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Carbonylative Sonogashira Coupling of Terminal Alkynes with Aqueous Ammonia

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Supporting Information

Experimental

General poecedure for the carbonylative coupling of phenylethyne (1) with an aryl iodide (2) summarized in Table 1: To a 25 mL of Schlenk tube equipped with a magnetic stirring bar under argon were added $PdCl_2(PPh_3)_2$ (3.5 mg, 0.005 mmol) and THF (3 mL). Phenylethyne (66 μL, 0.6 mmol) and **2** (0.5 mmol) were added successively to the mixture to form a pale yellow solution. Then, aqueous ammonia (0.5 M, 2 mL, 1.0 mmol) was added dropwise via syringe. The atmosphere was replaced with carbon monoxide with a balloon and stirring was continued at room temperature. After the period shown in Table 1, the mixture was passed through a Celite pad and the filtrate was washed with brine. The aqueous layer was extracted with diethyl ether (3 × 15 mL) and the combined organic layers were dried over anhydrous $MgSO_4$, and concentrated in vacuo. The residue was purified by flash chromatography using hexanes-ethyl acetate to afford the corresponding α,β-alkynyl ketones **3**.

1-(4-Methoxyphenyl)-3-phenyl-2-propynone (3a)[1]: Reaction time: 41 h. Purification by flash chromatography (30:1 hexanes:ethyl acetate) afforded the product in 72% yield (85 mg) as a colorless solid. Mp 98 °C, Lit. mp 100 °C. IR (KBr): ν = 2201, 1630 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 3.90 ppm (s, 3H), 6.99 (d, *J* = 8.7 Hz, 2H), 7.38-7.50 (m, 3H), 7.67 (dd, *J* = 8.1, 1.5 Hz, 2H), 8.20 (d, *J* = 8.7 Hz, 2H). ^{13}C NMR (300 MHz, $CDCl_3$): δ 55.54, 86.85, 92.27, 113.83, 120.26, 128.60, 130.22 130.55, 131.93, 132.90, 164.43, 176.63.

1-(2-Methoxyphenyl)-3-phenyl-2-propynone (3b)[2]: Reaction time: 24 h. Purification by flash chromatography (50:1 hexanes:ethyl acetate) afforded the product in 76% yield (90 mg) as a colorless oil. IR (neat): ν = 2199, 1641, 1620, 1597 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 3.97 (s, 3H), 7.01-7.10 (m, 2H), 7.36-7.76 (m, 6H), 8.10 (dd, *J* = 7.5, 1.5 Hz, 2H). ^{13}C NMR (300 MHz, $CDCl_3$): 55.88, 89.12, 91.55, 112.14, 120.26, 120.63, 126.64, 128.54, 130.41, 132.65, 132.92, 135.00, 159.77, 176.73.

1-(3-Methoxyphenyl)-3-phenyl-2-propynone (3c): Reaction time: 51 h. Purification by flash chromatography (50:1 hexanes:ethyl acetate) afforded the product in 81% yield (95 mg) as a colorless oil. IR (neat): ν = 2205, 1642, 1597, 1582 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 3.90 (s, 3H), 7.18 (ddd, *J* = 8.1, 1.7, 1.2 Hz, 1H), 7.40-7.53 (m, 4H), 7.67-7.72

[1] Johnston, K. M.; Shotter, R. G. *J. Chem. Soc., C*. **1967**, *23*, 2476.
[2] Larock, R. C.; Harrison, L. W. *J. Am. Chem. Soc.* **1984**, *106*, 4218.

(m, 3H), 7.87 (ddd, *J* = 7.8, 1.5, 1.2 Hz, 1H). ^{13}C NMR (300 MHz, $CDCl_3$): 55.44, 86.90, 92.97, 112.72, 120.03, 120.94, 122.85, 128.65, 129.61, 130.79, 133.05, 138.17, 159.74., 177.75. HRMS (EI) m/z calcd for $C_{16}H_{16}O_2$= 236.0837, found 236.0845.

3-Phenyl-1-(4-methylphenyl)-2-propynone (3d)[3]: Reaction time: 47 h. Purification by flash chromatography (50:1 hexanes:ethyl acetate) afforded the product in 64% yield (70 mg) as a colorless solid. Mp 70 ^{o}C, Lit. mp 71 ^{o}C. IR (KBr): ν = 2203, 1634, 1608, 1570 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.45 ppm (s, 3H), 7.30-7.45 (m, 5H), 7.68 (d, *J* = ,8.7 Hz, 2H), 8.12 (d, *J* = 8.7 Hz, 2H). ^{13}C NMR (300 MHz, $CDCl_3$): δ 21.79, 86.91, 92.56, 120.17, 128.61, 129.31, 129.66, 130.64, 132.98, 134.53, 145.21, 177.68.

1,3-Diphenyl-2-propynone (3e) [4] : Reaction time: 25 h. Purification by flash chromatography (50:1 hexanes:ethyl acetate) afforded the product in 76% yield (78 mg) as a colorless solid. Mp 46-47 ^{o}C, Lit mp 46-48 ^{o}C. IR (KBr): ν = 2201, 1651 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$): 7.40-7.56 (m, 5H), 7.60 (m, 1H), 7.70 (m, 2H), 8.23 (ddd, *J* = 8.4, 1.8, 1.2 Hz, 2H). ^{13}C NMR (300 MHz, $CDCl_3$): 86.85, 93.11, 120.09, 128.61, 128.67, 129.57, 130.80, 133.06, 134.12, 136.84 178.03.

1-(1-Nahpthyl)-3-phenyl-2-propynone (3f)[5]: Reaction time: 34 h. Purification by flash chromatography (50:1 hexanes:ethyl acetate) afforded the product in 50% yield (64 mg) as a colorless solid. Mp 95 ^{o}C, Lit. 95 ^{o}C. IR (KBr): ν = 2193, 1632, 16.19 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.43-7.71 (m, 8H), 7.91 (d, *J* = 8.7 Hz, 1H), 8.11 (d, *J* = 8.4 Hz, 1H), 8.65 (d, *J* = 6.9 Hz, 1H), 9.23 (d, *J* = 8.4 Hz, 1H). ^{13}C NMR (300 MHz, $CDCl_3$): 88.46, 91.68, 120.32, 124.45, 125.96, 126.74, 128.55, 128.64, 128.93, 130.58, 130.71, 132.92, 133.84, 134.50, 135.08, 179.71.

1-{4-(1-Oxoethyl)phenyl}-3-phenyl-2-propynone (3g): Reaction time: 25 h. Purification by flash chromatography (50:1 hexanes:ethyl acetate) afforded the product in 75% yield (93 mg) as a colorless solid. Mp 103-104 ^{o}C. IR (KBr): ν = 2199, 1686, 1632 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.67 ppm (s, 3H), 7.42-7.54 (m, 3H), 7.69-7.73 (m, 2H), 8.09 (d, *J* = 8.7 Hz, 2H), 8.31 (d, J = 8.7 Hz, 2H). ^{13}C NMR (300 MHz, $CDCl_3$): δ 26.95, 86.75, 94.28, 119.70, 128.42, 128.74, 129.67, 131.10, 133.15, 139.77, 140.75, 177.11, 197.42. HRMS (EI) m/z calcd for $C_{17}H_{12}O_2$ = 248.0837, found 248.0831.

1-(4-Chlorophenyl)-3-phenyl-2-propynone (3i)[6]: Reaction time: 12 h. Purification by flash chromatography (50:1 hexanes:ethyl acetate) afforded the product in 67% yield (80 mg) as a colorless solid. Mp 104-105 ^{o}C, Lit. mp 105 ^{o}C. IR (KBr): ν = 2201, 1655 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.43-7.51 ppm (m, 5H), 7.70 (d, *J* = 8.4 Hz, 2H), 8.16 (d, J = 8.4 Hz, 2H). ^{13}C NMR (300 MHz, $CDCl_3$): 86.52, 93.59, 119.80, 128.68, 128.94, 130.81, 130.93, 133.05, 135.21, 140.65, 176.59.

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[3] Baddar, F. G.; Al-Hajjar, F. H.; El-Rayyes, N. R. *J. Heterocyclic Chem.*, **1976**, *13*, 195.
[4] Nahm, S.; Weinreb, S. M. *Tetrahydron Lett*. **1981**, *22*, 3815.
[5] Arcadi, A.; Cacchi, S.; Marinelli, F.; Pace, P.; Sanzi, G. *Synlett*, **1995**, 823.
[6] Delaude, L.; Masdue, A. M.; Alper, H. *Synthesis*, **1994**, 1149.

General procedure for the carbonylative coupling of alkylalkynes 5 with aryl halides 2 summarized in Table 2: To a solution of an alkylalkyne (**5**, 0.6 mmol), **2** (0.5 mmol), $PdCl_2(PPh_3)_2$ (17.5 mg, 0.025 mmol) and CuI (1.9 mg, 0.01 mmol) in THF (3 mL) was added aqueous ammonia (0.5 M, 2 mL, 1.0 mmol). The atmosphere was replaced with carbon monoxide with a balloon and stirring was continued at room temperature for the period shown in Table 2. The resulting mixture was passed through a Celite pad and the filtrate was washed with brine. The aqueous layer was extracted with diethyl ether (3 × 15 mL) and the combined organic layers were dried over $MgSO_4$, and concentrated in vacuo. The residue was purified by flash chromatography on silica gel.

1-(4-Methoxyphenyl)-2-nonyn-1-one (6aa): Reaction time: 24 h. Purification by flash chromatography (30:1 hexanes:ethyl acetate) afforded the product in 74% yield (90 mg) as a colorless oil. IR (neat): ν = 2932, 2858, 2199, 1638, 1597 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 0.93 ppm (t, *J* = 6.6 Hz, 3H), 1.31-1.68 (m, 8H), 2.48 (t, *J* = 6.9 Hz, 2H), 3.88 (s, 3H), 6.94 (d, *J* = 8.4 Hz, 2H), 8.11 (d, *J* = 6.6 Hz, 2H). ^{13}C NMR (300 MHz, $CDCl_3$): 13.98, 19.15, 22.45, 27.78, 28.60, 31.19, 55.51, 79.59, 95.97, 113.65, 130.29, 131.86, 164.21, 176.95. HRMS (EI) m/z calcd for $C_{16}H_{20}O_2$ = 244.1463, found 244.1461.

1-(4-Methylphenyl)-2-nonyn-1-one (6ad): Reaction time: 24 h. Purification by flash chromatography (30:1 hexanes:ethyl acetate) afforded the product in 78% yield (89 mg) as a colorless oil. IR (neat): ν = 2955, 2936, 2859, 2199, 1646, 1605 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 0.91 ppm (t, *J* = 7.2 Hz, 3H), 1.20-1.75 (m, 8H), 2.49 (t, *J* = 7.2 Hz, 2H), 7.27 (d, *J* = 8.7 Hz, 2H), 8.03 (d, *J* = 7.2 Hz, 2H). ^{13}C NMR (300 MHz, $CDCl_3$): δ 13.94, 19.13, 21.69, 22.42, 27.73, 28.56, 31.16, 79.67, 96.27, 129.12, 129.60, 134.60, 144.81, 177.90. HRMS (EI) m/z calcd for $C_{16}H_{20}O$ = 228.1514, found 228.1511.

1-(1-Naphthyl)-2-nonyn-1-one (6af): Reaction time: 25 h. Purification by flash chromatography (30:1 hexanes:ethyl acetate) afforded the product in 60% yield (79 mg) as a colorless oil. IR (neat): ν = 3050, 2955, 2936, 2859, 2209, 1638 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 0.91 ppm (t, *J* = 6.9 Hz, 3H), 1.32-1.69 (m, 8H), 2.51 (t, *J* = 7.2 Hz, 2H), 7.54-7.66 (m, 3H), 7.89 (d, *J* = 7.2 Hz, 1H), 8.06 (d, *J* = 8.1 Hz, 1H), 8.54 (dd, *J* = 7.5, 1.5 Hz, 1H), 9.18 (d, *J* = 8.7 Hz, 1H). ^{13}C NMR (300 MHz, $CDCl_3$): δ 13.99, 19.18, 22.46, 27.74, 28.63, 31.19, 81.25, 95.37, 124.34, 125.95, 126.58, 128.44, 128.73, 130.63, 132.94, 133.75, 134.41, 134.76, 180.00. HRMS (EI) m/z calcd for $C_{19}H_{20}O$ = 264.1514, found 264.1518.

1-{4-(1-Oxoethyl)phenyl}-2-nonyn-1-one (6ag): Reaction time: 30 h. Purification by flash chromatography (30:1 hexanes:ethyl acetate) afforded the product in 47% yield (60 mg) as a colorless oil. IR (neat): ν = 2967, 2930, 2859, 2238, 2203, 1691, 1649 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 0.91 ppm (t, *J* = 6.9 Hz, 3H), 1.20-1.75 (m, 8H), 2.53 (t, *J* = 7.8 Hz, 2H), 2.66 (s, 3H), 8.04 (d, *J* = 6.6 Hz, 2H), 8.22 (d, *J* = 6.6 Hz, 2H). ^{13}C NMR (300 MHz, $CDCl_3$): δ 13.99, 19.23, 22.44, 26.95, 27.65, 28.60, 31.16, 79.57, 98.27, 128.29, 129.64, 139.82, 140.56, 177.33, 197.51. HRMS (EI) m/z calcd for $C_{17}H_{20}O_2$ = 256.1463, found 256.1474.

1-(2-Aminophenyl)-2-nonyn-1-one (6ah): Reaction time: 40 h. Purification by flash chromatography (30:1 hexanes:ethyl acetate) afforded the product in 71% yield (81 mg) as a pale yellow oil. IR (neat): ν = 3449, 3341, 2955, 2930, 2859, 2226, 1622, 1586 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 0.90 ppm (t, *J* = 6.9 Hz, 3H), 1.25-1.72 (m, 8H), 2.48 (t, *J* = 7.8 Hz, 2H), 6.29 (brs, 2H), 6.61-6.71 (m, 2H), 7.29-7.31 (m, 1H), 8.06-8.09 (m, 1H). ^{13}C NMR (300 MHz, $CDCl_3$): δ 13.93, 19.08, 22.40, 27.73, 28.53, 31.13, 79.75, 95.77, 115.80, 116.58, 118.68, 134.53, 134.92, 150.89, 179.83. HRMS (EI) m/z calcd for $C_{15}H_{19}ON$ = 229.1467, found 229.1474. Anal. Calcd for $C_{15}H_{19}ON$: C, 78.56; H, 8.35; N, 6.11. Found: C, 78.76; H, 8.28; N, 5.99.

1-(4-Chlorophenyl)-2-nonyn-1-one (6ai): Reaction time: 26 h. Purification by flash chromatography (30:1 hexanes:ethyl acetate) afforded the product in 70% yield (87 mg) as a colorless oil. IR (neat): ν = 2930, 2859, 2238, 2201, 1637, 1597, 1576 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 0.91 (s, 3H), 1.20-1.75 (m, 8H), 2.50 (t, *J* = 7.2 Hz, 2H), 7.44 (d, *J* = 8.1 Hz, 2H), 8.05 (d, *J* = 8.1 Hz, 2H). ^{13}C NMR (300 MHz, $CDCl_3$): 13.95, 19.16, 22.42, 27.66, 28.58, 31.14, 79.33, 97.45, 128.77, 130.78, 135.27, 140.37, 176.81 HRMS (EI) m/z calcd for $C_{15}H_{17}ClO$ = 248.0968, found 248.0967.

1,4-Di(1-oxo-2-nonyn-1-yl)benzene (8): Reaction time: 27 h. Purification by flash chromatography (30:1 hexanes:ethyl acetate) afforded the product in 53% yield (93 mg) as a colorless oil. IR (neat): ν = 2957, 2930, 2859, 2236, 2199, 1657 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 0.91 ppm (t, *J* = 7.2 Hz, 6H), 1.30-1.76 (m, 16H), 2.53 (t, *J* = 6.9 Hz, 4H), 8.22 (s, 4H). ^{13}C NMR (300 MHz, $CDCl_3$): δ 13.99, 19.25, 22.45, 27.66, 28.62, 31.17, 79.63, 98.40, 129.47, 140.33, 177.33. HRMS (EI) m/z calcd for $C_{24}H_{30}O_2$ = 350.2246, found 350.2237.

1-(1-Octyn-1-yl)-4-(1-oxo-2-nonyn-1-yl)benzene (9): Reaction time: 27 h. Purification by flash chromatography (30:1 hexanes:ethyl acetate) afforded the product in 37% yield (60 mg) as a colorless oil. IR (neat): ν = 2957, 2932, 2859, 2234, 2201, 1647, 1601 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 0.90 ppm (t, *J* = 6.9 Hz, 6H), 1.25-1.70 (m, 16H), 2.40-2.55 (m, 4H), 7.46 (d, *J* = 8.7 Hz, 2H), 8.04 (d, *J* = 8.7 Hz,2H). ^{13}C NMR (300 MHz, $CDCl_3$): δ 14.00, 14.03, 19.21, 19.55, 22.47, 22.52. 27.72, 28.46, 28.59, 28.62, 31.19, 31.30, 79.59, 80.18, 95.15, 97.13, 129.34, 129.96, 131.52, 135.51, 177.43. HRMS (EI) m/z calcd for $C_{23}H_{30}O$ = 322.2297, found 322.2288.

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4,4-Dimethyl-1-(4-methoxyphenyl)-2-pentyn-1-one (6ba): Reaction time: 20 h. Purification by flash chromatography (30:1 hexanes:ethyl acetate) afforded the product in 87% yield (94 mg) as a colorless oil. IR (neat): ν = 2993, 2932, 2903, 2842, 2211, 1647, 1597, 1576 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 1.37 (s, 9H), 3.88 (s, 3H), 6.95 (d, *J* = 8.4 Hz, 2H), 8.10 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR (300 MHz, $CDCl_3$): 27.95, 30.18, 55.50, 78.02, 103.05, 113.66, 130.41, 131.83, 164.17, 177.06. HRMS (EI) m/z calcd for $C_{14}H_{16}O_2$ = 216.1150, found 216.1158.

4,4-Dimethyl-1-phenyl-2-pentyn-1-one (6be)[7]: Reaction time: 24 h. Purification by flash chromatography (30:1 hexanes:ethyl acetate) afforded the product in 77% yield (71

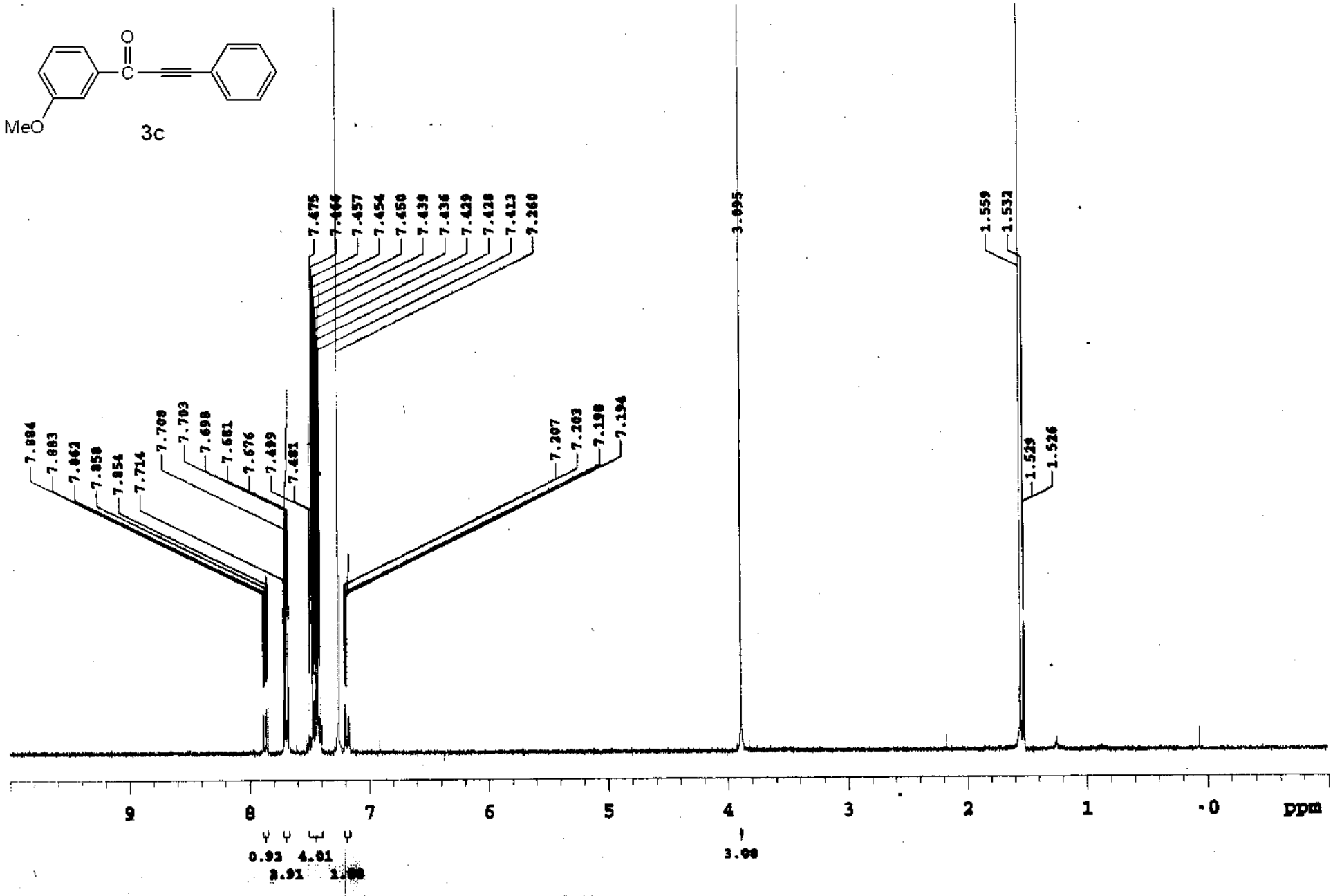
[7] Smith III, A. B.; Levenberg, P. A.; Suits, J. Z. *Synthesis*, **1986**, 184.

mg) as a colorless oil. IR (neat): ν = 3065, 2973, 2932, 2870, 2212, 1647, 1619, 1599, 1582 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 1.39 (s, 9H), 7.48-7.623 (m, 3H), 8.12 (dd, *J* = 8.1, 1.2 Hz, 2H). ^{13}C NMR (300 MHz, $CDCl_3$): 28.01, 30.14, 78.06, 103.96, 128.44, 129.47, 133.79, 136.97, 178.36.

4,4-Dimethyl-1-(1-naphthyl)-2-pentyn-1-one (6bf): Reaction time: 19 h. Purification by flash chromatography (30:1 hexanes:ethyl acetate) afforded the product in 69% yield (82 mg) as a colorless oil. IR (neat): ν = 2973, 2930, 2869, 2207, 1637, 1619, 1591, 1574 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 1.40 (s, 9H), 7.53-7.65 (m, 3H), 7.88 (d, *J* = 7.2 Hz, 1H), 8.06 (d, *J* = 8.1 Hz, 1H), 8.50 (d, *J* = 7.2 Hz, 1H), 9.15 (d, *J* = 8.7 Hz, 1H). ^{13}C NMR (300 MHz, $CDCl_3$): 27.96, 30.14, 79.79, 102.31, 124.35, 125.96, 126.57, 128.43, 128.67, 130.64, 133.19, 133.76, 134.13, 134.66, 180.13. HRMS (EI) m/z calcd for $C_{17}H_{16}O$ = 236.1201, found 236.1208.

4-Hydroxy-1-(4-methoxyphenyl)-4-methyl-2-pentyn-1-one (6ca): Reaction time: 46 h. Purification by flash chromatography (30:1 hexanes:ethyl acetate) afforded the product in 55% yield based on ^1H NMR. Calculation was based on signals of methoxy groups at 3.89 (s, 3H for **6ca**) and 3.78 (s, 3H for **2a**).

5-Hydroxy-1-(4-methoxyphenyl)-2-pentyn-1-one (6da): Reaction time: 48 h. Purification by flash chromatography (3:1 hexanes:ethyl acetate) afforded the product in 56% yield (57 mg) as a colorless solid. Mp 104 ^oC. IR (KBr): ν = 3420, 2204, 1635 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 2.44 (brs, 1H), 2.76(t, *J* = 6.3 Hz, 2H), 3.88 (s, 3H), 3.89 (t, *J* = 6.3 Hz, 2H), 6.95 (d, *J* = 90 Hz, 2H), 8.11 (d, *J* = 9.0 Hz, 2H). ^{13}C NMR (300 MHz, $CDCl_3$): 23.52, 55.54, 60.27, 80.65, 92.62, 113.78, 129.93, 132.03, 164.48, 176.90. HRMS (EI) m/z calcd for $C_{12}H_{12}O_3$ = 204.0786, found 204.0794.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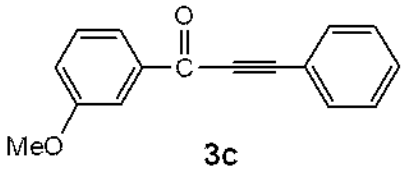
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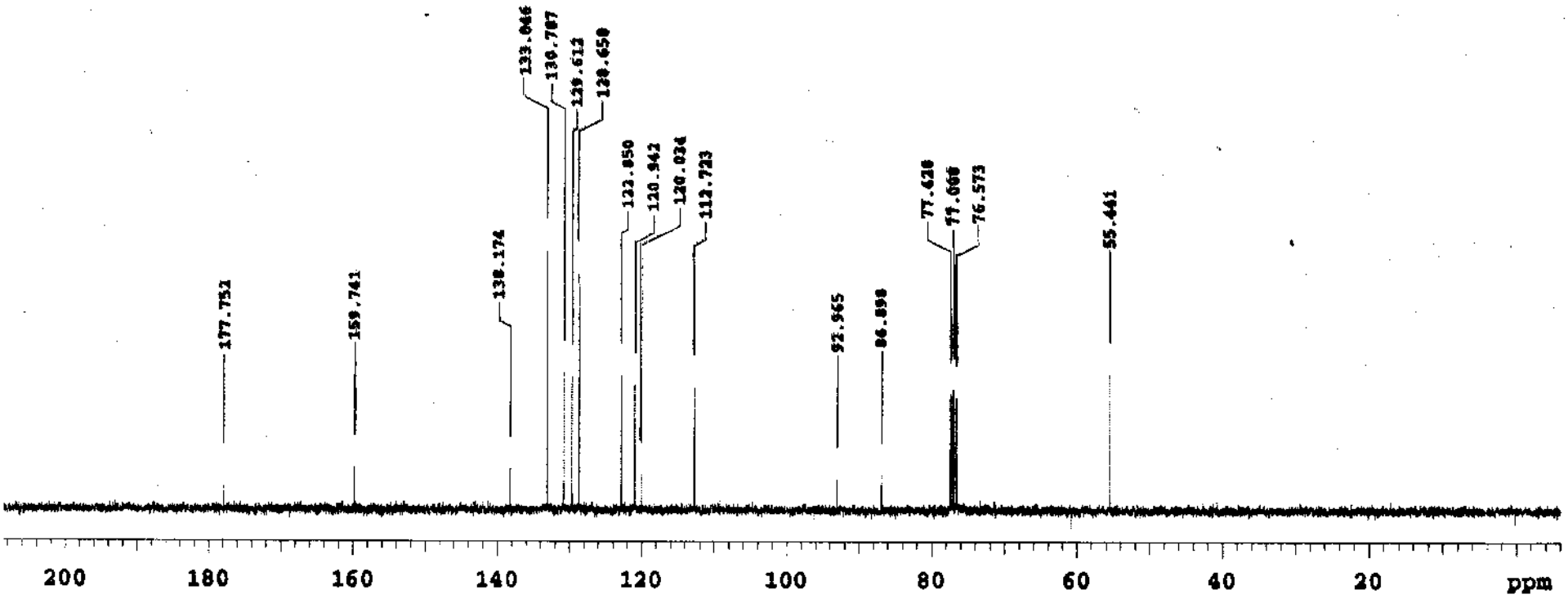
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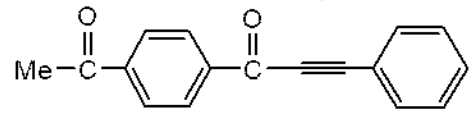
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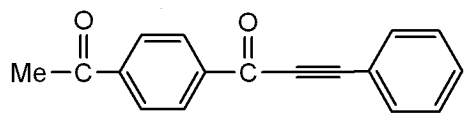
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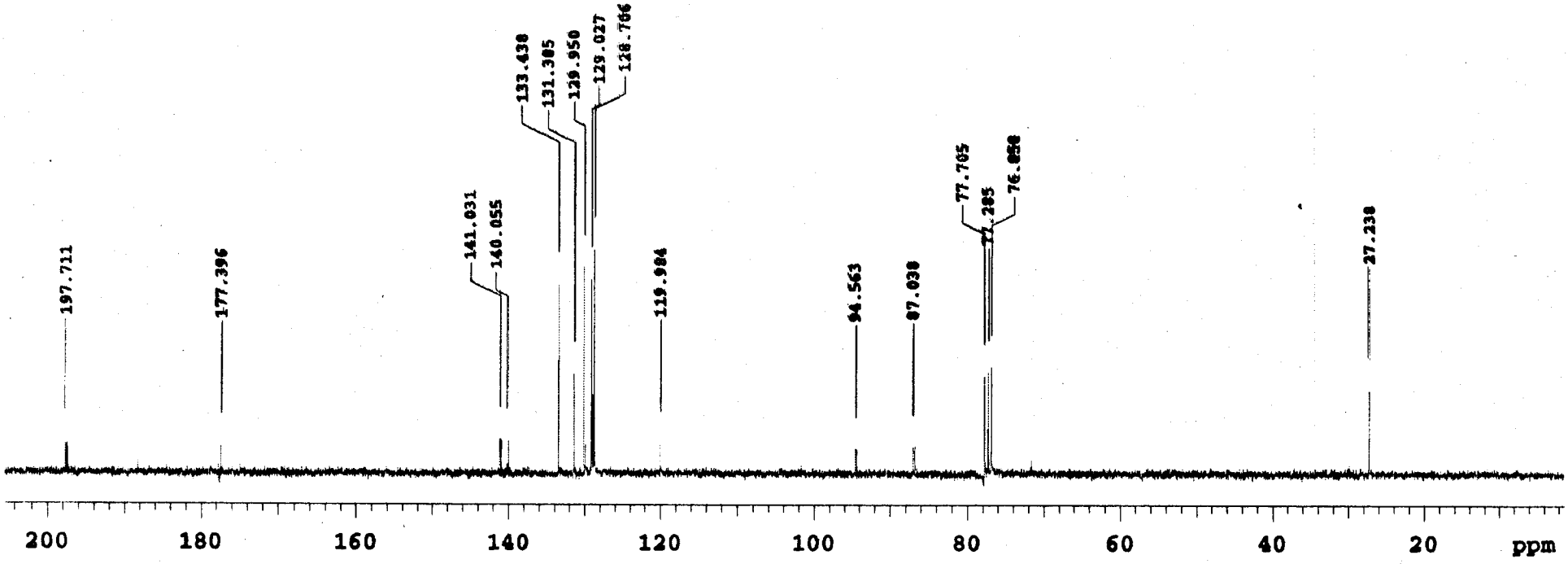
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140
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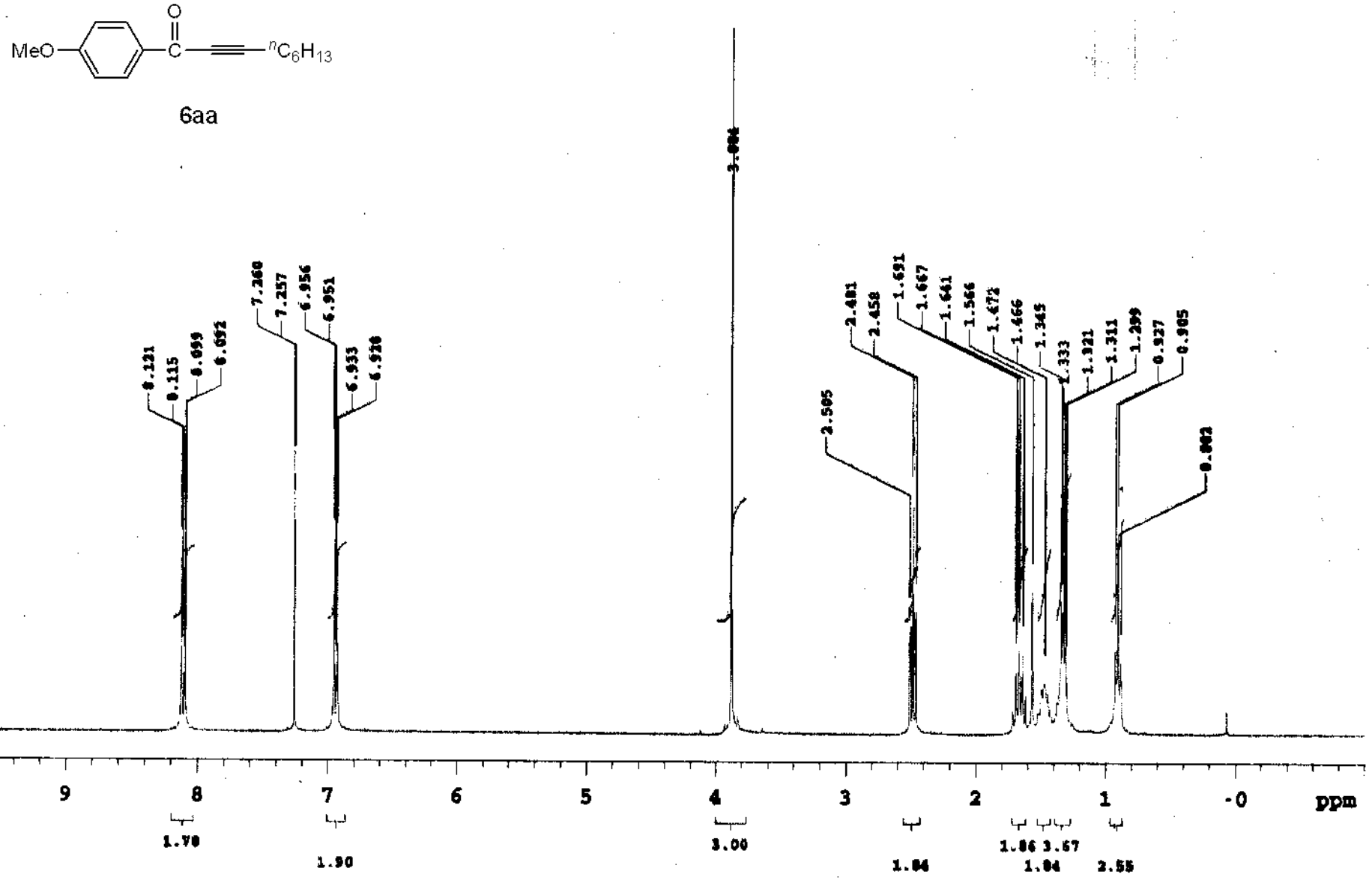
100
80
60
40
20
ppm


MeO
O
C
$^{n}C_6H_{13}$
6aa
8.121
8.115
8.099
8.092
7.260
7.257
6.956
6.951
6.933
6.926
3.884
2.481
2.458
2.585
1.691
1.667
1.641
1.566
1.472
1.466
1.345
1.333
1.321
1.311
1.299
0.927
0.905
0.862
9
8
7
6
5
4
3
2
1
-0
ppm
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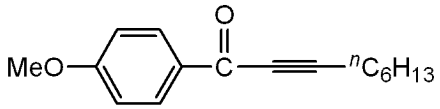
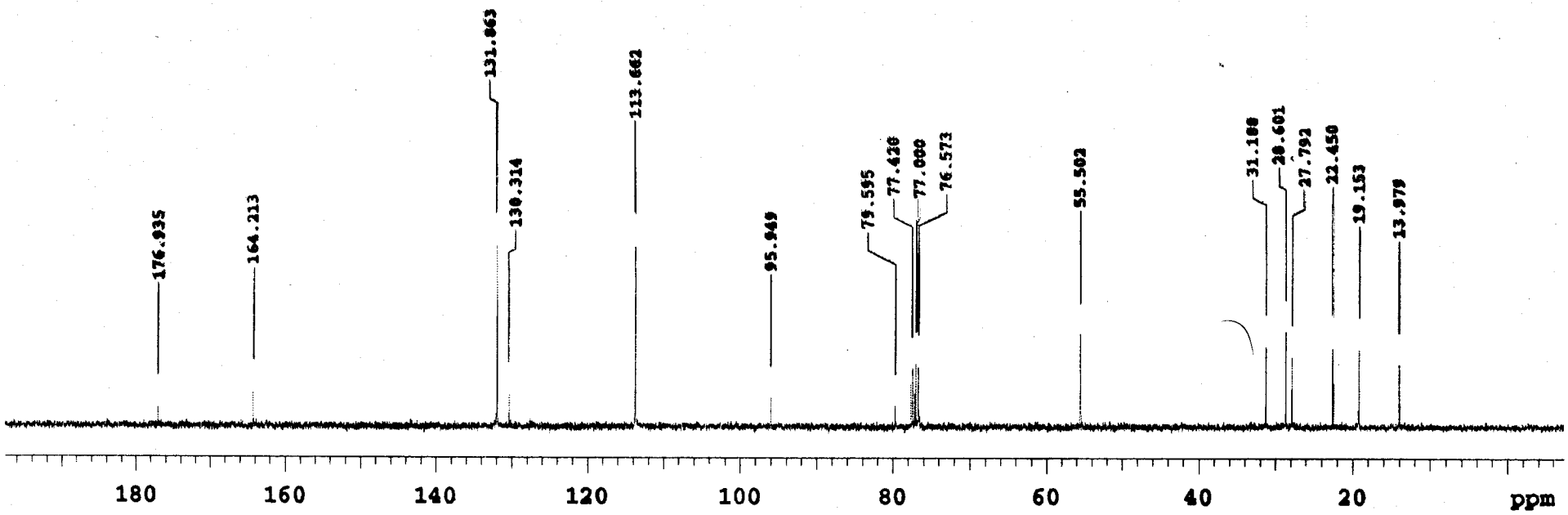
1.90
3.00
1.84
1.86
3.67
1.84
2.55


MeO
O
C
$^{n}C_6H_{13}$

6aa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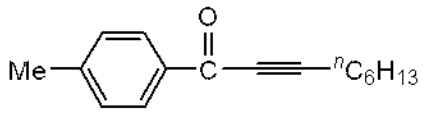
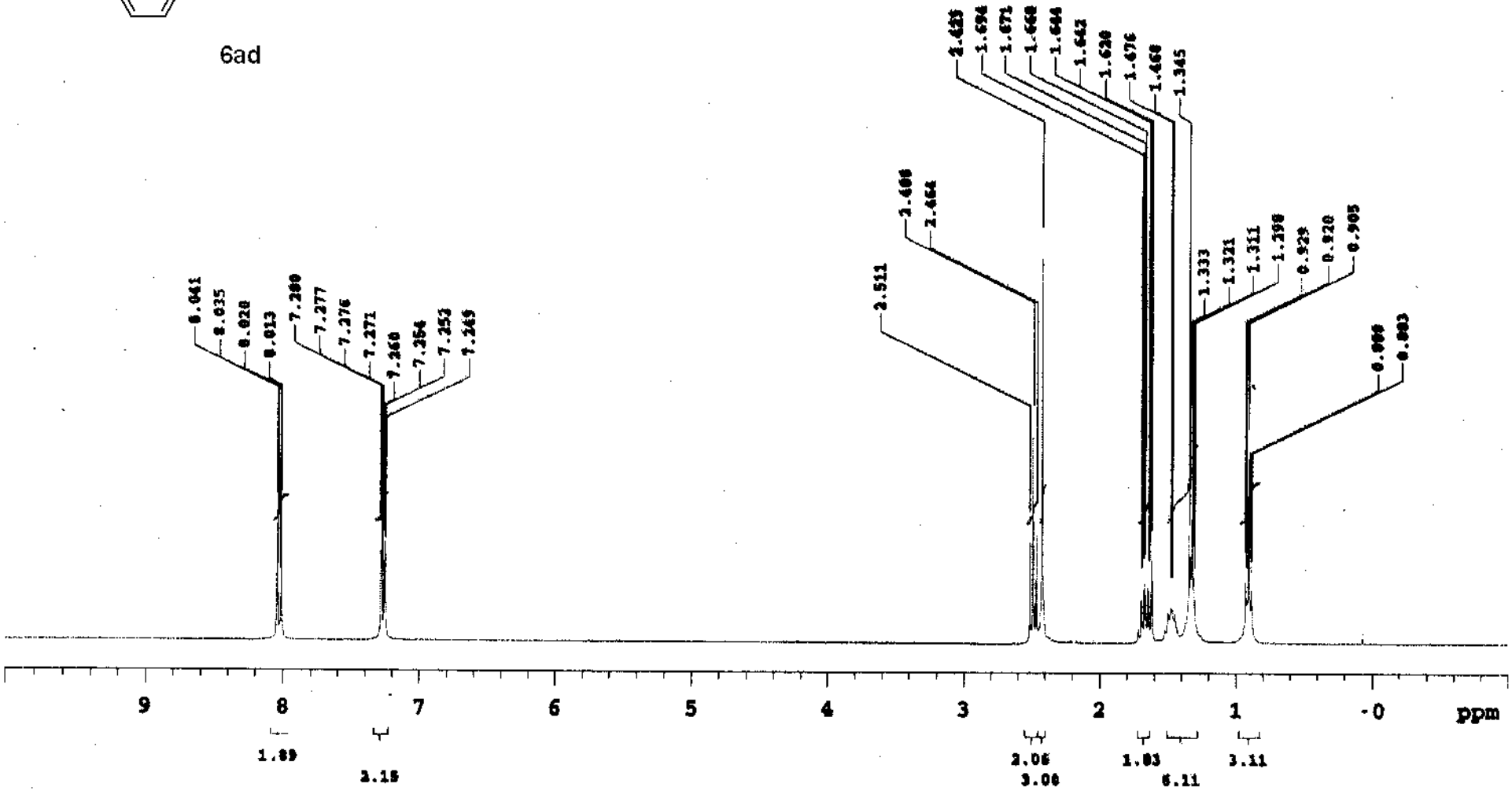
131.863
130.314
113.662
95.949
79.595
77.426
77.000
76.573
55.502
31.188
28.601
27.792
22.450
19.153
13.979
180
160
140
120
100
80
60
40
20
ppm


O
Me
C
$^{n}C_6H_{13}$

6ad


8.041
8.035
8.028
8.013
7.280
7.277
7.276
7.271
7.260
7.254
7.253
7.245
2.511
2.466
2.464
1.633
1.624
1.621
1.618
1.614
1.612
1.620
1.476
1.468
1.345
1.333
1.321
1.311
1.298
0.923
0.920
0.905
0.866
0.863
9
8
7
6
5
4
3
2
1
-0
ppm
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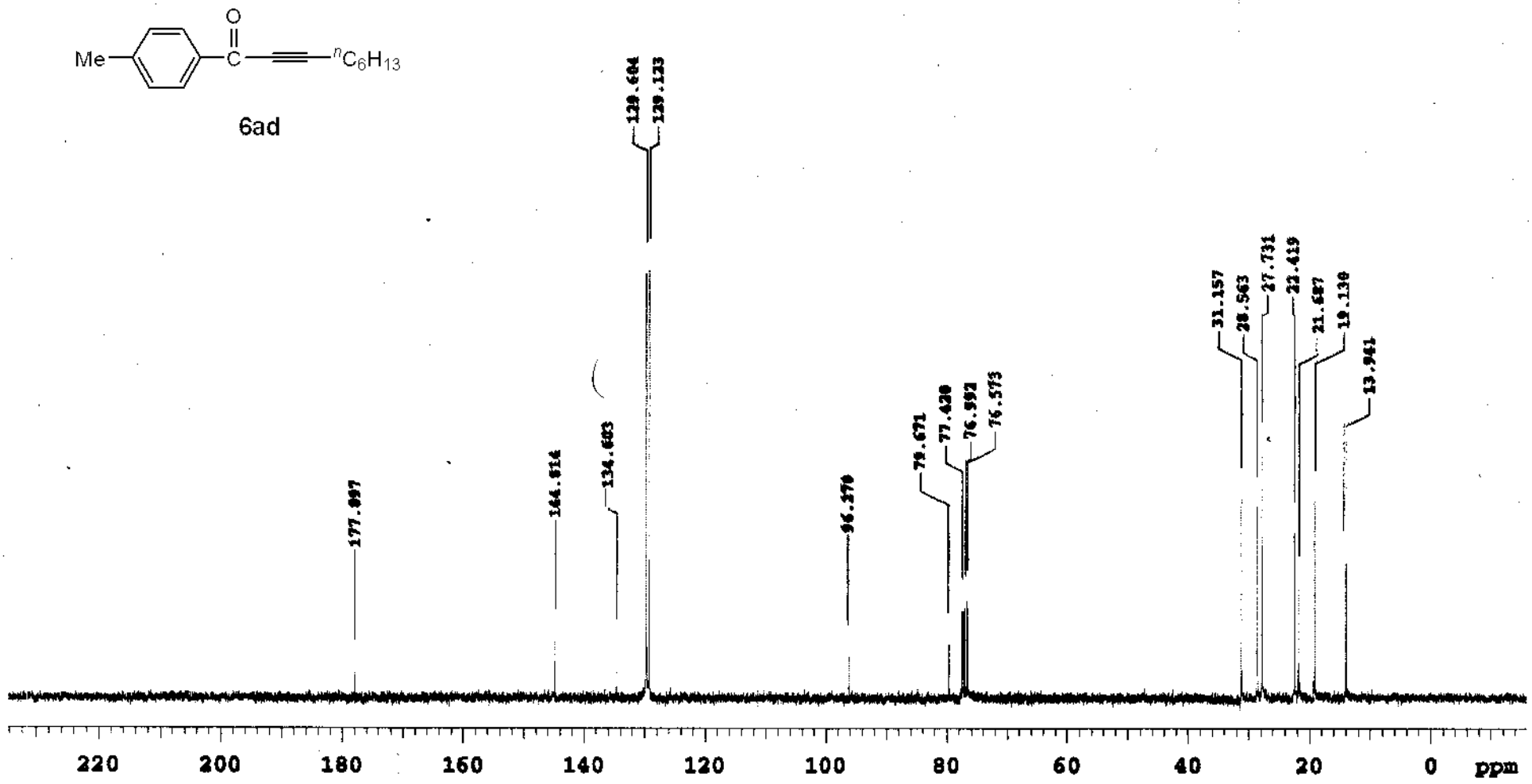
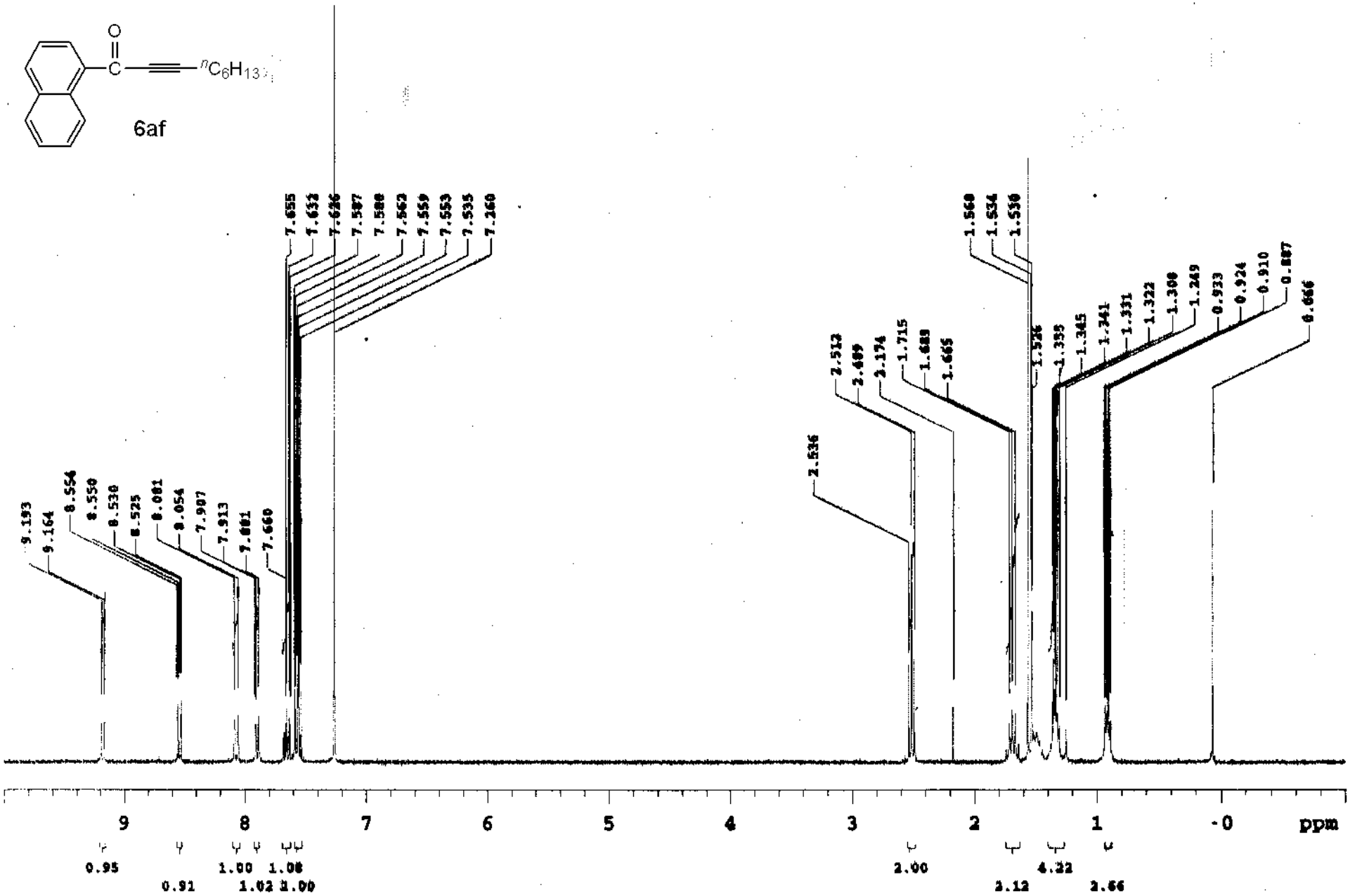
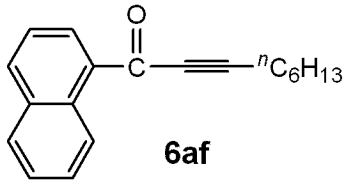
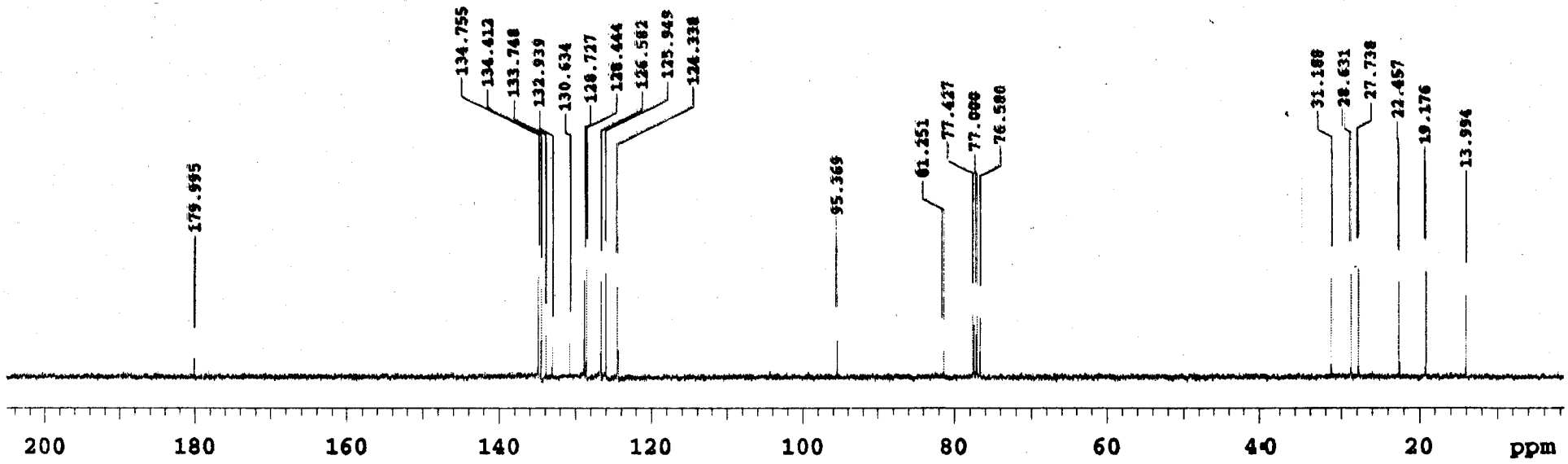
3.15
2.06
3.00
1.83
6.11
3.11

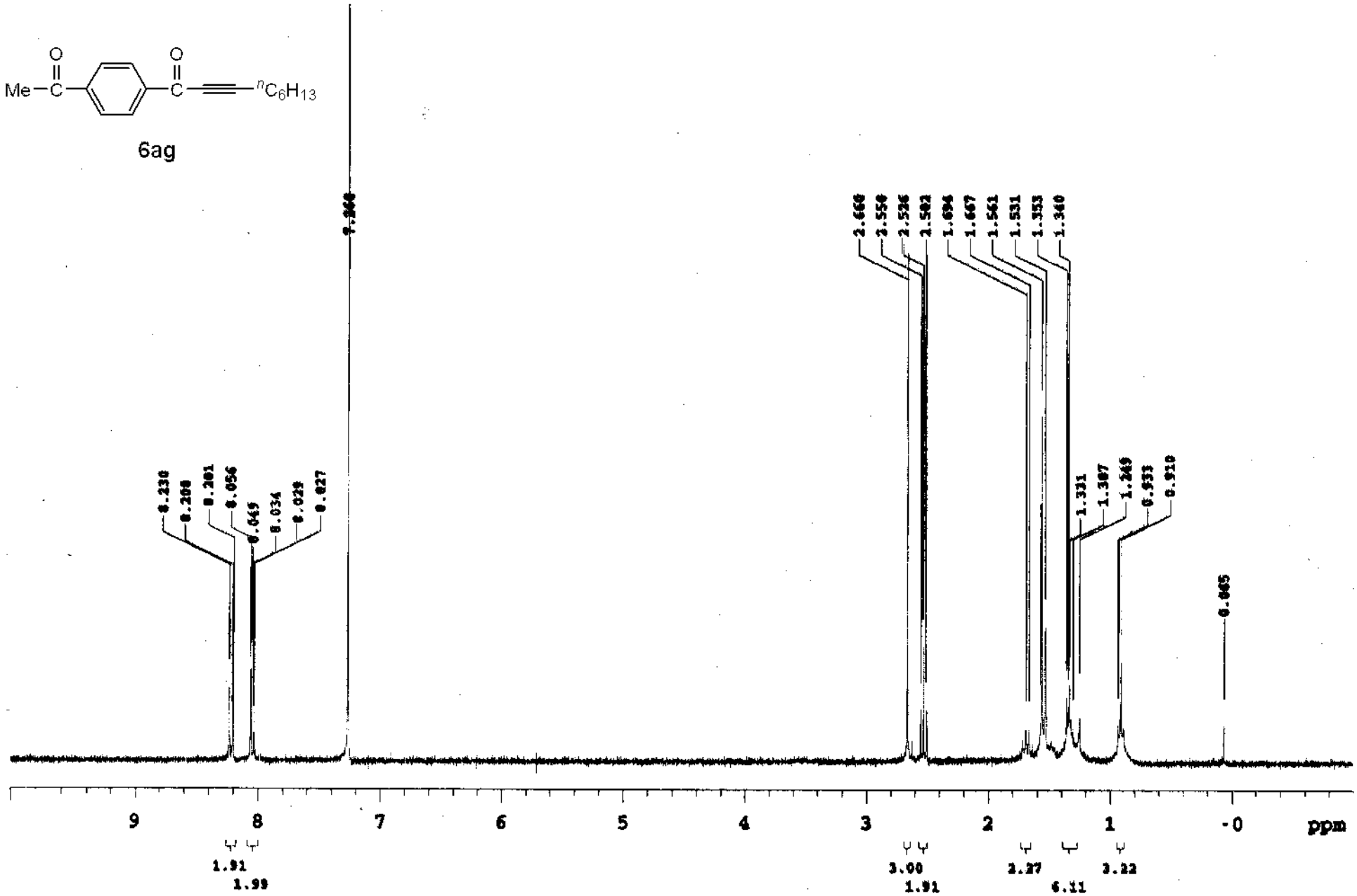

Me
O
C
$^{n}C_6H_{13}$
6ad
177.897
144.914
134.603
129.684
129.123
96.570
79.671
77.420
76.992
76.573
31.157
28.563
27.731
22.419
21.627
19.130
13.941
220
200
180
160
140
120
100
80
60
40
20
0
ppm

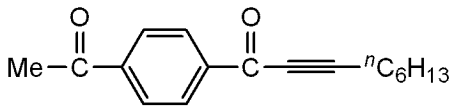

6af
9.193
9.164
8.554
8.550
8.530
8.525
8.081
8.054
7.987
7.913
7.881
7.660
7.655
7.632
7.626
7.587
7.588
7.563
7.559
7.553
7.535
7.360
2.536
2.513
2.489
2.174
1.715
1.688
1.665
1.568
1.534
1.536
1.526
1.339
1.345
1.341
1.331
1.322
1.308
1.249
0.933
0.924
0.910
0.887
0.666
9
8
7
6
5
4
3
2
1
0
ppm
0.95
0.91
1.00
1.02
1.08
1.00
2.00
3.12
4.22
3.66


O
C
$^{n}C_6H_{13}$

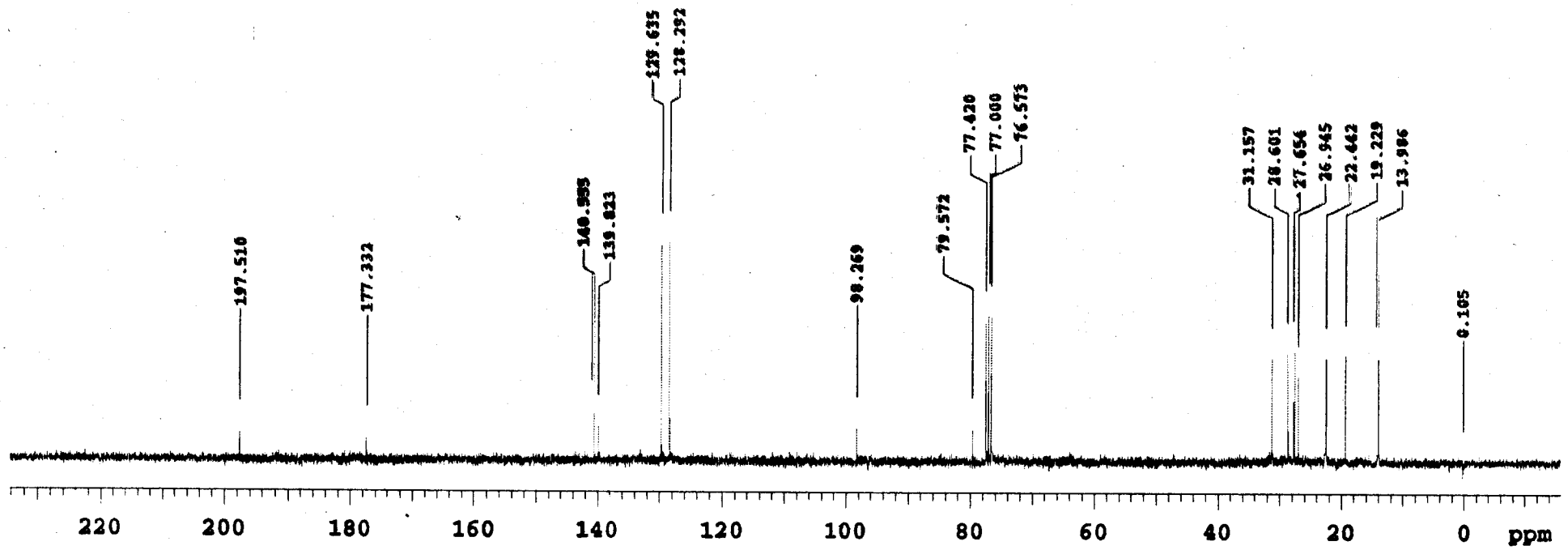
6af

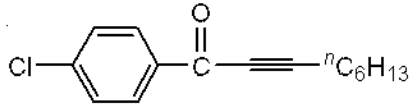

179.995
134.755
134.412
133.748
132.939
130.634
128.727
128.444
126.562
125.948
124.338
95.369
81.451
77.427
77.000
76.580
31.188
28.631
27.738
22.457
19.176
13.954
200
180
160
140
120
100
80
60
40
20
ppm


Me
O
O
$^{n}C_6H_{13}$
6ag
8.230
8.208
8.201
8.056
8.049
8.034
8.028
8.027
7.266
2.666
2.558
2.536
2.502
1.694
1.667
1.561
1.531
1.353
1.340
1.331
1.307
1.269
0.933
0.910
0.865
9
8
7
6
5
4
3
2
1
-0
ppm
1.91
1.99
3.00
1.91
2.27
6.11
3.22

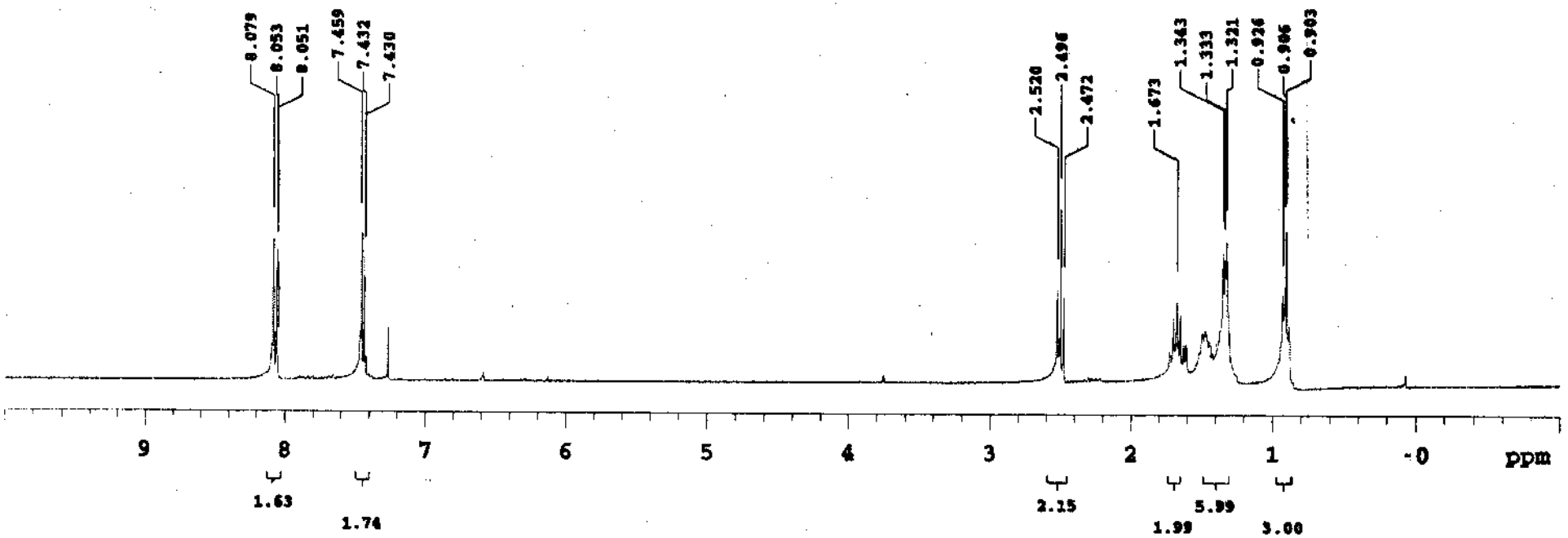

Me
O
C
O
C
$^{n}C_6H_{13}$

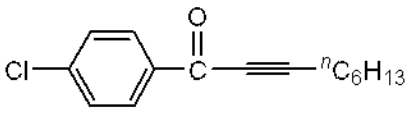
6ag


197.510
177.332
140.935
139.823
129.635
128.292
98.269
79.572
77.420
77.000
76.573
31.157
28.601
27.654
26.945
22.442
19.229
13.986
0.165
220
200
180
160
140
120
100
80
60
40
20
0
ppm

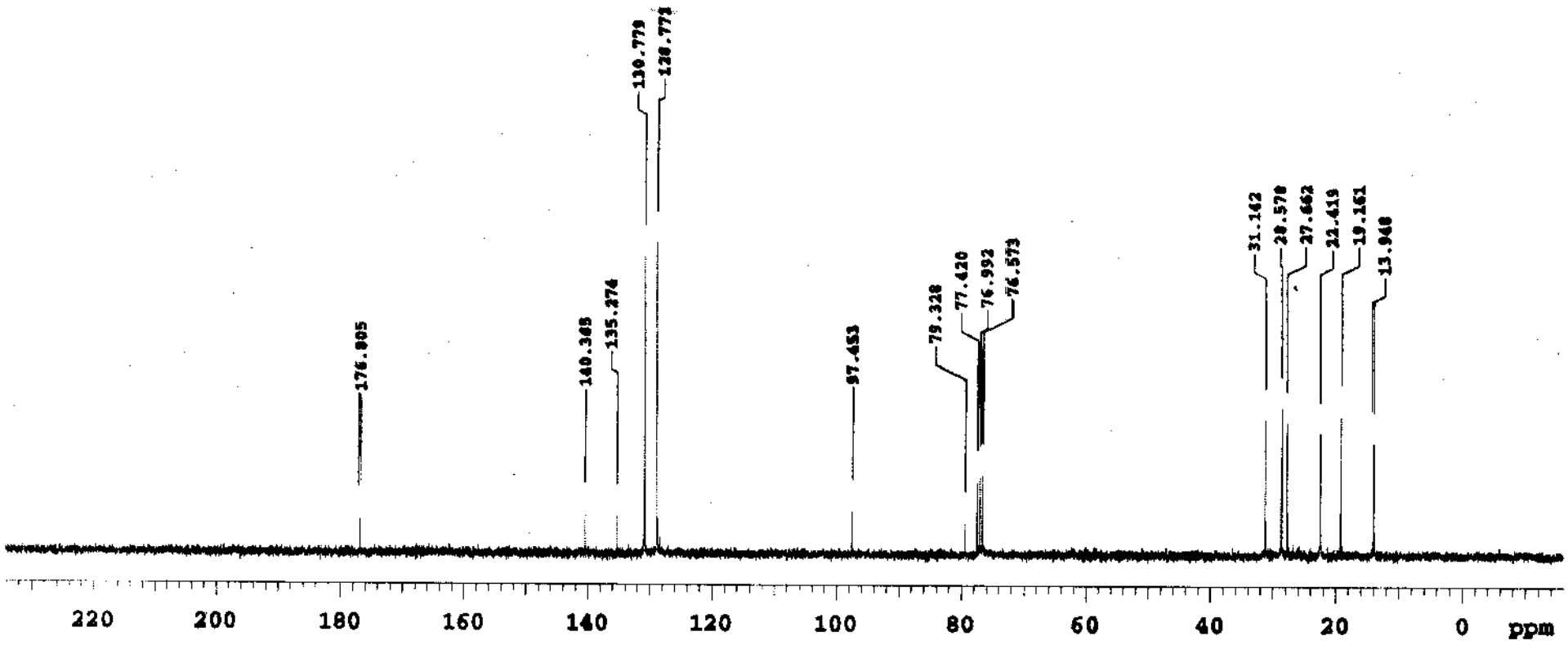

O
Cl
C
$^{n}C_6H_{13}$

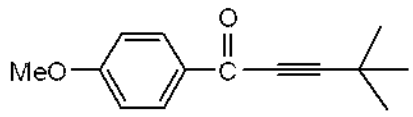
6ai


8.079
8.053
8.051
7.459
7.432
7.430
2.520
2.496
2.472
1.673
1.343
1.333
1.321
0.926
0.906
0.903
9
8
7
6
5
4
3
2
1
0
ppm
1.63
1.74
2.15
1.99
5.99
3.00


O
Cl
C
$^{n}C_6H_{13}$

6ai


176.805
140.365
135.374
130.779
128.771
97.453
79.328
77.420
76.992
76.573
31.142
28.578
27.662
22.419
19.161
13.948
220
200
180
160
140
120
100
80
60
40
20
0
ppm


MeO
O
C

6ba

8.103
8.076
7.268
7.257
6.960
6.931
3.883
1.576
1.574
9
8
7
6
5
4
3
2
1
-0
ppm
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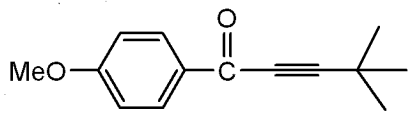
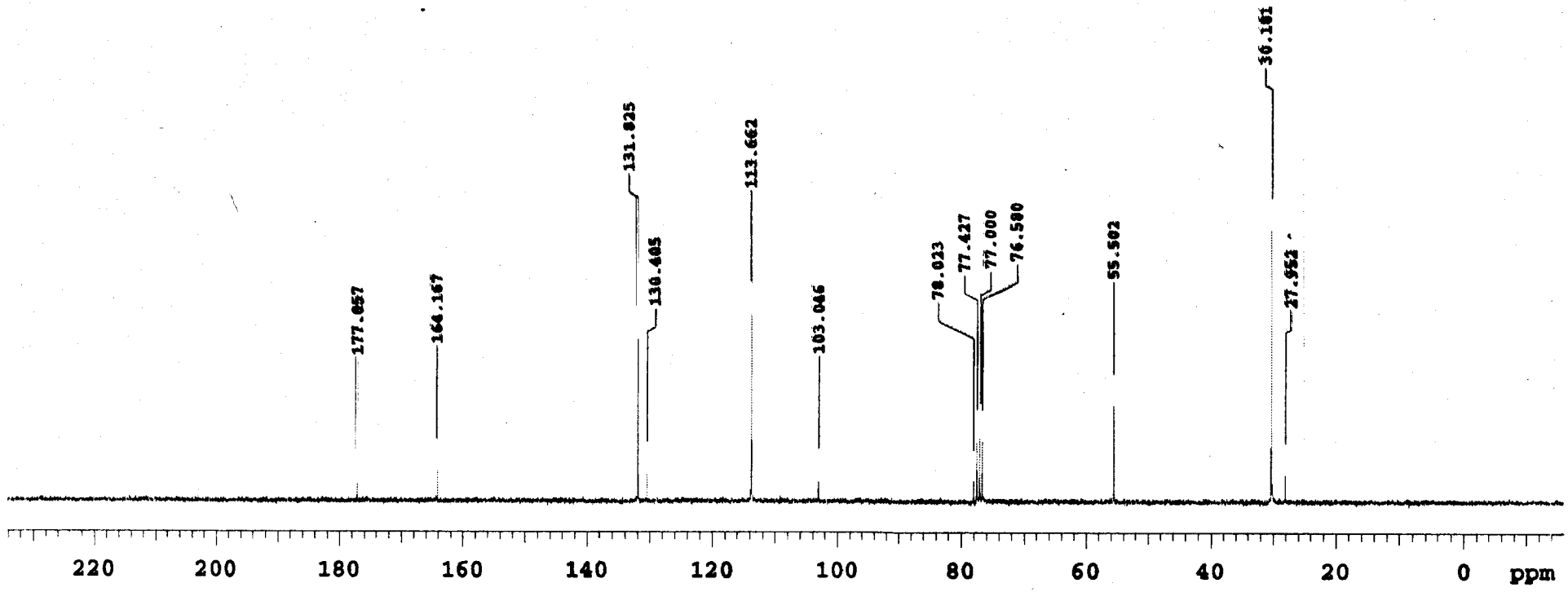
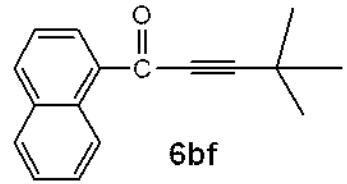
1.00
2.78
9.00


MeO
O
C

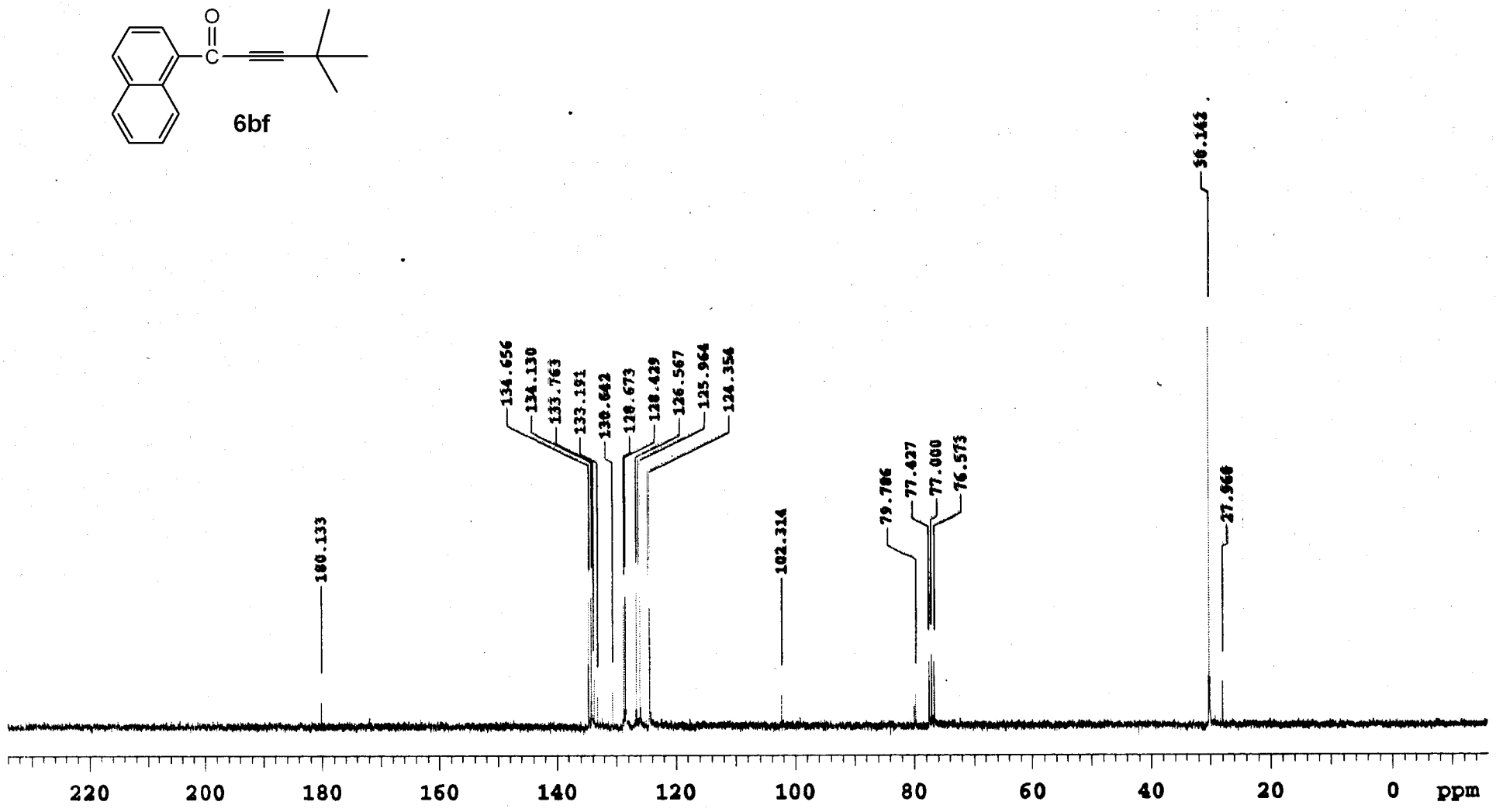
6ba

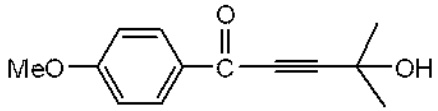

177.057
164.167
131.825
130.405
113.662
103.046
78.023
77.427
77.000
76.580
55.502
36.161
27.954
220
200
180
160
140
120
100
80
60
40
20
0
ppm



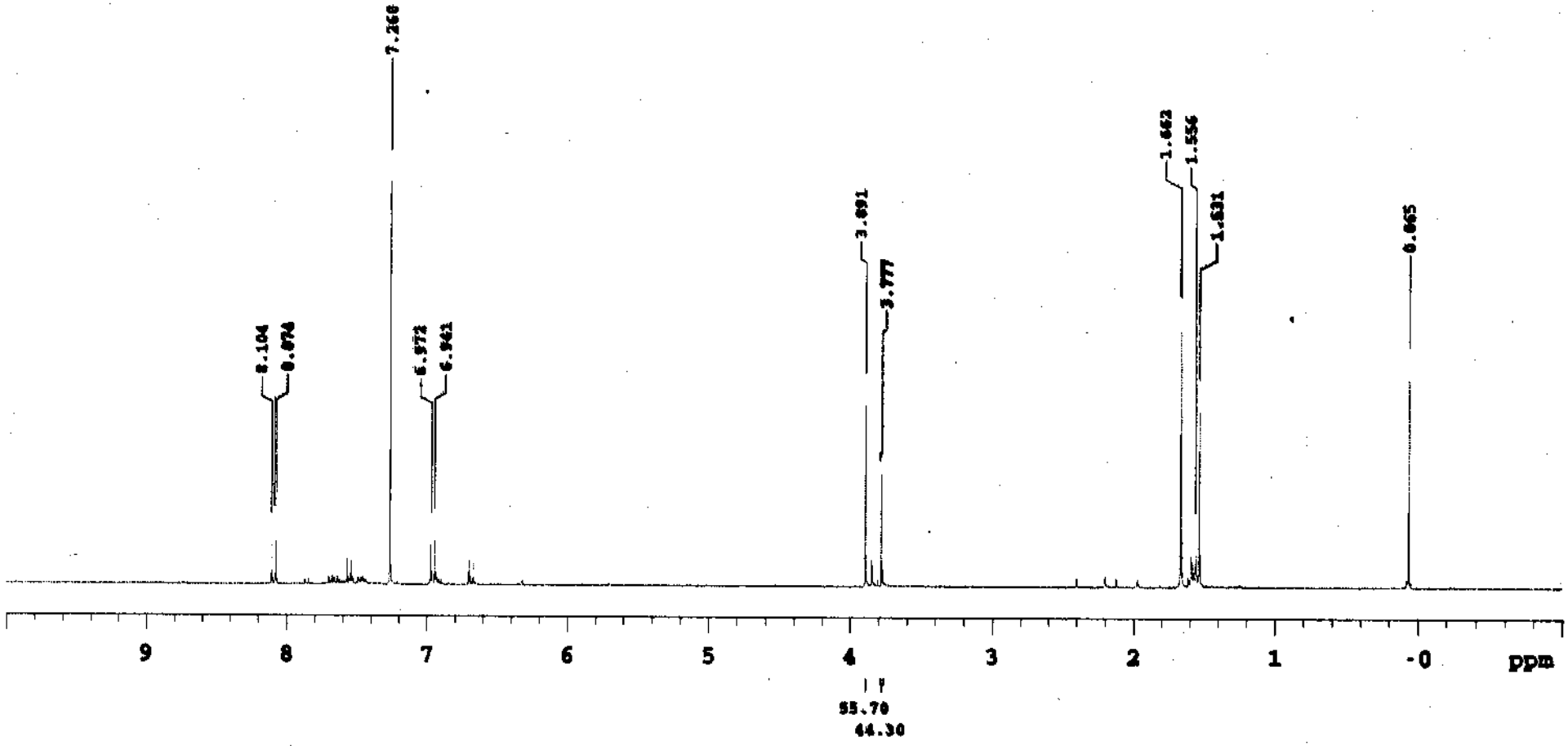
6bf

9.178
9.165
8.509
8.505
8.485
8.480
8.074
8.047
7.904
7.880
7.876
7.651
7.627
7.585
7.579
7.574
7.562
7.558
7.553
7.549
7.537
7.260
1.398
1.440
9
8
7
6
5
4
3
2
1
0
ppm
0.97
0.98
1.07
1.11
1.21
1.10
9.00


6bf
180.133
134.656
134.130
133.763
133.191
130.642
128.673
128.429
126.567
125.964
124.354
102.314
79.786
77.427
77.000
76.573
30.143
27.966
220
200
180
160
140
120
100
80
60
40
20
0
ppm


MeO
O
C
OH

6ca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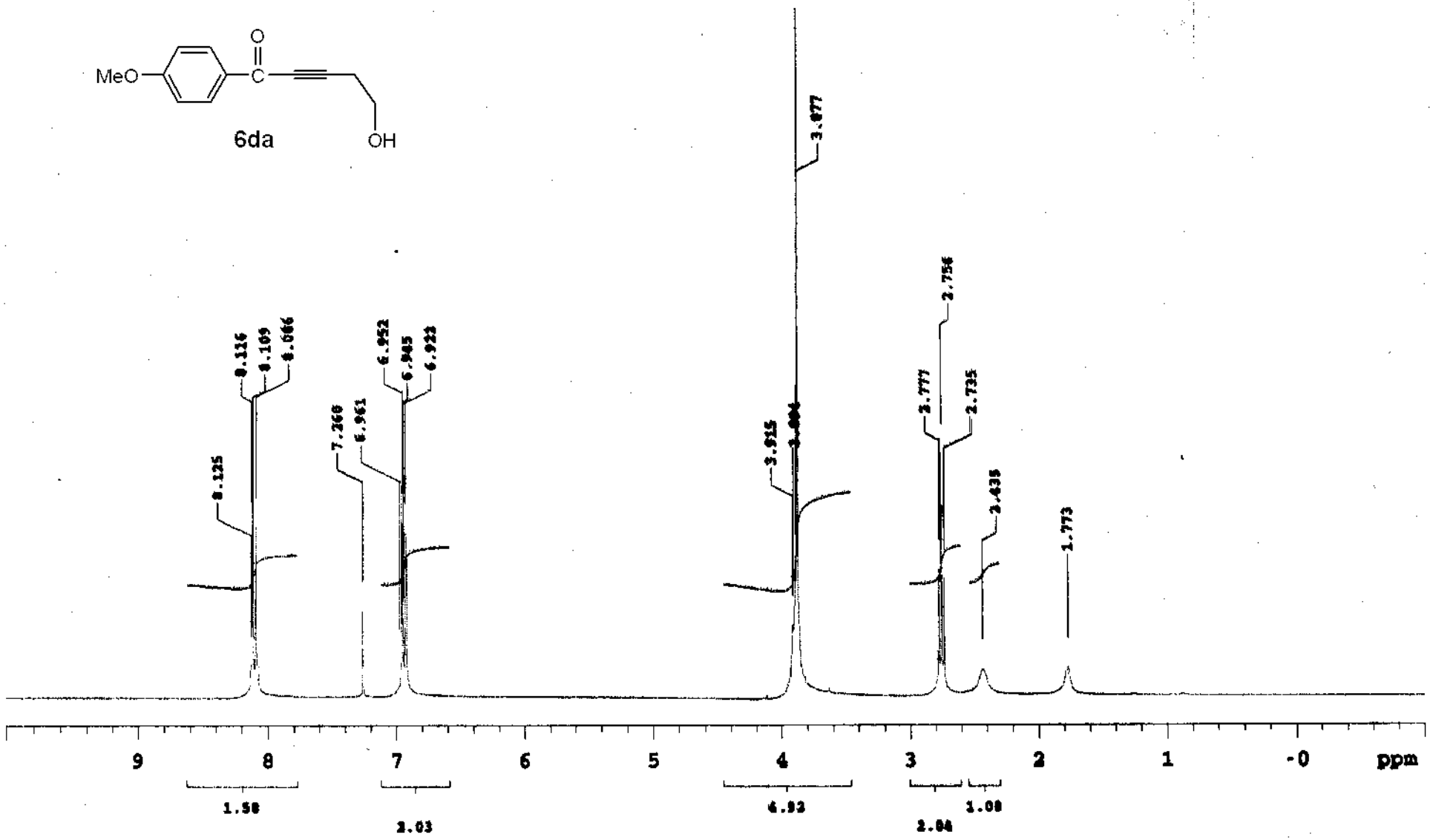
8.076
7.260
6.972
6.941
3.891
3.777
1.662
1.556
1.531
0.865
9
8
7
6
5
4
3
2
1
-0
ppm
55.70
44.30


MeO
O
OH
6da
8.125
8.116
8.109
8.066
7.266
6.961
6.952
6.945
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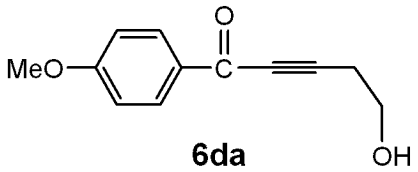
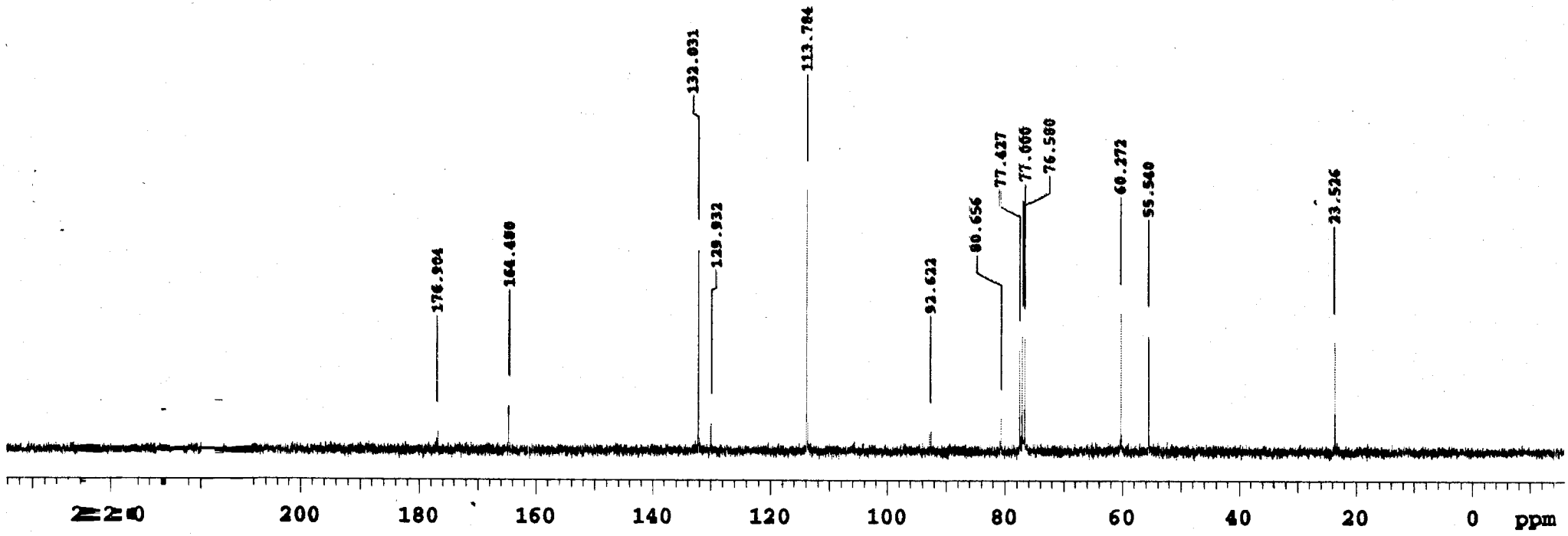
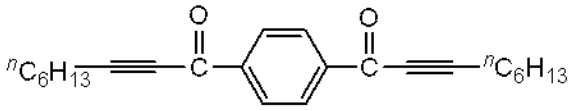
3.915
3.877
3.777
2.756
2.735
2.435
1.773
9
8
7
6
5
4
3
2
1
-0
ppm
1.58
2.03
4.93
2.04
1.08

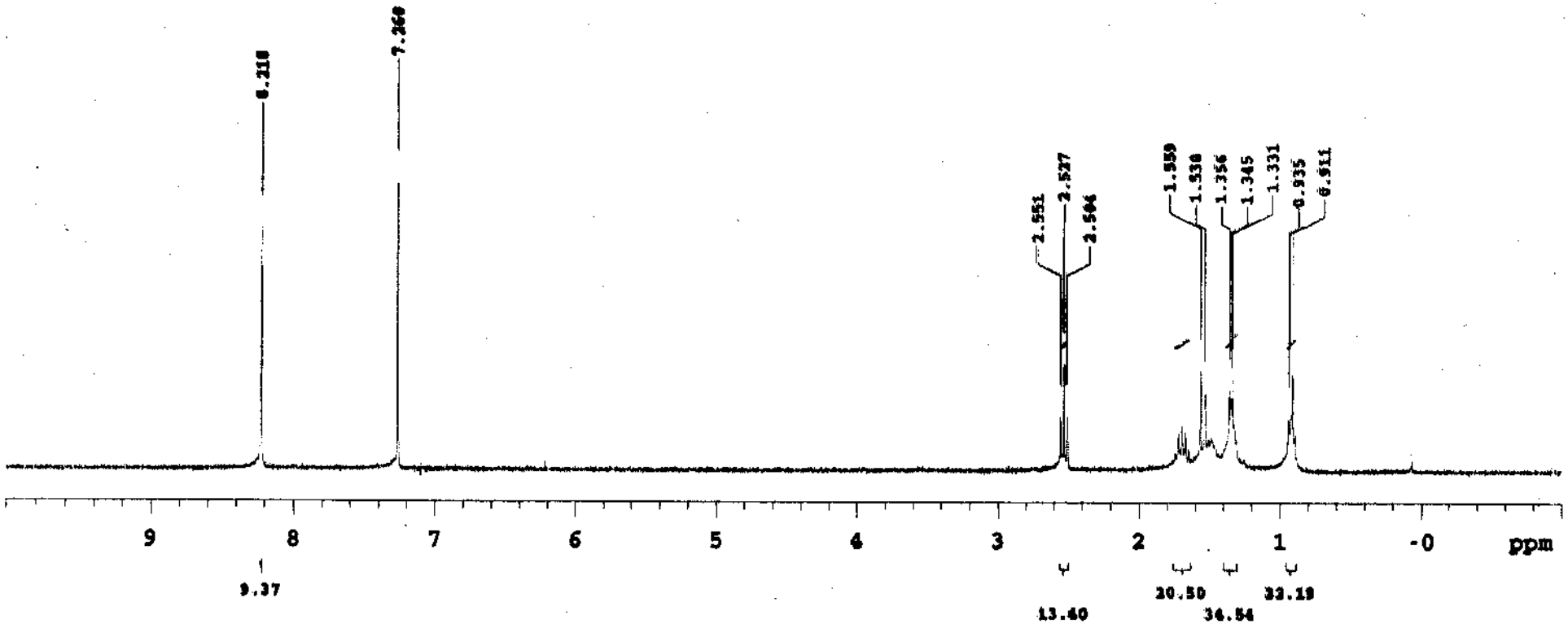

MeO
O
C
OH

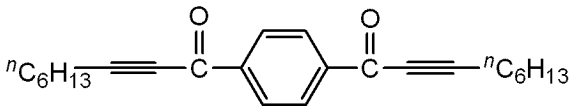
6da


176.904
164.480
133.031
129.932
113.784
92.622
80.656
77.427
77.066
76.580
60.272
55.540
23.526
200
180
160
140
120
100
80
60
40
20
0
ppm

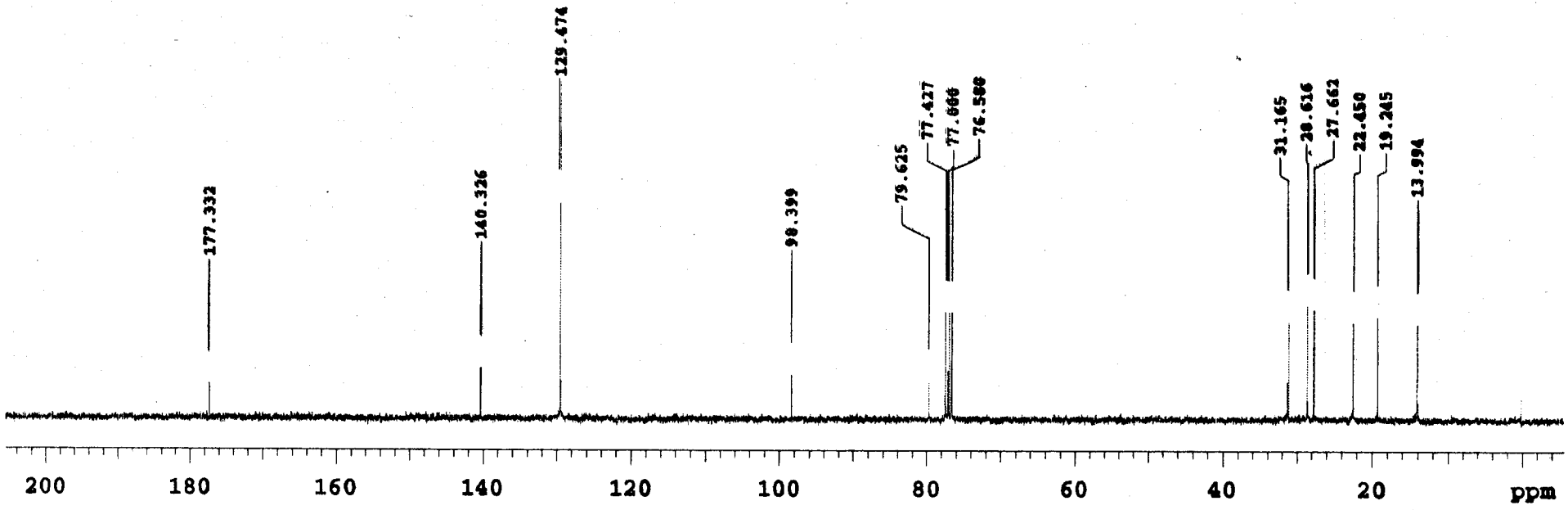

O
O
$^{n}C_6H_{13}$
C
C
$^{n}C_6H_{13}$

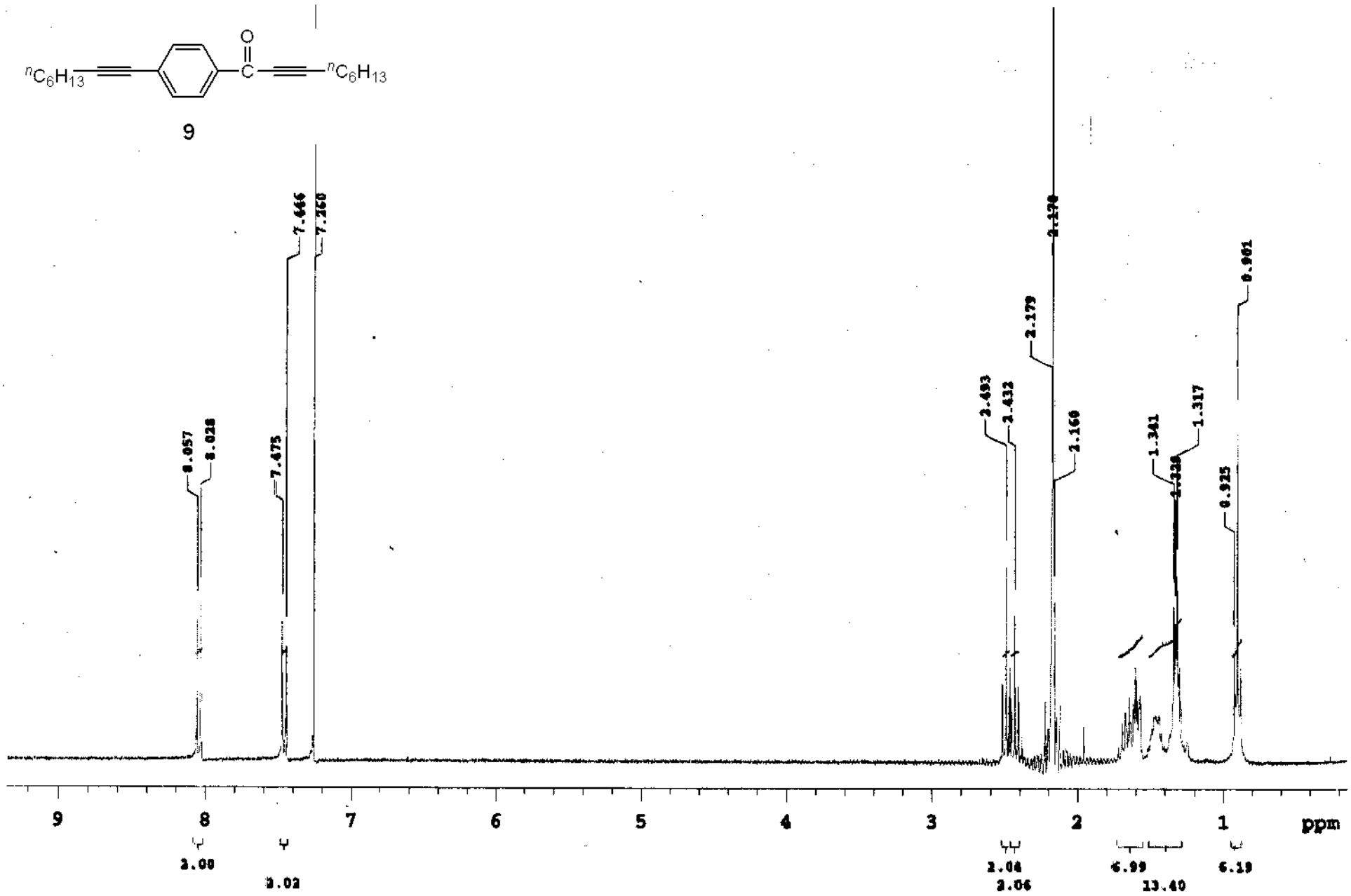
8

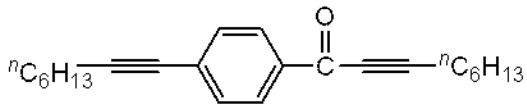

8.218
7.268
2.551
2.527
2.504
1.559
1.538
1.356
1.345
1.331
0.935
0.911
9
8
7
6
5
4
3
2
1
-0
ppm
1
9.37
13.40
20.50
34.54
32.19


O
O
$^{n}C_6H_{13}$
C
C
$^{n}C_6H_{13}$

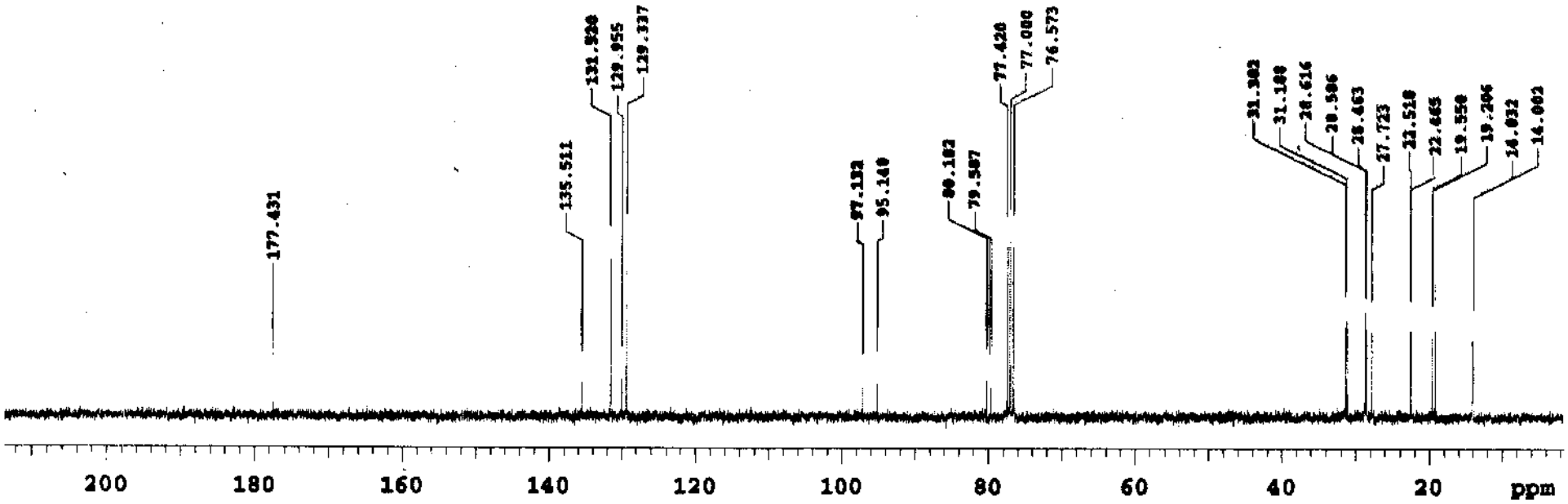
8


177.332
140.326
129.474
98.399
79.625
77.427
77.000
76.580
31.165
28.616
27.662
22.450
19.245
13.994
200
180
160
140
120
100
80
60
40
20
ppm


$^{n}C_6H_{13}$
O
C
$^{n}C_6H_{13}$
9
8.057
8.028
7.475
7.446
7.260
2.493
2.432
2.179
2.160
1.341
1.317
0.925
0.901
9
8
7
6
5
4
3
2
1
ppm
2.00
2.02
2.04
2.06
6.99
13.40
6.19


O
C
$^{n}C_6H_{13}$
$^{n}C_6H_{13}$

9


177.431
135.511
131.926
129.955
129.337
97.132
95.140
80.182
79.587
77.426
77.000
76.573
31.303
31.188
28.616
28.586
28.463
27.723
22.518
22.465
19.558
19.206
14.032
14.002
200
180
160
140
120
100
80
60
40
20
ppm