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Supporting Information

Cross-coupling reactivity of 1,1-dichloroalkenes under palladium catalysis: Domino synthesis of diarylalkynes

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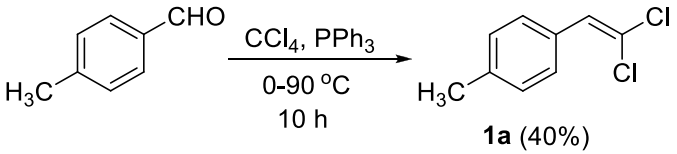
1. General Information:

The coupling reactions were performed using an oven-dried Schlenk tube under inert nitrogen atmosphere conditions. Solvents have been dried according to standard procedures. All the products were purified by silica gel column chromatography using ethyl acetate/hexane as eluent. Spectral measurements for ^{1}H and ^{13}C NMR were recorded using JEOL ECS 400 (400 MHz) and JEOL ECX 500 (500 MHz) spectrometer in $CDCl_3$ and chemical shift (δ) values are expressed in parts per million (ppm). Melting points reported are uncorrected. HRMS spectra were obtained using Electron Ionization (EI) and Electrospray Ionization (ESI) techniques with Waters CAB155 GCT Premier analyzer and Waters HAB 213 Q-TOF Premier analyzer. IR spectra were recorded using PerkinElmer FT-IR spectrum.

2. 1 Experimental procedure for substrate preparation:

Representative procedure for preparation of 1,1-dichloro-1-alkenes (1a-1k):[1]



1a (40%)

In a three neck 250 mL round bottom flask, to a solution of triphenylphosphine (39.68 mmol, 4 equiv) in CCl_4 (20 mL) was added 4-methylbenzaldehyde (9.92 mmol, 1 equiv) in portions under N_2 around 0 °C (ice bath). Then the cooling bath was detached and the reaction mixture was furthermore stirred for 10 h at 90 °C. After being cooled to rt, the reaction mixture was quenched with hexane and deposited material was filtered through a column of silica gel. The solvent was evaporated and the residue was purified by column chromatography using hexane to afford 1-(2,2-dichlorovinyl)-4-methylbenzene (**1a**) in 40% yield. The remaining substituted 1,1-dichloro-1-alkenes (**1b-1k**) were prepared based on above procedure and characterized by ^{1}H NMR, ^{13}C NMR, HRMS and IR.

2.2 Triarylbismuth reagents have been prepared using standard synthetic procedures given in "*Organobismuth Chemistry"*, by H. Suzuki and Y. Matano, Elsevier, Amsterdam, 2001.

3. Characterization data for 1,1-dichloroalkenes (1a-1k):

1-(2,2-Dichlorovinyl)-4-methylbenzene (**1a**):[2] Low melting solid (0.750 g, 40%); mp 38 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 (d, J = 8.2 Hz, 2H), 7.18 (d, J = 8.2 Hz, 2H), 6.82 (s, 1H), 2.35 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.53, 130.56, 129.16, 128.53, 128.47, 120.03, 21.31 ppm. IR (neat, cm^{-1}): 2922, 1607, 1509, 1411, 1328, 1274, 1127, 905, 810, 518. HRMS (EI^{+}): calcd for $C_9H_8Cl_2$ $[M]^{+}$ 186.0003; found 186.0009.

(2,2-Dichlorovinyl)benzene (**1b**):[2] Colorless liquid (0.865 g, 51%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55-7.53 (m, 2H), 7.40-7.30 (m, 3H), 6.87 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 133.38, 128.62, 128.54, 128.45, 120.97 ppm. IR (neat, cm^{-1}): 1608, 1491, 1446, 1273, 925, 855, 819, 751, 691. HRMS (EI^{+}): calcd for $C_8H_6Cl_2$ $[M]^{+}$ 171.9847; found 171.9845.

4-(2,2-Dichlorovinyl)benzonitrile (**1c**): White solid (0.589 g, 30%); mp 61-63 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68-7.62 (m, 4H), 6.88 (s, 1H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 137.72, 132.24, 129.15, 126.99, 124.68, 118.45, 111.89 ppm. IR (neat, cm^{-1}): 3028, 2227, 1739, 1604, 1504, 1409, 1177, 916, 874, 831, 814. HRMS (EI^{+}): calcd for $C_9H_5Cl_2N$ $[M]^{+}$ 196.9799; found 196.9797.

1-(2,2-Dichlorovinyl)-4-nitrobenzene (**1d**):[2] Light yellow solid (1 g, 46%); mp 88 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25-8.22 (m, 2H), 7.72-7.68 (m, 2H), 6.94 (s, 1H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 147.15, 139.56, 129.37, 126.70, 125.28, 123.75 ppm. IR (neat, cm^{-1}): 3109, 3042, 1605, 1593, 1491, 1342, 1279, 1111, 867, 860, 689, 505. HRMS (EI^{+}): calcd for $C_8H_5Cl_2NO_2$ $[M]^{+}$ 216.9697; found 216.9692.

1-(2,2-Dichlorovinyl)-4-methoxybenzene (**1e**):[2] Colorless liquid (0.950 g, 47%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50 (d, J = 9.2 Hz, 2H), 6.90 (d, J = 9.2 Hz, 2H), 6.79 (s, 1H), 3.83 (s, 3H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.55, 130.05, 128.03, 125.98, 118.80, 113.87, 55.29 ppm. IR (neat, cm^{-1}): 1605, 1510, 1252, 1178, 1032, 908, 821. HRMS (EI^{+}): calcd for $C_9H_8Cl_2O$ $[M]^{+}$ 201.9952; found 201.9955.

2-(2,2-Dichlorovinyl)naphthalene (**1f**):[3] White solid (0.935 g, 42%); mp 84 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25 (s, 1H), 8.08-8.06 (m, 3H), 7.91-7.88 (m, 1H), 7.75-7.73 (m, 2H), 7.50 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 133.04, 132.93, 130.81, 128.64, 128.36, 128.27, 128.04, 127.62, 126.73, 126.47, 125.87, 121.17 ppm. IR (neat, cm^{-1}): 3031, 1588, 1366, 1272, 908, 895, 873, 823, 750. HRMS (EI^{+}): calcd for $C_{12}H_8Cl_2$ $[M]^{+}$ 222.0003; found 222.0008.

1-(Benzyloxy)-4-(2,2-dichlorovinyl)-2-methoxybenzene (**1g**): Off-white solid (1.72 g, 56%); mp 84-86 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44-7.31 (m, 5H), 7.18 (d, J = 2.3 Hz, 1H), 7.02 (dd, J = 8.7, 1.8 Hz, 1H), 6.86 (d, J = 8.2 Hz, 1H), 6.77 (s, 1H), 5.18 (s, 2H), 3.90 (s, 3H) ppm. ^{13}C

NMR (100 MHz, $CDCl_3$): δ 149.20, 148.32, 136.74, 128.60, 128.17, 127.94, 127.19, 126.55, 121.92, 119.02, 113.31, 111.85, 70.82, 55.99 ppm. IR (neat, cm^{-1}): 2867, 1597, 1578, 1521, 1457, 1410, 1290, 1258, 1239, 1140, 1029, 997, 895, 876, 742. HRMS (EI^+): calcd for $C_{16}H_{14}Cl_2O_2$ $[M]^+$ 308.0371; found 308.0379.

1-Bromo-4-(2,2-dichlorovinyl)benzene (**1h**):[2] Colorless liquid (1.56 g, 63%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.50 (d, J = 8.2 Hz, 2H), 7.40 (d, J = 8.3 Hz, 2H), 6.79 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 132.21, 131.66, 130.10, 127.46, 122.48, 121.81 ppm. IR (neat, cm^{-1}): 1607, 1585, 1486, 1397, 1264, 1076, 1010, 912, 861, 821, 808, 512. HRMS (EI^+): calcd for $C_8H_5BrCl_2$ $[M]^+$ 249.8952; found 249.8954.

1-Chloro-4-(2,2-dichlorovinyl)benzene (**1i**):[2] Colorless liquid (1.38 g, 67%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.47 (d, J = 8.2 Hz, 2H), 7.34 (d, J = 8.2 Hz, 2H), 6.81(s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 134.23, 131.78, 129.86, 128.70, 127.40, 121.71 ppm. IR (neat, cm^{-1}): 1610, 1592, 1564, 1490, 1402, 1265, 1092, 1013, 912, 862, 826, 812, 514. HRMS (EI^+): calcd for $C_8H_5Cl_3$ $[M]^+$ 205.9457; found 205.9453.

2-(2,2-Dichlorovinyl)furan (**1j**):[2] Colorless liquid (0.791 g, 49%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.43 (d, J =1.8 Hz, 1H), 6.80-6.79 (m, 2H), 6.48-6.46 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.51, 142.44, 119.26, 118.48, 111.72, 111.26 ppm. IR (neat, cm^{-1}): 1616, 1491, 1144, 1022, 956, 909, 846, 739, 672. HRMS (EI^+): calcd for $C_6H_4Cl_2O$ $[M]^+$ 161.9639; found 161.9634.

2-(2,2-Dichlorovinyl)thiophene (**1k**): Colorless liquid (0.695 g, 39%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.38 (d, J = 5.5 Hz, 1H), 7.20 (d, J = 3.2 Hz, 1H), 7.06-7.03 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 136.24, 129.35, 127.21, 126.64, 122.85, 118.61 ppm. IR (neat, cm^{-1}): 1601, 1420, 1280, 1212, 1052, 908, 868, 861, 712, 752, 647. HRMS (EI^+): calcd for $C_6H_4Cl_2S$ $[M]^+$ 177.9411; found 177.9416.

4. Representative coupling procedure for diarylalkyne preparation from 1,1-dichloroalkenes:

To an oven-dried Schlenk tube under nitrogen atmosphere, 1,1-dichloroalkene (0.75 mmol, 3 equiv), Ar_3Bi (0.25 mmol, 1 equiv), $PdCl_2$ (0.0225 mmol, 0.09 equiv), PPh_3 (0.09 mmol, 0.36 equiv), Cs_2CO_3 (1.5 mmol, 6 equiv), TBAB (0.25 mmol, 1 equiv) and DMSO (3 mL) were added. The reaction mixture was stirred in an oil bath at 90 ^oC for 2 h. After that, the contents were brought to rt, quenched with water and then extracted with ethyl acetate (30 mL). The organic extract was treated with brine, dried over anhydrous $MgSO_4$ and concentrated. The crude was subjected to silica gel column chromatography using hexane and ethyl acetate as eluent. Considering threefold coupling from the bismuth reagent, 0.75 mmol of product yield was calculated as 100% yield.

5. Characterization data for diarylalkynes (2.1-2.10 and 3.1-3.30):

1-methyl-4-(phenylethynyl)benzene (**2.1**):[4] White solid (101 mg, 71%); mp 69-70 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.52-7.54 (m, 2H), 7.43 (d, J = 8.0 Hz, 2H), 7.32-7.36 (m, 3H), 7.16 (d, J = 8.0 Hz, 2H), 2.37 (s, 3H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 138.37, 131.54, 131.49, 129.10, 128.30, 128.05, 123.48, 120.19, 89.54, 88.70, 21.50 ppm. IR (neat, cm^{-1}): 3050, 3029, 2919, 1593, 1509, 1484, 1440, 1210, 1017, 817, 754, 689, 515. HRMS (EI$^+$): calcd for $C_{15}H_{12}$ $[M]^+$ 192.0939; found 192.0938.

1,2-dip-tolylethyne (**2.2**):[5] White solid (117 mg, 75%); mp 138-139 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41 (d, J = 8.3 Hz, 4H), 7.15 (d, J = 7.8 Hz, 4H), 2.36 (s, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.16, 131.40, 129.07, 120.32, 88.83, 21.50 ppm. IR (neat, cm^{-1}): 3436, 3024, 2920, 2856, 1655, 1515, 1436, 1034, 814, 514. HRMS (ESI$^+$): calcd for $C_{16}H_{14}$ $[M]^+$ 206.1096; found 206.1093.

1-fluoro-4-(p-tolylethynyl)benzene (**2.3**):[6] White solid (115 mg, 73%); mp 90-91 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48-7.52 (m, 2H), 7.42 (d, J = 7.8 Hz, 2H), 7.16 (d, J = 8.2 Hz, 2H), 7.04 (t, J = 8.7 Hz, 2H), 2.37 (s, 3H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 162.38 (d, $J_{C\text{-}F}$ = 246.8 Hz), 138.46, 133.38 (d, $J_{C\text{-}F}$ = 8.3 Hz), 131.43, 129.13, 119.99, 119.58, 115.57 (d, $J_{C\text{-}F}$ = 21.5 Hz), 89.20, 87.61, 21.50 ppm. IR (neat, cm^{-1}): 3026, 2923, 2856, 1595, 1511, 1499, 1234, 1216, 1157, 836, 815, 518. HRMS (EI$^+$): calcd for $C_{15}H_{11}F$ $[M]^+$ 210.0845; found 210.0849.

1-chloro-4-(p-tolylethynyl)benzene (**2.4**):[7] White solid (87 mg, 51%); mp 148-150 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41-7.46 (m, 4H), 7.31 (d, J = 8.7 Hz, 2H), 7.16 (d, J = 7.8 Hz, 2H), 2.37 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.66, 134.01, 132.72, 131.47, 129.16, 128.64, 121.97, 119.80, 90.49, 87.59, 21.52 ppm. IR (neat, cm^{-1}): 2923, 2215, 1912, 1658, 1509, 1487, 1397, 1087, 1011, 832, 816, 512. HRMS (EI$^+$): calcd for $C_{15}H_{11}Cl$ $[M]^+$ 226.0549; found 226.0543.

1-chloro-3-(p-tolylethynyl)benzene (**2.5**):[13] White solid (76 mg, 45%); mp 71-73 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50-7.51 (m, 1H), 7.37-7.42 (m, 3H), 7.23-7.30 (m, 2H), 7.16 (d, J = 7.8 Hz, 2H), 2.36 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.82, 134.12, 131.55, 131.36, 129.62, 129.52, 129.16, 128.29, 125.20, 119.63, 90.75, 87.29, 21.54 ppm. IR (neat, cm^{-1}): 3031, 2918, 2224, 1590, 1557, 1508, 1407, 1072, 893, 882, 815, 789, 681, 523, 445. HRMS (EI$^+$): calcd for $C_{15}H_{11}Cl$ $[M]^+$ 226.0549; found 226.0544.

2-(p-tolylethynyl)naphthalene (**2.6**):[14] White solid (133 mg, 73%); mp 127-129 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05 (s, 1H), 7.80-7.82 (m, 3H), 7.58 (d, J = 8.5 Hz, 1H), 7.46-7.52 (m, 4H), 7.18 (d, J = 7.8 Hz, 2H), 2.38 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ

138.45, 133.02, 132.69, 131.52, 131.25, 129.14, 128.43, 127.94, 127.74, 126.54, 126.48, 120.75, 120.15, 89.90, 89.11, 21.53 ppm. IR (neat, cm^{-1}): 3055, 3023, 1592, 1508, 864, 817, 743, 520, 480. HRMS (EI^+): calcd for $C_{19}H_{14}$ $[M]^+$ 242.1096; found 242.1098.

1-isopropoxy-4-(p-tolylethynyl)benzene (**2.7**): White solid (118 mg, 63%); mp 158-160 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 7.39-7.45 (m, 4H), 7.14 (d, J = 7.8 Hz, 2H), 6.84 (d, J = 8.7 Hz, 2H), 4.54-4.60 (m, 1H), 2.36 (s, 3H), 1.34 (d, J = 6.4 Hz, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.82, 137.94, 132.97, 131.30, 129.05, 120.51, 115.65, 115.17, 88.74, 88.01, 69.87, 21.97, 21.48 ppm. IR (neat, cm^{-1}): 2974, 2926, 1599, 1515, 1382, 1284, 1247, 1117, 951, 838, 813, 522. HRMS (EI^+): calcd for $C_{18}H_{18}O$ $[M]^+$ 250.1358; found 250.1355.

1-methyl-3-(p-tolylethynyl)benzene (**2.8**):[8] White solid (103 mg, 66%); mp 71-72 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 7.42 (d, J = 7.8 Hz, 2H), 7.32-7.36 (m, 2H), 7.21-7.25 (m, 1H), 7.12-7.16 (m, 3H), 2.37 (s, 3H), 2.35 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.28, 137.95, 132.11, 131.45, 129.08, 128.96, 128.58, 128.19, 123.20, 120.23, 89.17, 88.85, 21.51, 21.24 ppm. IR (neat, cm^{-1}): 2960, 2924, 2853, 1577, 1510, 1455, 1260, 1088, 1018, 816, 799, 783, 690. HRMS (ESI^+): calcd for $C_{16}H_{15}$ $[M+H]^+$ 207.1174; found 207.1178.

1-methoxy-4-(p-tolylethynyl)benzene (**2.9**): Light yellow solid (120 mg, 72%); mp 119-120 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 7.46 (d, J = 8.7 Hz, 2H), 7.41 (d, J = 8.2 Hz, 2H), 7.14 (d, J = 7.8 Hz, 2H), 6.87 (d, J = 9.2 Hz, 2H), 3.83 (s, 3H), 2.36 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.46, 138.0, 132.96, 131.32, 129.06, 120.47, 115.57, 113.94, 88.63, 88.17, 55.28, 21.48 ppm. IR (neat, cm^{-1}): 3028, 2967, 2936, 2841, 1603, 1511, 1463, 1451, 1286, 1246, 1027, 829, 817, 525. HRMS (EI^+): calcd for $C_{16}H_{14}O$ $[M]^+$ 222.1045; found 222.1048.

1-methoxy-3-(p-tolylethynyl)benzene (**2.10**):[8] White solid (114 mg, 68%); mp 60-61 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 7.43 (d, J = 7.8 Hz, 2H), 7.23-7.27 (m, 1H), 7.12-7.17 (m, 3H), 7.05-7.06 (m, 1H), 6.87-6.90 (m, 1H), 3.83 (s, 3H), 2.37 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.31, 138.44, 131.51, 129.35, 129.11, 124.44, 124.12, 120.07, 116.23, 114.79, 89.37, 88.62, 55.28, 21.51 ppm. IR (neat, cm^{-1}): 2918, 1594, 1573, 1509, 1477, 1463, 1321, 1222, 1039, 815, 777, 685. HRMS (ESI^+): calcd for $C_{16}H_{14}O$ $[M]^+$ 222.1045; found 222.1040.

1,2-diphenylethyne (**3.1**):[4] White solid (98 mg, 74%); mp 59-61 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 7.53-7.56 (m, 4H), 7.32-7.39 (m, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 131.59, 128.32, 128.23, 123.24, 89.35 ppm. IR (neat, cm^{-1}): 3079, 3063, 3019, 1600, 1571, 1493, 1442, 1069, 1025, 915, 755, 688, 508. HRMS (EI^+): calcd for $C_{14}H_{10}$ $[M]^+$ 178.0783; found 178.0787.

1-methyl-4-(phenylethynyl)benzene (**3.2**):[4] White solid (112 mg, 78%); mp 69-70 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52-7.54 (m, 2H), 7.43 (d, J = 8.2 Hz, 2H), 7.32-7.37 (m, 3H), 7.16 (d, J = 7.8 Hz, 2H), 2.37 (s, 3H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 138.37, 131.54, 131.49, 129.10, 128.30, 128.05, 123.48, 120.19, 89.54, 88.71, 21.52 ppm. IR (neat, cm^{-1}): 3050, 3029, 2919, 1593, 1509, 1484, 1440, 1210, 1017, 817, 754, 689, 515. HRMS (EI$^+$): calcd for $C_{15}H_{12}$ $[M]^+$ 192.0939; found 192.0938.

1-chloro-4-(phenylethynyl)benzene (**3.3**):[13] White solid (79 mg, 50%); mp 78-80 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51-7.54 (m, 2H), 7.45-7.47 (m, 2H), 7.32-7.36 (m, 5H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 134.23, 132.80, 131.58, 128.68, 128.47, 128.38, 122.89, 121.75, 90.28, 88.21 ppm. IR (neat, cm^{-1}): 3049, 1590, 1496, 1481, 1440, 1399, 1265, 1091, 751, 732, 687, 512, 432. HRMS (EI$^+$): calcd for $C_{14}H_9Cl$ $[M]^+$ 212.0393; found 212.0394.

1-methoxy-4-(phenylethynyl)benzene (**3.4**):[14] Light yellow solid (110 mg, 71%); mp 52-54 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47-7.53 (m, 4H), 7.31-7.36 (m, 3H), 6.88 (d, J = 9.2 Hz, 2H), 3.83 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.59, 133.04, 131.44, 128.29, 127.92, 123.57, 115.36, 113.98, 89.35, 88.05, 55.29 ppm. IR (neat, cm^{-1}): 3054, 2960, 2836, 1605, 1595, 1510, 1440, 1248, 1108, 1030, 833, 753, 691, 523. HRMS (EI$^+$): calcd for $C_{15}H_{12}O$ $[M]^+$ 208.0888; found 208.0880.

4-(phenylethynyl)benzonitrile (**3.5**):[14] Yellow solid (76 mg, 50%); mp 99-101 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.60-7.65 (m, 4H), 7.54-7.56 (m, 2H), 7.38-7.39 (m, 3H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 132.06, 132.04, 131.78, 129.12, 128.50, 128.25, 122.22, 118.51, 111.47, 93.78, 87.71 ppm. IR (neat, cm^{-1}): 3088, 2227, 1603, 1503, 1443, 1408, 1071, 843, 762, 692, 557, 532. HRMS (EI$^+$): calcd for $C_{15}H_9N$ $[M]^+$ 203.0735; found 203.0735.

4-((4-methoxyphenyl)ethynyl)benzonitrile (**3.6**):[14] White solid (109 mg, 75%); mp 146-148 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56-7.63 (m, 4H), 7.48 (d, J = 8.7 Hz, 2H), 6.90 (d, J = 8.7 Hz, 2H), 3.84 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.26, 133.35, 132.01, 131.84, 128.63, 118.65, 114.23, 114.15, 111.00, 94.07, 86.70, 55.35 ppm . IR (neat, cm^{-1}): 2971, 2226, 2213, 1599, 1566, 1511, 1445, 1408, 1256, 1108, 834, 556, 538. HRMS (EI$^+$): calcd for $C_{16}H_{11}NO$ $[M]^+$ 233.0841; found 233.0856.

4-((4-fluorophenyl)ethynyl)benzonitrile (**3.7**):[9] Off-white solid (116 mg, 70%); mp 116-118 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58-7.65 (m, 4H), 7.51 -7.55 (m, 2H), 7.05-7.10 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.95 (d, $J_{C\text{-}F}$ = 252 Hz), 133.76 (d, $J_{C\text{-}F}$ = 8.6 Hz), 132.06, 131.99, 128.02, 118.48, 118.28, 115.88 (d, $J_{C\text{-}F}$ = 22 Hz), 111.53, 92.64, 87.42 ppm. IR (neat, cm^{-1}): 3056, 2963, 2227, 2209, 1593, 1508, 1229, 1153, 1130, 837, 810, 554, 532. HRMS (EI$^+$): calcd for $C_{15}H_8NF$ $[M]^+$ 221.0641; found 221.0652.

4-((4-chlorophenyl)ethynyl)benzonitrile (**3.8**):[9] White solid (84 mg, 51%); mp 179-180 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59-7.66 (m, 4H), 7.46-7.48 (m, 2H), 7.34-7.37 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 135.25, 132.98, 132.09, 132.05, 128.89, 127.85, 120.68, 118.44, 111.69, 92.52, 88.56 ppm. IR (neat, cm^{-1}): 3058, 2965, 2229, 1603, 1500, 1486, 1398, 1273, 1092, 829, 558, 529. HRMS (EI^+): calcd for $C_{15}H_8ClN$ $[M]^+$ 237.0345; found 237.0348.

1-nitro-4-(phenylethynyl)benzene (**3.9**):[14] Light yellow solid (85 mg, 51%); mp 106-108 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.22 (d, J = 9.2 Hz, 2H), 7.67 (d, J = 9.2 Hz, 2H), 7.55-7.57 (m, 2H), 7.37-7.40 (m, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.96, 132.26, 131.83, 130.26, 129.27, 128.53, 123.64, 122.08, 94.69, 87.53 ppm. IR (neat, cm^{-1}): 3082, 3103, 2925, 2215, 1591, 1510, 1494, 1345, 1335, 1309, 1286, 1105, 858, 764, 689. HRMS (EI^+): calcd for $C_{14}H_9NO_2$ $[M]^+$ 223.0633; found 223.0631.

1-fluoro-4-((4-nitrophenyl)ethynyl)benzene (**3.10**):[10] Pale yellow solid (100 mg, 55%); mp 111-113 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.22 (d, J = 8.7 Hz, 2H), 7.65 (d, J = 8.7 Hz, 2H), 7.53-7.56 (m, 2H), 7.09 (t, J = 8.7 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.04 (d, $J_{C\text{-}F}$ = 249.8 Hz), 146.98, 133.84 (d, $J_{C\text{-}F}$ = 8.6 Hz), 132.21, 130.04, 123.65, 118.17, 115.93 (d, $J_{C\text{-}F}$ = 21.9 Hz), 93.57, 87.26 ppm. IR (neat, cm^{-1}): 3097, 3078, 2929, 2210, 1588, 1517, 1343, 1232, 1149, 853, 838, 748, 685, 511. HRMS (EI^+): calcd for $C_{14}H_8FNO_2$ $[M]^+$ 241.0539; found 241.0531.

1-chloro-4-((4-nitrophenyl)ethynyl)benzene (**3.11**):[15] Pale yellow solid (87 mg, 45%); mp 136-138 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.23 (d, J = 9.2 Hz, 2H), 7.66 (d, J = 8.7 Hz, 2H), 7.49 (d, J = 8.7 Hz, 2H), 7.37 (d, J = 8.7 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.09, 135.42, 133.03, 132.28, 129.85, 128.93, 123.68, 120.55, 93.41, 88.38 ppm. IR (neat, cm^{-1}): 3087, 2925, 2854, 2210, 1586, 1536, 1492, 1344, 1106, 1088, 853, 836, 747, 684, 645, 506. HRMS (EI^+): calcd for $C_{14}H_8ClNO_2$ $[M]^+$ 257.0244; found 257.0247.

1-methoxy-4-(phenylethynyl)benzene (**3.12**):[14] Light yellow solid (95 mg, 61%); mp 56-57 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47-7.53 (m, 4H), 7.31-7.36 (m, 3H), 6.88 (d, J = 9.2 Hz, 2H), 3.83 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.59, 133.04, 131.44, 128.29, 127.92, 123.57, 115.36, 113.98, 89.35, 88.05, 55.29 ppm. IR (neat, cm^{-1}): 3054, 2960, 2936, 2836, 1605, 1595, 1510, 1440, 1248, 1180, 1108, 1030, 833, 753, 691, 523. HRMS (EI^+): calcd for $C_{15}H_{12}O$ $[M]^+$ 208.0888; found 208.0880.

1-methoxy-4-((4-vinylphenyl)ethynyl)benzene (**3.13**): White solid (108 mg, 61%); mp 104-106 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 (d, J = 9.2 Hz, 4H), 7.38 (d, J = 8.2 Hz, 2H), 6.88 (d, J = 9.2 Hz, 2H), 6.71 (dd, J = 17.4, 11.0 Hz, 1H), 5.77 (d, J = 17.4 Hz, 1H), 5.29 (d, J = 11.0 Hz, 1H), 3.83 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.61, 137.08, 136.28, 133.03, 131.59, 126.12, 122.87,

115.35, 114.54, 113.99, 90.05, 88.13, 55.30 ppm. IR (neat, cm^{-1}): 2967, 2933, 2838, 1605, 1598, 1511, 1440, 1403, 1247, 1028, 917, 845, 833, 511. HRMS (ESI^+): calcd for $C_{17}H_{15}O$ $[M+H]^+$ 235.1123; found 235.1120.

1-fluoro-4-((4-methoxyphenyl)ethynyl)benzene (**3.14**):[14] White solid (89 mg, 53%); mp 83-84 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 7.45-7.50 (m, 4H), 7.03 (t, J = 8.9 Hz, 2H), 6.88 (d, J = 9.2 Hz, 2H), 3.83 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.28 (d, J_{C-F} = 247.9 Hz), 159.63, 133.25 (d, J_{C-F} = 7.6 Hz), 132.98, 119.63, 115.56 (d, J_{C-F} = 21.9 Hz), 115.13, 113.99, 88.97, 86.94, 55.29 ppm. IR (neat, cm^{-1}): 2971, 2846, 1902, 1605, 1585, 1513, 1467, 1288, 1253, 1092, 1029, 838, 824, 528. HRMS (EI^+): calcd for $C_{15}H_{11}FO$ $[M]^+$ 226.0794; found 226.0792.

1-chloro-4-((4-methoxyphenyl)ethynyl)benzene (**3.15**):[14] White solid (75 mg, 41%); mp 114-115 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 7.42-7.47 (m, 4H), 7.31 (d, J = 8.7 Hz, 2H), 6.88 (d, J = 8.7 Hz, 2H), 3.83 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.75, 133.84, 133.04, 132.62, 128.62, 122.09, 114.97, 114.02, 90.33, 86.96, 55.30 ppm. IR (neat, cm^{-1}): 2967, 2842, 1908, 1606, 1514, 1396, 1288, 1250, 1108, 1098, 1029, 830, 523. HRMS (EI^+): calcd for $C_{15}H_{11}ClO$ $[M]^+$ 242.0498; found 242.0496.

1,2-bis(4-methoxyphenyl)ethyne (**3.16**):[14] White solid (111 mg, 62%); mp 113-115 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 7.45 (d, J = 9.0 Hz, 4H), 6.87 (d, J = 8.7 Hz, 4H), 3.83 (s, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.35, 132.85, 115.68, 113.93, 87.91, 55.28 ppm. IR (neat, cm^{-1}): 2963, 2838, 1606, 1518, 1456, 1285, 1247, 1171, 1105, 1025, 834, 822, 533. HRMS (EI^+): calcd for $C_{16}H_{14}O_2$ $[M]^+$ 238.0994; found 238.0996.

2-(phenylethynyl)naphthalene (**3.17**):[14] White solid (108 mg, 63%); mp 105-106 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 8.07 (s, 1H), 7.81-7.84 (m, 3H), 7.57-7.61 (m, 3H), 7.48-7.53 (m, 2H), 7.35-7.41 (m, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 133.0, 132.78, 131.65, 131.42, 128.41, 128.37, 128.30, 127.99, 127.77, 126.65, 126.53, 123.25, 120.55, 89.77, 89.71 ppm. IR (neat, cm^{-1}): 3052, 1598, 1488, 1441, 1268, 965, 865, 822, 754, 744, 688, 481, 469. HRMS (EI^+): calcd for $C_{18}H_{12}$ $[M]^+$ 228.0939; found 228.0936.

2-((4-methoxyphenyl)ethynyl)naphthalene (**3.18**):[14] Pale yellow solid (134 mg, 69%); mp 118-120 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 8.04 (s, 1H), 7.80-7.83 (m, 3H), 7.48-7.59 (m, 5H), 6.91 (d, J = 8.8 Hz, 2H), 3.84 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.64, 133.09, 133.04, 132.62, 131.07, 128.41, 127.92, 127.73, 127.70, 126.46, 120.90, 115.36, 114.02, 89.75, 88.49, 55.29 ppm. IR (neat, cm^{-1}): 3052, 2965, 2934, 2841, 2213, 1604, 1592, 1511, 1285, 1270, 1246, 1029, 866, 834, 821, 741, 540, 528, 479. HRMS (EI^+): calcd for $C_{19}H_{14}O$ $[M]^+$ 258.1045; found 258.1041.

2-((4-fluorophenyl)ethynyl)naphthalene (**3.19**): White solid (121 mg, 66%); mp 106-108 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05 (s, 1H), 7.81-7.84 (m, 3H), 7.54-7.58 (m, 3H), 7.49-7.52 (m, 2H), 7.07 (t, J = 8.9 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.51 (d, $J_{C\text{-}F}$ = 250 Hz), 133.51 (d, $J_{C\text{-}F}$ = 8.6 Hz), 132.98, 132.80, 131.39, 128.29, 128.03, 127.76, 126.70, 126.58, 120.35, 119.37, 115.68 (d, $J_{C\text{-}F}$ = 22.0 Hz), 89.44, 88.61 ppm. IR (neat, cm^{-1}): 3052, 1589, 1505, 1218, 1138, 901, 836, 819, 740, 525, 479. HRMS (EI^{+}): calcd for $C_{18}H_{11}F$ $[M]^{+}$ 246.0845; found 246.0849.

2-((4-chlorophenyl)ethynyl)naphthalene (**3.20**):[11] White solid (112 mg, 57%); mp 122-123 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06 (s, 1H), 7.81-7.85 (m, 3H), 7.56-7.58 (m, 1H), 7.49-7.52 (m, 4H), 7.35 (d, J = 8.7 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 134.27, 132.95, 132.82, 131.52, 128.72, 128.25, 128.06, 127.78, 126.78, 126.61, 121.76, 120.17, 90.71, 88.56 ppm. IR (neat, cm^{-1}): 3054, 1909, 1587, 1487, 1396, 1270, 1085, 900, 867, 820, 740, 520, 479. HRMS (EI^{+}): calcd for $C_{18}H_{11}Cl$ $[M]^{+}$ 262.0549; found 262.0540.

1-(benzyloxy)-2-methoxy-4-(phenylethynyl)benzene (**3.21**): White solid (139 mg, 59%); mp 78-80 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50-7.53 (m, 2H), 7.44 (d, J = 7.3 Hz, 2H), 7.36–7.39 (m, 2H), 7.29-7.34 (m, 4H), 7.05–7.08 (m, 2H), 6.85 (d, J = 8.7 Hz, 1H), 5.18 (s, 2H), 3.92 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.23, 148.56, 136.70, 131.45, 128.59, 128.31, 128.02, 127.95, 127.24, 124.75, 123.40, 115.88, 114.70, 113.57, 89.42, 88.02, 70.87, 55.99 ppm. IR (neat, cm^{-1}): 3034, 2936, 2865, 2208, 1593, 1575, 1512, 1410, 1247, 1222, 1124, 1031, 1002, 848, 806, 756, 743, 692. HRMS (EI^{+}): calcd for $C_{22}H_{18}O_2$ $[M]^{+}$ 314.1307; found 314.1307.

1-(benzyloxy)-4-((4-fluorophenyl)ethynyl)-2-methoxybenzene (**3.22**): White Solid (136 mg, 55%); mp 102-104 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47-7.50 (m, 2H), 7.43-7.44 (m, 2H), 7.35-7.39 (m, 2H), 7.31-7.33 (m, 1H), 7.01-7.06 (m, 4H), 6.85 (d, J = 8.7 Hz, 1H), 5.18 (s, 2H), 3.91 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.34 (d, $J_{C\text{-}F}$ = 250 Hz), 149.25, 148.62, 136.67, 133.30 (d, $J_{C\text{-}F}$ = 8.6 Hz), 128.59, 127.96, 127.24, 124.70, 119.48, 115.59 (d, $J_{C\text{-}F}$ = 22.0 Hz), 114.63, 113.56, 89.06, 86.93, 70.87, 55.99 ppm. IR (neat, cm^{-1}): 3032, 2935, 1602, 1576, 1518, 1455, 1251, 1226, 1121, 992, 842, 815, 806, 743, 695. HRMS (EI^{+}): calcd for $C_{22}H_{17}FO_2$ $[M]^{+}$ 332.1213; found 332.1212.

1-(benzyloxy)-4-((4-chlorophenyl)ethynyl)-2-methoxybenzene (**3.23**): White Solid (99 mg, 38%); mp 112-114 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30-7.44 (m, 9H), 7.04-7.06 (m, 2H), 6.85 (d, J = 9.2 Hz, 1H), 5.18 (s, 2H), 3.91 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.25, 148.75, 136.64, 133.96, 132.64, 128.65, 128.60, 127.98, 127.24, 124.79, 121.93, 115.49, 114.64, 113.54, 90.40, 86.93, 70.86, 56.0 ppm. IR (neat, cm^{-1}): 3032, 2935, 2874, 1597, 1576, 1455, 1410, 1224, 1123, 1034, 990, 842, 828, 816, 806, 743, 693. HRMS (EI^{+}): calcd for $C_{22}H_{17}ClO_2$ $[M]^{+}$ 348.0917; found 348.0910.

1-bromo-4-(phenylethynyl)benzene (**3.24**):[13] White solid (133 mg, 69%); mp 82-83 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47-7.54 (m, 4H), 7.34-7.40 (m, 5H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 133.01, 131.60, 131.58, 128.50, 128.39, 122.87, 122.45, 122.22, 90.47, 88.28 ppm. IR (neat, cm^{-1}): 3050, 1908, 1599, 1504, 1492, 1478, 1441, 1265, 1068, 1008, 828, 821, 751, 739, 688, 510. HRMS (EI^+): calcd for $C_{14}H_9Br$ $[M]^+$ 255.9888; found 255.9888.

1-chloro-4-(phenylethynyl)benzene (**3.25**):[7] White solid (107 mg, 67%); mp 78-79 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51-7.54 (m, 2H), 7.45-7.47 (m, 2H), 7.32-7.36 (m, 5H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 134.23, 132.80, 131.58, 128.68, 128.47, 128.38, 122.89, 121.75, 90.28, 88.21 ppm. IR (neat, cm^{-1}): 3049, 1911, 1590, 1496, 1481, 1440, 1399, 1265, 1091, 831, 751, 732, 687, 512. HRMS (EI^+): calcd for $C_{14}H_9Cl$ $[M]^+$ 212.0393; found 212.0394.

2-(phenylethynyl)furan (**3.26**):[12] Colorless oil (77 mg, 61%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51-7.54 (m, 2H), 7.43 (dd, J = 1.8, 0.9 Hz, 1H), 7.34-7.36 (m, 3H), 6.66-6.67 (m, 1H), 6.43 (dd, J = 3.2, 1.8 Hz, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.63, 137.10, 131.39, 128.68, 128.37, 122.23, 115.22, 111.06, 93.21, 79.34 ppm. IR (neat, cm^{-1}): 3061, 2201, 1771, 1722, 1670, 1597, 1490, 1444, 1179, 1095, 1015, 756, 689. HRMS (ESI^+): calcd for $C_{12}H_8O$ $[M]^+$ 168.0575; found 168.0573.

2-((4-methoxyphenyl)ethynyl)furan (**3.27**):[14] Pale brown liquid (92 mg, 62%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41-7.48 (m, 3H), 6.86-6.89 (m, 2H), 6.62 (d, J = 3.2 Hz, 1H), 6.41-6.43 (m, 1H), 3.83 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.95, 143.34, 137.40, 133.03, 114.64, 114.29, 114.04, 110.99, 93.18, 78.06, 55.30 ppm. IR (neat, cm^{-1}): 2933, 2839, 2193, 1606, 1573, 1510, 1481, 1295, 1250, 1175, 1163, 1030, 832, 742. HRMS (EI^+): calcd for $C_{13}H_{10}O_2$$[M]^+$ 198.0681; found 198.0689.

2-((4-methoxyphenyl)ethynyl)thiophene (**3.28**):[14] Pale yellow solid (100 mg, 62%); mp 52-53 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44 (d, J = 9.2 Hz, 2H), 7.23-7.26 (m, 2H), 6.98-7.00 (m, 1H), 6.87 (d, J = 8.7 Hz, 2H), 3.82 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.73, 132.91, 131.41, 127.01, 126.79, 123.68, 114.97, 114.01, 92.98, 81.22, 55.29 ppm. IR (neat, cm^{-1}): 3106, 3003, 2960, 2836, 2541, 2205, 1605, 1568, 1525, 1503, 1463, 1441, 1426, 1292, 1248, 1215, 1173, 1109, 1029, 853, 830, 700, 544. HRMS (EI^+): calcd for $C_{13}H_{10}OS$ $[M]^+$ 214.0452; found 214.0450.

2-((4-ethoxyphenyl)ethynyl)thiophene (**3.29**):[14] Pale yellow solid (72 mg, 42%); mp 77-78 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 (d, J = 8.7 Hz, 2H) , 7.23-7.25 (m, 2H), 6.97-7.00 (m, 1H), 6.85 (d, J = 8.7 Hz, 2H), 4.01-4.06 (m, 2H), 1.41 (t, J = 7.1 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.13, 132.90, 131.37, 127.0, 126.75, 123.72, 114.76, 114.50, 93.07, 81.13, 63.50, 14.74 ppm. IR (neat,

cm^{-1}): 3116, 2973, 2928, 1601, 1521, 1496, 1290, 1242, 1213, 1181, 1117, 1105, 950, 850, 837, 709, 698. HRMS (EI^+): calcd for $C_{14}H_{12}OS$ $[M]^+$ 228.0609; found 228.0606.

2-((4-isopropoxyphenyl)ethynyl)thiophene (**3.30**):[14] Pale yellow solid (79 mg, 44%); mp 57-58 ^oC. ^1H NMR (400 MHz, $CDCl_3$): δ 7.42 (d, J = 9.2 Hz, 2H), 7.22-7.25 (m, 2H), 6.97-6.99 (m, 1H), 6.84 (d, J = 9.2 Hz, 2H), 4.55-4.58 (m, 1H), 1.33 (d, J = 6.0 Hz, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.15, 132.93, 131.34, 126.99, 126.71, 123.78, 115.70, 114.59, 93.12, 81.07, 69.93, 21.96 ppm. IR (neat, cm^{-1}): 3116, 2979, 2924, 2878, 1602, 1524, 1501, 1474, 1286, 1246, 1210, 1175, 1112, 1041, 919, 852, 811, 704, 558, 537. HRMS (EI^+): calcd for $C_{15}H_{14}OS$ $[M]^+$ 242.0765; found 242.0766.

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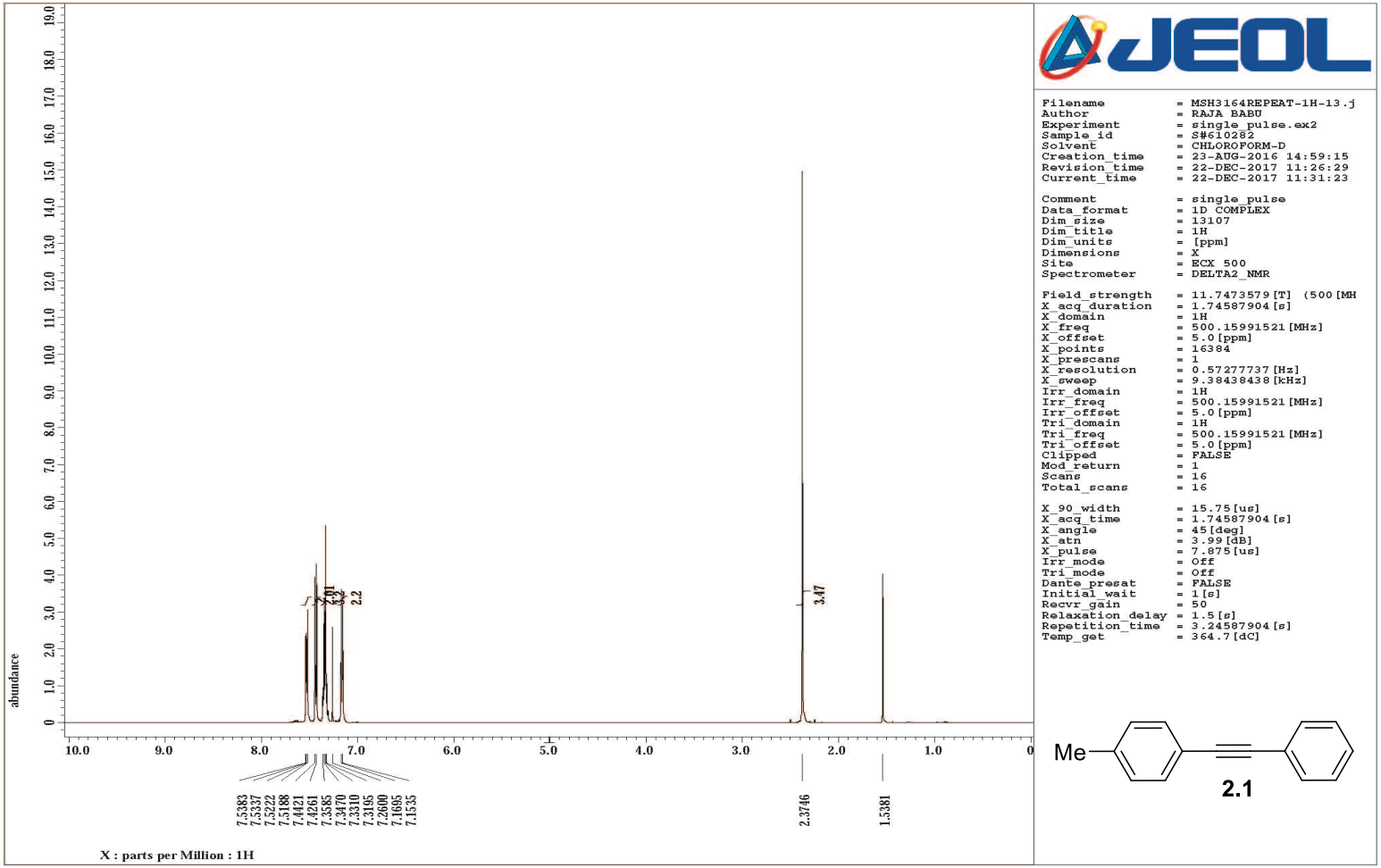
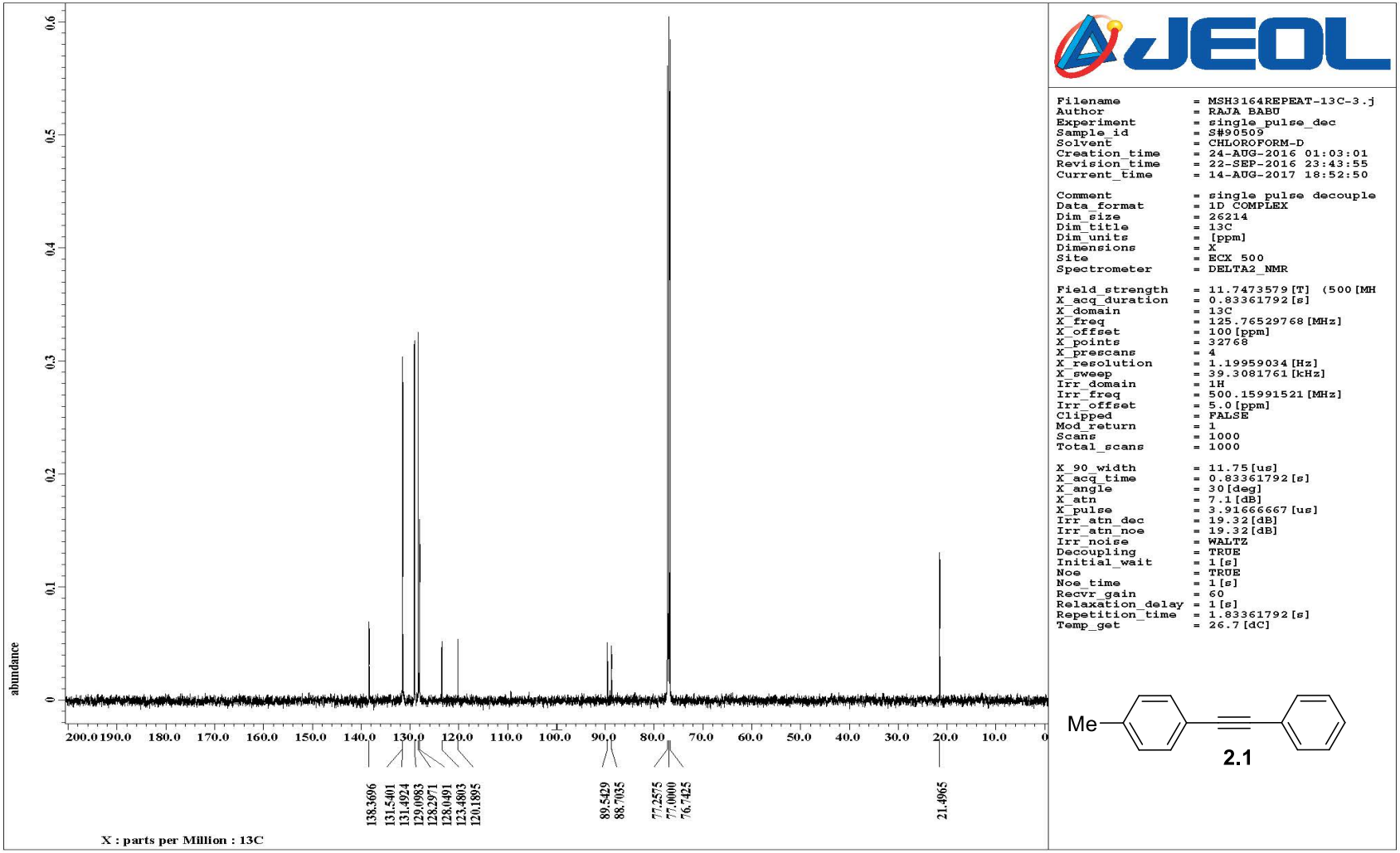
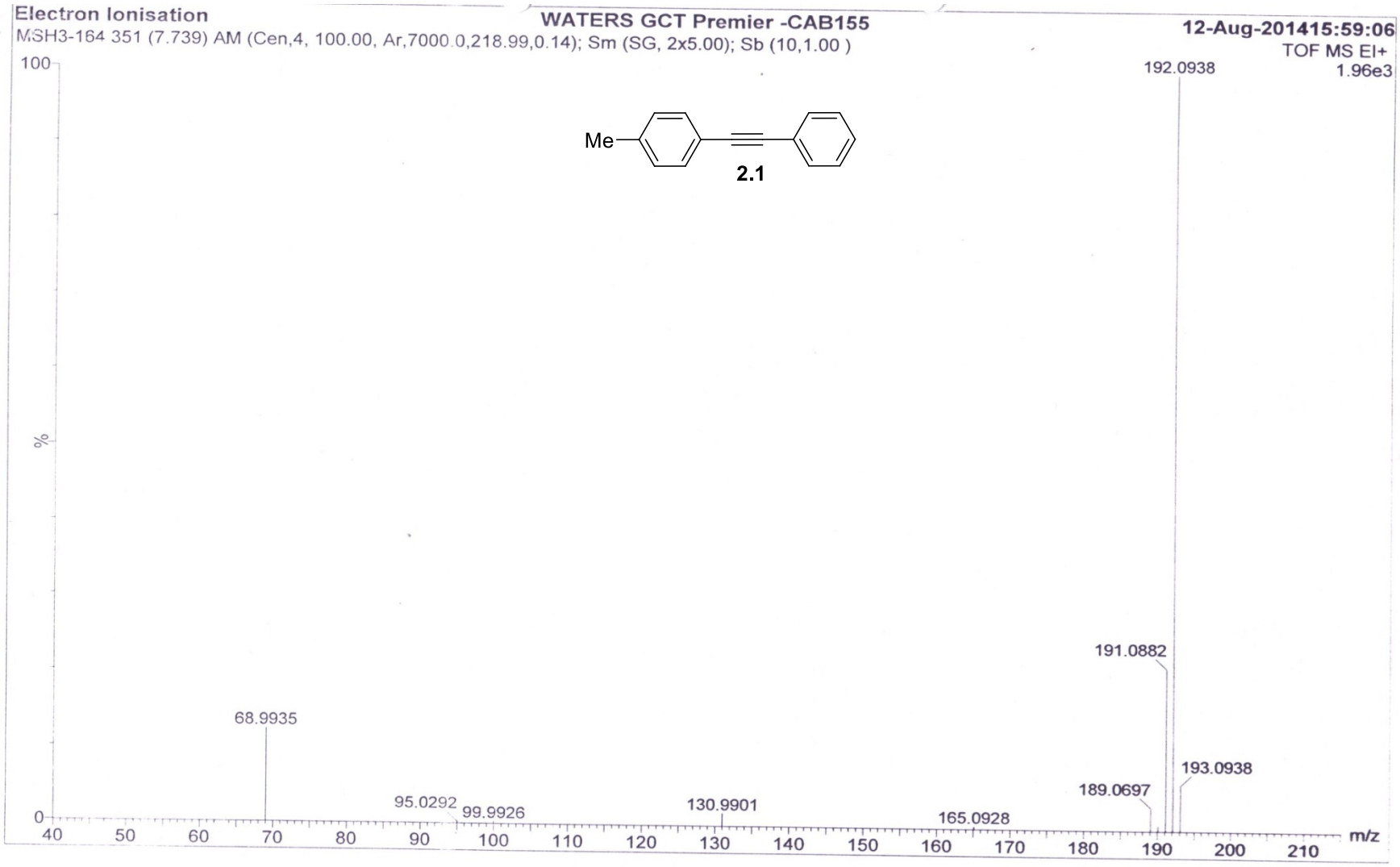
7. Copies of ^{1}H NMR, ^{13}C NMR and HRMS spectra of 2.1 -2.10 (Table 2):



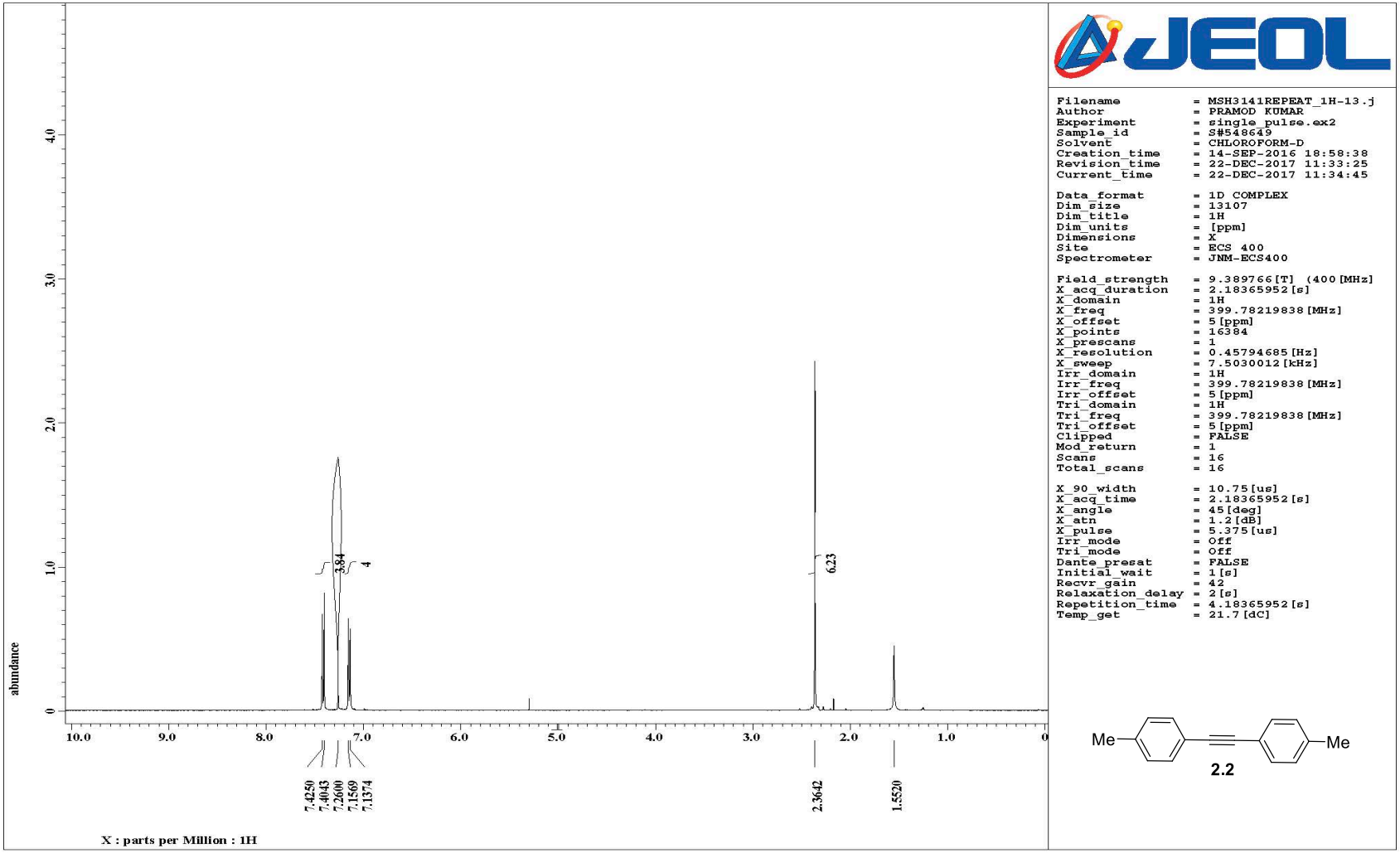
^{1}H NMR spectrum of 1-methyl-4-(phenylethynyl)benzene **(2.1)**



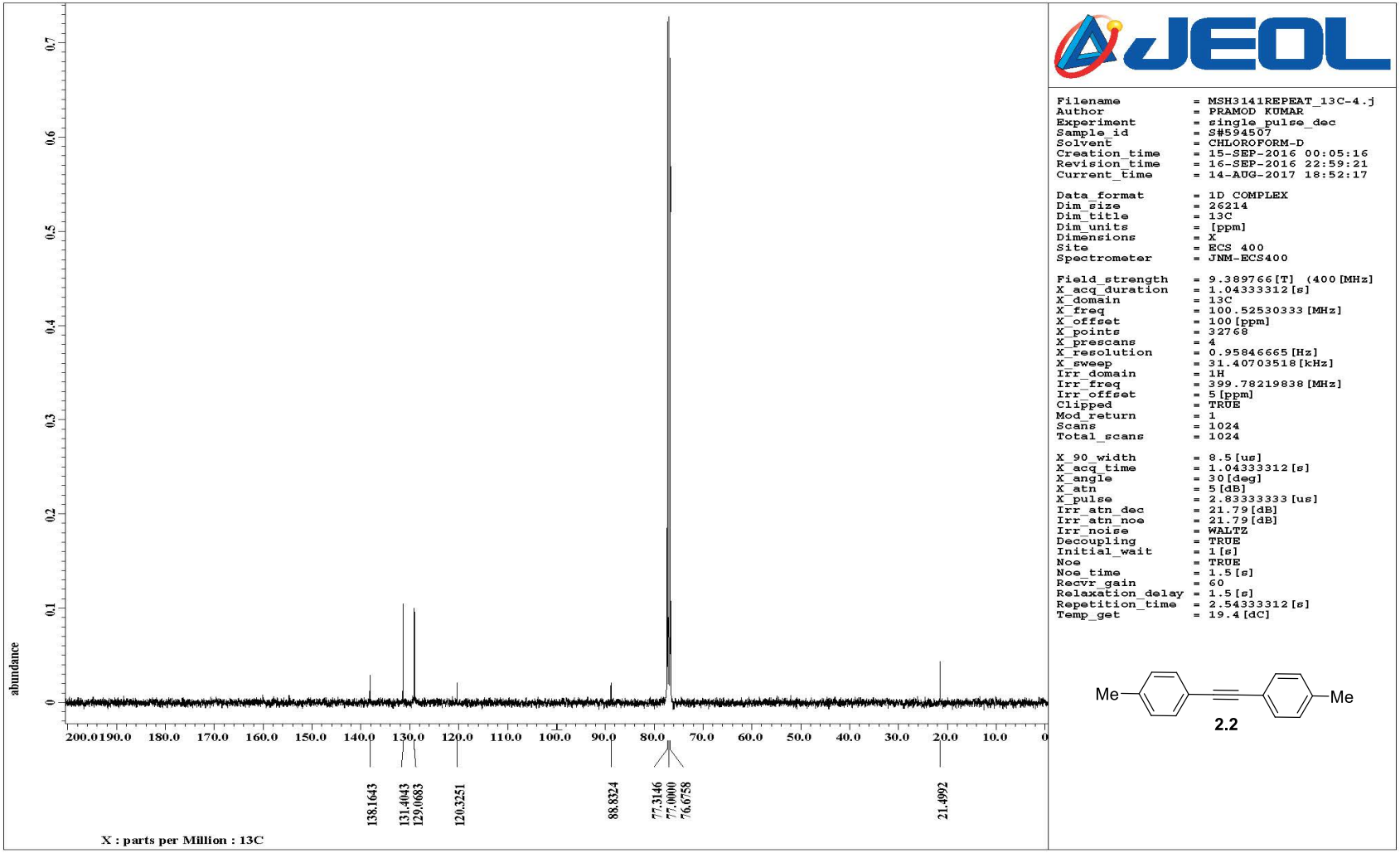
^{13}C NMR spectrum of 1-methyl-4-(phenylethynyl)benzene **(2.1)**



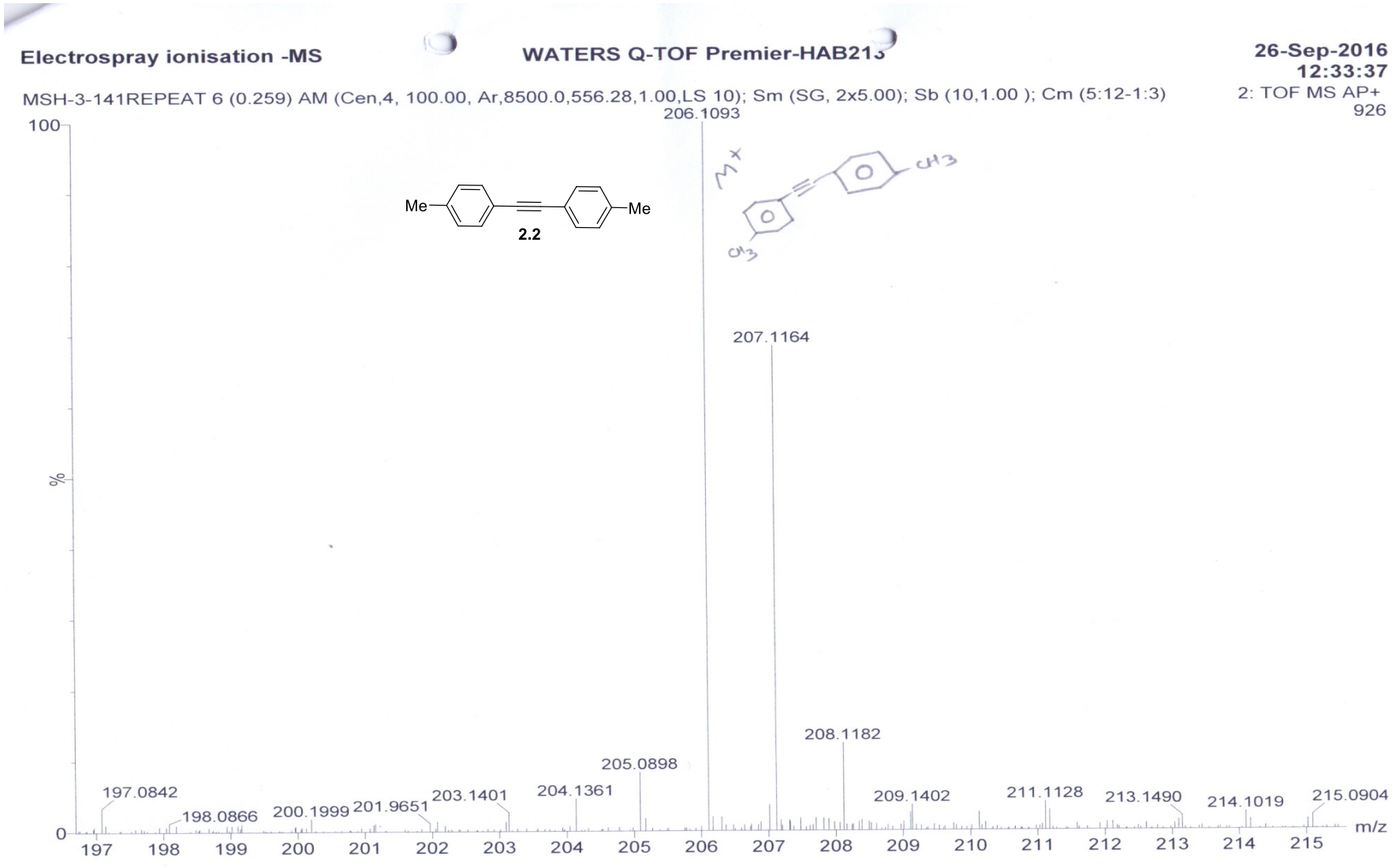
HRMS spectrum of 1-methyl-4-(phenylethynyl)benzene **(2.1)**



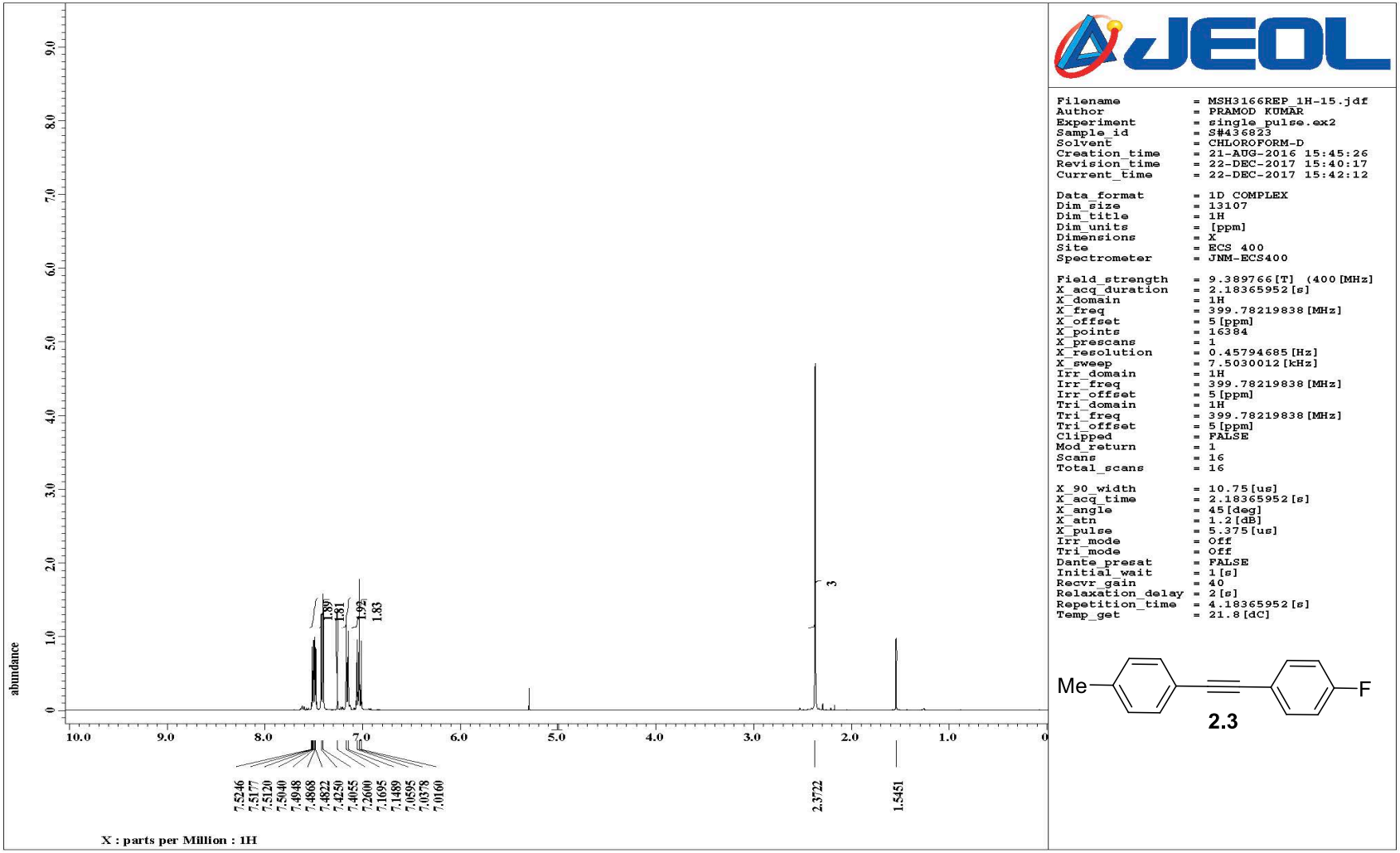
^{1}H NMR spectrum of 1,2-di*p*-tolylethyne **(2.2)**



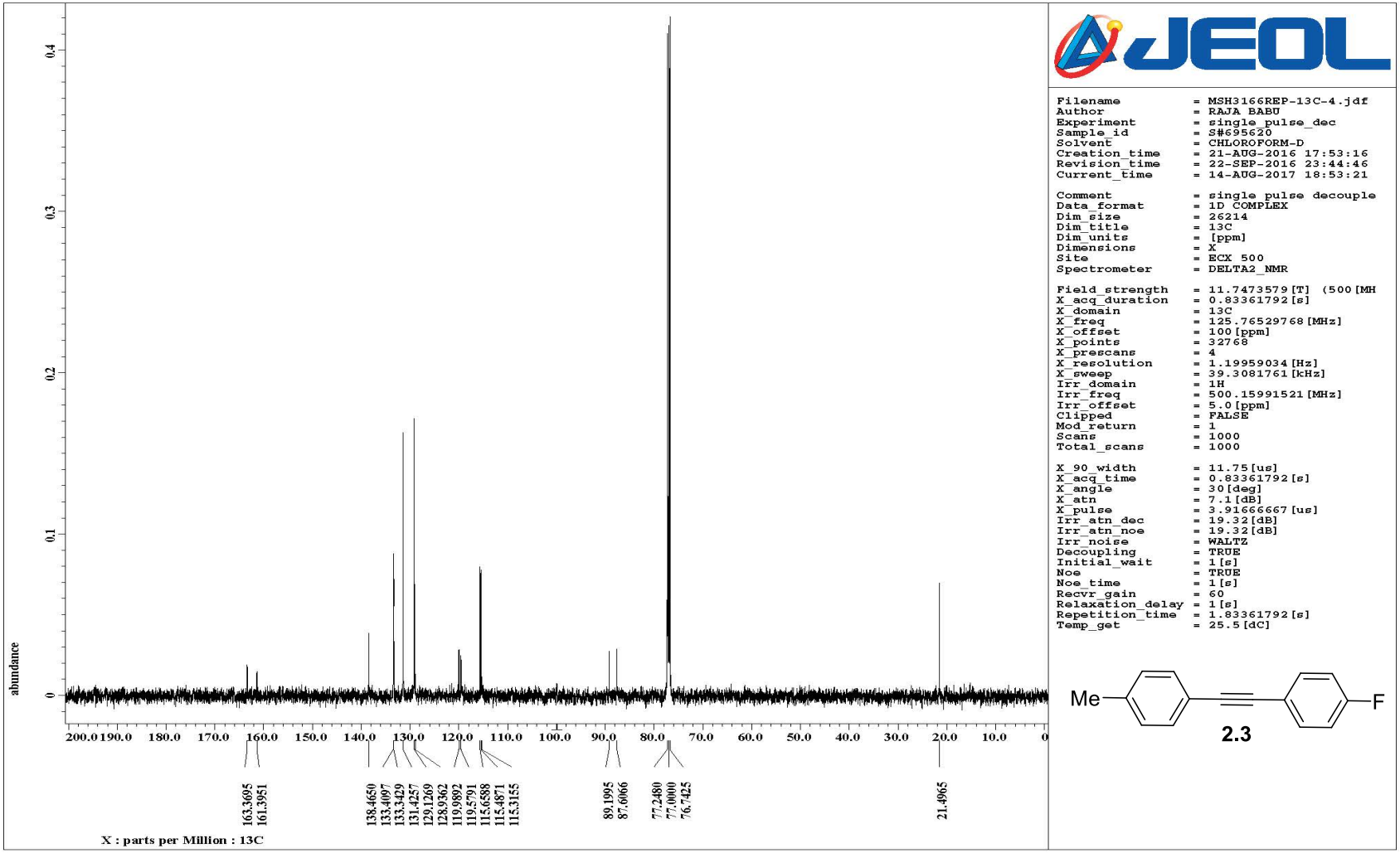
^{13}C NMR spectrum of 1,2-di*p*-tolylethyne (**2.2**)



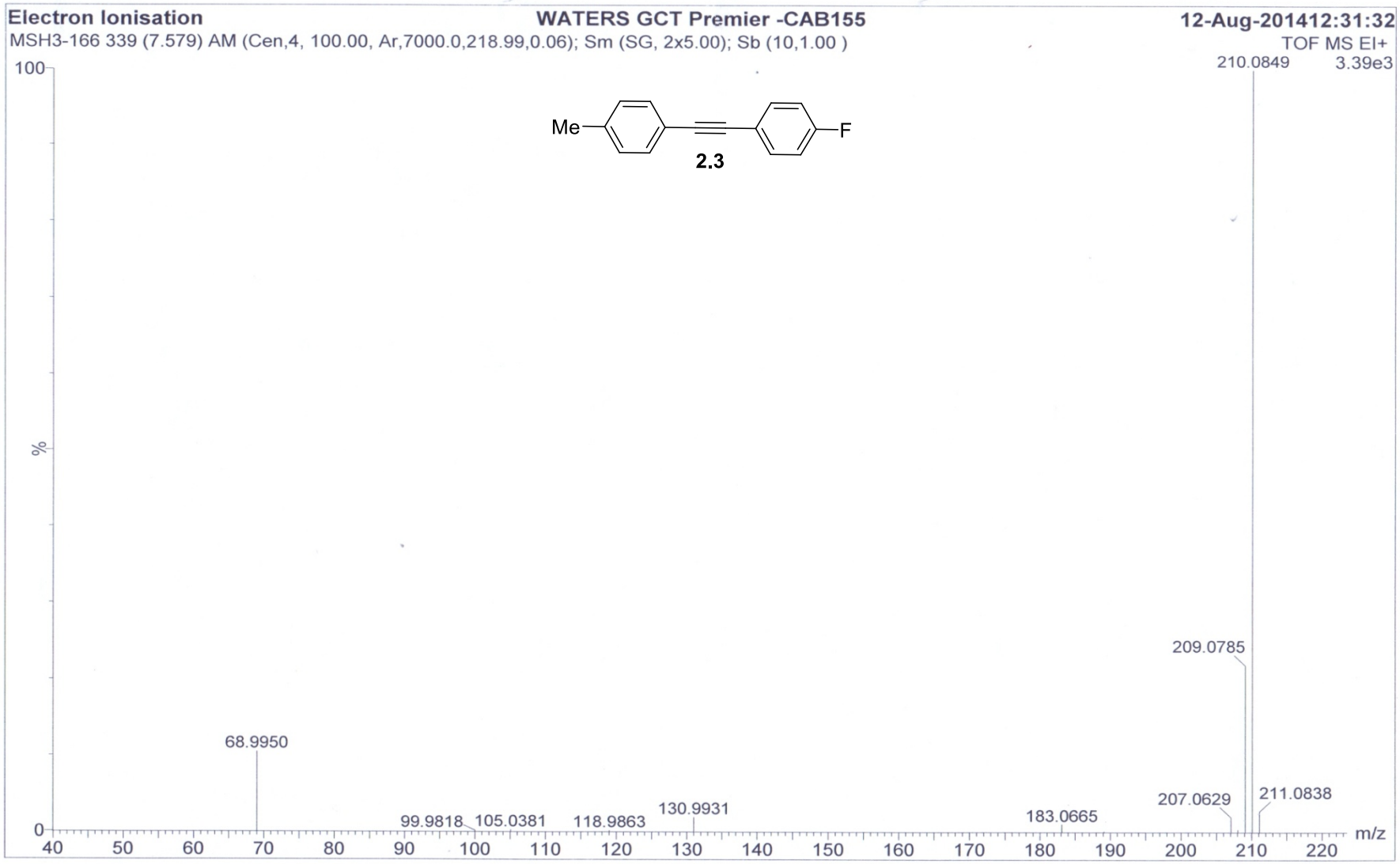
HRMS spectrum of 1,2-di*p*-tolylethyne (**2.2)**



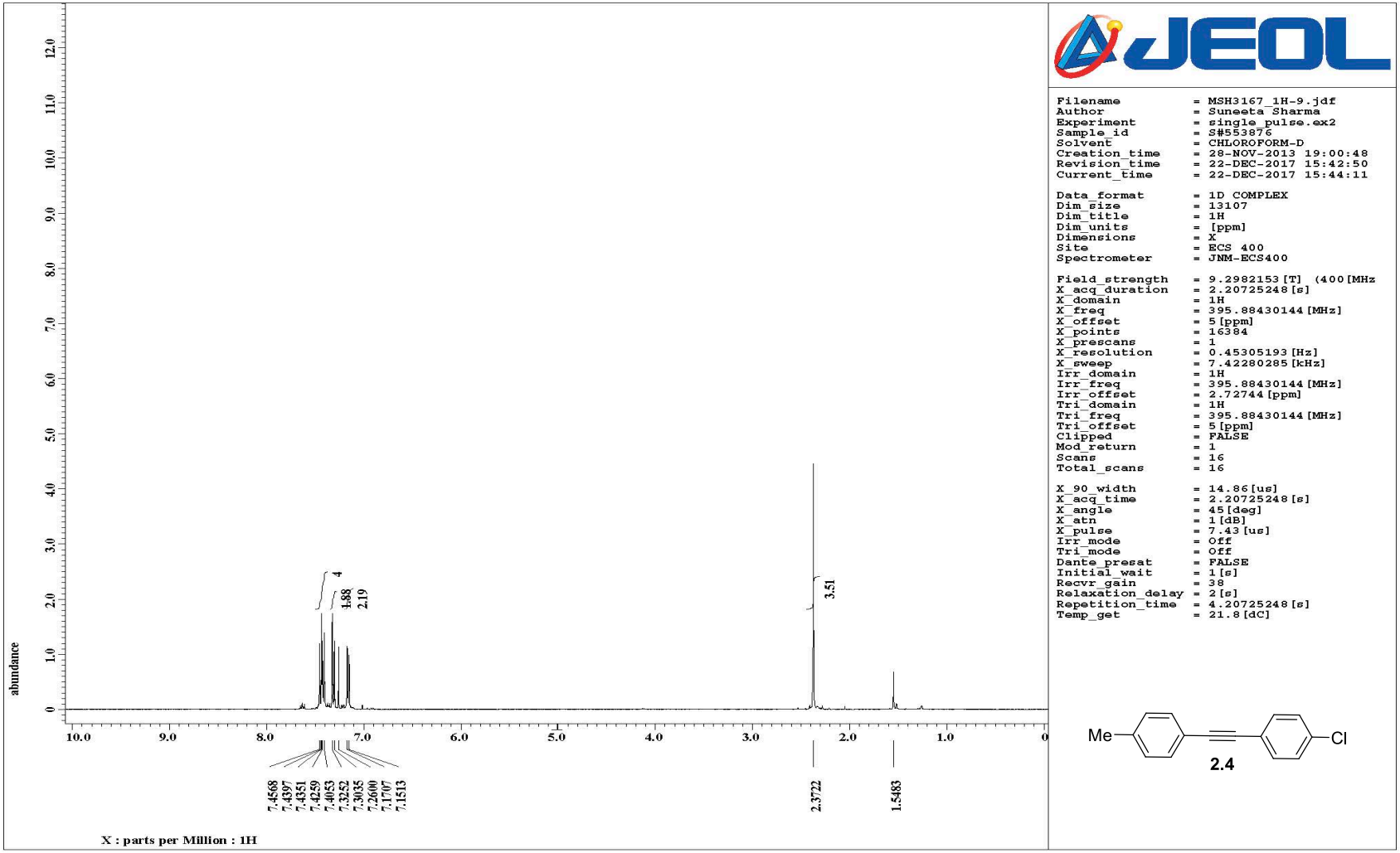
^{1}H NMR spectrum of 1-fluoro-4-(*p*-tolylethynyl)benzene **(2.3)**



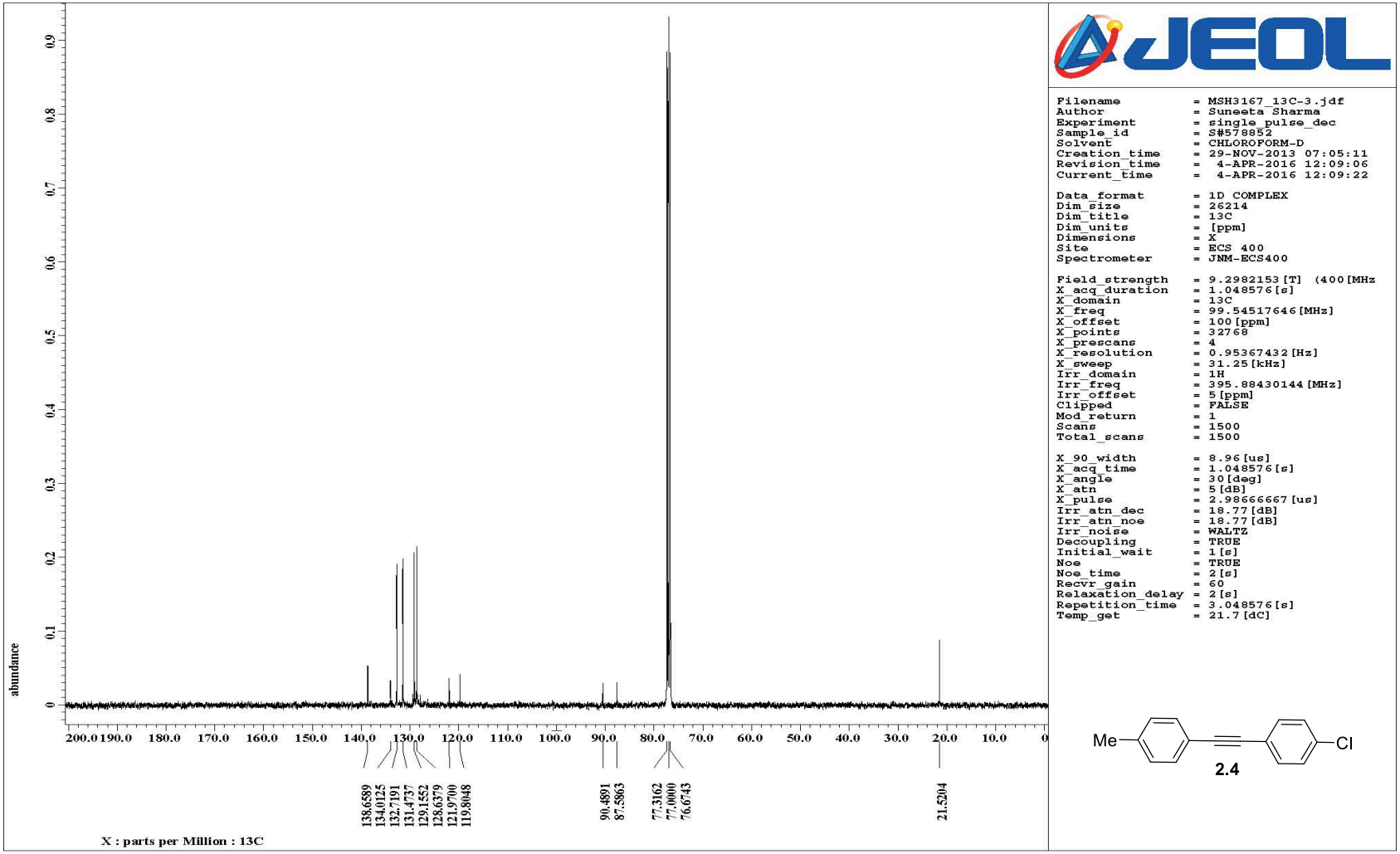
^{13}C NMR spectrum of 1-fluoro-4-(*p*-tolylethynyl)benzene **(2.3)**



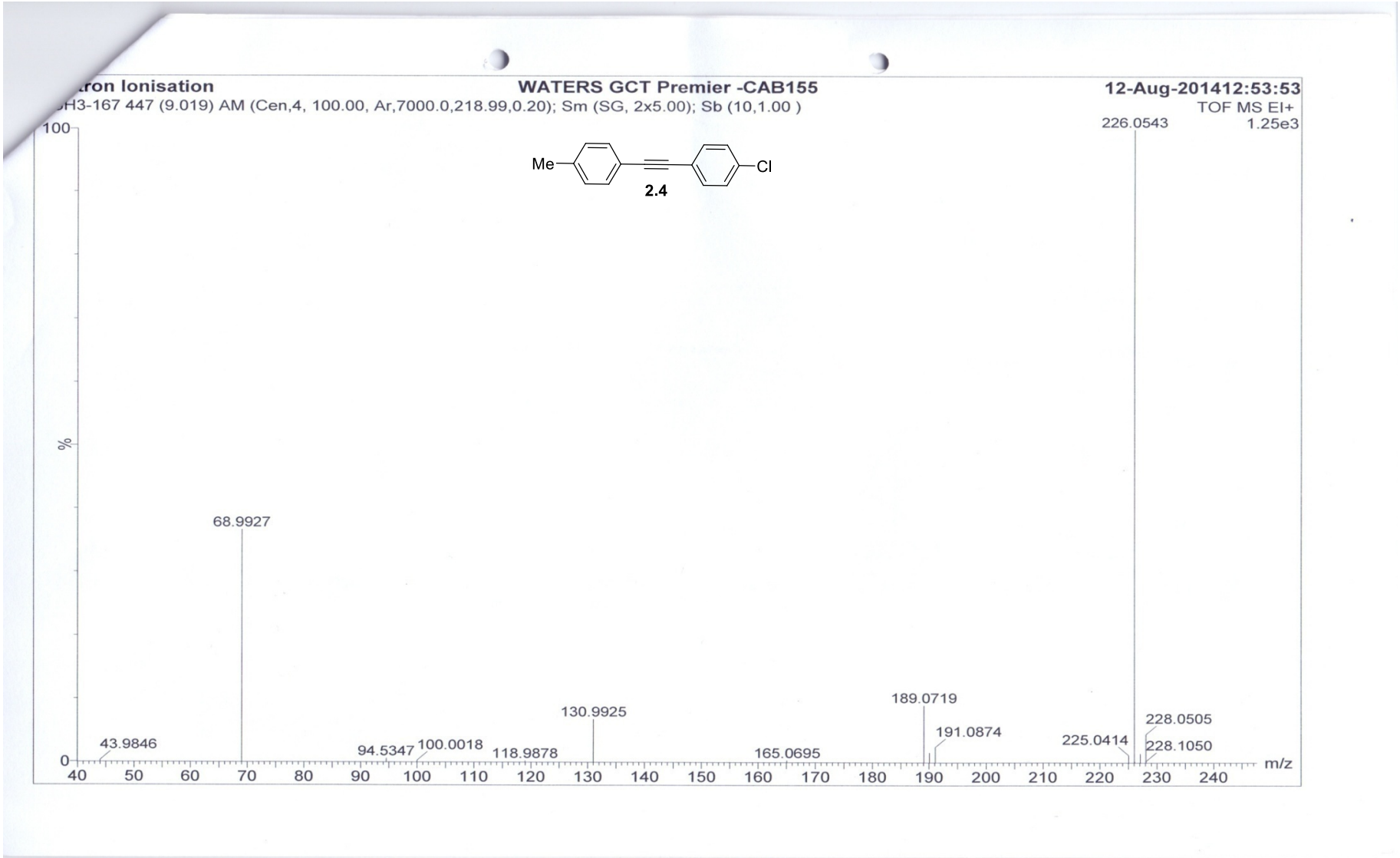
HRMS spectrum of 1-fluoro-4-(*p*-tolylethynyl)benzene (**2.3)**



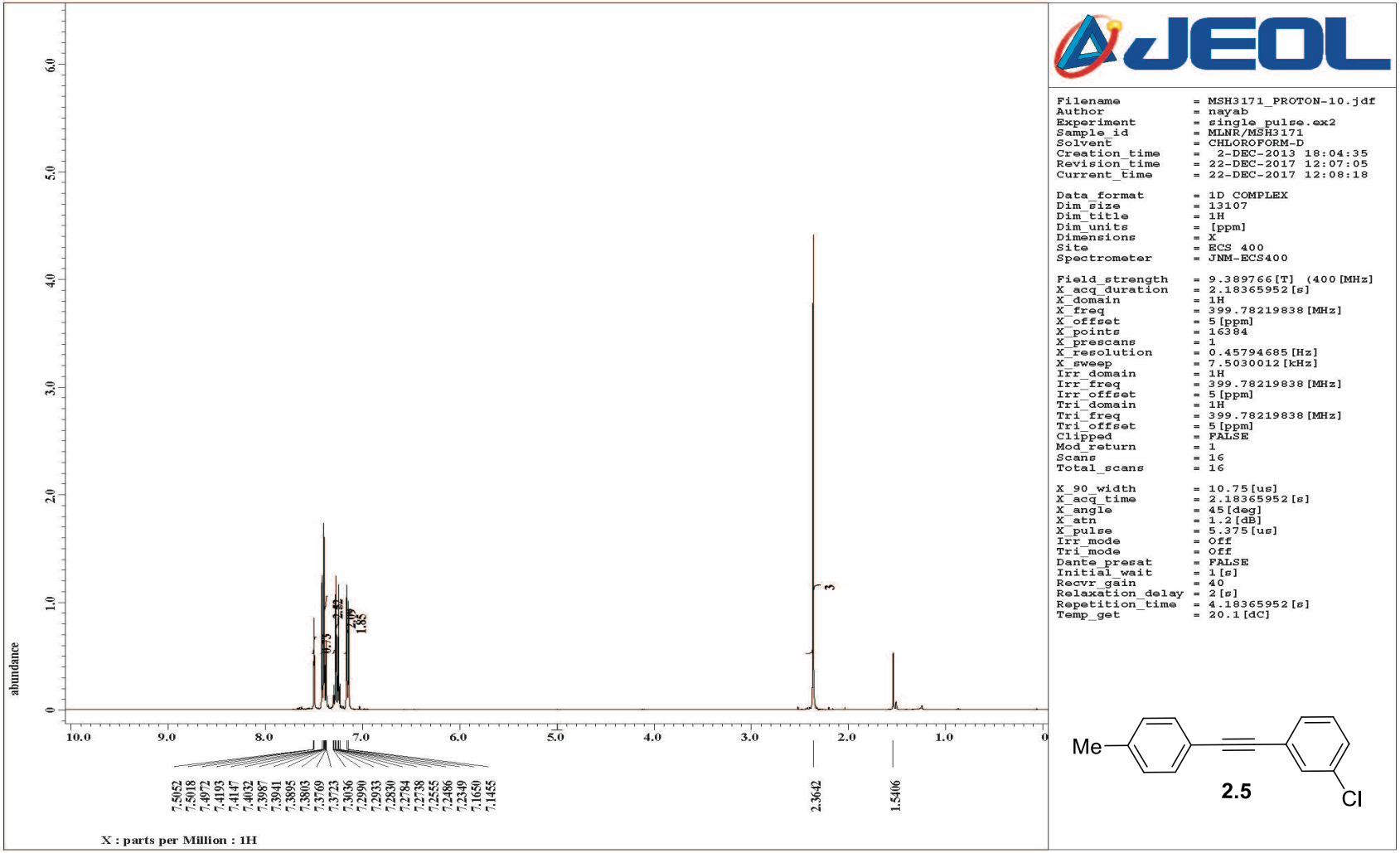
^{1}H NMR spectrum of 1-chloro-4-(*p*-tolylethynyl)benzene (**2.4**)



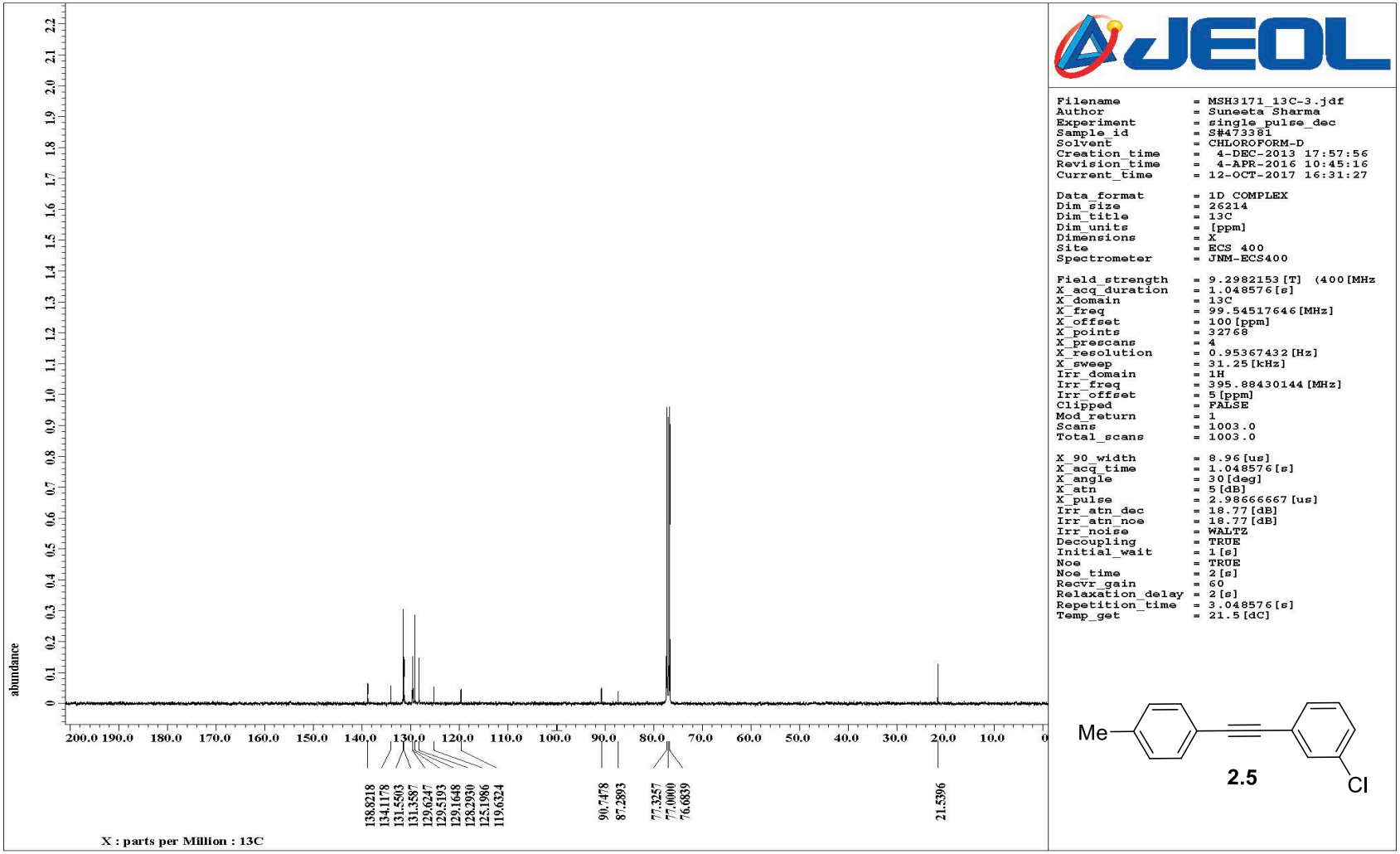
^{13}C NMR spectrum of 1-chloro-4-(*p*-tolylethynyl)benzene (**2.4**)



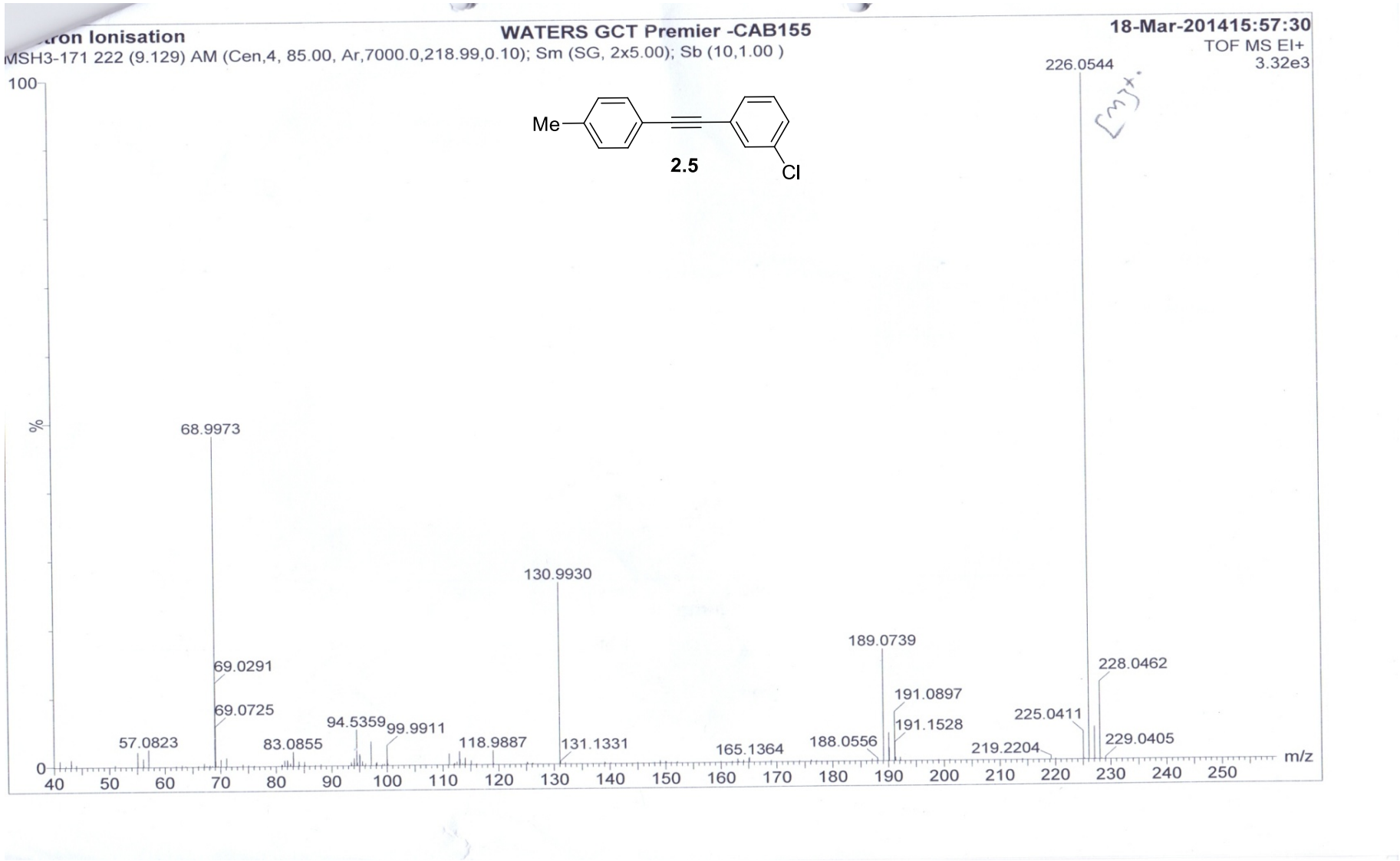
HRMS spectrum of 1-chloro-4-(*p*-tolylethynyl)benzene (**2.4)**



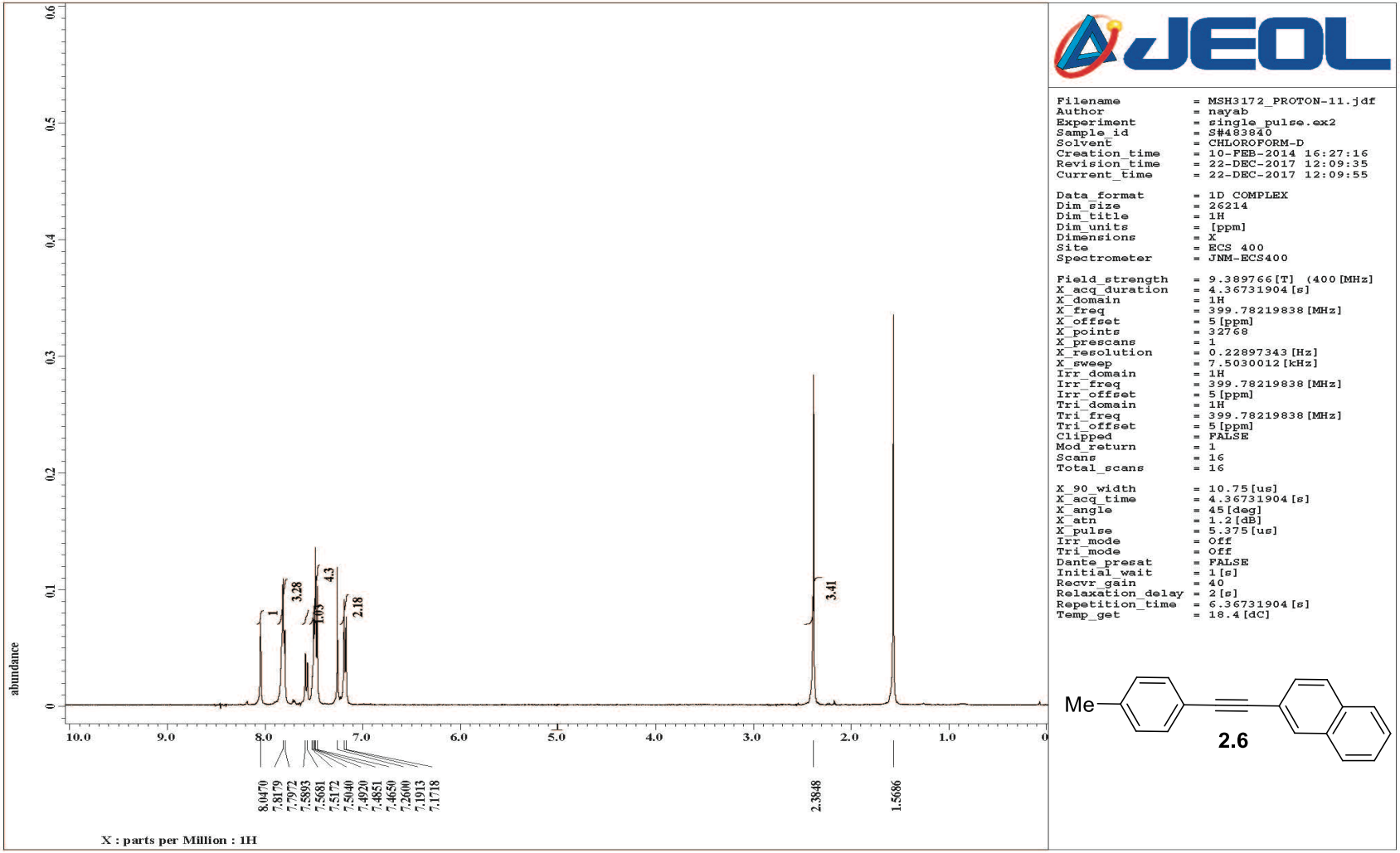
^{1}H NMR spectrum of 1-chloro-3-(*p*-tolylethynyl)benzene **(2.5)**



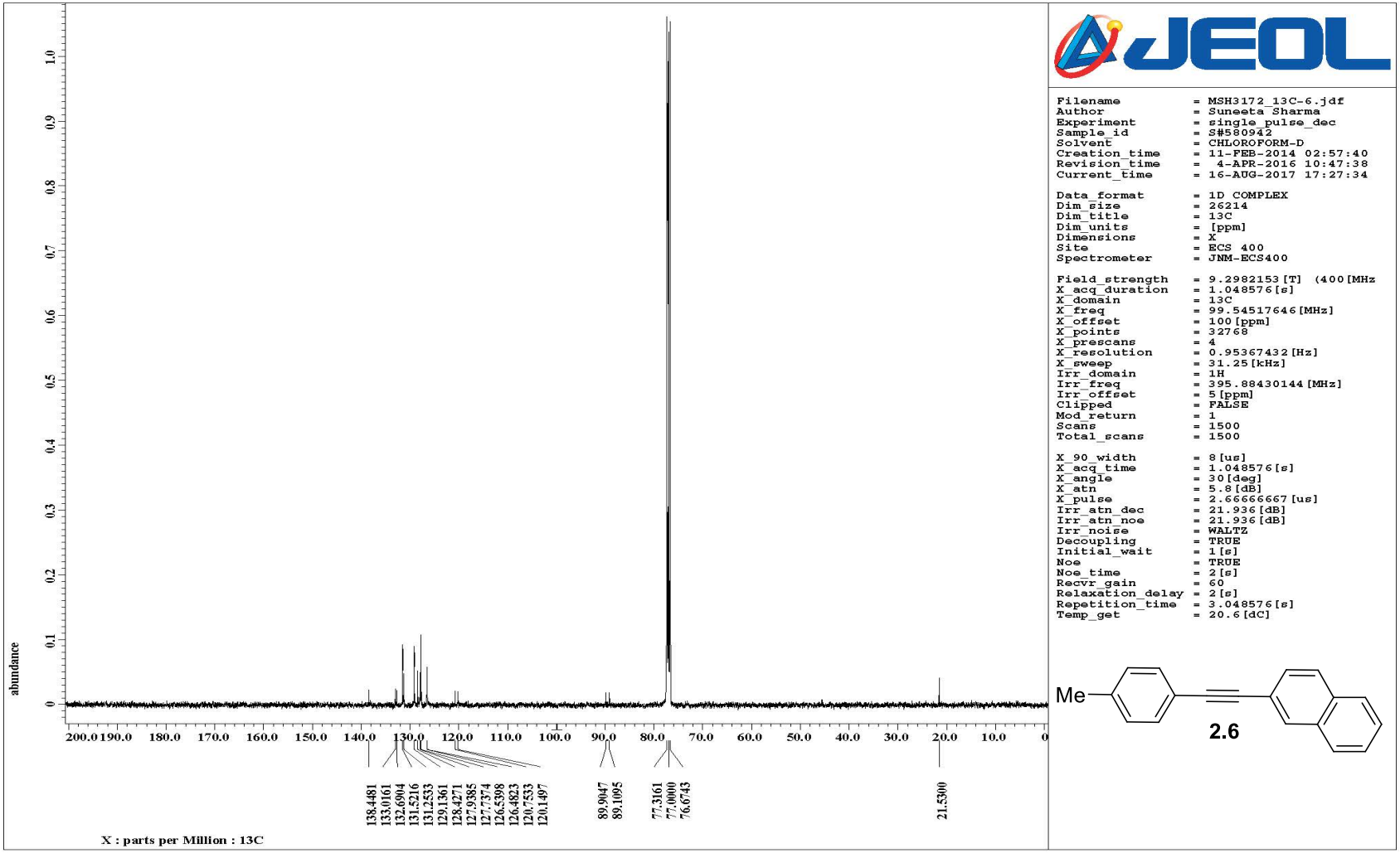
^{13}C NMR spectrum of 1-chloro-3-(*p*-tolylethynyl)benzene **(2.5)**



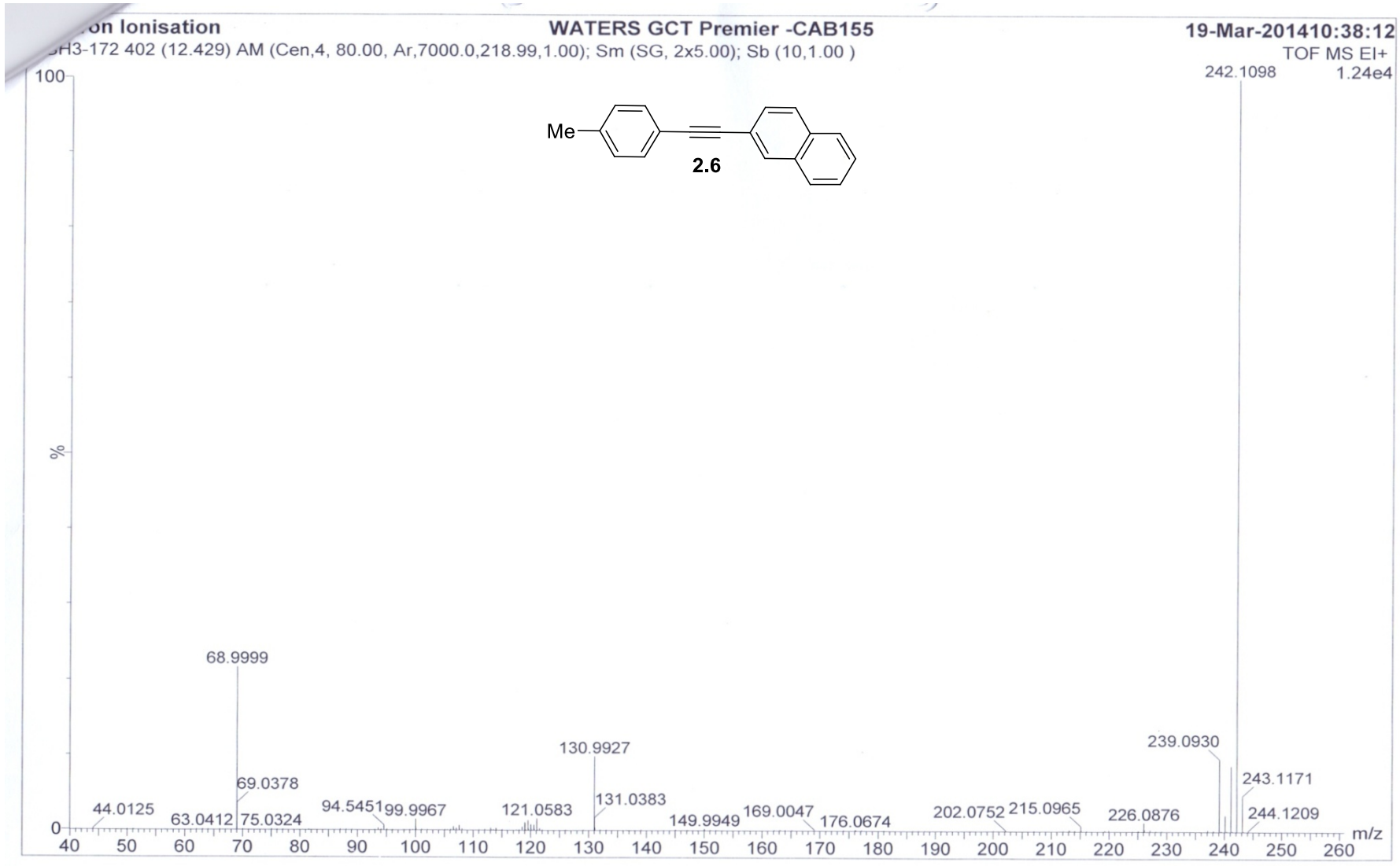
HRMS spectrum of 1-chloro-3-(*p*-tolylethynyl)benzene **(2.5)**



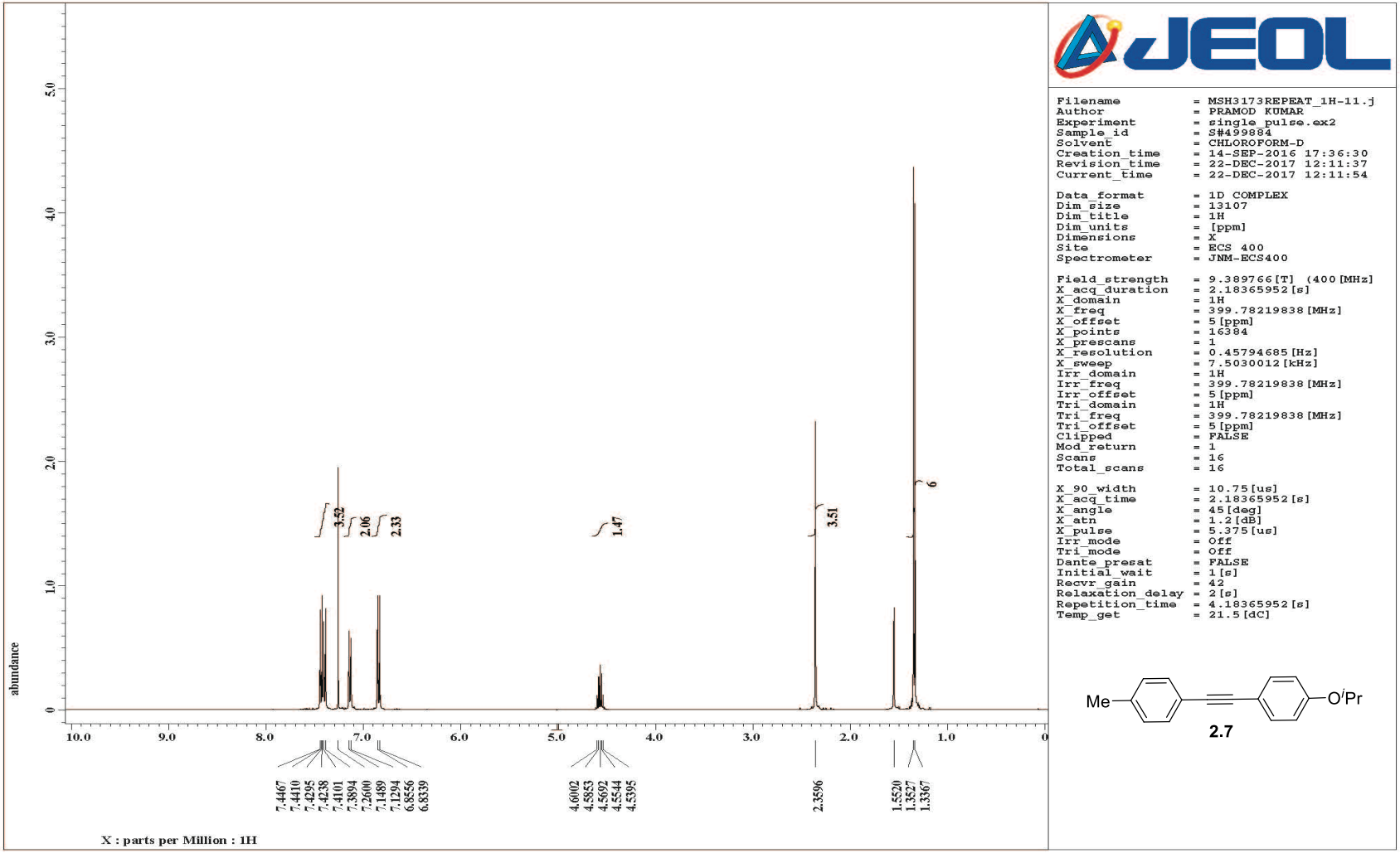
^{1}H NMR spectrum of 2-(*p*-tolylethynyl)naphthalene **(2.6)**



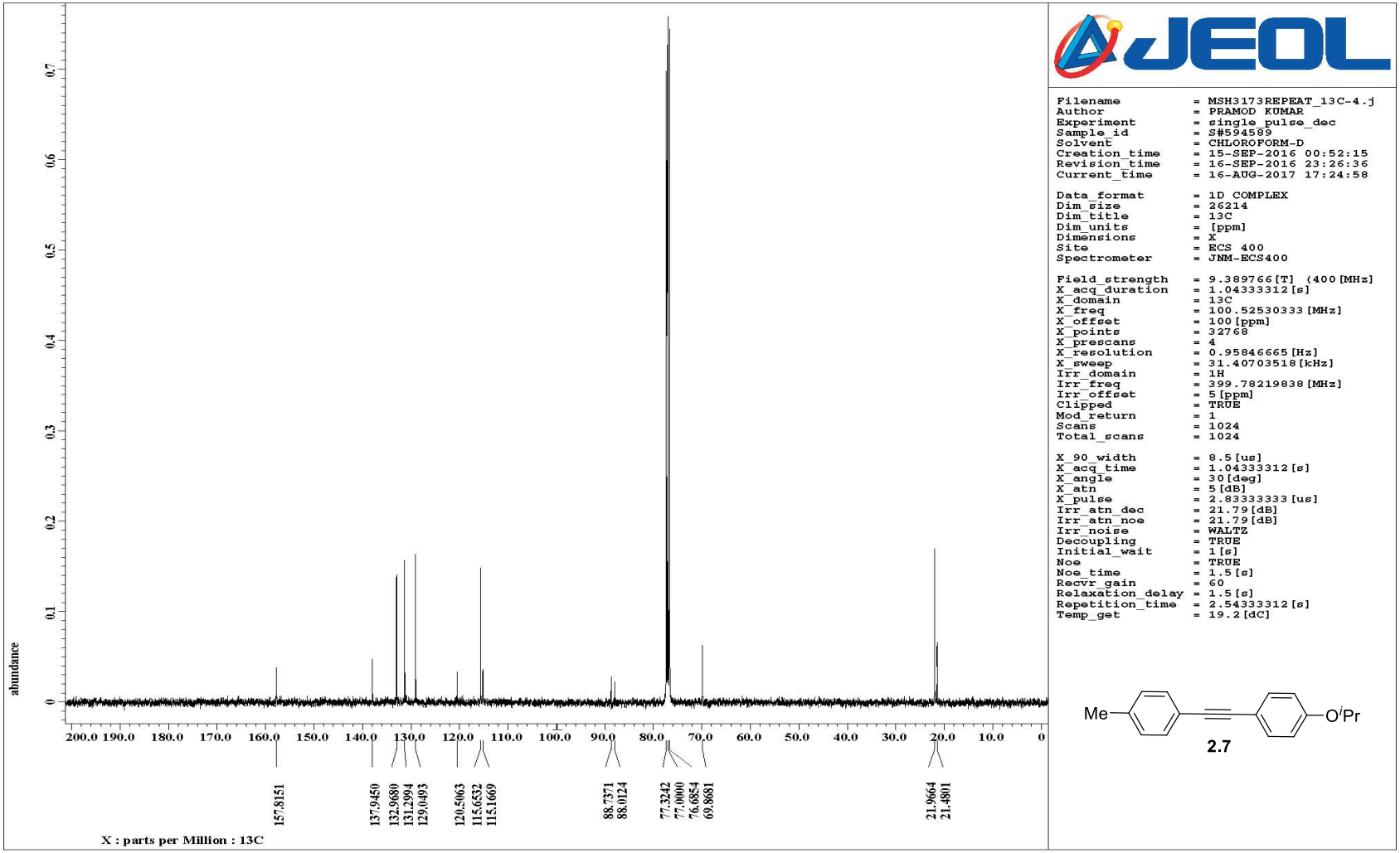
^{13}C NMR spectrum of 2-(*p*-tolylethynyl)naphthalene **(2.6)**



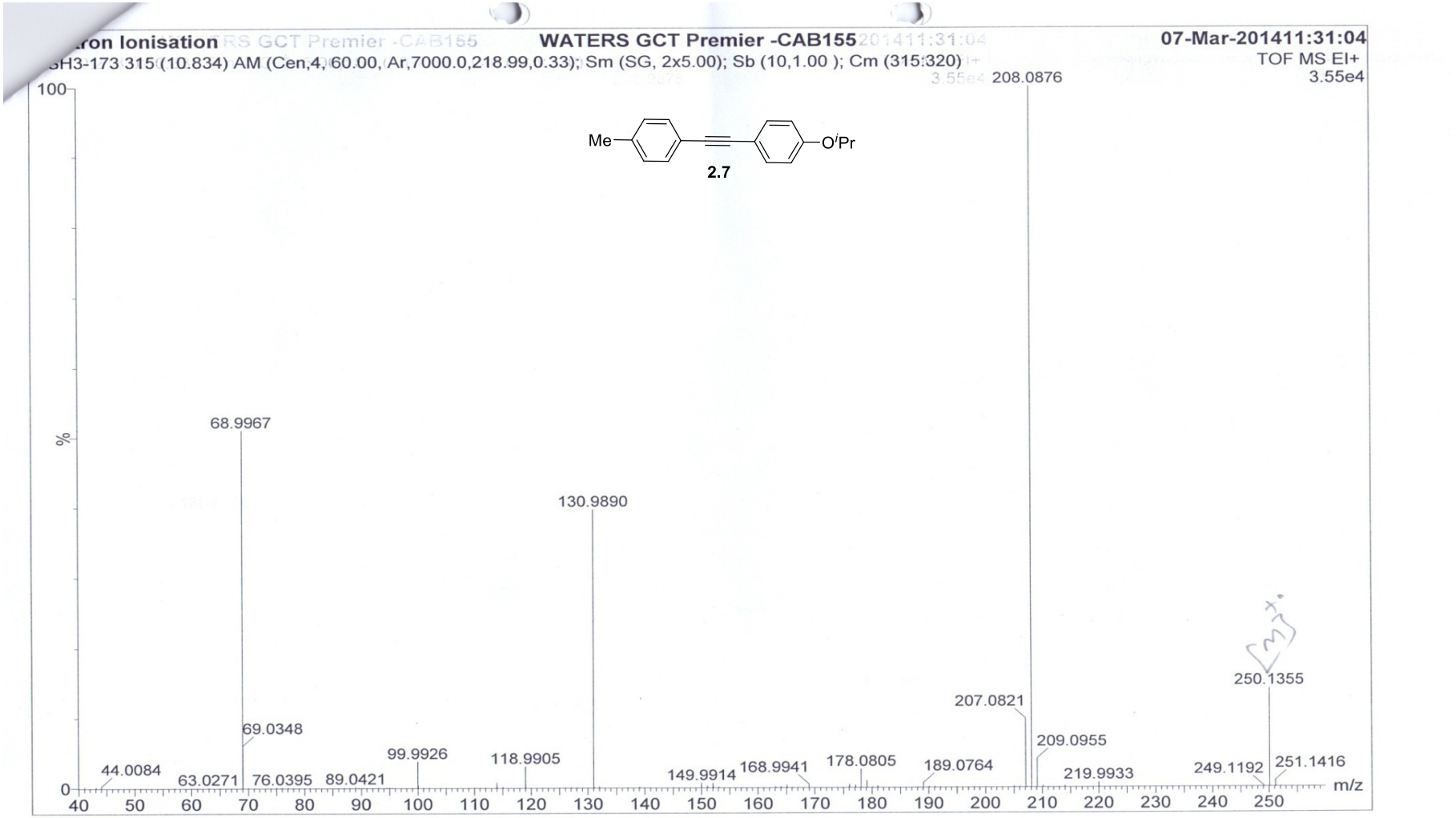
HRMS spectrum of 2-(*p*-tolylethynyl)naphthalene **(2.6)**



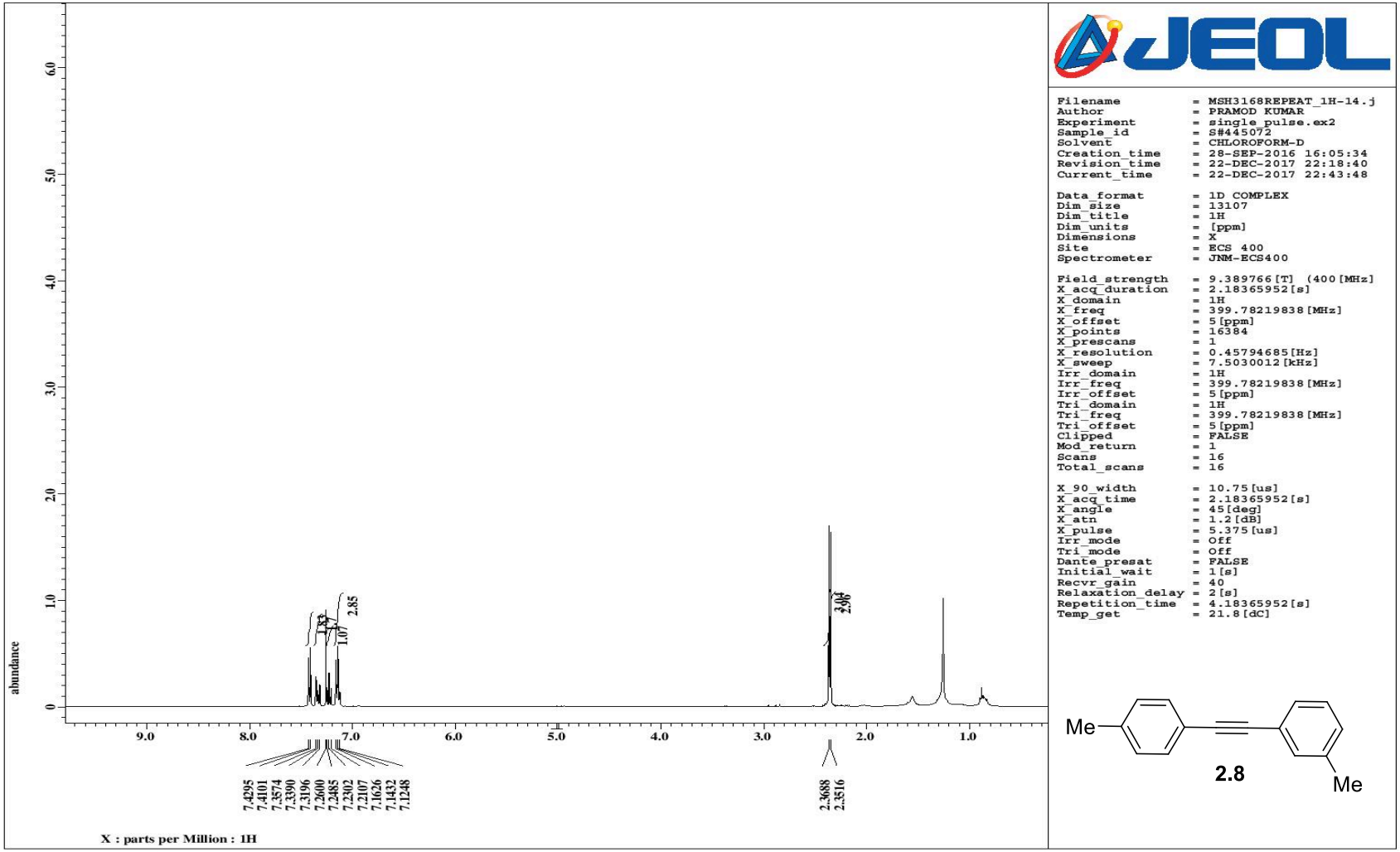
^{1}H NMR spectrum of 1-isopropoxy-4-(*p*-tolylethynyl)benzene **(2.7)**



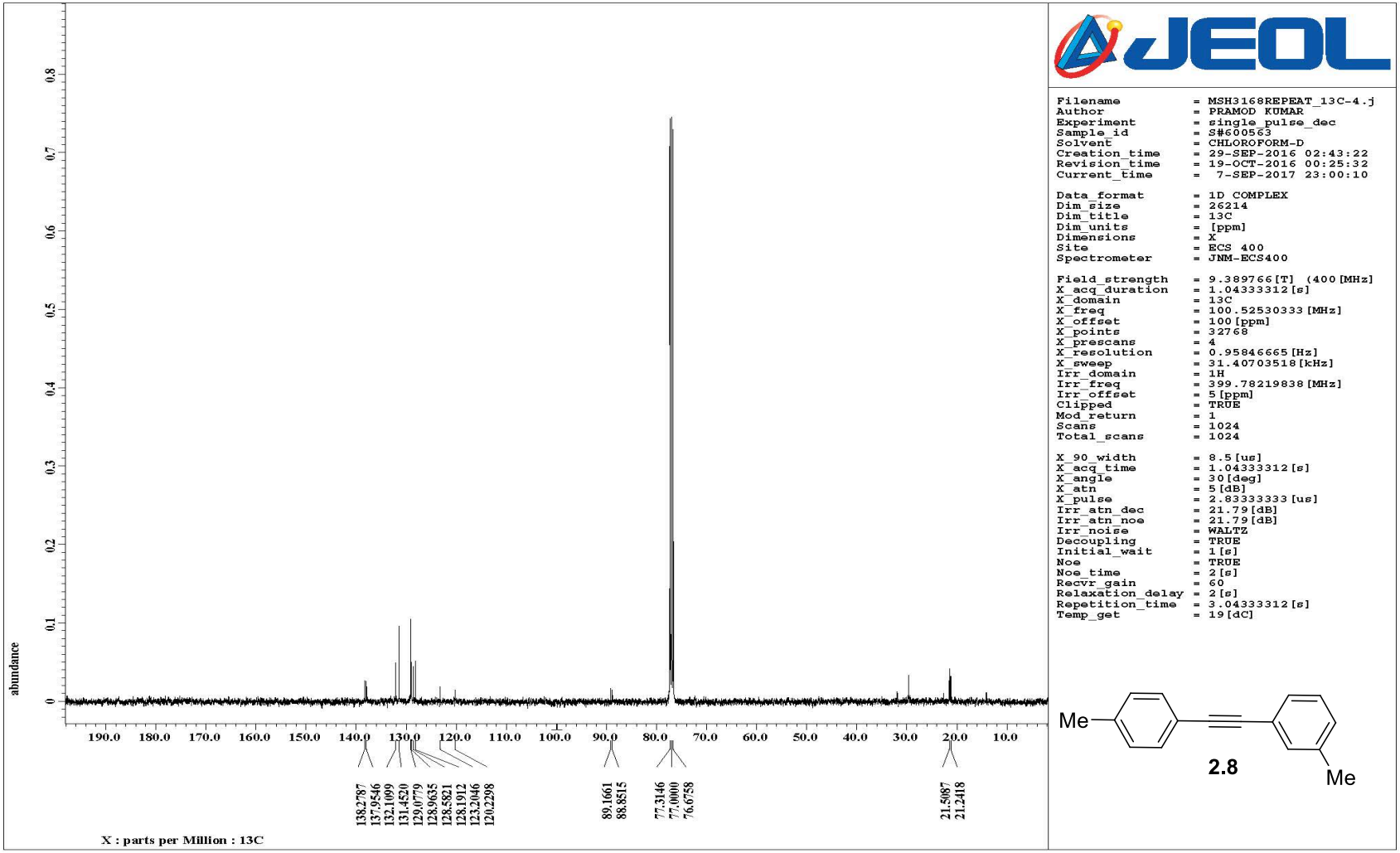
^{13}C NMR spectrum of 1-isopropoxy-4-(*p*-tolylethynyl)benzene **(2.7)**



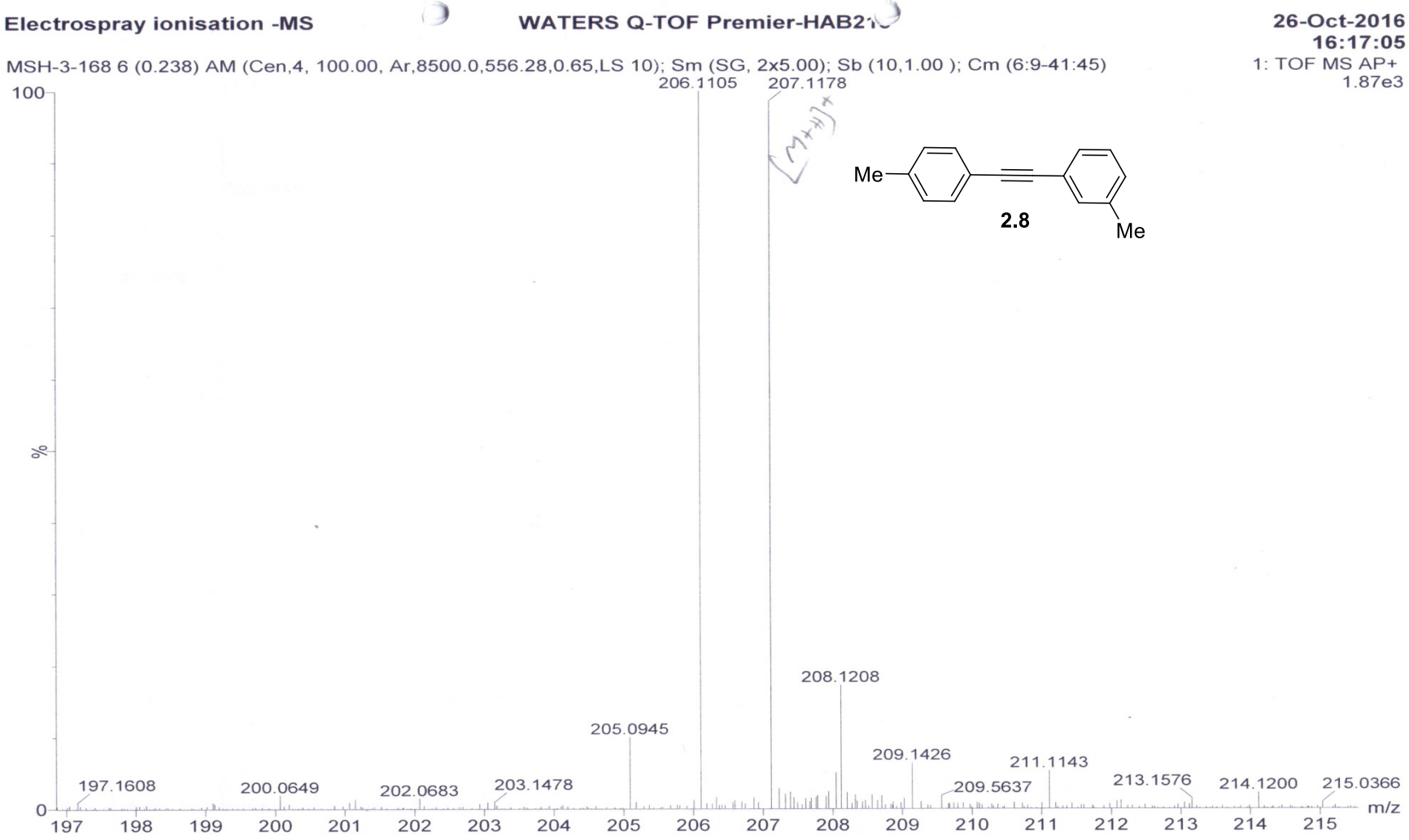
HRMS spectrum of 1-isopropoxy-4-(*p*-tolylethynyl)benzene **(2.7)**



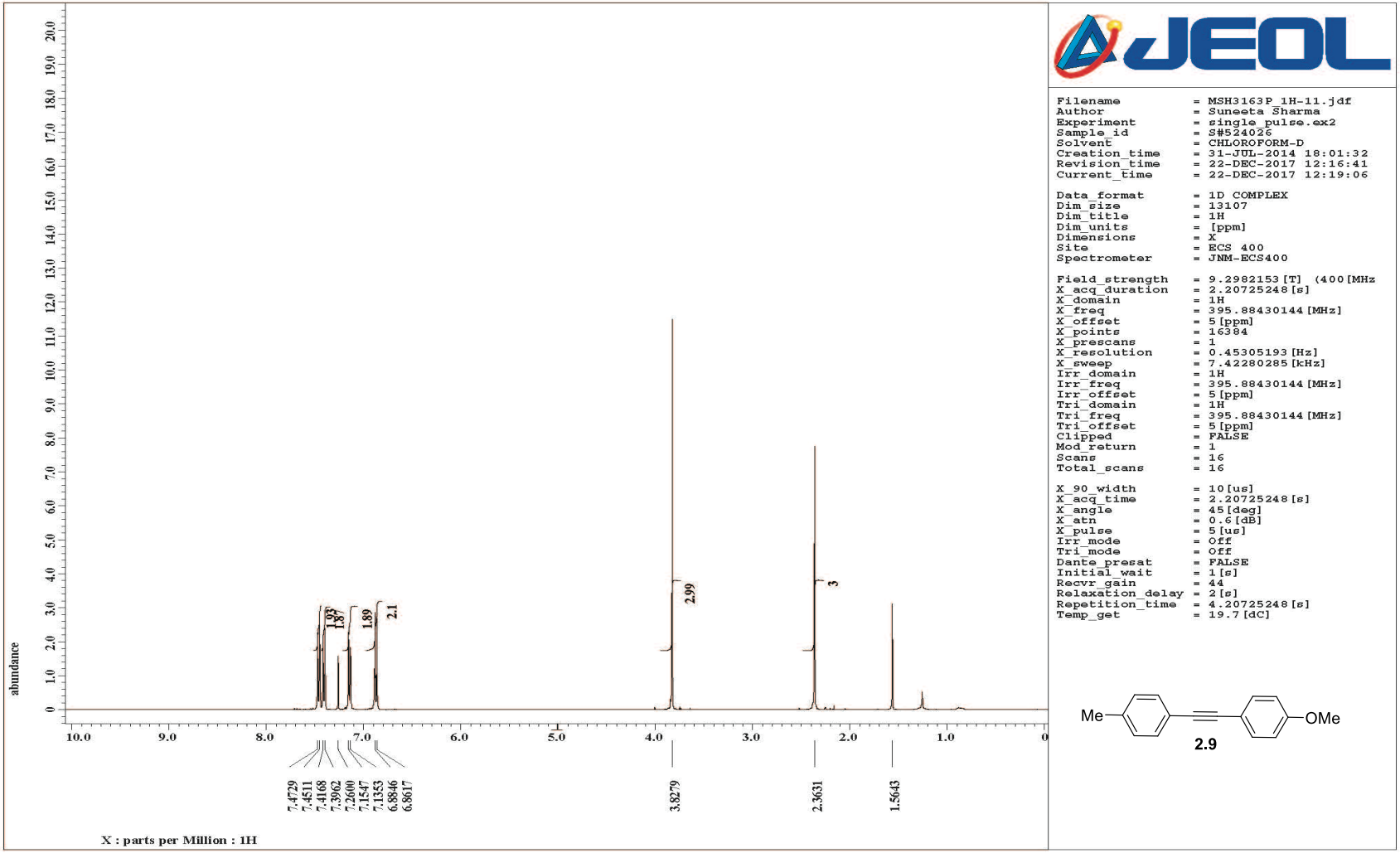
^{1}H NMR spectrum of 1-methyl-3-(*p*-tolylethynyl)benzene **(2.8)**



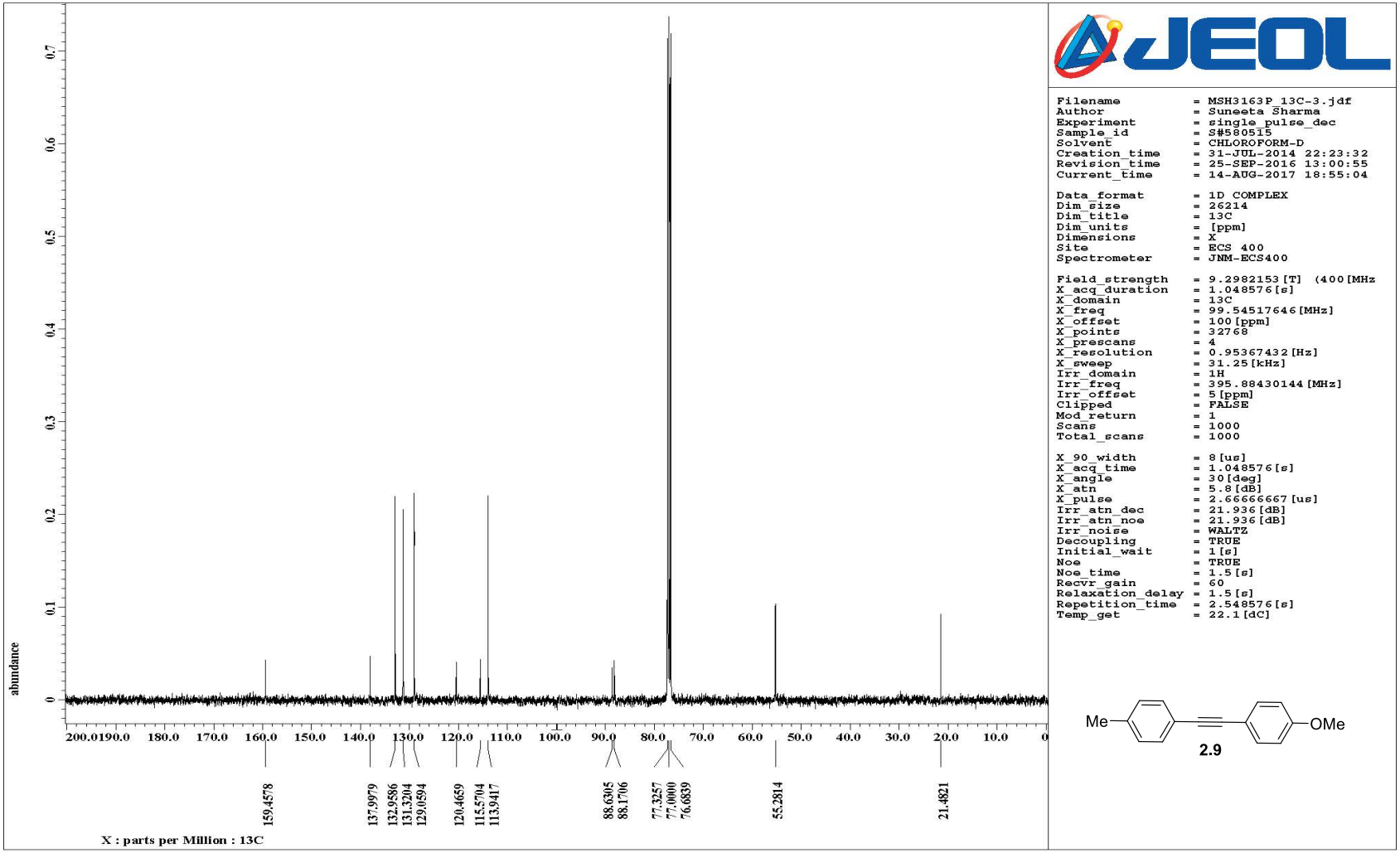
^{13}C NMR spectrum of 1-methyl-3-(*p*-tolylethynyl)benzene **(2.8)**



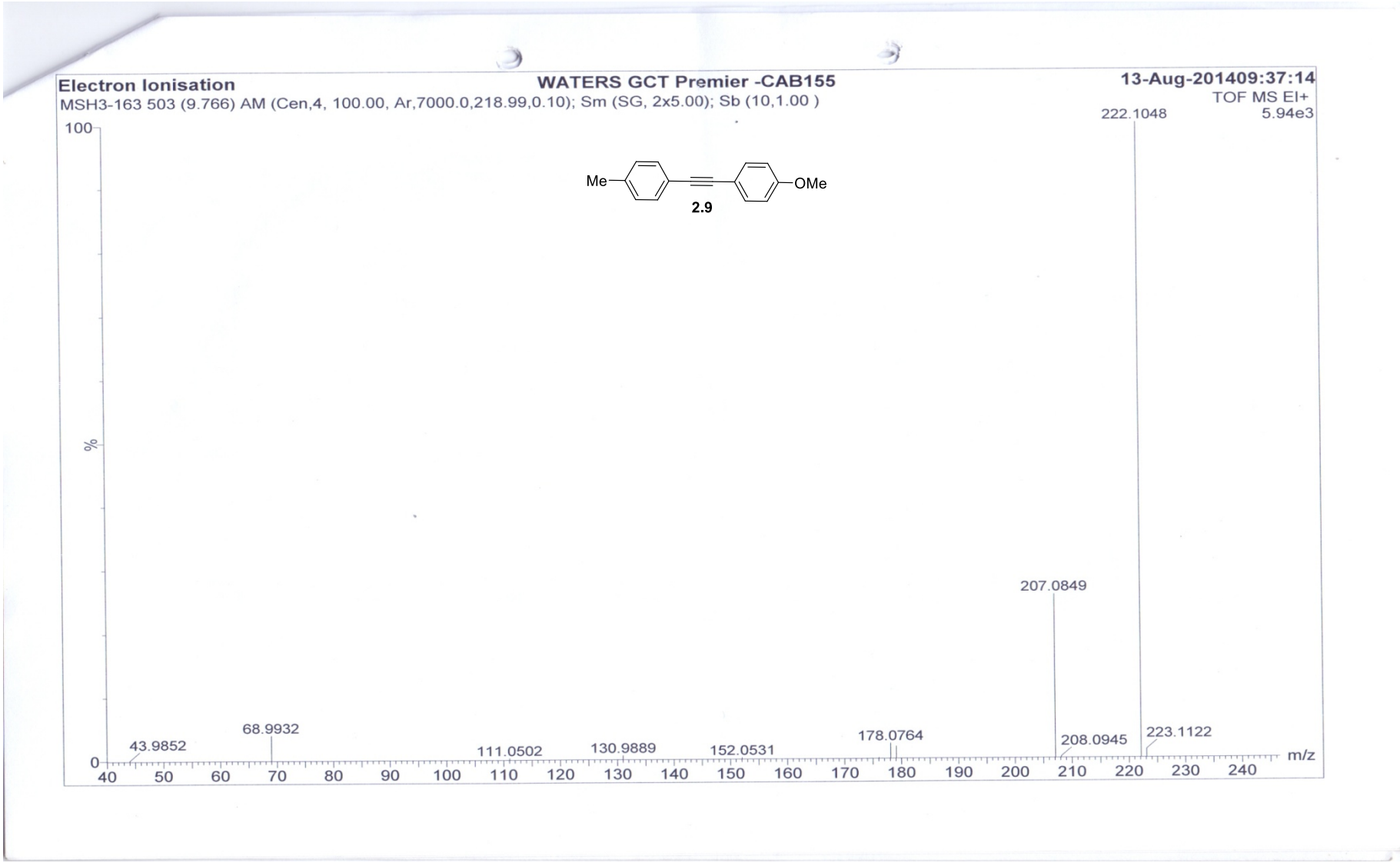
HRMS spectrum of 1-methyl-3-(*p*-tolylethynyl)benzene **(2.8)**



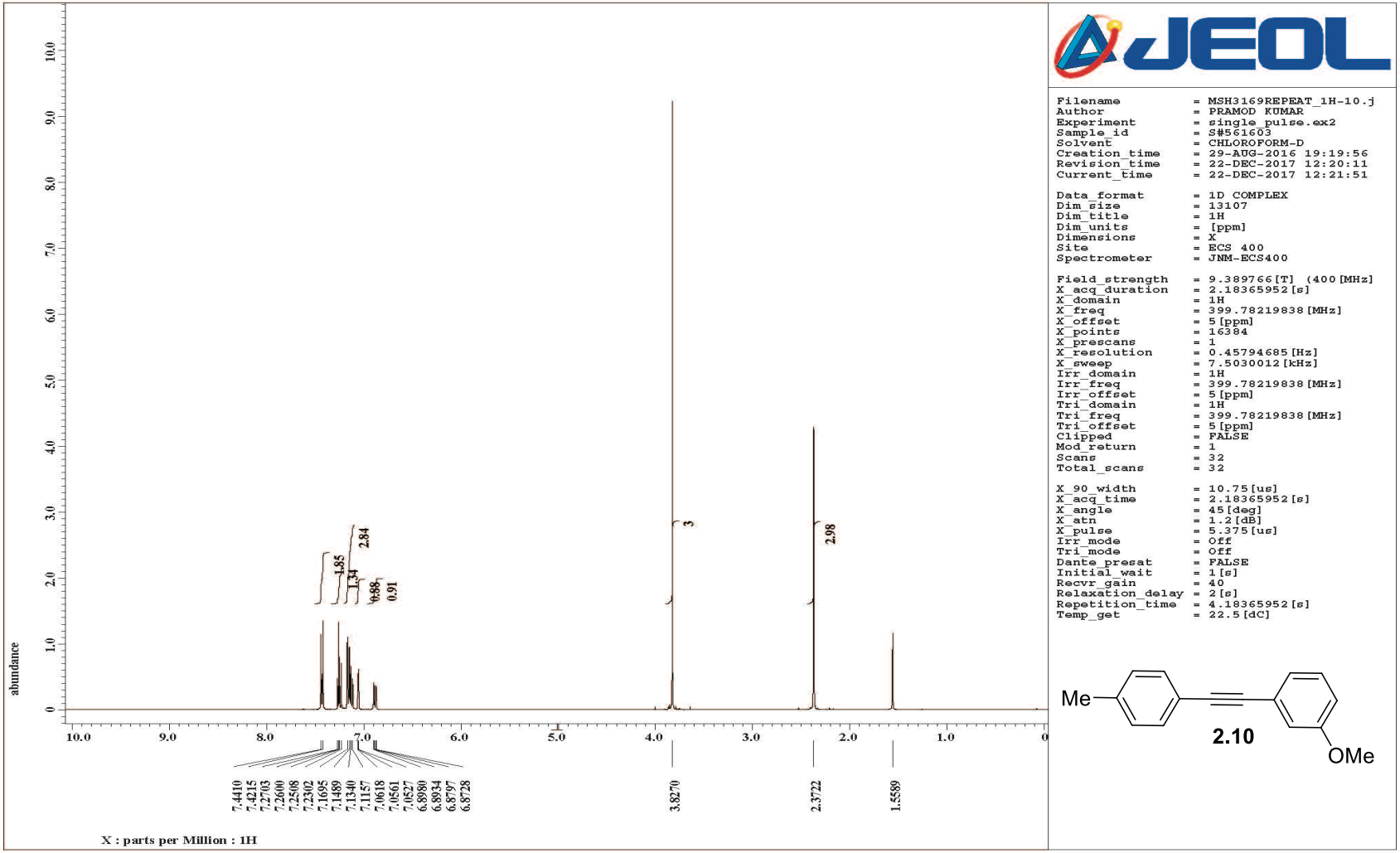
^{1}H NMR spectrum of 1-methoxy-4-(*p*-tolylethynyl)benzene **(2.9)**



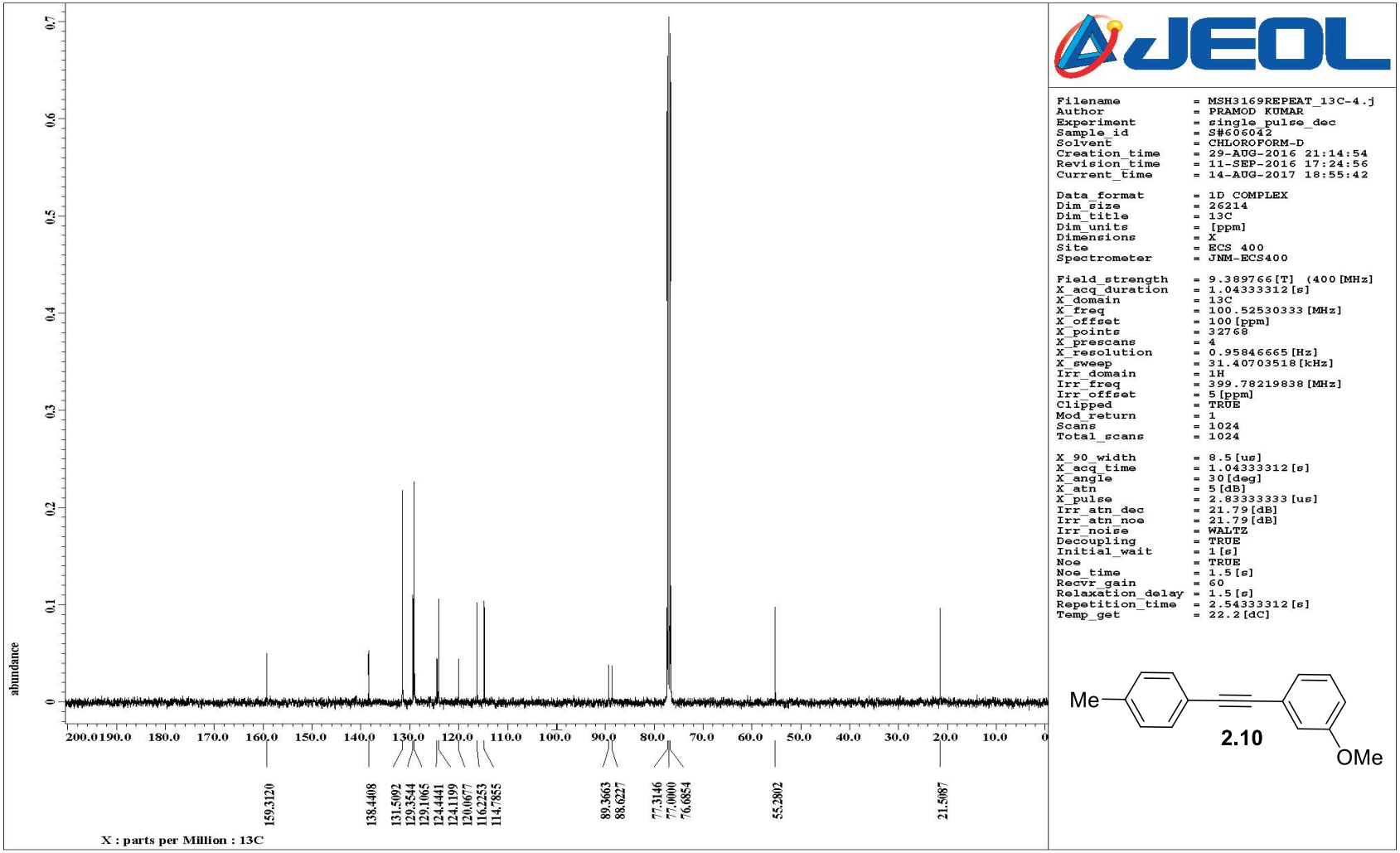
^{13}C NMR spectrum of 1-methoxy-4-(*p*-tolylethynyl)benzene **(2.9)**



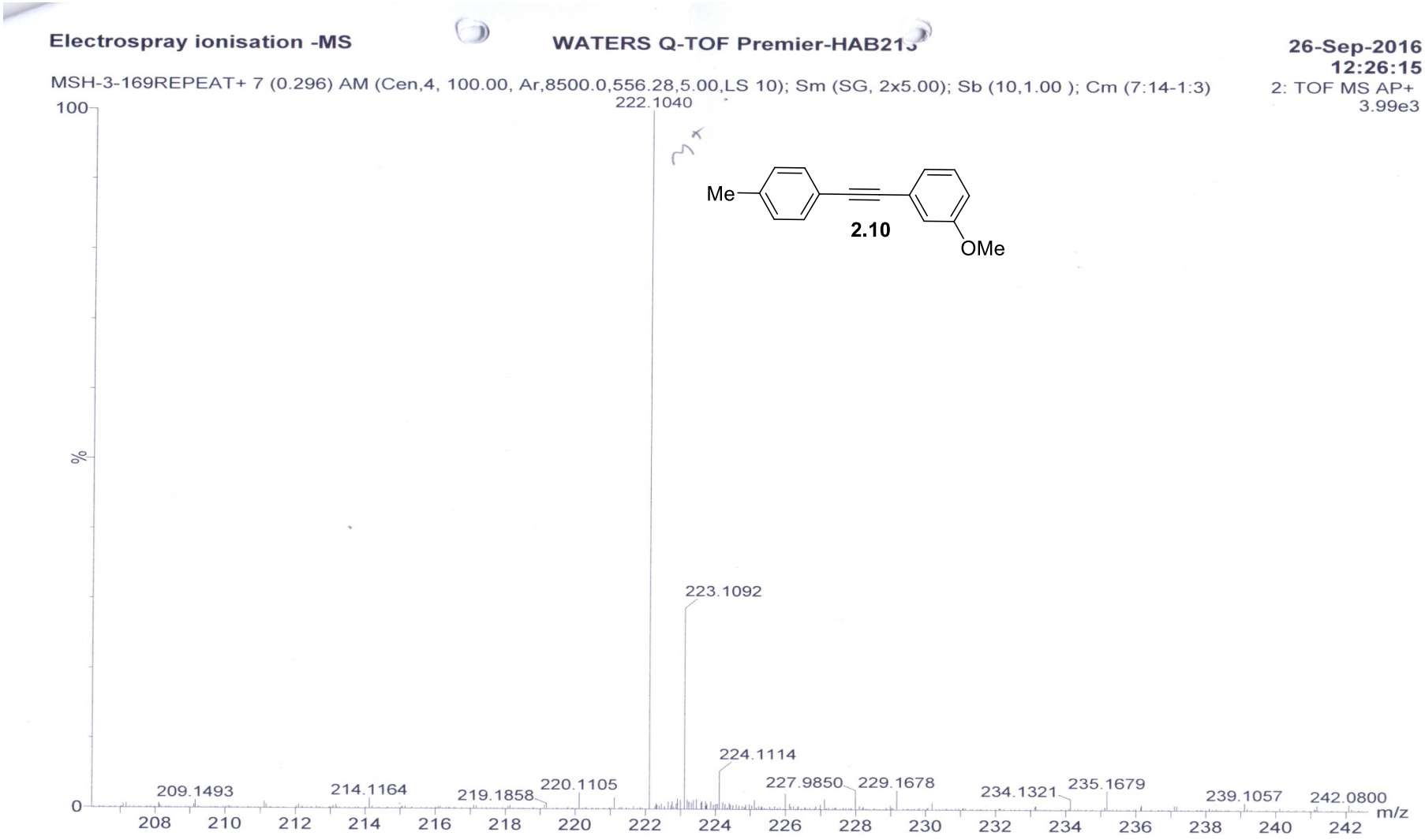
HRMS spectrum of 1-methoxy-4-(*p*-tolylethynyl)benzene **(2.9)**



^{1}H NMR spectrum of 1-methoxy-3-(*p*-tolylethynyl)benzene **(2.10)**

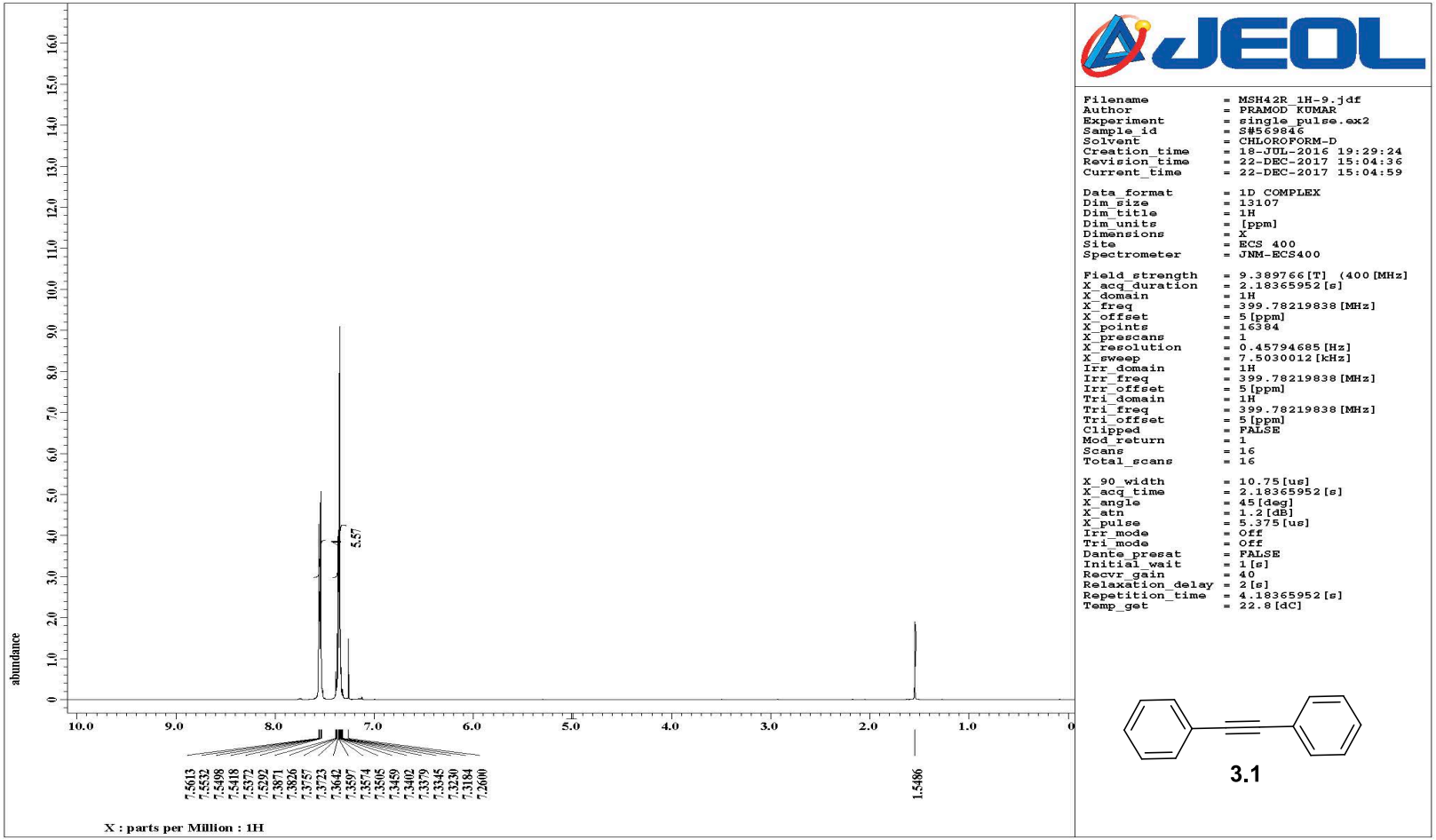


^{13}C NMR spectrum of 1-methoxy-3-(*p*-tolylethynyl)benzene **(2.10)**

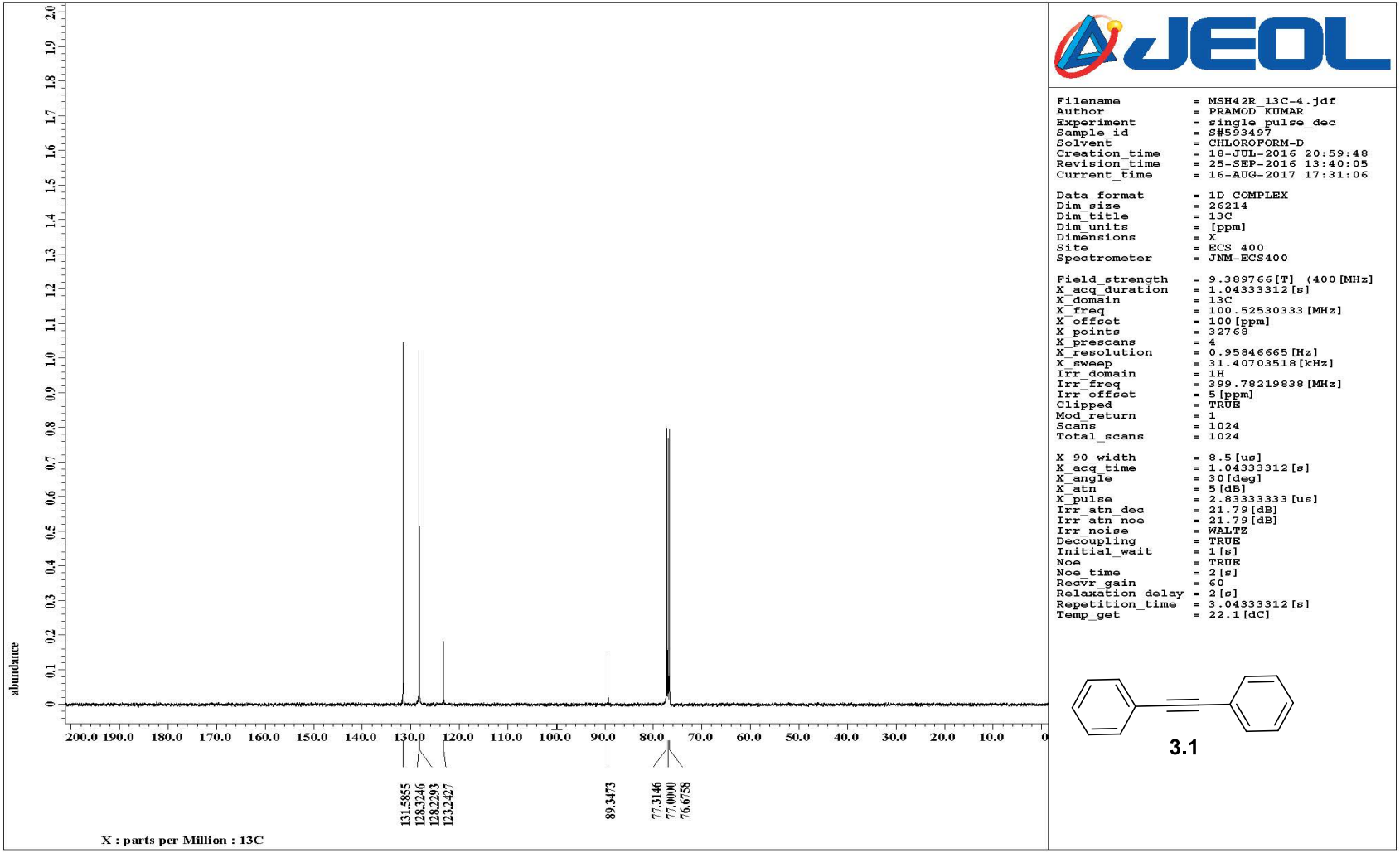


HRMS spectrum of 1-methoxy-3-(*p*-tolylethynyl)benzene **(2.10)**

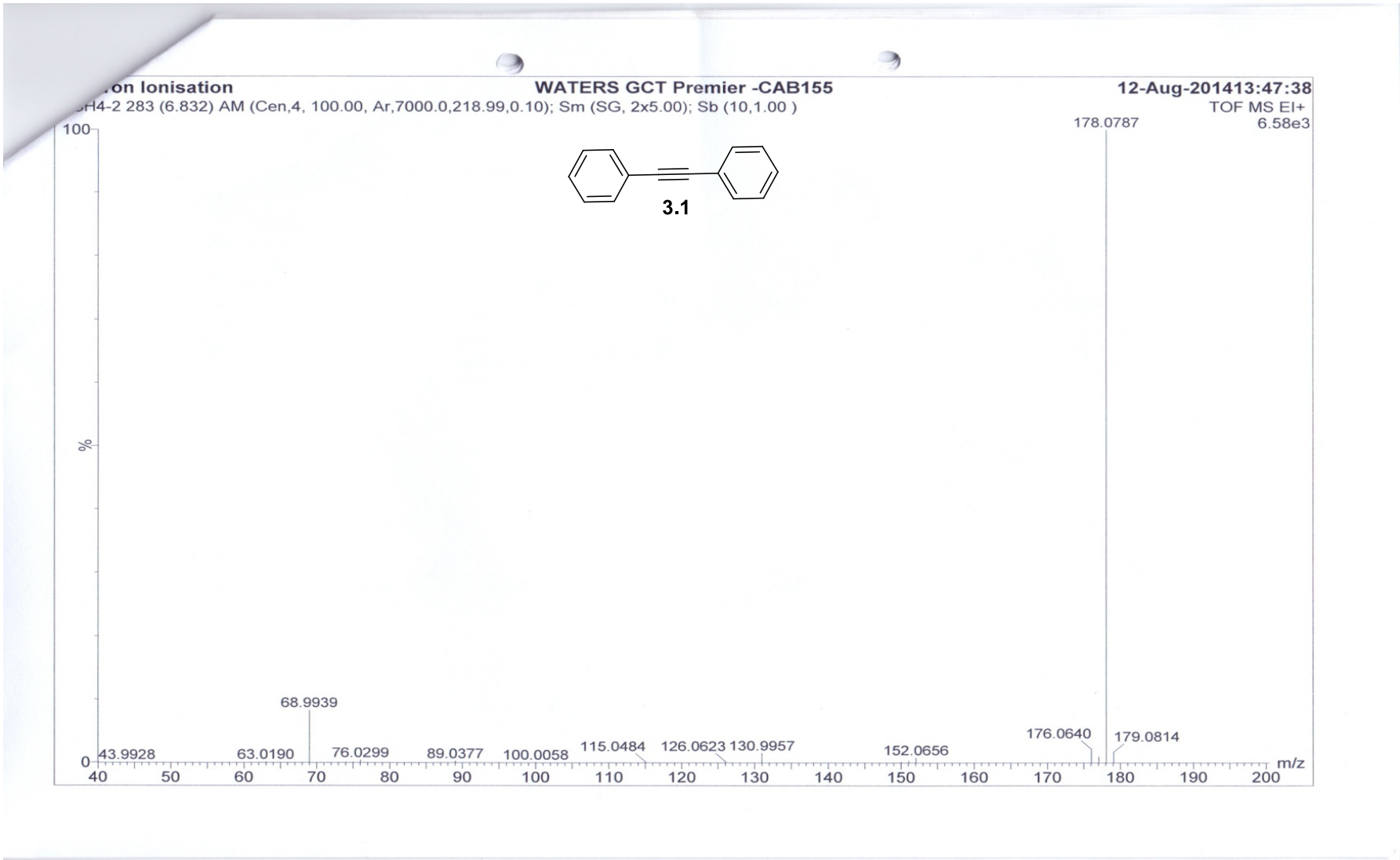
8. Copies of ^{1}H NMR, ^{13}C NMR and HRMS spectra of 3.1-3.30 (Table 3):



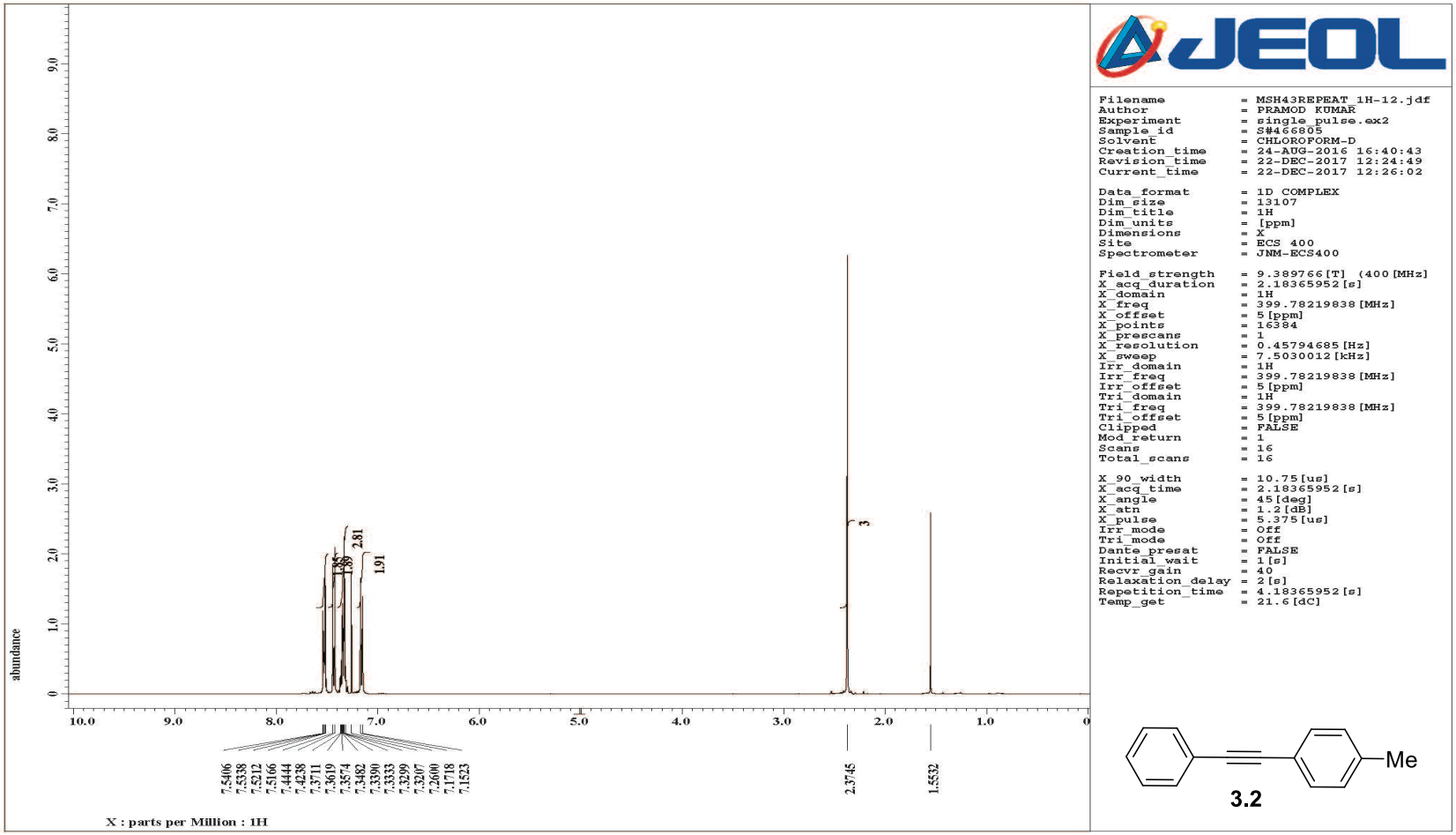
^{1}H NMR spectrum of 1,2-diphenylethyne **(3.1)**



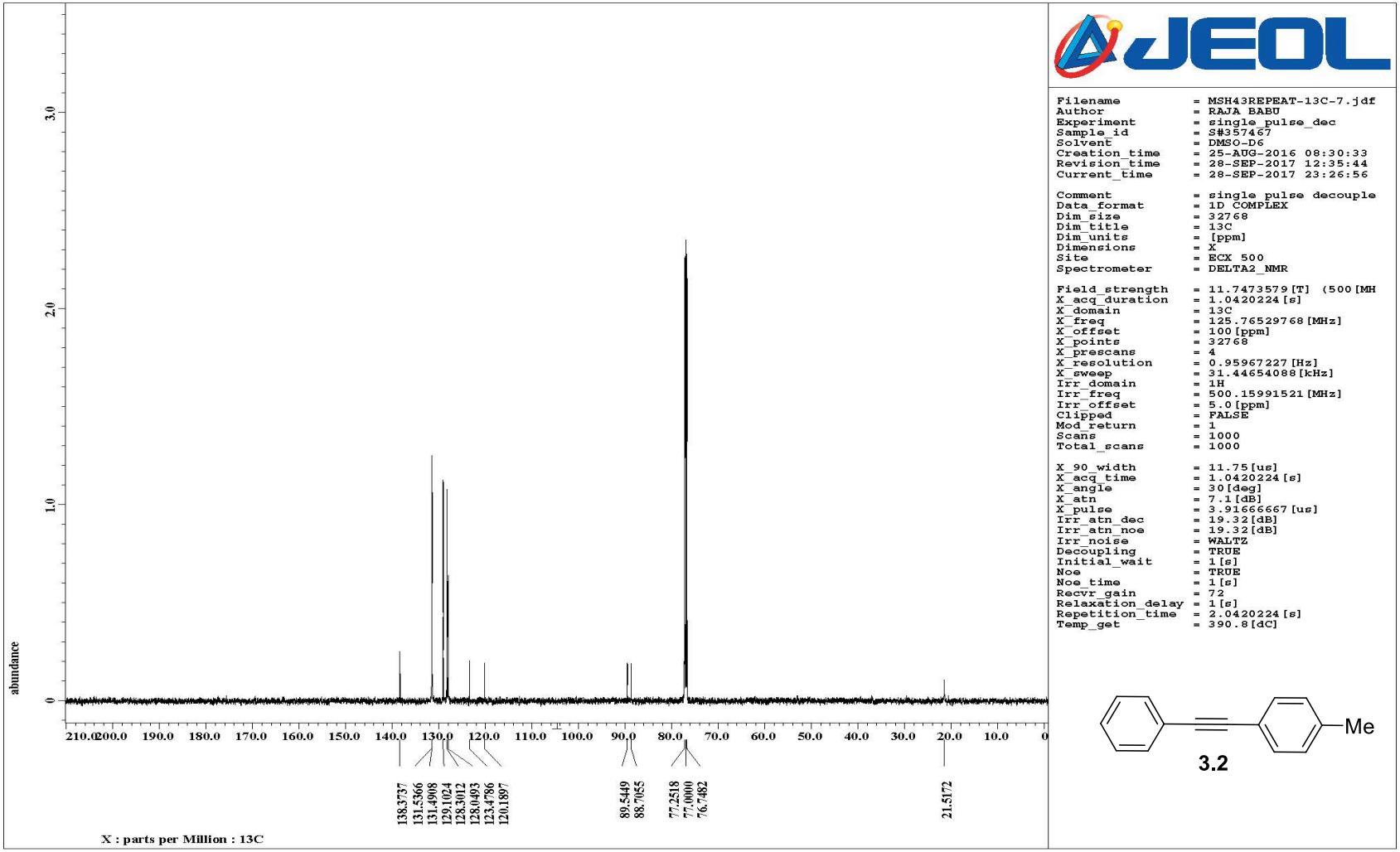
^{13}C NMR spectrum of 1,2-diphenylethyne **(3.1)**



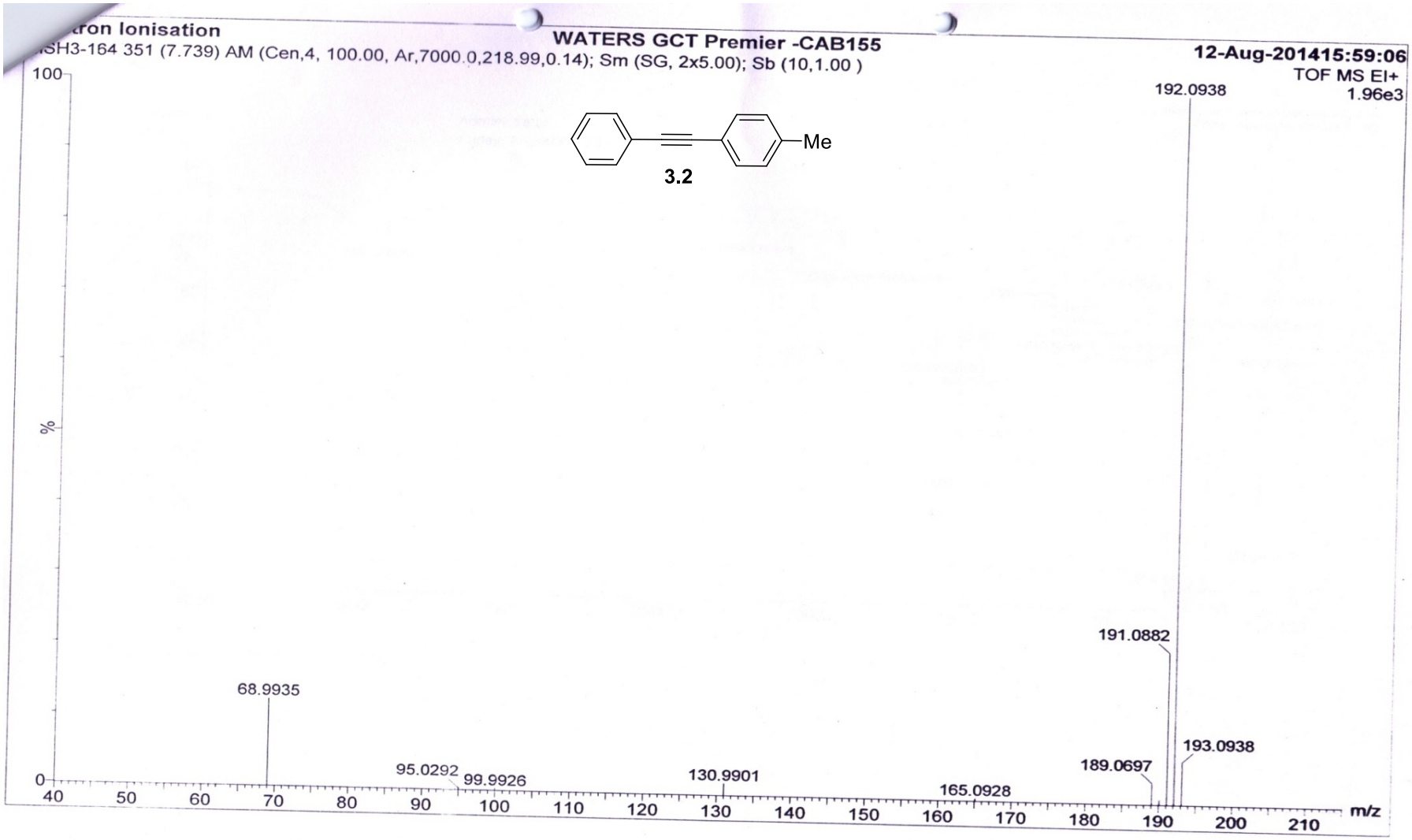
HRMS spectrum of 1,2-diphenylethyne **(3.1)**



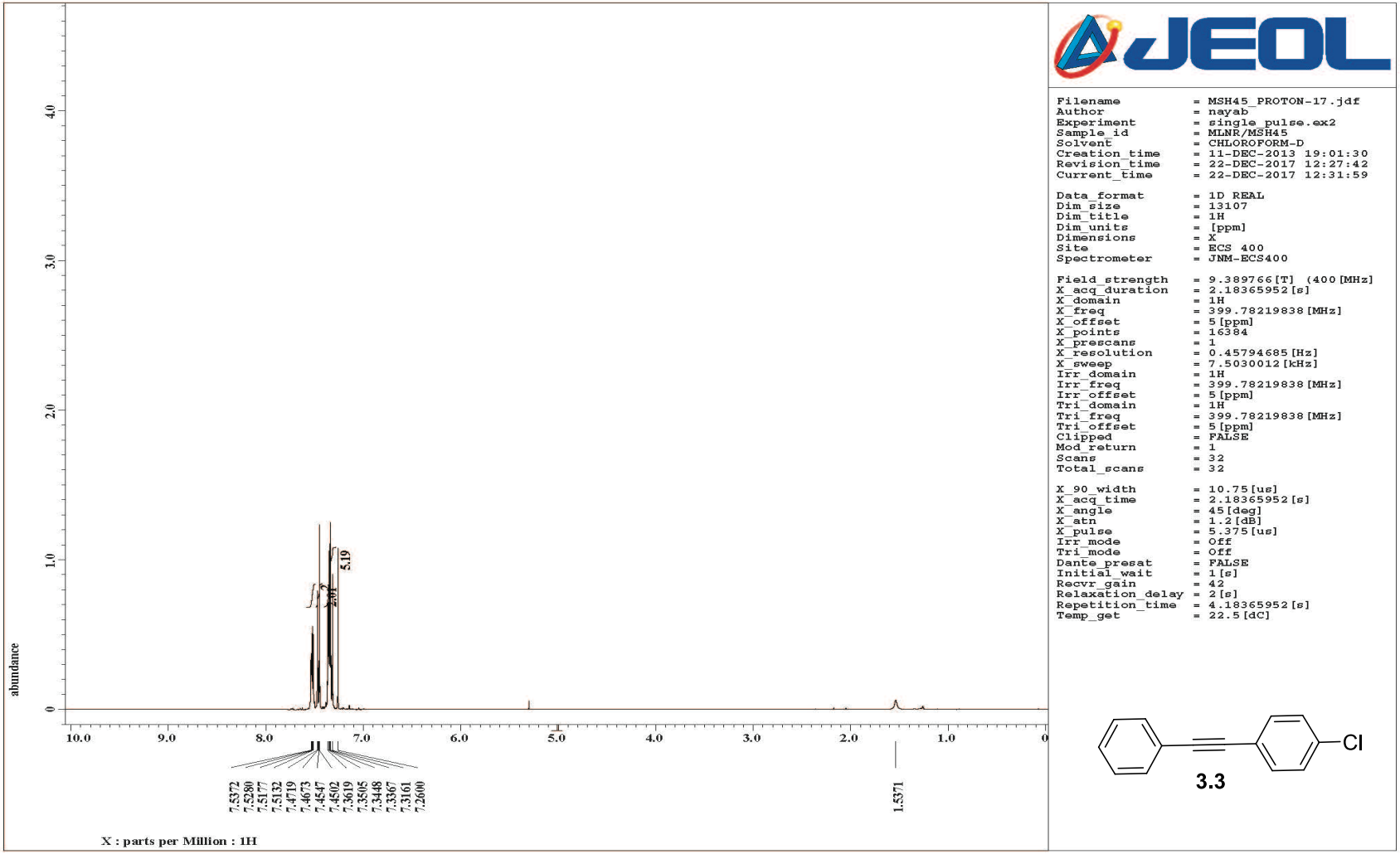
^{1}H NMR spectrum of 1-methyl-4-(phenylethynyl)benzene **(3.2)**



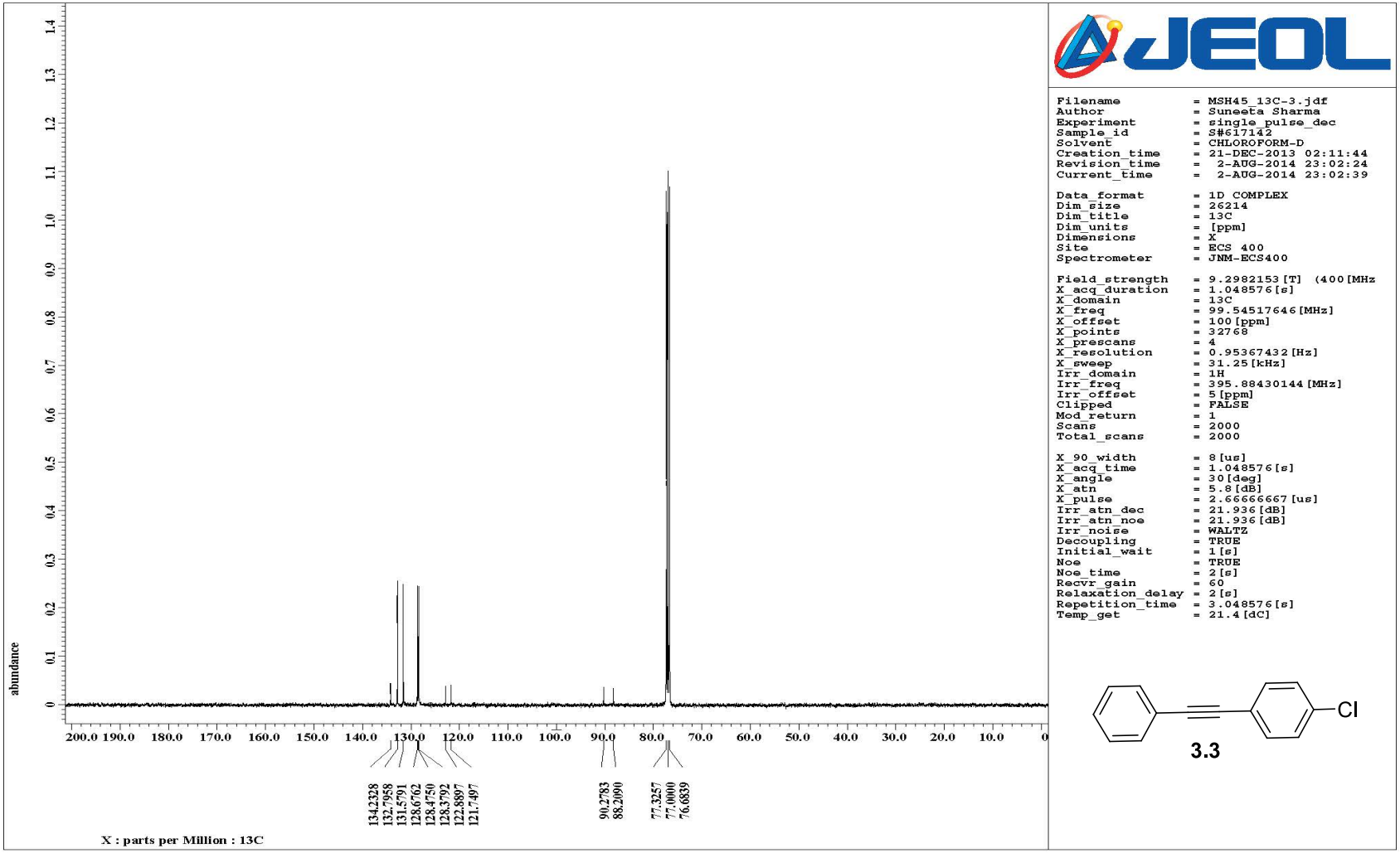
^{13}C NMR spectrum of 1-methyl-4-(phenylethynyl)benzene **(3.2)**



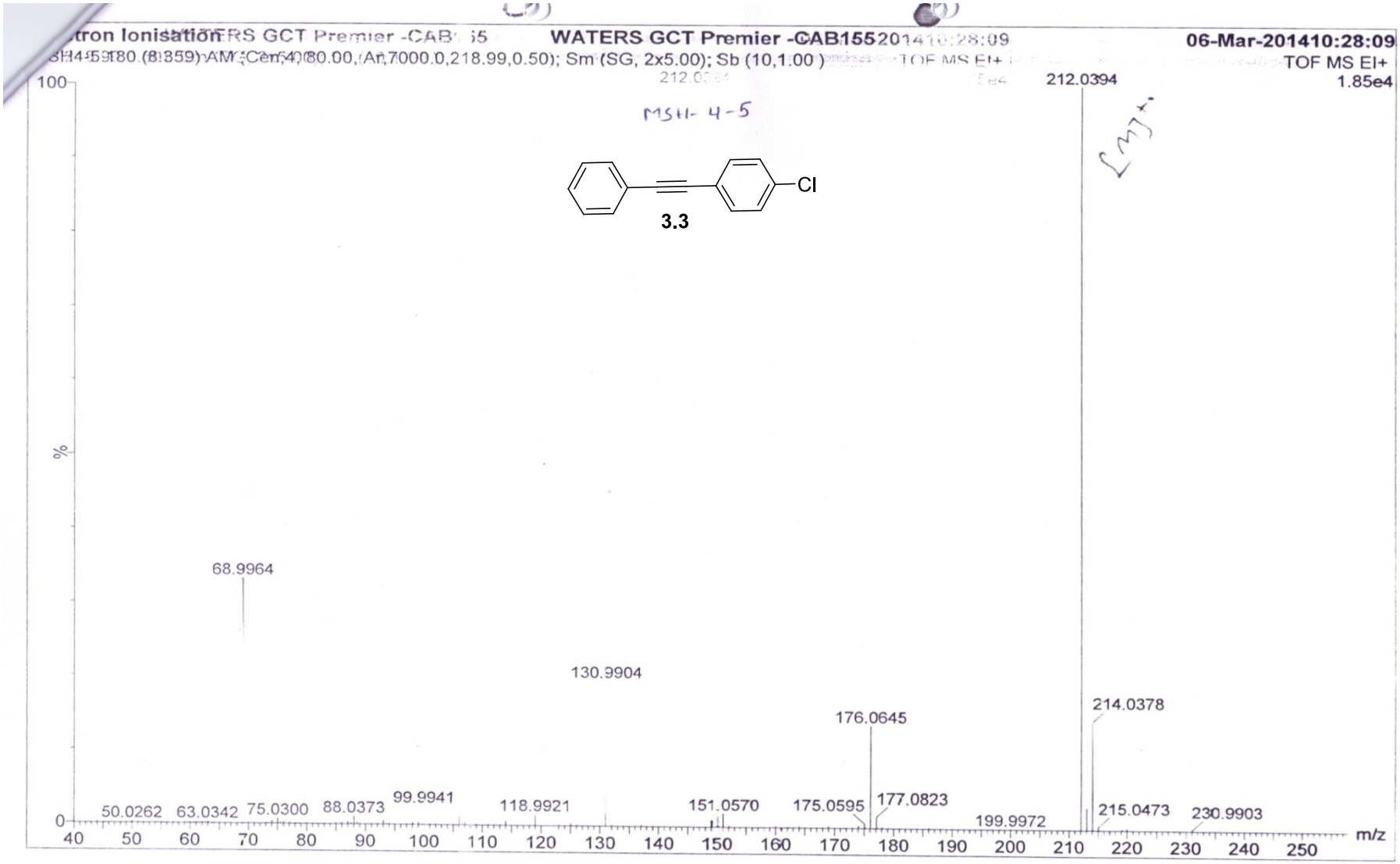
HRMS spectrum of 1-methyl-4-(phenylethynyl)benzene **(3.2)**



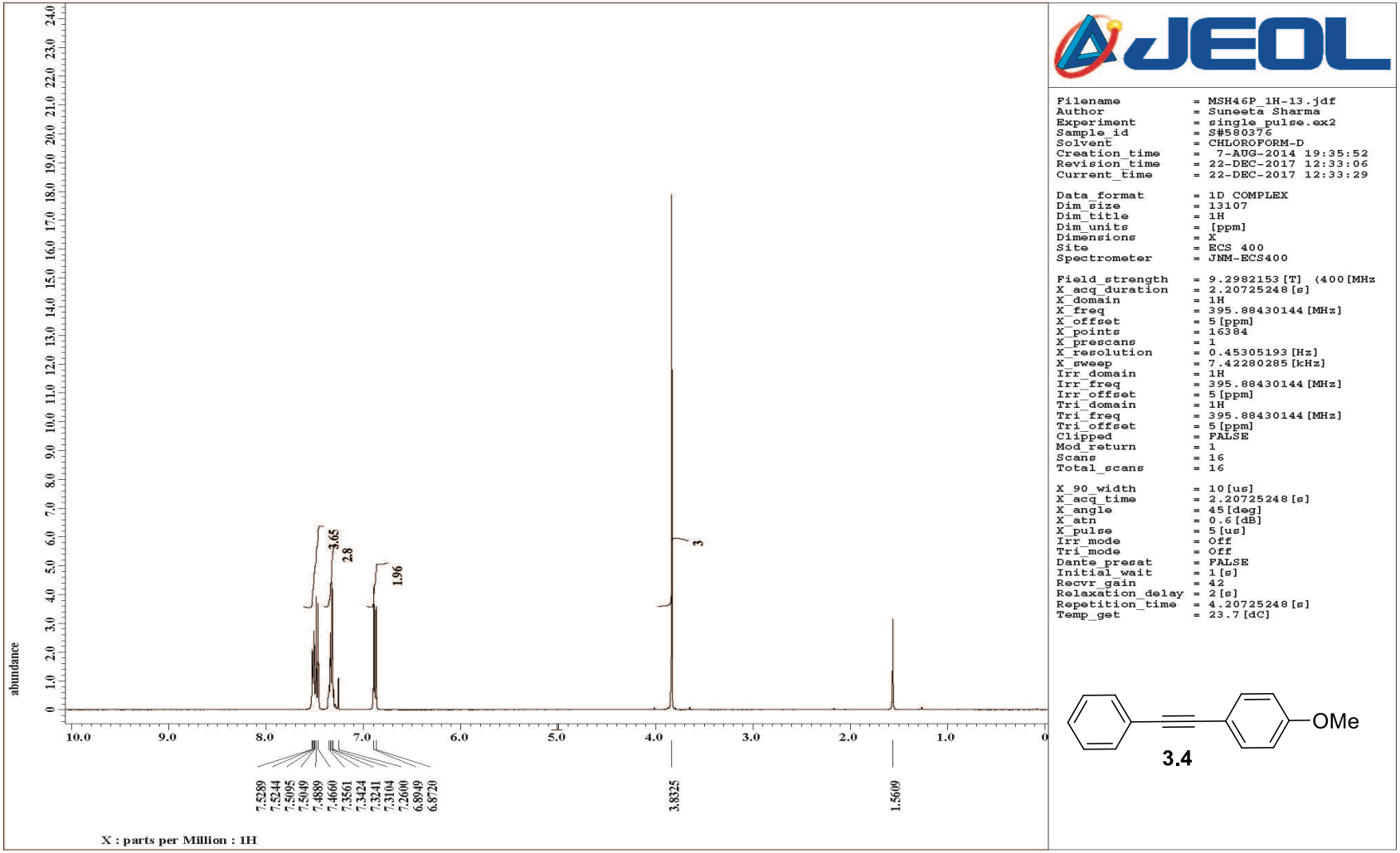
^{1}H NMR spectrum of 1-chloro-4-(phenylethynyl)benzene **(3.3)**



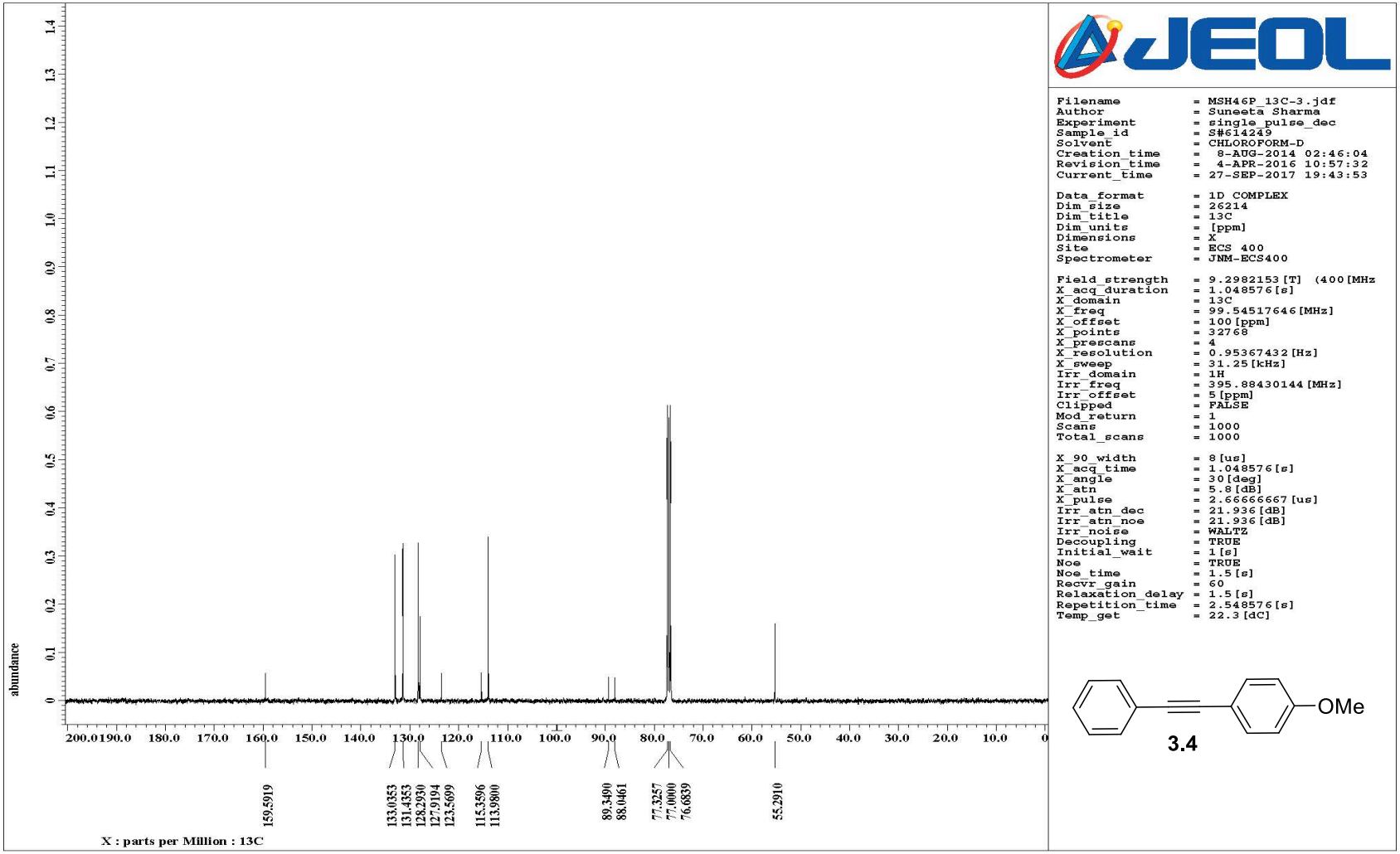
^{13}C NMR spectrum of 1-chloro-4-(phenylethynyl)benzene **(3.3)**



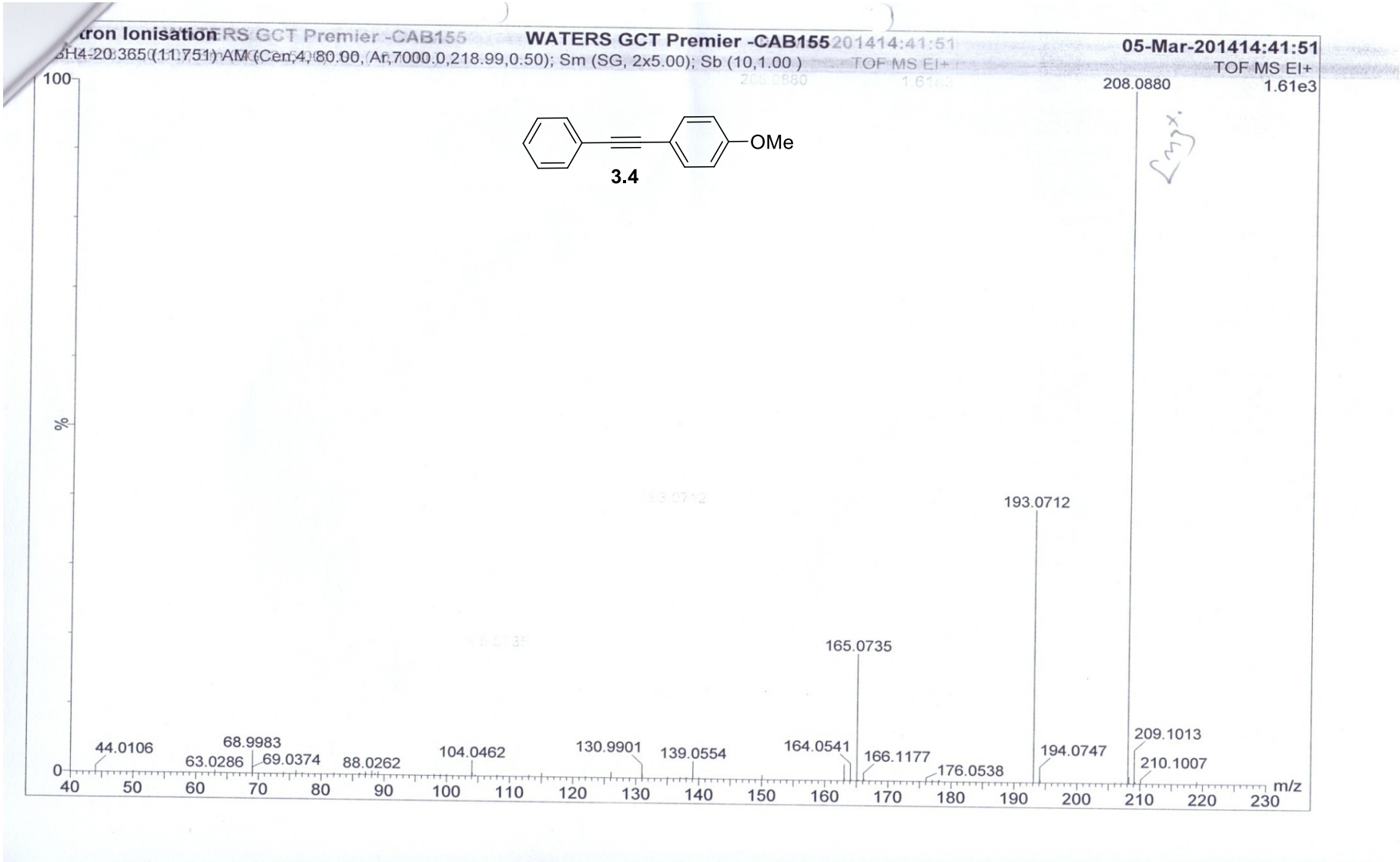
HRMS spectrum of 1-chloro-4-(phenylethynyl)benzene **(3.3)**



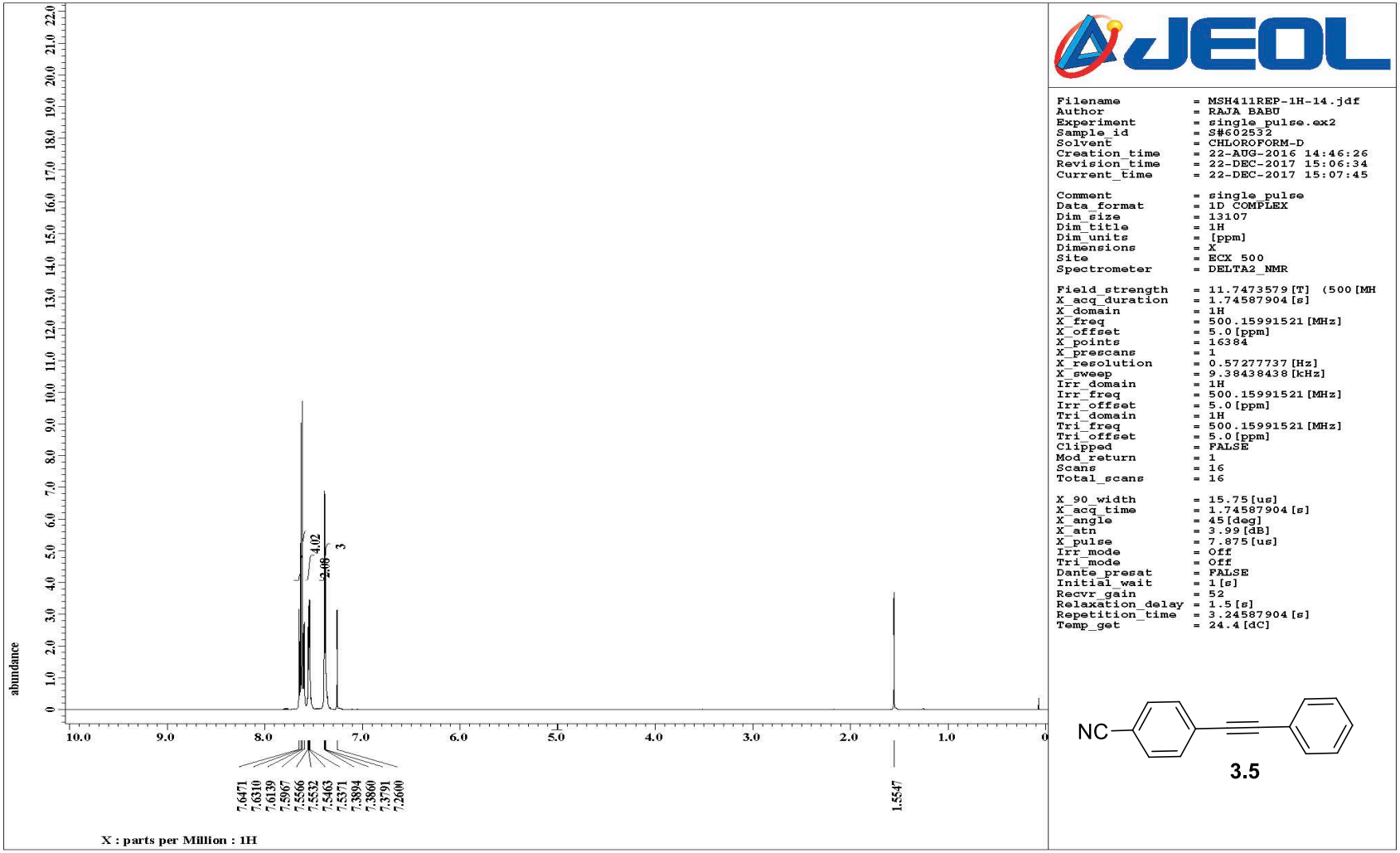
^{1}H NMR spectrum of 1-methoxy-4-(phenylethynyl)benzene **(3.4)**



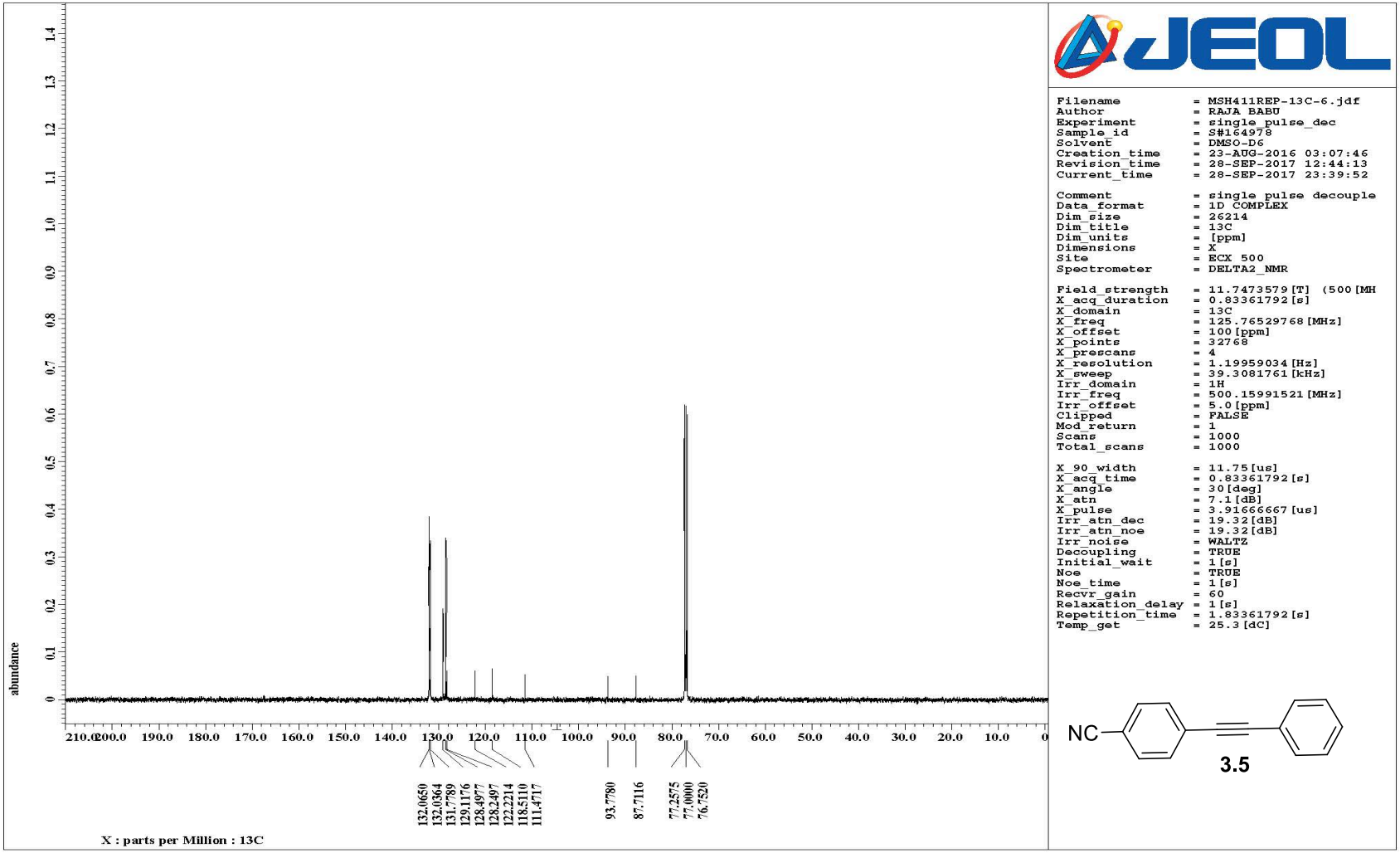
^{13}C NMR spectrum of 1-methoxy-4-(phenylethynyl)benzene **(3.4)**



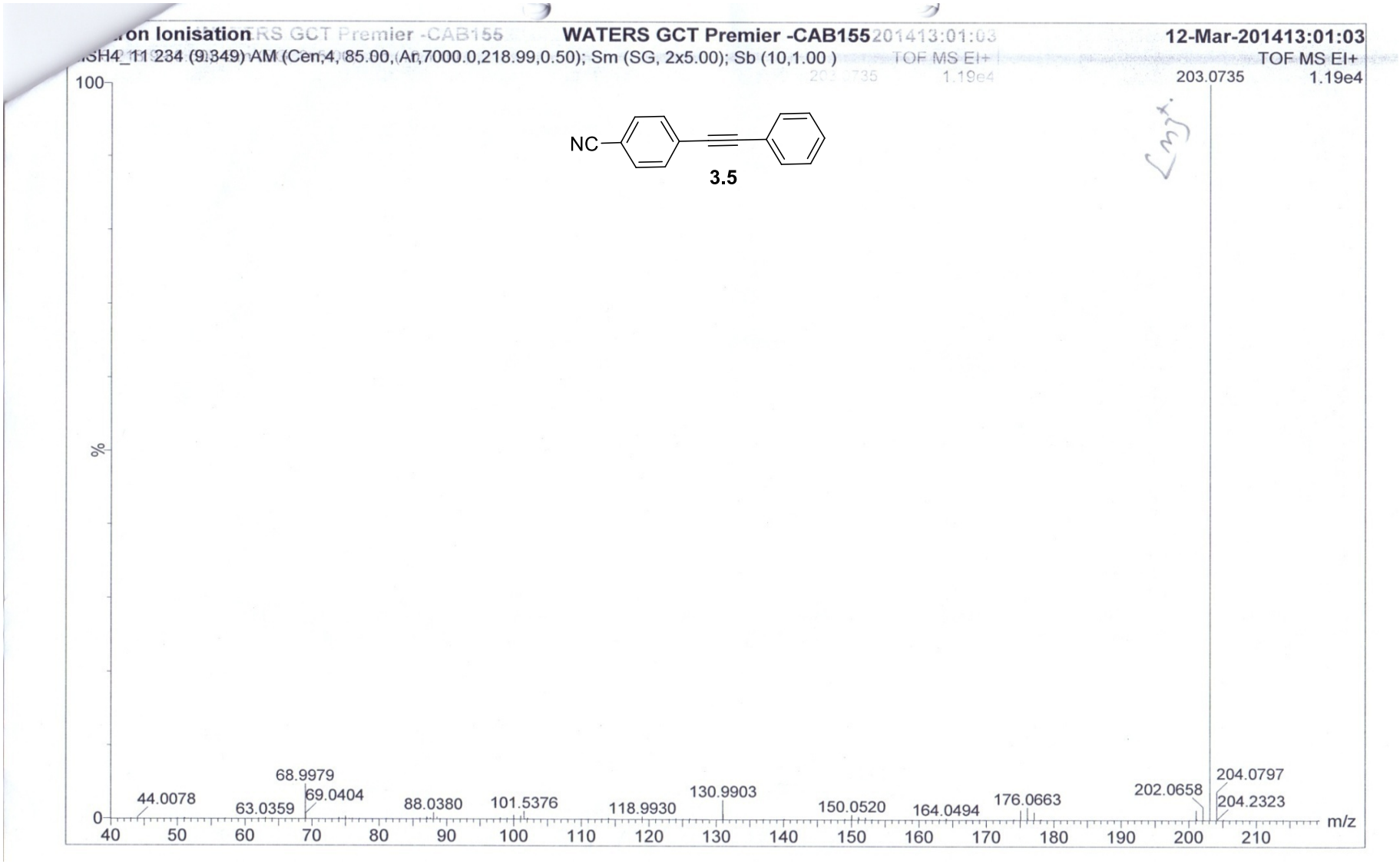
HRMS spectrum of 1-methoxy-4-(phenylethynyl)benzene **(3.4)**



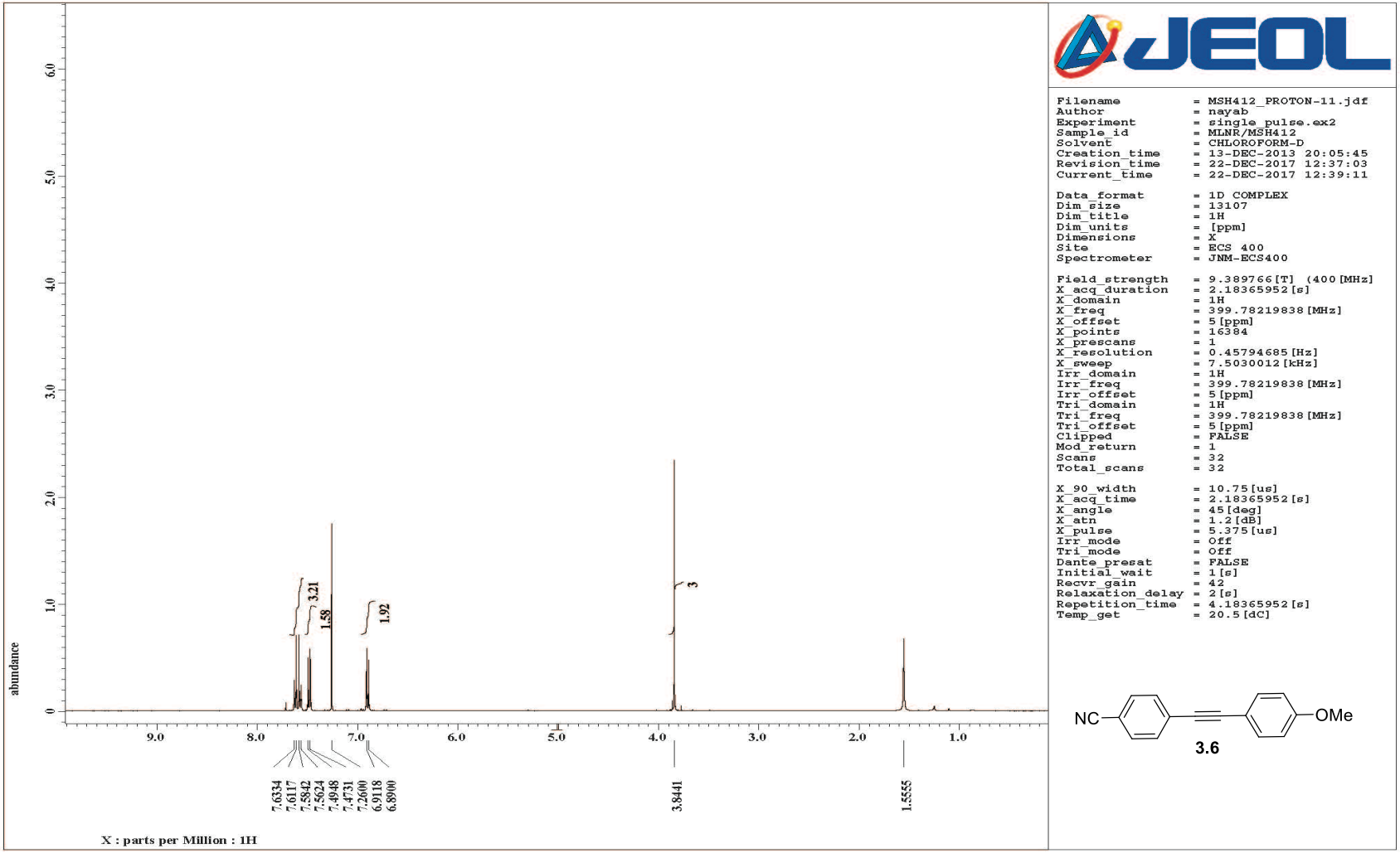
^{1}H NMR spectrum of 4-(phenylethynyl)benzonitrile **(3.5)**



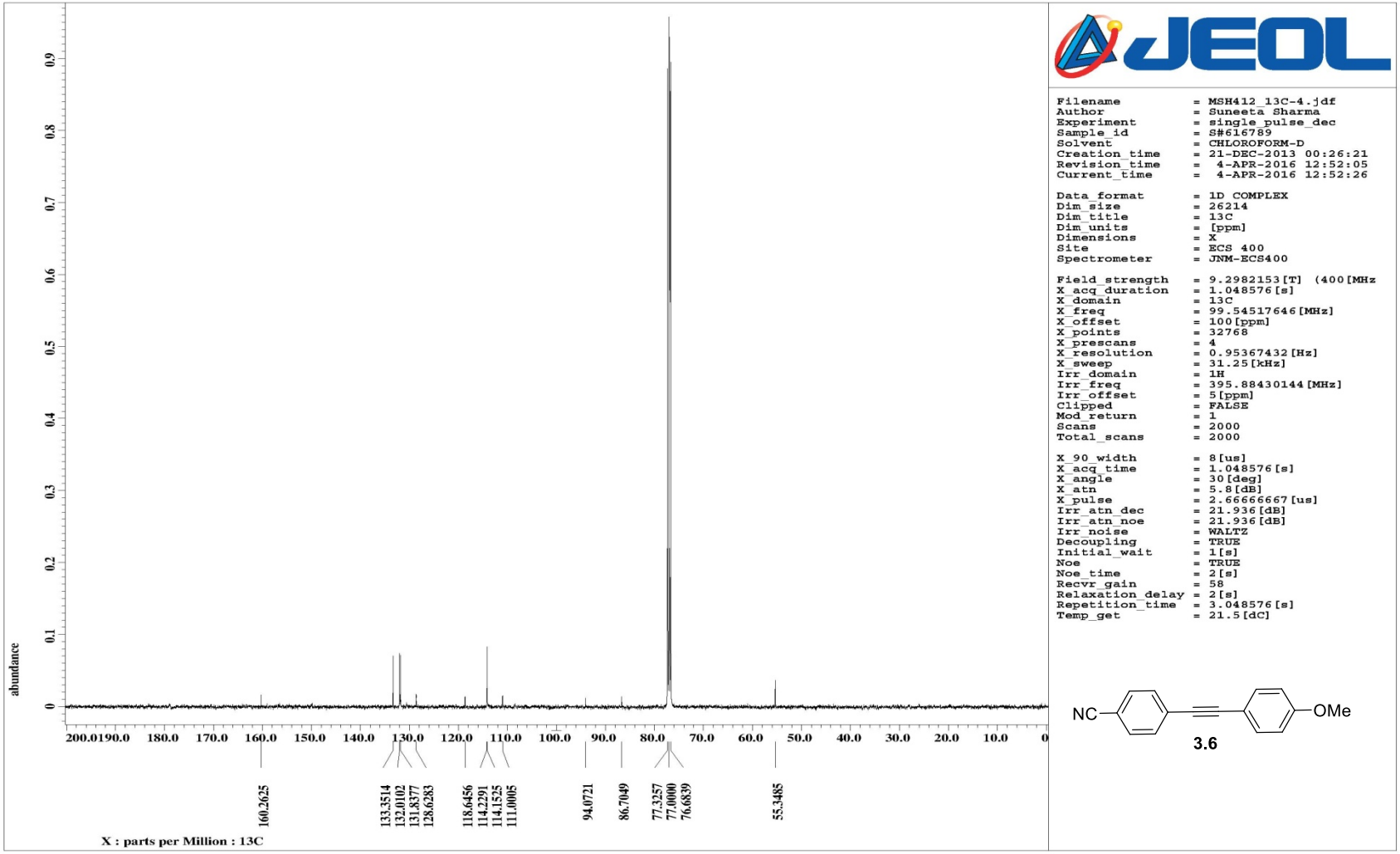
^{13}C NMR spectrum of 4-(phenylethynyl)benzonitrile **(3.5)**



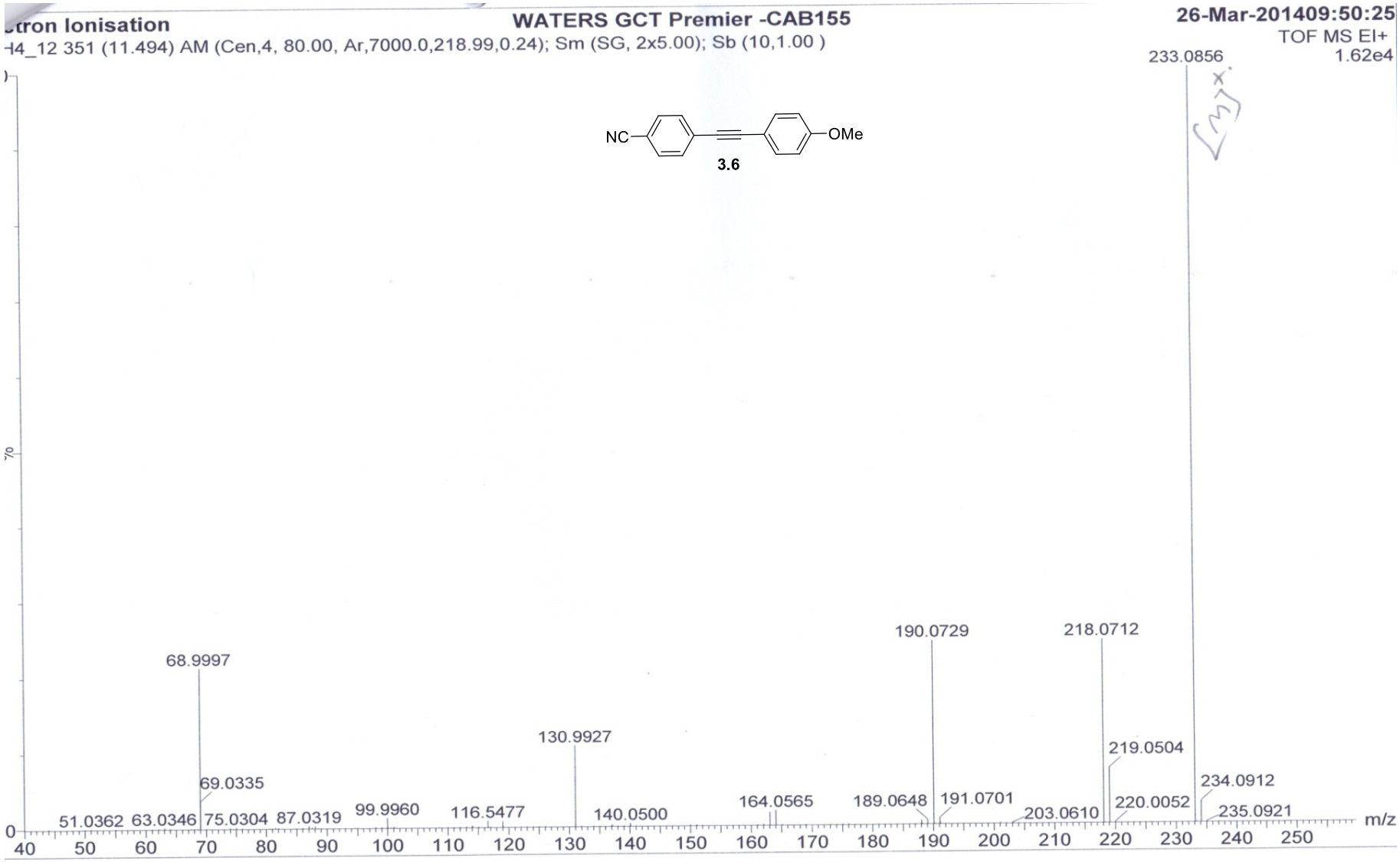
HRMS spectrum of 4-(phenylethynyl)benzonitrile **(3.5)**



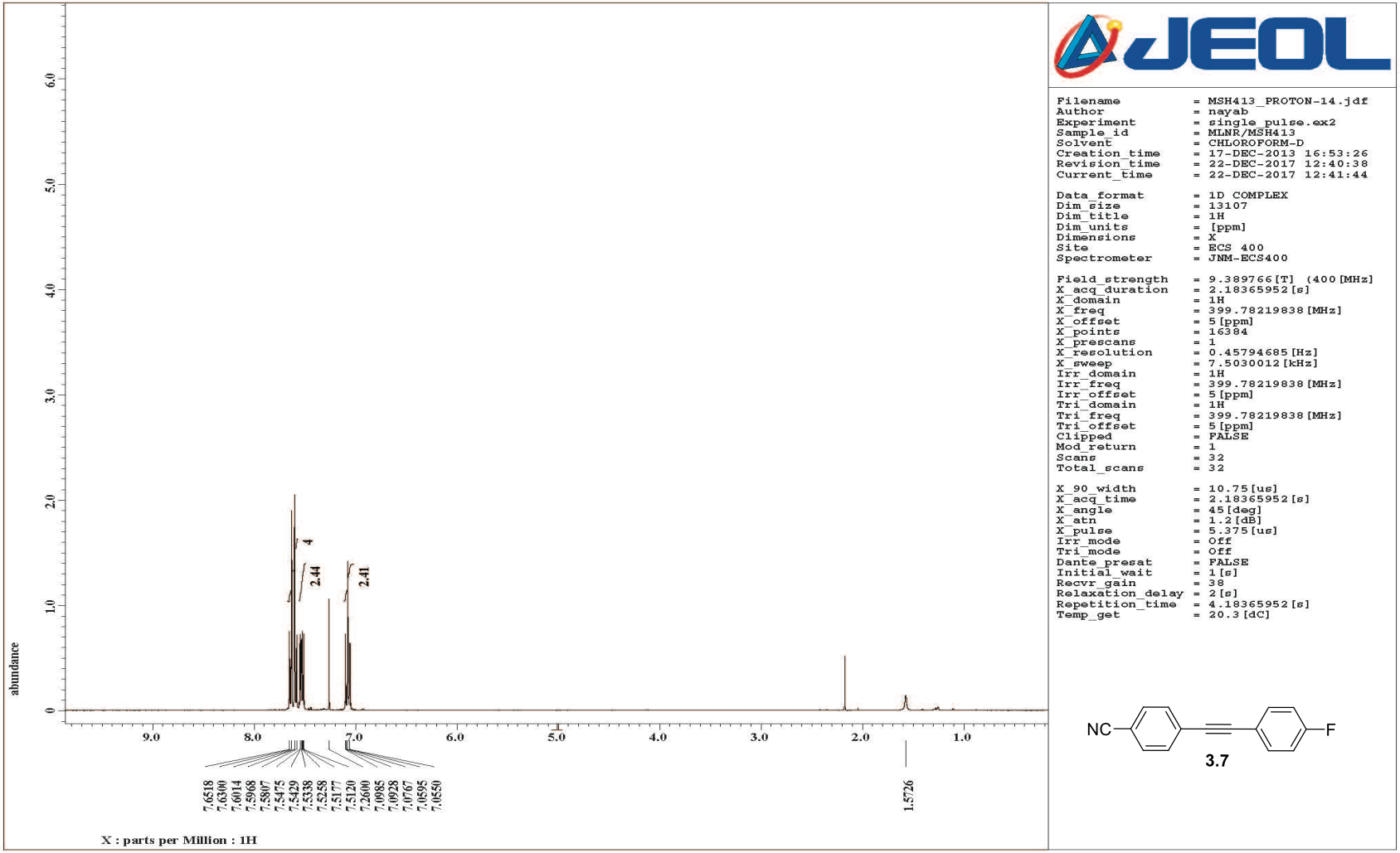
^{1}H NMR spectrum of 4-((4-methoxyphenyl)ethynyl)benzonitrile **(3.6)**



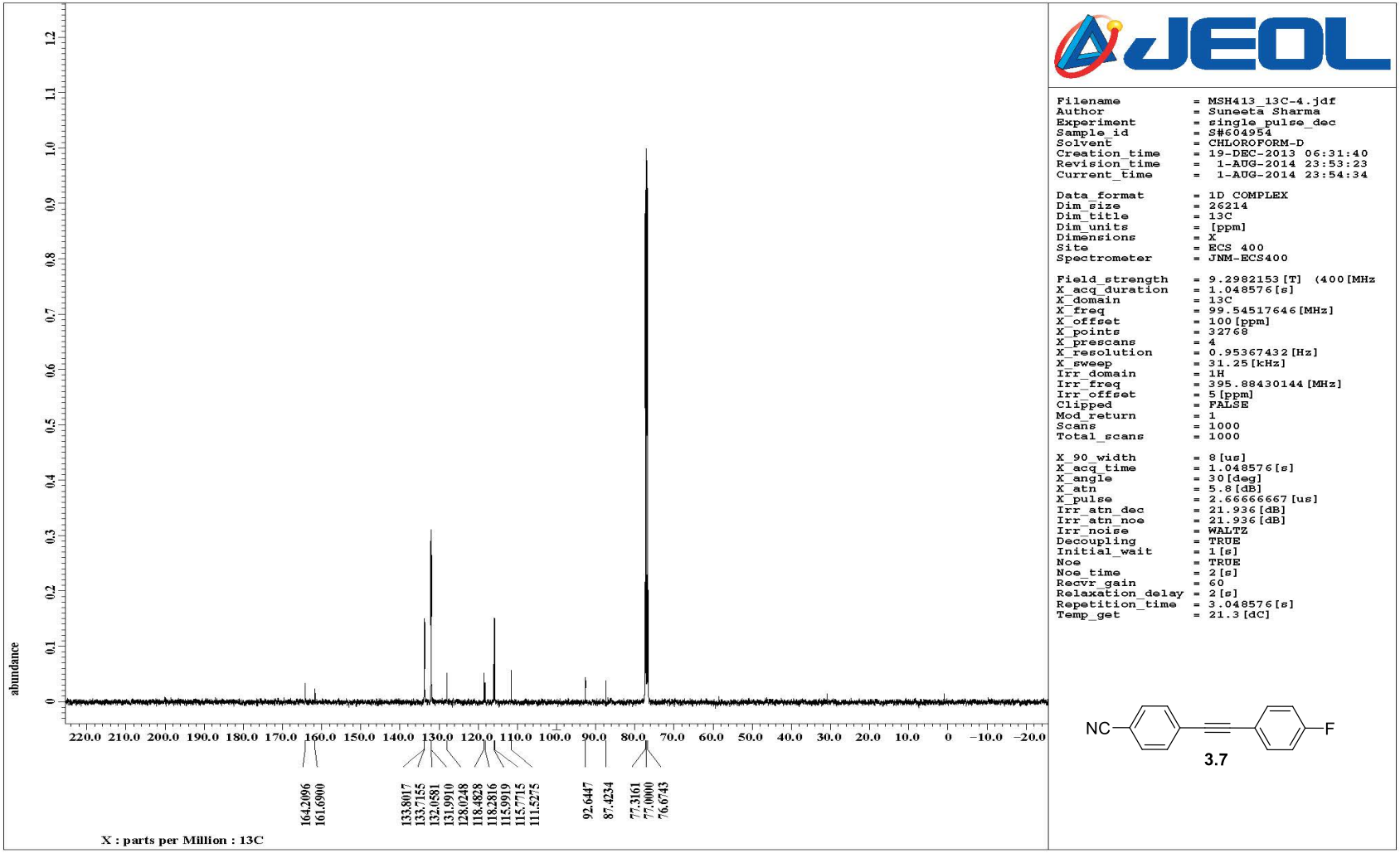
^{13}C NMR spectrum of 4-((4-methoxyphenyl)ethynyl)benzonitrile **(3.6)**



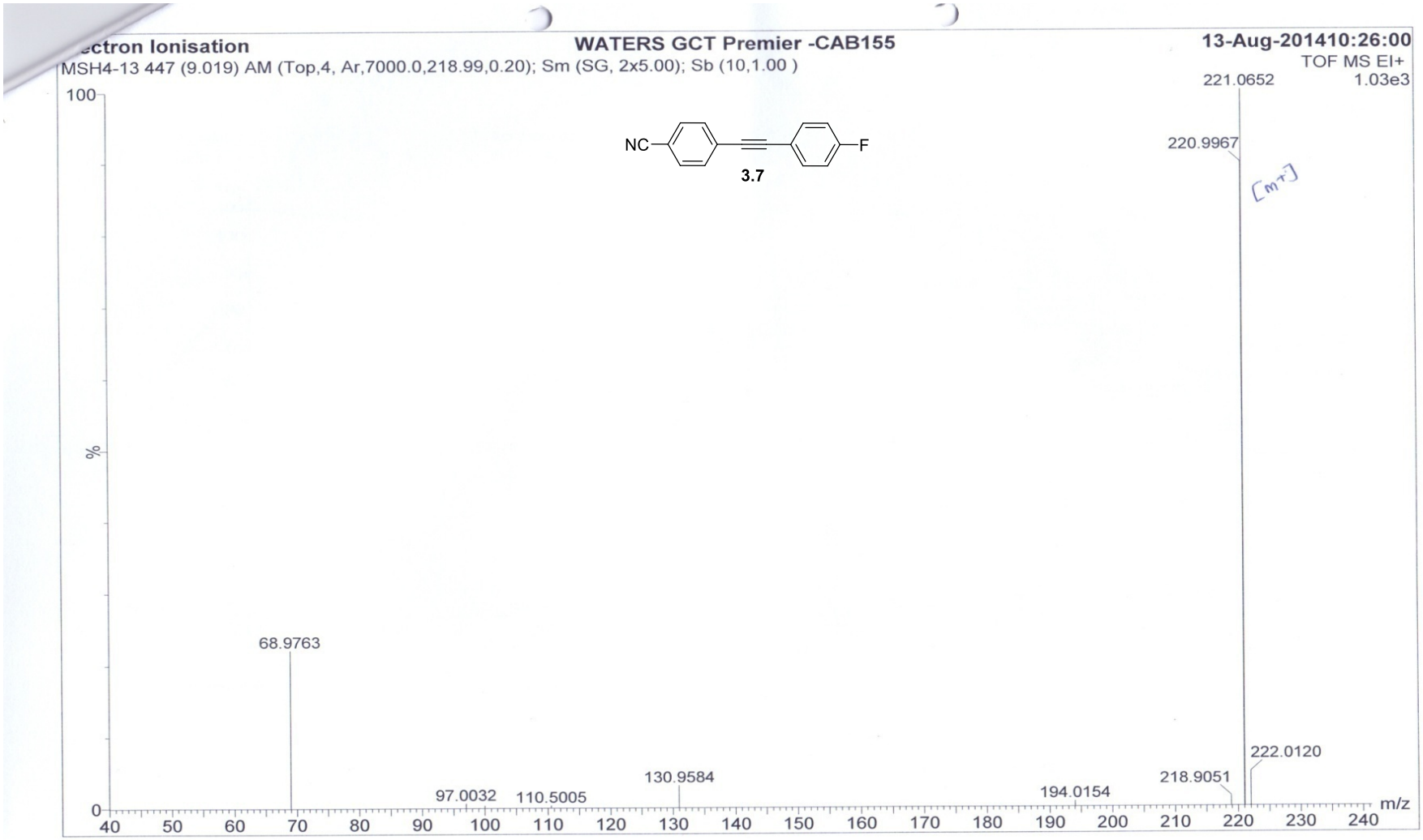
HRMS spectrum of 4-((4-methoxyphenyl)ethynyl)benzonitrile **(3.6)**



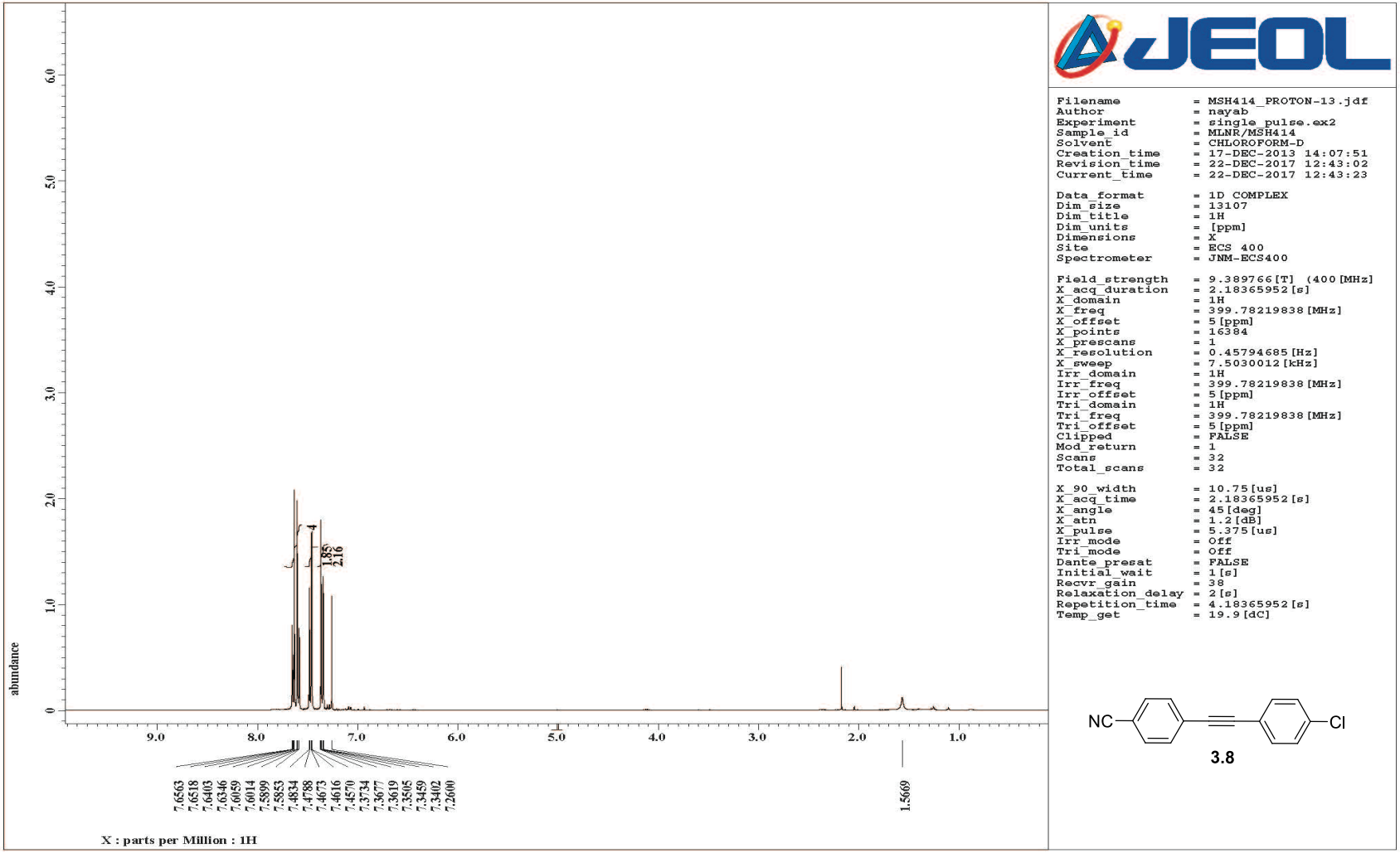
^{1}H NMR spectrum of 4-((4-fluorophenyl)ethynyl)benzonitrile **(3.7)**



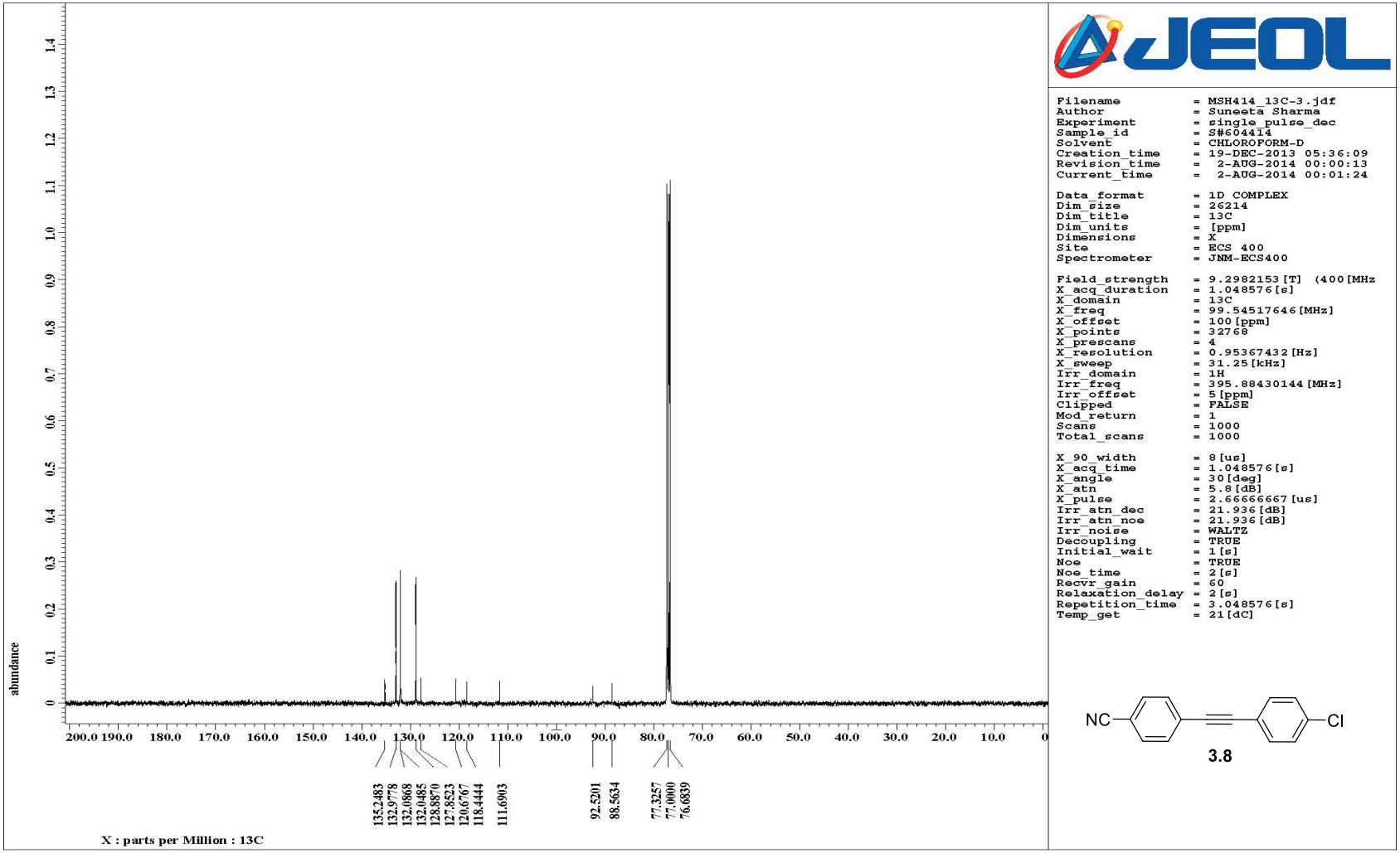
^{13}C NMR spectrum of 4-((4-fluorophenyl)ethynyl)benzonitrile **(3.7)**



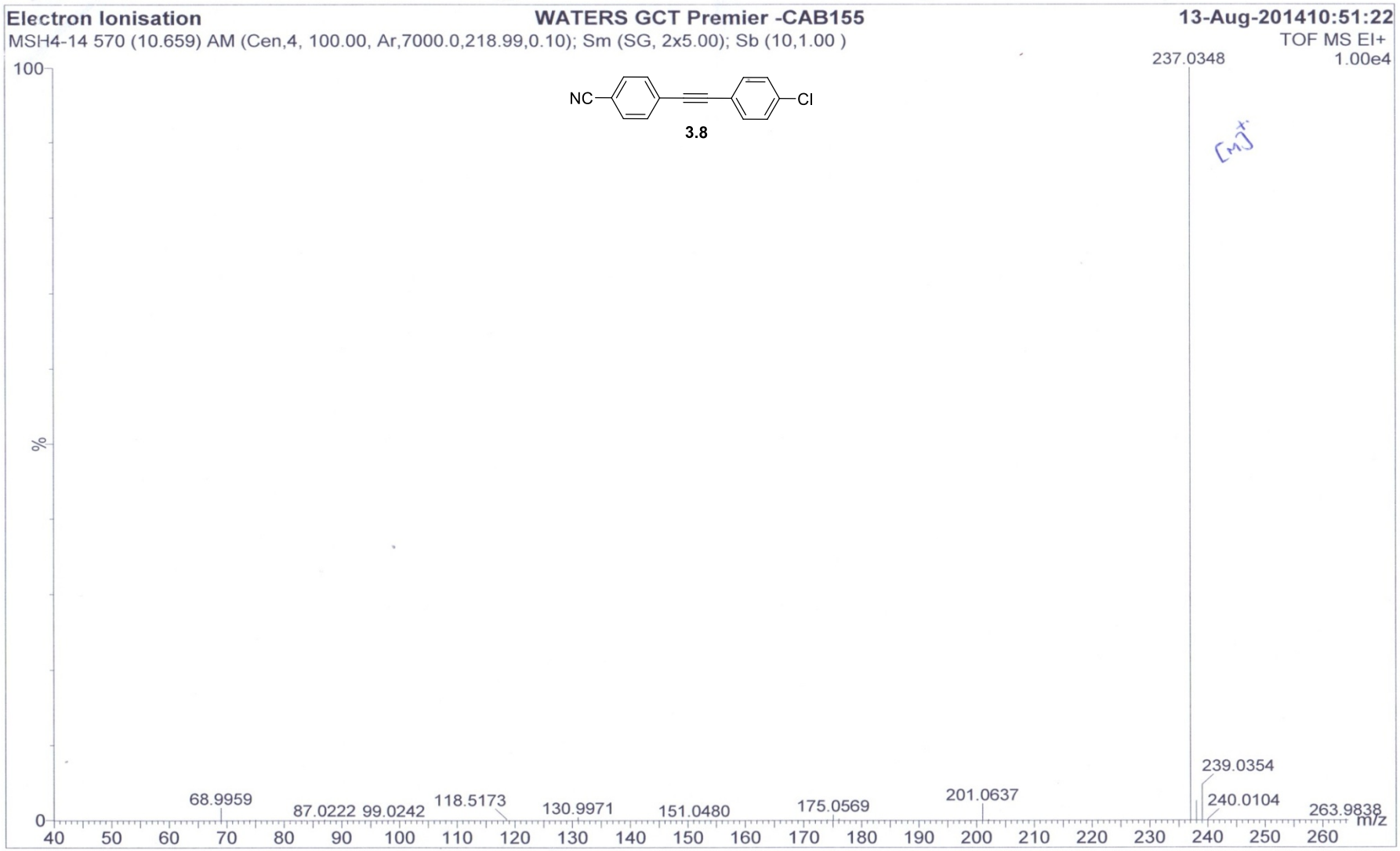
HRMS spectrum of 4-((4-fluorophenyl)ethynyl)benzonitrile **(3.7)**



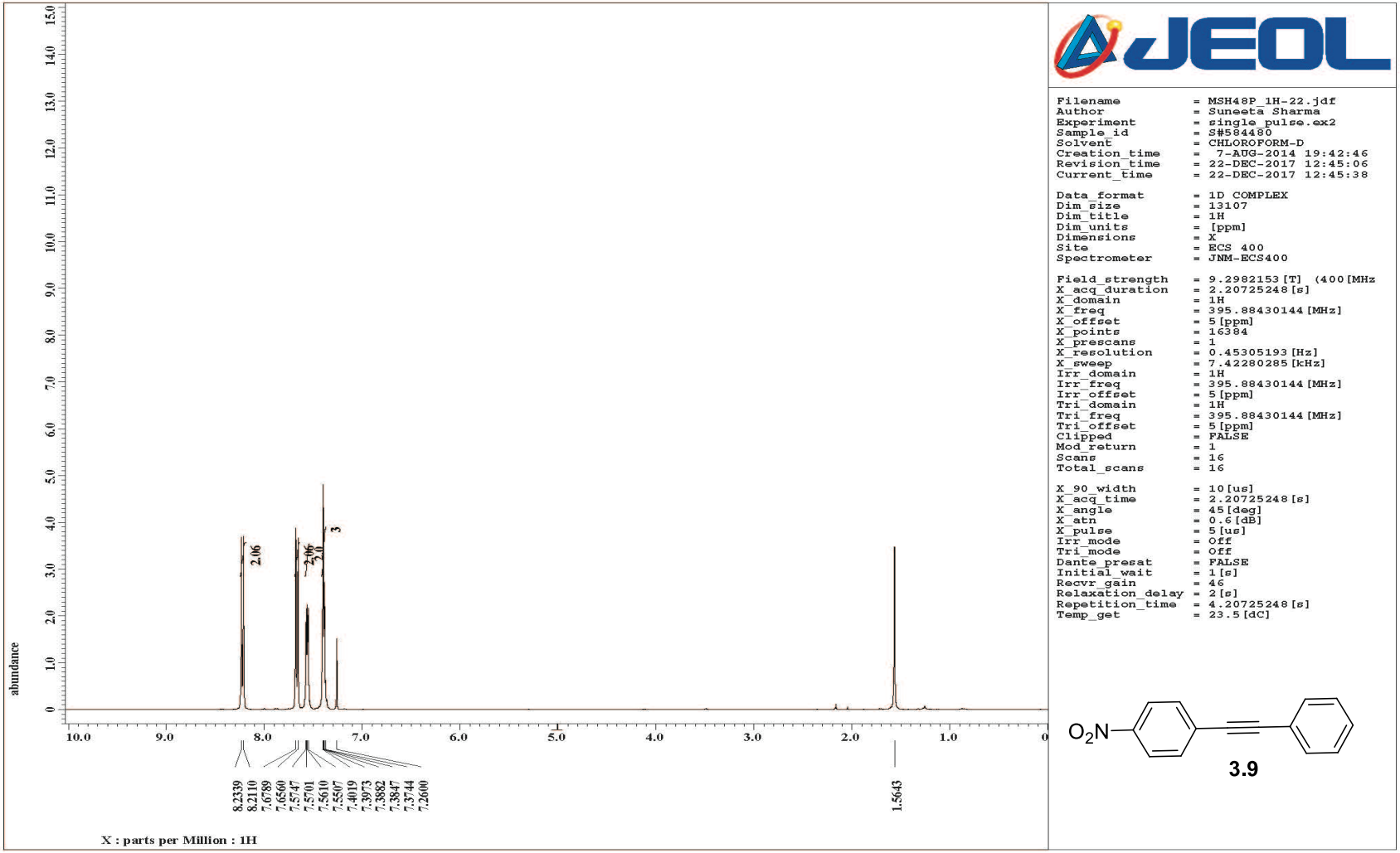
^{1}H NMR spectrum of 4-((4-chlorophenyl)ethynyl)benzonitrile **(3.8)**



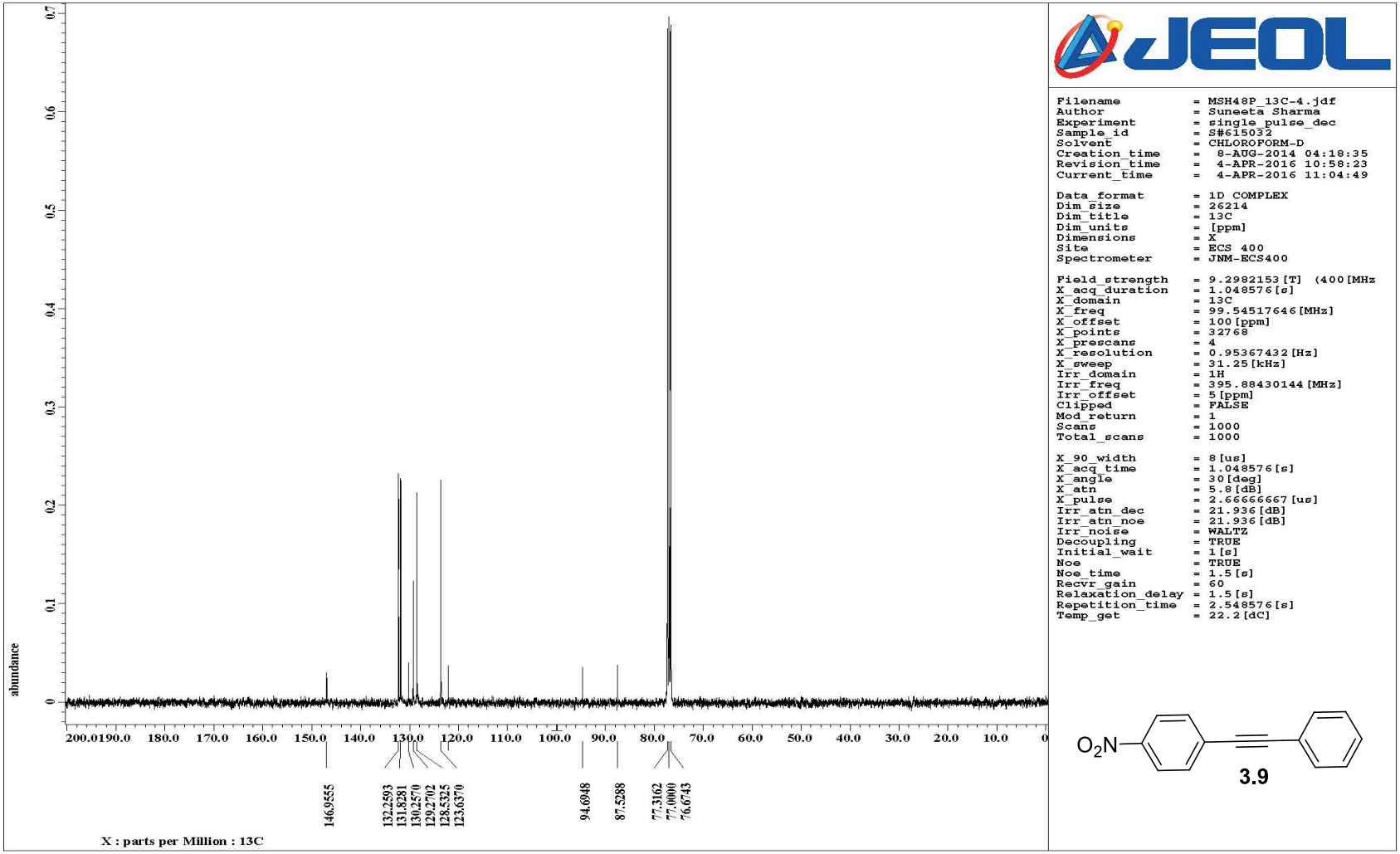
^{13}C NMR spectrum of 4-((4-chlorophenyl)ethynyl)benzonitrile **(3.8)**



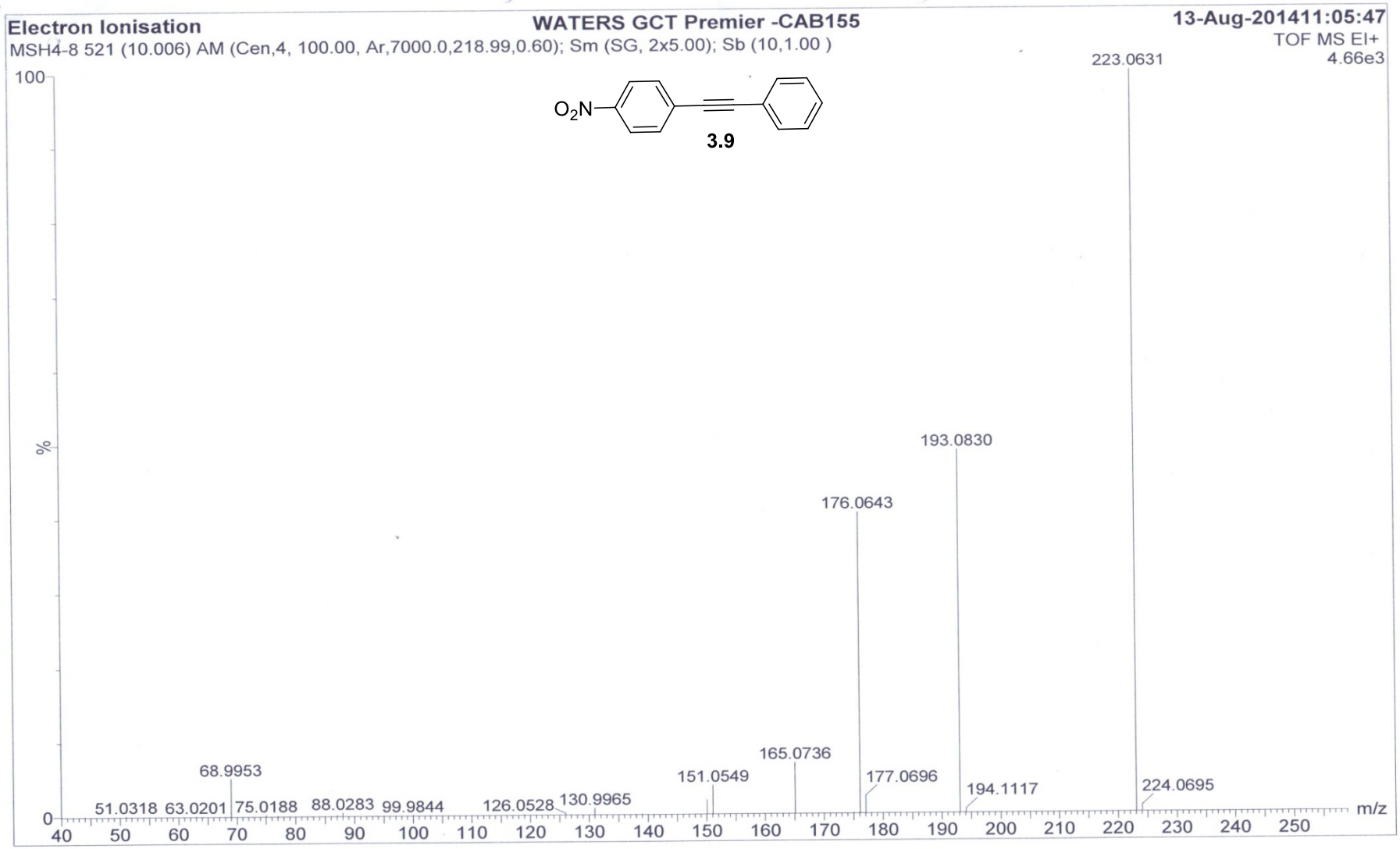
HRMS spectrum of 4-((4-chlorophenyl)ethynyl)benzonitrile **(3.8)**



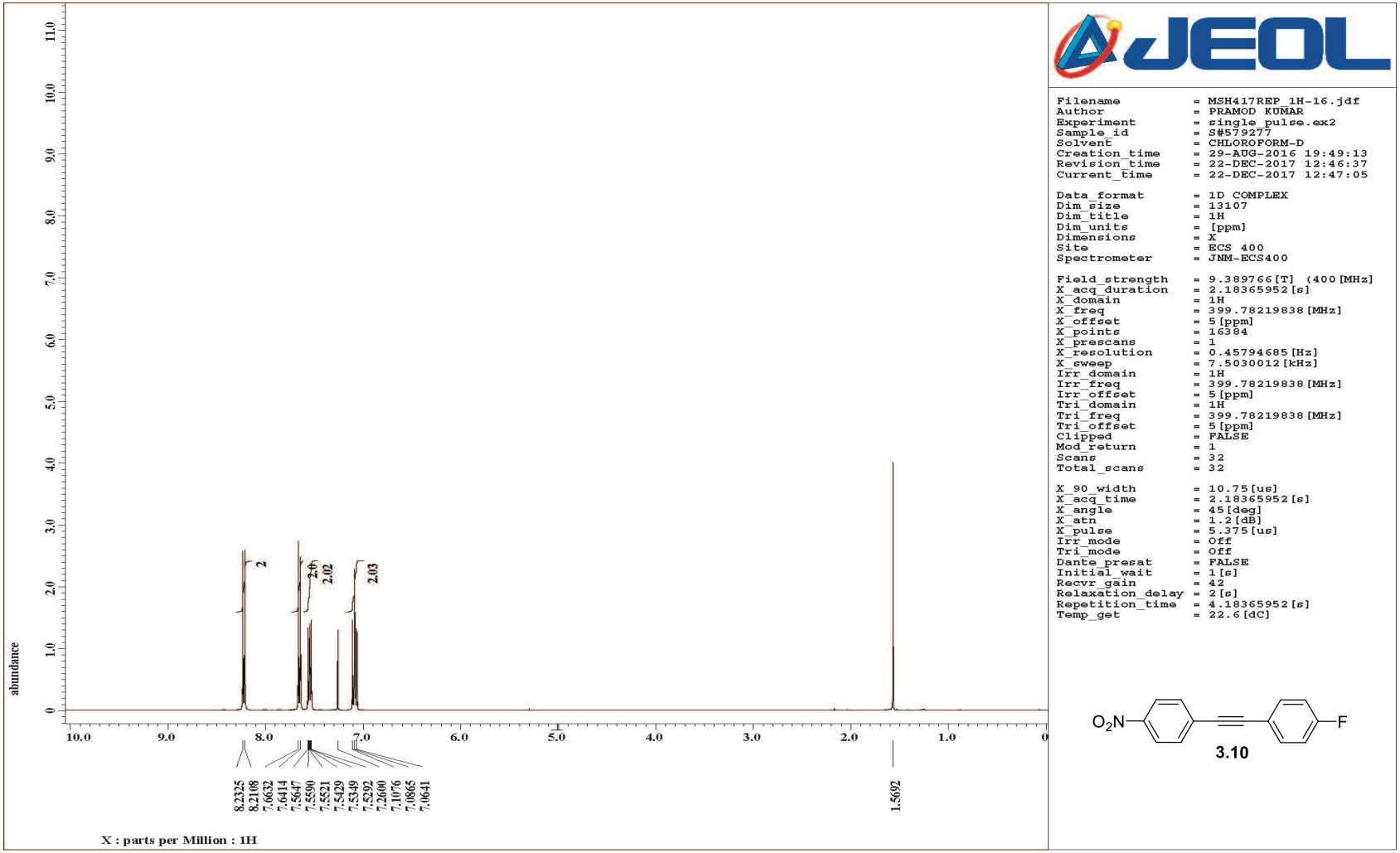
^{1}H NMR spectrum of 1-nitro-4-(phenylethynyl)benzene **(3.9)**



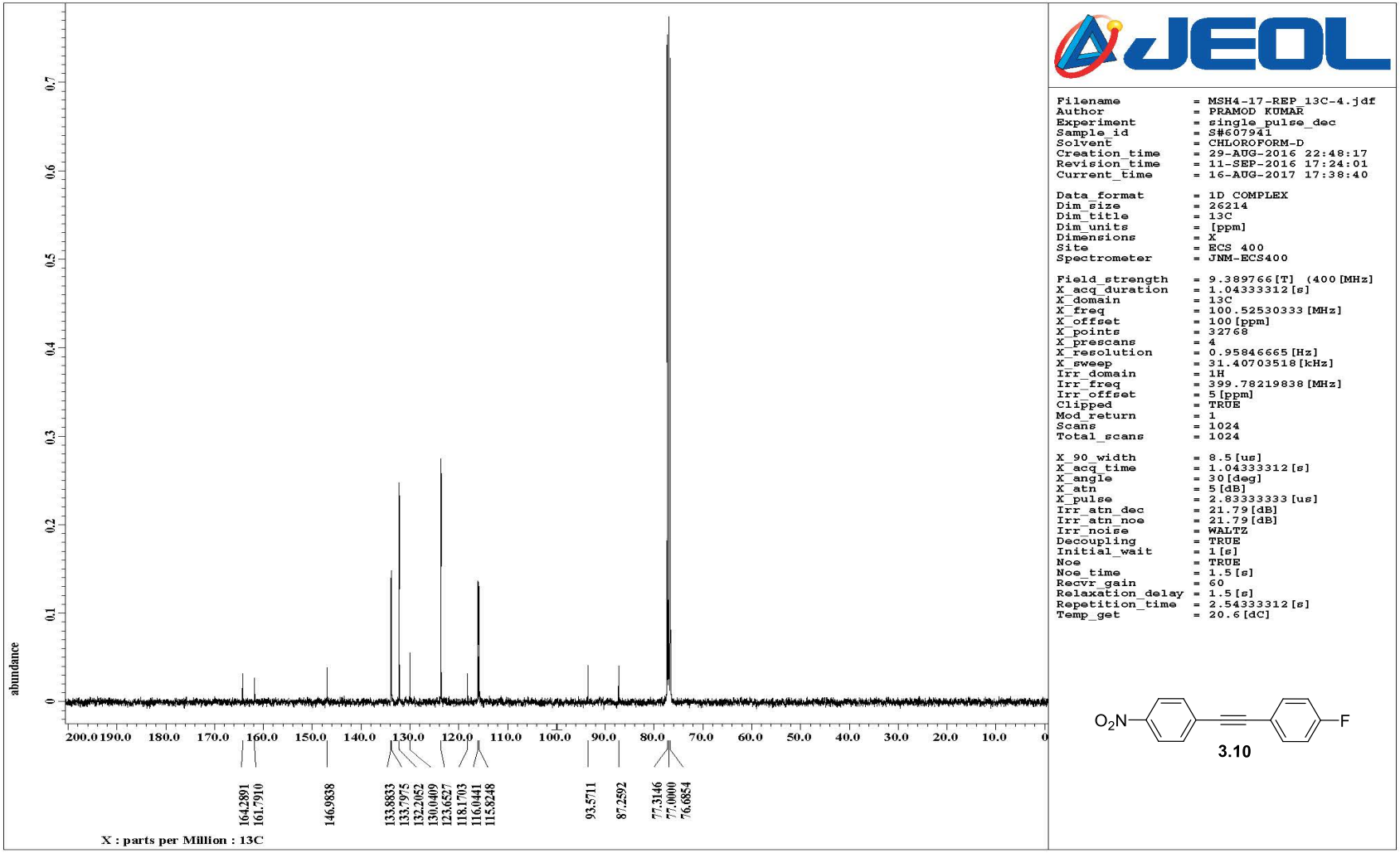
^{13}C NMR spectrum of 1-nitro-4-(phenylethynyl)benzene **(3.9)**



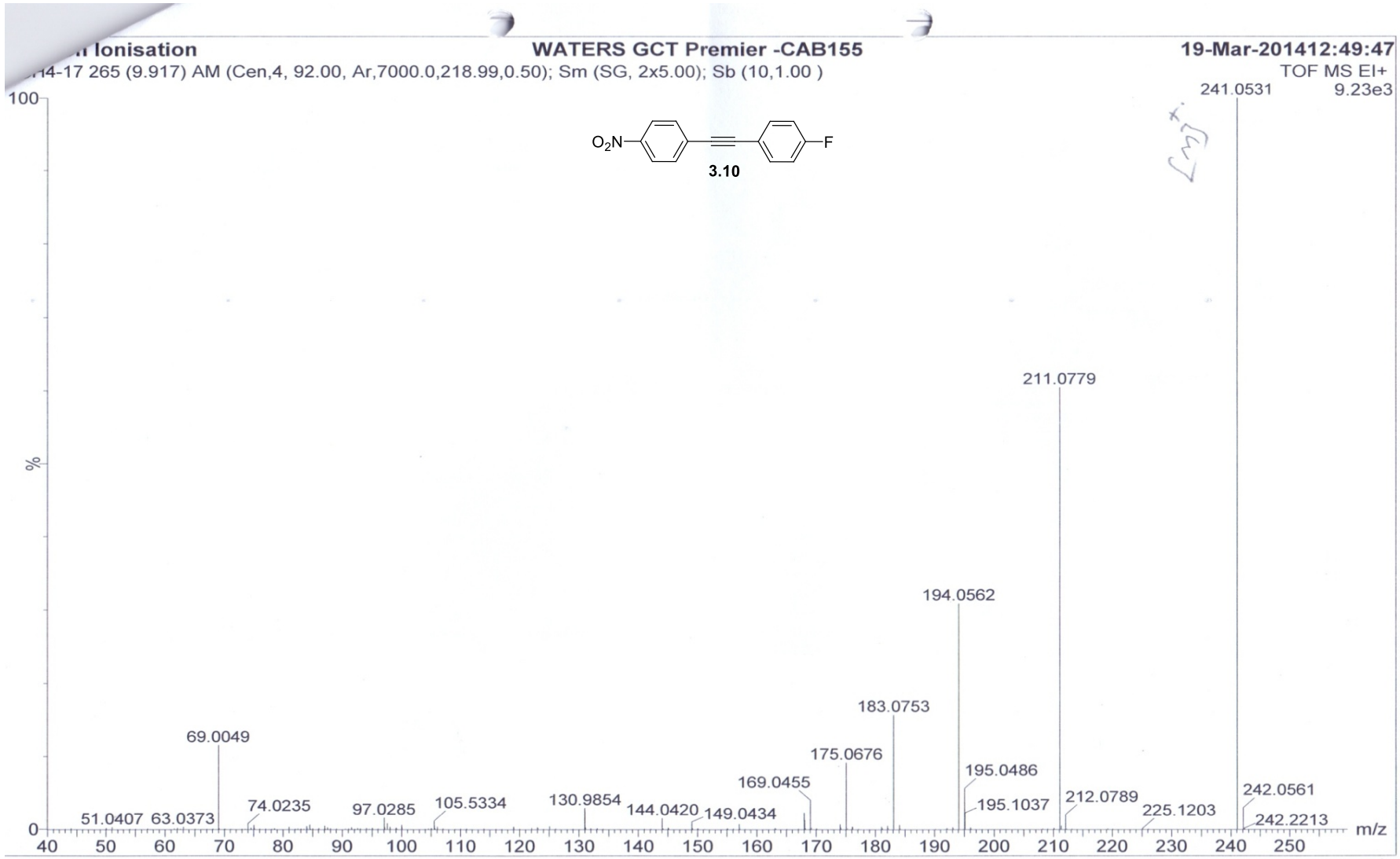
HRMS spectrum of 1-nitro-4-(phenylethynyl)benzene **(3.9)**



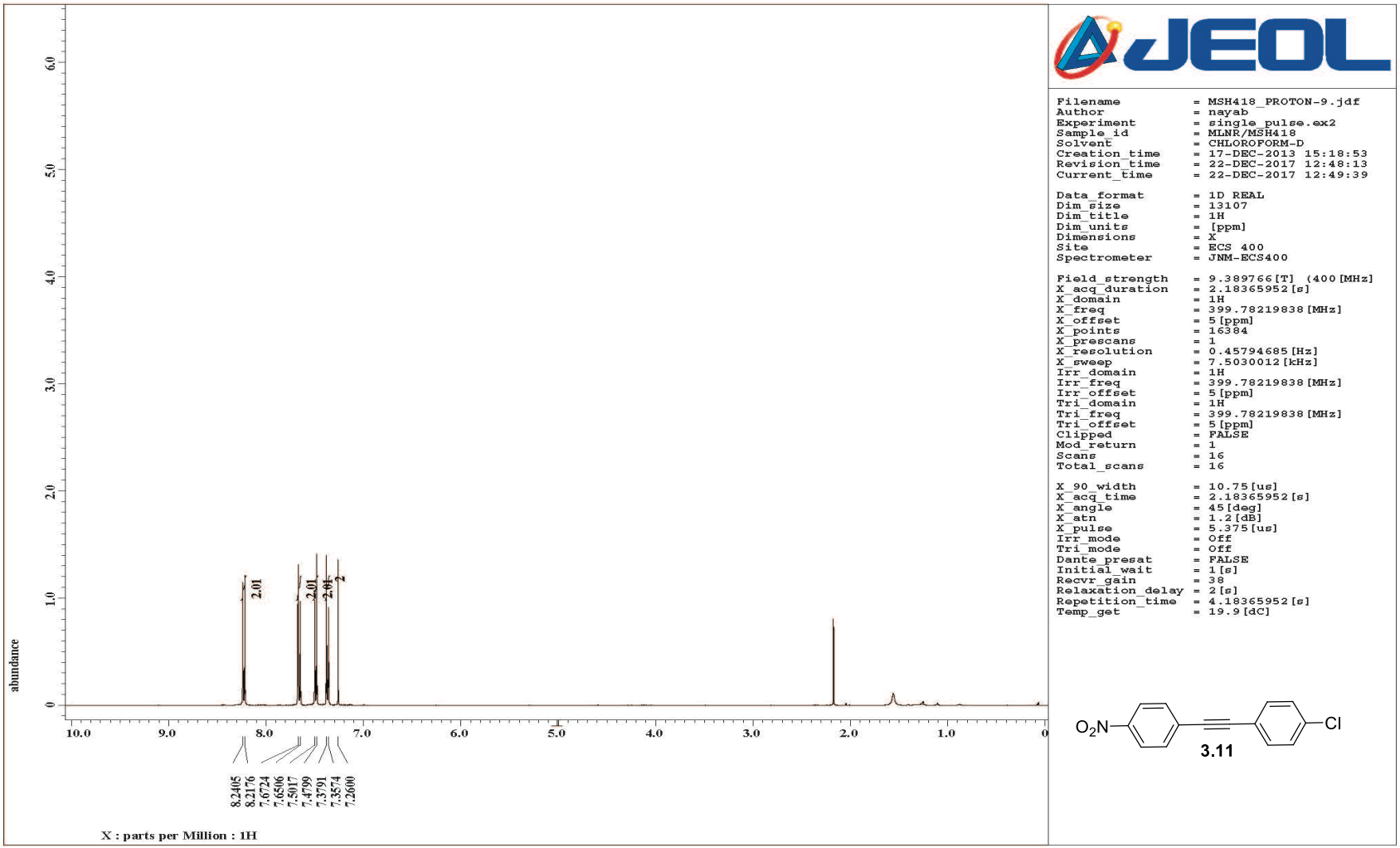
^{1}H NMR spectrum of 1-fluoro-4-((4-nitrophenyl)ethynyl)benzene **(3.10)**



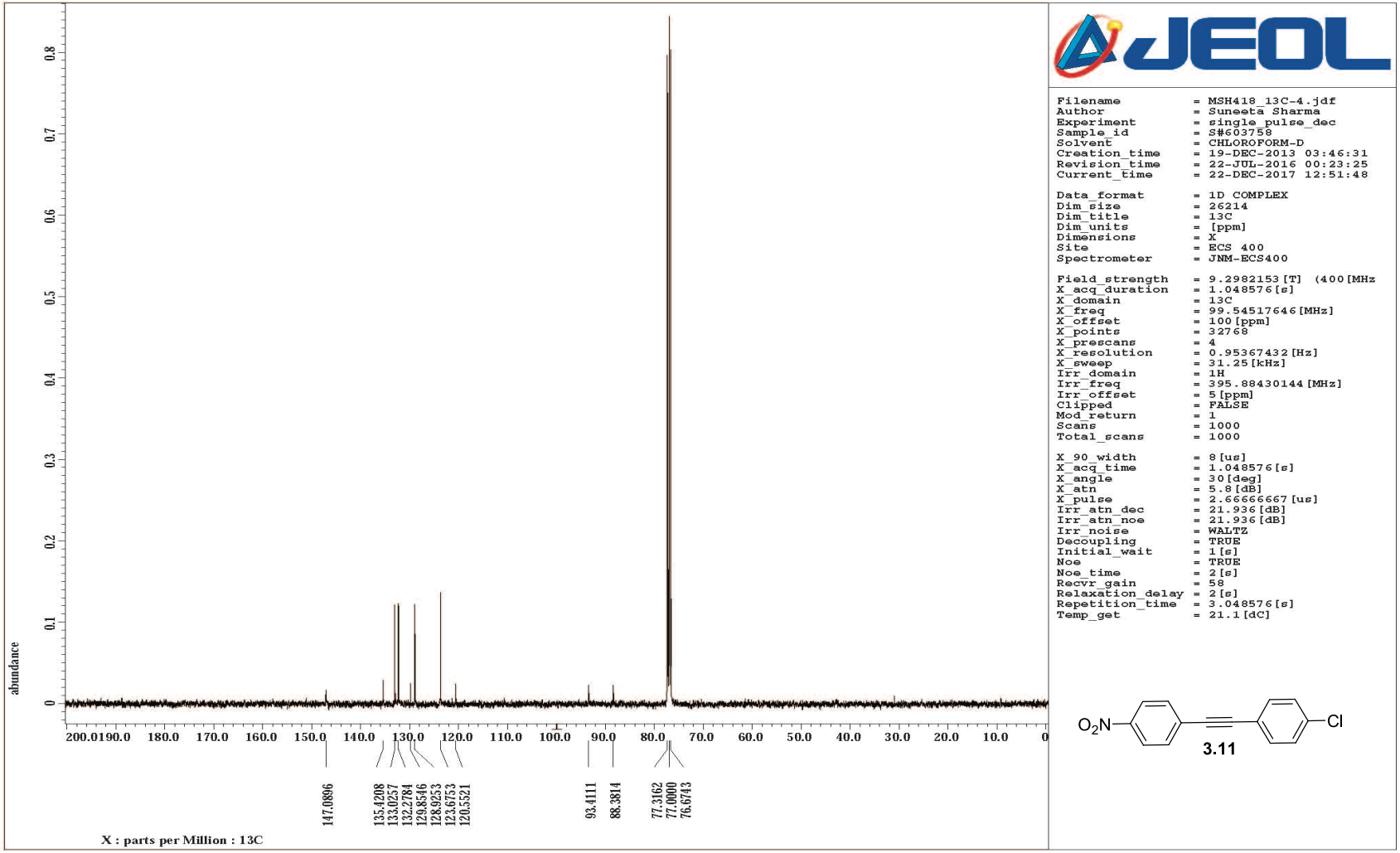
^{13}C NMR spectrum of 1-fluoro-4-((4-nitrophenyl)ethynyl)benzene **(3.10)**



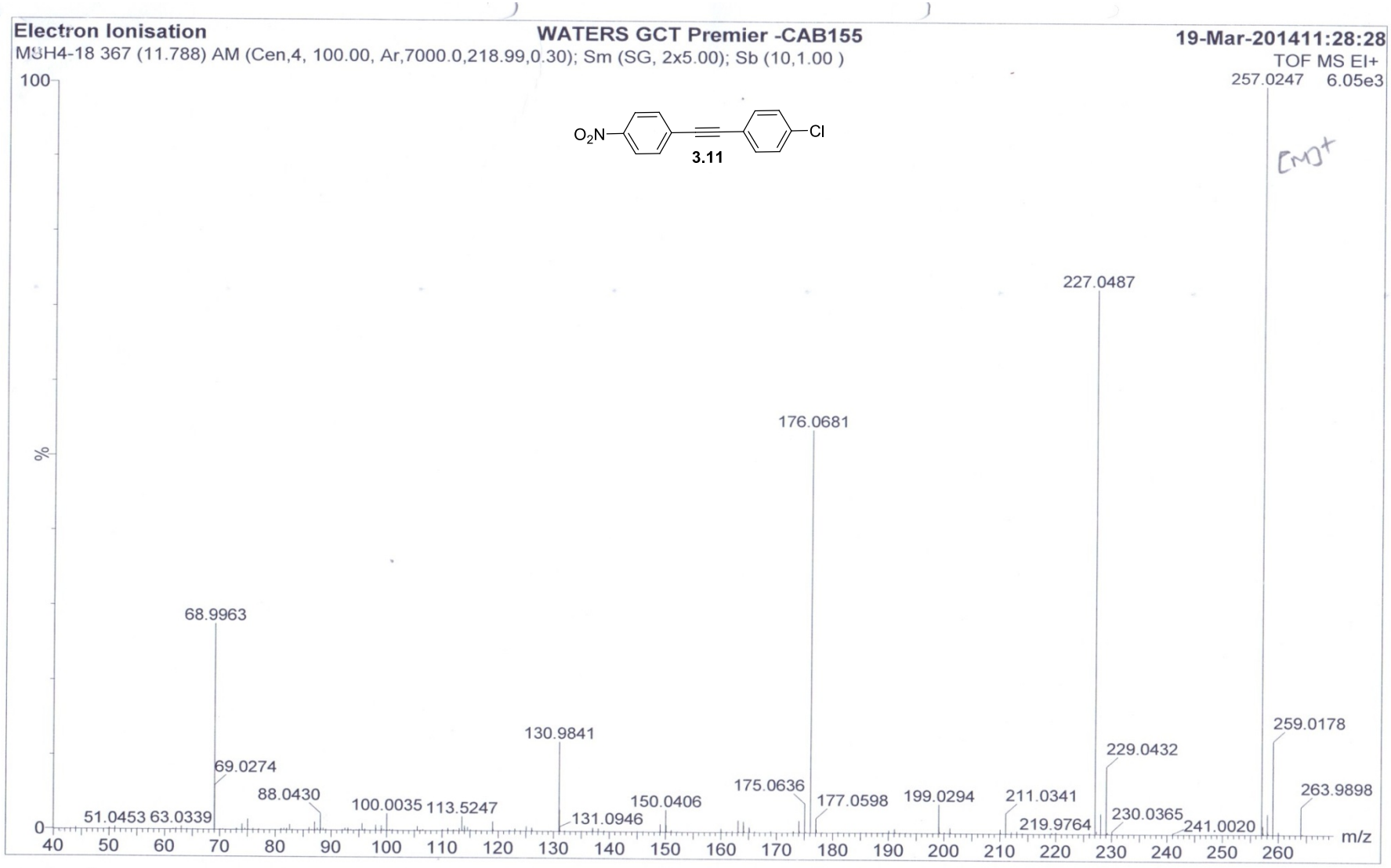
HRMS spectrum of 1-fluoro-4-((4-nitrophenyl)ethynyl)benzene **(3.10)**



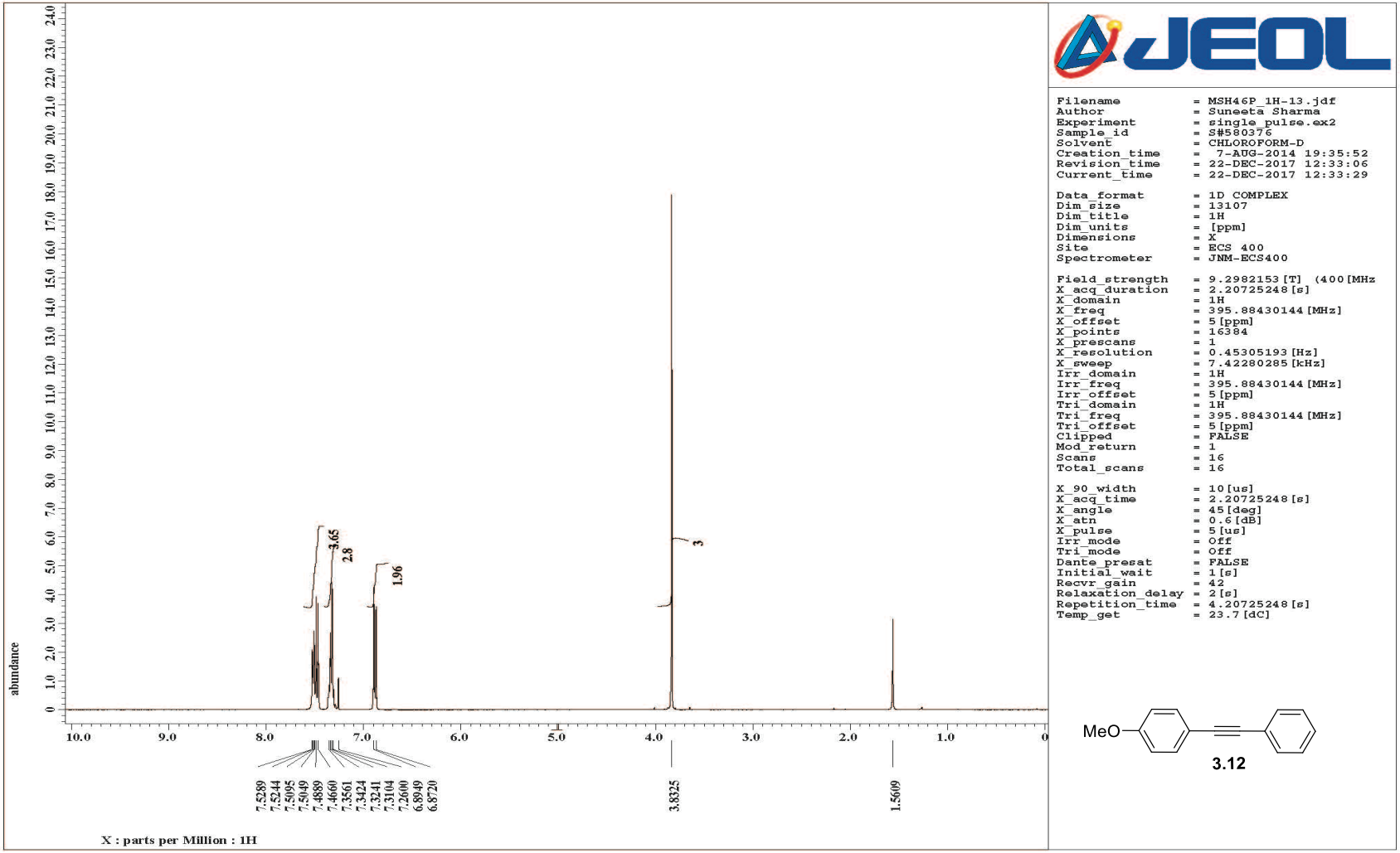
^{1}H NMR spectrum of 1-chloro-4-((4-nitrophenyl)ethynyl)benzene **(3.11)**



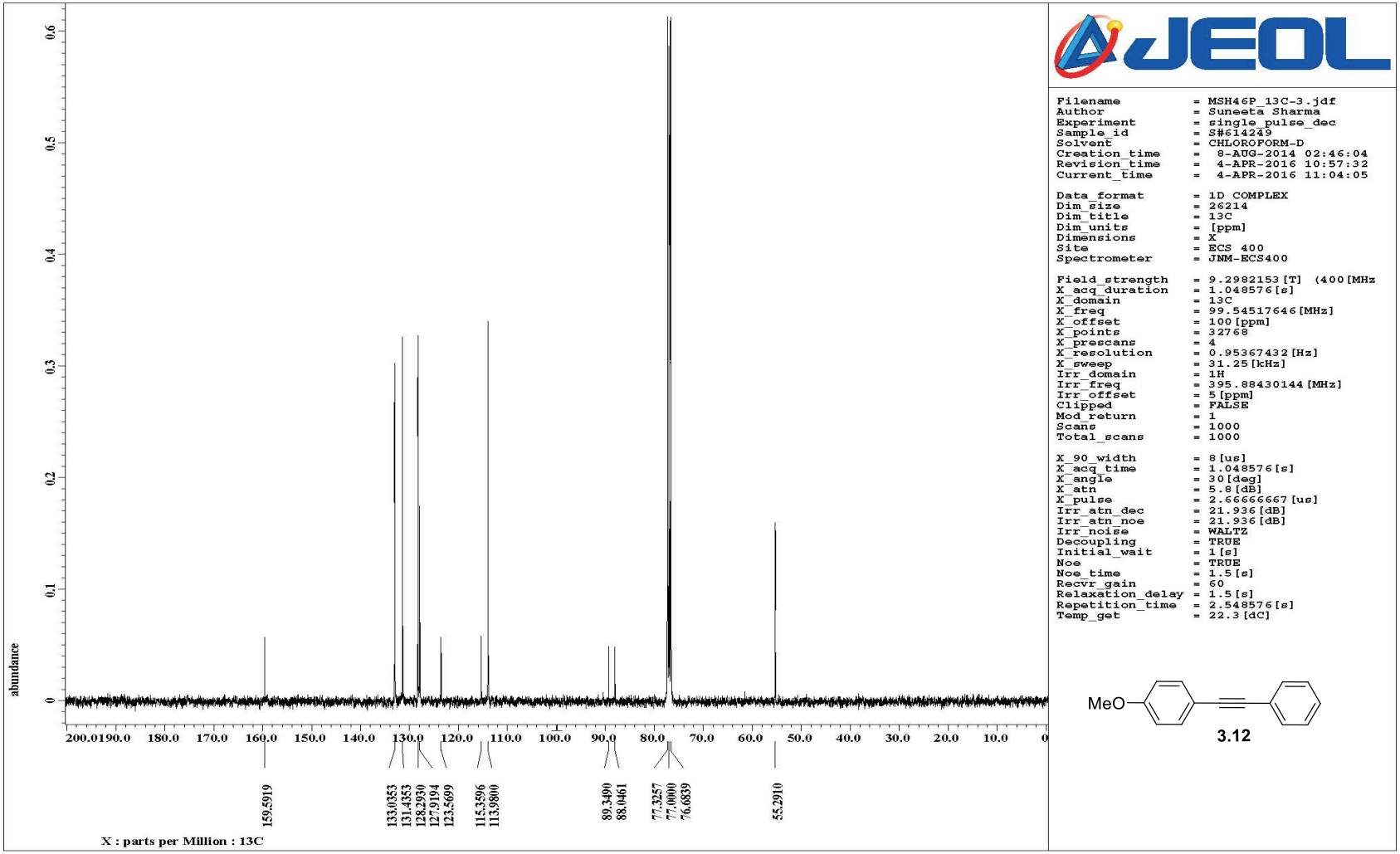
^{13}C NMR spectrum of 1-chloro-4-((4-nitrophenyl)ethynyl)benzene **(3.11)**



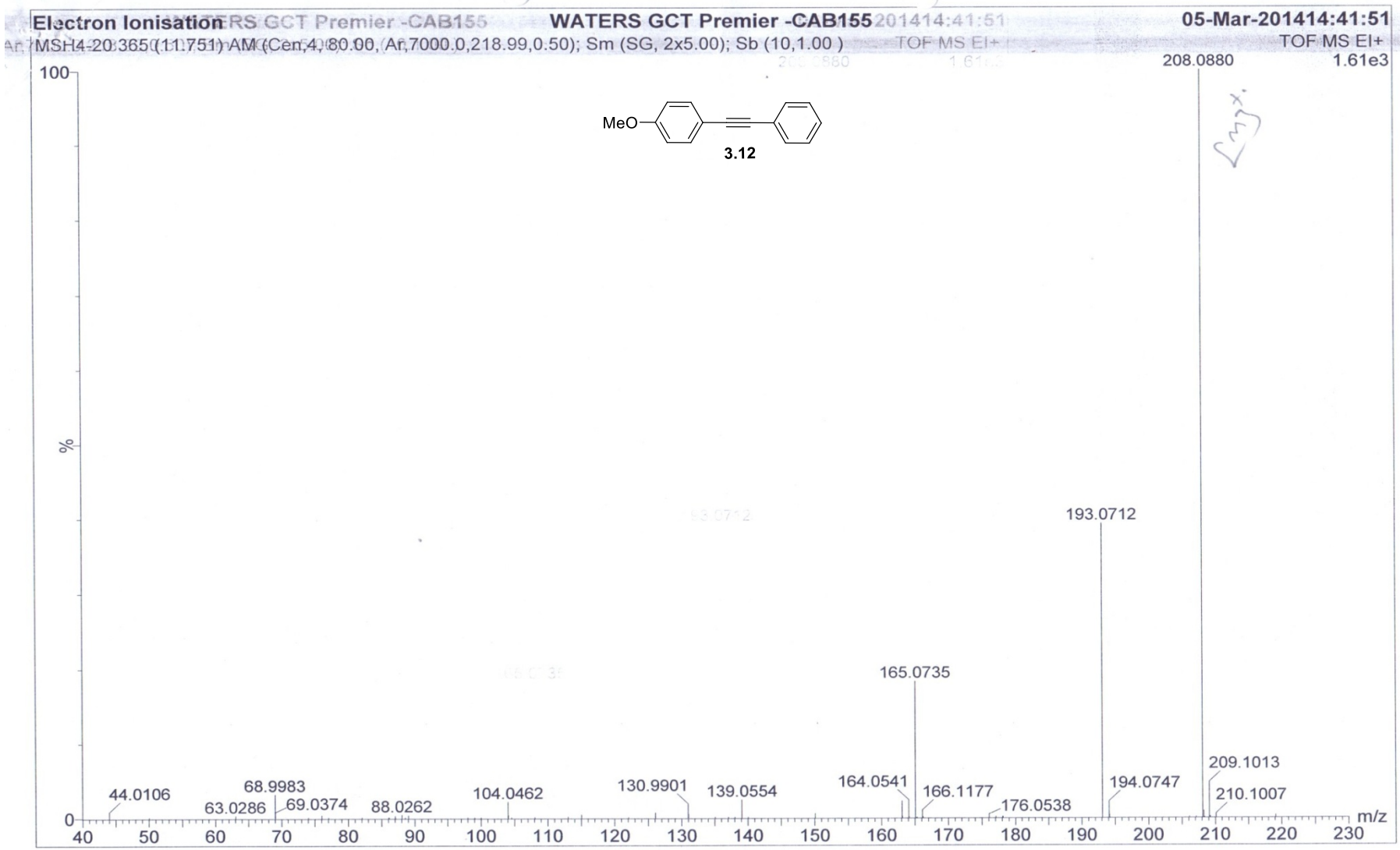
HRMS spectrum of 1-chloro-4-((4-nitrophenyl)ethynyl)benzene **(3.11)**



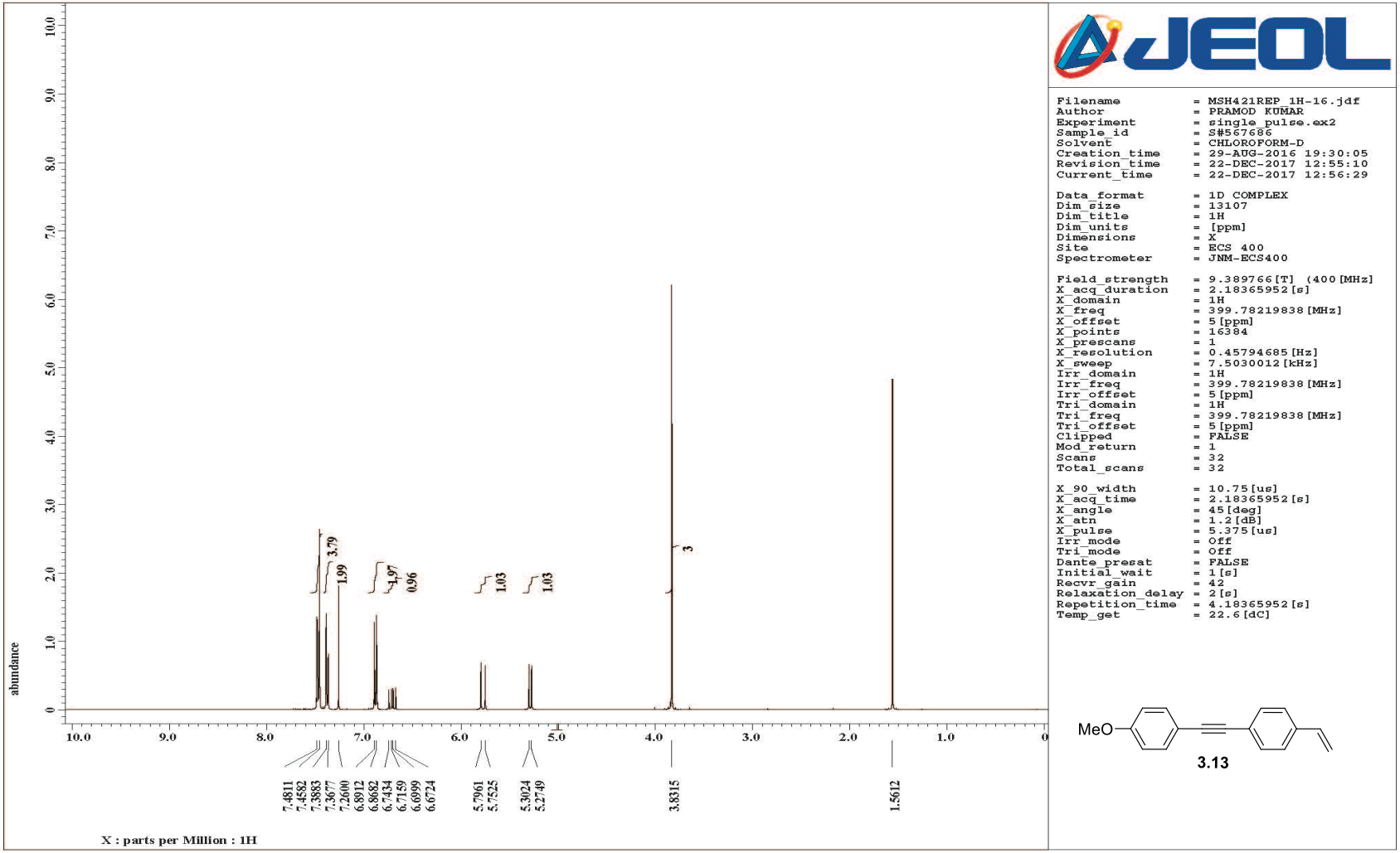
^{1}H NMR spectrum of 1-methoxy-4-(phenylethynyl)benzene **(3.12)**



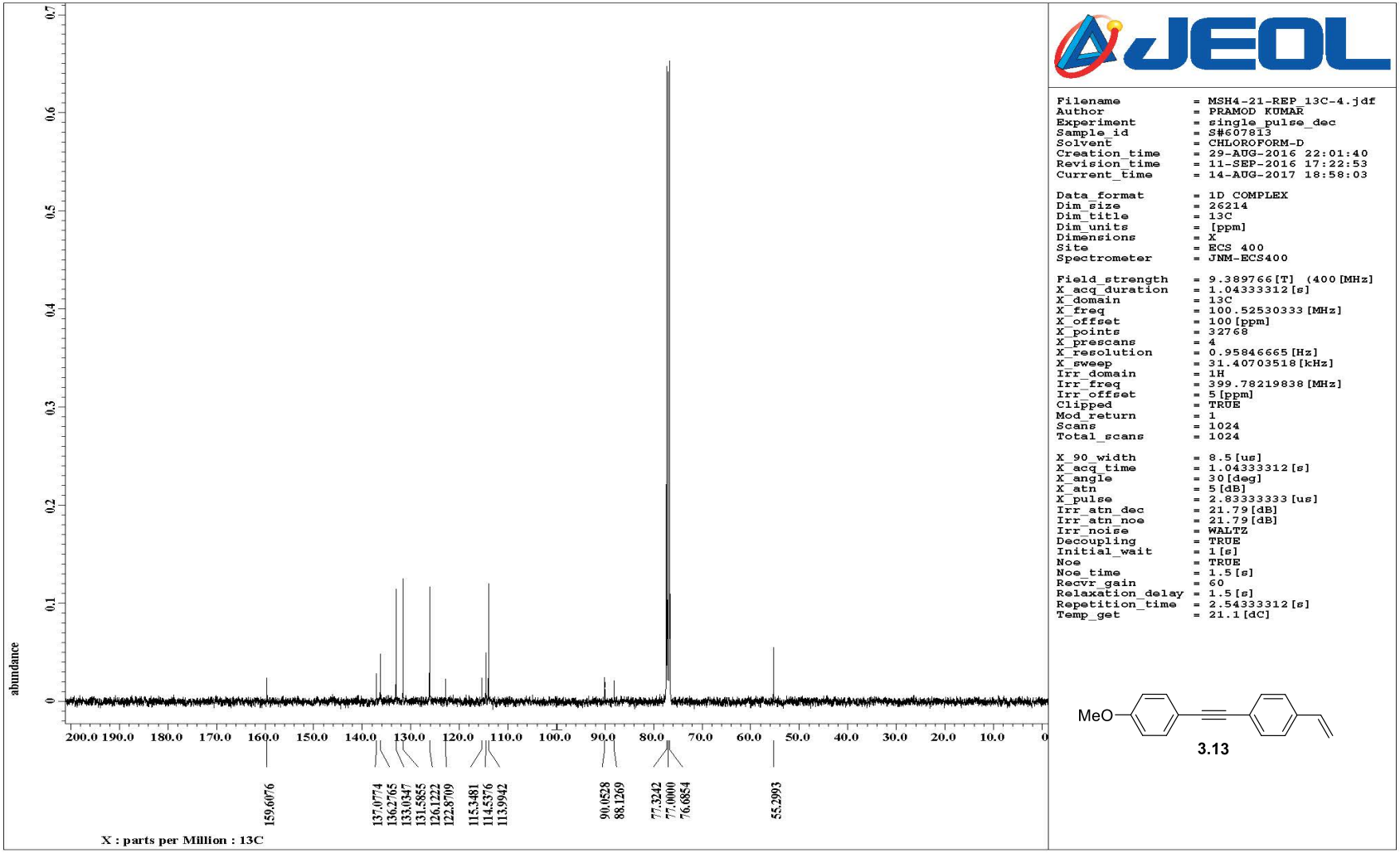
^{13}C NMR spectrum of 1-methoxy-4-(phenylethynyl)benzene **(3.12)**



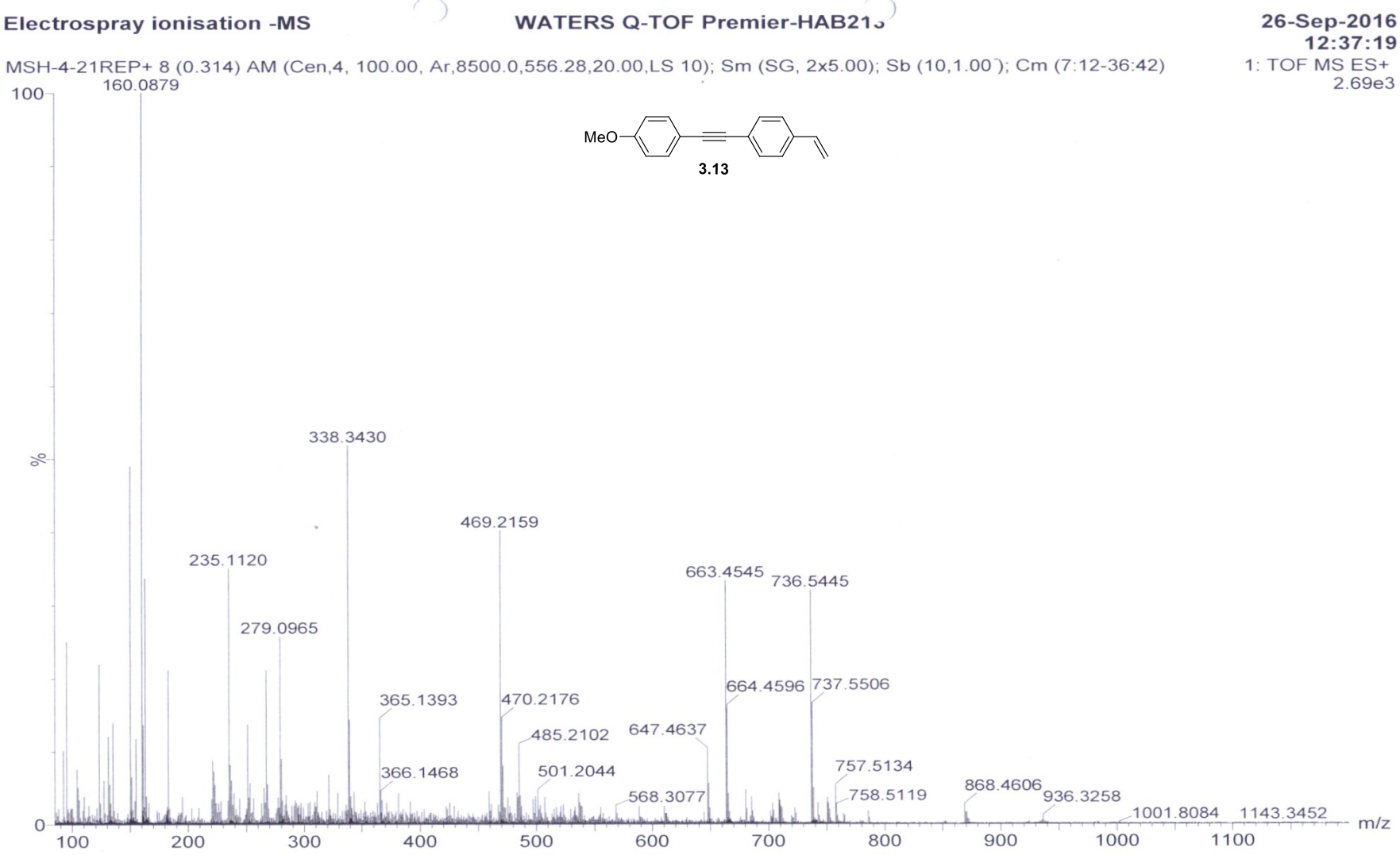
HRMS spectrum of 1-methoxy-4-(phenylethynyl)benzene **(3.12)**



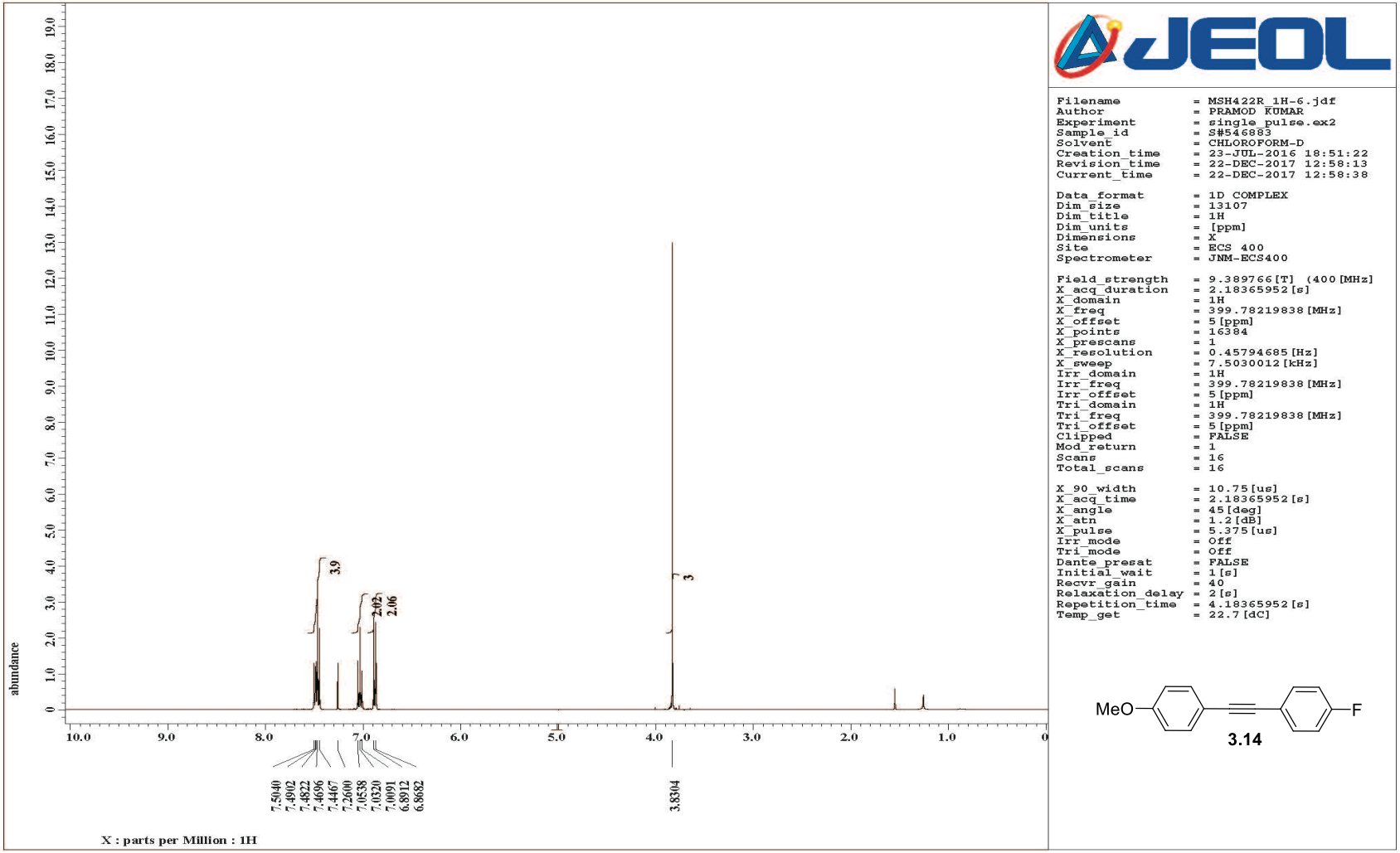
^{1}H NMR spectrum of 1-methoxy-4-((4-vinylphenyl)ethynyl)benzene **(3.13)**



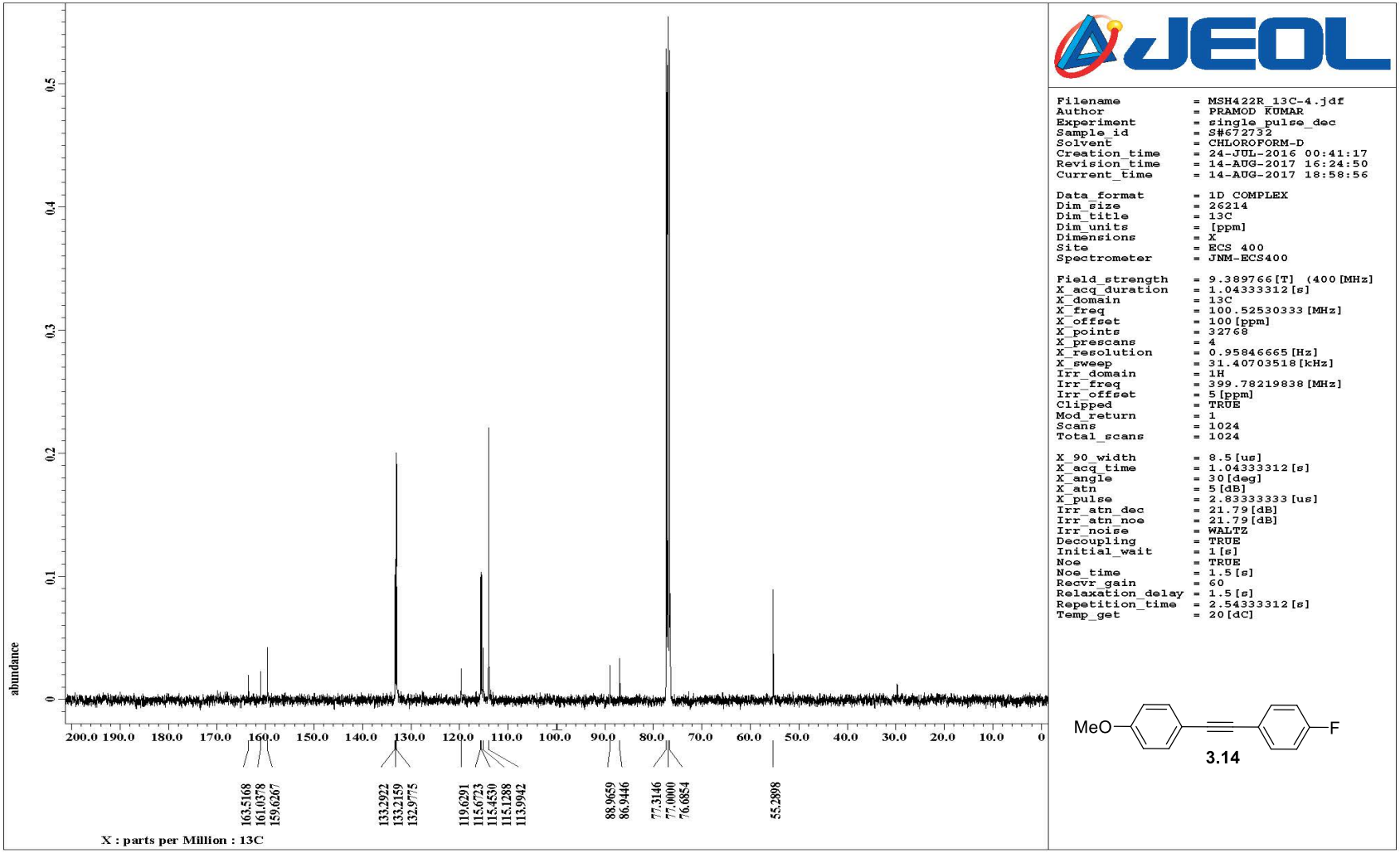
^{13}C NMR spectrum of 1-methoxy-4-((4-vinylphenyl)ethynyl)benzene **(3.13)**



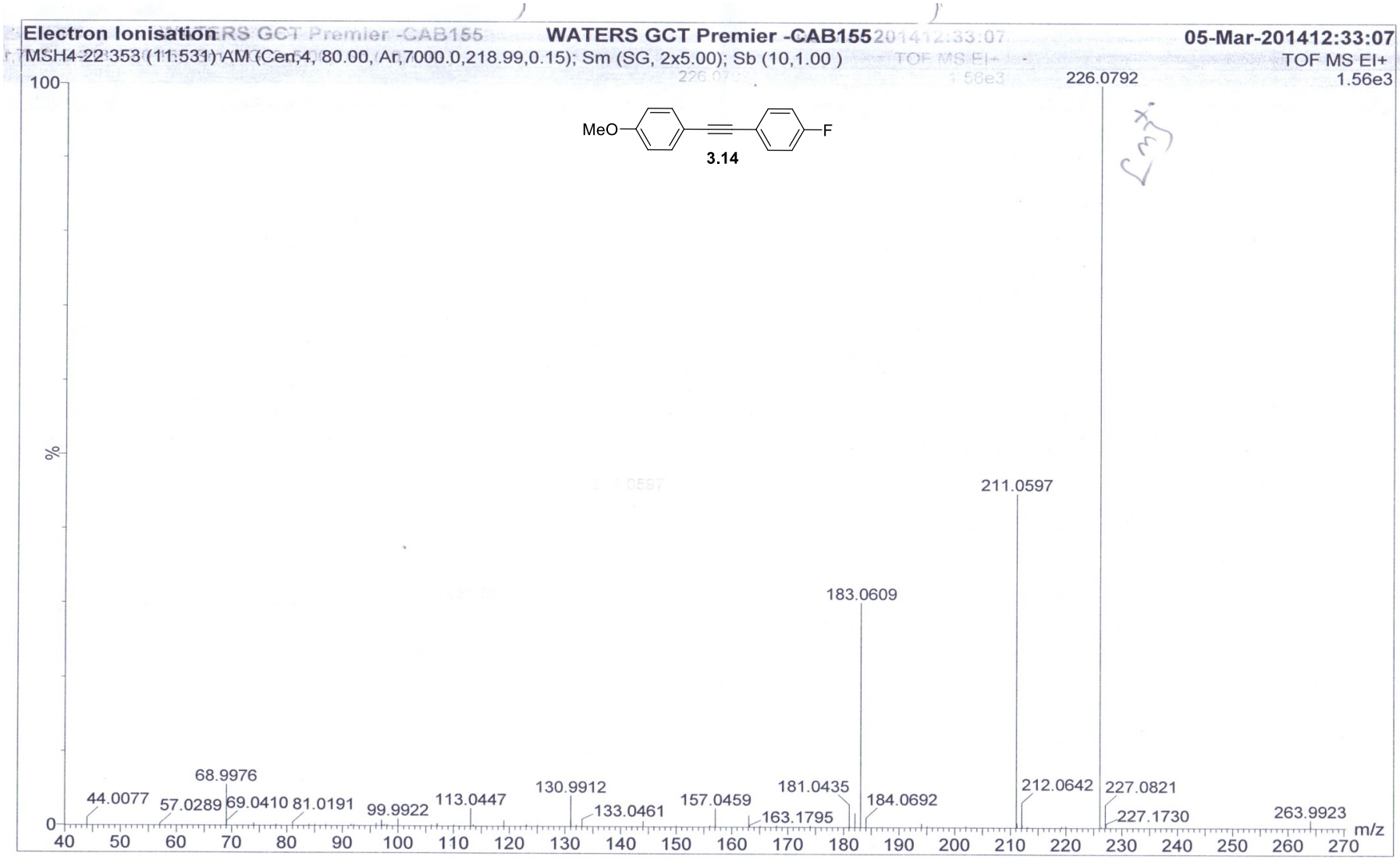
HRMS spectrum of 1-methoxy-4-((4-vinylphenyl)ethynyl)benzene **(3.13)**



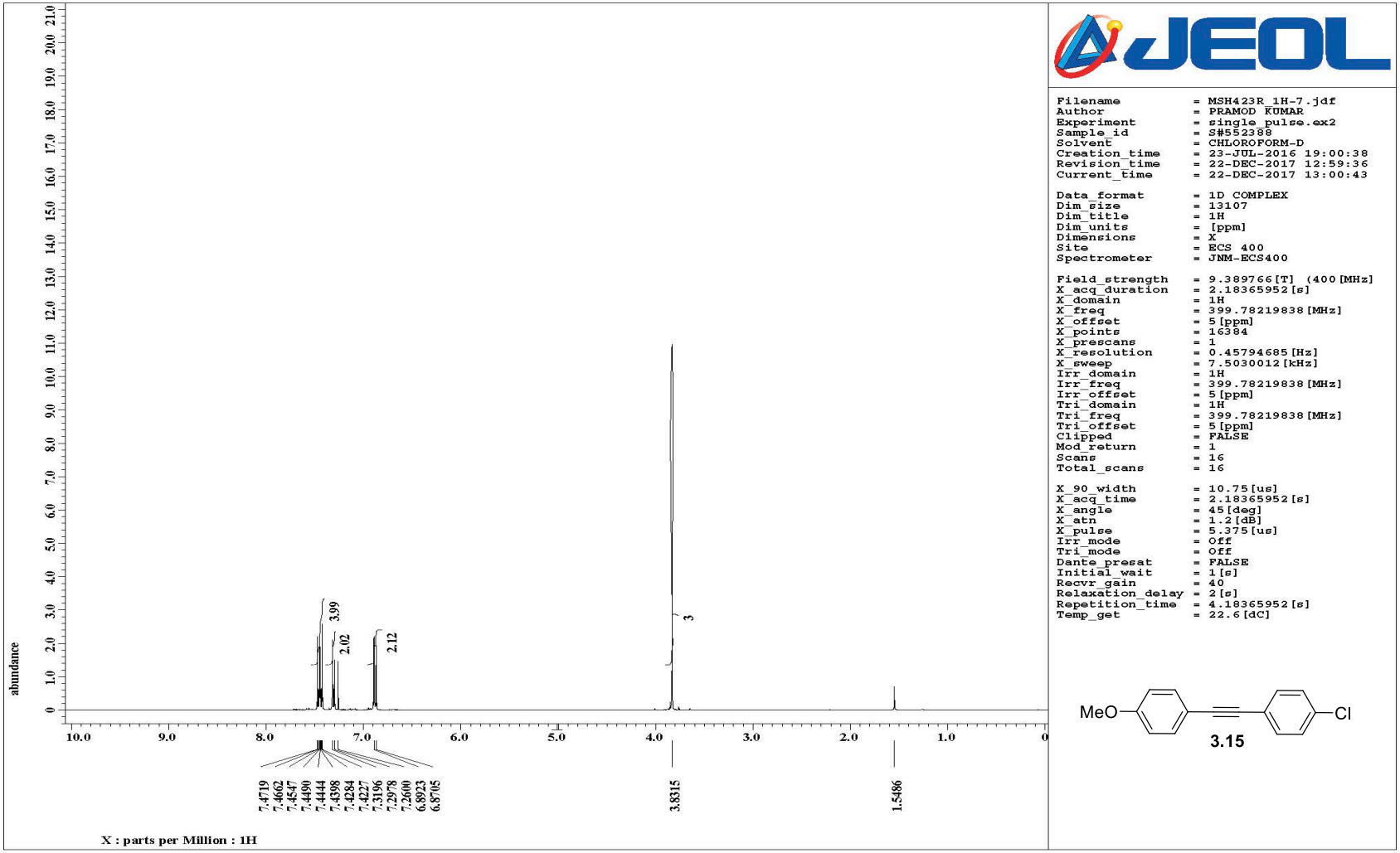
^{1}H NMR spectrum of 1-fluoro-4-((4-methoxyphenyl)ethynyl)benzene **(3.14)**



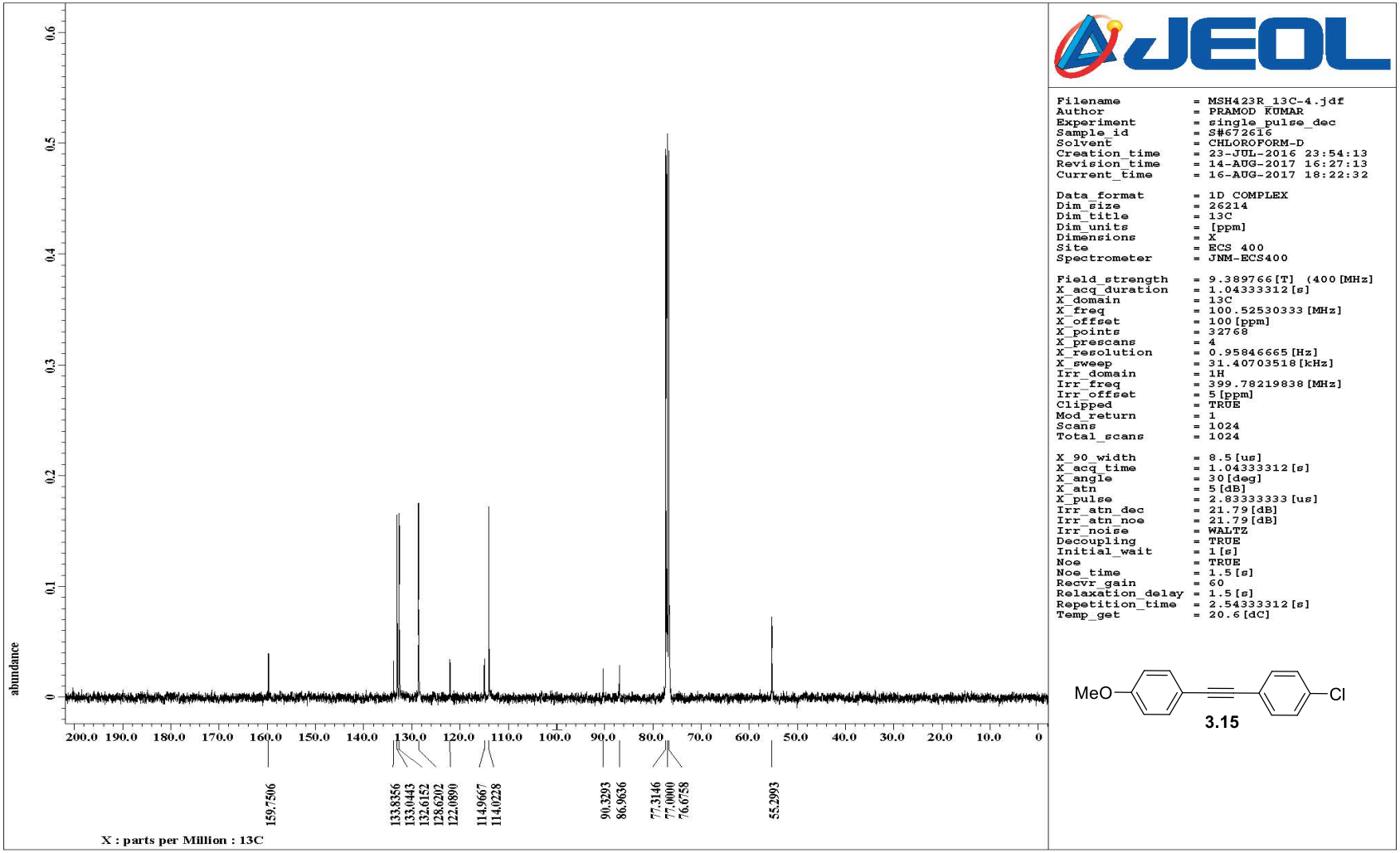
^{13}C NMR spectrum of 1-fluoro-4-((4-methoxyphenyl)ethynyl)benzene **(3.14)**



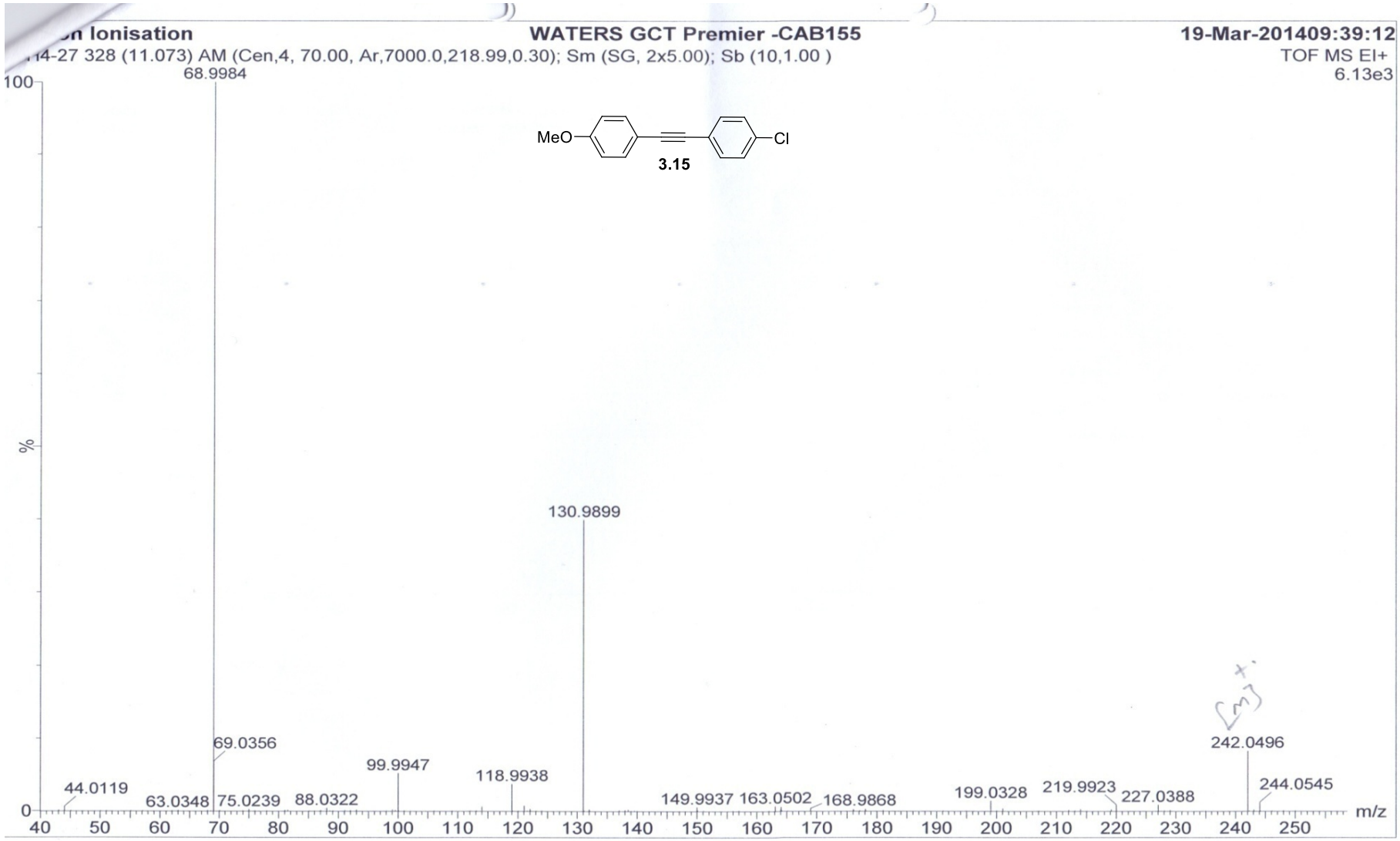
HRMS spectrum of 1-fluoro-4-((4-methoxyphenyl)ethynyl)benzene **(3.14)**



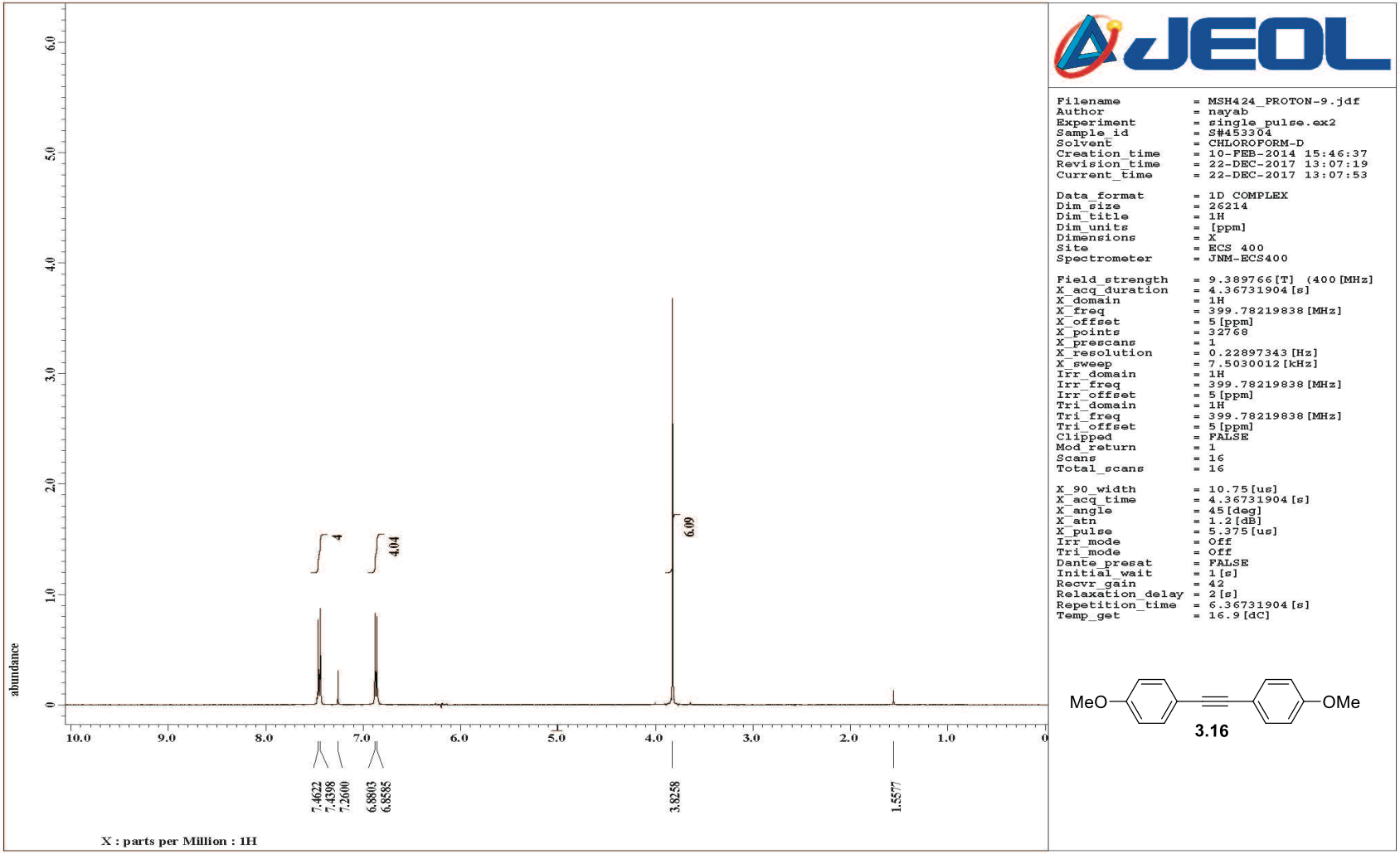
^{1}H NMR spectrum of 1-chloro-4-((4-methoxyphenyl)ethynyl)benzene **(3.15)**



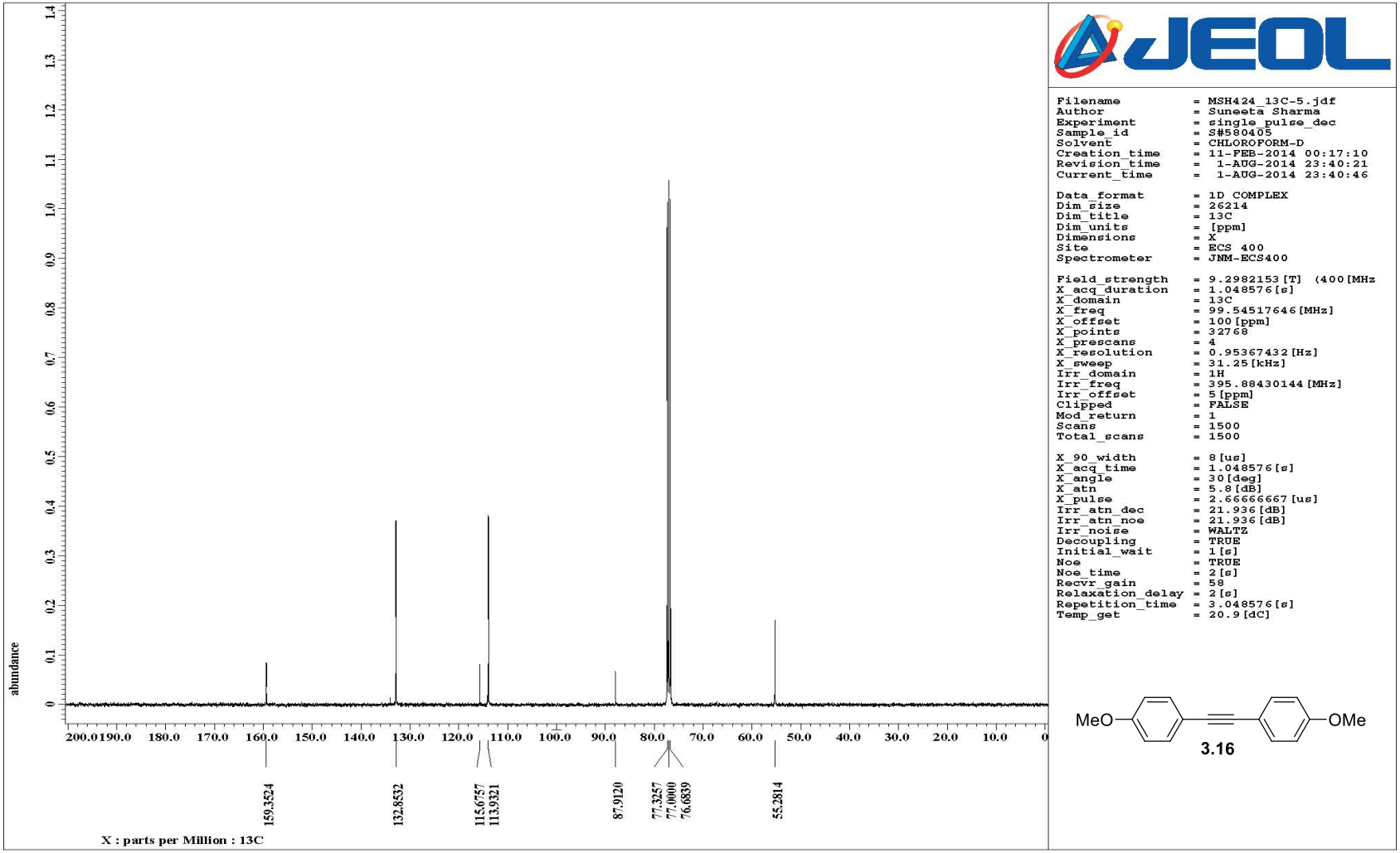
^{13}C NMR spectrum of 1-chloro-4-((4-methoxyphenyl)ethynyl)benzene **(3.15)**



HRMS spectrum of 1-chloro-4-((4-methoxyphenyl)ethynyl)benzene **(3.15)**



^{1}H NMR spectrum of 1,2-bis(4-methoxyphenyl)ethyne **(3.16)**



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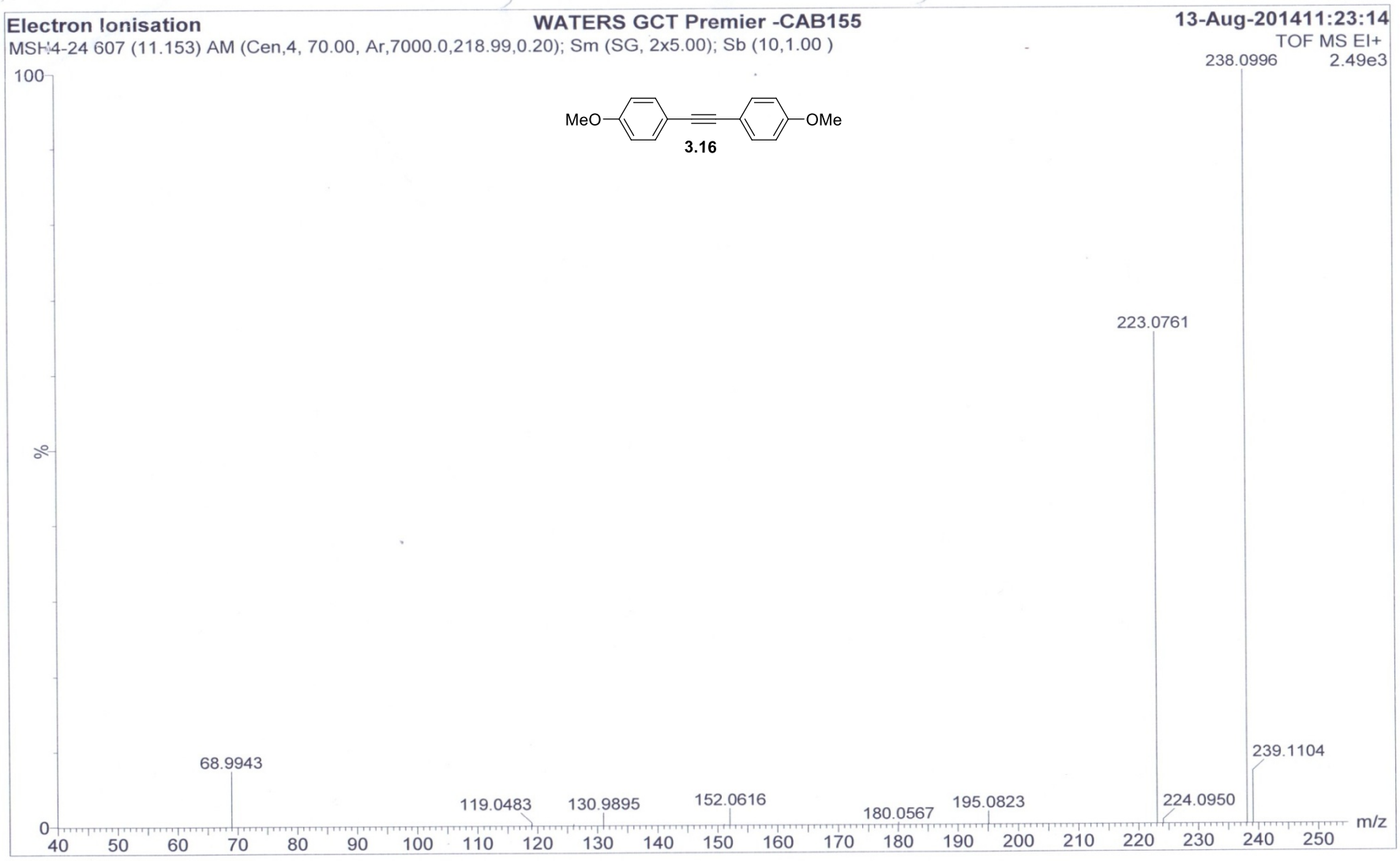
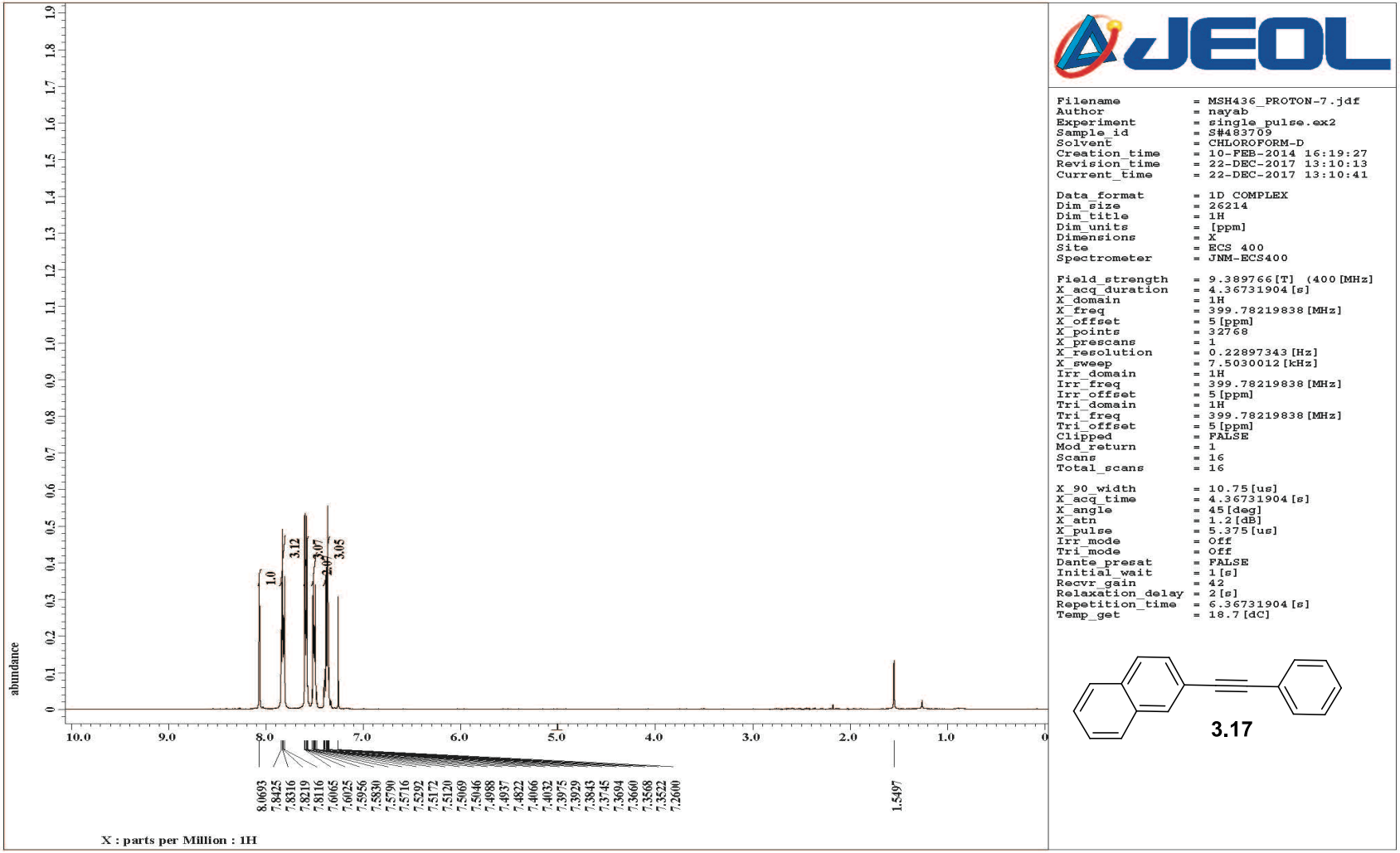
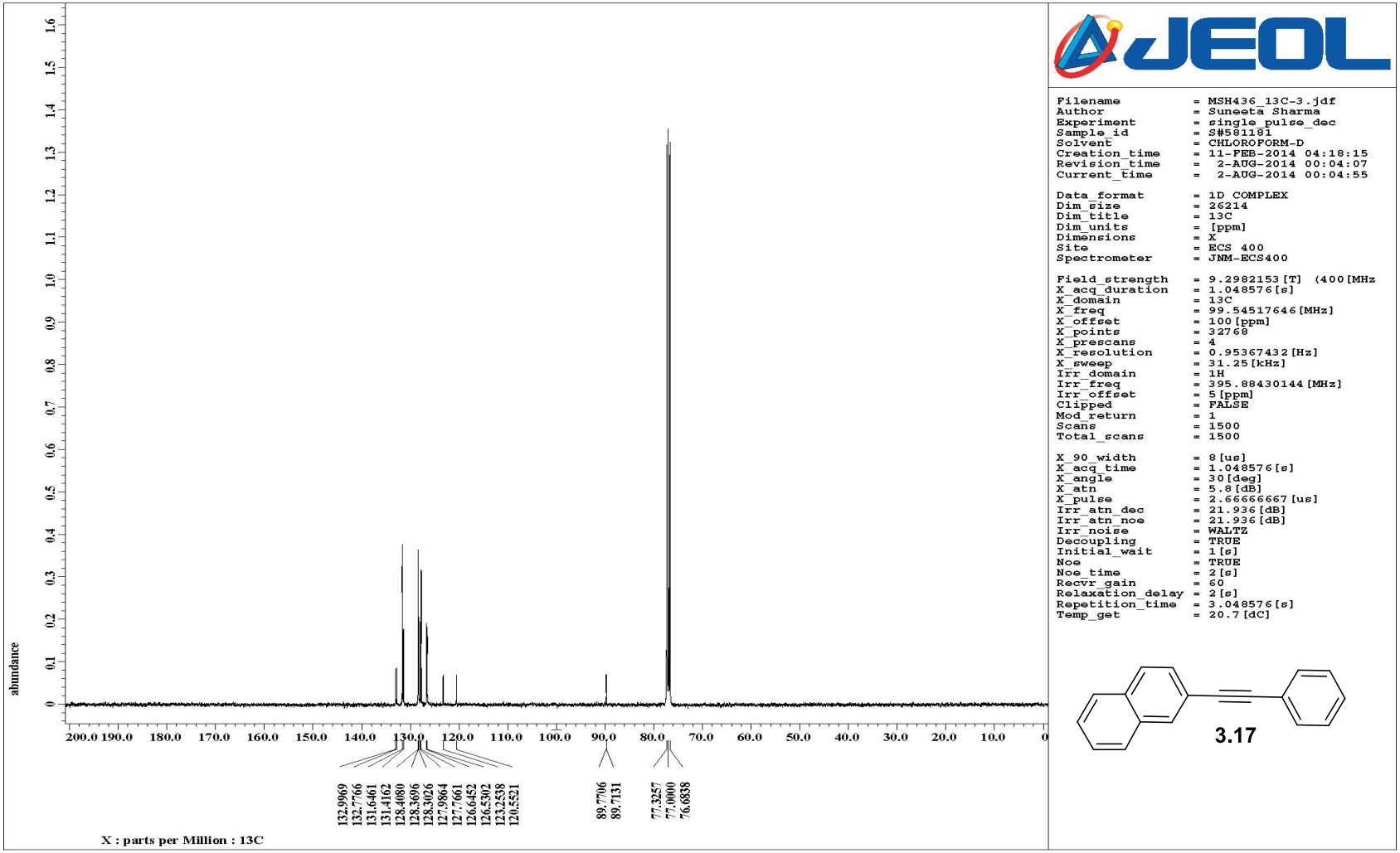
^{13}C NMR spectrum of 1,2-bis(4-methoxyphenyl)ethyne **(3.16)**



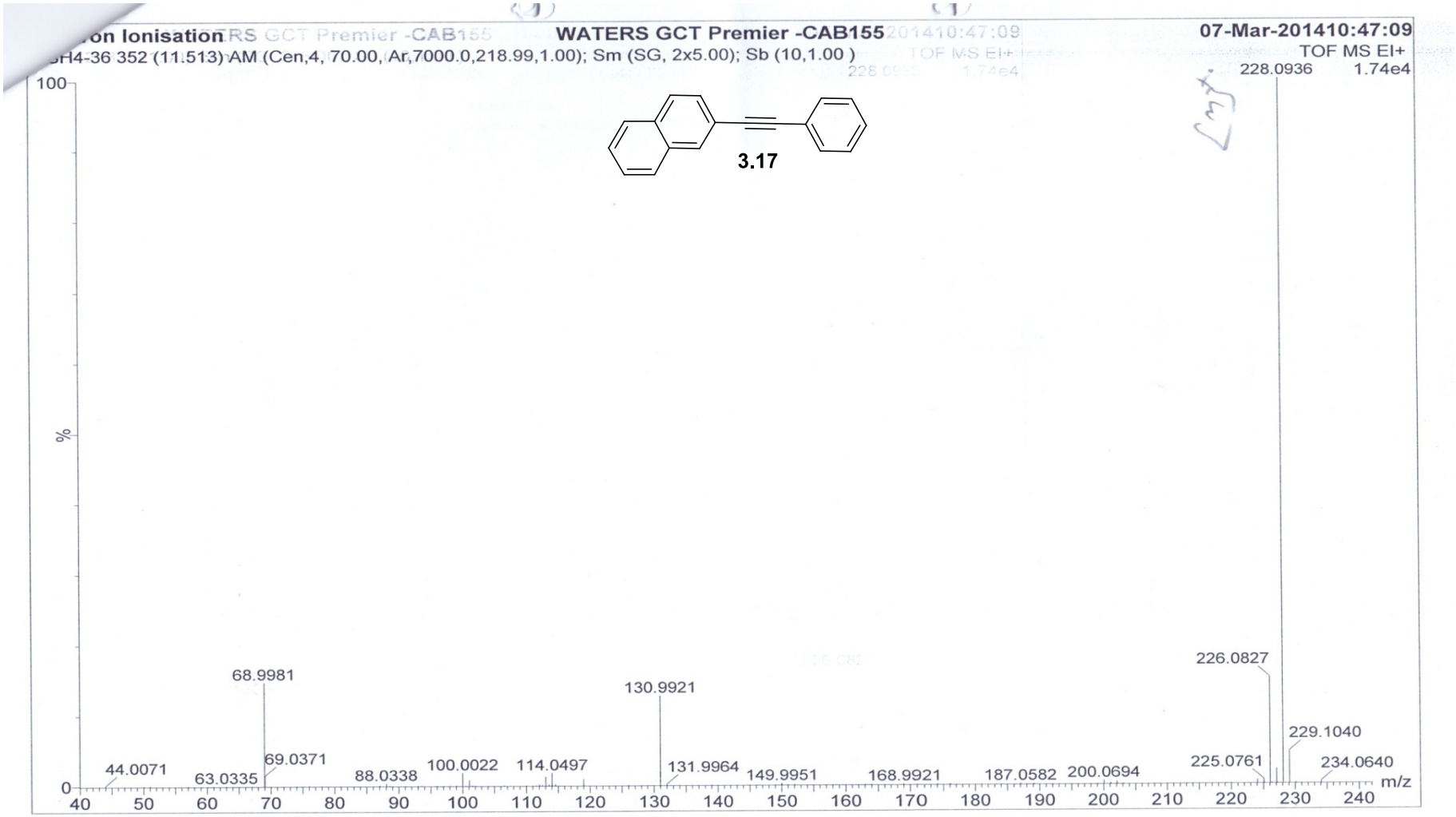
HRMS spectrum of 1,2-bis(4-methoxyphenyl)ethyne **(3.16)**



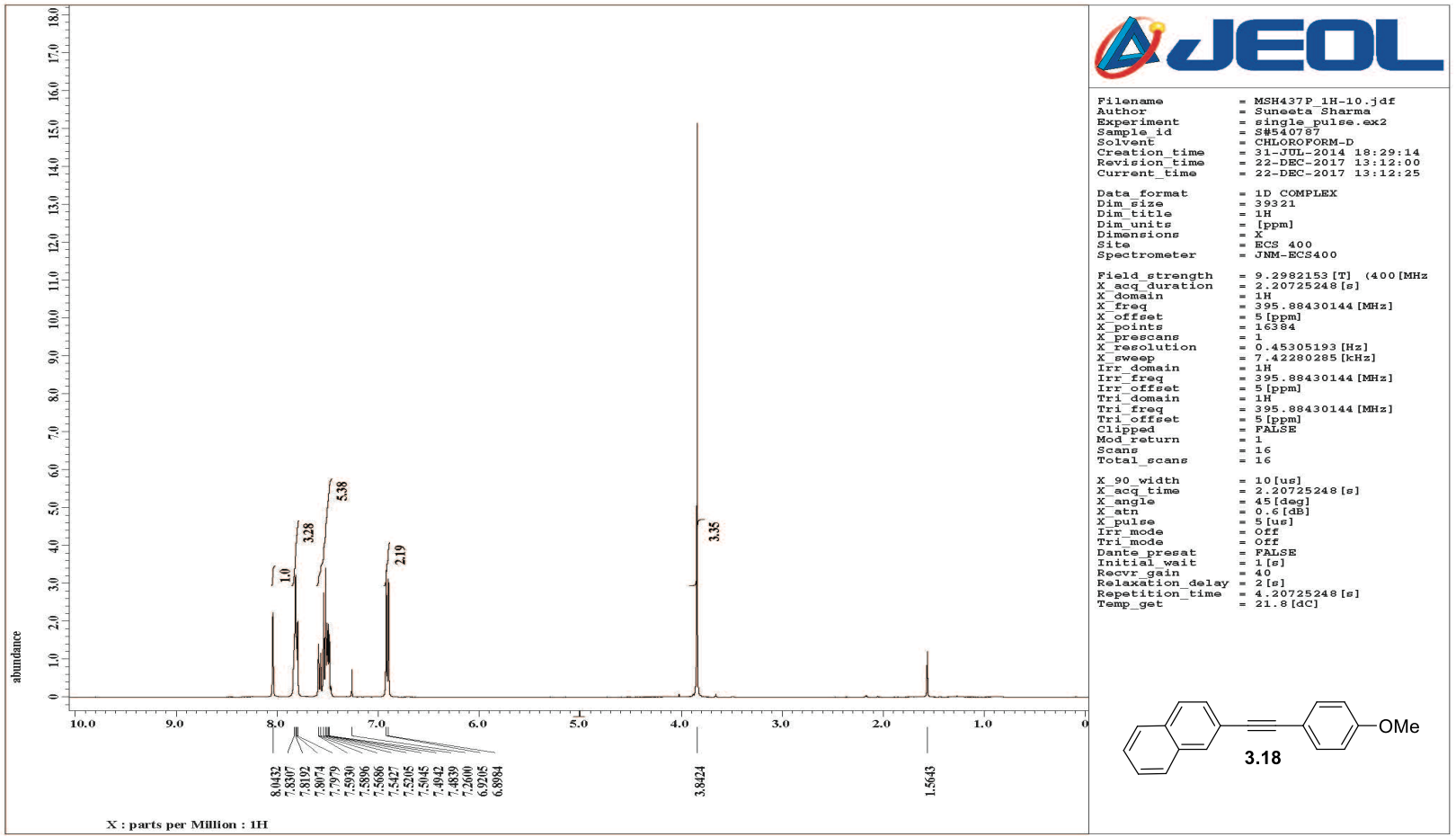
^{1}H NMR spectrum of 2-(phenylethynyl)naphthalene **(3.17)**



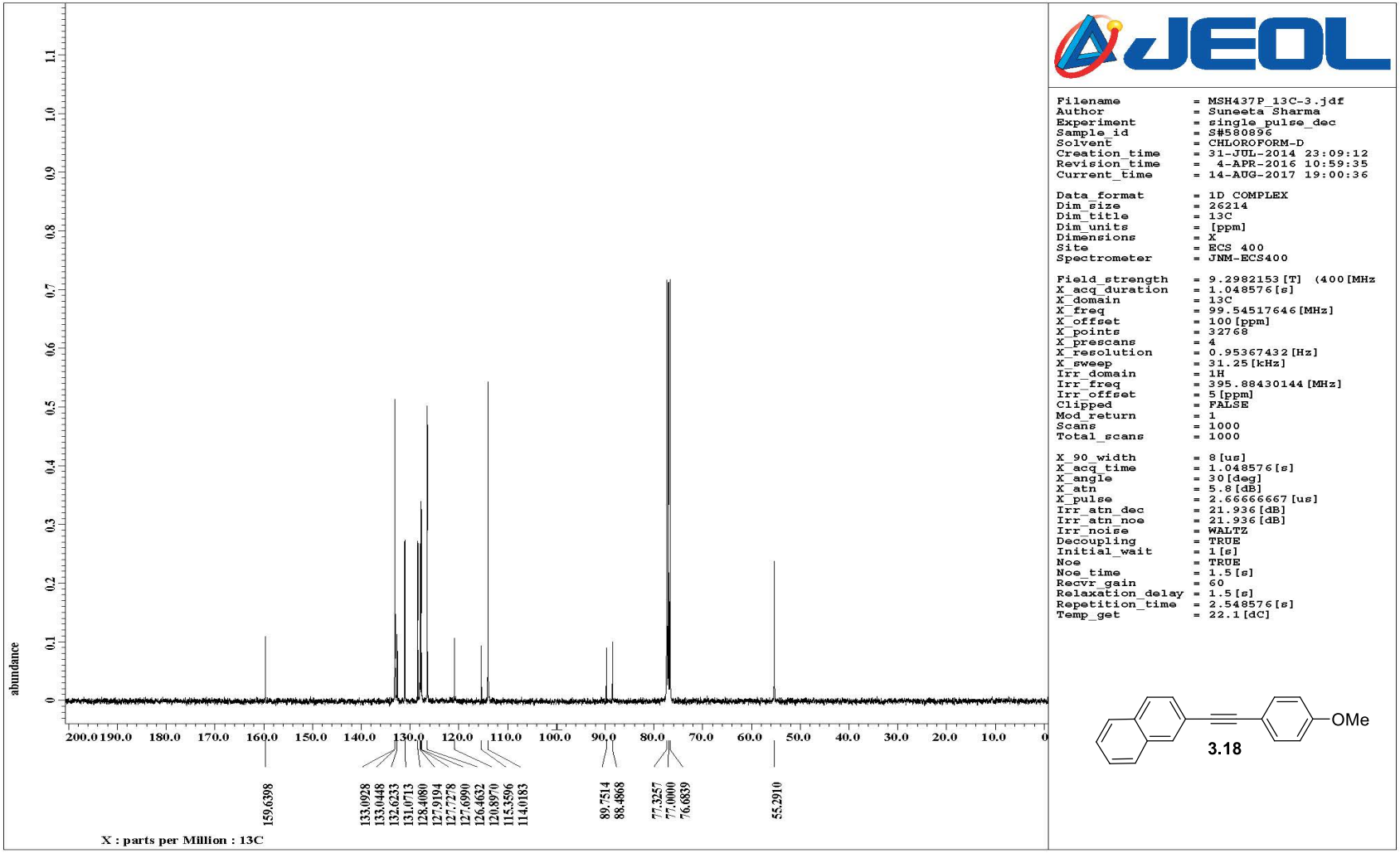
^{13}C NMR spectrum of 2-(phenylethynyl)naphthalene **(3.17)**



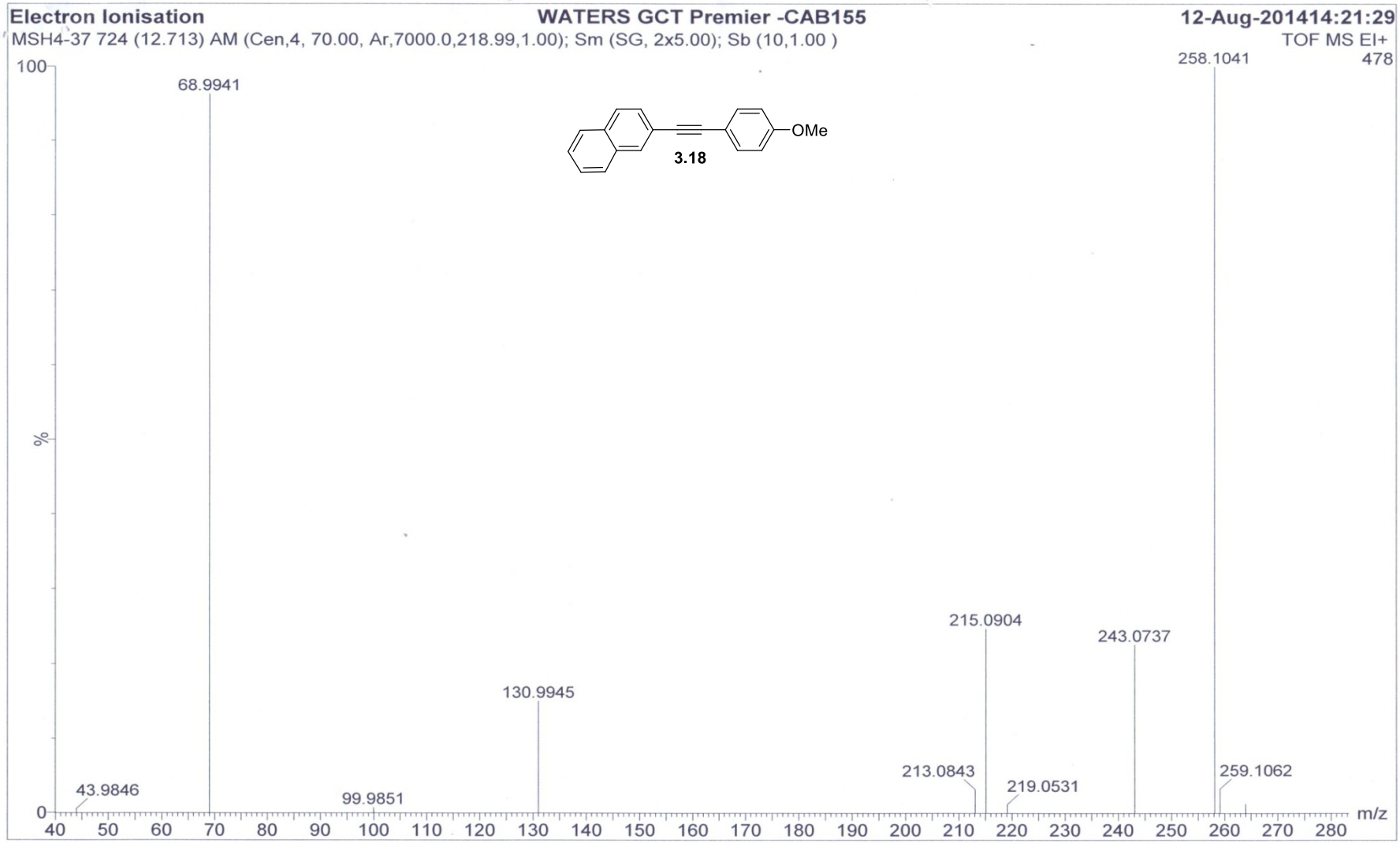
HRMS spectrum of 2-(phenylethynyl)naphthalene **(3.17)**



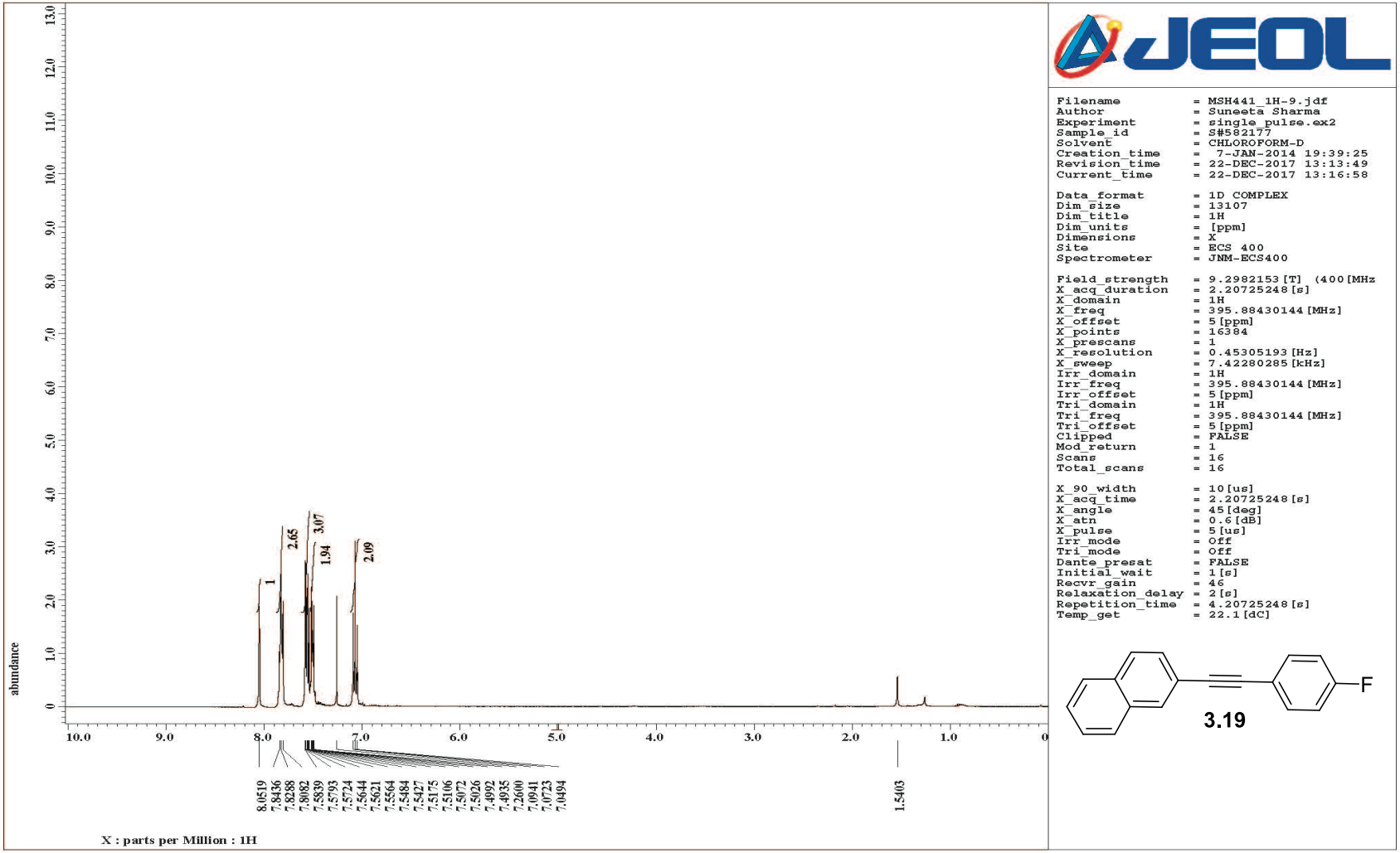
^{1}H NMR spectrum of 2-((4-methoxyphenyl)ethynyl)naphthalene **(3.18)**



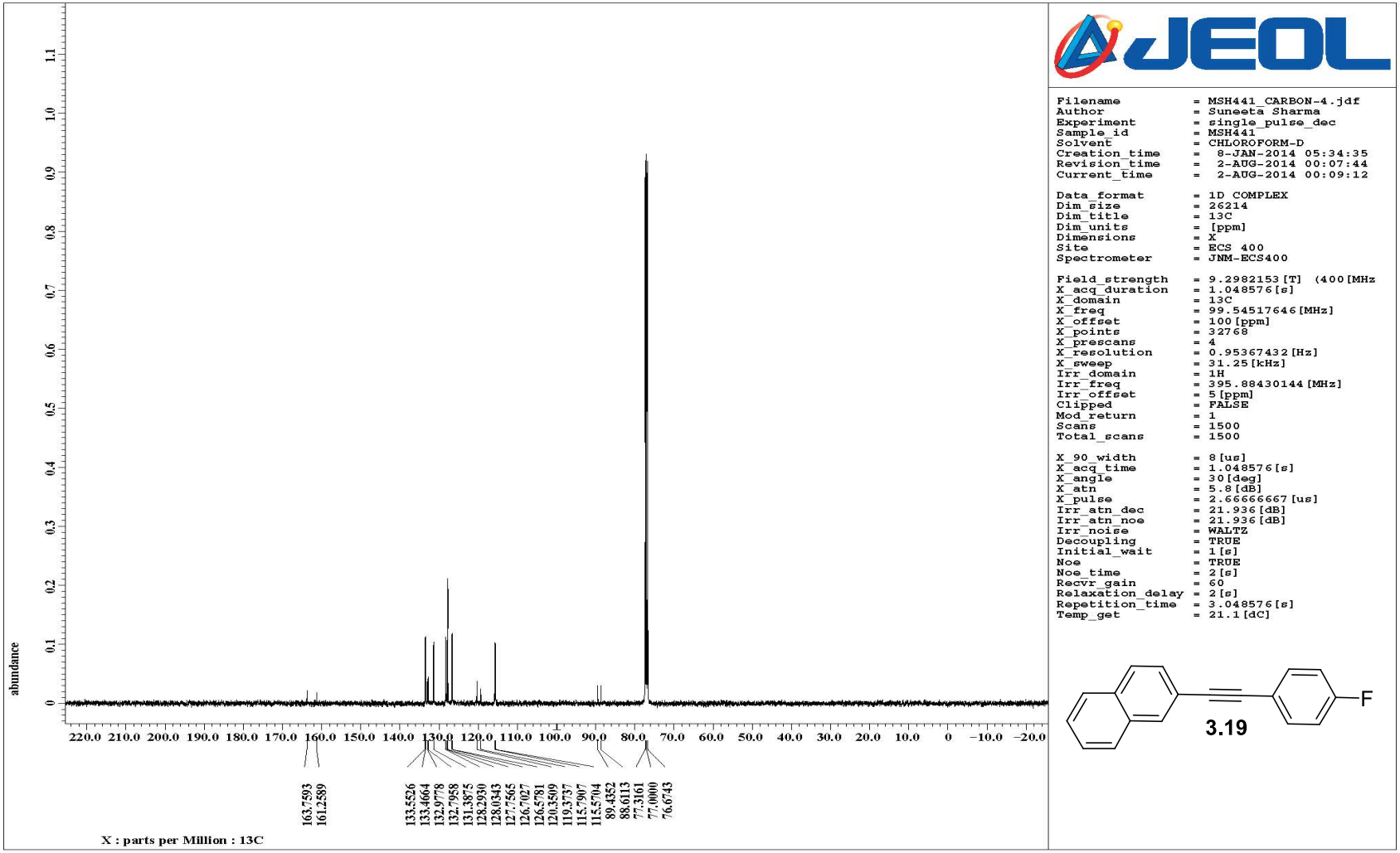
^{13}C NMR spectrum of 2-((4-methoxyphenyl)ethynyl)naphthalene **(3.18)**



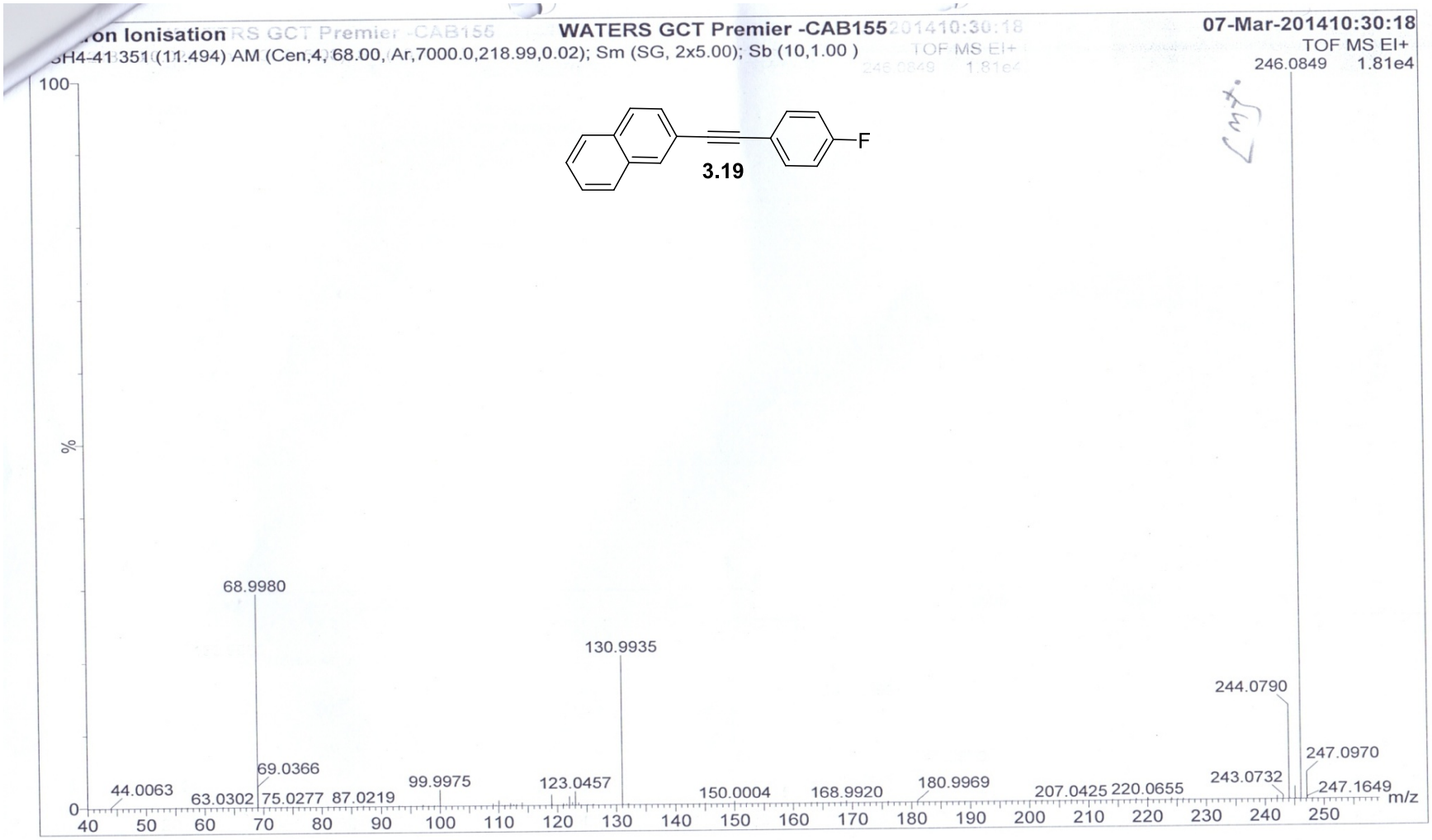
HRMS spectrum of 2-((4-methoxyphenyl)ethynyl)naphthalene **(3.18)**



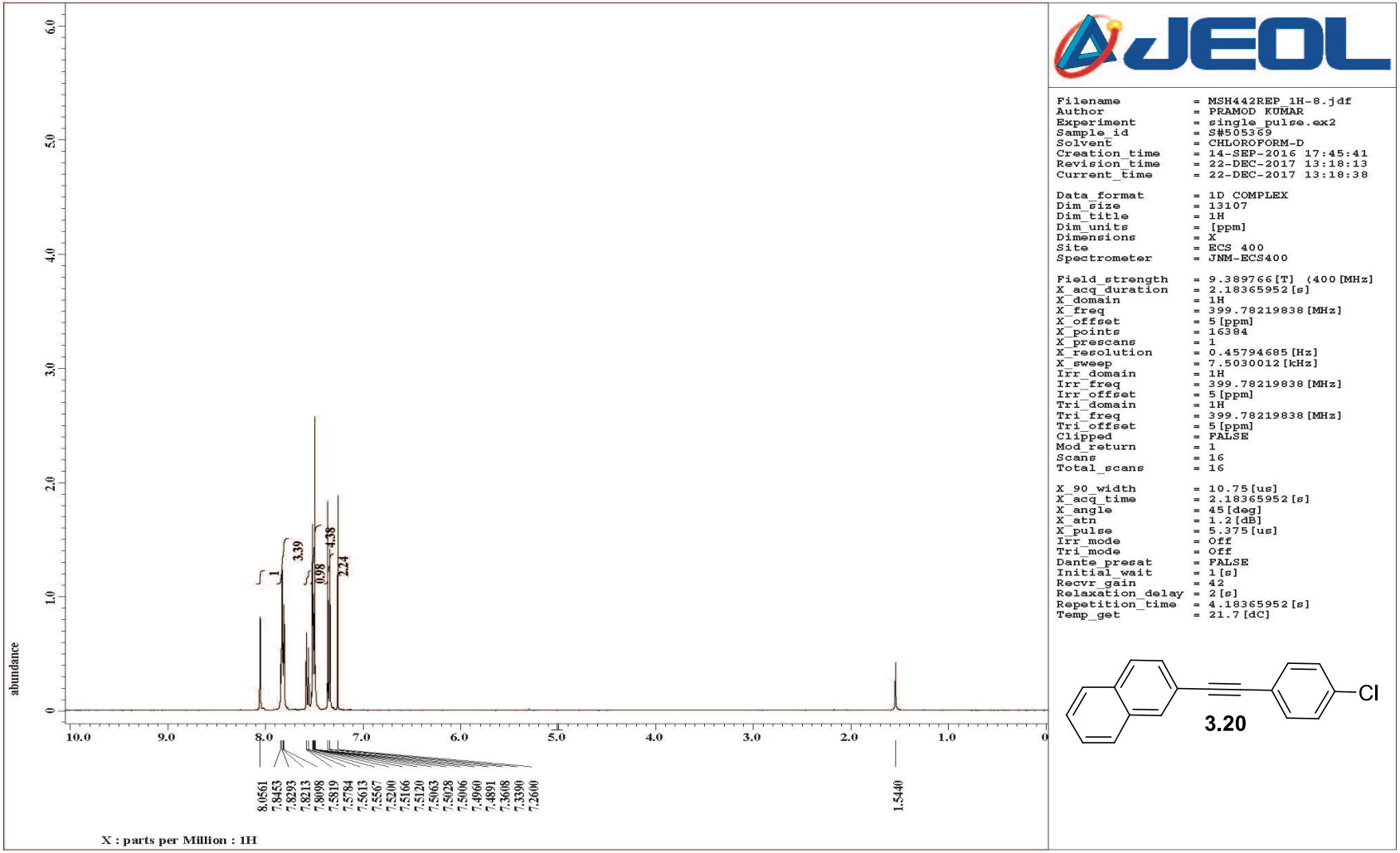
^{1}H NMR spectrum of 2-((4-fluorophenyl)ethynyl)naphthalene **(3.19)**



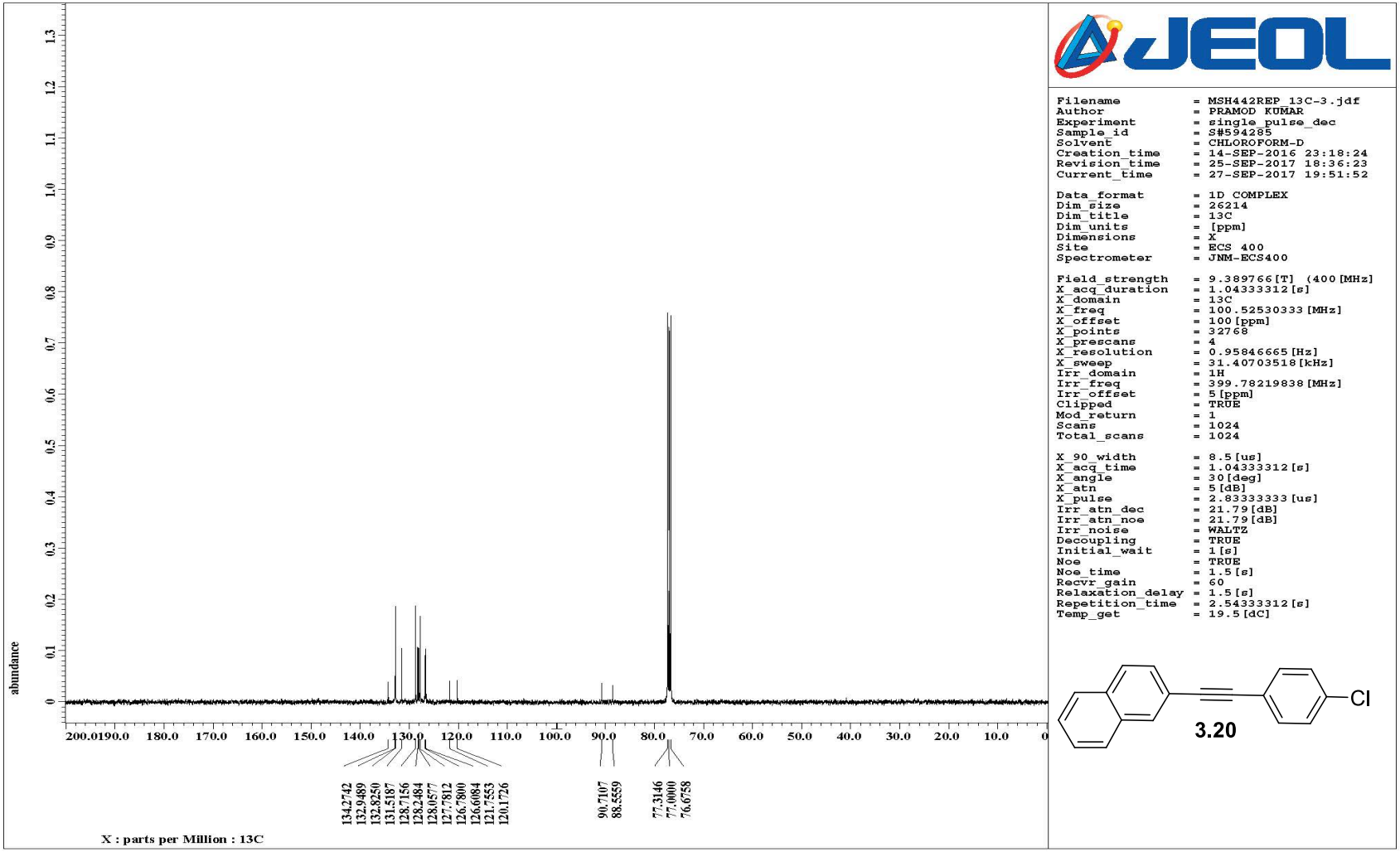
^{13}C NMR spectrum of 2-((4-fluorophenyl)ethynyl)naphthalene **(3.19)**



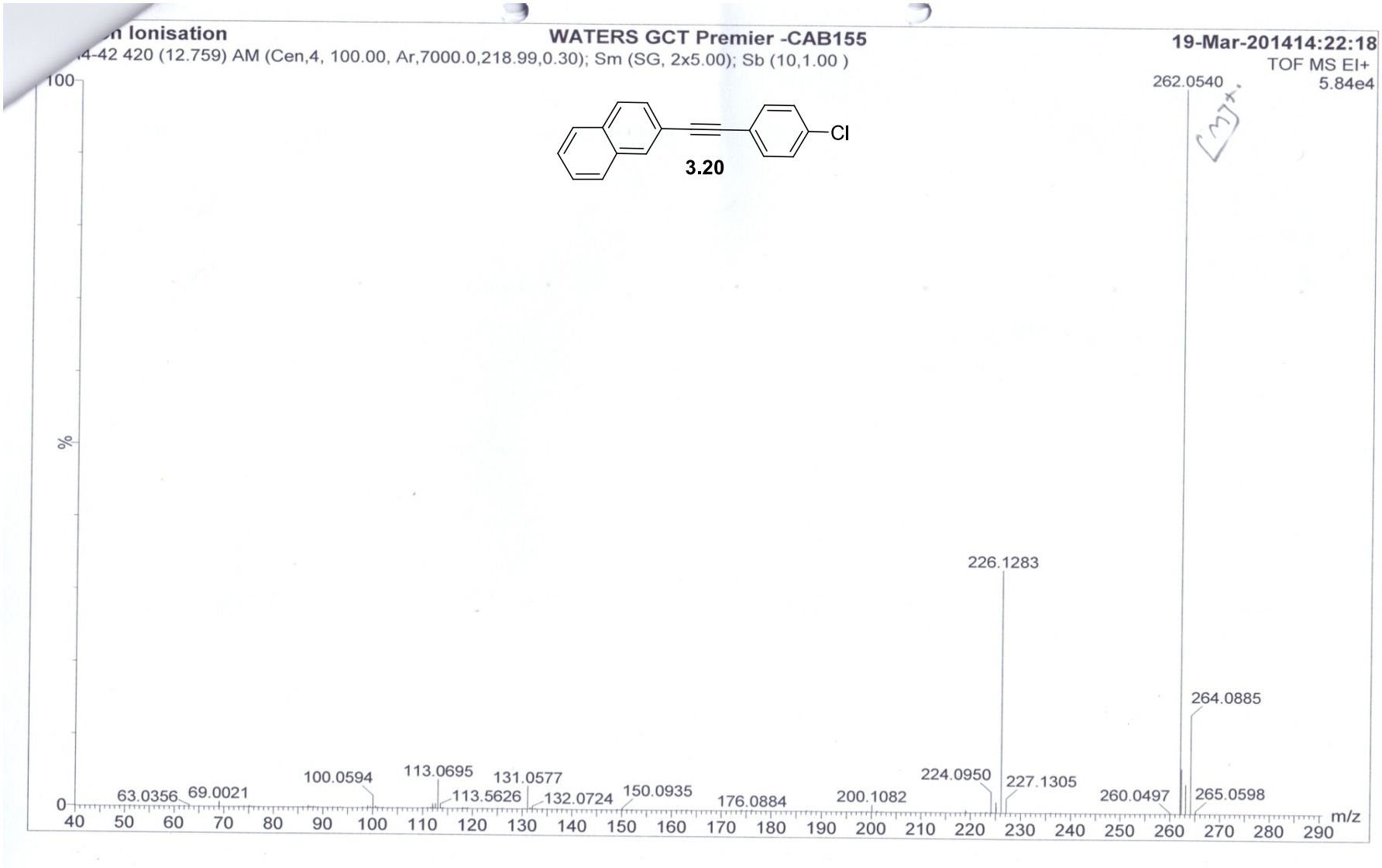
HRMS spectrum of 2-((4-fluorophenyl)ethynyl)naphthalene **(3.19)**



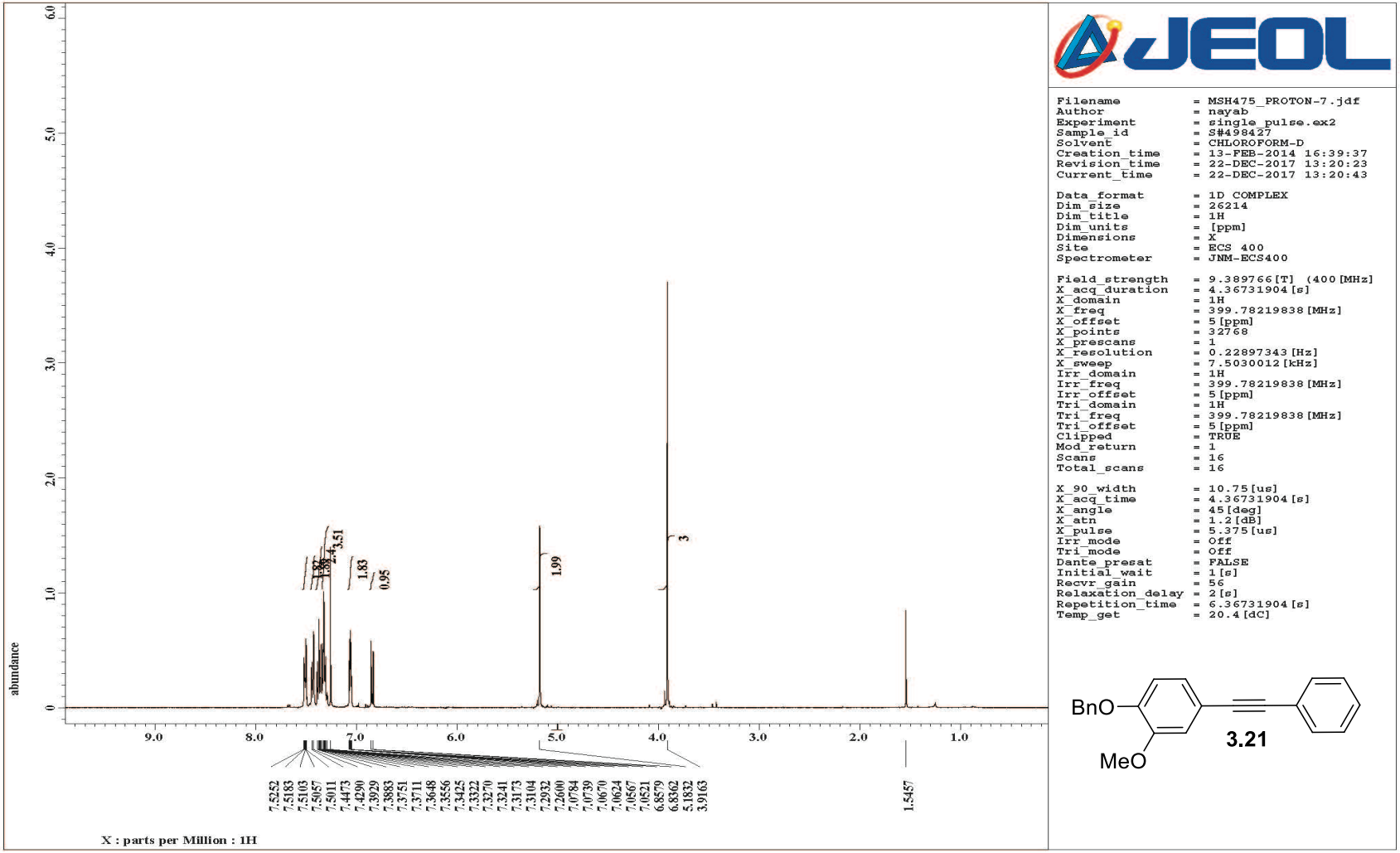
^{1}H NMR spectrum of 2-((4-chlorophenyl)ethynyl)naphthalene **(3.20)**



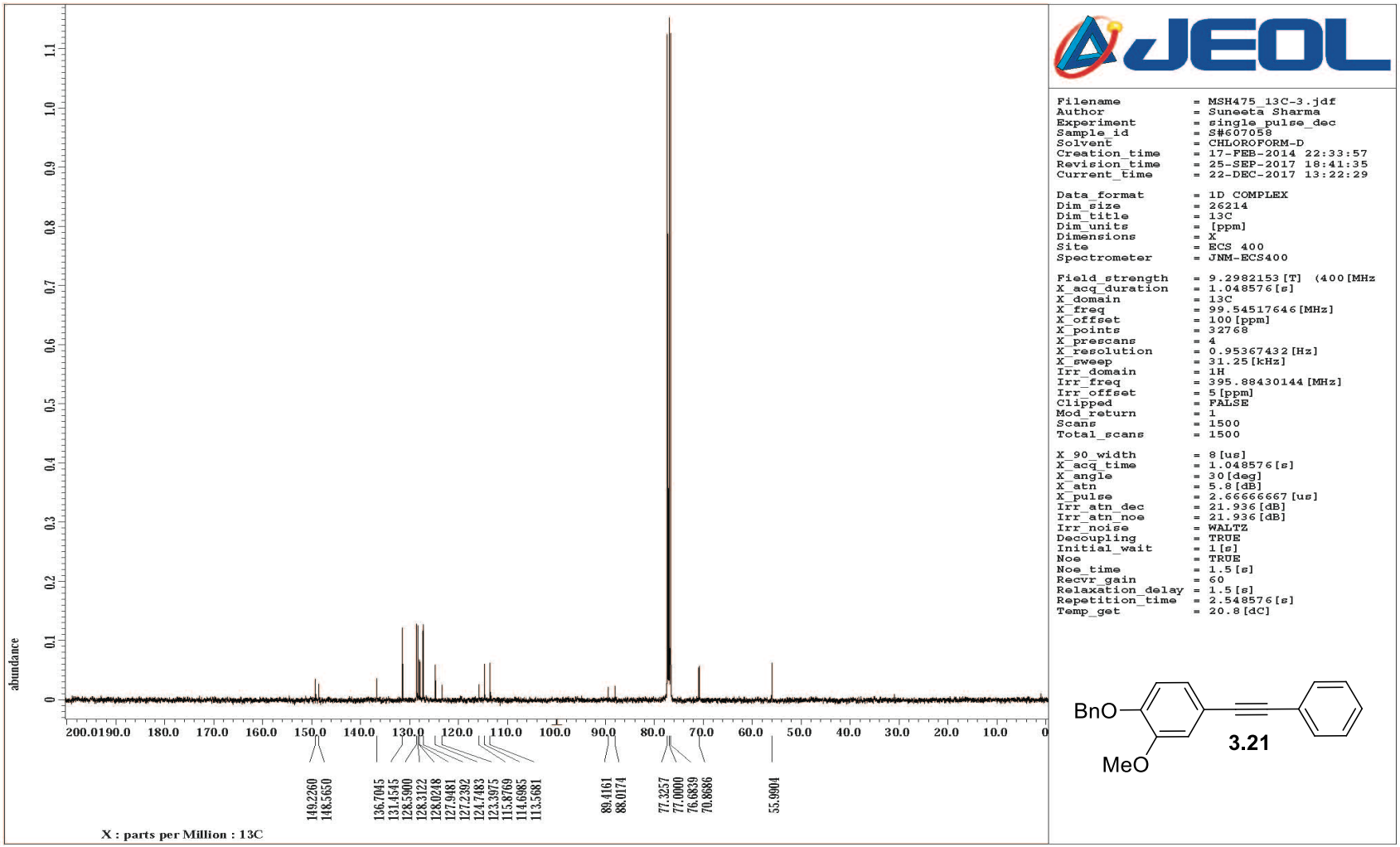
^{13}C NMR spectrum of 2-((4-chlorophenyl)ethynyl)naphthalene **(3.20)**



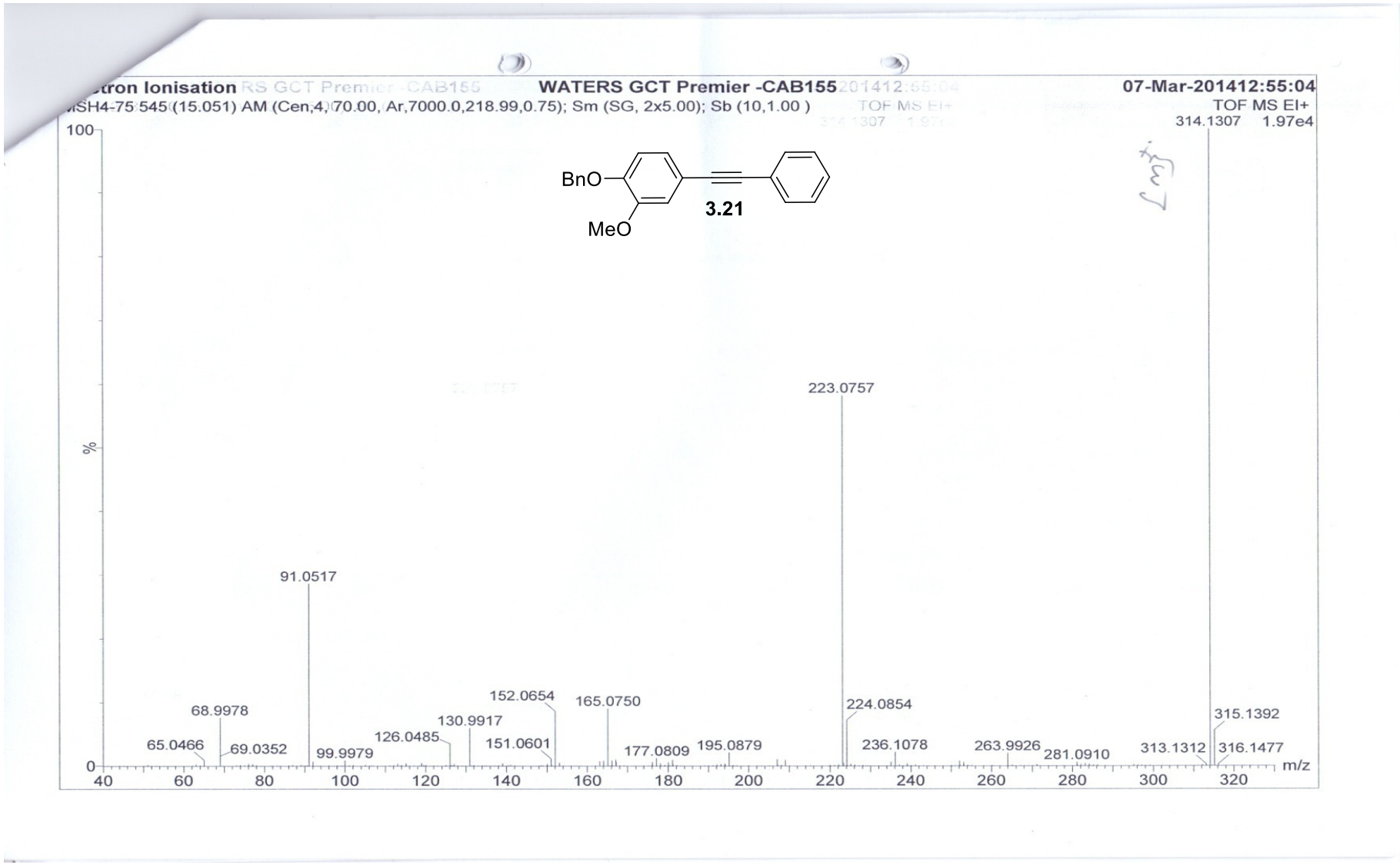
HRMS spectrum of 2-((4-chlorophenyl)ethynyl)naphthalene **(3.20)**



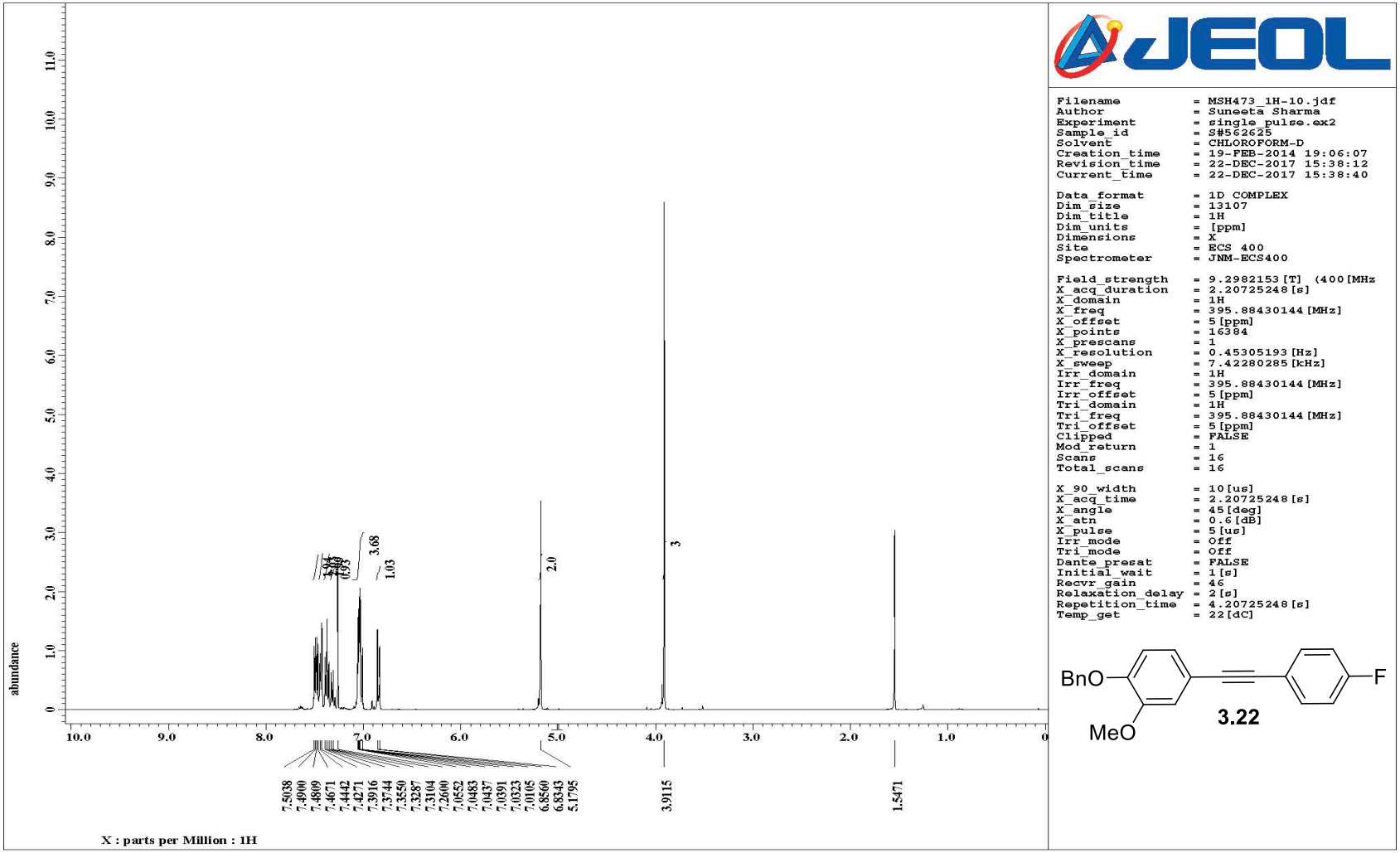
^{1}H NMR Spectrum of 1-(benzyloxy)-2-methoxy-4-(phenylethynyl)benzene **(3.21)**



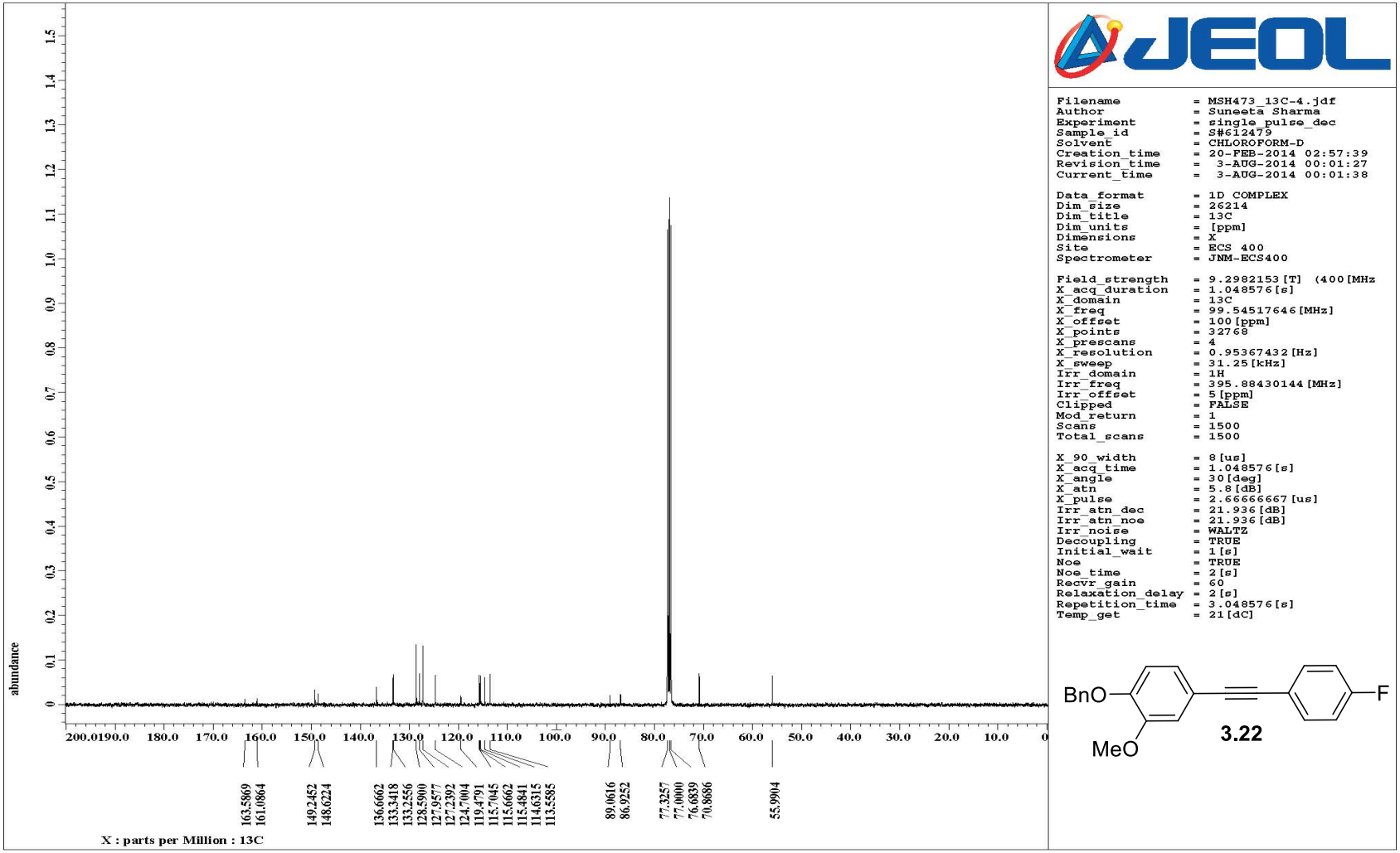
^{13}C NMR Spectrum of 1-(benzyloxy)-2-methoxy-4-(phenylethynyl)benzene **(3.21)**



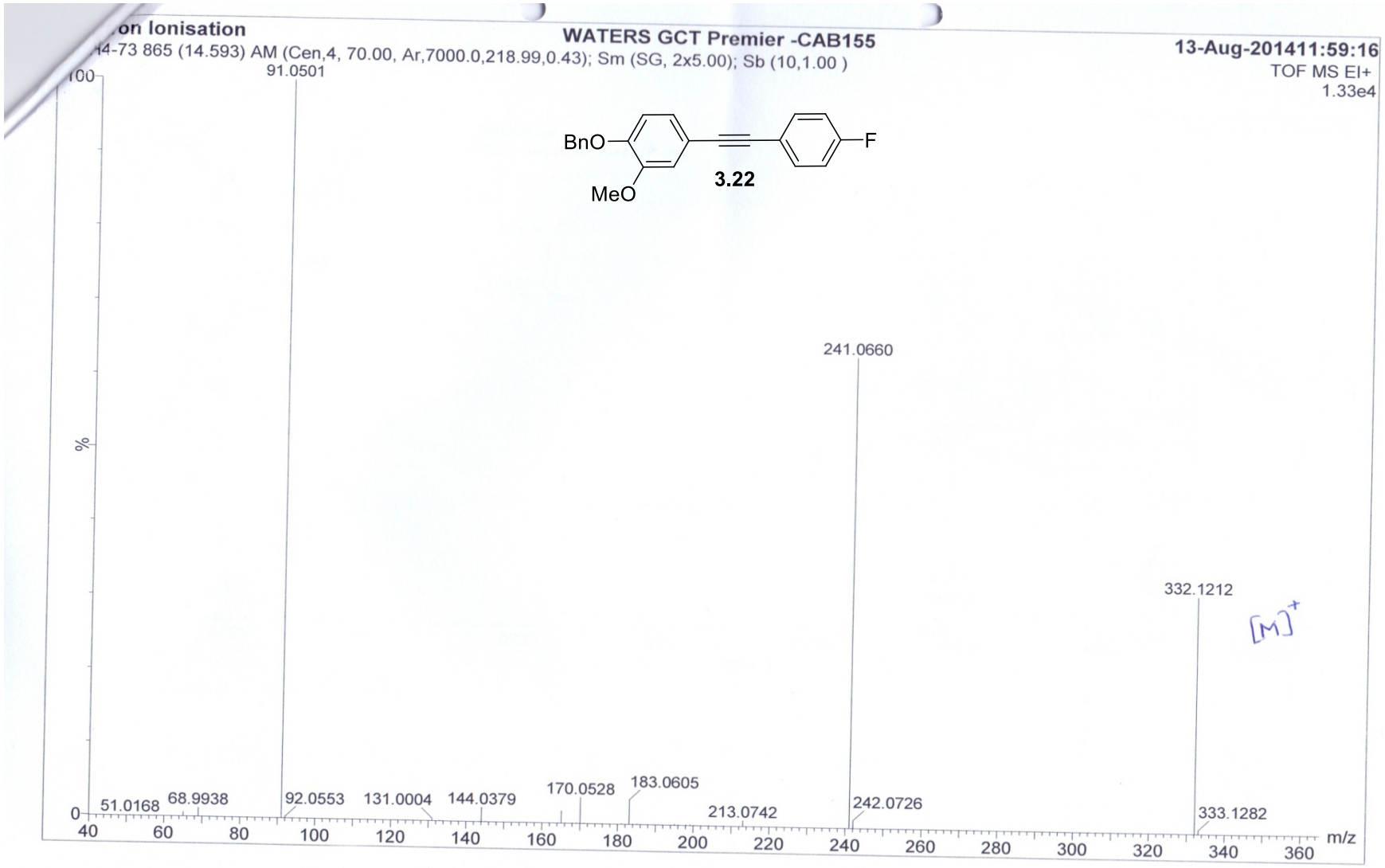
HRMS Spectrum of 1-(benzyloxy)-2-methoxy-4-(phenylethynyl)benzene **(3.21)**



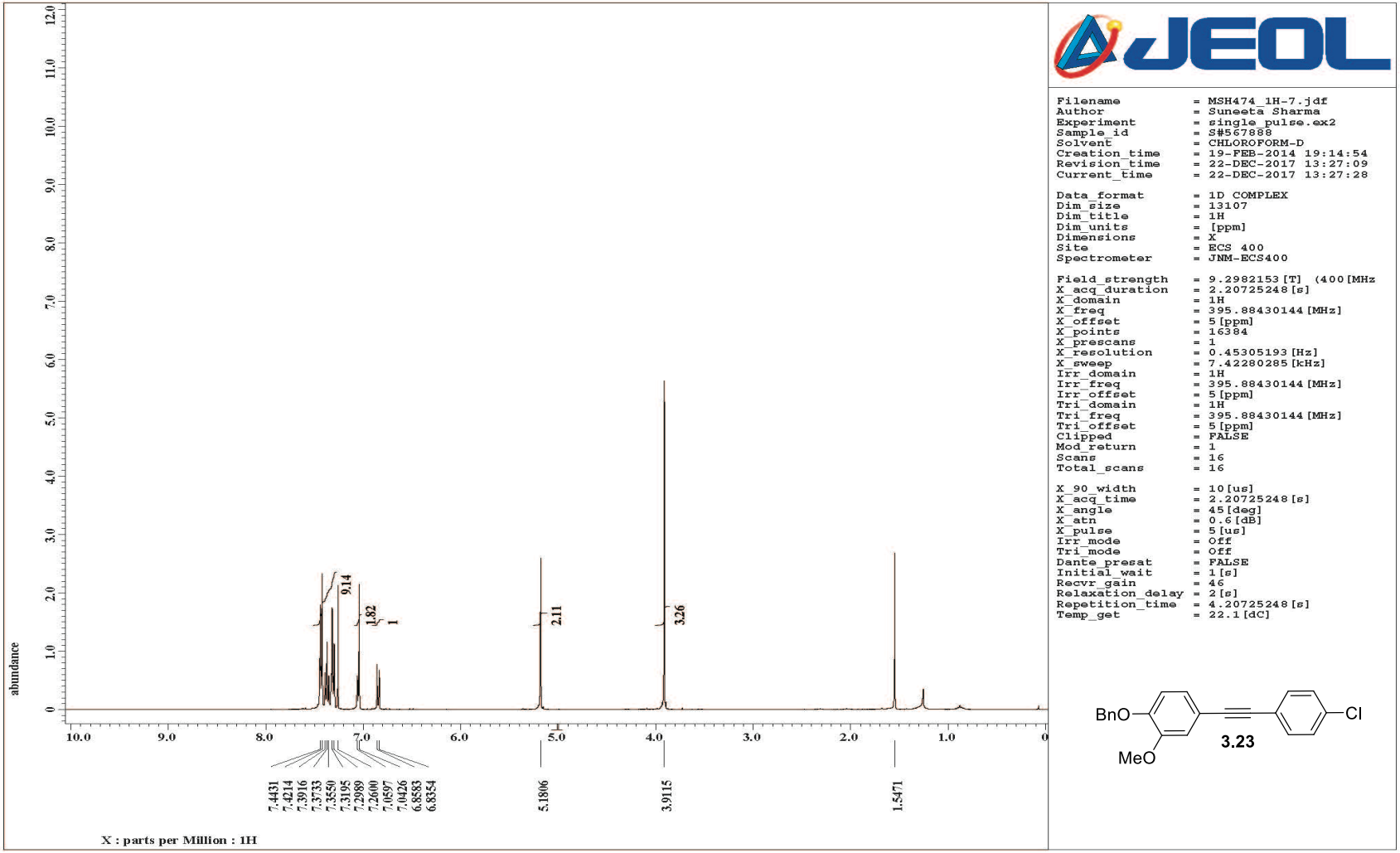
^{1}H NMR Spectrum of 1-(benzyloxy)-4-((4-fluorophenyl)ethynyl)-2-methoxybenzene **(3.22)**



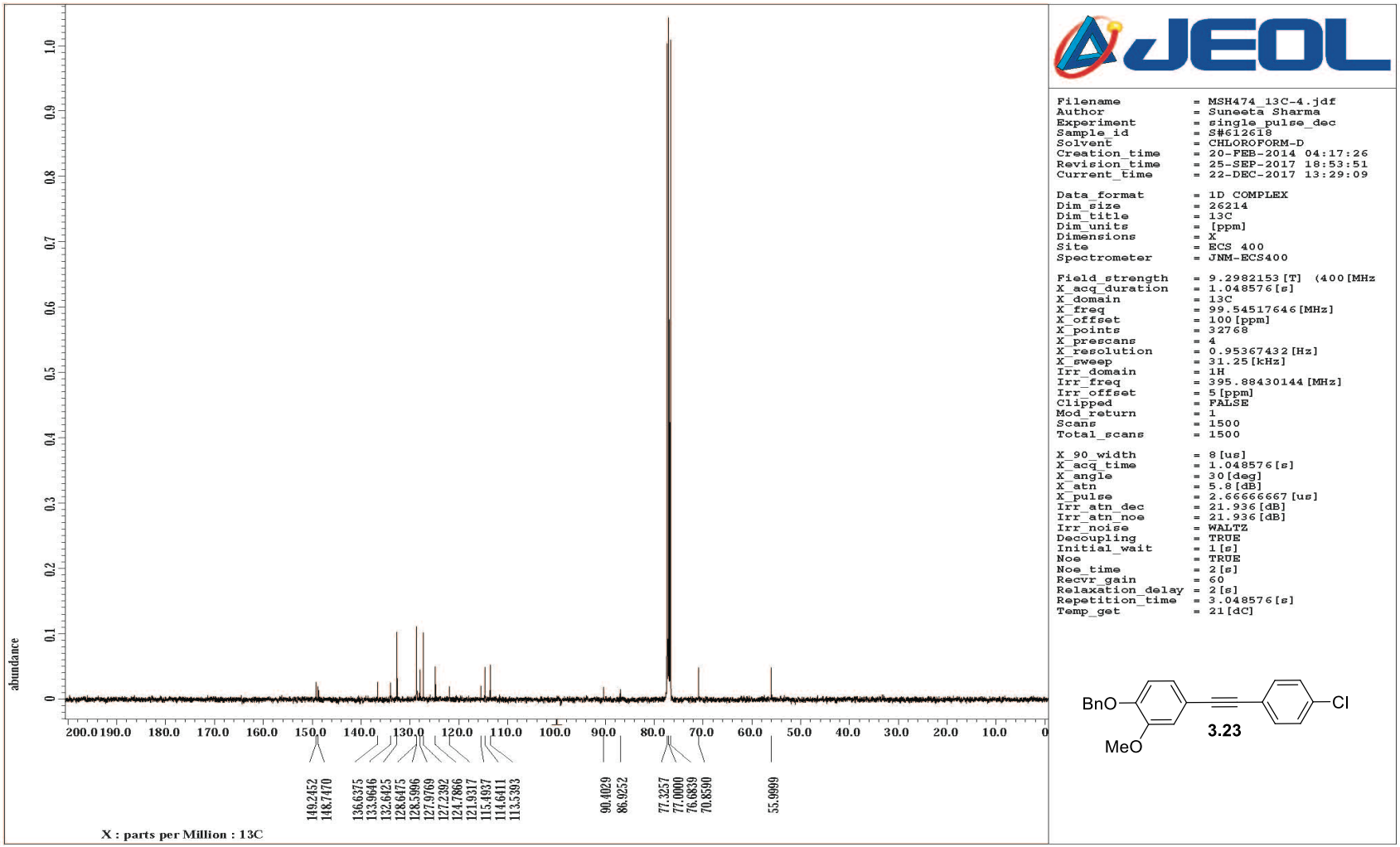
^{13}C NMR Spectrum of 1-(benzyloxy)-4-((4-fluorophenyl)ethynyl)-2-methoxybenzene **(3.22)**



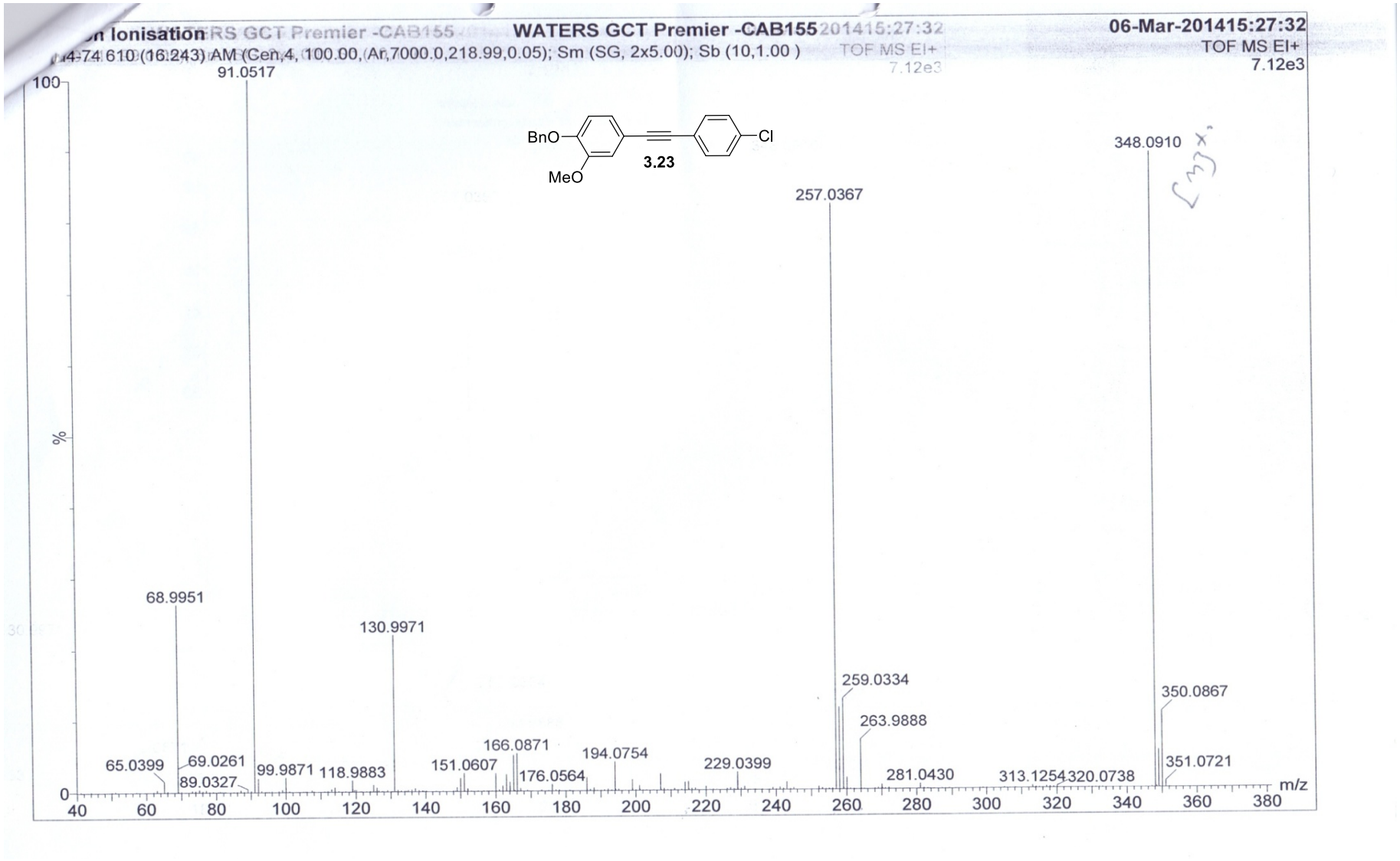
HRMS Spectrum of 1-(benzyloxy)-4-((4-fluorophenyl)ethynyl)-2-methoxybenzene **(3.22)**



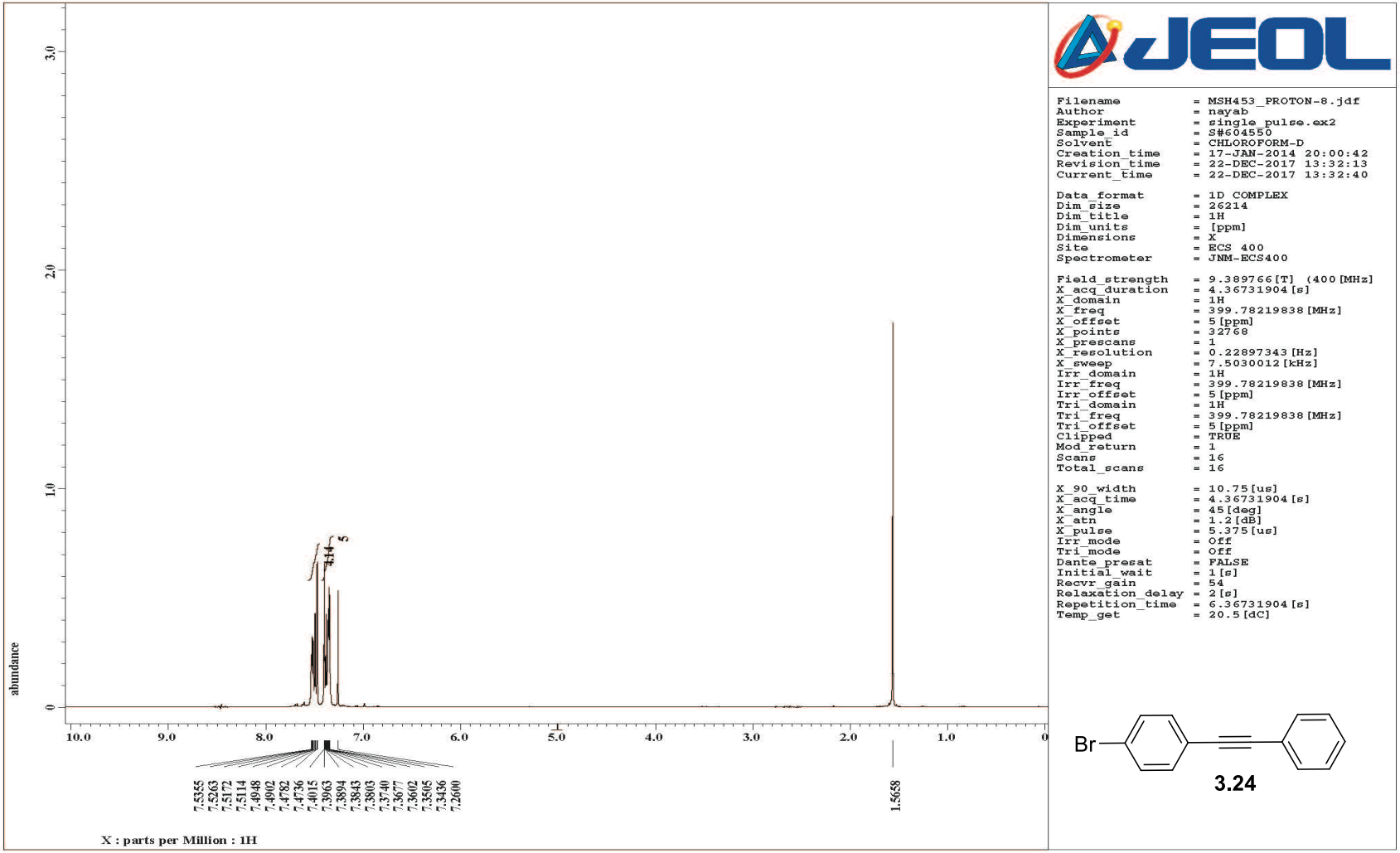
^{1}H NMR Spectrum of 1-(benzyloxy)-4-((4-chlorophenyl)ethynyl)-2-methoxybenzene **(3.23)**



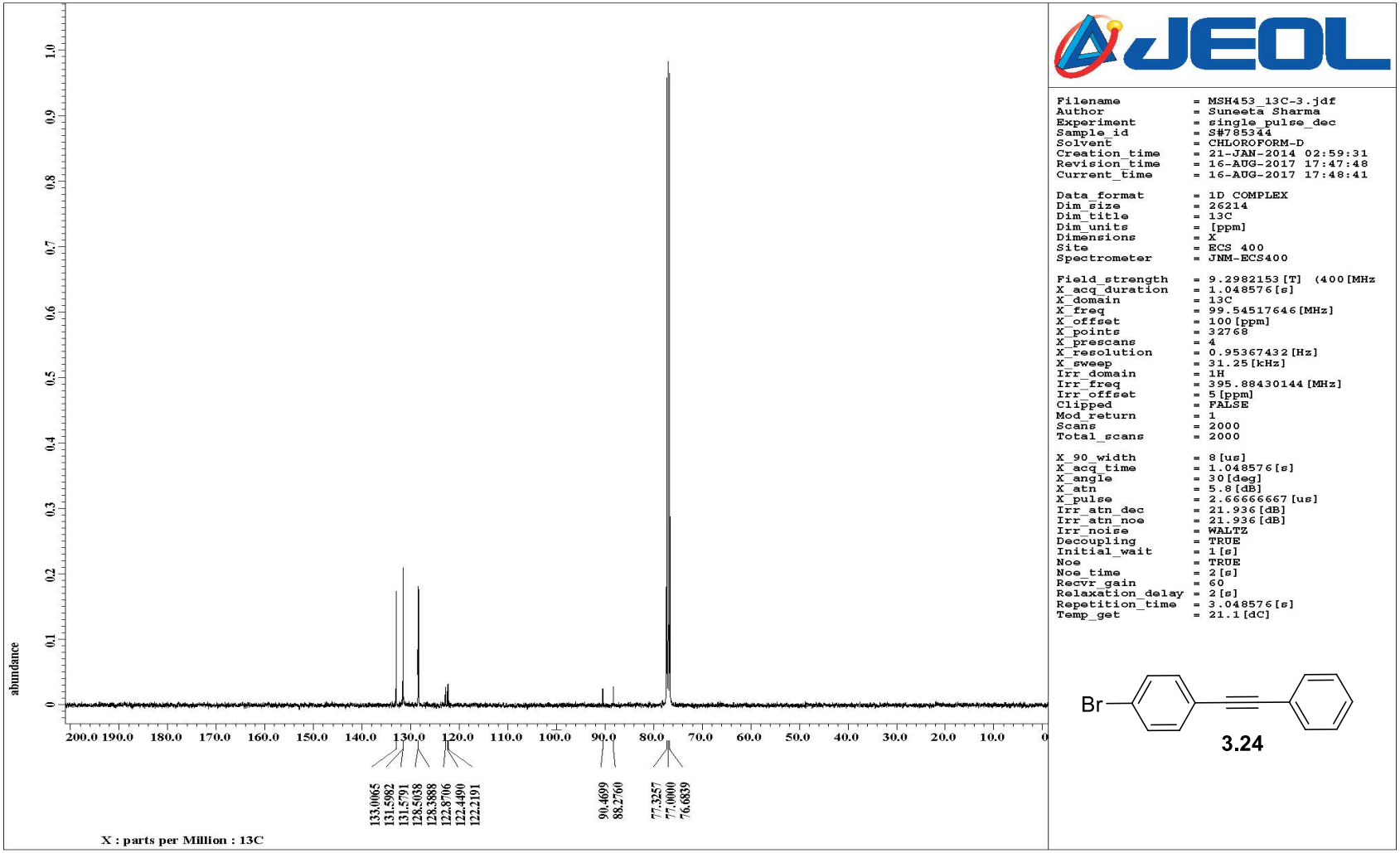
^{13}C NMR Spectrum of 1-(benzyloxy)-4-((4-chlorophenyl)ethynyl)-2-methoxybenzene **(3.23)**



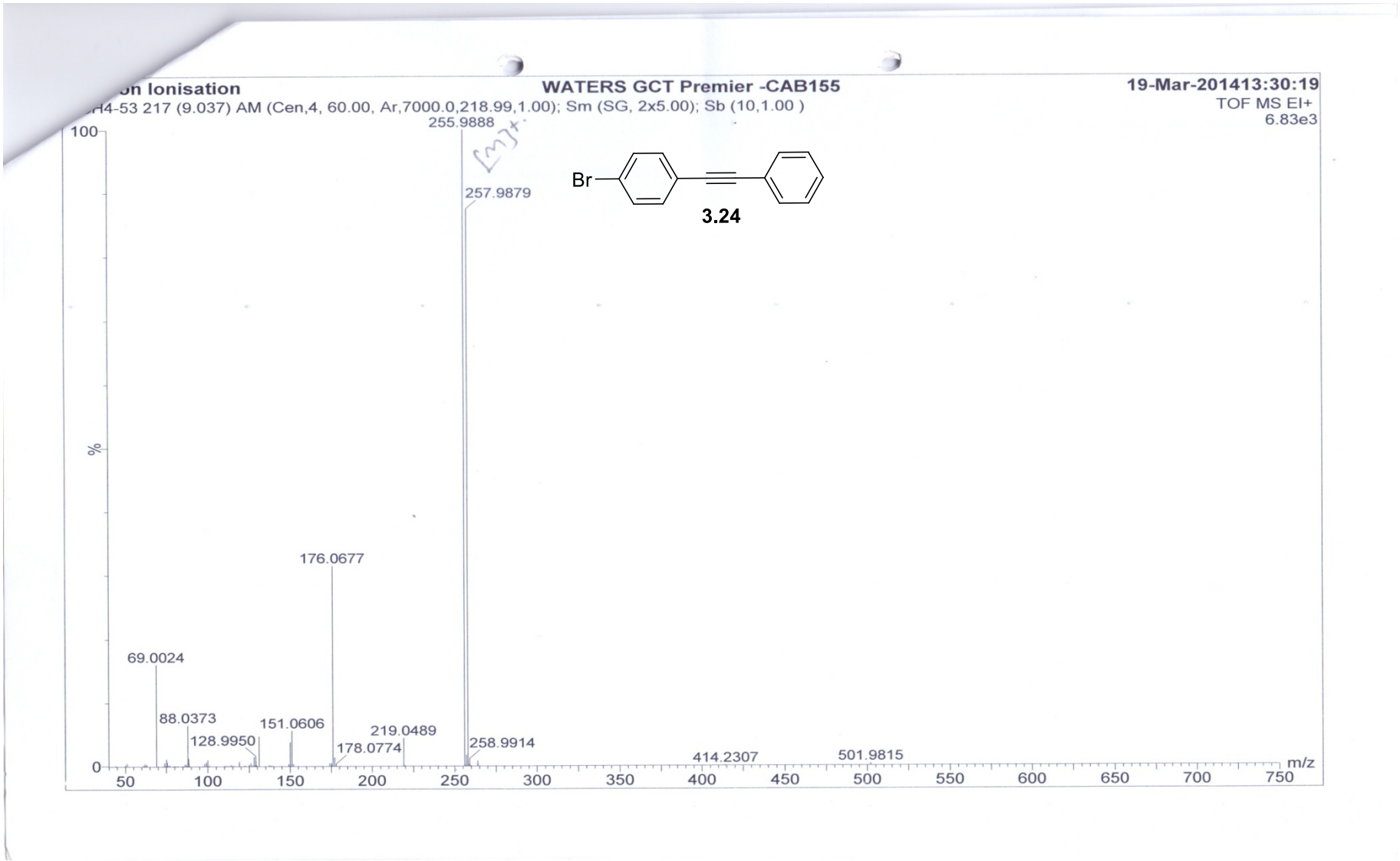
HRMS Spectrum of 1-(benzyloxy)-4-((4-chlorophenyl)ethynyl)-2-methoxybenzene **(3.23)**



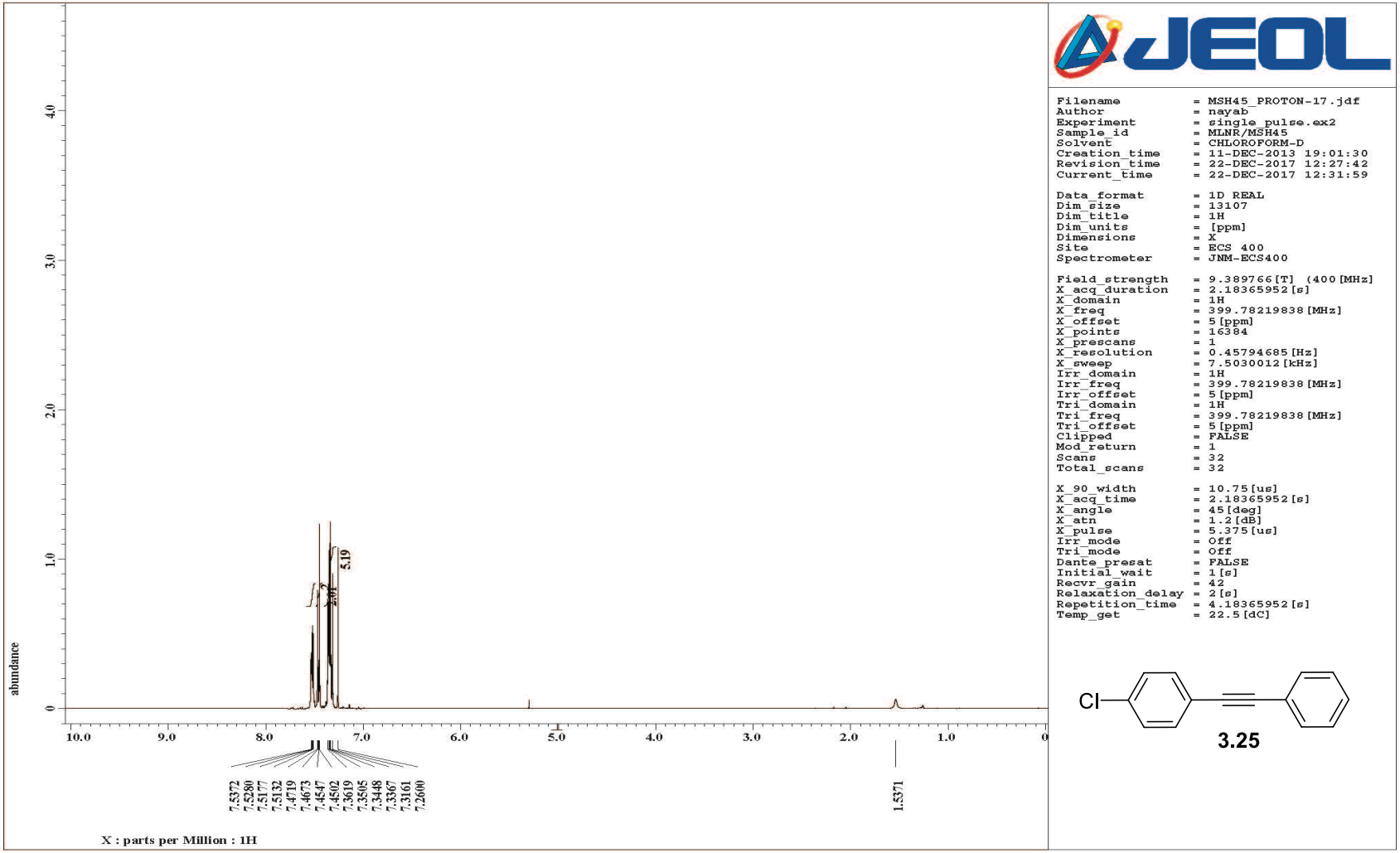
^{1}H NMR Spectrum of 1-bromo-4-(phenylethynyl)benzene **(3.24)**



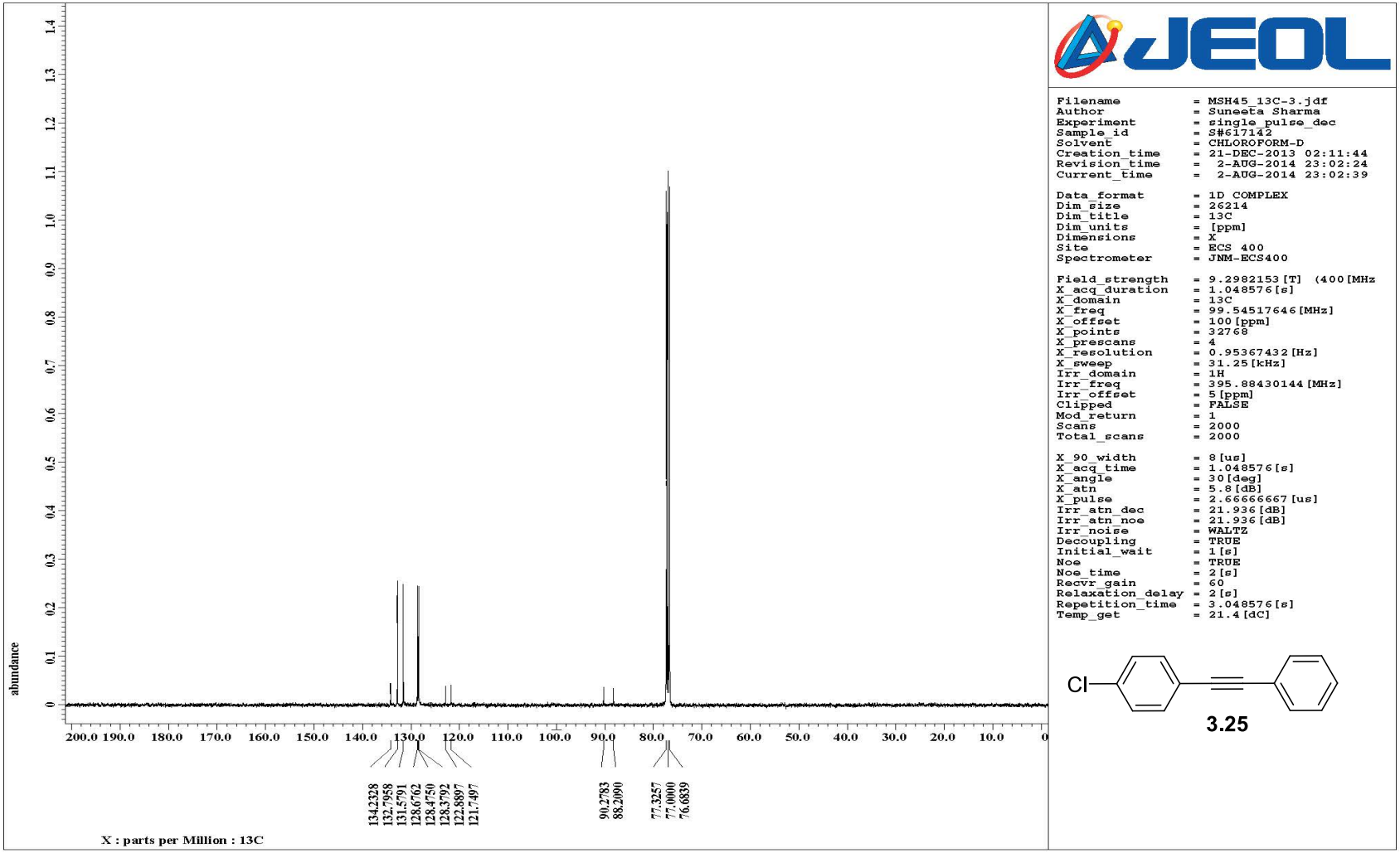
^{13}C NMR Spectrum of 1-bromo-4-(phenylethynyl)benzene **(3.24)**



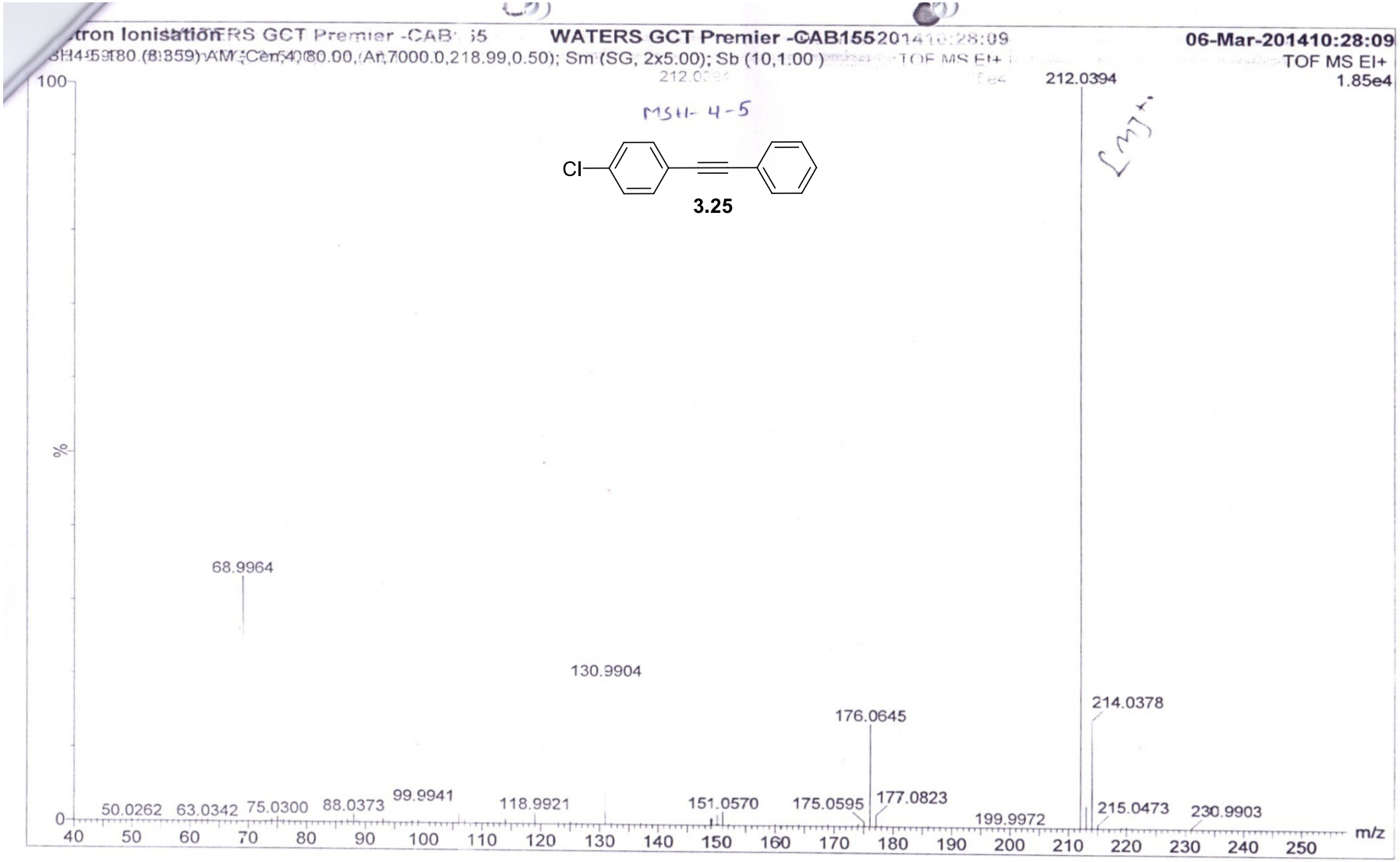
HRMS Spectrum of 1-bromo-4-(phenylethynyl)benzene **(3.24)**



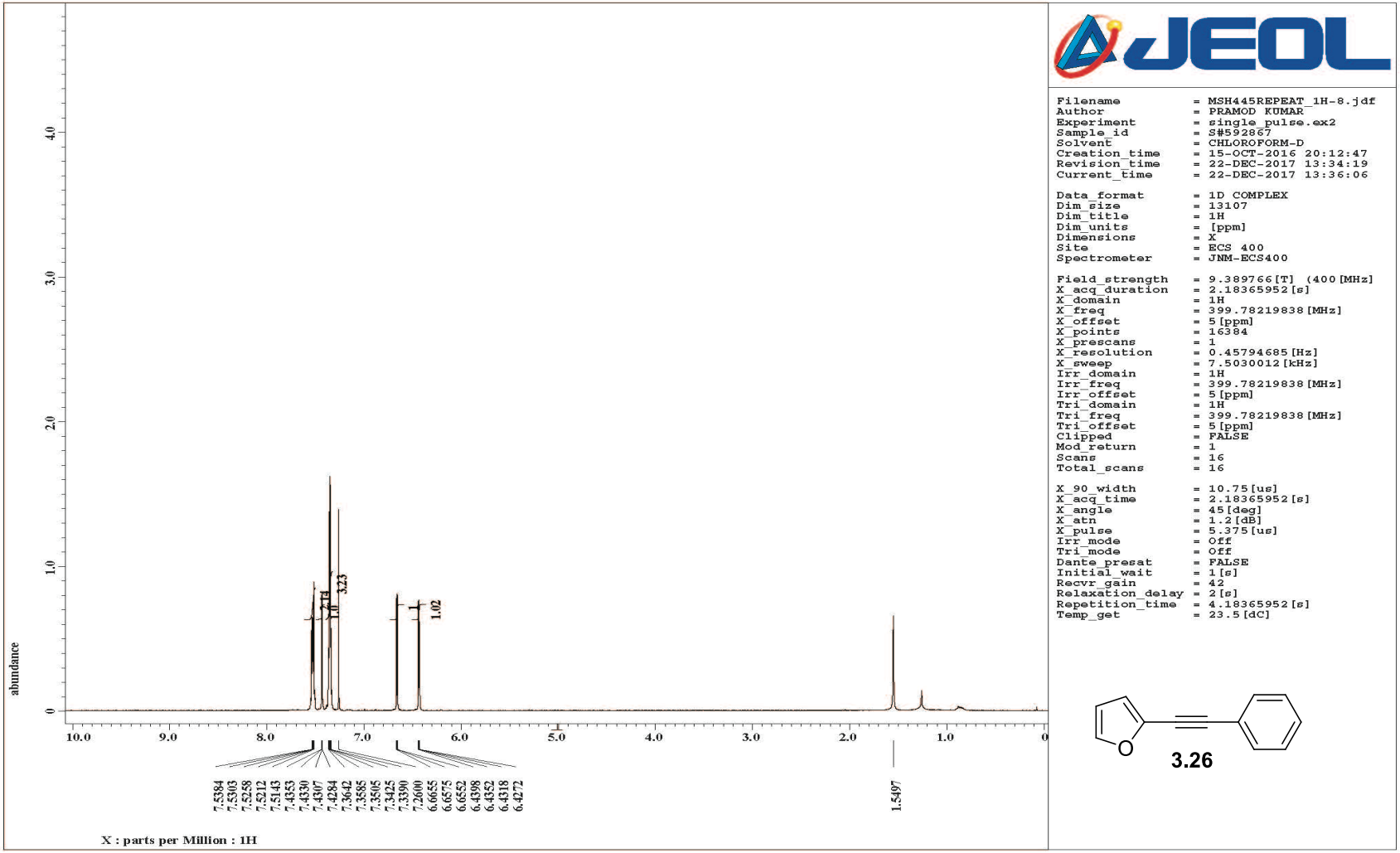
^{1}H NMR Spectrum of 1-chloro-4-(phenylethynyl)benzene **(3.25)**



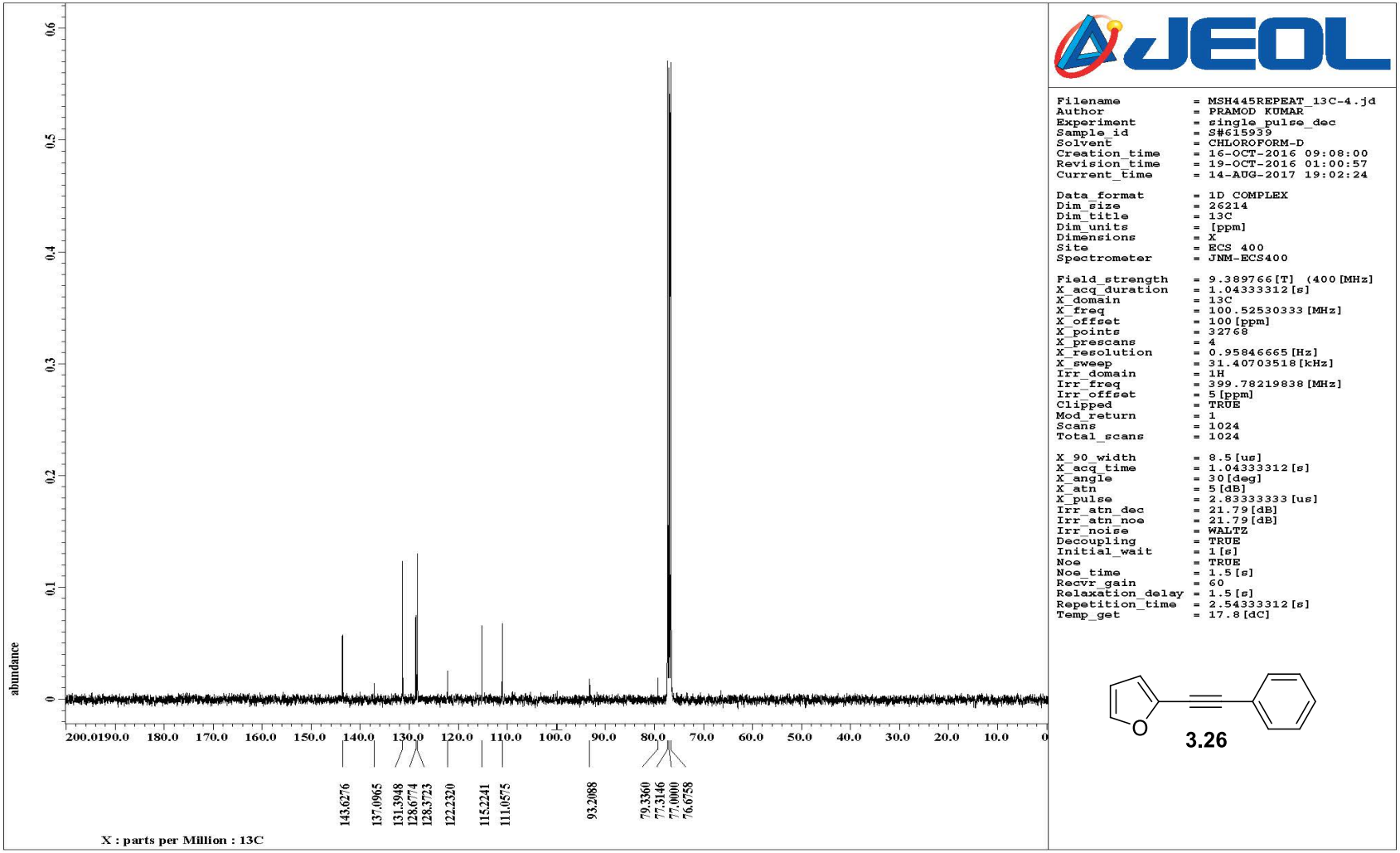
^{13}C NMR Spectrum of 1-chloro-4-(phenylethynyl)benzene **(3.25)**



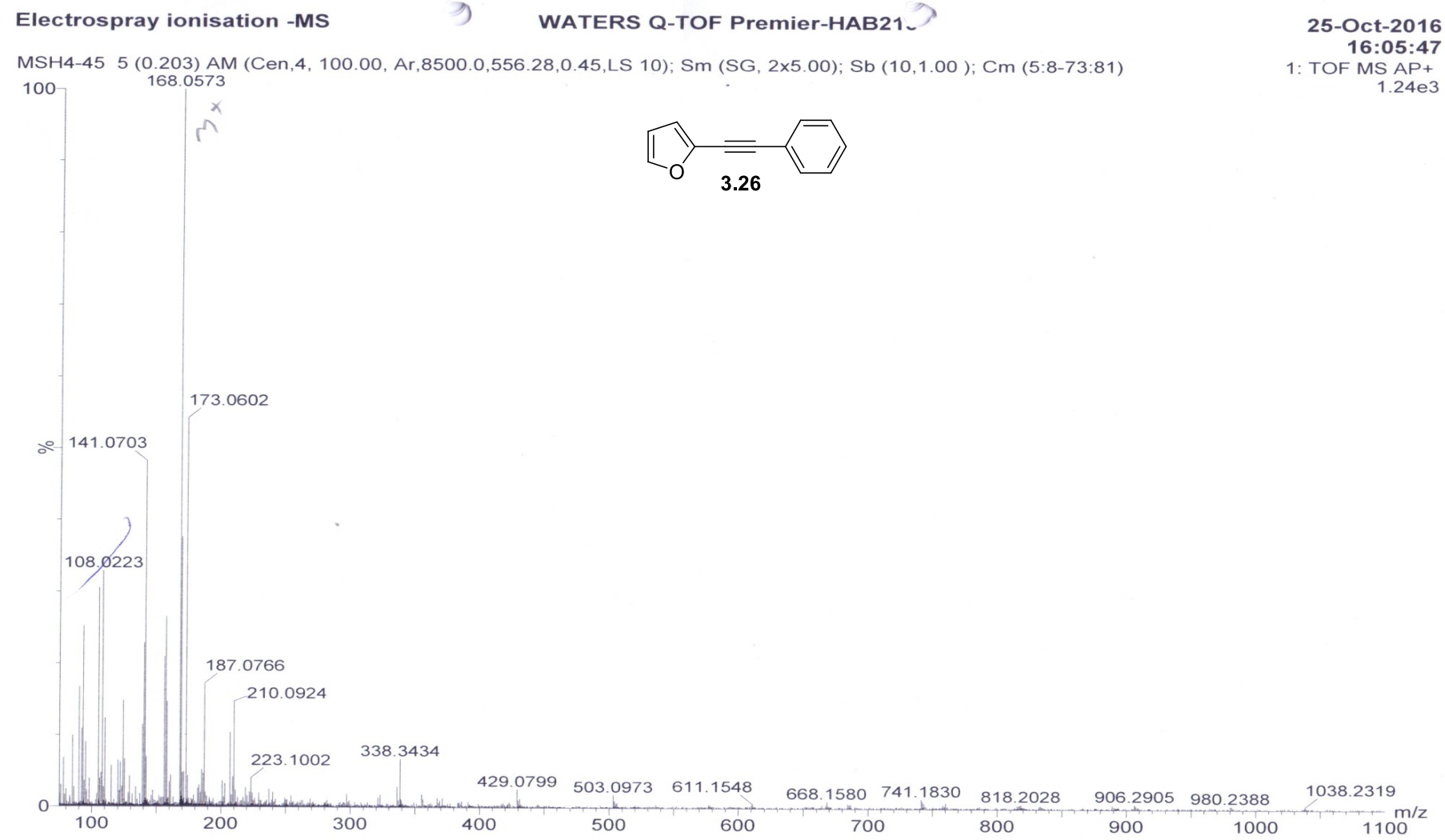
HRMS Spectrum of 1-chloro-4-(phenylethynyl)benzene **(3.25)**



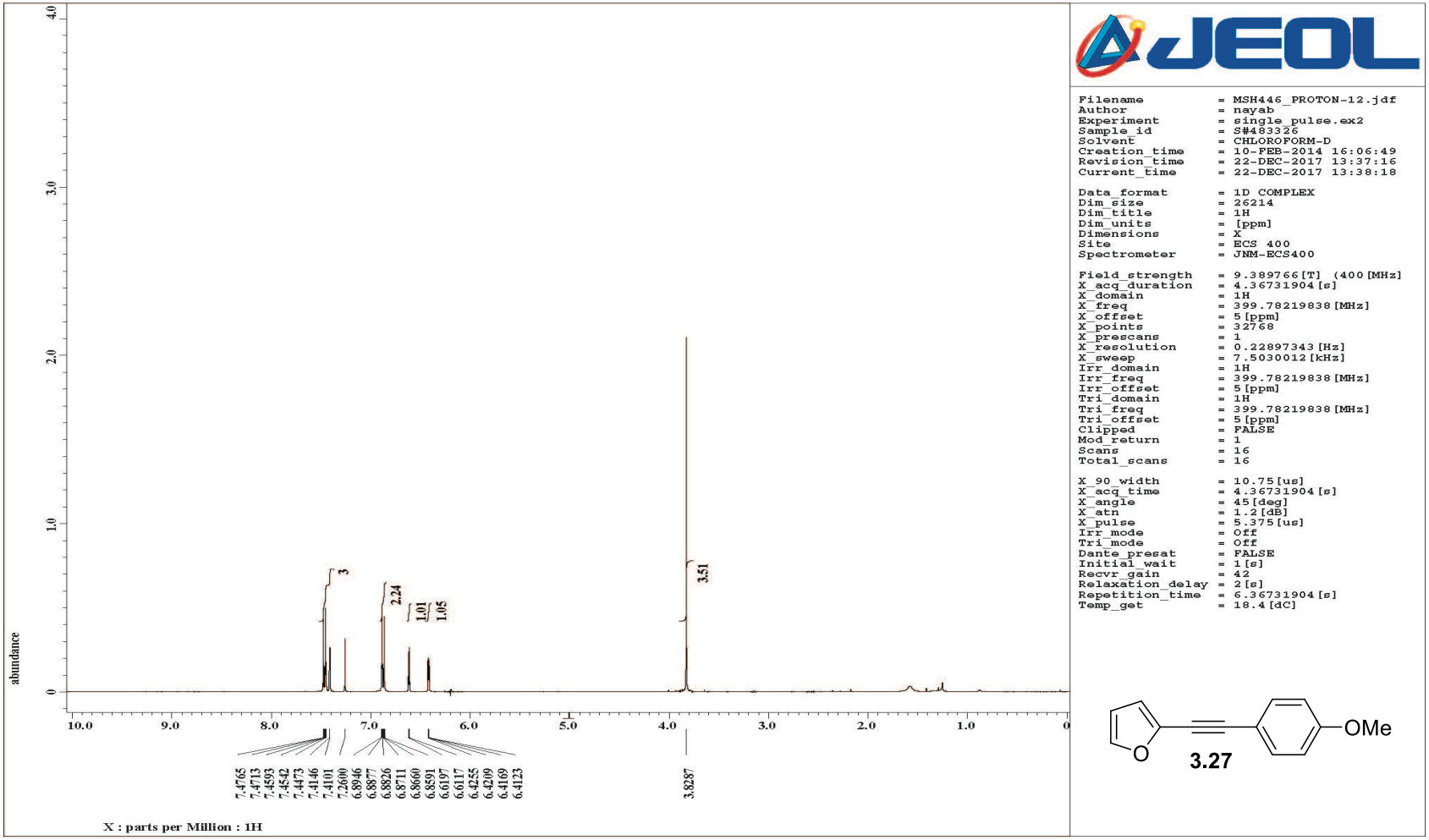
^{1}H NMR spectrum of 2-(phenylethynyl)furan **(3.26)**



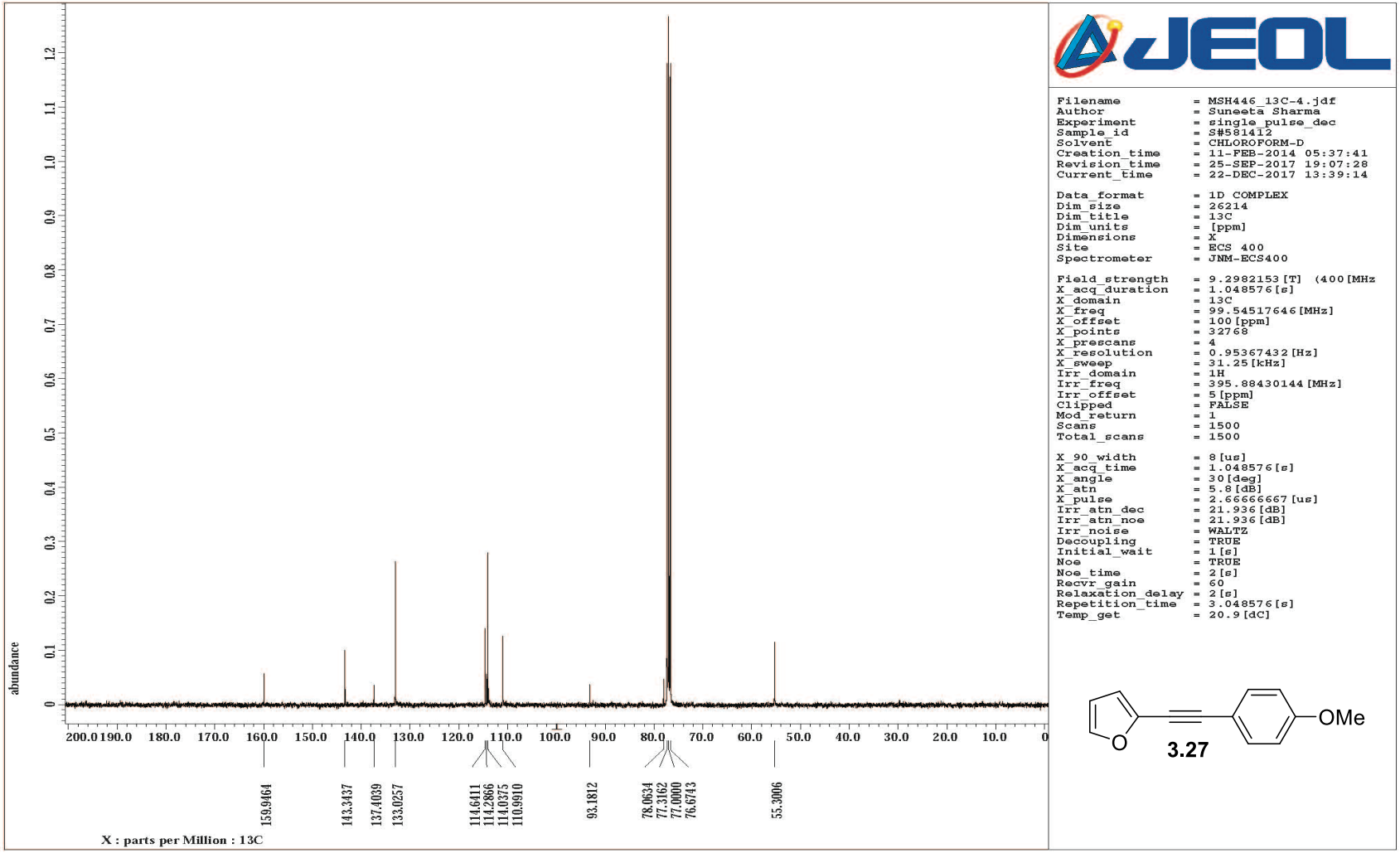
^{13}C NMR spectrum of 2-(phenylethynyl)furan **(3.26)**



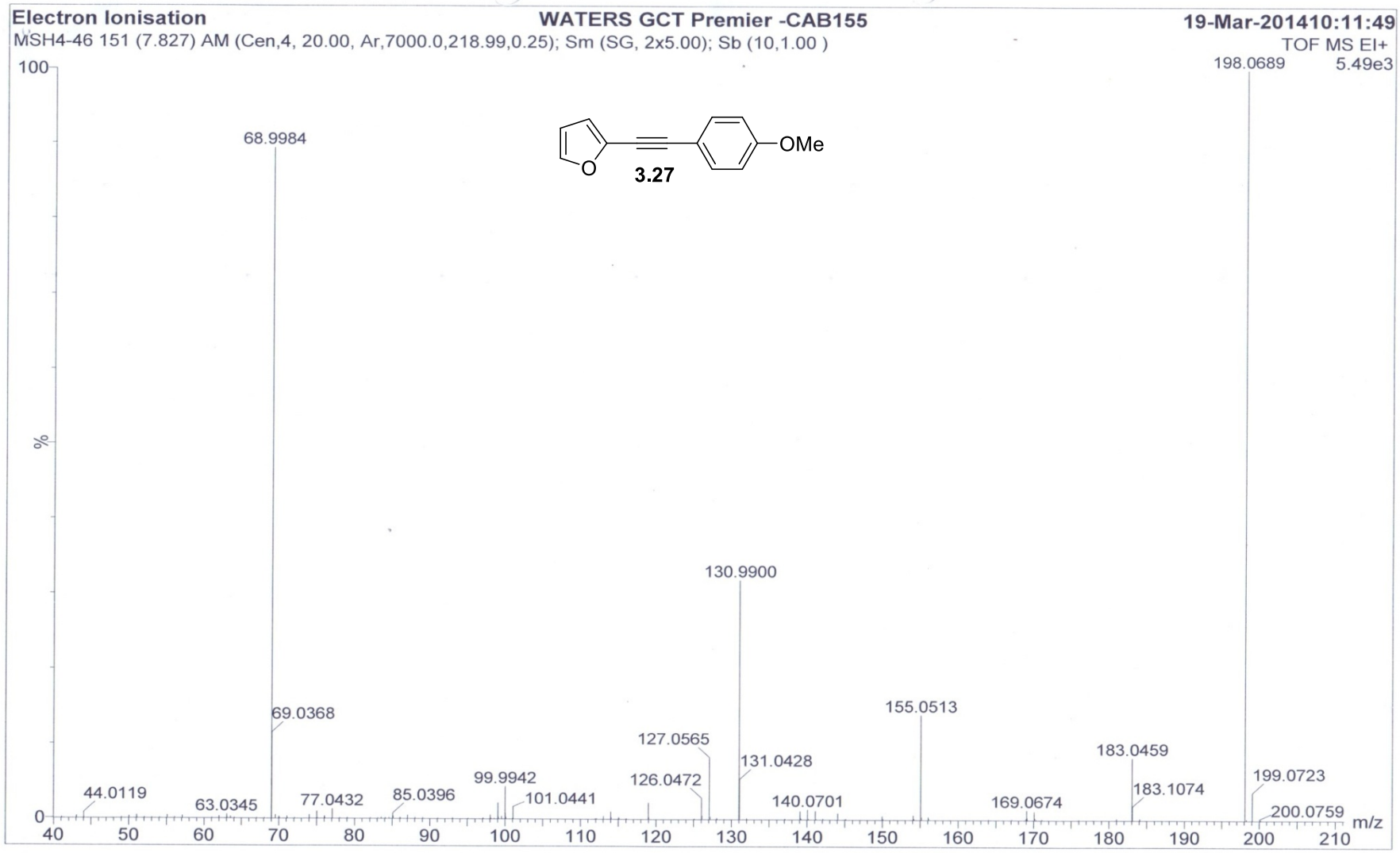
HRMS spectrum of 2-(phenylethynyl)furan **(3.26)**



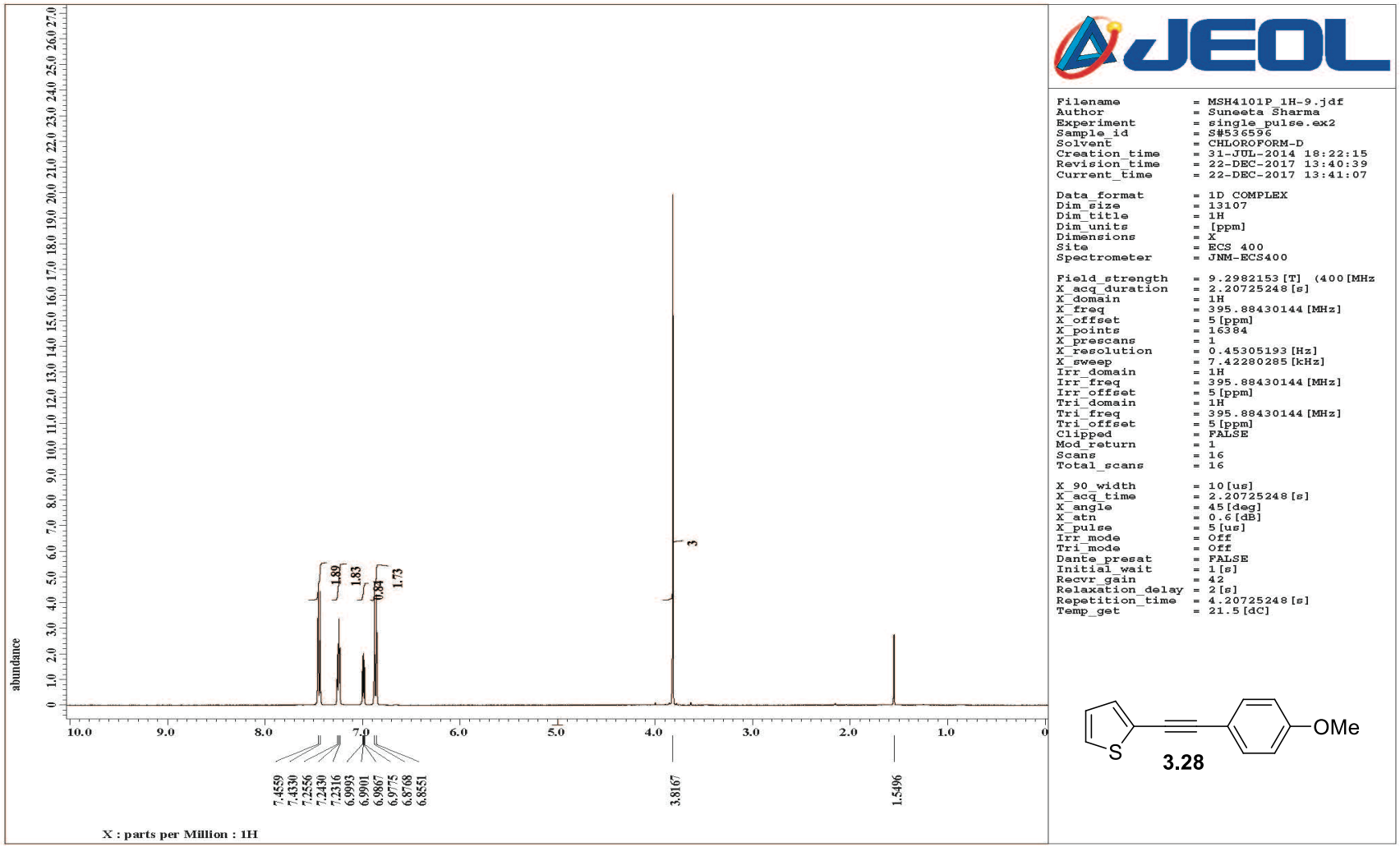
^{1}H NMR spectrum of 2-((4-methoxyphenyl)ethynyl)furan **(3.27)**



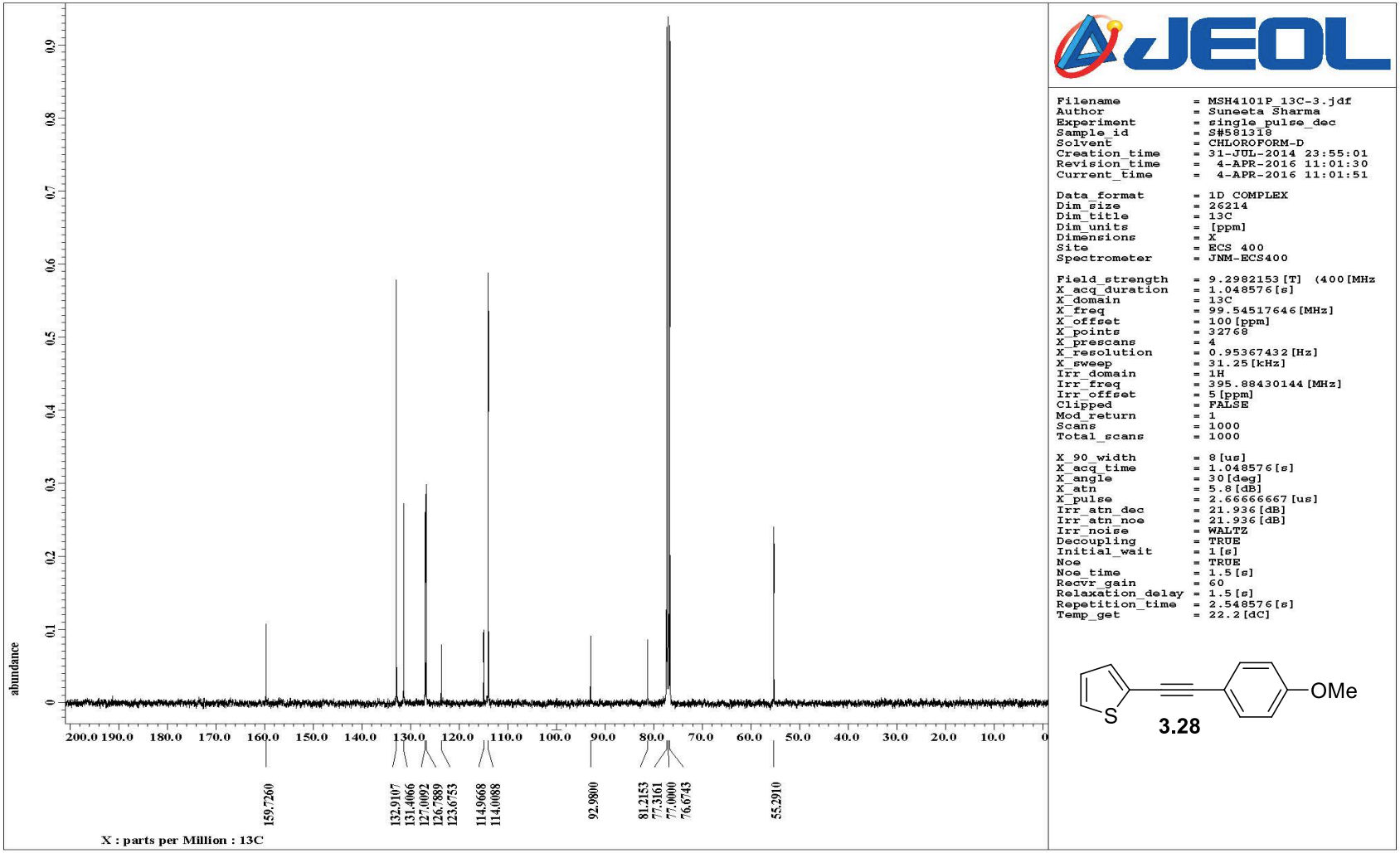
^{13}C NMR spectrum of 2-((4-methoxyphenyl)ethynyl)furan **(3.27)**



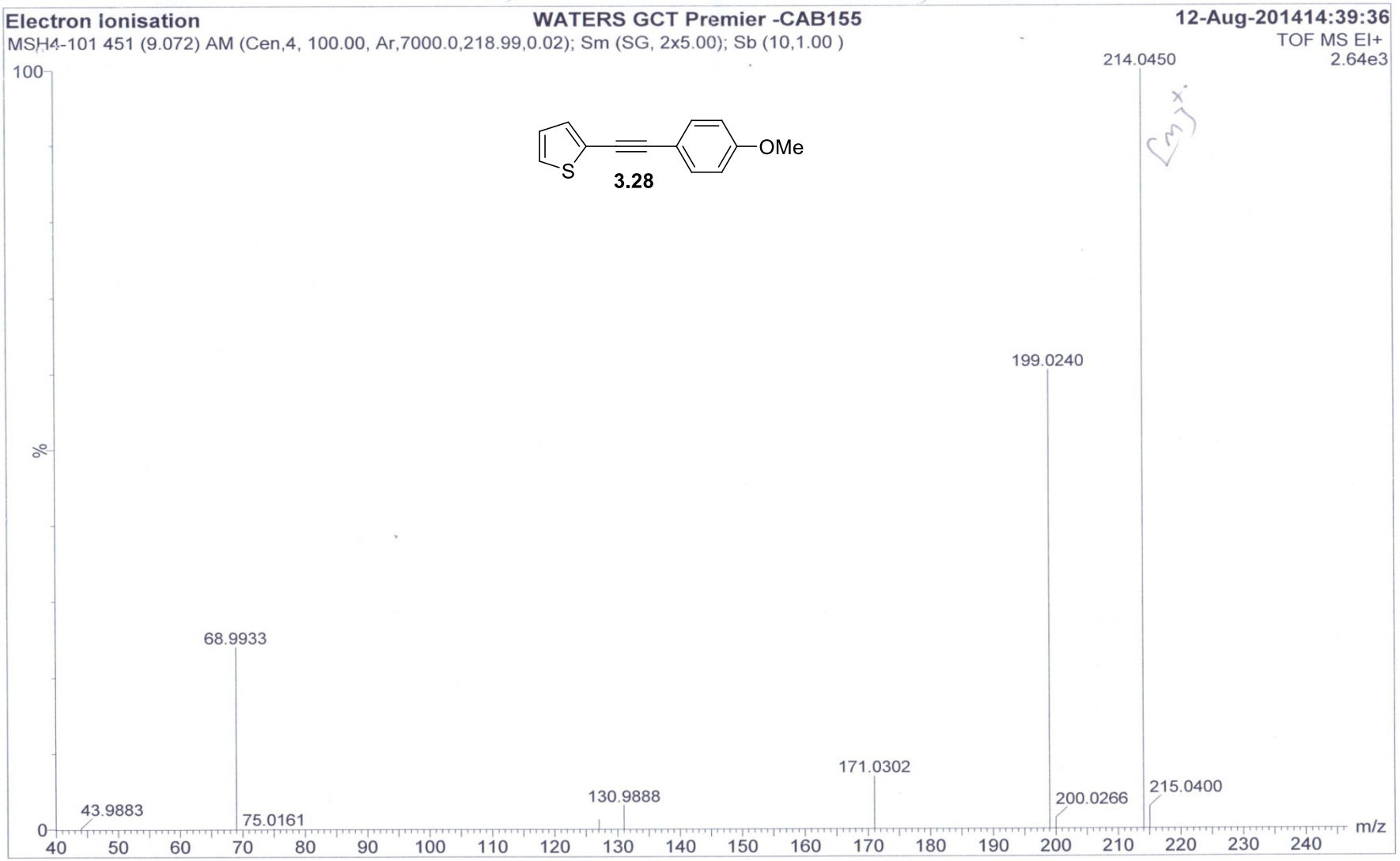
HRMS spectrum of 2-((4-methoxyphenyl)ethynyl)furan **(3.27)**



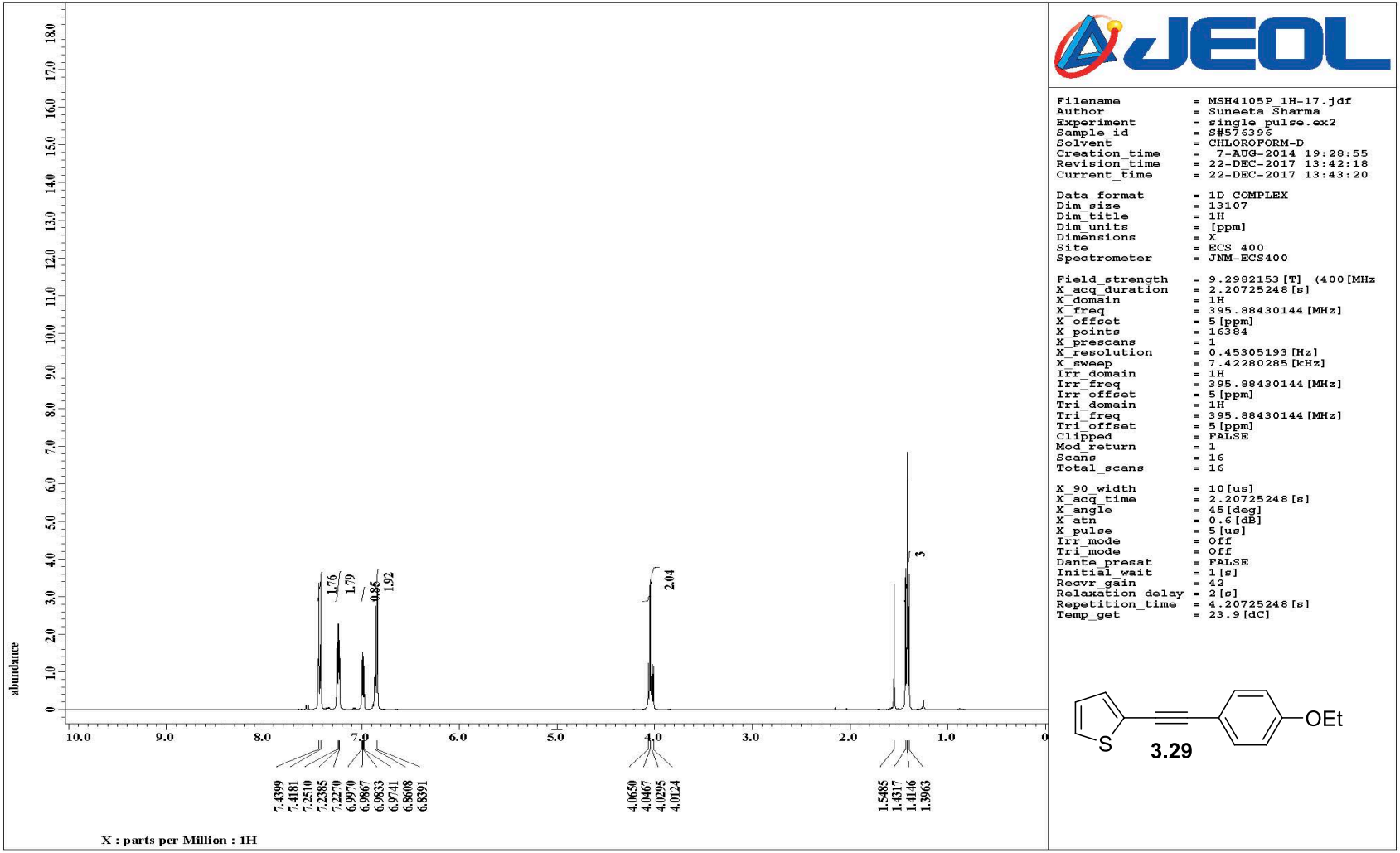
^{1}H NMR Spectrum of 2-((4-methoxyphenyl)ethynyl)thiophene **(3.28)**



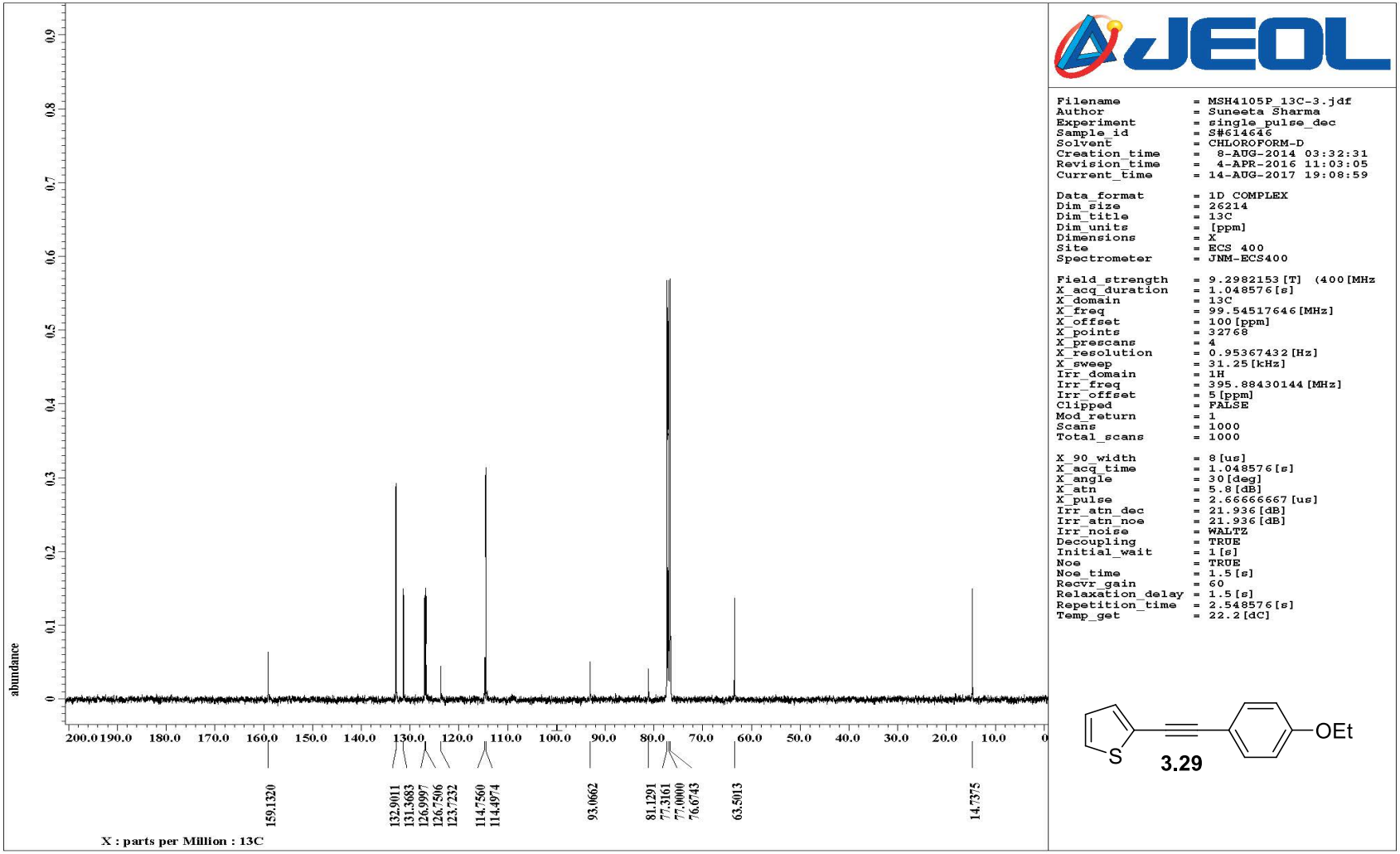
^{13}C NMR Spectrum of 2-((4-methoxyphenyl)ethynyl)thiophene **(3.28)**



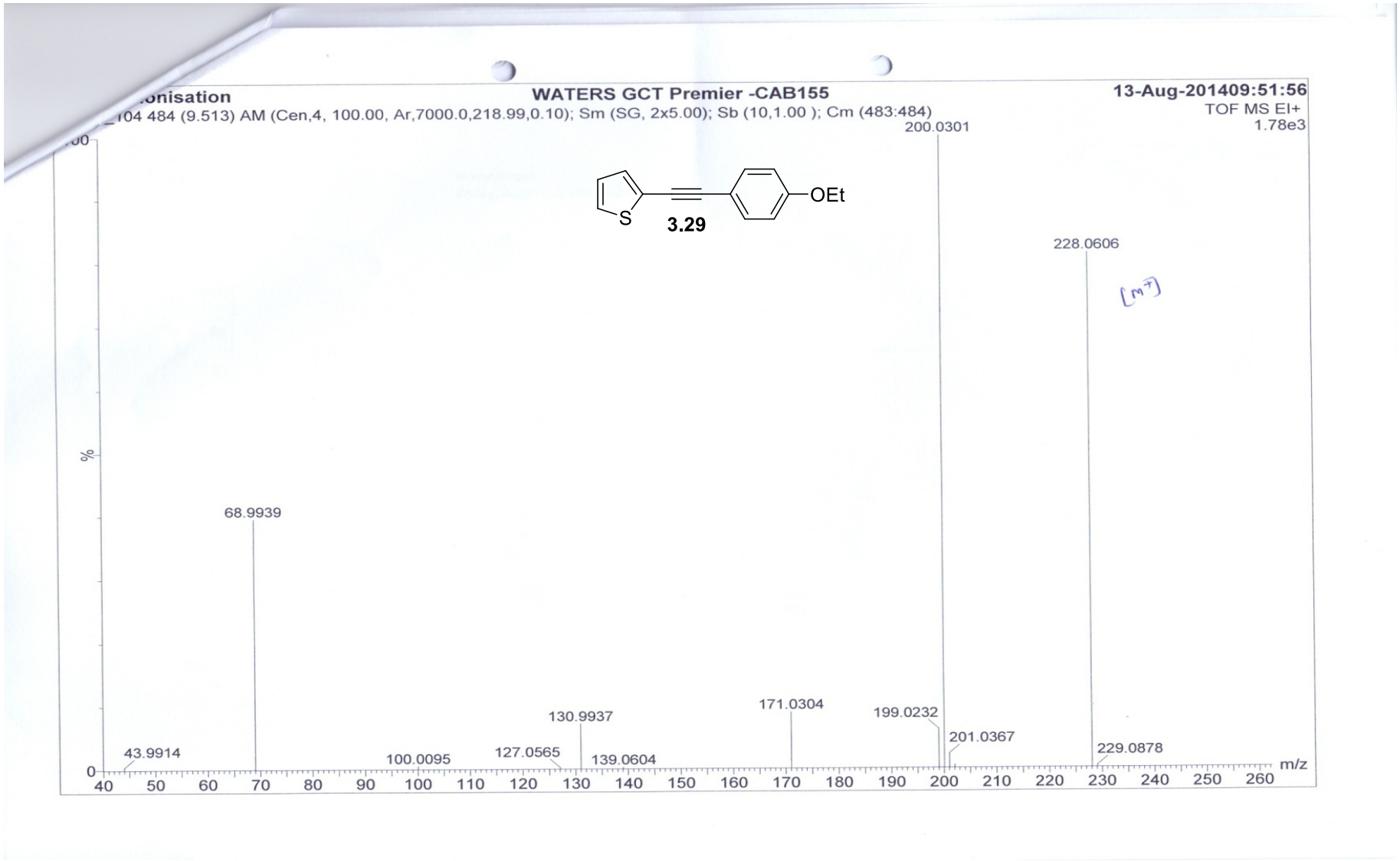
HRMS Spectrum of 2-((4-methoxyphenyl)ethynyl)thiophene **(3.28)**



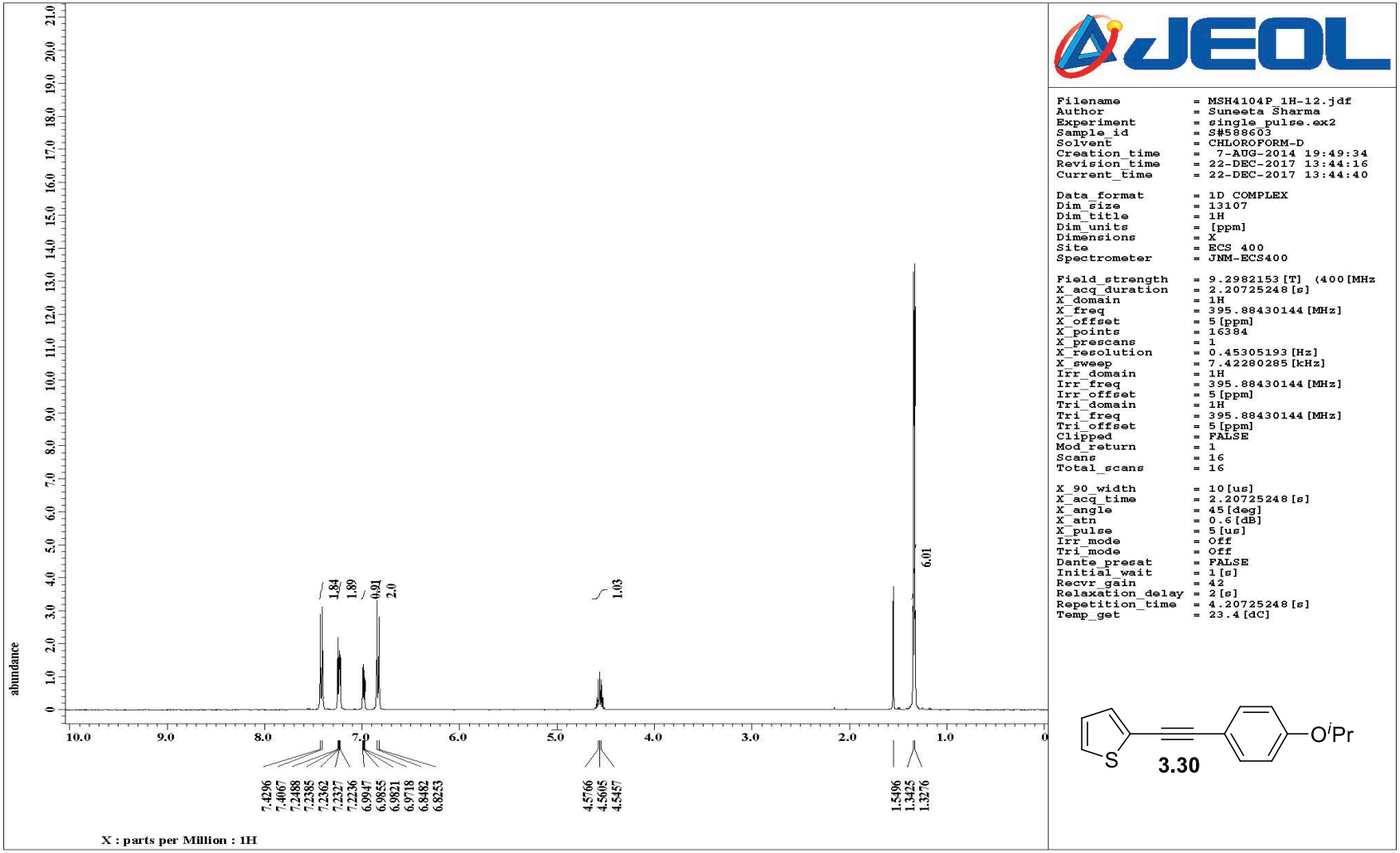
^{1}H NMR Spectrum of 2-((4-ethoxyphenyl)ethynyl)thiophene **(3.29)**



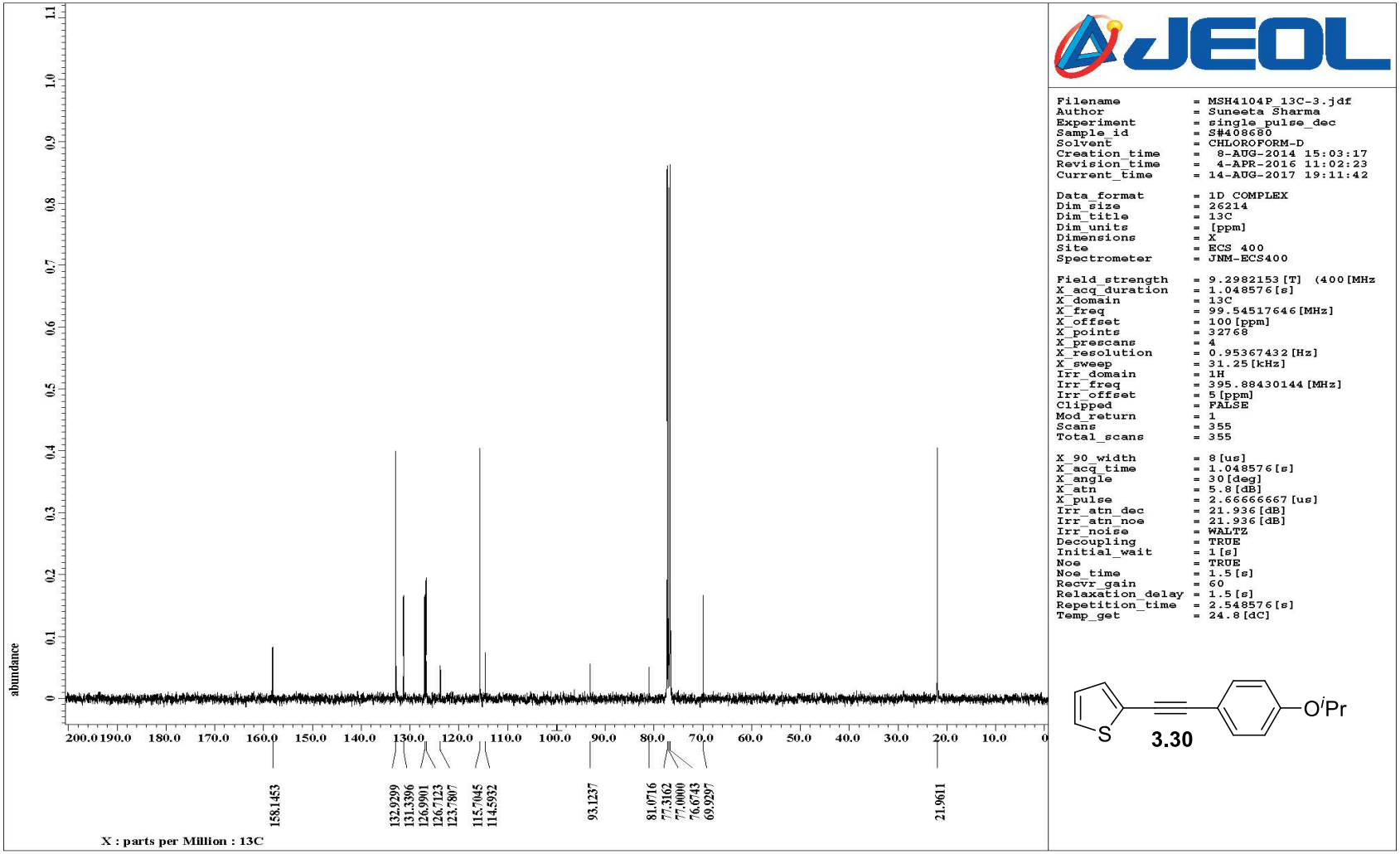
^{13}C NMR Spectrum of 2-((4-ethoxyphenyl)ethynyl)thiophene **(3.29)**



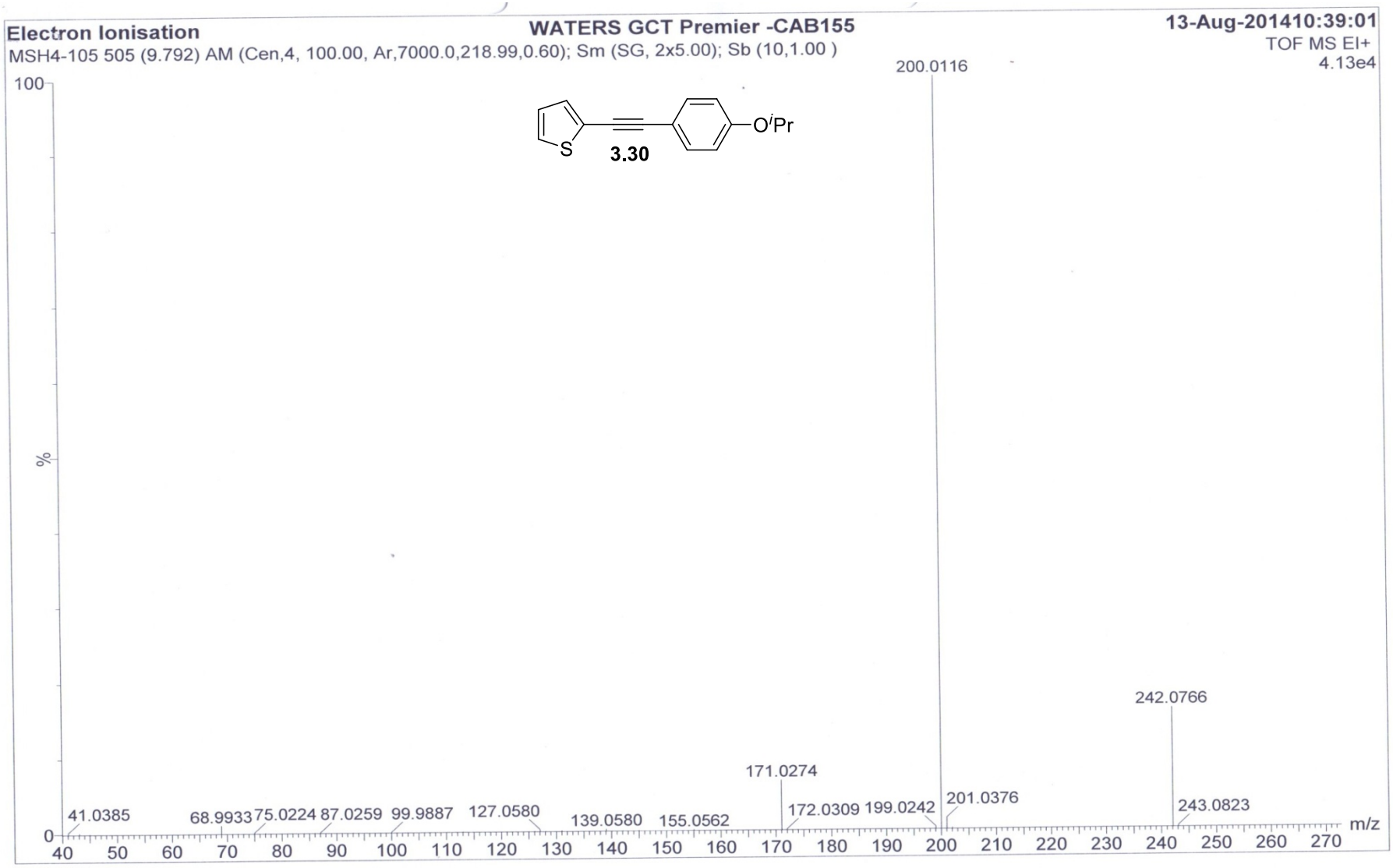
HRMS Spectrum of 2-((4-ethoxyphenyl)ethynyl)thiophene **(3.29)**



^{1}H NMR Spectrum of 2-((4-isopropoxyphenyl)ethynyl)thiophene **(3.30)**



^{13}C NMR Spectrum of 2-((4-isopropoxyphenyl)ethynyl)thiophene **(3.30)**



HRMS Spectrum of 2-((4-isopropoxyphenyl)ethynyl)thiophene **(3.30)**