Electronic Supplementary Material (ESI) for Reaction Chemistry & Engineering.

Integrated Continuous Flow/Batch Protocol for the Photoreduction of *ortho*-Methyl Phenyl Ketones using Water as Hydrogen Source

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1. General

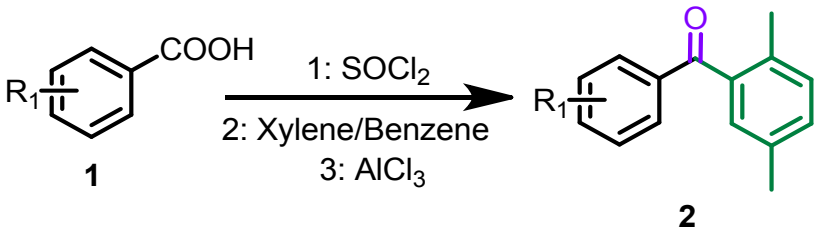
1.1. Materials: Most of the reagents, chemicals and TiO_2 anatase nanopowder bought from Sigma-Aldrich as used as such without any further modification. Common organic chemicals and salts were purchased from Avra chemicals, India. Deionized water (18.2 mS conductivity) was used in all experiments. All work-up and purification procedures were carried out with reagent-grade solvents in air. Analytical thin-layer chromatography (TLC) was performed using analytical chromatography silica gel 60 F254 precoated plates (0.25 mm). The developed chromatogram was analysed by UV lamp (254 nm). PTFE (id = 500 µm) tubing, T-junction, high-purity PFA tubing was purchased from Upchurch IDEX Health & Science. Asia syringe pump, heating system, back pressure controller (BPR), valve, catalytic reactor, Asia Manager PC software system bought from Syrris Asia System. Homemade photo-batch reactor bought from lelesil Mumbai, India and modified for the continuous flow reaction.

1.2. Analysis: High-resolution mass spectra (HRMS) were obtained from a JMS-T100TD instrument (DART) and Thermo Fisher Scientific Exactive (APCI). Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker 600, 500, 400 or 300 MHz in $CDCl_3$ or DMSO-d_6 solvent. Chemical shifts for ^1H NMR are expressed in parts per million (ppm) relative to tetramethyl silane (δ 0.00 ppm). Chemical shifts for ^{13}C NMR are expressed in ppm relative to $CDCl_3$ (δ 77.0 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, dd = doublet of doublets, t = triplet, q = quartet, quin = quintet, sext = sextet, m = multiplet), coupling constant (Hz), and integration. GC/MS analysis was conducted on an Simadzu technology GCMS-QP2010 instrument equipped with a HP-5 column (30 m × 0.25 mm, Hewlett-Packard) and inbuilt MS 5975C VL MSD system with triple

axis detector. SEM images were collected using JEOL JSM-7401F high resolution scanning electron microscope operating at 3 kV. The powder XRD diffractograms were obtained by a Rigaku X-ray diffractometer (Cu Kα radiation) at 49 100 kV, 30 mA.

2. General reaction procedure and characterization of products in details.

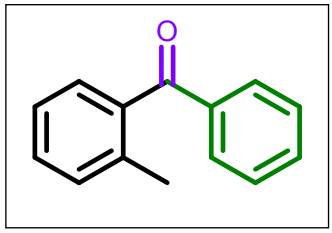
2.1. Preparation of starting materials 2b-2w:



1 **2**

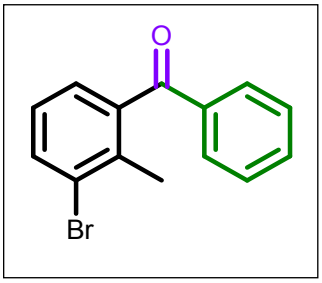
Reported method has been applied for the synthesis of starting materials. At first, substituted aromatic acid (2.0 mmol) and then slowly $SOCl_2$ (8 mmol) was added to the reaction mixture and then refluxed until a clear solution had formed (ca. 2 h). Excess $SOCl_2$ was removed *in vacuo* (under nitrogen), and the resulting acid chloride (2.0 mmol) was slowly mixed with substituted benzene (4mmol) and $AlCl_3$ (3 mmol) at 0 ^{0}C and then stirred at RT for 10-12h. After the reaction completion, 40 ml ice cold water was added to reaction mixture and then then quenched with 1M HCl (1 ml). Organic product was extracted with DCM (3 x 40 mL). Further to remove unused aromatic acid, we have added saturated $NaHCO_3$ (20 mL). The combined organic layers were washed with brine solution (20 mL), dried over Na_2SO_4, and the solvent removed *in vacuo* to yield the crude product. The title compound was obtained after silica gel flash chromatography (Hexane/ethyl acetate (98:02, v/v) as a followed phase product.

Phenyl (o-tolyl) methanone (2a):



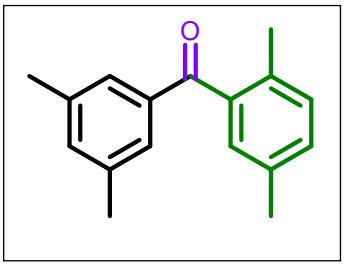
Directly purchased form Sigma-Aldrich and used as such.

(3-bromo-2-methylphenyl) (phenyl)methanone (2b).



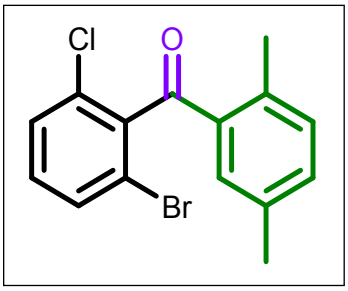
Starting material **2b** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a colorless liquid (471.1 mg, 78%); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.84 – 7.76 (m, 2H), 7.66 (d, *J* = 7.9 Hz, 1H), 7.58 (s, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.21 (d, *J* = 7.6 Hz, 1H), 7.12 (d, *J* = 7.7 Hz, 1H), 2.31 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 197.3, 140.8, 136.7, 135.6, 133.9, 133.5, 130.0, 128.5, 126.6, 126.5, 20.3; **IR (ν_{max}):** 3063, 2926, 2862, 1665, 1588, 1492, 1438, 1386, 1263, 1196, 1114, 1006, 940, 859, 774, 713, 654 cm^{-1}; **HRMS (ESI):** m/z calcd. for $C_{14}H_{12}BrO$ $[M+H]^+$: 275.0072, found: 275.0062.

(2,5-dimethylphenyl) (3,5-dimethylphenyl) methanone (2c).



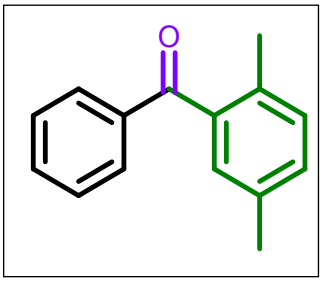
Starting material **2c** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide Colourless liquid (395.0 mg, 83%); The spectra data matched with values reported in the literature.[1] **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.40 (s, 2H), 7.22 – 7.14 (m, 3H), 7.10 (s, 1H), 2.35 (s, 6H), 2.33 (s, 3H), 2.25 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 199.4, 139.0, 138.1, 137.9, 134.8, 134.7, 133.3, 130.7, 130.7, 128.7, 127.8, 21.2, 20.9, 19.5; **IR (ν_{max}):** 2920, 2864, 1662, 1600, 1446, 1388, 1308, 1242, 1179, 1034, 963, 904, 866, 819, 776, 737, 639 cm^{-1}; **HRMS (ESI):** m/z calcd. for $C_{17}H_{18}O$ $[M+H]^+$: 239.1433, found: 239.1436.

(2-bromo-6-chlorophenyl) (2,5-dimethylphenyl) methanone (2d).



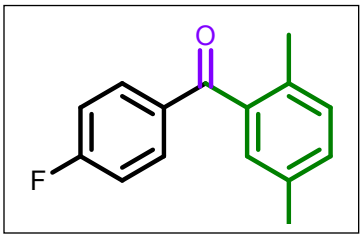
Starting material **2d** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a yellow color liquid (520.0 mg, 81%); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.53 (dd, *J* = 7.2, 2.2 Hz, 2H), 7.33 – 7.28 (m, 1H), 7.26 – 7.18 (m, 2H), 7.11 (s, 1H), 2.51 (s, 3H), 2.28 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 195.7, 141.43, 136.8, 136.0, 135.3, 134.2, 133.3, 132.3, 132.0, 131.9, 131.7, 130.7, 120.5, 20.8; **IR (ν_{max}):** 2965, 2924, 2861, 1672, 1567, 1494, 1454, 1384, 1291, 1211, 1127, 1053, 960, 877, 824, 771, 683, 657 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{12}BrClO$ $[M+H]^+$: 322.9838, found: 322.9841.

(2,5-dimethylphenyl) (phenyl) methanone (2e):



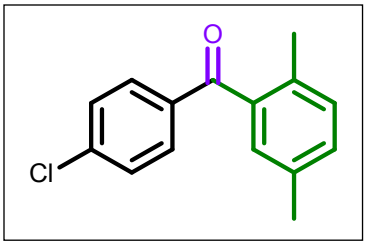
Starting material **2e** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a Colorless liquid (344.4 mg, 82%); The spectra data matched with values reported in the literature.[1, 2] **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.79 (d, *J* = 7.6 Hz, 2H), 7.51 (s, 1H), 7.39 (t, *J* = 7.0 Hz, 2H), 7.18 – 7.08 (m, 3H), 2.28 (s, 3H), 2.25 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 198.7, 138.6, 137.8, 134.8, 133.5, 133.1, 131.0, 130.9, 130.1, 128.9, 128.5, 20.9, 19.5; **IR (ν_{max}):** 3030**,** 2926, 2864, 1664, 1587, 1492, 1447, 1391, 1302, 1267, 1212, 1168, 952, 851, 814, 770, 739, 695, 649 cm-1; **HRMS (ESI):** m/z calcd for $C_{15}H_{12}Cl_2O$ $[M+H]^+$: 211.1123, found: 211.112.

(2,5-dimethylphenyl) (4-fluorophenyl) methanone (2f):



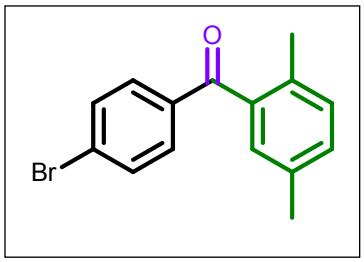
Starting material **2f** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide Yellow colour liquid (396.7 mg, 86%); The spectra data matched with values reported in the literature.[1] **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.87 – 7.77 (m, 2H), 7.22 – 7.08 (m, 5H), 2.33 (s, 3H), 2.25 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 197.29, 167.03, 164.49, 138.32, 134.88, 134.12 (d, *J*= 2.9 Hz), 133.29, 132.69 (d, *J* = 8.8 Hz), 130.95 (d, *J* = 11.7 Hz), 128.63, 115.68, 115.46, 20.84, 19.39; **IR (ν_{max}):** 3024, 2926, 1665, 1597, 1497, 1450, 1407, 1376, 1298, 1273, 1221, 1153, 1104, 1030, 953, 894, 855, 817, 765, 681, 641 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{13}FO$ $[M+H]^+$: 229.1029, found 229.1026.

(2,5-dimethylphenyl) (4-chlorophenyl) methanone (2g):



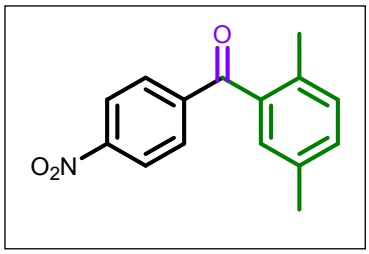
Starting material **2g** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a yellow liquid (405.0 mg, 83%); **^{1}H NMR (500 MHz, $CDCl_3$):** δ 7.75 – 7.71 (m, 2H), 7.39 (d, *J* = 8.2 Hz, 2H), 7.17 (d, *J* = 9.3 Hz, 2H), 7.09 (s, 1H), 2.31 (s, 3H), 2.25 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 197.21, 139.33, 137.87, 135.99, 134.71, 133.36, 131.28, 131.06, 130.85, 128.68, 128.60, 20.70, 19.33; **IR (ν_{max}):** 3021, 1664, 1585, 1488, 1400, 1300, 1272, 1215, 1168, 1092, 1008, 954, 850, 814, 761, 672 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{13}ClO$ $[M+H]^+$: 245.0733, found 245.0728.

(2,5-dimethylphenyl) (4-bromophenyl) methanone (2h):



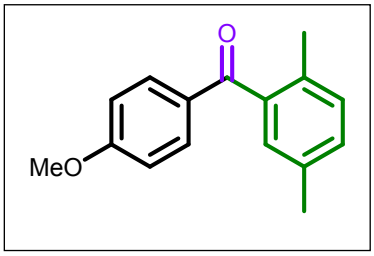
Starting material **2h** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a white solid (466.5 mg, 81%); The spectra data matched with values reported in the literature.[2] **^{1}H NMR (500 MHz, $CDCl_3$):** δ 7.67 – 7.64 (m, 2H), 7.56 – 7.53 (m, 2H), 7.17 (dt, *J* = 15.9, 4.7 Hz, 2H), 7.10 (s, 1H), 2.31 (s, 3H), 2.27 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ 197.0, 137.6, 136.3, 134.5, 133.2, 131.4, 131.3, 131.2, 131.1, 130.9, 130.7, 128.6, 127.9, 77.0, 20.6, 19.2; **IR (ν_{max}):** 2923, 2866, 1664, 1577, 1486, 1448, 1394, 1298, 1267, 1210, 1168, 1106, 1067, 1004, 952, 903, 845, 768, 731, 667 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{13}BrO$ $[M+H]^+$: 289.0228, found: 289.0228.

(2,5-dimethylphenyl) (4-nitrophenyl) methanone (2i):



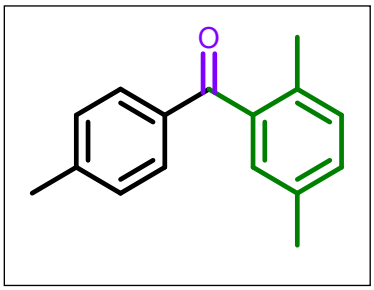
Starting material **2i** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a white solid (433.5 mg, 85%); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.33 – 8.26 (m, 2H), 7.94 (d, *J* = 8.8 Hz, 2H), 7.24 (dd, *J* = 19.9, 8.4 Hz, 2H), 7.12 (s, 1H), 2.34 (s, 3H), 2.31 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ 196.70, 150.05, 142.79, 136.86, 135.07, 134.27, 132.00, 131.32, 130.76, 129.43, 123.55, 20.76, 19.65; **IR (ν_{max}):** 3105, 3033, 2926, 2866, 1669, 1600, 1526,1452, 1396, 1348, 1302, 1268, 1210, 1162, 1109, 1002, 955, 871, 831, 775, 736, 705, 669 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{13}NO_3$ $[M+H]^+$: 256.0974, found: 256.0970.

(2, 5-dimethylphenyl) (4-methoxyphenyl) methanone (2j):



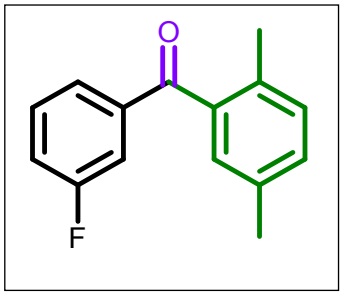
Starting material **2j** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a white solid (379.2 mg, 79%); The spectra data matched with values reported in the literature.[1, 3] **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.82 – 7.75 (m, 2H), 7.20 – 7.12 (m, 2H), 7.09 (s, 1H), 6.96 – 6.89 (m, 2H), 3.87 (s, 3H), 2.33 (s, 3H), 2.23 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 197.61, 163.63, 139.15, 134.72, 132.87, 132.45, 130.65, 130.58, 130.47, 128.33, 113.66, 55.47, 29.68, 20.86, 19.27; **IR (ν_{max}):** 3017, 2956, 1599, 1506, 1456, 1428, 1306, 1260, 1217, 1169, 1111, 1030, 954, 851, 813, 761, 673; **HRMS (ESI):** m/z calcd for $C_{16}H_{16}O_2$ $[M+H]^+$: 241.1229, found: 241.1229.

(2, 5-dimethylphenyl) (4-methylphenyl) methanone (2k):



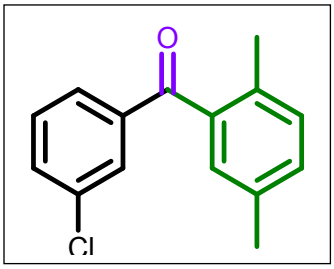
Starting material **2k** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a White solid (398.7 mg, 89%); The spectra data matched with values reported in the literature;[1, 3] **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.70 (d, *J* = 8.1 Hz, 2H), 7.23 (d, *J* = 8.1 Hz, 2H), 7.15 (d, *J* = 4.3 Hz, 2H), 7.09 (s, 1H), 2.40 (s, 3H), 2.31 (s, 3H), 2.24 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ 198.42, 143.86, 138.84, 135.12, 134.61, 133.09, 130.66, 130.62, 130.15, 129.06, 128.55, 21.57, 20.76, 19.30; **IR (ν_{max}):** 2925, 2865, 1665, 1608, 1568, 1452, 1409, 1304, 1282, 1215, 1178, 1114, 1037, 958, 851, 826, 772 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{16}H_{16}O$ $[M+H]^+$: 225.1279, found: 225.1276.

(2, 5-dimethylphenyl) (3-fluorophenyl) methanone (2l):

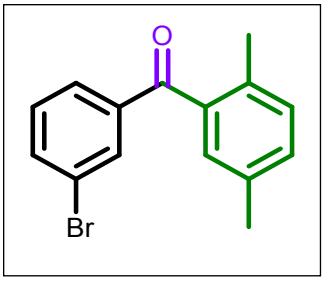


Starting material **2l** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a brownish liquid (487.0 mg, 83%); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.52 (d, *J* = 7.2 Hz, 2H), 7.37 (d, *J* = 5.6 Hz, 1H), 7.28 – 7.06 (m, 4H), 2.30 (s, 3H), 2.27 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ196.91, 163.42, 161.45, 139.80 (d, *J*=6.4), 137.68, 134.62, 133.40, 131.09, 130.81, 129.87 (d, *J*=7.3), 128.70, 125.73 (d, *J*=2.7), 119.81, 119.64, 116.24, 116.13 (d, *J*=21.8), 20.53, 19.19; **IR (ν_{max}):** 2927, 2867, 1670, 1587, 1486, 1443, 1392, 1301, 1271, 1230, 1177, 1145, 1103, 1042, 971, 898, 813, 770, 706, 663 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{13}FO$ $[M+H]^+$: 229.1029, found: 229.1026.

(2, 5-dimethylphenyl) (3-chlorophenyl) methanone (2m):



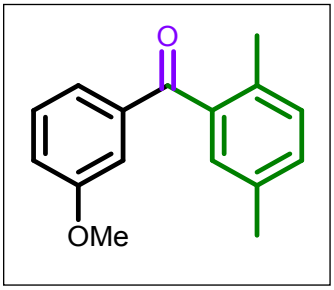
Starting material **2m** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide Yellow colour liquid (410.0 mg, 86%); The spectra data matched with values reported in the literature.[1] **^{1}H NMR (500 MHz, $CDCl_3$):** δ 7.83 – 7.77 (m, 1H), 7.65 – 7.58 (m, 1H), 7.45 (ddd, *J* = 7.9, 2.0, 0.9 Hz, 1H), 7.30 (t, *J* = 7.9 Hz, 1H), 7.17 – 7.07 (m, 3H), 2.28 (s, 3H), 2.25 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ 196.32, 139.13, 137.31, 134.38, 134.26, 133.26, 132.37, 130.97, 130.66, 129.35, 129.18, 128.56, 127.82, 20.35, 19.07; **IR (ν_{max}):** 3024, 2965, 2925, 2866, 1667, 1578, 1438, 1389, 1296, 1264, 1211, 1157, 1123, 1048, 992, 951, 889, 856, 821, 751, 680, 643 cm^{-1}; **HRMS (ESI)**: m/z calcd for $C_{15}H_{13}ClO$ $[M+H]^+$: 245.0733, found: 245.0733.



(2, 5-dimethylphenyl) (3-bromophenyl) methanone (2n):

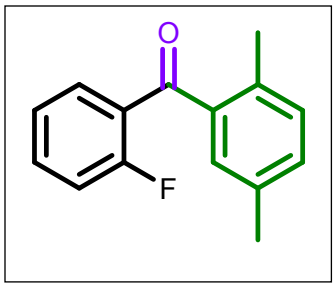
Starting material **2n** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a white solid (437.7 mg, 76%); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.95 (t, *J* = 1.8 Hz, 1H), 7.70 – 7.63 (m, 2H), 7.29 (t, *J* = 7.9 Hz, 1H), 7.21 – 7.13 (m, 2H), 7.10 (s, 1H), 2.31 (s, 3H), 2.26 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ 196.88, 139.54, 137.52, 135.62, 134.72, 133.51, 132.46, 131.25, 130.89, 129.85, 128.81, 128.51, 122.62, 20.70, 19.38; **IR (ν_{max}):** 2925, 2867, 1665, 1566, 1457, 1415, 1295, 1261, 1203, 1159, 1116, 1069, 995, 959, 903, 861, 760, 733, 663 cm^{-1}; **HRMS (ESI)**: m/z calcd for $C_{15}H_{13}BrO$ $[M+H]^+$: 289.0228, found: 289.0227.

(2, 5-dimethylphenyl) (3-methoxyphenyl) methanone (2o):

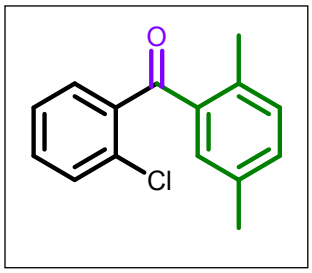


Starting material **2o** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a white solid (374.4 mg, 78%); **^{1}H NMR (500 MHz, $CDCl_3$):** δ 7.44 (d, *J*=1.4, 1H), 7.35 – 7.28 (m, 2H), 7.21 – 7.10 (m, 4H), 3.84 (s, 3H), 2.33 (s, 3H), 2.27 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 198.30, 159.55, 138.96, 138.45, 134.49, 133.17, 130.74, 130.63, 129.18, 128.60, 122.96, 119.35, 113.64, 55.14, 20.62, 19.22; **IR (ν_{max}):** 2925, 2867, 1665, 1566, 1457, 1415, 1295, 1261, 1203, 1159, 1116, 1069, 995, 959, 903, 861, 760, 733, 663 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{16}H_{16}O_2$ $[M+H]^+$: 241.1229, found: 241.1223

(2, 5-dimethylphenyl) (2-fluorophenyl) methanone (2p):



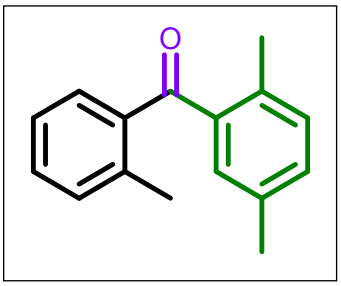
Starting material **2p** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a Yellow color liquid (392.2 mg, 86%); The spectra data matched with values reported in the literature.[1] **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.56 (d, *J* = 4.3 Hz, 1H), 7.43 (s, 1H), 7.21 – 7.09 (m, 4H), 7.04 (d, *J* = 7.8 Hz, 1H), 2.40 (s, 3H), 2.23 (d, *J* = 3.2 Hz, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ 194.8 (s), 160.3 (d, *J* = 254.3 Hz), 138.0, 134.5, 134.3, 133.3 (d, *J* = 8.2 Hz), 131.7 (s), 131.0, 130.7, 129.8, 127.5 (d, *J* = 11.8 Hz), 123.8 (d, *J* = 3.6 Hz), 116.13, 116.0 (d, *J* = 21.8 Hz), 20.2, 19.6; **IR (ν_{max}):** 3029, 2927, 1668, 1611, 1575, 1487, 1451, 1391, 1304, 1272, 1216, 1156, 1108, 1039, 955, 862, 820, 766, 645 cm^{-1}; **HRMS (ESI)**: m/z calcd for $C_{15}H_{13}FO$ $[M+H]^+$: 229.1029, found: 229.1027.



(2, 5-dimethylphenyl) (2-chlorophenyl) methanone (2q):

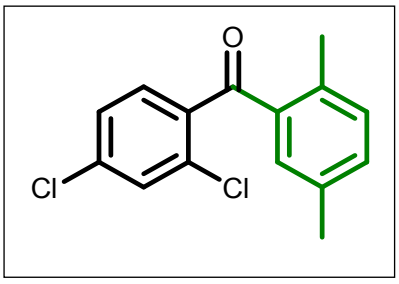
Starting material **2q** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a Brown colour liquid (405 mg, 83%); The spectra data matched with values reported in the literature.[1] **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.43 – 7.37 (m, 3H), 7.34 – 7.30 (m, 1H), 7.19 (q, *J* = 7.6 Hz, 2H), 7.12 (s, 1H), 2.50 (s, 3H), 2.26 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 197.4, 139.6, 136.8, 136.2, 135.0, 132.7, 131.7, 131.7, 131.6, 131.3, 130.2, 129.86, 126.6, 20.7, 20.6; **IR (ν_{max}):** 2925, 2866, 1667, 1578, 1438, 1389, 1296, 1263, 1210, 1157, 1123, 1048, 992, 950, 890, 855, 821, 748, 679, 639 cm^{-1}; **HRMS (ESI)**: m/z calcd for $C_{15}H_{13}ClO$ $[M+H]^+$: 245.0733, found: 245.0730.

(2, 5-dimethylphenyl) (2-methylphenyl) methanone (2r):



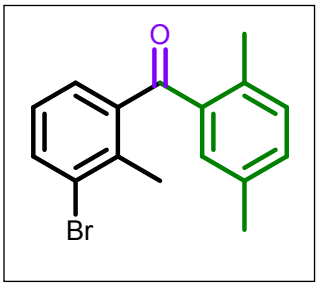
Starting material **2r** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a white solid (353.9 mg, 79%); The spectra data matched with values reported in the literature;[3] **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.31 (dd, J = 14.4, 7.4 Hz, 2H), 7.22 (d, J = 7.5 Hz, 1H), 7.17 – 7.07 (m, 4H), 2.44 (s, 3H), 2.36 (s, 3H), 2.23 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 197.6, 163.6, 139.1, 134.7, 132.8, 132.4, 130.6, 130.5, 130.4, 128.3, 113.6, 55.4, 20.8, 19.2; **IR (ν_{max}):** 3024, 2964, 2925, 2867, 1663, 1605, 1569, 1489, 1449, 1388, 1299, 1263, 1210, 1154, 1039, 950, 893, 858, 814, 759, 651 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{16}H_{16}O$ $[M+H]^+$: 225.1279, found: 225.1271.

(2,4-dichlorophenyl) (2,5-dimethylphenyl) methanone (2s):



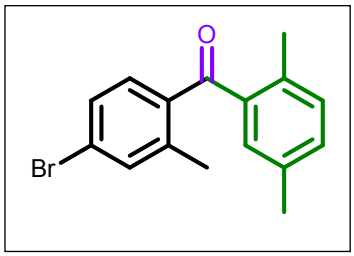
Starting material **2s** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide Pale brown solid (483.8 mg, 87%); The spectra data matched with values reported in the literature.[1] **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.41 (d, J = 1.8 Hz, 1H), 7.34 (d, J = 8.2 Hz, 1H), 7.28 (dd, J = 8.3, 1.9 Hz, 1H), 7.18 (dd, J = 18.5, 7.7 Hz, 2H), 7.10 (s, 1H), 2.48 (s, 3H), 2.25 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 196.0, 137.8, 136.7, 136.3, 136.2, 135.0, 132.85, 132.7, 131.7, 131.3, 130.8, 130.0, 126.9, 20.6, 20.5; **IR (ν_{max}):** 3024, 2925, 2863, 1668, 1578, 1456, 1377, 1297, 1264, 1210, 1142, 1106, 1051, 950, 861, 823, 778, 672 cm^{-1}; **HRMS (ESI)**: m/z calcd for $C_{15}H_{12}Cl_2O$ $[M+H]^+$: 279.0343, found: 279.0345.

(3-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanone (2t):



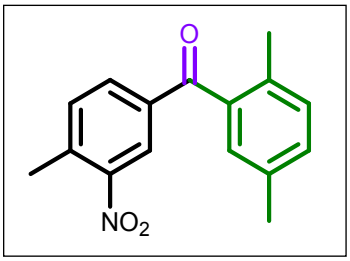
Starting material **2t** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a white solid (446.9 mg, 74%); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.64 (d, *J*=7.8, 1H), 7.18 (dd, *J*=16.1, 7.4, 3H), 7.11 (s, 1H), 7.05 (t, *J*=7.7, 1H), 2.45 (s, 3H), 2.41 (s, 3H), 2.25 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 199.61, 142.08, 137.39, 136.47, 135.96, 135.05, 134.44, 132.60, 131.66, 131.55, 127.88, 126.88, 126.58, 20.70, 20.58, 20.19; **IR (ν_{max}):** 2964, 2925, 2864, 1664, 1565, 1493, 1437, 1386, 1294, 1260, 1194, 1150, 1105, 1002, 953, 860, 810, 765, 721, 660 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{16}H_{16}BrO$ $[M+H]^+$: 303.0385, found: 303.0384.

(4-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanone (2u):



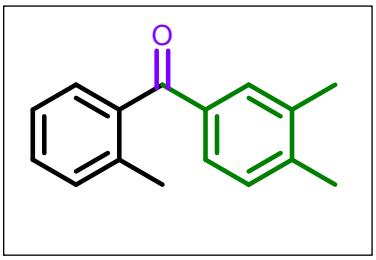
Starting material **2u** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a white solid (471.1 mg, 78%); **^{1}H NMR (500 MHz, $CDCl_3$)**: δ 7.44 (d, *J* = 1.2 Hz, 1H), 7.33 (dd, *J* = 8.2, 1.8 Hz, 1H), 7.20 – 7.13 (m, 3H), 7.08 (s, 1H), 2.41 (s, 3H), 2.36 (s, 3H), 2.28 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)**: δ 199.75, 140.31, 138.33, 137.75, 135.00, 134.98, 134.21, 132.03, 131.69, 131.38, 130.53, 128.51, 125.44, 20.75, 20.42, 20.13; **IR (ν_{max}):** 2965, 2925, 2864, 1663, 1618, 1579, 1485, 1443, 1387, 1296, 1261, 1206, 1148, 1093, 1038, 992, 948, 868, 818, 775, 665 cm^{-1}; **HRMS (ESI)**: m/z calcd for $C_{16}H_{16}BrO$ $[M+H]^+$: 303.0385, found: 303.0388.

(2,5-dimethylphenyl) (4-methyl-3-nitrophenyl) methanone (2v):



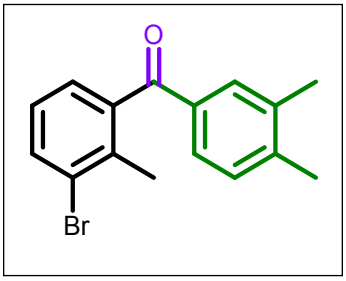
Starting material **2v** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a white solid (468.0 mg, 87%); **^{1}H NMR (400 MHz, $CDCl_3$)**: δ 8.34 (d, J = 1.7 Hz, 1H), 7.94 (dd, J = 7.9, 1.7 Hz, 1H), 7.47 (d, J = 7.9 Hz, 1H), 7.23 (dt, J = 16.4, 4.6 Hz, 2H), 7.11 (s, 1H), 2.67 (s, 3H), 2.34 (s, 3H), 2.29 (s, 3H**)**; **^{13}C NMR (101 MHz, $CDCl_3$)**: δ 196.0, 149.2, 138.3, 137.0, 136.9, 135.0, 133.8, 133.5, 133.0, 131.7, 131.2, 128.9, 126.0, 20.8, 20.5, 19.5; **IR (ν_{max}):** 2928, 2872, 1667, 1614, 1529, 1447, ,1391, 1348, 1299, 1259, 1204, 1156,1116, 1071, 1038, 967,907, 828, 781, 750, 710, 674 cm^{-1}; **HRMS (ESI)**: m/z calcd for $C_{16}H_{16}NO_3$ $[M+H]^+$: 270.1130, found: 270.1127.

(2-methylphenyl) (3,4-dimethylphenyl) methanone (2w):



Starting material **2w** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a white solid (322.5. mg, 72%); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.62 (s, 1H), 7.49 (dt, *J* = 10.6, 5.3 Hz, 1H), 7.36 (td, *J* = 7.4, 1.6 Hz, 1H), 7.28 (dd, *J* = 10.9, 4.7 Hz, 2H), 7.25 – 7.17 (m, 2H), 2.32 (s, 3H), 2.31 (s, 3H), 2.29 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ 198.49, 142.77, 139.03, 136.82, 136.31, 135.44, 130.92, 130.77, 129.83, 129.62, 128.12, 128.09, 125.05, 19.99, 19.81, 19.65; **IR (ν_{max}):** 3024, 2927, 1660, 1603, 1568, 1448, 1396, 1299, 1263, 1218, 1114, 1018, 963, 904, 842, 775, 738, 677 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{16}H_{16}O$ $[M+H]^+$: 225.1279, found: 225.1278.

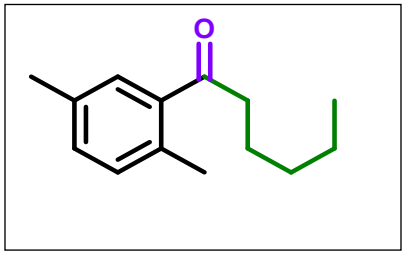
(3-bromo-2-methylphenyl) (3,4-dimethylphenyl) methanone (2x):



Starting material **2x** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a white solid (422.8 mg, 70%); **^{1}H NMR (500 MHz, $CDCl_3$):** δ 7.65 – 7.60 (m, 2H), 7.49 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.19 (t, *J* = 7.2 Hz, 2H), 7.09 (t, *J* = 7.7 Hz, 1H), 2.32 (s, 3H), 2.31 (s, 3H), 2.28 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 196.8, 143.1, 141.1, 136.8, 135.2, 134.5, 133.5, 130.6, 129.6, 127.9, 126.4, 126.3, 126.2, 20.0, 19.8, 19.4; **IR (ν_{max}):** 2928, 1661, 1602, 1565, 1439, 1395, 1292, 1262, 1213, 1139, 1124, 1011, 965, 908, 842, 734, 634 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{16}H_{16}BrO$ $[M+H]^+$: 303.0385, found: 303.0385.

1-(2,5-dimethylphenyl) hexan-1-one (2y):

Starting material **2y** was prepared according to general procedure 2.1. The crude material



was purified by silica gel column chromatography (hexane/ethyl acetate; 98:02) to provide a pale yellow liquid (315.1 mg, 78%); **^{1}H NMR (500 MHz, $CDCl_3$):** δ 7.28 (s, 1H), 7.03 – 6.89 (m, 2H), 2.72 (s, 2H), 2.31 (s, 3H), 2.21 (s, 3H), 1.57 (s, 2H), 1.22 (s, 4H), 0.79 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** 204.35, 138.03, 134.72, 134.26, 131.45, 131.38, 128.59, 128.54, 41.22, 31.27, 23.83, 22.32, 20.57, 20.39, 13.68; **IR (ν_{max}):** 2928, 2864, 1685, 1568, 1454, 1368, 1298, 1251, 1212, 1171, 1013, 969, 879, 815, 770, 669 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{14}H_{21}O$ $[M+H]^+$: 205.1592, found: 205.1609.

2.2. Photo-transfer hydrogenation reaction in batch process:

At first, 0.25 mmol of 2a was added to 50 ml borosilicate glass vials (dried by a heat gun) and then 5 mL water were added using a syringe. Then the glass was sealed by a septa and degassed with three freeze-pump-thaw cycles. After the mixture was thoroughly degassed, the glass vial placed in a light chamber for irradiation of light and their basic set-up has shown in Figure S1. The reaction mixture was allowed stir while the reaction proceeded. Resultant mixture was extracted with copious quantities of diethyl ether (DEE). The combined organic phase was concentrated under vacuum. The product was purified using flash column chromatography on 200-300 mesh silica gel with hexane/ethyl acetate as eluent. The results are summarized in the Table S1.

Note: Degassing is very crucial point of this reaction. If small amount of oxygen gas or air is available in reaction medium oxidized side product was observed.

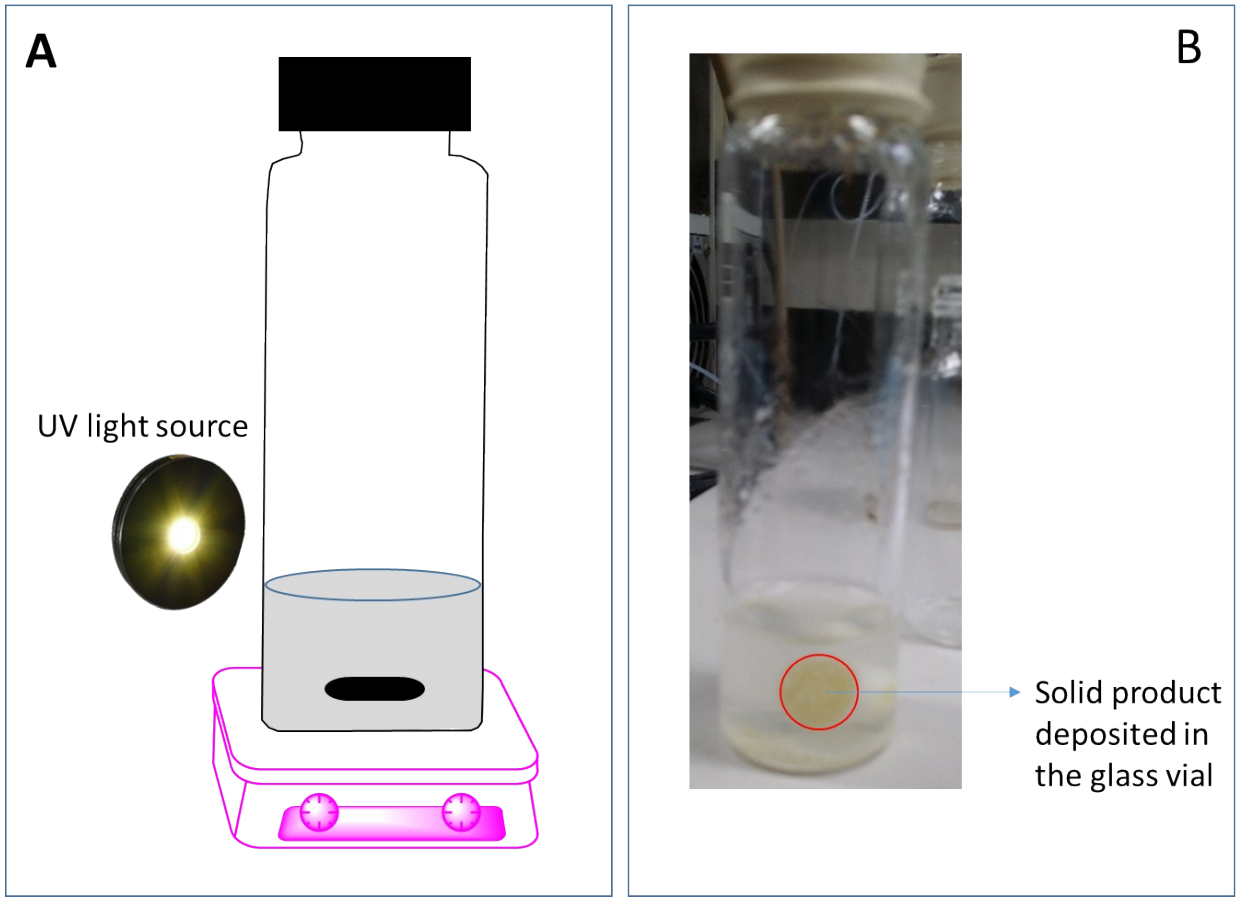
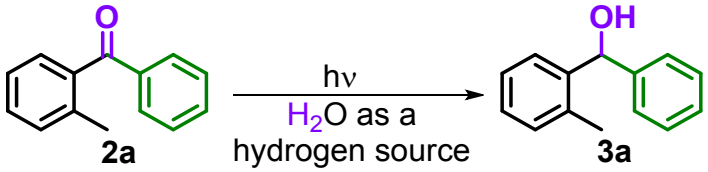


Figure S1: (A) Basic set-up for batch process photo-transfer hydrogenation reaction; (B) solid product precipitate in glass vials.

Table S1. Optimization of Batch reaction.



2a **3a**

Entry	Additive	Time (h)	Yield [%][a]
1	NA	12	9
2	NA	24	38
3	NA	48	52
4	NA	72	58
5	NA	98	59
6[b]	TiO_2 nanoparticle	24	3
7[c]	ACN	24	55
8[c]	ACN	48	72
9[c]	ACN	72	68
10[c]	DMSO	24	4

Reaction condition: Reactant (0.25 mmol), water 5 mL; (b) TiO_2 anatase nanopowder, <25 nm particle size (30 mg), (c) solvent 1 ml; Yield is based on GC analysis with anisole as internal standard.

2.2.1. Fabrication of homemade Photo-flow transfer hydrogenation (PTH) reactor design:

As shown in Figure S2, a Syrris asia pump was used to deliver the water solution (syringe 1, with various flow rate) and solution of phenyl (o-tolyl) methanone (0.0125M) in MeCN (syringe 2, with various flow rate), which was connected to the polytetrafluoroethylene (PTFE) tubing. The solution phase was mixed with water at a T-mixer, and the combined mixture was introduced to a perfluoroalkoxy (PFA) coil reactor (OD 1/16”, ID 1.0”, volume = 3.0 mL). The tubing reactor was wrapped within the helical grooves around a cylindrical-shaped frame. The reactor was cooled by circulating chilled water. To harvest maximum light finally tubing reactor was covered with aluminum foil. The cylindrical reactor was irradiated by medium pressure lamp beam of 250W Hg(Xe) arc lamp. The entire reactor system was covered by blue metal board. The final exiting product mixture was collected into a flask. As mentioned in Table 1, various reaction parameters (retention time, temperature, bulb power, pressure) were regulated to optimize reaction performance. Eventually, medium pressure lamp (250 W, max. medium pressure lamp), 29.0 min. retention time at RT generated the best yield 89% of phenyl(o-tolyl) methanol production (Table 1, entry 11).

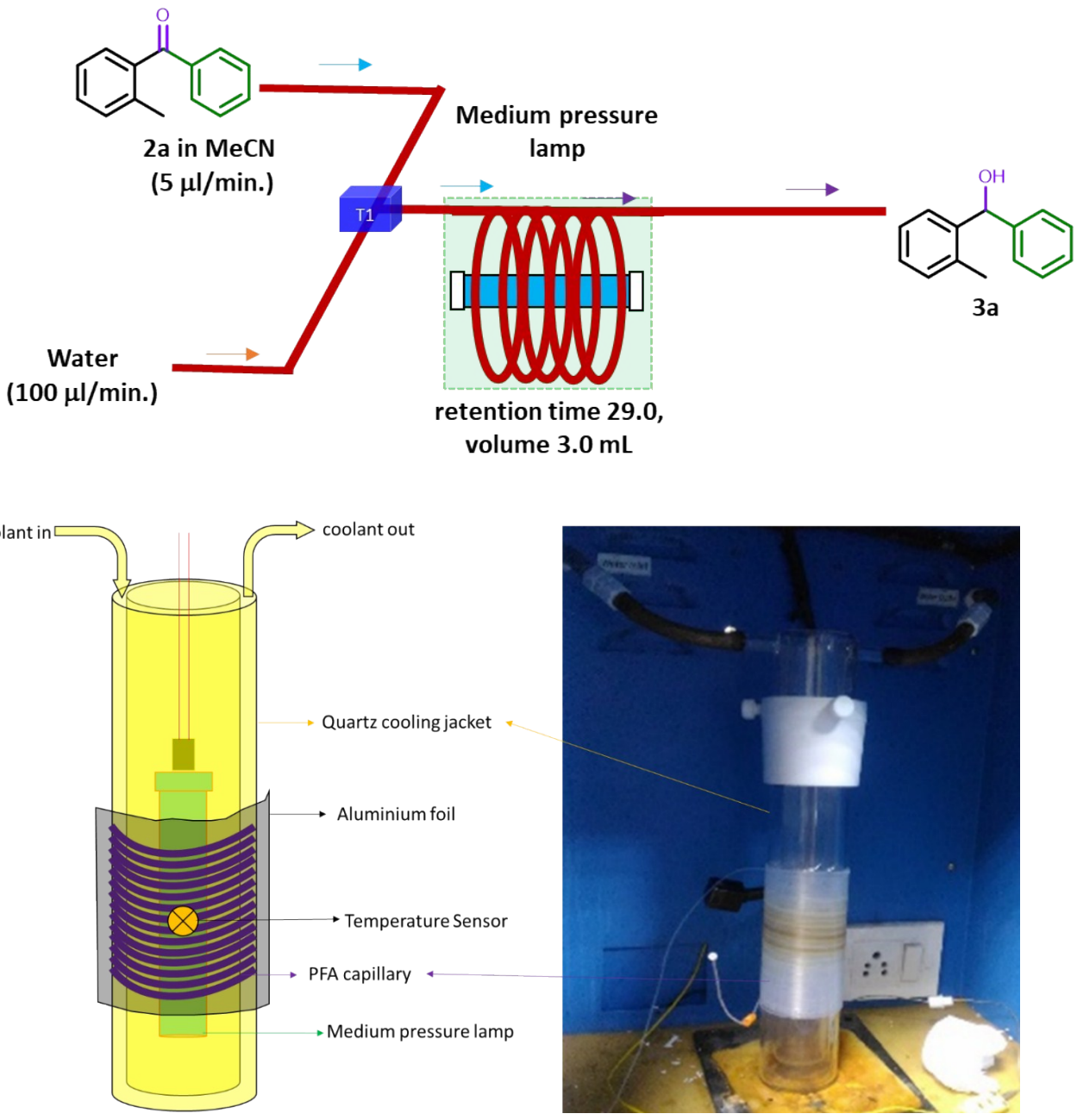


Figure S2: Basic set-up for photo-flow transfer hydrogenation reaction.

3. Typical procedure to extract and to separate the product in a PTHR platform:

3.1. Micro-separator design and work: To switch the solvent containing the product from MeCN to low volatile solvent DEE, the additional PTFE membrane embedded phase separator was connected to outlet of the photo-reactor as shown in Figure S3. The homemade microseparator was fabricated as following: to protect the metal corrosion, firstly, we were place the laser cutted PTFE grooves kit (60 mm x 60 mm x 2 μm thickness) as shown in Figure S2, with metal holder. Secondly, laser cutted PTFE (60 mm x 60 mm x 2 mm thickness) spiral line groove with rectangular shape (2 mm x 100 mm, volume 400 μl). The 4-corners of two PTFE film were holed (1 mm diameter) to align the film patterns. Polytetrafluoroethylene (PTFE) membrane (Whatmann, 0.45 μm pore, 37 mm dia.) was sandwiched by two PTFE sheets with identical dimension of groove channels, and aligned to each other by inserting metal pins through the holes at the film corners. Finally, the metal holder was tightly pressed by screw to seal the device with no leak. A serial process of droplet formation, extraction and separation for purification of the alcohol was conducted in droplet microfluidics equipped with the PTFE membrane microseparator, as explained in a step-wise manner at the below.

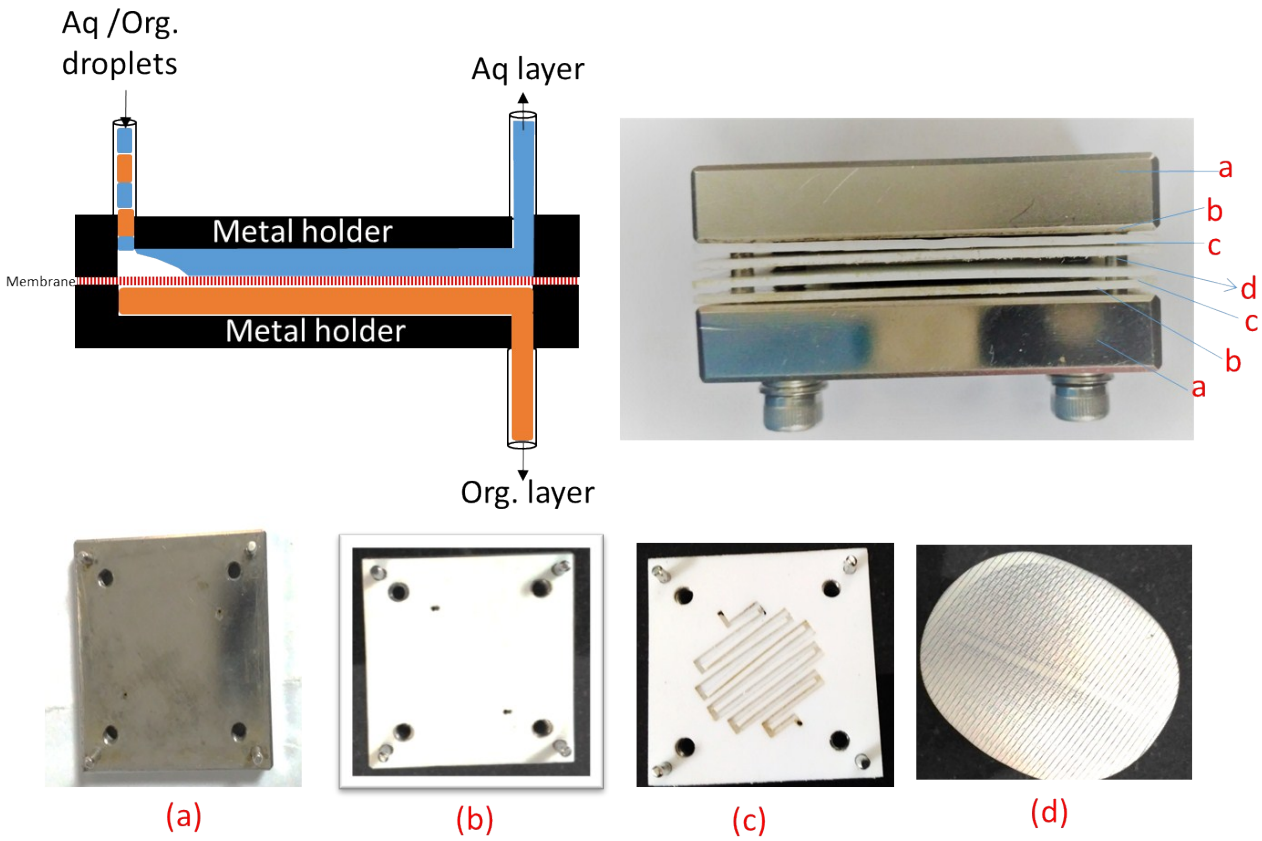
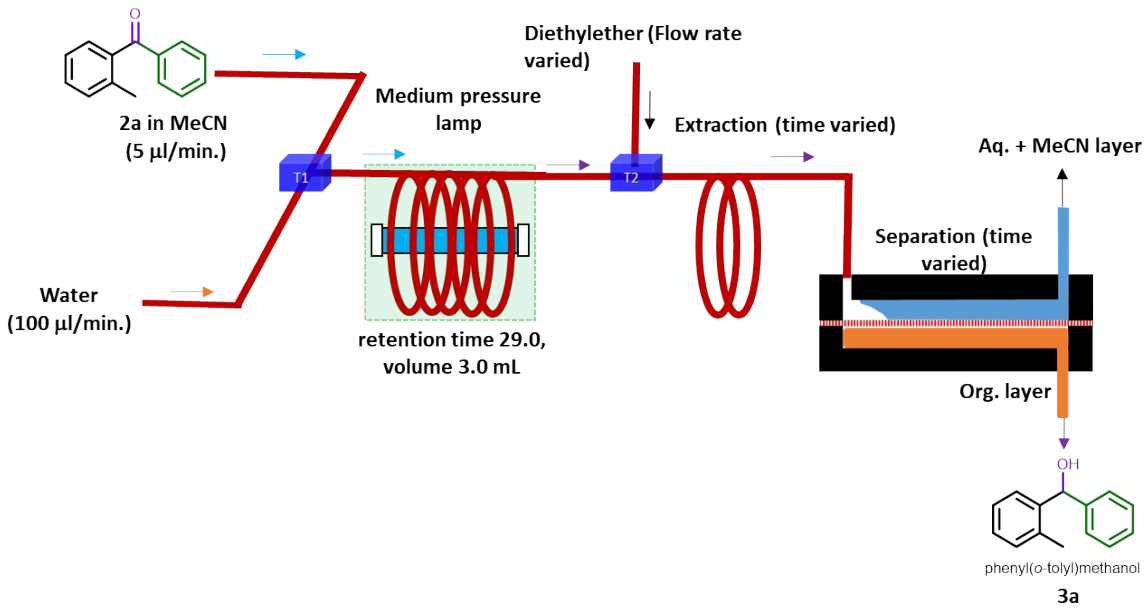


Figure S3. Illustration of a fluoropolymer PTFE membrane microseparation sandwiched between two PTFE films with laser grooved channel; (a) original image of metal holder; (b) original image of laser cutted PTFE grooves kit (60 mm x 60 mm x 2000 µm thickness); (c) original image of laser cutted PTFE (60 mm x 60 mm x 2mm thickness) grooves (single groove with zig-zag rectangular shape (2 mm x 2 mm x 100 mm, vol. = 400 µl); (d) original image of PTFE membrane (Whatmann, 0.45 µm pore, 37 mm diameter).

Step 1: Formation of alternating organic-aqueous droplets: Water was introduced into the product mixture in DEE through X-junction.

Step 2: Extraction: The MeCN solvent in the reaction mixture were gradually moved to aqueous droplet phase and real time extraction through a PTFE capillary (id = 500 µm, length = 2.5 m, vol. = 0.5 mL).

Step 3: Complete separation: The organic phase containing product could wet thin PTFE membrane and permeated to the opposite channel of the separator, whereas the waste containing aqueous phase did not wet the membrane and maintained at the original stream. The obtained product dissolved in DEE was analyzed by GC-MS, which showed a no ACN and as confirmed by absence of the corresponded peaks in NMR analysis (^{1}H and ^{13}C NMR spectra of **3a**). Point to be noted that there was no workup such as washing the product with aq. NH_4Cl, there was no need to be dried with Na_2SO_4. The reaction mixture was purified by column chromatography (hexane/ethyl acetate 95:5) to give the product 3a (40.5 mg, 82%).



Entry	DEE (Flow rate: µl/min)	%Yields (3a)
1	5	48
2	50	75
3	85	80
4	100	82

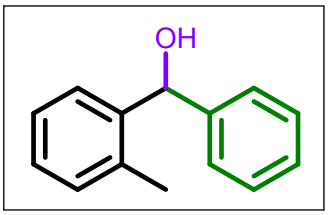
Yield is based on isolated yields;

Figure S4. Integrated process of in-line transfer photo-hydrogenation reaction, extraction and separation of synthesized phenyl(o-tolyl) methanol.

3.2 Typical procedure to extract and to separate the product in a PTHR platform:

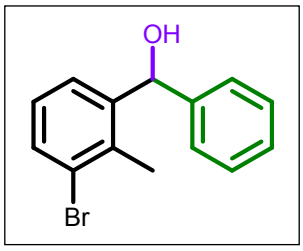
In-line, extraction and separation of hydrogenated product (S4): A solution of phenyl (o-tolyl) methanone (0.0125M) in MeCN taken in one bottles and de-oxygenated water was taken in another bottle and connected through the pump. Two reactants were introduced through T-mixer (T1) in a flow rate (20:1 ratio) of water and phenyl(o-tolyl) methanone to maintain the stoichiometry (Table 1), and then passed through a PFA tubing (id = 1000 µm, length = 3.9 m) to homemade photo-flow reactor and used medium pressure 250W lamp for the irradiation of light. As mentioned in Table 1, retention time 29.0 min., temperature 25±5 ^{o}C, power 250 W, and pressure 1 atm was the optimum reaction condition. The light irradiation might generate heat; therefore, the temperature of the reactor was maintained at room temperature by circulating chilled water in it. Next the reaction mixture was extracted by introducing DEE (flow rate 100 µl/min) through additional T2-mixer to form droplets, then the extraction process between the organic-aqueous segments was occurred for 2.4 min by flowing along PFA tubing (id = 500 µm, length = 2.5 m). The out-coming aq. organic phase was conducted in droplet microfluidics equipped with the PTFE dual channel microseparator. Out-flowing crude solution was again purified with column chromatography.

phenyl(o-tolyl) methanol (3a):



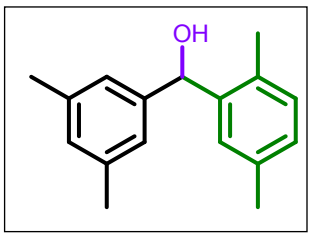
Compound **3a** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 40.5 mg (82% Yield); **m.p.:** 114 ºC; The spectra data matched with values reported in the literature;[4] **^{1}H NMR (300 MHz, $CDCl_3$):** δ 7.55 – 7.47 (m, 1H), 7.35 – 7.11 (m, 8H), 6.00 (d, *J* = 3.9 Hz, 1H), 2.24 (s, 3H), 2.14 (d, *J* = 3.9 Hz, 1H); **^{13}C NMR (75 MHz, $CDCl_3$):** δ 142.90, 141.46, 135.40, 130.57, 128.49, 127.59, 127.56, 127.12, 126.30, 126.15, 77.47, 77.04, 76.62, 73.42, 19.41; **IR (ν_{max}):** 3222, 3023, 2965, 2880, 1598, 1480, 1459, 1345, 1292, 1249, 1213, 1024, 865, 832, 758, 732, 704 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{14}H_{14}O$ $[M+H-H_2O]^+$: 181.1017, found: 181.1020.

(3-bromo-2-methylphenyl) (Phenyl) methanol (3b):



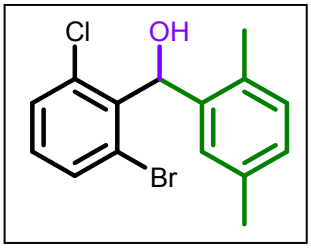
Compound **3b** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 53.1 mg (77% Yield); **m.p.:** 74 ºC; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.44 (dd, *J* = 7.9, 1.1 Hz, 1H), 7.36 (d, *J* = 7.7 Hz, 1H), 7.25 – 7.20 (m, 3H), 7.17 – 7.13 (m, 2H), 6.99 (t, *J* = 7.8 Hz, 1H), 5.76 (s, 1H), 3.05 (s, 1H), 2.17 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 143.21, 142.11, 134.89, 131.64, 128.44, 127.66, 127.05, 126.95, 126.20, 125.49, 73.57, 18.85; **IR (ν_{max}):** 3317, 3066, 3029, 2923, 1565, 1486, 1445, 1385, 1229, 1176, 1126, 1080, 1004, 862, 770, 702 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{14}H_{13}BrO$ $[M+H-H_2O]^+$: 259.0122, found: 259.0118

(2,5-dimethylphenyl) (3,5-dimethylphenyl) methanol (3c):



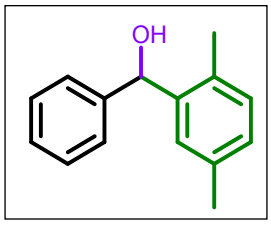
Compound **3c** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 50.4 mg (84% Yield); **m.p.:** 95 ºC; **^{1}H NMR (500 MHz, $CDCl_3$):** δ 7.39 (s, 1H), 7.06 (s, 2H), 6.96 (d, *J* = 14.5 Hz, 3H), 5.91 (s, 1H), 2.39 (s, 3H), 2.33 (s, 6H), 2.25 (d, *J* = 5.8 Hz, 1H), 2.24 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ 142.88, 141.27, 137.87, 135.41, 132.04, 130.30, 129.12, 127.96, 126.76, 124.77, 73.25, 21.29, 21.15, 18.92; **IR (ν_{max}):** 3341, 3014, 2920, 2868, 1606, 1493, 1458, 1381, 1289, 1231, 1156, 1113, 1040, 897, 854, 807, 779, 758, 716, 679 cm^{-1}; **HRMS (ESI):** m/z calcd. for $C_{17}H_{20}O$ $[M+H-H_2O]^+$: 223.1487, found: 223.1497.

(2-bromo-6-chlorophenyl) (2,5-dimethylphenyl) methanol (3d).



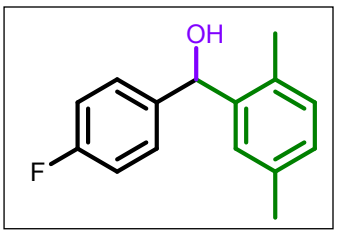
Compound **3d** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 54.3 mg (67% Yield); **m.p.:** 128 ºC; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.64 (d, *J* = 1.7 Hz, 1H), 7.40 – 7.33 (m, 1H), 7.21 (d, *J* = 8.4 Hz, 1H), 7.05 (dd, *J* = 18.9, 7.6 Hz, 2H), 6.97 (s, 1H), 6.17 (d, *J* = 6.0 Hz, 1H), 2.53 (s, 1H), 2.31 (s, 3H), 2.28 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)**: δ 142.63, 138.97, 135.82, 132.95, 131.91, 131.79, 131.41, 130.98, 130.63, 128.93, 126.99, 120.95, 69.64, 21.21, 18.69; **IR (ν_{max}):** 3275, 3013, 2923, 1499, 1453, 1336, 1354, 1291, 1249, 1220, 1123, 1023, 892, 810, 754 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{14}ClBrO$ $[M+H-H_2O]^+$: 306.9889, found: 306.9889.

(2,5-dimethylphenyl) (phenyl)methanol (3e):



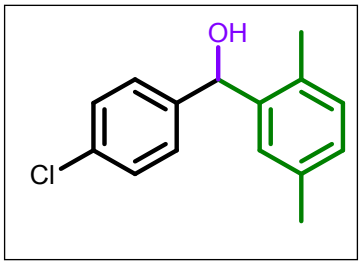
Compound **3e** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 45.8 mg (87% Yield); The spectra data matched with values reported in the literature;[5] **m.p.:** 109 ºC; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.28 (dd, *J* = 4.4, 1.3 Hz, 5H), 7.25 – 7.21 (m, 1H), 7.02 – 6.95 (m, 2H), 5.89 (s, 1H), 2.33 (s, 1H), 2.30 (s, 3H), 2.15 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$)**: δ 142.92, 141.15, 135.49, 132.08, 130.40, 128.37, 128.11, 127.40, 127.01, 126.84, 73.24, 21.12, 18.86; **IR (ν_{max}):** 3340, 3027, 2921, 1495, 1451, 1385, 1294, 1236, 1188, 1160, 1024, 1113, 1021, 809, 753, 701, 660 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{16}O$ $[M+H-H_2O]^+$: 195.1174, found: 195.1169.

(2,5-dimethylphenyl) (4-fluorophenyl) methanol (3f):



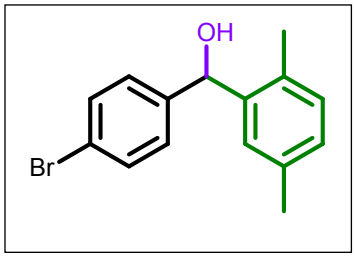
Compound **3f** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 44.5 mg (78% Yield); **m.p.:** 110 ºC; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.27 – 7.13 (m, 3H), 6.94 (dd, *J* = 19.0, 10.3 Hz, 4H), 5.78 (s, 1H), 2.82 (s, 1H), 2.29 (s, 3H), 2.10 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ 163.13, 161.17, 141.15, 138.83, 135.72, 132.15, 130.64, 128.85 (d, *J* = 8.2 Hz), 128.38, 126.87, 115.35, 115.18, 72.67, 21.23, 18.91; **^{19}F NMR (376 MHz, $CDCl_3$):** δ -114.99; **IR (ν_{max}):** 3297, 3032, 2924, 1605, 1504, 1452, 1292, 1223, 1157, 1106, 1023, 825, 750, 655 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{15}FO$ $[M+H-H_2O]^+$: 213.1080, found: 213.1077.

(2,5-dimethylphenyl) (4-chlorophenyl) methanol (3g):



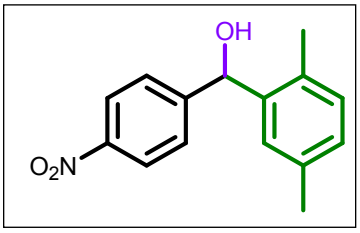
Compound 3**g** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide white solid; 44.3mg (72% Yield); **m.p.:** 131 ºC; **^{1}H NMR (500 MHz, $CDCl_3$):** δ 7.33 – 7.29 (m, 2H), 7.27 (dd, *J* = 5.8, 2.8 Hz, 3H), 7.08 – 7.03 (m, 2H), 5.94 (d, *J* = 3.5 Hz, 1H), 2.35 (s, 3H), 2.26 (d, *J* = 3.8 Hz, 1H), 2.22 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ 141.42, 140.79, 135.73, 133.15, 132.12, 130.60, 128.50, 128.43, 128.35, 126.92, 72.66, 21.11, 18.84; **IR (ν_{max}):** 3204, 3020, 2964, 2915, 1490, 1450, 1399, 1289, 1247, 1247, 1093, 1025, 857, 820, 779, 710, 673 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{15}ClO$ $[M+H-H_2O]^+$: 229.0796, found: 229.0784.

(2,5-dimethylphenyl) (4-bromophenyl) methanol (3h):



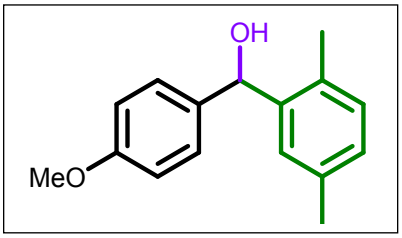
Compound 3**h** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide white solid; 48.6 mg (67% Yield); **m.p.:** 127 ºC; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.47 – 7.42 (m, 2H), 7.25 (d, *J* = 4.3 Hz, 1H), 7.19 (d, *J* = 8.4 Hz, 2H), 7.07 – 7.01 (m, 2H), 5.89 (s, 1H), 2.43 (s, 1H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 141.90, 140.69, 135.68, 132.10, 131.40, 130.57, 128.66, 128.41, 126.91, 121.27, 72.62, 21.10, 18.84; **IR (ν_{max}):** 3550, 3340, 3023, 2958, 2918, 1491, 1451, 1405, 1292, 1224, 1163, 1112, 1071, 1020, 816, 772 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{15}BrO$ $[M+H-H_2O]^+$: 273.0279, found: 273.0278.

(2,5-dimethylphenyl) (4-nitrophenyl) methanol (3i):



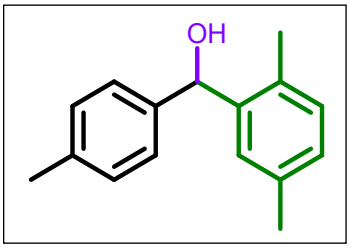
Compound **3i** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 25.0 mg (39% Yield); **m.p.:** 123 ºC; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 8.12 (d, *J* = 8.9 Hz, 2H), 7.48 (d, *J* = 8.7 Hz, 2H), 7.12 (s, 1H), 7.05 (q, *J* = 7.8 Hz, 2H), 5.99 (d, *J* = 3.5 Hz, 1H), 2.96 (d, *J* = 3.8 Hz, 1H), 2.29 (s, 3H), 2.24 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)**: δ 150.39, 146.76, 140.04, 135.81, 132.19, 130.74, 128.78, 127.39, 127.30, 123.32, 72.31, 20.88, 18.71; **IR (ν_{max}):** 3414, 3020, 2927, 2965, 2880, 1602, 1516, 1452, 1343, 1110, 862, 813, 746, 664 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{15}NO_3$ $[M+H-H_2O]^+$: 240.1025, found: 240.1022.

(2, 5-dimethylphenyl) (4-methoxyphenyl) methanol (3j):



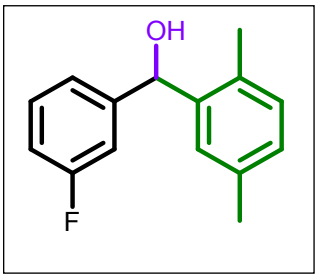
Compound **3j** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 53.2 mg (88% Yield); **m.p.:** 69 ºC; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.35 (s, 1H), 7.21 – 7.15 (m, 2H), 6.98 (d, *J* = 1.0 Hz, 2H), 6.84 – 6.77 (m, 2H), 5.84 (s, 1H), 3.74 (s, 3H), 2.34 (s, 1H), 2.32 (s, 3H), 2.12 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$)**: δ 158.81, 141.34, 135.35, 135.16, 131.86, 130.28, 128.35, 127.87, 126.48, 113.69, 72.73, 55.12, 21.11, 18.77; **IR (ν_{max}):** 3441, 2928, 1613, 1509, 1456, 1382, 1298, 1247, 1174, 1111, 1031, 823 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{16}H_{18}O_2$ $[M+H-H_2O]^+$: 225.1279, found: 225.1274.

(2,5-dimethylphenyl) (p-tolyl) methanone (3k):



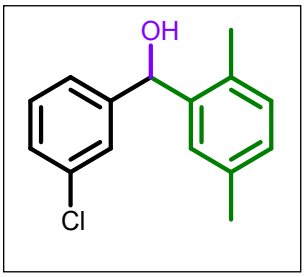
Compound **3k** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 39.0 mg (69% Yield); **m.p.:** 103 ºC; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.29 (s, 1H), 7.12 (d, *J* = 8.1 Hz, 2H), 7.06 (d, *J* = 7.9 Hz, 2H), 6.95 (s, 2H), 5.78 (s, 1H), 2.49 (s, 1H), 2.28 (d, *J* = 3.1 Hz, 6H), 2.10 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 141.59, 140.28, 137.16, 135.55, 132.19, 130.51, 129.22, 128.14, 127.26, 126.94, 73.15, 21.35, 21.27, 19.05; **IR (ν_{max}):** 3554, 3445**,** 3412, 3345, 3228, 3097, 3021, 2927, 2862, 1507, 1458, 1295, 1168, 1111, 1023, 819, 779, cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{16}H_{18}O$ $[M+H-H_2O]^+$: 209.1230, found: 209.1220.

(2, 5-dimethylphenyl) (3-fluorophenyl) methanol (3l):



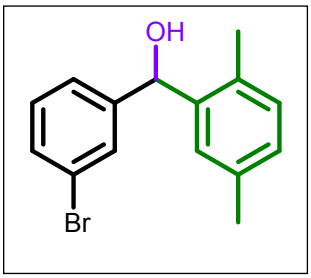
Compound **3l** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 90:10) to provide a white solid; 43.1 mg (75% Yield); **m.p.:** 81 ºC; **^{1}H NMR (500 MHz, $CDCl_3$):** δ 7.24 – 7.11 (m, 2H), 6.96 (t, *J* = 11.1 Hz, 4H), 6.87 (t, *J* = 8.3 Hz, 1H), 5.74 (s, 1H), 2.91 (d, *J* = 12.4 Hz, 1H), 2.25 (s, 3H), 2.12 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ 163.98, 162.02, 145.90, 145.85, 140.87, 135.79, 132.36, 130.73, 129.95, 129.88, 128.59, 127.26, 122.70, 122.69, 114.39, 114.22, 114.09, 113.91, 72.66, 21.22, 18.94; **^{19}F NMR (376 MHz, $CDCl_3$):** δ -112.87; **IR (ν_{max}):** 3342, 3019, 2925, 1598, 1489, 1447, 1241, 1124, 1027, 947, 881, 762, 695 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{15}FO$ $[M+H-H_2O]^+$: 213.1080, found: 213.1083

(2, 5-dimethylphenyl) (3-chlorophenyl) methanol (3m):



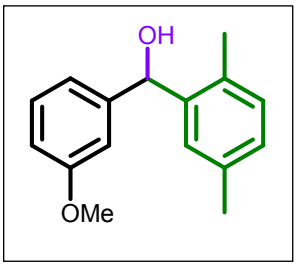
Compound **3m** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 90:10) to provide a yellow liquid; 43.7 mg (71% Yield); **^{1}H NMR (500 MHz, $CDCl_3$):** δ 7.25 (s, 1H), 7.18 – 7.12 (m, 3H), 7.09 (t, *J* = 6.0 Hz, 1H), 6.99 – 6.94 (m, 2H), 5.73 (s, 1H), 2.86 (d, *J* = 25.8 Hz, 1H), 2.26 (s, 3H), 2.12 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ 144.98, 140.47, 135.54, 134.10, 132.08, 130.48, 129.46, 128.36, 127.35, 126.99, 126.92, 124.98, 77.00, 72.38, 21.00, 18.74; **IR (ν_{max}):** 3318, 3019, 2923, 1583, 1465, 1428, 1289, 1227, 1189, 1087, 1026, 890, 805, 756, 693 cm^{-1}; **HRMS (ESI)**: m/z calcd for $C_{15}H_{15}ClO$ $[M+H-H_2O]^+$: 229.0784, found: 229.0772.

(2, 5-dimethylphenyl) (3-bromophenyl) methanol (3n):



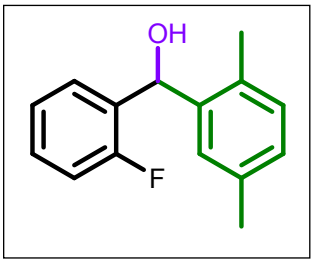
Compound **3n** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a pale brown liquid; 50.2 mg (69% Yield); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.35 (s, 1H), 7.26 (dt, *J* = 7.5, 1.7 Hz, 1H), 7.07 (s, 1H), 7.04 – 6.97 (m, 2H), 6.92 (d, *J* = 7.1 Hz, 2H), 5.55 (s, 1H), 3.67 (s, 1H), 2.19 (s, 3H), 2.04 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 145.13, 140.26, 135.28, 131.93, 130.31, 130.06, 129.66, 129.59, 128.18, 126.90, 125.30, 122.21, 72.00, 20.91, 18.64; **IR (ν_{max}):** 3341, 3013, 2923, 1577, 1463, 1425, 1288, 1219, 1184, 1108, 1026, 892, 809, 753, 699 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{15}BrO$ $[M+H-H_2O]^+$: 273.0279, found: 273.0279.

(2, 5-dimethylphenyl) (3-bromophenyl) methanol (3o):



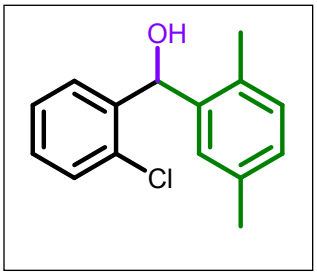
Compound **3o** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a yellow liquid; 50.8 mg (84% Yield); **^{1}H NMR (500 MHz, $CDCl_3$):** δ 7.25 (s, 1H), 7.18 (t, *J*=7.9, 1H), 7.01 – 6.94 (m, 2H), 6.89 – 6.81 (m, 2H), 6.76 (dd, *J*=8.1, 2.0, 1H), 5.84 (s, 1H), 3.72 (s, 3H), 2.50 (s, 1H), 2.28 (s, 3H), 2.17 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 159.48, 144.66, 141.03, 135.36, 132.06, 130.30, 129.25, 128.03, 126.86, 119.31, 112.70, 112.52, 72.93, 55.00, 21.03, 18.79; **IR (ν_{max}):** 3422, 2929, 1596, 1486 1449, 1385, 1391, 1254, 1151, 1034, 878, 767, 699 cm^{-1}; **HRMS (ESI)**: m/z calcd for $C_{15}H_{15}O_2$ $[M+H-H_2O]^+$: 225.1279, found: 225.1274.

(2, 5-dimethylphenyl) (2-fluorophenyl) methanol (3p):



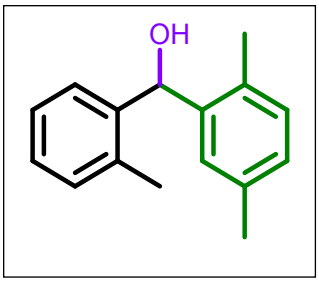
Compound **3p** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 40.3 mg (70% Yield); **m.p.:** 101 ºC; **^{1}H NMR (500 MHz, $CDCl_3$):** δ 7.33 – 7.19 (m, 3H), 7.07 (t, *J* = 6.6 Hz, 1H), 7.01 (s, 3H), 6.23 (s, 1H), 2.54 (d, *J* = 11.7 Hz, 1H), 2.29 (s, 3H), 2.20 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$)**: δ 161.34, 159.37, 140.24, 135.59, 132.41, 130.54, 130.41, 130.31, 129.23, 129.17, 128.54, 128.52, 128.46, 127.01, 124.31, 124.28, 115.42, 115.25, 66.71, 66.69, 21.28, 18.63; **^{19}F NMR (376 MHz, $CDCl_3$):** δ -118.38; **IR (ν_{max}):** 3334, 3013, 2926, 1585, 1490, 1452, 1225, 1163, 1104, 1024, 849, 807, 755, 653 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{15}FO$ $[M+H-H_2O]^+$: 213.1080, found: 213.1077.

(2, 5-dimethylphenyl) (2-chlorophenyl) methanol (3q):



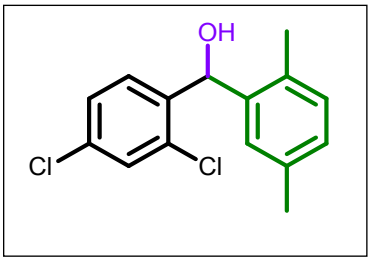
Starting material **3q** was prepared according to general procedure 2.1. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 43.7 mg (71% Yield); **m.p.:** 141 ºC; **^{1}H NMR (400 MHz, $CDCl_3$)**: δ 7.39 – 7.31 (m, 2H), 7.25 – 7.19 (m, 2H), 7.13 (s, 1H), 7.03 (t, *J* = 7.1 Hz, 2H), 6.29 (d, *J* = 4.2 Hz, 1H), 2.28 (s, 3H), 2.26 (s, 1H), 2.22 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 140.40, 139.69, 135.57, 133.27, 132.73, 130.46, 129.57, 128.88, 128.61, 128.48, 127.02, 126.96, 69.84, 21.22, 18.67; **IR (ν_{max}):** 3118, 3027, 2962, 2917, 2861, 1499, 1444, 1343, 1252, 1154, 1117, 1027, 813, 756, 710 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{15}ClO$ $[M+H-H_2O]^+$: 229.0784, found: 229.0795.

(2, 5-dimethylphenyl) (2-methylphenyl) methanol (3r):



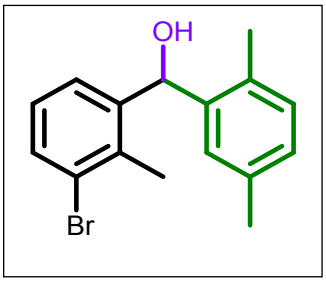
Starting material **3r** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 20.9 mg (37% Yield); **m.p.:** 134 ºC; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.32 – 7.28 (m, 1H), 7.23 – 7.17 (m, 3H), 7.14 (s, 1H), 7.05 (dt, *J* = 8.6, 4.3 Hz, 2H), 6.08 (d, *J* = 4.5 Hz, 1H), 2.31 (s, 3H), 2.30 (s, 3H), 2.23 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ 140.82, 140.52, 135.80, 135.43, 132.53, 130.36, 130.31, 128.14, 127.45, 127.02, 126.44, 125.99, 70.11, 21.13, 19.01, 18.53; **IR (ν_{max}):** 3189, 3024, 2960, 2914, 2861, 1490, 1446, 1379, 1340, 1291, 1249, 1213, 1152, 1108, 1029, 940, 885, 808, 749, 716 cm^{-1}; **HRMS (ESI)**: m/z calcd for $C_{16}H_{18}O$ $[M+H-H_2O]^+$: 209.1330, found: 209.1366.

(2,4-dichlorophenyl) (2,5-dimethylphenyl) methanol (3s):



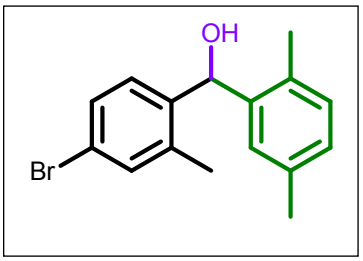
Starting material **3s** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 47.0 mg (67% Yield); **m.p.:** 143 ºC; **^{1}H NMR (500 MHz, $CDCl_3$)**: δ 7.38 (d, *J* = 2.1 Hz, 1H), 7.35 (d, *J* = 8.4 Hz, 1H), 7.24 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.09 – 7.01 (m, 3H), 6.20 (d, *J* = 4.0 Hz, 1H), 2.47 (d, *J* = 4.1 Hz, 1H), 2.29 (s, 3H), 2.25 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ 139.19, 139.02, 135.63, 133.81, 133.68, 132.65, 130.49, 129.42, 129.24, 128.63, 127.19, 126.80, 69.34, 21.10, 18.54; **IR (ν_{max}):** 3186, 2974, 2919, 2862, 1577, 1498, 1457, 1379, 1338, 1293, 1250, 1198, 1145, 1102, 1027, 859, 803, 779, 712 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{15}H_{14}Cl_2O$ $[M+H-H_2O]^+$: 263.0394, found: 263.0393.

(3-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanol (3t):



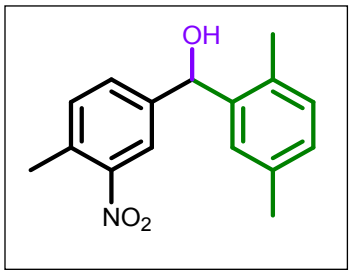
Compound **3t** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 31.3 mg (41% Yield); **m.p.:** 141 ºC; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.52 (d, *J* = 7.9 Hz, 1H), 7.29 (d, *J* = 7.7 Hz, 1H), 7.09 – 7.00 (m, 4H), 6.09 (s, 1H), 2.33 (s, 3H), 2.28 (s, 3H), 2.24 (s, 3H), 2.02 (s, 1H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 142.94, 140.08, 135.69, 135.46, 132.59, 131.81, 130.52, 128.52, 127.01, 126.22, 125.76, 77.00, 70.68, 21.13, 18.64, 18.55; **IR (ν_{max}):** 3261, 3013, 2923, 1565, 1496, 1444, 1384, 1337, 1286, 1220, 1163, 1122, 1026, 811, 763, 718, 678, 632 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{16}H_{17}BrO$ $[M+H-H_2O]^+$: 287.0435, found: 287.0434.

(4-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanol (3u):



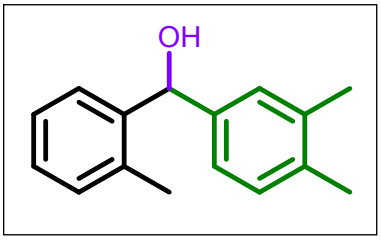
Compound **3u** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 34.3 mg (45% Yield); **m.p.:** 125 ºC; **^{1}H NMR (400 MHz, $CDCl_3$):**δ 7.52 (d, *J* = 7.9 Hz, 1H), 7.29 (d, *J* = 7.7 Hz, 1H), 7.09 – 7.00 (m, 4H), 6.09 (s, 1H), 2.33 (s, 3H), 2.28 (s, 3H), 2.24 (s, 3H), 2.02 (s, 1H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 142.94, 140.08, 135.69, 135.46, 132.59, 131.81, 130.52, 128.52, 127.01, 126.22, 125.76, 77.00, 70.68, 21.13, 18.64, 18.55; **IR (ν_{max}):** 3271, 3016, 2921, 1594, 1485, 1448, 1390, 1289, 1213, 1163, 1103, 1024, 866, 805, 757, 651 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{16}H_{17}BrO$ $[M+H-H_2O]^+$: 287.0435, found: 287.0359.

(2,5-dimethylphenyl) (4-methyl-3-nitrophenyl) methanol (3v):



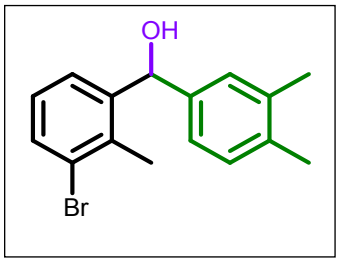
Compound **3v** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 35.9 mg (53% Yield); **m.p.:** **93** ºC; **^{1}H NMR (500 MHz, $CDCl_3$):** δ 7.87 (d, *J* = 1.2 Hz, 1H), 7.32 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.18 (d, *J* = 7.9 Hz, 1H), 7.11 (s, 1H), 7.00 – 6.93 (m, 2H), 5.81 (s, 1H), 3.35 (s, 1H), 2.49 (s, 3H), 2.24 (s, 3H), 2.15 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ 148.75, 142.49, 140.05, 135.60, 132.49, 132.14, 131.99, 131.15, 130.53, 128.51, 126.97, 122.63, 71.73, 20.85, 19.88, 18.62; **IR (ν_{max}):** 3344, 2927, 1617, 1527, 1451, 1345, 1201, 1156, 1034, 900, 821, 769 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{16}H_{17}NO_3$ $[M+H-H_2O]^+$: 254.1181, found: 254.1195.

(3,4-dimethylphenyl) (o-tolyl) methanol (3w):



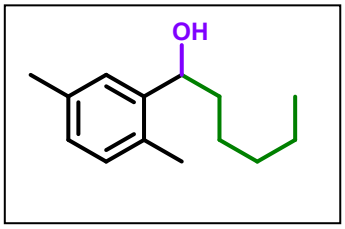
Compound **3w** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a pale brown liquid; 39.0 mg (69% Yield); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.70 (d, *J*=7.3, 1H), 7.43 – 7.34 (m, 2H), 7.27 (dd, *J*=19.9, 7.3, 3H), 7.17 (d, *J*=7.7, 1H), 5.95 (s, 1H), 3.16 (s, 1H), 2.43 (s, 3H), 2.41 (s, 3H), 2.37 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$):** δ 141.55, 140.26, 136.15, 135.34, 134.90, 130.05, 129.33, 128.28, 126.90, 125.92, 125.72, 124.47, 72.62, 19.56, 19.20, 19.11; **IR (ν_{max}):** 3381, 3333, 3023, 2930, 1494, 1386, 1290, 1215, 1118, 1028, 829, 758 cm^{-1}; **HRMS (ESI)**: m/z calcd for $C_{16}H_{18}O$ $[M+H-H_2O]^+$: 209.1330, found: 209.1360.

(3-bromo-2-methylphenyl) (3,4-dimethylphenyl) methanol (3x):



Compound **3x** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 51.1 mg (67% Yield); **m.p.:** 103 ºC; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.57 – 7.50 (m, 2H), 7.14 – 7.05 (m, 3H), 6.99 (dd, *J* = 7.7, 1.7 Hz, 1H), 5.92 (s, 1H), 2.32 (d, *J* = 3.1 Hz, 1H), 2.29 (s, 3H), 2.25 (s, 3H), 2.24 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 143.51, 139.81, 136.85, 136.29, 134.86, 131.59, 129.77, 128.38, 127.01, 126.22, 125.38, 124.56, 77.00, 73.72, 19.82, 19.44, 18.93; **IR (ν_{max}):** 3333, 3010, 2928, 1567, 1500, 1446, 1385, 1326, 1218, 1163, 1127, 1031, 1000, 829, 768, 720, 679 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{16}H_{17}BrO$ $[M+H-H_2O]^+$: 287.0435, found: 287.0430.

1-(2,5-dimethylphenyl) hexan-1-ol (3y):



Compound **3y** was prepared according to general procedure 2.3. The crude material was purified by silica gel column chromatography (hexane/ethyl acetate; 95:05) to provide a white solid; 25.2 mg (49% Yield); **m.p.:** 77 ºC; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.57 – 7.50 (m, 2H), 7.14 – 7.05 (m, 3H), 6.99 (dd, *J* = 7.7, 1.7 Hz, 1H), 5.92 (s, 1H), 2.32 (d, *J* = 3.1 Hz, 1H), 2.29 (s, 3H), 2.25 (s, 3H), 2.24 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 143.51, 139.81, 136.85, 136.29, 134.86, 131.59, 129.77, 128.38, 127.01, 126.22, 125.38, 124.56, 77.00, 73.72, 19.82, 19.44, 18.93; **IR (ν_{max}):** 3345, 2927, 2862, 1500, 1456, 1379, 1301, 1149, 1123, 1048, 811, 770 cm^{-1}; **HRMS (ESI):** m/z calcd for $C_{14}H_{22}O$ $[M+H-H_2O]^+$: 189.1643, found: 189.1642.

Control experiments for mechanistic study: A solution of phenyl (o-tolyl) methanone (0.0125M) in MeCN was mix with 10 eq. of (2,2,6,6-Tetramethylpiperidin-1-yl)oxyl (TEMPO) taken in one bottles and de-oxygenated water was taken in another bottle and connected through the pump. Two reactants were introduced through T-mixer (T1) in a flow rate (20:1 ratio) of water and phenyl(o-tolyl) methanone to maintain the stoichiometry (Table 1), and then passed through a PFA tubing (id = 1000 μm, length = 3.9 m) to homemade photo-flow reactor and used medium pressure 250W lamp for the irradiation of light. As mentioned in Table 1, retention time 29.0 min., temperature 25±5 ºC, power 250 W, and pressure 1 atm was the optimum reaction condition. The light irradiation might generate heat; therefore, the temperature of the reactor was maintained at room temperature by circulating chilled water in it. Next the reaction mixture was extracted by introducing DEE (flow rate 100 μl/min) through additional T2-mixer to form droplets, then

the extraction process between the organic-aqueous segments was occurred for 2.4 min by flowing along PFA tubing (id = 500 µm, length = 2.5 m). The out-coming aq. organic phase was conducted in droplet microfluidics equipped with the PTFE dual channel microseparator. Obtained organic layer was analysed by the GC-MS but it's showed no product formation.

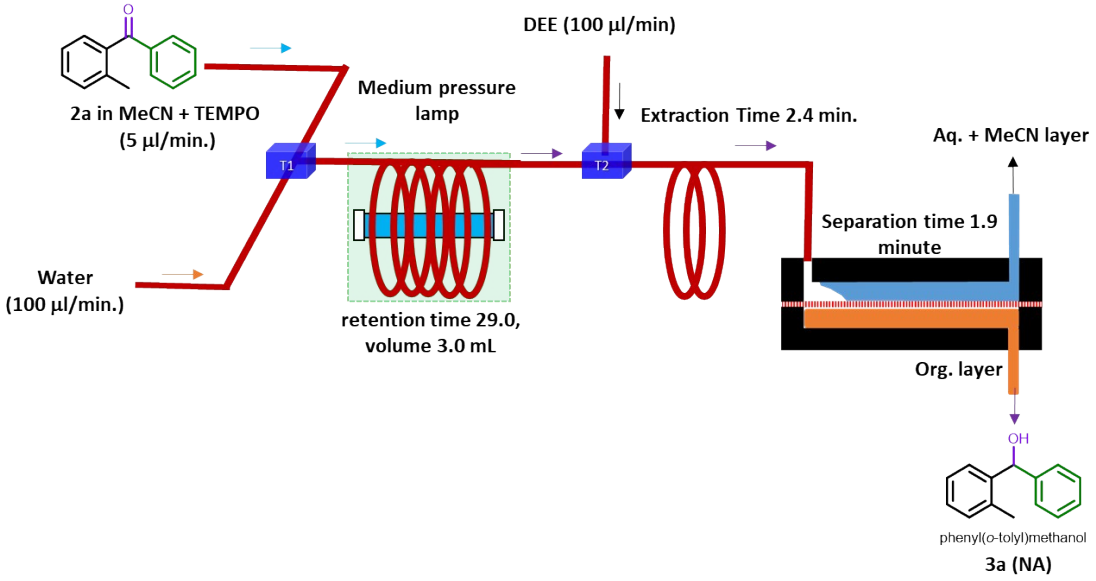


Figure S5. Free radical-trapping Integrated process set-up.

Isotopic labelling experiment in batch system:

At first, 0.25 mmol of 2a was added to 5 ml borosilicate glass vials (dried by a heat gun) and then 1 mL D_2O were added using a syringe. Then the glass was sealed by a septa and degassed with three freeze-pump-thaw cycles. After the mixture was thoroughly degassed, the glass vial placed in a light chamber for irradiation of light and their basic set-up has shown in Figure S1. The reaction mixture was allowed stir while the reaction proceeded. Resultant mixture was extracted with copious quantities of diethyl ether (DEE). The combined organic phase was concentrated under vacuum. The crude mixture was analyzed by GC-MS and NMR.

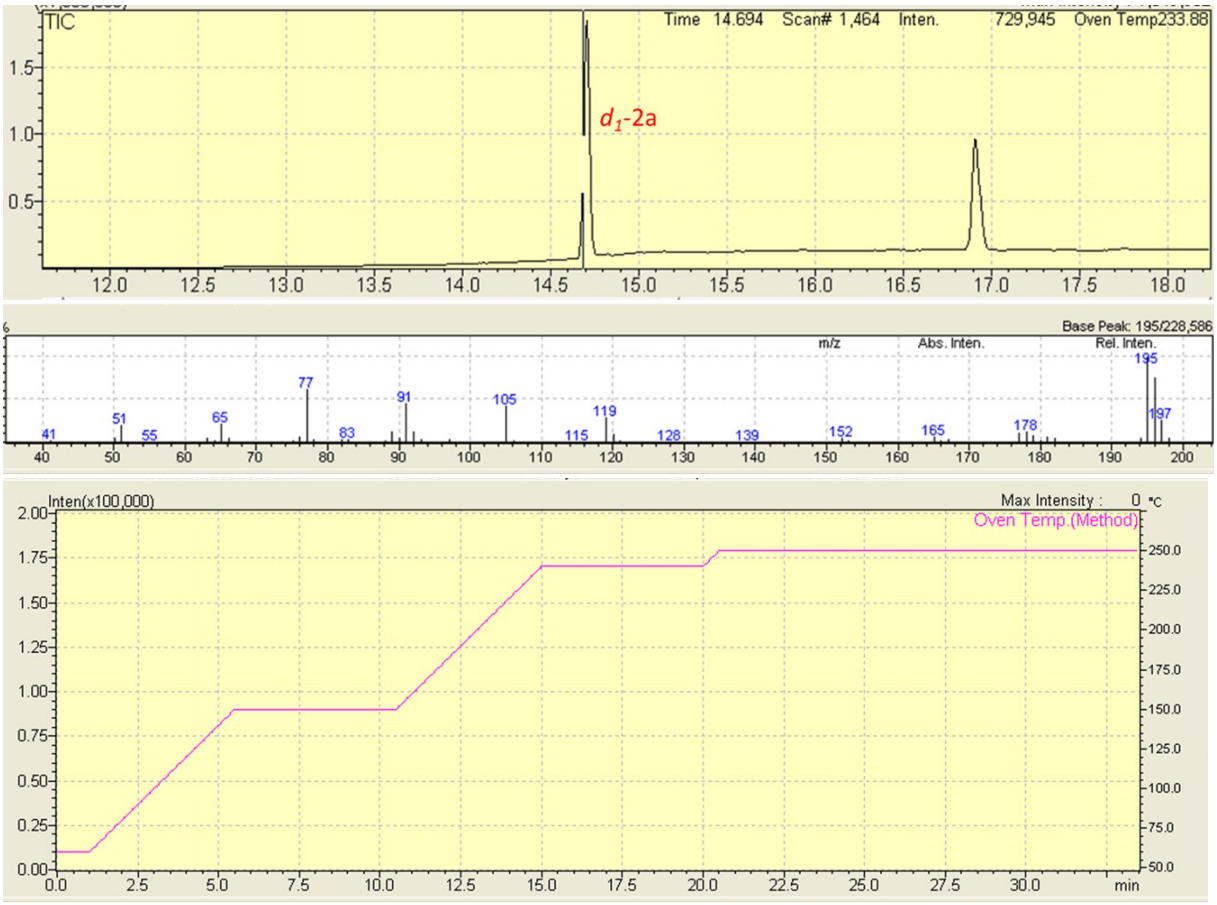


Figure S6a. GC-MS spectrum of ***d*$_1$-2a** with fast oven temperature variation.

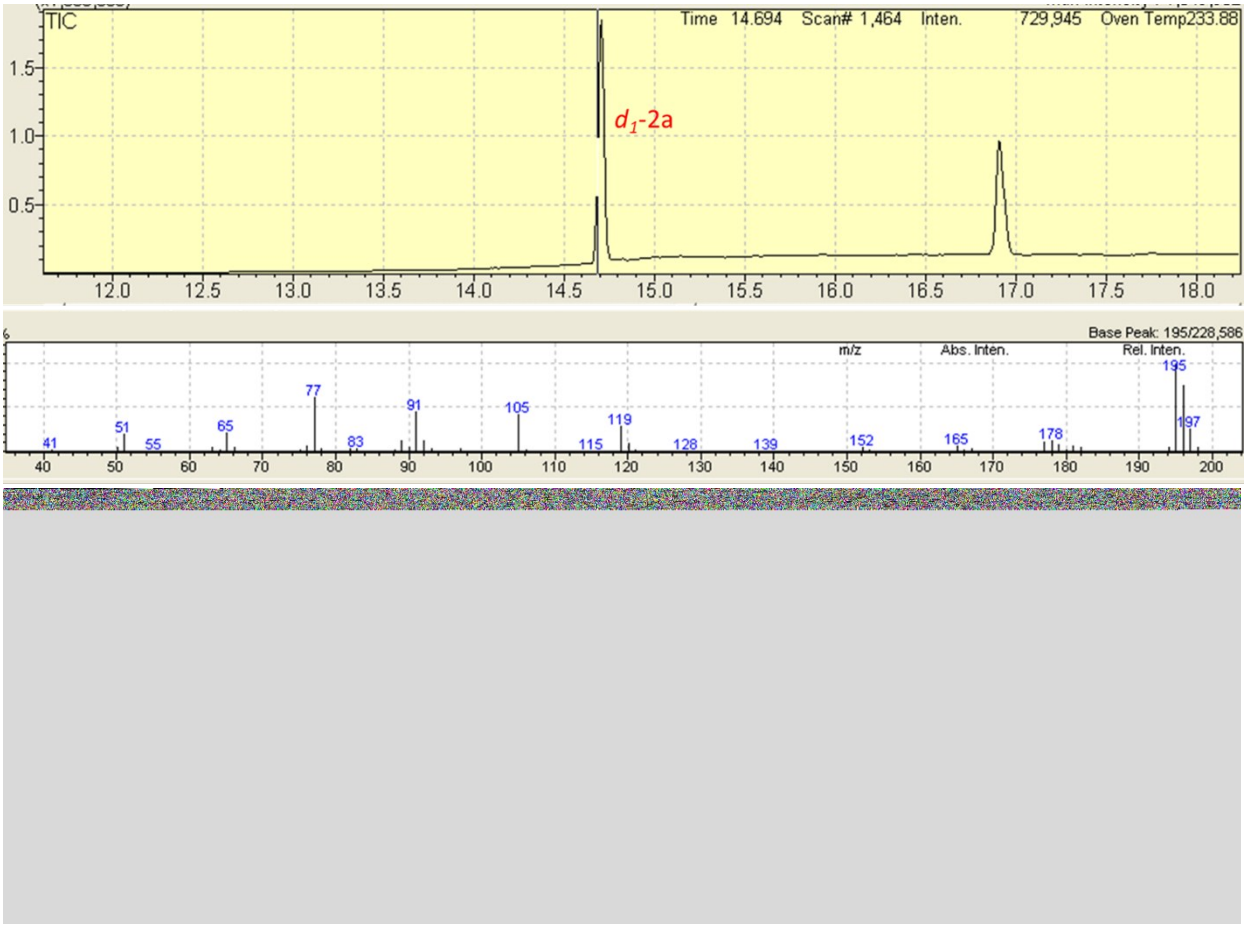


Figure S6b. GC-MS spectrum of ***d_2*-2a** with fast oven temperature variation

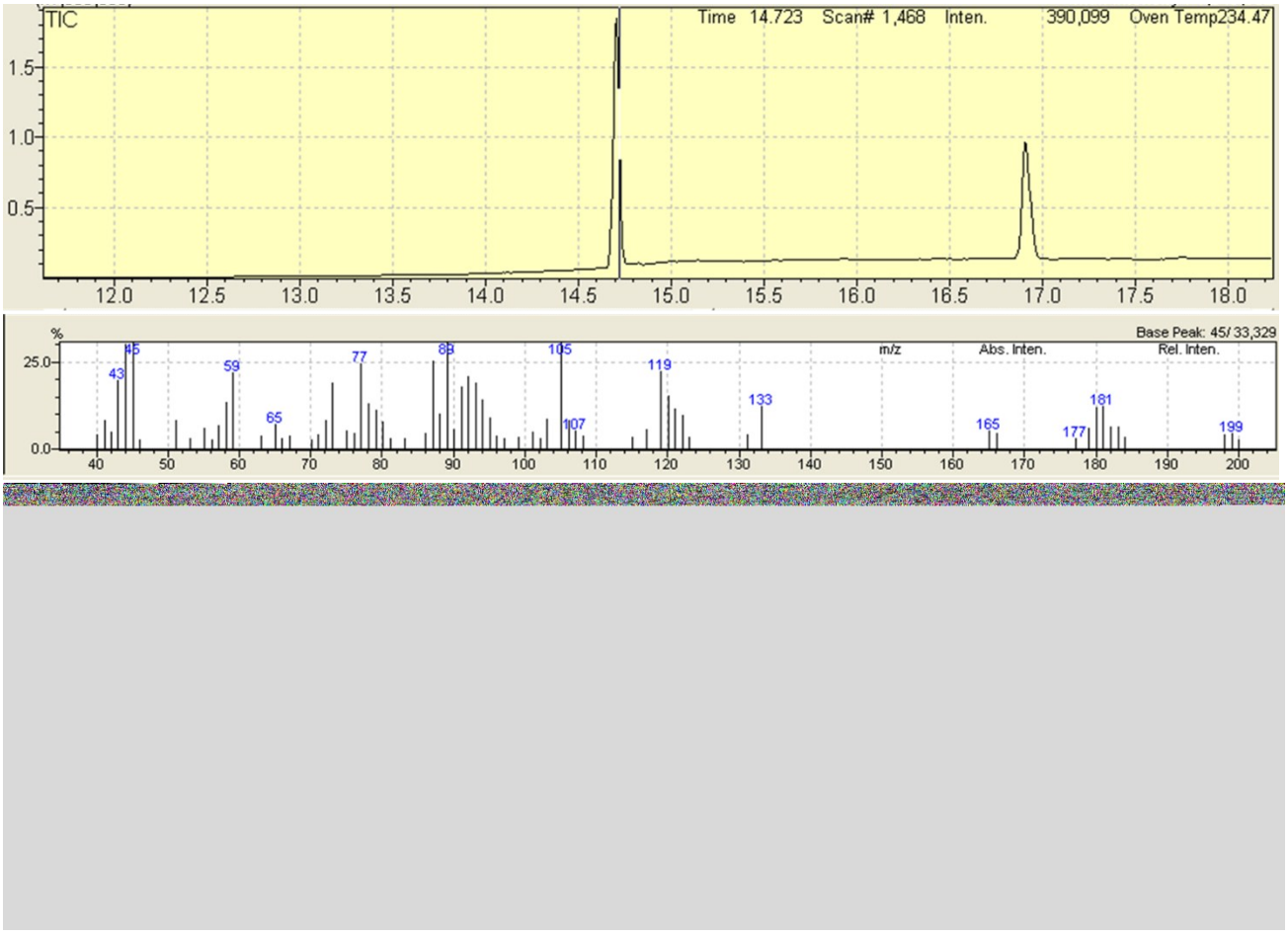


Figure S6c. GC-MS spectrum of ***d*$_1$-2a** with fast oven temperature variation.

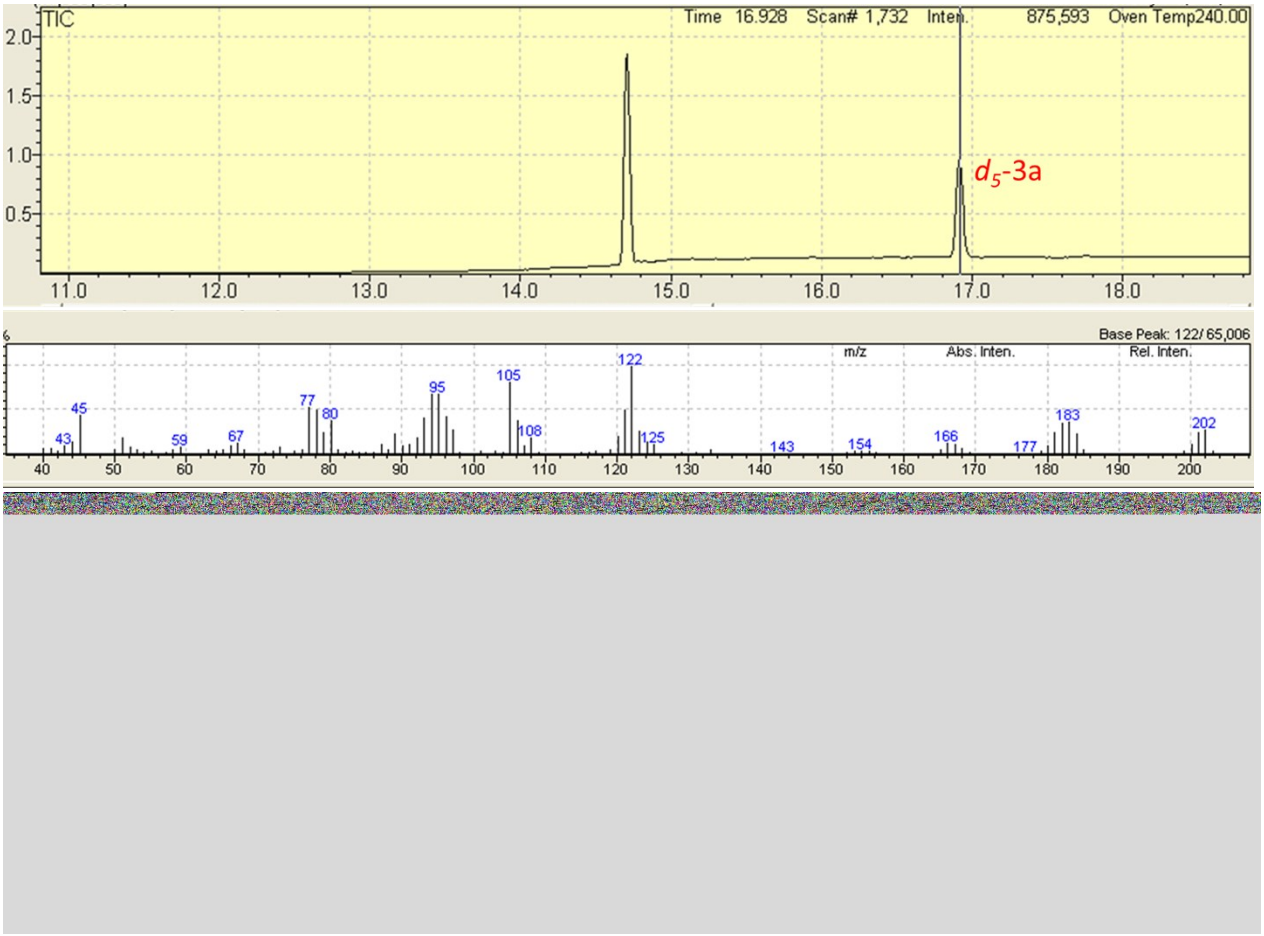


Figure S7a. GC-MS spectrum of **d_5-3a** with fast oven temperature variation

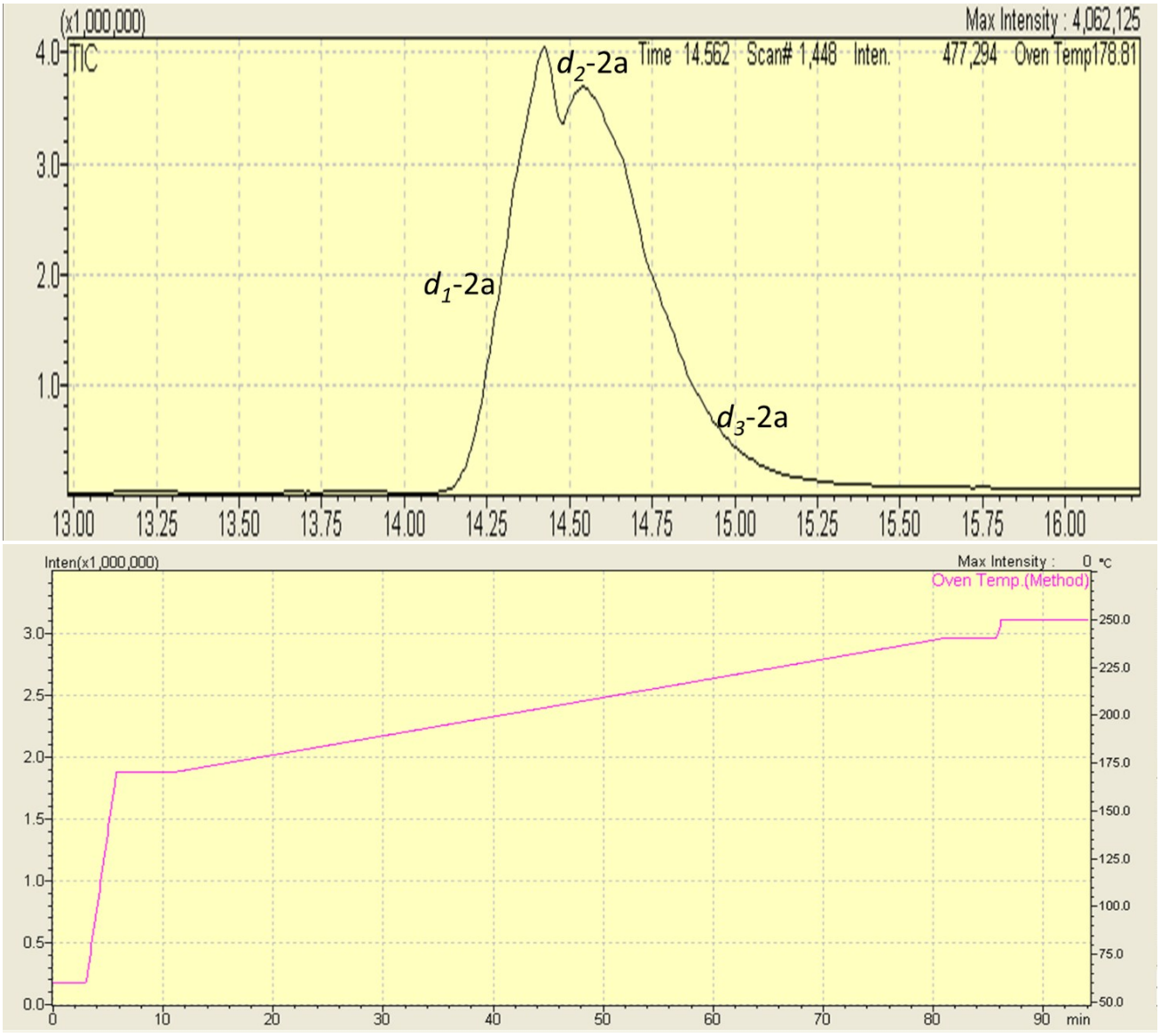


Figure S7b. GC-MS spectrum of ***d*$_5$-3a** with very slow oven temperature variation.

7.Spectra

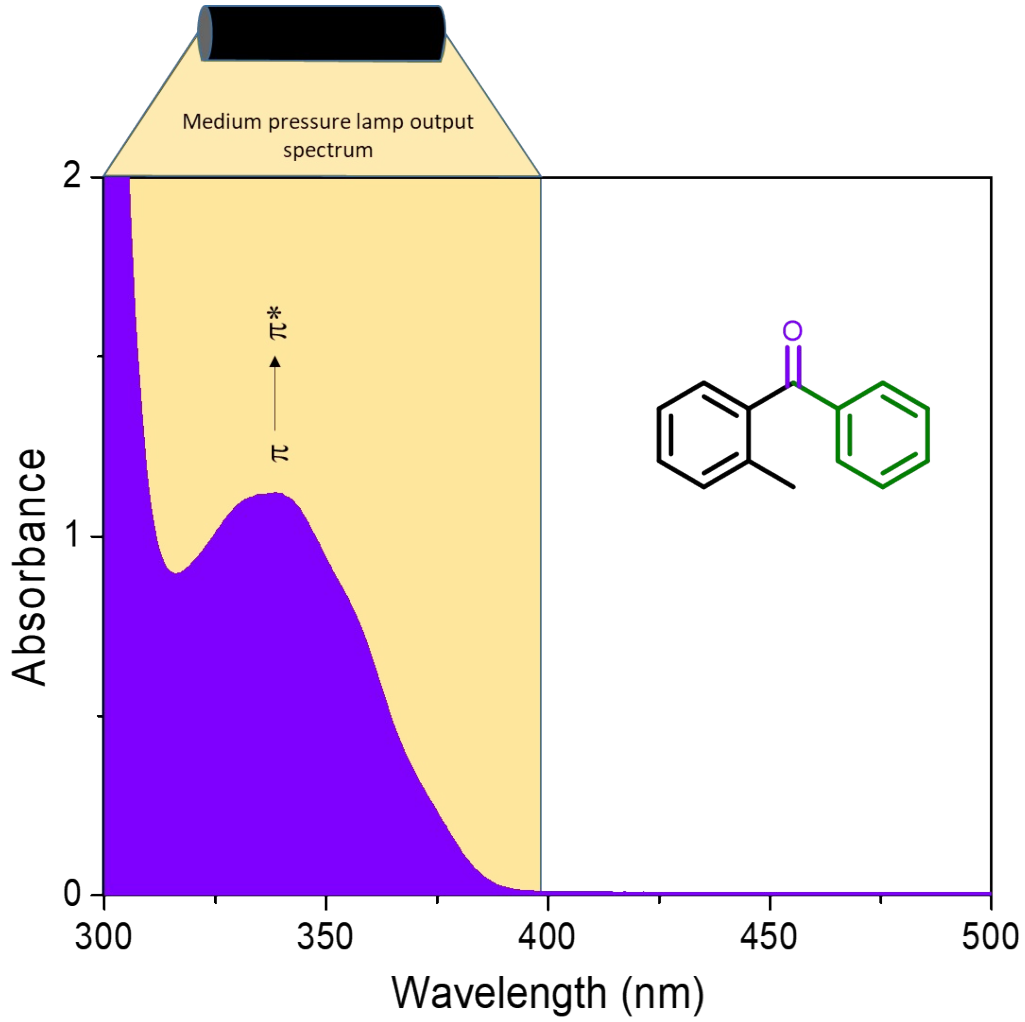


Figure S8. UV-Vis spectra of (phenyl(o-tolyl)methanone (**2a**) in 0.005M MeCN.

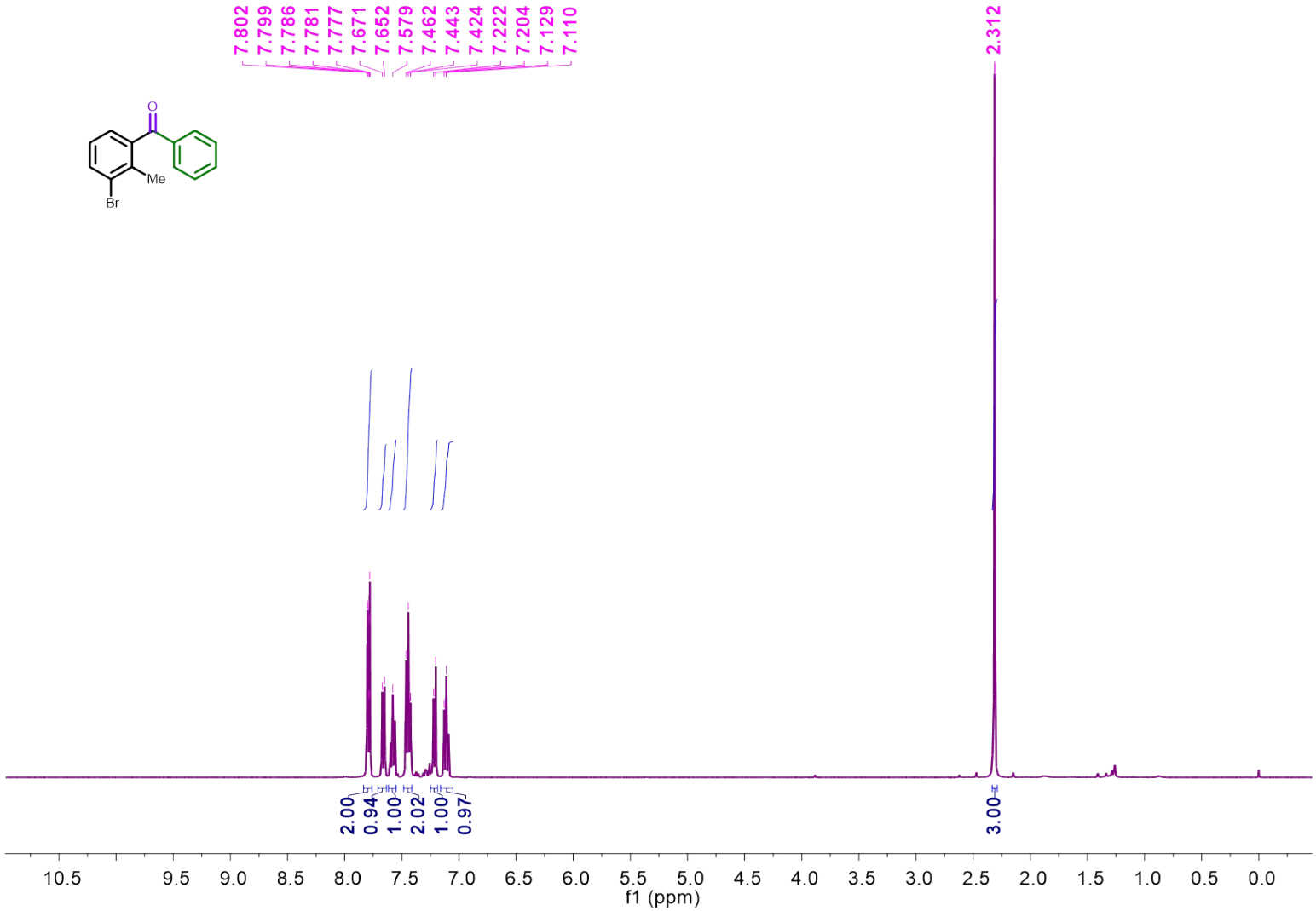


Figure S9. ^{1}H NMR spectra of (3-bromo-2-methylphenyl) (phenyl) methanone (**2b**) in $CDCl_3$.

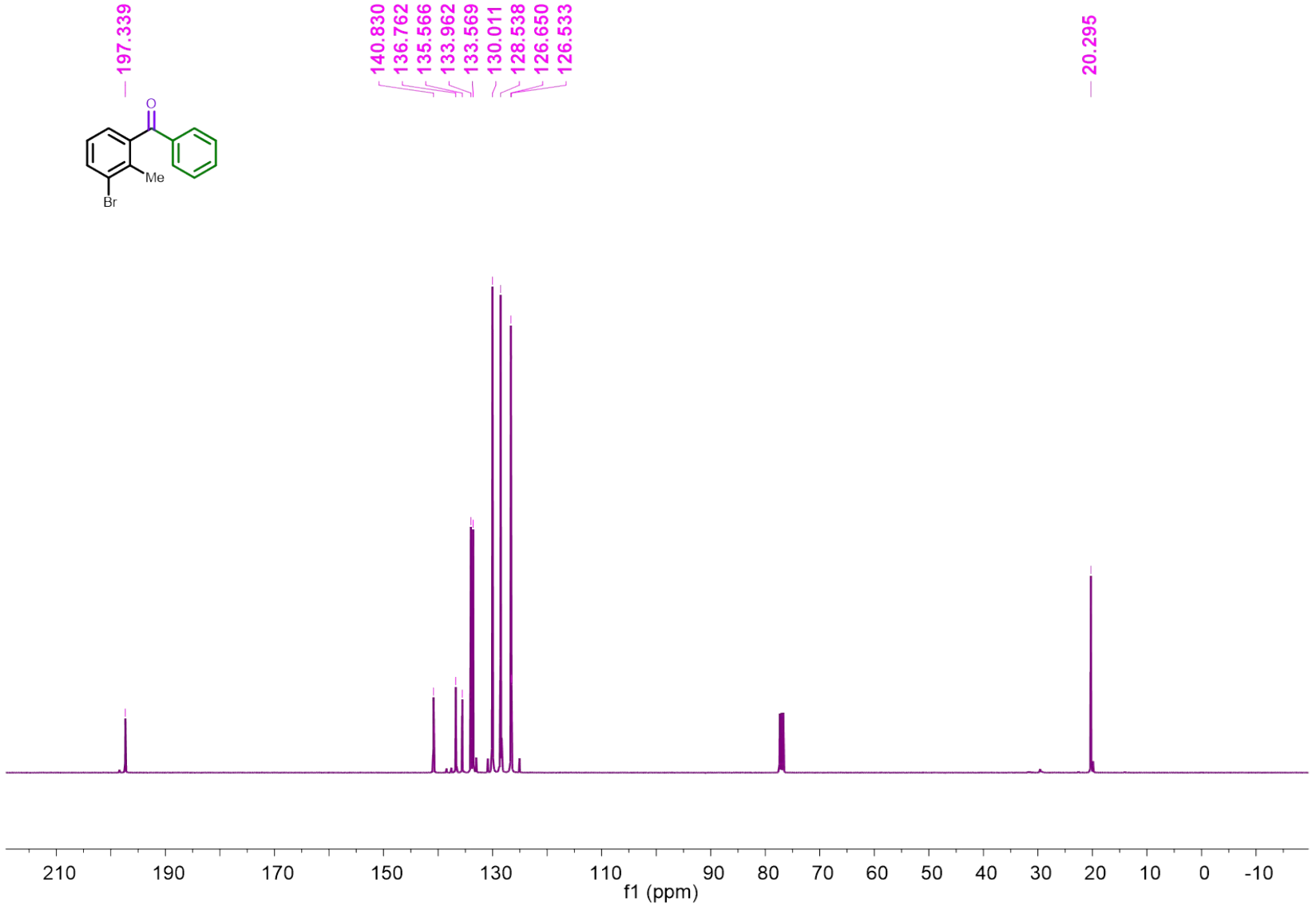


Figure S10. ^{13}C NMR spectra of (3-bromo-2-methylphenyl) (phenyl) methanone (**2b**) in $CDCl_3$.

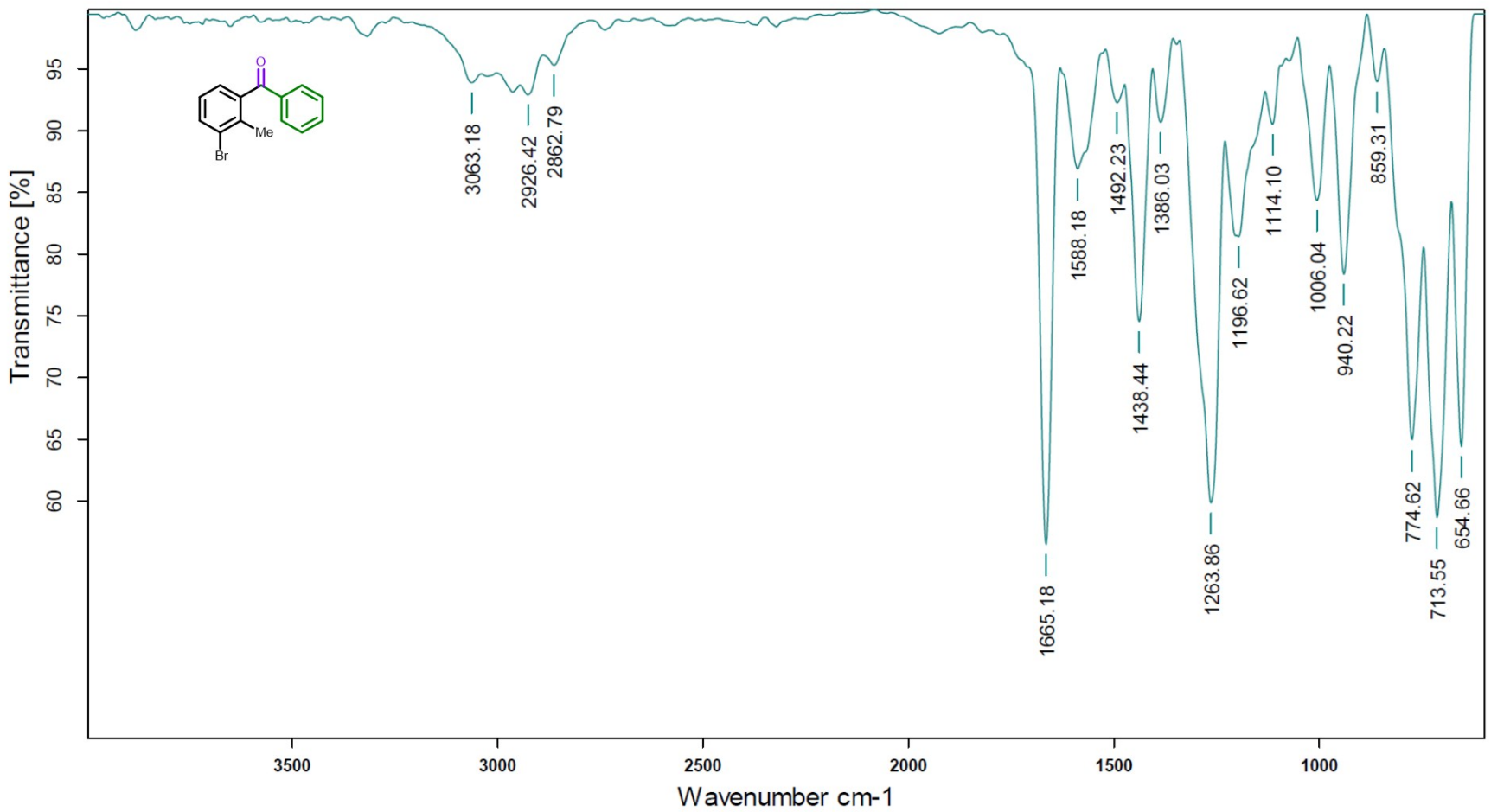


Figure S11. IR spectra of (3-bromo-2-methylphenyl) (phenyl) methanone (**2b**).

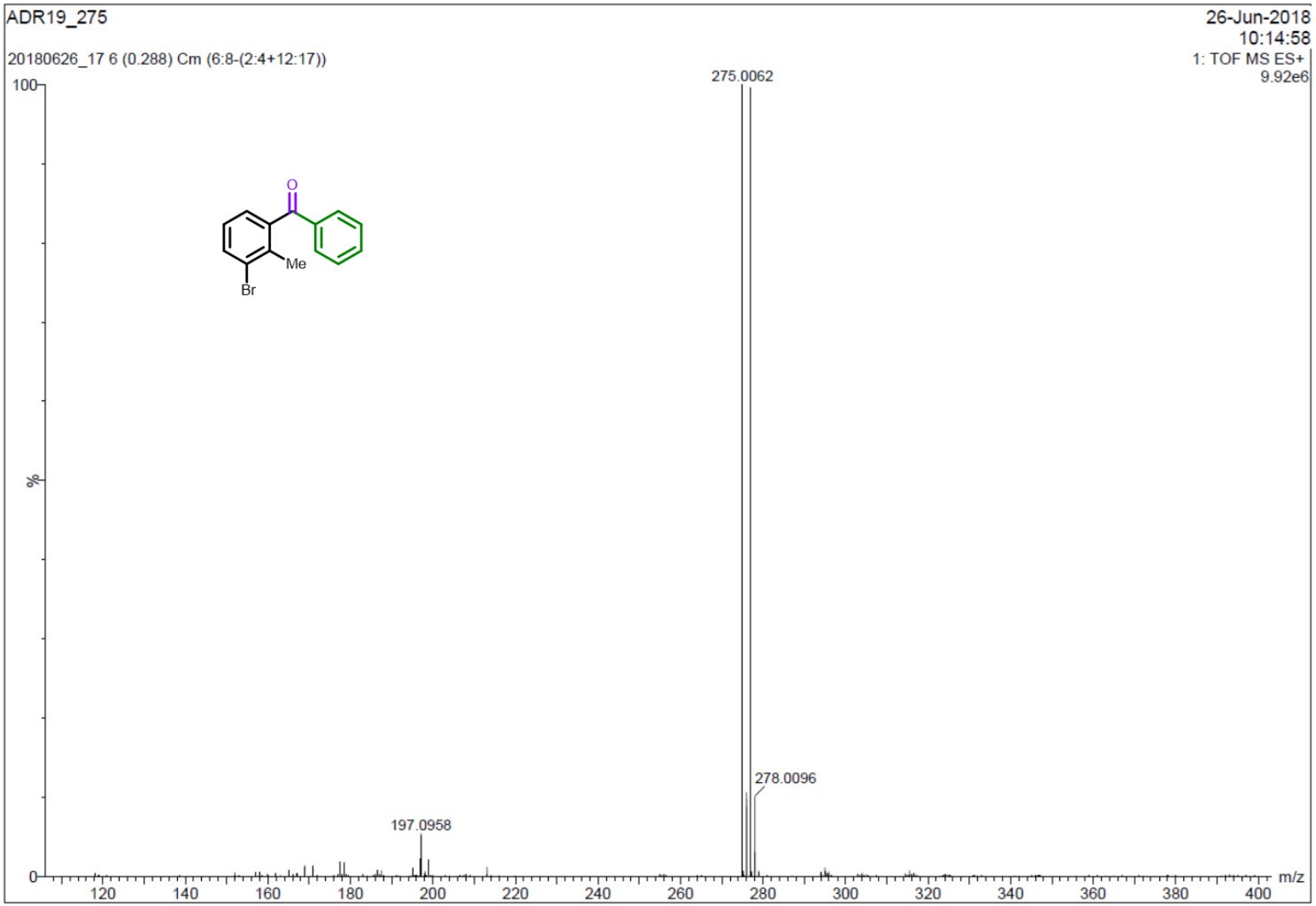


Figure S12. HRMS spectra of (3-bromo-2-methylphenyl) (phenyl) methanone (**2b**).

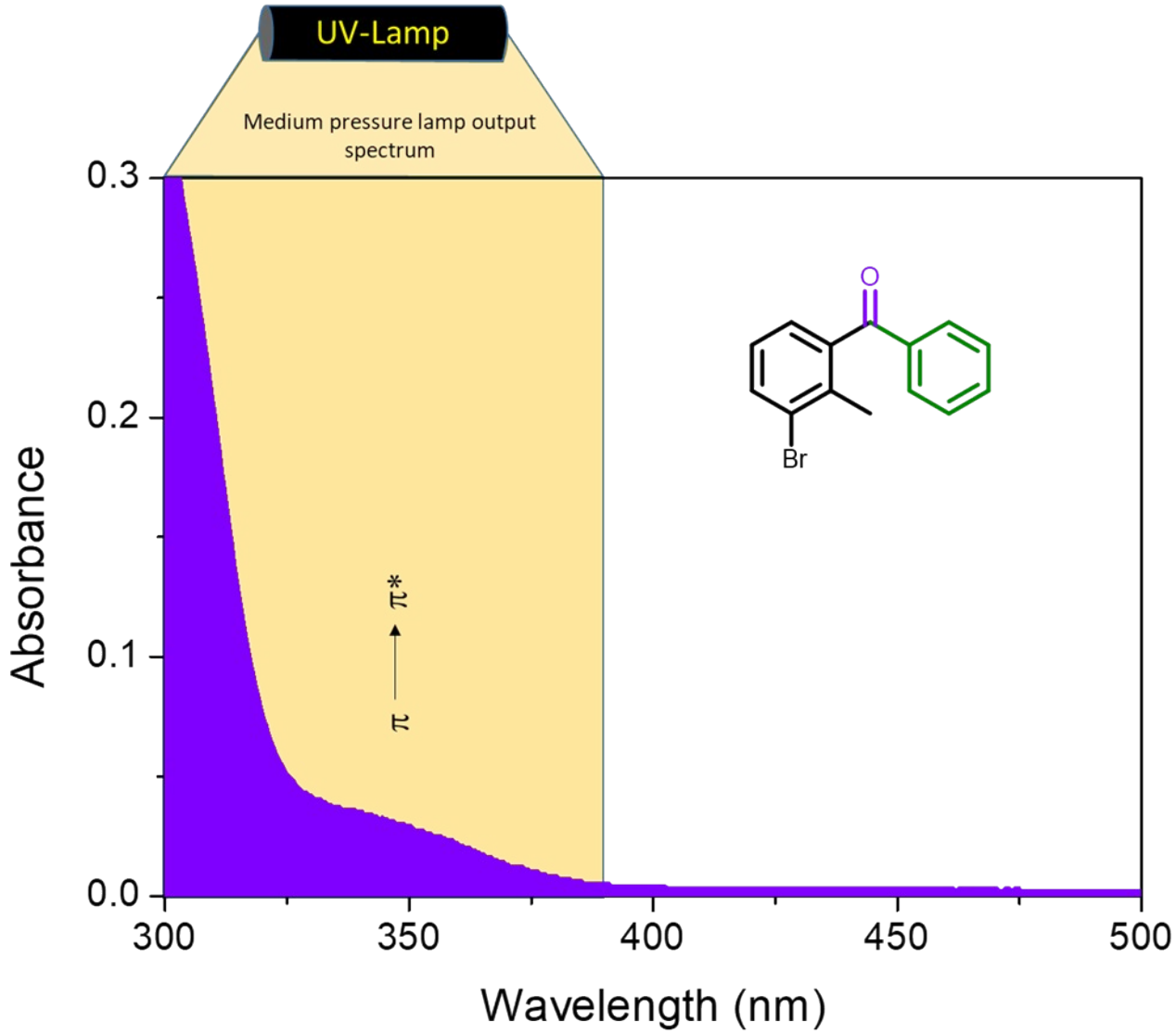


Figure S13. UV-Vis spectra of (3-bromo-2-methylphenyl) (phenyl) methanone (**2b**) in 0.005M MeCN.

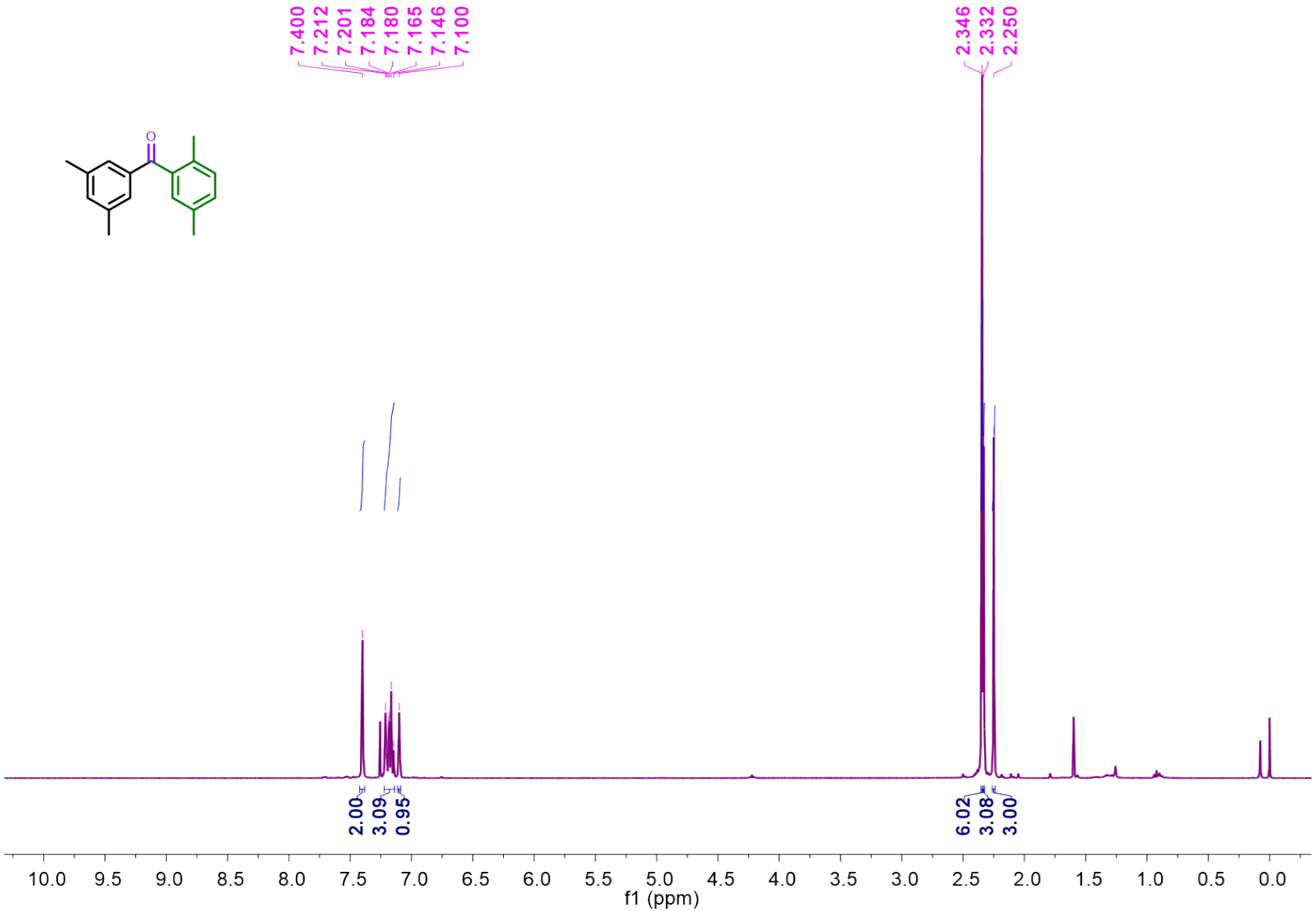


Figure S14. ^{1}H NMR spectra of (2,5-dimethylphenyl) (3,5-dimethylphenyl) methanone **(2c)**.

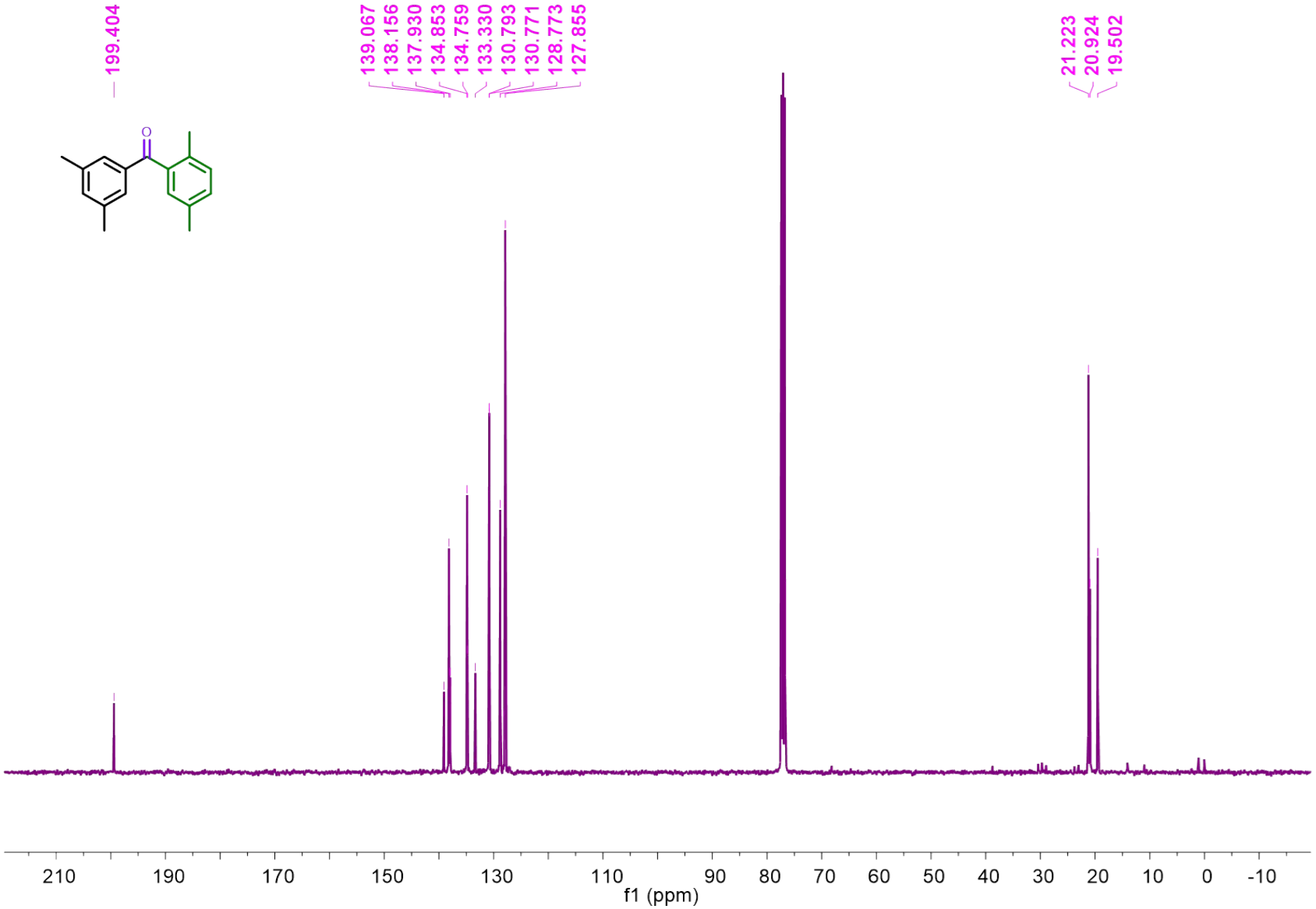


Figure S15. ^{13}C NMR spectra of (2,5-dimethylphenyl) (3,5-dimethylphenyl) methanone **(2c)** in $CDCl_3$.

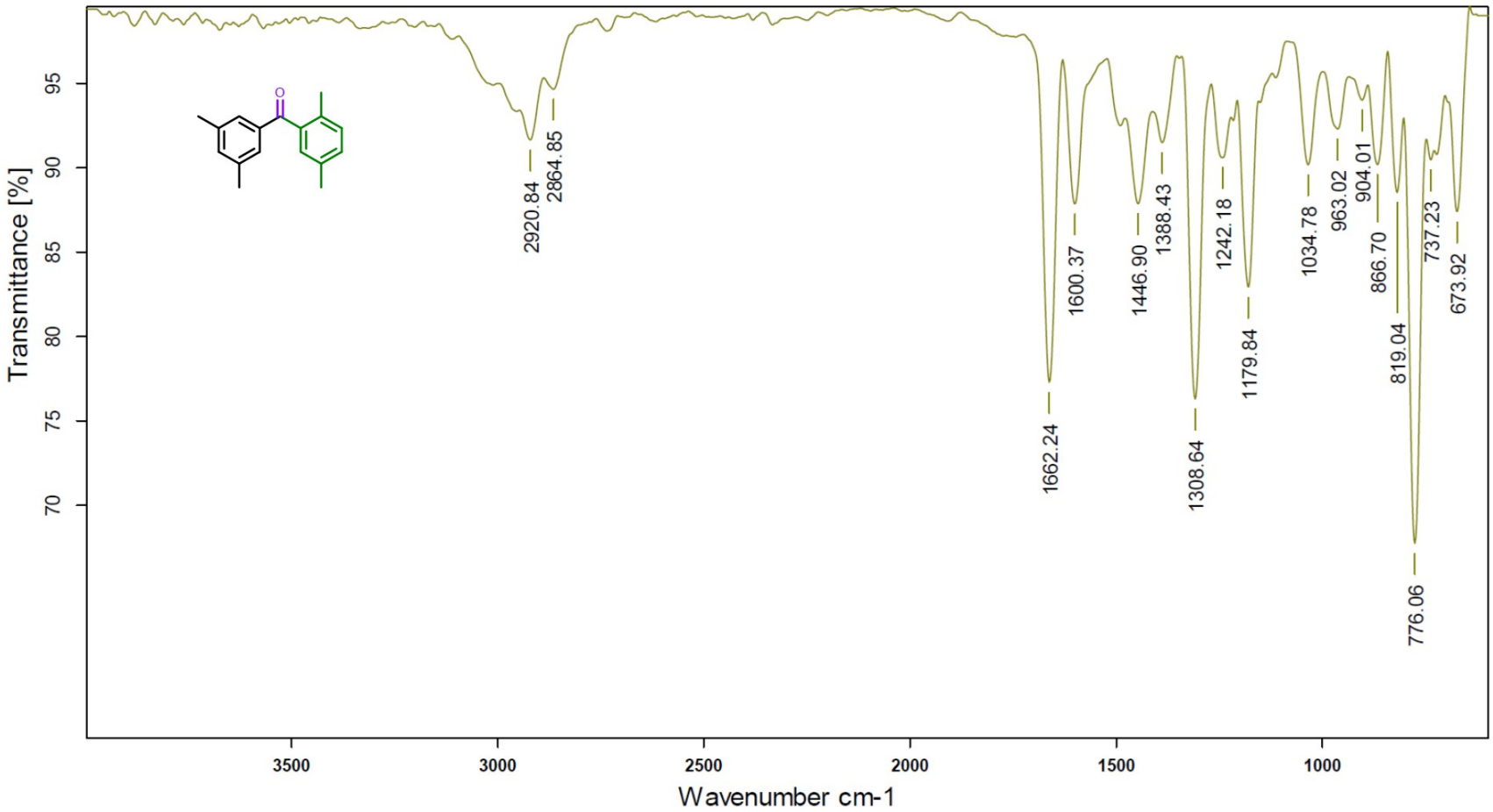


Figure S16. IR spectra of (2,5-dimethylphenyl) (3,5-dimethylphenyl) methanone **(2c**).

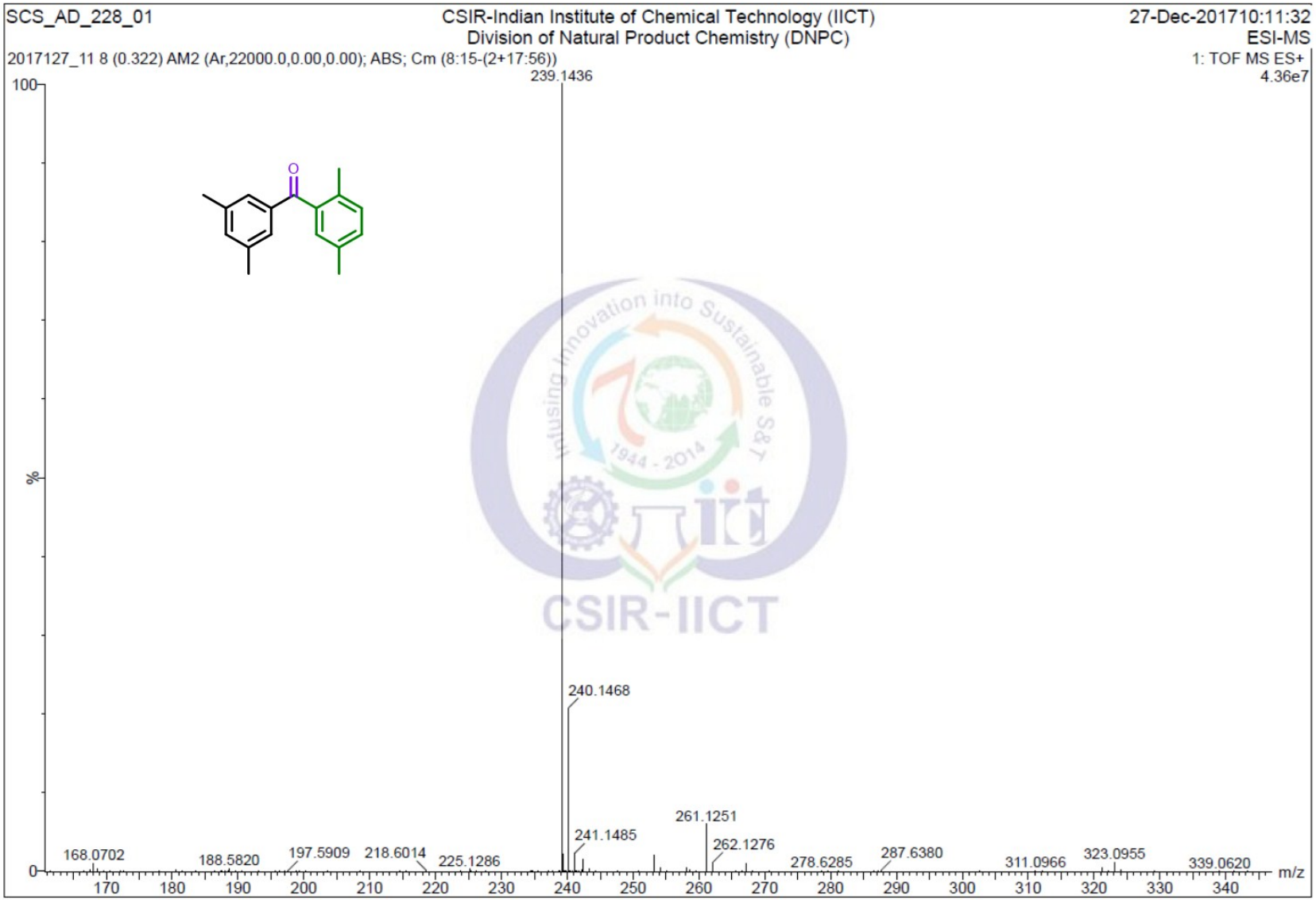


Figure S17. HRMS spectra of (2,5-dimethylphenyl) (3,5-dimethylphenyl) methanone **(2c)**.

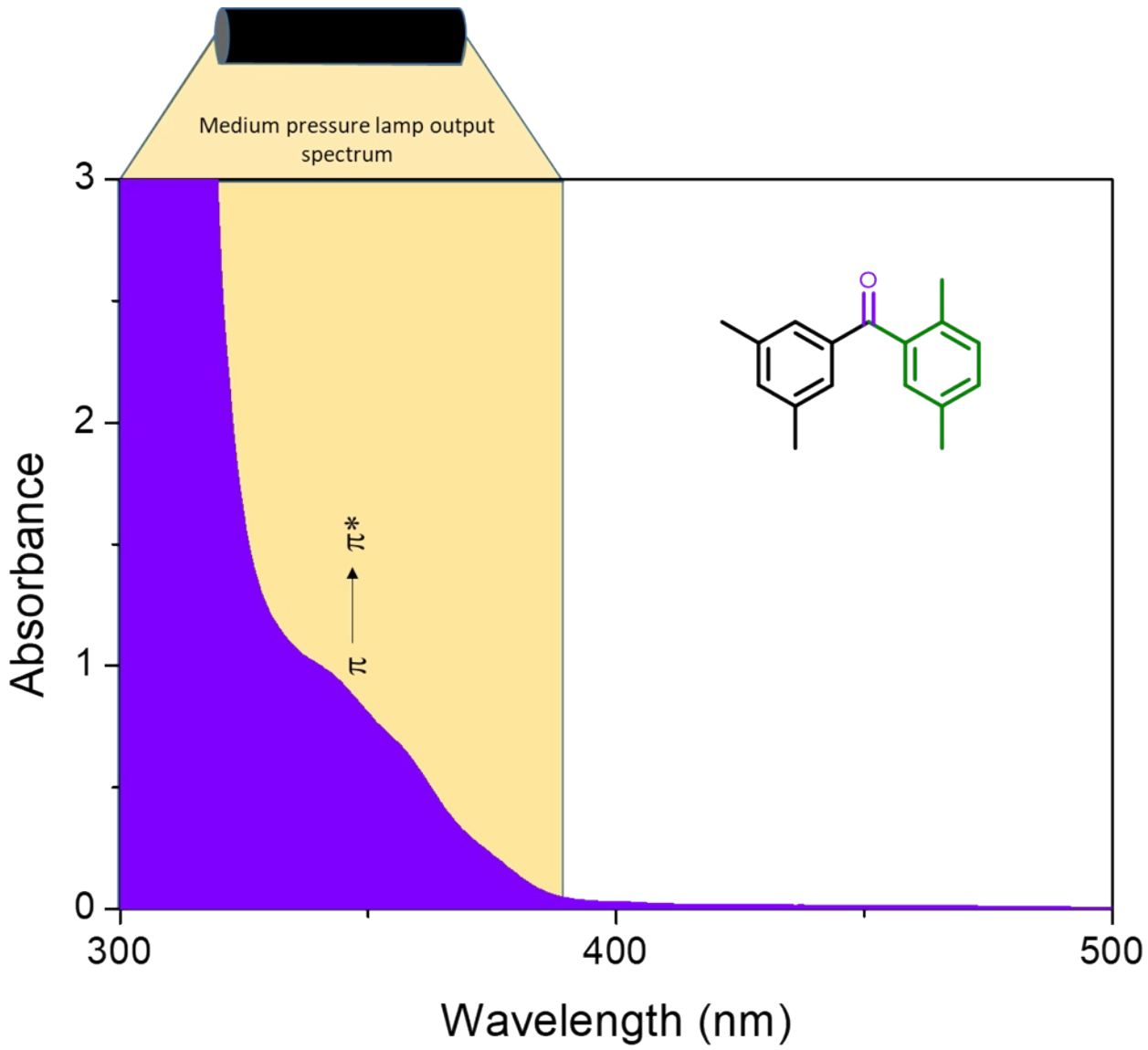


Figure S18. UV-Vis spectra of (2,5-dimethylphenyl) (3,5-dimethylphenyl) methanone **(2c**) in 0.005M MeCN.

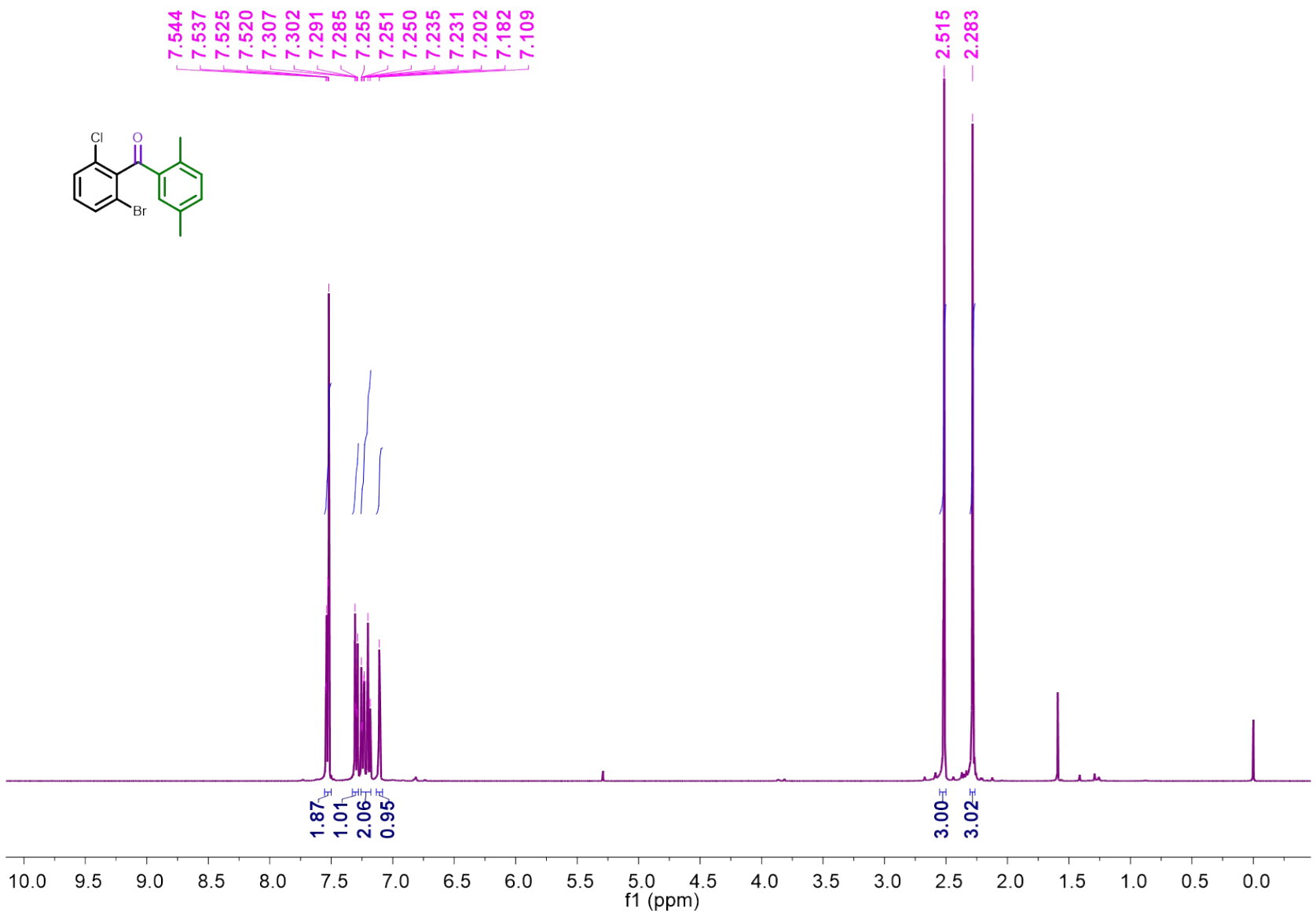


Figure S19. ^{1}H NMR spectra of (2-bromo-6-chlorophenyl) (2,5-dimethylphenyl) methanone **(2d)** in $CDCl_3$.

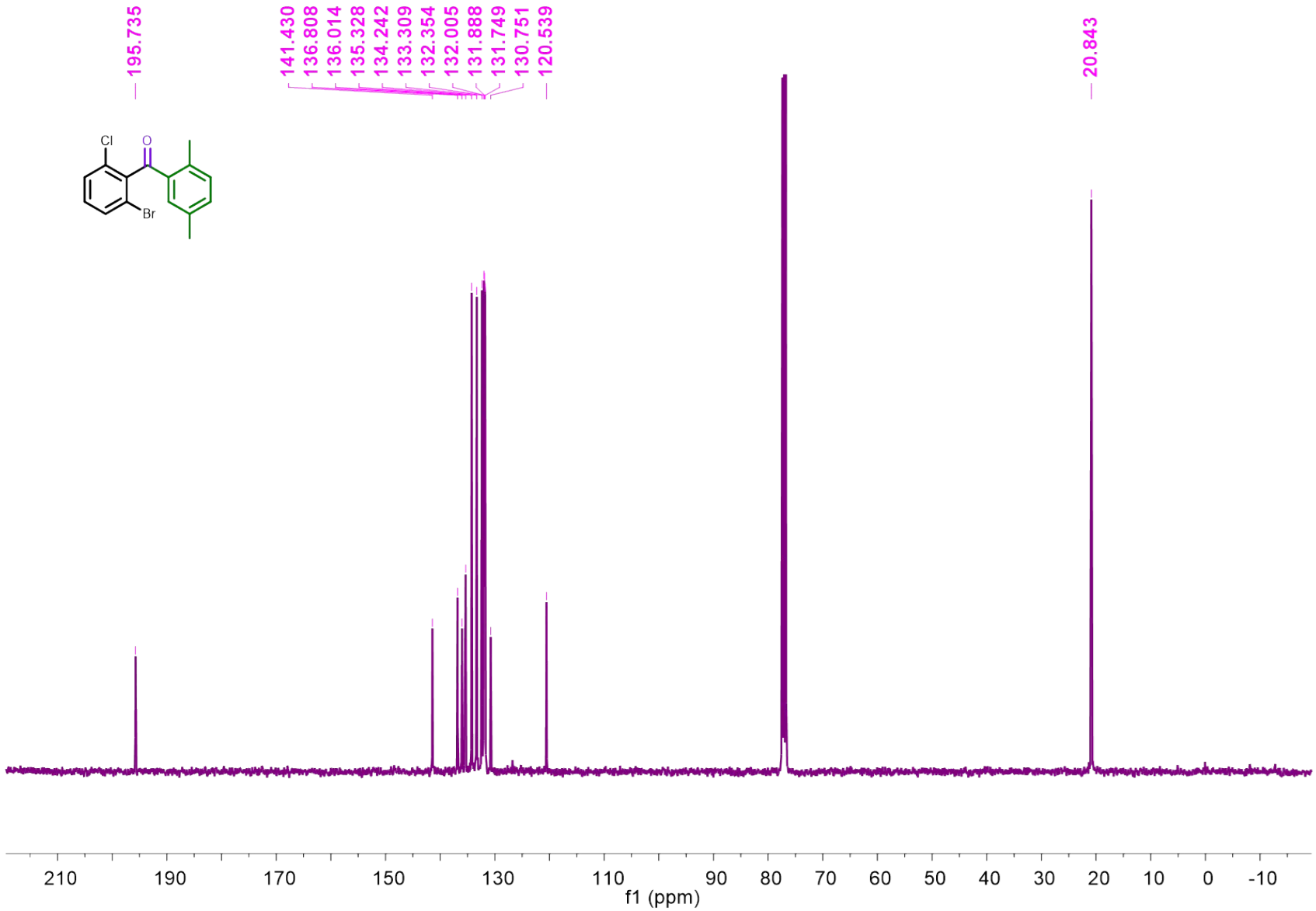


Figure S20. ^{13}C NMR spectra of (2-bromo-6-chlorophenyl) (2,5-dimethylphenyl) methanone **(2d)** in $CDCl_3$.

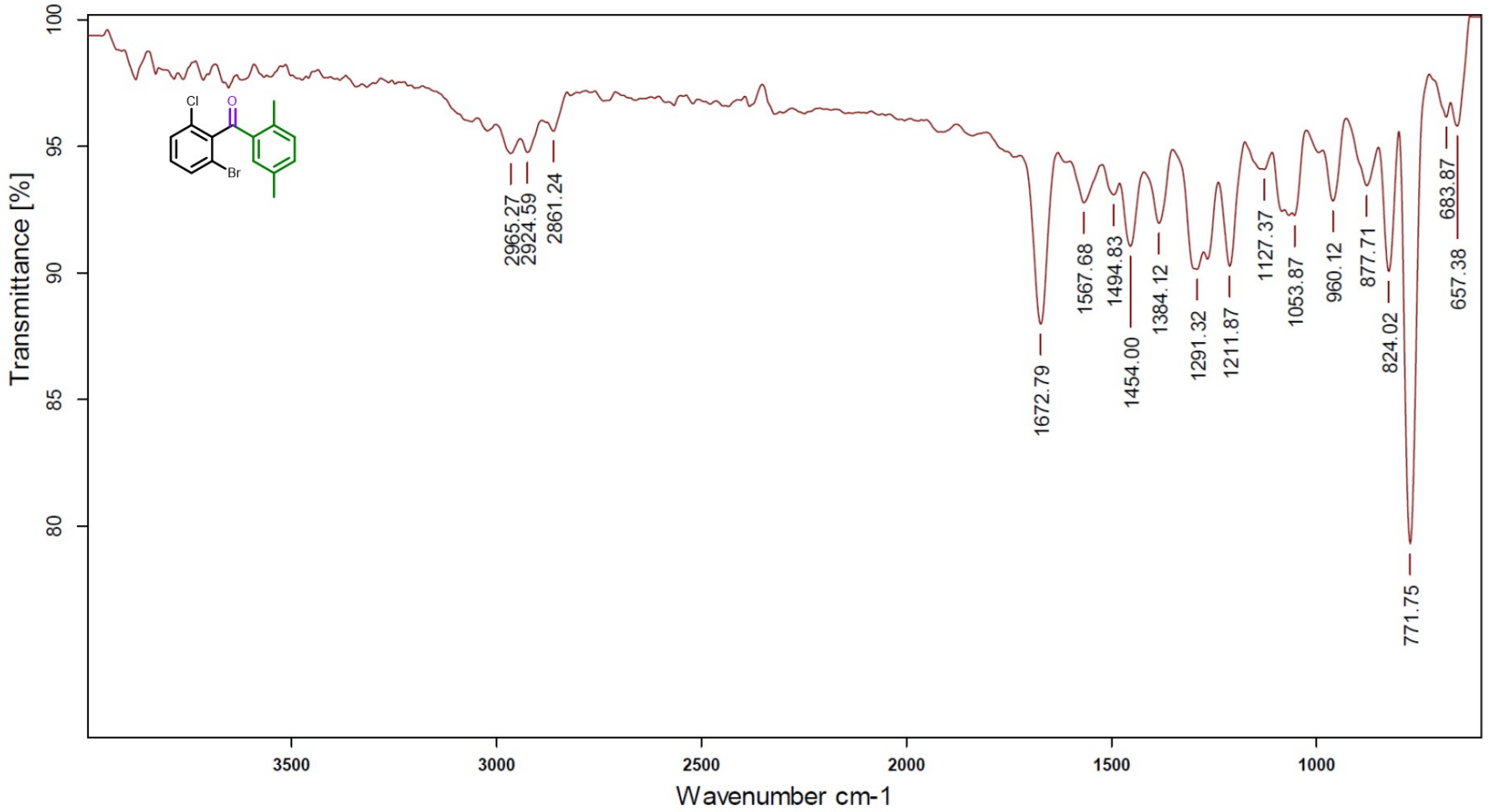


Figure S21. IR spectra of (2-bromo-6-chlorophenyl) (2,5-dimethylphenyl) methanone **(2d)**.

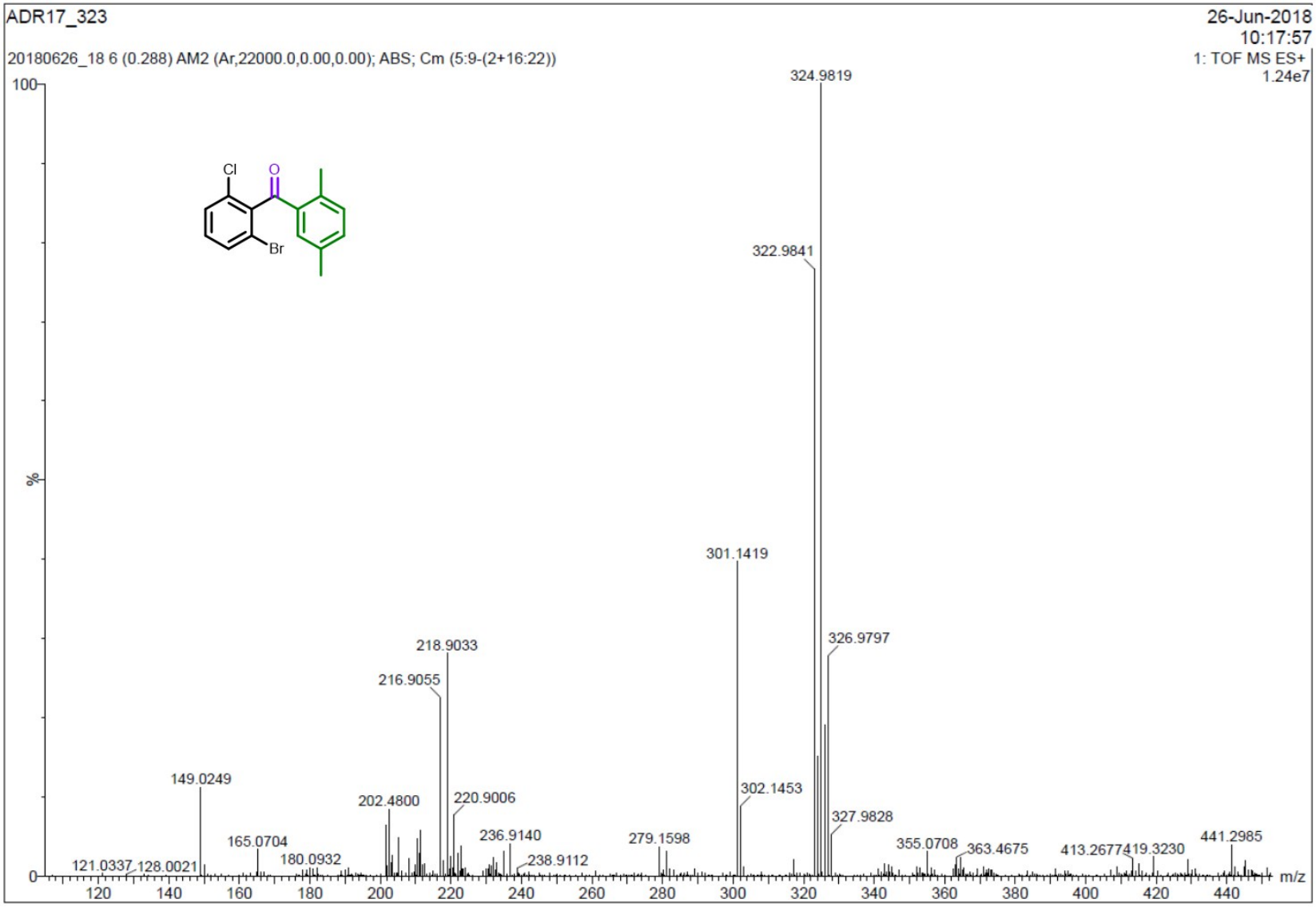


Figure S22. HRMS spectra of (2-bromo-6-chlorophenyl) (2,5-dimethylphenyl) methanone **(2d**).

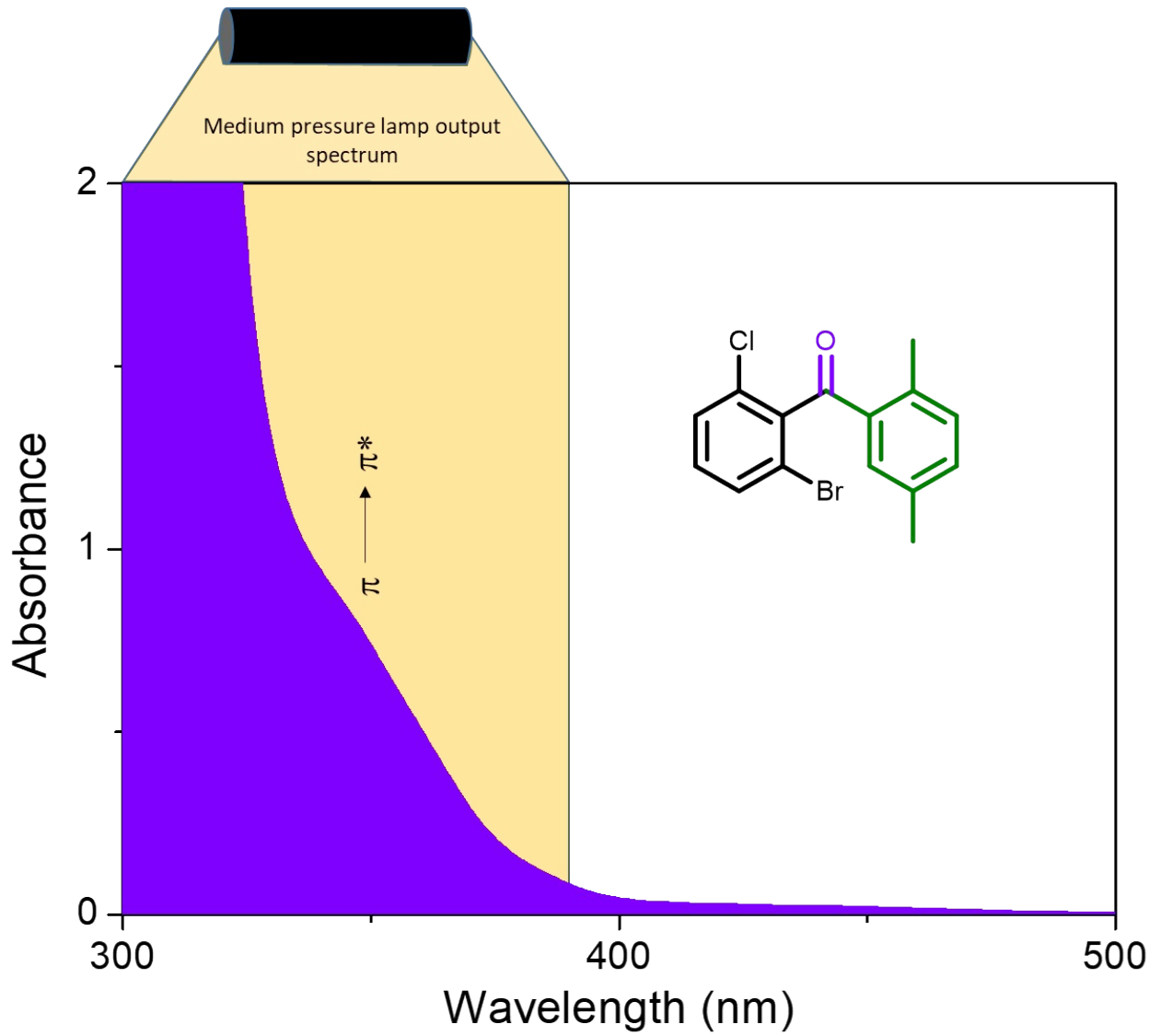


Figure S23. UV-Vis spectra of (2-bromo-6-chlorophenyl) (2,5-dimethylphenyl) methanone **(2d**) in 0.005M MeCN.

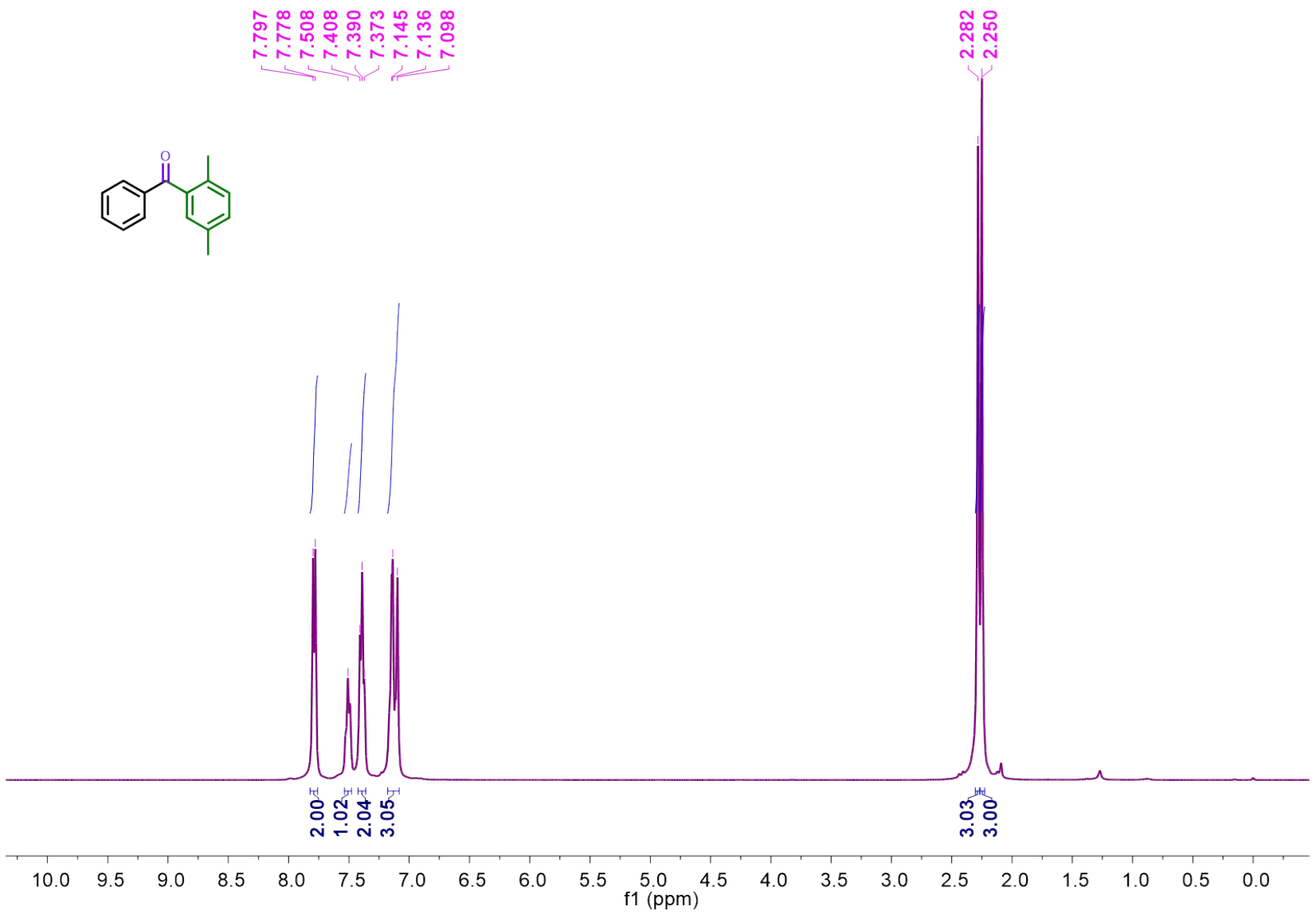


Figure S24. ^{1}H NMR spectra of (2,5-dimethylphenyl)(phenyl) methanone **(2e)** in $CDCl_3$.

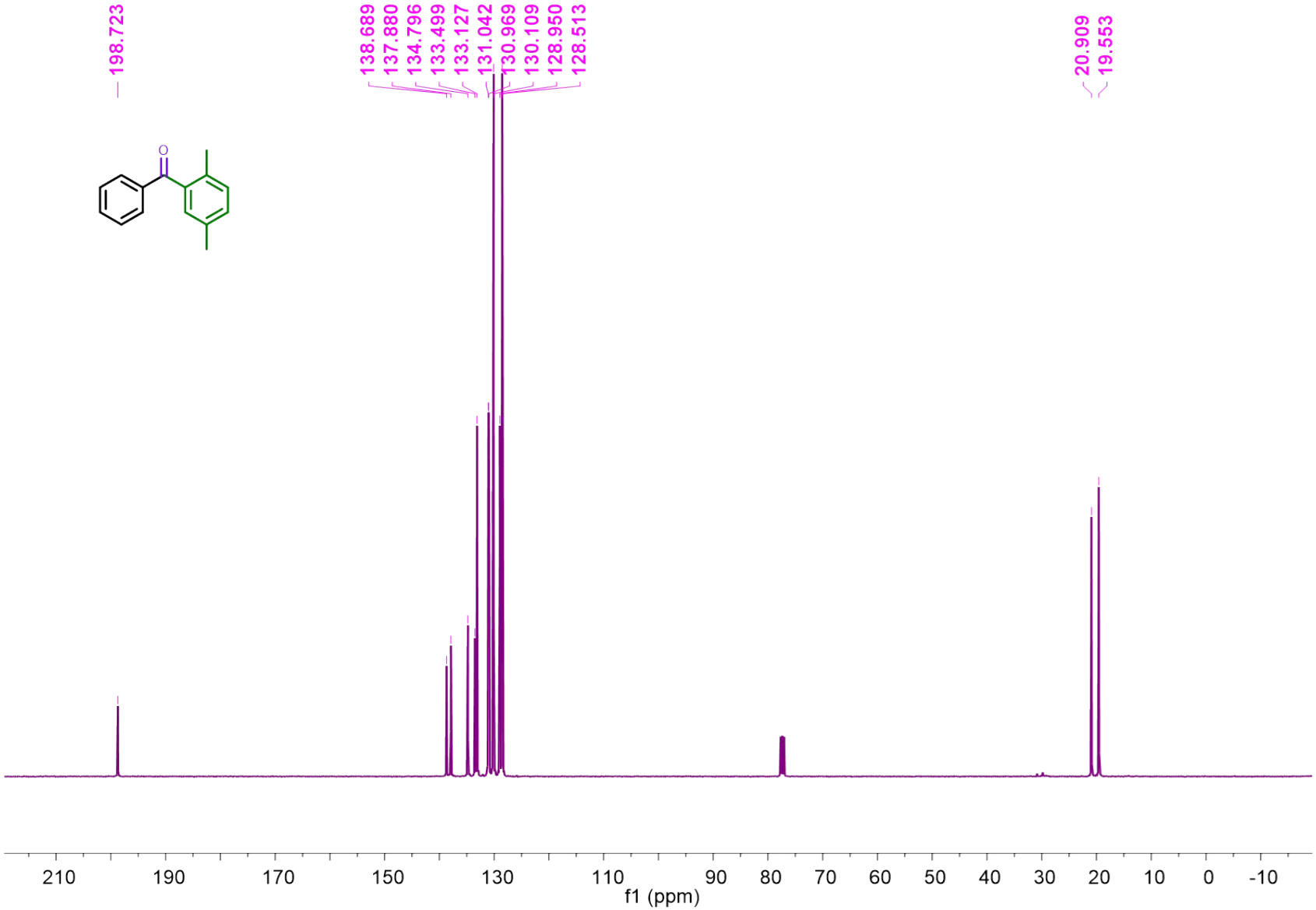


Figure S25. ^{13}C NMR spectra of (2,5-dimethylphenyl) (phenyl) methanone **(2e)** in $CDCl_3$.

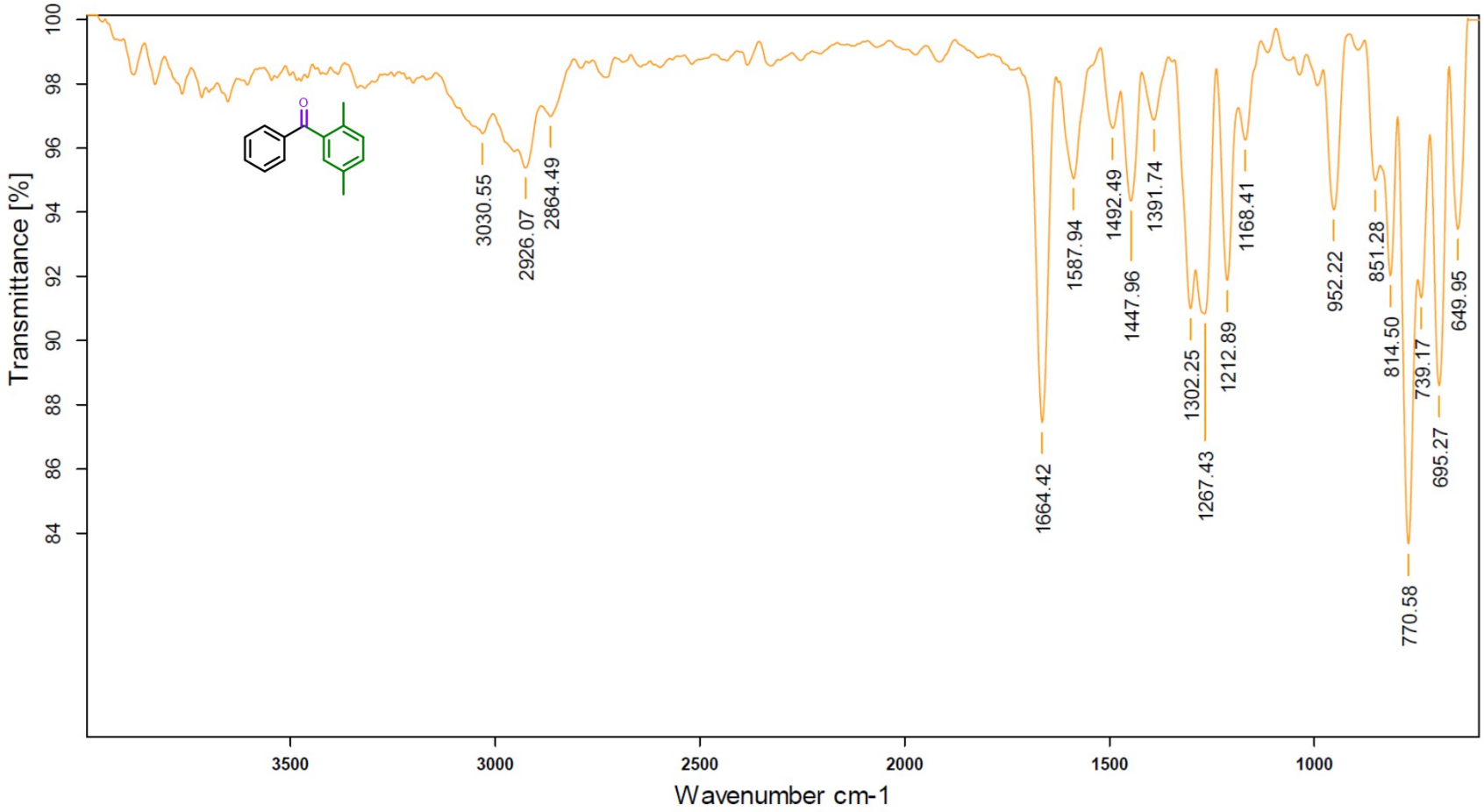


Figure S26. IR spectra of (2,5-dimethylphenyl) (phenyl) methanone **(2e**).

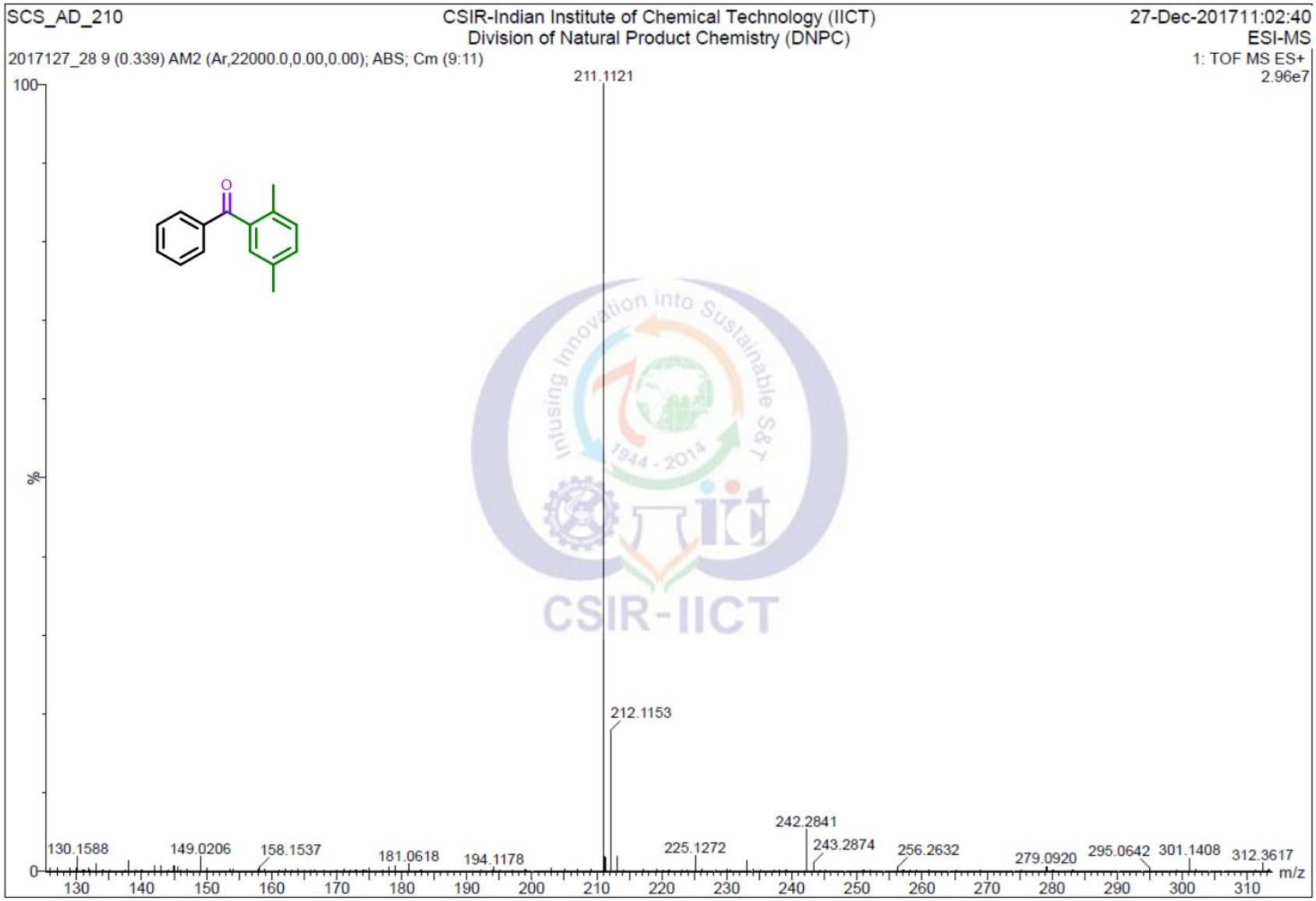


Figure S27. HRMS spectra of (2,5-dimethylphenyl) (phenyl) methanone **(2e)**.

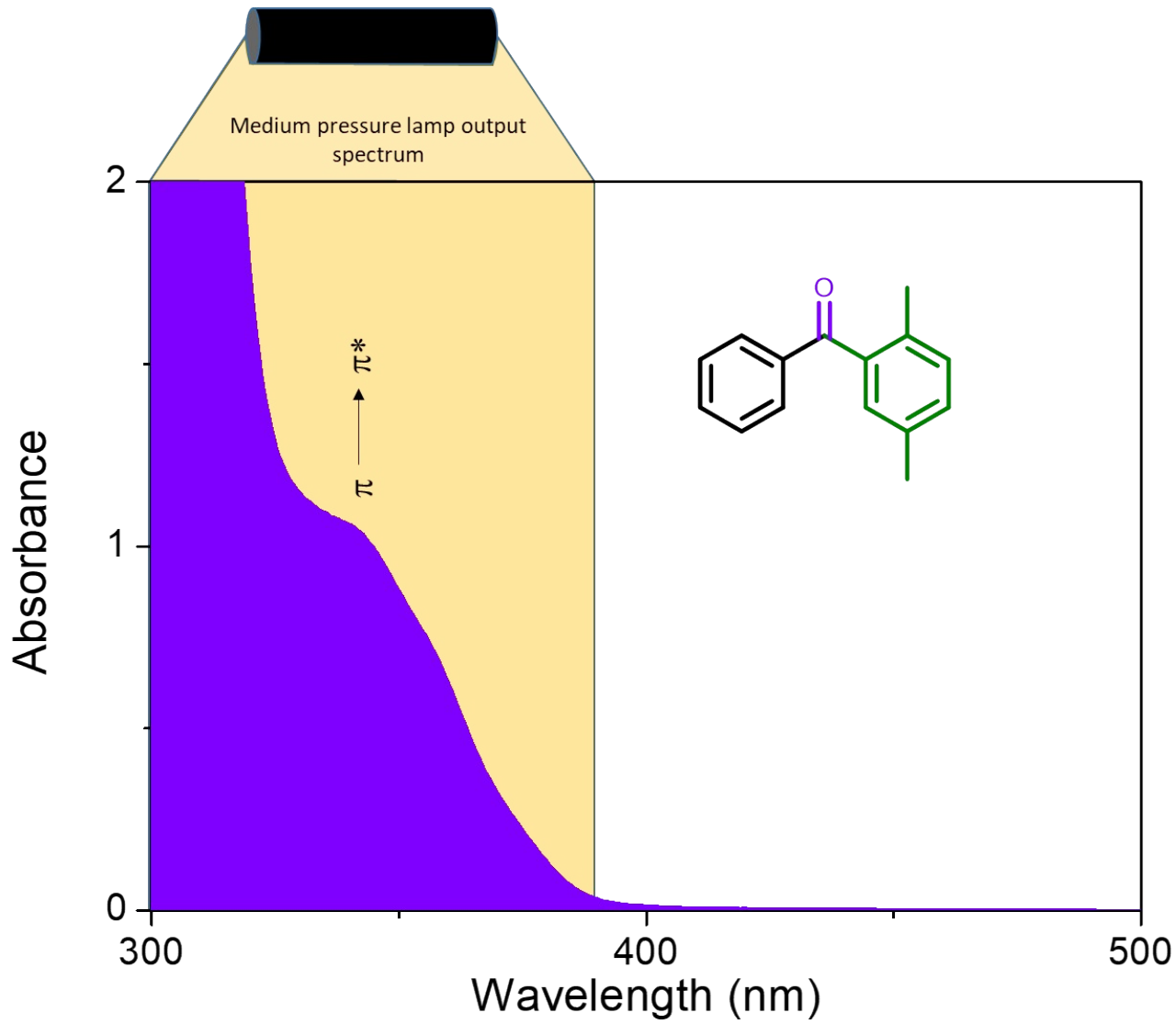


Figure S28. UV-Vis spectra of (2,5-dimethylphenyl) (phenyl) methanone **(2e**) in 0.005M MeCN.

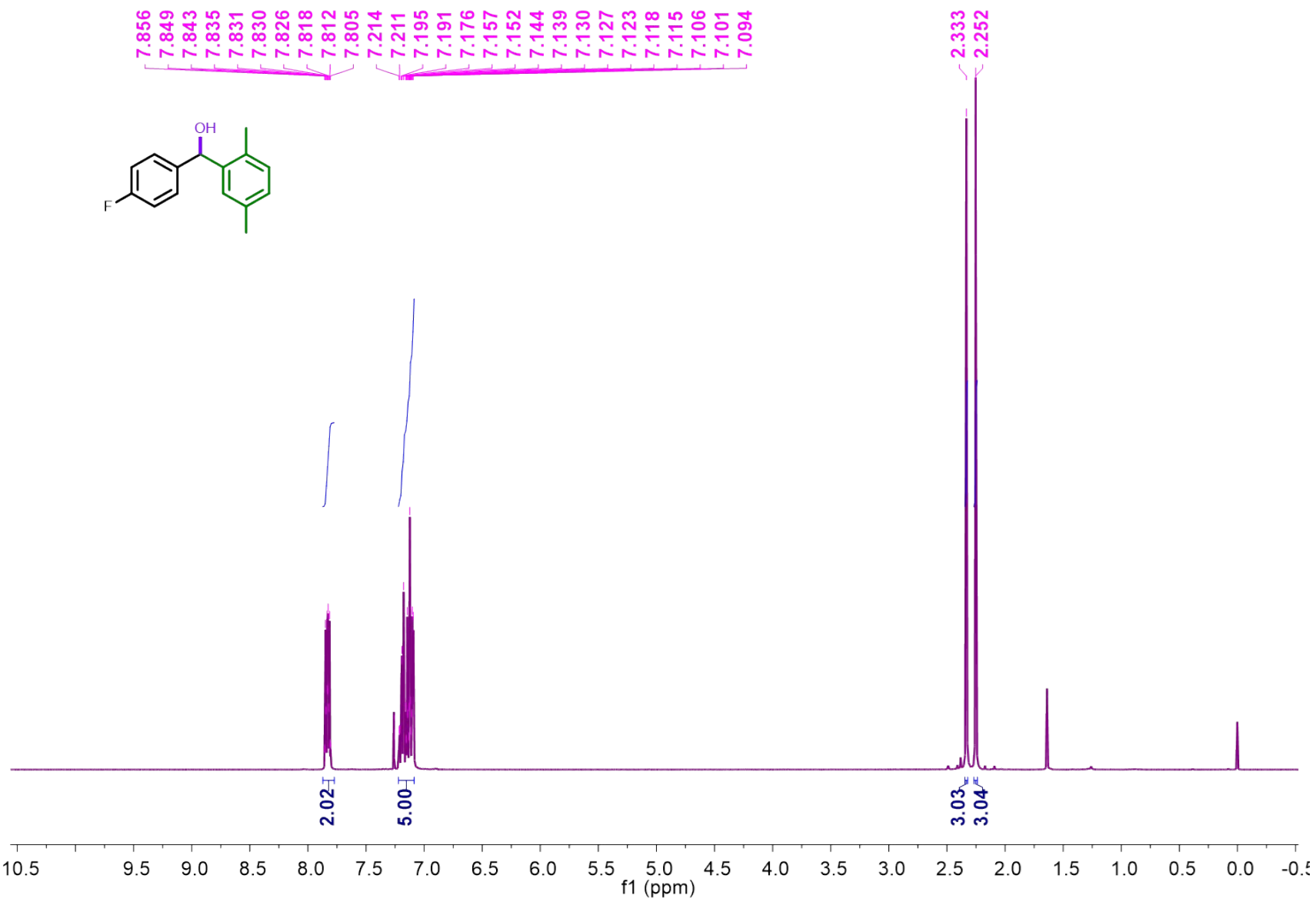


Figure S29. ^{1}H NMR spectra of (2,5-dimethylphenyl) (4-fluorophenyl) methanone **(2f)** in $CDCl_3$.

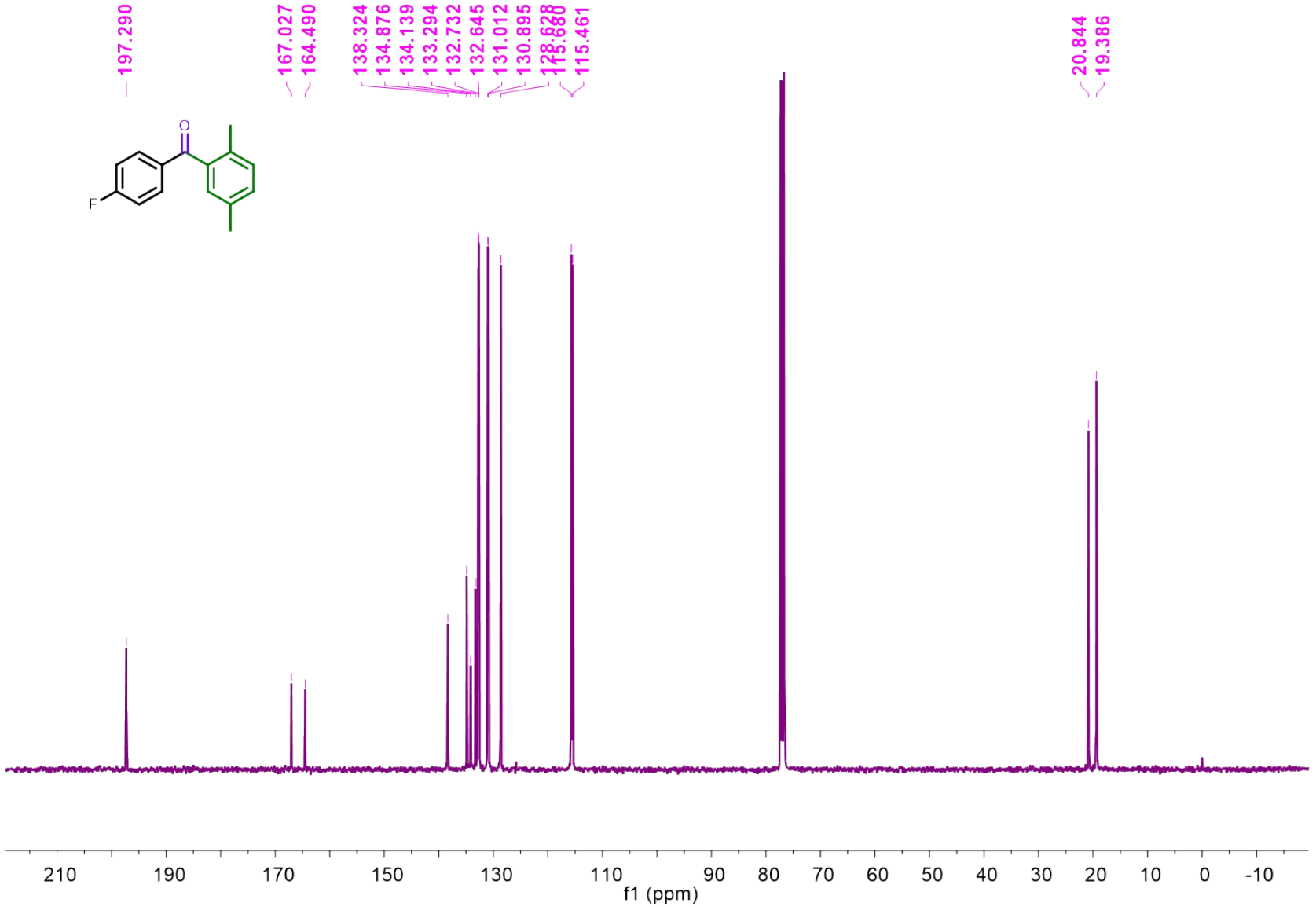


Figure S30. ^{13}C NMR spectra of (2,5-dimethylphenyl) (4-fluorophenyl) methanone **(2f)** in $CDCl_3$.

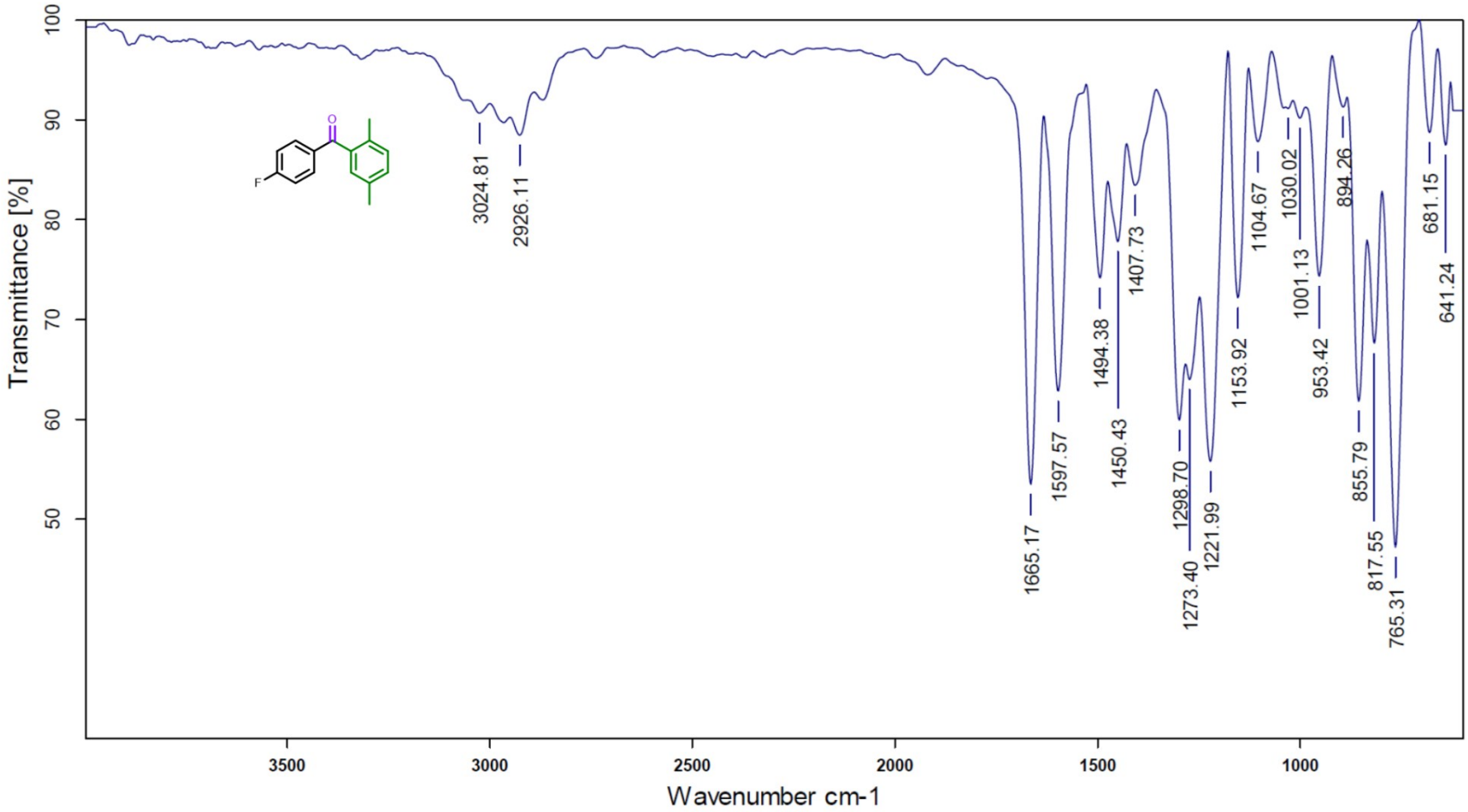


Figure S31. IR spectra of (2,5-dimethylphenyl) (4-fluorophenyl) methanone **(2f**).

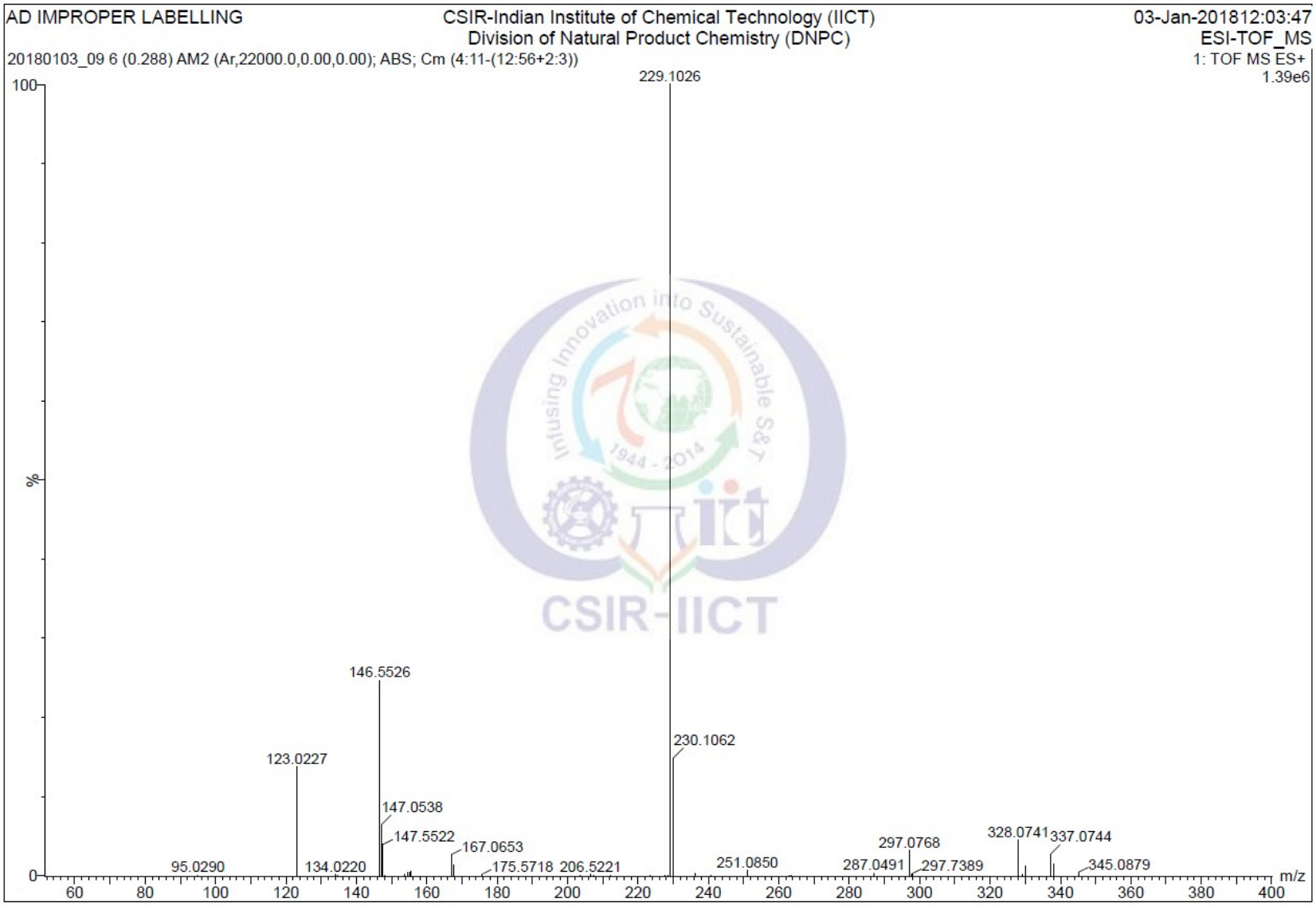


Figure S32. HRMS spectra of (2, 5-dimethylphenyl) (4-fluorophenyl) methanone (**2f**).

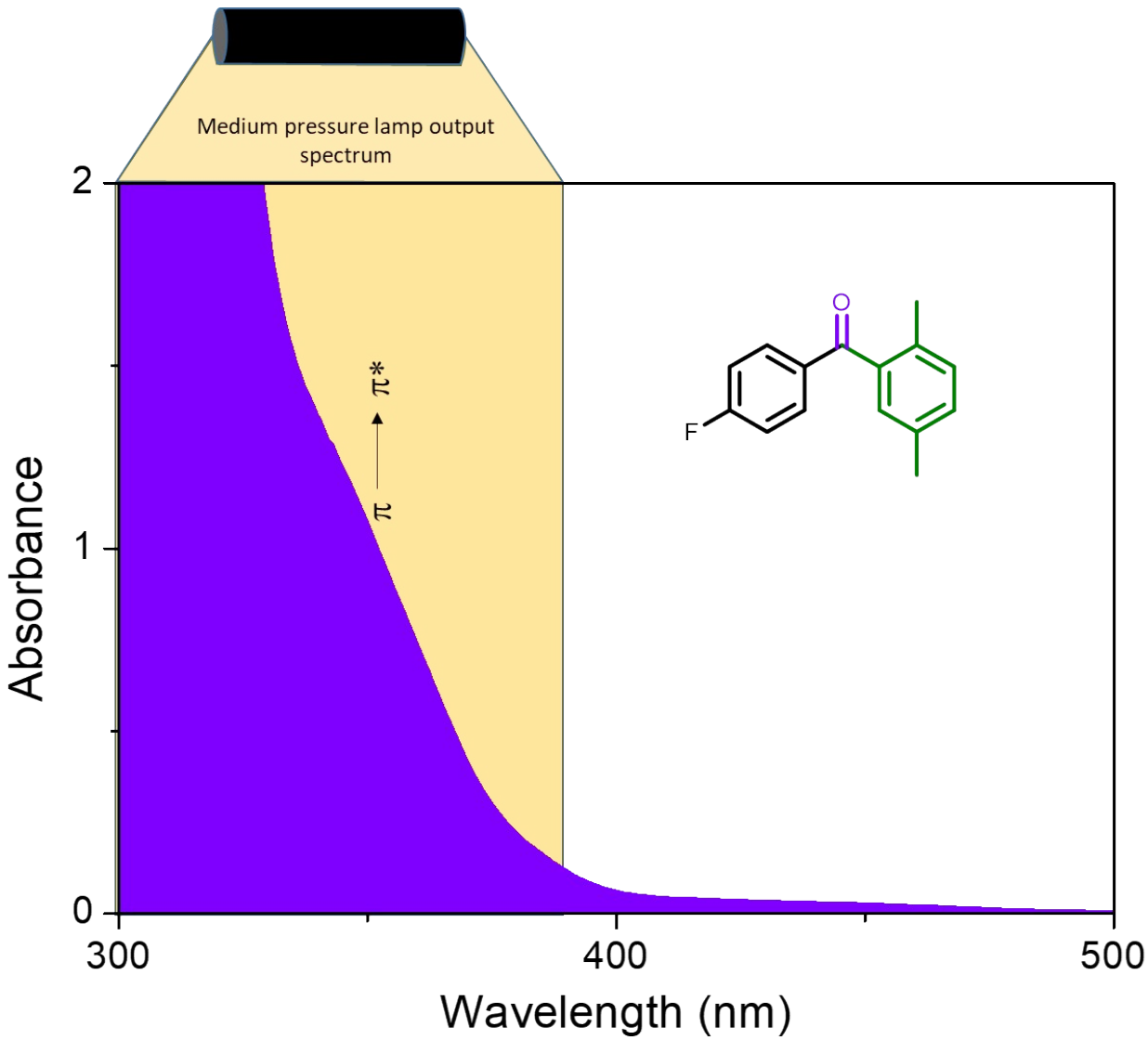


Figure S33. UV-Vis spectra of (2, 5-dimethylphenyl) (4-fluorophenyl) methanone (**2f**) in 0.005M MeCN.

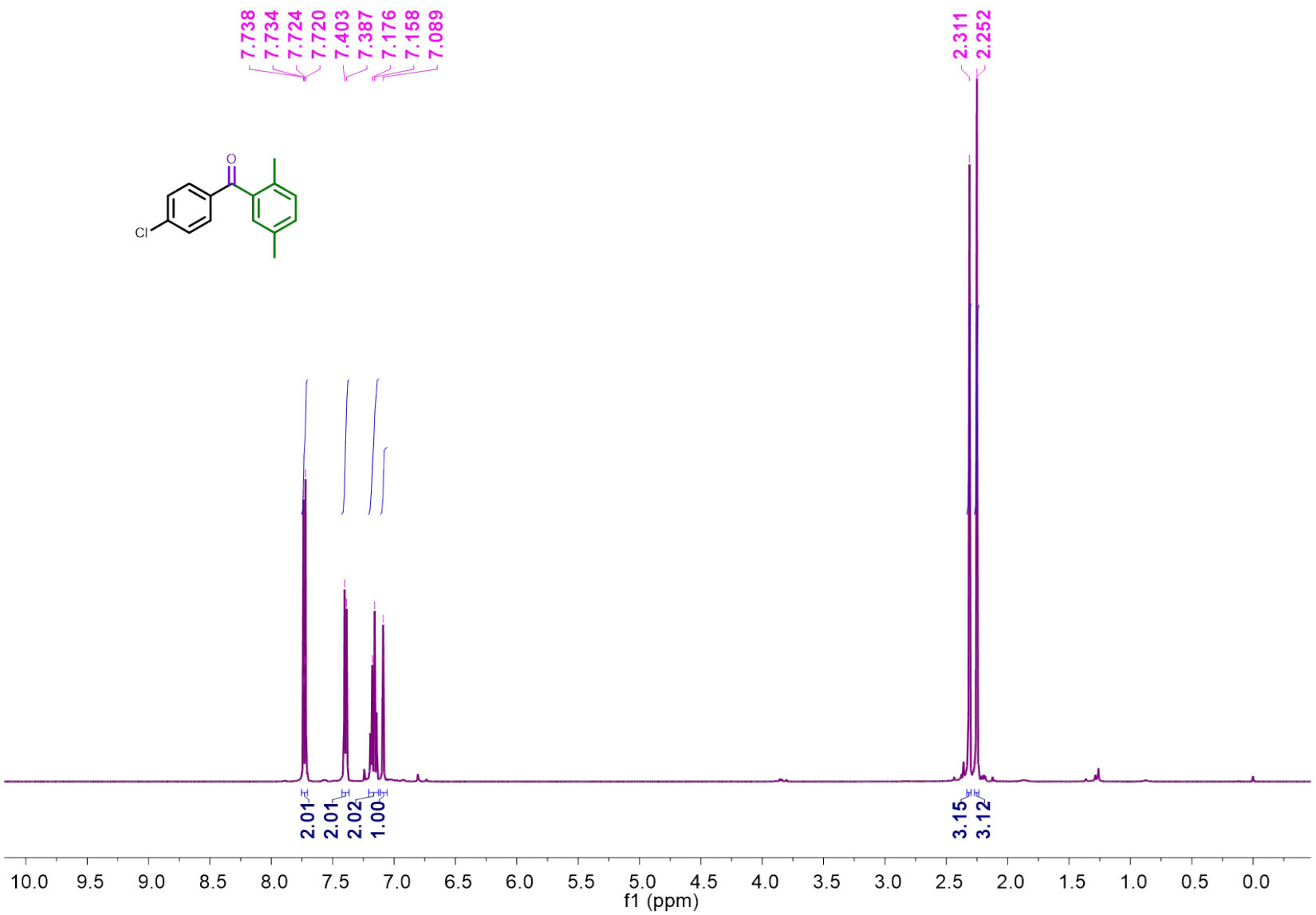


Figure S34. ^{1}H NMR spectra of (2,5-dimethylphenyl) (4-chlorophenyl) methanone **(2g)** in $CDCl_3$.

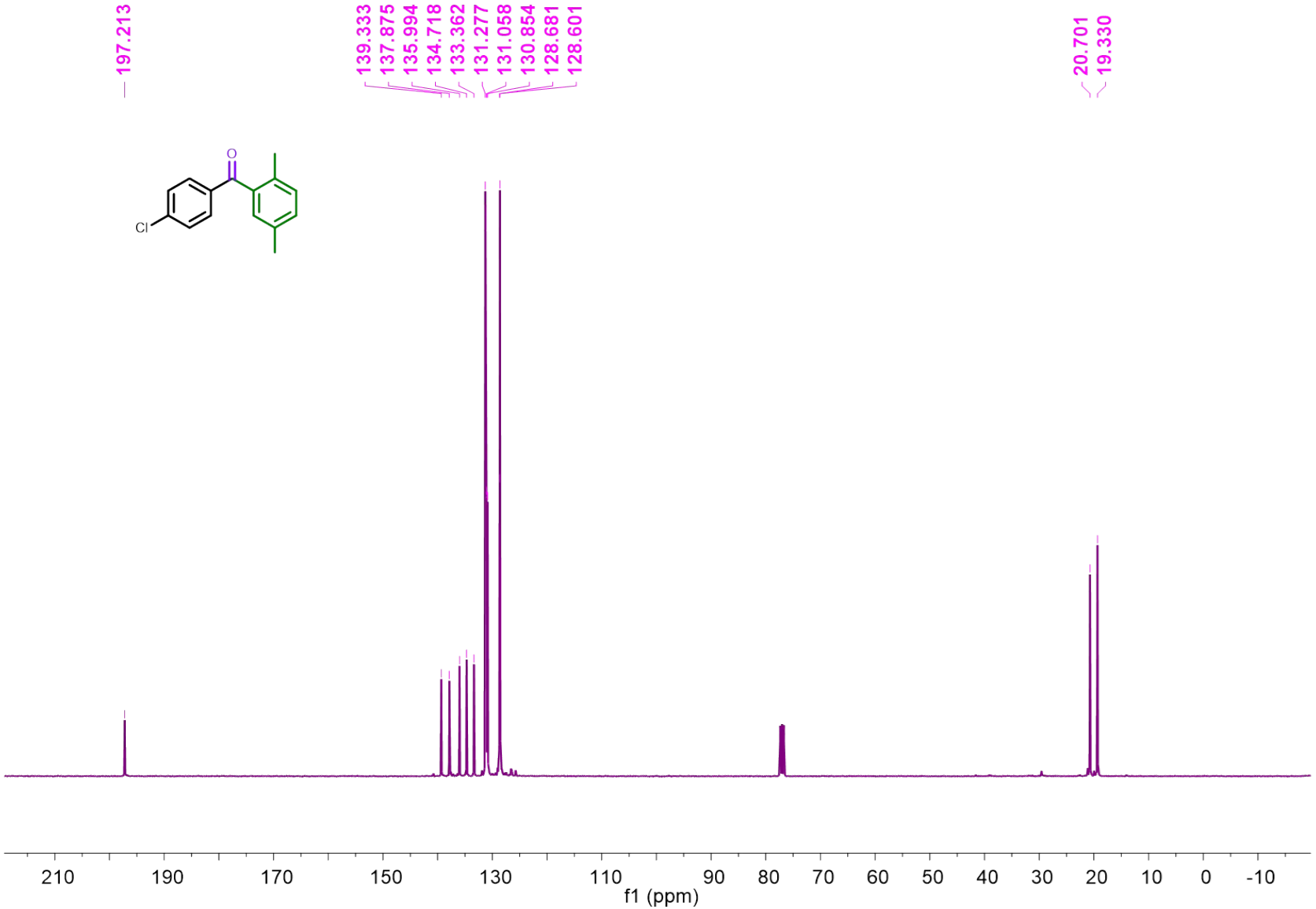


Figure S35. ^{13}C NMR spectra of (2,5-dimethylphenyl) (4-chlorophenyl) methanone **(2g)** in $CDCl_3$.

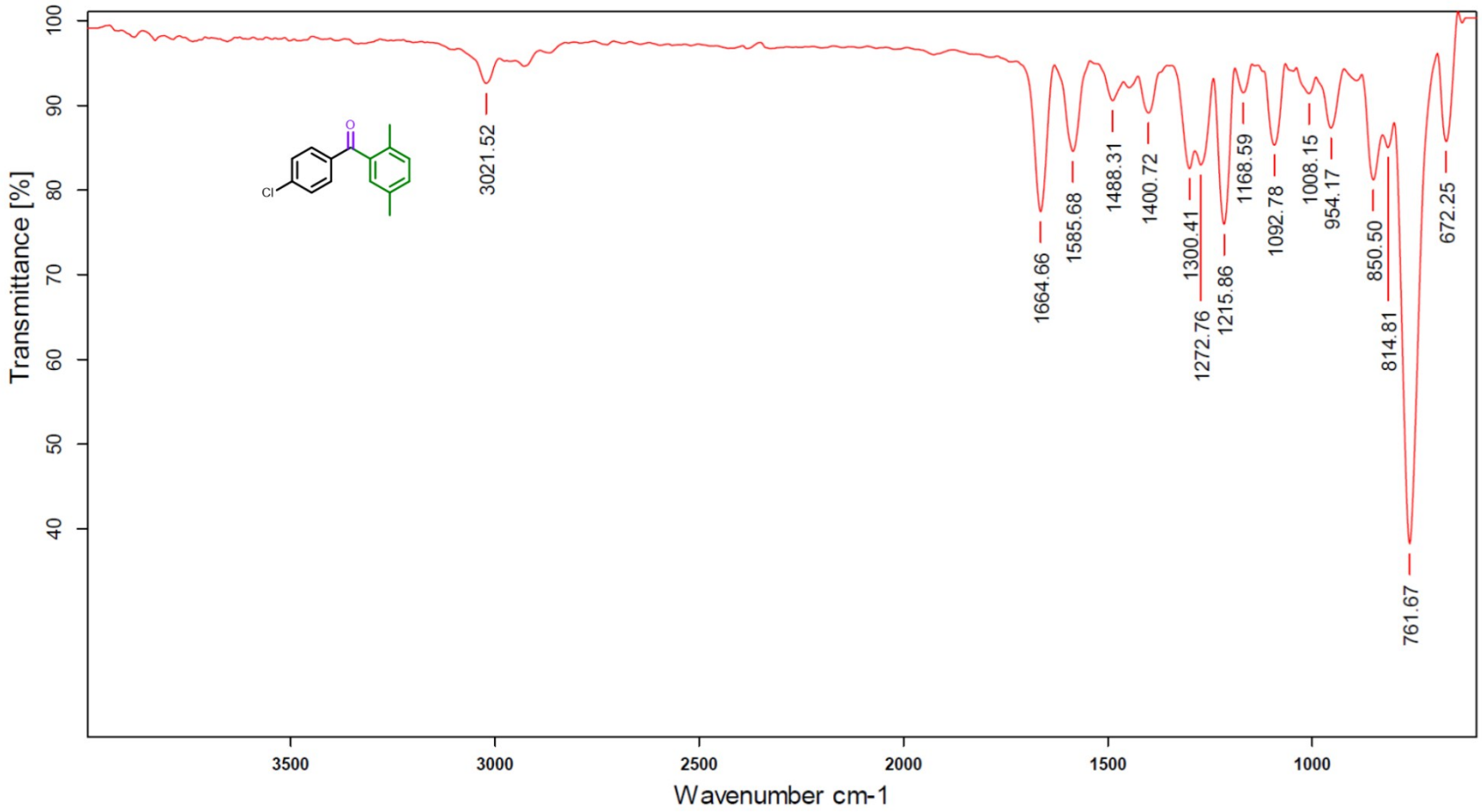


Figure S36. IR spectra of (2,5-dimethylphenyl) (4-chlorophenyl) methanone **(2g)**.

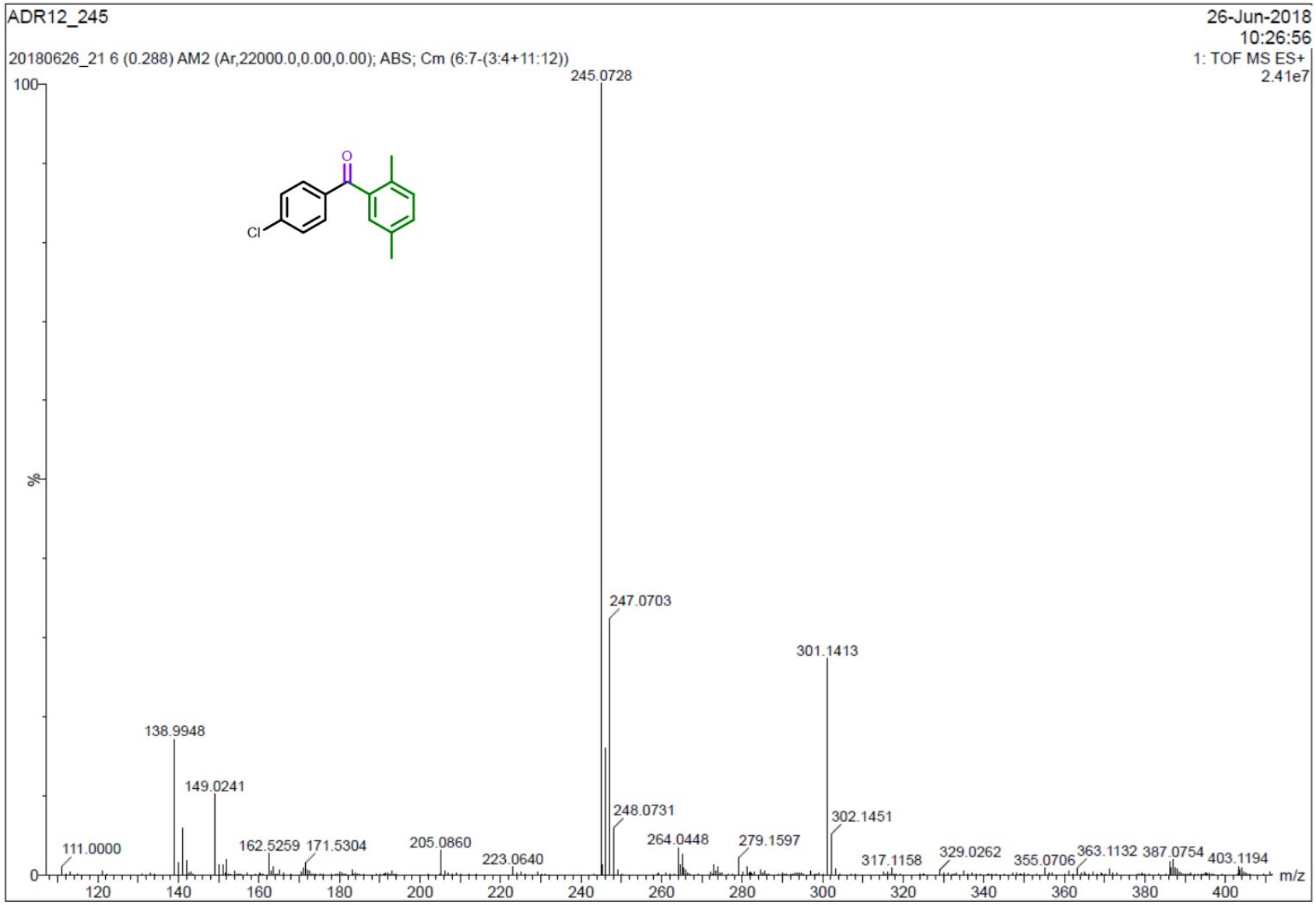


Figure S37. HRMS spectra of (2,5-dimethylphenyl) (4-chlorophenyl) methanone **(2g)**.

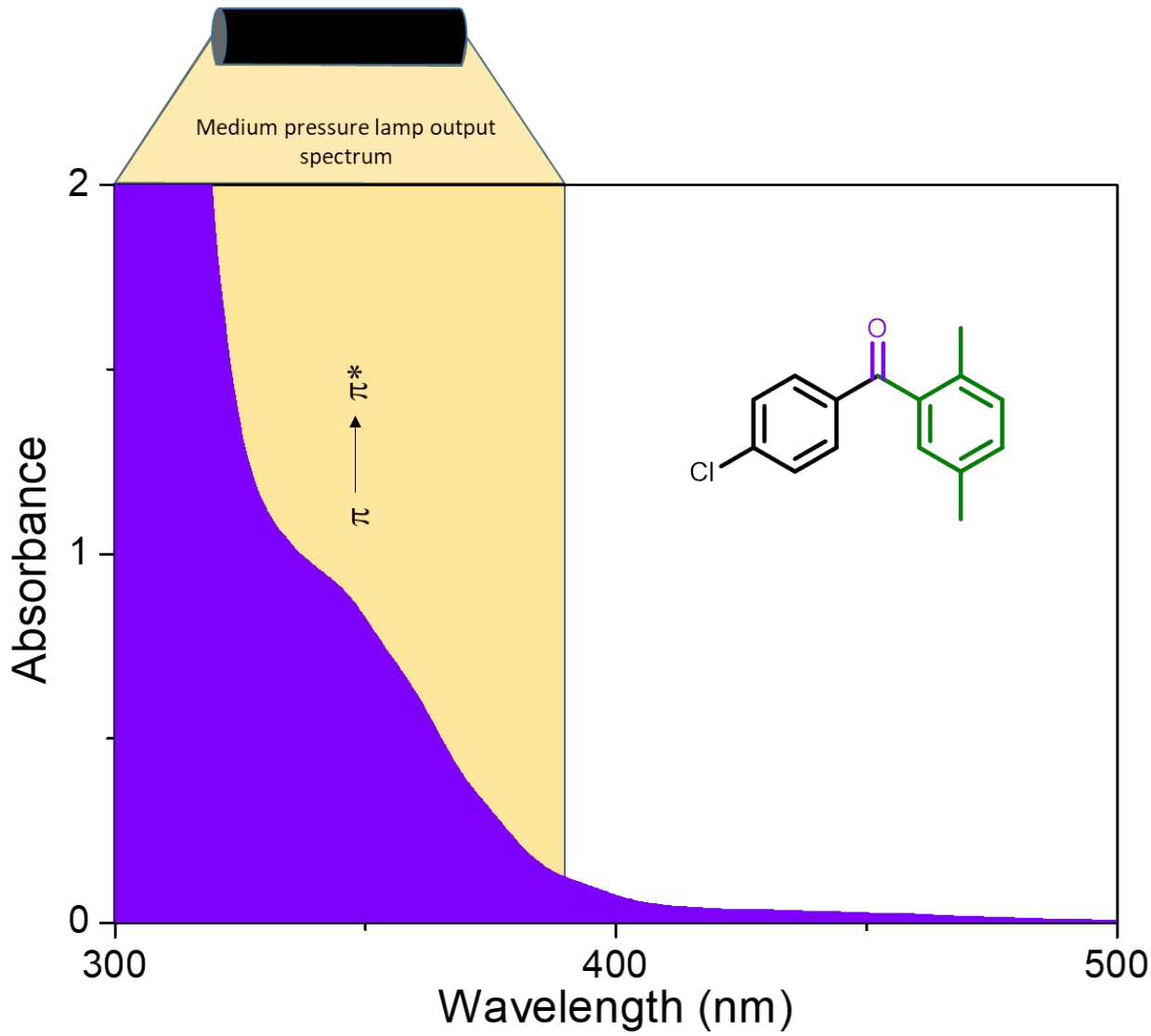


Figure S38. UV-Vis spectra of (2,5-dimethylphenyl) (4-chlorophenyl) methanone **(2g)** in 0.005M ACN.

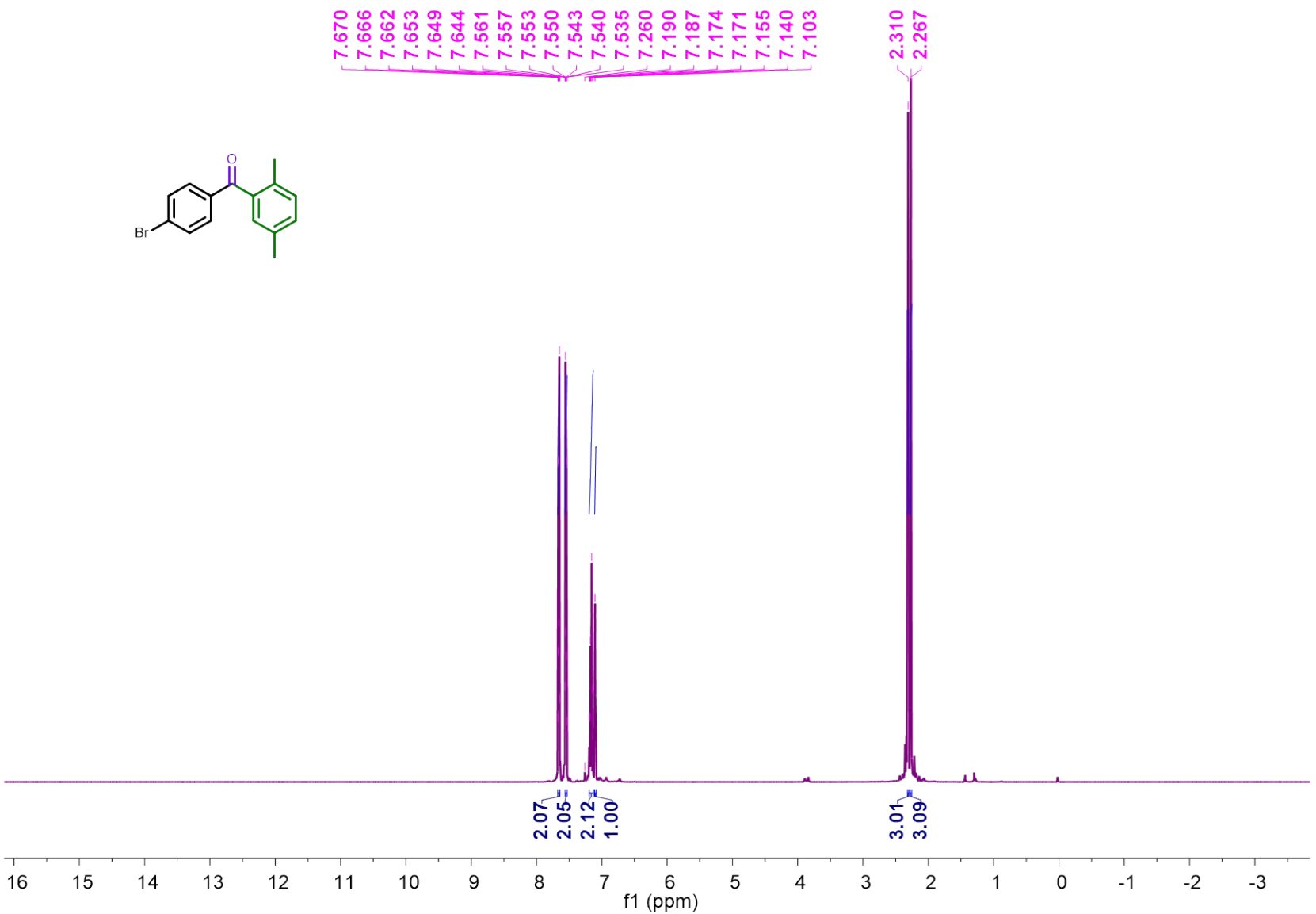


Figure S39. ^{1}H NMR spectra of (2,5-dimethylphenyl) (4-bromophenyl) methanone **(2h)** in $CDCl_3$.

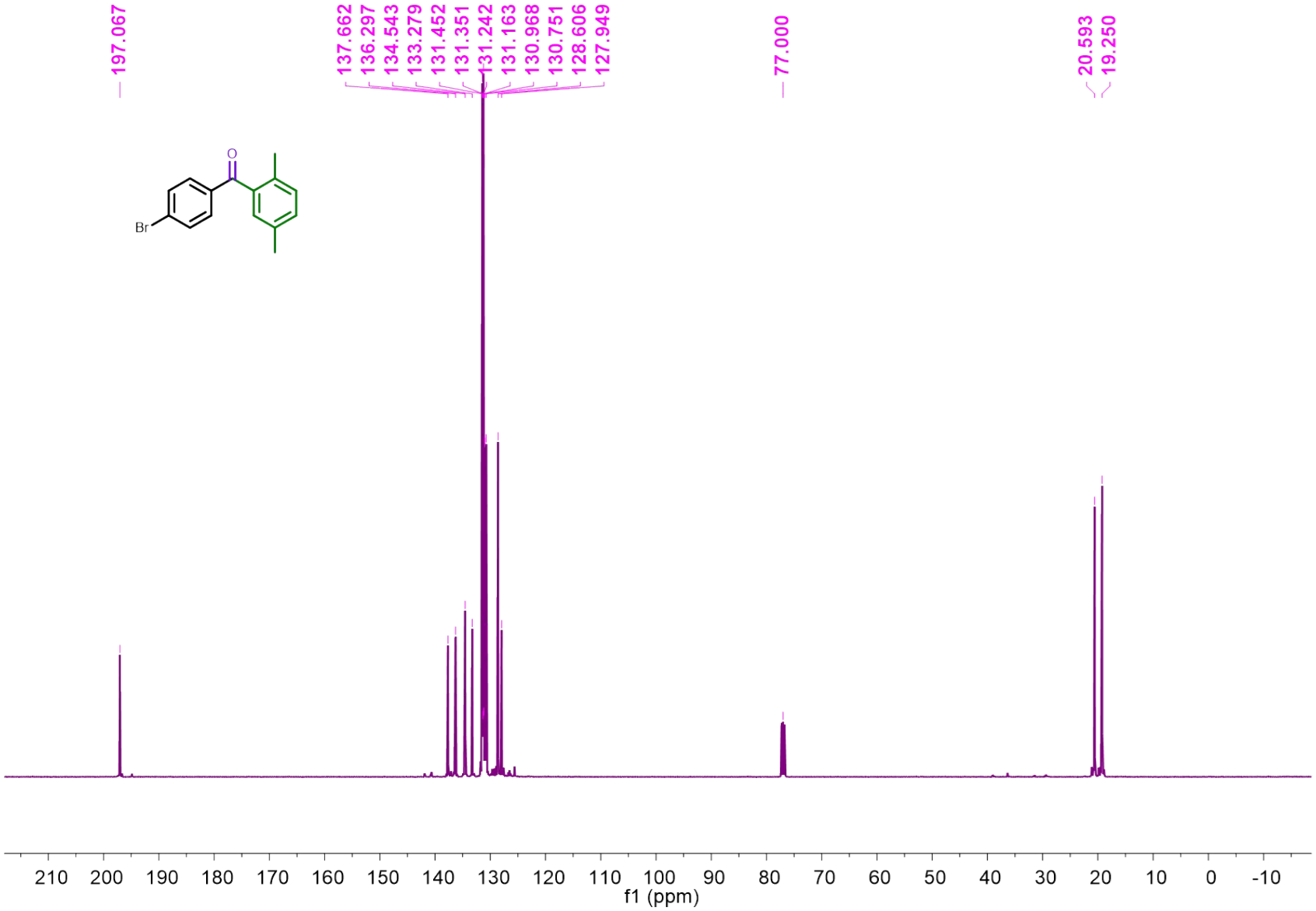


Figure S40. ^{13}C NMR spectra of (2,5-dimethylphenyl) (4-bromophenyl) methanone **(2h)** in $CDCl_3$.

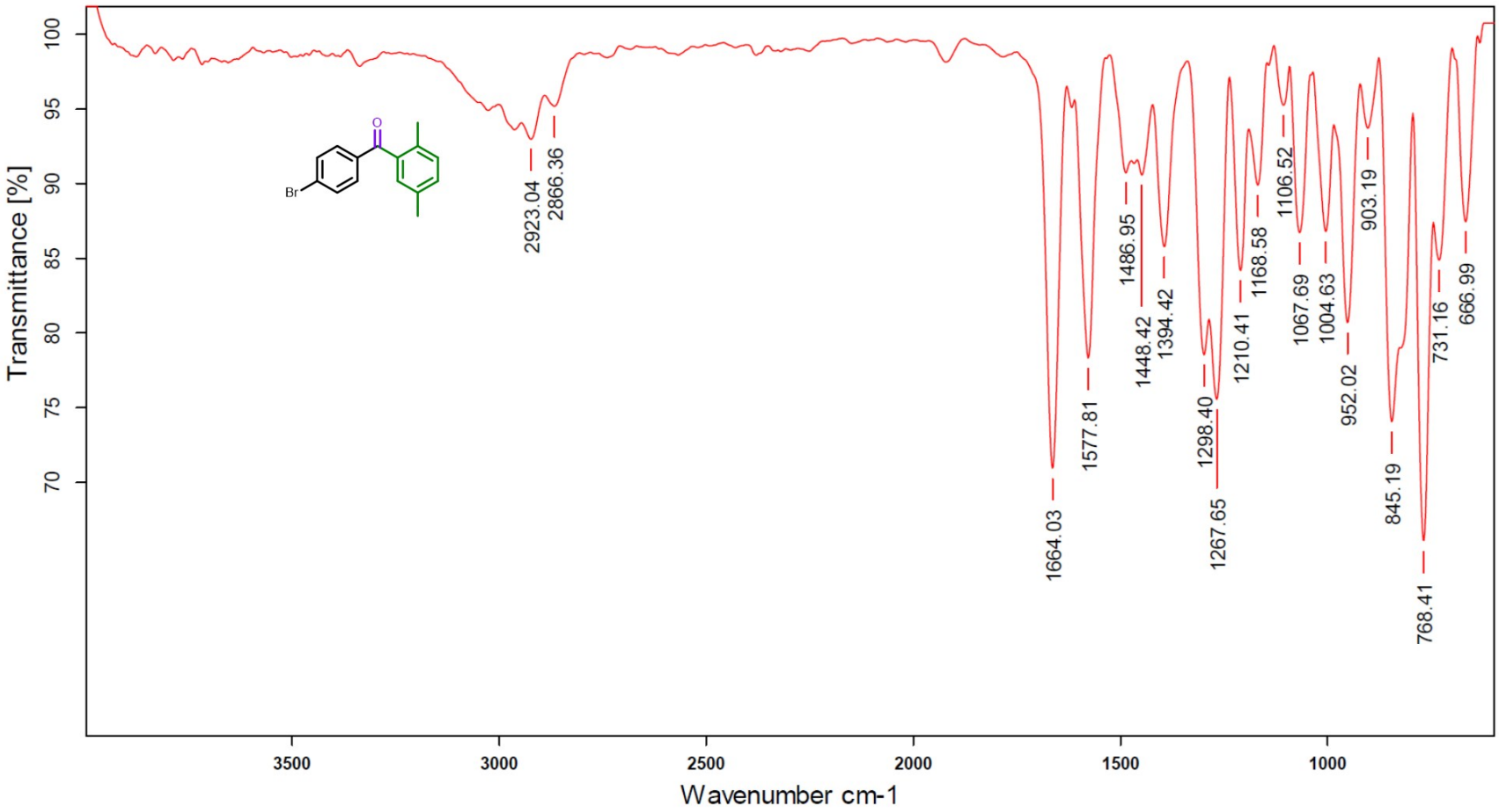


Figure S41. IR spectra of (2,5-dimethylphenyl) (4-bromophenyl) methanone **(2h**).

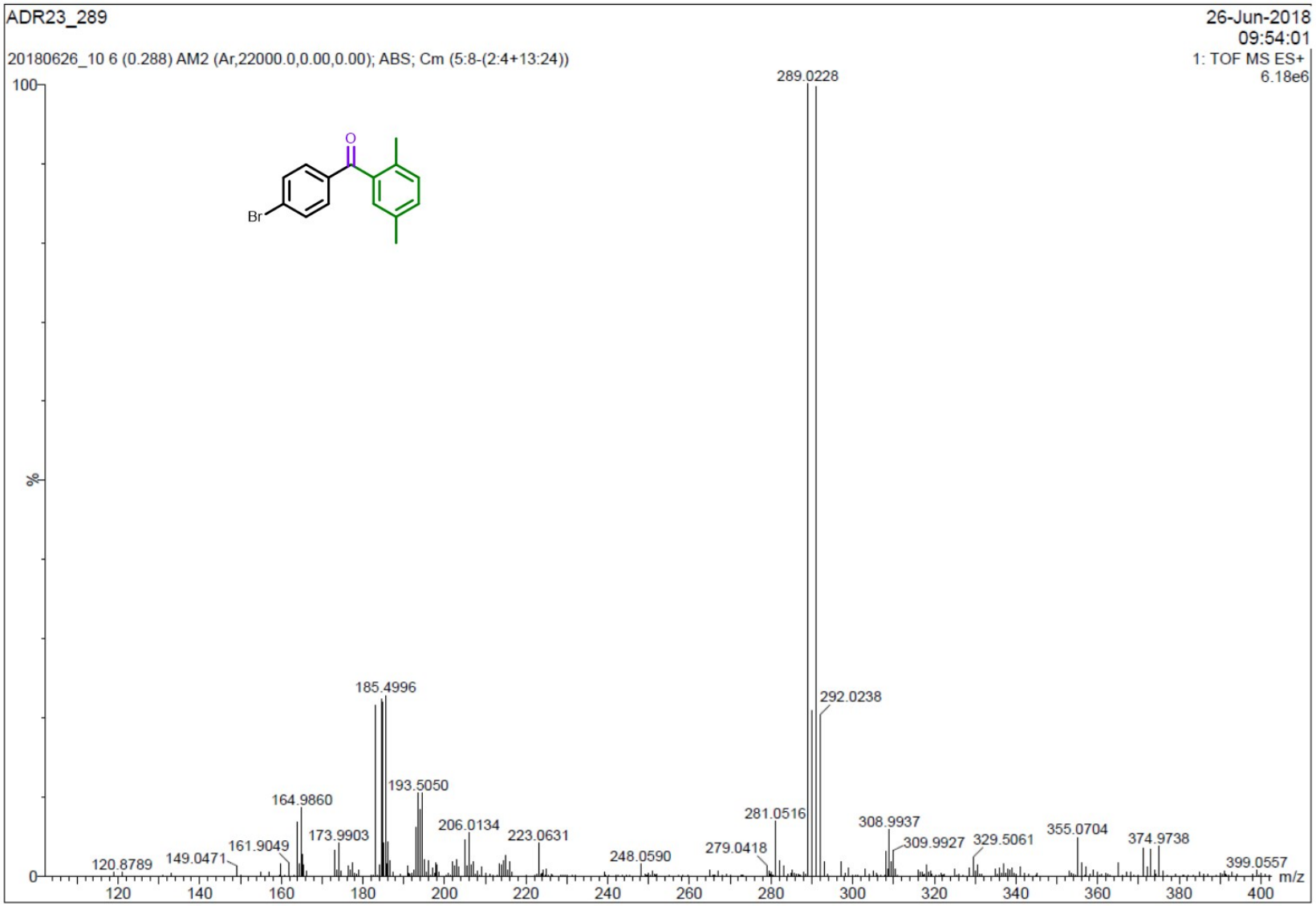


Figure S42. HRMS spectra of (2,5-dimethylphenyl) (4-bromophenyl) methanone **(2h)**.

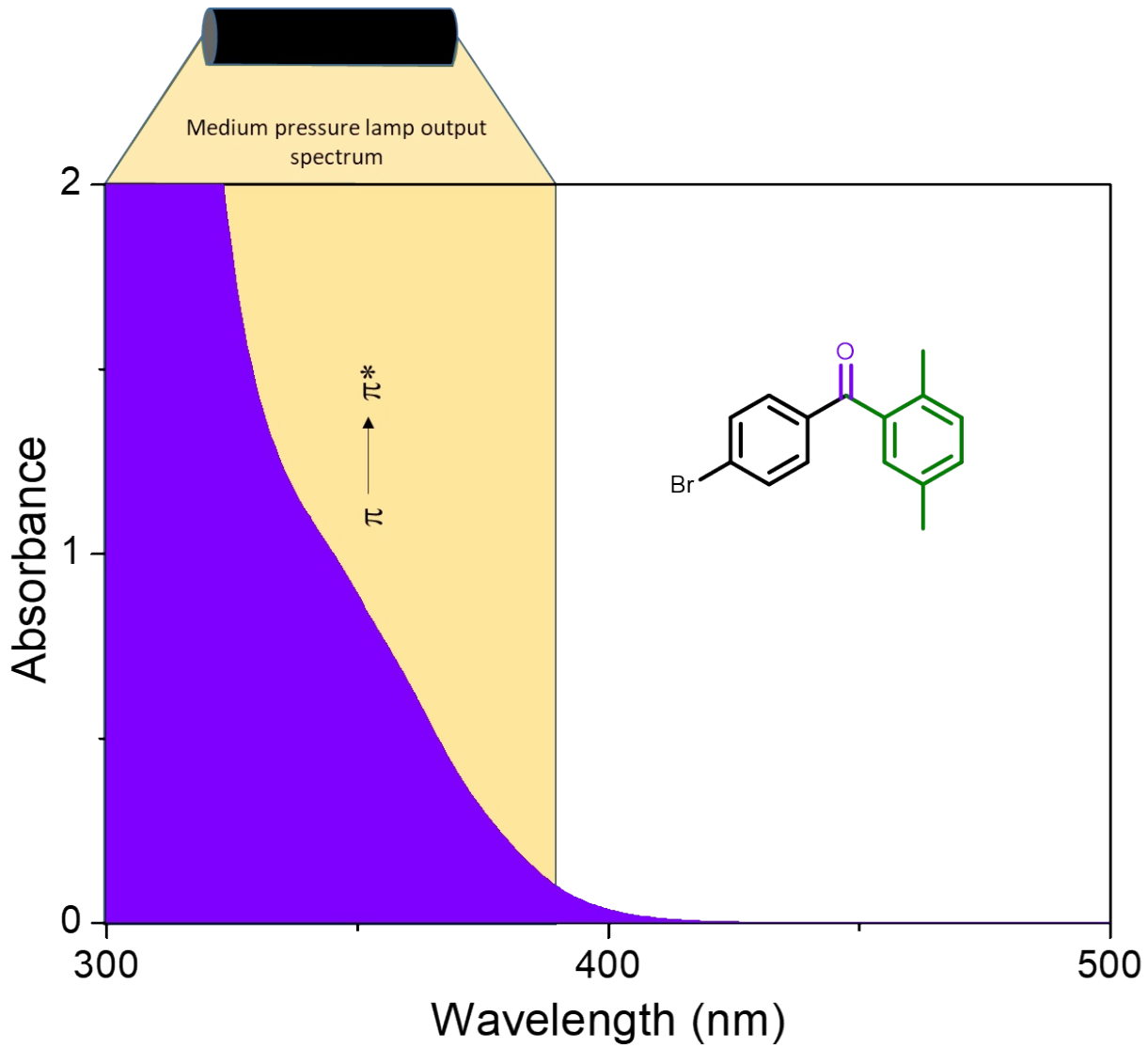


Figure S43. UV-Vis spectra of (2,5-dimethylphenyl) (4-bromophenyl) methanone **(2h)** in 0.005M ACN.

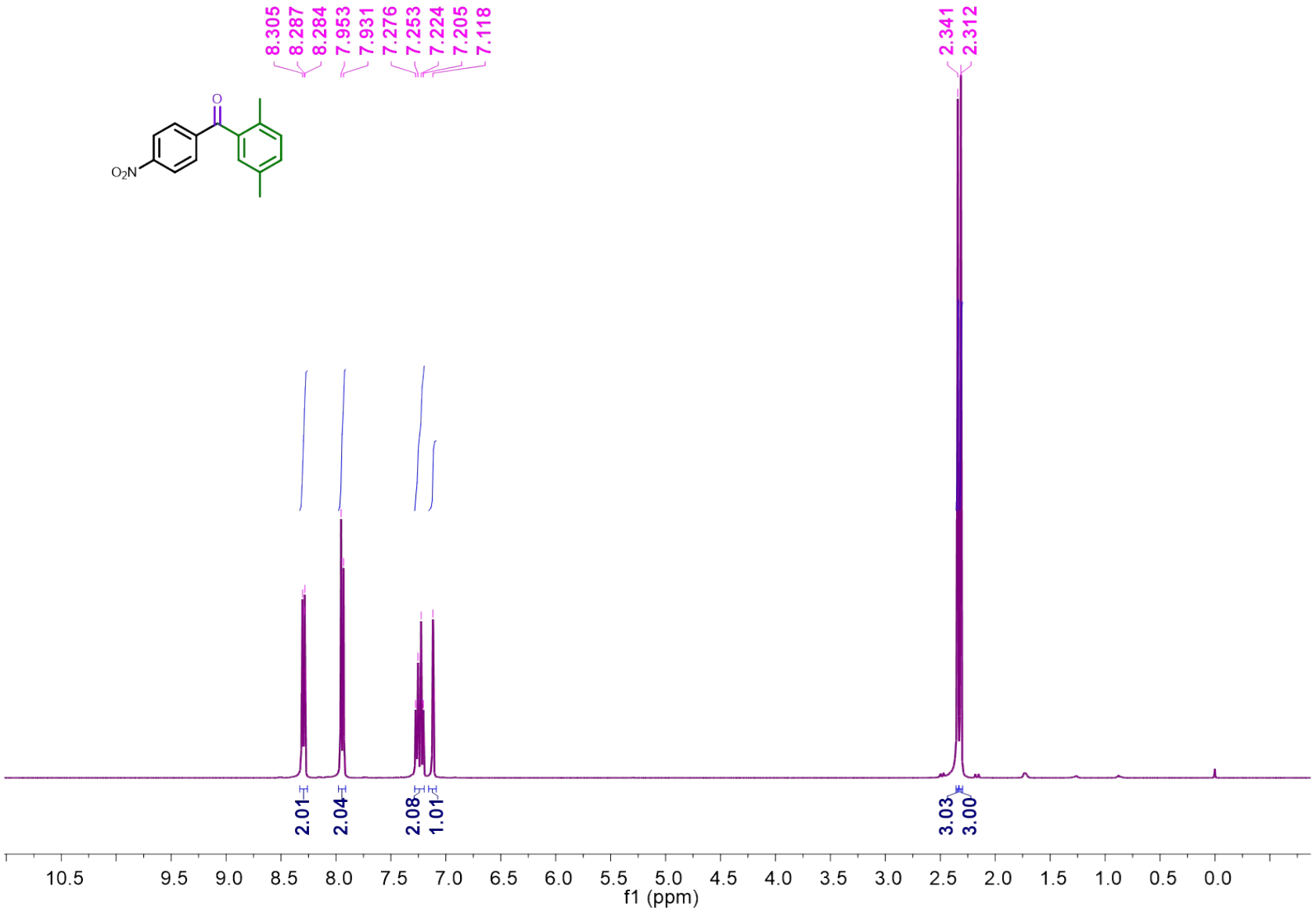


Figure S44. ^{1}H NMR spectra of (2,5-dimethylphenyl) (4-nitrophenyl) methanone **(2i)** in $CDCl_3$.

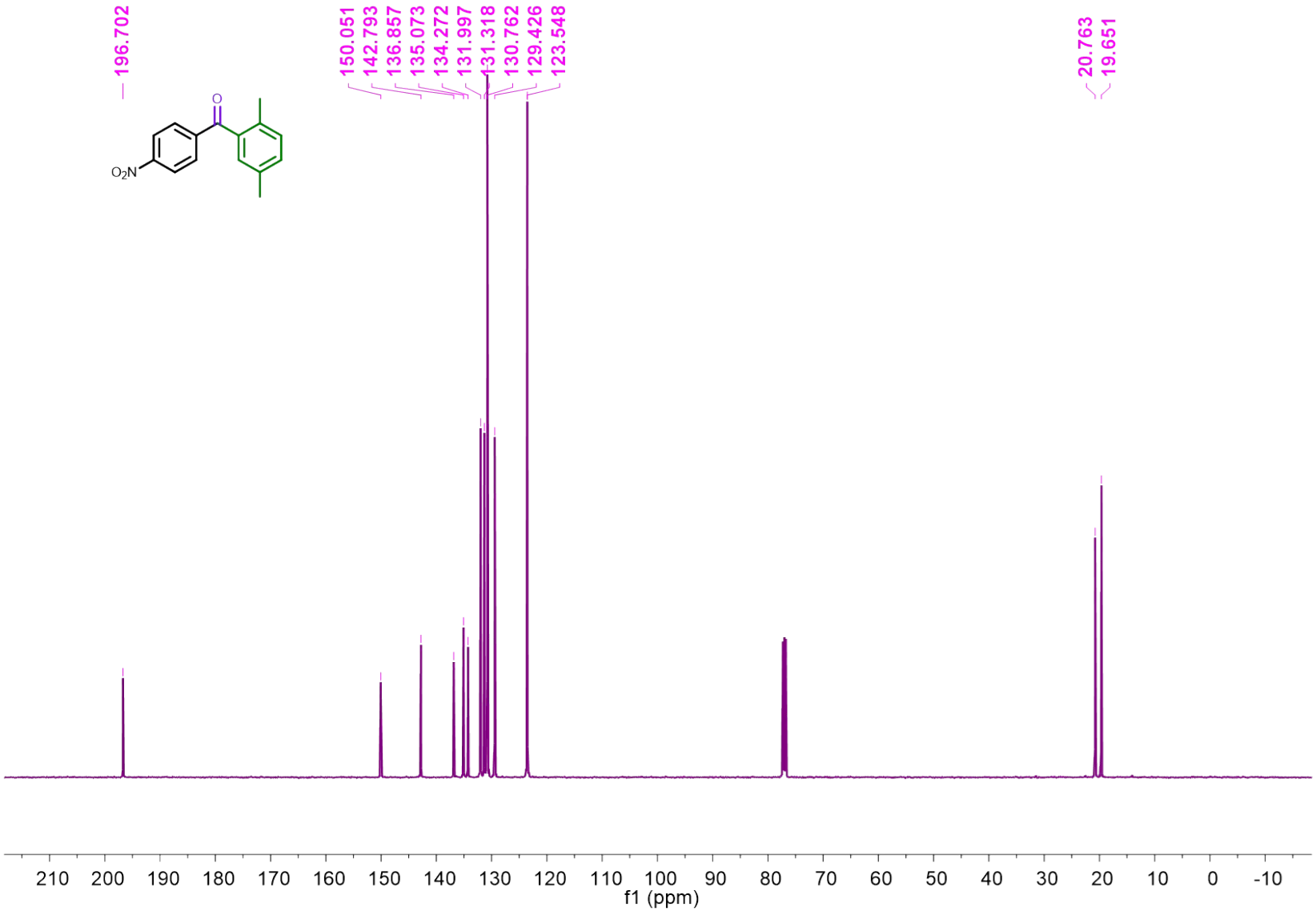


Figure S45. ^{13}C NMR spectra of (2,5-dimethylphenyl) (4-nitrophenyl) methanone **(2i)** in $CDCl_3$.

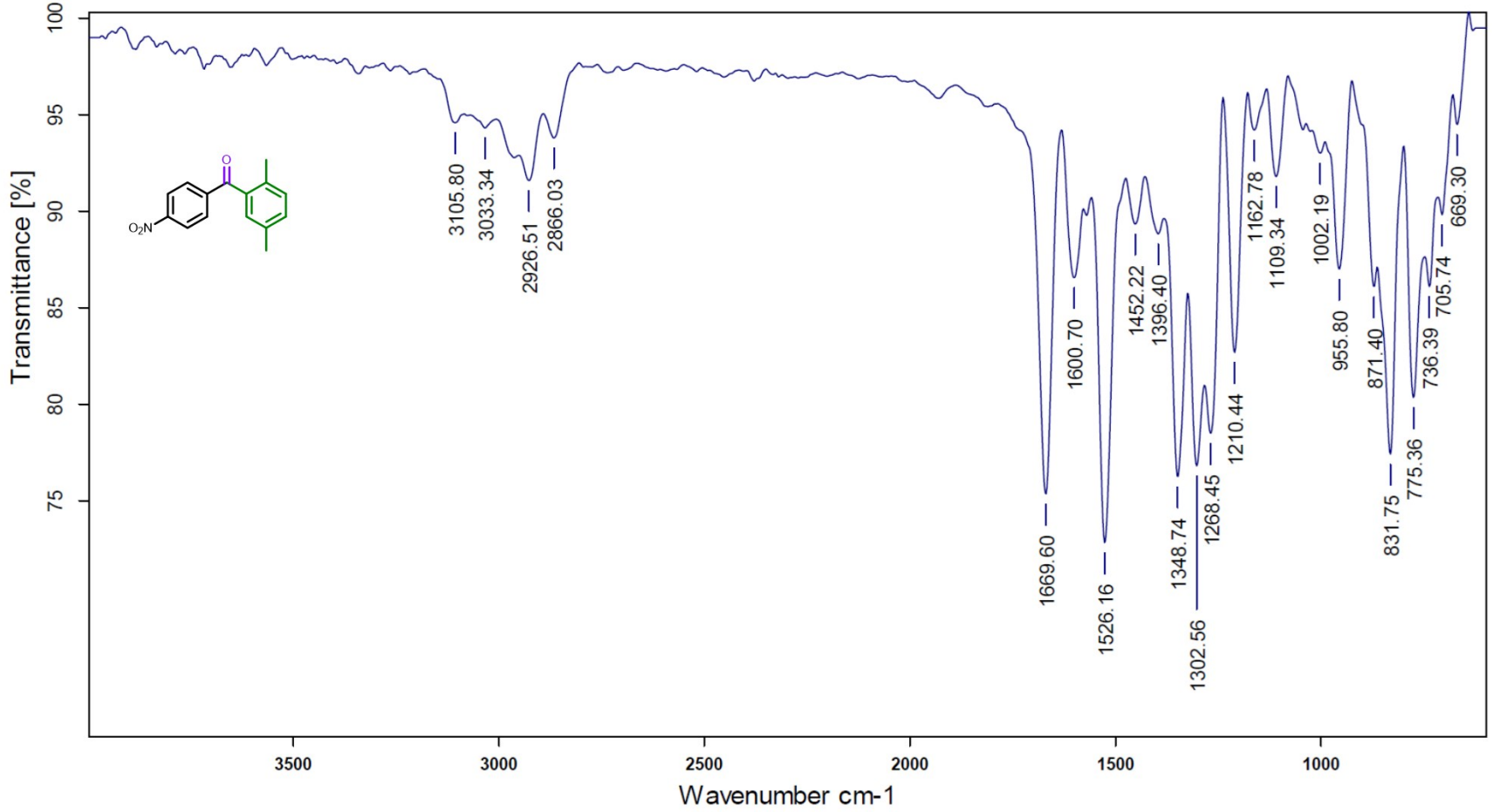
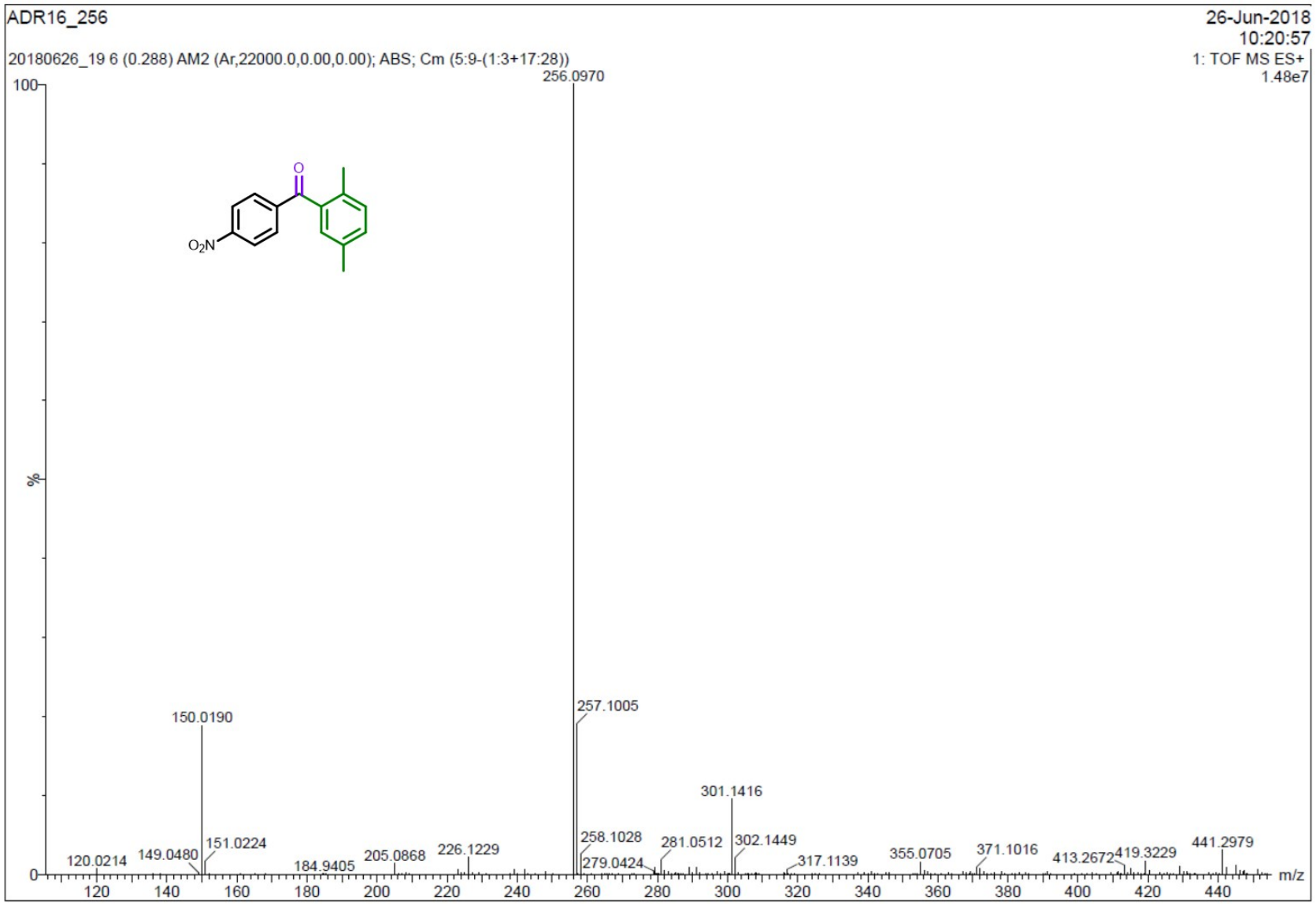


Figure S46. IR spectra of (2,5-dimethylphenyl) (4-nitrophenyl) methanone **(2i)**.


ADR16_256
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10:20:57
20180626_19 6 (0.288) AM2 (Ar,22000.0,0.00,0.00); ABS; Cm (5:9-(1:3+17:28))
1: TOF MS ES+
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257.1005
150.0190
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120.0214
149.0480
151.0224
184.9405
205.0868
226.1229
258.1028
279.0424
281.0512
302.1449
317.1139
355.0705
371.1016
413.2672
419.3229
441.2979
m/z
120
140
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440

Fi

gure S47. HRMS spectra of (2,5-dimethylphenyl) (4-nitrophenyl) methanone **(2i**).

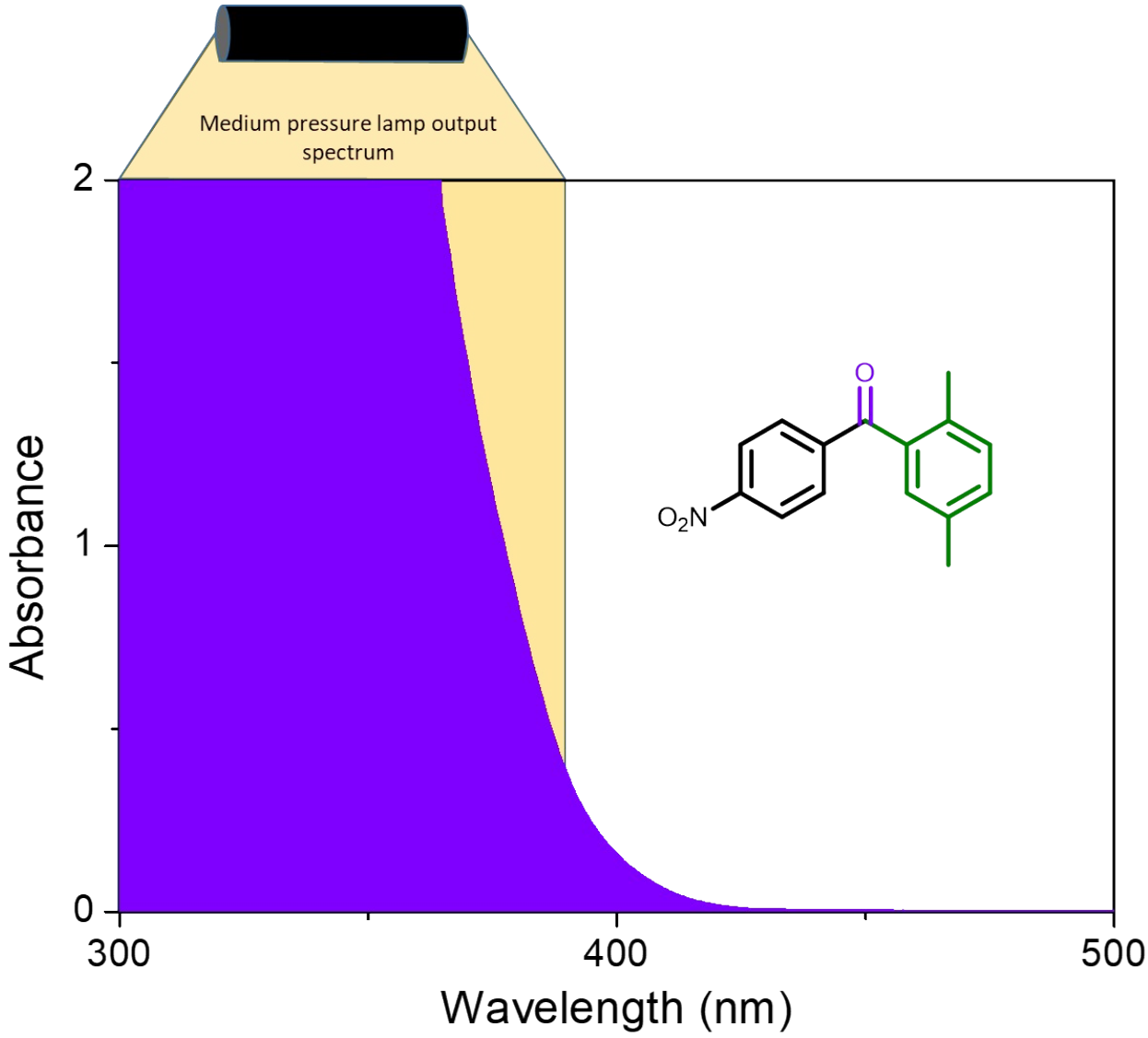


Figure S48. UV-Vis spectra of (2,5-dimethylphenyl) (4-nitrophenyl) methanone **(2i**) in 0.005M ACN.

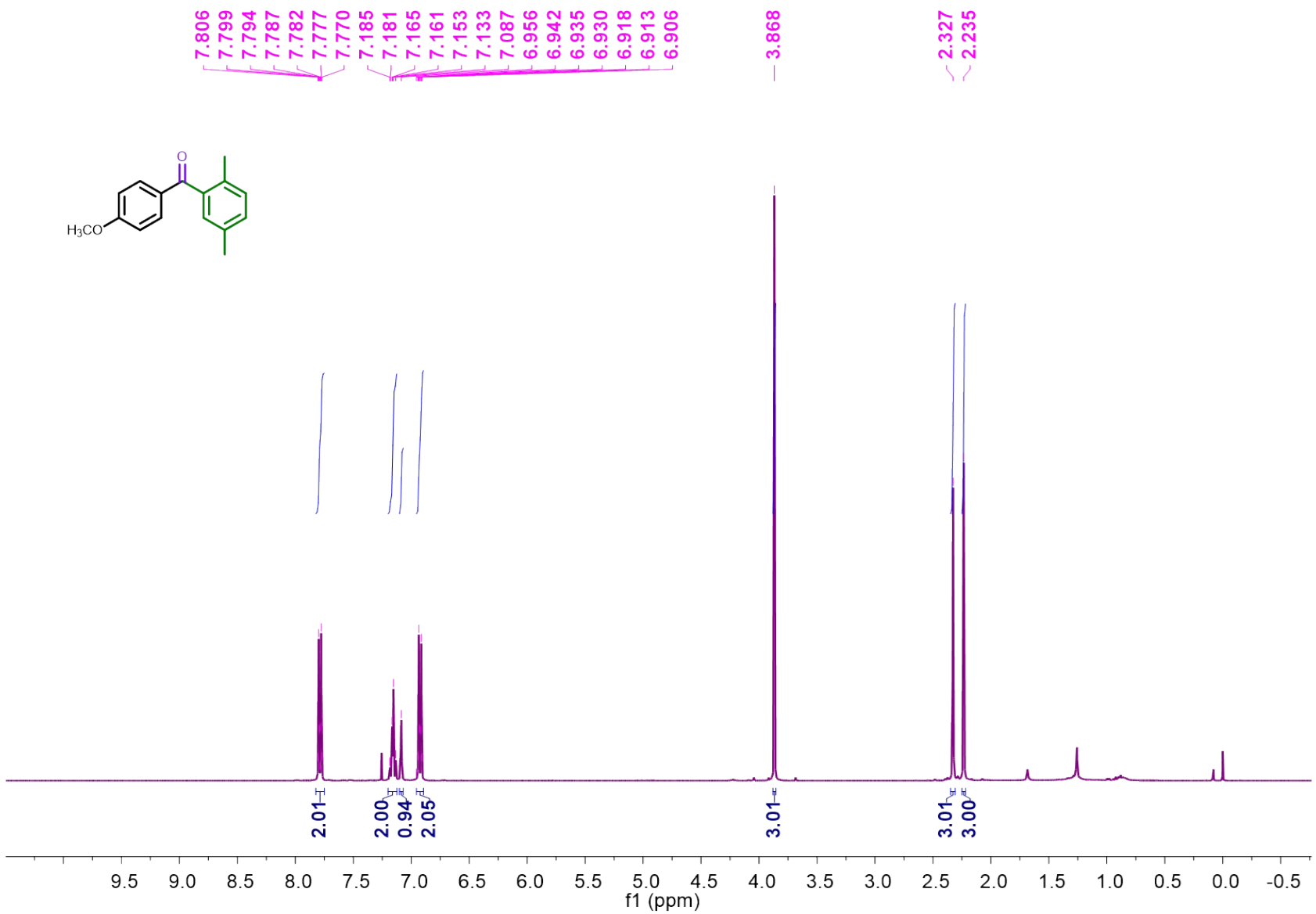


Figure S49. ^{1}H NMR spectra of (2, 5-dimethylphenyl) (4-methoxyphenyl) methanone **(2j)** in $CDCl_3$.

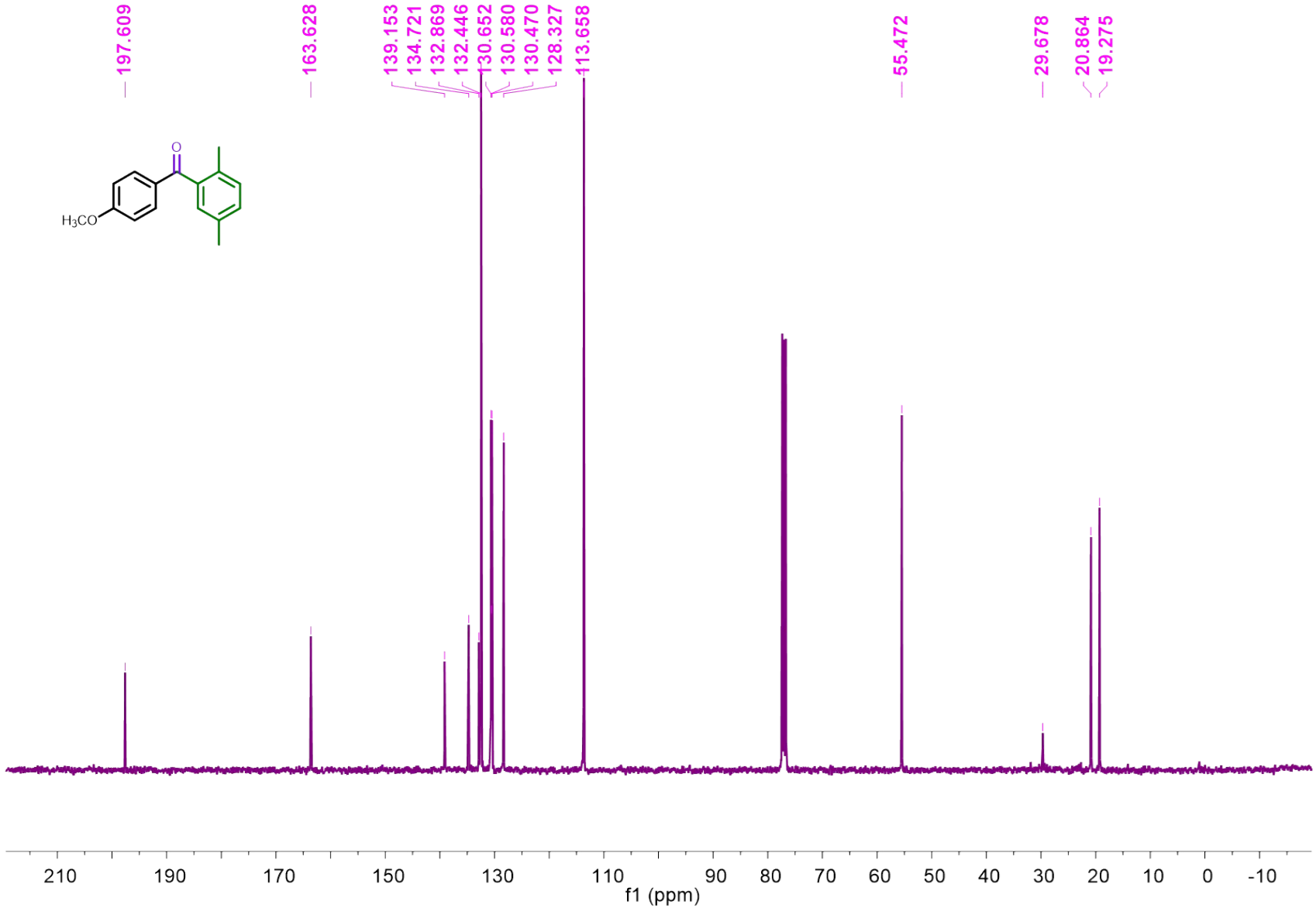


Figure S50. ^{13}C NMR spectra of (2, 5-dimethylphenyl) (4-methoxyphenyl) methanone **(2j)** in $CDCl_3$.

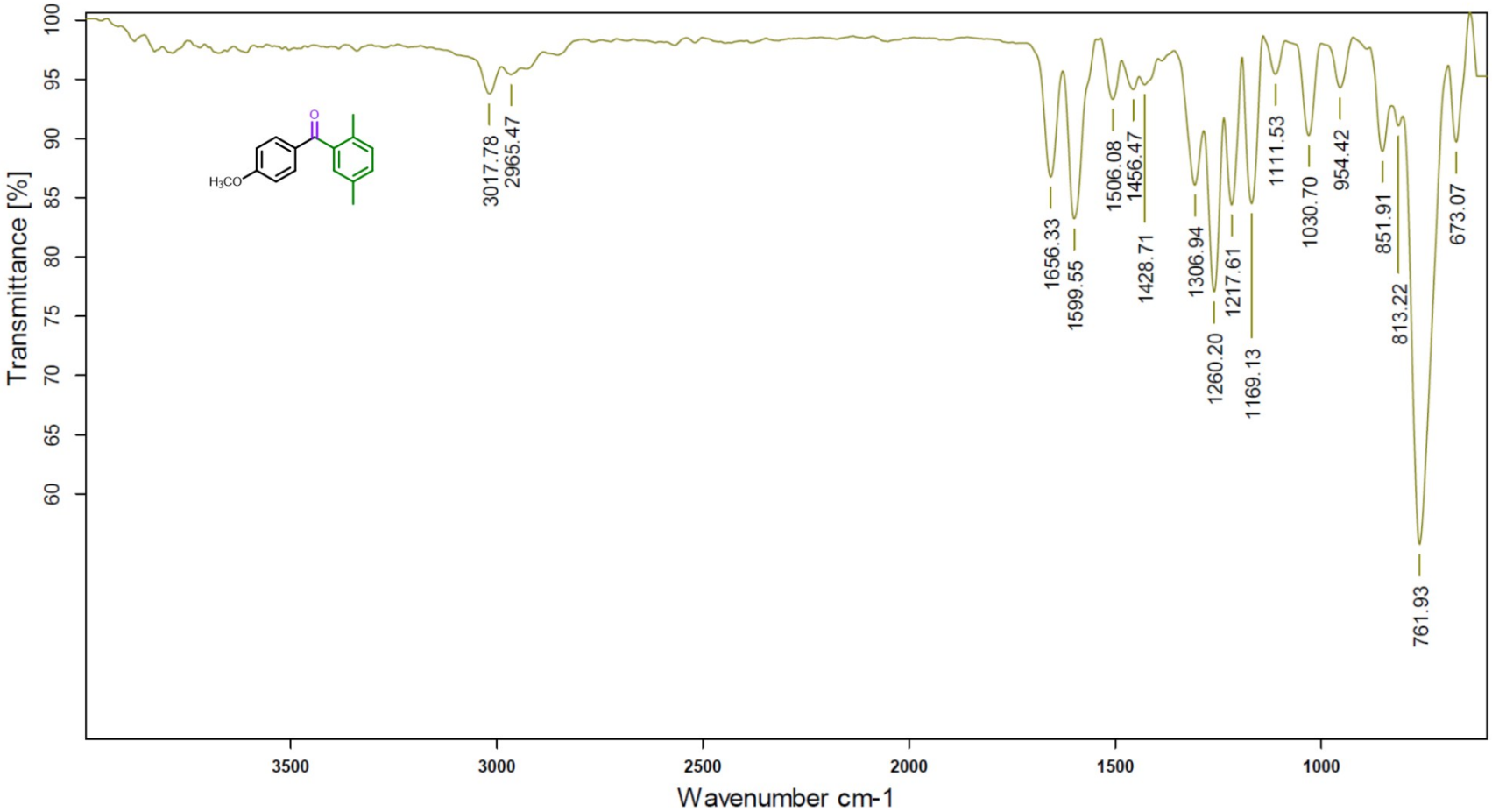


Figure S51. IR spectra of (2, 5-dimethylphenyl) (4-methoxyphenyl) methanone **(2j)**.

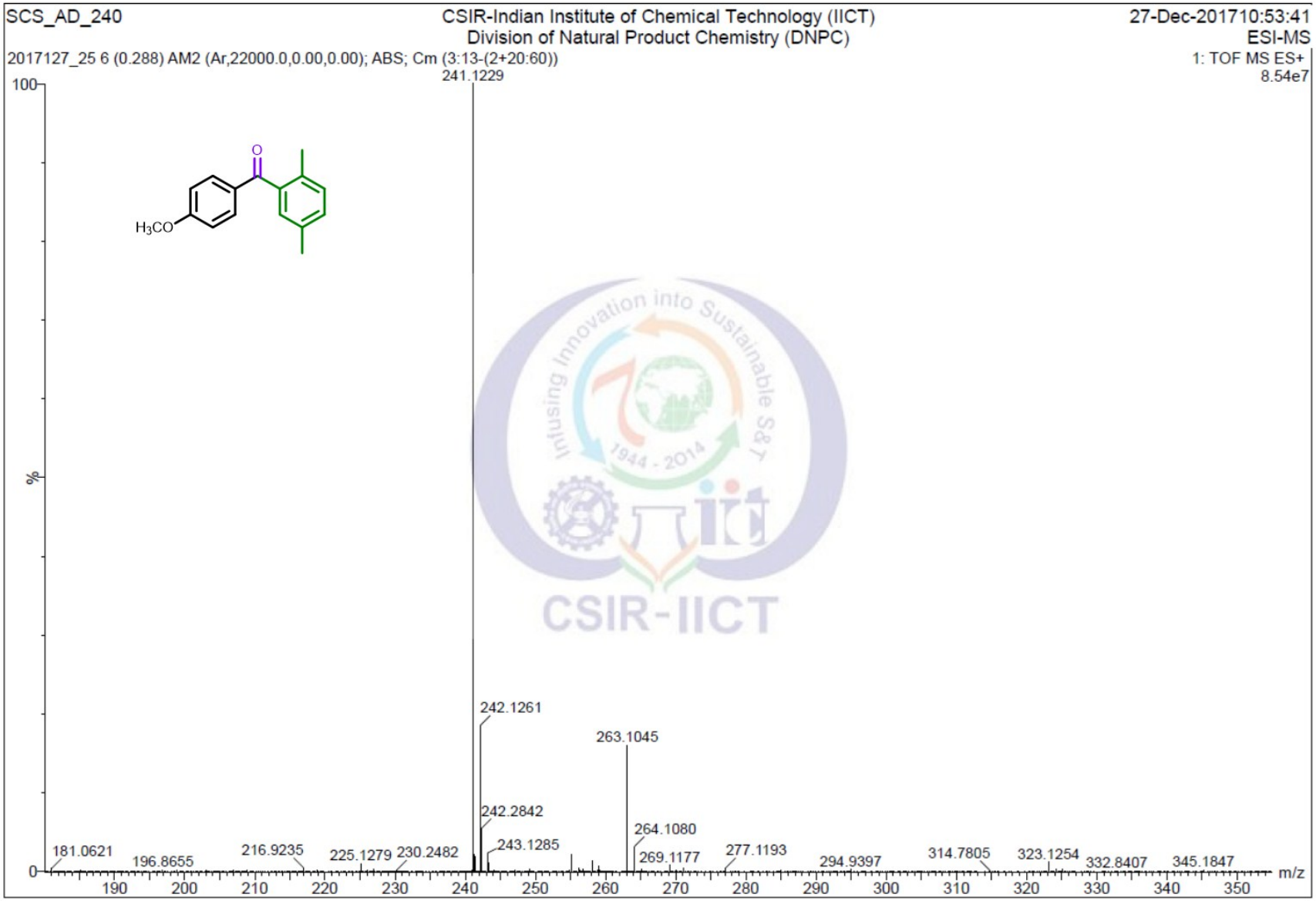


Figure S52. HRMS spectra of (2, 5-dimethylphenyl) (4-methoxyphenyl) methanone **(2j)**.

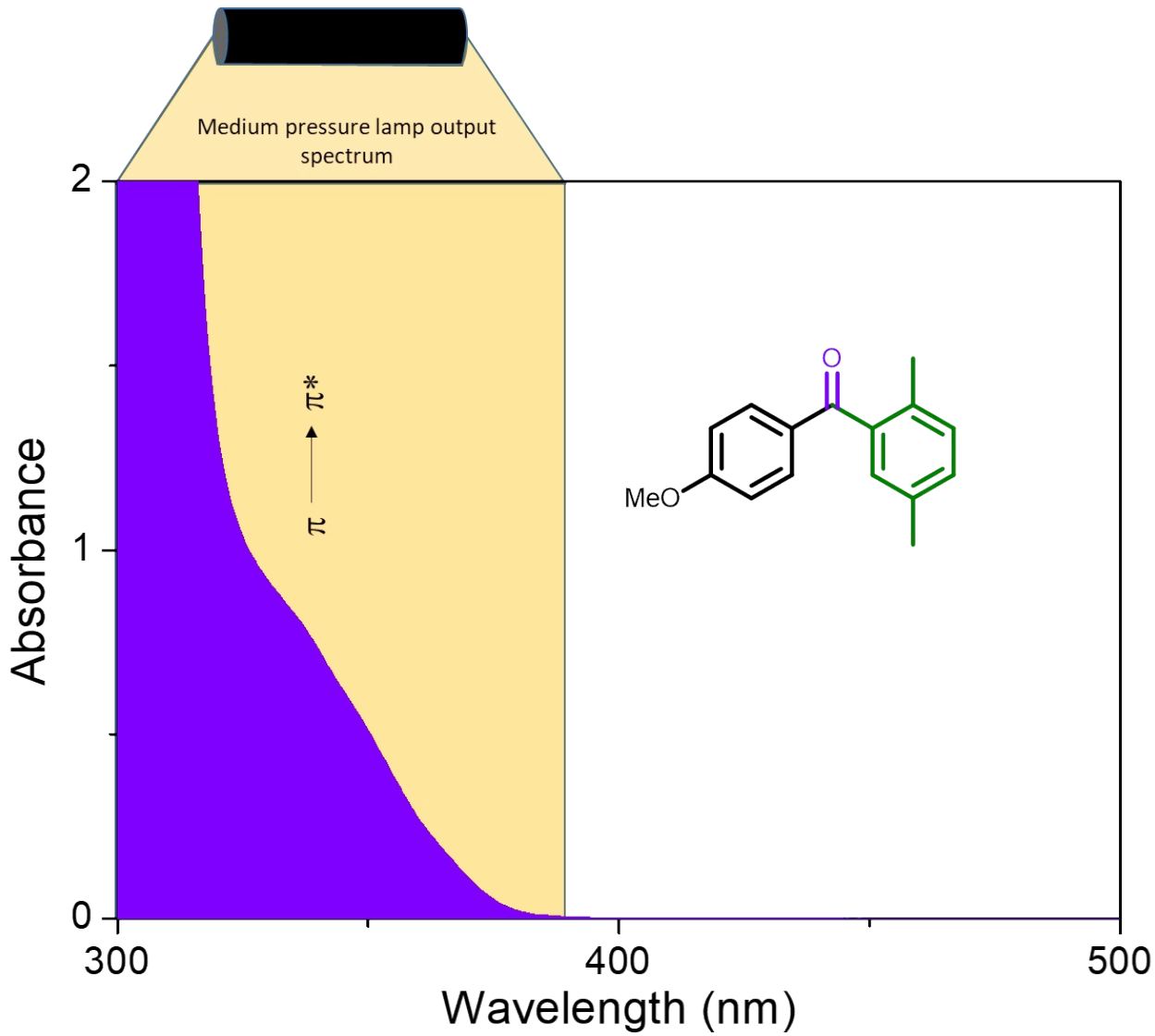


Figure S53. UV-Vis spectra of (2, 5-dimethylphenyl) (4-methoxyphenyl) methanone **(2j)** in 0.005M ACN.

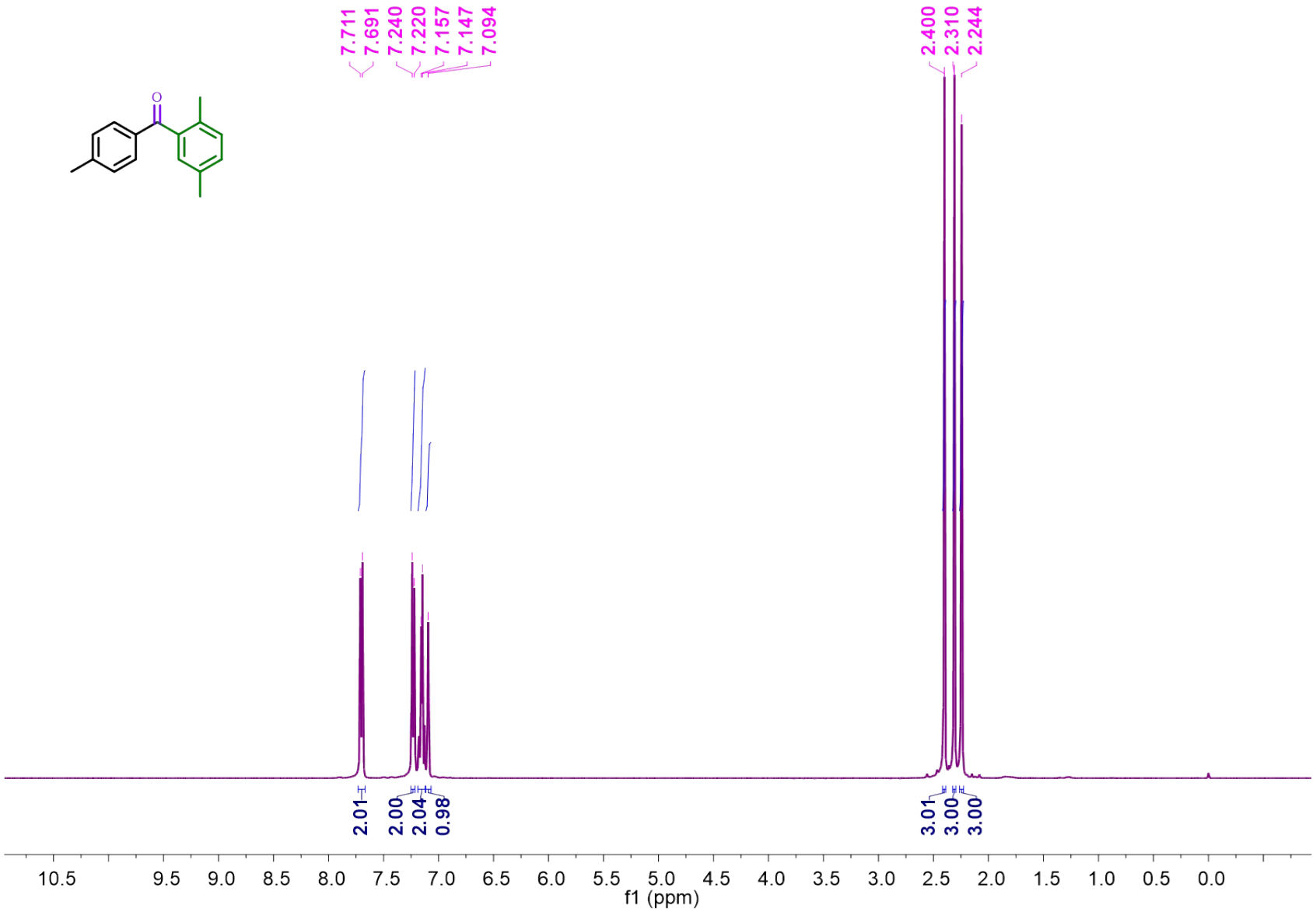


Figure S54. ^{1}H NMR spectra of (2,5-dimethylphenyl) (p-tolyl) methanone **(2k)** in $CDCl_3$.

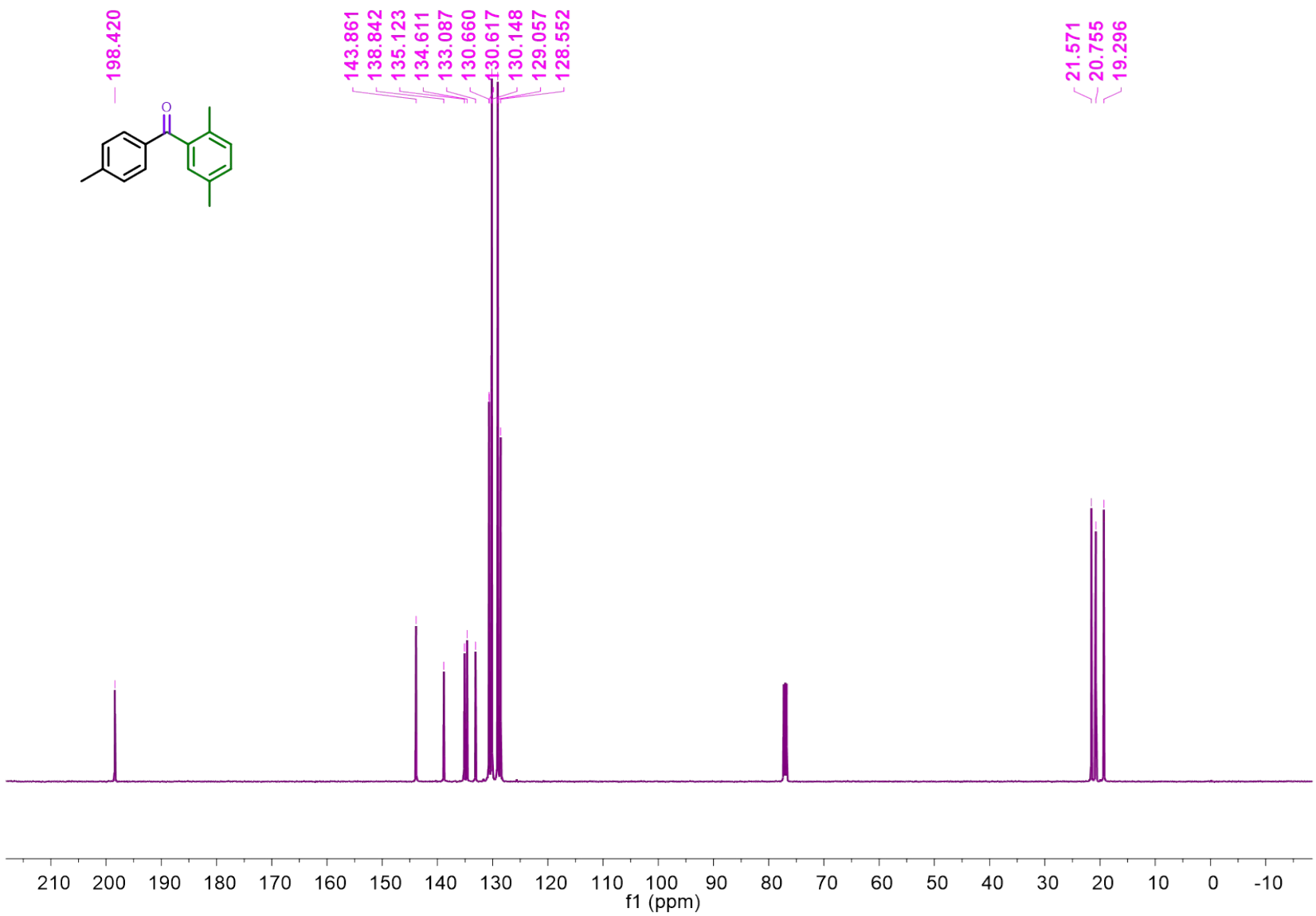


Figure S55. ^{13}C NMR spectra of (2,5-dimethylphenyl)(p-tolyl)methanone **(2k)** in $CDCl_3$.

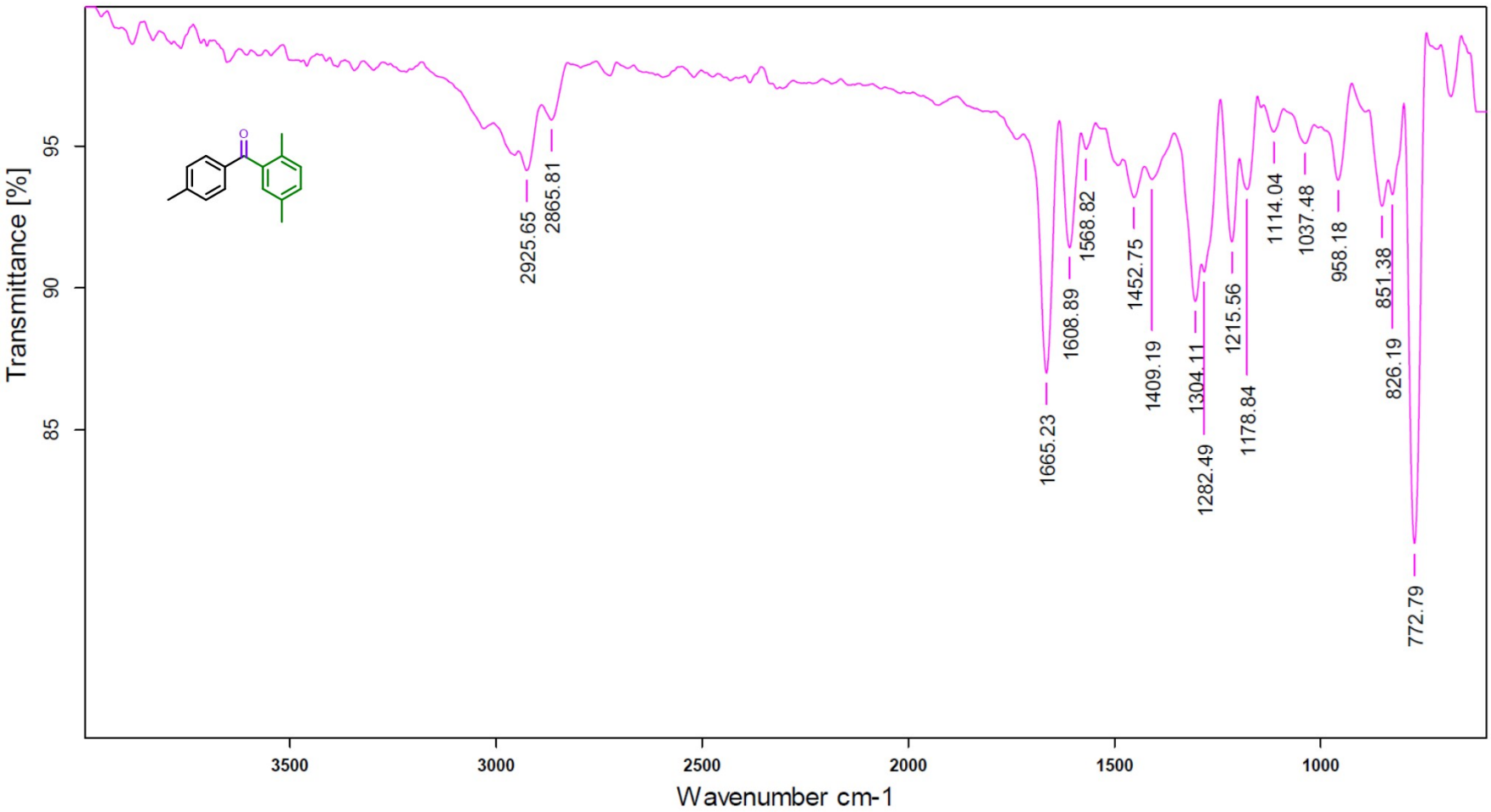


Figure S56. IR spectra of (2,5-dimethylphenyl) (p-tolyl) methanone **(2k)**.

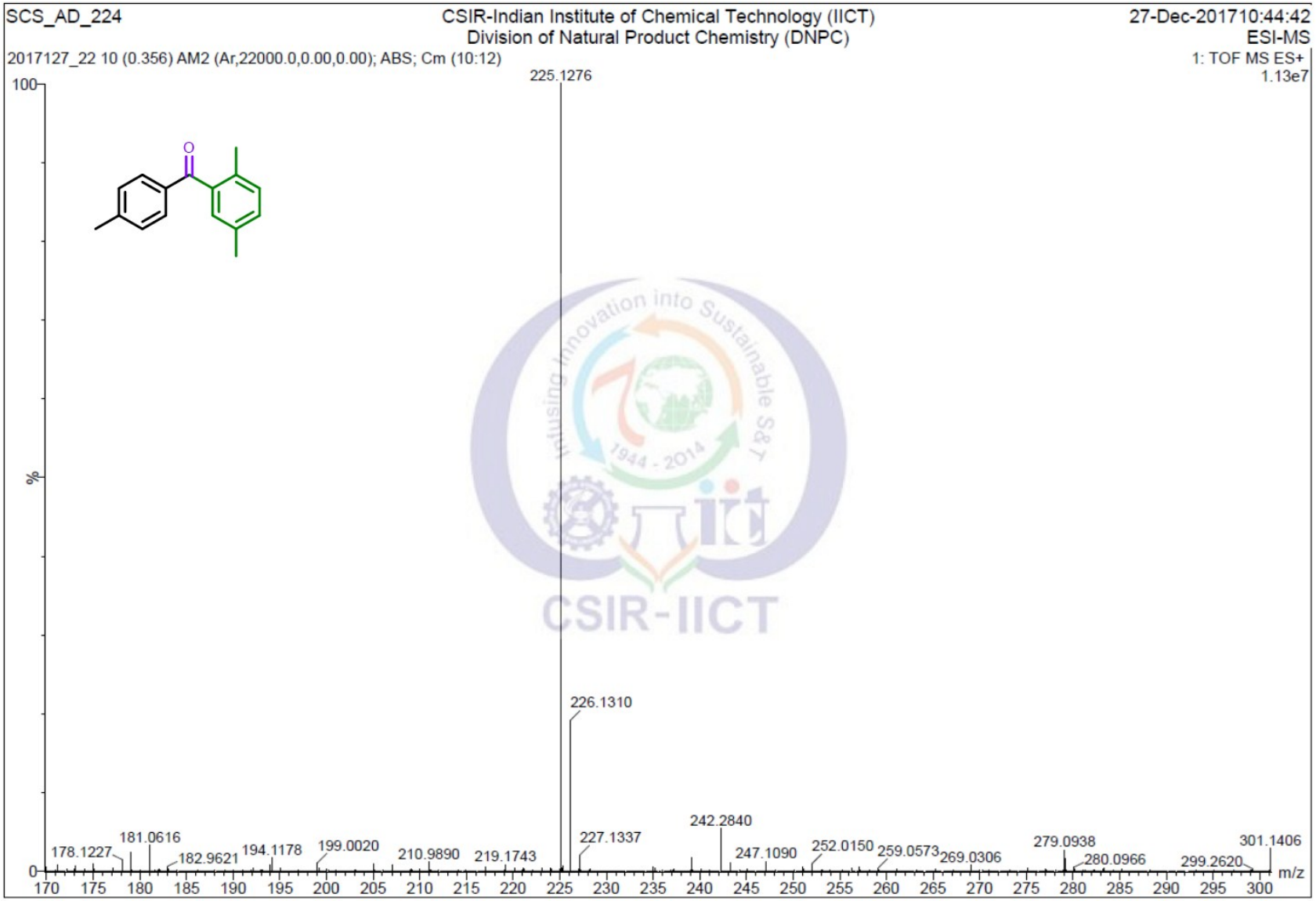


Figure S57. HRMS spectra of (2,5-dimethylphenyl) (p-tolyl) methanone **(2k)**.

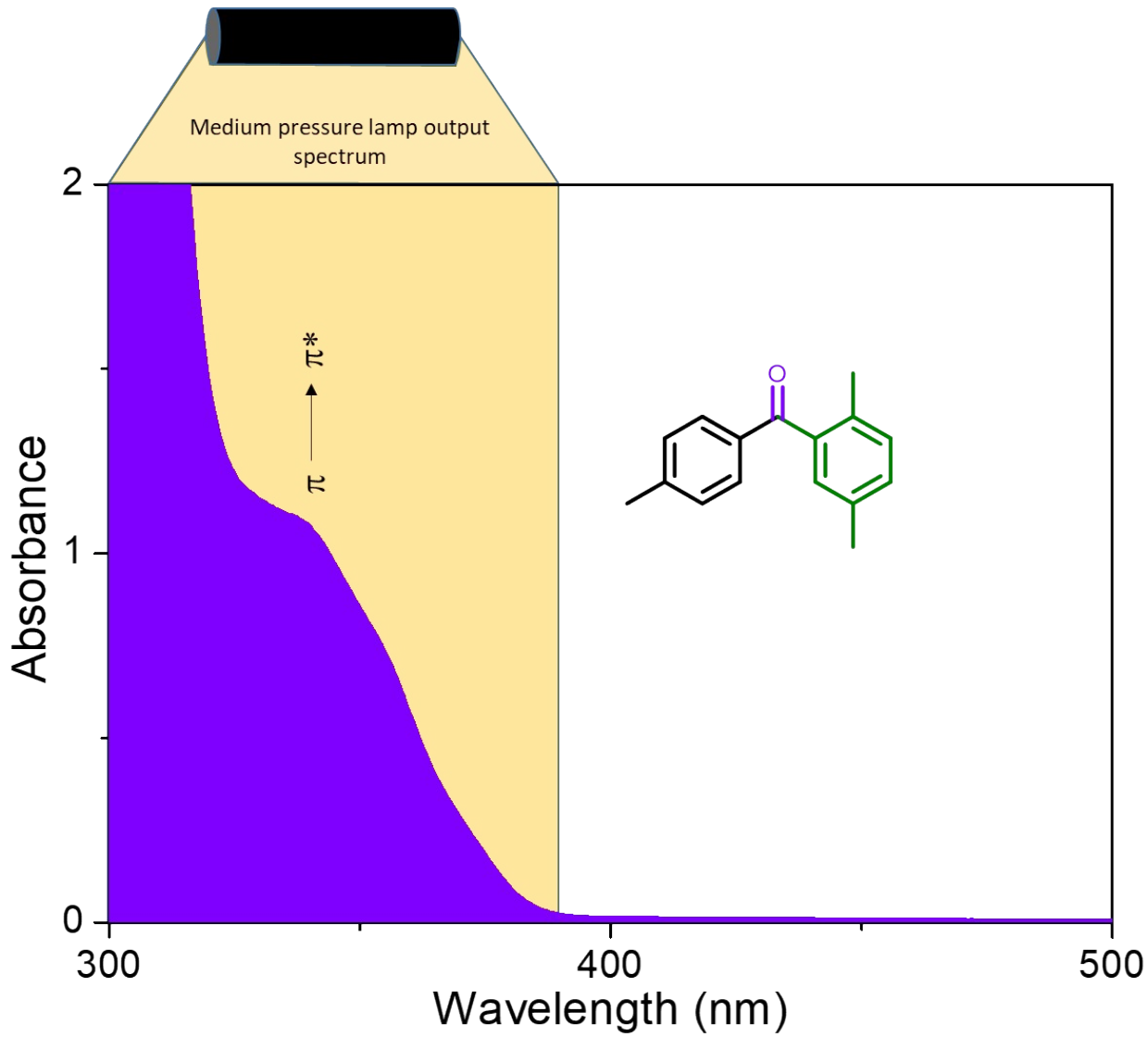


Figure S58. UV-Vis spectra of (2,5-dimethylphenyl) (p-tolyl) methanone (**2k**) in 0.005M ACN.

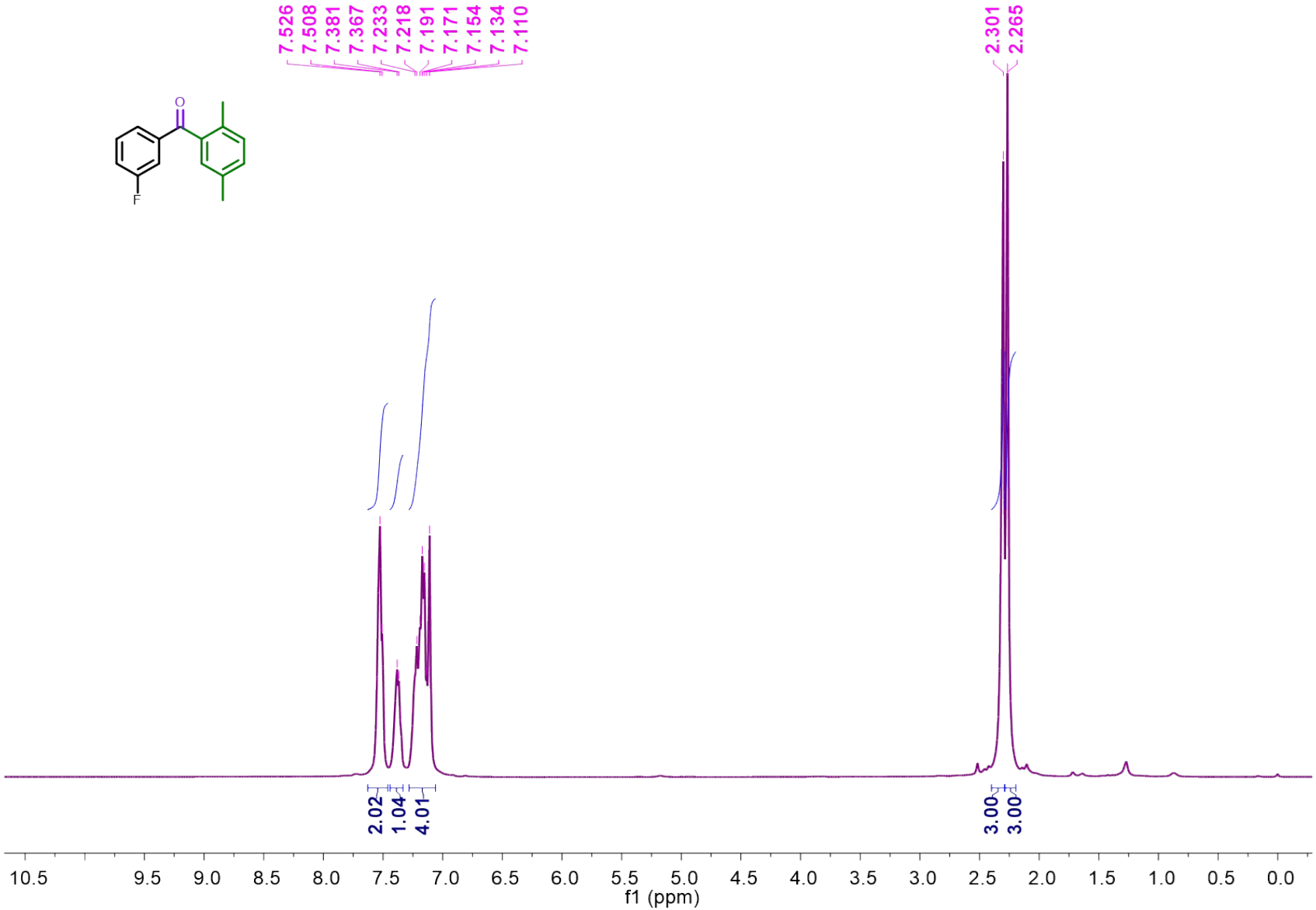


Figure S59. ^{1}H NMR spectra of (2, 5-dimethylphenyl) (3-fluorophenyl) methanone **(2l)** in $CDCl_3$.

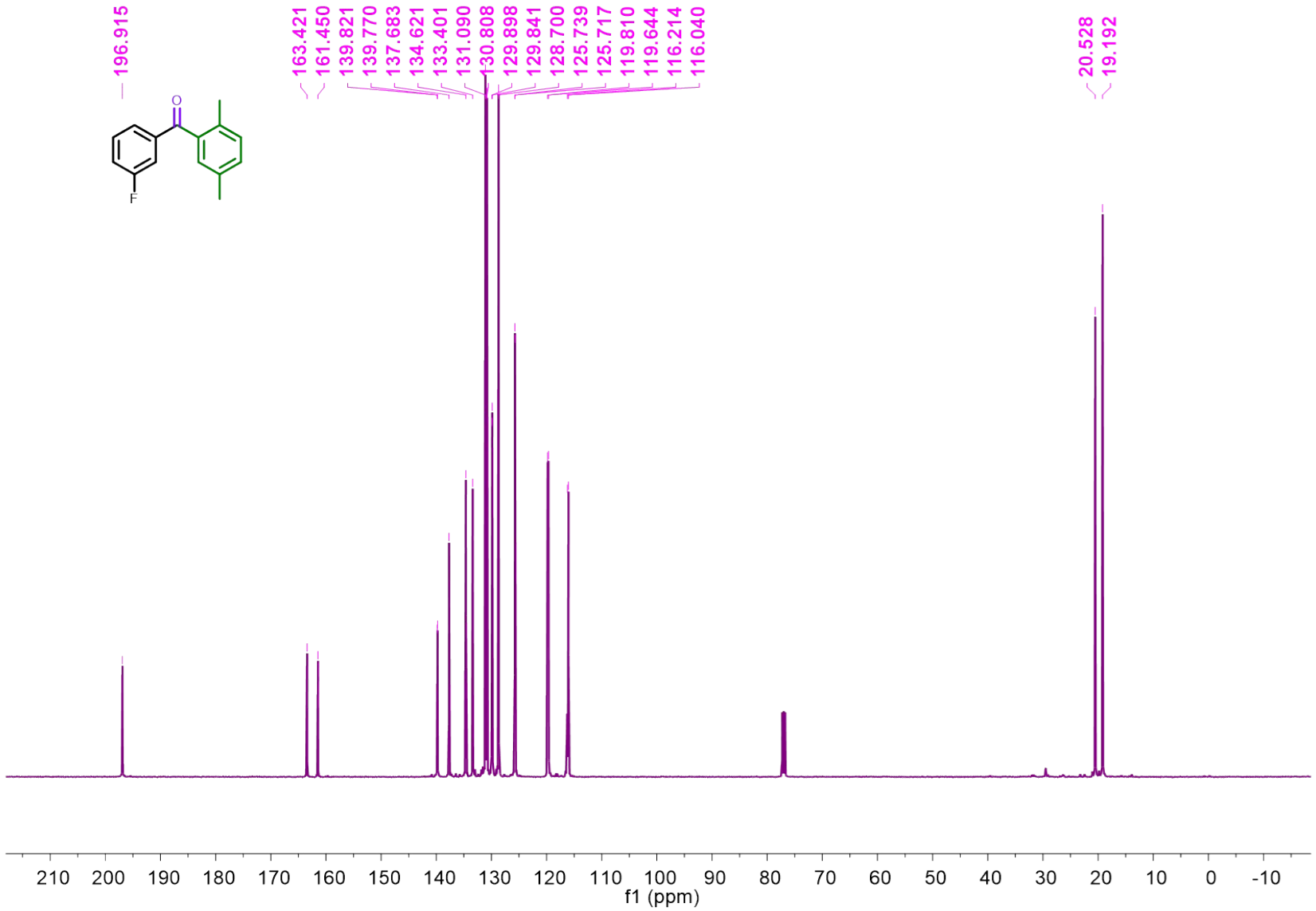


Figure S60. ^{13}C NMR spectra of (2, 5-dimethylphenyl) (3-fluorophenyl) methanone **(2l)** in $CDCl_3$.

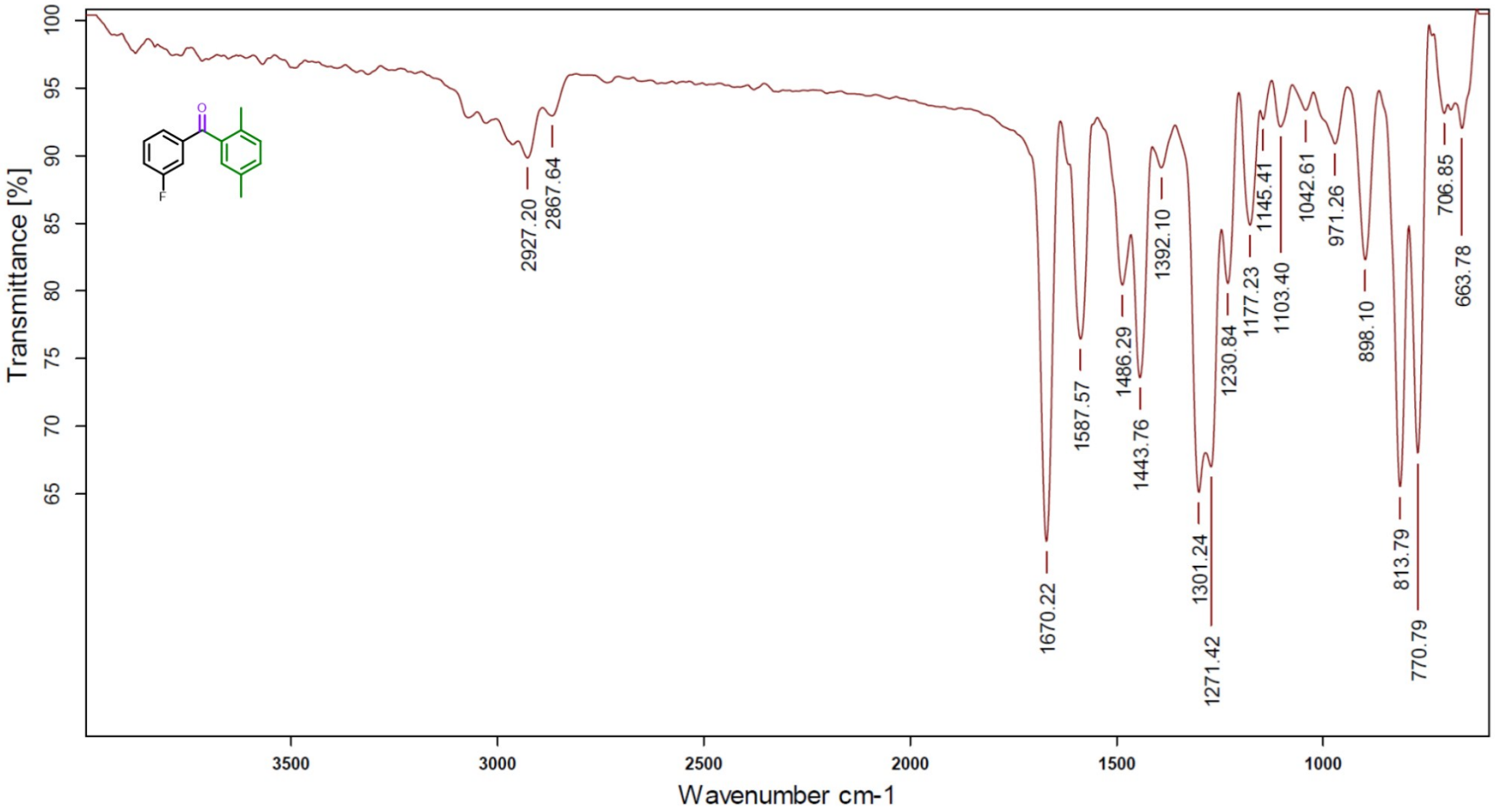


Figure S61. IR spectra of (2, 5-dimethylphenyl) (3-fluorophenyl) methanone **(2l)**.

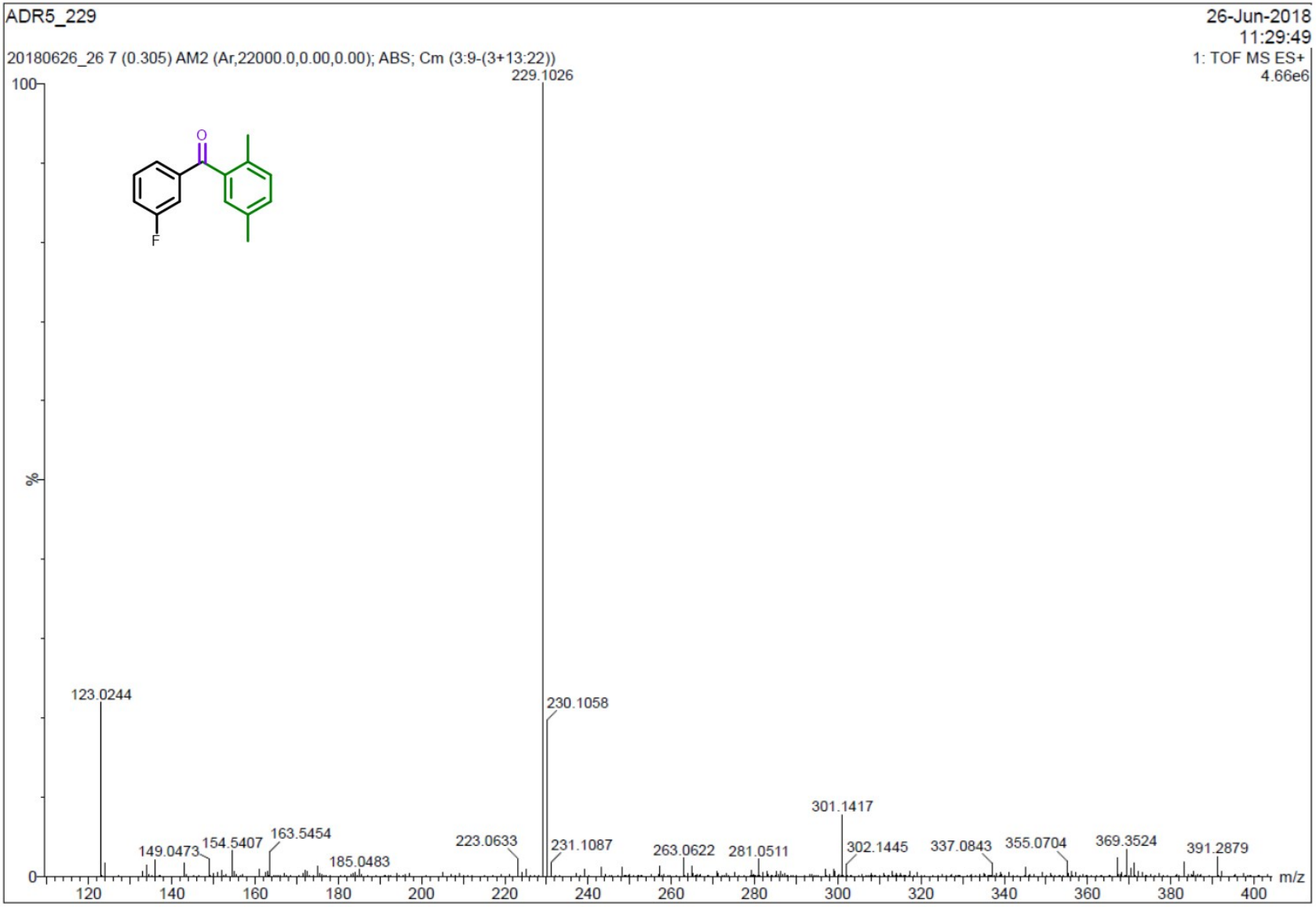


Figure S62. HRMS spectra of (2, 5-dimethylphenyl) (3-fluorophenyl) methanone **(2l)**.

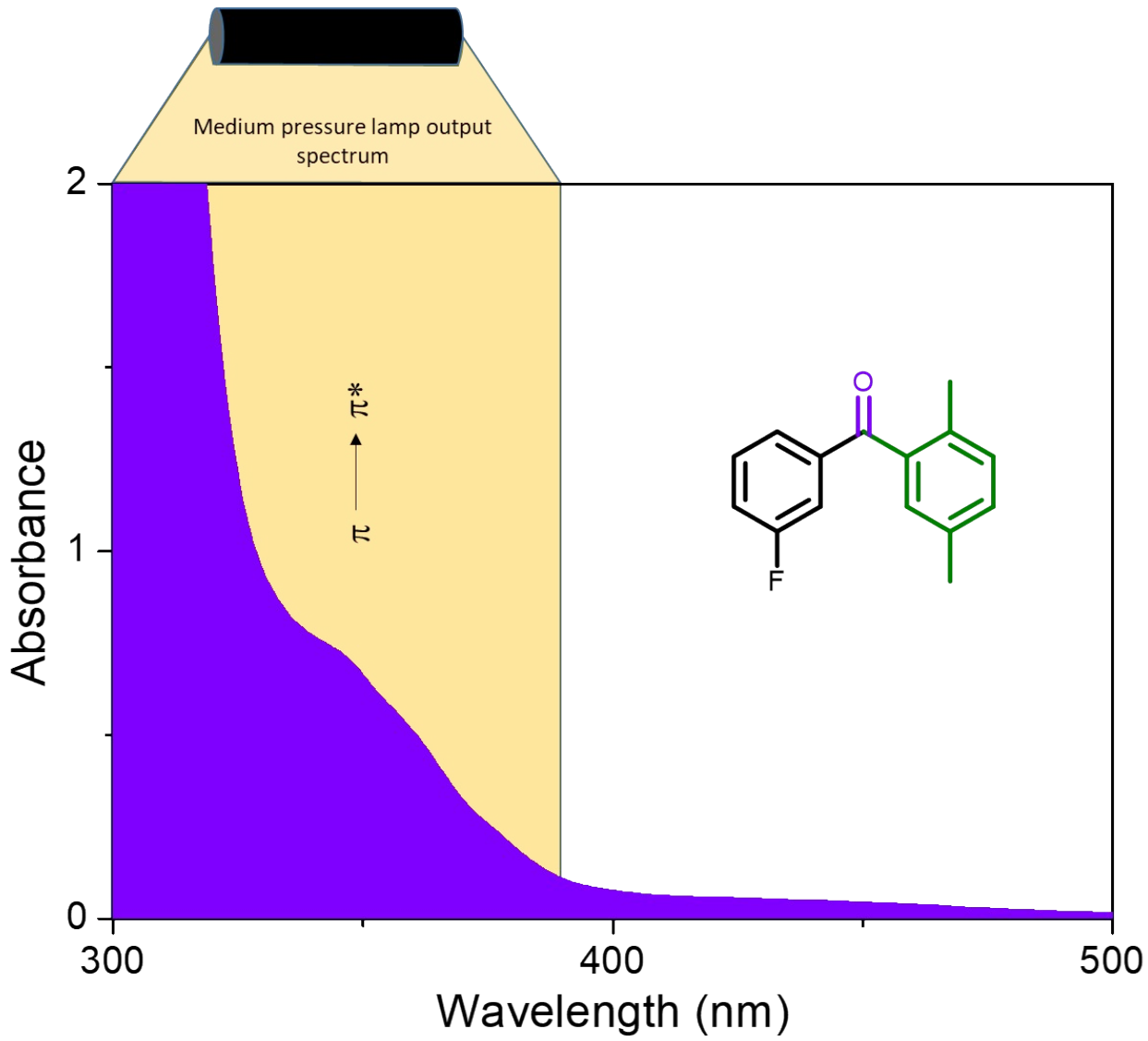


Figure S63. UV-Vis spectra of (2, 5-dimethylphenyl) (3-fluorophenyl) methanone **(2l**) in 0.005M ACN.

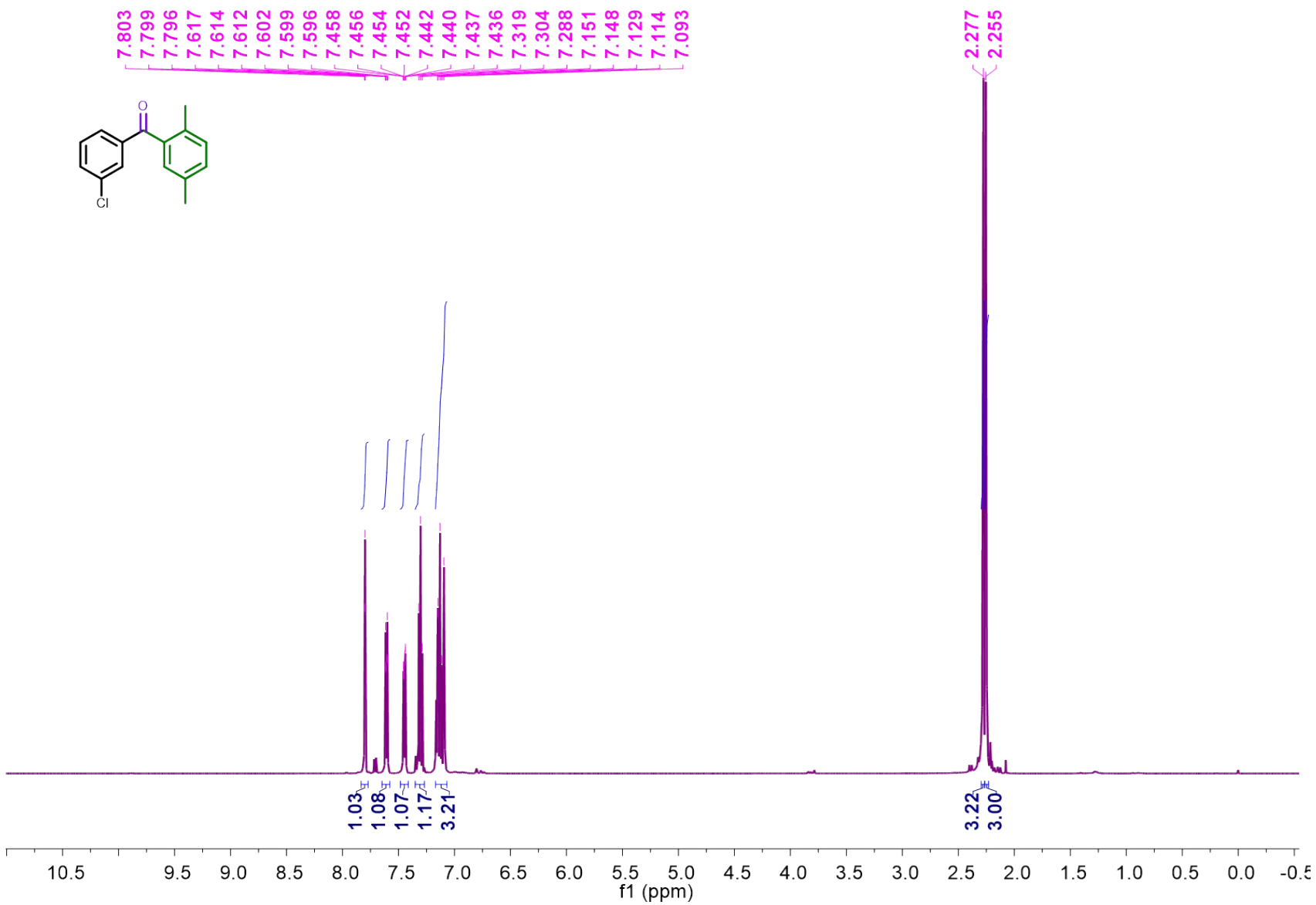


Figure S64. ^{1}H NMR spectra of (2, 5-dimethylphenyl) (3-chlorophenyl) methanone **(2m)** in $CDCl_3$.

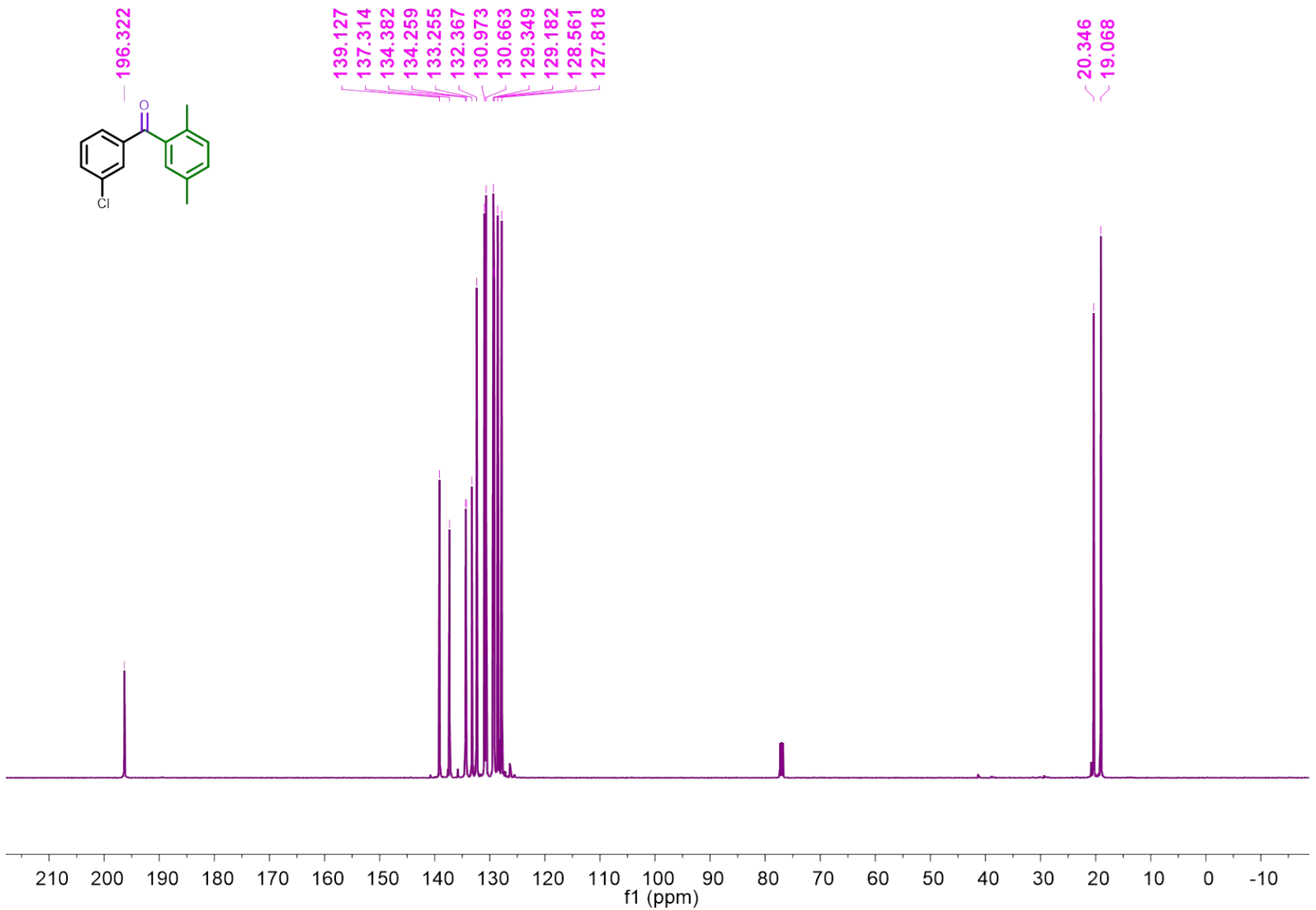


Figure S65. ^{13}C NMR spectra of (2, 5-dimethylphenyl) (3-chlorophenyl) methanone **(2m)** in $CDCl_3$.

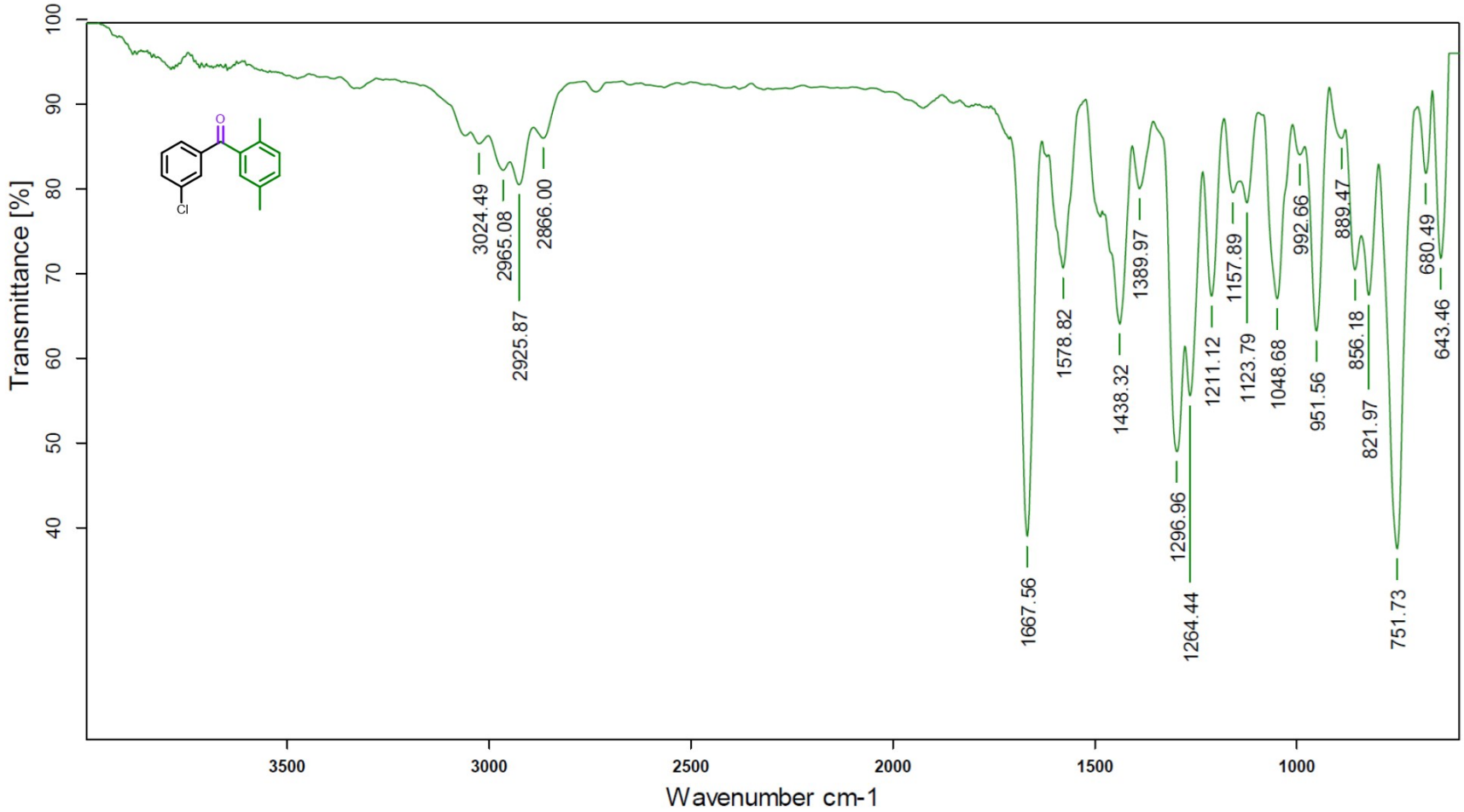


Figure S66. IR spectra of (2, 5-dimethylphenyl) (3-chlorophenyl) methanone **(2m**).

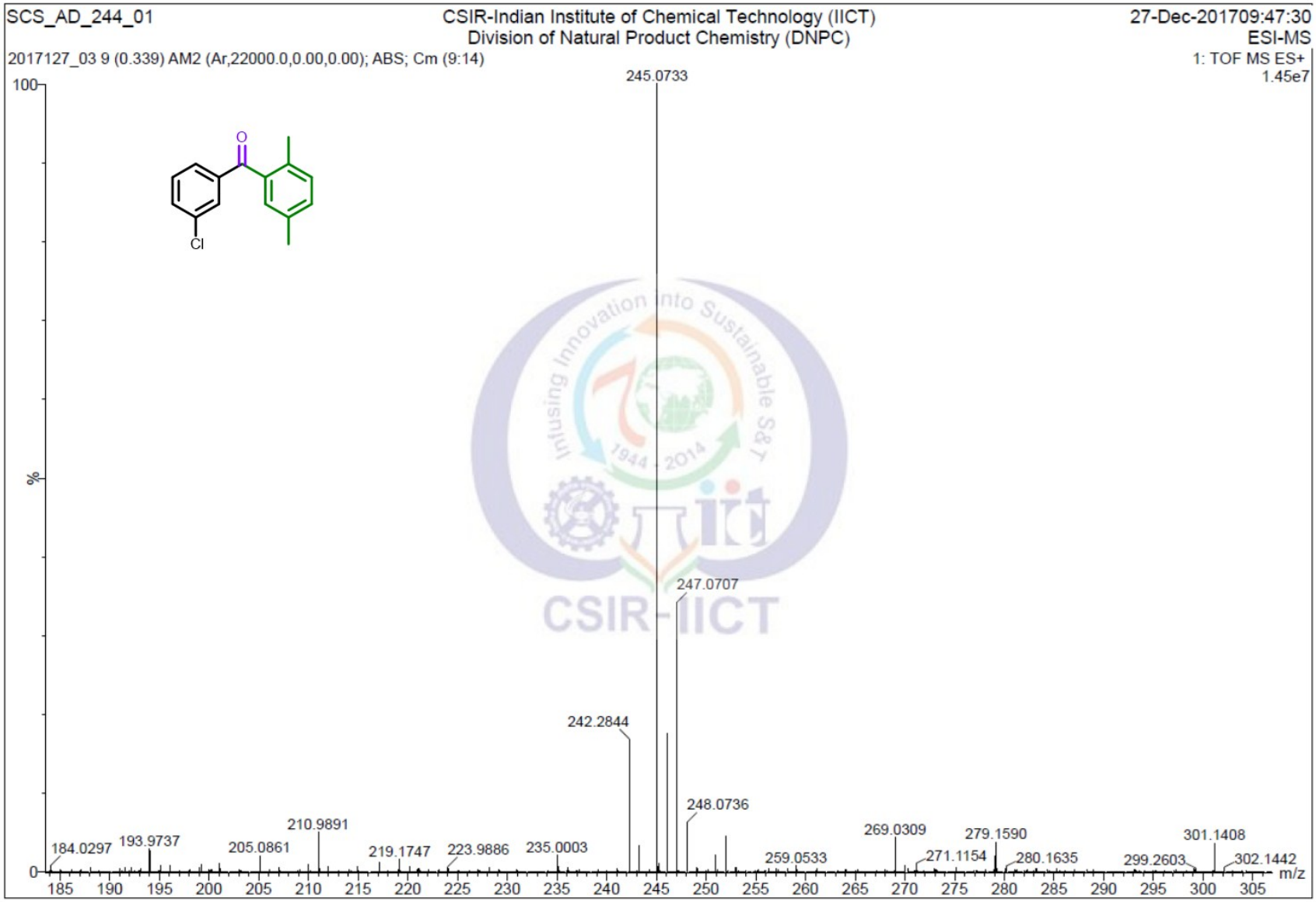


Figure S67. HRMS spectra of (2, 5-dimethylphenyl) (3-chlorophenyl) methanone **(2m)**.

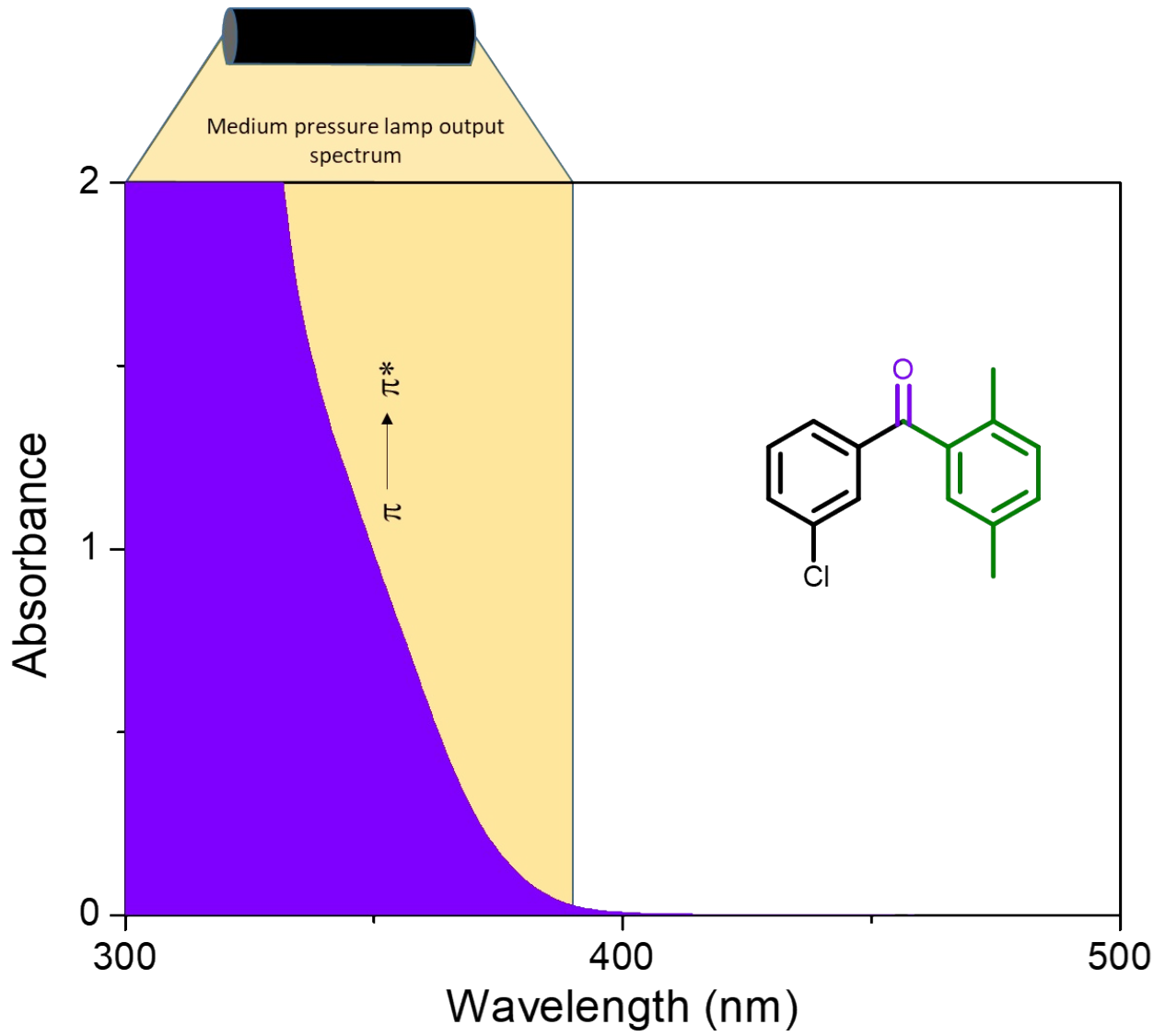


Figure S68. UV-Vis spectra of (2, 5-dimethylphenyl) (3-chlorophenyl) methanone **(2m**) in 0.005M ACN.

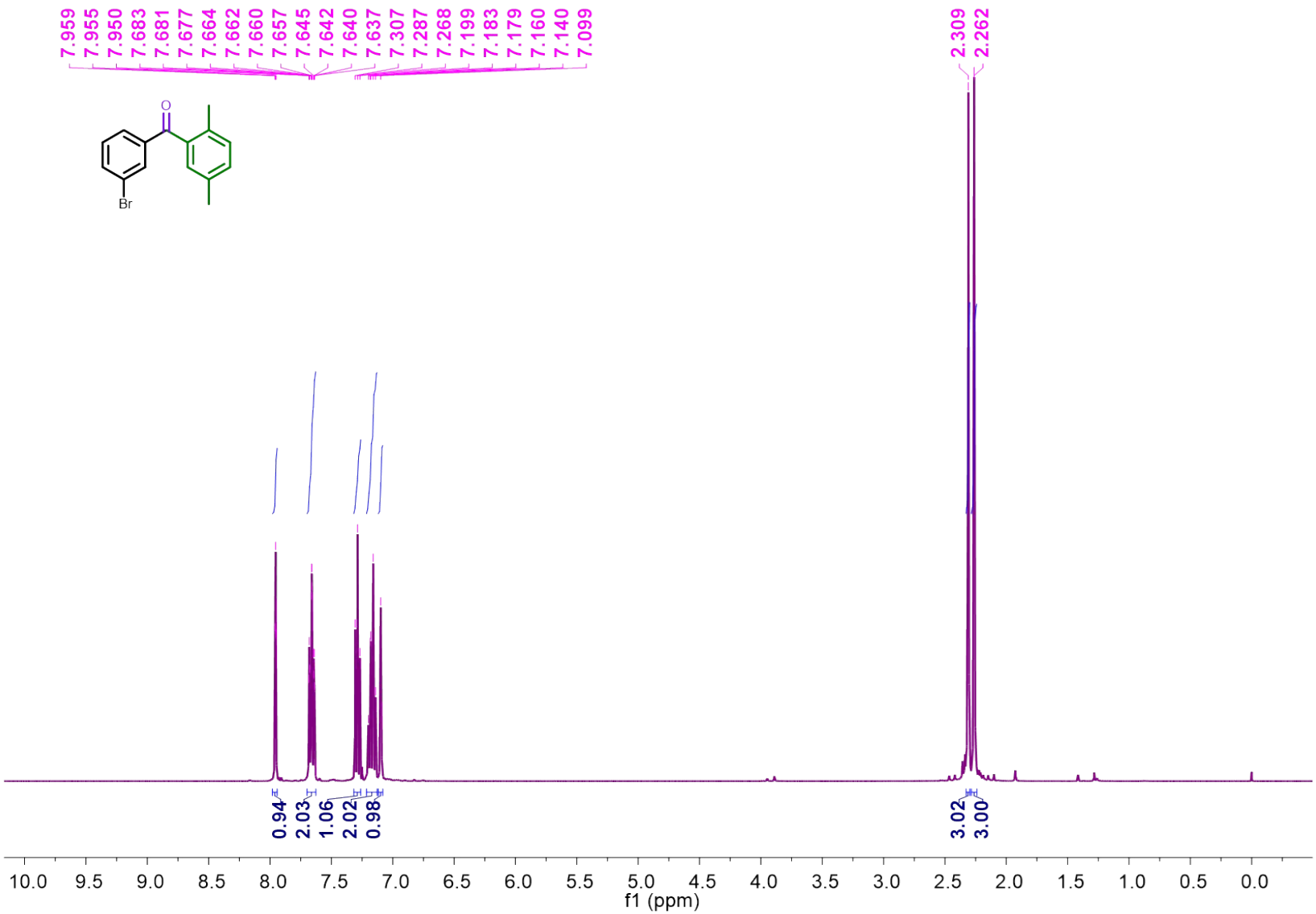


Figure S69. ^{1}H NMR spectra of (2, 5-dimethylphenyl) (3-bromophenyl) methanone **(2n)** in $CDCl_3$.

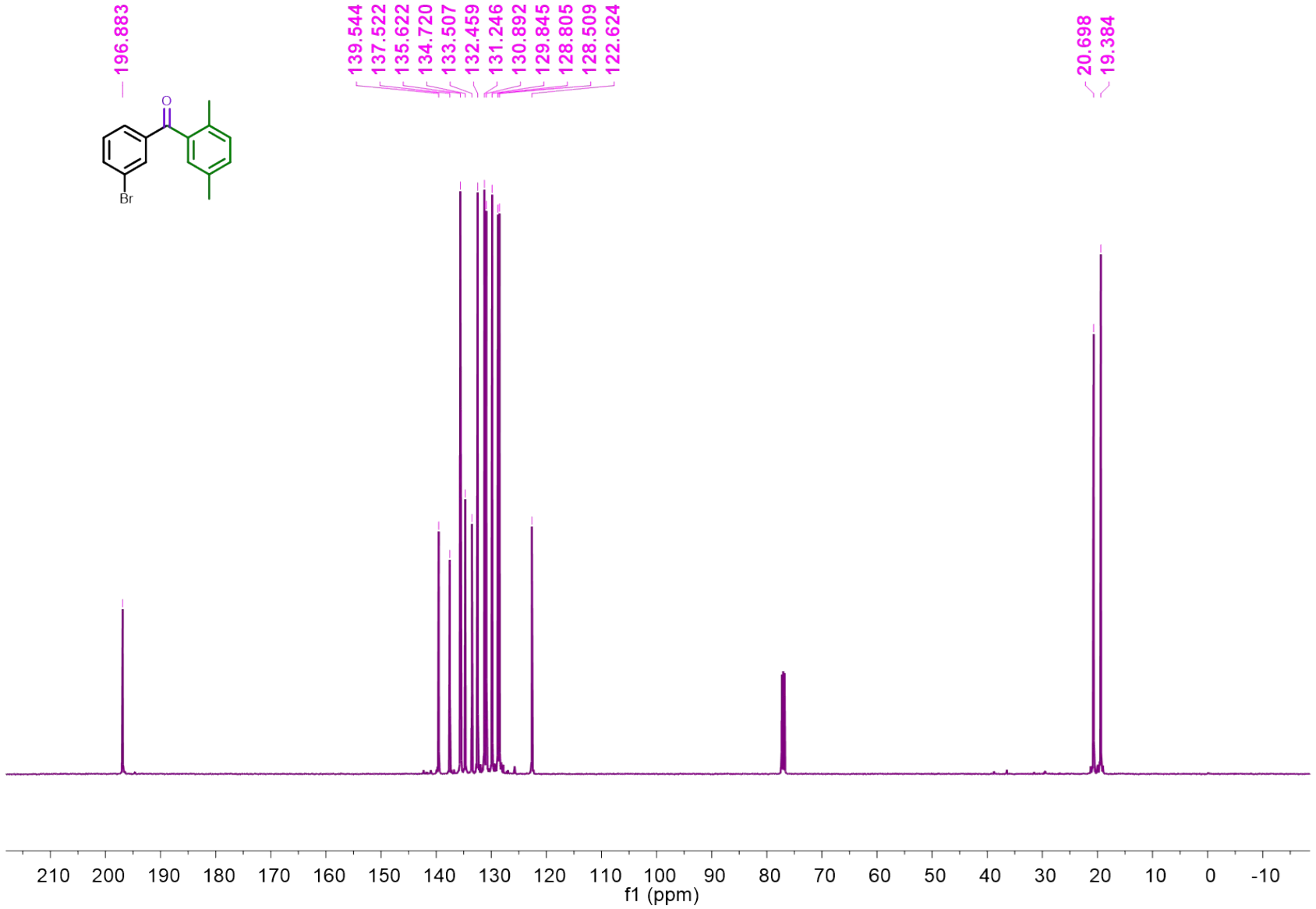


Figure S70. ^{13}C NMR spectra of (2, 5-dimethylphenyl) (3-bromophenyl) methanone **(2n)** in $CDCl_3$.

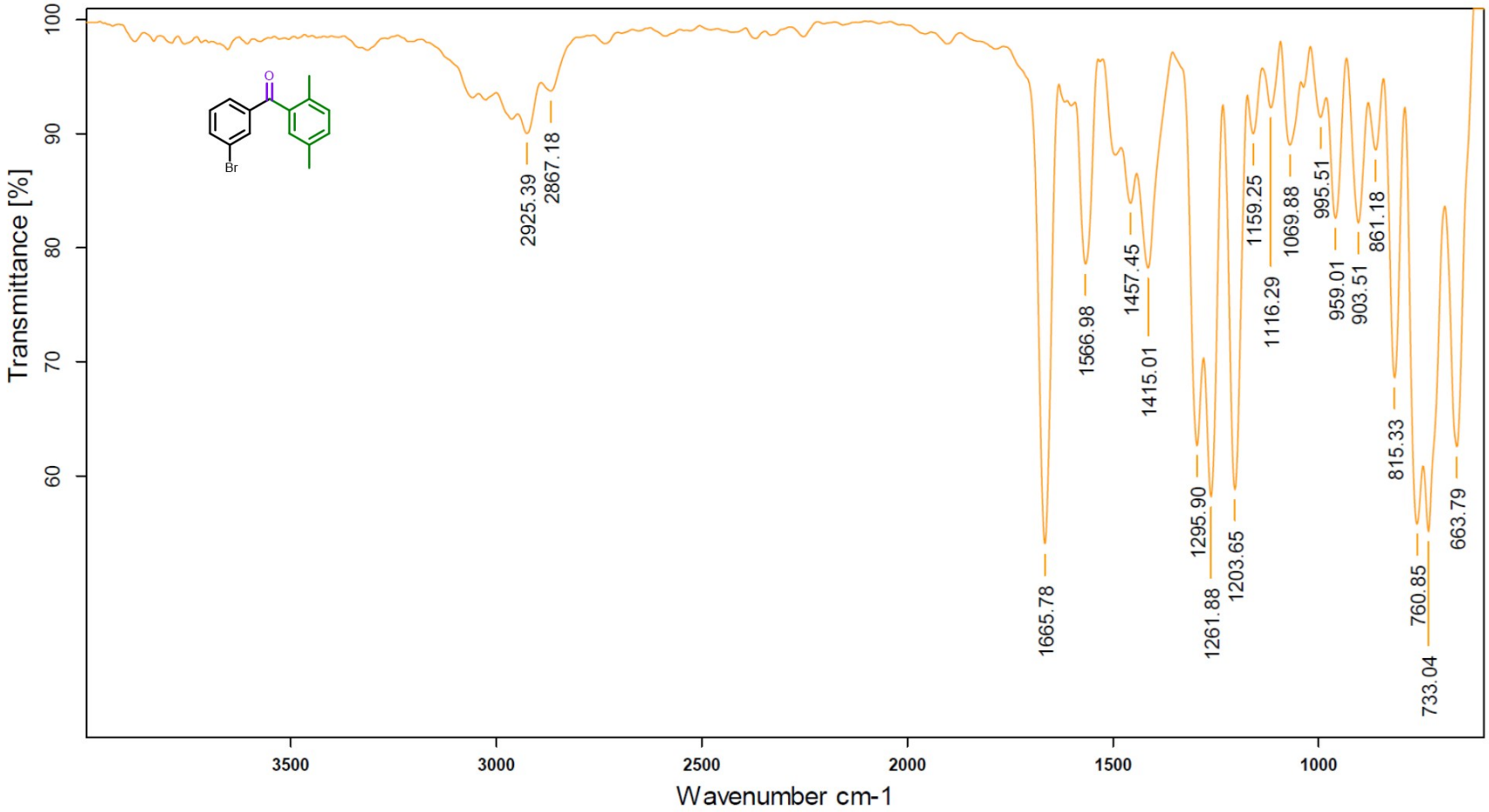


Figure S71. IR spectra of (2, 5-dimethylphenyl) (3-bromophenyl) methanone **(2n)**.

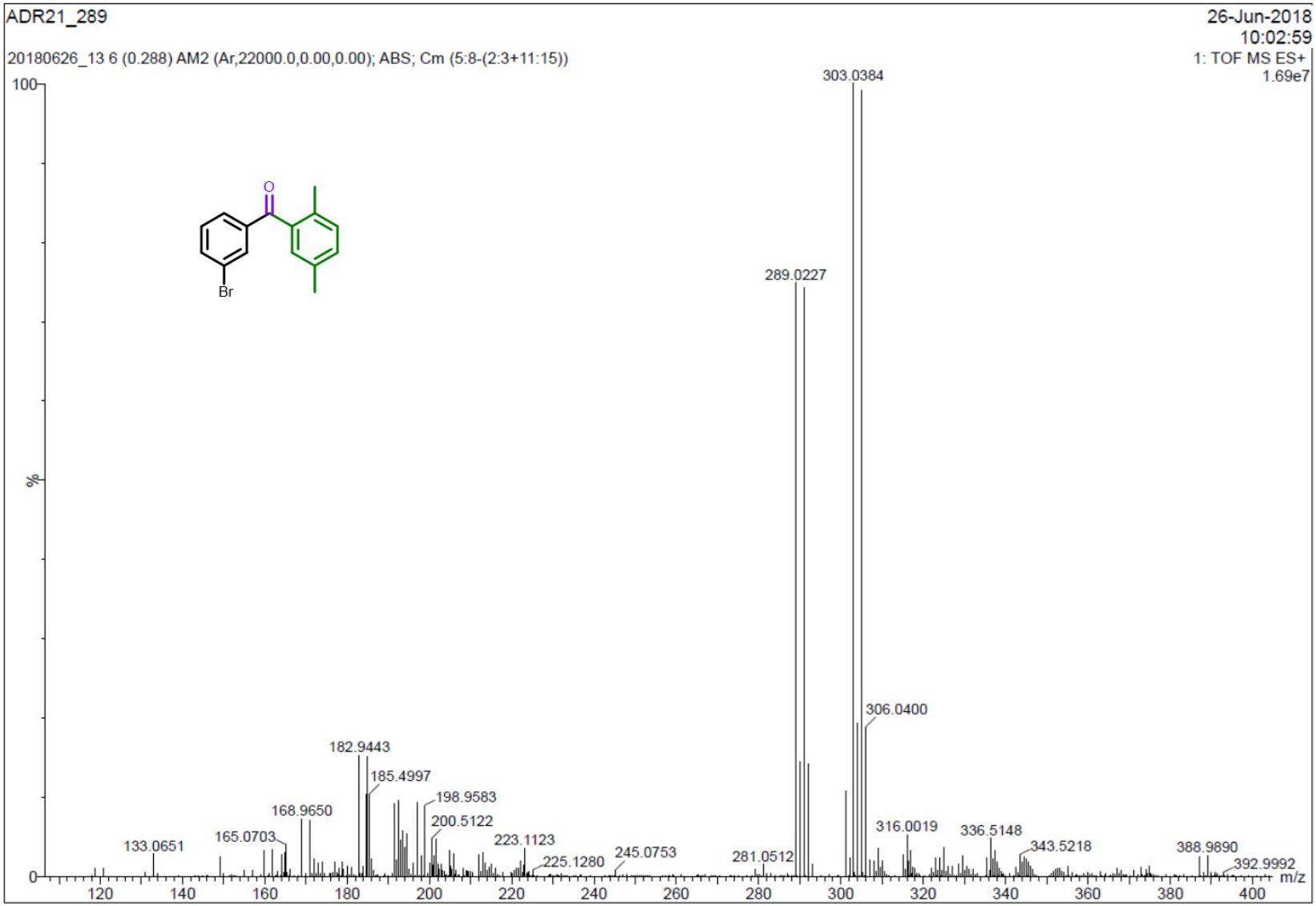


Figure S72. HRMS spectra of (2, 5-dimethylphenyl) (3-bromophenyl) methanone **(2n)**.

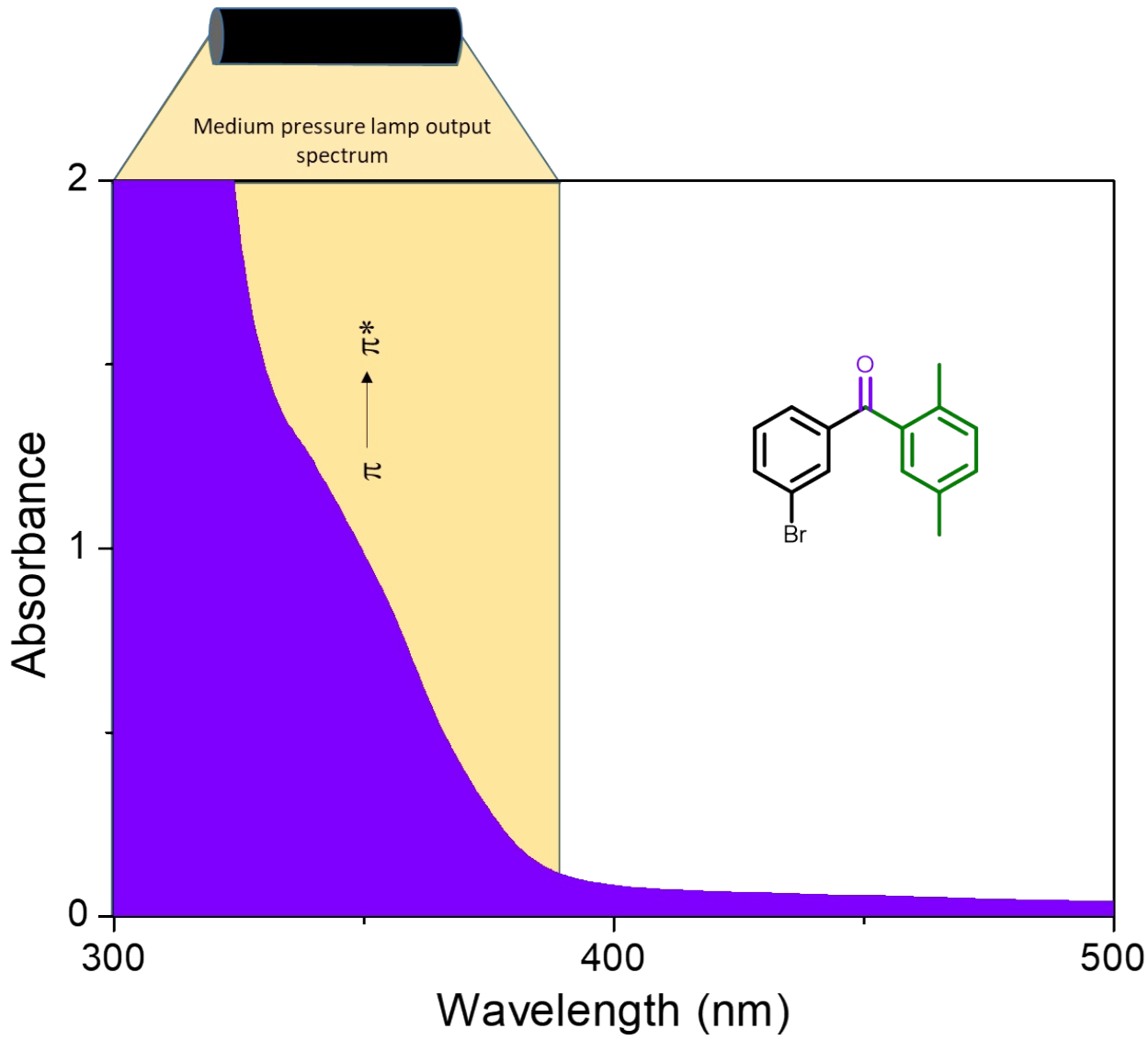


Figure S73. UV-Vis spectra of (2, 5-dimethylphenyl) (3-bromophenyl) methanone **(2n**) in 0.005M ACN.

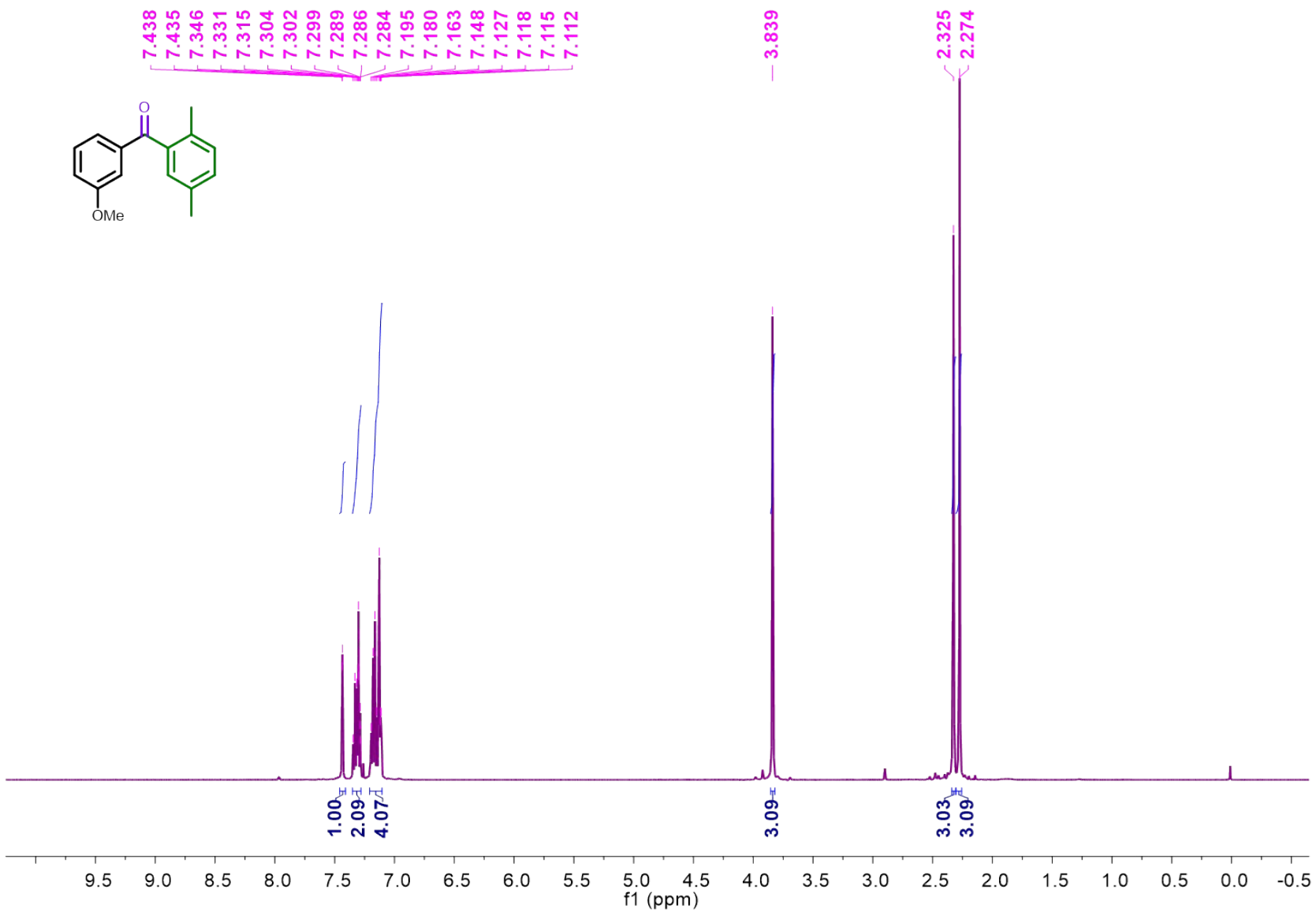


Figure S74. ^{1}H NMR spectra of (2, 5-dimethylphenyl) (3-methoxyphenyl) methanone **(2o)** in $CDCl_3$.

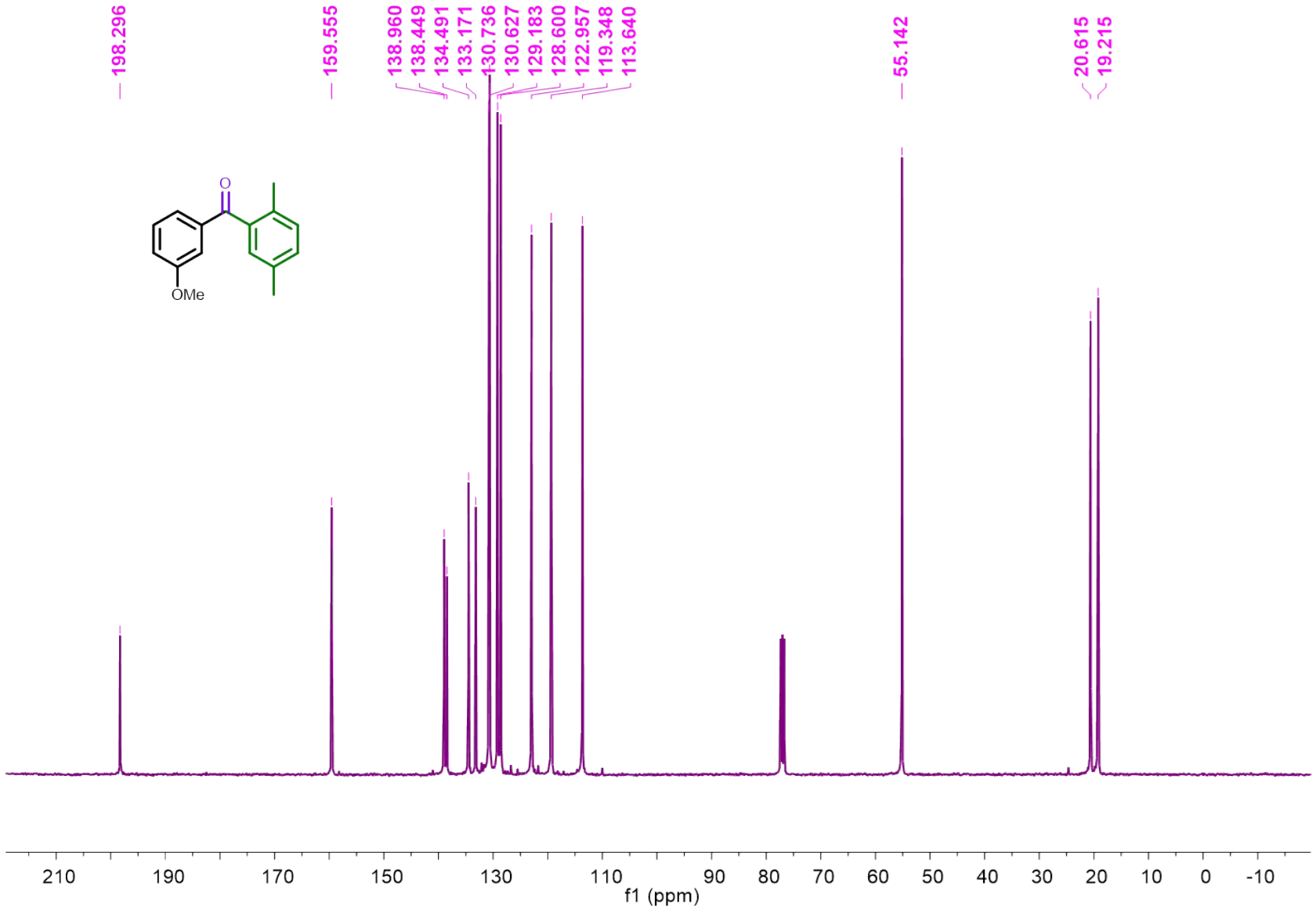


Figure S75. ^{13}C NMR spectra of (2, 5-dimethylphenyl) (3-methoxyphenyl) methanone **(2o)** in $CDCl_3$.

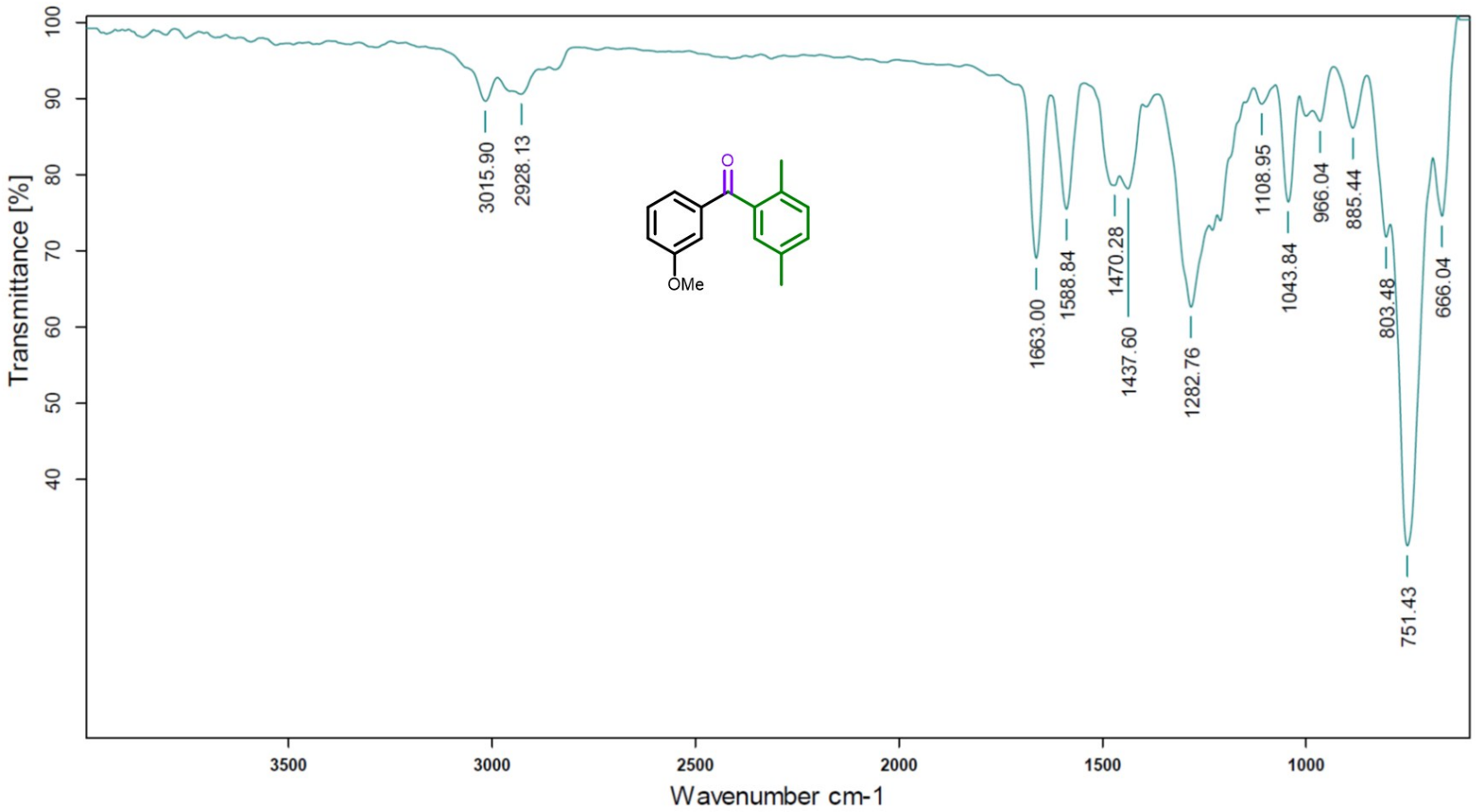
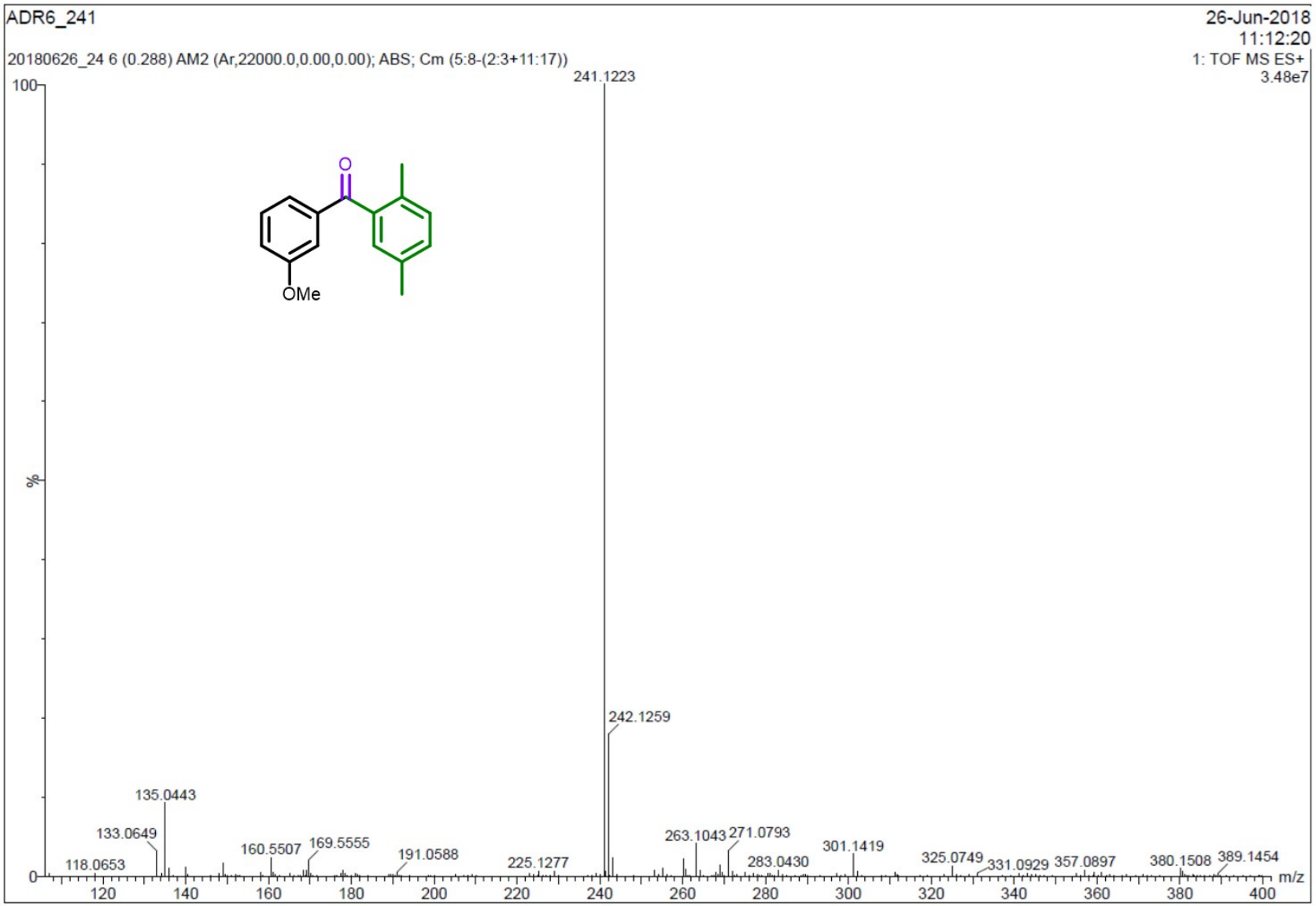


Figure S76. IR spectra of (2, 5-dimethylphenyl) (3-methoxyphenyl) methanone **(2o**).


ADR6_241
26-Jun-2018
11:12:20
20180626_24 6 (0.288) AM2 (Ar,22000.0,0.00,0.00); ABS; Cm (5:8-(2:3+11:17))
1: TOF MS ES+
3.48e7
100
%
0
241.1223
242.1259
135.0443
133.0649
118.0653
160.5507
169.5555
191.0588
225.1277
263.1043
271.0793
283.0430
301.1419
325.0749
331.0929
357.0897
380.1508
389.1454
OMe
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
m/z

Fig

ure S77. HRMS spectra of (2, 5-dimethylphenyl) (3-methoxyphenyl) methanone **(2o**).

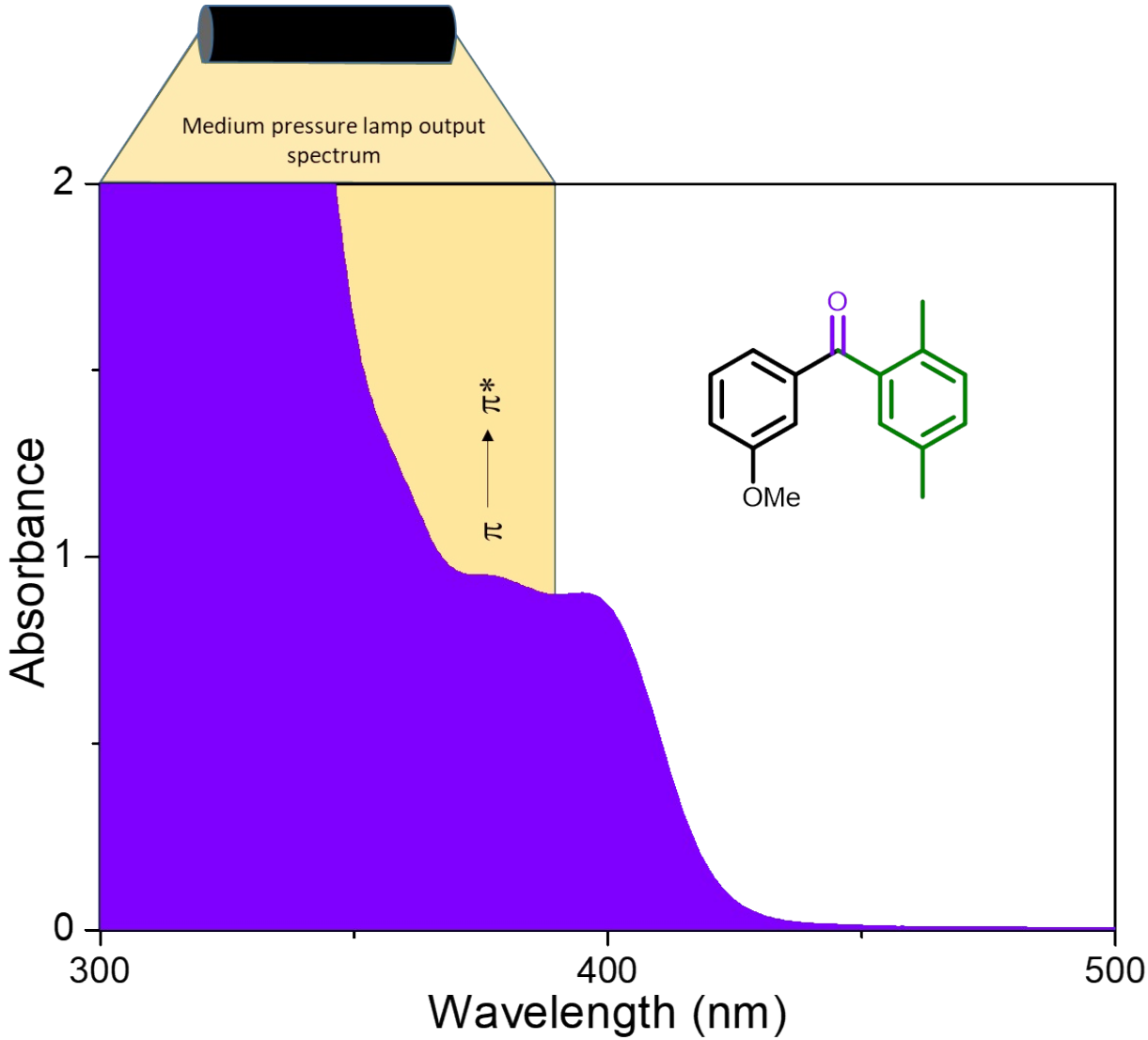


Figure S78. UV-Vis spectra of (2, 5-dimethylphenyl) (3-methoxyphenyl) methanone **(2o)** in 0.005M ACN.

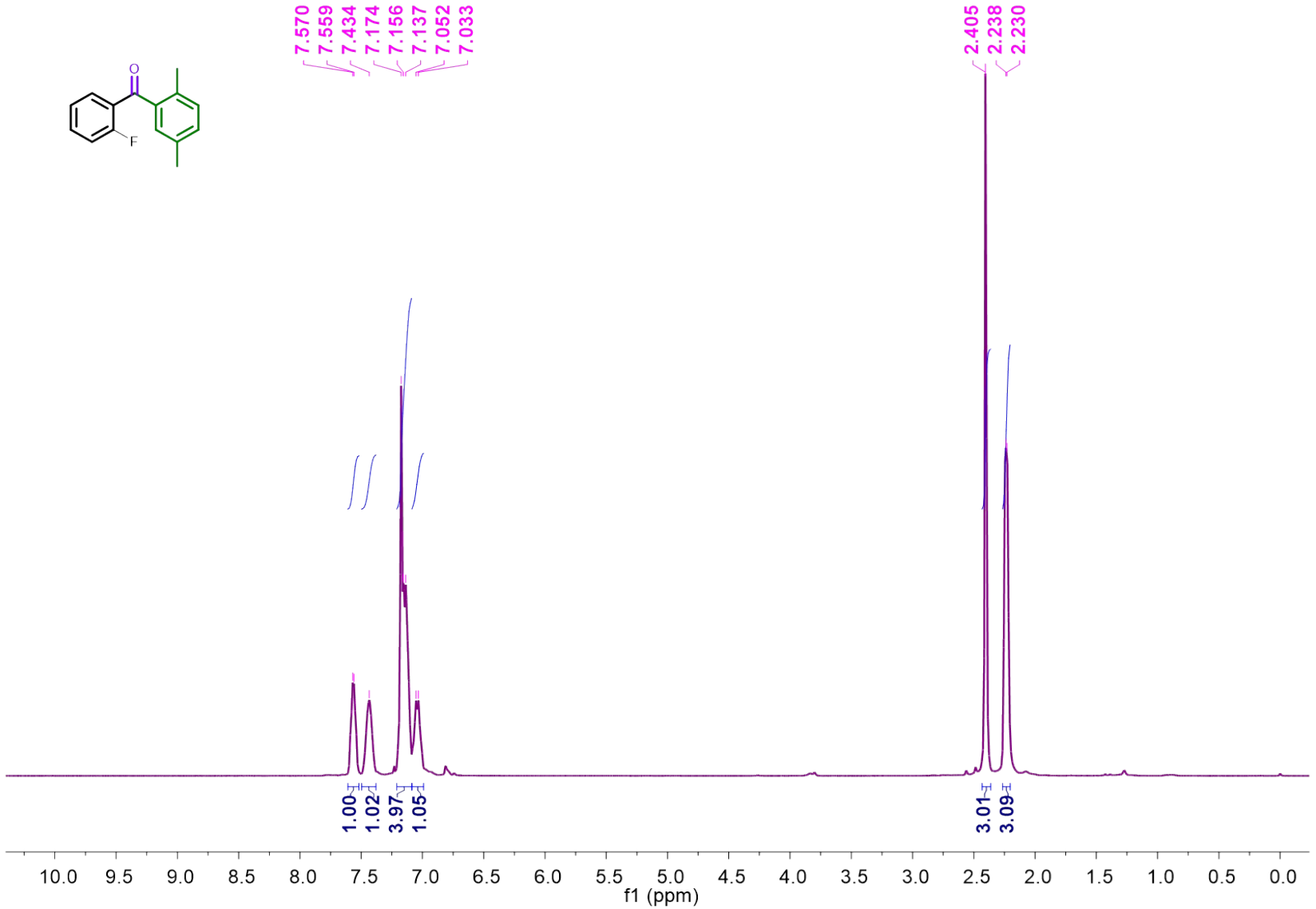


Figure S79. ^{1}H NMR spectra of (2, 5-dimethylphenyl) (2-fluorophenyl) methanone **(2p**) in $CDCl_3$.

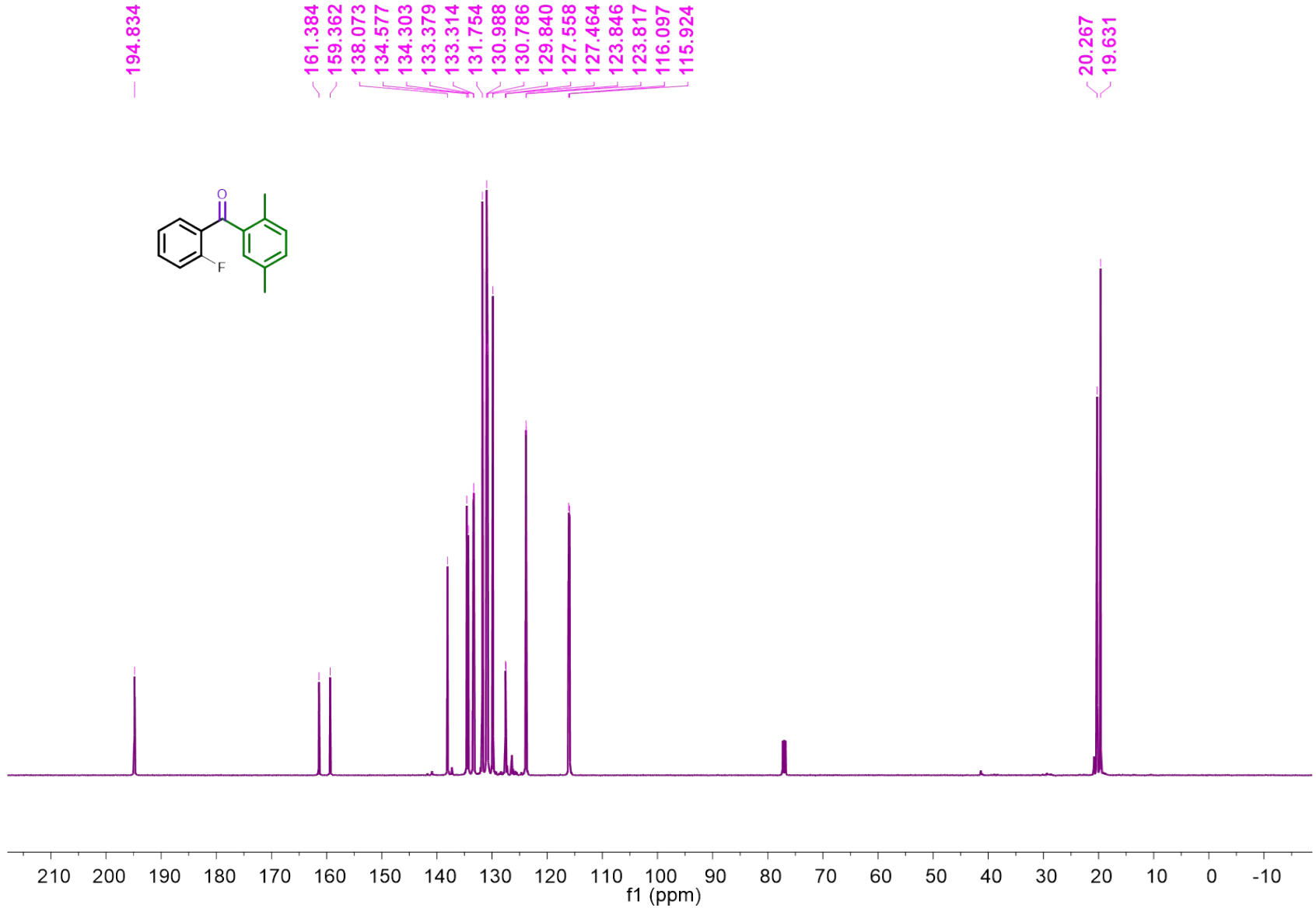


Figure S80.^{13}C NMR spectra of (2, 5-dimethylphenyl) (2-fluorophenyl) methanone **(2p)** in $CDCl_3$.

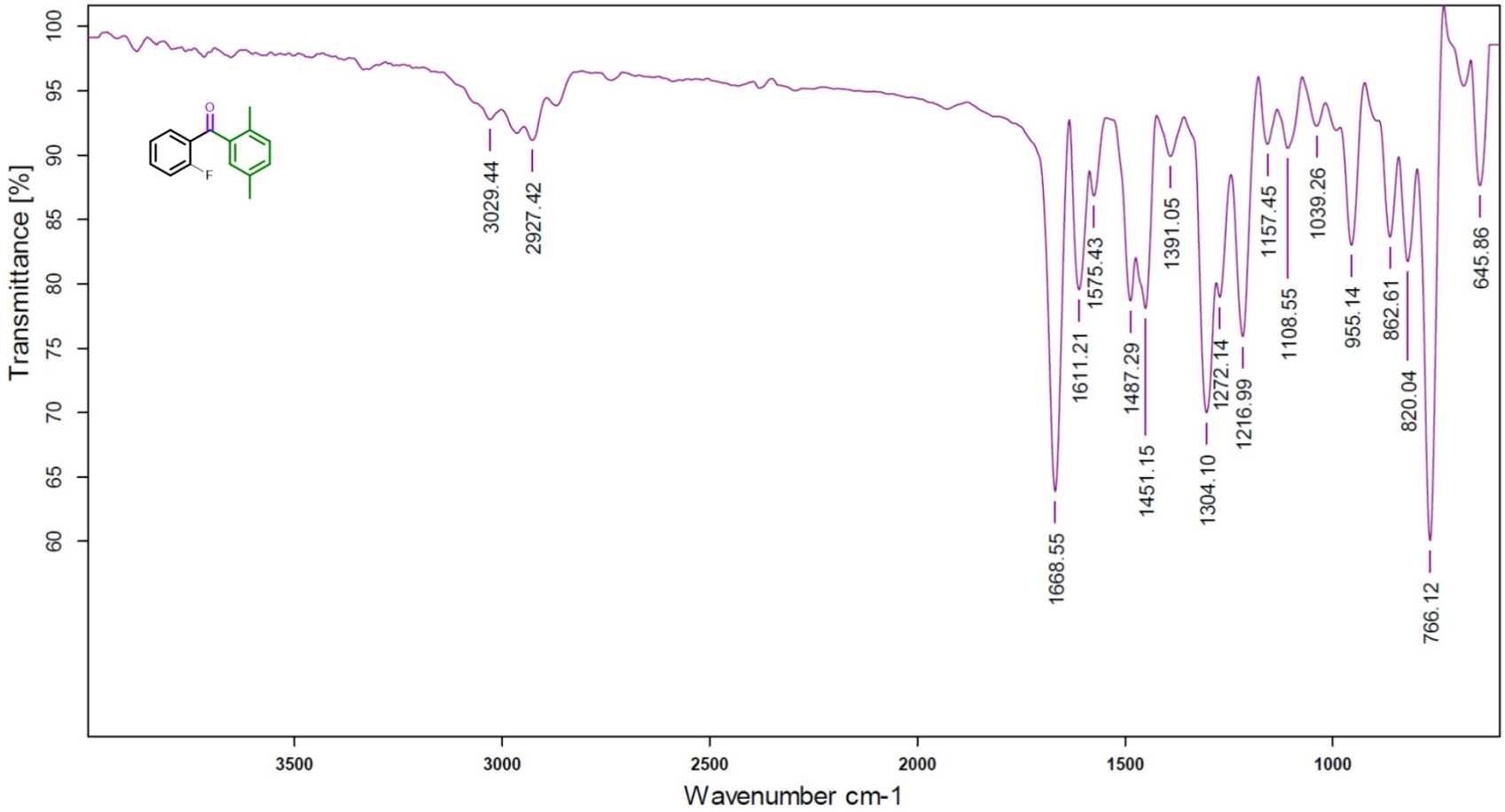


Figure S81. IR spectra of (2, 5-dimethylphenyl) (2-fluorophenyl) methanone **(2p)**.

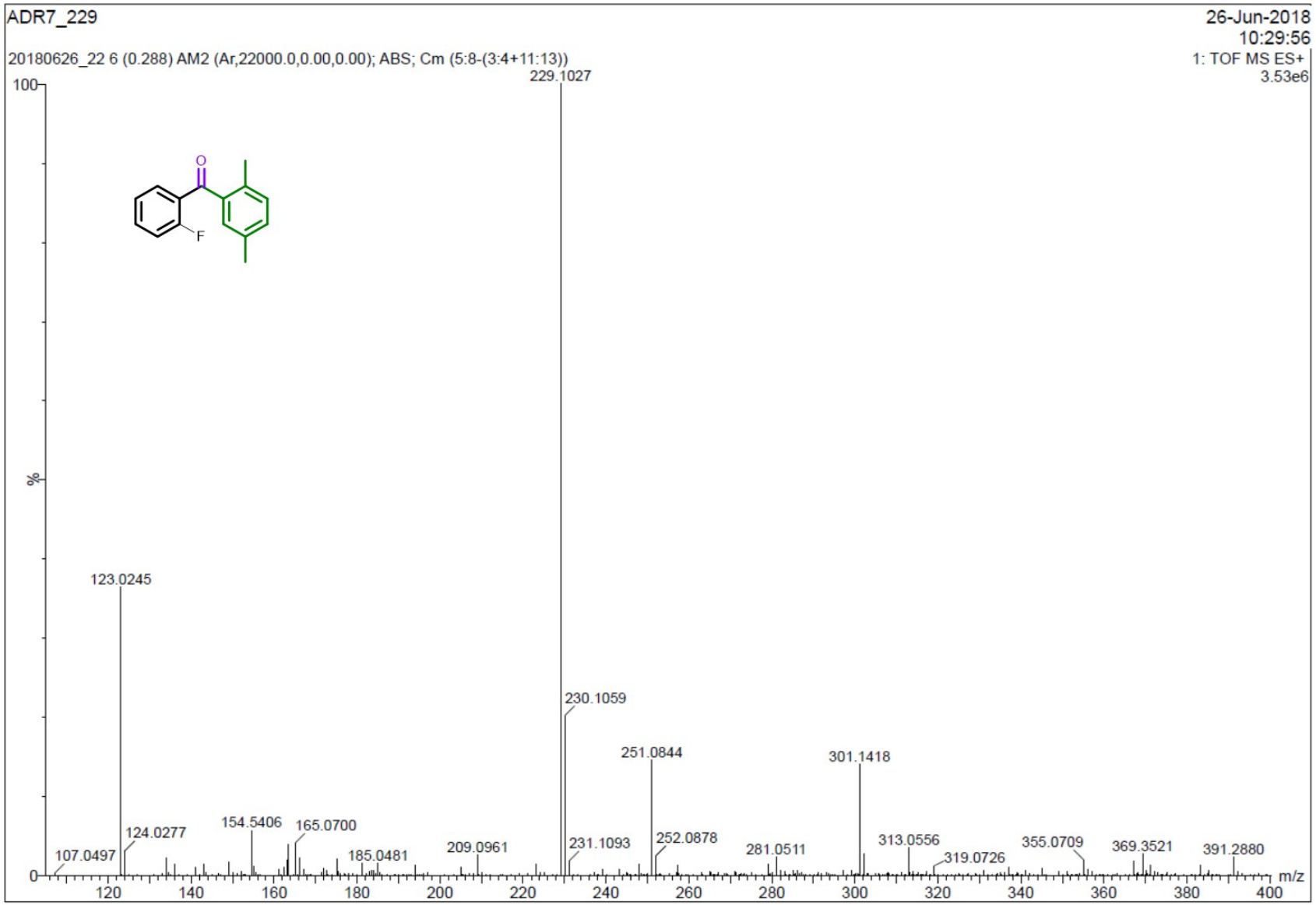


Figure S82. HRMS spectra of (2, 5-dimethylphenyl) (2-fluorophenyl) methanone **(2p)**.

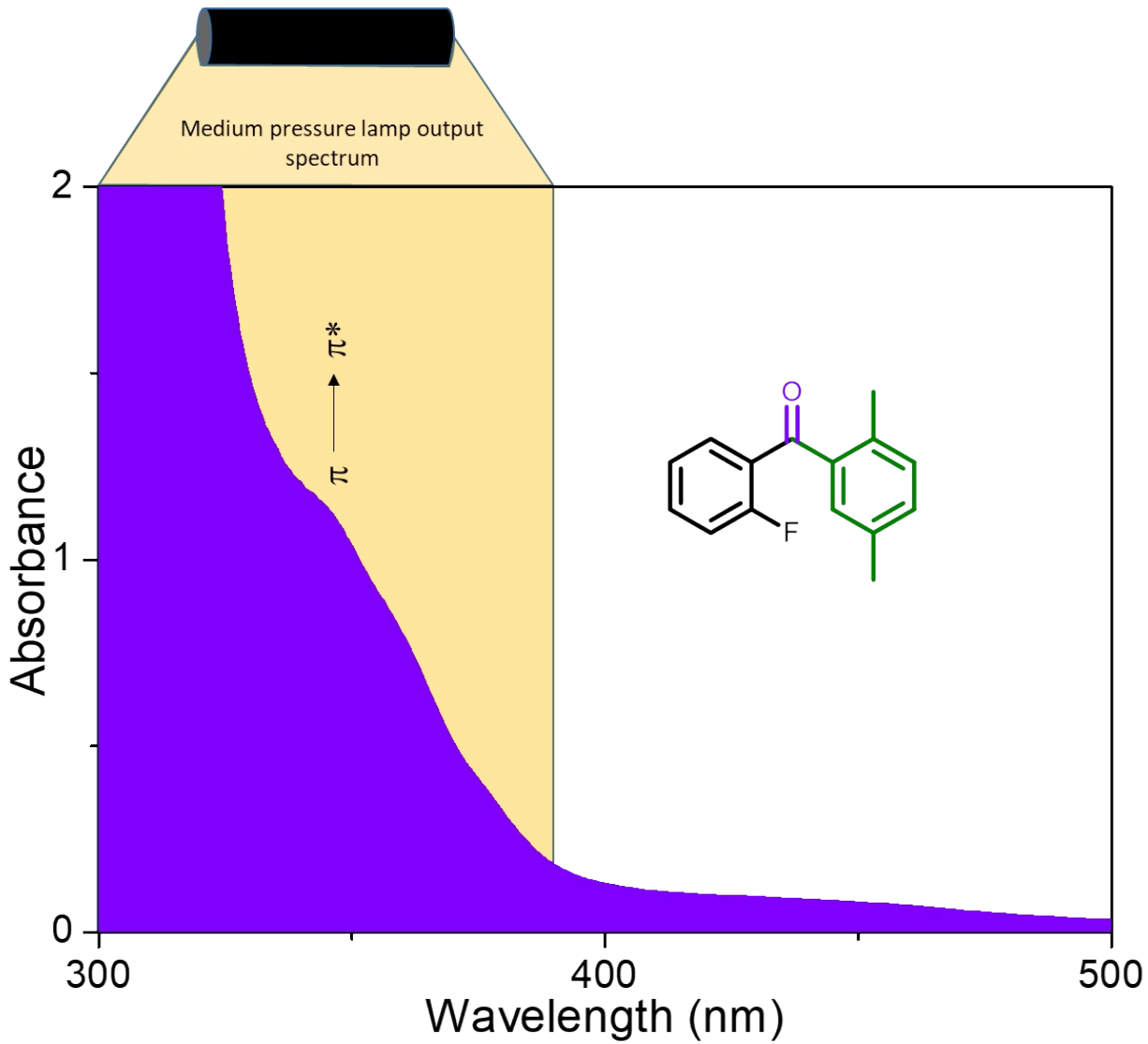


Figure S83. UV-Vis spectra of (2, 5-dimethylphenyl) (2-fluorophenyl) methanone (**2p**) in 0.005M ACN.

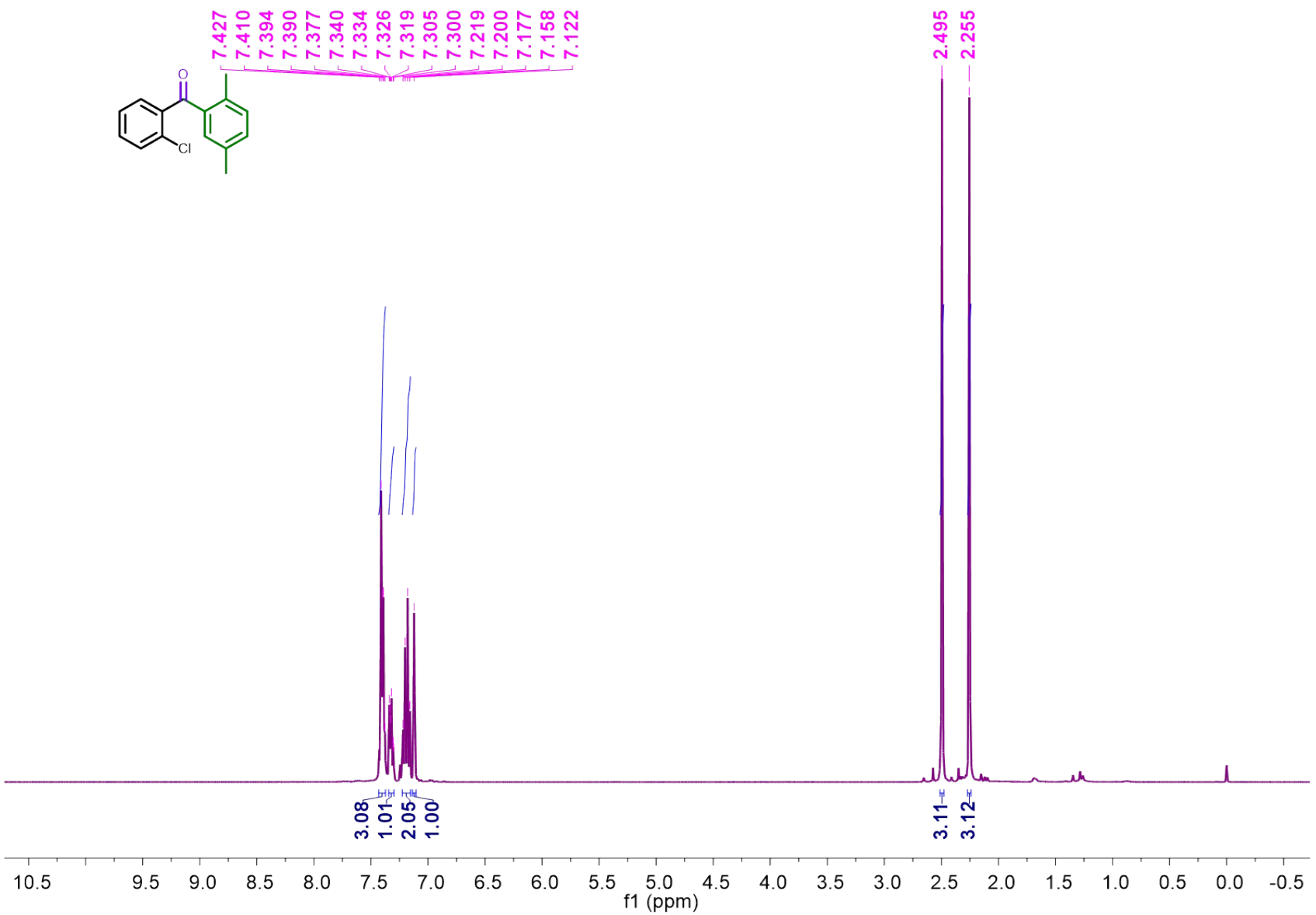


Figure S84. ^{1}H NMR spectra of (2, 5-dimethylphenyl) (2-chlorophenyl) methanone **(2q)** in $CDCl_3$.

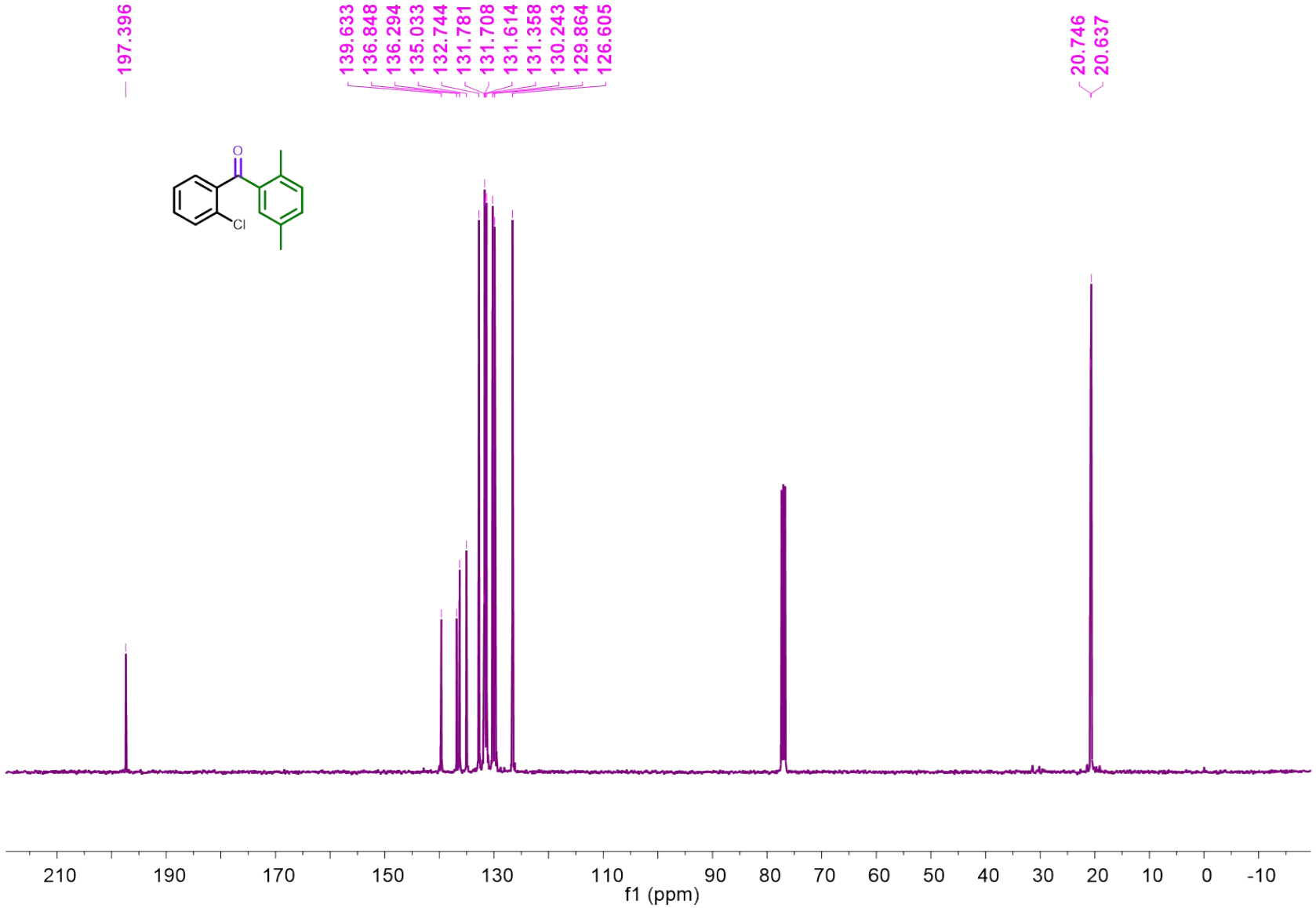


Figure S85. ^{13}C NMR spectra of (2, 5-dimethylphenyl) (2-chlorophenyl) methanone **(2q)** in $CDCl_3$.

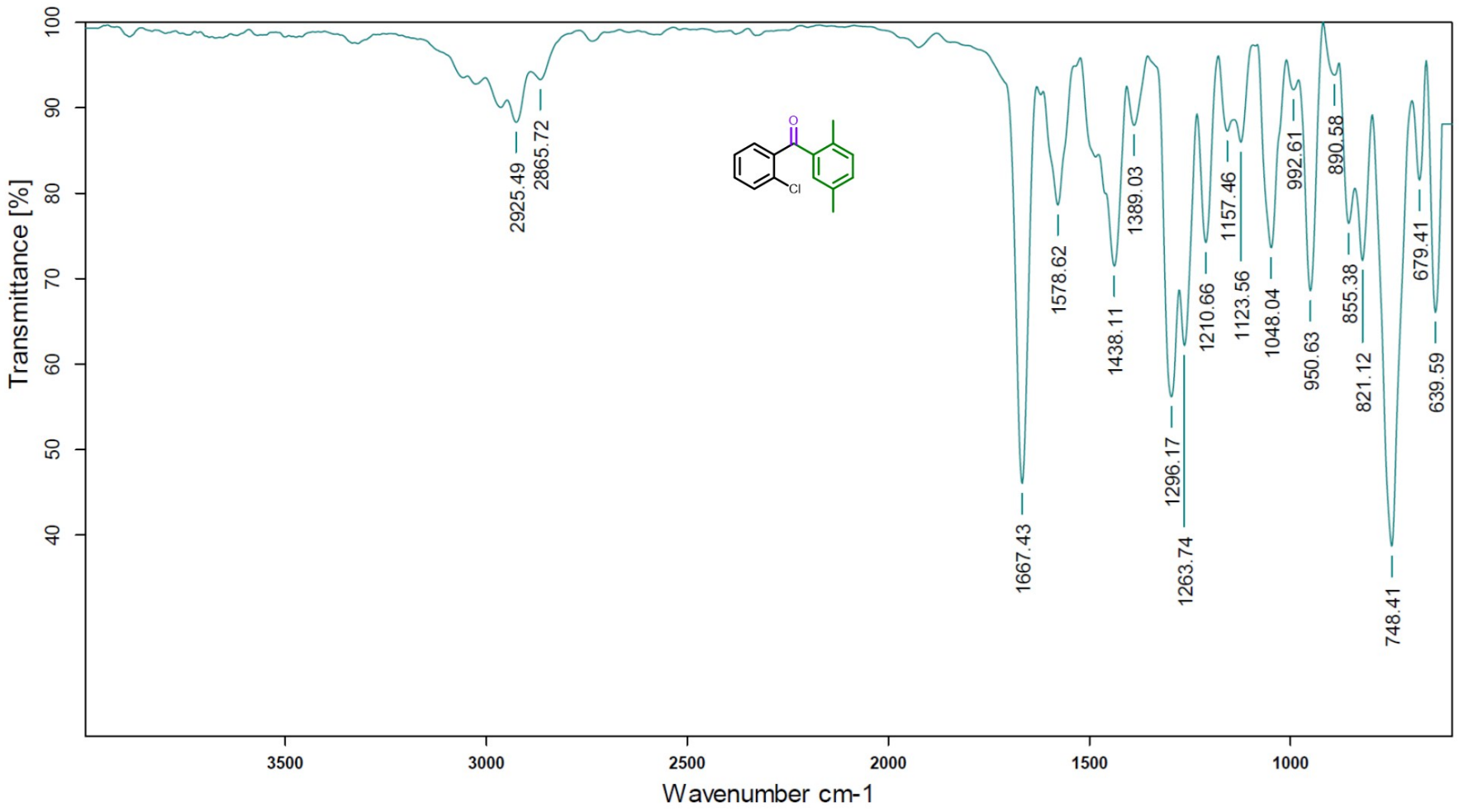


Figure S86. IR spectra of (2, 5-dimethylphenyl) (2-chlorophenyl) methanone **(2q**).

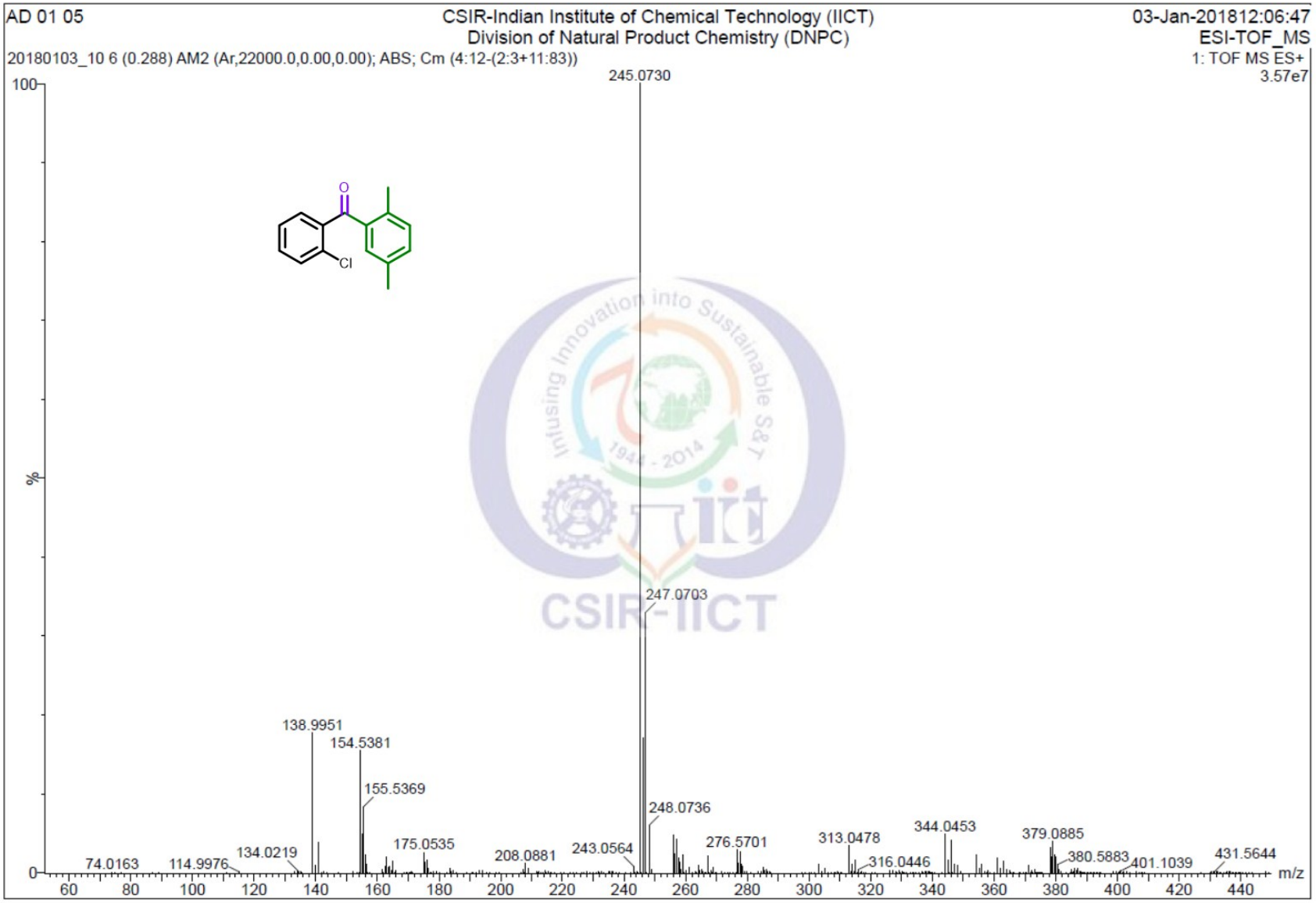


Figure S87. HRMS spectra of (2, 5-dimethylphenyl) (2-chlorophenyl) methanone **(2q)**.

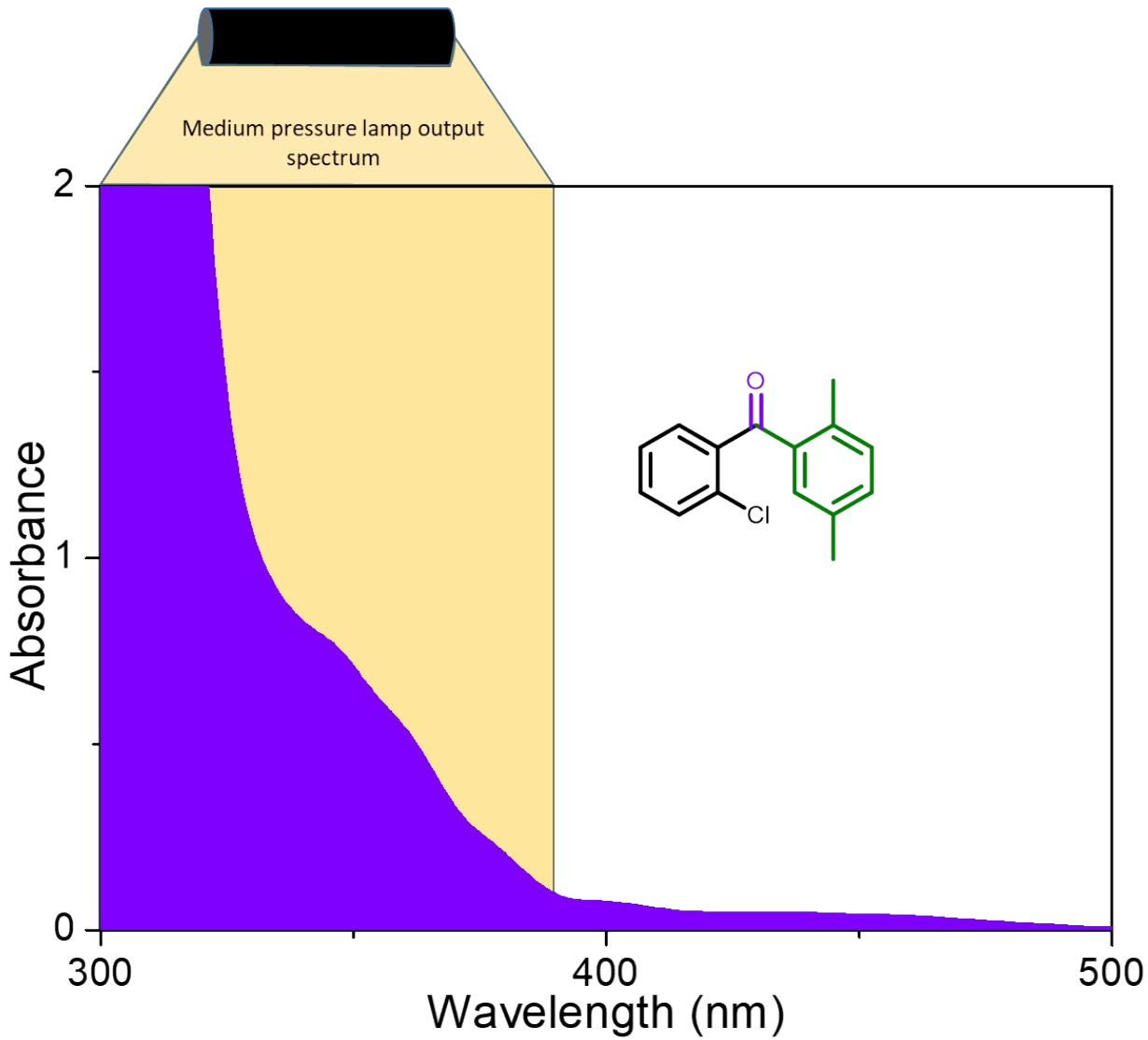


Figure S88. UV-Vis spectra of (2, 5-dimethylphenyl) (2-chlorophenyl) methanone **(2q**) in 0.005M ACN.

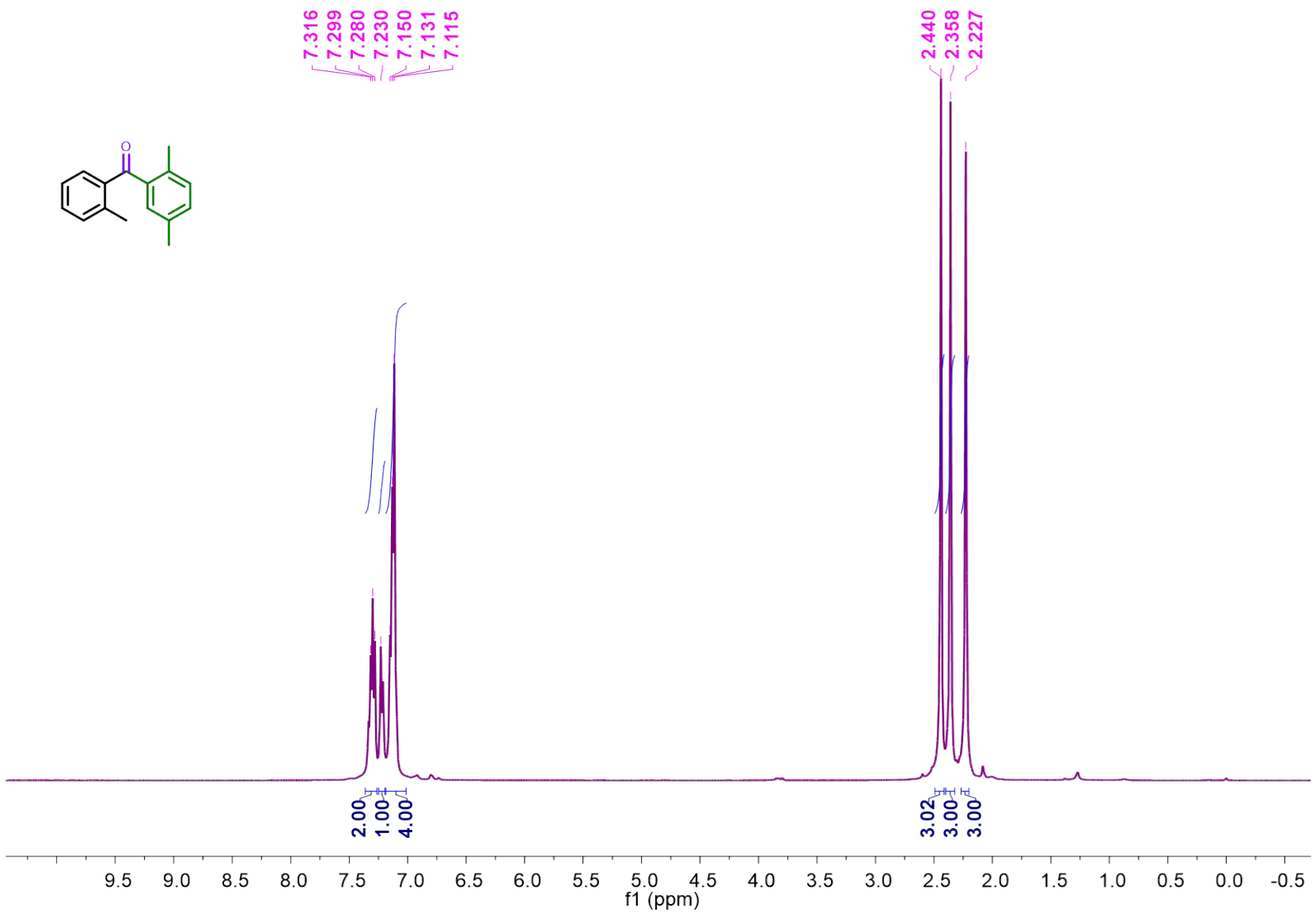


Figure S89. ^{1}H NMR spectra of (2,5-dimethylphenyl) (o-tolyl) methanone **(2r)** in $CDCl_3$.

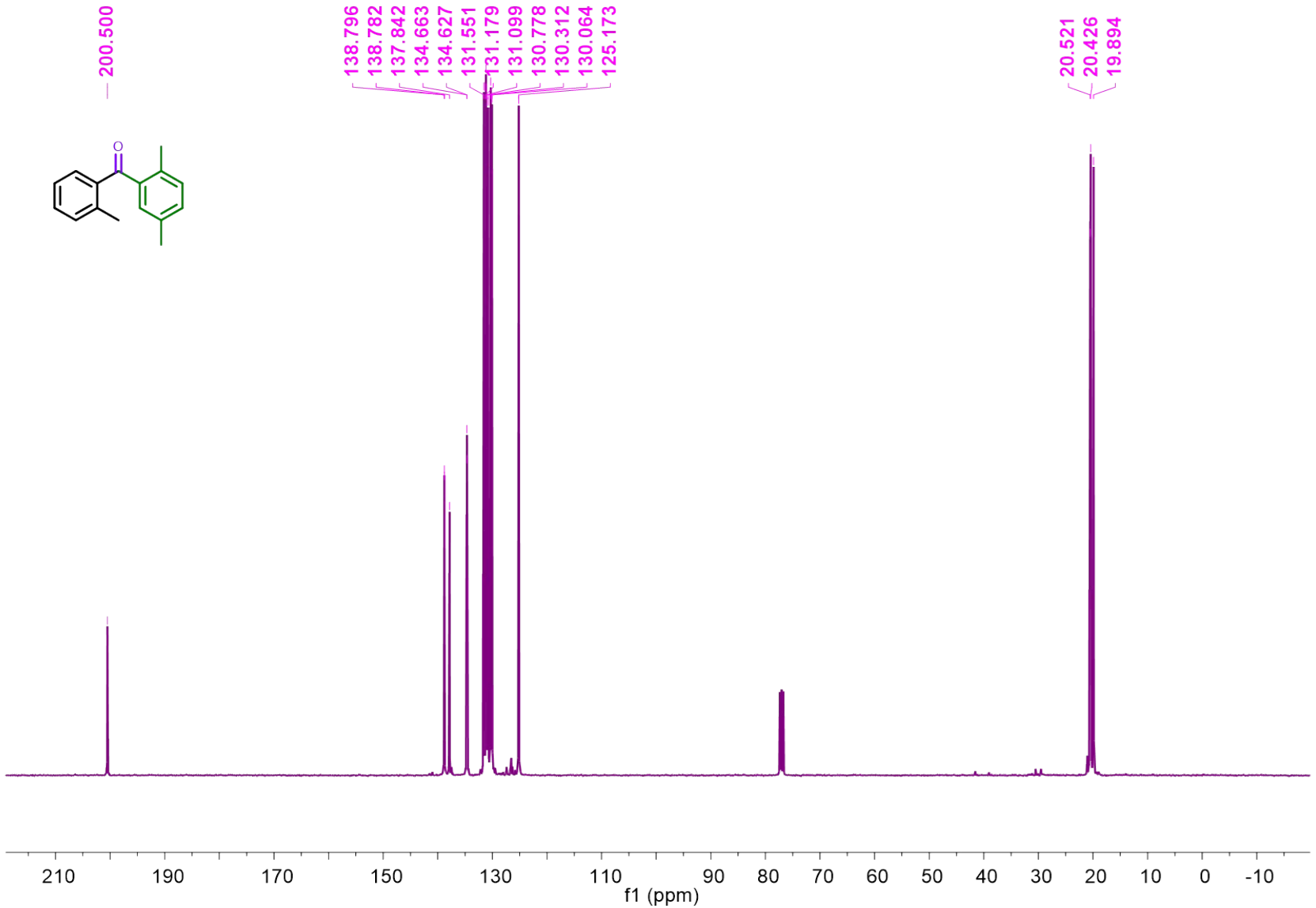


Figure S90. ^{13}C NMR spectra of (2,5-dimethylphenyl) (o-tolyl) methanone (**2r**) in $CDCl_3$.

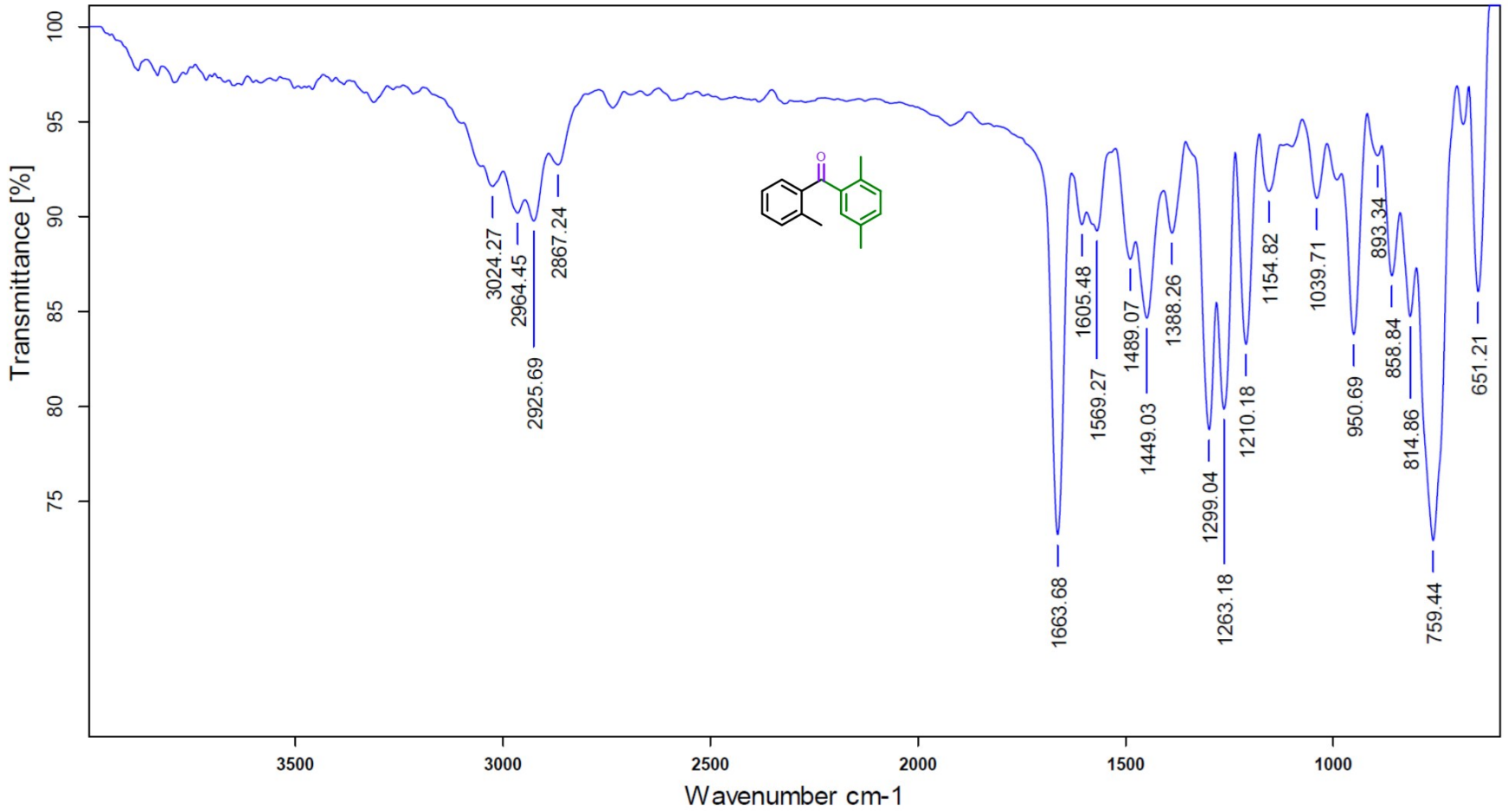


Figure S91. IR spectra of (2,5-dimethylphenyl) (o-tolyl) methanone **(2r**).

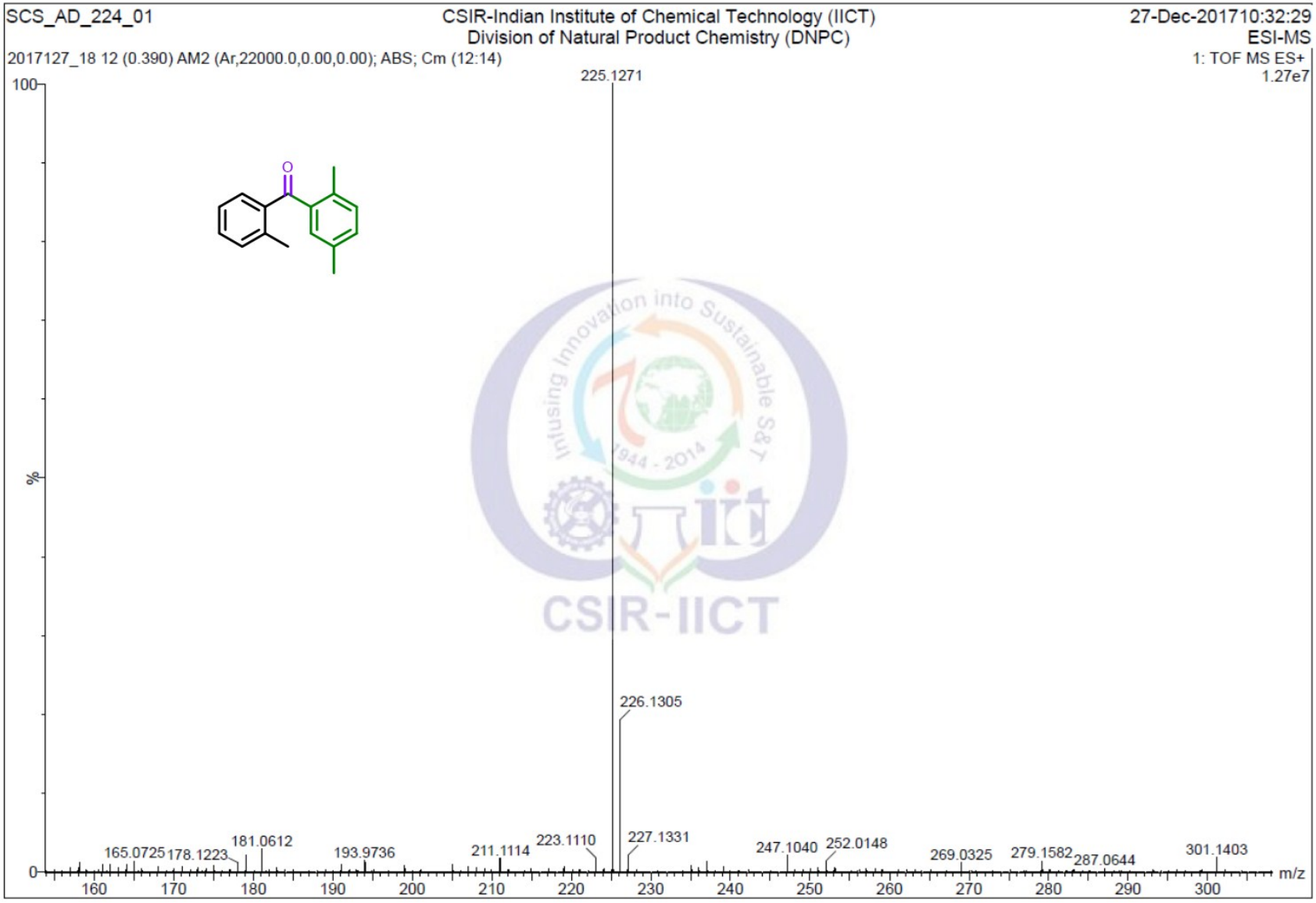


Figure S92. HRMS spectra of (2,5-dimethylphenyl) (o-tolyl) methanone **(2r)**.

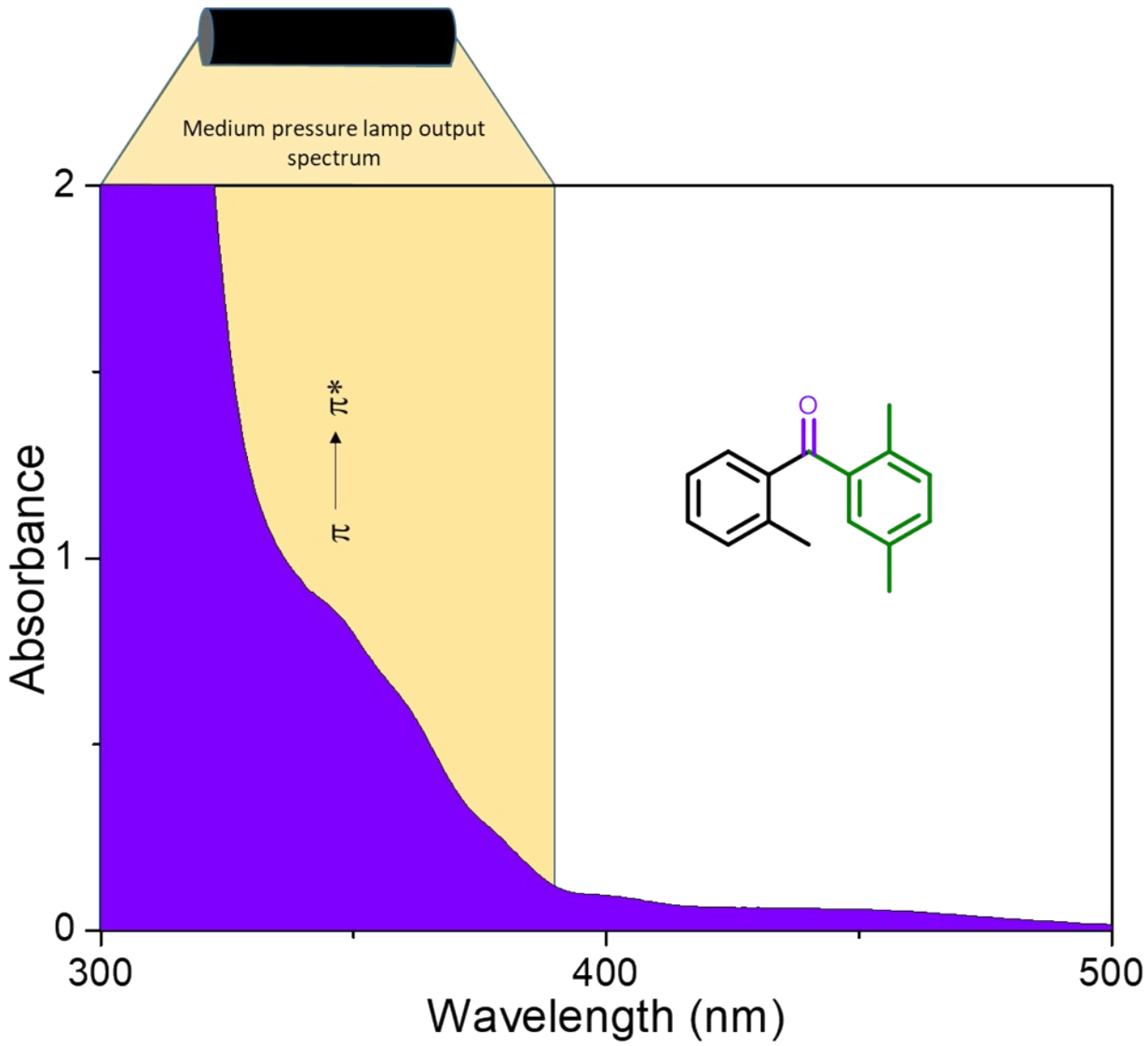


Figure S93. UV-Vis spectra of (2,5-dimethylphenyl) (o-tolyl) methanone **(2r**) in 0.005M ACN.

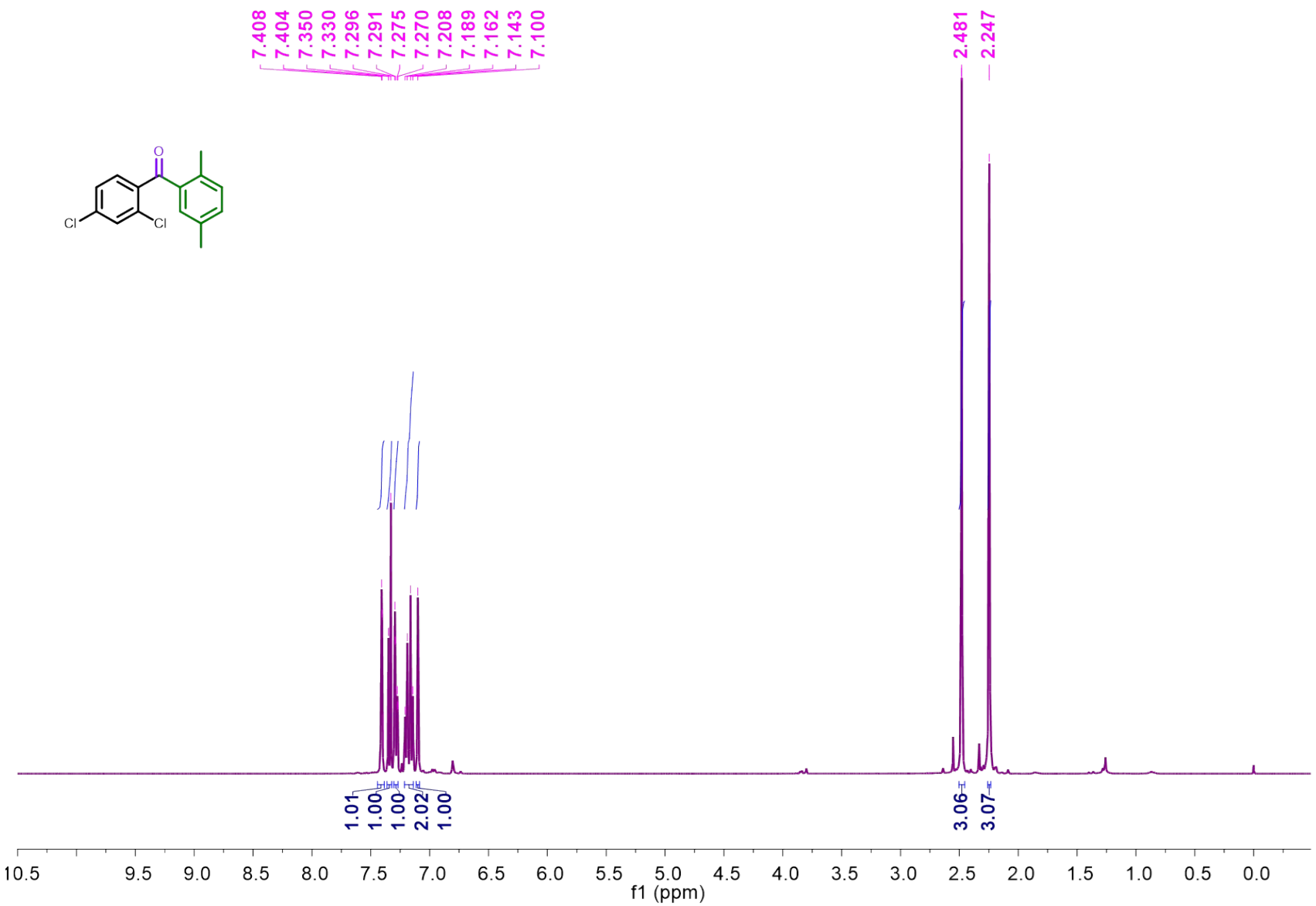


Figure S94. ^{1}H NMR spectra of (2,4-dichlorophenyl) (2,5-dimethylphenyl) methanone **(2s)** in $CDCl_3$.

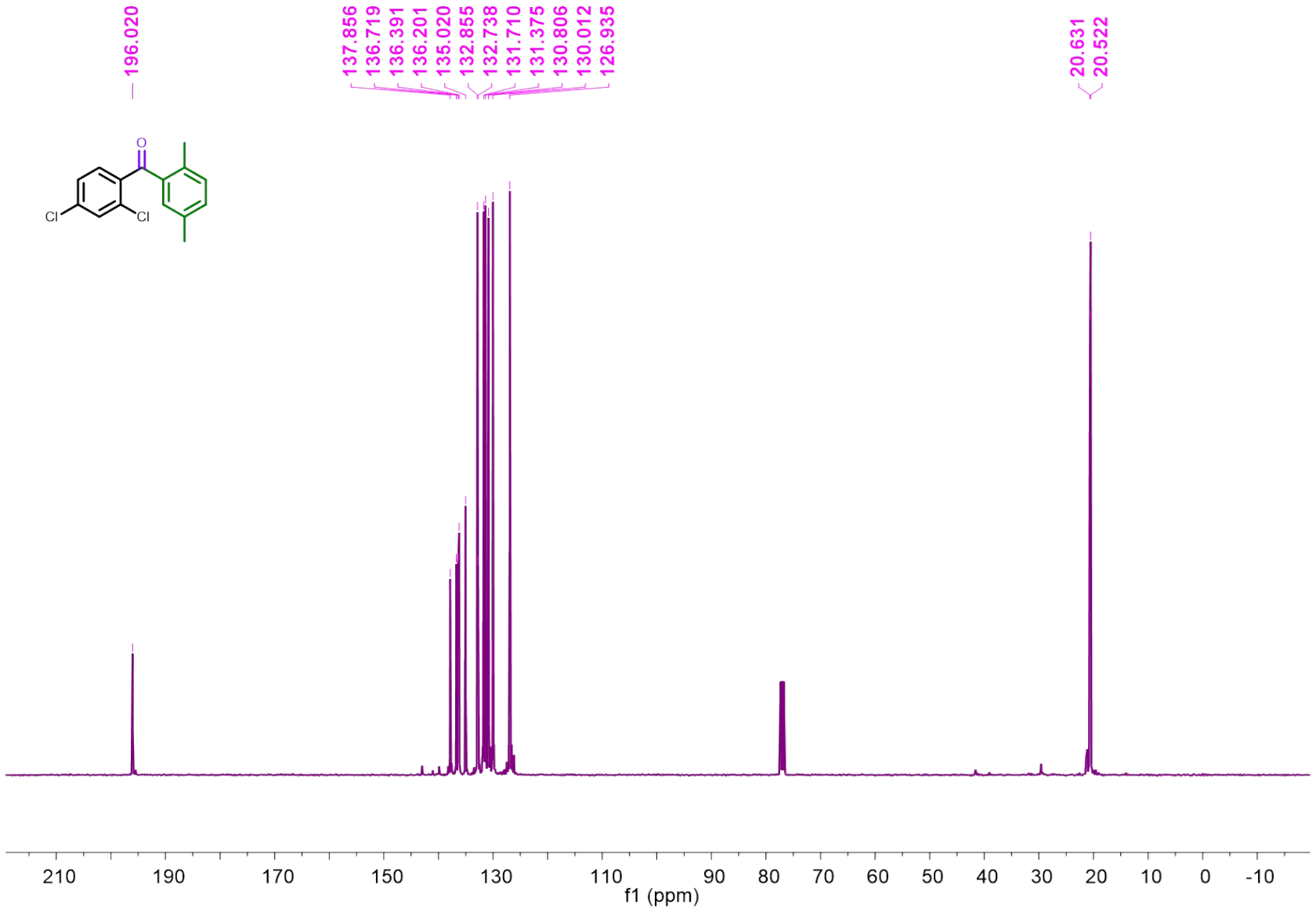


Figure S95. ^{13}C NMR spectra of (2,4-dichlorophenyl) (2,5-dimethylphenyl) methanone **(2s)** in $CDCl_3$.

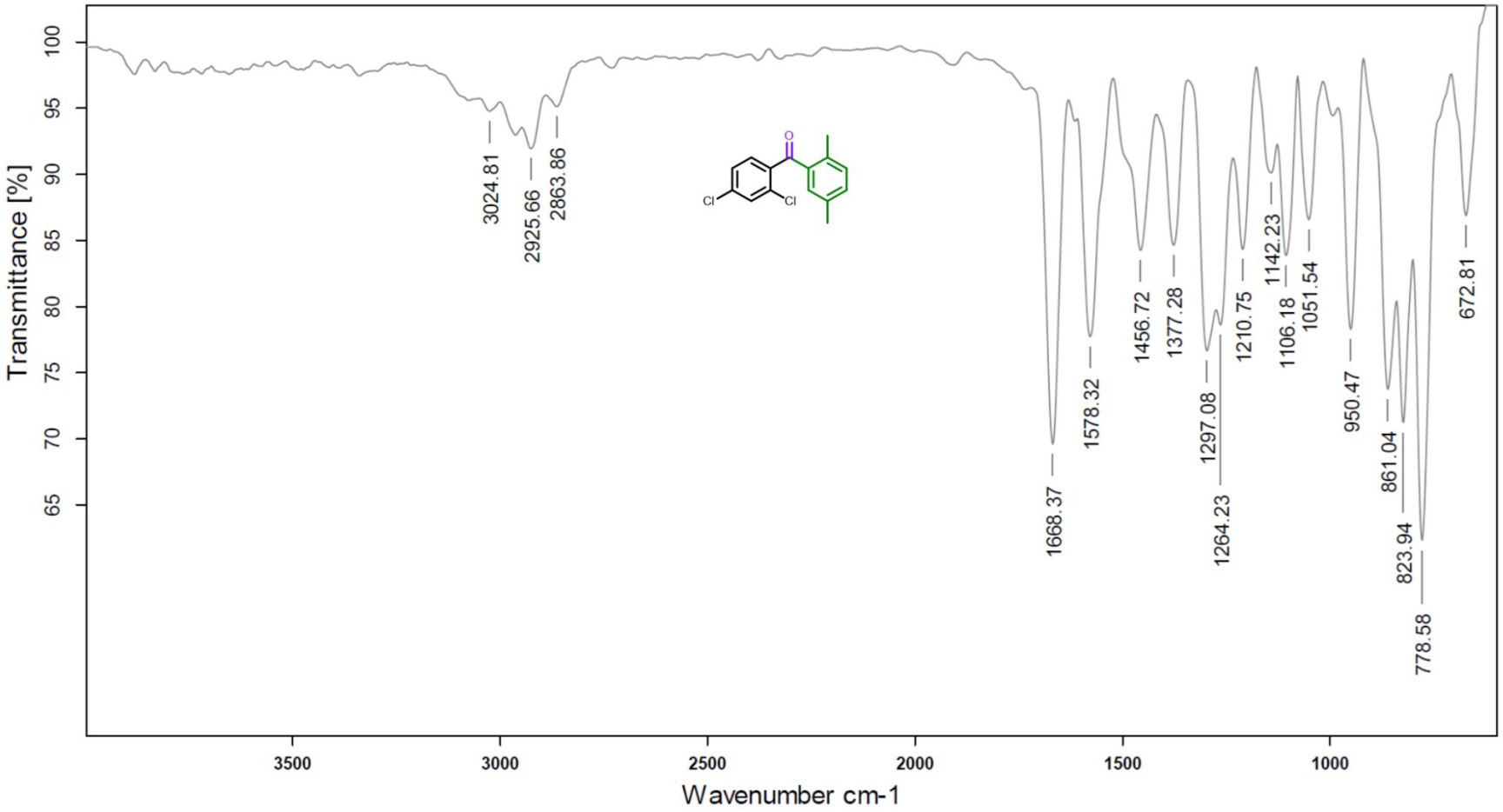


Figure S96. IR spectra of (2,4-dichlorophenyl) (2,5-dimethylphenyl) methanone **(2s**).

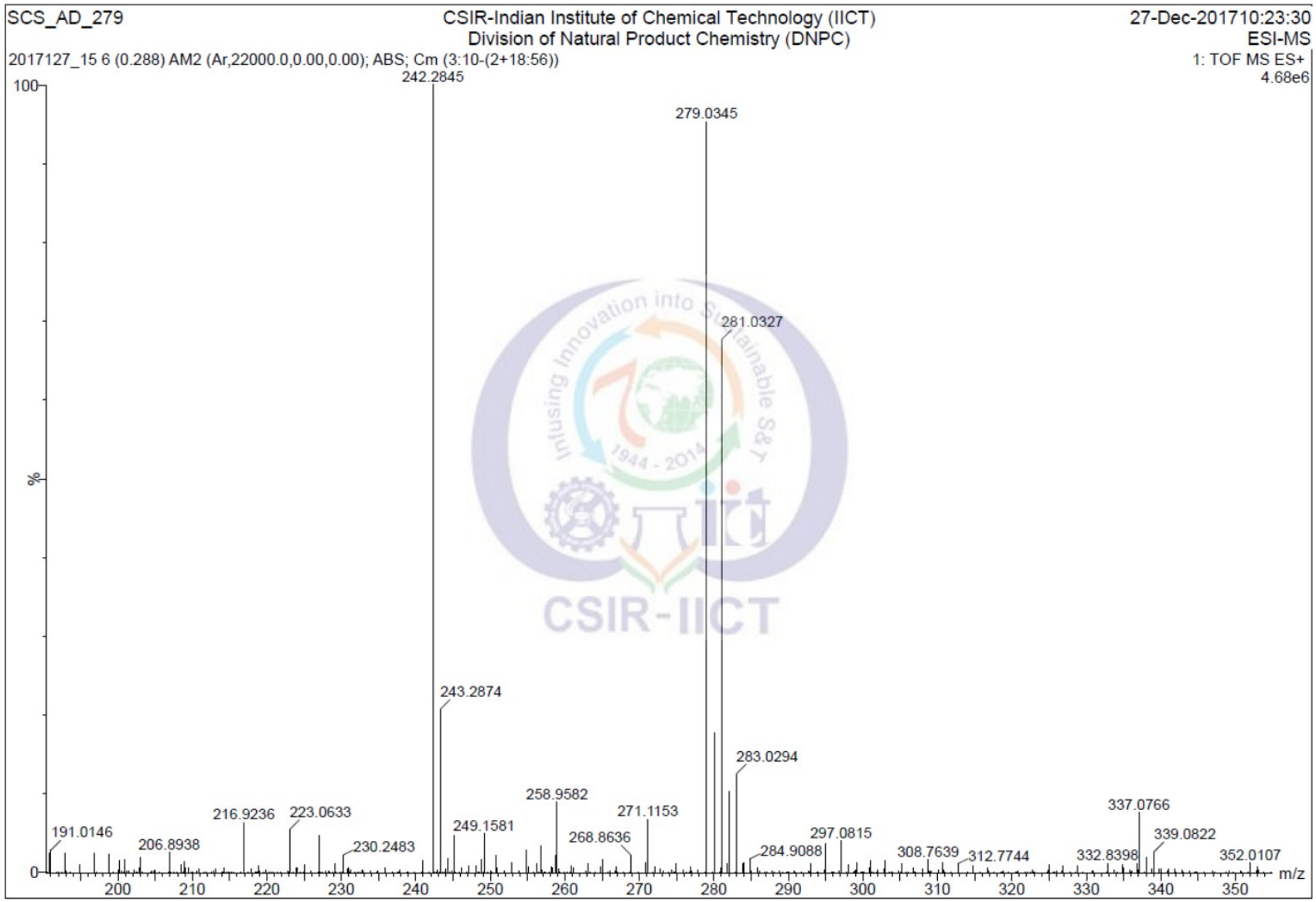


Figure S97. HRMS spectra of (2,4-dichlorophenyl) (2,5-dimethylphenyl) methanone **(2s**).

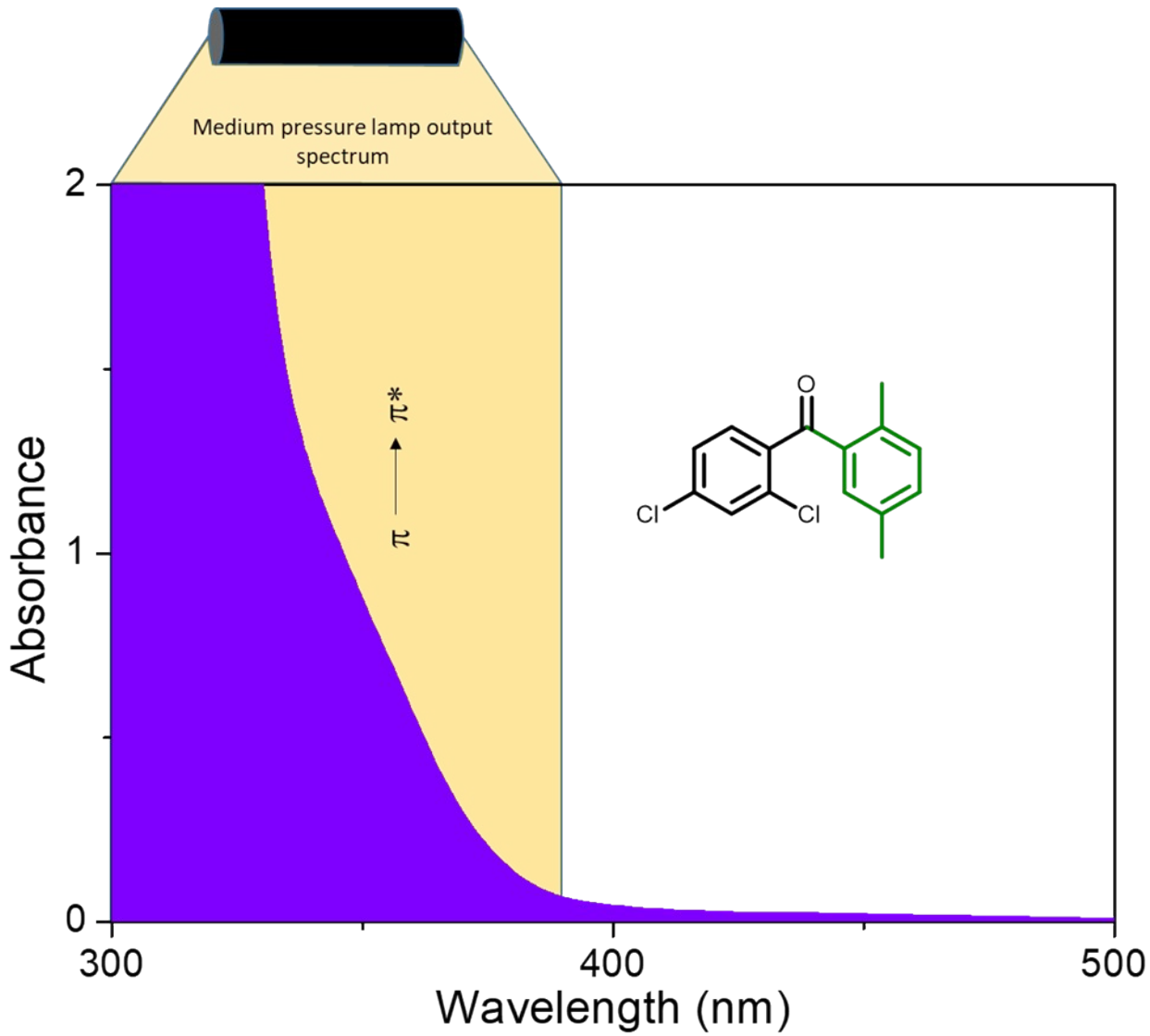


Figure S98. UV-Vis spectra of (2,4-dichlorophenyl) (2,5-dimethylphenyl) methanone **(2s**) in 0.005M ACN.

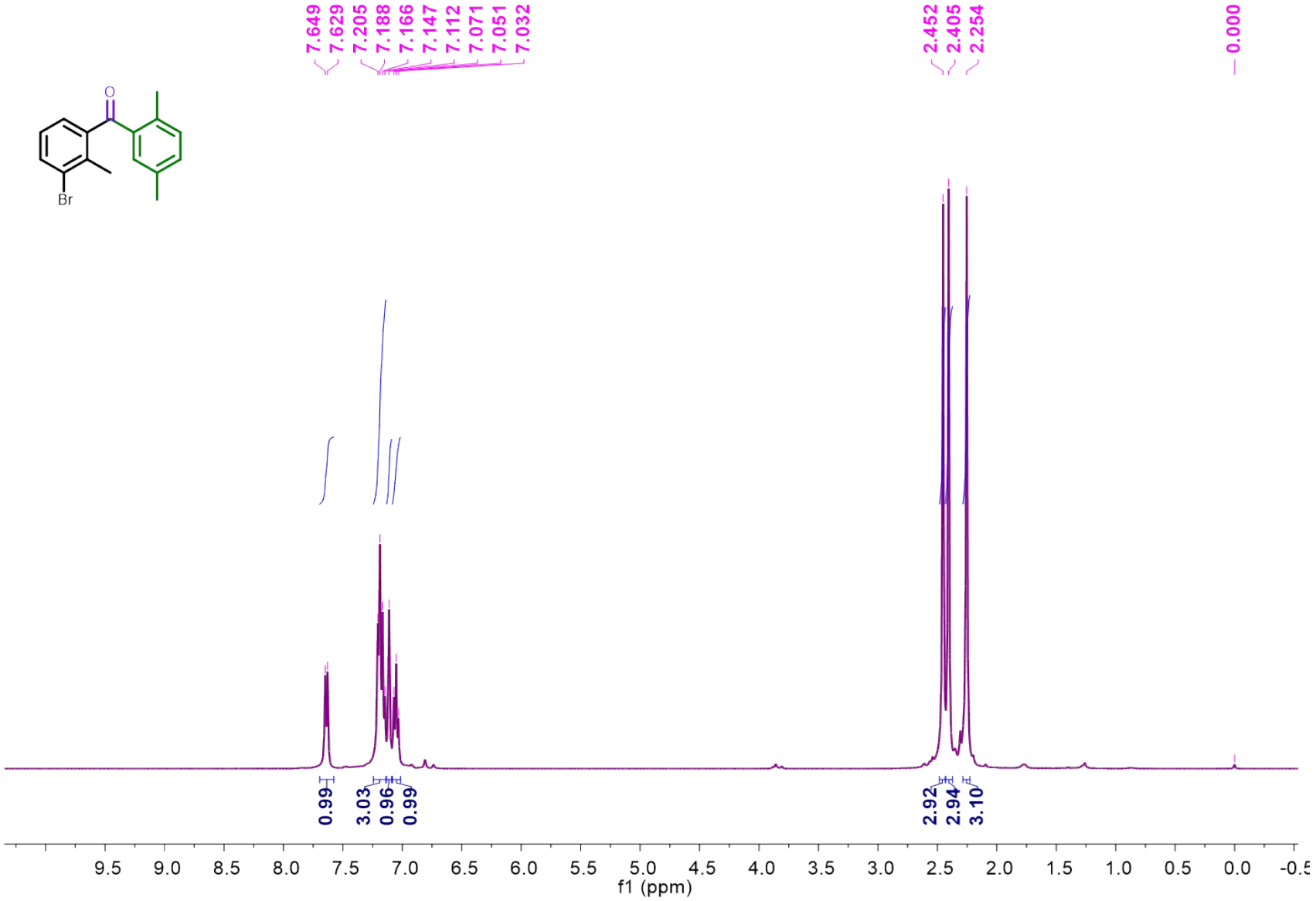


Figure S99. ^{1}H NMR spectra of (3-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanone **(2t)** in $CDCl_3$.

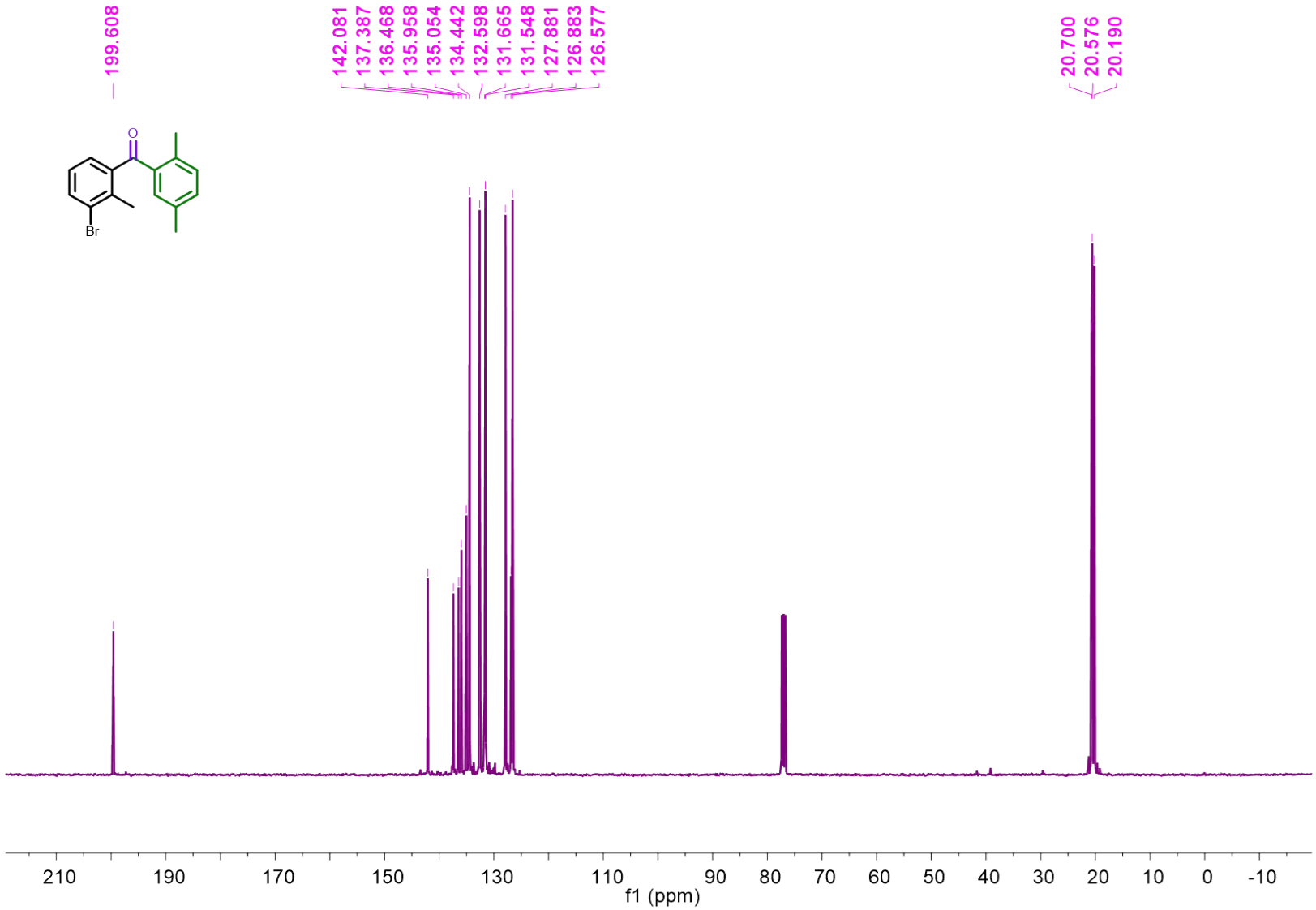


Figure S100. ^{13}C NMR spectra of (3-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanone **(2t)** in $CDCl_3$.

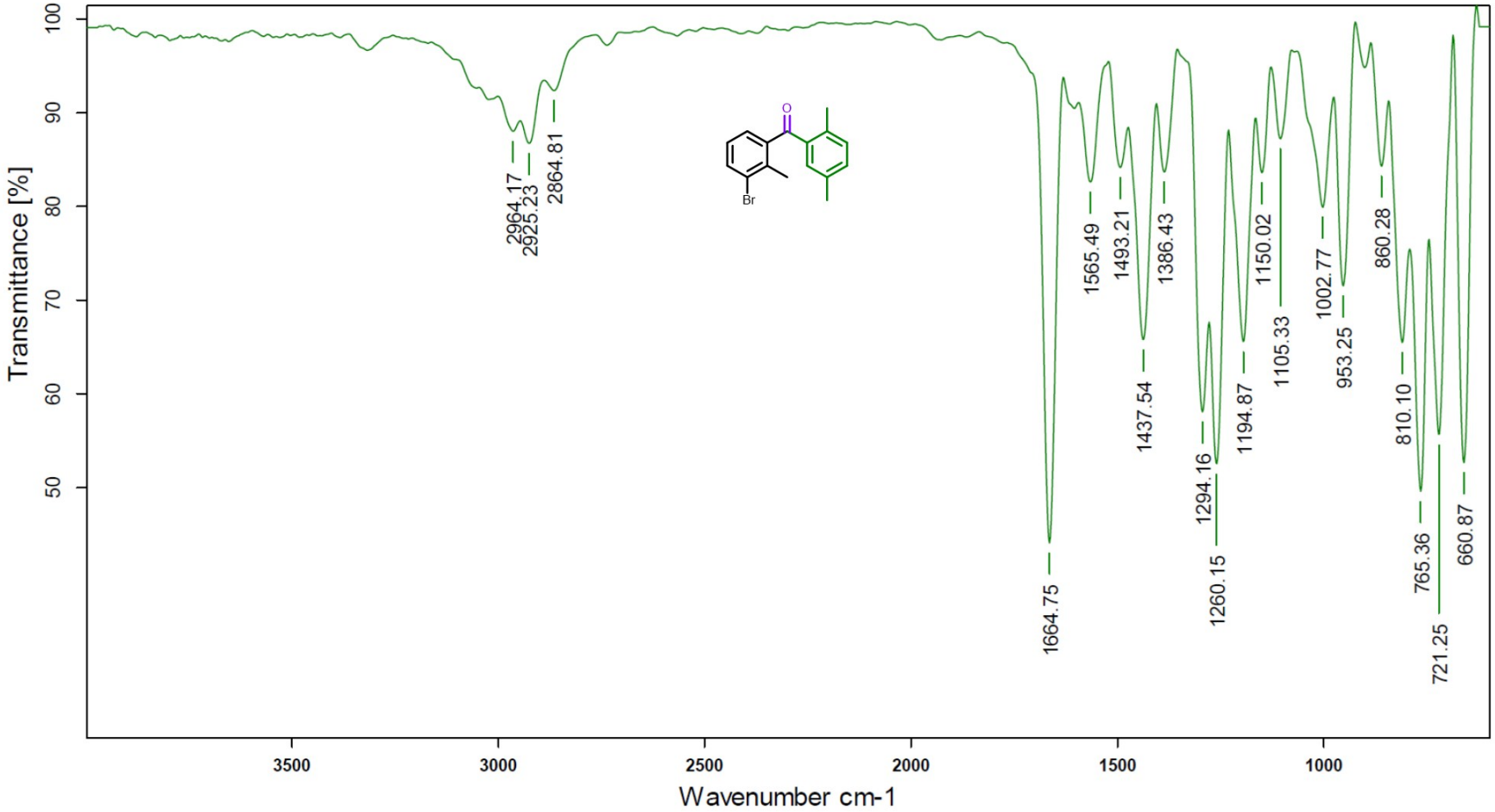


Figure S101. IR spectra of (3-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanone **(2t)**.

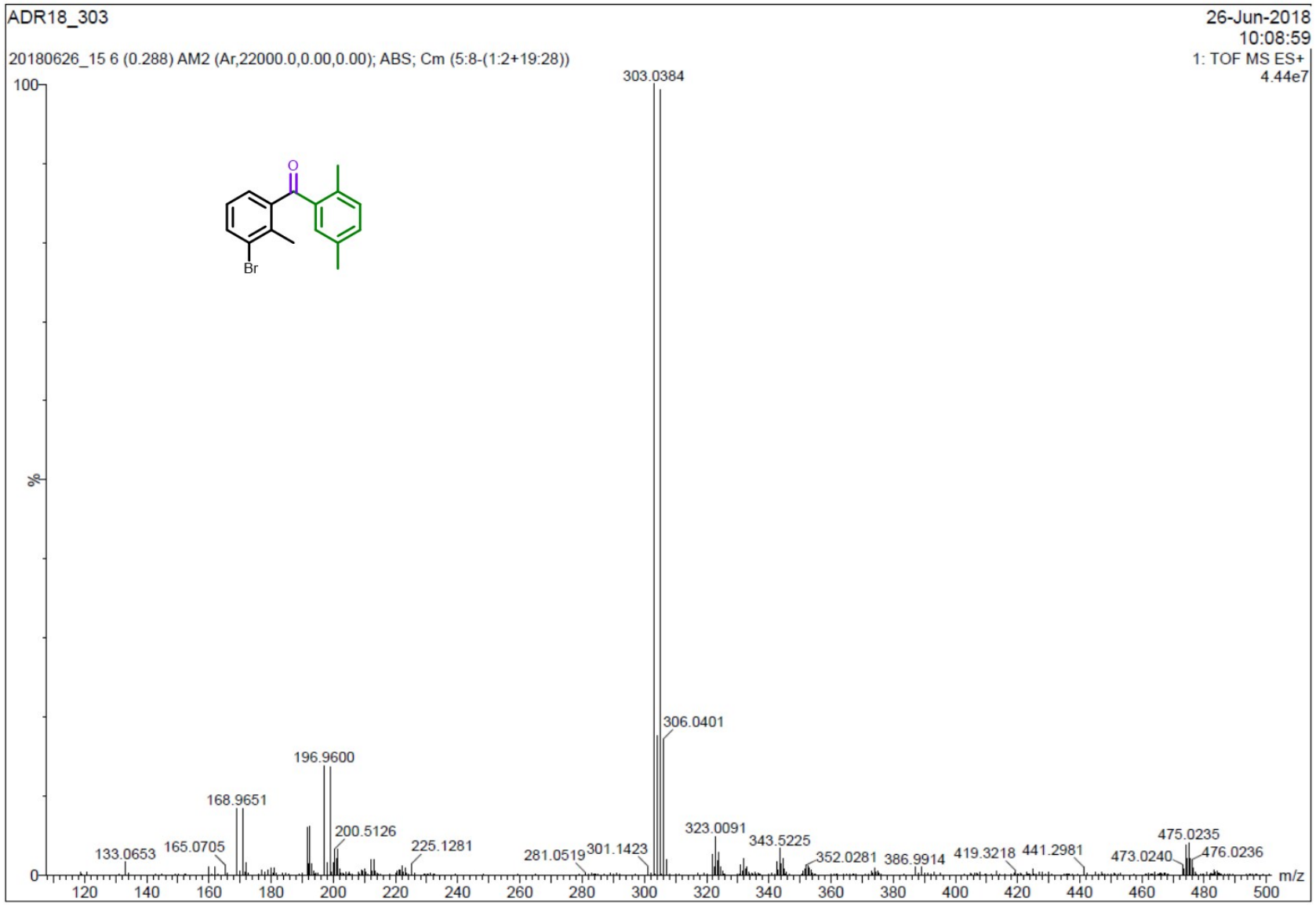


Figure S102. HRMS spectra of (3-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanone **(2t)**.

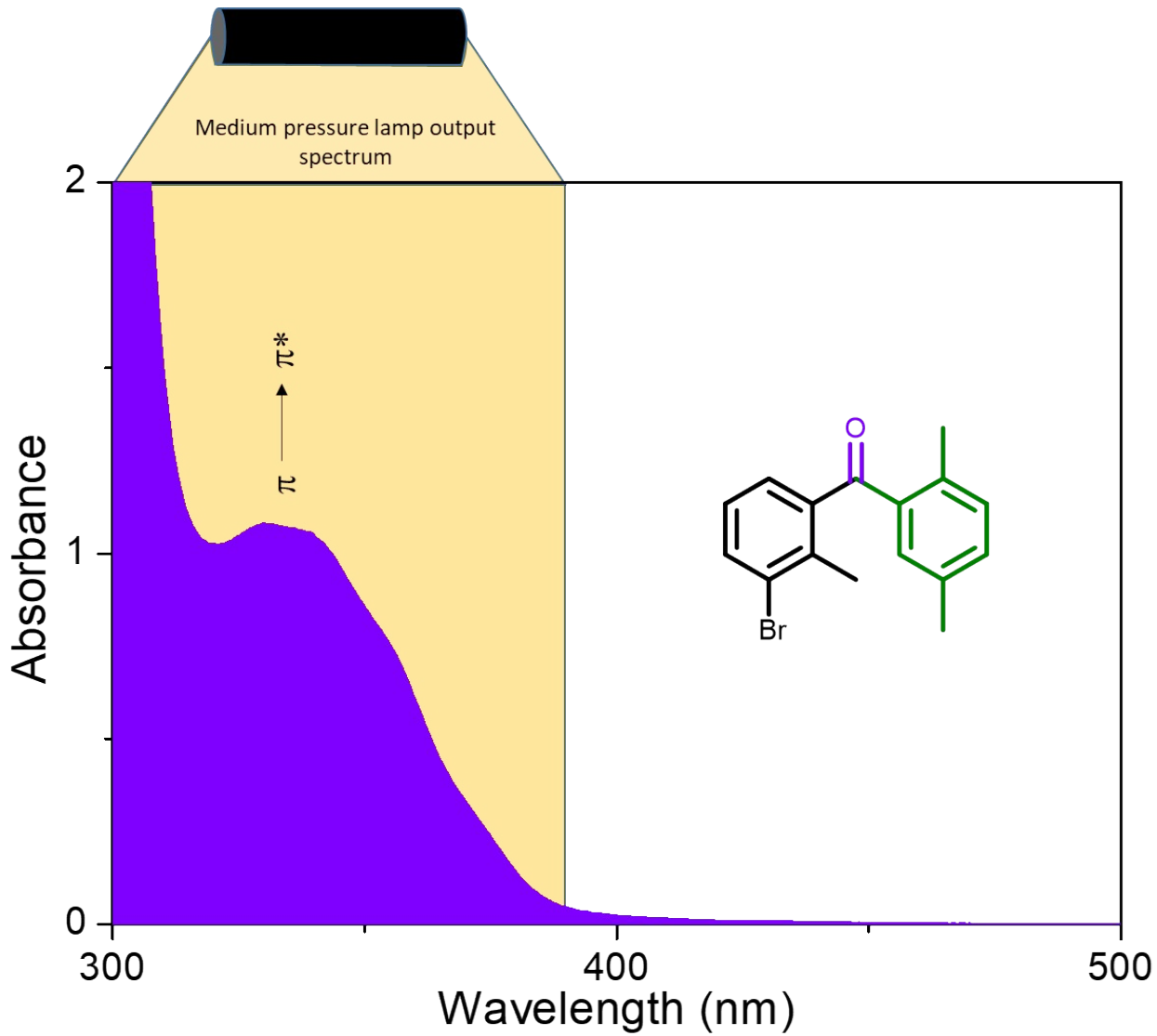


Figure S103. UV-Vis spectra of (3-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanone **(2t)** in 0.005M ACN.

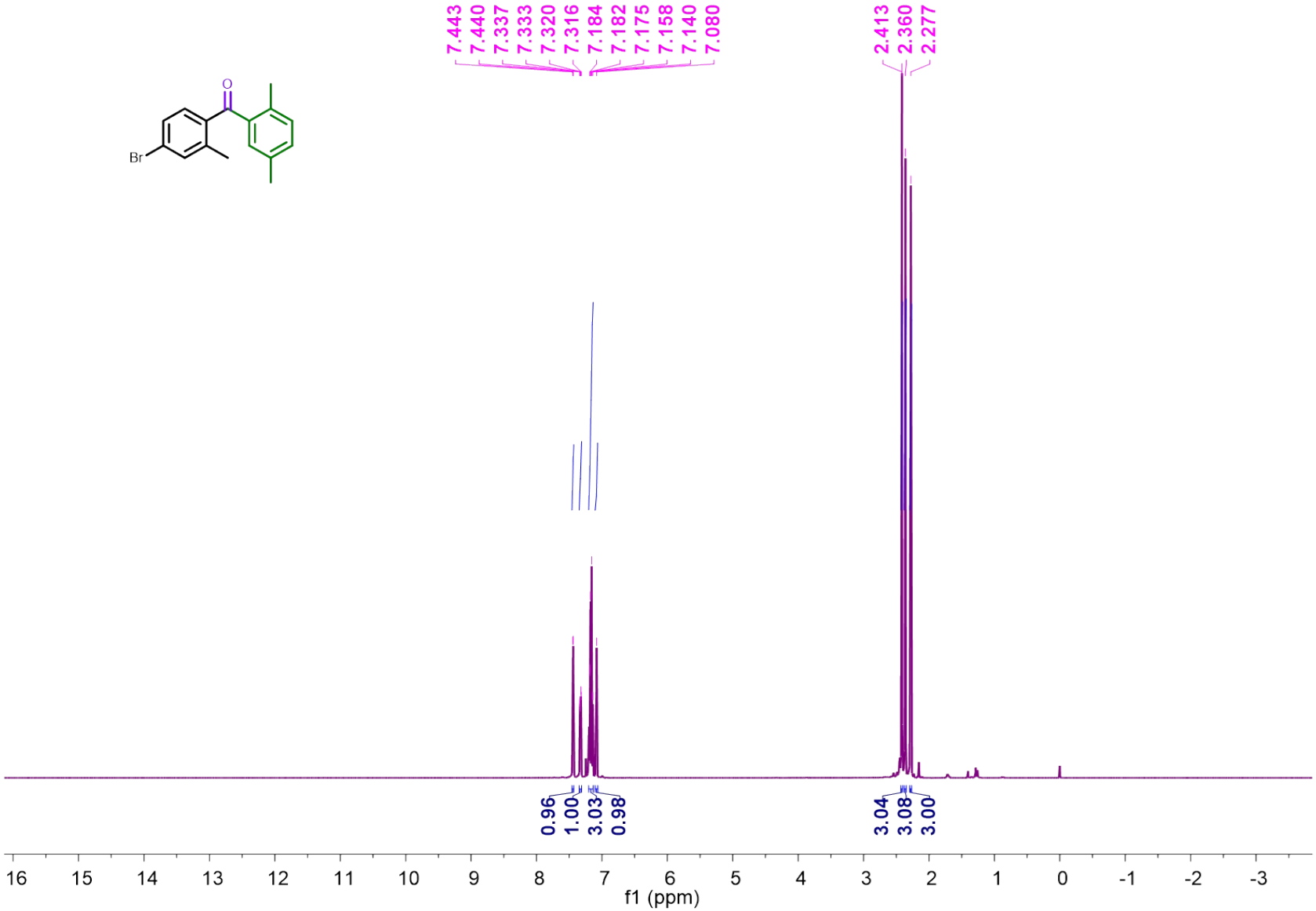


Figure S104. ^{1}H NMR spectra of (4-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanone **(2u)** in $CDCl_3$.

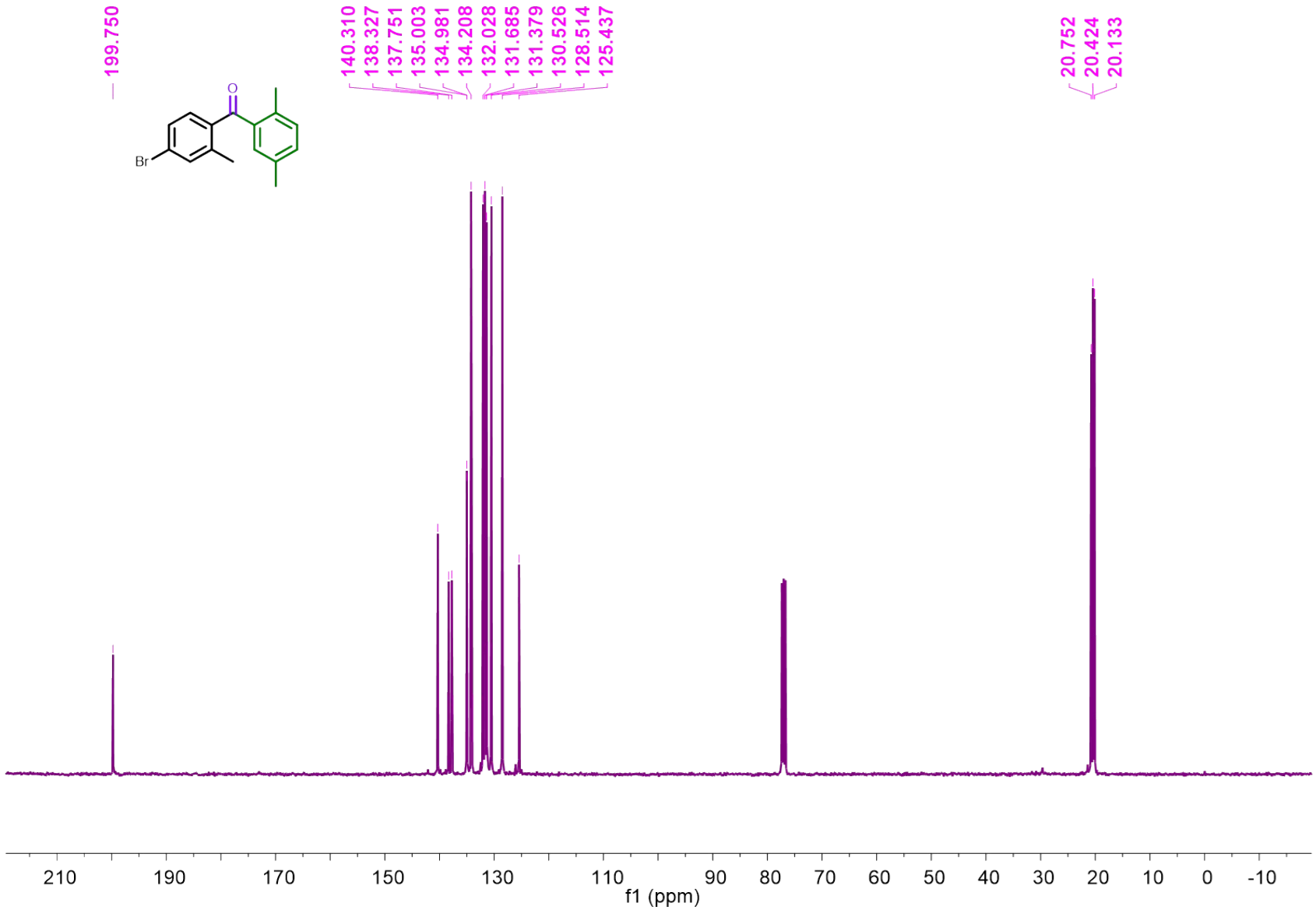


Figure S105. ^{13}C NMR spectra of (4-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanone **(2u)** in $CDCl_3$.

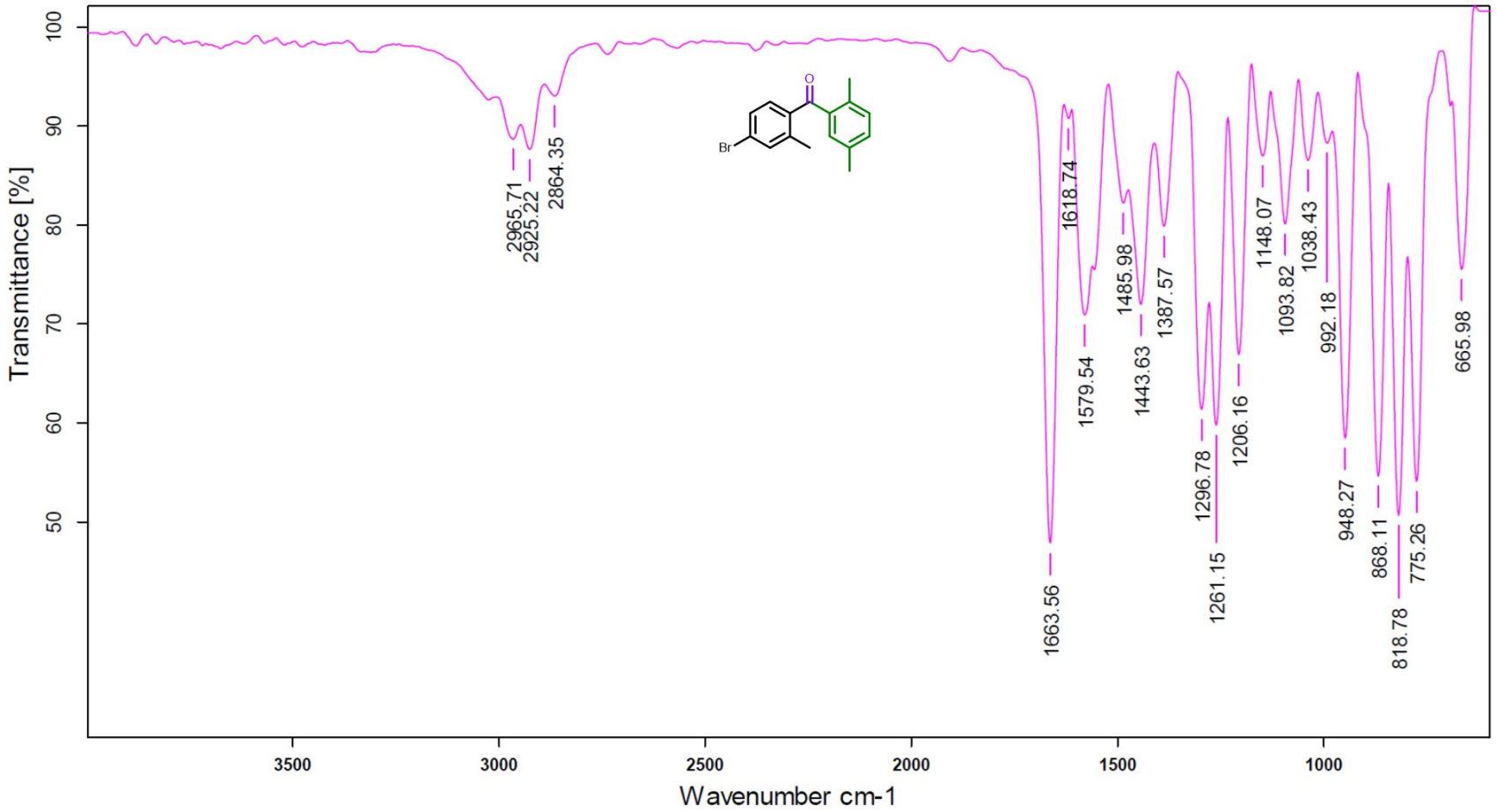


Figure S106. IR spectra of (4-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanone **(2u)**.

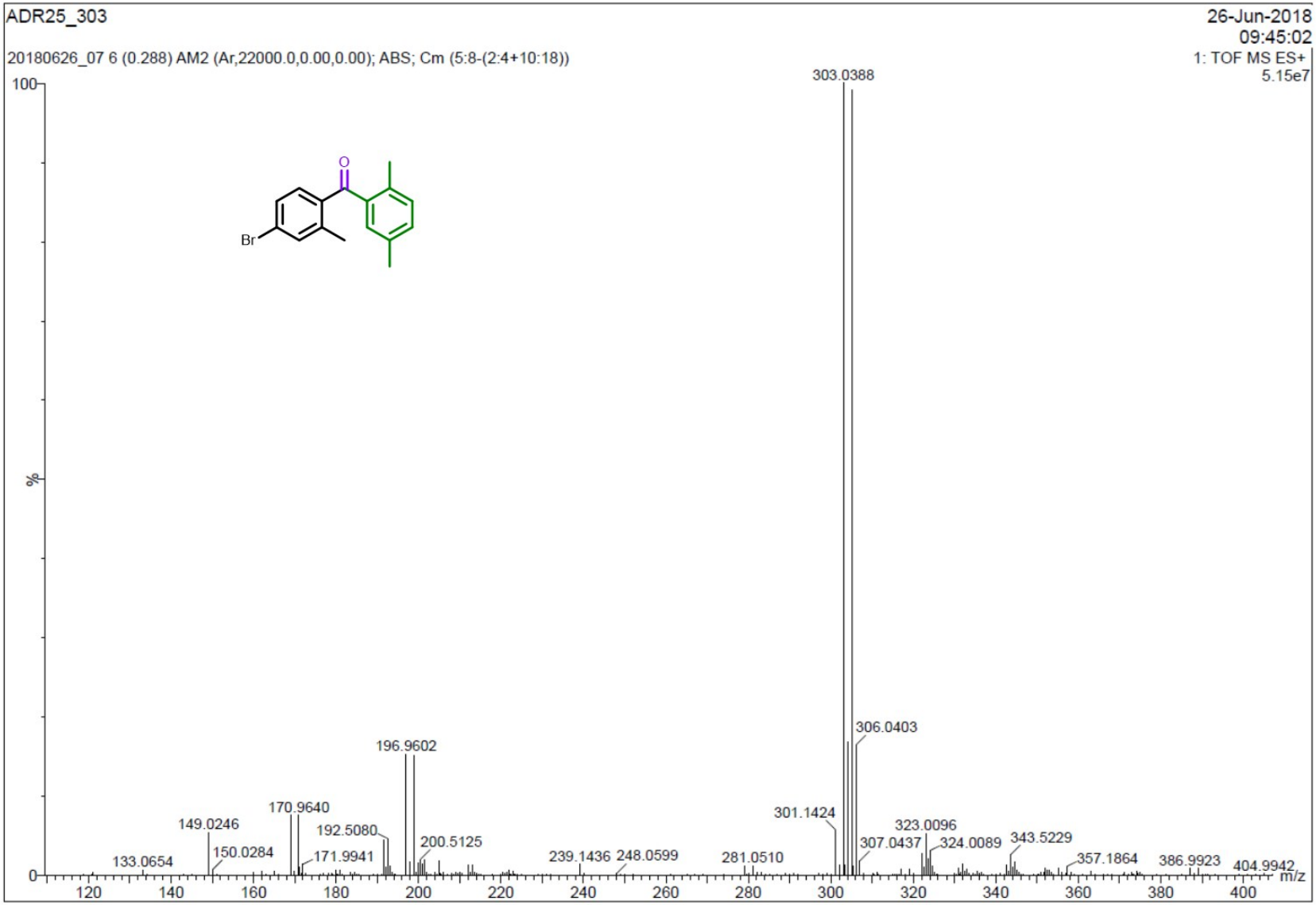


Figure S107. HRMS spectra of (4-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanone **(2u)**.

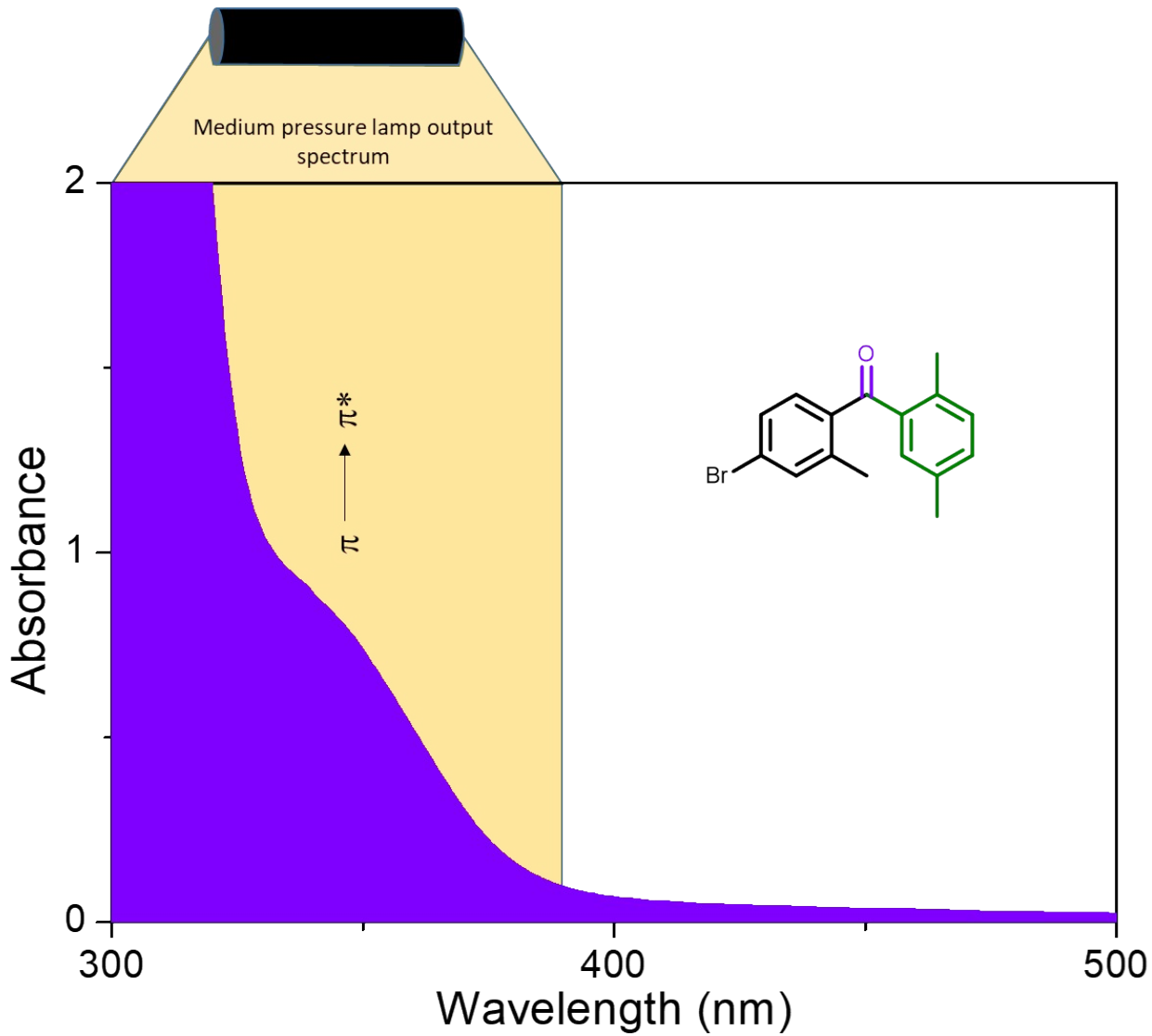


Figure S108. HRMS spectra of (4-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanone **(2u**) in 0.005M ACN.

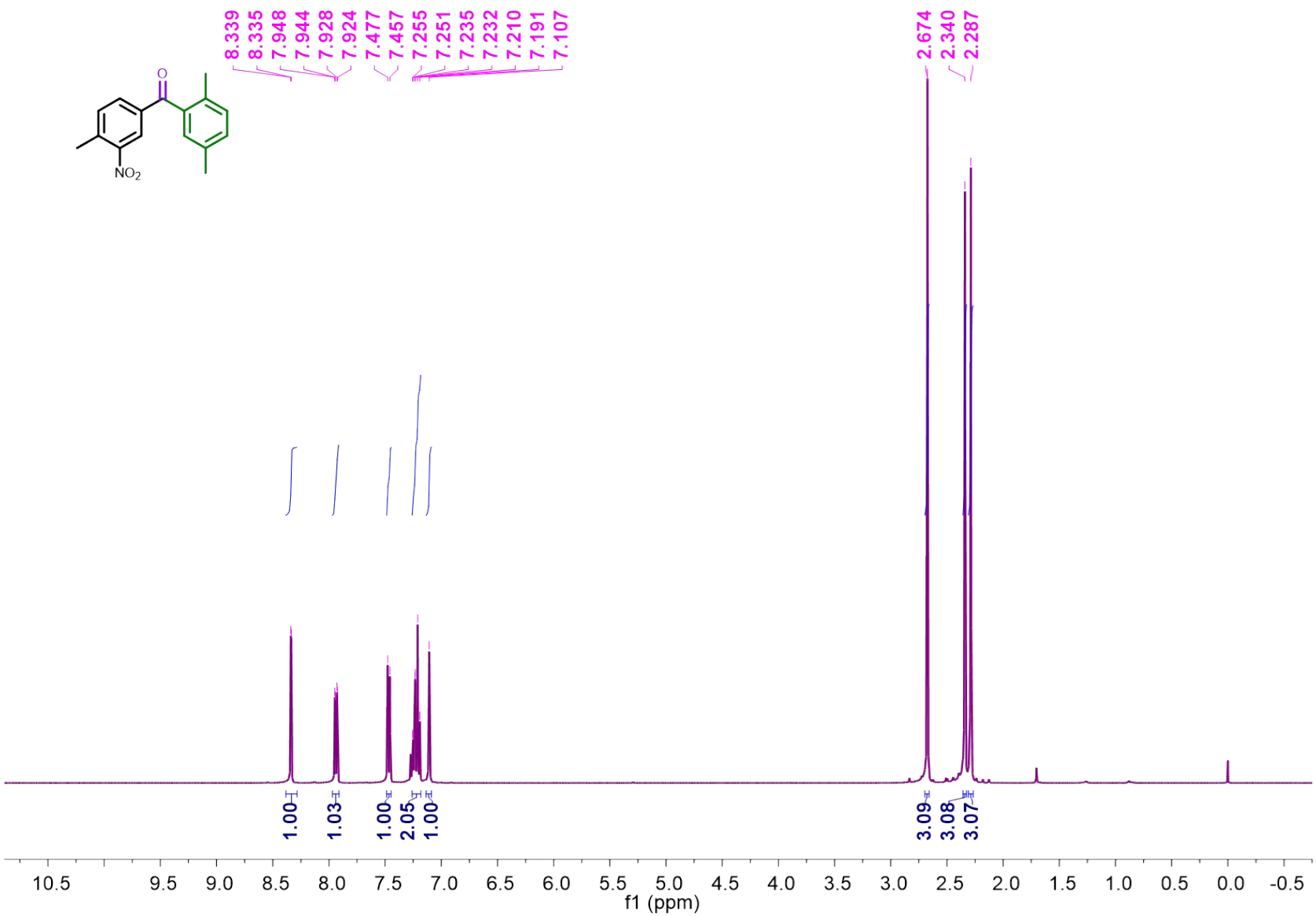


Figure S109. ^{1}H NMR spectra of (2,5-dimethylphenyl) (4-methyl-3-nitrophenyl) methanone **(2v)** in $CDCl_3$.

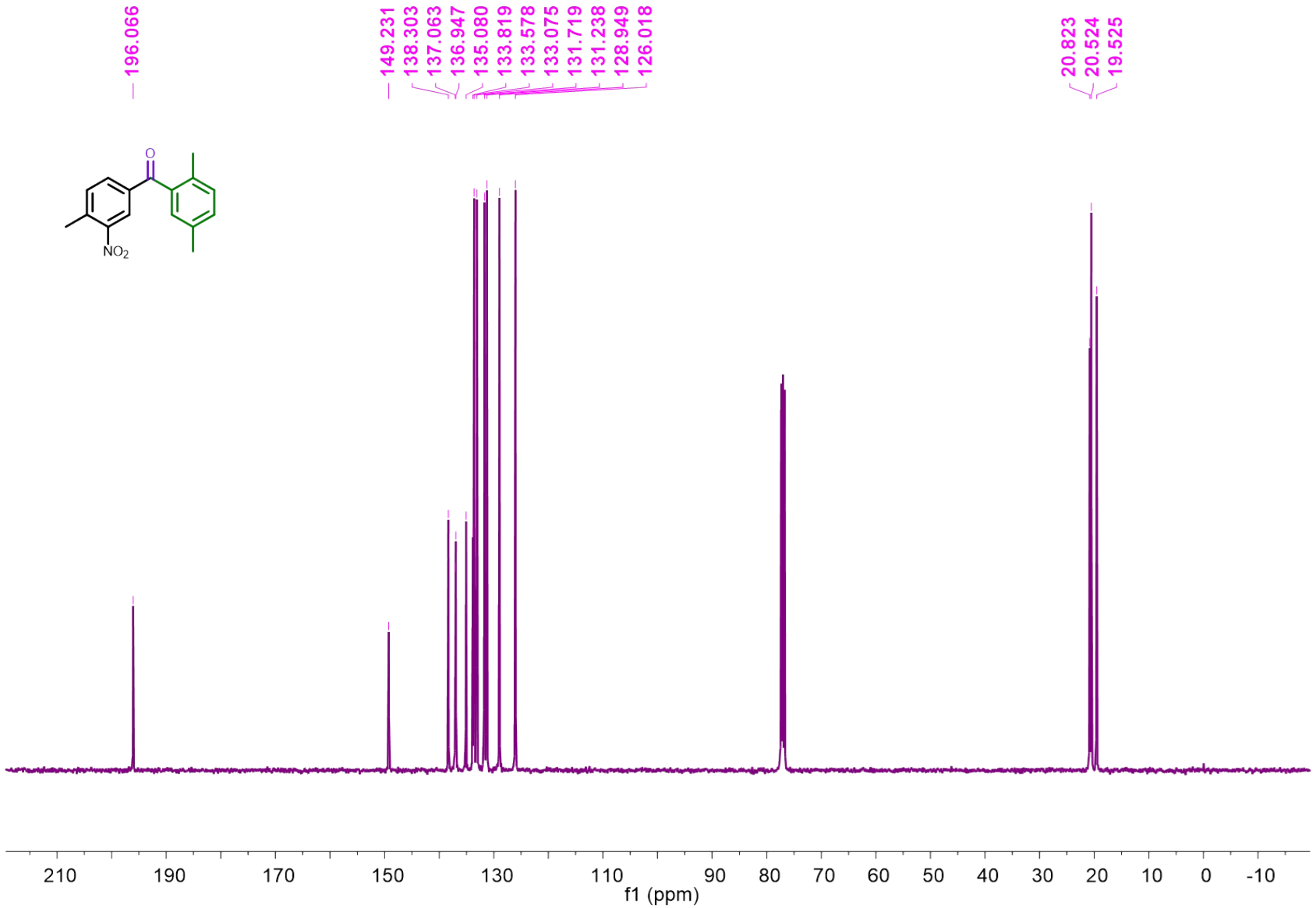


Figure S110. ^{13}C NMR spectra of (2,5-dimethylphenyl) (4-methyl-3-nitrophenyl) methanone **(2v)** in $CDCl_3$.

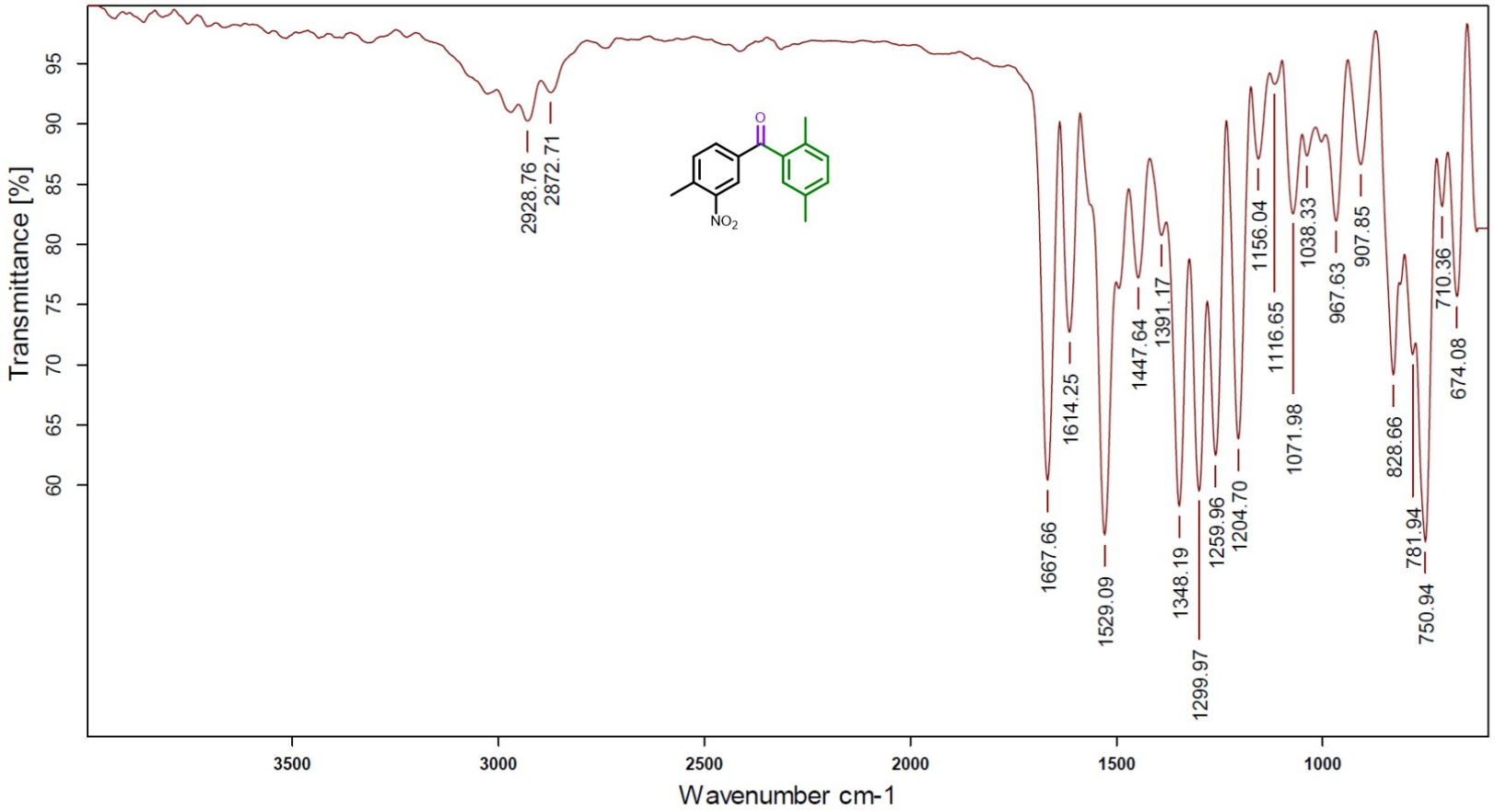
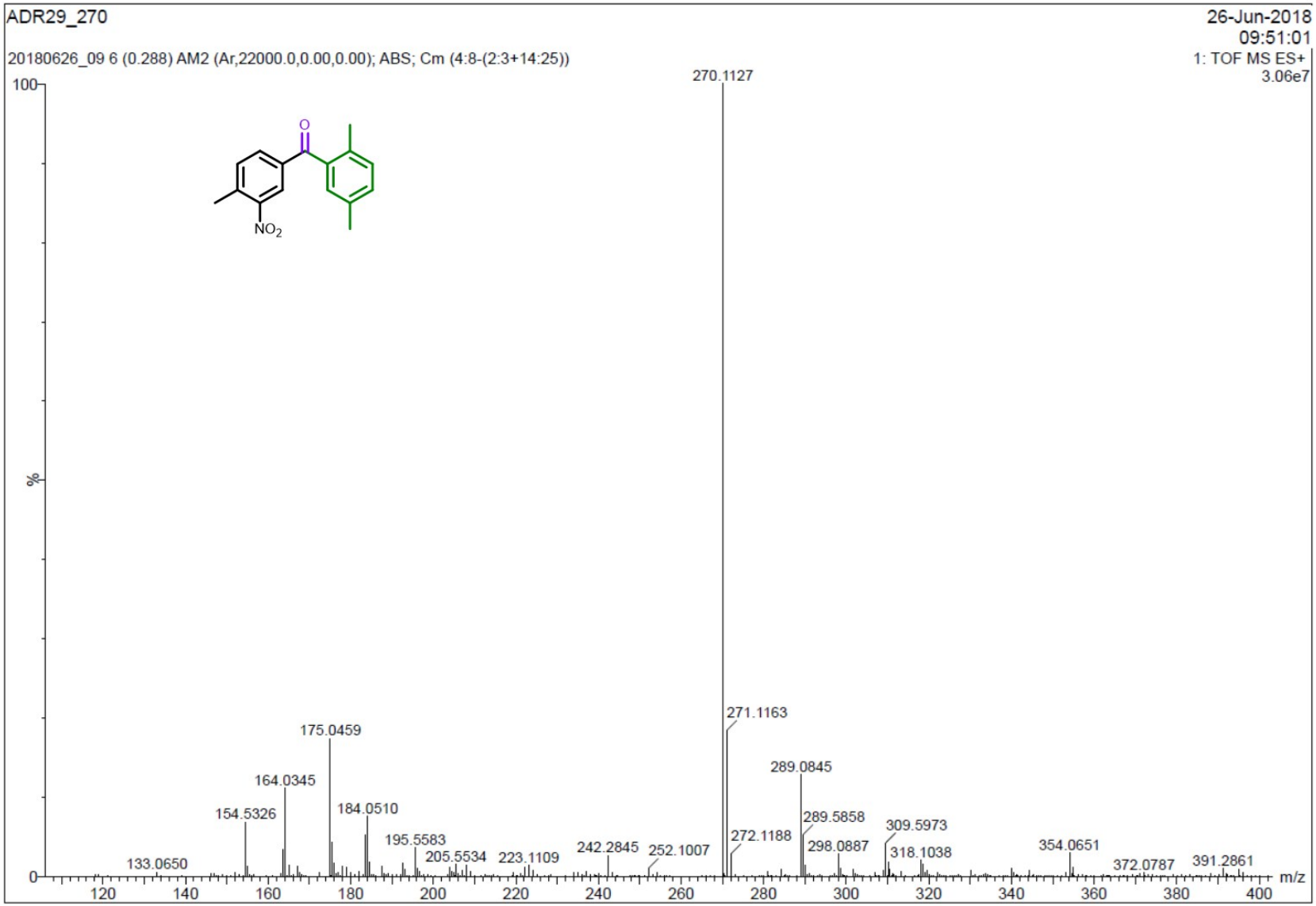


Figure S111. IR NMR spectra of (2,5-dimethylphenyl) (4-methyl-3-nitrophenyl) methanone **(2v)**.


ADR29_270
26-Jun-2018
09:51:01
20180626_09 6 (0.288) AM2 (Ar,22000.0,0.00,0.00); ABS; Cm (4:8-(2:3+14:25))
1: TOF MS ES+
3.06e7
100
%
0
O
NO2
270.1127
271.1163
175.0459
289.0845
164.0345
184.0510
154.5326
289.5858
309.5973
195.5583
272.1188
298.0887
318.1038
354.0651
205.5534
223.1109
242.2845
252.1007
133.0650
372.0787
391.2861
m/z
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400

Fi

gure S112. HRMS spectra of (2,5-dimethylphenyl) (4-methyl-3-nitrophenyl) methanone **(2v)**.

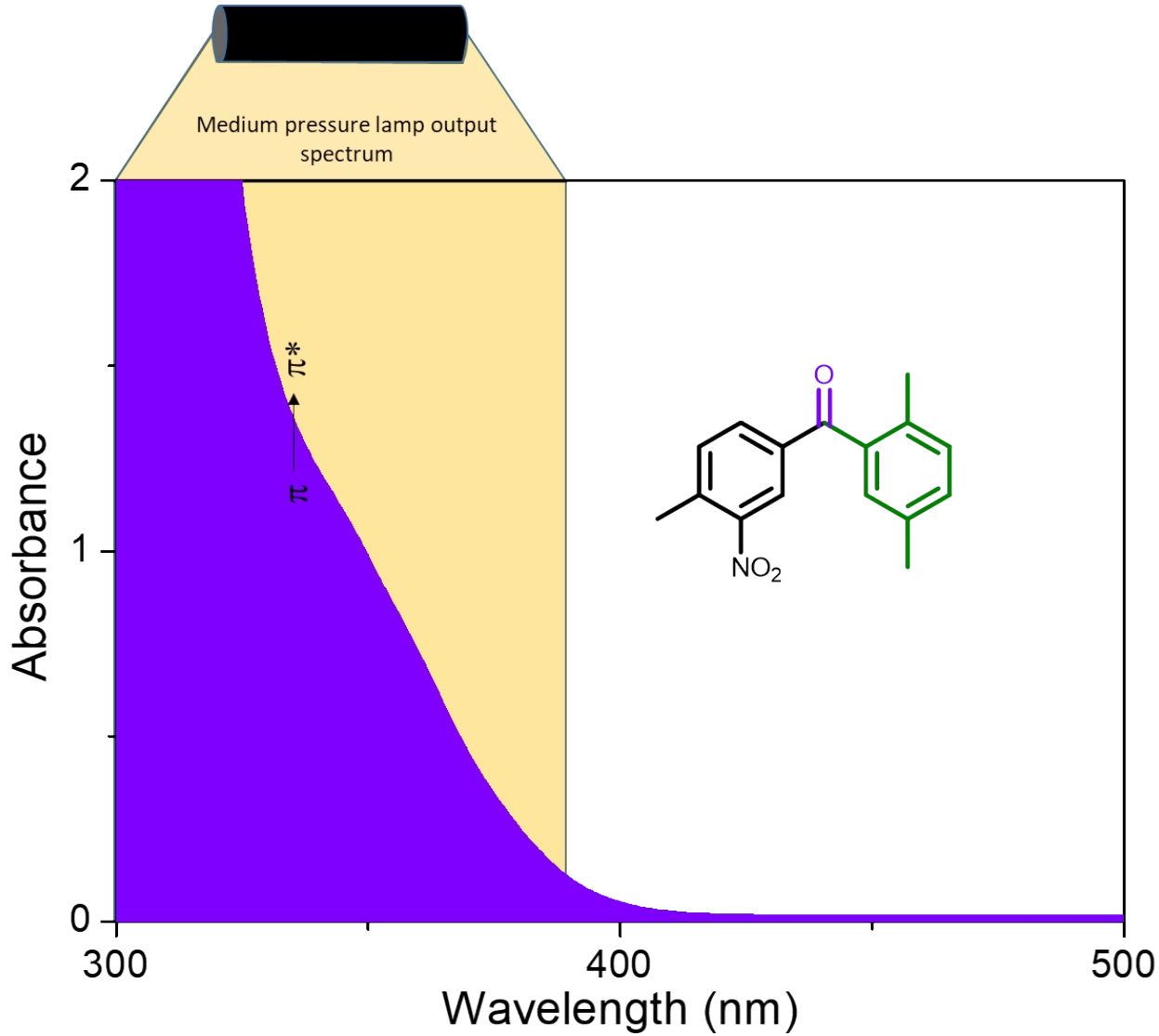


Figure S113. HRMS spectra of (2,5-dimethylphenyl) (4-methyl-3-nitrophenyl) methanone **(2v**) in 0.005M ACN.

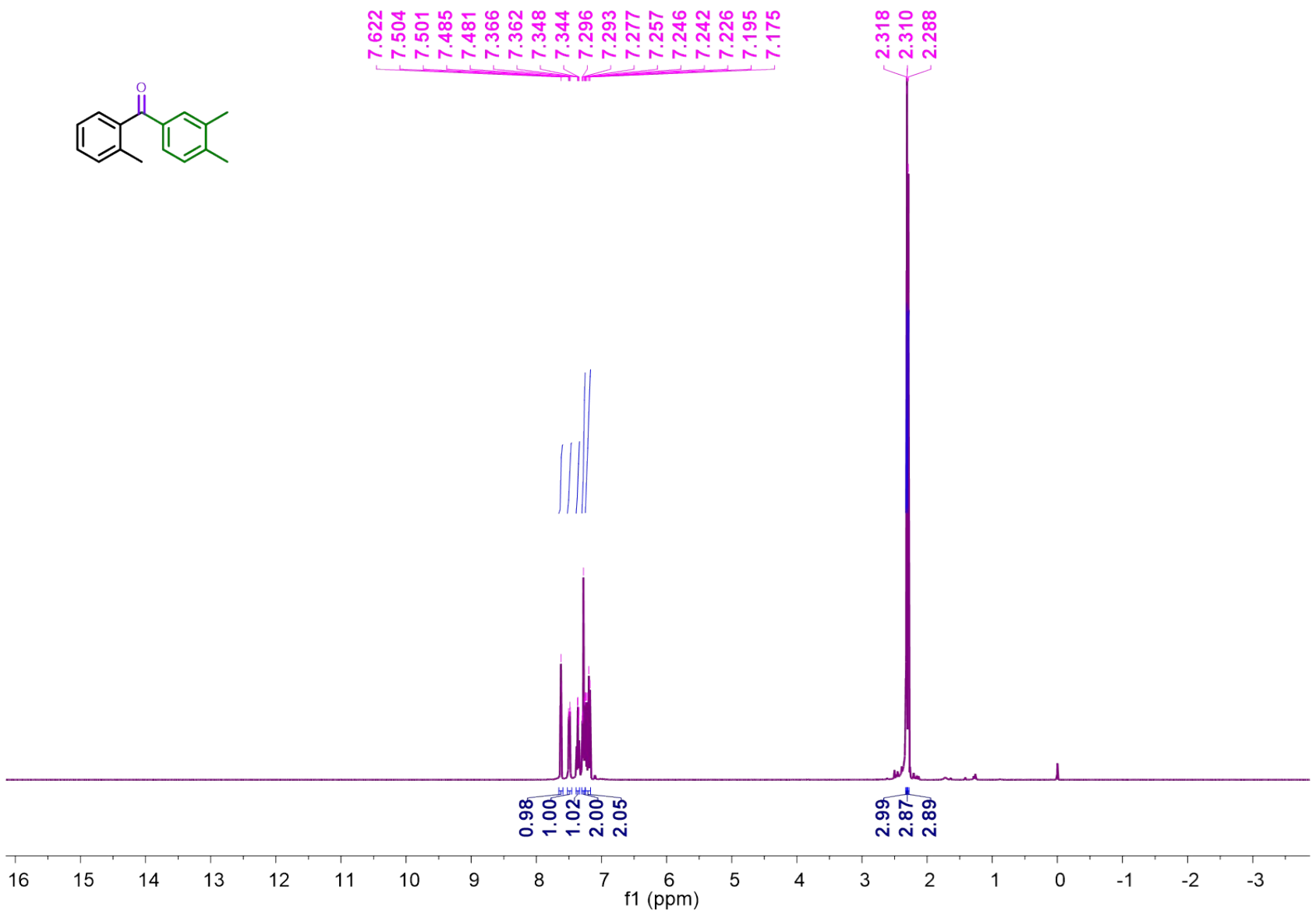


Figure S114. ^{1}H NMR spectra of (2-methylphenyl) (3,4-dimethylphenyl) methanone **(2w)** in $CDCl_3$.

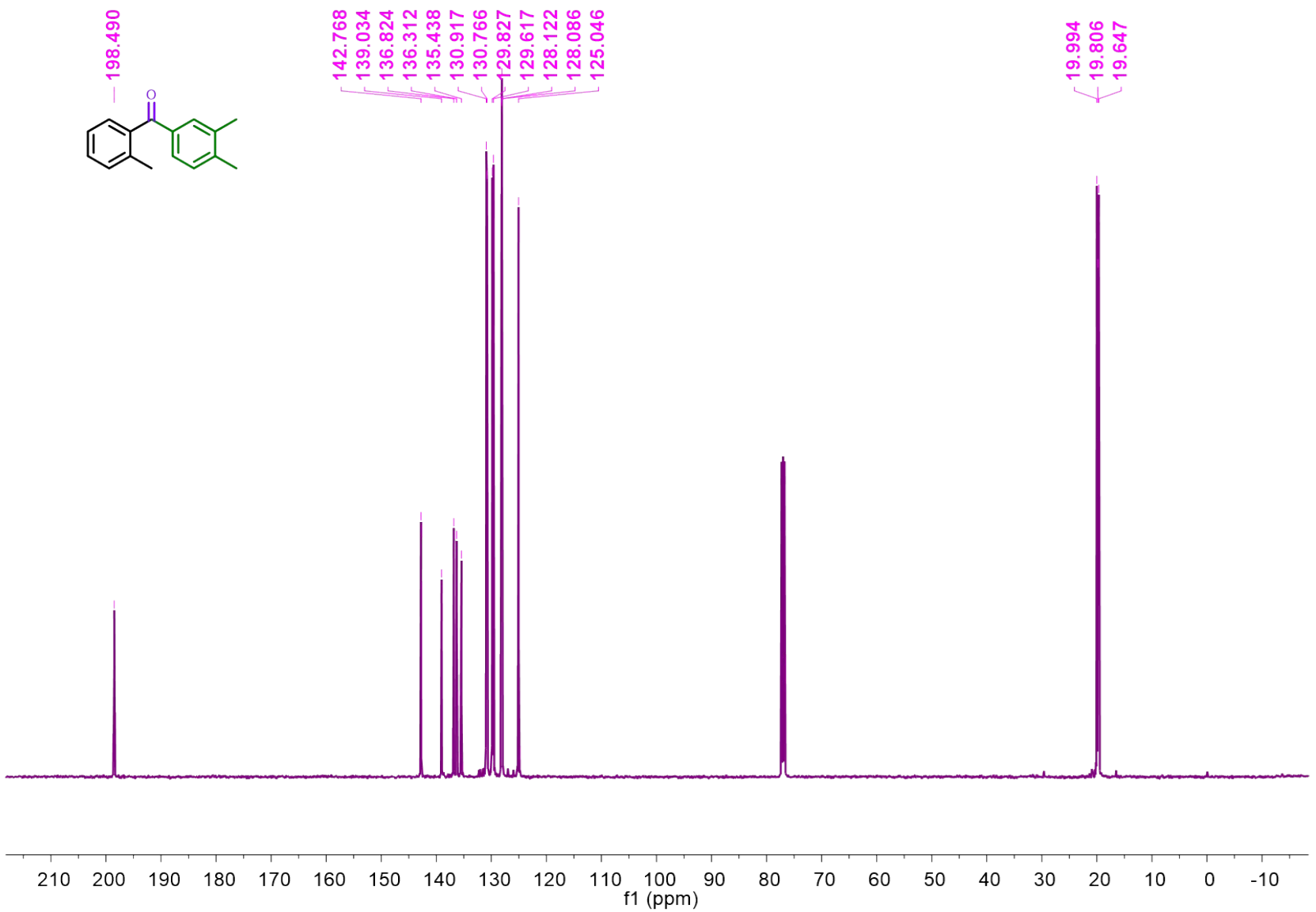


Figure S115. ^{13}C NMR spectra of (2-methylphenyl) (3,4-dimethylphenyl) methanone **(2w)** in $CDCl_3$.

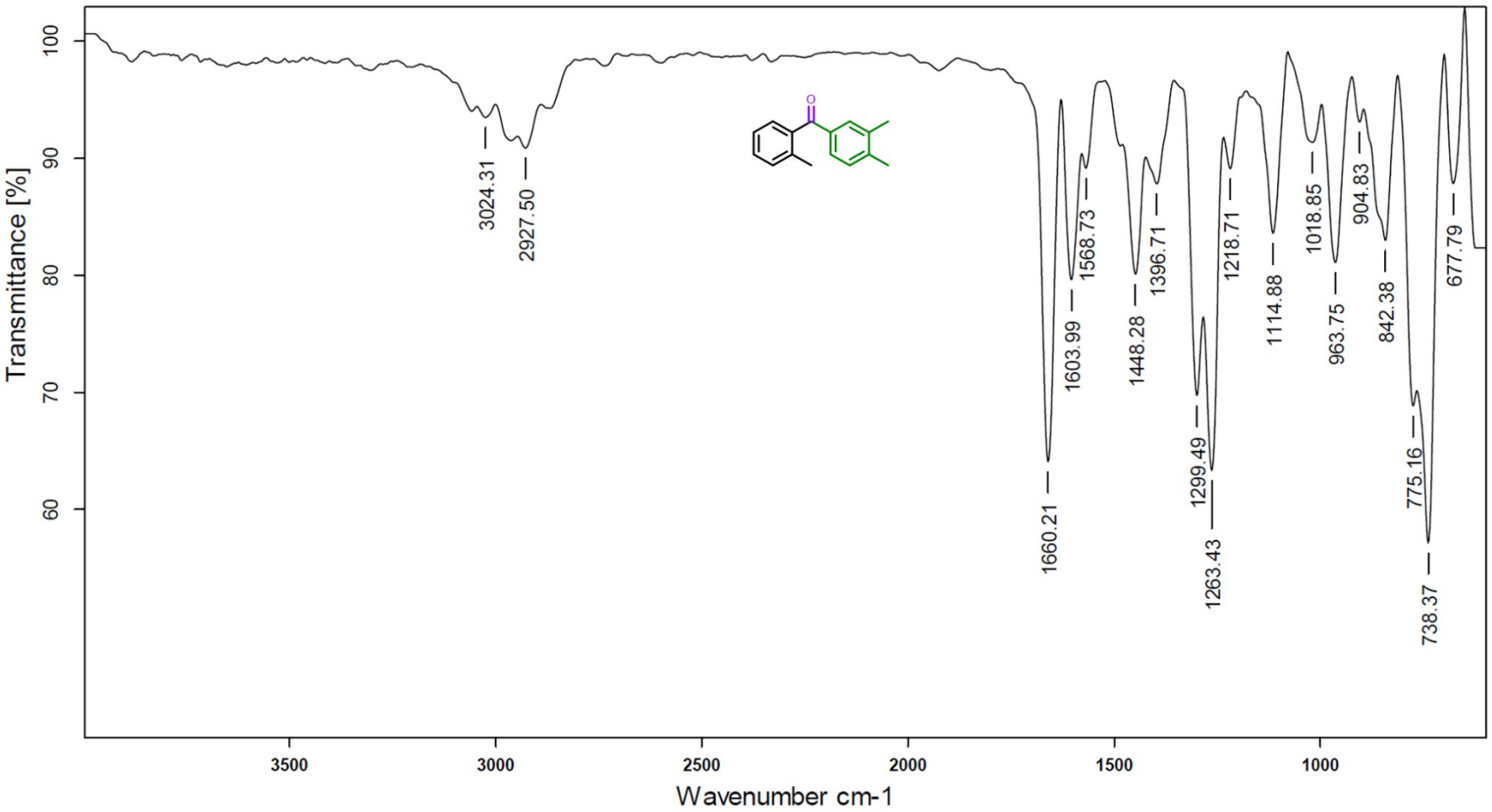


Figure S116. IR spectra of (2-methylphenyl) (3,4-dimethylphenyl) methanone **(2w**).

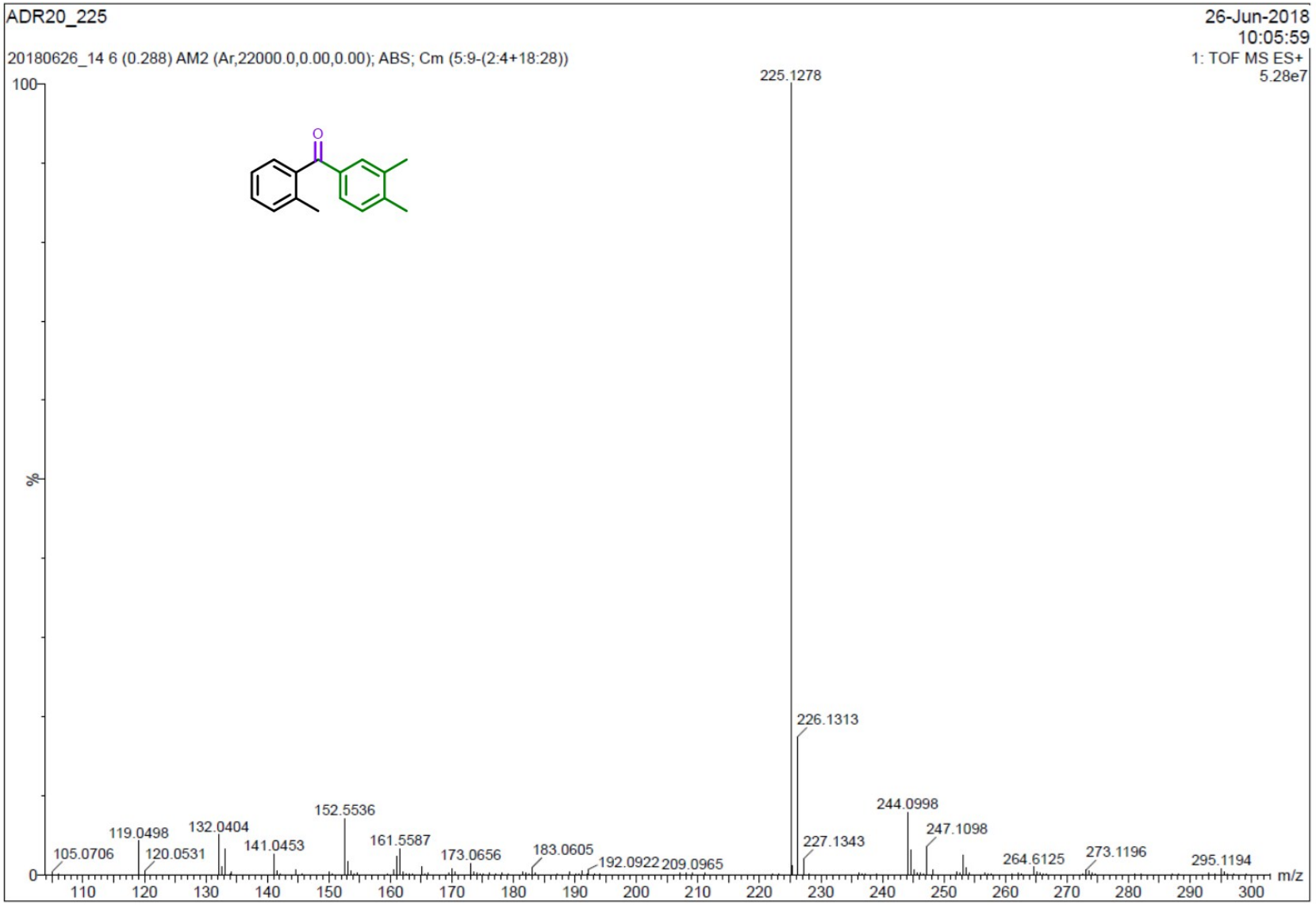


Figure S117. HRMS spectra of (2-methylphenyl) (3,4-dimethylphenyl) methanone **(2w)**.

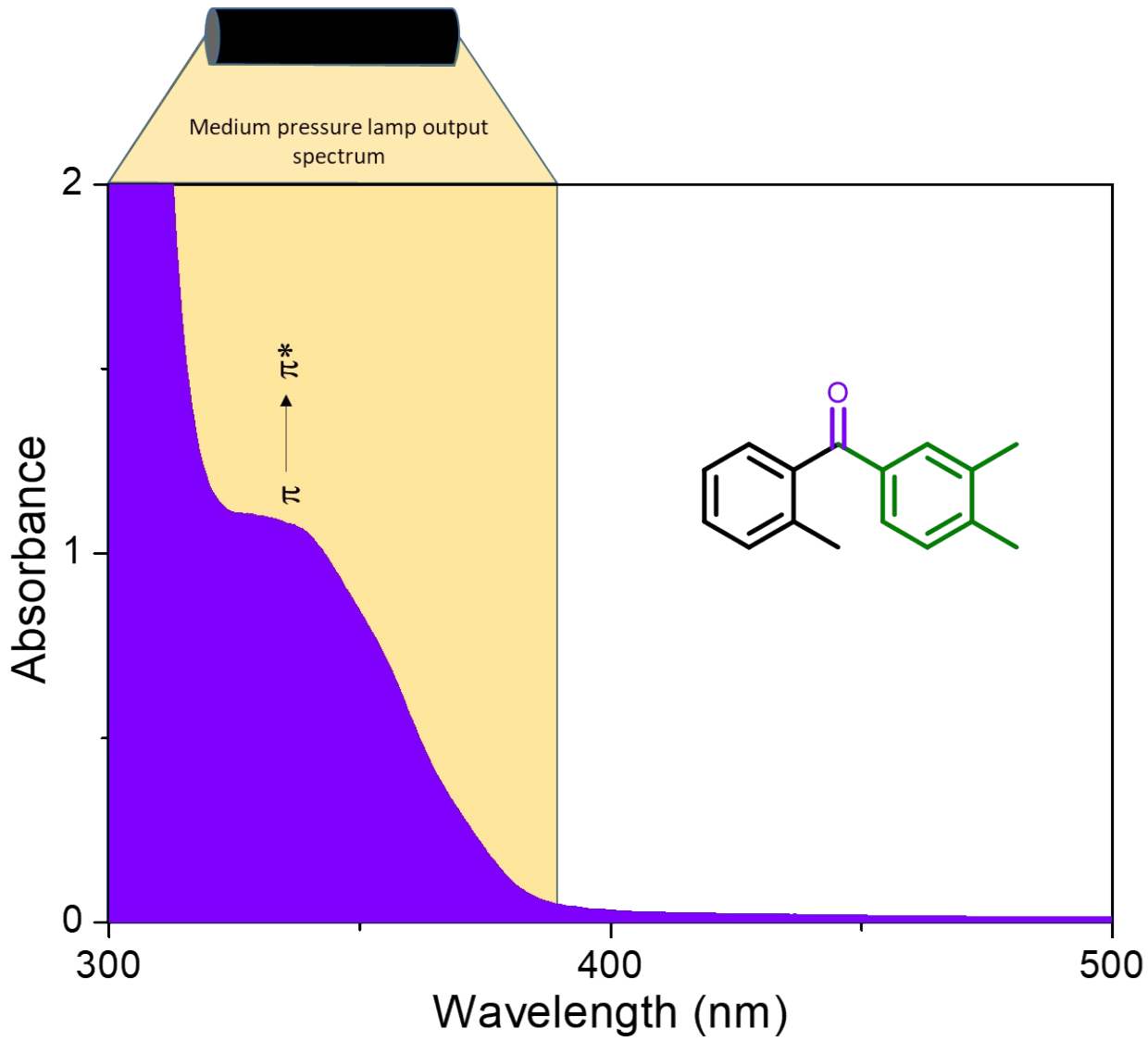


Figure S118. HRMS spectra of (2-methylphenyl) (3,4-dimethylphenyl) methanone **(2w**) in 0.005M ACN.

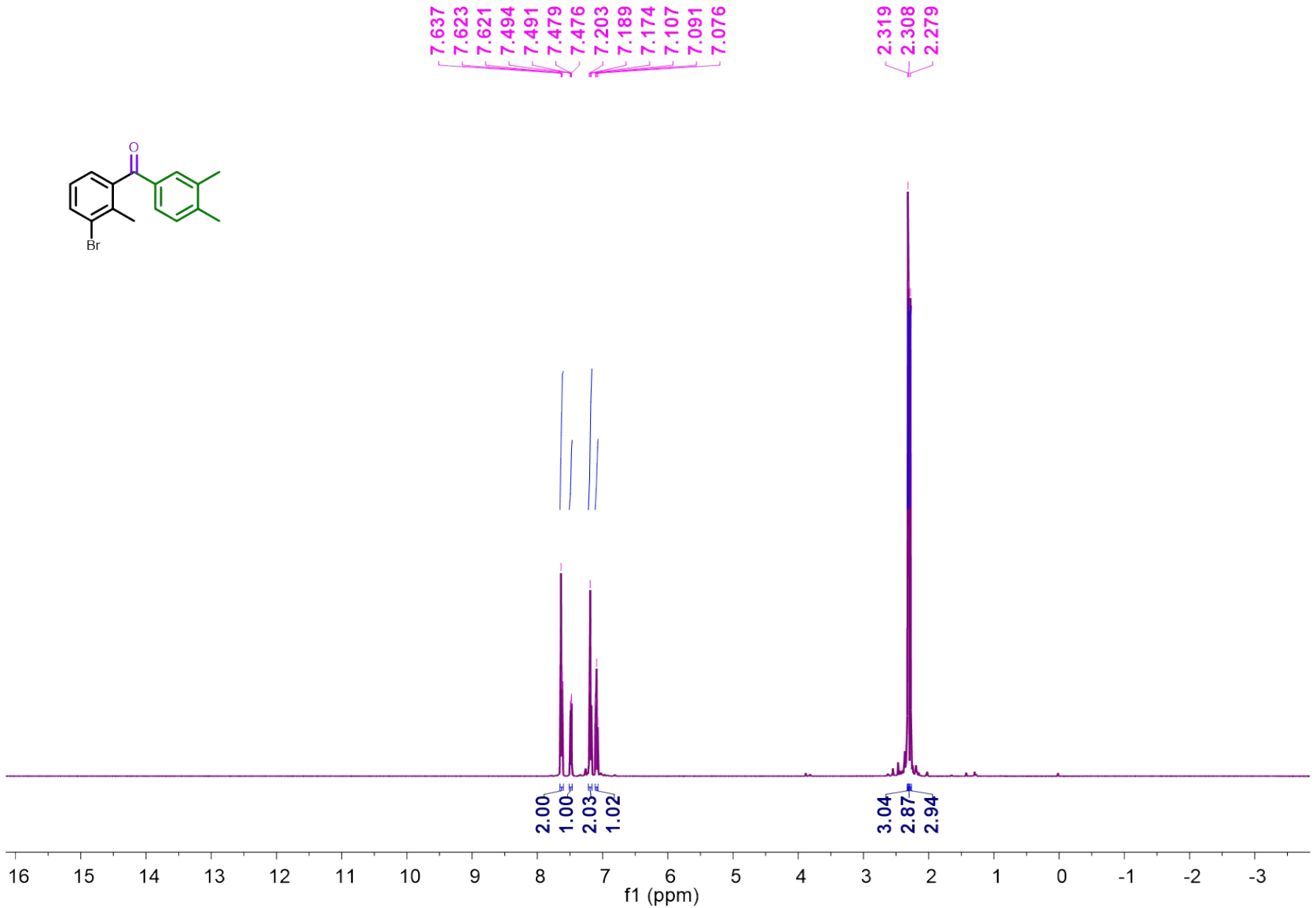


Figure S119. ^{1}H NMR spectra of (3-bromo-2-methylphenyl) (3,4-dimethylphenyl) methanone **(2x)** in $CDCl_3$.

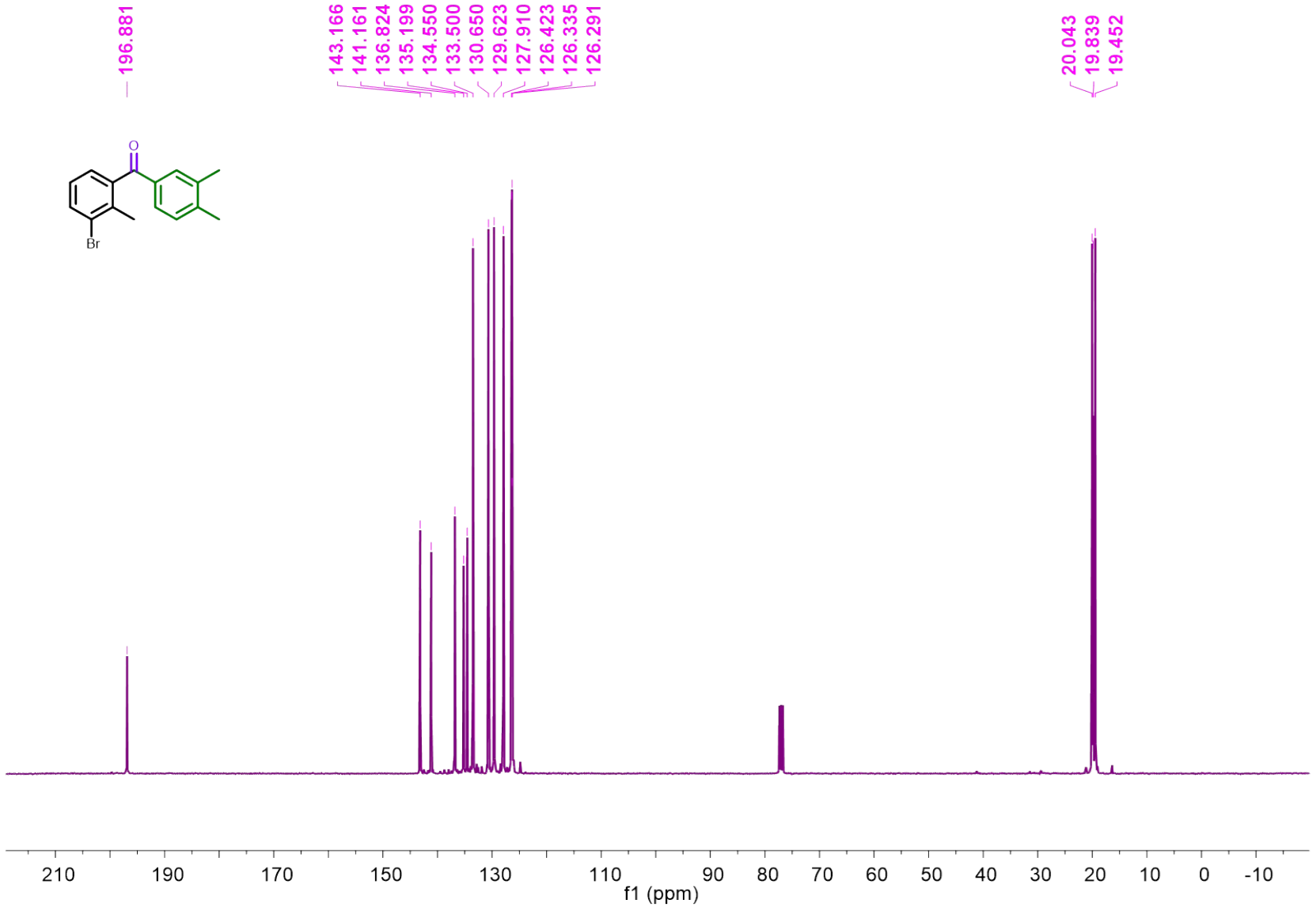


Figure S120. ^{13}C NMR spectra of (3-bromo-2-methylphenyl) (3,4-dimethylphenyl) methanone **(2x)** in $CDCl_3$.

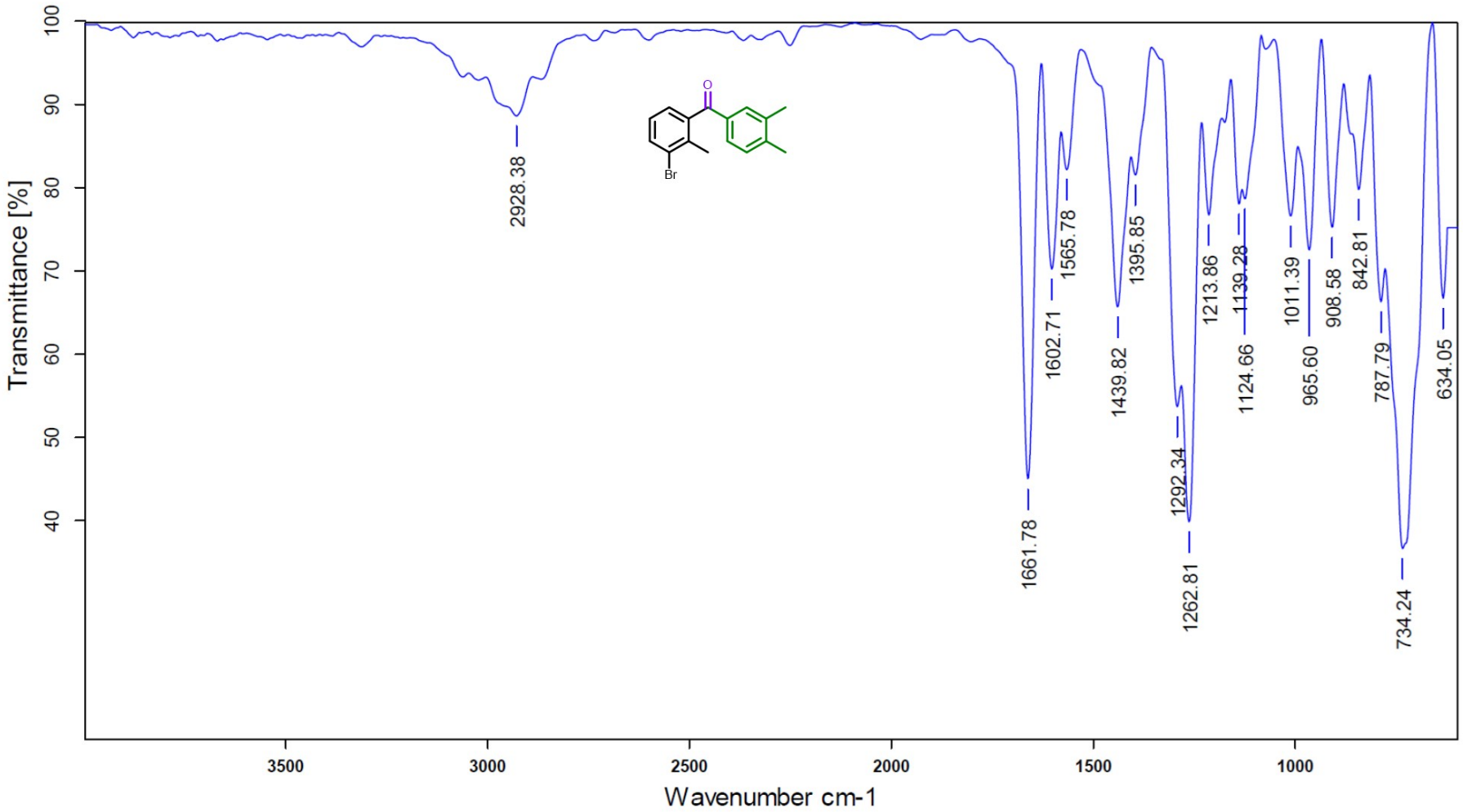


Figure 121. IR spectra of (3-bromo-2-methylphenyl) (3,4-dimethylphenyl) methanone **(2x**).

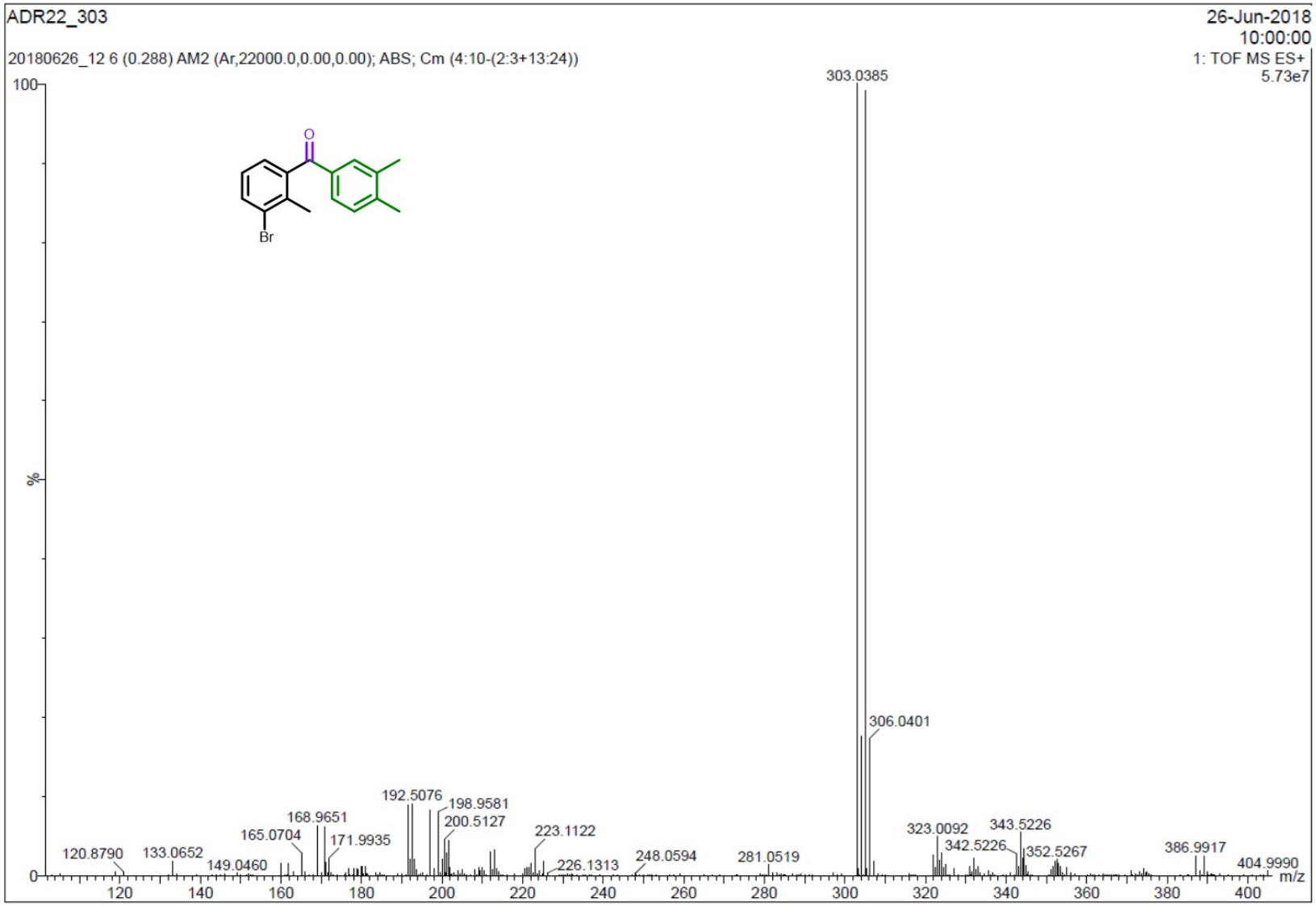


Figure S122. HRMS spectra of (3-bromo-2-methylphenyl) (3,4-dimethylphenyl) methanone **(2x**).

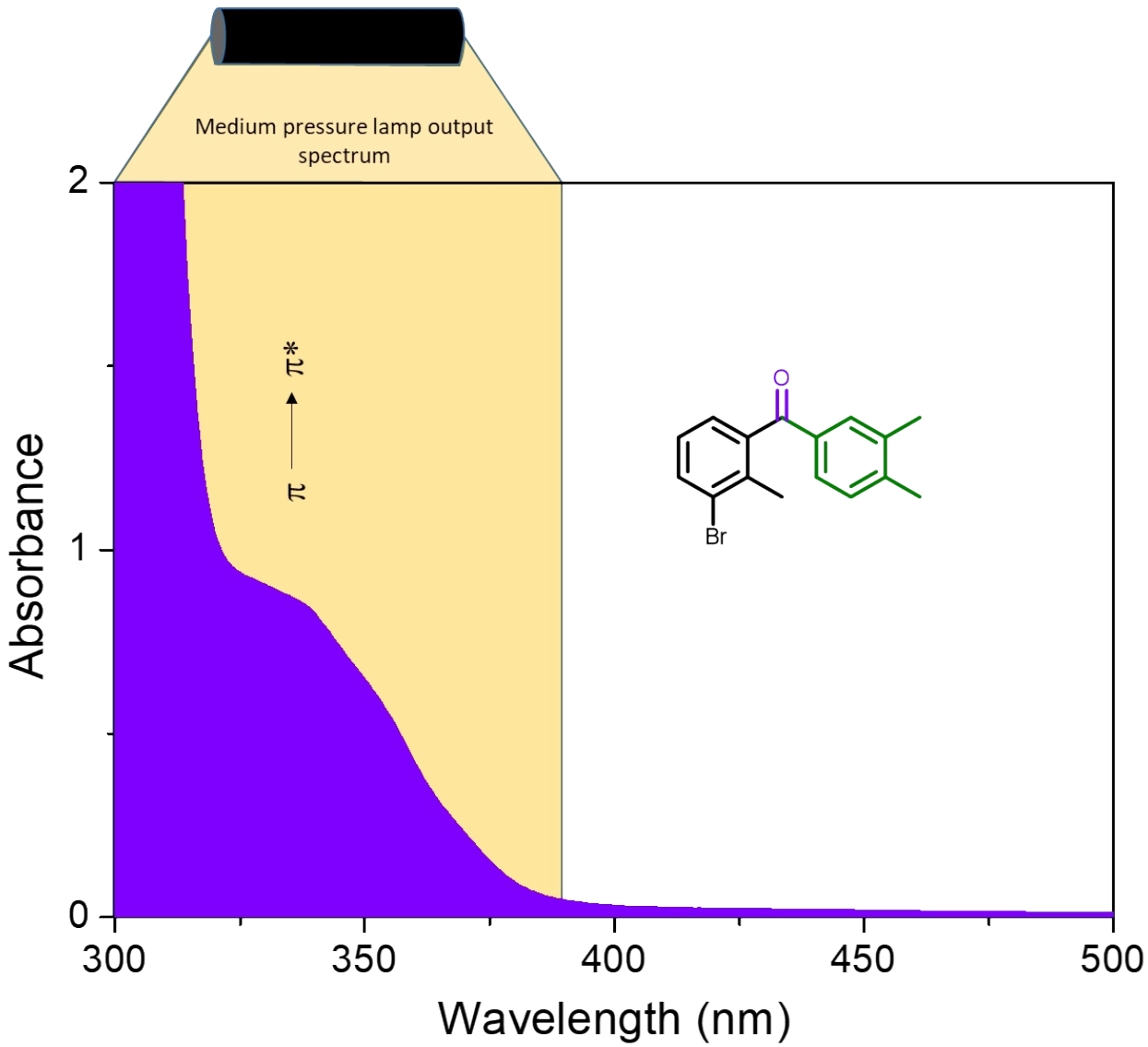


Figure S123. HRMS spectra of (3-bromo-2-methylphenyl) (3,4-dimethylphenyl) methanone **(2x)** in 0.005M ACN.

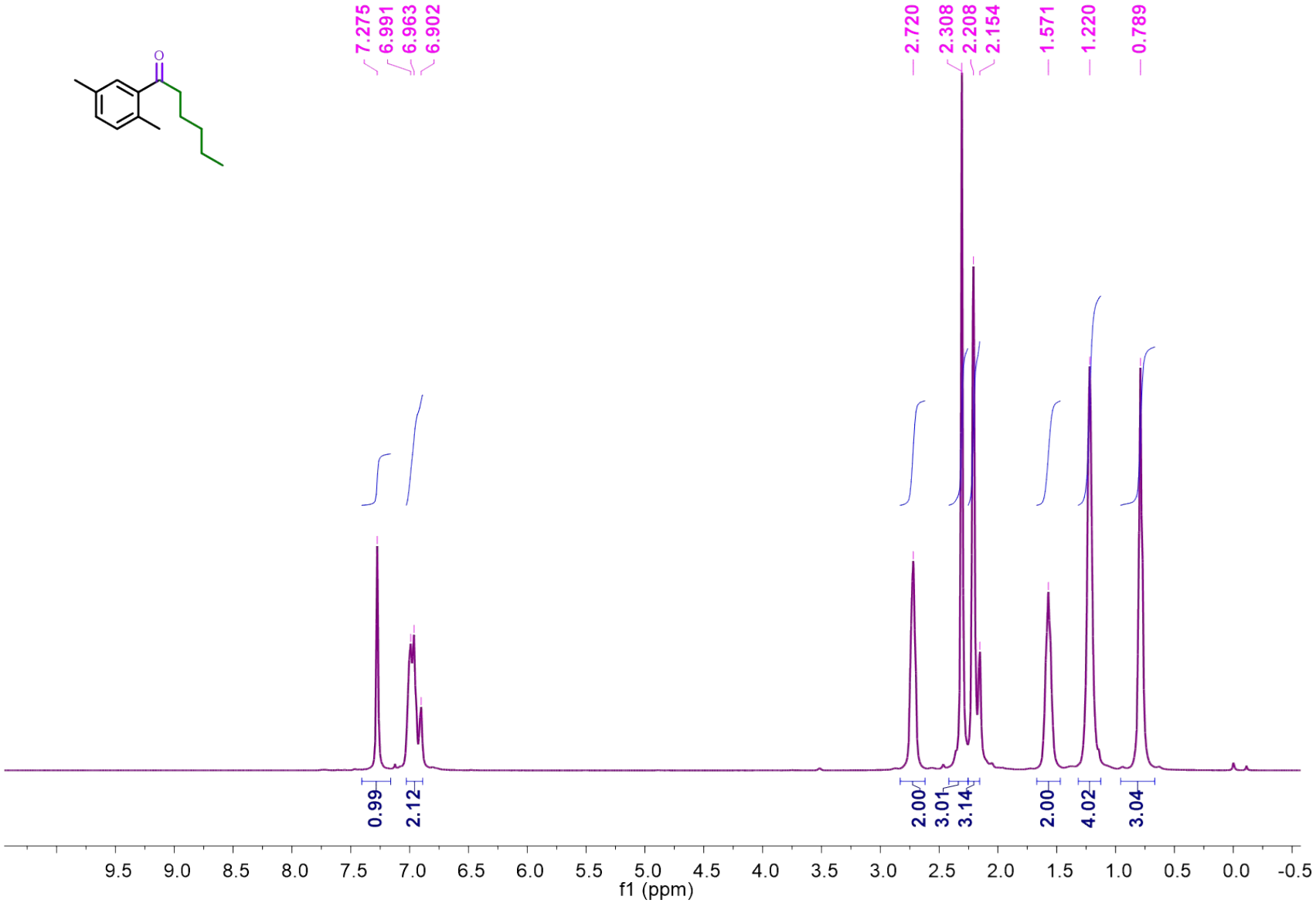


Figure S124. ^{1}H NMR spectra of 1-(2,5-dimethylphenyl) hexan-1-one **(2y)** in $CDCl_3$.

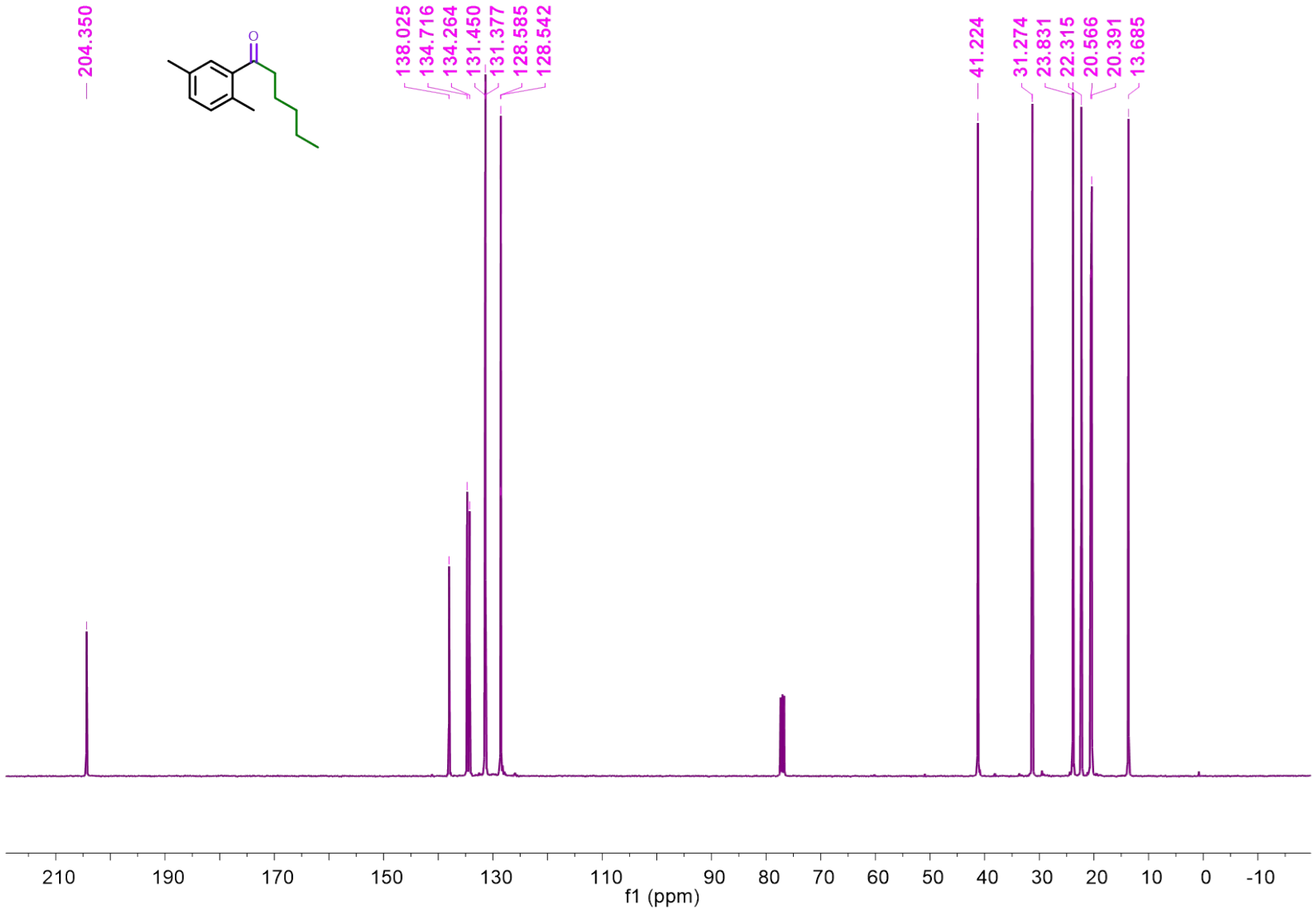


Figure S125. ^{13}C NMR spectra of 1-(2,5-dimethylphenyl) hexan-1-one **(2y)** in $CDCl_3$.

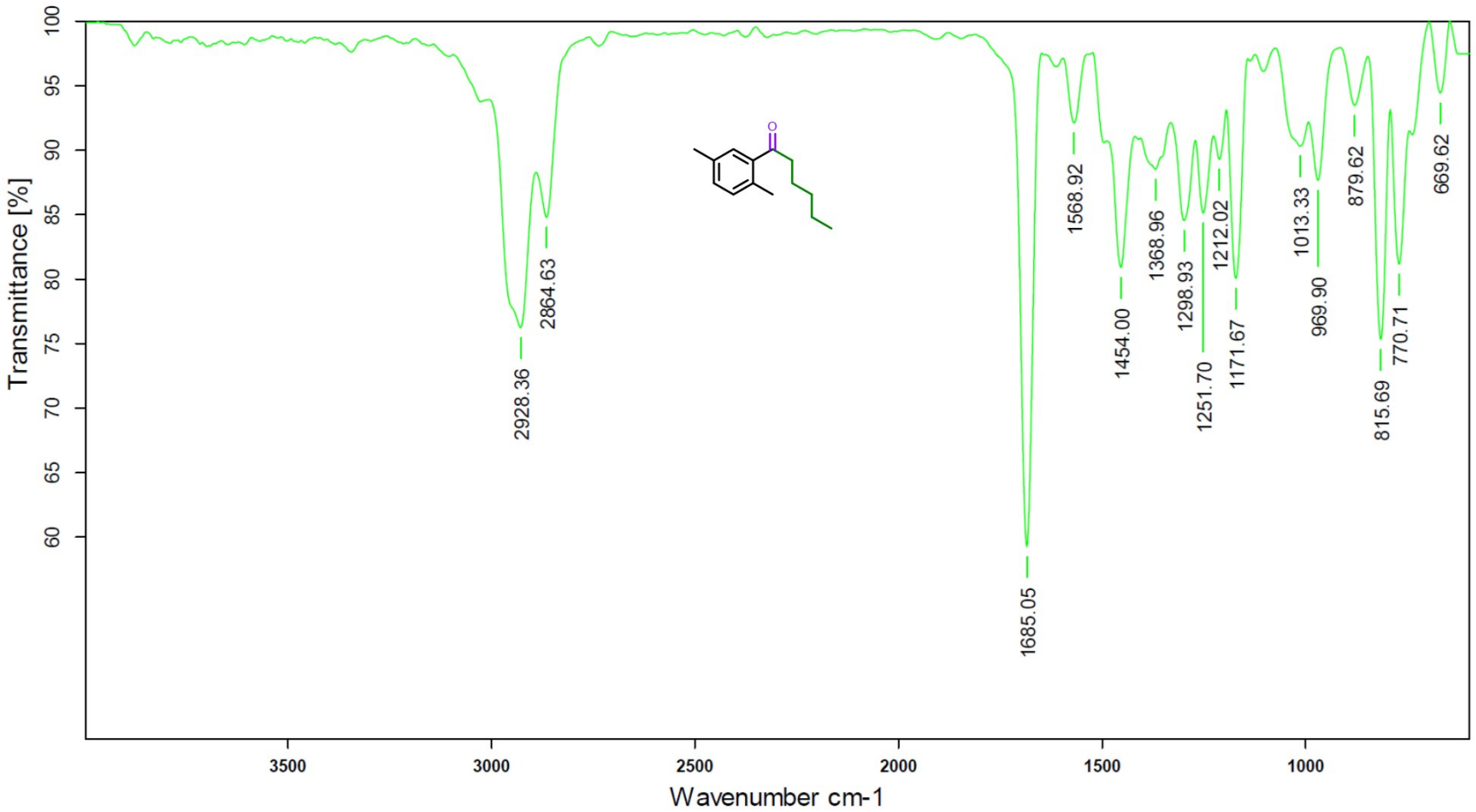


Figure S126. IR spectra of 1-(2,5-dimethylphenyl) hexan-1-one **(2y**).

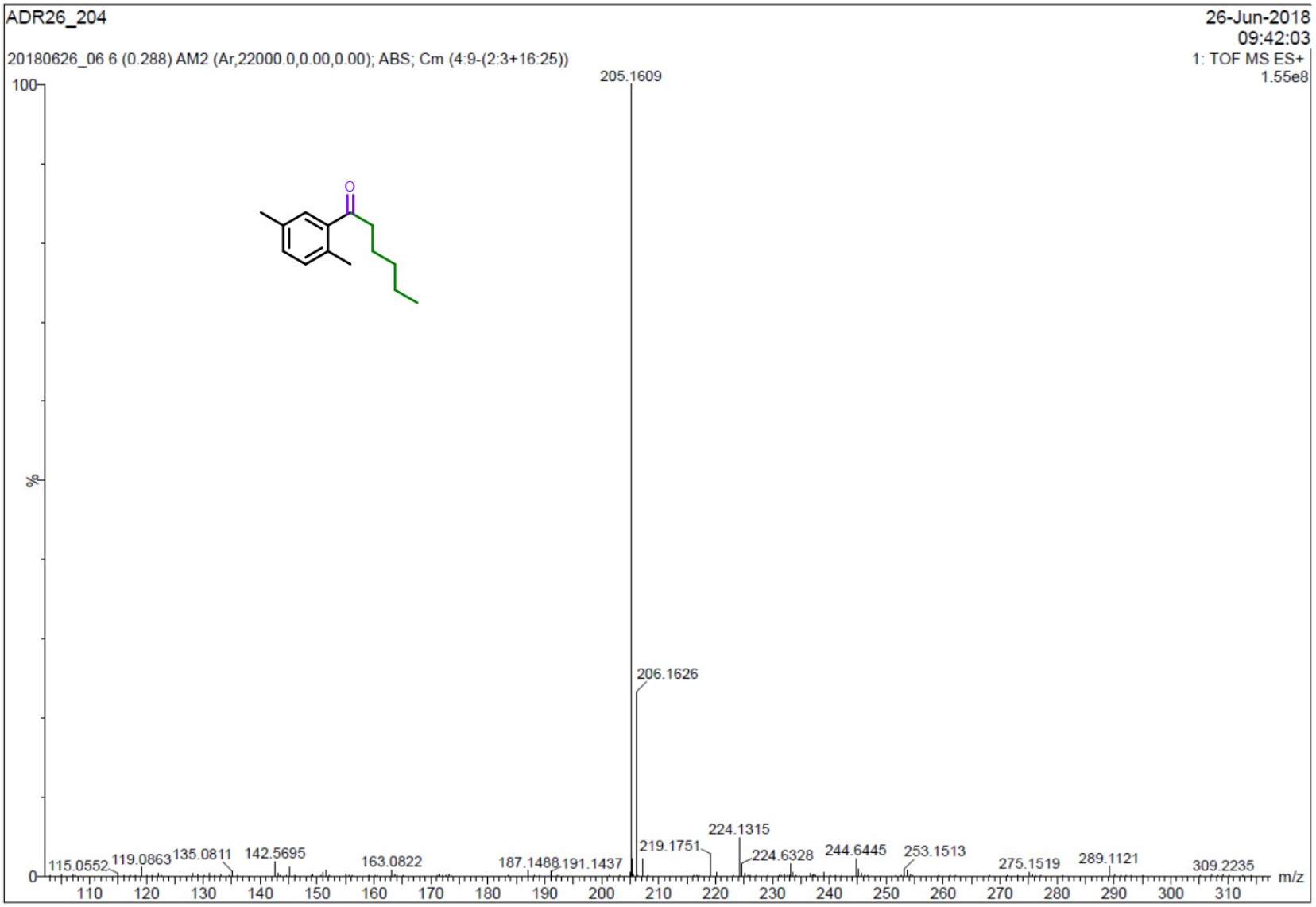


Figure S127. HRMS spectra of 1-(2,5-dimethylphenyl) hexan-1-one **(2y)**.

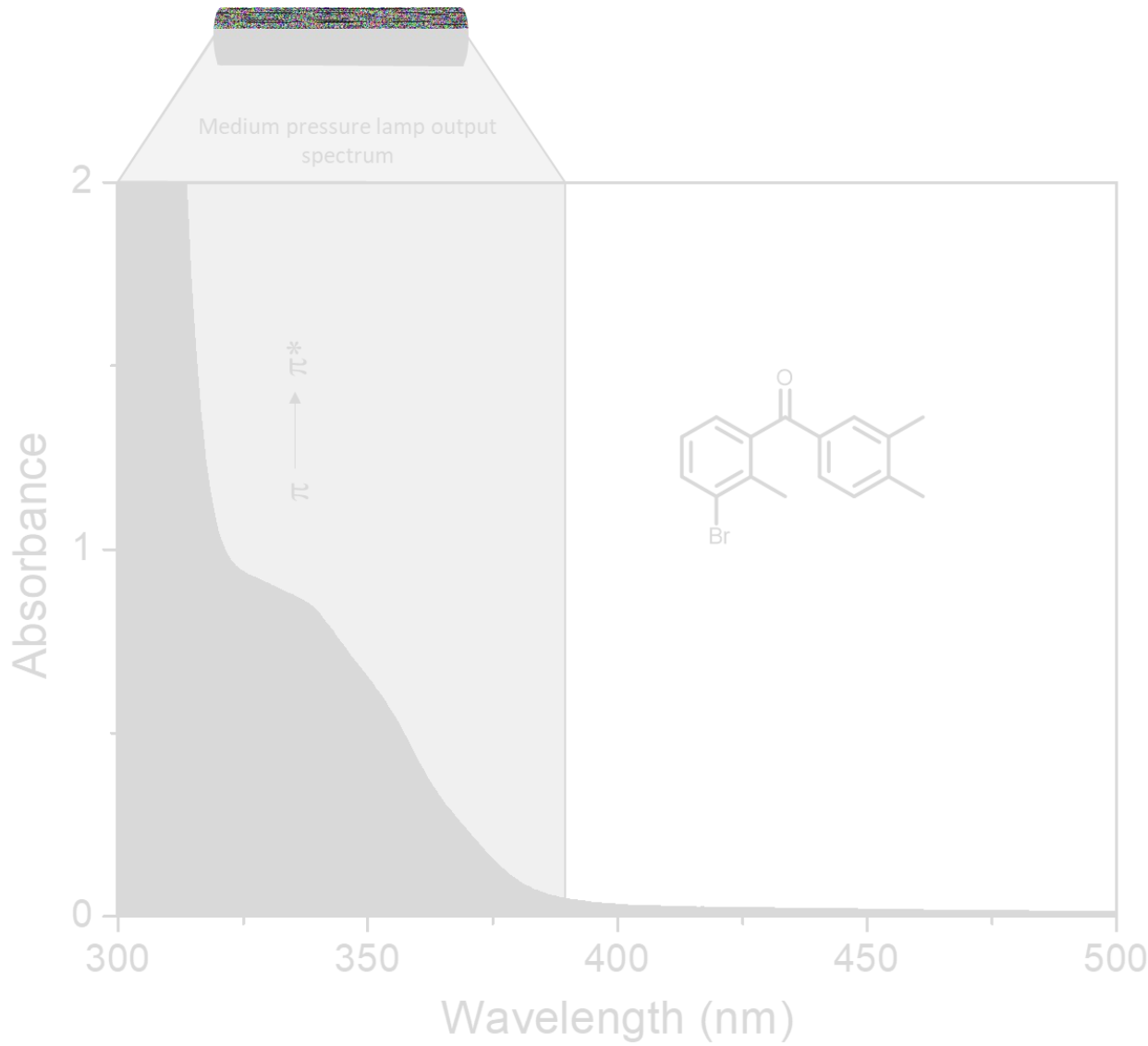
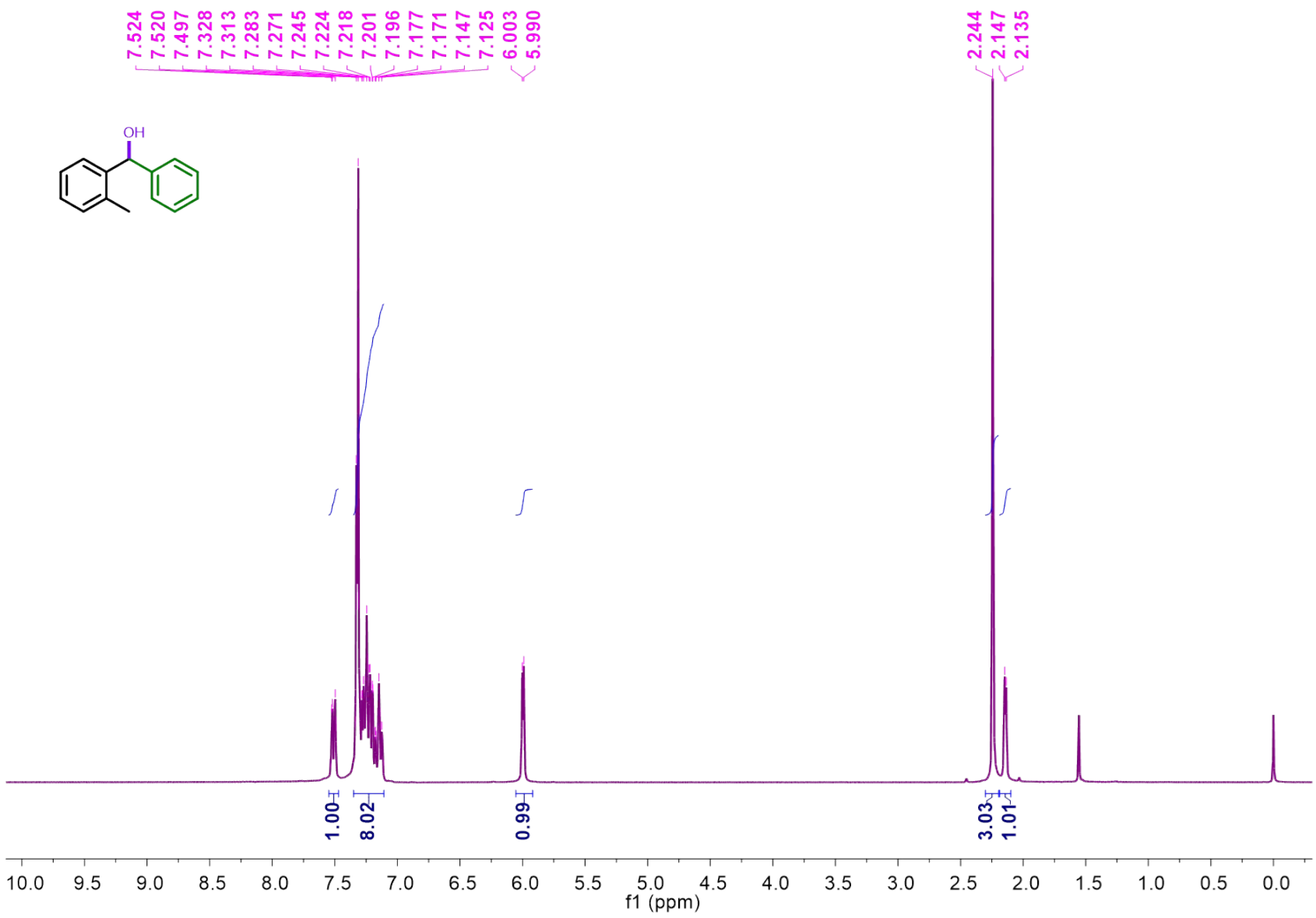


Figure S128. HRMS spectra of 1-(2,5-dimethylphenyl) hexan-1-one **(2y**) in 0.005M ACN.


7.524
7.520
7.497
7.328
7.313
7.283
7.271
7.245
7.224
7.218
7.201
7.196
7.177
7.171
7.147
7.125
6.003
5.990
2.244
2.147
2.135
OH
1.00
8.02
0.99
3.03
1.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

F

igure S129. ^{1}H NMR spectra of phenyl(o-tolyl) methanol **(3a)** in $CDCl_3$.

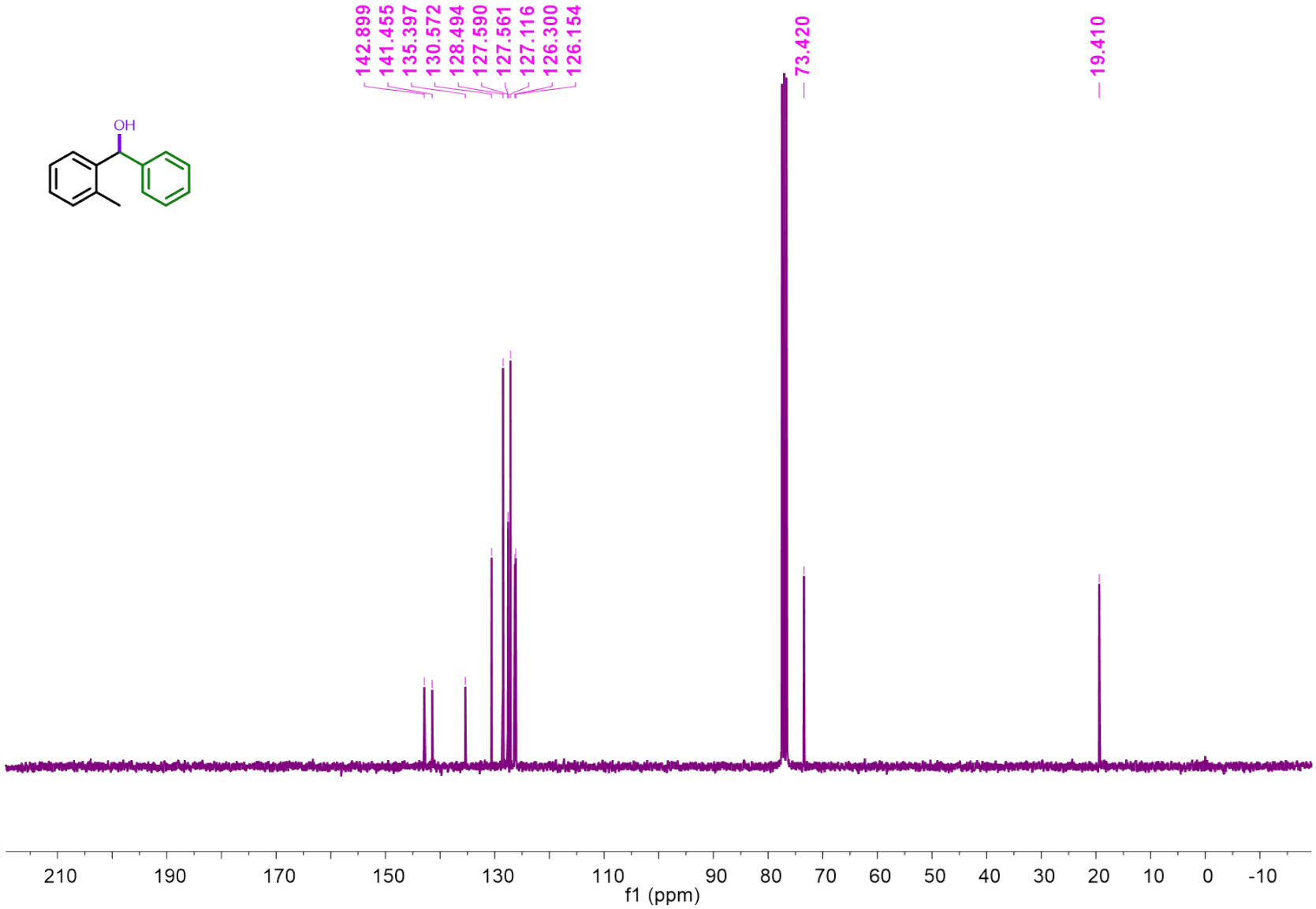


Figure S130. ^{13}C NMR spectra of phenyl(o-tolyl) methanol **(3a)** in $CDCl_3$.

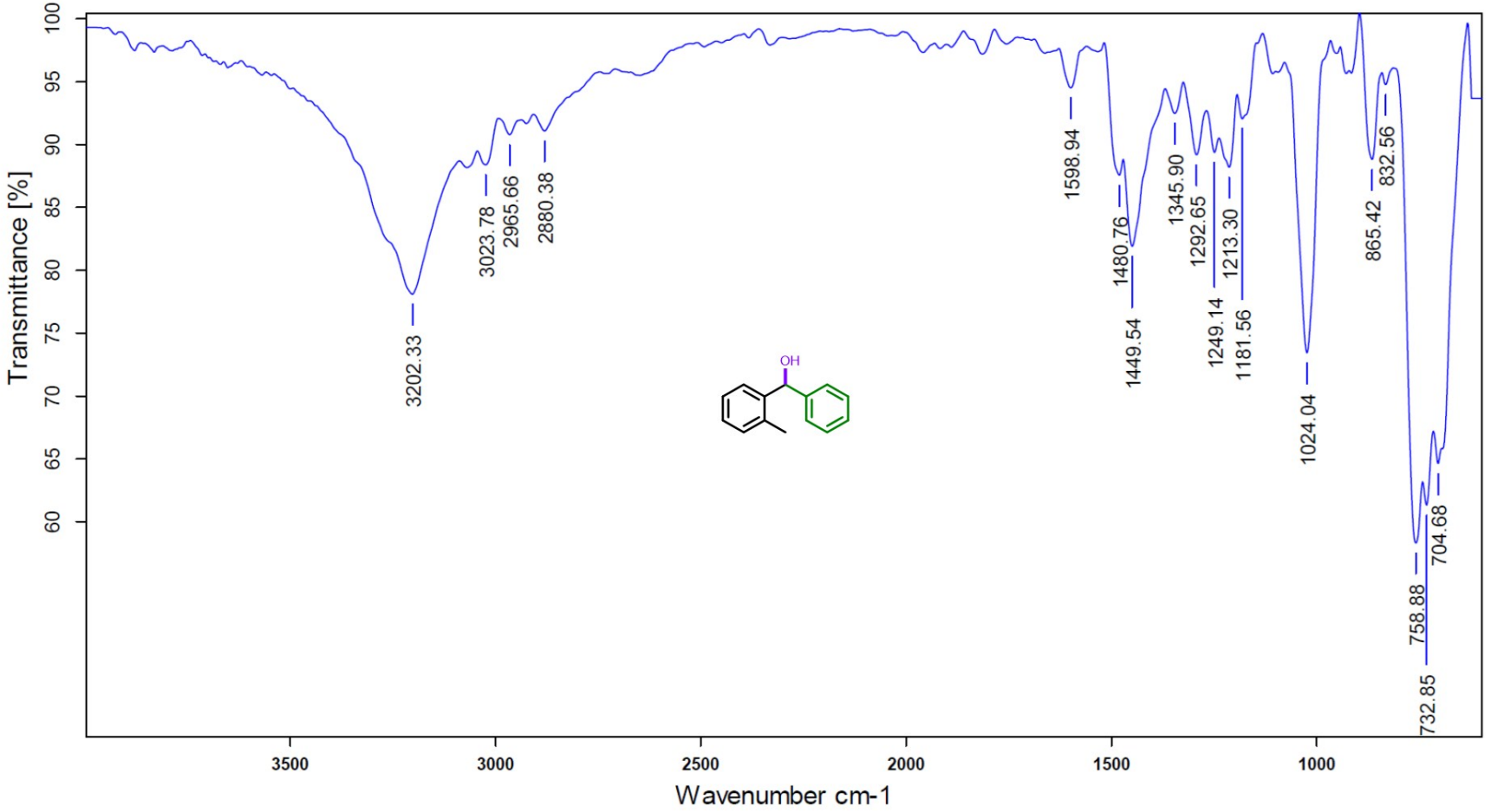


Figure S131. IR spectra of phenyl(o-tolyl) methanol **(3a)**.

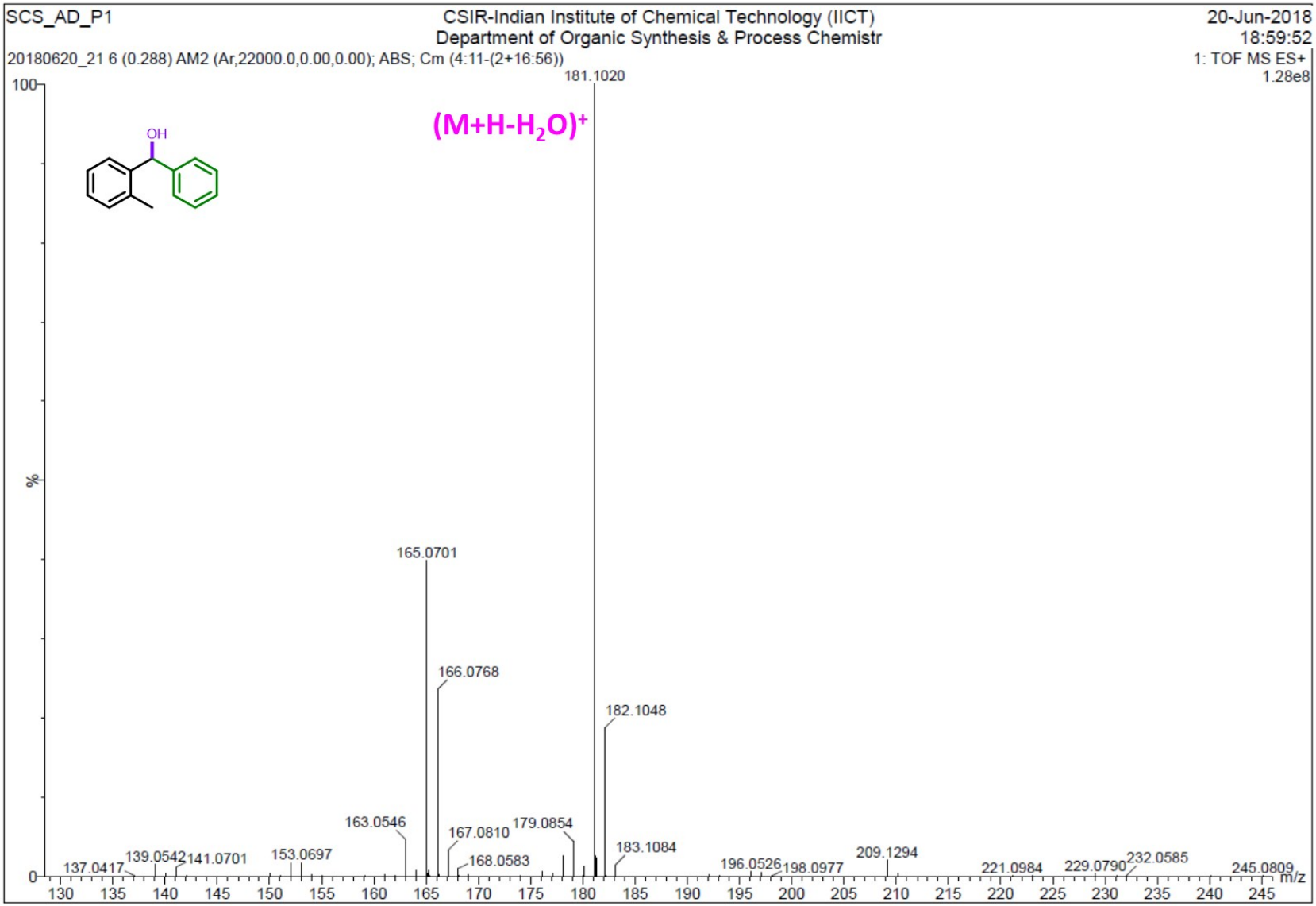


Figure S132. HRMS spectra of phenyl(o-tolyl) methanol **(3a)**.

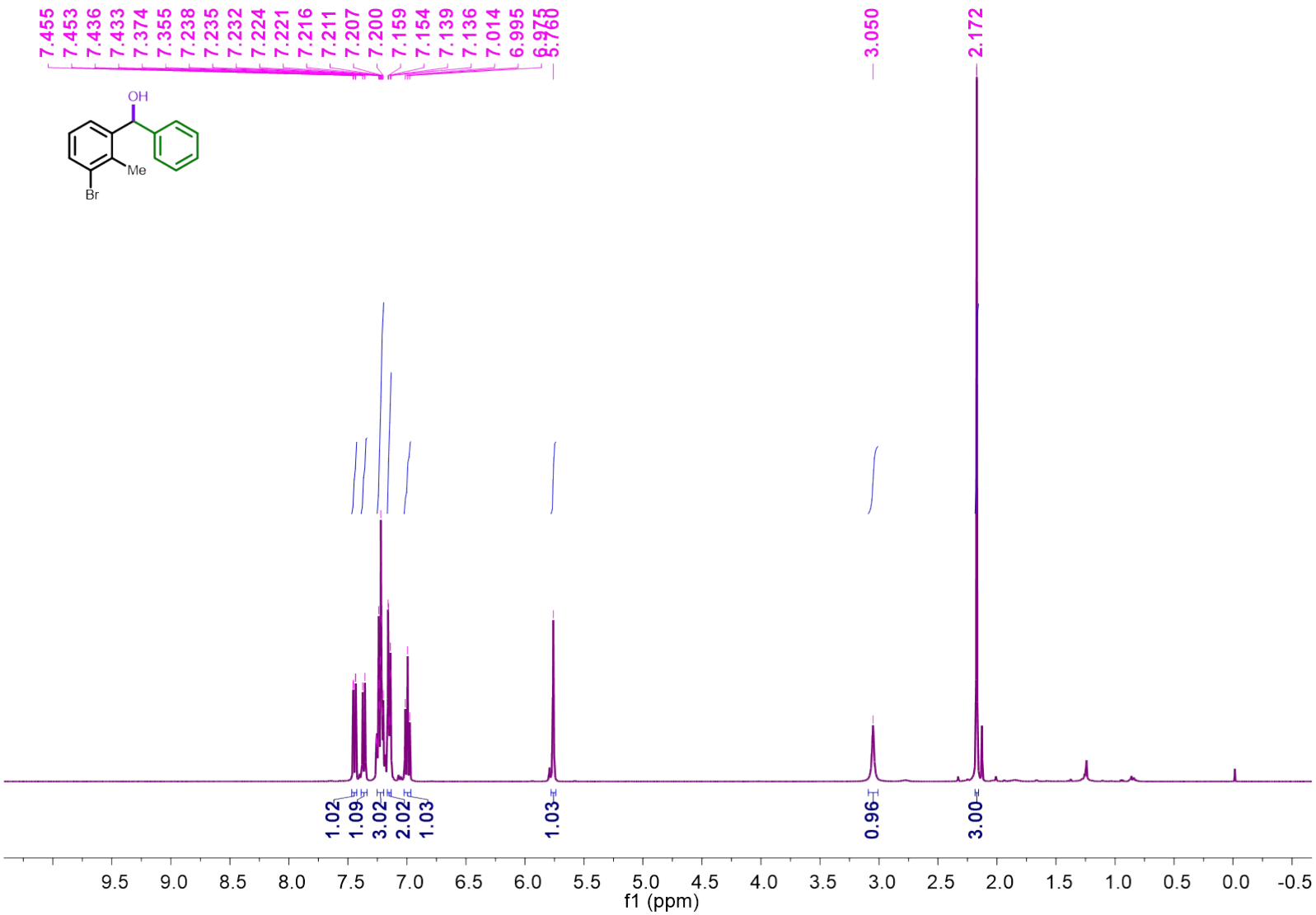


Figure S133. ^{1}H NMR spectra of (3-bromo-2-methylphenyl) (phenyl) methanol **(3b)** in $CDCl_3$.

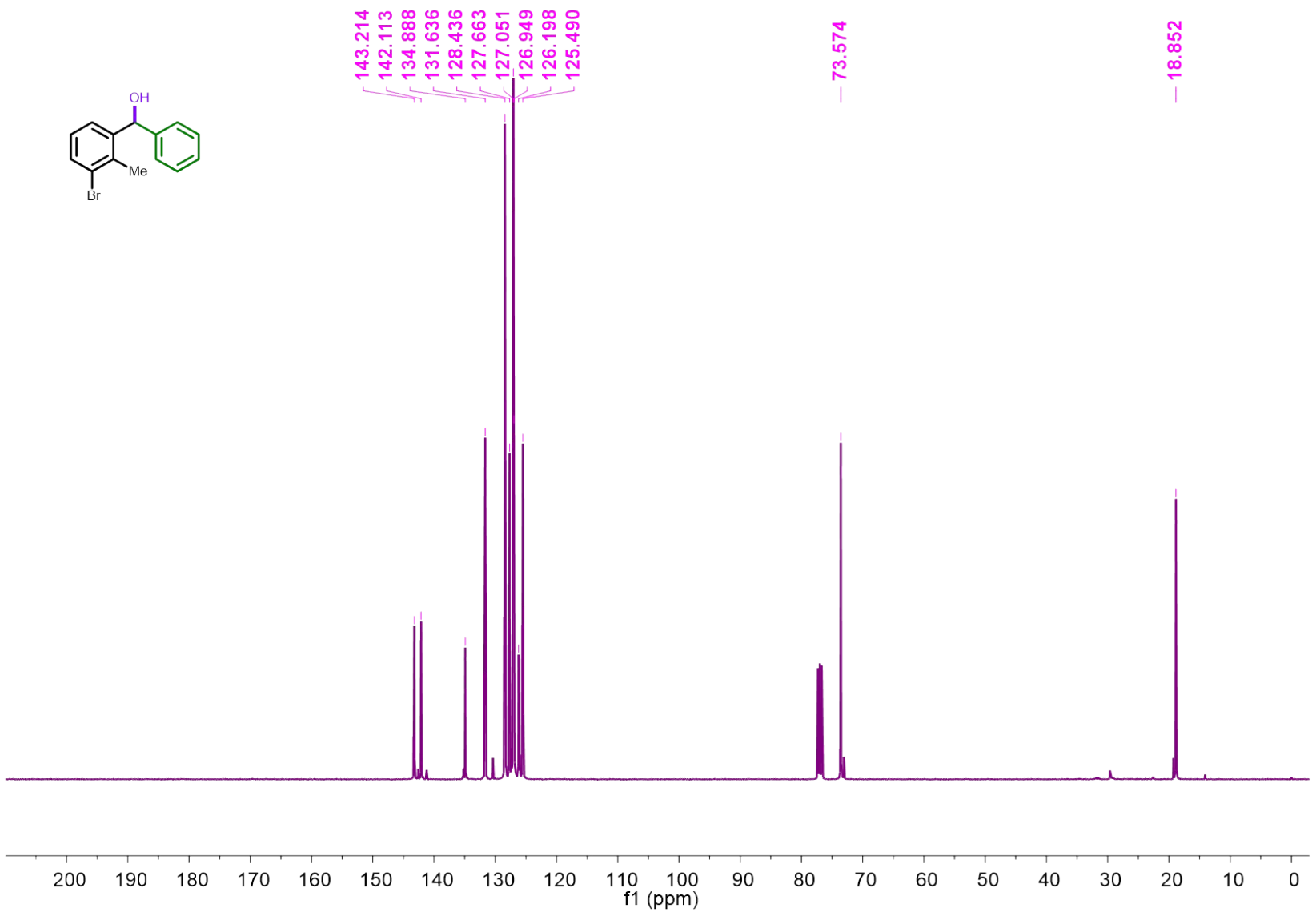


Figure S134. ^{13}C NMR spectra of (3-bromo-2-methylphenyl) (phenyl) methanol **(3b)** in $CDCl_3$.

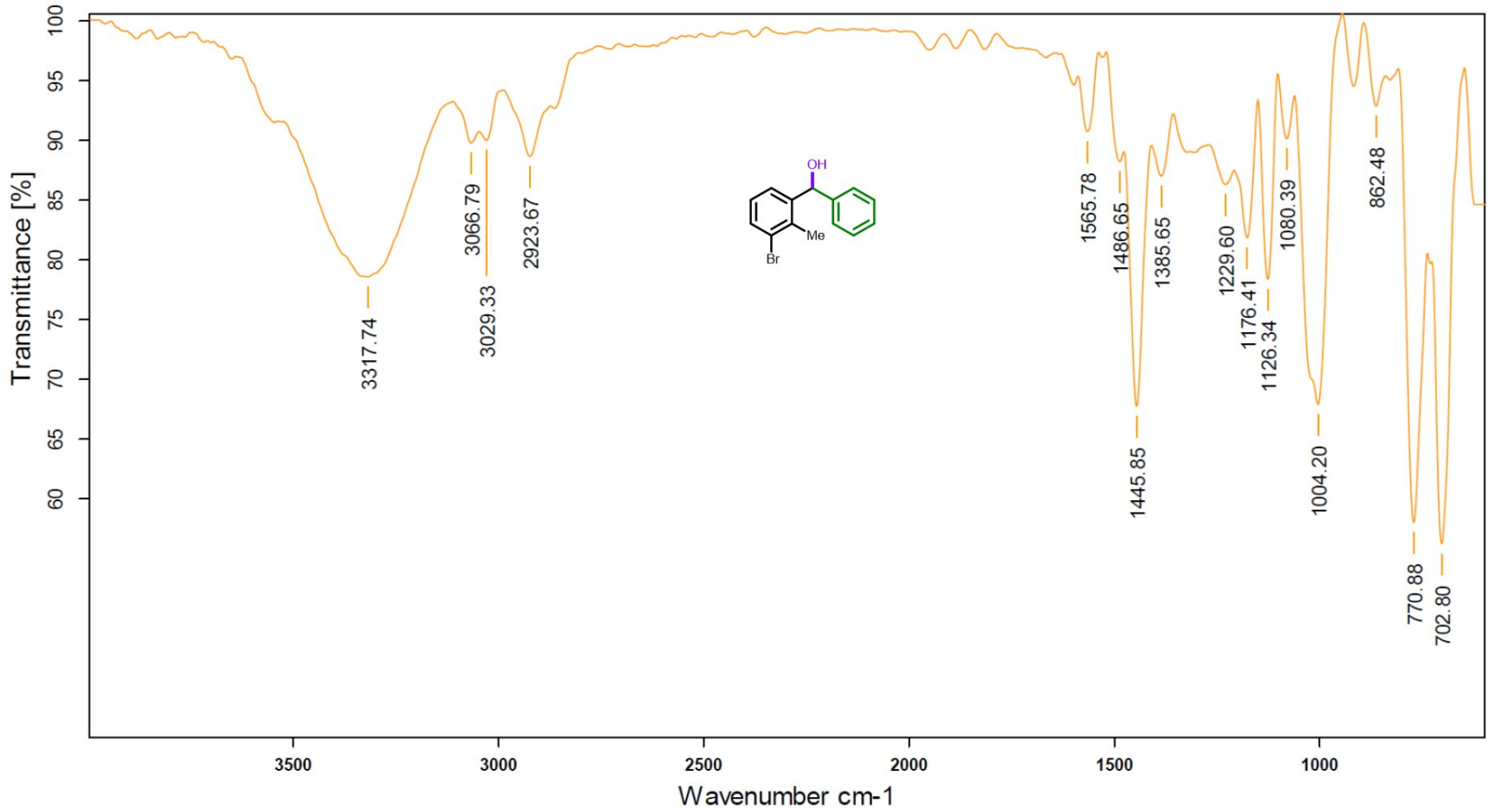


Figure S135. IR spectra of (3-bromo-2-methylphenyl) (phenyl) methanol **(3b)**.

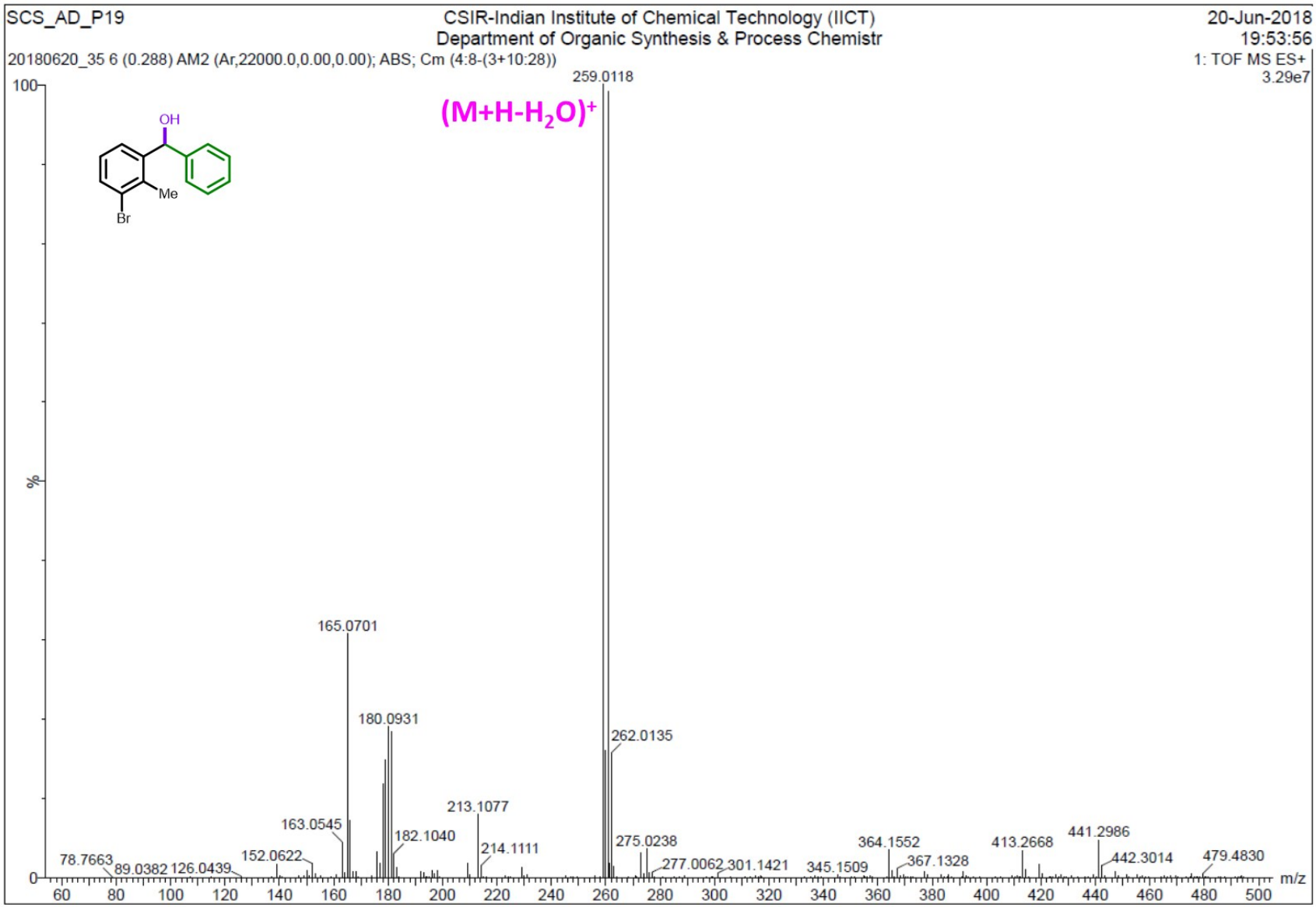


Figure S136. HRMS spectra of (3-bromo-2-methylphenyl) (phenyl) methanol **(3b)**.

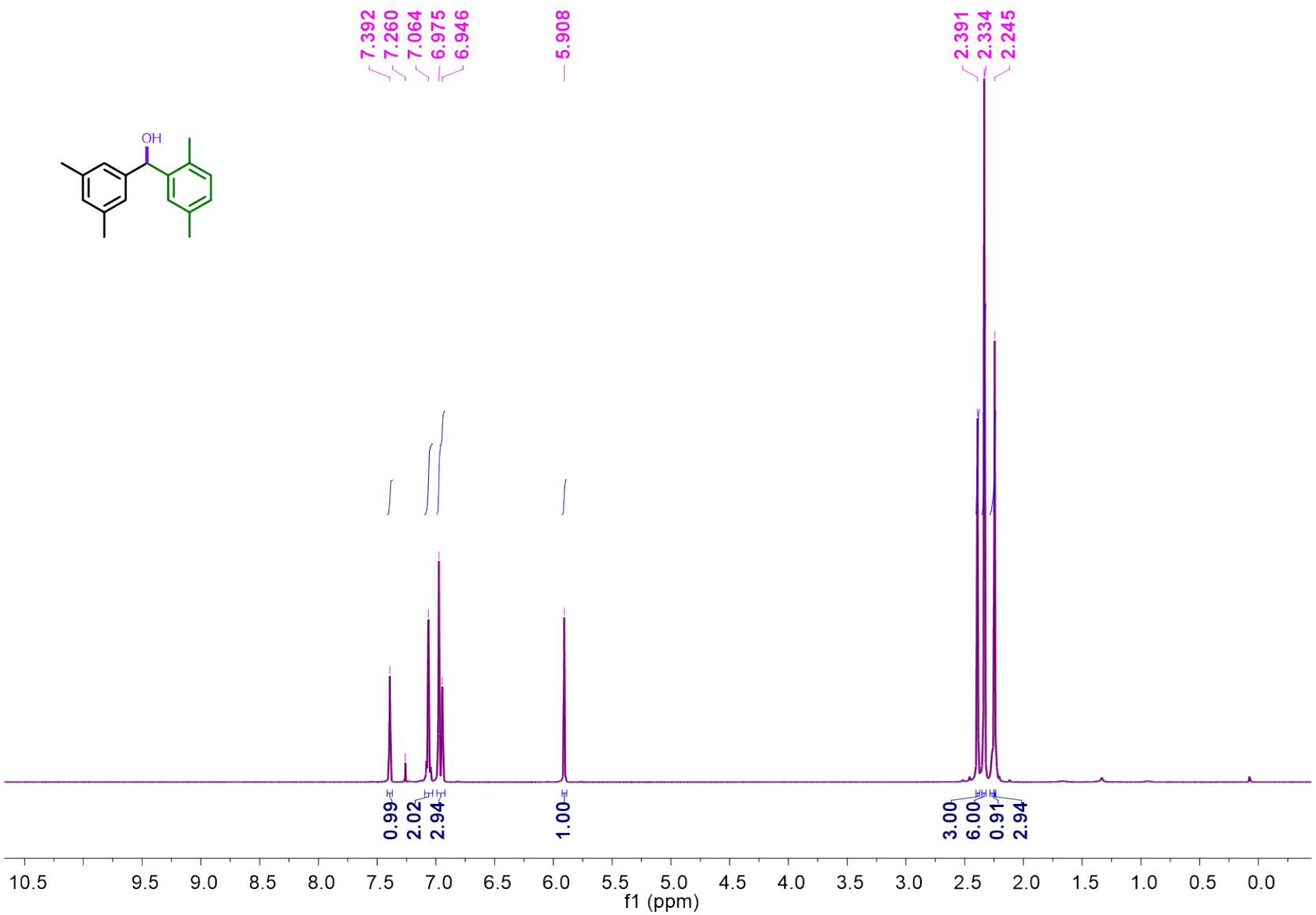


Figure S137. ^{1}H NMR spectra of (2,5-dimethylphenyl) (3,5-dimethylphenyl) methanol **(3c)** in $CDCl_3$.

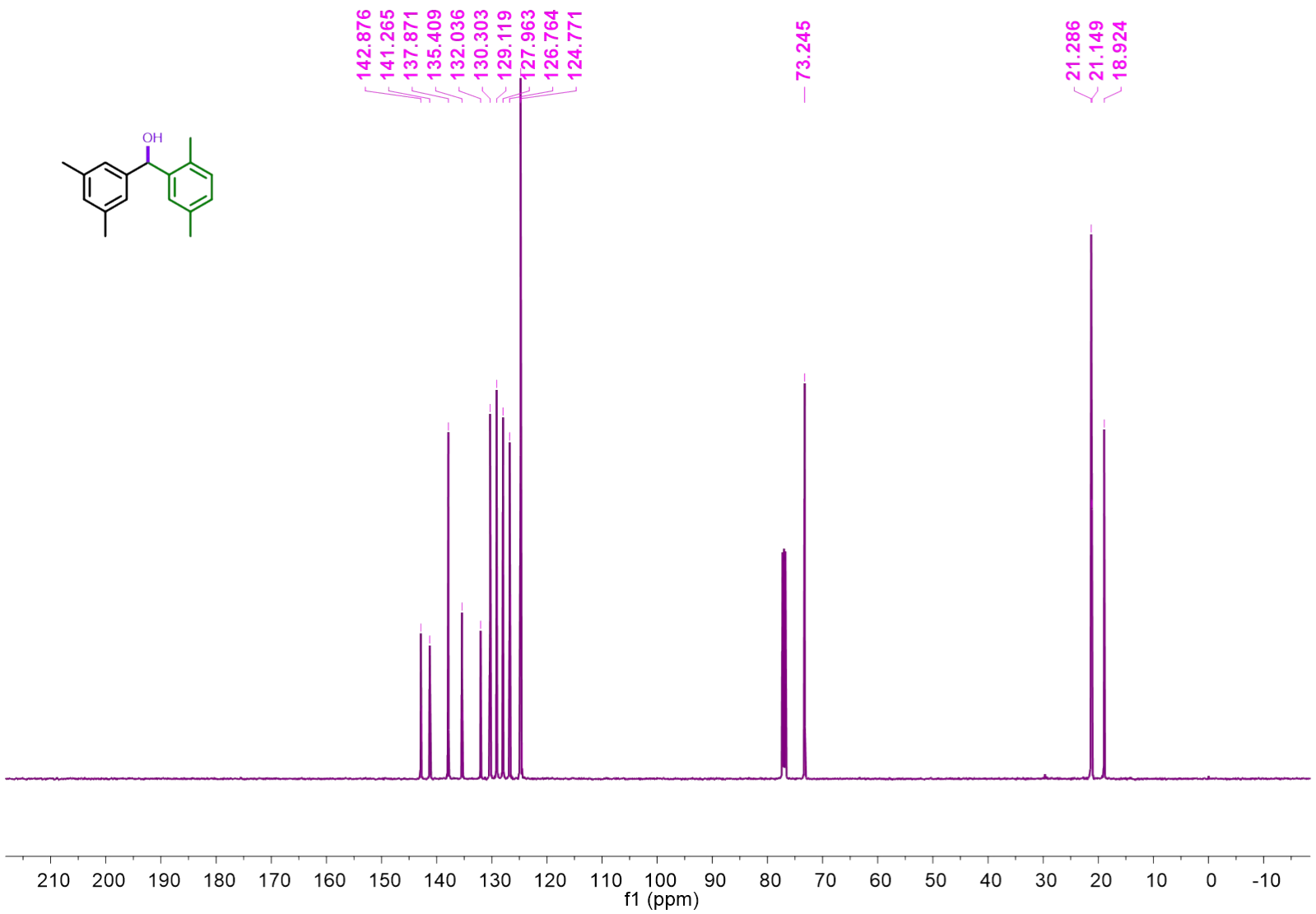


Figure S138. ^{13}C NMR spectra of (2,5-dimethylphenyl) (3,5-dimethylphenyl) methanol **(3c)** in $CDCl_3$.

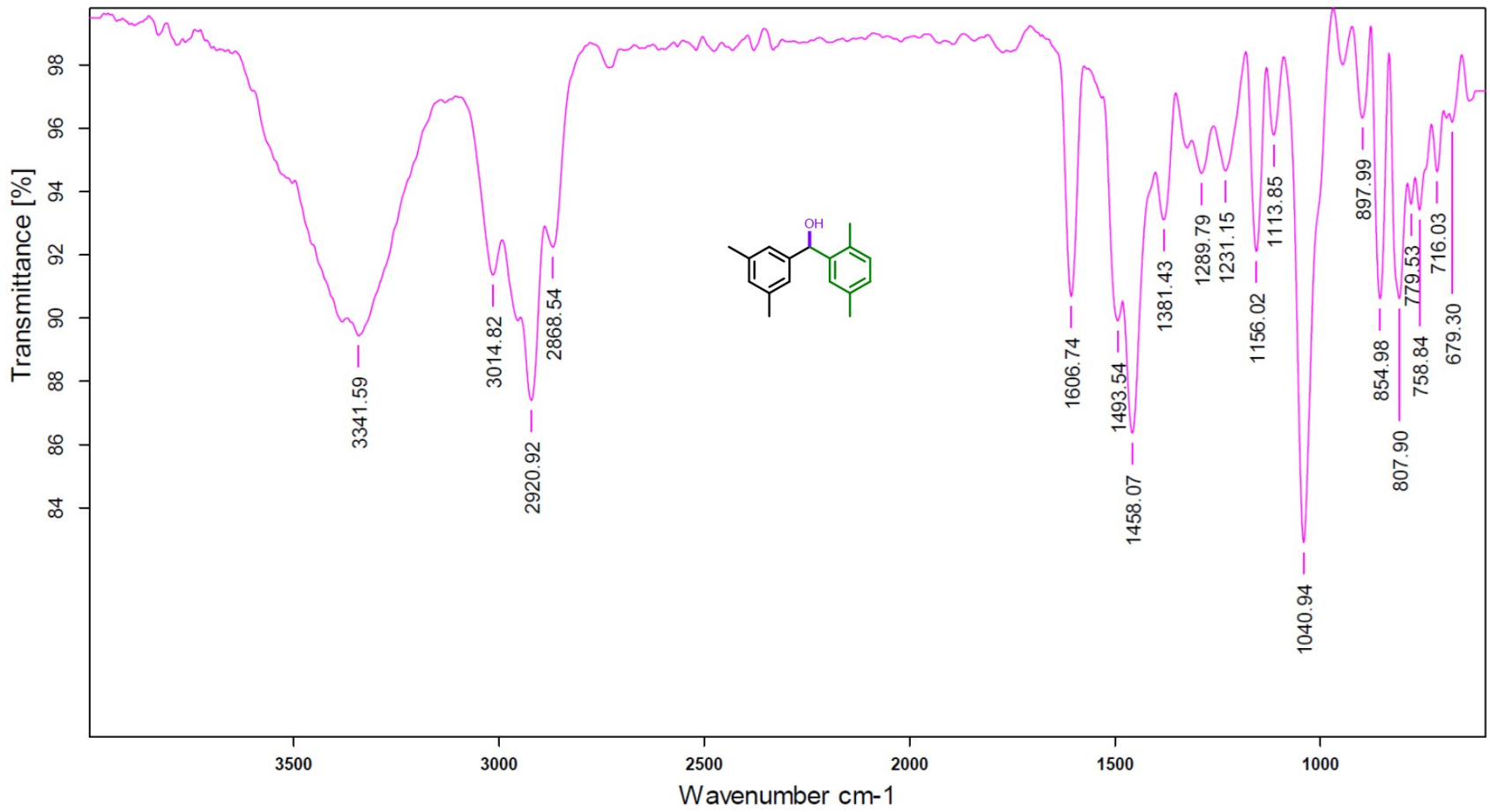


Figure S139. IR spectra of (2,5-dimethylphenyl) (3,5-dimethylphenyl) methanol **(3c)**.

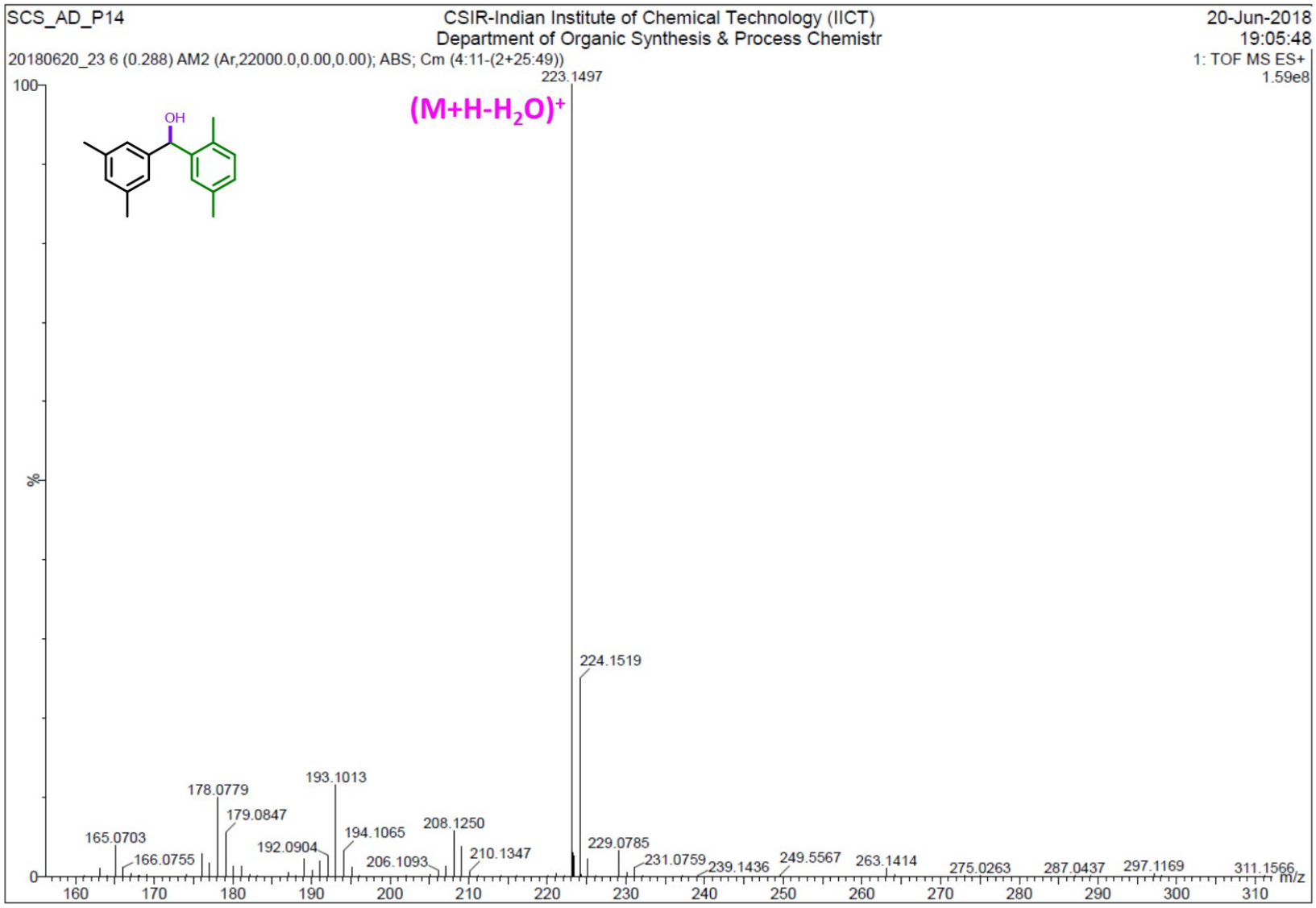


Figure S140. HRMS spectra of (2,5-dimethylphenyl) (3,5-dimethylphenyl) methanol **(3c)**.

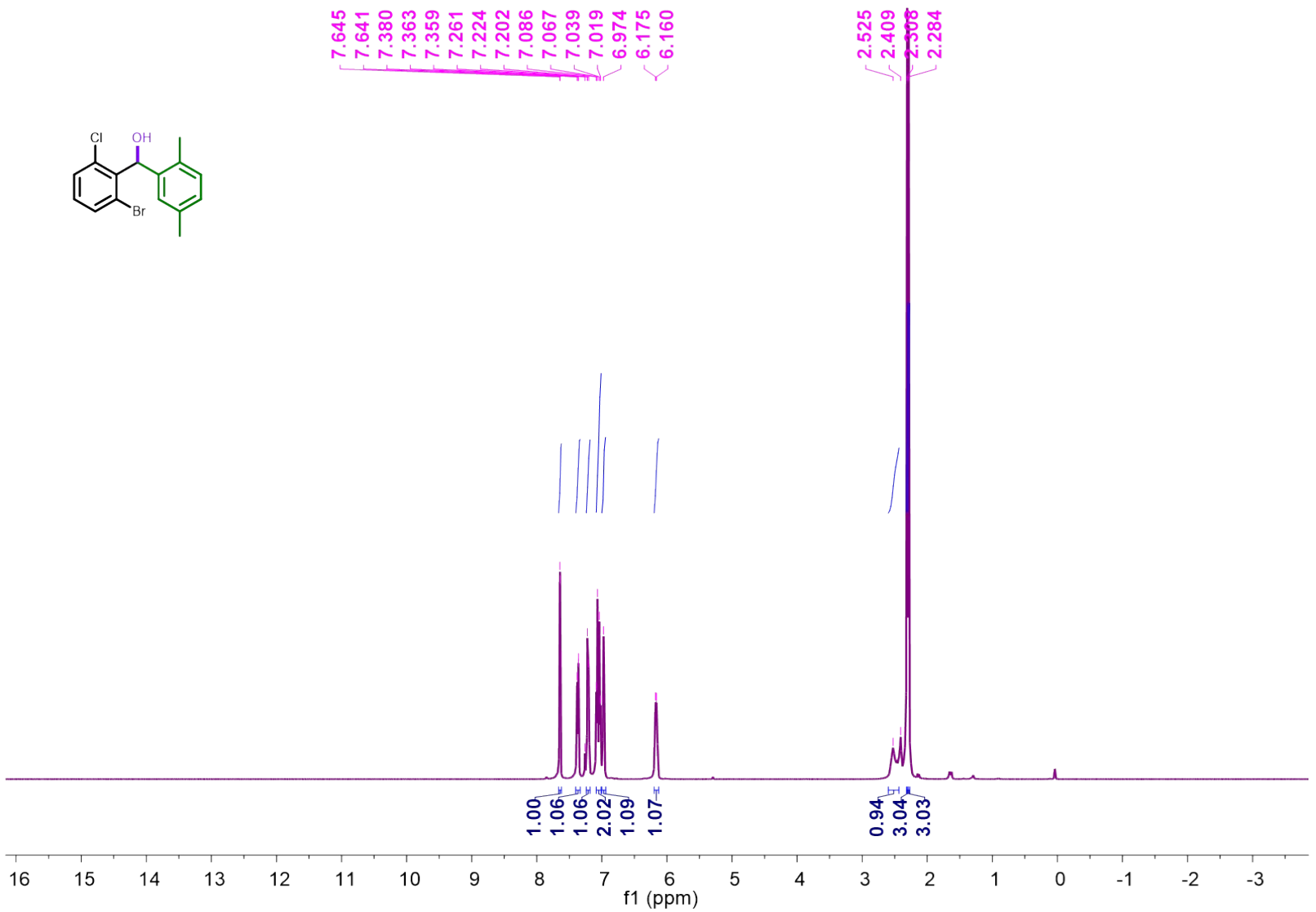
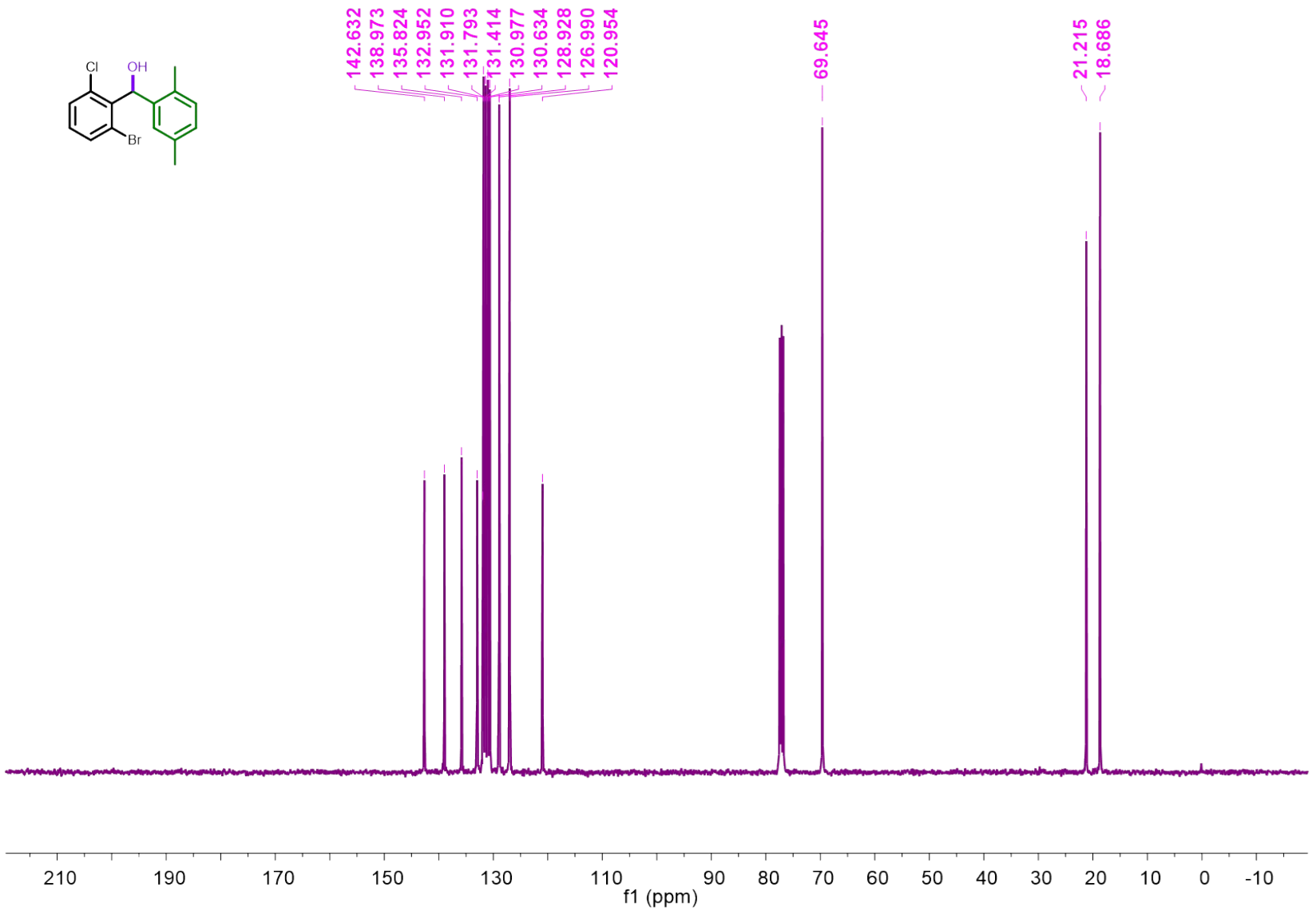


Figure S141. ^{1}H NMR spectra of (2-bromo-6-chlorophenyl) (2,5-dimethylphenyl) methanol **(3d)** in $CDCl_3$.


Cl
OH
Br
142.632
138.973
135.824
132.952
131.910
131.793
131.414
130.977
130.634
128.928
126.990
120.954
69.645
21.215
18.686
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

Figu

re S142. ^{13}C NMR spectra of (2-bromo-6-chlorophenyl) (2,5-dimethylphenyl) methanol **(3d)** in $CDCl_3$.

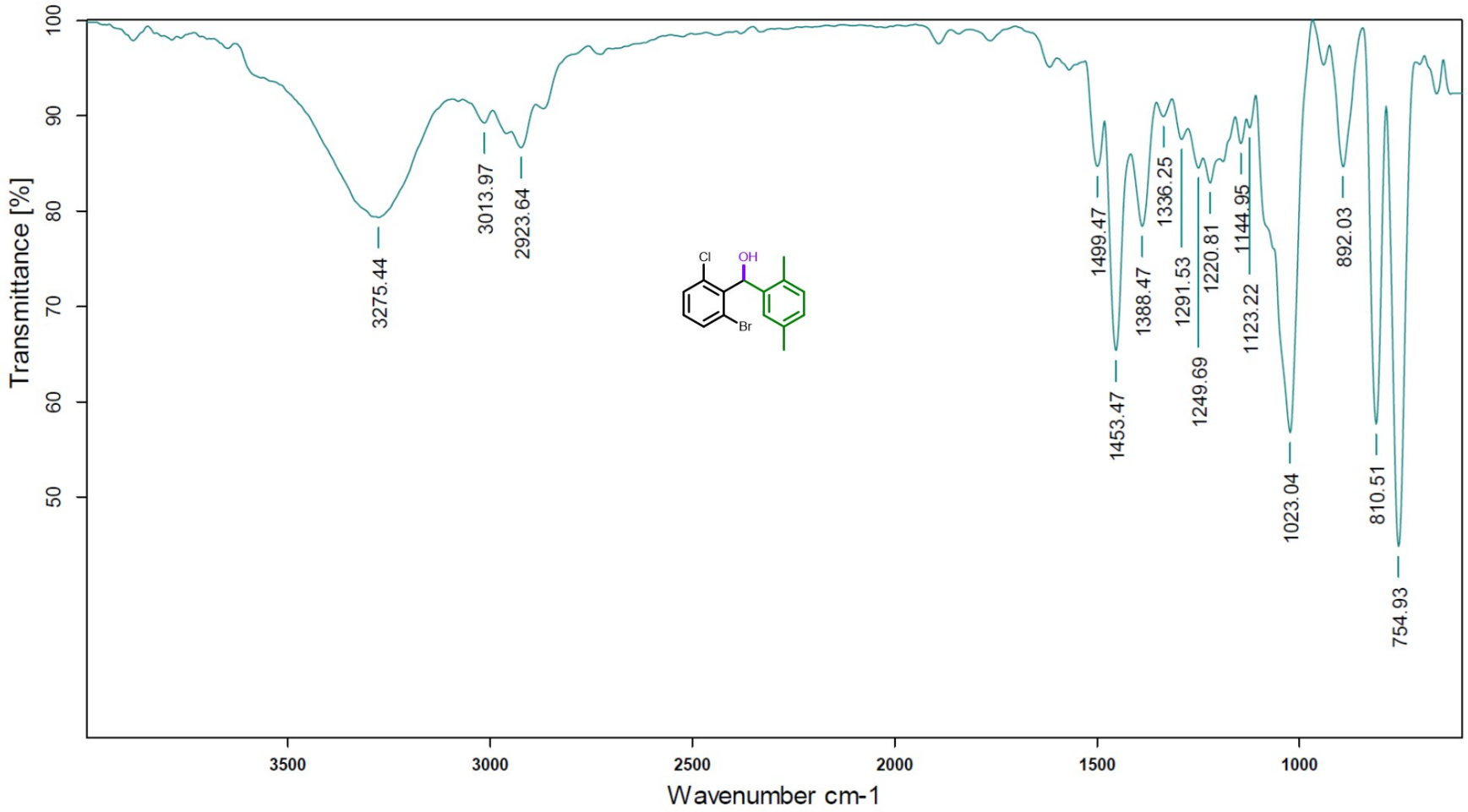


Figure S143. IR spectra of (2-bromo-6-chlorophenyl) (2,5-dimethylphenyl) methanol **(3d)**.

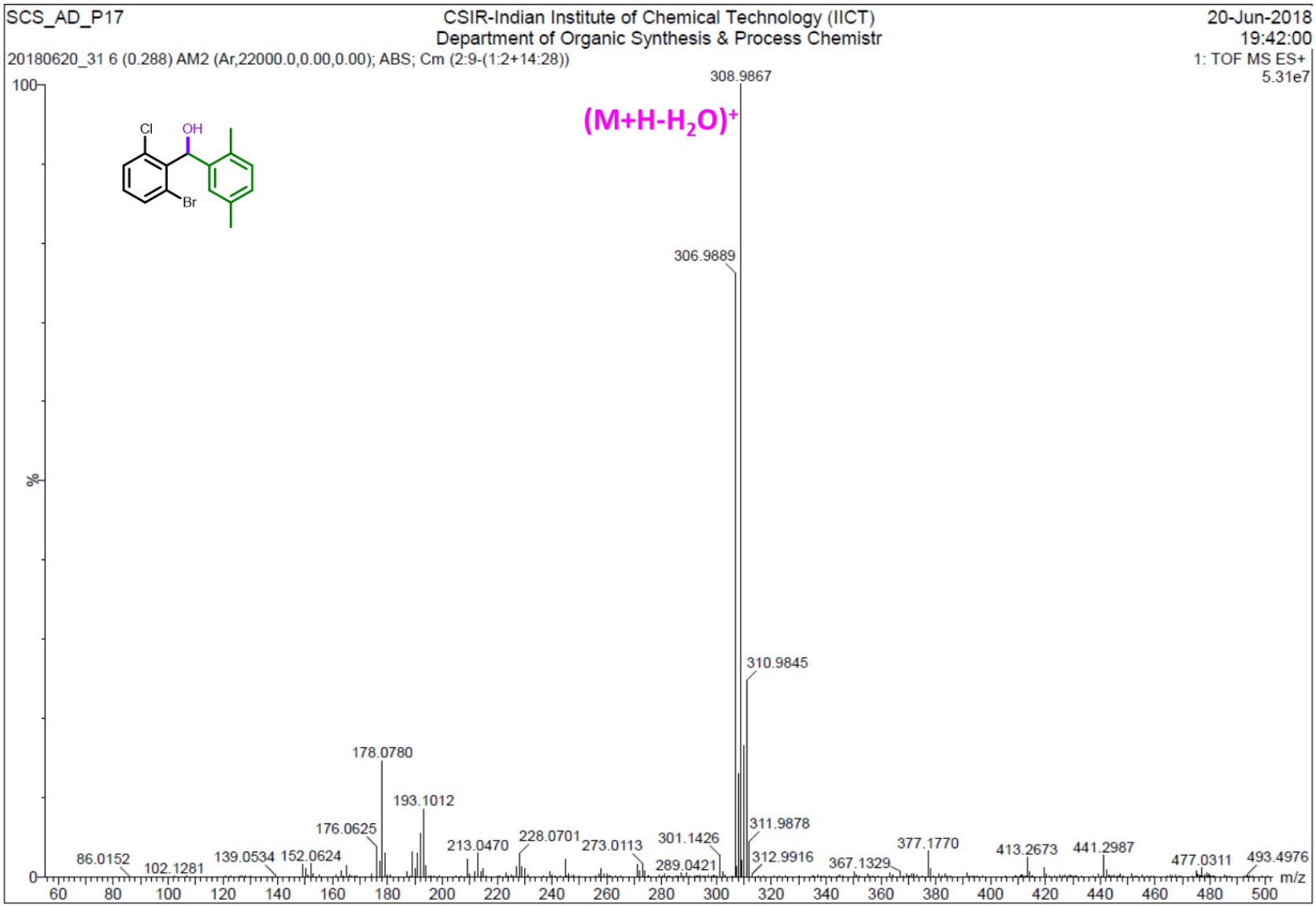


Figure S144. HRMS spectra of (2-bromo-6-chlorophenyl) (2,5-dimethylphenyl) methanol **(3d).**

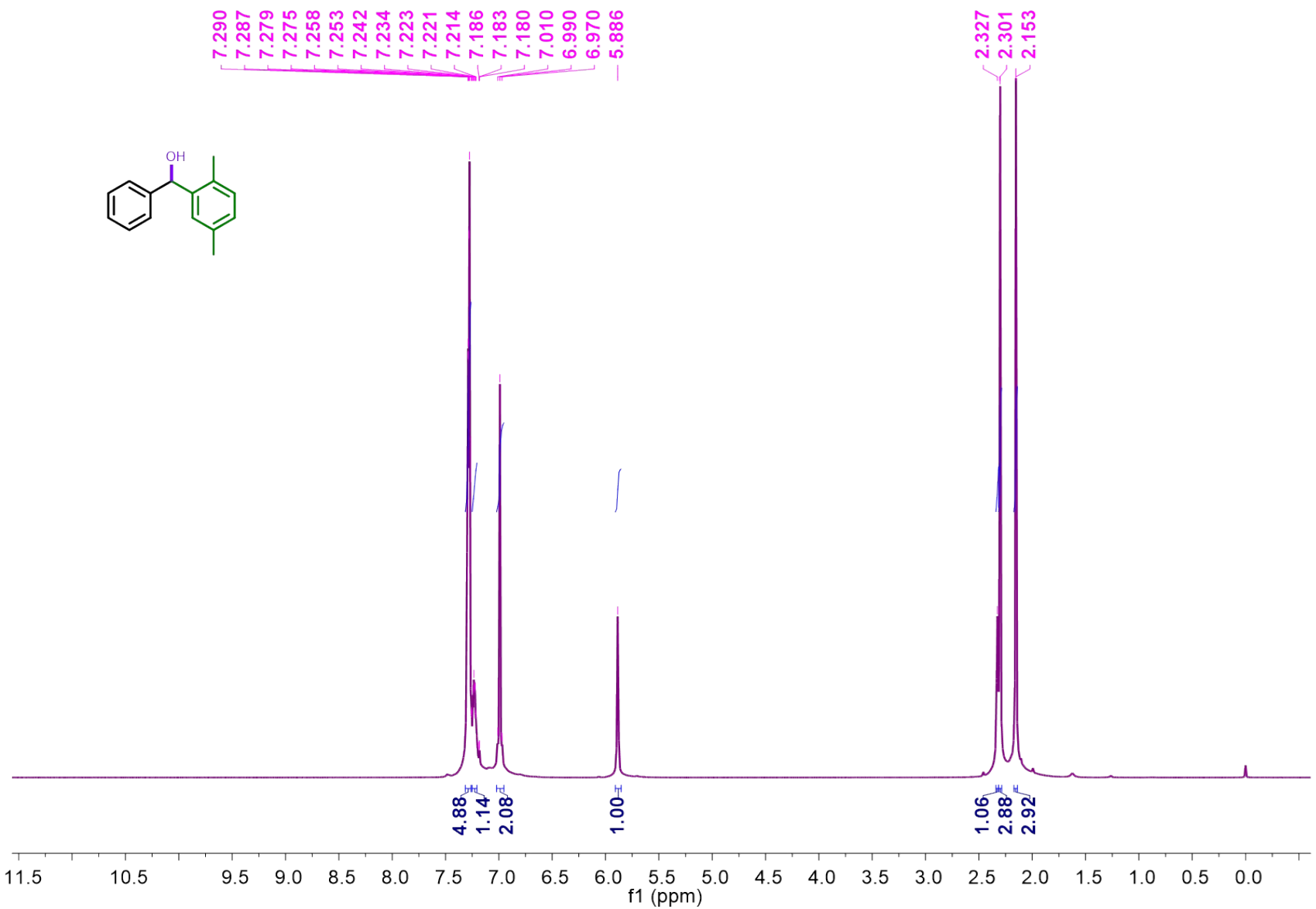


Figure S145. ^{1}H NMR spectra of (2,5-dimethylphenyl) phenyl) methanol **(3e)** in $CDCl_3$.

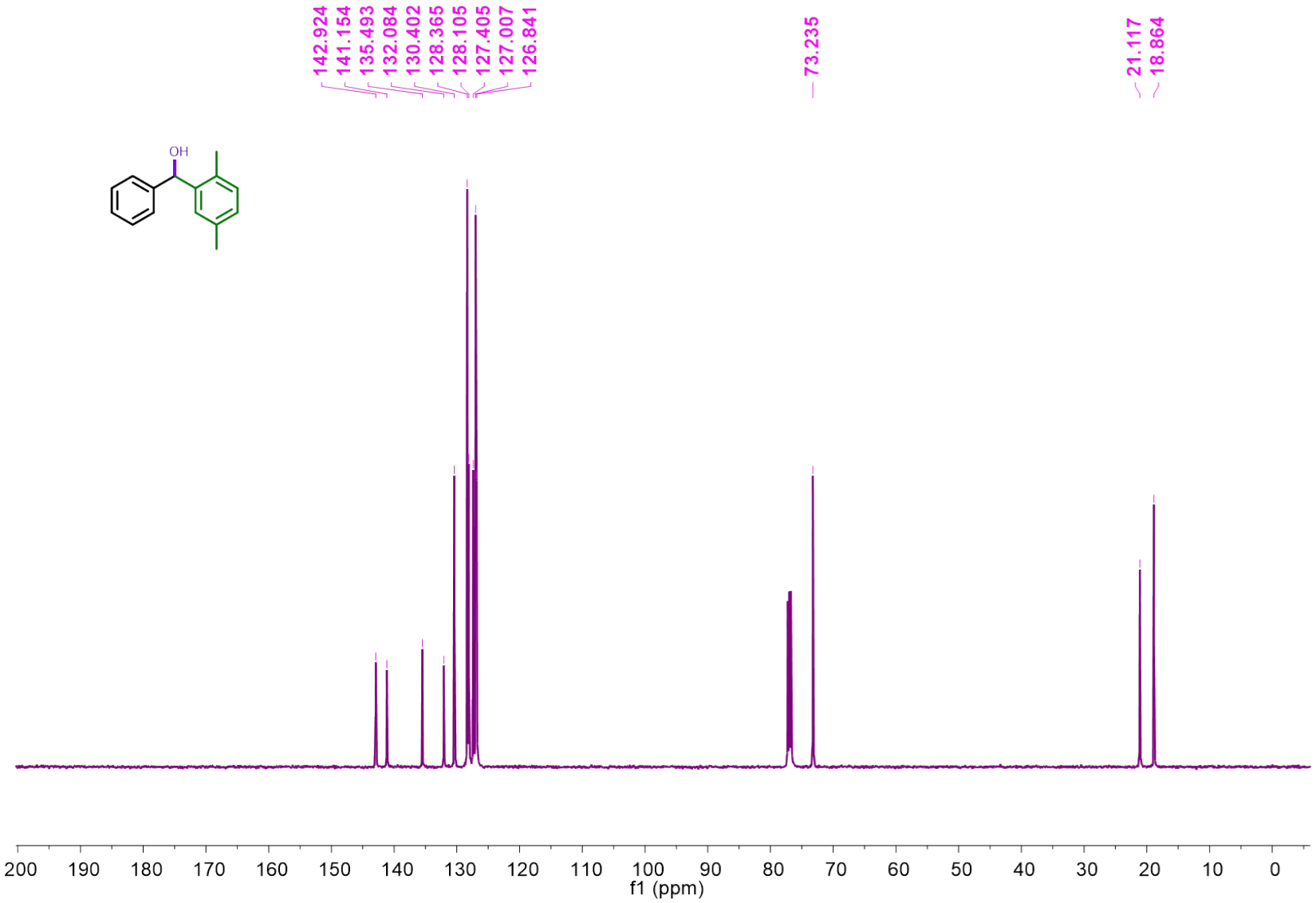


Figure S146. ^{13}C NMR spectra of (2,5-dimethylphenyl) phenyl) methanol **(3e)** in $CDCl_3$.

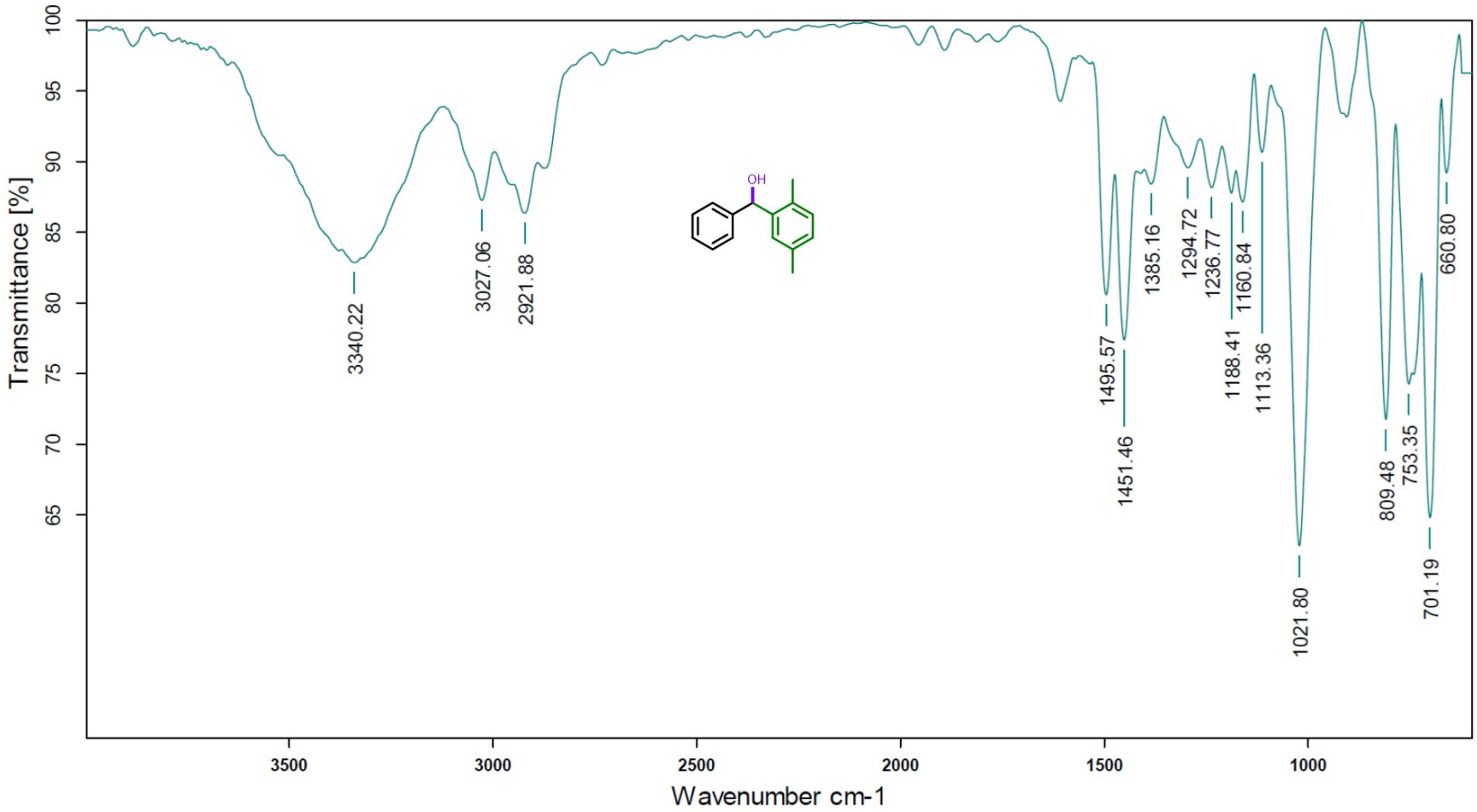


Figure S147. IR spectra of (2,5-dimethylphenyl) phenyl) methanol **(3e)**.

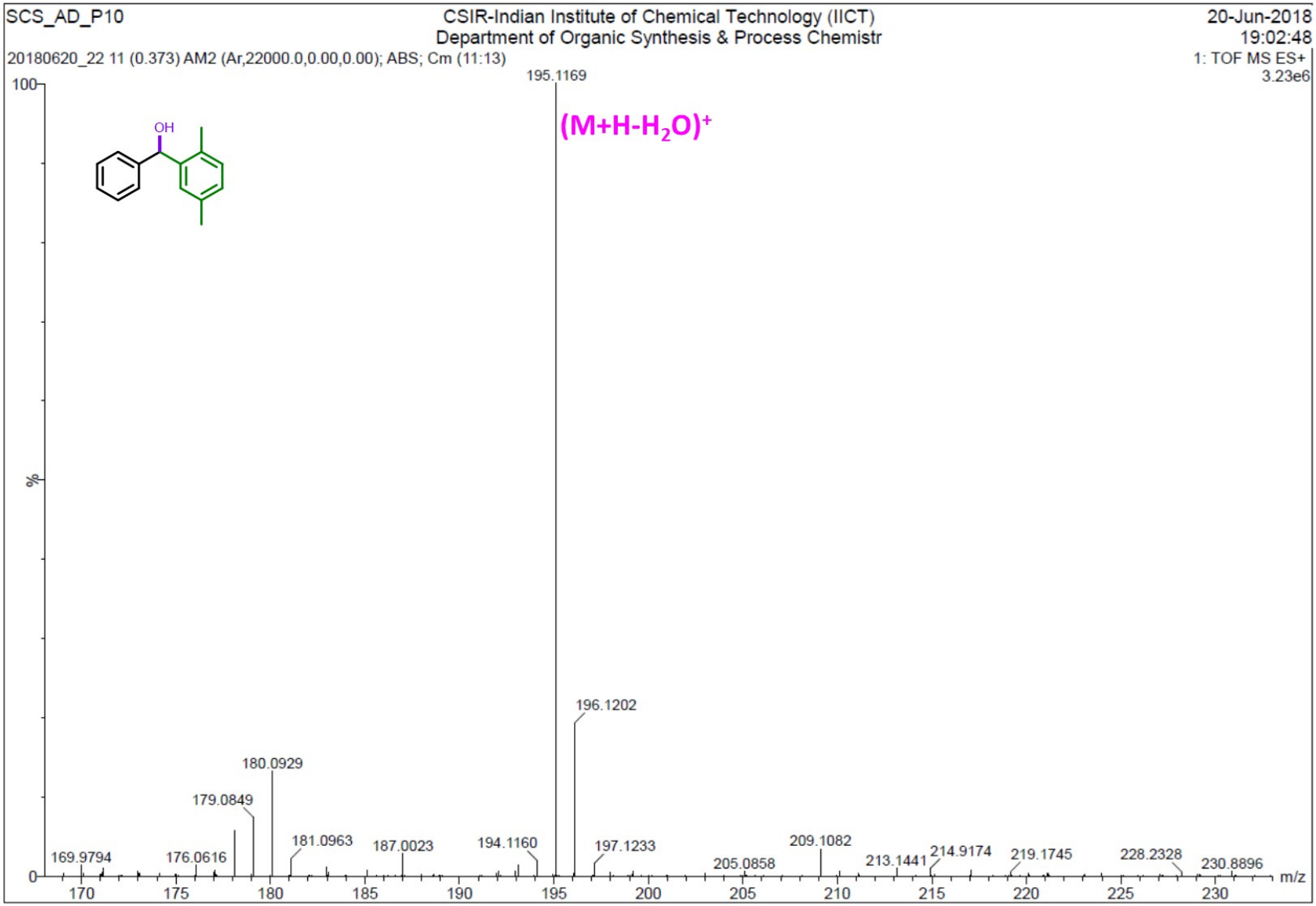
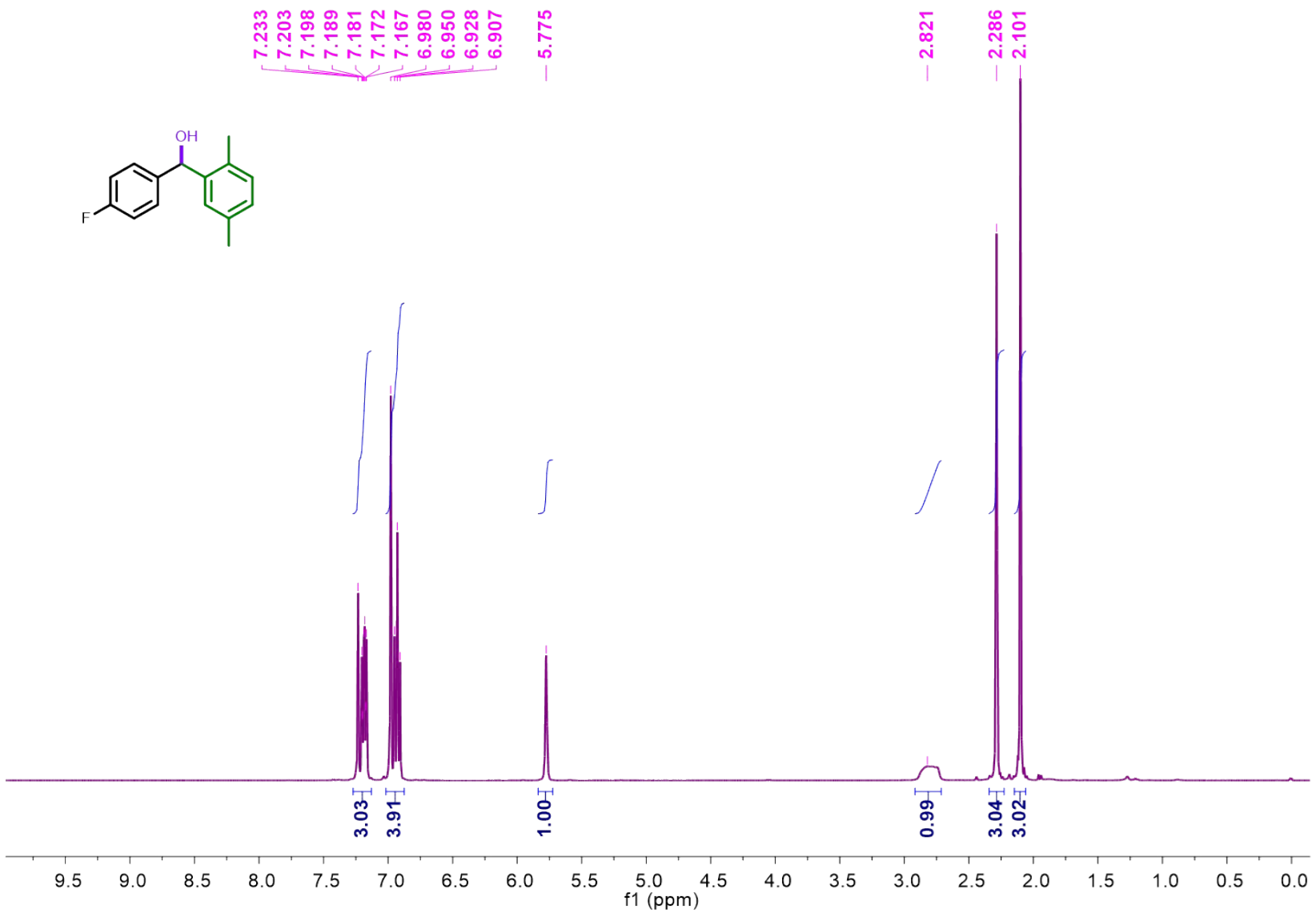
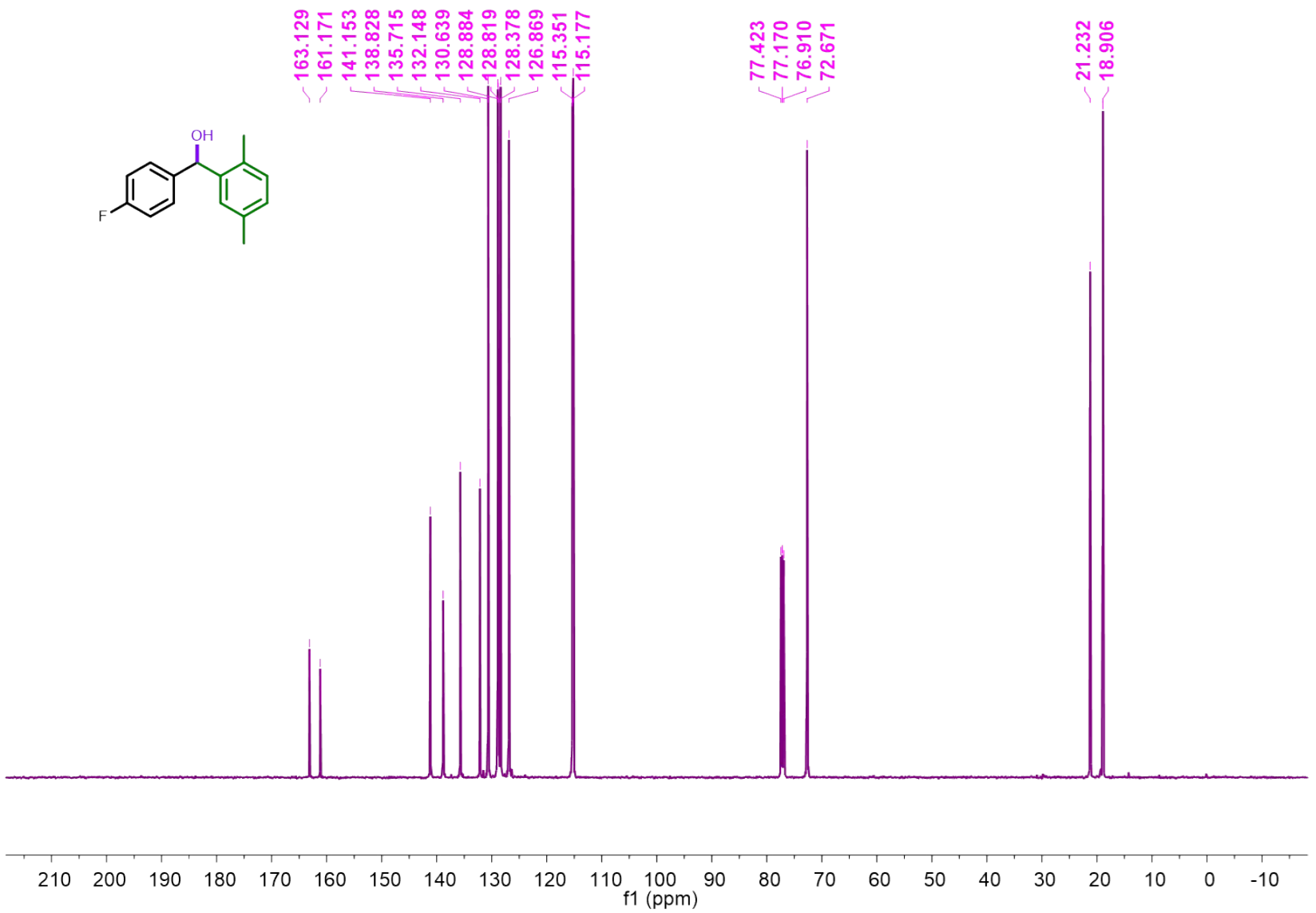


Figure S148. HRMS spectra of (2,5-dimethylphenyl) phenyl) methanol **(3e)**.


7.233
7.203
7.198
7.189
7.181
7.172
7.167
6.980
6.950
6.928
6.907
5.775
2.821
2.286
2.101
OH
F
3.03
3.91
1.00
0.99
3.04
3.02
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

Figu

re S149. ^{1}H NMR spectra of (2,5-dimethylphenyl) (4-fluorophenyl) methanol **(3f)** in $CDCl_3$.


163.129
161.171
141.153
138.828
135.715
132.148
130.639
128.884
128.819
128.378
126.869
115.351
115.177
77.423
77.170
76.910
72.671
21.232
18.906
OH
F
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Figu

re S150. ^{13}C NMR spectra of (2,5-dimethylphenyl) (4-fluorophenyl) methanol **(3f)** in $CDCl_3$.

Figure S151. ^{19}F NMR spectra of (2,5-dimethylphenyl) (4-fluorophenyl) methanol **(3f)** in $CDCl_3$.

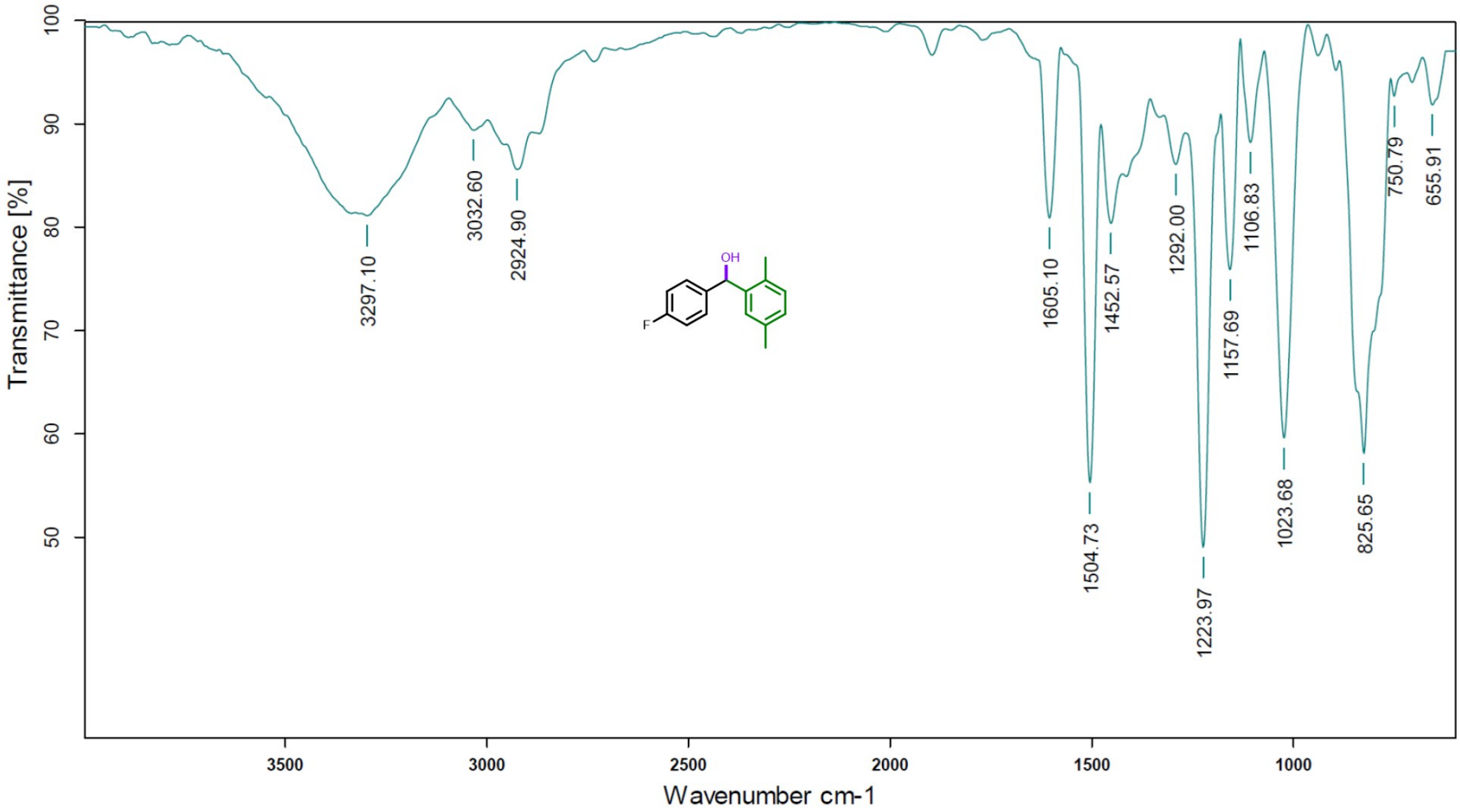
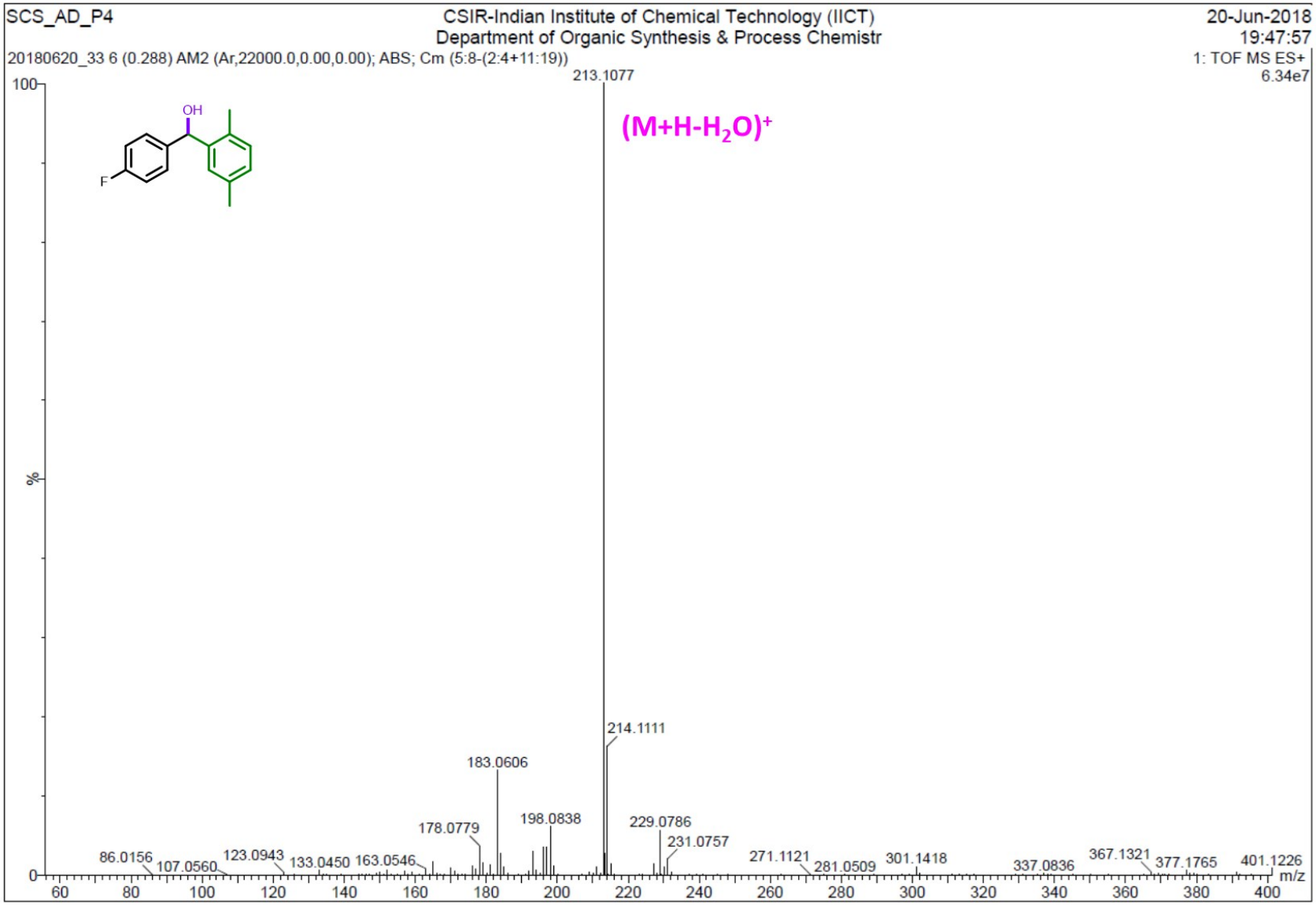
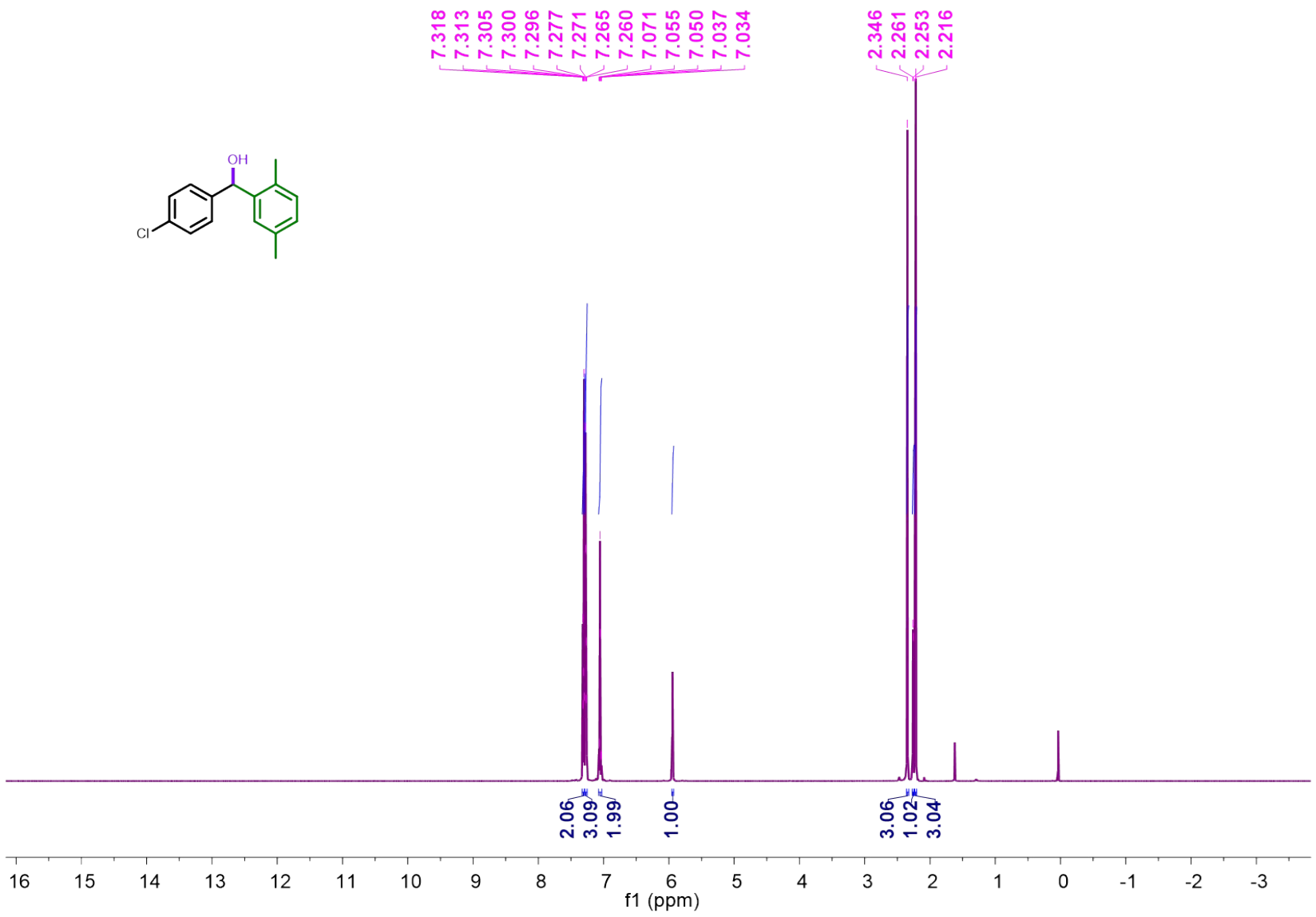


Figure S152. IR spectra of (2,5-dimethylphenyl) (4-fluorophenyl) methanol **(3f).**


SCS_AD_P4
CSIR-Indian Institute of Chemical Technology (IICT)
Department of Organic Synthesis & Process Chemistr
20-Jun-2018
19:47:57
20180620_33 6 (0.288) AM2 (Ar,22000.0,0.00,0.00); ABS; Cm (5:8-(2:4+11:19))
1: TOF MS ES+
6.34e7
213.1077
(M+H-H2O)+
OH
F
214.1111
183.0606
178.0779
198.0838
229.0786
231.0757
86.0156
107.0560
123.0943
133.0450
163.0546
271.1121
281.0509
301.1418
337.0836
367.1321
377.1765
401.1226
%
m/z

Fig

ure S153. HRMS spectra of (2,5-dimethylphenyl) (4-fluorophenyl) methanol **(3f).**


7.318
7.313
7.305
7.300
7.296
7.277
7.271
7.265
7.260
7.071
7.055
7.050
7.037
7.034
2.346
2.261
2.253
2.216
OH
Cl
2.06
3.09
1.99
1.00
3.06
1.02
3.04
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

Fig

ure S154. ^{1}H NMR spectra of (2,5-dimethylphenyl) (4-chlorophenyl) methanol**(3g)** in $CDCl_3$.

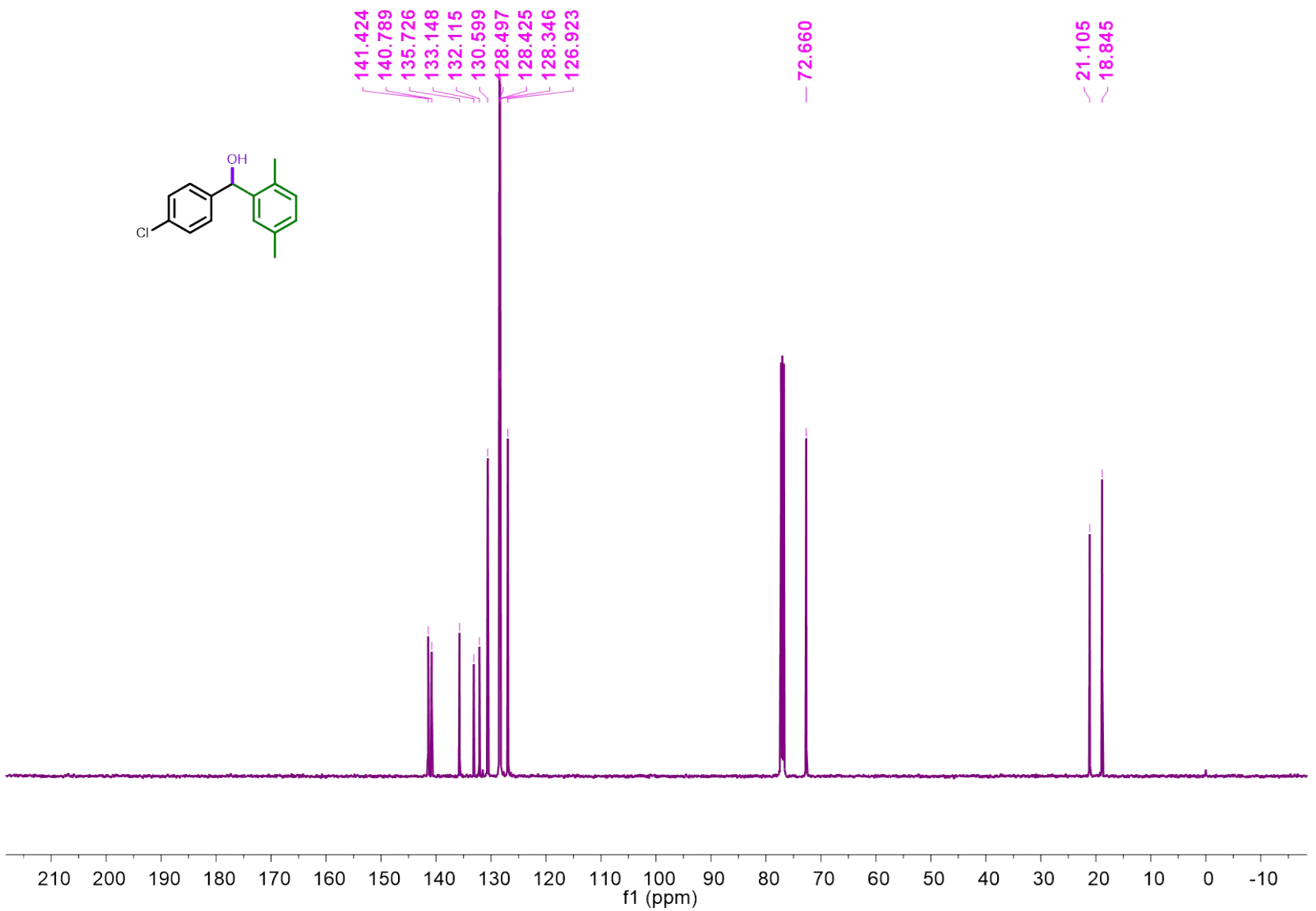


Figure S155. ^{13}C NMR spectra of (2,5-dimethylphenyl) (4-chlorophenyl) methanol**(3g)** in $CDCl_3$.

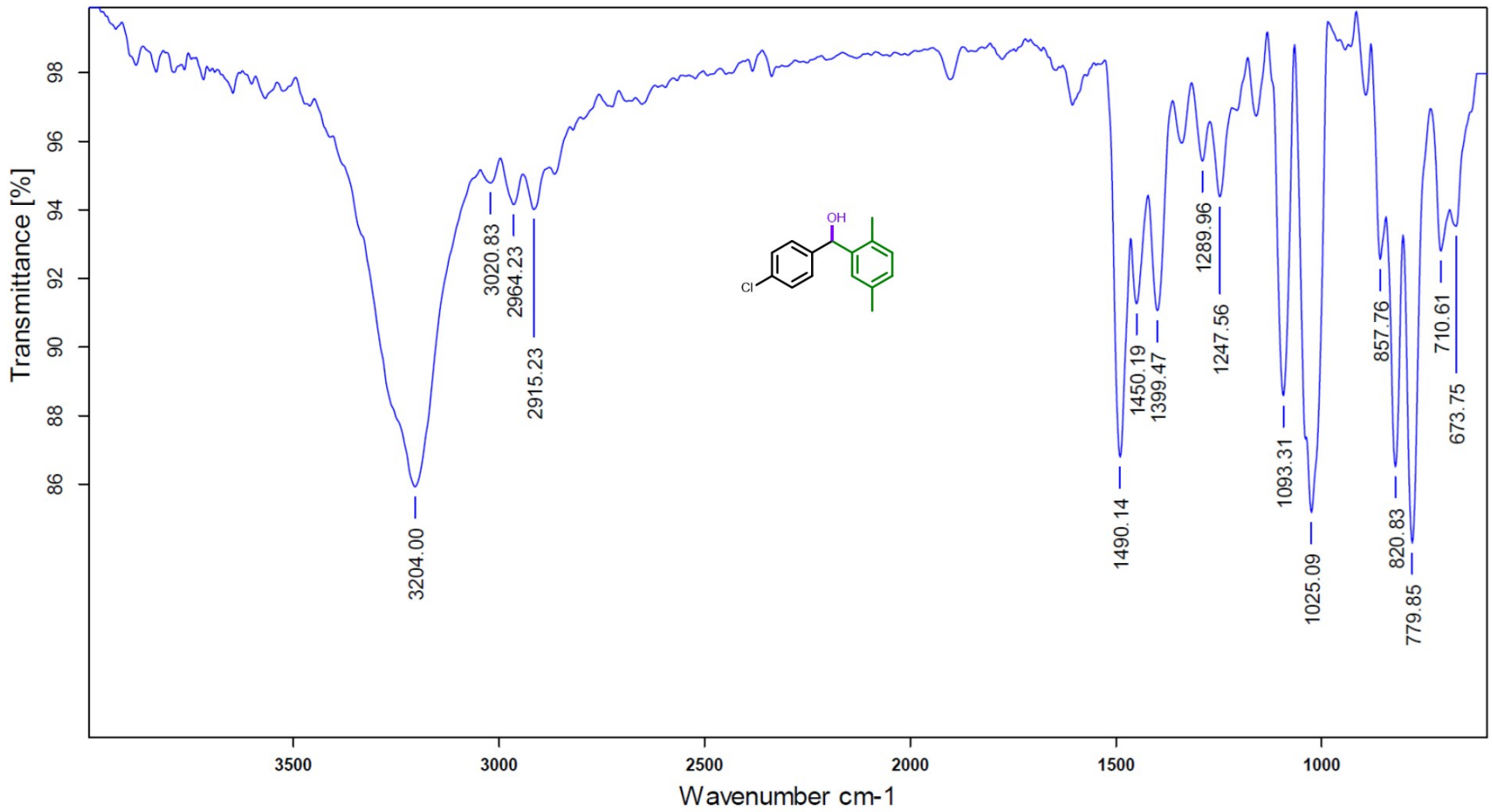


Figure S156. IR spectra of (2,5-dimethylphenyl) (4-chlorophenyl) methanol**(3g)**.

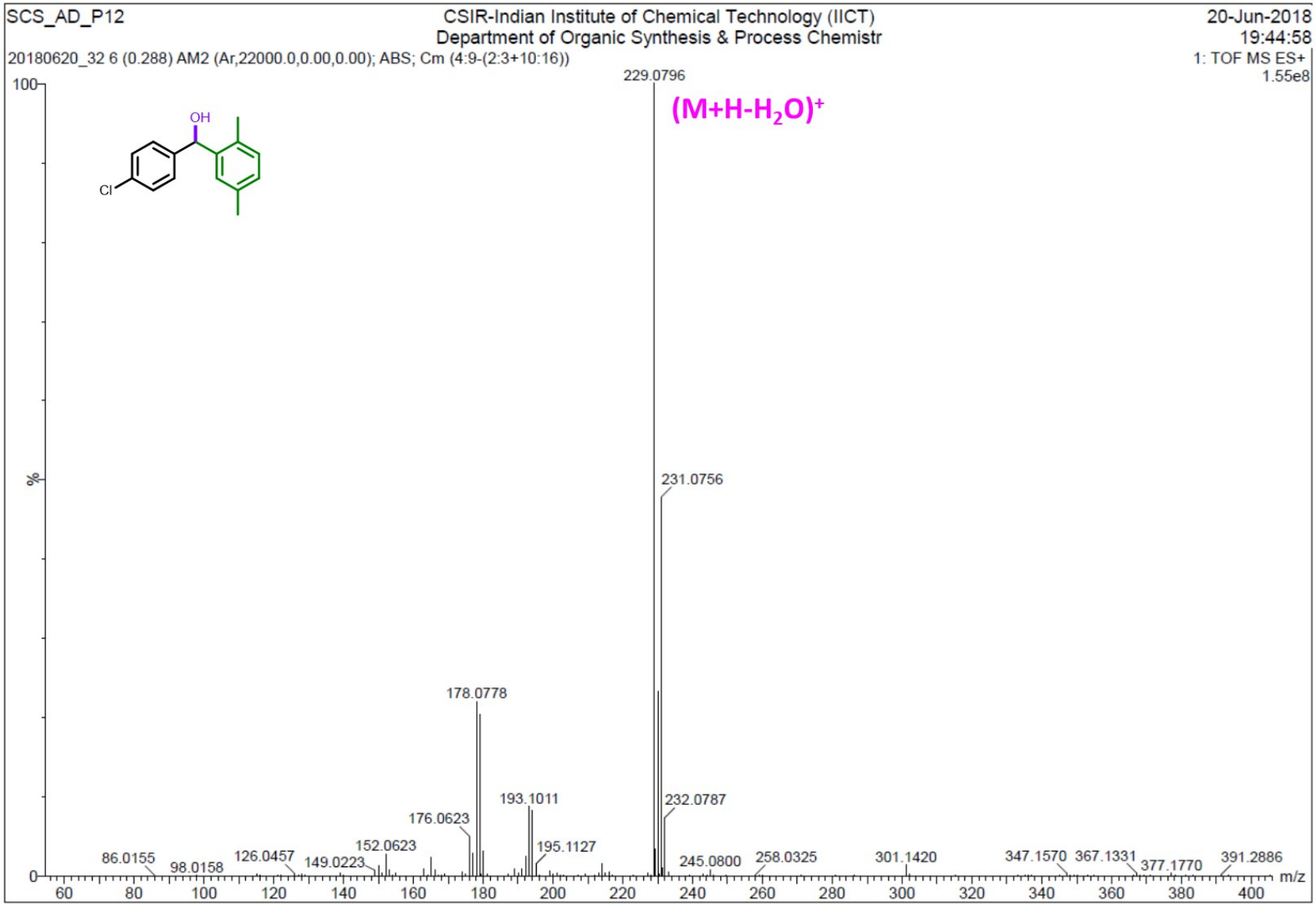


Figure S157. HRMS spectra of (2,5-dimethylphenyl) (4-chlorophenyl) methanol**(3g)**.

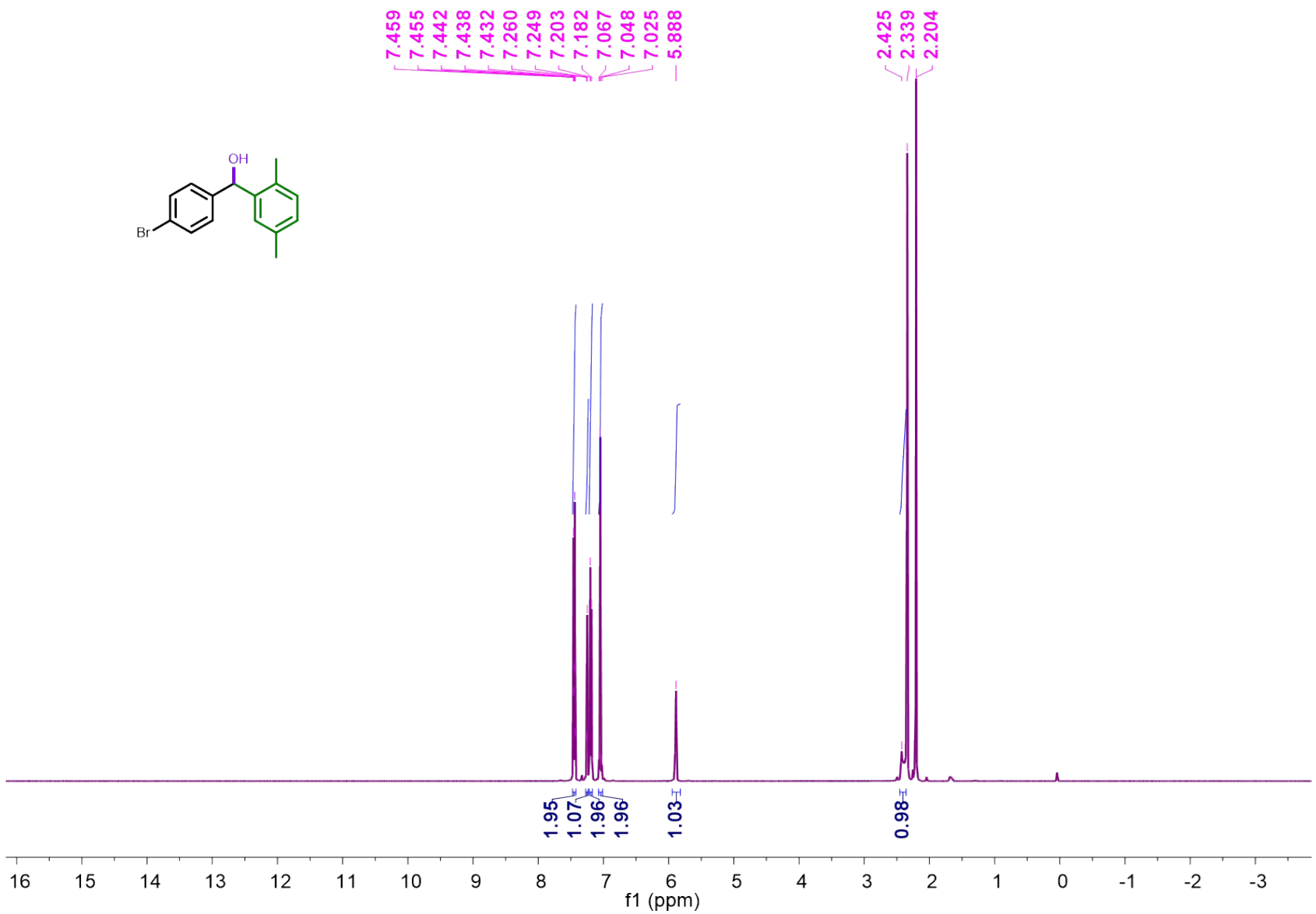


Figure S158. ^{1}H NMR spectra of (2,5-dimethylphenyl) (4-bromophenyl) methanol **(3h)** in $CDCl_3$.

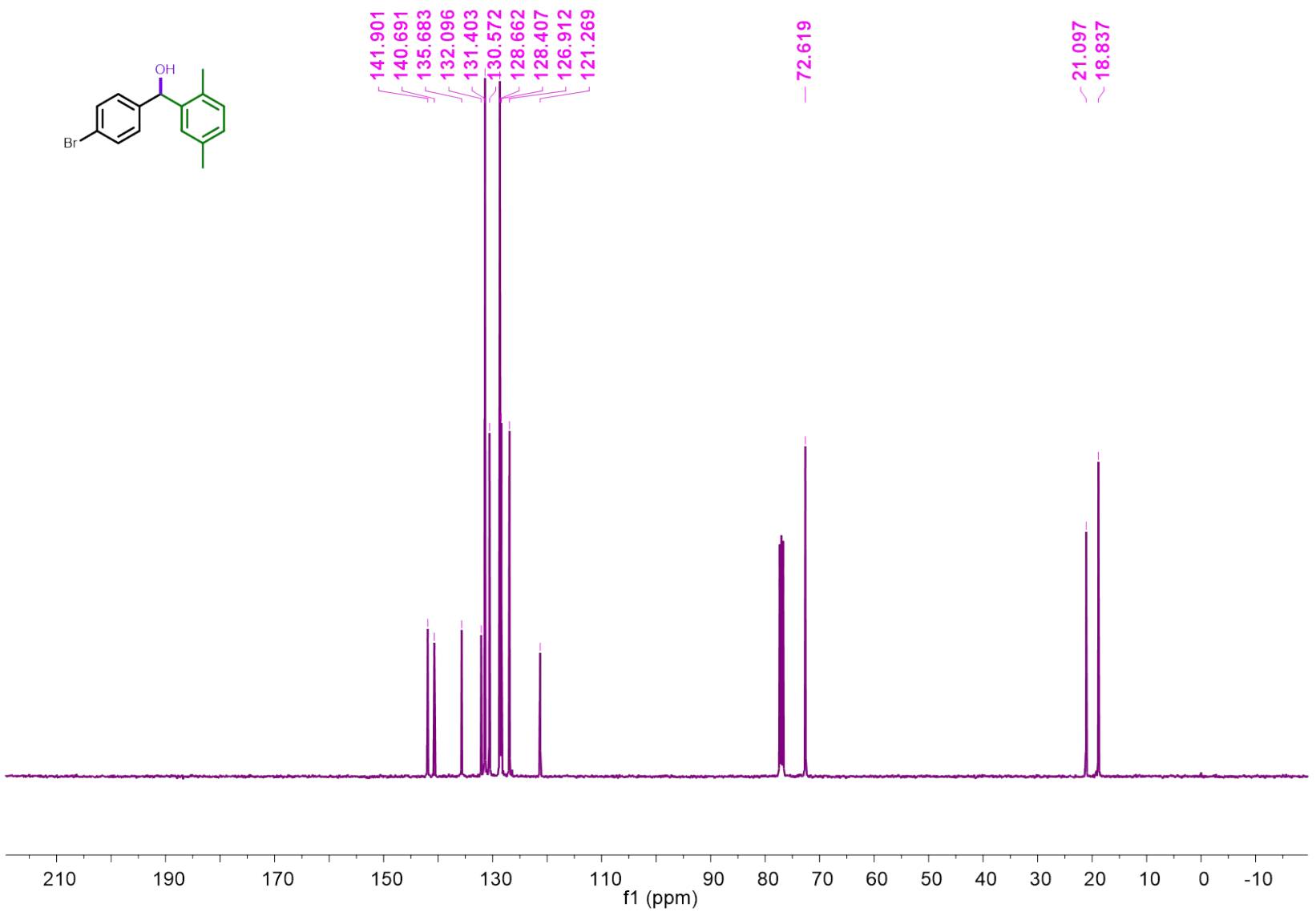


Figure S159. ^{13}C NMR spectra of (2,5-dimethylphenyl)(4-bromophenyl)methanol **(3h)** in $CDCl_3$.

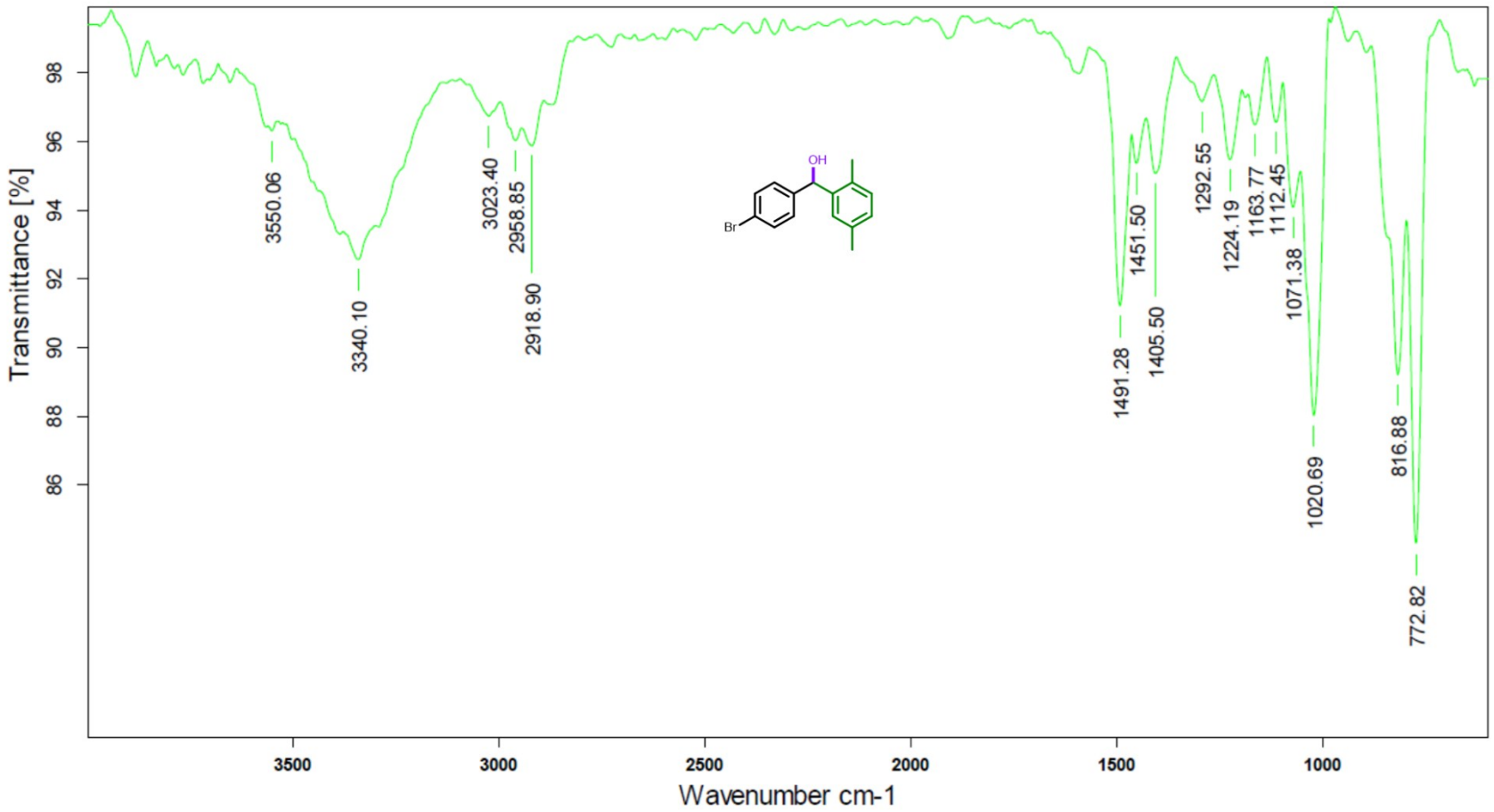


Figure S160. IR spectra of (2,5-dimethylphenyl)(4-bromophenyl)methanol **(3h**).

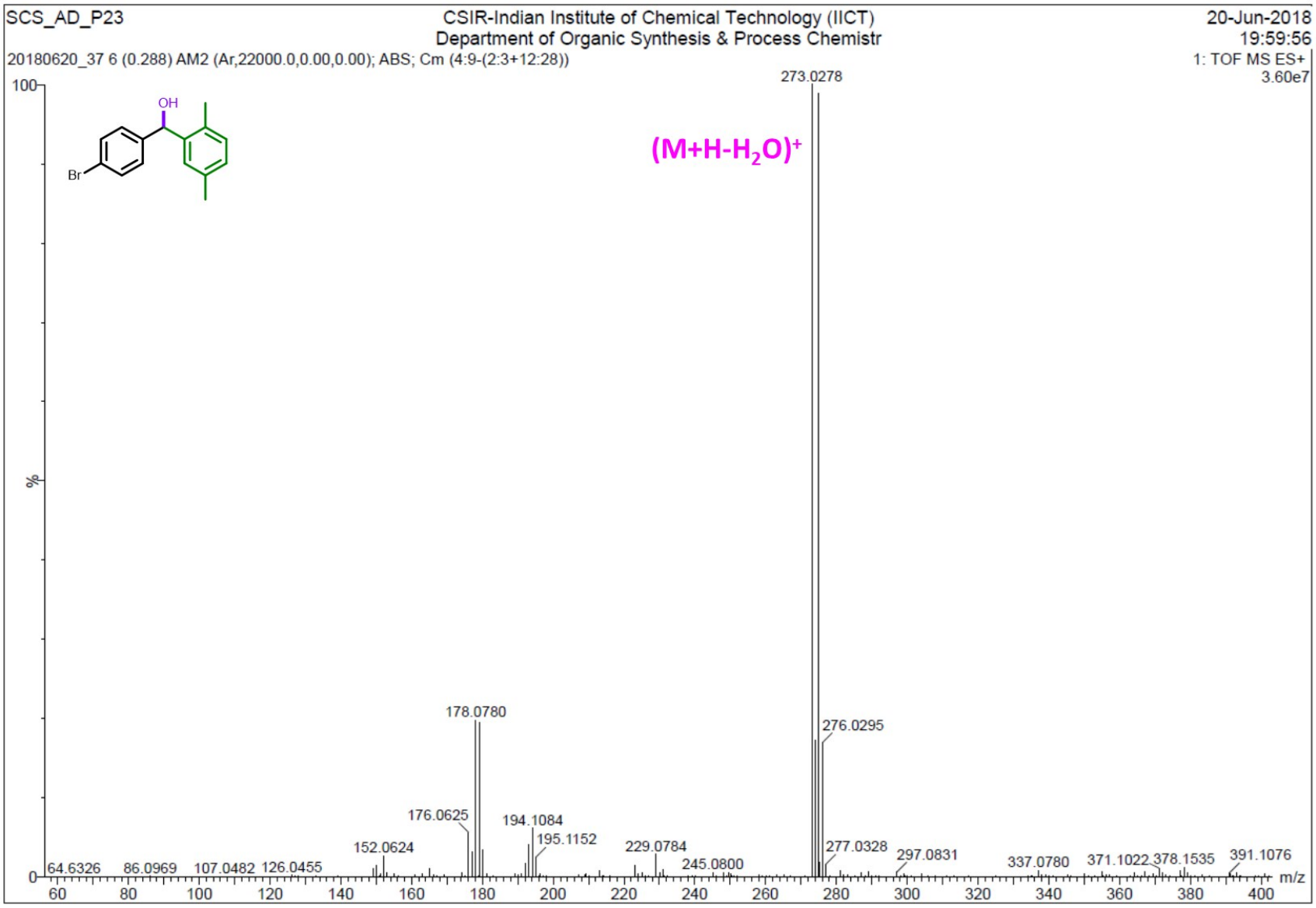


Figure S161. HRMS spectra of (2,5-dimethylphenyl)(4-bromophenyl)methanol **(3h)**.

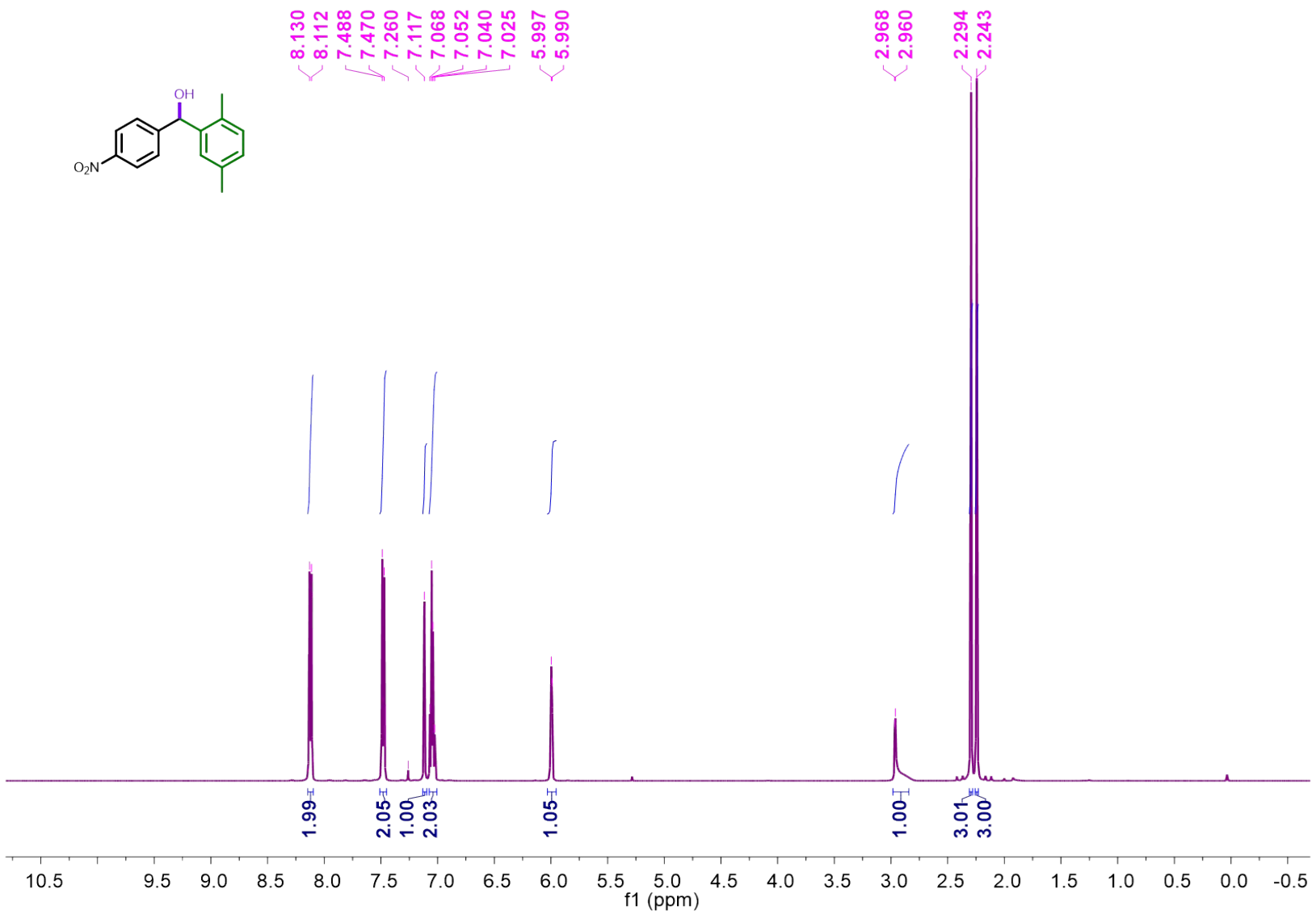


Figure S162. ^{1}H NMR spectra of (2,5-dimethylphenyl) (4-nitrophenyl)methanol **(3i)** in $CDCl_3$.

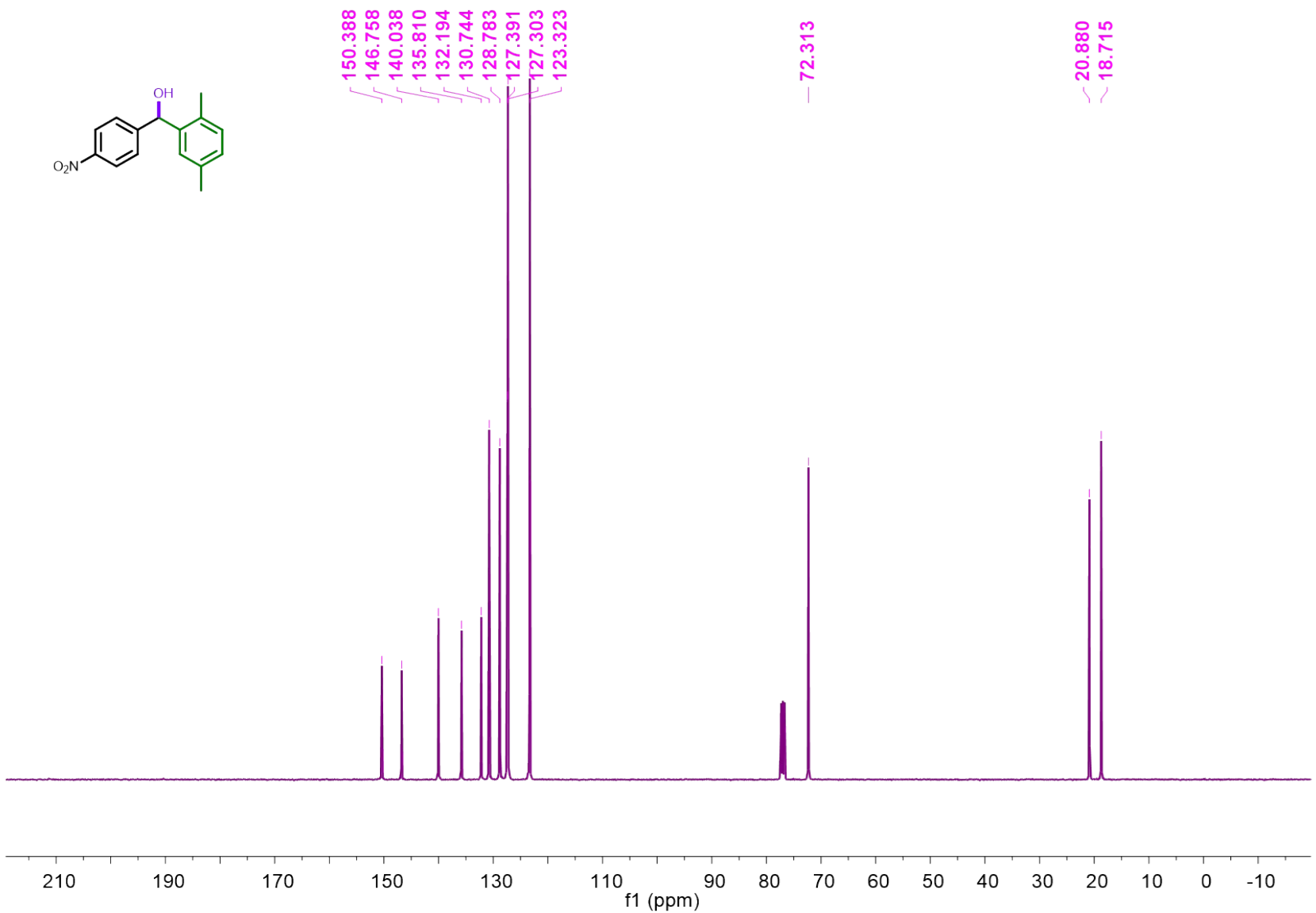


Figure S163. ^{13}C NMR spectra of (2,5-dimethylphenyl) (4-nitrophenyl)methanol **(3i)** in $CDCl_3$.

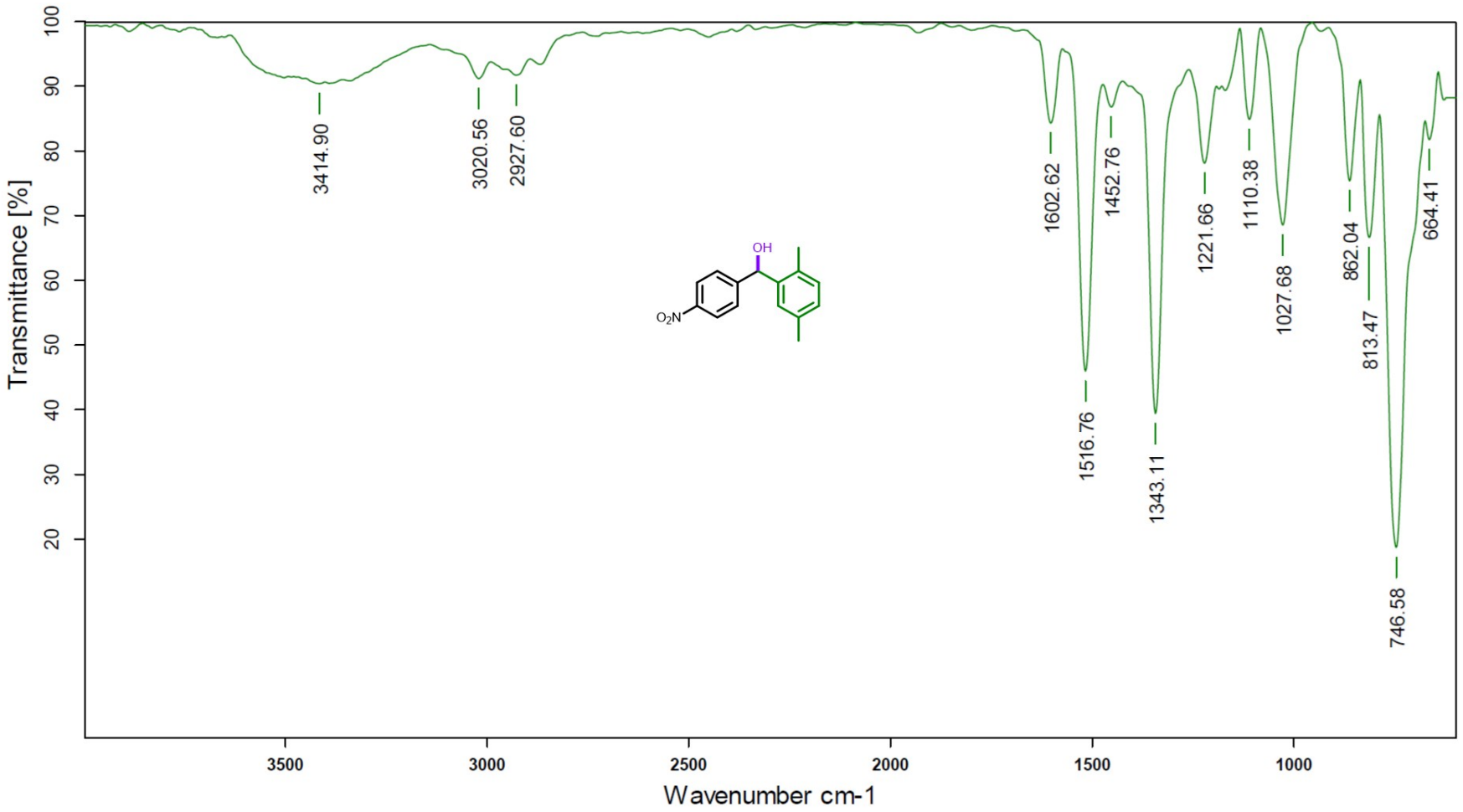


Figure S164. IR spectra of (2,5-dimethylphenyl) (4-nitrophenyl)methanol **(3i)**.

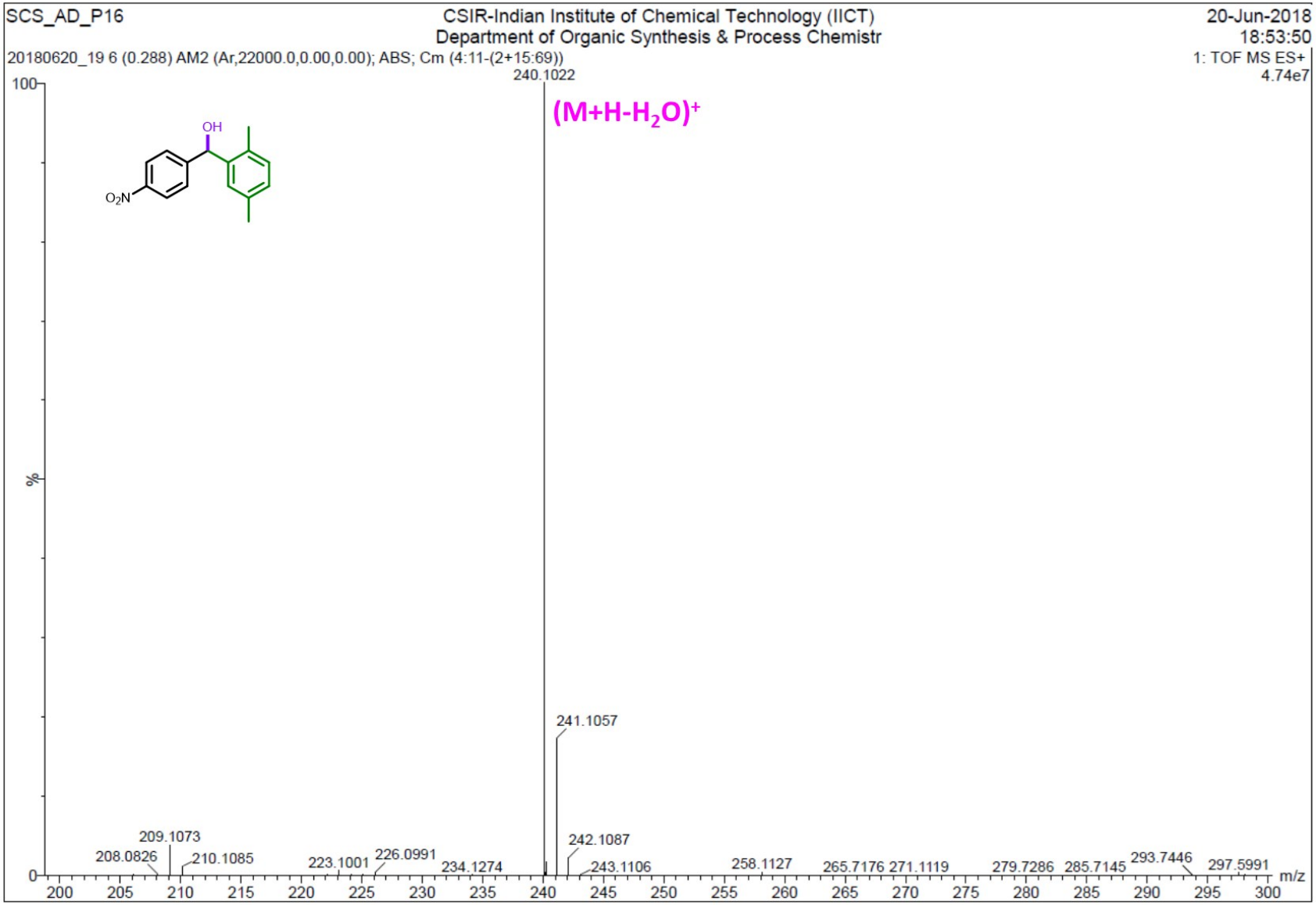
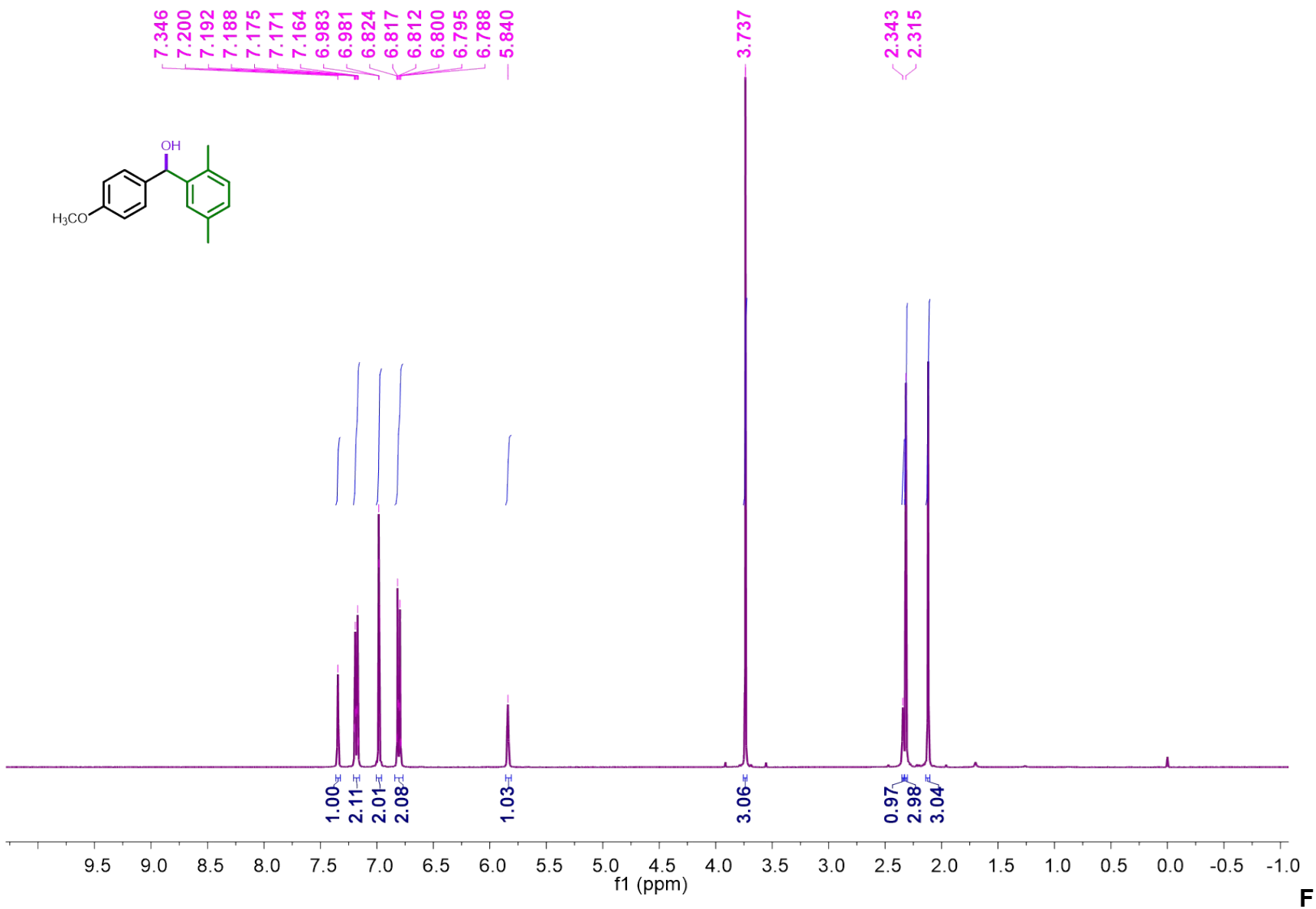


Figure S165. HRMS spectra of (2,5-dimethylphenyl) (4-nitrophenyl)methanol **(3i)**.



F

igure S166. ^{1}H NMR spectra of (2, 5-dimethylphenyl) (4-methoxyphenyl) methanol **(3j)** in $CDCl_3$.

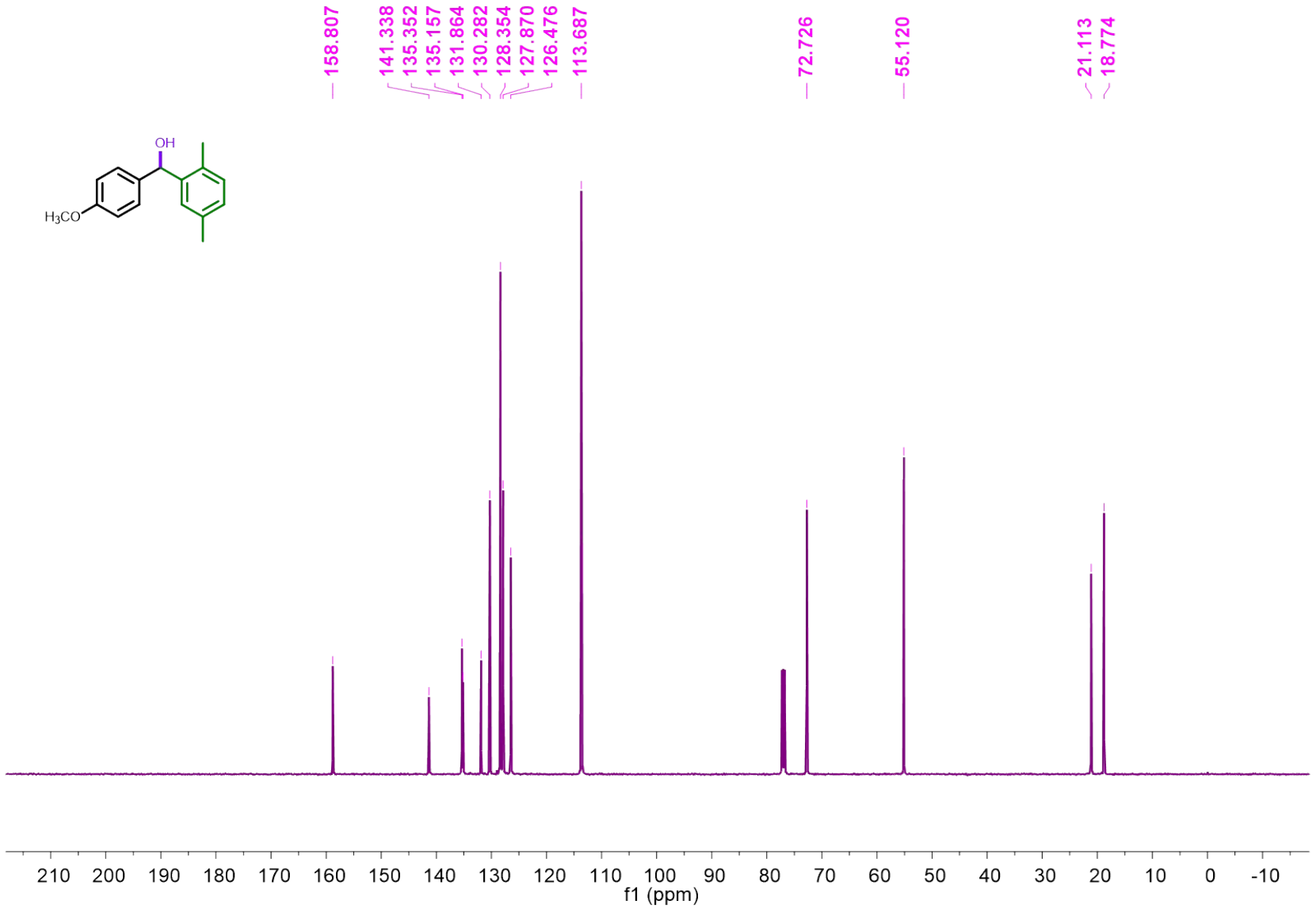


Figure S167. ^{13}C NMR spectra of (2, 5-dimethylphenyl) (4-methoxyphenyl) methanol **(3j)** in $CDCl_3$.

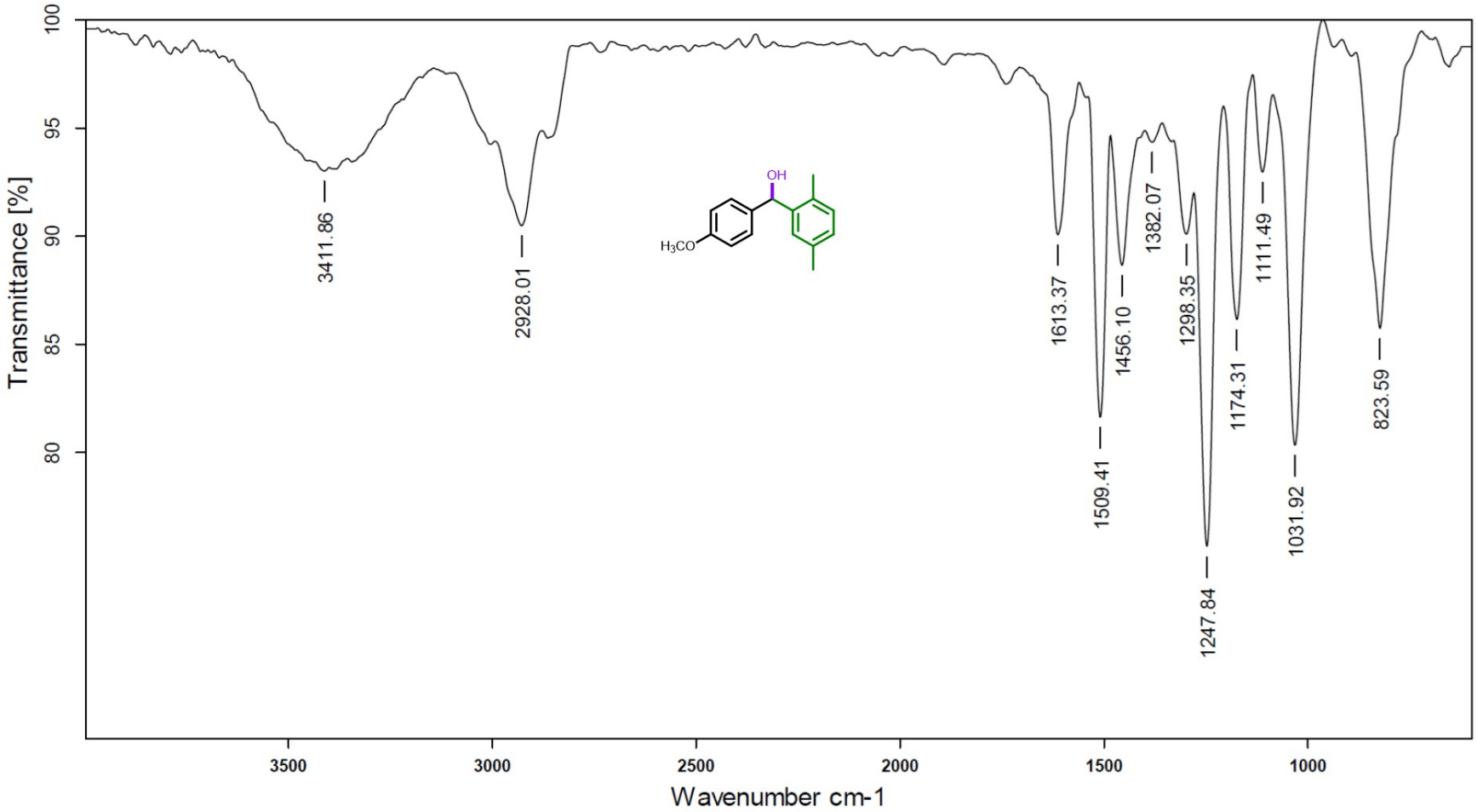


Figure S168. IR spectra of (2, 5-dimethylphenyl) (4-methoxyphenyl) methanol **(3j)**.

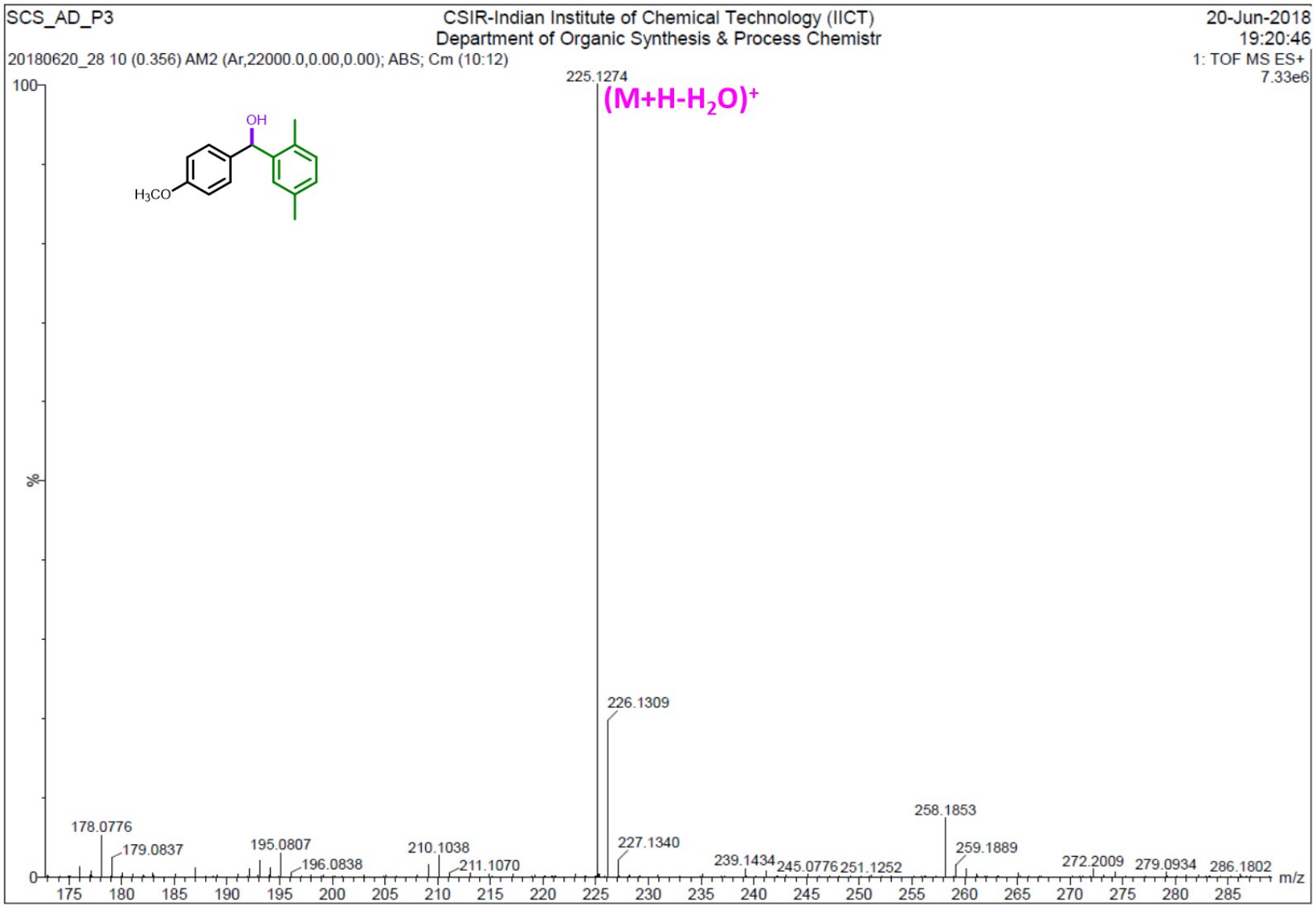


Figure S169. HRMS spectra of (2, 5-dimethylphenyl) (4-methoxyphenyl) methanol **(3j)**.

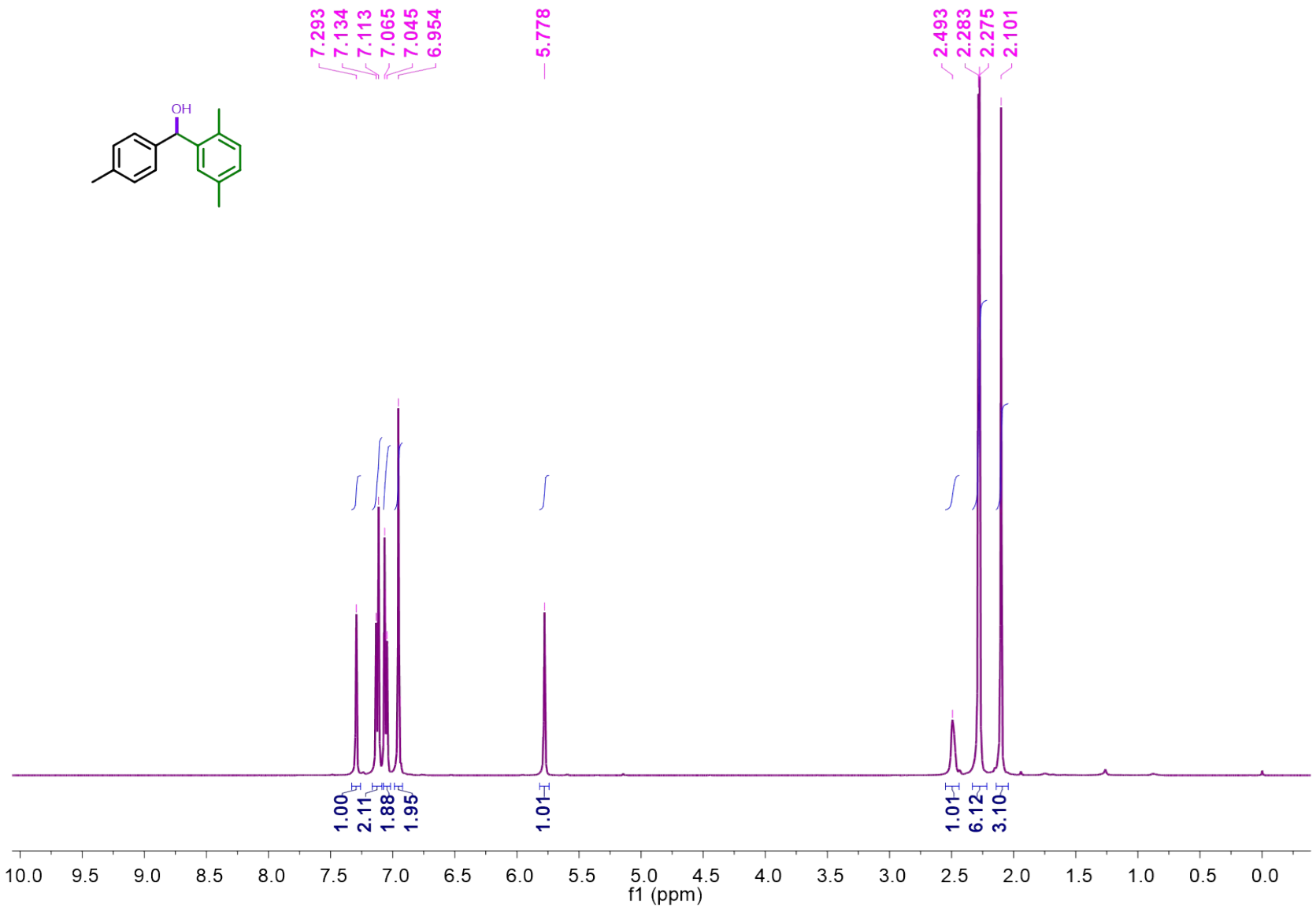


Figure S170. ^{1}H NMR spectra of (2, 5-dimethylphenyl) (4-methylphenyl) methanol **(3k)** in $CDCl_3$.

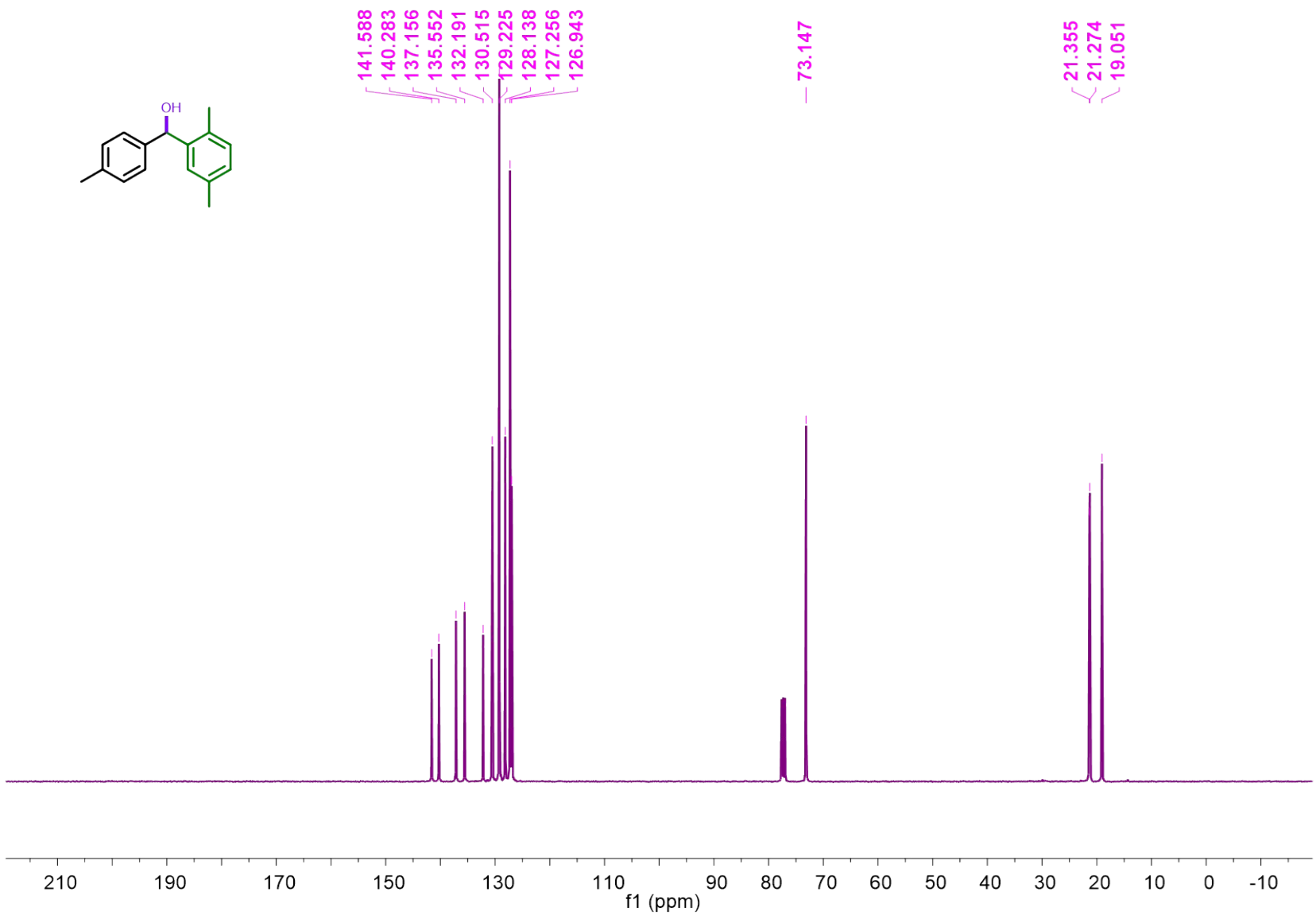


Figure S171. ^{13}C NMR spectra of (2, 5-dimethylphenyl) (4-methylphenyl) methanol **(3k)** in $CDCl_3$.

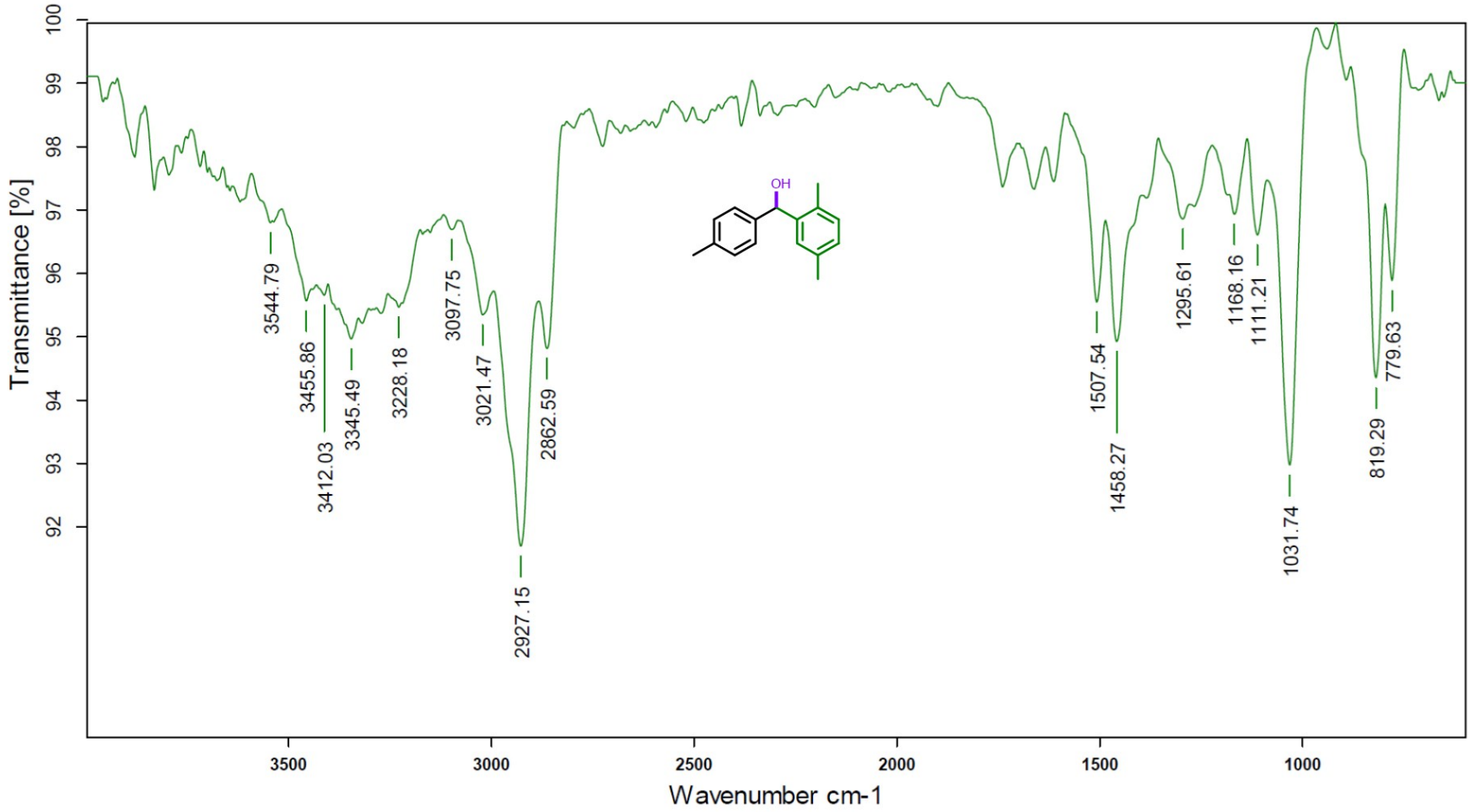


Figure S172. IR spectra of (2, 5-dimethylphenyl) (4-methylphenyl) methanol **(3k)**.

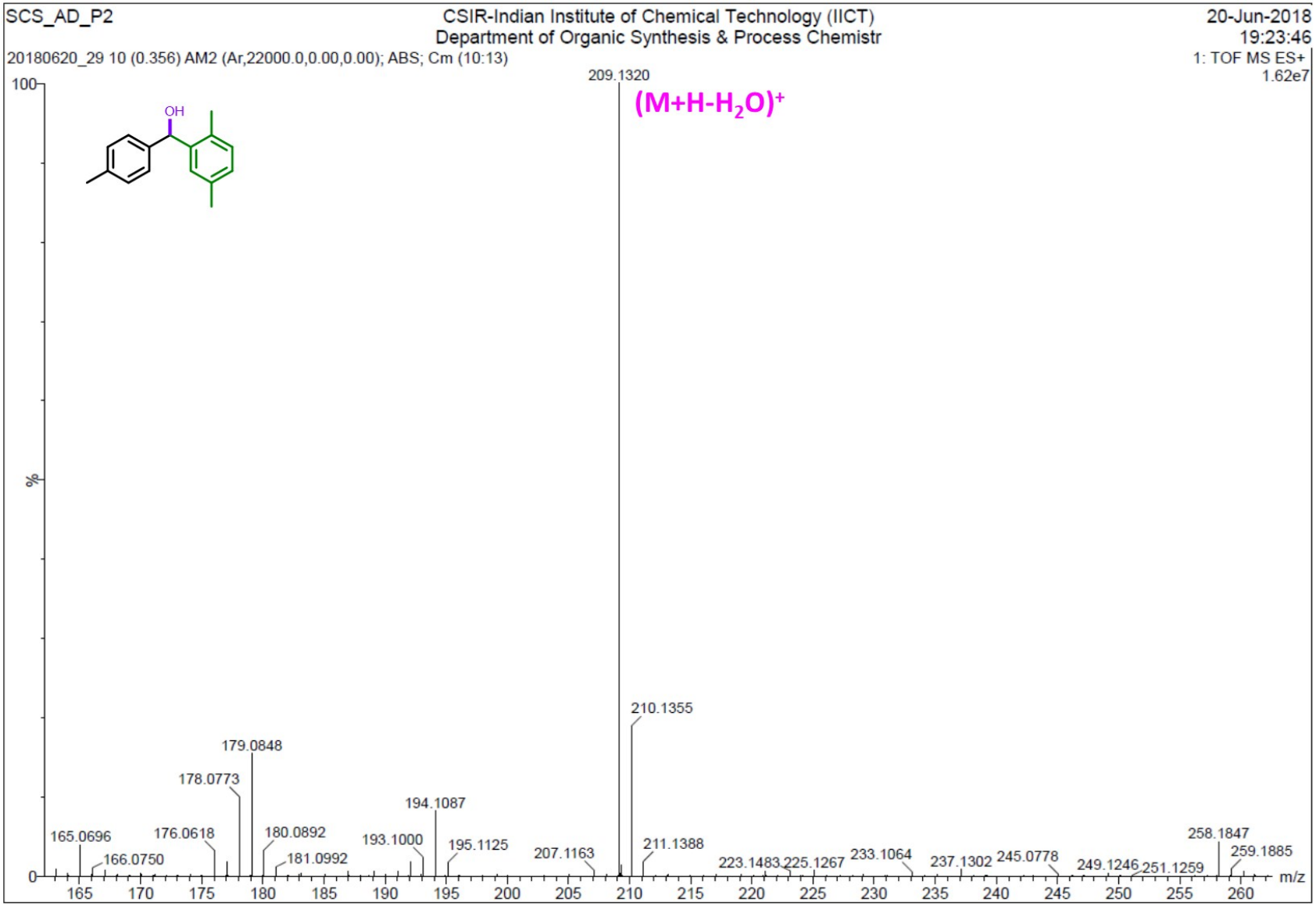


Figure S173. HRMS spectra of (2, 5-dimethylphenyl) (4-methylphenyl) methanol **(3k)**.

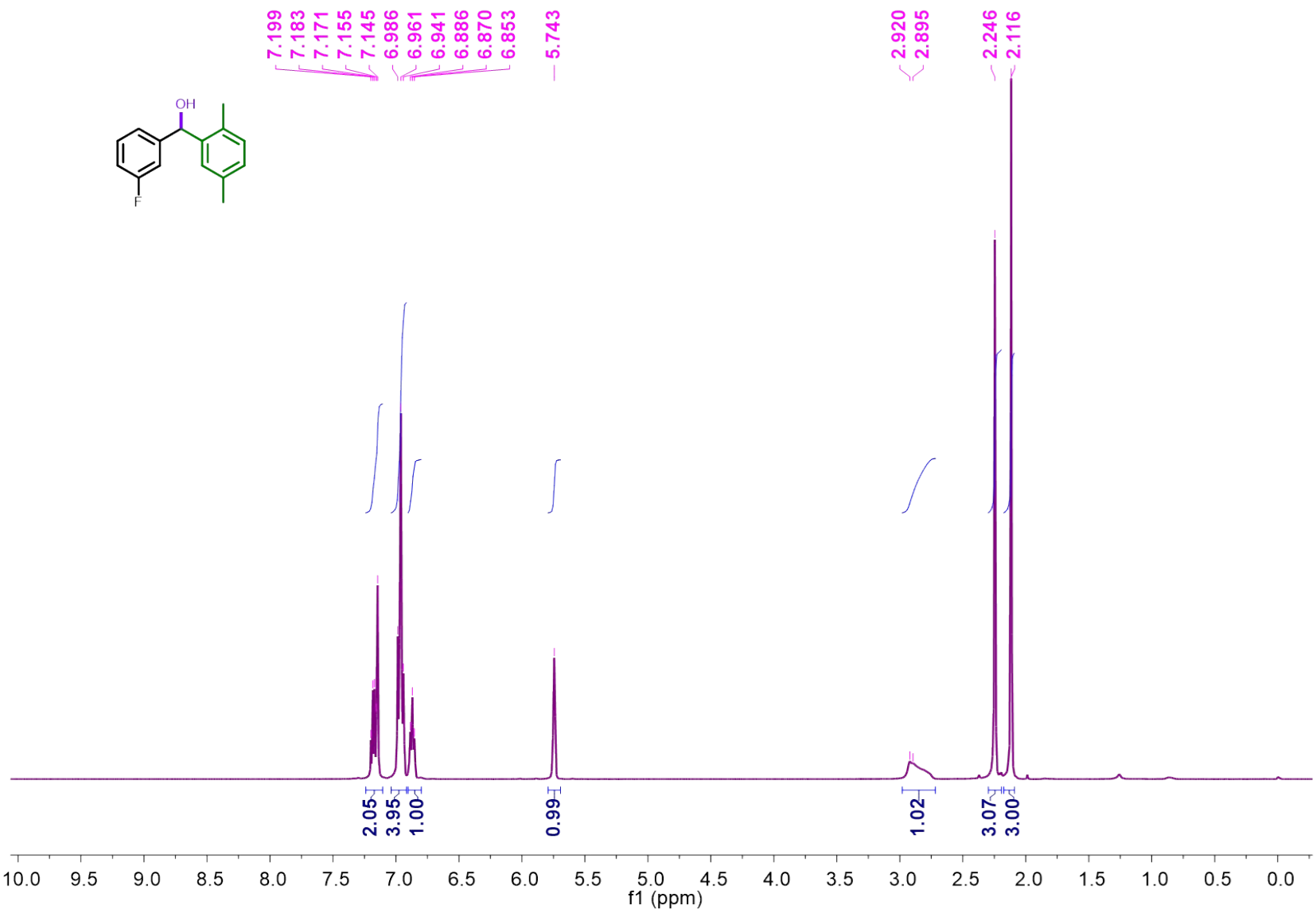


Figure S174. ^{1}H NMR spectra of (2, 5-dimethylphenyl) (3-fluorophenyl) methanol **(3l)** in $CDCl_3$.

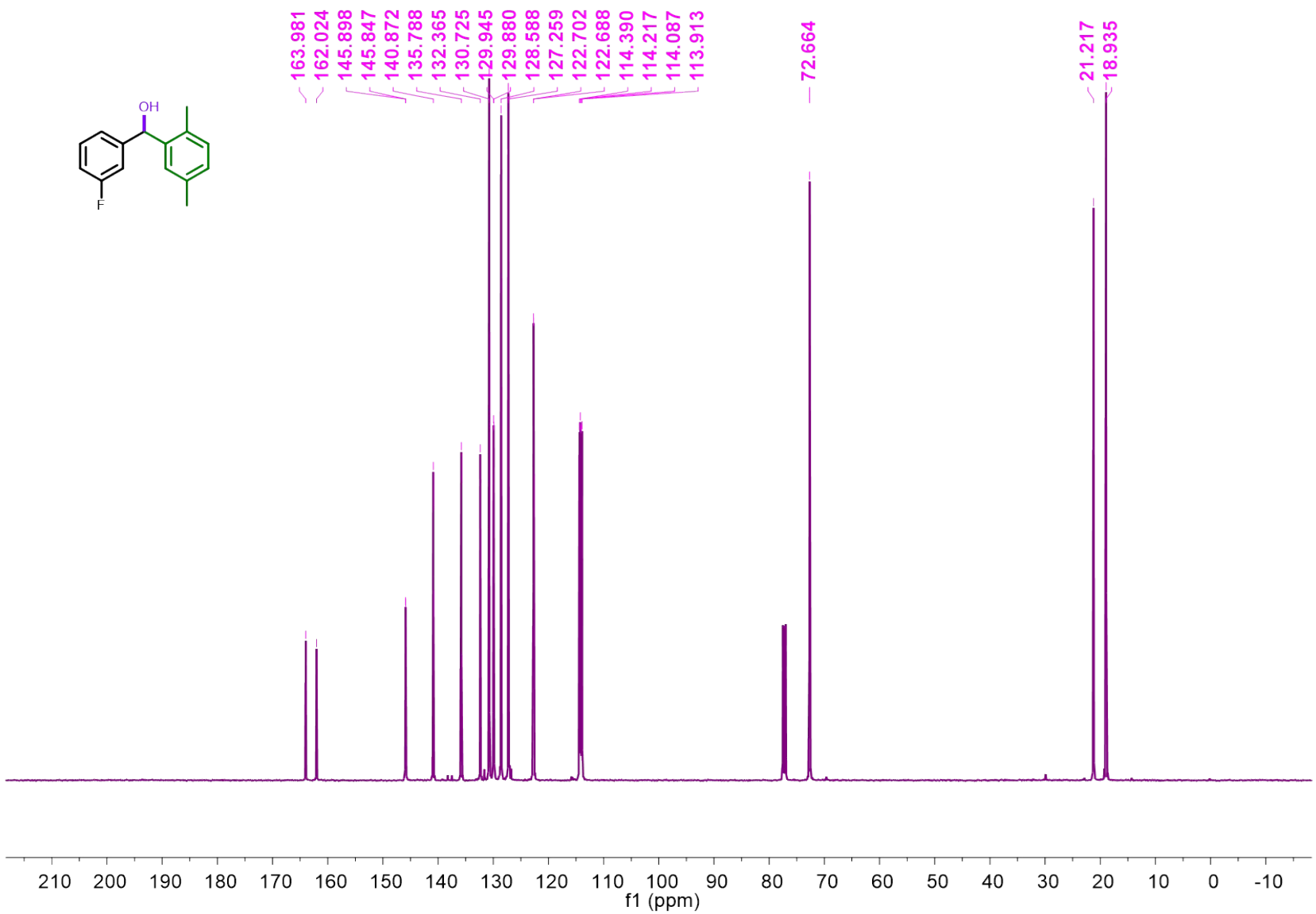


Figure S175. ^{13}C NMR spectra of (2, 5-dimethylphenyl) (3-fluorophenyl) methanol **(3l)** in $CDCl_3$.

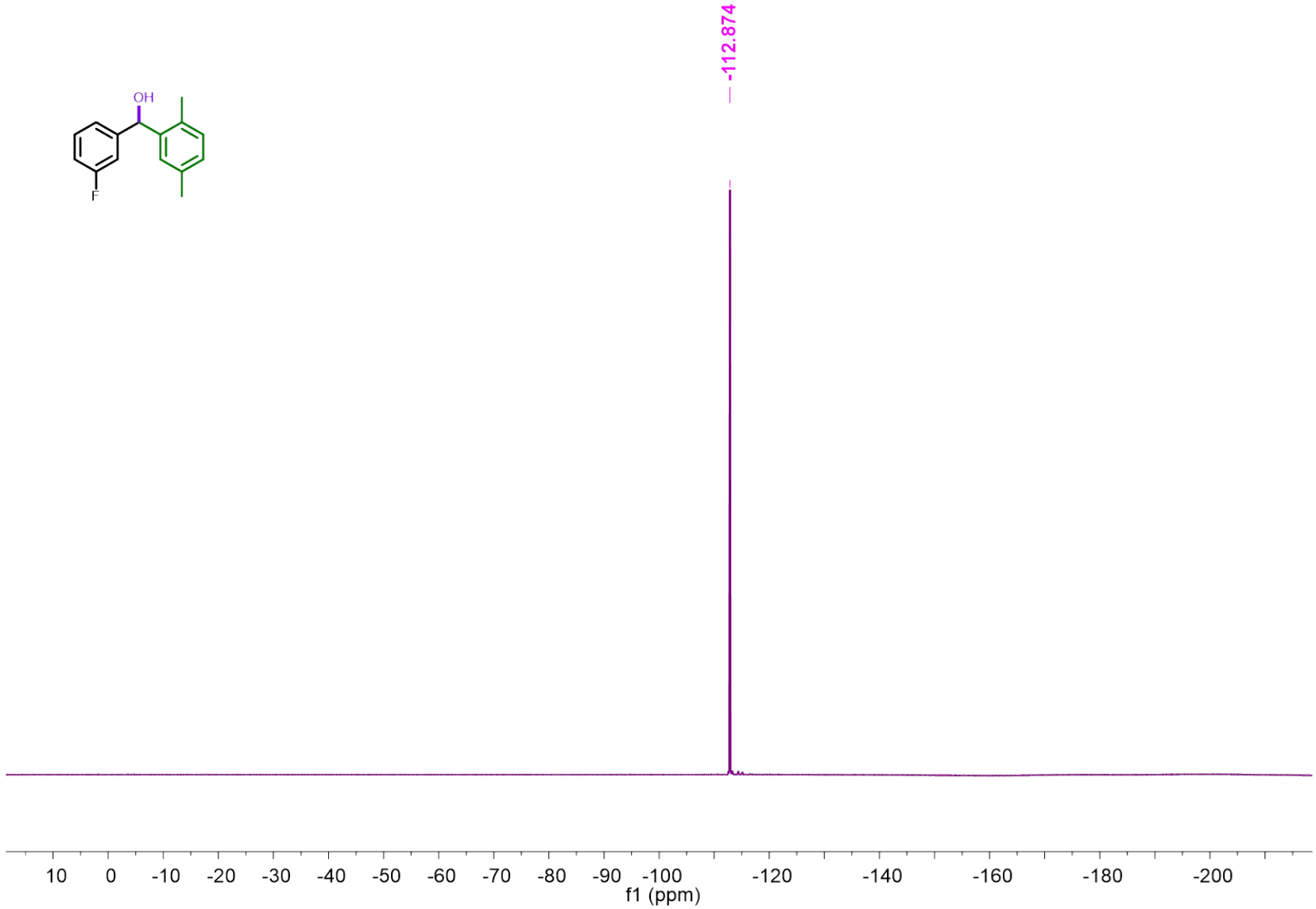


Figure S176. ^{19}F NMR spectra of (2, 5-dimethylphenyl) (3-fluorophenyl) methanol **(3l)** in $CDCl_3$.

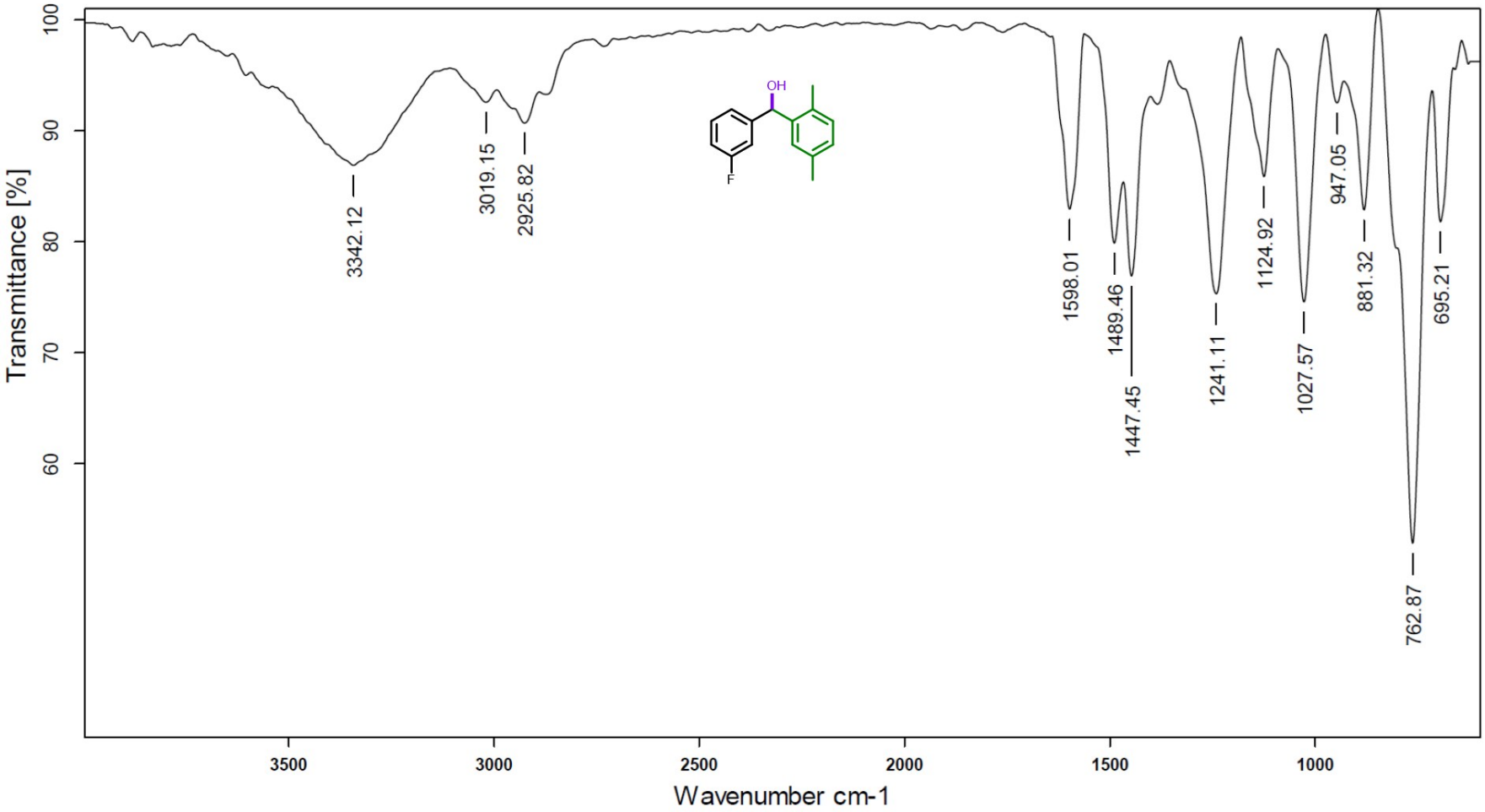


Figure S177. IR spectra of (2, 5-dimethylphenyl) (3-fluorophenyl) methanol **(3l**).

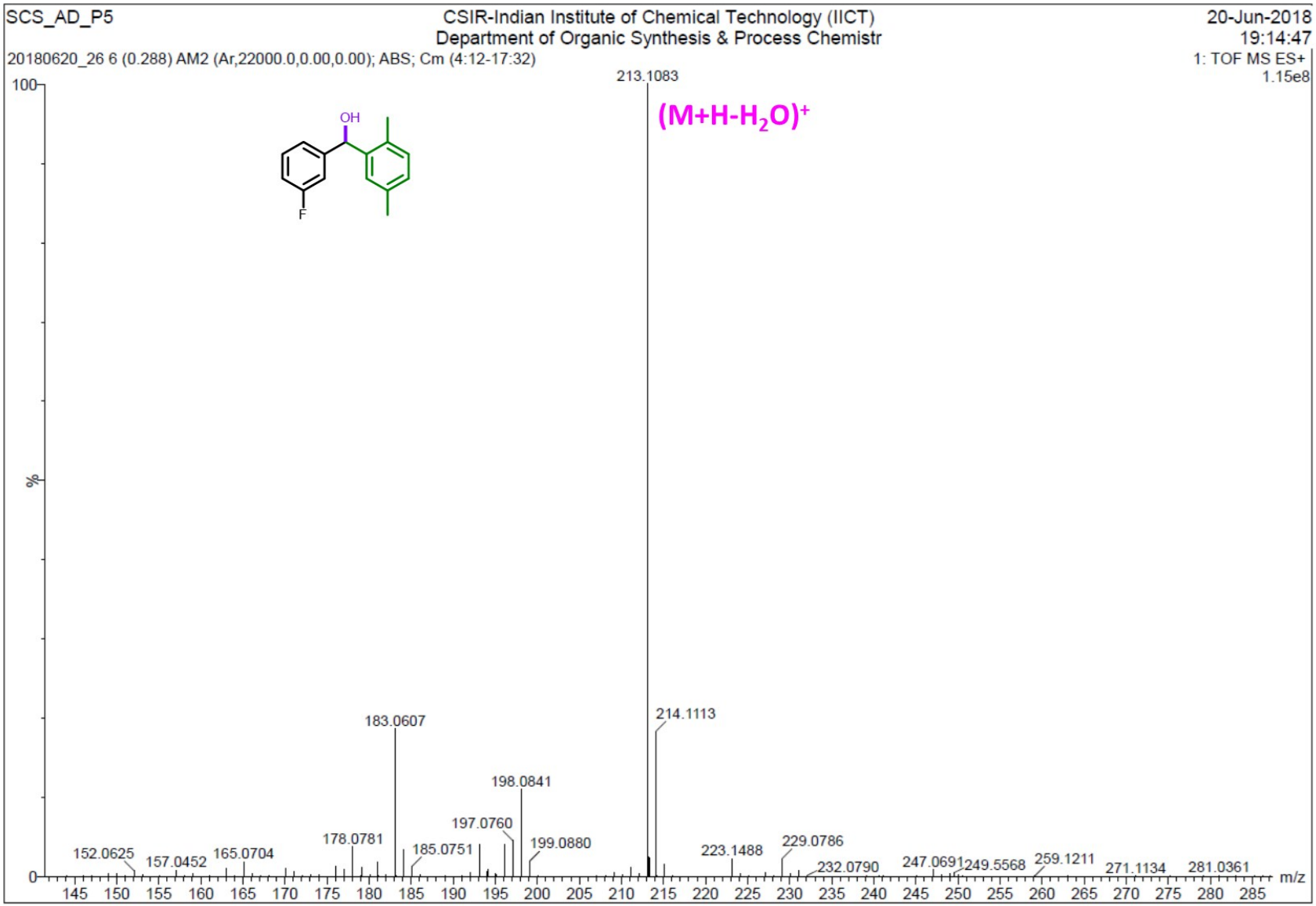


Figure S178. HRMS spectra of (2, 5-dimethylphenyl) (3-fluorophenyl) methanol **(3l**).

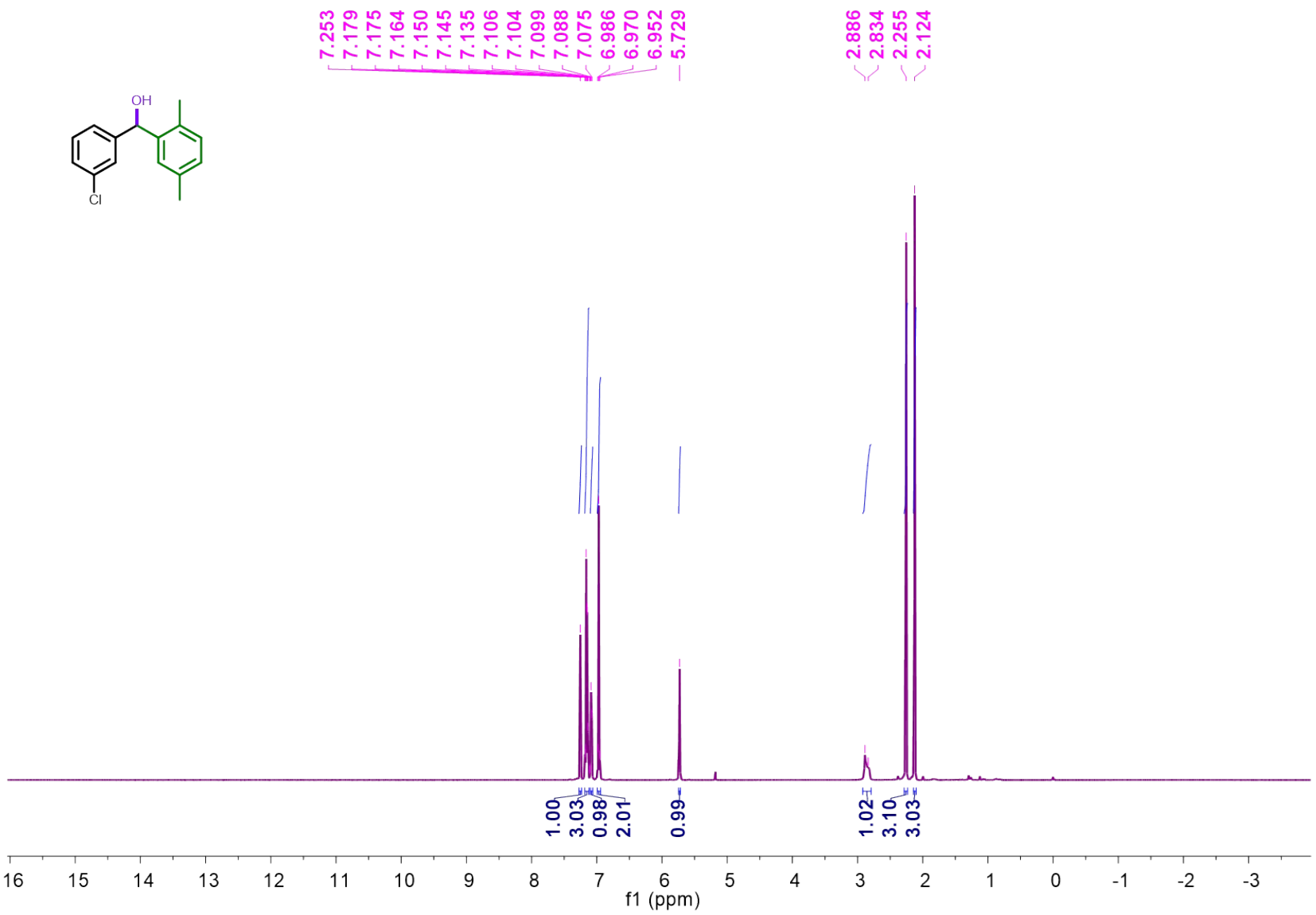


Figure S179. ^{1}H NMR spectra of (2, 5-dimethylphenyl) (3-chlorophenyl) methanol **(3m)** in $CDCl_3$.

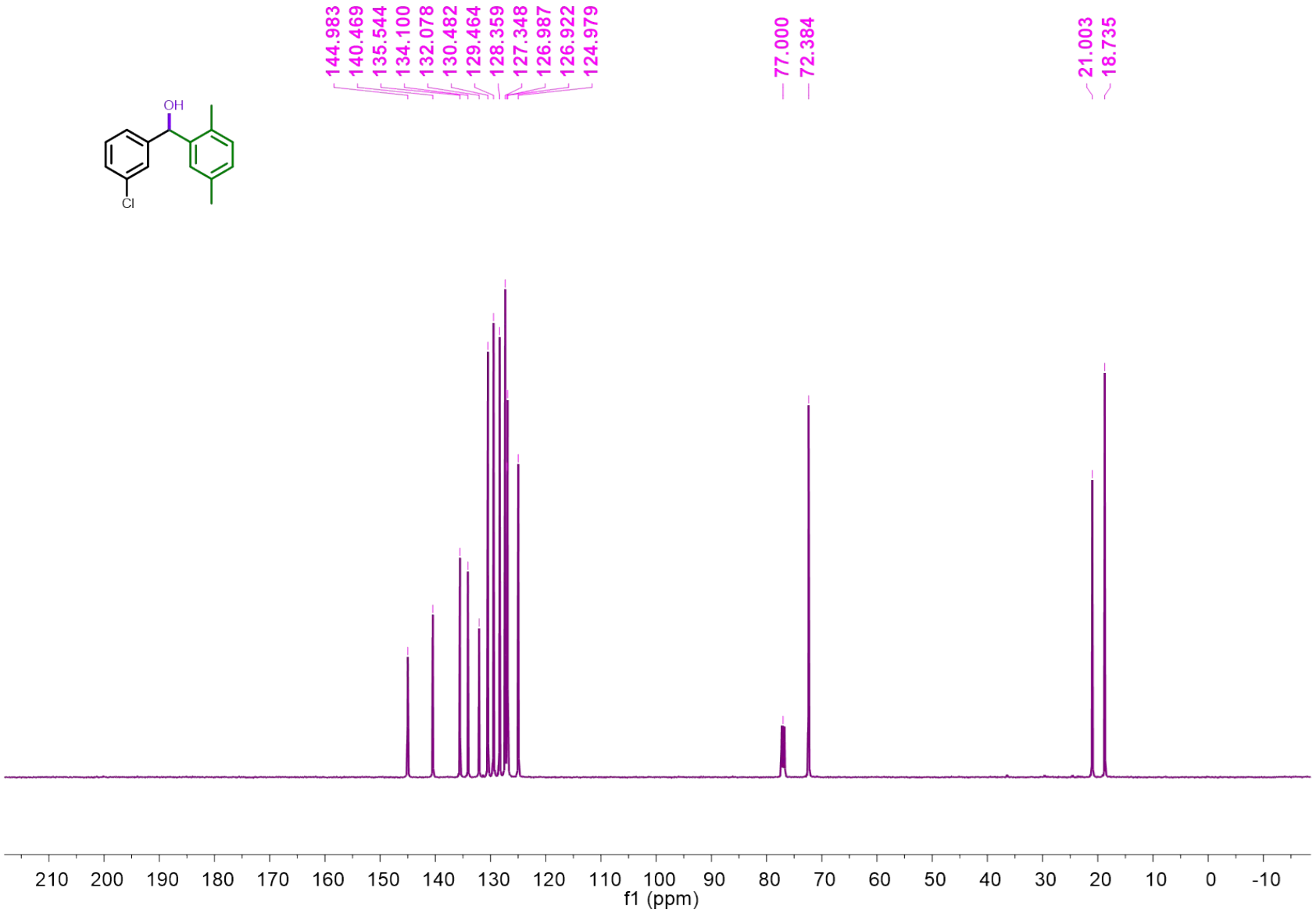


Figure S180. ^{13}C NMR spectra of (2, 5-dimethylphenyl) (3-chlorophenyl) methanol **(3m)** in $CDCl_3$.

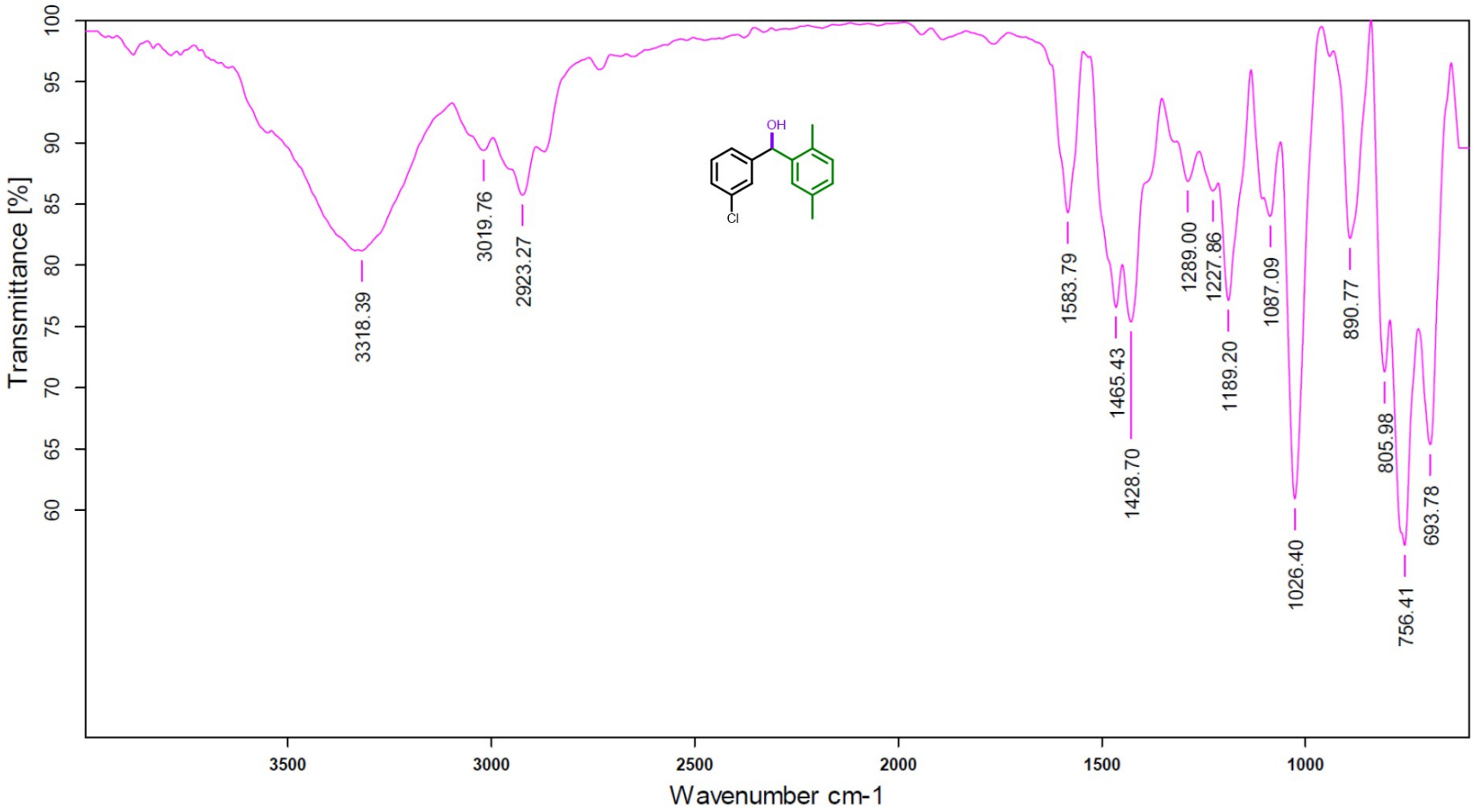


Figure S181. IR spectra of (2, 5-dimethylphenyl) (3-chlorophenyl) methanol **(m)**.

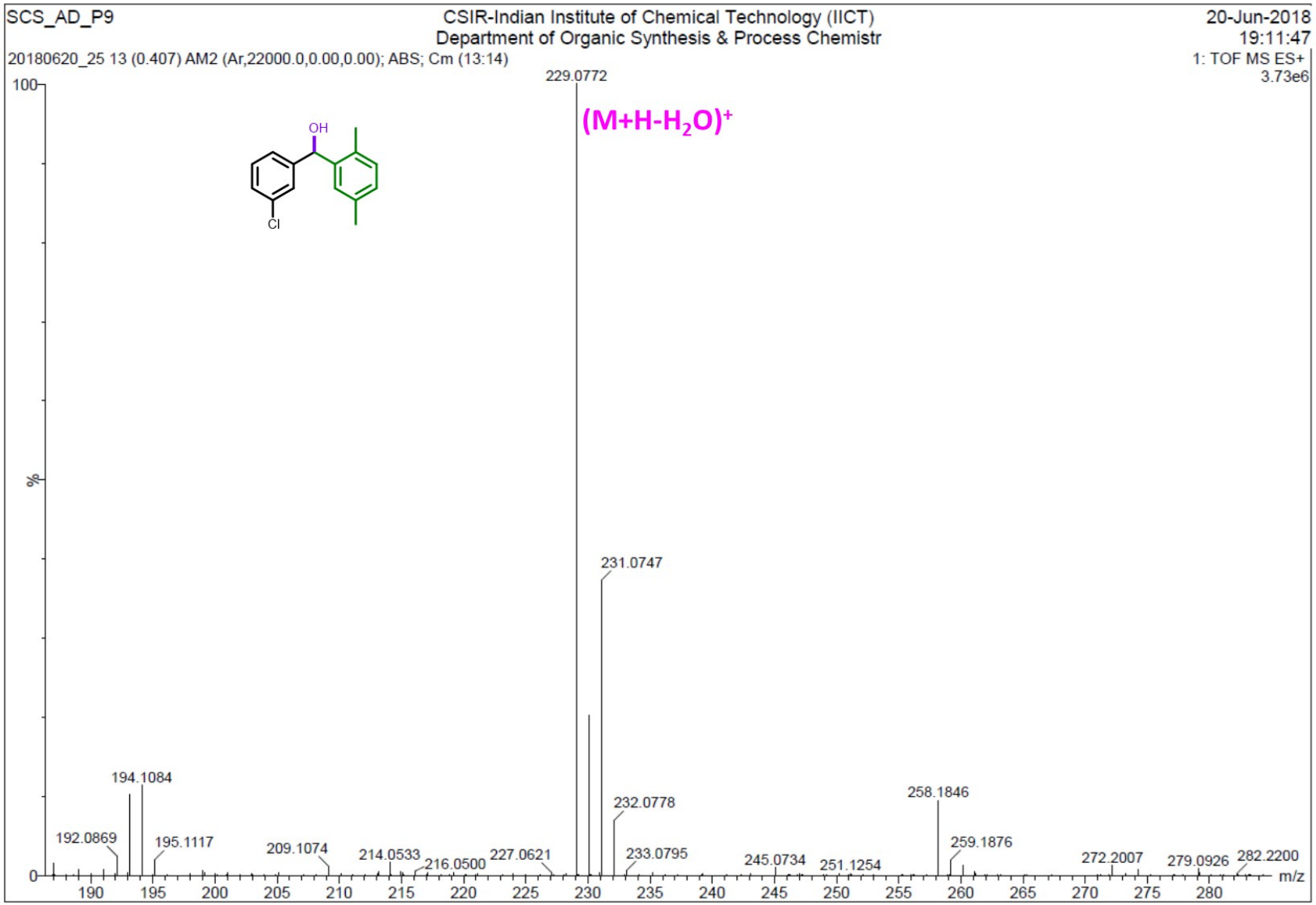


Figure S182. HRMS spectra of (2, 5-dimethylphenyl) (3-chlorophenyl) methanol **(3m)**.

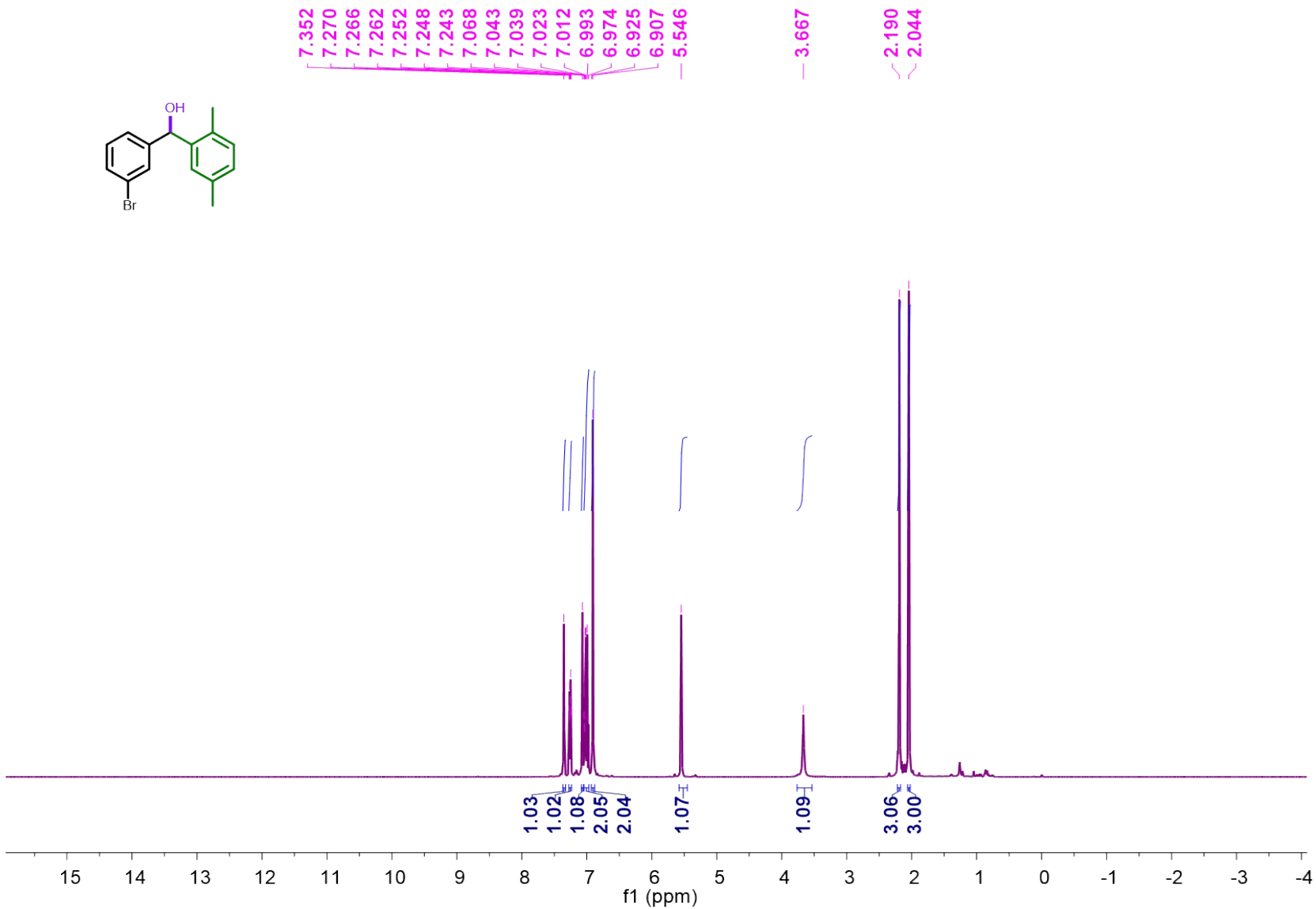


Figure S183. ^{1}H NMR spectra of (2, 5-dimethylphenyl) (3-bromophenyl) methanol **(3n)** in $CDCl_3$.

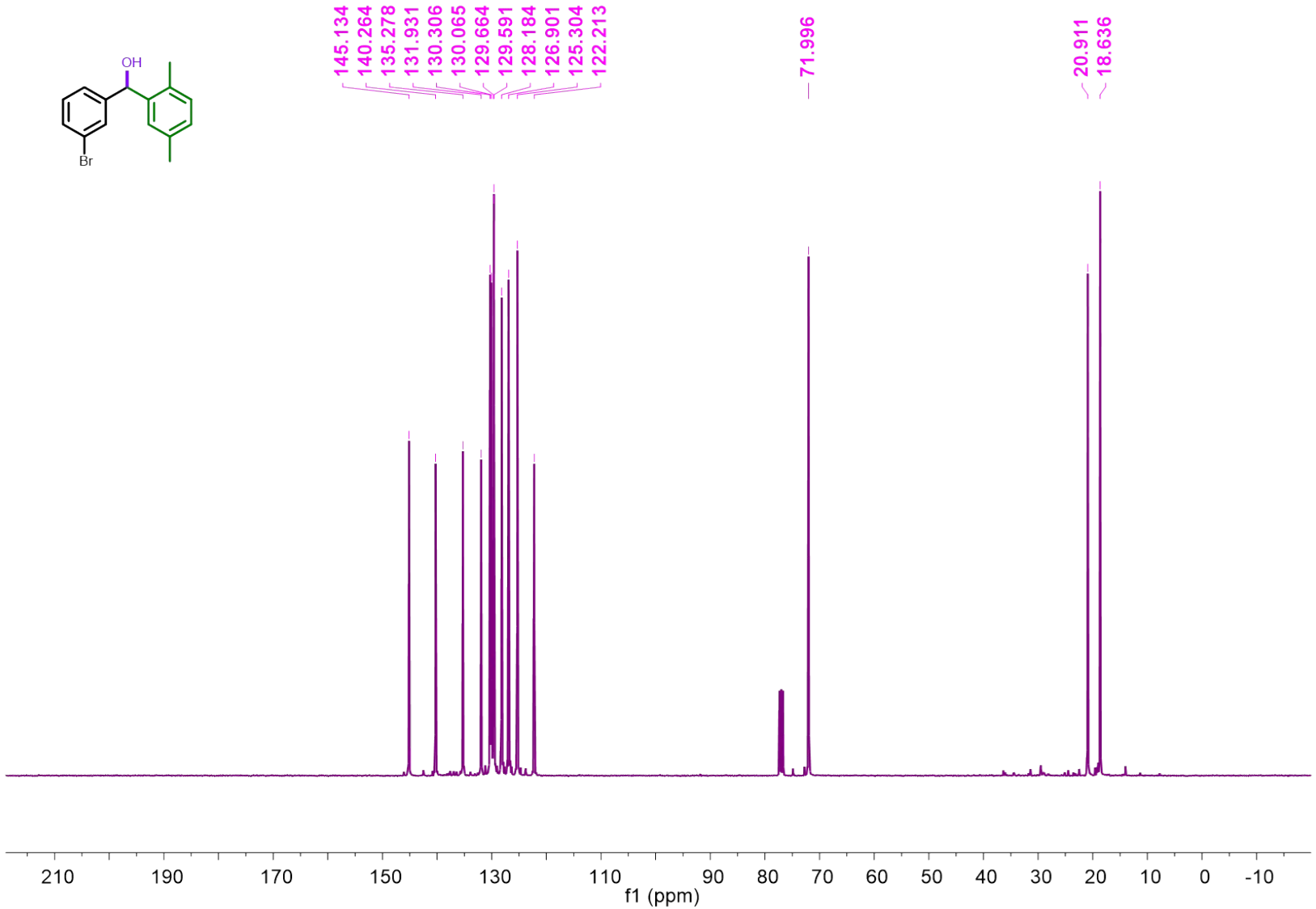


Figure S184. ^{13}C NMR spectra of (2, 5-dimethylphenyl) (3-bromophenyl) methanol **(3n)** in $CDCl_3$.

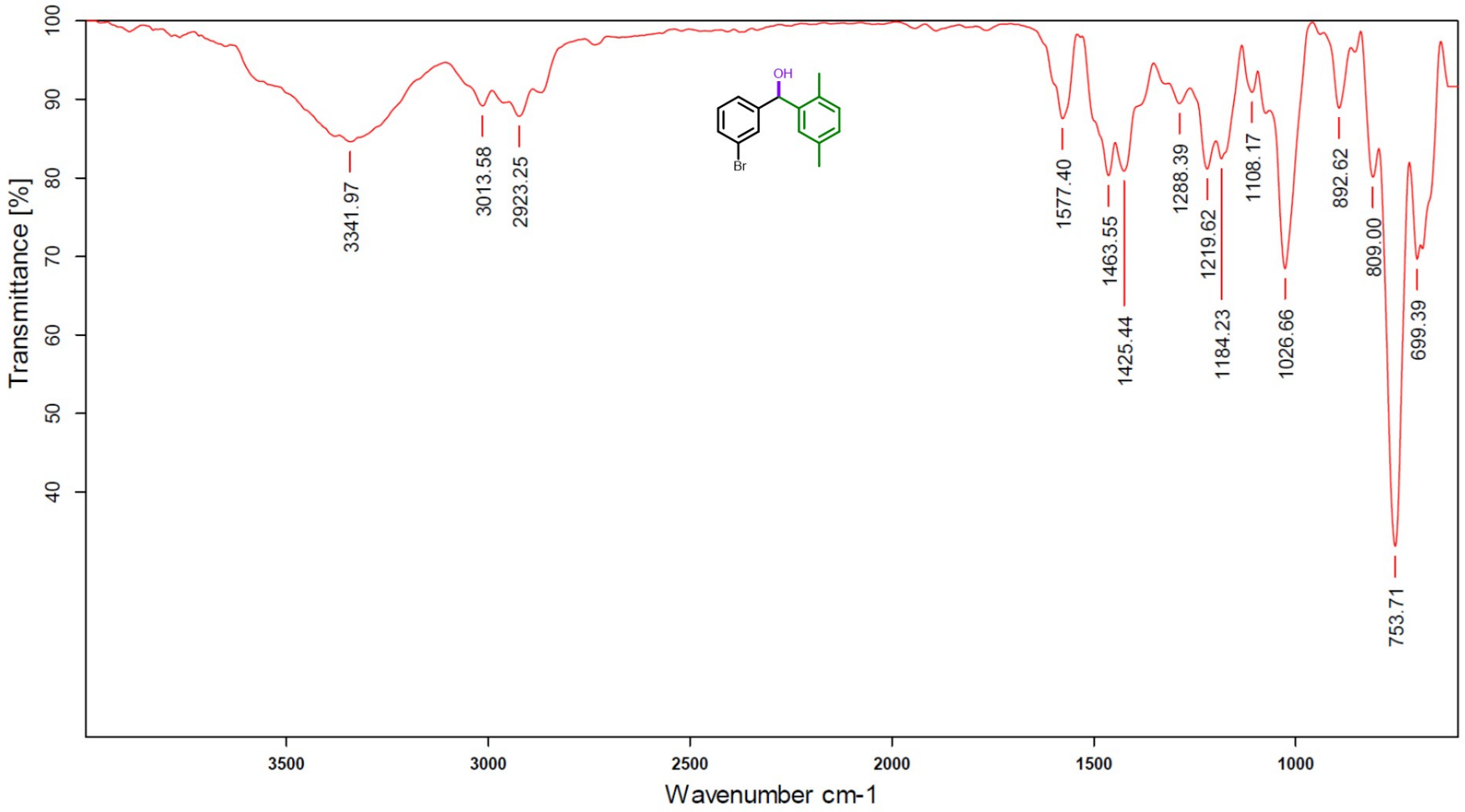


Figure S185. IR spectra of (2, 5-dimethylphenyl) (3-bromophenyl) methanol **(3n**).

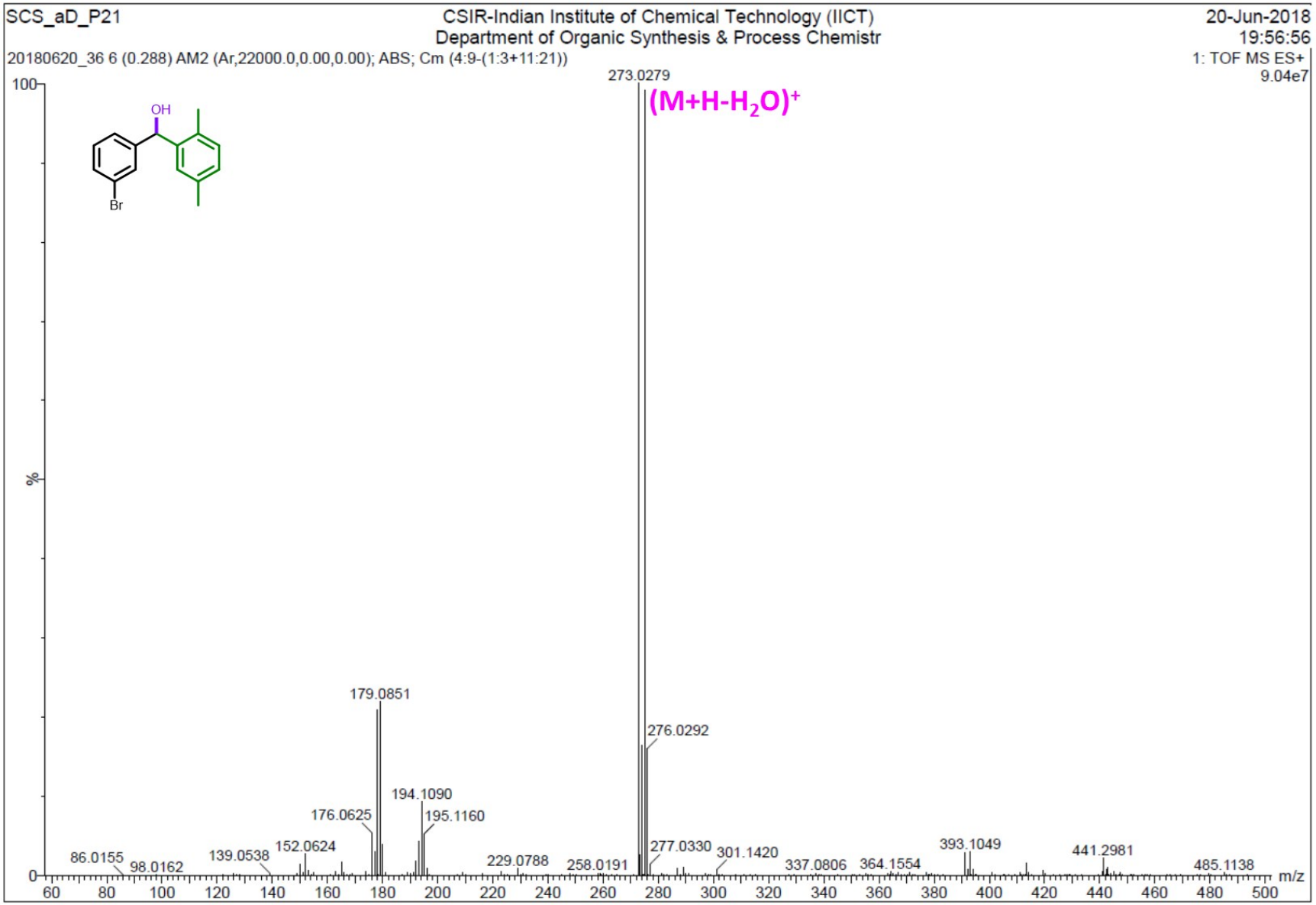


Figure S186. HRMS spectra of (2, 5-dimethylphenyl) (3-bromophenyl) methanol **(3n)**.

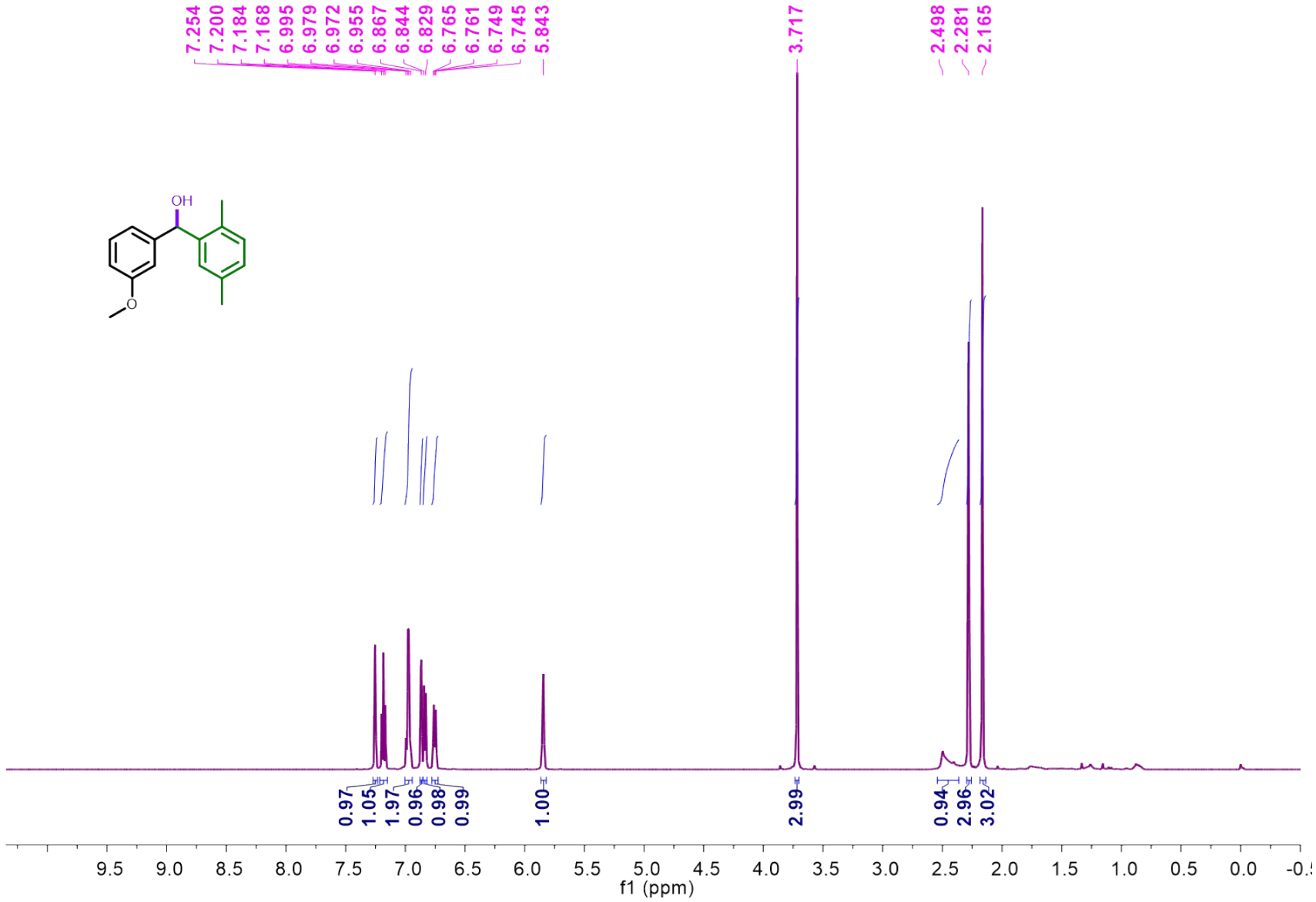


Figure S187. HRMS spectra of (2, 5-dimethylphenyl) (3-bromophenyl) methanol **(3o)**.

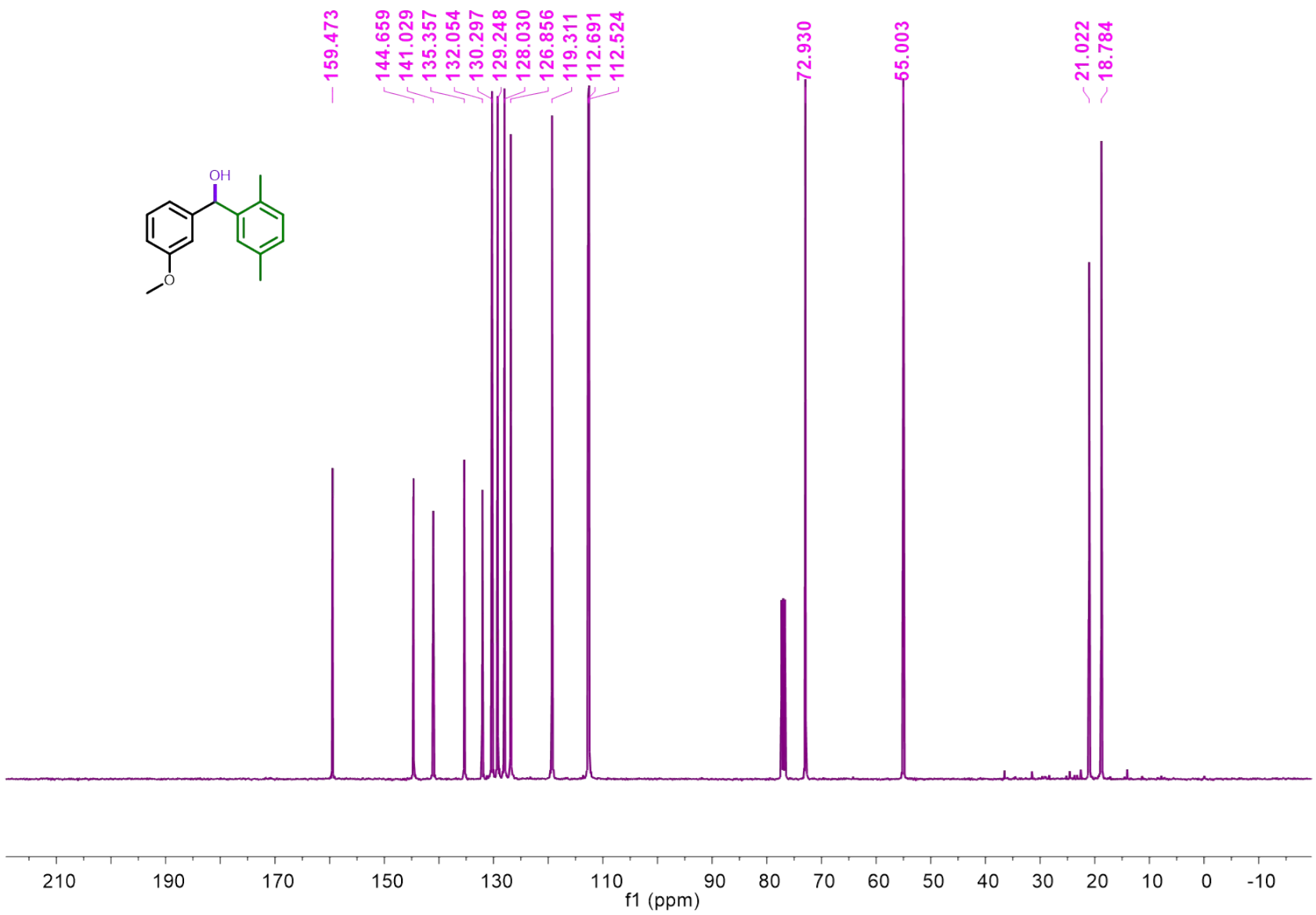


Figure S188. HRMS spectra of (2, 5-dimethylphenyl) (3-bromophenyl) methanol **(3o)**.

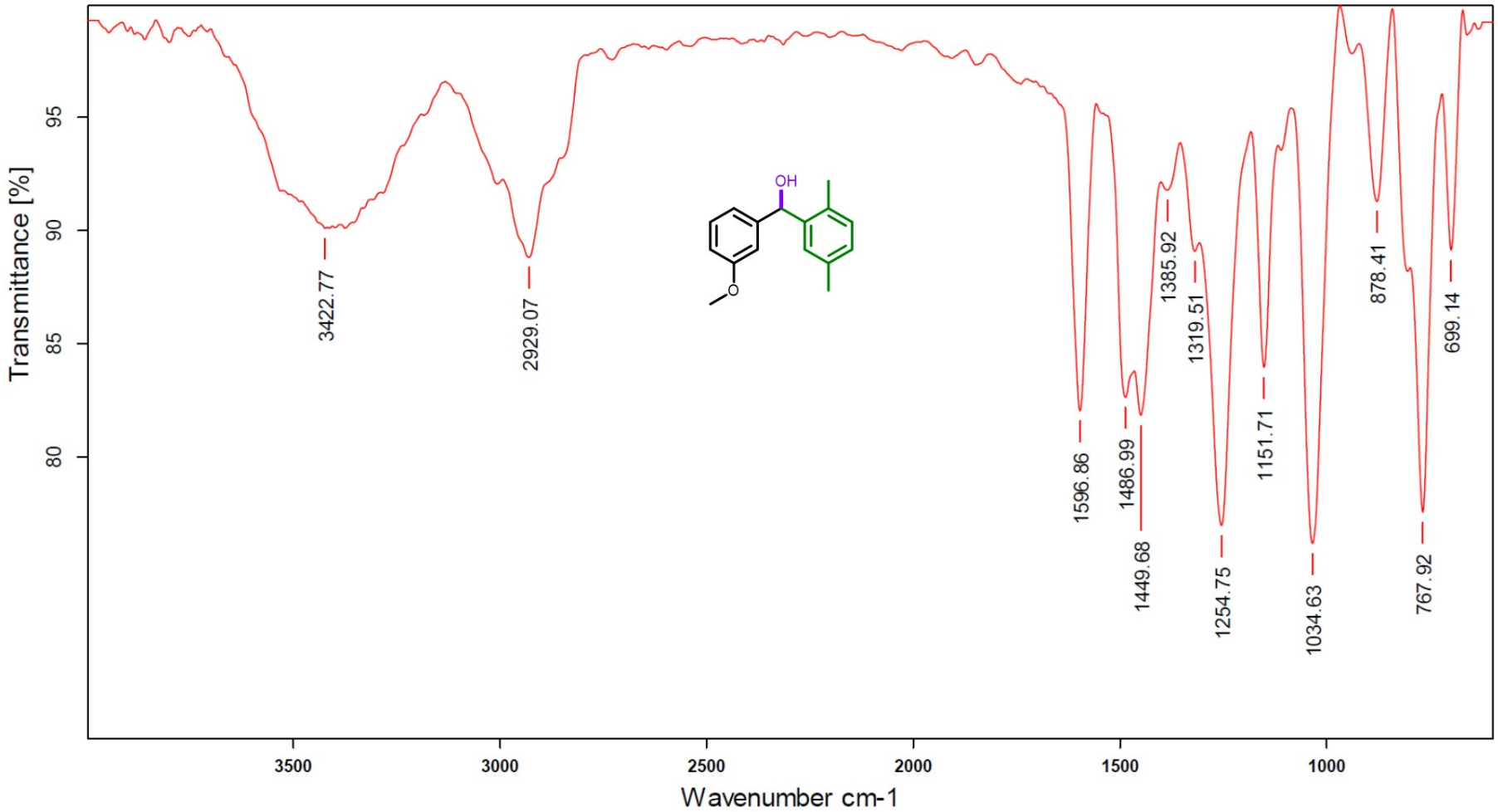


Figure S189. HRMS spectra of (2, 5-dimethylphenyl) (3-bromophenyl) methanol **(3o)**.

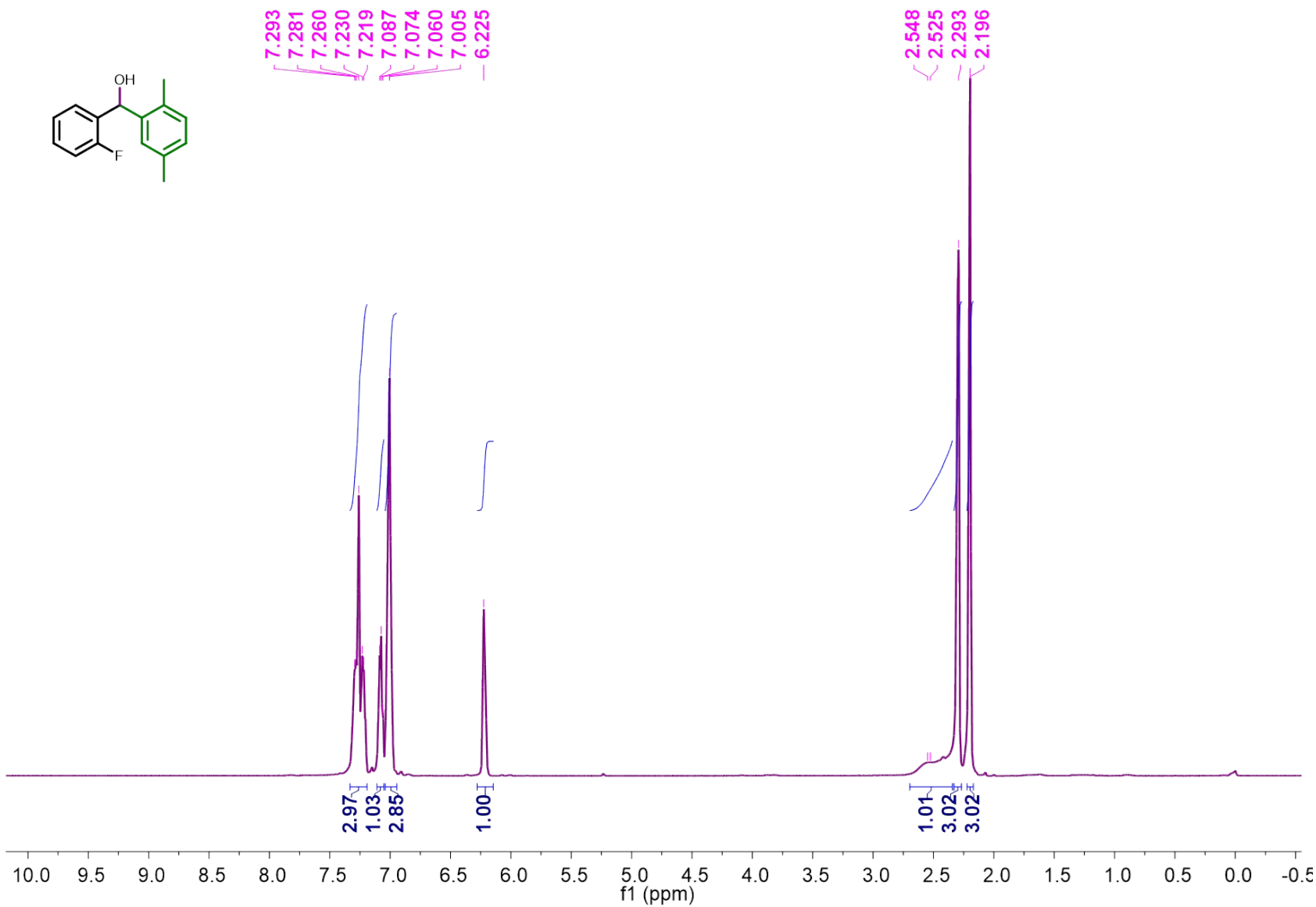
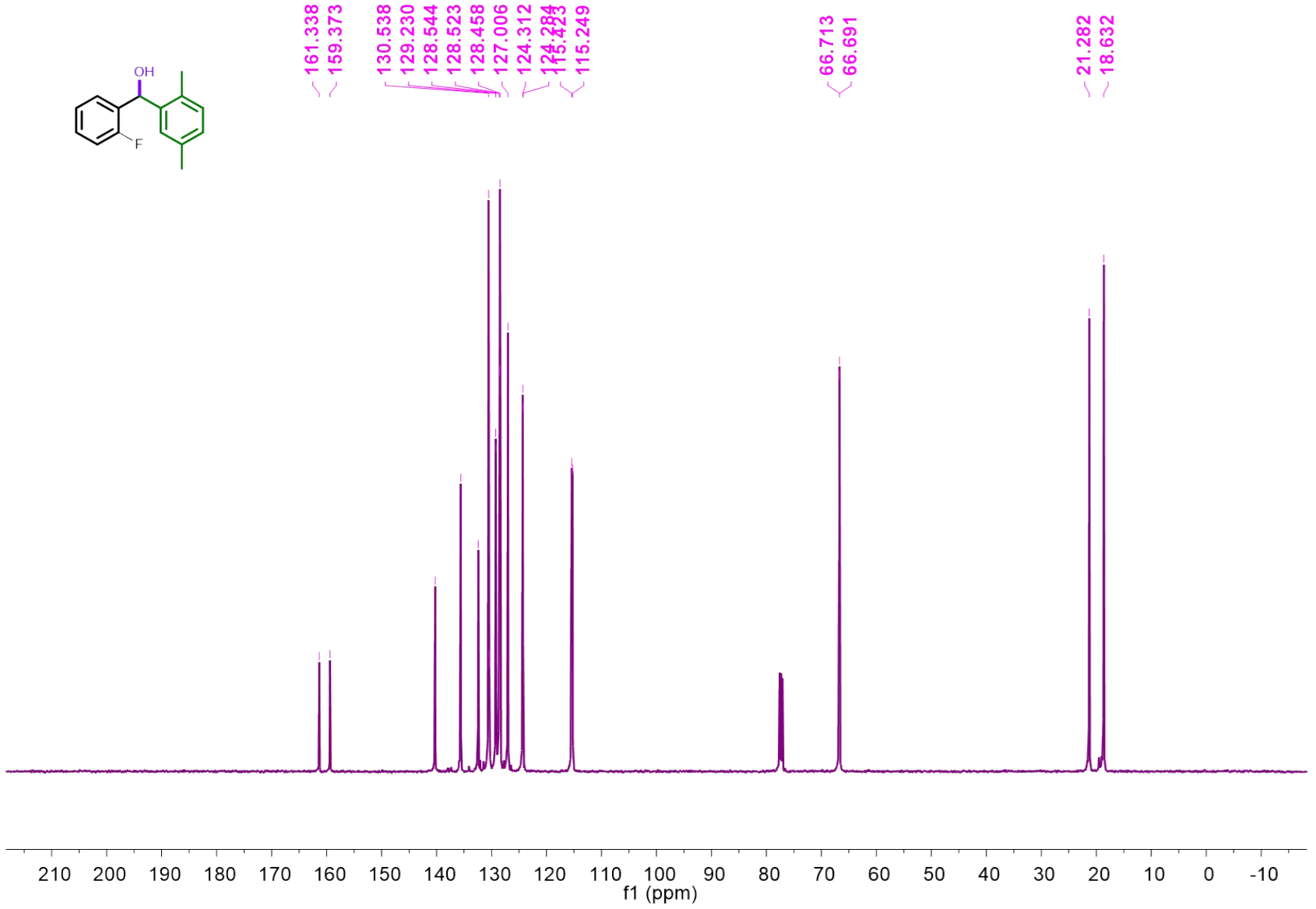


Figure S190. ^{1}H NMR spectra of (2, 5-dimethylphenyl) (2-fluorophenyl) methanol **(3p)** in $CDCl_3$.



F

igure S191. ^{13}C NMR spectra of (2, 5-dimethylphenyl) (2-fluorophenyl) methanol **(3p)** in $CDCl_3$.

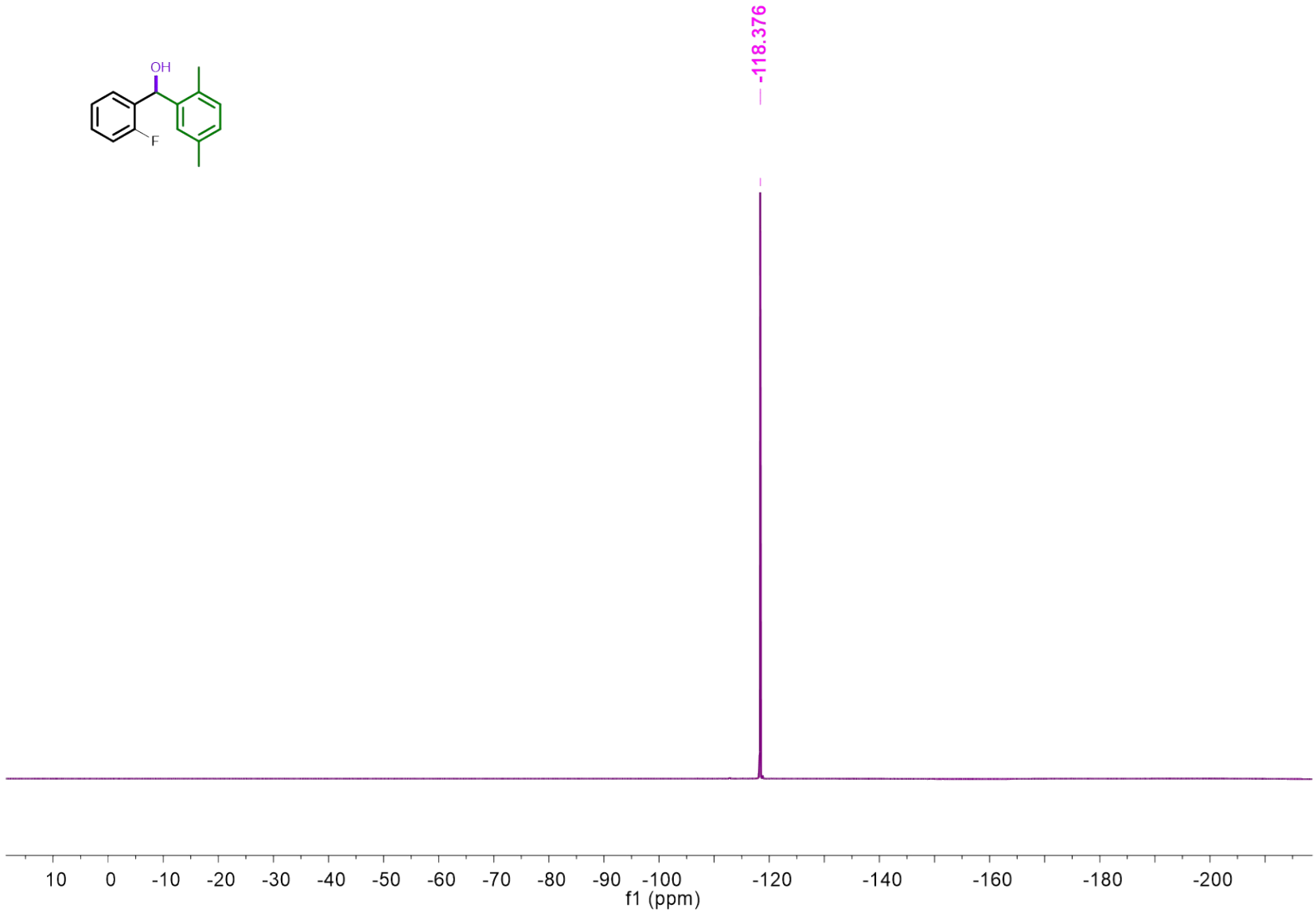


Figure S192. ^{19}F NMR spectra of (2, 5-dimethylphenyl) (2-fluorophenyl) methanol **(3p)** in $CDCl_3$.

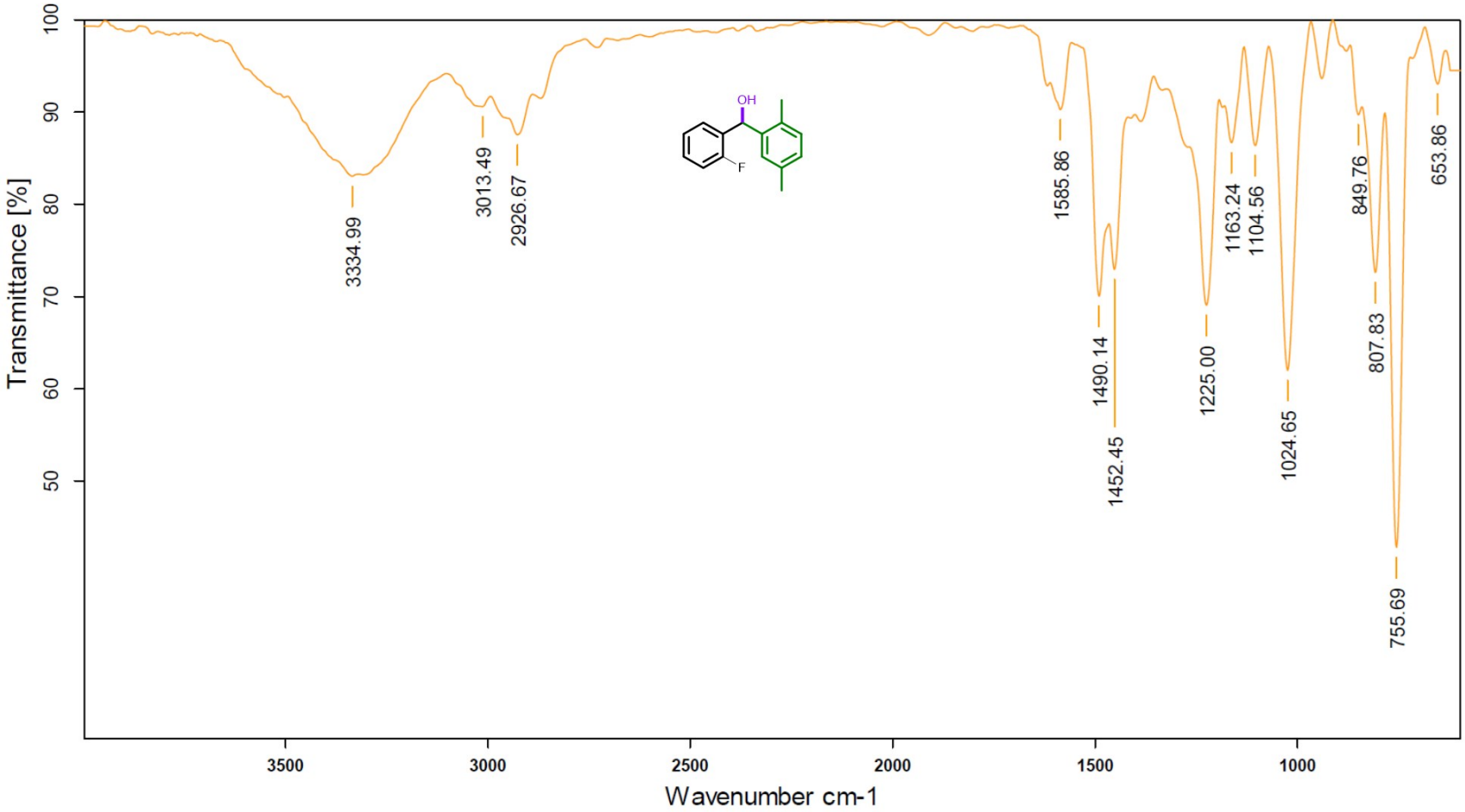


Figure S193. IR spectra of (2, 5-dimethylphenyl) (2-fluorophenyl) methanol **(3p)**.

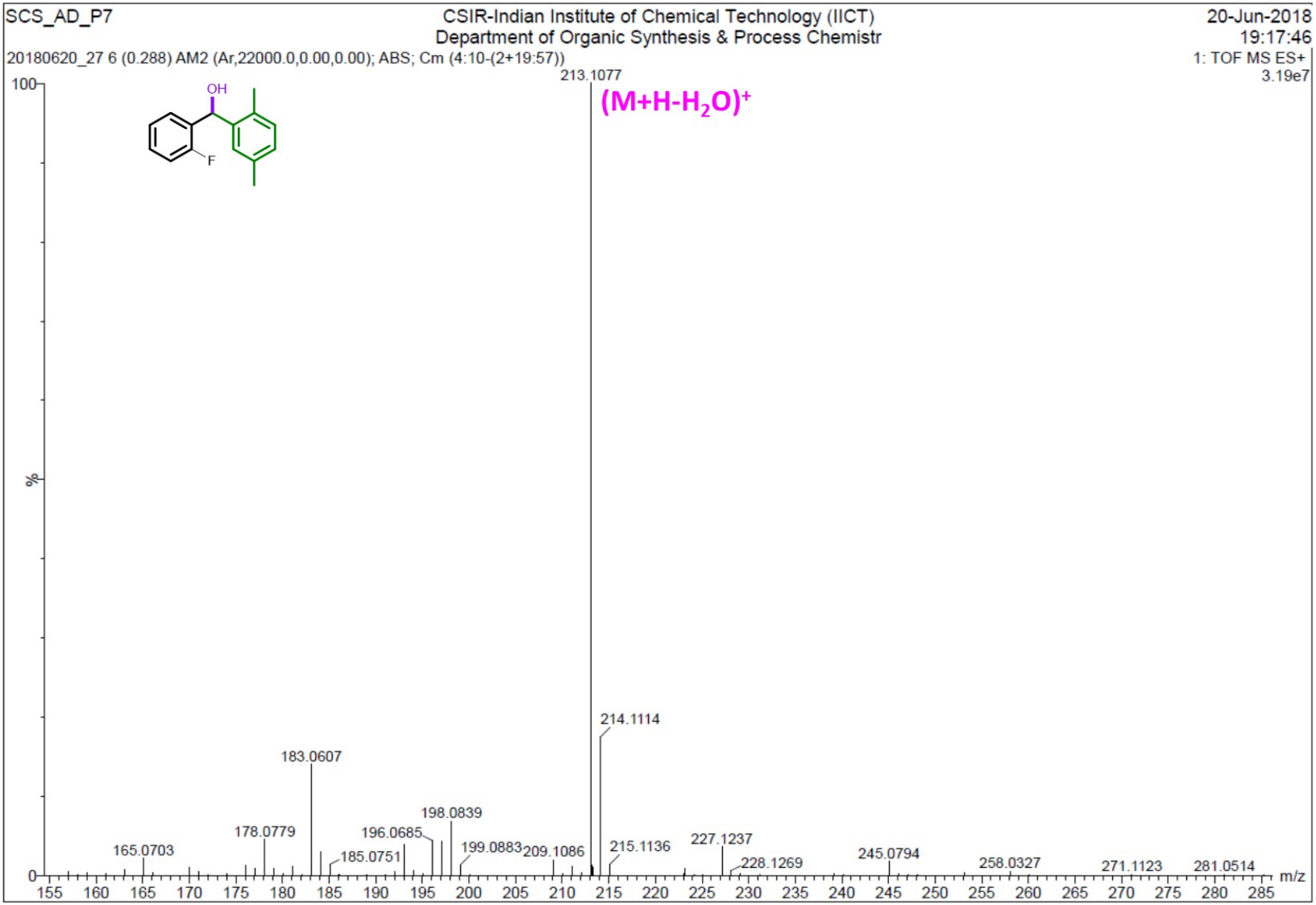


Figure S194. HRMS spectra of (2, 5-dimethylphenyl) (2-fluorophenyl) methanol **(3p)**.

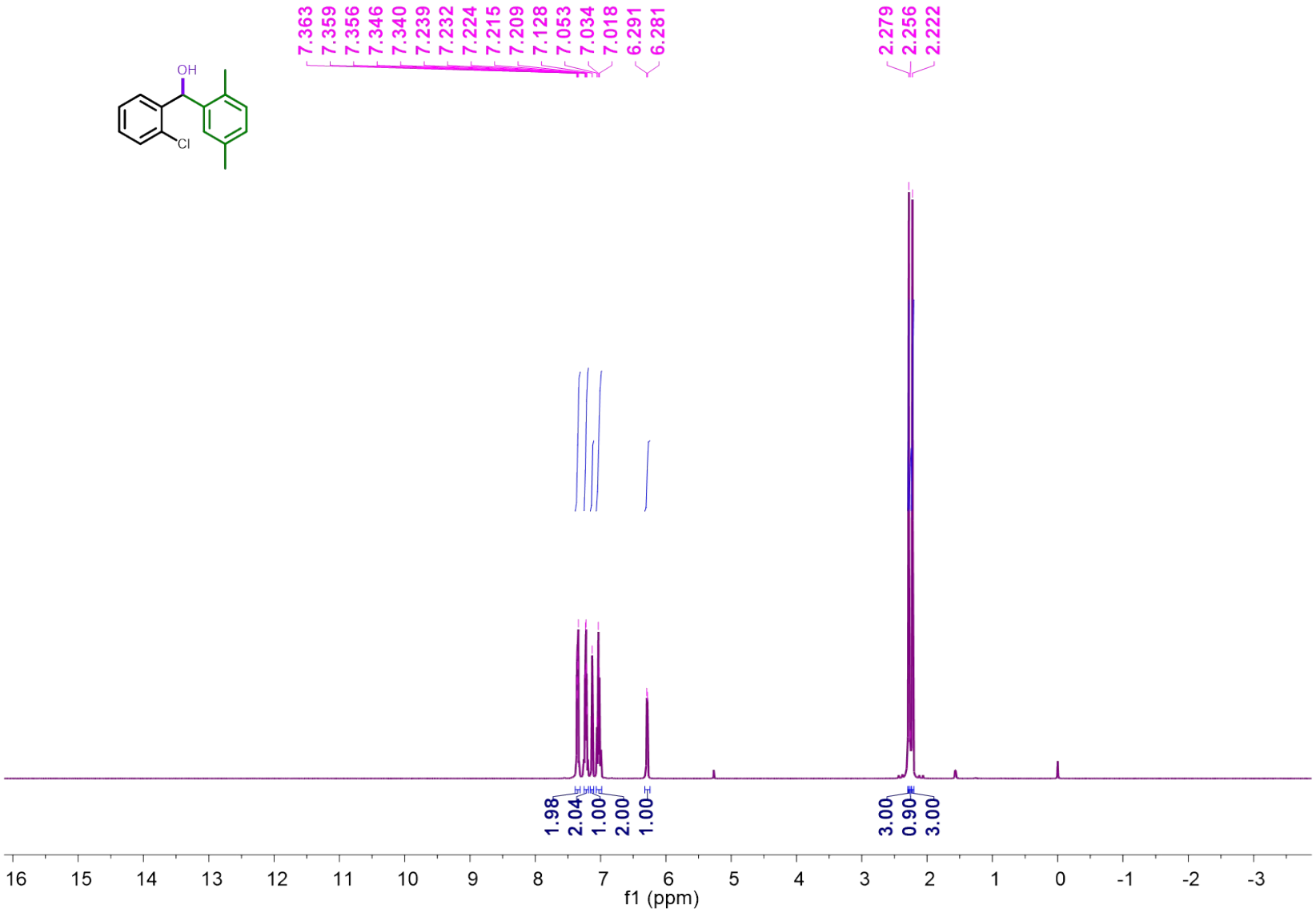


Figure S195. ^{1}H NMR spectra of (2, 5-dimethylphenyl) (2-chlorophenyl) methanol **(3q)** in $CDCl_3$.

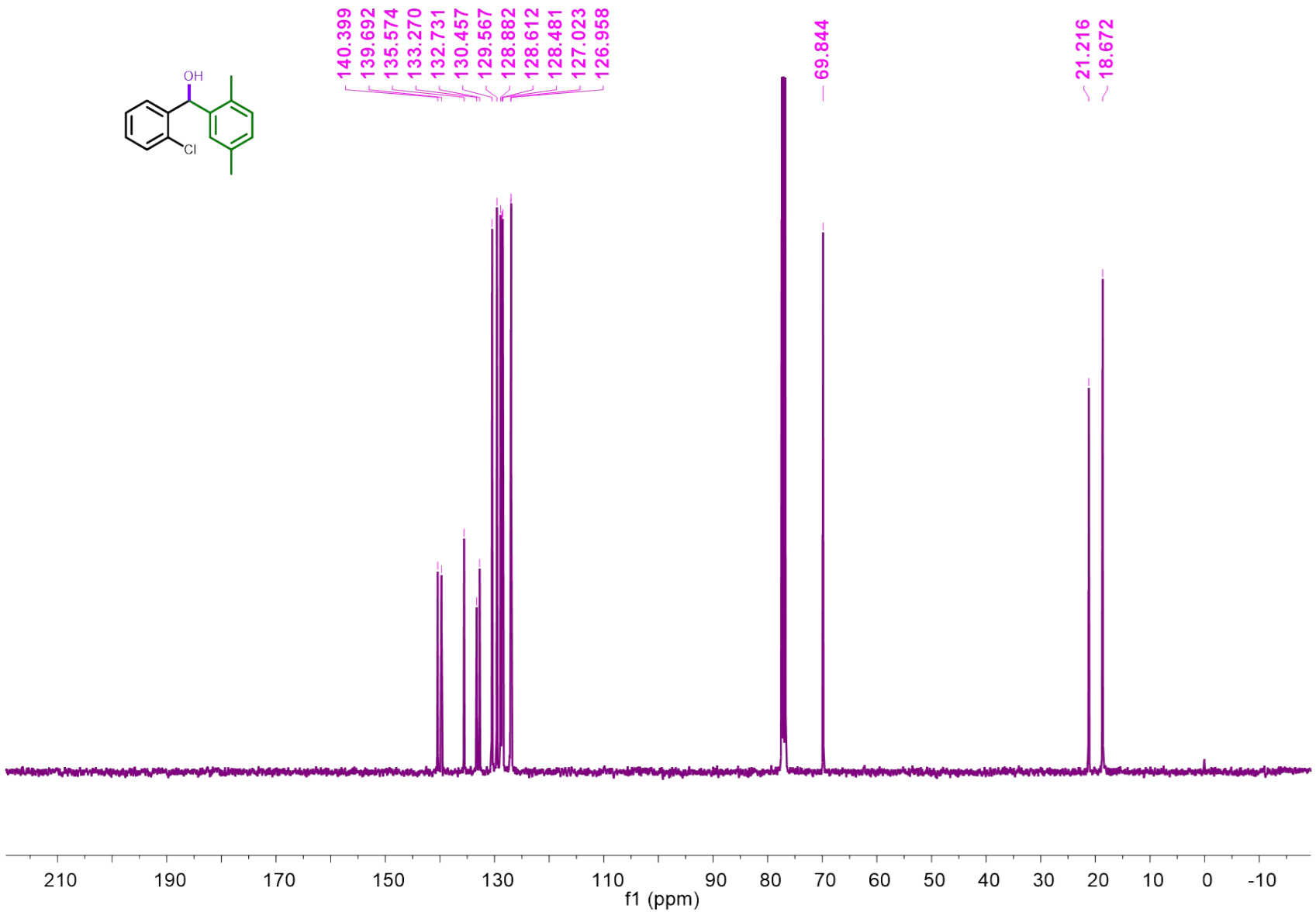


Figure S196. ^{13}C NMR spectra of (2, 5-dimethylphenyl) (2-chlorophenyl) methanol **(3q)** in $CDCl_3$.

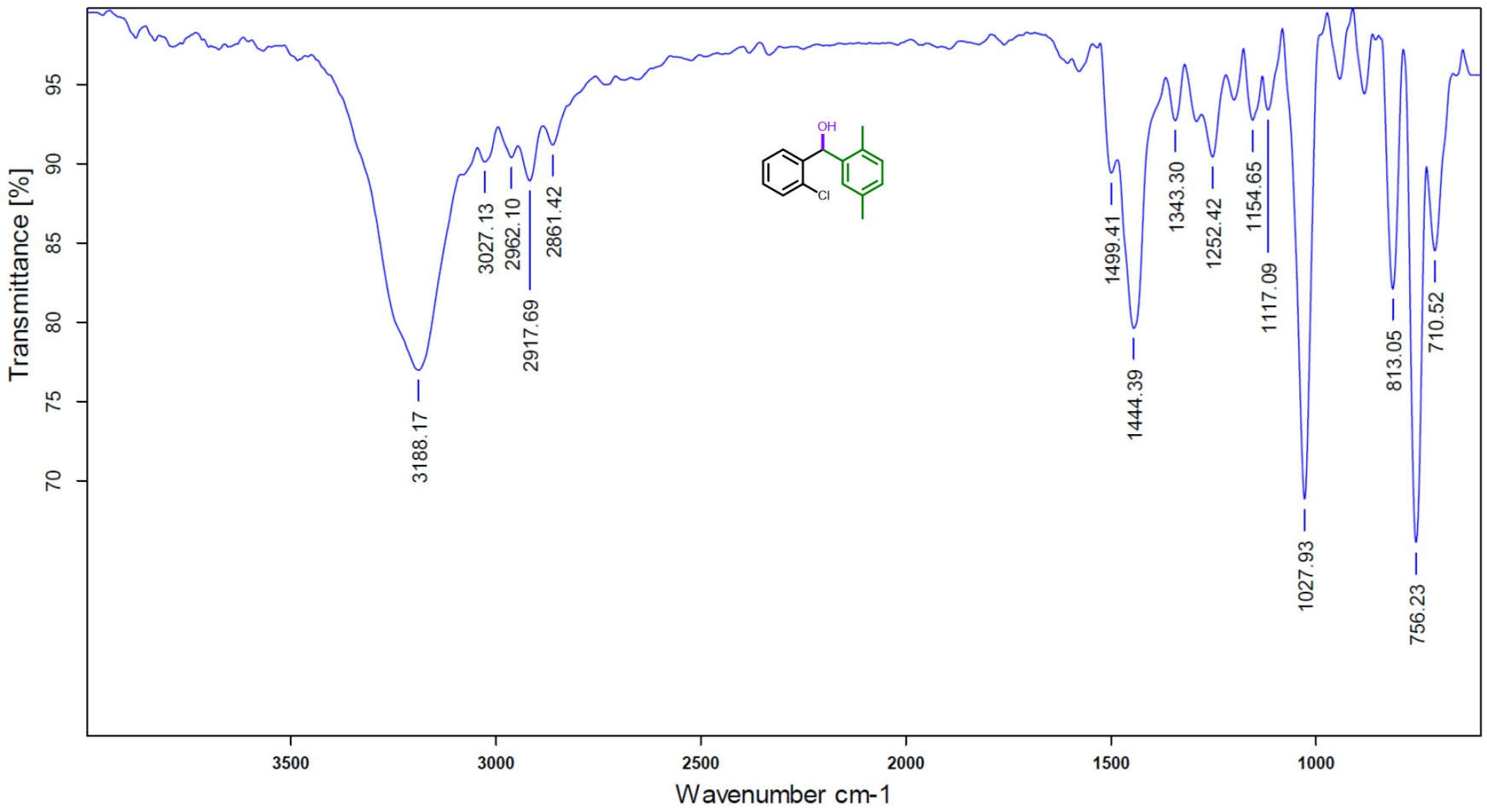


Figure S197. IR spectra of (2, 5-dimethylphenyl) (2-chlorophenyl) methanol **(3q**).

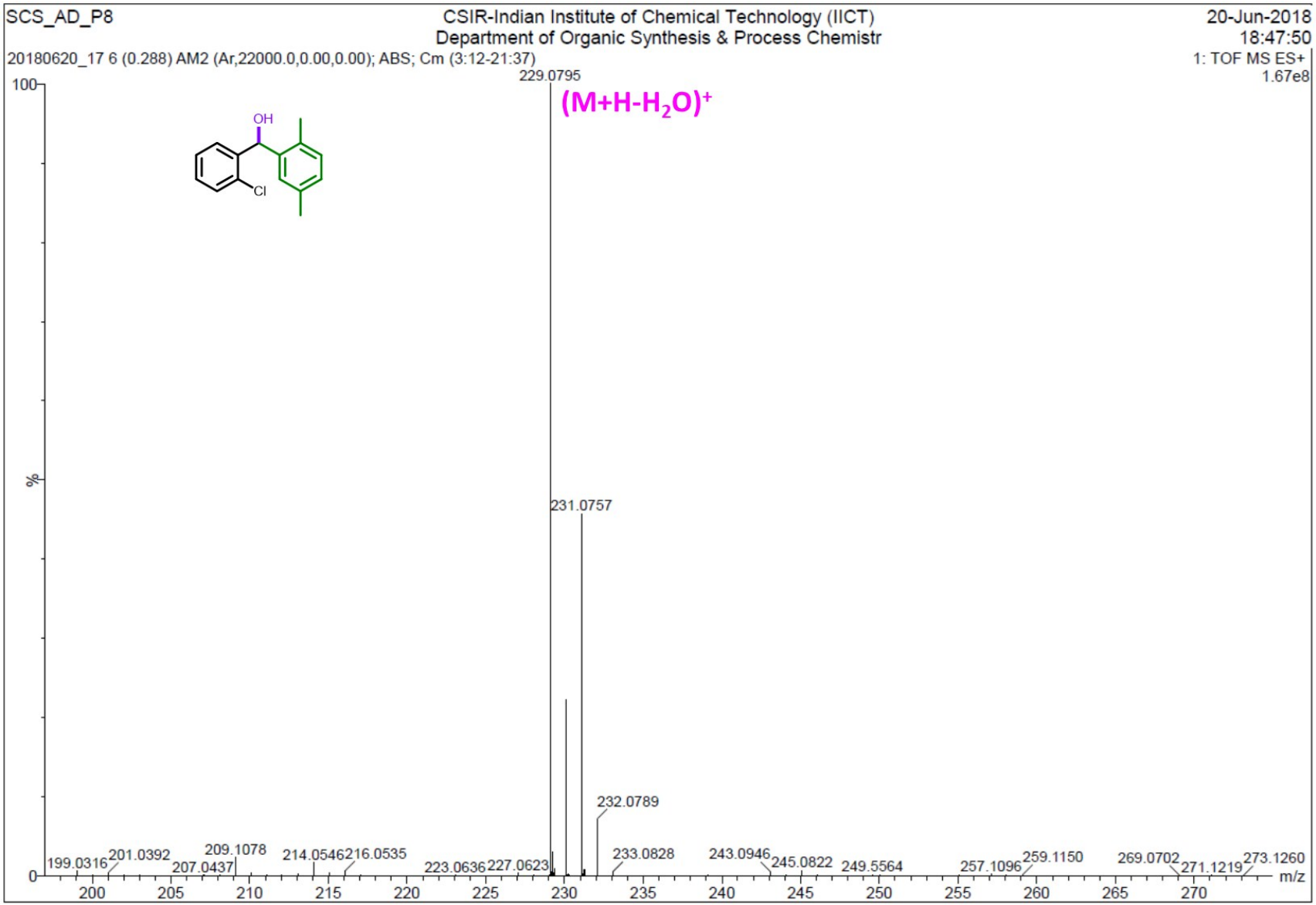


Figure S198. HRMS spectra of (2, 5-dimethylphenyl) (2-chlorophenyl) methanol **(3q)**.

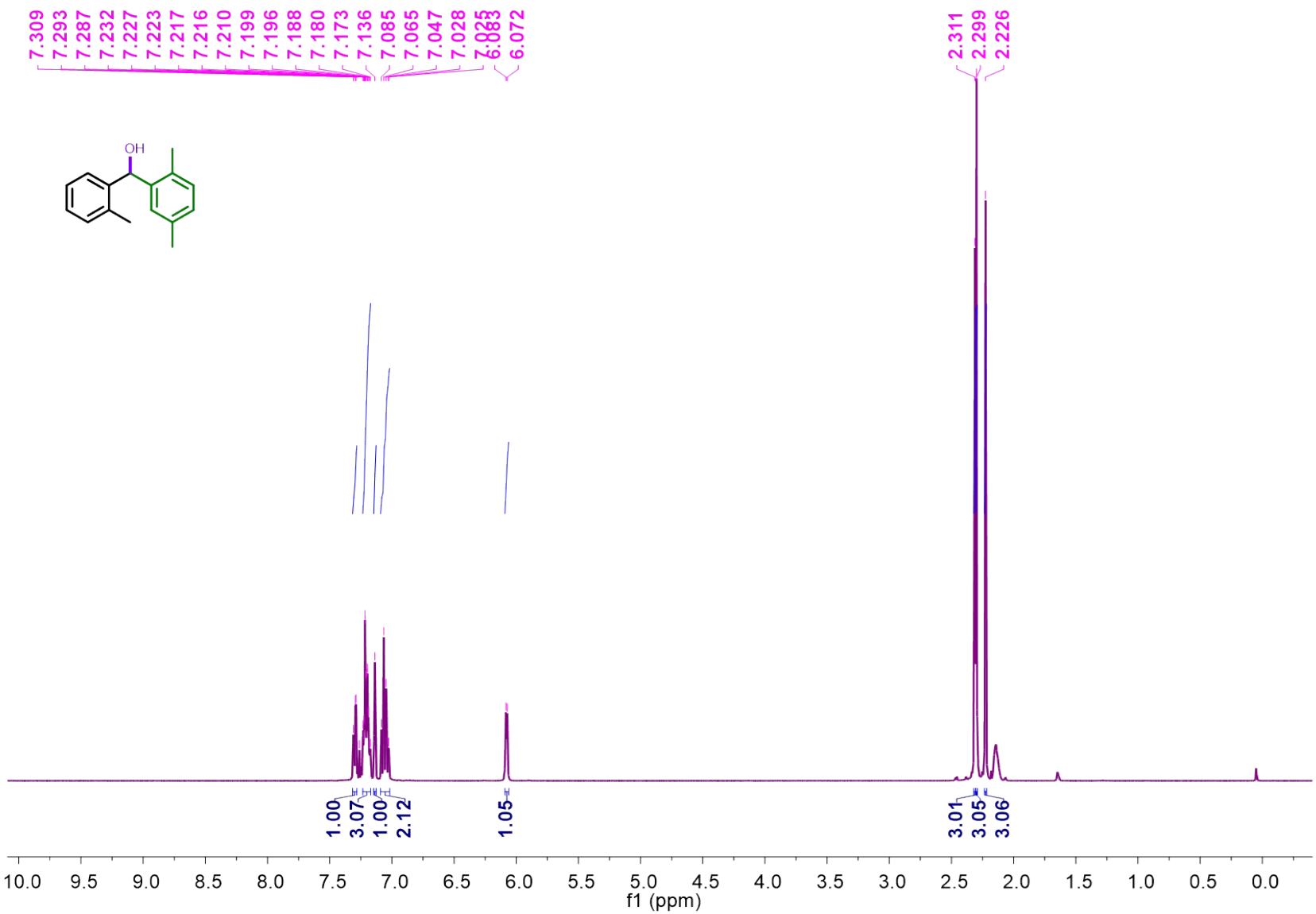


Figure S199. ^{1}H NMR spectra of (2, 5-dimethylphenyl) (2-methylphenyl) methanol **(3r)** in $CDCl_3$.

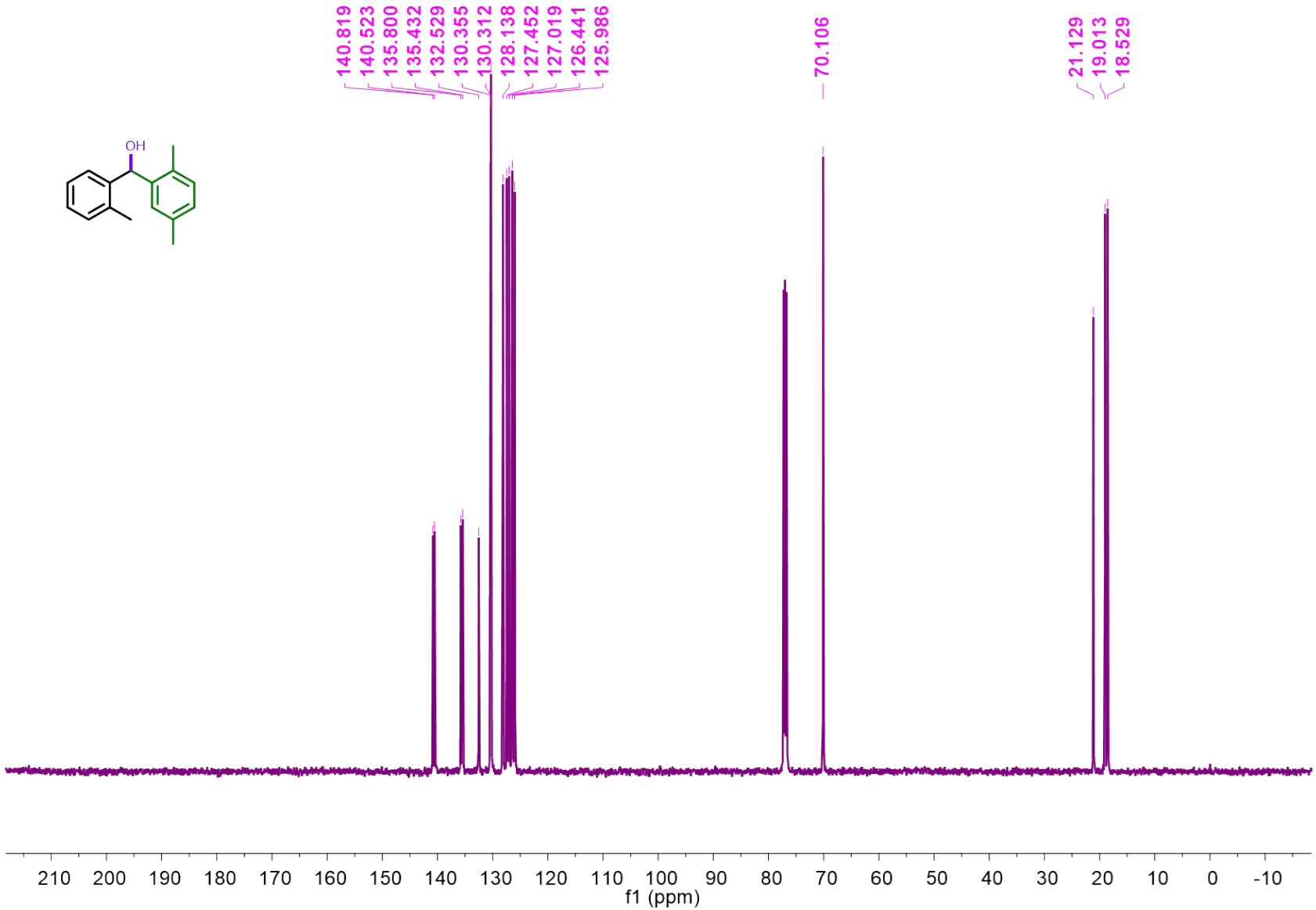


Figure S200. ^{13}C NMR spectra of (2, 5-dimethylphenyl) (2-methylphenyl) methanol **(3r)** in $CDCl_3$.

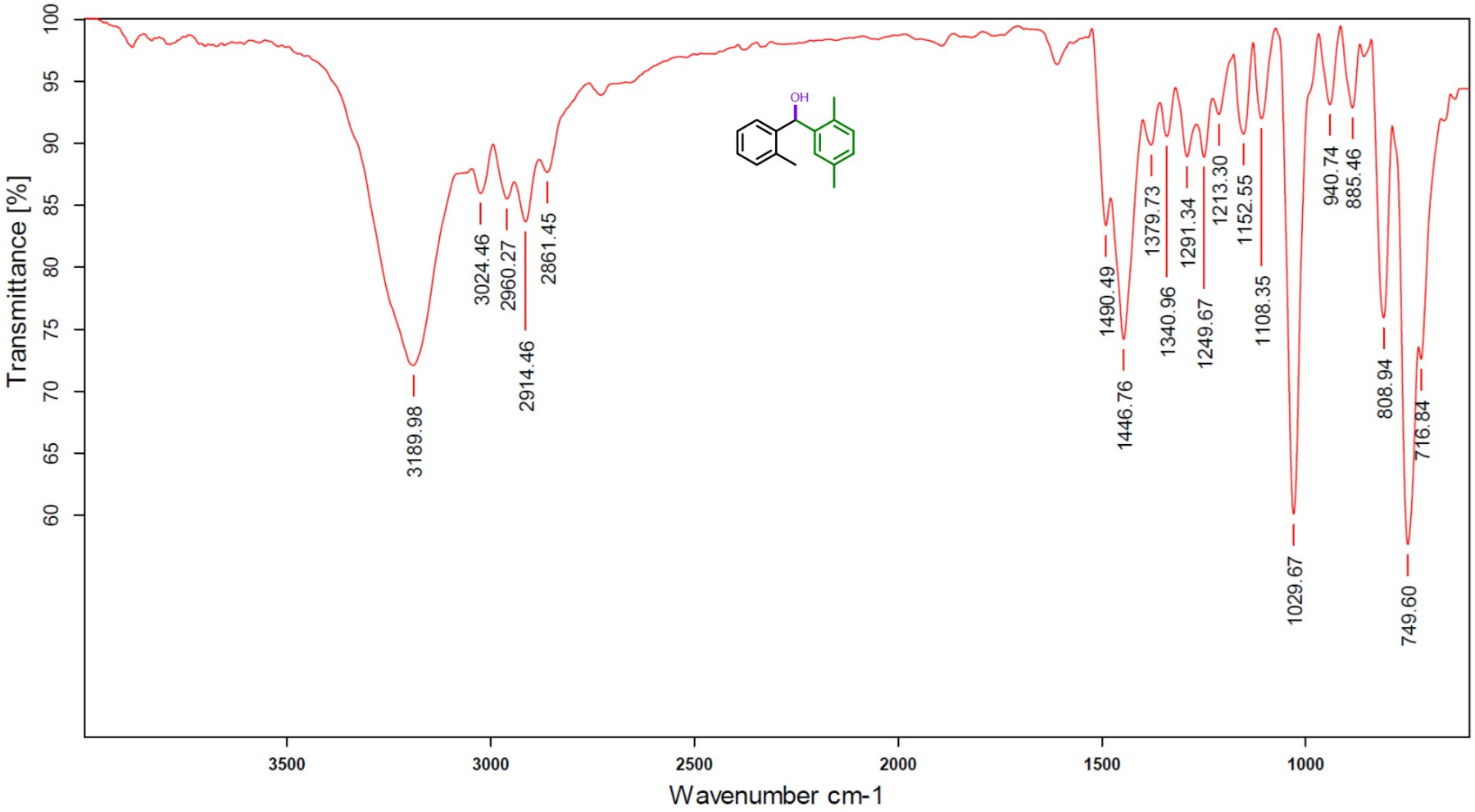


Figure S201. IR spectra of (2, 5-dimethylphenyl) (2-methylphenyl) methanol **(3r)**.

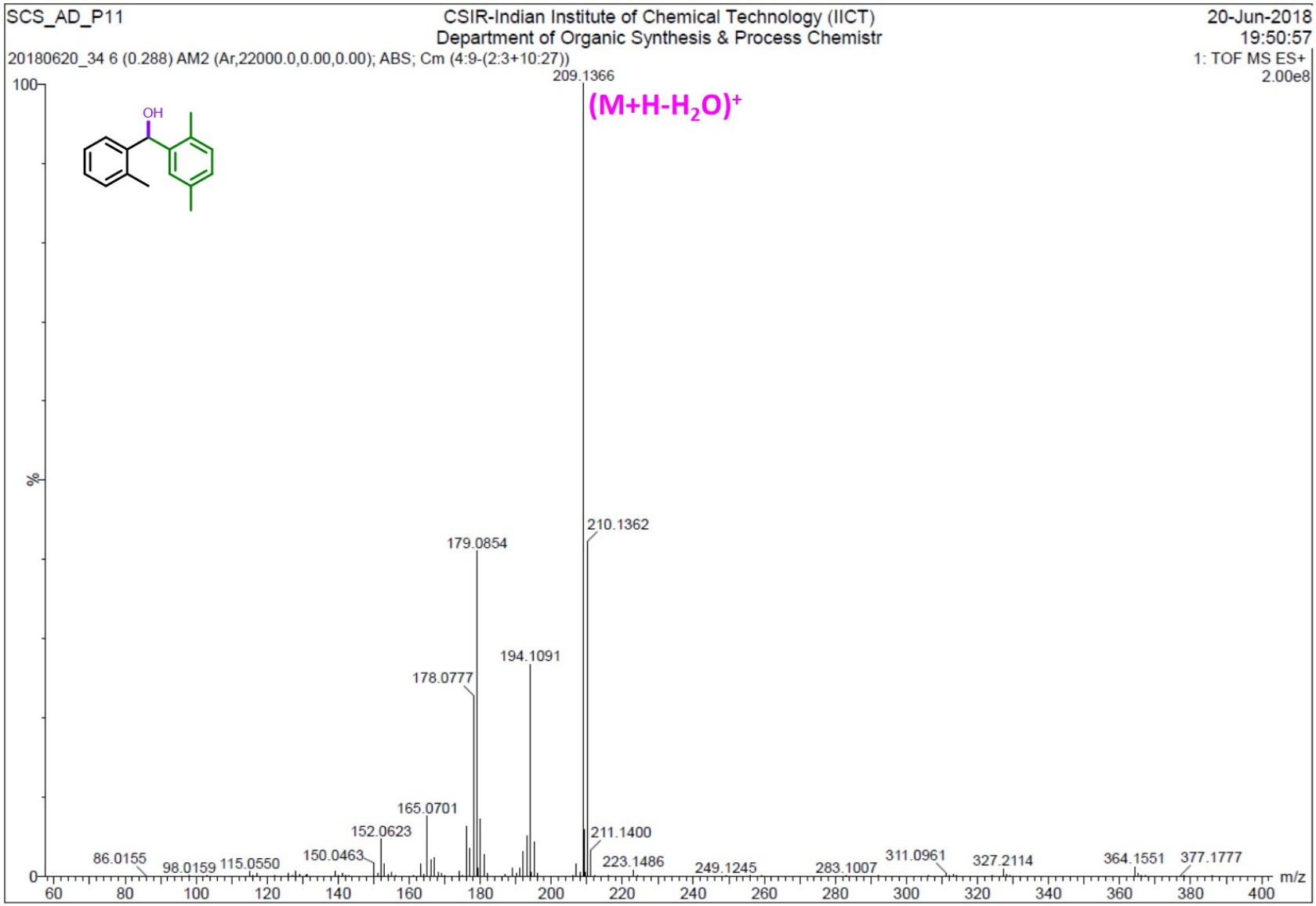


Figure S202. HRMS spectra of (2, 5-dimethylphenyl) (2-methylphenyl) methanol **(3r)**.

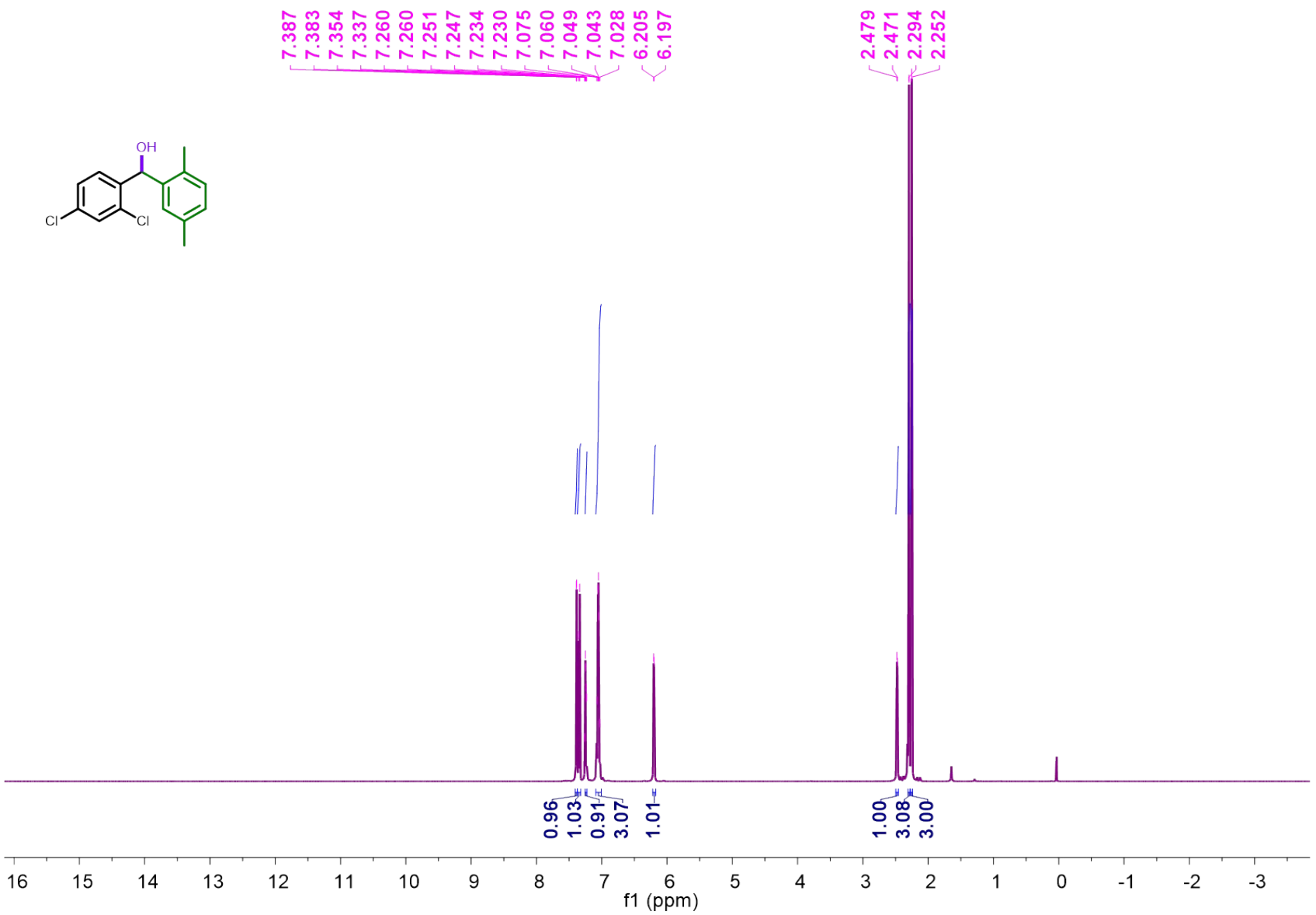


Figure S203. ^{1}H NMR spectra of (2,4-dichlorophenyl) (2,5-dimethylphenyl) methanol **(3s)** in $CDCl_3$.

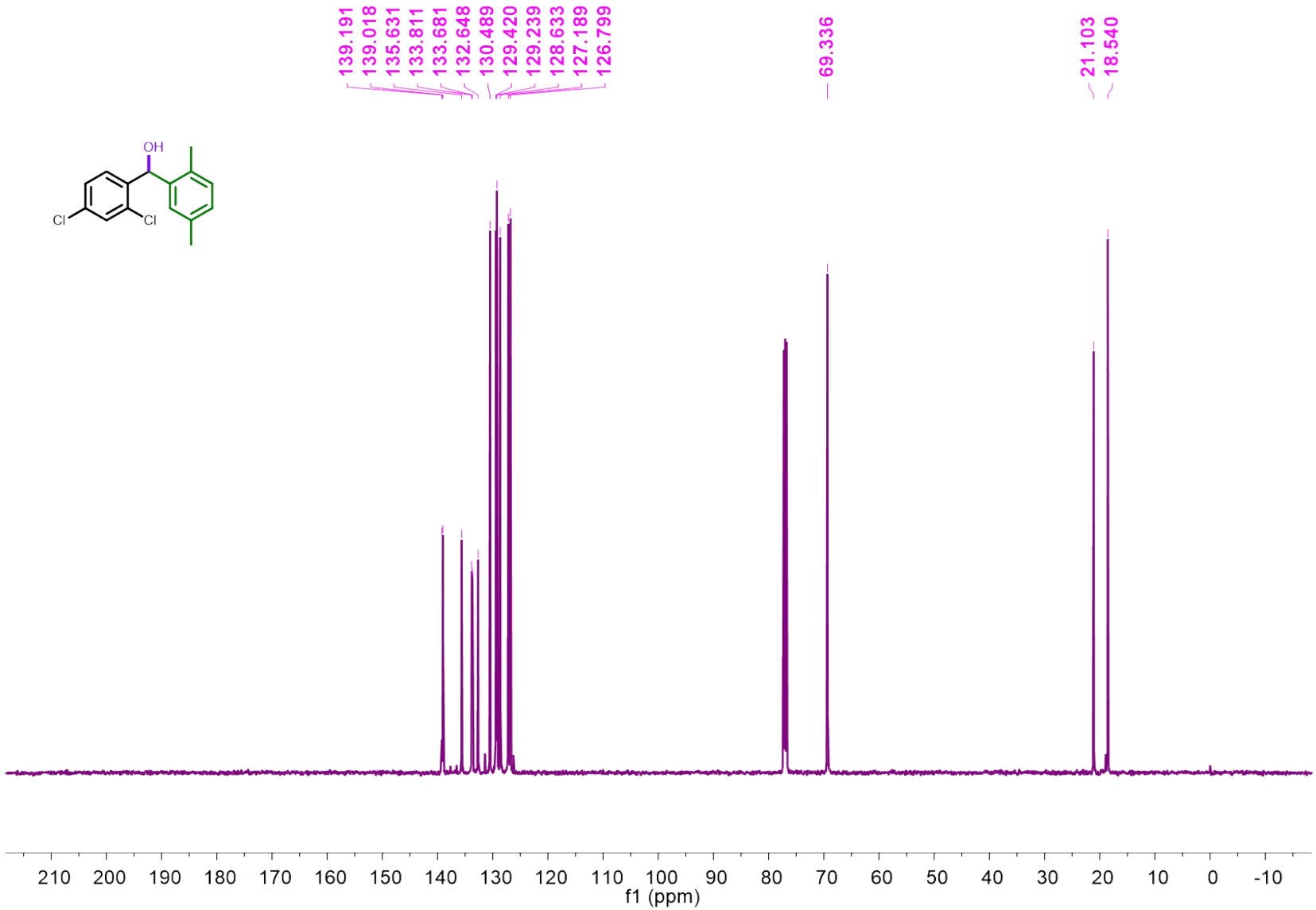


Figure S204. ^{13}C NMR spectra of (2,4-dichlorophenyl) (2,5-dimethylphenyl) methanol **(3s)** in $CDCl_3$.

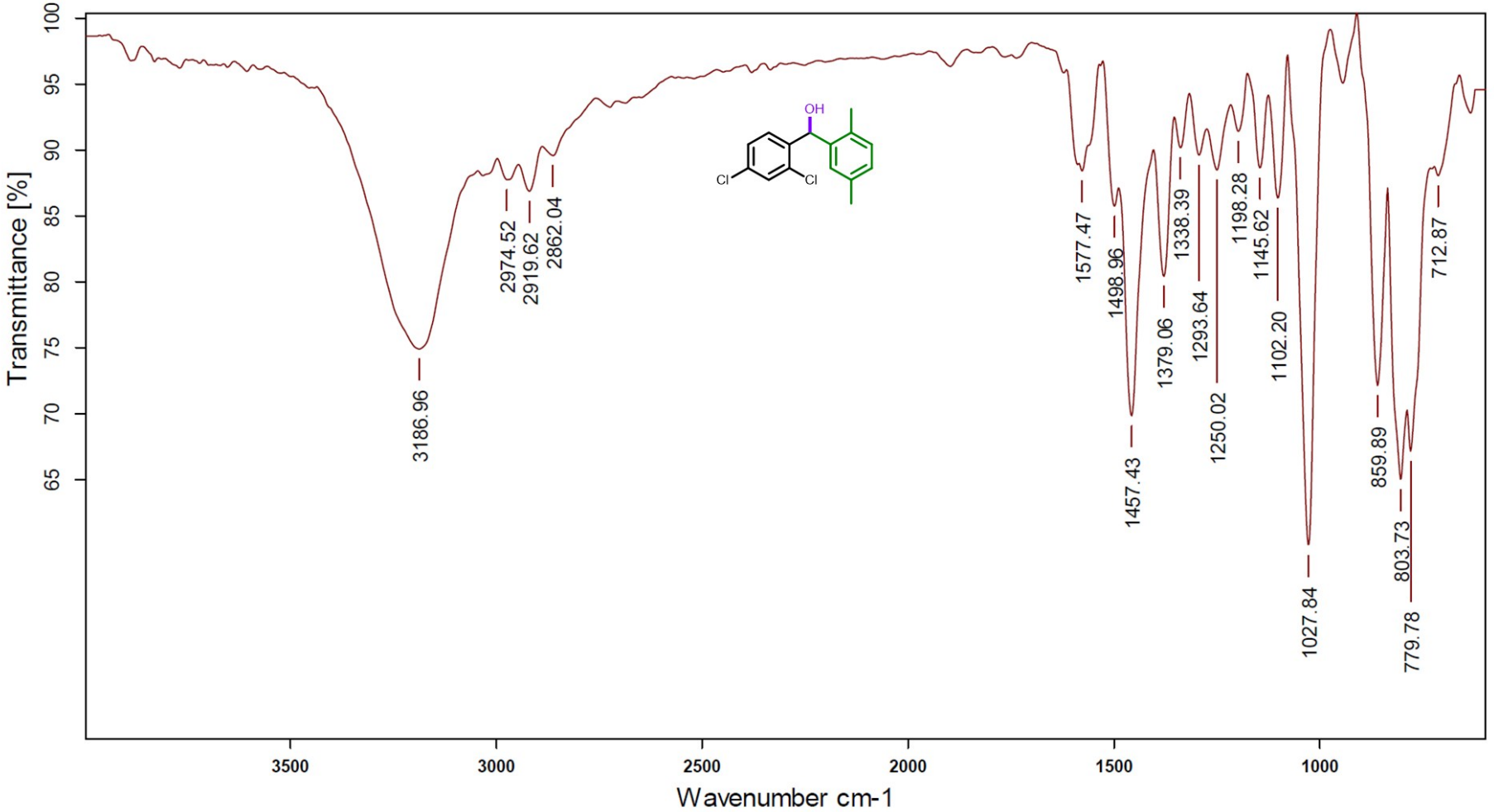


Figure S205. IR spectra of (2,4-dichlorophenyl) (2,5-dimethylphenyl) methanol **(3s**).

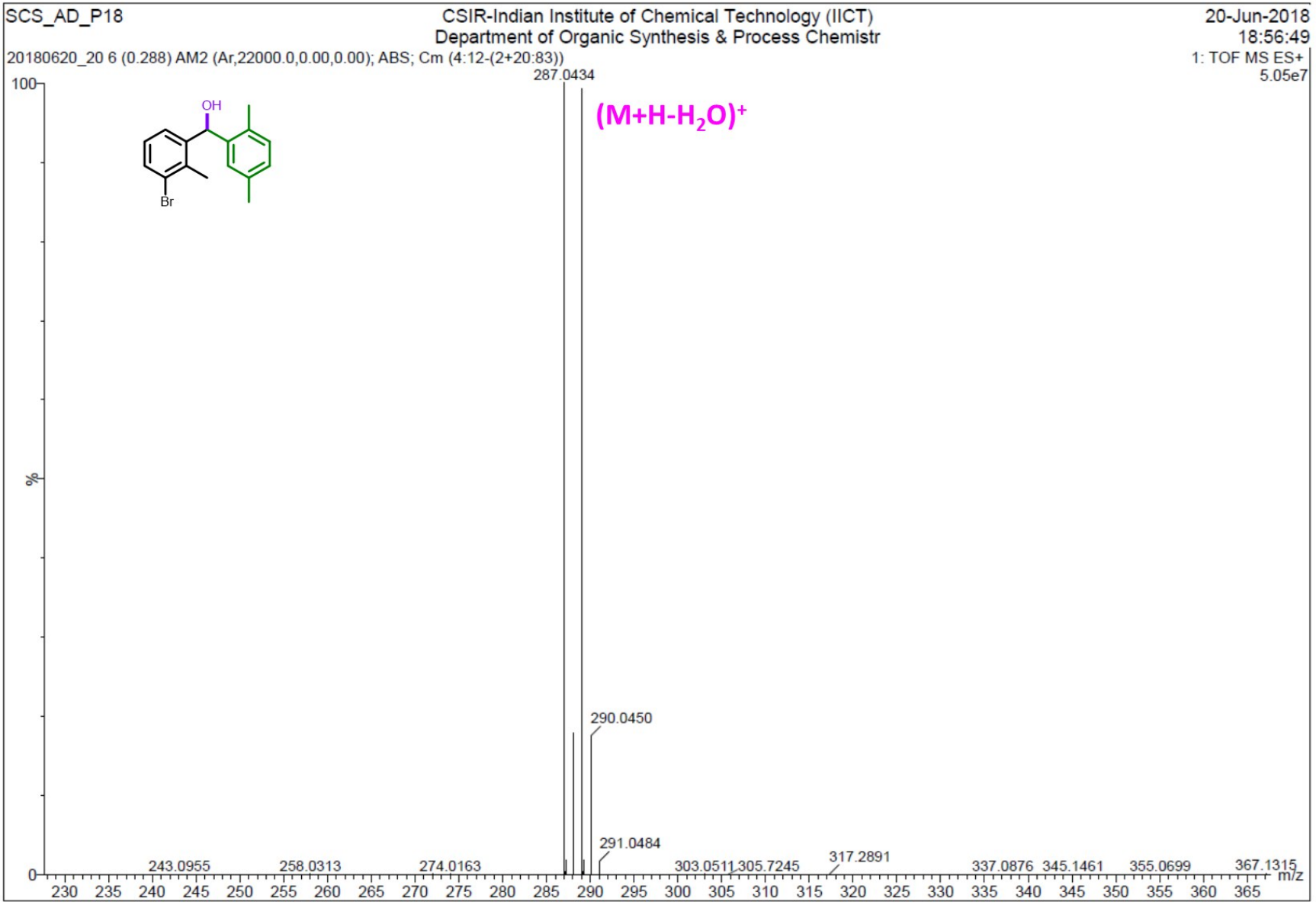


Figure S206. HRMS spectra of (2,4-dichlorophenyl) (2,5-dimethylphenyl) methanol **(3s**).

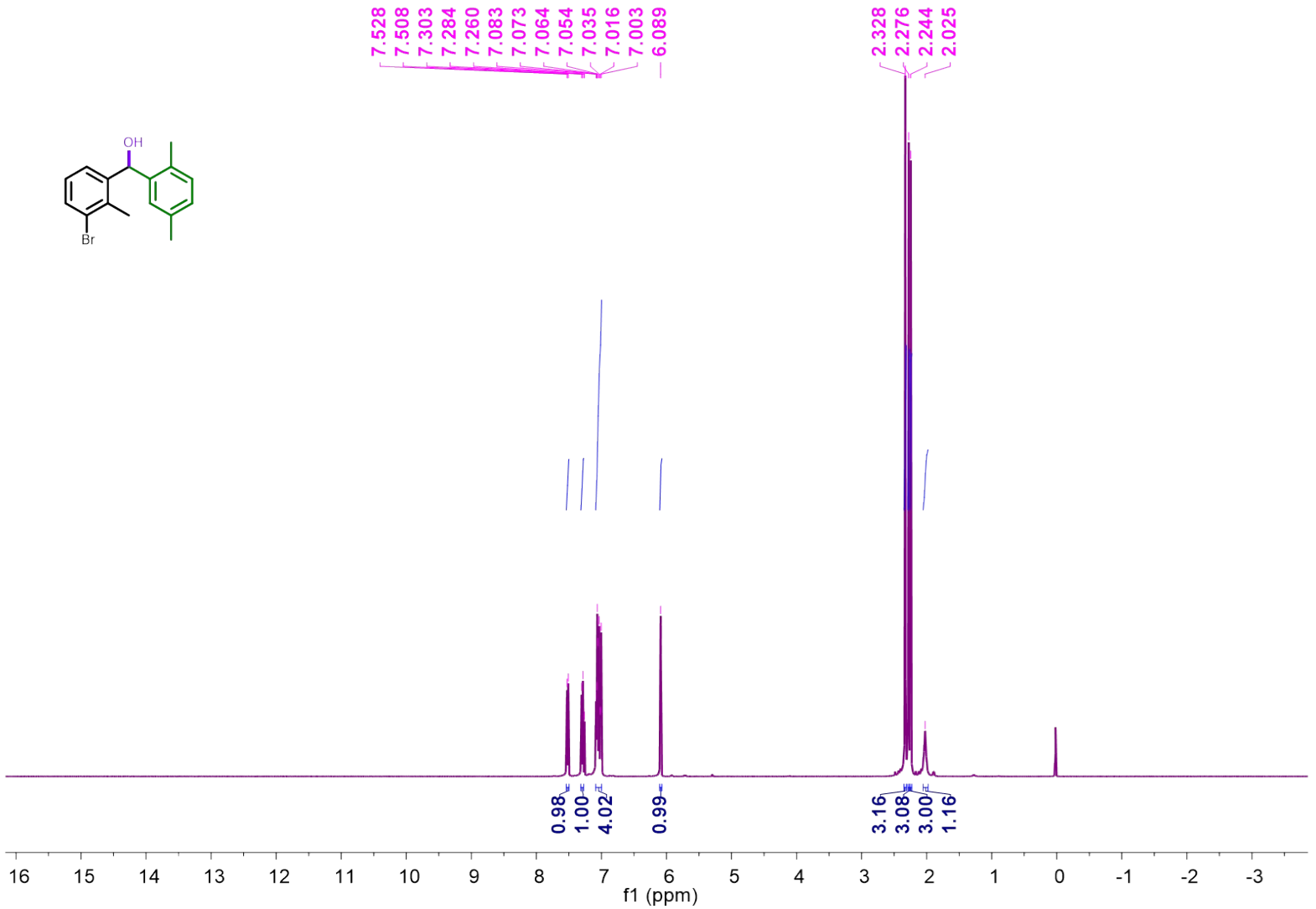


Figure S207. ^{1}H NMR spectra of (3-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanol **(3t)** in $CDCl_3$

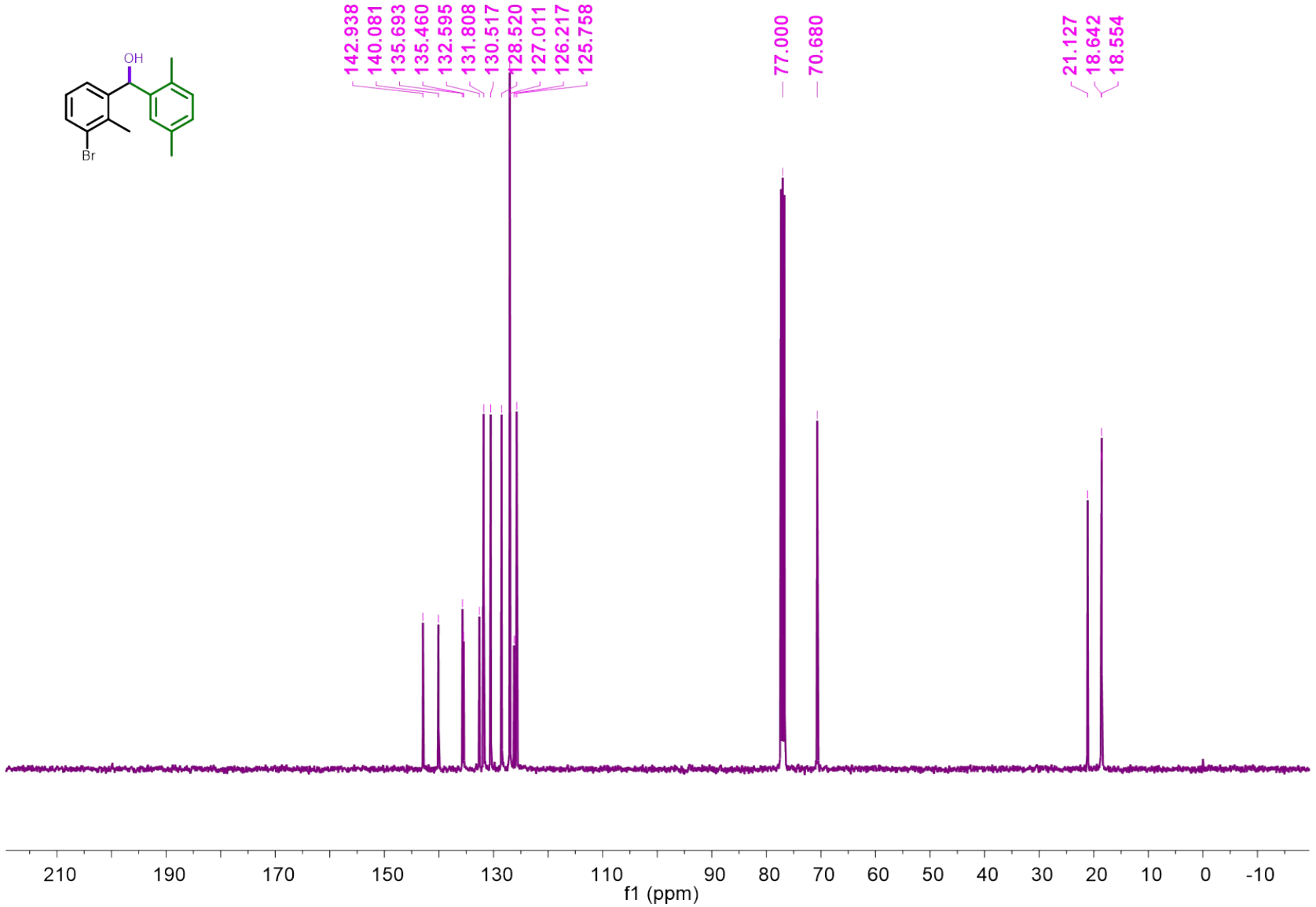


Figure S208. ^{13}C NMR spectra of (3-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanol **(3t)** in $CDCl_3$.

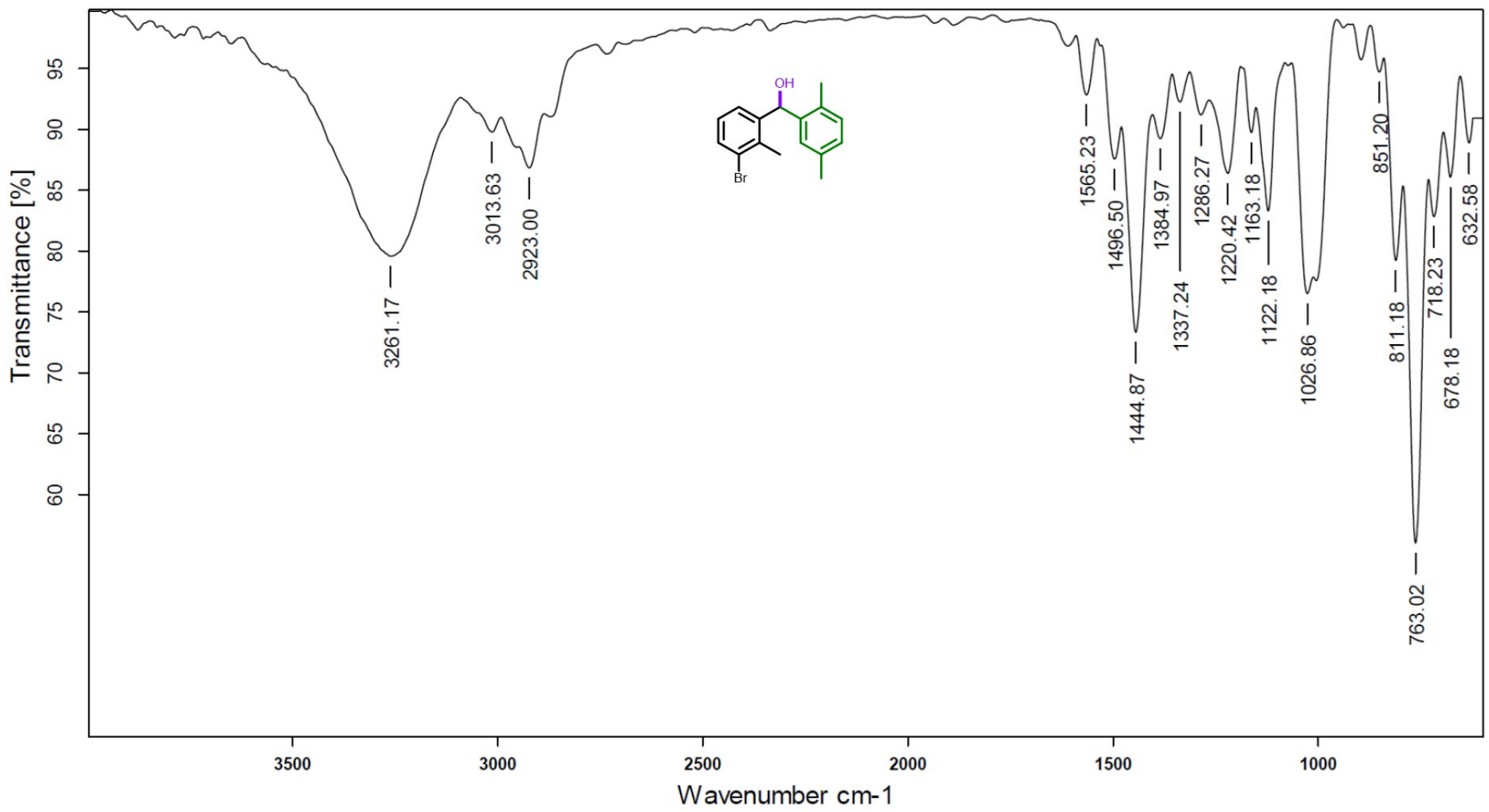


Figure S209. IR spectra of (3-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanol **(3t)**.

Figure S210. HRMS spectra of (3-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanol **(3t**).

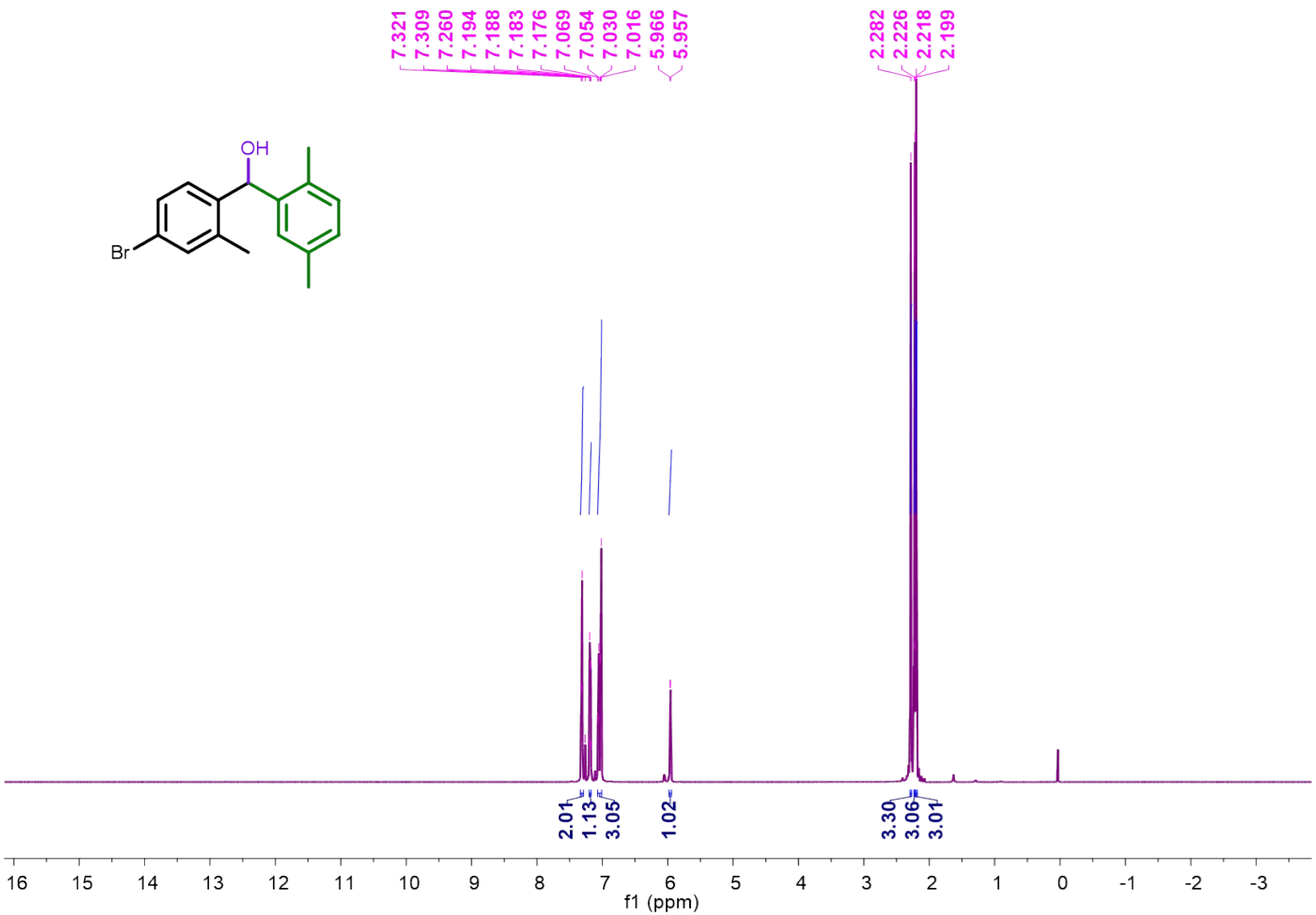


Figure S211. ^{1}H NMR spectra of (4-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanol **(3u)** in $CDCl_3$

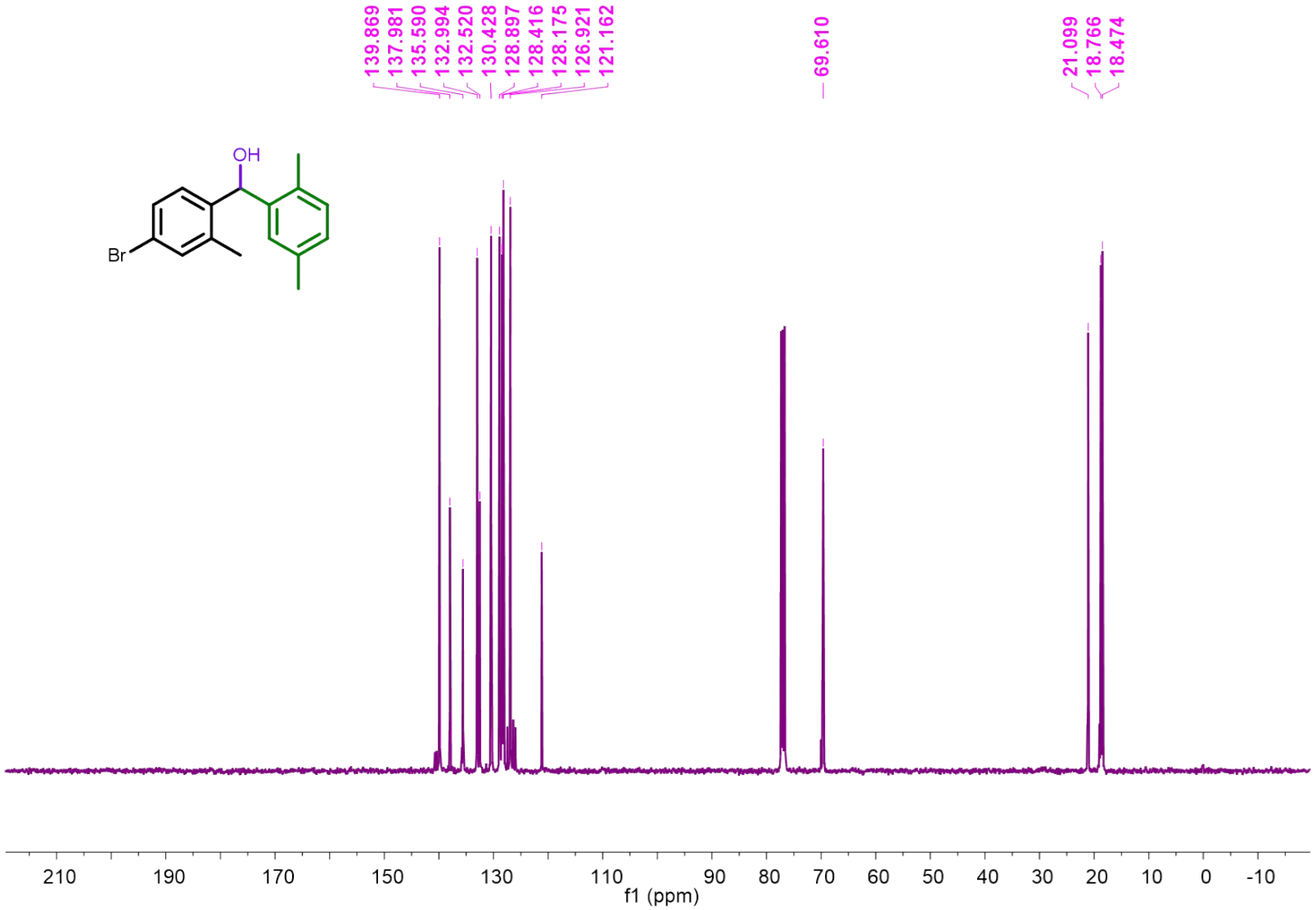


Figure S212. ^{13}C NMR spectra of (4-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanol **(3u)** in $CDCl_3$.

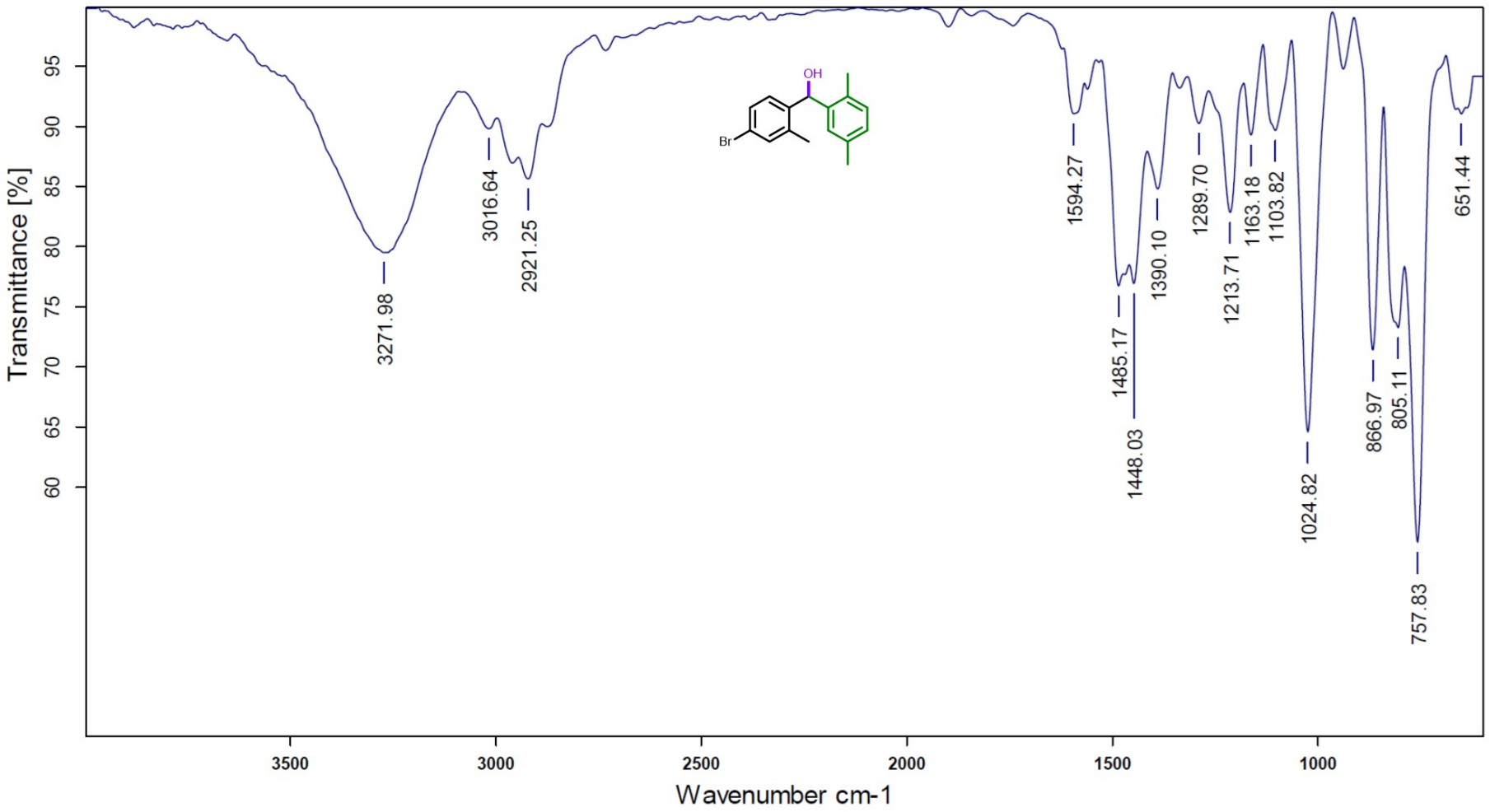


Figure S213. IR spectra of (4-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanol **(3u**).

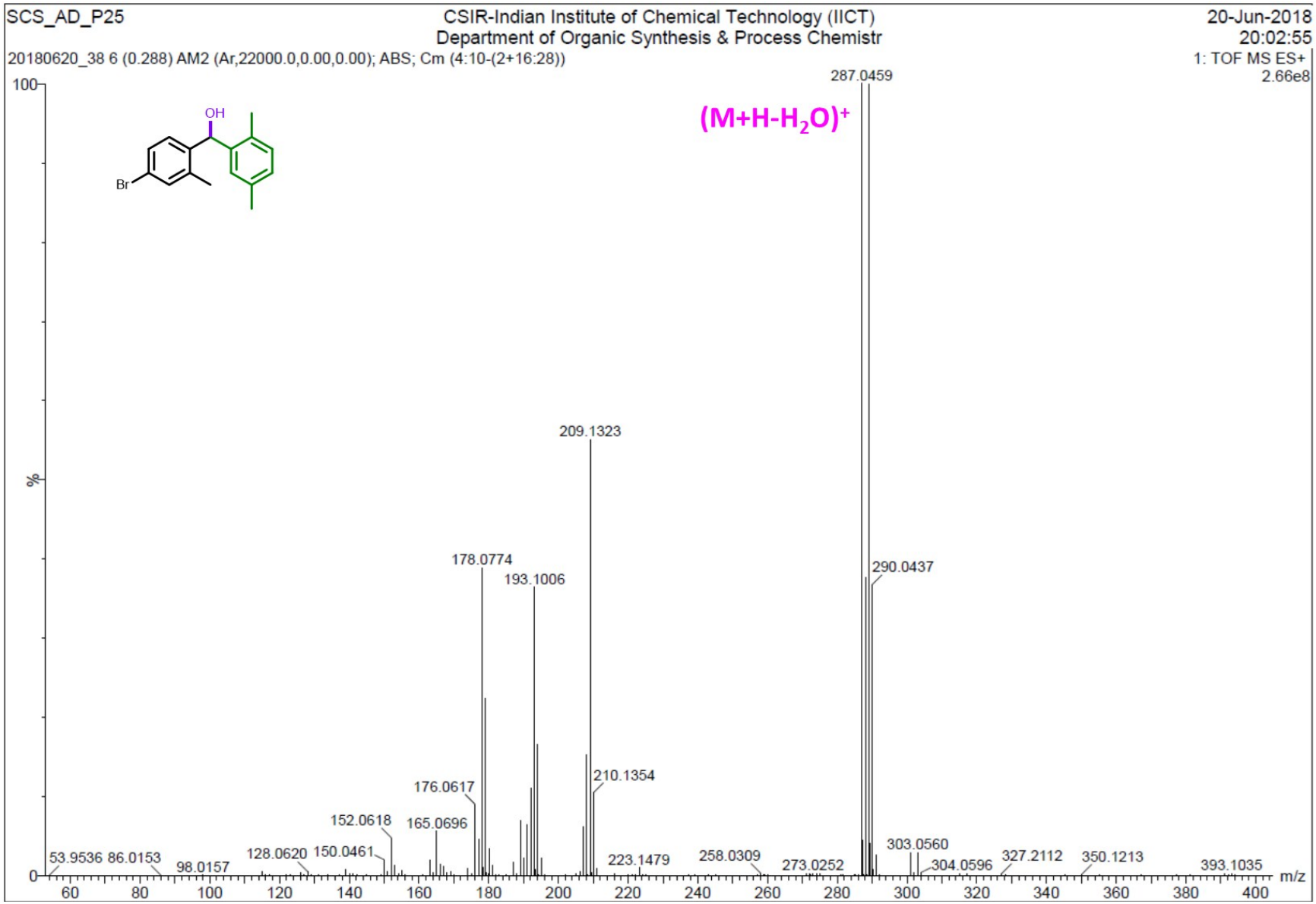
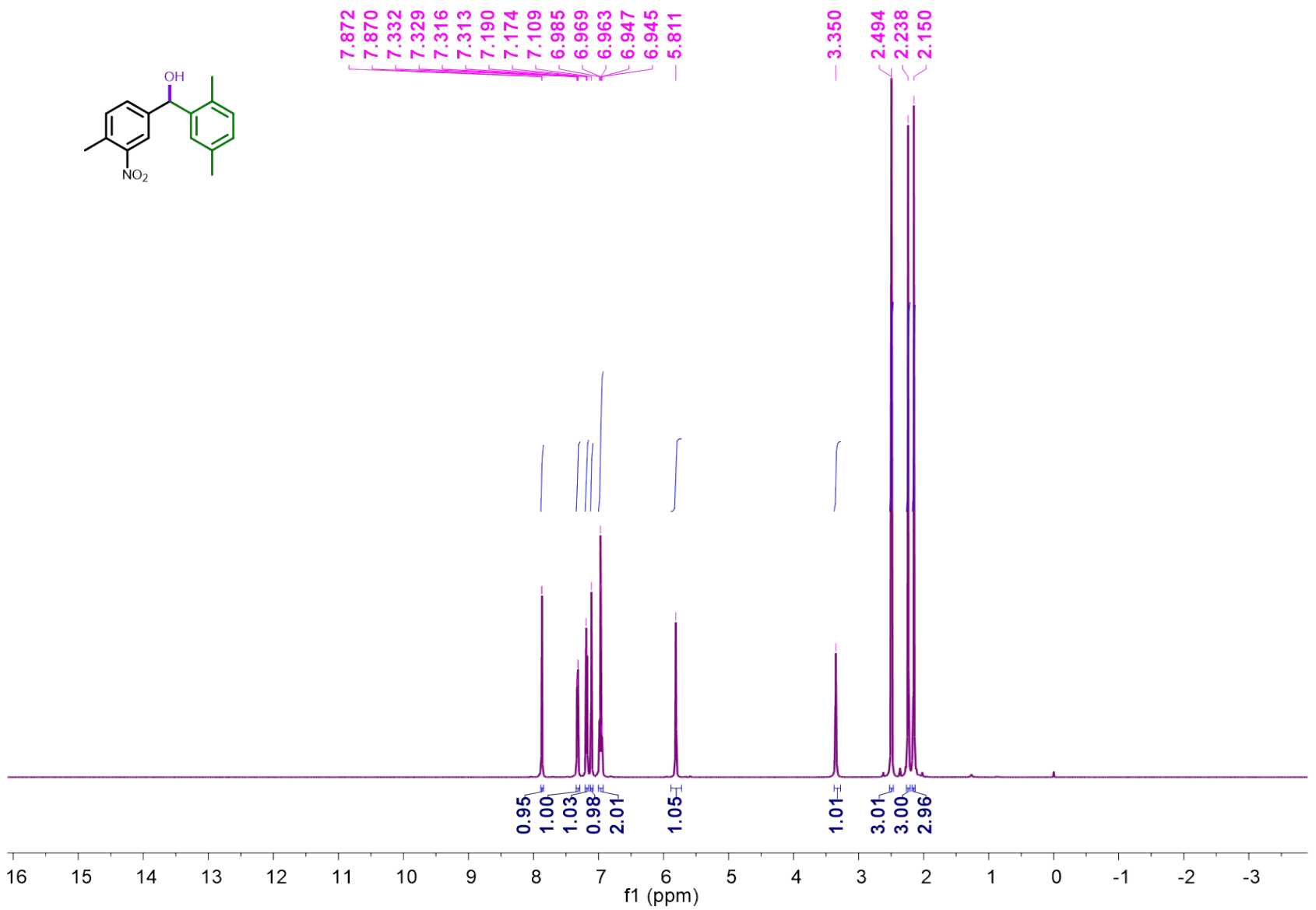


Figure S214. HRMS spectra of (4-bromo-2-methylphenyl) (2,5-dimethylphenyl) methanol **(3u)**.



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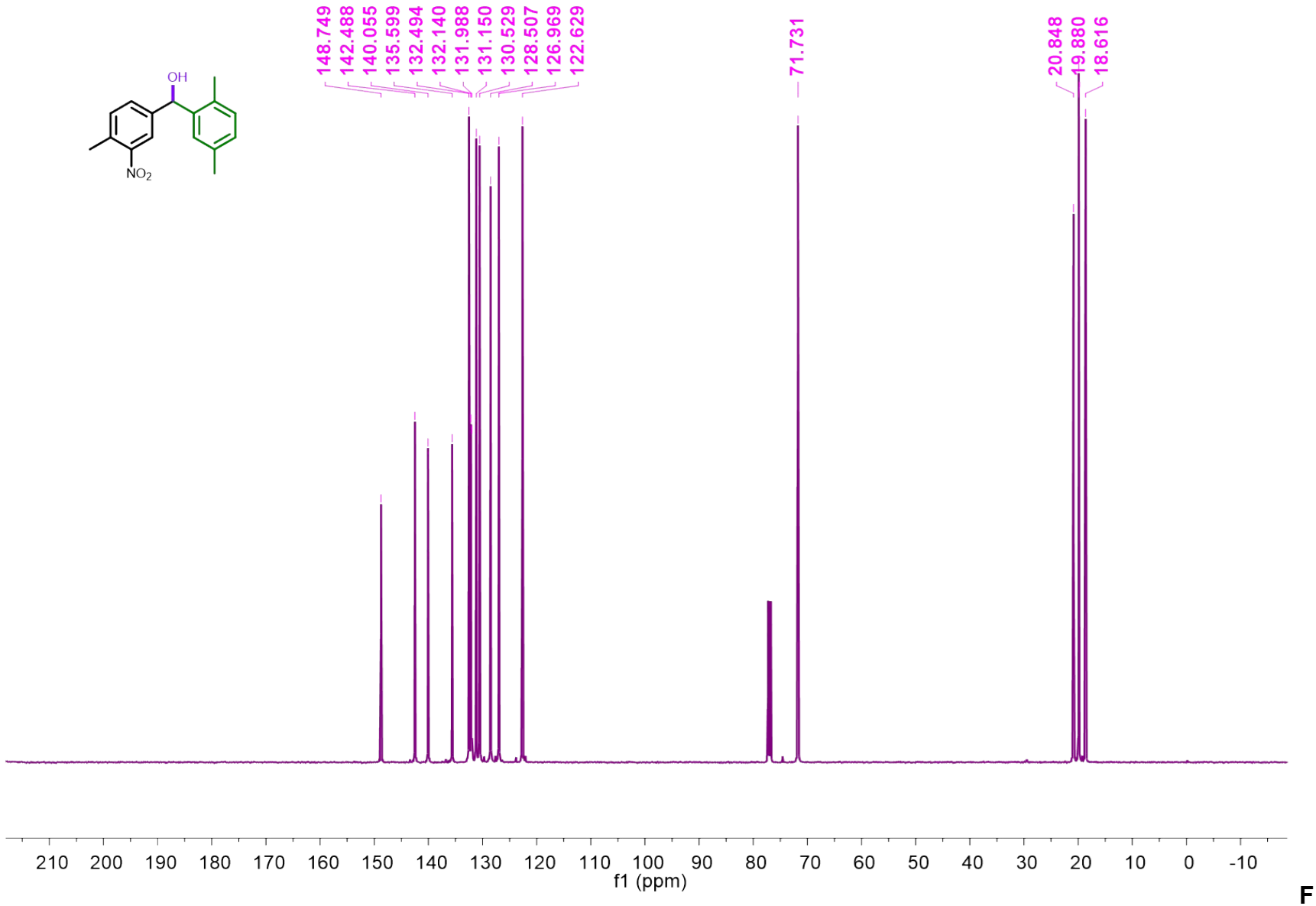
igure S215. ^{1}H NMR spectra of (2,5-dimethylphenyl) (4-methyl-3-nitrophenyl) methanol **(3v)** in $CDCl_3$.


148.749
142.488
140.055
135.599
132.494
132.140
131.988
131.150
130.529
128.507
126.969
122.629
71.731
20.848
19.880
18.616
OH
NO2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

F

igure S216. ^{13}C NMR spectra of (2,5-dimethylphenyl) (4-methyl-3-nitrophenyl) methanol **(3v)** in $CDCl_3$.

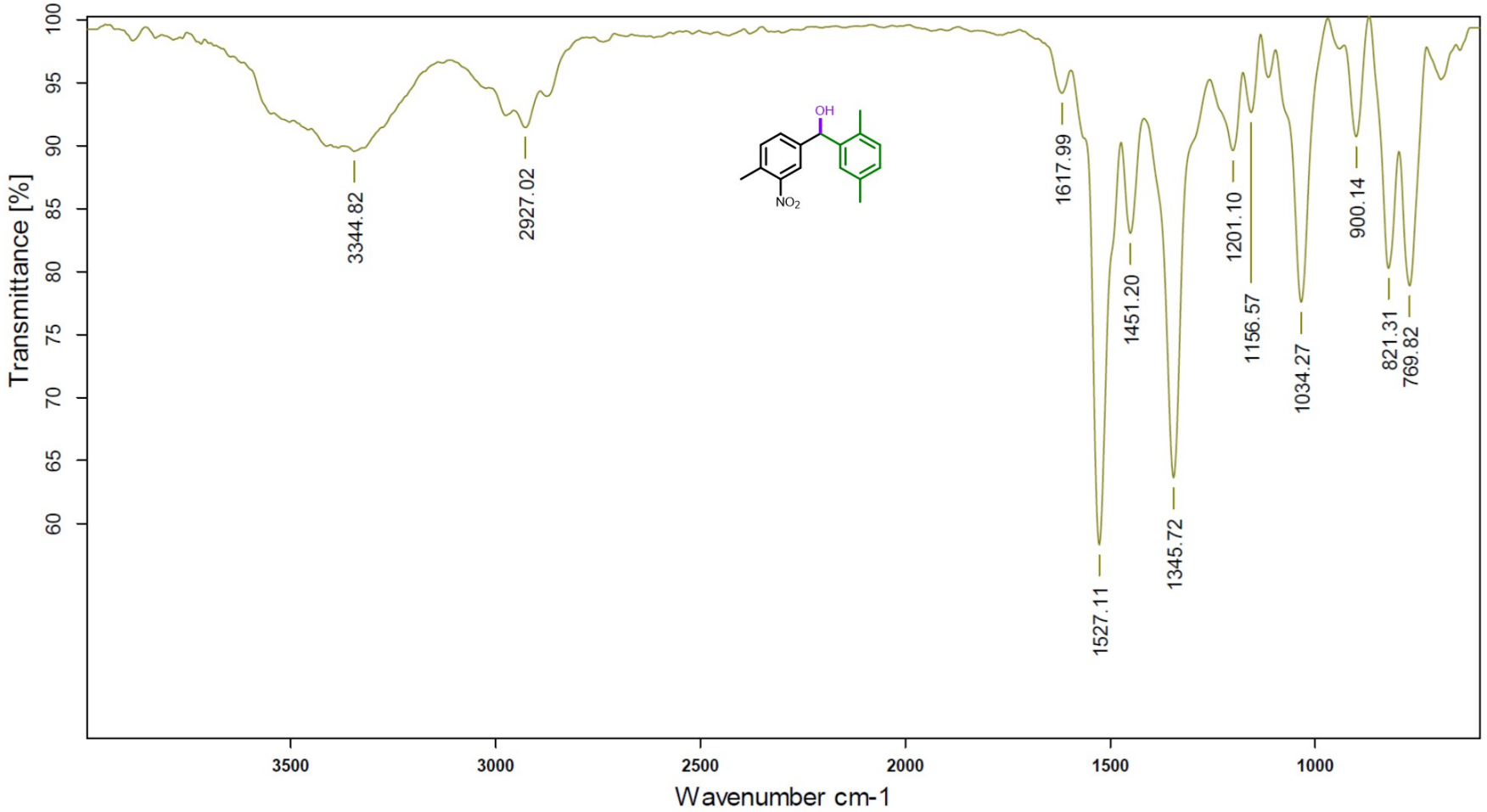


Figure S217. IR spectra of (2,5-dimethylphenyl) (4-methyl-3-nitrophenyl) methanol **(3v)**.

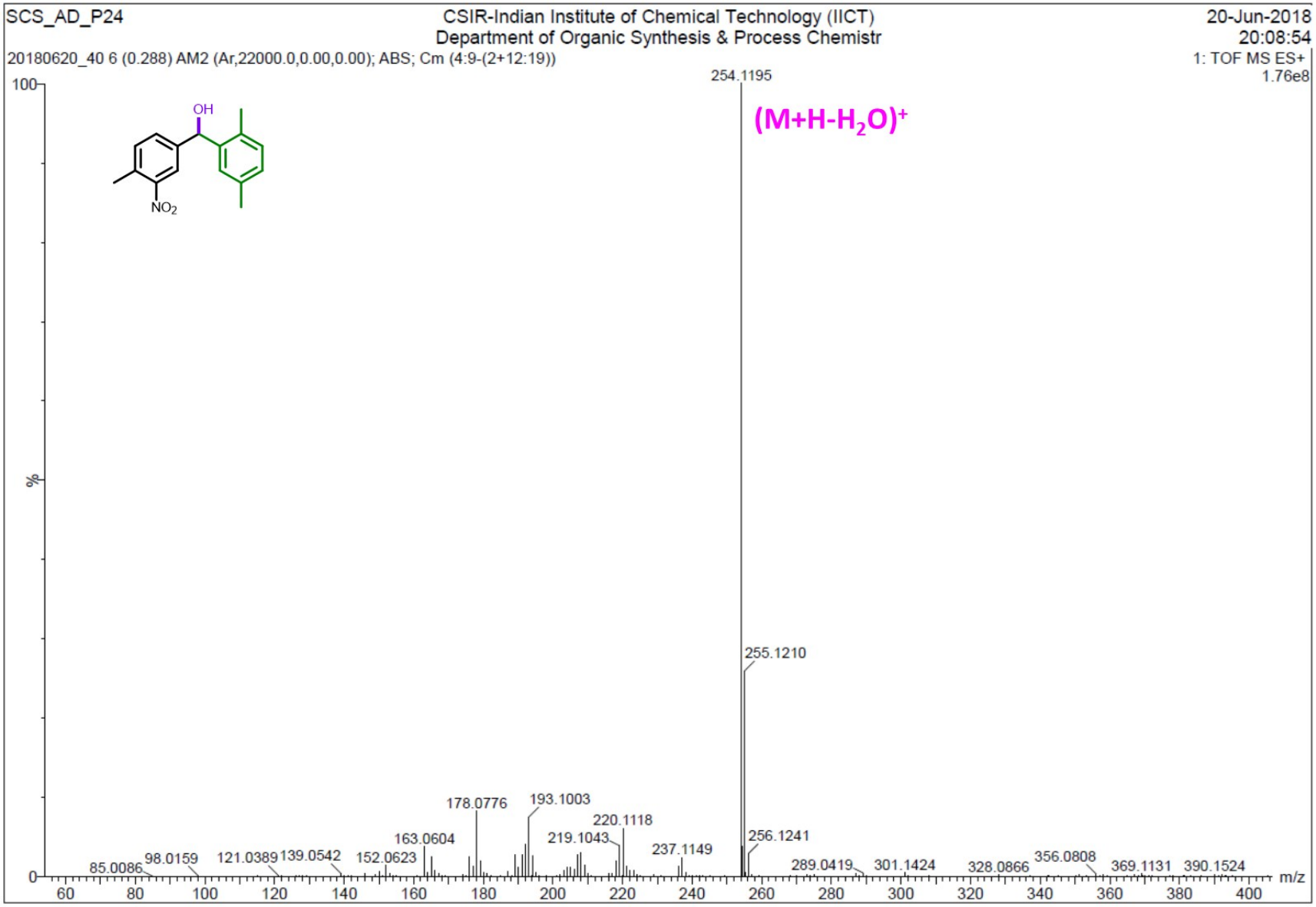


Figure S218. HRMS spectra of (2,5-dimethylphenyl) (4-methyl-3-nitrophenyl) methanol **(3v)**.

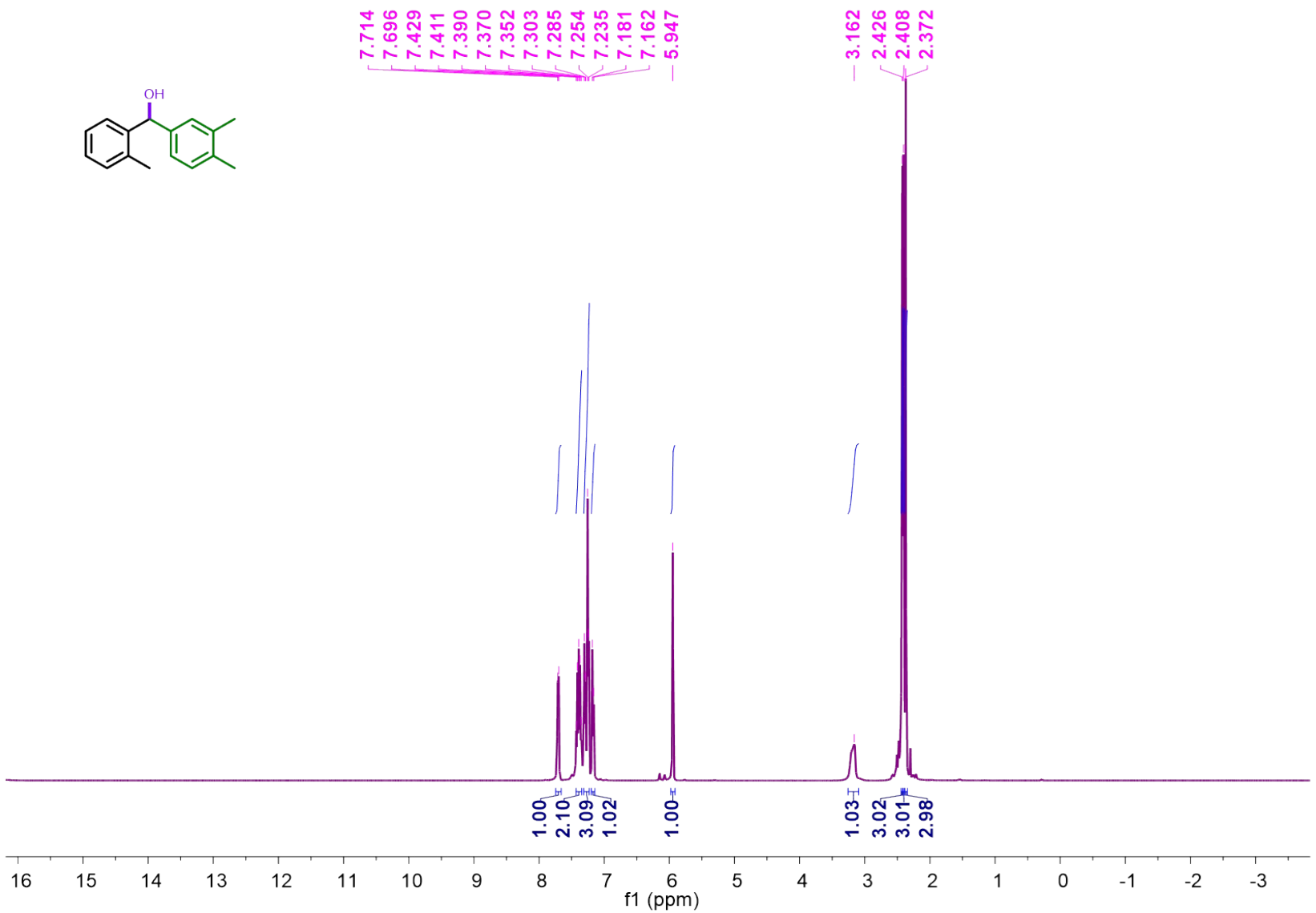


Figure S219. ^{1}H NMR spectra of (3-bromo-2-methylphenyl) (3,4-dimethylphenyl) methanol **(3w)** in $CDCl_3$.

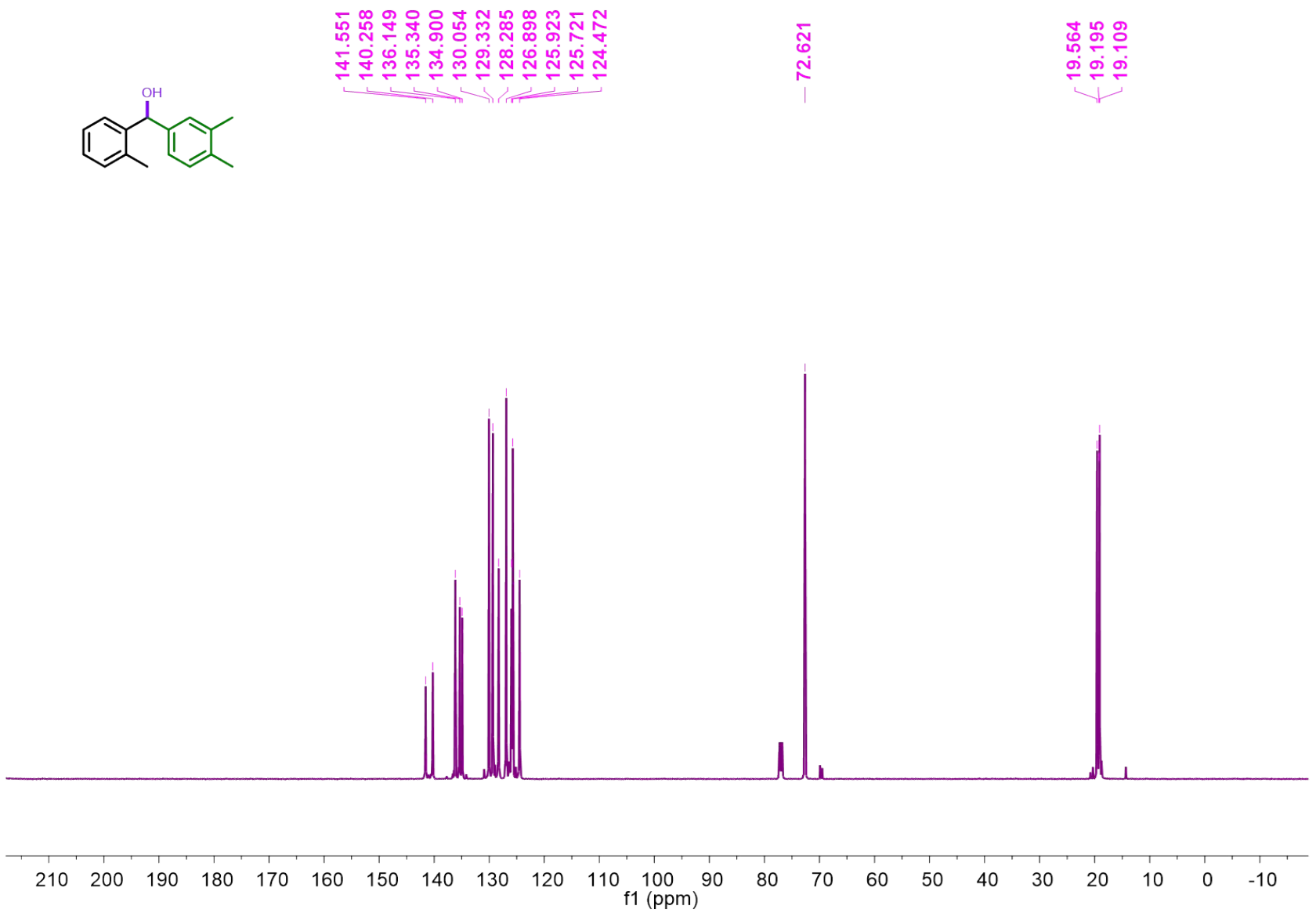


Figure S220. ^{13}C NMR spectra of (3-bromo-2-methylphenyl) (3,4-dimethylphenyl) methanol **(3w)** in $CDCl_3$.

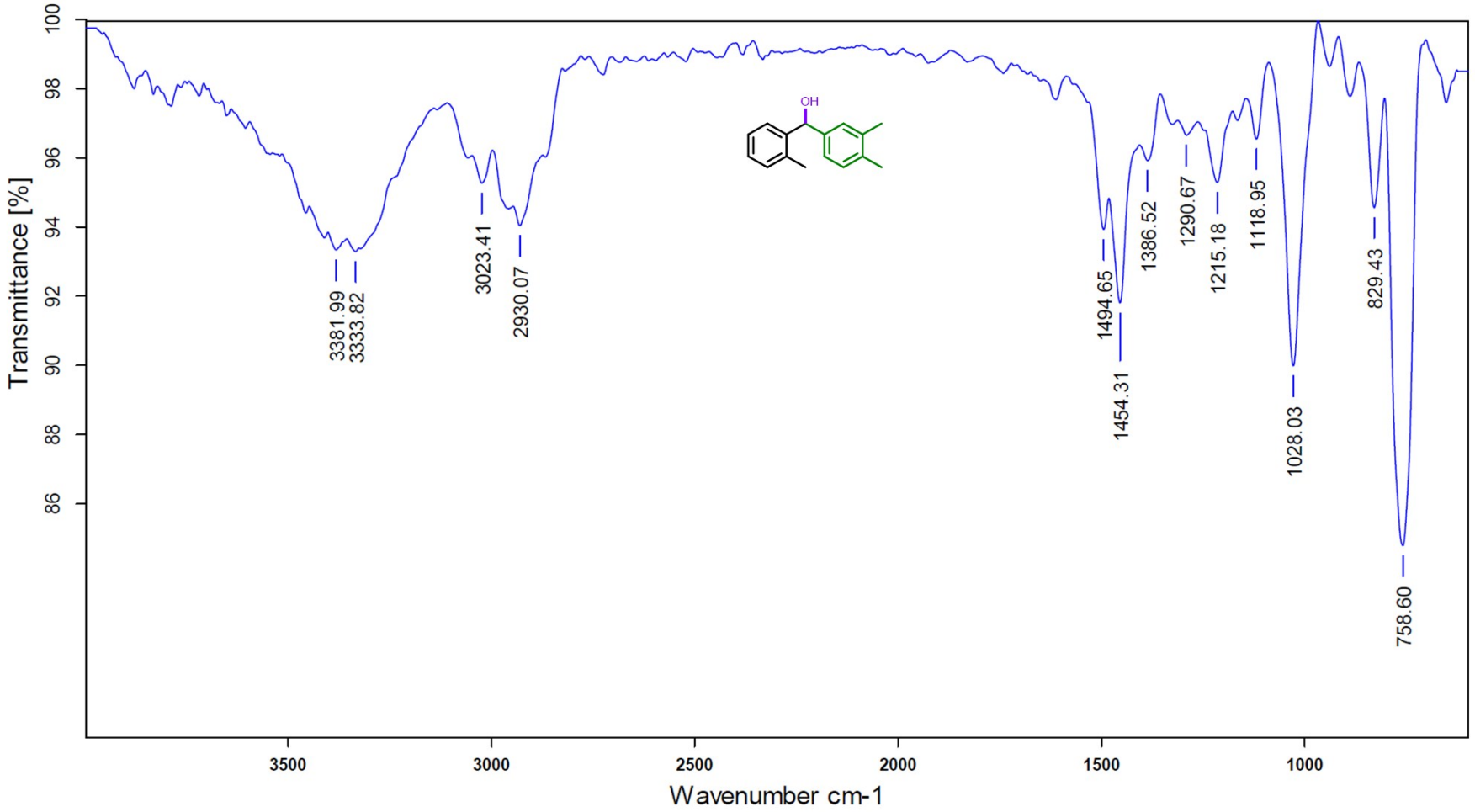


Figure S221. IR spectra of (3-bromo-2-methylphenyl) (3,4-dimethylphenyl) methanol **(3w)**.

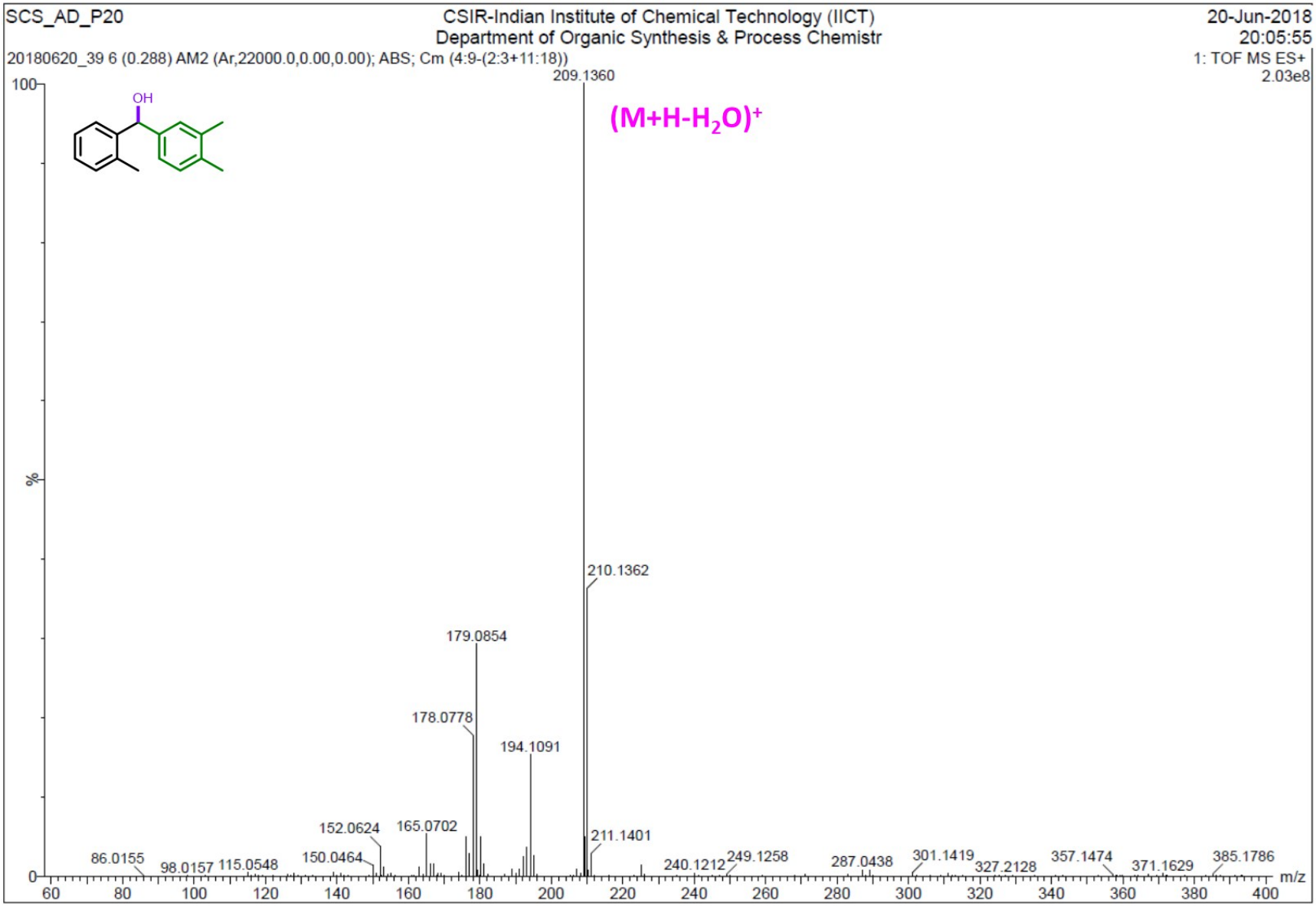


Figure S222. HRMS spectra of (3-bromo-2-methylphenyl) (3,4-dimethylphenyl) methanol **(3w)**.

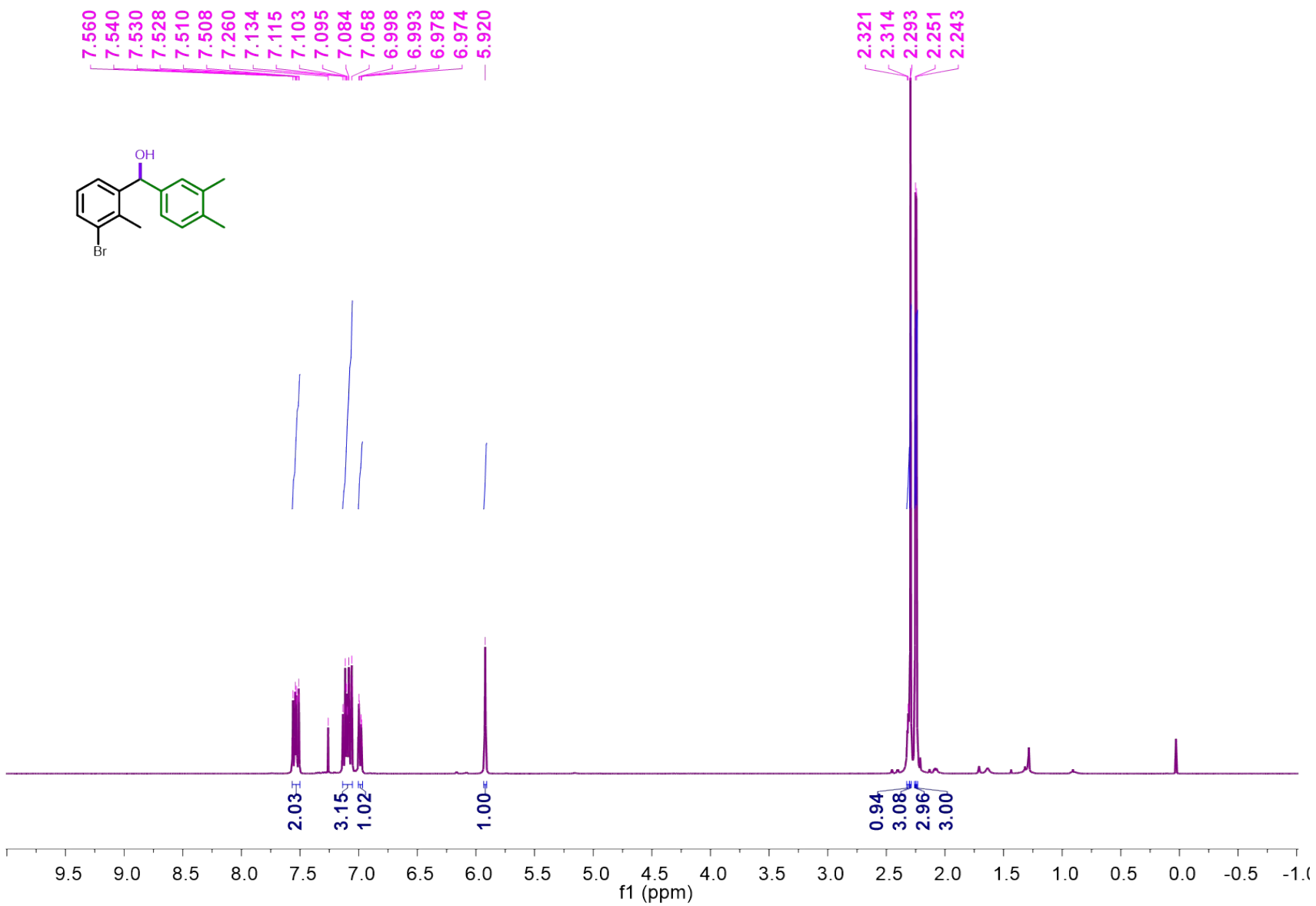


Figure S223. ^{1}H NMR spectra of (3-bromo-2-methylphenyl) (3,4-dimethylphenyl) methanol **(3x)** in $CDCl_3$.

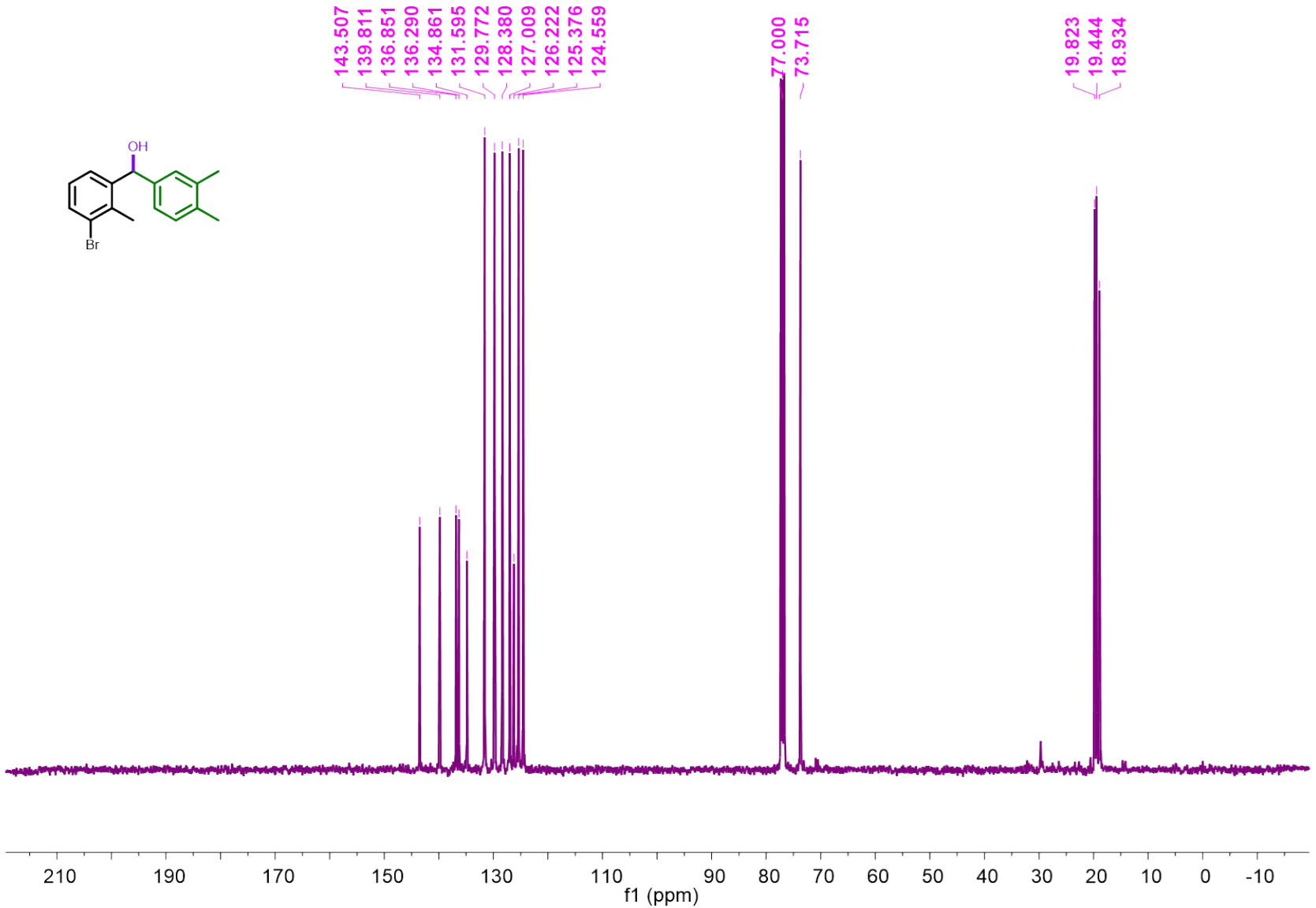


Figure S224. ^{13}C NMR spectra of (3-bromo-2-methylphenyl) (3,4-dimethylphenyl) methanol **(3x)** in $CDCl_3$.

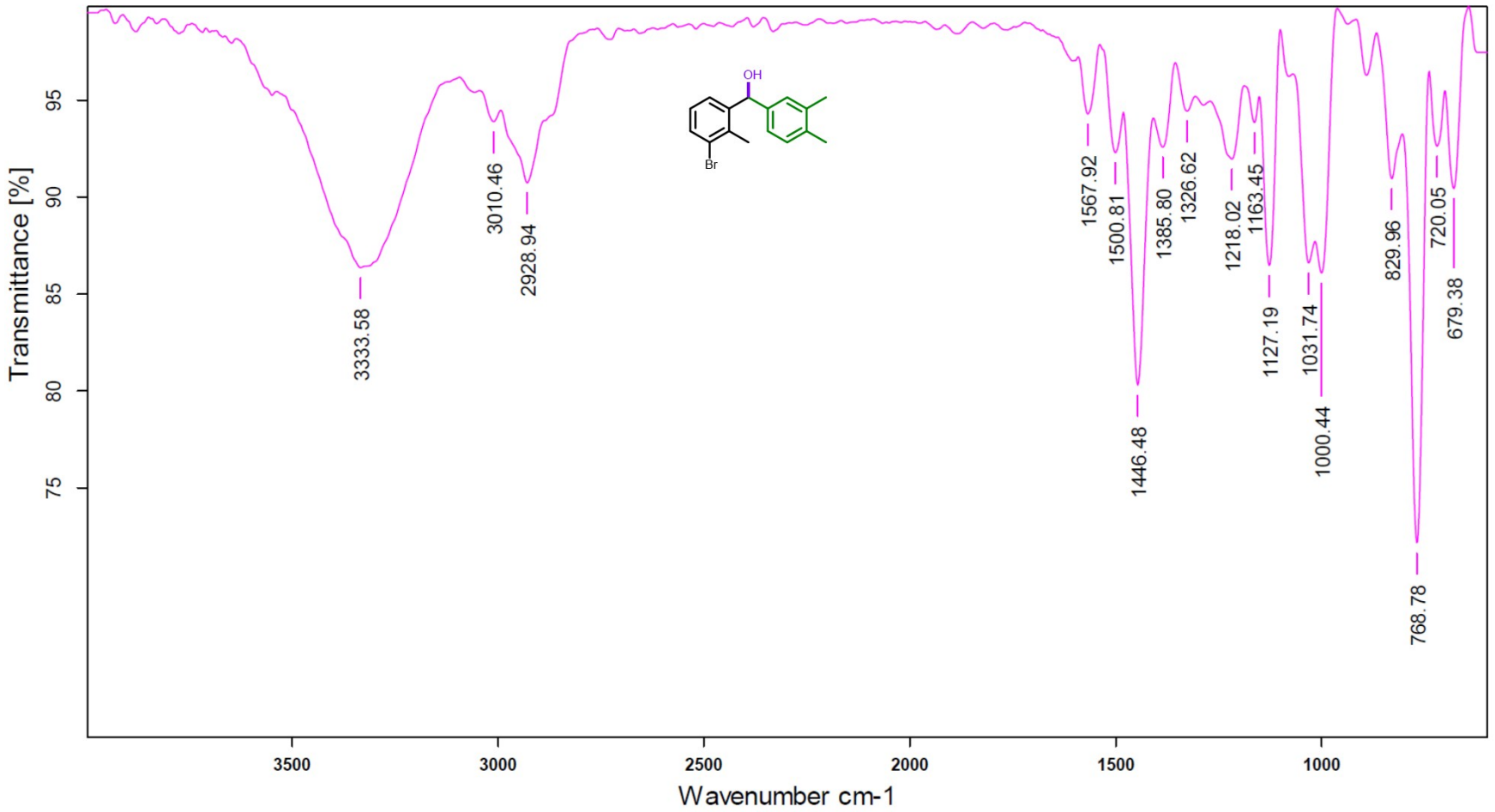


Figure S225. IR spectra of (3-bromo-2-methylphenyl) (3,4-dimethylphenyl) methanol **(3x)**.

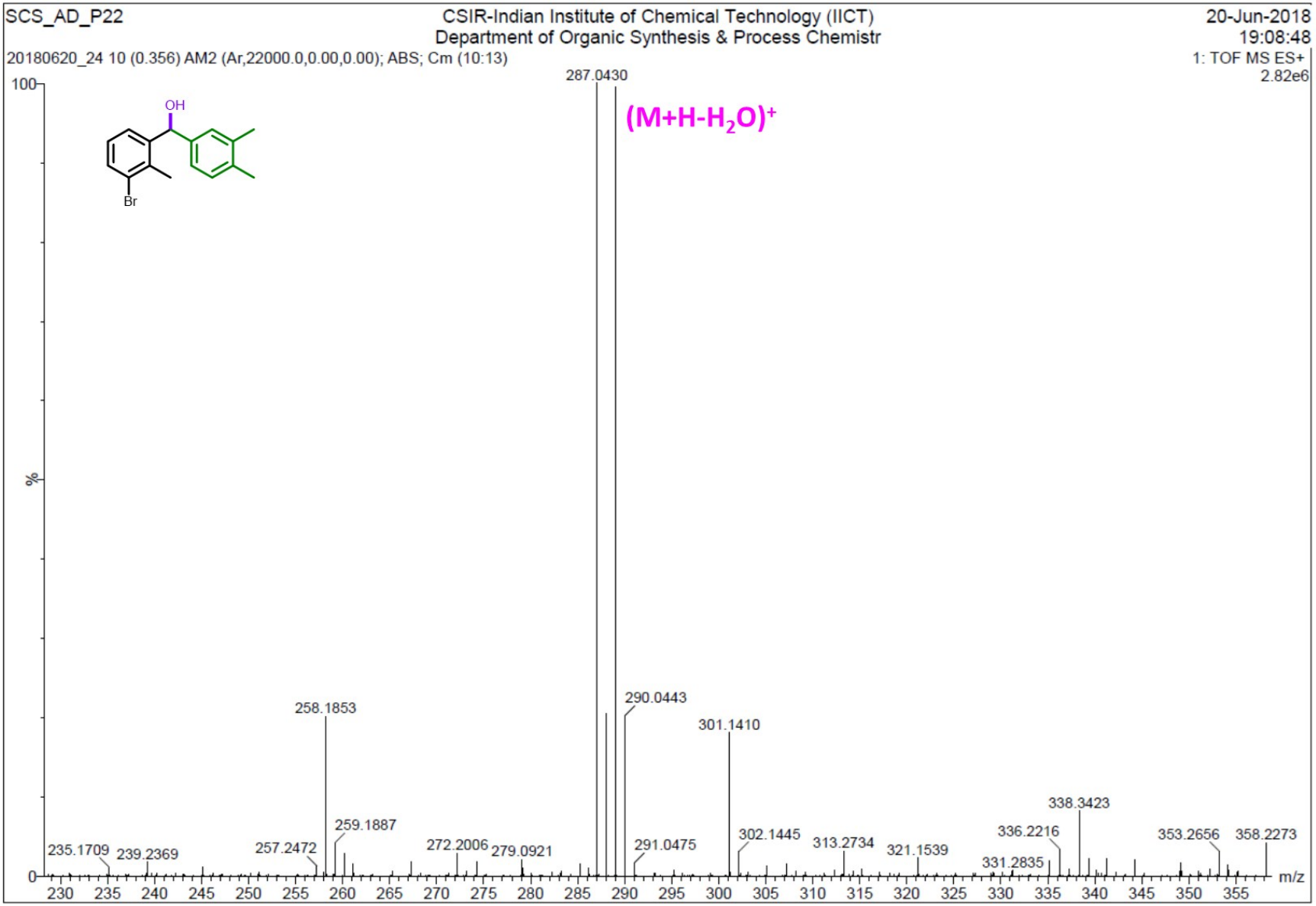


Figure S226. HRMS spectra of (3-bromo-2-methylphenyl) (3,4-dimethylphenyl) methanol **(3x)**.

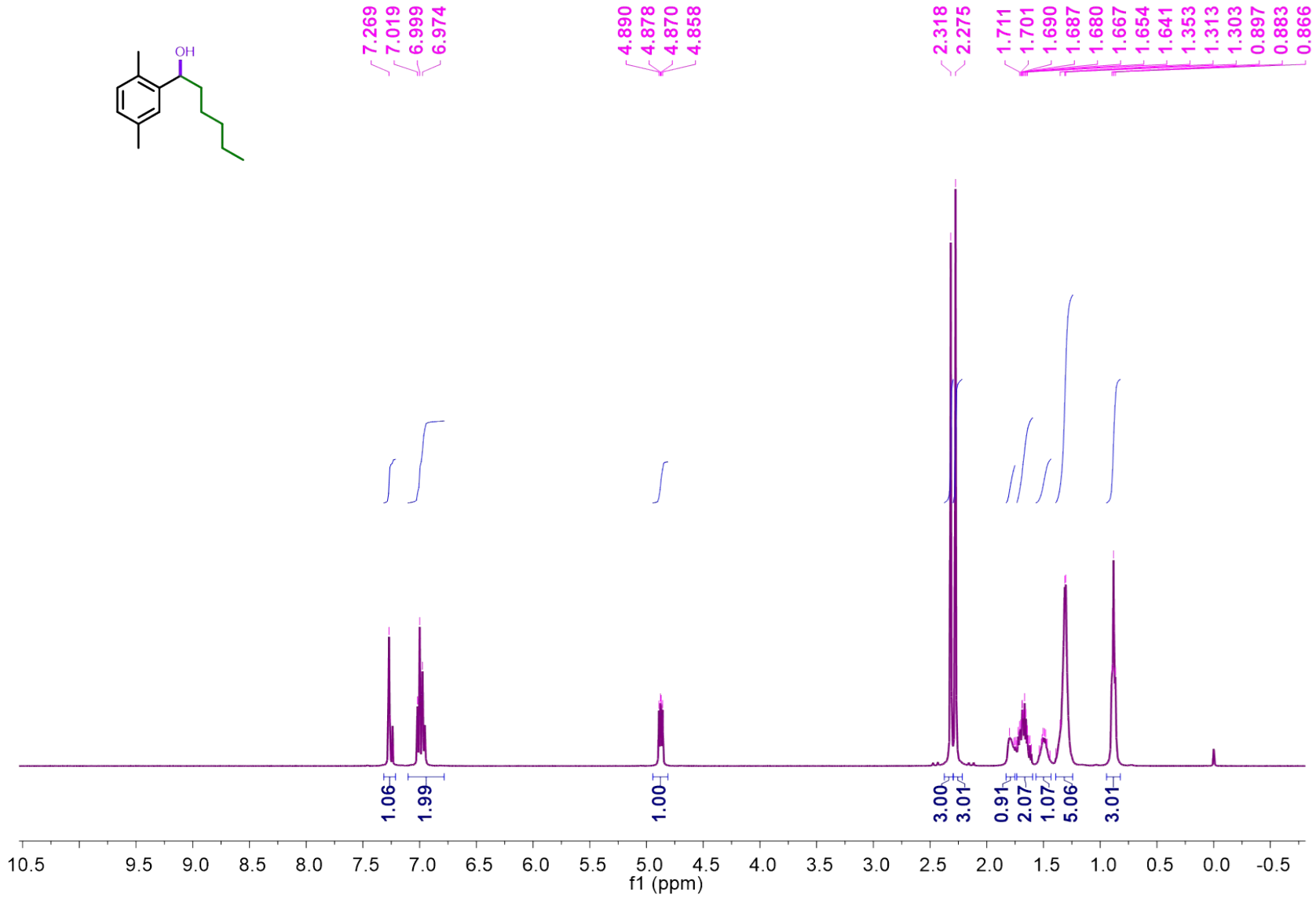


Figure S227. ^{1}H NMR spectra of 1-(2,5-dimethylphenyl) hexan-1-ol **(3y)** in $CDCl_3$.

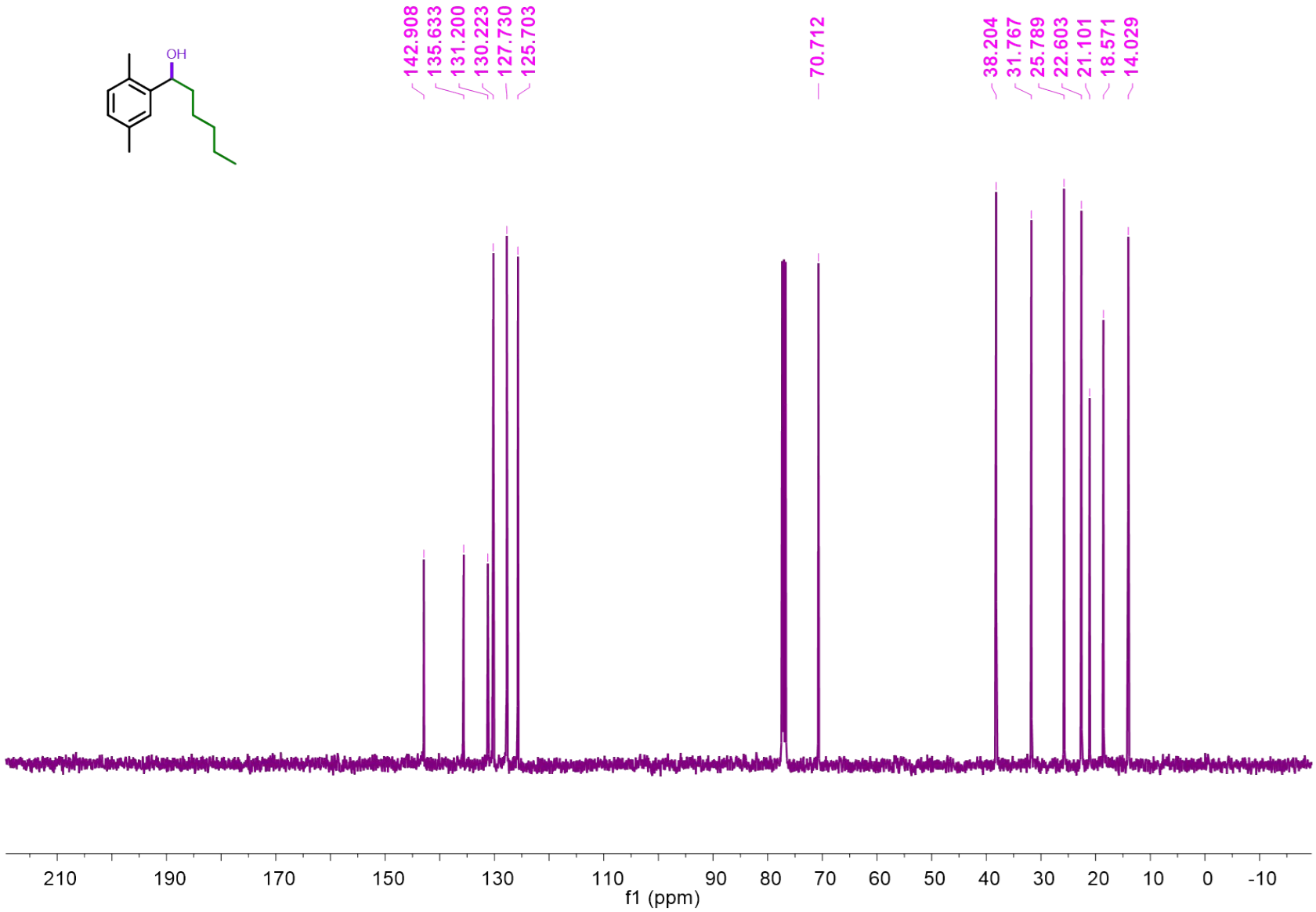


Figure S228. ^{13}C NMR spectra of 1-(2,5-dimethylphenyl) hexan-1-ol **(3y)** in $CDCl_3$.

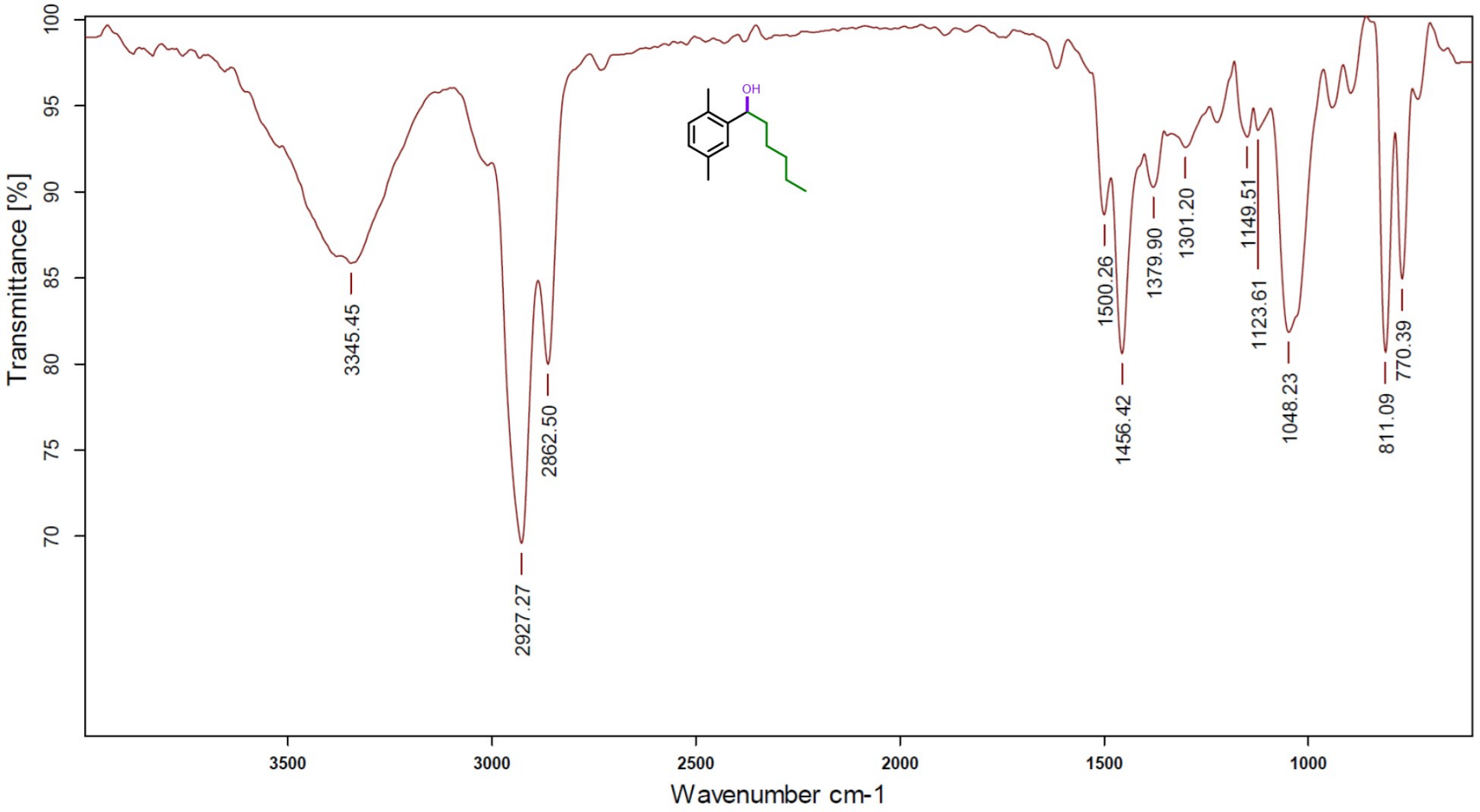


Figure S229. IR spectra of 1-(2,5-dimethylphenyl) hexan-1-ol **(3y)**

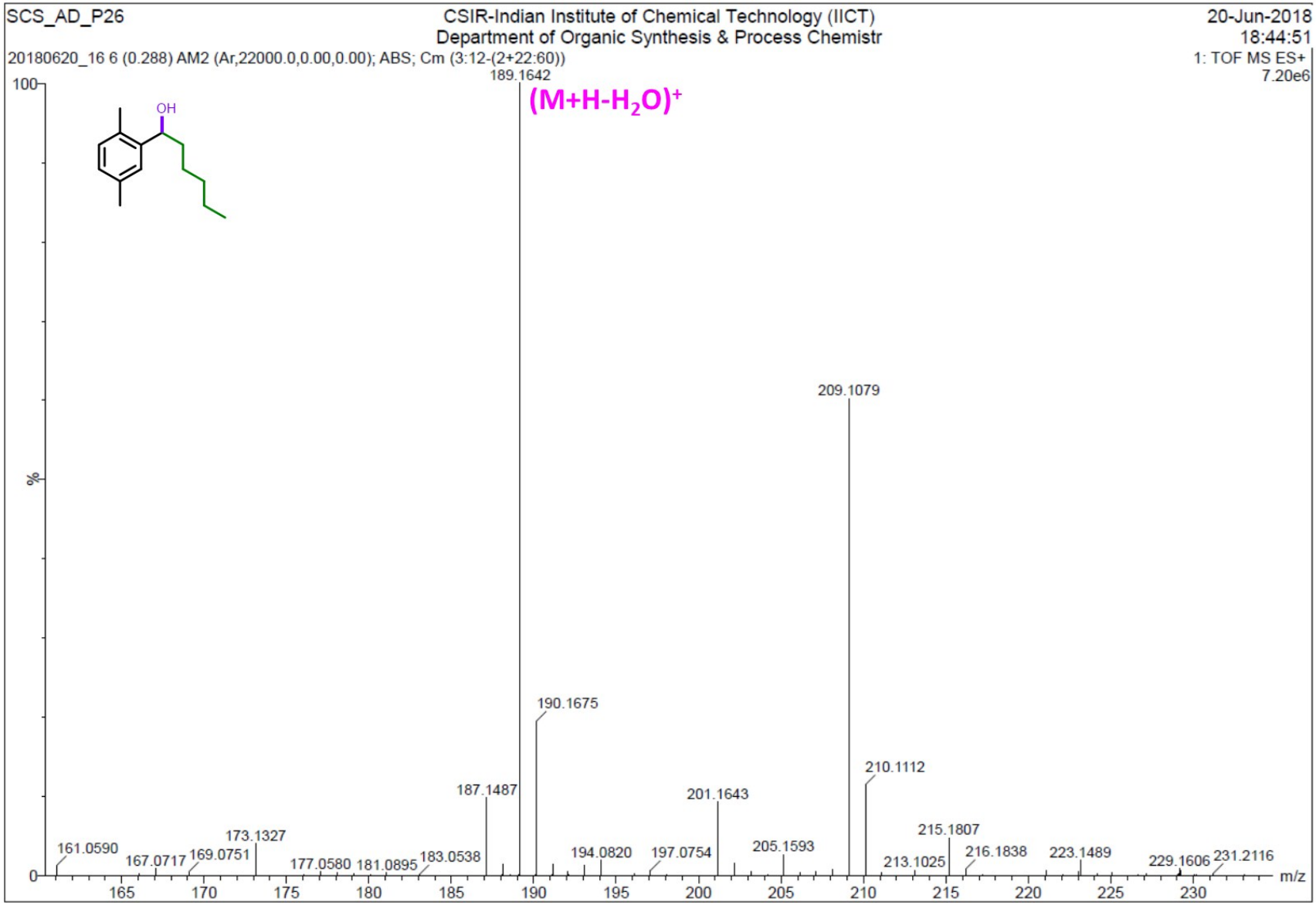


Figure S230. HRMS spectra of 1-(2,5-dimethylphenyl) hexan-1-ol **(3y)**.

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