

Pd-catalyzed desulfitative arylation of olefins by *N*-methoxysulfonamide

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Table of contents

- | | |
|---|---------------------------------|
| 1. Synthesis and analytical data | S ₃ -S ₇ |
| 2. References | S ₇ -S ₈ |
| 3. ¹ H and ¹³ C NMR spectra | S ₈ -S ₈₆ |

General procedure for the desulfitative Heck reaction of *N*-methoxy arylsulfonamides with alkenes (procedure A):

An oven-dried Schlenk tube equipped with a stir bar was charged with *N*-methoxy sulfonamide (**1**, 0.5 mmol), Pd(OAc)₂ (5.6 mg, 5 mol %), CuCl₂ (1 mmol), NaOAc (1 mmol), alkene (**2**, 0.75 mmol) and 3 mL of anhydrous ethyl acetate. The resulting reaction mixture was stirred at 130 °C (oil bath temperature) for 12 h until complete consumption of starting material as monitored by TLC. After cooling to room temperature, the reaction mixture was triturated with water (10 mL) and extracted with ethyl acetate (3 × 10 mL). The combined organic layer was dried over anhydrous sodium sulfate and concentrated under reduced pressure. The crude reaction mixture was purified by column chromatography over silica gel to afford the desired product (**3**).

General procedure for the homocoupling of *N*-methoxy arylsulfonamides (procedure B):

To a Schlenk tube were added *N*-methoxy sulfonamide (**1**, 0.5 mmol), CuCl₂ (1.25 mmol), NaOAc (1 mmol) and anhydrous ethyl acetate (3 mL). Then the tube was stirred at 160 °C (oil bath temperature) for 12 h until complete consumption of starting material as monitored by TLC. After cooling to room temperature, the reaction mixture was triturated with water (10 mL) and extracted with ethyl acetate (3 × 10 mL). The combined organic layer was dried over anhydrous sodium sulfate and concentrated under reduced pressure. The crude reaction mixture was purified by column chromatography over silica gel to afford the desired product (**4**).

3-p-Tolyl-acrylic acid methyl ester^[1] (3aa). Following the general procedure A, the desired compound was obtained from the reaction of *N*-methoxy-4-methyl-benzenesulfonamide (**1a**) and methyl acrylate (**2a**) as a white solid in 73% yield (64 mg). M.P. 58 °C; IR 1708, 1630, 1315, 1158, 983, 809, 503 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.69 (d, 1H, J = 16.0 Hz), 7.44 (d, 2H, J = 8.0 Hz), 7.21 (d, 2H, J = 8.0 Hz), 6.42 (d, 1H, J = 16.0 Hz), 3.82 (s, 3H), 2.39 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 167.6, 144.9, 140.7, 131.6, 129.6, 128.0, 116.7, 51.6, 21.4.

3-phenyl-acrylic acid methyl ester^[1] (3ba). Following the general procedure A, the desired compound was obtained from the reaction of *N*-methoxy-benzenesulfonamide (**1b**) and methyl acrylate (**2a**) as a white solid in 68% yield (55 mg). M.P. 35 °C; IR 1717, 1630, 1446, 1307, 1272, 1167, 983, 765, 687 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.72 (d, 1H, J = 16.0 Hz), 7.58-7.51 (m, 2H), 7.43-7.38 (m, 3H), 6.47 (d, 1H, J = 16.0 Hz), 3.83 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 167.4, 144.8, 134.4, 130.3, 128.9, 128.0, 117.8, 51.7.

3-(4-Chloro-phenyl)-acrylic acid methyl ester^[1] (3ca). Following the general procedure A, the desired compound was obtained from the reaction of 4-chloro-*N*-methoxy-benzenesulfonamide (**1c**) and methyl acrylate (**2a**) as a white solid in 65 % yield (64 mg). M.P. 73 °C; IR 1700, 1630, 1489, 1315, 1167, 1001, 817, 495 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.66 (d, 1H, J = 16.0 Hz), 7.47 (d, 2H, J = 8.4 Hz), 7.38 (d, 2H, J = 8.4 Hz), 6.43 (d, 1H, J = 16.0 Hz), 3.83 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 167.1, 143.5, 133.3, 132.1, 124.5, 118.5, 51.8.

3-(4-Bromo-phenyl)-acrylic acid methyl ester^[2] (3da). Following the general procedure A, the desired compound was obtained from the reaction of 4-bromo-*N*-methoxy-benzenesulfonamide (**1d**) and methyl acrylate (**2a**) as a white solid in 62 % yield (74 mg). M.P. 90 °C; IR 1694, 1630, 1489, 1429, 1307, 1167, 1071, 1001, 813, 495 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.64 (d, 1H, J = 16.0 Hz), 7.53 (d, 2H, J = 8.4 Hz), 7.40 (d, 2H, J = 8.4 Hz), 6.44 (d, 1H, J = 16.0 Hz), 3.82 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 167.1, 143.4, 133.3, 132.1, 129.4, 124.5, 118.5, 51.7.

3-(4-Nitro-phenyl)-acrylic acid methyl ester^[2] (3ea). Following the general procedure A, the desired compound was obtained from the reaction of *N*-methoxy-4-nitro-benzenesulfonamide (**1e**) and methyl acrylate (**2a**) as a white solid in 48 % yield (50 mg). M.P. 123 °C; IR 1798, 1532, 1432, 1331, 1158, 1052, 994, 837, 768 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.31-8.23 (m, 2H), 7.78-7.66 (m, 3H), 6.58 (d, 1H, J = 16.0 Hz), 3.86 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 166.4, 148.5, 141.9, 140.4, 128.6, 124.1, 122.1, 52.0.

3-(2-Methoxycarbonyl-vinyl)-benzoic acid methyl ester^[3] (3fa). Following the general procedure A, the desired compound was obtained from the reaction of 3-methoxysulfamoyl-benzoic acid methyl ester (**1f**) and methyl acrylate (**2a**) as a white solid in 53 % yield (58 mg). M.P. 89 °C; IR 1717, 1437, 1307, 1224, 1080, 983, 739, 695 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.22 (s, 1H), 8.06 (d, 1H, J = 7.6 Hz), 7.77-7.68 (m, 2H), 7.49 (t, 1H, J = 8.0 Hz), 6.53 (d, 1H, 16.0 Hz), 3.96 (s, 3H), 3.83 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 167.1, 166.4, 143.6, 134.7, 132.2, 131.0, 130.9, 129.0, 129.0, 119.1, 52.3, 51.8.

3-(3,4-Dichloro-phenyl)-acrylic acid methyl ester^[4] (3ga). Following the general procedure A, the desired compound was obtained from the reaction of 3,4-dichloro-N-methoxy-benzenesulfonamide (**1g**) and methyl acrylate (**2a**) as a white solid in 58 % yield (67 mg). M.P. 92 °C; IR 1717, 1638, 1429, 1315, 992, 861, 809, 747, 530 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.62 (s, 1H), 7.60 (d, 1H, J = 15.6 Hz), 7.48 (d, 1H, J = 8.4 Hz), 7.36 (dd, 1H, J = 6.8 Hz), 6.44 (d, 1H, J = 16.0 Hz), 3.83 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 166.7, 142.0, 134.4, 134.2, 133.2, 130.9, 129.6, 126.9, 119.7, 51.8.

3-(4-Ethyl-phenyl)-acrylic acid methyl ester^[5] (3ha). Following the general procedure A, the desired compound was obtained from the reaction of 4-ethyl-N-methoxy-benzenesulfonamide (**1h**) and methyl acrylate (**2a**) as an oily liquid in 75% yield (71 mg). IR 1717, 1630, 1446, 1307, 1272, 1167, 983, 765, 687 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.70 (d, 1H, J = 16.0 Hz), 7.46 (d, 2H, J = 8.4 Hz), 7.23 (d, 2H, J = 8.0 Hz), 6.42 (d, 1H, J = 16.0 Hz), 3.82 (s, 3H), 2.67 (q, 2H, J = 7.6 Hz), 1.26 (t, 3H, J = 7.6 Hz); ¹³C NMR (100 MHz, CDCl₃): δ 167.6, 147.0, 144.8, 131.9, 128.4, 128.1, 116.7, 51.5, 28.8, 15.3.

3-Biphenyl-4-yl-acrylic acid methyl ester^[1] (3ja). Following the general procedure A, the desired compound was obtained from the reaction of biphenyl-4-sulfonic acid methoxy-amide (**1j**) and methyl acrylate (**2a**) as a white solid in 66 % yield (63 mg). M.P. 147 °C; IR 1717, 1638, 1559, 1489, 1437, 1307, 1158, 983, 835, 765 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.73 (d, 1H, J = 16.0 Hz), 7.65-7.56 (m, 6H), 7.45 (t, 2H, J = 7.6 Hz), 7.38 (d, 1H, J = 7.2 Hz), 6.48 (d, 1H, J = 16.0 Hz), 3.82 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 167.4, 144.3, 143.1, 140.1, 133.4, 128.8, 128.5, 127.8, 127.5, 127.0, 117.7, 51.6.

3-Naphthalen-2-yl-acrylic acid methyl ester^[3] (3ka). Following the general procedure A, the desired compound was obtained from the reaction of naphthalene-2-sulfonic acid methoxy-amide (**1k**) and methyl acrylate (**2a**) as an oily liquid in 61 % yield (64 mg). IR 1708, 1638, 1437, 1307, 1158, 974, 774 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.53 (d, 1H, J = 15.6 Hz), 8.18 (d, 1H, J = 8.4 Hz), 7.87 (t, 2H, J = 8.0 Hz), 7.74 (d, 1H, J = 7.2 Hz), 7.60-7.43 (m, 3H), 6.52 (d, 1H, J = 15.6 Hz), 3.85 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 167.2, 141.9, 133.7, 131.8, 131.4, 130.5, 128.7, 126.8, 126.2, 125.4, 125.0, 123.3, 120.5, 51.7.

3-(4-Methoxy-phenyl)-acrylic acid methyl ester^[2] (3la). Following the general procedure A, the desired compound was obtained from the reaction of 4,N-dimethoxy-benzenesulfonamide (**1l**) and methyl acrylate (**2a**) as a white solid in 66 % yield (63 mg). M.P. 86 °C; IR 1717, 1638, 1603, 1559, 1507, 1437, 1289, 1167, 983, 817 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.67 (d, 1H, J = 16.0 Hz), 7.49 (d, 2H, J = 8.8 Hz), 6.92 (d, 2H, J = 8.4 Hz), 6.33 (d, 1H, J = 16.0 Hz), 3.86 (s, 3H), 3.81 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 167.7, 161.4, 144.5, 129.7, 127.1, 115.2, 114.3, 55.3, 51.9.

3-(3,4-Dimethyl-phenyl)-acrylic acid methyl ester^[6] (3ma). Following the general procedure A, the desired compound was obtained from the reaction of N-methoxy-3,4-dimethyl-benzenesulfonamide (**1m**) and methyl acrylate (**2a**) as a white solid in 75 % yield (71 mg). M.P. 74 °C; IR 1700, 1442, 1272, 1175, 992, 826, 556 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.67 (d, 1H, J = 16.0 Hz), 7.33-7.26 (m, 2H), 7.16 (d, 1H, J = 7.6 Hz), 6.41 (d, 1H, J = 16.0 Hz), 3.82 (s, 3H), 2.30 (s, 6H); ¹³C NMR (100 MHz, CDCl₃): δ 167.6, 145.0, 139.4, 137.1, 132.0, 130.1, 129.3, 125.7, 116.5, 51.5, 19.7, 19.7.

3-(3,4-Dimethoxy-phenyl)-acrylic acid methyl ester^[7] (3na). Following the general procedure A, the desired compound was obtained from the reaction of 3,4,N-Trimethoxy-benzenesulfonamide (**1n**) and methyl acrylate (**2a**) as a white solid in 61 % yield (68 mg). M.P. 141 °C; IR 1690, 1507, 1437, 1228, 1140, 1009, 983, 804 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.65 (d, 1H, J = 15.6 Hz), 7.12 (dd, 1H, J = 8.0 Hz), 7.06 (s, 1H), 6.88 (d, 1H, J = 8.0 Hz), 6.33 (d, 1H, J = 16.0 Hz), 3.93 (s, 6H), 3.81 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 167.6, 151.1, 149.2, 144.7, 127.4, 122.5, 115.5, 111.1, 109.7, 55.9, 55.9, 51.5.

3-(5-Bromo-2-methoxy-phenyl)-acrylic acid methyl ester^[8] (3oa). Following the general procedure A, the desired compound was obtained from the reaction of 5-bromo-2,N-dimethoxy-benzenesulfonamide (**1o**) and methyl acrylate (**2a**) as a white solid in 61 % yield (82 mg); IR 1717, 1636, 1481, 1315, 1175, 983, 861, 809, 625, 455 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.89 (d, 1H, J = 16.0 Hz), 7.60 (s, 1H), 7.42 (dd, 1H, J₁ = 8.8 Hz, J₂ = 2.4 Hz), 6.79 (d, 1H, J = 8.8 Hz), 6.49 (d, 1H, J = 16.4 Hz), 3.86 (s, 3H), 3.80 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 167.4, 157.2, 138.6, 133.7, 131.1, 125.3, 119.5, 112.9, 112.9, 55.7, 51.6.

3-(5-Acetyl-2-methoxy-phenyl)-acrylic acid methyl ester (3pa). Following the general procedure A, the desired compound was obtained from the reaction of 5-acetyl-2,N-dimethoxy-benzenesulfonamide (**1p**) and methyl acrylate (**2a**) as a white solid in 58 % yield (68 mg). IR 1700, 1673, 1437, 1241, 1123, 983, 826, 565 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.14 (d, 1H, J = 2.0 Hz), 8.03-7.95 (m, 2H), 6.98 (d, 1H, J = 8.8 Hz), 6.62 (d, 1H, J = 16.4 Hz), 3.98 (s, 3H), 3.83 (s, 3H), 2.60 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 196.3, 167.5, 161.7, 139.2, 131.9, 130.2, 129.4, 123.4, 119.7, 110.7, 55.8, 51.7, 26.2. HRMS (ESI) calcd for C₁₃H₁₅O₄⁺ [M + H]⁺ 235.065; found 235.078.

3-(2,5-Dichloro-phenyl)-acrylic acid methyl ester^[9] (3qa). Following the general procedure A, the desired compound was obtained from the reaction of 2,5-dichloro-N-methoxy-benzenesulfonamide (**1q**) and methyl acrylate (**2a**) as a white solid in 57 % yield (65 mg). M.P. 95 °C; IR 1708, 1454, 1394, 1272, 1175, 1036, 983, 800, 573, 486 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.01 (d, 1H, J = 16.0 Hz), 7.60 (s, 1H), 7.37 (d, 1H, J = 8.4 Hz), 7.33-7.27 (m, 1H), 6.44 (d, 1H, J = 16.0 Hz), 3.84 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 166.5, 139.3, 134.1, 133.1, 133.0, 131.2, 130.8, 127.4, 121.7, 51.9.

3-(2,4-Dimethyl-phenyl)-acrylic acid methyl ester^[10] (3ra). Following the general procedure A, the desired compound was obtained from the reaction of N-methoxy-2,4-dimethyl-benzenesulfonamide (**1r**) and methyl acrylate (**2a**) as a colourless oil in 70 % yield (66 mg). IR 1717, 1612, 1437, 1315, 1280, 1158, 983, 817, 713, 547 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.98 (d, 1H, J = 16.0 Hz), 7.47 (d, 1H, J = 8.4 Hz), 7.04 (s, 2H), 6.35 (s, 1H, J = 16.0 Hz), 3.82 (s, 3H), 2.42 (s, 3H), 2.34 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 167.6, 142.4, 140.3, 137.6, 131.5, 130.5, 127.1, 126.3, 117.6, 51.5, 21.2, 19.6.

3-Thiophen-2-yl-acrylic acid methyl ester^[9] (3sa). Following the general procedure A, the desired compound was obtained from the reaction of thiophene-2-sulfonic acid methoxy-amide (**1s**) and methyl acrylate (**2a**) as a white solid in 52 % yield (44 mg). M.P. 55 °C; IR 1700, 1638, 1419, 1307, 1210, 1158, 974, 844, 722, 590 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.80 (d, 1H, J = 15.6 Hz), 7.39 (d, 1H, J = 5.2 Hz), 7.27 (d, 1H, J = 6.4 Hz), 7.07 (dd, 1H, J₁ = 5.2 Hz, J₂ = 3.6 Hz), 6.26 (d, 1H, J = 16.0 Hz), 3.80 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 167.3, 139.5, 137.3, 130.9, 128.4, 128.0, 116.5, 51.7.

3-p-Tolyl-acrylic acid butyl ester^[11] (3ab). Following the general procedure A, the desired compound was obtained from the reaction of N-methoxy-4-methyl-benzenesulfonamide (**1a**) and acrylic acid butyl ester (**2b**) as a yellow oil in 76 % yield (83 mg). IR 1708, 1638, 1516, 1315, 1167, 974, 809, 503 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.68 (d, 1H, J = 16.0 Hz), 7.44 (d, 2H, J = 8.4 Hz), 7.21 (d, 2H, J = 8.0 Hz), 6.41 (d, 1H, J = 16.0 Hz), 4.22 (t, 2H, J = 6.8 Hz), 2.39 (s, 3H), 1.75-1.65 (m, 2H), 1.53-1.39 (m, 2H), 0.99 (t, 3H, J = 7.2 Hz); ¹³C NMR (100 MHz, CDCl₃): δ 162.4, 139.7, 135.8, 127.0, 124.8, 123.2, 112.5, 59.5, 26.0, 16.6, 14.4, 8.9.

3-p-Tolyl-acrylic acid tert-butyl ester^[11] (3ac). Following the general procedure A, the desired compound was obtained from the reaction of N-methoxy-4-methyl-benzenesulfonamide (**1a**) and acrylic acid tert-butyl ester (**2c**) as a yellow oil in 77 % yield (84 mg). IR 1708, 1630, 1507, 1367, 1324, 1150, 974, 870, 809 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.58 (d, 1H, J = 16.0 Hz), 7.42 (d, 2H, J = 8.4 Hz), 7.19 (d, 2H, J = 7.6 Hz), 6.36 (d, 1H, J = 16.0 Hz), 2.38 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 166.5, 143.5, 140.2, 131.9, 129.5, 127.9, 119.1, 80.3, 28.2, 21.3.

3-p-Tolyl-acrylic acid^[12] (3ad). Following the general procedure A, the desired compound was obtained from the reaction of N-methoxy-4-methyl-benzenesulfonamide (**1a**) and acrylic acid (**2d**) as a white solid in 60 % yield (49 mg). M.P. 180°C; IR 1682, 1655, 1559, 1419, 1315, 983, 931, 809, 687, 495 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): δ 7.59-7.51 (m, 3H), 7.22 (d, 2H, J = 7.2 Hz), 6.45 (d, 1H, J = 16.0 Hz), 2.31 (s, 3H); ¹³C NMR (100 MHz, DMSO-d₆): δ 168.1, 144.4, 140.6, 131.9, 129.9, 128.6, 118.5, 21.4.

N,N-Dimethyl-3-p-tolyl-acrylamide^[13] (3ae). Following the general procedure A, the desired compound was obtained from the reaction of N-methoxy-4-methyl-benzenesulfonamide (**1a**) and N,N-dimethyl-acrylamide (**2e**) as a white solid in 76 % yield (71 mg). M.P. 99 °C; IR 2215, 1603, 1507, 1272, 1175, 974, 792, 538 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): δ 7.57 (d, 2H, J = 7.2 Hz), 7.45 (d, 1H, J = 15.2 Hz), 7.20 (d, 2H, J = 7.6 Hz), 7.12 (d, 1H, J = 15.6 Hz), 3.13 (s, 3H), 2.92 (s, 3H), 2.30 (s, 3H); ¹³C NMR (100 MHz, DMSO-d₆): δ 170.9, 146.1, 144.4, 137.6, 134.5, 133.1, 122.5, 42.0, 40.5, 26.1.

3-p-Tolyl-acrylonitrile^[14] (3af). Following the general procedure A, the desired compound was obtained from the reaction of N-methoxy-4-methyl-benzenesulfonamide (**1a**) and acrylonitrile (**2f**) as a yellow oil in 78 % yield (56

mg). IR 2215, 1603, 1507, 1272, 1185, 974, 800, 459 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.43-7.35 (m, 3H), 7.23 (s, 2H), 5.85 (d, 2H, J = 16.4 Hz), 2.41 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 150.5, 141.8, 130.9, 129.8, 127.3, 118.4, 95.0, 21.5.

1-Methyl-4-styryl-benzene^[1] (3ag). Following the general procedure A, the desired compound was obtained from the reaction of *N*-methoxy-4-methyl-benzenesulfonamide (**1a**) and vinyl-benzene (**2g**) as a white solid in 46 % yield (45 mg). M.P. 73°C; IR 1655, 1507, 1446, 974, 809, 747, 687, 530 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.53 (d, 2H, J = 7.2 Hz), 7.44 (d, 2H, J = 8.0 Hz), 7.37 (t, 2H, J = 8.0 Hz), 7.27 (d, 1H, J = 7.2 Hz), 7.19 (d, 2H, J = 7.6 Hz), 7.10 (d, 2H, J = 2.4 Hz), 2.38 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): 137.5, 137.5, 134.6, 129.3, 128.6, 128.6, 127.7, 127.3, 126.4, 126.4, 21.2.

1-Methyl-4-(2-phenyl-ethenesulfonyl)-benzene^[15] (3ag'). Following the general procedure A, the desired compound was obtained from the reaction of *N*-methoxy-4-methyl-benzenesulfonamide (**1a**) and vinyl-benzene (**2g**) as a white solid in 32 % yield (41 mg). M.P. 126°C; ¹H NMR (400 MHz, CDCl₃): δ 7.85 (d, 2H, J = 8.0 Hz), 7.68 (d, 1H, J = 15.6 Hz), 7.52-7.46 (m, 2H), 7.47-7.38 (m, 3H), 7.36 (d, 2H, J = 8.0 Hz), 7.86 (d, 1H, J = 15.2 Hz), 2.45 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): 144.4, 141.9, 137.7, 132.4, 131.1, 129.9, 129.0, 128.5, 127.7, 127.6, 21.6.

1-Methyl-4-(3-phenoxy-propenyl)-benzene^[16] (3ah). Following the general procedure A, the desired compound was obtained from the reaction of *N*-methoxy-4-methyl-benzenesulfonamide (**1a**) and allyloxy-benzene (**2h**) as a white solid in 75 % yield (84 mg). M.P. 58 °C; IR 1655, 1586, 1489, 1454, 1385, 1228, 1175, 1009, 966, 747, 695, 512 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.36-7.28 (d, 4H), 7.16 (d, 2H, J = 8.0 Hz), 6.98 (t, 3H, J = 6.8 Hz), 6.73 (d, 1H, J = 16.0 Hz), 6.45-6.34 (m, 1H), 4.71 (d, 2H, J = 6.0 Hz), 2.36 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 158.6, 137.8, 133.6, 133.0, 129.4, 129.3, 126.5, 123.4, 120.8, 114.8, 68.7, 21.2.

Carbonic acid methyl ester 3-p-tolyl-allyl-ester^[17] (3ai). Following the general procedure A, the desired compound was obtained from the reaction of *N*-methoxy-4-methyl-benzenesulfonamide (**1a**) and Carbonic acid allyl ester methyl ester (**2i**) as a white solid in 74 % yield (76 mg). M.P. 56 °C; IR 1655, 1595, 1507, 1446, 974, 800, 747, 687, 530 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.31 (d, 2H, J = 8.0 Hz), 7.15 (d, 2H, J = 7.6 Hz), 6.68 (d, 1H, J = 16.0 Hz), 6.32-6.21 (m, 1H), 4.80 (d, 2H, J = 6.4 Hz), 3.82 (s, 3H), 2.36 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 155.7, 138.1, 134.9, 133.2, 129.3, 126.6, 121.3, 68.6, 54.8, 21.2.

1,1'-(1E)-1-Propene-1,3-diylbis[4-methylbenzene]^[18] (3aj). Following the general procedure A, the desired compound was obtained from the reaction of *N*-methoxy-4-methyl-benzenesulfonamide (**1a**) and 3-Bromo-propene (**2j**) as a white solid in 58 % yield (39 mg). ¹H NMR (400 MHz, CDCl₃): δ 7.27 (d, 2H, J = 6.8 Hz), 7.17-6.99 (m, 6H), 6.44 (d, 1H, J = 15.6 Hz), 6.37-6.25 (m, 1H), 3.52 (d, 2H, J = 6.8 Hz), 2.35 (s, 3H), 2.34 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 137.2, 136.7, 135.6, 134.7, 130.6, 129.1, 129.1, 128.5, 128.4, 126.0, 38.9, 21.1, 21.0.

3-p-Tolyl-chromen-2-one^[19] (3ak). Following the general procedure A, the desired compound was obtained from the reaction of *N*-methoxy-4-methyl-benzenesulfonamide (**1a**) and Chromen-2-one (**2k**) as a white solid in 72 % yield (85 mg). M.P. 158 °C; IR 1655, 1586, 1489, 1454, 1385, 1228, 1175, 1009, 966, 747, 695, 512 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.81 (s, 1H), 7.63 (d, 2H, J = 8.0 Hz), 7.59-7.50 (m, 2H), 7.39 (d, 1H, J = 8.0 Hz), 7.35-7.26 (m, 3H), 2.42 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 160.7, 153.4, 139.2, 138.9, 131.8, 131.2, 129.2, 128.4, 128.3, 127.8, 124.4, 119.8, 116.4, 21.3.

3-p-Tolyl-but-2-enoic acid methyl ester^[20] (3al). Following the general procedure A, the desired compound was obtained from the reaction of *N*-methoxy-4-methyl-benzenesulfonamide (**1a**) and But-2-enoic acid methyl ester (**2l**) as a yellow oil in 68 % yield (64 mg). ¹H NMR (400 MHz, CDCl₃): δ 7.39 (d, 2H, J = 8.0 Hz), 7.1 (d, 2H, J = 8.0 Hz), 6.15 (s, 1H), 3.77 (s, 3H), 2.59 (s, 3H), 2.39 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 167.3, 155.7, 139.1, 139.1, 129.1, 126.1, 115.7, 51.0, 21.1, 17.8. HRMS (ESI) calcd for C₁₂H₁₅O₂⁺ [M + H]⁺ 191.1072; found 191.1081.

2-p-Tolyl-but-2-enedioic acid dimethyl ester^[21] (3am). Following the general procedure A, the desired compound was obtained from the reaction of *N*-methoxy-4-methyl-benzenesulfonamide (**1a**) and But-2-enedioic acid dimethyl ester (**2m**) as a colourless oil in 71 % yield (E:Z::8:2) (81 mg). ¹H NMR (400 MHz, CDCl₃): δ 7.39 (d, 2H, J = 8.0 Hz), 7.22 (d, 2H, J = 8.0 Hz), 6.31 (s, 1H), 3.96 (s, 3H), 3.80 (s, 3H), 2.39 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 169.3, 168.5, 165.5, 149.0, 141.2, 138.9, 135.8, 130.3, 129.7, 129.3, 126.7, 125.9, 115.8, 53.7, 52.6, 52.1, 51.9, 46.1, 21.3, 21.0. HRMS (ESI) calcd for C₁₃H₁₄NaO₄⁺ [M + Na]⁺ 257.0790; found 257.0786.

2-Methyl-3-p-tolyl-acrylic acid methyl ester^[22] (3an). Following the general procedure A, when *N*-methoxy-4-methyl-benzenesulfonamide (**1a**) reacted with 2-Methyl-acrylic acid methyl ester (**2n**), we got an inseparable mixture of 2-methyl-3-p-tolyl-acrylic acid methyl ester and 2-(4-methyl-benzyl)-acrylic acid methyl ester (**3an**)^[23] in 1:1 ratio as a yellow oil in 64 % yield (61 mg). ¹H NMR (400 MHz, CDCl₃): δ 7.69 (s, 1H), 7.33 (d, 2H, *J* = 8.0 Hz), 7.22 (d, 2H, *J* = 8.0 Hz), 7.16-7.07 (m, 4H), 6.23 (s, 1H), 5.48 (s, 1H), 3.83 (s, 3H), 3.75 (s, 3H), 3.61 (s, 2H), 2.39 (s, 3H), 2.34 (s, 3H), 2.15 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 169.2, 167.4, 140.3, 138.9, 138.4, 135.8, 135.5, 133.0, 129.7, 129.1, 128.9, 127.3, 126.0, 52.0, 51.8, 37.6, 21.3, 21.0, 14.0.

4,4'-Dimethyl-biphenyl^[24] (4a). Following the general procedure B, when *N*-Methoxy-4-methyl-benzenesulfonamide (**1a**) was stirred at 160 °C the desired compound (**4a**) was obtained as a white crystalline solid in 74% yield (33 mg). M.P. 127 °C; IR 2922, 2852, 1655, 1454, 1001, 800, 547, 503 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.50 (d, 4H, *J* = 8.4 Hz), 7.26 (d, 4H, *J* = 8.0 Hz), 2.41 (s, 6H); ¹³C NMR (100 MHz, CDCl₃): δ 138.0, 136.6, 129.4, 126.8, 21.0.

Biphenyl^[24] (4b). Following the general procedure B, when *N*-Methoxy-benzenesulfonamide (**1b**) was stirred at 160 °C the desired compound (**4b**) was obtained as a white crystalline solid in 70% yield (27 mg). M.P. 72 °C; IR 1481, 1429, 1167, 1001, 904, 730, 687, 608 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.68-7.62 (m, 4H), 7.53-7.45 (m, 4H), 7.44-7.35 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 141.2, 128.7, 127.2, 127.2.

4,4'-Dichloro-biphenyl^[24] (4c). Following the general procedure B, when 3,4-Dichloro-*N*-methoxy-benzenesulfonamide (**1c**) was stirred at 160 °C the desired compound (**4c**) was obtained as a white crystalline solid in 64% yield (36 mg). M.P. 151 °C; IR 1655, 1559, 1472, 1385, 1088, 1001, 809, 704, 547, 503 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.50 (d, 4H, *J* = 8.8 Hz), 7.43 (d, 4H, *J* = 8.8 Hz); ¹³C NMR (100 MHz, CDCl₃): δ 138.4, 133.7, 129.0, 128.2.

4,4'-Dibromo-biphenyl^[24] (4d). Following the general procedure B, when 4-Bromo-*N*-methoxy-benzenesulfonamide (**1d**) was stirred at 160 °C the desired compound (**4d**) was obtained as a white crystalline solid in 62% yield (47 mg). M.P. 166 °C; IR 1582, 1472, 1385, 1071, 1001, 804, 722, 669, 538, 502 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.58 (d, 2H, *J* = 8.4 Hz), 7.43 (d, 2H, *J* = 8.4 Hz); ¹³C NMR (100 MHz, CDCl₃): δ 138.9, 132.0, 128.5, 121.9.

4,4'-Dinitro-biphenyl^[24] (4e). Following the general procedure B, when *N*-Methoxy-4-nitro-benzenesulfonamide (**1e**) was stirred at 160 °C the desired compound (**4e**) was obtained as a white crystalline solid in 45% yield (28 mg). M.P. 226 °C; IR 1577, 1507, 1472, 1341, 1088, 1009, 844, 739, 530 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.20 (d, 4H, *J* = 8.8 Hz), 7.54 (d, 4H, *J* = 8.4 Hz); ¹³C NMR (100 MHz, CDCl₃): δ 146.5, 141.3, 129.5, 124.9.

Biphenyl-3,3'-dicarboxylic acid dimethyl ester^[24] (4f). Following the general procedure B, when biphenyl-4-sulfonic acid methoxy-amide (**1f**) was stirred at 160 °C the desired compound (**4f**) was obtained as a white crystalline solid in 53% yield (35 mg). M.P. 103 °C; IR 1708, 1446, 1376, 1175, 1036, 835, 687, 590, 495 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.32 (s, 2H), 8.07 (d, 2H, *J* = 7.6 Hz), 7.84 (d, 2H, *J* = 8.0 Hz), 7.56 (t, 2H, *J* = 8.0 Hz), 3.98 (s, 6H); ¹³C NMR (100 MHz, CDCl₃): δ 166.9, 140.3, 131.5, 130.8, 129.0, 128.8, 128.2, 52.2.

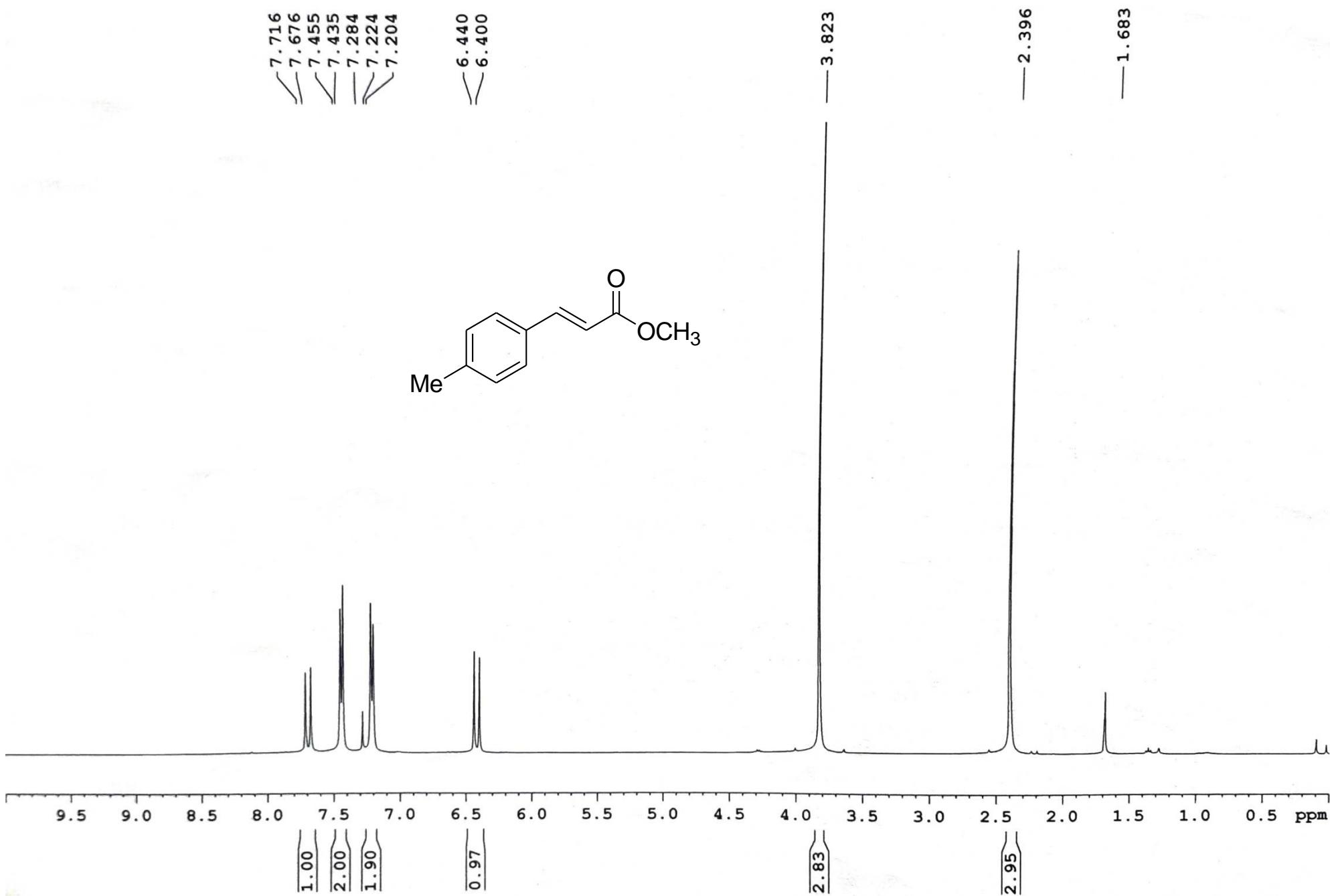
3,4,3',4'-Tetrachloro-biphenyl^[24] (4g). Following the general procedure B, when 3,4-Dichloro-*N*-methoxy-benzenesulfonamide (**1g**) was stirred at 160 °C the desired compound (**4g**) was obtained as a white crystalline solid in 57% yield (41 mg). M.P. 173 °C; IR 1655, 1542, 1454, 1359, 1132, 817, 747, 669, 441 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.65 (d, 2H, *J* = 2.0 Hz), 7.54 (d, 2H, *J* = 8.0 Hz), 7.38 (dd, 2H, *J*₁ = 8.0 Hz, *J*₂ = 2.0 Hz); ¹³C NMR (100 MHz, CDCl₃): δ 138.7, 133.2, 132.4, 130.9, 128.8, 126.1.

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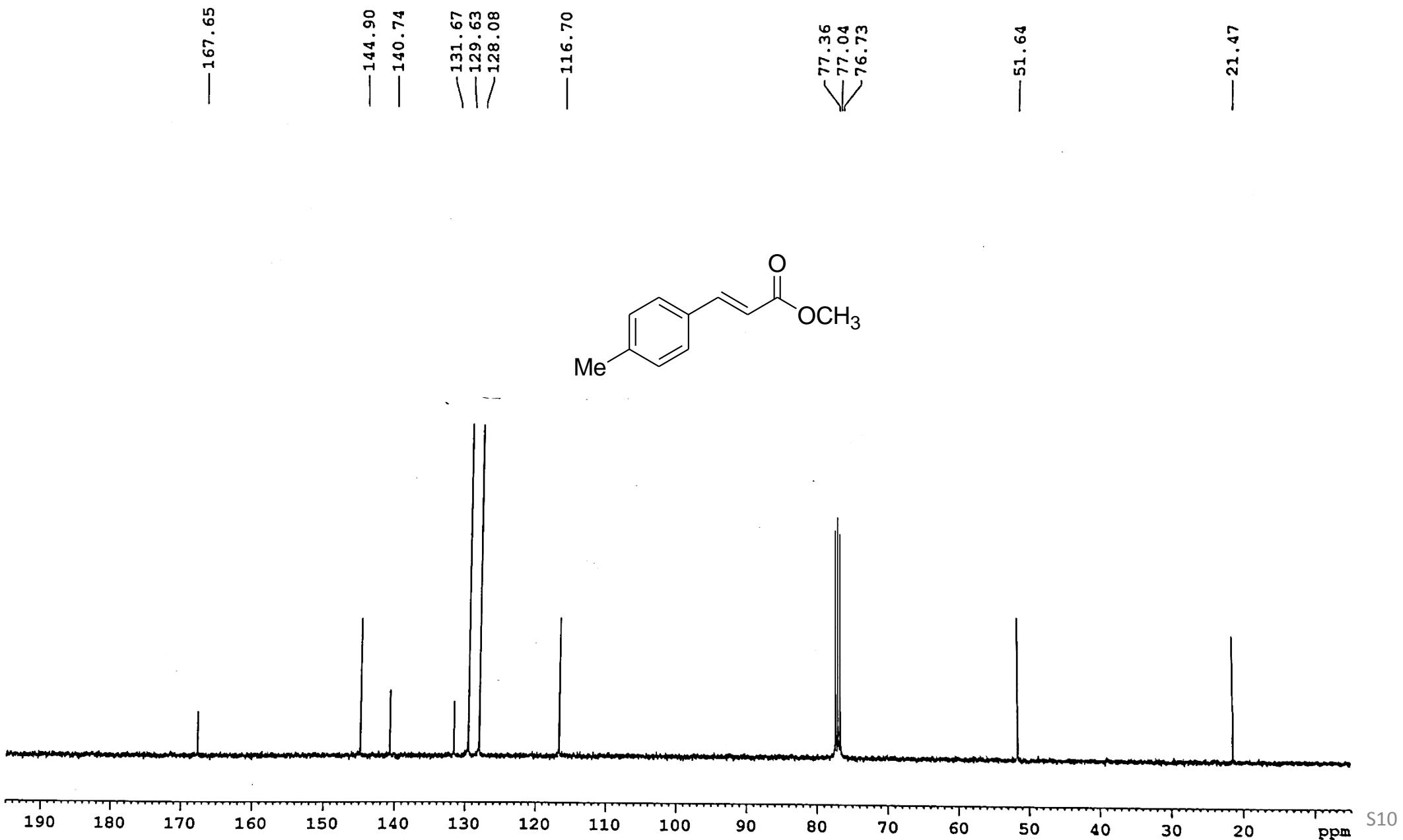
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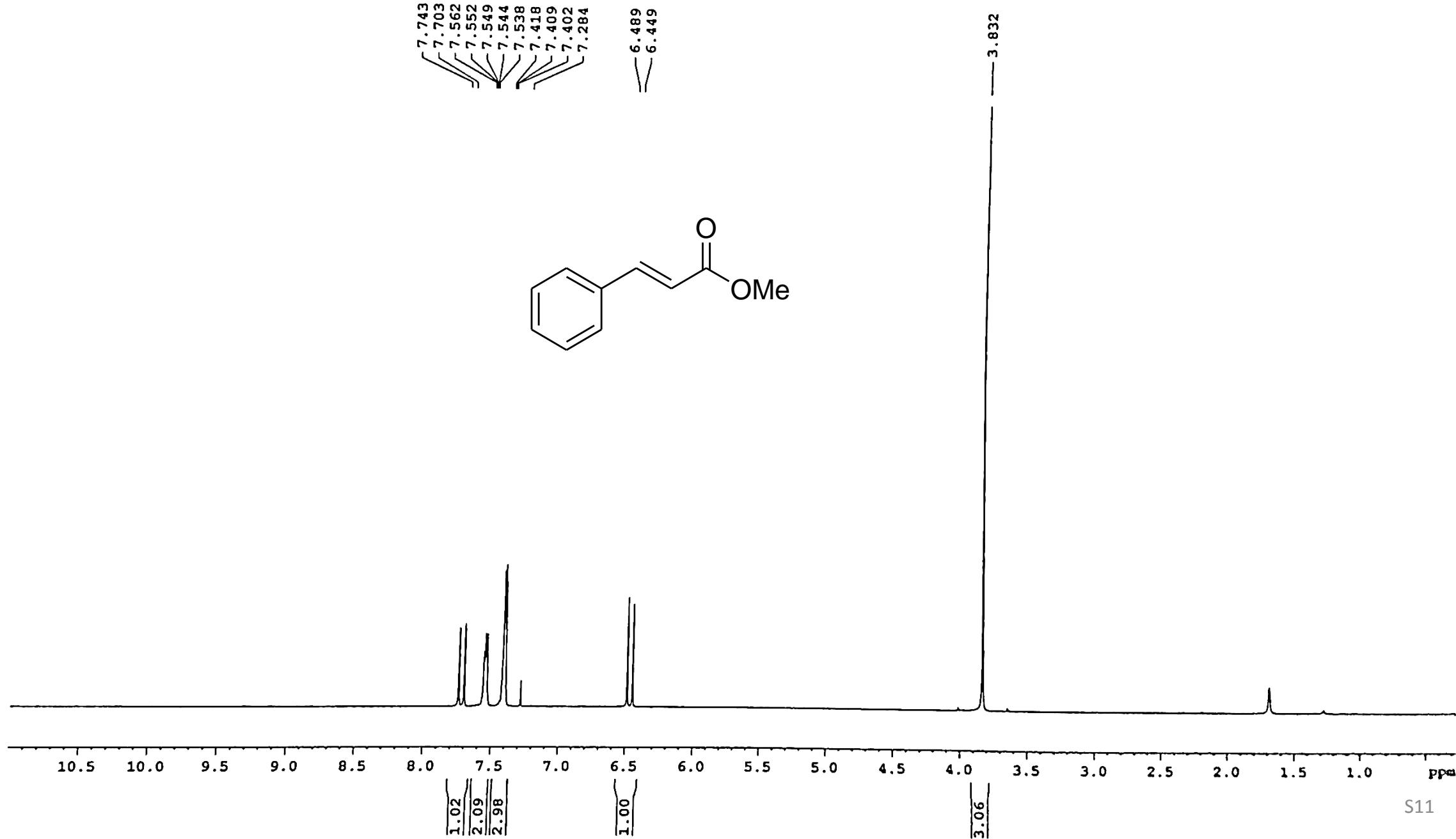
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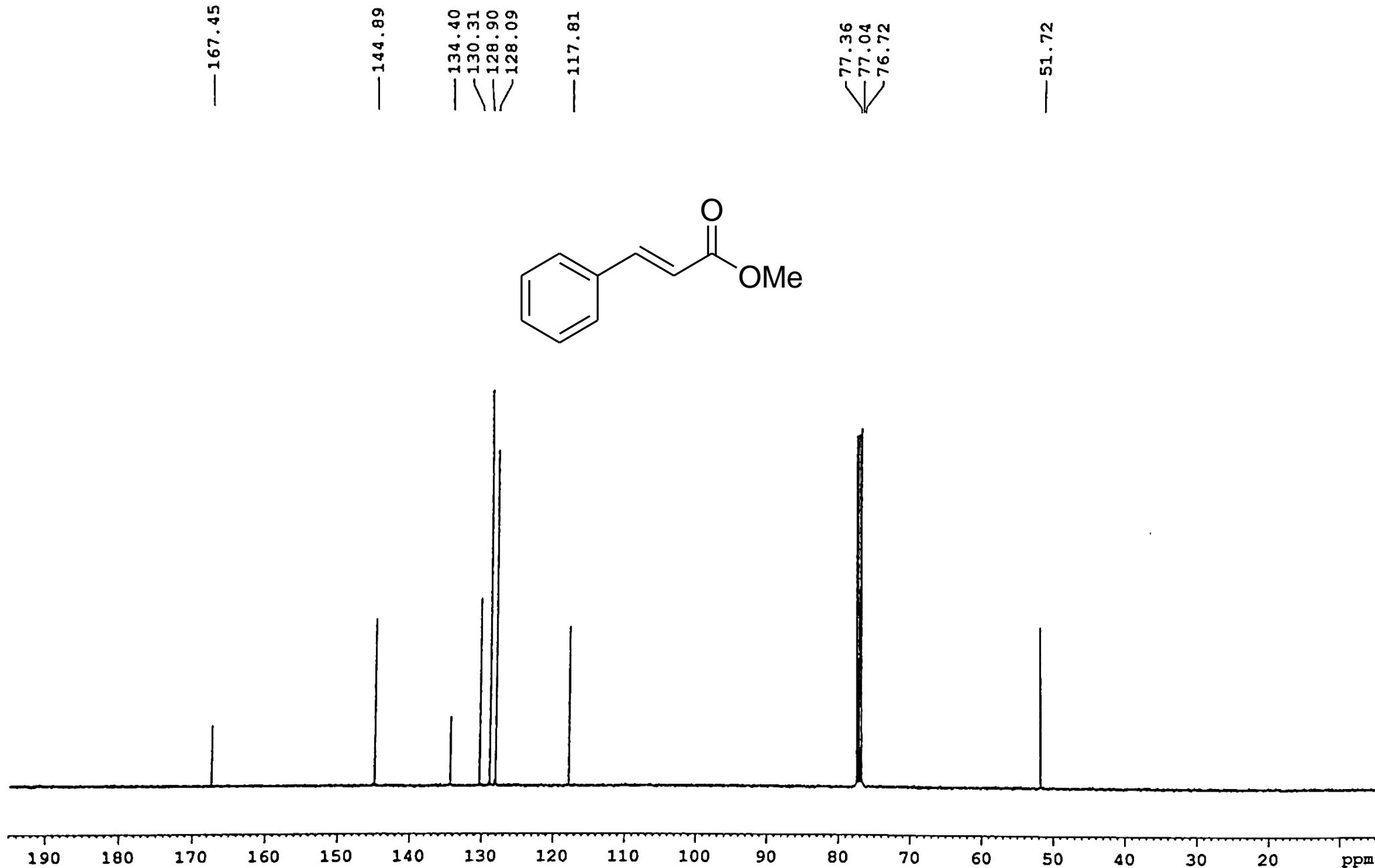
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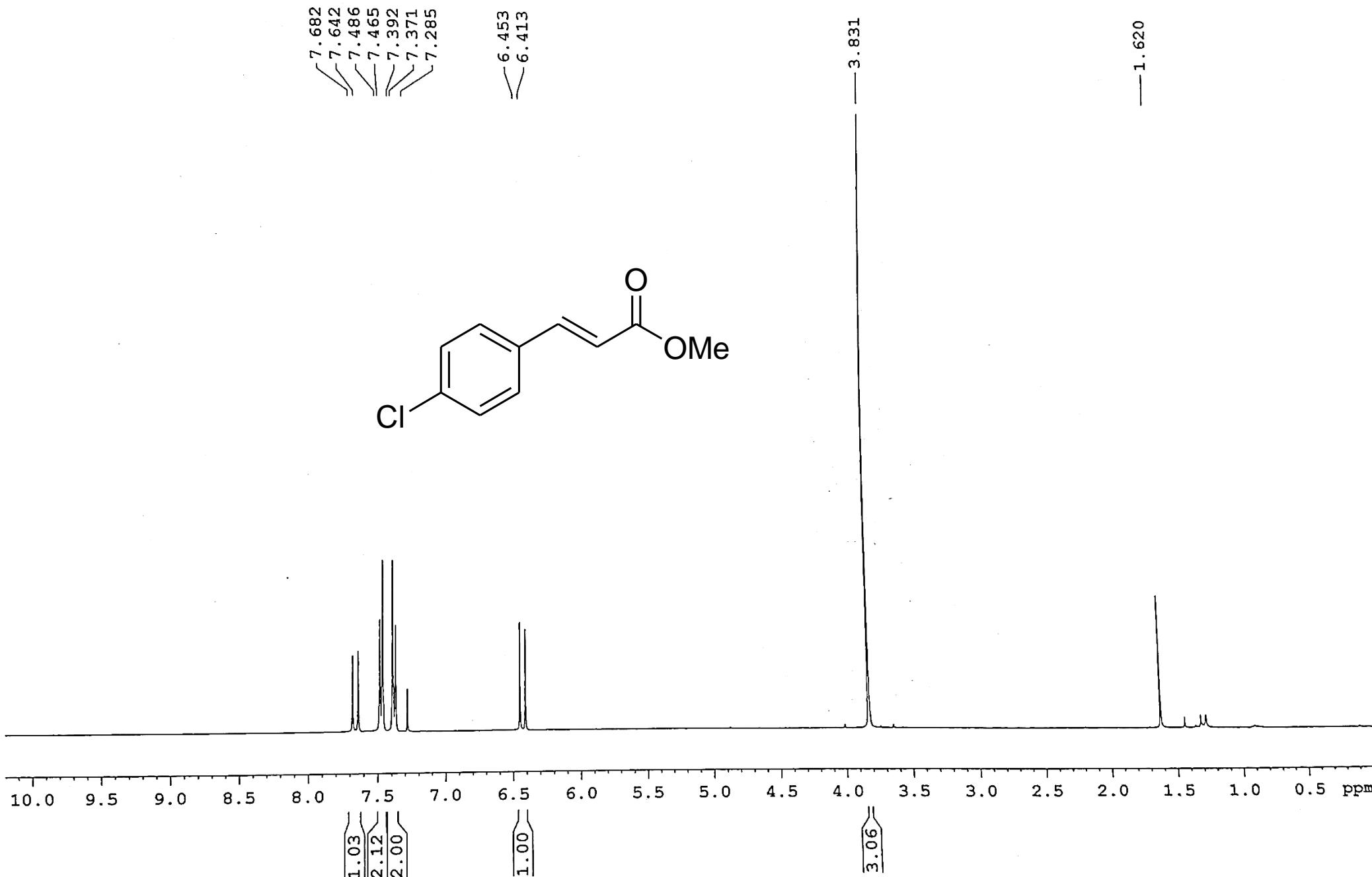
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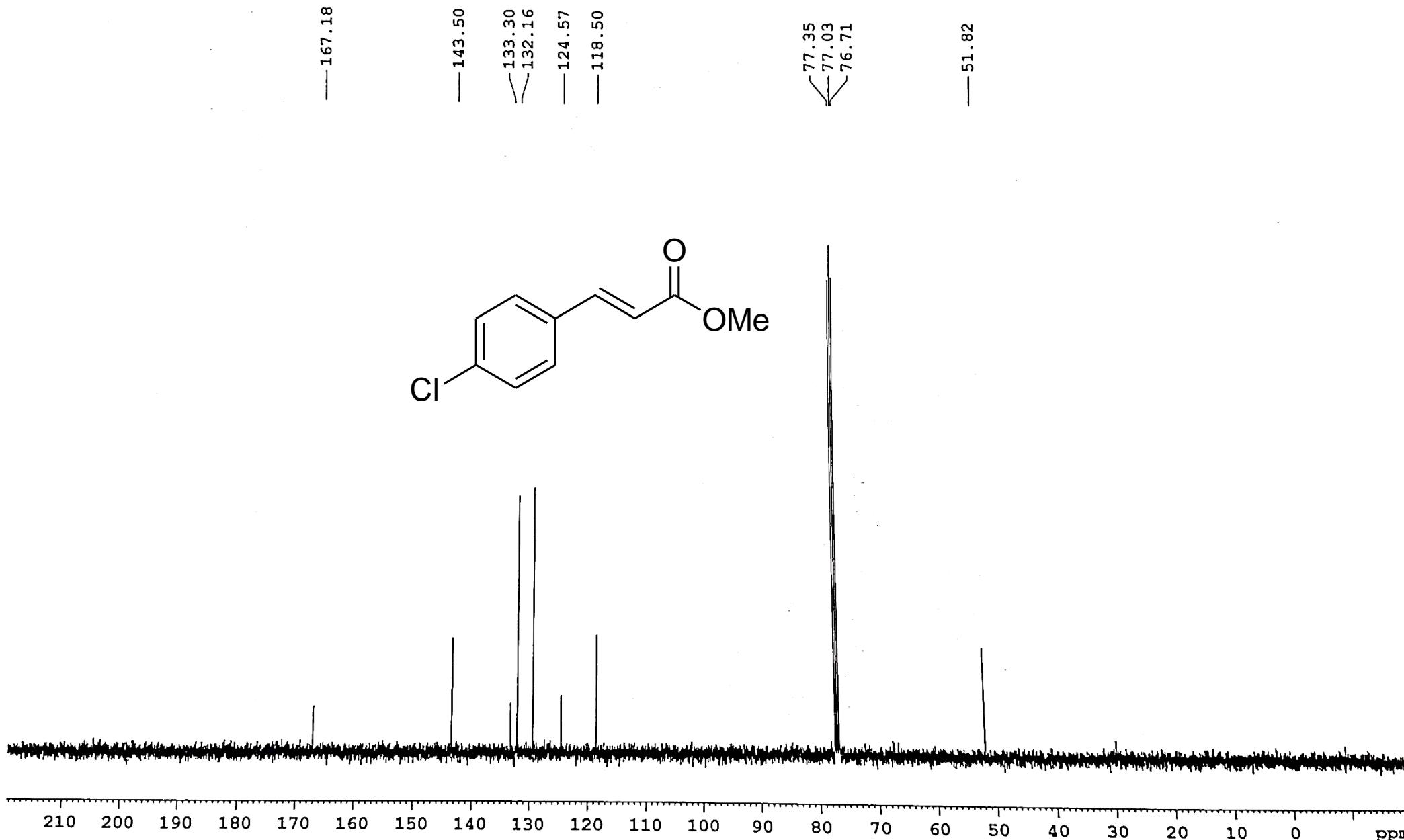
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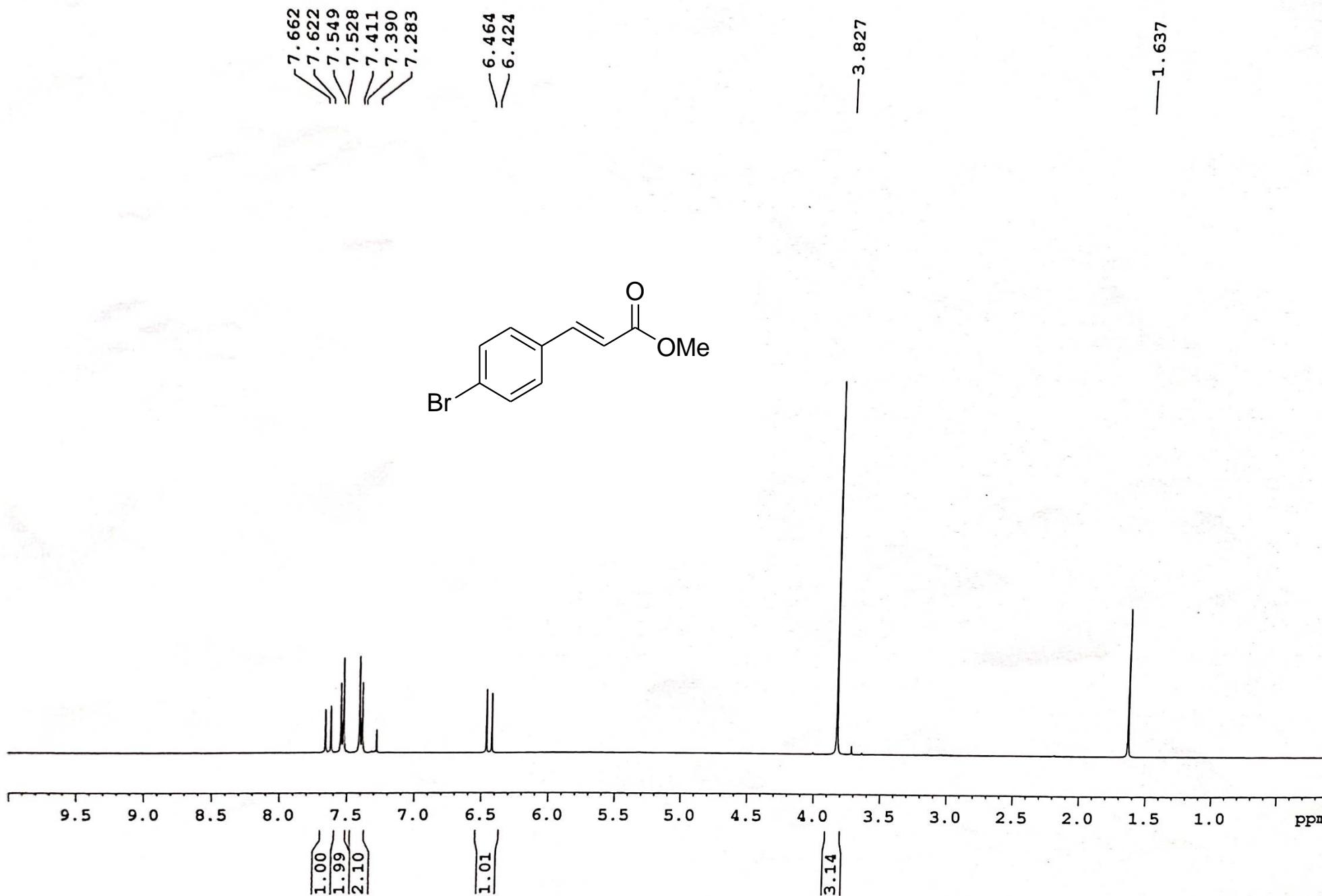
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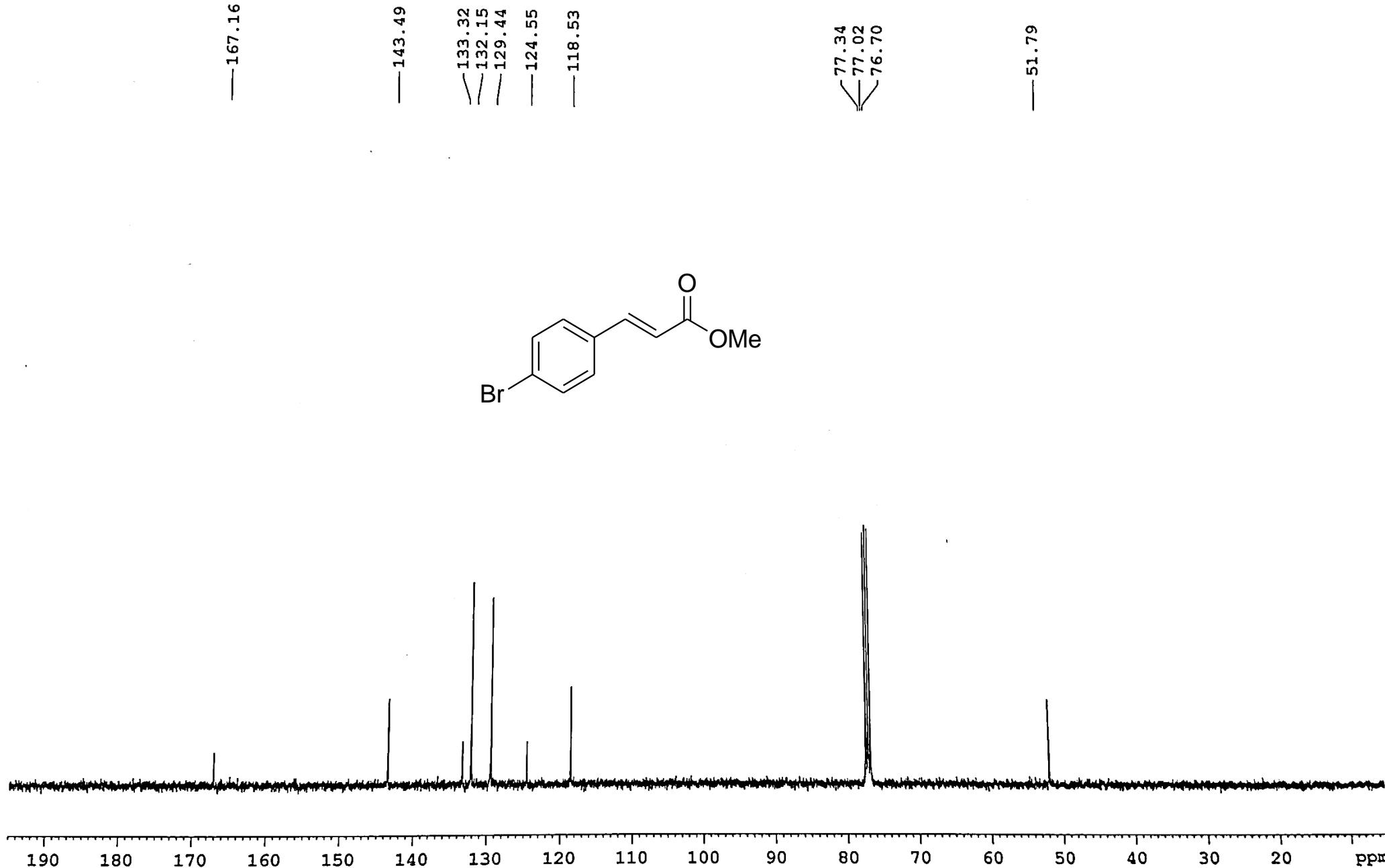
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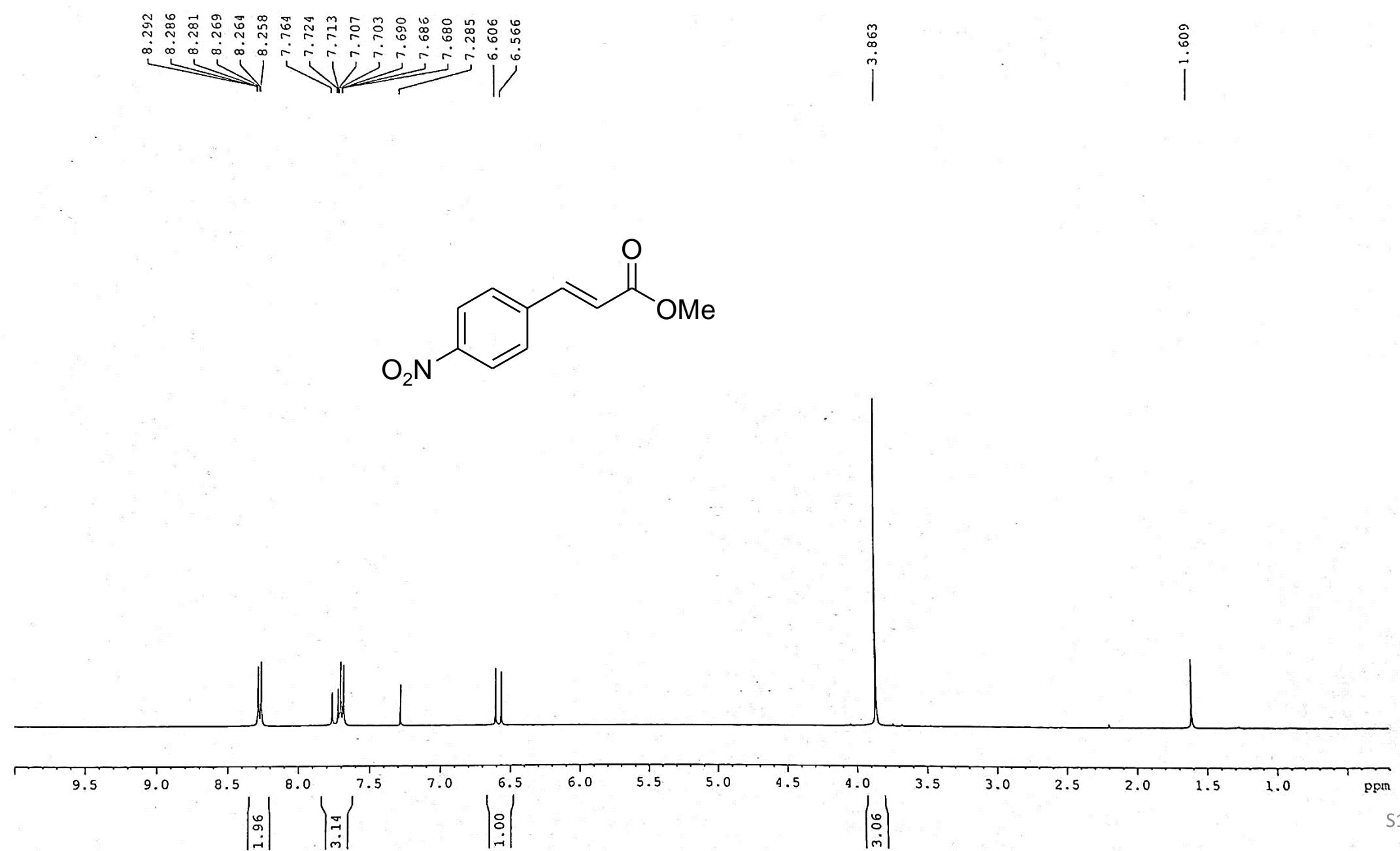
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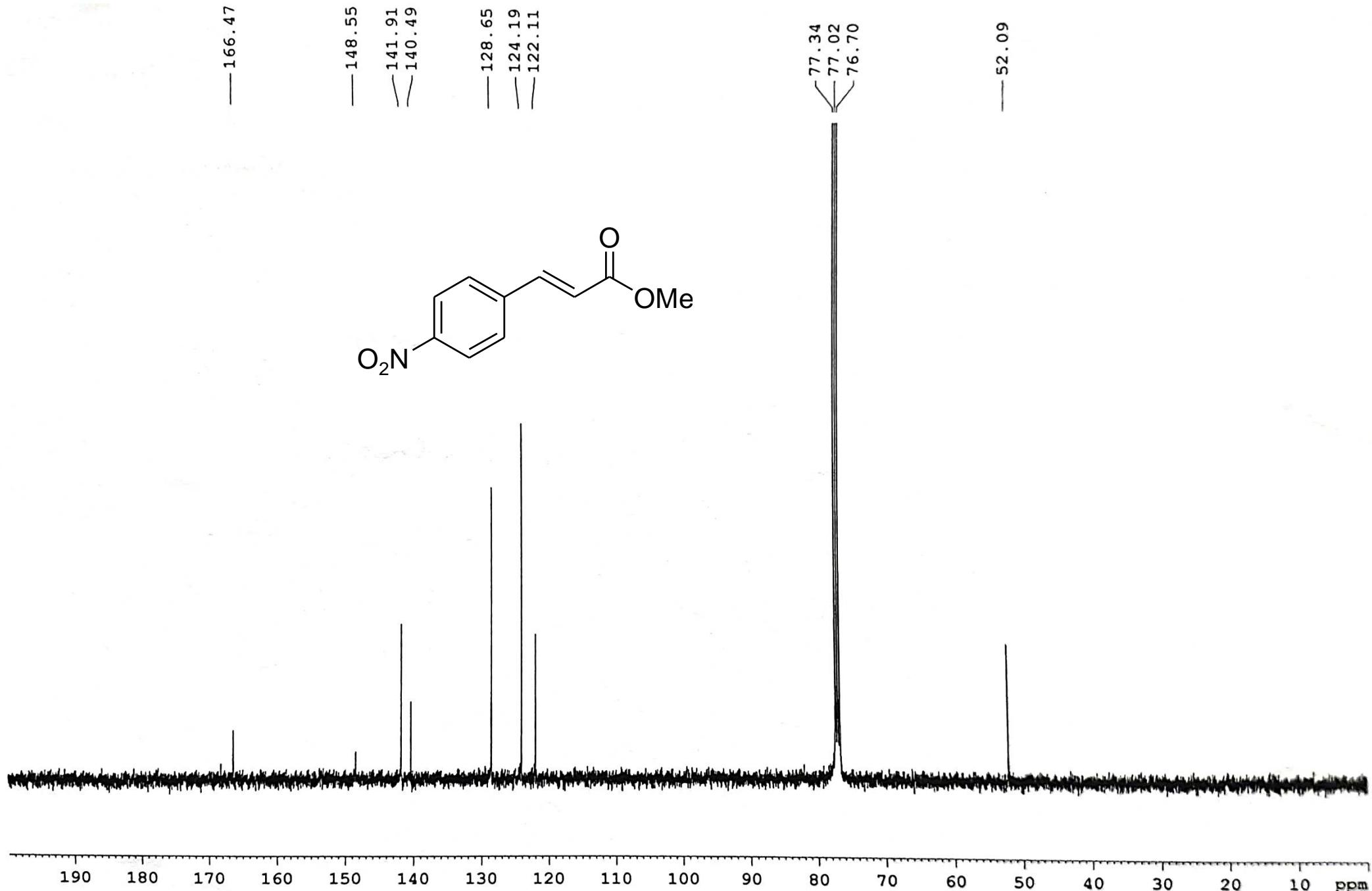
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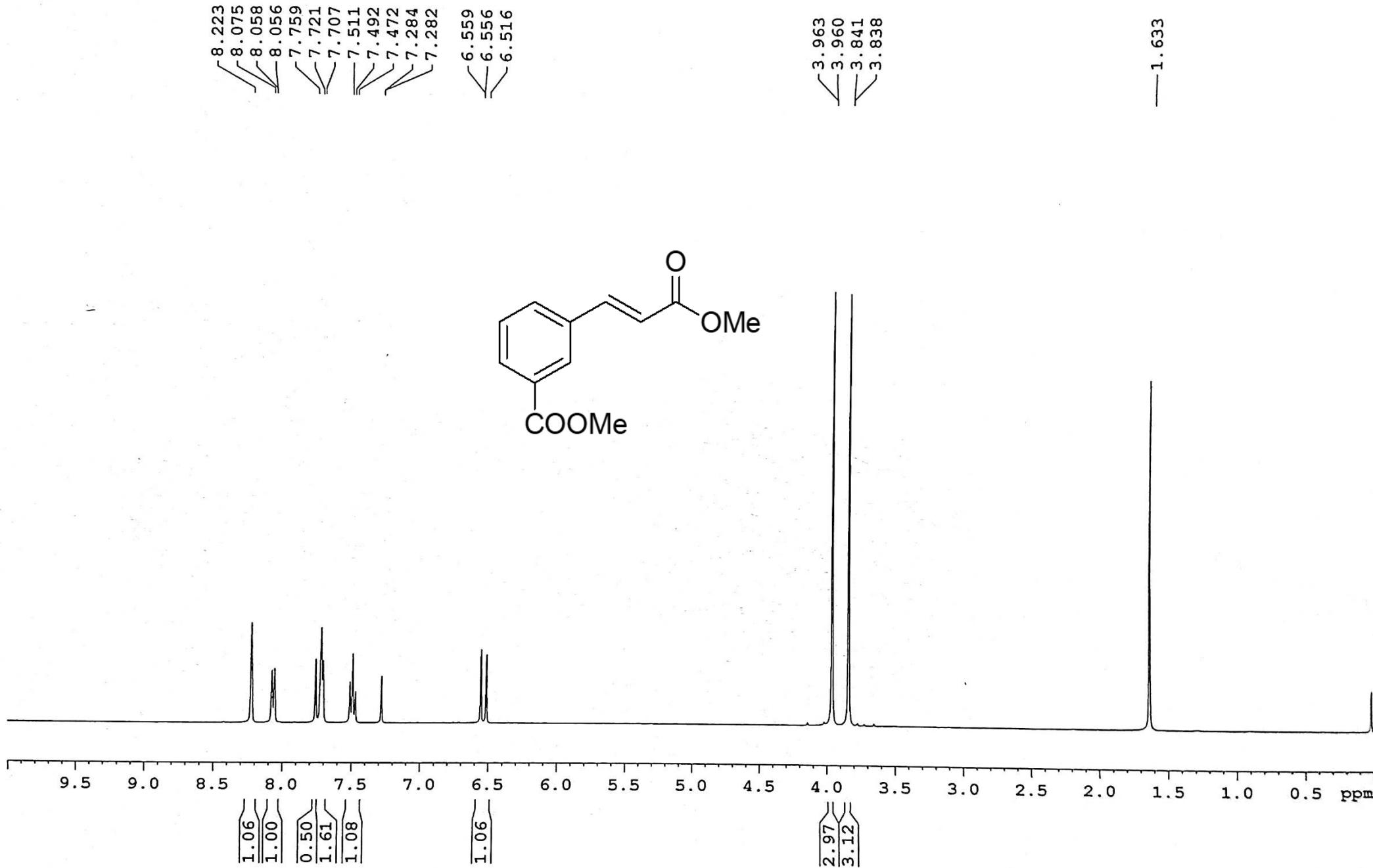
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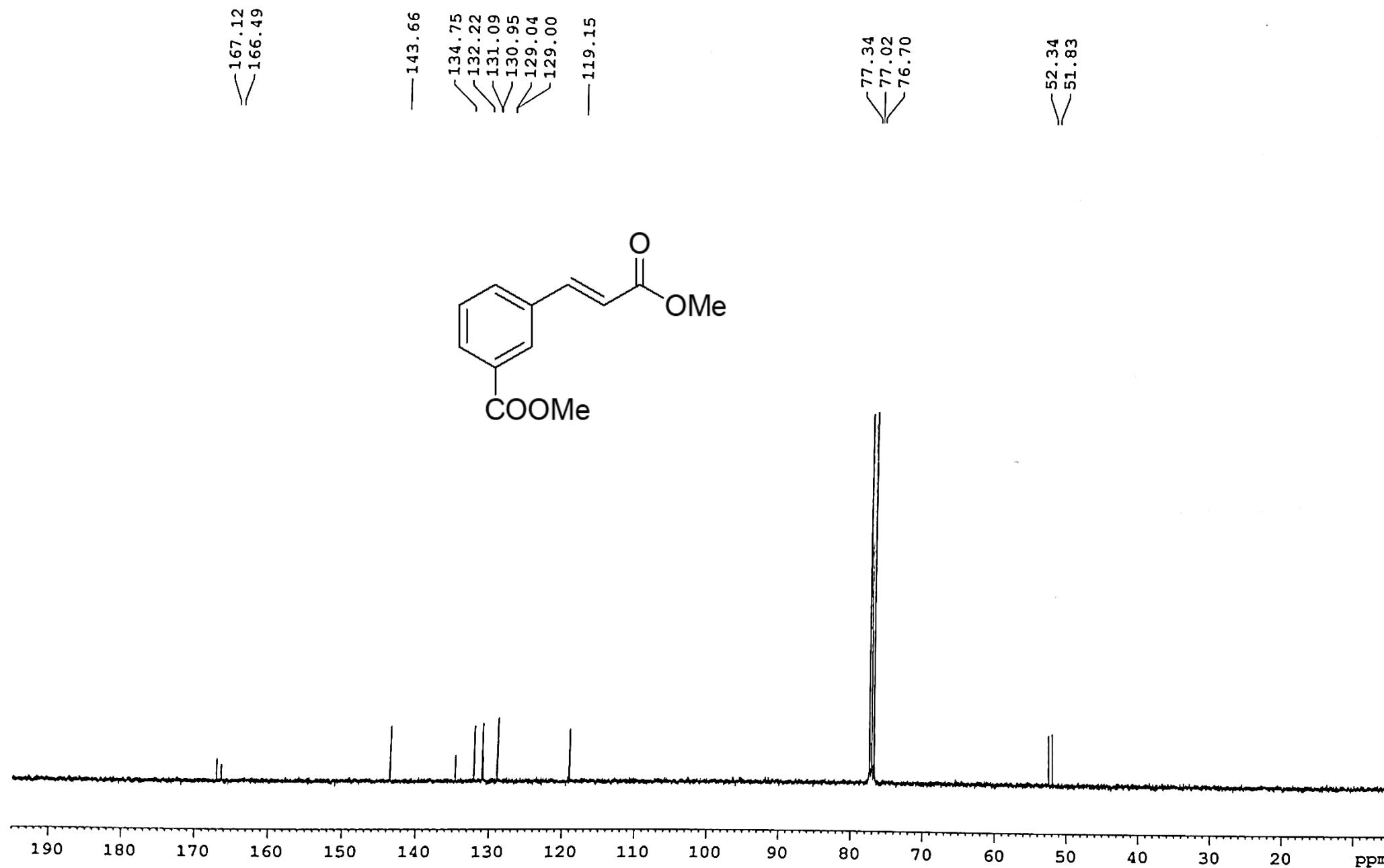
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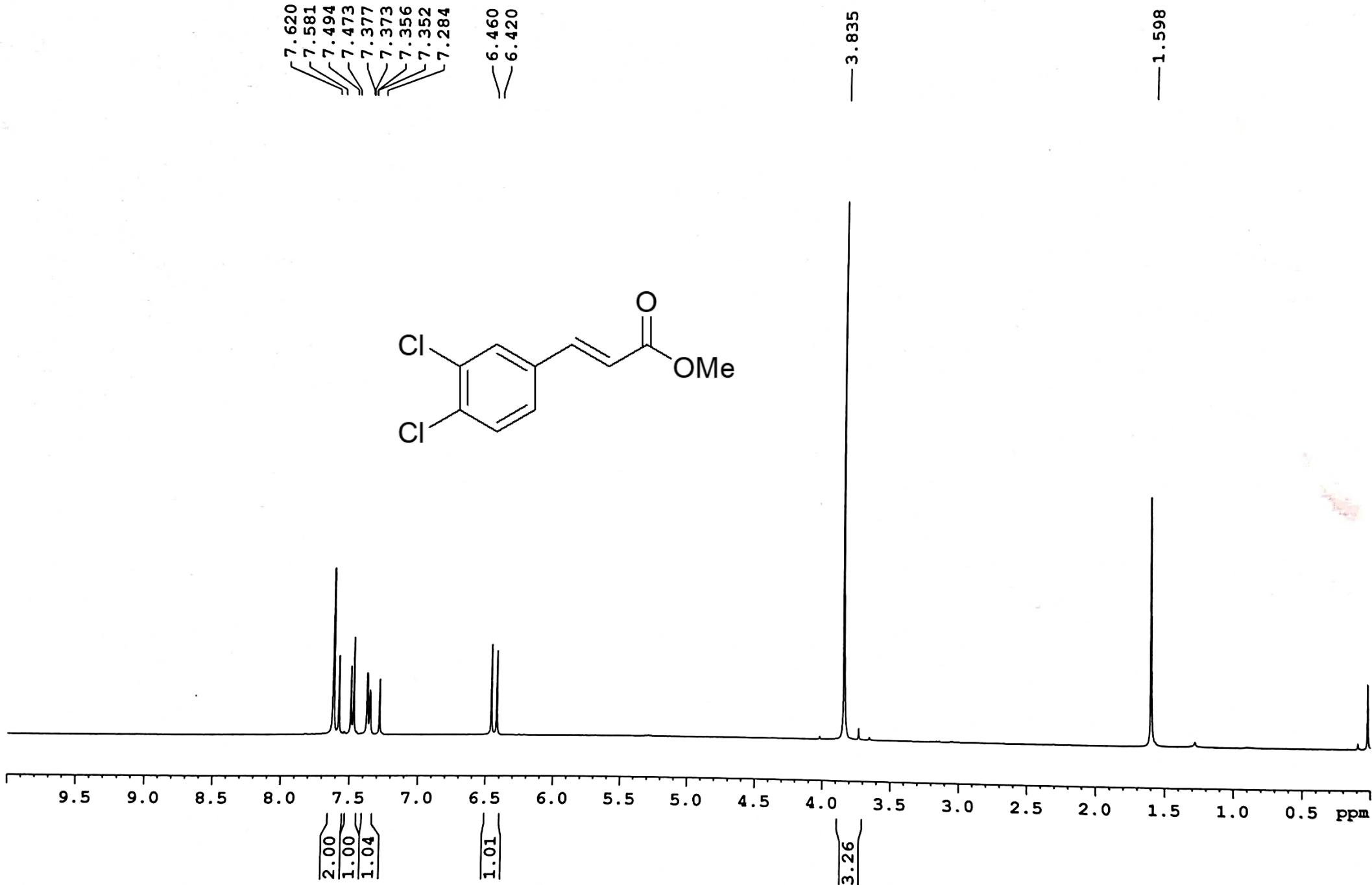
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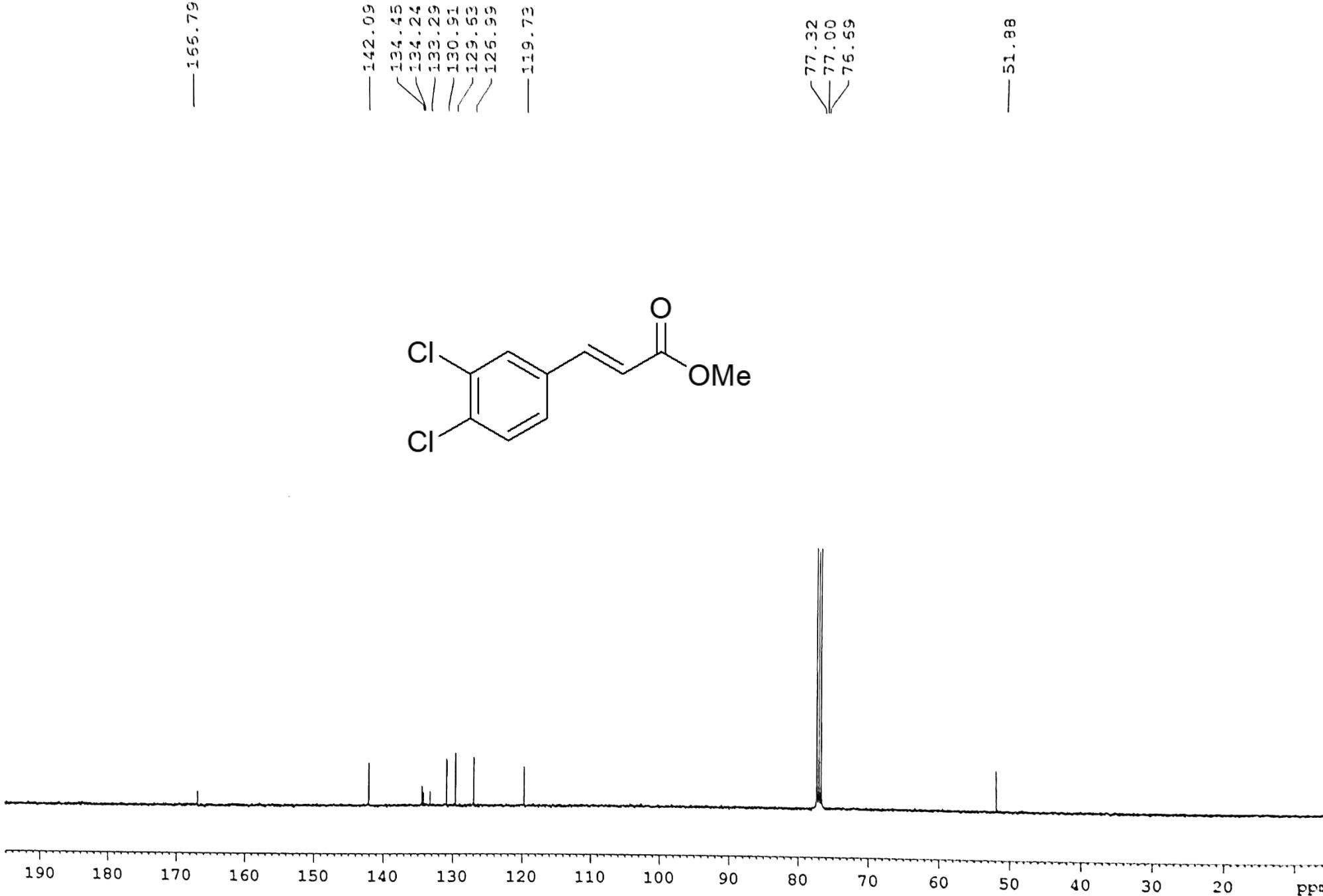
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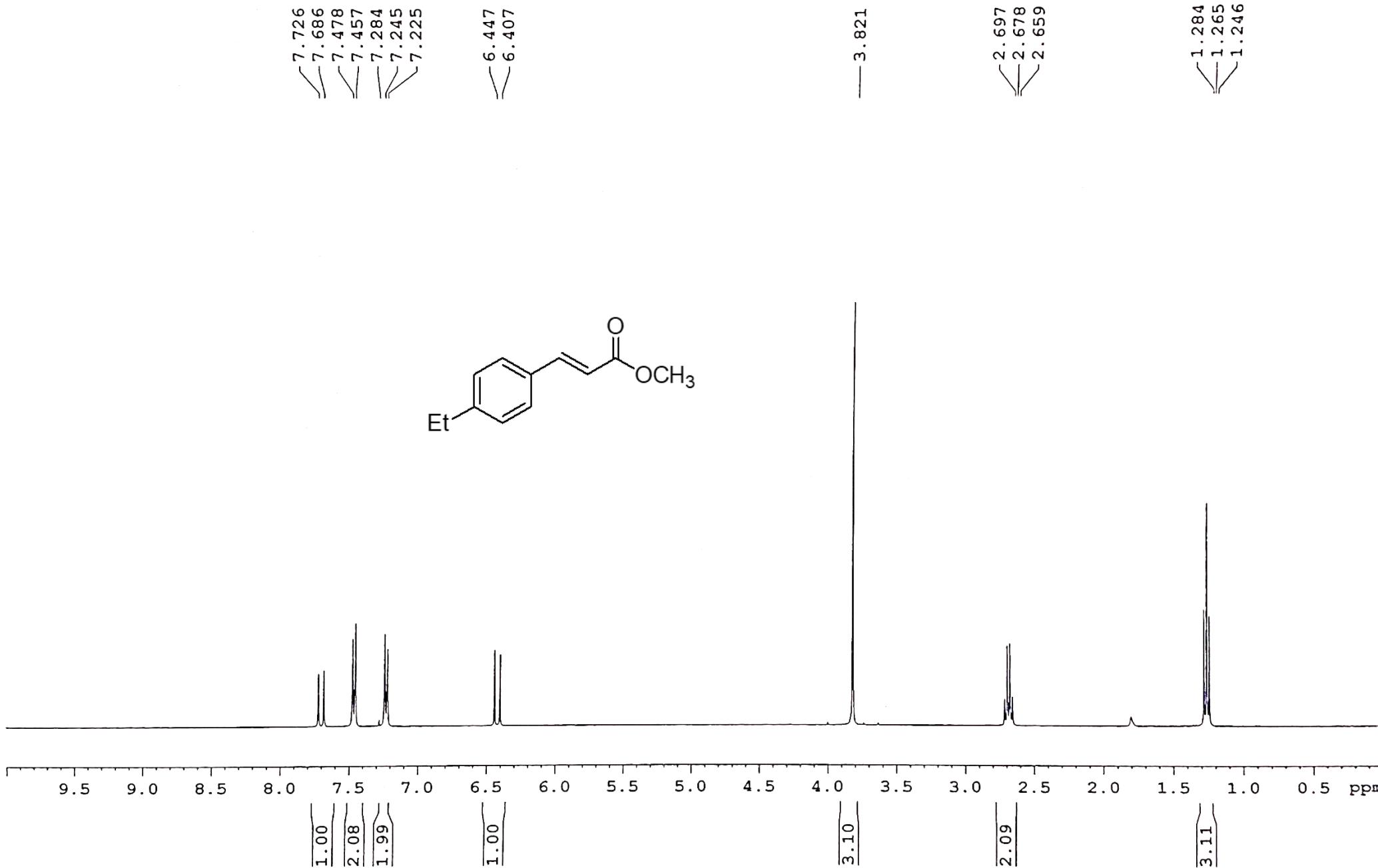
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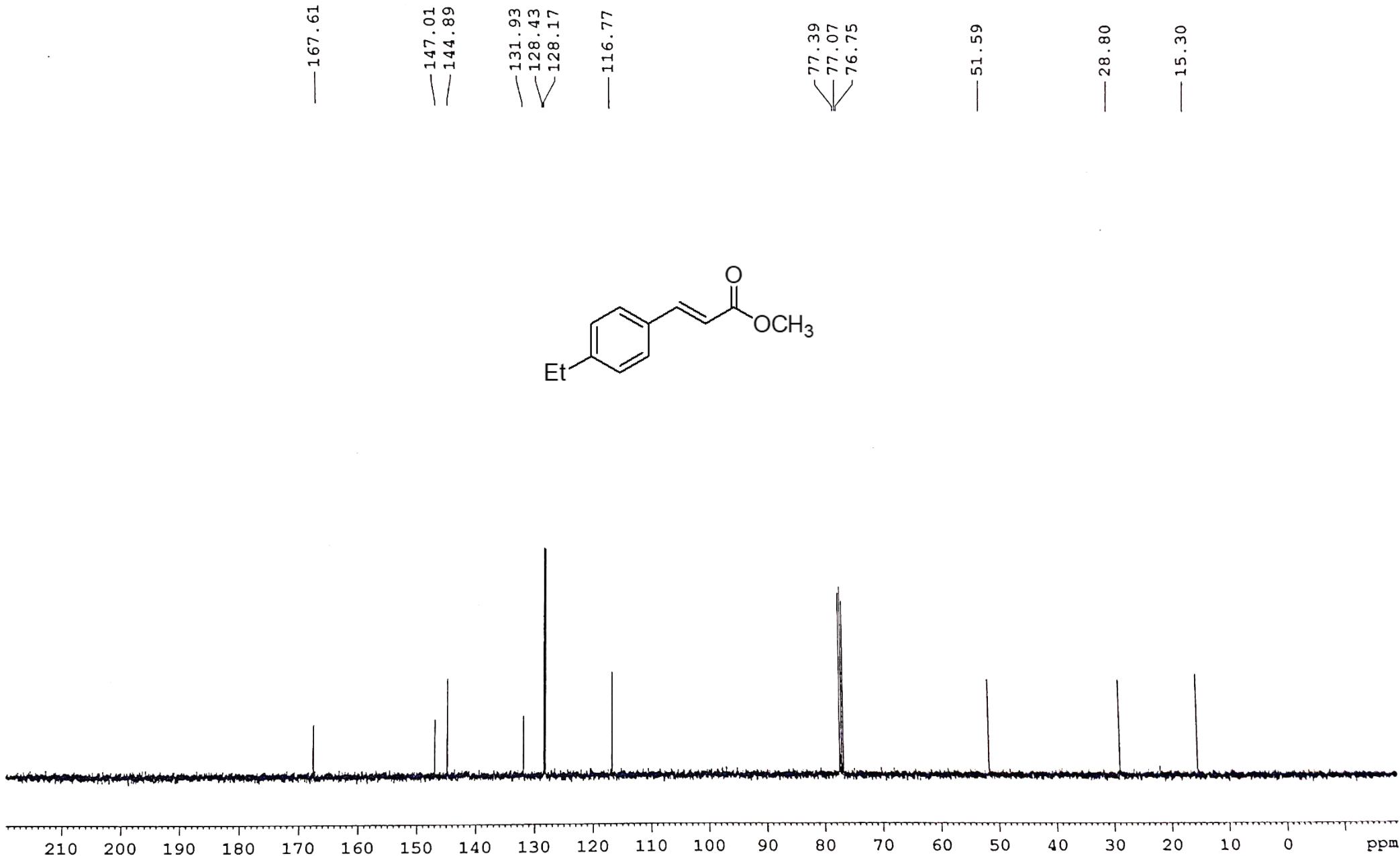
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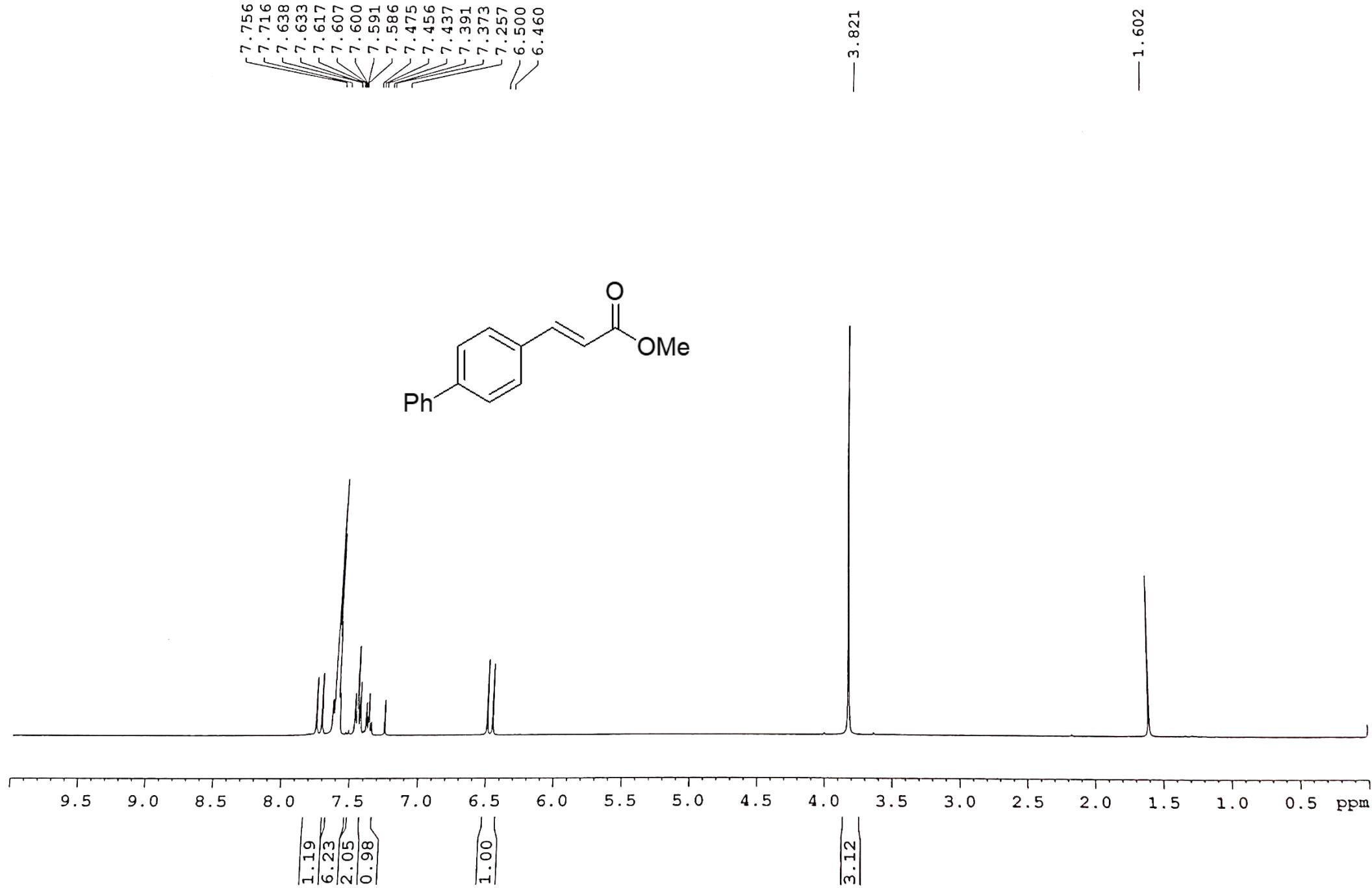
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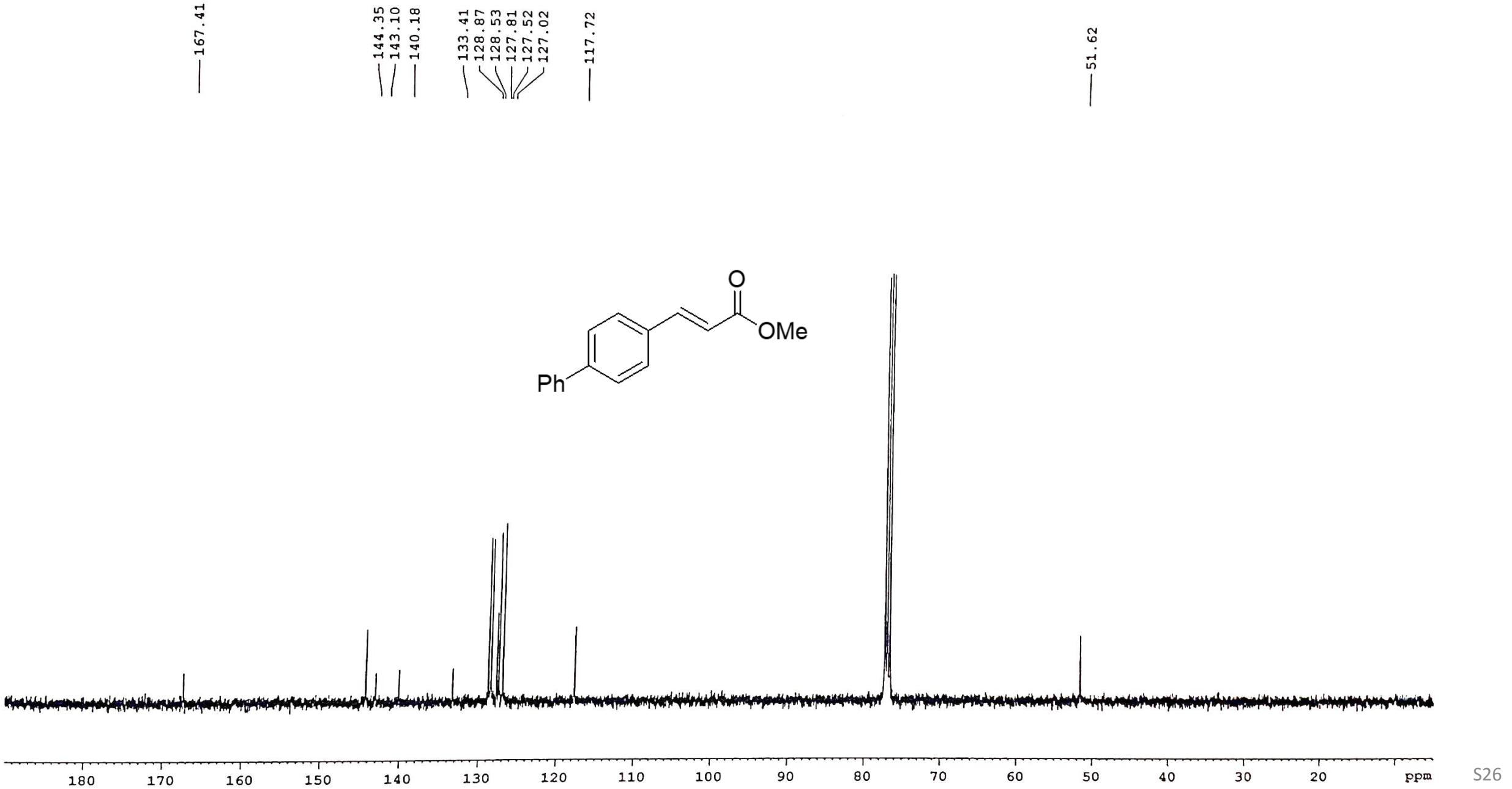
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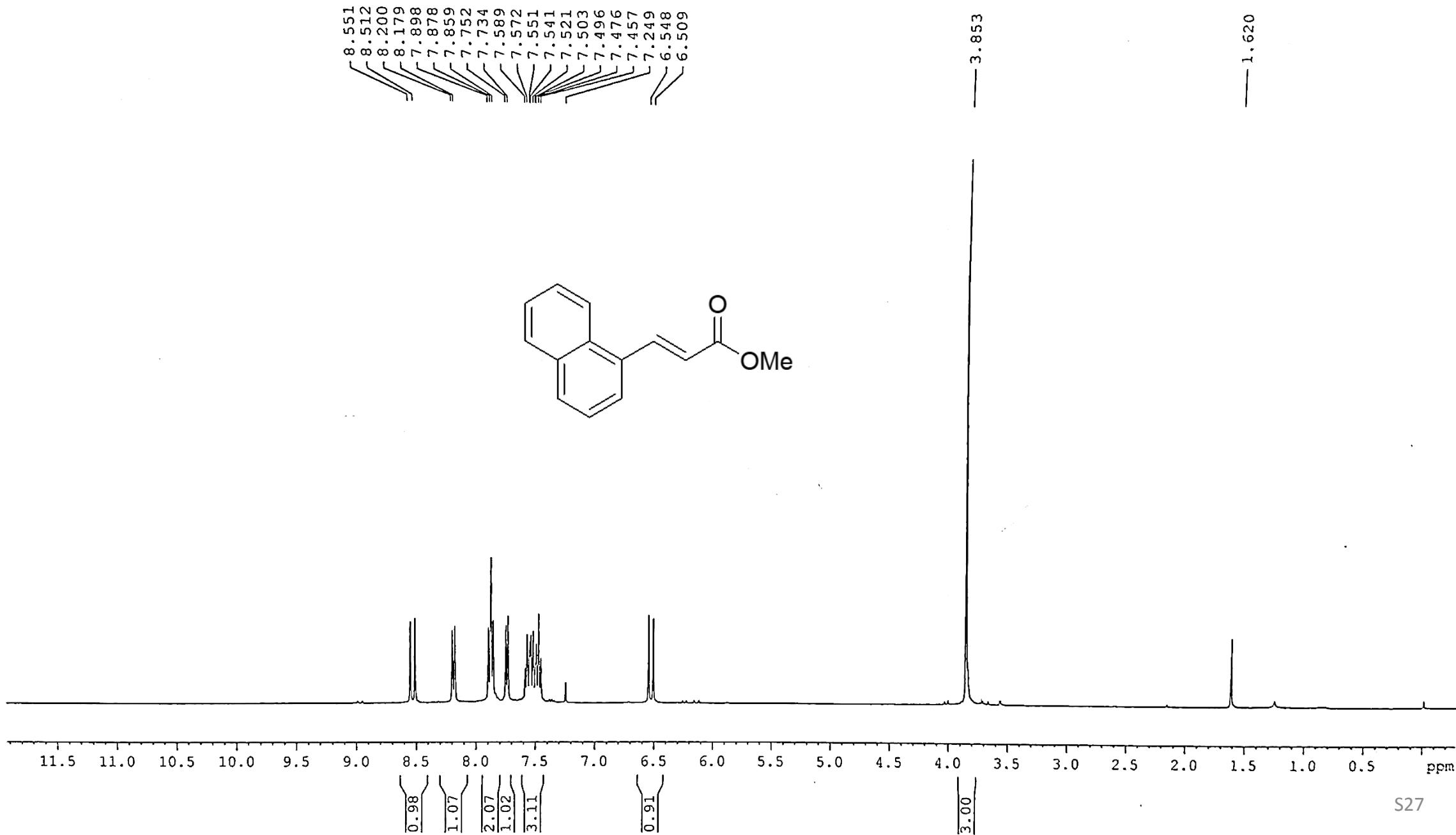
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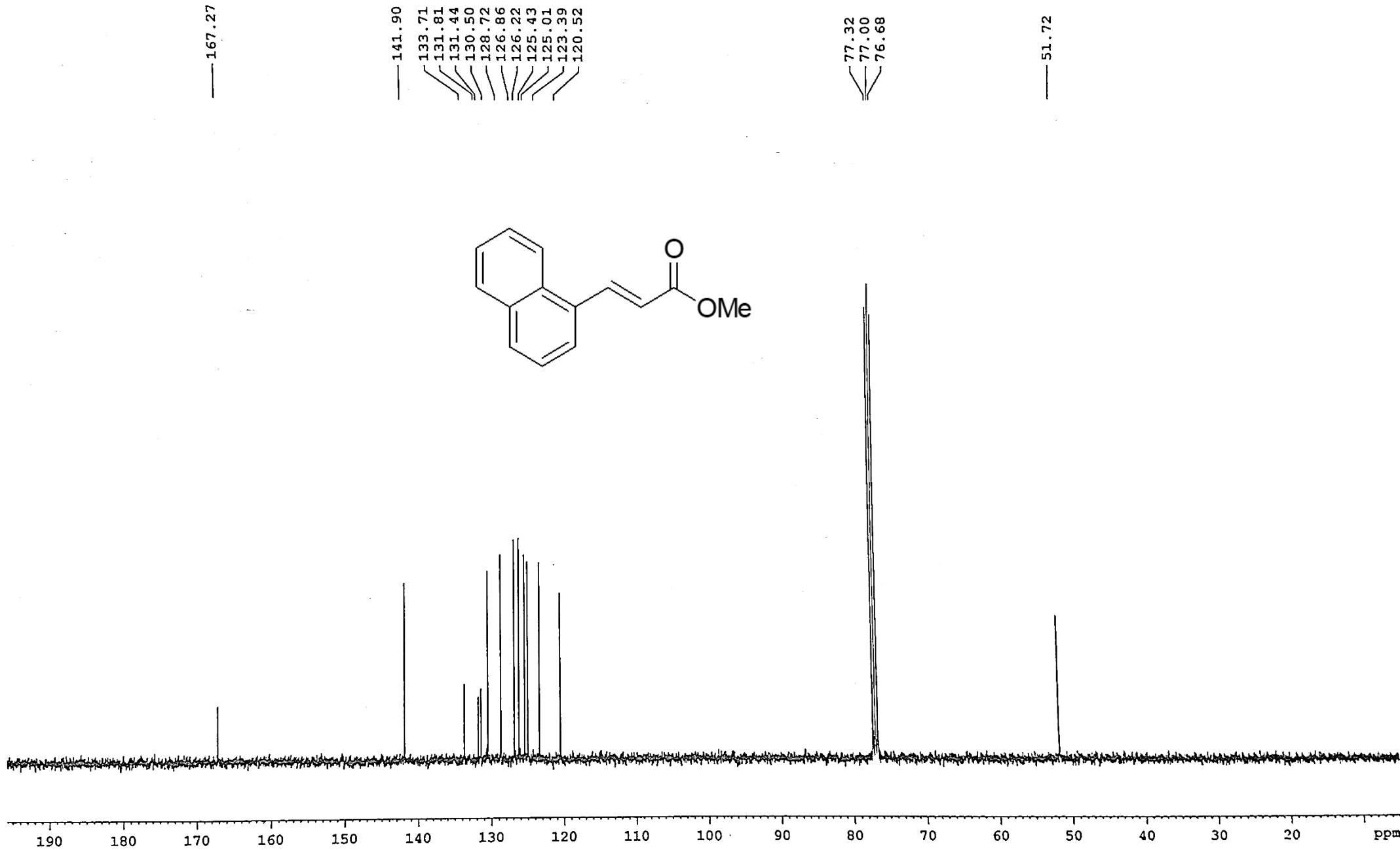
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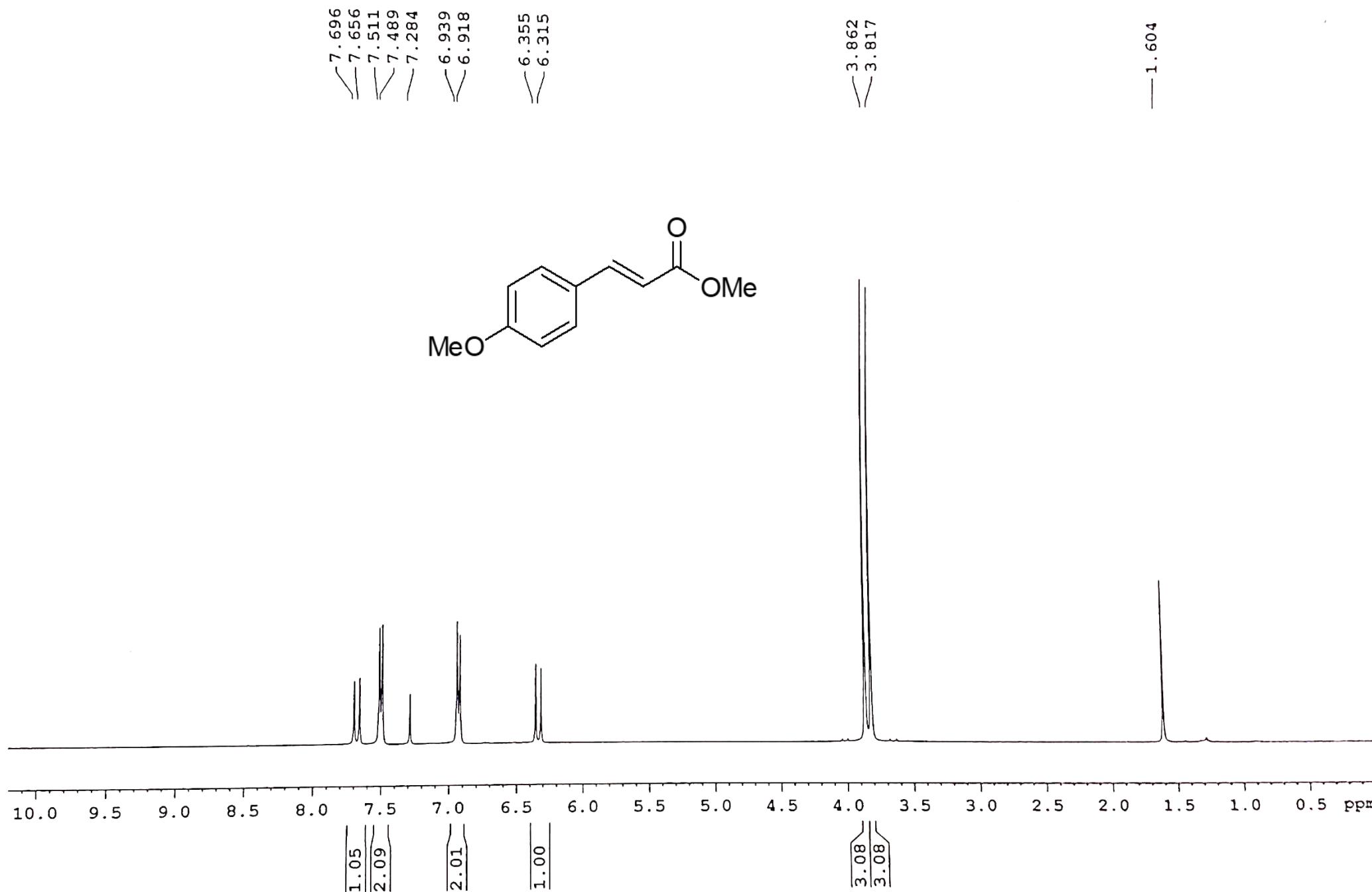
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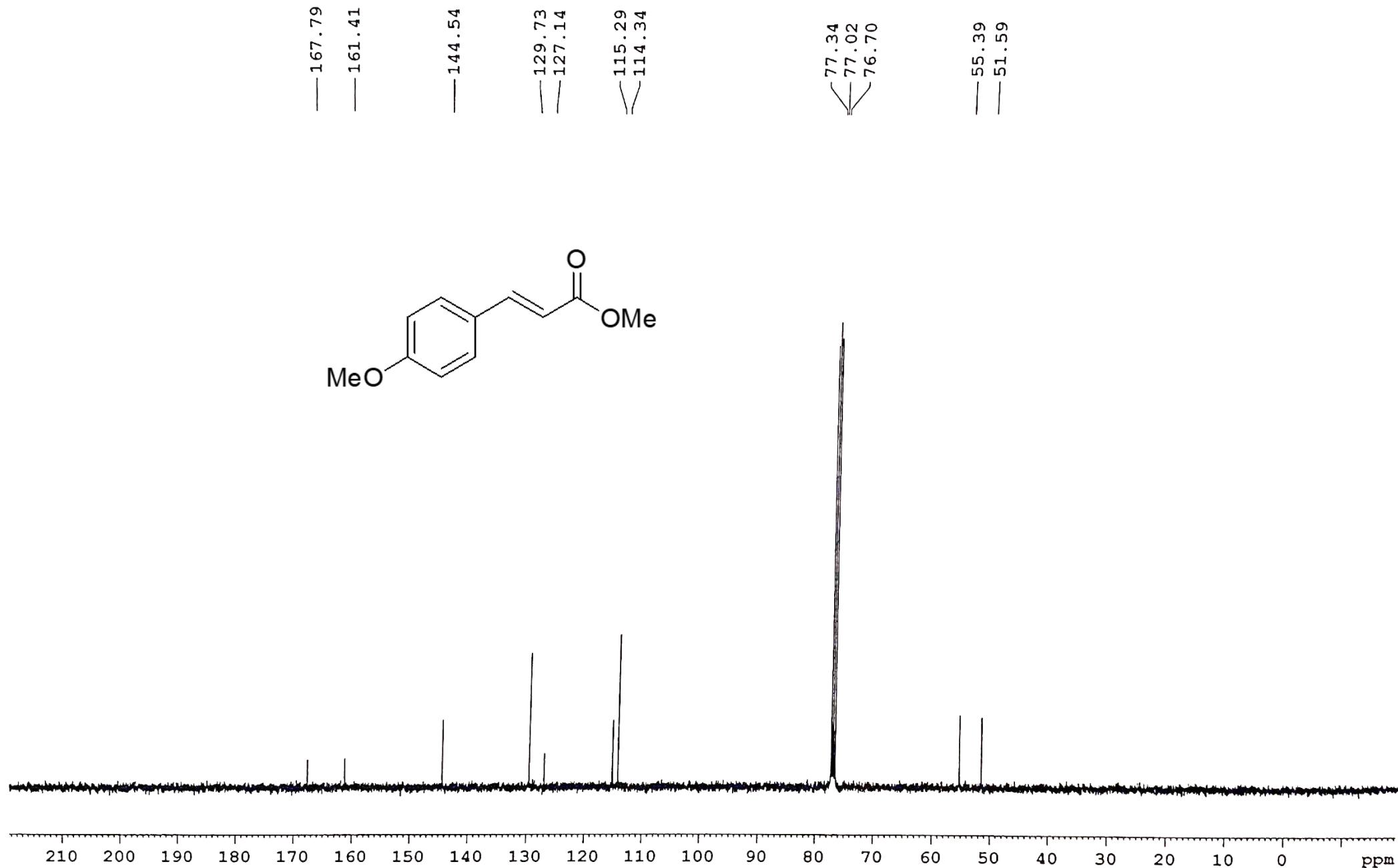
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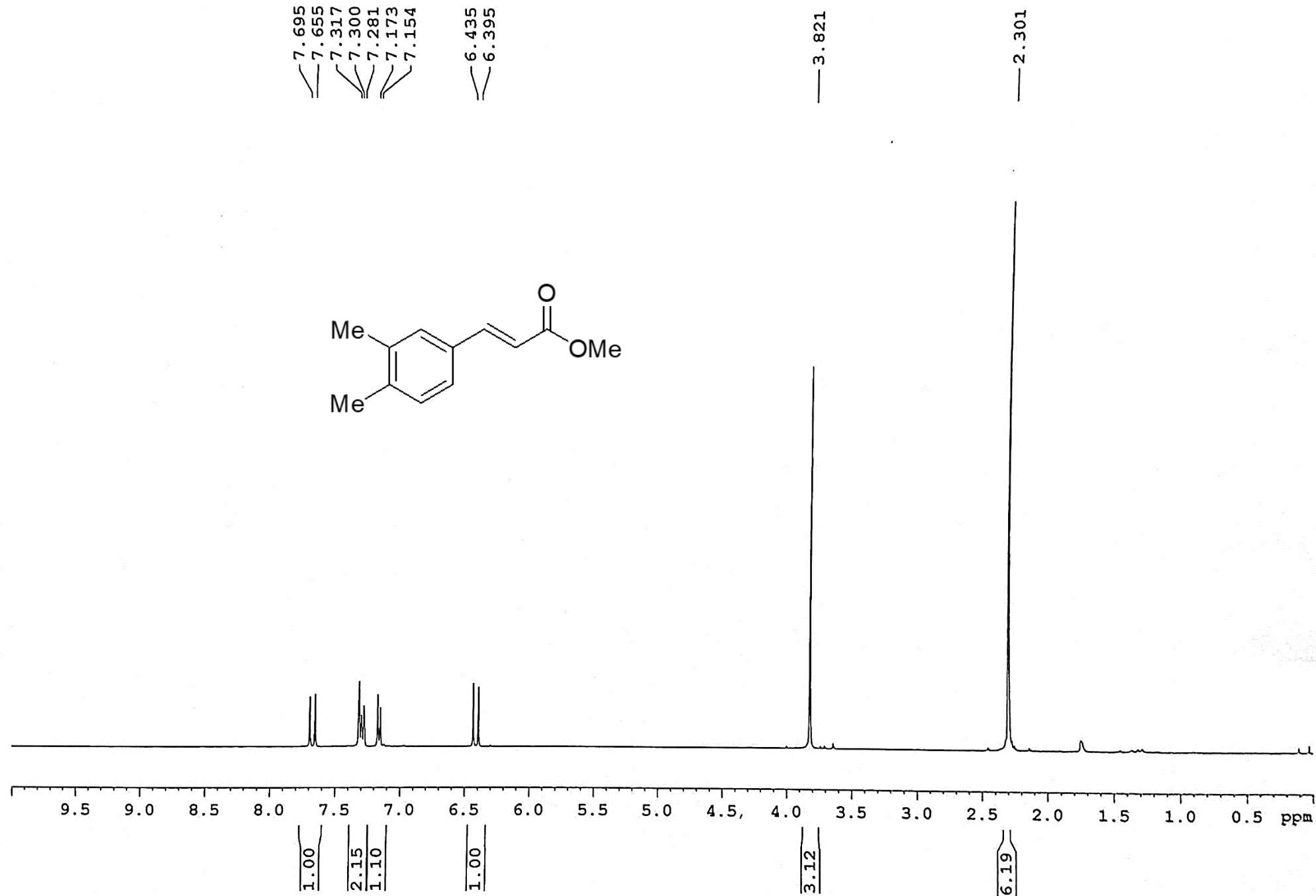
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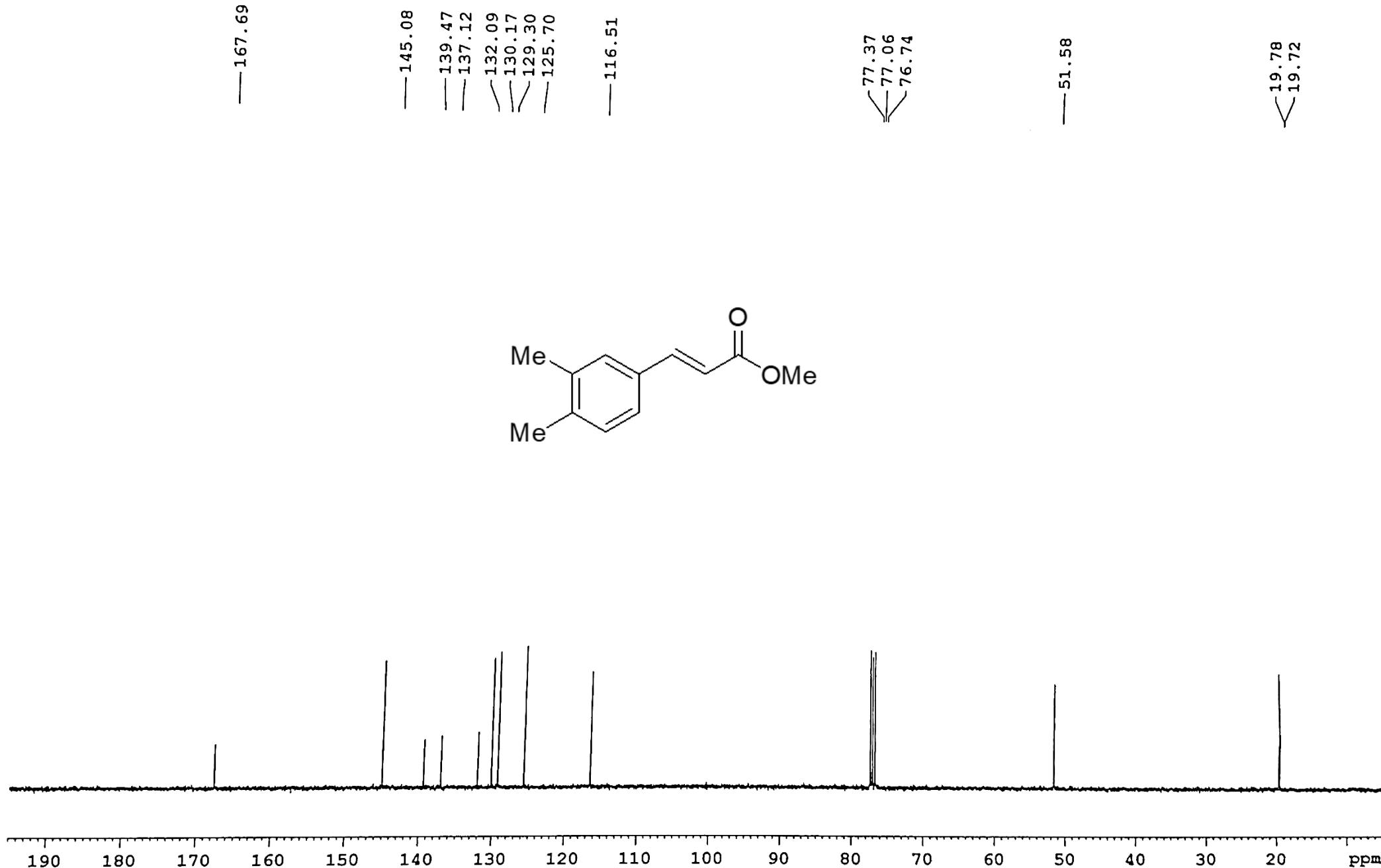
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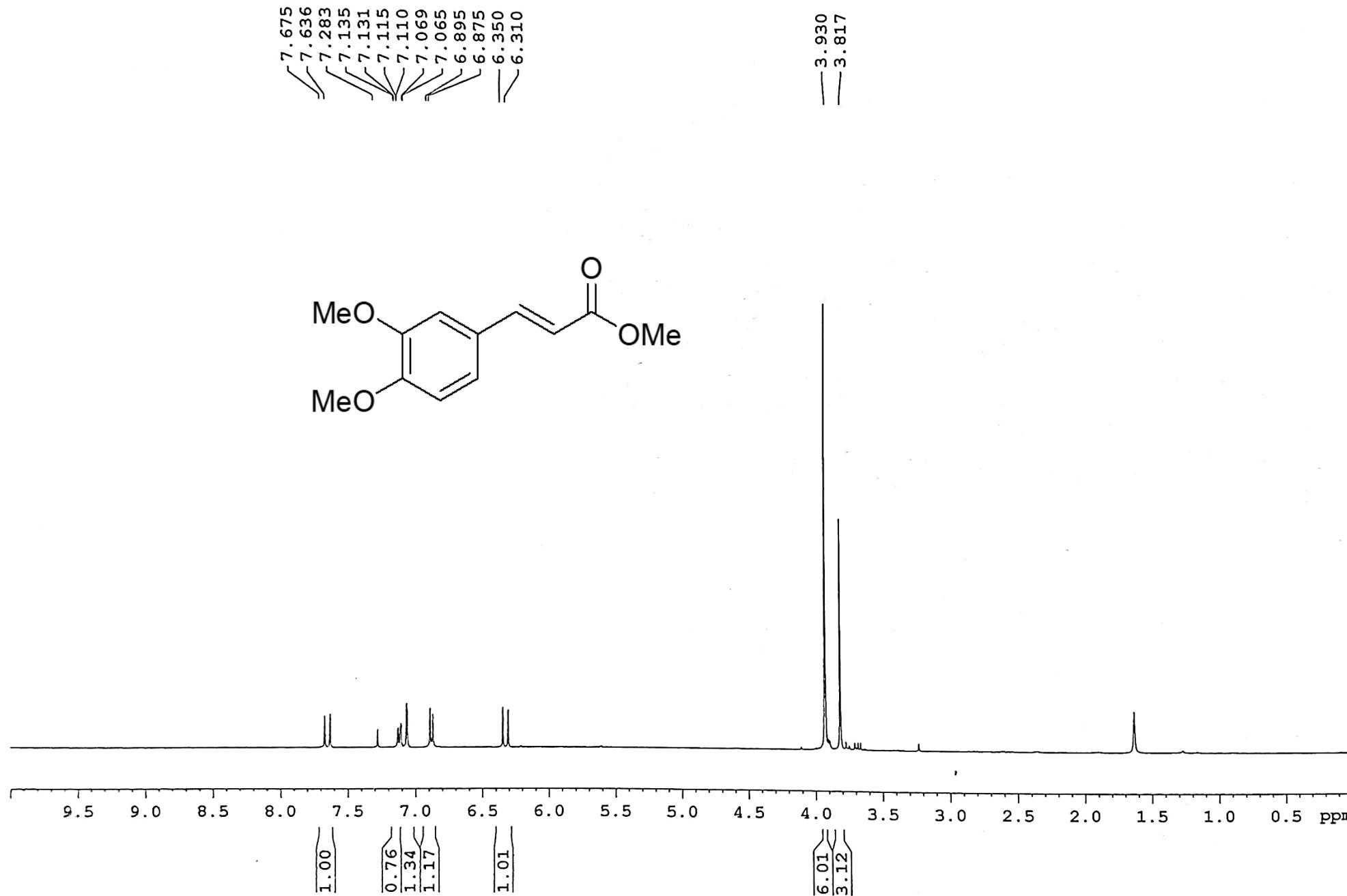
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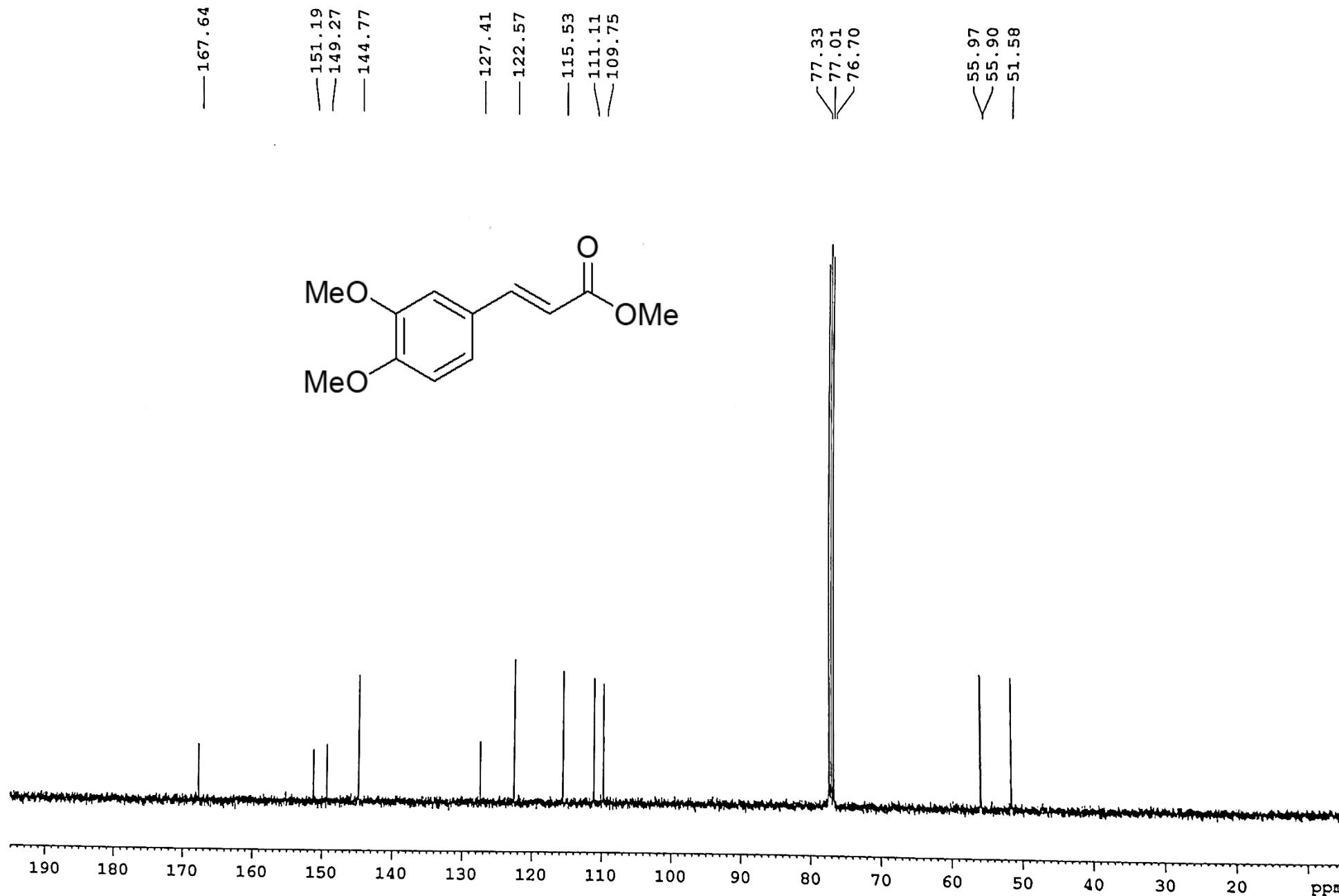
^{13}C NMR OF 3-(3,4-Dimethyl-phenyl)-acrylic acid methyl ester (3ma)



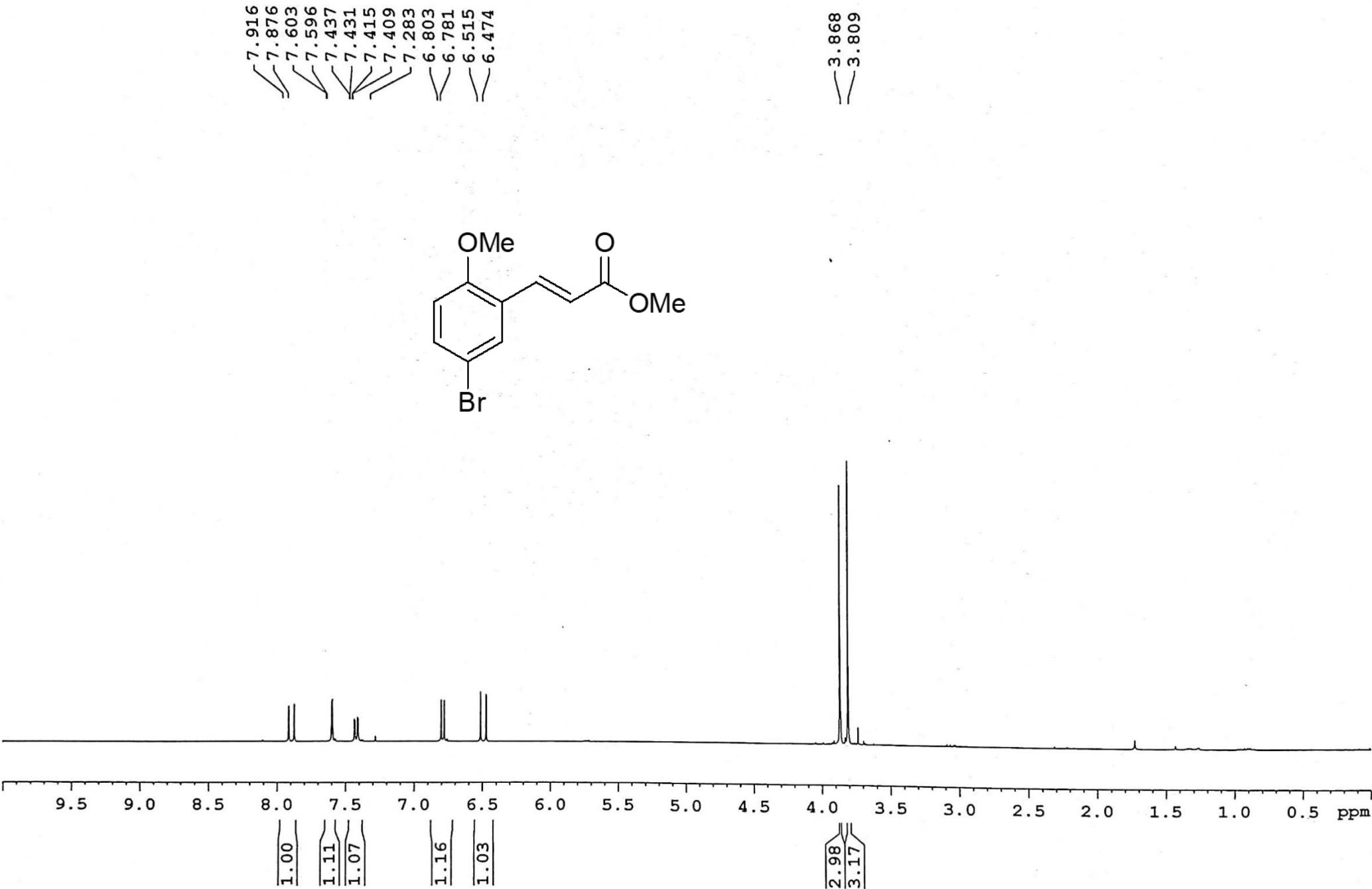
¹H NMR OF 3-(3,4-Dimethoxy-phenyl)-acrylic acid methyl ester (3na)



¹³C NMR OF 3-(3,4-Dimethoxy-phenyl)-acrylic acid methyl ester (3na)

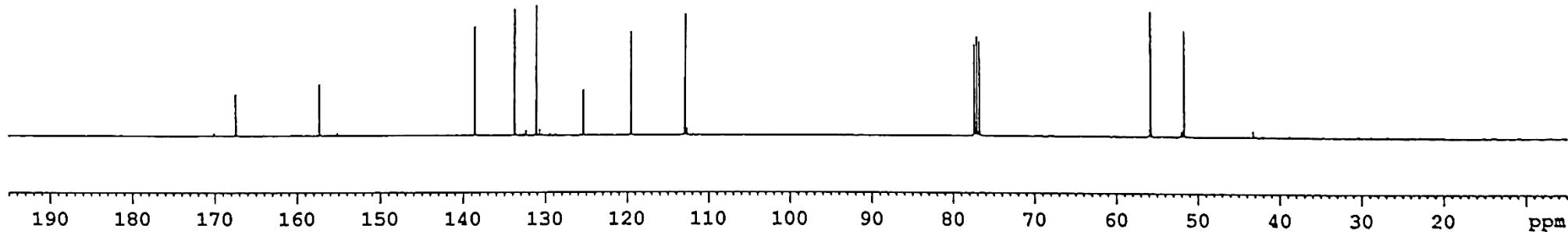
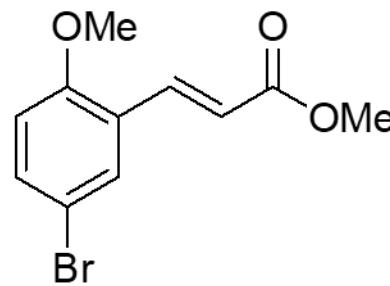


¹H NMR OF 3-(5-Bromo-2-methoxy-phenyl)-acrylic acid methyl ester (3oa)

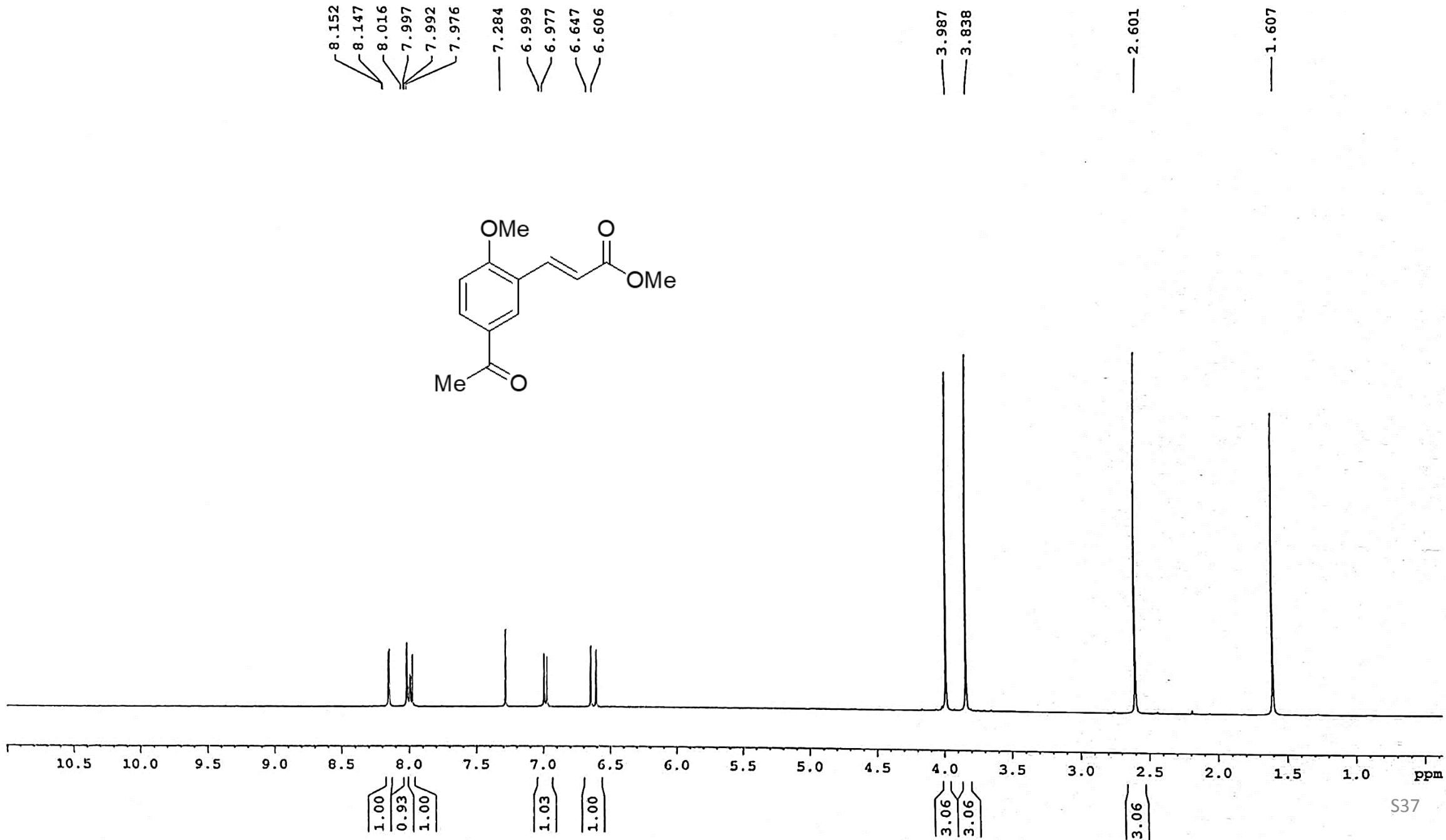


¹³C NMR OF 3-(5-Bromo-2-methoxy-phenyl)-acrylic acid methyl ester (3oa)

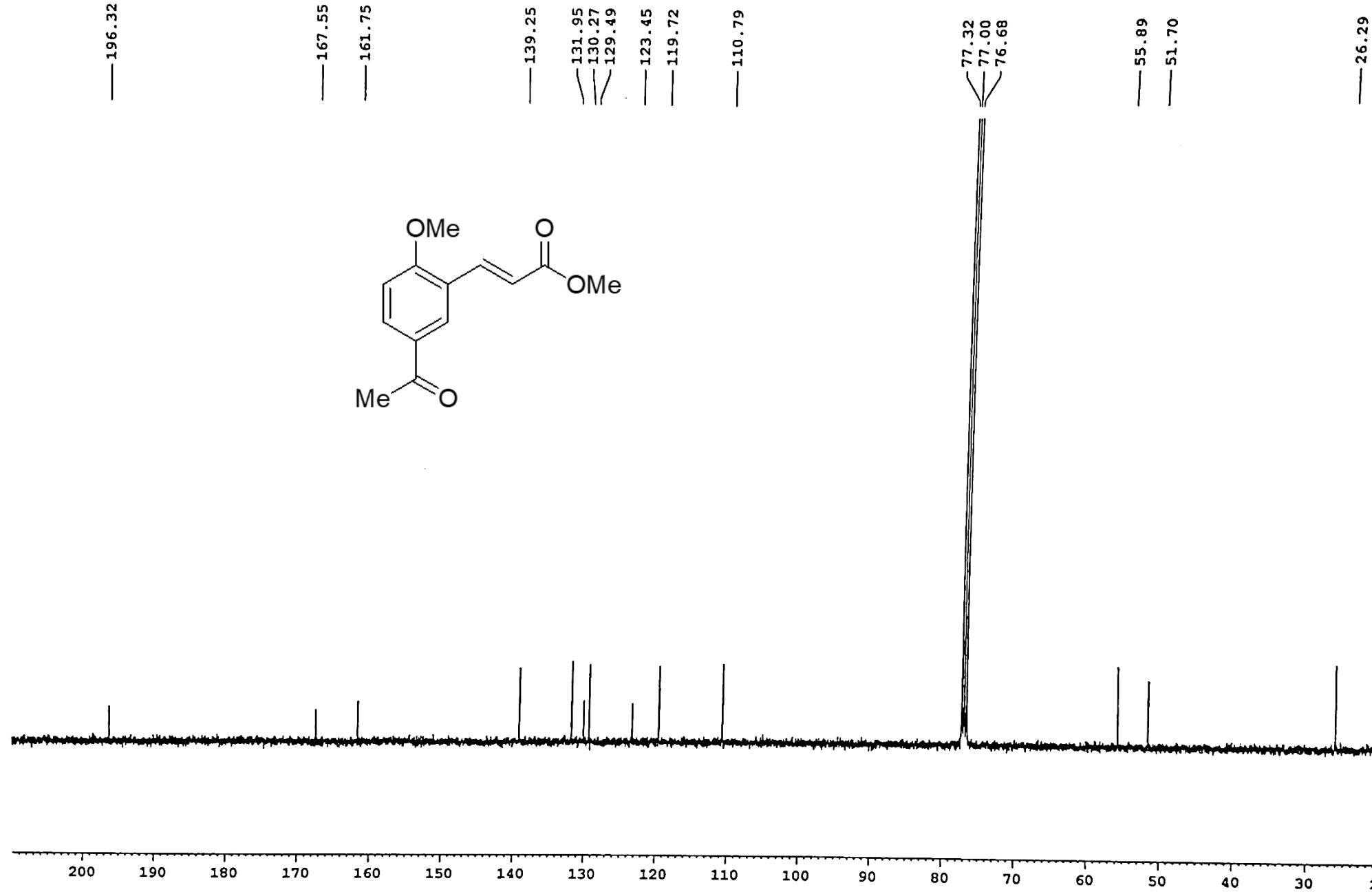
—167.41
—157.27
—138.61
—133.77
—131.12
—125.39
—119.55
—112.99
—112.91
—77.38
—77.06
—76.74
—55.77
—51.66



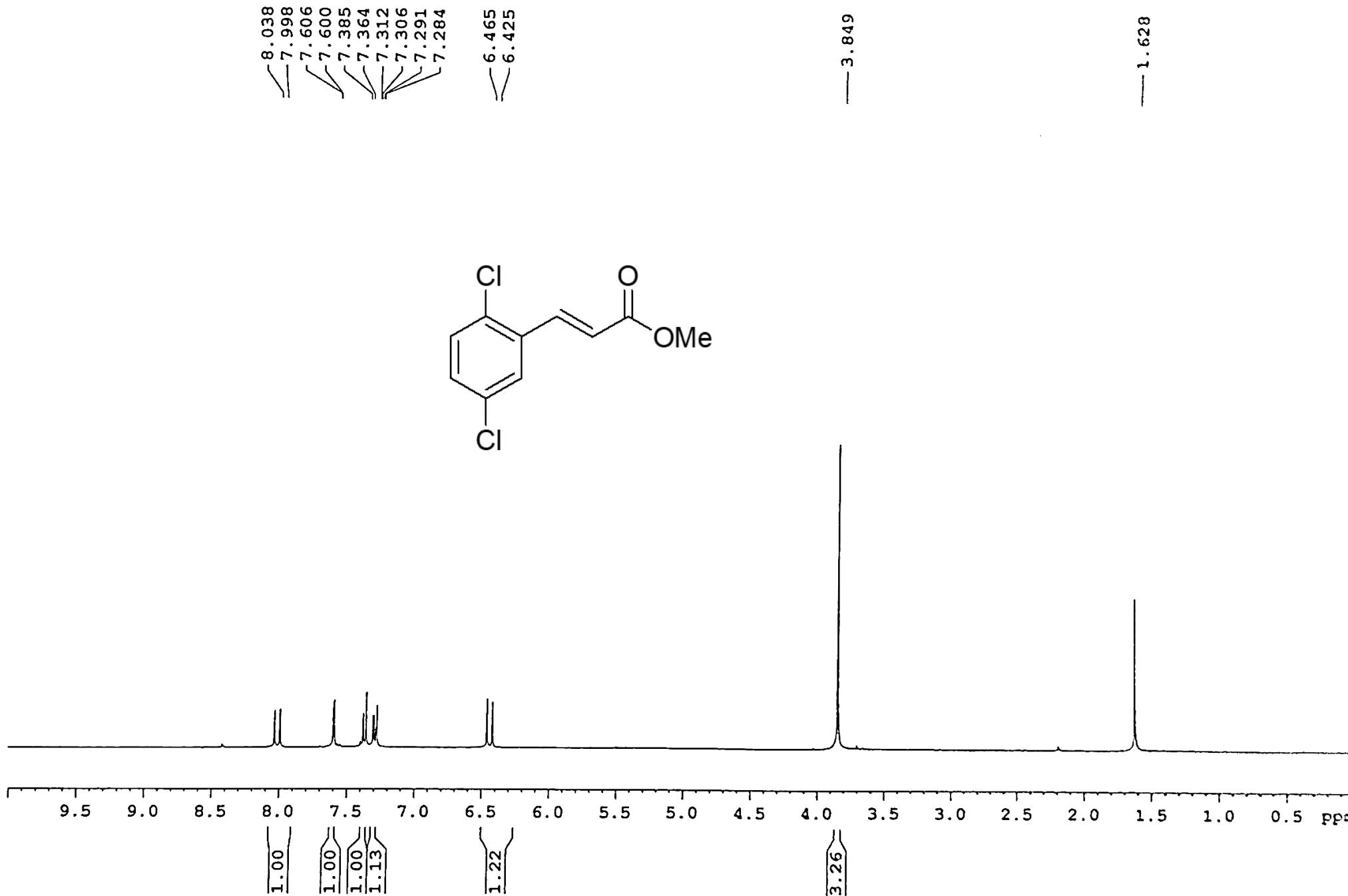
¹H NMR OF 3-(5-Acetyl-2-methoxy-phenyl)-acrylic acid methyl ester (3pa)



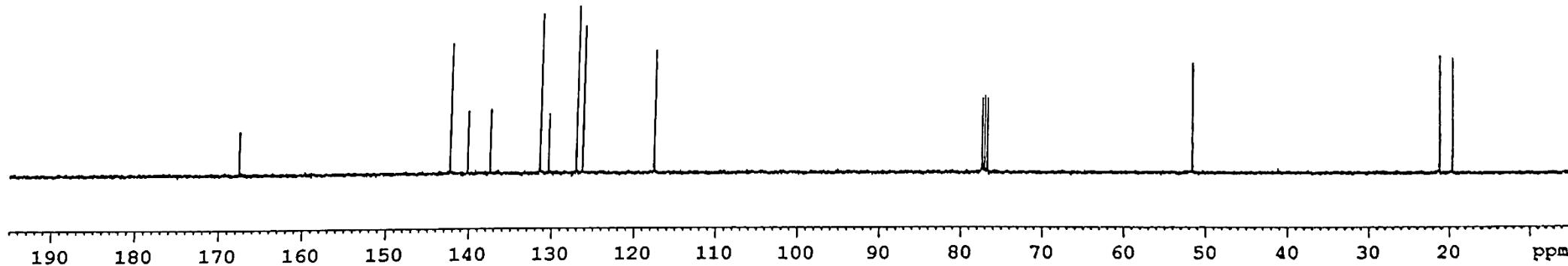
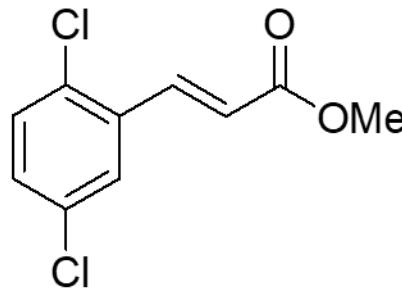
¹³C NMR OF 3-(5-Acetyl-2-methoxy-phenyl)-acrylic acid methyl ester (3pa)



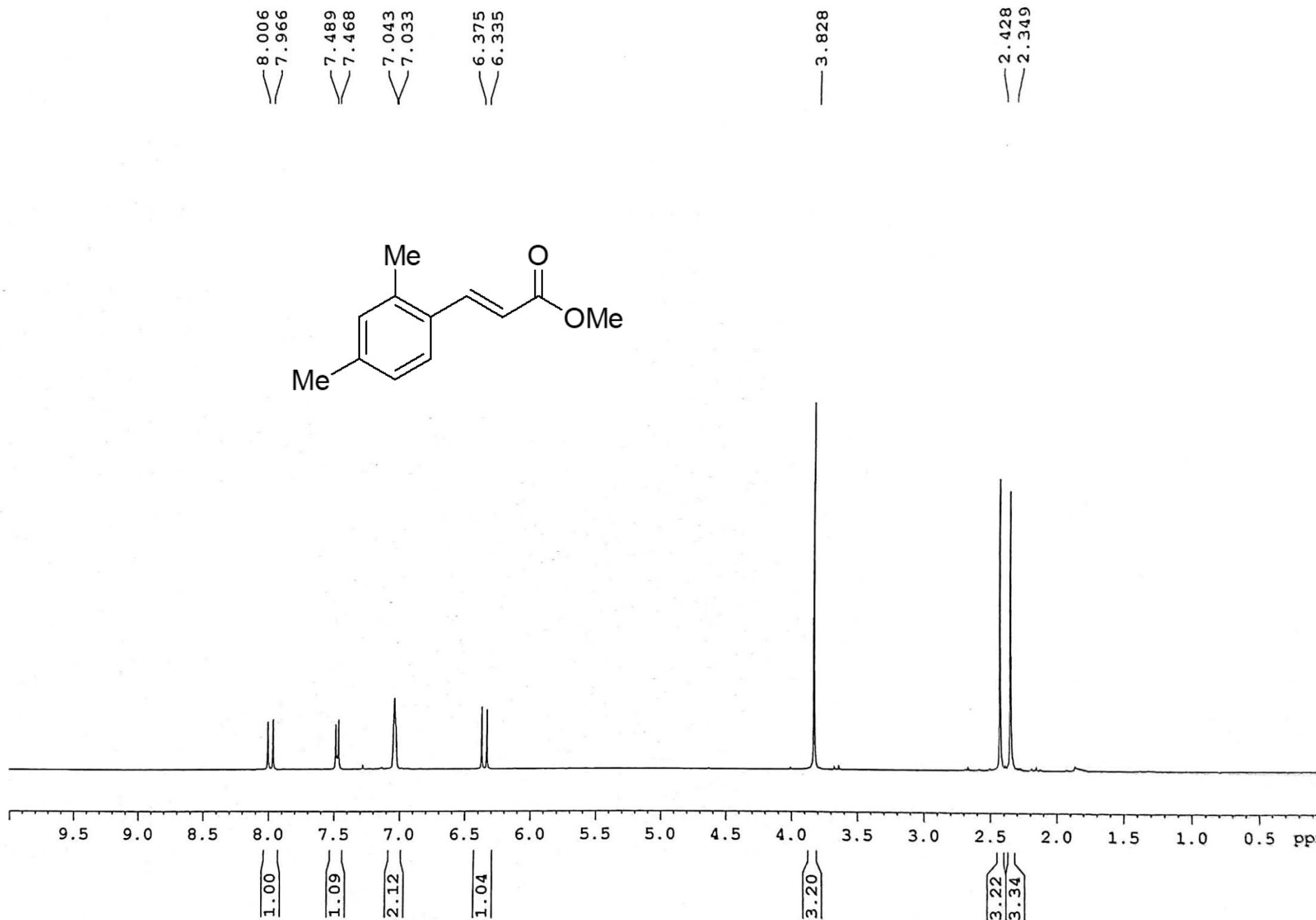
¹H NMR OF 3-(2,5-Dichloro-phenyl)-acrylic acid methyl ester (3qa)



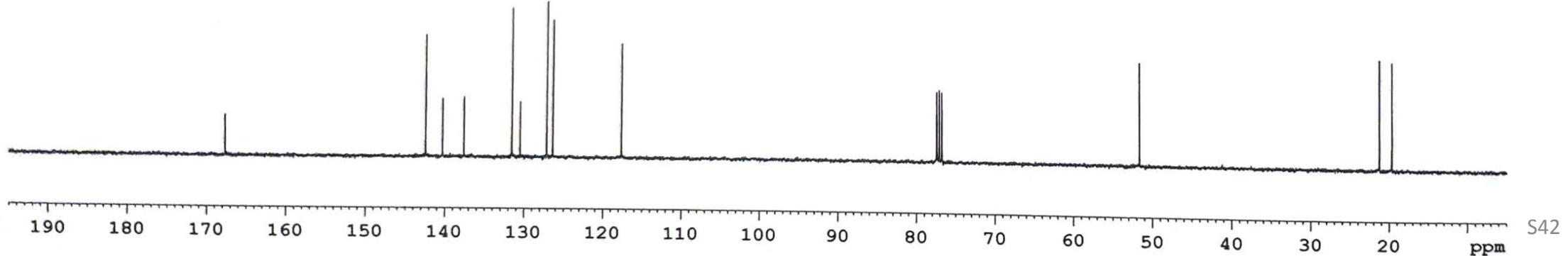
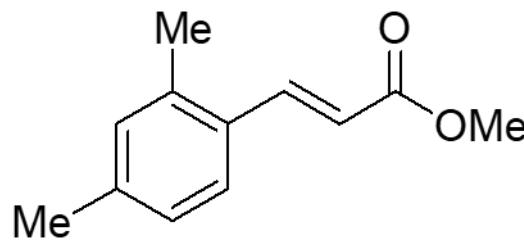
¹³C NMR OF 3-(2,5-Dichloro-phenyl)-acrylic acid methyl ester (3qa)



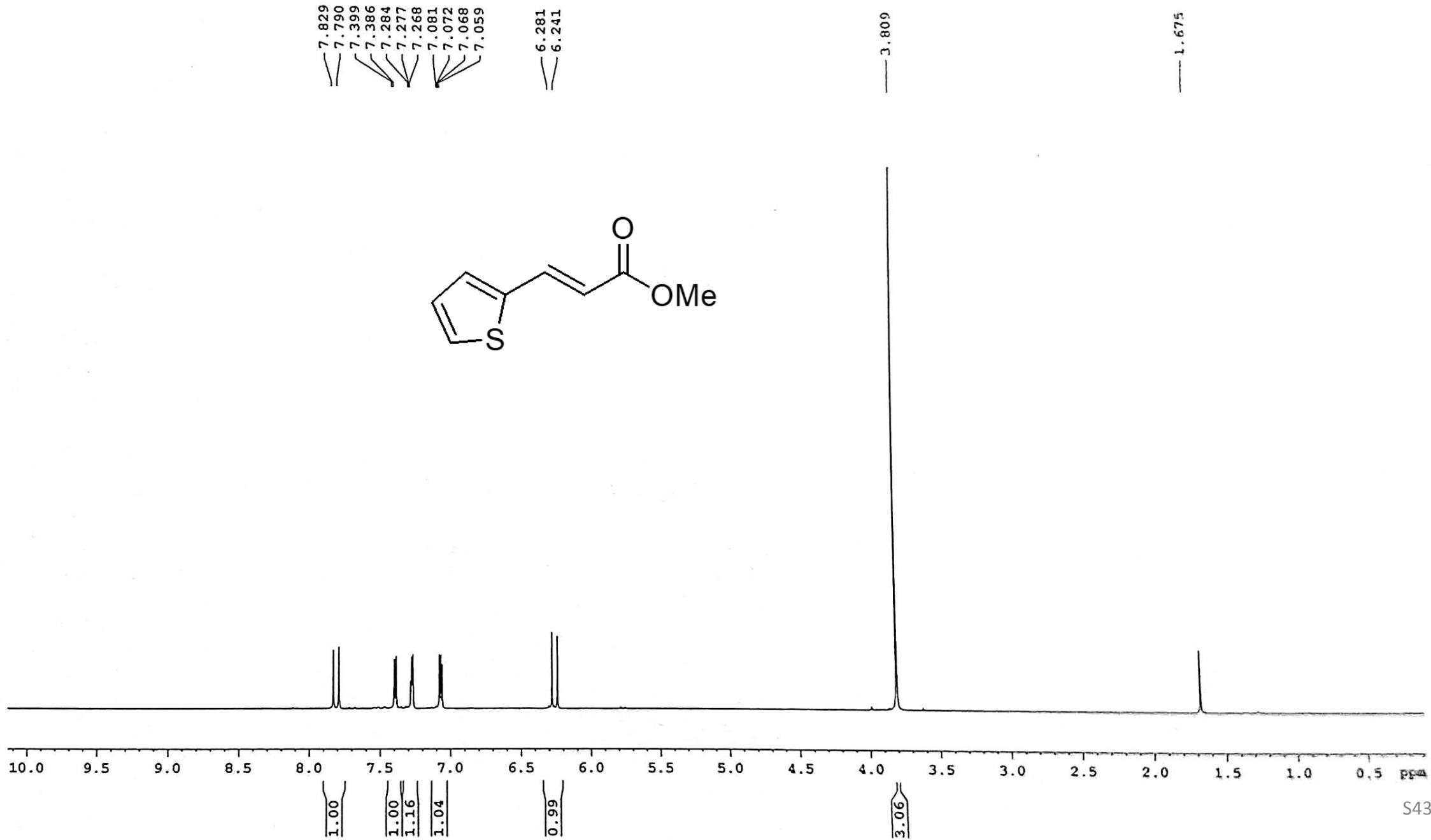
¹H NMR OF 3-(2,4-Dimethyl-phenyl)-acrylic acid methyl ester (3ra)



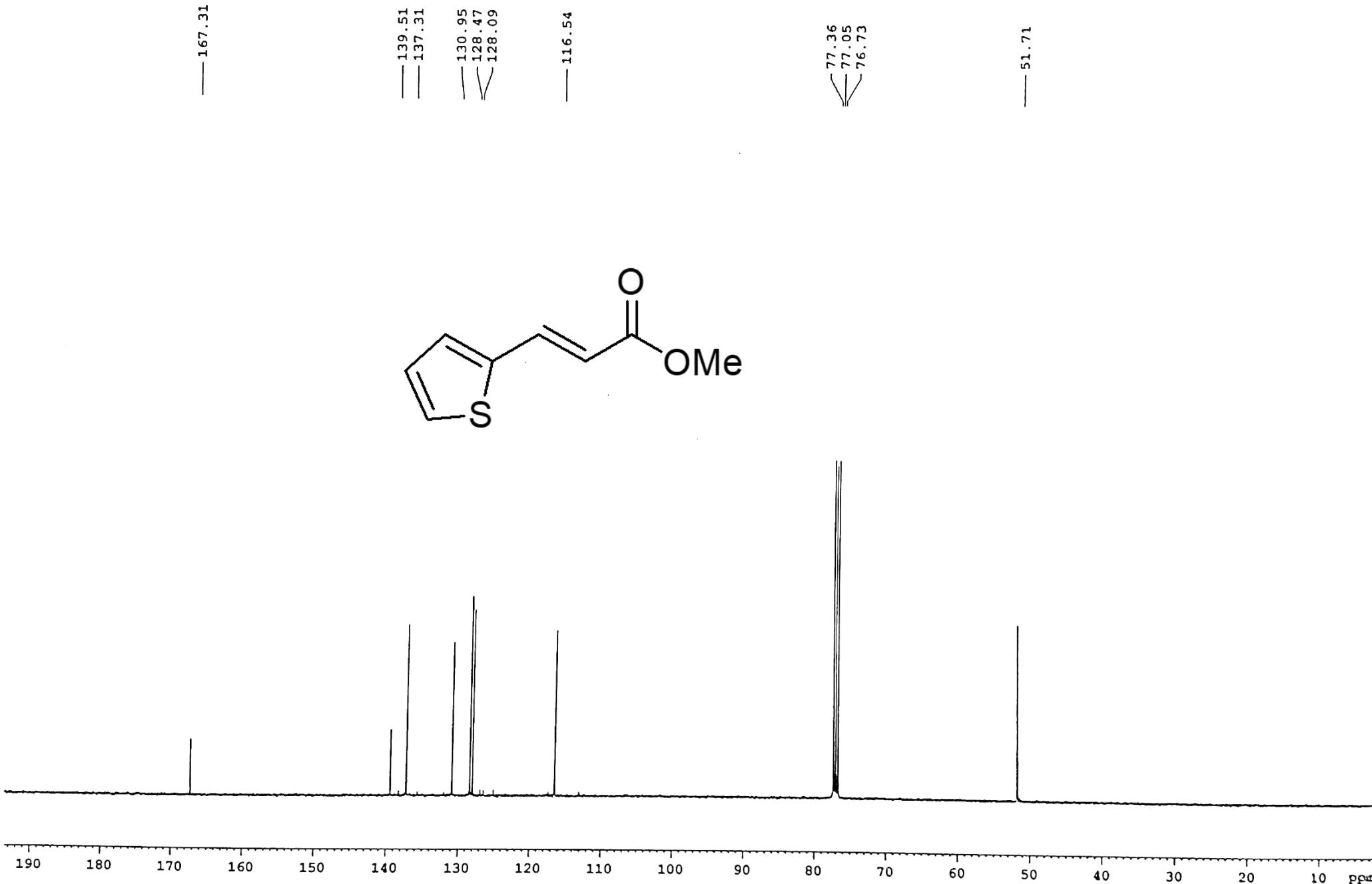
¹³C NMR OF 3-(2,4-Dimethyl-phenyl)-acrylic acid methyl ester (3ra)



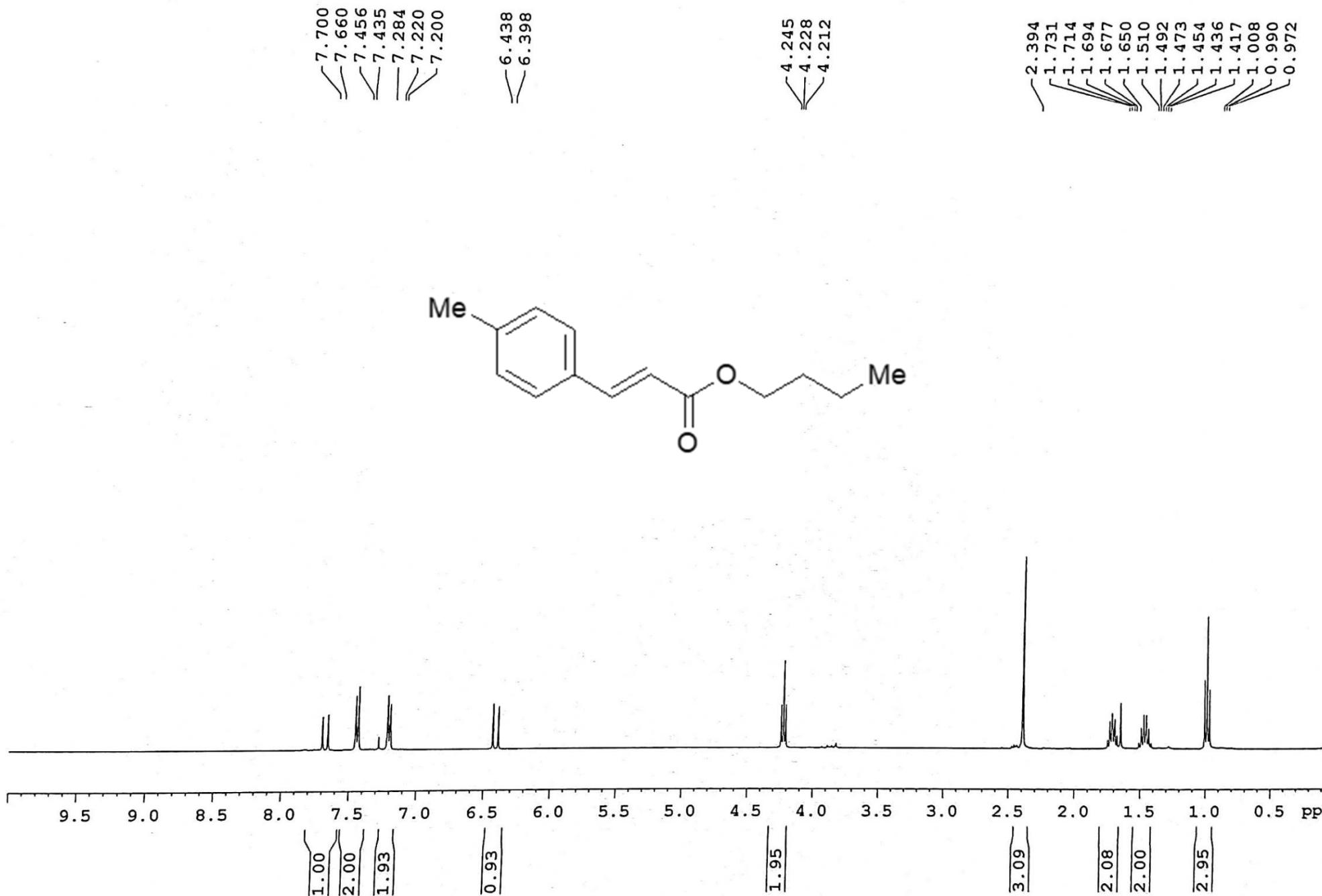
¹H NMR OF 3-Thiophen-2-yl-acrylic acid methyl ester (3sa)



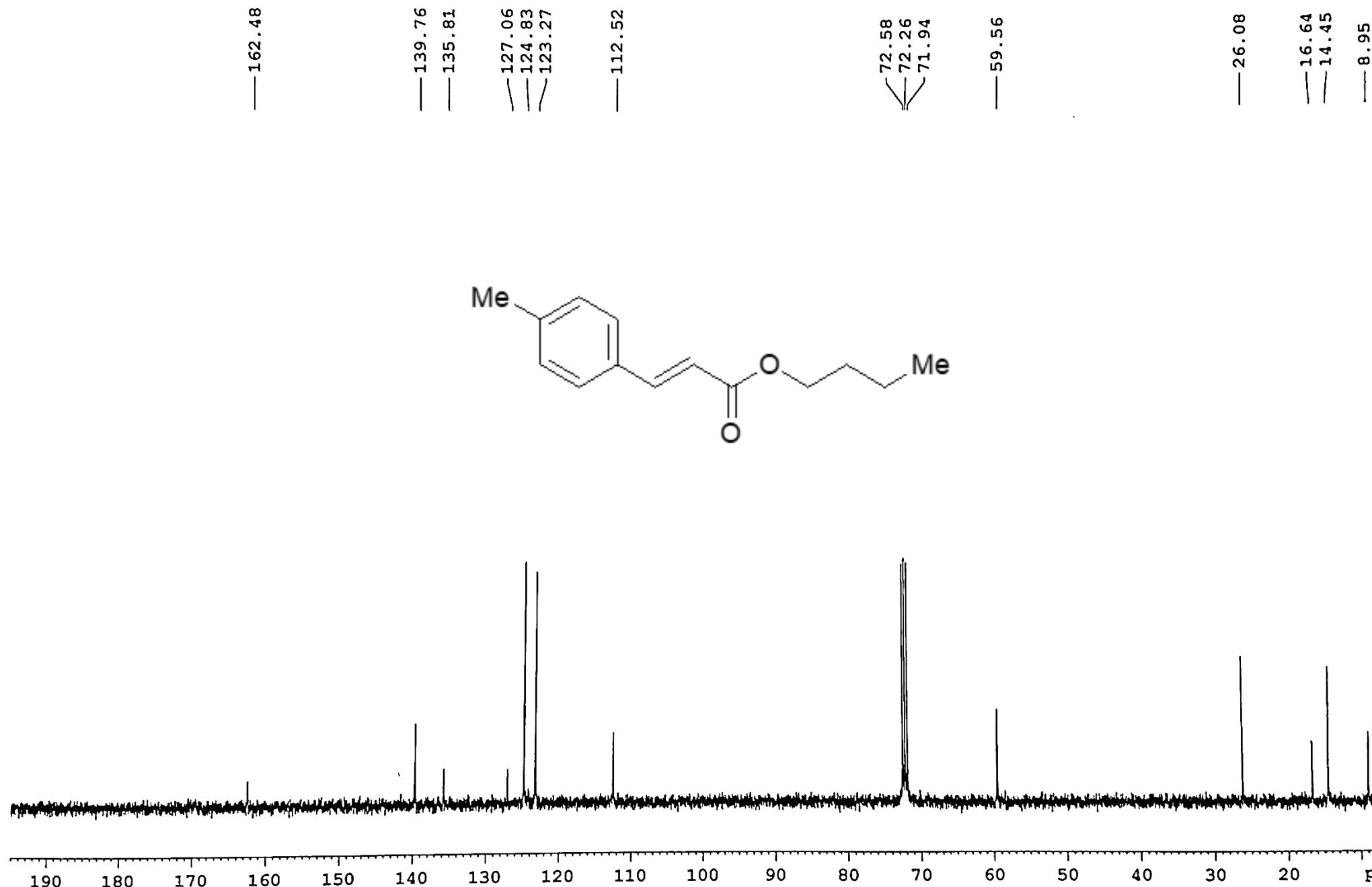
¹³C NMR OF 3-Thiophen-2-yl-acrylic acid methyl ester (3sa)



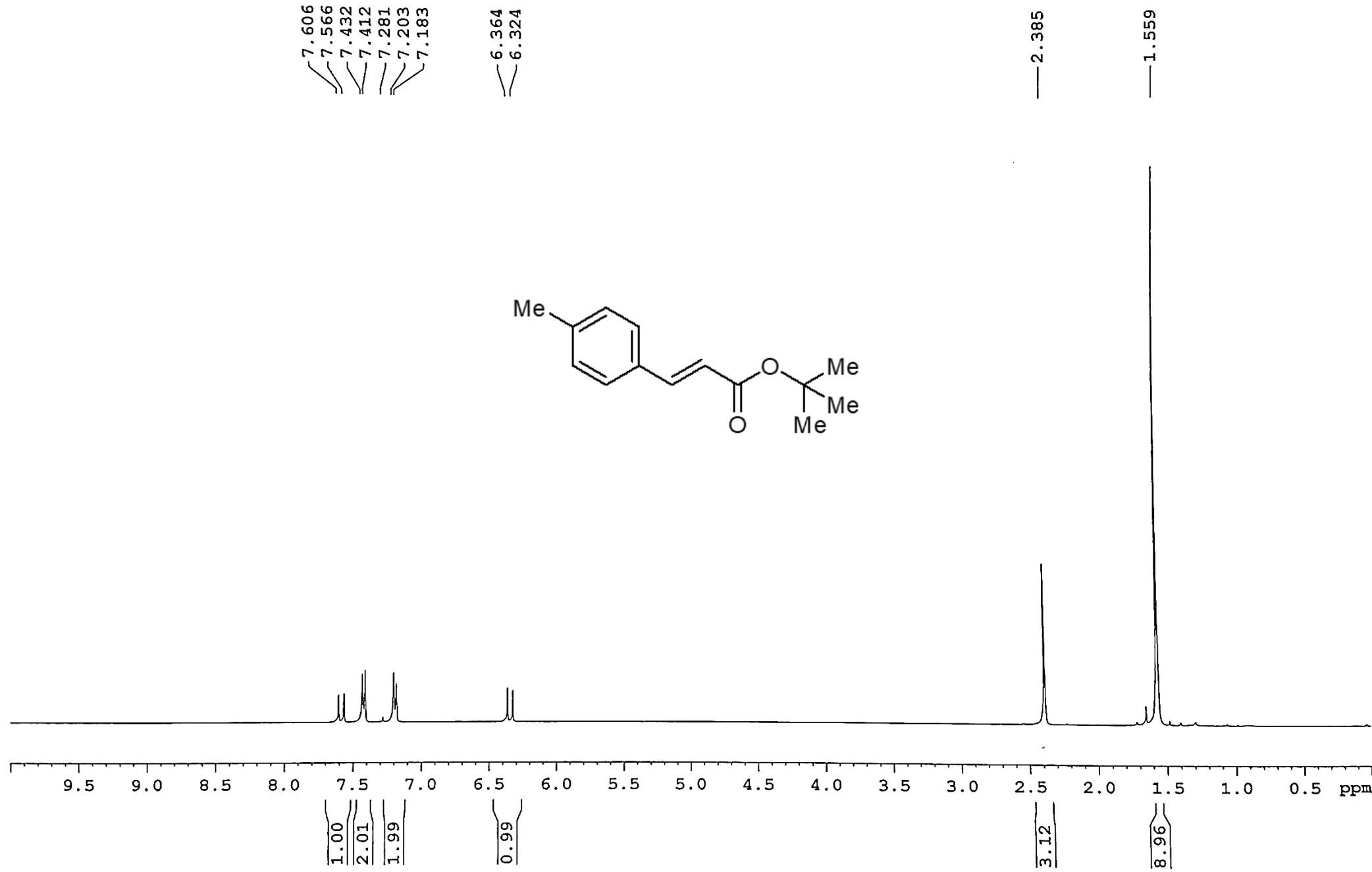
¹H NMR OF 3-p-Tolyl-acrylic acid butyl ester (3ab)



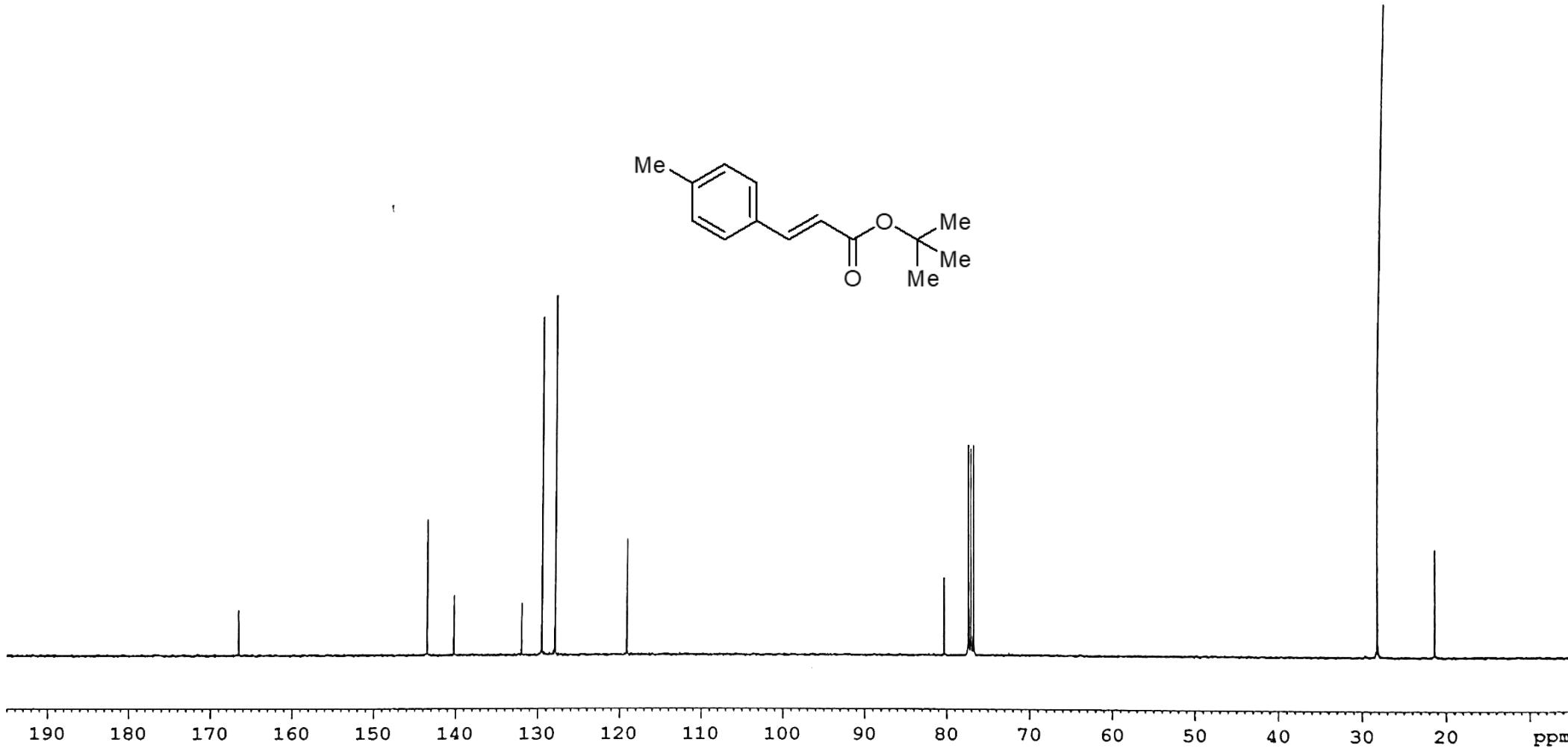
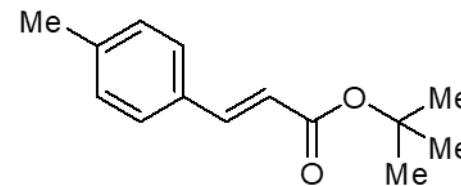
^{13}C NMR OF 3-p-Tolyl-acrylic acid butyl ester (3ab)



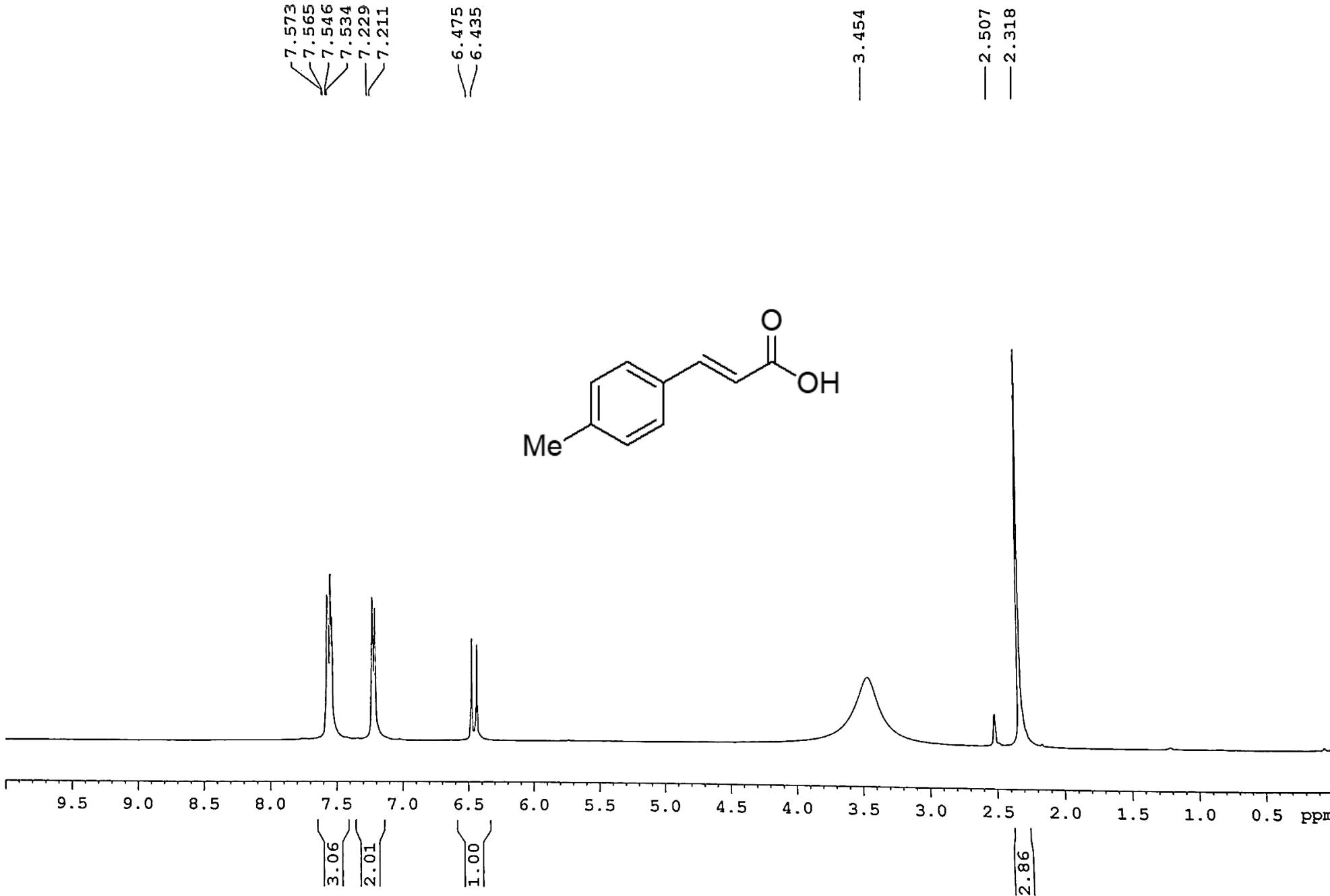
¹H NMR OF 3-p-Tolyl-acrylic acid tert-butyl ester (3ac)



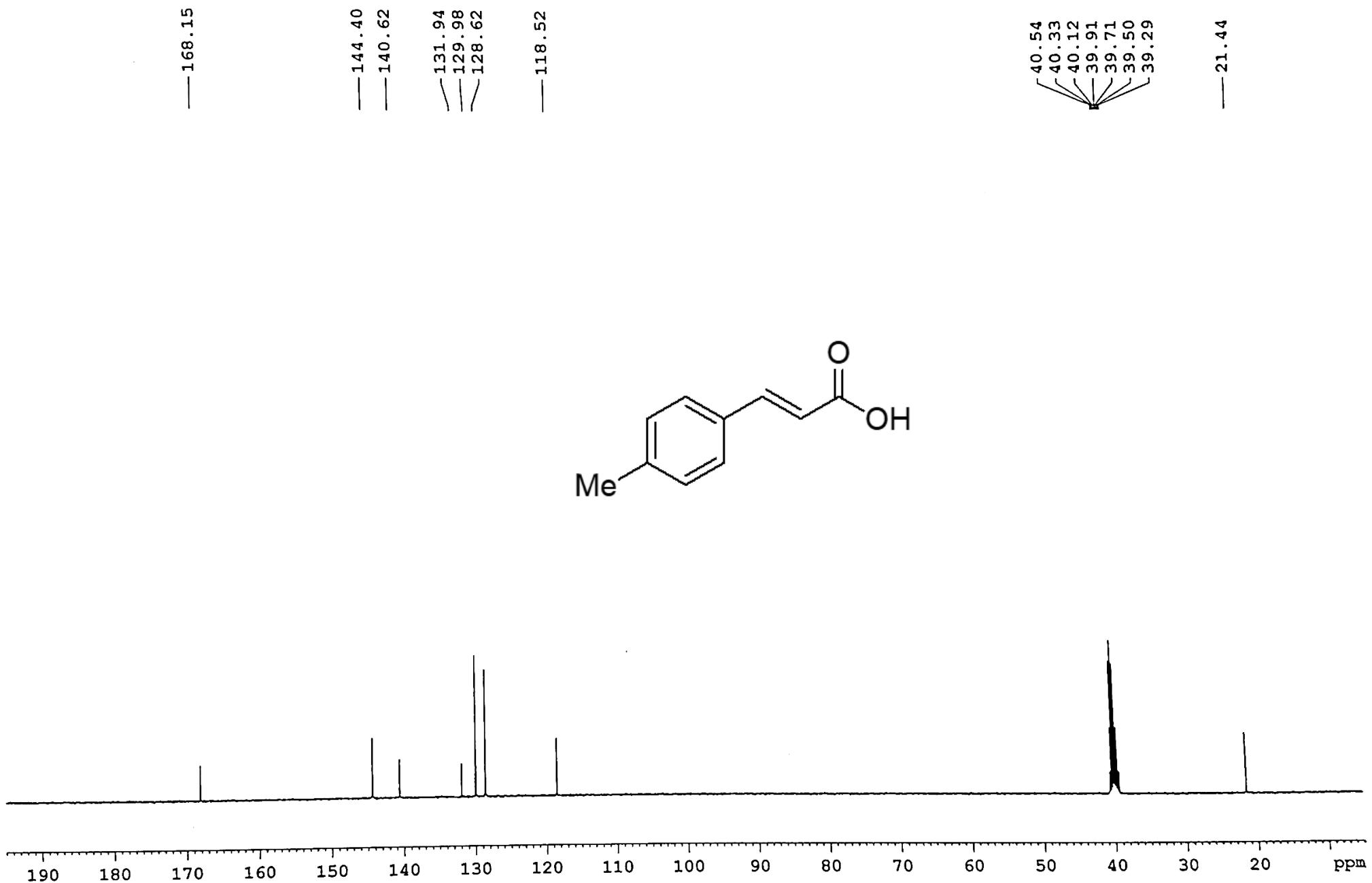
¹³C NMR OF 3-p-Tolyl-acrylic acid tert-butyl ester (3ac)



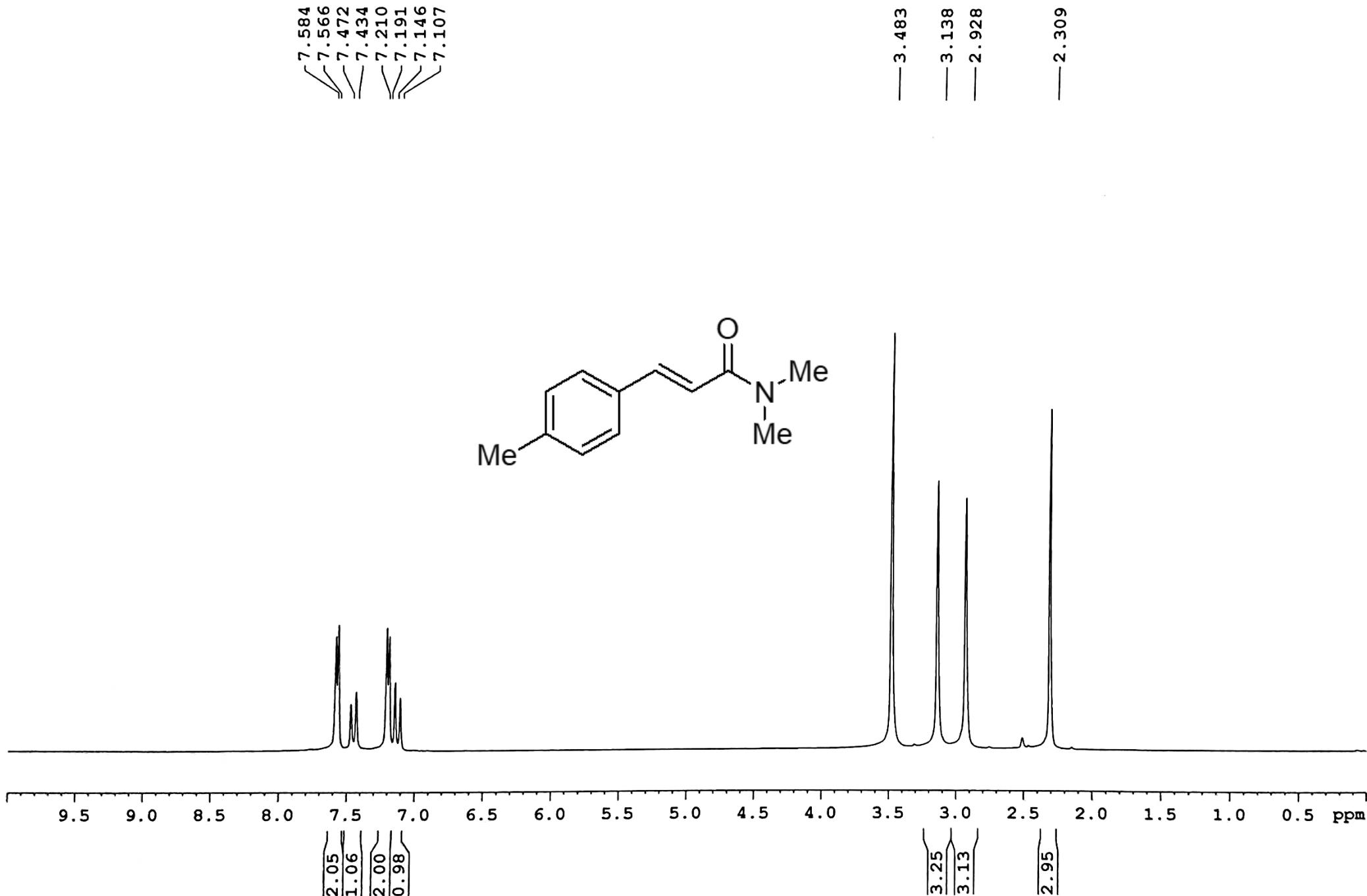
¹H NMR OF 3-p-Tolyl-acrylic acid (3ad)



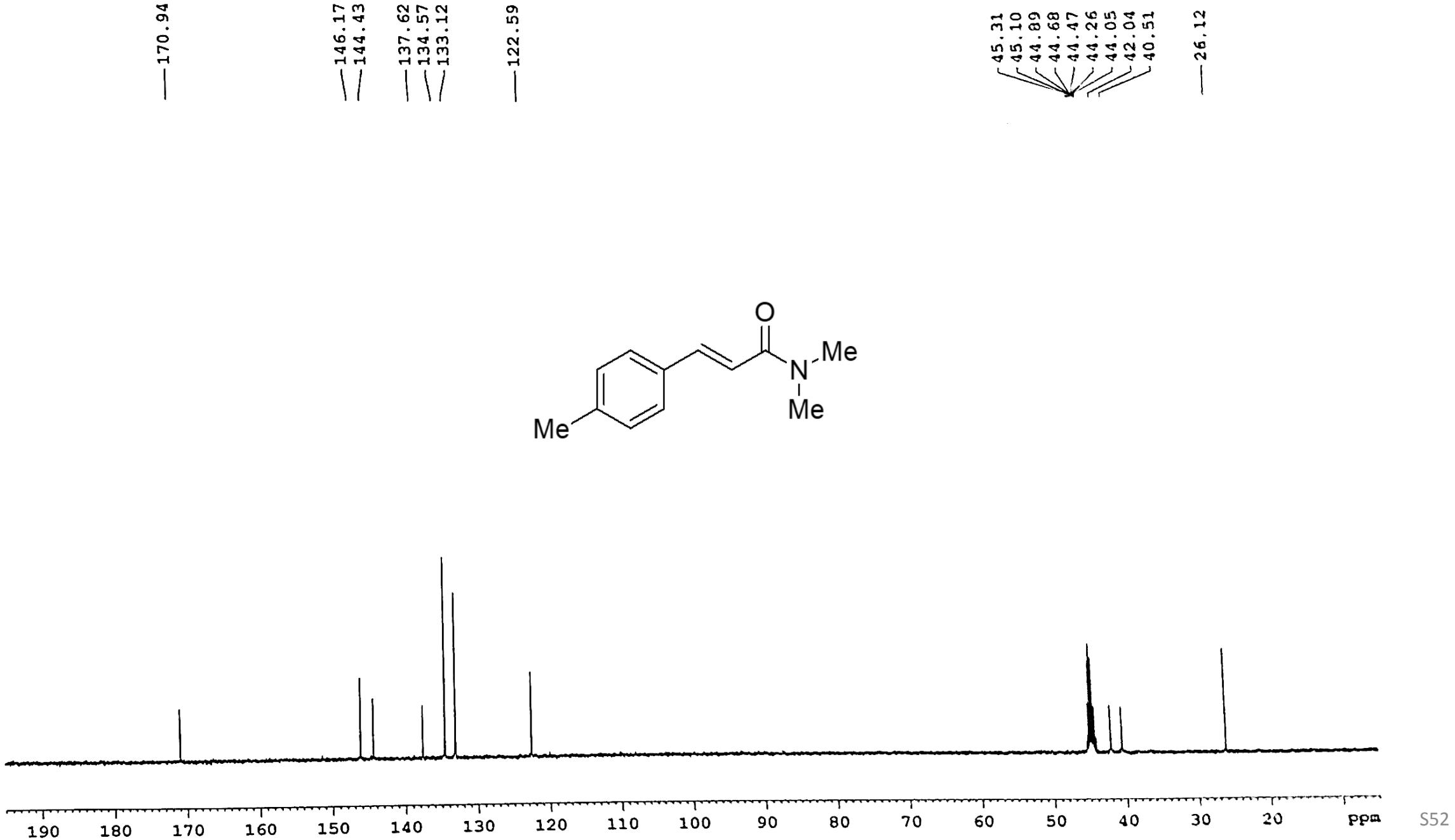
¹³C NMR OF 3-p-Tolyl-acrylic acid (3ad)



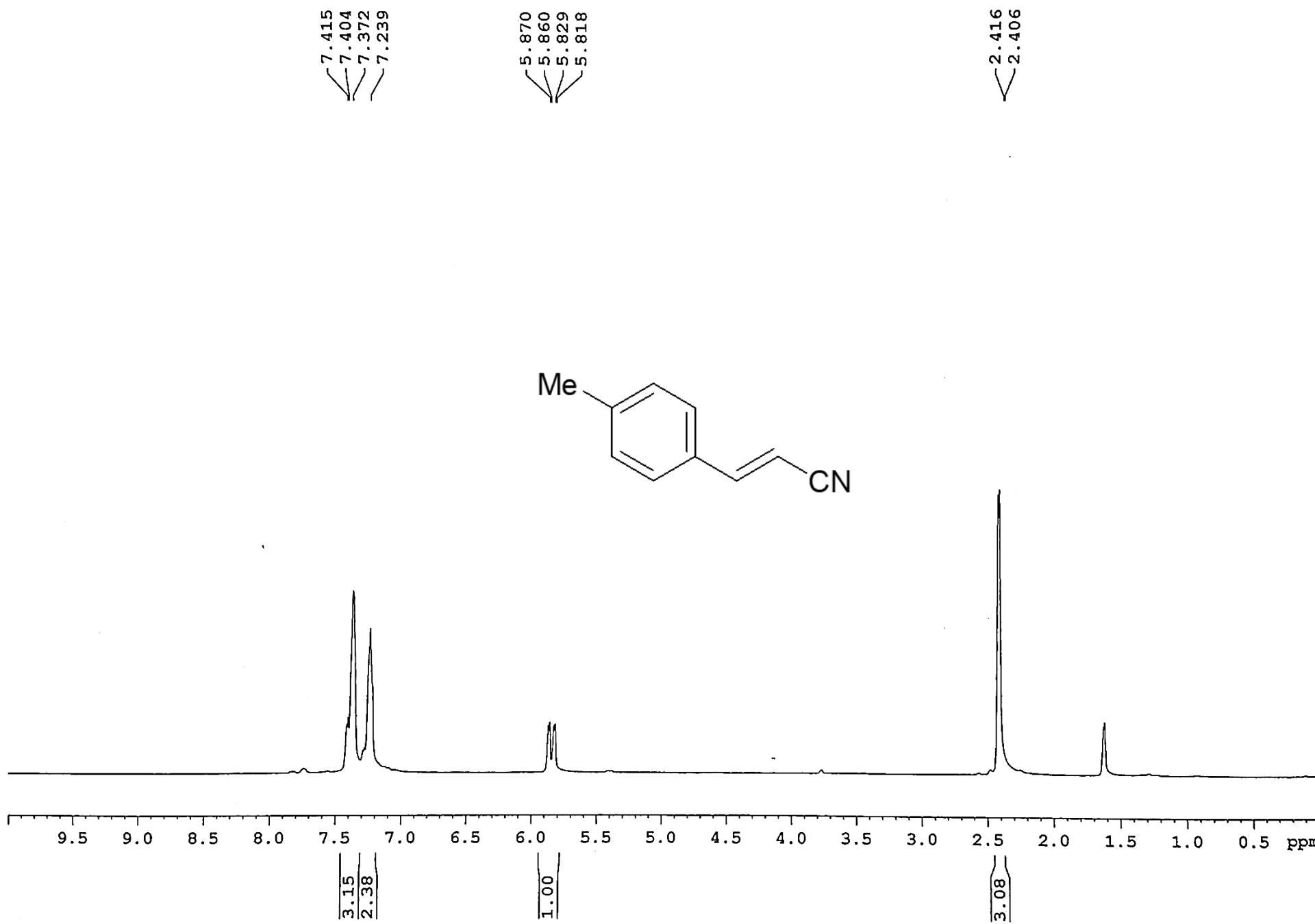
¹H OF NMR *N,N*-Dimethyl-3-p-tolyl-acrylamide (3ae)



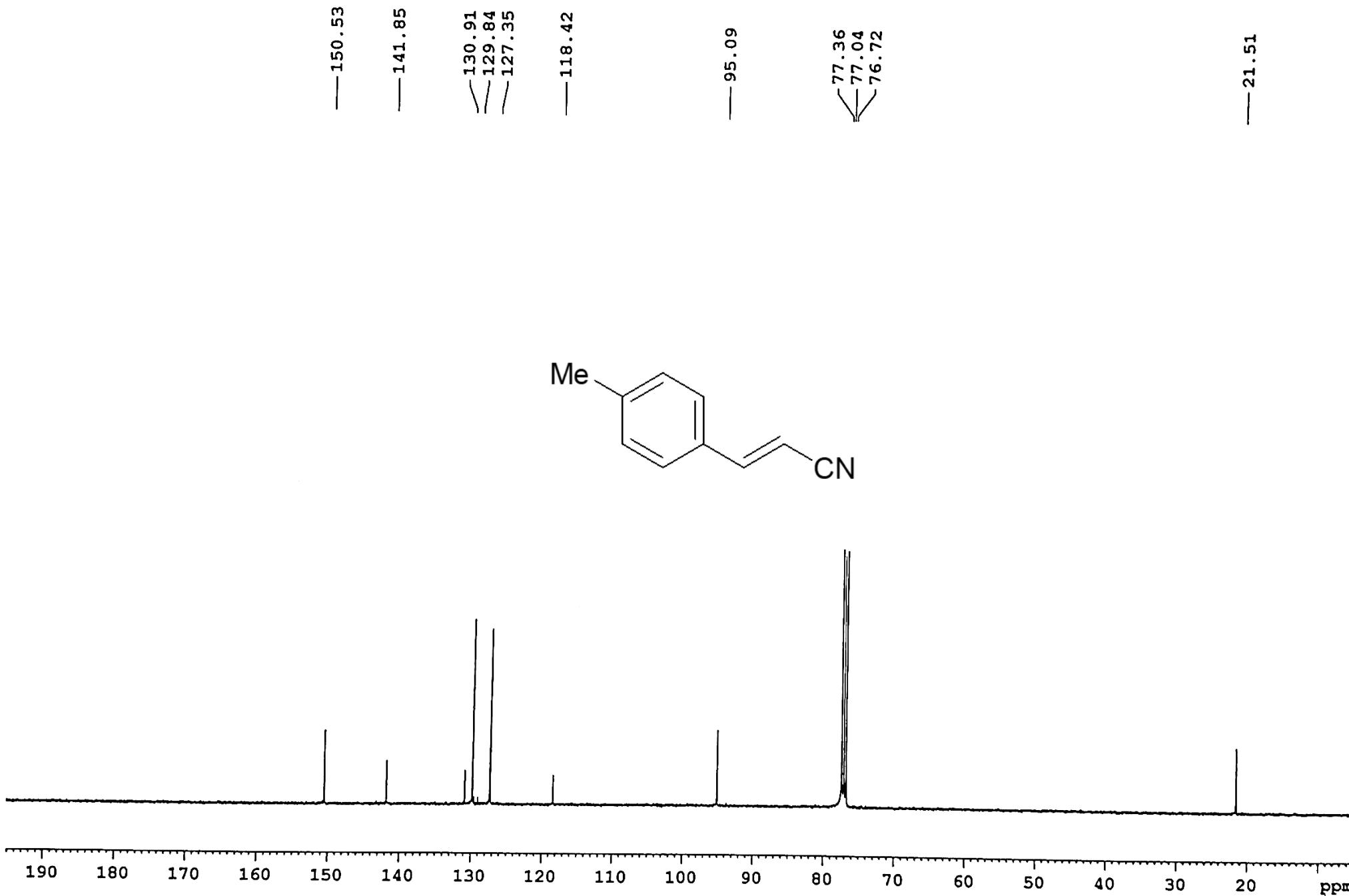
¹³C OF NMR N,N-Dimethyl-3-p-tolyl-acrylamide (3ae)



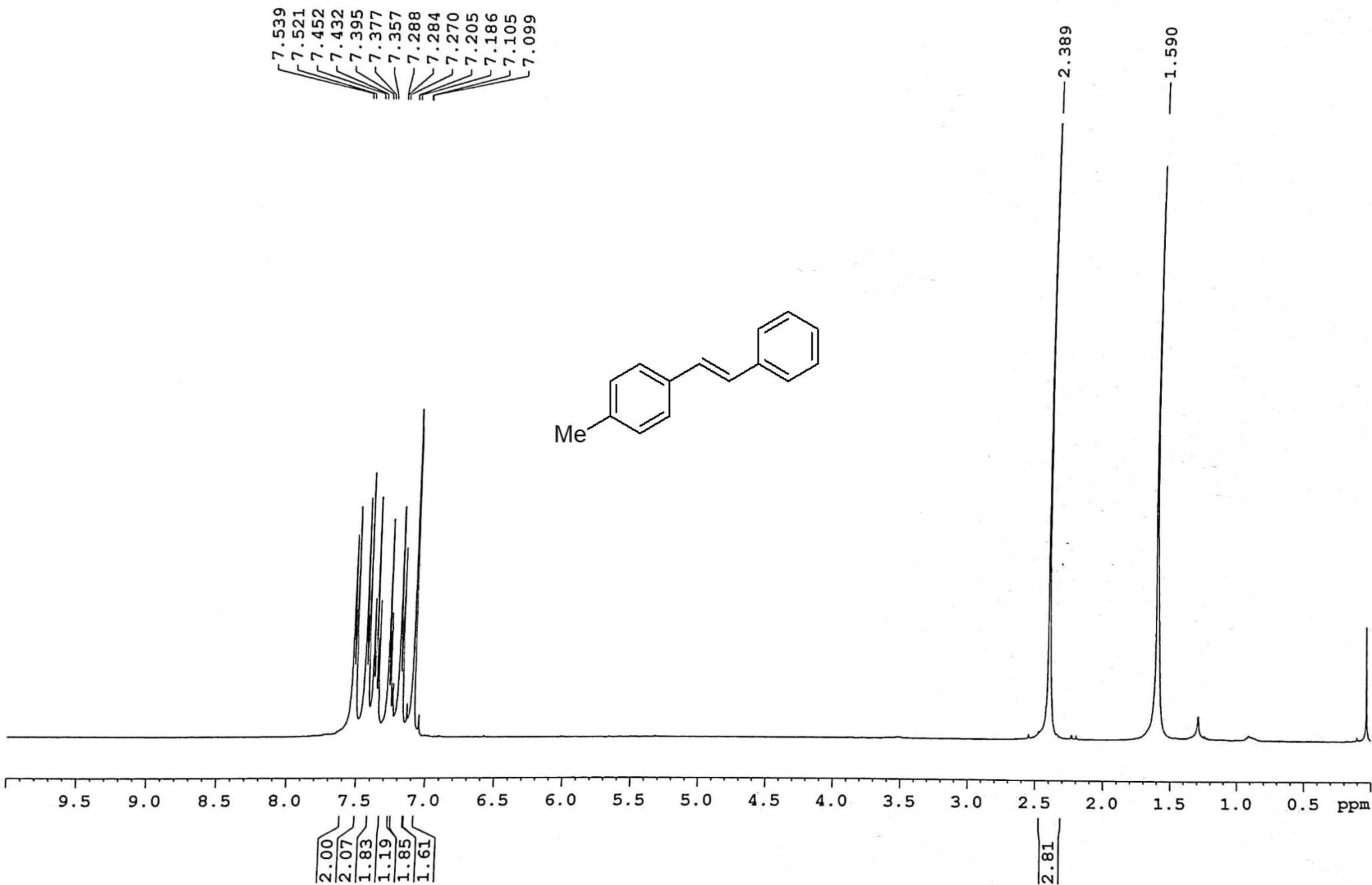
¹H NMR OF 3-*p*-Tolyl-acrylonitrile (3af)



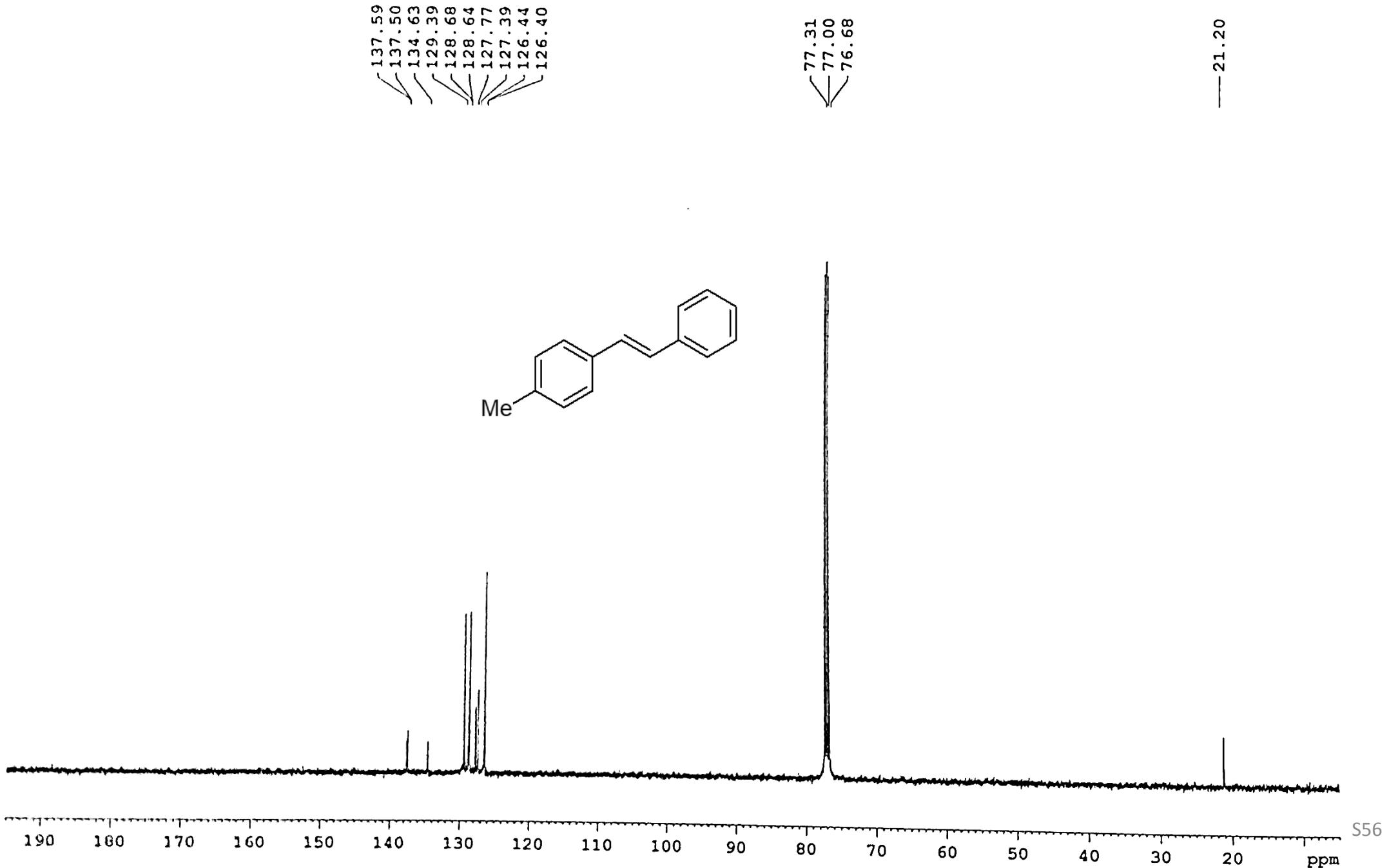
¹³C NMR OF 3-*p*-Tolyl-acrylonitrile (3af)



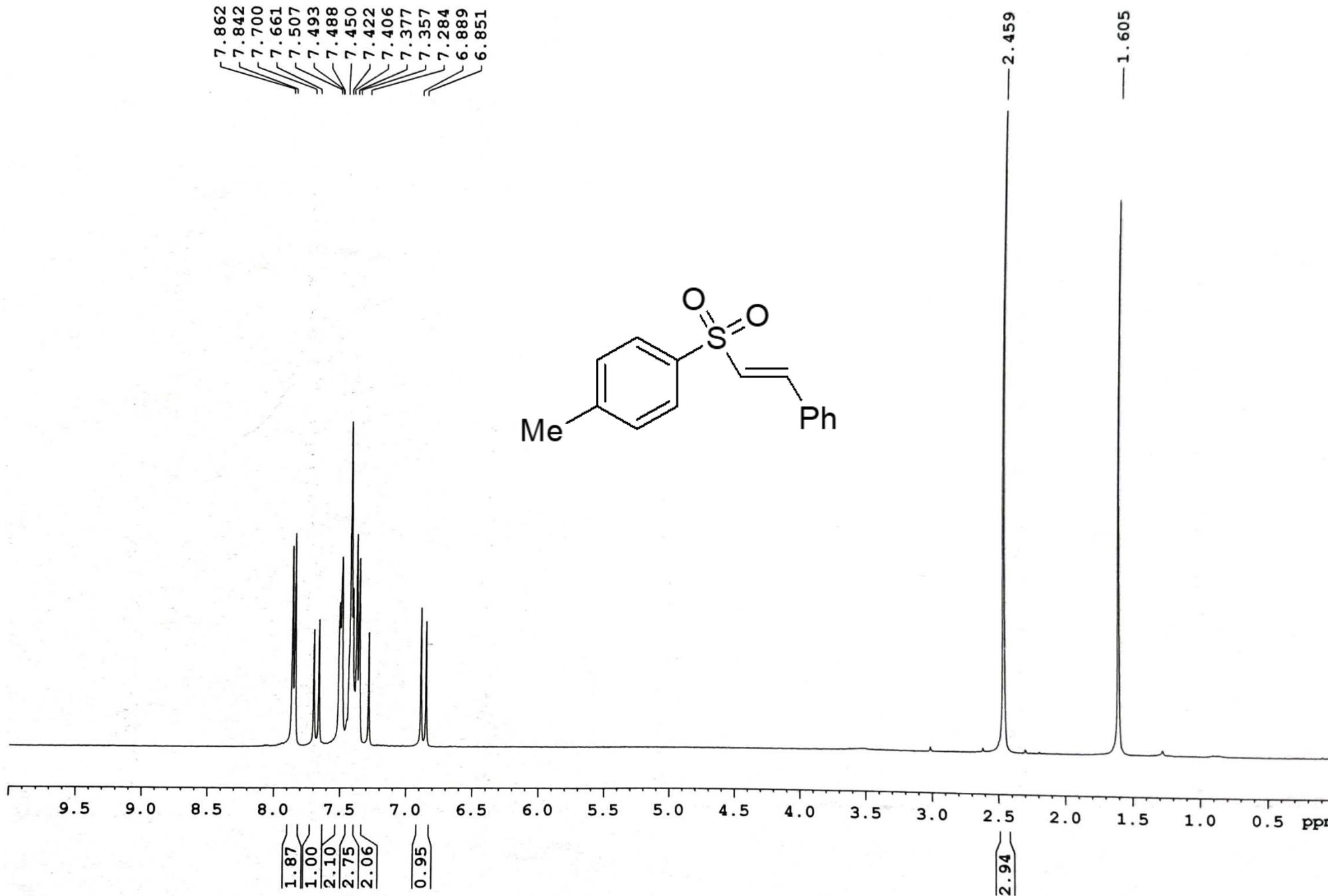
¹H NMR OF 1-Methyl-4-styryl-benzene (3ag)



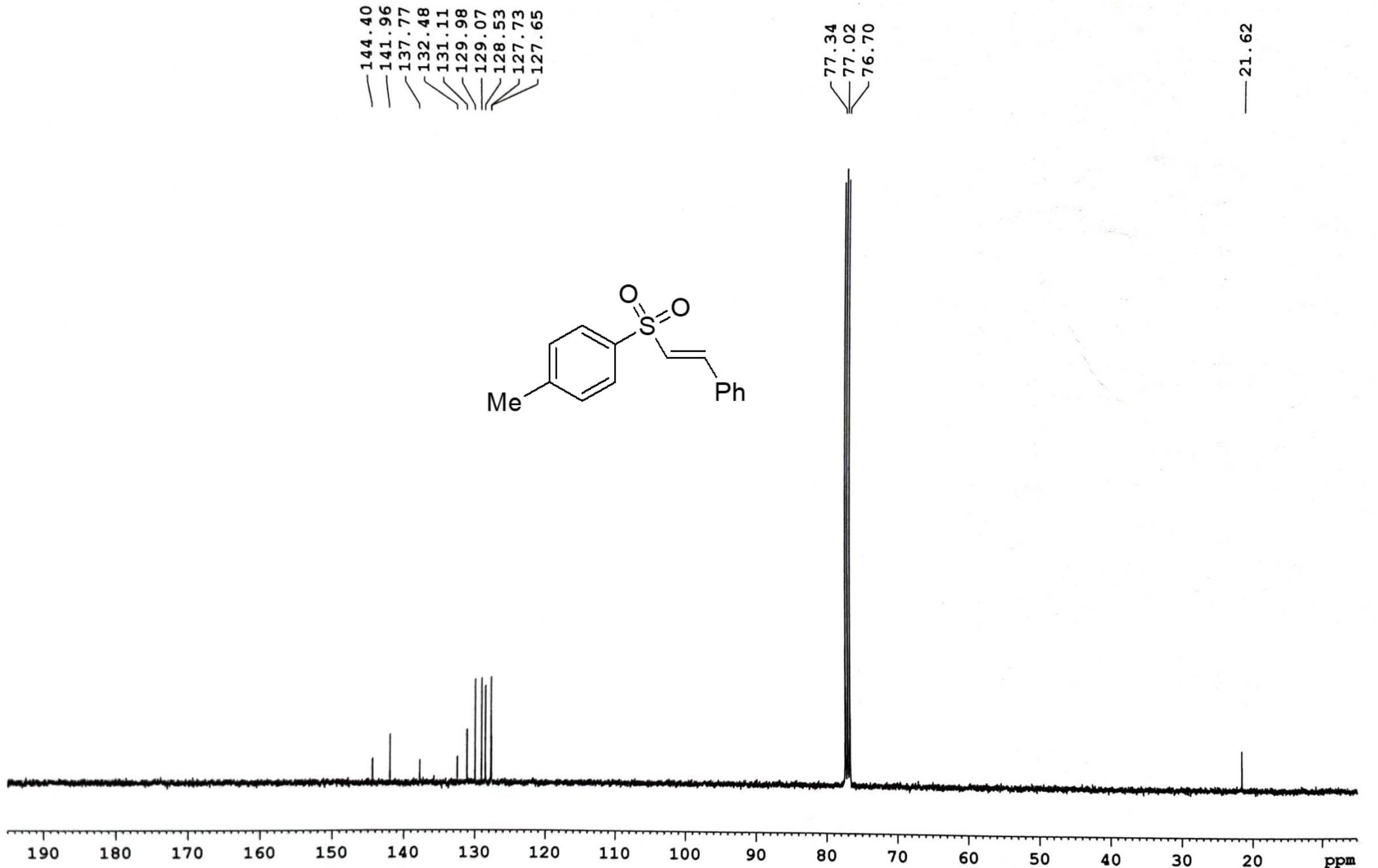
¹³C NMR OF 1-Methyl-4-styryl-benzene (3ag)



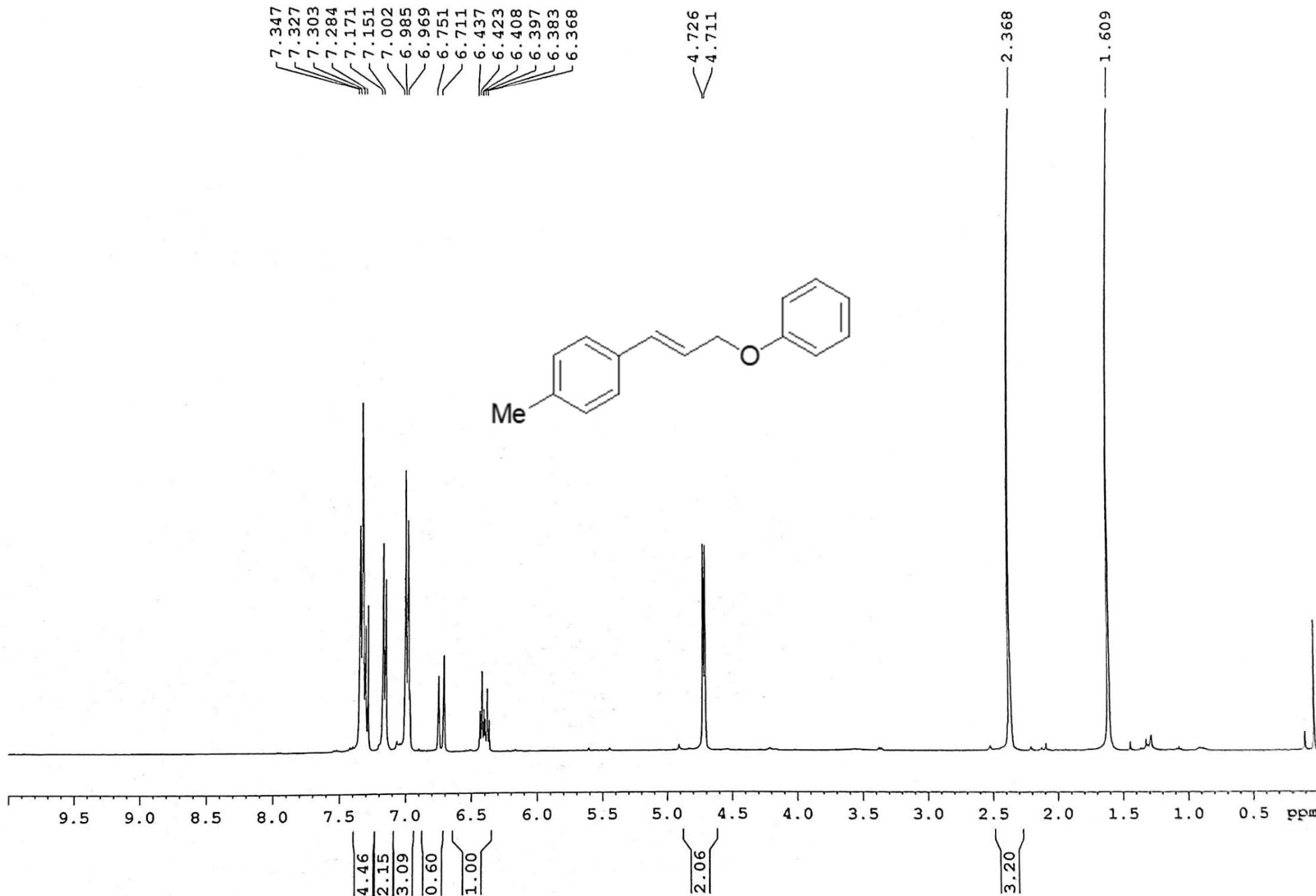
¹H NMR OF 1-Methyl-4-(2-phenyl-ethenesulfonyl)-benzene (3ag')



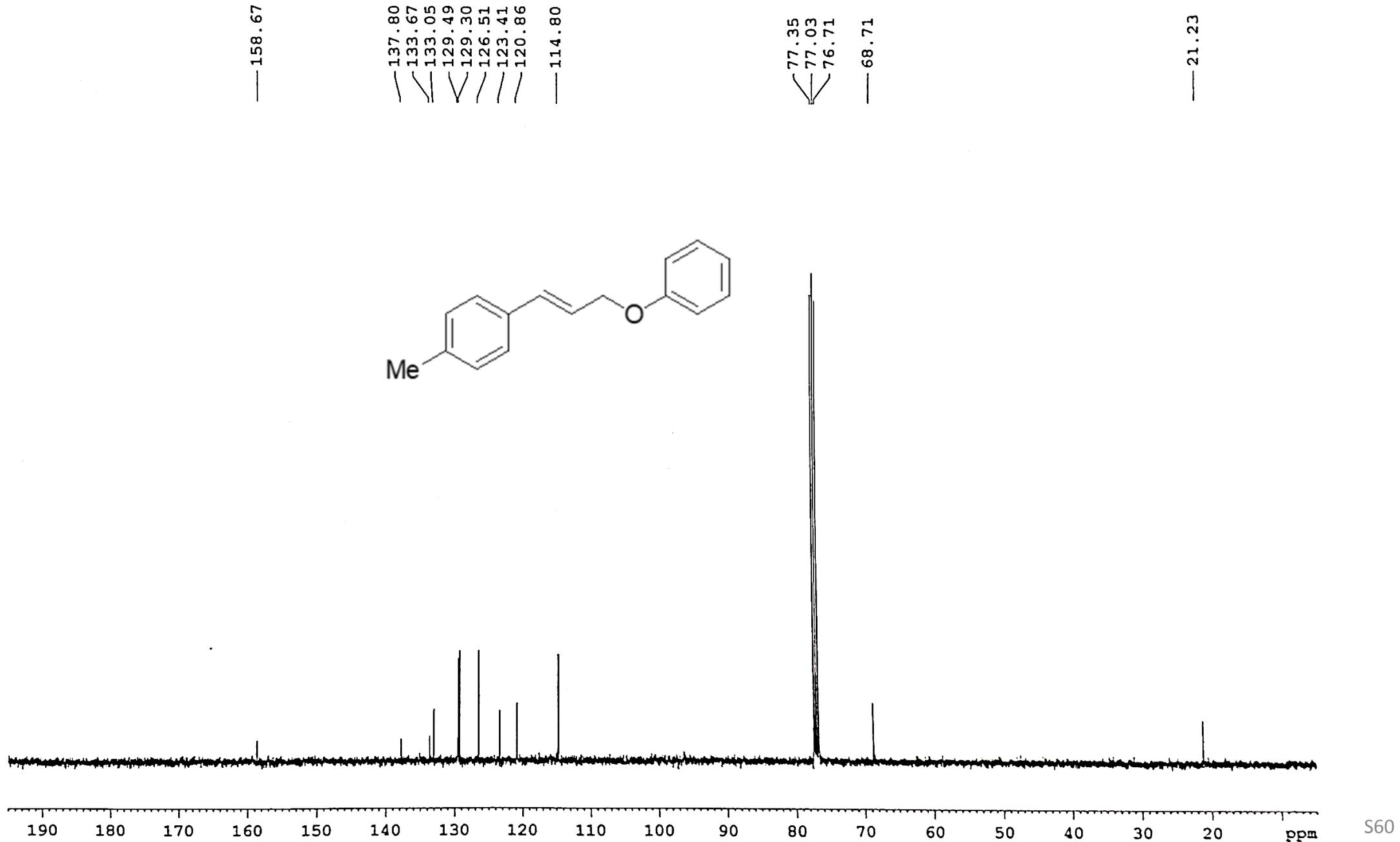
¹³C NMR OF 1-Methyl-4-(2-phenyl-ethenesulfonyl)-benzene (3ag')



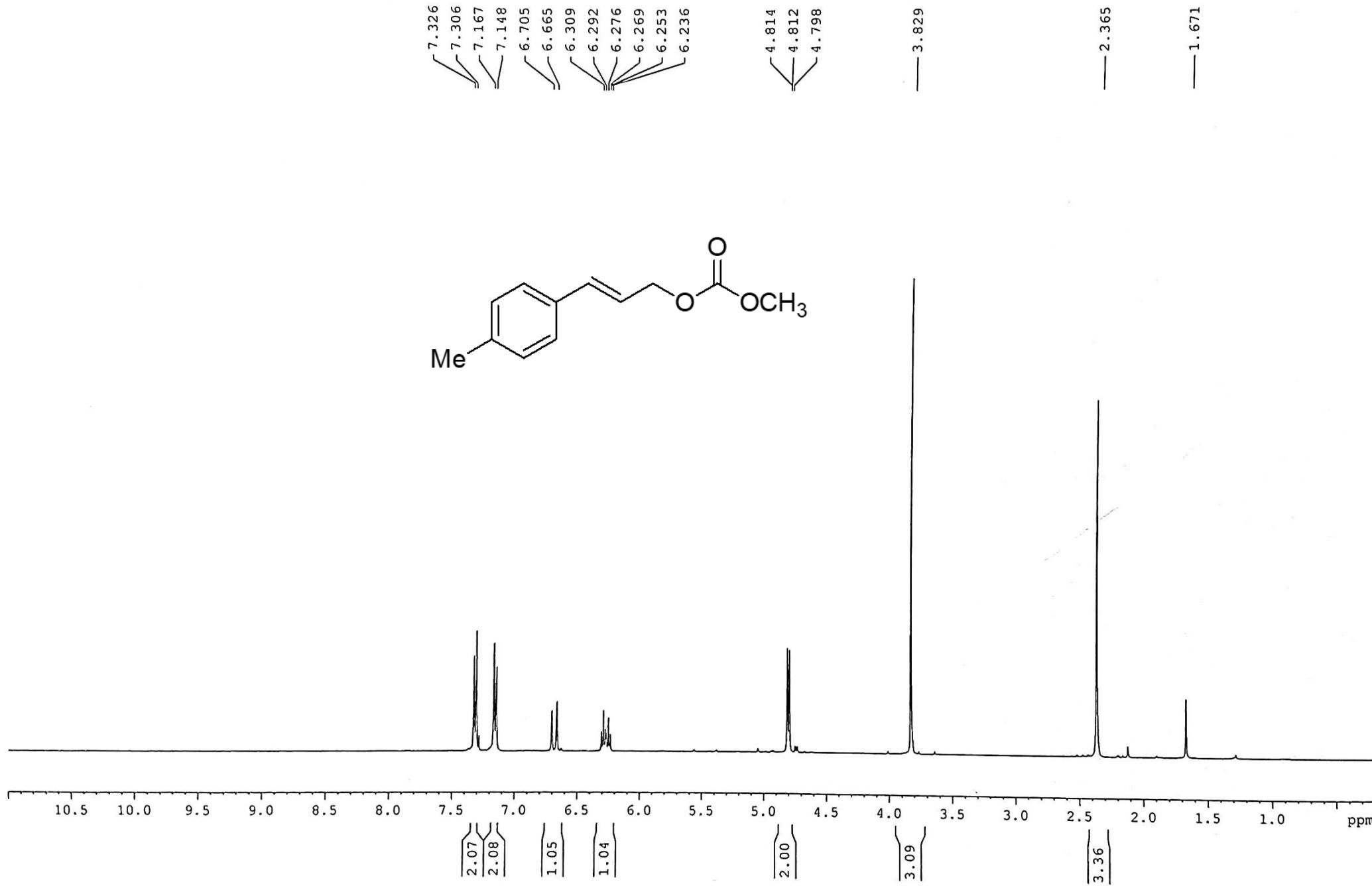
¹H NMR OF 1-Methyl-4-(3-phenoxy-propenyl)-benzene (3ah)



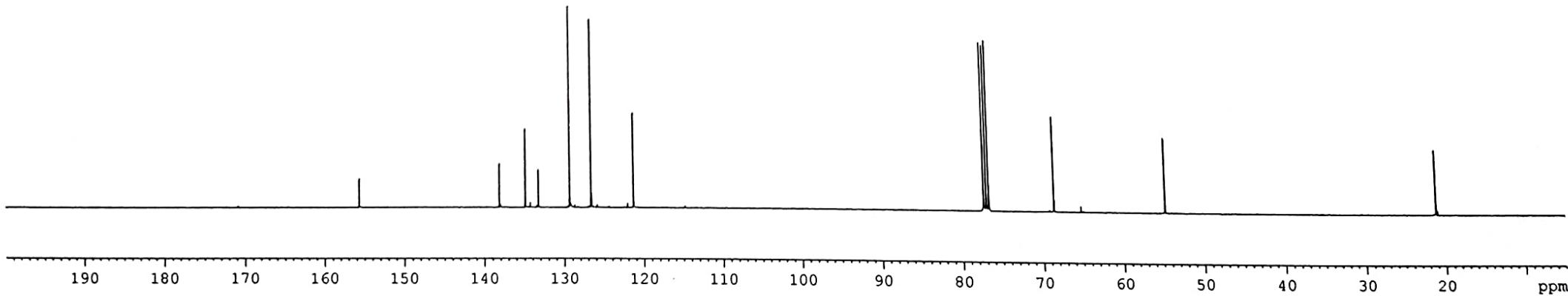
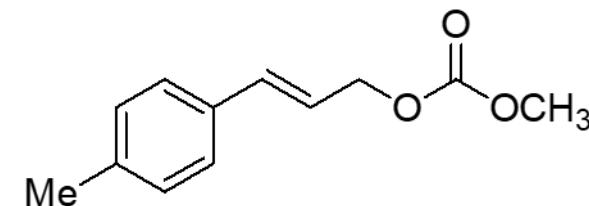
¹³C NMR OF 1-Methyl-4-(3-phenoxy-propenyl)-benzene (3ah)



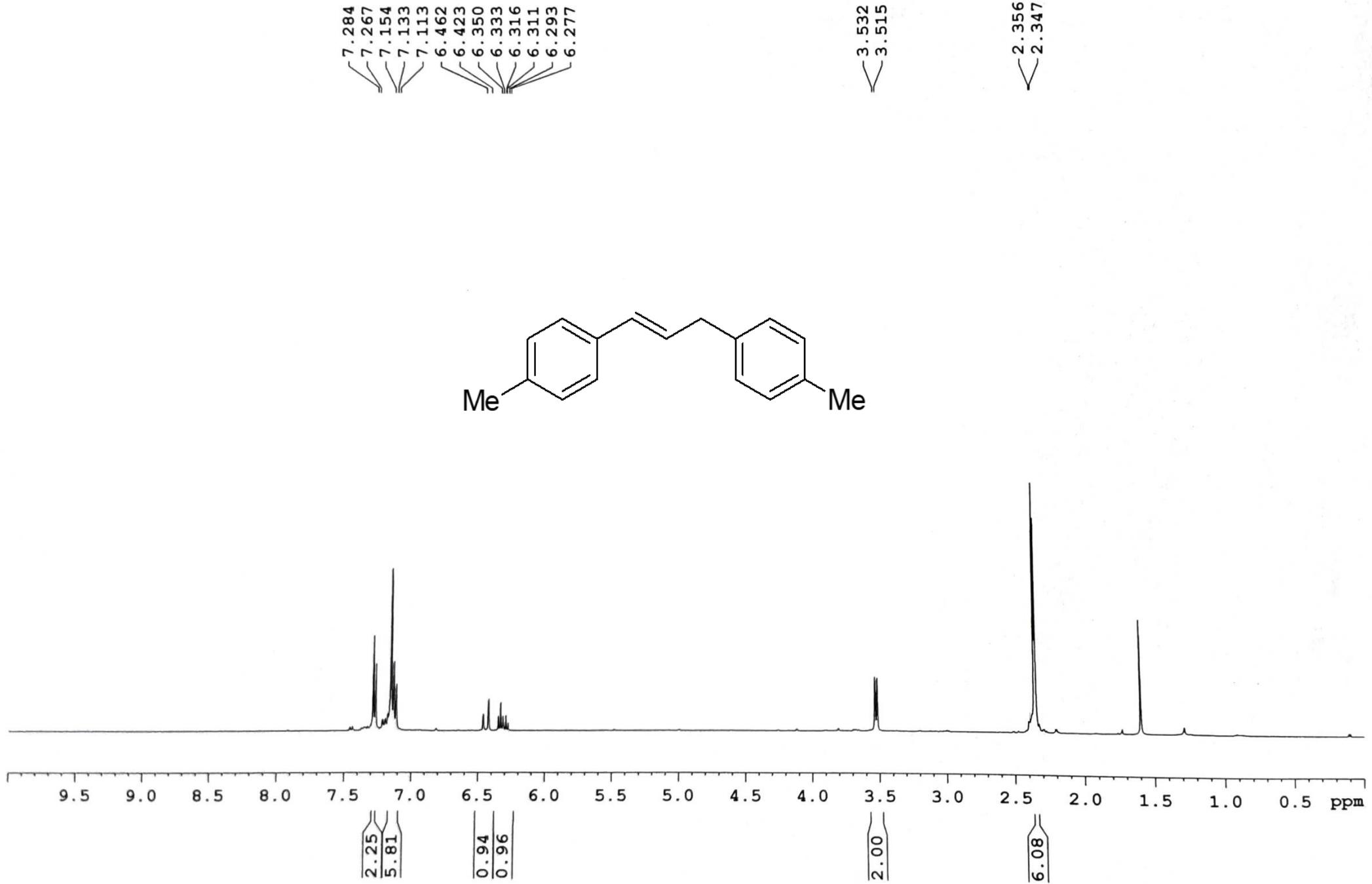
¹H NMR OF Carbonic acid methyl ester 3-p-tolyl-allyl-ester (3ai)



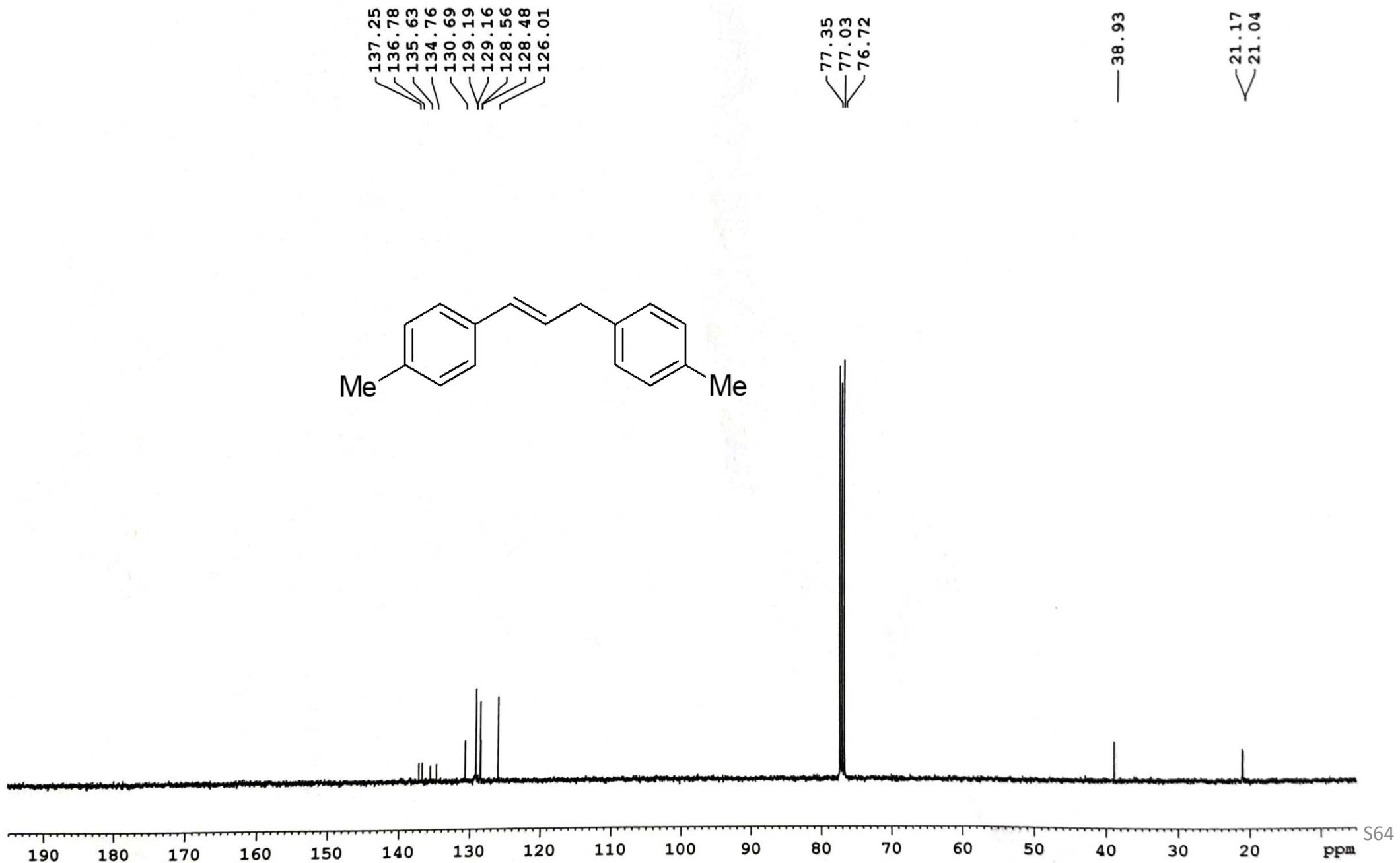
¹³C NMR OF Carbonic acid methyl ester 3-p-tolyl-allyl-ester (3ai)



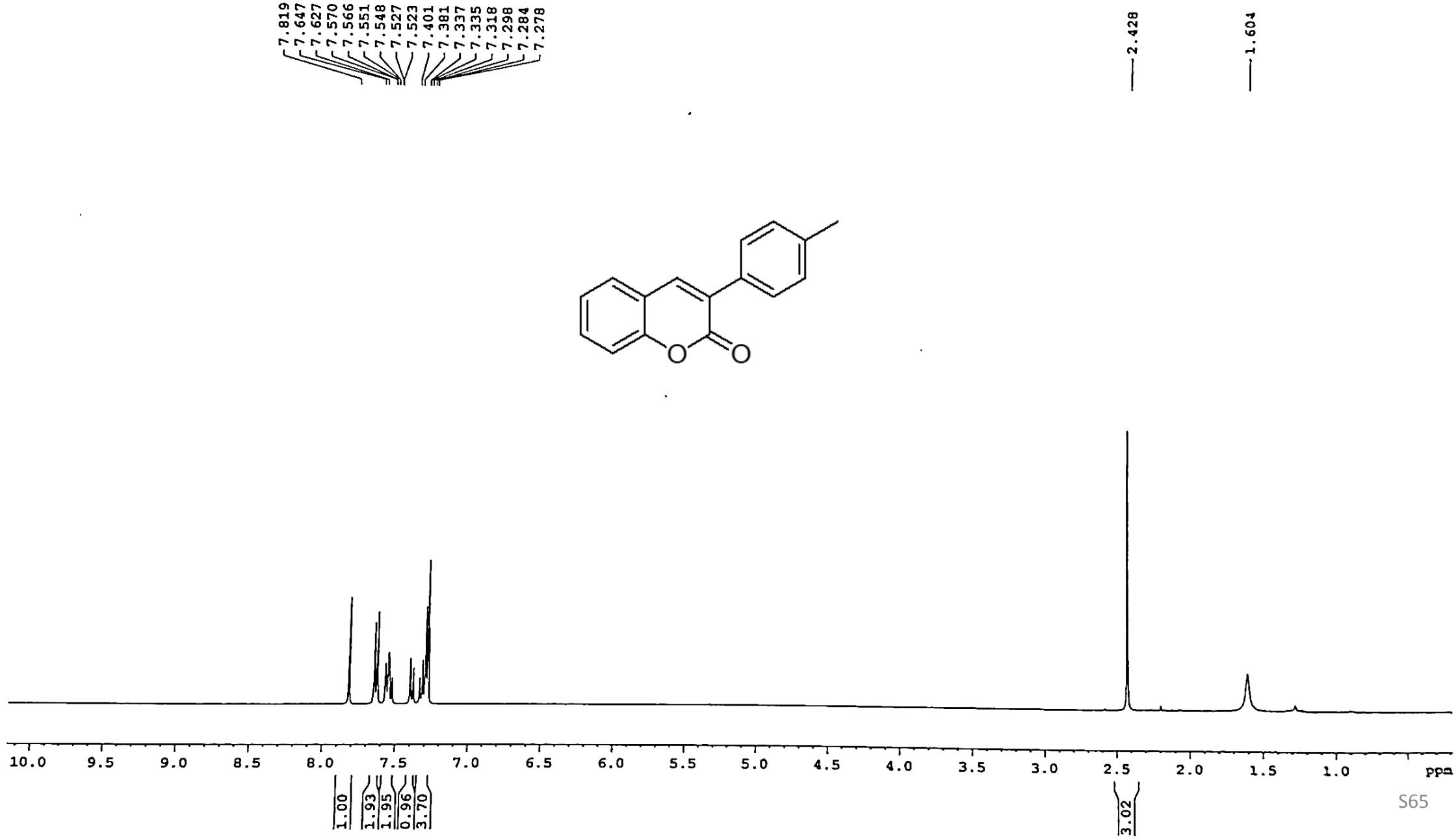
^1H NMR OF 1,1'-(1*E*)-1-Propene-1,3-diylbis[4-methylbenzene] (3aj)



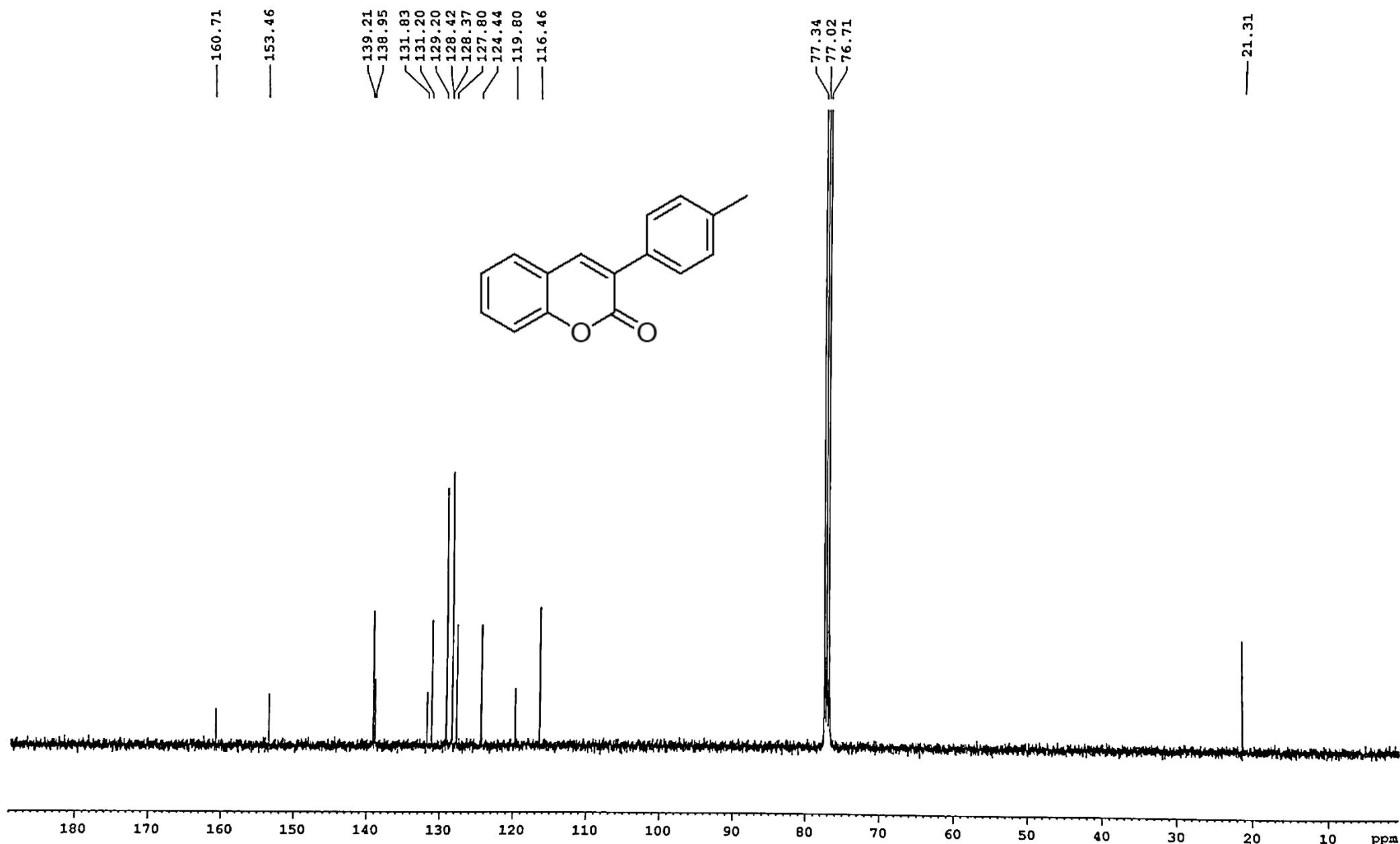
^{13}C NMR OF 1,1'-(1*E*)-1-Propene-1,3-diylbis[4-methylbenzene] (3aj)



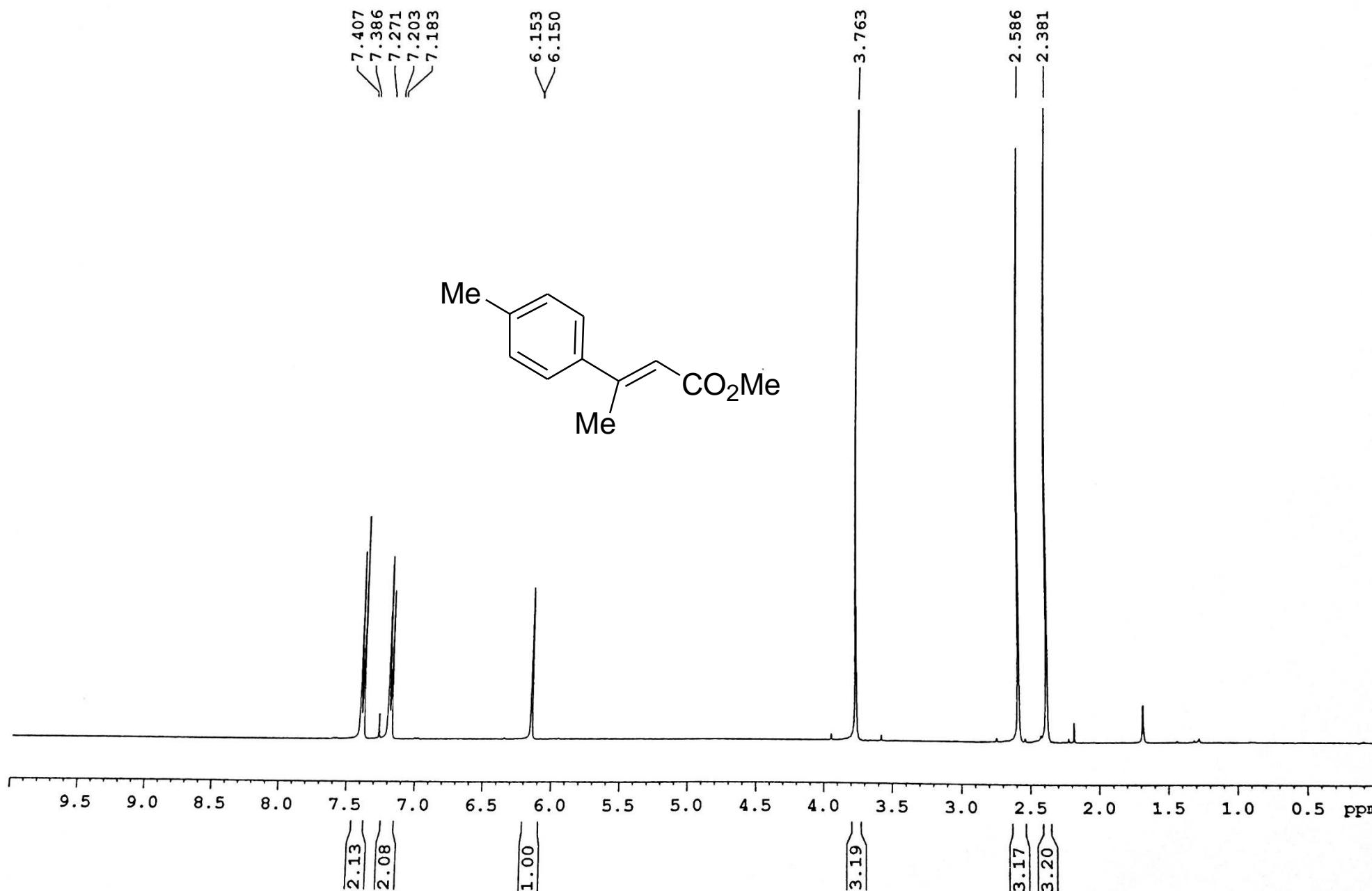
¹H NMR OF 3-p-Tolyl-chromen-2-one (3ak)



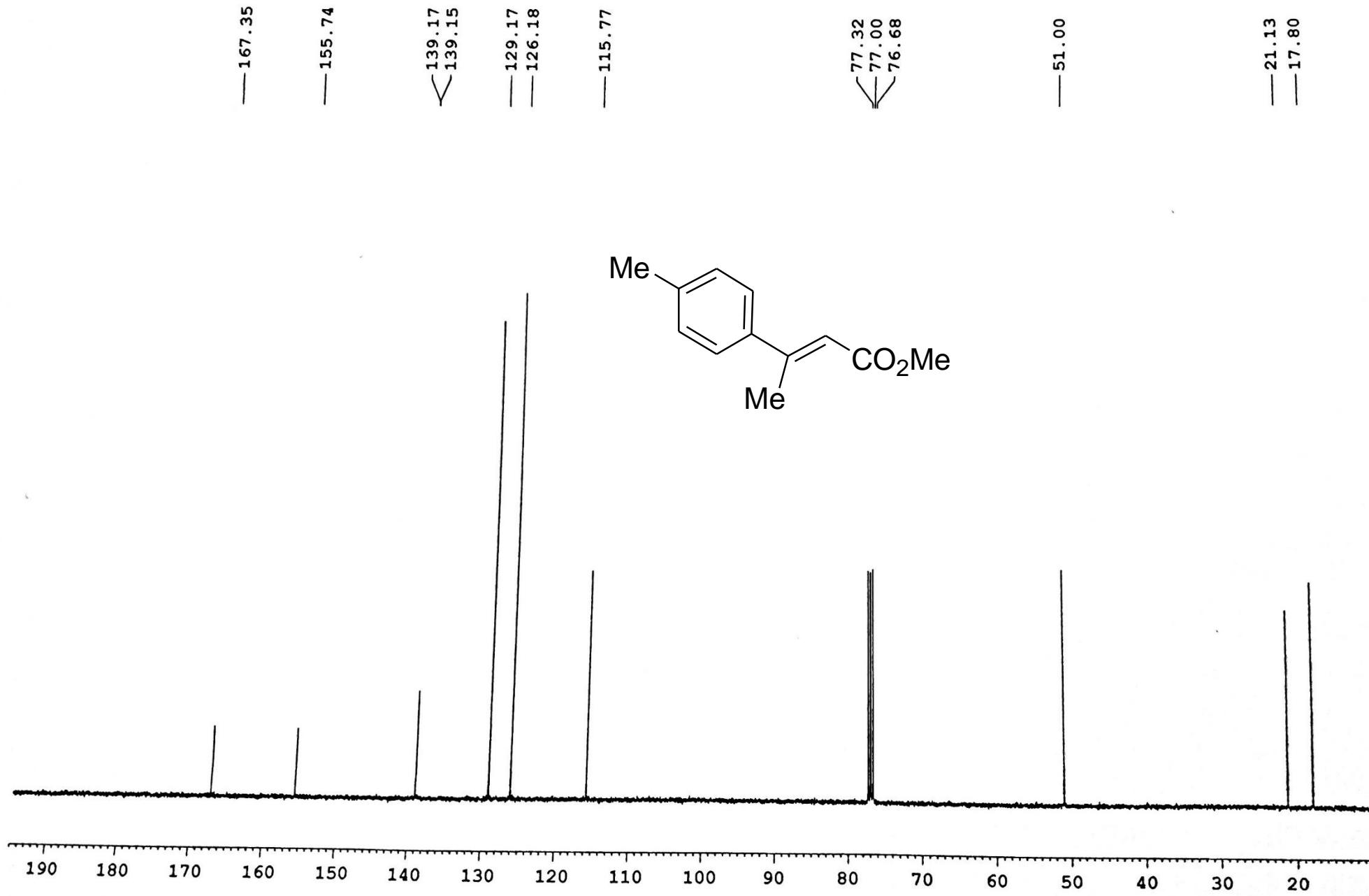
¹³C NMR OF 3-p-Tolyl-chromen-2-one (3ak)



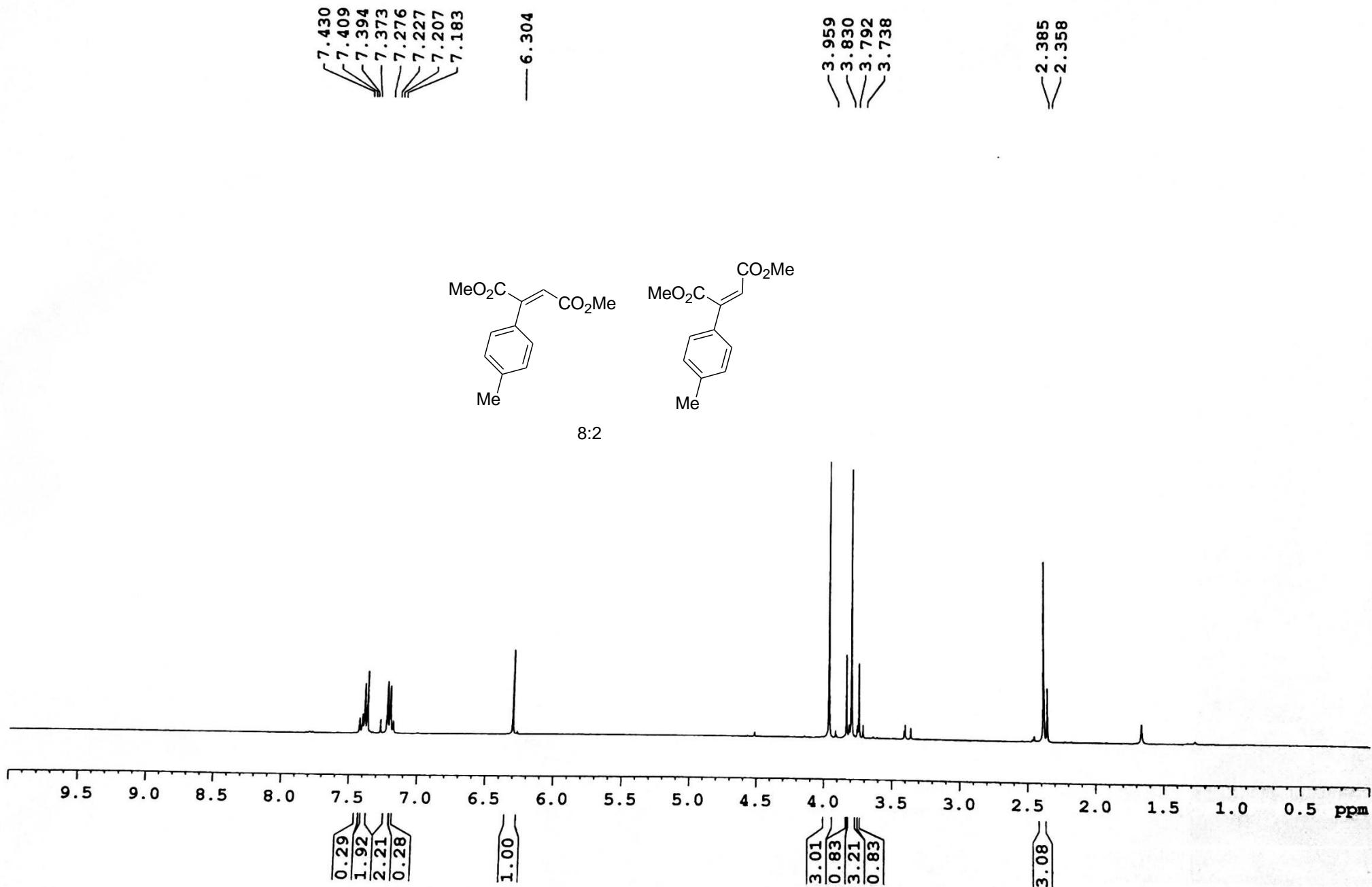
^1H NMR OF 3-p-Tolyl-but-2-enoic acid methyl ester (3al)



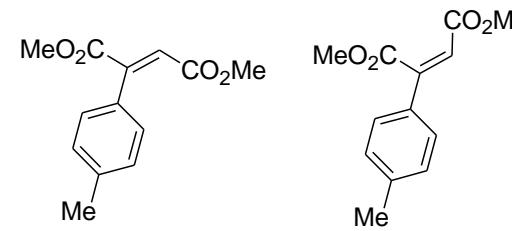
¹³C NMR OF 3-p-Tolyl-but-2-enoic acid methyl ester (3al)



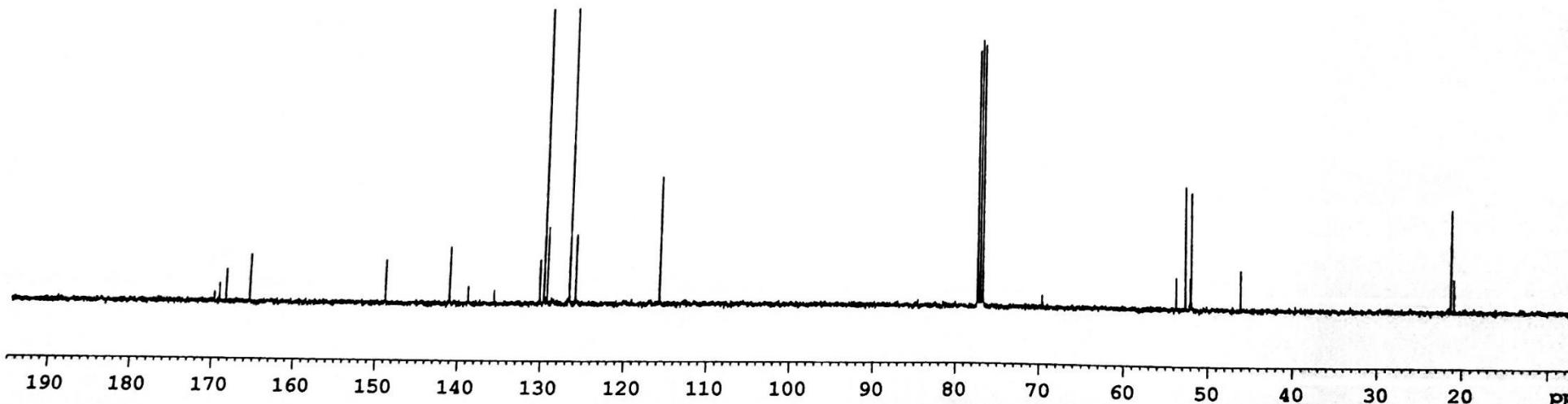
¹H NMR OF 2-p-Tolyl-but-2-enedioic acid dimethyl ester (3am)



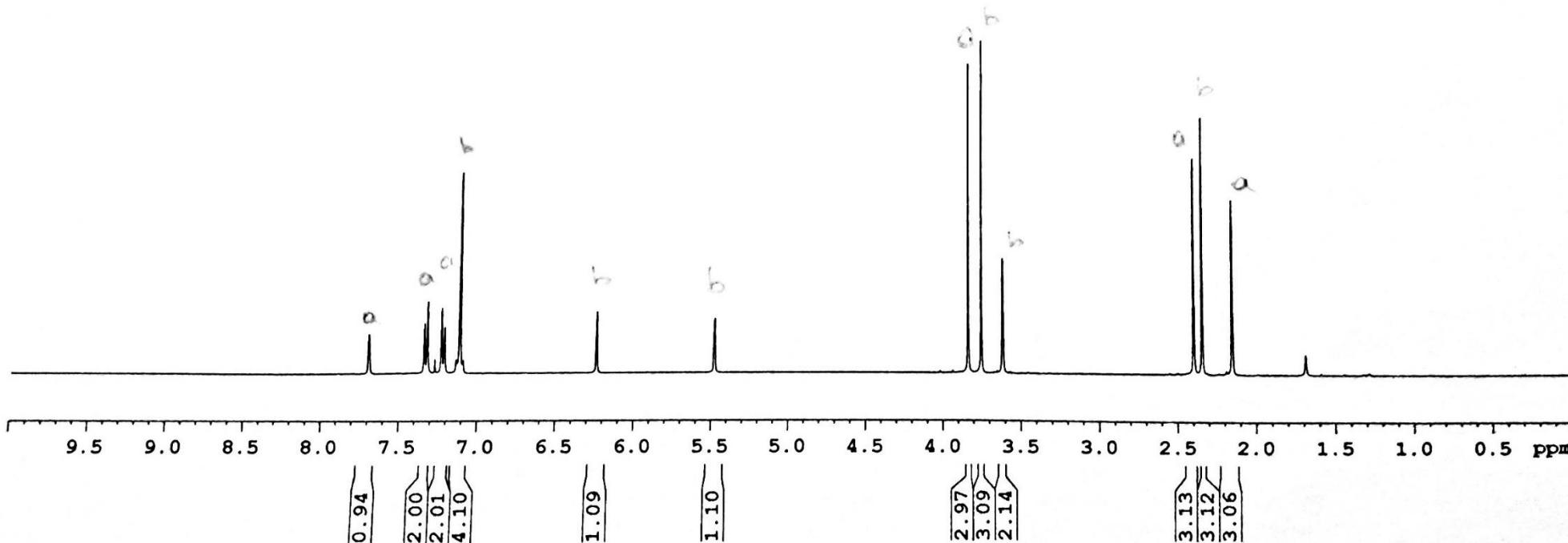
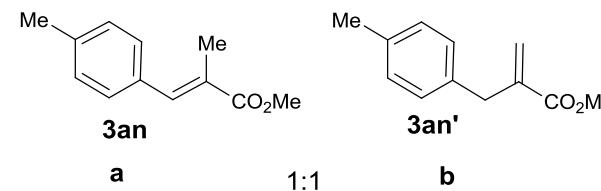
¹³C NMR OF 2-p-Tolyl-but-2-enedioic acid dimethyl ester (3am)



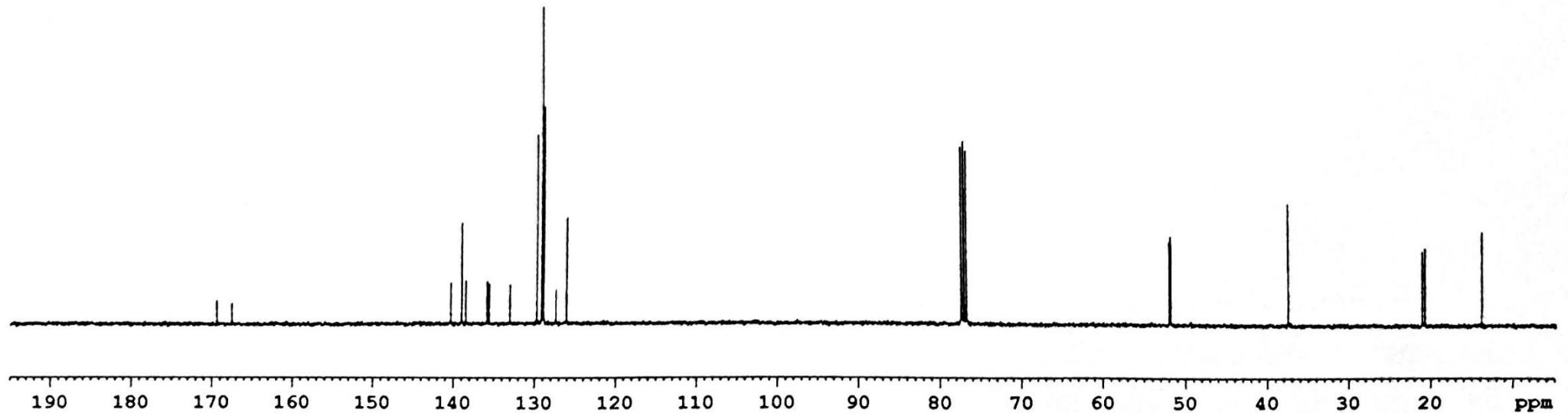
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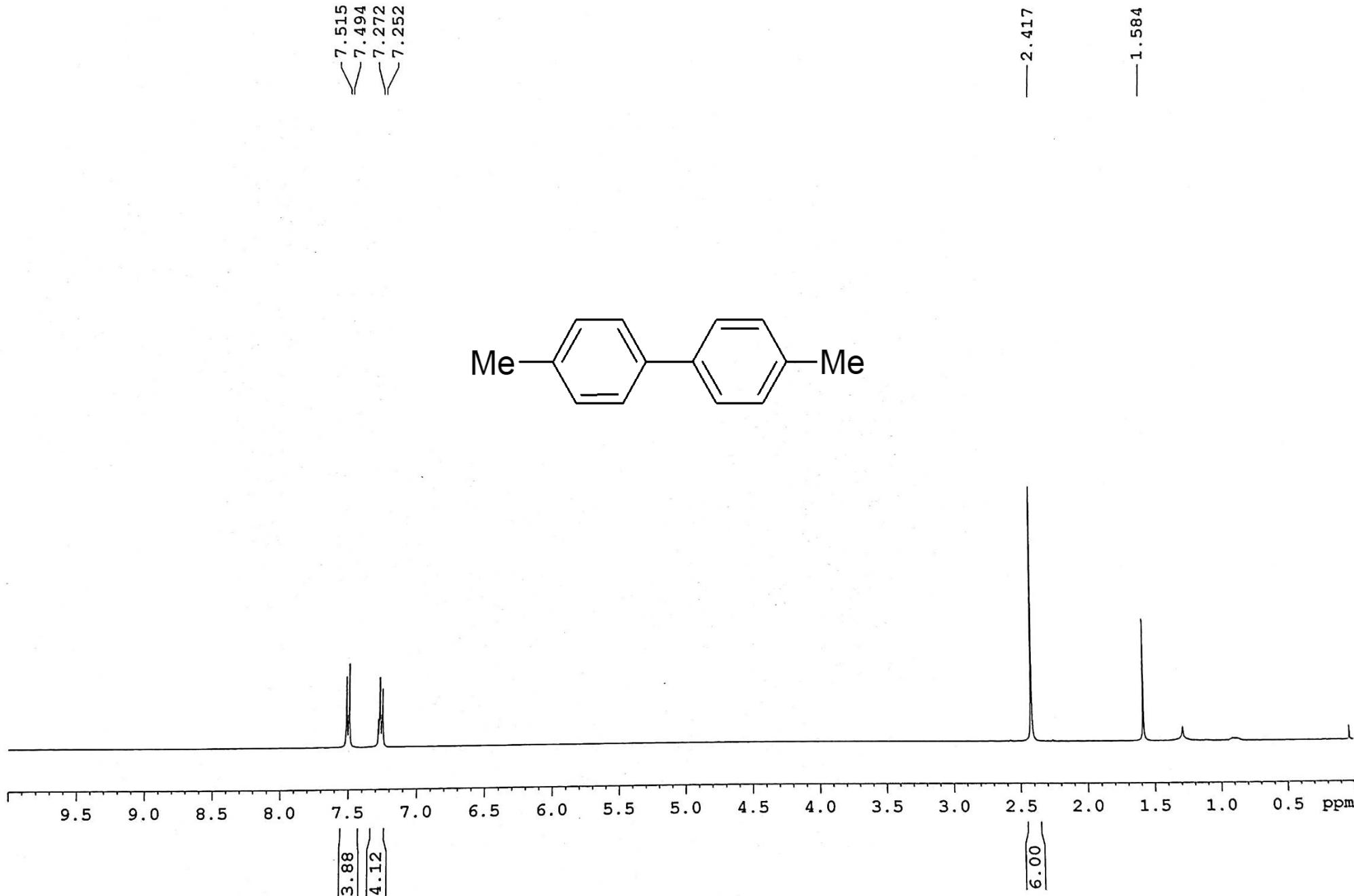
¹H NMR Of 2-Methyl-3-p-tolyl-acrylic acid methyl ester (3an)



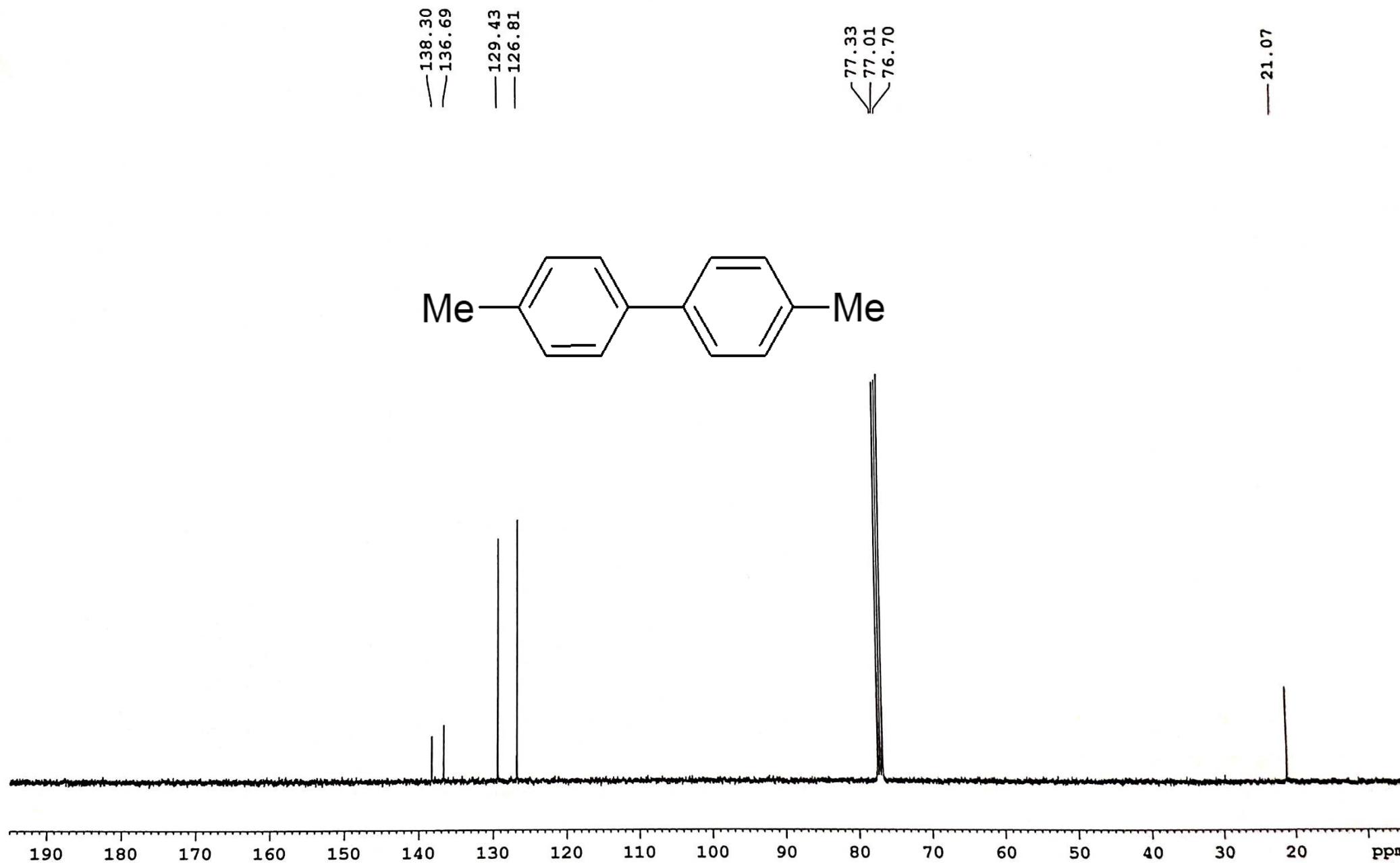
¹³H NMR OF 2-Methyl-3-p-tolyl-acrylic acid methyl ester (3an)



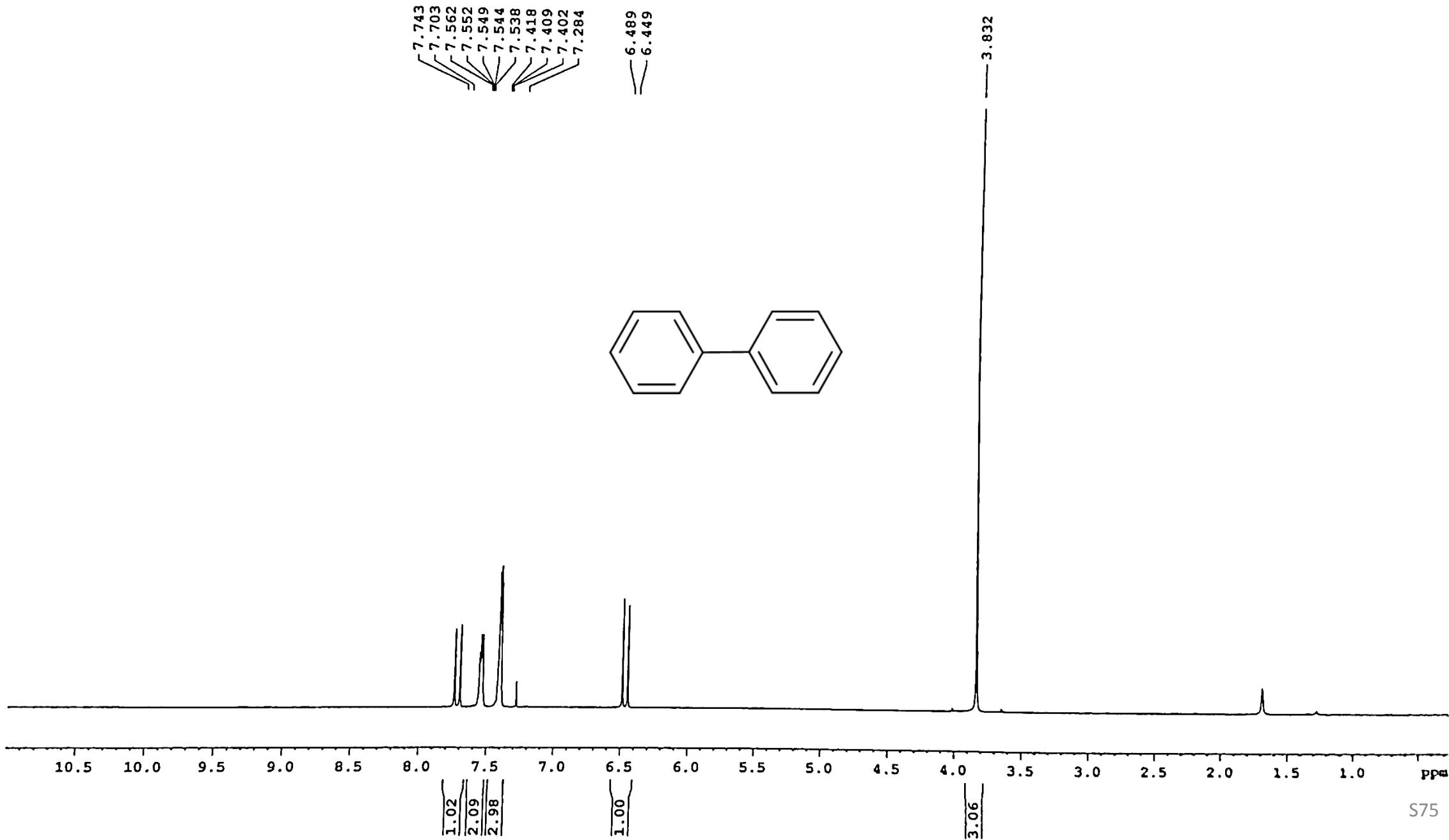
¹H NMR OF 4,4'-Dimethyl-biphenyl (4a)



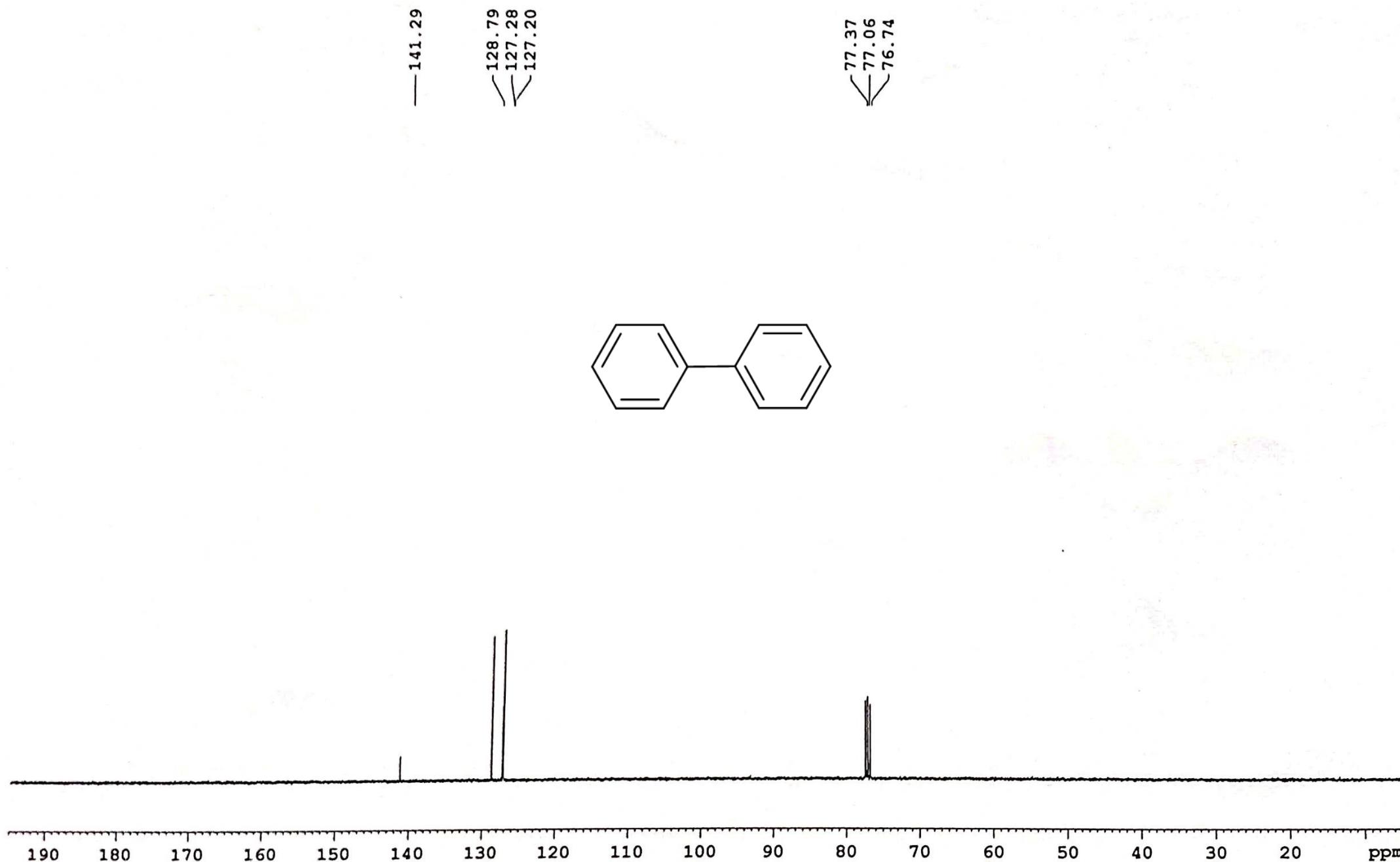
¹³C NMR OF 4,4'-Dimethyl-biphenyl (4a)



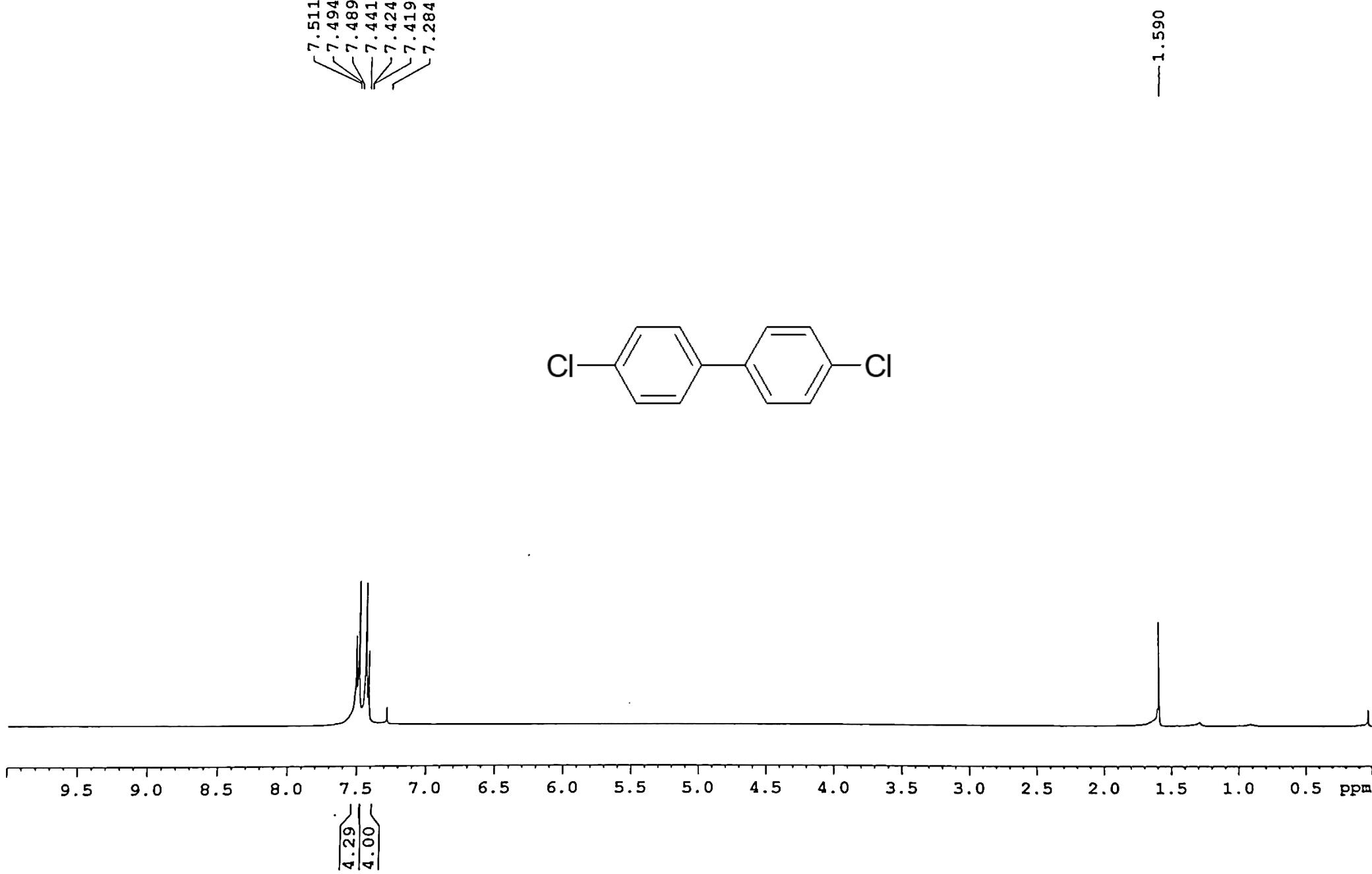
¹H NMR OF Biphenyl (4b)



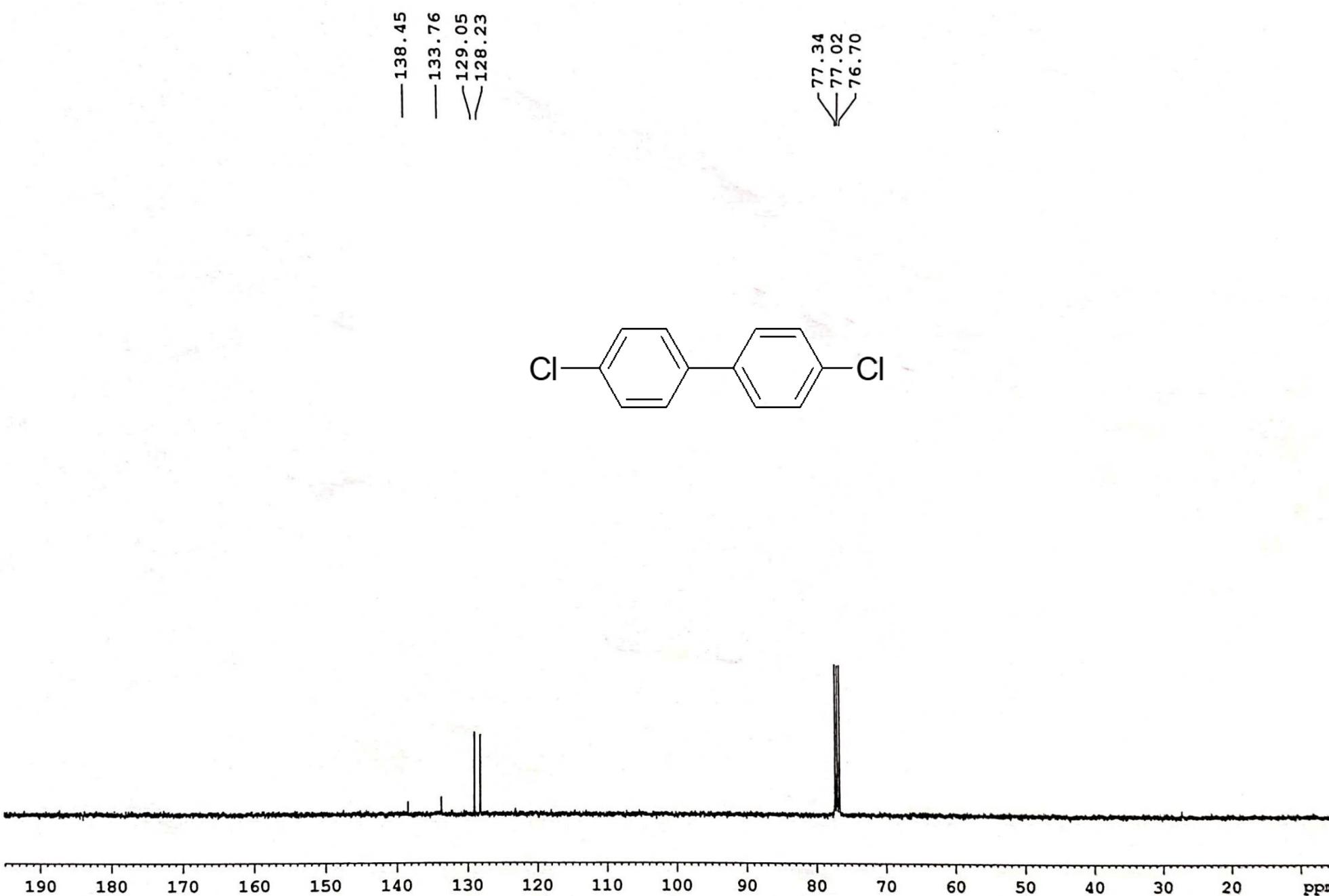
¹³C NMR OF Biphenyl (4b)



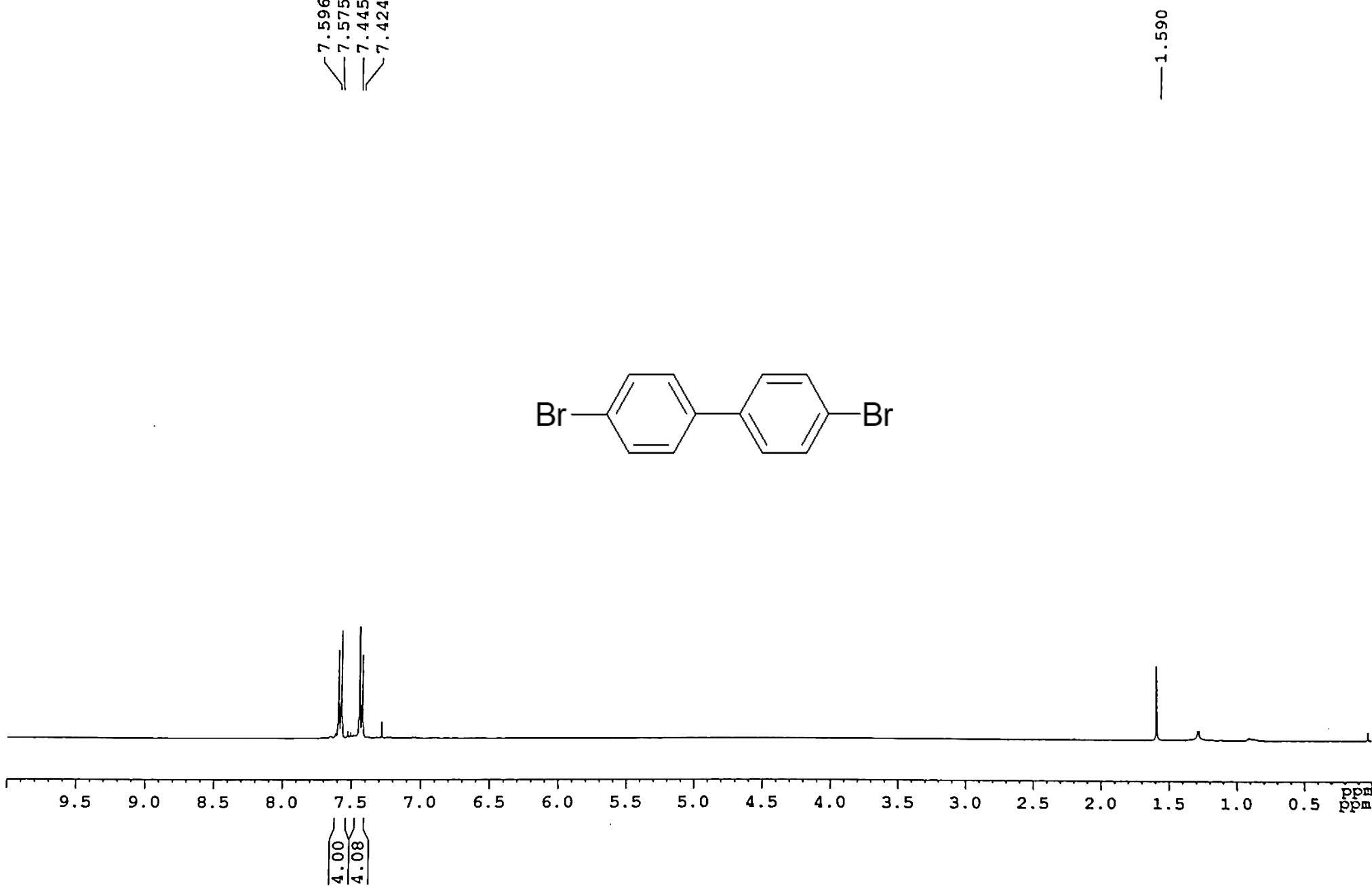
¹H NMR OF 4,4'-Dichloro-biphenyl (4c)



¹³C NMR OF 4,4'-Dichloro-biphenyl (4c)



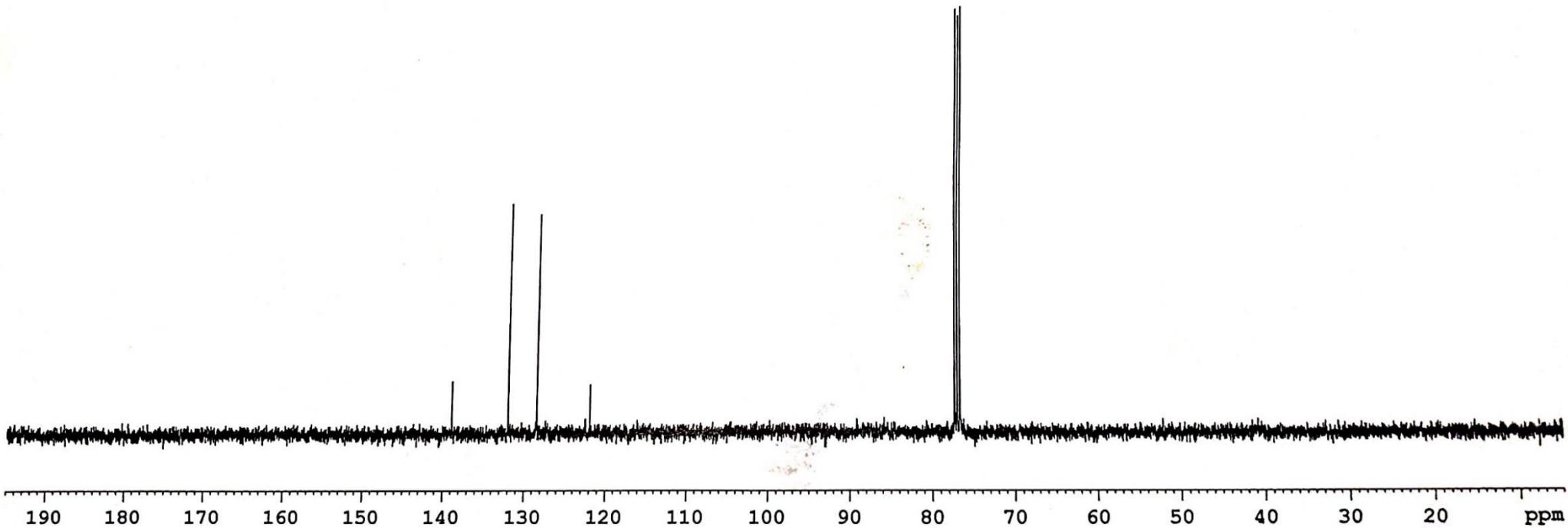
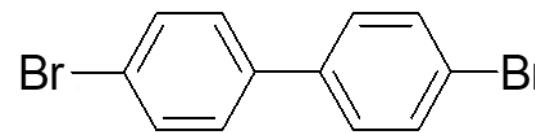
¹H NMR OF 4,4'-Dibromo-biphenyl (4d)



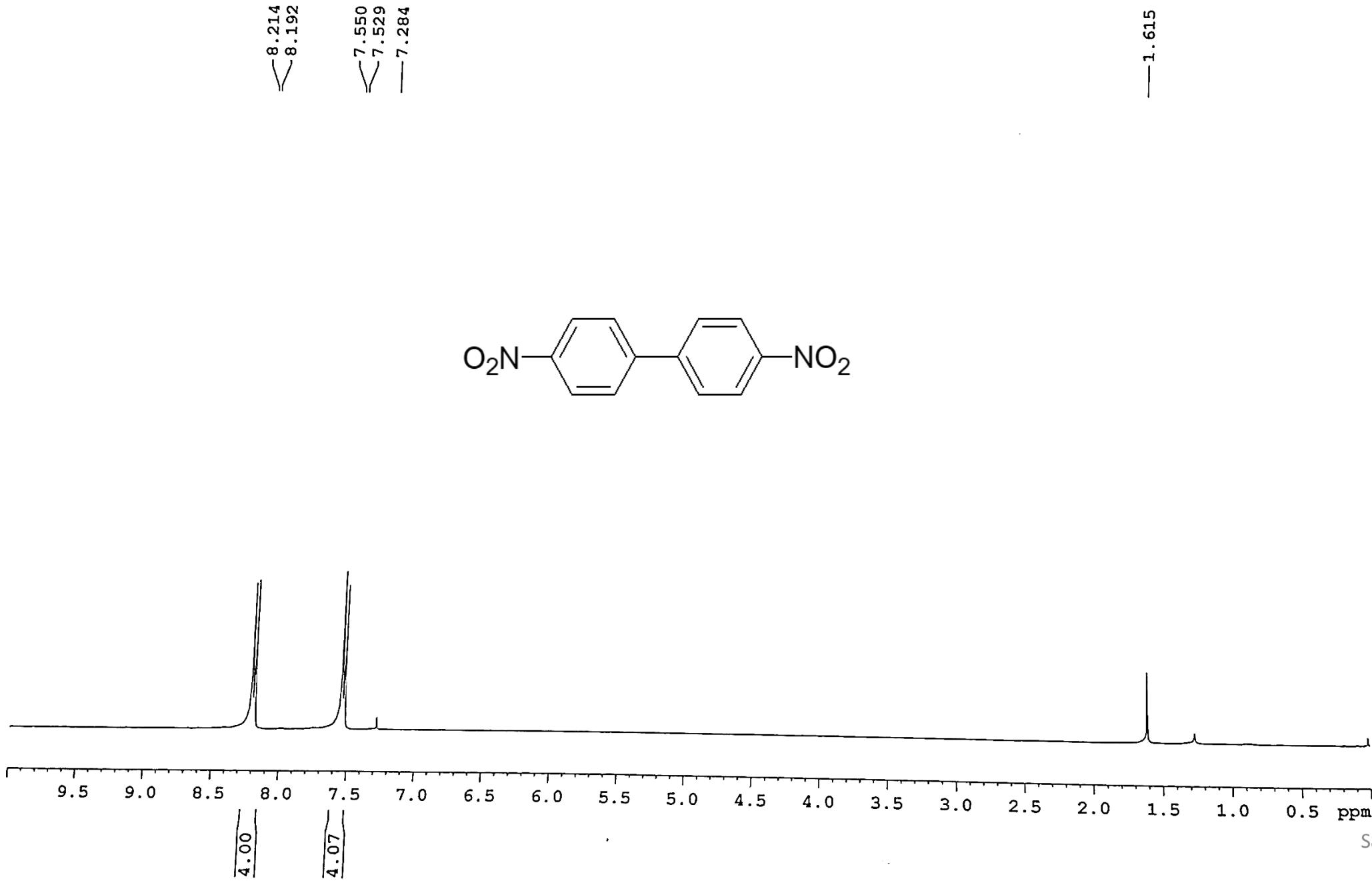
^{13}C NMR OF 4,4'-Dibromo-biphenyl (4d)

— 138.93 — 132.03 — 128.52 — 121.96

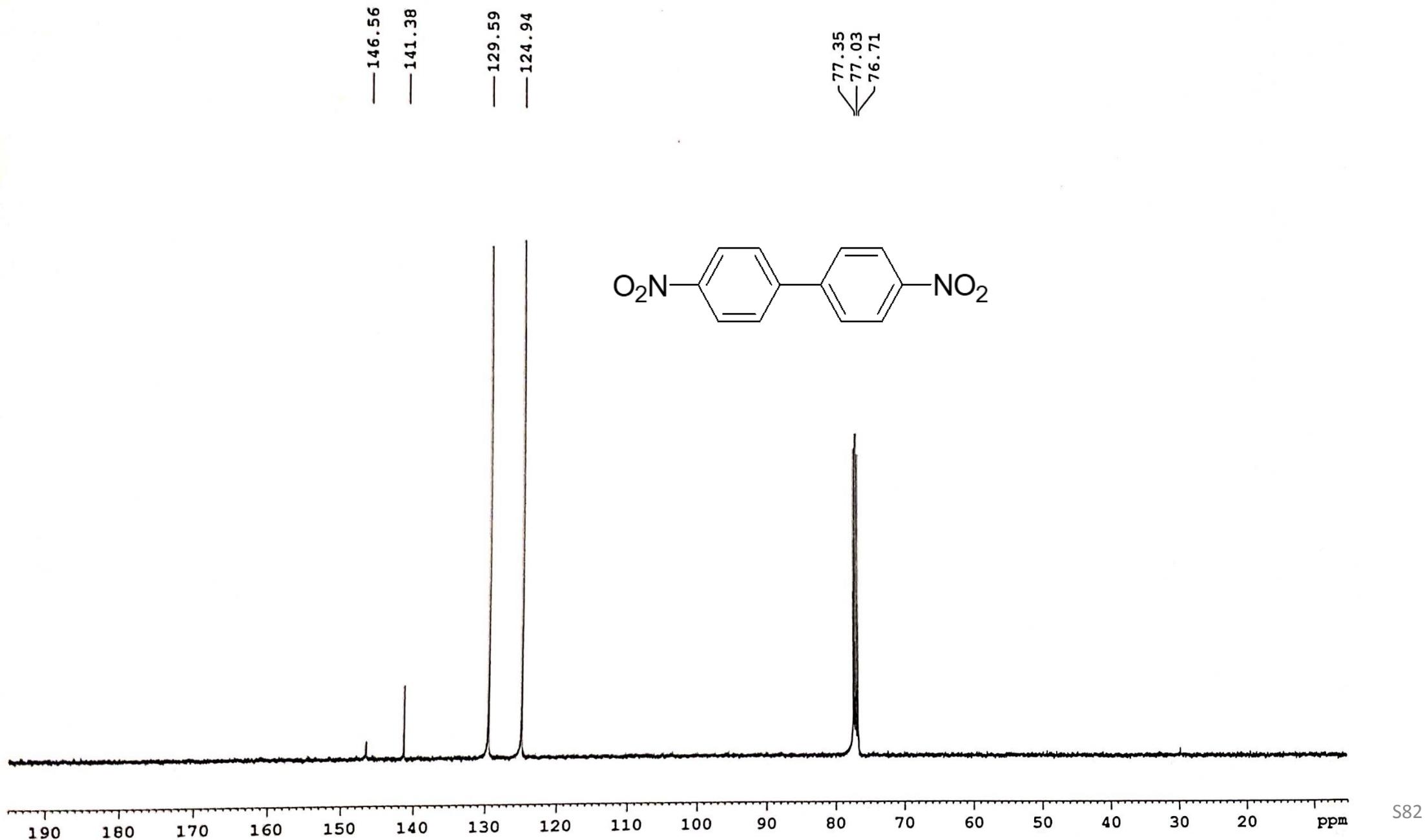
— 77.34 — 77.02 — 76.70



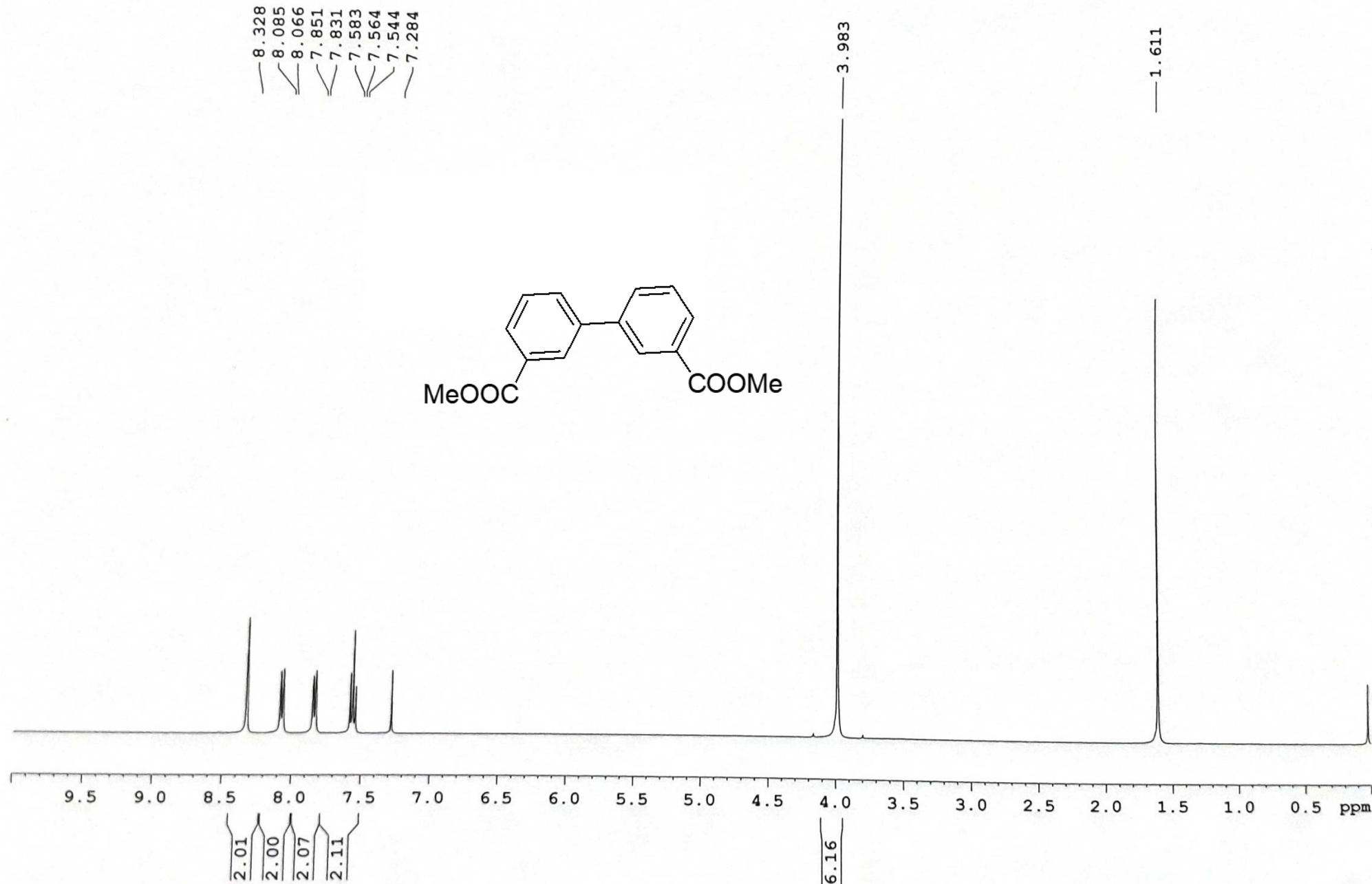
¹H NMR OF 4,4'-Dinitro-biphenyl (4e)



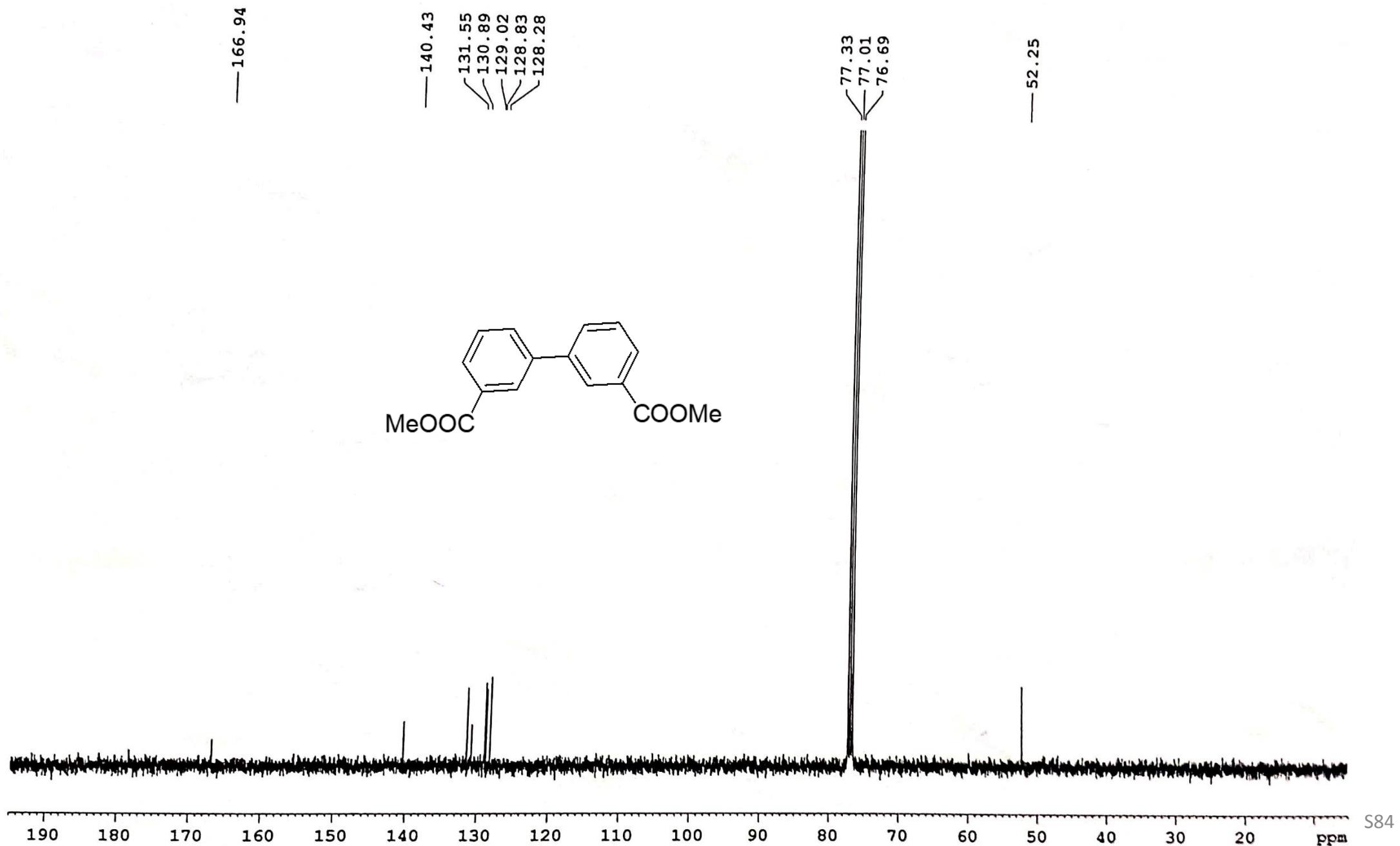
¹³C NMR OF 4,4'-Dinitro-biphenyl (4e)



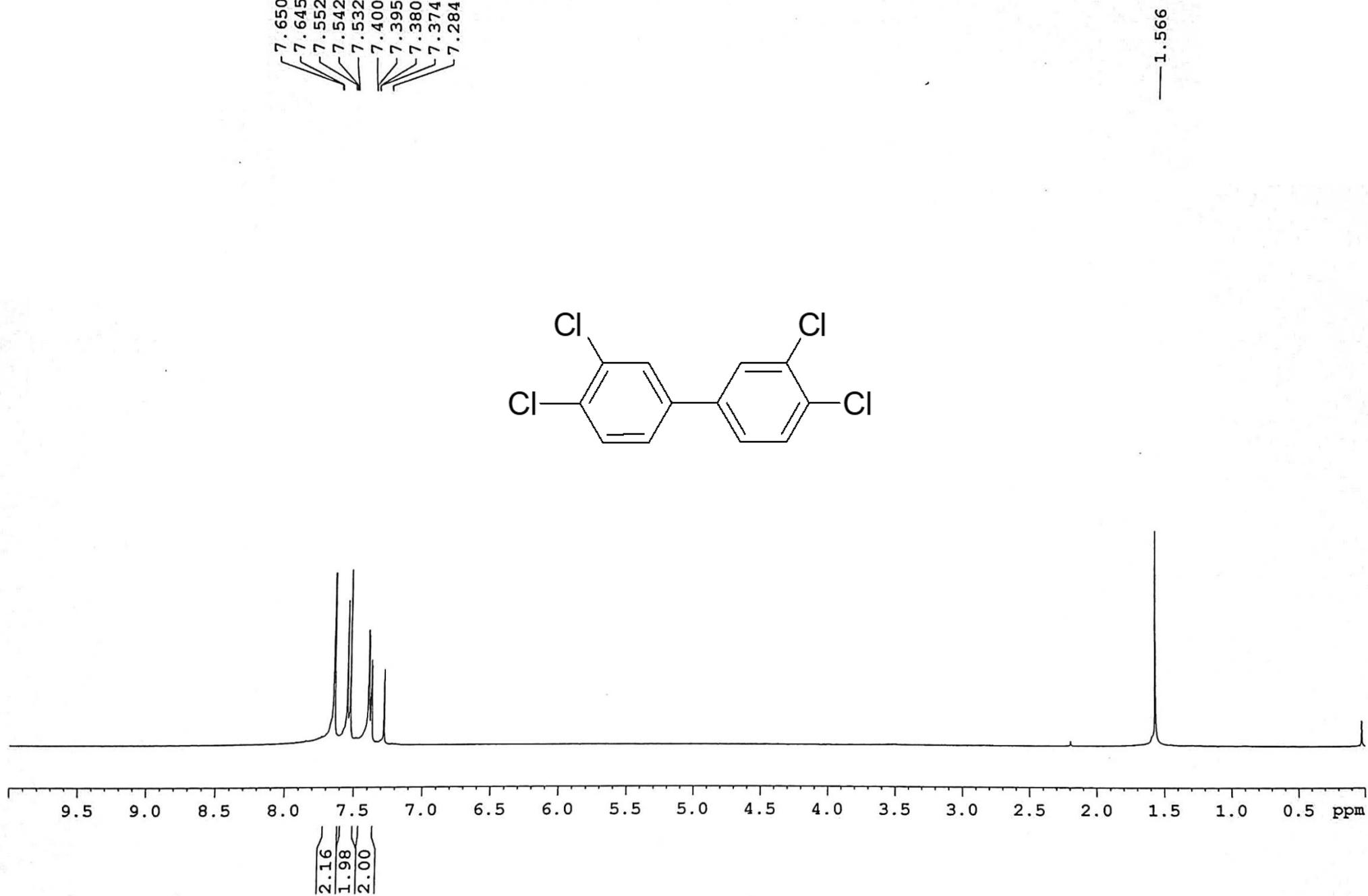
¹H NMR OF Biphenyl-3,3'-dicarboxylic acid dimeethyl ester (4f)



¹³C NMR OF Biphenyl-3,3'-dicarboxylic acid dimeethyl ester (4f)



¹H NMR OF 3,4,3',4'-Tetrachloro-biphenyl (4g)



¹³C NMR OF 3,4,3',4'-Tetrachloro-biphenyl (4g)

