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ELECTRONIC SUPPLEMENTARY INFORMATION

One-pot Process for Palladium Catalyzed Direct C-H Acylation of Anilines in Water Using a Removable Ortho Directing Group

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Table of contents

General 2
Characterization of products 3
NMR spectras 17
GC-MS chromatograms 73

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General

Unless otherwise indicated, all starting materials were obtained from commercial suppliers, and were used without further purification. Analytical thin-layer chromatography (TLC) was performed on Merck DC pre coated TLC plates with 0.25 mm Kieselgel 60 F254. Visualizationwas performed with a 254 nm UV lamp. The ^{1}H and ^{13}C NMR spectra were recorded on a 250 spectrometer in $CDCl_3$. Chemical shifts are expressed in parts per million (δ) using residual solvent protons as internal standards (δ 7.26 for ^{1}H, δ 77.0 for ^{13}C). Coupling constants (*J*) are reported in Hertz (Hz). Splitting patterns are designated as s (singlet), d (doublet), t (triplet), m (multiplet). Combination gas chromatography and low resolution mass spectrometry was obtained on Gas Chromatograph (30 m x 0.25 mm column with 0.25 μm HP-5MS coating, He carrier gas) and Mass Spectrometer (Ion source: EI+, 70eV, 230°C; interface: 300°C). IR spectra were obtained on a spectrometer on a single-reflection diamond ATR unit. All melting points were measured on an appropriate apparatus and are uncorrected. High-resolution mass spectra were acquired on time-of-flight mass spectrometer equipped with a Jet Stream electrospray ion source in positive ion mode. Injections of 0.1-0.3 μl were directed to the mass spectrometer at a flow rate 0.5 ml/min (70% acetonitrile-water mixture, 0.1 % formic acid), using HPLC system. Jet Stream parameters: drying gas (N_2) flow and temperature: 10.0 l/min and 325 °C, respectively; nebulizer gas (N_2) pressure: 10 psi; capillary voltage: 4000V; sheath gas flow and temperature: 325 °C and 7.5 l/min; TOFMS parameters: fragmentor voltage: 120 V; skimmer potential: 120V; OCT 1 RF Vpp:750 V. Full-scan mass spectra were acquired over the m/z range 100-2500 at an acquisition rate of 250 ms/spectrum and processed by software.

Caution! TBHP is a dangerous, highly reactive, flammable and toxic chemical. Reactions should be performed with special care.

General procedure for the synthesis of N-(2-benzoylphenyl)acetamides 4.

A round bottom flask was charged with 14.4 mg (0.05 mmol, 5 mol%, method A) or 21 mg (0.075 mmol, 7.5 mol%, method B) SDS, 4 mL water and 1 mmol (1 eq.) aniline. 141.7 μL acetic anhydride was added dropwise during stirring at RT. The reaction was monitored by TLC. After completition (0.5 – 1 hours), 11.2 mg (0.05 mmol, 5 mol%, method A) or 17 mg (0.075 mmol, 7.5 mol%, method B) $Pd(OAc)_2$, 20 μL (0.26 mmol, 26 mol%, method A) or 30 μL (0.39 mmol, 39 mol%, method B) TFA, 2 mmol (2 eq.) aldehyde and 250 μL (2 mmol, 2 eq., 70 wt% in water) TBHP was added during vigorous stirring. After 24 hours, the heterogeneous mixture was neutralized with $NaHCO_3$, extracted with EtOAc, and the combined organic layers were washed with water. The EtOAc solution was dried over $MgSO_4$, concentrated under reduced pressure, and the crude product was purified by silica gel column chromatography (hexane:EtOAc).

General procedure for the synthesis of (2-aminophenyl)(phenyl)methanones 5.

Following the general procedure for the synthesis of **4** described above 3.5 mL HCl (37 wt% in water) was added, and the reaction mixture was stirred at 125 °C for 3 hours. The reaction mixture was cooled to RT, and 4 mL EtOAc was added. The mixture was neutralized with $NaHCO_3$ during stirring. Than the layers were separated, the water phase was extracted with EtOAc, and the summarized organic phase was washed with water, and dryed over $MgSO_4$. The crude product was purified by silica gel column chromatography (hexane/EtOAc).

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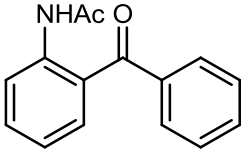
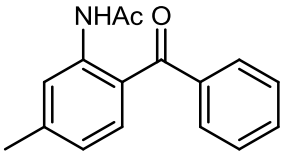
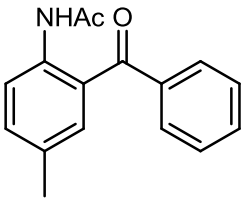
Characterization of products



N-(2-benzoylphenyl)acetamide[1] (**4a)** Method A. White, solid; 147 mg (0.62 mmol, yield: 68%). m.p.: 73–82 °C. R_f : 0.68 (hexane:EtOAc = 1:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 10.80 (s, 1H), 8.61 (d, 1H, *J* = 8.37), 7.70-7.44 (m, 7H), 7.06 (t , 1H, *J* = 7.42 Hz), 2.21 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 199.6, 169.1, 140.4, 138.5, 134.1, 133.4, 132.4, 129.8, 128.2, 123.1, 121.9, 121.4, 25.2 ppm. IR (ATR): ν_{max} = 3212.6, 3163.4, 3027.3, 1673.9, 1600.6, 1477.0, 1287.1, 920.3, 750.4, 701.8, 634.5 cm^{-1}. MS (EI, 70 eV) *m/z* (%): 239 (16 [M+]), 196 (100), 167 (15), 134 (13), 120 (24), 105 (10), 92 (12), 77 (24).



N-(2-benzoyl-5-methylphenyl)acetamide[1] **(4b)** Method A. White, solid; 193 mg (0.76 mmol, yield: 76%). m.p. 125-129°C Rf: 0.59 (in Hexane:EtOAc = 1:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 11.00 (s, 1H), 8.47 (s, 1H), 7.66-7.41 (m, 6H), 6.85 (d, 1H *J* = 8.69 Hz), 2.40 (s, 3H), 2.20 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 199.4, 169.1, 145.7, 140.6, 138.8, 133.8, 132.1, 129.6, 128.1, 122.7, 121.5, 120.3 25.2, 22.0 ppm. IR (ATR): ν_{max} = 3177, 2962, 1668, 1608, 1280, 1260, 1016, 794, 742, 698, 617 cm^{-1} MS (EI, 70 eV): *m/z* (%): 253 (41, [M$^+$]), 210 (100), 180 (16), 148 (20), 134 (30), 77 (28).



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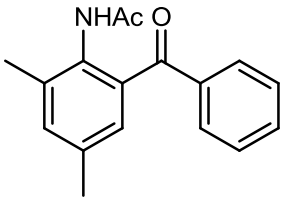
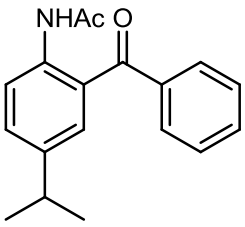
N-(2-benzoyl-4-methylphenyl)acetamide[1] **(4c)** Method A. White, solid; 174 mg (0.69 mmol, yield: 69%). m.p.: 158 – 163 °C. R_f: 0.51 (in Hexane:EtOAc = 1:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 10.62 (s, 1H), 8.47 (d, 1H, *J* = 8.37 Hz), 7.70 - 7.32 (m, 7H), 2.27 (s, 3H), 2.18 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 199.6, 168.9, 138.6, 137.8, 134.8, 133.4, 132.4, 131.6, 129.8, 128.2, 123.4, 121.5, 25.1, 20.6 ppm. IR (ATR) ν_{max} = 3207, 3162, 3026, 1669, 1487, 1291, 825, 742, 702 cm^{-1}; MS (EI, 70 eV) *m/z* (%): 253 (25, [M^+]), 210 (100), 180 (85), 134 (19), 106 (10), 77 (24).



N-(2-benzoyl-4,6-dimethylphenyl)acetamide[1] **(4d)** Method B. Yellow, solid; 133 mg (0.5 mmol, yield: 50 %). m.p.: 125–141°C. R_f : 0.25 (Hexane:EtOAc = 1:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 8.50 (s, 1H), 7.81 (d, 2H, *J* = 7.11 Hz), 7.58 (t, 1H, *J* = 7.27 Hz), 7.44 (t, 2H, *J* = 7.50 Hz), 7.18 (s, 1H), 7.03 (s, 1H), 2.29 (s, 3H), 2.23 (s, 3H), 1.98 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 197.9, 168.7, 137.3, 135.9, 135.2, 134.7, 133.7, 133.0, 132.0, 130.3, 128.4, 128.2, 23.3, 20.8, 18.6 ppm. IR ν = 3222, 3015, 2922, 1665, 1649, 1528, 1298, 1216.2, 710.2 cm^{-1}; MS (EI, 70 eV) *m/z* (%): 267 (17, [M+]), 224 (100), 208 (10), 180 (13), 162 (24), 148 (18), 120 (14), 105 (16), 91 (11), 77 (40).



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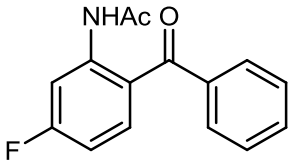
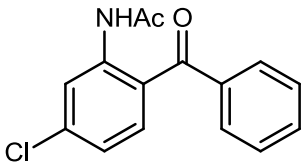
N-(2-benzoyl-4-isopropylphenyl)acetamide[1] **(4e)** Method A. White, solid; 154 mg (0.55 mmol, yield: 55%). m.p. 83-88 °C. Rf: 0.43 (in Hexane:EtOAc = 1:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 10.62 (s, 1H), 8.49 (d, 1H *J* = 8.53 Hz), 7.72 – 7.37 (m, 7H), 2.85 (septet, 1H *J* = 6.79 Hz), 2.20 (s, 3H), 1.19 (d, 6H *J* = 6. 95 Hz) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 199.6, 169.0, 142.7, 138.6, 138.1, 132.5, 132.2, 131.2, 129.9, 128.3, 123.5, 121.7, 33.3, 25.1, 23.8 ppm. IR (ATR): ν_{max} = 3188, 3166, 2963, 1666, 1594, 1492, 1370, 1286, 1274, 827, 698 cm^{-1} MS (EI, 70 eV): *m/z* (%): 281 (42, [M^{+}]), 239 (40), 224 (100), 105 (31), 77 (28).



***N*-(2-benzoyl-5-fluoro-phenyl)acetamide**[1] **(4f)** Method B. Offwhite, oil. 138 mg (0.54 mmol, yield: 54%). R_f : 0.72 (hexane:EtOAc = 1:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 11.20 (s, 1H), 8.48 (dd, 1H, J_1=11.85, J_2=2.53), 7.64 - 7.43 (m, 6H), 6.77-6.69 (m, 1H), 2.20 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 198.7, 169.3, 165.6 (d, J_{CF} = 254.6 Hz), 143.3 (d, J_{CF} = 13.3 Hz), 138.6, 136.2, 136.1, 132.3, 129.4, 128.3, 118.8 (d, J_{CF} = 2.8 Hz), 109.0 (d, J_{CF} = 22.1 Hz), 108.2 (d, J_{CF} = 28.0 Hz), 25.2 ppm. IR (ATR): ν_{max} = 3270, 2926, 2854, 1591, 1521, 1425, 1255, 1235, 695 cm^{-1}. MS (EI, 70 eV) *m/z* (%): 257 (18, [M+]), 214 (100), 198 (7), 185 (15), 152 (15), 138 (49), 110 (15), 105 (15), 83 (8), 77 (29).



N-(2-benzoyl-5-chlorophenyl)acetamide[1] **(4g)** Method B. Offwhite, solid; 115 mg (0.42 mmol, yield: 42%). m.p. 108-118°C (lit.: 70.5-75°C). Rf: 0.57 (in Hexane:EtOAc =1:2). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 10.96 (s, 1H), 8.74 (s, 1H), 7.66-7.57 (m, 3H), 7.51-7.45 (m,

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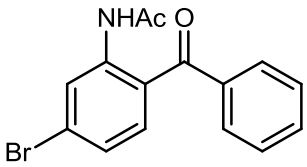
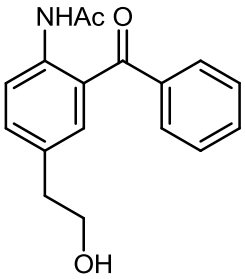
3H), 7.02 (dd, 1H, J_1 = 8.53 Hz J_2 = 2.05 Hz), 2.21 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 198.9, 169.2, 141.5, 140.6, 138.3, 134.6, 132.6, 129.6, 128.4, 122.1, 121.1, 120.9, 25.2 ppm. IR (ATR): ν_{max} = 3200, 2919, 1669, 1595, 1470, 1271, 926, 749, 697, 613 cm^{-1} MS (EI, 70 eV): *m/z* (%): 273 (16, [M+], 230 (100), 168 (30), 154 (35), 105 (32), 77 (60).

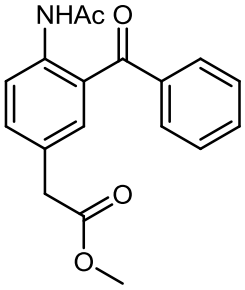


N-(2-benzoyl-5-bromophenyl)acetamide[1] **(4h)** Method B. Offwhite, solid; 127 mg (0.40 mmol, yield: 40%). m.p. 132-137 °C Rf: 0.51 (in Hexane:EtOAc = 1:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 10.90 (s, 1H), 8.88 (d, 1H, *J* = 1.74 Hz), 7.66-7.57 (m, 3H), 7.50-7.37 (m, 3H), 7.19 (dd, 1H, J_1 = 8.53 Hz J_2 = 1.90 Hz), 2.21 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 199.0, 169.1, 141.4, 138.2, 134.5, 132.6, 129.6, 129.3, 128.4, 125.1, 124.0, 121.3, 25.2 ppm. IR (ATR): ν_{max} = 3230, 1690, 1631, 1567, 1395, 1251, 915, 879, 753, 700, 654, 485 cm^{-1} MS (EI, 70 eV): *m/z* (%): 317 (25, [M+]), 276 (100), 200 (28), 167 (45), 105 (61), 77 (100), 51 (40).



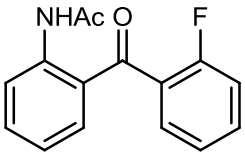
N-(2-benzoyl-4-(2-hydroxyethyl)phenyl)-acetamide (4i) Method A. Off-white, solid; 238 mg (0.84 mmol, yield: 84%). m.p. 87-92 °C Rf: 0.5 (in Hexane:EtOAc = 2:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 10.63 (s, 1H), 8.47 (d, 1H, *J* = 8.21 Hz), 7.69–7.39 (m, 7H), 3.79 (t, 2H, *J*

= 6.48 Hz), 3.03 (br, 1H), 2.77 (t, 2H, *J* = 6.48 Hz), 2.17 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 199.3, 169.2, 138.3, 134.5, 133.6, 132.8, 132.5, 129.8, 128.2, 123.6, 121.7, 63.1, 38.1, 25.0 ppm. IR (ATR): ν_{max} = 3437, 2931, 1659, 1493, 1273, 705, 590 cm^{-1} MS (EI, 70 eV): *m/z* (%): 281 (8, [M$^+$]), 265 (60), 223 (100), 210 (28), 180 (10), 132 (13), 105 (15), 77 (30). HRMS: *m/z* [M + H]$^+$ calcd for $C_{17}H_{18}NO_3$: 284.1287; found: 284.1293.



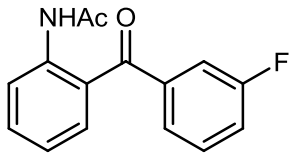
Methyl 2-(4-acetamido-3-benzoylphenyl) acetate (4j)

Method B. Light yellow, oil; 58 mg (0.19 mmol, yield: 37%). Rf: 0.44 (in Hexane:EtOAc = 1:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 10.64 (s, 1H), 8.50 (d, 1H *J* = 9.16 Hz), 7.65 – 7.39 (m, 7H), 3.59 (s, 3H), 3.49 (s, 2H), 2.14 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 199.3, 171.4, 169.2, 139.3, 138.3, 135.0, 134.1, 132.6, 130.0, 128.3, 127.7, 123.4, 121.7, 52.1, 40.2, 25.2 ppm. IR (ATR): ν_{max} = 3315, 2931, 1730, 1589, 1510, 1256, 705 cm^{-1} MS (EI, 70 eV): *m/z* (%): 311 (15, [M$^+$]), 269 (37), 210 (100), 180 (28), 132 (20), 77 (35). HRMS: *m/z* [M + H]$^+$ calcd for $C_{18}H_{19}FNO_2$: 312.1236; found: 312.1242.

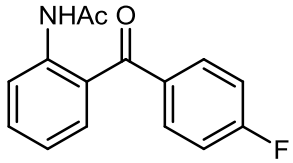


N-(2-(2-fluorobenzoyl)phenyl)acetamide[1] **(4k)** Method A. White, solid; 171 mg (0.67 mmol, yield: 67%). m.p. 81-93°C. Rf: 0.41 (in Hexane:EtOAc = 1:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 11.26 (s, 1H), 8.66 (d, 1H, *J* = 8.53 Hz), 7.52 - 7.33 (m, 4H), 7.21-6.93 (m, 3H),

2.17 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 197.0, 169.6, 162.3 (d, J_{CF} = 260.6 Hz), 141.2, 135.4, 134.0 (d, J_{CF} = 1.8 Hz), 133.0 (d, J_{FC} = 8.3 Hz), 130.1 (d, J_{CF} = 2.3 Hz), 127.4 (d, J_{CF} = 15.2 Hz), 124.2 (d, J_{CF} = 3.7 Hz), 122.3, 122.2, 120.7, 116.3 (d, J_{FC} = 21.6 Hz), 25.4 ppm. IR (ATR): ν_{max} = 3242, 1689, 1633, 1583, 1528, 1447, 1280, 1214, 1152, 925, 751, 632, 524 cm^{-1} MS (EI, 70 eV): *m/z* (%): 257 (40, [M$^+$]), 214 (100), 196 (36), 134 (35), 120 (45), 95 (30).

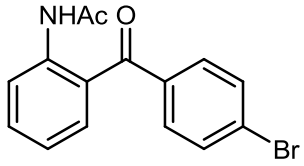


N-(2-(3-fluorobenzoyl)phenyl)acetamide[1] **(4l)** Method A. Pale-white, solid; 220 mg (0.86 mmol, yield: 86 %). m.p.: 63 – 65 °C. R_f : 0.47 (Hexane:EtOAc = 1:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 10.64 (s, 1H), 8.50 (d, 1H, *J* = 8.37 Hz), 7.49 – 7.26 (m, 5H), 7.21 – 7.13 (m, 1H), 6.97 (t, 1H *J* = 7.66 Hz), 2.11 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 198.0 (d, J_{CF} = 1.8 Hz), 169.2, 162.3 (d, J_{CF} = 248.6 Hz), 140.4, 134.6, 133.3, 129.9 (d, J_{CF} = 7.8 Hz), 125.6 (d, J_{CF} = 3.2 Hz), 122.7, 122.1, 121.6, 119.4 (d, J_{CF} = 21.1 Hz), 116.5 (d, J_{CF} = 22.5 Hz), 25.2 ppm. IR ν = 3221, 3077, 2962, 1667, 1479, 1292, 1254, 1010, 753, 719, 529 cm^{-1}; MS (EI, 70 eV) *m/z* (%): 257 (22, [M+]), 214 (100), 185 (17), 134 (23), 120 (53), 95 (33), 92 (24).

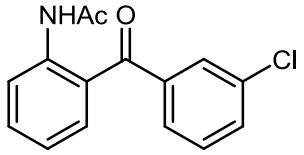


N-(2-(4-fluorobenzoyl)phenyl)-acetamide[1] **(4m)** Method A. Pale-white, solid; 182 mg (0.71 mmol, yield: 71 %). m.p.: 100 – 102 °C. R_f : 0.49 (Hexane:EtOAc = 1:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 10.63 (s, 1H), 8.58 (d, 1H, *J* = 8.37 Hz), 7.76 – 7.71 (m, 2H), 7.59 – 7.48 (m, 2H), 7.19 – 7.05 (m, 3H), 2.21 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 197.9,

169.1, 165.3 (d, J_{CF} = 254.6 Hz), 140.2, 134.6 (d, J_{CF} = 2.8 Hz), 134.2, 133.0, 132.5 (d, J_{CF} = 9.2 Hz), 123.3, 122.1, 121.7, 115.5 (d, J_{CF} = 22.1 Hz), 25.2 ppm. IR ν = 3221, 3070, 1662, 1595, 1479, 1289, 1225, 930.1, 847.9, 755.7, 599.8, 507.6 cm^{-1}; MS (EI, 70 eV) *m/z* (%): 257 (17, [M+]), 214 (100), 185 (15), 134 (19), 123 (25), 120 (30), 95 (34), 92 (16).



N-(2-(4-bromobenzoyl)phenyl)-acetamide[1] **(4n)** Method A. Offwhite, solid; 215 mg (0.68 mmol, yield: 62%). m.p.: 142 – 143 °C. R_f: 0.66 (hexane:EtOAc = 1:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 10.70 (s, 1H), 8.60 (d, 1H *J* = 8.37 Hz), 7.64-7.48 (m, 6H), 7.08 (t, 1H *J* = 7.19 Hz), 2.21 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 198.4, 169.1, 140.4, 137.3, 134.5, 133.1, 131.6, 131.3, 127.6, 122.9, 122.1, 121.6, 25.2 ppm. IR (ATR): ν_{max} = 3310, 3057, 1515, 1260, 757, 471 cm^{-1}. MS (EI, 70 eV) *m/z* (%): 317 (26, [M+]), 276 (100), 196 (47), 167 (41), 157 (23), 134 (42), 120 (58), 92 (34).



N-(2-(3-chlorobenzoyl)phenyl)acetamide[1] **(4o)** Method A. Offwhite, solid; 233 mg (0.85 mmol, yield: 85%). m.p.: 76 – 82 °C. R_f : 0.76 (hexane:EtOAc = 1:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 10.75 (s, 1H), 8.60 (d, 1H, *J* = 8.37 Hz), 7.66-7.37 (m, 6H), 7.07 (t, 1H, *J* = 8.37 Hz), 2.20 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 198.0, 169.0, 140.5, 140.1, 134.6, 134.5, 133.2, 132.3, 129.5, 127.8, 122.6, 122.1, 121.5, 25.2 ppm. IR (ATR): ν_{max} = 3242, 3209, 3056, 1691, 1633, 1585, 1534, 1252, 752, 710 cm^{-1}. MS (EI, 70 eV) *m/z* (%): 273 (25 [M+]), 230 (100), 196 (50), 167 (16), 139 (18), 134 (25), 120 (50), 111 (27), 92 (26).

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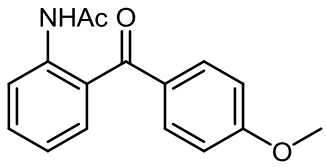
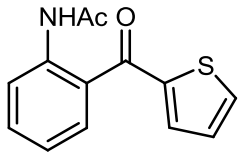


N-(2-(4-methoxybenzoyl)phenyl)acetamide[1] **(4p)** Method A. White, solid; 208 mg (0.78 mmol, yield: 78%). m.p. 115-121°C (lit.: 107-121°C). Rf: 0.25 (in Hexane:EtOAc = 1:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 10.51 (s, 1H), 8.54 (d, 1H *J* = 8.37 Hz), 7.72 (d, 2H *J* = 8.85 Hz), 7.55 - 7.50 (m, 2H), 7.07 (t, 1H *J* = 7.58 Hz), 6.95 (d, 2H *J* = 8.85 Hz), 3.87 (s, 3H), 2.18 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 197.8, 169.0, 163.3, 139.7, 133.4, 132.7, 132.5, 130.8, 124.2, 122.0, 121.6, 113.6, 55.5, 25.1 ppm. IR (ATR): ν_{max} = 3307, 2927, 1671, 1646, 1497, 1253, 1020, 926, 758, 690, 598 cm^{-1} MS (EI, 70 eV): *m/z* (%): 269 (40, [M^{+}]), 226 (100), 219 (38), 135 (35), 92 (26).



***N*-(2-(thiophene-2-carbonyl)phenyl)-acetamide**[1] **(4q)** Method B. Yellow, oil; 78 mg (0.32 mmol, yield: 32%). Rf: 0.27 (in hexane:EtOAc = 3:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 10.25 (s, 1H), 8.52 (d, 1H *J* = 8.21 Hz), 7.80–7.74 (m, 2H), 7.57–7.51 (m, 2H), 7.17– 7.10 (m, 2H), 2.18 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 189.8, 168.9, 143.9, 139.1, 135.6, 134.9, 133.6, 131.6, 128.0, 124.2, 122., 121.8 ppm. IR (ATR): ν_{max} = 3320, 3091, 1692, 1614, 1507, 1406, 1261, 712, 645 cm^{-1}; MS (EI, 70 eV): *m/z* (%): 245 (18, [M^{+}]), 202 (100), 170 (40), 134 (18), 119 (40), 111 (39).

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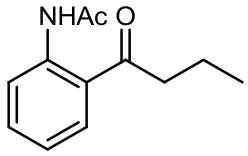
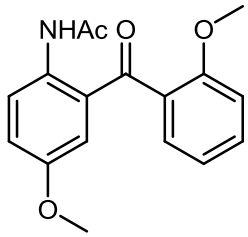


N-(2-butyrylphenyl)acetamide[1] **(4r)** Method B. Offwhite, solid; 90 mg (0.44 mmol, yield:44%). m.p 43-46 °C (lit.: 46-47 °C). Rf: 0.75 (in hexane:EtOAc = 1:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 11.70 (s, 1H), 8.68 (d, 1H, *J* = 8.53 Hz), 7.86 (d, 1H, *J* = 7.90 Hz), 7.48 (t, 1H, *J* = 7.42 Hz), 7.05 (t, 1H, *J* = 7.42 Hz), 2.95 (t, 2H, *J* = 7.42 Hz), 2.18 (s, 3H), 1.71 (sextet, 2H, *J* = 7.42 Hz), 0.97 (t, 3H, *J* = 7.42 Hz) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 204.9, 169.3, 140.8, 134.6, 130.6, 122.1, 121.4, 120.6, 41.7, 25.4, 17.8, 13.6 ppm. IR (ATR): ν_{max} = 3214, 2961, 1691, 1646, 1586, 1521, 1467, 1362, 1199, 897, 750, 725, 515, 485 cm^{-1}. MS (EI, 70 eV): *m/z* (%): 205 (10, [M^{+}]), 162 (82), 120 (100), 92 (35), 65 (27).



N-(4-methoxy-2-(2-methoxybenzoyl)phenyl)-acetamide[1] **(4s)** Method B. Offwhite, solid; 98 mg (0.33 mmol, yield: 33%). m.p. 147-152 °C Rf: 0.57 (in Hexane:EtOAc = 1:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 12.06 (s, 1H), 8.42 (d, 1H, *J* = 2.53 Hz), 7.46 – 7.33 (m, 2H), 7.21 (dd, 1H, J_1 = 7.42 Hz J_2 = 1.74 Hz), 7.03 – 6.95 (m, 2H), 6.47 (dd, 1H, J_1 = 8.85 Hz J_2 = 2.53 Hz), 3.85 (s, 3H), 3.74 (s, 3H), 2.25 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 198.9, 169.8, 164.9, 156.2, 144.1, 136.8, 131.3, 129.4, 128.5, 120.3, 115.5, 111.2, 109.1, 103.7, 55.5, 25.6 ppm. IR (ATR): ν_{max} = 2921, 1694, 1580, 1510, 1431, 1239, 757, 625 cm^{-1} MS (EI, 70 eV): *m/z* (%): 299 (80), 256 (100), 242 (57), 226 (55), 164 (60), 150 (82), 135 (77), 123 (40), 92 (47), 77 (62).

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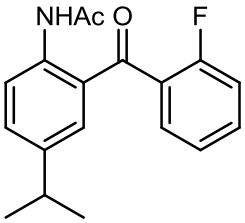
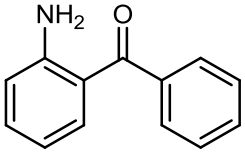


N-(2-(2-fluorobenzoyl)-4-isopropylphenyl)-acetamide (4t) Method B. Brown, solid; 98 mg (0.36 mmol, yield: 36%). m.p. 88-90 °C (lit 162-163°C) Rf: 0.68 (in Hexane:EtOAc = 1:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 11.12 (s, 1H), 8.55 (d, 1H, *J* = 8.69 Hz), 7.50-7.35 (m, 3H), 7.25 - 7.06 (m, 3H), 2.72 (sextet, *J* = 6.95 Hz), 2.16 (s, 3H), 1.07 (d, 6H, *J* = 6.95 Hz) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 196.9, 169.2, 161.5 (d, J_{CF} = 252.3 Hz), 142.7, 139.0, 133.5, 133.3 (d, J_{CF} = 8.3 Hz), 131.7 (d, J_{CF} = 2.3 Hz), 130.2 (d, J_{CF} = 2.8 Hz), 127.5 (d, J_{CF} = 14.3 Hz), 124.1 (d, JCF = 3.7 Hz), 122.3, 120.9, 116.2 (d, J_{CF} = 21.6 Hz), 33.2, 25.3, 23.6 ppm. IR (ATR): ν_{max} = 2962, 1664, 1609, 1491, 1271, 825, 760, 597 cm^{-1} MS (EI, 70 eV): 299 (30, [M$^+$]), 257 (27), 242 (100), 123 (34), 95 (20) *m/z* (%). HRMS: *m/z* [M + H]$^+$ calcd for $C_{18}H_{19}FNO_2$: 300.1400; found: 300.1408.



(2-aminophenyl)(phenyl)methanone[2] (5a) Method A. Brown, solid; 133 mg (0.56 mmol, yield: 56%). m.p.: 100 °C. R_f: 0.65 (Hexane:EtOAc = 1:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.6 –7.43 (m, 6H), 7.26 (t, 1H, *J* = 7.27 Hz), 6.71 (d, 1H, *J* = 7.58 Hz), 6.57 (t, 1H, *J* = 7.11 Hz), 6.1 (s, 2H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 199.0, 150.9, 140.0, 134.5, 134.2, 131.0, 129.1, 128.0, 118.1, 116.9, 115.4 ppm. IR ν = 3429, 3312, 1612, 1549, 1070, 935, 741, 700, 642, 428 cm^{-1}; MS (EI, 70 eV) *m/z* (%): 196 (100, [M+]), 120 (51), 105 (15), 92 (24), 77 (36), 65 (25), 51 (14).

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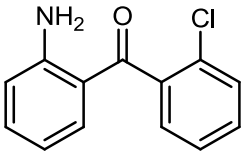
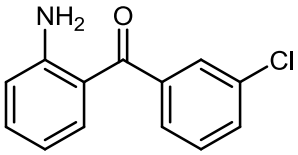
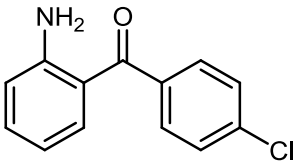


(2-aminophenyl)(2-chlorophenyl)methanone (5b) Method A. Yellow, oil; 82 mg (0.35 mmol, yield: 35 %). R_f : 0.62 (Hexane:EtOAc = 1:1). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.38–7.17 (m, 5H), 7.08 (dd, 1H, J_1 = 8.2 Hz J_2 = 1.3 Hz), 6.63 (d, 1H, J = 8.2 Hz), 6.49–6.42 (m, 1H), 6.20 (s, 2H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 197.2, 151.3, 139.8, 135.2, 134.6, 130.6, 130.3, 129.8, 128.3, 126.5, 117.4, 116.9, 115.7 ppm. IR ν = 3460, 3342, 2922, 1614, 1544, 1450, 1242, 930.1, 743.0, 636.6 cm^{-1}; MS (EI, 70 eV) *m/z* (%): 231 (32, [M+]), 196 (100), 167 (17), 139 (25), 120 (52), 111 (24), 92 (32), 65 (33).



(2-aminophenyl)(4-chlorophenyl)methanone[3] (5c) Method A. Yellow, solid; 107 mg (0.46 mmol, yield: 61%). m.p. 96-100 °C Rf: 0.63 (Hexane:EtOAc = 2:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.51–7.48 (m, 2H), 7.35–7.28 (m, 3H), 7.23–7.17 (m, 1H), 6.64 (d, 1H, J = 8.37 Hz), 6.51 (t, 1H, J = 8.06 Hz), 5.99 (s, 2H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 197.6, 150.9, 138.3, 137.2, 134.4, 134.1 130.5, 128.3, 117.7, 117.0, 115.5 ppm. IR (ATR): ν_{max} = 3474, 3367, 1579, 1545, 1239, 1090, 923, 757, 661 cm^{-1} MS (EI, 70 eV): *m/z* (%):230 (100, [M$^+$]), 214 (15), 196 (23), 139 (25), 120 (50), 111 (45), 92 (42), 65 (47), 75 (20), 65 (47).



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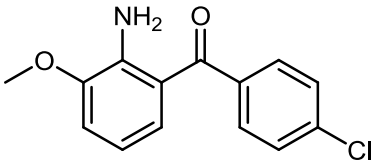
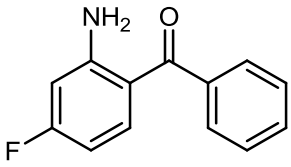
(2-aminophenyl)(3-chlorophenyl)-methanone[4] (5d) Method A. Yellow, solid; 130 mg (0.57 mmol, yield: 51%). m.p. 82-87 °C Rf: 0.64 (Hexane:EtOAc = 2:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.52 (s, 1H), 7.42–7.17 (m, 5H), 6.64 (d, 1H, *J* = 8.37 Hz), 6.51 (t, 1H, *J* = 8.06 Hz), 6.06 (br s, 2H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 197.3, 151.1, 141.7, 134.6, 134.3, 134.2, 130.9, 129.4, 128.9, 127.1, 117.4, 117.0, 115.6 ppm. IR (ATR): ν_{max} = 3440, 3331, 1610, 1541, 1442, 1305, 1241, 944, 746, 709 cm^{-1} MS (EI, 70 eV): *m/z* (%): 230 (80, [M$^+$]), 196 (55), 167 (20), 139 (25), 120 (100), 111 (45), 92 (57), 65 (60).

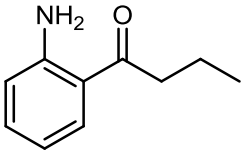


(2-amino-3-methoxyphenyl)(4-chlorophenyl)-methanone (5e) Method B. Yellow, solid; 68 mg (0.26 mmol, yield: 26%). m.p. 82-88 °C Rf: 0.68 (Hexane:EtOAc = 2:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.59 (d, 2H, *J* = 8.37 Hz), 7.42 (d, 2H, *J* = 8.53 Hz), 7.02 (dd, 1H, J_1 = 8.21 Hz J_2 = 1.11 Hz), 6.88 (d, 1H, *J* = 7.74 Hz), 6.53 (t, 1H, J = 8.06 Hz), 6.39 (br s, 2H), 3.90 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 197.5, 147.2, 142.2, 138.5, 137.1, 130.5, 128.2, 125.5, 117.0, 113.9, 112.9, 55.7 ppm. IR (ATR): ν_{max} = 3455, 3342, 1545, 1449, 1221, 1082, 959, 730 cm^{-1} MS (EI, 70 eV): *m/z* (%): 261 (100, [M$^+$]), 246 (45), 211 (63), 183 (26), 139 (48), 11 (50). HRMS: *m/z* [M + H]$^+$ calcd for $C_{14}H_{13}ClNO_2$: 262.0635; found: 262.0633.

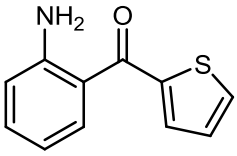


(2-amino-4-fluorophenyl)(phenyl)-methanone[5] (5f) Method B**.** Yellow, oil; 56 mg (0.26 mmol, yield: 26%). Rf: 0.52 (Hexane:EtOAc = 2:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.61–7.43 (m, 5H), 6.42–6.26 (m, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 198.0, 168.5 (d, J_{CF}

= 253.2 Hz), 153.4 (d, J_{CF} = 12.9 Hz), 140.0, 137.5 (d, J_{CF} = 11.5 Hz), 131.0, 128.9, 128.1, 115.0, 103.6 (d, J_{CF} = 22.5 Hz), 102.4 (d, J_{CF} = 24.4 Hz) ppm. IR (ATR): ν_{max} = 3386, 2964, 1602, 1241, 931, 714, 658 cm^{-1} MS (EI, 70 eV): *m/z* (%): 214 (100, [M+]), 138 (73), 110 (18), 105 (116), 83 (20), 77 (45).



1-(2-aminophenyl)butan-1-one[6] (5g) Method B. Off-white, solid; 56 mg (0.34 mmol, yield: 34%). m.p. 40-45 °C Rf: 0.73 (Hexane:EtOAc = 2:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.66 (dd, 1H, J_1 = 8.53 Hz J_2 = 1.58 Hz), 7.20–7.13 (m, 1H), 6.58-6.52 (m, 2H), 6.20 (s, 2H), 2.83 (t, 2H, J = 7.27 Hz), 1.67 (sextet, 2H, J = 7.42 Hz), 0.92 (t, 3H, J = 7.42 Hz) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 202.9, 150.2, 134.0, 131.2, 117.9, 117.3, 115.6, 41.1, 18.2, 13.9 ppm. IR (ATR): ν_{max} = 3436, 3323, 2956, 1616, 1204, 1160, 747 cm^{-1} MS (EI, 70 eV): *m/z* (%): 163 (30, [M+]), 120 (100), 92 (33), 65 (30).



(2-aminophenyl)(thiophen-2-yl)methanone[7] (5h) Method B. brown, oil; 49 mg (0.24 mmol, yield: 24%). Rf: 0.73 (Hexane:EtOAc = 2:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.77 (dd, 1H, J_1 = 8.06 Hz J_2 = 1.58 Hz), 7.65 (dd, 1H, J_1 = 5.06 Hz J_2 = 1.11 Hz), 7.57 (dd, 1H, J_1 = 3.63 Hz J_2 = 0.95 Hz), 7.34–7.26 (m, 1H), 7.15–7.11 (m, 1H), 6.75–6.66 (m, 2H), 5.71 (s, 2H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 189.4, 149.8, 144.7, 133.8, 133.7, 132.8, 132.7, 127.5, 119.0, 116.9, 115.9 ppm. IR (ATR): ν_{max} = 3350, 2915, 1578, 1249, 1161, 719, 649

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cm^{-1} MS (EI, 70 eV): *m/z* (%): 202 (100, [M+]), 170 (50), 119 (47), 111 (48), 92 (49), 65 (47).

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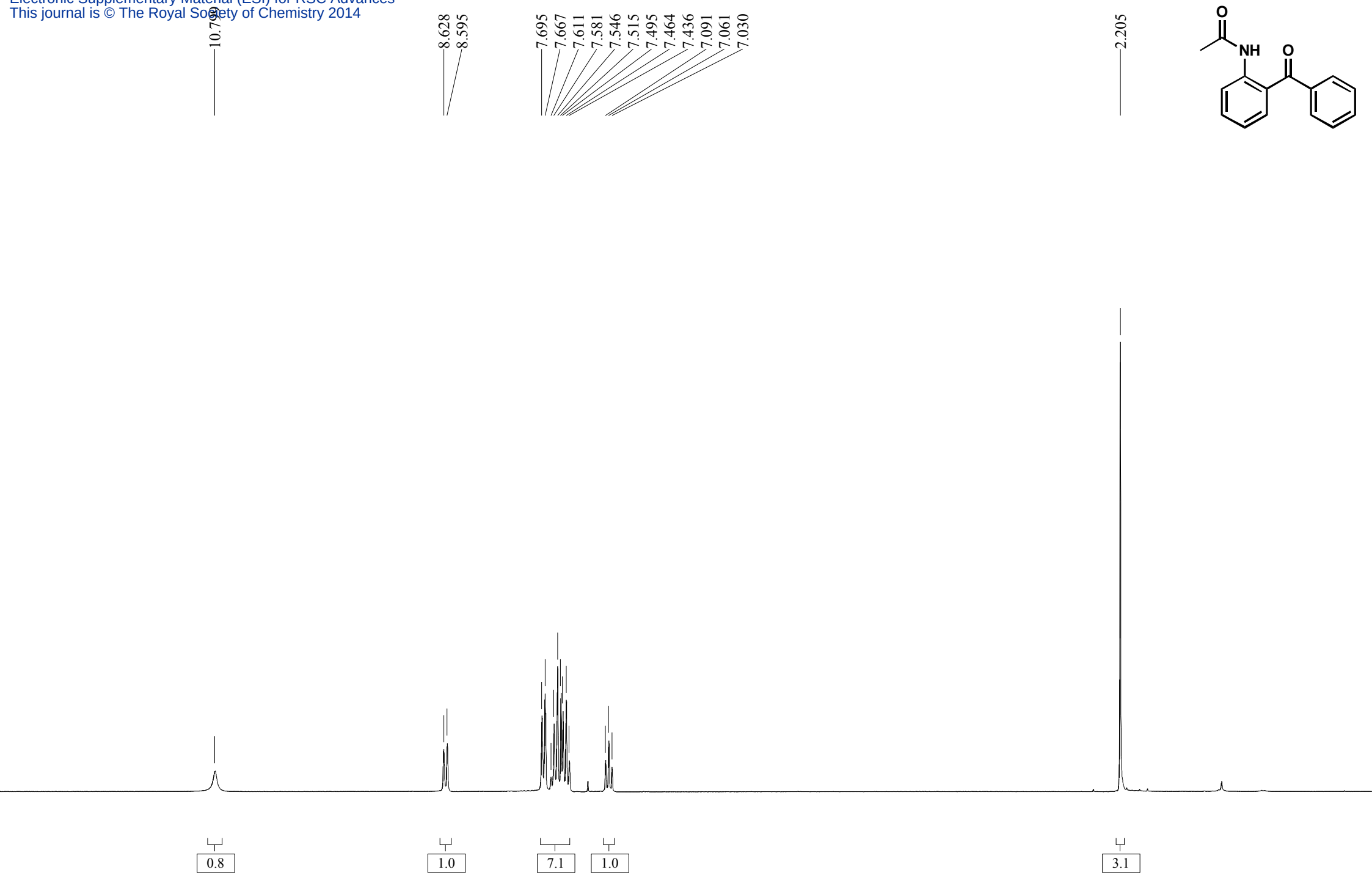

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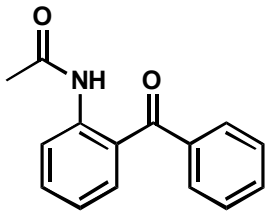

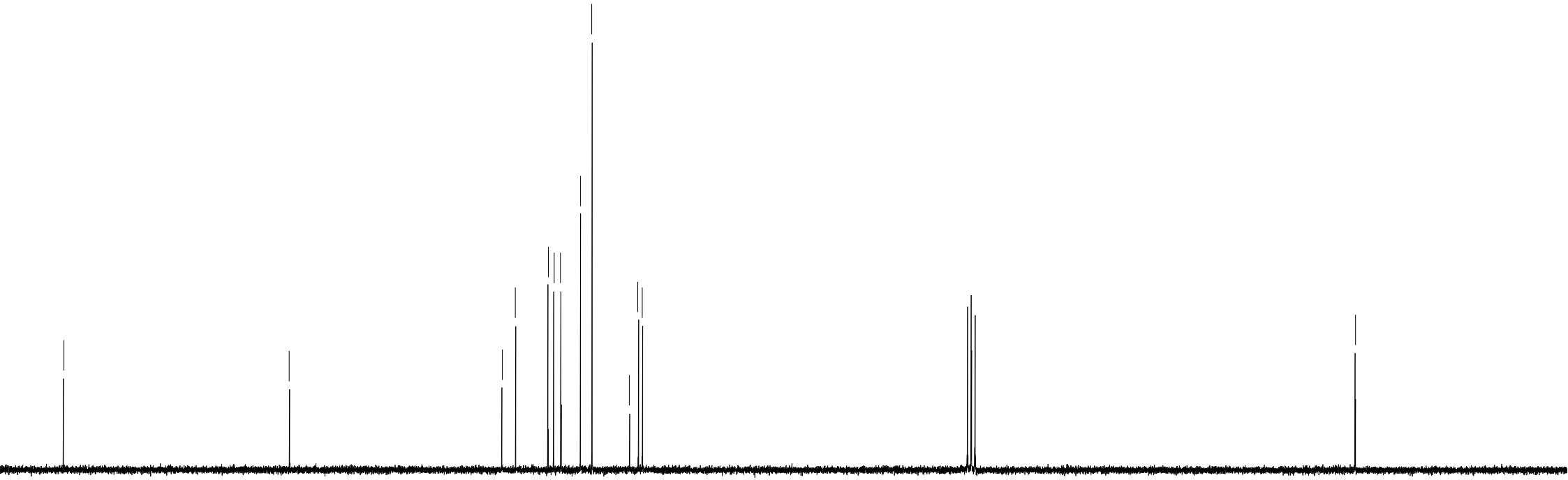

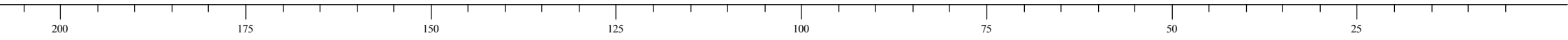

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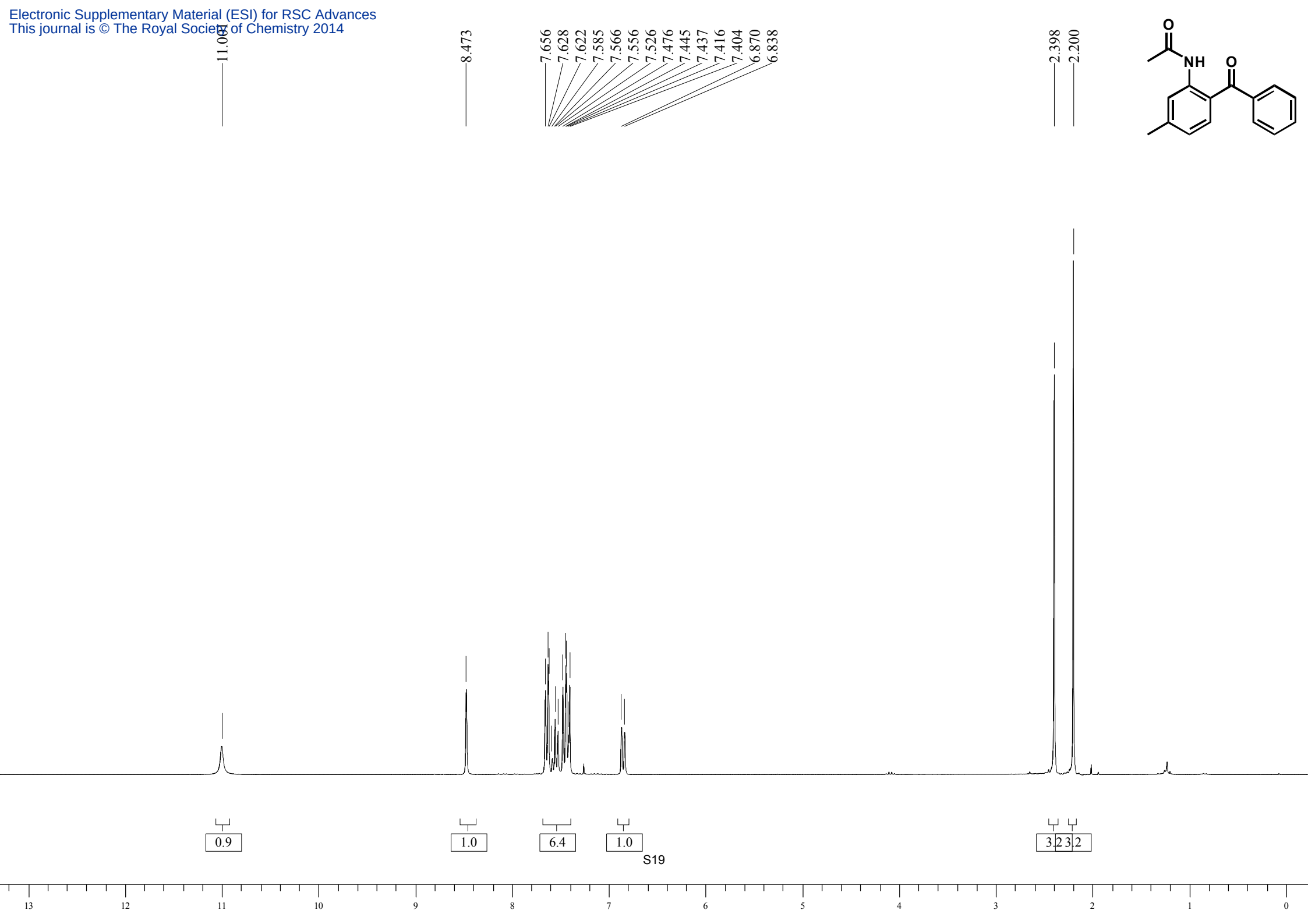

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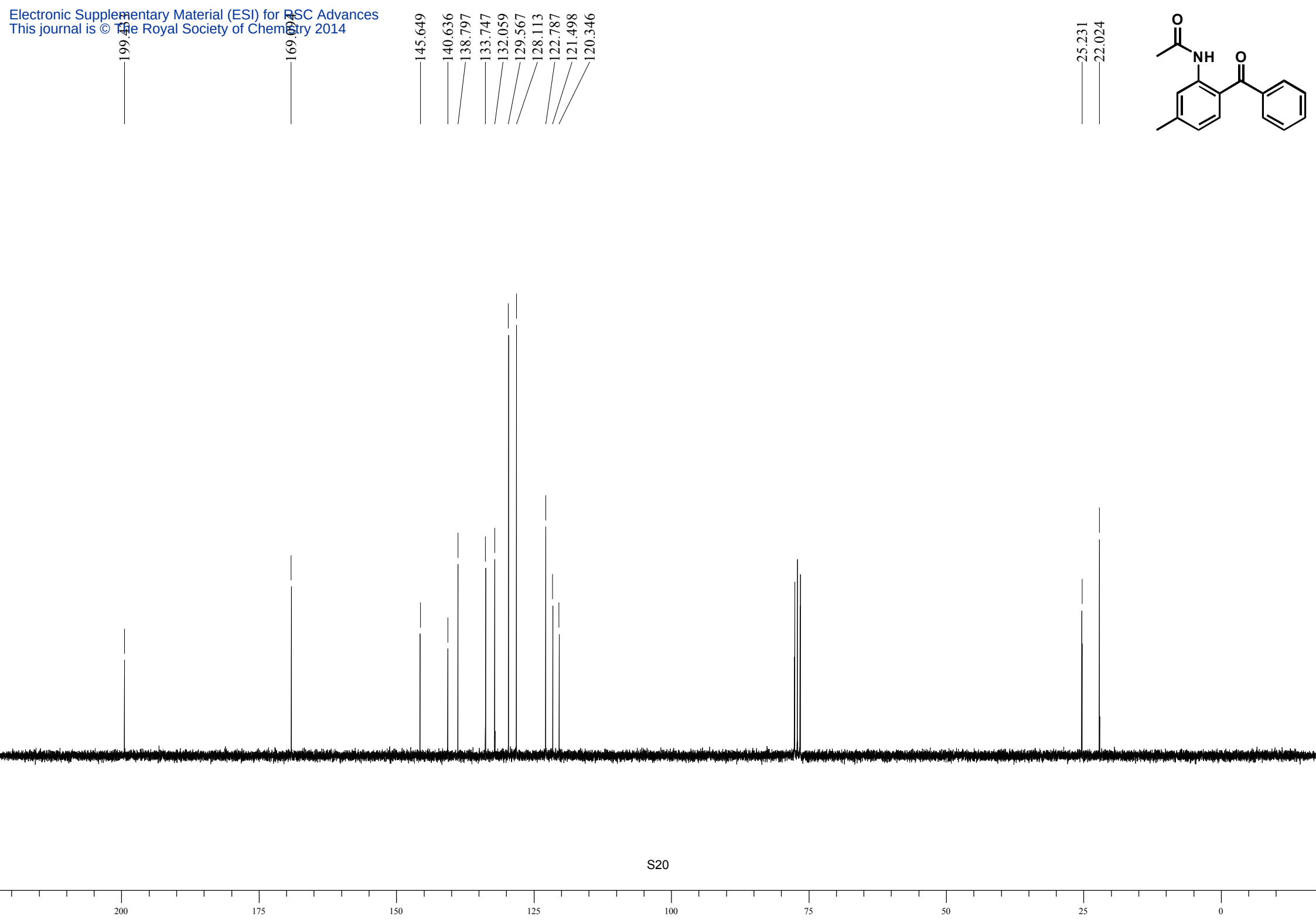

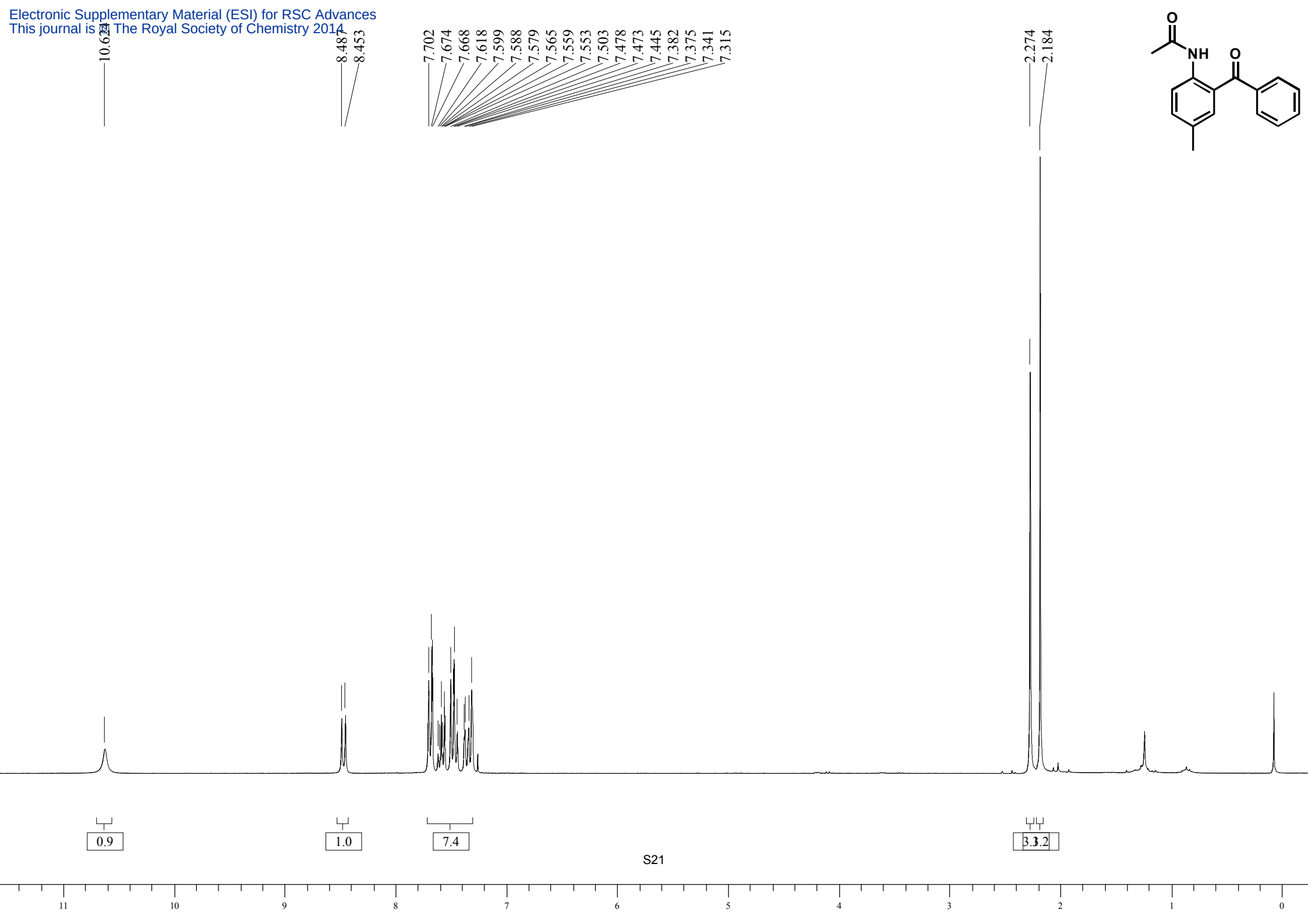
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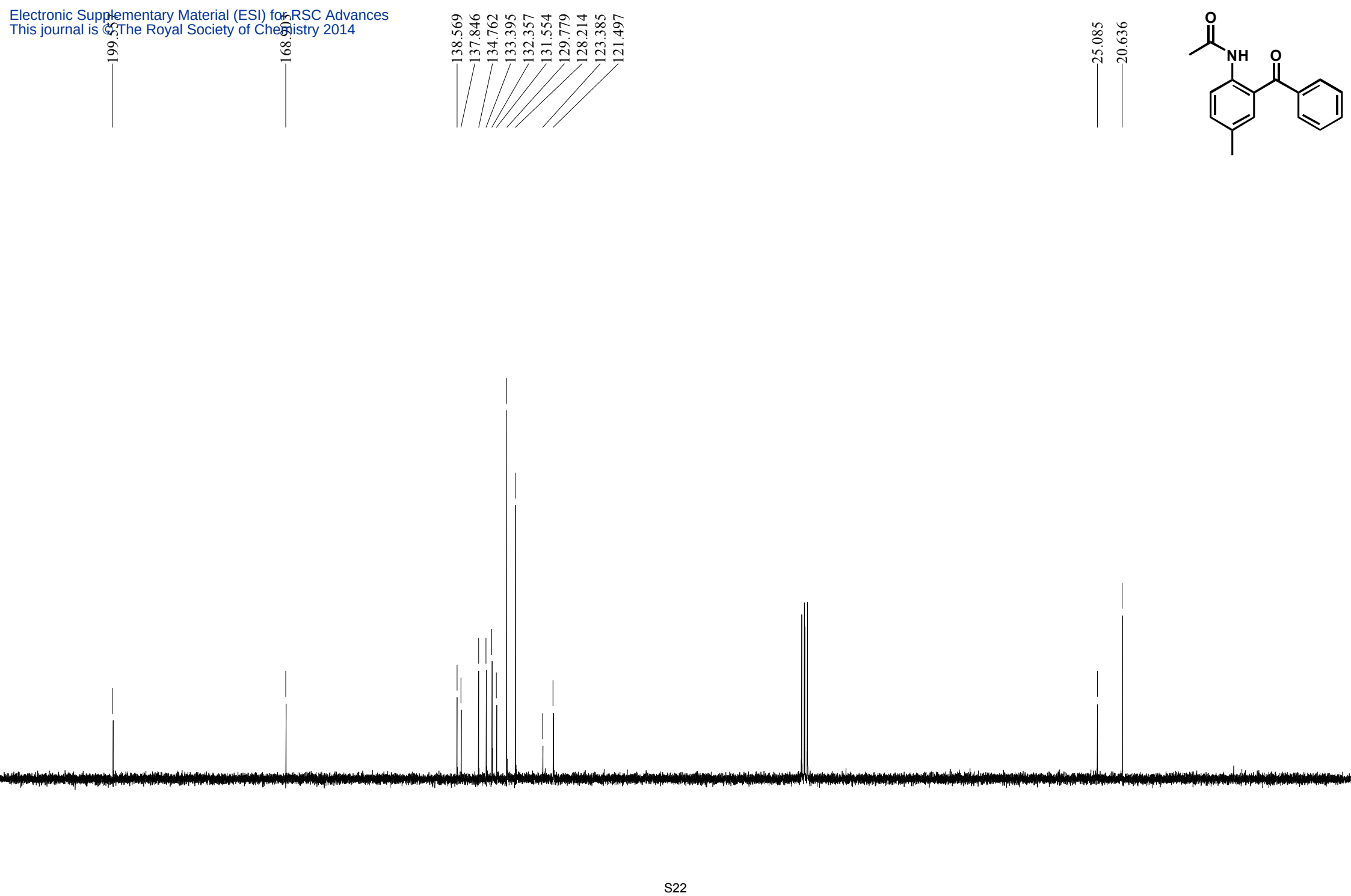

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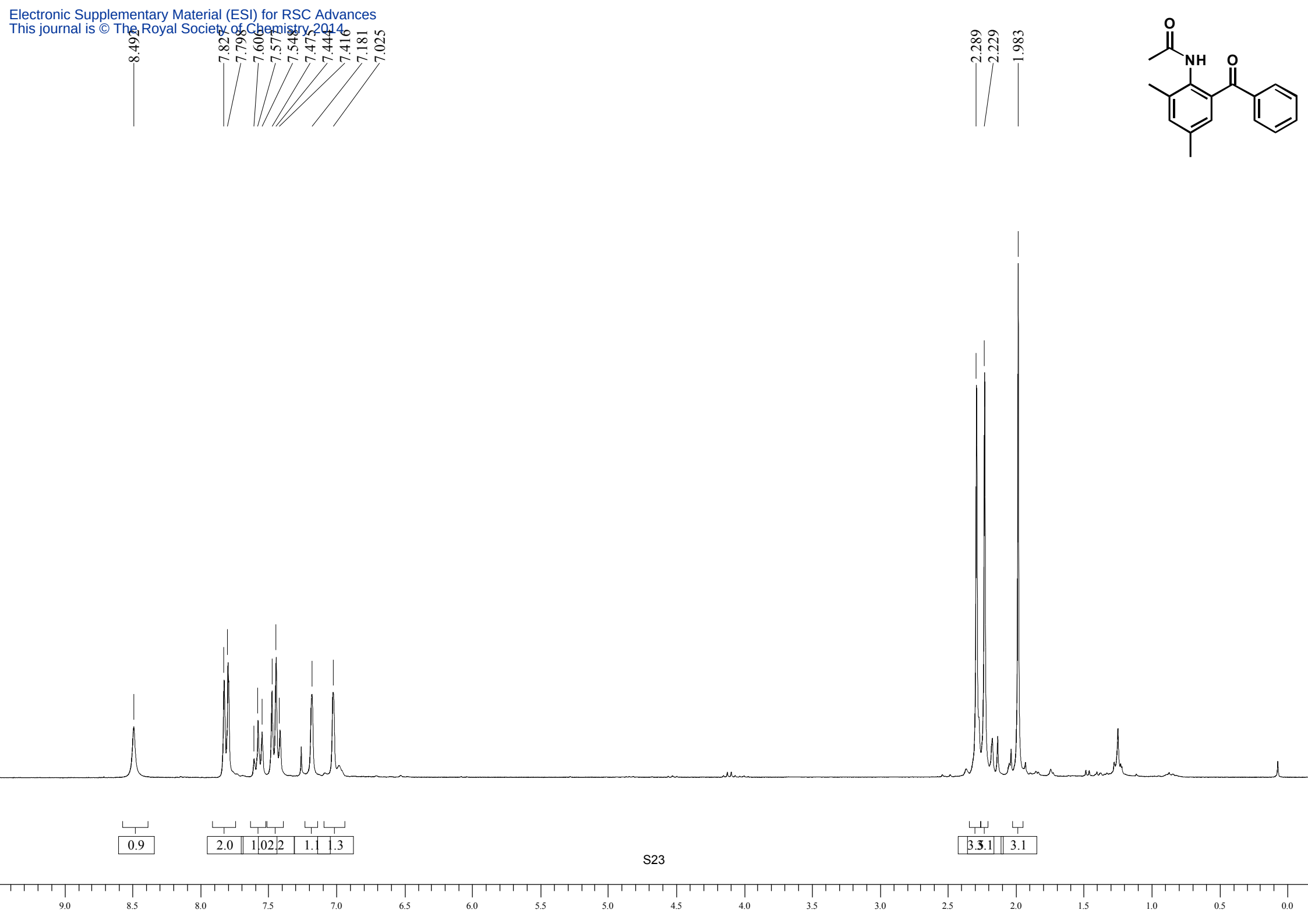

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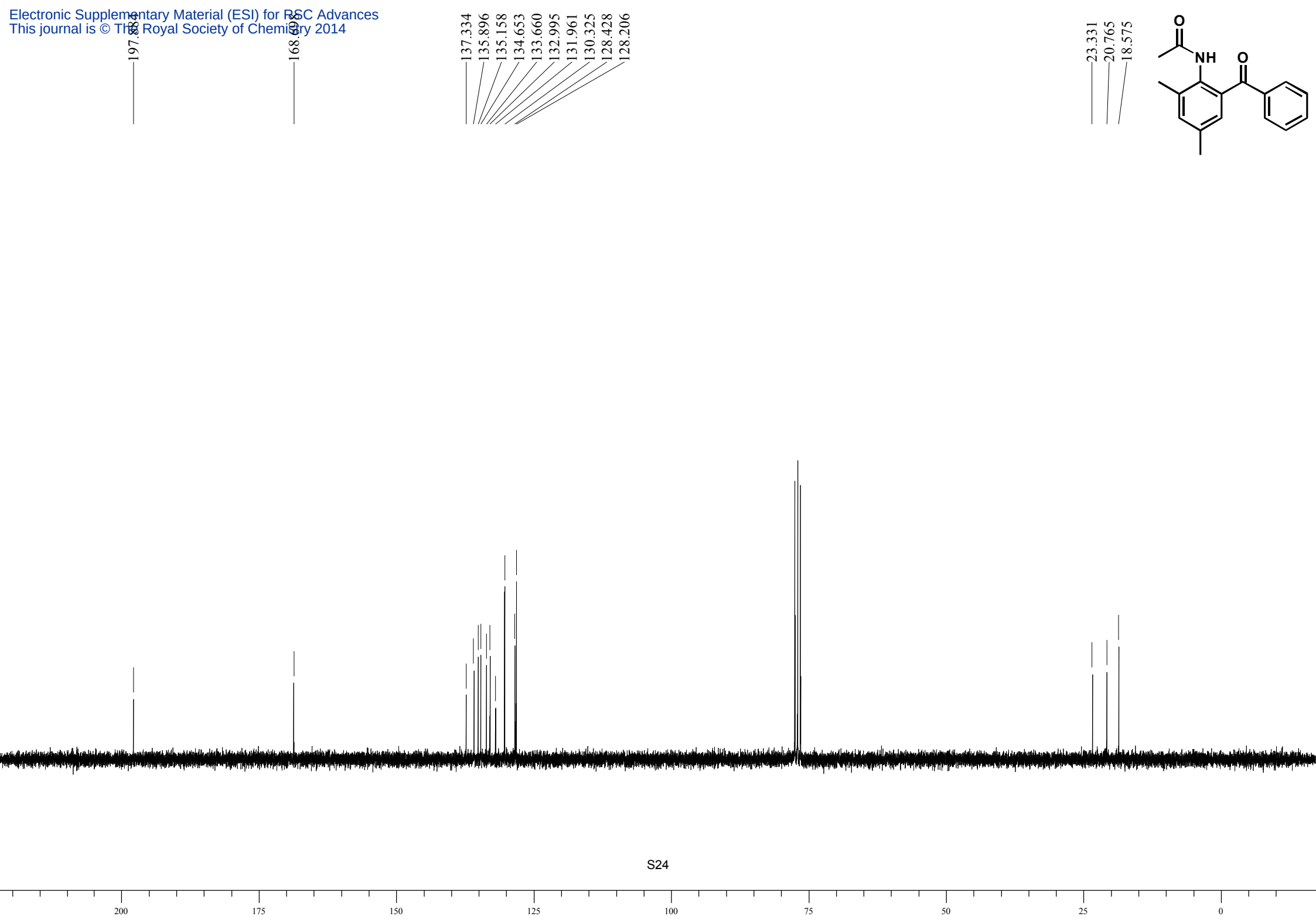

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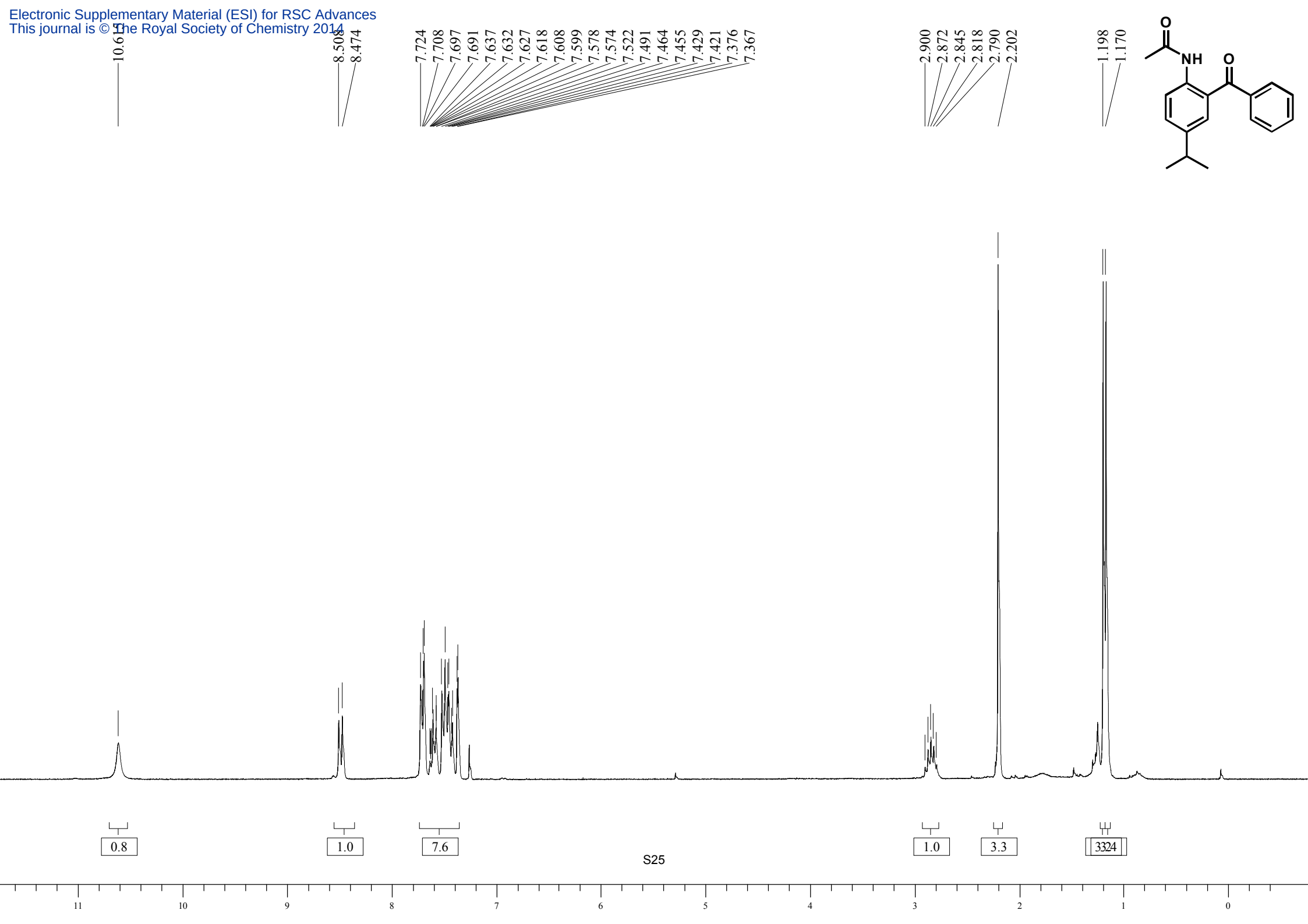

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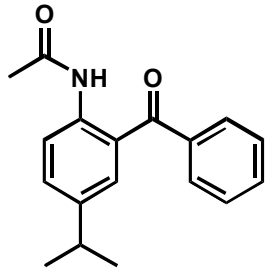

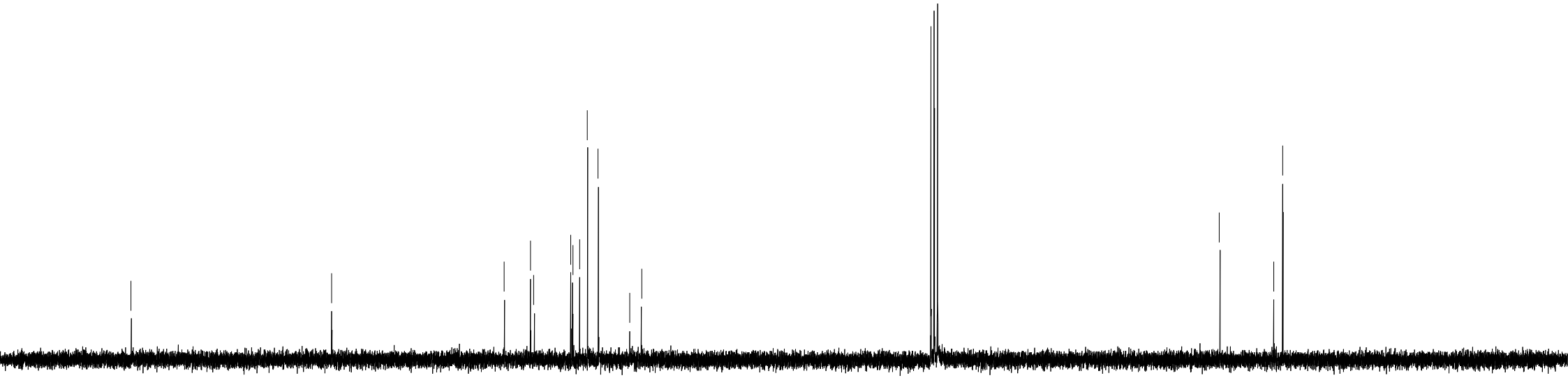

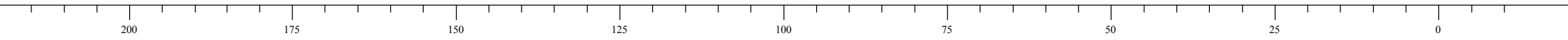

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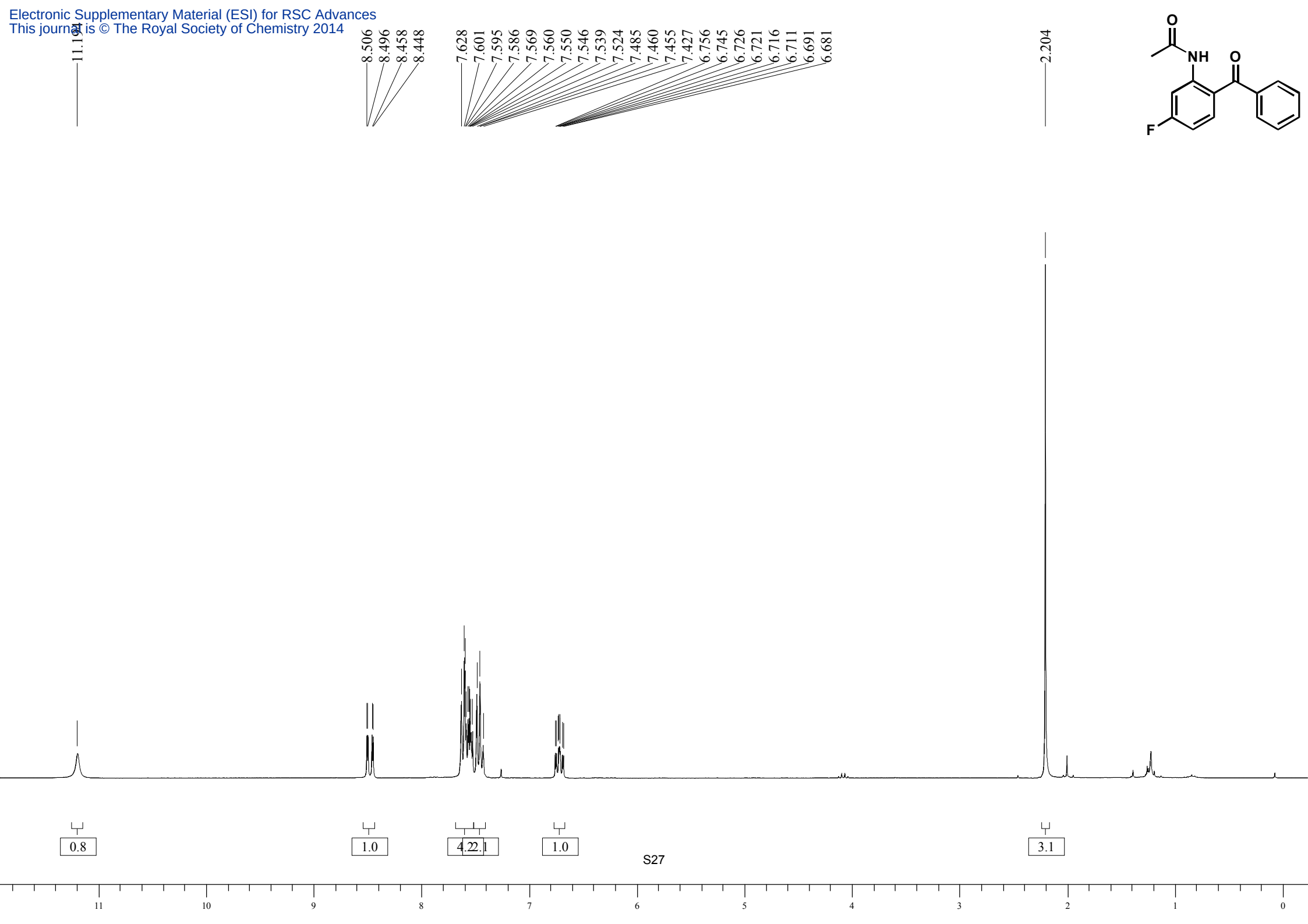

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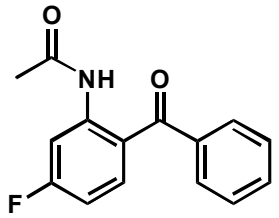

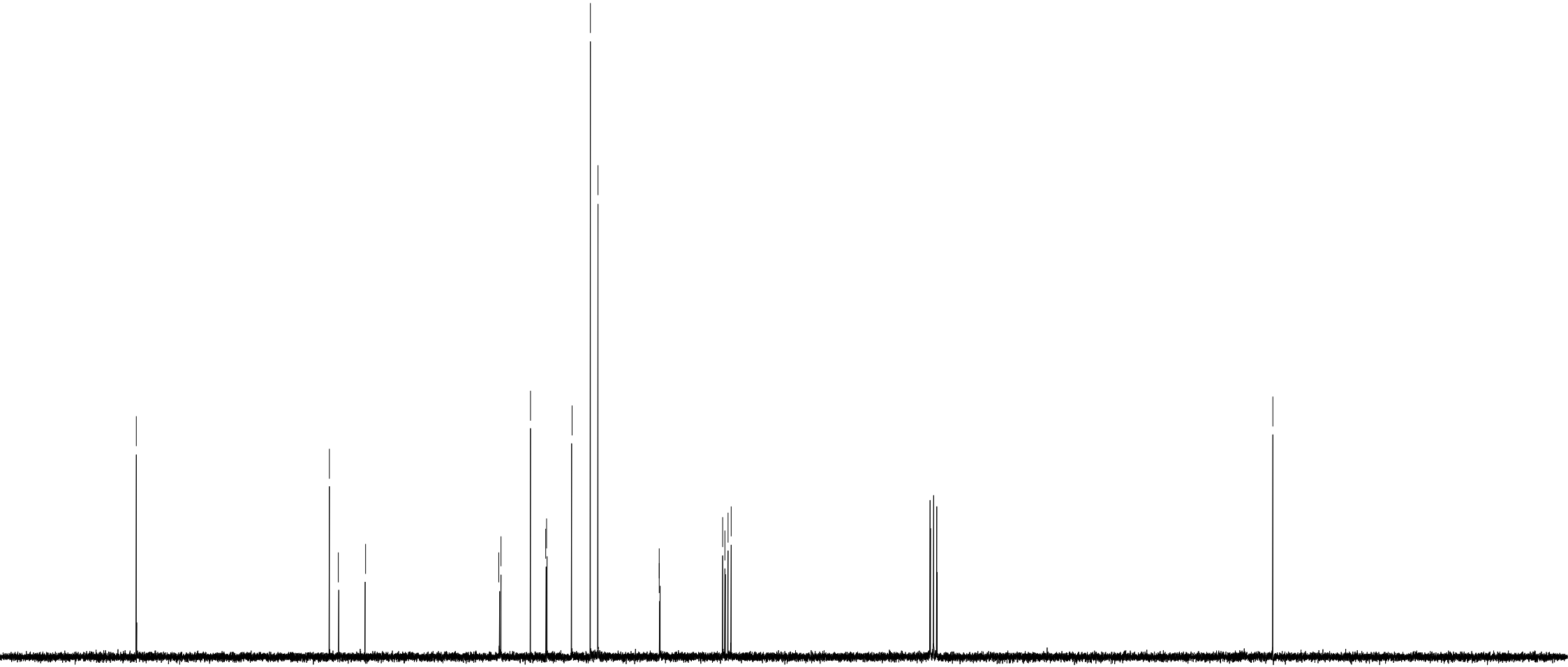

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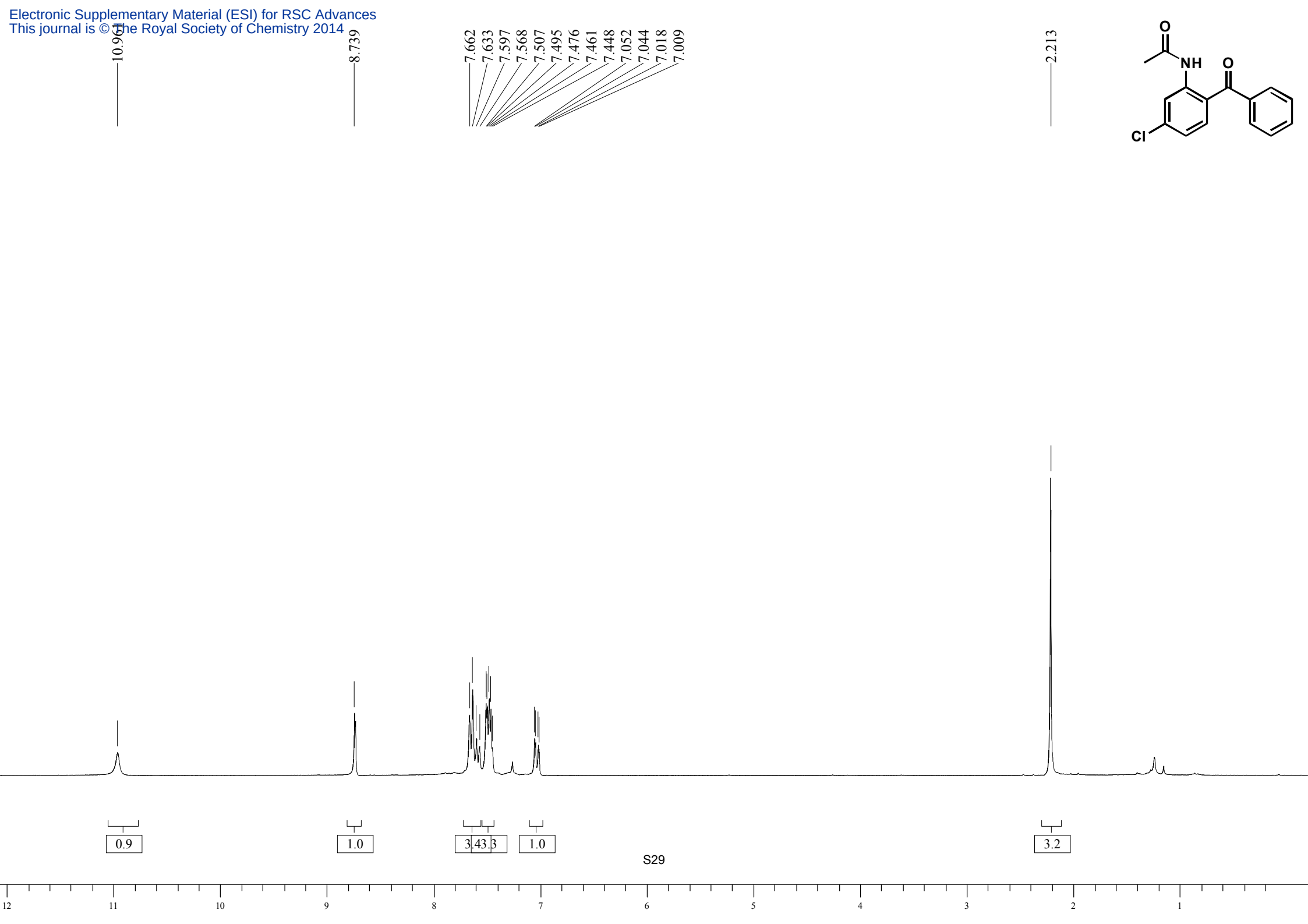

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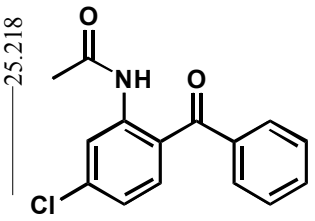

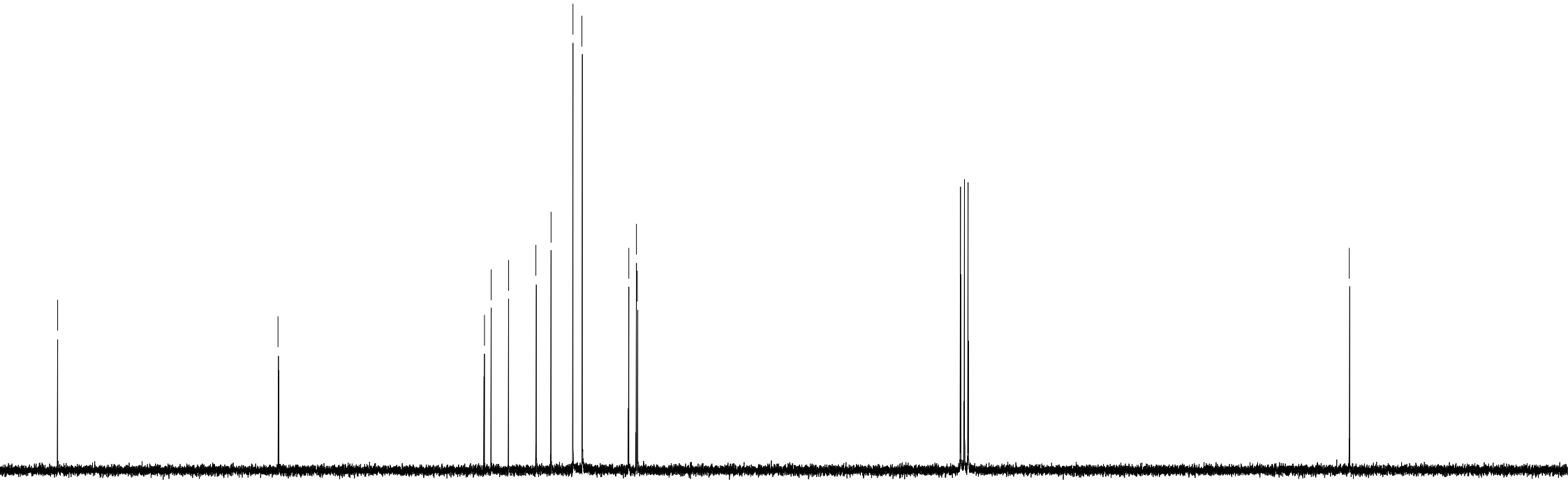

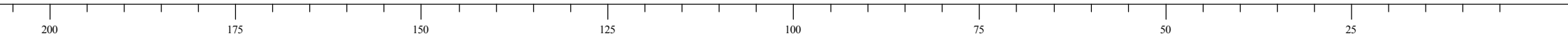

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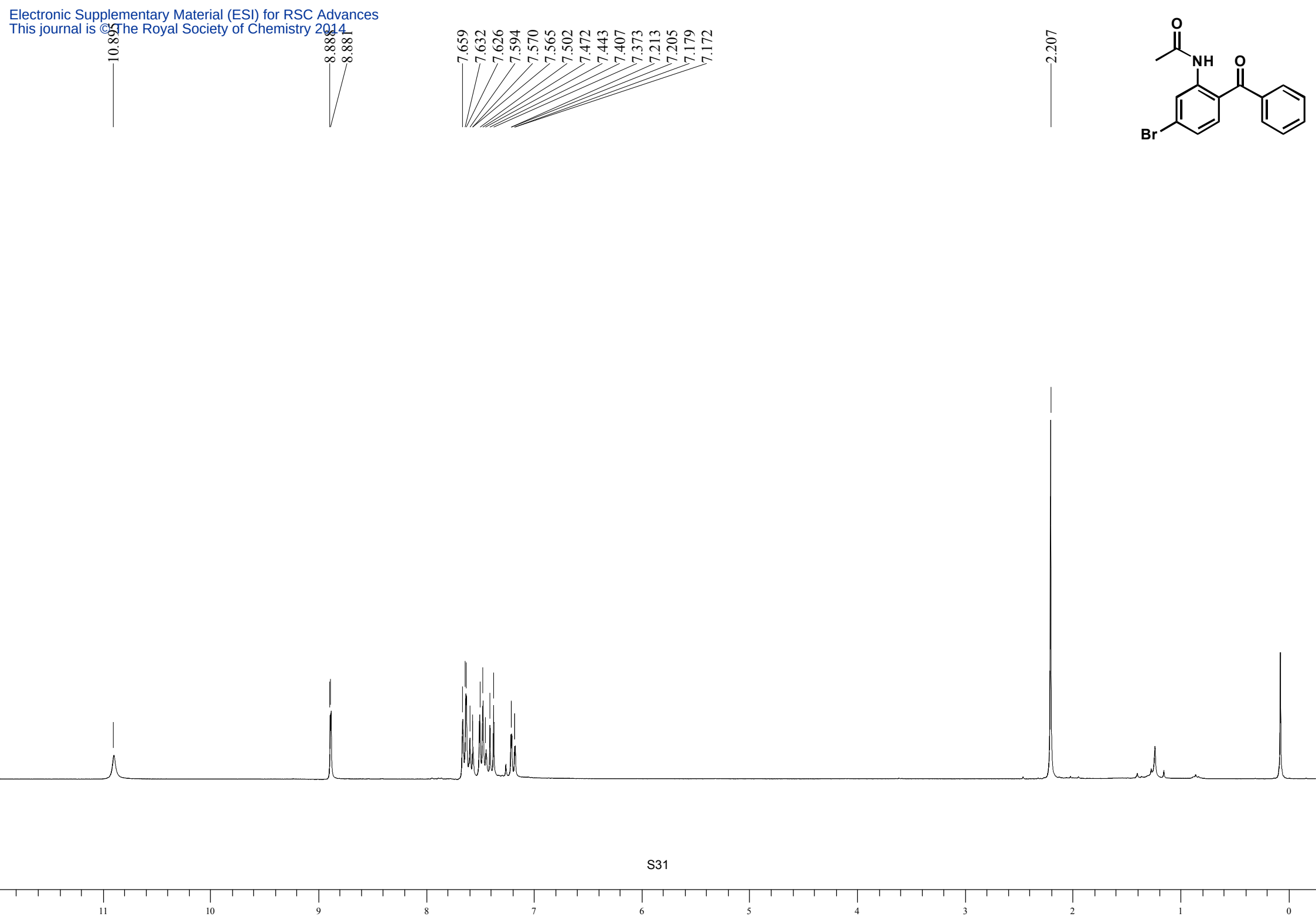

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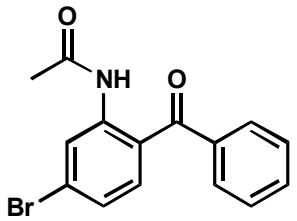

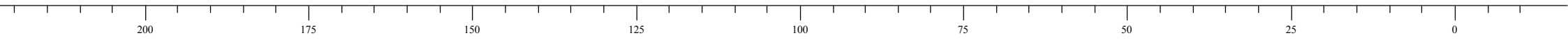

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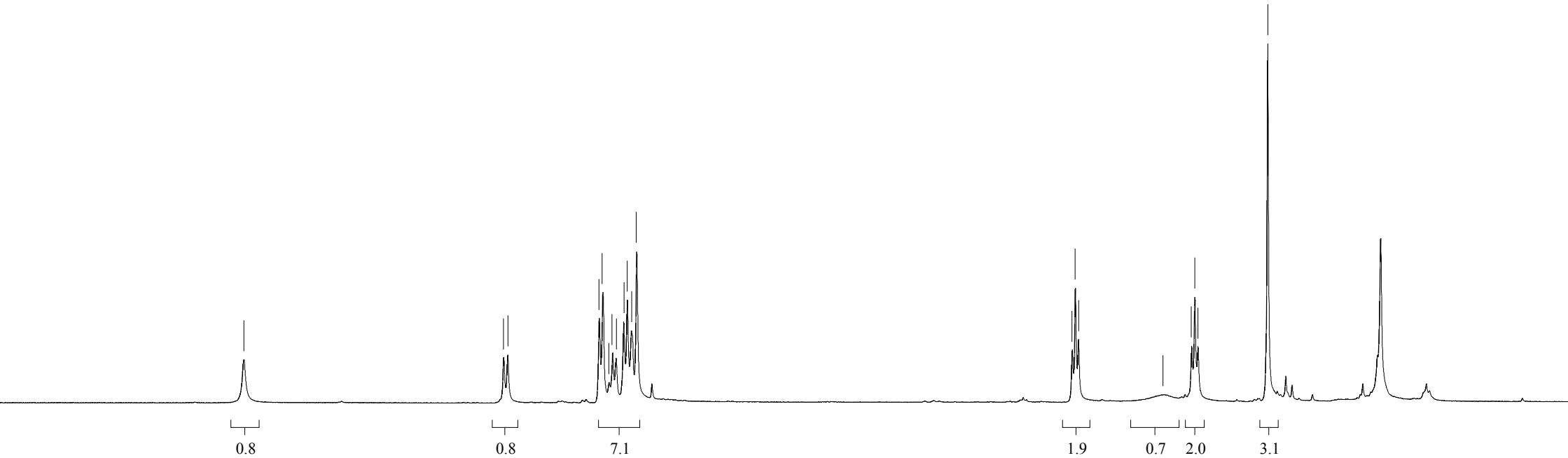

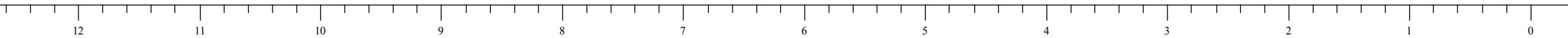

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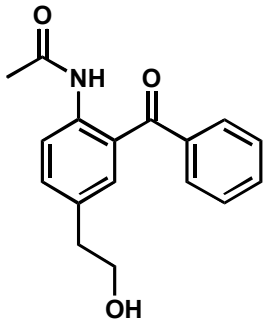

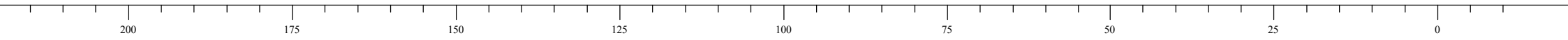

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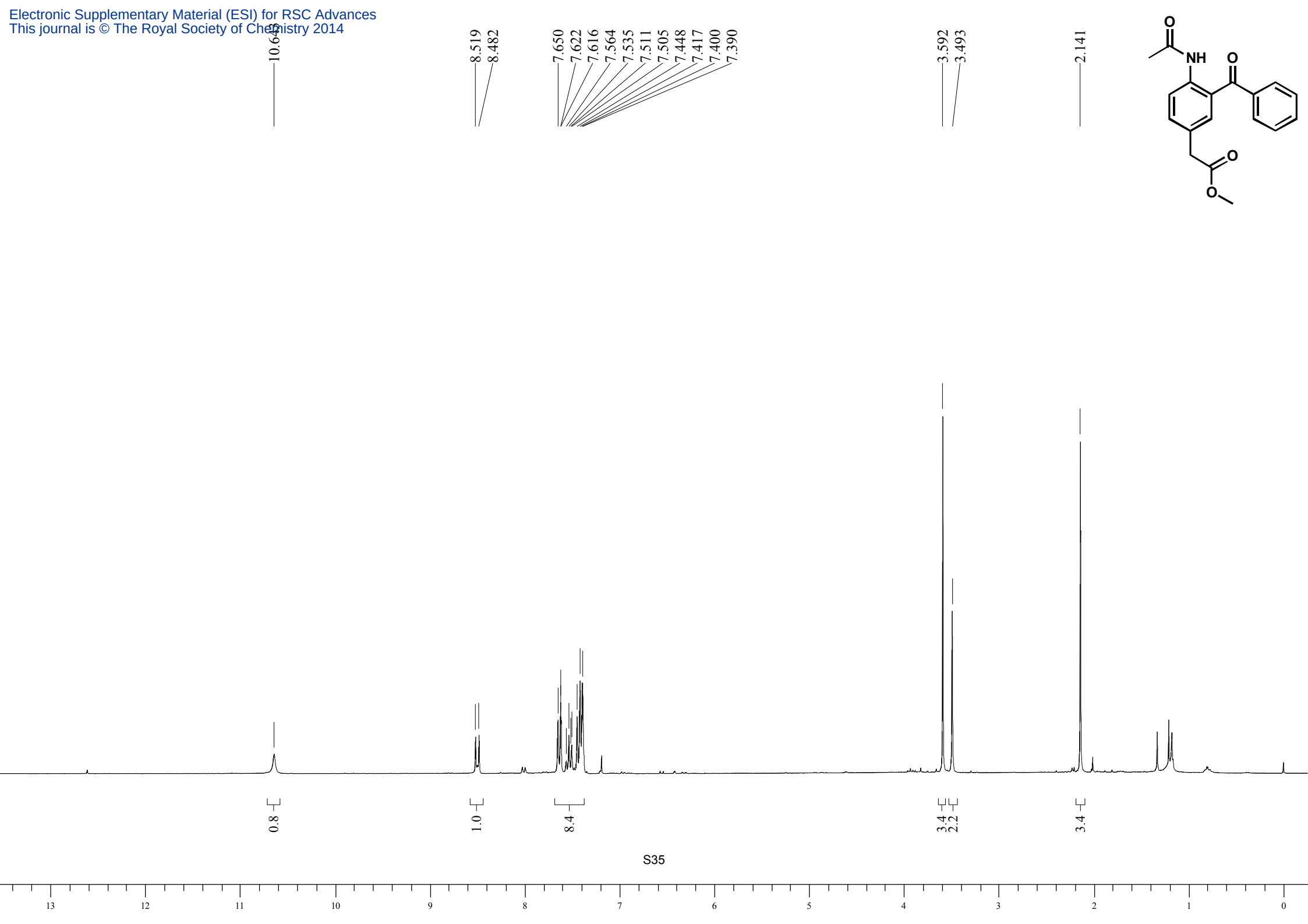

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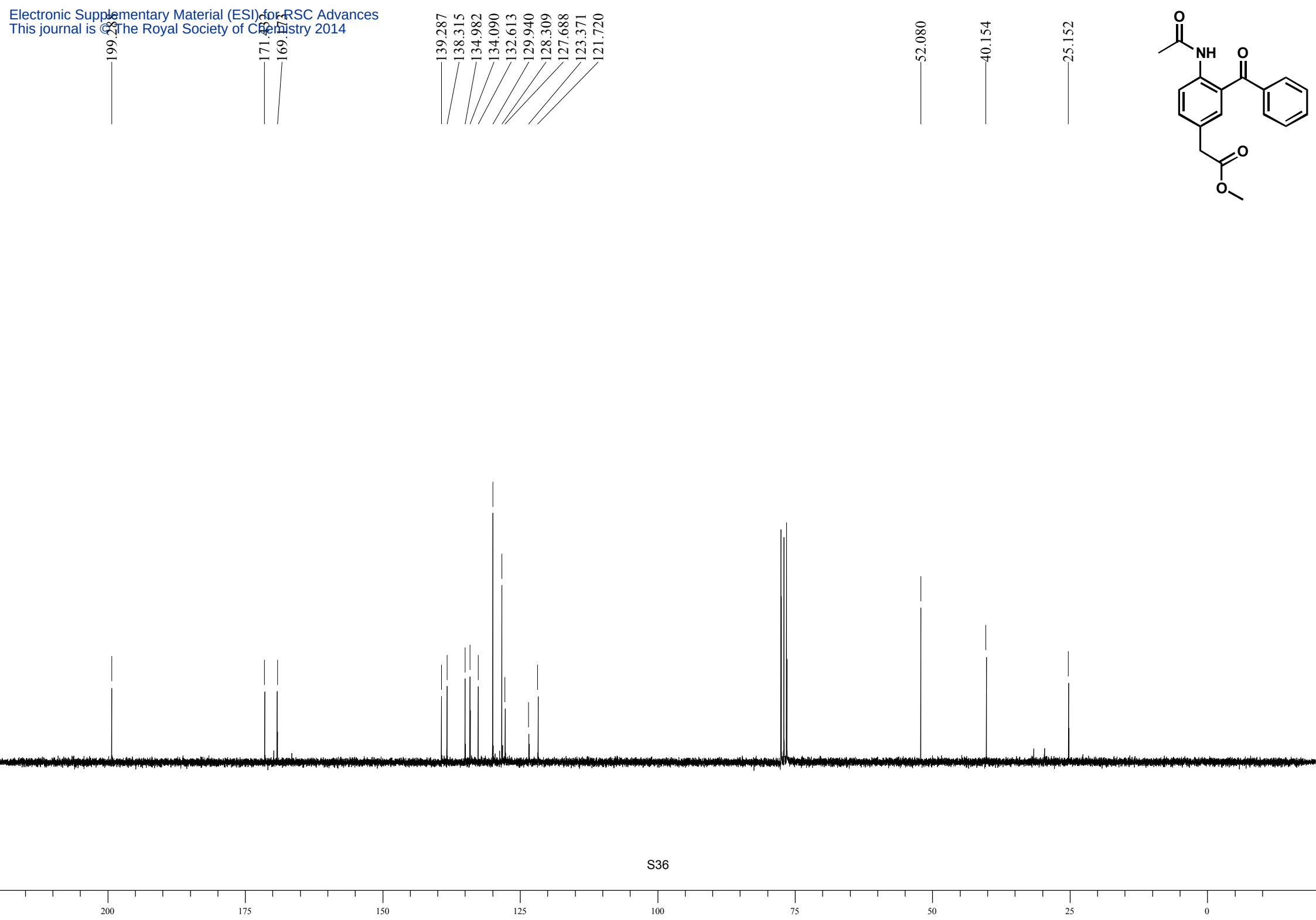

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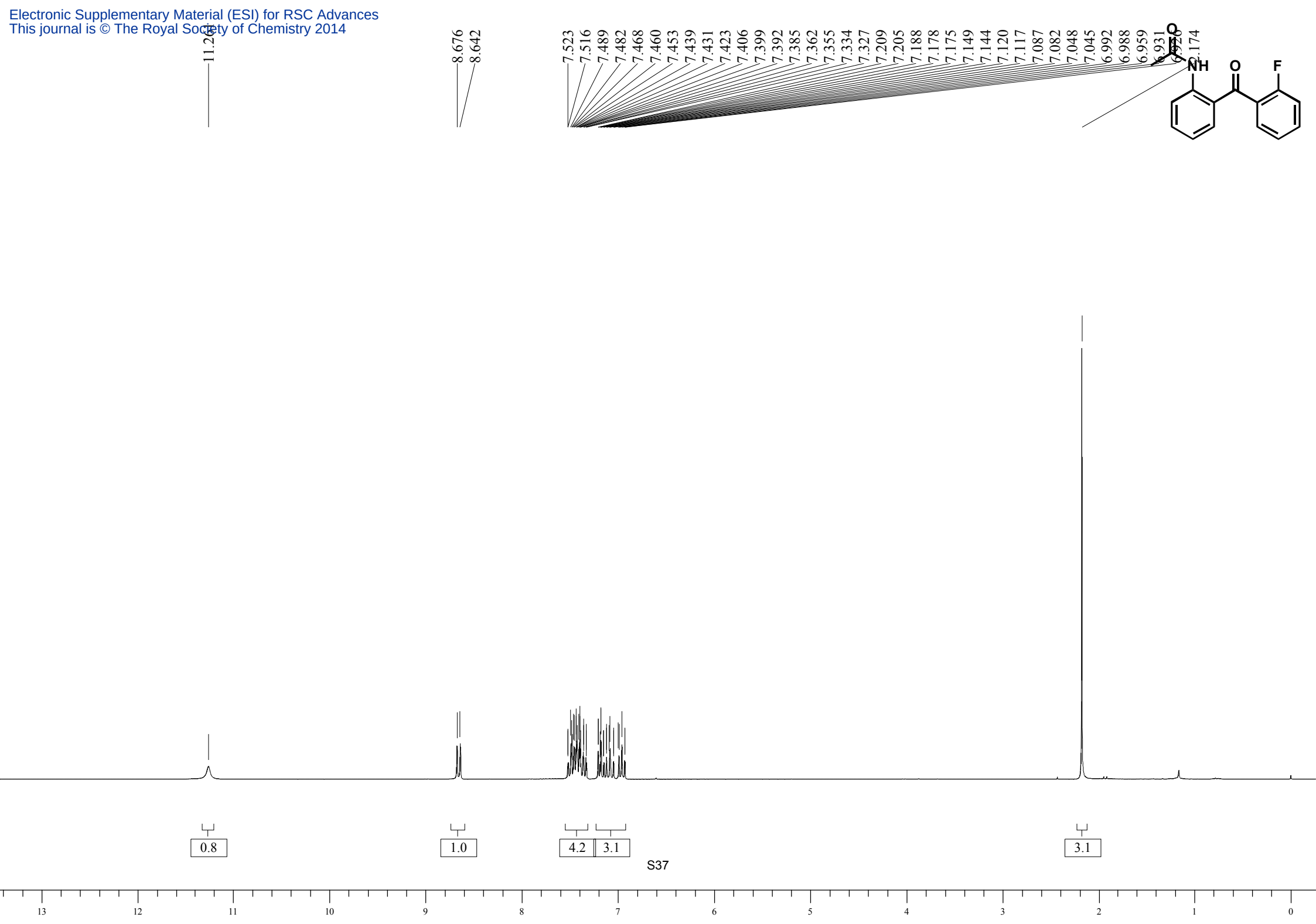

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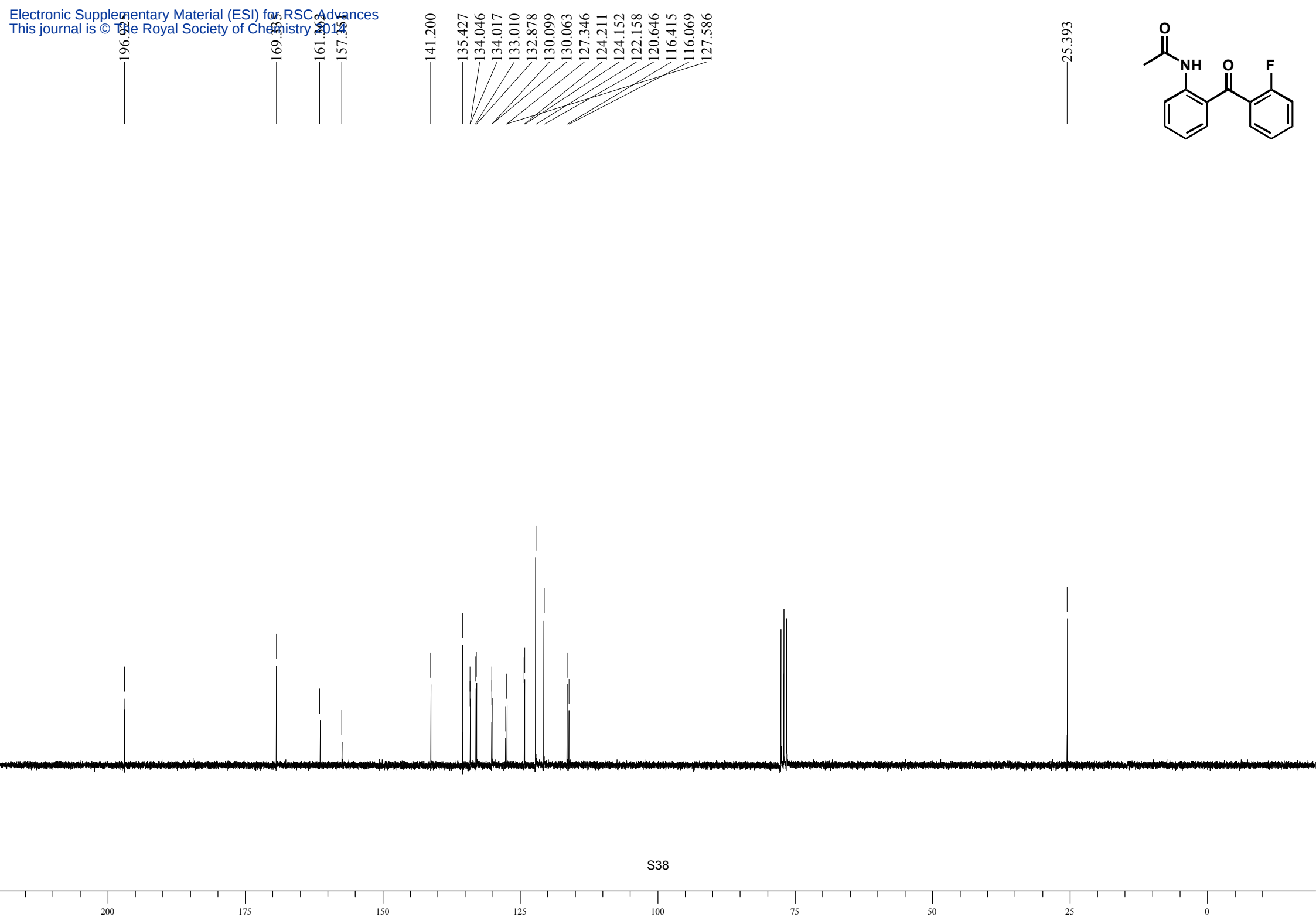

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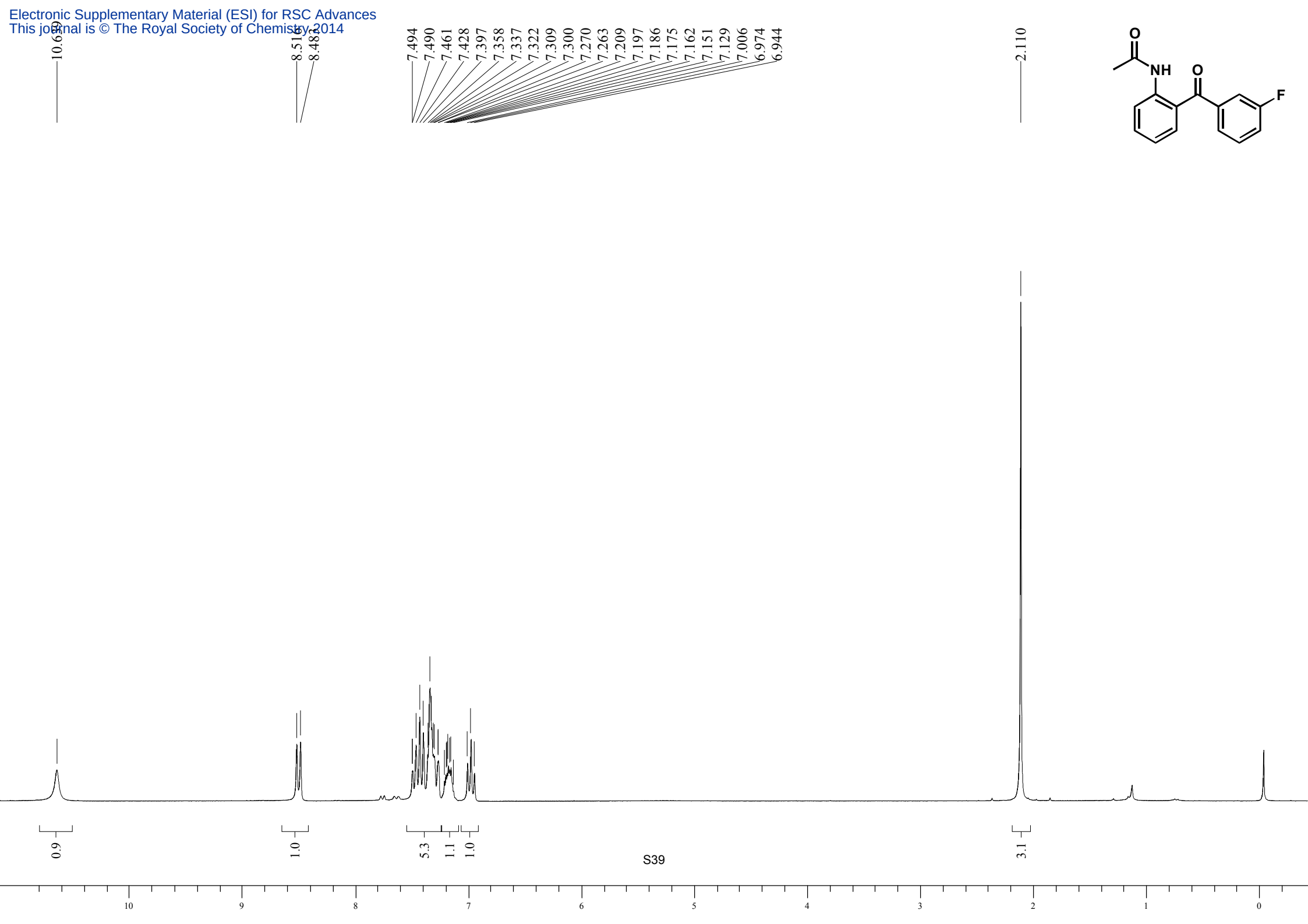

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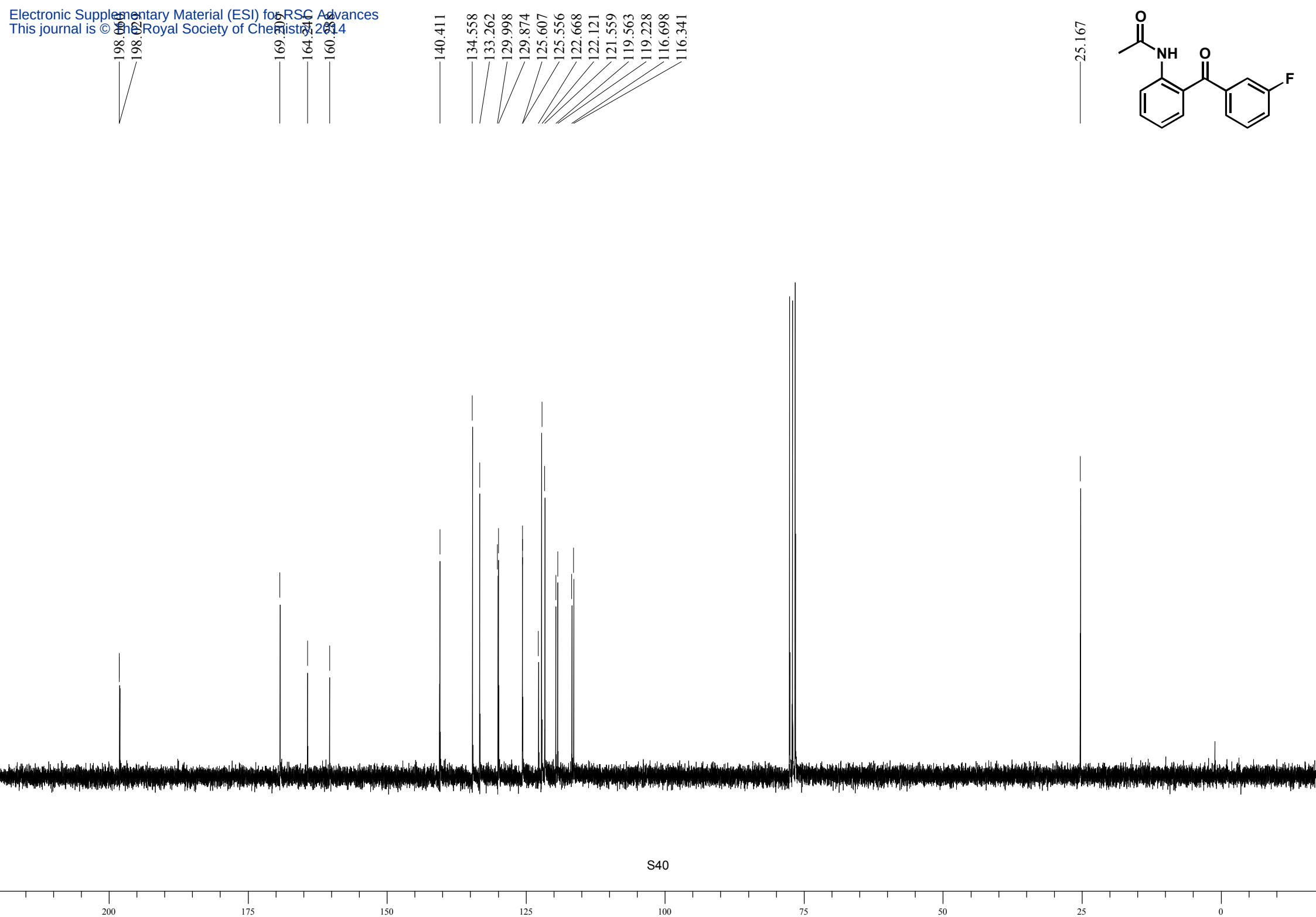

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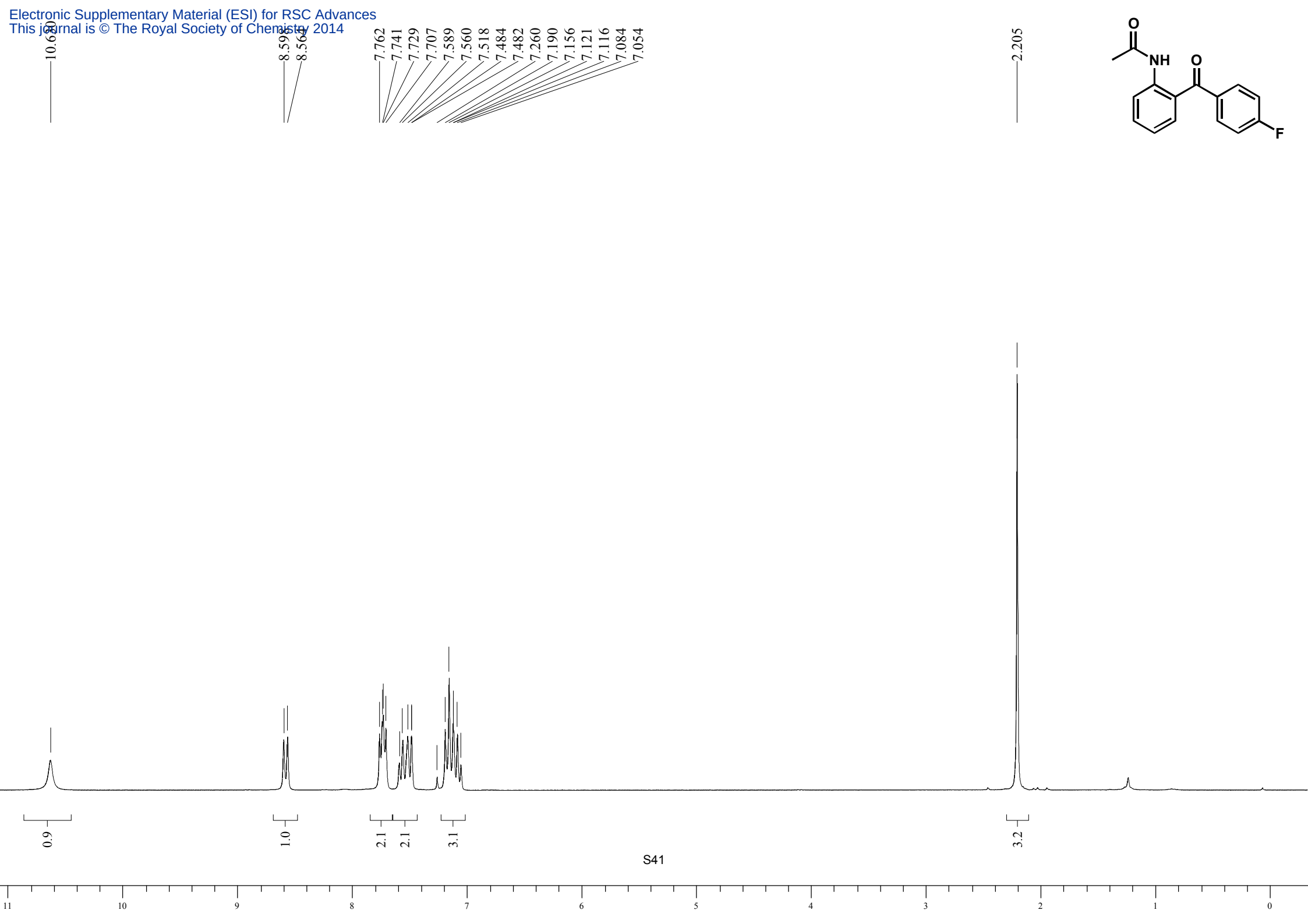

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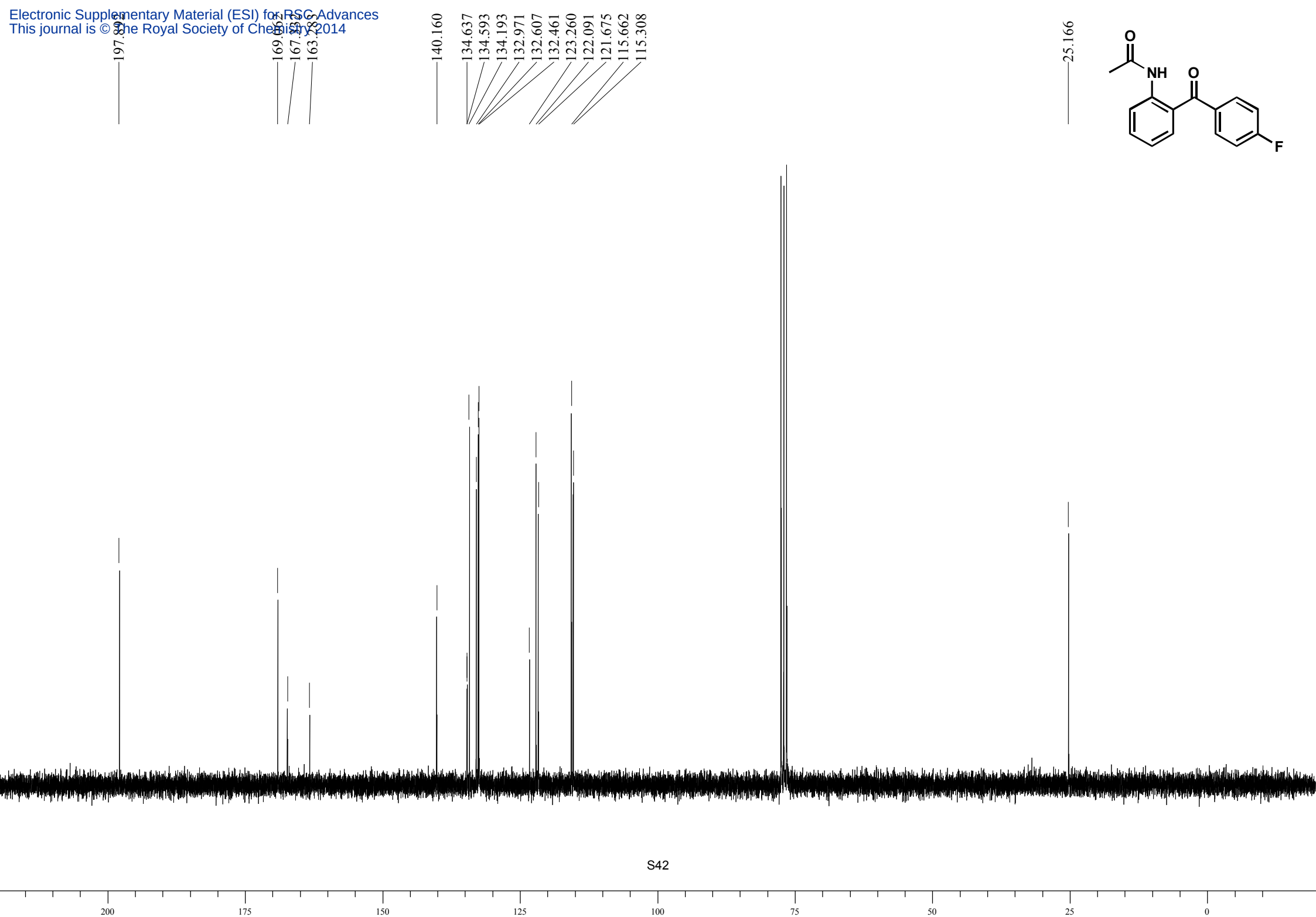

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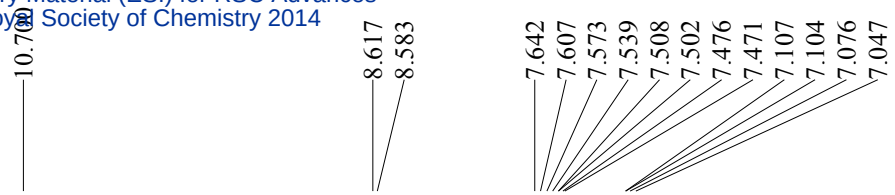

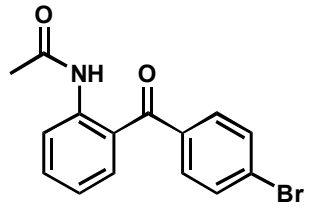

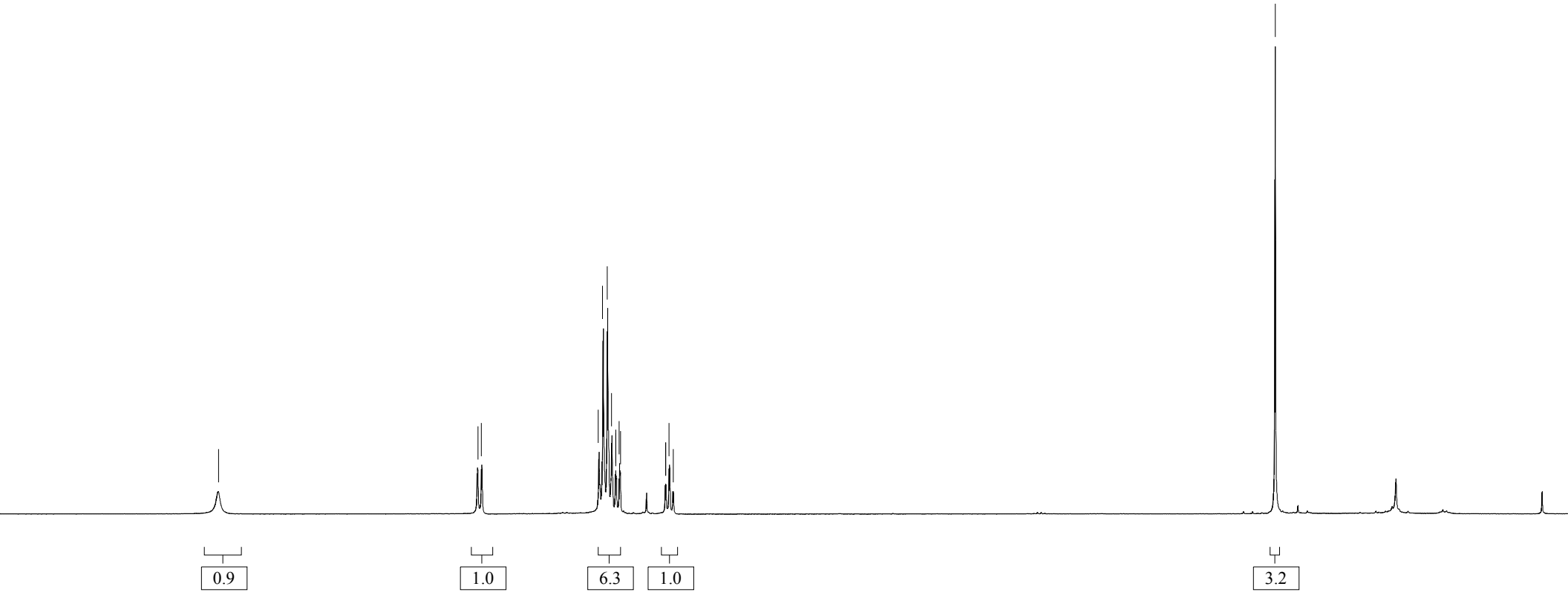

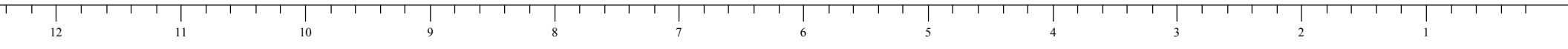

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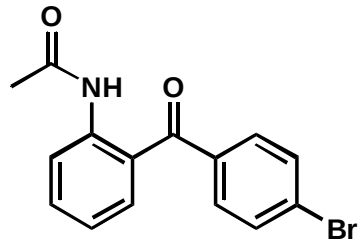

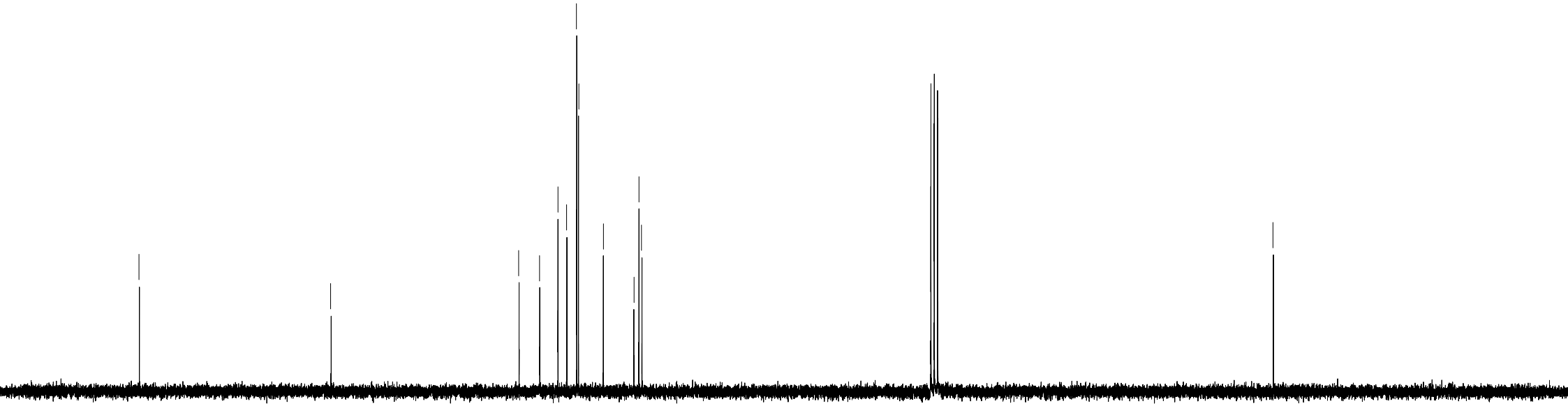

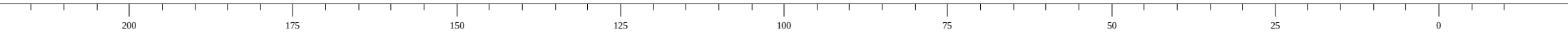

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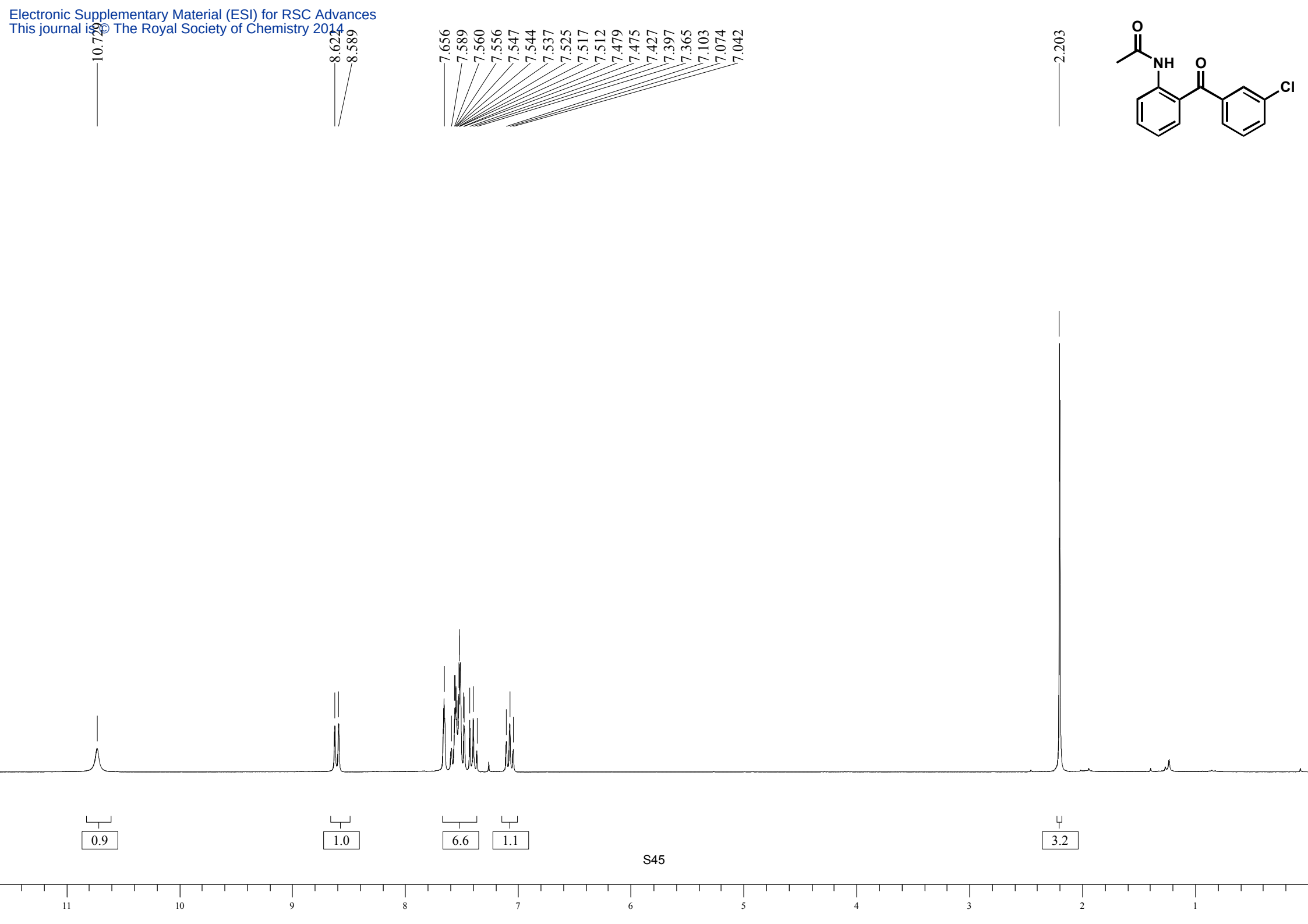

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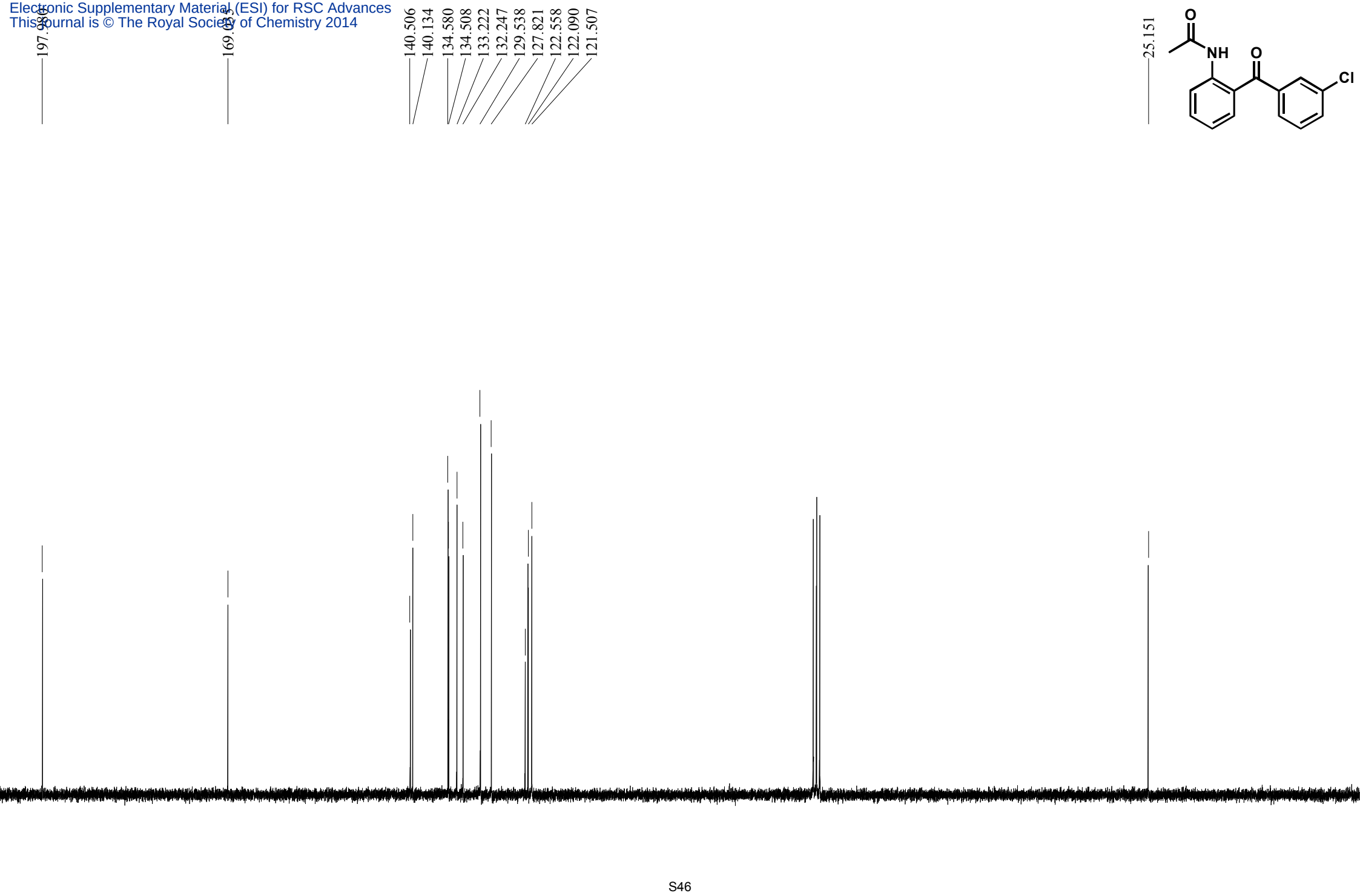

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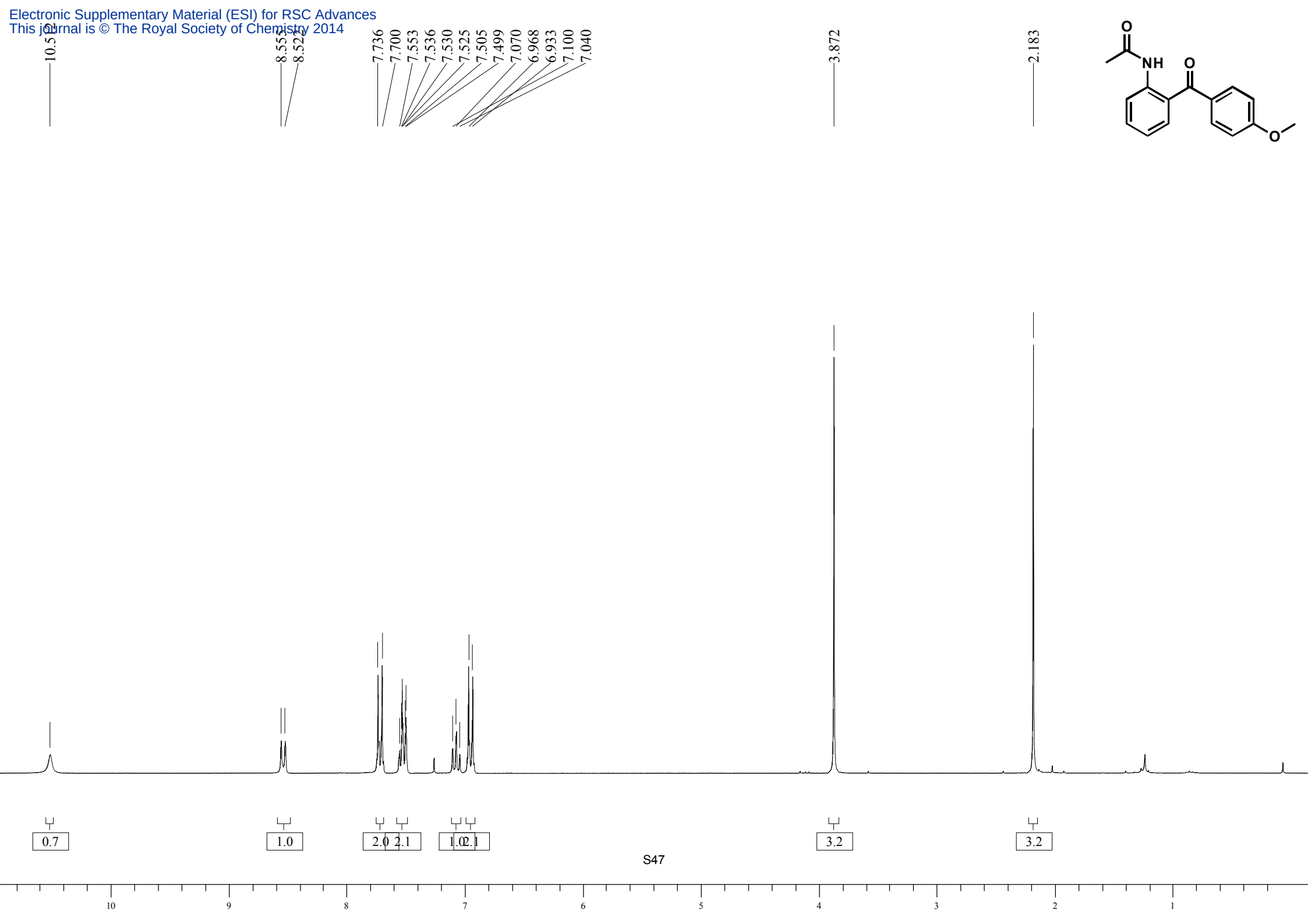

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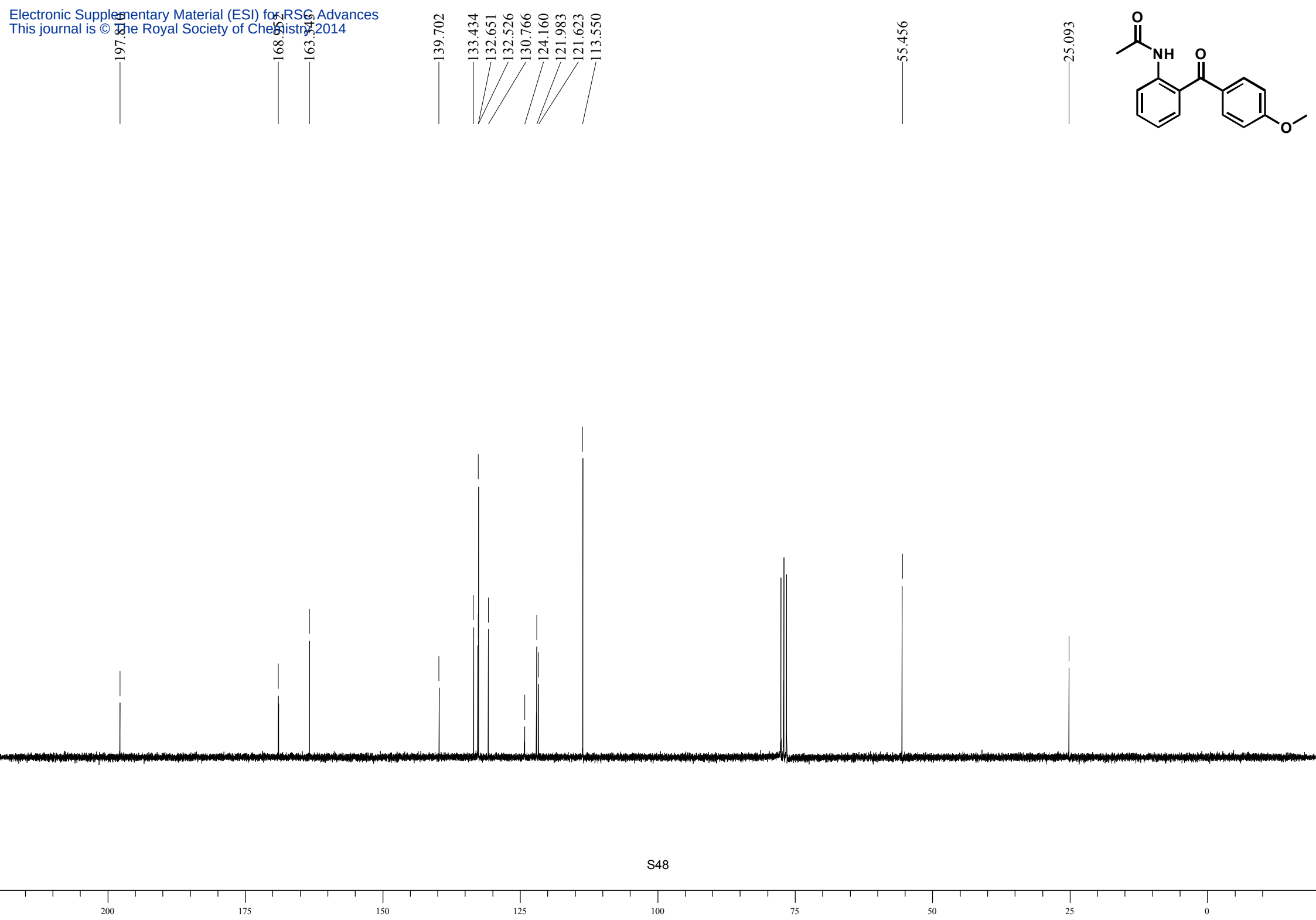

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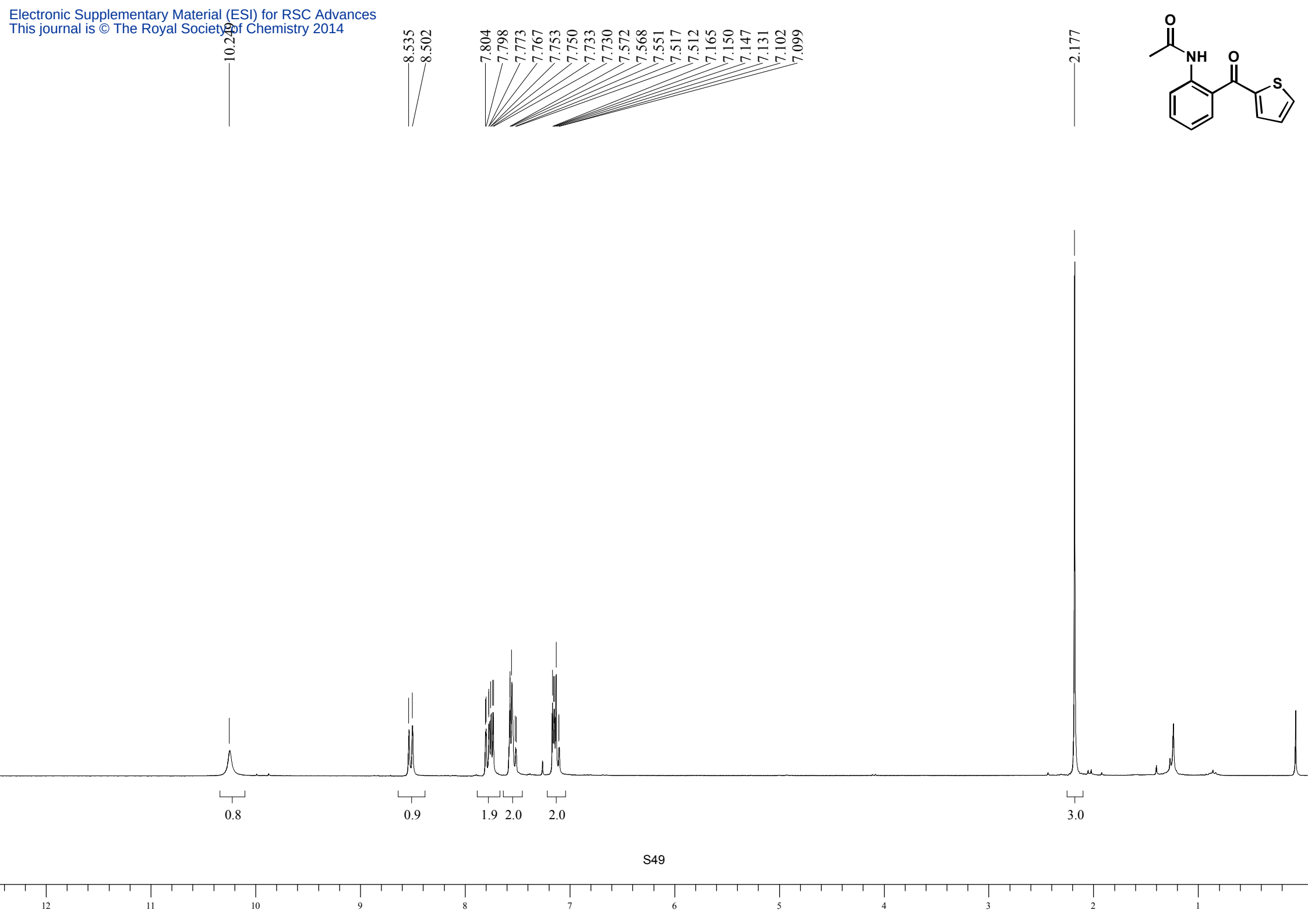

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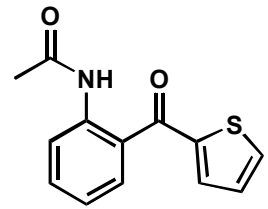

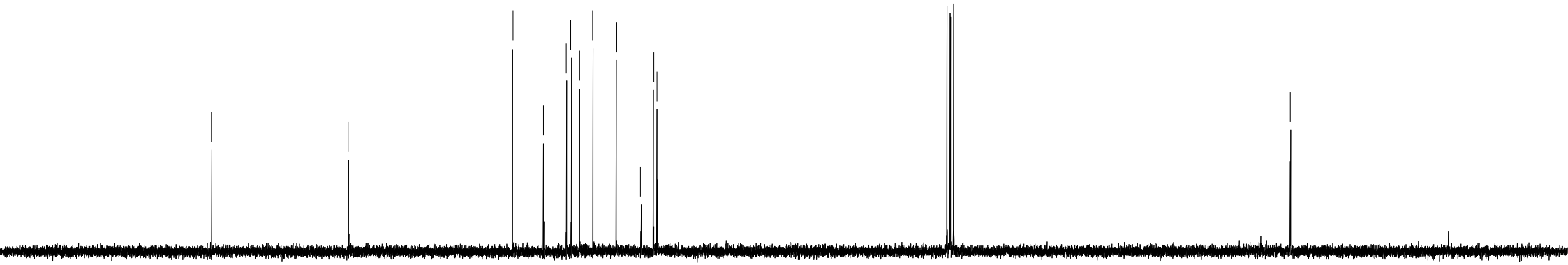

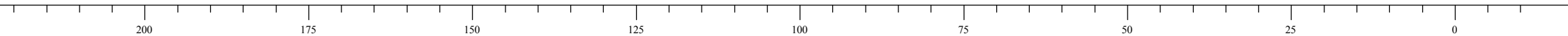

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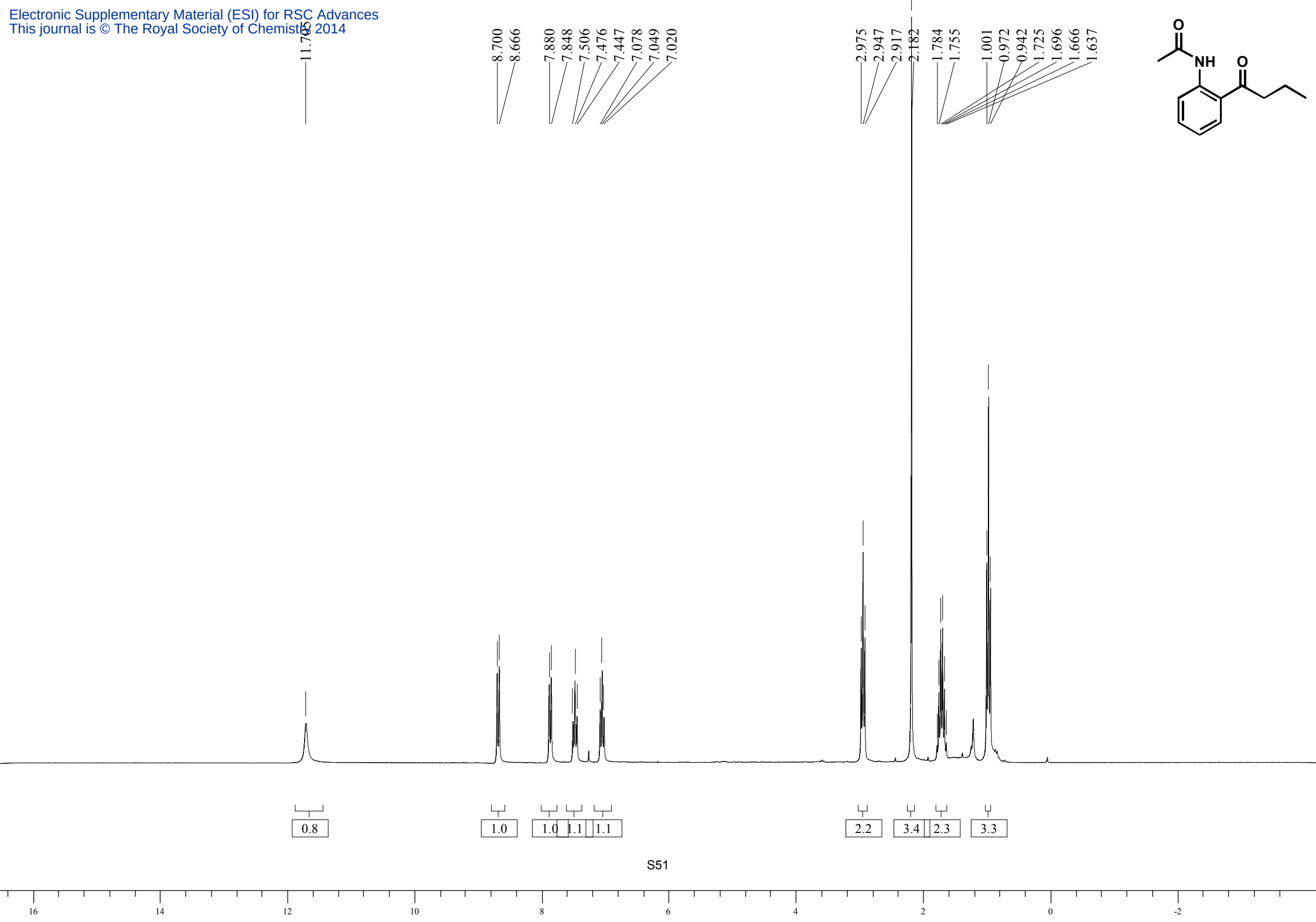

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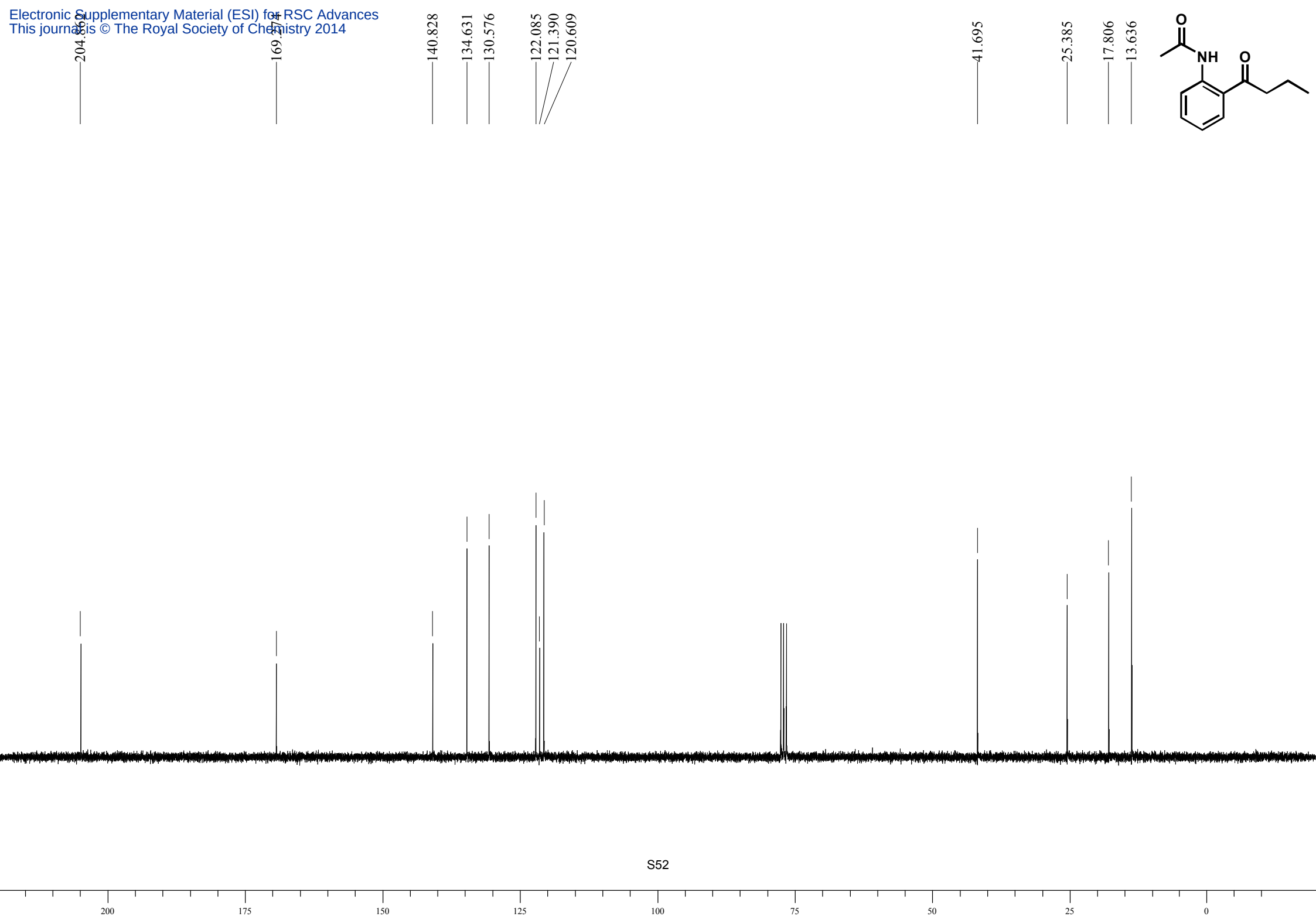

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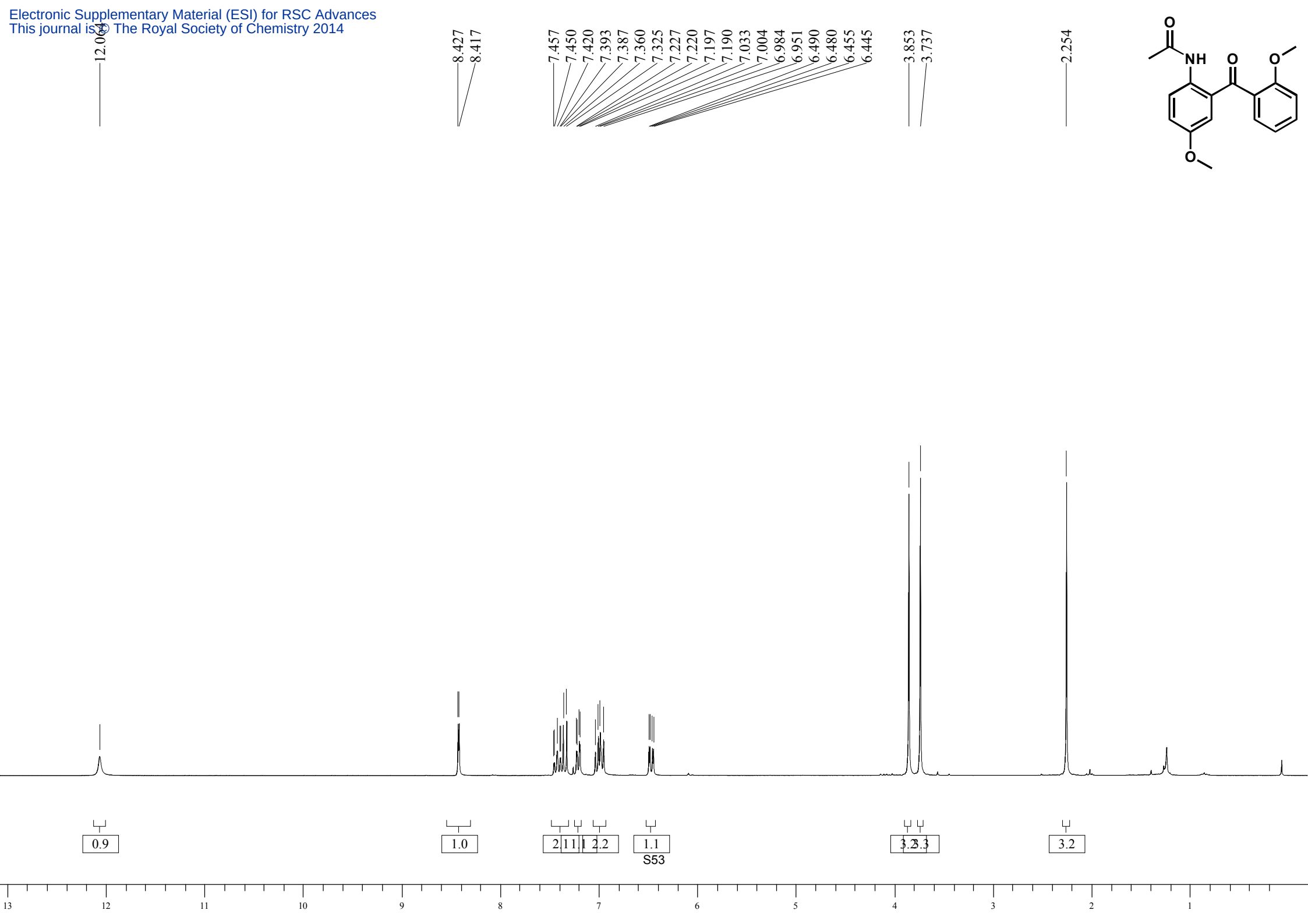

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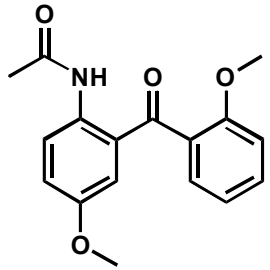

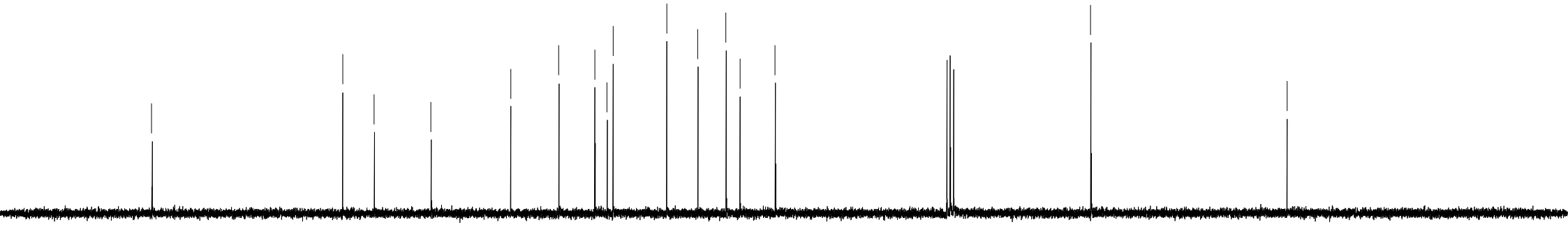

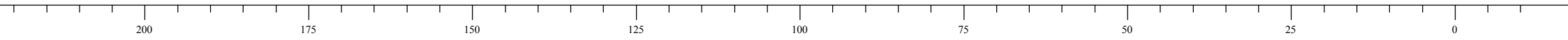

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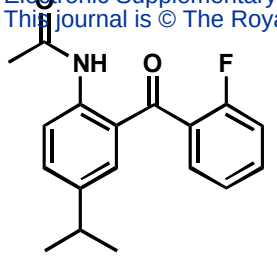

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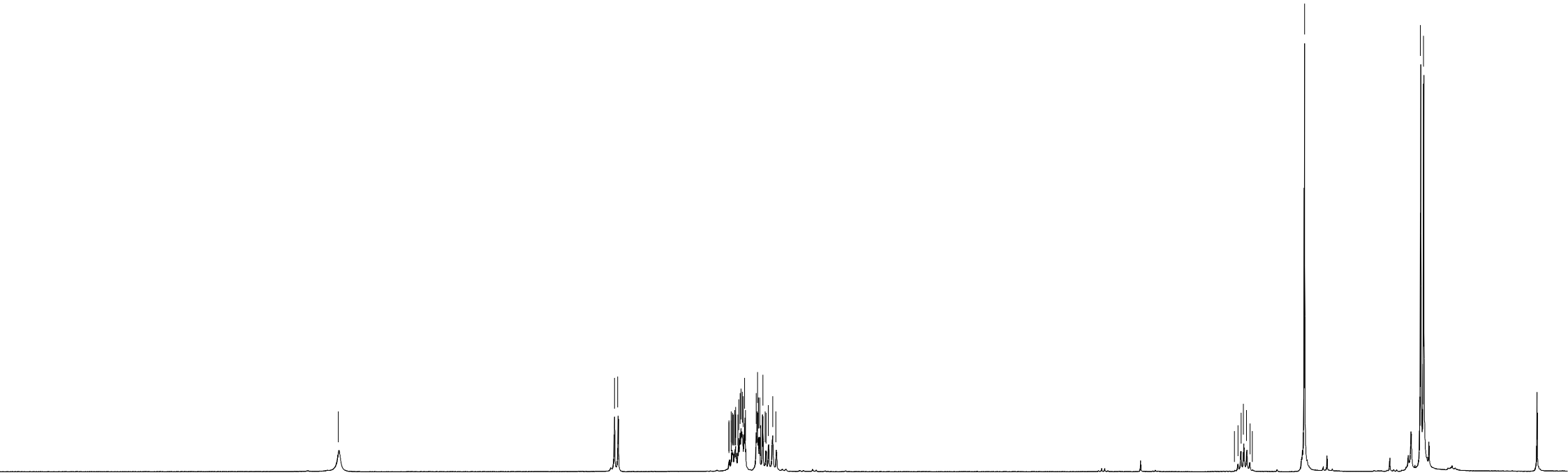

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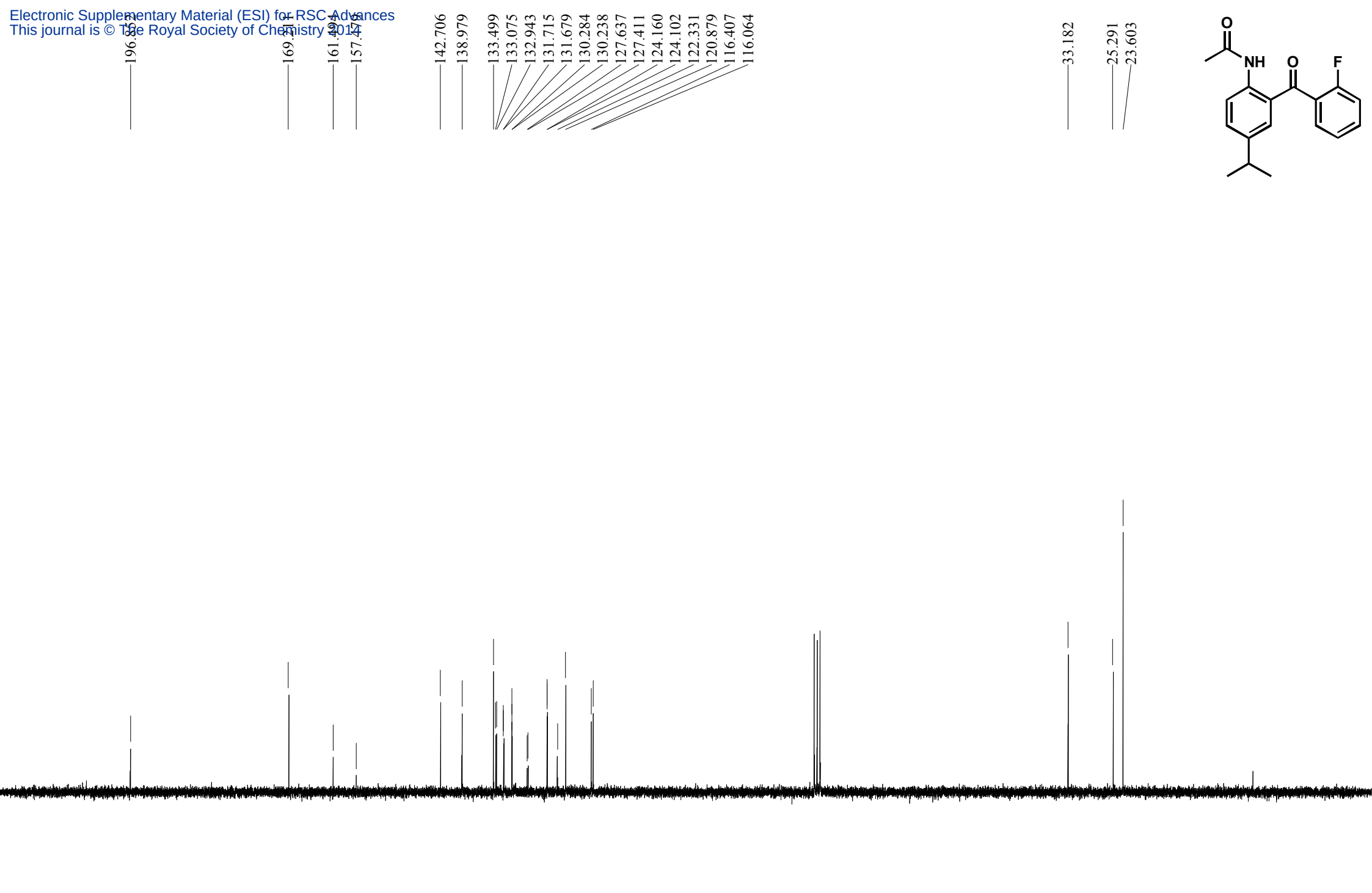

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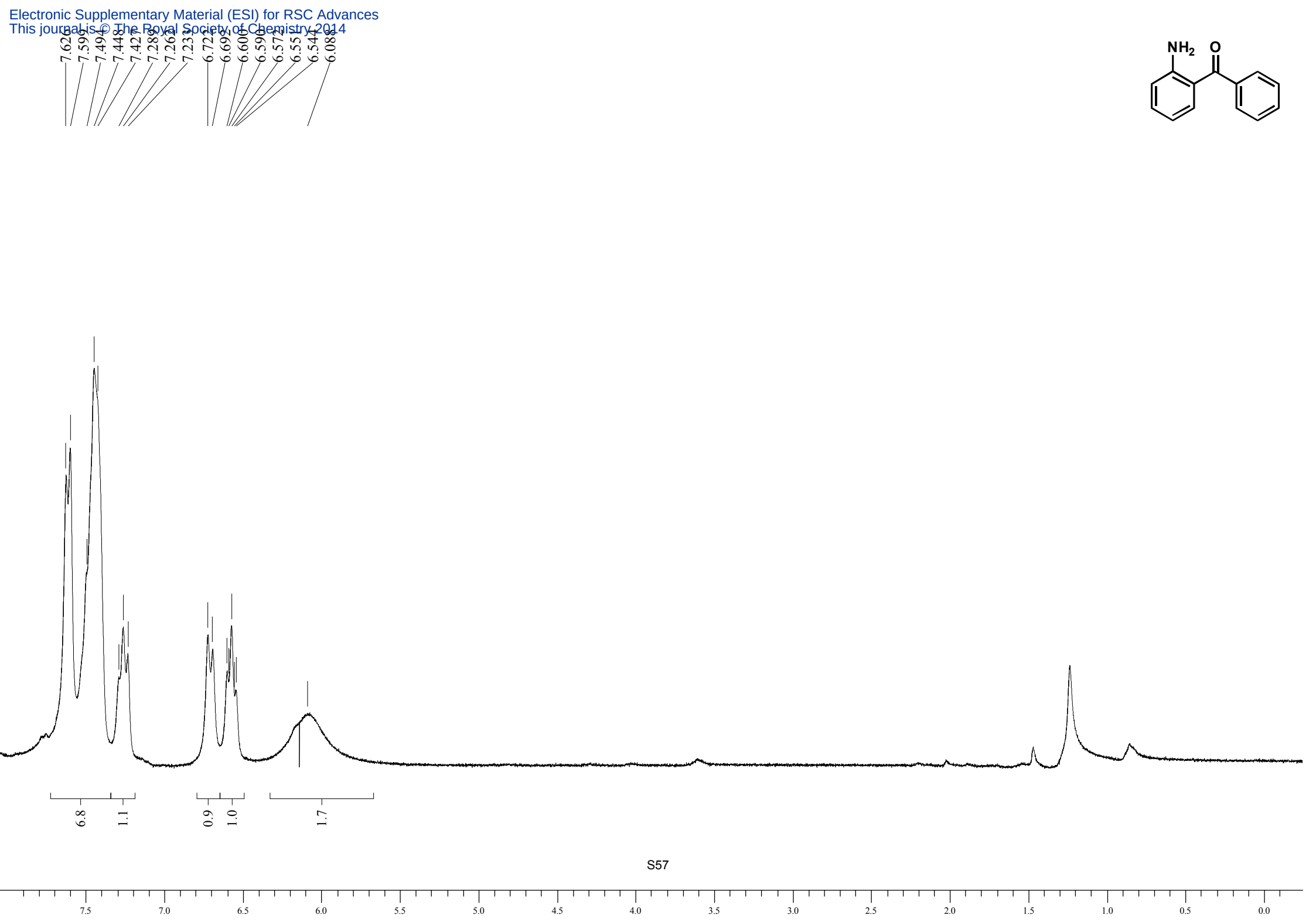

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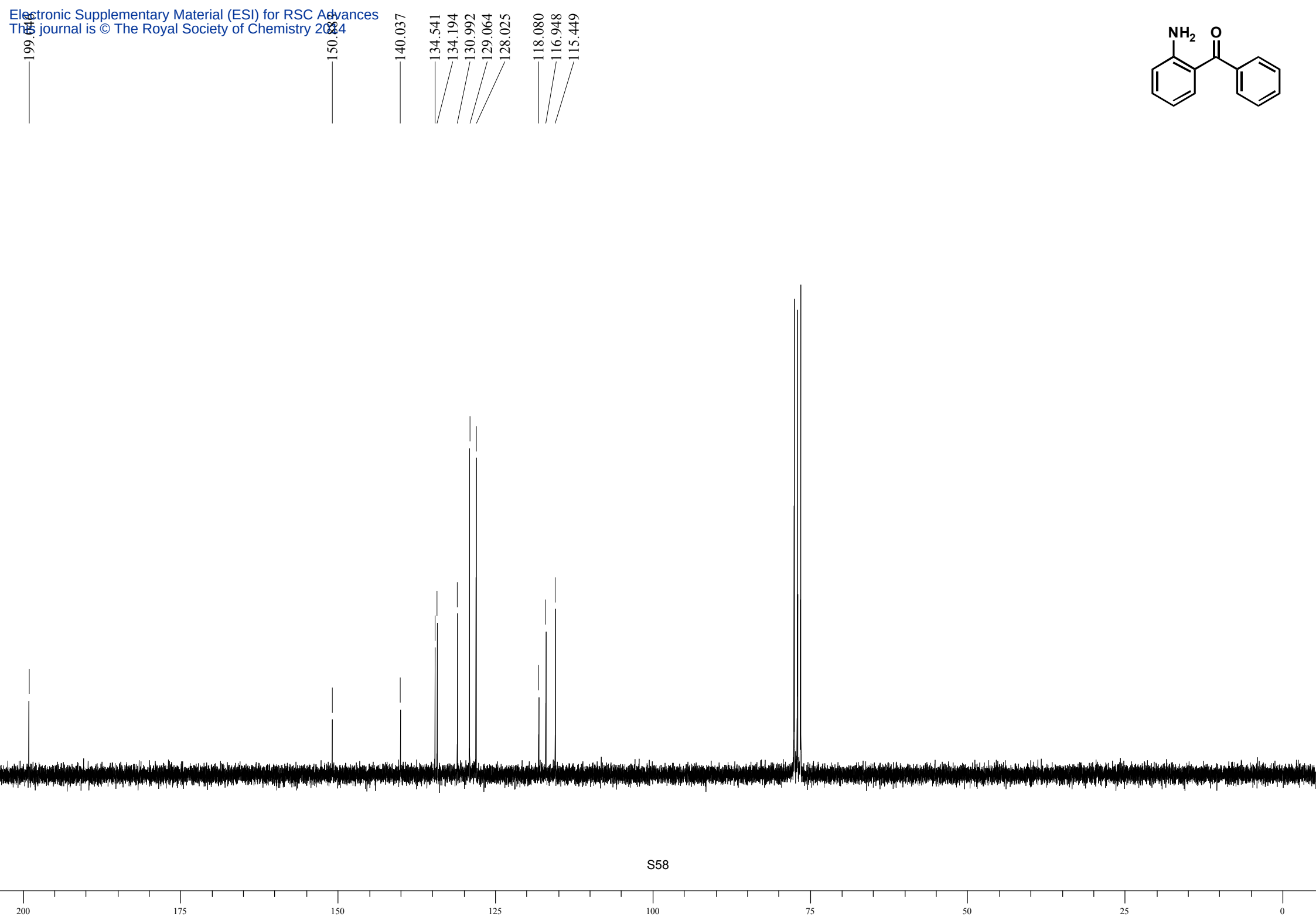

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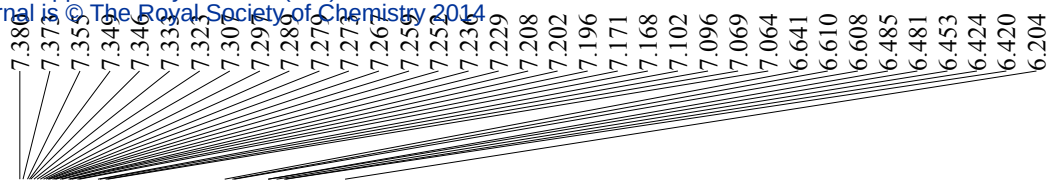

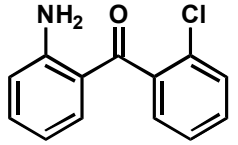

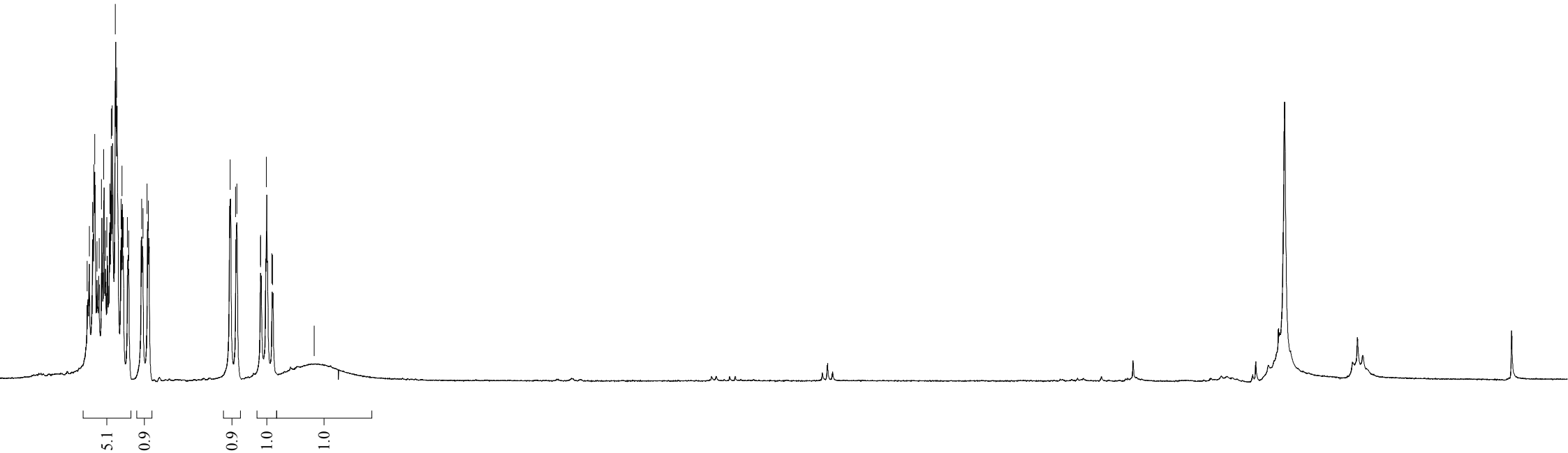

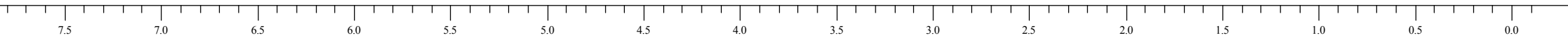

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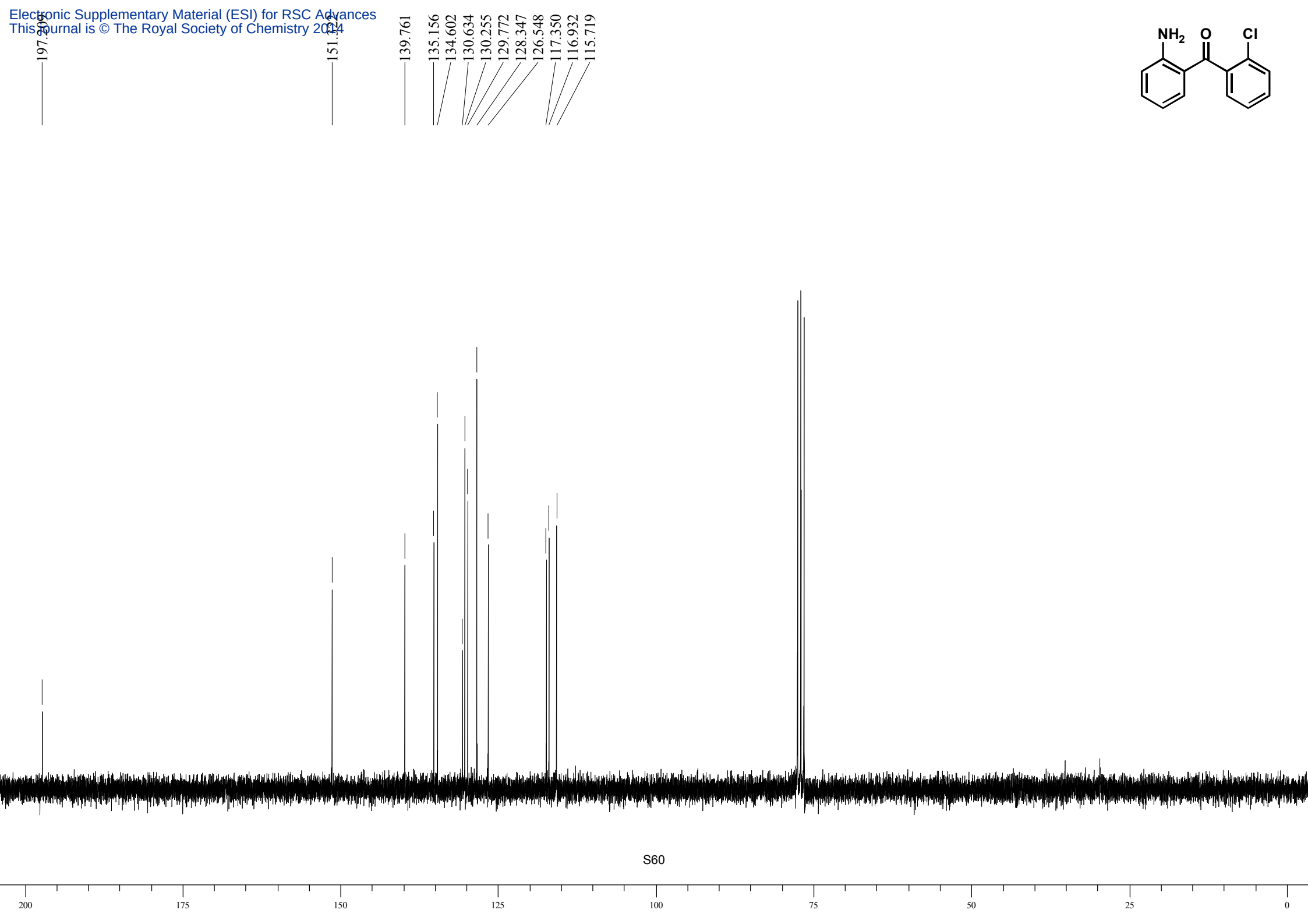

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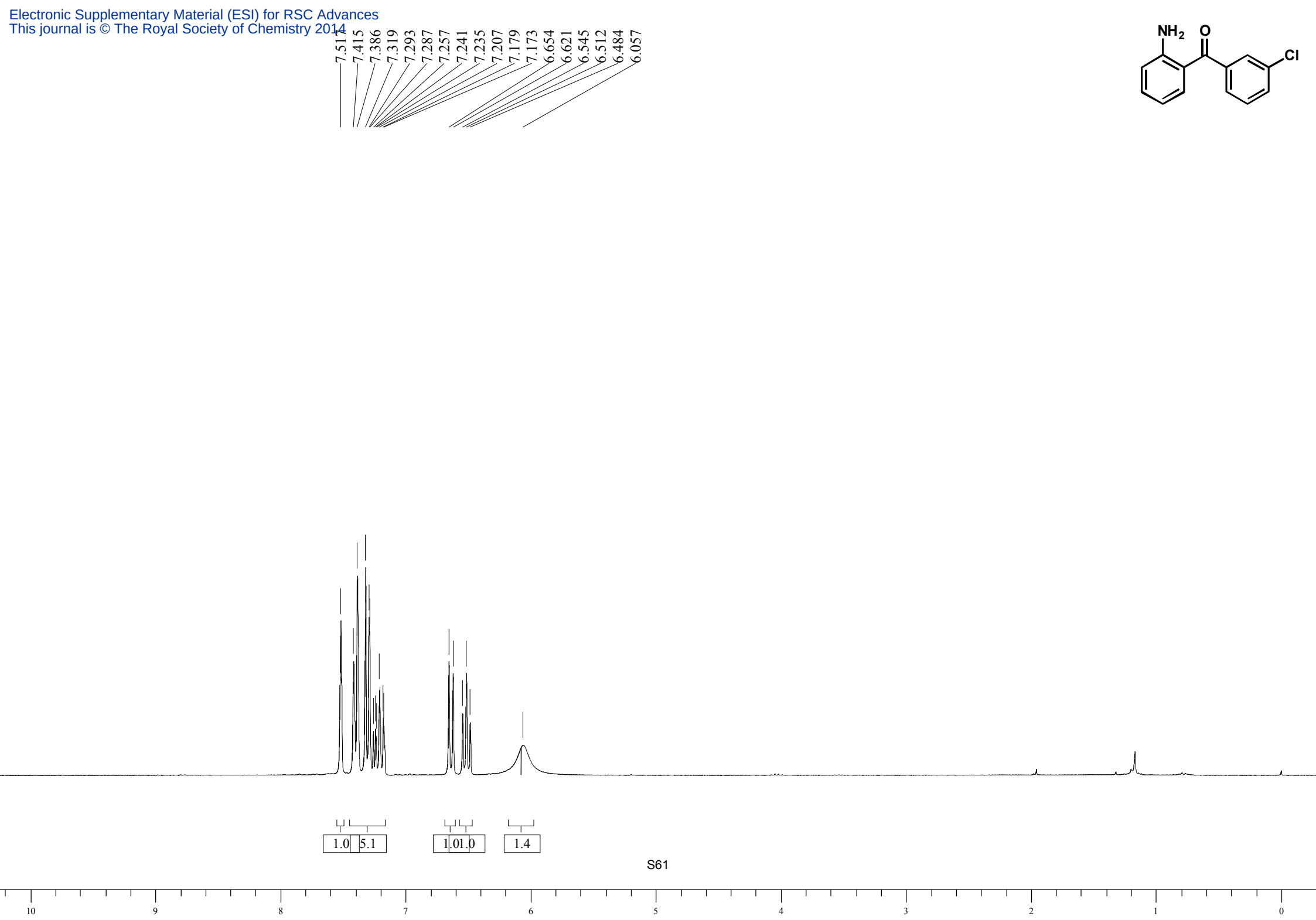

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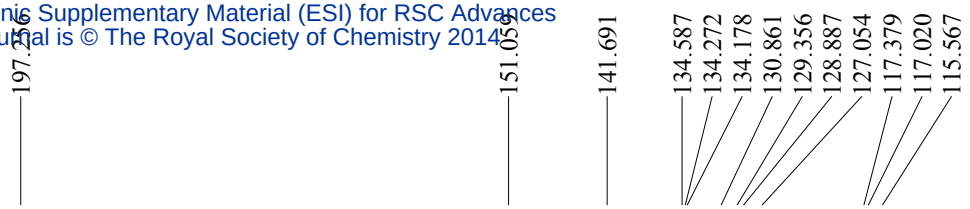

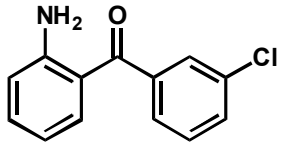

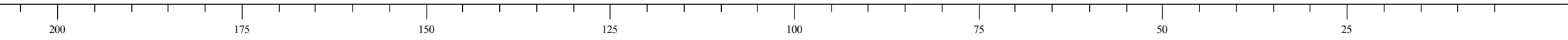

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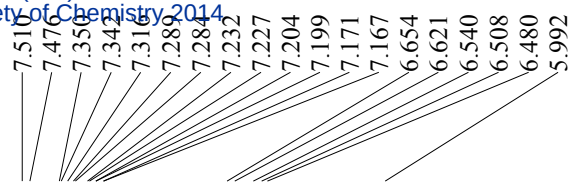

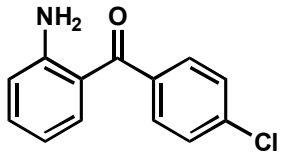

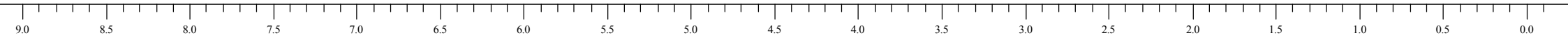

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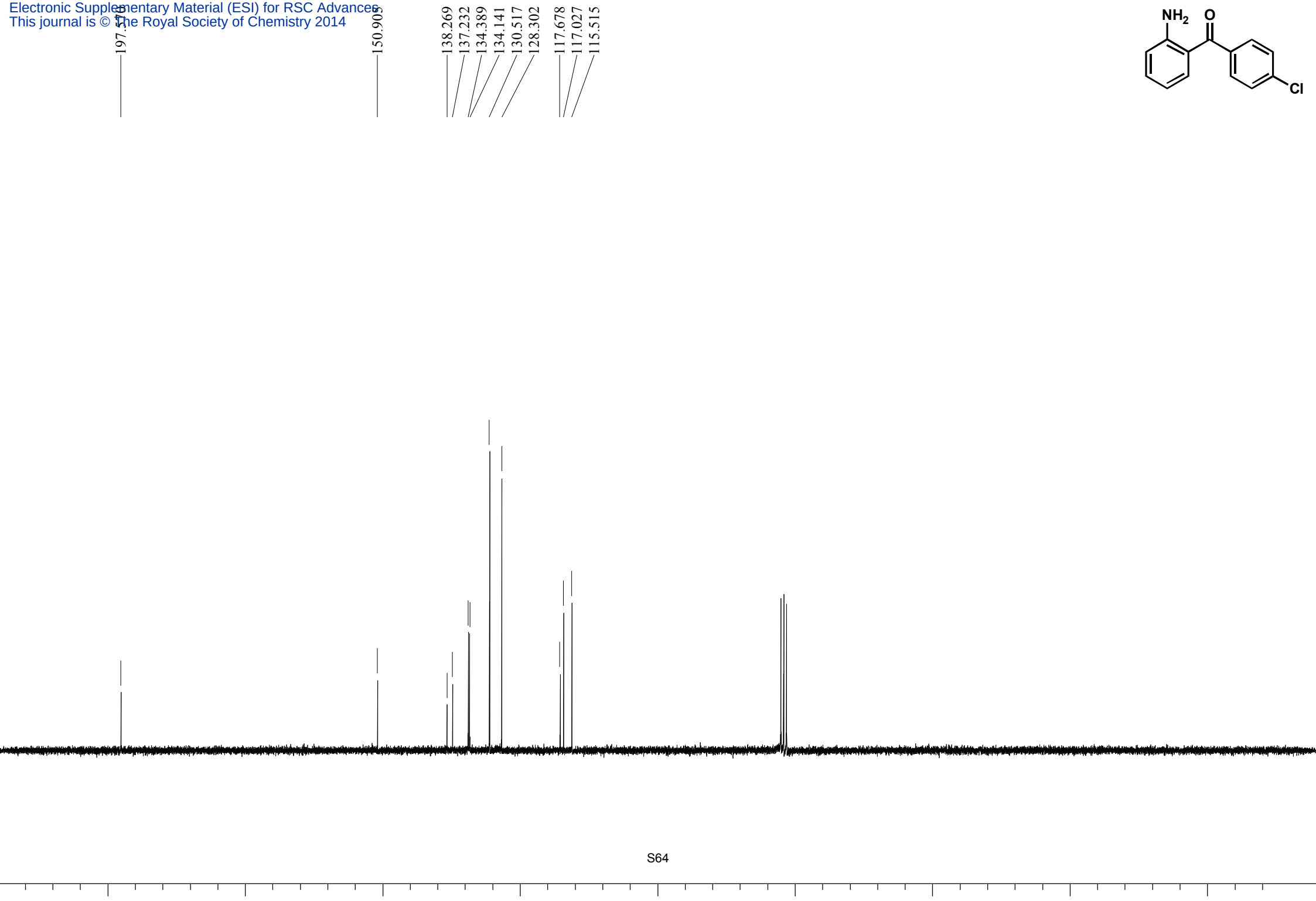

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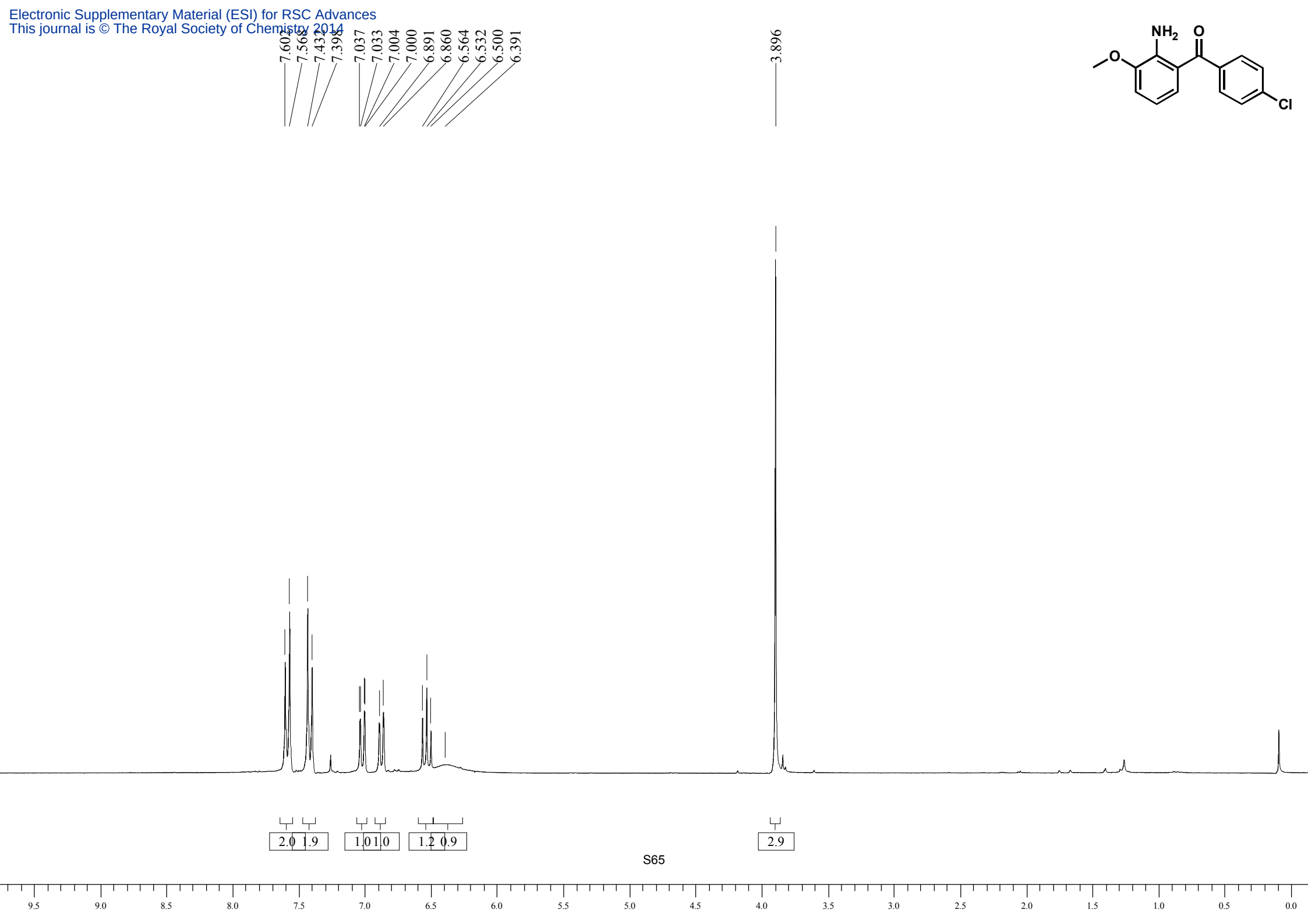

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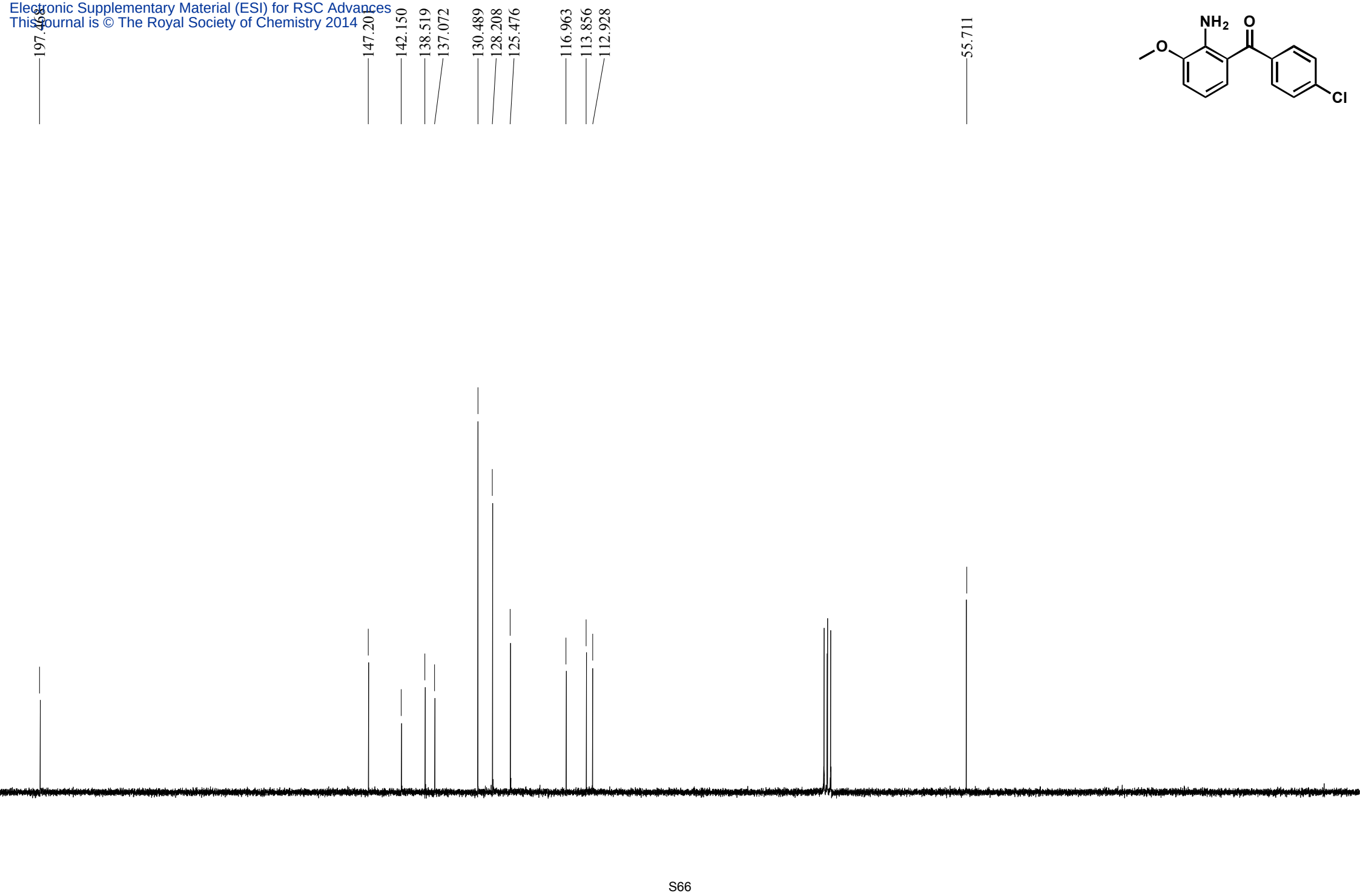

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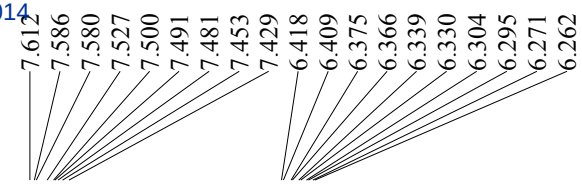

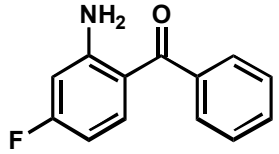

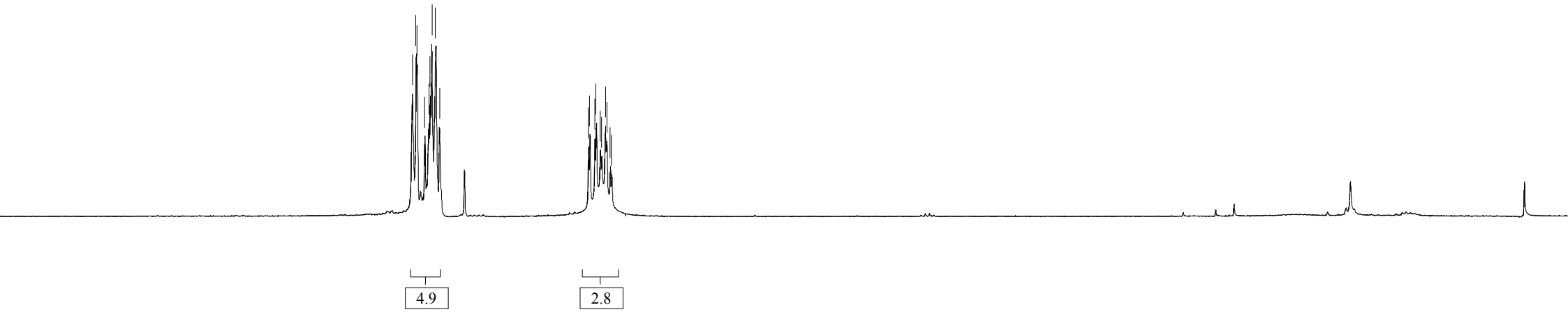

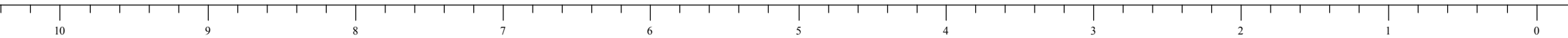

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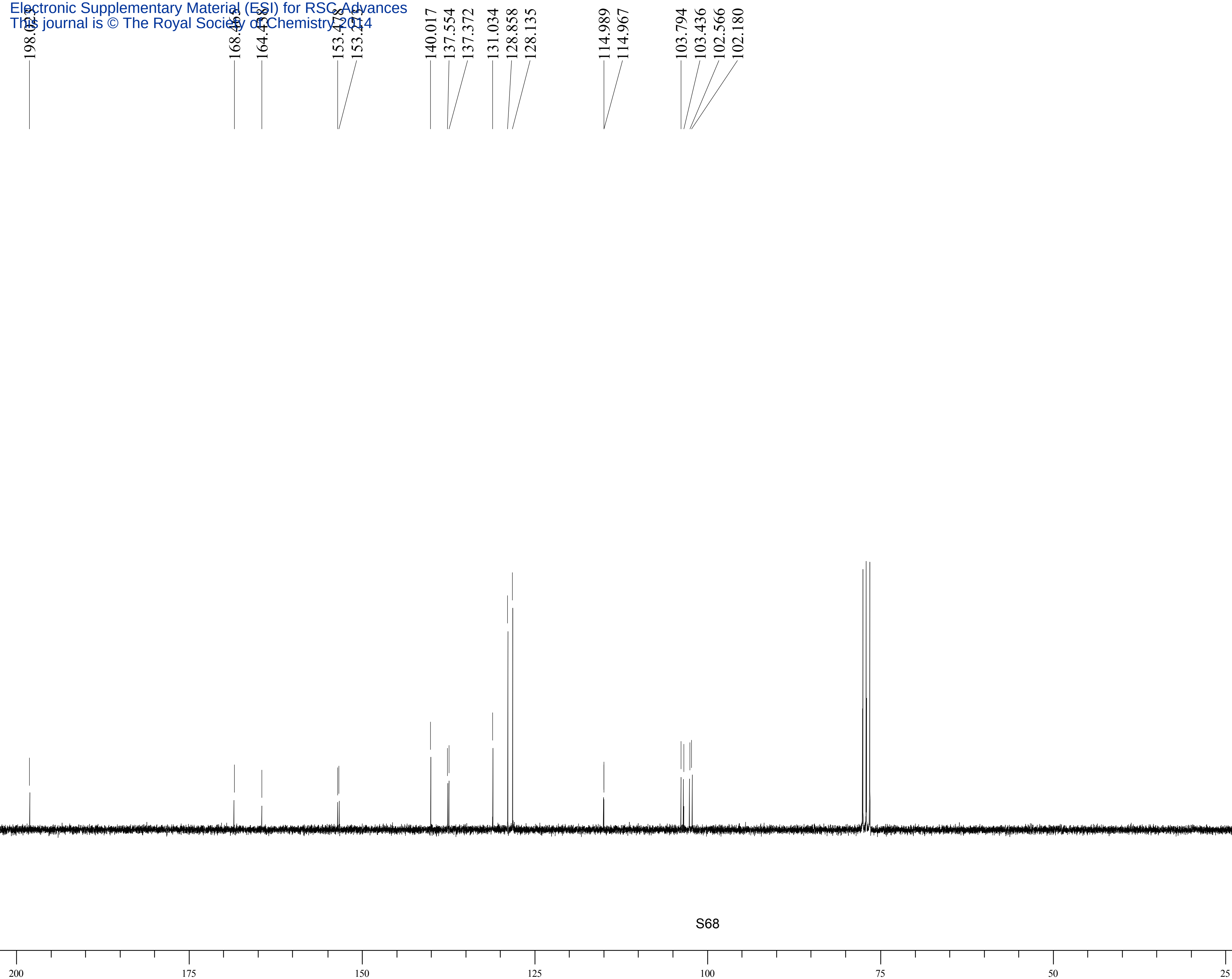

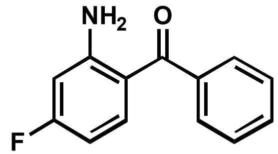

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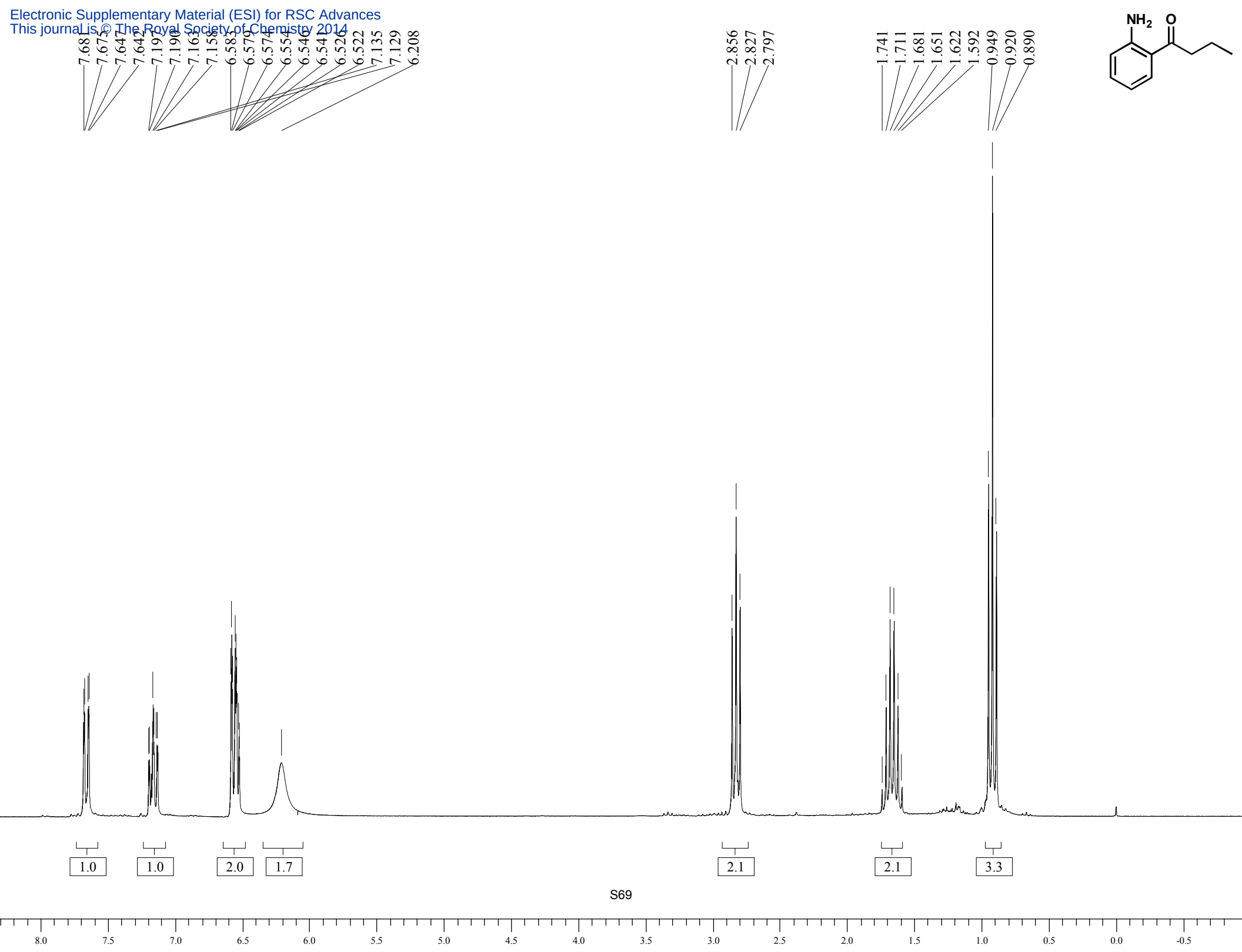

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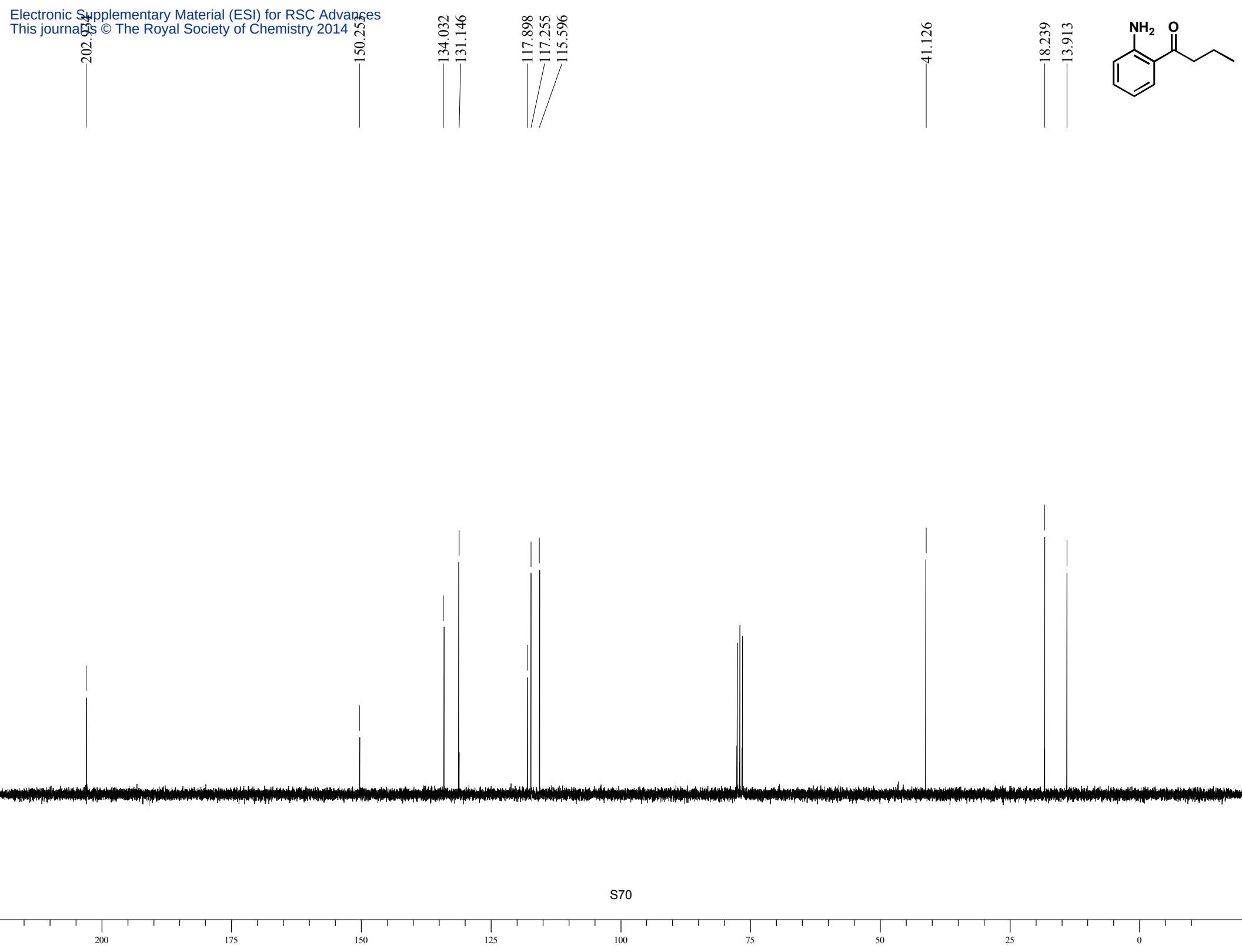

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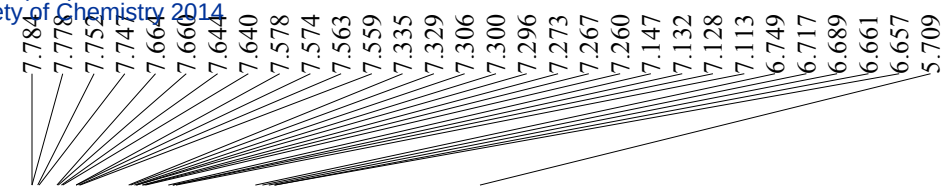

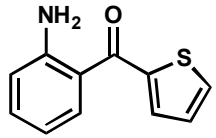

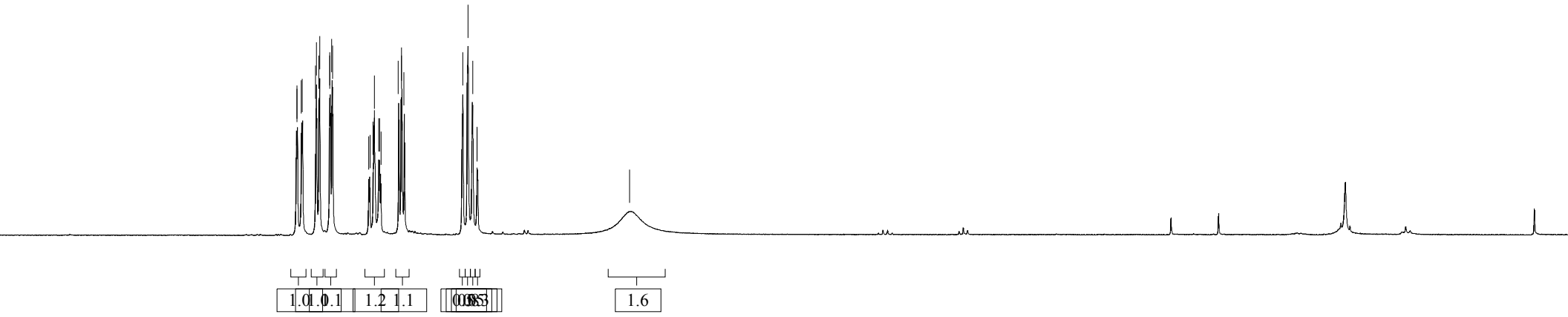

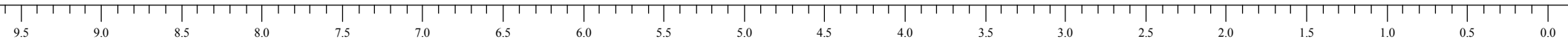

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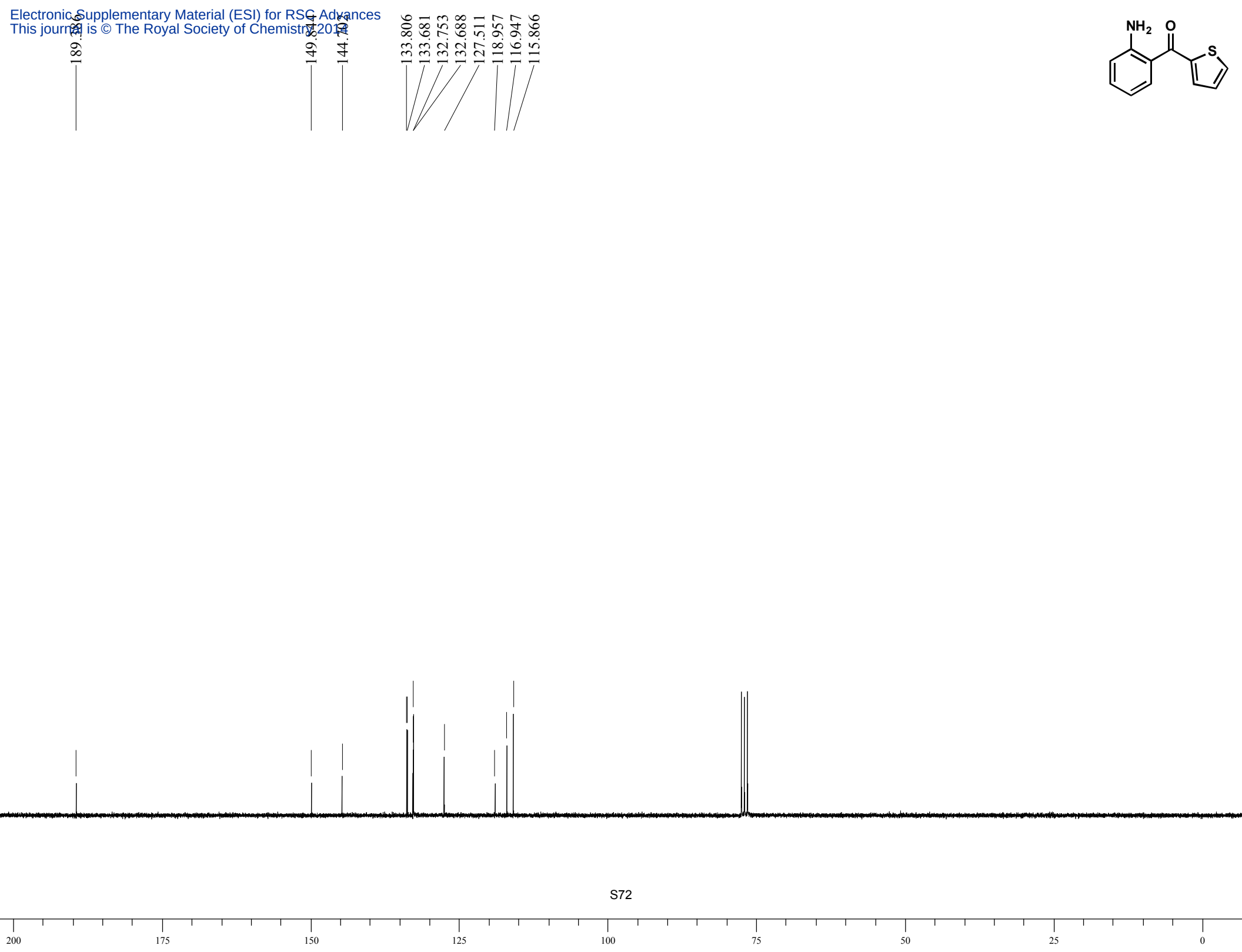

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GC-MS chromatograms

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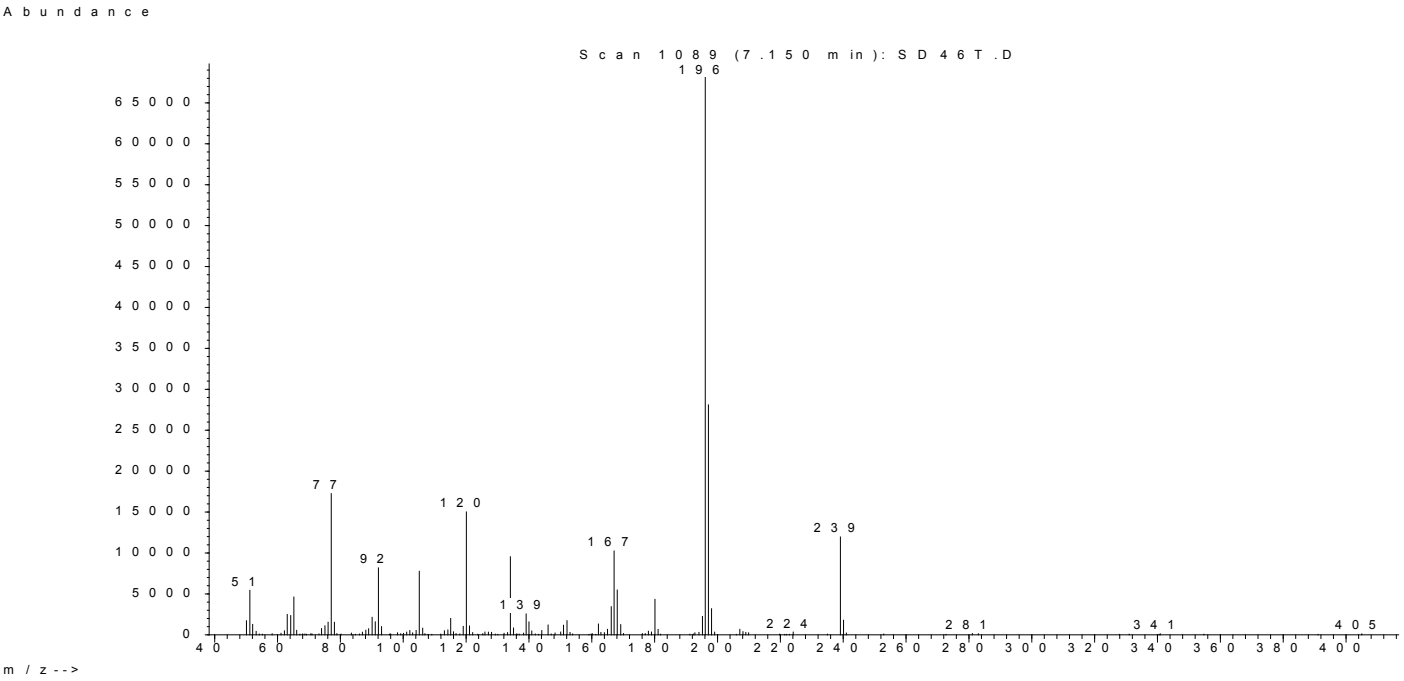
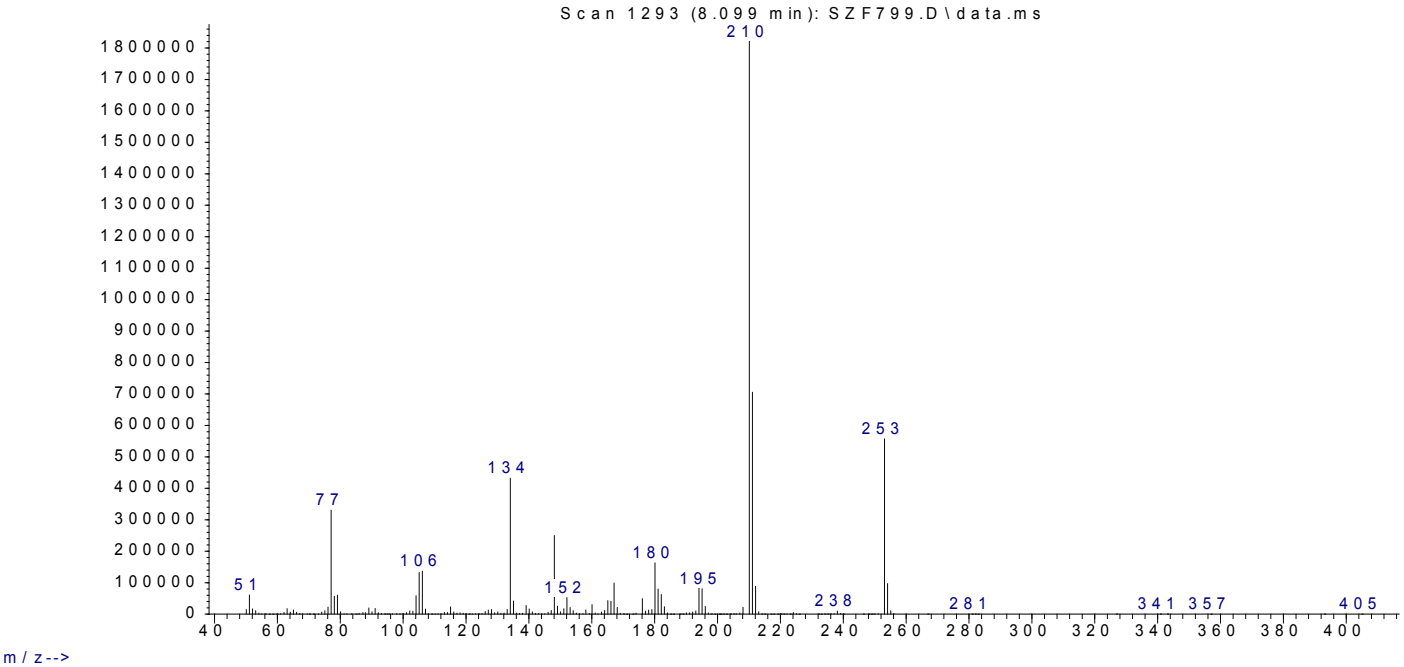


N-(2-benzoyl-5-methylphenyl)acetamide (4b)



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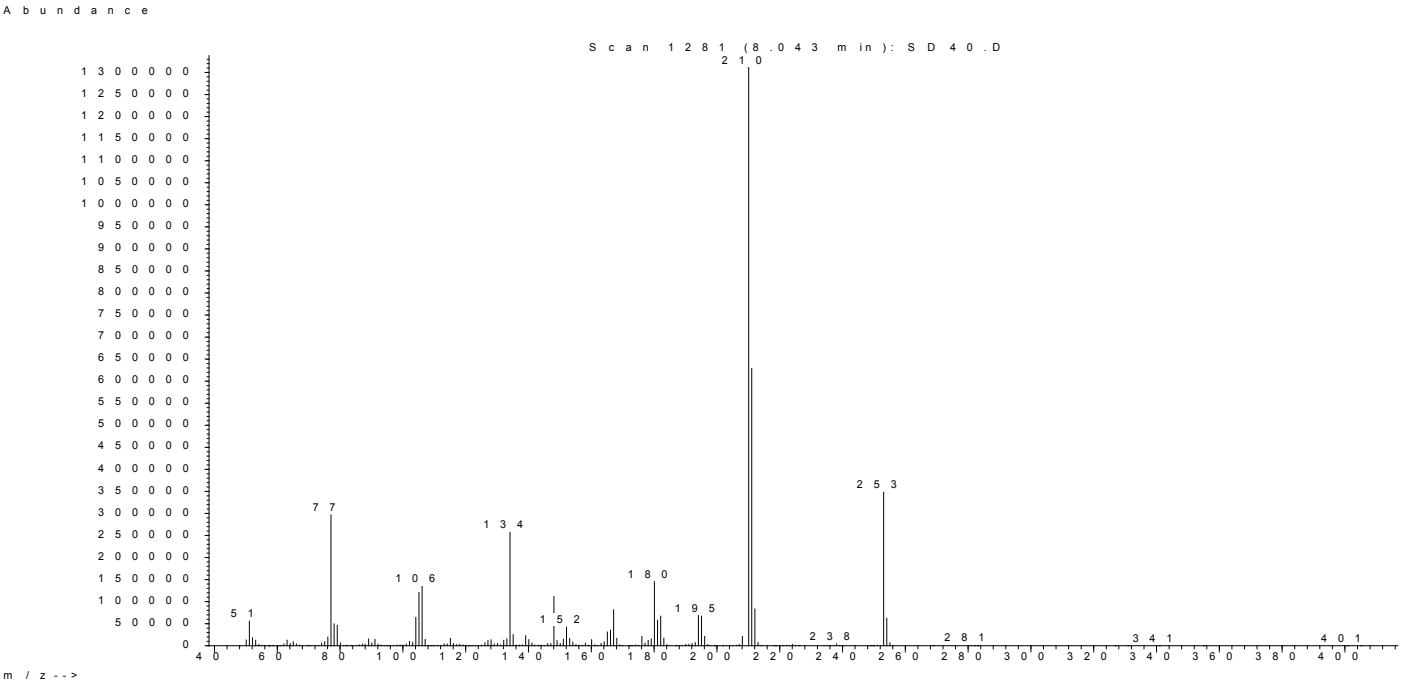
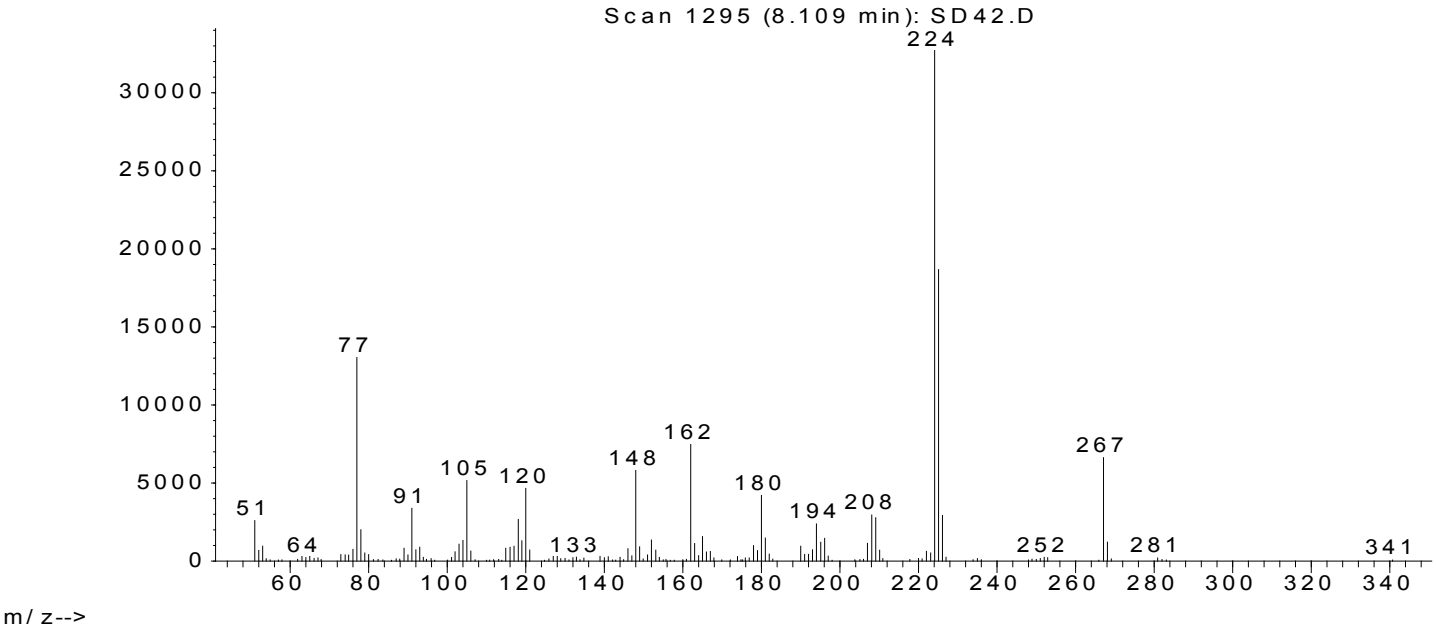
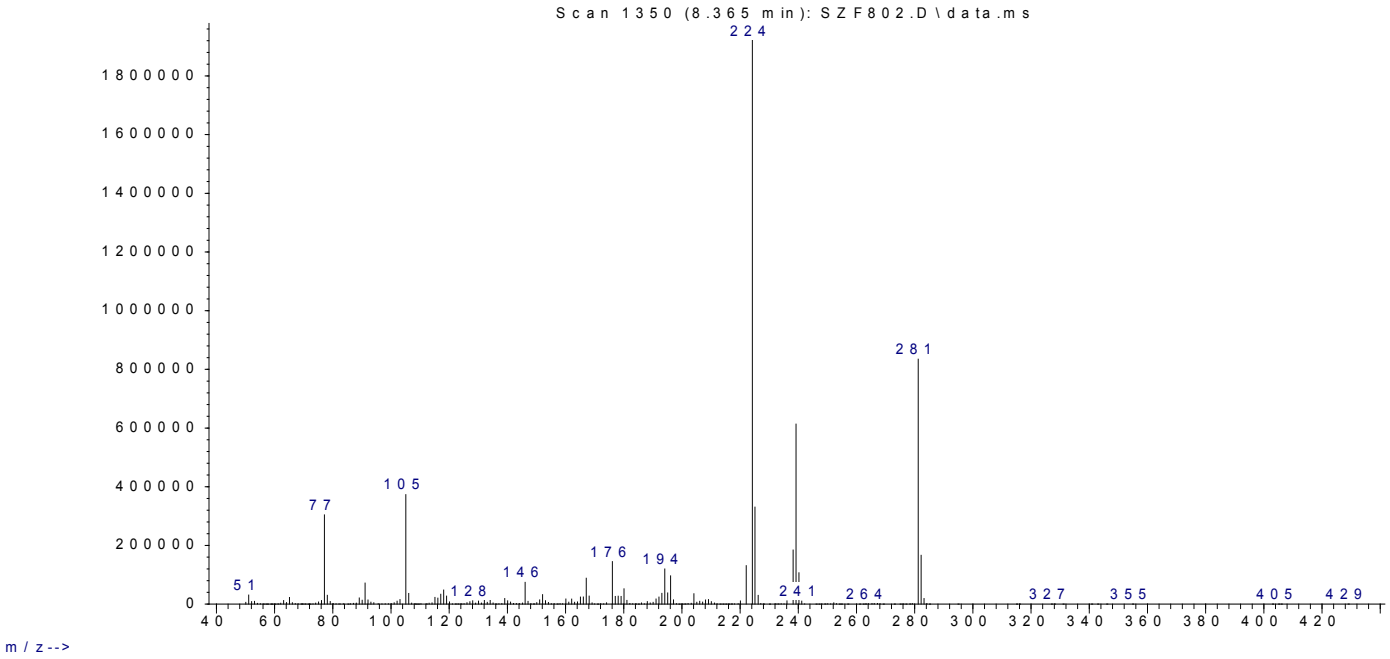
N-(2-benzoyl-4-methylphenyl)acetamide (4c)



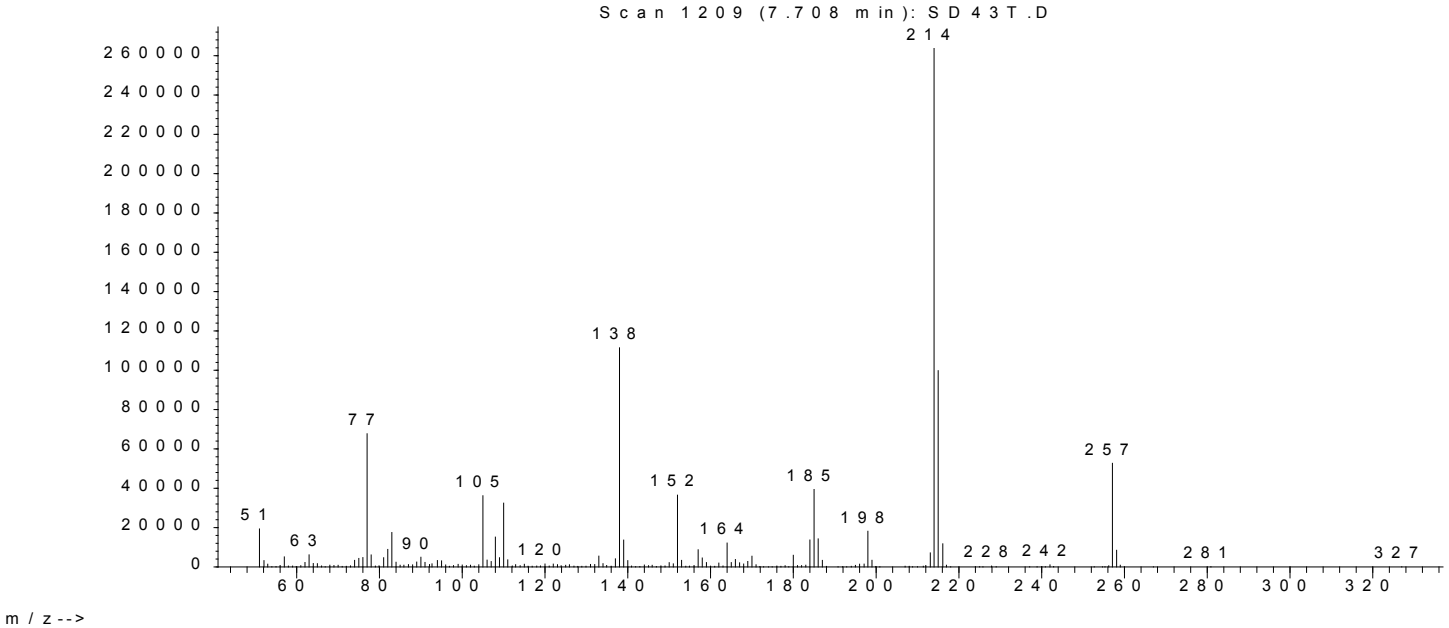
N-(2-benzoyl-4,6-dimethylphenyl)acetamide (4d)



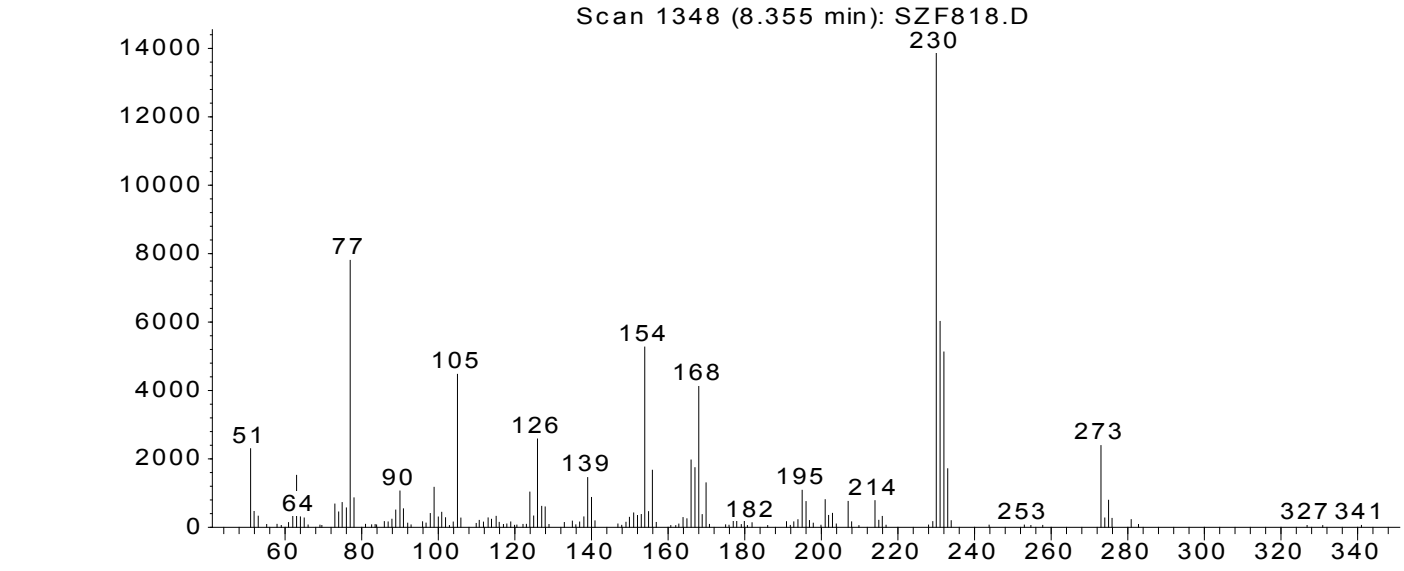
N-(2-benzoyl-4-isopropylphenyl)acetamide (4e)



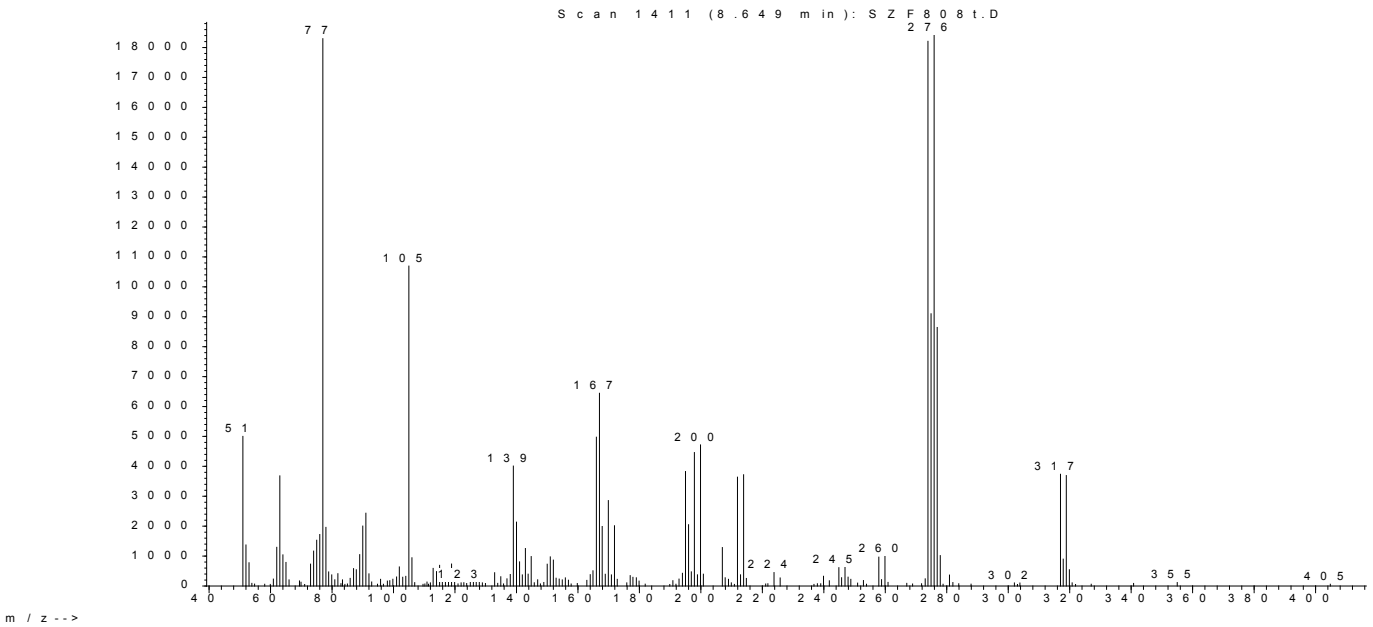
N-(2-benzoyl-5-fluoro-phenyl)acetamide (4f)



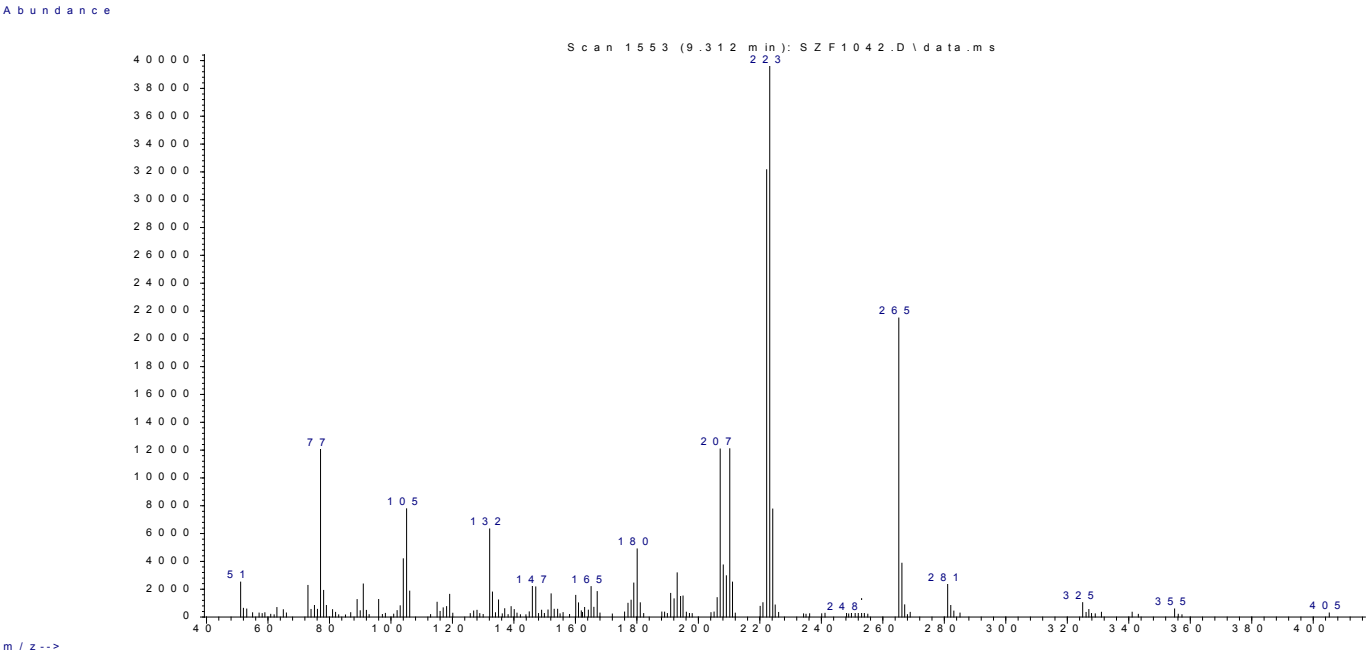
N-(2-benzoyl-5-chlorophenyl)acetamide (4g)



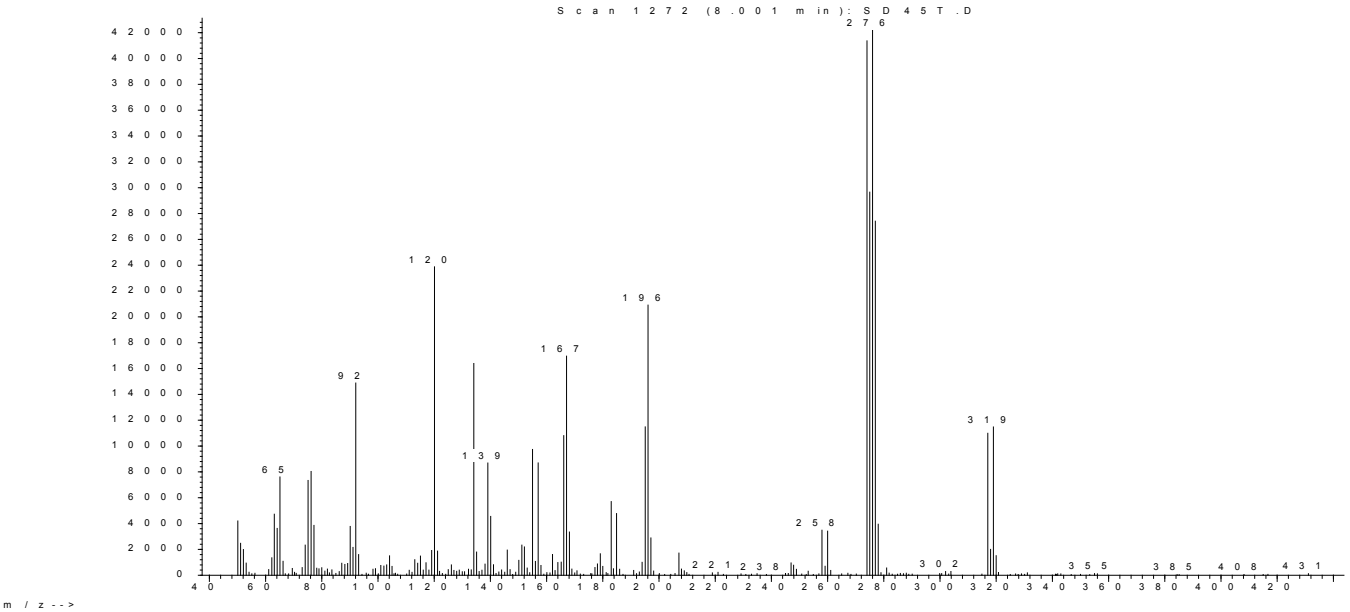
N-(2-benzoyl-5-bromophenyl)acetamide (4h)



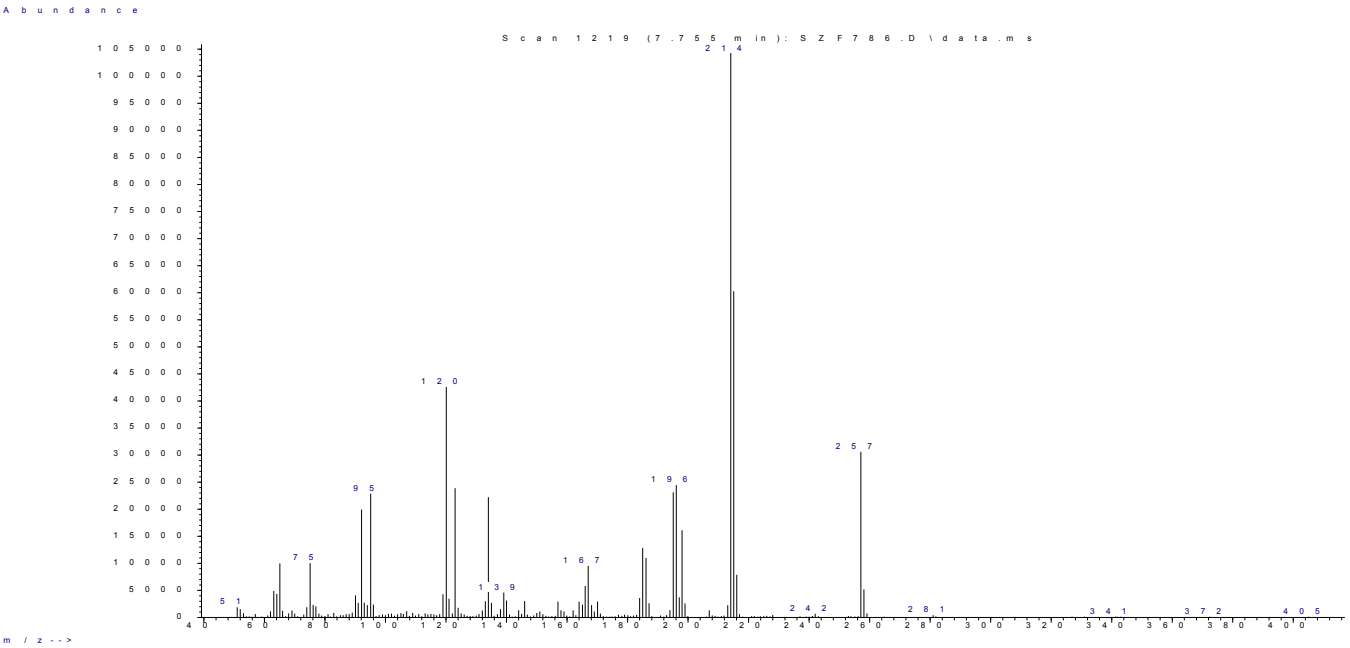
N-(2-benzoyl-4-(2-hydroxyethyl)phenyl)acetamide (4i)



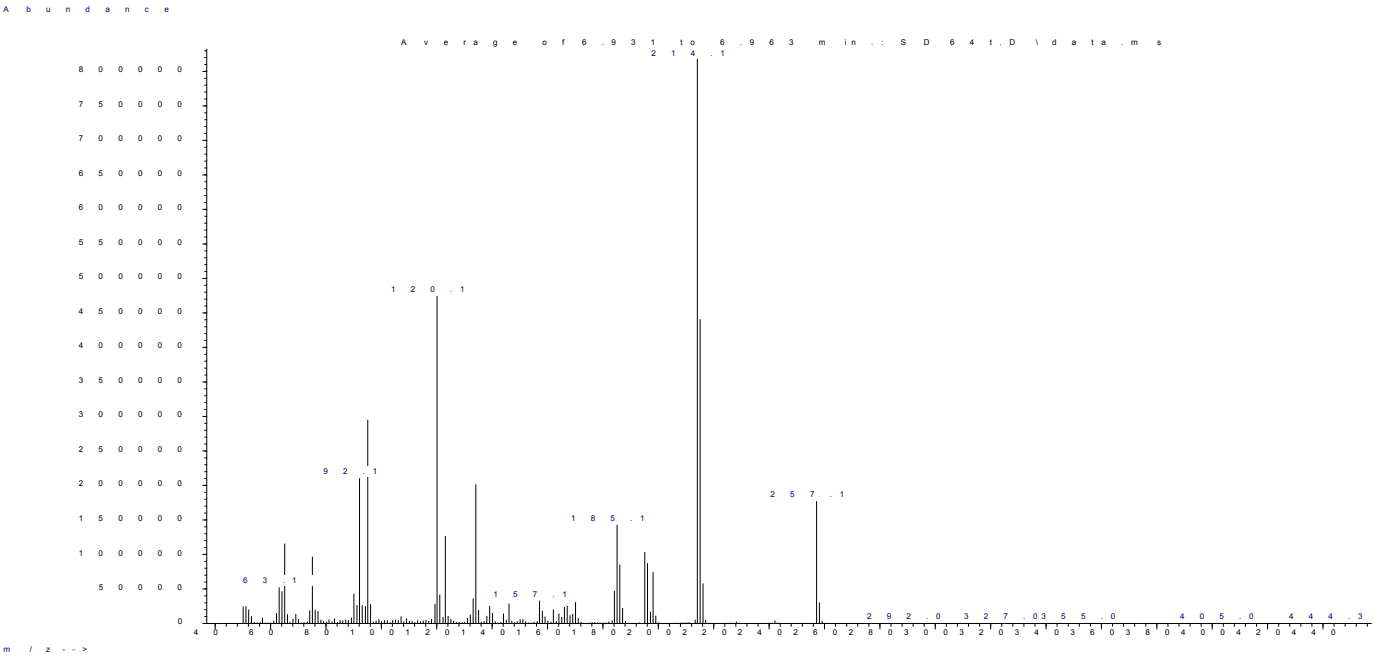
Methyl 2-(4-acetamido-3-benzoylphenyl) acetate (4j)



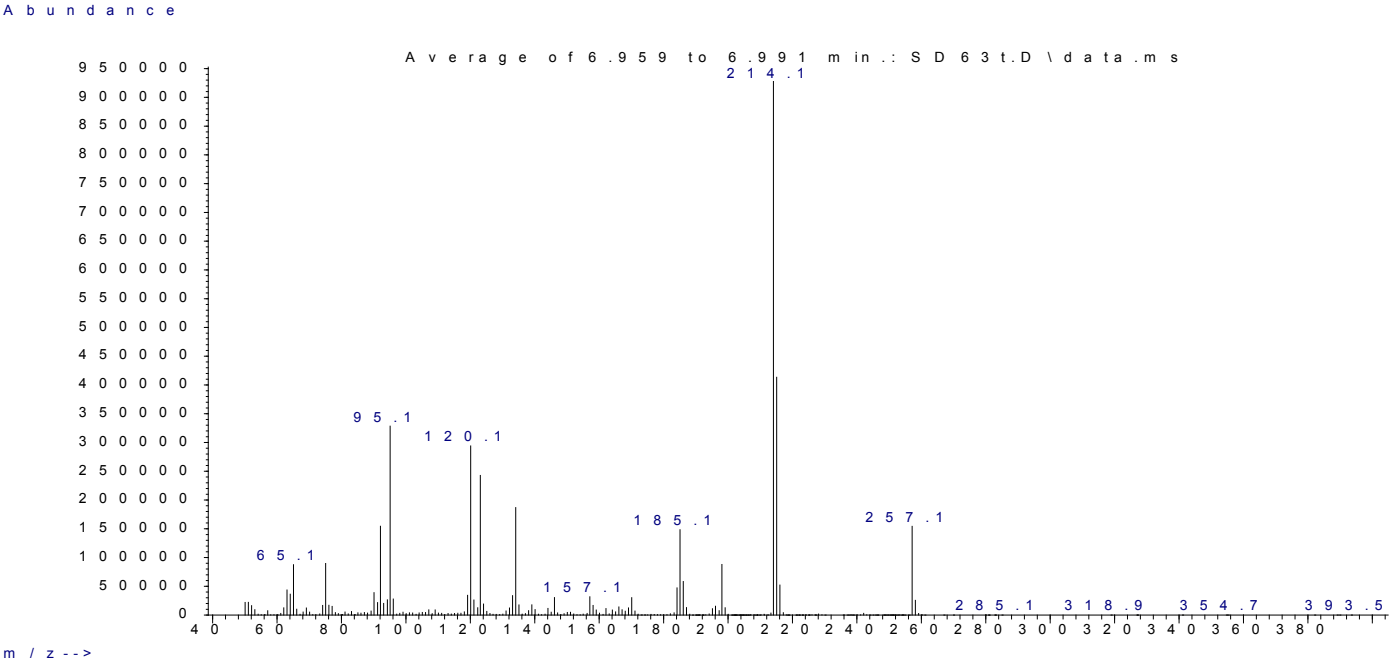
N-(2-(2-fluorobenzoyl)phenyl)acetamide (4k)



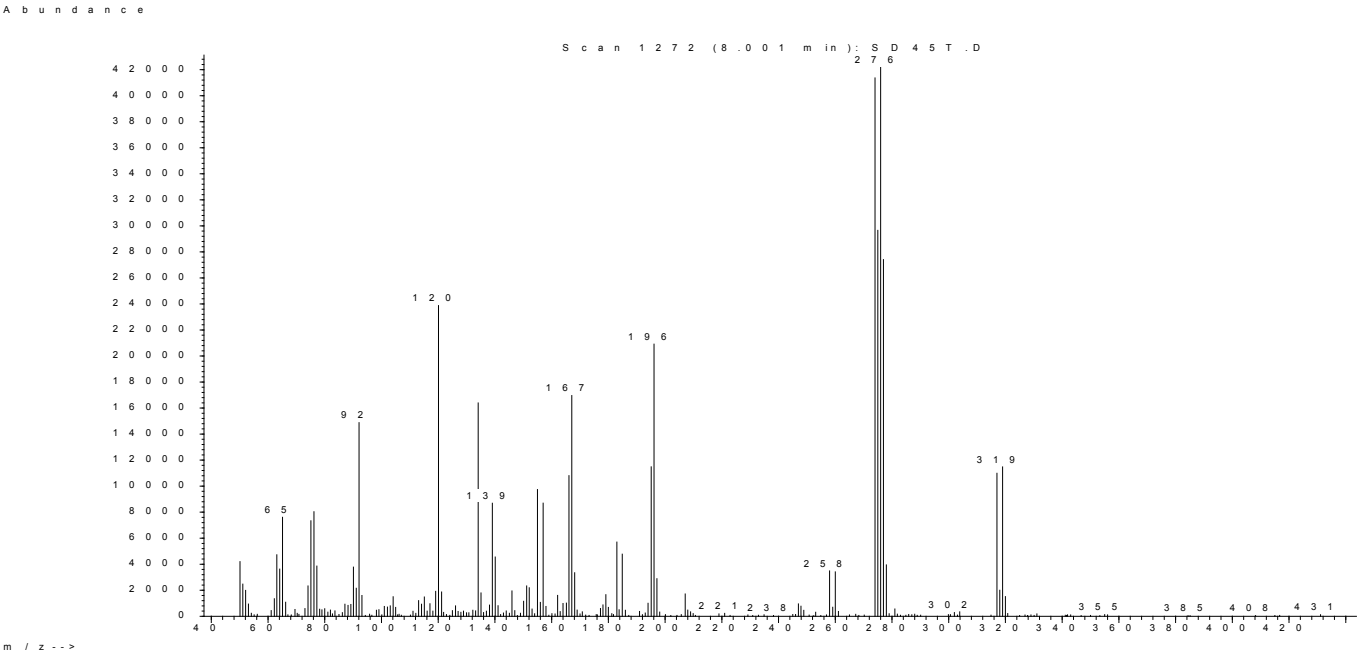
N-(2-(3-fluorobenzoyl)phenyl)acetamide (4l)



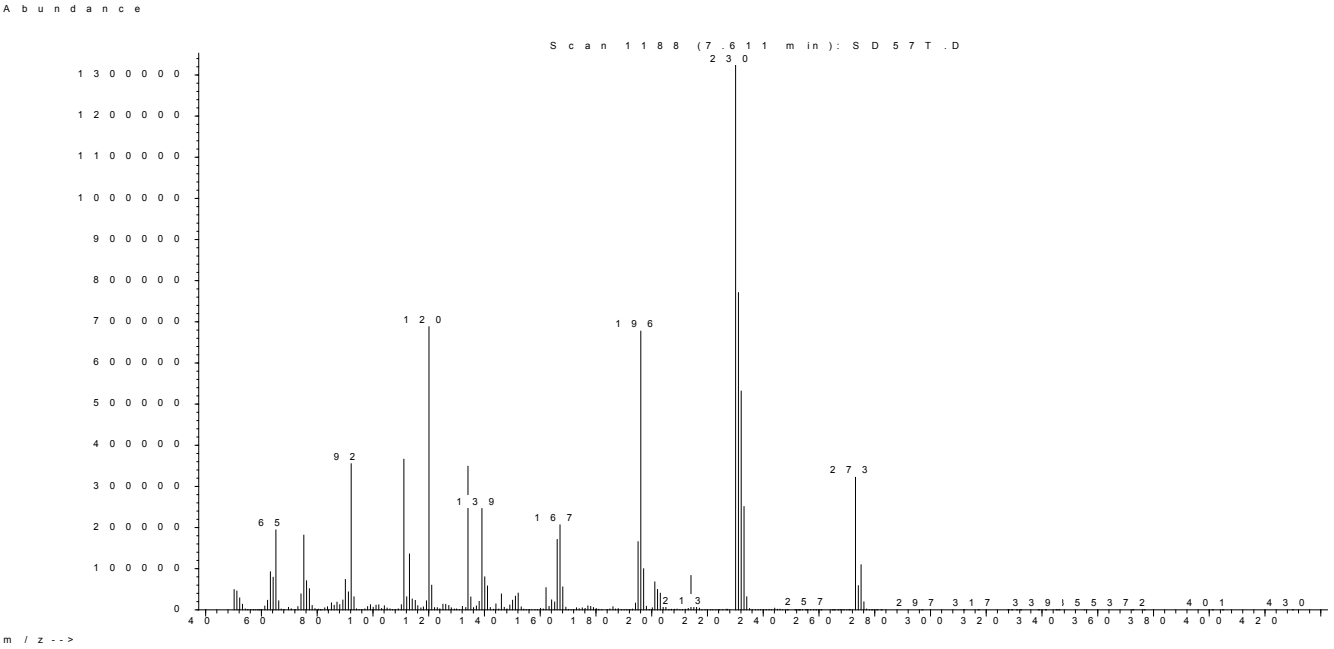
N-(2-(4-fluorobenzoyl)phenyl)acetamide (4m)



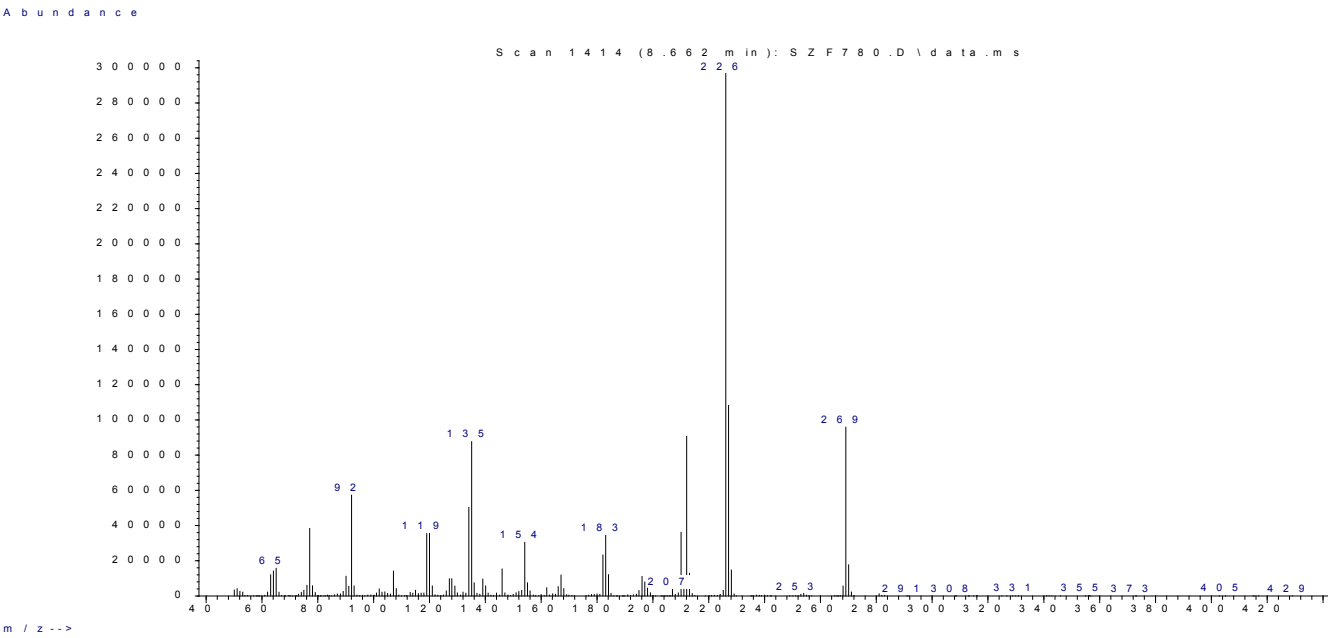
N-(2-(4-bromobenzoyl)phenyl)acetamide (4n)



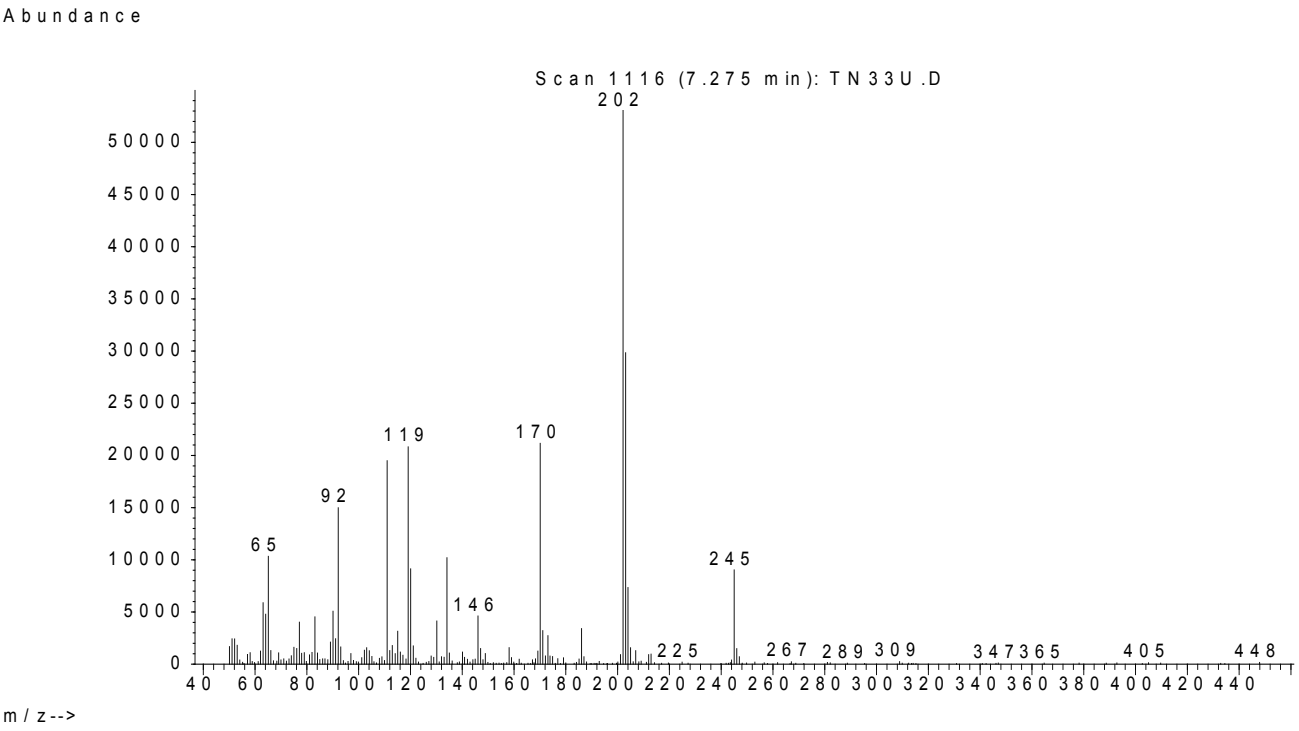
N-(2-(3-chlorobenzoyl)phenyl)acetamide (4o)



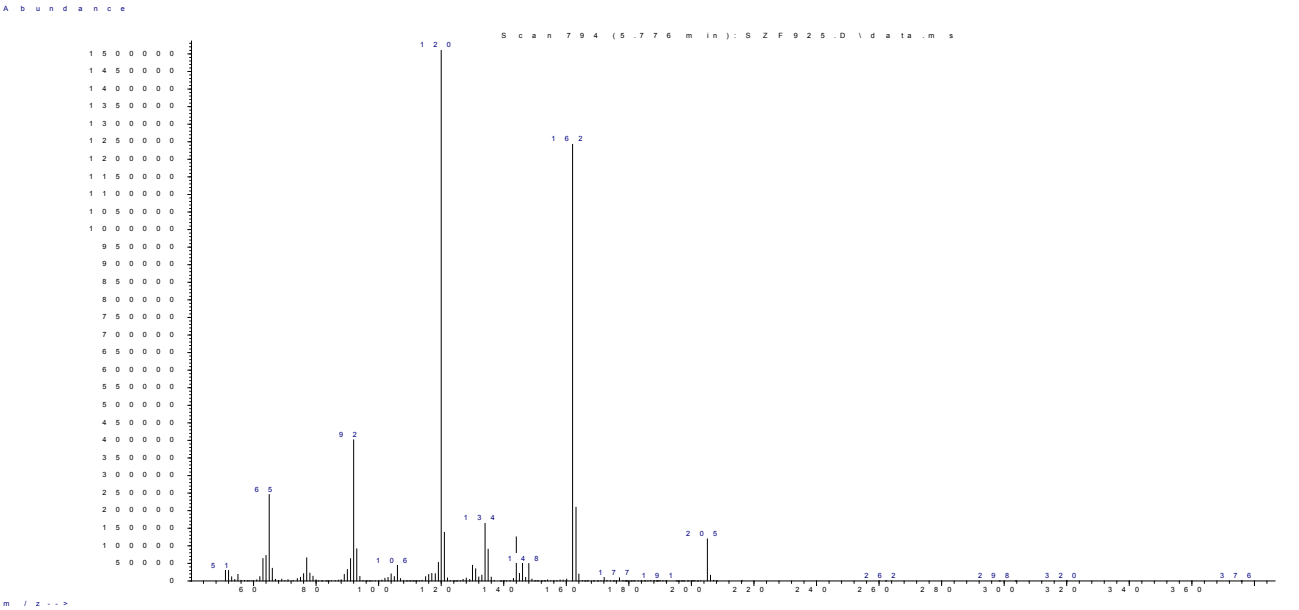
N-(2-(4-methoxybenzoyl)phenyl)acetamide (4p)



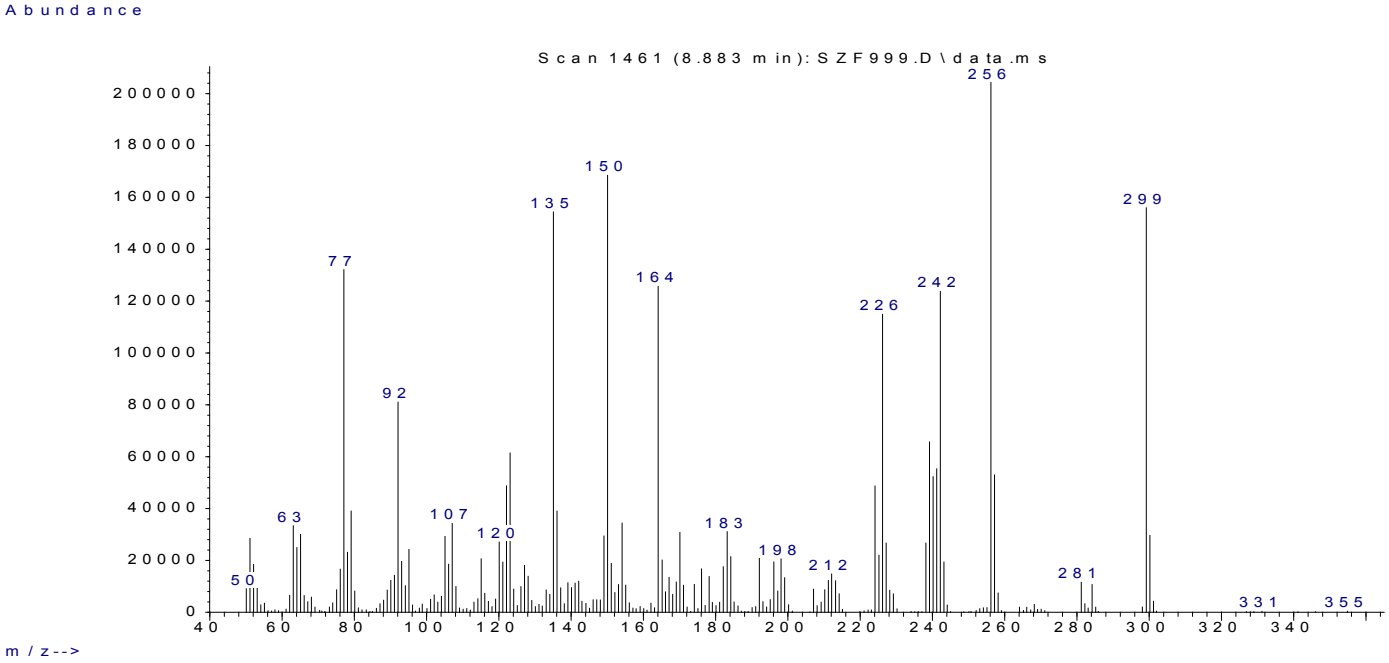
N-(2-(thiophene-2-carbonyl)phenyl)acetamide (4q)



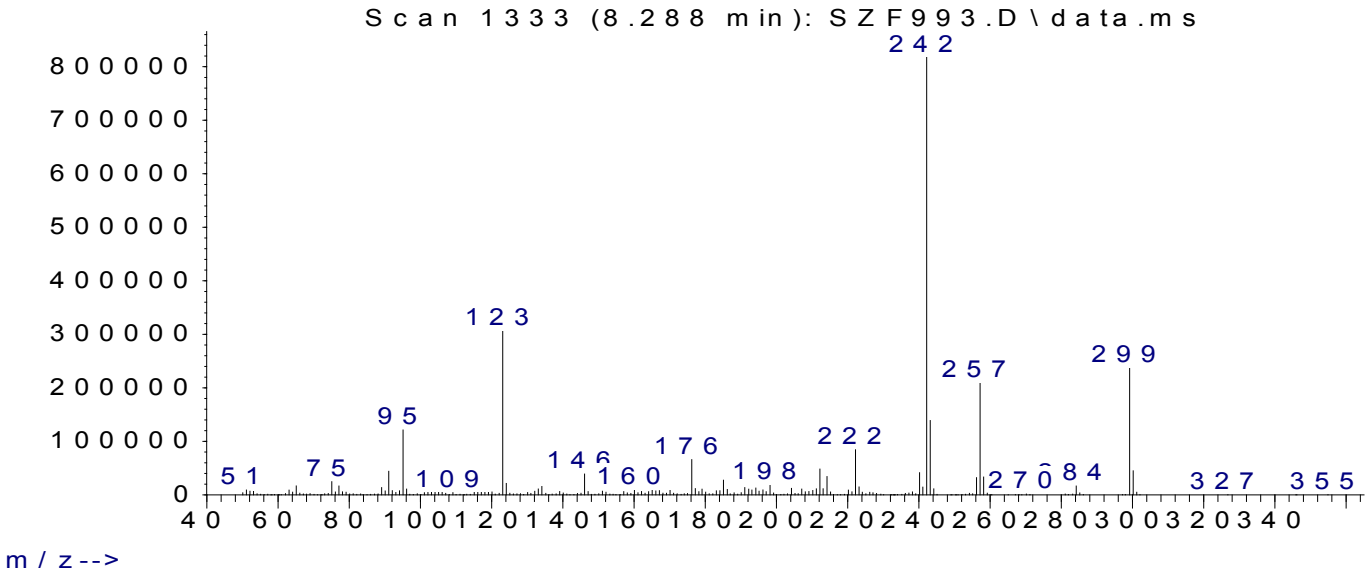
N-(2-butyrylphenyl)acetamide (4r)



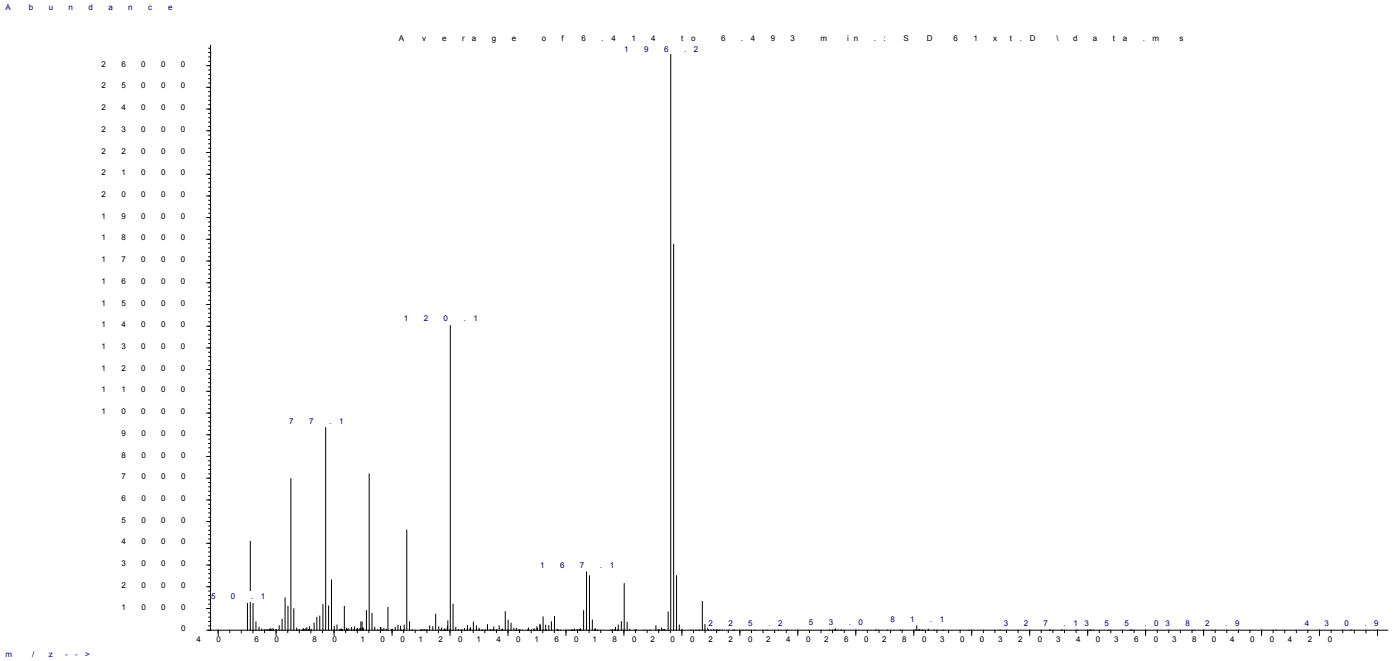
N-(4-methoxy-2-(2-methoxybenzoyl)phenyl)acetamide (4s)



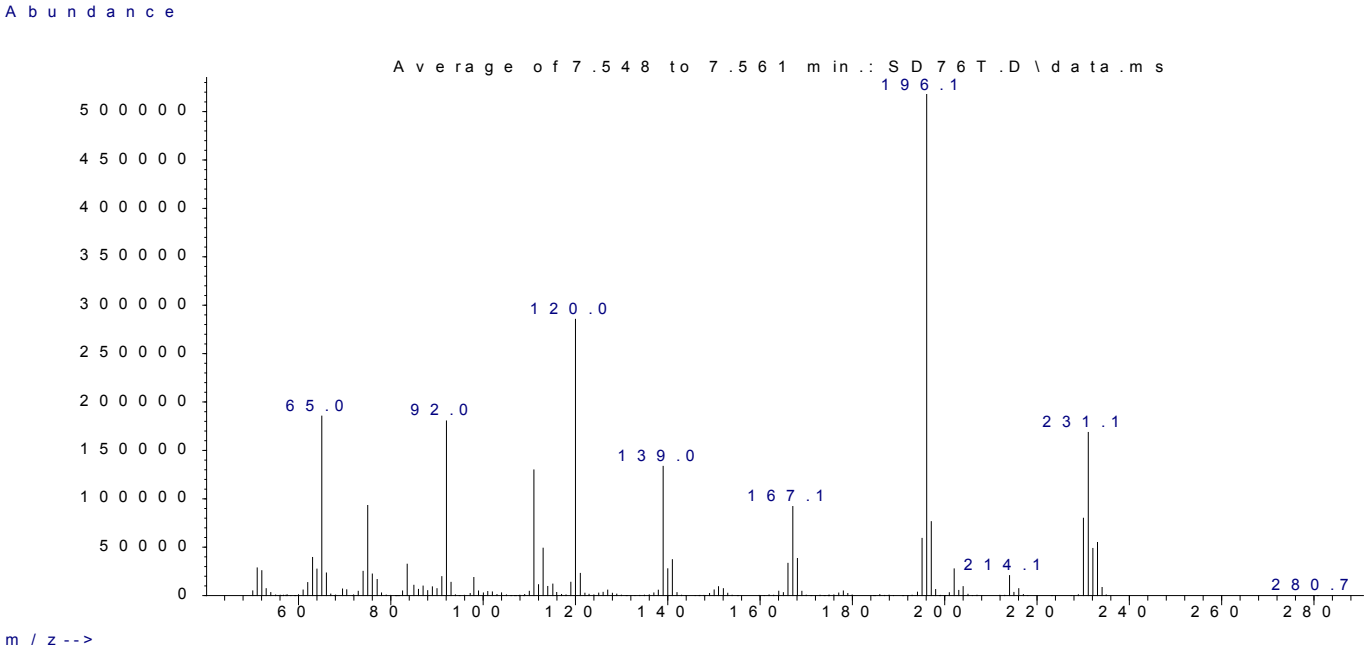
N-(2-(2-fluorobenzoyl)-4-isopropylphenyl)acetamide (4t)



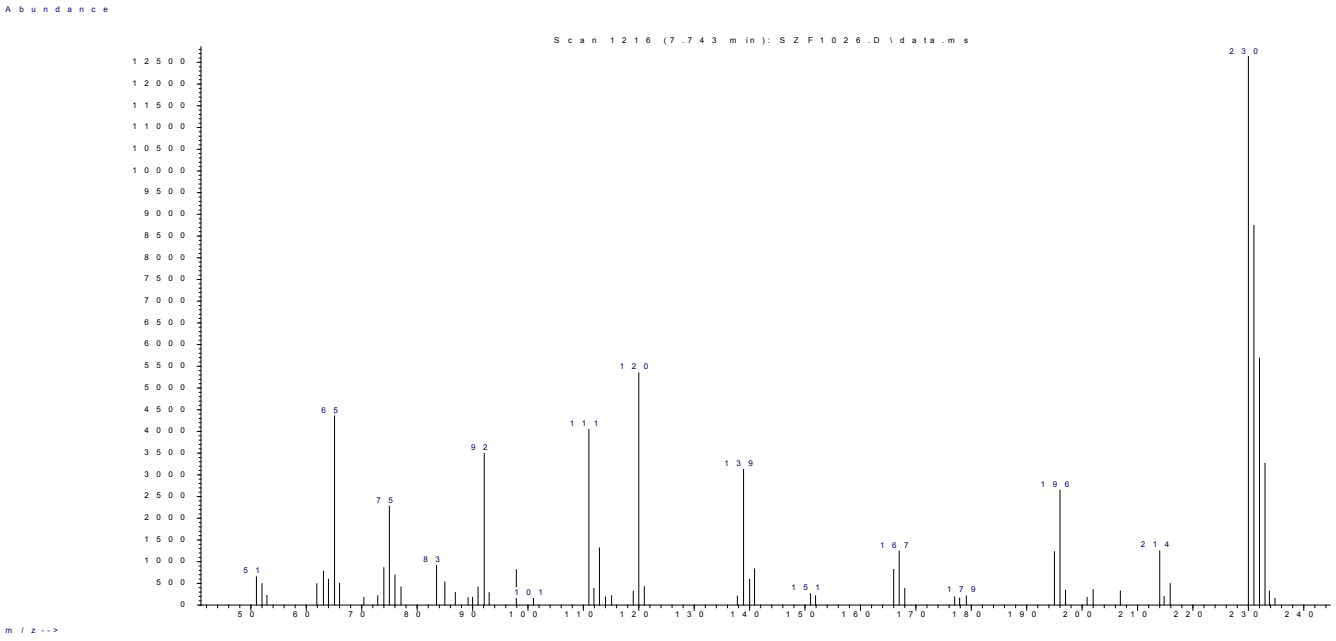
(2-aminophenyl)(phenyl)methanone (5a)



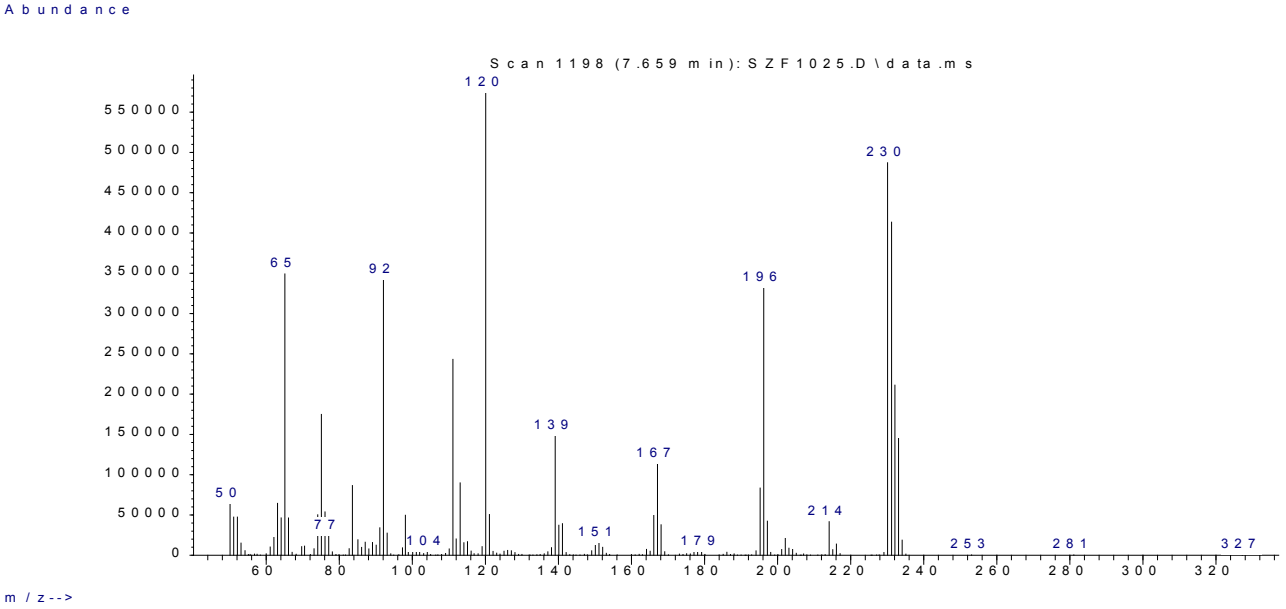
(2-aminophenyl)(2-chlorophenyl)methanone (5b)



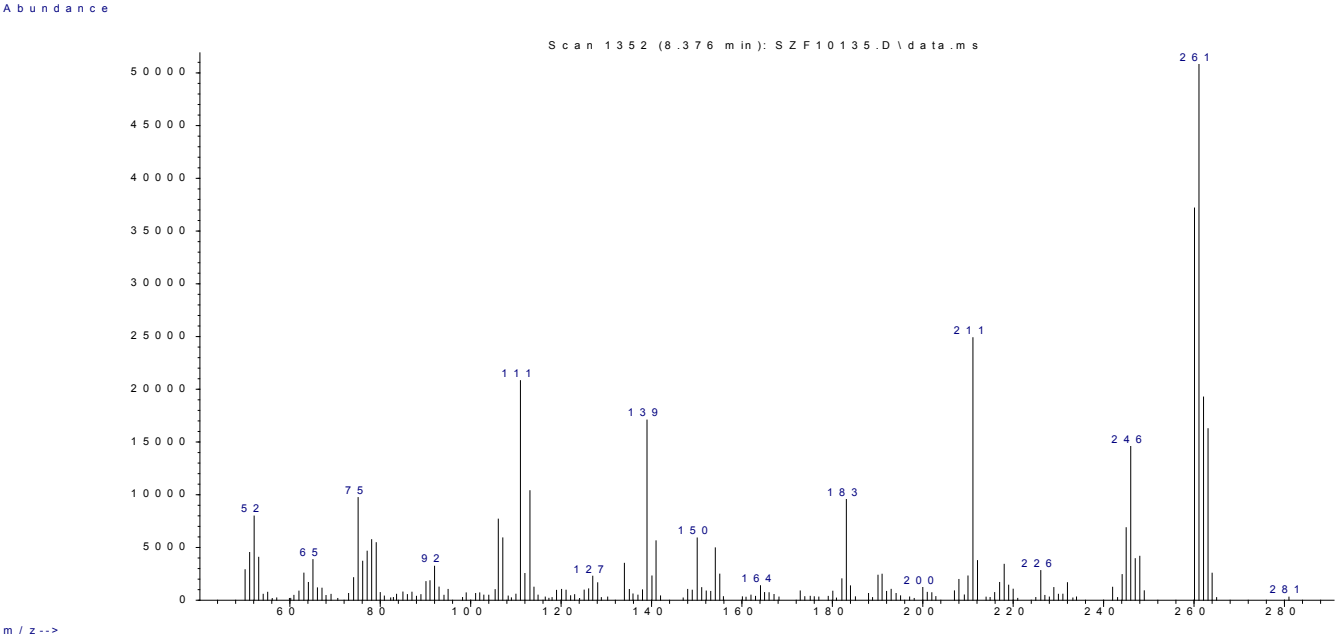
(2-aminophenyl)(4-chlorophenyl)methanone (5c)



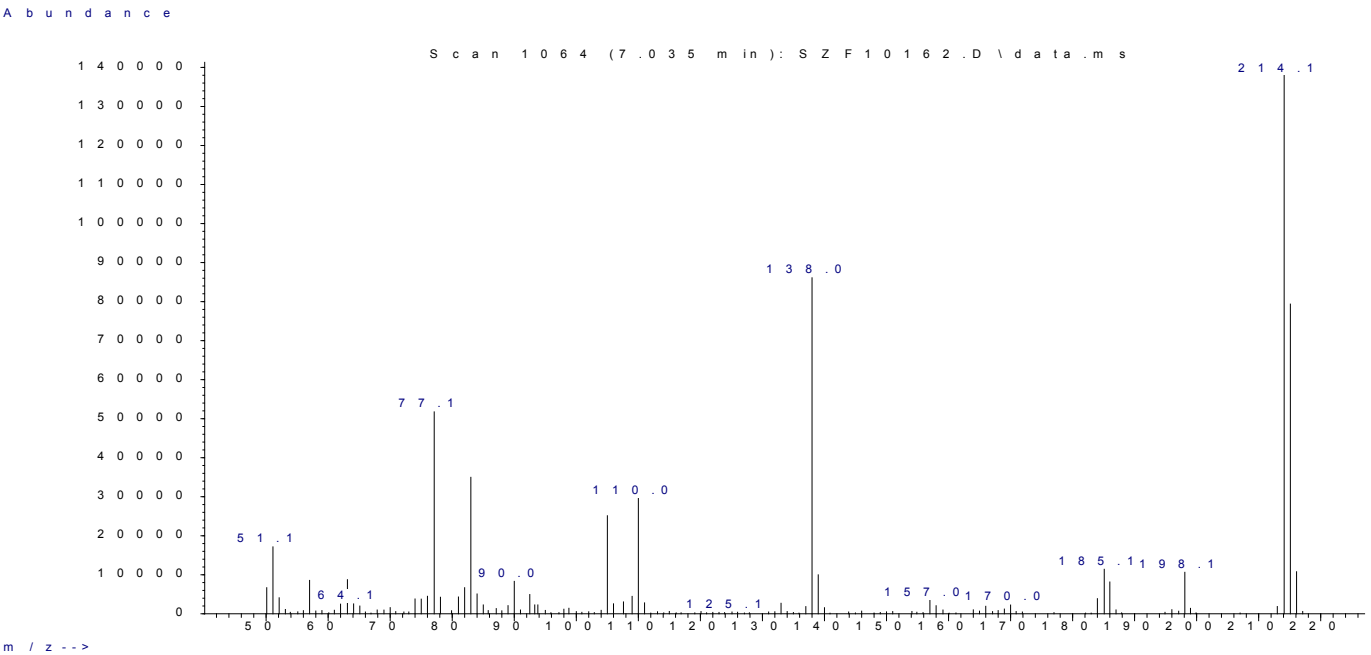
(2-aminophenyl)(3-chlorophenyl)methanone (5d)



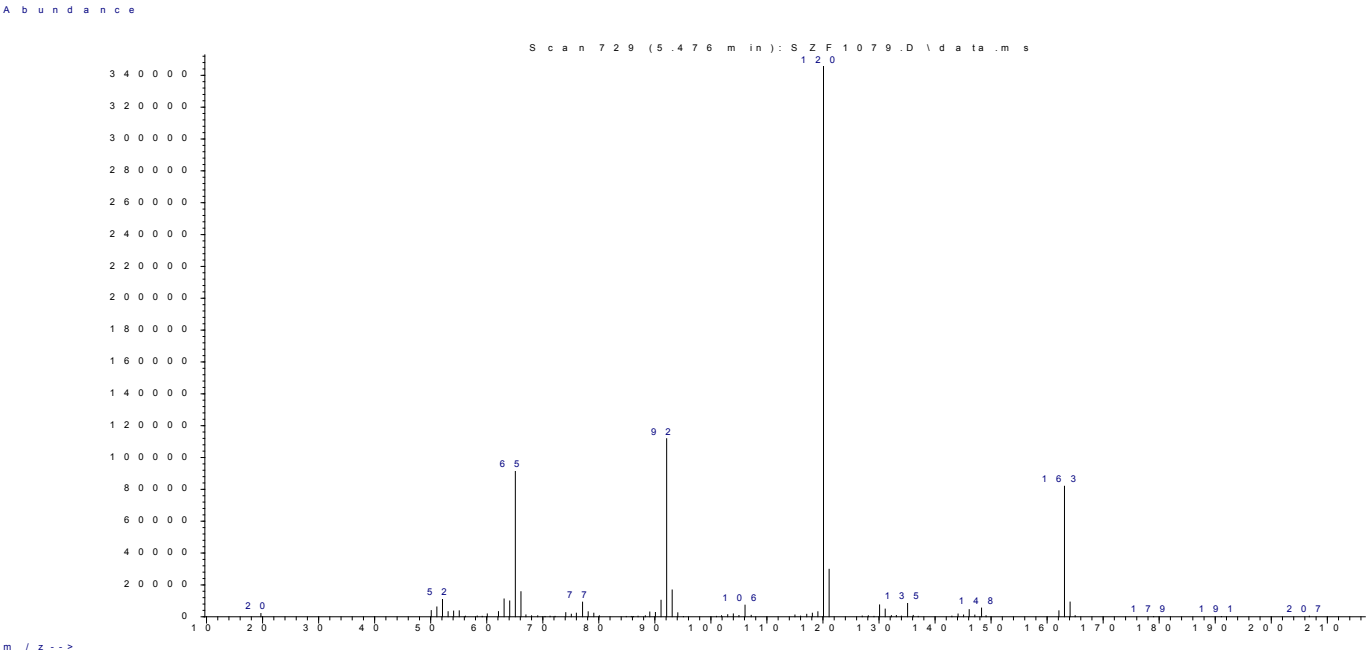
(2-amino-3-methoxyphenyl)(4-chlorophenyl)methanone (5e)



(2-amino-4-fluorophenyl)(phenyl)methanone (5f)



1-(2-aminophenyl)butan-1-one (5g)



(2-aminophenyl)(thiophen-2-yl)methanone (5h)