Electronic Supplementary Information

Total Synthesis of Pseudouridimycin and Its Epimer via Ugi-type Multicomponent Reaction

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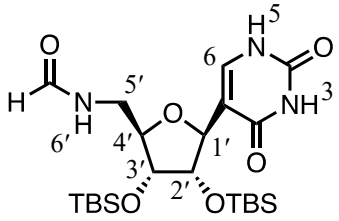
Table of Contents

General experimental methods

All reactions except that carried out in aqueous phase were performed under argon atmosphere, unless otherwise noted. Materials were purchased from commercial suppliers and used without further purification, unless otherwise noted. Solvents were distilled according to the standard protocol. Isolated yields were calculated by weighing products. The weight of the starting materials and the products were not calibrated. Analytical thin layer chromatography (TLC) was performed on Merck silica gel 60F_{254} plates. Normal-phase column chromatography was performed on Merck silica gel 5715 or Wakogel 60N. Flash column chromatography was performed on Kanto Chemical Silica Gel 60N (spherical, neutral, 40-50 μm). Hi-flash column chromatography was performed on YAMAZEN Hi-Flash^{TM} column silica gel (40 μm) or Fuji Silysia Chromatorex MB/PSQ (50-200 μm). Preparative thin-layer chromatography was performed on sigmaaldrich TLC Silica gel 60 F_{254} 25 Glass plates 20×20 cm. ^{1}H NMR spectra were measured in $CDCl_3$ or DMSO-d_6 solution and reported in parts per million (δ) relative to tetramethylsilane (0.00 ppm) as internal standard or residual solvent peaks of DMSO-d_6 (2.50 ppm) using JEOL ECS400 and ECX400P, unless otherwise noted. ^{13}C NMR spectra were measured in $CDCl_3$ or DMSO-d_6 solution and referenced to residual solvent peaks of $CDCl_3$ (77.0 ppm) or DMSO-d_6 (39.5 ppm) using JEOL ECS400 and ECX400P. Coupling constant (*J*) was reported in hertz (Hz). Abbreviations of multiplicity were as follows; s: singlet, d; doublet, t: triplet, q: quartet, m: multiplet, br: broad. Data were presented as follows; chemical shift (multiplicity, integration, coupling constant). Assignment was based on ^{1}H–^{1}H COSY, HMBC and HMQC NMR spectra. Mass spectra were obtained on Waters MICRO MASS LCT–premier and the mass analyzer type used for the HRMS measurements was TOF. Optical rotation was measured on a Rudolph Research Analytical Autopol IV automatic polarimeter.

1) Preparation of compounds

2′,3′-Di-*O*-*tert*-butyldimethylsilyl- 5′-deoxy-5′-*N*-formylaminopseudouridine (9)



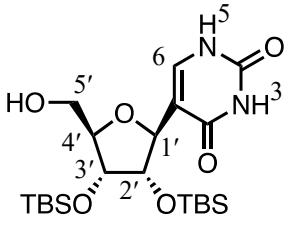
Preparation by route A. A suspension of pseudouridine (**6**, 3.00 g, 12.3 mmol) and imidazole (6.69 g, 98.3 mmol) in DMF was treated with *tert*-butyldimethylsilyl chloride (7.41 g, 49.1 mmol) at 50 °C for 20 h. Further imidazole (1.67 g, 24.6 mol) and *tert*-butyldimethylsilyl chloride (1.85 g, 12.3 mmol) were added to the reaction mixture, which was stirred for 24 h. The resulting mixture was concentrated *in vacuo* to give a crude 2′,3′,5′-tri-*O*-*tert*-butyldimethylsilylpseudouridine. 50% TFA/H_2O (65 mL) was slowly added to a solution of the residue in THF (130 mL) at 0 °C, and the mixture was stirred for 3.5 h. The resulting mixture was diluted with H_2O (50 mL) and extracted with AcOEt (100 mL × 3). The organic phase was washed with brine (80 mL), dried (Na_2SO_4), filtered, and concentrated *in vacuo* to give a crude 2′,3′-di-*O*-*tert*-butyldimethylsilylpseudouridine. A suspension of the residue in pyridine (100 mL) was treated with *p*-toluenesulfonyl chloride (5.86 g, 30.7 mmol) at 0 °C for 3 h. Further *p*-toluenesulfonyl chloride (2.93 g, 15.4 mmol) was added to the reaction mixture, which was stirred for further 3 h. The reaction mixture was quenched by adding cooled H_2O (0 °C, 50 mL) and extracted with AcOEt (100 mL). The organic phase was washed with brine (100 mL), dried (Na_2SO_4), filtered, and concentrated *in vacuo* to give a crude 2′,3′-di-*O*-*tert*-butyldimethylsilyl-5′-*O*-*p*-toluenesulfonylpseudouridine. A solution of the residue and NaN_3 (2.00 g, 30.7 mmol) in DMF (80 mL) was stirred at 70 °C for 10 h. Further NaN_3 (2.00 g, 30.7 mmol) was added to the reaction mixture, which was stirred for 22 h. The resulting mixture was coolded to room temperature and partitioned between hexane/AcOEt = 4/1 (200 mL) and H_2O (80 mL). The organic phase was washed with brine (80 mL), dried (Na_2SO_4), filtered, and concentrated *in vacuo* to give a crude 5′-azido-2′,3′-di-*O*-*tert*-butyldimethylsilyl-5′-deoxypseudouridine (**7**). A mixture of the residue and 5% Pd/C (1.00 g) in MeOH (40 mL) was vigorously stirred under H_2 atmosphere at 40 °C for 12 h. The catalyst was filtered off through a Celite pad, and the filtrate was concentrated *in vacuo* to give a crude 5′-amino-2′,3′-di-*O*-*tert*-butyldimethylsilyl-5′-deoxypseudouridine (**8**). A suspension of the residue and EDCI·HCl (5.89 g, 30.7 mmol) in CH_2Cl_2 (100 mL) was treated with formic acid (2.19 mL, 41.8 mmol) at 0 °C for 16 h. The reaction mixture was washed with H_2O (30 mL) and brine (80 mL), dried (Na_2SO_4), filtered, and concentrated *in vacuo.* The residue was purified by silica gel column chromatography (ϕ 10.5 × 15 cm, $CHCl_3$/MeOH: 100/0→99/1→98/2→97/3→96/4) to afford **9** (2.15 g, 4.30 mmol, 35% over 6 steps) as a white solid. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 11.14 (s, 1H, H3), 10.95 (s, 1H, H5), 8.18 (s, 1H, H6′), 8.05 (s, 1H, C*H*O), 7.49 (s, 1H, H6), 4.47 (d, 1H, H1′, $J_{1',2'}$ = 5.6 Hz), 4.35 (dd, 1H, H2′, $J_{2',1'}$ = 5.6, $J_{2',3'}$ = 4.2 Hz), 3.94 (dd, 1H, H3′, $J_{3',2'}$ = 4.2, $J_{3',4'}$ = 4.0 Hz), 3.81-3.76 (m, 1H, H4′), 3.38-3.22 (m, 2H, H5′), 0.85 (s, 18H, $Si(CH_3)_2C(CH_3)_3$), 0.03 (s, 12H, $Si(CH_3)_2C(CH_3)_3$); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 163.6, 161.2, 151.2, 141.3, 109.5, 82.0, 79.3, 74.0, 74.0, 48.6, 25.8, 17.8, -4.68; ESIMS-LR

m/z 522.24 [(M+Na)$^+$]; ESIMS-HR *m/z* calcd for $C_{22}H_{41}O_6N_3NaSi_2$ [(M+Na)$^+$] 522.2426, found 522.2417; $[\alpha]^{20}_D$ −49.93 (*C* 1.0, MeOH).

Preparation by route B. A suspension of pseudouridine (**6**, 3.00 g, 12.3 mmol), PPh_3 (4.83g, 18.4 mmol), imidazole (6.69 g, 98.3 mmol) and NaN_3 (2.40 g, 36.9 mmol) in DMF (60 mL) was treated with I_2 (4.68 g, 18.4 mmol) at room temperature for 2 h. The mixture was warmed to 40 °C and stirred for 20 h. *tert*-Butyldimethylsilyl chloride (7.41 g, 49.1 mmol) and imidazole (6.69 g, 98.3 mmol) were added to the mixture, which was stirred at the same temperature for 20 h. Further *tert*-butyldimethylsilyl chloride (3.70 g, 24.6 mmol) and imidazole (3.35 g, 49.1 mmol) were added to the mixture, which was stirred at the same temperature for 5 h. The mixture was diluted with THF (45 mL) and H_2O (25 mL), then PPh_3 (6.44 g, 24.6 mmol) was added to the mixture. The resulting whole mixture was stirred at the same temperature for 26 h. After cooling the reaction mixture to room temperature, the resulting mixture was diluted with H_2O (35 mL) and extracted with AcOEt (180 mL × 3). The organic phase was washed with brine (80 mL), dried (Na_2SO_4), filtered, and concentrated *in vacuo* to give a crude 5′-amino-2′,3′-di-*O*-*tert*-butyldimethylsilyl-5′-deoxypseudouridine. A suspension of the residue and EDCI·HCl (5.89 g, 30.7 mmol) in CH_2Cl_2 (100 mL) was treated with formic acid (2.19 mL, 41.8 mmol) at 0 °C for 4 h. The reaction mixture was washed with H_2O (30 mL) and brine (80 mL), dried (Na_2SO_4), filtered, and concentrated *in vacuo.* The residue was purified by silica gel column chromatography (ϕ 10.5 × 15 cm, $CHCl_3$/MeOH: 100/0→99/1→98/2→97/3→96/4) to afford **9** (3.74 g, 7.48 mmol, 61% over 4 steps) as a white solid.

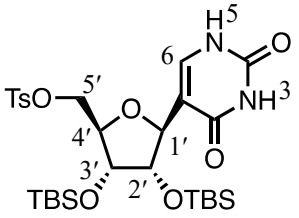
In route A, the analytically pure materials for compounds **6-9** were obtained by purification of less than 1% of the crude material in each step. Purification was conducted by preparative thin-layer chromatography (0-10% MeOH/$CHCl_3$) and all compounds were obtained as white solids. Data for each compound was described below.

2′,3′-Di-*O*-*tert*-butyldimethylsilylpseudouridine



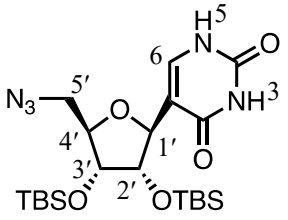
^{1}H NMR (DMSO-d_6, 400 MHz) δ 11.12 (s, 1H, H3), 10.99 (s, 1H, H5), 7.62 (s, 1H, H6), 4.90 (dd, 1H, O*H*, $J_{OH,5'}$ = 6.0, $J_{OH,5'}$ = 4.4 Hz), 4.48 (d, 1H, H1′, $J_{1',2'}$ = 4.4 Hz), 4.16 (dd, 1H, H2′, $J_{2',1'}$ = 4.4, $J_{2',3'}$ = 4.0 Hz), 4.06 (dd, 1H, H3′, $J_{3',\ 2'}$ = 4.0, $J_{3',4'}$ = 5.2 Hz), 3.76 (ddd, 1H, H4′, $J_{4',3'}$ = 5.2, $J_{4',5'}$ = 5.6, $J_{4',5'}$ = 3.2 Hz), 3.65 (ddd, 1H, H5′, $J_{5',4'}$ = 5.6, $J_{5',4'}$ = 11.6, $J_{5',OH}$ = 6.0 Hz), 3.43 (ddd, 1H, H5′, $J_{5',4'}$ = 3.2, $J_{5',5'}$ = 11.6, $J_{H5',OH}$ = 4.4 Hz), 0.87 (s, 18H, $Si(CH_3)_2C(CH_3)_3$), 0.04 (s, 12H, $Si(CH_3)_2C(CH_3)_3$); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 163.7, 151.2, 140.5, 110.5, 83.0, 79.0, 75.0, 71.7, 60.3, 25.8, 17.8, –4.69; ESIMS-LR *m/z* 495.23 [(M+Na)$^+$]; ESIMS-HR *m/z* calcd for $C_{21}H_{40}O_6N_2NaSi_2$ [(M+Na)$^+$] 495.2317, found 495.2308; $[\alpha]^{20}_D$ –44.82 (*C* 1.0, MeOH).

2′,3′-Di-*O-tert*-butyldimethylsilyl-5′-*O-p*-toluenesulfonylpseudouridine



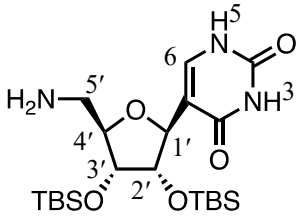
^{1}H NMR (DMSO-d_6, 400 MHz) δ 11.12 (s, 1H, H3), 10.99 (d, 1H, H5, $J_{5,6}$ = 5.0 Hz), 7.81 (d, 2H, H-*o*-Ph, $J_{o\text{-Ph},m\text{-Ph}}$ = 8.6 Hz), 7.49 (d, 2H, H-*m*-Ph, $J_{m\text{-Ph},o\text{-Ph}}$ = 8.6 Hz), 7.32 (d, 1H, H6, $J_{6,5}$ = 5.0 Hz), 4.44 (d, 1H, H1′, $J_{1',2'}$ = 5.0 Hz), 4.23 – 4.17 (m, 2H, H2′, H5′), 4.10 (dd, 1H, H5′, $J_{5',4'}$ = 5.4, $J_{5',5'}$ = 11.0 Hz), 3.93 (dd, 1H, H3′, $J_{3',2'}$ = 4.0, $J_{3',4'}$ = 5.4 Hz), 3.85 (dd, 1H, H4′, $J_{4',3'}$ = 5.4, $J_{4',5'}$ = 5.4 Hz), 2.42 (s, 3H, *p*-Me), 0.82 (s, 18H, Si(CH$_3$)$_2$C(C*H*$_3$)$_3$), 0.00 (s, 12H, Si(C*H*$_3$)$_2$C(CH$_3$)$_3$); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 163.2, 151.2, 145.2, 140.0, 132.0, 130.2, 127.7, 109.6, 79.9, 79.3, 74.0, 71.7, 69.4, 25.7, 21.1, 17.7, -4.75; ESIMS-LR *m/z* 649.24 [(M+Na)$^+$]; ESIMS-HR *m/z* calcd for $C_{28}H_{46}O_8N_2NaSSi_2$ [(M+Na)$^+$] 649.2406, found 649.2390; [α]$^{20}_D$ 16.31 (*C* 1.0, MeOH).

5′-Azido-2′,3′-di-*O-tert*-butyldimethylsilyl-5′-deoxypseudouridine (7)



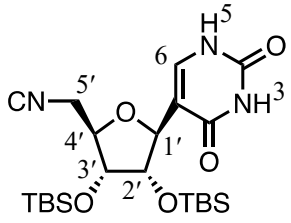
^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.99 (s, 1H, H5), 7.45 (s, 1H, H6), 4.50 (d, 1H, H1′, $J_{1',2'}$ = 4.0 Hz), 4.29 (dd, 1H, H2′, $J_{2',1'}$ = 4.0, $J_{2',3'}$ = 4.8 Hz), 4.01 (dd, 1H, H3′, $J_{3',2'}$ = 4.8, $J_{3',4'}$ = 4.8 Hz), 3.87 (dd, 1H, H4′, $J_{4',3'}$ = 4.8, $J_{4',5'}$ = 5.2 Hz), 3.54 (d, 2H, H5′, $J_{5',4'}$ = 5.2 Hz), 0.86 (s, 18H, Si(CH$_3$)$_2$C(C*H*$_3$)$_3$), 0.05 (s, 12H, Si(C*H*$_3$)$_2$C(CH$_3$)$_3$); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 163.3, 151.2, 140.4, 109.9, 80.7, 80.2, 74.4, 72.6, 51.4, 25.8, 17.8, –4.62; ESIMS-LR *m/z* 520.24 [(M+Na)$^+$]; ESIMS-HR *m/z* calcd for $C_{21}H_{39}O_5N_5NaSi_2$ [(M+Na)$^+$] 520.2382, found 520.2376; [α]$^{20}_D$ 63.20 (*C* 0.69, MeOH).

5′-*O*-Amino-2′,3′-di-*O-tert*-butyldimethylsilyl-5′-deoxypseudouridine (8)



^{1}H NMR (DMSO-d_6, 400 MHz) δ 7.55 (s, 1H, H6), 4.45 (d, 1H, H1′, $J_{1',2'}$ = 5.8 Hz), 4.34 (dd, 1H, H2′, $J_{2',1'}$ = 5.8, $J_{2',3'}$ = 4.4 Hz), 4.07 (dd, 1H, H3′, $J_{3',2'}$ = 4.4, $J_{3',4'}$ = 4.0 Hz), 3.89 (dd, 1H, H4′, $J_{4',3'}$ = 4.0, $J_{4',5'}$ = 6.4 Hz), 2.89 (d, 2H, H5′, $J_{5',4'}$ = 6.4 Hz), 0.86 (s, 18H, Si(CH$_3$)$_2$C(C*H*$_3$)$_3$), 0.04 (s, 12H, Si(C*H*$_3$)$_2$C(CH$_3$)$_3$); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 163.7, 151.1, 141.7, 109.0, 81.5, 79.9, 73.7, 73.6, 41.8, 25.8, 17.8, –4.64; ESIMS-LR *m/z* 472.27 [(M+H)$^+$]; ESIMS-HR *m/z* calcd for $C_{21}H_{42}O_5N_3Si_2$ [(M+H)$^+$] 472.2658, found 472.2651; [α]$^{20}_D$ –29.66 (*C* 1.00, MeOH).

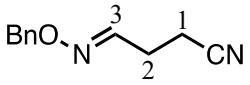
2′,3′-Di-*O-tert*-butyldimethylsilyl-5′-deoxy-5′-isocyanopseudouridine (10)



A mixture of **9** (0.50 g, 1.00 mmol) and Et$_3$N (1.40 mL, 10.0 mmol) in CH_2Cl_2 was treated with triphosgene (0.37 g, 1.25 mmol) at –78 °C for 2 h. After MeOH was added, the resulting mixture was stirred for additional 45 min and partitioned between AcOEt (50 mL) and H_2O (15 mL). The organic phase was washed with brine (15 mL), dried (Na_2SO_4), filtered, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (ϕ 2 × 12 cm, $CHCl_3$/MeOH: 100/0→99/1→98/2→97/3) to afford **10** (0.216 g, 0.449 mmol, 45%) as a white amorphous. ^{1}H NMR

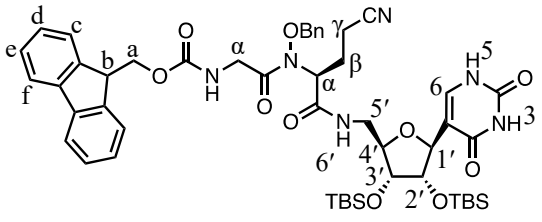
(DMSO-d_6, 400 MHz) δ 11.15 (s, 1H, H3), 10.97 (d, 1H, H5, $J_{5,6}$ = 5.4 Hz), 7.46 (d, 1H, H6, $J_{6,5}$ = 5.4 Hz), 4.52 (d, 1H, H1′, $J_{1',2'}$ = 4.4 Hz), 4.33 (dd, 1H, H2′, $J_{2',1'} = J_{2',3'}$ = 4.4 Hz), 4.02 (dd, 1H, H3′, $J_{3',2'}$ = 4.4, $J_{3',4'}$ = 4.8 Hz), 3.90-3.86 (m, 2H, H4′, H5′), 3.68 (dd, 1H, H5′, $J_{5',4'}$ = 6.4, $J_{H5'}$ = 16.8 Hz), 0.87 (s, 18H, $Si(CH_3)_2C(CH_3)_3$), 0.06 (s, 12H, $Si(CH_3)_2C(CH_3)_3$); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 163.3, 157.5, 151.2, 140.2, 109.6, 80.1, 78.9, 74.1, 72.6, 42.9, 25.8, 17.7, –4.87; ESIMS-LR *m/z* 504.23 [(M+Na)$^+$]; IR (neat) ν 2146.38 cm^{-1}; ESIMS-HR *m/z* calcd for $C_{22}H_{39}O_5N_3NaSi_2$ [(M+Na)$^+$] 504.2321, found 504.2310; $[\alpha]^{20}_D$ –6.73 ($_C$ 1.00, MeOH).

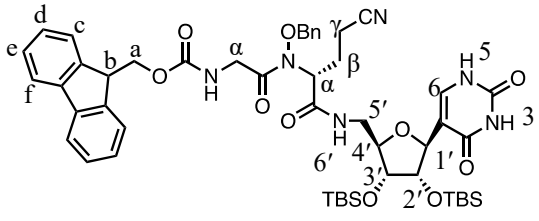
4-*N*-Benzyloxyiminobutanenitrile (11)



A solution of *O*-benzylhydroxylamine hydrochloride (1.50 g, 9.40 mmol) and 3-cyanopropionaldehyde dimethyl acetal (0.607 g, 4.70 mmol) in MeCN (7.5 mL) and H_2O (7.5 mL) was stirred at 80 °C for 5 h. The reaction mixture was cooled to room temperature, and diluted with Et_2O (45 mL). The organic phase was washed with brine (10 mL), dried (Na_2SO_4), filtered, and concentrated *in vacuo.* The residue was purified by silica gel column chromatography (ϕ 3 × 10 cm, hexane/AcOEt: 3/1) to afford **11** (0.829 g, 4.40 mmol, 94%) as a colorless oil. ^{1}H NMR (DMSO-d_6, 400 MHz, 5:3 geometric mixture) δ 7.49 (t, 1H, H3, $J_{3,2}$ = 4.6 Hz), 7.39-7.30 (m, 5H, Ph), 5.08 (s, 2H, $PhCH_2$), 2.61-2.51 (m, 4H, H1, H2); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 146.4, 137.4, 128.6, 128.2, 118.9, 76.5, 26.0, 22.1, 14.3; ESIMS-LR *m/z* 211.08 [(M+Na)$^+$]; ESIMS-HR *m/z* calcd for $C_{11}H_{12}ON_2Na$ [(M+Na)$^+$] 211.0842, found 211.0839.

5′-[2-(*S*)-*N*-Benzyloxy-*N*-(fluorenylmethyloxycarbonylaminoacetyl)amino-4-cyanobutanoylamino]-5′-deoxypseudouridine (13) and 5′-[2-(*R*)-*N*-benzyloxy-*N*-(fluorenylmethyloxycarbonylaminoacetyl)amino-4-cyanobutanoylamino]-5′-deoxypseudouridine (14)





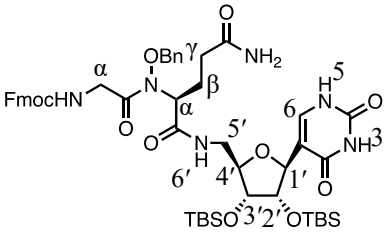
A suspension of **10** (0.40 g, 0.83 mmol), **11** (0.24 g, 1.25 mmol), Fmoc-Gly-OH (0.30 g, 1.00 mmol) and MS4A (1.60 g) in CH_2Cl_2 (8.3 mL) was treated with $ZnCl_2 \cdot Et_2O$ (1.66 mL, 1.66 mmol) at room temperature for 48 h. Molecular sieves 4A was filtered off through a Celite pad, and the filtrate was diluted with AcOEt (120 mL) and washed with saturated aqueous $NaHCO_3$ (60 mL) three times. The organic phase was washed with brine (50 mL), dried (Na_2SO_4), filtered, and concentrated *in vacuo.* The residue was purified by silica gel column chromatography (ϕ 4.5 × 15 cm, $CHCl_3$/MeOH: 100/0→99/1→98/2→97/3) to afford **13** (0.21 g, 0.22 mmol, 27%) and **14** (0.24 g, 0.28 mmol, 30%) as a white amorphous, respectively. Data for **13**: ^{1}H NMR (DMSO-d_6, 400 MHz) δ 11.14 (s, 1H, H3), 10.98 (s, 1H, H5), 8.34 (s, 1H, H6′), 7.89 (d, 2H, H-f, $J_{f,e}$ = 8.0 Hz), 7.73 (d, 2H, H-c, $J_{c,d}$ = 7.2 Hz), 7.60 (t, 1H, Gly-NH, $J_{Gly\text{-}NH,Gly\text{-}\alpha\text{-}CH}$ = 5.6 Hz), 7.50 (s, 1H, H6), 7.42-

7.31 (m, 9H, H-d, H-e, Ph), 5.07 (d, 1H, PhCH_2, $J_{PhCH_2,PhCH_2}$ = 9.8 Hz), 4.95 (d, 1H, PhCH_2, $J_{PhCH_2,PhCH_2}$ = 9.8 Hz), 4.80 (t, 1H, Gln-α-CH, $J_{Gln\text{-}\alpha\text{-}CH,Gln\text{-}\beta\text{-}CH}$ = 7.2 Hz), 4.42 (d, 1H, H1′, $J_{1',2'}$ = 5.6 Hz), 4.31-4.22 (m, 4H, H2′, H-a, H-b), 4.15 (dd, 1H, Gly-α-CH, $J_{Gly\text{-}\alpha\text{-}CH,Gly\text{-}NH}$ = 5.6, $J_{Gly\text{-}\alpha\text{-}CH,Gly\text{-}\alpha\text{-}CH}$ = 17.6 Hz), 4.03 (dd, 1H, Gly-α-CH, $J_{Gly\text{-}\alpha\text{-}CH,Gly\text{-}NH}$ = 5.6, $J_{Gly\text{-}\alpha\text{-}CH,Gly\text{-}\alpha\text{-}CH}$ = 17.6 Hz), 3.95-3.94 (m, 1H, H3′), 3.85-3.84 (m, 1H, H4′), 3.35-3.33 (m, 2H, H5′), 2.54-2.41 (m, 2H, Gln-γ-CH), 2.21 (td, 2H, Gln-β-CH, $J_{Gly\text{-}\beta\text{-}CH,Gln\text{-}\alpha\text{-}CH}$ = 7.2, $J_{Gly\text{-}\beta\text{-}CH,Gln\text{-}\gamma\text{-}CH}$ = 8.6 Hz), 0.84 (s, 18H, $Si(CH_3)_2C(CH_3)_3$), 0.02 (s, 12H, $Si(CH_3)_2C(CH_3)_3$); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 168.2, 163.5, 156.6, 151.2, 143.9, 141.2, 140.8, 134.4, 129.4, 128.9, 128.6, 127.7, 127.1, 125.3, 120.1, 119.9, 109.5, 81.5, 79.5, 79.2, 77.7, 74.0, 74.0, 65.8, 60.1, 46.6, 41.8, 41.8, 25.8, 24.4, 17.7, 13.8, –4.70; ESIMS-LR *m/z* 989.43 [(M+Na)$^+$]; ESIMS-HR *m/z* calcd for $C_{50}H_{66}O_{10}N_6NaSi_2$ [(M+Na)$^+$] 989.4271, found 989.4259; $[\alpha]^{20}_D$ –52.0 (C 0.94, $CHCl_3$). Data for **14**: ^{1}H NMR (DMSO-d_6, 400 MHz) δ 11.13 (s, 1H, H3), 10.96 (s, 1H, H5), 8.35 (s, 1H, H6′), 7.89 (d, 2H, H-f, $J_{f,e}$ = 7.6 Hz), 7.73 (d, 2H, H-c, $J_{c,d}$ = 7.2 Hz), 7.58 (t, 1H, Gly-NH, $J_{Gly\text{-}NH,Gly\text{-}\alpha\text{-}CH}$ = 6.0 Hz), 7.49 (s, 1H, H6), 7.44-7.31 (m, 9H, H-d, H-e, Ph), 5.09 (d, 1H, PhCH_2, $J_{PhCH_2,PhCH_2}$ = 10.0 Hz), 4.95 (d, 1H, PhCH_2, $J_{PhCH_2,PhCH_2}$ = 10.0 Hz), 4.80 (t, 1H, Gln-α-CH, $J_{Gln\text{-}\alpha\text{-}CH,Gln\text{-}\beta\text{-}CH}$ = 6.4 Hz), 4.42 (d, 1H, H1′, $J_{1',2'}$ = 4.4 Hz), 4.32-4.21 (m, 4H, H2′, H-a, H-b), 4.14 (dd, 1H, Gly-α-CH, $J_{Gly\text{-}\alpha\text{-}CH,Gly\text{-}NH}$ = 5.6, $J_{Gly\text{-}\alpha\text{-}CH,Gly\text{-}\alpha\text{-}CH}$ = 18.0 Hz), 4.02 (dd, 1H, Gly-α-CH, $J_{Gly\text{-}\alpha\text{-}CH,Gly\text{-}NH}$ = 5.6, $J_{Gly\text{-}\alpha\text{-}CH,Gly\text{-}\alpha\text{-}CH}$ = 18.0 Hz), 3.93-3.92 (m, 1H, H3′), 3.85-3.84 (m, 1H, H4′), 3.39-3.27 (m, 2H, H5′), 2.57-2.42 (m, 2H, Gln-γ-CH), 2.27-2.15 (m, 2H, Gln-β-CH), 0.84 (s, 18H, $Si(CH_3)_2C(CH_3)_3$), 0.02 (s, 12H, $Si(CH_3)_2C(CH_3)_3$); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 168.2, 163.5, 156.6, 151.2, 143.9, 141.0, 140.8, 134.5, 129.3, 128.8, 128.5, 127.7, 127.1, 125.3, 120.2, 119.9, 109.7, 81.4, 79.6, 79.2, 77.7, 74.2, 74.0, 65.8, 60.2, 46.6, 42.0, 41.7, 25.8, 24.5, 17.7, 13.8, –4.70; ESIMS-LR *m/z* 989.43 [(M+Na)$^+$]; ESIMS-HR *m/z* calcd for $C_{50}H_{66}O_{10}N_6NaSi_2$ [(M+Na)$^+$] 989.4271, found 989.4254; $[\alpha]^{20}_D$ –15.26 (C 1.00, $CHCl_3$).

General procedure of consideration of Ugi reaction in Table

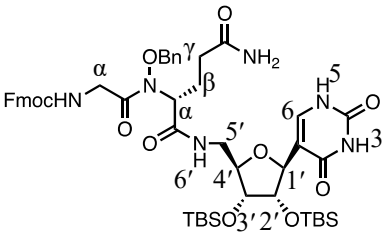
A suspension of **10** (5.0 mg, 10.4 μmol), **11** (2.93 mg, 15.6 μmol), Fmoc-Gly-OH (3.7 mg, 12.5 μmol) and MS4A (20 mg) in CH_2Cl_2 (100 μL) was treated with Lewis acid (20.8 μmol) at room temperature for 48 h. Molecular sieves 4A was filtered off through a Celite pad, and the filtrate was purified by preparative thin-layer chromatography ($CHCl_3$/MeOH: 96/4) to afford **13** and **14** as a white amorphous, respectively. The results are described in Table 1.

5′-(2-(*S*)- *N*-Benzyloxy-*N*-fluorenylmethyloxycarbonylaminoacetylamino-4-carbamoyl-butanoyl)amino-5′-deoxypseudouridine (15)



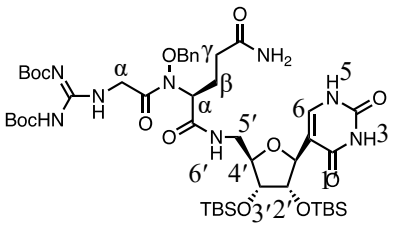
A solution of **13** (40.1 mg, 41.5 μmol) and acetaldoxime (73.5 μL, 1.24 mmol) in toluene (420 μL) was treated with $InCl_3$ (13.8 mg, 62.4 mmol) at room temperature for 12 h. The resulting mixture was concentrated *in vacuo* with silica gel. The silica gel charging the crude material was purified by silica gel column chromatography (ϕ 2 × 7 cm, $CHCl_3$/MeOH: 100/0→99/1→98/2→97/3) to afford **15** (42.3 mg, 41.5 mmol, quant.) as a white solid. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 11.13 (s, 1H, H3), 10.95 (s, 1H, H5), 8.25 (s, 1H, H6′), 7.90 (d, 2H, H-f, $J_{f,e}$ = 7.6 Hz), 7.73 (d, 2H, H-c, $J_{c,d}$ = 7.2 Hz), 7.56-7.22 (m, 12H, Gly-NH, H6, H-d, H-e, Ph, Gln-NH), 6.78 (s, 1H, Gln-NH), 5.11 (m, 1H, PhCH_2), 4.95 (m, 1H, PhCH_2), 4.76 (s, 1H, Gln-α-CH), 4.42 (d, 1H, H1′, $J_{1',2'}$ = 5.6 Hz), 4.31-4.29 (m, 4H, H2′, H-a, H-b), 4.13 (dd, 1H, Gly-α-CH, $J_{\text{Gly-α-CH,Gly-NH}}$ = 3.6, $J_{\text{Gly-α-CH,Gly-α-CH}}$ = 10.8 Hz), 4.03 (dd, 1H, Gly-α-CH, $J_{\text{Gly-α-CH,Gly-NH}}$ = 3.6, $J_{\text{Gly-α-CH,Gly-α-CH}}$ = 10.8 Hz), 3.93 (s, 1H, H3′), 3.84 (m, 1H, H4′), 3.36-3.34 (m, 2H, H5′), 2.09-2.07 (m, 4H, Gln-β-CH, Gln-γ-CH), 0.83 (s, 18H, $Si(CH_3)_2C(CH_3)_3$), 0.02 (s, 12H, $Si(CH_3)_2C(CH_3)_3$); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 173.3, 172.5, 169.2, 163.5, 156.6, 151.2, 143.8, 141.1, 140.7, 134.6, 129.3, 128.7, 128.5, 127.6, 127.1, 125.3, 120.1, 109.6, 81.6, 79.5, 79.2, 77.6, 74.1, 74.0, 65.8, 61.0, 46.6, 41.7, 31.7, 26.2, 24.1, 17.7, –4.73; ESIMS-LR *m/z* 1007.44 [(M+Na)$^+$]; ESIMS-HR *m/z* calcd for $C_{50}H_{68}O_{11}N_6NaSi_2$ [(M+Na)$^+$] 1007.4377, found 1007.4370; $[\alpha]^{20}_D$ –18.53 ($_C$ 1.00, $CHCl_3$).

5′-(2-(*R*)-*N*-Benzyloxy-*N*-fluorenylmethyloxycarbonylaminoacetylamino-4-carbamoyl-butanoyl)amino-5′-deoxypseudouridine (18)



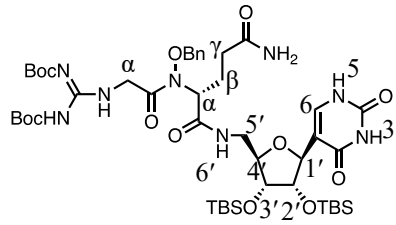
A solution of **14** (52.8 mg, 54.6 μmol) and acetaldoxime (96.7 μL, 1.64 mmol) in toluene (550 μL) was treated with $InCl_3$ (18.1 mg, 81.8 mmol) at room temperature for 9 h. The resulting mixture was concentrated *in vacuo* with silica gel. The silica gel charging the crude material was purified by silica gel column chromatography (ϕ 2 × 7 cm, $CHCl_3$/MeOH: 100/0→99/1→98/2→97/3) to afford **18** (54.8 mg, 54.6 mmol, quant.) as a white solid. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 11.12 (s, 1H, H3), 10.94 (s, 1H, H5), 8.22 (s, 1H, H6′), 7.89 (d, 2H, H-f, $J_{f,e}$ = 7.6 Hz), 7.73 (d, 2H, H-c, $J_{c,d}$ = 7.2 Hz), 7.59-7.23 (m, 12H, Gly-NH, H6, H-d, H-e, Ph, Gln-NH), 6.79 (s, 1H, Gln-NH), 5.11 (m, 1H, PhCH_2), 4.93 (m, 1H, PhCH_2), 4.75 (s, 1H, Gln-α-CH), 4.42 (d, 1H, H1′, $J_{1',2'}$ = 5.2 Hz), 4.33-4.22 (m, 4H, H2′, H-a, H-b), 4.12 (dd, 1H, Gly-α-CH, $J_{\text{Gly-α-CH,Gly-NH}}$ = 6.0, $J_{\text{Gly-α-CH,Gly-α-CH}}$ = 17.6 Hz), 4.03 (dd, 1H, Gly-α-CH, $J_{\text{Gly-α-CH,Gly-NH}}$ = 6.0, $J_{\text{Gly-α-CH,Gly-α-CH}}$ = 17.6 Hz), 3.93-3.92 (m, 1H, H3′), 3.84-3.82 (m, 1H, H4′), 3.35-3.27 (m, 2H, H5′), 2.08-2.06 (m, 4H, Gln-β-CH, Gln-γ-CH), 0.83 (s, 18H, $Si(CH_3)_2C(CH_3)_3$), 0.00 (s, 12H, $Si(CH_3)_2C(CH_3)_3$); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 173.4, 172.4, 169.2, 163.5, 156.6, 151.2, 143.9, 141.0, 140.8, 134.7, 129.3, 128.7, 128.5, 127.7, 127.1, 125.3, 120.1, 109.8, 81.6, 79.7, 79.2, 77.7, 74.2, 74.1, 65.9, 61.1, 46.7, 41.8, 31.7, 25.8, 24.2, 17.8, –4.69; ESIMS-LR *m/z* 1007.44 [(M+Na)$^+$]; ESIMS-HR *m/z* calcd for $C_{50}H_{68}O_{11}N_6NaSi_2$ [(M+Na)$^+$] 1007.4377, found 1007.4367; $[\alpha]^{20}_D$ –20.52 ($_C$ 0.50, $CHCl_3$).

5′-[2-(*S*)- *N*-Benzyloxy-(*N*,*N*′-di-*tert*-butoxycarbonyl-1-carboxamidylaminoacetyl)amino-4-carbamoyl-butanoyl]amino -5′-deoxypseudouridine (17)



A solution of **15** (23.7 mg, 24.1 μmol) and *N*,*N*′-di-Boc-1*H*-pyrazole-1-carboxamidine (11.2 mg, 36.1 μmol) in DMF (220 μL) was treated with DBU (4.39 μL, 28.9 μmol) at room temperature for 4 h. The resulting mixture was concentrated *in vacuo* with silica gel. The silica gel charging the crude material was purified by silica gel column chromatography (ϕ 1.5 × 9 cm, $CHCl_3$/MeOH: 100/0→99/1→98/2→97/3→96/4) to afford **17** (19.6 mg, 19.5 μmol, 81%) as a white solid. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 11.45 (s, 1H, Gua-NH), 11.13 (s, 1H, H3), 10.93 (d, 1H, H5, J_{H6} = 5.6 Hz), 8.76 (s, 1H, Gly-NH), 8.21 (s, 1H, H6′), 7.49 (d, 1H, H6, J_5 = 5.6 Hz), 7.44-7.39 (m, 5H, Ph), 7.19 (s, 1H, Gln-NH), 6.76 (s, 1H, Gln-NH), 5.13 (d, 1H, PhCH_2, $J_{PhCH_2,PhCH_2}$ = 10.4 Hz), 5.00 (d, 1H, PhCH_2, $J_{PhCH_2,PhCH_2}$ = 10.4 Hz), 4.76-4.72 (m, 1H, Gln-α-CH), 4.41-4.40 (m, 3H, H1′, Gly-α-CH), 4.34 (dd, 1H, H2′, $J_{2',1'}$ = 5.2, $J_{2',3'}$ = 4.4 Hz), 3.92 (dd, 1H, H3′, $J_{3',2'}$ = 4.4, $J_{3,'4'}$ = 4.0 Hz), 3.84 (dd, 1H, H4′, $J_{4',3'}$ = 4.0, $J_{4',5'}$ = 5.6 Hz), 3.31 (m, 2H, H5′), 2.18-2.07 (m, 4H, Gln-β-CH Gln-γ-CH), 1.49 (s, 9H, H-^{t}Bu), 1.38 (s, 9H, H-^{t}Bu), 0.84 (s, 18H, $Si(CH_3)_2C(CH_3)_3$), 0.02 (s, 12H, $Si(CH_3)_2C(CH_3)_3$); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 173.3, 171.1, 169.0, 163.5, 162.8, 155.0, 151.9, 151.1, 141.2, 134.5, 129.2, 128.7, 128.5, 109.5, 83.1, 81.9, 79.5, 79.2, 78.3, 77.6, 73.9, 61.1, 42.5, 41.6, 31.6, 27.6, 25.7, 24.0, 17.7, –4.77; ESIMS-LR *m/z* 1027.49 [(M+Na)$^+$]; ESIMS-HR *m/z* calcd for $C_{46}H_{76}O_{13}N_8NaSi_2$ [(M+Na)$^+$] 1027.4963, found 1027.4934; $[\alpha]^{20}_D$ –40.16 ($_C$ 0.45, $CHCl_3$).

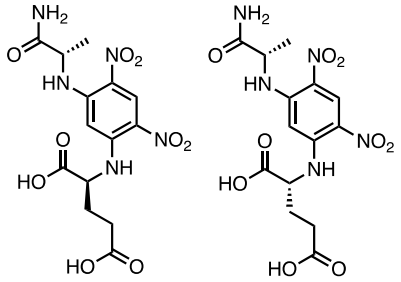
5′-[2-(*R*)-*N*-Benzyloxy-(*N*,*N*′-di-*tert*-butoxycarbonyl-1-carboxamidylaminoacetyl)amino-4-carbamoyl-butanoyl]amino-5′-deoxypseudouridine (21)



A solution of **18** (20.9 mg, 21.2 μmol) and *N*,*N*′-di-Boc-1*H*-pyrazole-1-carboxamidine (9.87 mg, 31.8 μmol) in DMF (194 μL) was treated with DBU (3.84 μL, 25.5 μmol) at room temperature for 4 h. The resulting mixture was concentrated *in vacuo* with silica gel. The silica gel charging the crude material was purified by silica gel column chromatography (ϕ 1.5 × 7 cm, $CHCl_3$/MeOH: 100/0→99/1→98/2→97/3) to afford **21** (18.3 mg, 18.2 μmol, 86%) as a white solid. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 11.46 (s, 1H, Gua-NH), 11.11 (s, 1H, H3), 10.94 (s, 1H, H5), 8.75 (s, 1H, Gly-NH), 8.22 (s, 1H, H6′), 7.50 (s, 1H, H6), 7.44-7.38 (m, 5H, Ph), 7.21 (s, 1H, Gln-NH), 6.77 (s, 1H, Gln-NH), 5.16 (d, 1H, PhCH_2, $J_{PhCH_2,\ PhCH_2}$ = 9.6 Hz), 4.99 (d, 1H, PhCH_2, $J_{PhCH_2,\ PhCH_2}$ = 9.6 Hz), 4.77-4.71 (m, 1H, Gln-α-CH), 4.41-4.40 (m, 3H, H1′, Gly-α-CH), 4.34 (dd, 1H, H2′, $J_{2',1'}$ = 5.6, $J_{2',3'}$ = 4.2 Hz), 3.93 (dd, 1H, H3′, $J_{3',2'}$ = 4.2, $J_{3,'4'}$ = 4.0 Hz), 3.84 (dd, 1H, H4′, $J_{4',3'}$ = 4.0, $J_{4',5'}$ = 6.0 Hz), 3.32 (m, 2H, H5′), 2.17-2.08 (m, 4H, Gln-β-CH Gln-γ-CH), 1.49 (s, 9H, H-^{t}Bu), 1.39 (s, 9H, H-^{t}Bu), 0.84 (s, 18H, $Si(CH_3)_2C(CH_3)_3$), 0.02 (s, 12H, $Si(CH_3)_2C(CH_3)_3$); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 173.3, 170.9, 169.1, 163.5, 162.8, 155.1, 152.0, 151.2, 141.2, 134.6, 129.2, 128.8, 128.5, 109.5, 83.1, 81.9, 79.6, 79.2, 78.3, 77.7, 74.0, 61.3, 42.5, 41.7, 31.6, 27.8, 25.8,

24.1, 17.7, –4.81; ESIMS-LR *m/z* 1027.49 [(M+Na)$^+$]; ESIMS-HR *m/z* calcd for $C_{46}H_{76}O_{13}N_8NaSi_2$ [(M+Na)$^+$] 1027.4963, found 1027.4928; $[\alpha]^{20}_D$ −35.47 (*c* 1.00, $CHCl_3$).

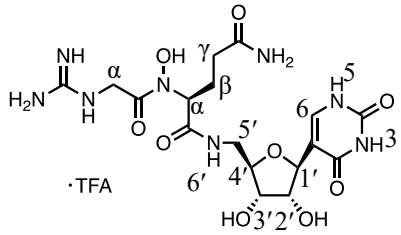
Preparation of L-FDAA derivatives (19) and (20)



A solution of **15** or **16** (1.00 mg, 0.99 μmol) and 5% Pd/C (10.0 mg) in MeOH (1 mL) was vigorously stirred under H_2 atmosphere at room temperature for 14 h. The catalyst was filtered off through a Celite pad, and the filtrate was concentrated *in vacuo*. The residue or amino acid standard (Boc-Gln-OH or Boc-D-Gln-OH) (1 mg, 4.06 μmol) was dissolved in 6 N aqueous HCl at 95 °C for 24 h. The resulting mixture was cooled to room temperature and *in vacuo*. The residue in saturated aqueous $NaHCO_3$ (50 μL) and acetone (200 μL) was treated with Marfey's reagent (*N*-α-(2,4-dinitro-5-fluorophenyl)-L-alaninamide, L-FDAA) (1.6 mg, 5.97 μL) at 40 °C for 16 h. The resulting mixture was diluted with MeOH and filtered (0.45 μm PTFE) prior to LC-MS analysis.

Conditions of LC-MS analysis: LC-MS equipment: Shimadzu, Prominence-I LC-2030CPlus, LCMS-8040; column: COSMOSIL Packed Column 5C18-PAQ, 4.6ID × 250mm, column oven: 30 °C; eluent: isocratic elution of 15% B over 1 min; linear gradient elution of 15% B-50% B over 40 min; linear gradient elution of 50% B-90% B over 1 min; isocratic elution of 90% B over 5 min; linear gradient elution of 90% B-15% B over 1 min; isocratic elution of 15% B over 3 min (where solvent A was 0.1% TFA in H_2O and solvent B was MeCN), flow rate: 0.40 mL/min, detection: UV (340 nm), method file: 15-50% MeCN over 45 min SIM l cm.

Pseudouridimycin trifluoroacetate (1)

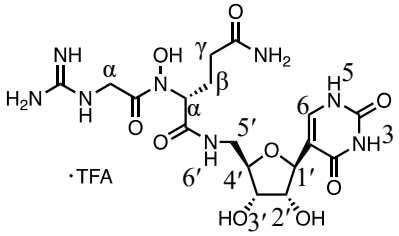


A solution of **17** (19.0 mg, 18.9 μmol) in CH_2Cl_2 (1.6 mL) was treated with 1 M BCl_3 in CH_2Cl_2 (1.6 mL, 1.6 mmol) at −78 °C for 5 min. The mixture was warmed to 0 °C and stirred for additional 2 h. The reaction mixture was added by cooled MeOH, then warmed to room temperature and stirred for 1.5 h. The mixure was concentrated *in vacuo*, and the resulting residue was purified by ODS silica gel column chromatography (ϕ 2.3 × 8 cm, 2 mM heptafluorobutyric acid (HFBA) in MeCN/2 mM HFBA in H_2O: 0-10%) to afford **1** as a HFBA salt (9.20 mg, 13.1 μmol, 69%) as a white form. To exchange the counter anion, the HFBA salt was treated with 0.1% TFA/MeCN and azeotroped several times to give **1** as a white solid. **1**: ^{1}H NMR (DMSO-d_6, 400 MHz) δ 11.13 (s, 1H, H3), 10.89 (d, 1H, H5, $J_{5,6}$ = 5.6 Hz), 9.84 (s, 1H, Gua-NH), 7.91 (t, 1H, H6′, $J_{6',5'}$ = 4.8 Hz), 7.40 (s, 1H, Gly-NH), 7.33 (d, 1H, H6, $J_{6,5}$ = 5.6 Hz), 7.11 (s, 1H, Gln-NH), 6.90 (s, 1H, Gln-NH), 4.79-4.77 (m, 1H, Gln-α-CH), 4.41 (d, 1H, H1′, $J_{1',2'}$ = 4.4 Hz), 4.21 (dd, 1H, Gly-α-CH, $J_{\text{Gly-α-CH,Gly-NH}}$ = 4.8, $J_{\text{Gly-α-CH,Gly-α-CH}}$ = 18.4 Hz), 4.11 (dd, 1H, Gly-α-CH, $J_{\text{Gly-α-CH,Gly-}}$

$_{NH}$ = 4.8, $J_{Gly\text{-}\alpha\text{-}CH,Gly\text{-}\alpha\text{-}CH}$ = 18.4 Hz), 3.99-3.98 (m, 3H, H2′), 3.73-3.71 (m, 2H, H3′, H4′), 3.34-3.28 (m, 2H, H5′), 2.11-2.09 (m, 3H, Gln-β-CH, Gln-γ-CH), 2.00-1.95 (m, 1H, Gln-β-CH); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 174.1, 169.0, 168.7, 163.5, 157.0, 151.2, 140.1, 110.5, 81.0, 79.6, 73.3, 72.3, 59.3, 42.6, 41.6, 31.6, 23.4; ^{19}F NMR (DMSO-d_6, 376 MHz) δ −74.4; ESIMS-LR *m/z* 487.19 [(M+H)$^+$]; ESIMS-HR *m/z* calcd for $C_{17}H_{27}O_9N_8$ [(M+H)$^+$] 487.1896, found 487.1888; $[\alpha]^{20}_D$ −4.13 (*c* 0.16, MeOH).

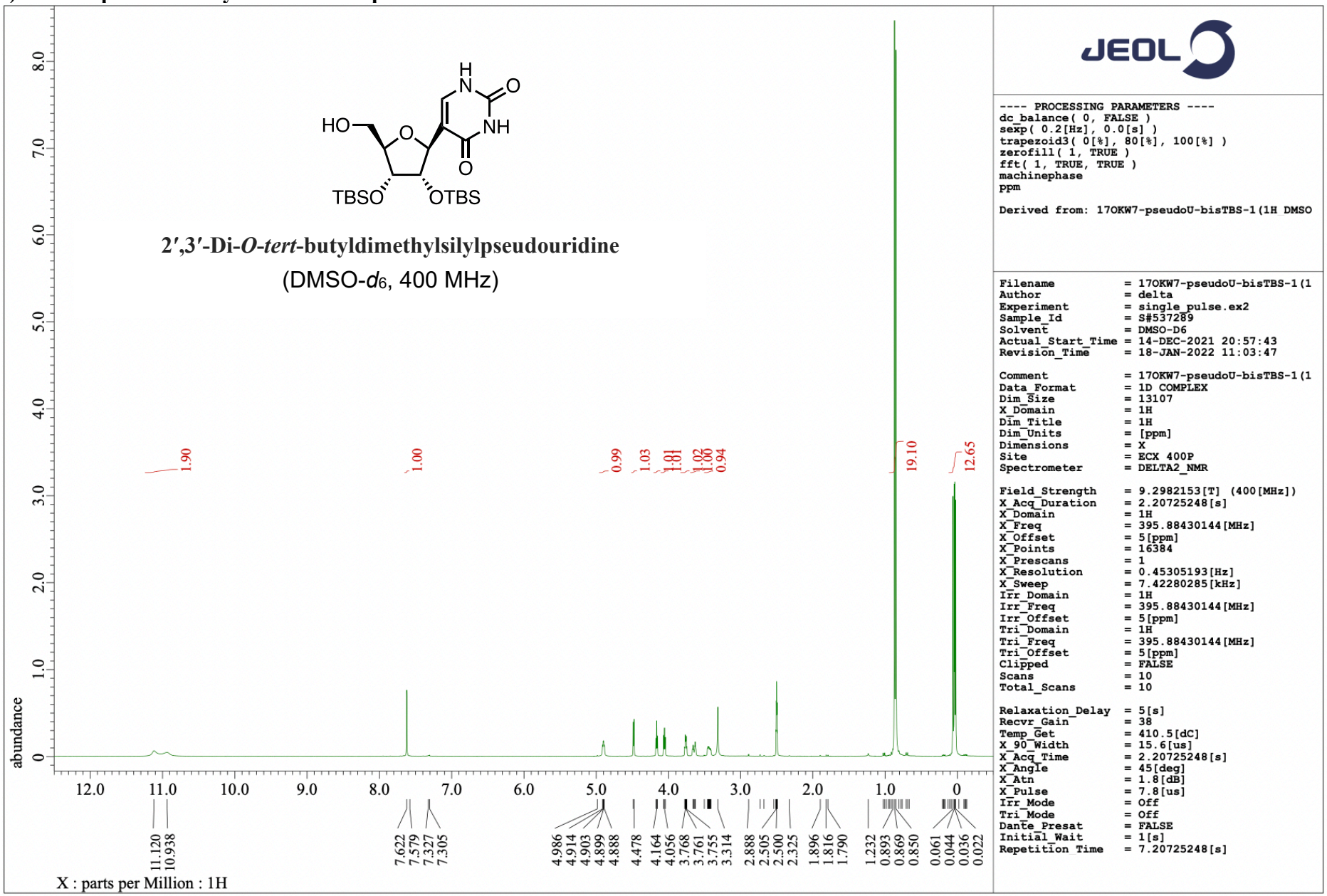
※ Optical rotation of the sample of pseudouridimycin (MCE®) (purity 89%) was −5.79 (*c* 0.098, MeOH).

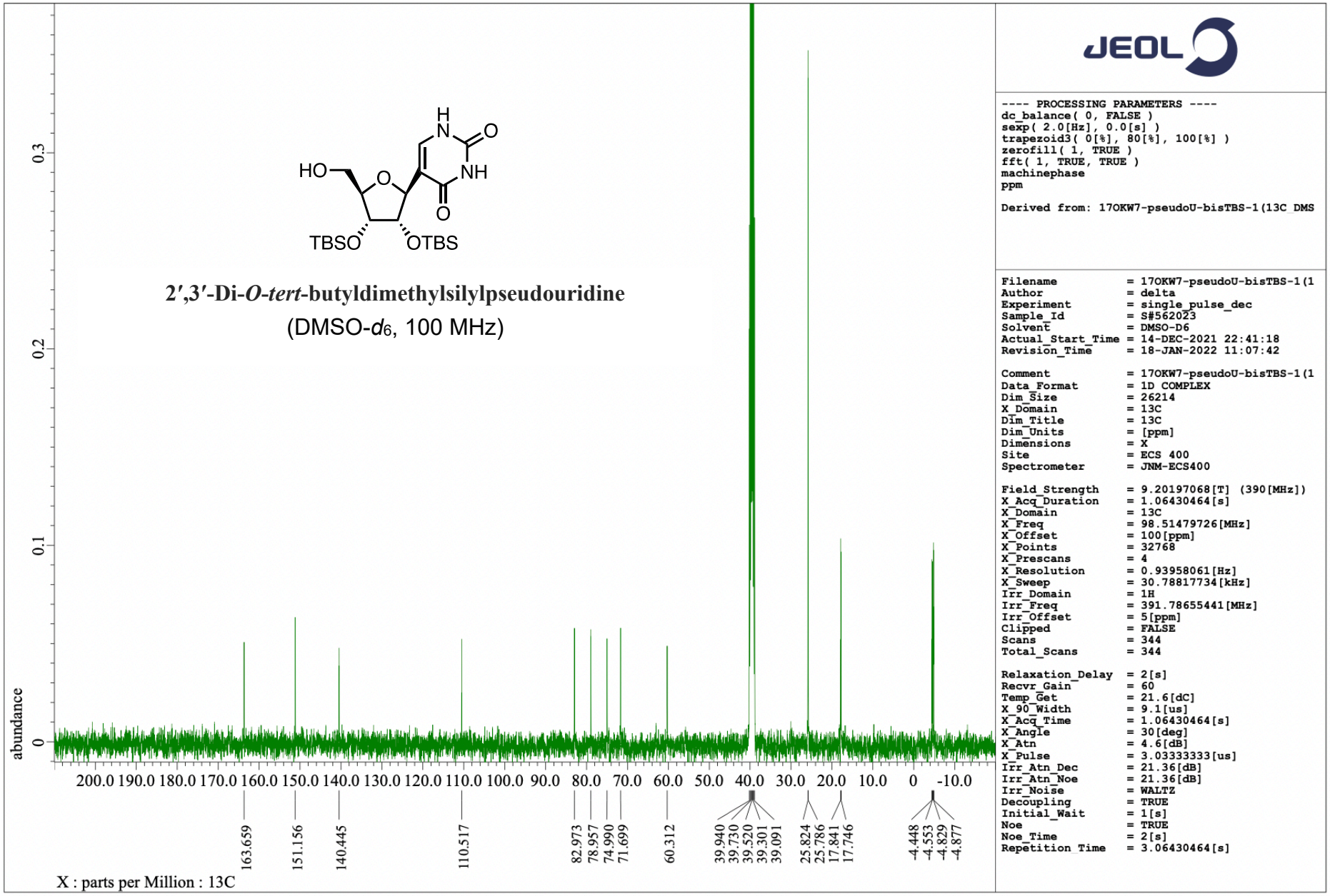
epi-Pseudouridimycin trifluoroacetate (_epi_-1)



A solution of **21** (19.8 mg, 19.7 μmol) in CH_2Cl_2 (1.6 mL) was treated with 1 M BCl_3 in CH_2Cl_2 (1.6 mL, 1.6 mmol) at -78 °C for 5 min. The mixture was warmed to 0 °C and stirred for additional 2 h. The reaction mixture was added by cooled MeOH, then warmed to room temperature and stirred for 1.5 h. The mixure was concentrated *in vacuo*, and the resulting residue was purified by ODS silica gel column chromatography (ϕ 2.3 × 8 cm, 2mM heptafluorobutyric acid (HFBA) in MeCN/2 mM HFBA in H_2O: 0-10%) to afford **1** as a HFBA salt (9.80 mg, 14.0 μmol, 71%) as a white form. To exchange the counter anion, the HFBA salt was treated with 0.1% TFA/MeCN and azeotroped several times to give **1** as a white solid. **1**: ^{1}H NMR (DMSO-d_6, 400 MHz) δ 11.13 (s, 1H, H3), 10.93 (d, 1H, H5, J_{H6} = 5.2 Hz), 9.85 (s, 1H, Gua-NH), 7.91 (t, 1H, H6′, $J_{6',5'}$ = 5.2 Hz), 7.41 (s, 1H, Gly-NH), 7.34 (d, 1H, H6, $J_{6,5}$ = 5.2 Hz), 7.14 (s, 1H, Gln-NH), 6.90 (s, 1H, Gln-NH), 4.79-4.77 (m, 1H, Gln-α-CH), 4.41 (d, 1H, H1′, $J_{1',2'}$ = 5.2 Hz), 4.22-4.17 (m, 1H, Gly-α-CH), 4.10 (dd, 1H, Gly-α-CH, $J_{Gly\text{-}\alpha\text{-}CH,Gly\text{-}NH}$ = 4.4, $J_{Gly\text{-}\alpha\text{-}CH,Gly\text{-}\alpha\text{-}CH}$ = 18.0 Hz), 4.00-3.99 (m, 3H, H2′), 3.73-3.71 (m, 2H, H3′, H4′), 3.42-3.38 (m, 1H, H5′), 3.19-3.16 (m, 1H, H5′), 2.11-2.08 (m, 3H, Gln-β-CH, Gln-γ-CH), 1.99-1.92 (m, 1H, Gln-β-CH); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 174.2, 169.0, 168.7, 163.6, 157.0, 151.2, 140.3, 110.5, 81.2, 79.6, 73.2, 72.3, 59.3, 42.6, 41.6, 31.6, 23.4; ^{19}F NMR (DMSO-d_6, 376 MHz) δ -73.4; ESIMS-LR *m/z* 487.18 [(M+H)$^+$]; ESIMS-HR *m/z* calcd for $C_{17}H_{27}O_9N_8$ [(M+H)$^+$] 487.1896, found 487.1890; $[\alpha]^{20}_D$ −10.55 (*c* 0.91, MeOH).

2) NMR spectrum of synthesized compounds




HO
O
H
N
O
NH
O
TBSO
OTBS
2′,3′-Di-O-tert-butyldimethylsilylpseudouridine
(DMSO-d6, 100 MHz)
abundance
0
0.1
0.2
0.3
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
163.659
151.156
140.445
110.517
82.973
78.957
74.990
71.699
60.312
39.940
39.730
39.520
39.301
39.091
25.824
25.786
17.841
17.746
-4.448
-4.553
-4.829
-4.877
X : parts per Million : 13C
JEOL
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW7-pseudoU-bisTBS-1(13C DMS
Filename = 17OKW7-pseudoU-bisTBS-1(1
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#562023
Solvent = DMSO-D6
Actual_Start_Time = 14-DEC-2021 22:41:18
Revision_Time = 18-JAN-2022 11:07:42
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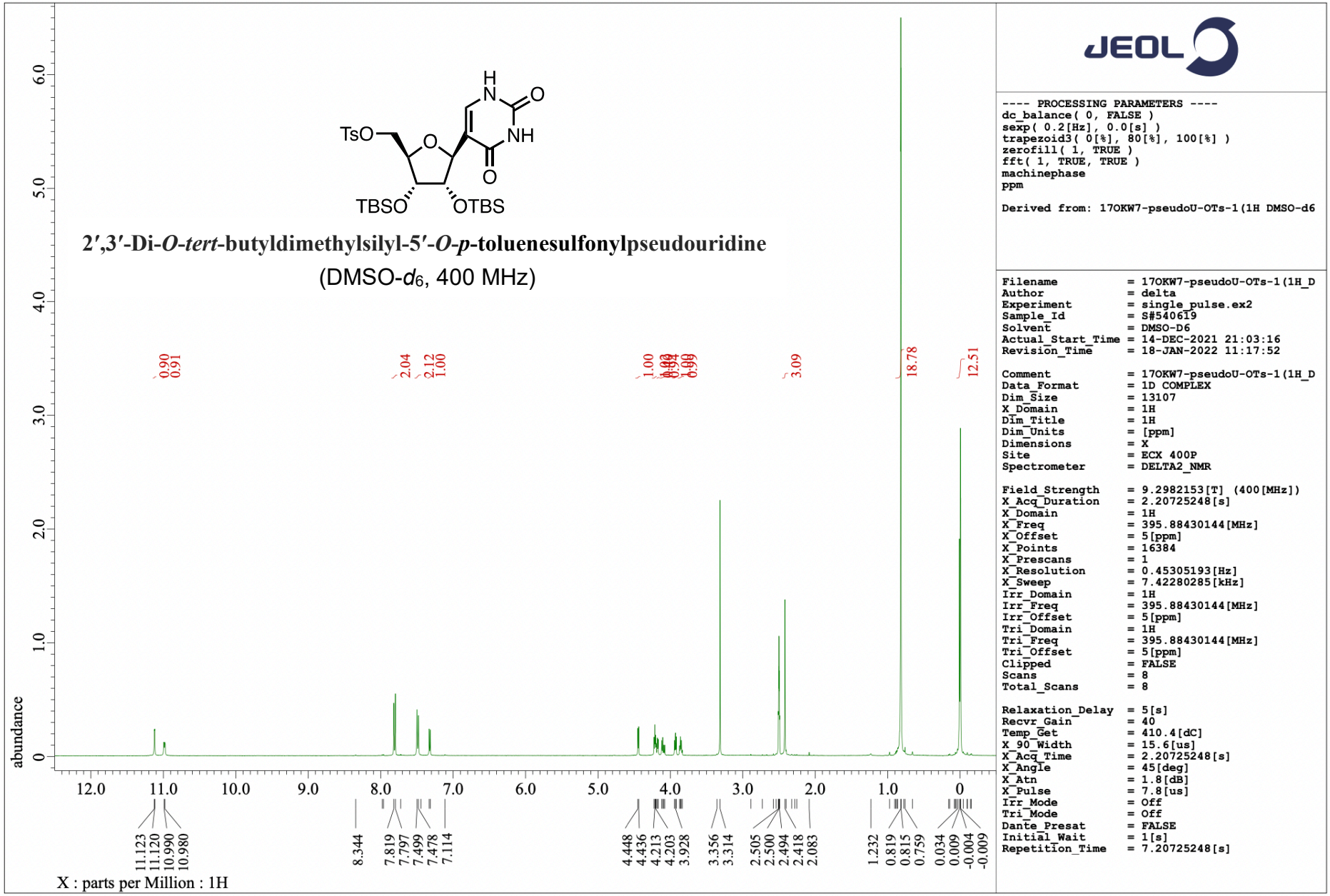
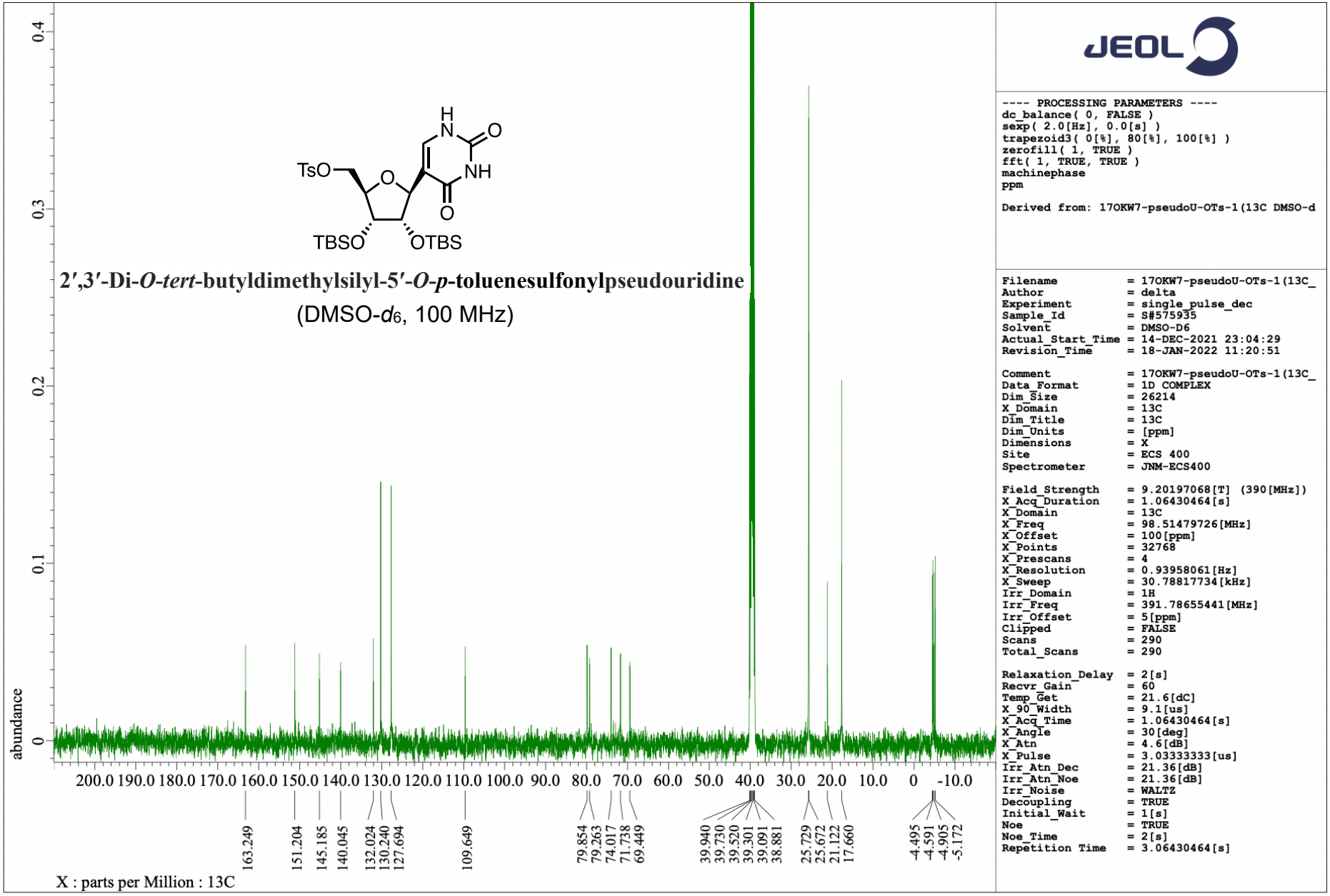
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = 13C
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain = 13C
X_Freq = 98.51479726[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.93958061[Hz]
X_Sweep = 30.78817734[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 344
Total_Scans = 344
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 21.6[dC]
X_90_Width = 9.1[us]
X_Acq_Time = 1.06430464[s]
X_Angle = 30[deg]
X_Atn = 4.6[dB]
X_Pulse = 3.03333333[us]
Irr_Atn_Dec = 21.36[dB]
Irr_Atn_Noe = 21.36[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.06430464[s]


TsO
TBSO
OTBS
H
N
O
NH
O
O
2′,3′-Di-O-tert-butyldimethylsilyl-5′-O-p-toluenesulfonylpseudouridine
(DMSO-d6, 400 MHz)
abundance
X : parts per Million : 1H
0.90
0.91
2.04
2.12
1.00
1.00
3.09
18.78
12.51
11.123
11.120
10.990
10.980
8.344
7.819
7.797
7.499
7.478
7.114
4.448
4.436
4.213
4.203
3.928
3.356
3.314
2.505
2.500
2.494
2.418
2.083
1.232
0.819
0.815
0.759
0.034
0.009
-0.004
-0.009
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW7-pseudoU-OTs-1(1H DMSO-d6
Filename = 17OKW7-pseudoU-OTs-1(1H_D
Author = delta
Experiment = single_pulse.ex2
Sample_Id = S#540619
Solvent = DMSO-D6
Actual_Start_Time = 14-DEC-2021 21:03:16
Revision_Time = 18-JAN-2022 11:17:52
Comment = 17OKW7-pseudoU-OTs-1(1H_D
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = 1H
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.2982153[T] (400[MHz])
X_Acq_Duration = 2.20725248[s]
X_Domain = 1H
X_Freq = 395.88430144[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45305193[Hz]
X_Sweep = 7.42280285[kHz]
Irr_Domain = 1H
Irr_Freq = 395.88430144[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 395.88430144[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 40
Temp_Get = 410.4[dC]
X_90_Width = 15.6[us]
X_Acq_Time = 2.20725248[s]
X_Angle = 45[deg]
X_Atn = 1.8[dB]
X_Pulse = 7.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.20725248[s]


TsO
O
H
N
O
NH
O
TBSO
OTBS
2′,3′-Di-O-tert-butyldimethylsilyl-5′-O-p-toluenesulfonylpseudouridine
(DMSO-d6, 100 MHz)
abundance
0
0.1
0.2
0.3
0.4
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
163.249
151.204
145.185
140.045
132.024
130.240
127.694
109.649
79.854
79.263
74.017
71.738
69.449
39.940
39.730
39.520
39.301
39.091
38.881
25.729
25.672
21.122
17.660
-4.495
-4.591
-4.905
-5.172
X : parts per Million : 13C
JEOL
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
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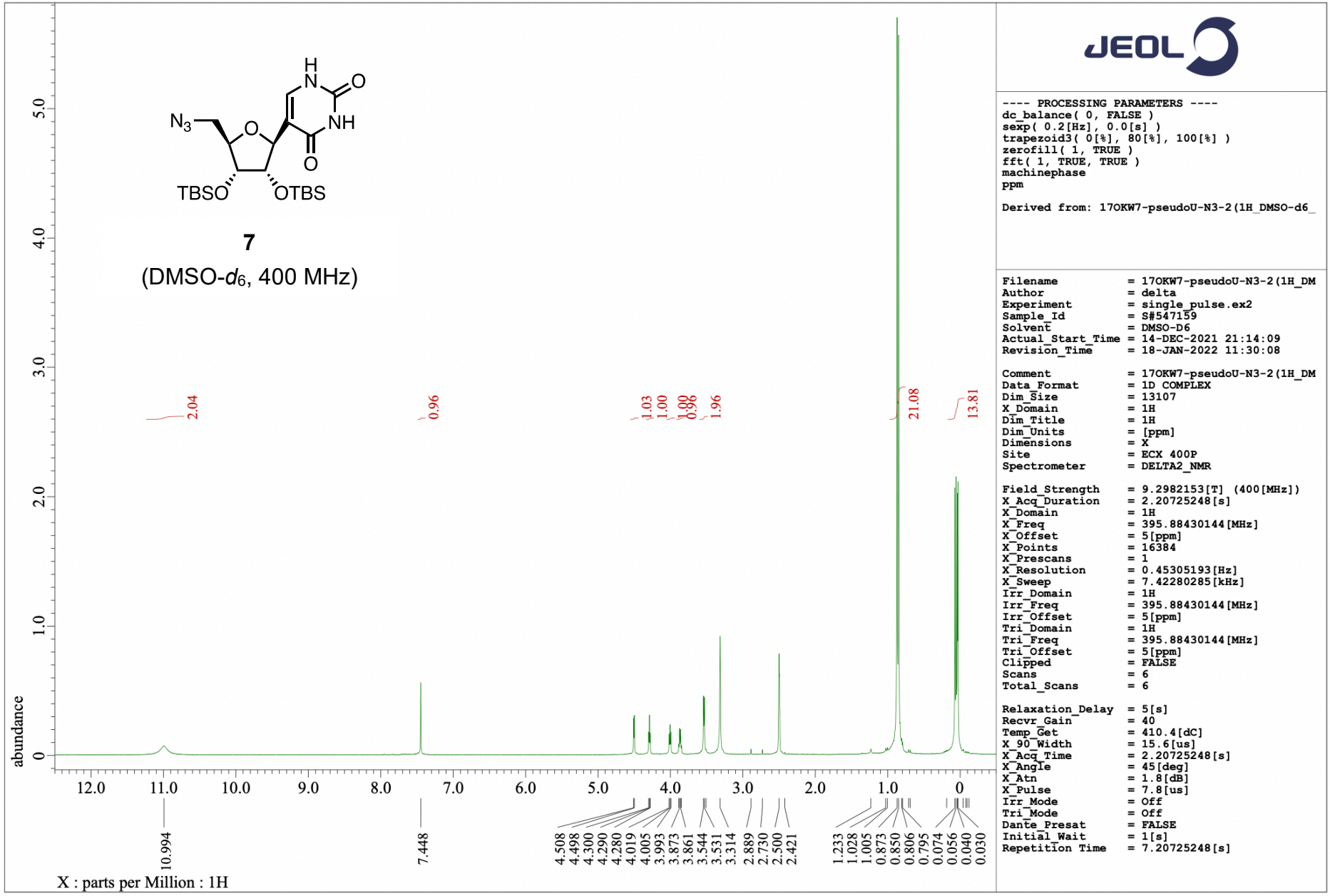
Filename = 17OKW7-pseudoU-OTs-1(13C_
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#575935
Solvent = DMSO-D6
Actual_Start_Time = 14-DEC-2021 23:04:29
Revision_Time = 18-JAN-2022 11:20:51
Comment = 17OKW7-pseudoU-OTs-1(13C_
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = 13C
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain = 13C
X_Freq = 98.51479726[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.93958061[Hz]
X_Sweep = 30.78817734[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 290
Total_Scans = 290
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 21.6[dC]
X_90_Width = 9.1[us]
X_Acq_Time = 1.06430464[s]
X_Angle = 30[deg]
X_Atn = 4.6[dB]
X_Pulse = 3.03333333[us]
Irr_Atn_Dec = 21.36[dB]
Irr_Atn_Noe = 21.36[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition Time = 3.06430464[s]


7
(DMSO-d_6, 400 MHz)
abundance
X : parts per Million : 1H
---- PROCESSING PARAMETERS ----
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sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW7-pseudoU-N3-2(1H_DMSO-d6_
Filename = 17OKW7-pseudoU-N3-2(1H_DM
Author = delta
Experiment = single_pulse.ex2
Sample_Id = S#547159
Solvent = DMSO-D6
Actual_Start_Time = 14-DEC-2021 21:14:09
Revision_Time = 18-JAN-2022 11:30:08
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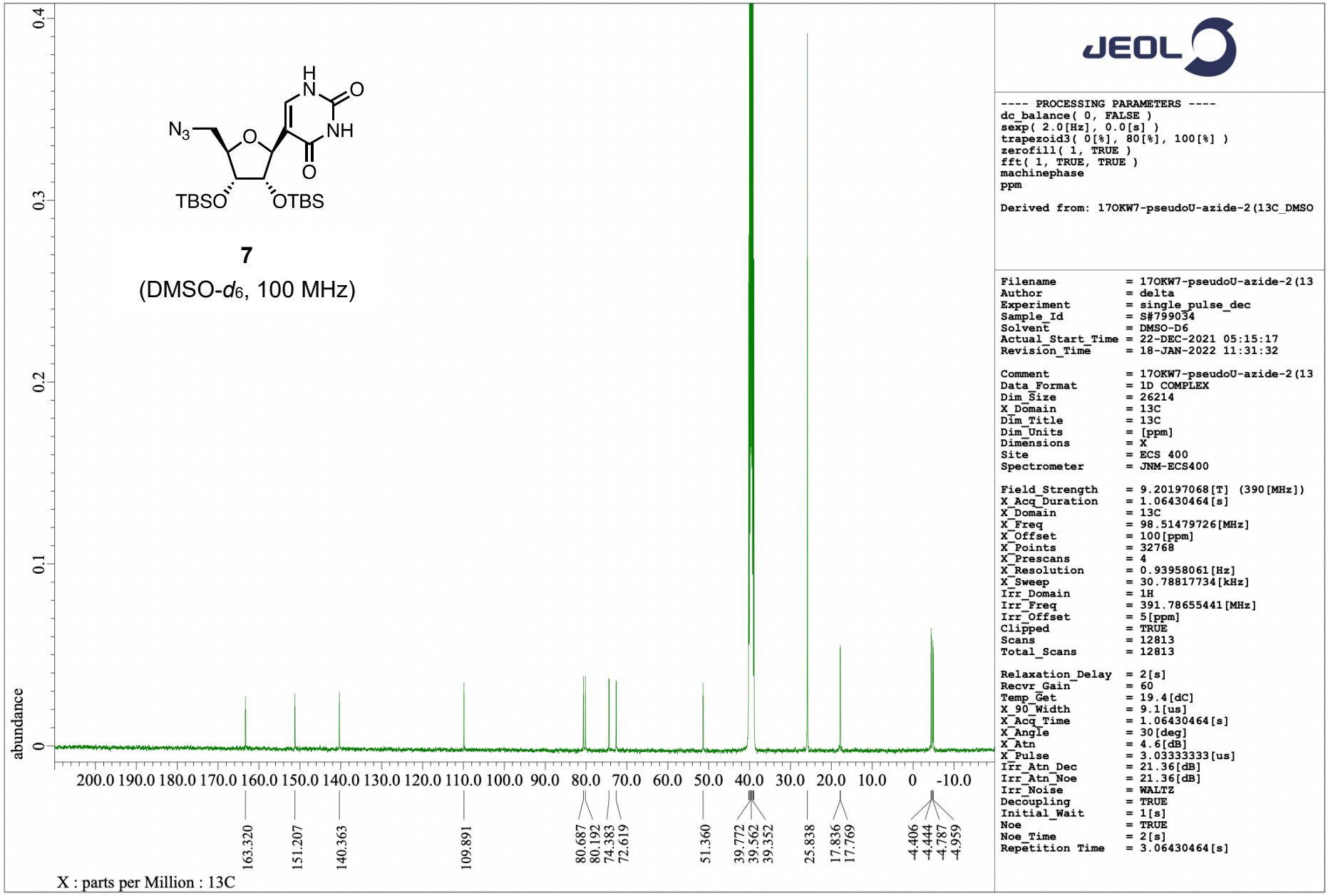
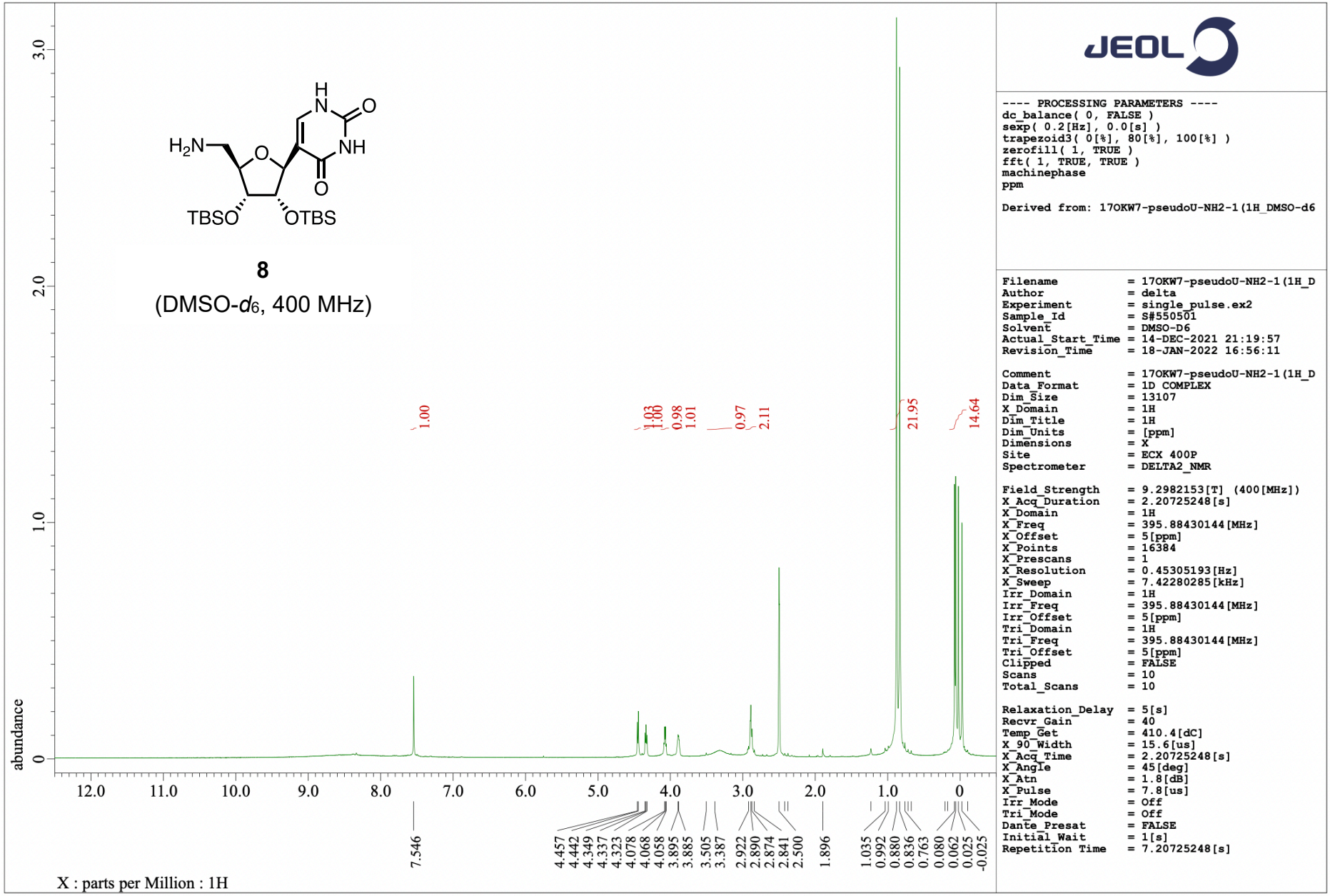
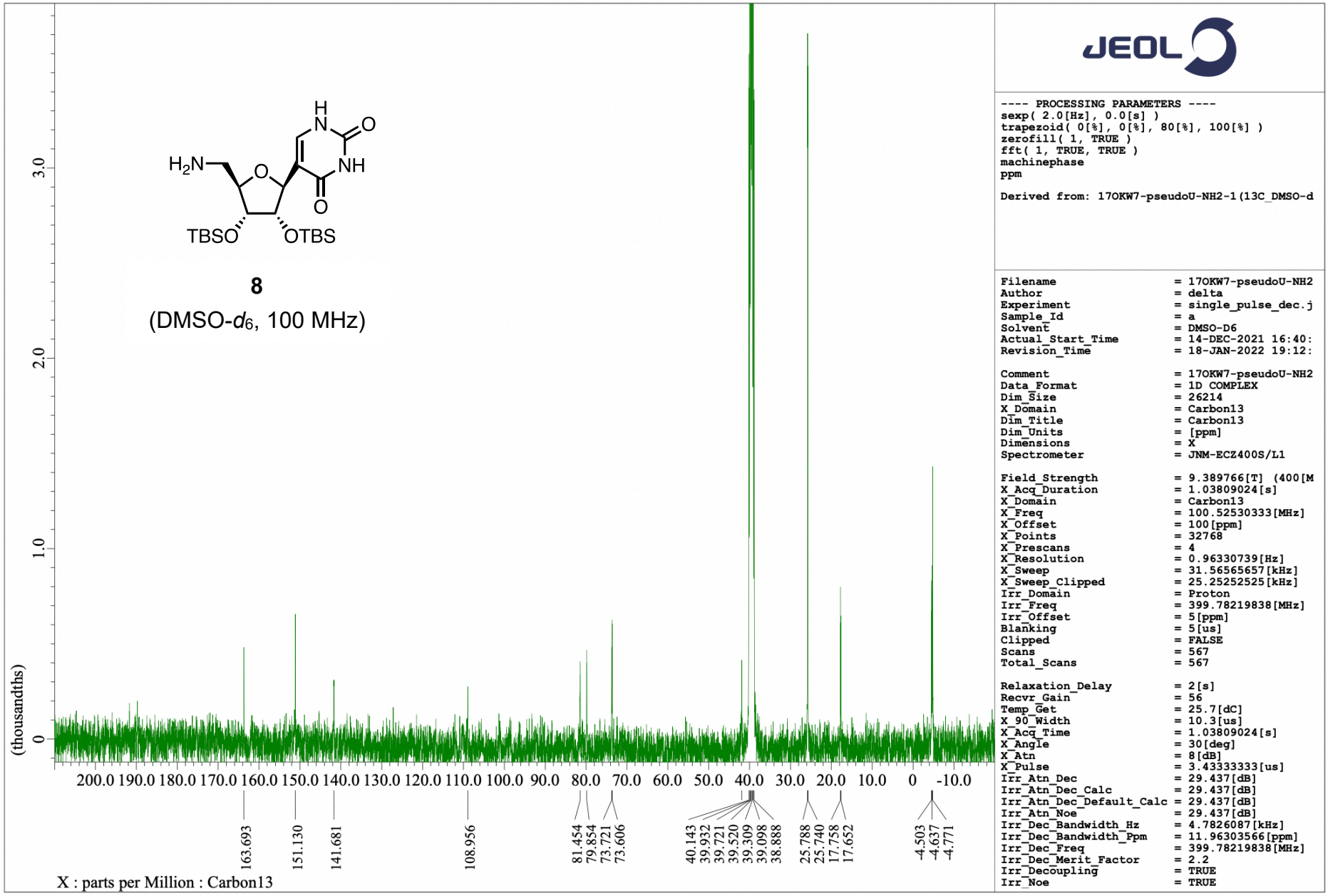
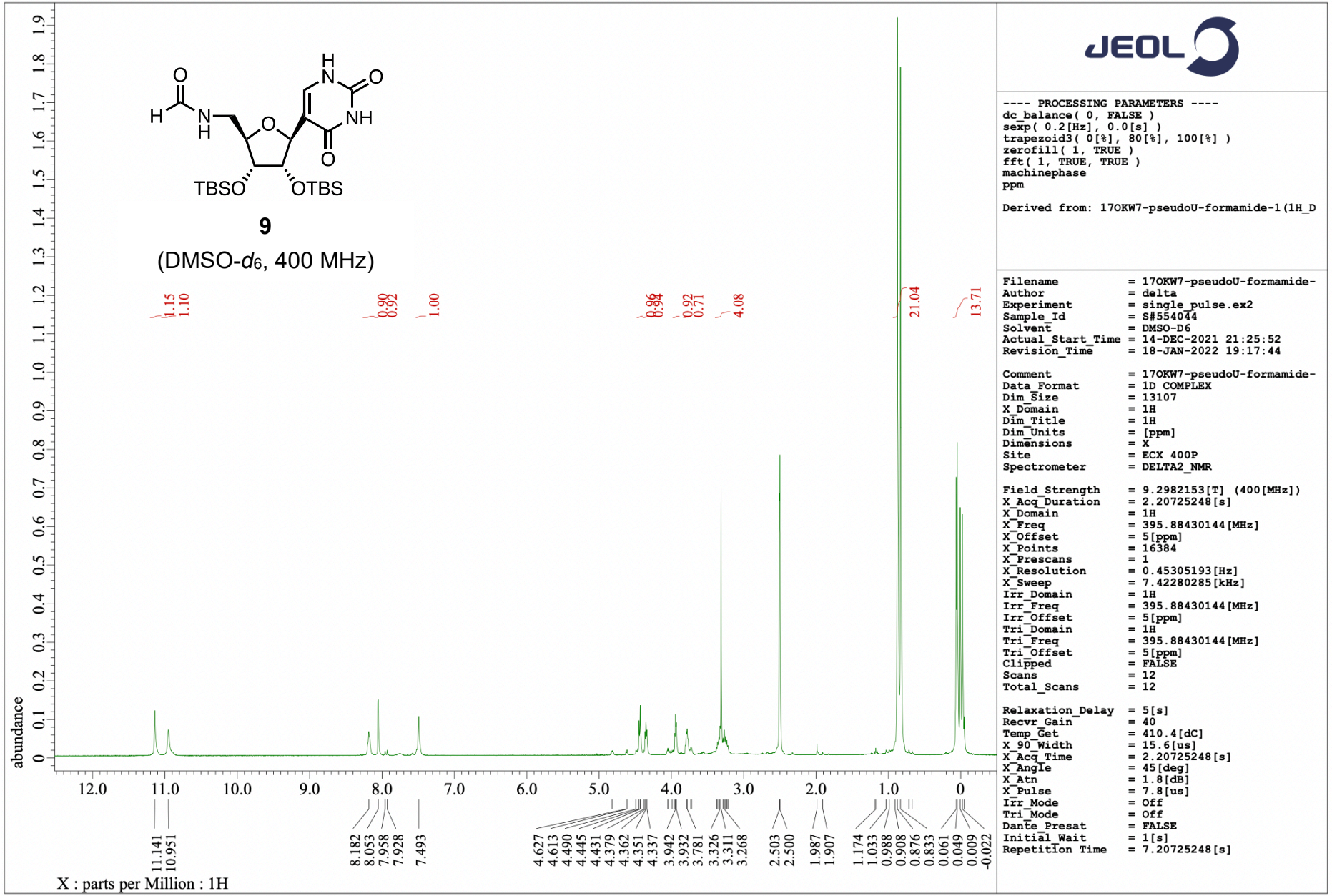
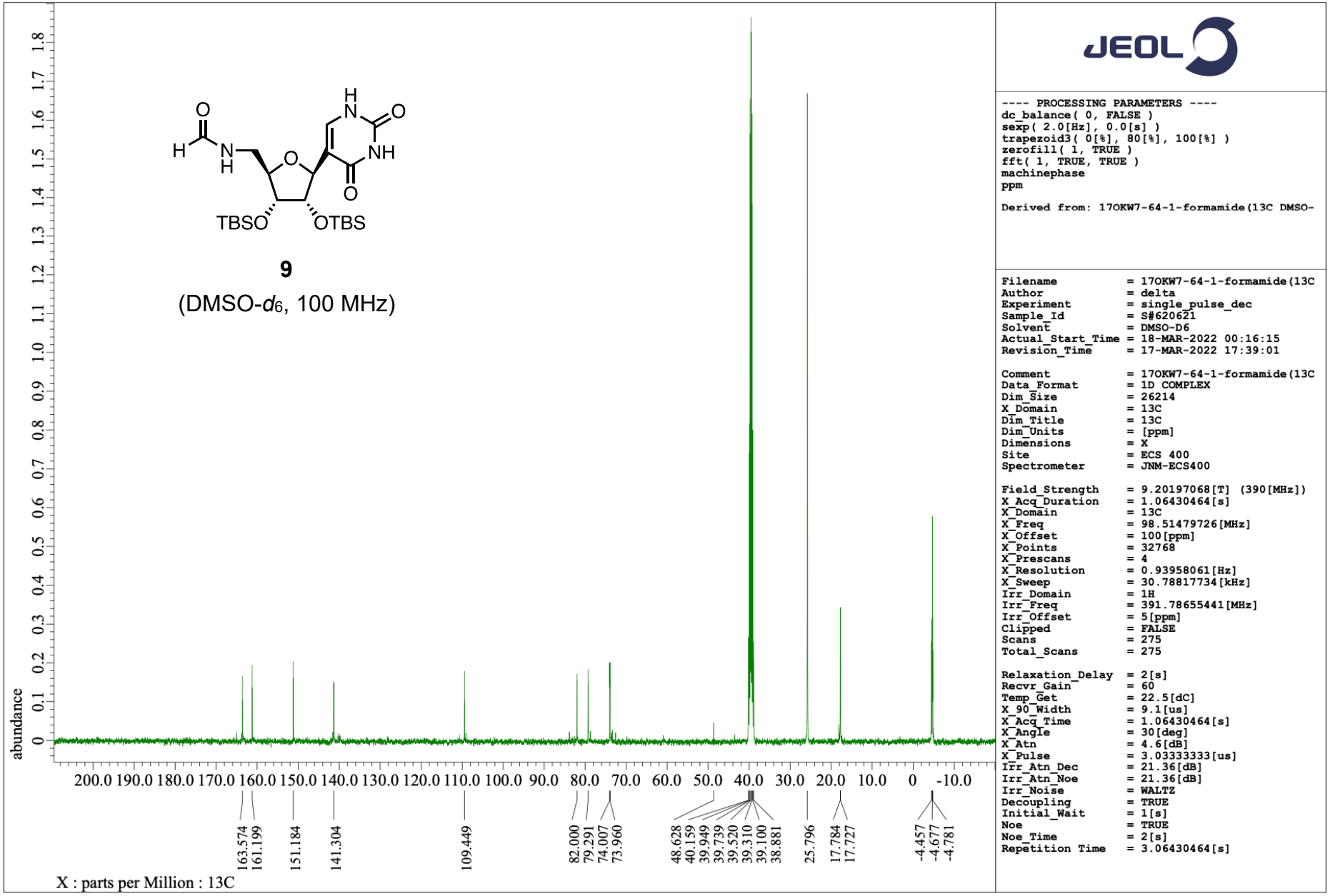
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = 1H
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.2982153[T] (400[MHz])
X_Acq_Duration = 2.20725248[s]
X_Domain = 1H
X_Freq = 395.88430144[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45305193[Hz]
X_Sweep = 7.42280285[kHz]
Irr_Domain = 1H
Irr_Freq = 395.88430144[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 395.88430144[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 6
Total_Scans = 6
Relaxation_Delay = 5[s]
Recvr_Gain = 40
Temp_Get = 410.4[dC]
X_90_Width = 15.6[us]
X_Acq_Time = 2.20725248[s]
X_Angle = 45[deg]
X_Atn = 1.8[dB]
X_Pulse = 7.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition Time = 7.20725248[s]

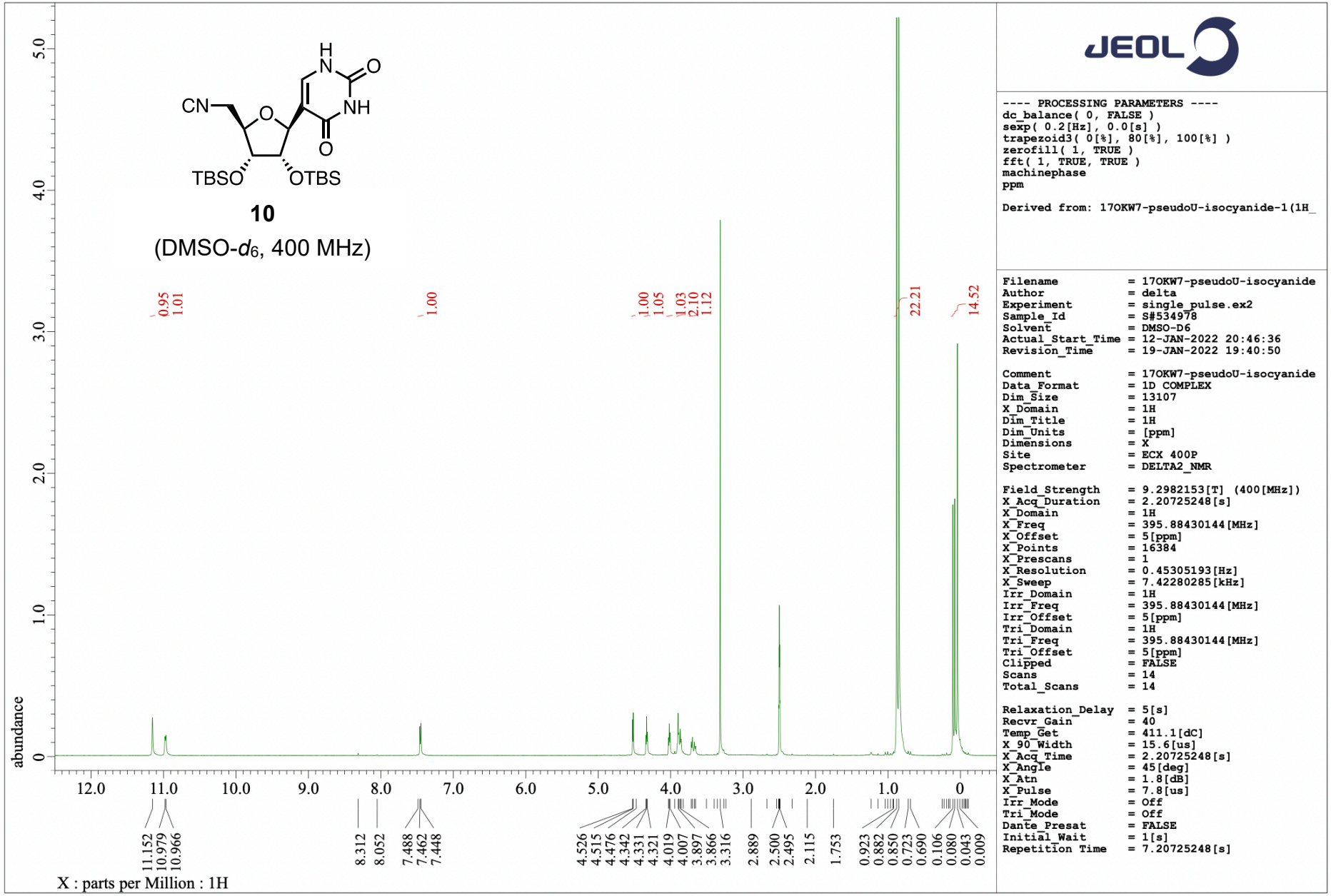

7
(DMSO-d6, 100 MHz)
abundance
X : parts per Million : 13C
163.320
151.207
140.363
109.891
80.687
80.192
74.383
72.619
51.360
39.772
39.562
39.352
25.838
17.836
17.769
-4.406
-4.444
-4.787
-4.959
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW7-pseudoU-azide-2(13C_DMSO
Filename = 17OKW7-pseudoU-azide-2(13
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#799034
Solvent = DMSO-D6
Actual_Start_Time = 22-DEC-2021 05:15:17
Revision_Time = 18-JAN-2022 11:31:32
Comment = 17OKW7-pseudoU-azide-2(13
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = 13C
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain = 13C
X_Freq = 98.51479726[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.93958061[Hz]
X_Sweep = 30.78817734[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 12813
Total_Scans = 12813
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 19.4[dC]
X_90_Width = 9.1[us]
X_Acq_Time = 1.06430464[s]
X_Angle = 30[deg]
X_Atn = 4.6[dB]
X_Pulse = 3.03333333[us]
Irr_Atn_Dec = 21.36[dB]
Irr_Atn_Noe = 21.36[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition Time = 3.06430464[s]

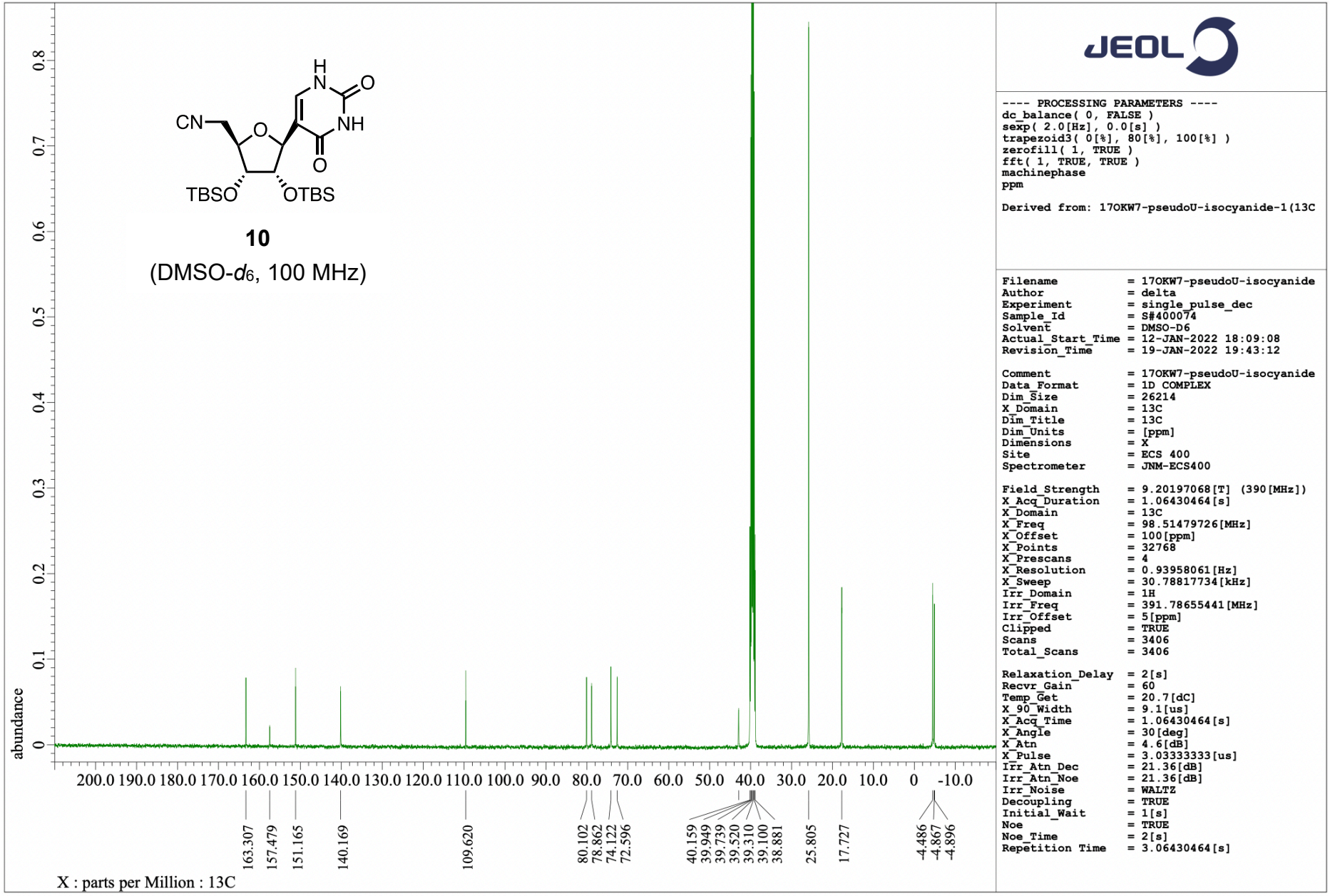

H2N
O
N
H
O
NH
O
TBSO
OTBS
8
(DMSO-d6, 400 MHz)
abundance
X : parts per Million : 1H
1.00
1.03
1.00
0.98
1.01
0.97
2.11
21.95
14.64
7.546
4.457
4.442
4.349
4.337
4.323
4.078
4.068
4.058
3.895
3.885
3.505
3.387
2.922
2.890
2.874
2.841
2.500
1.896
1.035
0.992
0.880
0.836
0.763
0.080
0.062
0.025
-0.025
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW7-pseudoU-NH2-1(1H_DMSO-d6
Filename = 17OKW7-pseudoU-NH2-1(1H_D
Author = delta
Experiment = single_pulse.ex2
Sample_Id = S#550501
Solvent = DMSO-D6
Actual_Start_Time = 14-DEC-2021 21:19:57
Revision_Time = 18-JAN-2022 16:56:11
Comment = 17OKW7-pseudoU-NH2-1(1H_D
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = 1H
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.2982153[T] (400[MHz])
X_Acq_Duration = 2.20725248[s]
X_Domain = 1H
X_Freq = 395.88430144[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45305193[Hz]
X_Sweep = 7.42280285[kHz]
Irr_Domain = 1H
Irr_Freq = 395.88430144[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 395.88430144[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 10
Total_Scans = 10
Relaxation_Delay = 5[s]
Recvr_Gain = 40
Temp_Get = 410.4[dC]
X_90_Width = 15.6[us]
X_Acq_Time = 2.20725248[s]
X_Angle = 45[deg]
X_Atn = 1.8[dB]
X_Pulse = 7.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition Time = 7.20725248[s]

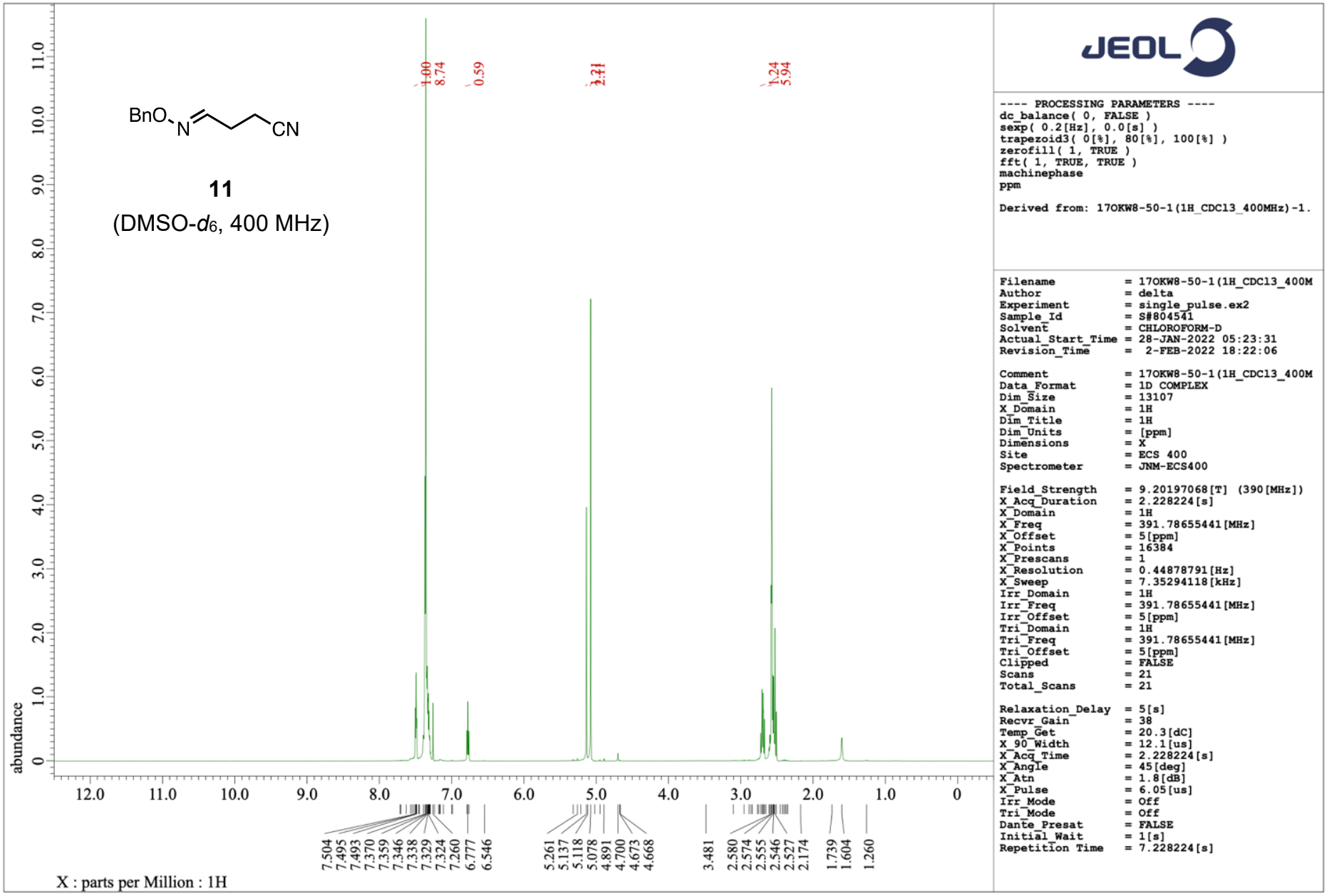

8
(DMSO-d_6, 100 MHz)
(thousandths)
X : parts per Million : Carbon13
163.693
151.130
141.681
108.956
81.454
79.854
73.721
73.606
40.143
39.932
39.721
39.520
39.309
39.098
38.888
25.788
25.740
17.758
17.652
-4.503
-4.637
-4.771
---- PROCESSING PARAMETERS ----
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW7-pseudoU-NH2-1(13C_DMSO-d
Filename = 17OKW7-pseudoU-NH2
Author = delta
Experiment = single_pulse_dec.j
Sample_Id = a
Solvent = DMSO-D6
Actual_Start_Time = 14-DEC-2021 16:40:
Revision_Time = 18-JAN-2022 19:12:
Comment = 17OKW7-pseudoU-NH2
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon13
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[M
X_Acq_Duration = 1.03809024[s]
X_Domain = Carbon13
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Blanking = 5[us]
Clipped = FALSE
Scans = 567
Total_Scans = 567
Relaxation_Delay = 2[s]
Recvr_Gain = 56
Temp_Get = 25.7[dC]
X_90_Width = 10.3[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 8[dB]
X_Pulse = 3.43333333[us]
Irr_Atn_Dec = 29.437[dB]
Irr_Atn_Dec_Calc = 29.437[dB]
Irr_Atn_Dec_Default_Calc = 29.437[dB]
Irr_Atn_Noe = 29.437[dB]
Irr_Dec_Bandwidth_Hz = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm = 11.96303566[ppm]
Irr_Dec_Freq = 399.78219838[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noe = TRUE

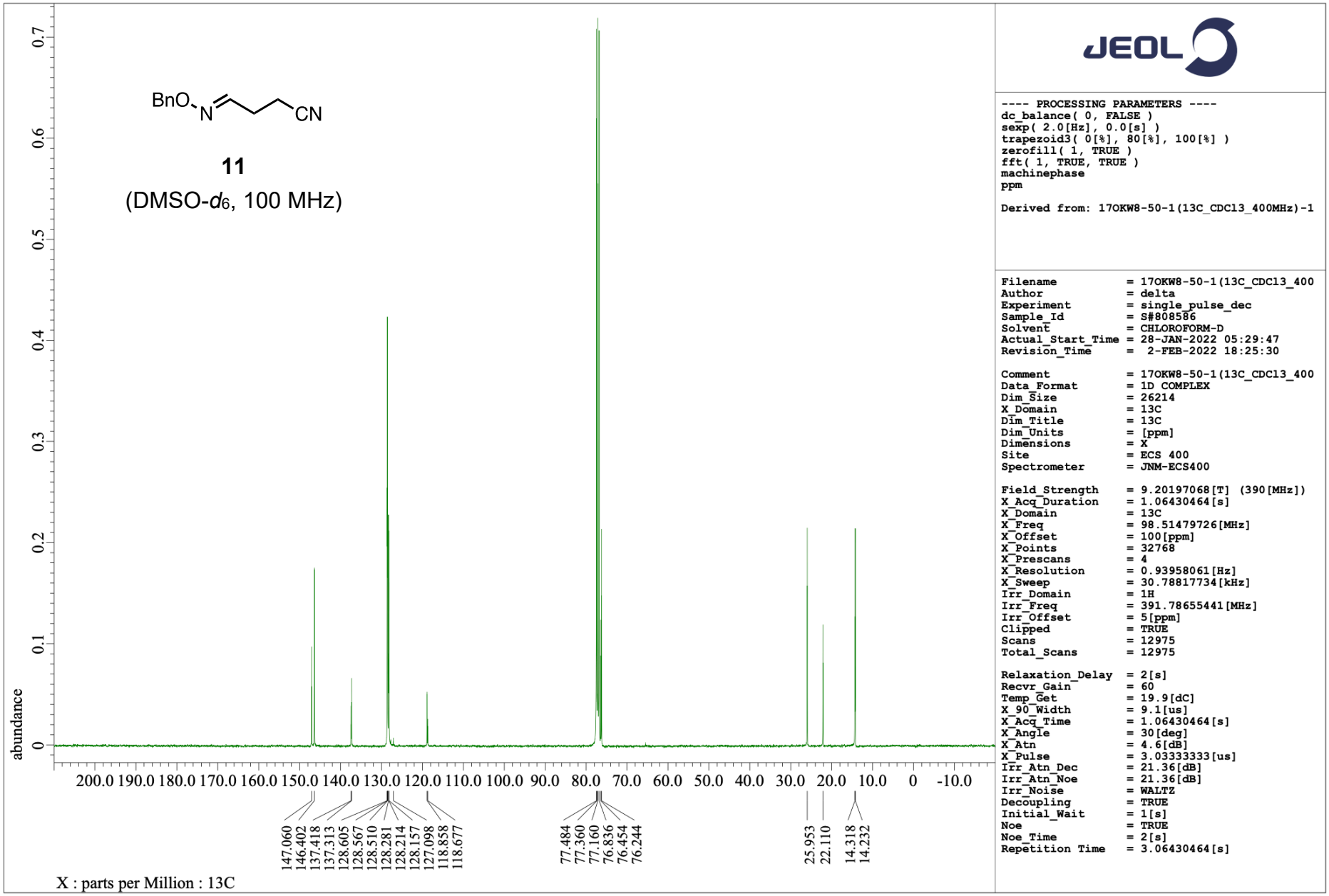

TBSO
OTBS
9
(DMSO-d6, 400 MHz)
abundance
X : parts per Million : 1H
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW7-pseudoU-formamide-1(1H_D
Filename = 17OKW7-pseudoU-formamide-
Author = delta
Experiment = single_pulse.ex2
Sample_Id = S#554044
Solvent = DMSO-D6
Actual_Start_Time = 14-DEC-2021 21:25:52
Revision_Time = 18-JAN-2022 19:17:44
Comment = 17OKW7-pseudoU-formamide-
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = 1H
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.2982153[T] (400[MHz])
X_Acq_Duration = 2.20725248[s]
X_Domain = 1H
X_Freq = 395.88430144[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45305193[Hz]
X_Sweep = 7.42280285[kHz]
Irr_Domain = 1H
Irr_Freq = 395.88430144[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 395.88430144[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 12
Total_Scans = 12
Relaxation_Delay = 5[s]
Recvr_Gain = 40
Temp_Get = 410.4[dC]
X_90_Width = 15.6[us]
X_Acq_Time = 2.20725248[s]
X_Angle = 45[deg]
X_Atn = 1.8[dB]
X_Pulse = 7.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition Time = 7.20725248[s]

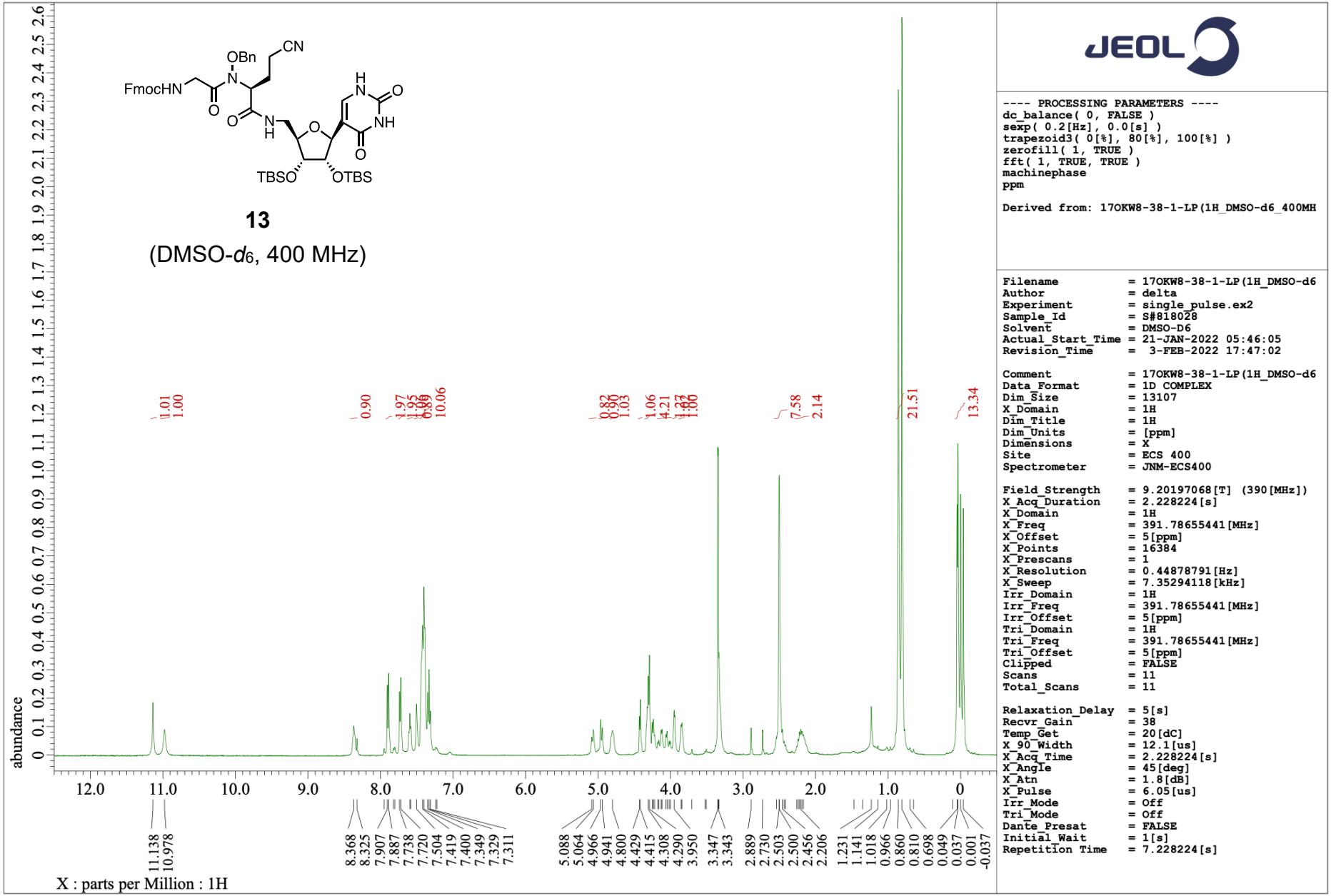

TBSO
OTBS
9
(DMSO-d6, 100 MHz)
abundance
X : parts per Million : 13C
163.574
161.199
151.184
141.304
109.449
82.000
79.291
74.007
73.960
48.628
40.159
39.949
39.739
39.520
39.310
39.100
38.881
25.796
17.784
17.727
-4.457
-4.677
-4.781
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW7-64-1-formamide(13C DMSO-
Filename = 17OKW7-64-1-formamide(13C
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#620621
Solvent = DMSO-D6
Actual_Start_Time = 18-MAR-2022 00:16:15
Revision_Time = 17-MAR-2022 17:39:01
Comment = 17OKW7-64-1-formamide(13C
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = 13C
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain = 13C
X_Freq = 98.51479726[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.93958061[Hz]
X_Sweep = 30.78817734[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 275
Total_Scans = 275
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 22.5[dC]
X_90_Width = 9.1[us]
X_Acq_Time = 1.06430464[s]
X_Angle = 30[deg]
X_Atn = 4.6[dB]
X_Pulse = 3.03333333[us]
Irr_Atn_Dec = 21.36[dB]
Irr_Atn_Noe = 21.36[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition Time = 3.06430464[s]

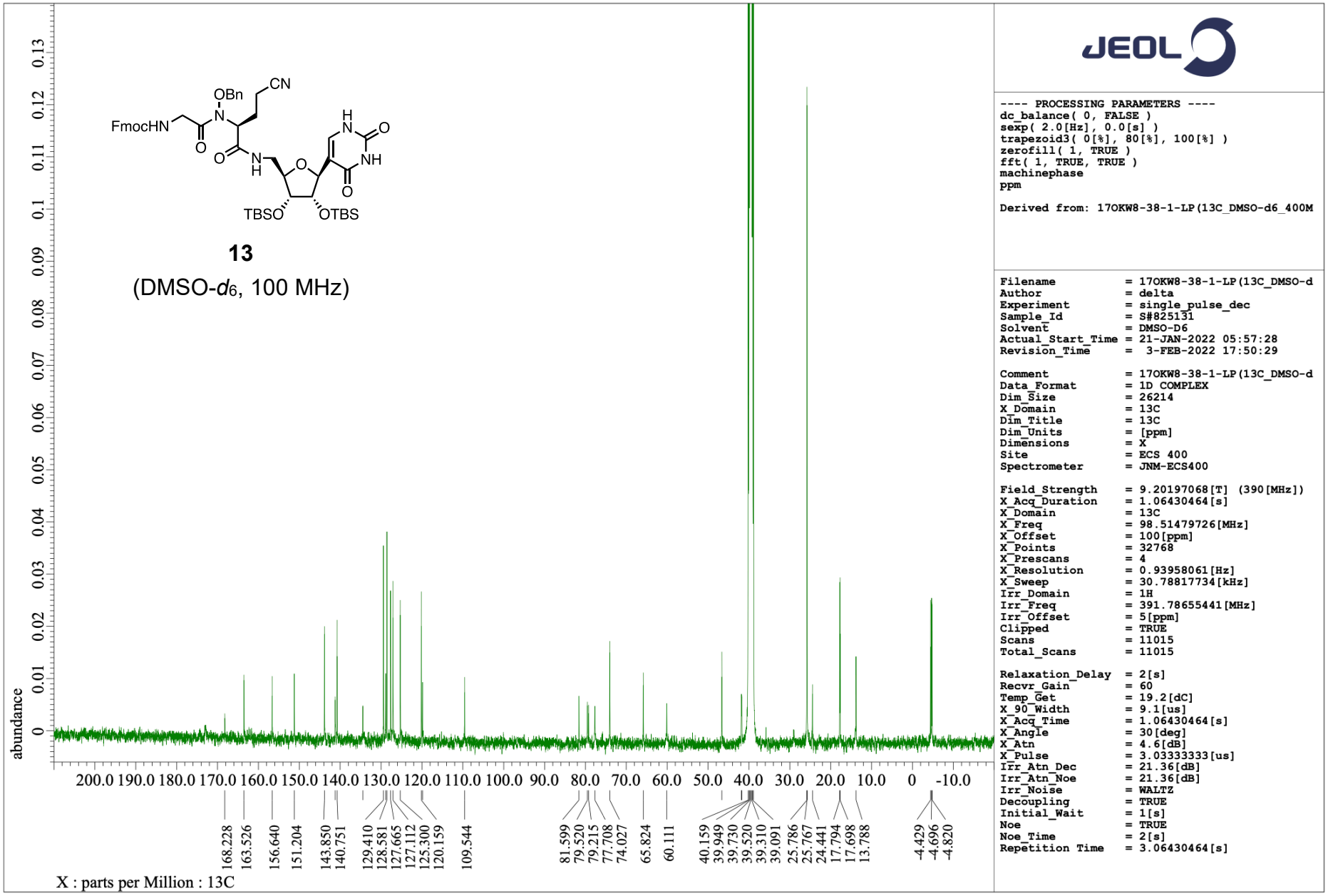

10
(DMSO-d_6, 400 MHz)
abundance
X : parts per Million : 1H
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW7-pseudoU-isocyanide-1(1H_
Filename = 17OKW7-pseudoU-isocyanide
Author = delta
Experiment = single_pulse.ex2
Sample_Id = S#534978
Solvent = DMSO-D6
Actual_Start_Time = 12-JAN-2022 20:46:36
Revision_Time = 19-JAN-2022 19:40:50
Comment = 17OKW7-pseudoU-isocyanide
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = 1H
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.2982153[T] (400[MHz])
X_Acq_Duration = 2.20725248[s]
X_Domain = 1H
X_Freq = 395.88430144[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45305193[Hz]
X_Sweep = 7.42280285[kHz]
Irr_Domain = 1H
Irr_Freq = 395.88430144[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 395.88430144[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 14
Total_Scans = 14
Relaxation_Delay = 5[s]
Recvr_Gain = 40
Temp_Get = 411.1[dC]
X_90_Width = 15.6[us]
X_Acq_Time = 2.20725248[s]
X_Angle = 45[deg]
X_Atn = 1.8[dB]
X_Pulse = 7.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition Time = 7.20725248[s]

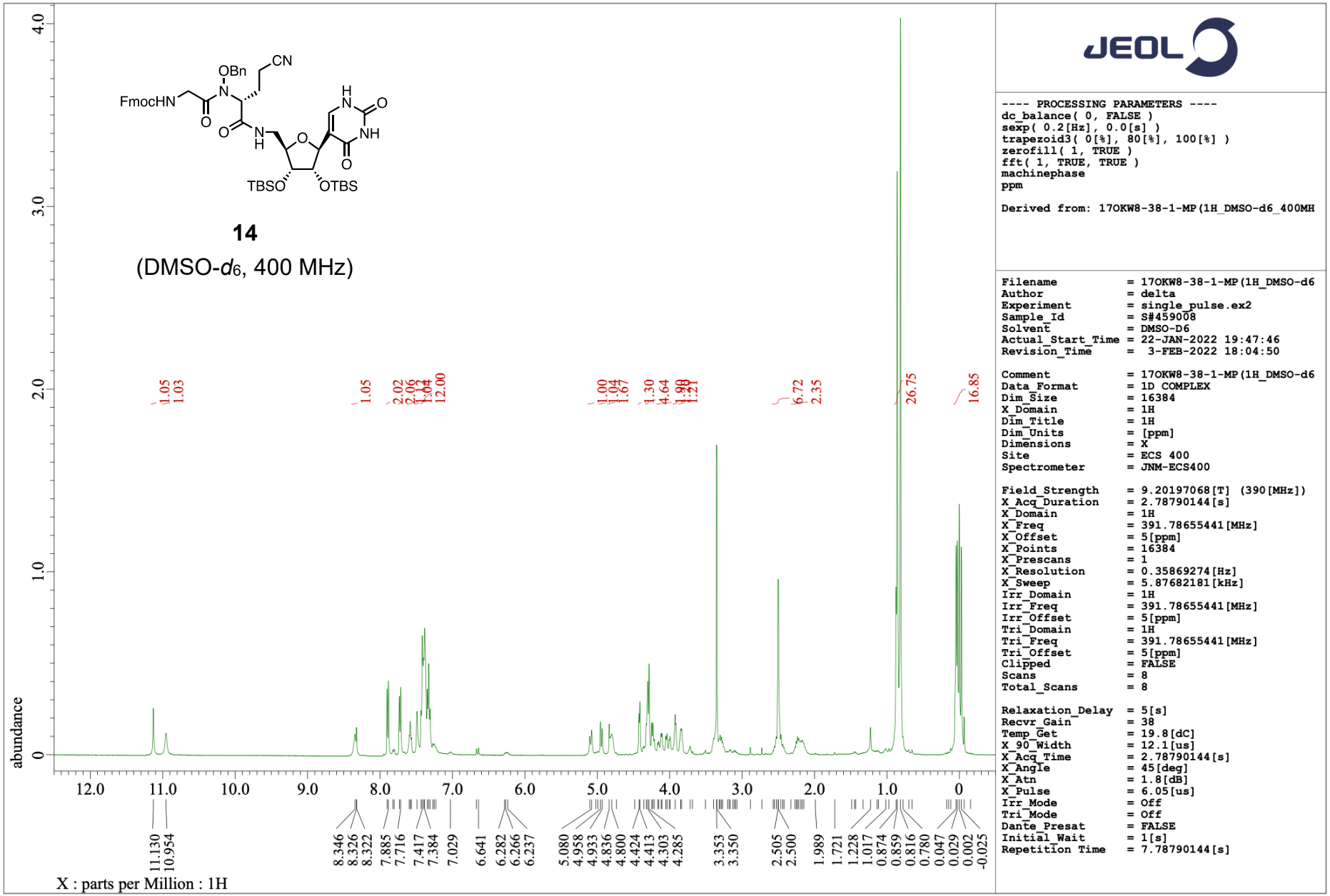

CN
O
H
N
O
NH
O
TBSO
OTBS
10
(DMSO-d6, 100 MHz)
abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
163.307
157.479
151.165
140.169
109.620
80.102
78.862
74.122
72.596
40.159
39.949
39.739
39.520
39.310
39.100
38.881
25.805
17.727
-4.486
-4.867
-4.896
X : parts per Million : 13C
JEOL
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW7-pseudoU-isocyanide-1(13C
Filename = 17OKW7-pseudoU-isocyanide
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#400074
Solvent = DMSO-D6
Actual_Start_Time = 12-JAN-2022 18:09:08
Revision_Time = 19-JAN-2022 19:43:12
Comment = 17OKW7-pseudoU-isocyanide
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = 13C
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain = 13C
X_Freq = 98.51479726[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.93958061[Hz]
X_Sweep = 30.78817734[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 3406
Total_Scans = 3406
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 20.7[dC]
X_90_Width = 9.1[us]
X_Acq_Time = 1.06430464[s]
X_Angle = 30[deg]
X_Atn = 4.6[dB]
X_Pulse = 3.03333333[us]
Irr_Atn_Dec = 21.36[dB]
Irr_Atn_Noe = 21.36[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition Time = 3.06430464[s]

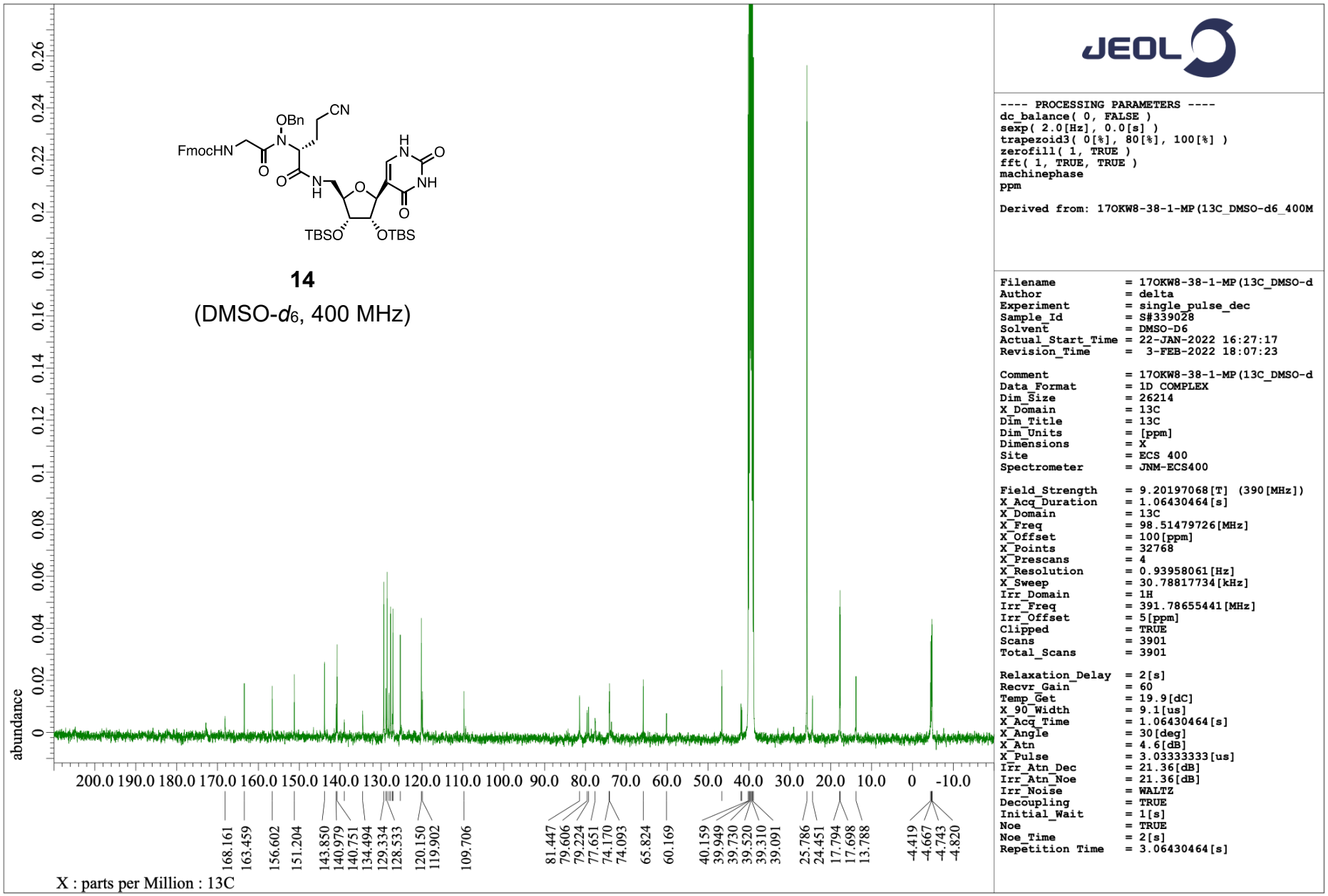

BnO
N
CN
11
(DMSO-d6, 400 MHz)
1.00
8.74
0.59
3.41
1.24
5.94
abundance
X : parts per Million : 1H
7.504
7.495
7.493
7.370
7.359
7.346
7.338
7.329
7.324
7.260
6.777
6.546
5.261
5.137
5.118
5.078
4.891
4.700
4.673
4.668
3.481
2.580
2.574
2.555
2.546
2.527
2.174
1.739
1.604
1.260
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW8-50-1(1H_CDCl3_400MHz)-1.
Filename = 17OKW8-50-1(1H_CDCl3_400M
Author = delta
Experiment = single_pulse.ex2
Sample_Id = S#804541
Solvent = CHLOROFORM-D
Actual_Start_Time = 28-JAN-2022 05:23:31
Revision_Time = 2-FEB-2022 18:22:06
Comment = 17OKW8-50-1(1H_CDCl3_400M
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = 1H
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 2.228224[s]
X_Domain = 1H
X_Freq = 391.78655441[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.44878791[Hz]
X_Sweep = 7.35294118[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 391.78655441[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 21
Total_Scans = 21
Relaxation_Delay = 5[s]
Recvr_Gain = 38
Temp_Get = 20.3[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.228224[s]
X_Angle = 45[deg]
X_Atn = 1.8[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition Time = 7.228224[s]

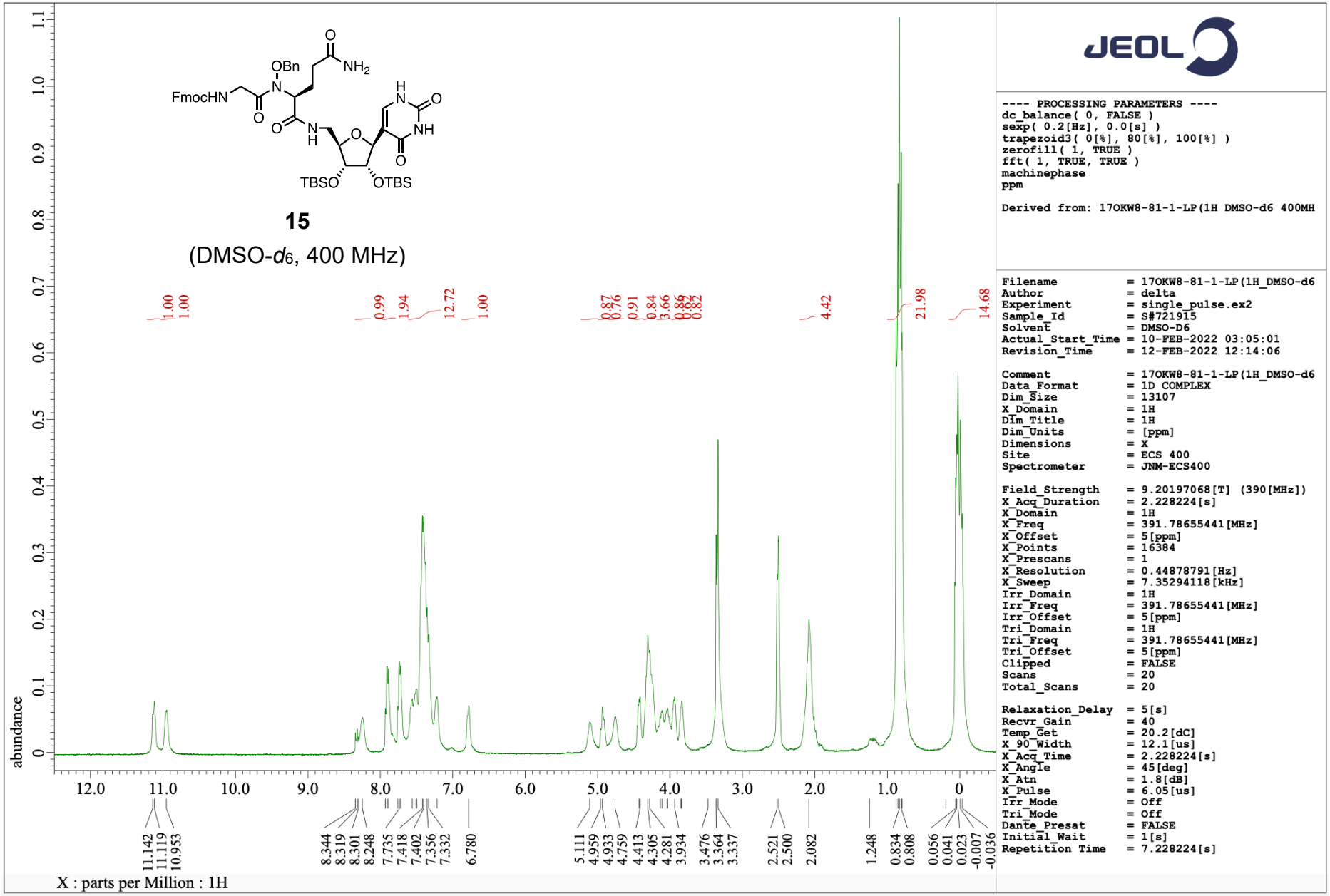

BnO
N
CN
11
(DMSO-d6, 100 MHz)
abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
147.060
146.402
137.418
137.313
128.605
128.567
128.510
128.281
128.214
128.157
127.098
118.858
118.677
77.484
77.360
77.160
76.836
76.454
76.244
25.953
22.110
14.318
14.232
X : parts per Million : 13C
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW8-50-1(13C_CDCl3_400MHz)-1
Filename = 17OKW8-50-1(13C_CDCl3_400
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#808586
Solvent = CHLOROFORM-D
Actual_Start_Time = 28-JAN-2022 05:29:47
Revision_Time = 2-FEB-2022 18:25:30
Comment = 17OKW8-50-1(13C_CDCl3_400
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = 13C
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain = 13C
X_Freq = 98.51479726[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.93958061[Hz]
X_Sweep = 30.78817734[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 12975
Total_Scans = 12975
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 19.9[dC]
X_90_Width = 9.1[us]
X_Acq_Time = 1.06430464[s]
X_Angle = 30[deg]
X_Atn = 4.6[dB]
X_Pulse = 3.03333333[us]
Irr_Atn_Dec = 21.36[dB]
Irr_Atn_Noe = 21.36[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition Time = 3.06430464[s]


FmocHN
OBn
CN
TBSO
OTBS
13
(DMSO-d6, 400 MHz)
abundance
X : parts per Million : 1H
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW8-38-1-LP(1H_DMSO-d6_400MH
Filename = 17OKW8-38-1-LP(1H_DMSO-d6
Author = delta
Experiment = single_pulse.ex2
Sample_Id = S#818028
Solvent = DMSO-D6
Actual_Start_Time = 21-JAN-2022 05:46:05
Revision_Time = 3-FEB-2022 17:47:02
Comment = 17OKW8-38-1-LP(1H_DMSO-d6
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = 1H
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 2.228224[s]
X_Domain = 1H
X_Freq = 391.78655441[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.44878791[Hz]
X_Sweep = 7.35294118[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 391.78655441[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 11
Total_Scans = 11
Relaxation_Delay = 5[s]
Recvr_Gain = 38
Temp_Get = 20[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.228224[s]
X_Angle = 45[deg]
X_Atn = 1.8[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition Time = 7.228224[s]


FmocHN
OBn
CN
TBSO
OTBS
13
(DMSO-d6, 100 MHz)
abundance
X : parts per Million : 13C
168.228
163.526
156.640
151.204
143.850
140.751
129.410
128.581
127.665
127.112
125.300
120.159
109.544
81.599
79.520
79.215
77.708
74.027
65.824
60.111
40.159
39.949
39.730
39.520
39.310
39.091
25.786
25.767
24.441
17.794
17.698
13.788
-4.429
-4.696
-4.820
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW8-38-1-LP(13C_DMSO-d6_400M
Filename = 17OKW8-38-1-LP(13C_DMSO-d
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#825131
Solvent = DMSO-D6
Actual_Start_Time = 21-JAN-2022 05:57:28
Revision_Time = 3-FEB-2022 17:50:29
Comment = 17OKW8-38-1-LP(13C_DMSO-d
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = 13C
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain = 13C
X_Freq = 98.51479726[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.93958061[Hz]
X_Sweep = 30.78817734[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 11015
Total_Scans = 11015
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 19.2[dC]
X_90_Width = 9.1[us]
X_Acq_Time = 1.06430464[s]
X_Angle = 30[deg]
X_Atn = 4.6[dB]
X_Pulse = 3.03333333[us]
Irr_Atn_Dec = 21.36[dB]
Irr_Atn_Noe = 21.36[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition Time = 3.06430464[s]

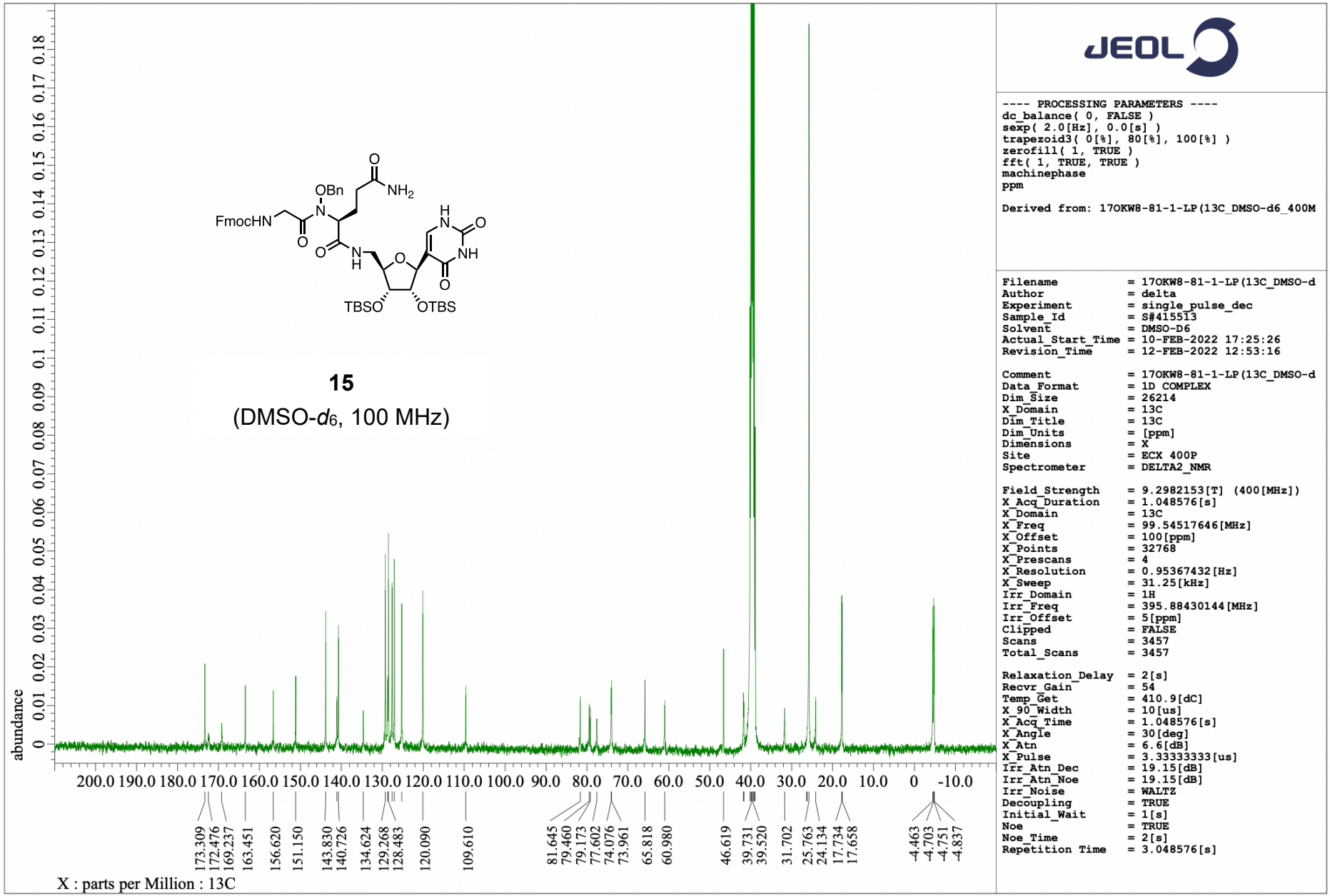

FmocHN
OBn
CN
TBSO
OTBS
14
(DMSO-d6, 400 MHz)
abundance
X : parts per Million : 1H
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW8-38-1-MP(1H_DMSO-d6_400MH
Filename = 17OKW8-38-1-MP(1H_DMSO-d6
Author = delta
Experiment = single_pulse.ex2
Sample_Id = S#459008
Solvent = DMSO-D6
Actual_Start_Time = 22-JAN-2022 19:47:46
Revision_Time = 3-FEB-2022 18:04:50
Comment = 17OKW8-38-1-MP(1H_DMSO-d6
Data_Format = 1D COMPLEX
Dim_Size = 16384
X_Domain = 1H
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 2.78790144[s]
X_Domain = 1H
X_Freq = 391.78655441[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.35869274[Hz]
X_Sweep = 5.87682181[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 391.78655441[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 38
Temp_Get = 19.8[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.78790144[s]
X_Angle = 45[deg]
X_Atn = 1.8[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition Time = 7.78790144[s]

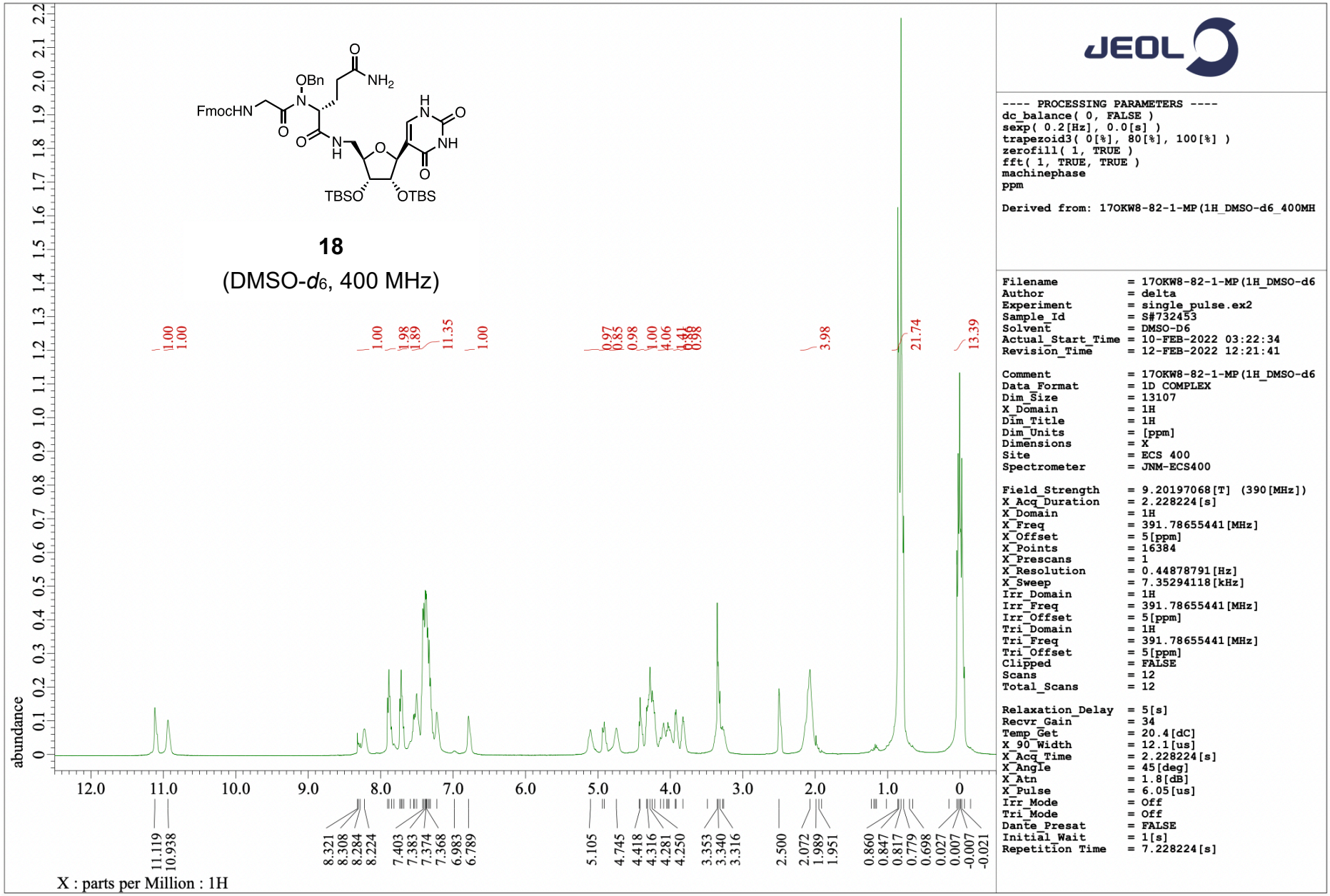

FmocHN
OBn
CN
TBSO
OTBS
14
(DMSO-d6, 400 MHz)
abundance
X : parts per Million : 13C
168.161
163.459
156.602
151.204
143.850
140.979
140.751
134.494
129.334
128.533
120.150
119.902
109.706
81.447
79.606
79.224
77.651
74.170
74.093
65.824
60.169
40.159
39.949
39.730
39.520
39.310
39.091
25.786
24.451
17.794
17.698
13.788
-4.419
-4.667
-4.743
-4.820
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW8-38-1-MP(13C_DMSO-d6_400M
Filename = 17OKW8-38-1-MP(13C_DMSO-d
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#339028
Solvent = DMSO-D6
Actual_Start_Time = 22-JAN-2022 16:27:17
Revision_Time = 3-FEB-2022 18:07:23
Comment = 17OKW8-38-1-MP(13C_DMSO-d
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = 13C
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain = 13C
X_Freq = 98.51479726[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.93958061[Hz]
X_Sweep = 30.78817734[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 3901
Total_Scans = 3901
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 19.9[dC]
X_90_Width = 9.1[us]
X_Acq_Time = 1.06430464[s]
X_Angle = 30[deg]
X_Atn = 4.6[dB]
X_Pulse = 3.03333333[us]
Irr_Atn_Dec = 21.36[dB]
Irr_Atn_Noe = 21.36[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition Time = 3.06430464[s]

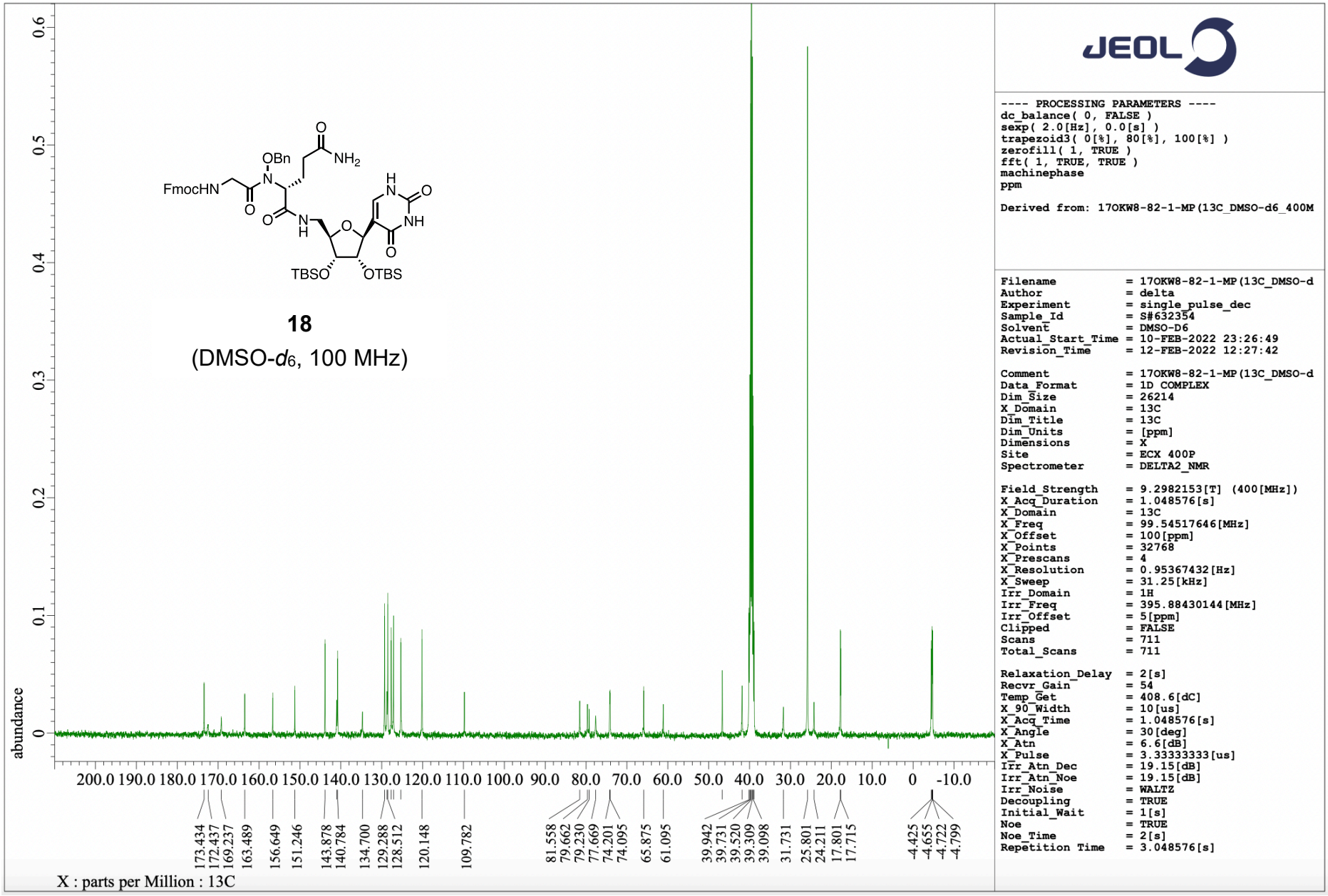


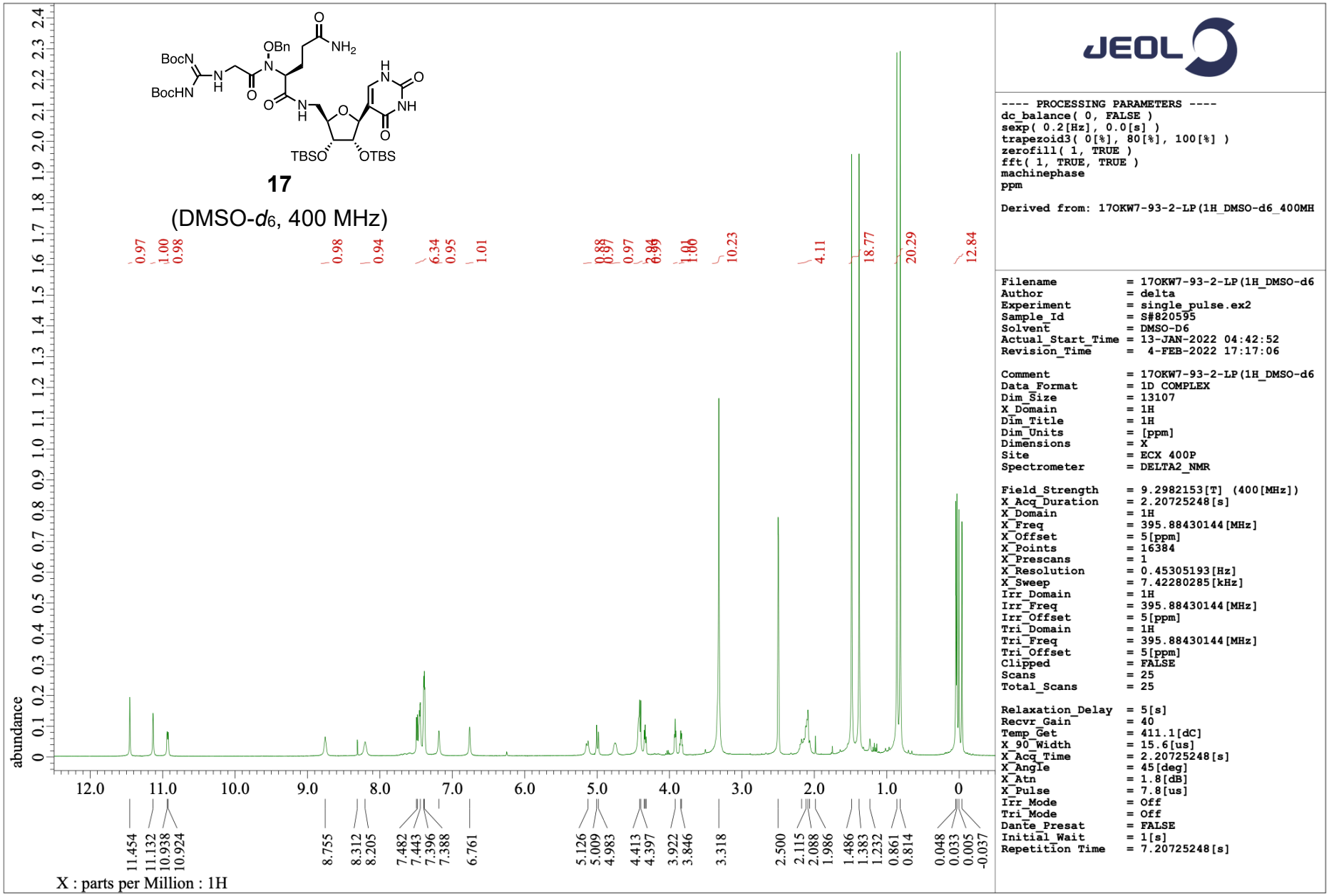

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: 17OKW8-81-1-LP(1H DMSO-d6 400MH
```

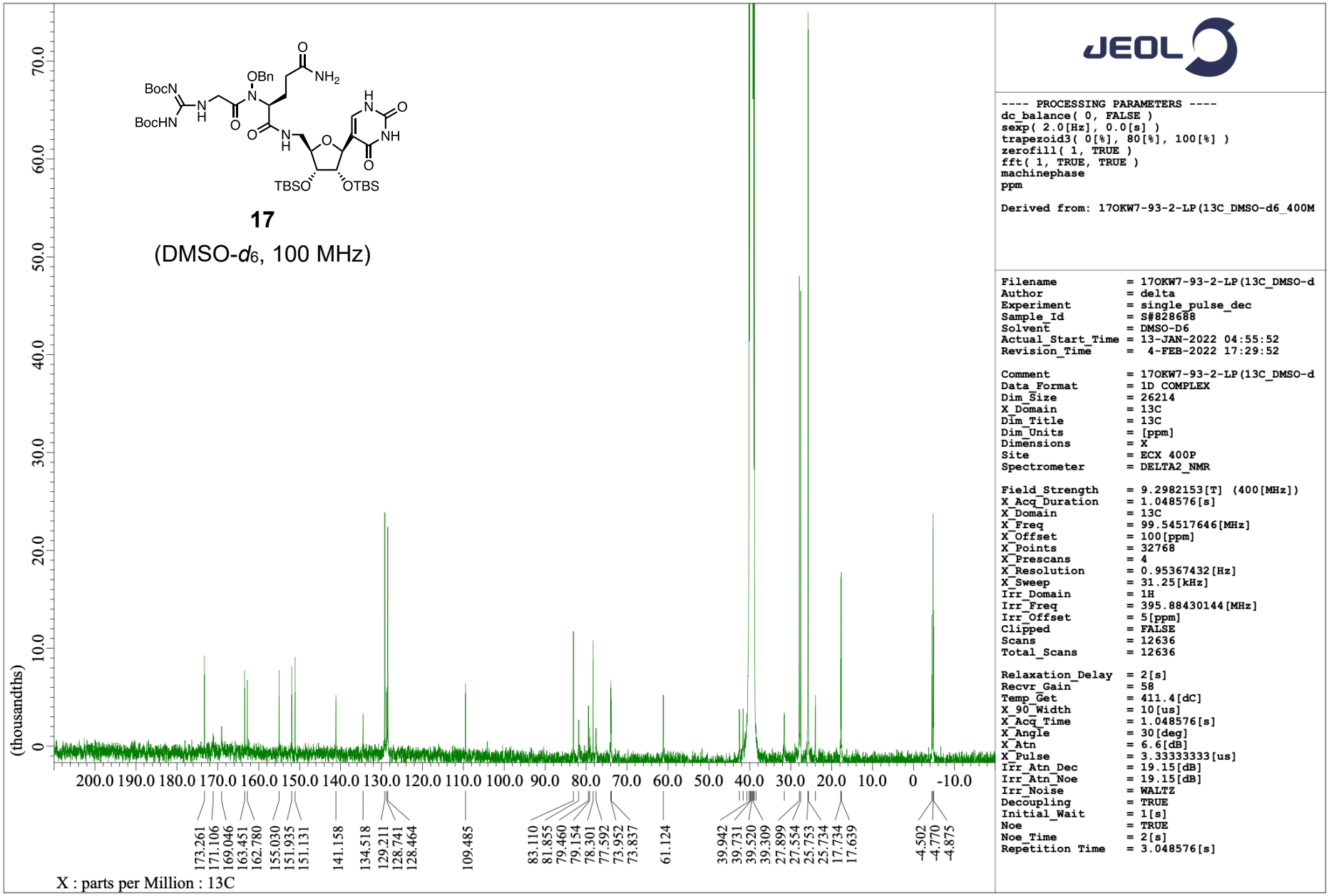
Filename	= 17OKW8-81-1-LP(1H_DMSO-d6
Author	= delta
Experiment	= single_pulse.ex2
Sample_Id	= S#721915
Solvent	= DMSO-D6
Actual_Start_Time	= 10-FEB-2022 03:05:01
Revision_Time	= 12-FEB-2022 12:14:06
Comment	= 17OKW8-81-1-LP(1H_DMSO-d6
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= 1H
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 2.228224[s]
X_Domain	= 1H
X_Freq	= 391.78655441[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.44878791[Hz]
X_Sweep	= 7.35294118[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 391.78655441[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 20
Total_Scans	= 20
Relaxation_Delay	= 5[s]
Recvr_Gain	= 40
Temp_Get	= 20.2[dC]
X_90_Width	= 12.1[us]
X_Acq_Time	= 2.228224[s]
X_Angle	= 45[deg]
X_Atn	= 1.8[dB]
X_Pulse	= 6.05[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition Time	= 7.228224[s]

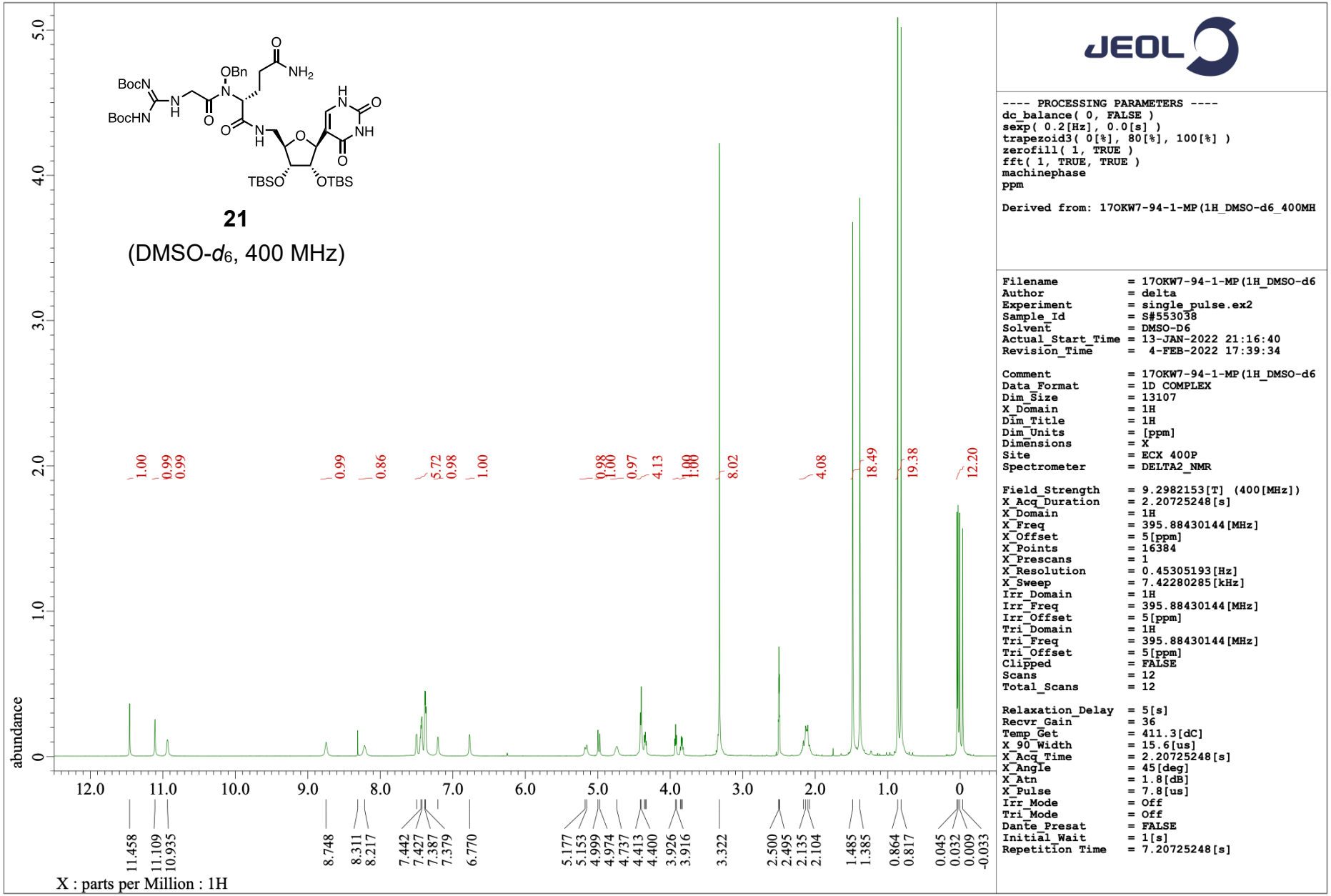

FmocHN
OBn
NH2
TBSO
OTBS
15
(DMSO-d6, 100 MHz)
abundance
X : parts per Million : 13C
173.309
172.476
169.237
163.451
156.620
151.150
143.830
140.726
134.624
129.268
128.483
120.090
109.610
81.645
79.460
79.173
77.602
74.076
73.961
65.818
60.980
46.619
39.731
39.520
31.702
25.763
24.134
17.734
17.658
-4.463
-4.703
-4.751
-4.837
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW8-81-1-LP(13C_DMSO-d6_400M
Filename = 17OKW8-81-1-LP(13C_DMSO-d
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#415513
Solvent = DMSO-D6
Actual_Start_Time = 10-FEB-2022 17:25:26
Revision_Time = 12-FEB-2022 12:53:16
Comment = 17OKW8-81-1-LP(13C_DMSO-d
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = 13C
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.2982153[T] (400[MHz])
X_Acq_Duration = 1.048576[s]
X_Domain = 13C
X_Freq = 99.54517646[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95367432[Hz]
X_Sweep = 31.25[kHz]
Irr_Domain = 1H
Irr_Freq = 395.88430144[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 3457
Total_Scans = 3457
Relaxation_Delay = 2[s]
Recvr_Gain = 54
Temp_Get = 410.9[dC]
X_90_Width = 10[us]
X_Acq_Time = 1.048576[s]
X_Angle = 30[deg]
X_Atn = 6.6[dB]
X_Pulse = 3.33333333[us]
Irr_Atn_Dec = 19.15[dB]
Irr_Atn_Noe = 19.15[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition Time = 3.048576[s]

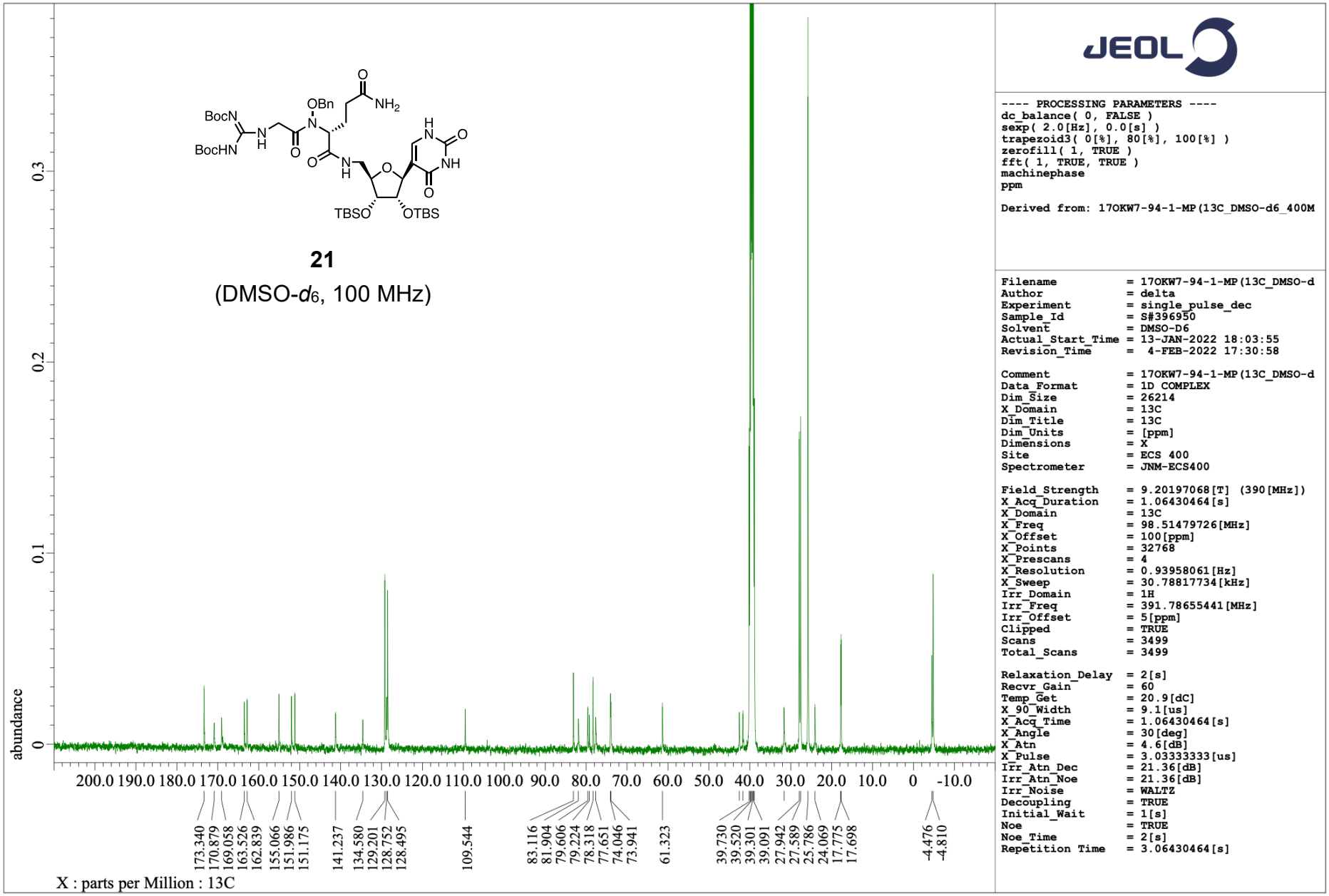

FmocHN
OBn
NH2
TBSO
OTBS
18
(DMSO-d6, 400 MHz)
abundance
X : parts per Million : 1H
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW8-82-1-MP(1H_DMSO-d6_400MH
Filename = 17OKW8-82-1-MP(1H_DMSO-d6
Author = delta
Experiment = single_pulse.ex2
Sample_Id = S#732453
Solvent = DMSO-D6
Actual_Start_Time = 10-FEB-2022 03:22:34
Revision_Time = 12-FEB-2022 12:21:41
Comment = 17OKW8-82-1-MP(1H_DMSO-d6
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = 1H
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 2.228224[s]
X_Domain = 1H
X_Freq = 391.78655441[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.44878791[Hz]
X_Sweep = 7.35294118[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 391.78655441[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 12
Total_Scans = 12
Relaxation_Delay = 5[s]
Recvr_Gain = 34
Temp_Get = 20.4[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.228224[s]
X_Angle = 45[deg]
X_Atn = 1.8[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition Time = 7.228224[s]

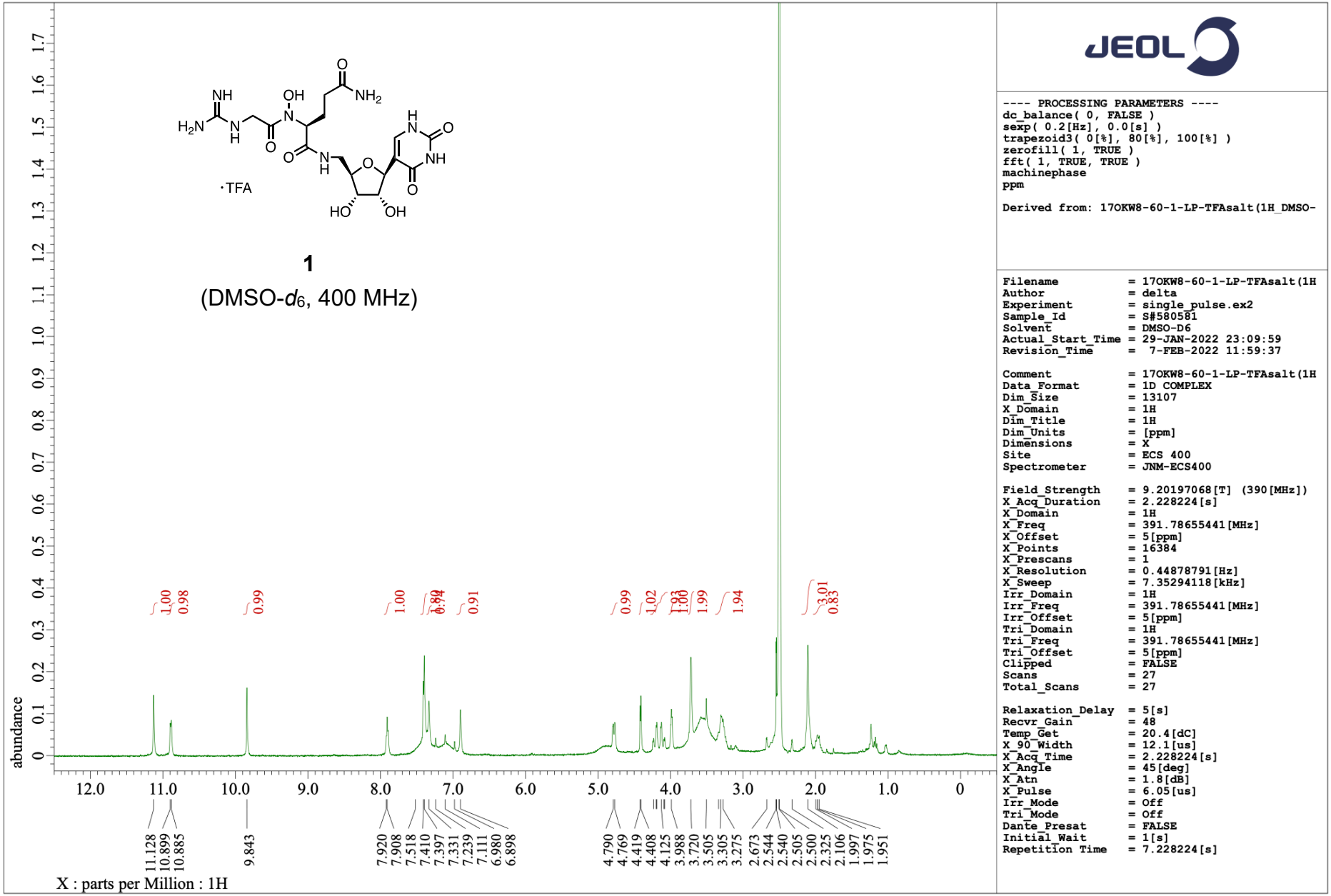

FmocHN
OBn
NH2
TBSO
OTBS
18
(DMSO-d6, 100 MHz)
abundance
X : parts per Million : 13C
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW8-82-1-MP(13C_DMSO-d6_400M
Filename = 17OKW8-82-1-MP(13C_DMSO-d
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#632354
Solvent = DMSO-D6
Actual_Start_Time = 10-FEB-2022 23:26:49
Revision_Time = 12-FEB-2022 12:27:42
Comment = 17OKW8-82-1-MP(13C_DMSO-d
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = 13C
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.2982153[T] (400[MHz])
X_Acq_Duration = 1.048576[s]
X_Domain = 13C
X_Freq = 99.54517646[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95367432[Hz]
X_Sweep = 31.25[kHz]
Irr_Domain = 1H
Irr_Freq = 395.88430144[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 711
Total_Scans = 711
Relaxation_Delay = 2[s]
Recvr_Gain = 54
Temp_Get = 408.6[dC]
X_90_Width = 10[us]
X_Acq_Time = 1.048576[s]
X_Angle = 30[deg]
X_Atn = 6.6[dB]
X_Pulse = 3.33333333[us]
Irr_Atn_Dec = 19.15[dB]
Irr_Atn_Noe = 19.15[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition Time = 3.048576[s]

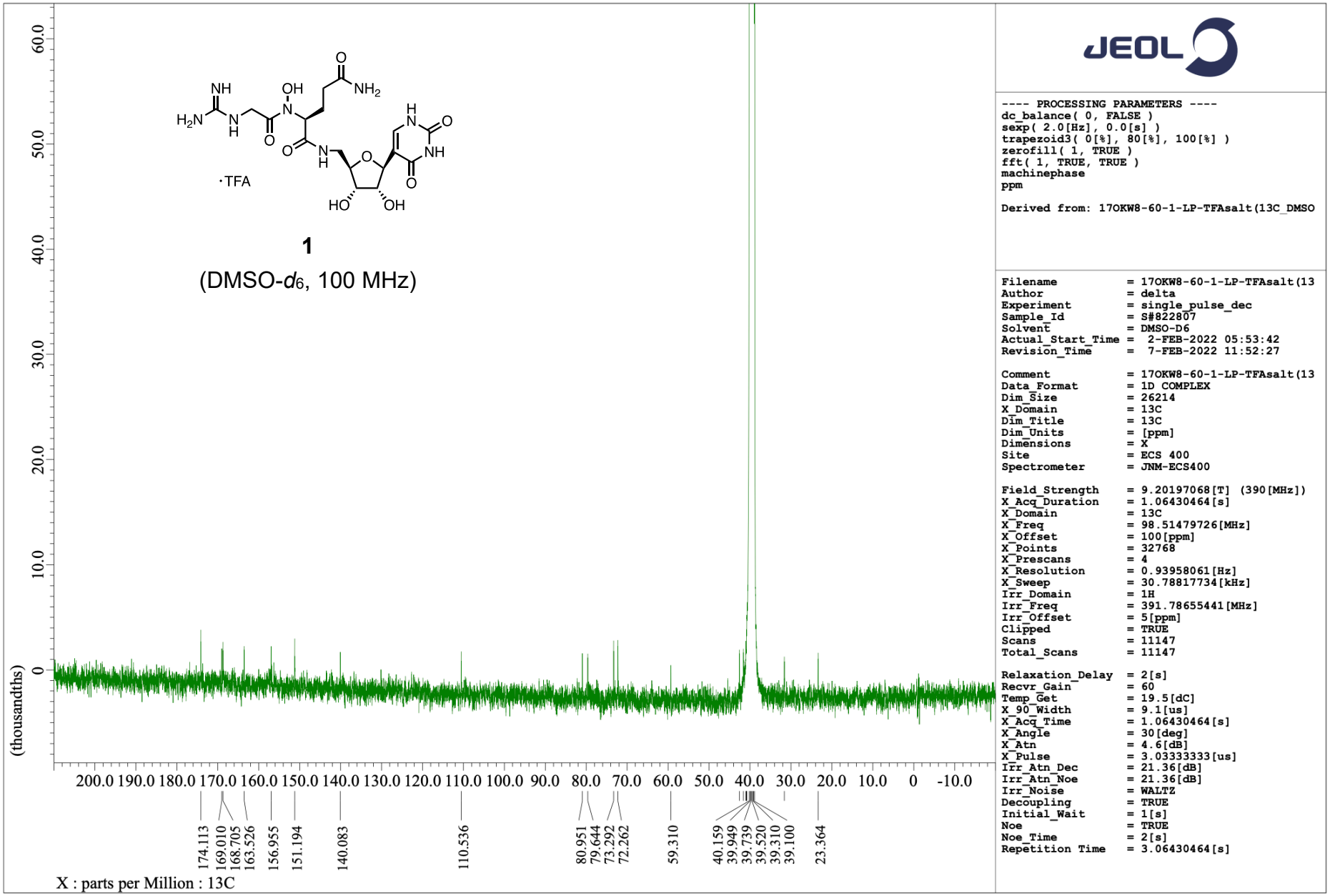

BocN
BocHN
OBn
NH2
TBSO
OTBS
17
(DMSO-d6, 400 MHz)
abundance
X : parts per Million : 1H
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW7-93-2-LP(1H_DMSO-d6_400MH
Filename = 17OKW7-93-2-LP(1H_DMSO-d6
Author = delta
Experiment = single_pulse.ex2
Sample_Id = S#820595
Solvent = DMSO-D6
Actual_Start_Time = 13-JAN-2022 04:42:52
Revision_Time = 4-FEB-2022 17:17:06
Comment = 17OKW7-93-2-LP(1H_DMSO-d6
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = 1H
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.2982153[T] (400[MHz])
X_Acq_Duration = 2.20725248[s]
X_Domain = 1H
X_Freq = 395.88430144[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45305193[Hz]
X_Sweep = 7.42280285[kHz]
Irr_Domain = 1H
Irr_Freq = 395.88430144[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 395.88430144[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 25
Total_Scans = 25
Relaxation_Delay = 5[s]
Recvr_Gain = 40
Temp_Get = 411.1[dC]
X_90_Width = 15.6[us]
X_Acq_Time = 2.20725248[s]
X_Angle = 45[deg]
X_Atn = 1.8[dB]
X_Pulse = 7.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition Time = 7.20725248[s]

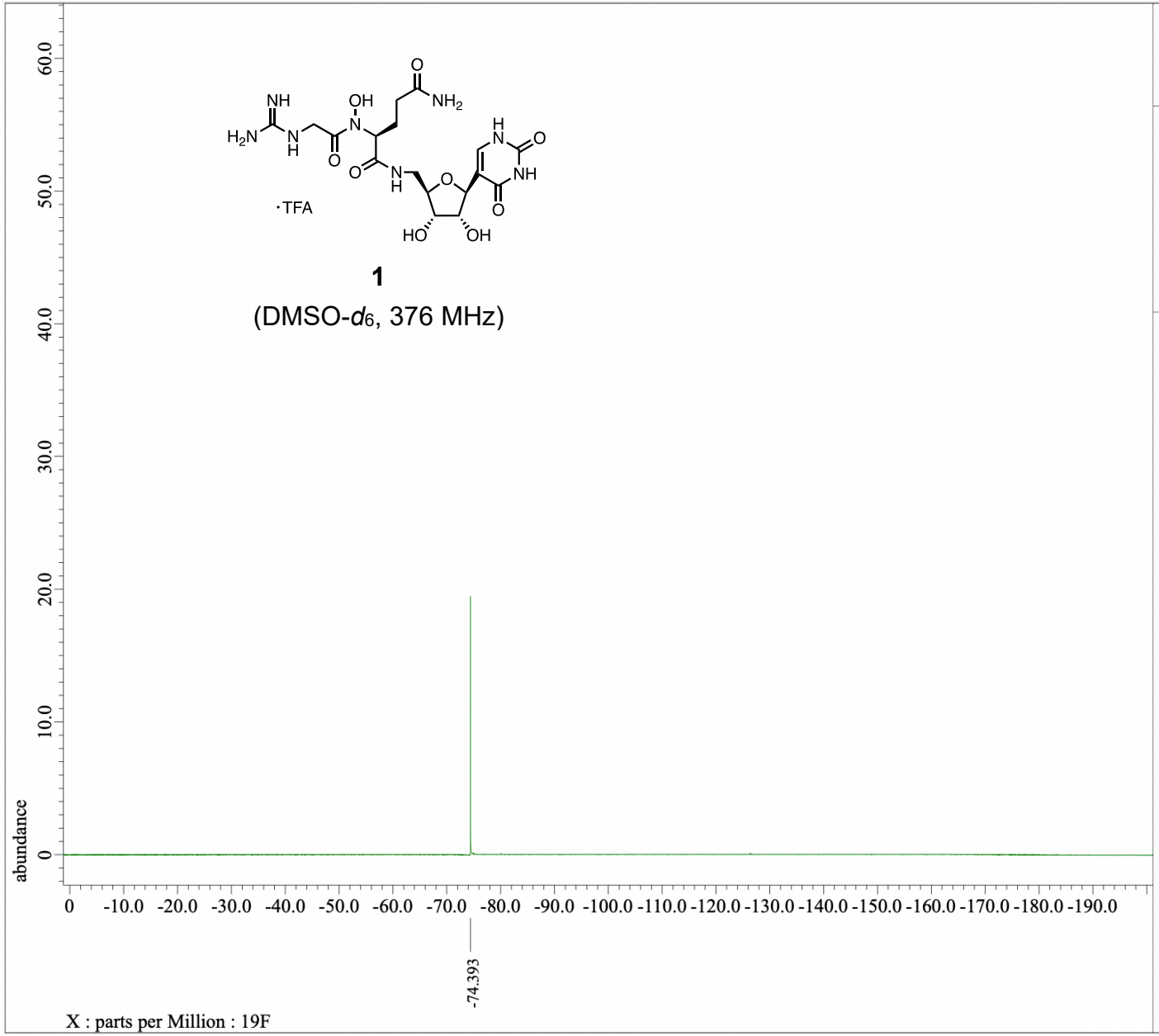

BocN
BocHN
OBn
NH2
TBSO
OTBS
17
(DMSO-d6, 100 MHz)
(thousandths)
X : parts per Million : 13C
173.261
171.106
169.046
163.451
162.780
155.030
151.935
151.131
141.158
134.518
129.211
128.741
128.464
109.485
83.110
81.855
79.460
79.154
78.301
77.592
73.952
73.837
61.124
39.942
39.731
39.520
39.309
27.899
27.554
25.753
25.734
17.734
17.639
-4.502
-4.770
-4.875
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW7-93-2-LP(13C_DMSO-d6_400M
Filename = 17OKW7-93-2-LP(13C_DMSO-d
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#828688
Solvent = DMSO-D6
Actual_Start_Time = 13-JAN-2022 04:55:52
Revision_Time = 4-FEB-2022 17:29:52
Comment = 17OKW7-93-2-LP(13C_DMSO-d
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = 13C
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.2982153[T] (400[MHz])
X_Acq_Duration = 1.048576[s]
X_Domain = 13C
X_Freq = 99.54517646[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95367432[Hz]
X_Sweep = 31.25[kHz]
Irr_Domain = 1H
Irr_Freq = 395.88430144[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 12636
Total_Scans = 12636
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 411.4[dC]
X_90_Width = 10[us]
X_Acq_Time = 1.048576[s]
X_Angle = 30[deg]
X_Atn = 6.6[dB]
X_Pulse = 3.33333333[us]
Irr_Atn_Dec = 19.15[dB]
Irr_Atn_Noe = 19.15[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition Time = 3.048576[s]


21
(DMSO-d_6, 400 MHz)
abundance
X : parts per Million : 1H
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW7-94-1-MP(1H_DMSO-d6_400MH
Filename = 17OKW7-94-1-MP(1H_DMSO-d6
Author = delta
Experiment = single_pulse.ex2
Sample_Id = S#553038
Solvent = DMSO-D6
Actual_Start_Time = 13-JAN-2022 21:16:40
Revision_Time = 4-FEB-2022 17:39:34
Comment = 17OKW7-94-1-MP(1H_DMSO-d6
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = 1H
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.2982153[T] (400[MHz])
X_Acq_Duration = 2.20725248[s]
X_Domain = 1H
X_Freq = 395.88430144[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45305193[Hz]
X_Sweep = 7.42280285[kHz]
Irr_Domain = 1H
Irr_Freq = 395.88430144[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 395.88430144[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 12
Total_Scans = 12
Relaxation_Delay = 5[s]
Recvr_Gain = 36
Temp_Get = 411.3[dC]
X_90_Width = 15.6[us]
X_Acq_Time = 2.20725248[s]
X_Angle = 45[deg]
X_Atn = 1.8[dB]
X_Pulse = 7.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition Time = 7.20725248[s]


BocN
BocHN
OBn
NH2
TBSO
OTBS
21
(DMSO-d6, 100 MHz)
abundance
0
0.1
0.2
0.3
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
173.340
170.879
169.058
163.526
162.839
155.066
151.986
151.175
141.237
134.580
129.201
128.752
128.495
109.544
83.116
81.904
79.606
79.224
78.318
77.651
74.046
73.941
61.323
39.730
39.520
39.301
39.091
27.942
27.589
25.786
24.069
17.775
17.698
-4.476
-4.810
X : parts per Million : 13C
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW7-94-1-MP(13C_DMSO-d6_400M
Filename = 17OKW7-94-1-MP(13C_DMSO-d
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#396950
Solvent = DMSO-D6
Actual_Start_Time = 13-JAN-2022 18:03:55
Revision_Time = 4-FEB-2022 17:30:58
Comment = 17OKW7-94-1-MP(13C_DMSO-d
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = 13C
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain = 13C
X_Freq = 98.51479726[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.93958061[Hz]
X_Sweep = 30.78817734[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 3499
Total_Scans = 3499
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 20.9[dC]
X_90_Width = 9.1[us]
X_Acq_Time = 1.06430464[s]
X_Angle = 30[deg]
X_Atn = 4.6[dB]
X_Pulse = 3.03333333[us]
Irr_Atn_Dec = 21.36[dB]
Irr_Atn_Noe = 21.36[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition Time = 3.06430464[s]

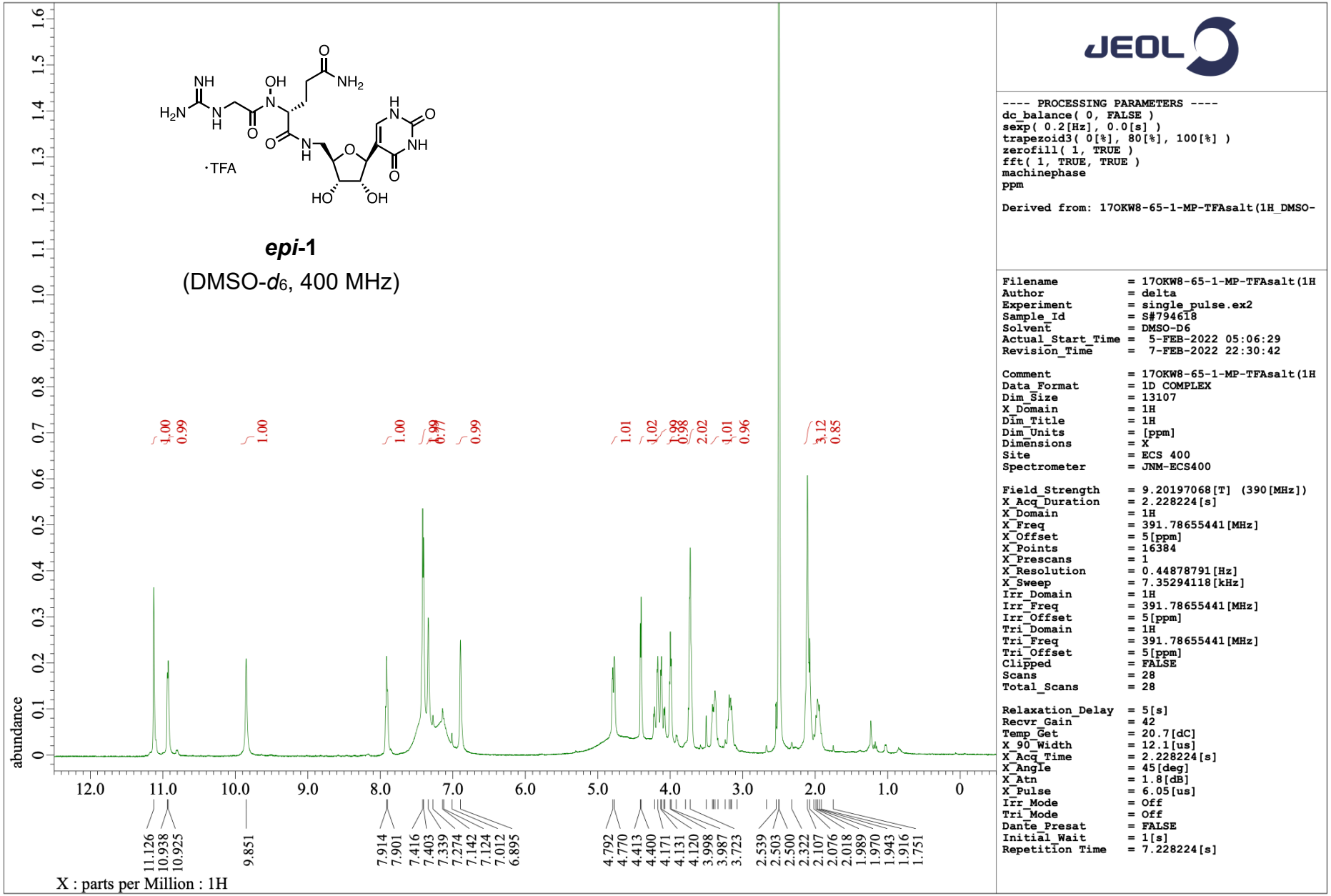

·TFA
1
(DMSO-d_6, 400 MHz)
abundance
X : parts per Million : 1H
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW8-60-1-LP-TFAsalt(1H_DMSO-
Filename = 17OKW8-60-1-LP-TFAsalt(1H
Author = delta
Experiment = single_pulse.ex2
Sample_Id = S#580581
Solvent = DMSO-D6
Actual_Start_Time = 29-JAN-2022 23:09:59
Revision_Time = 7-FEB-2022 11:59:37
Comment = 17OKW8-60-1-LP-TFAsalt(1H
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = 1H
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 2.228224[s]
X_Domain = 1H
X_Freq = 391.78655441[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.44878791[Hz]
X_Sweep = 7.35294118[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 391.78655441[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 27
Total_Scans = 27
Relaxation_Delay = 5[s]
Recvr_Gain = 48
Temp_Get = 20.4[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.228224[s]
X_Angle = 45[deg]
X_Atn = 1.8[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition Time = 7.228224[s]


·TFA
1
(DMSO-d6, 100 MHz)
(thousandths)
60.0
50.0
40.0
30.0
20.0
10.0
0
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
174.113
169.010
168.705
163.526
156.955
151.194
140.083
110.536
80.951
79.644
73.292
72.262
59.310
40.159
39.949
39.739
39.520
39.310
39.100
23.364
X : parts per Million : 13C
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW8-60-1-LP-TFAsalt(13C_DMSO
Filename = 17OKW8-60-1-LP-TFAsalt(13
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#822807
Solvent = DMSO-D6
Actual_Start_Time = 2-FEB-2022 05:53:42
Revision_Time = 7-FEB-2022 11:52:27
Comment = 17OKW8-60-1-LP-TFAsalt(13
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = 13C
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain = 13C
X_Freq = 98.51479726[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.93958061[Hz]
X_Sweep = 30.78817734[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 11147
Total_Scans = 11147
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 19.5[dC]
X_90_Width = 9.1[us]
X_Acq_Time = 1.06430464[s]
X_Angle = 30[deg]
X_Atn = 4.6[dB]
X_Pulse = 3.03333333[us]
Irr_Atn_Dec = 21.36[dB]
Irr_Atn_Noe = 21.36[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition Time = 3.06430464[s]



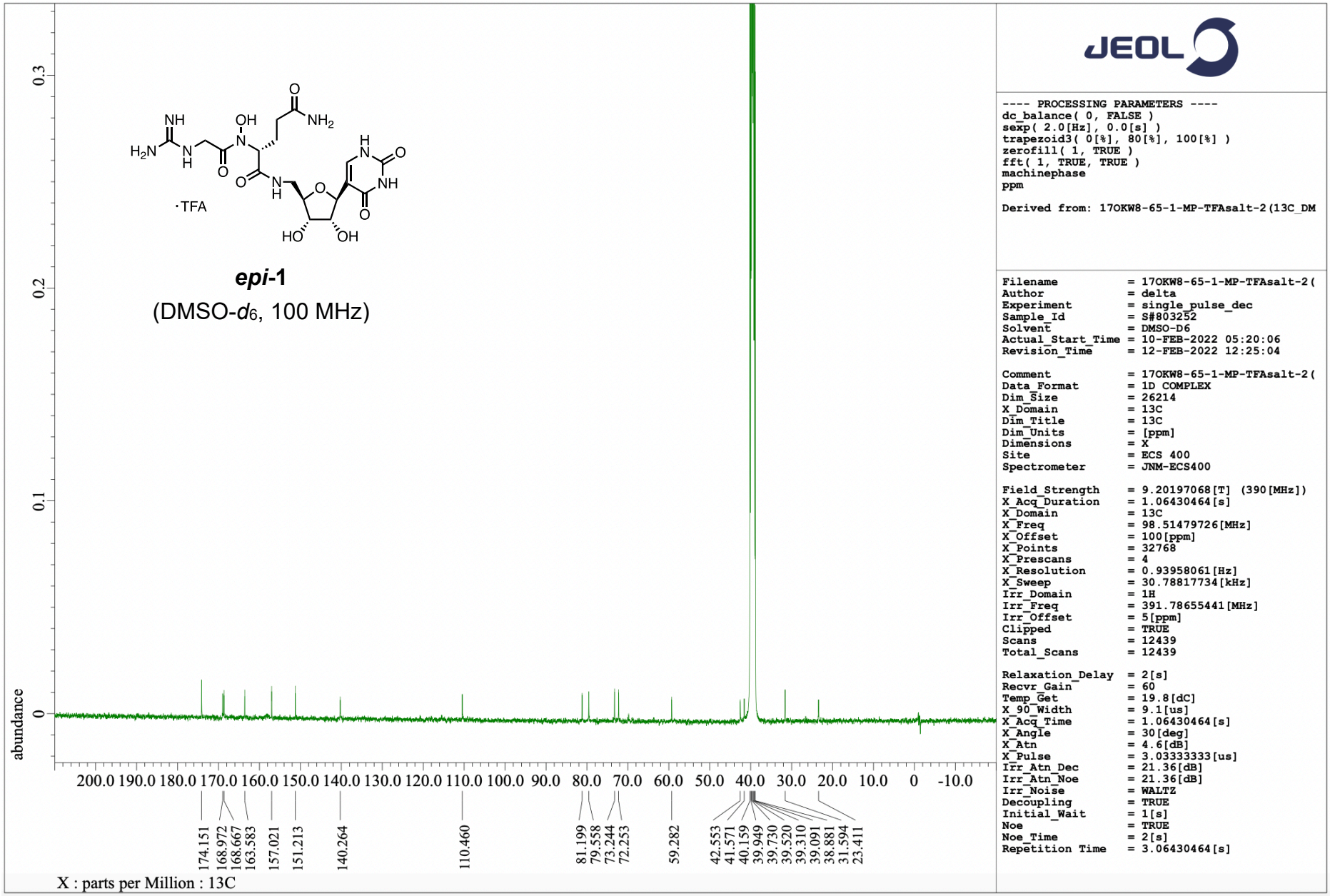

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -5, 0, 50[%] )

Derived from: 17OKW8-60-1-LP-TFAsalt-4(19F_DM
```

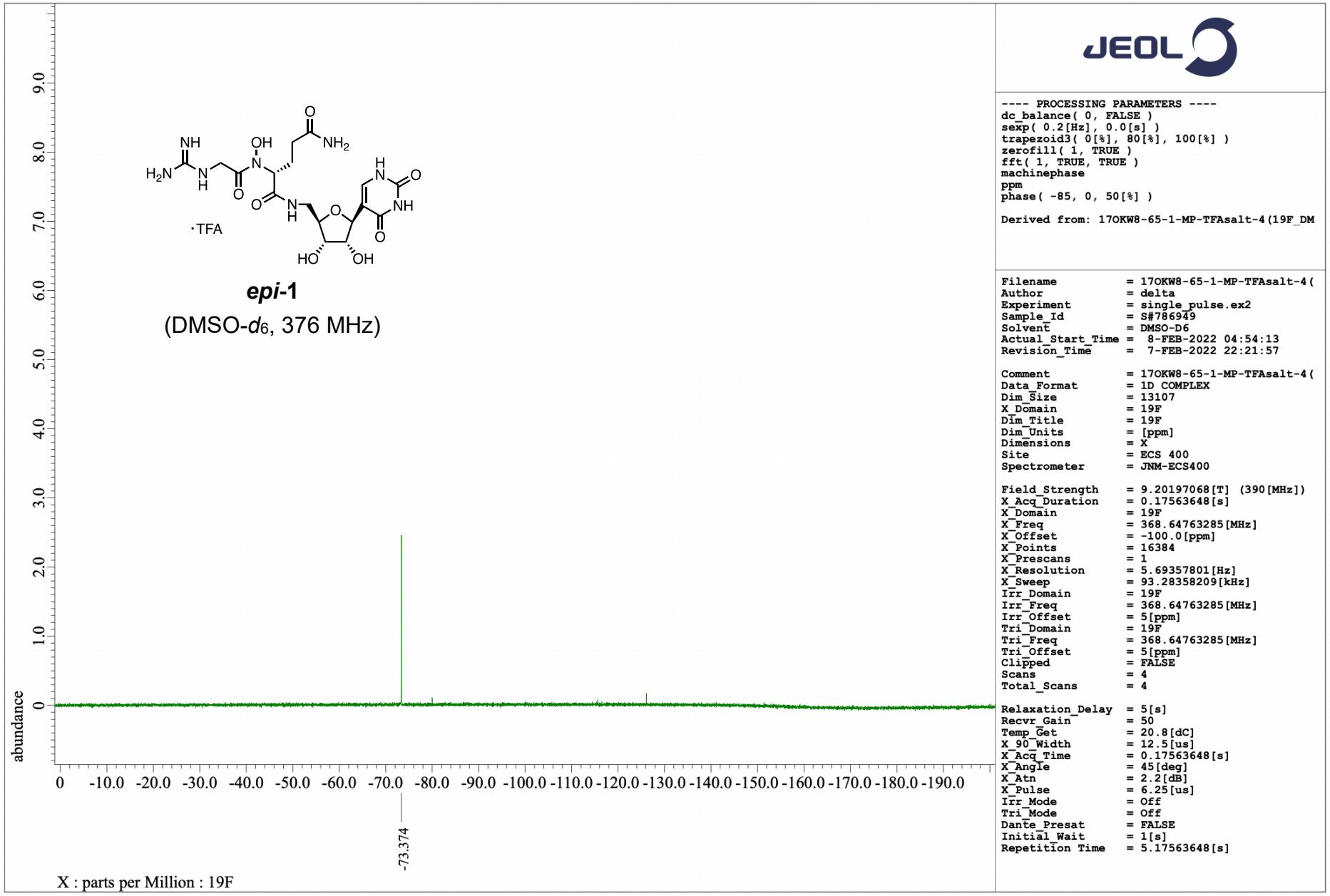
```
Filename            = 17OKW8-60-1-LP-TFAsalt-4(
Author              = delta
Experiment          = single_pulse.ex2
Sample_Id           = S#757858
Solvent             = DMSO-D6
Actual_Start_Time   =  8-FEB-2022 04:05:45
Revision_Time       =  7-FEB-2022 22:22:42

Comment             = 17OKW8-60-1-LP-TFAsalt-4(
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = 19F
Dim_Title           = 19F
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = JNM-ECS400

Field_Strength      = 9.20197068[T]  (390[MHz])
X_Acq_Duration      = 0.17563648[s]
X_Domain            = 19F
X_Freq              = 368.64763285[MHz]
X_Offset            = -100.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 5.69357801[Hz]
X_Sweep             = 93.28358209[kHz]
Irr_Domain          = 19F
Irr_Freq            = 368.64763285[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = 19F
Tri_Freq            = 368.64763285[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 4
Total_Scans         = 4

Relaxation_Delay    = 5[s]
Recvr_Gain          = 50
Temp_Get            = 20.6[dC]
X_90_Width          = 12.5[us]
X_Acq_Time          = 0.17563648[s]
X_Angle             = 45[deg]
X_Atn               = 2.2[dB]
X_Pulse             = 6.25[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition Time     = 5.17563648[s]
```



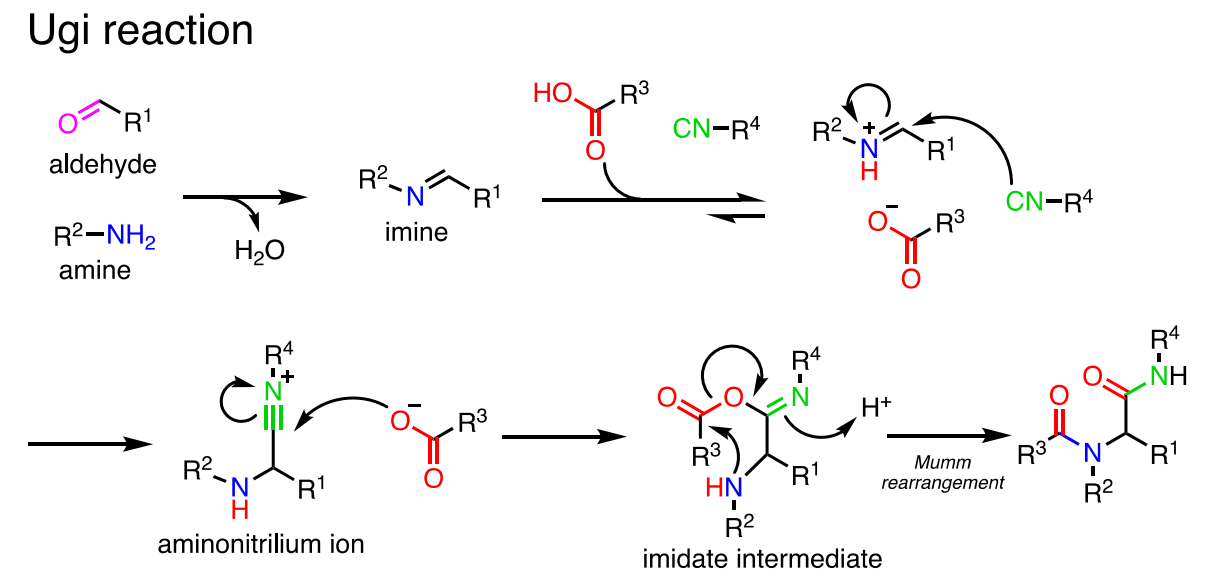

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: 17OKW8-65-1-MP-TFAsalt(1H_DMSO-
```

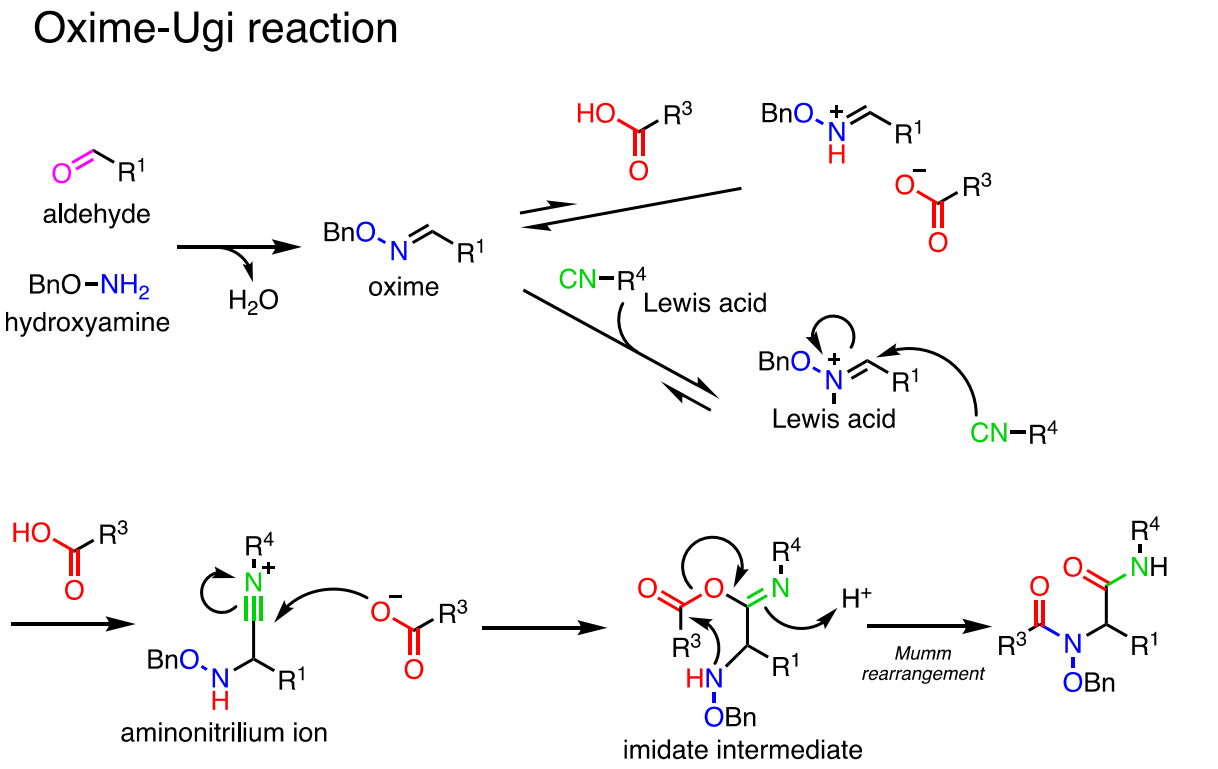
```
Filename            = 17OKW8-65-1-MP-TFAsalt(1H
Author              = delta
Experiment          = single_pulse.ex2
Sample_Id           = S#794618
Solvent             = DMSO-D6
Actual_Start_Time   =  5-FEB-2022 05:06:29
Revision_Time       =  7-FEB-2022 22:30:42

Comment             = 17OKW8-65-1-MP-TFAsalt(1H
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = 1H
Dim_Title           = 1H
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = JNM-ECS400

Field_Strength      = 9.20197068[T] (390[MHz])
X_Acq_Duration      = 2.228224[s]
X_Domain            = 1H
X_Freq              = 391.78655441[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.44878791[Hz]
X_Sweep             = 7.35294118[kHz]
Irr_Domain          = 1H
Irr_Freq            = 391.78655441[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = 1H
Tri_Freq            = 391.78655441[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 28
Total_Scans         = 28

Relaxation_Delay    = 5[s]
Recvr_Gain          = 42
Temp_Get            = 20.7[dC]
X_90_Width          = 12.1[us]
X_Acq_Time          = 2.228224[s]
X_Angle             = 45[deg]
X_Atn               = 1.8[dB]
X_Pulse             = 6.05[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition Time     = 7.228224[s]
```


·TFA
***epi*-1**
(DMSO-d_6, 100 MHz)
abundance
0.3
0.2
0.1
0
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
174.151
168.972
168.667
163.583
157.021
151.213
140.264
110.460
81.199
79.558
73.244
72.253
59.282
42.553
41.571
40.159
39.949
39.730
39.520
39.310
39.091
38.881
31.594
23.411
X : parts per Million : 13C
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: 17OKW8-65-1-MP-TFAsalt-2(13C_DM
Filename = 17OKW8-65-1-MP-TFAsalt-2(
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#803252
Solvent = DMSO-D6
Actual_Start_Time = 10-FEB-2022 05:20:06
Revision_Time = 12-FEB-2022 12:25:04
Comment = 17OKW8-65-1-MP-TFAsalt-2(
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = 13C
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain = 13C
X_Freq = 98.51479726[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.93958061[Hz]
X_Sweep = 30.78817734[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 12439
Total_Scans = 12439
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 19.8[dC]
X_90_Width = 9.1[us]
X_Acq_Time = 1.06430464[s]
X_Angle = 30[deg]
X_Atn = 4.6[dB]
X_Pulse = 3.03333333[us]
Irr_Atn_Dec = 21.36[dB]
Irr_Atn_Noe = 21.36[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition Time = 3.06430464[s]

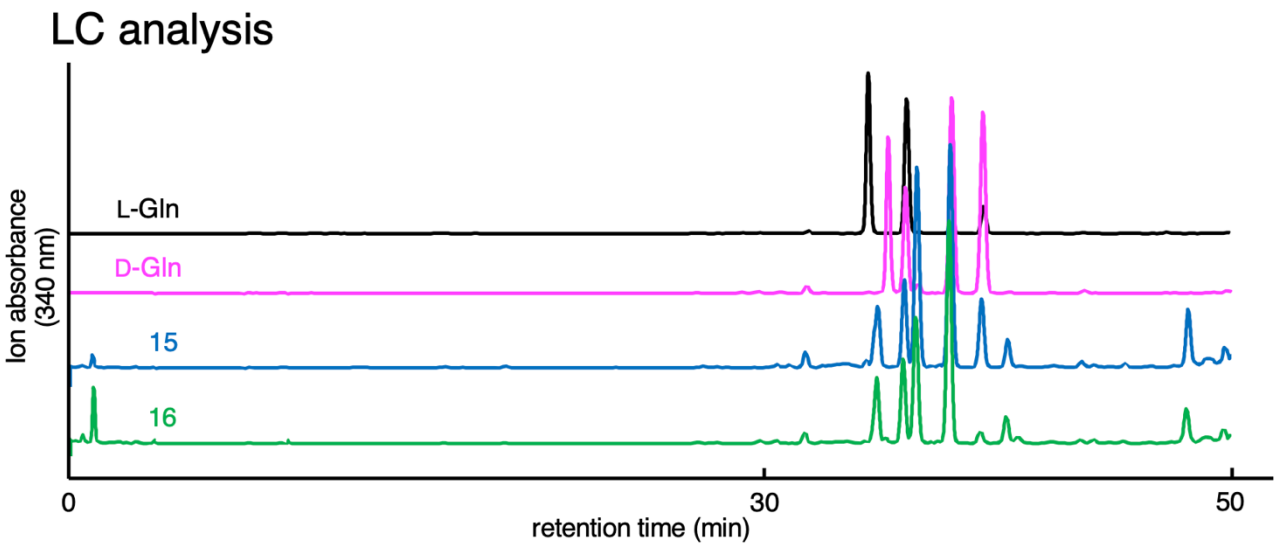

·TFA
***epi*-1**
(DMSO-*d*6, 376 MHz)
abundance
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
0 -10.0 -20.0 -30.0 -40.0 -50.0 -60.0 -70.0 -80.0 -90.0 -100.0 -110.0 -120.0 -130.0 -140.0 -150.0 -160.0 -170.0 -180.0 -190.0
-73.374
X : parts per Million : 19F
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
phase(-85, 0, 50[%])
Derived from: 17OKW8-65-1-MP-TFAsalt-4(19F_DM
Filename = 17OKW8-65-1-MP-TFAsalt-4(
Author = delta
Experiment = single_pulse.ex2
Sample_Id = S#786949
Solvent = DMSO-D6
Actual_Start_Time = 8-FEB-2022 04:54:13
Revision_Time = 7-FEB-2022 22:21:57
Comment = 17OKW8-65-1-MP-TFAsalt-4(
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = 19F
Dim_Title = 19F
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 0.17563648[s]
X_Domain = 19F
X_Freq = 368.64763285[MHz]
X_Offset = -100.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 5.69357801[Hz]
X_Sweep = 93.28358209[kHz]
Irr_Domain = 19F
Irr_Freq = 368.64763285[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 19F
Tri_Freq = 368.64763285[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 4
Total_Scans = 4
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 20.8[dC]
X_90_Width = 12.5[us]
X_Acq_Time = 0.17563648[s]
X_Angle = 45[deg]
X_Atn = 2.2[dB]
X_Pulse = 6.25[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition Time = 5.17563648[s]

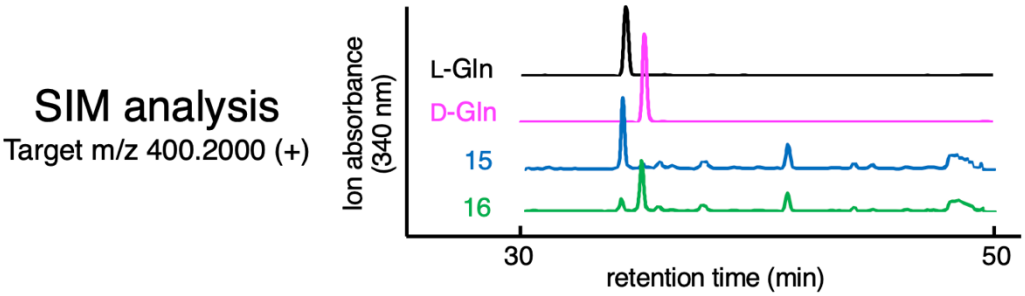
3) Reaction mechanism of Ugi multicomponent reaction



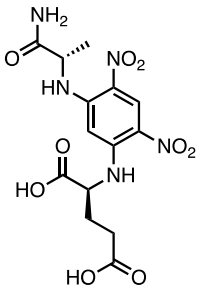


4) Marfey's analysis of compound 15 and 16

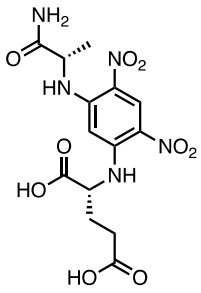




L-FDAA derivatives



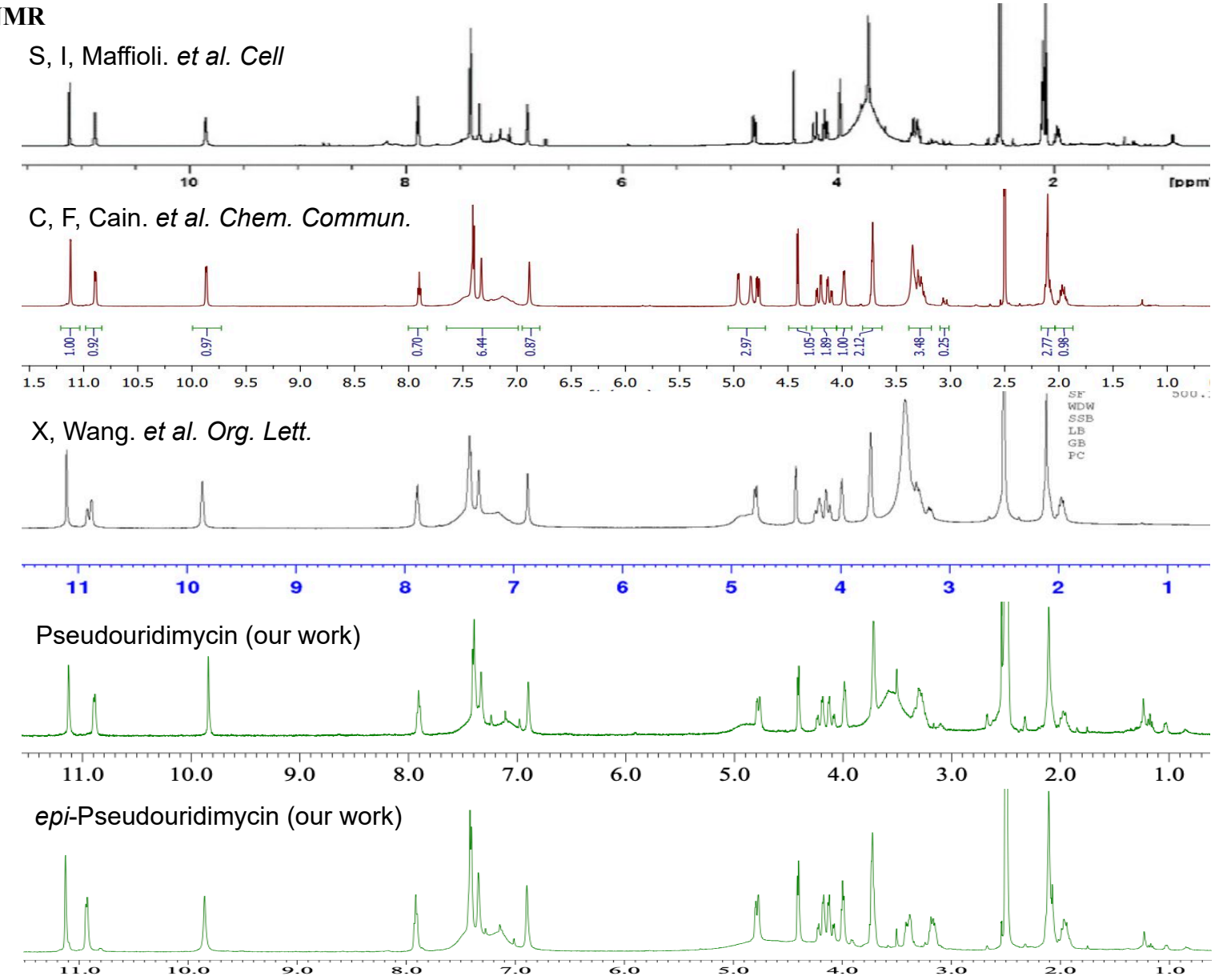
Exact Mass: 399.10



Exact Mass: 399.10

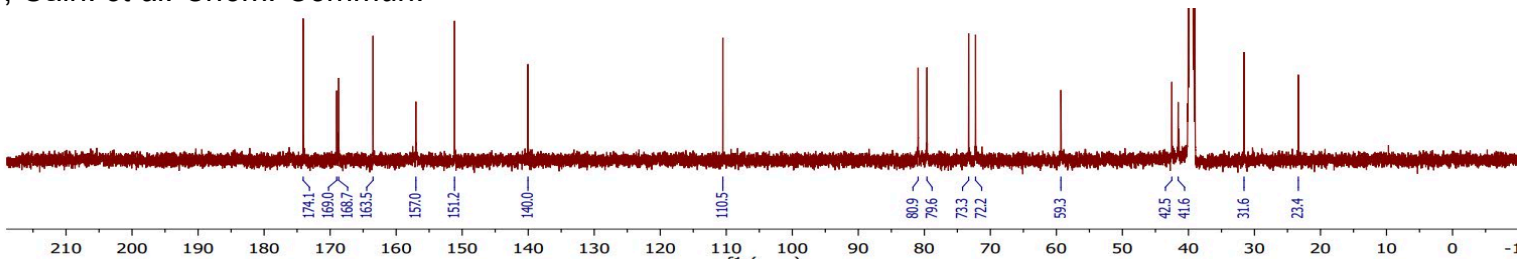
5) Comparison of ^{1}H NMR and ^{13}C NMR charts of natural and synthetic pseudouridimycin and *epi*-psuedouridimycin (DMSO-d_6)

^{1}H NMR

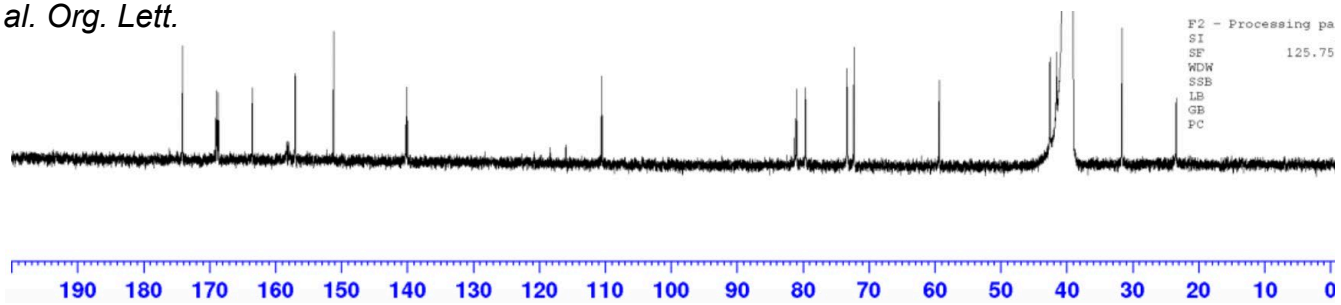


^{13}C NMR

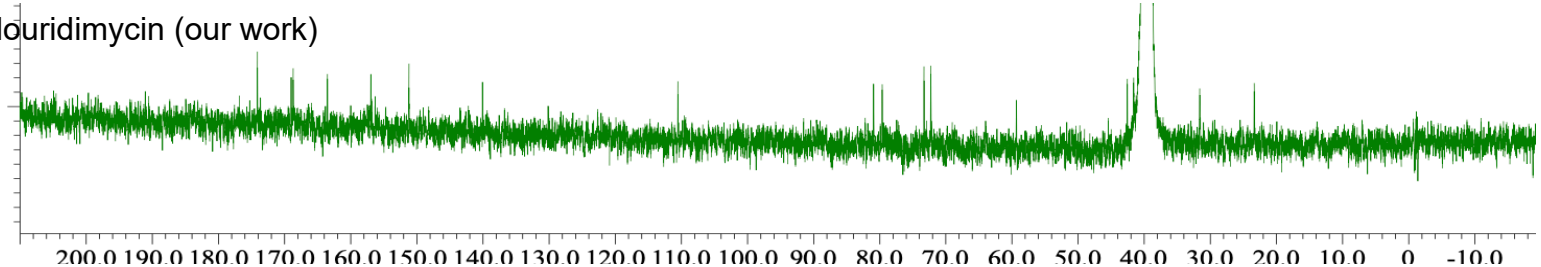
C, F, Cain. *et al. Chem. Commun.*



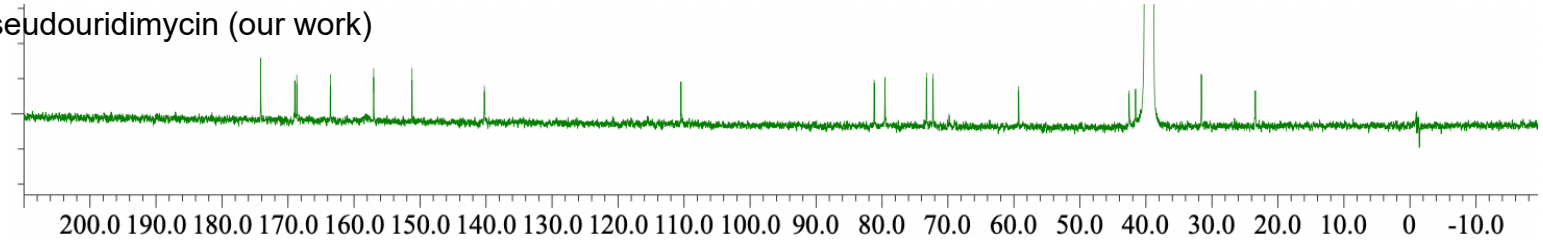
X, Wang. *et al. Org. Lett.*



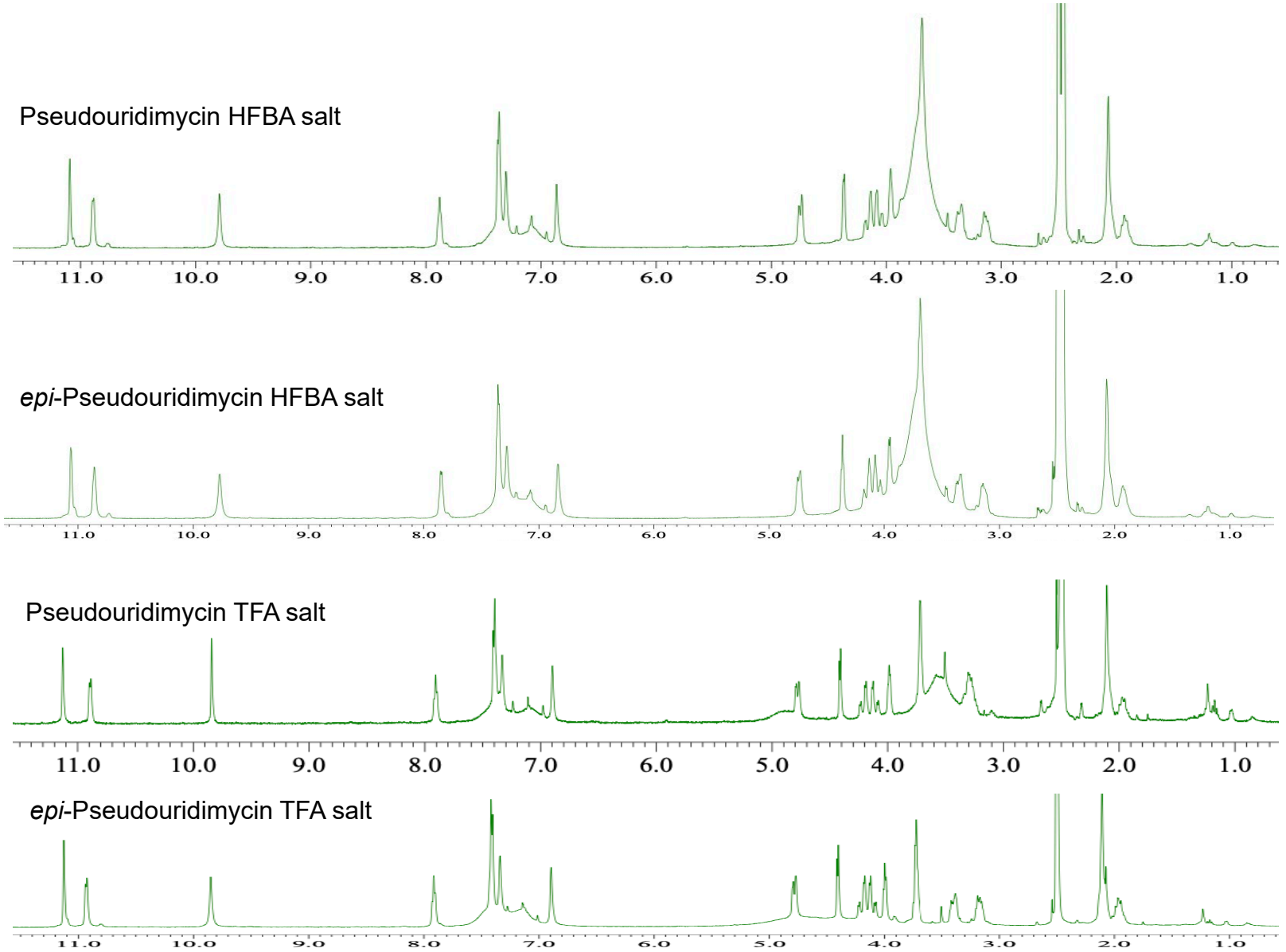
Pseudouridimycin (our work)

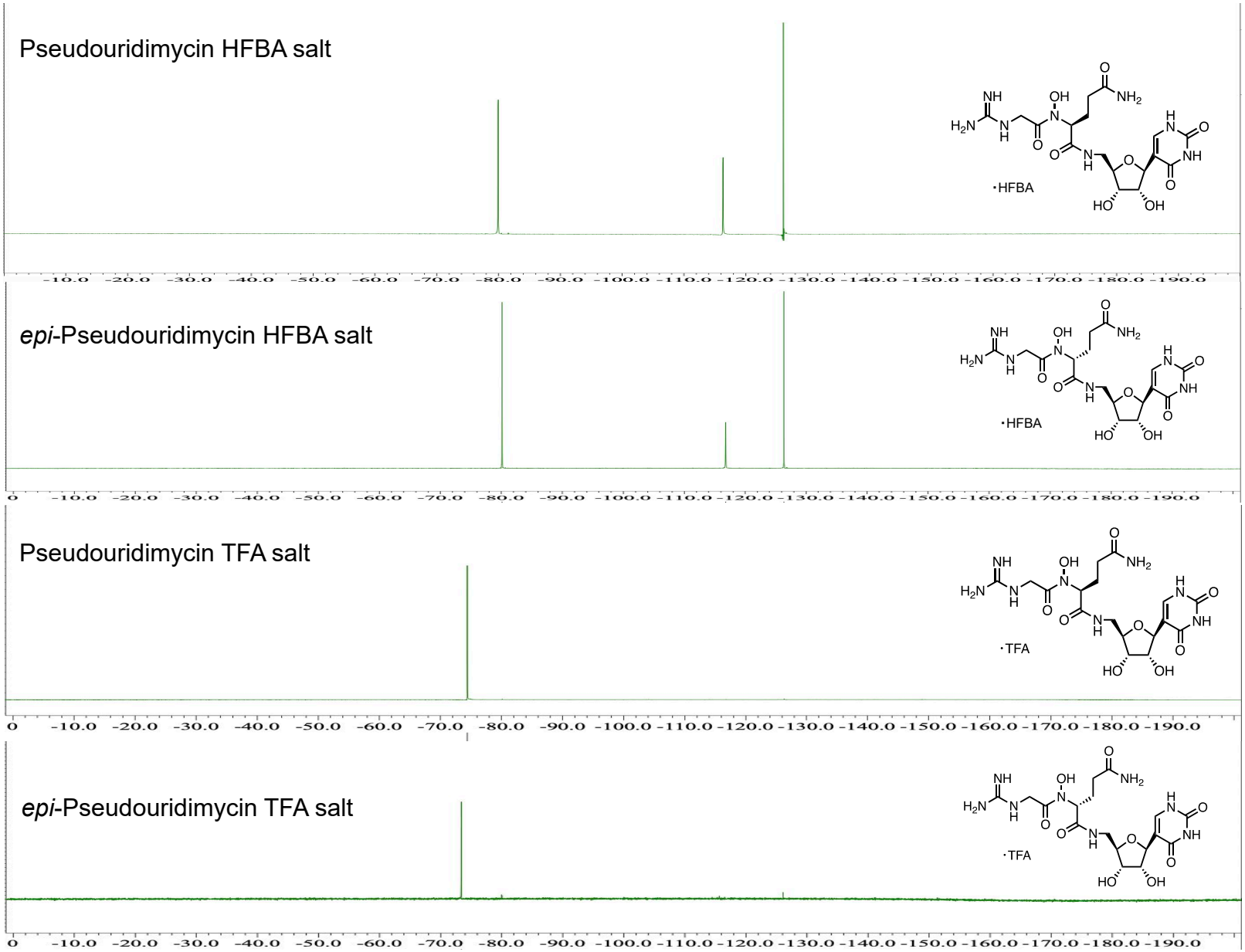


epi-Pseudouridimycin (our work)

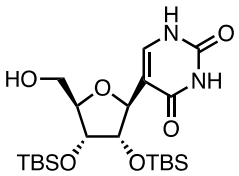


6) comparison of ^{1}H-NMR and ^{19}F-NMR charts of two types of pseudopuridimycin and *epi*-pseudouridimycin salts (DMSO-d_6)



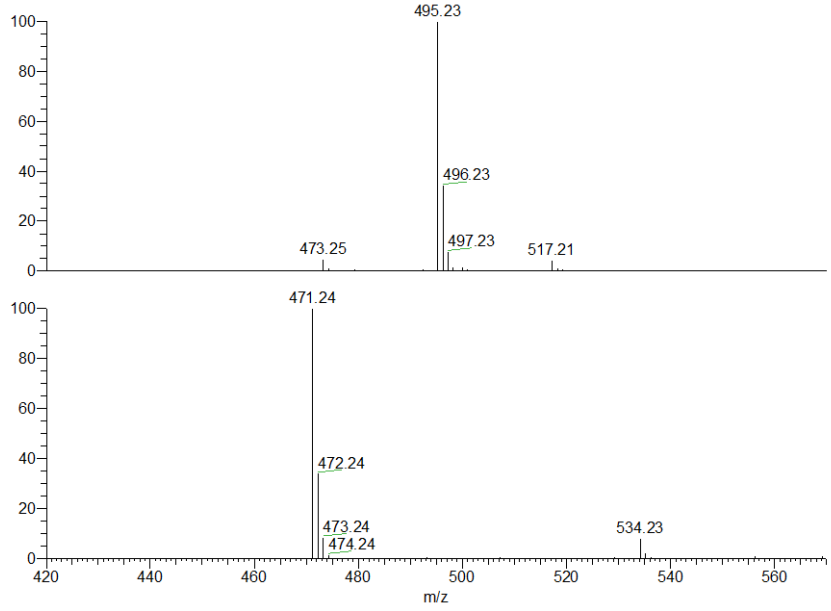

Pseudouridimycin HFBA salt
·HFBA
epi-Pseudouridimycin HFBA salt
·HFBA
Pseudouridimycin TFA salt
·TFA
epi-Pseudouridimycin TFA salt
·TFA

7) LR-MS and HR-MS charts of synthesized compounds

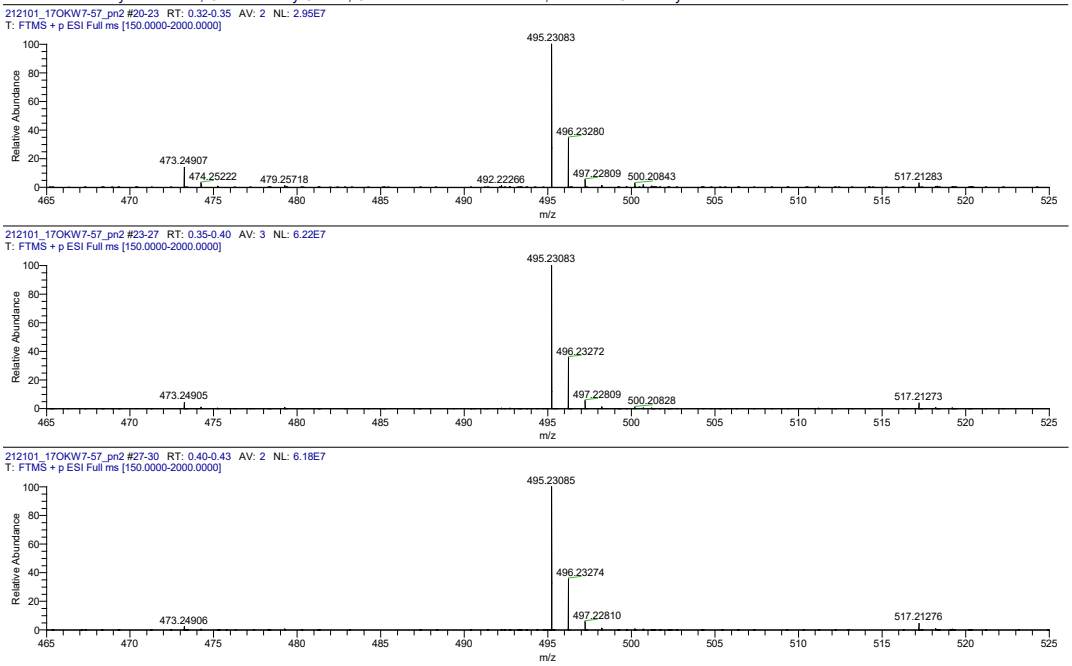


2′,3′-Di-*O*-*tert*-butyldimethylsilylpseudouridine

Sample No. : C:\Xcalibur\...\BG_212101_17OKW _ 2 Instrument : Exactive Plus Mobile phase solvent : MeOH
Operator name : Yamashita Nao Sample solvent : MeOH
Date : 02/03/22 10:44:01
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University



Sample No. : C:\Xcalibur\...\212101_17OKW7-57_pn2 Instrument : Exactive Plus Mobile phase solvent : MeOH
Operator name : Yamashita Nao Sample solvent : MeOH
Date : 02/03/22 10:21:42
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University



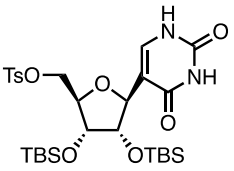
Elemental composition search on mass 495.23

m/z= 490.23-500.23

Isotope	Min	Max
N-14	0	2
O-16	0	15
C-12	0	100
H-1	0	200
Na-23	0	1
Si-28	0	2

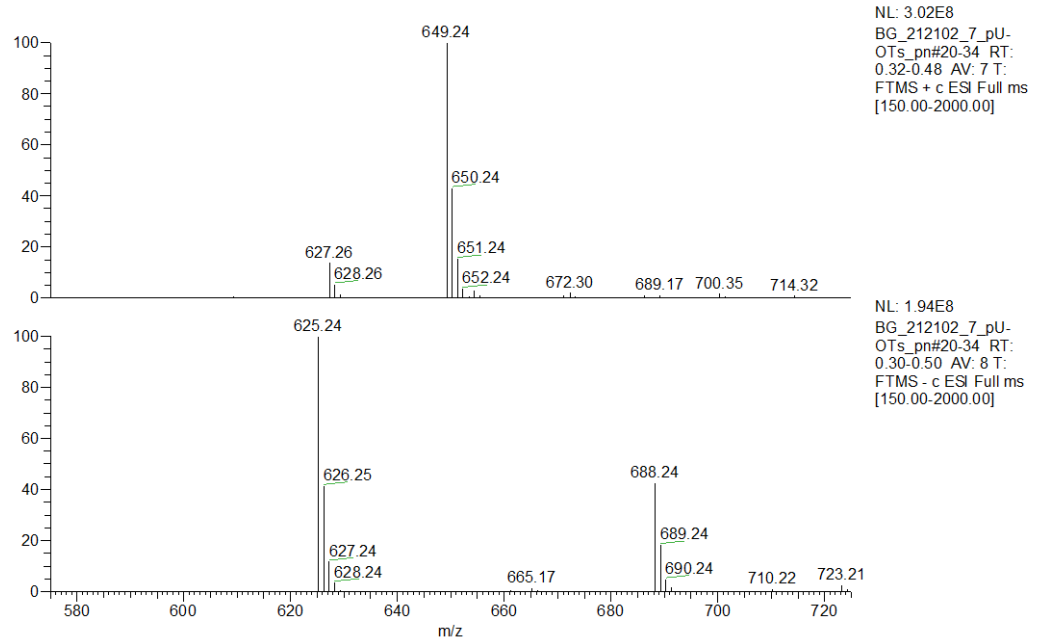
Charge 1
Mass tolerance 5.00 ppm
Nitrogen rule not used
RDB equiv -1.00-100.00
max results 100

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
495.23083	495.23098	-0.29	12.5	$C_{27}H_{35}O_5N_2Si$
	495.23103	-0.40	4.0	$C_{21}H_{37}O_{12}N$
	495.23130	-0.95	5.5	$C_{22}H_{36}O_9N_2Na$
	495.23144	-1.22	3.0	$C_{20}H_{41}O_9NSi_2$
	495.23171	-1.78	4.5	$C_{21}H_{40}O_6N_2NaSi_2$
	495.23186	-2.07	21.5	$C_{36}H_{31}O_2$
	495.22945	2.78	18.5	$C_{34}H_{32}O_2Na$
	495.23259	-3.56	13.5	$C_{30}H_{36}O_3NaSi$
	495.22903	3.63	0.0	$C_{18}H_{42}O_9NNaSi_2$
	495.22862	4.46	1.0	$C_{19}H_{38}O_{12}NNa$
	495.22857	4.56	9.5	$C_{25}H_{36}O_5N_2NaSi$

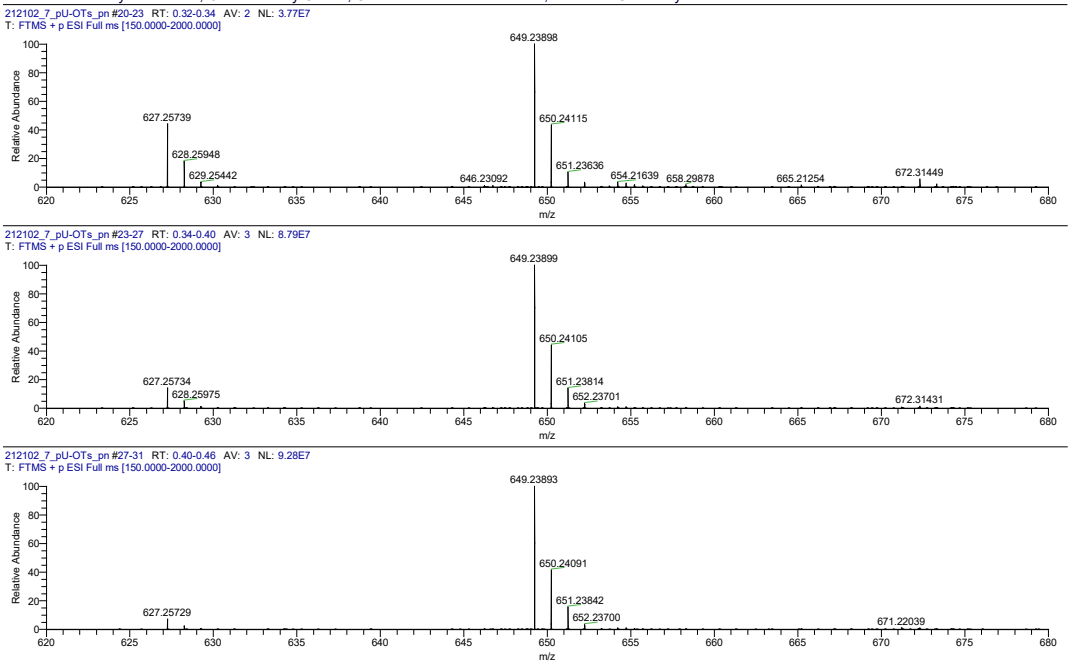


2′,3′-Di-*O*-*tert*-butyldimethylsilyl-5′-*O*-*p*-toluenesulfonylpseudouridine

Sample No. : C:\Xcalibur\...\BG_212102_7_pU-O` _,
Operator name : Yamashita Nao
Date : 02/03/22 11:09:11
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University
Instrument : Exactive Plus
Mobile phase solvent : MeOH
Sample solvent : MeOH



Sample No. : C:\Xcalibur\...\0203\212102_7_pU-OTs_pn
Operator name : Yamashita Nao
Date : 02/03/22 10:40:51
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University
Instrument : Exactive Plus
Mobile phase solvent : MeOH
Sample solvent : MeOH



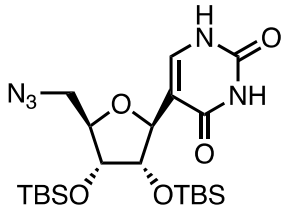
Elemental composition search on mass 649.24

m/z= 644.24-654.24

Isotope	Min	Max
N-14	0	2
O-16	0	15
C-12	0	100
H-1	0	200
Na-23	1	1
Si-28	0	2
S-32	0	1

Charge 1
Mass tolerance 5.00 ppm
Nitrogen rule not used
RDB equiv -1.00-100.00
max results 20

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
649.23899	649.23871	0.43	21.5	$C_{40}H_{42}ONaSSi_2$
	649.23830	1.06	22.5	$C_{41}H_{38}O_4NaS$
	649.23807	1.41	22.5	$C_{40}H_{38}O_5NaSi$
	649.23992	-1.44	9.5	$C_{28}H_{42}O_{12}N_2NaSi$
	649.23788	1.71	4.0	$C_{25}H_{48}O_{11}NNaSSi_2$
	649.24015	-1.79	9.5	$C_{29}H_{42}O_{11}N_2NaS$
	649.23761	2.12	32.0	$C_{47}H_{32}ONNa$
	649.23747	2.34	5.0	$C_{26}H_{44}O_{14}NNaS$
	649.23742	2.42	13.5	$C_{32}H_{42}O_7N_2NaSSi$
	649.24056	-2.42	8.5	$C_{28}H_{46}O_8N_2NaSSi_2$
	649.24061	-2.50	0.0	$C_{22}H_{48}O_{15}NNaSSi$
	649.23724	2.69	5.0	$C_{25}H_{44}O_{15}NNaSi$
	649.24075	-2.71	27.0	$C_{43}H_{36}O_2NNaSi$
	649.23719	2.77	13.5	$C_{31}H_{42}O_8N_2NaSi_2$
	649.24080	-2.79	18.5	$C_{37}H_{38}O_9Na$
	649.24098	-3.07	27.0	$C_{44}H_{36}ONNaS$
	649.23678	3.40	14.5	$C_{32}H_{38}O_{11}N_2Na$
	649.24121	-3.42	17.5	$C_{36}H_{42}O_6NaSi_2$
	649.24144	-3.78	17.5	$C_{37}H_{42}O_5NaSSi$

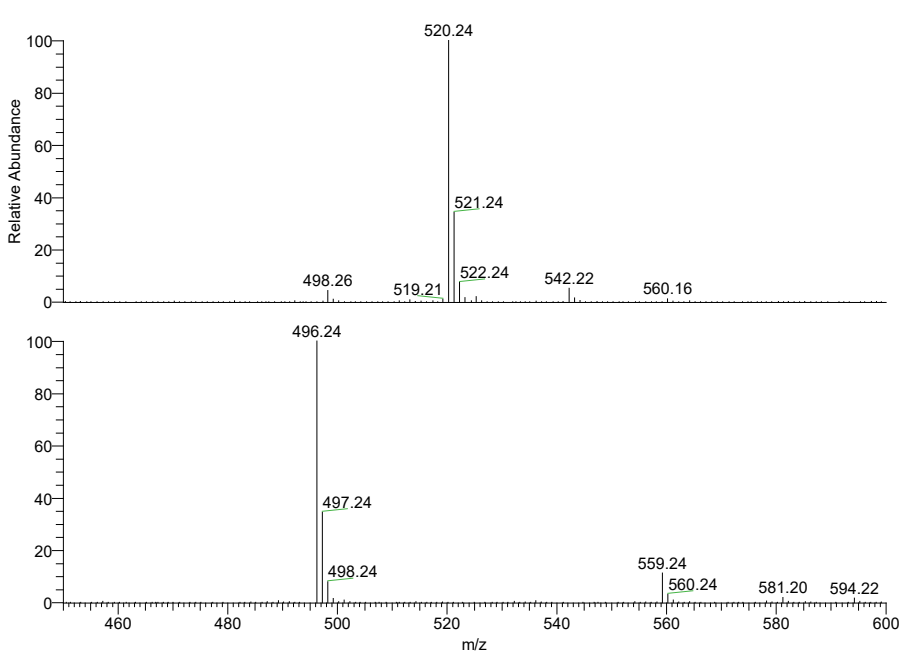


7

Sample No. : C:\Xcalibur\...\BG_212103_7_pU_N3_pn
Operator name : Yamashita Nao
Date : 02/03/22 11:23:09
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

Instrument : Exactive Plus

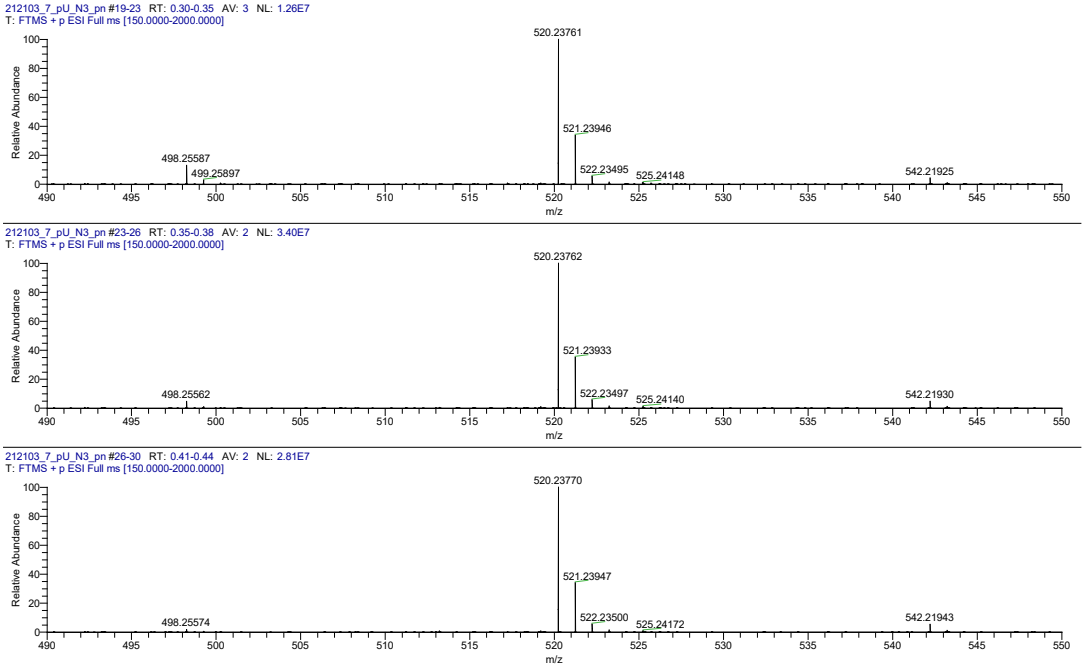
Mobile phase solvent : MeOH
Sample solvent : MeOH



Sample No. : C:\Xcalibur\...\0203\212103_7_pU_N3_pn
Operator name : Yamashita Nao
Date : 02/03/22 10:45:37
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

Instrument : Exactive Plus

Mobile phase solvent : MeOH
Sample solvent : MeOH



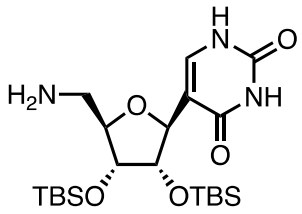
Elemental composition search on mass 520.24

m/z= 515.24-525.24

Isotope	Min	Max
N-14	0	10
O-16	0	15
C-12	0	100
H-1	0	200
Na-23	1	1
Si-28	0	2

Charge 1
Mass tolerance 5.00 ppm
Nitrogen rule not used
RDB equiv -1.00-100.00
max results 20

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
520.23762	520.23773	-0.22	16.0	$C_{28}H_{33}ON_6NaSi$
	520.23778	-0.32	7.5	$C_{22}H_{35}O_8N_5Na$
	520.23728	0.66	20.0	$C_{36}H_{33}O_2Na$
	520.23819	-1.10	6.5	$C_{21}H_{39}O_5N_5NaSi_2$
	520.23686	1.47	1.5	$C_{20}H_{43}O_9NNaSi_2$
	520.23685	1.48	7.0	$C_{19}H_{37}O_4N_8NaSi_2$
	520.23645	2.26	2.5	$C_{21}H_{39}O_{12}NNa$
	520.23644	2.27	8.0	$C_{20}H_{33}O_7N_8Na$
	520.23639	2.35	11.0	$C_{27}H_{37}O_5N_2NaSi$
	520.23639	2.36	16.5	$C_{26}H_{31}N_9NaSi$
	520.23908	-2.80	15.5	$C_{30}H_{35}O_2N_3NaSi$
	520.23912	-2.89	12.5	$C_{23}H_{31}O_4N_9Na$
	520.23913	-2.90	7.0	$C_{24}H_{37}O_9N_2Na$
	520.23593	3.24	20.5	$C_{34}H_{31}ON_3Na$
	520.23953	-3.67	11.5	$C_{22}H_{35}ON_9NaSi_2$
	520.23954	-3.68	6.0	$C_{23}H_{41}O_6N_2NaSi_2$
	520.23958	-3.77	3.0	$C_{16}H_{37}O_8N_8NaSi$
	520.23556	3.96	-1.0	$C_{11}H_{37}O_{10}N_{10}NaSi$
	520.23551	4.05	2.0	$C_{18}H_{41}O_8N_4NaSi_2$
	520.23510	4.84	3.0	$C_{19}H_{37}O_{11}N_4Na$

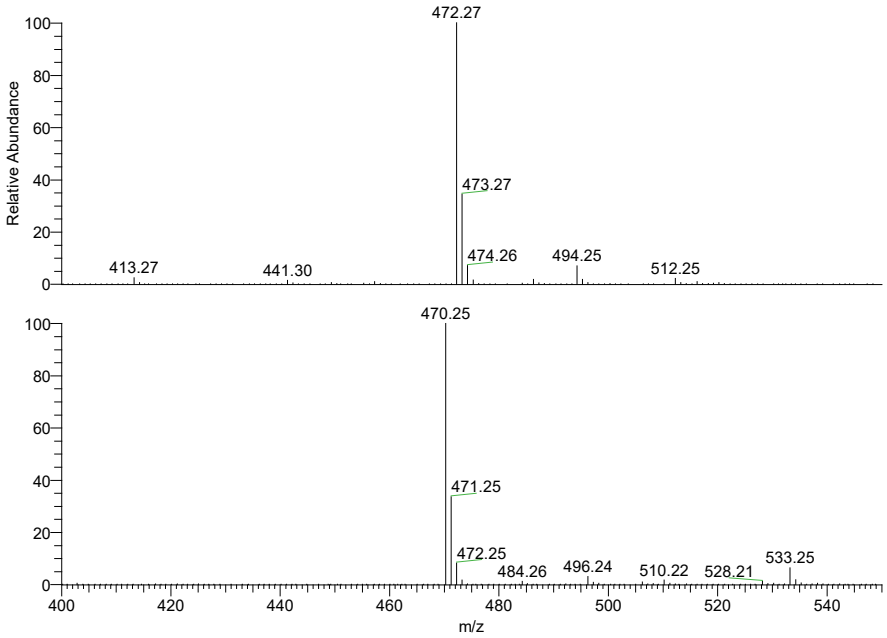


8

Sample No. : C:\Xcalibur\...\BG_212104_7_pU_NH2_pn
Operator name : Yamashita Nao
Date : 02/03/22 11:52:24
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

Instrument : Exactive Plus

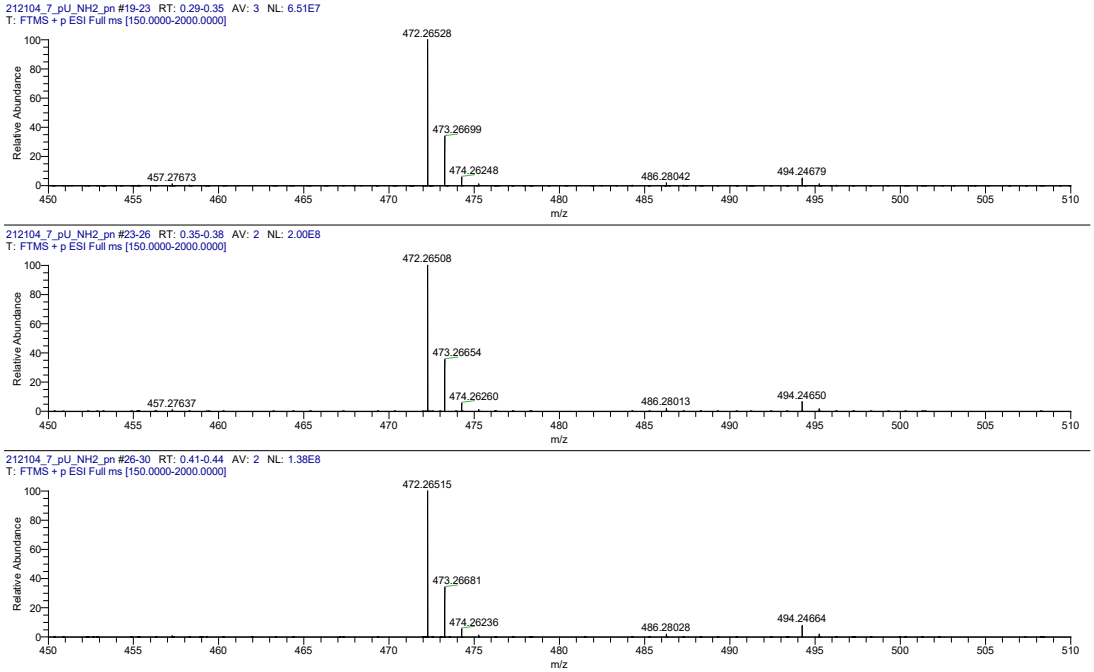
Mobile phase solvent : MeOH
Sample solvent : MeOH



Sample No. : C:\Xcalibur\...\0203\212104_7_pU_NH2_pn
Operator name : Yamashita Nao
Date : 02/03/22 11:45:03
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

Instrument : Exactive Plus

Mobile phase solvent : MeOH
Sample solvent : MeOH



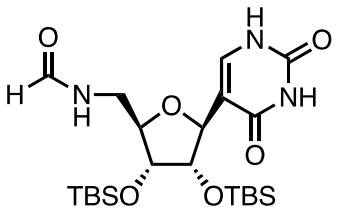
Elemental composition search on mass 472.27

m/z= 467.27-477.27

Isotope	Min	Max
N-14	0	5
O-16	0	10
C-12	0	100
H-1	0	200
Si-28	0	2

Charge 1
Mass tolerance 5.00 ppm
Nitrogen rule not used
RDB equiv -1.00-100.00
max results 20

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
472.26508	472.26529	-0.44	14.0	$C_{28}H_{36}ON_4Si$
	472.26534	-0.55	5.5	$C_{22}H_{38}O_8N_3$
	472.26575	-1.42	4.5	$C_{21}H_{42}O_5N_3Si_2$
	472.26395	2.39	9.0	$C_{27}H_{40}O_5Si$
	472.26663	-3.29	13.5	$C_{30}H_{38}O_2NSi$
	472.26349	3.36	18.5	$C_{34}H_{34}ON$
	472.26668	-3.40	5.0	$C_{24}H_{40}O_9$
	472.26307	4.25	0.0	$C_{18}H_{44}O_8N_2Si_2$
	472.26709	-4.26	4.0	$C_{23}H_{44}O_6Si_2$

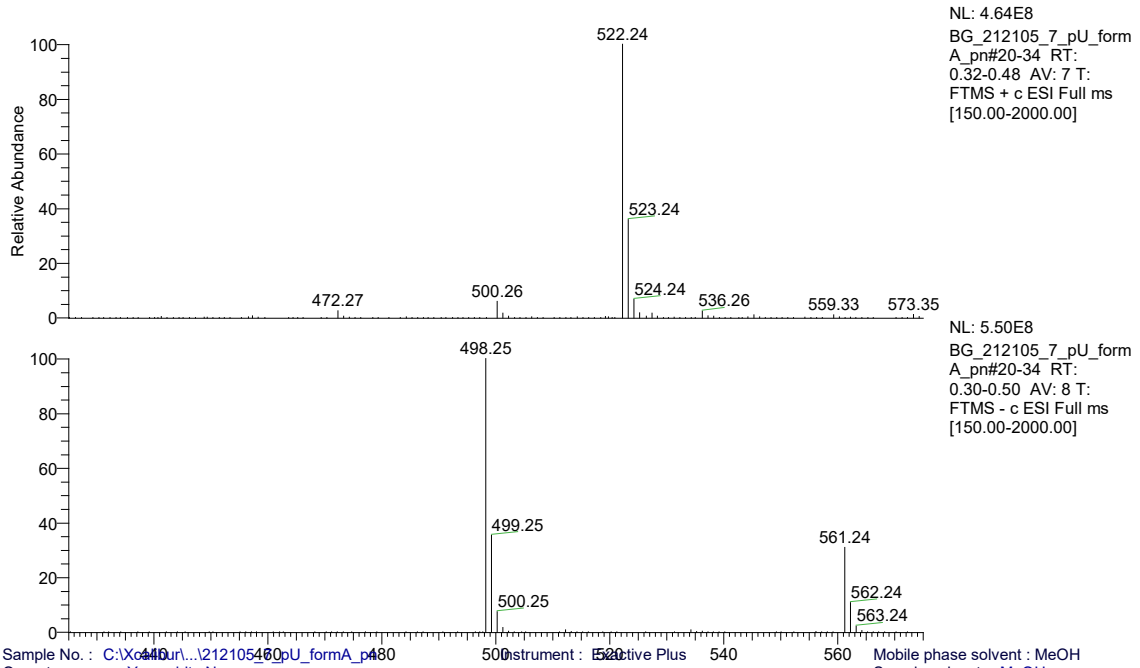


9

Sample No. : C:\Xcalibur\...\BG_212105_7_pU_formA_pn
Operator name : Yamashita Nao
Date : 02/03/22 13:12:41
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

Instrument : Exactive Plus

Mobile phase solvent : MeOH
Sample solvent : MeOH

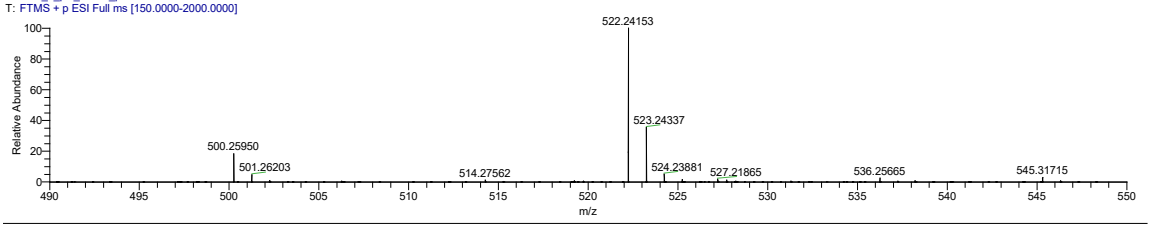


Sample No. : C:\Xcalibur\...\212105_7_pU_formA_pn
Operator name : Yamashita Nao
Date : 02/03/22 11:50:23
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

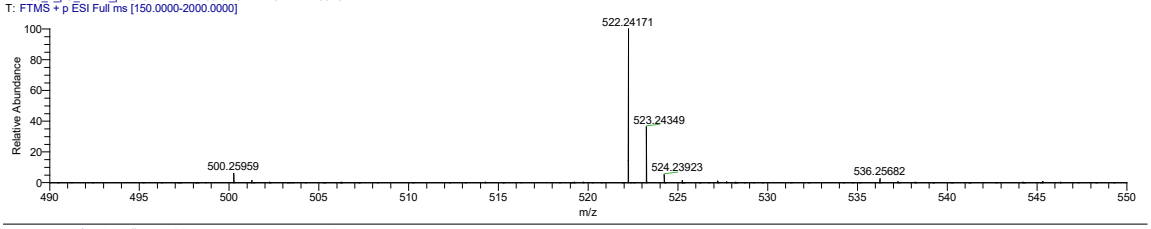
Instrument : Exactive Plus

Mobile phase solvent : MeOH
Sample solvent : MeOH

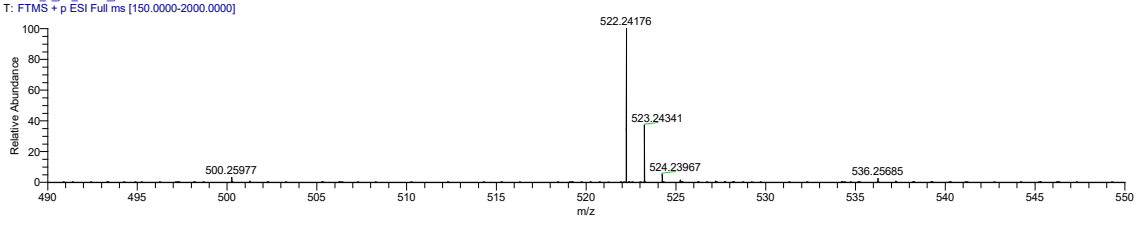
212105_7_pU_formA_pn #20-23 RT: 0.32-0.35 AV: 2 NL: 6.45E7
T: FTMS + p ESI Full ms [150.0000-2000.0000]



212105_7_pU_formA_pn #23-27 RT: 0.35-0.40 AV: 3 NL: 1.30E8
T: FTMS + p ESI Full ms [150.0000-2000.0000]



212105_7_pU_formA_pn #27-31 RT: 0.40-0.46 AV: 3 NL: 1.36E8
T: FTMS + p ESI Full ms [150.0000-2000.0000]



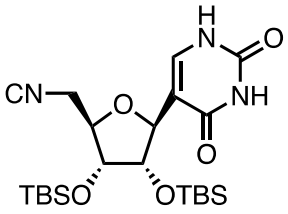
Elemental composition search on mass 522.24

m/z= 517.24-527.24

Isotope	Min	Max
N-14	0	5
O-16	0	10
C-12	0	100
H-1	0	200
Si-28	0	2
Na-23	0	1

Charge 1
Mass tolerance 5.00 ppm
Nitrogen rule not used
RDB equiv -1.00-100.00
max results 20

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
522.24171	522.24187	-0.31	13.5	$C_{28}H_{36}O_5N_3Si$
	522.24141	0.57	23.0	$C_{35}H_{30}ON_4$
	522.24215	-0.84	15.0	$C_{29}H_{35}O_2N_4NaSi$
	522.24220	-0.94	6.5	$C_{23}H_{37}O_9N_3Na$
	522.24234	-1.20	4.0	$C_{21}H_{42}O_9N_2Si_2$
	522.24099	1.37	4.5	$C_{19}H_{40}O_8N_5Si_2$
	522.24081	1.72	10.0	$C_{28}H_{39}O_6NaSi$
	522.24261	-1.72	5.5	$C_{22}H_{41}O_6N_3NaSi_2$
	522.24276	-2.00	22.5	$C_{37}H_{32}O_2N$
	522.24048	2.35	17.0	$C_{33}H_{38}O_2Si_2$
	522.24035	2.60	19.5	$C_{35}H_{33}O_2NNa$
	522.24322	-2.89	13.0	$C_{30}H_{38}O_6Si$
	522.24008	3.13	18.0	$C_{34}H_{34}O_5$
	522.23993	3.41	1.0	$C_{19}H_{43}O_9N_2NaSi_2$
	522.24349	-3.41	14.5	$C_{31}H_{37}O_3NNaSi$
	522.24354	-3.51	6.0	$C_{25}H_{39}O_{10}Na$
	522.23947	4.29	10.5	$C_{26}H_{37}O_5N_3NaSi$
	522.24395	-4.29	5.0	$C_{24}H_{43}O_7NaSi_2$
	522.23919	4.82	9.0	$C_{25}H_{38}O_8N_2Si$
	522.23914	4.92	17.5	$C_{31}H_{36}ON_3Si_2$

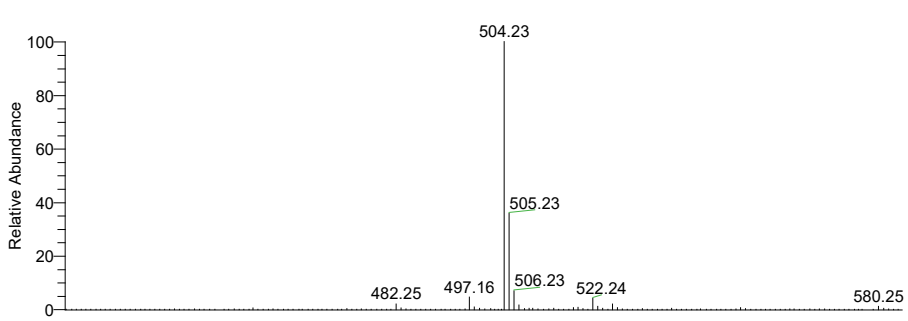


10

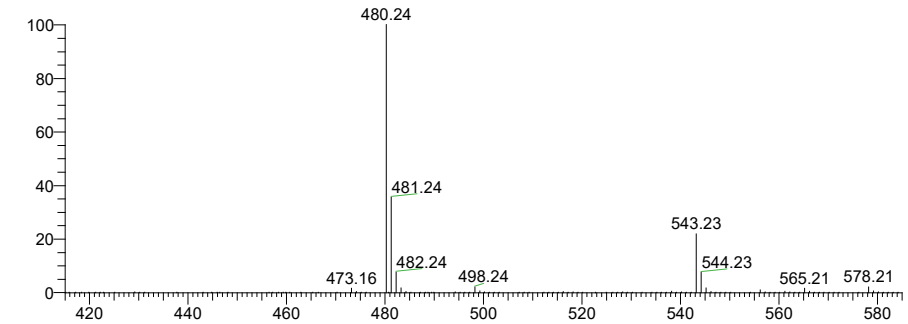
Sample No. : C:\Xcalibur\...\BG_212106_7_pU_isocy_pn
Operator name : Yamashita Nao
Date : 02/03/22 13:20:12
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

Instrument : Exactive Plus

Mobile phase solvent : MeOH
Sample solvent : MeOH



NL: 5.07E8
BG_212106_7_pU_isocy_pn#20-34 RT: 0.32-0.49 AV: 7 T: FTMS + c ESI Full ms [150.00-2000.00]



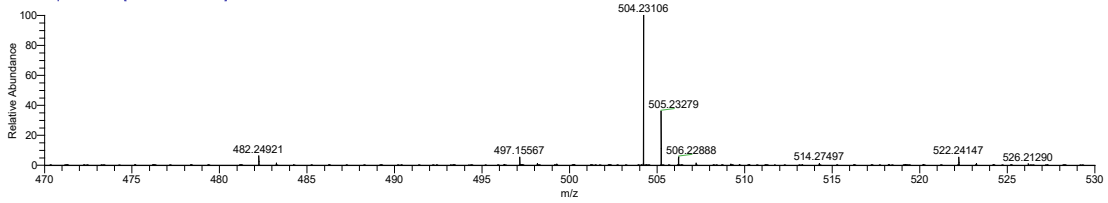
NL: 5.07E8
BG_212106_7_pU_isocy_pn#20-34 RT: 0.31-0.50 AV: 8 T: FTMS - c ESI Full ms [150.00-2000.00]

Sample No. : C:\Xcalibur\...\212106_7_pU_isocy_pn
Operator name : Yamashita Nao
Date : 02/03/22 11:55:09
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

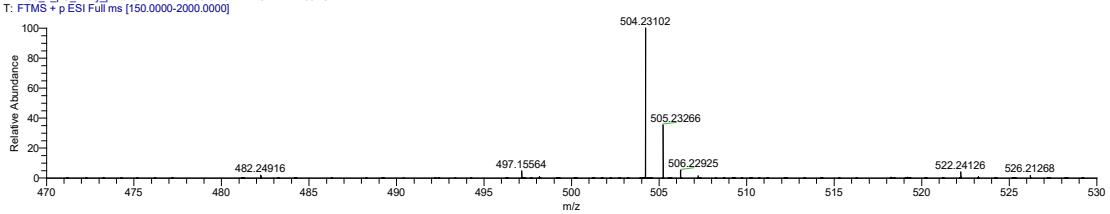
Instrument : Exactive Plus

Mobile phase solvent : MeOH
Sample solvent : MeOH

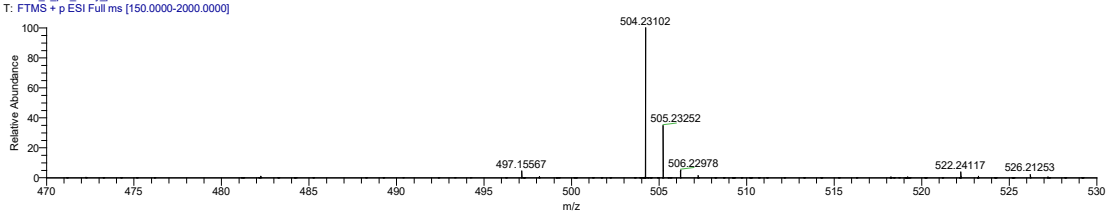
212106_7_pU_isocy_pn #20-23 RT: 0.32-0.35 AV: 2 NL: 7.36E7
T: FTMS + p ESI Full ms [150.0000-2000.0000]



212106_7_pU_isocy_pn #23-27 RT: 0.35-0.40 AV: 3 NL: 1.59E8
T: FTMS + p ESI Full ms [150.0000-2000.0000]



212106_7_pU_isocy_pn #27-30 RT: 0.40-0.43 AV: 2 NL: 1.71E8
T: FTMS + p ESI Full ms [150.0000-2000.0000]



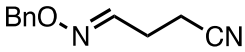
Elemental composition search on mass 504.23

m/z= 499.23-509.23

Isotope	Min	Max
N-14	0	5
O-16	0	10
C-12	0	100
H-1	0	200
Si-28	0	2
Na-23	0	1

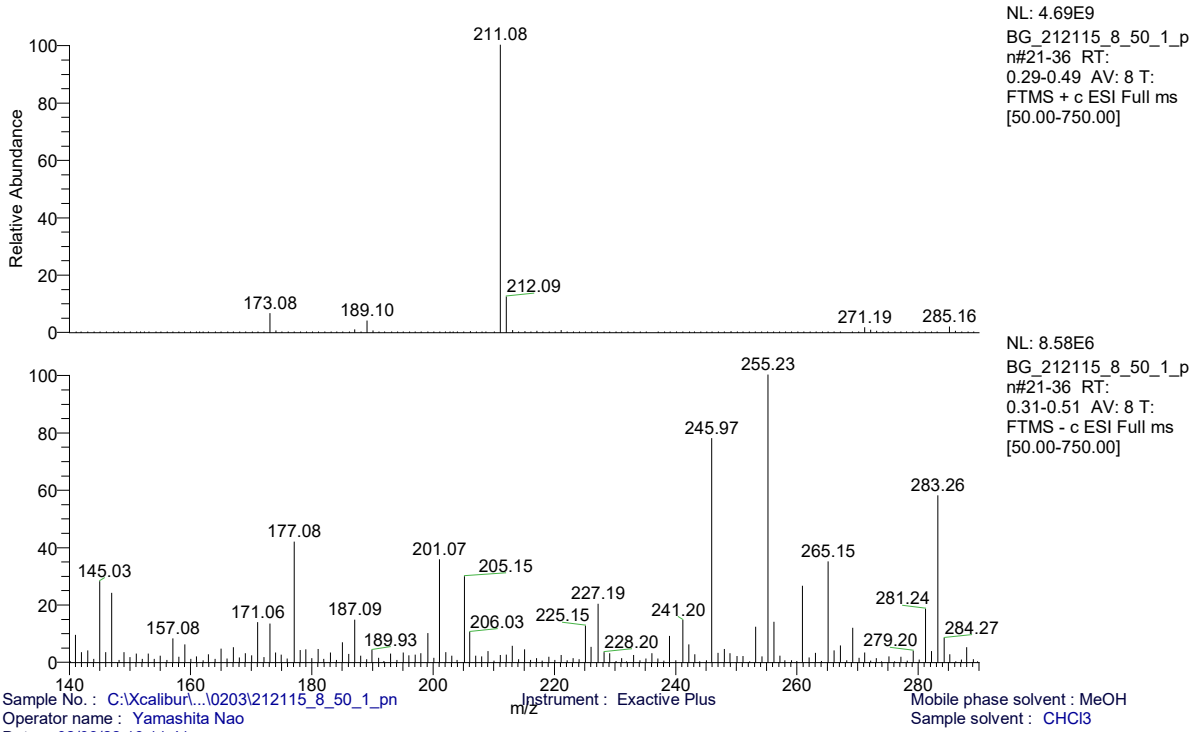
Charge 1
Mass tolerance 5.00 ppm
Nitrogen rule not used
RDB equiv -1.00-100.00
max results 20

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
504.23102	504.23085	0.34	24.0	$C_{35}H_{28}N_4$
	504.23131	-0.57	14.5	$C_{28}H_{34}O_4N_3Si$
	504.23158	-1.12	16.0	$C_{29}H_{33}ON_4NaSi$
	504.23043	1.17	5.5	$C_{19}H_{38}O_7N_5Si_2$
	504.23164	-1.22	7.5	$C_{23}H_{35}O_8N_3Na$
	504.23177	-1.49	5.0	$C_{21}H_{40}O_8N_2Si_2$
	504.23025	1.53	11.0	$C_{28}H_{37}O_5NaSi$
	504.23002	1.99	6.5	$C_{20}H_{34}O_{10}N_5$
	504.23205	-2.03	6.5	$C_{22}H_{39}O_5N_3NaSi_2$
	504.22992	2.18	18.0	$C_{33}H_{36}OSi_2$
	504.23219	-2.32	23.5	$C_{37}H_{30}ON$
	504.22979	2.45	20.5	$C_{35}H_{31}ONNa$
	504.22951	2.99	19.0	$C_{34}H_{32}O_4$
	504.23265	-3.24	14.0	$C_{30}H_{36}O_5Si$
	504.22937	3.28	2.0	$C_{19}H_{41}O_8N_2NaSi_2$
	504.23293	-3.78	15.5	$C_{31}H_{35}O_2NNaSi$
	504.23298	-3.88	7.0	$C_{25}H_{37}O_9Na$
	504.22890	4.20	11.5	$C_{26}H_{35}O_4N_3NaSi$
	504.23339	-4.70	6.0	$C_{24}H_{41}O_6NaSi_2$
	504.22863	4.74	10.0	$C_{25}H_{36}O_7N_2Si$

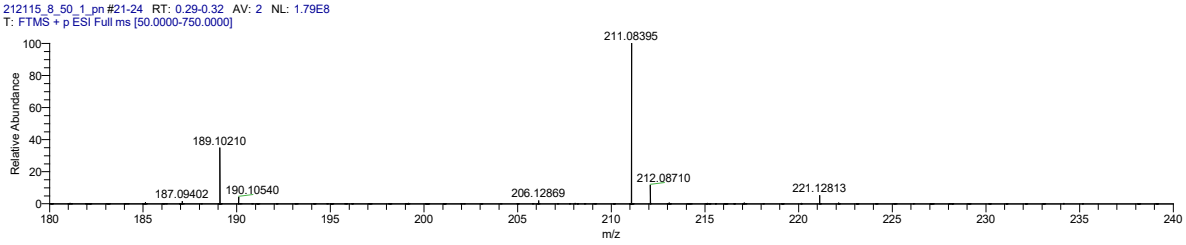


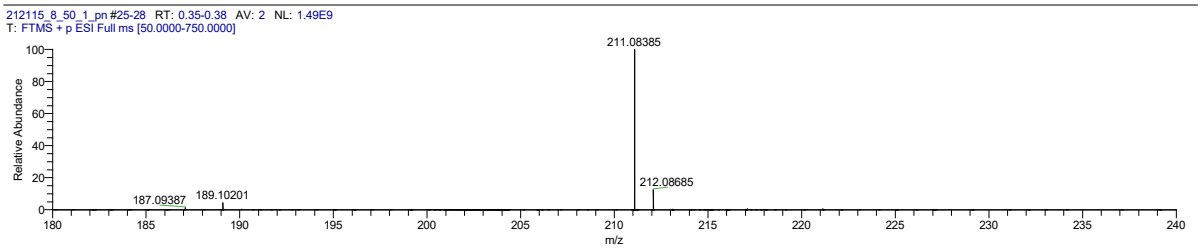
11

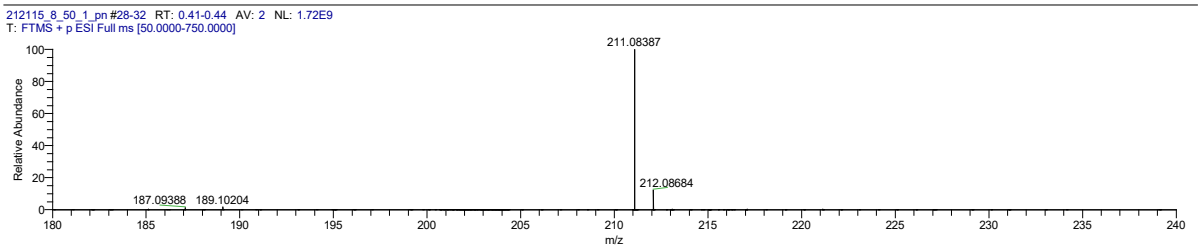
Sample No. : C:\Xcalibur\...\0203\BG_212115_8_50_1_pn
Operator name : Yamashita Nao
Date : 02/03/22 16:21:18
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz50_750pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University
Instrument : Exactive Plus
Mobile phase solvent : MeOH
Sample solvent : CHCl3



Sample No. : C:\Xcalibur\...\0203\212115_8_50_1_pn
Operator name : Yamashita Nao
Date : 02/03/22 16:11:41
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz50_750pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University
Instrument : Exactive Plus
Mobile phase solvent : MeOH
Sample solvent : CHCl3







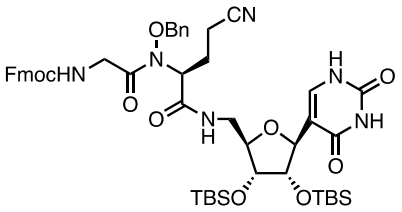
Elemental composition search on mass 211.08

m/z= 206.08-216.08

Isotope	Min	Max
N-14	0	10
O-16	0	10
C-12	0	100
H-1	0	200
Na-23	0	1

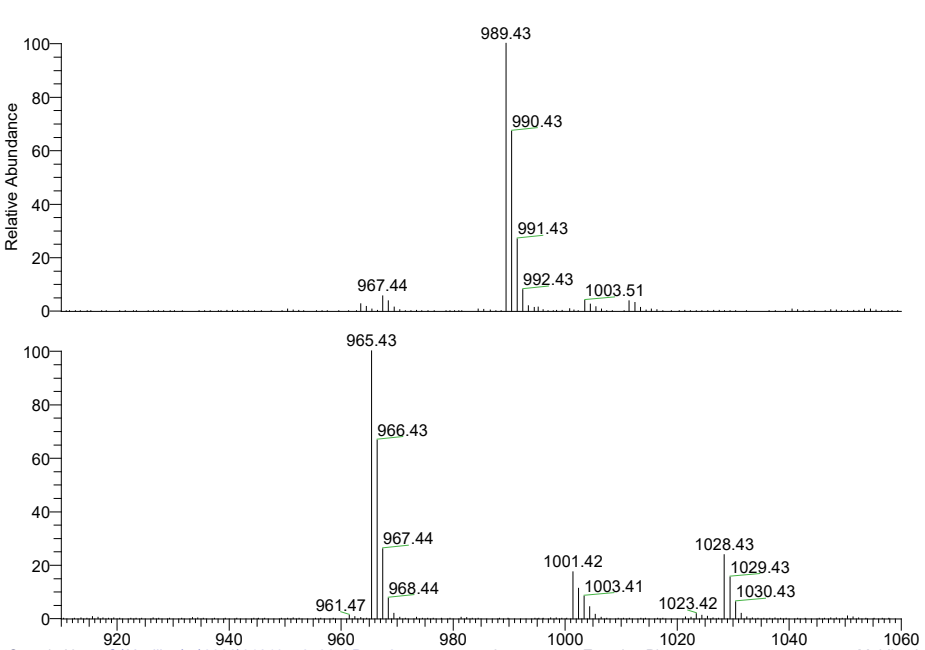
Charge 1
Mass tolerance 5.00 ppm
Nitrogen rule not used
RDB equiv -1.00-100.00
max results 50

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
211.08385	211.08391	-0.28	5.0	$C_{10}H_{13}O_4N$
	211.08418	-1.58	6.5	$C_{11}H_{12}ONNa$
	211.08284	4.78	7.0	$C_9H_{10}N_5Na$

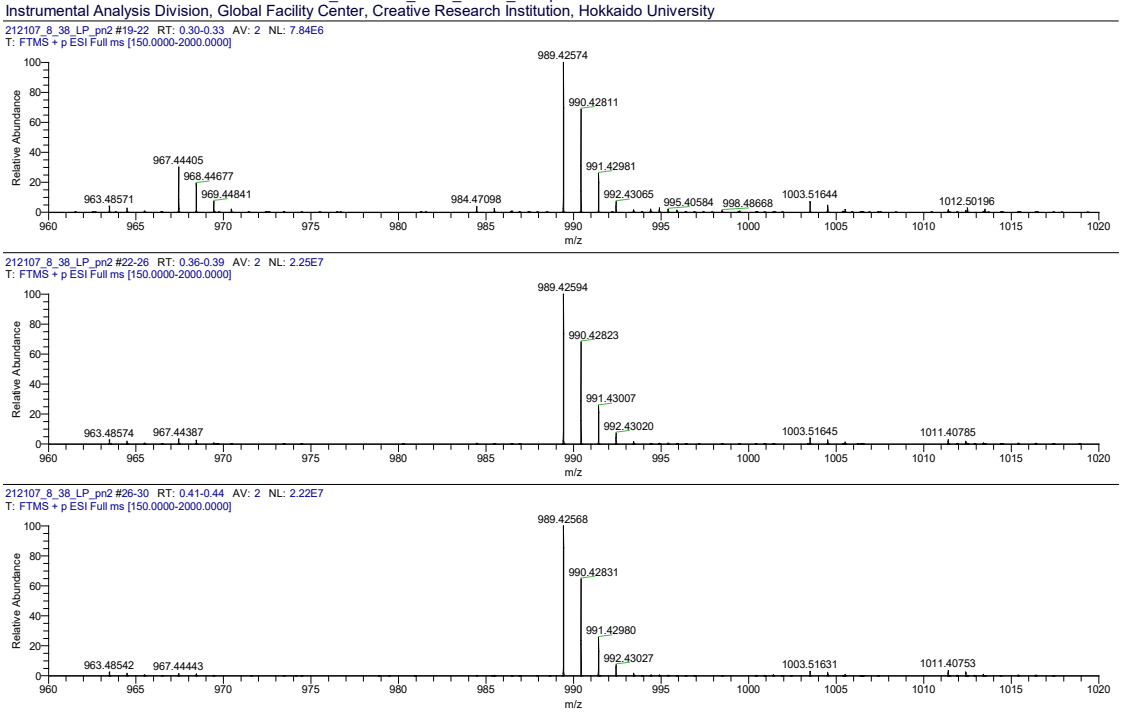


13

Sample No. : C:\Xcalibur\...\BG_212107_8_38_LP_pn2
Operator name : Yamashita Nao
Date : 02/03/22 14:28:09
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University
Instrument : Exactive Plus
Mobile phase solvent : MeOH
Sample solvent : CHCl3



Sample No. : C:\Xcalibur\...\0203\212107_8_38_LP_pn2
Operator name : Yamashita Nao
Date : 02/03/22 14:16:05
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University
Instrument : Exactive Plus
Mobile phase solvent : MeOH
Sample solvent : CHCl3



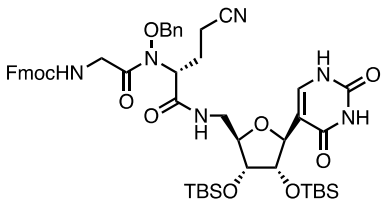
Elemental composition search on mass 989.43

m/z= 984.43-994.43

Isotope	Min	Max
N-14	0	6
O-16	0	10
C-12	0	100
H-1	0	200
Na-23	1	1
Si-28	0	2
S-32	0	0

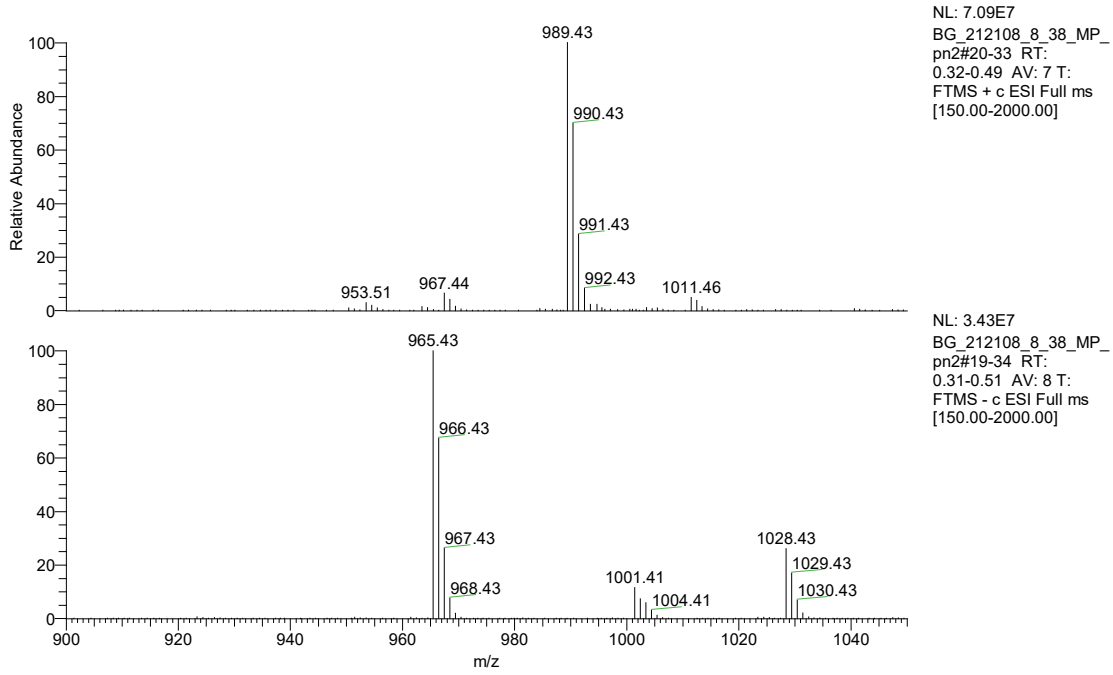
Charge 1
Mass tolerance 5.00 ppm
Nitrogen rule not used
RDB equiv -1.00-100.00
max results 100

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
989.42594	989.42615	-0.21	44.5	$C_{71}H_{58}N_2NaSi$
	989.42620	-0.26	36.0	$C_{65}H_{60}O_7NNa$
	989.42532	0.63	27.0	$C_{56}H_{64}O_{10}N_3NaSi$
	989.42661	-0.68	35.0	$C_{64}H_{64}O_4NNaSi_2$
	989.42527	0.68	35.5	$C_{62}H_{62}O_3N_4NaSi_2$
	989.42486	1.09	36.5	$C_{63}H_{58}O_6N_4Na$
	989.42712	-1.19	22.5	$C_{50}H_{66}O_{10}N_6NaSi_2$
	989.42754	-1.61	41.0	$C_{66}H_{56}O_3N_5Na$
	989.42398	1.99	27.5	$C_{54}H_{62}O_9N_6NaSi$
	989.42795	-2.03	40.0	$C_{65}H_{60}N_5NaSi_2$
	989.42393	2.03	30.5	$C_{61}H_{66}O_7NaSi_2$
	989.42800	-2.08	31.5	$C_{59}H_{62}O_7N_4NaSi$
	989.42352	2.45	31.5	$C_{62}H_{62}O_{10}Na$
	989.42347	2.50	40.0	$C_{68}H_{60}O_3NNaSi$
	989.42888	-2.97	40.5	$C_{68}H_{58}O_4N_2Na$
	989.42929	-3.38	39.5	$C_{67}H_{62}ON_2NaSi_2$
	989.42259	3.39	31.0	$C_{59}H_{64}O_6N_3NaSi_2$
	989.42934	-3.44	31.0	$C_{61}H_{64}O_8NNaSi$
	989.42218	3.80	32.0	$C_{60}H_{60}O_9N_3Na$
	989.42212	3.86	40.5	$C_{66}H_{58}O_2N_4NaSi$
	989.43022	-4.32	45.5	$C_{69}H_{54}N_6Na$
	989.42124	4.75	31.5	$C_{57}H_{62}O_5N_6NaSi_2$

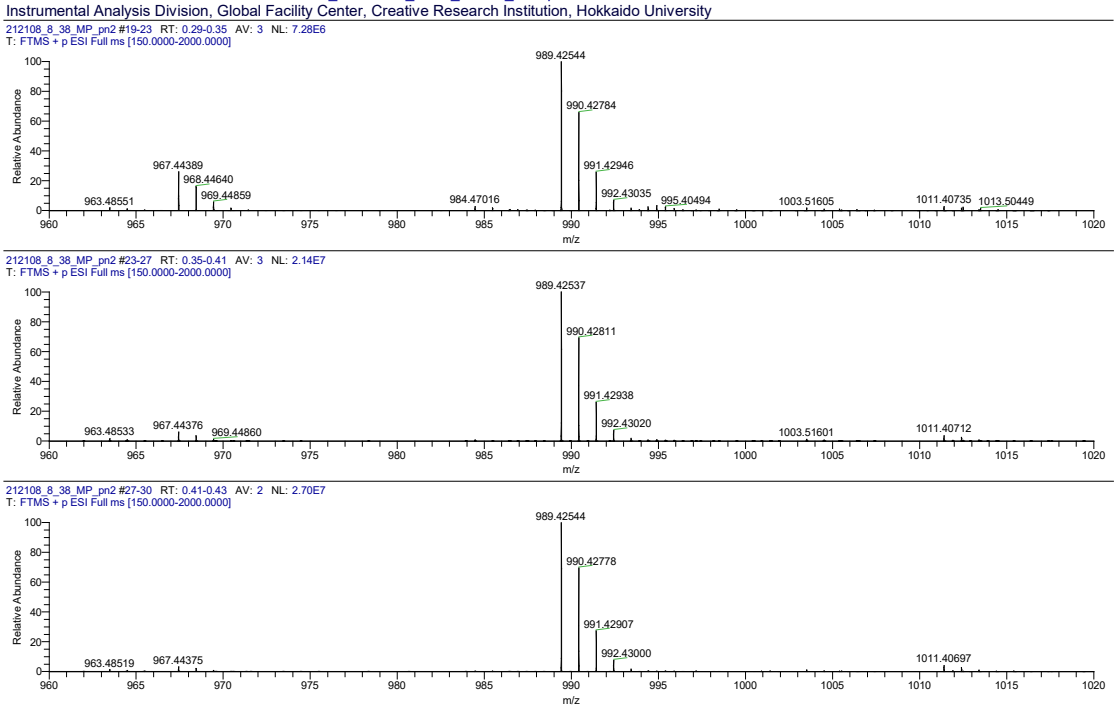


14

Sample No. : C:\Xcalibur\...\BG_212108_8_38_MP_pn2 Instrument : Exactive Plus Mobile phase solvent : MeOH
Operator name : Yamashita Nao Sample solvent : CHCl3
Date : 02/03/22 14:40:22
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University



Sample No. : C:\Xcalibur\...\0203\212108_8_38_MP_pn2 Instrument : Exactive Plus Mobile phase solvent : MeOH
Operator name : Yamashita Nao Sample solvent : CHCl3
Date : 02/03/22 14:30:23
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University



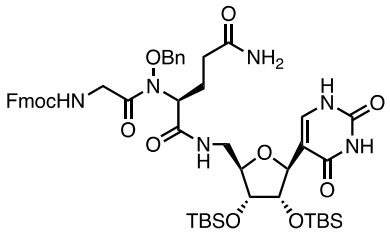
Elemental composition search on mass 989.43

m/z= 984.43-994.43

Isotope	Min	Max
N-14	0	6
O-16	0	10
C-12	0	100
H-1	0	200
Na-23	1	1
Si-28	0	2
S-32	0	0

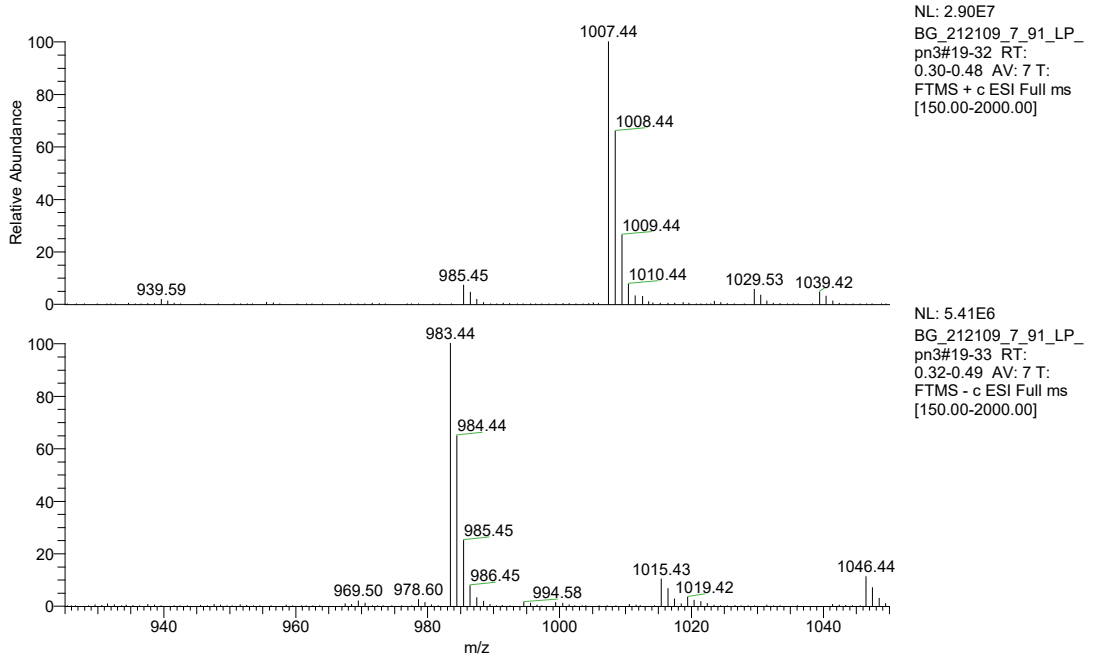
Charge 1
Mass tolerance 5.00 ppm
Nitrogen rule not used
RDB equiv -1.00-100.00
max results 100

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
989.42537	989.42532	0.05	27.0	$C_{56}H_{64}O_{10}N_3NaSi$
	989.42527	0.11	35.5	$C_{62}H_{62}O_3N_4NaSi_2$
	989.42486	0.52	36.5	$C_{63}H_{58}O_6N_4Na$
	989.42615	-0.79	44.5	$C_{71}H_{58}N_2NaSi$
	989.42620	-0.84	36.0	$C_{65}H_{60}O_7NNa$
	989.42661	-1.25	35.0	$C_{64}H_{64}O_4NNaSi_2$
	989.42398	1.41	27.5	$C_{54}H_{62}O_9N_6NaSi$
	989.42393	1.46	30.5	$C_{61}H_{66}O_7NaSi_2$
	989.42712	-1.76	22.5	$C_{50}H_{66}O_{10}N_6NaSi_2$
	989.42352	1.87	31.5	$C_{62}H_{62}O_{10}Na$
	989.42347	1.92	40.0	$C_{68}H_{60}O_3NNaSi$
	989.42754	-2.19	41.0	$C_{66}H_{56}O_3N_5Na$
	989.42795	-2.60	40.0	$C_{65}H_{60}N_5NaSi_2$
	989.42800	-2.66	31.5	$C_{59}H_{62}O_7N_4NaSi$
	989.42259	2.81	31.0	$C_{59}H_{64}O_6N_3NaSi_2$
	989.42218	3.23	32.0	$C_{60}H_{60}O_9N_3Na$
	989.42212	3.28	40.5	$C_{66}H_{58}O_2N_4NaSi$
	989.42888	-3.55	40.5	$C_{68}H_{58}O_4N_2Na$
	989.42929	-3.96	39.5	$C_{67}H_{62}ON_2NaSi_2$
	989.42934	-4.01	31.0	$C_{61}H_{64}O_8NNaSi$
	989.42124	4.17	31.5	$C_{57}H_{62}O_5N_6NaSi_2$
	989.42083	4.58	32.5	$C_{58}H_{58}O_8N_6Na$

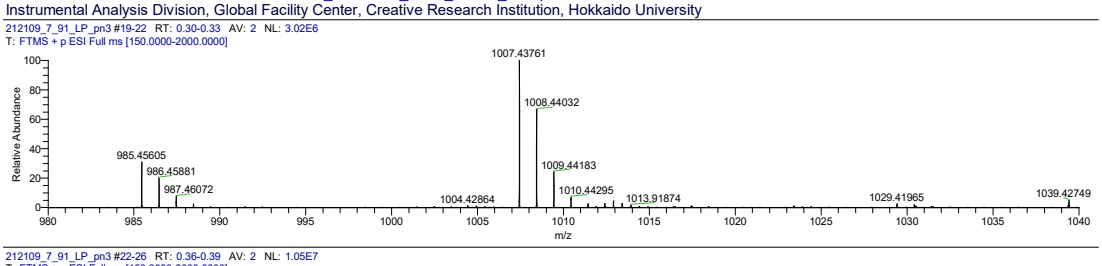


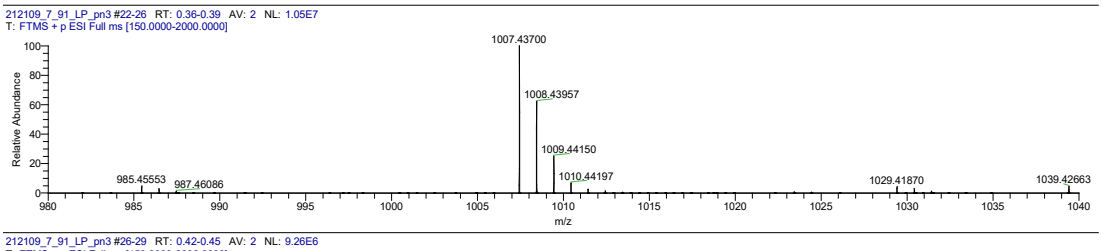
15

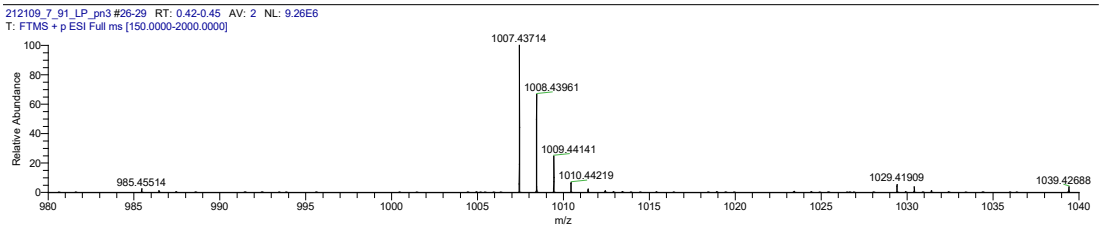
Sample No. : C:\Xcalibur\...\BG_212109_7_91_LP_pn3 Instrument : Exactive Plus Mobile phase solvent : MeOH
Operator name : Yamashita Nao Sample solvent : CHCl3
Date : 02/04/22 16:29:45
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University



Sample No. : C:\Xcalibur\...\0204\212109_7_91_LP_pn3 Instrument : Exactive Plus Mobile phase solvent : MeOH
Operator name : Yamashita Nao Sample solvent : CHCl3
Date : 02/04/22 16:25:59
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University







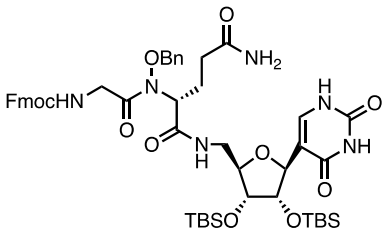
Elemental composition search on mass 1007.44

m/z= 1002.44-1012.44

Isotope	Min	Max
O-16	10	15
C-12	0	100
H-1	0	200
Na-23	0	1
Si-28	2	2
N-14	5	10

Charge 1
Mass tolerance 5.00 ppm
Nitrogen rule not used
RDB equiv -1.00-100.00
max results 10

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
1007.43700	1007.43741	-0.40	20.0	$C_{49}H_{69}O_{14}N_5Si_2$
	1007.43634	0.66	22.0	$C_{48}H_{66}O_{10}N_9NaSi_2$
	1007.43768	-0.68	21.5	$C_{50}H_{68}O_{11}N_6NaSi_2$
	1007.43606	0.93	20.5	$C_{47}H_{67}O_{13}N_8Si_2$
	1007.43874	-1.73	25.0	$C_{50}H_{65}O_{10}N_9Si_2$
	1007.43500	1.98	17.0	$C_{47}H_{70}O_{14}N_5NaSi_2$
	1007.44009	-3.06	24.5	$C_{52}H_{67}O_{11}N_6Si_2$
	1007.43366	3.32	17.5	$C_{45}H_{68}O_{13}N_8NaSi_2$
	1007.43204	4.92	16.5	$C_{42}H_{67}O_{15}N_{10}Si_2$

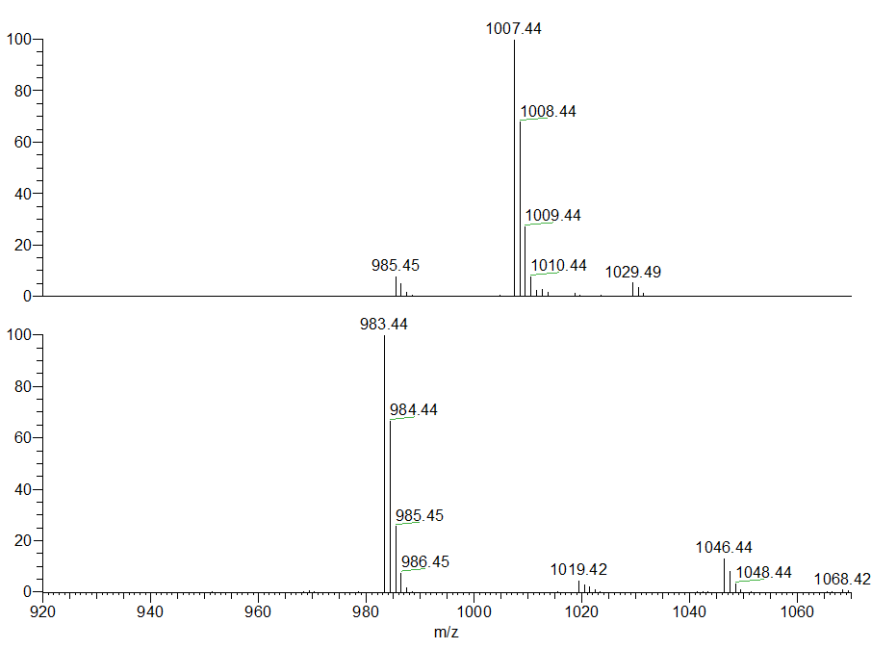


18

Sample No. : C:\Xcalibur\...\BG_212110_7_29_M _,
Operator name : Yamashita Nao
Date : 02/03/22 15:14:00
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

Instrument : Exactive Plus

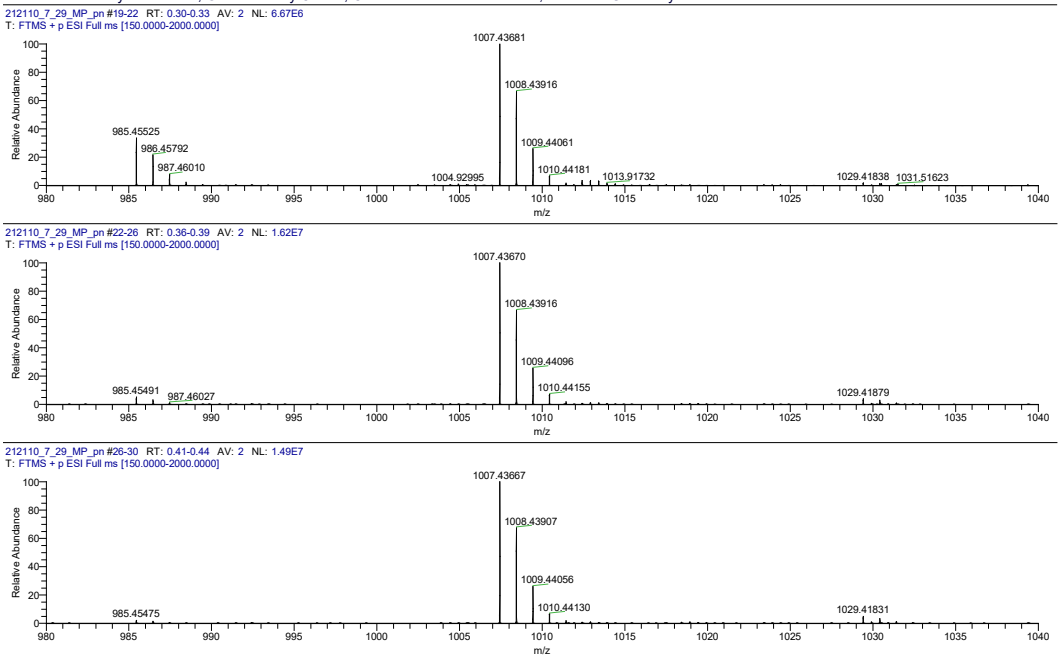
Mobile phase solvent : MeOH
Sample solvent : CHCl3



Sample No. : C:\Xcalibur\...\0203\212110_7_29_MP_pn
Operator name : Yamashita Nao
Date : 02/03/22 15:11:54
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

Instrument : Exactive Plus

Mobile phase solvent : MeOH
Sample solvent : CHCl3



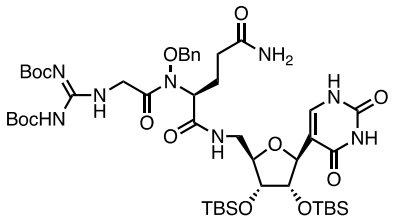
Elemental composition search on mass 1007.44

m/z= 1002.44-1012.44

Isotope	Min	Max
N-14	2	6
O-16	0	11
C-12	0	100
H-1	0	200
Na-23	1	1
Si-28	0	2
S-32	0	0

Charge 1
Mass tolerance 5.00 ppm
Nitrogen rule not used
RDB equiv -1.00-100.00
max results 100

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
1007.43670	1007.43671	-0.01	43.5	$C_{71}H_{60}ON_2NaSi$
	1007.43588	0.81	26.0	$C_{56}H_{66}O_{11}N_3NaSi$
	1007.43583	0.86	34.5	$C_{62}H_{64}O_4N_4NaSi_2$
	1007.43768	-0.97	21.5	$C_{50}H_{68}O_{11}N_6NaSi_2$
	1007.43542	1.27	35.5	$C_{63}H_{60}O_7N_4Na$
	1007.43537	1.32	44.0	$C_{69}H_{58}N_5NaSi$
	1007.43810	-1.39	40.0	$C_{66}H_{58}O_4N_5Na$
	1007.43851	-1.80	39.0	$C_{65}H_{62}ON_5NaSi_2$
	1007.43856	-1.85	30.5	$C_{59}H_{64}O_8N_4NaSi$
	1007.43454	2.14	26.5	$C_{54}H_{64}O_{10}N_6NaSi$
	1007.43944	-2.72	39.5	$C_{68}H_{60}O_5N_2Na$
	1007.43357	3.11	48.5	$C_{75}H_{56}N_2Na$
	1007.43985	-3.13	38.5	$C_{67}H_{64}O_2N_2NaSi_2$
	1007.43315	3.52	30.0	$C_{59}H_{66}O_7N_3NaSi_2$
	1007.43274	3.93	31.0	$C_{60}H_{62}O_{10}N_3Na$
	1007.43269	3.98	39.5	$C_{66}H_{60}O_3N_4NaSi$
	1007.44078	-4.05	44.5	$C_{69}H_{56}ON_6Na$
	1007.44124	-4.51	35.0	$C_{62}H_{62}O_5N_5NaSi$
	1007.43181	4.86	30.5	$C_{57}H_{64}O_6N_6NaSi_2$
	1007.44170	-4.97	25.5	$C_{55}H_{68}O_9N_4NaSi_2$

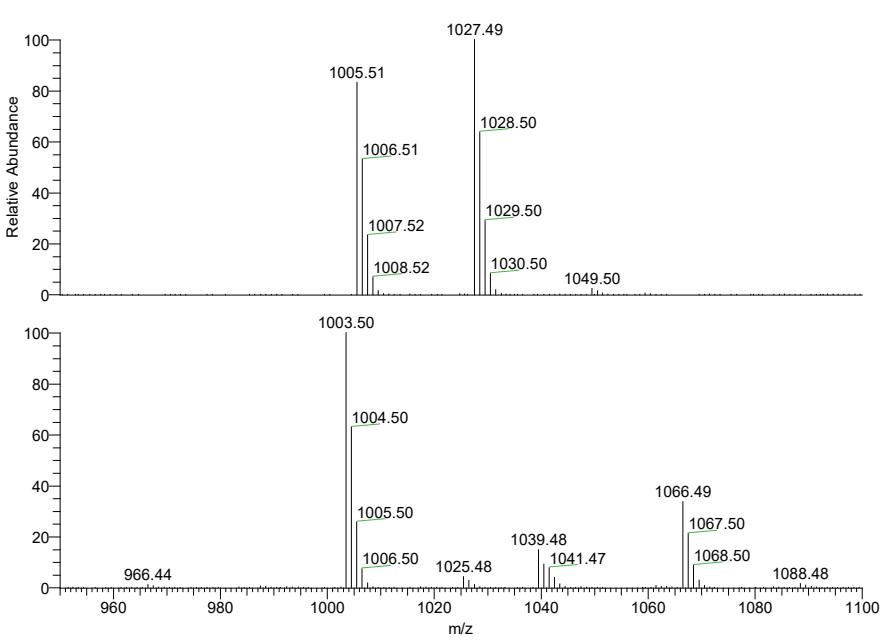


17

Sample No. : C:\Xcalibur\...\BG_212111_7_93_LP_pn
Operator name : Yamashita Nao
Date : 02/03/22 15:23:54
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz200_3000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

Instrument : Exactive Plus

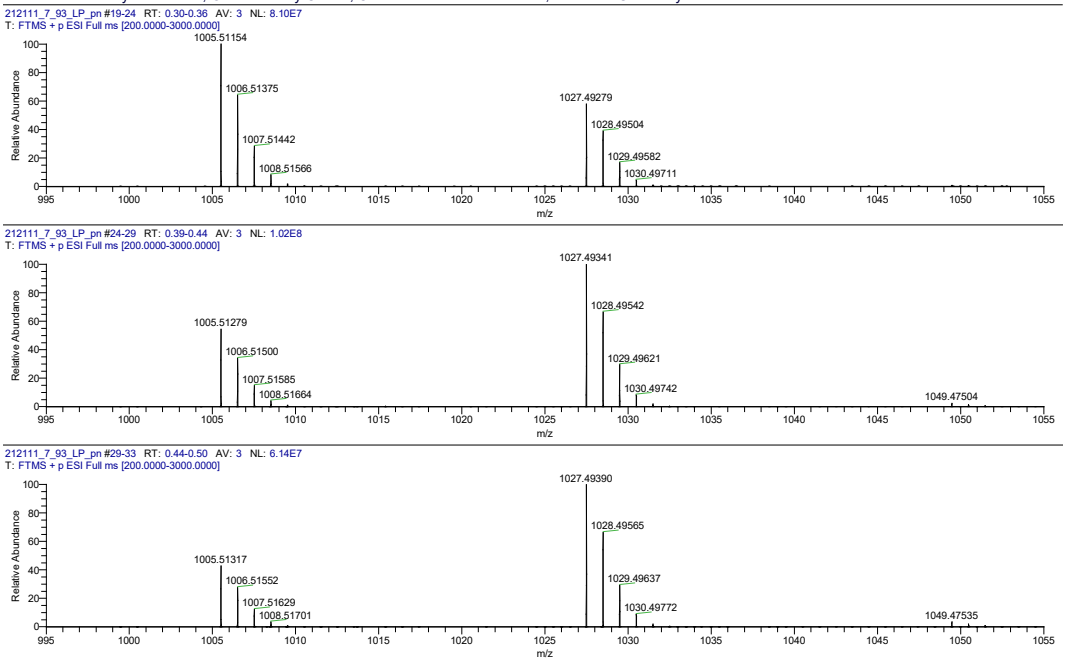
Mobile phase solvent : MeOH
Sample solvent : CHCl3



Sample No. : C:\Xcalibur\...\0203\212111_7_93_LP_pn
Operator name : Yamashita Nao
Date : 02/03/22 15:17:00
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz200_3000pn.meth
IInstrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

Instrument : Exactive Plus

Mobile phase solvent : MeOH
Sample solvent : CHCl3



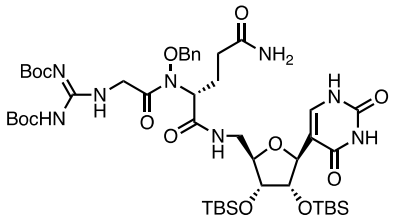
Elemental composition search on mass 1027.49

m/z= 1022.49-1032.49

Isotope	Min	Max
N-14	0	10
O-16	5	15
C-12	0	100
H-1	0	200
Si-28	2	2
Na-23	1	1

Charge 1
Mass tolerance 5.00 ppm
Nitrogen rule not used
RDB equiv -1.00-100.00
max results 50

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
1027.49341	1027.49307	0.33	22.5	$C_{57}H_{76}O_{10}N_2NaSi_2$
	1027.49307	0.34	28.0	$C_{56}H_{70}O_5N_9NaSi_2$
	1027.49441	-0.97	27.5	$C_{58}H_{72}O_6N_6NaSi_2$
	1027.49224	1.14	10.5	$C_{41}H_{76}O_{15}N_{10}NaSi_2$
	1027.49173	1.64	23.0	$C_{55}H_{74}O_9N_5NaSi_2$
	1027.49575	-2.28	27.0	$C_{60}H_{74}O_7N_3NaSi_2$
	1027.49626	-2.77	14.5	$C_{46}H_{76}O_{13}N_8NaSi_2$
	1027.49039	2.94	18.0	$C_{54}H_{78}O_{13}NNaSi_2$
	1027.49039	2.94	23.5	$C_{53}H_{72}O_8N_8NaSi_2$
	1027.49709	-3.58	26.5	$C_{62}H_{76}O_8NaSi_2$
	1027.49760	-4.08	14.0	$C_{48}H_{78}O_{14}N_5NaSi_2$
	1027.48905	4.25	18.5	$C_{52}H_{76}O_{12}N_4NaSi_2$

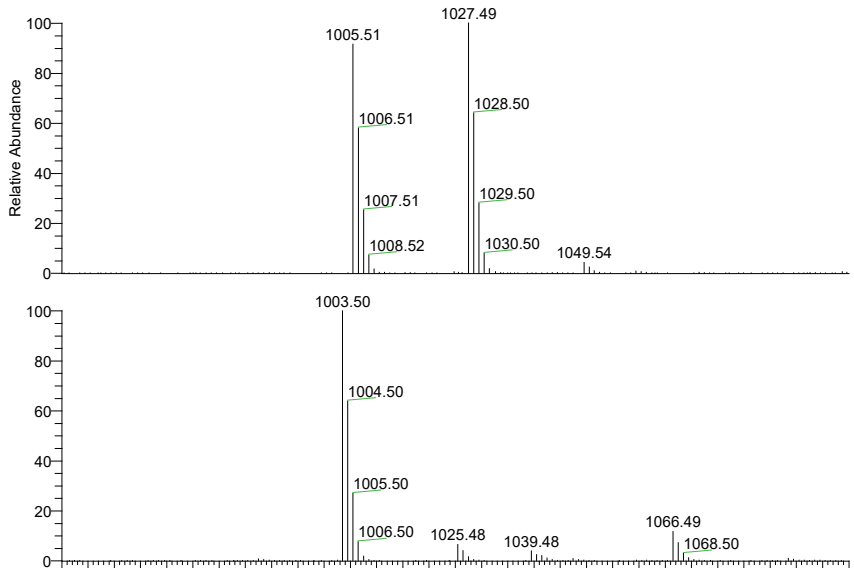


21

Sample No. : C:\Xcalibur\...\BG_212112_7_94_MP_pn
Operator name : Yamashita Nao
Date : 02/03/22 15:37:55
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz200_3000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

Instrument : Exactive Plus

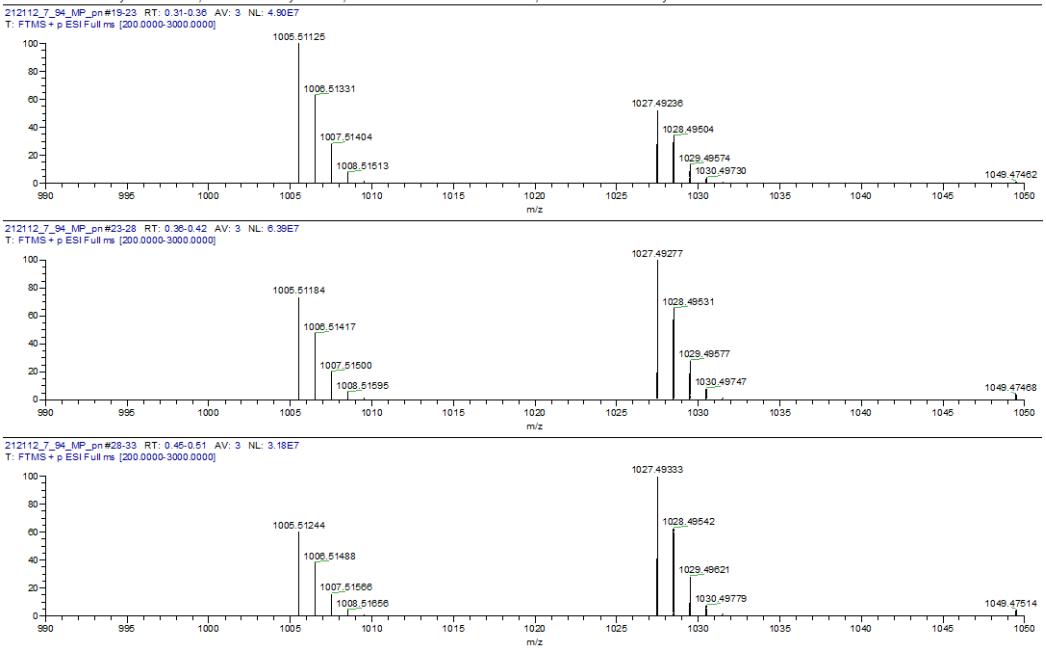
Mobile phase solvent : MeOH
Sample solvent : CHCl3



Sample No. : C:\Xcalibur\...\0203\212112_7_94_MP_pn
Operator name : Yamashita Nao
Date : 02/03/22 15:21:46
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz200_3000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

Instrument : Exactive Plus

Mobile phase solvent : MeOH
Sample solvent : CHCl3



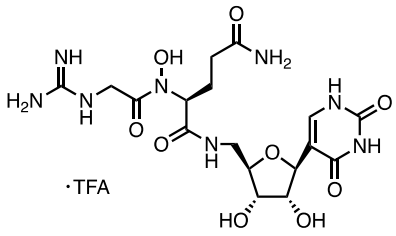
Elemental composition search on mass 1027.49

m/z= 1022.49-1032.49

Isotope	Min	Max
N-14	0	10
O-16	5	15
C-12	0	100
H-1	0	200
Si-28	2	2
Na-23	1	1

Charge 1
Mass tolerance 5.00 ppm
Nitrogen rule not used
RDB equiv -1.00-100.00
max results 50

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
1027.49277	1027.49307	-0.29	28.0	$C_{56}H_{70}O_5N_9NaSi_2$
	1027.49307	-0.29	22.5	$C_{57}H_{76}O_{10}N_2NaSi_2$
	1027.49224	0.52	10.5	$C_{41}H_{76}O_{15}N_{10}NaSi_2$
	1027.49173	1.01	23.0	$C_{55}H_{74}O_9N_5NaSi_2$
	1027.49441	-1.59	27.5	$C_{58}H_{72}O_6N_6NaSi_2$
	1027.49039	2.32	18.0	$C_{54}H_{78}O_{13}NNaSi_2$
	1027.49039	2.32	23.5	$C_{53}H_{72}O_8N_8NaSi_2$
	1027.49575	-2.90	27.0	$C_{60}H_{74}O_7N_3NaSi_2$
	1027.49626	-3.40	14.5	$C_{46}H_{76}O_{13}N_8NaSi_2$
	1027.48905	3.62	18.5	$C_{52}H_{76}O_{12}N_4NaSi_2$
	1027.49709	-4.21	26.5	$C_{62}H_{76}O_8NaSi_2$
	1027.49760	-4.70	14.0	$C_{48}H_{78}O_{14}N_5NaSi_2$
	1027.48771	4.93	19.0	$C_{50}H_{74}O_{11}N_7NaSi_2$

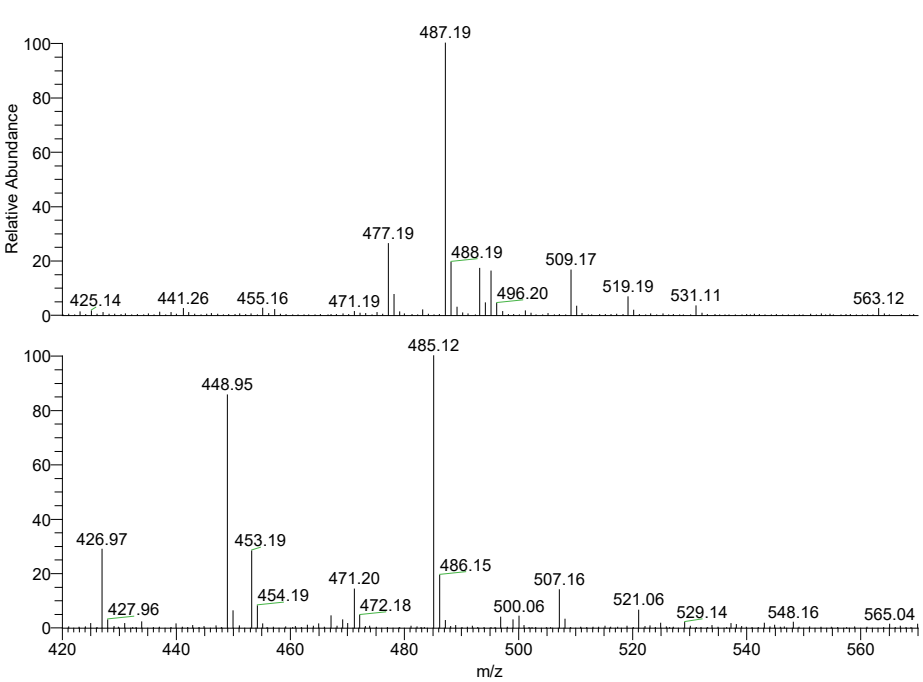


1

Sample No. : C:\Xcalibur\...\BG_212113_8_60_LP_pn
Operator name : Yamashita Nao
Date : 02/03/22 16:03:40
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

Instrument : Exactive Plus

Mobile phase solvent : MeOH
Sample solvent : MeOH

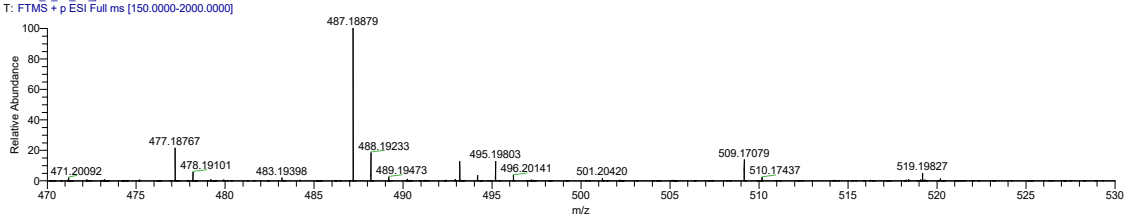


Sample No. : C:\Xcalibur\...\0203\212113_8_60_LP_pn
Operator name : Yamashita Nao
Date : 02/03/22 15:57:48
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

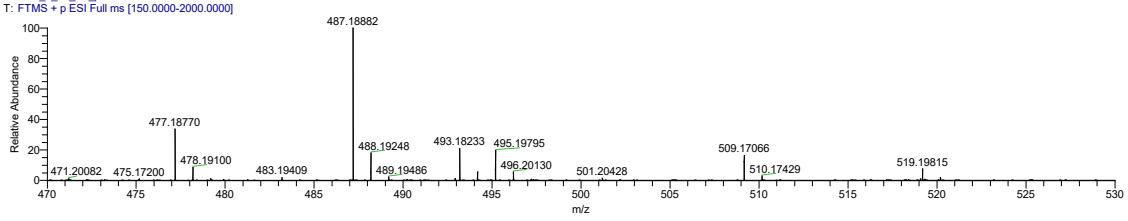
Instrument : Exactive Plus

Mobile phase solvent : MeOH
Sample solvent : MeOH

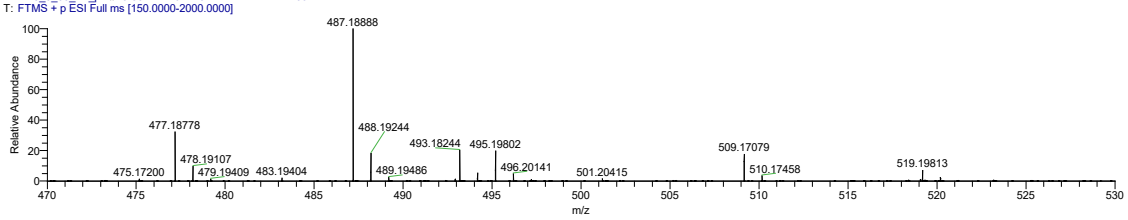
212113_8_60_LP_pn #19-23 RT: 0.30-0.36 AV: 3 NL: 7.24E6
T: FTMS + p ESI Full ms [150.0000-2000.0000]



212113_8_60_LP_pn #23-26 RT: 0.36-0.38 AV: 2 NL: 1.30E7
T: FTMS + p ESI Full ms [150.0000-2000.0000]



212113_8_60_LP_pn #26-30 RT: 0.41-0.44 AV: 2 NL: 1.09E7
T: FTMS + p ESI Full ms [150.0000-2000.0000]



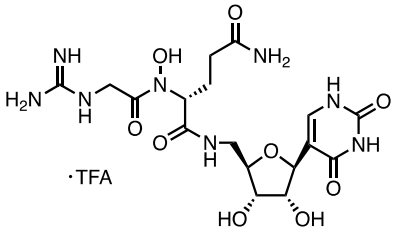
Elemental composition search on mass 487.19

m/z= 482.19-492.19

Isotope	Min	Max
N-14	0	10
O-16	0	10
C-12	0	100
H-1	0	200

Charge 1
Mass tolerance 5.00 ppm
Nitrogen rule not used
RDB equiv -1.00-100.00
max results 50

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
487.18882	487.18904	-0.46	21.0	$C_{31}H_{25}O_3N_3$
	487.18955	-1.50	8.5	$C_{17}H_{27}O_9N_8$
	487.18770	2.30	21.5	$C_{29}H_{23}O_2N_6$
	487.19039	-3.21	20.5	$C_{33}H_{27}O_4$
	487.19089	-4.26	8.0	$C_{19}H_{29}O_{10}N_5$



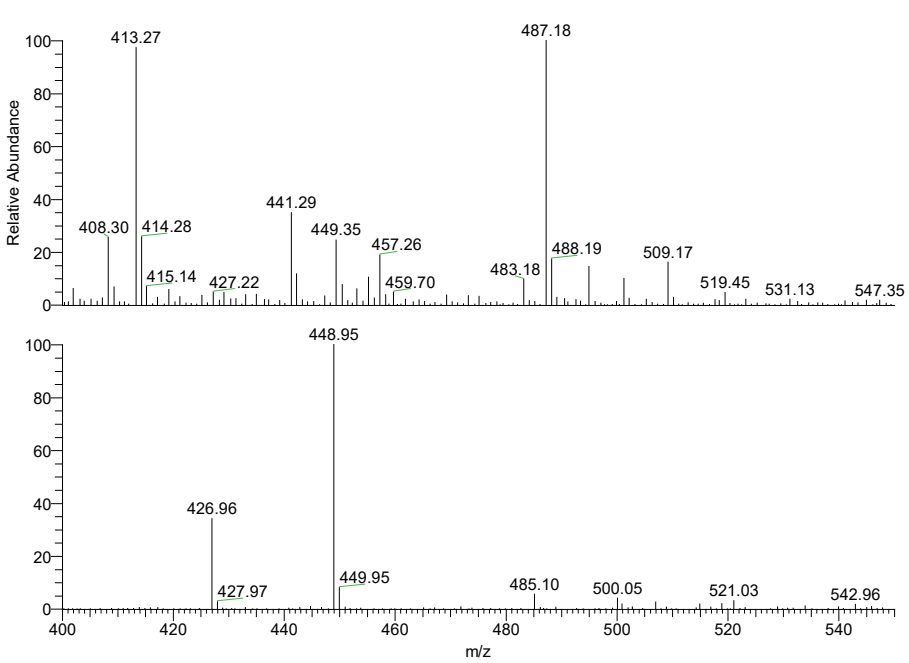
·TFA

epi-1

Sample No. : C:\Xcalibur\...\BG_212114_8_65_MP_pn
Operator name : Yamashita Nao
Date : 02/03/22 16:13:36
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

Instrument : Exactive Plus

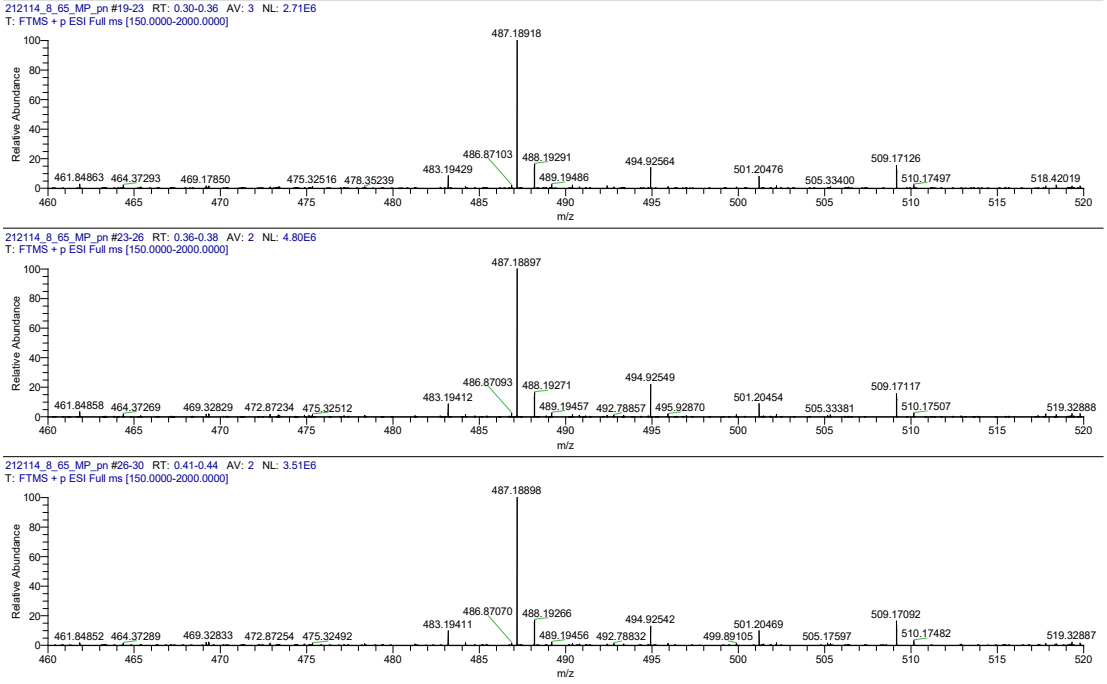
Mobile phase solvent : MeOH
Sample solvent : MeOH



Sample No. : C:\Xcalibur\...\0203\212114_8_65_MP_pn
Operator name : Yamashita Nao
Date : 02/03/22 16:02:36
Instrumental method : C:\Xcalibur\methods\ESI_100ul\S60_100ul_mz150_2000pn.meth
Instrumental Analysis Division, Global Facility Center, Creative Research Institution, Hokkaido University

Instrument : Exactive Plus

Mobile phase solvent : MeOH
Sample solvent : MeOH



Elemental composition search on mass 487.19

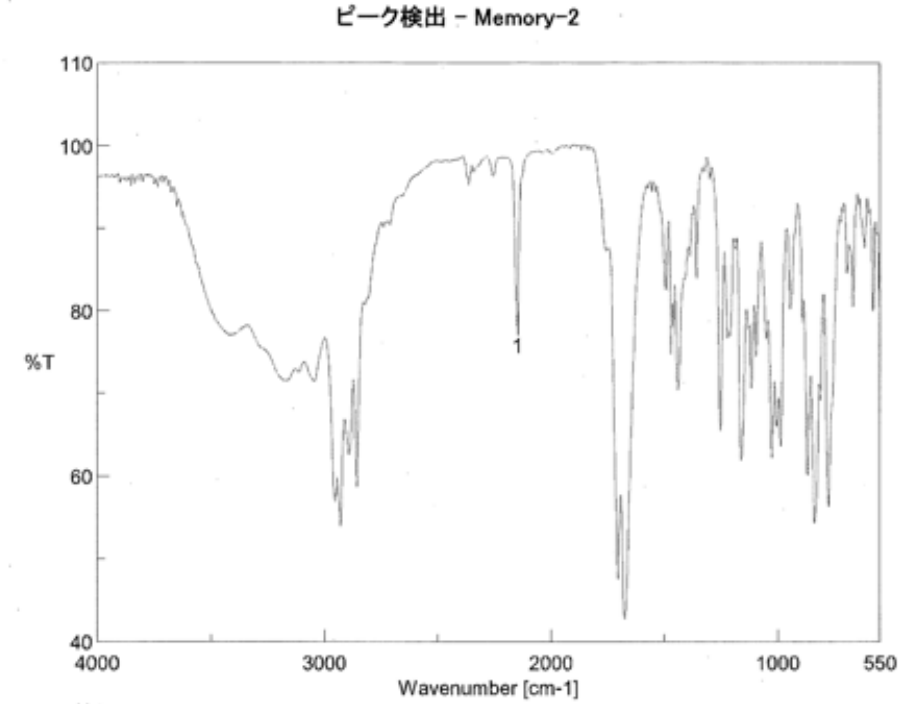
m/z= 482.19-492.19

Isotope	Min	Max
N-14	0	10
O-16	0	10
C-12	0	100
H-1	0	200

Charge 1
Mass tolerance 5.00 ppm
Nitrogen rule not used
RDB equiv -1.00-100.00
max results 50

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
487.18897	487.18904	-0.15	21.0	$C_{31}H_{25}O_3N_3$
	487.18955	-1.19	8.5	$C_{17}H_{27}O_9N_8$
	487.18770	2.61	21.5	$C_{29}H_{23}O_2N_6$
	487.19039	-2.91	20.5	$C_{33}H_{27}O_4$
	487.19089	-3.95	8.0	$C_{19}H_{29}O_{10}N_5$

8) The IR spectrum of isocyanide (10)



[コメント情報]
試料名 17OKW-pseudoU-isocyanide
コメント NaCl
測定者 Ryotaro Okawa
所属
会社 北海道大学

[データ情報]
作成日時 2022/01/28 15:17
更新日時 2022/01/28 15:25

データタイプ 等間隔データ
横軸 Wavenumber [cm-1]
縦軸 %T
スタート 549.613 cm-1
エンド 4000.6 cm-1
データ間隔 0.964233 cm-1
データ数 3580

[測定情報]
機種名 FT/IR-4100typeA
シリアル番号 B066661016

測定日時 2022/01/28 15:11

光源 標準光源
検出器 TGS
積算回数 100
分解 4 cm-1
ゼロフィリング On
アポダイゼーション Cosine
ゲイン Auto (4)
アパーチャー Auto (7.1 mm)
スキャンスピード Auto (2 mm/sec)
フィルタ Auto (30000 Hz)

[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	2146.38	78.4548			