Electronic Supplementary Information

Development of dichloroacetamide pyrimidines as pyruvate dehydrogenases kinase inhibitors to reduce cancer cell growth: Synthesis and biological evaluation

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1. Lactate formation was decreased by the treatment with compounds 1b and 40

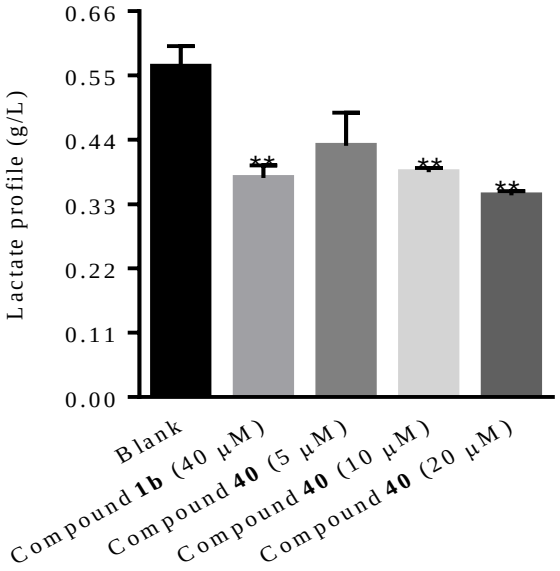

Fig S1 Lactate formation was decreased by the treatment with compounds **1b** and **40** for 24 h, * $P < 0.05$, versus blank group.

2. General methods

All the building blocks were bought from sigma, JK chemical, Wako, and Acros and were used as received. Solvents were purchased from Anaqua Chemicals Supply (ACS grade). Dimethyl sulfoxide-d_6 was bought from CIL, USA. All new compounds were characterized by ^{1}H NMR, ^{13}C NMR and HRMS. NMR spectra were recorded on a Bruker AV-400 instrument. All ^{1}H NMR spectra are reported in ppm downfield from DMSO (2.50 ppm), and ^{13}C NMR spectra are reported in ppm relative to residual DMSO (39.6 ppm). Coupling constants are reported in Hz with multiplicities denoted as s (singlet), d (doublet), t (triplet), and m (multiplet). Reactions were monitored by thin-layer chromatography (TLC) carried out on commercial silica gel plates (GF254) using UV light as a visualizing agent. High resolution mass spectra (HRMS) were obtained on a Xevo G2-XS QTof spectrometer.

*2.1 Synthesis of compounds **1a**, **1b**, and **3–5***

Compounds **1a**, **1b**, and **3**–**5** were prepared according to the literatures procedures [1, 2, 3].

*2.2 2,2-Dichloro-N-(4-chloro-6-(cyclopropylamino)pyrimidin-5-yl)acetamide (**6**)*

A mixture of compound **1a** (0.27 g, 1 mmol) and triethylamine (0.12 g, 1.2 mmol) in dimethyl sulfoxide (15 mL) was stirred under room temperature. Then cyclopropanamine (0.085 g, 1.5 mmol) in dimethyl sulfoxide (5 mL) was added dropwise. Upon completion of the reaction, the resulting mixture was extracted with dichlormethane (3 × 20 mL), then the combined organic phase was dried over anhydrous sodium sulfate and subsequently the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography (eluent, ethyl acetate / dichlormethane, 1/2, V/V) to give the compound **6** (0.22 g) as light yellow solid. Yield: 75.1 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.98 (s, 1H), 8.30 (s, 1H), 7.66 (d, J = 2.8 Hz, 1H), 6.49 (s, 1H), 2.80–2.74 (m, 1H), 0.7–0.78 (m, 2H), 0.50–0.52 (m, 2H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.58, 161.40, 156.59, 155.48, 111.77, 79.28, 67.26, 24.10, 6.49; HRMS (ESI) calcd. for $C_9H_{10}Cl_3N_4O$ $[M+H]^+$, 294.9920; found, 294.9911.

*2.3 2,2-Dichloro-N-(4-chloro-6-(cyclobutylamino)pyrimidin-5-yl)acetamide (**7**)*

Compound **7** was prepared according to the experimental procedure described for compound **6**, starting from compound **1a** (0.27 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and cyclobutanamine (0.1 g, 1.41 mmol). The desired compound **7** (0.24 g) was obtained as slight yellow solid. Yield: 77.9%; ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.99 (s, 1H), 8.23 (s, 1H), 7.58 (d, J = 7.2 Hz, 1H), 6.55 (s, 1H), 4.54–4.44 (m, 1H), 2.29–2.22 (m, 2H), 2.06–1.96 (m, 2H), 1.72–1.64 (m, 2H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.55, 159.12, 156.49, 155.70, 111.36, 67.25, 45.93, 30.17, 14.74; HRMS (ESI) calcd. for $C_{10}H_{12}Cl_3N_4O$ $[M+H]^+$, 309.0077; found, 309.0069.

*2.4 2,2-Dichloro-N-(4-chloro-6-(pyrrolidin-1-yl)pyrimidin-5-yl)acetamide (**8**)*

Compound **8** was prepared according to the experimental procedure described for compound **6**, starting from compound **1a** (0.27 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and pyrrolidine (0.1 g, 1.41 mmol). The desired compound **8** (0.26 g) was obtained as slight yellow solid. Yield: 83.9 %; ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.41 (s,

1H), 8.26 (s, 1H), 6.75 (s, 1H), 3.67 (t, *J* = 10 Hz, 2H), 3.43 (t, *J* = 10 Hz, 2H), 1.89–1.84 (m, 4H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 163.36, 158.51, 158.37, 155.91, 110.58, 66.60, 48.74, 24.86; HRMS (ESI) calcd. for $C_{10}H_{12}Cl_3N_4O$ $[M+H]^+$, 309.0077; found, 309.0083.

*2.5 2,2-Dichloro-N-(4-chloro-6-(piperidin-1-yl)pyrimidin-5-yl)acetamide (**9**)*

Compound **9** was prepared according to the experimental procedure described for compound **6**, starting from compound **1a** (0.27 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and piperidine (0.12 g, 1.5 mmol). The desired compound **9** (0.25 g) was obtained as slight yellow solid. Yield: 76.8 %; ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.46 (s, 1H), 8.30 (s, 1H), 6.74 (s, 1H), 3.63 (t, *J* = 10 Hz, 4H), 1.62–1.58 (m, 2H), 1.55–1.49 (m, 4H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.41, 160.32, 159.29, 155.53, 112.16, 66.57, 47.70, 25.83, 23.99; HRMS (ESI) calcd. for $C_{11}H_{14}Cl_3N_4O$ $[M+H]^+$, 323.0233; found, 323.0233.

*2.6 2,2-Dichloro-N-(4-chloro-6-thiomorpholinopyrimidin-5-yl)acetamide (**10**)*

Compound **10** was prepared according to the experimental procedure described for compound **6**, starting from compound **1a** (0.27 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and piperidine (0.15 g, 1.5 mmol). The desired compound **10** (0.28 g) was obtained as slight yellow solid. Yield: 81.2 %; ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.51 (s, 1H), 8.37 (s, 1H), 6.78 (s, 1H), 3.92 (t, *J* = 9.6 Hz, 4H), 2.65 (t, *J* = 9.6 Hz, 4H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.68, 160.54, 159.71, 155.66, 112.84, 66.55, 49.50, 26.58; HRMS (ESI) calcd. for $C_{10}H_{12}Cl_3N_4O$ $[M+H]^+$, 340.9797; found, 340.9789.

*2.7 2,2-Dichloro-N-(4-chloro-6-(4-methylpiperazin-1-yl)pyrimidin-5-yl)acetamide (**11**)*

Compound **11** was prepared according to the experimental procedure described for compound **6**, starting from compound **1a** (0.27 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and morpholine (0.13 g, 1.5 mmol). The desired compound **11** (0.25 g) was obtained as slight yellow solid. Yield: 78.5 %; ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.51 (s, 1H), 8.37 (s, 1H), 6.78 (s, 1H), 3.63 (s, 8H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.40, 160.39, 159.37, 155.63, 112.77, 66.54, 66.15, 46.96; HRMS (ESI) calcd. for $C_{10}H_{12}Cl_3N_4O_2$ $[M+H]^+$, 325.0026; found, 325.0015.

*2.8 2,2-Dichloro-N-(4-chloro-6-(4-methylpiperazin-1-yl)pyrimidin-5-yl)acetamide (**12**)*

Compound **12** was prepared according to the experimental procedure described for compound **6**, starting from compound **1a** (0.27 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and 1-methylpiperazine (0.15 g, 1.5 mmol). The desired compound **12** (0.27 g) was obtained as slight yellow solid. Yield: 79.4 %; ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.49 (s, 1H), 8.34 (s, 1H), 6.76 (s, 1H), 3.65 (s, 4H), 2.36–2.33 (m, 4H), 2.17 (s, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.40, 160.38, 159.37, 155.59, 112.65, 66.60, 54.60, 46.39, 45.65; HRMS (ESI) calcd. for $C_{11}H_{15}Cl_3N_5O$ $[M+H]^+$, 338.0342; found, 338.0345.

*2.9 Tert-butyl 4-(6-chloro-5-(2,2-dichloroacetamido)pyrimidin-4-yl)piperazine-1-carboxylate (**13**)*

Compound **13** was prepared according to the experimental procedure described for compound **6**, starting from compound **1a** (0.27 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and tert-butyl piperazine-1-carboxylate (0.28 g, 1.5 mmol). The desired compound **13** (0.36 g) was obtained as slight yellow solid. Yield: 84.1 %; ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.52 (s, 1H), 8.38 (s, 1H), 6.80 (s, 1H), 3.66 (t, J = 10 Hz, 4H), 3.40 (t, J = 10 Hz, 4H), 1.40 (s, 9H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.51, 160.49, 159.43, 155.63, 153.92, 112.77, 79.37, 66.60, 46.12, 43.56, 42.60, 28.13; HRMS (ESI) calcd. for $C_{15}H_{21}Cl_3N_5O_3$ $[M+H]^+$, 424.0710; found, 424.0667.

*2.10 4-(6-Chloro-5-(2,2-dichloroacetamido)pyrimidin-4-yl)piperazin-1-ium 2,2,2-trifluoroacetate (**14**)*

Compound **13** (0.21 g, 0.5 mmol) was stirred in dichloromethane, then trifluoroacetic acid (0.22 g, 2 mmol) in dichloromethane (5 mL) was added dropwise. Upon completion of the reaction, the resulting mixture was concentrated, the residues was purified by silica gel column chromatography (eluent, methanol / dichlormethane, 1/10, V/V) to give the compound **14** (0.21 g) as light yellow solid. Yield: 96.2 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.91 (s, 1H), 9.34 (s, 2H), 8.43 (s, 1H), 6.88 (s, 1H), 3.90 (m, 4H), 2.51 (m, 4H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.72, 160.52, 159.66, 155.75, 113.85, 66.64, 43.68, 42.81; HRMS (ESI) calcd. for $C_{10}H_{13}Cl_3N_5O$ $[M+H\text{-}CF_3COO^-]^+$, 324.0186; found, 324.0189.

*2.11 2,2-Dichloro-N-(4-chloro-6-(cyclopropylamino)-2-methylpyrimidin-5-yl)acetamide (**15**)*

Compound **15** was prepared according to the experimental procedure described for compound **6**, starting from compound **1b** (0.29 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and cyclopropanamine (0.085 g, 1.41 mmol). The desired compound **15** (0.27 g) was obtained as slight yellow solid. Yield: 86.4 %; ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.86 (s, 1H), 7.46 (d, J = 2.4 Hz, 1H), 6.48 (s, 1H), 2.83–2.77 (m, 1H), 2.39 (s, 3H), 0.77–0.72 (m, 2H), 0.51–0.47 (m, 2H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 165.79, 162.59, 161.15, 155.14, 109.03, 67.22, 25.52, 24.03, 6.41; HRMS (ESI) calcd. for $C_{10}H_{12}Cl_3N_4O$ $[M+H]^+$, 309.0077; found, 309.0068.

*2.12 2,2-dichloro-N-(4-chloro-6-(cyclobutylamino)-2-methylpyrimidin-5-yl)acetamide (**16**)*

Compound **16** was prepared according to the experimental procedure described for compound **6**, starting from compound **1b** (0.29 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and cyclobutanamine (0.11 g, 1.5 mmol). The desired compound **16** (0.26 g) was obtained as slight yellow solid. Yield: 83.1 %; ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.88 (s, 1H), 7.42 (d, J = 11.2 Hz, 1H), 6.53 (s, 1H), 4.56–4.46 (m, 1H), 2.35 (s, 3H), 2.24–2.21 (m, 2H), 2.05–1.95 (m, 2H), 1.71–1.63 (m, 2H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 165.71, 162.62, 158.93, 155.44, 108.64, 67.26, 45.67, 30.31, 25.48, 14.64; HRMS (ESI) calcd. for $C_{11}H_{14}Cl_3N_4O$ $[M+H]^+$, 323.0233; found, 323.0225.

*2.13 2,2-Dichloro-N-(4-chloro-2-methyl-6-(pyrrolidin-1-yl)pyrimidin-5-yl)acetamide (**17**)*

Compound **17** was prepared according to the experimental procedure described for compound **6**, starting from

compound **1b** (0.29 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and pyrrolidine (0.11 g, 1.5 mmol). The desired compound **17** (0.24 g) was obtained as slight yellow solid. Yield: 74.4 %; ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.23 (s, 1H), 6.73 (s, 1H), 3.65 (t, J = 9.6 Hz, 2H), 3.42 (t, J = 9.6 Hz, 2H), 2.36 (s, 3H), 1.87–1.81 (m, 4H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 165.05, 163.39, 158.25, 108.04, 66.63, 48.61, 25.34, 24.88; HRMS (ESI) calcd. for $C_{11}H_{14}Cl_3N_4O$ $[M+H]^+$, 323.0233; found, 323.0233.

2.14 2,2-Dichloro-N-(4-chloro-2-methyl-6-(piperidin-1-yl)pyrimidin-5-yl)acetamide ***(18)***

Compound **18** was prepared according to the experimental procedure described for compound **6**, starting from compound **1b** (0.29 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and piperidine (0.13 g, 1.5 mmol). The desired compound **18** (0.23 g) was obtained as slight yellow solid. Yield: 69.2 %; ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.34 (s, 1H), 6.72 (s, 1H), 3.61 (t, J = 9.6 Hz, 4H), 2.38 (s, 3H), 1.60–1.57 (m, 2H), 1.52–1.48 (m, 4H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 164.79, 162.48, 160.20, 159.06, 109.59, 66.60, 47.59, 25.82, 25.32, 24.02; HRMS (ESI) calcd. for $C_{12}H_{16}Cl_3N_4O$ $[M+H]^+$, 337.0390; found, 337.0394.

*2.15 2,2-Dichloro-N-(4-chloro-2-methyl-6-thiomorpholinopyrimidin-5-yl)acetamide (**19**)*

Compound **19** was prepared according to the experimental procedure described for compound **6**, starting from compound **1b** (0.29 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and thiomorpholine (0.15 g, 1.5 mmol). The desired compound **19** (0.25 g) was obtained as slight yellow solid. Yield: 72.1 %; ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.38 (s, 1H), 6.75 (s, 1H), 3.90 (t, J = 9.2 Hz, 4H), 2.64 (t, J = 9.2 Hz, 4H), 2.40 (s, 3H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 164.97, 162.70, 160.39, 159.44, 110.26, 66.56, 49.39, 26.57, 25.31; HRMS (ESI) calcd. for $C_{11}H_{14}Cl_3N_4OS$ $[M+H]^+$, 354.9954; found, 354.9951.

*2.16 2,2-Dichloro-N-(4-chloro-2-methyl-6-morpholinopyrimidin-5-yl)acetamide (**20**)*

Compound **20** was prepared according to the experimental procedure described for compound **6**, starting from compound **1b** (0.29 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and thiomorpholine (0.13 g, 1.5 mmol). The desired compound **20** (0.25 g) was obtained as slight yellow solid. Yield: 73.5 %; ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.40 (s, 1H), 6.74 (s, 1H), 3.62 (s, 8H), 2.60 (s, 3H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 164.96, 162.44, 160.22, 159.14, 110.18, 66.56, 66.15, 46.87, 25.29; HRMS (ESI) calcd. for $C_{11}H_{14}Cl_3N_4O_2$ $[M+H]^+$, 339.0182; found, 339.0178.

2.17 2,2-Dichloro-N-(4-chloro-2-methyl-6-(4-methylpiperazin-1-yl)pyrimidin-5-yl)acetamide ***(21)***

Compound **21** was prepared according to the experimental procedure described for compound **6**, starting from compound **1b** (0.29 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and thiomorpholine (0.15 g, 1.5 mmol). The desired compound **21** (0.27 g) was obtained as slight yellow solid. Yield: 77.4 %; ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.38 (s, 1H), 6.73 (s, 1H), 3.63 (s, 4H), 2.39 (s, 3H), 2.35–2.32 (m, 4H), 2.17 (s, 3H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 164.87, 162.42, 160.21, 159.14, 110.02, 66.59, 54.60, 46.27, 45.63, 25.30; HRMS (ESI) calcd. for $C_{12}H_{17}Cl_3N_5O$

$[M+H]^+$, 352.0499; found, 352.0481.

*2.18 Tert-butyl 4-(6-chloro-5-(2,2-dichloroacetamido)-2-methylpyrimidin-4-yl)piperazine-1-carboxylate (**22**)*

Compound **22** was prepared according to the experimental procedure described for compound **6**, starting from compound **1b** (0.29 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and tert-butyl piperazine-1-carboxylate (0.27 g, 1.5 mmol). The desired compound **22** (0.28 g) was obtained as slight yellow solid. Yield: 64.1 %; ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.39 (s, 1H), 6.77 (s, 1H), 3.63 (t, J = 10 Hz, 4H), 3.37 (t, J = 10 Hz, 4H), 2.40 (s, 3H), 1.40 (s, 9H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 164.93, 162.53, 160.31, 159.17, 153.88, 110.22, 79.30, 66.62, 46.03, 28.14, 28.10, 25.28; HRMS (ESI) calcd. for $C_{16}H_{23}Cl_3N_5O_3$ $[M+H]^+$, 438.0866; found, 438.0857.

*2.19 4-(6-Chloro-5-(2,2-dichloroacetamido)-2-methylpyrimidin-4-yl)piperazin-1-ium 2,2,2-trifluoroacetate (**23**)*

Compound **22** (0.22 g, 0.5 mmol) was stirred in dichloromethane, then trifluoroacetic acid (0.22 g, 2 mmol) in dichloromethane (5 mL) was added dropwise. Upon completion of the reaction, the resulting mixture was concentrated, the residues was purified by silica gel column chromatography (eluent, methanol / dichlormethane, 1/10, V/V) to give the compound **23** (0.21 g) as light yellow solid. Yield: 93.2 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.72 (s, 1H), 9.26 (s, 2H), 6.85 (s, 1H), 3.82 (s, 4H), 3.15 (s, 4H), 2.42 (s, 3H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 165.04, 162.57, 160.12, 159.22, 111.02, 66.50, 43.40, 42.62, 25.23; HRMS (ESI) calcd. for $C_{11}H_{15}Cl_3N_5O_3$ $[M + H - CF_3COO^-]^+$, 338.0342; found, 338.0347.

*2.20 N-(4,6-Bis(cyclobutylamino)pyrimidin-5-yl)-2,2-dichloroacetamide (**24**)*

A mixture of compound **1a** (0.27 g, 1 mmol) and triethylamine (0.12 g, 1.2 mmol) in dimethyl sulfoxide (15 mL) was stirred under room temperature. Then cyclobutanamine (0.15 g, 2.5 mmol) in dimethyl sulfoxide (5 mL) was added dropwise, then the reaction temperature was increased to 60 °C. Upon completion of the reaction, the resulting mixture was extracted with dichlormethane (3 × 20 mL), then the combined organic phase was dried over anhydrous sodium sulfate and subsequently the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography (eluent, ethyl acetate / dichlormethane, 1/5, V/V) to give the compound **24** (0.3 g) as white solid. Yield: 87.9 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.94 (s, 1H), 8.26 (s, 1H), 7.59 (d, J = 7.2 Hz, 2H), 6.56 (s, 1H), 4.56–4.43 (m, 2H), 2.31–2.24 (m, 4H), 2.11–1.98 (m, 4H), 1.75–1.68 (m, 4H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.57, 159.25, 155.54, 111.58, 45.95, 30.18, 14.75; HRMS (ESI) calcd. for $C_{14}H_{20}Cl_2N_5O$ $[M+H]^+$, 344.1045; found, 344.1047.

*2.21 2,2-Dichloro-N-(4,6-di(pyrrolidin-1-yl)pyrimidin-5-yl)acetamide (**25**)*

Compound **25** was prepared according to the experimental procedure described for compound **6**, starting from compound **1a** (0.27 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and pyrrolidine (0.15 g, 2.5 mmol). The desired compound **25** (0.25 g) was obtained as white solid. Yield: 73.5 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.77 (s, 1H),

7.92 (s, 1H), 6.70 (s, 1H), 3.52–3.48 (m, 4H), 3.43–3.90 (m, 4H), 1.87–1.83 (m, 4H), 1.76–1.71 (m, 4H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.58, 159.26, 154.81, 94.51, 66.91, 48.81, 25.09; HRMS (ESI) calcd. for $C_{14}H_{20}Cl_2N_5O$ $[M+H]^+$, 344.1045; found, 344.1051.

*2.22 2,2-Dichloro-N-(4,6-di(piperidin-1-yl)pyrimidin-5-yl)acetamide (**26**)*

Compound **26** was prepared according to the experimental procedure described for compound **6**, starting from compound **1a** (0.27 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and pyrrolidine (0.21 g, 2.5 mmol). The desired compound **25** (0.28 g) was obtained as white solid. Yield: 74.9 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.68 (s, 1H), 8.09 (s, 1H), 6.64 (s, 1H), 3.37–3.40 (m, 8H), 1.54–1.50 (m, 12H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 163.51, 161.16, 155.09, 101.32, 66.89, 48.76, 25.83, 24.23; HRMS (ESI) calcd. for $C_{16}H_{24}Cl_2N_5O$ $[M+H]^+$, 372.1358; found, 372.1360.

*2.23 2,2-Dichloro-N-(4,6-dithiomorpholinopyrimidin-5-yl)acetamide (**27**)*

Compound **27** was prepared according to the experimental procedure described for compound **6**, starting from compound **1a** (0.27 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and thiomorpholine (0.25 g, 2.5 mmol). The desired compound **27** (0.28 g) was obtained as white solid. Yield: 69.2 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.82 (s, 1H), 8.17 (s, 1H), 6.73 (s, 1H), 3.67 (t, J = 10 Hz, 8H), 2.61–2.56 (m, 4H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 163.58, 161.43, 154.94, 102.22, 66.83, 50.42, 26.75; HRMS (ESI) calcd. for $C_{14}H_{20}Cl_2N_5OS_2$ $[M+H]^+$, 408.0486; found, 408.0486.

*2.24 2,2-Dichloro-N-(4,6-dimorpholinopyrimidin-5-yl)acetamide (**28**)*

Compound **28** was prepared according to the experimental procedure described for compound **6**, starting from compound **1a** (0.27 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and morpholine (0.22 g, 2.5 mmol). The desired compound **28** (0.32 g) was obtained as white solid. Yield: 84.3 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.78 (s, 1H), 8.18 (s, 1H), 6.70 (s, 1H), 3.60 (t, J = 7.2 Hz, 8H), 3.41 (m, 8H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 163.14, 161.33, 155.24, 102.19, 66.91, 66.30, 48.09; HRMS (ESI) calcd. for $C_{14}H_{20}Cl_2N_5O_3$ $[M+H]^+$, 376.0943; found, 376.0941.

*2.25 N-(4,6-bis(4-methylpiperazin-1-yl)pyrimidin-5-yl)-2,2-dichloroacetamide (**29**)*

Compound **29** was prepared according to the experimental procedure described for compound **6**, starting from compound **1a** (0.27 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and 1-methylpiperazine (0.25 g, 2.5 mmol). The desired compound **29** (0.33 g) was obtained as white solid. Yield: 82.7 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.85 (s, 1H), 8.12 (s, 1H), 6.71 (s, 1H), 3.41 (s, 8H), 2.33 (s, 8H), 2.17 (s, 6H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 163.16, 161.25, 155.09, 101.68, 66.90, 54.77, 47.38, 45.78; HRMS (ESI) calcd. for $C_{16}H_{26}Cl_2N_7O$ $[M+H]^+$, 402.1576; found, 402.1534.

*2.26 Di-tert-butyl 4,4'-(5-(2,2-dichloroacetamido)pyrimidine-4,6-diyl)bis(piperazine-1-carboxylate) (**30**)*

Compound **30** was prepared according to the experimental procedure described for compound **6**, starting from compound **1a** (0.27 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and tert-butyl piperazine-1-carboxylate (0.49 g, 2.5 mmol). The desired compound **30** (0.35 g) was obtained as white solid. Yield: 61.5 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.81 (s, 1H), 8.18 (s, 1H), 6.74 (s, 1H), 3.40 (s, 16H), 1.40 (s, 18H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 163.19, 161.38, 155.16, 153.97, 102.20, 79.20, 66.94, 47.31, 28.13; HRMS (ESI) calcd. for $C_{24}H_{38}Cl_2N_7O_5$ $[M+H]^+$, 574.2311; found, 574.2289.

*2.27 N-(4,6-Bis(cyclobutylamino)-2-methylpyrimidin-5-yl)-2,2-dichloroacetamide (**31**)*

A mixture of compound **1b** (0.29 g, 1 mmol) and triethylamine (0.12 g, 1.2 mmol) in dimethyl sulfoxide (15 mL) was stirred under room temperature. Then cyclobutanamine (0.15 g, 2.5 mmol) in dimethyl sulfoxide (5 mL) was added dropwise, then the reaction temperature was increased to 60 ºC. Upon completion of the reaction, the resulting mixture was extracted with dichlormethane (3 × 20 mL), then the combined organic phase was dried over anhydrous sodium sulfate and subsequently the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography (eluent, ethyl acetate / dichlormethane, 1/5, V/V) to give the compound **31** (0.22 g) as white solid. Yield: 60.7 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.89 (s, 1H), 8.26 (s, 1H), 7.44 (d, J = 7.2 Hz, 2H), 6.54 (s, 1H), 4.57–4.50 (m, 2H), 2.35 (s, 3H), 2.25–2.22 (m, 4H), 2.02–1.98 (m, 4H), 1.70–1.67 (m, 4H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 165.78, 162.81, 158.91, 108.64, 67.29, 45.84, 30.32, 25.51, 14.71; HRMS (ESI) calcd. for $C_{15}H_{22}Cl_2N_5O$ $[M+H]^+$, 358.1201; found, 358.1207.

*2.28 2,2-Dichloro-N-(2-methyl-4,6-di(pyrrolidin-1-yl)pyrimidin-5-yl)acetamide (**32**)*

Compound **32** was prepared according to the experimental procedure described for compound **6**, starting from compound **1b** (0.29 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and pyrrolidine (0.17 g, 2.5 mmol). The desired compound **32** (0.26 g) was obtained as white solid. Yield: 72.4 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.68 (s, 1H), 6.67 (s, 1H), 3.49–3.45 (m, 4H), 3.42–3.38 (m, 4H), 2.20 (s, 3H), 1.85–1.79 (m, 4H), 1.76–1.65 (m, 4H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.88, 162.56, 159.40, 92.61, 66.94, 48.75, 25.93, 25.10; HRMS (ESI) calcd. for $C_{15}H_{22}Cl_2N_5O$ $[M+H]^+$, 358.1201; found, 358.1195.

*2.29 2,2-Dichloro-N-(2-methyl-4,6-di(piperidin-1-yl)pyrimidin-5-yl)acetamide (**33**)*

Compound **33** was prepared according to the experimental procedure described for compound **6**, starting from compound **1b** (0.29 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and piperidine (0.21 g, 2.5 mmol). The desired compound **33** (0.3 g) was obtained as white solid. Yield: 79.1 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.57 (s, 1H), 6.61 (s, 1H), 3.35–3.31 (m, 8H), 2.26 (s, 3H), 1.51–1.49 (m, 12H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 163.62, 163.57, 161.27, 99.22, 66.93, 48.79, 25.95, 25.88, 24.31; HRMS (ESI) calcd. for $C_{17}H_{26}Cl_2N_5O$ $[M+H]^+$, 386.1514; found, 386.1523.

*2.30 2,2-Dichloro-N-(2-methyl-4,6-dithiomorpholinopyrimidin-5-yl)acetamide (**34**)*

Compound **34** was prepared according to the experimental procedure described for compound **6**, starting from compound **1b** (0.29 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and thiomorpholine (0.25 g, 2.5 mmol). The desired compound **34** (0.32 g) was obtained as white solid. Yield: 76.2 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.67 (s, 1H), 6.69 (s, 1H), 3.62 (s, 8H), 2.60 (s, 8H), 2.30 (s, 3H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 163.85, 163.82, 161.43, 100.18, 66.89, 50.41, 26.80, 25.86; HRMS (ESI) calcd. for $C_{15}H_{22}Cl_2N_5OS_2$ $[M+H]^+$, 422.0643; found, 422.0651.

*2.31 2,2-Dichloro-N-(2-methyl-4,6-dimorpholinopyrimidin-5-yl)acetamide (**35**)*

Compound **35** was prepared according to the experimental procedure described for compound **6**, starting from compound **1b** (0.29 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and morpholine (0.21 g, 2.5 mmol). The desired compound **35** (0.31 g) was obtained as white solid. Yield: 80.6 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.68 (s, 1H), 6.67 (s, 1H), 3.59 (d, J = 4 Hz, 8H), 3.36 (s, 8H), 2.30 (s, 3H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 163.90, 163.19, 161.41, 100.00, 66.94, 66.36, 48.12, 25.87; HRMS (ESI) calcd. for $C_{15}H_{22}Cl_2N_5O_3$ $[M+H]^+$, 390.1100; found, 390.1095.

*2.32 2,2-dichloro-N-(2-methyl-4,6-bis(4-methylpiperazin-1-yl)pyrimidin-5-yl)acetamide (**36**)*

Compound **36** was prepared according to the experimental procedure described for compound **6**, starting from compound **1b** (0.29 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and 1-methylpiperazine (0.25 g, 2.5 mmol). The desired compound **36** (0.31 g) was obtained as white solid. Yield: 73.8 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.72 (s, 1H), 6.67 (s, 1H), 3.59 (d, J = 4 Hz, 8H), 3.41 (s, 8H), 2.32 (s, 8H), 2.29 (s, 3H), 2.17 (s, 6H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 163.69, 163.19, 161.33, 99.48, 66.94, 54.81, 47.35, 45.78, 25.89; HRMS (ESI) calcd. for $C_{17}H_{28}Cl_2N_7O$ $[M+H]^+$, 416.1732; found, 416.1721.

*2.33 Di-tert-butyl 4,4'-(5-(2,2-dichloroacetamido)-2-methylpyrimidine-4,6-diyl)bis(piperazine-1-carboxylate) (**37**)*

Compound **37** was prepared according to the experimental procedure described for compound **6**, starting from compound **1b** (0.29 g, 1 mmol), triethylamine (0.12 g, 1.2 mmol) and tert-butyl piperazine-1-carboxylate (0.47 g, 2.5 mmol). The desired compound **37** (0.41 g) was obtained as white solid. Yield: 69.5 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.70 (s, 1H), 6.71 (s, 1H), 3.29 (s, 8H), 2.35 (s, 8H), 2.30 (s, 3H), 1.38 (s, 18H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 163.84, 163.24, 161.44, 154.00, 100.08, 79.17, 66.97, 47.33, 28.14, 25.85; HRMS (ESI) calcd. for $C_{25}H_{40}Cl_2N_7O_5$ $[M+H]^+$, 588.2468; found, 588.2477.

*2.34 4,4'-(5-(2,2-Dichloroacetamido)-2-methylpyrimidine-4,6-diyl)bis(piperazin-1-ium) 2,2,2-trifluoroacetate (**38**)*

Compound **37** (0.29 g, 0.5 mmol) was stirred in dichloromethane, then trifluoroacetic acid (0.22 g, 2 mmol) in dichloromethane (5 mL) was added dropwise. Upon completion of the reaction, the resulting mixture was concentrated, the residues was purified by silica gel column chromatography (eluent, methanol / dichlormethane, 1/10, V/V) to give the compound **38** (0.27 g) as light yellow solid. Yield: 89.4 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.08 (s, 1H), 9.01 (s, 4H), 6.85 (s, 1H), 3.60 (s, 8H), 3.12-3.09 (m, 8H), 2.35 (s, 3H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6):

δ 164.15, 162.75, 161.64, 101.38, 66.94, 44.58, 42.93, 25.81; HRMS (ESI) calcd. for $C_{15}H_{24}F_3Cl_2N_7O$ $[M+H-2CF_3COO^-]^+$, 388.1419; found, 388.1432.

*2.35 N-(4,6-Bis(4-(2-hydroxyethyl)piperazin-1-yl)-2-methylpyrimidin-5-yl)-2,2-dichloroacetamide (**39**)*

Compound **38** (0.31 g, 0.5 mmol) and anhydrous potassium carbonate (0.28 g, 2 mmol) were stirred in acetonitrile for 0.5 h. Then 2-bromoethan-1-ol (0.15 g, 1.2 mmol) was added dropwise at room temperature. After completion of the reaction, the resulting mixture was concentrated, the residues was purified by silica gel column chromatography (eluent, methanol / dichlormethane, 1/15, V/V) to give the compound **39** (0.15 g) as white solid. Yield: 64.7 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.65 (s, 1H), 6.68 (s, 1H), 3.94 (m, 4H), 3.68 (m, 4H), 3.31 (m, 8H), 3.25 (s, 1H), 2.34 (m, 8H), 2.27 (s, 3H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 163.89, 163.25, 161.52, 101.62, 66.94, 61.45, 54.97, 47.45, 45.95, 25.83; HRMS (ESI) calcd. for $C_{19}H_{32}Cl_2N_7O_3$ $[M+H]^+$, 476.1944; found, 476.1916.

*2.36 N-(4,6-Bis(4-(2-hydroxyacetyl)piperazin-1-yl)-2-methylpyrimidin-5-yl)-2,2-dichloroacetamide (**40**)*

Compound **38** (0.31 g, 0.5 mmol) and triethylamine (0.73 mL, 5 mmol) were stirred in dichloromethane for 0.5 h. Then glycolic acid (0.16 g, 2 mmol) and two drops of DMF were dissolved in dichloromethane, oxalyl chloride (0.2 mL, 2.4 mmol) was added. After 2 h, mix the two reactants in room temperature. After completion of the reaction, the resulting mixture was concentrated, the residues was purified by silica gel column chromatography (eluent, methanol / dichlormethane, 1/15, V/V) to give the compound **40** (65 mg) as white solid. Yield: 24.7 %. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.66 (s, 1H), 6.68 (s, 1H), 4.31 (s, 4H), 3.32 (m, 8H), 2.66 (m, 8H), 2.27 (s, 3H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 174.2, 163.84, 163.33, 161.51, 101.63, 66.94, 54.94, 47.44, 25.83; HRMS (ESI) calcd. for $C_{19}H_{28}Cl_2N_7O_5$ $[M+H]^+$, 504.1529; found, 504.1533.

3. Biological evaluation

3.1 Cell culture

SF188, RKO, and MCF-7 cancer cells were cultured in DMEM medium supplemented with 10% fetal bovine serum and 1% penicillin/streptomycin in 5% CO_2 at 37 °C. Cells at exponentially growing stage were used for all the experiments.

3.2 Cell viability assay

Cancer cells viability upon treatment was assessed by Alamar Blue assay. A suspension of cancer cells (2000/well for MCF-7, 3000/well for SF188 and RKO) were seeded in 96-well plates and cultured overnight. Working compound solution (400 μM) were prepared by diluting the 40 mM compounds stock solutions in PBS buffer containing 1% DMSO. The working solutions were added to the corresponding plates (10 μL/well), in which cells were incubated for 72 h. Then, 10 μL of Alamar Blue solution (10 ×) was added to each well. After 4 h of incubation at 37 °C, the solvent was mixed with gentle shaking. Fluorescence of the solution was recorded around 590 nm with an excitation wavelength at 530 nm on a SpectraMax M5 Microplate Reader. After the potent compounds (**1b**, **37** and **40**) were identified, dose-response studies were undertaken to determine the exact IC_{50} values of these compounds. Eight different concentrations of **1b**, **37** and **40** were added into the 96-well plate. The fluorescence intensity were read, and the results were expressed as IC_{50} values, which were the mean values derived from three independent experiments.

3.3 IncuCyte Zoom live cells image for the growth of the cancer cells

The growth of the cancer cells after treating with compound **1b** and **40** was monitored in an IncuCyte Zoom system (Essen BioScience Company). The SF188 (3000/well) was seeded into each well of a 96-well plate for 24 h, then compound **40** at 40 μM were added into each well, followed by another 24, 48, and 72 h incubation. Cell images were recorded every three hours, and cells without adding others drugs were served as control.

3.4 Apoptosis detection by flow cytometry

SF188 cells were seeded at a density of 2×10^5 cells/mL on each well of a six-well plate and were allowed to grow overnight. Then cells were treated with compound **1b** (40 μM), and compound **40** (10, 20, and 40 μM) for 24 h at 37 °C. Cells were trypsinized, repeatedly washed with cold PBS for three times, and centrifuged at 800 rpm for 5 min, and the supernatants were discarded. Cells were resuspended in 1× Annexin binding buffer to ~5×10^5 cells/mL, preparing a sufficient volume to have 100 μL per assay. To 100 μL of cell suspension, 10 μL of Annexin V and 5 μL of PI were added and incubated for 15 min at room temperature. After incubation, 400 μL of PBS was added to each sample and was gently mixed and analyzed immediately on a BD Accuri™ C6 Plus flow cytometer.

3.5 Lactate measurement

A suspension of SF188 cells (2×10^5 / well) were seeded in 6-well plates and cultured overnight. Then compound **1b** (40 μM), and compound **40** (5, 10, 20 μM) were added to the corresponding wells, and the plates were incubated for

24 h. Then, the medium was transfered to microcentrifuge, which were centrifuged for 5 min (6000 r/min). At last 1 mL medium was collected, the lactate production was evaluated by Nova Bioprofile Flex analyzer. (Nova Biomedical).

3.6 PDK1 expression and purification

Recombinant human PDK1 (aa29−436) was prepared as we described in our previous work [1].

3.7 ITC Analysis

The concentration of PDK1 proteins were determined by measuring sample absorbance at 280 nm and diluted to 20 μM in the buffer solution containing 50 mM KH_2PO_4/K_2HPO_4, pH 7.4, 250 mM KCl, and 2 mM $MgCl_2$. Stock solution of the compound **40** was dissolved in DMSO (30 mM). Then the stock solution was diluted by buffer solution (50 mM KH_2PO_4/K_2HPO_4, pH 7.4, 250 mM KCl, 2 mM $MgCl_2$) into the 300 μM containing 1% DMSO. Then the corresponding solution was delivered into the titration syringe. ITC experiments were accomplished using a Microcal iTC200 microcalorimeter (GE Healthcare). The reaction cell contained 300 μL PDK1. Titrations were performed with the injection of 2.5 μL titrant(s) for every increment into the reaction cell which was maintained at 25 ℃. All of the ITC data were initially analyzed and the baseline was automatically determined by the Origin 7, then followed by curve fitting to one-site model to obtain K_d value.

3.8 PDK kinase activity assay

PDK1 kinase activity was measured by the Kinase-Glo® Luminescent Kinase Assays (Promega). Reagents were used in this assay: PDK1 250 μM (in 50 mM K_3PO_4, 250 mM KCl, 2 mM $MgCl_2$, pH = 7.4); Assay buffer (10 X, 250 mM Tris-HCl, 10 mM EDTA, 5 mM EGTA, 10 mM DTT, 50 mM $MgCl_2$), peptide 10 mM (amino acid sequences: RYHGHSMSDP, which is a fragment around S293 of PDC E1 subunit), ATP 10 mM. Firstly, 25 μL of dd water and 5 μL assay buffer (10 x) were added to 96-well plate, and marked the wells as well 1#, 2#, 3#, 4#, 5# and 6#. Then 5 μL of 1% DMSO PBS buffer was added to wells 1# and 6#, and 5 μL of compound **1b** (400 μM), 5 μL of compound **40** (400 μM), 5 μL of compound **40** (200 μM), 5 μL of compound **40** (100 μM) were added to wells 2#, 3#, 4#, and 5#. Subsequently, 5 μL ATP (20 μM) and 5 μL peptide (50 μM) were added to wells 1#, 2#, 3#, 4#, 5# and 6#, then 5 μL PDK1 (20 μM) was added to wells 1#, 2#, 3#, 4# and 5#, and 5 μL protein buffer to well 6#. At last, mix the plate and incubate at 37 ºC for 30 minutes. Then to each wells 50 μL of the appropriate kinase-Glo reagent were added. The plate was shake gently, and incubated for another 10 min at room temperature. Then the luminescence was recorded in the microplate reader.

3.9 Molecule docking

The crystal structure of PDK2 (PDB ID: 2BU2) was downloaded from the Protein Data Bank (http://www.pdb.org/), the protein was prepared in Protein Preparation Workflow of Sybyl-X 2.1 software package. Protocol of the preparation of the protein includes: Water molecules removing, side-chain reparation, termini treatment, hydrogens addition, *etc*. The structures of compound **40** were drawn using the structure drawing tool available in Sybyl-X 2.1

package and the energy of molecule was minimized using a tripos force field and charges were computed using a Gasteiger–Huckel method. The lowest binding energy conformer was searched out of 20 conformers for each docking simulation and the resultant one was used for further analysis.

References

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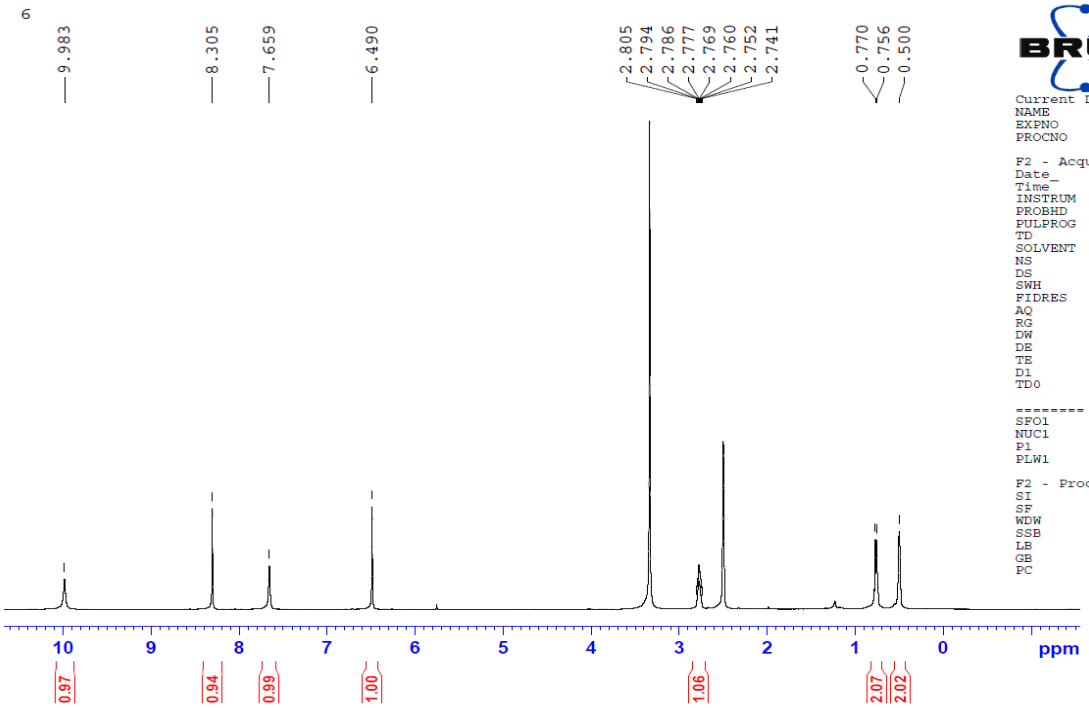
2. M. H. Norman, N. Chen, Z. D. Chen, C. Fotsch, C. Hale, N. Han, R. Hurt, T. Jenkins, J. Kincaid, L. B. Liu, Y. L. Lu, O. Moreno, V. J. Santora, J. D. Sonnenberg and W. Karbon, *J. Med. Chem.*, 2000, **43**, 4288.
3. N. Baindur, N. Chadha and M. R. Player, *J. Comb. Chem.*, 2003, **5**, 653.

4. NMR spectra

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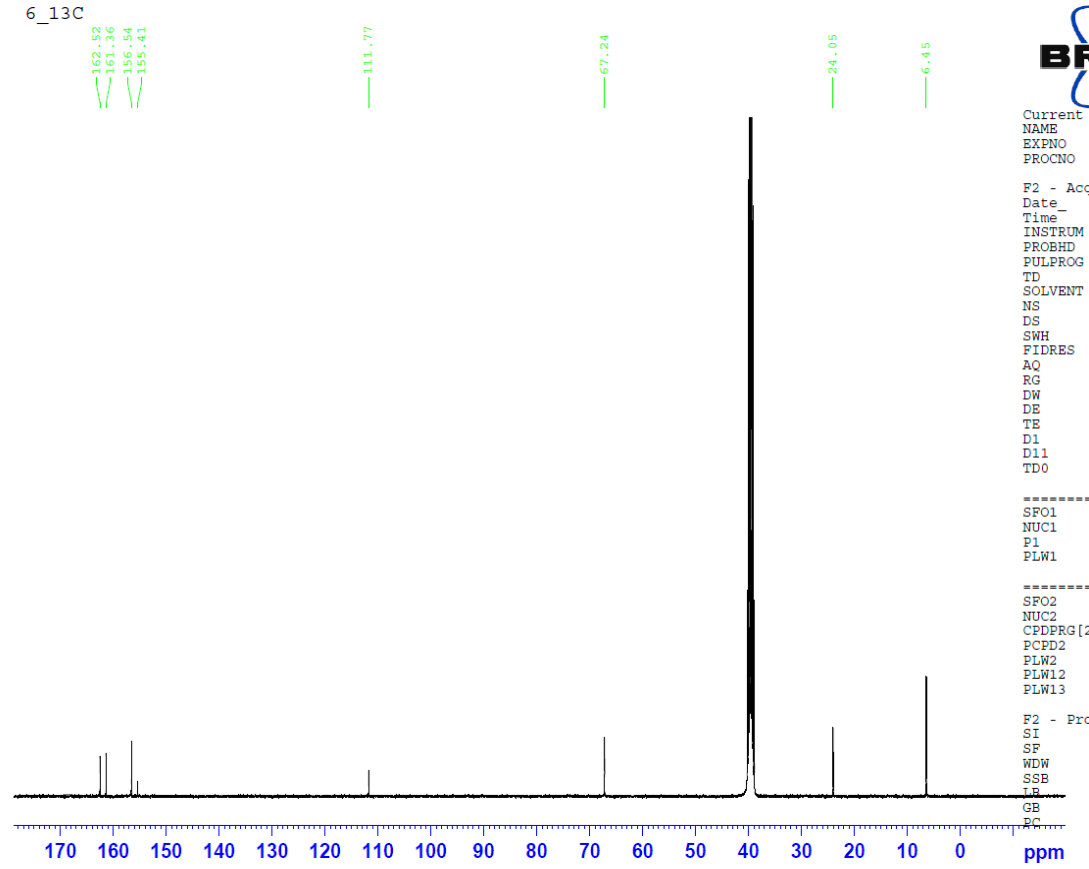


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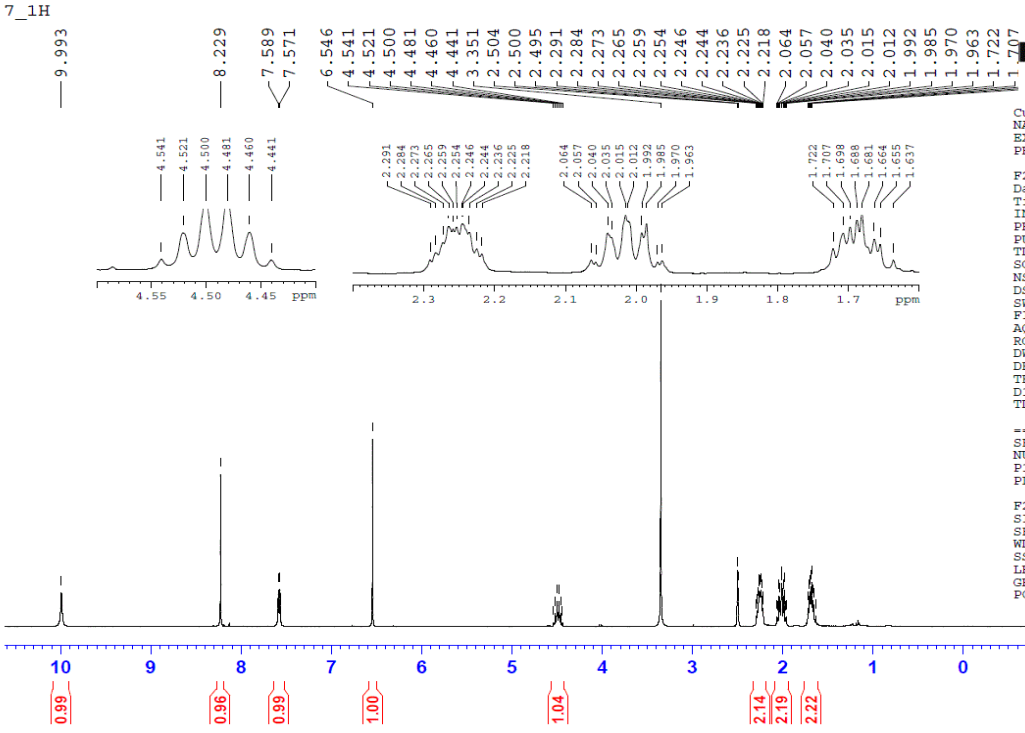

BRUKER

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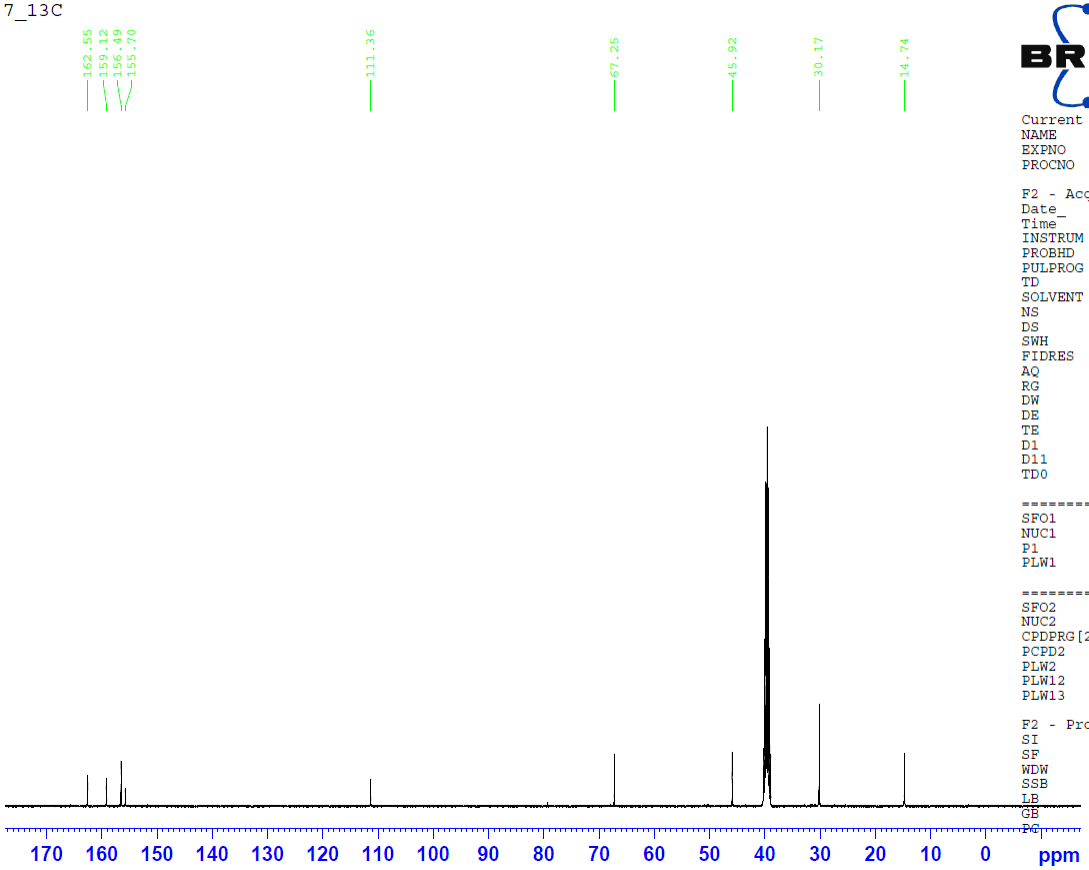

BRUKER

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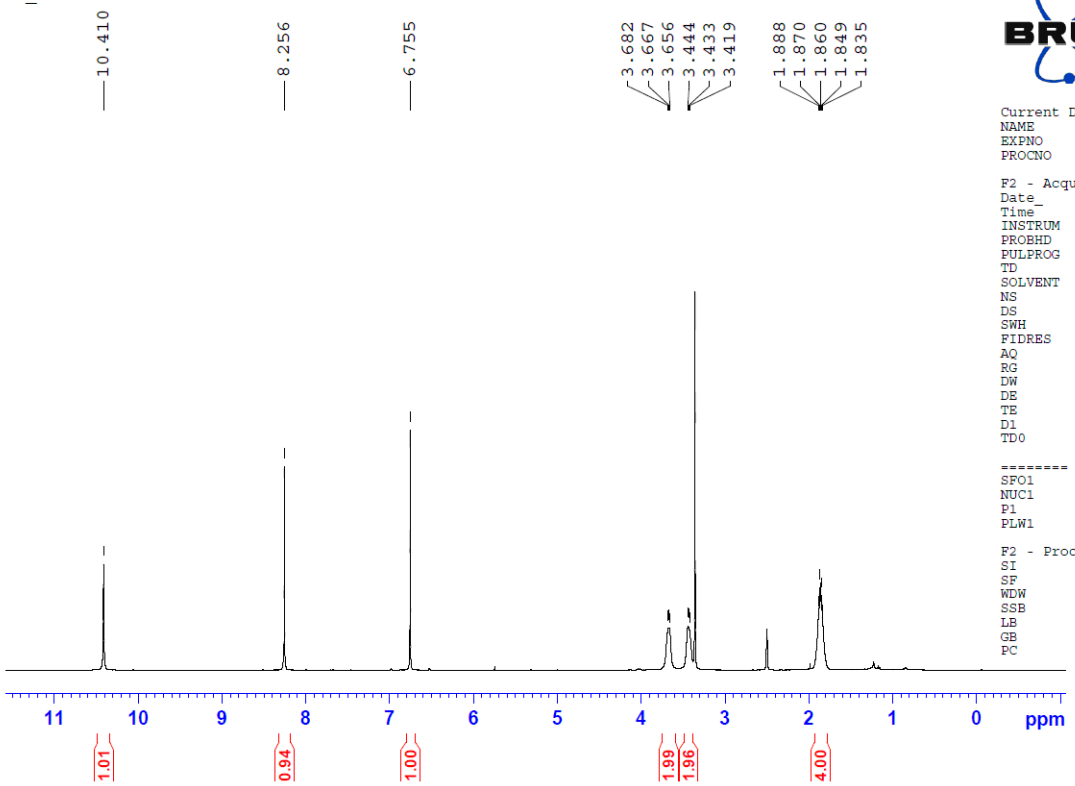
AQ                     1.3631488 sec
RG                         199.94
DW                        20.800 usec
DE                         6.50 usec
TE                        295.0 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                 100.6228293 MHz
NUC1                         13C
P1                         9.50 usec
PLW1                66.50000000 W

======== CHANNEL f2 ========
SFO2                 400.1316005 MHz
NUC2                         1H
CPDPRG[2                 waltz16
PCPD2                     90.00 usec
PLW2                18.00000000 W
PLW12                0.20056000 W
PLW13                0.16245000 W

F2 - Processing parameters
SI                           32768
SF                   100.6128046 MHz
WDW                             EM
SSB                  0
LB                         1.00 Hz
GB                   0
PC                         1.40
```

8_1H



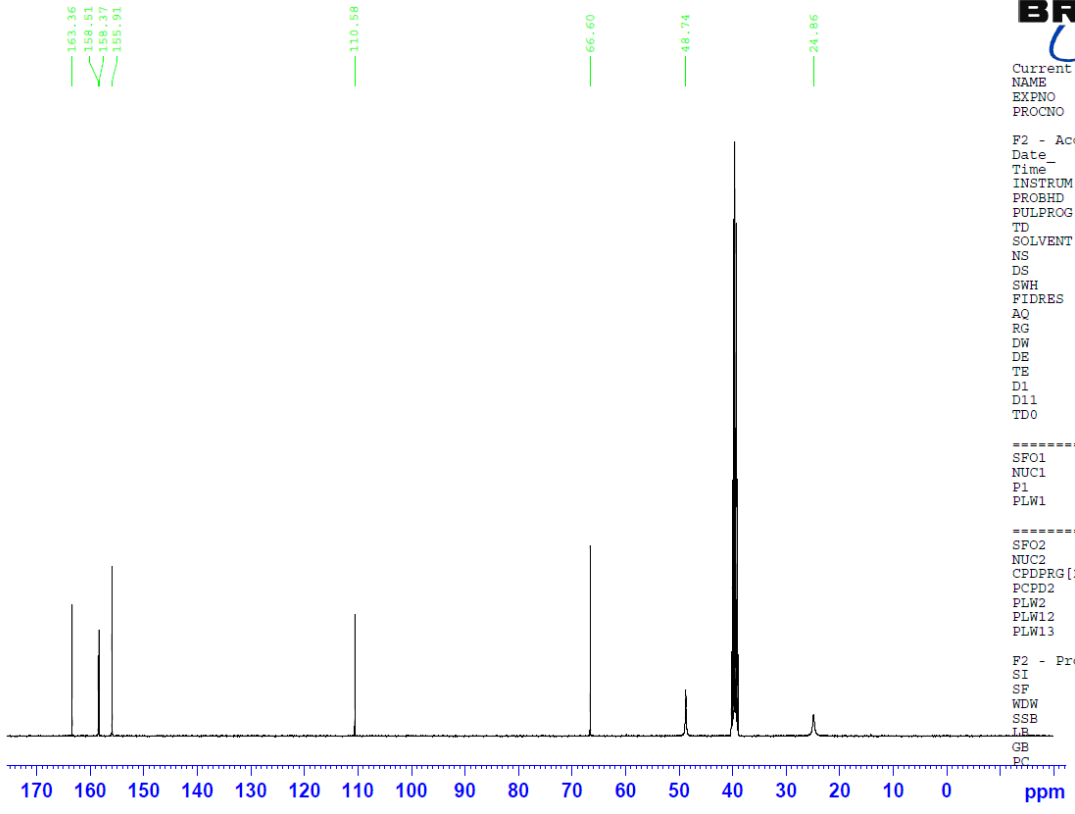

```
Current Data Parameters
NAME          Feb18-2016
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160218
Time               17.07
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 31.12
DW                62.400 usec
DE                 6.50 usec
TE                295.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                 9.50 usec
PLW1          15.80000019 W

F2 - Processing parameters
SI                 65536
SF            400.1300032 MHz
WDW                   EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```

8_13C




```
Current Data Parameters
NAME          Feb18-2016
EXPNO                  3
PROCNO                 1

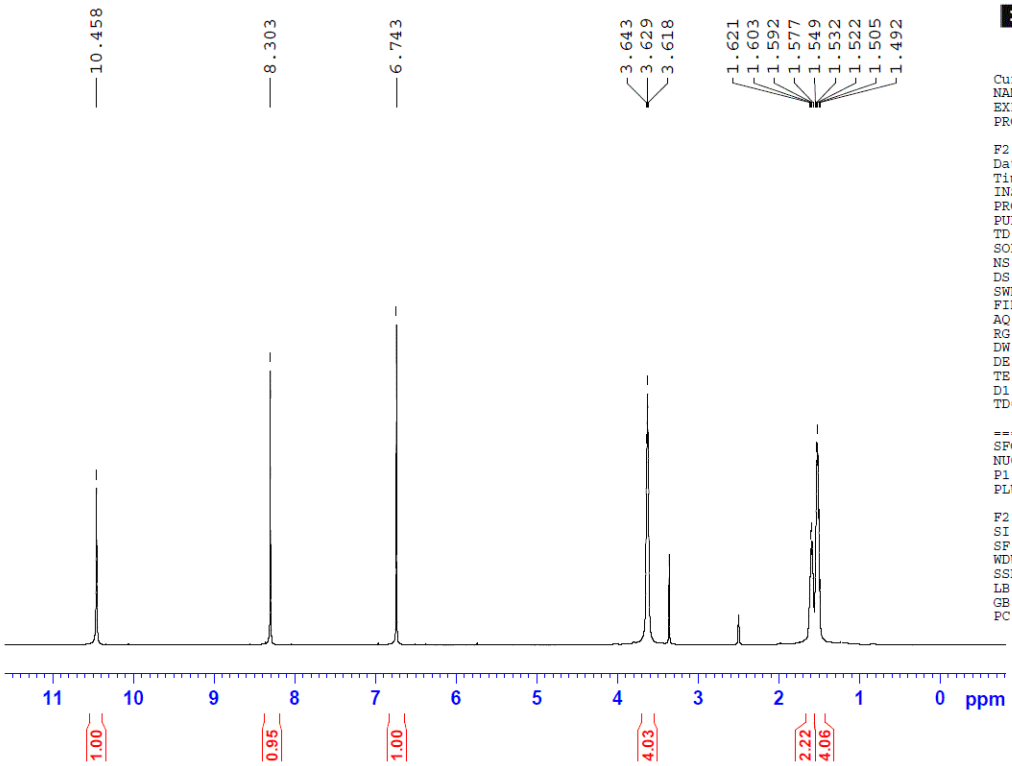
F2 - Acquisition Parameters
Date_           20160219
Time               1.10
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT             DMSO
NS                  8192
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                199.94
DW                20.800 usec
DE                 6.50 usec
TE                295.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228293 MHz
NUC1                  13C
P1                 9.50 usec
PLW1          66.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2          18.00000000 W
PLW12          0.20056000 W
PLW13          0.16245000 W

F2 - Processing parameters
SI                 32768
SF            100.6128048 MHz
WDW                   EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```

9_1H



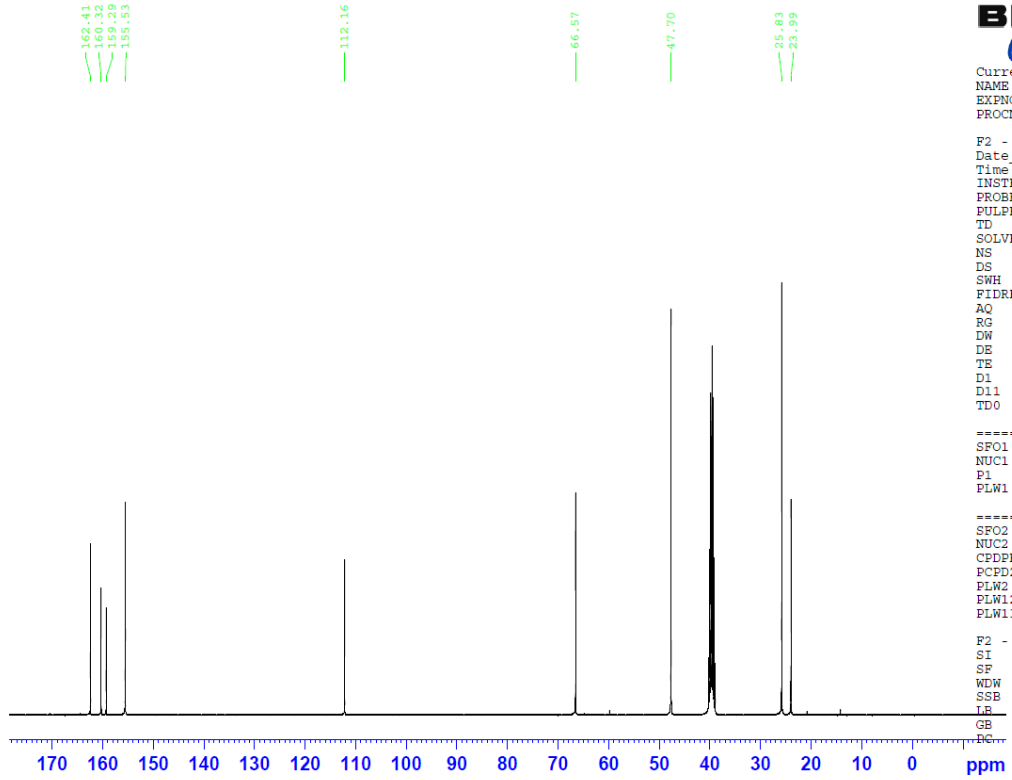

```
Current Data Parameters
NAME          Feb19-2016
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160219
Time               9.25
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 31.12
DW                62.400 usec
DE                 6.50 usec
TE                295.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  9.50 usec
PLW1         15.80000019 W

F2 - Processing parameters
SI                 65536
SF          400.1300032 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

9_13C




```
Current Data Parameters
NAME          Feb19-2016
EXPNO                  2
PROCNO                 1

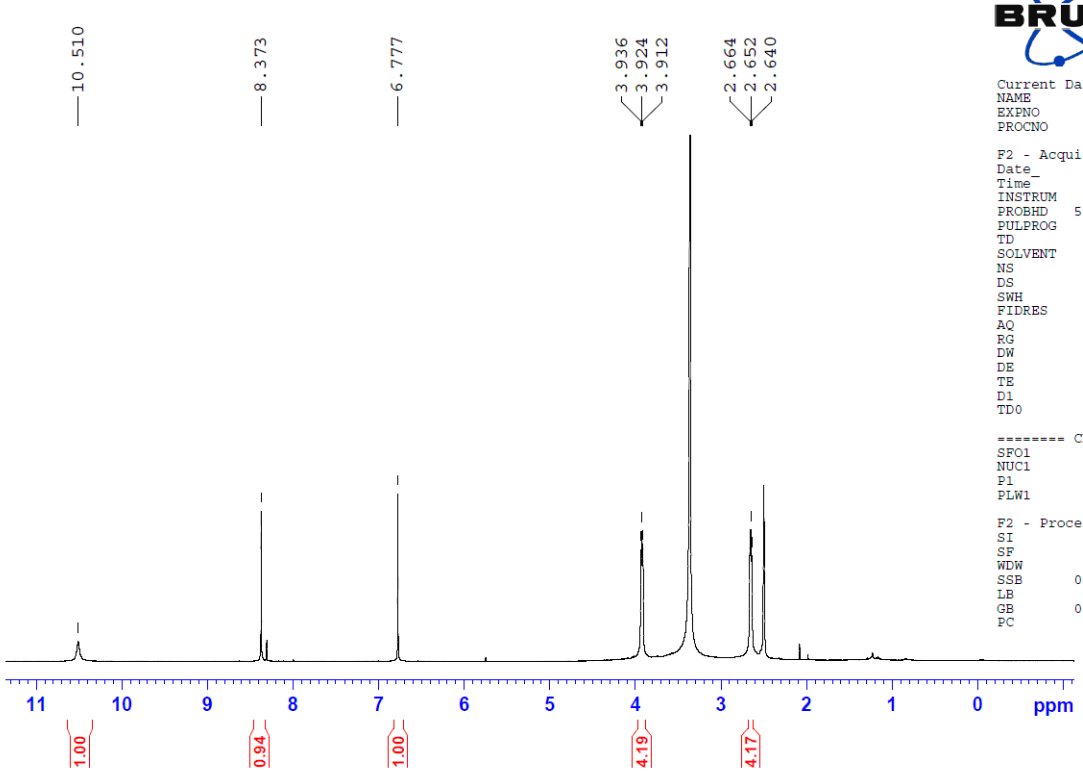
F2 - Acquisition Parameters
Date_           20160219
Time              13.23
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT             DMSO
NS                  4096
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                199.94
DW                20.800 usec
DE                 6.50 usec
TE                295.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                  13C
P1                  9.50 usec
PLW1         66.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         18.00000000 W
PLW12         0.20056000 W
PLW13         0.16245000 W

F2 - Processing parameters
SI                 32768
SF          100.6128056 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

10_1H



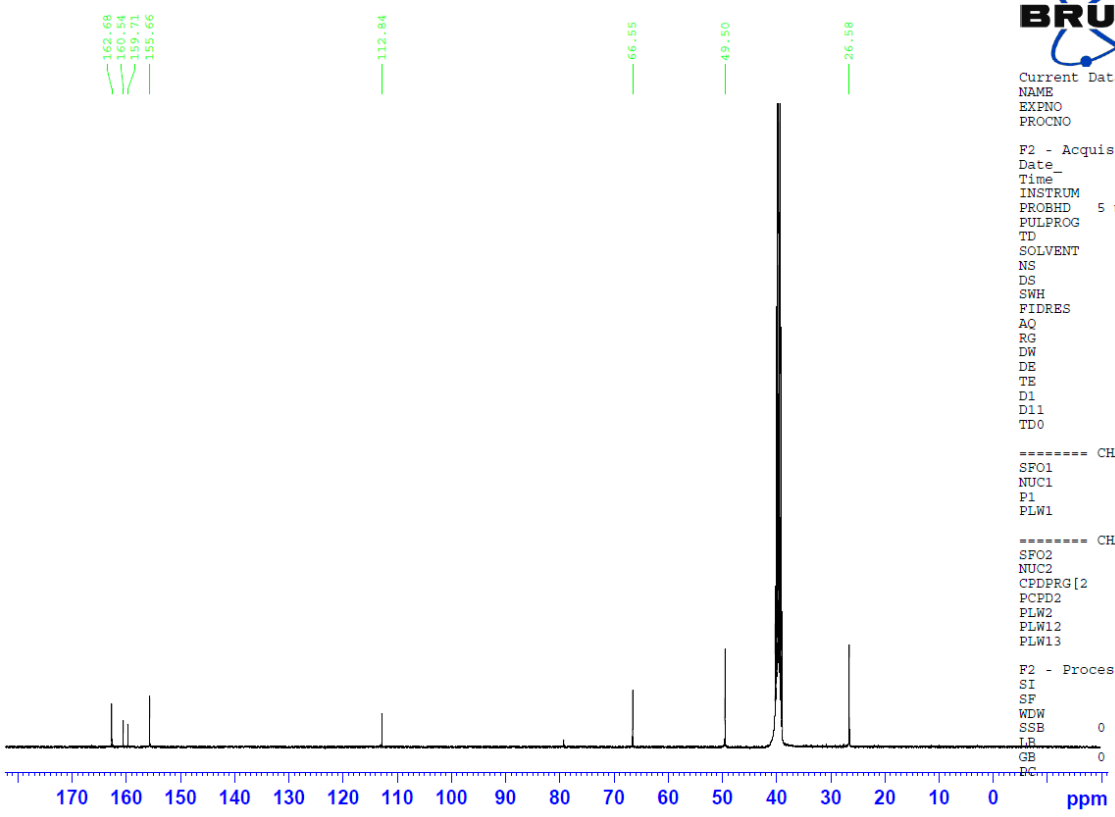

```
Current Data Parameters
NAME          Sep24-2015
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150924
Time              21.10
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT           DMSO
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG                78.4
DW              62.400 usec
DE               6.50 usec
TE              295.0 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1               1H
P1               9.50 usec
PLW1        15.80000019 W

F2 - Processing parameters
SI               65536
SF          400.1300037 MHz
WDW                EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```

10_13C




```
Current Data Parameters
NAME          Sep24-2015
EXPNO                  5
PROCNO                 1

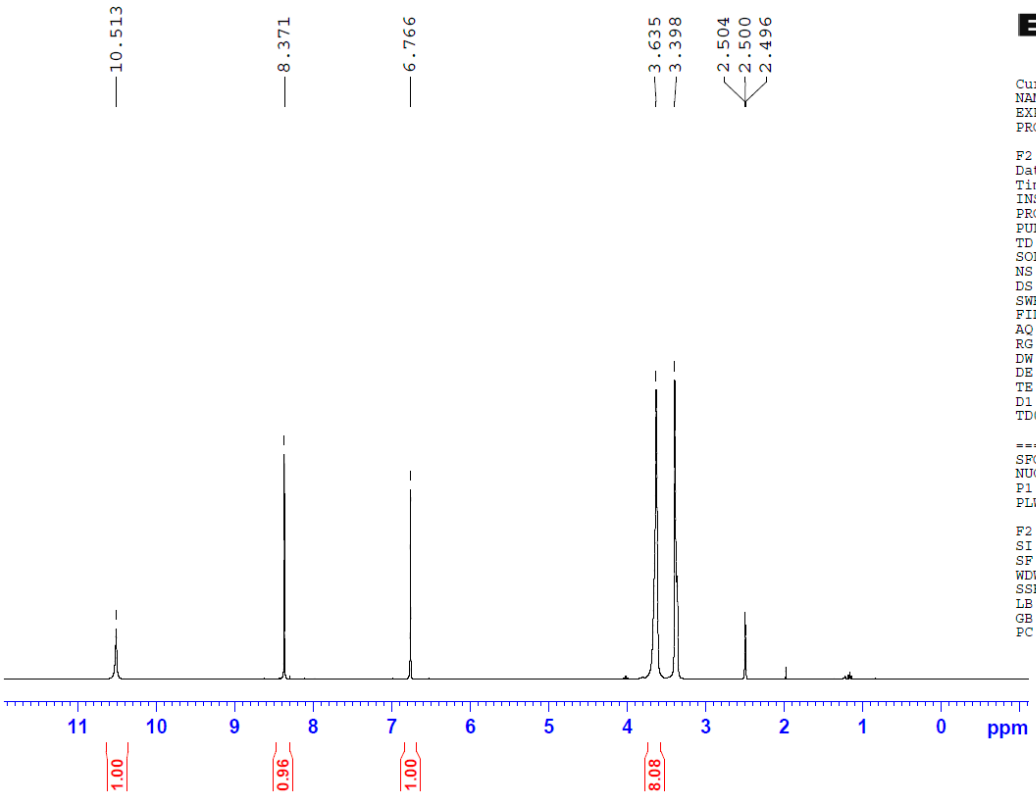
F2 - Acquisition Parameters
Date_           20150925
Time               7.24
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD               65536
SOLVENT           DMSO
NS                 9216
DS                    4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               199.94
DW              20.800 usec
DE               6.50 usec
TE              295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1               13C
P1               9.50 usec
PLW1        66.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2            90.00 usec
PLW2        18.00000000 W
PLW12        0.20056000 W
PLW13        0.16245000 W

F2 - Processing parameters
SI               32768
SF          100.6128032 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```

11_1H



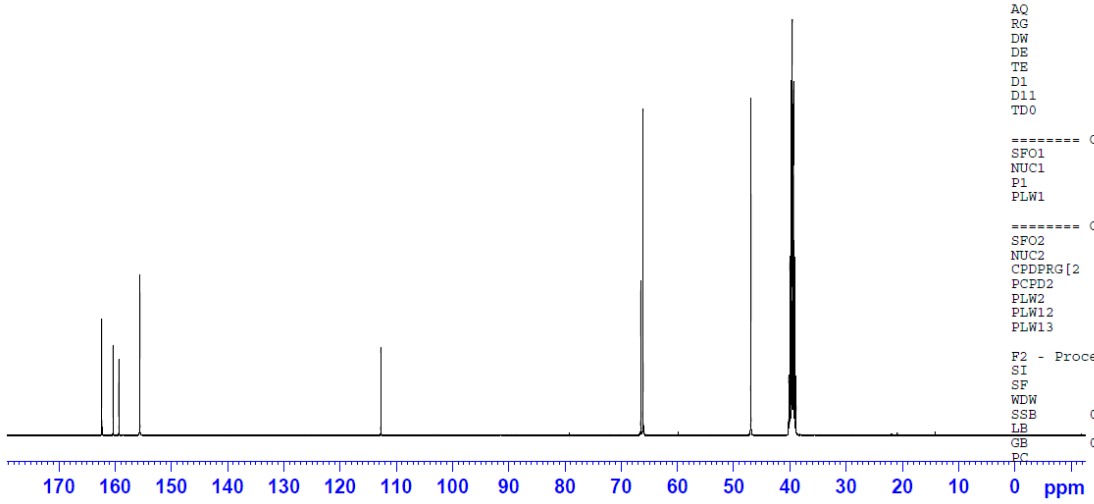

```
Current Data Parameters
NAME            Jan07-2016
EXPNO                    8
PROCNO                   1

F2 - Acquisition Parameters
Date_             20160107
Time                 21.27
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                     64
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  31.12
DW                62.400 usec
DE                 6.50 usec
TE                295.0 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                    1H
P1                   9.50 usec
PLW1          15.80000019 W

F2 - Processing parameters
SI                   65536
SF            400.1300032 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```

11_13C




```
Current Data Parameters
NAME            Jan07-2016
EXPNO                    9
PROCNO                   1

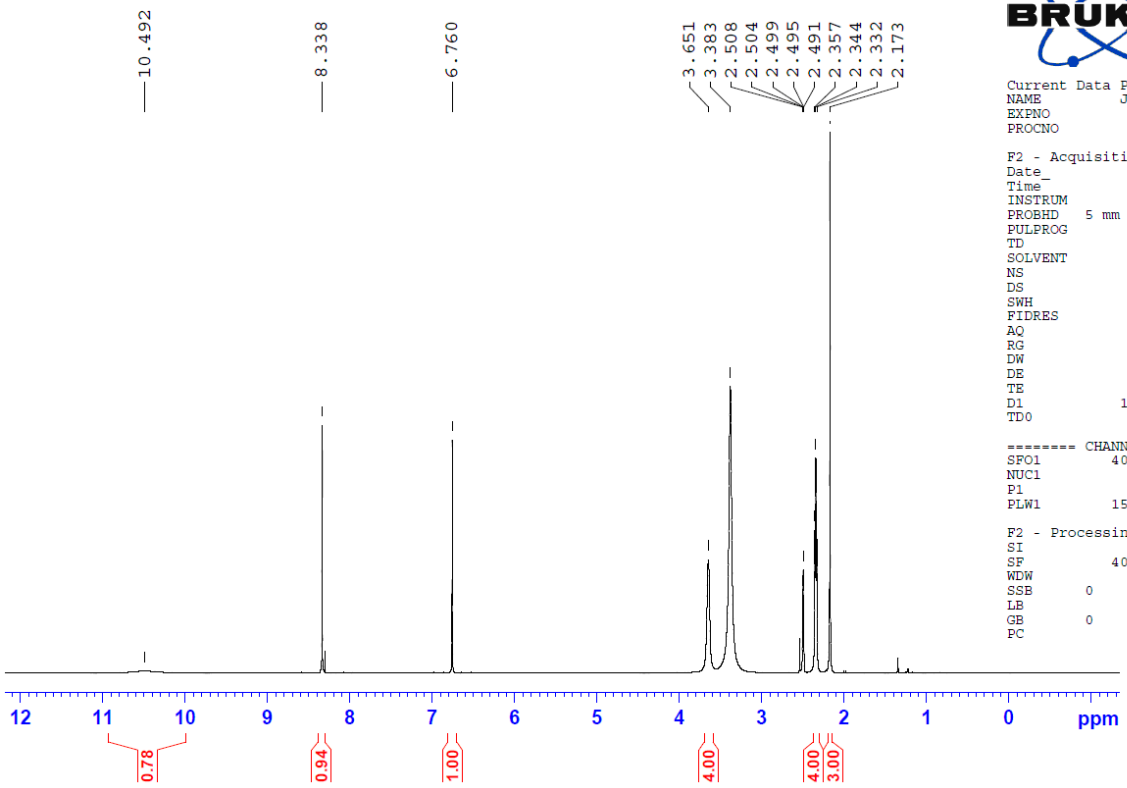
F2 - Acquisition Parameters
Date_             20160108
Time                  5.24
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT              DMSO
NS                   8192
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631488 sec
RG                  199.94
DW                20.800 usec
DE                 6.50 usec
TE                295.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          100.6228293 MHz
NUC1                   13C
P1                   9.50 usec
PLW1          66.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2               90.00 usec
PLW2          18.00000000 W
PLW12          0.20056000 W
PLW13          0.16245000 W

F2 - Processing parameters
SI                   32768
SF            100.6128036 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```

12_1H



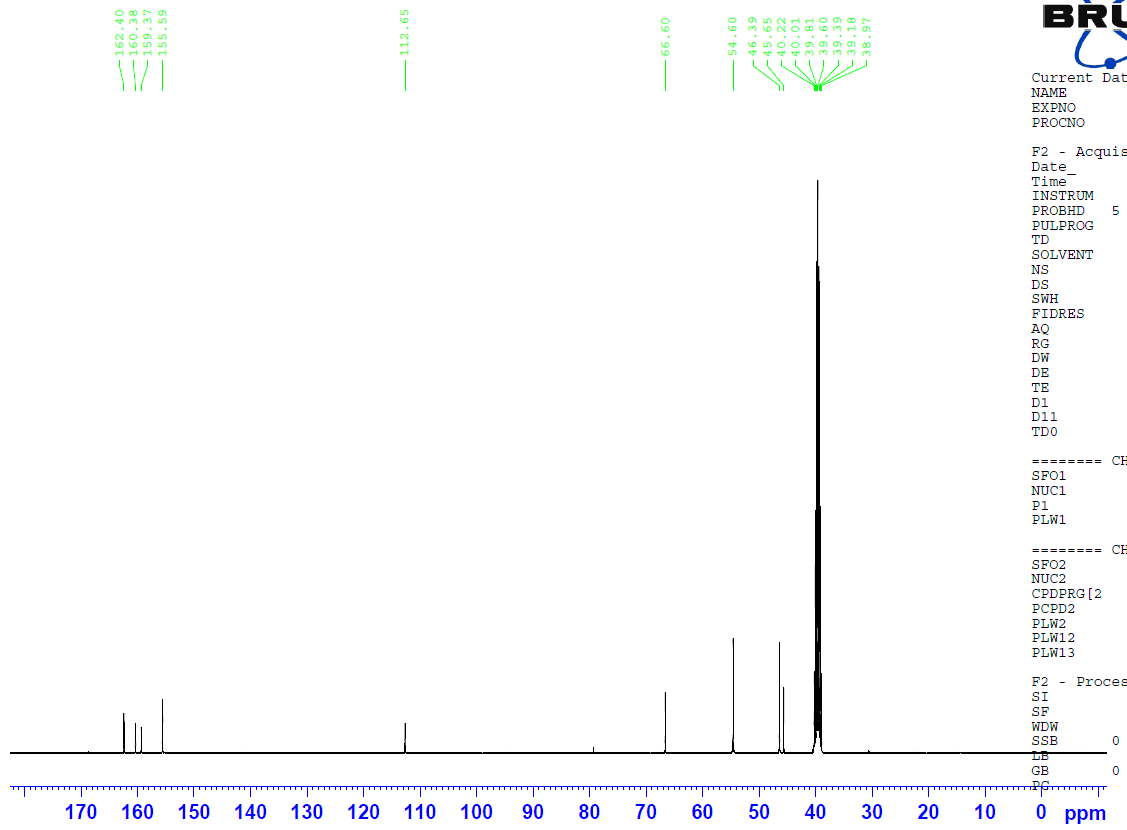

```
Current Data Parameters
NAME          Jan09-2016
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160109
Time              11.28
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                    32
DS                     2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                 48.54
DW                62.400 usec
DE                 6.50 usec
TE                295.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  9.50 usec
PLW1         15.80000019 W

F2 - Processing parameters
SI                 65536
SF           400.1300034 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

12_13C




```
Current Data Parameters
NAME          Jan09-2016
EXPNO                  2
PROCNO                 1

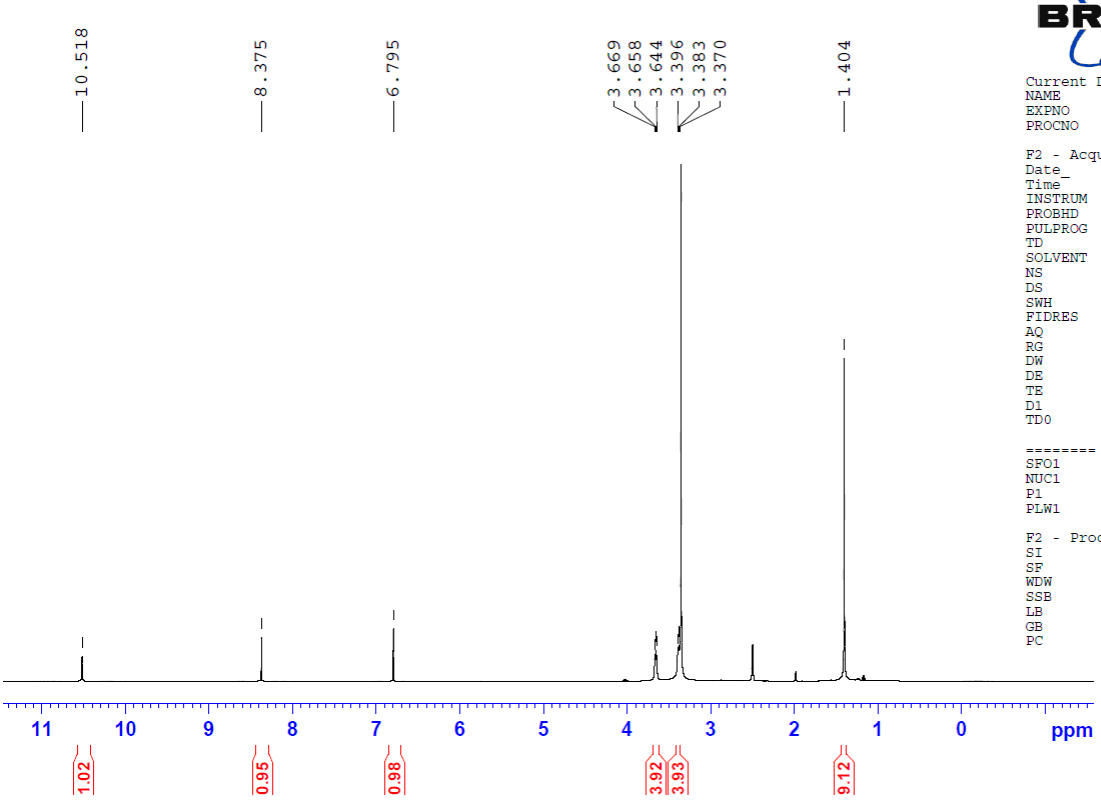
F2 - Acquisition Parameters
Date_           20160109
Time              19.23
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 8192
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                199.94
DW                20.800 usec
DE                 6.50 usec
TE                295.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                  13C
P1                  9.50 usec
PLW1         66.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         18.00000000 W
PLW12         0.20056000 W
PLW13         0.16245000 W

F2 - Processing parameters
SI                 32768
SF           100.6128026 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

13_1H



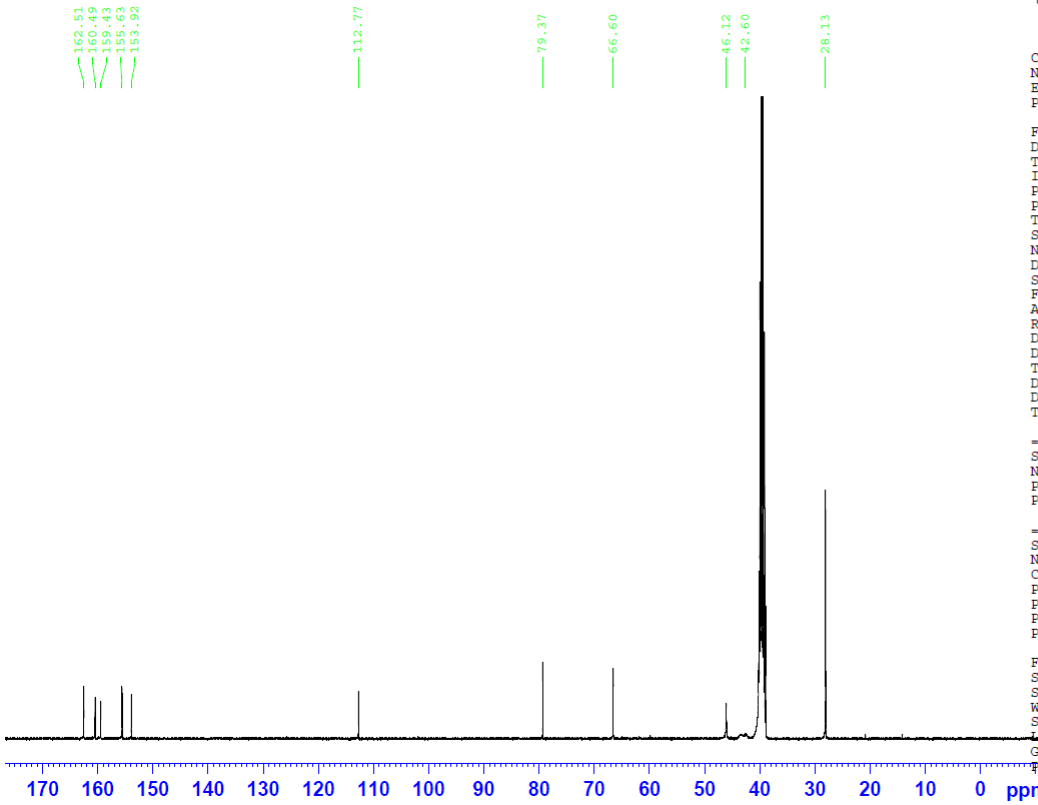

```
Current Data Parameters
NAME            Mar22-2016
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_             20160322
Time                 17.03
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                     16
DS                      2
SWH               8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894465 sec
RG                   71.13
DW                  62.400 usec
DE                   6.50 usec
TE                  295.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                    1H
P1                   9.50 usec
PLW1           15.80000019 W

F2 - Processing parameters
SI                  65536
SF              400.1300034 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```

13_13C




```
Current Data Parameters
NAME            Mar23-2016
EXPNO                    1
PROCNO                   1

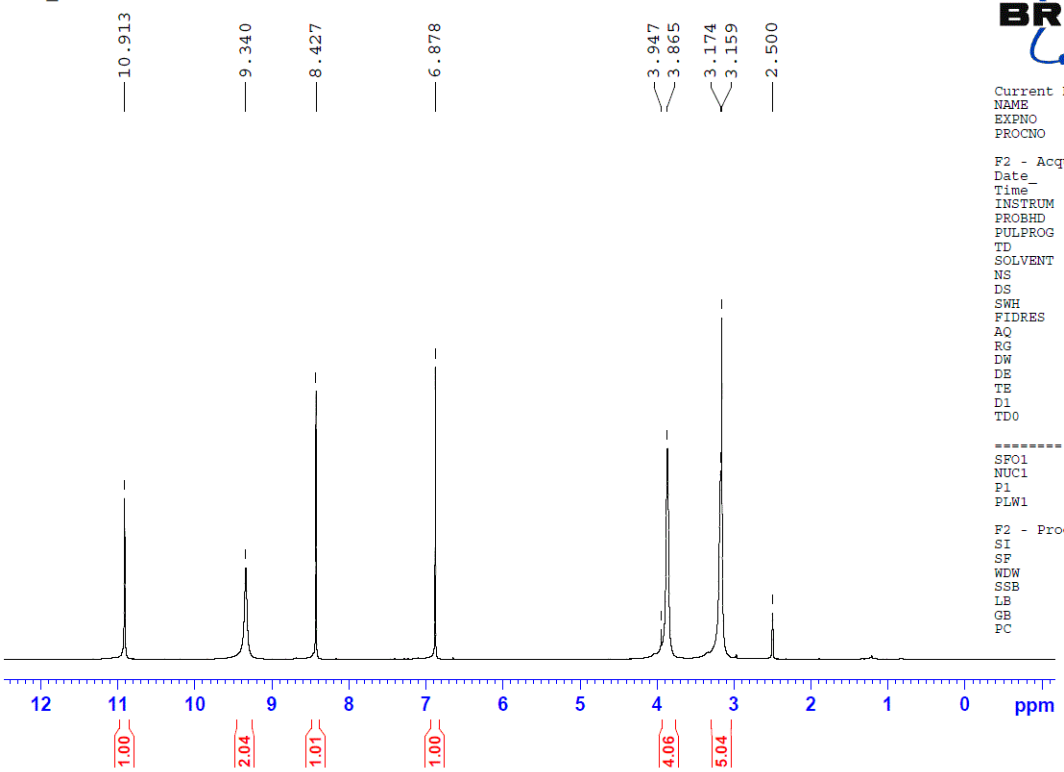
F2 - Acquisition Parameters
Date_             20160323
Time                 20.08
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT              DMSO
NS                    8192
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631488 sec
RG                   199.94
DW                  20.800 usec
DE                   6.50 usec
TE                  295.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1            100.6228293 MHz
NUC1                   13C
P1                   9.50 usec
PLW1           66.50000000 W

======== CHANNEL f2 ========
SFO2            400.1316005 MHz
NUC2                    1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2           18.00000000 W
PLW12           0.20056000 W
PLW13           0.16245000 W

F2 - Processing parameters
SI                  32768
SF              100.6128046 MHz
WDW                    EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```

14_1H



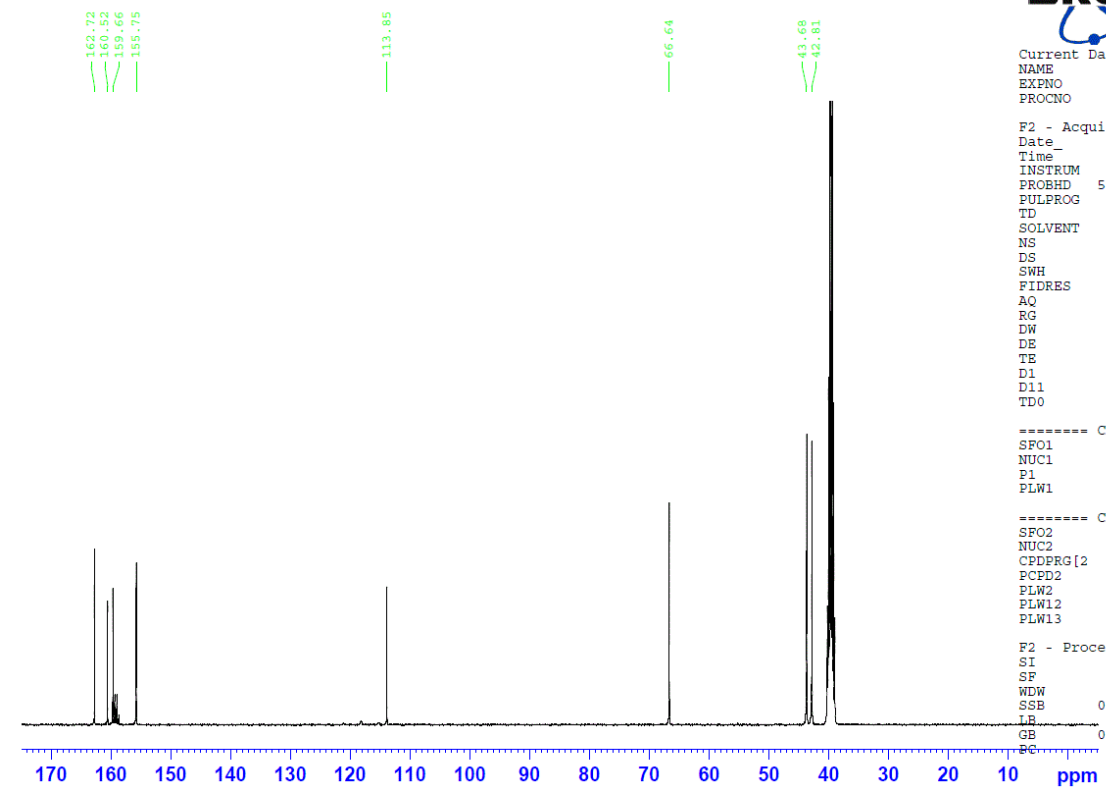

```
Current Data Parameters
NAME          Apr04-2016
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160404
Time               12.55
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    64
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 31.12
DW                62.400 usec
DE                 6.50 usec
TE                295.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  9.50 usec
PLW1         15.80000019 W

F2 - Processing parameters
SI                 65536
SF           400.1300036 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

14_13C




```
Current Data Parameters
NAME          Apr04-2016
EXPNO                  2
PROCNO                 1

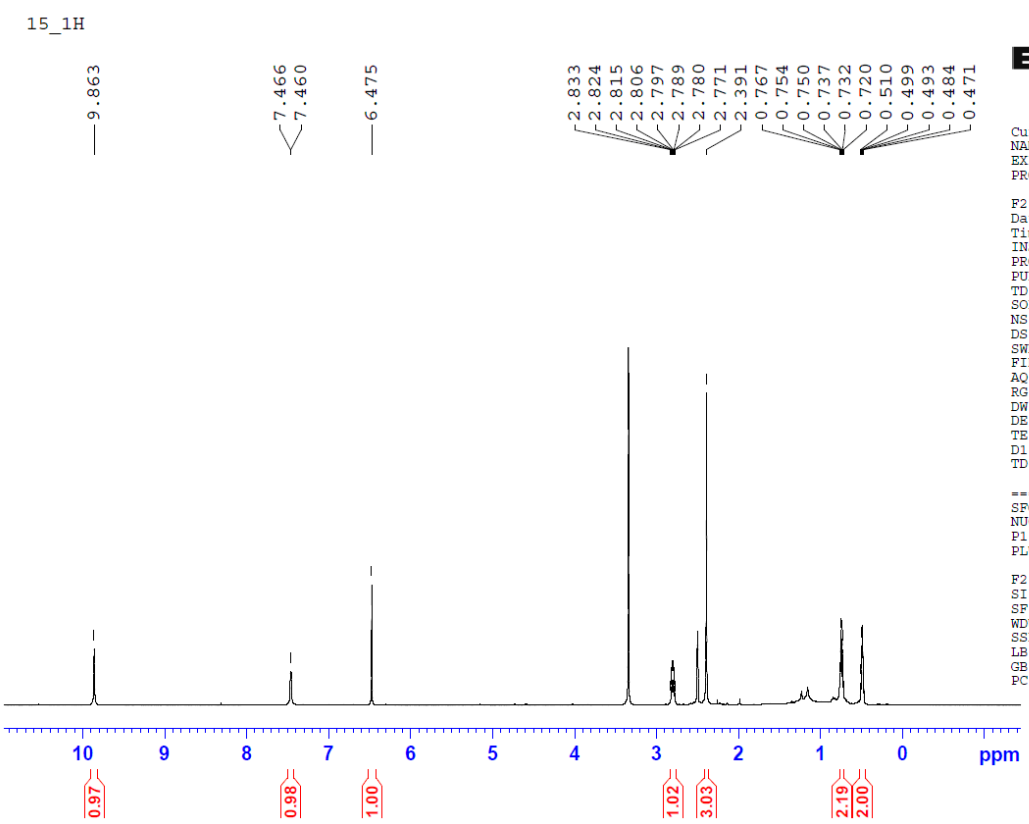
F2 - Acquisition Parameters
Date_           20160404
Time               20.44
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT             DMSO
NS                  8192
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                199.94
DW                20.800 usec
DE                 6.50 usec
TE                295.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                  13C
P1                  9.50 usec
PLW1         66.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         18.00000000 W
PLW12         0.20056000 W
PLW13         0.16245000 W

F2 - Processing parameters
SI                 32768
SF           100.6127875 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

15_1H



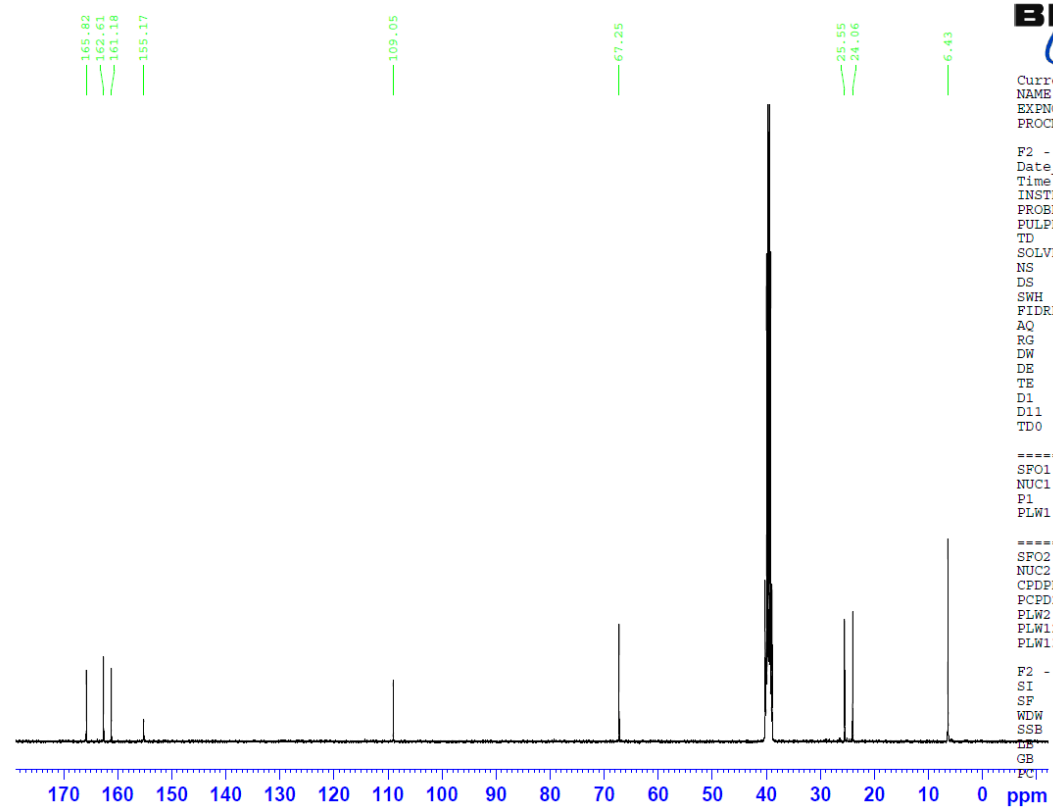

```
Current Data Parameters
NAME          Jan11-2016
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160111
Time              22.22
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                71.13
DW               62.400 usec
DE                6.50 usec
TE               295.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  9.50 usec
PLW1         15.80000019 W

F2 - Processing parameters
SI                 65536
SF           400.1300035 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

15_13C




```
Current Data Parameters
NAME          Jan11-2016
EXPNO                  2
PROCNO                 1

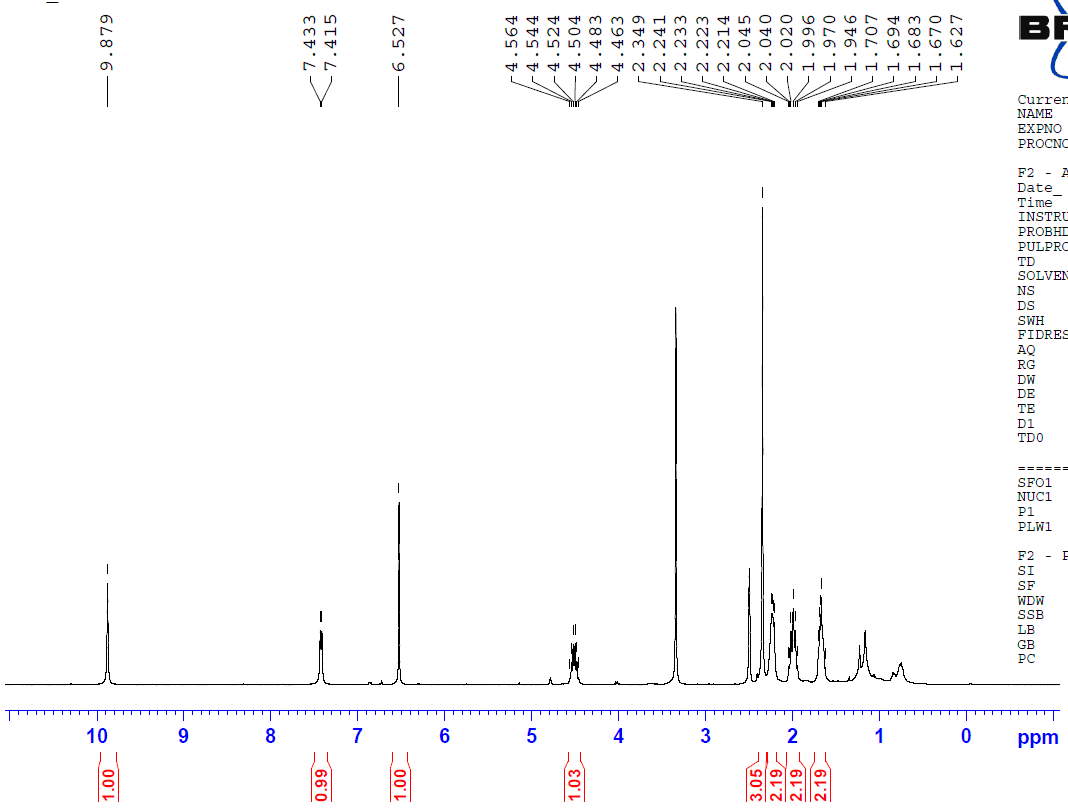
F2 - Acquisition Parameters
Date_           20160112
Time               6.24
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                  8192
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                199.94
DW               20.800 usec
DE                6.50 usec
TE               295.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                  13C
P1                  9.50 usec
PLW1         66.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         18.00000000 W
PLW12         0.20056000 W
PLW13         0.16245000 W

F2 - Processing parameters
SI                 32768
SF           100.6128055 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

16_1H



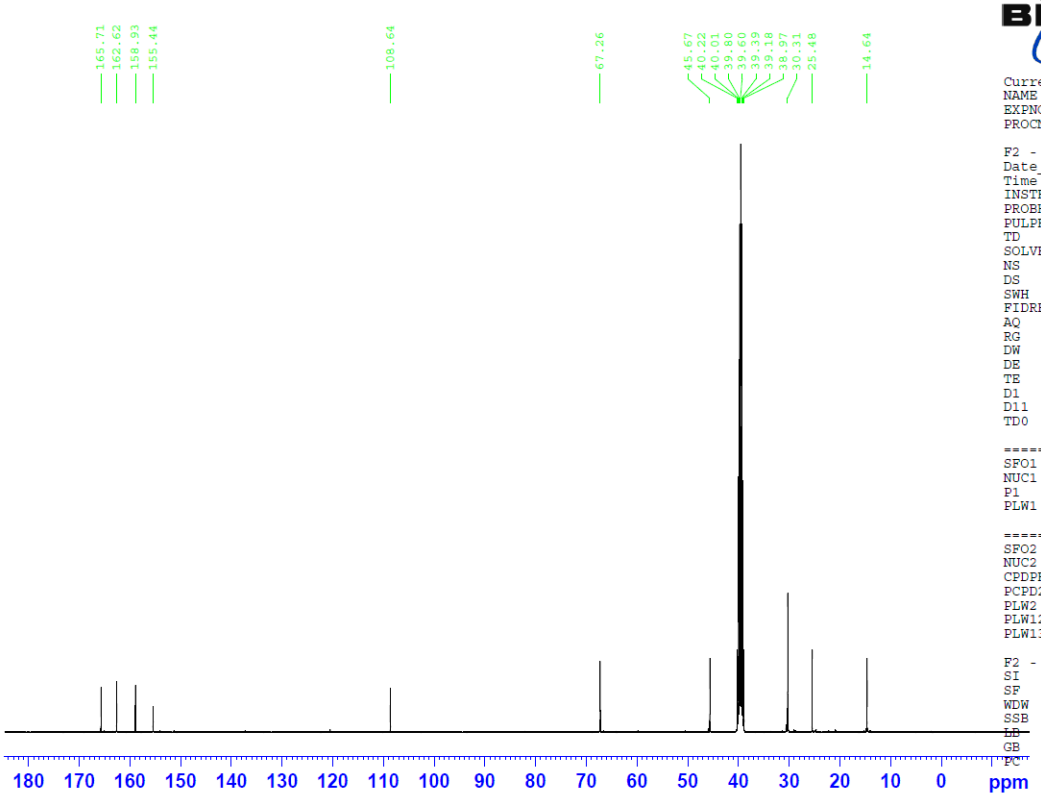

```
Current Data Parameters
NAME          Jan12-2016
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160112
Time               9.36
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 60.77
DW                62.400 usec
DE                 6.50 usec
TE                295.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                 9.50 usec
PLW1          15.80000019 W

F2 - Processing parameters
SI                 65536
SF            400.1300035 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

16_13C




```
Current Data Parameters
NAME          Jan12-2016
EXPNO                  3
PROCNO                 1

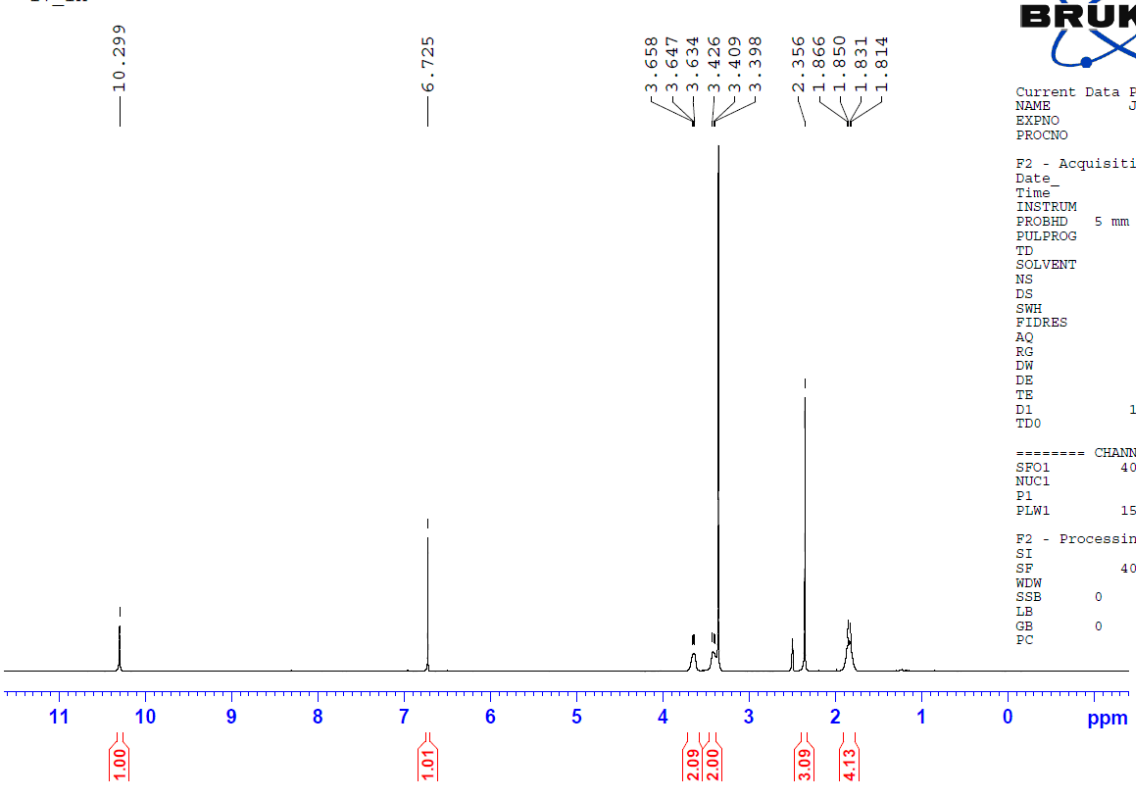
F2 - Acquisition Parameters
Date_           20160112
Time              17.58
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                  8192
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                199.94
DW                20.800 usec
DE                 6.50 usec
TE                295.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228293 MHz
NUC1                  13C
P1                 9.50 usec
PLW1          66.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          18.00000000 W
PLW12          0.20056000 W
PLW13          0.16245000 W

F2 - Processing parameters
SI                 32768
SF            100.6128058 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

17_1H



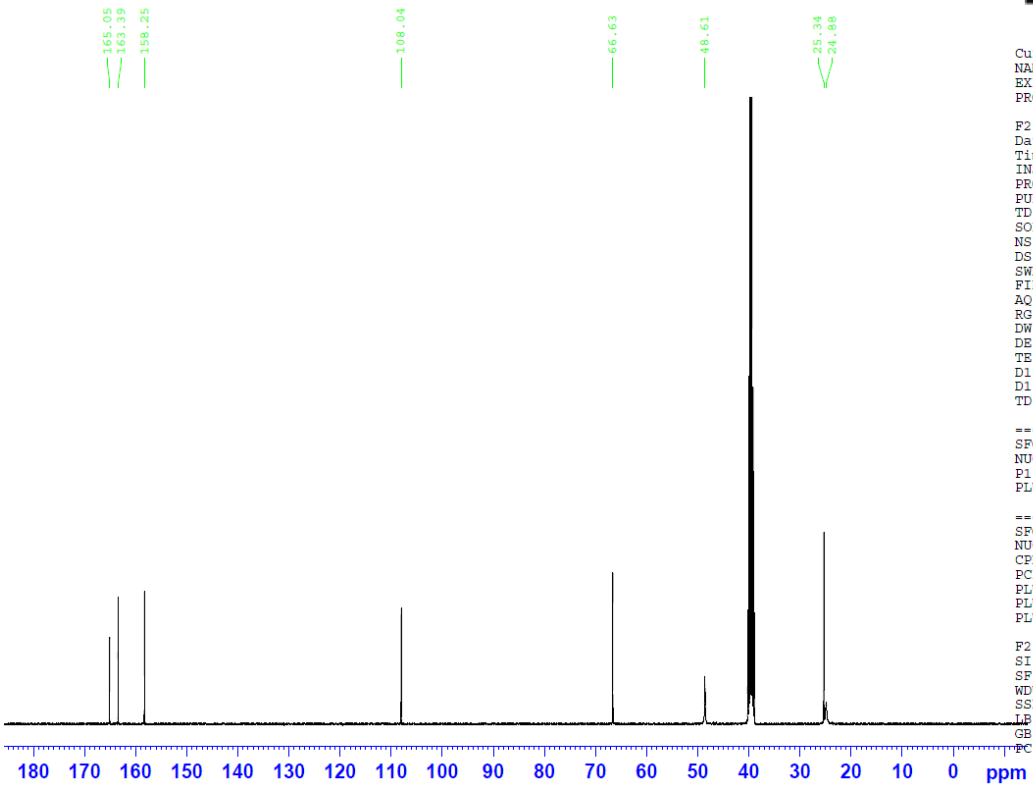

```
Current Data Parameters
NAME          Jan04-2016
EXPNO                  9
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160104
Time              22.06
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT           DMSO
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG                54.6
DW              62.400 usec
DE               6.50 usec
TE              295.0 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                9.50 usec
PLW1       15.80000019 W

F2 - Processing parameters
SI                65536
SF          400.1300033 MHz
WDW                 EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
```

17_13C




```
Current Data Parameters
NAME          Jan05-2016
EXPNO                  1
PROCNO                 1

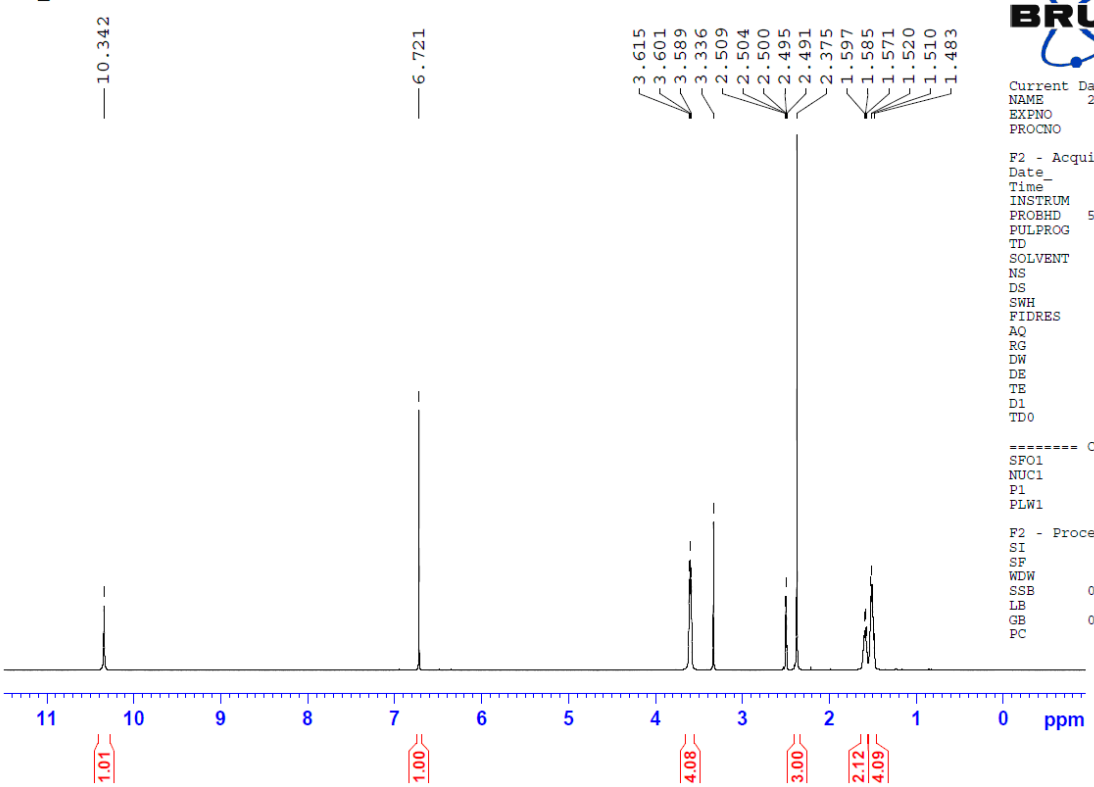
F2 - Acquisition Parameters
Date_           20160105
Time              13.06
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT           DMSO
NS                4096
DS                   4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               199.94
DW              20.800 usec
DE               6.50 usec
TE              295.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                13C
P1                9.50 usec
PLW1       66.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2       18.00000000 W
PLW12       0.20056000 W
PLW13       0.16245000 W

F2 - Processing parameters
SI                32768
SF          100.6128046 MHz
WDW                 EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```

18_1H



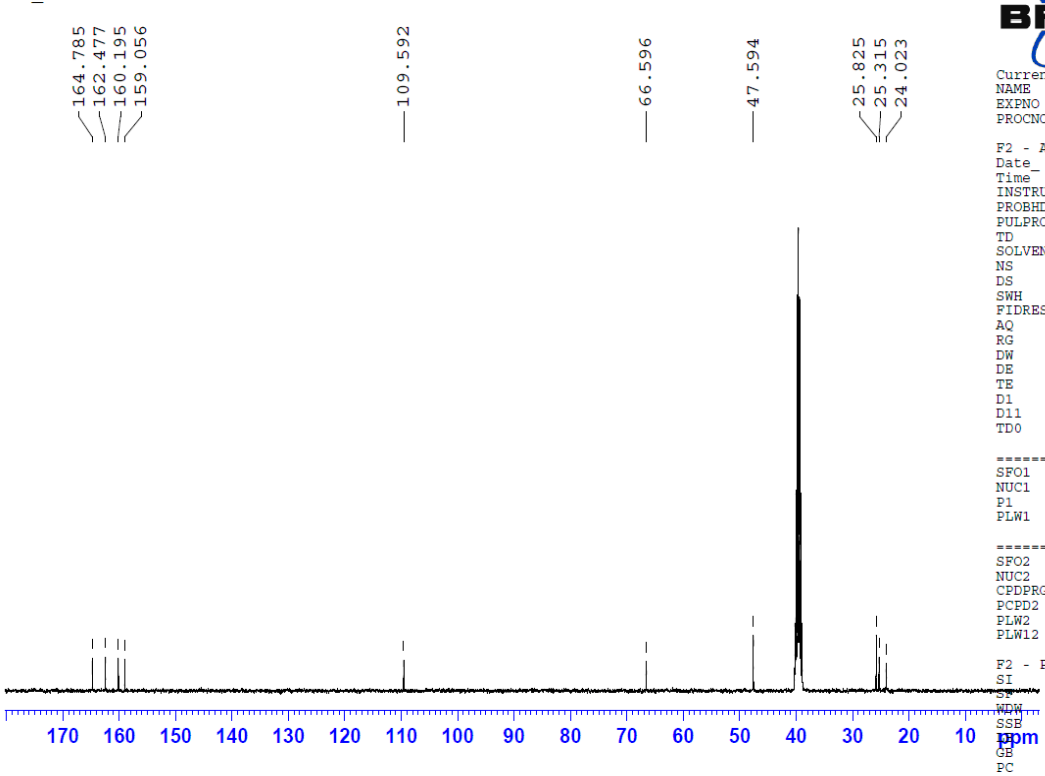

```
Current Data Parameters
NAME      20151229_DDC22_1H
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20151229
Time              20.26
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT           DMSO
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG               71.13
DW              62.400 usec
DE               6.50 usec
TE              295.0 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                9.50 usec
PLW1        15.80000019 W

F2 - Processing parameters
SI                65536
SF          400.1300032 MHz
WDW                  EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
```

18_13C




```
Current Data Parameters
NAME      20151229_DDC22_1H
EXPNO                    2
PROCNO                   1

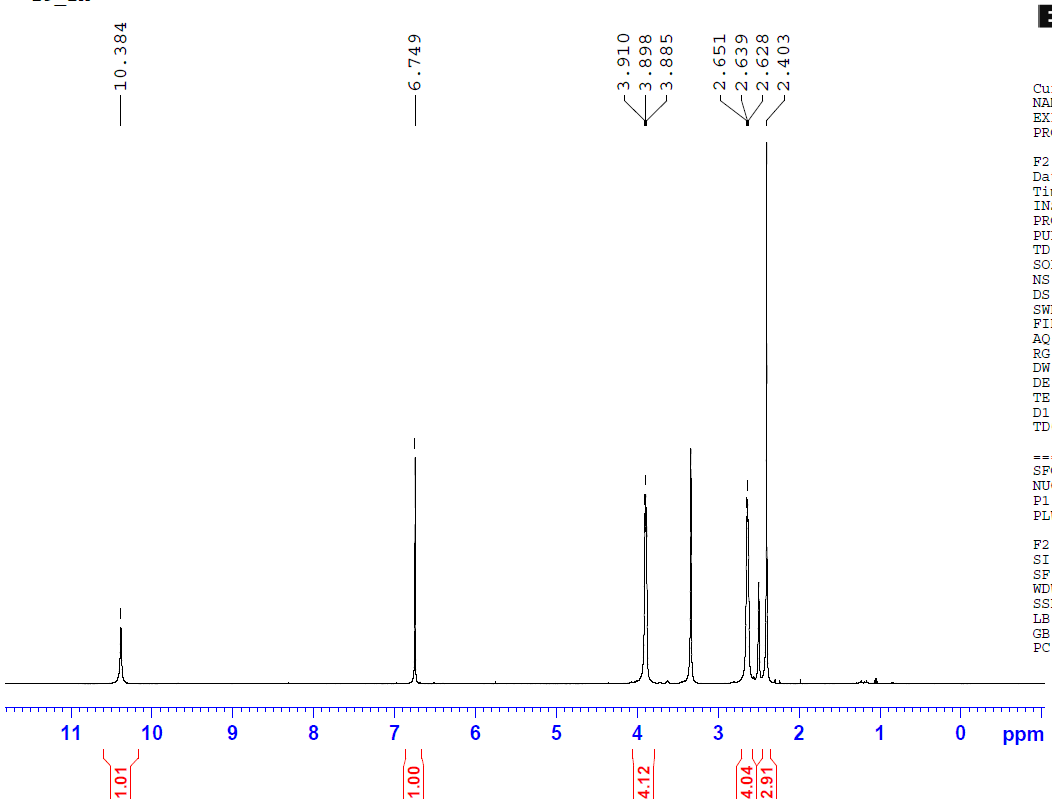
F2 - Acquisition Parameters
Date_           20151229
Time              21.07
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zgig
TD               65536
SOLVENT           DMSO
NS                 128
DS                   4
SWH          20161.291 Hz
FIDRES        0.307637 Hz
AQ            1.6252928 sec
RG               199.94
DW              24.800 usec
DE              10.98 usec
TE              295.0 K
D1          10.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6218237 MHz
NUC1                13C
P1                9.50 usec
PLW1        66.50000000 W

======== CHANNEL f2 ========
SFO2        400.1318529 MHz
NUC2                1H
CPDPRG[2         waltz17
PCPD2             90.00 usec
PLW2        18.00000000 W
PLW12        0.20056000 W

F2 - Processing parameters
SI                65536
SF          100.6128068 MHz
WDW                  EM
SSB       0
LB              3.00 Hz
GB        0
PC              1.00
```

19_1H



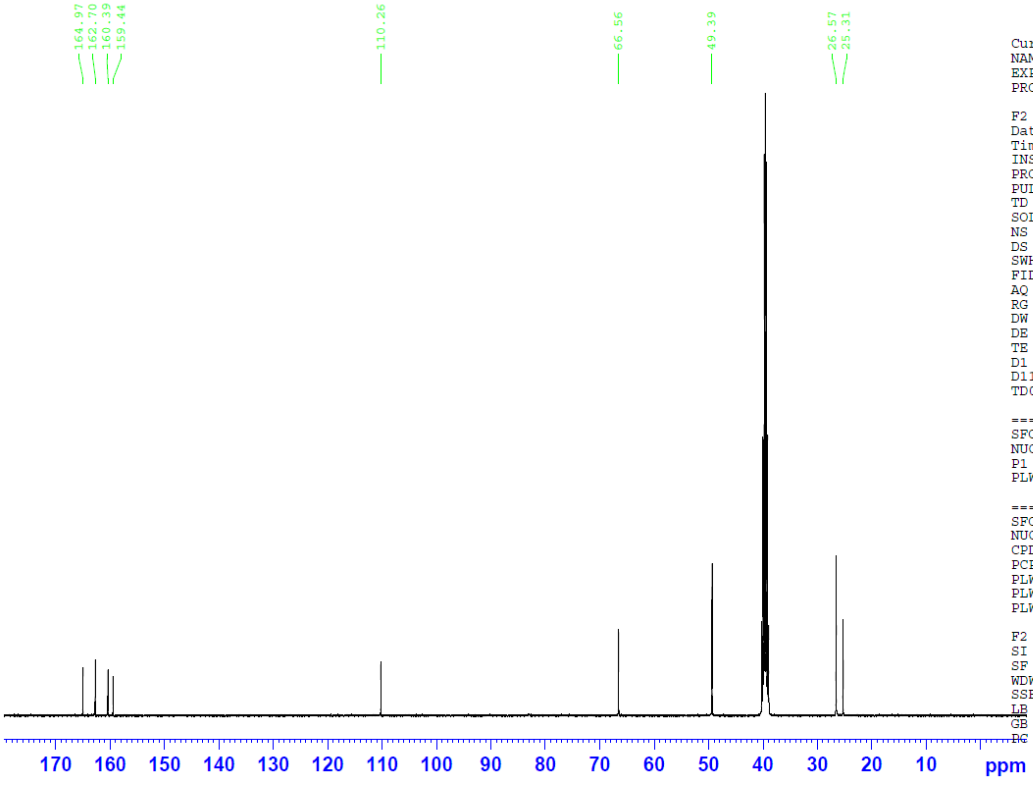

```
Current Data Parameters
NAME      20151230_DDC24_1H
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_           20151230
Time              17.46
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT           DMSO
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG               71.13
DW             62.400 usec
DE              6.50 usec
TE             295.0 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                   1H
P1                  9.50 usec
PLW1        15.80000019 W

F2 - Processing parameters
SI                   65536
SF          400.1300031 MHz
WDW                     EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

19_13C




```
Current Data Parameters
NAME      20151230_DDC24_13C
EXPNO                     1
PROCNO                    1

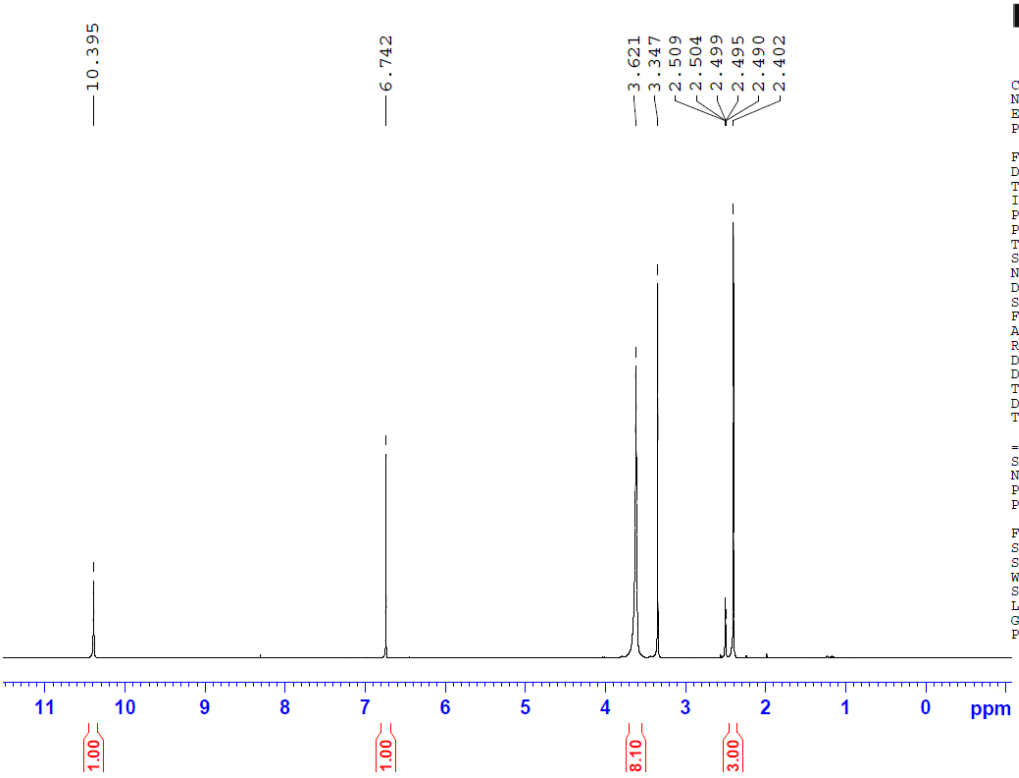
F2 - Acquisition Parameters
Date_           20151230
Time              21.50
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT           DMSO
NS                4096
DS                   4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               199.94
DW             20.800 usec
DE              6.50 usec
TE             295.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                   13C
P1                  9.50 usec
PLW1        66.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                   1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        18.00000000 W
PLW12        0.20056000 W
PLW13        0.16245000 W

F2 - Processing parameters
SI                   32768
SF          100.6128062 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

20_1H



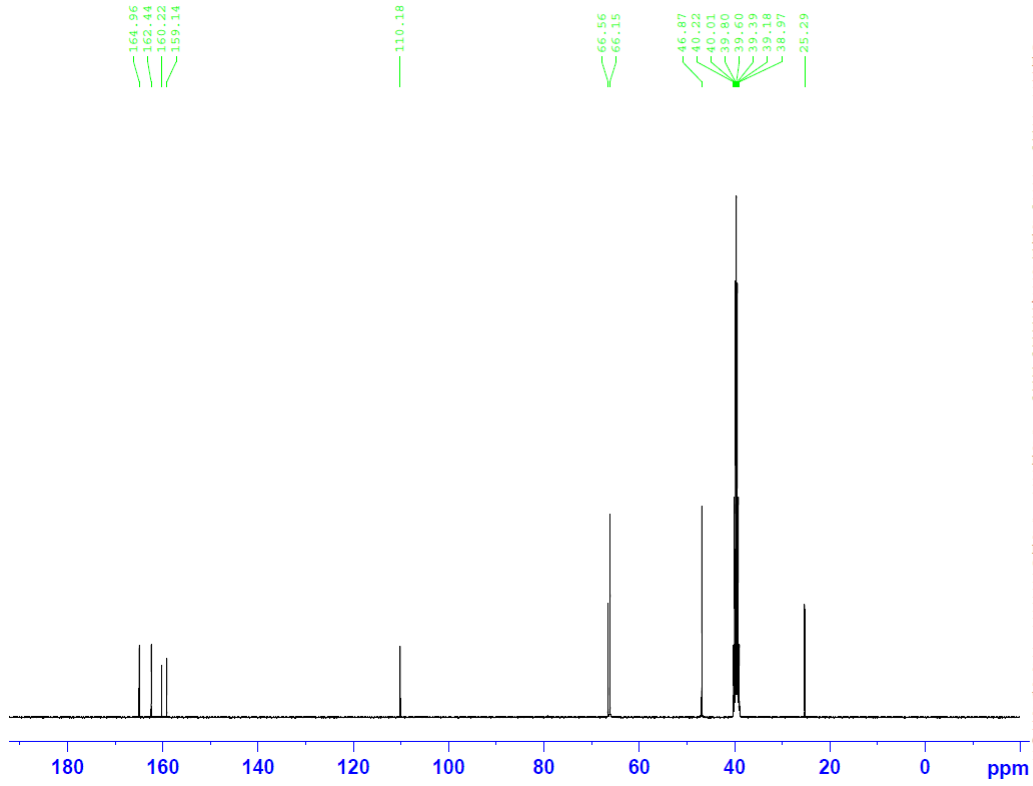

```
Current Data Parameters
NAME          Jan07-2016
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160107
Time               20.40
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    32
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                  54.6
DW                62.400 usec
DE                 6.50 usec
TE                295.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  9.50 usec
PLW1         15.80000019 W

F2 - Processing parameters
SI                 65536
SF          400.1300033 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

20_13C




```
Current Data Parameters
NAME          Jan07-2016
EXPNO                  3
PROCNO                 1

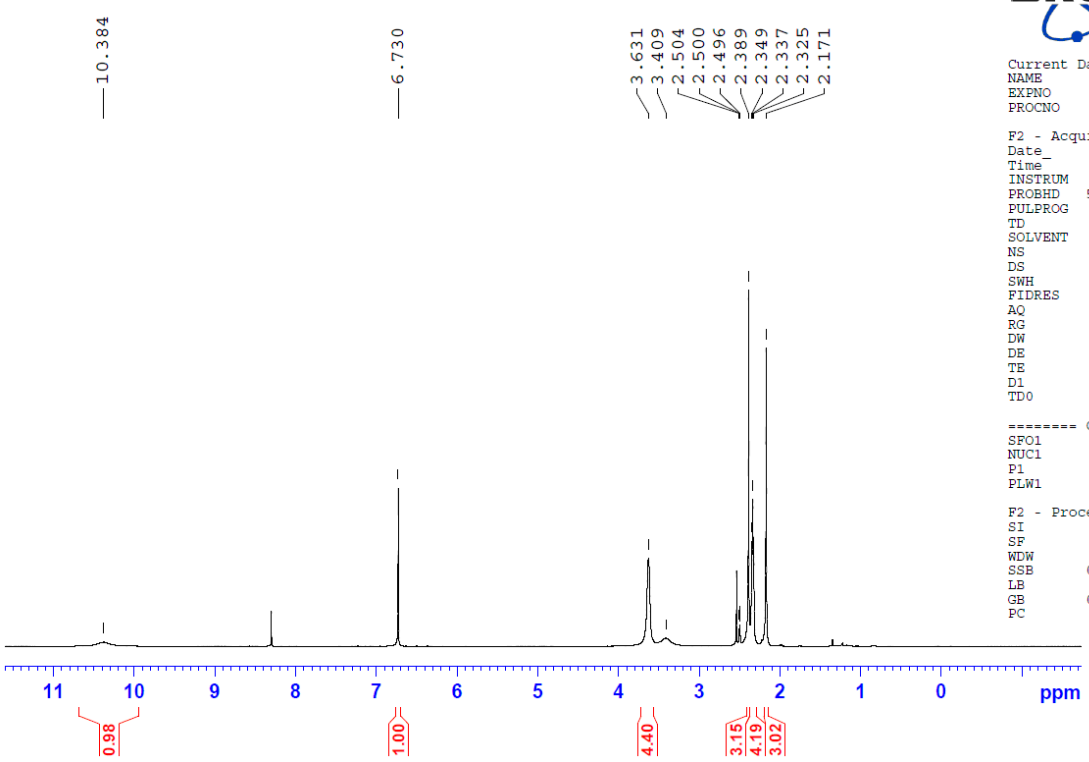
F2 - Acquisition Parameters
Date_           20160107
Time               18.00
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT             DMSO
NS                  4096
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                199.94
DW                20.800 usec
DE                 6.50 usec
TE                295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                  13C
P1                  9.50 usec
PLW1         66.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         18.00000000 W
PLW12         0.20056000 W
PLW13         0.16245000 W

F2 - Processing parameters
SI                 32768
SF          100.6128055 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

21_1H



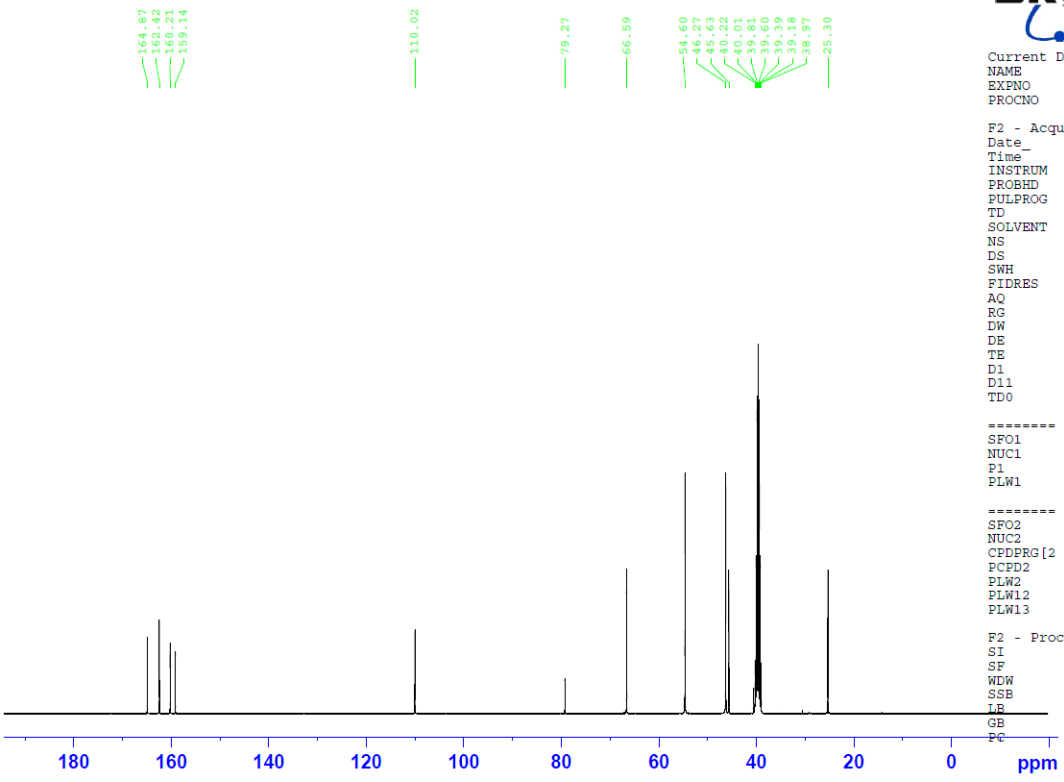

```
Current Data Parameters
NAME          Jan07-2016
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160107
Time               21.06
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 31.12
DW                62.400 usec
DE                 6.50 usec
TE                295.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                 9.50 usec
PLW1          15.80000019 W

F2 - Processing parameters
SI                 65536
SF            400.1300031 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

21_13C




```
Current Data Parameters
NAME          Jan10-2016
EXPNO                  1
PROCNO                 1

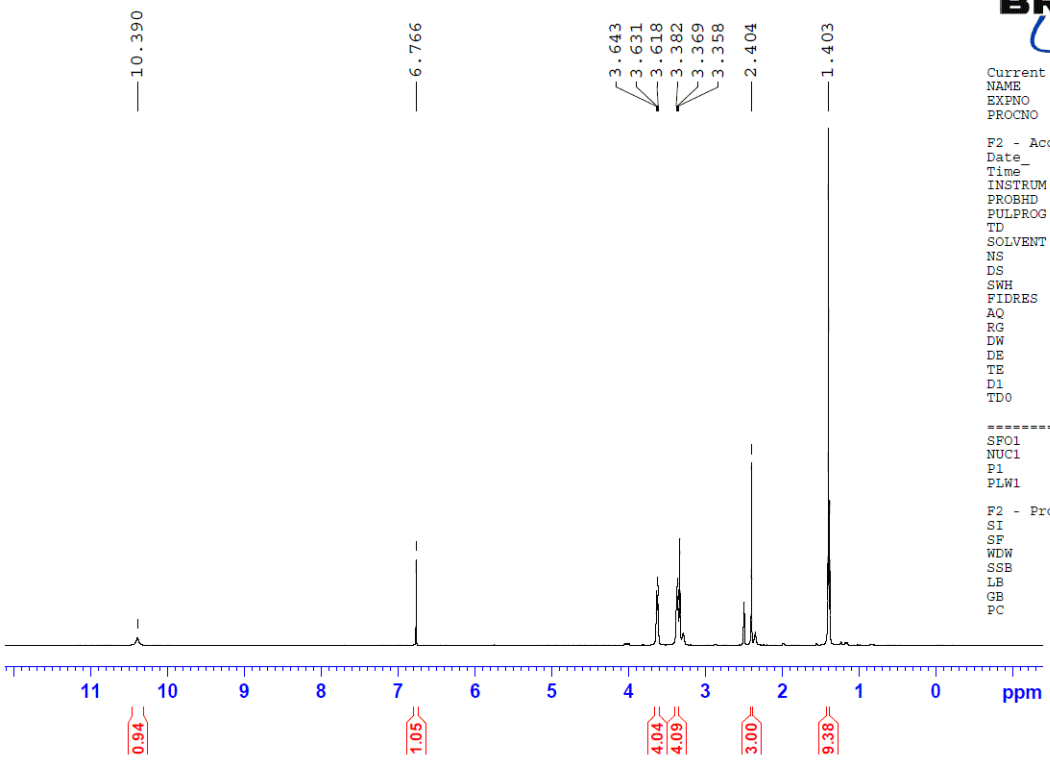
F2 - Acquisition Parameters
Date_           20160110
Time               17.33
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT             DMSO
NS                  8192
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                199.94
DW                20.800 usec
DE                 6.50 usec
TE                295.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228293 MHz
NUC1                  13C
P1                 9.50 usec
PLW1          66.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2          18.00000000 W
PLW12          0.20056000 W
PLW13          0.16245000 W

F2 - Processing parameters
SI                 32768
SF            100.6128056 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

22_1H



BRUKER

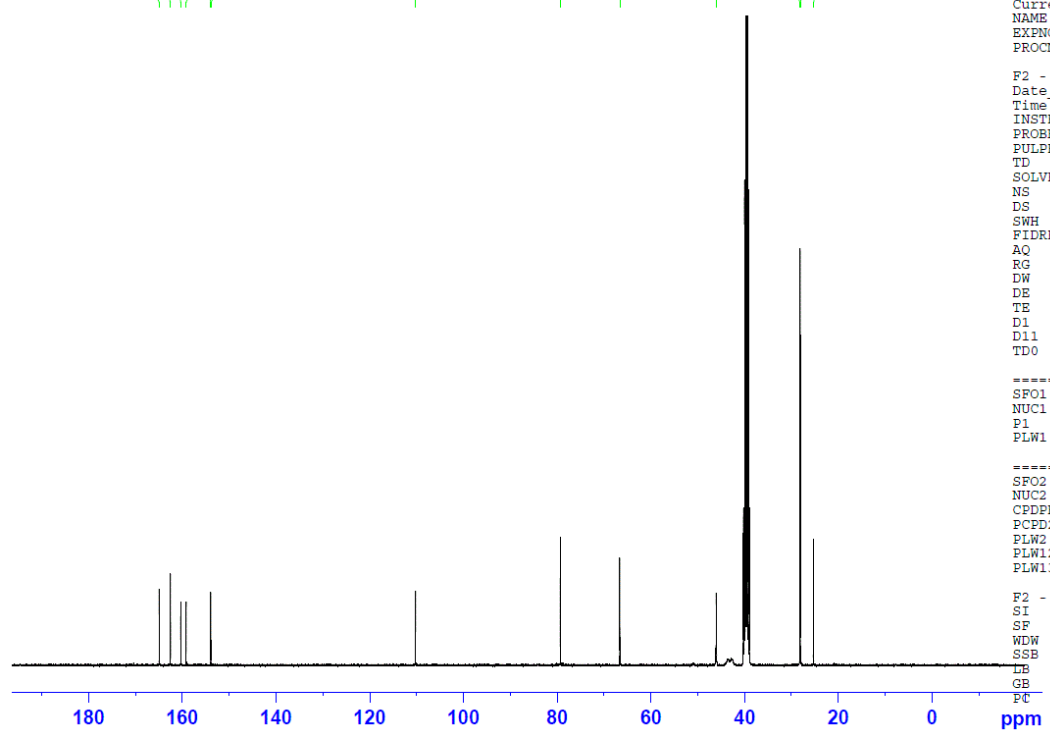
```
Current Data Parameters
NAME          Mar22-2016
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160322
Time              16.38
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT           DMSO
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ           4.0894465 sec
RG                54.6
DW              62.400 usec
DE               6.50 usec
TE              295.0 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1               1H
P1               9.50 usec
PLW1        15.80000019 W

F2 - Processing parameters
SI                65536
SF          400.1300034 MHz
WDW                EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```

22_13C



BRUKER

```
Current Data Parameters
NAME          Mar24-2016
EXPNO                  1
PROCNO                 1

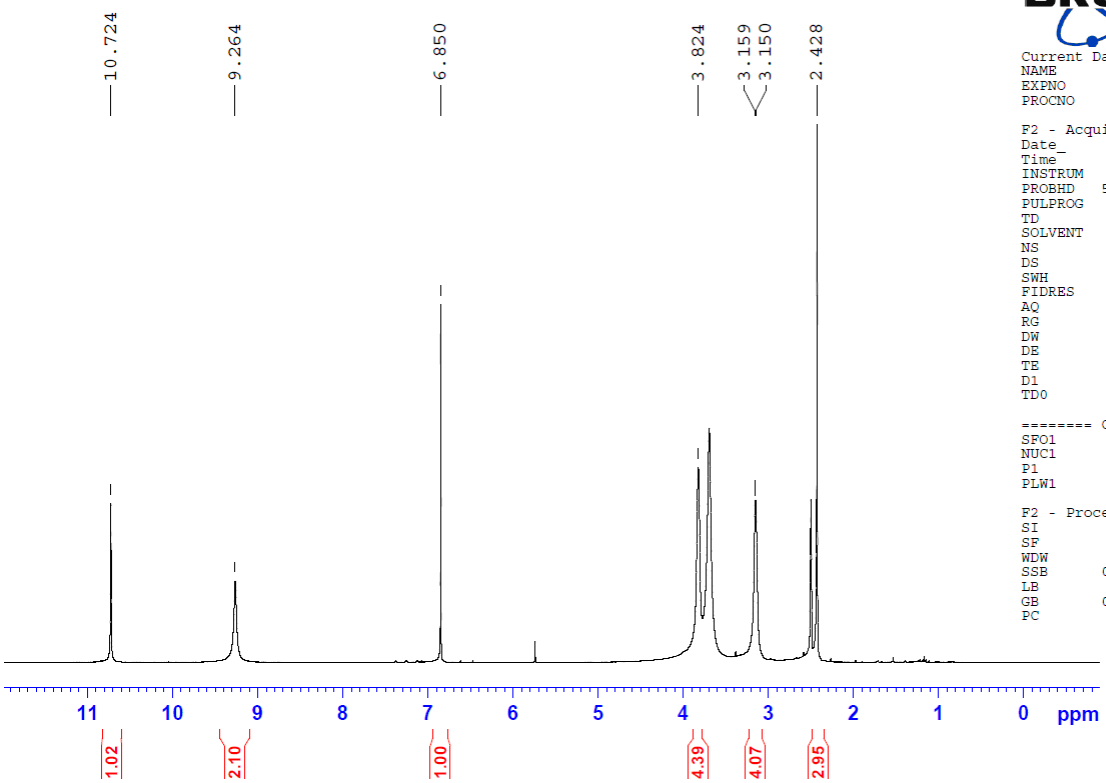
F2 - Acquisition Parameters
Date_           20160324
Time              16.31
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT           DMSO
NS                8192
DS                   4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ           1.3631488 sec
RG              199.94
DW              20.800 usec
DE               6.50 usec
TE              295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1               13C
P1               9.50 usec
PLW1        66.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2           90.00 usec
PLW2        18.00000000 W
PLW12        0.20056000 W
PLW13        0.16245000 W

F2 - Processing parameters
SI                32768
SF          100.6128074 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```

23_1H



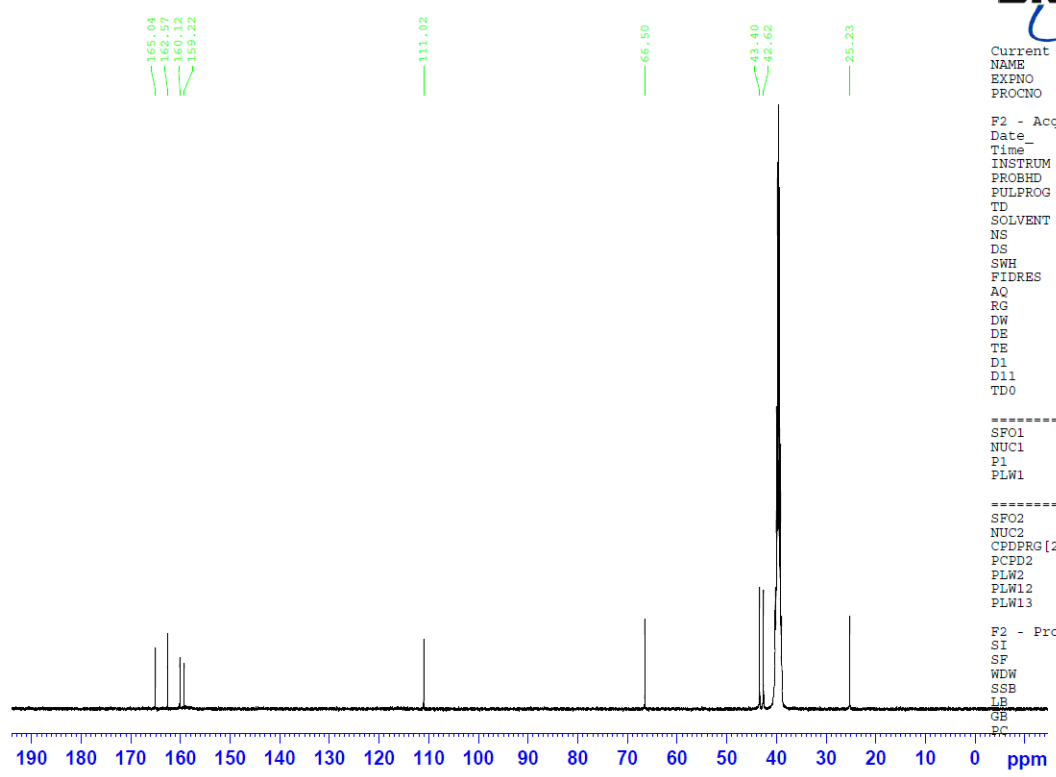

```
Current Data Parameters
NAME          Mar23-2016
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160323
Time              21.16
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                 54.6
DW               62.400 usec
DE                6.50 usec
TE                295.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                   9.50 usec
PLW1         15.80000019 W

F2 - Processing parameters
SI                 65536
SF           400.1300034 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

23_13C




```
Current Data Parameters
NAME          Mar23-2016
EXPNO                  4
PROCNO                 1

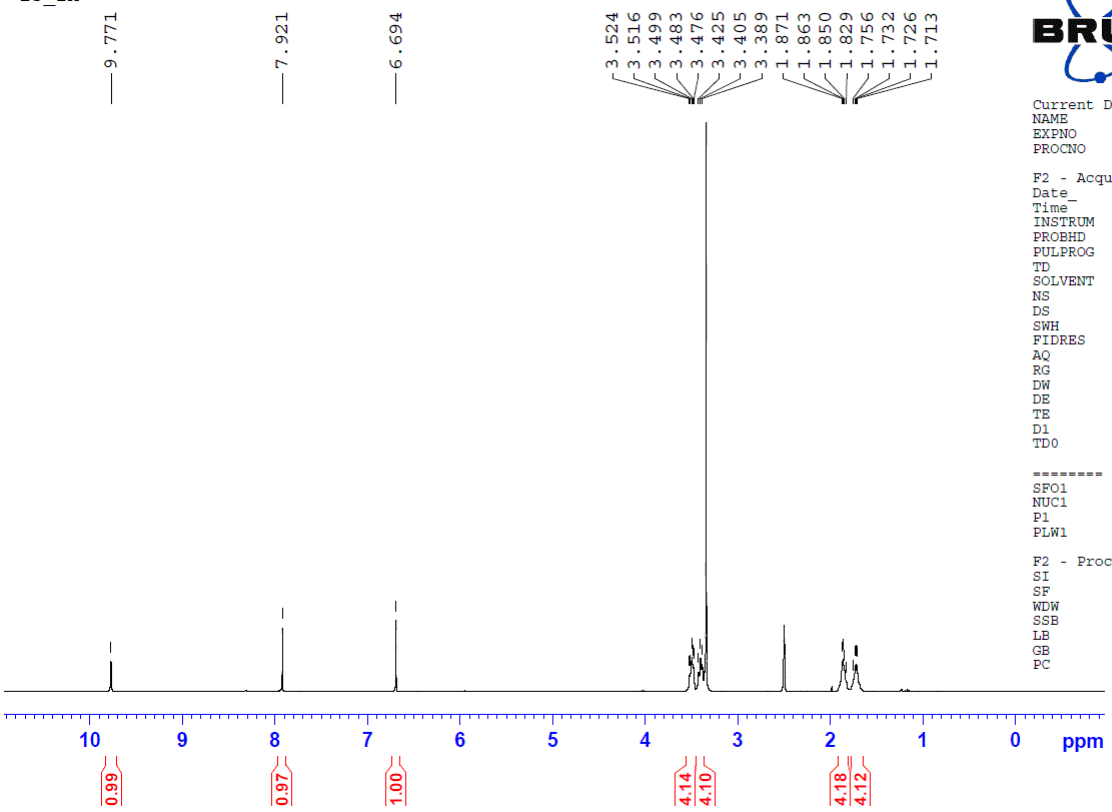
F2 - Acquisition Parameters
Date_           20160323
Time              23.23
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 2048
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                199.94
DW               20.800 usec
DE                6.50 usec
TE                295.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                  13C
P1                   9.50 usec
PLW1         66.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         18.00000000 W
PLW12         0.20056000 W
PLW13         0.16245000 W

F2 - Processing parameters
SI                 32768
SF           100.6128091 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

25_1H



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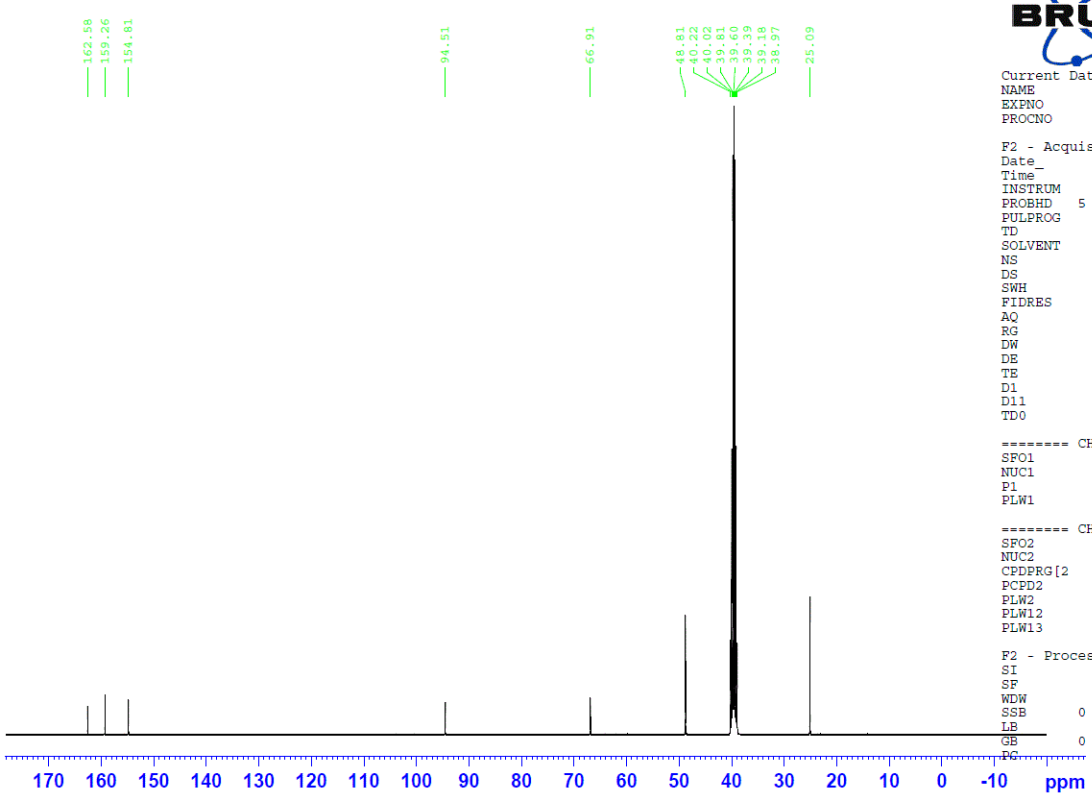
Current Data Parameters
NAME Jan08-2016
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160108
Time 17.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 78.4
DW 62.400 usec
DE 6.50 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 9.50 usec
PLW1 15.80000019 W

F2 - Processing parameters
SI 65536
SF 400.1300031 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

25_13C



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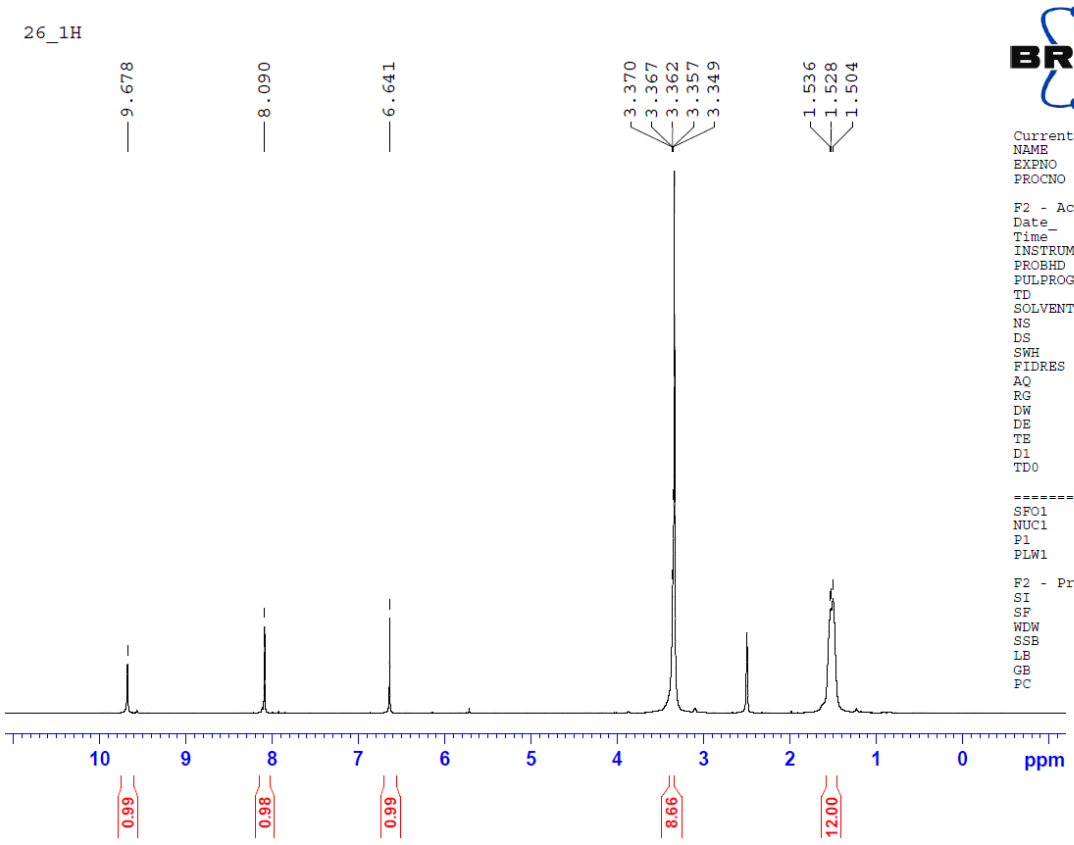
Current Data Parameters
NAME Jan08-2016
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160109
Time 1.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 8192
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 199.94
DW 20.800 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.50 usec
PLW1 66.50000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 18.00000000 W
PLW12 0.20056000 W
PLW13 0.16245000 W

F2 - Processing parameters
SI 32768
SF 100.6128054 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

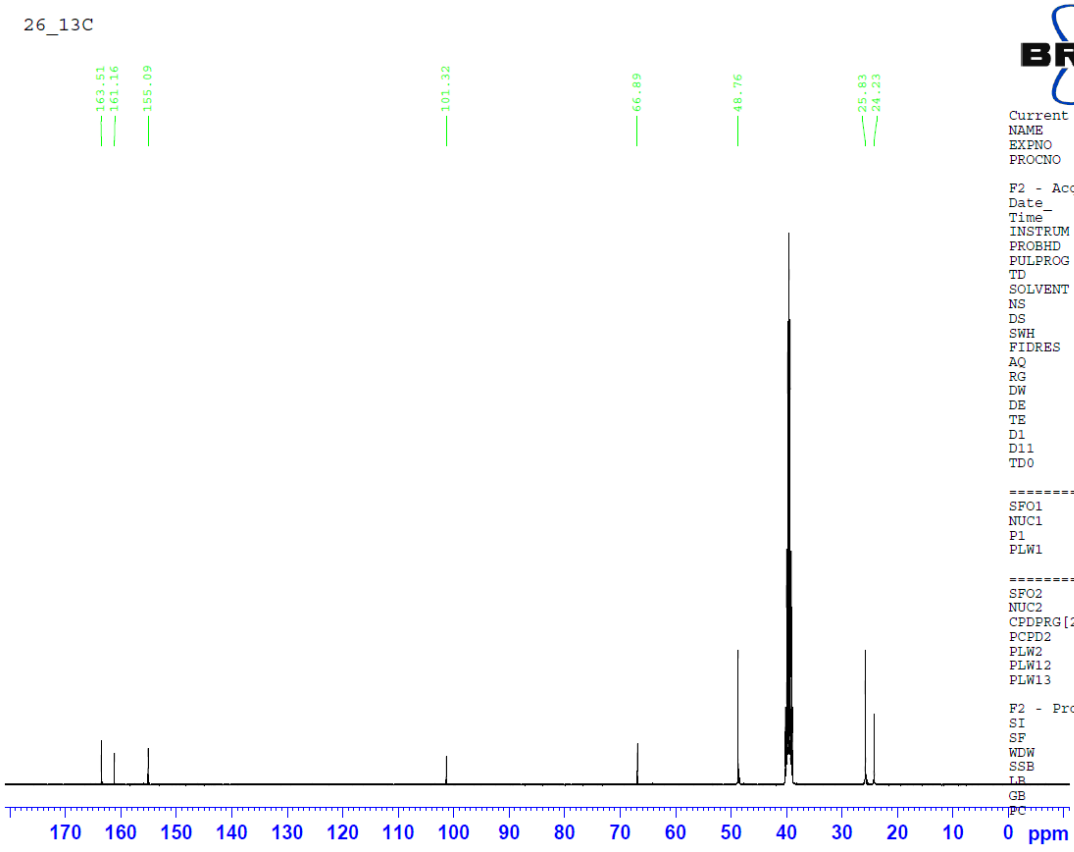



```
Current Data Parameters
NAME          Apr16-2016
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160416
Time              15.42
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT           DMSO
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG                60.77
DW              62.400 usec
DE               6.50 usec
TE              295.0 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                9.50 usec
PLW1        15.80000019 W

F2 - Processing parameters
SI                65536
SF          400.1300033 MHz
WDW                  EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
```



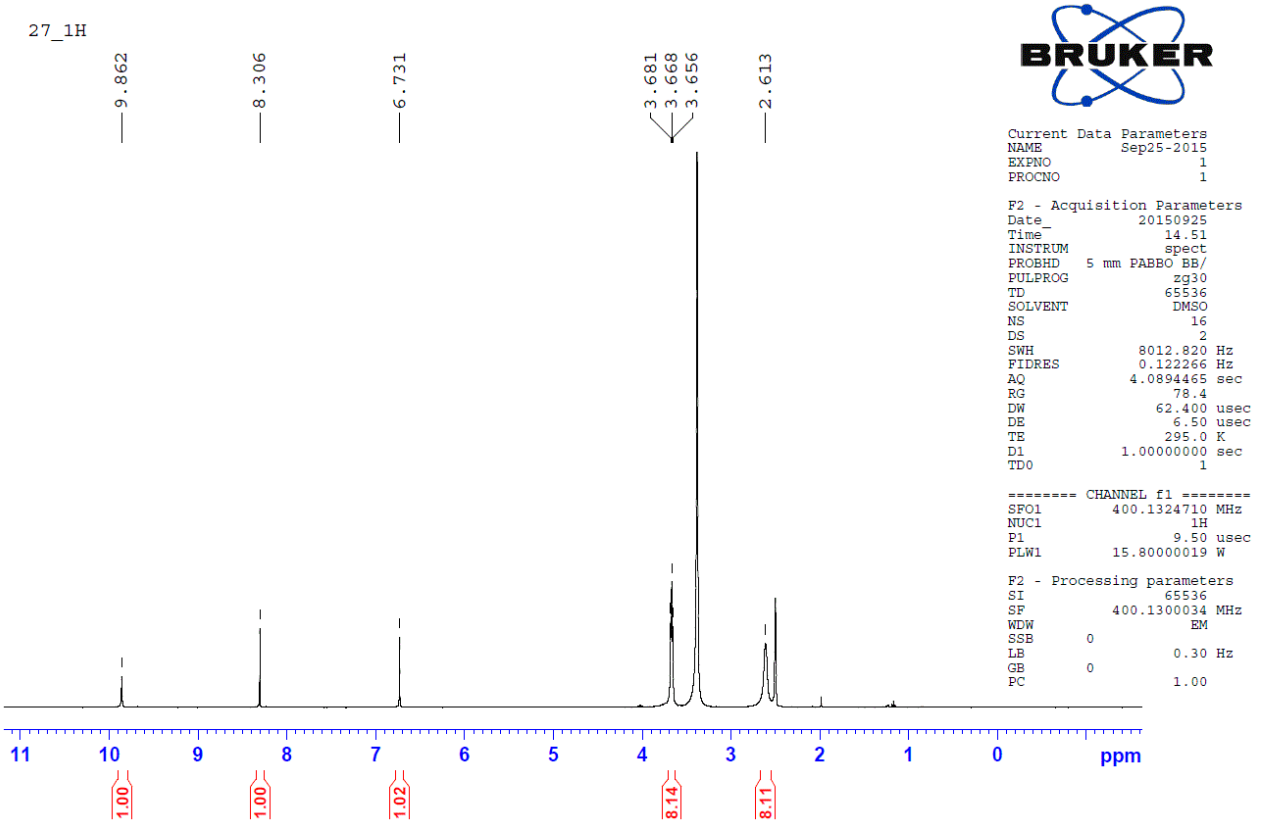

```
Current Data Parameters
NAME          Apr16-2016
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160416
Time              21.11
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT           DMSO
NS                5699
DS                   4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               199.94
DW              20.800 usec
DE               6.50 usec
TE              295.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                13C
P1                9.50 usec
PLW1        66.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2           90.00 usec
PLW2        18.00000000 W
PLW12        0.20056000 W
PLW13        0.16245000 W

F2 - Processing parameters
SI                32768
SF          100.6128060 MHz
WDW                  EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```

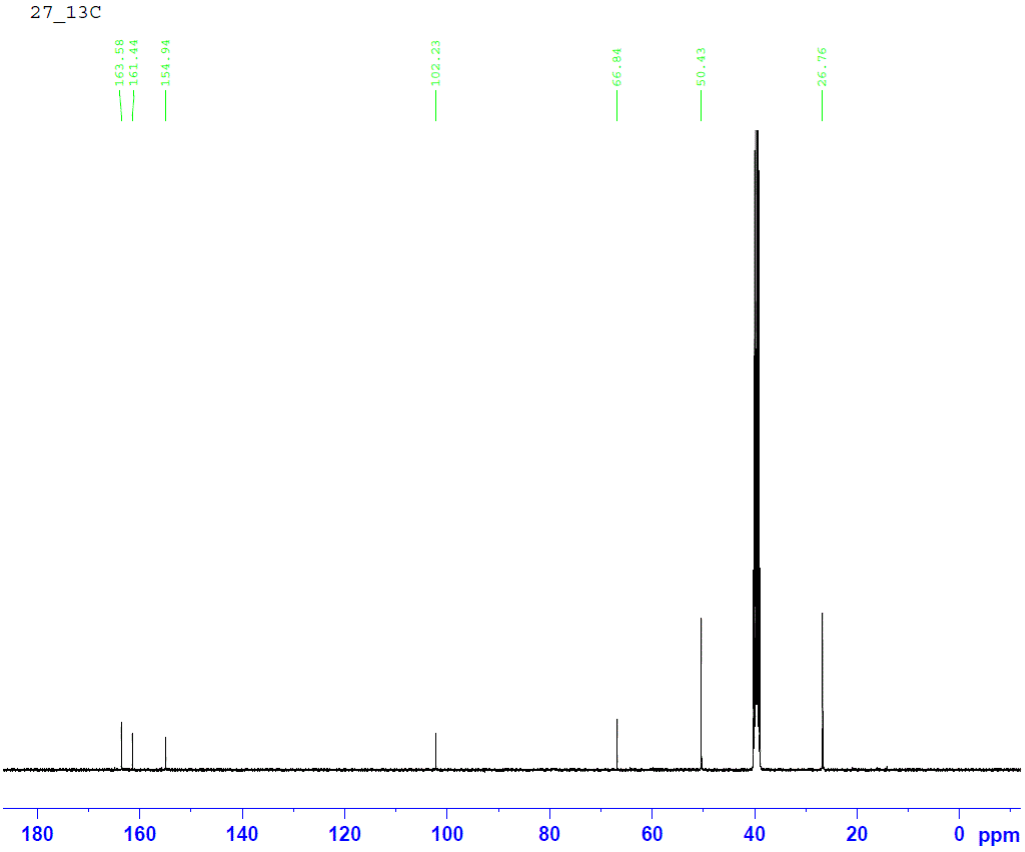



```
Current Data Parameters
NAME          Sep25-2015
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150925
Time_              14.51
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                  78.4
DW                62.400 usec
DE                 6.50 usec
TE                295.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                  9.50 usec
PLW1          15.80000019 W

F2 - Processing parameters
SI                 65536
SF            400.1300034 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



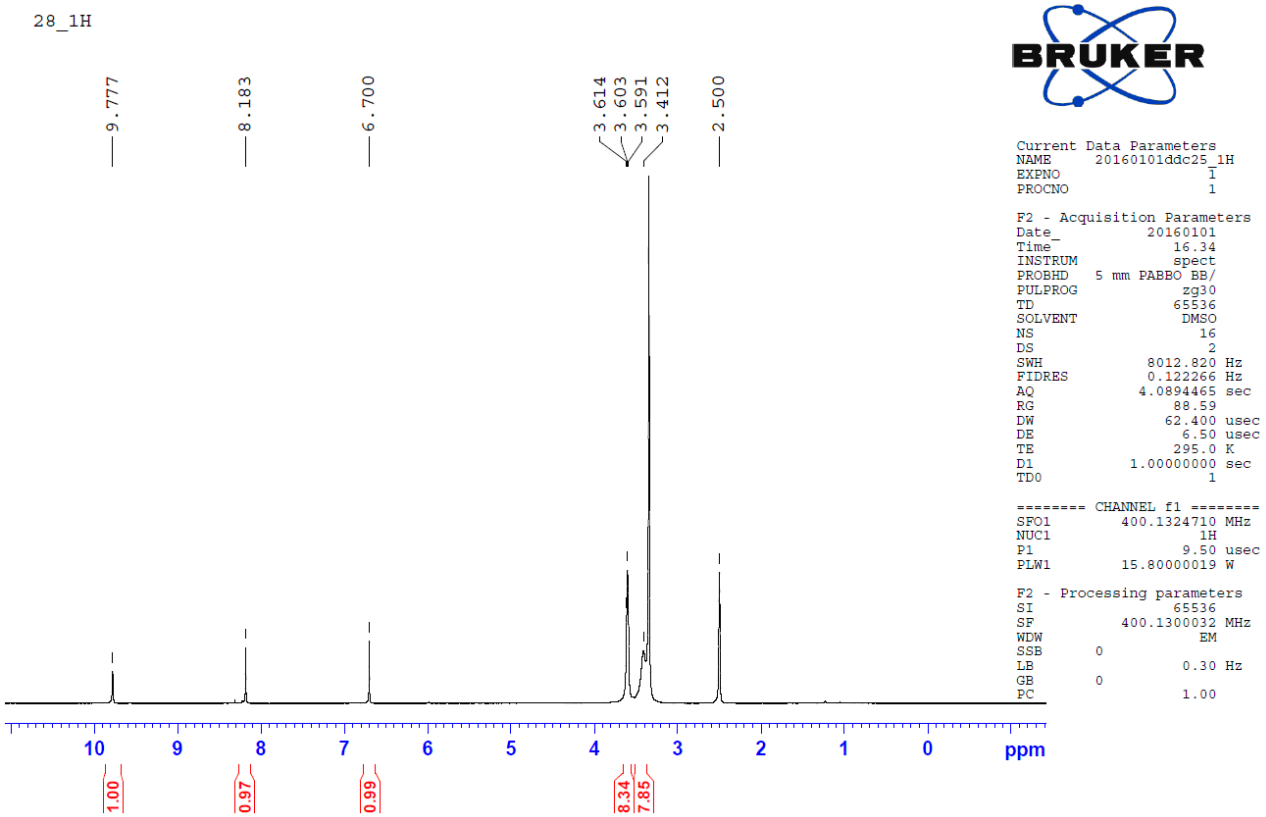

```
Current Data Parameters
NAME          Sep25-2015
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150925
Time               22.40
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                 65536
SOLVENT             DMSO
NS                  8192
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                199.94
DW                20.800 usec
DE                 6.50 usec
TE                295.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228293 MHz
NUC1                  13C
P1                  9.50 usec
PLW1          66.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          18.00000000 W
PLW12          0.20056000 W
PLW13          0.16245000 W

F2 - Processing parameters
SI                 32768
SF            100.6128045 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

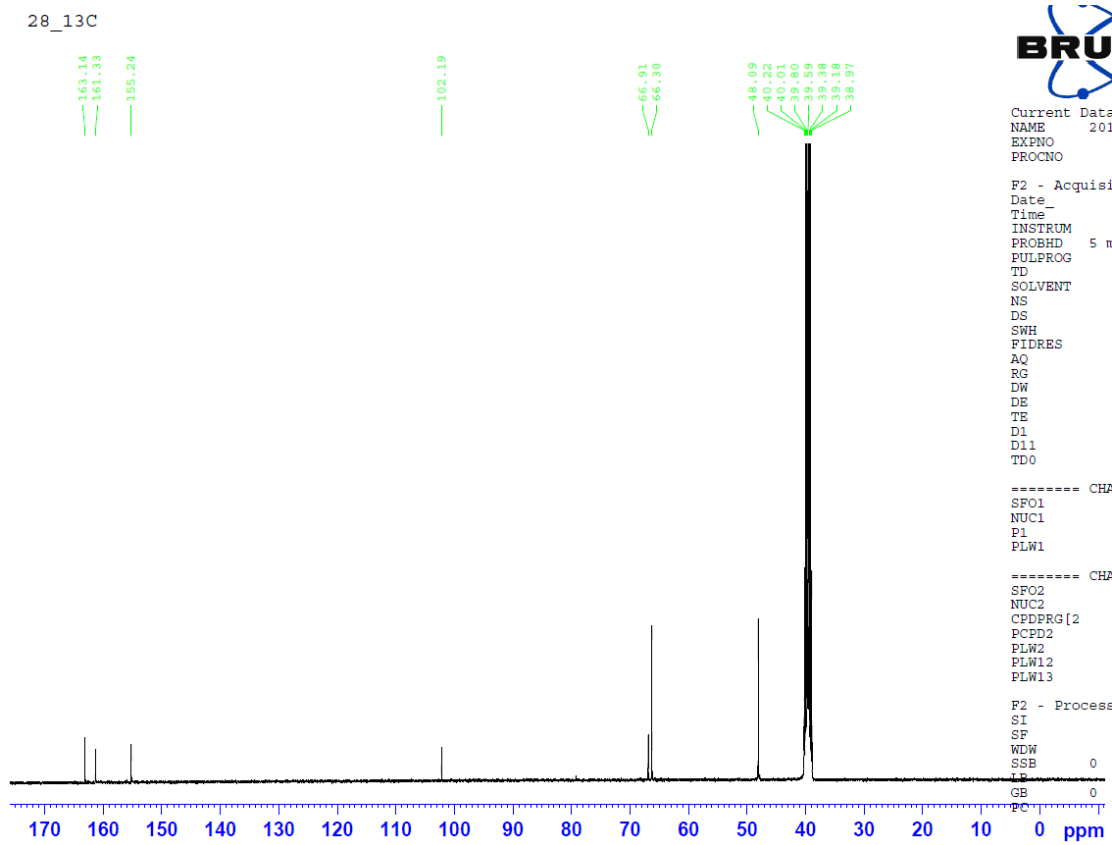



```
Current Data Parameters
NAME      20160101ddc25_1H
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160101
Time              16.34
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT           DMSO
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG                88.59
DW              62.400 usec
DE               6.50 usec
TE              295.0 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1               1H
P1                9.50 usec
PLW1       15.80000019 W

F2 - Processing parameters
SI               65536
SF          400.1300032 MHz
WDW                EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
```




```
Current Data Parameters
NAME      20160101ddc25_13C
EXPNO                    1
PROCNO                   1

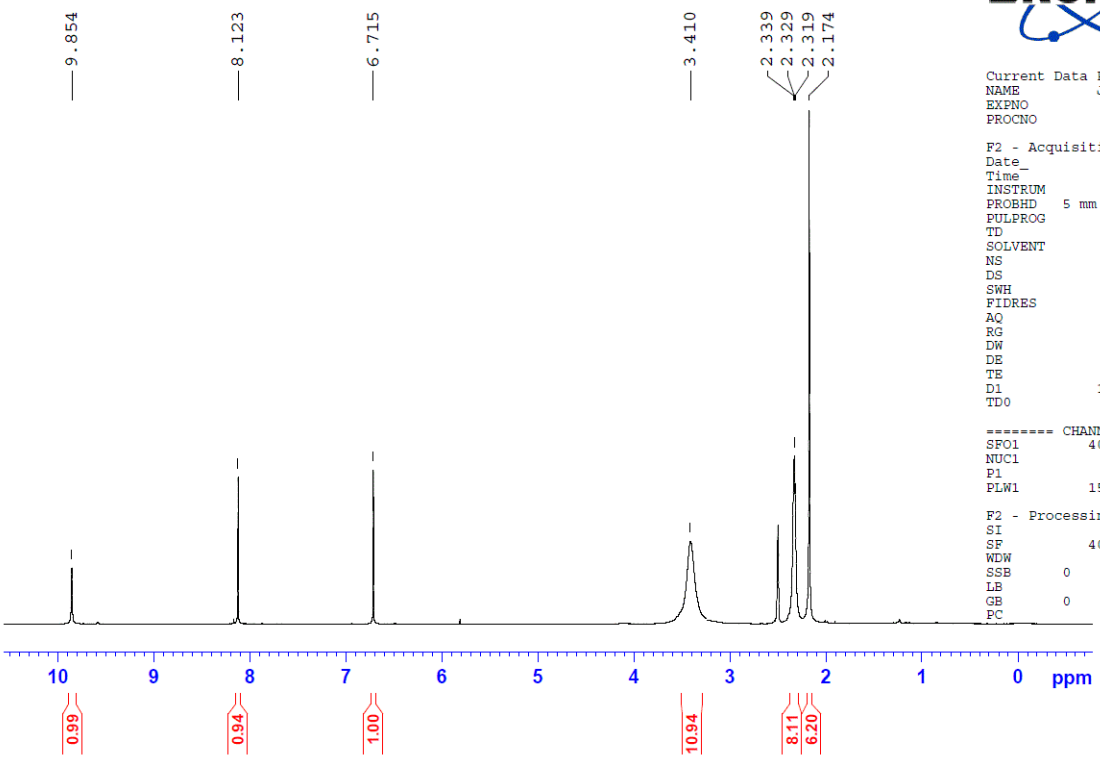
F2 - Acquisition Parameters
Date_           20160102
Time              0.32
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT           DMSO
NS                8192
DS                   4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               199.94
DW              20.800 usec
DE               6.50 usec
TE              295.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1               13C
P1                9.50 usec
PLW1       66.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2       18.00000000 W
PLW12       0.20056000 W
PLW13       0.16245000 W

F2 - Processing parameters
SI               32768
SF          100.6128054 MHz
WDW                EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```

29_1H



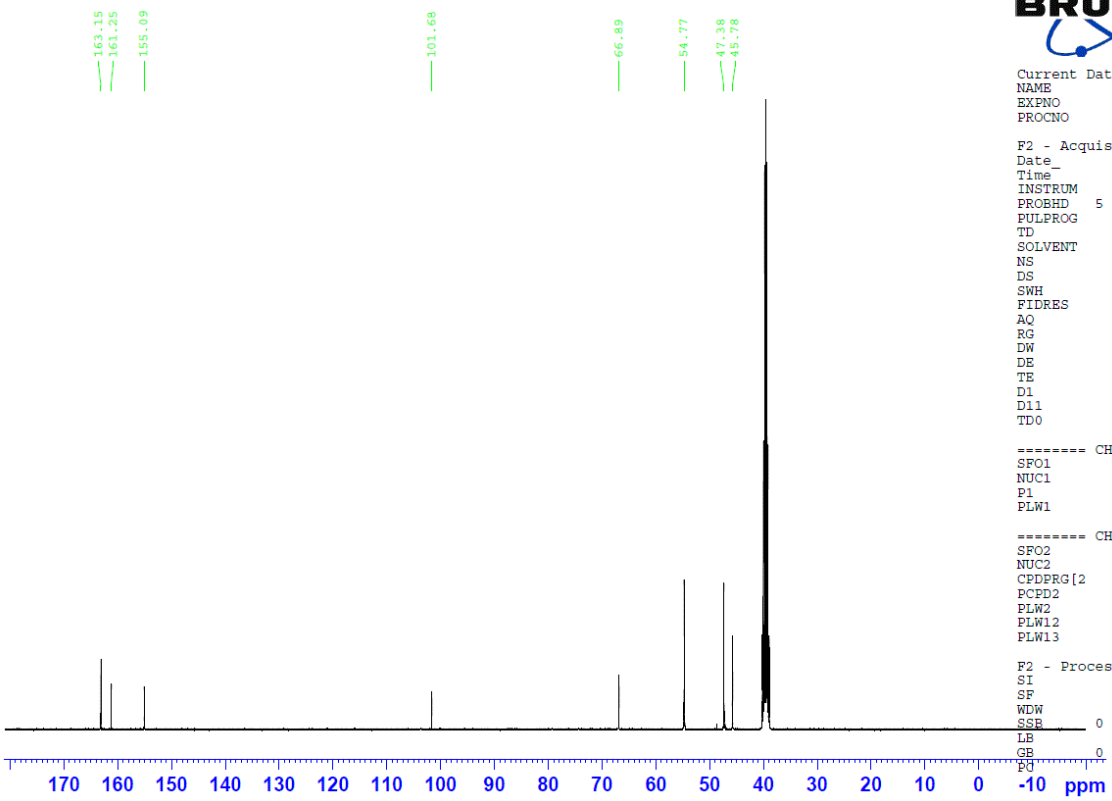

```
Current Data Parameters
NAME          Jan05-2016
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160105
Time              22.16
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                 60.77
DW               62.400 usec
DE                6.50 usec
TE                295.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                   9.50 usec
PLW1         15.80000019 W

F2 - Processing parameters
SI                 65536
SF           400.1300030 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

29_13C




```
Current Data Parameters
NAME          Jan06-2016
EXPNO                  2
PROCNO                 1

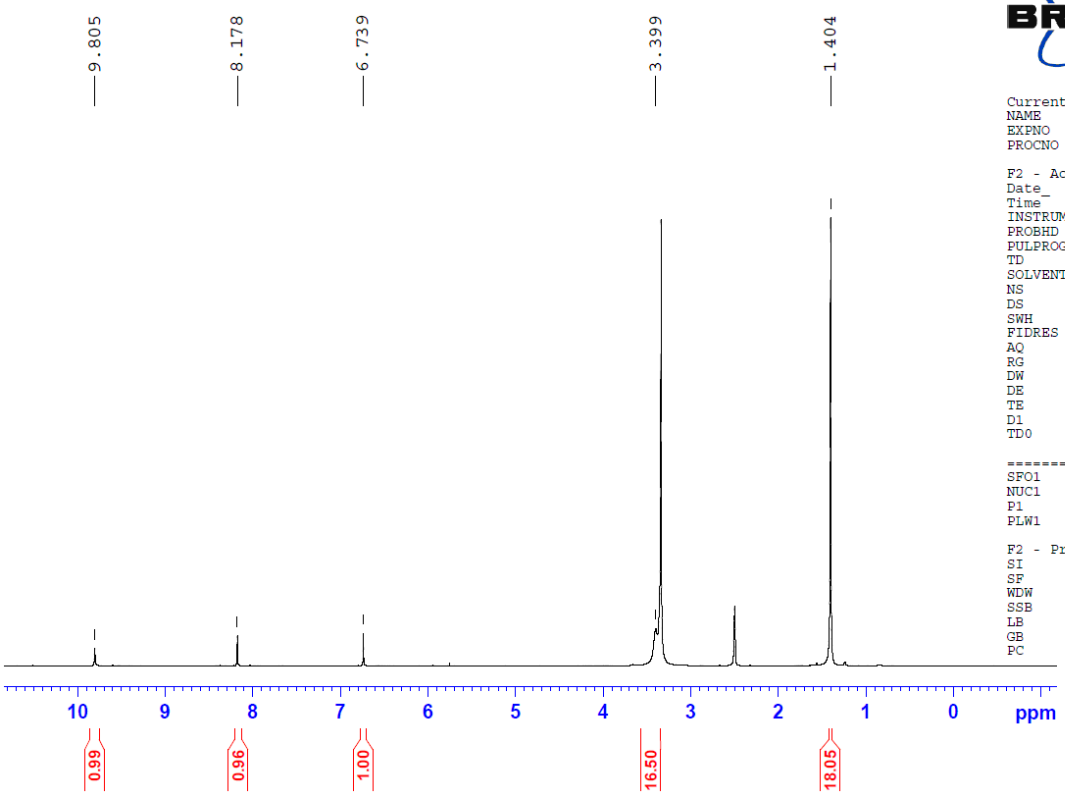
F2 - Acquisition Parameters
Date_           20160106
Time              14.41
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                  4096
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                199.94
DW               20.800 usec
DE                6.50 usec
TE                295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                  13C
P1                   9.50 usec
PLW1         66.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2         18.00000000 W
PLW12         0.20056000 W
PLW13         0.16245000 W

F2 - Processing parameters
SI                 32768
SF           100.6128062 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

30_1H



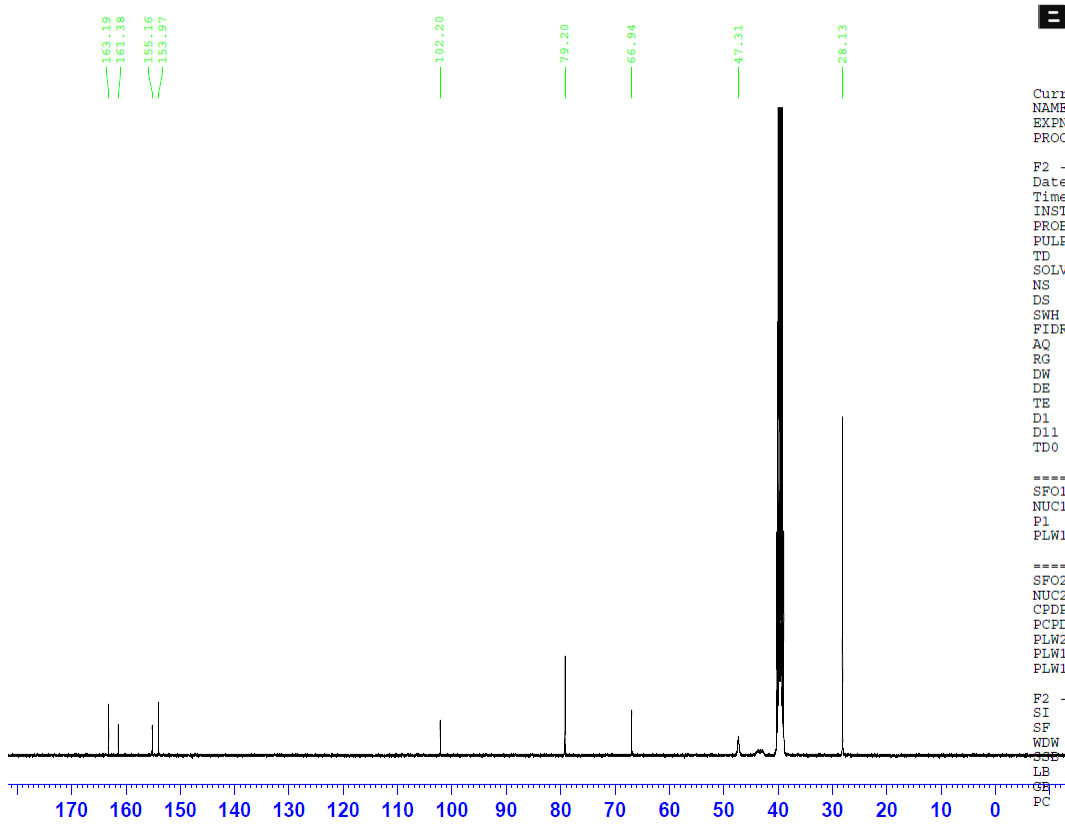

```
Current Data Parameters
NAME          Mar22-2016
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160322
Time               17.13
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                  78.4
DW                62.400 usec
DE                 6.50 usec
TE                295.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                   1H
P1                  9.50 usec
PLW1          15.80000019 W

F2 - Processing parameters
SI                 65536
SF            400.1300035 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

30_13C




```
Current Data Parameters
NAME          Mar22-2016
EXPNO                  5
PROCNO                 1

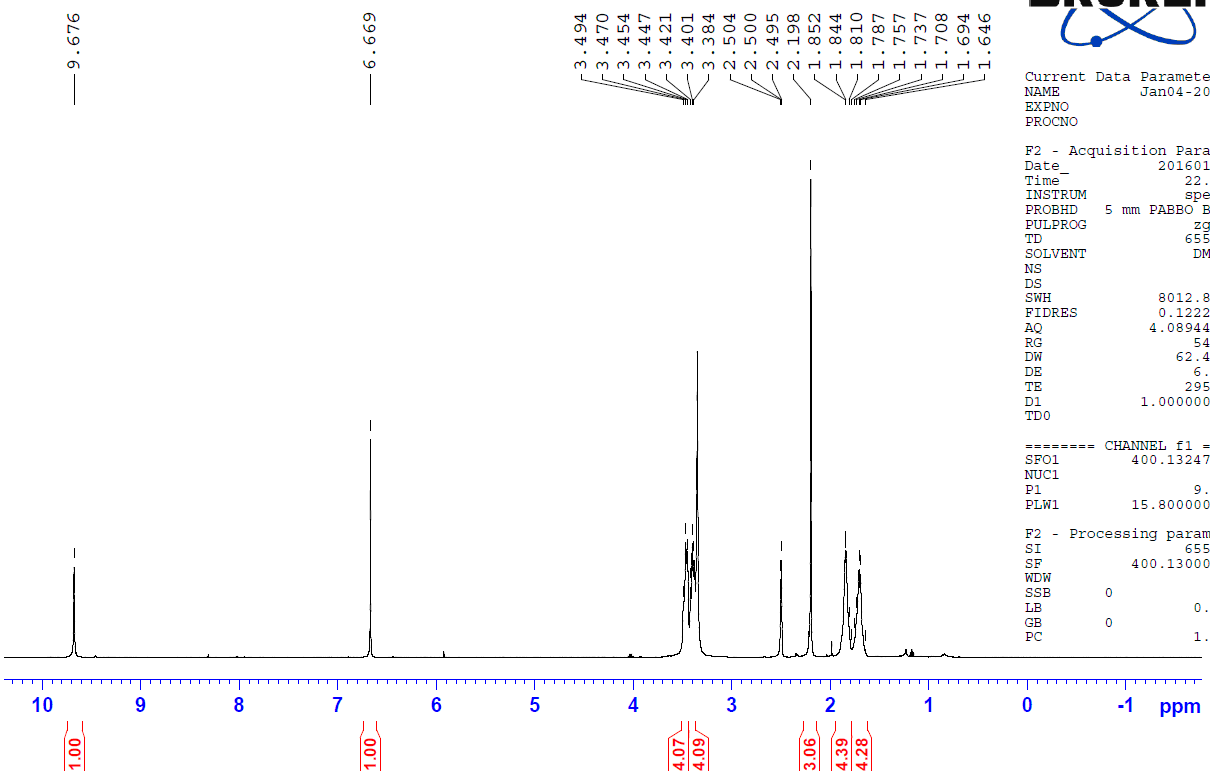
F2 - Acquisition Parameters
Date_           20160323
Time               1.02
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                 65536
SOLVENT             DMSO
NS                  8192
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                199.94
DW                20.800 usec
DE                 6.50 usec
TE                295.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228293 MHz
NUC1                  13C
P1                  9.50 usec
PLW1          66.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          18.00000000 W
PLW12          0.20056000 W
PLW13          0.16245000 W

F2 - Processing parameters
SI                 32768
SF            100.6128060 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

32_1H



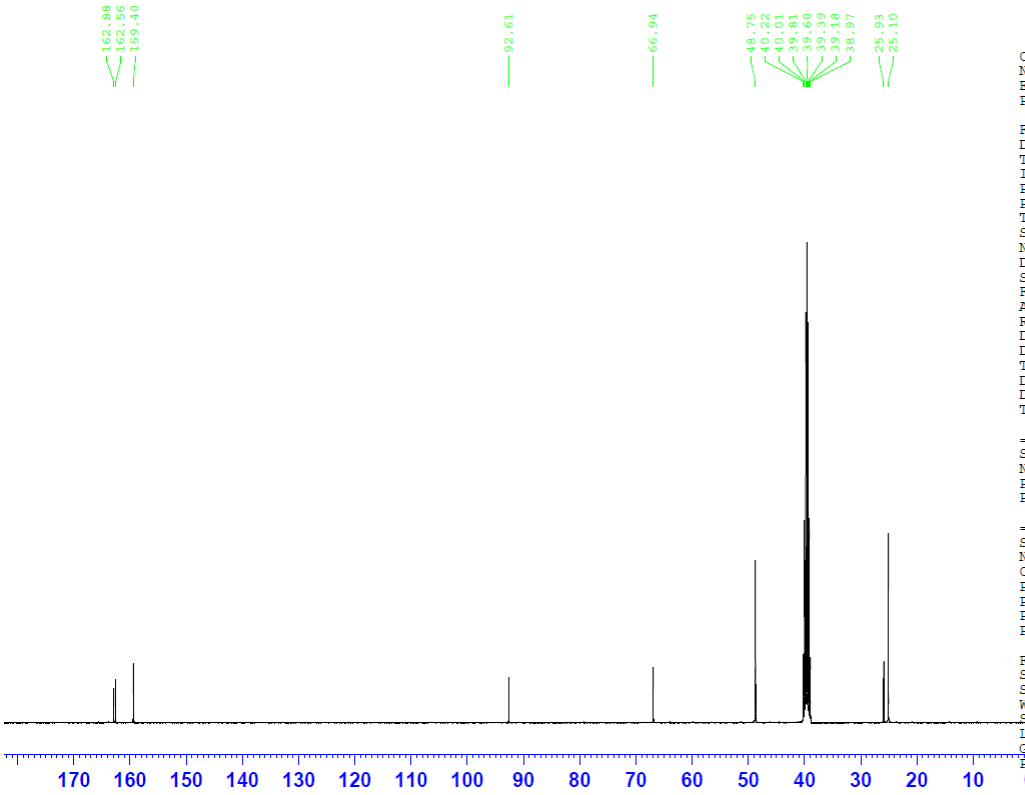

```
Current Data Parameters
NAME          Jan04-2016
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160104
Time               22.17
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                  54.6
DW                62.400 usec
DE                 6.50 usec
TE                295.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  9.50 usec
PLW1         15.80000019 W

F2 - Processing parameters
SI                 65536
SF          400.1300031 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

32_13C




```
Current Data Parameters
NAME          Jan04-2016
EXPNO                 11
PROCNO                 1

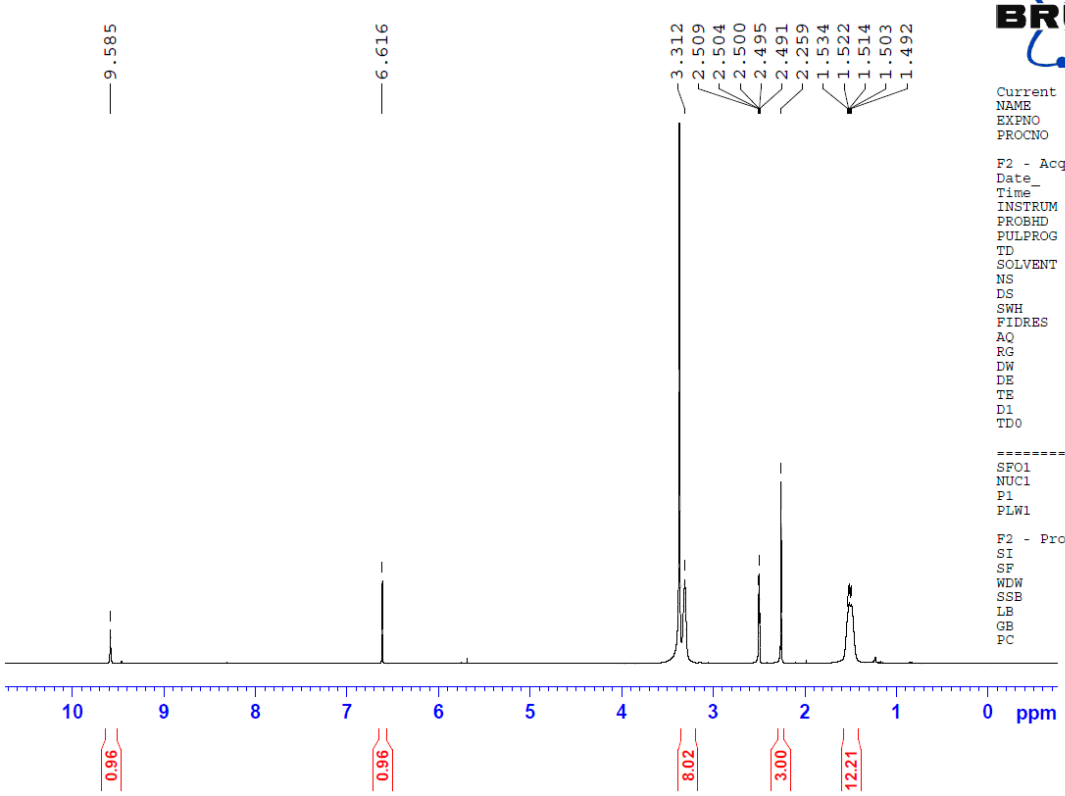
F2 - Acquisition Parameters
Date_           20160105
Time                6.14
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                 65536
SOLVENT             DMSO
NS                  8192
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                199.94
DW                20.800 usec
DE                 6.50 usec
TE                295.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                  13C
P1                  9.50 usec
PLW1         66.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         18.00000000 W
PLW12         0.20056000 W
PLW13         0.16245000 W

F2 - Processing parameters
SI                 32768
SF          100.6128055 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

33_1H



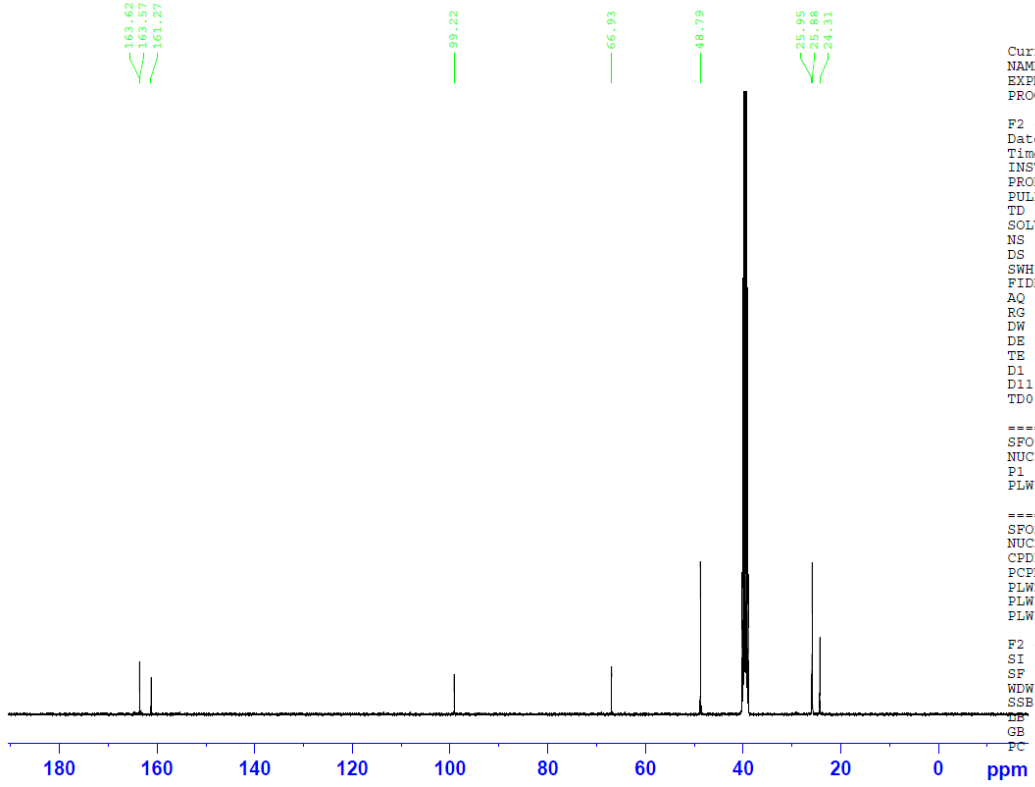

```
Current Data Parameters
NAME          Oct05-2015
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151005
Time              18.18
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                   64
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                71.13
DW              62.400 usec
DE               6.50 usec
TE              295.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                9.50 usec
PLW1         15.80000019 W

F2 - Processing parameters
SI                65536
SF           400.1300037 MHz
WDW                  EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
```

33_13C




```
Current Data Parameters
NAME          Oct05-2015
EXPNO                  3
PROCNO                 1

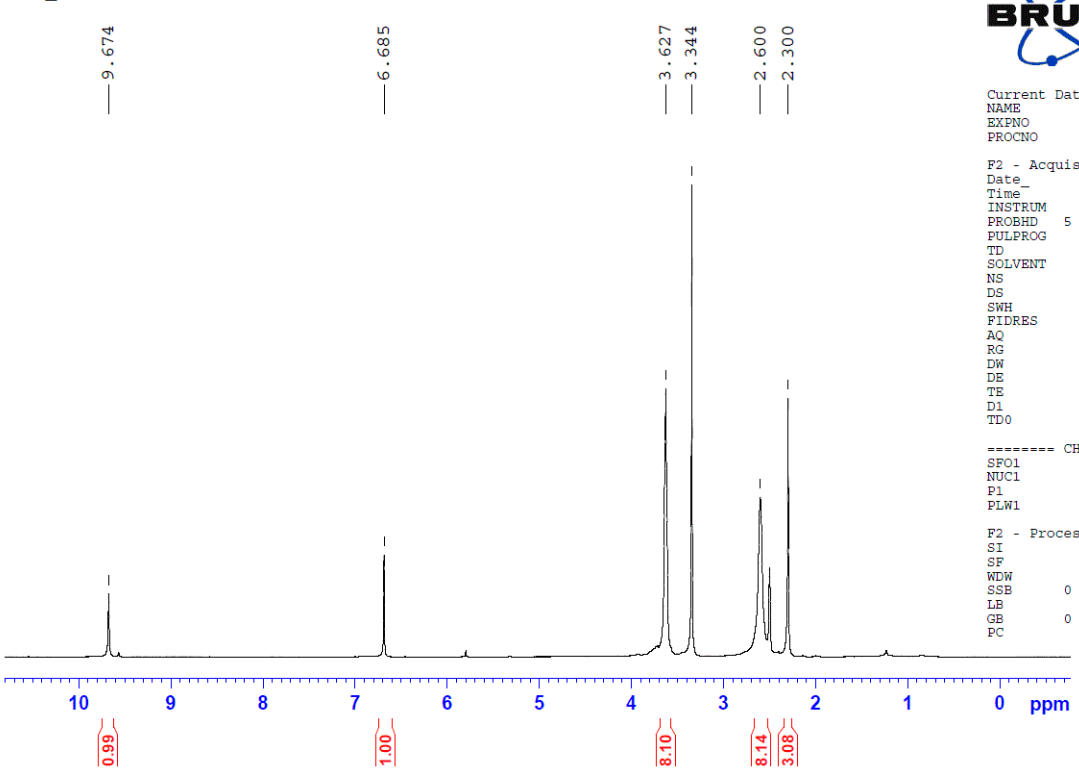
F2 - Acquisition Parameters
Date_           20151005
Time              22.13
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                199.94
DW              20.800 usec
DE               6.50 usec
TE              295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                 13C
P1                9.50 usec
PLW1         66.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         18.00000000 W
PLW12         0.20056000 W
PLW13         0.16245000 W

F2 - Processing parameters
SI                32768
SF           100.6128033 MHz
WDW                  EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```

34_1H



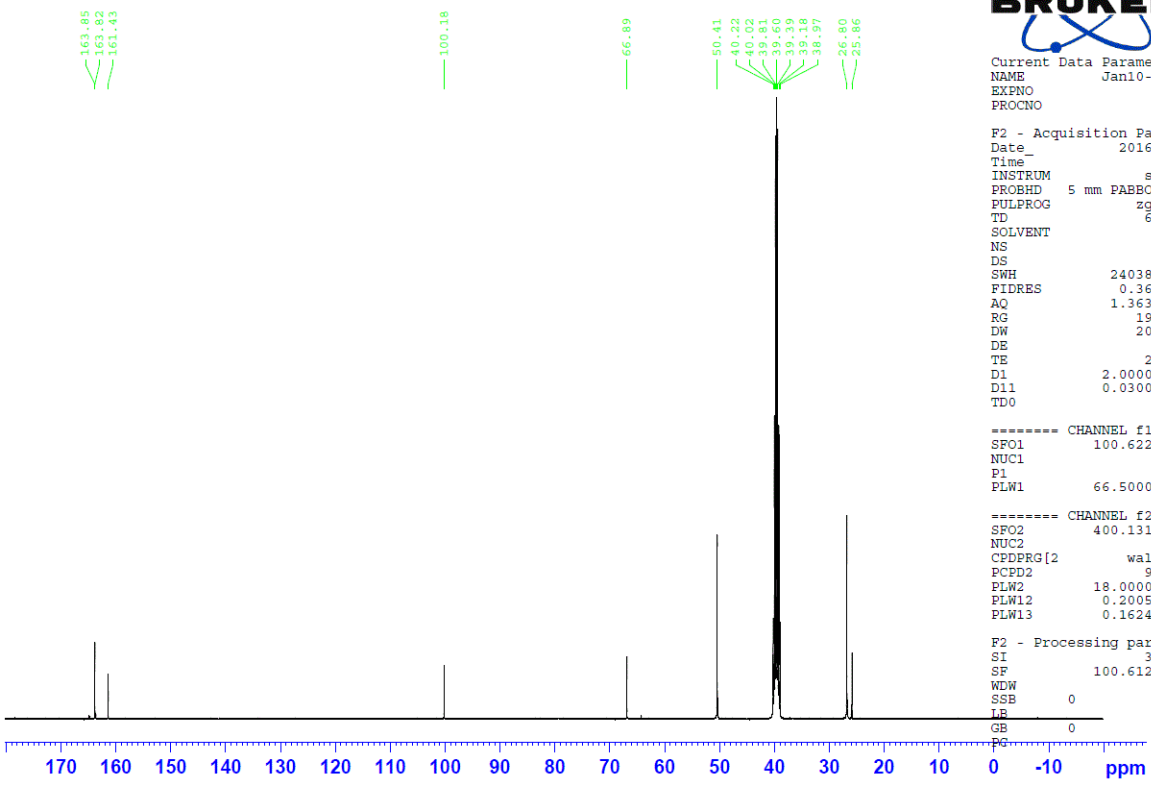

```
Current Data Parameters
NAME          Jan10-2016
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160110
Time              19.13
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                   64
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                60.77
DW              62.400 usec
DE               6.50 usec
TE               295.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                9.50 usec
PLW1         15.80000019 W

F2 - Processing parameters
SI                65536
SF           400.1300035 MHz
WDW                  EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
```

34_13C




```
Current Data Parameters
NAME          Jan10-2016
EXPNO                  3
PROCNO                 1

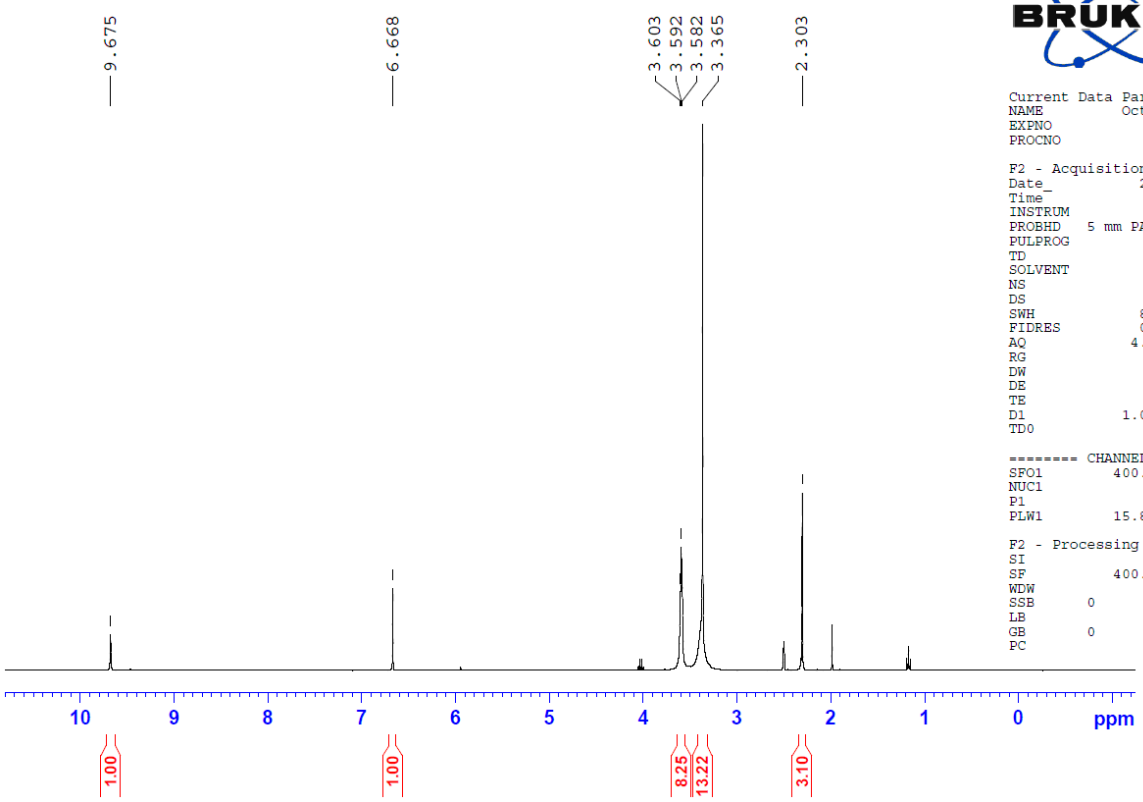
F2 - Acquisition Parameters
Date_           20160111
Time              3.09
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 8192
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                199.94
DW              20.800 usec
DE               6.50 usec
TE               295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                 13C
P1                9.50 usec
PLW1         66.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         18.00000000 W
PLW12         0.20056000 W
PLW13         0.16245000 W

F2 - Processing parameters
SI                32768
SF           100.6128058 MHz
WDW                  EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```

35_1H



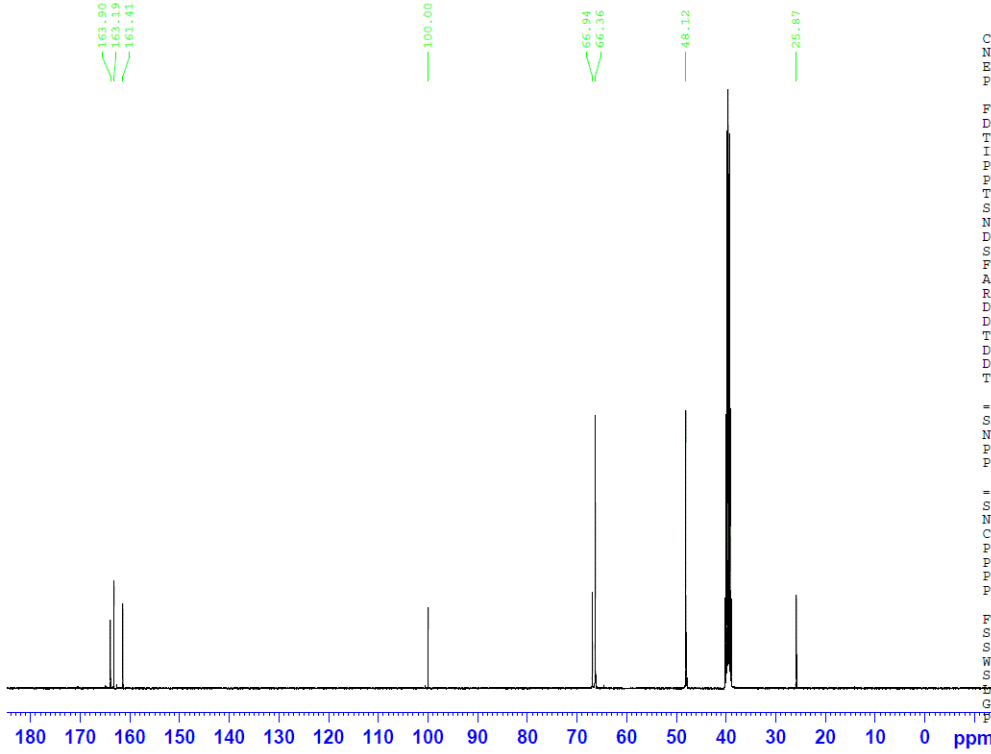

```
Current Data Parameters
NAME                 Oct06-2015
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20151006
Time                      15.02
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                   zg30
TD                       65536
SOLVENT                   DMSO
NS                          16
DS                           2
SWH                   8012.820 Hz
FIDRES                0.122266 Hz
AQ                    4.0894465 sec
RG                        31.12
DW                      62.400 usec
DE                        6.50 usec
TE                       295.0 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                400.1324710 MHz
NUC1                        1H
P1                        9.50 usec
PLW1                15.80000019 W

F2 - Processing parameters
SI                           65536
SF                  400.1300037 MHz
WDW                             EM
SSB                  0
LB                            0.30 Hz
GB                   0
PC                            1.00
```

35_13C



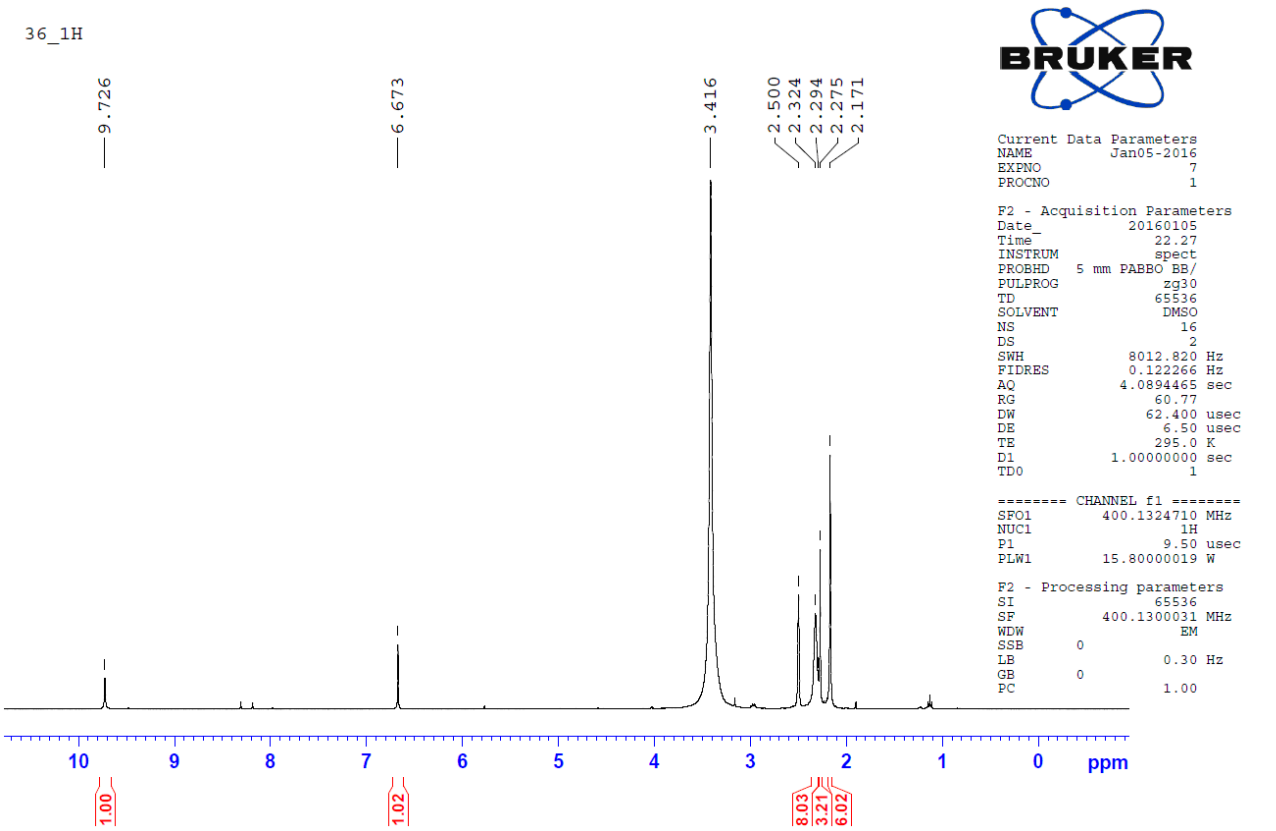

```
Current Data Parameters
NAME                 Oct06-2015
EXPNO                         4
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20151006
Time                      19.16
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                   DMSO
NS                         4096
DS                            4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631488 sec
RG                       199.94
DW                      20.800 usec
DE                        6.50 usec
TE                       295.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                100.6228293 MHz
NUC1                        13C
P1                        9.50 usec
PLW1                66.50000000 W

======== CHANNEL f2 ========
SFO2                400.1316005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     90.00 usec
PLW2                18.00000000 W
PLW12                0.20056000 W
PLW13                0.16245000 W

F2 - Processing parameters
SI                           32768
SF                  100.6128033 MHz
WDW                             EM
SSB                  0
LB                            1.00 Hz
GB                   0
PC                            1.40
```

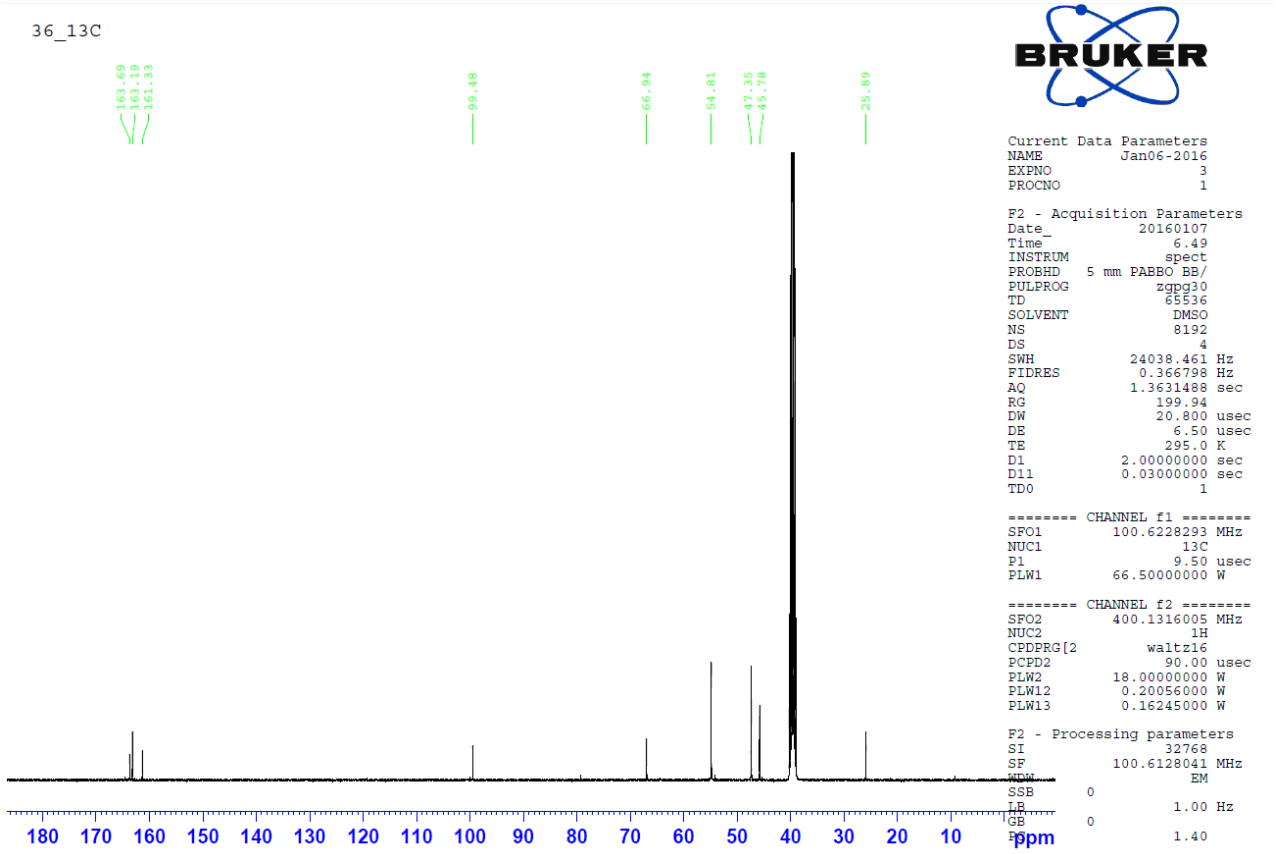



```
Current Data Parameters
NAME          Jan05-2016
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_              20160105
Time                 22.27
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                   60.77
DW                 62.400 usec
DE                  6.50 usec
TE                 295.0 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1           400.1324710 MHz
NUC1                   1H
P1                   9.50 usec
PLW1          15.80000019 W

F2 - Processing parameters
SI                  65536
SF             400.1300031 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```




```
Current Data Parameters
NAME          Jan06-2016
EXPNO                  3
PROCNO                 1

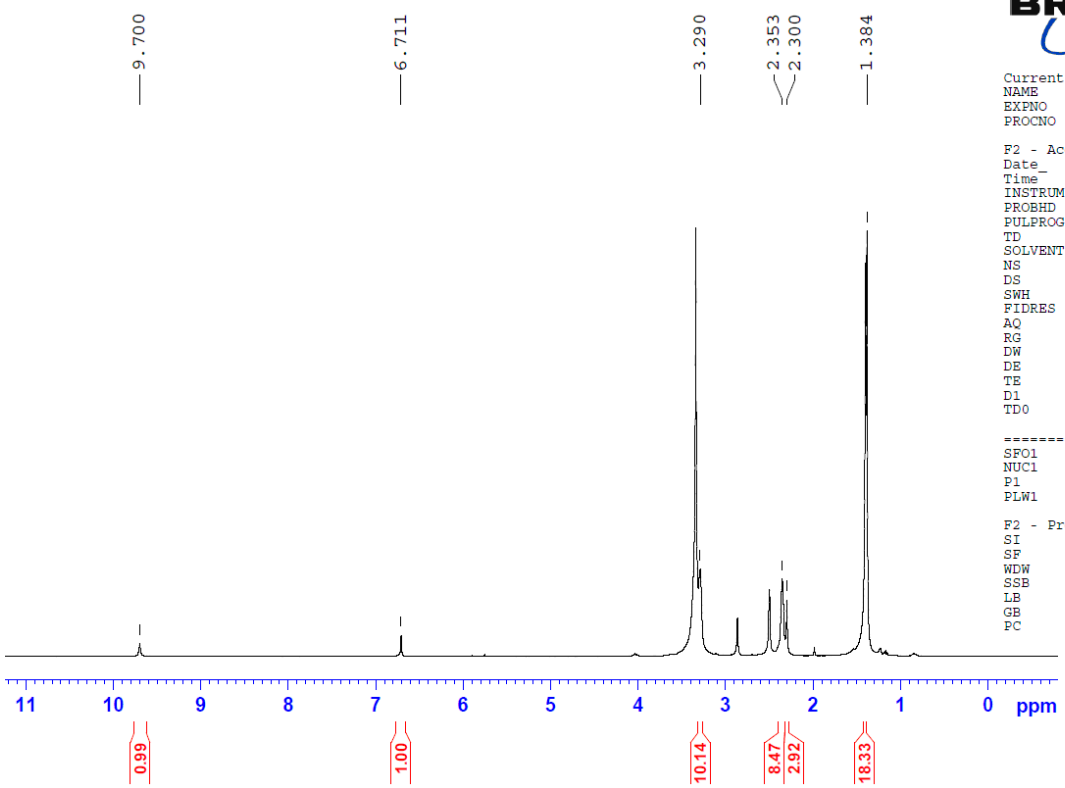
F2 - Acquisition Parameters
Date_              20160107
Time                  6.49
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT              DMSO
NS                   8192
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631488 sec
RG                  199.94
DW                 20.800 usec
DE                  6.50 usec
TE                 295.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1           100.6228293 MHz
NUC1                   13C
P1                   9.50 usec
PLW1          66.50000000 W

======== CHANNEL f2 ========
SFO2           400.1316005 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2               90.00 usec
PLW2          18.00000000 W
PLW12          0.20056000 W
PLW13          0.16245000 W

F2 - Processing parameters
SI                  32768
SF             100.6128041 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

37_1H



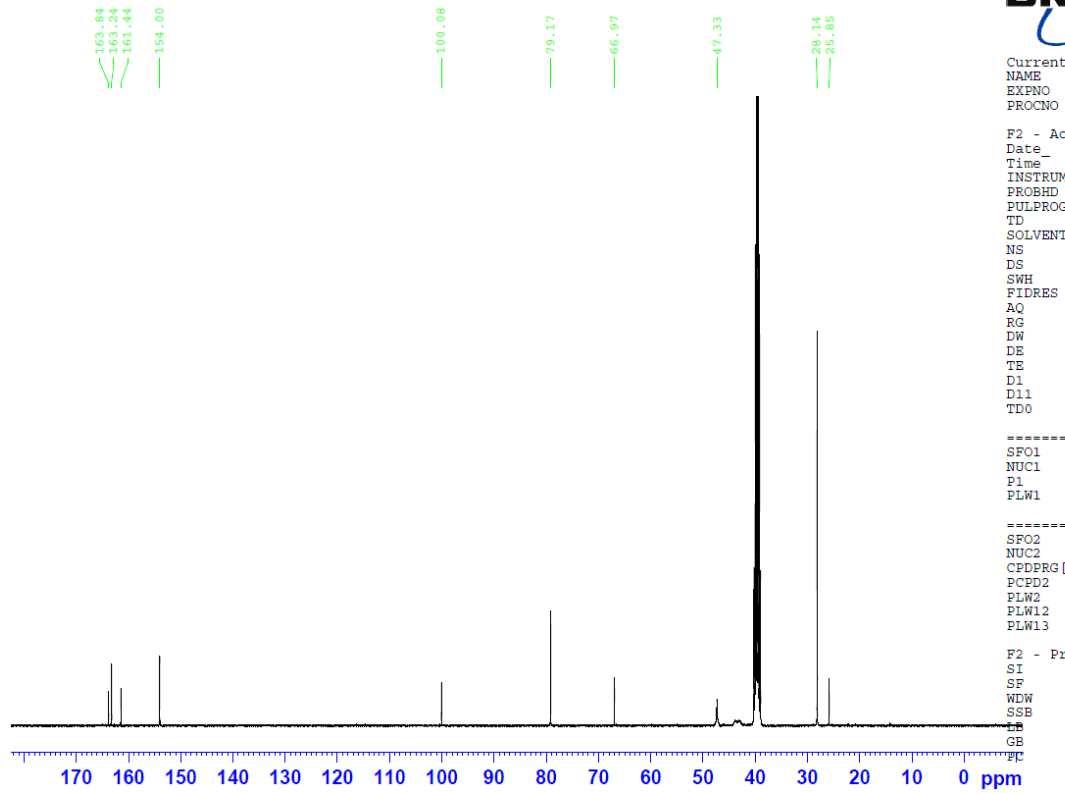

```
Current Data Parameters
NAME          Mar22-2016
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160322
Time              16.50
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT           DMSO
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG                54.6
DW              62.400 usec
DE               6.50 usec
TE              295.0 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                9.50 usec
PLW1        15.80000019 W

F2 - Processing parameters
SI               65536
SF          400.1300039 MHz
WDW                 EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
```

37_13C




```
Current Data Parameters
NAME          Mar17-2016
EXPNO                 10
PROCNO                 1

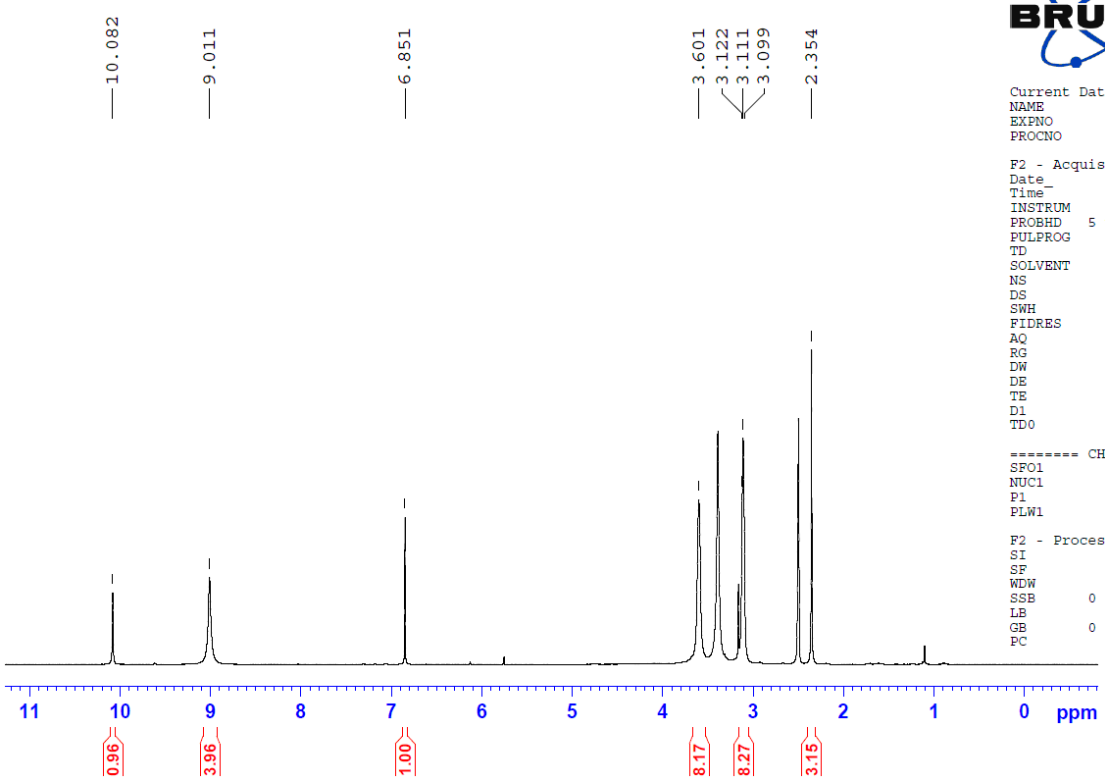
F2 - Acquisition Parameters
Date_           20160318
Time              1.39
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT           DMSO
NS                 8192
DS                    4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               199.94
DW              20.800 usec
DE               6.50 usec
TE              295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                13C
P1                9.50 usec
PLW1        66.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        18.00000000 W
PLW12        0.20056000 W
PLW13        0.16245000 W

F2 - Processing parameters
SI               32768
SF          100.6128052 MHz
WDW                 EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```

38_1H



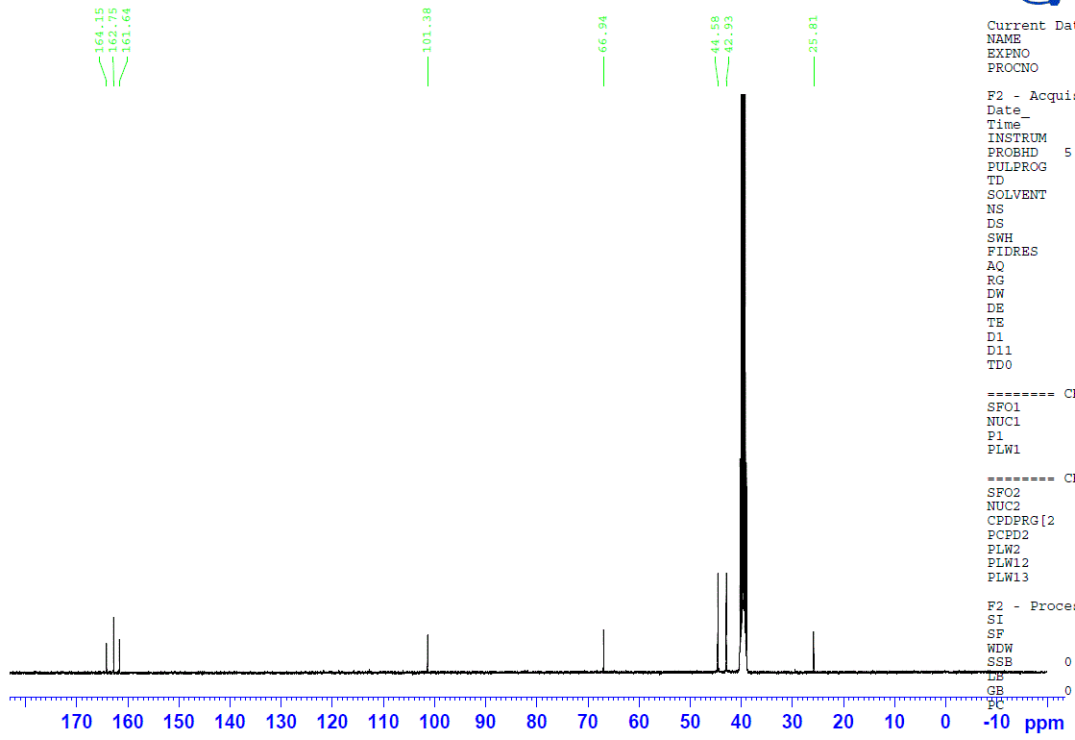

```
Current Data Parameters
NAME          Mar31-2016
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160331
Time               9.45
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                   64
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                88.59
DW              62.400 usec
DE               6.50 usec
TE              295.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 9.50 usec
PLW1         15.80000019 W

F2 - Processing parameters
SI                 65536
SF           400.1300035 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

38_13C




```
Current Data Parameters
NAME          Apr16-2016
EXPNO                  3
PROCNO                 1

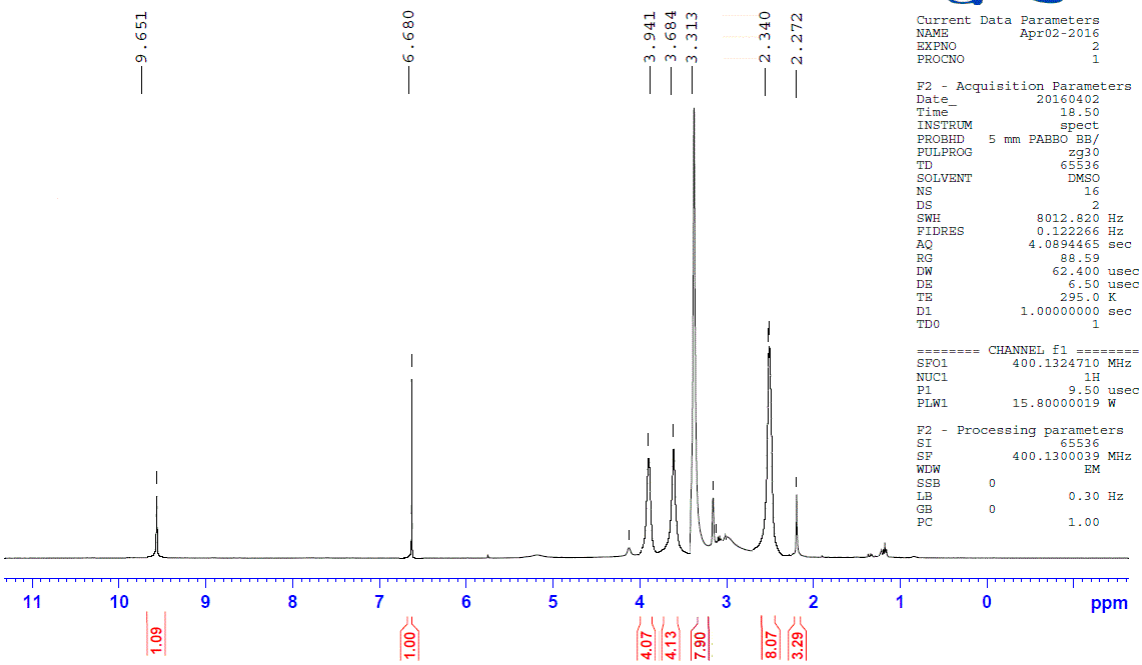
F2 - Acquisition Parameters
Date_           20160417
Time               5.09
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 8192
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                199.94
DW              20.800 usec
DE               6.50 usec
TE              295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                  13C
P1                 9.50 usec
PLW1         66.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         18.00000000 W
PLW12         0.20056000 W
PLW13         0.16245000 W

F2 - Processing parameters
SI                 32768
SF           100.6128045 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

39_1H



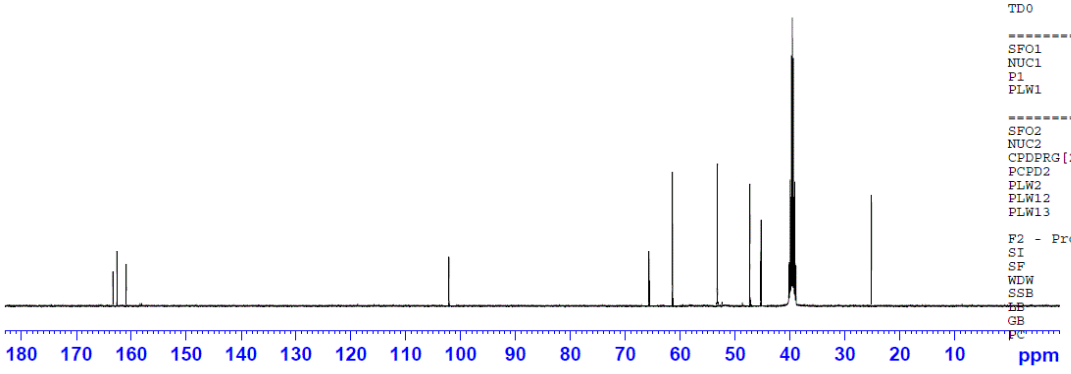

```
Current Data Parameters
NAME                  Apr02-2016
EXPNO                          2
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20160402
Time                       18.50
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG                    zg30
TD                        65536
SOLVENT                    DMSO
NS                           16
DS                            2
SWH                    8012.820 Hz
FIDRES                 0.122266 Hz
AQ                     4.0894465 sec
RG                         88.59
DW                        62.400 usec
DE                         6.50 usec
TE                        295.0 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                 400.1324710 MHz
NUC1                         1H
P1                         9.50 usec
PLW1                15.80000019 W

F2 - Processing parameters
SI                        65536
SF                   400.1300039 MHz
WDW                          EM
SSB                   0
LB                         0.30 Hz
GB                    0
PC                         1.00
```

39_13c



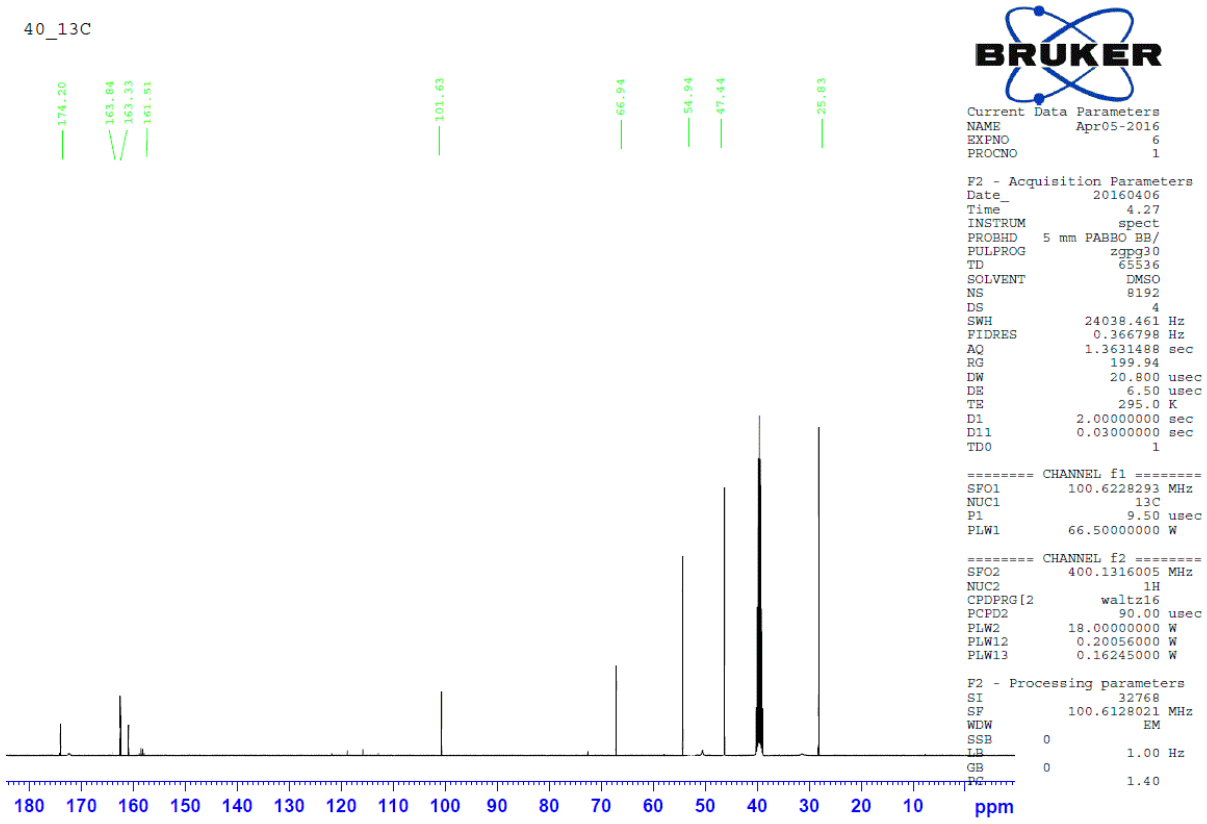

```
Current Data Parameters
NAME                  Apr02-2016
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20160402
Time                       19.56
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG                  zgpg30
TD                        65536
SOLVENT                    DMSO
NS                          1024
DS                             4
SWH                   24038.461 Hz
FIDRES                 0.366798 Hz
AQ                     1.3631488 sec
RG                         199.94
DW                        20.800 usec
DE                         6.50 usec
TE                        295.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                 100.6228293 MHz
NUC1                         13C
P1                         9.50 usec
PLW1                66.50000000 W

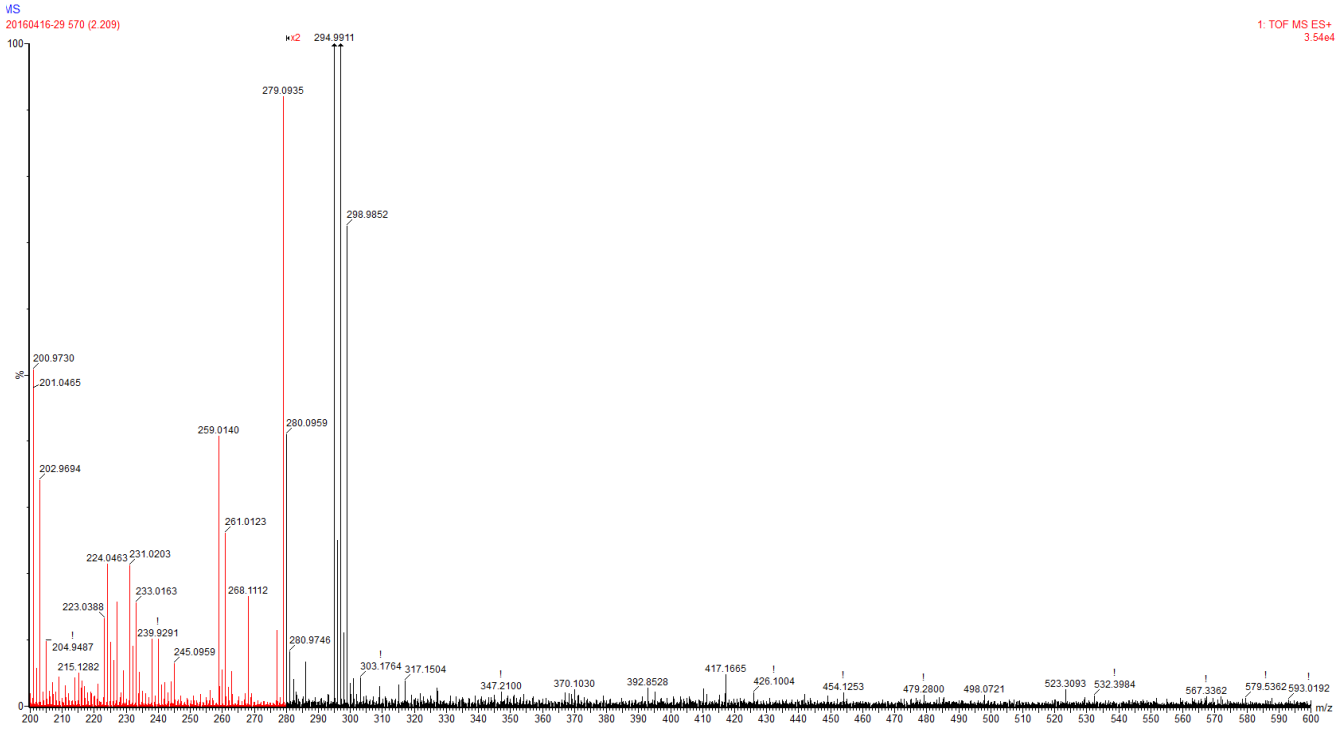
======== CHANNEL f2 ========
SFO2                 400.1316005 MHz
NUC2                         1H
CPDPRG[2                waltz16
PCPD2                     90.00 usec
PLW2                18.00000000 W
PLW12                0.20056000 W
PLW13                0.16245000 W

F2 - Processing parameters
SI                        32768
SF                   100.6128022 MHz
WDW                          EM
SSB                   0
LB                         1.00 Hz
GB                    0
PC                         1.40
```

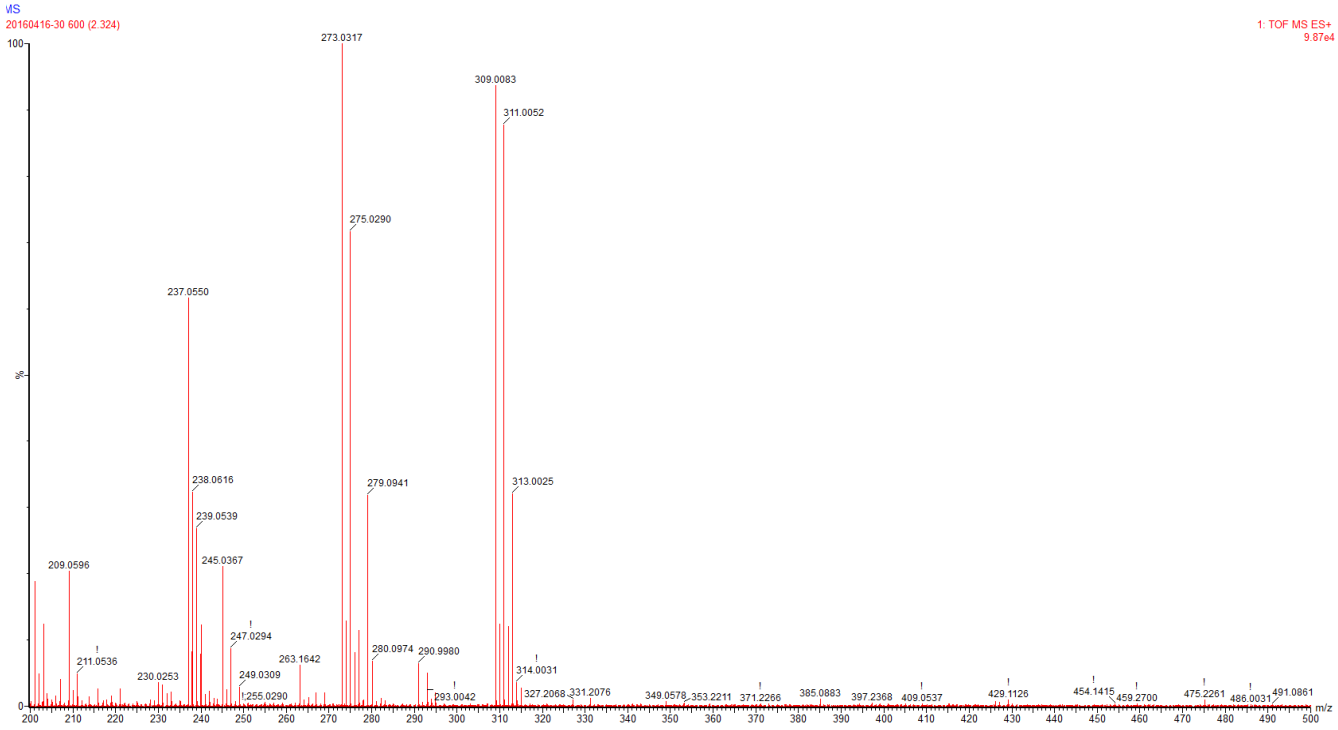

40_13C
174.20
163.84
163.33
161.51
101.63
66.94
54.94
47.44
25.83
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
Current Data Parameters
NAME Apr05-2016
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160406
Time 4.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 8192
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 199.94
DW 20.800 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.50 usec
PLW1 66.50000000 W
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 18.00000000 W
PLW12 0.20056000 W
PLW13 0.16245000 W
F2 - Processing parameters
SI 32768
SF 100.6128021 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

5. HRMS spectra

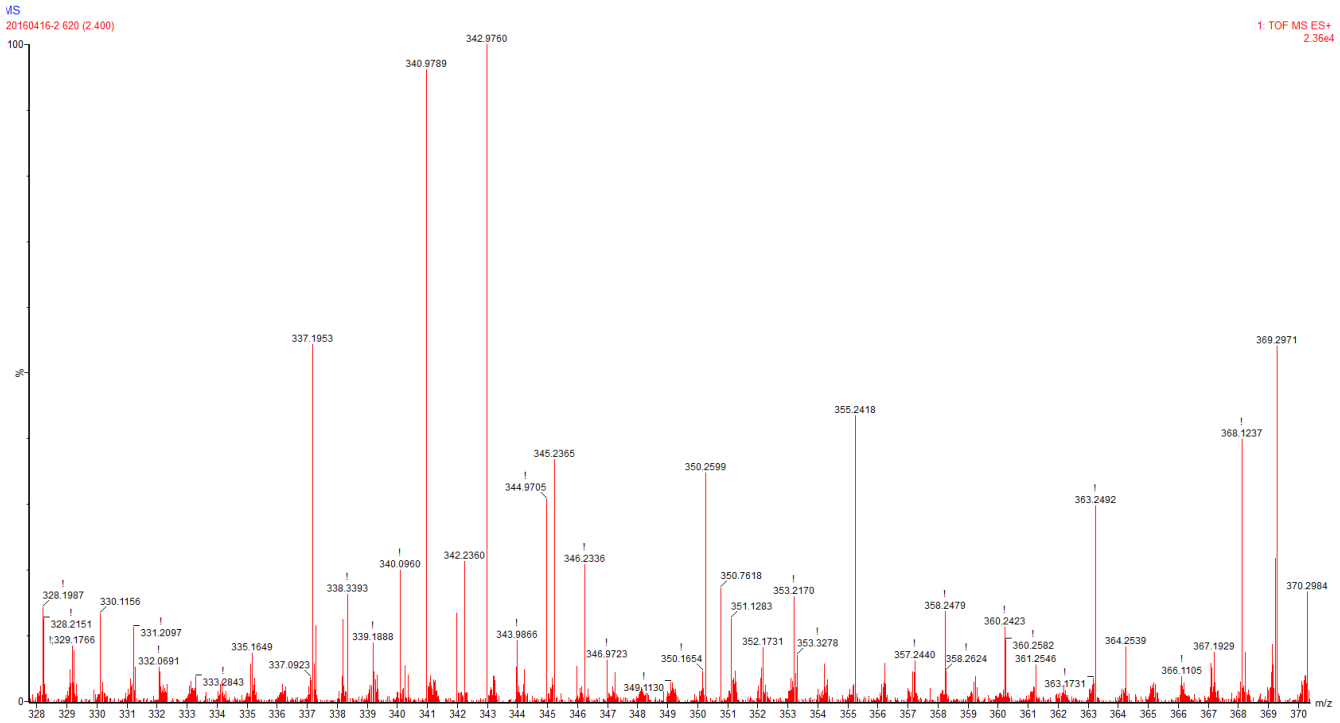
6



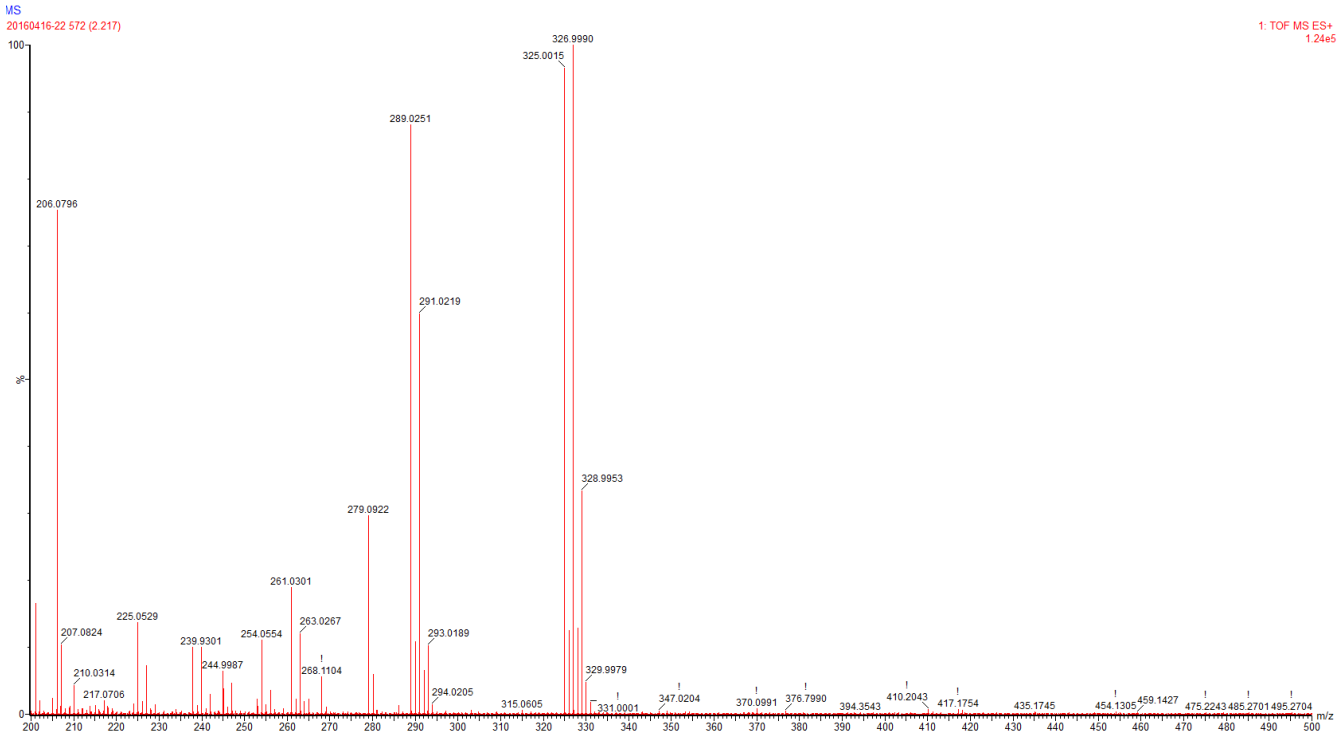
8



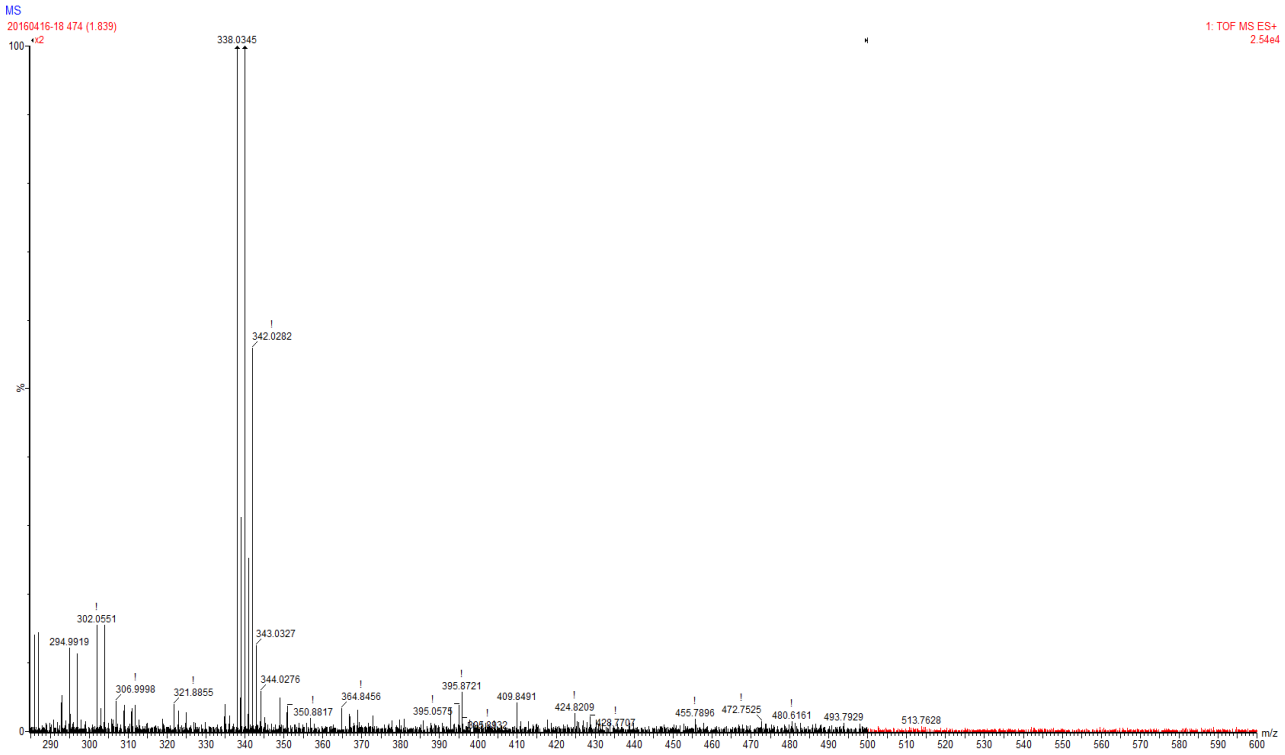
10


MS
20160416-2 620 (2.400)
1: TOF MS ES+
2.36e4
100
%
0
342.9760
340.9789
337.1953
369.2971
355.2418
368.1237
345.2365
350.2599
344.9705
363.2492
340.0960
342.2360
346.2336
370.2984
350.7618
353.2170
338.3393
351.1283
358.2479
328.1987
330.1156
360.2423
328.2151
331.2097
339.1888
343.9866
364.2539
329.1766
352.1731
353.3278
360.2582
335.1649
332.0691
346.9723
357.2440
358.2624
361.2546
367.1929
337.0923
350.1654
363.1731
366.1105
333.2843
349.1130
328
329
330
331
332
333
334
335
336
337
338
339
340
341
342
343
344
345
346
347
348
349
350
351
352
353
354
355
356
357
358
359
360
361
362
363
364
365
366
367
368
369
370
m/z

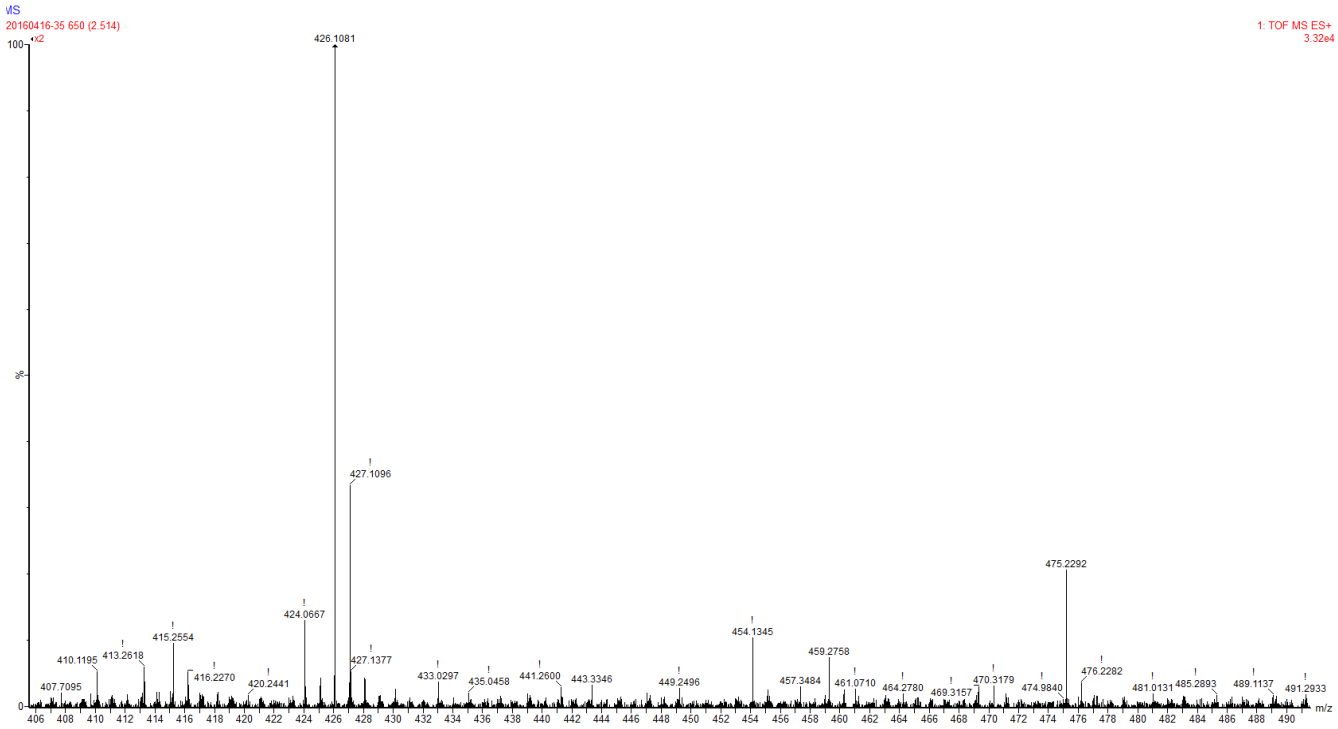
11


MS
20160416-22 572 (2.217)
1: TOF MS ES+
1.24e5
100
%
0
326.9990
325.0015
289.0251
206.0796
291.0219
328.9953
279.0922
261.0301
225.0529
263.0267
207.0824
239.9301
254.0554
293.0189
244.9987
329.9979
210.0314
268.1104
217.0706
294.0205
315.0605
331.0001
347.0204
370.0991
376.7990
394.3543
410.2043
417.1754
435.1745
454.1305
459.1427
475.2243
485.2701
495.2704
200
210
220
230
240
250
260
270
280
290
300
310
320
330
340
350
360
370
380
390
400
410
420
430
440
450
460
470
480
490
500
m/z

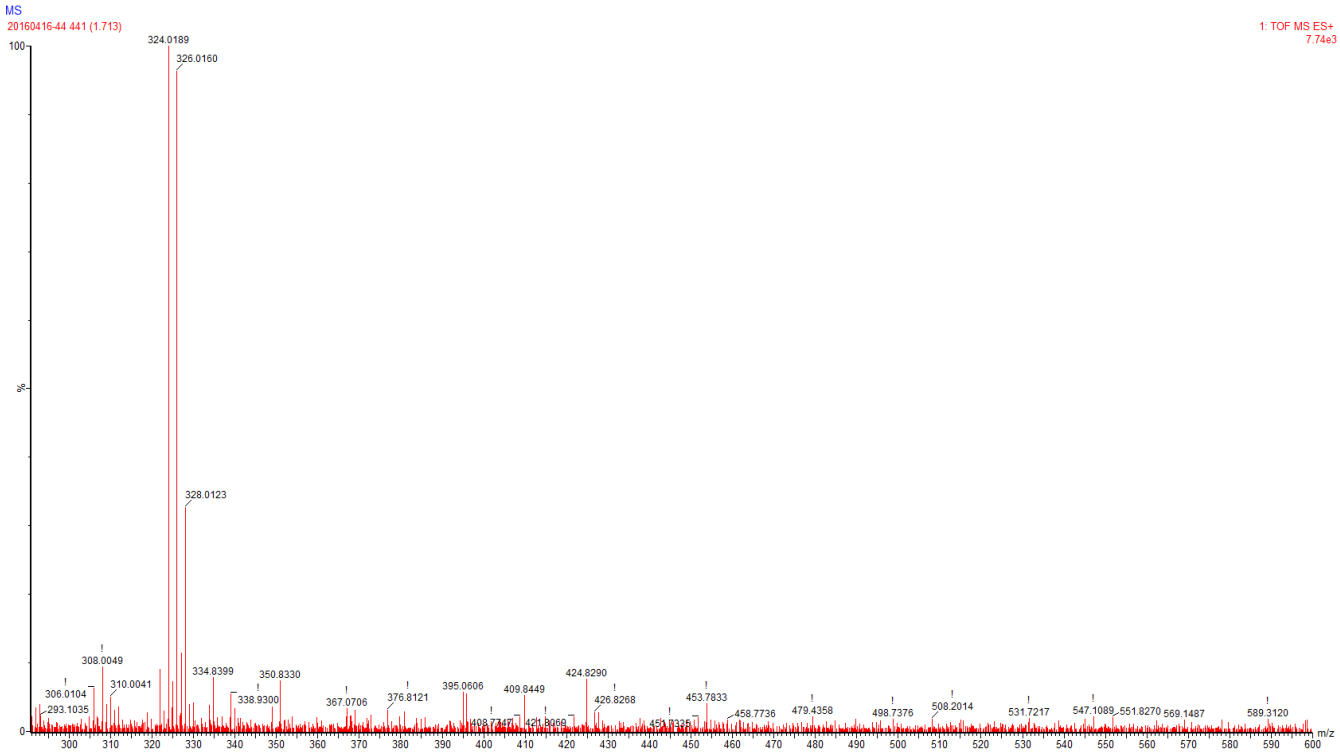
12


MS
20160416-18 474 (1.839)
1: TOF MS ES+
2.54e4
100
%
0
x2
338.0345
342.0282
302.0551
294.9919
343.0327
344.0276
306.9998
321.8855
364.8456
350.8817
395.0575
395.8721
409.8491
424.8209
455.7896
472.7525
480.6161
493.7929
513.7628
m/z

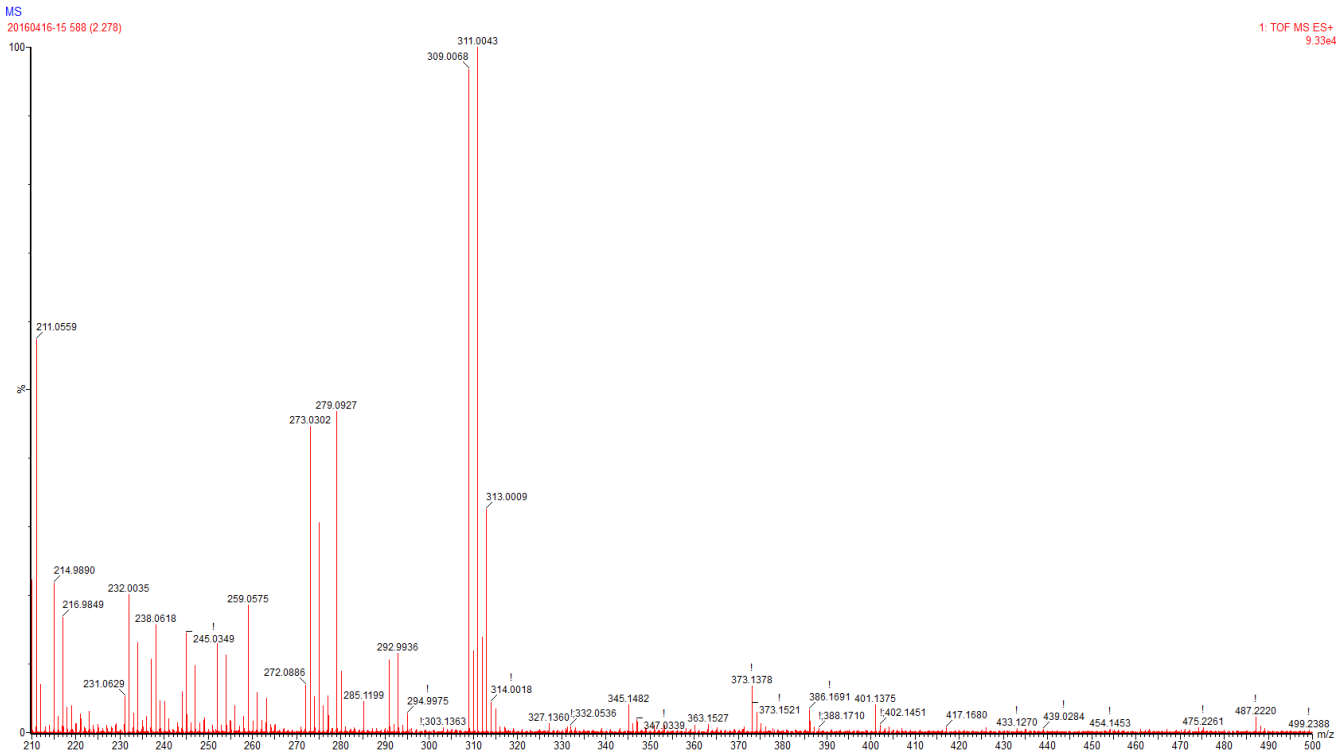
13


MS
20160416-35 650 (2.514)
1: TOF MS ES+
3.32e4
100
%
0
x2
426.1081
427.1096
475.2292
424.0667
454.1345
415.2554
459.2758
413.2618
410.1195
427.1377
476.2282
416.2270
407.7095
420.2441
433.0297
435.0458
441.2600
443.3346
449.2496
457.3484
461.0710
464.2780
469.3157
470.3179
474.9840
481.0131
485.2893
489.1137
491.2933
m/z

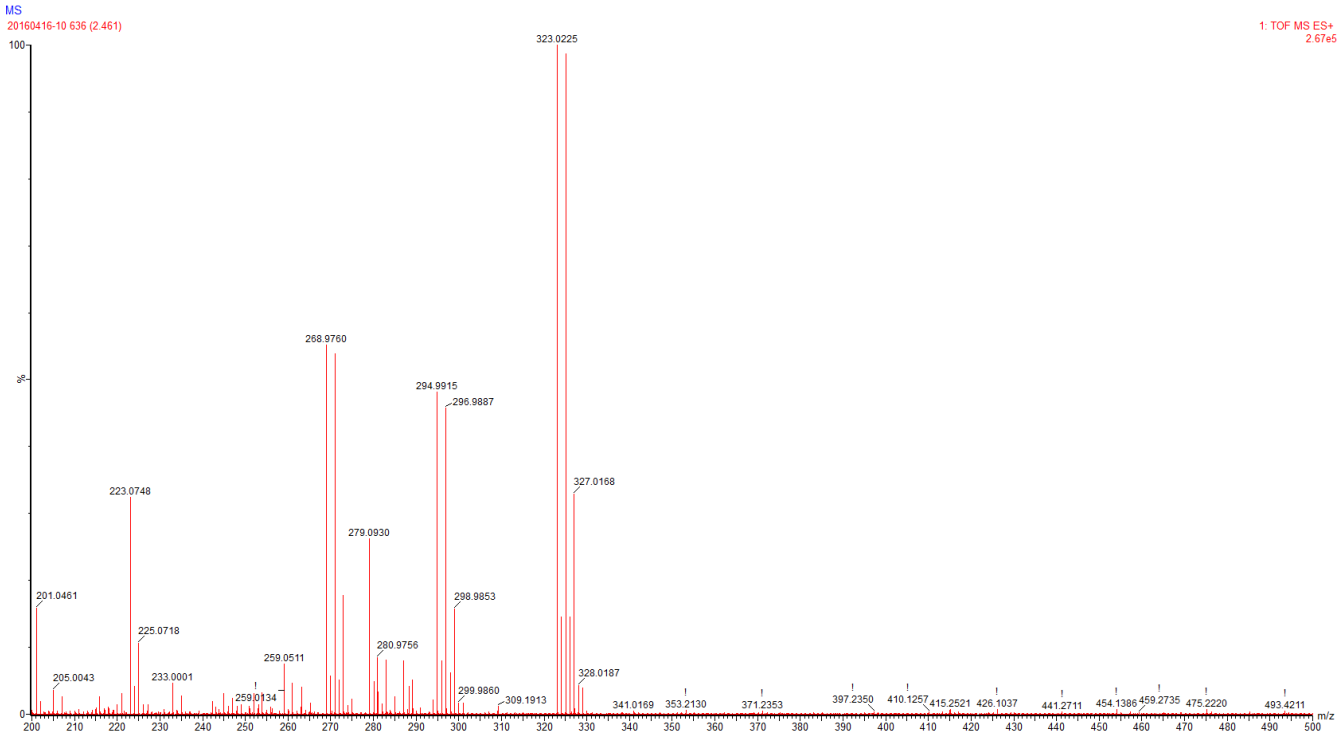
14


MS
20160416-44 441 (1.713)
1: TOF MS ES+
7.74e3
100
%
0
m/z
324.0189
326.0160
328.0123
308.0049
310.0041
306.0104
293.1035
334.8399
350.8330
338.9300
367.0706
376.8121
395.0606
409.8449
424.8290
426.8268
453.7833
458.7736
479.4358
498.7376
508.2014
531.7217
547.1089
551.8270
569.1487
589.3120

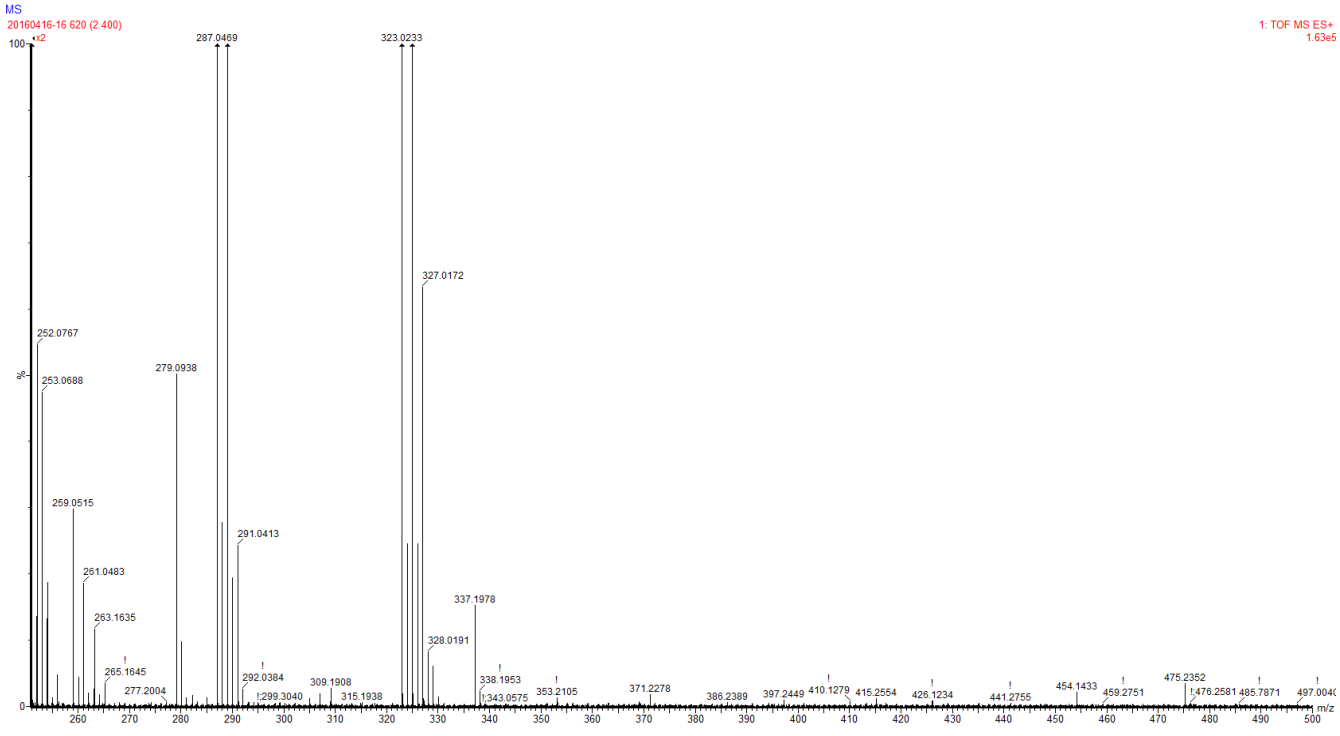
15


MS
20160416-15 588 (2.278)
1: TOF MS ES+
9.33e4
100
%
0
m/z
311.0043
309.0068
211.0559
279.0927
273.0302
313.0009
214.9890
232.0035
216.9849
238.0618
259.0575
245.0349
292.9936
272.0886
231.0629
285.1199
294.9975
314.0018
373.1378
373.1521
386.1691
401.1375
345.1482
327.1360
332.0536
347.0339
363.1527
388.1710
402.1451
417.1680
433.1270
439.0284
454.1453
475.2261
487.2220
499.2388

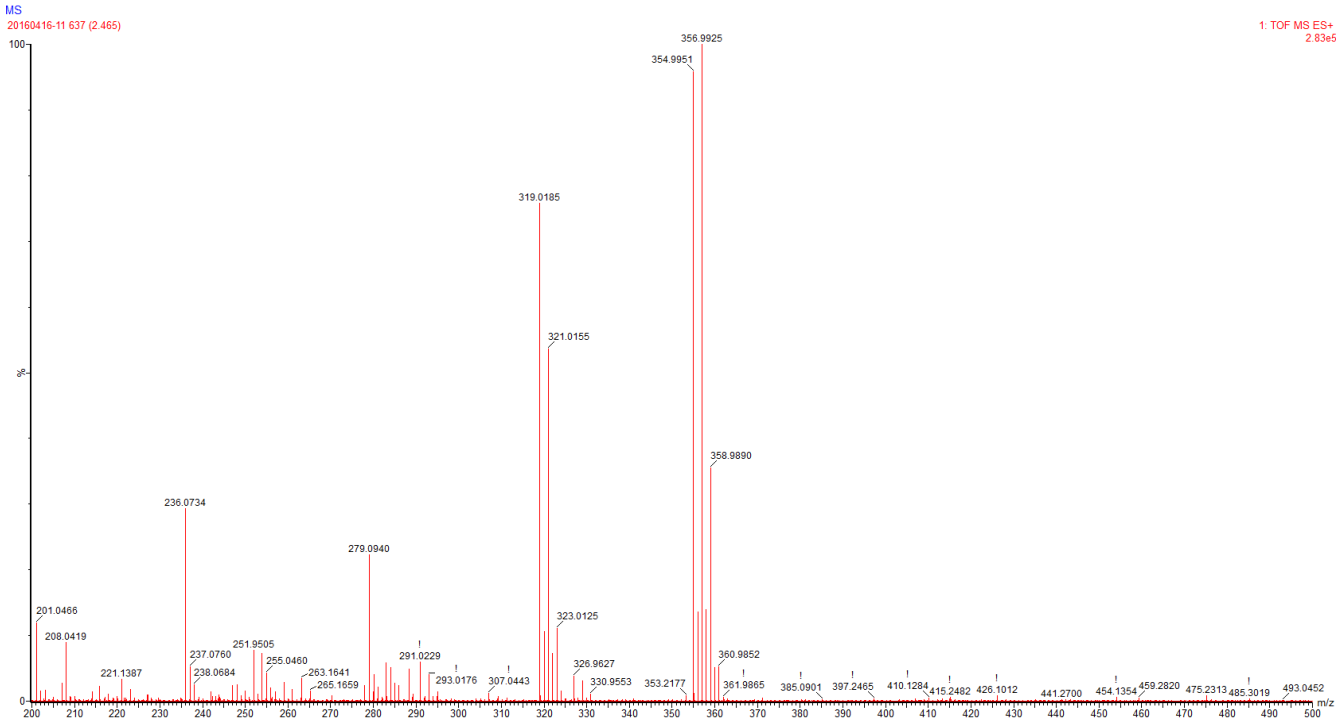
16


MS
20160416-10 636 (2.461)
1: TOF MS ES+
2.67e5
100
%
0
323.0225
268.9760
294.9915
296.9887
327.0168
223.0748
279.0930
201.0461
298.9853
225.0718
280.9756
259.0511
328.0187
205.0043
233.0001
259.0134
299.9860
309.1913
341.0169
353.2130
371.2353
397.2350
410.1257
415.2521
426.1037
441.2711
454.1386
459.2735
475.2220
493.4211
200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450 460 470 480 490 500
m/z

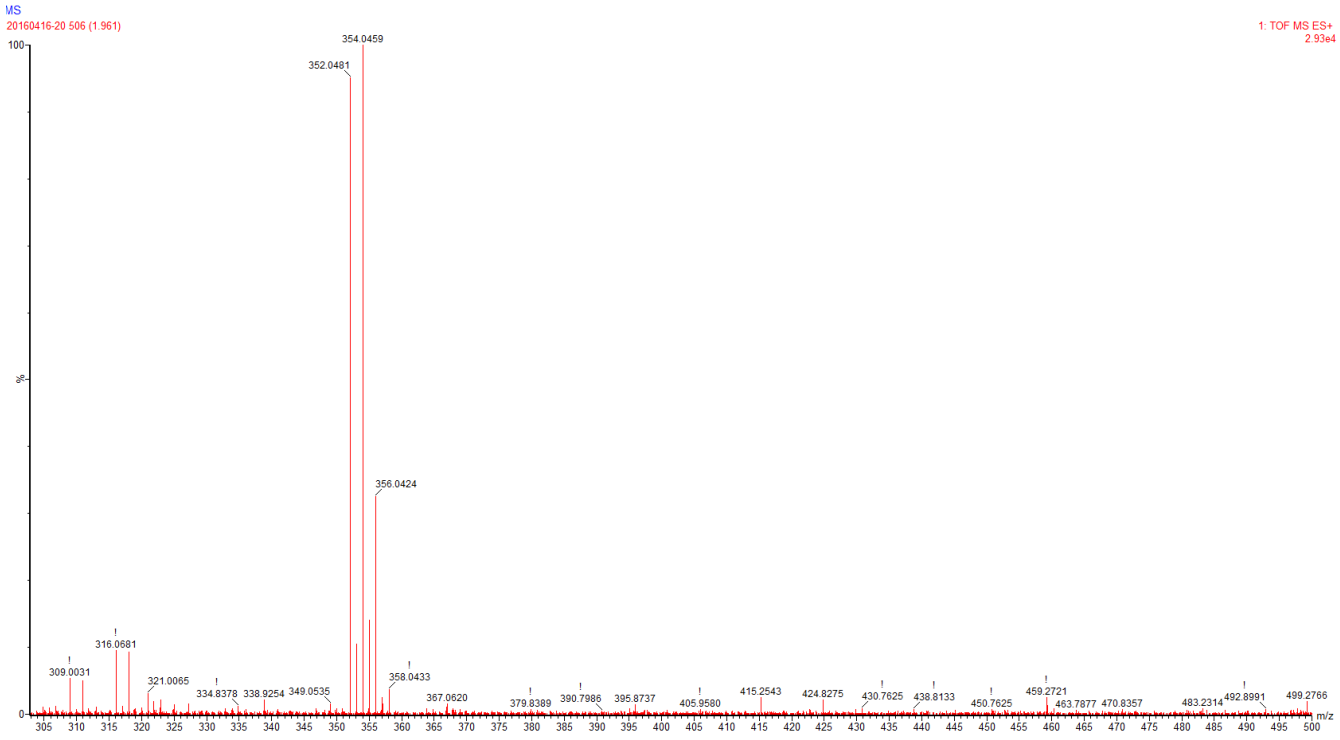
17


MS
20160416-16 620 (2.400)
1: TOF MS ES+
1.63e5
100
%
0
+2
287.0469
323.0233
327.0172
252.0767
253.0688
279.0938
259.0515
291.0413
261.0483
337.1978
263.1635
328.0191
265.1645
292.0384
277.2004
309.1908
315.1938
338.1953
353.2105
371.2278
386.2389
397.2449
410.1279
415.2554
426.1234
441.2755
454.1433
459.2751
475.2352
485.7871
260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450 460 470 480 490 500
m/z

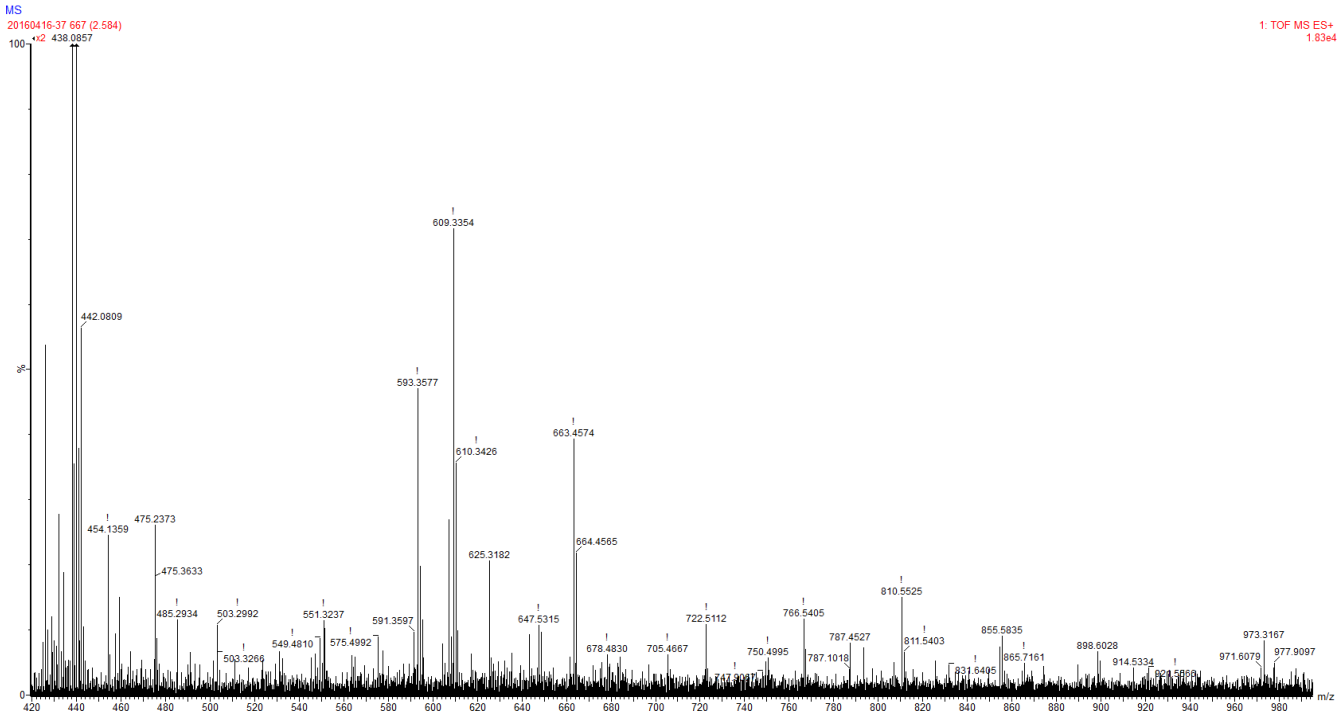
19


MS
20160416-11 637 (2.465)
1: TOF MS ES+
2.83e5
100
%
0
356.9925
354.9951
319.0185
321.0155
358.9890
236.0734
279.0940
201.0466
208.0419
323.0125
251.9505
237.0760
255.0460
238.0684
263.1641
265.1659
221.1387
291.0229
293.0176
307.0443
326.9627
330.9553
353.2177
360.9852
361.9865
385.0901
397.2465
410.1284
415.2482
426.1012
441.2700
454.1354
459.2820
475.2313
485.3019
493.0452
200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450 460 470 480 490 500
m/z

21


MS
20160416-20 506 (1.961)
1: TOF MS ES+
2.93e4
100
%
0
354.0459
352.0481
356.0424
316.0681
309.0031
321.0065
358.0433
334.8378
338.9254
349.0535
367.0620
379.8389
390.7986
395.8737
405.9580
415.2543
424.8275
430.7625
438.8133
450.7625
459.2721
463.7877
470.8357
483.2314
492.8991
499.2766
305 310 315 320 325 330 335 340 345 350 355 360 365 370 375 380 385 390 395 400 405 410 415 420 425 430 435 440 445 450 455 460 465 470 475 480 485 490 495 500
m/z

22


MS
20160416-37 667 (2.584)
1: TOF MS ES+
1.83e4
438.0857
609.3354
442.0809
593.3577
663.4574
610.3426
454.1359
475.2373
664.4565
625.3182
475.3633
810.5525
485.2934
503.2992
551.3237
591.3597
647.5315
722.5112
766.5405
787.4527
811.5403
855.5835
549.4810
575.4992
678.4830
705.4667
750.4995
787.1018
865.7161
898.6028
914.5334
971.6079
973.3167
977.9097
503.3266
831.6405
m/z

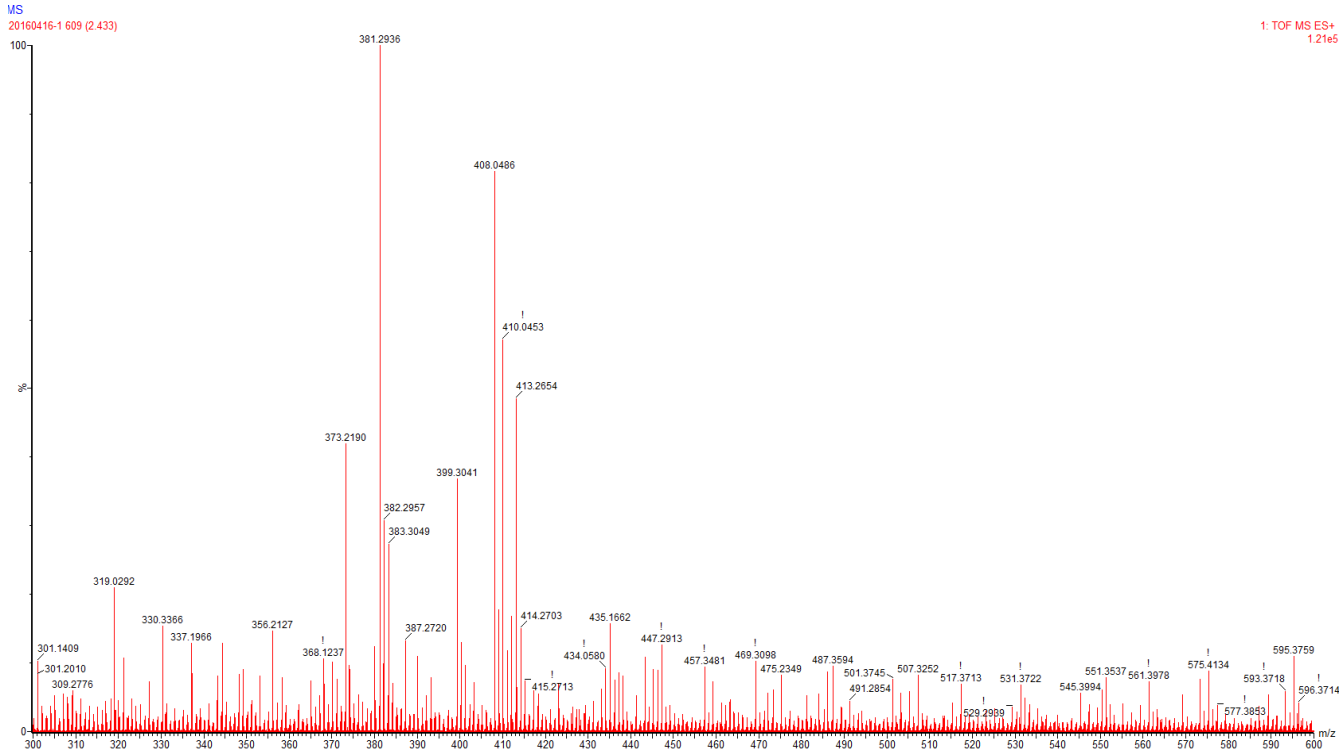
23


MS
20160416-39 503 (1.949)
1: TOF MS ES+
4.68e3
338.0347
340.0306
342.0302
322.0215
320.0200
323.0097
349.0536
424.8326
312.9306
319.8000
324.0087
334.8318
350.8869
354.8906
366.9256
367.0600
375.7947
392.8458
395.8771
403.9483
405.9642
415.2533
425.8212
439.7175
451.7414
457.2809
464.7689
475.7516
485.7541
489.0872
492.7583
499.2526
m/z

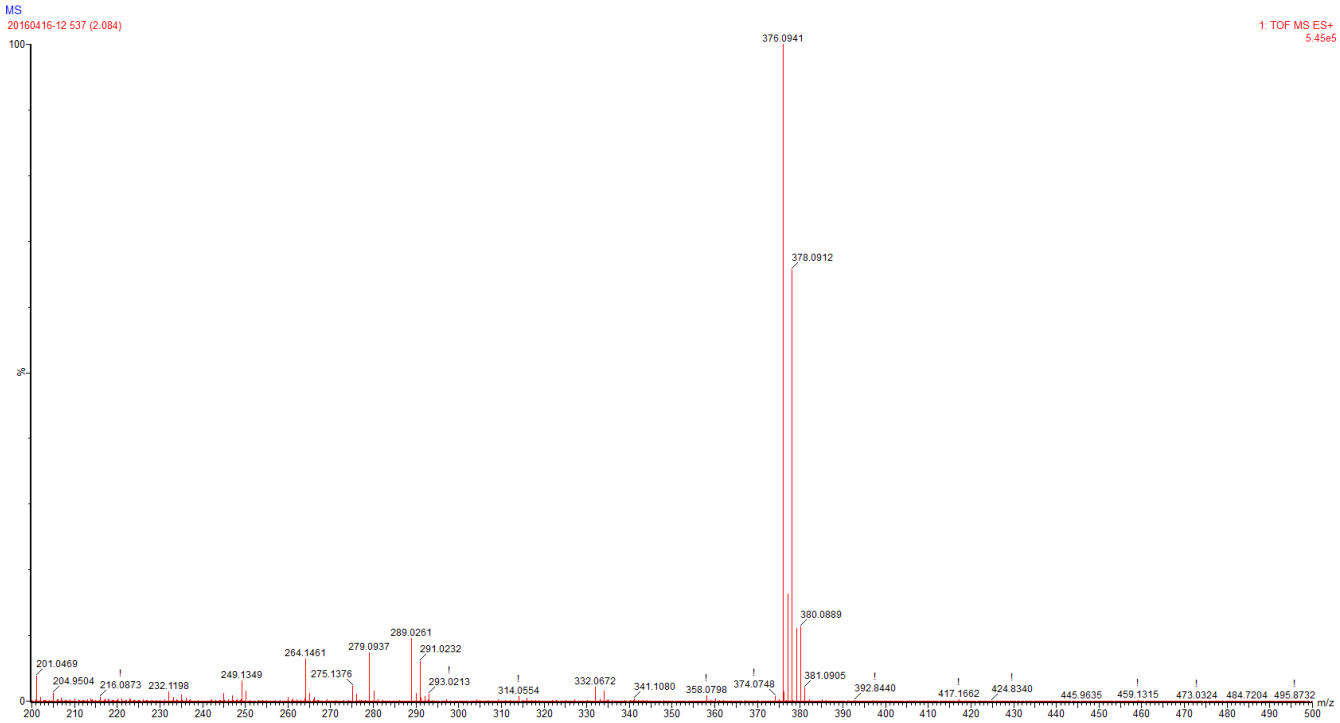
26


MS
20160416-32 580 (2.247)
1: TOF MS ES+
1.40e5
100
%
0
372.1360
374.1333
279.0938
376.1308
201.0474
260.1875
287.0470
289.0441
245.1767
204.9489
215.8882
231.0649
246.1784
261.1924
291.0432
317.1466
327.2013
345.1401
354.1183
377.1295
403.1380
410.2080
426.1109
435.1765
454.1394
465.0459
475.1716
493.1828
200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450 460 470 480 490 500
m/z

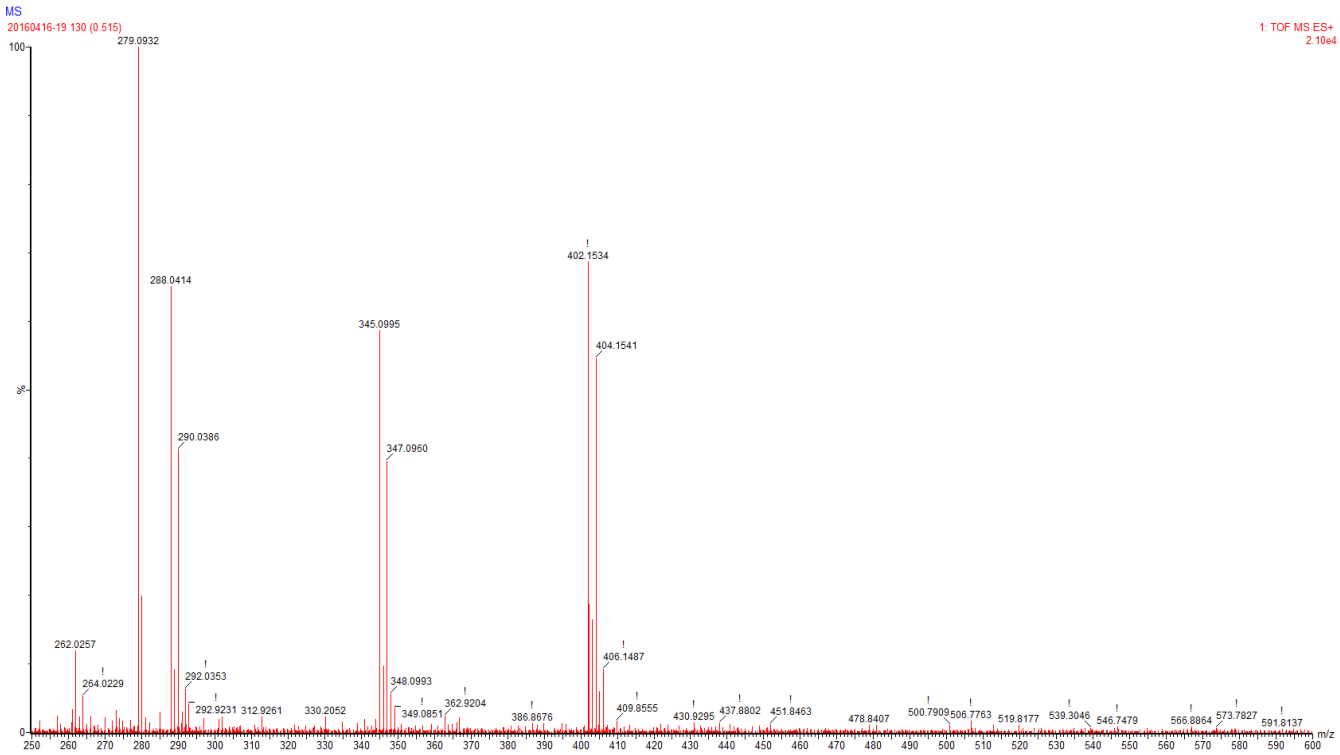
27


MS
20160416-1 609 (2.433)
1: TOF MS ES+
1.21e5
100
%
0
381.2936
408.0486
410.0453
413.2654
373.2190
399.3041
382.2957
383.3049
319.0292
330.3366
337.1966
356.2127
387.2720
414.2703
435.1662
301.1409
301.2010
309.2776
368.1237
434.0580
447.2913
457.3481
469.3098
475.2349
487.3594
491.2854
501.3745
507.3252
517.3713
529.2989
531.3722
545.3994
551.3537
561.3978
575.4134
577.3853
593.3718
595.3759
596.3714
300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450 460 470 480 490 500 510 520 530 540 550 560 570 580 590 600
m/z

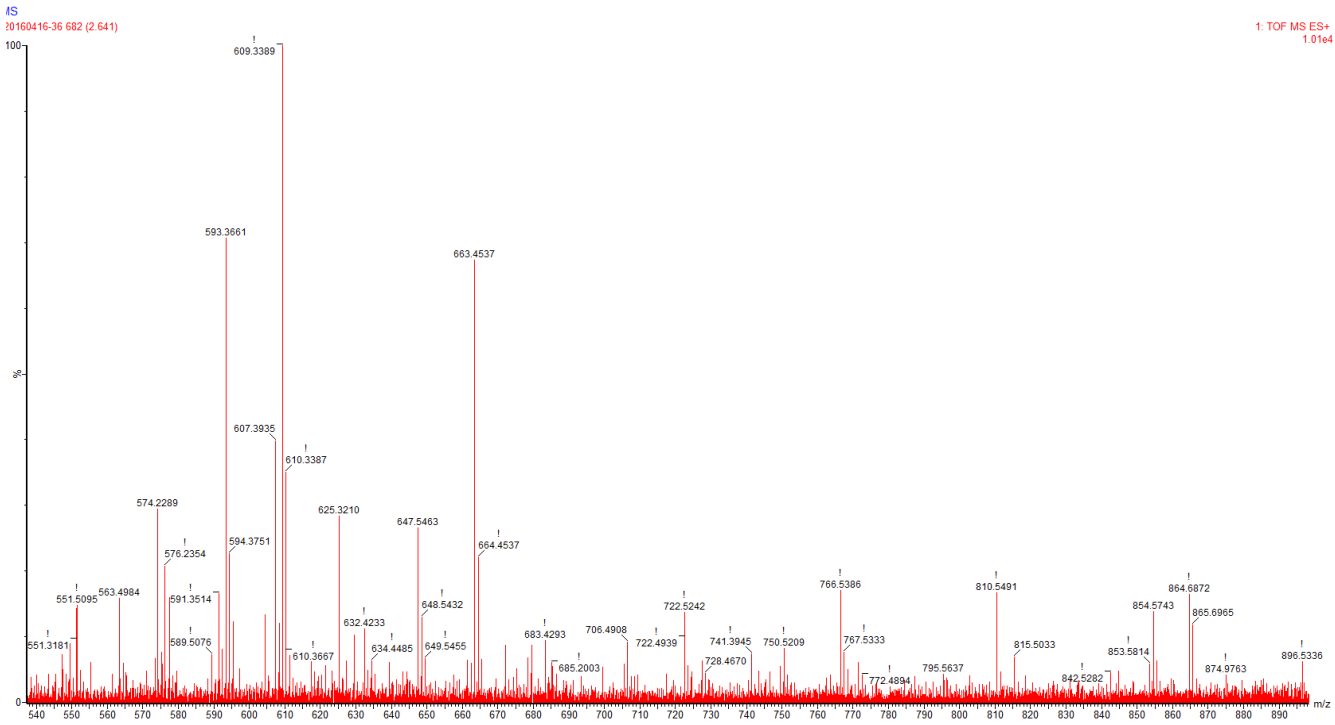
28


MS
20160416-12 537 (2.084)
1: TOF MS ES+
5.45e5
100
%
0
376.0941
378.0912
380.0889
381.0905
289.0261
279.0937
291.0232
264.1461
275.1376
293.0213
201.0469
204.9504
216.0873
232.1198
249.1349
314.0554
332.0672
341.1080
358.0798
374.0748
392.8440
417.1662
424.8340
445.9635
459.1315
473.0324
484.7204
495.8732
200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450 460 470 480 490 500
m/z

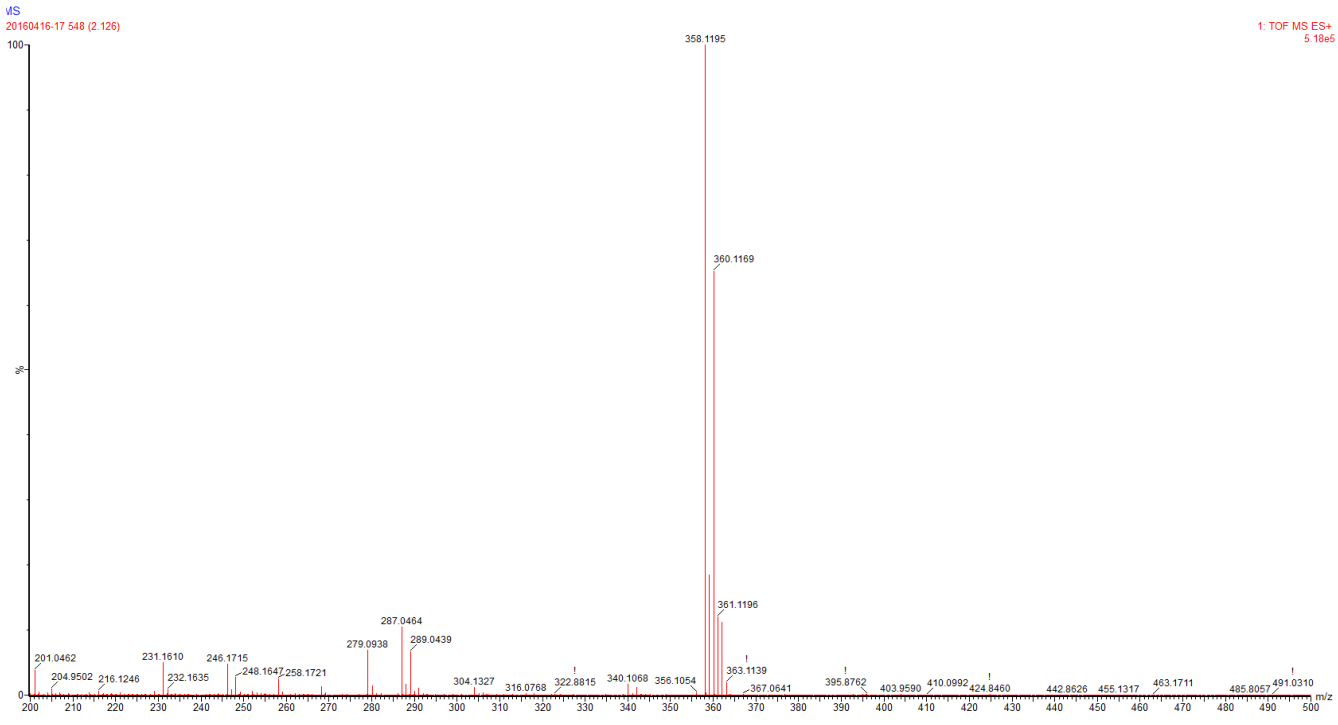
29


MS
20160416-19 130 (0.515)
1: TOF MS ES+
2.10e4
100
%
0
279.0932
288.0414
402.1534
345.0995
404.1541
290.0386
347.0960
406.1487
262.0257
264.0229
292.0353
348.0993
292.9231
312.9261
330.2052
349.0851
362.9204
386.8676
409.8555
430.9295
437.8802
451.8463
478.8407
500.7909
506.7763
519.8177
539.3046
546.7479
566.8864
573.7827
591.8137
250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450 460 470 480 490 500 510 520 530 540 550 560 570 580 590 600
m/z

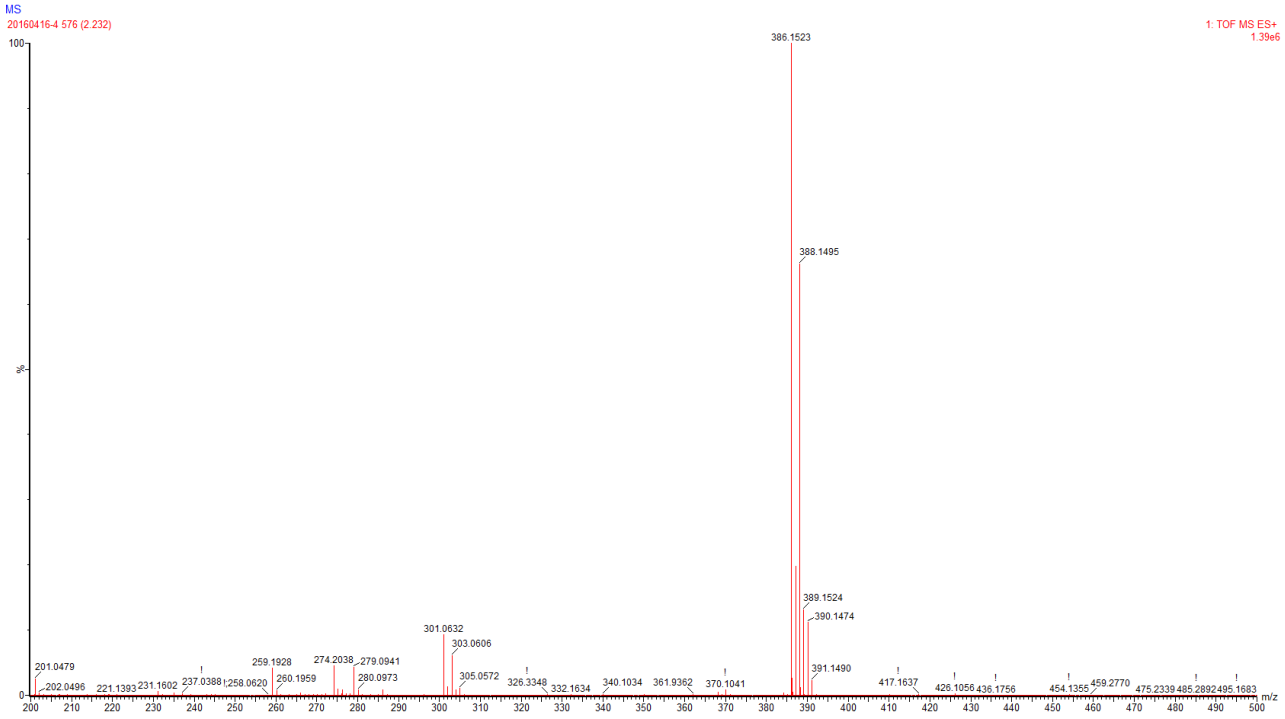
30


MS
20160416-36 682 (2.641)
1: TOF MS ES+
1.01e4
100
%
0
609.3389
593.3661
663.4537
607.3935
610.3387
625.3210
647.5463
574.2289
594.3751
576.2354
664.4537
563.4984
551.5095
591.3514
648.5432
632.4233
551.3181
589.5076
634.4485
649.5455
610.3667
683.4293
706.4908
722.5242
722.4939
685.2003
741.3945
750.5209
728.4670
766.5386
767.5333
772.4894
795.5637
810.5491
815.5033
842.5282
853.5814
854.5743
864.6872
865.6965
874.9763
896.5336
540 550 560 570 580 590 600 610 620 630 640 650 660 670 680 690 700 710 720 730 740 750 760 770 780 790 800 810 820 830 840 850 860 870 880 890
m/z

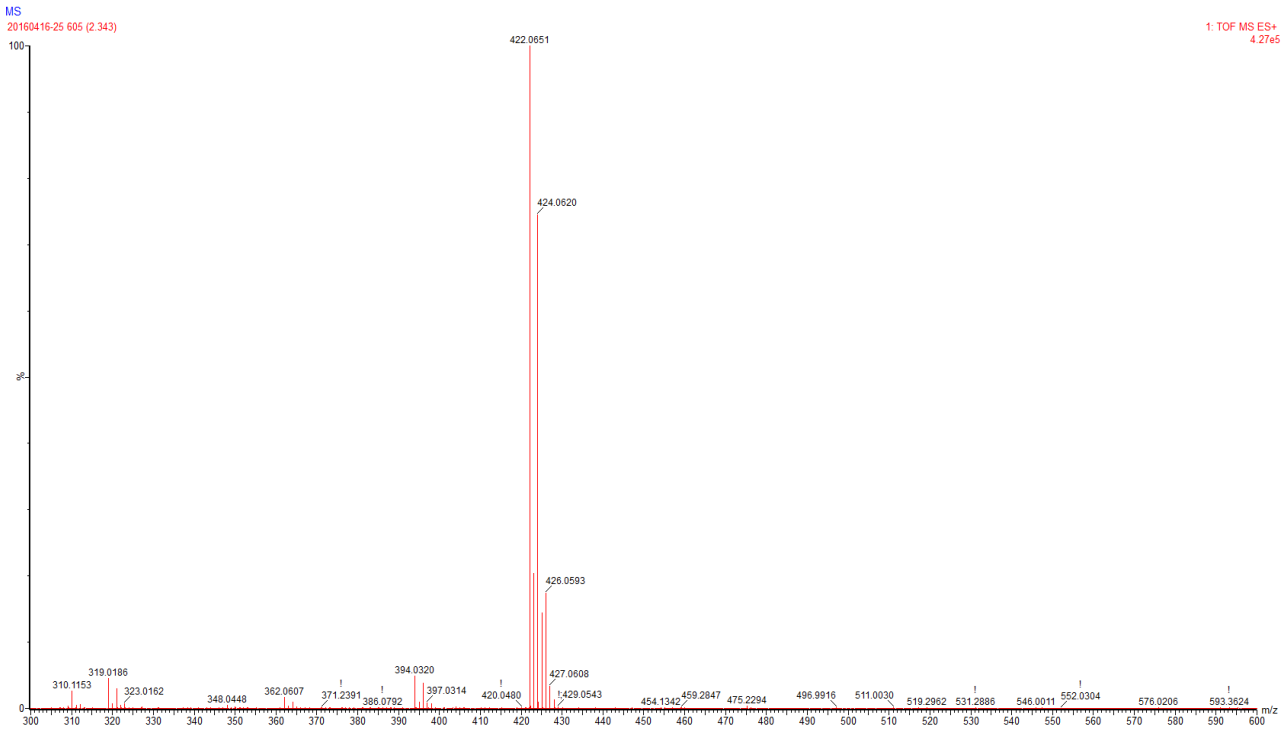
32


MS
20160416-17 548 (2.126)
1: TOF MS ES+
5.18e5
100
%
0
358.1195
360.1169
361.1196
287.0464
279.0938
289.0439
201.0462
231.1610
246.1715
204.9502
216.1246
232.1635
248.1647
258.1721
304.1327
316.0768
322.8815
340.1068
356.1054
363.1139
367.0641
395.8762
403.9590
410.0992
424.8460
442.8626
455.1317
463.1711
485.8057
491.0310
200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450 460 470 480 490 500
m/z

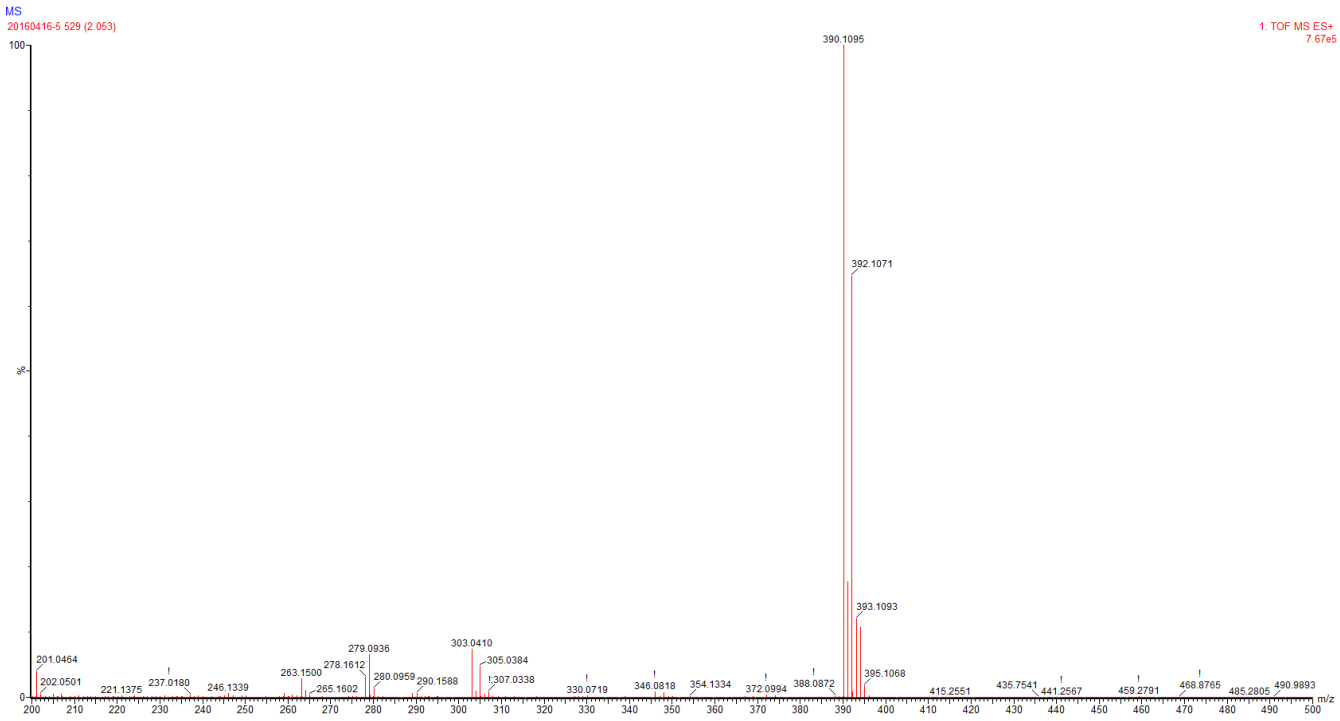
33


MS
20160416-4 576 (2.232)
1: TOF MS ES+
1.39e6
386.1523
388.1495
389.1524
390.1474
391.1490
301.0632
303.0606
305.0572
259.1928
274.2038
279.0941
280.0973
260.1959
201.0479
202.0496
221.1393
231.1602
237.0388
258.0620
326.3348
332.1634
340.1034
361.9362
370.1041
417.1637
426.1056
436.1756
454.1355
459.2770
475.2339
485.2892
495.1683
%
m/z

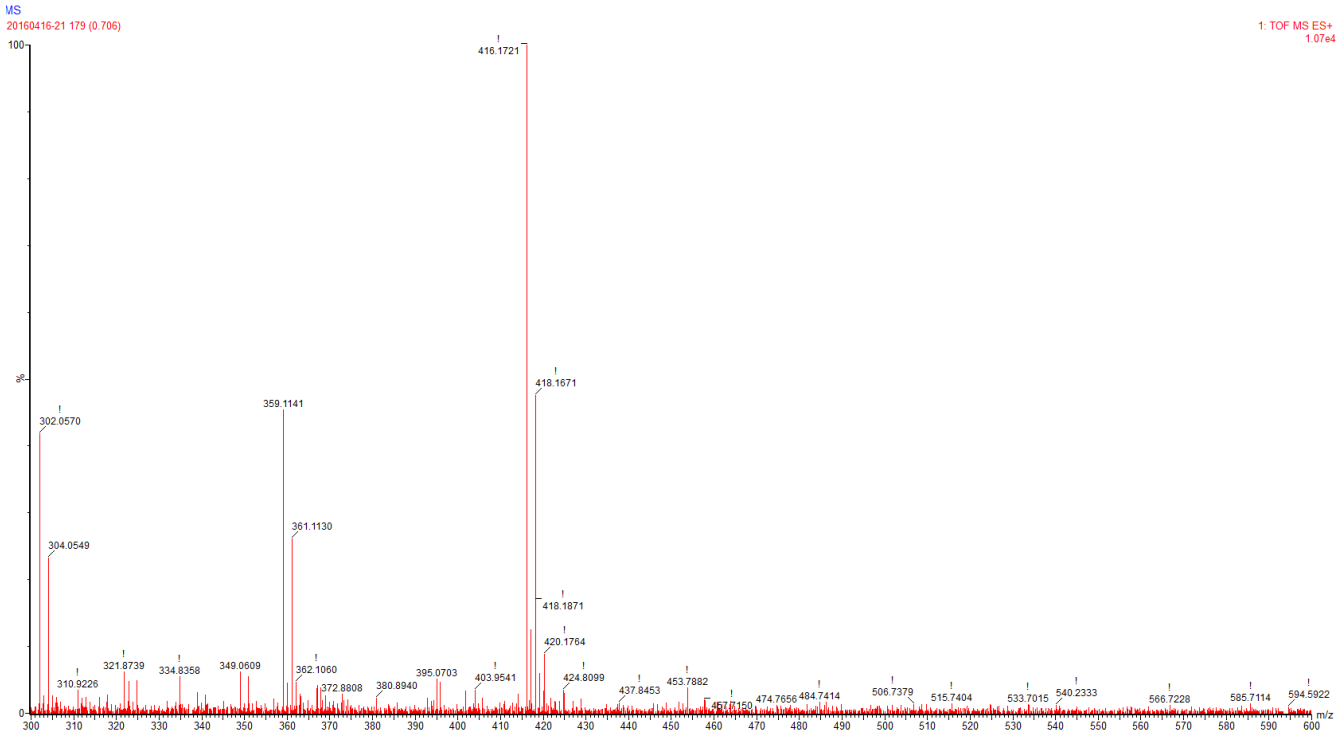
34


MS
20160416-25 605 (2.343)
1: TOF MS ES+
4.27e5
422.0651
424.0620
426.0593
427.0608
394.0320
319.0186
310.1153
323.0162
348.0448
362.0607
371.2391
386.0792
397.0314
420.0480
429.0543
454.1342
459.2847
475.2294
496.9916
511.0030
519.2962
531.2886
546.0011
552.0304
576.0206
593.3624
%
m/z

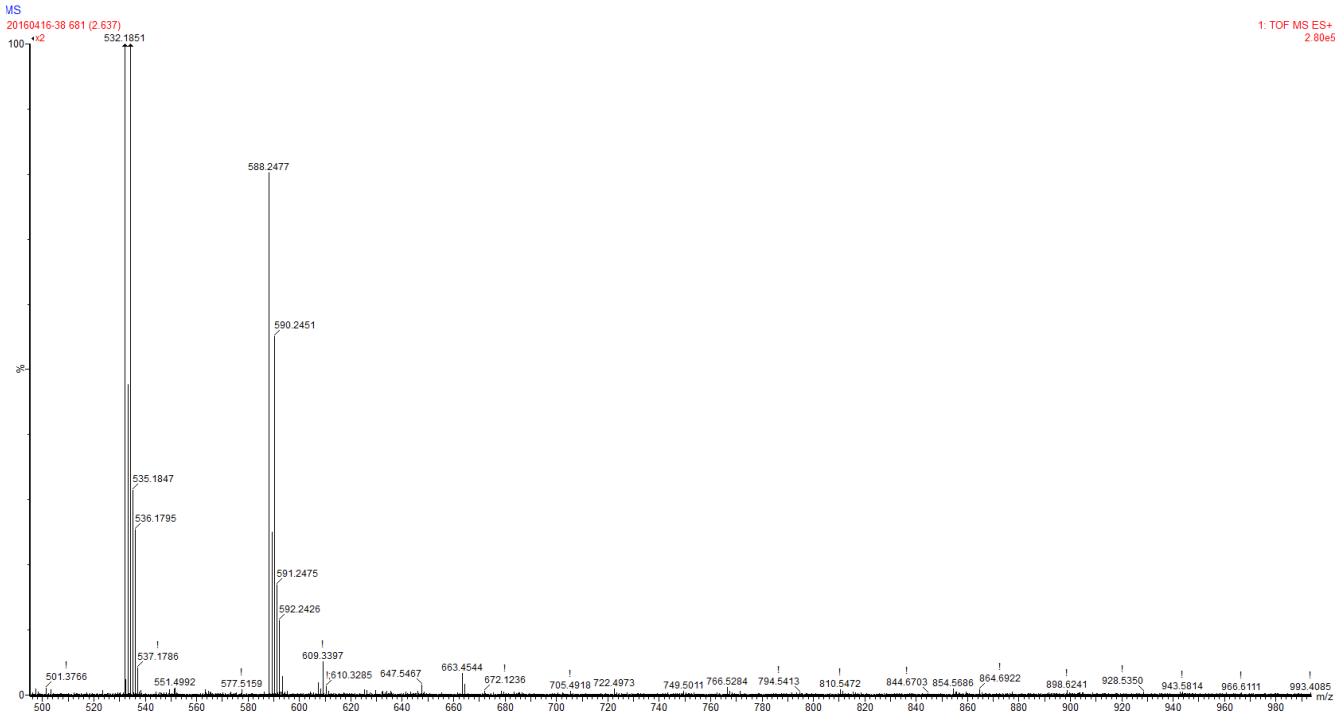
35


MS
20160416-5 529 (2.053)
1: TOF MS ES+
7.67e5
100
%
0
390.1095
392.1071
393.1093
303.0410
279.0936
278.1612
305.0384
263.1500
280.0959
290.1588
307.0338
201.0464
202.0501
221.1375
237.0180
246.1339
265.1602
330.0719
346.0818
354.1334
372.0994
388.0872
395.1068
415.2551
435.7541
441.2567
459.2791
468.8765
485.2805
490.9893
200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450 460 470 480 490 500
m/z

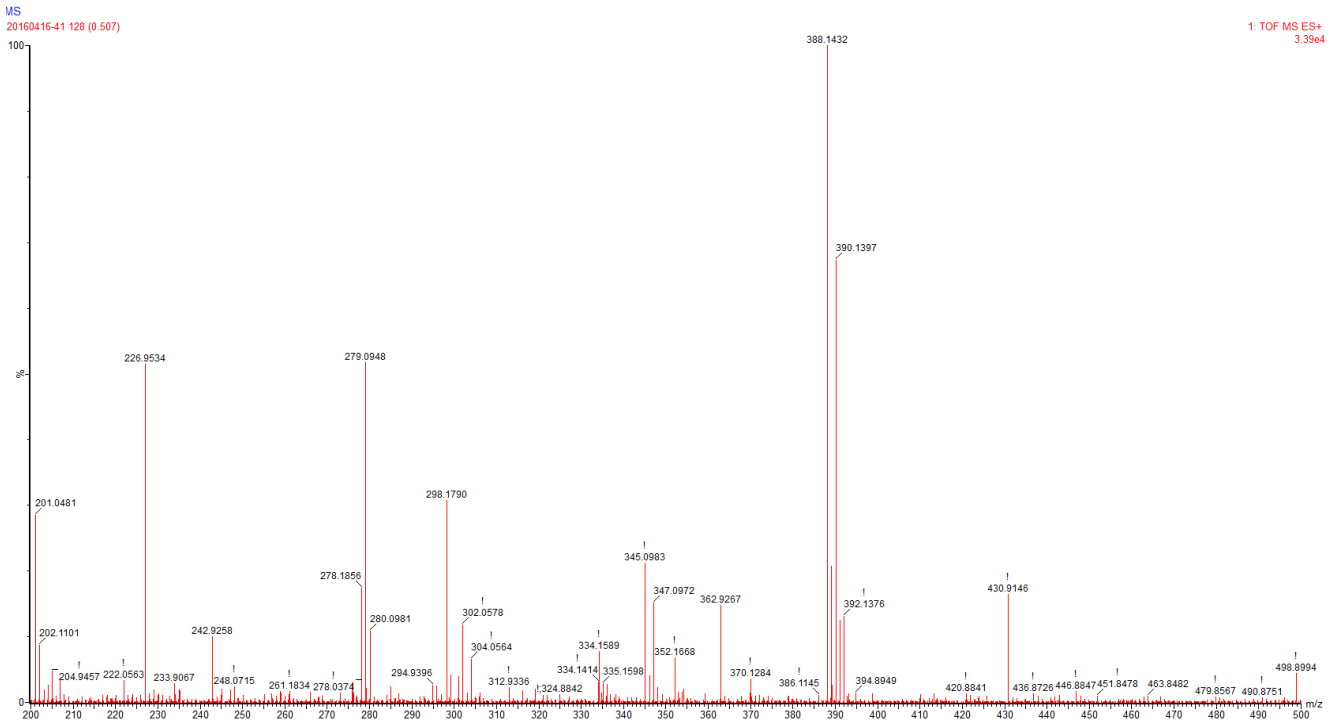
36


MS
20160416-21 179 (0.706)
1: TOF MS ES+
1.07e4
100
%
0
416.1721
418.1671
359.1141
302.0570
361.1130
304.0549
418.1871
420.1764
321.8739
334.8358
349.0609
310.9226
362.1060
372.8808
380.8940
395.0703
403.9541
424.8099
437.8453
453.7882
457.7150
474.7656
484.7414
506.7379
515.7404
533.7015
540.2333
566.7228
585.7114
594.5922
300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450 460 470 480 490 500 510 520 530 540 550 560 570 580 590 600
m/z

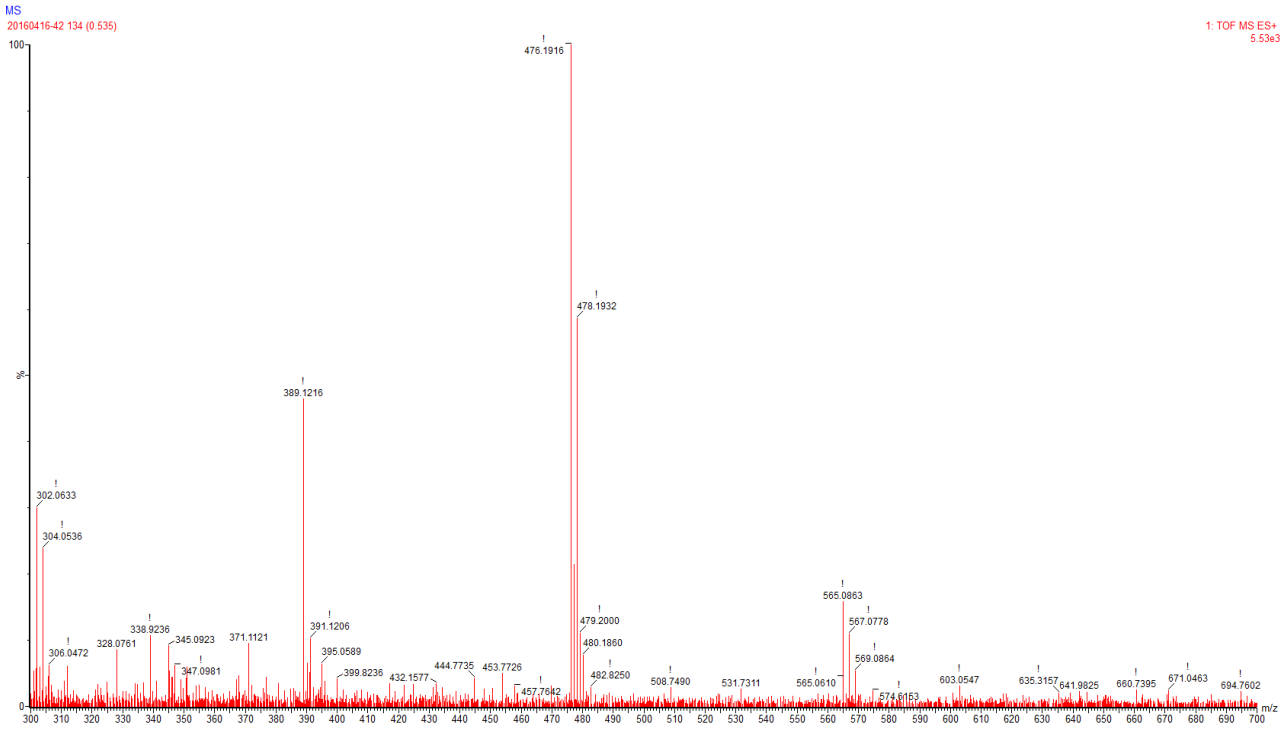
37


MS
20160416-38 681 (2.637)
1: TOF MS ES+
2.80e5
x2
532.1851
588.2477
590.2451
535.1847
536.1795
591.2475
592.2426
537.1786
609.3397
501.3766
551.4992
577.5159
610.3285
647.5467
663.4544
672.1236
705.4918
722.4973
749.5011
766.5284
794.5413
810.5472
844.6703
854.5686
864.6922
898.6241
928.5350
943.5814
966.6111
993.4085
m/z

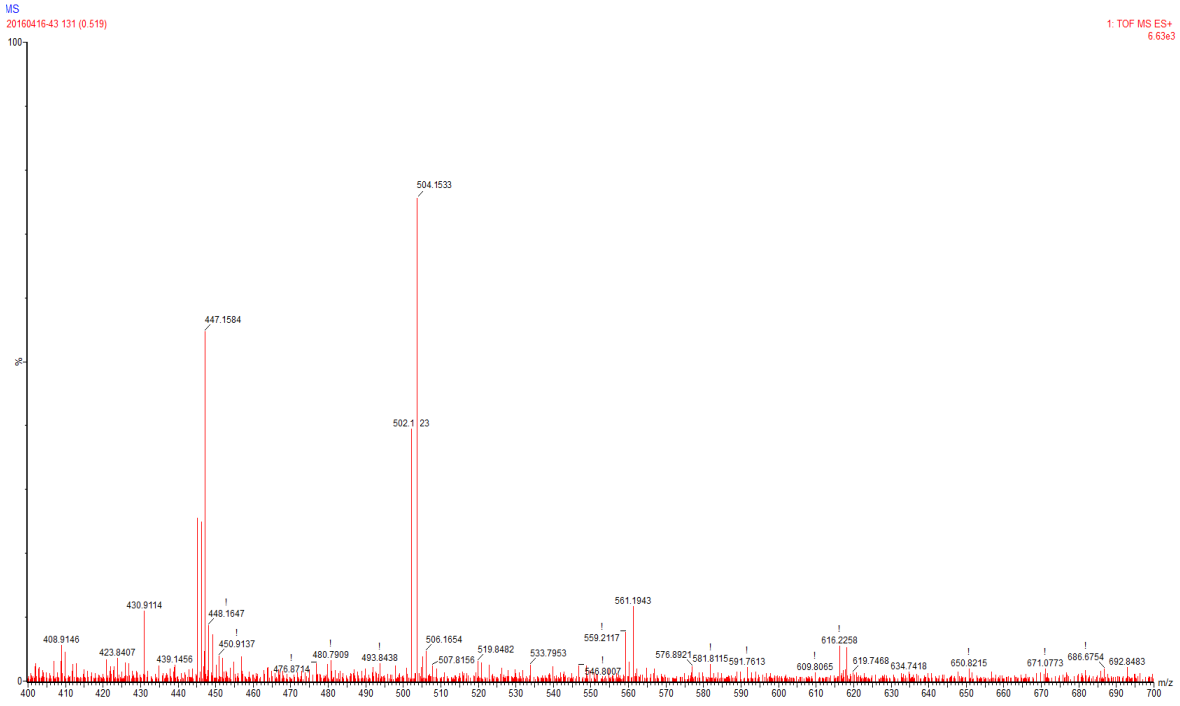
38


MS
20160416-41 128 (0.507)
1: TOF MS ES+
3.39e4
388.1432
390.1397
226.9534
279.0948
298.1790
201.0481
345.0983
278.1856
347.0972
362.9267
392.1376
430.9146
280.0981
302.0578
202.1101
242.9258
304.0564
334.1589
352.1668
204.9457
222.0563
233.9067
248.0715
261.1834
278.0374
294.9396
312.9336
324.8842
334.1414
335.1598
370.1284
386.1145
394.8949
420.8841
436.8726
446.8847
451.8478
463.8482
479.8567
490.8751
498.8994
m/z

39


MS
20160416-42 134 (0.535)
1: TOF MS ES+
5.53e3
100
%
0
476.1916
478.1932
389.1216
302.0633
304.0536
565.0863
567.0778
338.9236
345.0923
328.0761
371.1121
391.1206
306.0472
395.0589
347.0981
399.8236
432.1577
444.7735
453.7726
457.7642
479.2000
480.1860
482.8250
508.7490
531.7311
565.0610
569.0864
574.8153
603.0547
635.3157
641.9825
660.7395
671.0463
694.7602
m/z

40


MS
20160416-43 131 (0.519)
1: TOF MS ES+
6.63e3
100
%
0
504.1533
447.1584
502.1 23
561.1943
430.9114
448.1647
408.9146
450.9137
423.8407
439.1456
476.8714
480.7909
493.8438
506.1654
507.8156
519.8482
533.7953
559.2117
546.8007
576.8921
581.8115
591.7613
609.8065
616.2258
619.7468
634.7418
650.8215
671.0773
686.6754
692.8483
m/z