

Supporting Information

Functional group manoeuvring for tuning stability and reactivity: Synthesis of Cicerfuran, Moracin (D, E, M) and Chromene fused benzofuran natural products

Maddali L. N. Rao,* Venneti N. Murty, Sachchida Nand

Department of Chemistry, Indian Institute of Technology Kanpur, Kanpur-208016, India

maddali@iitk.ac.in

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1. General information: All cross-coupling reactions were performed in an oven-dried Schlenk tubes under N₂ atmosphere conditions. All other reactions were performed under anhydrous conditions using dry solvents.

2. Experimental procedures:

Compound 11:¹

A solution of compound **10** (3 g, 21.72 mmol) and DMAP (0.265 g, 2.17 mmol) in 100 mL of CH₂Cl₂ was taken in a 250 mL RB flask at 0 °C. To this, NEt₃ (2.42 g, 23.89 mmol) was added initially followed by the slow addition of acetyl chloride (1.87 g, 23.89 mmol). The reaction mixture was stirred at rt for 2 h and then poured into cold water. The organic layer was separated and washed with water followed by brine. It was dried over anhydrous MgSO₄, concentrated and purified by silica gel column chromatography (EtOAc/hexane, 10:90) to give compound **11** (2.1 g, 54%) as colourless solid, m.p. 56-58 °C.

¹H NMR (500 MHz, CDCl₃): δ = 11.22 (s, 1H), 9.85 (s, 1H), 7.57 (d, *J* = 8.55 Hz, 1H), 6.78 (dd, *J* = 8.25, 2.15 Hz, 1H), 6.75 (d, *J* = 1.8 Hz, 1H), 2.32 (s, 3H) ppm. ¹³C NMR (125 MHz, CDCl₃): δ = 195.47, 168.35, 163.05, 157.21, 134.93, 118.60, 113.87, 110.66, 21.15 ppm. IR (KBr): $\tilde{\nu}$ = 3214, 2941, 2857, 1752, 1663, 1625, 1586, 1502, 1444, 1378, 1303, 1198, 1145, 1117 cm⁻¹. HRMS (EI): calcd for C₉H₈O₄ [M]⁺ 180.0423; found 180.0424.

General procedure A for *ortho-gem*-dibromovinylphenols (Compounds 12b, 12c and 22)

A solution of CBr₄ (3 equiv) in CH₂Cl₂ (1.0 mL per mmol CBr₄) was put into a 250 mL round-bottomed flask and cooled to 0 °C. To this, a solution of PPh₃ (6 equiv) in CH₂Cl₂ (1.0 mL per mmol PPh₃) was added dropwise for about 15 minutes time period. The resulting mixture was stirred for 30 min and thereafter triethylamine (6 equiv) was added and stirred for 5 minutes. To this, a solution of salicylaldehyde derivative (1 equiv) in CH₂Cl₂ (1.0 mL per mmol salicylaldehyde) was added dropwise in 30 minutes time period. The resulting mixture was brought to rt and

stirred for additional 14 h. To this, hexane (one fourth volume of total CH₂Cl₂ was used) was added and stirred for 15 minutes. The resulting suspension was filtered through 100–200 silica and the filtrate was concentrated. The crude product was subjected to silica gel column chromatography to obtain *ortho-gem*-dibromovinylphenols.

Compound 12b:²

Following the general procedure A, reaction of compound **11b** (2 g, 8.76 mmol), CBr₄ (8.71 g, 26.28 mmol), PPh₃ (13.78 g, 52.57 mmol), NEt₃ (5.32 g, 52.57 mmol) gave **compound 12b** as a pale yellow solid (1.5 g, 50%), m.p. 88-90 °C.

¹H NMR (500 MHz, CDCl₃): δ = 7.52 (d, J = 8.73 Hz, 1H), 7.49 (s, 1H), 7.43-7.34 (m, 5H), 6.59 (dd, J = 8.73, 2.43 Hz, 1H), 6.44 (d, J = 2.43 Hz, 1H), 5.04 (s, 2H) ppm. ¹³C NMR (125 MHz, CDCl₃): δ = 160.19, 153.72, 136.47, 131.78, 129.91, 128.64, 128.12, 127.46, 115.83, 107.25, 102.40, 90.42, 70.11 ppm. IR (KBr): $\tilde{\nu}$ = 3393, 3031, 2943, 1610, 1513, 1435, 1299, 1222, 1109, 1176, 1015, 852, 735 cm⁻¹. HRMS (ESI): calcd for C₁₅H₁₃Br₂O₂ [M+H]⁺ 382.9282; found 382.9282.

Compound 12c:

Following the general procedure A, reaction of compound **11** (1.2 g, 6.66 mmol), CBr₄ (6.62 g, 19.98 mmol), PPh₃ (10.48 g, 39.96 mmol), NEt₃ (4.04 g, 39.96 mmol) gave **compound 12c** as a colourless solid (1.63 g, 73%), m.p. 120-122 °C.

¹H NMR (400 MHz, DMSO-*d*₆): δ = 10.31 (s, 1H), 7.59-7.57 (m, 2H), 6.63-6.60 (m, 2H), 2.25 (s, 3H) ppm. ¹³C NMR (100 MHz, DMSO-*d*₆): δ = 168.87, 155.61, 151.36, 132.52, 128.97, 119.78, 112.21, 108.97, 88.98, 20.89 ppm. IR (KBr): $\tilde{\nu}$ = 3316, 3022, 1718, 1605, 1513, 1430, 1366, 1257, 1149, 1100, 1018, 844 cm⁻¹. HRMS (ESI): calcd for C₁₀H₇Br₂O₃ [M-H]⁻ 332.8762; found 332.8766.

Compound 22:

Following the general procedure A, reaction of compound **20** (1.2 g, 5.82 mmol), CBr₄ (5.79 g, 17.47 mmol), PPh₃ (9.16 g, 34.93 mmol), NEt₃ (3.53 g, 34.93 mmol) gave **compound 22** as an off-white solid (1.2 g, 57%), m.p. 78-81 °C.

^1H NMR (400 MHz, CDCl_3): δ = 7.50 (s, 1H), 7.36 (s, 1H), 6.23 (s, 1H), 4.86 (s, 1H), 2.71 (t, J = 6.84 Hz, 2H), 1.79 (t, J = 6.84 Hz, 2H), 1.33 (s, 6H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ = 155.27, 152.05, 131.87, 129.51, 114.98, 113.42, 103.73, 88.87, 74.97, 32.82, 26.86, 21.74 ppm. IR (KBr): $\tilde{\nu}$ = 3335, 2975, 2931, 1620, 1590, 1501, 1387, 1318, 1192, 1104, 902 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{13}\text{H}_{15}\text{Br}_2\text{O}_2$ $[\text{M}+\text{H}]^+$ 360.9439; found 360.9435.

General procedure B for acetyl protected *ortho-gem*-dibromovinylphenols (Compounds 12ba, 12ca, 22a and 22b)

A solution of CBr_4 (2 equiv) and acetyl protected salicylaldehyde derivative (1 equiv) in CH_2Cl_2 (1.0 mL per each mmol) was put into a 250 mL round-bottomed flask, and cooled to 0 °C. To this, a solution of PPh_3 (4 equiv) in CH_2Cl_2 (1.0 mL per mmol PPh_3) was added dropwise for about 30 minutes time period. After complete addition, the mixture was brought to rt and stirred for 14 h. Then hexane (one fourth volume of total CH_2Cl_2 was used) was added with stirring for 15 minutes. The resulting suspension was filtered through 100–200 silica and the filtrate was concentrated. The crude product was purified by silica gel column chromatography to obtain the corresponding *gem*-dibromide.

Compound 12ba

Following the general procedure B, reaction of compound **11c** (1.5 g, 5.5 mmol), CBr_4 (3.7 g, 11.1 mmol), PPh_3 (5.8 g, 22.2 mmol) gave **compound 12ba** as a pale yellow solid (1.95 g, 83%), m.p. 46-48 °C.

^1H NMR (500 MHz, CDCl_3): δ = 7.64 (d, J = 8.59 Hz, 1H), 7.41-7.35 (m, 5H), 7.29 (s, 1H), 6.88 (dd, J = 8.59, 2.29 Hz, 1H), 6.75 (d, J = 2.29 Hz, 1H), 5.05 (s, 2H) 2.33 (s, 3H) ppm. ^{13}C NMR (125 MHz, CDCl_3): δ = 168.73, 159.67, 148.89, 136.19, 131.44, 129.95, 128.64, 128.19, 127.53, 121.15, 112.46, 108.99, 91.19, 70.31, 20.89 ppm. IR (KBr): $\tilde{\nu}$ = 3032, 1766, 1613, 1497, 1369, 1199, 1160, 1100 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{17}\text{H}_{18}\text{Br}_2\text{NO}_3$ $[\text{M}+\text{NH}_4]^+$ 441.9653; found 441.9656.

Compound 12ca

Following the general procedure B, reaction of compound **11a** (1 g, 4.5 mmol), CBr₄ (3 g, 9 mmol), PPh₃ (4.7 g, 18 mmol) gave **compound 12ca** as a pale yellow solid (1.7 g, 82%), m.p. 34-36 °C.

¹H NMR (400 MHz, CDCl₃): δ = 7.66 (d, J = 8.61 Hz, 1H), 7.32 (s, 1H), 7.03 (dd, J = 8.61, 2.27 Hz, 1H), 6.97 (d, J = 2.27 Hz, 1H), 2.32 (s, 3H), 2.29 (s, 3H) ppm. ¹³C NMR (100 MHz, CDCl₃): δ = 168.68, 168.40, 150.90, 148.18, 131.21, 129.78, 126.00, 118.99, 116.12, 92.88, 21.09, 20.88 ppm. IR (KBr): $\tilde{\nu}$ = 3023, 2923, 1769, 1650, 1491, 1369, 1192, 1145, 1096, 1014 cm⁻¹. HRMS (ESI): calcd for C₁₂H₁₄Br₂NO₄ [M+NH₄]⁺ 393.9290; found 393.9290.

Compound 22a

Following the general procedure B, reaction of compound **21** (1.5 g, 6.04 mmol), CBr₄ (4 g, 12.08 mmol), PPh₃ (6.34 g, 24.17 mmol) gave **compound 22a** as a colourless solid (2 g, 82%), m.p. 60-62 °C.

¹H NMR (400 MHz, CDCl₃): δ = 7.42 (s, 1H), 7.25 (s, 1H), 6.50 (s, 1H), 2.76 (t, J = 6.64 Hz, 2H), 2.29 (s, 3H), 1.80 (t, J = 6.64 Hz, 2H), 1.33 (s, 6H) ppm. ¹³C NMR (100 MHz, CDCl₃): δ = 169.05, 155.14, 147.20, 131.69, 129.69, 119.84, 118.74, 110.97, 90.28, 75.13, 32.56, 27.03, 22.19, 20.99 ppm. IR (KBr): $\tilde{\nu}$ = 2930, 2975, 1766, 1617, 1576, 1485, 1369, 1198, 1152, 1098, 1013, 928, 911 cm⁻¹. HRMS (ESI): calcd for C₁₅H₂₀Br₂NO₃ [M+NH₄]⁺ 419.9810; found 419.9815.

Compound 22b

Following the general procedure B, reaction of compound **21a** (1.2 g, 4.87 mmol), CBr₄ (4.85 g, 14.62 mmol), PPh₃ (7.67 g, 29.24 mmol) gave **compound 22b** as a pale yellow liquid (1.5 g, 76%).

¹H NMR (400 MHz, CDCl₃): δ = 7.31 (s, 1H), 7.24 (s, 1H), 6.52 (s, 1H), 6.29 (d, J = 9.62 Hz, 1H), 5.60 (d, J = 9.62 Hz, 1H), 2.30 (s, 3H), 1.44 (s, 6H) ppm. ¹³C NMR (100 MHz, CDCl₃): δ = 168.73, 153.99, 148.37, 131.39, 130.60, 126.38, 121.22, 120.63, 118.85, 110.39, 90.73, 77.11,

28.38, 29.91 ppm. IR (KBr): $\tilde{\nu}$ = 2976, 2932, 1768, 1640, 1613, 1566, 1485, 1366, 1289, 1198, 1164, 913, 787 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{15}\text{H}_{18}\text{Br}_2\text{NO}_3$ $[\text{M}+\text{NH}_4]^+$ 417.9653; found 417.9653.

Compound 13a:

Compound **12b** (0.5 g, 1.30 mmol), CuI (0.012 g, 0.065 mmol), K_3PO_4 (0.552 g, 2.6 mmol) and THF (3 mL) were put into an oven dried Schlenk tube under a nitrogen atmosphere. The mixture was heated in an oil bath at 65 °C for 7 h. The reaction mixture was then cooled to rt and extracted with ethyl acetate. The combined organic extract was washed with water, brine, dried over anhydrous MgSO_4 and concentrated. The crude product was purified by silica gel column chromatography (EtOAc/hexane 03:97) to obtain compound **13a** (0.320 g, 81%) as a pale yellow solid, m.p. 40-42 °C.

^1H NMR (500 MHz, CDCl_3): δ = 7.46-7.45 (m, 2H), 7.42-7.33 (m, 4H), 7.06 (d, J = 2.29 Hz, 1H), 6.95 (dd, J = 8.59, 2.29 Hz, 1H), 6.64 (d, J = 0.86 Hz, 1H), 5.10 (s, 2H) ppm. ^{13}C NMR (125 MHz, CDCl_3): δ = 156.88, 156.46, 136.70, 128.62, 128.06, 127.48, 126.20, 122.26, 120.16, 113.02, 108.01, 97.07, 70.62 ppm. IR (KBr): $\tilde{\nu}$ = 3090, 2902, 1533, 1292, 1035, 911, 825 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{15}\text{H}_{12}\text{BrO}_2$ $[\text{M}+\text{H}]^+$ 303.0021; found 303.0028.

Triarylbismuth reagent (TAB-3):

To a solution of 5-bromo-6-methoxybenzo[d][1,3]dioxole (5 g, 5 equiv) in dry THF (20 mL), *n*-BuLi (14.87 mL, 1.6 M, 5.5 equiv) was added dropwise at -78 °C and stirred for 2 h. The reaction mixture was brought to rt and ZnCl_2 (47.6 mL, 0.5 M in THF) was added and stirred for 30 minutes. To this, a solution of BiCl_3 (1.36 g, 1 equiv) in dry THF (20 mL) was added dropwise in about 30 minutes at 0 °C and stirred at rt for further 18 h. After that, the solvent was evaporated and the crude residue obtained was dissolved in CHCl_3 (100 mL), washed with saturated NaHCO_3 solution followed by brine. It was quickly passed through silica bed and concentrated. The crude product was triturated with hexane (10 mLx2), hexane:ethanol (1:1) (10 mLx2) and the solid obtained was washed with hexane (5 mL). This procedure furnished the triarylbismuth **TAB-3** (1.8 g, 63%) as colourless solid, m.p. 222-224 °C.

C₂₄H₂₁BiO₉ (662.10): calcd C 43.52, H 3.20; found C 43.47, H 3.08%.

¹H NMR (400 MHz, CDCl₃): δ = 6.89 (s, 1H), 6.64 (s, 1H), 5.88 (s, 2H), 3.70 (s, 3H) ppm. ¹³C NMR (100 MHz, CDCl₃): δ = 157.11, 148.52, 145.38, 133.64, 117.62, 100.91, 94.75, 56.62 ppm. IR (KBr): $\tilde{\nu}$ = 2896, 1615, 1592, 1495, 1455, 1443, 1379, 1264, 1189, 1163, 1107, 1038, 943, 927, 869, 817 cm⁻¹.

Compound 13b:

Compound **13a** (0.825 mmol, 0.25 g, 3.3 equiv), **TAB-2** (0.25 mmol, 0.155 g, 1.0 equiv), Cs₂CO₃ (0.75 mmol, 0.244 g, 3.0 equiv), Pd(OAc)₂ (0.025 mmol, 5.6 mg, 0.1 equiv), PPh₃ (0.1 mmol, 26 mg, 0.4 equiv), and NMP (3 mL) were put into an oven-dried Schlenk tube under a nitrogen atmosphere. The mixture was heated in an oil bath at 90 °C for 1 h. The contents were cooled to rt, and extracted with ethyl acetate. The combined organic extract was washed with water, brine, dried over anhydrous MgSO₄ and concentrated. The crude product was purified by silica gel column chromatography (EtOAc/hexane 05:95) to give compound **13 b** (0.219 g, 81%) as a colourless solid, m.p. 76-78 °C.

¹H NMR (500 MHz, CDCl₃): δ = 7.49-7.33 (m, 6H), 7.13 (d, *J* = 2 Hz, 1H), 6.98-6.94 (m, 4H), 6.45 (t, *J* = 2.29 Hz, 1H), 5.13 (s, 2H), 3.87 (s, 6H) ppm. ¹³C NMR (125 MHz, CDCl₃): δ = 161.04, 157.21, 155.67, 155.02, 136.89, 132.38, 128.61, 128.00, 127.49, 122.68, 121.07, 112.77, 102.53, 101.68, 100.59, 97.08, 70.57, 55.46 ppm. IR (KBr): $\tilde{\nu}$ = 2932, 1599, 1572, 1489, 1455, 1204, 1154, 821 cm⁻¹. HRMS (ESI): calcd for C₂₃H₂₁O₄ [M+H]⁺ 361.1440; found 361.1446.

Procedure for domino couplings (compounds 13, 23 and 27):

1,1-Dibromide (0.825 mmol), BiAr₃ (0.250 mmol), Cs₂CO₃ (2.25 mmol), Pd(PPh₃)₄ (0.022 mmol), and DMF (6 mL) were put into an oven-dried Schlenk tube under a nitrogen atmosphere. The mixture was heated in an oil bath at 90 °C for 7 h. The contents were cooled to rt and extracted with ethyl acetate. The organic portion was washed with water, brine, dried over anhydrous MgSO₄, and concentrated. The crude product was purified by silica gel column chromatography to obtain the benzofuran product.

Compound 13:³

Colourless solid (0.130 g, 64%), m.p. 110-112 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.40 (d, J = 8.32 Hz, 1H), 7.01 (d, J = 1.96 Hz, 1H), 6.97 (d, J = 2.44 Hz, 2H), 6.93 (d, J = 0.76 Hz, 1H), 6.78 (dd, J = 8.28, 2.2 Hz, 1H), 6.45 (t, J = 2.32 Hz, 1H), 4.99 (bs, 1H), 3.87 (s, 6H) ppm. ^{13}C NMR (125 MHz, CDCl_3): δ = 161.01, 155.65, 155.02, 153.66, 132.32, 122.75, 121.20, 112.08, 102.57, 101.68, 100.60, 98.24, 55.48 ppm. IR (KBr): $\tilde{\nu}$ = 3399, 2937, 2838, 1574, 1600, 1488, 1461, 1423, 1153, 1204, 1064, 825 cm^{-1} . HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{15}\text{O}_4$ $[\text{M}+\text{H}]^+$ 271.0970; found 271.0972.

Compound 23

Yellow colour solid (0.165 g, 65%), m.p. 104-106 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.21 (s, 1H), 6.96 (d, J = 2.28 Hz, 2H), 6.93 (s, 1H), 6.86 (s, 1H), 6.43 (t, J = 2.30 Hz, 1H), 3.86 (s, 6H), 2.89 (t, J = 6.88 Hz, 2H), 1.85 (t, J = 6.64 Hz, 2H), 1.37 (s, 6H) ppm. ^{13}C NMR (125 MHz, CDCl_3): δ = 161.05, 154.65, 152.40, 132.61, 122.19, 120.24, 117.40, 102.58, 101.31, 100.52, 99.28, 74.42, 55.48, 33.02, 26.91, 22.78 ppm. IR (KBr): $\tilde{\nu}$ = 2934, 2973, 1598, 1573, 1460, 1422, 1353, 1326, 1204, 1155, 1113, 1066, 928, 836 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{21}\text{H}_{23}\text{O}_4$ $[\text{M}+\text{H}]^+$ 339.1596; found 339.1592.

Compound 27:⁴

Pale yellow solid (0.162 g, 70%), m.p. 170-172 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.74 (d, 8.72 Hz, 2H), 7.19 (s, 1H), 6.95 (d, 8.72Hz, 2H), 6.92 (s, 1H), 6.74 (s, 1H), 3.85 (s, 3H), 2.89 (t, J = 6.86 Hz, 2H), 1.84 (t, J = 6.86 Hz, 2H), 1.37 (s, 6H) ppm. ^{13}C NMR (125 MHz, CDCl_3): δ = 159.52, 155.03, 154.49, 151.84, 125.94, 123.79, 122.56, 120.47, 117.07, 114.15, 99.21, 99.07, 74.30, 55.34, 33.05, 26.89, 22.77 ppm. IR (KBr): $\tilde{\nu}$ = 2972, 1504, 1460, 1249, 1180, 1116, 1033, 879, 831, 797 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{20}\text{H}_{21}\text{O}_3$ $[\text{M}+\text{H}]^+$ 309.1490; found 309.1498.

Compound 27a

Pale yellow solid (0.181 g, 69%), m.p. 156-158 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.39 (d, J = 9.16 Hz, 2H), 7.25 (s, 1H), 6.85 (d, J = 9.20 Hz, 2H), 6.53 (s, 1H), 3.82 (s, 3H), 2.74 (t, J = 7.10 Hz, 2H), 2.33 (s, 3H), 1.80 (t, J = 6.88 Hz, 2H), 1.33 (s, 6H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ = 168.99, 159.43, 154.84, 150.56, 133.25, 132.76, 118.87, 115.58, 113.93, 111.04, 108.56, 92.04, 83.18, 75.09, 55.27, 32.42, 26.87,

21.84, 20.86 ppm. IR (KBr): $\tilde{\nu}$ = 3416, 1766, 1616, 1571, 1513, 1249, 1202, 1149, 1119 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{22}\text{H}_{23}\text{O}_4$ $[\text{M}+\text{H}]^+$ 351.1596; found 351.1591.

Compound 13c:⁵

A solution of compound **13** (0.150 g, 0.555 mmol) and DMAP (0.007 g, 0.055 mmol) in 100 mL of CH_2Cl_2 was put in a 250 mL RB flask at 0 °C. To this, NEt_3 (0.084 g, 0.832 mmol) was added followed by the slow addition of acetyl chloride (0.065 g, 0.832 mmol). This reaction mixture was stirred for 2 h at rt and poured in to cold water. The CH_2Cl_2 layer was separated, washed with water and brine solution. The organic layer was dried over anhydrous MgSO_4 and concentrated. The crude obtained was purified by silica gel column chromatography (EtOAc/hexane, 10:90) to obtain compound **13c** (0.161 g, 93%) as colourless solid. m.p. 110–112 °C

^1H NMR (500 MHz, CDCl_3): δ = 7.54 (d, J = 8.25 Hz, 1H), 7.30 (d, J = 1.5 Hz, 1H), 6.99-6.97 (m, 4H), 6.48 (t, J = 2.12 Hz, 1H), 3.87 (s, 6H), 2.34 (s, 3H) ppm. ^{13}C NMR (125 MHz, CDCl_3): δ = 169.78, 161.06, 156.69, 154.56, 147.90, 131.92, 126.98, 120.89, 117.13, 105.11, 102.91, 101.57, 101.04, 55.49, 21.17 ppm. IR (KBr): $\tilde{\nu}$ = 2937, 2839, 1762, 1598, 1573, 1482, 1459, 1431, 1205, 1155, 1132, 1109, 966 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{18}\text{H}_{17}\text{O}_5$ $[\text{M}+\text{H}]^+$ 313.1076; found 313.1074.

Moracin M (1):³

To a solution of compound **13c** (0.100 g, 0.320 mmol) in CH_2Cl_2 (20 mL), BBr_3 (0.240 g, 0.960 mmol) solution in CH_2Cl_2 (5 mL) was added dropwise at –78 °C. The reaction mixture brought to rt and stirred for 24 h. It was then cooled to 0 °C, quenched with water (5 mL), and extracted with ethyl acetate. The organic extract was dried over anhydrous MgSO_4 , and concentrated. The crude product was purified by column chromatography (EtOAc/hexane, 80:20) to give compound **1** (0.063 g, 81%) as a pale yellow solid, m.p. 260-262 °C.

^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ = 9.60 (s, 1H), 9.46 (s, 2H), 7.38 (d, J = 8.24 Hz, 1H), 7.08 (s, 1H), 6.93 (d, J = 1.84 Hz, 1H), 6.74 (dd, J = 8.70, 2.30 Hz, 1H), 6.68 (d, J = 2.28 Hz, 2H), 6.21 (t, J = 2.3 Hz, 1H) ppm. ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$): δ = 158.86, 155.79, 155.33,

154.01, 131.75, 121.22, 120.86, 112.53, 102.68, 102.36, 101.66, 97.54 ppm. IR (KBr): $\tilde{\nu}$ = 3414, 2255, 2128, 1653, 1049, 1025, 1002, 826, 764 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{14}\text{H}_9\text{O}_4$ $[\text{M}-\text{H}]^-$ 241.0501; found 241.0503.

Compound 2:⁶

1,1-Dibromide (0.412 mmol), BiAr_3 (0.125 mmol), Cs_2CO_3 (0.75 mmol), $\text{Pd}(\text{PPh}_3)_4$ (0.11 mmol), CuI (0.025 mmol) and DMF (4 mL) were put into an oven-dried Schlenk tube under a nitrogen atmosphere. The mixture was heated in an oil bath at 90 °C for 7 h. The contents were cooled to rt and Cs_2CO_3 (0.375 mmol) was added again, stirred at 90 °C for additional 3 h. Thereafter, it was cooled to rt and extracted with ethyl acetate. The organic extract was washed with water, brine, dried over MgSO_4 , and concentrated. The crude product was purified by silica gel column chromatography to obtain benzofuran **2** (0.045 g, 42%) as a colourless solid, m.p. 150-152 °C.

^1H NMR (400 MHz, CDCl_3): δ = 7.48 (s, 1H), 7.38 (d, J = 8.72 Hz, 1H), 7.14 (s, 1H), 6.97 (d, J = 1.8 Hz, 1H), 6.75 (dd, J = 8.24, 2.28 Hz, 1H), 6.63 (s, 1H), 5.98 (s, 2H), 4.81 (s, 1H), 3.93 (s, 3H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ = 154.40, 153.15, 152.22, 151.64, 147.93, 141.47, 123.65, 120.95, 112.30, 111.59, 106.08, 104.43, 101.47, 97.91, 94.70, 56.27 ppm. IR (KBr): $\tilde{\nu}$ = 2896, 1615, 1593, 1495, 1444, 1455, 1264, 1189, 1164, 1038, 928 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{16}\text{H}_{12}\text{NaO}_5$ $[\text{M}+\text{Na}]^+$ 307.0582; found 307.0588.

Compound 14:

A solution of compound **13** (0.3 g, 1.11 mmol) and methanesulfonyl chloride (0.190 g, 1.66 mmol) in 100 mL of CH_2Cl_2 were put in a 250 mL RB flask at 0 °C. To this, NEt_3 (0.168 g, 1.66 mmol) was added with stirring for 2 h at rt. Thereafter, CH_2Cl_2 layer was separated and washed with water and brine. The organic layer was dried over anhydrous MgSO_4 , concentrated and purified by silica gel column chromatography (EtOAc/hexane, 30:70) to obtain compound **14** (0.310 g, 81%) as a pale brown solid, m.p. 106-108 °C.

^1H NMR (500 MHz, CDCl_3): δ = 7.57 (d, J = 8.55 Hz, 1H), 7.51 (d, J = 1.55 Hz, 1H), 7.18 (dd, J = 8.55, 2.15 Hz, 1H), 7.01 (s, 1H), 6.99 (d, J = 2.40 Hz, 2H), 6.49 (t, J = 2.3 Hz, 1H), 3.87 (s, 6H), 3.17 (s, 3H) ppm. ^{13}C NMR (125 MHz, CDCl_3): δ = 161.12, 157.65, 154.30, 146.19,

131.52, 128.41, 121.34, 117.42, 105.85, 103.05, 101.37, 55.50, 37.17 ppm. IR (KBr): $\tilde{\nu}$ = 3012, 2938, 2839, 1572, 1598, 1431, 1420, 1331, 1364, 1204, 1156, 1180, 1102, 960, 836.98 cm^{-1} . HRMS (EI): calcd for $\text{C}_{15}\text{H}_{12}\text{O}_6\text{S}$ $[\text{M}]^+$ 348.0668; found 348.0664.

Compound 15:

To a solution of compound **14** (0.3 g, 0.861 mmol) in CH_2Cl_2 (20 mL), BBr_3 (0.863 g, 3.44 mmol) solution in CH_2Cl_2 (5 mL) was added dropwise at -78°C . The reaction mixture brought to rt and stirred for 24 h. The contents were cooled to 0°C , quenched with water (5 mL), and extracted with ethyl acetate. The organic extract was dried over anhydrous MgSO_4 , and concentrated. The crude product was purified by column chromatography (methanol/DCM, 05:95) to obtain compound **15** (0.260 g, 94%) as a brown solid, m.p. $170\text{--}172^\circ\text{C}$.

^1H NMR (400 MHz, $\text{DMSO-}d_6$): δ = 9.54 (s, 2H), 7.70-7.68 (m, 2H), 7.30 (s, 1H), 7.25 (dd, J = 8.56, 2.20 Hz, 1H), 6.76 (d, J = 1.96 Hz, 2H), 6.28 (t, J = 2.20 Hz, 1H), 3.40 (s, 3H) ppm. ^{13}C NMR (125 MHz, $\text{DMSO-}d_6$): δ = 158.96, 157.32, 153.61, 146.19, 130.83, 128.01, 121.59, 118.06, 105.98, 103.63, 102.95, 101.50, 37.22 ppm. IR (KBr): $\tilde{\nu}$ = 3419, 2923, 1614, 1578, 1475, 1428, 1408, 1323, 1179, 1143, 1106, 962, 873, 851, 819 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{15}\text{H}_{13}\text{O}_6\text{S}$ $[\text{M}+\text{H}]^+$ 321.0433; found 321.0439.

Compound 16:

A mixture of compound **15** (0.433 g, 1.352 mmol), **15a** (0.257 g, 1.622 mmol), 3-picoline (0.031 g, 0.337 mmol) and xylene (9 mL) were taken in an oven-dried Schlenk tube under nitrogen atmosphere. It was heated in an oil bath at 130°C for 24 h and then cooled to rt. The xylene was evaporated and extracted with ethylacetate. The organic extract was washed with water, brine, dried over anhydrous MgSO_4 and concentrated. The crude product was purified by silica gel column chromatography (EtOAc/hexane, 40:60) to obtain compound mixture **16** (0.407 g, 78%) as a pale brown solid.

Compound 17: To a solution of compound mixture **16** (0.283 g, 0.732 mmol) in 1, 4-dioxane (10 mL), tetraethylammoniumhydroxide (4 mL) was added and stirred for 8 h at rt. Thereafter, it was extracted with ethylacetate. The organic extract was washed with water, brine, dried over

anhydrous MgSO₄ and concentrated. The crude product was purified by silica gel column chromatography (EtOAc/hexane, 60:40) to obtain compound mixture **17** as colourless solid (0.206 g, 91%).

Compounds 18a and 18b:

A solution of compound **17** (0.210 g, 0.681 mmol) and DMAP (0.008 g, 0.068 mmol) in 25 mL of CH₂Cl₂ put in a 100 mL RB flask at 0 °C. To this, NEt₃ (0.206 g, 2.04 mmol) was added followed by the slow addition of acetyl chloride (0.160 g, 2.04 mmol). The reaction mixture was stirred for 3 h. The contents were poured into cold water and extracted with CH₂Cl₂. The organic extract was washed with water, brine and dried over anhydrous MgSO₄, concentrated. The crude product was purified by silica gel column chromatography (EtOAc/hexane, 10:90) to obtain compound **18a** (0.163 g, 61%) as a colourless solid, m.p. 102-104 °C and compound **18b** (0.074 g, 28%) as a colourless solid, m.p. 64-66 °C.

Compound 18a (Moarcin D diacetate):⁷

¹H NMR (400 MHz, CDCl₃): δ = 7.51 (d, J = 8.24 Hz, 1H), 7.24 (s, 1H), 7.15 (d, J = 0.92 Hz, 1H), 7.11 (d, J = 1.40 Hz, 1H), 6.96 (dd, J = 8.46, 2.06 Hz, 1H), 6.93 (d, J = 0.88 Hz, 1H), 6.33 (d, J = 9.64 Hz, 1H), 5.69 (d, J = 10.08 Hz, 1H), 2.34 (s, 3H), 2.32 (s, 3H), 1.46 (s, 6H) ppm. ¹³C NMR (100 MHz, CDCl₃): δ = 169.69, 168.99, 155.85, 154.62, 154.04, 148.02, 146.65, 131.83, 130.62, 126.90, 120.92, 117.19, 115.92, 114.65, 110.94, 110.34, 105.04, 101.80, 76.56, 27.89, 21.13, 20.81 ppm. IR (KBr): $\tilde{\nu}$ = 2976, 2931, 1766, 1618, 1553, 1484, 1369, 1205, 1132, 1114, 1059, 1009, 972.18, 907.83 cm⁻¹. HRMS (EI): calcd for C₂₃H₂₀O₆ [M]⁺ 392.1260; found 392.1268.

Compound 18b (Moracin E diacetate):⁸

¹H NMR (400 MHz, CDCl₃): δ = 7.56 (d, J = 8.24 Hz, 1H), 7.30 (bs, 1H), 7.02-6.98 (m, 2H), 6.84 (s, 1H), 6.82 (d, J = 10.04 Hz, 1H), 6.63 (d, J = 2.28 Hz, 1H), 5.71 (d, J = 10.08 Hz, 1H), 2.34 (s, 3H), 2.30 (s, 3H), 1.48 (s, 6H) ppm. ¹³C NMR (100 MHz, CDCl₃): δ = 169.74, 169.14, 154.89, 154.60, 150.68, 148.06, 130.96, 127.83, 126.68, 121.00, 119.95, 117.23, 116.90, 113.62, 110.70, 106.03, 105.16, 75.76, 27.68, 21.12 ppm. IR (KBr): $\tilde{\nu}$ = 3070, 2975, 2929, 1766, 1618, 1553, 1484, 1435, 1419, 1369, 1205, 1132, 1114 cm⁻¹. HRMS (EI): calcd for C₂₃H₂₀O₆ [M]⁺ 392.1260; found 392.1261.

Compound 3 (Moracin D):⁷

A solution of compound **18a** (0.05 g, 0.127 mmol) and 1:1 mixture of aqueous ammonia in methanol (8 mL) stirred for 2 h at rt. It was extracted with ethyl acetate and washed with water and brine solution. The organic extract was dried over anhydrous MgSO₄, concentrated and dried under reduced pressure. The crude product was purified by silica gel column chromatography (EtOAc / hexane, 50:50) to obtain moracin D (0.036 mg, 92%) as a off white solid, m.p. 122-124 °C.

¹H NMR (500 MHz, Acetone-*d*₆): δ = 8.97 (bs, 1H), 8.77 (bs, 1H), 7.39 (d, *J* = 8.45 Hz, 1H), 7.05 (d, *J* = 0.72 Hz, 1H), 6.97 (d, *J* = 2.15 Hz, 1H), 6.93 (d, *J* = 1.43 Hz, 1H), 6.80 (dd, *J* = 8.45, 2.15 Hz, 1H), 6.77 (dd, *J* = 1.43, 0.72 Hz, 1H), 6.69 (dd, *J* = 9.88, 0.72 Hz, 1H), 5.67 (d, *J* = 9.88 Hz, 1H), 1.41 (s, 6H). ¹³C NMR (125 MHz, Acetone-*d*₆): δ = 156.99, 156.89, 155.30, 154.40, 132.10, 129.72, 122.59, 122.07, 117.69, 113.42, 110.58, 104.83, 104.50, 102.56, 98.47, 76.65, 28.12 ppm. IR (KBr): $\tilde{\nu}$ = 3351, 2975, 2925, 2854, 1616, 1561, 1488, 1425, 1146, 1115, 1062, 898, 818 cm⁻¹. HRMS (ESI): calcd for C₁₉H₁₅O₄, [M-H]⁻ 307.0971; found 307.0960.

Compound 4 (Moracin E):⁸

A solution of compound **18b** (0.05 mg, 0.127 mmol) and 1:1 mixture of aqueous ammonia in methanol (8 mL) stirred for 2 h at rt. It was extracted with ethyl acetate and washed with water and brine solution. The organic extract was dried over anhydrous MgSO₄ and concentrated. The crude product was purified by silica gel column chromatography (EtOAc/hexane, 50:50) to obtain moracin E (0.0365 g, 93%) as a pale brown solid, m.p. 166-168 °C.

¹H NMR (500 MHz, Acetone-*d*₆): δ = 8.65 (bs, 2H), 7.46 (d, *J* = 8.31 Hz, 1H), 7.00 (d, *J* = 2.15 Hz, 1H), 6.87 (d, *J* = 0.72 Hz, 1H), 6.84 (dd, *J* = 8.31, 2.15 Hz, 1H), 6.83 (dd, *J* = 10.10, 0.57 Hz, 1H), 6.79 (d, *J* = 2.43 Hz, 1H), 6.35 (dd, *J* = 2.43, 0.57 Hz, 1H), 5.67 (d, *J* = 10.02 Hz, 1H), 1.42 (s, 6H) ppm. ¹³C NMR (125 MHz, Acetone-*d*₆): δ = 159.08, 156.95, 156.89, 156.26, 154.29, 129.54, 128.97, 122.43, 122.25, 121.29,

113.41, 112.29, 108.15, 106.71, 104.83, 98.48, 76.04, 27.83 ppm. IR (KBr): $\tilde{\nu}$ = 3341, 2975, 2926, 2854, 1609, 1575, 1489, 1440, 1146, 1119, 1018, 969, 826 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{19}\text{H}_{17}\text{O}_4$ $[\text{M}+\text{H}]^+$ 309.1127; found 309.1120.

Compound 19:⁹

To a solution of resorcinol **9** (8.3 g, 75.47 mmol) in dichloroethane (150 mL) was added *p*-toluenesulfonic acid (1 g, 5.80 mmol). To this 2-methyl-3-buten-2-ol (5 g, 58.05 mmol) in dichloroethane (20 mL) was added dropwise in 30 minutes and stirred for 2 h at rt. Thereafter, it was refluxed at 80 °C for 10 h and cooled to rt. Then solvent was evaporated and the crude was extracted with ethyl acetate. The organic extract was washed with brine, dried over anhydrous MgSO_4 and concentrated. The crude product was purified by silica gel column chromatography (EtOAc/hexane 05:95) to obtain compound **19** (6.2 g, 60%) as a colourless solid, m.p. 62-64 °C.

^1H NMR (400 MHz, CDCl_3): δ = 6.90 (d, J = 8.24 Hz, 1H), 6.34 (dd, J = 8.24, 2.29 Hz, 1H), 6.28 (d, J = 2.29 Hz, 1H), 4.65 (bs, 1H) 2.69 (t, J = 6.87 Hz, 2H), 1.78 (t, J = 6.87 Hz, 2H), 1.32 (s, 6H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ = 154.77, 154.72, 130.10, 113.30, 107.29, 103.71, 74.40, 32.89, 26.80, 21.72 ppm. IR (KBr): $\tilde{\nu}$ = 3352, 2974, 2930, 1596, 1623, 1507, 1458, 1304, 1149, 1118, 994, 847 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{11}\text{H}_{15}\text{O}_2$ $[\text{M}+\text{H}]^+$ 179.1072; found 179.1071.

Compound 20:¹⁰

A mixture of compound **19** (5 g, 28.05 mmol), MgCl_2 (4 g, 42.08 mmol) and NEt_3 (10.64 g, 105.18 mmol) in dry THF (150 mL) stirred for 20 minutes at rt. Then paraformaldehyde (5.68 g, 189.34 mmol) was added portion wise, stirred for 1 h followed by reflux for 7 h. It was cooled to rt, evaporated THF and extracted with ethyl acetate. The organic extract was washed with dil. HCl, water followed by brine. The crude product was purified by column chromatography (EtOAc/hexane, 10:90) to obtain compound **20** (3.18 g, 55%) as a yellow solid, m.p. 82-84 °C.

^1H NMR (400 MHz, CDCl_3): δ = 11.06 (s, 1H), 9.66 (s, 1H), 7.21 (s, 1H), 6.31 (s, 1H), 2.75 (t, J = 6.64 Hz, 2H), 1.83 (t, J = 6.64 Hz, 2H), 1.36 (s, 6H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ = 194.16, 162.11, 162.01, 135.28, 115.19, 113.76, 104.34, 76.25, 32.54, 26.95, 21.42 ppm. IR (KBr): $\tilde{\nu}$ = 3023, 2933, 2978, 2844, 2742, 1629, 1652, 1582, 1493, 1458, 1327, 1344, 1296, 1173, 851, 760 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{12}\text{H}_{15}\text{O}_3$ $[\text{M}+\text{H}]^+$ 207.1021; found 207.1029.

Compound 20a:¹¹

A solution of compound **20** (2 g, 9.7 mmol) and DDQ (2.42 g, 10.7 mmol) in 1,4-dioxane (30 mL) was refluxed at 100 °C for 10 h then cooled to rt. It was extracted with ethyl acetate and washed with water followed by brine. The organic extract was dried over anhydrous MgSO_4 , concentrated and purified by silica gel column chromatography (EtOAc/hexane, 15:85) to obtain compound **20a** (1.4 g, 70%) as a pale yellow solid, m.p. 88-90 °C.

^1H NMR (400 MHz, CDCl_3): δ = 11.43 (s, 1H), 9.66 (s, 1H), 7.11 (s, 1H), 6.33 (s, 1H), 6.29 (d, J = 9.62 Hz, 1H), 5.59 (d, J = 9.62 Hz, 1H), 1.45 (s, 6H) ppm. ^{13}C NMR (100, CDCl_3): δ = 194.06, 164.26, 161.22, 131.34, 128.95, 120.59, 115.19, 114.42, 104.15, 78.28, 28.61 ppm. IR (KBr): $\tilde{\nu}$ = 3059, 2971, 2934, 1643, 1589, 1487, 1370, 1340, 1286, 1261, 1214, 1169, 1140, 1105, 882, 777, 763 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{12}\text{H}_{13}\text{O}_3$ $[\text{M}+\text{H}]^+$ 205.0865; found 205.0860.

Compound 21:

A solution of compound **20** (4 g, 19.33 mmol) and DMAP (0.236 g, 1.93 mmol) in 100 mL of CH_2Cl_2 were put in a 250 mL RB flask at 0 °C. To this, NEt_3 (3.92 g, 38.77 mmol) was added followed by slow addition of acetyl chloride (3.04 g, 38.77 mmol). The reaction mixture was stirred for 3 h at rt and poured into cold water. The organic layer was separated and washed with water followed by brine. The organic extract was dried over anhydrous MgSO_4 , concentrated and purified by silica gel column chromatography (EtOAc/hexane, 20:80) to obtain compound **21** (4.4 g, 92%) as a yellow solid, m.p. 78-80 °C.

^1H NMR (400 MHz, CDCl_3): δ = 9.88 (s, 1H), 7.58 (s, 1H), 6.53 (s, 1H), 2.80 (t, J = 6.42 Hz, 2H), 2.36 (s, 3H), 1.84 (t, J = 6.64 Hz, 2H), 1.36 (s, 6H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ = 187.60, 169.34, 160.29, 151.09, 133.34, 120.75, 119.22, 111.85, 76.32, 32.24, 26.94, 21.76, 20.85 ppm. IR (KBr): $\tilde{\nu}$ = 2977, 2935, 2853, 1767, 1688, 1610, 1572, 1491, 1370, 1290, 1200, 1149, 1111, 912 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{14}\text{H}_{17}\text{O}_4$ $[\text{M}+\text{H}]^+$ 249.1127; found 249.1123.

Compound 21a:

A solution of compound **20a** (1.1 g, 5.4 mmol) and DMAP (0.066 g, 0.54 mmol) in 60 mL of CH_2Cl_2 were put in a 100 mL RB flask at 0 °C. To this, NEt_3 (1.09 g, 10.77 mmol) was added followed by the slow addition of acetyl chloride (0.845 g, 10.77 mmol). The reaction mixture was stirred for 2 h and poured into cold water. Thereafter, organic layer was separated, washed with water and brine. The organic extract was dried under anhydrous MgSO_4 , concentrated and purified by silica gel column chromatography (EtOAc/hexane, 20:80) to obtain compound **21a** (0.900 g, 68%) as a pale yellow solid, m.p. 72-74 °C.

^1H NMR (400 MHz, CDCl_3): δ = 9.90 (s, 1H), 7.47 (s, 1H), 6.54 (s, 1H), 6.34 (d, J = 10.02 Hz, 1H), 5.66 (d, J = 10.02 Hz, 1H), 2.36 (s, 3H), 1.46 (s, 6H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ = 187.27, 169.07, 159.20, 152.84, 130.88, 129.00, 121.44, 120.58, 119.02, 111.11, 78.38, 28.67, 20.87 ppm. IR (KBr): $\tilde{\nu}$ = 2977, 2934, 2855, 1769, 1687, 1642, 1605, 1568, 1486, 1365, 1286, 912, 770 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{14}\text{H}_{14}\text{NaO}_4$ $[\text{M}+\text{Na}]^+$ 269.0790; found 269.0799.

Compound 24:

To a solution of compound **23** (0.610 g, 1.80 mmol) in CH_2Cl_2 (50 mL), BBr_3 (1.129 g, 4.50 mmol) solution in CH_2Cl_2 (10 mL) was added dropwise at -78 °C. The reaction mixture was brought to rt and stirred for 24 h. The mixture was then cooled to 0 °C, quenched with water (10 mL) and extracted with ethyl acetate. The organic extract was dried over anhydrous MgSO_4 and concentrated. The crude product was purified by column chromatography (EtOAc/hexane, 60:40) to obtain compound **24** (0.475 g, 85%) as pale brown solid, m.p. 120-124 °C.

^1H NMR (400 MHz, DMSO- d_6): δ = 9.43 (s, 2H), 7.28 (s, 1H), 7.06 (s, 1H), 6.91 (s, 1H), 6.68 (d, J = 2.28 Hz, 2H), 6.21 (t, J = 2.06 Hz, 1H), 2.84 (t, J = 6.64 Hz, 2H), 1.78 (t, J = 6.64 Hz, 2 H), 1.30 (s, 6H) ppm. ^{13}C NMR (125 MHz, DMSO- d_6): δ = 158.77, 154.35, 153.71, 151.86, 131.54, 121.73, 120.51, 117.37, 102.72, 102.38, 101.08, 98.51, 74.29, 32.25, 26.59, 22.06 ppm. IR (KBr): $\tilde{\nu}$ = 3364, 2975, 2931, 1702, 1621, 1578, 1460, 1354, 1325, 1264, 1148, 1108, 948.82, 926.90 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{19}\text{H}_{17}\text{O}_4$ $[\text{M}-\text{H}]^-$ 309.1127; found 309.1126.

Compound 25:

A solution of compound **24** (0.250 g, 0.805 mmol) and DMAP (0.010 g, 0.080 mmol) in 50 mL of CH_2Cl_2 put in a 100 mL RB flask at 0 $^\circ\text{C}$. To this, NEt_3 (0.326 g, 3.22 mmol) was added followed by the slow addition of acetyl chloride (0.253 g, 3.22 mmol). The reaction mixture was stirred for 2 h and then poured into cold water. Thereafter, organic layer was washed with water followed by brine. It was dried over anhydrous MgSO_4 , concentrated and purified by silica gel column chromatography (EtOAc/hexane, 5:95) to obtain compound **25** (0.302 g, 95%) as a colourless solid, m.p. 178-180 $^\circ\text{C}$.

^1H NMR (400 MHz, CDCl_3): δ = 7.41 (d, J = 1.8 Hz, 2H), 7.22 (s, 1H), 6.90 (s, 1H), 6.88 (s, 1H), 6.86 (t, J = 2.06 Hz, 1H), 2.89 (t, J = 6.86 Hz, 2H), 2.32 (s, 6H), 1.84 (t, J = 6.86 Hz, 2H), 1.37 (s, 6H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ = 168.97, 154.77, 152.92, 152.74, 151.35, 132.86, 121.90, 120.56, 117.65, 114.95, 114.66, 102.37, 99.26, 74.55, 32.91, 26.90, 22.75, 21.14 ppm. IR (KBr): $\tilde{\nu}$ = 2975, 2932, 1770, 1615, 1594, 1573, 1459, 1369, 1352, 1195, 1147, 1123, 1022, 1065, 1023 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{23}\text{H}_{23}\text{O}_6$ $[\text{M}+\text{H}]^+$ 395.1495; found 395.1492.

Compound 26:

A solution of compound **25** (0.100 g, 0.253 mmol) and DDQ (0.057 g, 0.253 mmol) in 1,4-dioxane (5 mL) was refluxed at 100 $^\circ\text{C}$ for 20 h. The contents were brought to rt, extracted with ethyl acetate, washed with water followed by brine. The organic extract was dried over anhydrous MgSO_4 , concentrated. The crude was purified by silica gel column chromatography (EtOAc/hexane, 5:95) to obtain compound **26** (0.089 g, 89%) as colourless solid, m.p. 138-140 $^\circ\text{C}$.

^1H NMR (400 MHz, CDCl_3): δ = 7.41 (d, J = 1.84 Hz, 2H), 7.14 (s, 1H), 6.92 (s, 1H), 6.90 (s, 1H), 6.87 (t, J = 2.06 Hz, 1H), 6.41 (d, J = 9.6 Hz, 1H), 5.64 (d, J = 10.08 Hz, 1H), 2.32 (s, 6H), 1.46 (s, 6H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ = 168.91, 155.65, 153.22, 151.95, 151.40, 132.62, 130.20, 122.61, 122.47, 118.53, 117.87, 114.94, 114.77, 102.74, 99.50, 76.43, 27.86, 21.12 ppm. IR (KBr): $\tilde{\nu}$ = 3042, 2926, 2971, 1769, 1616, 1574, 1462, 1433, 1370, 1346, 1197, 1126, 1023 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{23}\text{H}_{21}\text{O}_6$ $[\text{M}+\text{H}]^+$ 393.1338; found 393.1336.

Compound 6:¹²

A solution of compound **23** (0.500 g, 1.478 mmol) and DDQ (0.336 g, 1.478 mmol) in 1,4-dioxane (15 mL) was refluxed at 100 °C for 20 h. Thereafter, contents were cooled to rt and extracted with ethyl acetate, washed with water followed by brine. The organic extract was dried over anhydrous MgSO_4 , concentrated and purified by silica gel column chromatography (EtOAc/hexane, 5:95) to obtain compound **5** (0.447 g, 90%) as a yellow solid, m.p. 114-116 °C.

^1H NMR (400 MHz, CDCl_3): δ = 7.13 (s, 1H), 6.96-6.95 (m, 3H), 6.88 (s, 1H), 6.44 (t, 2.3 Hz, 1H), 6.42 (d, 9.6 Hz, 1H), 5.64 (d, J = 9.6 Hz, 1H), 3.86 (s, 6H), 1.46 (s, 6H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ = 161.03, 155.46, 154.94, 151.55, 132.34, 130.05, 122.74, 118.34, 117.62, 102.51, 101.69, 100.57, 99.51, 76.31, 55.47, 27.79 ppm. IR (KBr): $\tilde{\nu}$ = 2973, 2936, 2837, 1574, 1599, 1461, 1350, 1423, 1204, 1156, 1126, 1066, 1035, 911, 837, 732 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{21}\text{H}_{21}\text{O}_4$ $[\text{M}+\text{H}]^+$ 337.1440 found 337.1440.

Compound 5:¹²

A solution of compound **26** (0.050 g, 0.127 mmol) in 1:1 mixture of aqueous ammonia and methanol (8 mL) was stirred at rt for 5 h. It was extracted with ethyl acetate and washed with water followed by brine. The organic extract was dried over anhydrous MgSO_4 and concentrated. The crude product was purified by silica gel column chromatography (EtOAc/hexane, 70:30) to obtain compound **6** (0.035 g, 90%) as a brown solid, m.p. 138-140 °C.

^1H NMR (500 MHz, $\text{DMSO-}d_6$): δ = 9.43 (s, 2H), 7.28 (s, 1H), 7.10 (s, 1H), 6.99 (s, 1H), 6.69 (d, J = 2.3 Hz, 2H), 6.50 (d, J = 9.7 Hz, 1H), 6.22 (t, J = 2.42 Hz, 1H), 5.75 (d, J = 9.7 Hz, 1H), 1.39 (s, 6H) ppm. ^{13}C NMR (125 MHz, $\text{DMSO-}d_6$): δ = 158.80, 154.76, 154.53, 150.96, 131.33, 130.23, 122.41, 122.26, 118.04, 117.87, 102.85, 102.41, 101.52, 98.90, 76.15, 27.49 ppm. IR (KBr): $\tilde{\nu}$ = 3615, 3290, 2972, 2923, 2852, 1613, 1581, 1466, 1440, 1353, 1149, 1128, 1001, 838 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{19}\text{H}_{17}\text{O}_4$ $[\text{M}+\text{H}]^+$ 309.1127; found 309.1125.

Compound 7:^{4,12,13}

A solution of compound **27** (0.100 g, 0.324 mmol) and DDQ (0.074 g, 0.324 mmol) in 1,4-dioxane (5 mL) was refluxed at 100 °C for 20 h. Thereafter, it was cooled to rt and extracted with ethyl acetate and washed with water followed by brine. The organic layer was dried over anhydrous MgSO_4 , concentrated and purified by silica gel column chromatography (EtOAc / hexane, 5:95) to obtain compound **7** (0.092 g, 93%) as a semi white solid, m.p. 172-174 °C.

^1H NMR (400 MHz, CDCl_3): δ = 7.73 (d, J = 8.68 Hz, 2H), 7.11 (s, 1H), 6.96 (s, 1H), 6.95 (d, J = 8.72 Hz, 2H), 6.75 (d, J = 0.92 Hz, 1H), 6.42 (d, J = 9.6 Hz, 1H), 5.63 (d, J = 9.6 Hz, 1H), 3.85 (s, 3H), 1.46 (s, 6H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ = 159.53, 155.27, 150.99, 129.85, 125.89, 123.47, 123.08, 122.81, 118.09, 117.26, 114.14, 99.44, 76.18, 55.30, 27.73 ppm. IR (KBr): $\tilde{\nu}$ = 2965, 1612, 1504, 1461, 1250, 1179, 1087, 1033, 832, 797, 732 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{20}\text{H}_{19}\text{O}_3$ $[\text{M}+\text{H}]^+$ 307.1334; found 307.1331.

Compound 28:

To a solution of compound **27** (0.100 g, 0.324 mmol) in CH_2Cl_2 (15 mL), BBr_3 (0.097 g, 0.389 mmol) solution in CH_2Cl_2 (5 mL) was added dropwise at -78 °C. The reaction mixture was brought to rt and stirred for 12 h. Thereafter, the mixture was cooled to 0 °C, quenched with water (5 mL) and extracted with ethyl acetate. The organic extract was dried over anhydrous MgSO_4 and concentrated. The crude product was purified by column chromatography (EtOAc/hexane 20:80) to obtain compound **28** (0.076 g, 80%) as a semi white solid, m.p. 178-180 °C.

^1H NMR (400 MHz, CDCl_3): δ = 7.69 (d, J = 8.72 Hz, 2H), 7.19 (s, 1H), 6.92 (s, 1H), 6.88 (d, J = 8.72 Hz, 2H), 6.73 (s, 1H), 2.89 (t, J = 6.64 Hz, 2H), 1.84 (t, J = 6.64 Hz, 2H), 1.37 (s, 6H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ = 155.47, 154.94, 154.47, 151.85, 126.18, 124.05, 122.54, 119.88, 117.12, 115.63, 99.21, 99.10, 74.36, 33.04, 26.89, 22.78 ppm. IR (KBr): $\tilde{\nu}$ = 3411, 2973, 1596, 1507, 1459, 1353, 1252, 1147, 1116, 836.98, 798, 518.70 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{19}\text{H}_{19}\text{O}_3$ $[\text{M}+\text{H}]^+$ 295.1334; found 295.1330.

Compound 29:

A solution of compound **28** (0.200 g, 0.679 mmol) and DMAP (0.008 g, 0.068 mmol) in 50 mL of CH_2Cl_2 were put in a 100 mL RB flask at 0 $^\circ\text{C}$. To this, NEt_3 (0.137 g, 1.359 mmol) was added followed by the slow addition of acetyl chloride (0.107 g, 1.359 mmol). The reaction mixture was stirred for 2 h and poured into cold water. The organic portion was separated and washed with water followed by brine. It was dried over anhydrous MgSO_4 , concentrated and purified by silica gel column chromatography (EtOAc/hexane, 5:95) to obtain compound **29** (0.194 g, 85%) as a colourless solid, m.p. 156-158 $^\circ\text{C}$.

^1H NMR (400 MHz, CDCl_3): δ = 7.81 (d, J = 8.72 Hz, 2H), 7.22 (s, 1H), 7.14 (d, J = 8.72 Hz, 2H), 6.93 (s, 1H), 6.84 (s, 1H), 2.89 (t, J = 6.64 Hz, 2H), 2.32 (s, 3H), 1.85 (t, J = 6.88 Hz, 2H), 1.37 (s, 6H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ = 169.44, 154.70, 154.12, 152.29, 150.22, 128.63, 125.55, 122.23, 121.89, 120.24, 117.38, 100.83, 99.27, 74.43, 32.96, 26.89, 22.76, 21.16 ppm. IR (KBr): $\tilde{\nu}$ = 2969, 2927, 1755, 1629, 1590, 1499, 1459, 1368, 1199, 1149, 1119, 913, 849 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{21}\text{H}_{21}\text{O}_4$ $[\text{M}+\text{H}]^+$ 337.1440; found 337.1441.

Compound 30:

A solution of compound **29** (0.100 g, 0.297 mmol) and DDQ (0.067 g, 0.297 mmol) in 1,4-dioxane (5 mL) was refluxed at 100 $^\circ\text{C}$ for 20 h. The contents were brought to rt and extracted with ethyl acetate, washed with water followed by brine. The organic extract was dried over anhydrous MgSO_4 , concentrated and purified by silica gel column chromatography (EtOAc/hexane, 5:95) to obtain compound **30** (0.090 g, 90%) as a semi white solid, m.p. 154-156 $^\circ\text{C}$.

^1H NMR (400 MHz, CDCl_3): δ = 7.80 (d, 8.72 Hz, 2H), 7.15 (d, 8.68 Hz, 2H), 7.13 (s, 1H), 6.95 (s, 1H), 6.86 (s, 1H), 6.42 (d, 10.08 Hz, 1H), 5.64 (d, 10.08 Hz, 1H), 2.32 (s, 3H), 1.46 (s, 6H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ = 169.38, 155.56, 154.40, 151.51, 150.34, 130.06, 128.42, 125.54, 122.72, 121.94, 118.34, 117.59, 101.22, 99.53, 76.31, 27.80, 21.15 ppm. IR (KBr): $\tilde{\nu}$ = 2973, 1754, 1593, 1503, 1461, 1368, 1345, 1227, 1210, 1195, 1166, 1013, 838, 849 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{21}\text{H}_{19}\text{O}_4$ $[\text{M}+\text{H}]^+$ 335.1283; found 335.1286.

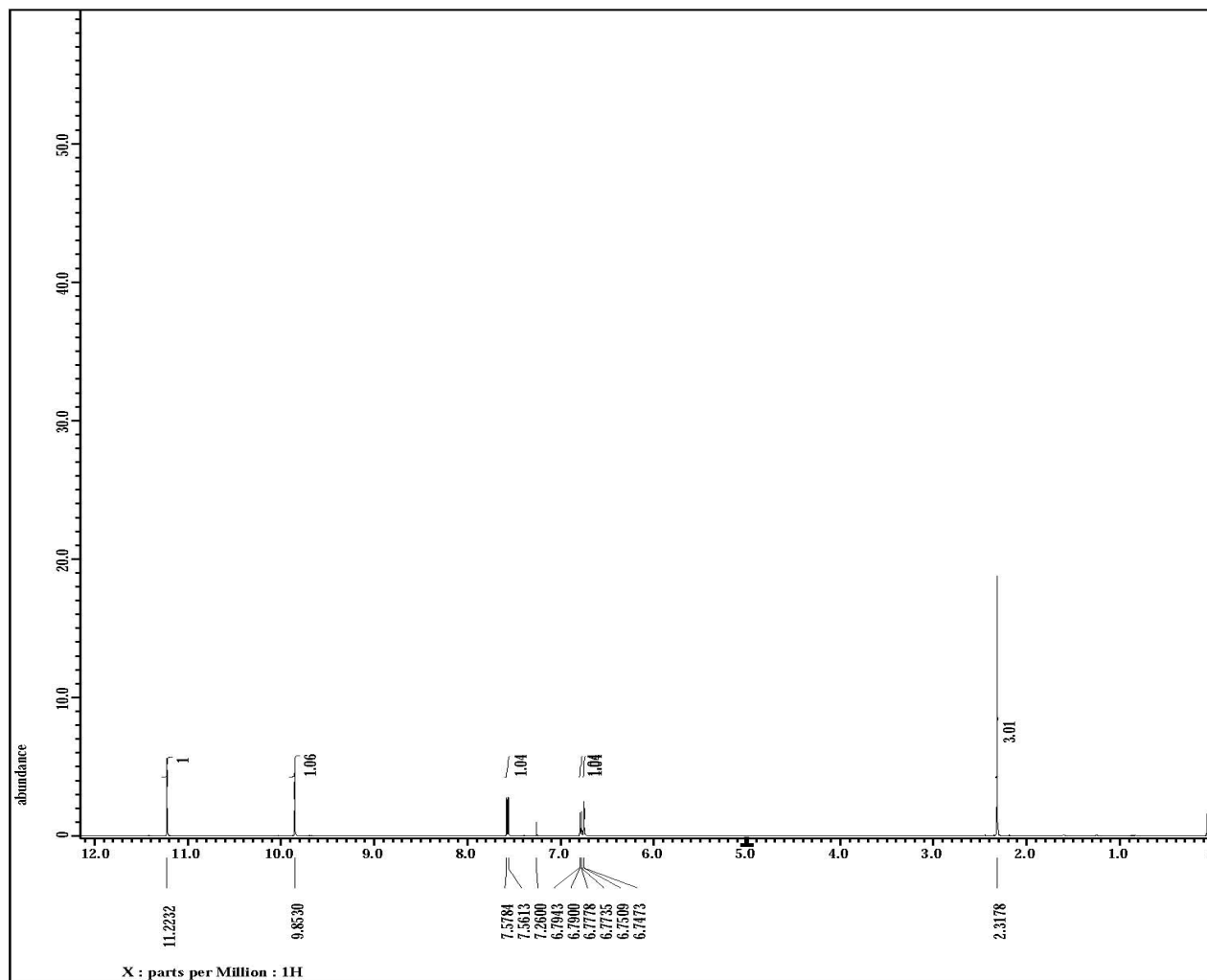
Compound 8:^{12,13}

A solution of compound **29** (0.050 g, 0.149 mmol) in 1:1 mixture of aqueous ammonia and methanol (5 mL) was stirred at rt for 5 h. Thereafter, it was extracted with ethyl acetate, washed with water followed by brine. The organic extract was dried over anhydrous MgSO_4 and concentrated. The crude product was purified by silica gel column chromatography (EtOAc/hexane, 70:30) to obtain compound **8** (0.039 g, 89%) as a brown solid, m.p. 140-142 $^\circ\text{C}$.

^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ = 9.78 (s, 1H), 7.64 (d, J = 8.68 Hz, 2H), 7.24 (s, 1H), 7.02 (s, 1H), 6.95 (s, 1H), 6.84 (d, J = 8.72 Hz, 2H), 6.49 (d, J = 10.08 Hz, 1H), 5.73 (d, J = 10.08 Hz, 1H), 1.38 (s, 6H) ppm. ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$): δ = 157.87, 155.16, 154.42, 150.46, 130.14, 125.90, 122.83, 122.38, 121.08, 117.92, 117.50, 115.81, 99.24, 98.91, 76.10, 27.51 ppm. IR (KBr): $\tilde{\nu}$ = 2971, 2925, 1612, 1507, 1461, 1347, 1253, 1127, 837 cm^{-1} . HRMS (ESI): calcd for $\text{C}_{19}\text{H}_{15}\text{O}_3$ $[\text{M}-\text{H}]^-$ 291.1021; found 291.1023.

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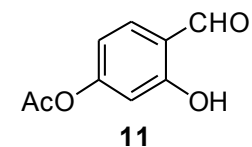
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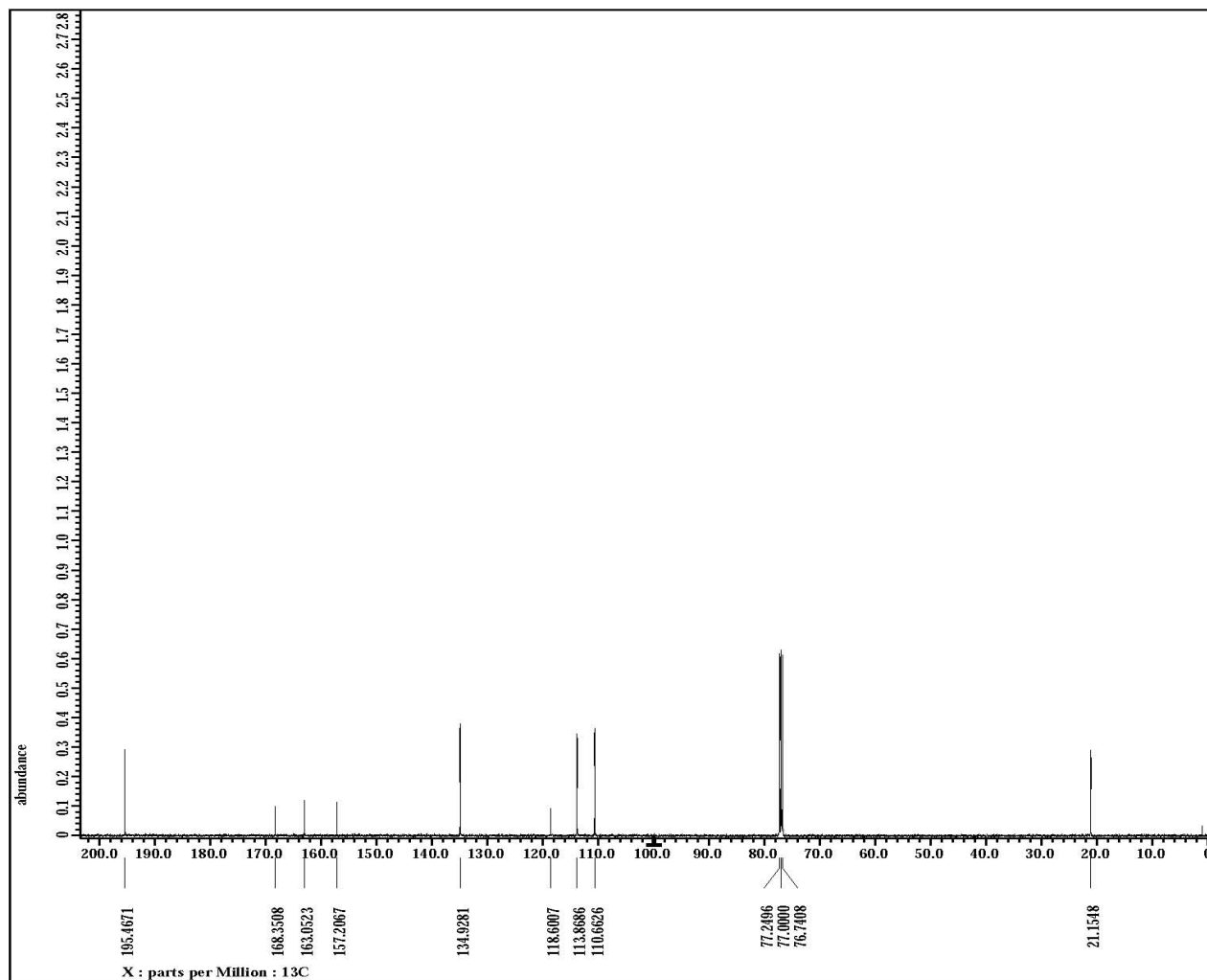
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^1H NMR spectrum of Compound **11**



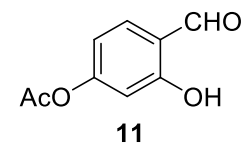
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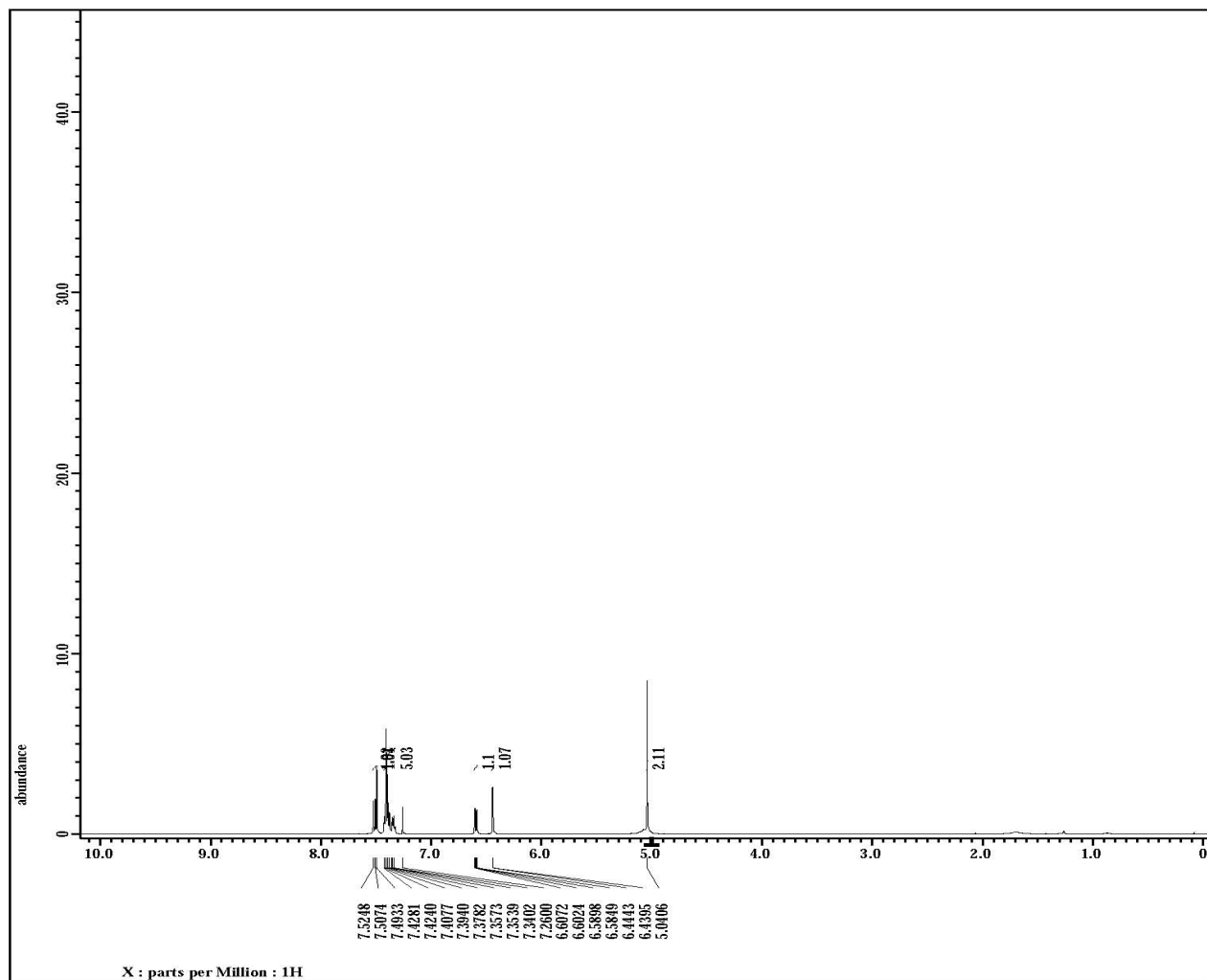
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^{13}C NMR spectrum of Compound 11



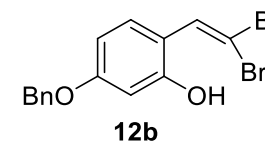
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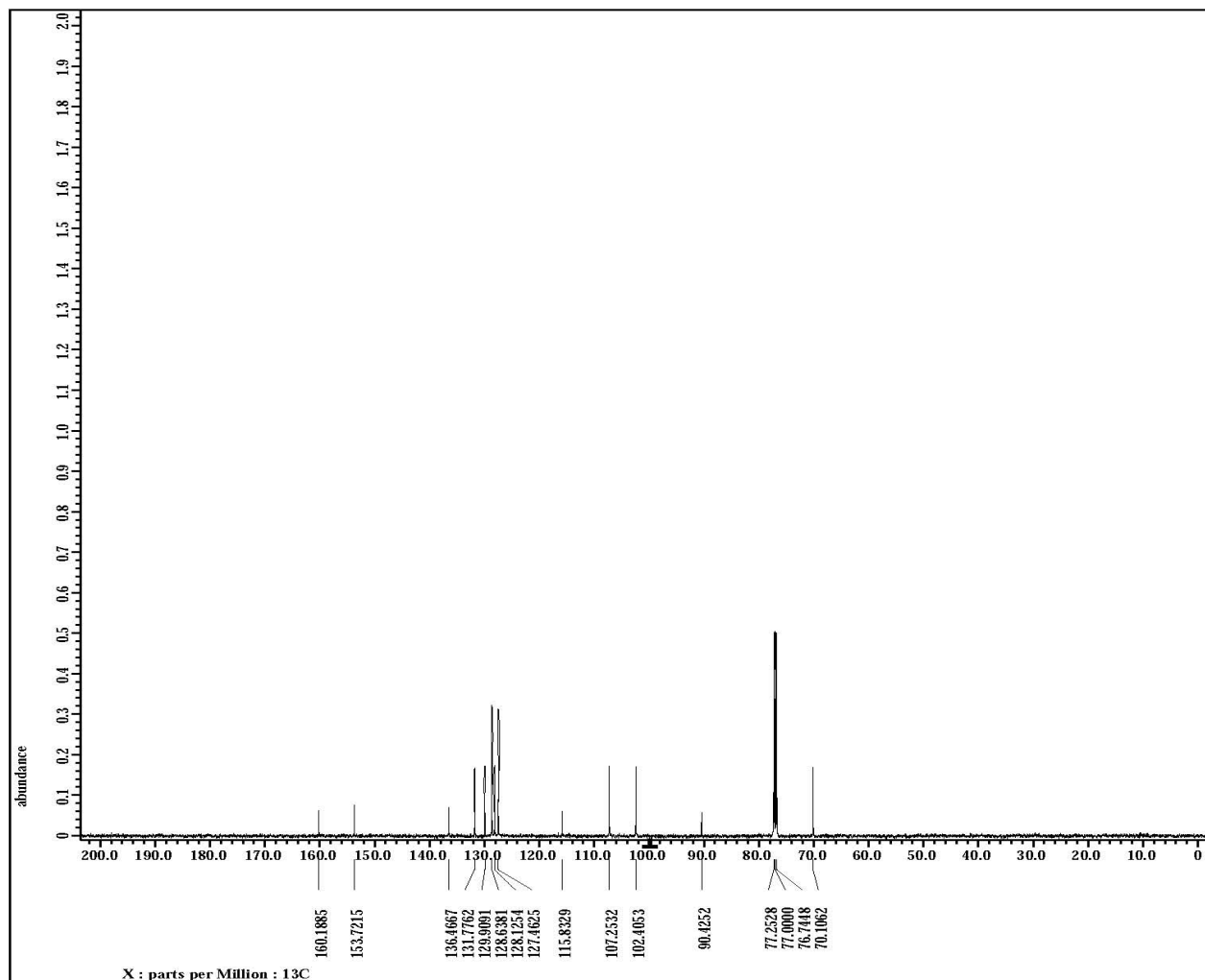
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^1H NMR spectrum of Compound **12b**



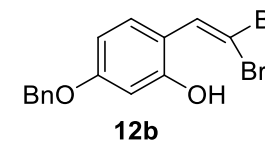
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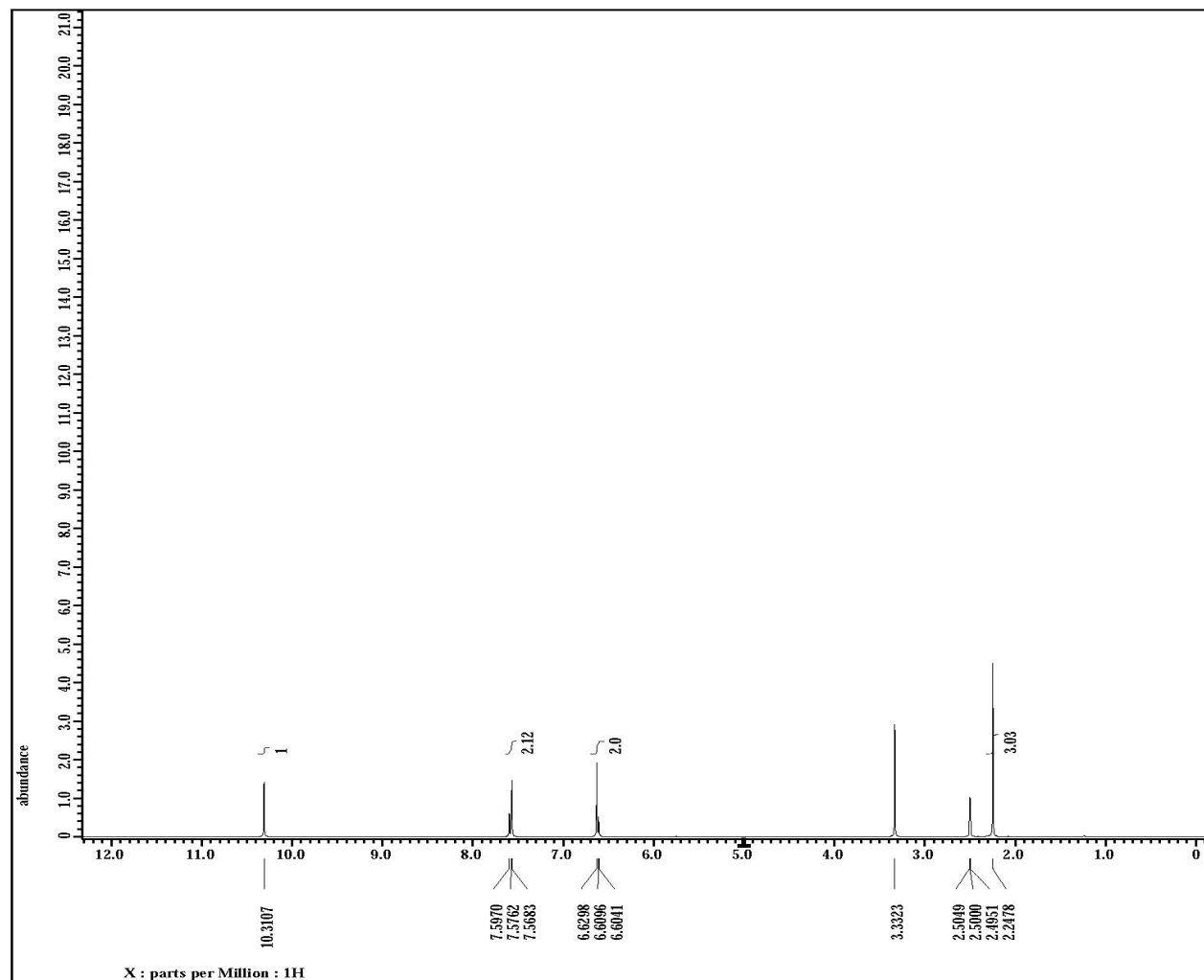
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^{13}C NMR spectrum of Compound **12b**



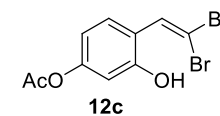
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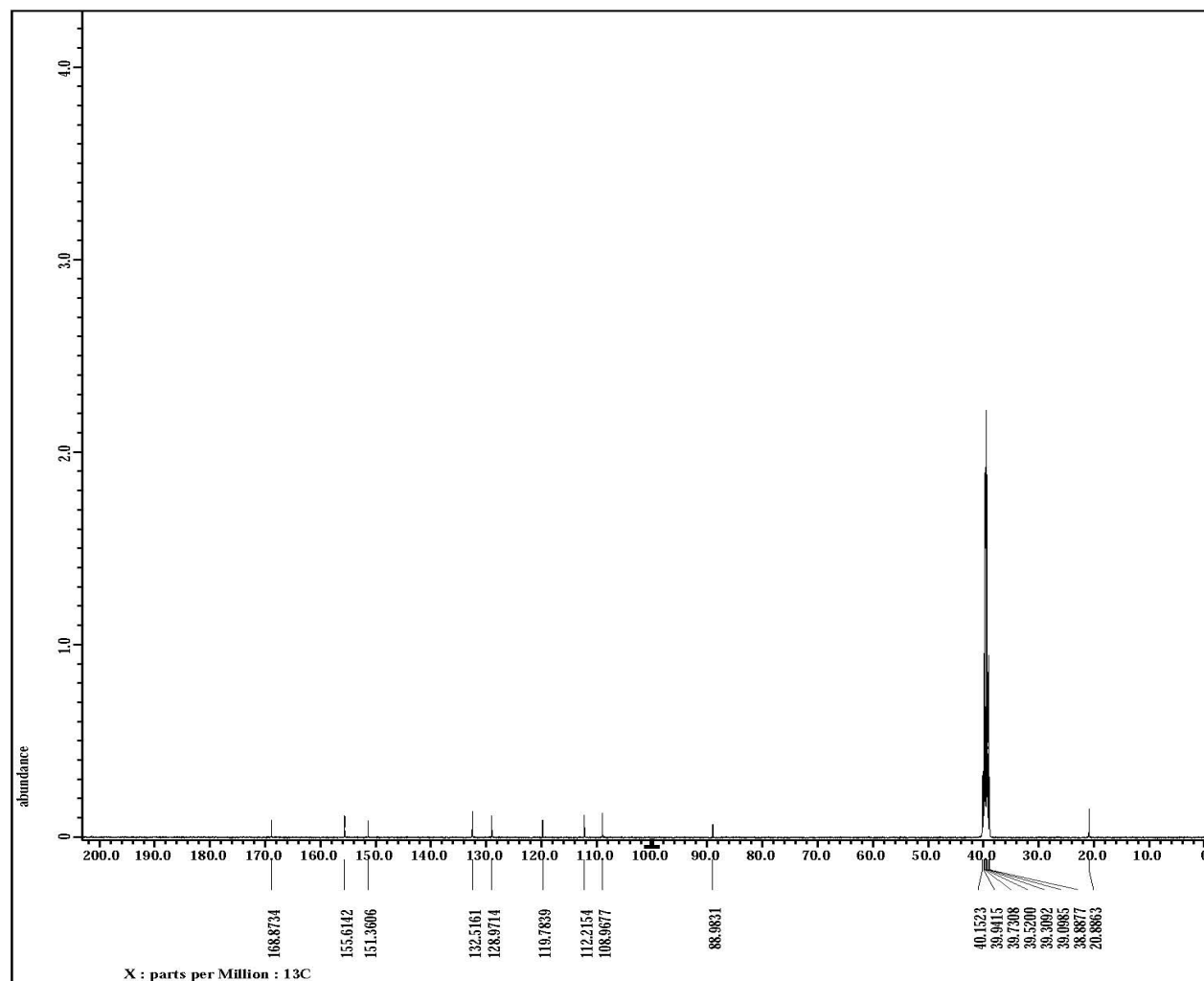
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^1H NMR spectra of Compound **12c**



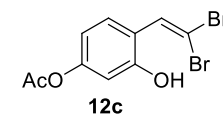
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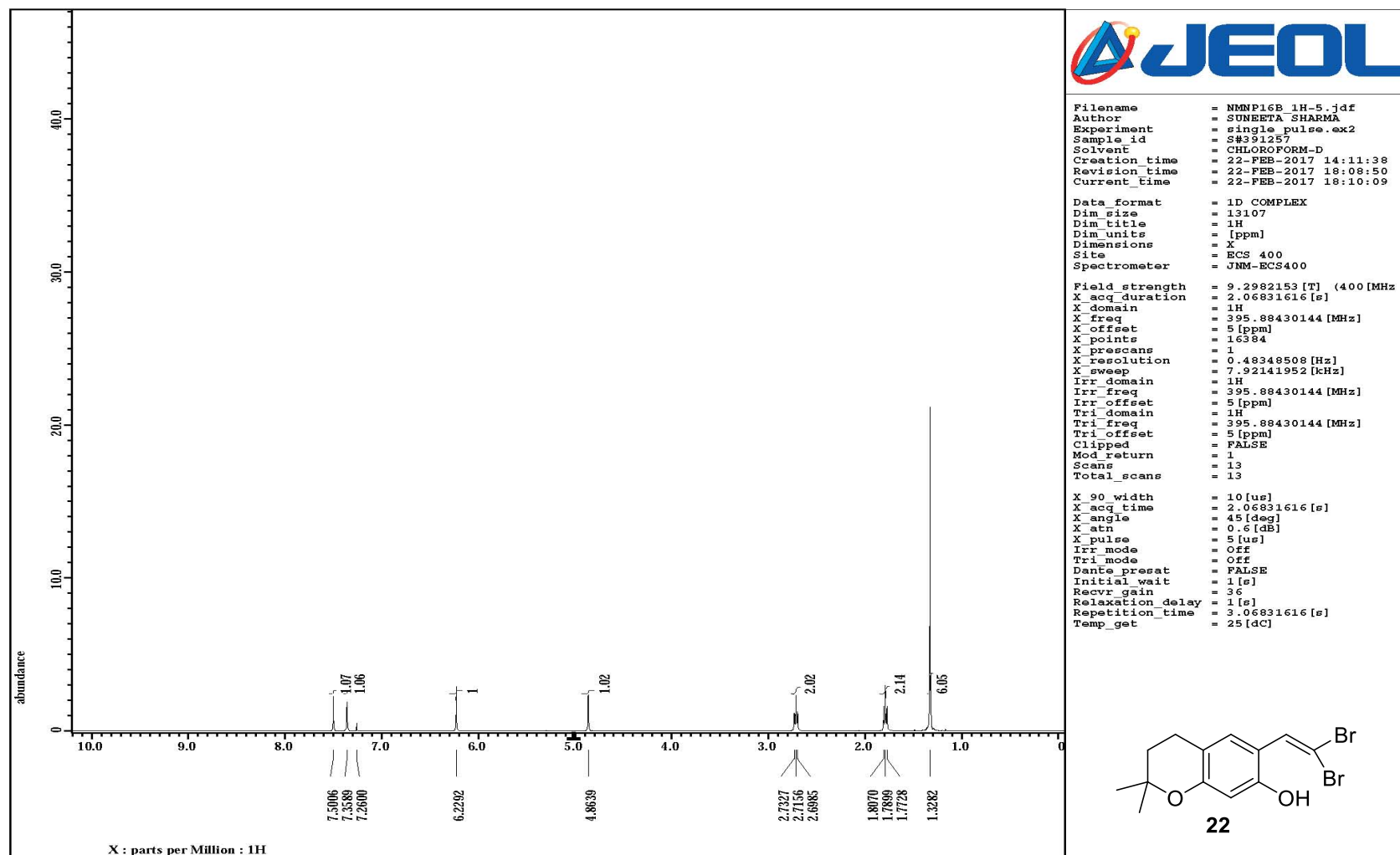
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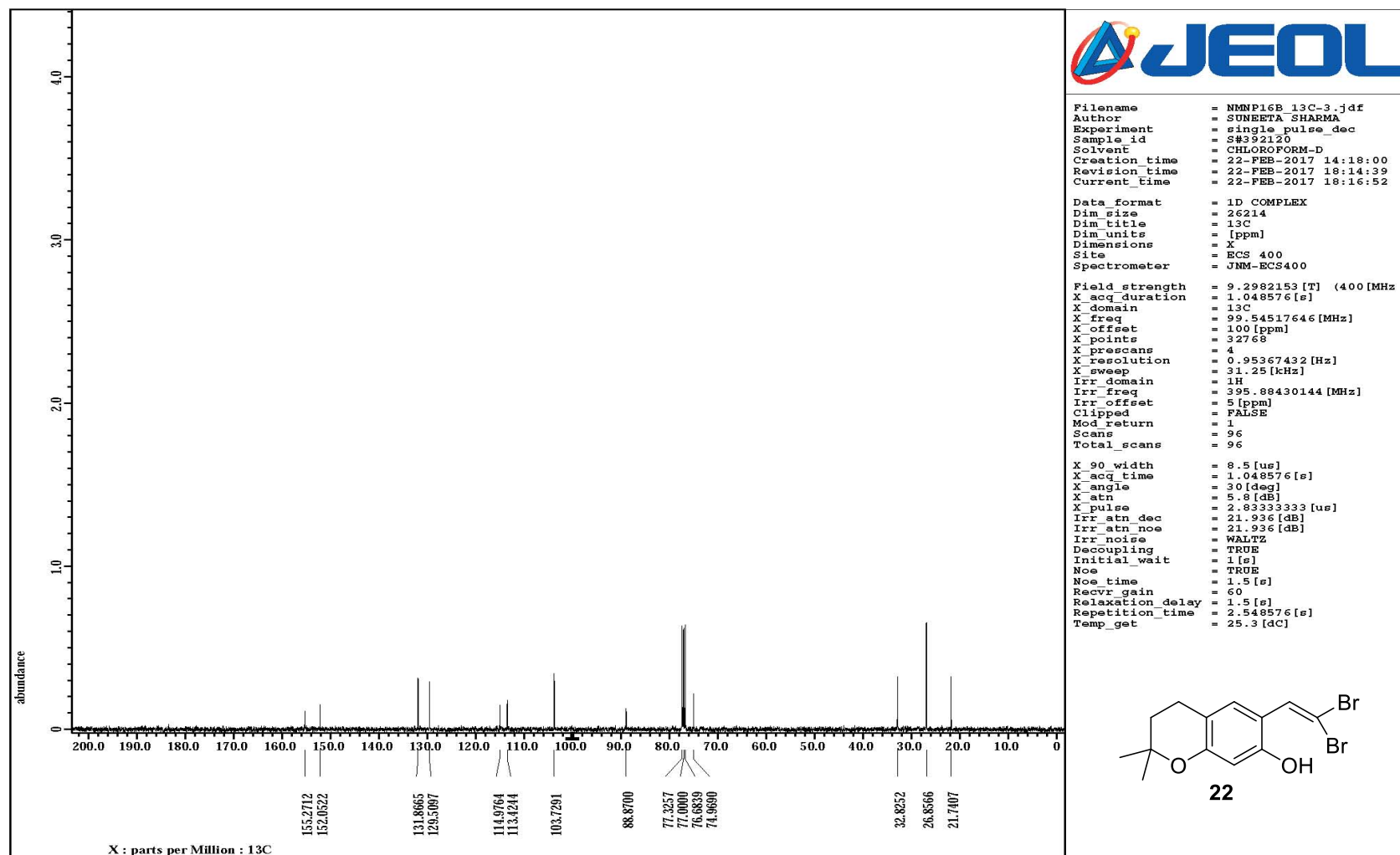
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Irr_atn_dec   = 21.936 [dB]
Irr_atn_noe   = 21.936 [dB]
Irr_noise     = WALTZ
Decoupling    = TRUE
Initial_wait  = 1 [s]
Noe           = TRUE
Noe_time      = 2 [s]
Recvr_gain    = 60
Relaxation_delay = 2 [s]
Repetition_time = 3.048576 [s]
Temp_get      = 25.1 [dC]
  
```



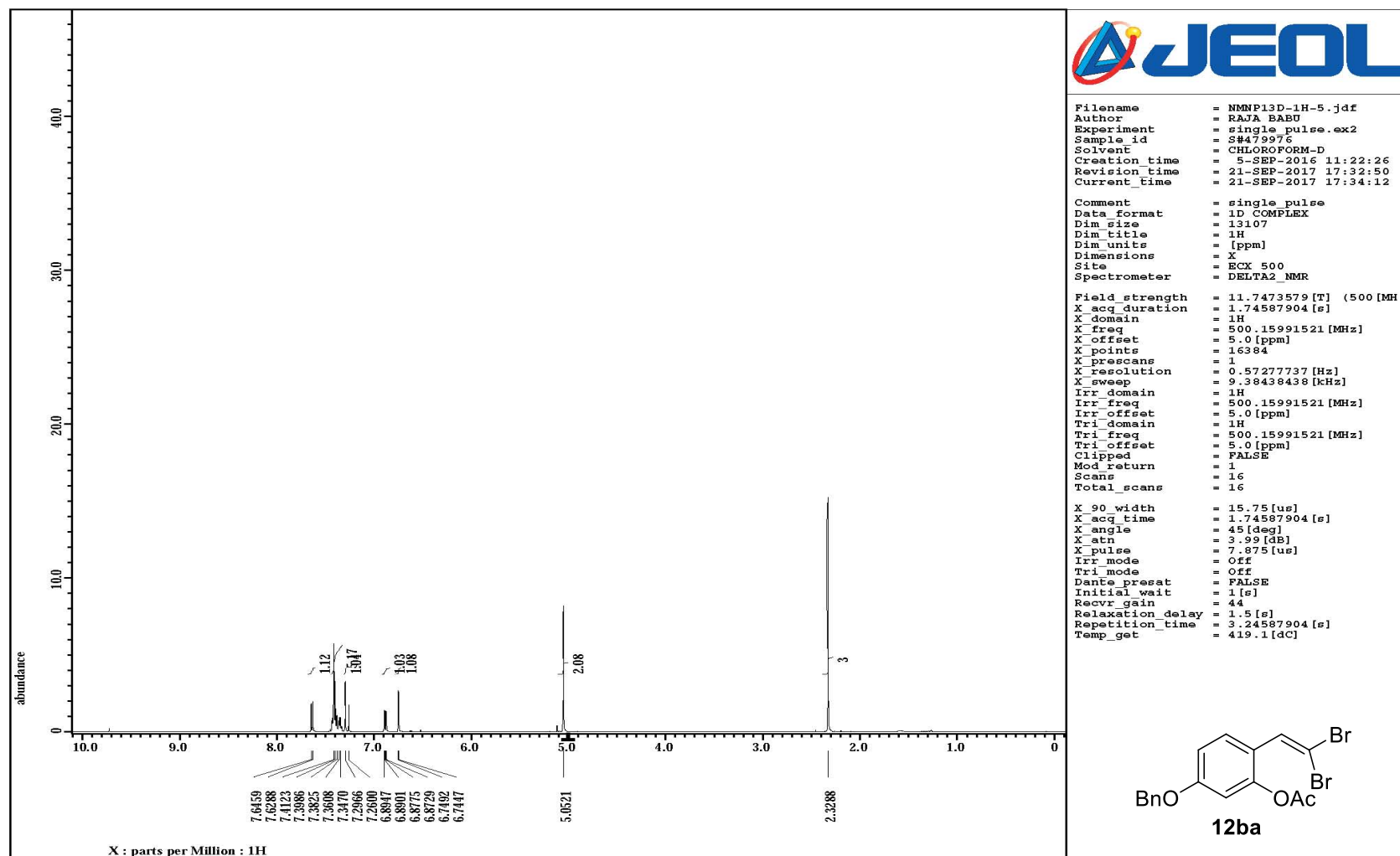
^{13}C NMR spectra of Compound **12c**



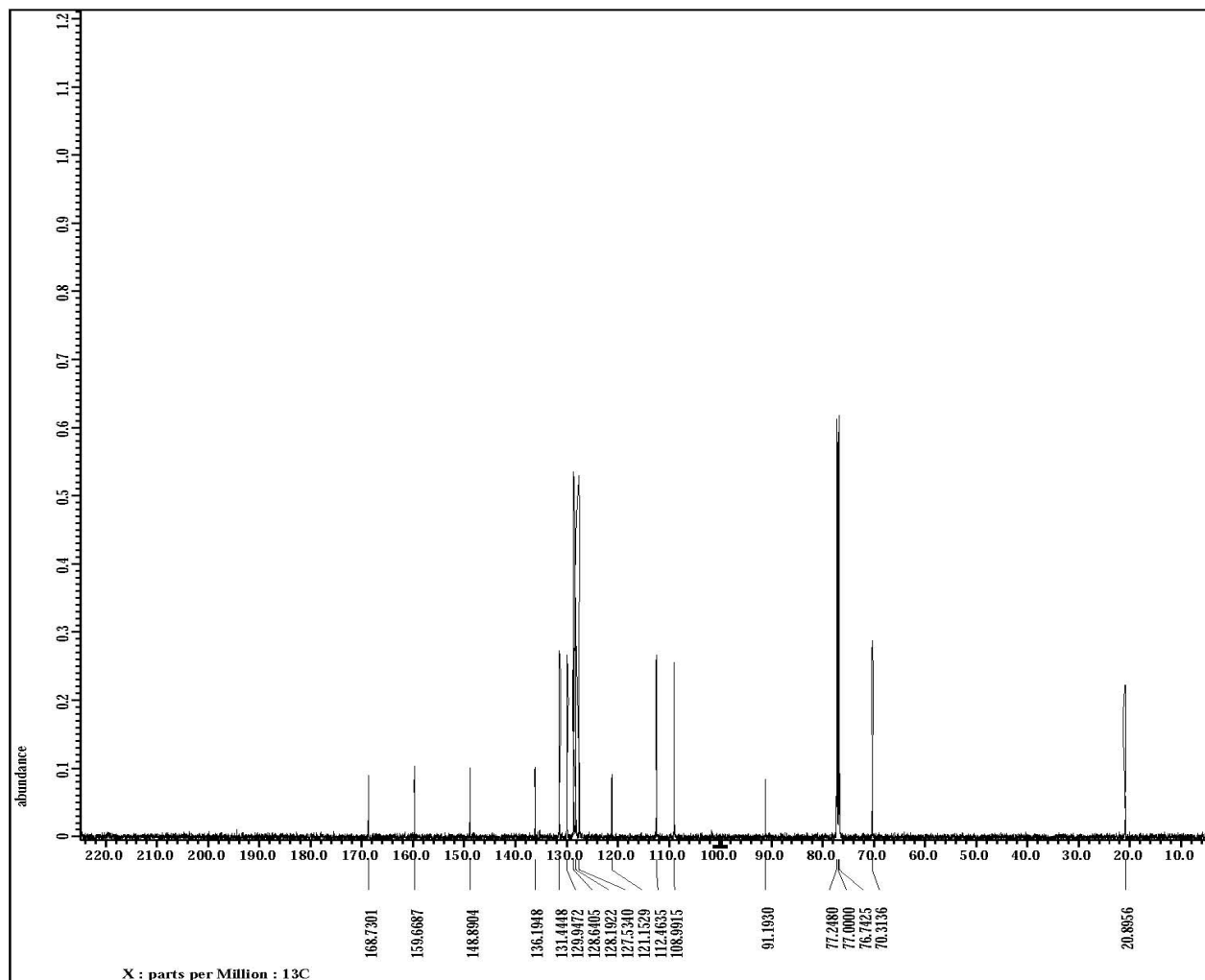
¹H NMR spectra of Compound **22**



¹³C NMR spectra of Compound 22



¹H NMR spectrum of Compound **12ba**



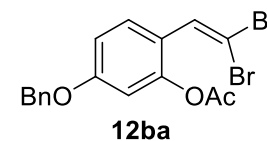
```

Filename      = NMNP13D-13C-3.jdf
Author       = RAJA BABU
Experiment    = single pulse_dec
Sample_id     = S#116902
Solvent       = CHLOROFORM-D
Creation_time = 6-SEP-2016 01:47:12
Revision_time = 21-SEP-2017 17:35:01
Current_time  = 21-SEP-2017 17:40:26

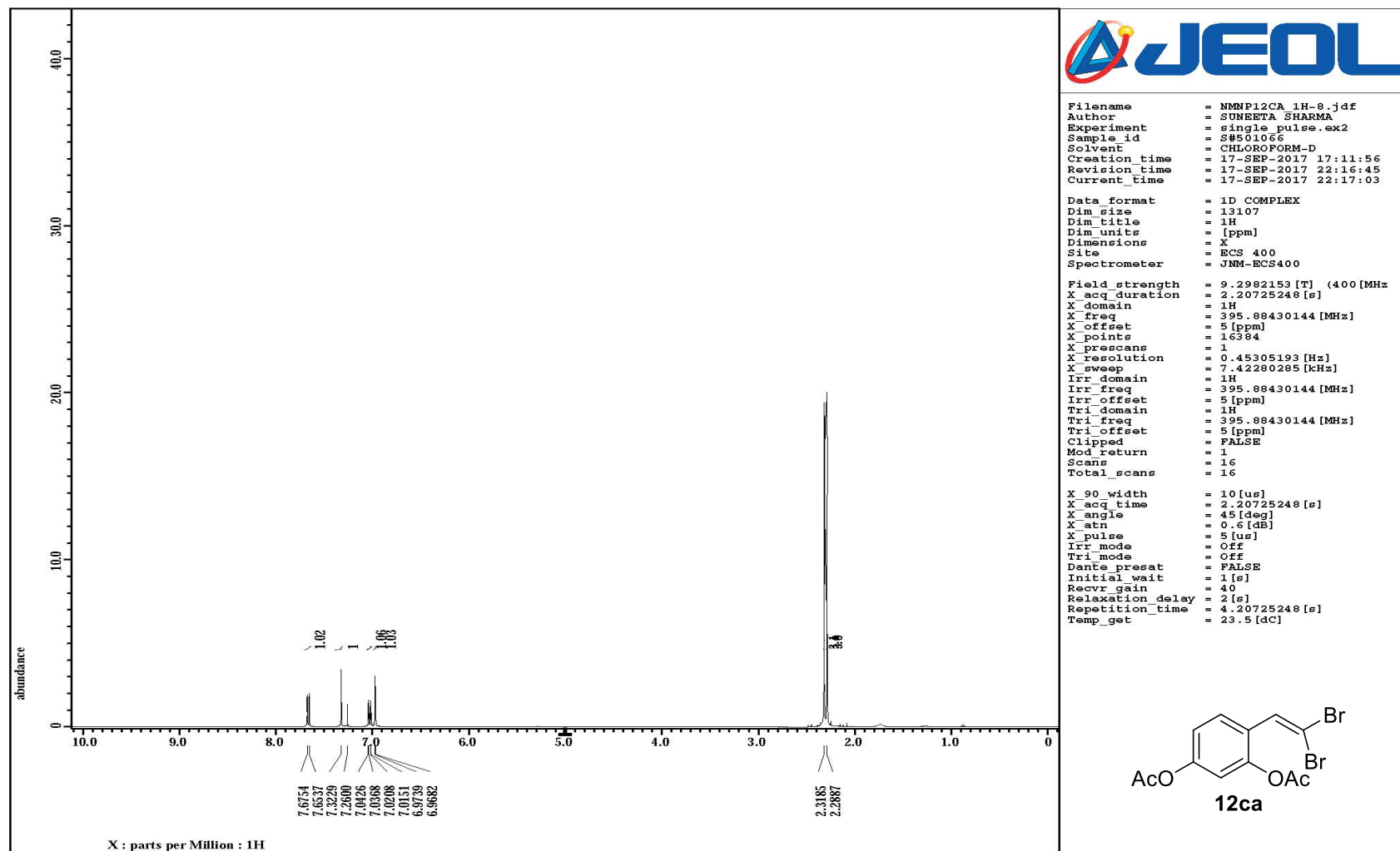
Comment       = single pulse decouple
Data_format   = 1D COMPLEX
Dim_size      = 26214
Dim_title     = 13C
Dim_units     = [ppm]
Dimensions    = X
Site          = ECX 500
Spectrometer  = DELTA2 NMR

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 0.83361792 [s]
X_domain       = 13C
X_freq         = 125.76529768 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034 [Hz]
X_sweep        = 39.3081761 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 1000
Total_scans    = 1000

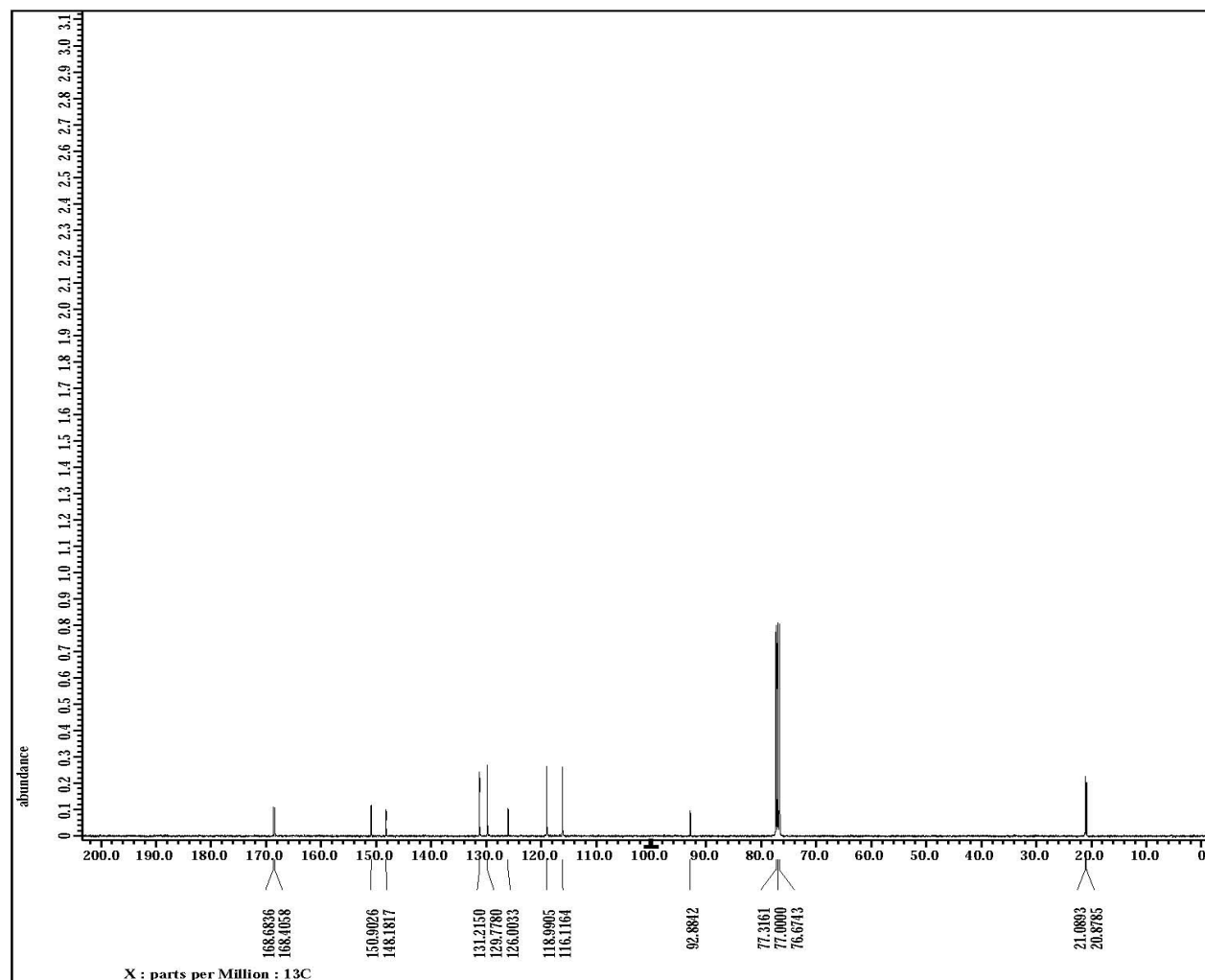
X_90_width     = 11.75 [us]
X_acq_time     = 0.83361792 [s]
X_angle        = 30 [deg]
X_atn          = 7.1 [dB]
X_pulse        = 3.91666667 [us]
Irr_atn_dec    = 19.32 [dB]
Irr_atn_noe    = 19.32 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 1 [s]
Recvr_gain     = 60
Relaxation_delay = 1 [s]
Repetition_time = 1.83361792 [s]
Temp_get       = 403.6 [dC]
  
```



^{13}C NMR spectrum of Compound **12ba**



¹H NMR spectrum of Compound **12ca**



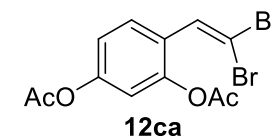
```

Filename      = NMNP12CA 13C-4.jdf
Author       = SUNEETA SHARMA
Experiment    = single pulse_dec
Sample_id     = S#596731
Solvent       = CHLOROFORM-D
Creation_time = 17-SEP-2017 22:42:14
Revision_time = 18-SEP-2017 16:36:03
Current_Time  = 18-SEP-2017 16:45:46

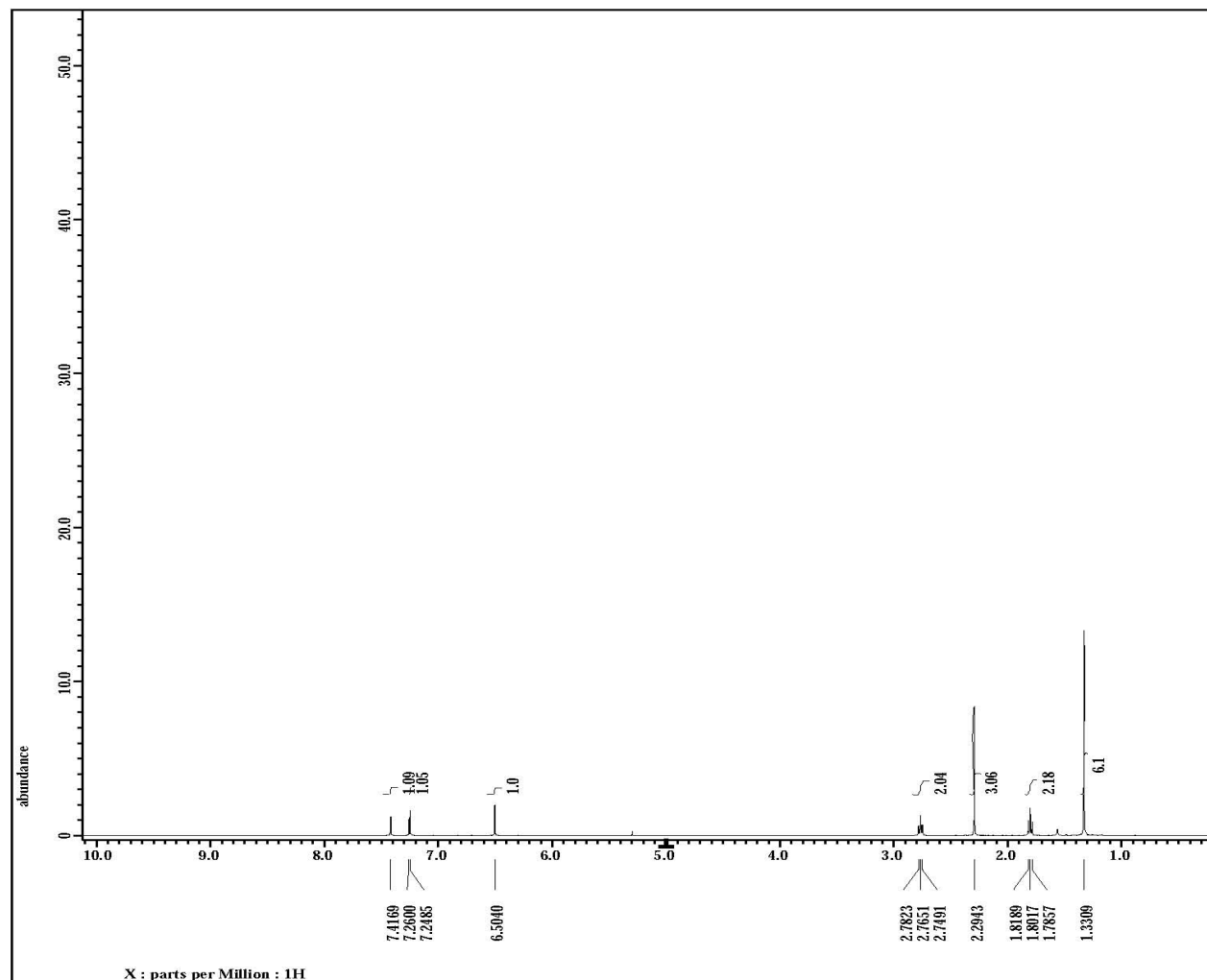
Data_format   = 1D COMPLEX
Dim_size      = 26214
Dim_title     = 13C
Dim_units     = [ppm]
Dimensions    = X
Site          = ECS 400
Spectrometer  = JNM-ECS400

Field_strength = 9.2982153 [T] (400 [MHz]
X_acq_duration = 1.048576 [s]
X_domain       = 13C
X_freq         = 99.54517646 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 0.95367432 [Hz]
X_sweep        = 31.25 [kHz]
Irr_domain     = 1H
Irr_freq       = 395.88430144 [MHz]
Irr_offset     = 5 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 2048
Total_scans    = 2048

X_90_width     = 8.5 [us]
X_acq_time     = 1.048576 [s]
X_angle        = 30 [deg]
X_atn          = 5.8 [dB]
X_pulse        = 2.83333333 [us]
Irr_atn_dec    = 21.936 [dB]
Irr_atn_noe    = 21.936 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 2 [s]
Recvr_gain     = 60
Relaxation_delay = 2 [s]
Repetition_time = 3.048576 [s]
Temp_get       = 24.1 [dC]
  
```



^{13}C NMR spectrum of Compound 12ca



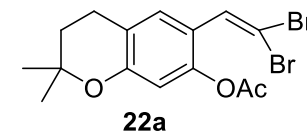
```

Filename      = NM111202 1H-5.jdf
Author       = PRAMOD KUMAR
Experiment   = single pulse.ex2
Sample id    = S#379477
Solvent      = CHLOROFORM-D
Creation time = 12-APR-2016 14:17:57
Revision time = 30-AUG-2016 21:46:37
Current Time = 30-AUG-2016 21:49:17

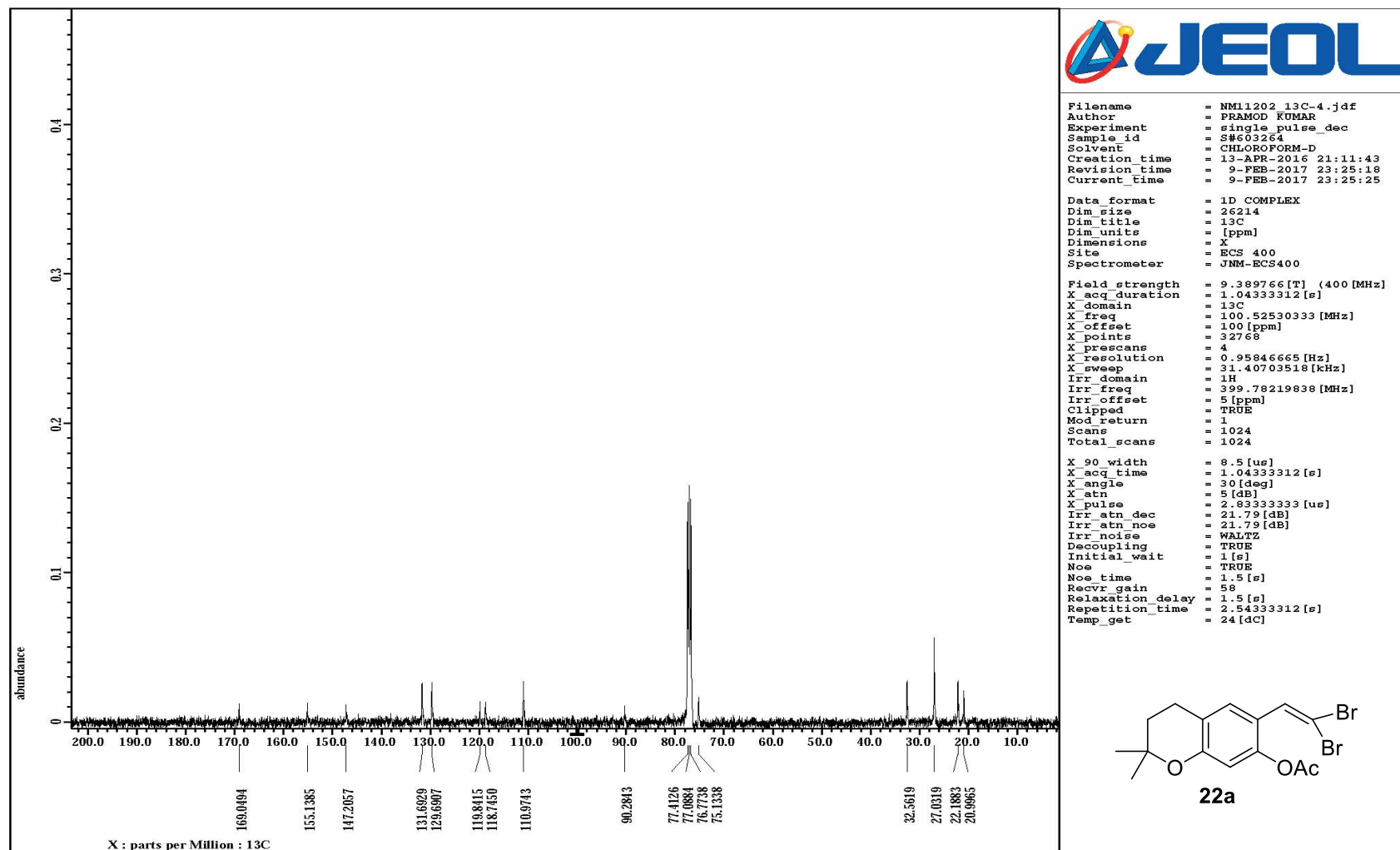
Data format   = 1D COMPLEX
Dim size      = 13107
Dim title     = 1H
Dim units     = [ppm]
Dimensions    = X
Site          = ECS 400
Spectrometer  = JNM-ECS400

Field strength = 9.389766[T] (400 [MHz])
X_acq_duration = 2.18365952[s]
X_domain      = 1H
X_freq        = 399.78219838 [MHz]
X_offset      = 5 [ppm]
X_points      = 16384
X_prescans    = 1
X_resolution  = 0.45794685 [Hz]
X_sweep       = 7.5030012 [kHz]
Irr_domain    = 1H
Irr_freq      = 399.78219838 [MHz]
Irr_offset    = 5 [ppm]
Tri_domain    = 1H
Tri_freq      = 399.78219838 [MHz]
Tri_offset    = 5 [ppm]
Clipped       = FALSE
Mod_return    = 1
Scans         = 16
Total_scans   = 16

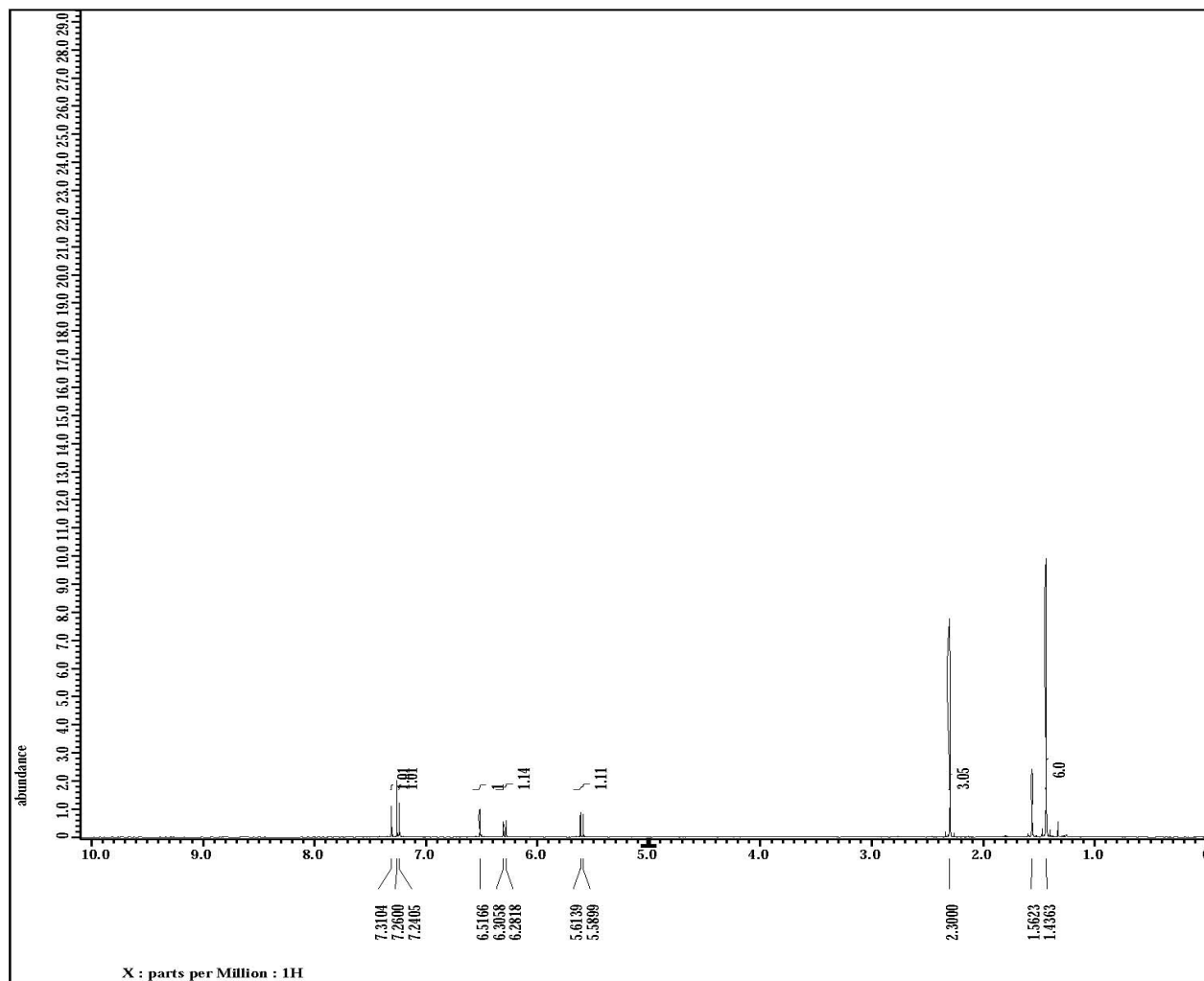
X_90_width    = 10.75 [us]
X_acq_time    = 2.18365952 [s]
X_angle       = 45 [deg]
X_atn         = 1.2 [dB]
X_pulse       = 5.375 [us]
Irr_mode      = Off
Tri_mode      = Off
Dante_preset  = FALSE
Initial_wait  = 1 [s]
Recvr_gain    = 40
Relaxation_delay = 2 [s]
Repetition_time = 4.18365952 [s]
Temp_get      = 23.3 [dC]
  
```



^1H NMR spectra of Compound **22a**



¹³C NMR spectra of Compound **22a**



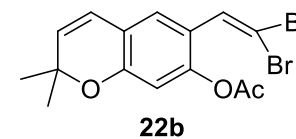
```

Filename      = NMNP18AR 1H-4.jdf
Author       = PRAMOD KUMAR
Experiment   = single pulse.ex2
Sample id    = S#359635
Solvent      = CHLOROFORM-D
Creation time = 22-SEP-2017 13:40:01
Revision time = 22-SEP-2017 15:12:18
Current Time = 22-SEP-2017 15:13:43

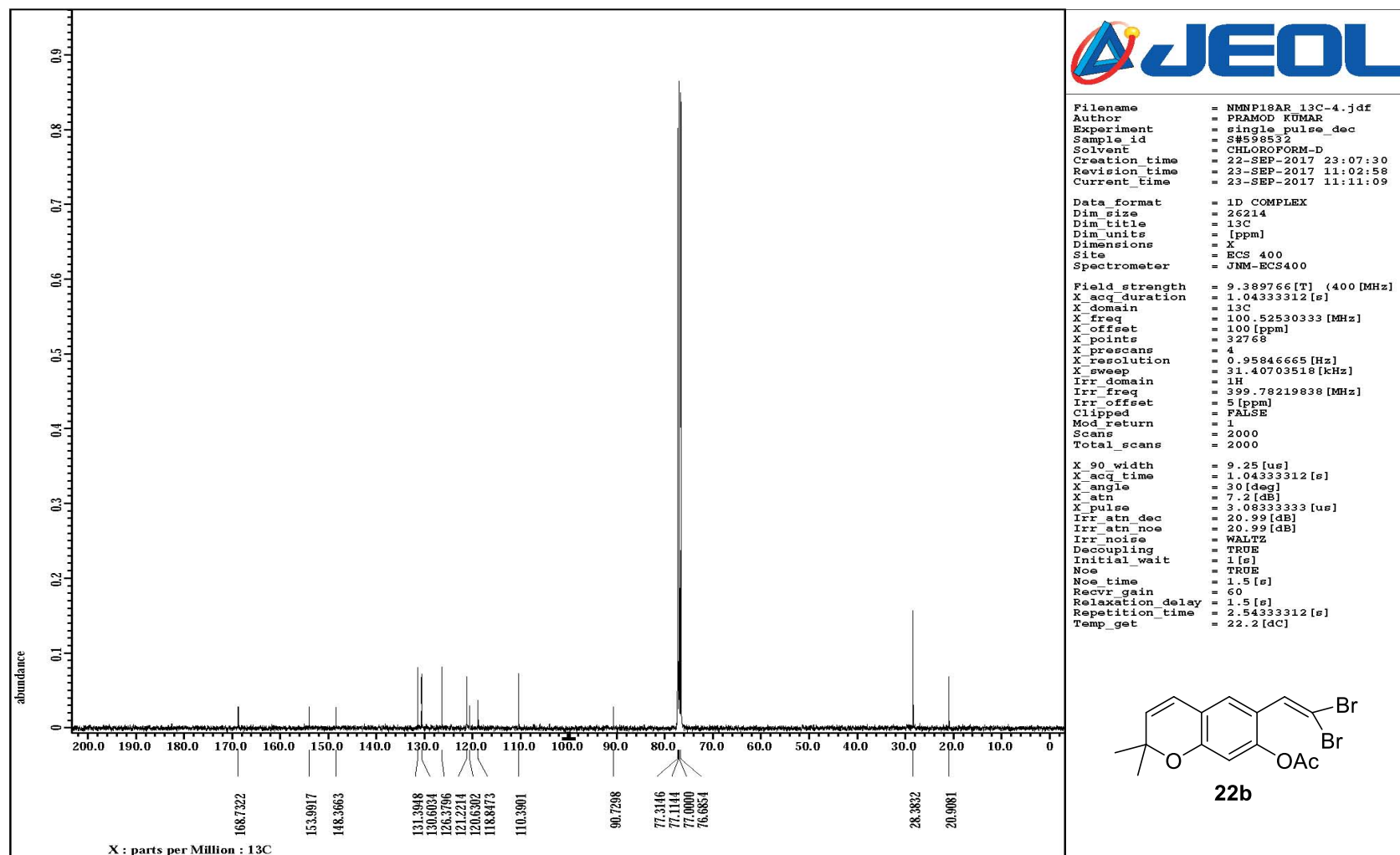
Data format   = 1D COMPLEX
Dim size      = 13107
Dim title     = 1H
Dim units     = [ppm]
Dimensions    = X
Site          = ECS 400
Spectrometer  = JNM-ECS400

Field strength = 9.389766[T] (400 [MHz])
X_acq_duration = 2.18365952[s]
X_domain      = 1H
X_freq        = 399.78219838 [MHz]
X_offset      = 5 [ppm]
X_points      = 16384
X_prescans    = 1
X_resolution  = 0.45794685 [Hz]
X_sweep       = 7.5030012 [kHz]
Irr_domain    = 1H
Irr_freq      = 399.78219838 [MHz]
Irr_offset    = 5 [ppm]
Tri_domain    = 1H
Tri_freq      = 399.78219838 [MHz]
Tri_offset    = 5 [ppm]
Clipped       = FALSE
Mod_return    = 1
Scans         = 16
Total_scans   = 16

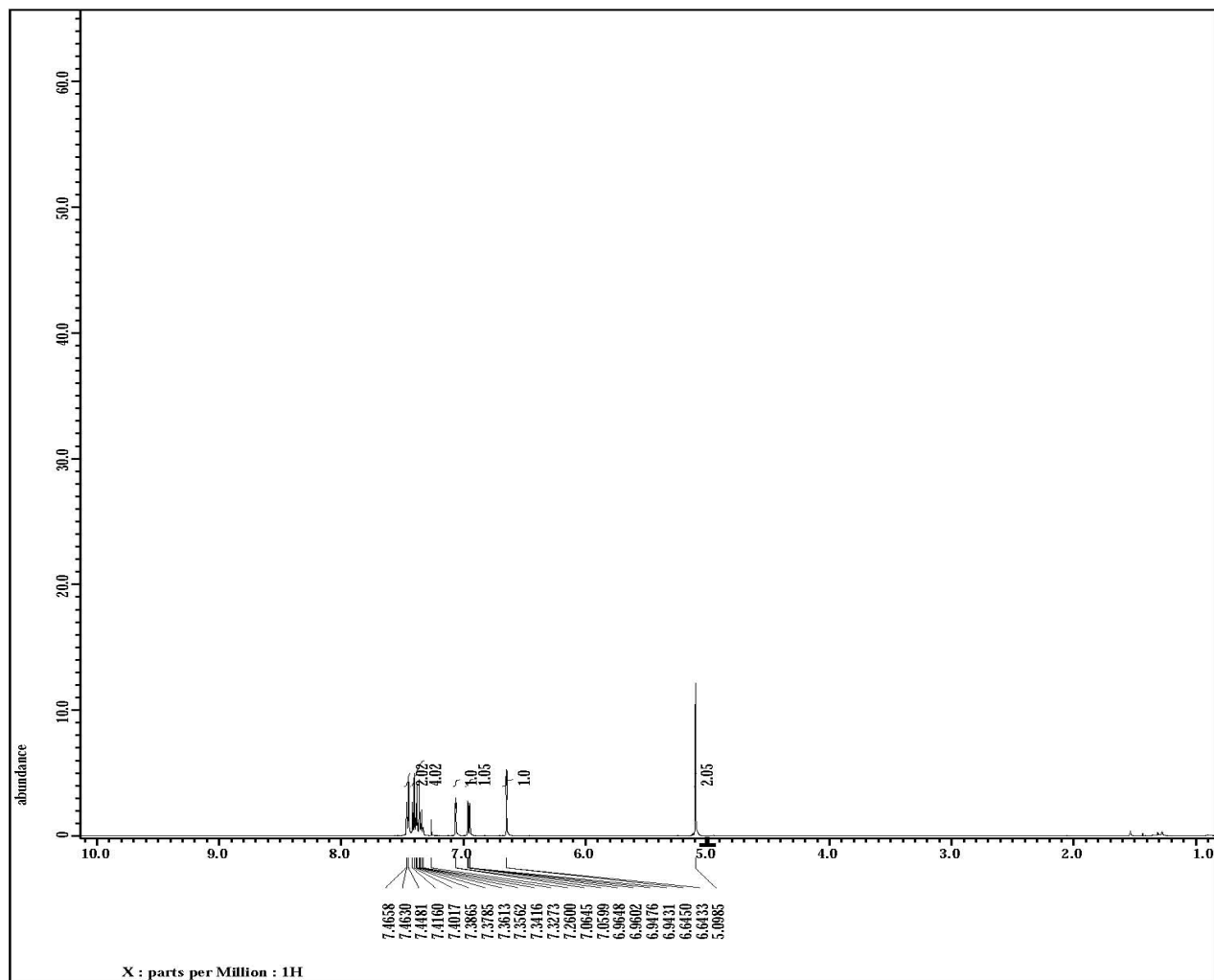
X_90_width    = 10.5 [us]
X_acq_time     = 2.18365952[s]
X_angle       = 45 [deg]
X_atn         = 0.2 [dB]
X_pulse       = 5.25 [us]
Irr_mode      = Off
Tri_mode      = Off
Dante_preset  = FALSE
Initial_wait   = 1 [s]
Recvr_gain     = 40
Relaxation_delay = 2 [s]
Repetition_time = 4.18365952[s]
Temp_get      = 22.5 [dC]
  
```



^1H NMR spectrum of Compound **22b**



^{13}C NMR spectrum of Compound **22b**



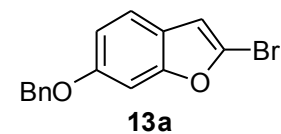
```

Filename      = NMNP13ARR 1H-2.jdf
Author       = RAJA BABU
Experiment   = single pulse.ex2
Sample id    = S#582020
Solvent      = CHLOROFORM-D
Creation time = 25-SEP-2017 13:57:47
Revision time = 26-SEP-2017 15:34:11
Current Time = 26-SEP-2017 15:35:05

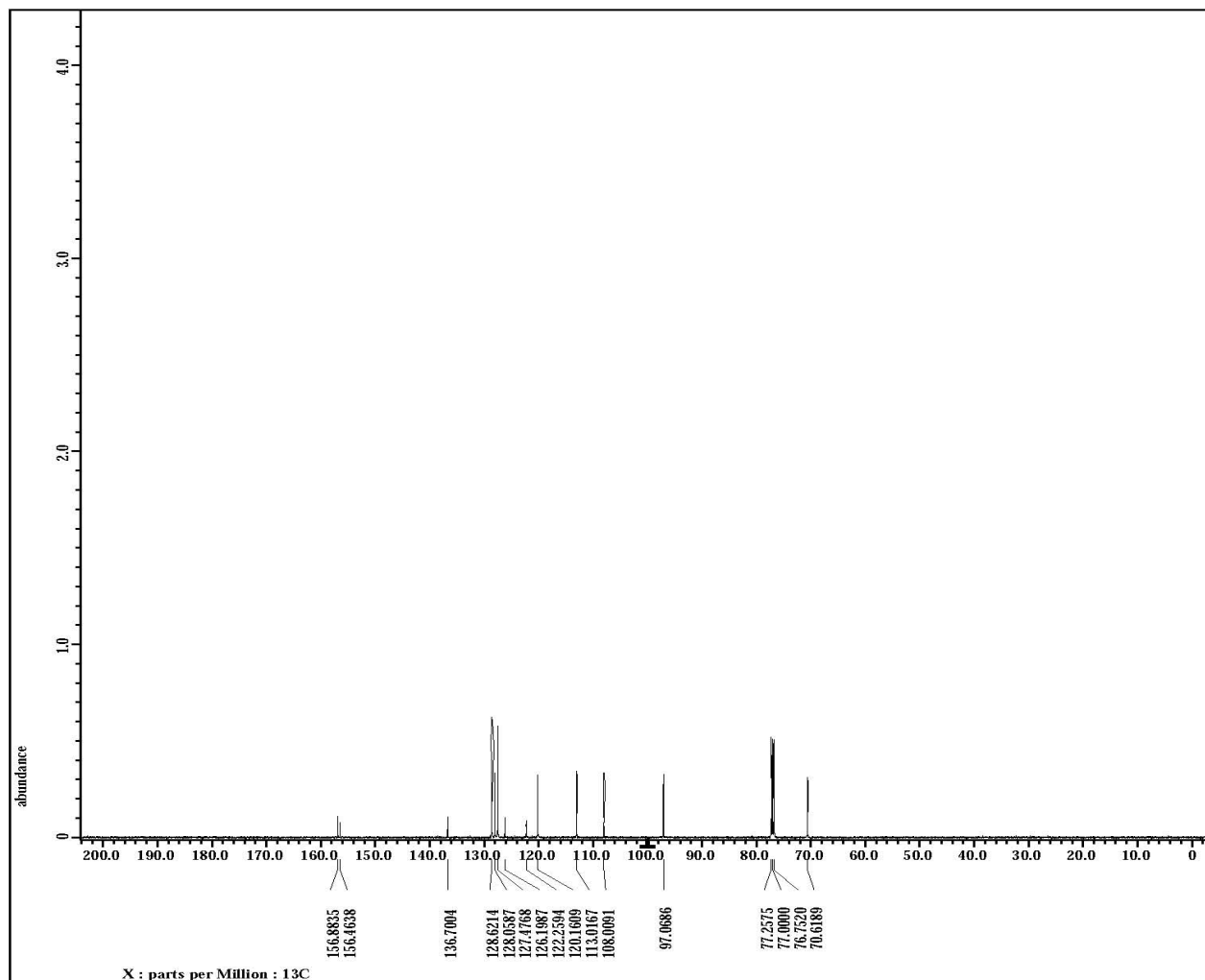
Comment      = single pulse
Data format  = 1D COMPLEX
Dim size     = 52428
Dim title    = 1H
Dim units    = [ppm]
Dimensions   = X
Site         = ECX 500
Spectrometer = DELTA2 NMR

Field strength = 11.7473579 [T] (500 [MH]
X_acq_duration = 1.74587904 [s]
X_domain       = 1H
X_freq         = 500.15991521 [MHz]
X_offset       = 5.0 [ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.57277737 [Hz]
X_sweep        = 9.38438458 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Tri_domain     = 1H
Tri_freq       = 500.15991521 [MHz]
Tri_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 16
Total_scans    = 16

X_90_width     = 15.75 [us]
X_acq_time     = 1.74587904 [s]
X_angle        = 45 [deg]
X_atn          = 3.99 [dB]
X_pulse        = 7.875 [us]
Irr_mode       = Off
Tri_mode       = Off
Dante_presat   = FALSE
Initial_wait   = 1 [s]
Recvr_gain     = 46
Relaxation_delay = 2 [s]
Repetition_time = 3.74587904 [s]
Temp_get       = 396.9 [dC]
  
```



^1H NMR spectrum of Compound **13a**



```

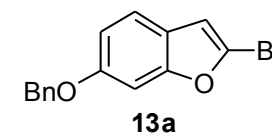
Filename      = NMNP13ARR 13C-3.jdf
Author       = RAJA BABU
Experiment    = single pulse_dec
Sample_id     = S#794142
Solvent       = CHLOROFORM-D
Creation_time = 25-SEP-2017 20:21:35
Revision_time = 26-SEP-2017 15:29:29
Current_Time  = 26-SEP-2017 15:33:33

Comment       = single pulse decouple
Data_format   = 1D COMPLEX
Dim_size      = 26214
Dim_title     = 13C
Dim_units     = [ppm]
Dimensions    = X
Site          = ECX 500
Spectrometer  = DELTA2_NMR

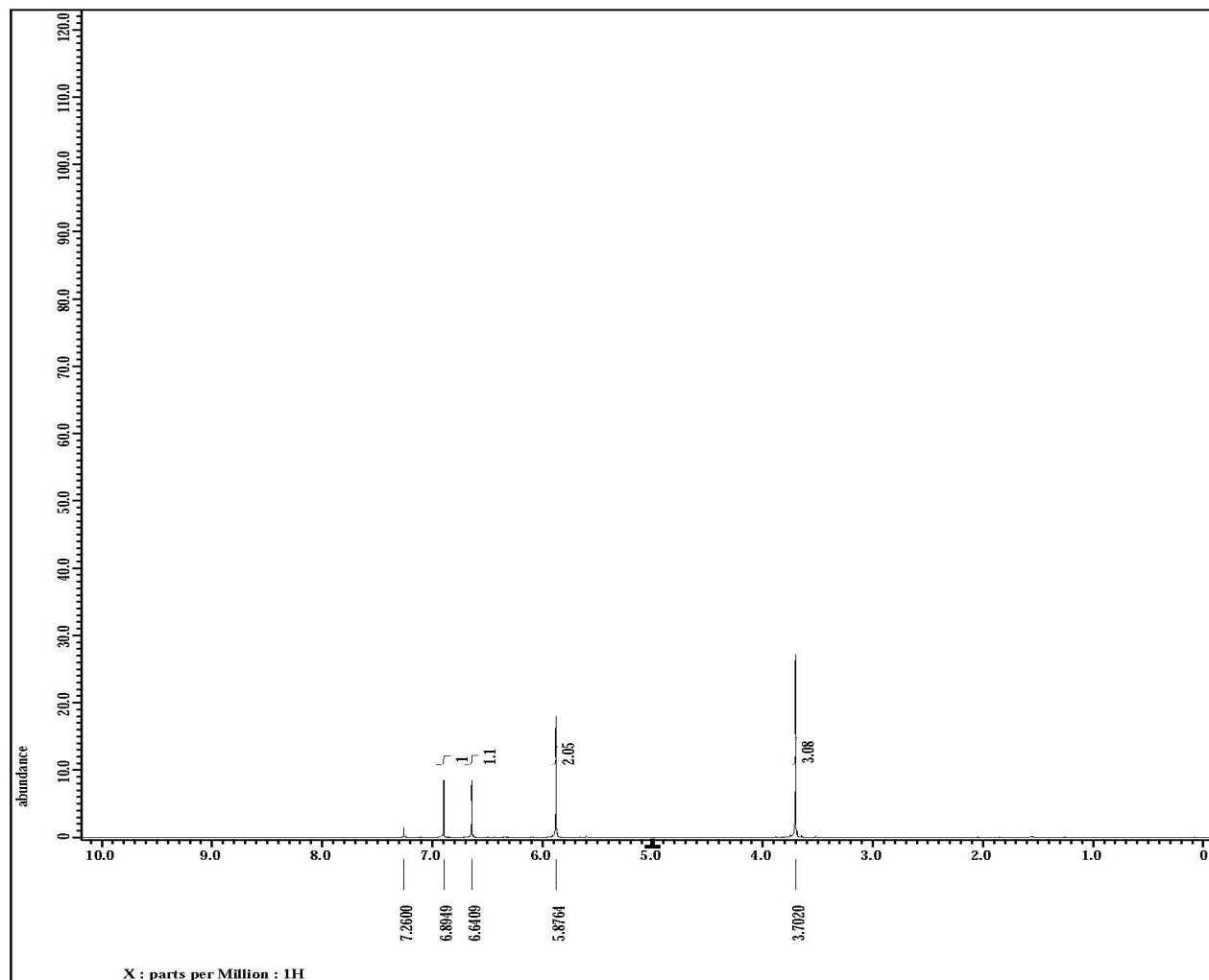
Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 0.83361792 [s]
X_domain       = 13C
X_freq         = 125.76529768 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034 [Hz]
X_sweep        = 39.3081761 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 1024
Total_scans    = 1024

X_90_width     = 11.75 [us]
X_acq_time     = 0.83361792 [s]
X_angle        = 30 [deg]
X_atn          = 7.1 [dB]
X_pulse        = 3.91666667 [us]
Irr_atn_dec    = 19.32 [dB]
Irr_atn_noe    = 19.32 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 1 [s]
Recvr_gain     = 60
Relaxation_delay = 1 [s]
Repetition_time = 1.83361792 [s]
Temp_get       = 395.7 [dC]

```



^{13}C NMR spectrum of Compound **13a**



```

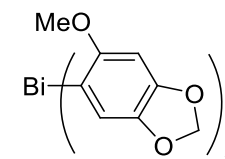
Filename      = NM3240_1H-4.jdf
Author       = Sunesta Sharma
Experiment   = single_pulse.ex2
Sample_id    = S#488294
Solvent      = CHLOROFORM-D
Creation_time = 10-FEB-2014 17:07:17
Revision_time = 12-NOV-2016 13:02:05
Current_time  = 12-NOV-2016 13:03:12

Data_format   = 1D COMPLEX
Dim_size      = 13107
Dim_title     = 1H
Dim_units     = [ppm]
Dimensions    = X
Site          = ECS 400
Spectrometer  = JNM-ECS400

Field_strength = 9.2982153 [T] (400 [MHz]
X_acq_duration = 2.20725248 [s]
X_domain       = 1H
X_freq         = 395.88430144 [MHz]
X_offset       = 5 [ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.45305193 [Hz]
X_sweep        = 7.42280285 [kHz]
Irr_domain     = 1H
Irr_freq       = 395.88430144 [MHz]
Irr_offset     = 5 [ppm]
Tri_domain     = 1H
Tri_freq       = 395.88430144 [MHz]
Tri_offset     = 5 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 16
Total_scans    = 16

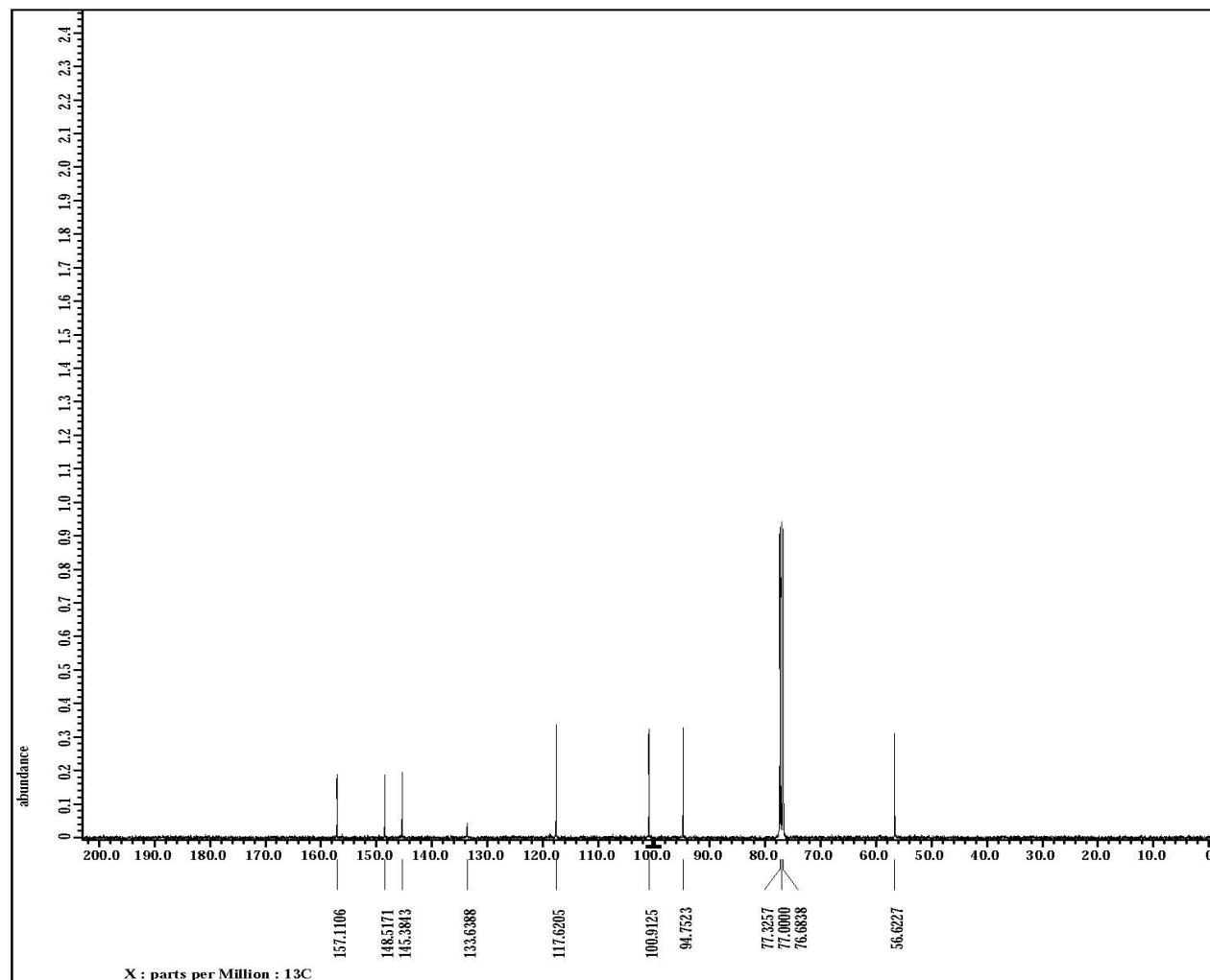
X_90_width     = 10 [us]
X_acq_time      = 2.20725248 [s]
X_angle         = 45 [deg]
X_atn          = 0.6 [dB]
X_pulse        = 5 [us]
Irr_mode        = Off
Tri_mode        = Off
Dante_preset    = FALSE
Initial_wait    = 1 [s]
Recvr_gain      = 40
Relaxation_delay = 2 [s]
Repetition_time = 4.20725248 [s]
Temp_get        = 20.9 [dC]

```



TAB-3

¹H NMR spectra of **TAB-3**



```

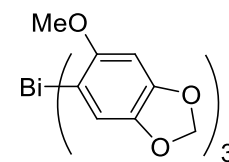
Filename      = NM3240_13C-5.jdf
Author        = Suneeta Sharma
Experiment     = single pulse_dec
Sample id     = S#493004
Solvent       = CHLOROFORM-D
Creation time  = 10-FEB-2014 17:34:20
Revision time  = 12-NOV-2016 13:05:23
Current time   = 12-NOV-2016 13:05:31

Data format   = 1D COMPLEX
Dim size      = 26214
Dim title     = 13C
Dim units     = [ppm]
Dimensions    = X
Site          = ECS 400
Spectrometer  = JNM-ECS400

Field strength = 9.2982153 [T] (400 [MHz]
X_acq duration = 1.048576 [s]
X_domain      = 13C
X_freq        = 99.54517646 [MHz]
X_offset      = 100 [ppm]
X_points      = 32768
X_prescans    = 4
X_resolution  = 0.95367432 [Hz]
X_sweep       = 31.25 [kHz]
F1 domain     = 1H
F1_freq       = 395.88430144 [MHz]
F1_offset     = 5 [ppm]
Clipped       = FALSE
Mod return    = 1
Scans         = 502.0
Total_scans   = 502.0

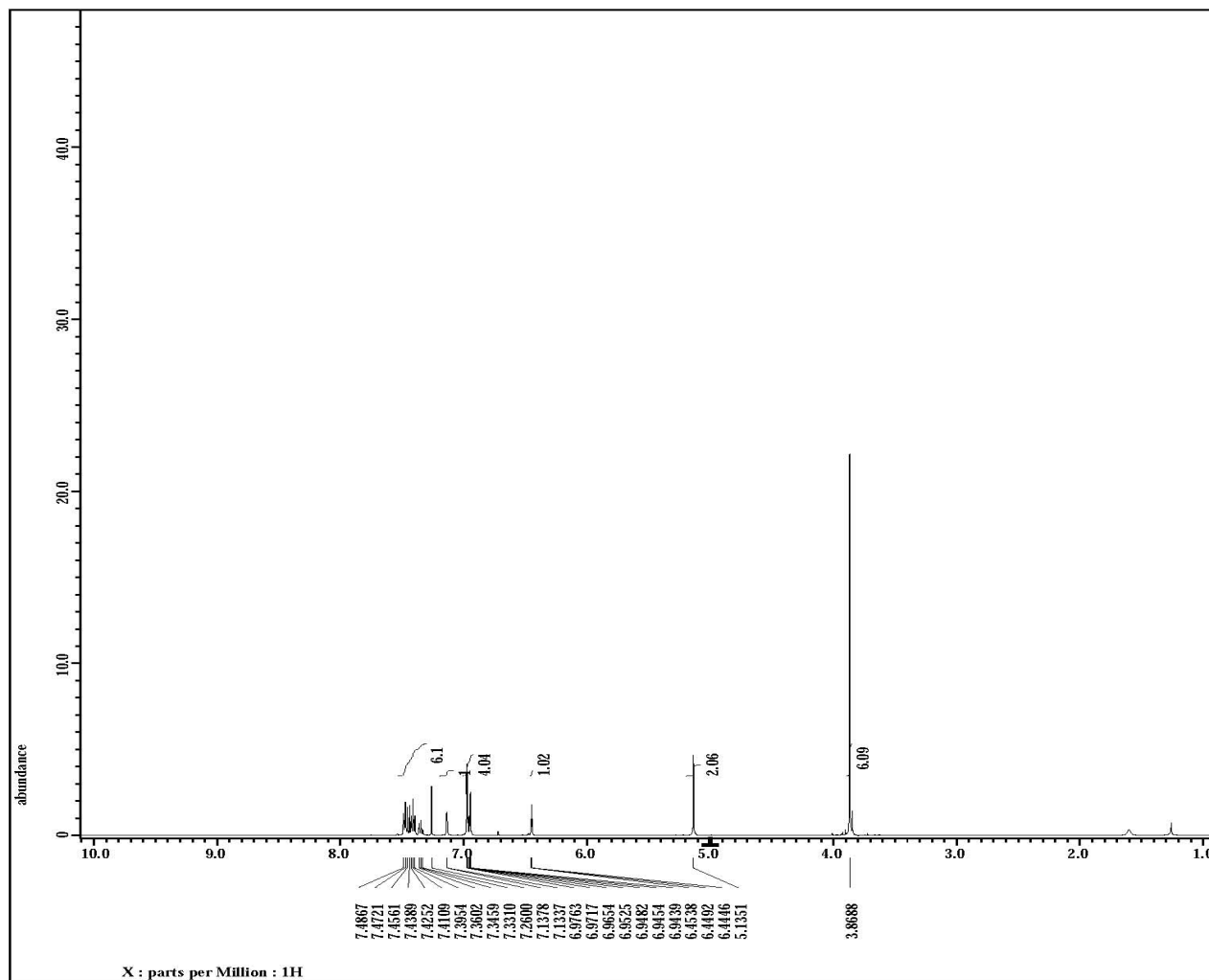
X_90 width    = 8 [us]
X_acq time    = 1.048576 [s]
X_angle       = 30 [deg]
X_atn         = 5.8 [dB]
X_pulse       = 2.66666667 [us]
Irr_atn_dec   = 21.936 [dB]
Irr_atn_noe   = 21.936 [dB]
Irr_noise     = WALTZ2
Decoupling    = TRUE
Initial_wait  = 1 [s]
Noe           = TRUE
Noe time      = 2 [s]
RecVr gain    = 58
Relaxation delay = 2 [s]
Repetition_time = 3.048576 [s]
Temp_get      = 21 [dC]

```



TAB-3

^{13}C NMR spectra of **TAB-3**



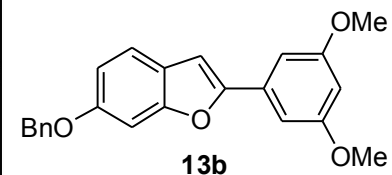
```

Filename      = NMP2NP13A-1H-6.jdf
Author       = RAJA BABU
Experiment    = single pulse.ex2
Sample id    = S#374996
Solvent      = CHLOROFORM-D
Creation time = 13-SEP-2017 08:12:47
Revision time = 18-SEP-2017 17:37:31
Current Time  = 18-SEP-2017 17:39:55

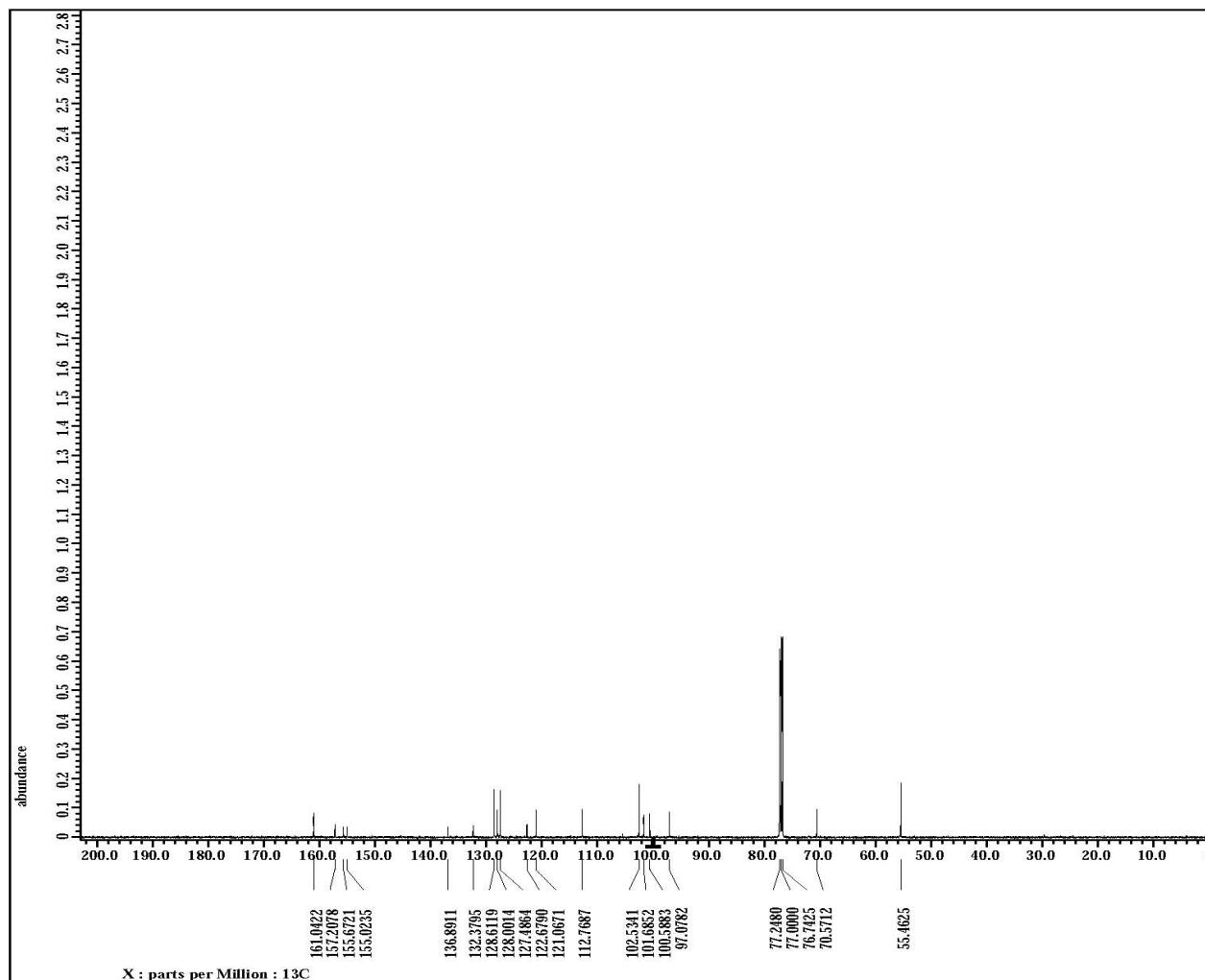
Comment      = single pulse
Data format  = 1D COMPLEX
Dim size     = 52428
Dim title    = 1H
Dim units    = [ppm]
Dimensions   = X
Site         = ECX 500
Spectrometer = DELTA2_NMR

Field strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain       = 1H
X_freq         = 500.15991521 [MHz]
X_offset       = 5.0 [ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.57277737 [Hz]
X_sweep        = 9.38438458 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Tri_domain     = 1H
Tri_freq       = 500.15991521 [MHz]
Tri_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 16
Total_scans    = 16

X_90_width     = 15.75 [us]
X_acq_time     = 1.74587904 [s]
X_angle        = 45 [deg]
X_atn          = 3.99 [dB]
X_pulse        = 7.875 [us]
Irr_mode       = Off
Tri_mode       = Off
Dante_presat   = FALSE
Initial_wait   = 1 [s]
Recvr_gain     = 50
Relaxation_delay = 2 [s]
Repetition_time = 3.74587904 [s]
Temp_get       = 391.4 [dC]
  
```



^1H NMR spectrum of Compound **13b**



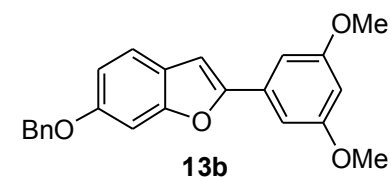
```

Filename      = NM2NP13A-13C-4.jdf
Author       = RAJA BABU
Experiment    = single pulse_dec
Sample_id     = S#120729
Solvent       = CHLOROFORM-D
Creation_time = 14-SEP-2017 02:09:03
Revision_time = 18-SEP-2017 17:42:46
Current_time  = 18-SEP-2017 17:47:28

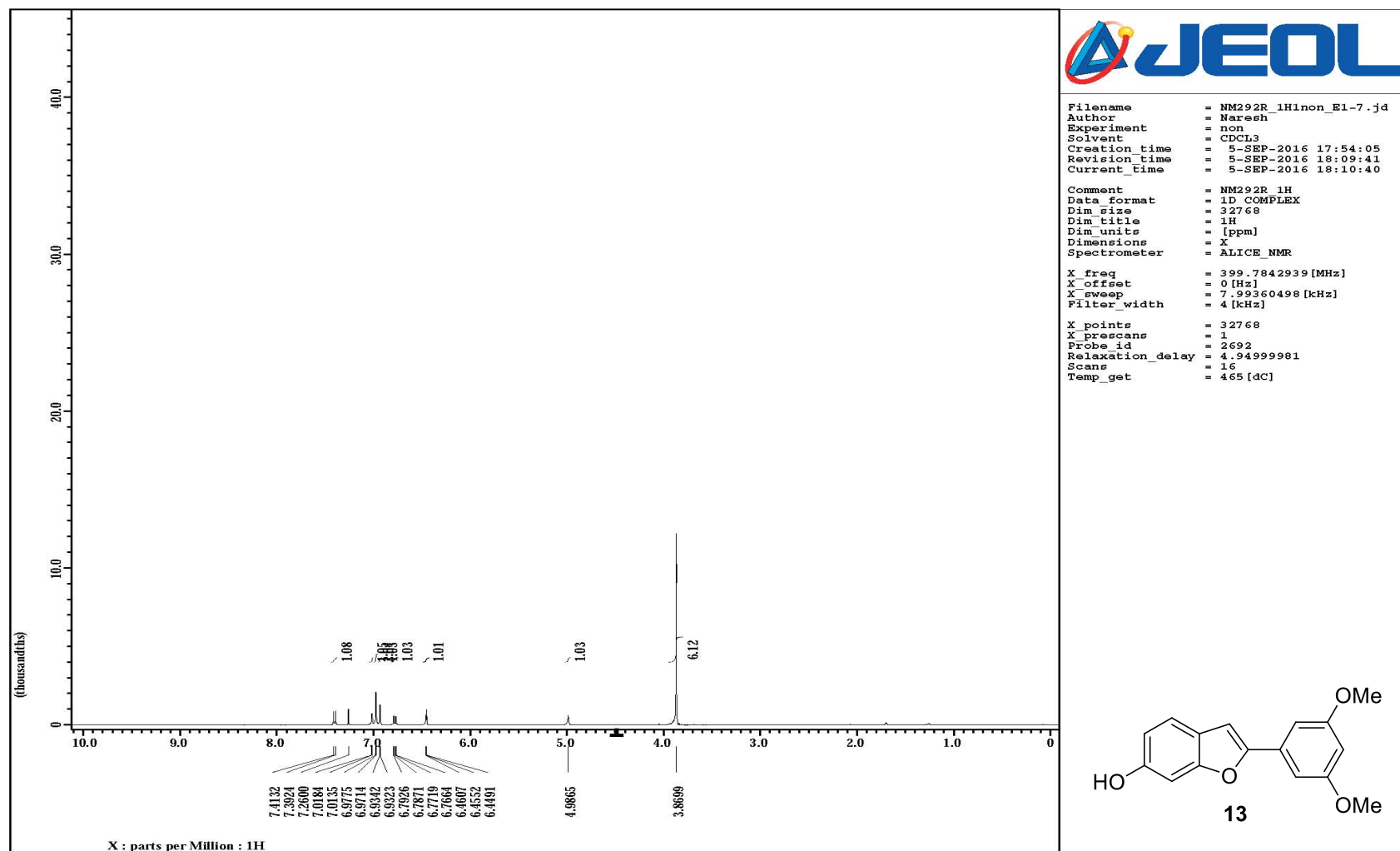
Comment      = single pulse decouple
Data_format   = 1D COMPLEX
Dim_size      = 26214
Dim_title     = 13C
Dim_units     = [ppm]
Dimensions    = X
Site          = ECX 500
Spectrometer  = DELTA2_NMR

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 0.83361792 [s]
X_domain       = 13C
X_freq         = 125.76529768 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034 [Hz]
X_sweep        = 39.3081761 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 2000
Total_scans    = 2000

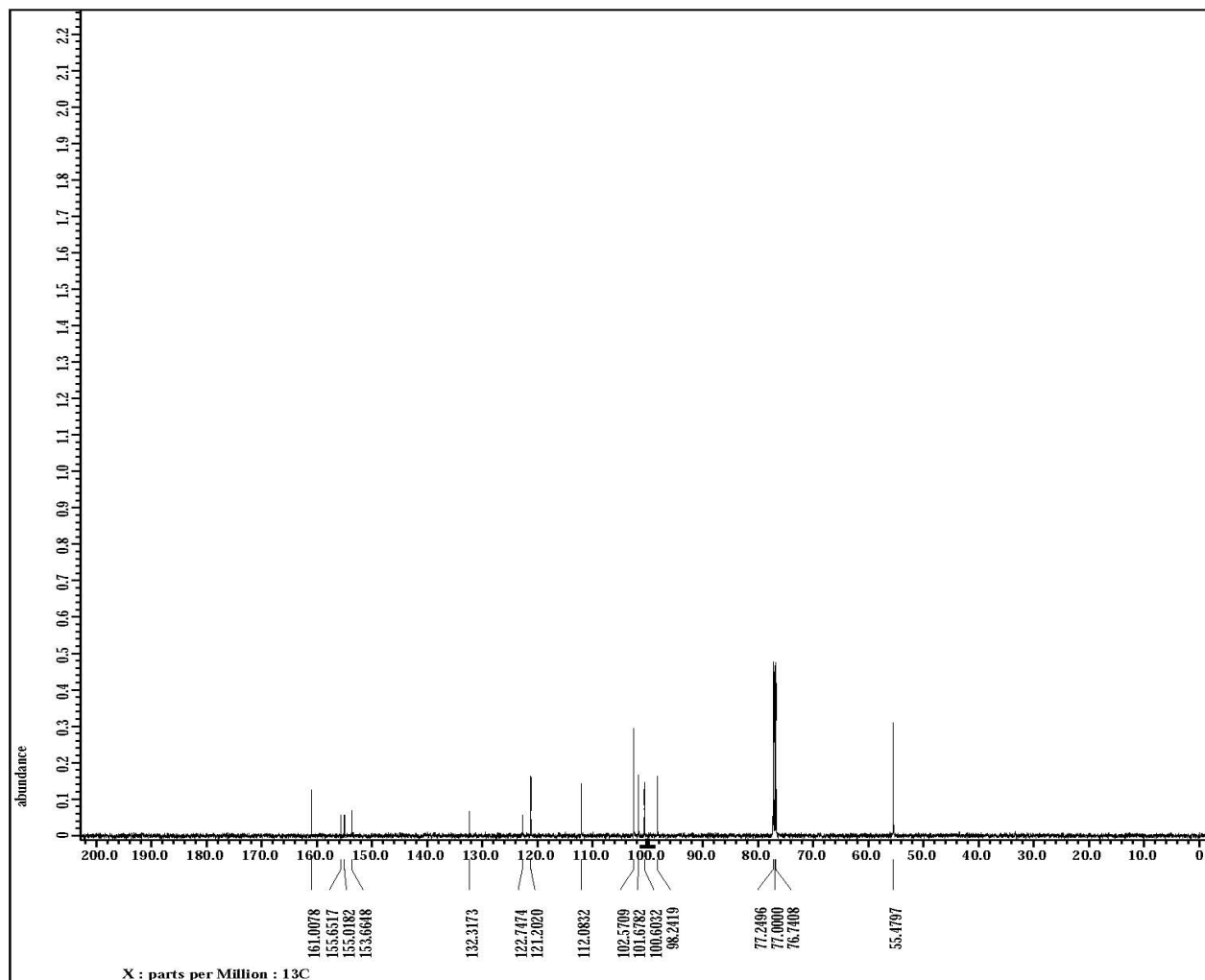
X_90_width     = 11.75 [us]
X_acq_time     = 0.83361792 [s]
X_angle        = 30 [deg]
X_atn          = 7.1 [dB]
X_pulse        = 3.91666667 [us]
Irr_atn_dec    = 19.32 [dB]
Irr_atn_noe    = 19.32 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 1 [s]
Recvr_gain     = 60
Relaxation_delay = 1 [s]
Repetition_time = 1.83361792 [s]
Temp_get       = 393.5 [dC]
  
```



^{13}C NMR spectrum of Compound **13b**



¹H NMR spectra of Compound 13



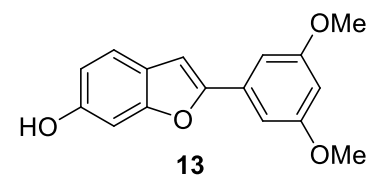
```

Filename      = NM292R_13C-5.jdf
Author       = N.Ahmed
Experiment   = single_pulse_dec
Sample_id    = S#358544
Solvent      = CHLOROFORM-D
Creation_time = 11-OCT-2012 09:00:11
Revision_time = 5-SEP-2016 18:17:01
Current_Time = 5-SEP-2016 18:17:08

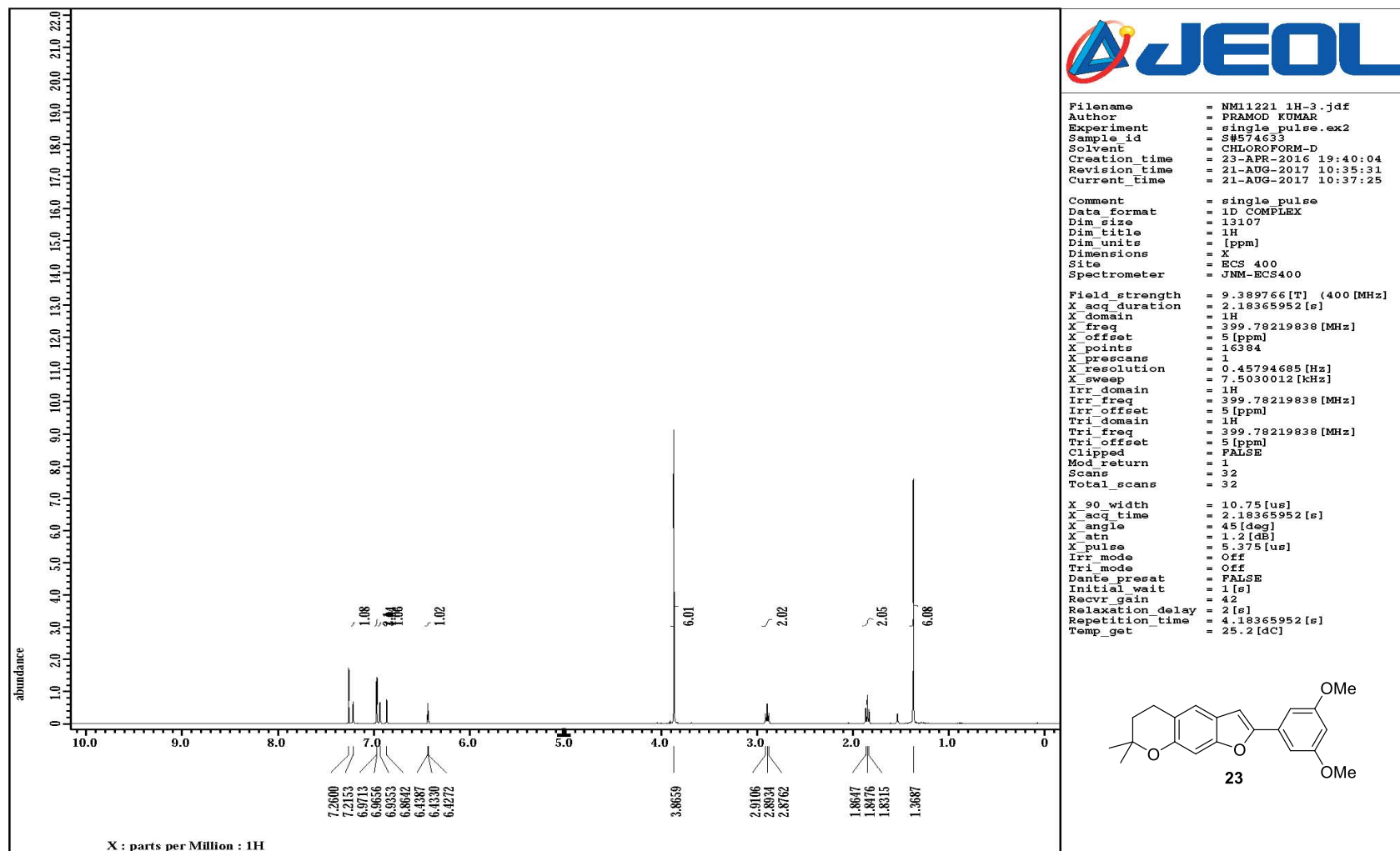
Data_format  = 1D_COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECX 500
Spectrometer = DELTA2_NMR

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 0.82837504 [s]
X_domain       = 13C
X_freq         = 125.76529768 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.20718268 [Hz]
X_sweep        = 39.55696203 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 447
Total_scans    = 447

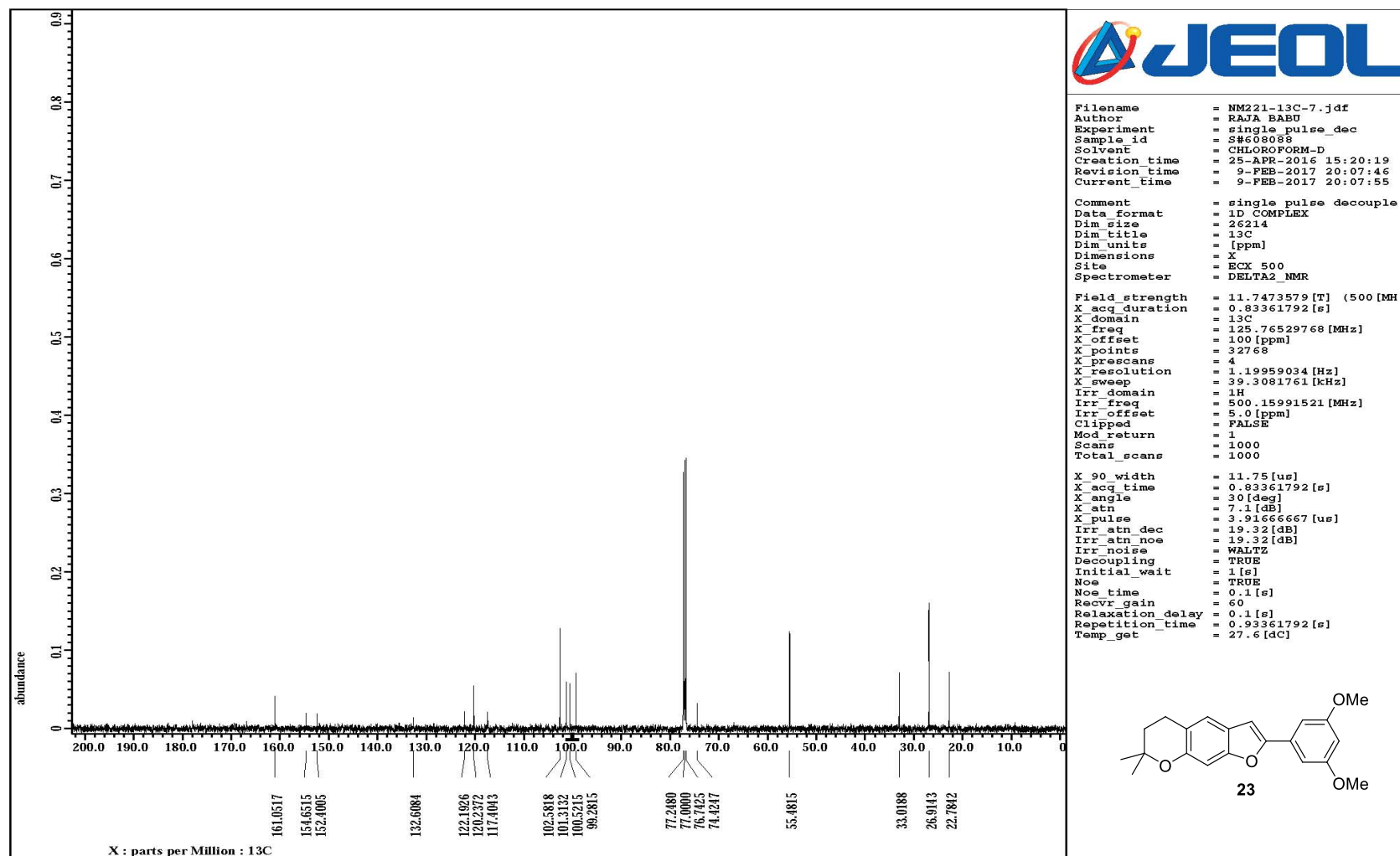
X_90_width    = 9.62 [us]
X_acq_time     = 0.82837504 [s]
X_angle        = 30 [deg]
X_atn          = 7.1 [dB]
X_pulse        = 3.20666667 [us]
Irr_atn_dec    = 19.5 [dB]
Irr_atn_noe    = 21.5 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 0.6 [s]
Recvr_gain     = 58
Relaxation_delay = 0.6 [s]
Repetition_time = 1.42837504 [s]
Temp_get       = 20.7 [dC]
  
```



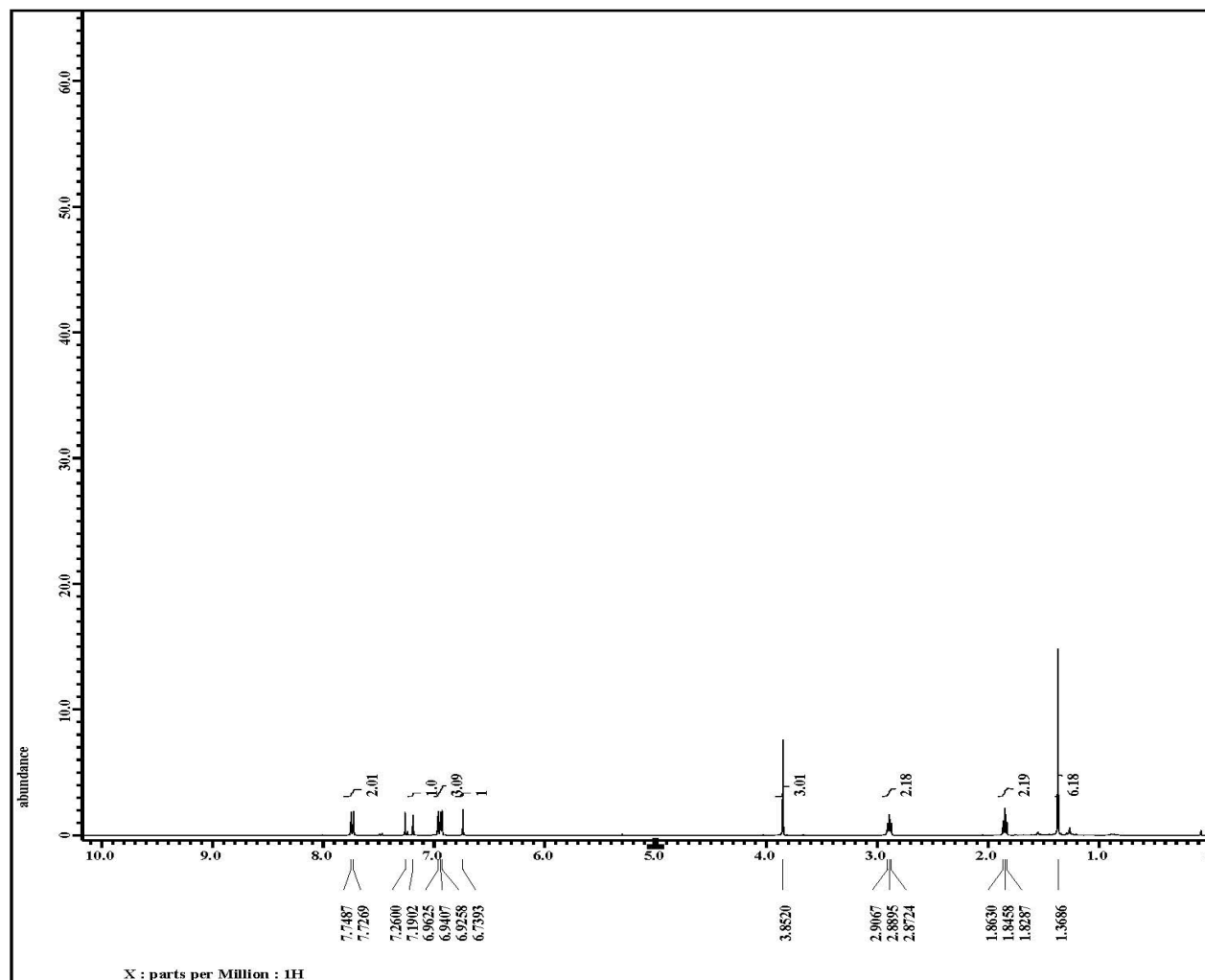
^{13}C NMR spectra of Compound 13



¹H NMR spectra of Compound 23



¹³C NMR spectra of Compound 23



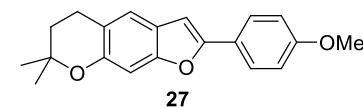
```

Filename      = NM11228R4_1H-5.jdf
Author       = Bhanu Tiwari
Experiment   = single pulse.ex2
Sample id    = S#373288
Solvent      = CHLOROFORM-D
Creation_time = 29-APR-2016 13:47:04
Revision_time = 30-AUG-2016 22:35:15
Current_time  = 30-AUG-2016 22:35:58

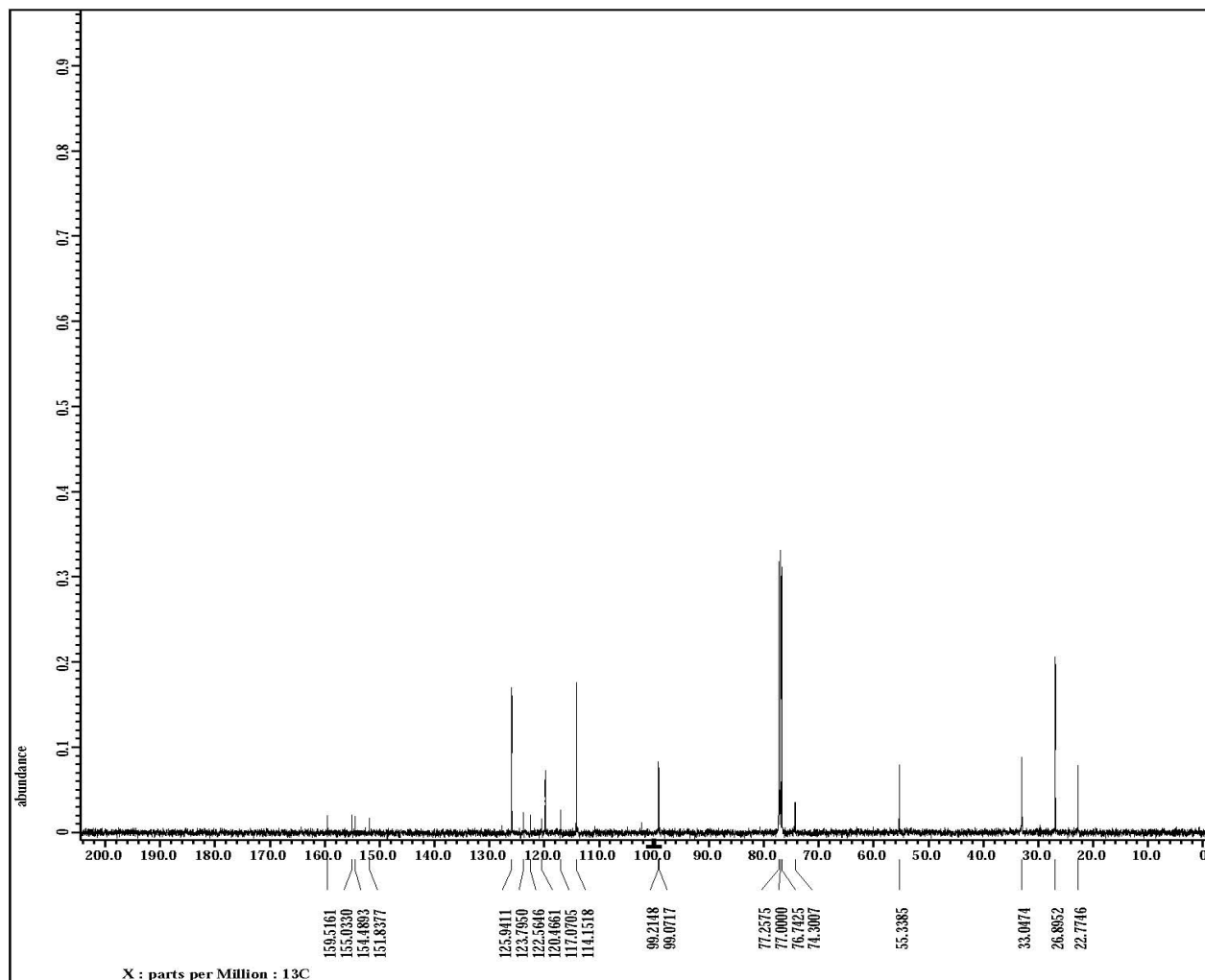
Data format   = 1D COMPLEX
Dim size      = 13107
Dim title     = 1H
Dim units     = [ppm]
Dimensions    = X
Site          = ECS 400
Spectrometer  = JNM-ECS400

Field strength = 9.2982153 [T] (400 [MHz]
X_acq_duration = 2.20725248 [s]
X_domain       = 1H
X_freq         = 395.88430144 [MHz]
X_offset       = 5 [ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.45305193 [Hz]
X_sweep        = 7.42280285 [kHz]
Irr_domain     = 1H
Irr_freq       = 395.88430144 [MHz]
Irr_offset     = 5 [ppm]
Tri_domain     = 1H
Tri_freq       = 395.88430144 [MHz]
Tri_offset     = 5 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 32
Total_scans    = 32

X_90_width     = 10.25 [us]
X_acq_time     = 2.20725248 [s]
X_angle        = 45 [deg]
X_atn          = 0.6 [dB]
X_pulse        = 5.125 [us]
Irr_mode       = Off
Tri_mode       = Off
Dante_preset   = FALSE
Initial_wait   = 1 [s]
Recvr_gain     = 44
Relaxation_delay = 2 [s]
Repetition_time = 4.20725248 [s]
Temp_get       = 23.8 [dC]
  
```



^1H NMR spectra of Compound **27**



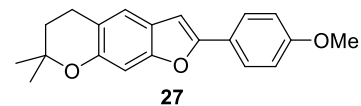
```

Filename      = NM11228RH-13C-6.jdf
Author       = RAJA BABU
Experiment   = single pulse_dec
Sample_id    = S#58825
Solvent      = CHLOROFORM-D
Creation_time = 30-APR-2016 00:06:17
Revision_time = 4-SEP-2016 16:08:26
Current_Time  = 4-SEP-2016 16:09:10

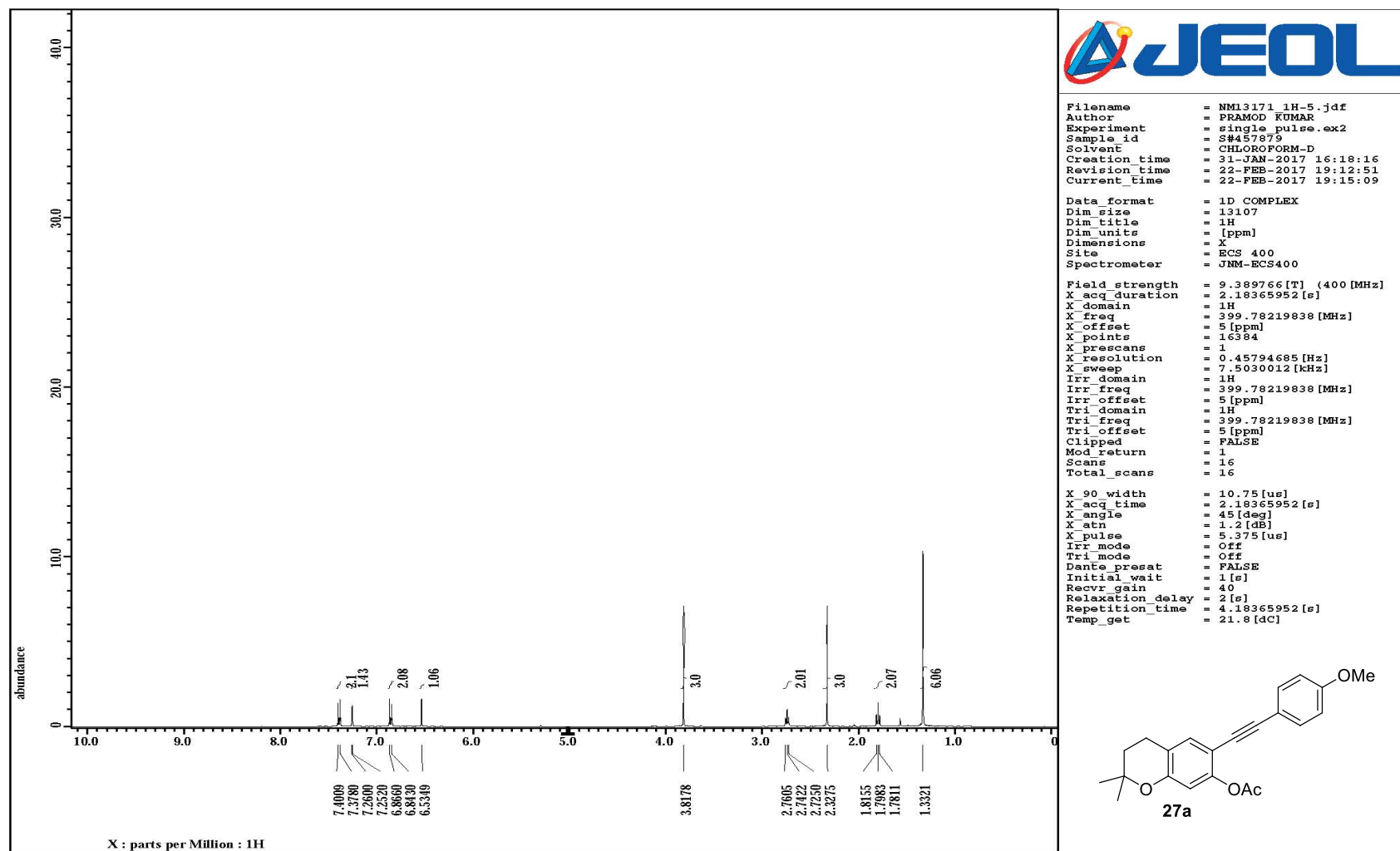
Comment      = single pulse decouple
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECX 500
Spectrometer = DELTA2_NMR

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 0.83361792 [s]
X_domain       = 13C
X_freq         = 125.76529768 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034 [Hz]
X_sweep        = 39.3081761 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 1000
Total_scans    = 1000

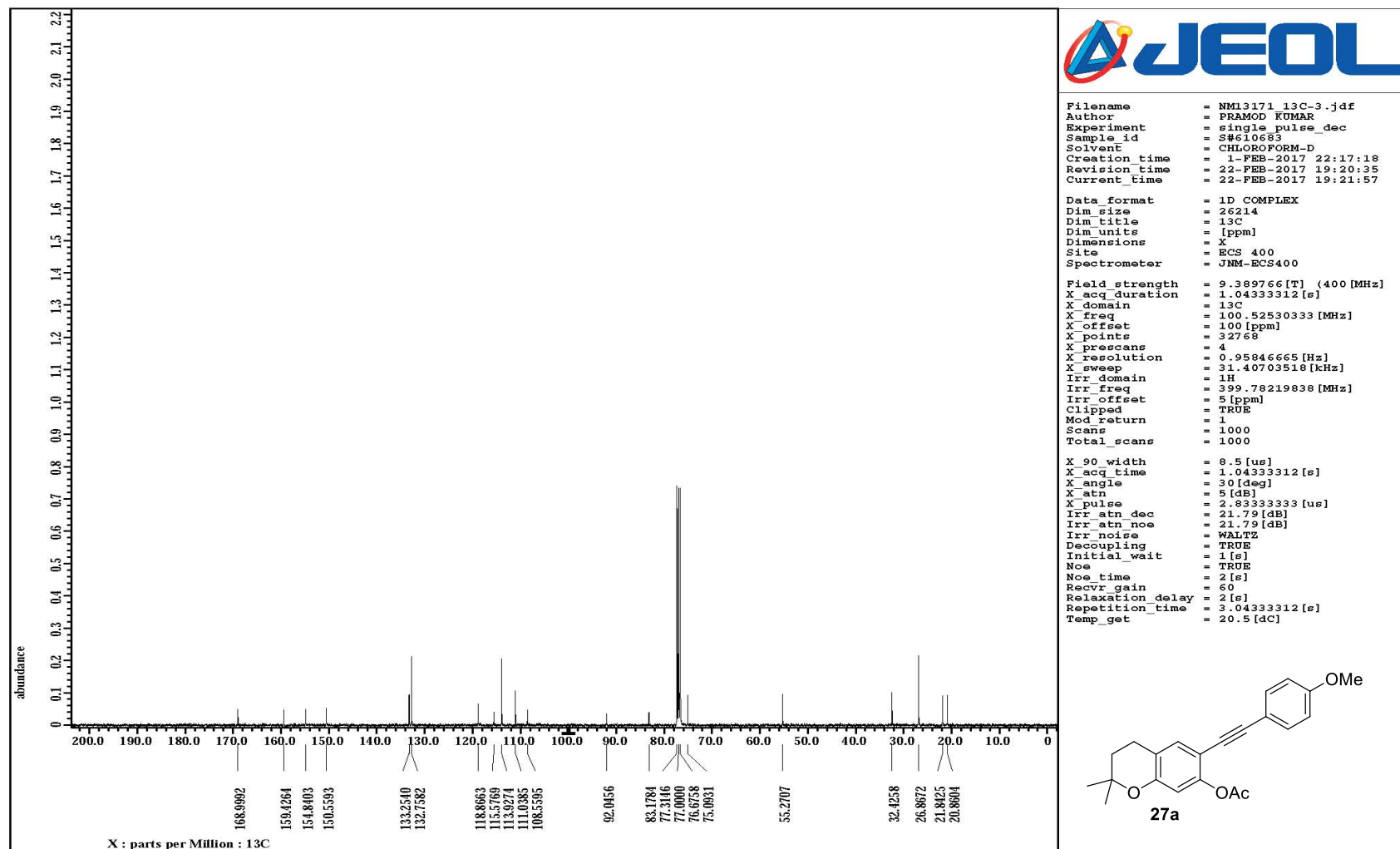
X_90_width    = 11.75 [us]
X_acq_time     = 0.83361792 [s]
X_angle        = 30 [deg]
X_atn          = 7.1 [dB]
X_pulse        = 3.91666667 [us]
Irr_atn_dec    = 19.32 [dB]
Irr_atn_noe    = 19.32 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 0.1 [s]
Recvr_gain     = 60
Relaxation_delay = 0.1 [s]
Repetition_time = 0.93361792 [s]
Temp_get       = 395.7 [dC]
  
```



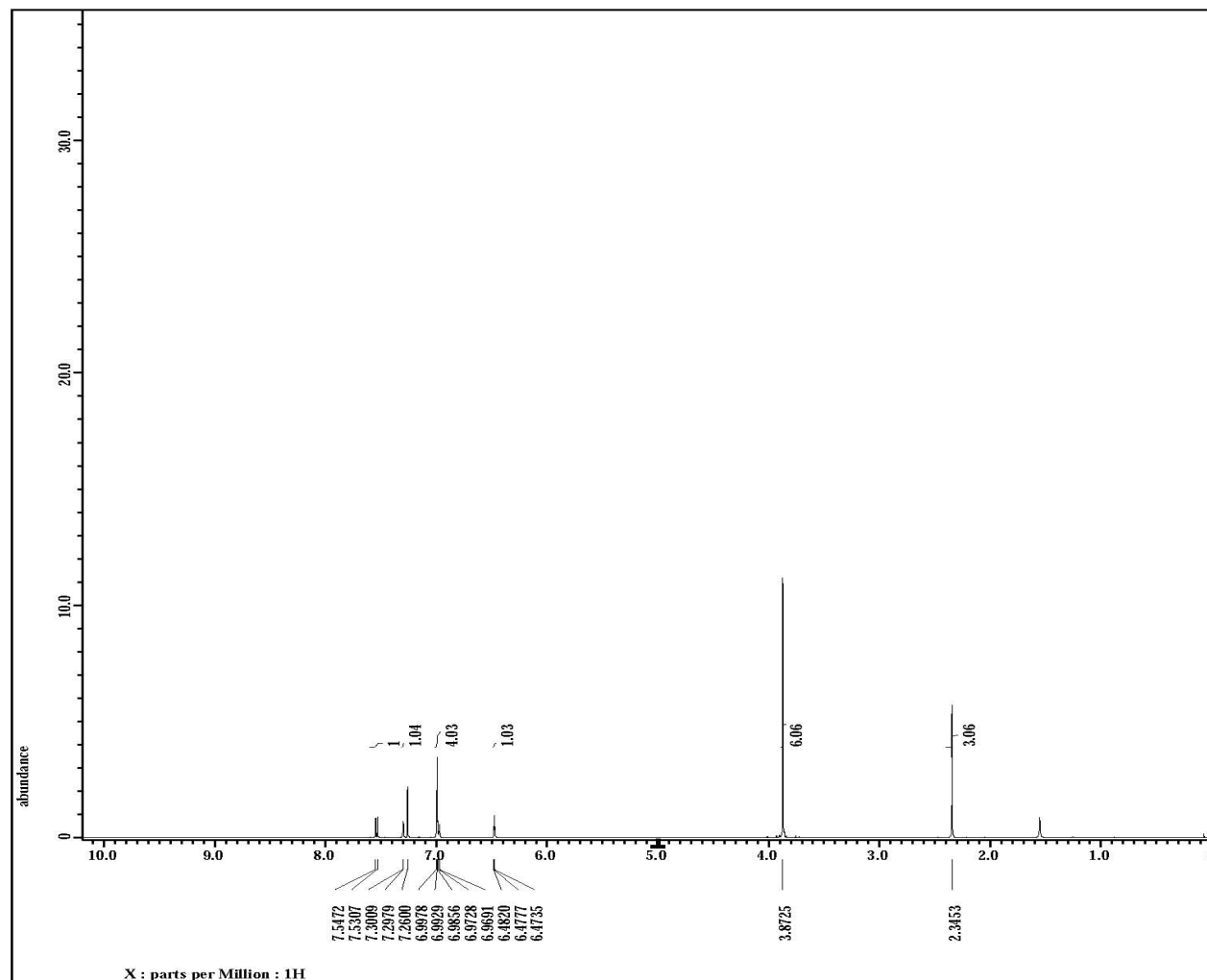
^{13}C NMR spectra of Compound 27



¹H NMR spectra of Compound **27a**



¹³C NMR spectra of Compound **27a**



```

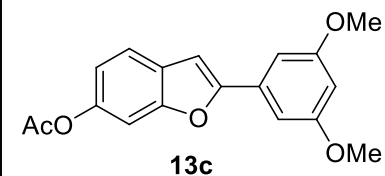
Filename      = NSH1257F2_1H-5.jdf
Author       = N.Ahmed
Experiment    = single_pulse.ex2
Sample_id    = S#427583
Solvent      = CHLOROFORM-D
Creation_time = 25-FEB-2012 11:10:53
Revision_time = 9-SEP-2016 22:37:31
Current_Time  = 9-SEP-2016 22:39:47

Data_format   = 1D COMPLEX
Dim_size      = 26214
Dim_title     = 1H
Dim_units     = [ppm]
Dimensions    = X
Site          = ECX 500
Spectrometer  = DELTA2_NMR

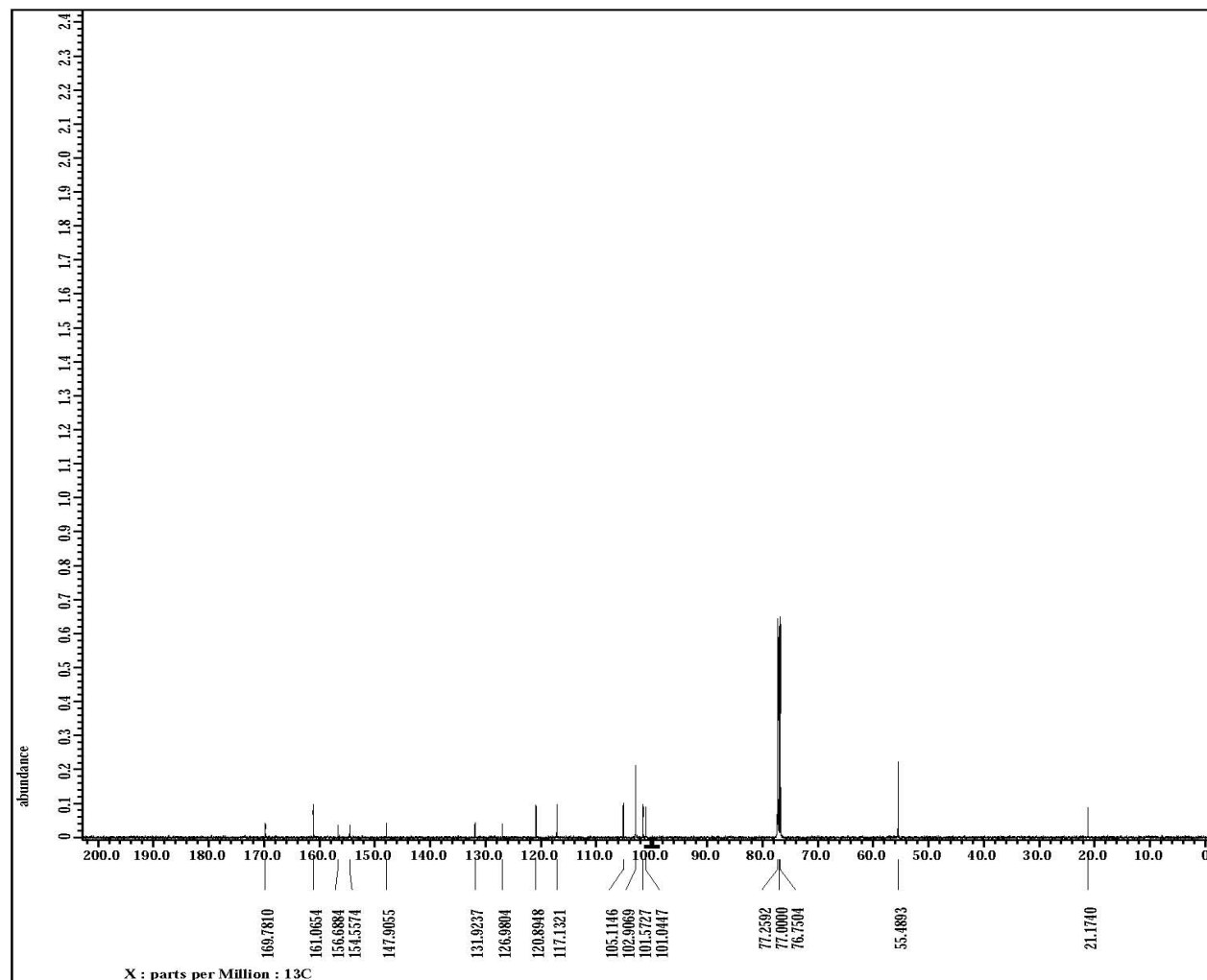
Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 3.27155712 [s]
X_domain       = 1H
X_freq         = 500.15991521 [MHz]
X_offset       = 5.0 [ppm]
X_points       = 32768
X_prescans     = 1
X_resolution   = 0.30566485 [Hz]
X_sweep        = 10.01602564 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Tri_domain     = 1H
Tri_freq       = 500.15991521 [MHz]
Tri_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 9
Total_scans    = 9

X_90_width     = 13.25 [us]
X_acq_time      = 3.27155712 [s]
X_angle         = 45 [deg]
X_atn           = 3.99 [dB]
X_pulse         = 6.625 [us]
Irr_mode        = Off
Tri_mode        = Off
Dante_preset    = FALSE
Initial_wait    = 1 [s]
Recvr_gain      = 54
Relaxation_delay = 1 [s]
Repetition_time = 4.27155712 [s]
Temp_get        = 20.1 [dC]

```



^1H NMR spectra of Compound **13c**



```

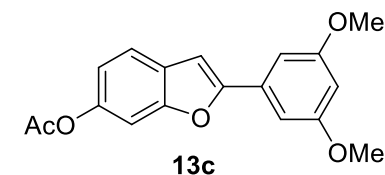
Filename      = NSH1257F2_13C-3.jdf
Author       = N.Ahmed
Experiment   = single_pulse_dec
Sample_id    = S#590257
Solvent      = CHLOROFORM-D
Creation_time = 27-FEB-2012 20:14:22
Revision_time = 9-SEP-2016 22:48:25
Current_Time = 9-SEP-2016 22:49:53

Data_format   = 1D COMPLEX
Dim_size      = 26214
Dim_title     = 13C
Dim_units     = [ppm]
Dimensions    = X
Site          = ECX 500
Spectrometer  = DELTA2_NMR

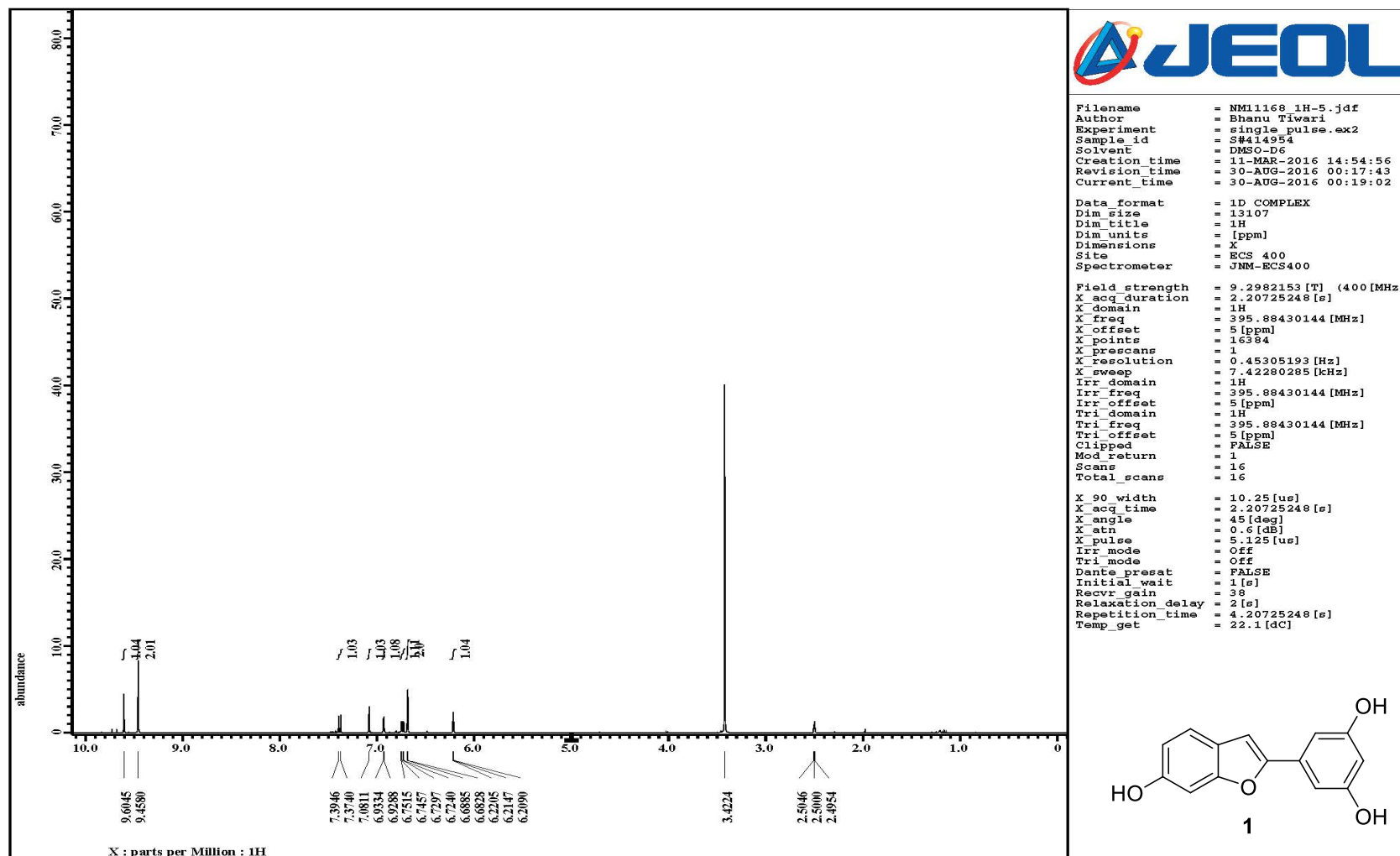
Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 0.82837504 [s]
X_domain      = 13C
X_freq        = 125.76529768 [MHz]
X_offset      = 100 [ppm]
X_points      = 32768
X_prescans    = 4
X_resolution  = 1.20718268 [Hz]
X_sweep       = 39.55696203 [kHz]
Irr_domain    = 1H
Irr_freq      = 500.15991521 [MHz]
Irr_offset    = 5.0 [ppm]
Clipped       = FALSE
Mod_return    = 1
Scans         = 1000
Total_scans   = 1000

X_90_width    = 9.62 [us]
X_acq_time    = 0.82837504 [s]
X_angle       = 30 [deg]
X_atn         = 7.1 [dB]
X_pulse       = 3.20666667 [us]
Irr_atn_dec   = 19.5 [dB]
Irr_atn_noe   = 21.5 [dB]
Irr_noise     = WALTZ
Decoupling    = TRUE
Initial_wait  = 1 [s]
Noe           = TRUE
Noe_time      = 1 [s]
Recvr_gain    = 58
Relaxation_delay = 1 [s]
Repetition_time = 1.82837504 [s]
Temp_get      = 19.1 [dC]

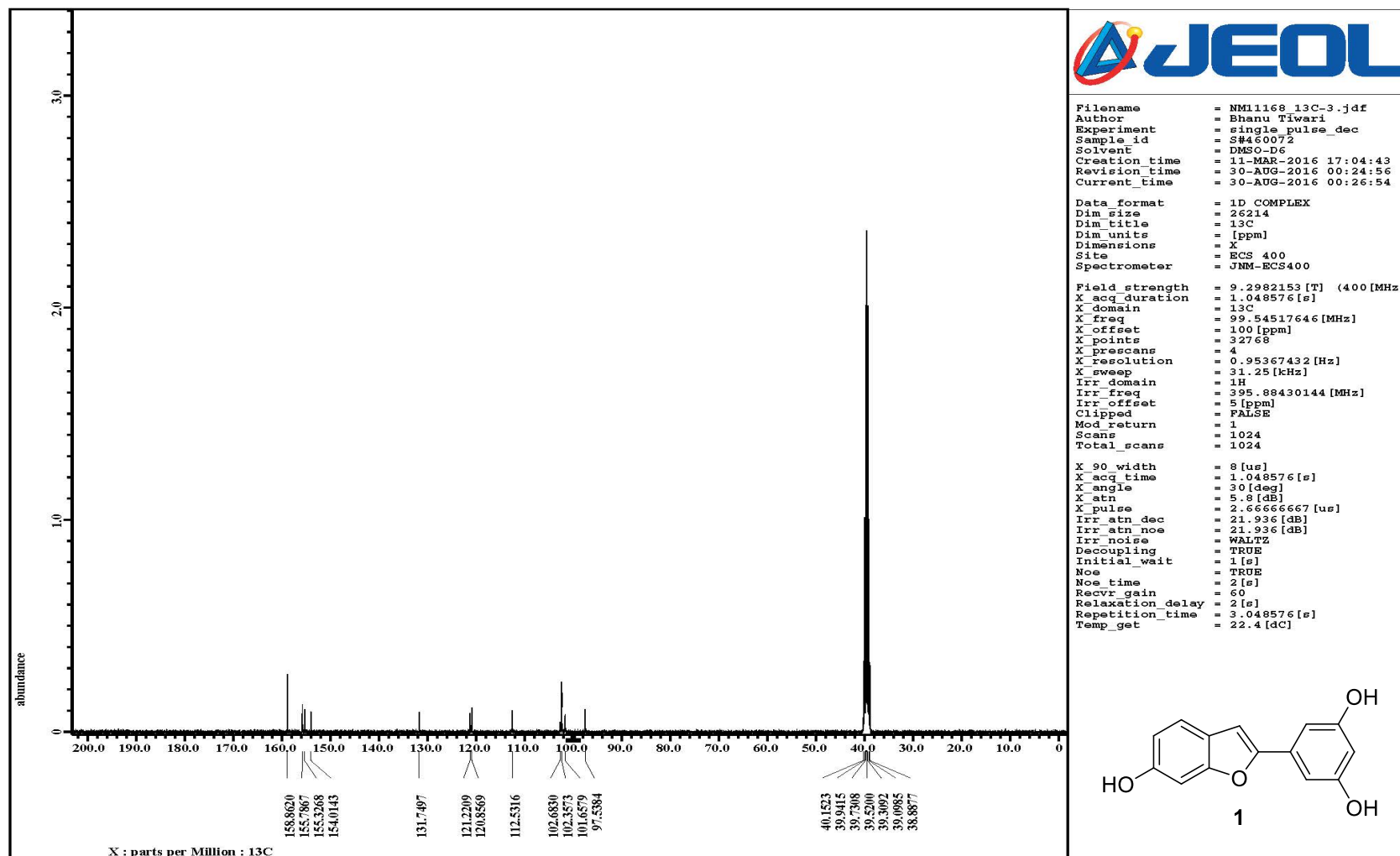
```



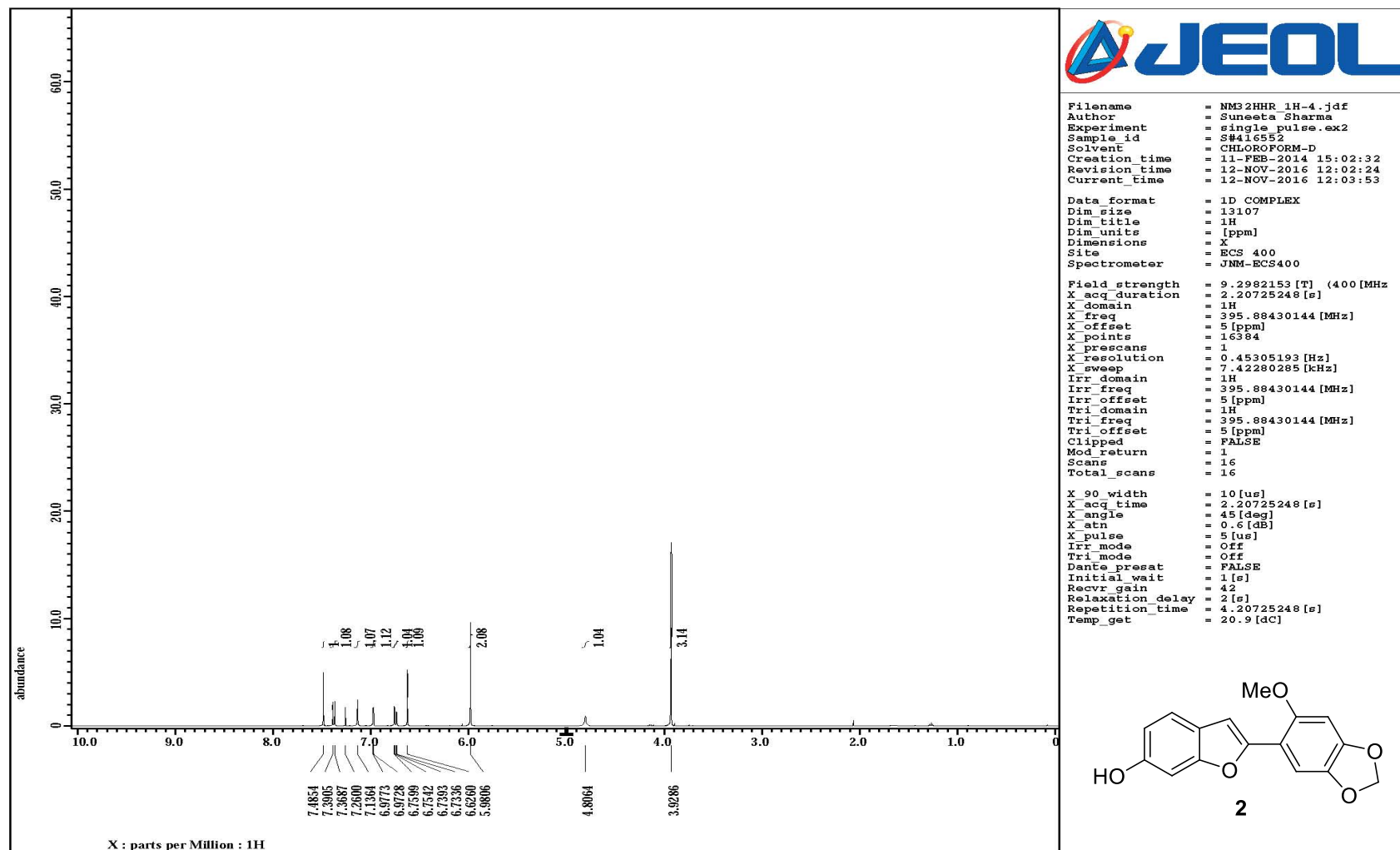
^{13}C NMR spectra of Compound **13c**



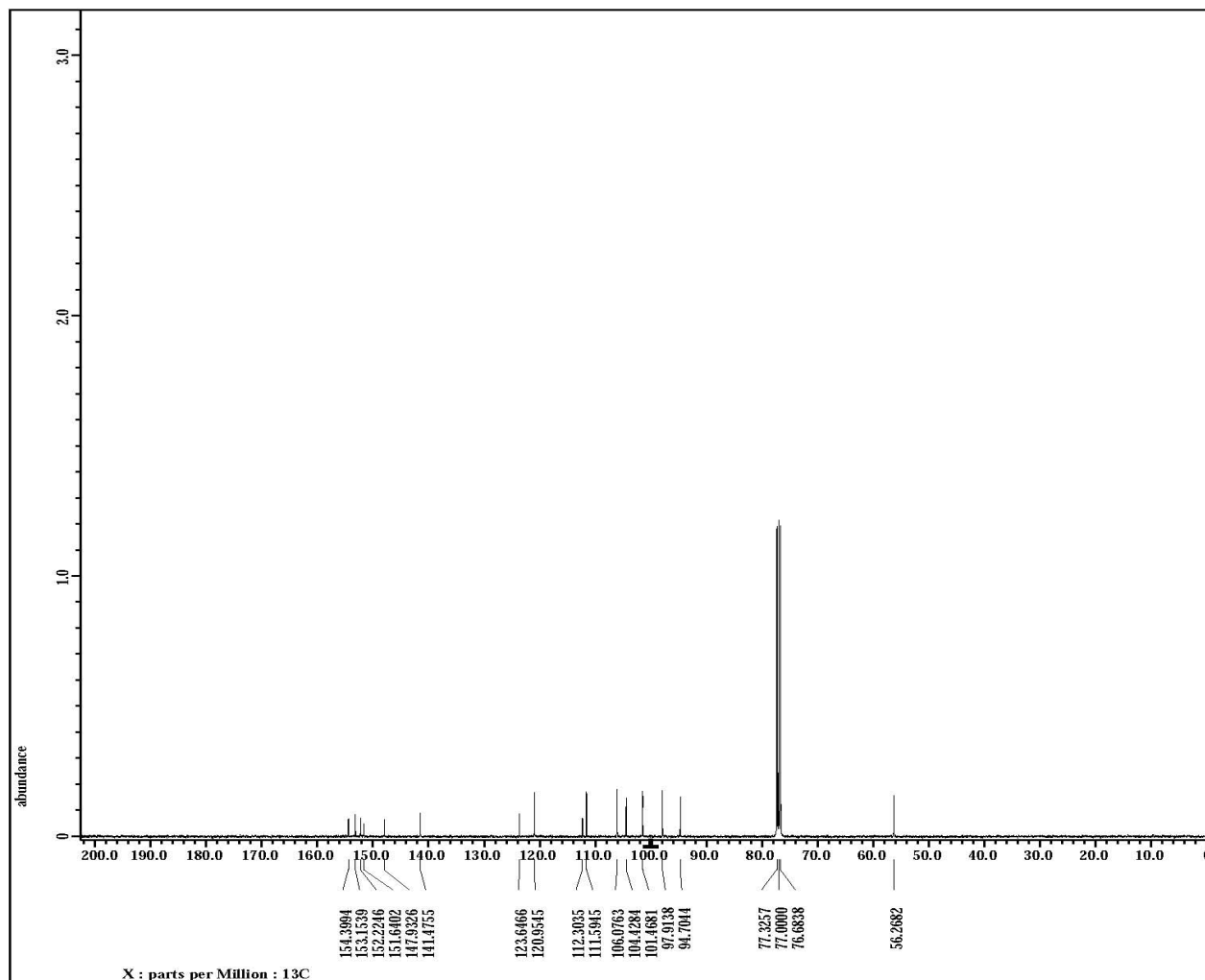
¹H NMR spectra of moracin M (1)



¹³C NMR spectra of moracin M (1)



¹H NMR spectra of cicerfuran (2)



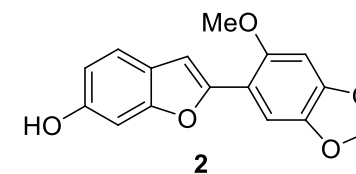
```

Filename      = NM32HHR_13C-3.jdf
Author       = Sunseta Sharma
Experiment    = single pulse_dec
Sample_id     = S#417587
Solvent       = CHLOROFORM-D
Creation_time = 11-FEB-2014 16:19:25
Revision_time = 12-NOV-2016 12:11:20
Current_time  = 12-NOV-2016 12:12:24

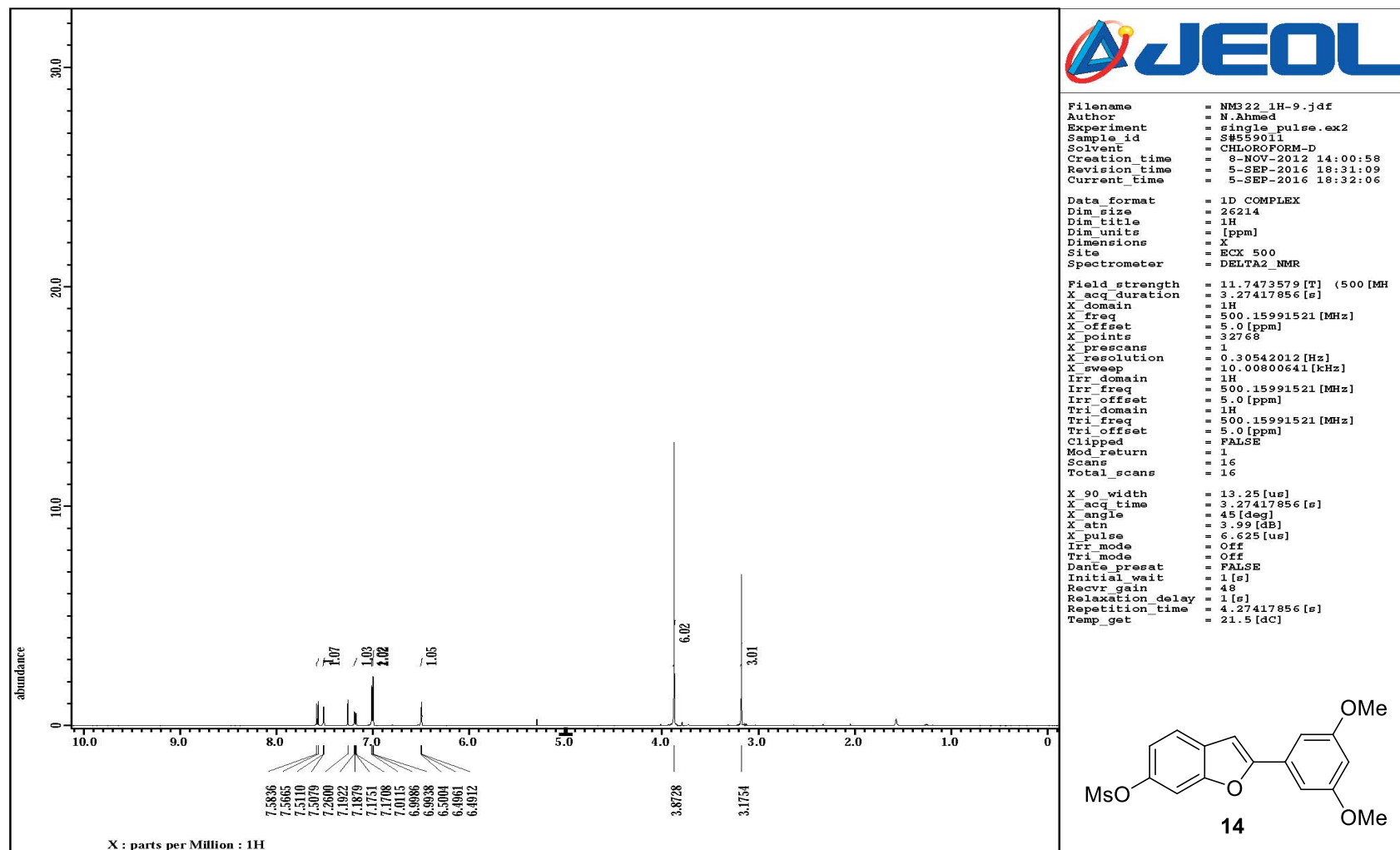
Data_format   = 1D COMPLEX
Dim_size      = 26214
Dim_title     = 13C
Dim_units     = [ppm]
Dimensions    = X
Site          = ECS 400
Spectrometer  = JNM-ECS400

Field_strength = 9.2982153 [T] (400 [MHz]
X_acq_duration = 1.048576 [s]
X_domain       = 13C
X_freq         = 99.54517646 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 0.95367432 [Hz]
X_sweep        = 31.25 [kHz]
Irr_domain     = 1H
Irr_freq       = 395.88430144 [MHz]
Irr_offset     = 5 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 1500
Total_scans    = 1500

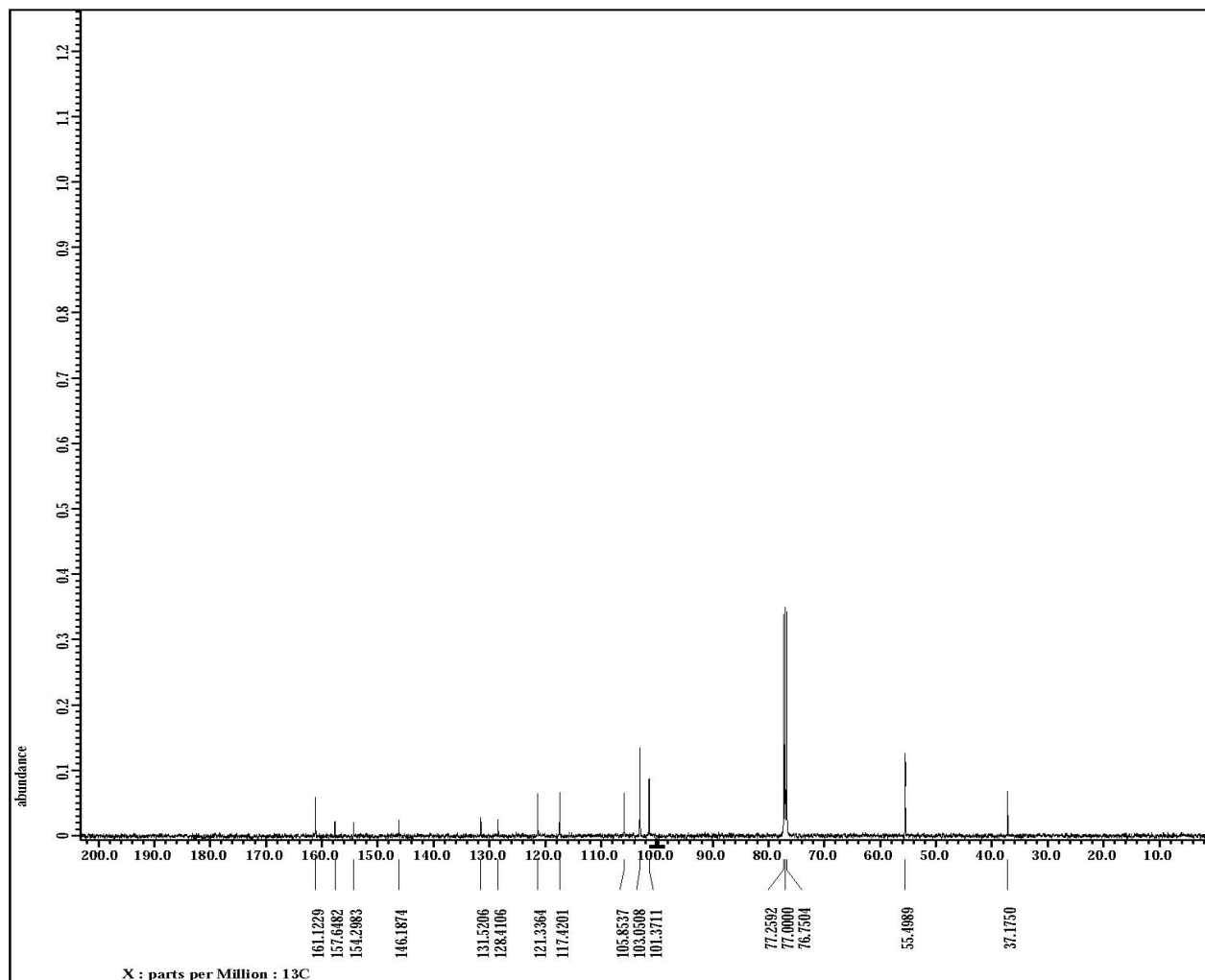
X_90_width     = 8 [us]
X_acq_time     = 1.048576 [s]
X_angle        = 30 [deg]
X_atn          = 5.8 [dB]
X_pulse        = 2.66666667 [us]
Irr_atn_dec    = 21.936 [dB]
Irr_atn_noe    = 21.936 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 2 [s]
Recvr_gain     = 60
Relaxation_delay = 2 [s]
Repetition_time = 3.048576 [s]
Temp_get       = 21.2 [dC]
  
```



^{13}C NMR spectra of cicerfuran (2)



¹H NMR spectra of compound **14**



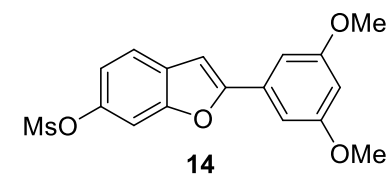
```

Filename      = NM322_13C-8.jdf
Author       = N.Ahmed
Experiment    = single_pulse_dec
Sample_id    = S#568044
Solvent      = CHLOROFORM-D
Creation_time = 8-NOV-2012 14:55:42
Revision_time = 5-SEP-2016 18:34:55
Current_time  = 5-SEP-2016 18:35:04

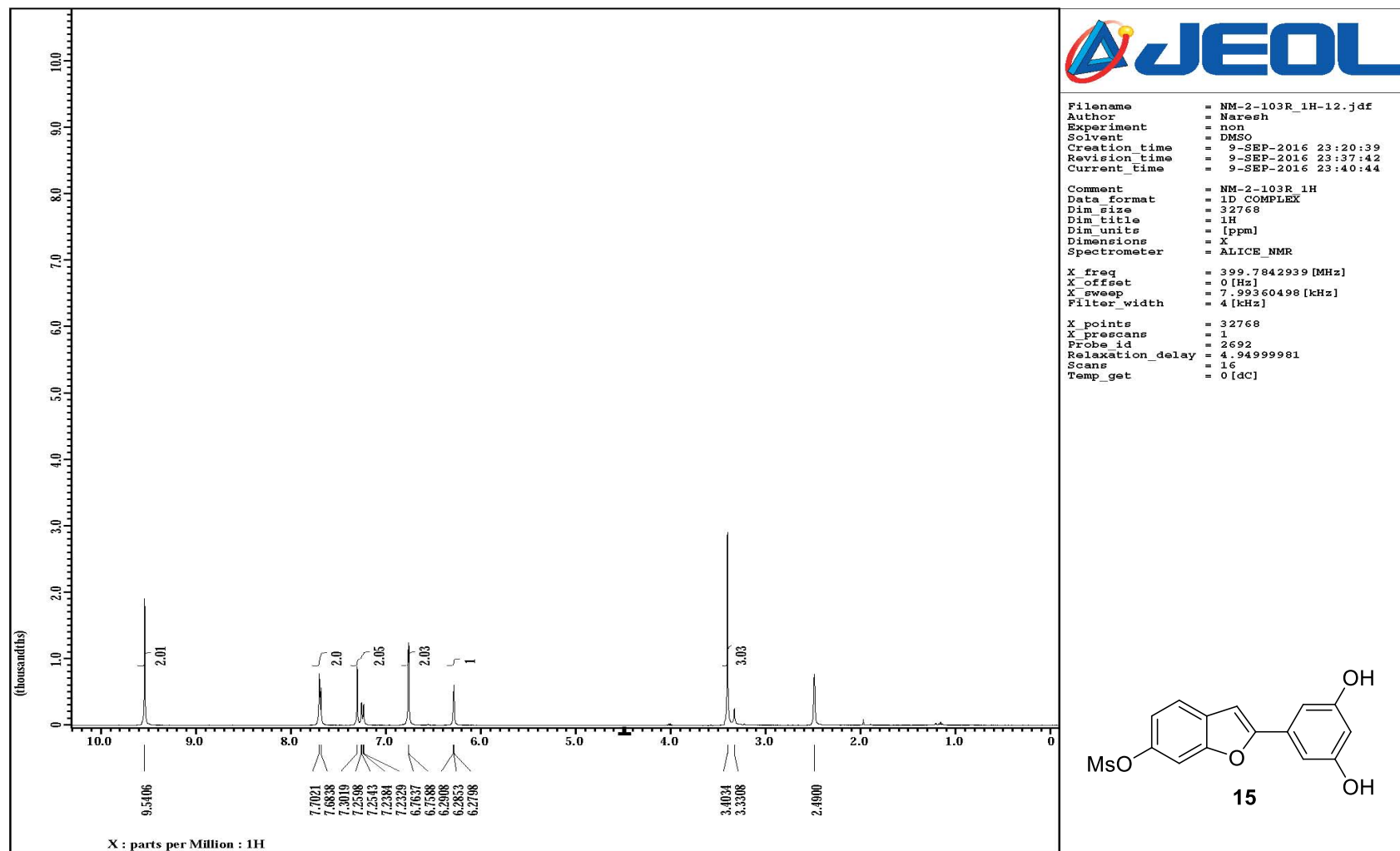
Data_format   = 1D_COMPLEX
Dim_size      = 26214
Dim_title     = 13C
Dim_units     = [ppm]
Dimensions    = X
Site          = ECX 500
Spectrometer  = DELTA2_NMR

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 0.82837504 [s]
X_domain      = 13C
X_freq        = 125.76529768 [MHz]
X_offset      = 100 [ppm]
X_points      = 32768
X_prescans    = 4
X_resolution  = 1.20718268 [Hz]
X_sweep       = 39.55696203 [kHz]
Irr_domain    = 1H
Irr_freq      = 500.15991521 [MHz]
Irr_offset    = 5.0 [ppm]
Clipped       = FALSE
Mod_return    = 1
Scans         = 649
Total_scans   = 649

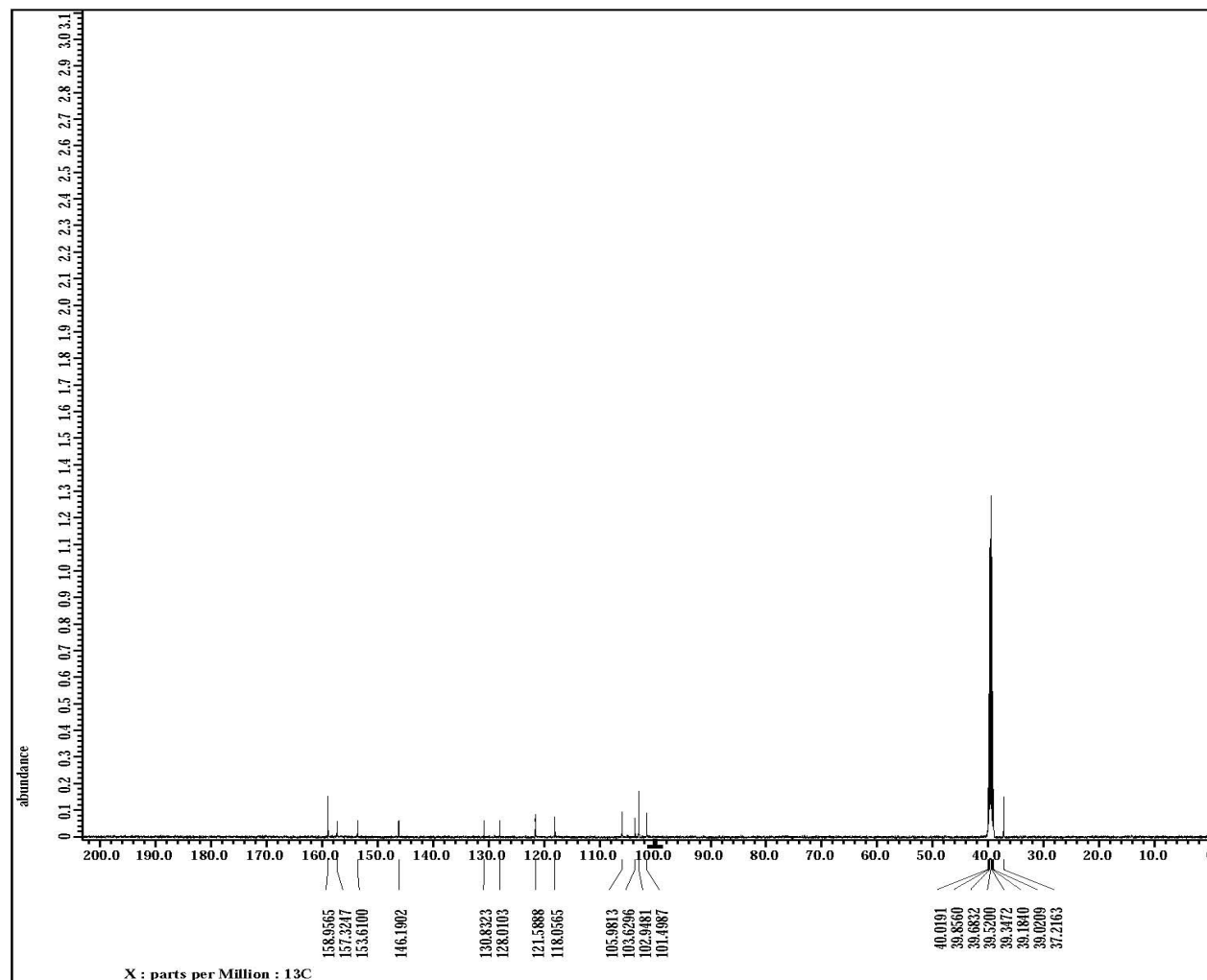
X_90_width    = 9.62 [us]
X_acq_time    = 0.82837504 [s]
X_angle       = 30 [deg]
X_atn         = 7.1 [dB]
X_pulse       = 3.20666667 [us]
Irr_atn_dec   = 19.5 [dB]
Irr_atn_noe   = 21.5 [dB]
Irr_noise     = WALTZ
Decoupling    = TRUE
Initial_wait  = 1 [s]
Noe           = TRUE
Noe_time      = 0.6 [s]
Recvr_gain    = 58
Relaxation_delay = 0.6 [s]
Repetition_time = 1.42837504 [s]
Temp_get      = 20.9 [dC]
  
```



¹³C NMR spectra of Compound 14



^1H NMR spectra of Compound 15



```

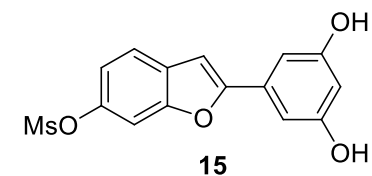
Filename      = NM2103R_13C-3.jdf
Author       = N.Ahmed
Experiment    = single_pulse_dec
Sample_id    = S#563783
Solvent      = DMSO-D6
Creation_time = 19-JUN-2012 18:59:34
Revision_time = 10-SEP-2016 00:32:05
Current_time  = 10-SEP-2016 00:33:38

Data_format   = 1D_COMPLEX
Dim_size      = 26214
Dim_title     = 13C
Dim_units     = [ppm]
Dimensions    = X
Site          = ECX 500
Spectrometer  = DELTA2_NMR

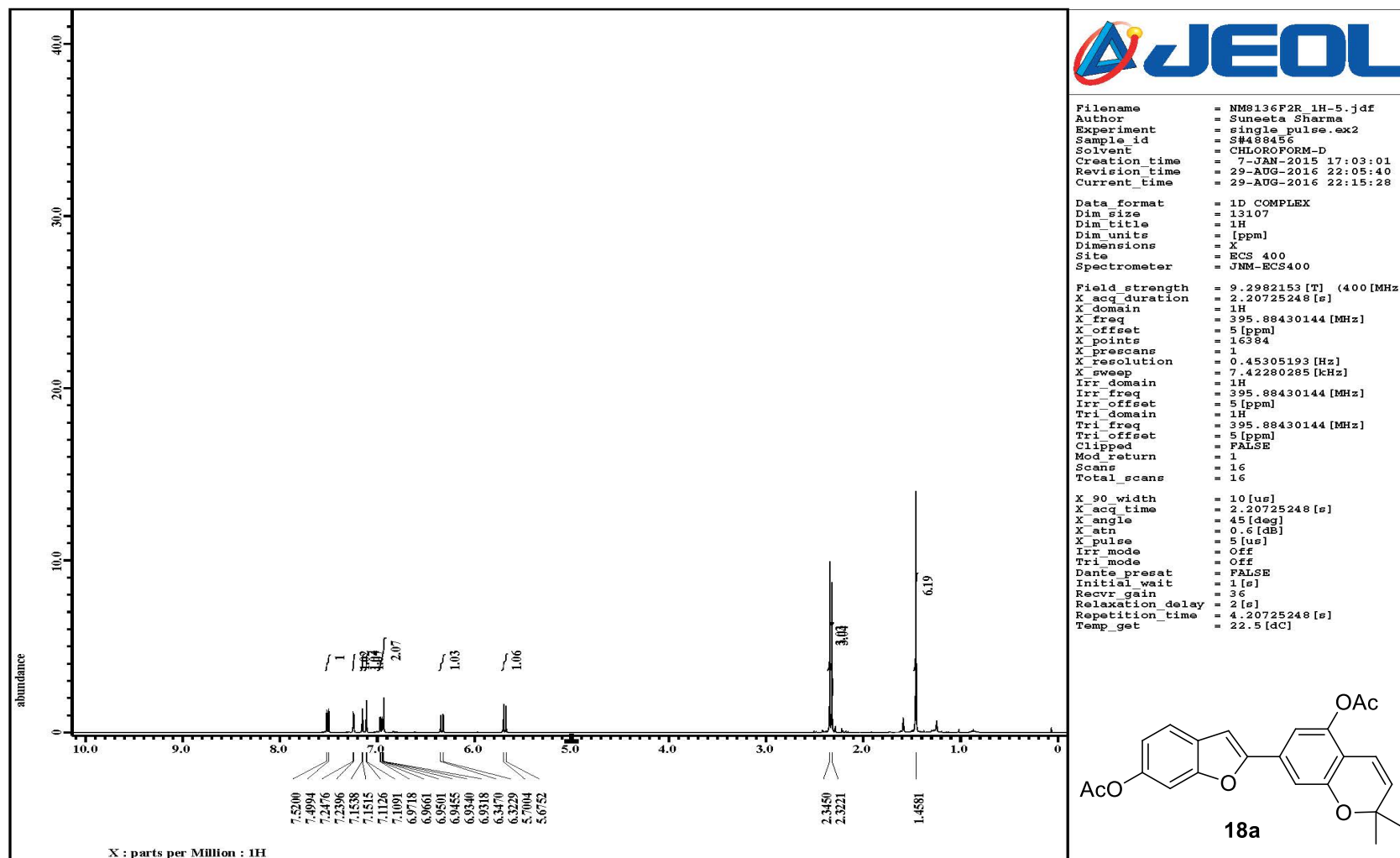
Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 0.82837504 [s]
X_domain       = 13C
X_freq         = 125.76529768 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.20718268 [Hz]
X_sweep        = 39.55696203 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 1000
Total_scans    = 1000

X_90_width     = 9.62 [us]
X_acq_time     = 0.82837504 [s]
X_angle        = 30 [deg]
X_atn          = 7.1 [dB]
X_pulse        = 3.20666667 [us]
Irr_atn_dec    = 19.5 [dB]
Irr_atn_noe    = 21.5 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 1 [s]
Recvr_gain     = 58
Relaxation_delay = 1 [s]
Repetition_time = 1.82837504 [s]
Temp_get       = 21 [dC]

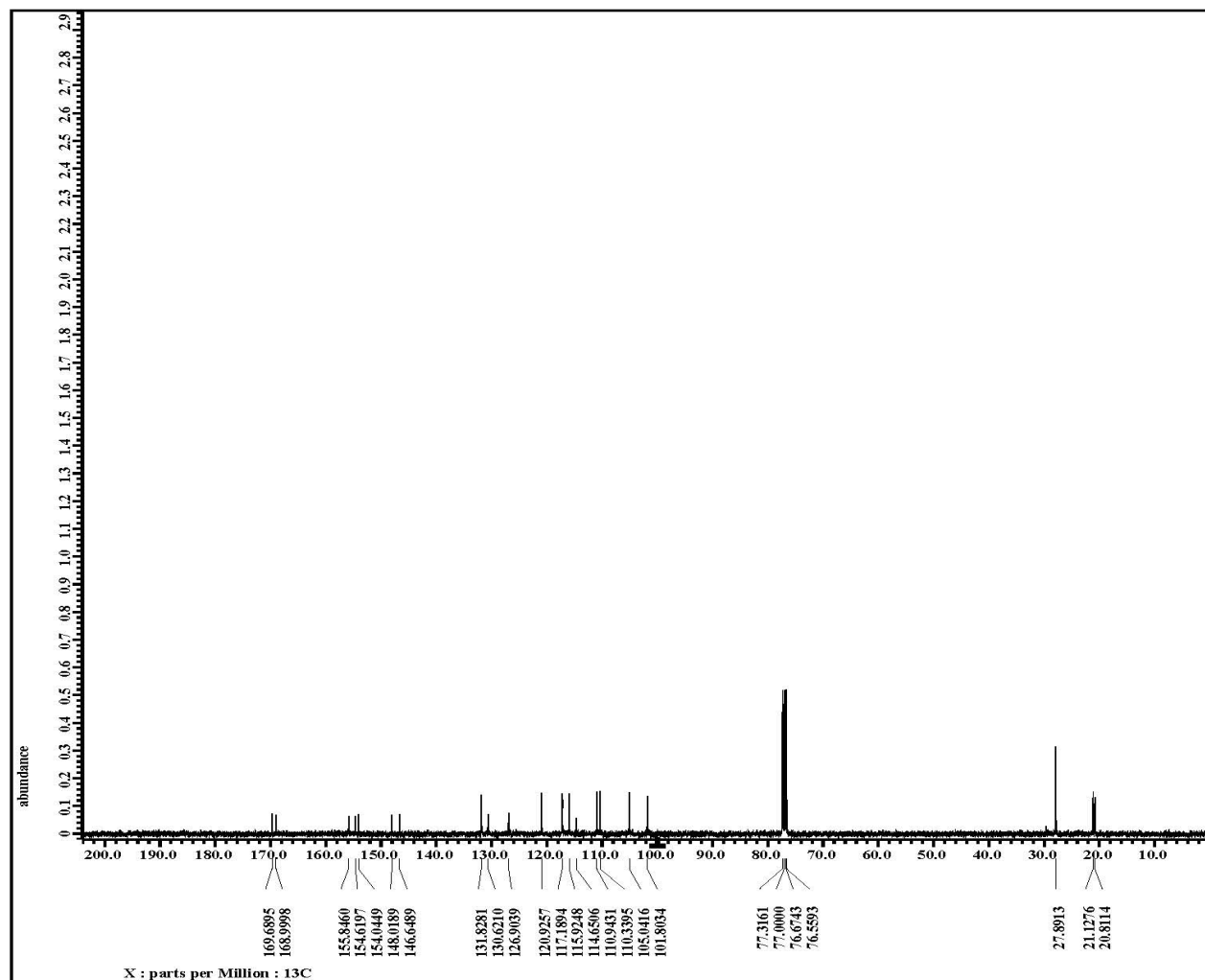
```



^{13}C NMR spectra of Compound 15



¹H NMR spectra of Compound **18a**



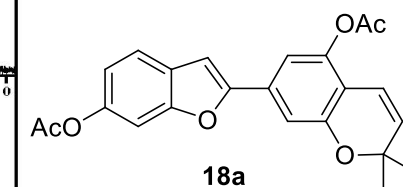
```

Filename      = NM8136F2R_13C-3.jdf
Author       = Suneeta Sharma
Experiment   = single pulse_dec
Sample_id    = S#582410
Solvent      = CHLOROFORM-D
Creation_time = 7-JAN-2015 19:56:51
Revision_time = 29-AUG-2016 22:19:46
Current_time  = 29-AUG-2016 22:21:30

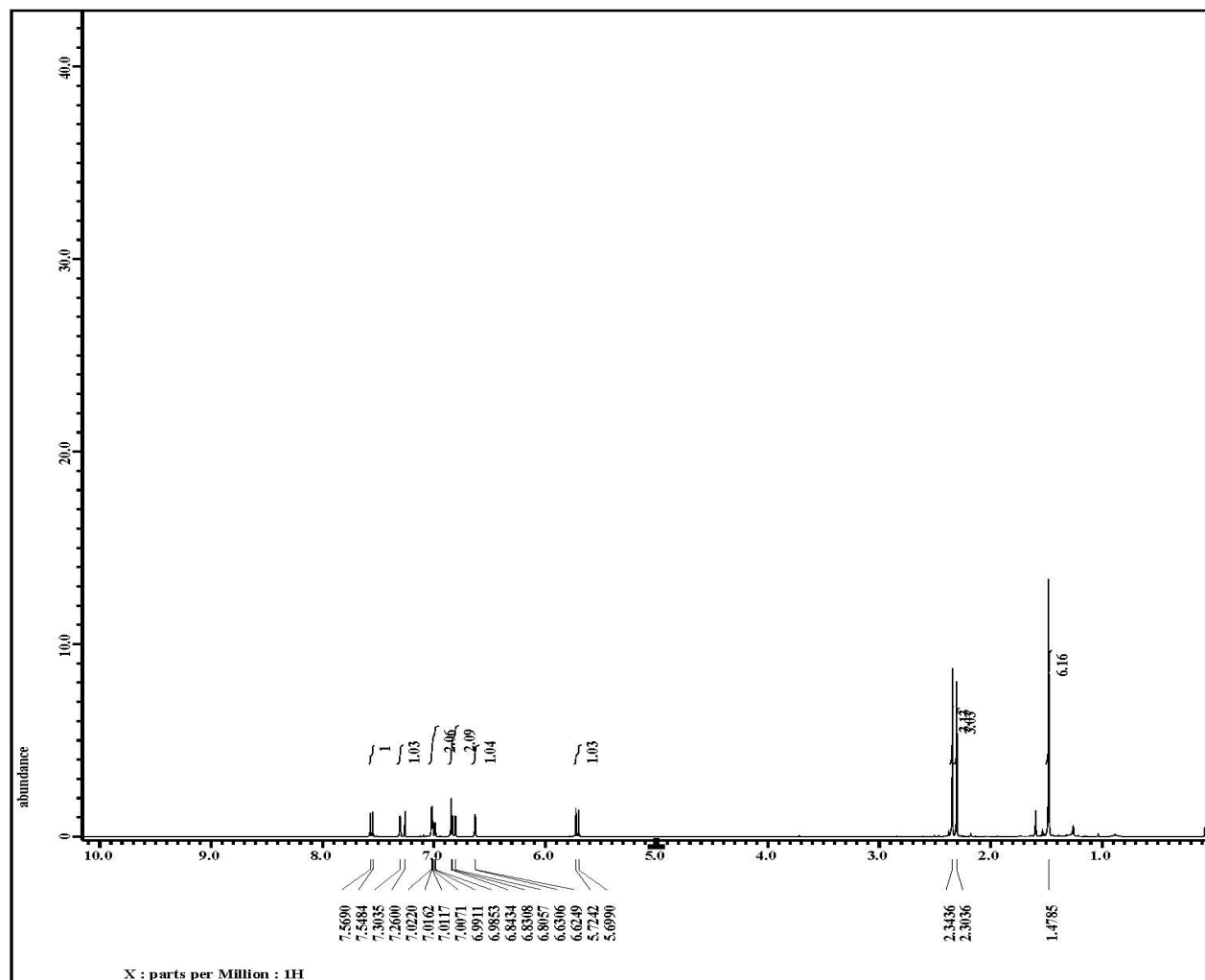
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECS 400
Spectrometer = JNM-ECS400

Field_strength = 9.2982153 [T] (400 [MHz]
X_acq_duration = 1.048576 [s]
X_domain       = 13C
X_freq         = 99.54517646 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 0.95367432 [Hz]
X_sweep        = 31.25 [kHz]
Irr_domain     = 1H
Irr_freq       = 395.88430144 [MHz]
Irr_offset     = 5 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 510.0
Total_scans    = 510.0

X_90_width     = 8 [us]
X_acq_time     = 1.048576 [s]
X_angle        = 30 [deg]
X_atn          = 5.8 [dB]
X_pulse        = 2.66666667 [us]
Irr_atn_dec    = 21.936 [dB]
Irr_atn_noe    = 21.936 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 1 [s]
Recvr_gain     = 60
Relaxation_delay = 1 [s]
Repetition_time = 2.048576 [s]
Temp_get       = 23.3 [dC]
  
```



^{13}C NMR spectra of Compound **18a**

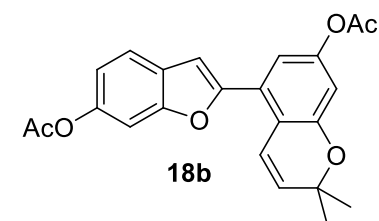


Filename = NM8136F1R_1H-5.jdf
 Author = Suneeta Sharma
 Experiment = single pulse.ex2
 Sample id = S#492838
 Solvent = CHLOROFORM-D
 Creation time = 7-JAN-2015 17:10:25
 Revision time = 29-AUG-2016 21:25:11
 Current time = 29-AUG-2016 21:38:44

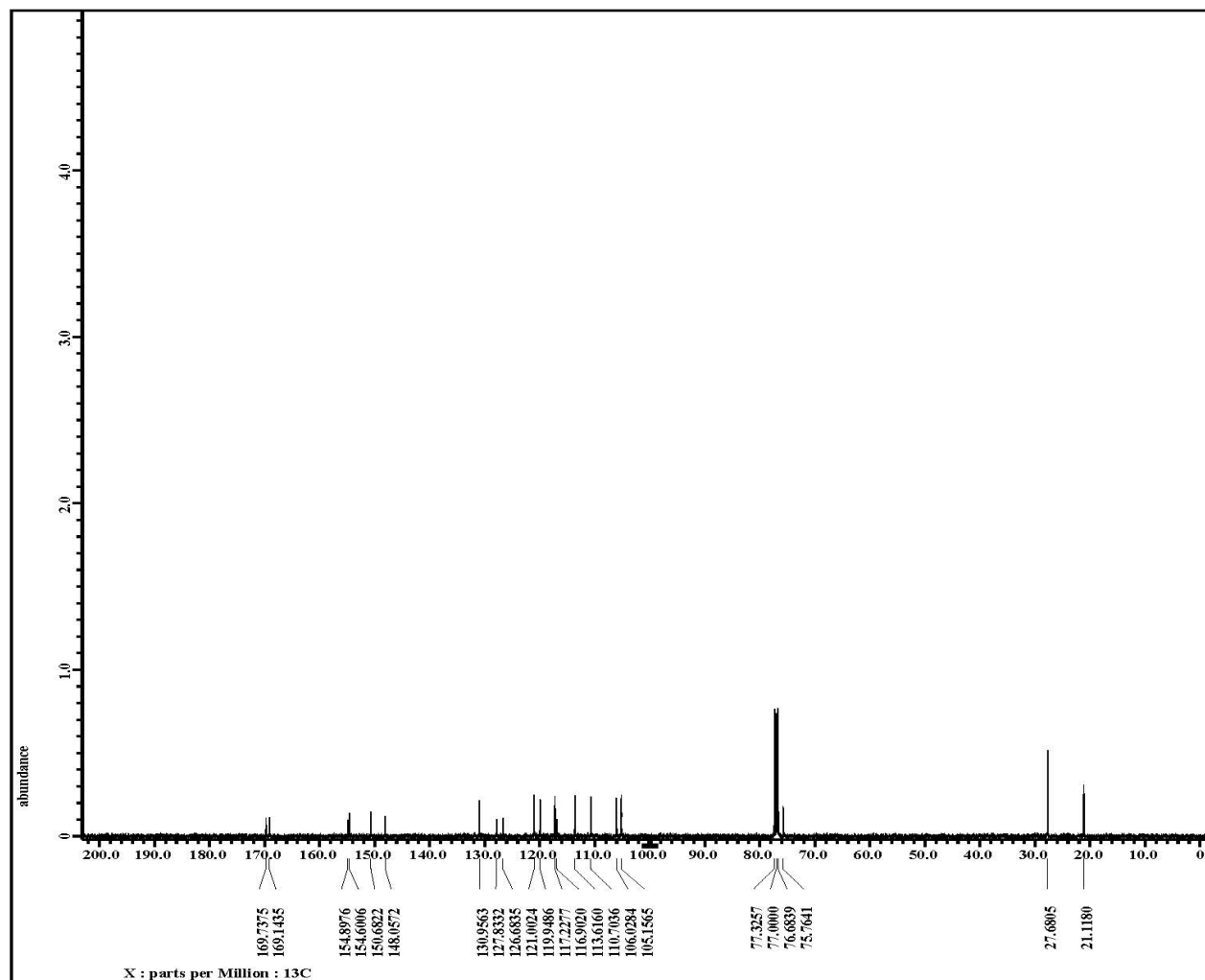
 Data format = 1D COMPLEX
 Dim size = 13107
 Dim title = 1H
 Dim units = [ppm]
 Dimensions = X
 Site = ECS 400
 Spectrometer = JNM-ECS400

 Field strength = 9.2982153 [T] (400 [MHz]
 X acq duration = 2.20725248 [s]
 X domain = 1H
 X freq = 395.88430144 [MHz]
 X offset = 5 [ppm]
 X points = 16384
 X prescans = 1
 X resolution = 0.45305193 [Hz]
 X sweep = 7.42280285 [kHz]
 Irr domain = 1H
 Irr freq = 395.88430144 [MHz]
 Irr offset = 5 [ppm]
 Tri domain = 1H
 Tri freq = 395.88430144 [MHz]
 Tri offset = 5 [ppm]
 Clipped = FALSE
 Mod return = 1
 Scans = 16
 Total scans = 16

 X 90 width = 10 [us]
 X acq time = 2.20725248 [s]
 X angle = 45 [deg]
 X atn = 0.6 [dB]
 X pulse = 5 [us]
 Irr mode = Off
 Tri mode = Off
 Dante preset = FALSE
 Initial wait = 1 [s]
 Recvr gain = 38
 Relaxation delay = 2 [s]
 Repetition time = 4.20725248 [s]
 Temp get = 22.5 [dc]



^1H NMR spectra of Compound **18b**



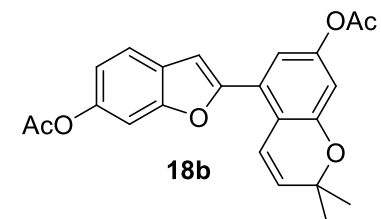
```

Filename      = NMS136F1 13C-3.jdf
Author       = Suneeta Sharma
Experiment   = single pulse_dec
Sample_id    = S#408085
Solvent      = CHLOROFORM-D
Creation_time = 7-JAN-2015 15:00:22
Revision_time = 29-AUG-2016 21:46:52
Current_time  = 29-AUG-2016 21:53:50

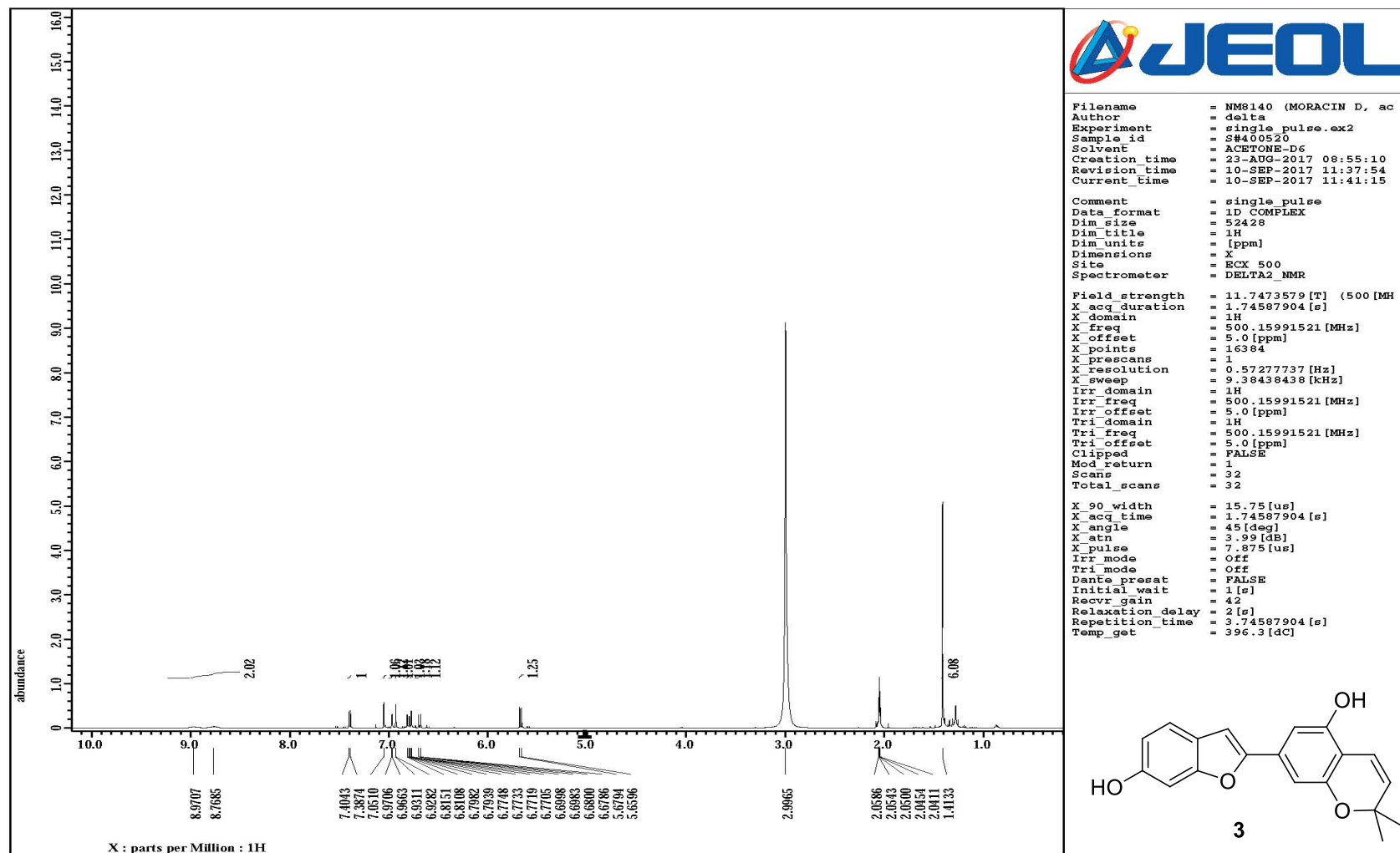
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECS 400
Spectrometer = JNM-ECS400

Field_strength = 9.2982153 [T] (400 [MHz]
X_acq_duration = 1.048576 [s]
X_domain       = 13C
X_freq         = 99.54517646 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 0.95367432 [Hz]
X_sweep        = 31.25 [kHz]
Irr_domain     = 1H
Irr_freq       = 395.88430144 [MHz]
Irr_offset     = 5 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 405
Total_scans    = 405

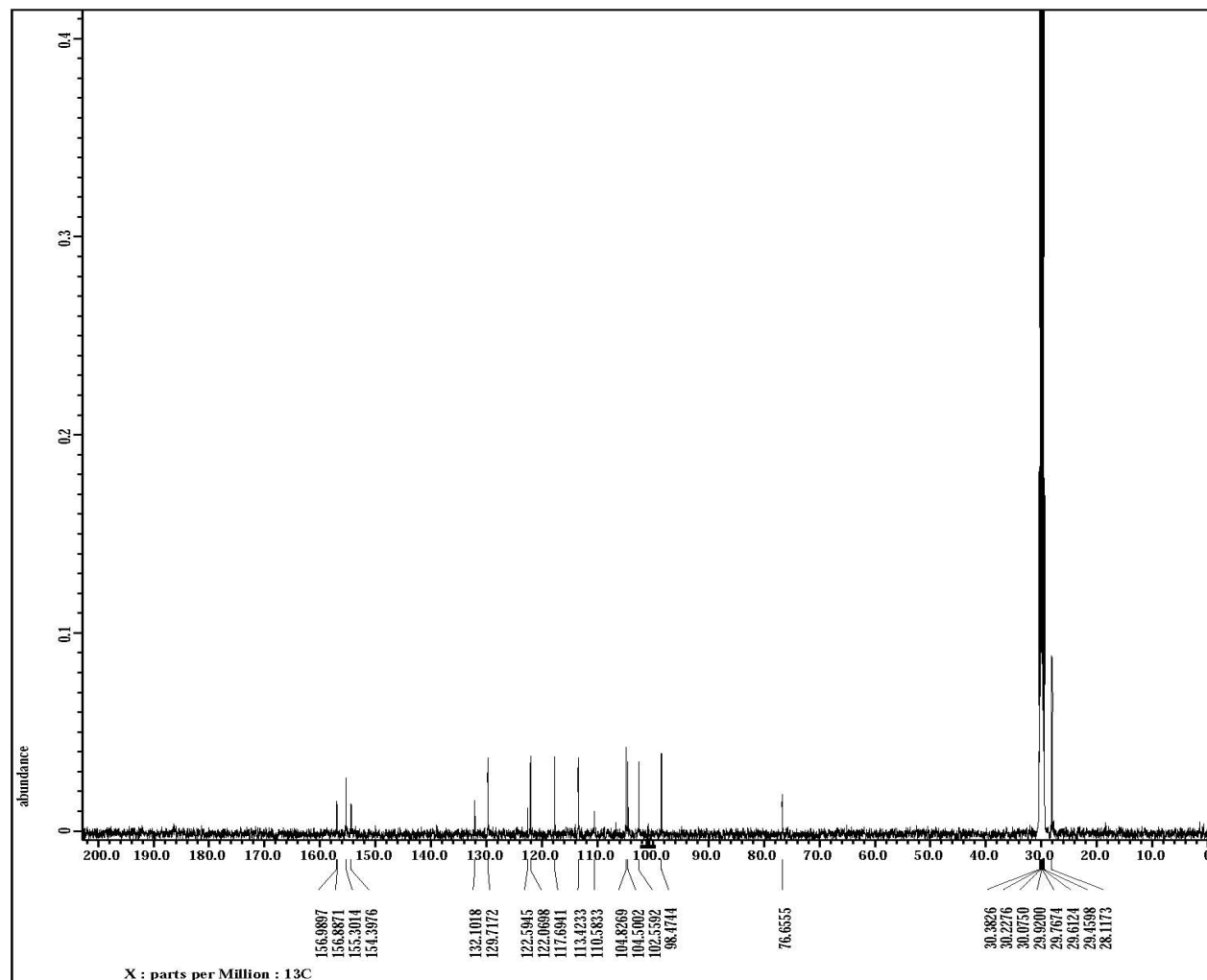
X_90_width    = 8 [us]
X_acq_time     = 1.048576 [s]
X_angle        = 30 [deg]
X_atn          = 5.8 [dB]
X_pulse        = 2.66666667 [us]
Irr_atn_dec    = 21.936 [dB]
Irr_atn_noe    = 21.936 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 1 [s]
Recvr_gain     = 60
Relaxation_delay = 1 [s]
Repetition_time = 2.048576 [s]
Temp_get       = 22 [dC]
  
```



^{13}C NMR spectra of Compound **18b**



^1H NMR spectra of moracin D (**3**)



```

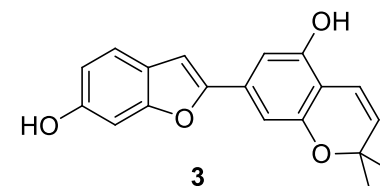
Filename      = NMMORACINDF2-13C-7.jd
Author        = delta
Experiment    = single pulse_dec
Sample_id     = S#462828
Solvent       = ACETONE-D6
Creation_time  = 23-AUG-2017 11:37:52
Revision_time = 10-SEP-2017 11:52:56
Current_time  = 10-SEP-2017 11:57:47

Comment       = single pulse decouple
Data_format   = 1D COMPLEX
Dim_size      = 104856
Dim_title     = 13C
Dim_units     = [ppm]
Dimensions    = X
Site          = ECX 500
Spectrometer  = DELTA2_NMR

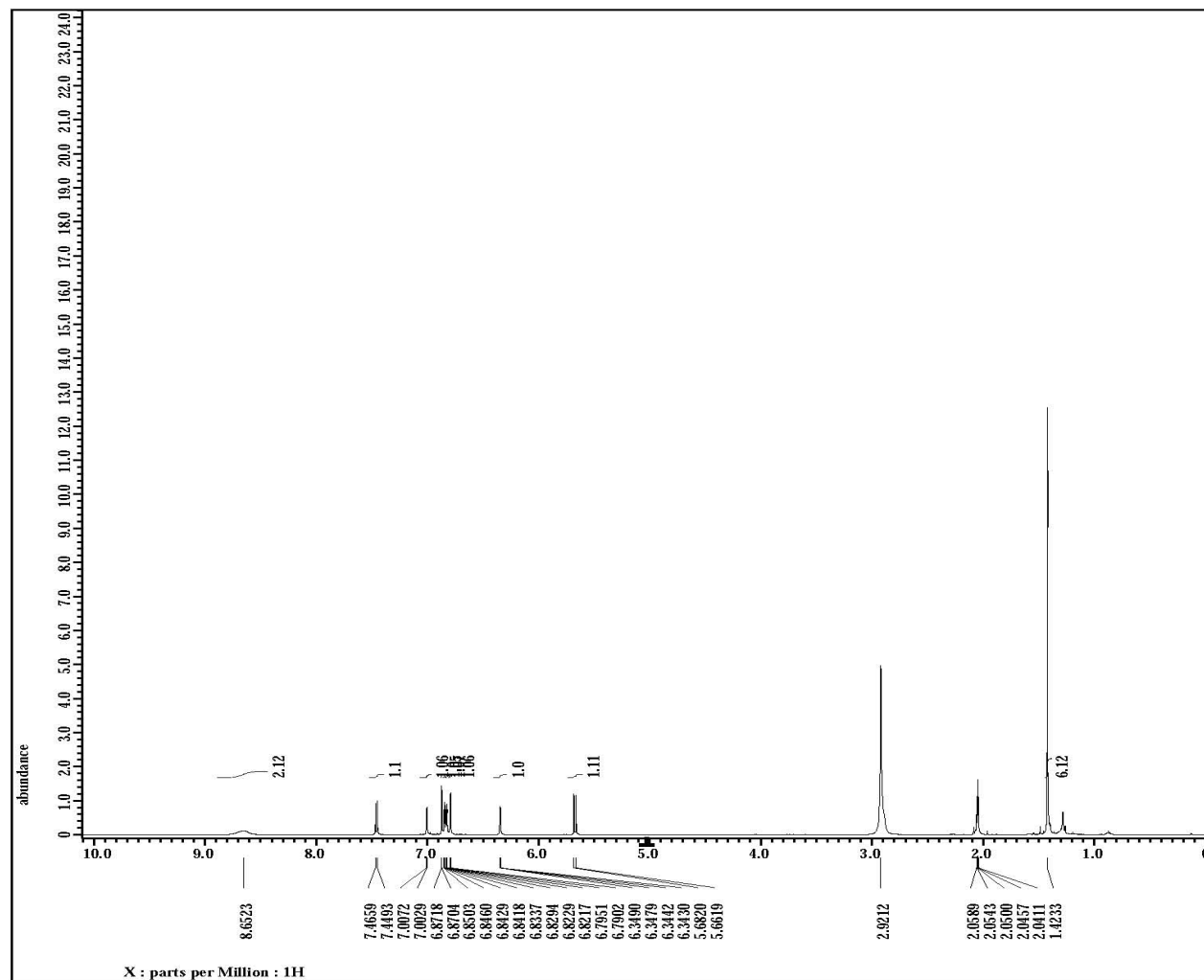
Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 0.83361792 [s]
X_domain       = 13C
X_freq         = 125.76529768 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034 [Hz]
X_sweep        = 39.3081761 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 2000
Total_scans    = 2000

X_90_width     = 11.75 [us]
X_acq_time     = 0.83361792 [s]
X_angle        = 30 [deg]
X_atn          = 7.1 [dB]
X_pulse        = 3.91666667 [us]
Irr_atn_dec    = 19.32 [dB]
Irr_atn_noe    = 19.32 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 1 [s]
Recvr_gain     = 60
Relaxation_delay = 1 [s]
Repetition_time = 1.83361792 [s]
Temp_get       = 396.4 [dC]

```



^{13}C NMR spectra of moracin D (3)



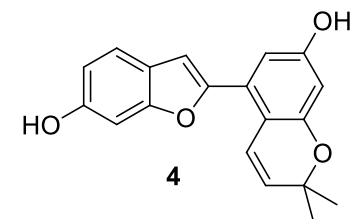
```

Filename      = NMS141 (MOARACIN E, A
Author        = delta
Experiment    = single pulse.ex2
Sample id     = S#537881
Solvent       = ACETONE-D6
Creation time  = 30-AUG-2017 12:43:51
Revision time  = 10-SEP-2017 12:32:21
Current Time  = 10-SEP-2017 12:34:19

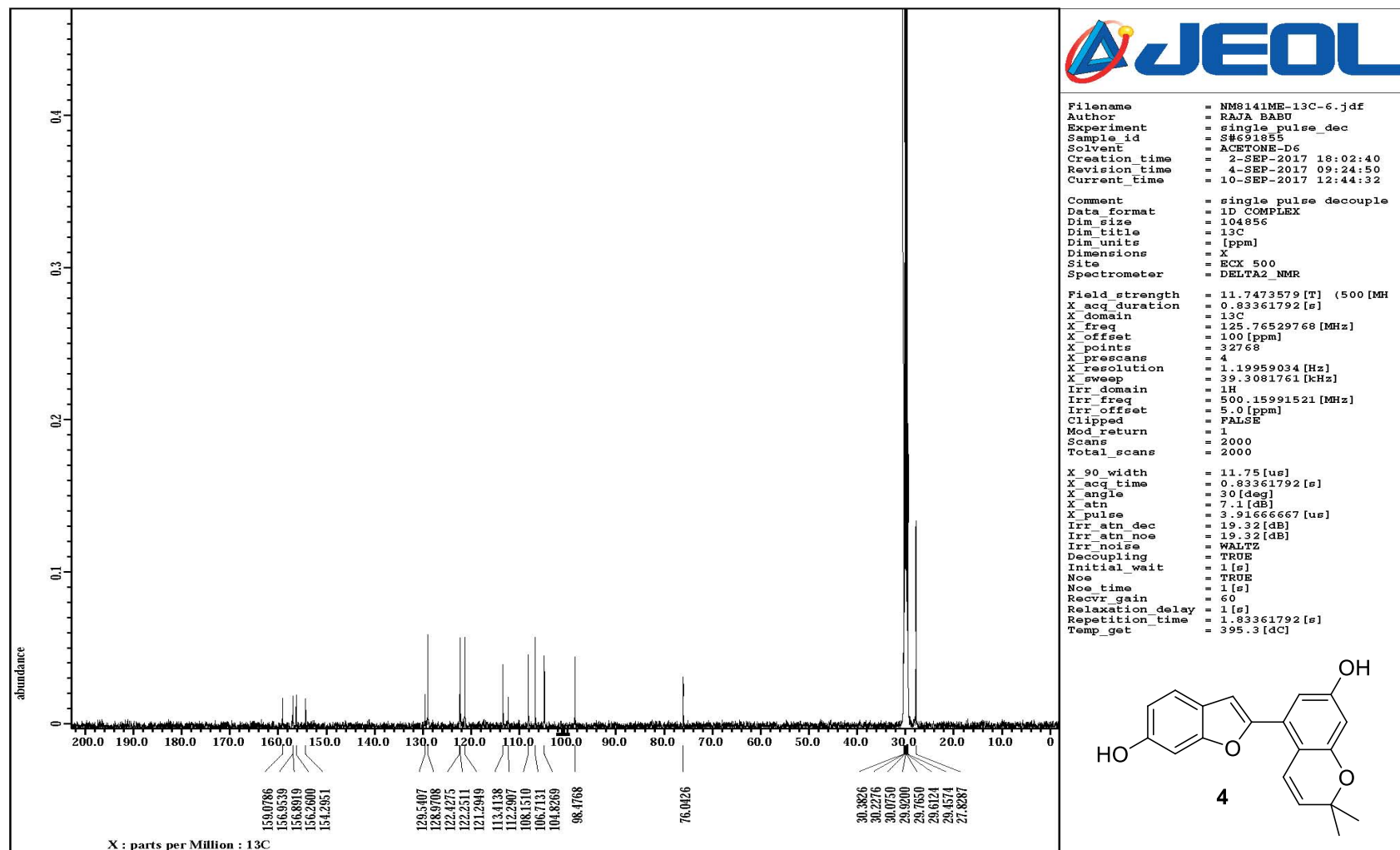
Comment       = single pulse
Data format   = 1D COMPLEX
Dim size      = 52428
Dim title     = 1H
Dim units     = [ppm]
Dimensions    = X
Site          = ECX 500
Spectrometer  = DELTA2_NMR

Field strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain       = 1H
X_freq         = 500.15991521 [MHz]
X_offset       = 5.0 [ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.57277737 [Hz]
X_sweep        = 9.38438438 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Tri_domain     = 1H
Tri_freq       = 500.15991521 [MHz]
Tri_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 32
Total_scans    = 32

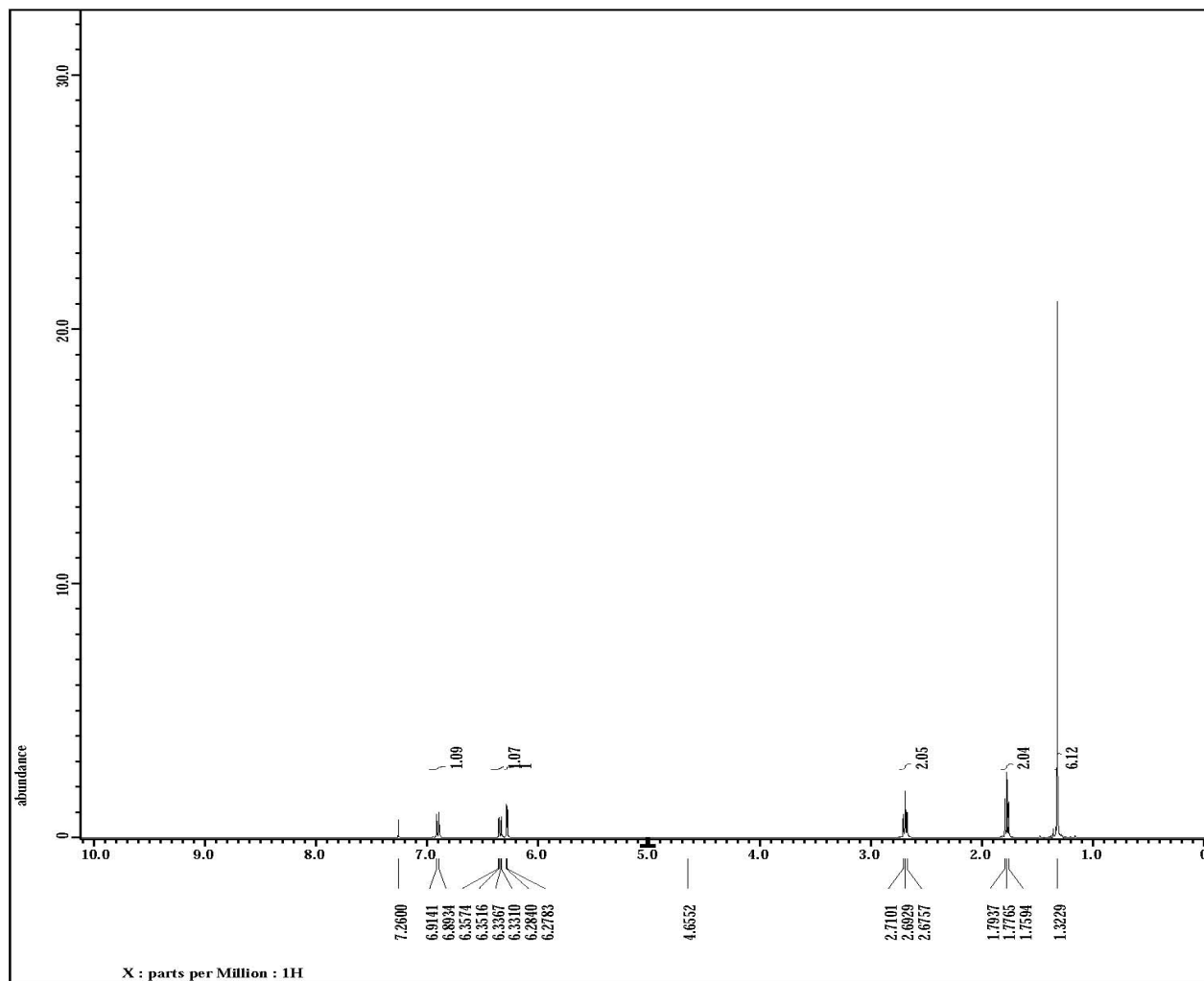
X_90_width     = 15.75 [us]
X_acq_time     = 1.74587904 [s]
X_angle        = 45 [deg]
X_atn          = 3.99 [dB]
X_pulse        = 7.875 [us]
Irr_mode       = Off
Tri_mode       = Off
Dante_presat   = FALSE
Initial_wait    = 1 [s]
Recvr_gain     = 48
Relaxation_delay = 2 [s]
Repetition_time = 3.74587904 [s]
Temp_get       = 394.6 [dC]
  
```



^1H NMR spectra of moracin E (4)



¹³C NMR spectra of moracin E (4)



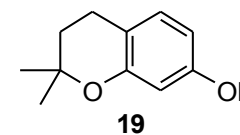
```

Filename      = NMP2NP19 1H-10.jdf
Author       = PRAMOD KUMAR
Experiment   = single pulse.ex2
Sample id    = S#153559
Solvent      = CHLOROFORM-D
Creation time = 12-SEP-2017 19:57:01
Revision time = 13-SEP-2017 11:02:08
Current Time = 13-SEP-2017 11:05:30

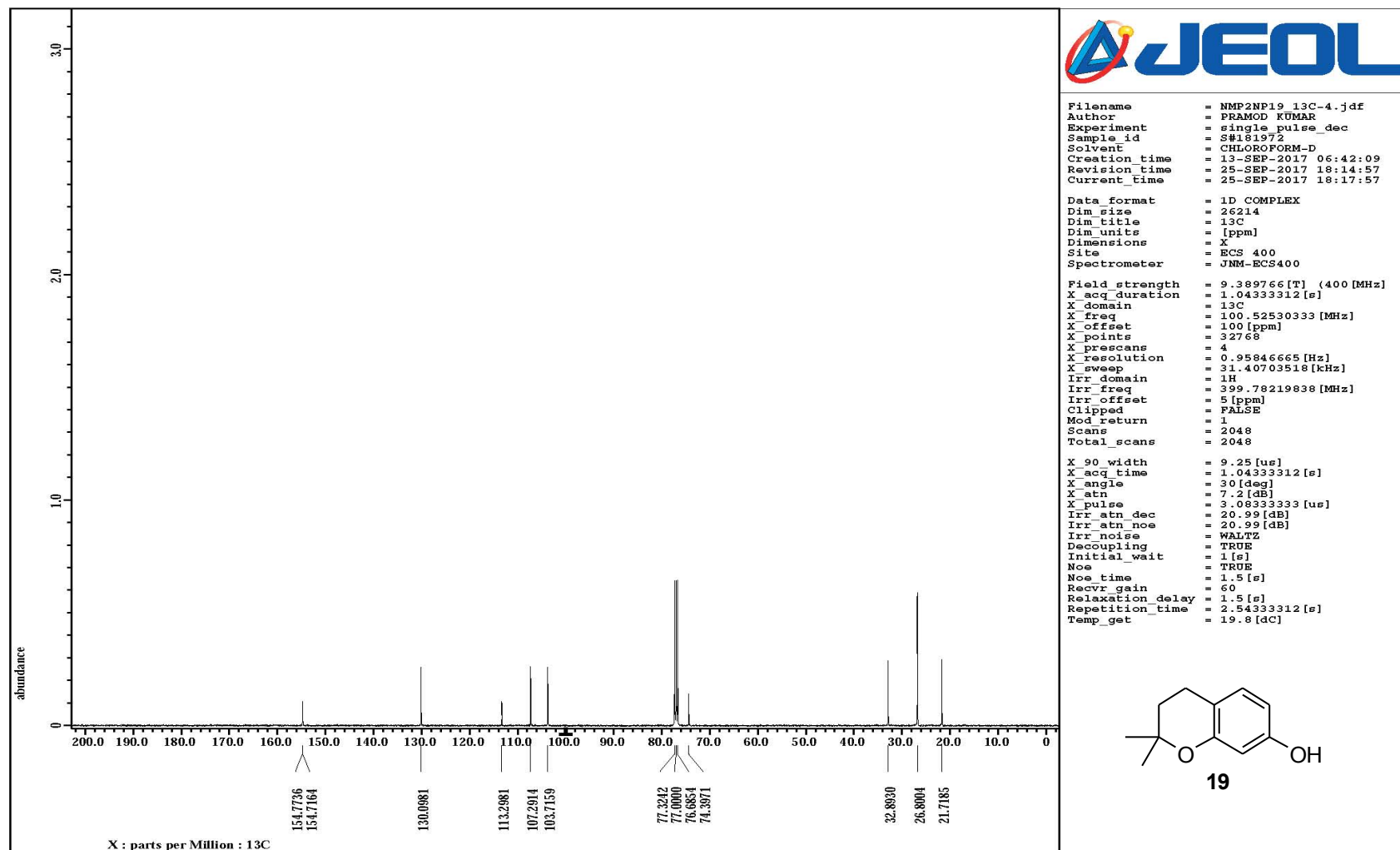
Data format   = 1D COMPLEX
Dim size      = 13107
Dim title     = 1H
Dim units     = [ppm]
Dimensions    = X
Site          = ECS 400
Spectrometer  = JNM-ECS400

Field strength = 9.389766[T] (400 [MHz])
X_acq_duration = 2.18365952[s]
X_domain      = 1H
X_freq        = 399.78219838 [MHz]
X_offset      = 5 [ppm]
X_points      = 16384
X_prescans    = 1
X_resolution  = 0.45794685 [Hz]
X_sweep       = 7.5030012 [kHz]
Irr_domain    = 1H
Irr_freq      = 399.78219838 [MHz]
Irr_offset    = 5 [ppm]
Tri_domain    = 1H
Tri_freq      = 399.78219838 [MHz]
Tri_offset    = 5 [ppm]
Clipped       = FALSE
Mod_return    = 1
Scans         = 16
Total_scans   = 16

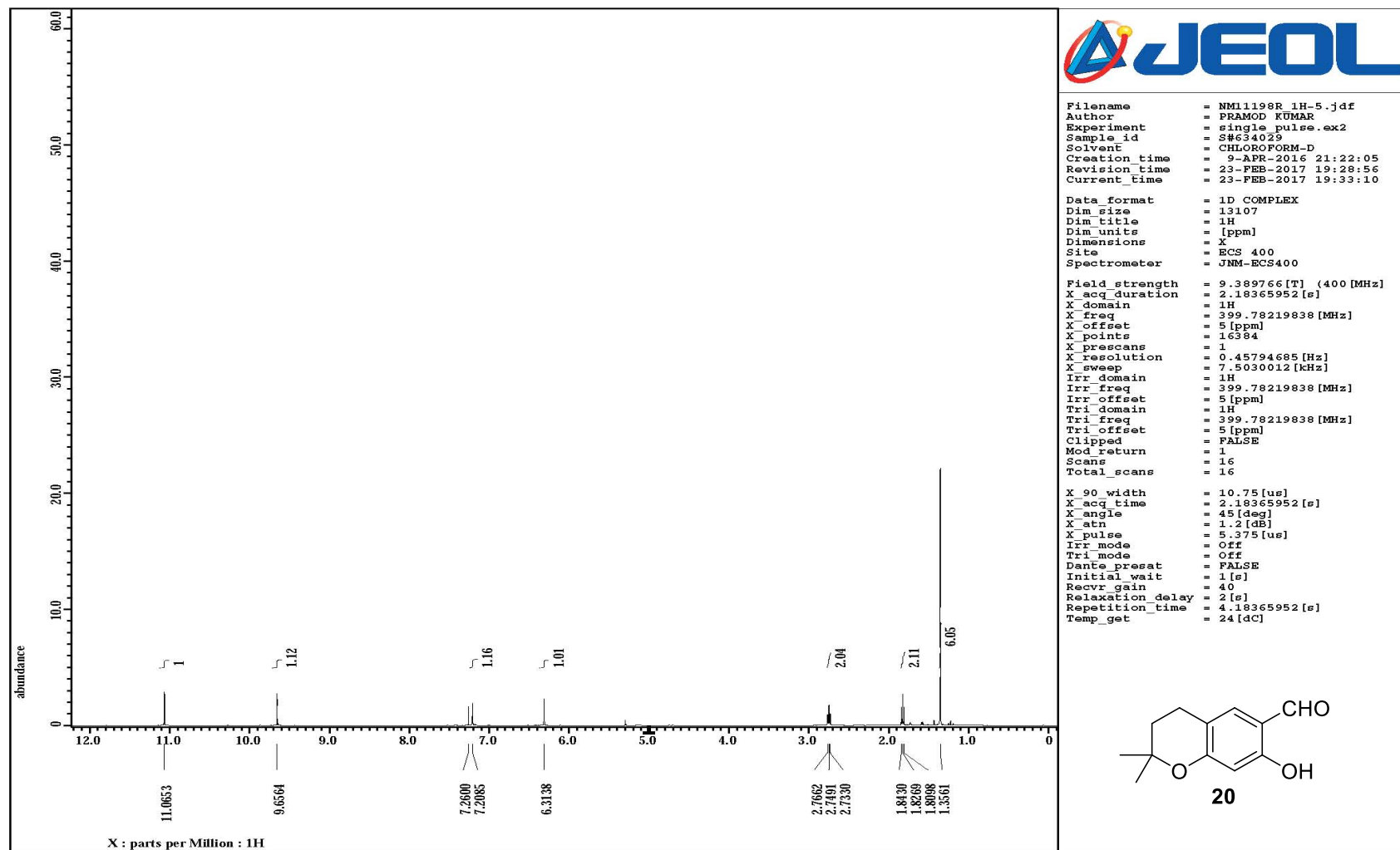
X_90_width    = 10.5 [us]
X_acq_time     = 2.18365952[s]
X_angle       = 45 [deg]
X_atn         = 0.2 [dB]
X_pulse       = 5.25 [us]
Irr_mode      = Off
Tri_mode      = Off
Dante_preset  = FALSE
Initial_wait   = 1 [s]
Recvr_gain    = 32
Relaxation_delay = 2 [s]
Repetition_time = 4.18365952[s]
Temp_get      = 23.6 [dC]
  
```



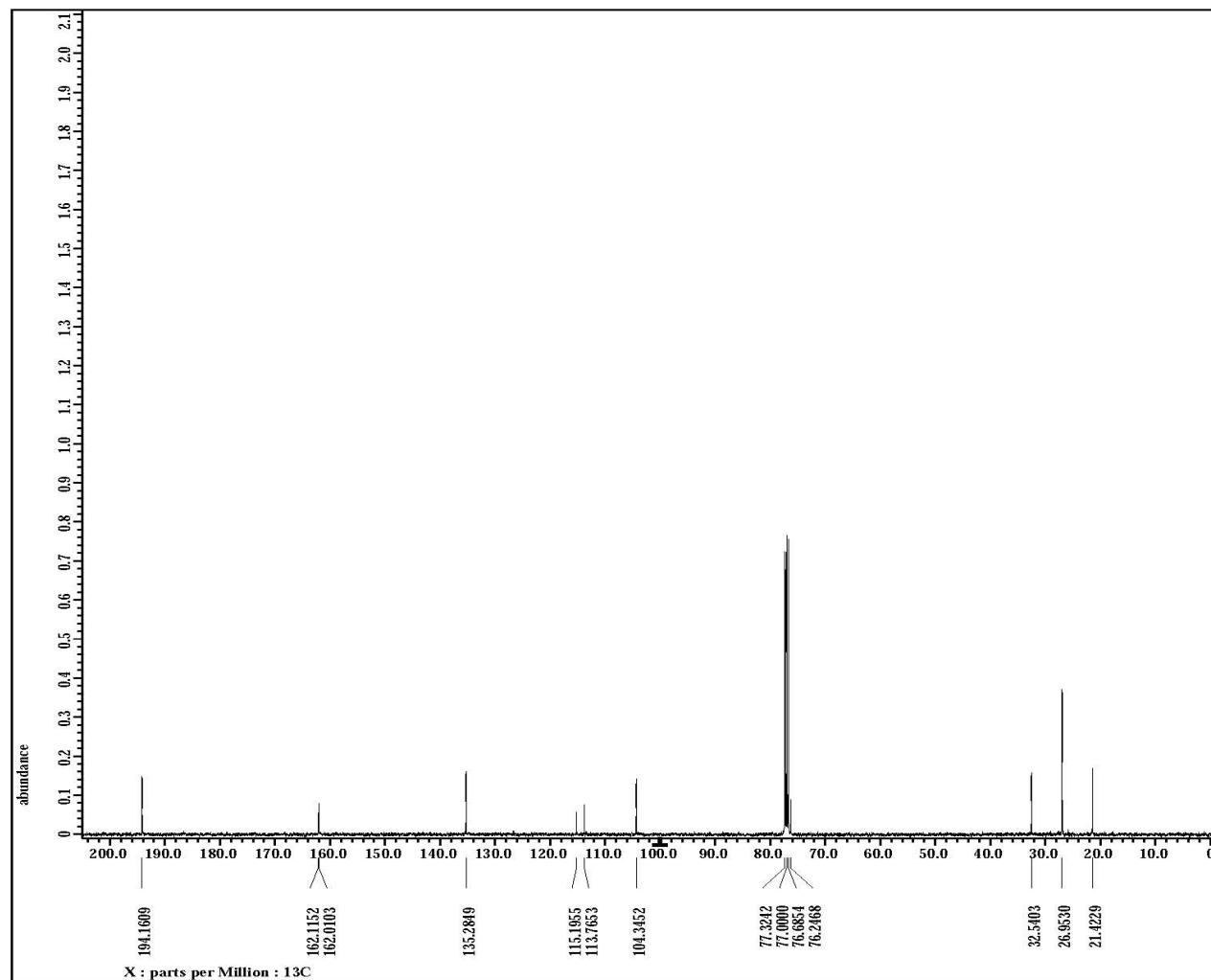
^1H NMR spectrum of Compound 19



^{13}C NMR spectrum of Compound **19**



¹H NMR spectra of Compound **20**



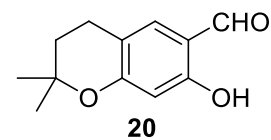
```

Filename      = NM11198R 13C-3.jdf
Author       = PRAMOD KUMAR
Experiment    = single pulse_dec
Sample id    = S#673764
Solvent      = CHLOROFORM-D
Creation time = 10-APR-2016 07:36:36
Revision time = 23-FEB-2017 19:33:58
Current time  = 23-FEB-2017 19:36:34

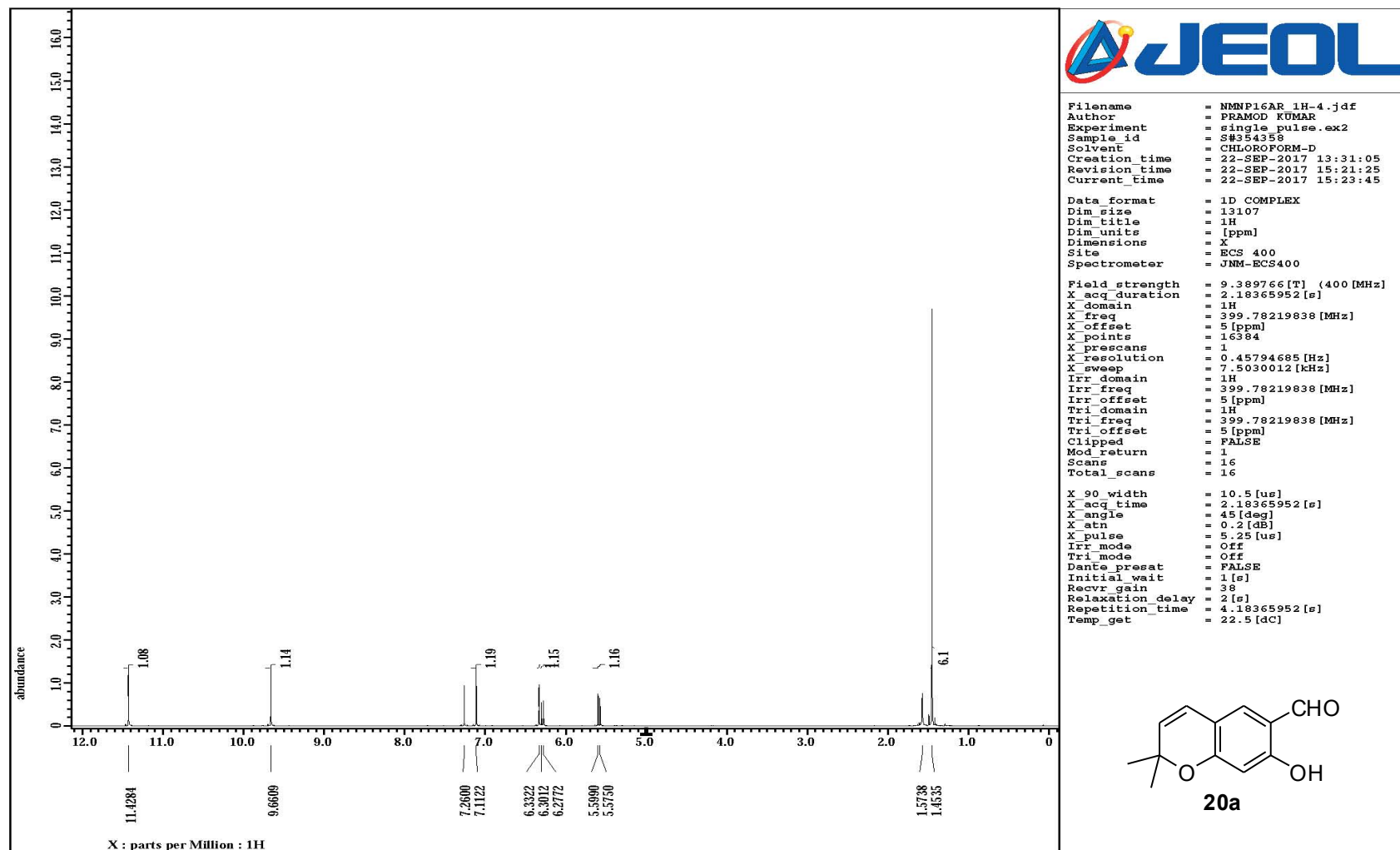
Data format   = 1D COMPLEX
Dim size      = 26214
Dim title     = 13C
Dim units     = [ppm]
Dimensions    = X
Site          = ECS 400
Spectrometer  = JNM-ECS400

Field strength = 9.389766[T] (400 [MHz])
X_acq duration = 1.04333312 [s]
X_domain      = 13C
X_freq        = 100.52530333 [MHz]
X_offset      = 100 [ppm]
X_points      = 32768
X_prescans    = 4
X_resolution  = 0.95846665 [Hz]
X_sweep       = 31.40703518 [kHz]
Irr_domain    = 1H
Irr_freq      = 399.78219838 [MHz]
Irr_offset    = 5 [ppm]
Clipped       = TRUE
Mod return    = 1
Scans         = 1024
Total_scans   = 1024

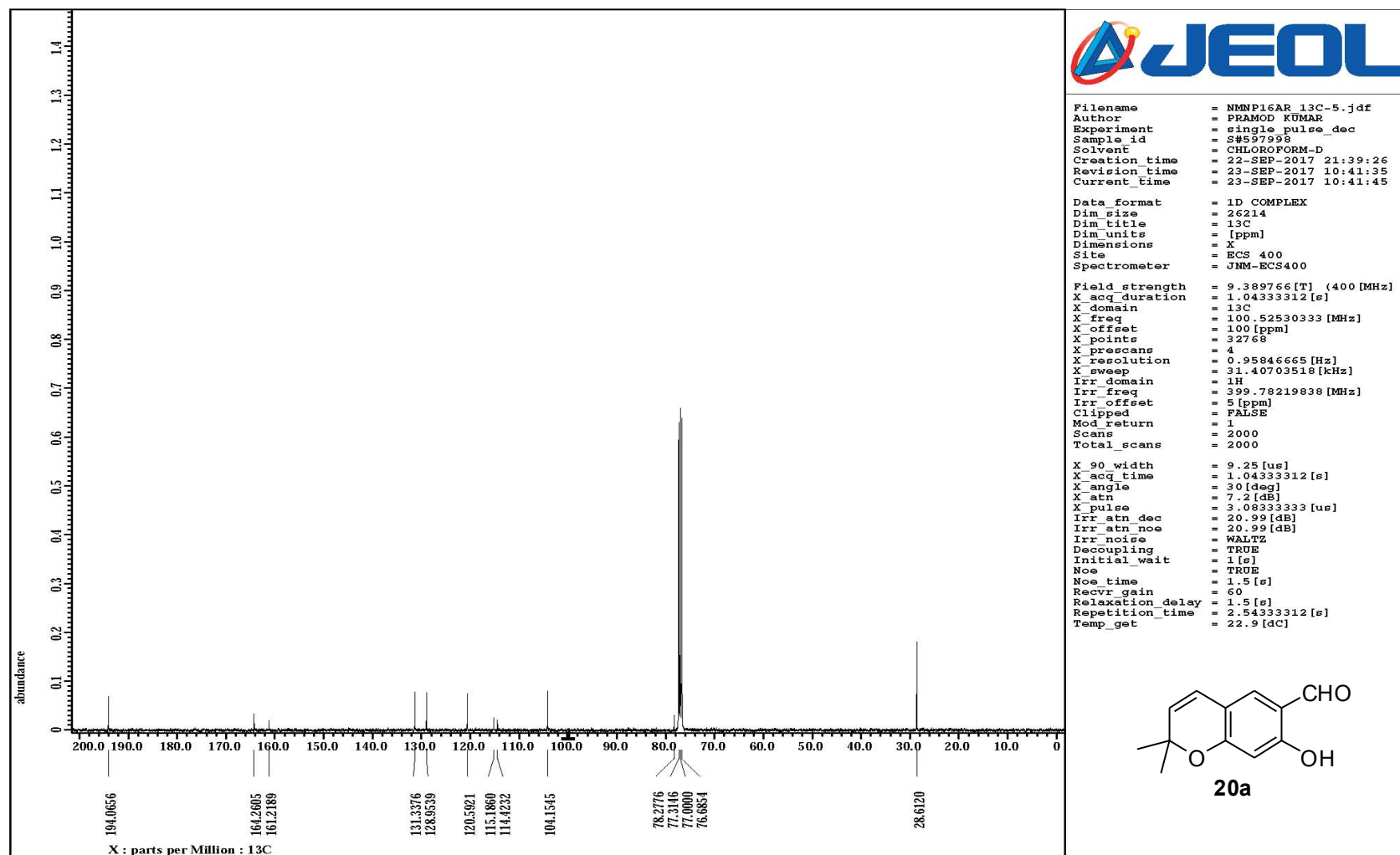
X_90 width    = 8.5 [us]
X_acq time    = 1.043333312 [s]
X_angle       = 30 [deg]
X_atn         = 5 [dB]
X_pulse       = 2.83333333 [us]
Irr_atn_dec   = 21.79 [dB]
Irr_atn_noe   = 21.79 [dB]
Irr_noise     = WALTZ
Decoupling    = TRUE
Initial_wait  = 1 [s]
Noe           = TRUE
Noe time      = 1.5 [s]
RecVr gain    = 60
Relaxation delay = 1.5 [s]
Repetition_time = 2.543333312 [s]
Temp_get      = 23.2 [dC]
  
```



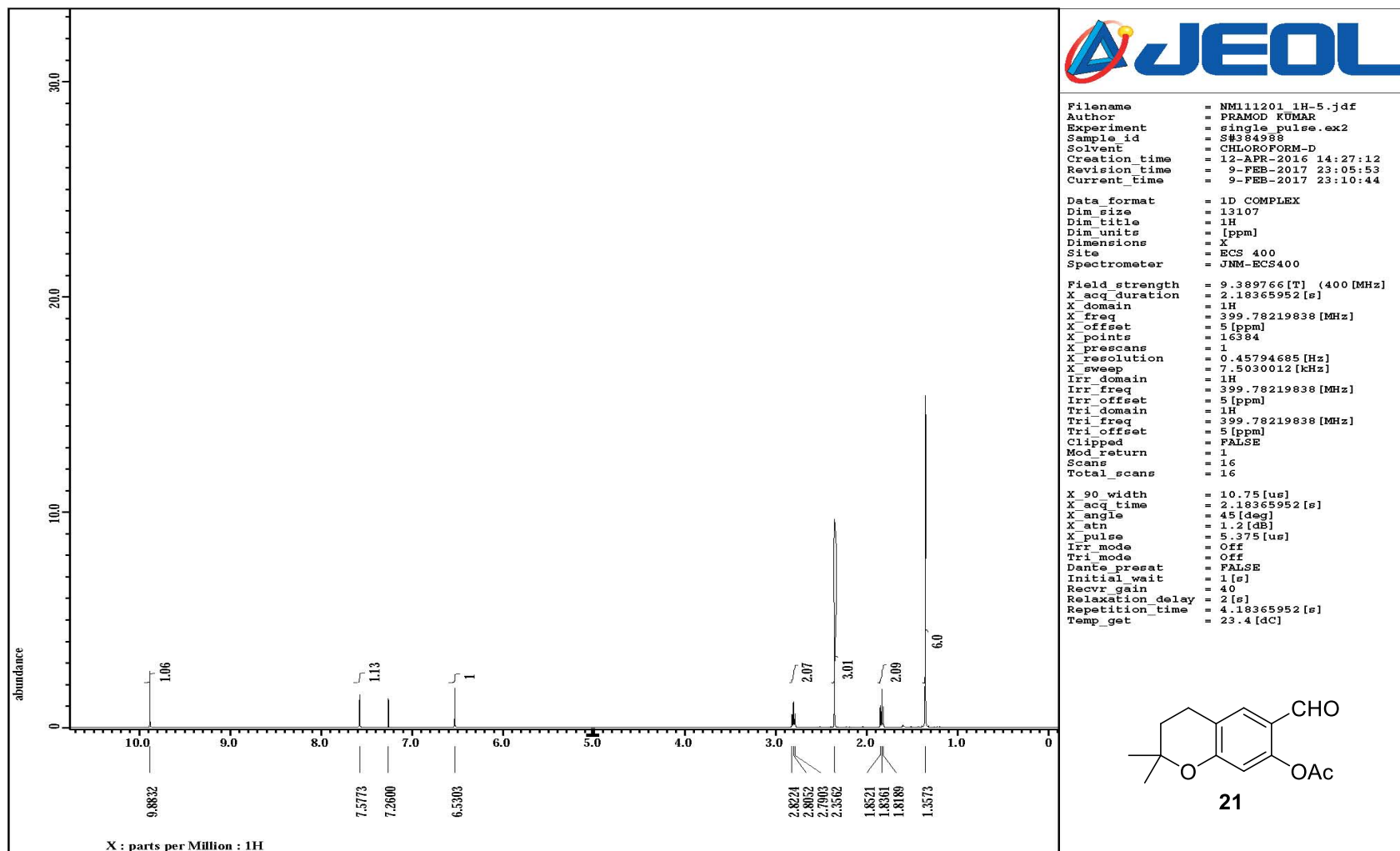
^{13}C NMR spectra of Compound 20



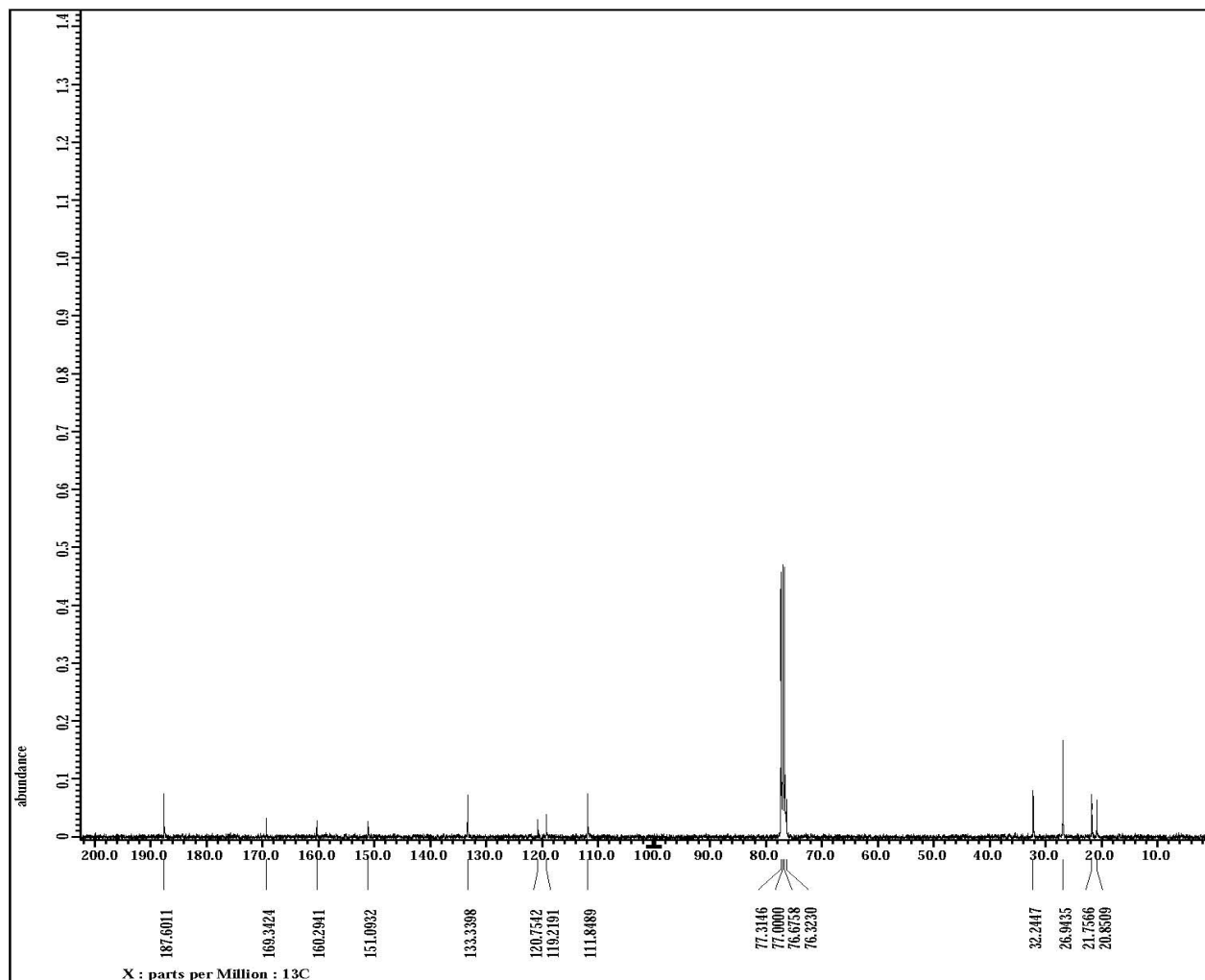
¹H NMR spectrum of Compound **20a**



^{13}C NMR spectrum of Compound **20a**



¹H NMR spectra of Compound **21**



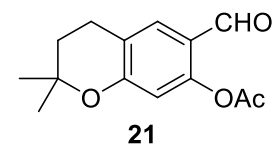
```

Filename      = NM11201_13C-3.jdf
Author       = PRAMOD KUMAR
Experiment   = single pulse_dec
Sample_id    = S#603554
Solvent      = CHLOROFORM-D
Creation_time = 13-APR-2016 22:00:31
Revision_time = 9-FEB-2017 23:18:30
Current_Time = 9-FEB-2017 23:20:18

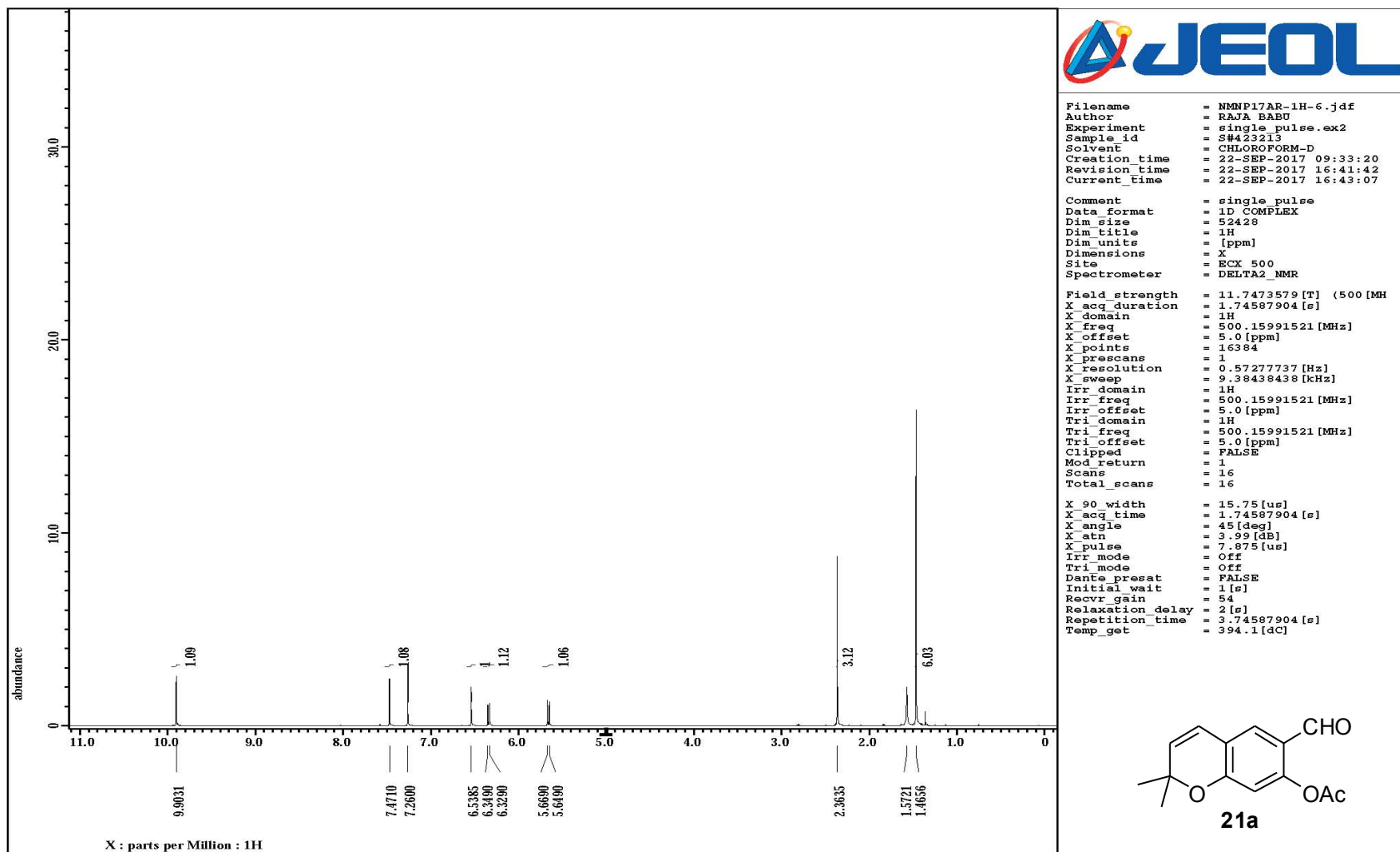
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECS 400
Spectrometer = JNM-ECS400

Field_strength = 9.389766[T] (400 [MHz])
X_acq_duration = 1.04333312[s]
X_domain      = 13C
X_freq        = 100.52530333 [MHz]
X_offset      = 100 [ppm]
X_points      = 32768
X_prescans    = 4
X_resolution  = 0.95846665 [Hz]
X_sweep       = 31.40703518 [kHz]
Irr_domain    = 1H
Irr_freq      = 399.78219838 [MHz]
Irr_offset    = 5 [ppm]
Clipped       = TRUE
Mod_return    = 1
Scans         = 1024
Total_scans   = 1024

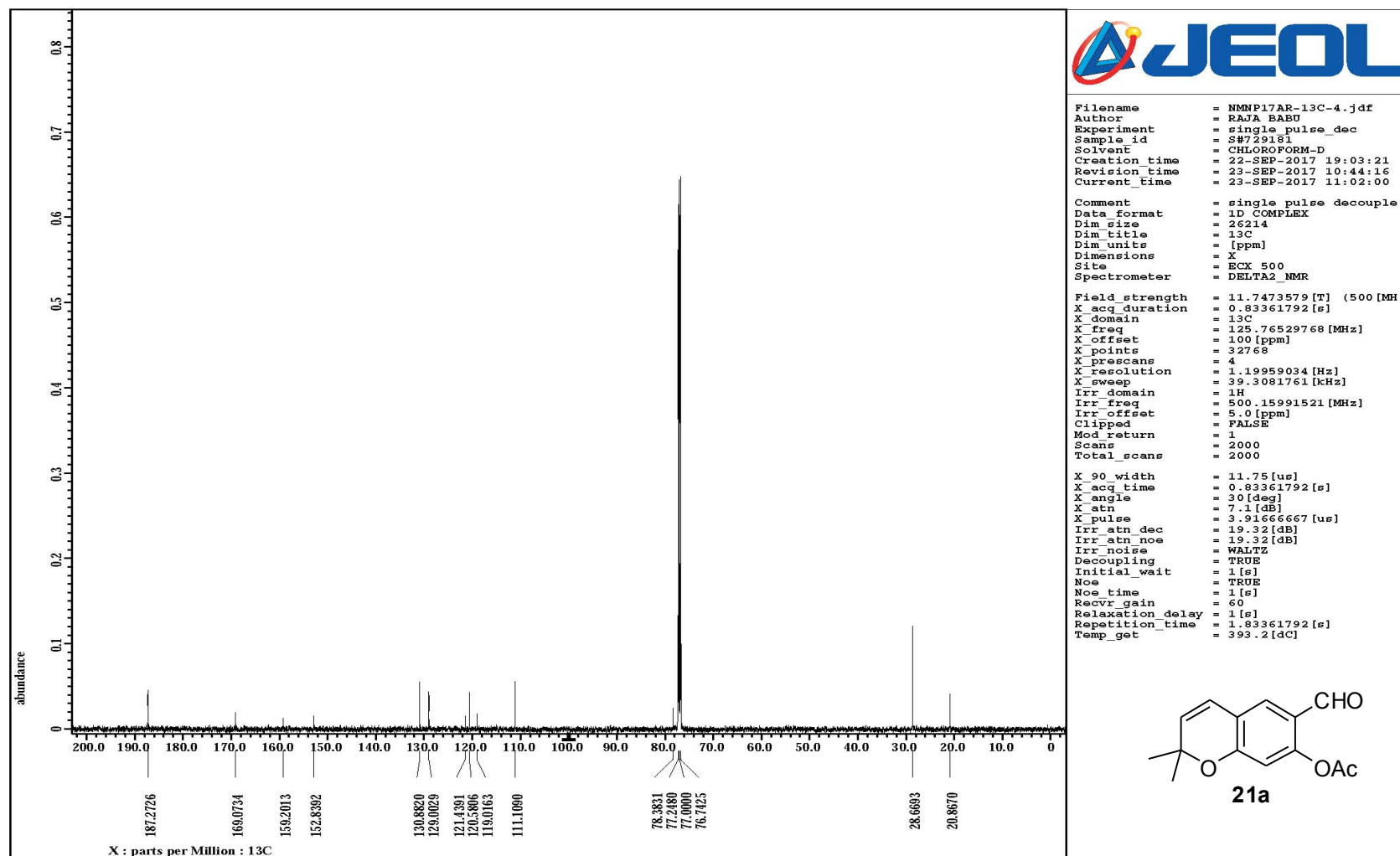
X_90_width    = 8.5 [us]
X_acq_time    = 1.04333312 [s]
X_angle       = 30 [deg]
X_atn         = 5 [dB]
X_pulse       = 2.83333333 [us]
Irr_atn_dec   = 21.79 [dB]
Irr_atn_noe   = 21.79 [dB]
Irr_noise     = WALTZ
Decoupling    = TRUE
Initial_wait  = 1 [s]
Noe           = TRUE
Noe_time      = 1.5 [s]
Recvr_gain    = 60
Relaxation_delay = 1.5 [s]
Repetition_time = 2.54333312 [s]
Temp_get      = 23.6 [dC]
  
```



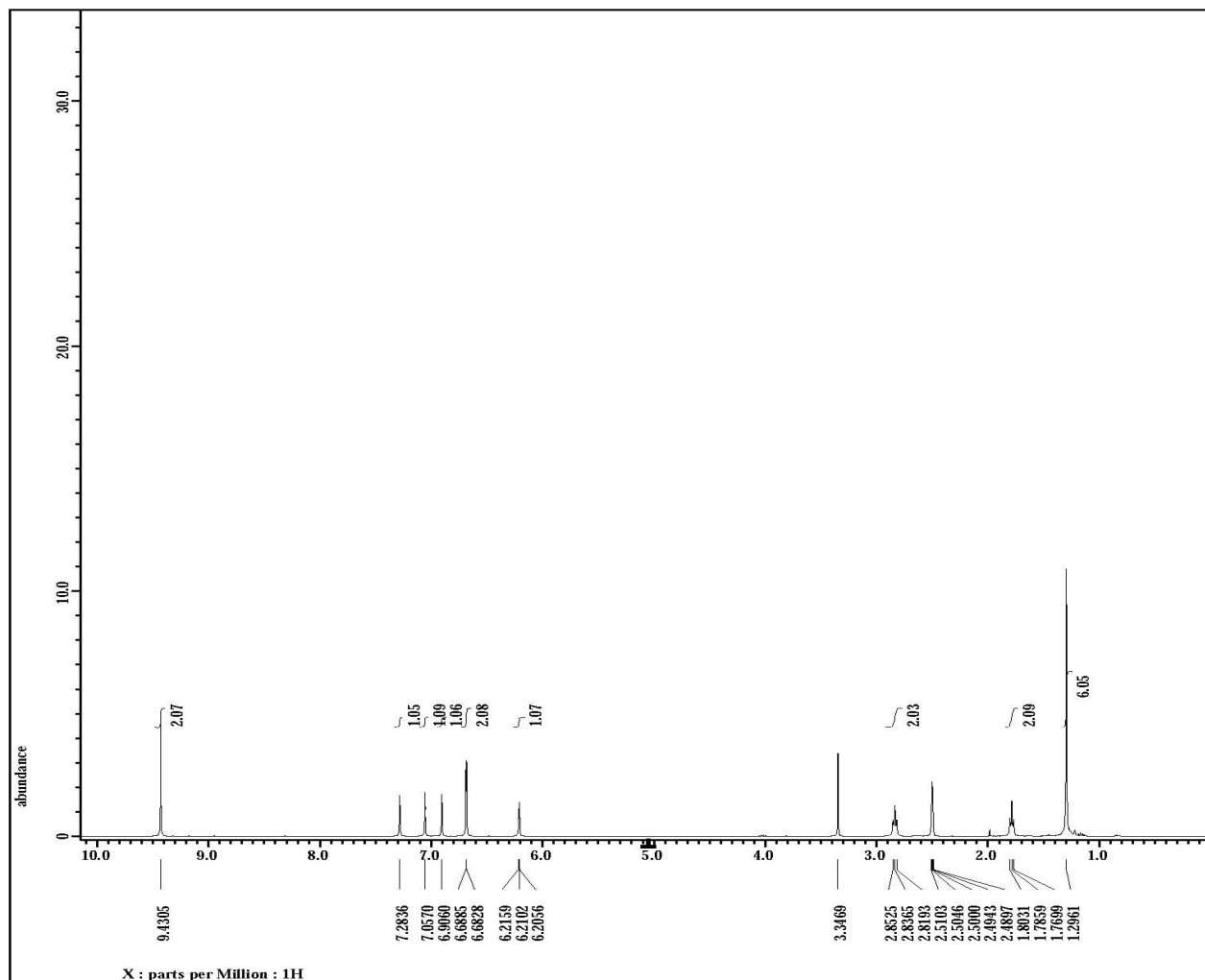
^{13}C NMR spectra of Compound 21



^1H NMR spectrum of Compound **21a**



¹³C NMR spectrum of Compound **21a**



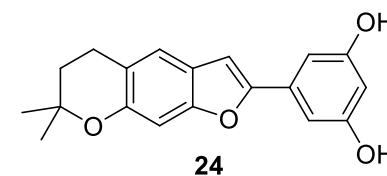
```

Filename      = NM11227R2 1H-2.jdf
Author       = Bhanu Tiwari
Experiment   = single pulse.ex2
Sample id    = S#344033
Solvent      = DMSO-D6
Creation time = 25-APR-2016 12:57:31
Revision time = 9-FEB-2017 22:02:57
Current Time = 9-FEB-2017 22:14:24

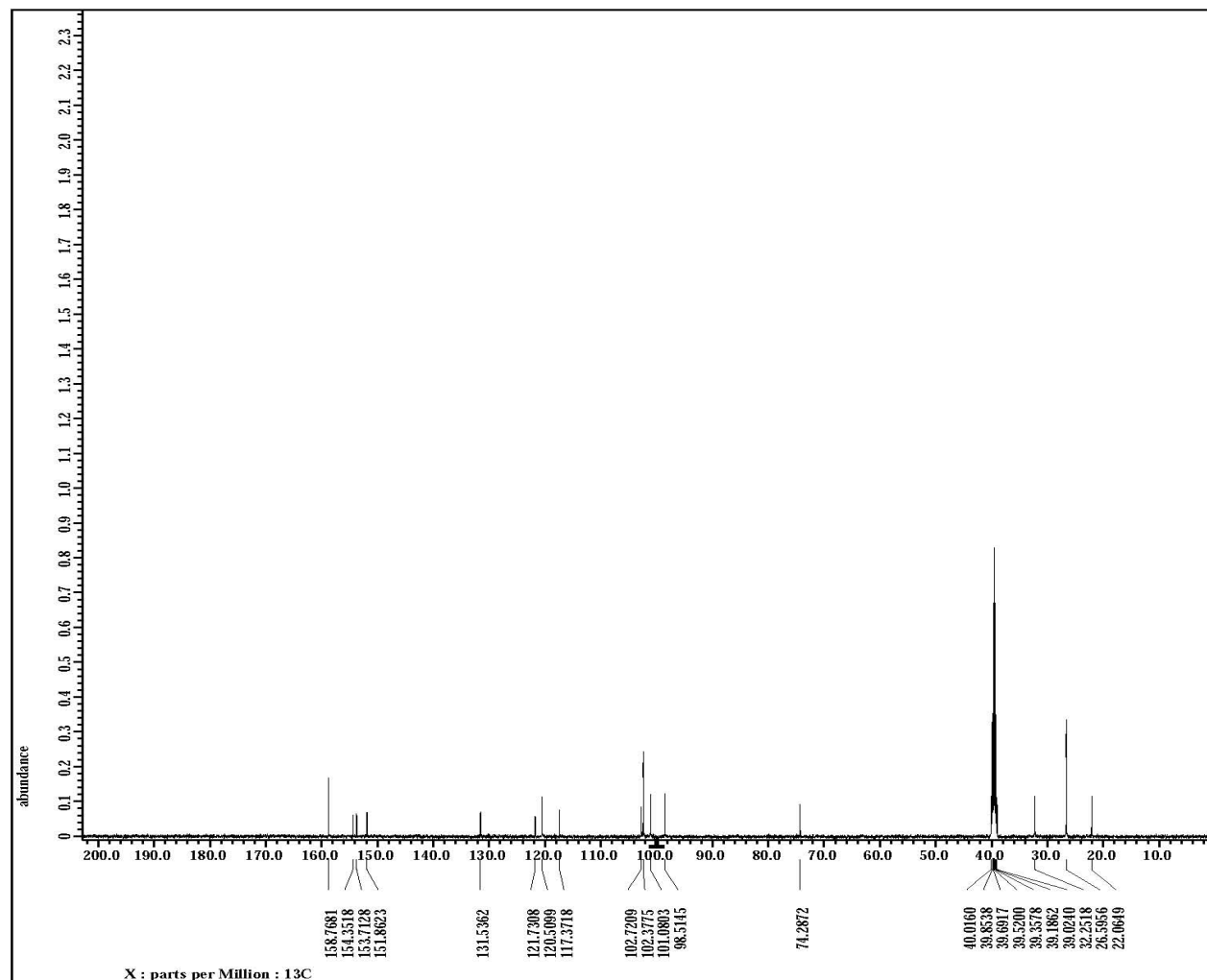
Data format   = 1D COMPLEX
Dim size      = 13107
Dim title     = 1H
Dim units     = [ppm]
Dimensions    = X
Site          = ECS 400
Spectrometer  = JNM-ECS400

Field strength = 9.2982153 [T] (400 [MHz]
X_acq_duration = 2.20725248 [s]
X_domain      = 1H
X_freq        = 395.88430144 [MHz]
X_offset      = 5 [ppm]
X_points      = 16384
X_prescans    = 1
X_resolution  = 0.45305193 [Hz]
X_sweep       = 7.42280285 [kHz]
Irr_domain    = 1H
Irr_freq      = 395.88430144 [MHz]
Irr_offset    = 5 [ppm]
Tri_domain    = 1H
Tri_freq      = 395.88430144 [MHz]
Tri_offset    = 5 [ppm]
Clipped       = FALSE
Mod_return    = 1
Scans         = 32
Total_scans   = 32

X_90_width    = 10.25 [us]
X_acq_time     = 2.20725248 [s]
X_angle       = 45 [deg]
X_atn         = 0.6 [dB]
X_pulse       = 5.125 [us]
Irr_mode      = Off
Tri_mode      = Off
Dante_preset  = FALSE
Initial_wait   = 1 [s]
Recvr_gain    = 42
Relaxation_delay = 2 [s]
Repetition_time = 4.20725248 [s]
Temp_get      = 22 [dC]
  
```



^1H NMR spectra of Compound **24**



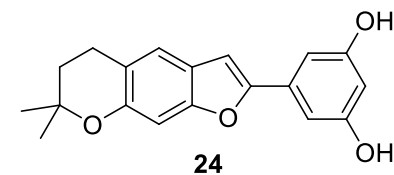
```

Filename      = NM11227R2-13C-4.jdf
Author       = RAJA BABU
Experiment   = single pulse_dec
Sample_id    = S#720030
Solvent      = DMSO-D6
Creation_time = 25-APR-2016 18:26:45
Revision_time = 9-FEB-2017 22:09:42
Current_Time = 9-FEB-2017 22:11:17

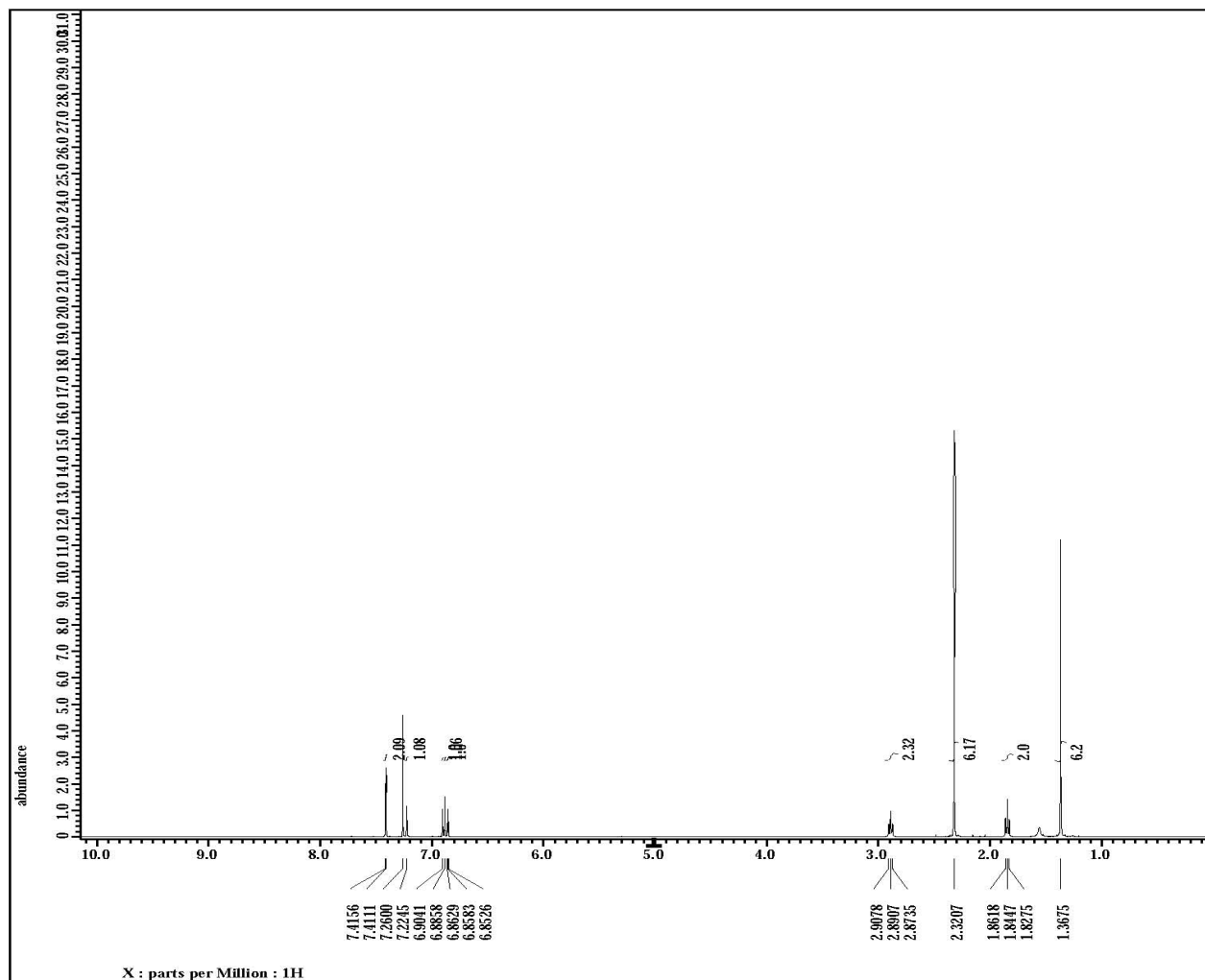
Comment      = single pulse decouple
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECX 500
Spectrometer = DELTA2_NMR

Field_strength = 11.7473579 [T] (500 [MH]
X_acq_duration = 0.83361792 [s]
X_domain       = 13C
X_freq         = 125.76529768 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034 [Hz]
X_sweep        = 39.3081761 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 1000
Total_scans    = 1000

X_90_width     = 11.75 [us]
X_acq_time     = 0.83361792 [s]
X_angle        = 30 [deg]
X_atn          = 7.1 [dB]
X_pulse        = 3.91666667 [us]
Irr_atn_dec    = 19.32 [dB]
Irr_atn_noe    = 19.32 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 0.1 [s]
Recvr_gain     = 60
Relaxation_delay = 0.1 [s]
Repetition_time = 0.93361792 [s]
Temp_get       = 28.6 [dC]
  
```



^{13}C NMR spectra of Compound 24

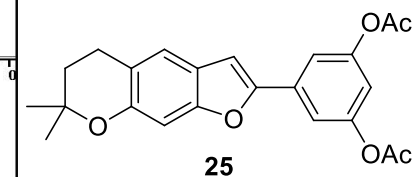


Filename = NMNP30R_1H-5.jdf
 Author = SUNEETA SHARMA
 Experiment = single pulse.ex2
 Sample id = S#579482
 Solvent = CHLOROFORM-D
 Creation time = 10-FEB-2017 19:24:04
 Revision time = 10-FEB-2017 16:38:13
 Current Time = 10-FEB-2017 16:38:26

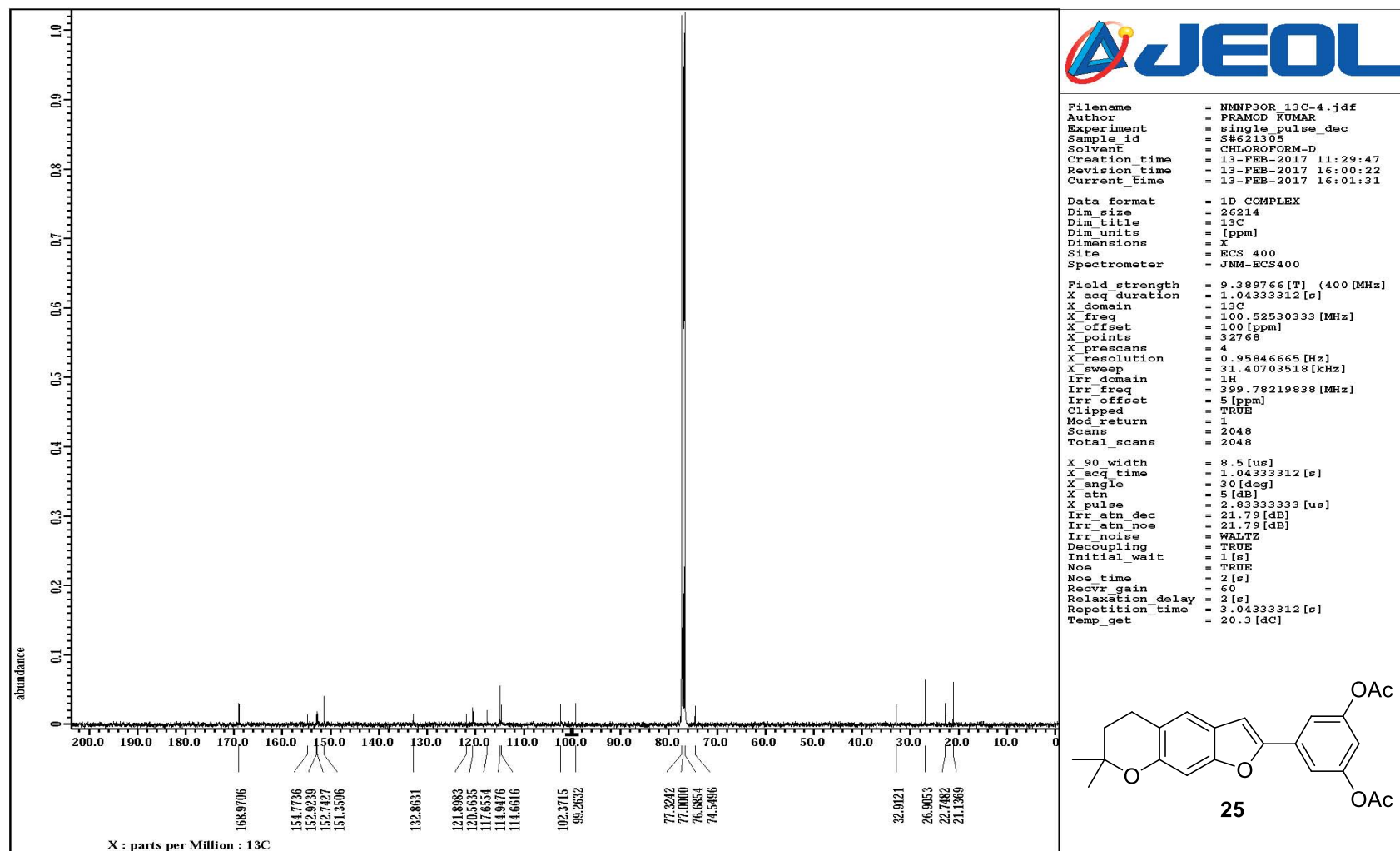
 Data format = 1D COMPLEX
 Dim size = 13107
 Dim title = 1H
 Dim units = [ppm]
 Dimensions = X
 Site = ECS 400
 Spectrometer = JNM-ECS400

 Field strength = 9.2982153 [T] (400 [MHz]
 X_acq duration = 2.20725248 [s]
 X_domain = 1H
 X_freq = 395.88430144 [MHz]
 X_offset = 5 [ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.45305193 [Hz]
 X_sweep = 7.42280285 [kHz]
 Irr_domain = 1H
 Irr_freq = 395.88430144 [MHz]
 Irr_offset = 5 [ppm]
 Tri_domain = 1H
 Tri_freq = 395.88430144 [MHz]
 Tri_offset = 5 [ppm]
 Clipped = FALSE
 Mod return = 1
 Scans = 14
 Total_scans = 14

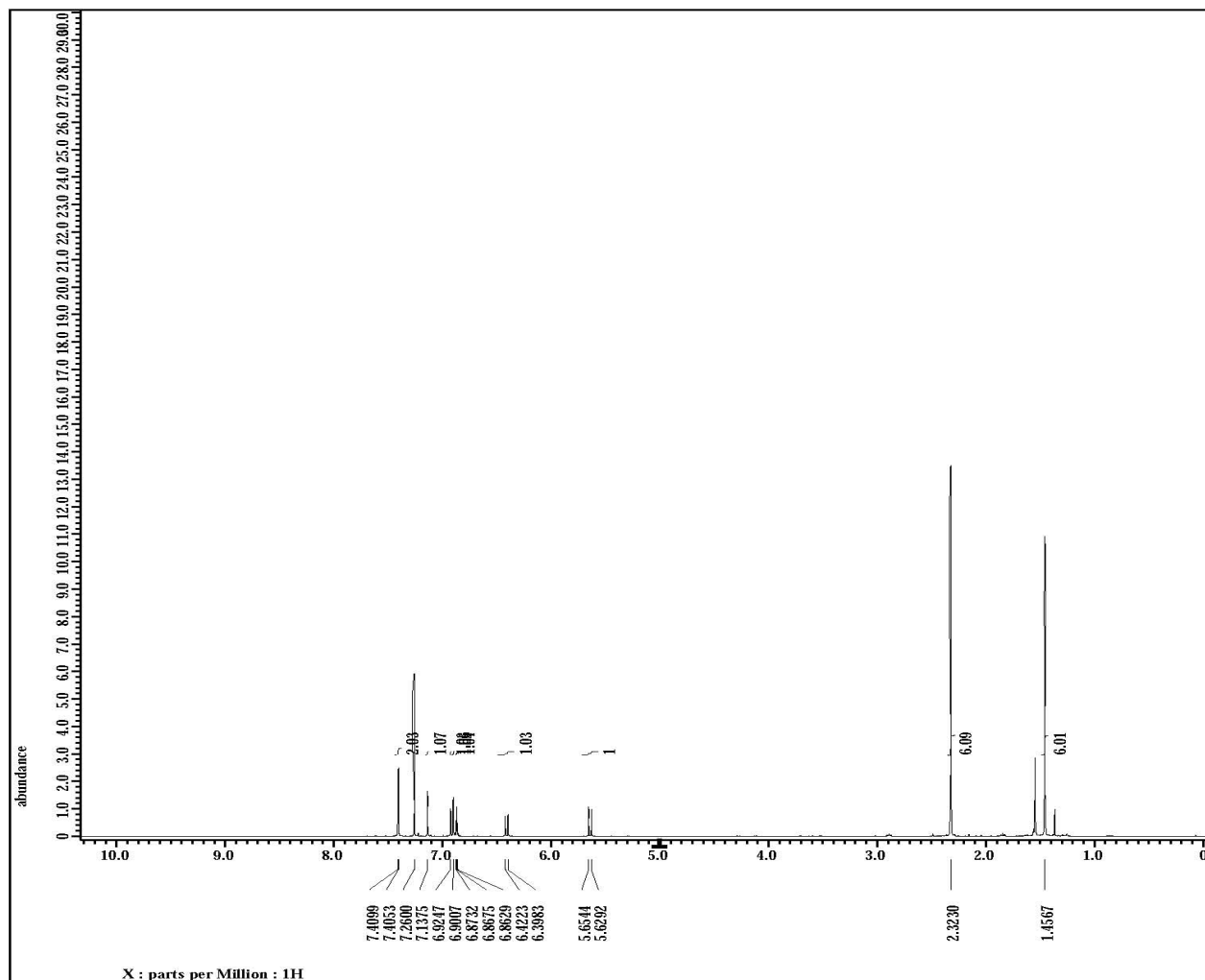
 X_90 width = 10 [us]
 X_acq time = 2.20725248 [s]
 X_angle = 45 [deg]
 X_atn = 0.6 [dB]
 X_pulse = 5 [us]
 Irr mode = Off
 Tri mode = Off
 Dante preset = FALSE
 Initial wait = 1 [s]
 Recvr gain = 48
 Relaxation delay = 1 [s]
 Repetition_time = 3.20725248 [s]
 Temp_get = 23.4 [dC]



^1H NMR spectra of Compound **25**



^{13}C NMR spectra of Compound **25**



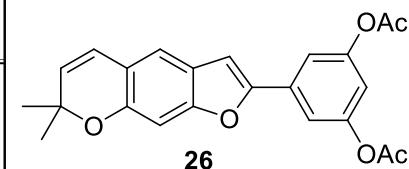
```

Filename      = NMNP31R_1H-5.jdf
Author       = SUNEETA SHARMA
Experiment   = single pulse.ex2
Sample_id    = S#575493
Solvent      = CHLOROFORM-D
Creation_time = 10-FEB-2017 19:17:15
Revision_time = 10-FEB-2017 16:53:43
Current_time  = 10-FEB-2017 17:19:34

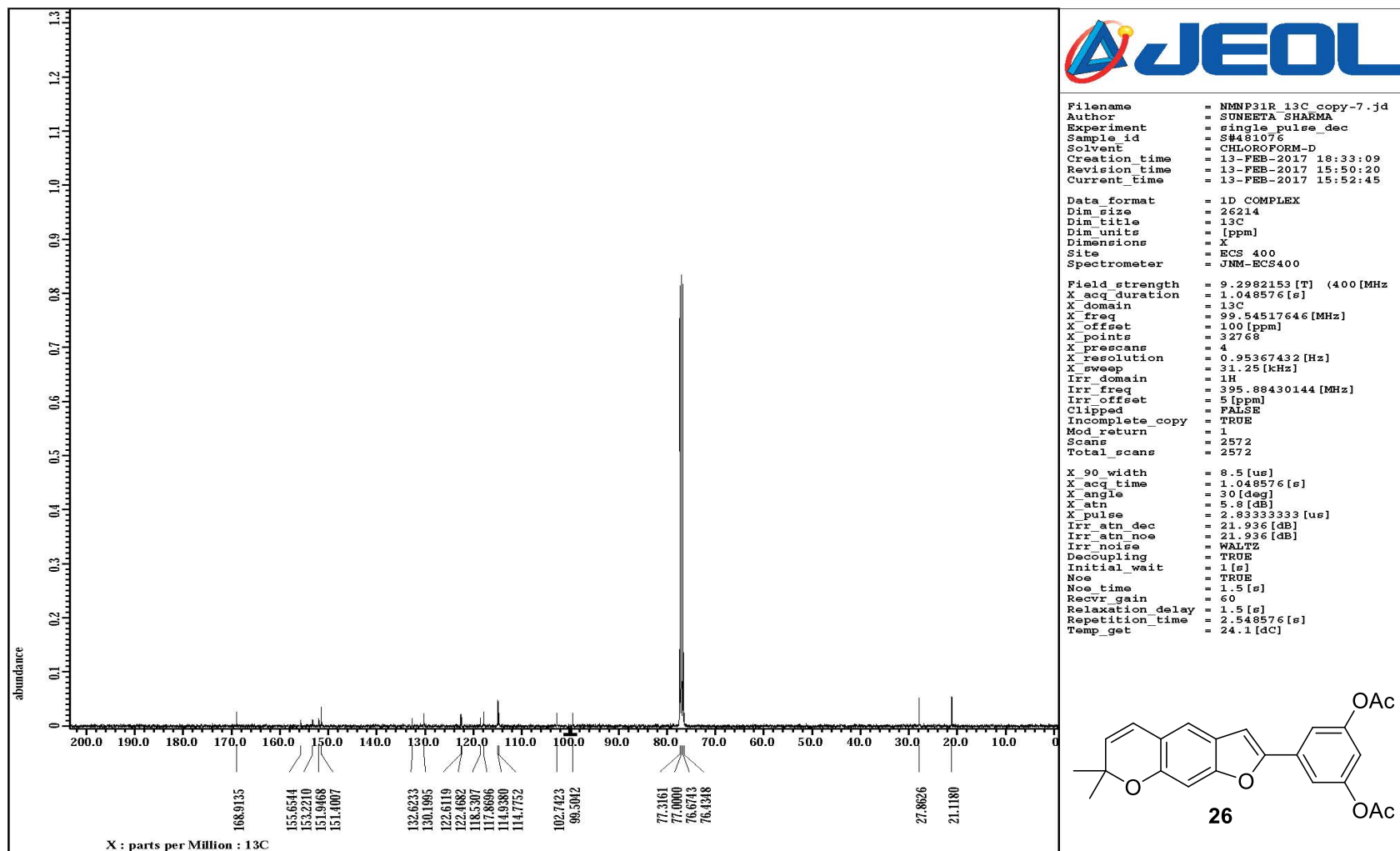
Data_format   = 1D COMPLEX
Dim_size      = 13107
Dim_title     = 1H
Dim_units     = [ppm]
Dimensions    = X
Site          = ECS 400
Spectrometer  = JNM-ECS400

Field_strength = 9.2982153 [T] (400 [MHz]
X_acq_duration = 2.20725248 [s]
X_domain      = 1H
X_freq        = 395.88430144 [MHz]
X_offset      = 5 [ppm]
X_points      = 16384
X_prescans    = 1
X_resolution  = 0.45305193 [Hz]
X_sweep       = 7.42280285 [kHz]
Irr_domain    = 1H
Irr_freq      = 395.88430144 [MHz]
Irr_offset    = 5 [ppm]
Tri_domain    = 1H
Tri_freq      = 395.88430144 [MHz]
Tri_offset    = 5 [ppm]
Clipped       = FALSE
Mod_return    = 1
Scans         = 11
Total_scans   = 11

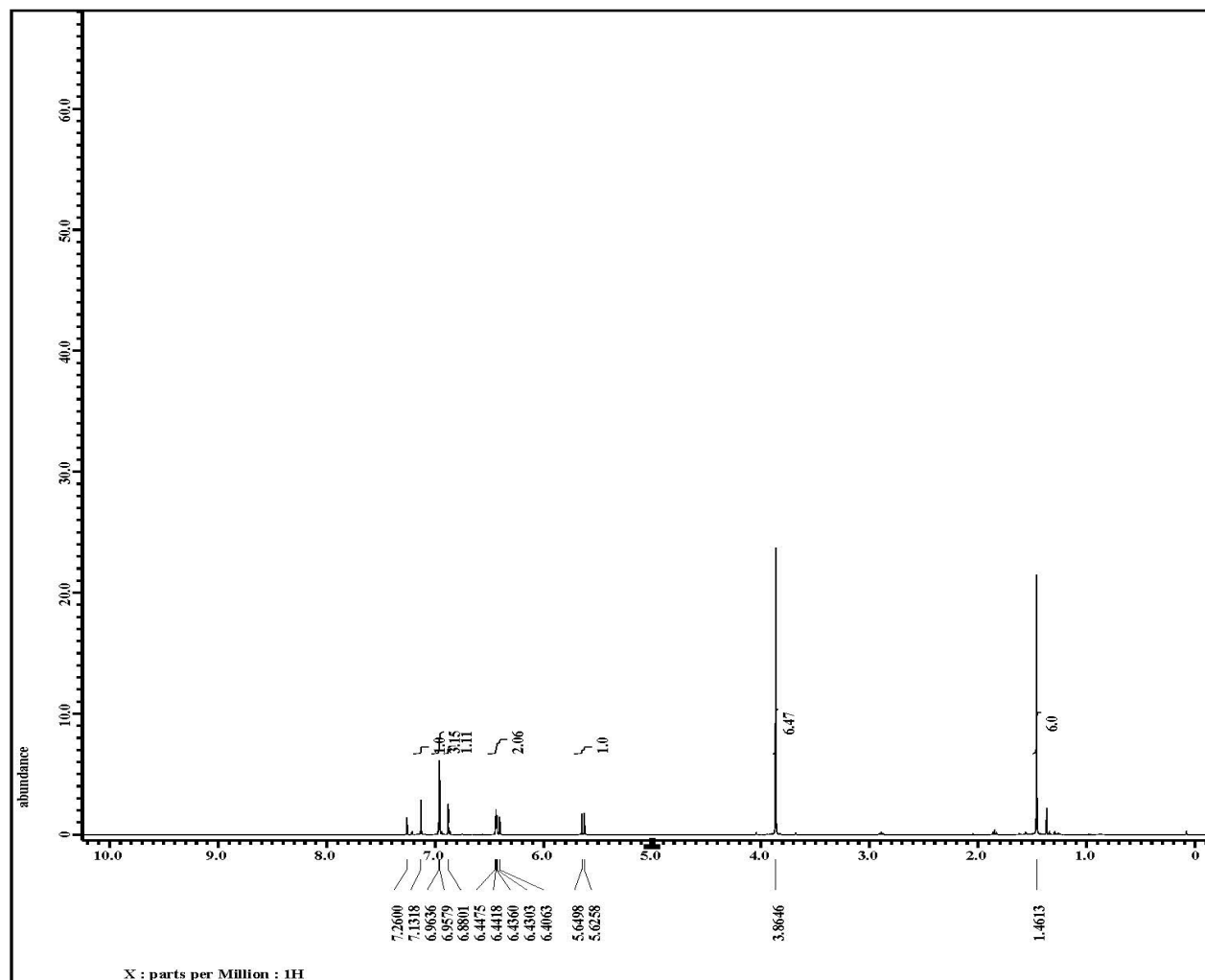
X_90_width    = 10 [us]
X_acq_time     = 2.20725248 [s]
X_angle       = 45 [deg]
X_atn         = 0.6 [dB]
X_pulse       = 5 [us]
Irr_mode      = Off
Tri_mode      = Off
Dante_presat  = FALSE
Initial_wait   = 1 [s]
Recvr_gain    = 50
Relaxation_delay = 1 [s]
Repetition_time = 3.20725248 [s]
Temp_get      = 23.3 [dC]
  
```



^1H NMR spectra of Compound **26**



¹³C NMR spectra of Compound 26



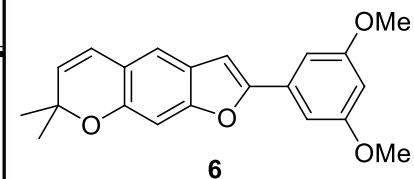
```

Filename      = NM124_1H-5.jdf
Author       = Bhanu Tiwari
Experiment   = single pulse.ex2
Sample_id    = S#524720
Solvent      = CHLOROFORM-D
Creation_time = 28-APR-2016 17:59:21
Revision_time = 30-AUG-2016 22:13:55
Current_time  = 30-AUG-2016 22:15:11

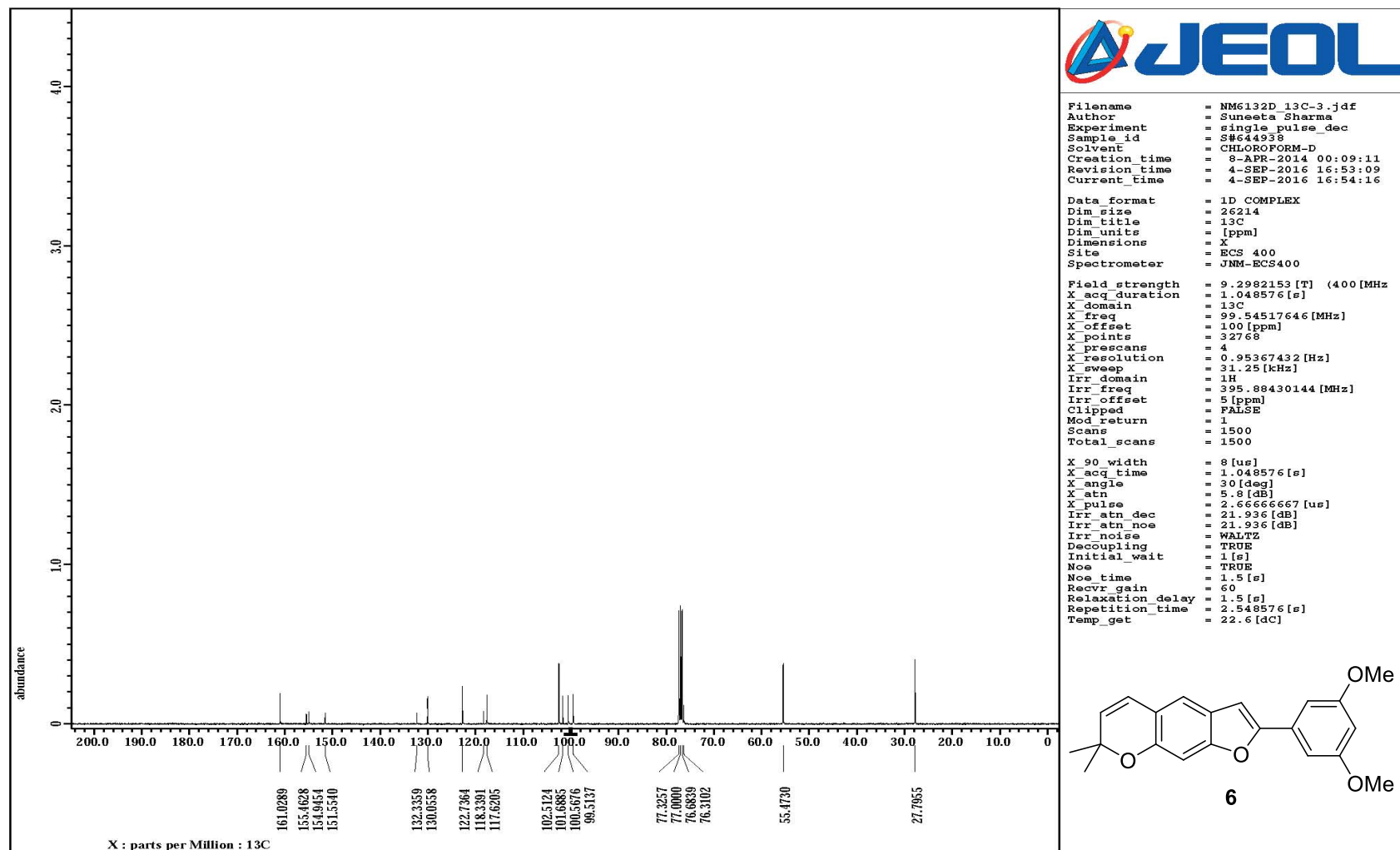
Data_format  = 1D_COMPLEX
Dim_size     = 13107
Dim_title    = 1H
Dim_units    = [ppm]
Dimensions   = X
Site         = ECS 400
Spectrometer = JNM-ECS400

Field_strength = 9.2982153 [T] (400 [MHz]
X_acq_duration = 2.20725248 [s]
X_domain       = 1H
X_freq         = 395.88430144 [MHz]
X_offset       = 5 [ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.45305193 [Hz]
X_sweep        = 7.42280285 [kHz]
Irr_domain     = 1H
Irr_freq       = 395.88430144 [MHz]
Irr_offset     = 5 [ppm]
Tri_domain     = 1H
Tri_freq       = 395.88430144 [MHz]
Tri_offset     = 5 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 32
Total_scans    = 32

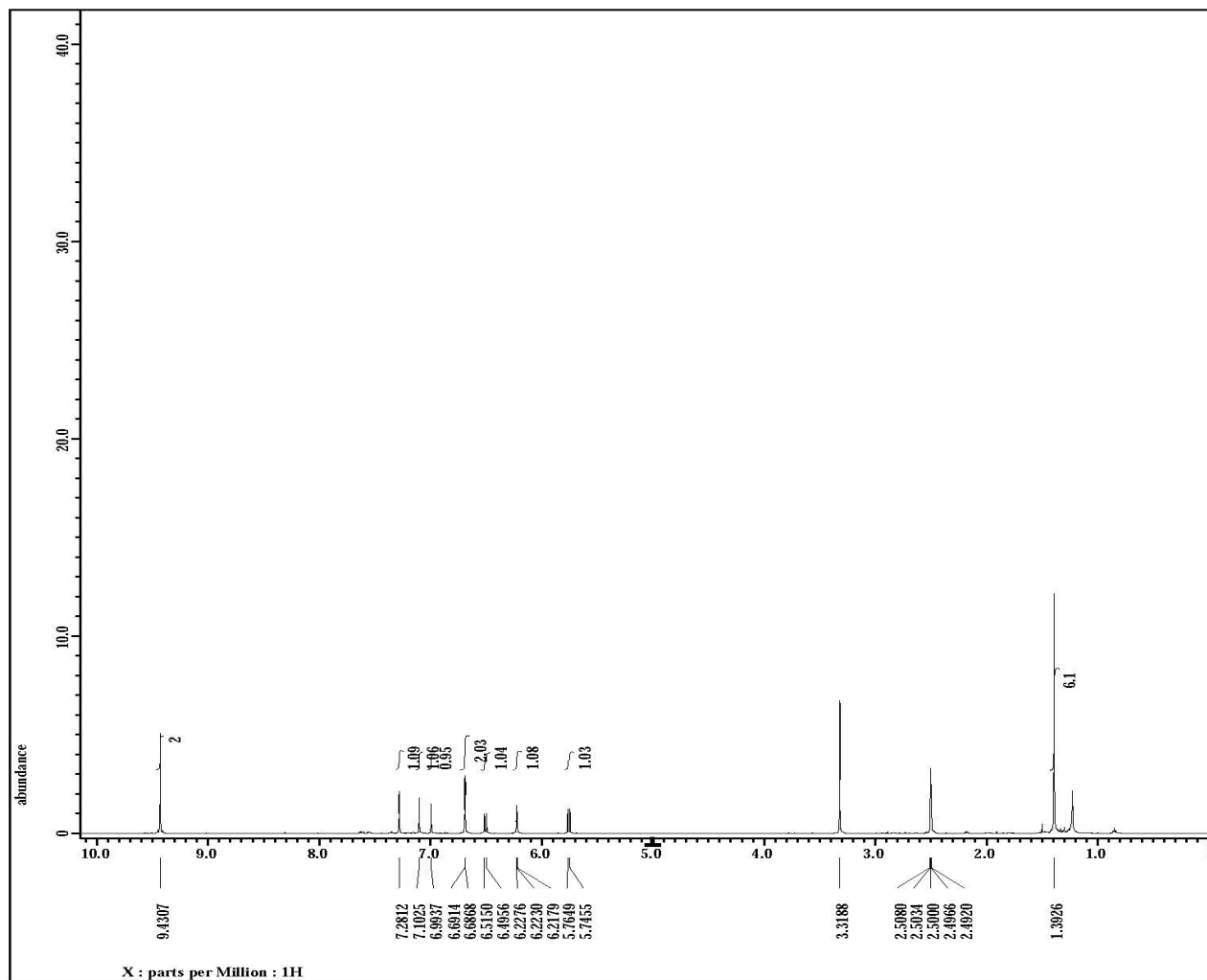
X_90_width    = 10.25 [us]
X_acq_time     = 2.20725248 [s]
X_angle        = 45 [deg]
X_atn          = 0.6 [dB]
X_pulse        = 5.125 [us]
Irr_mode       = Off
Tri_mode       = Off
Dante_preset   = FALSE
Initial_wait   = 1 [s]
Recvr_gain     = 40
Relaxation_delay = 2 [s]
Repetition_time = 4.20725248 [s]
Temp_get       = 24.4 [dC]
  
```



^1H NMR spectra of Compound 6



^{13}C NMR spectra of Compound 6



```

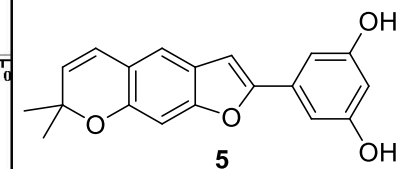
Filename      = NM12109-1H-6.jdf
Author       = RAJA BABU
Experiment   = single pulse.ex2
Sample id    = S#614776
Solvent      = DMSO-D6
Creation time = 20-JUN-2016 15:11:45
Revision time = 9-FEB-2017 18:55:31
Current Time = 9-FEB-2017 18:57:15

Comment      = single pulse
Data format  = 1D COMPLEX
Dim size     = 13107
Dim title    = 1H
Dim units    = [ppm]
Dimensions   = X
Site         = ECX 500
Spectrometer = DELTA2_NMR

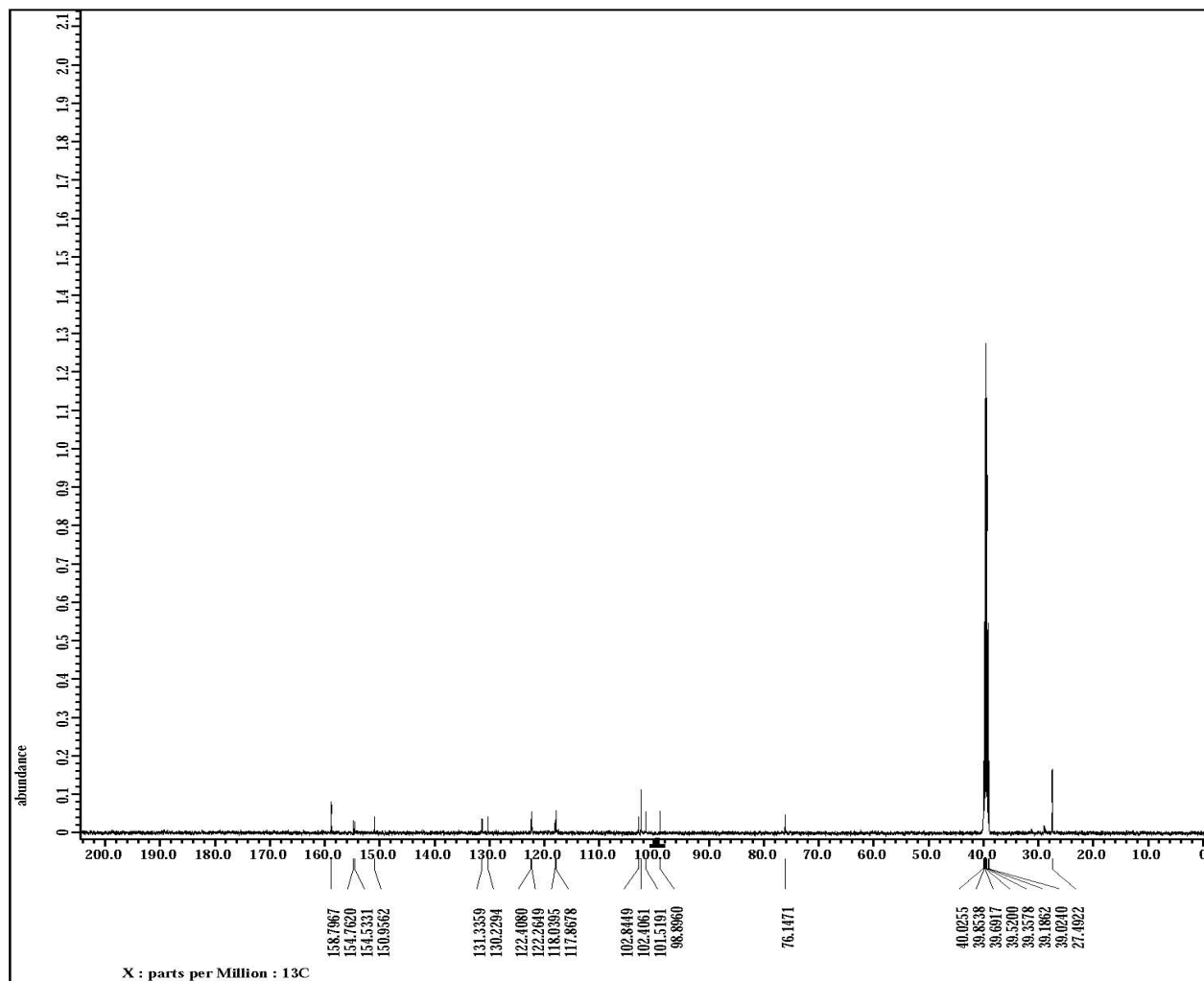
Field strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain       = 1H
X_freq         = 500.15991521 [MHz]
X_offset       = 5.0 [ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.57277737 [Hz]
X_sweep        = 9.38438458 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Tri_domain     = 1H
Tri_freq       = 500.15991521 [MHz]
Tri_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 16
Total_scans    = 16

X_90_width     = 15.75 [us]
X_acq_time     = 1.74587904 [s]
X_angle        = 45 [deg]
X_stn          = 3.99 [dB]
X_pulse        = 7.875 [us]
Irr_mode       = Off
Tri_mode       = Off
Dante_presat   = FALSE
Initial_wait    = 1 [s]
Recvr_gain     = 50
Relaxation_delay = 1.5 [s]
Repetition_time = 3.24587904 [s]
Temp_get       = 268 [dc]

```



^1H NMR spectra of Compound 5



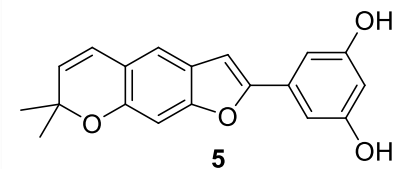
```

Filename      = NM12109-13C-5.jdf
Author       = RAJA BABU
Experiment    = single pulse_dec
Sample id    = S#188452
Solvent      = DMSO-D6
Creation time = 21-JUN-2016 03:50:50
Revision time = 9-FEB-2017 19:03:55
Current Time  = 9-FEB-2017 19:12:40

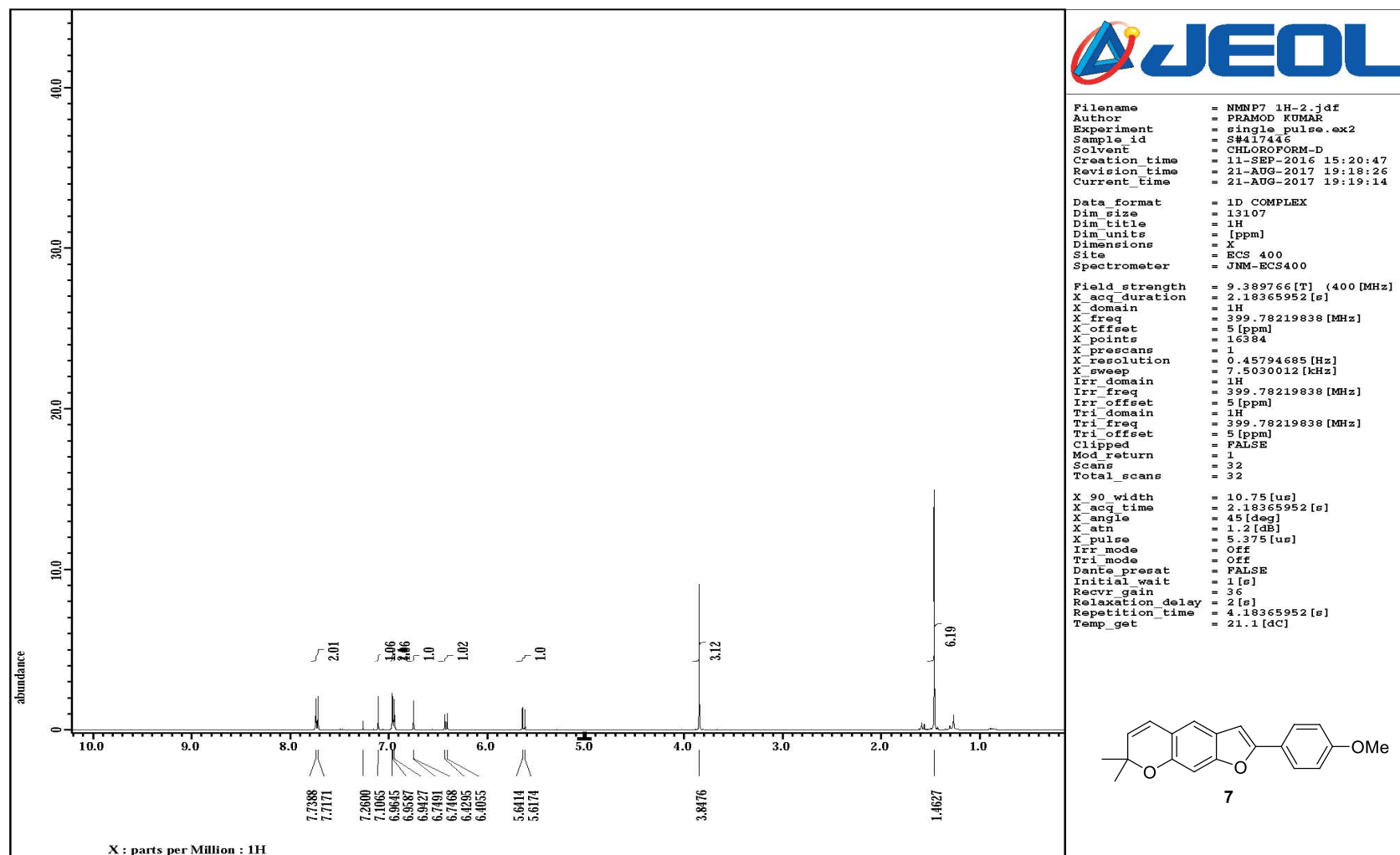
Comment      = single pulse decouple
Data format  = 1D COMPLEX
Dim size     = 26214
Dim title    = 13C
Dim units    = [ppm]
Dimensions   = X
Site         = ECX 500
Spectrometer = DELTA2_NMR

Field strength = 11.7473579 [T] (500 [MH]
X_acq_duration = 0.83361792 [s]
X_domain       = 13C
X_freq         = 125.76529768 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034 [Hz]
X_sweep        = 39.3081761 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod return     = 1
Scans          = 1000
Total scans    = 1000

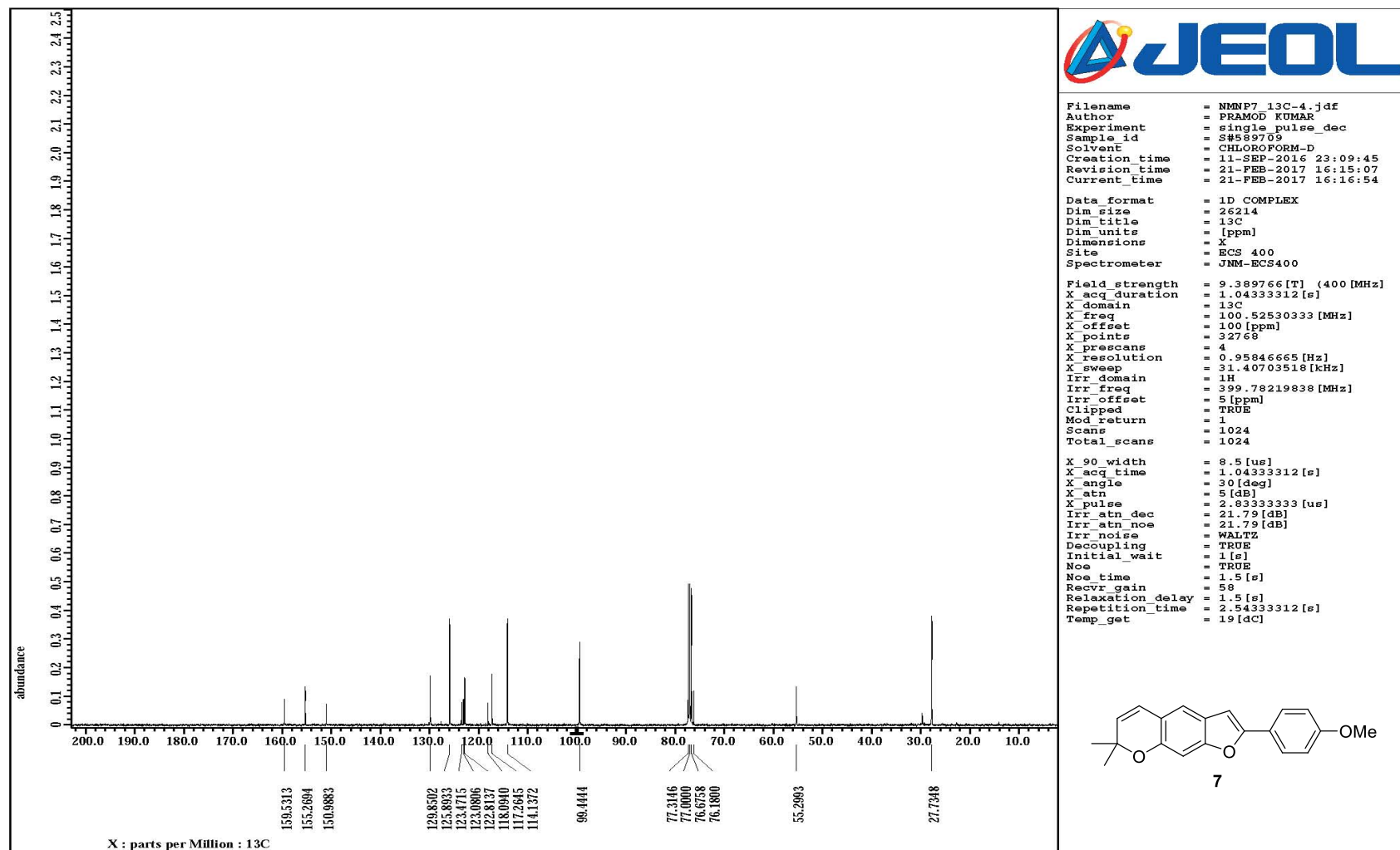
X_90_width     = 11.75 [us]
X_acq_time     = 0.83361792 [s]
X_angle        = 30 [deg]
X_atn          = 7.1 [dB]
X_pulse        = 3.91666667 [us]
Irr_atn_dec    = 19.32 [dB]
Irr_atn_noe    = 19.32 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial wait   = 1 [s]
Noe            = TRUE
Noe time       = 1 [s]
Recvr gain     = 60
Relaxation delay = 1 [s]
Repetition_time = 1.83361792 [s]
Temp_get       = 27.7 [dC]
  
```



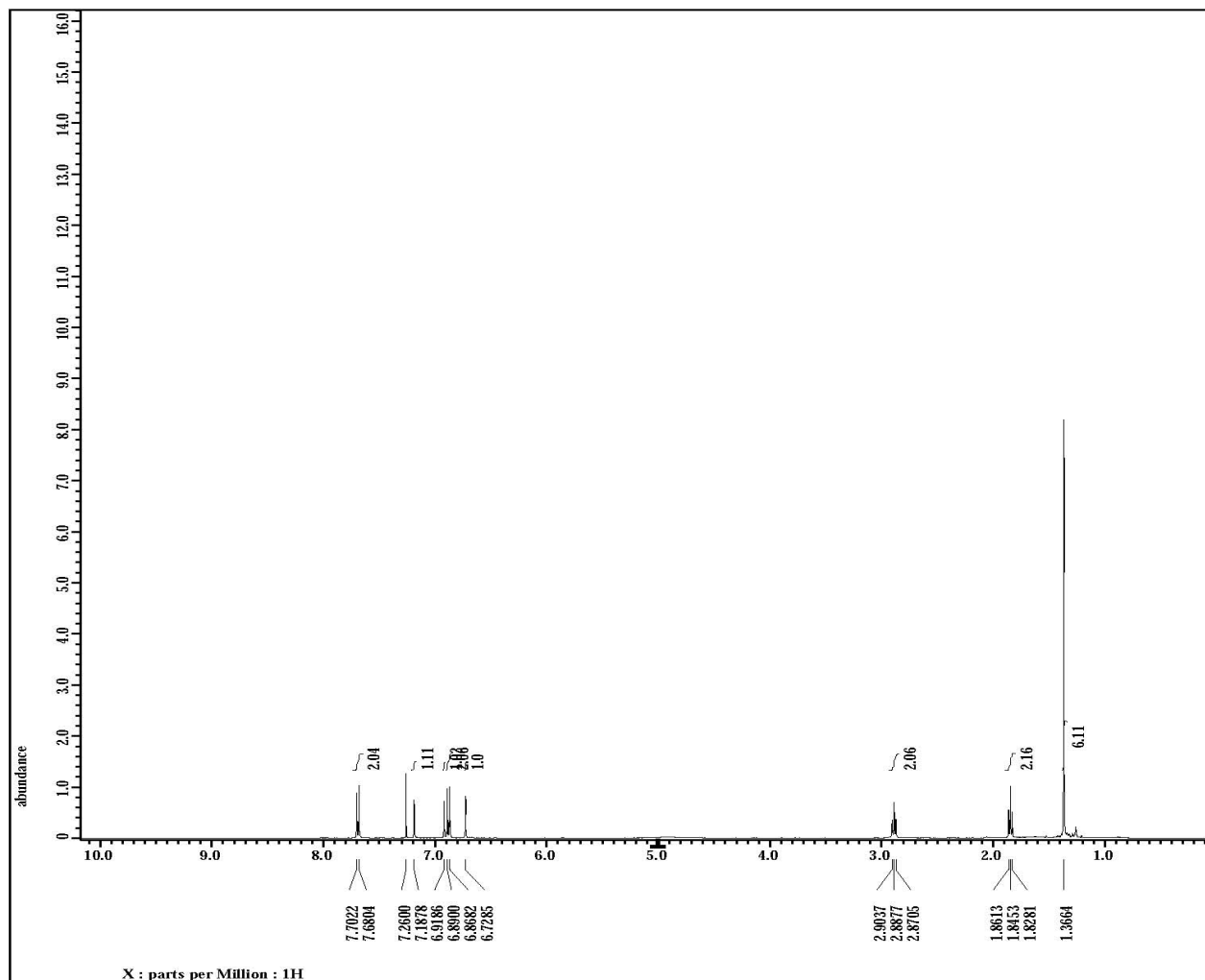
^{13}C NMR spectra of Compound 5



^1H NMR spectra of Compound 7



^{13}C NMR spectra of Compound 7

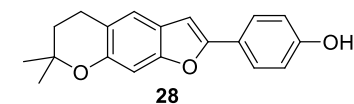


Filename = NM11223 1H-5.jdf
 Author = PRAMOD KUMAR
 Experiment = single pulse.ex2
 Sample id = S#556956
 Solvent = CHLOROFORM-D
 Creation time = 20-APR-2016 19:11:42
 Revision time = 9-FEB-2017 19:37:11
 Current Time = 9-FEB-2017 19:39:25

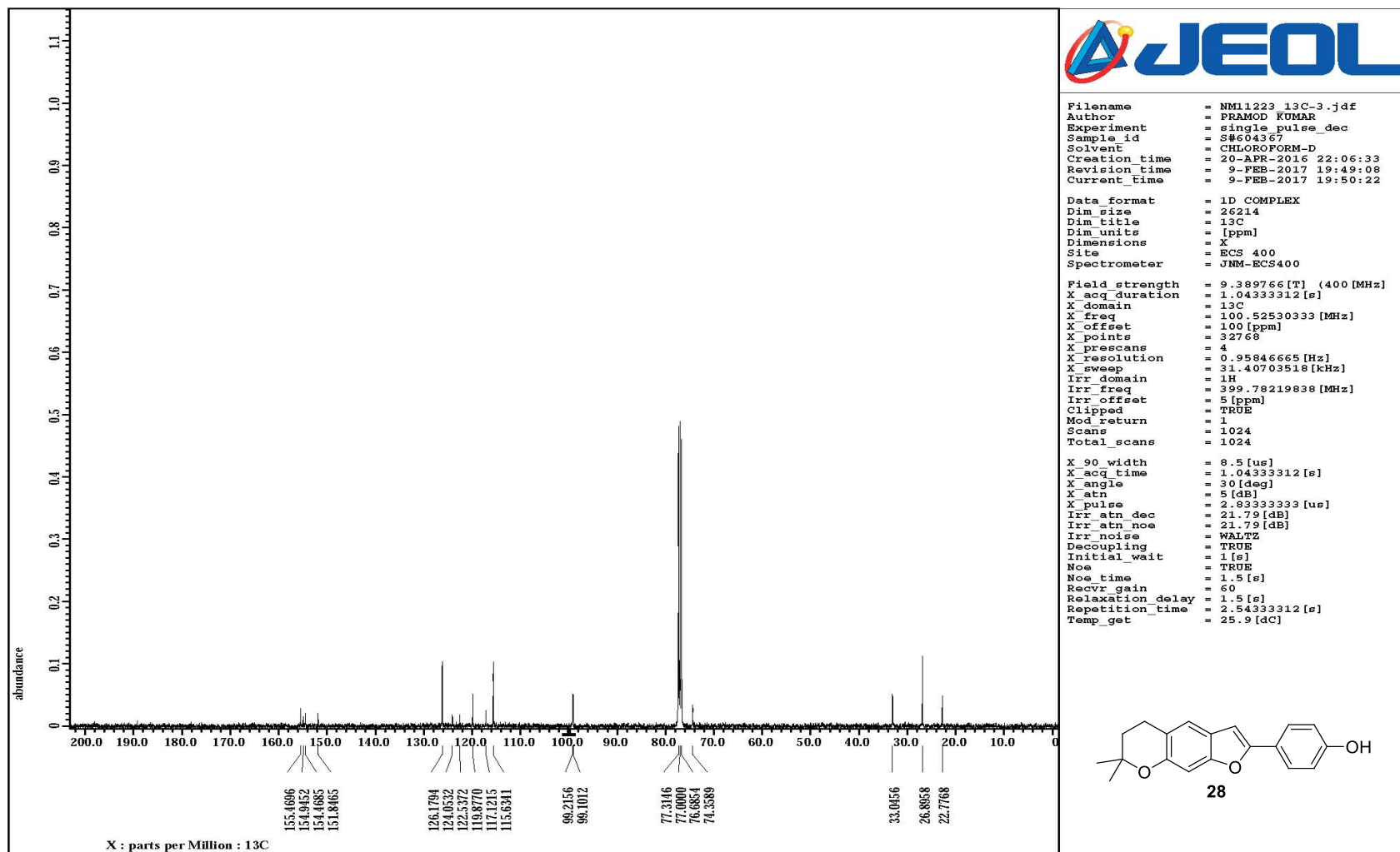
 Data format = 1D COMPLEX
 Dim size = 13107
 Dim title = 1H
 Dim units = [ppm]
 Dimensions = X
 Site = ECS 400
 Spectrometer = JNM-ECS400

 Field strength = 9.389766[T] (400 [MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 399.78219838 [MHz]
 X_offset = 5 [ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.45794685 [Hz]
 X_sweep = 7.5030012 [kHz]
 Irr_domain = 1H
 Irr_freq = 399.78219838 [MHz]
 Irr_offset = 5 [ppm]
 Tri_domain = 1H
 Tri_freq = 399.78219838 [MHz]
 Tri_offset = 5 [ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 16
 Total_scans = 16

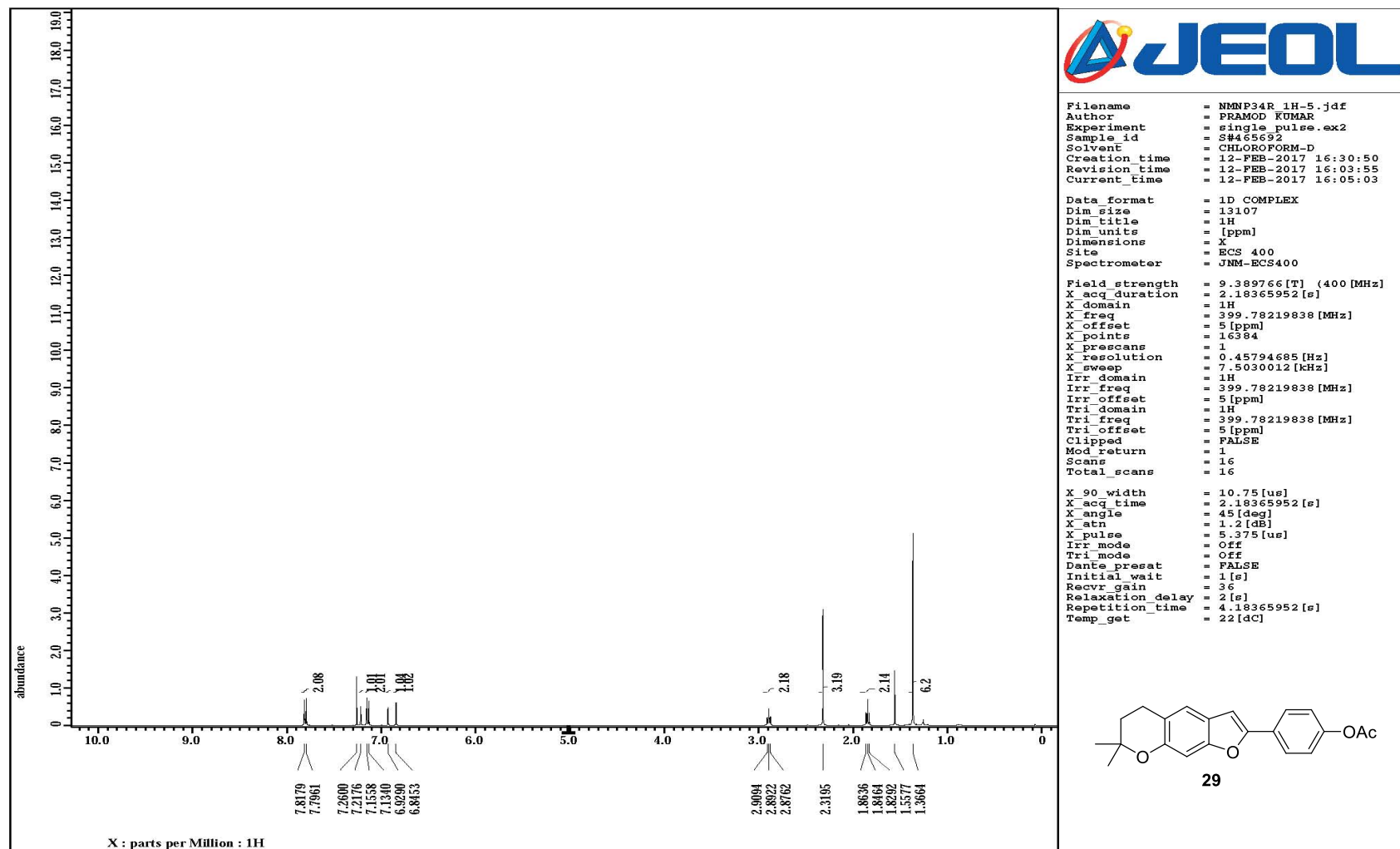
 X_90_width = 10.75[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 1.2[db]
 X_pulse = 5.375[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_preset = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 40
 Relaxation_delay = 2[s]
 Repetition_time = 4.18365952[s]
 Temp_get = 25.8 [dC]



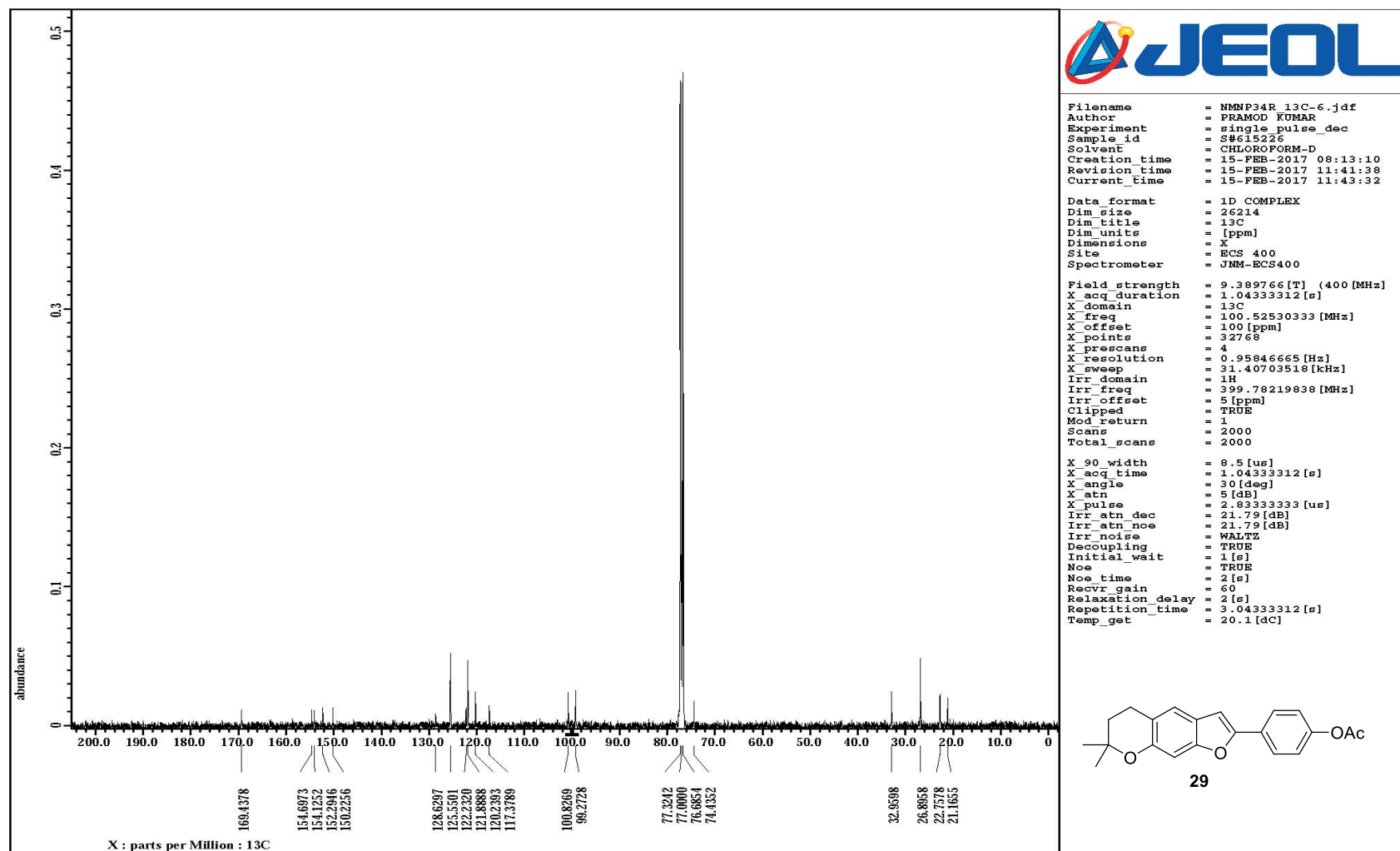
^1H NMR spectra of Compound 28



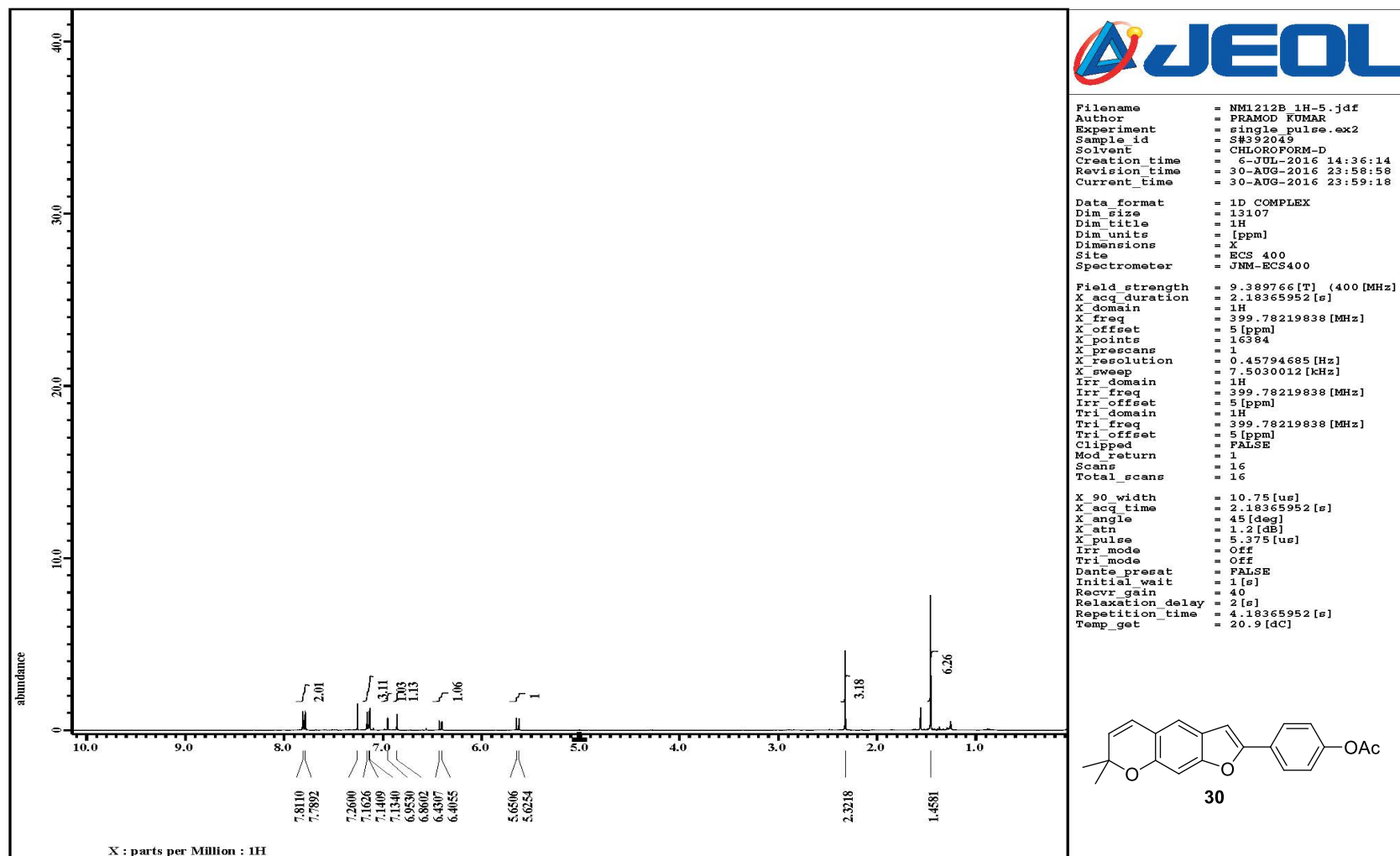
¹³C NMR spectra of Compound 28

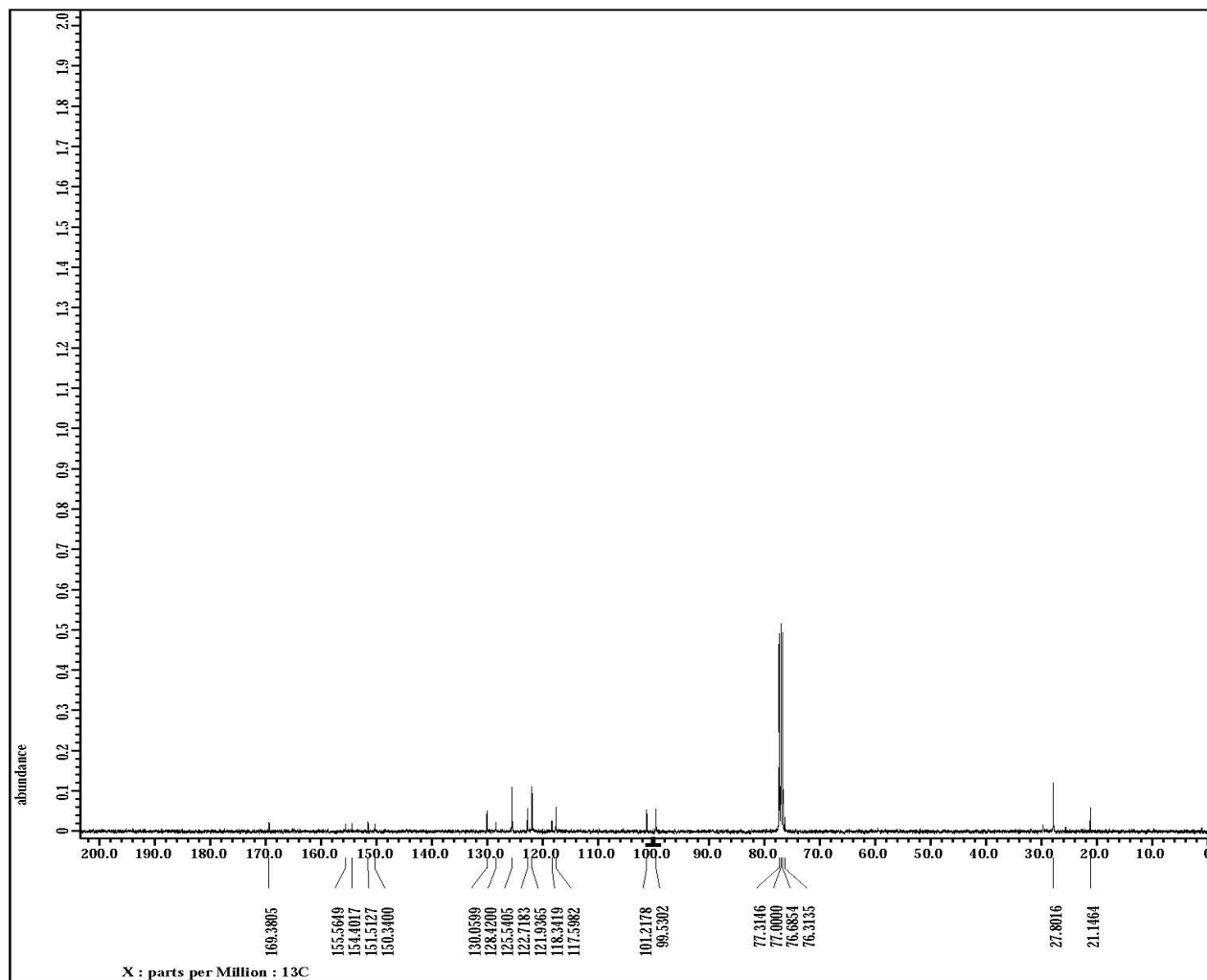


¹H NMR spectra of Compound **29**



¹³C NMR spectra of Compound 29





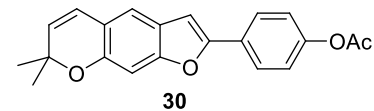
```

Filename      = NM1212B_13C-4.jdf
Author       = PRAMOD KUMAR
Experiment    = single pulse_dec
Sample_id     = S#599144
Solvent       = CHLOROFORM-D
Creation_time = 8-JUL-2016 21:00:59
Revision_time = 4-SEP-2016 16:38:06
Current_Time  = 4-SEP-2016 16:41:00

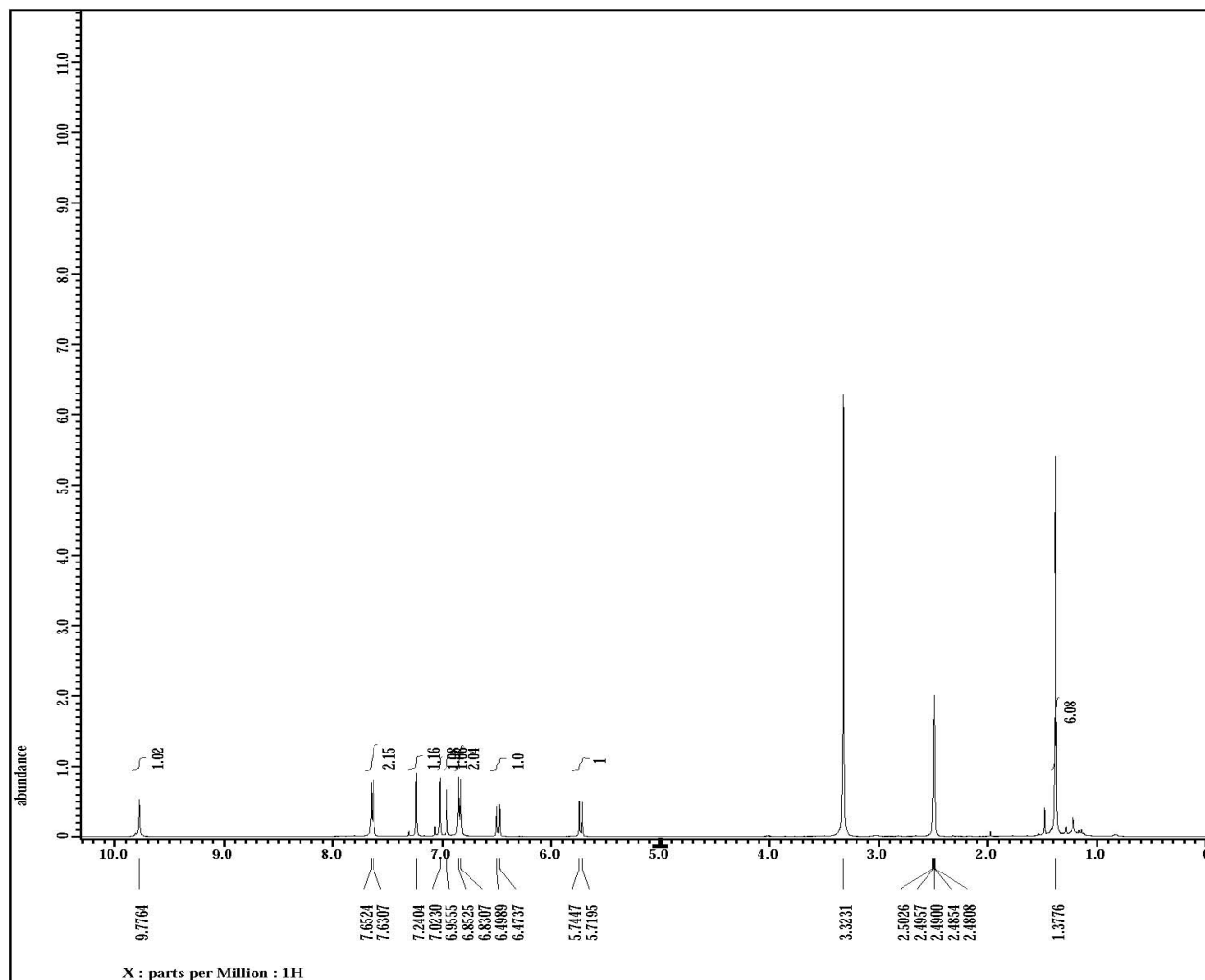
Data_format   = 1D COMPLEX
Dim_size      = 26214
Dim_title     = 13C
Dim_units     = [ppm]
Dimensions    = X
Site          = ECS 400
Spectrometer  = JNM-ECS400

Field_strength = 9.389766[T] (400 [MHz])
X_acq_duration = 1.04333312[s]
X_domain      = 13C
X_freq        = 100.52530333 [MHz]
X_offset      = 100 [ppm]
X_points      = 32768
X_prescans    = 4
X_resolution  = 0.95846665 [Hz]
X_sweep       = 31.40703518 [kHz]
Irr_domain    = 1H
Irr_freq      = 399.78219838 [MHz]
Irr_offset    = 5 [ppm]
Clipped       = TRUE
Mod_return    = 1
Scans         = 1024
Total_scans   = 1024

X_90_width    = 8.5 [us]
X_acq_time     = 1.043333312 [s]
X_angle       = 30 [deg]
X_atn         = 5 [dB]
X_pulse       = 2.83333333 [us]
Irr_atn_dec   = 21.79 [dB]
Irr_atn_noe   = 21.79 [dB]
Irr_noise     = WALTZ
Decoupling    = TRUE
Initial_wait   = 1 [s]
Noe           = TRUE
Noe_time      = 1.5 [s]
Recvr_gain    = 60
Relaxation_delay = 1.5 [s]
Repetition_time = 2.543333312 [s]
Temp_get      = 23 [dC]
  
```



^{13}C NMR spectra of Compound **30**



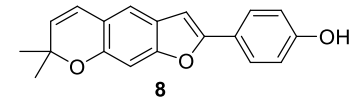
```

Filename      = NMN08R 1H-4.jdf
Author       = SUNEETA SHARMA
Experiment    = single pulse.ex2
Sample id    = S#571435
Solvent      = DMSO-D6
Creation time = 10-FEB-2017 19:11:56
Revision time = 10-FEB-2017 17:00:49
Current Time  = 10-FEB-2017 17:02:15

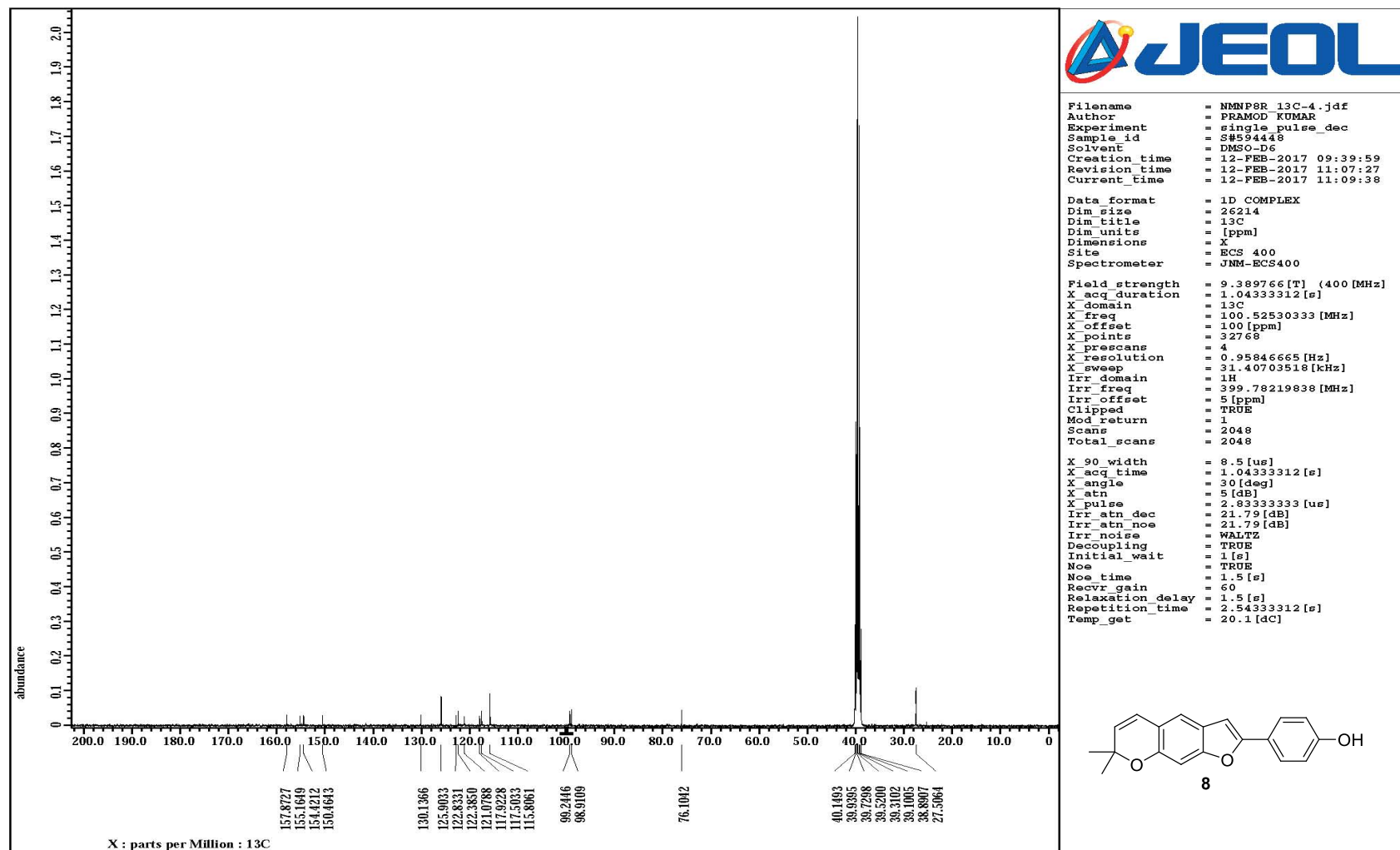
Data format   = 1D COMPLEX
Dim size      = 13107
Dim title     = 1H
Dim units     = [ppm]
Dimensions    = X
Site          = ECS 400
Spectrometer  = JNM-ECS400

Field strength = 9.2982153 [T] (400 [MHz]
X_acq duration = 2.20725248 [s]
X_domain       = 1H
X_freq         = 395.88430144 [MHz]
X_offset       = 5 [ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.45305193 [Hz]
X_sweep        = 7.42280285 [kHz]
Irr_domain     = 1H
Irr_freq       = 395.88430144 [MHz]
Irr_offset     = 5 [ppm]
Tri_domain     = 1H
Tri_freq       = 395.88430144 [MHz]
Tri_offset     = 5 [ppm]
Clipped        = FALSE
Mod return     = 1
Scans          = 34
Total_scans    = 34

X_90 width    = 10 [us]
X_acq time     = 2.20725248 [s]
X_angle        = 45 [deg]
X_atn          = 0.6 [dB]
X_pulse        = 5 [us]
Irr mode       = Off
Tri mode       = Off
Dante preset   = FALSE
Initial wait    = 1 [s]
Recvr gain     = 42
Relaxation delay = 1 [s]
Repetition_time = 3.20725248 [s]
Temp_get       = 23.2 [dC]
  
```



^1H NMR spectra of Compound 8



¹³C NMR spectra of Compound 8