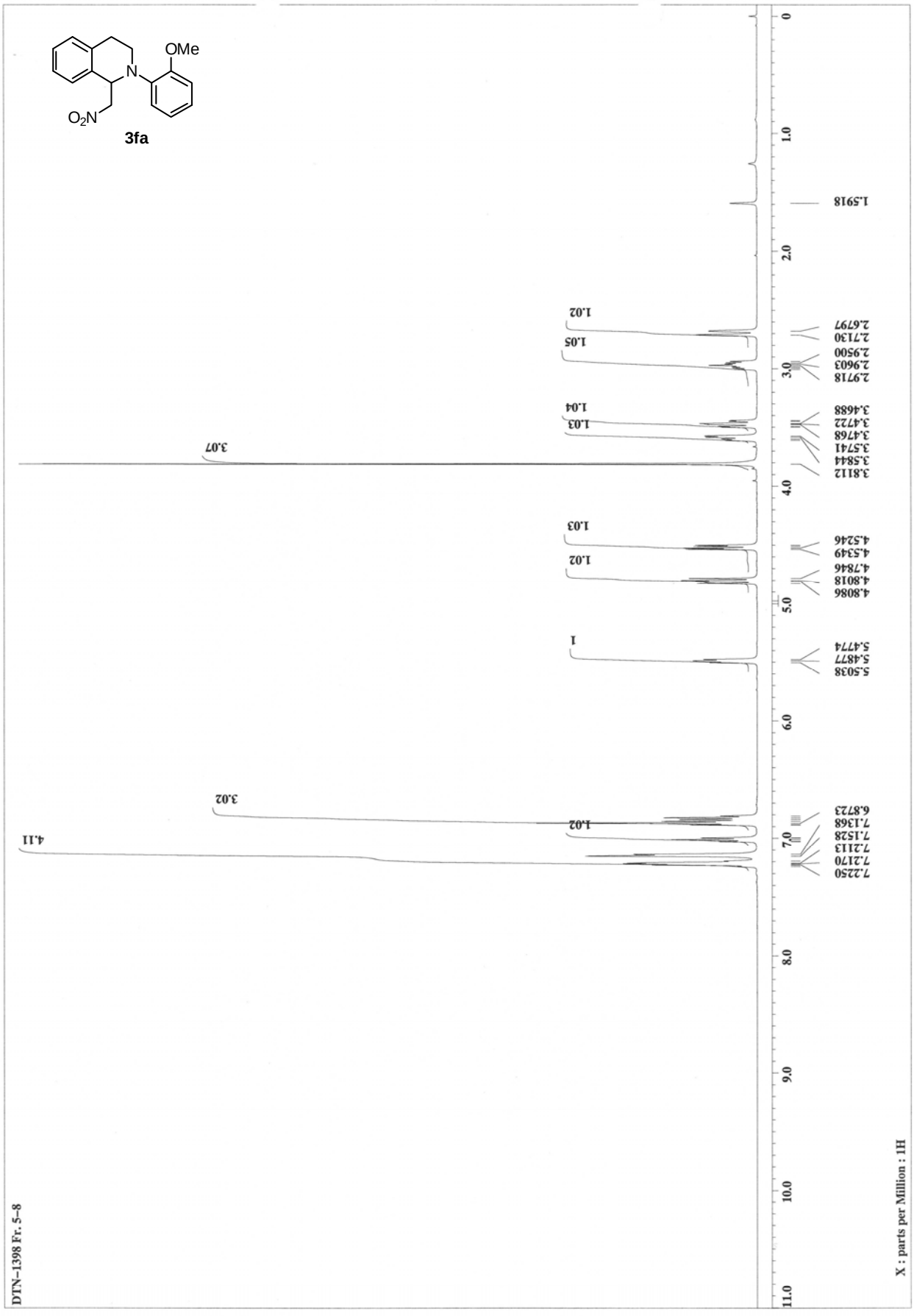
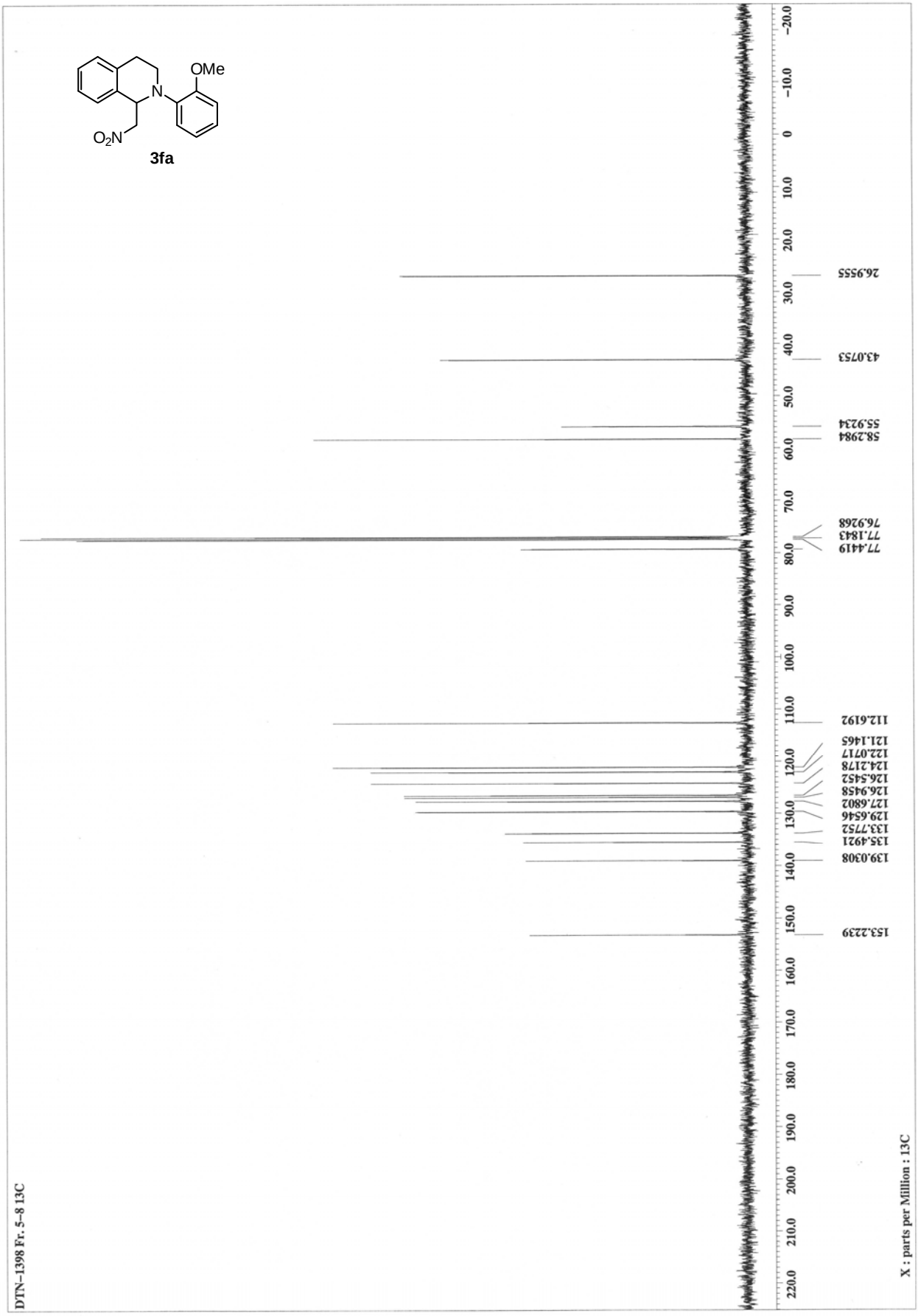
55.7040
59.0234
76.9268
77.1843
77.4323
114.8035
118.9431
126.7264
127.0316
128.0045
129.5784
132.9740
135.5493
143.1705
154.0632


OMe
N
O_2N
3fa
DTN-1398 Fr. 5-8
X : parts per Million : 1H


OMe
N
O_2N
3fa
DTN-1398 Fr. 5-8 13C
X : parts per Million : 13C
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55.9234
58.2984
76.9268
77.1843
77.4419
112.6192
121.1465
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126.5452
126.9458
127.6802
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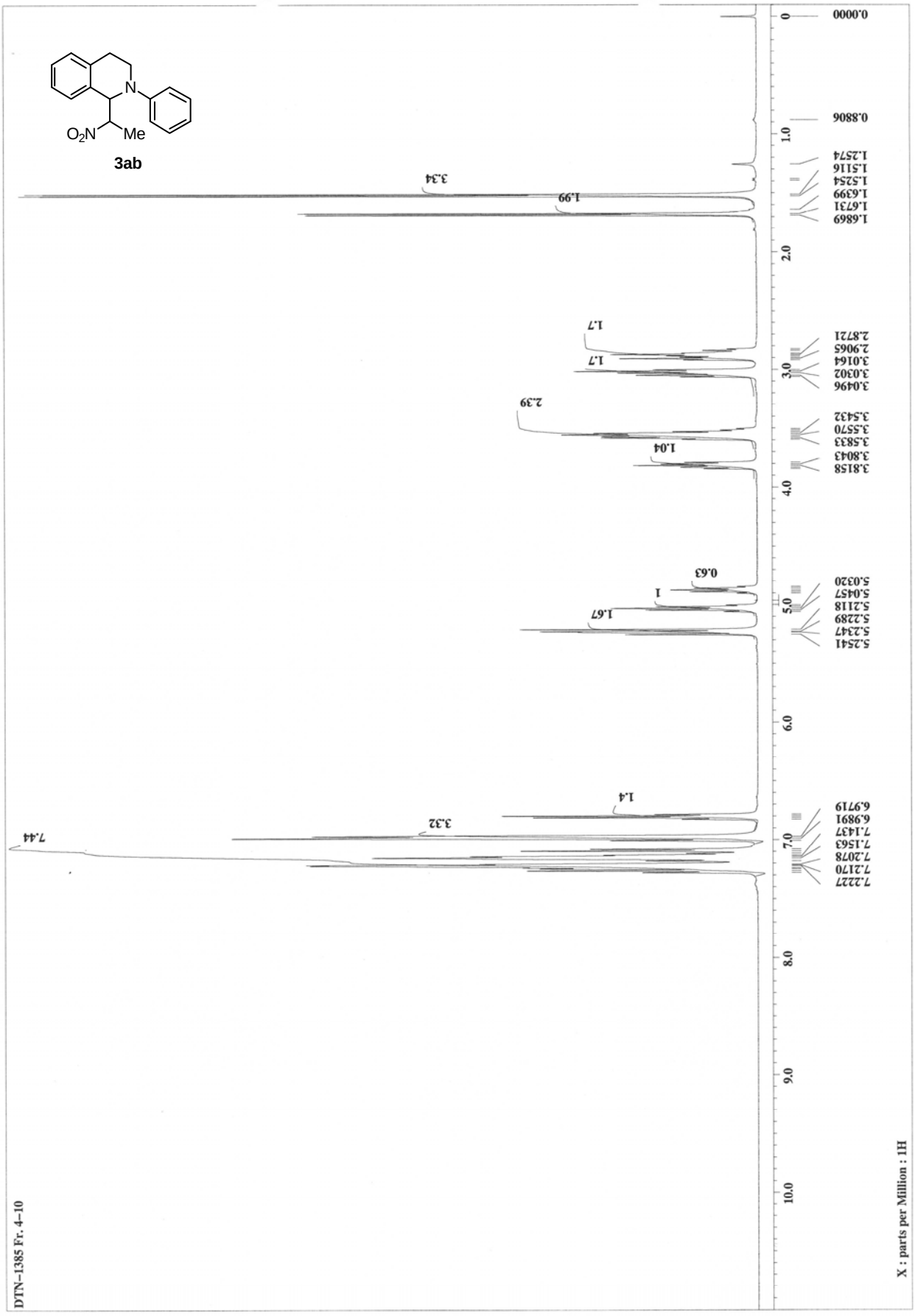
133.7752
135.4921
139.0308
153.2239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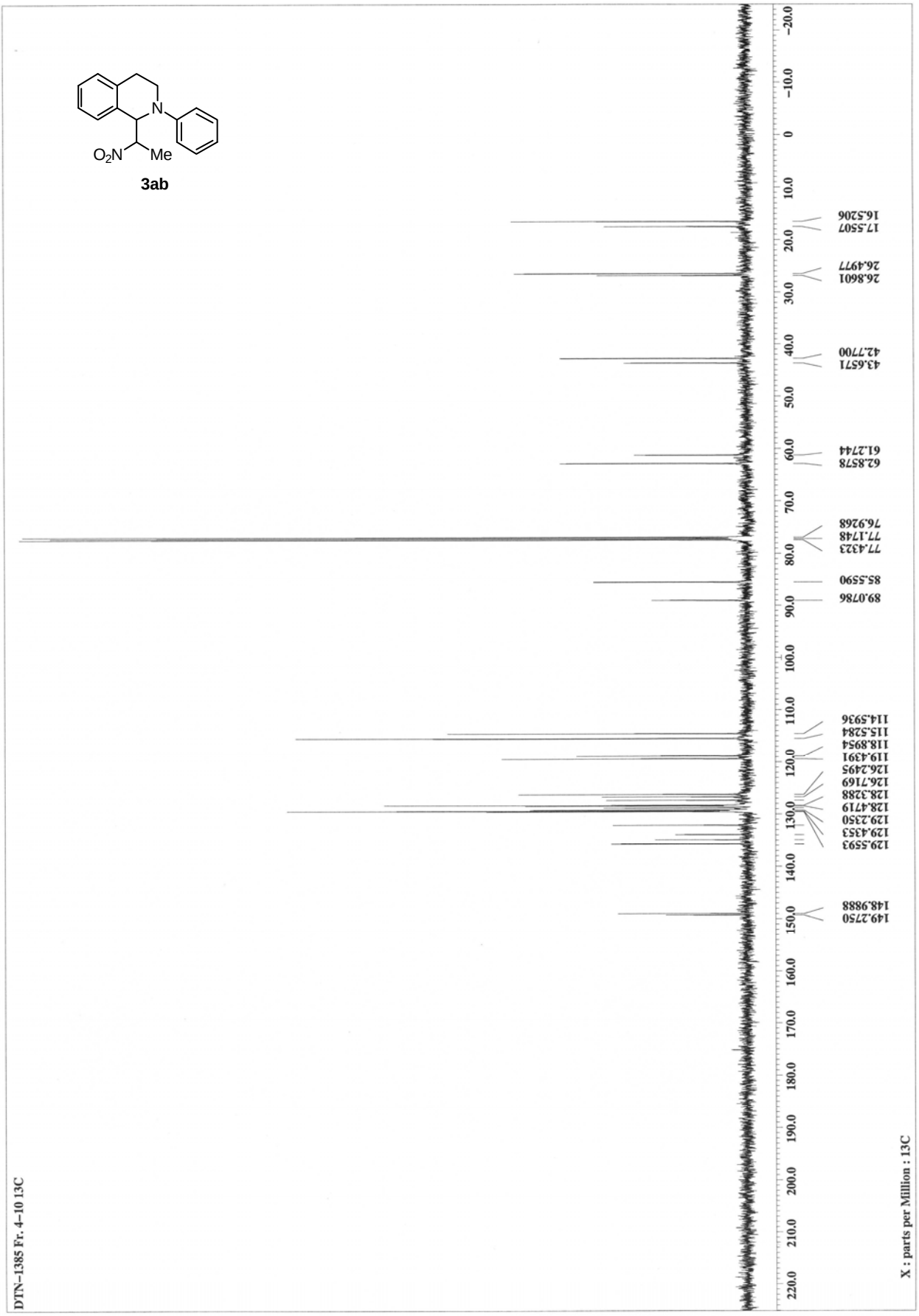
DTN-1385 Fr. 4-10
X : parts per Million : 1H


O2N
Me
N
3ab
DTN-1385 Fr. 4-10 13C
X : parts per Million : 13C
16.5206
17.5507
26.8601
26.4977
43.6571
42.7700
62.8578
61.2744
77.4323
77.1748
76.9268
89.0786
85.5590
114.5936
115.5284
118.8954
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129.2350
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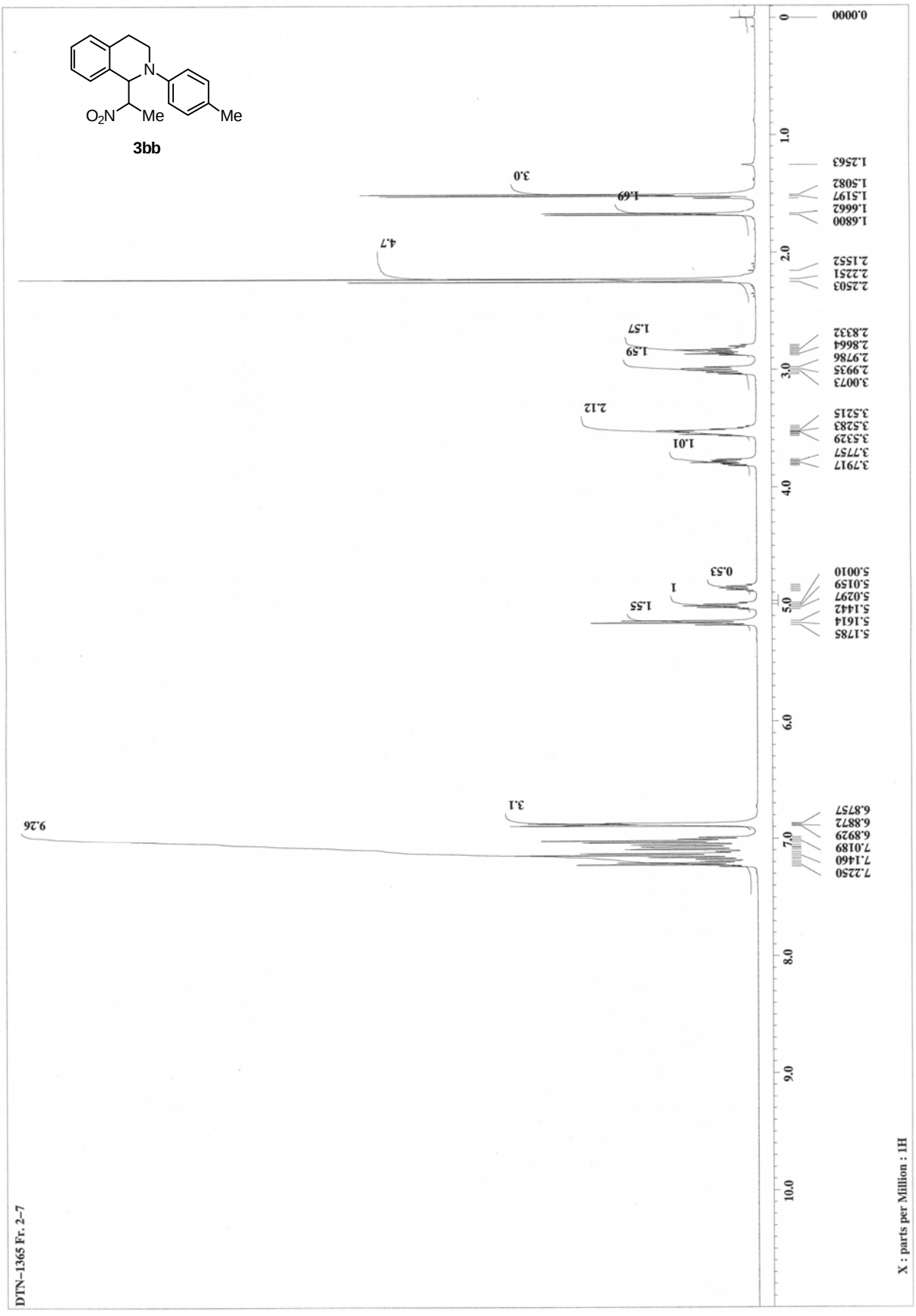
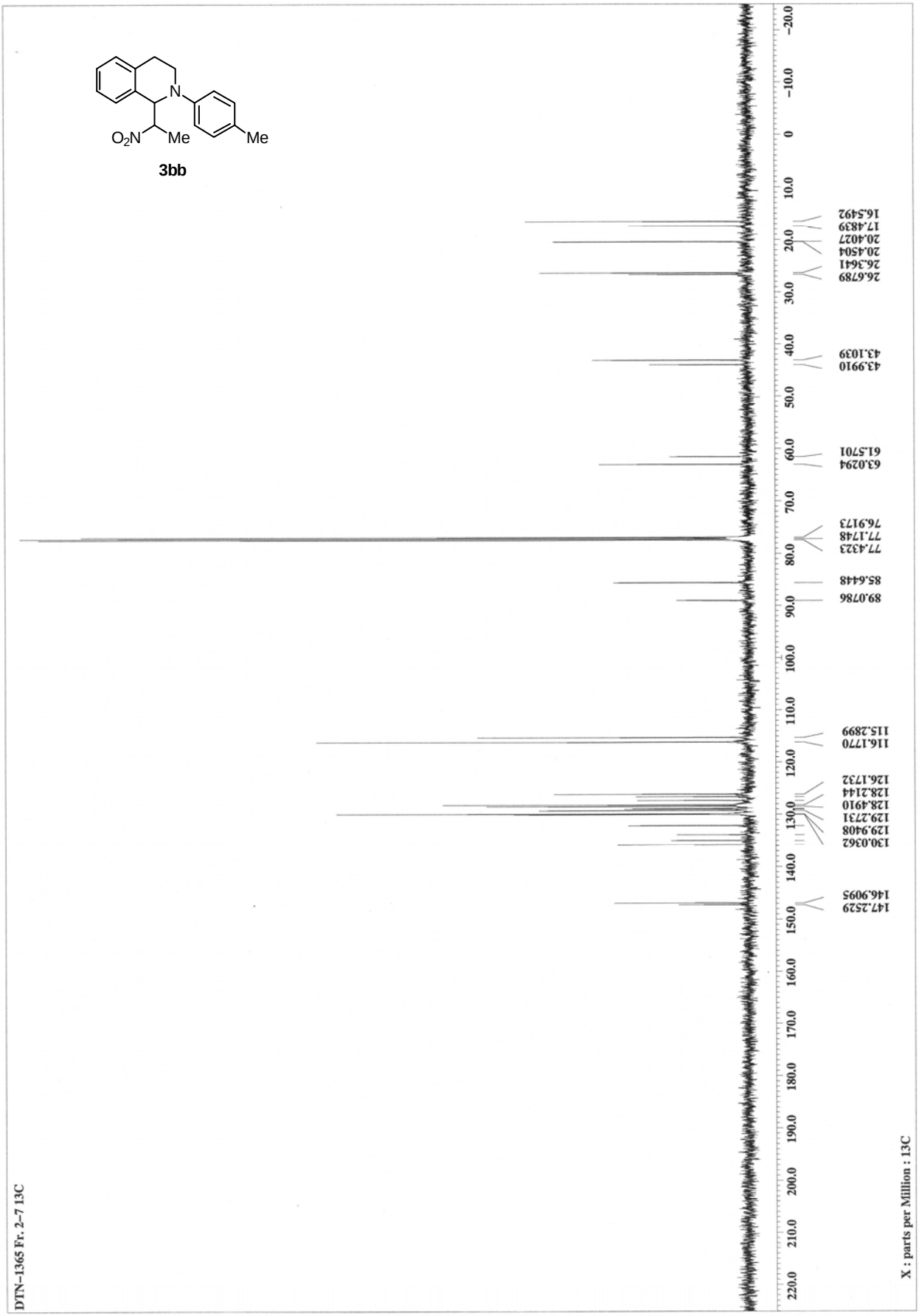
149.2750


O2N
Me
Me
3bb
DTN-1365 Fr. 2-7
X : parts per Million : 1H


N
O2N
Me
Me
3bb
DTN-1365 Fr. 2-7 13C
X : parts per Million : 13C
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20.4027
20.4504
26.3641
26.6789
43.1039
43.9910
61.5701
63.0294
76.9173
77.1748
77.4323
85.6448
89.0786
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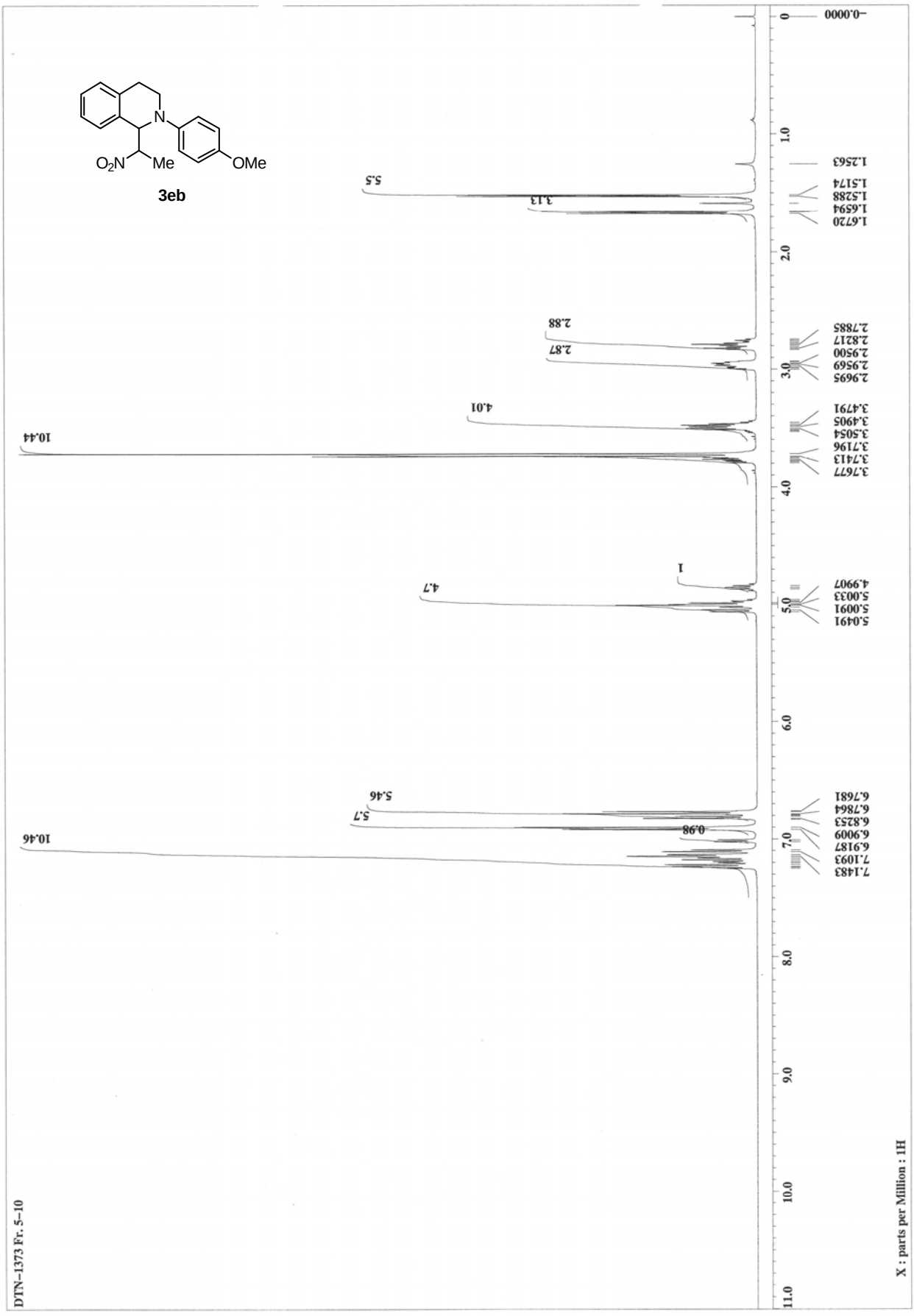
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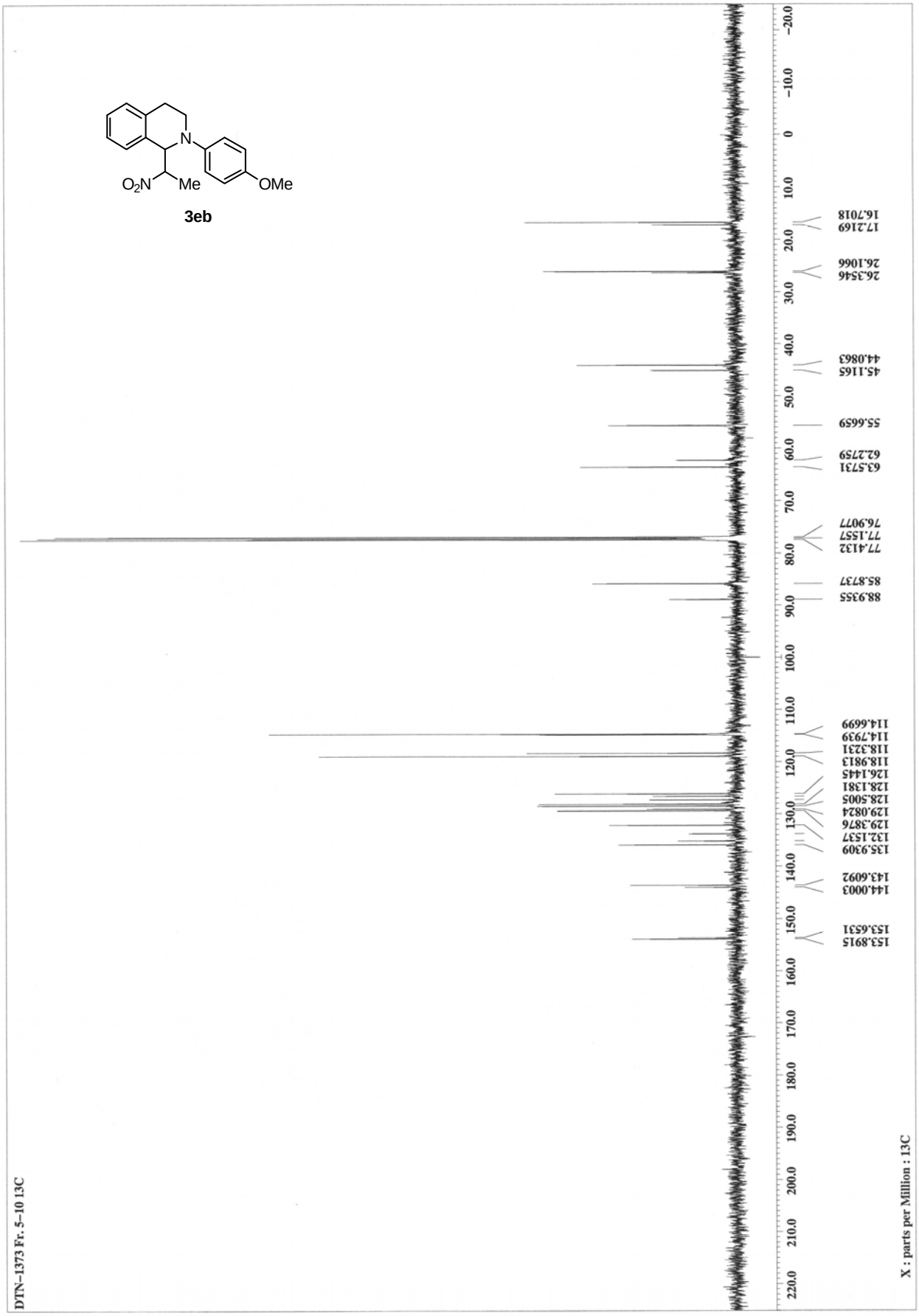
Me
OMe
3eb
DTN-1373 Fr. 5-10
X : parts per Million : 1H


O2N
Me
N
OMe
3eb
DTN-1373 Fr. 5-10 13C
X : parts per Million : 13C
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26.3546
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44.0863
45.1165
55.6659
62.2759
63.5731
76.9077
77.1557
77.4132
85.8737
88.9355
114.6699
114.7939
118.3231
118.9813
126.1445
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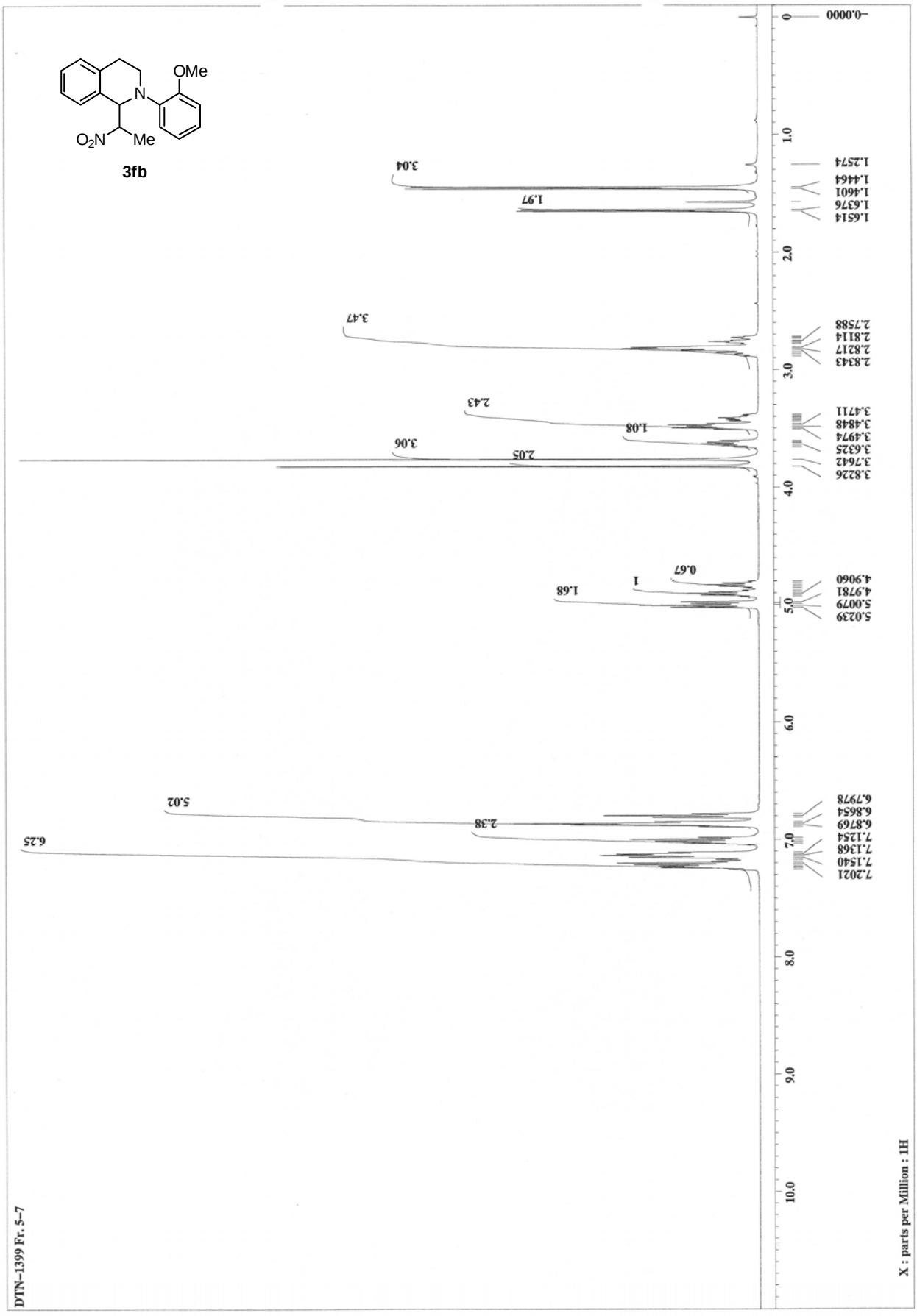
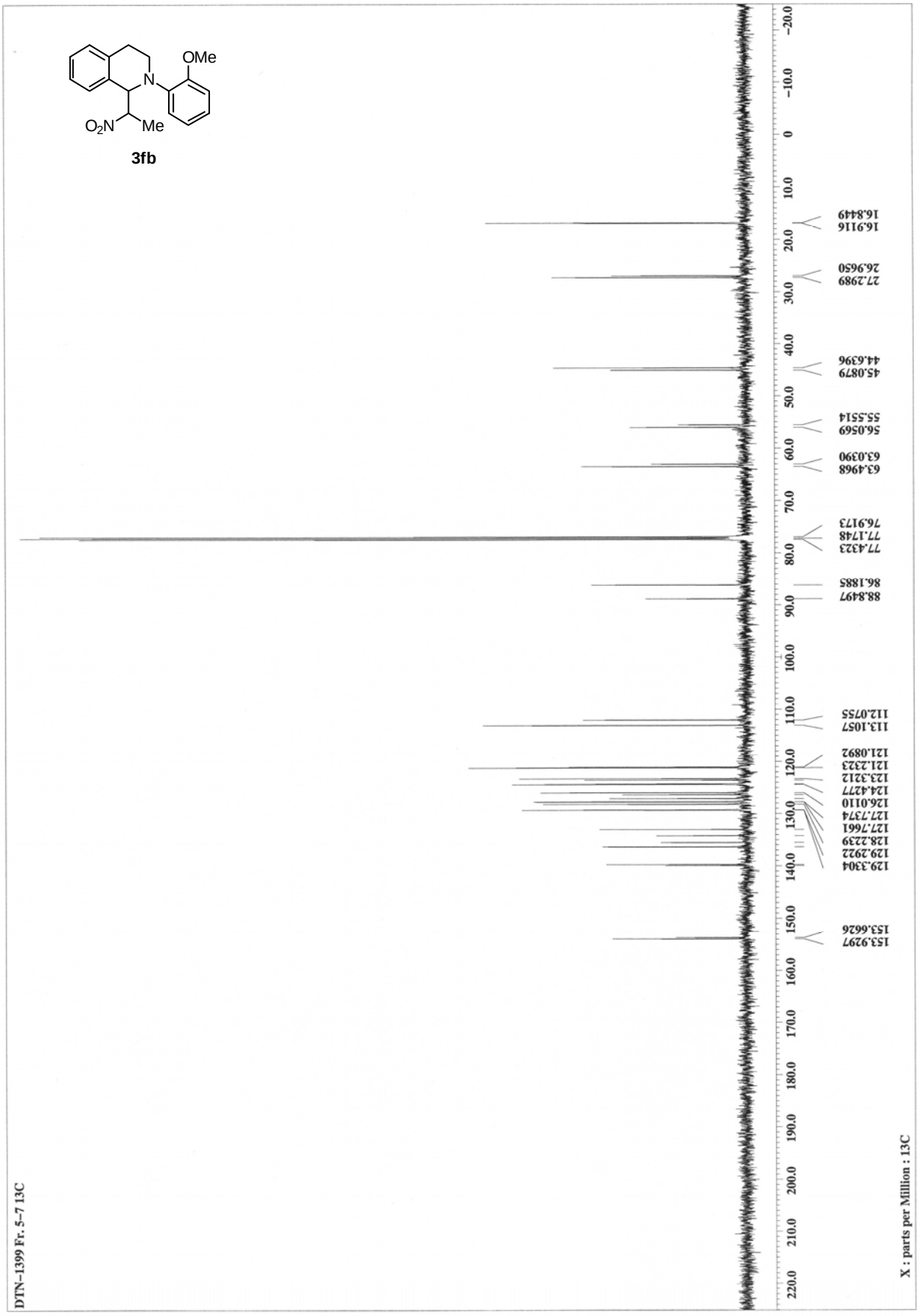
153.6531
153.8915


OMe
N
O2N
Me
3fb
DTN-1399 Fr. 5-7
X : parts per Million : 1H


OMe
N
O2N
Me
3fb
DTN-1399 Fr. 5-7 13C
X : parts per Million : 13C
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26.9650
27.2989
44.6396
45.0879
55.5514
56.0569
63.0390
63.4968
76.9173
77.1748
77.4323
86.1885
88.8497
112.0755
113.1057
121.0892
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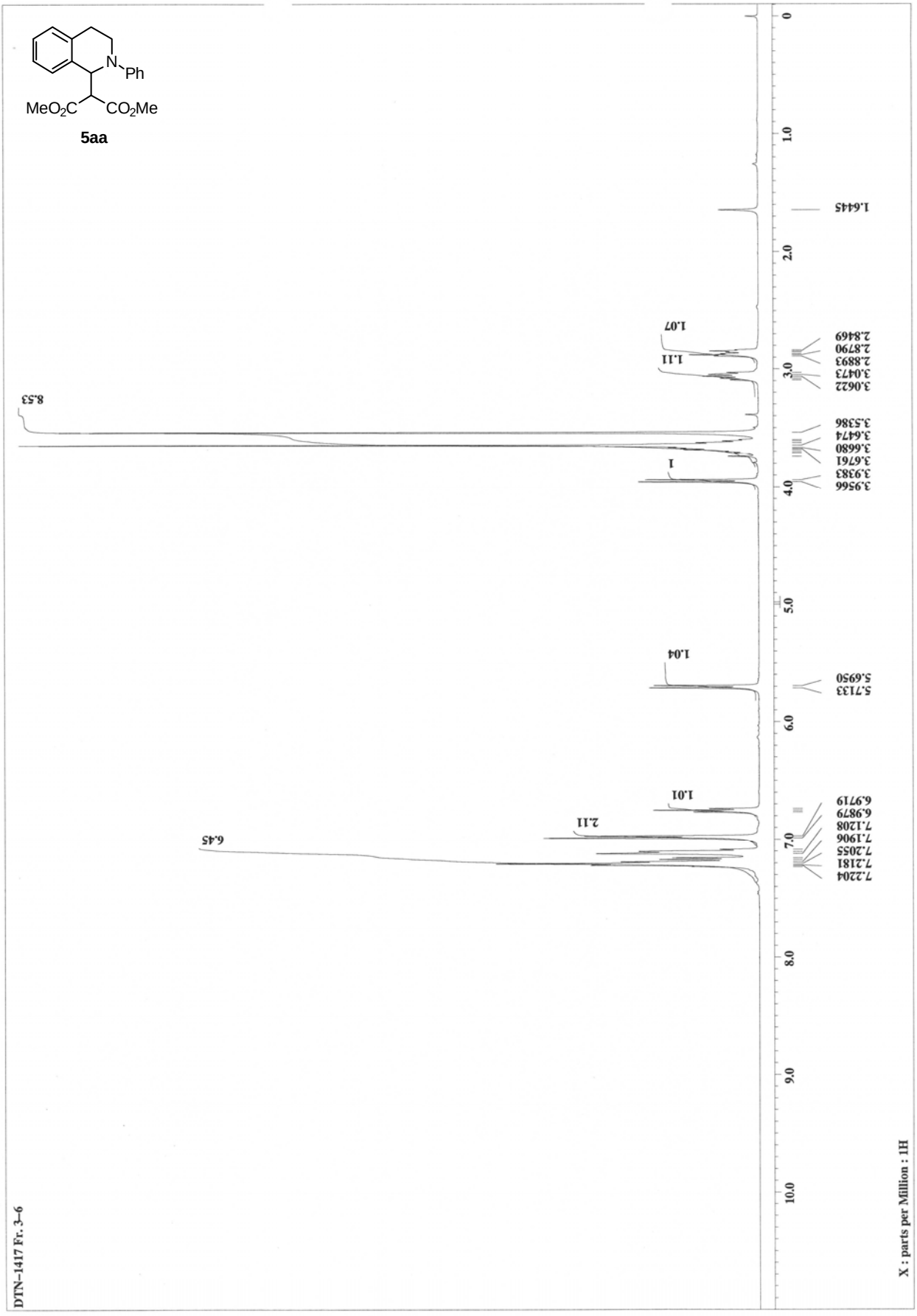
N
Ph
MeO2C
CO2Me
5aa
DTN-1417 Fr. 3-6
X : parts per Million : 1H
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1.07
1.11
8.53
1
1.04
1.01
2.11
6.45
3.0622
3.0473
2.8893
2.8790
2.8469
3.5386
3.6474
3.6680
3.6761
3.9383
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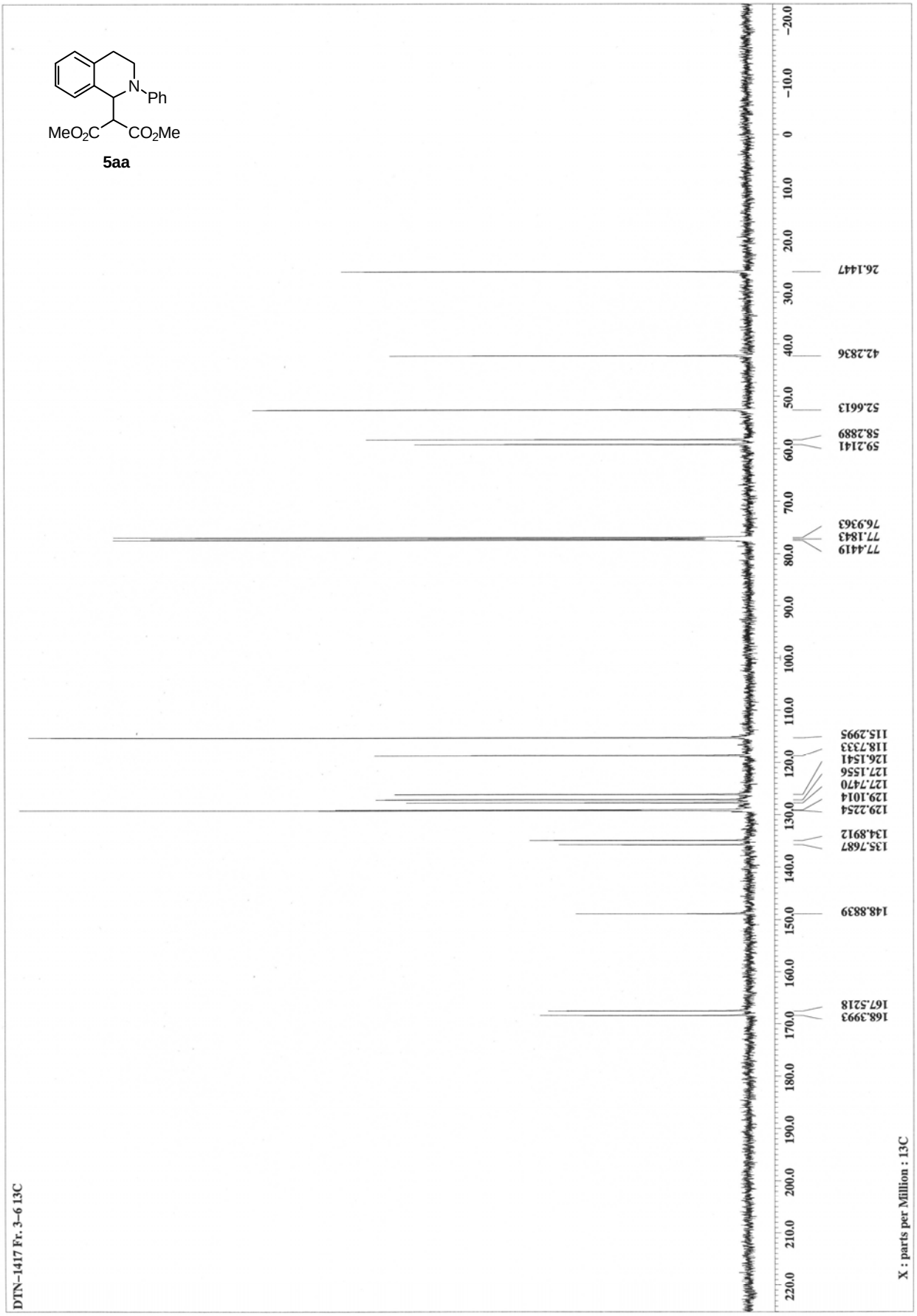
N
Ph
MeO2C
CO2Me
5aa
DTN-1417 Fr. 3-6 13C
X : parts per Million : 13C
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52.6613
58.2889
59.2141
76.9363
77.1843
77.4419
115.2995
118.7333
126.1541
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127.7470
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129.2254
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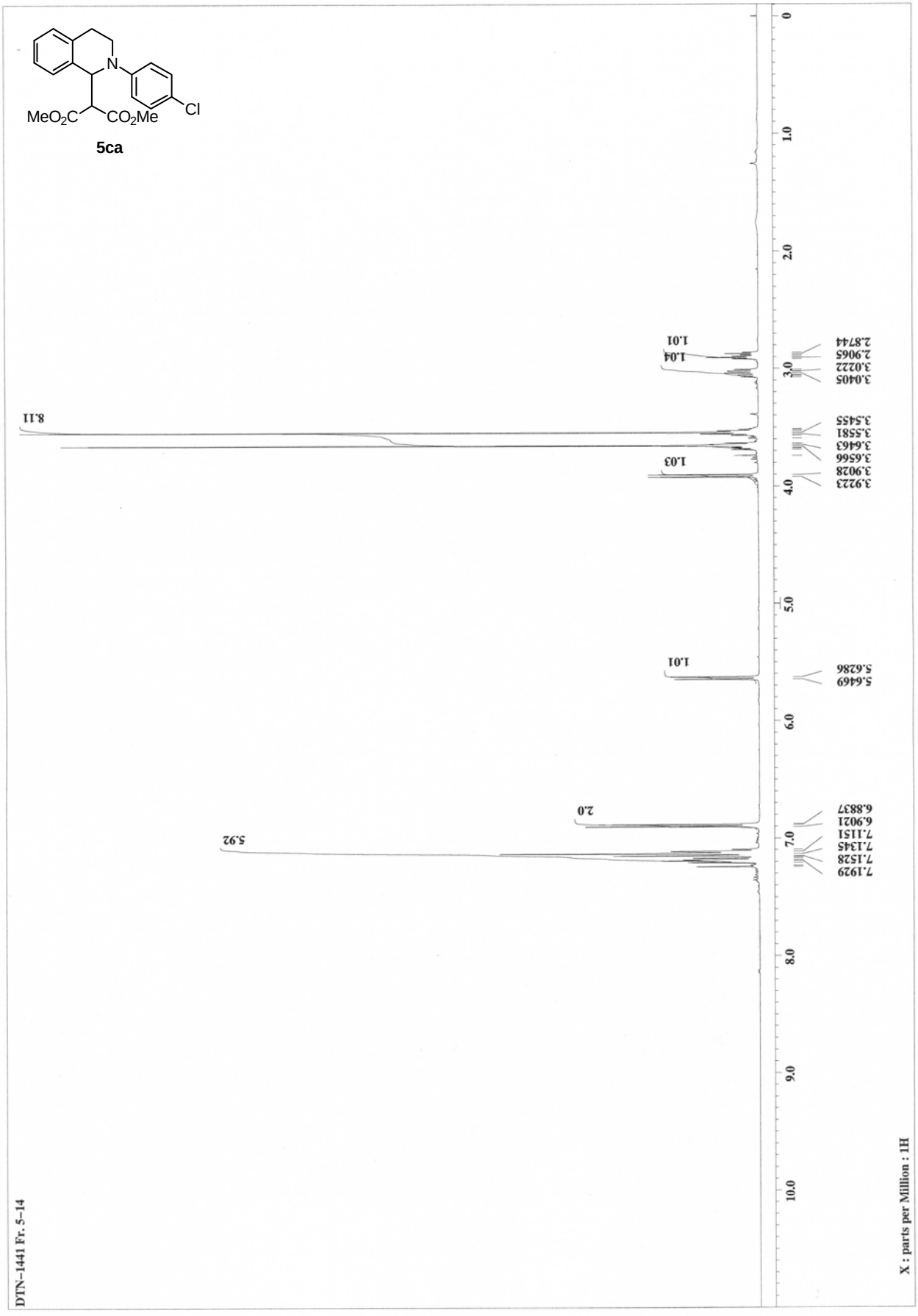
167.5218
168.3993


MeO2C
CO2Me
Cl
N
5ca
DTN-1441 Fr. 5-14
X : parts per Million : 1H
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7.1528
7.1345
7.1151
6.9021
6.8837
5.6469
5.6286
3.9223
3.9028
3.6566
3.6463
3.5581
3.5455
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2.9065
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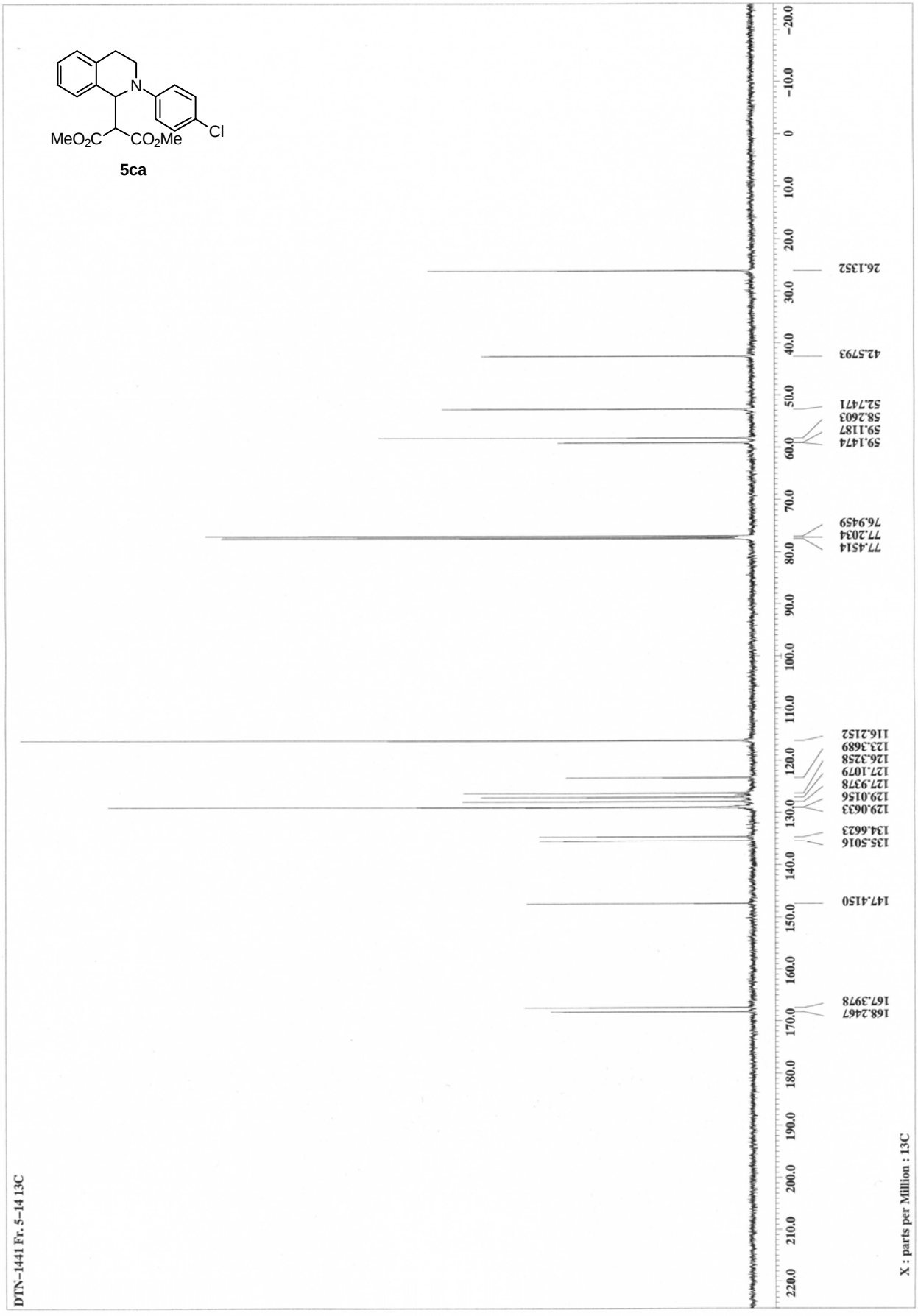
8.11
1.03
1.01
1.04
1.01


N
MeO2C
CO2Me
Cl
5ca
DTN-1441 Fr. 5-14 13C
X : parts per Million : 13C
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42.5793
52.7471
58.2603
59.1187
59.1474
76.9459
77.2034
77.4514
116.2152
123.3689
126.3258
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127.9378
129.0156
129.0633
134.6623
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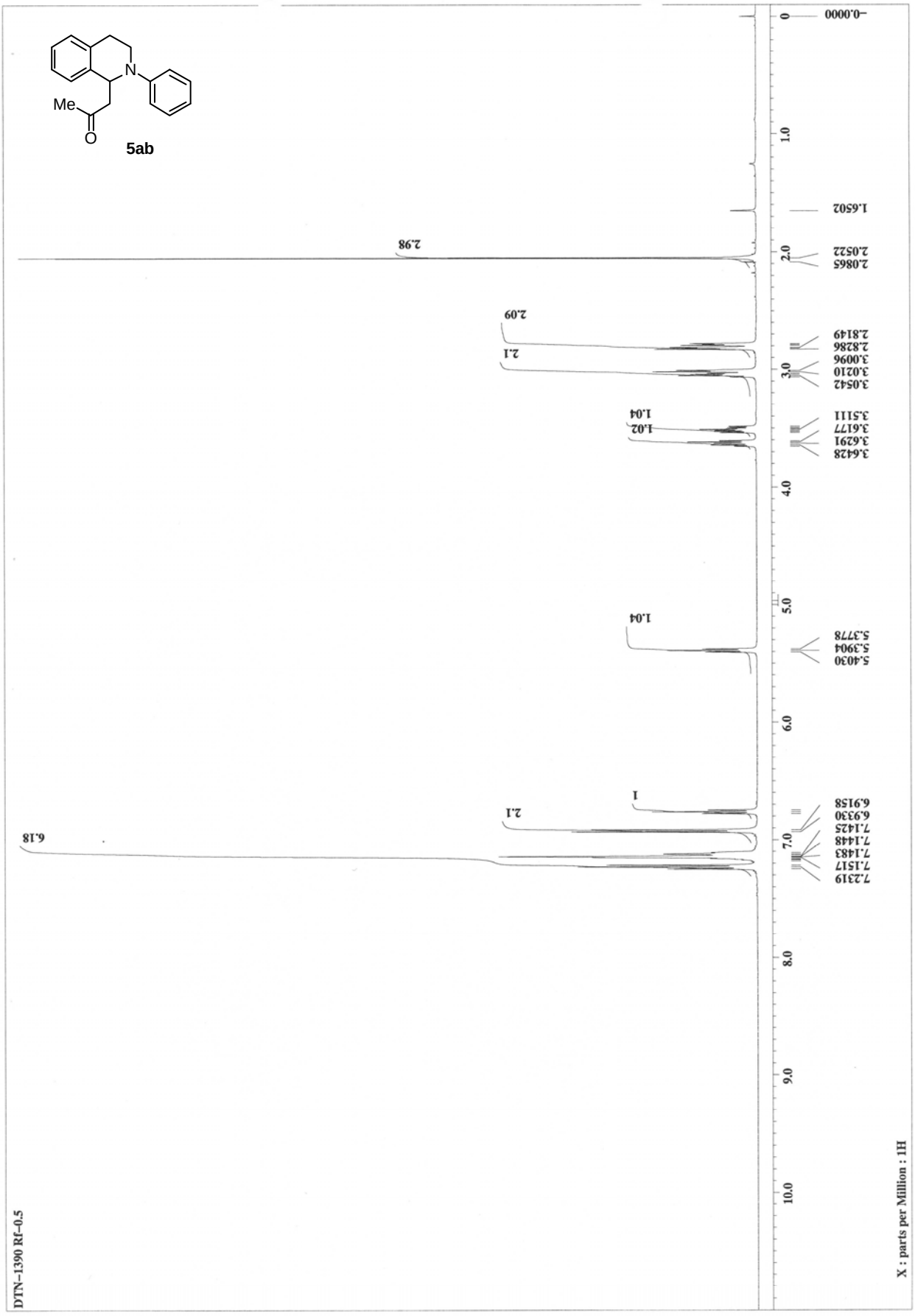
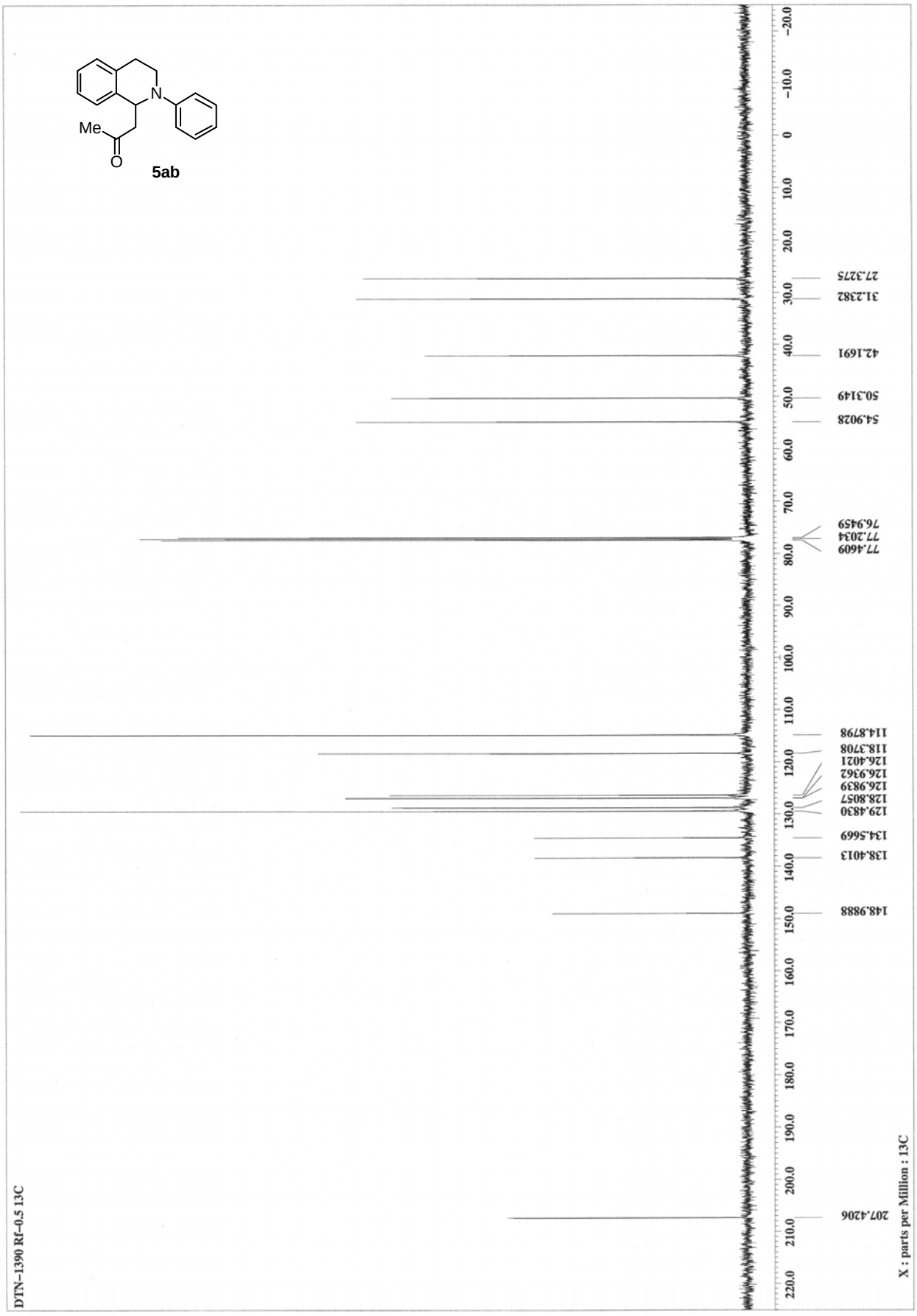
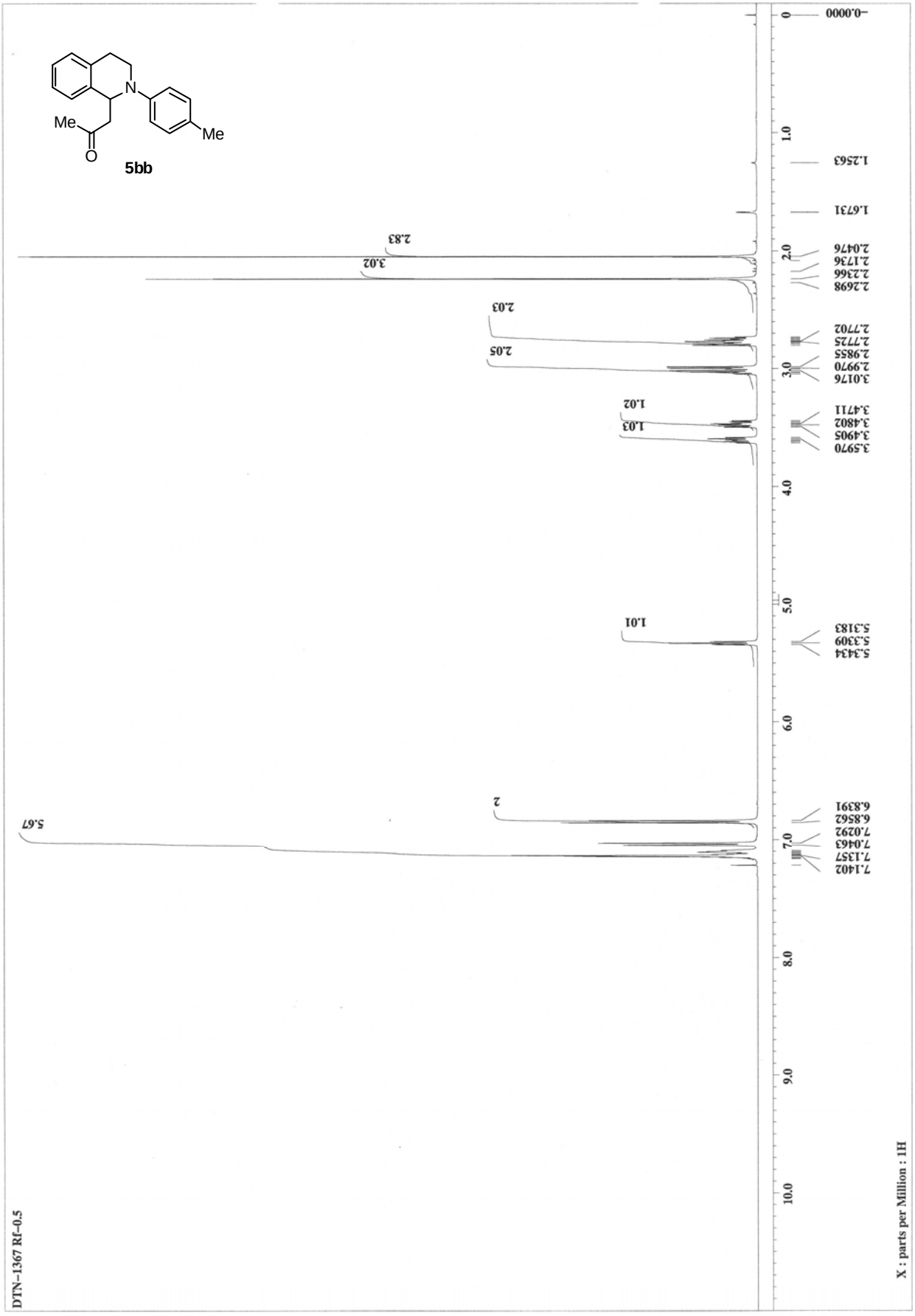
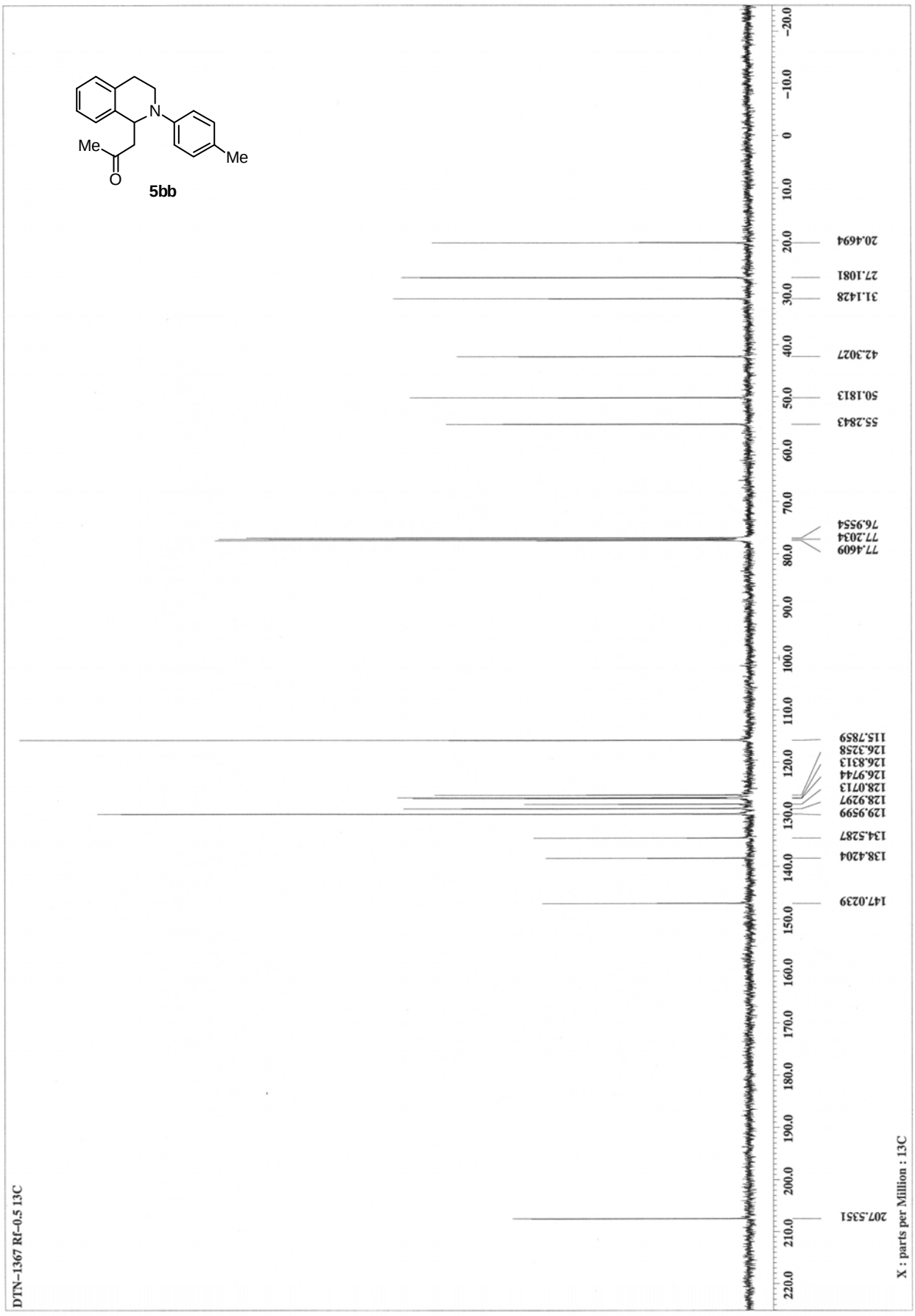
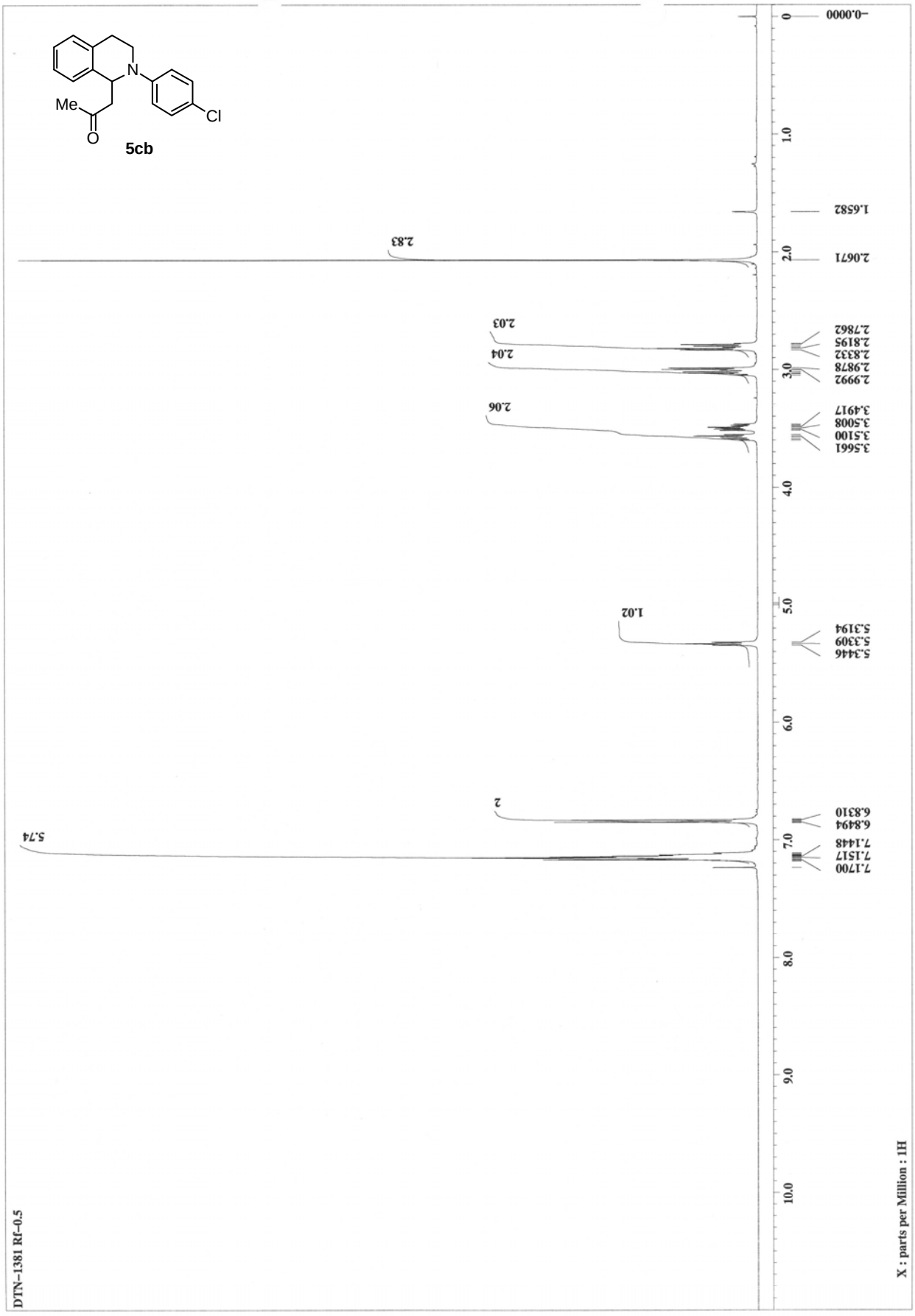
168.2467

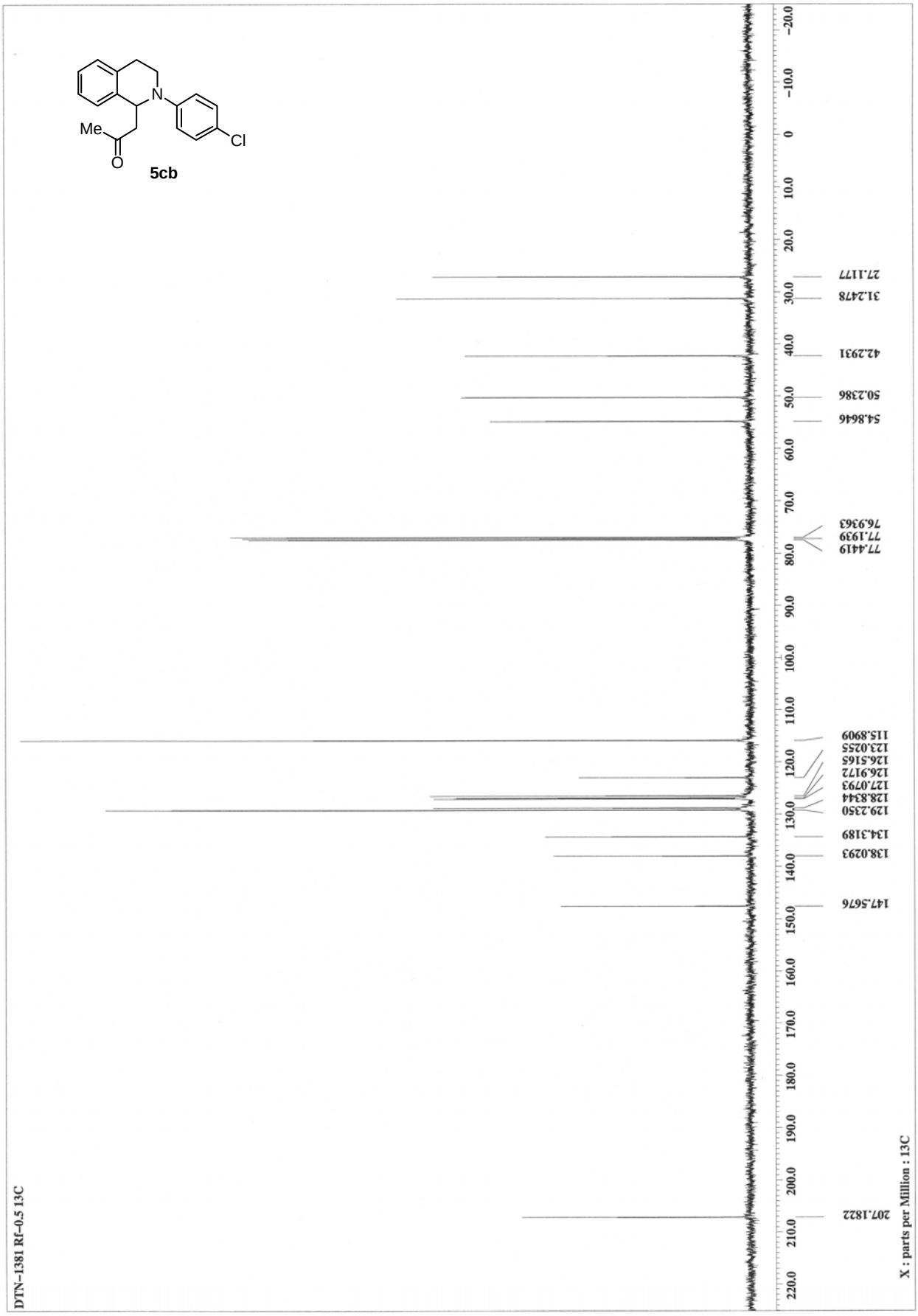

Me
O
N
5ab
DTN-1390 RF-0.5
X : parts per Million : 1H

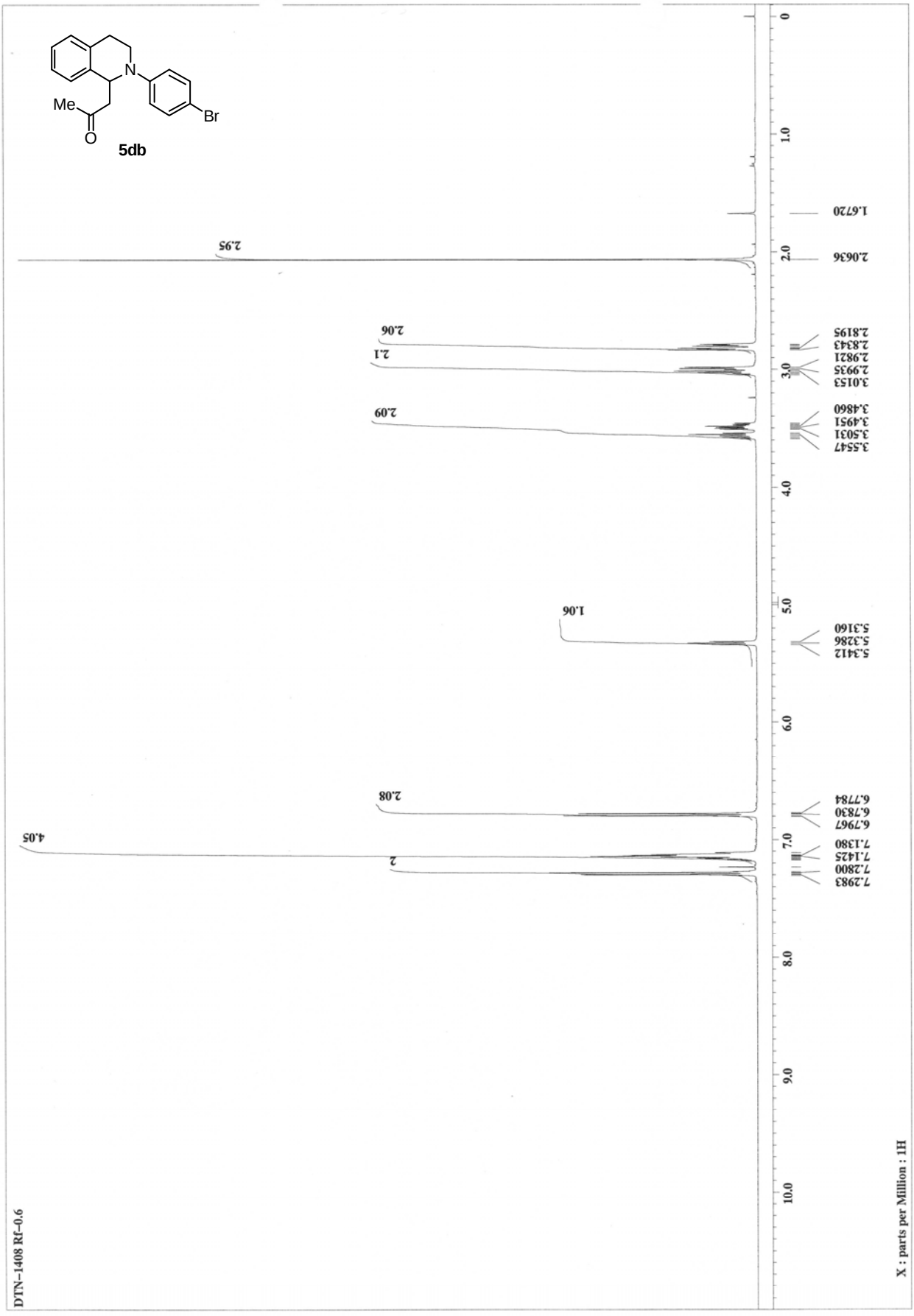

Me
O
N
5ab
DTN-1390 Rf-0.5 13C
X : parts per Million : 13C
27.3275
31.2382
42.1691
50.3149
54.9028
76.9459
77.2034
77.4609
114.8798
118.3708
126.4021
126.9362
126.9839
128.8057
129.4830
134.5669
138.4013
148.9888
207.4206

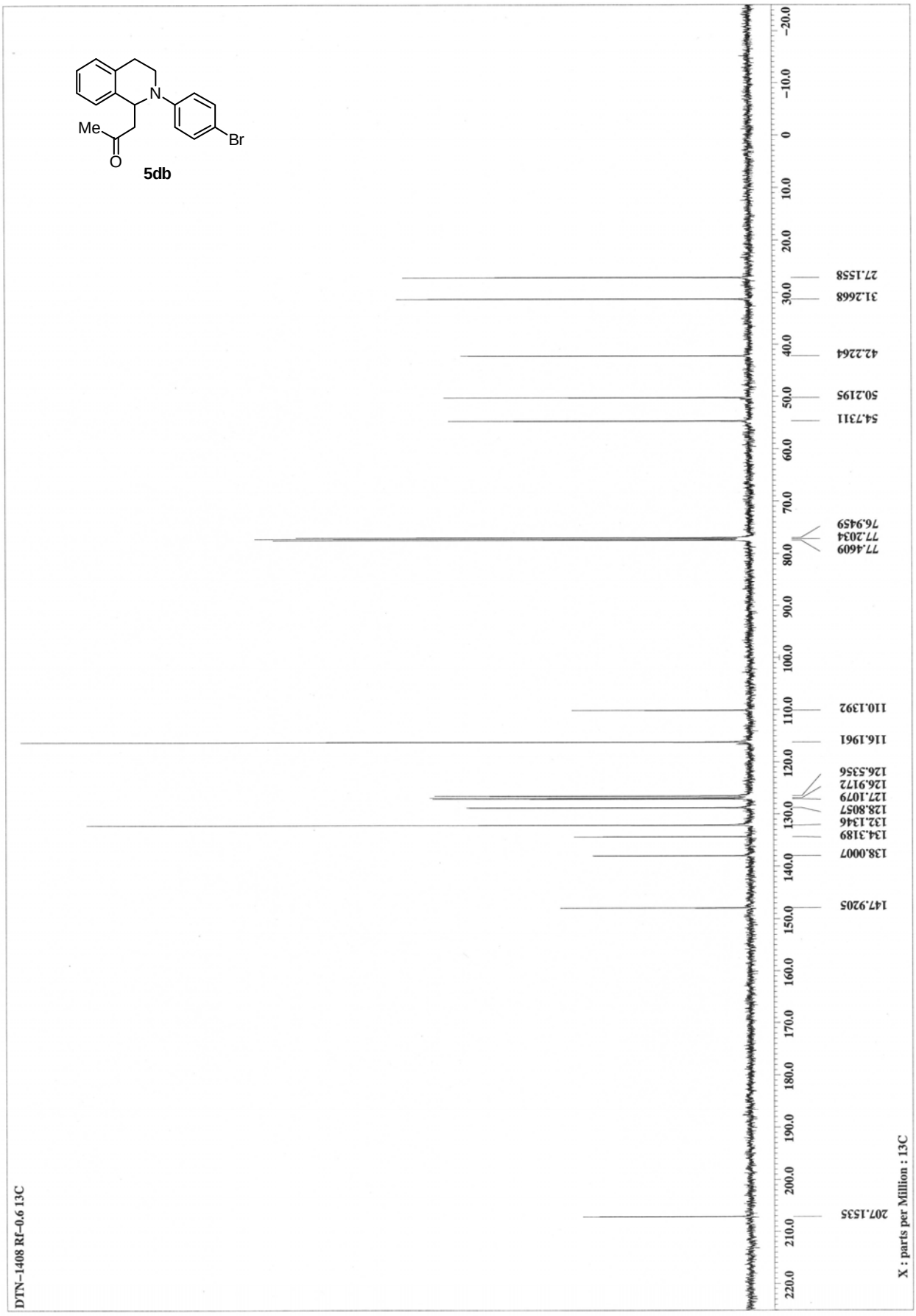

Me
N
Me
O
5bb
DTN-1367 Rf-0.5
X : parts per Million : 1H

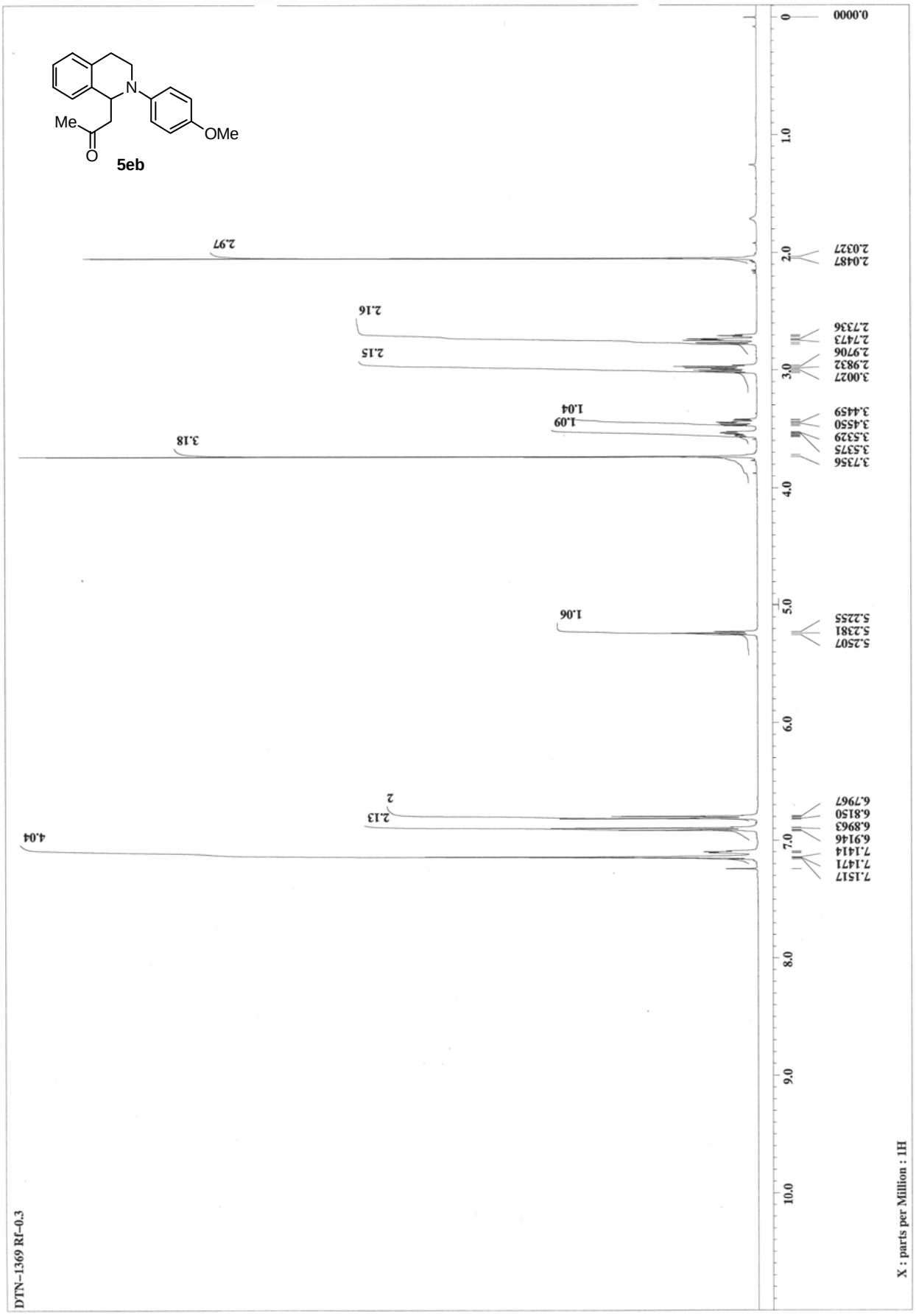

Me
N
Me
O
5bb
DTN-1367 Rf-0.5 13C
X : parts per Million : 13C
20.4694
27.1081
31.1428
42.3027
50.1813
55.2843
76.9554
77.2034
77.4609
115.7859
126.3258
126.8313
126.9744
128.0713
128.9297
129.9599
134.5287
138.4204
147.0239
207.5351

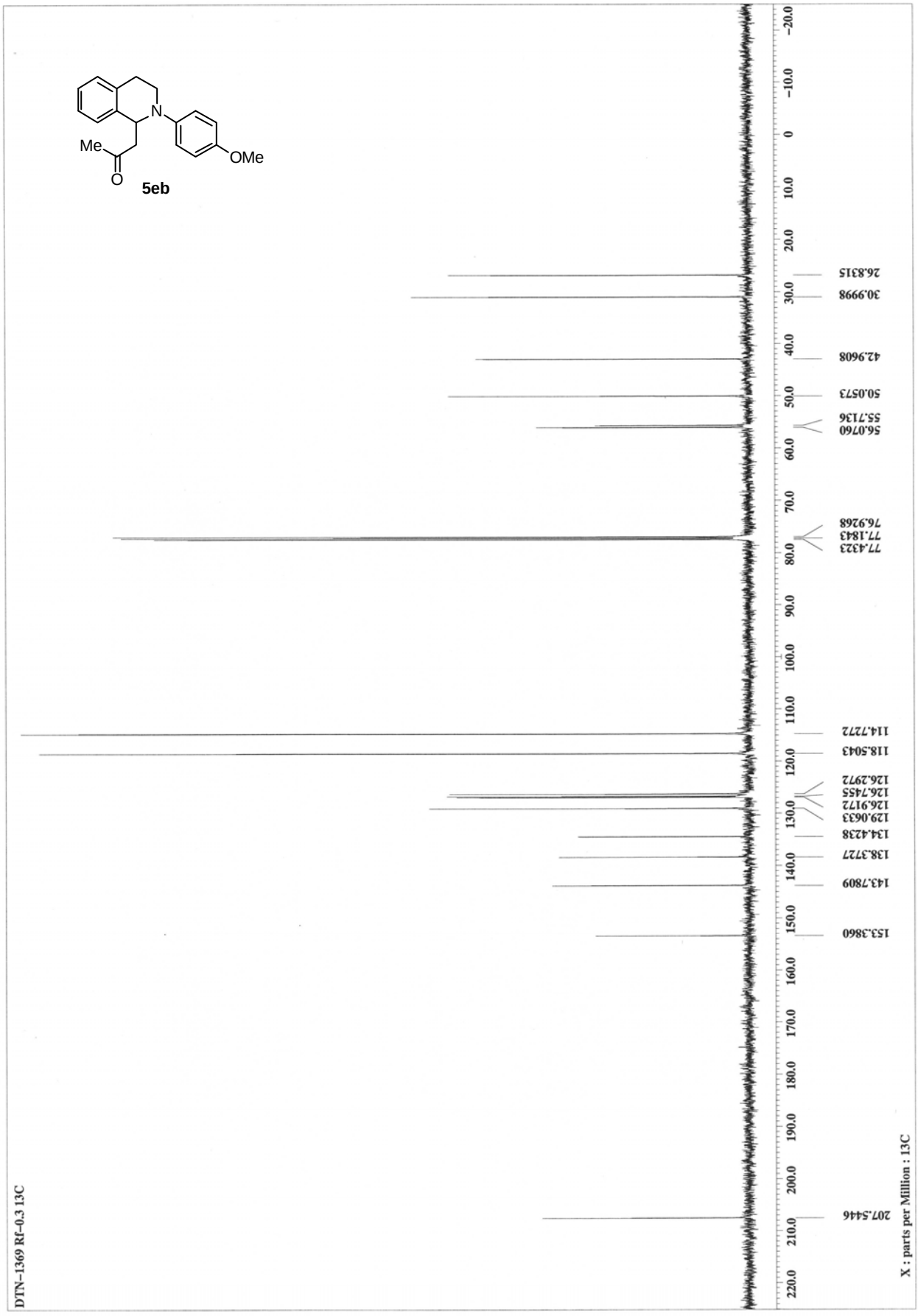

Me
O
N
Cl
5cb
DTN-1381 Rf-0.5
X : parts per Million : 1H
-0.0000
1.6582
2.0671
2.83
2.03
2.04
2.06
1.02
2
5.74
7.1700
7.1517
7.1448
6.8494
6.8310
5.3446
5.3309
5.3194
3.5661
3.5100
3.5008
3.4917
2.9992
2.9878
2.8332
2.8195
2.7862

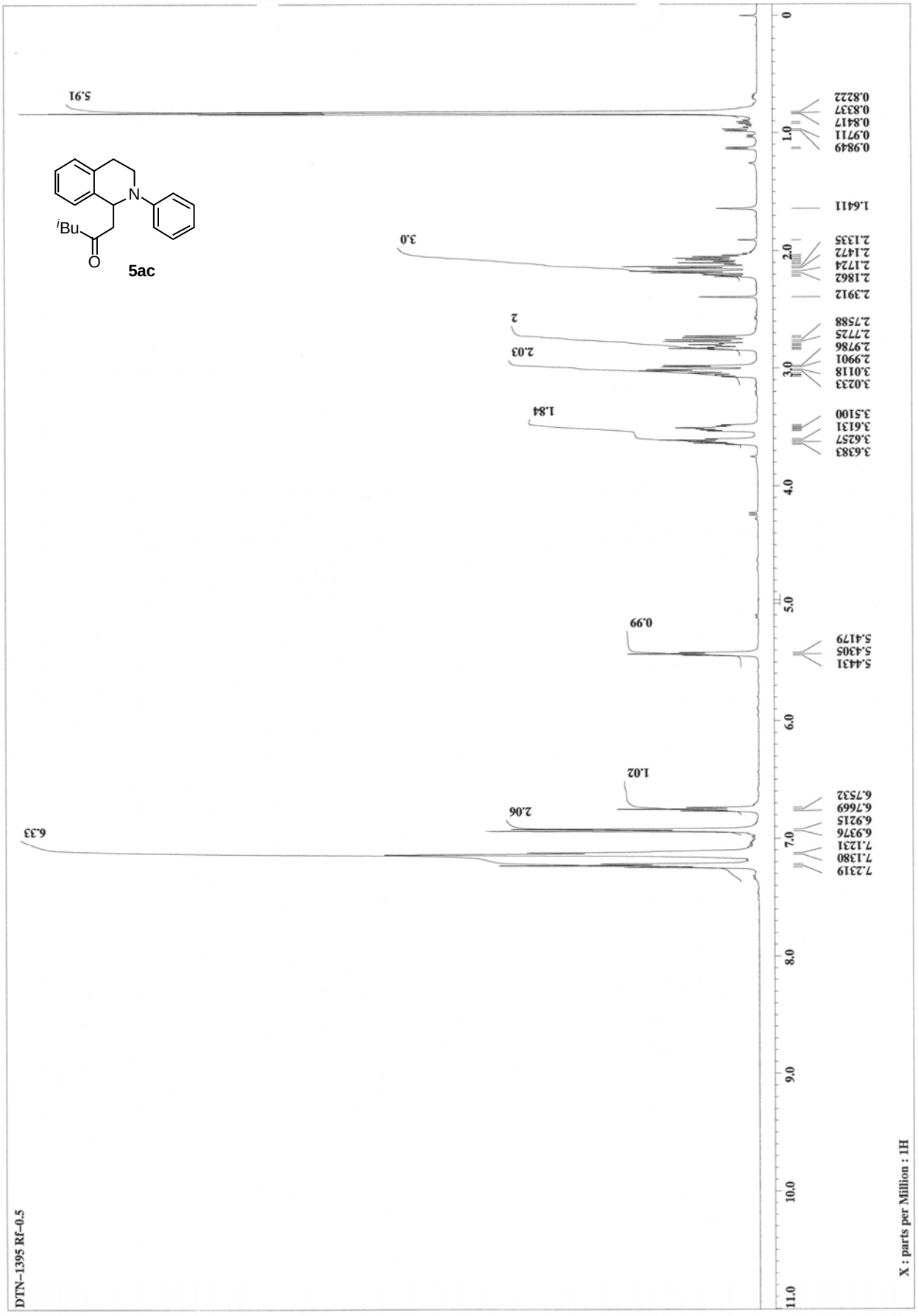

Me
O
N
Cl
5cb
DTN-1381 Rf-0.5 13C
X : parts per Million : 13C
27.1177
31.2478
42.2931
50.2386
54.8646
76.9363
77.1939
77.4419
115.8909
123.0255
126.5165
126.9172
127.0793
128.8344
129.2350
134.3189
138.0293
147.5676
207.1822

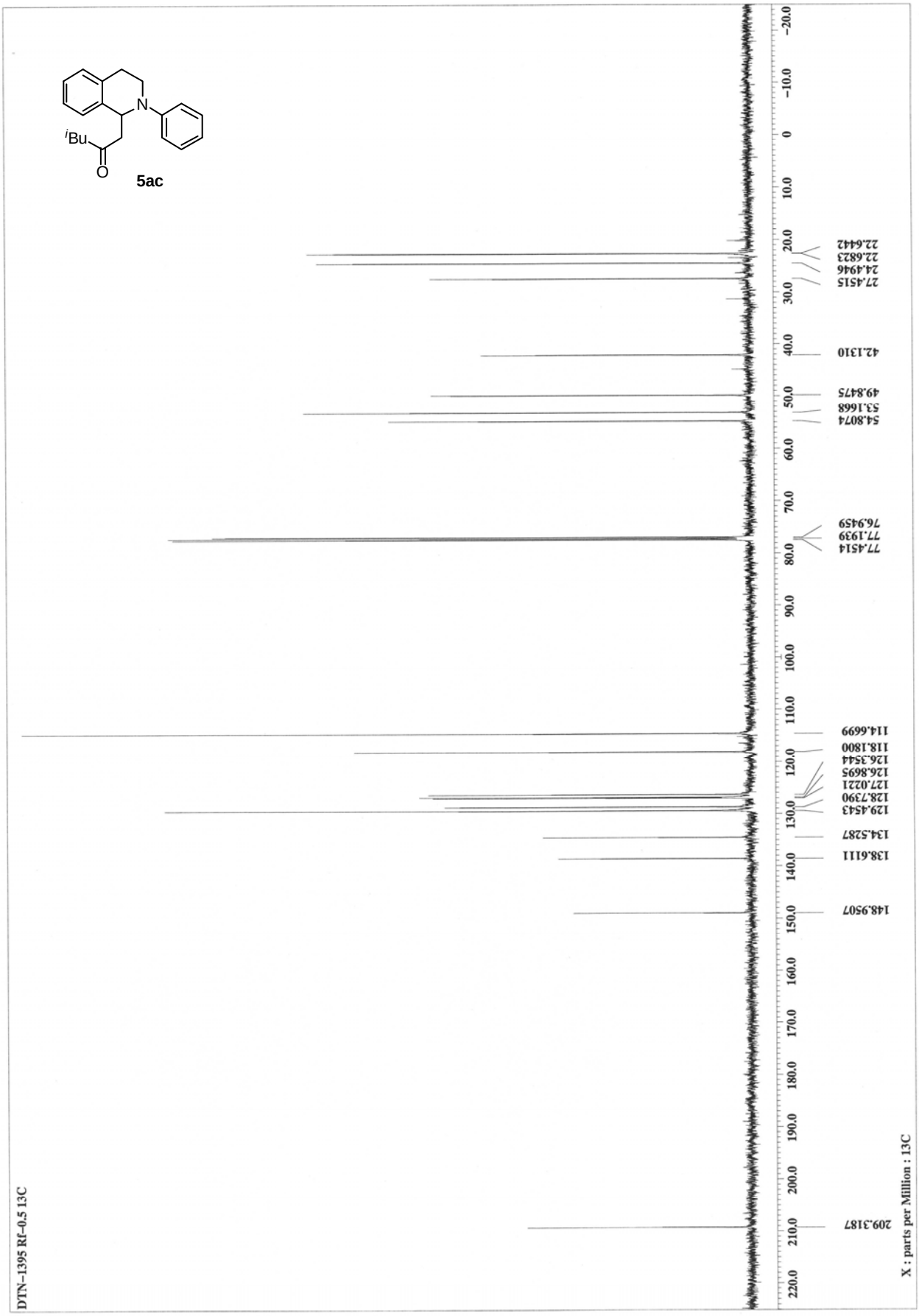

Me
O
N
Br
5db
DTN-1408 Rf-0.6
X : parts per Million : 1H


Me
O
N
Br
5db
DTN-1408 RF-0.6 13C
X : parts per Million : 13C
207.1535
147.9205
138.0007
134.3189
132.1346
128.8057
127.1079
126.9172
126.5356
116.1961
110.1392
77.4609
77.2034
76.9459
54.7311
50.2195
42.2264
31.2668
27.1558


Me
O
N
OMe
5eb
DTN-1369 RF-0.3
X : parts per Million : 1H


Me
O
N
OMe
5eb
DTN-1369 Rf-0.3 13C
X : parts per Million : 13C
26.8315
30.9998
42.9608
50.0573
55.7136
56.0760
76.9268
77.1843
77.4323
114.7272
118.5043
126.2972
126.7455
126.9172
129.0633
134.4238
138.3727
143.7809
153.3860
207.5446


5.91
3.0
2
2.03
1.84
0.99
1.02
2.06
6.33
5ac
DTN-1395 Rf-0.5
X : parts per Million : 1H


5ac
DTN-1395 RF-0.5 13C
X : parts per Million : 13C
22.6442
22.6823
24.4946
27.4515
42.1310
49.8475
53.1668
54.8074
76.9459
77.1939
77.4514
114.6699
118.1800
126.3544
126.8695
127.0221
128.7390
129.4543
134.5287
138.6111
148.9507
209.3187