Electronic Supplementary Information for

A gold-complex initiated functionalization of biologically active polyphenols applied to ^{18}F-labeled chemical probe

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General Information

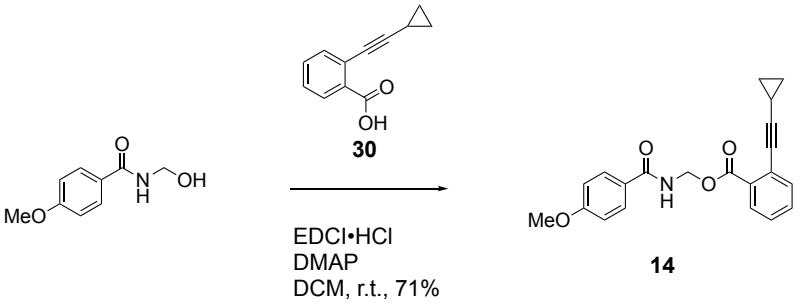
NMR spectra were recorded on a JEOL Model ECP-400 (400 MHz for ^{1}H, 400 MHz for ^{13}C, 376 MHz for ^{19}F) and Bruker AVANCE III HD 400 (400 MHz for ^{1}H, 400 MHz for ^{13}C, 376 MHz for ^{19}F) instrument in the indicated solvent. Chemical shifts are reported in units of parts per million (ppm) relative to the signal for internal tetramethylsilane (0 ppm for ^{1}H) for solutions in $CDCl_3$. ^{1}H NMR spectral data are reported as follows: $CDCl_3$ (7.26 ppm). ^{13}C NMR spectral data are reported as follows: $CDCl_3$ (77.16 ppm). ^{19}FNMR spectral are reported as follows: $C_6H_5CF_3$ (-63.72 ppm) as an external standard. Multiplicities are reported by using the following abbreviations: s, singlet; br-s, broaded-singlet; d, doublet; br-d, broaded-doublet; dd, doublet of doublets; br-dd, broaded-doublet of doublets; t, triplet; dq, doublet of quartets; q, quartet, m, multiplet; and, *J*, coupling constants in Hertz.

IR spectra were recorded on a Perkin Elmer Spectrum One FT-IR spectrophotometer or JASCO FT/IR-4200 spectrophotometer. Only the strongest and/or structurally important absorption is reported as the IR data in cm^{-1}.

All reactions were monitored by thin-layer chromatography carried out on 0.2 mm E. Merck silica gel plates (60F-254) with UV light, visualized by p-anisaldehyde solution, ceric sulfate or ethanolic phosphomolybdic acid. Column chromatography separations were performed using silica gel (Merck silica gel 60, 0.063 – 0.200 mm).

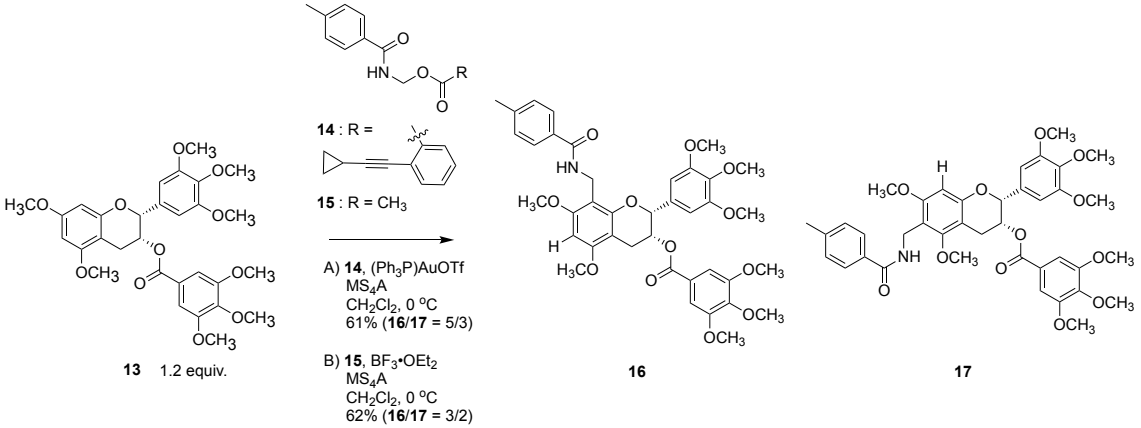
Gel permeation chromatography (GPC) for quantitative analysis were performed on a Japan Analytical Industry Model LC 605 (recycling preparative HPLC), with a Japan Analytical Industry Model RI-5 refractive index detector and a Japan Analytical Industry Model 301 ultra violet detector with polystyrene gel column (JAIGEL1H, 20 mm x 600 mm) using chloroform as a solvent (3.50 mL/min).

(4-methoxybenzamido)methyl 2-(cyclopropylethynyl)benzoate (14)



To a stirred solution of *N*-(hydroxymethyl)-4-methoxybenzamide (50.0 mg, 276 μmol) and 2-(cyclopropylethynyl)benzoic acid (**30**) (103 mg, 552 μmol) in dry DCM (2.76 mL) were added DMAP (67.4 mg, 552 μmol) and EDCI•HCl (159 mg, 828 μmol) at 0 ˚C. After being stirred at room temperature for 30 min, the reaction mixture was poured into NH_4Cl aq. and EtOAc at 0 ˚C. The aqueous layer was extracted with ethyl acetate twice. The combined extract was washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel with hexane/ethyl acetate (1/1) to give **14** (70.5 mg, 202 μmol, 71%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, 1H, *J* = 7.2 Hz), 7.79 (d, 2H, *J* = 8.8 Hz), 7.47 (d, 1H, *J* = 6.8 Hz), 7.43 ~ 7.37 (m, 2H), 7.28 (d, 1H, *J* = 7.6 Hz), 6.93 (d, 2H, *J* = 8.8 Hz), 5.70 (d, 2H, *J* = 7.6 Hz), 3.85 (s, 3H, 1.55–1.48 (m, 1H), 0.926 ~ 0.867 (m, 4H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 166.4, 166.1, 162.1, 133.6, 131.4, 130.0, 128.6, 126.4, 124.9, 124.3, 113.2, 99.2, 73.7, 64.6, 54.8, 8.34; FT-IR (neat) 3335, 2229, 1717, 1654, 1606, 1540, 1505, 1255, 1243, 1181, 1057, 1037, 930, 846, 757 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{21}H_{19}NO_4$ 372.12118$[M+Na]^+$, found 372.12140.

An electrophilic aromatic substitution of *O*-octamethyl EGCG



A gold-catalyzed reaction

To MS4A (53.8 mg, 2.00 g/mmol) heated by heat gun for 15 min was added **14** (9.60 mg, 26.9 μmol)

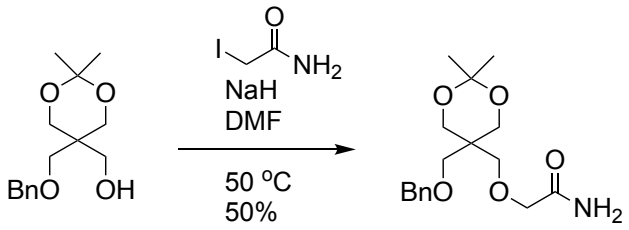
and (2*R*,3*R*)-5,7-dimethoxy-2-(3,4,5-trimethoxyphenyl)chroman-3-yl 3,4,5-trimethoxybenzoate (**13**) (18.4 mg, 32.3 μmol) in DCM (0.50 mL) at 0 ˚C. After being stirred at room temperature for 1 h, (PPh_3)AuOTf (26.9 μL, 26.9 μmol, 1.00 mmol/mL in DCM)) was added to the reaction mixture at 0 ˚C. After being stirred at 0 ºC for 10 min, the reaction mixture was filtered through celite, washed by DCM, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel with hexane/ethyl acetate (1/4)) and further purified by a gel permeation chromatography (GPC) to give mixture of (2*R*,3*R*)-5,7-dimethoxy-8-((4-methoxybenzamido)methyl)-2-(3,4,5-trimethoxyphenyl)chroman-3-yl 3,4,5-trimethoxybenzoate (**16**) and (2*R*,3*R*)-5,7-dimethoxy-6-((4-methoxybenzamido)methyl)-2-(3,4,5-trimethoxyphenyl)chroman-3-yl 3,4,5-trimethoxybenzoate (**17**) (12.1 mg, 19.7 μmol, 61%). Then ratio of the mixture was decided by 1H NMR which showed **16**/**17** = 5/3.

An acid-catalyzed reaction

To a stirred solution of (4-methoxybenzamide)methyl acetate (100 mg, 448 μmol) and **13** (307 mg, 538 μmol) in dry DCM (4.50 mL) were dropped distilled BF_3OEt_2 (56.3 μL, 63.5 mg, 1.13 g/mL, 448 μmol) at –40 ºC. Then warmed up to -20 ºC. After reacting at -20 ºC for 10 min, the reaction mixture was quenched by Et_3N (62.4μL, 45.3 mg, 0.726 g/mL, 448 μmol) and poured into $NaHCO_3$ aq. and ethyl acetate at 0 ℃. The aqueous layer was extracted with ethyl acetate twice. The combined extract was washed with brine, dried over $MgSO_4$, filtered, and concentrated in vacuo. The residue was short passed by column chromatography on silica gel with hexane/ethyl acetate (1/4) and further purified by Gel Permeation Chromatography (GPC) to give mixture of **16**/**17** (203 mg, 277 μmol, 62%). Then ratio of the mixture was decided by ^{1}H NMR which showed **16**/**17** = 3/2. Then further purification was done by column chromatography on silica gel with hexane/ethyl acetate (3/7) to give pure **16** and **17**. **16**:$[\alpha]_D^{21}$ -60.7 (*c* 1.32, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, 2H, *J* = 8.8 Hz), 7.08 (s, 2H), 6.84 (d, 2H, *J* = 8.8 Hz), 6.81 (s, 2H), 6.60 (t, 1H, *J* = 5.4 Hz), 6.18 (s, 1H), 5.70 (br, 1H), 5.17 (s, 1H), 5.04, 4.56 (dd, 2H, *J* = 6.6, 13.8 Hz), 3.91 (s, 3H), 3.85 (s, 3H), 3.83 (s, 3H), 3.82 (s, 3H), 3.81 (s, 3H); 3.78 (s, 6H), 3.64 (s, 6H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 165.8, 165.3, 161.9, 158.1, 157.8, 153.2, 153.1, 152.8, 142.3, 137.6, 133.3, 128.5, 127.2, 124.9, 113.6, 106.8, 106.1, 103.3, 100.4, 88.2, 77.5, 77.2, 68.4, 60.8, 56.1, 56.0, 55.9, 55.8, 55.4, 55.3, 32.8, 25.9; FT-IR (neat) 3000, 2940, 2838, 1714, 1593, 1503, 1460, 1416, 1359, 1332, 1252, 1224, 1127, 765 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{39}H_{43}NO_{13}$ 756.26321 [M+Na] +, found 756.26306; **17**: $[\alpha]_D^{21}$ -55.6 (*c* 1.00 , $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, 2H, *J* = 6.8 Hz), 7.14 (s, 2H), 6.89 (d, 2H, *J* = 6.8 Hz), 6.70 (s, 2H), 6.61 (t, 1H, *J* = 5.6 Hz), 6.47 (s, 1H), 5.68 (br, 1H), 5.13 (s, 1H), 4.80, 4.56 (dd, 2H, *J* = 6.0, 14.0 Hz), 3.81 (s, 3H), 3.85 (s, 3H), 3.84 (s, 3H), 3.82 (s, 3H), 3.80 (s, 9H), 3.73 (s, 6H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 166.3, 165.1, 162.0, 158.0,

155.2, 153.2, 152.9, 142.7, 138.0, 133.0, 128.6, 127.2, 124.8, 113.6, 112.7, 107.3, 104.9, 103.9, 95.9, 77.9, 77.2, 68.4, 61.5, 60.9, 60.8, 56.3, 56.0, 55.9, 55.3, 33.6, 26.2; FT-IR (neat) 2940, 2838, 1715, 1644, 1606, 1591, 1542, 1456, 1417, 1252, 1220, 1127, 772 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{39}H_{43}NO_{13}$ 756.26321 [M+Na] +, found 756.26271.

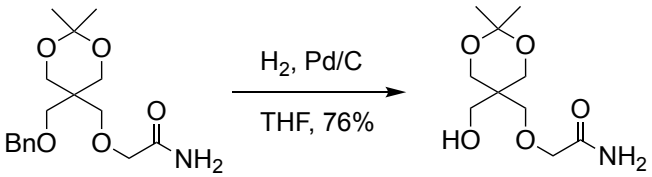
2-((5-((benzyloxy)methyl)-2,2-dimethyl-1,3-dioxan-5-yl)methoxy)acetamide (25)



24 **25**

To a stirred solution of sodium hydride (51.0 mg, 1.28 mmol) in dry DMF (1.0 mL) was added methyl (2,2-Dimethyl-5-(benzyloxymethyl)-1,3-dioxane-5-yl)methanol (**24**) (170 mg, 638 μmol) in dry DMF (2.0 mL) at 0 ˚C. After the reaction mixture was stirred at room temperature for 1h, 2-iodoacetamide (177 mg, 957 μmol) was added to reaction mixture. After being stirred at room temperature for 12 h, the reaction mixture was poured into water and EtOAc at 0 ˚C. The aqueous layer was extracted with ethyl acetate twice. The combined extract was washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo*. evaporated. The residue was purified by column chromatography on silica gel with hexane/ethyl acetate (1/4) to give **25** (103 mg, 320 μmol, 50%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28-7.38 (m, 5H), 6.77 (br, 1H), 5.41 (br, 1H), 4.51 (s, 2H), 3.94 (s, 2H), 3.70-3.80 (m, 4H), 3.60 (s, 2H), 3.47 (s, 2H), 1.40 (d, 6H, J = 6.8 Hz); ^{13}C NMR (400 MHz, $CDCl_3$) δ 172.8, 137.7, 128.5, 127.9, 127.7, 98.5, 73.6, 70.9, 70.3, 69.9, 62.9, 39.0, 24.8, 22.5; FT-IR (neat) 2992, 2870, 1697, 1684, 1455, 1373, 1200, 1119, 1085, 1029, 829, 771, 740, 699(cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{17}H_{25}NO_5$ 346.16304 $[M+Na]^+$, found 346.16244.

2-((5-(hydroxymethyl)-2,2-dimethyl-1,3-dioxan-5-yl)methoxy)acetamide (26)

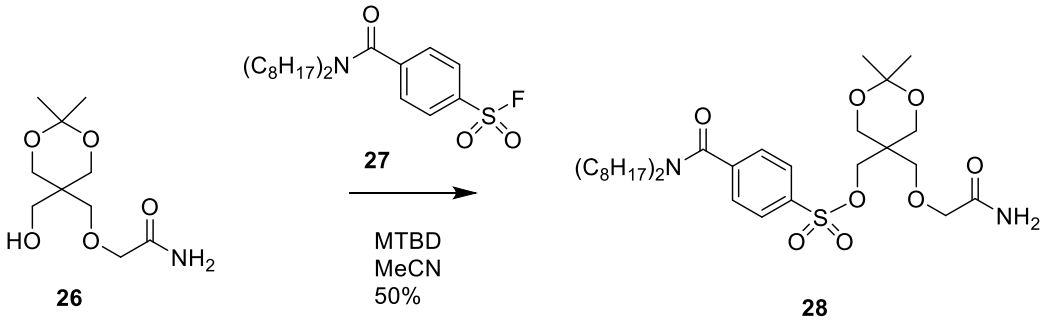


25 **26**

To a stirred solution of Pd/C (6.93 g, 0.30 g/mmol, 10%) in THF (348 mL) was added **25** (7.48 g, 23.1 mmol) at 0 °C. Then H_2 gas was added to reaction mixture. After being stirred at room temperature for 2 h, the reaction mixture was filtered through celite, washed by ethyl acetate, and then evaporated. The residue was recrystallized from hexane/EtOAc to give **26** (4.13 g, 17.7 mmol,

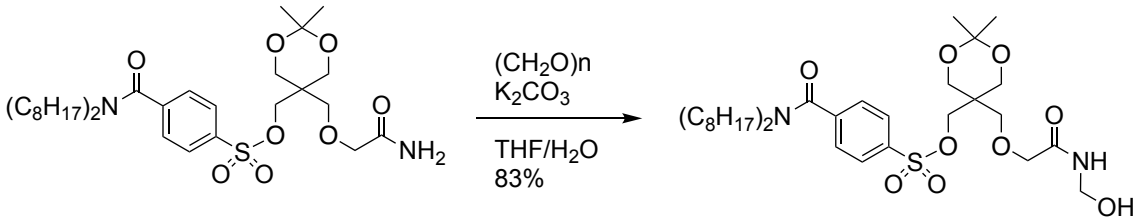
76%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.78 (br, 1H), 5.50 (br, 1H), 4.01 (s, 2H), 3.74 (s, 4H), 3.69 (s, 2H), 3.63 (s, 2H), 3.48 (s, 2H), 1.42 (d, 6H, J = 6.4 Hz);^{13}C NMR (400 MHz, $CDCl_3$) δ 172.6, 98.6, 71.5, 70.2, 63.2, 62.7, 39.3, 24.7, 22.7; FT-IR (neat) 3420, 2992, 2940, 2879, 1682, 1385, 1200, 1153, 1123, 1082, 1048, 828 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{10}H_{19}NO_5$ 256.11609 $[M+Na]^+$, found 256.11573.

(5-((2-amino-2-oxoethoxy)methyl)-2,2-dimethyl-1,3-dioxan-5-yl)methyl 4-(didodecylcarbamoyl)benzenesulfonate (28)



To a stirred solution of **26** (20.6 mg, 88.3 μmol) in dry MeCN (0.88 mL) was added 7-Methyl-1,5,7-triazabicyclo[4.4.0]dec-5-ene (MTBD) (27.1 mg, 25.5 μL, 177 μmol, 1.06 g/mL) and 4-(didodecylcarbamoyl)benzenesulfonyl fluoride (**27**) (57.2 mg, 106 μmol) at 0 °C. After being stirred at room temperature for 2.0 h, the reaction mixture was poured into water and EtOAc at 0 °C. The aqueous layer was extracted with ethyl acetate twice. The combined extract was washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel with hexane/ethyl acetate (1/3, 0/1) to give **28** (33.3 mg, 44.1 μmol, 50%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, 2H, J = 8.4 Hz), 7.53 (d, 2H, J = 8.0 Hz), 6.34(br, 1H), 5.80 (br, 1H), 4.13 (s, 2H), 3.88 (s, 2H), 3.64-3.71 (m, 4H), 3.46-3.50 (m, 4H), 3.10-3.14 (m, 2), 1.73, 1.66 (m, 4H), 1.50 (d, 6H J = 7.2 Hz),1.27 (s, 36), 0.881(t, 6H, J = 6.4 Hz); ^{13}C NMR (400 MHz, $CDCl_3$) δ 171.6, 169.4, 143.1, 135.6, 128.3, 127.4, 98.9, 70.8, 69.9, 69.3, 62.0, 49.0, 44.9, 38.6, 31.9, 29.6, 29.4, 29.3, 29.1, 27.4, 26.6, 24.1, 22.8, 22.7, 14.1; FT-IR (neat) 3465, 2923, 2853, 1686, 1629, 1428, 1369, 1187, 1089, 971, 831, 612(cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{41}H_{72}N_2O_8S$ 775.49071 $[M+Na]^+$, found 775.49179.

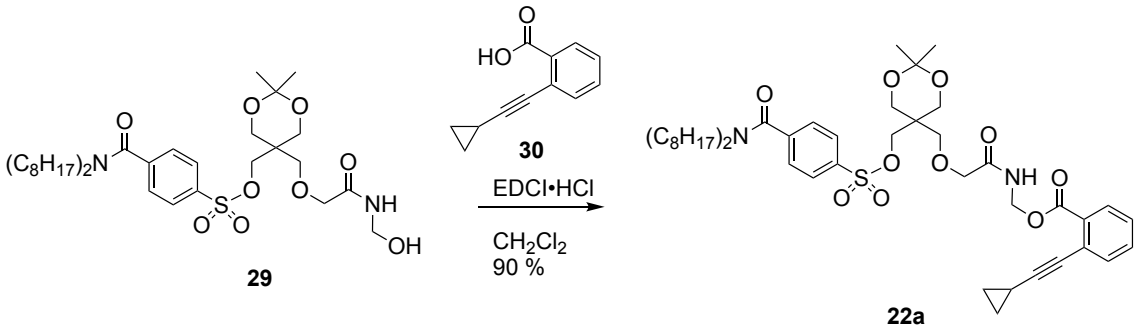
(5-((2-amino-2-oxoethoxy)methyl)-2,2-dimethyl-1,3-dioxan-5-yl)methyl 4-(didodecylcarbamoyl)benzenesulfonate (29)



28 **29**

To a stirred solution of **29** (23.5 mg, 31.2 μmol) in THF (0.25 mL) and water (0.25 mL) was added potassium carbonate (12.9 mg, 93.6 μmol) and paraformaldehyde (28.1 mg, 936 μmol) at 0 °C. After being stirred at room temperature for 12 h, the reaction mixture was poured into NH_4Cl aq. and EtOAc at 0 °C. The aqueous layer was extracted with ethyl acetate twice. The combined extract was washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel with hexane/ethyl acetate (1/3) to give **29** (20.2 mg, 25.8 μmol, 83%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, 2H, *J* = 8.8 Hz), 7.53 (d, 2H, *J* = 8.4 Hz), 7.08 (br, 1H), 4.78-4.81 (m, 2H), 4.26-4.30 (m, 1H), 4.09 (s, 2H), 3.86 (s, 2H), 3.67 (s, 4), 3.46-3.50 (m, 4H), 3.08-3.10 (m, 2H), 1.38 (d, 6H, *J* = 5.6 Hz), 1.35 (m, 4H), 1.26 (m, 36H), 0.880(t, 6H, *J* = 6.6 Hz); ^{13}C NMR (400 MHz, $CDCl_3$) δ 169.6, 142.7, 135.5, 128.3, 127.4, 99.0, 70.7, 69.6, 69.3, 63.9, 62.0, 49.1, 45.0, 38.6, 31.9, 29.6, 29.5, 29.4, 29.3, 29.1, 28.7, 27.4, 27.0, 26.5, 23.8, 23.1, 22.7, 14.1; FT-IR (neat) 3401, 2926, 2854, 1683, 1635, 1540, 1456, 1373, 1188, 1089, 971, 842, 772(cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{43}H_{76}N_2O_8S$ 805.50127 [M+Na] +, found 805.50175.

(2-((5-((((4-(dioctylcarbamoyl)phenyl)sulfonyl)oxy)methyl)-2,2-dimethyl-1,3-dioxan-5-yl)methoxy)acetamido)methyl 2-(cyclopropylethynyl)benzoate (22a)



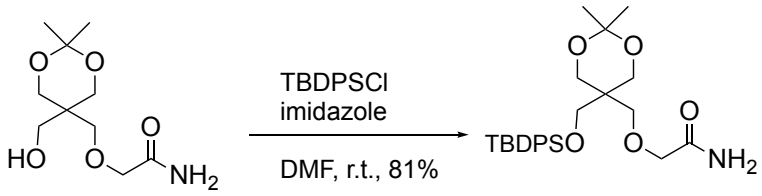
29 **22a**

To a stirred solution of **29** (14.5 mg, 21.6 μmol) and 2-(cyclopropylethynyl)benzoic acid (**30**) (8.85 mg, 47.5 μmol) in DCM (0.50 mL) were added $(PPh_3)AuOTf$ (12.4 mg, 64.8 μmol, 1.00 mmol/mL in DCM) and DMAP (5.28 mg, 43.2 μmol) at °C. After being stirred at room temperature for 2 h, the reaction mixture was poured into NH_4Cl aq. and EtOAc at 0 °C. The aqueous layer was extracted

with ethyl acetate twice. The combined extract was washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel with EtOAc/hexane (1/4, 1/1) to give **22a** (16.4 mg, 19.5 μmol, 90%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, 2H, *J* = 8.0 Hz), 7.92 (d, 1H, *J* = 8.0 Hz), 7.69 (t, 1H, *J* = 7.2 Hz), 7.52(d, 2H, *J* = 8.0 Hz), 7.38 -7.46 (m, 2H), 7.26-7.29 (m, 1H), 5.55 (d, 2H, *J* = 7.6 Hz), 4.17 (s, 2H), 3.98 (s, 2H), 3.64-3.73 (m, 4H), 3.46-3.50 (m, 4H), 3.11 (t, 1H, *J* = 7.2 Hz), 1.44-1.65 (m, 3H), 1.36 (s, 6H), 1.26 (m, 36H), 0.923-0.882 (m, 8H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 169.7, 165.6, 134.9, 133.6, 132.4, 131.3, 130.1, 129.2, 127.1, 126.3, 124.3, 99.3, 97.8, 73.7, 70.1, 63.3, 62.5, 61.9, 39.3, 26.2, 24.5, 21.7, 18.7, 8.33; FT-IR (neat) 3394, 2924, 2853, 2229, 1704, 1635, 1508, 1456, 1371, 1188, 1090, 956, 831, 757, 611(cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{54}H_{82}N_2O_{10}S$ 973.55878 [M+Na] +, found 973.55933.

Preparation of 22b

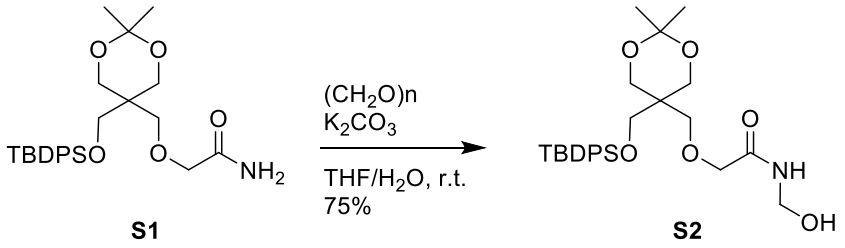
2-((5-(((tert-butyldiphenylsilyl)oxy)methyl)-2,2-dimethyl-1,3-dioxan-5-yl)methoxy)acetamide (S1)



28 **S1**

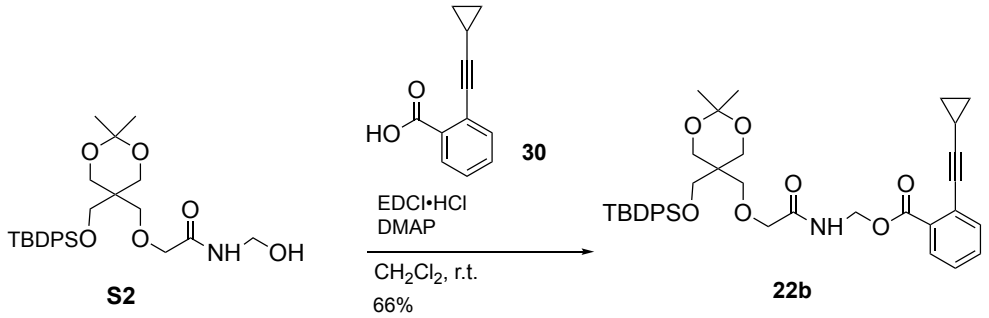
To a stirred solution of **28** (2.00 g, 8.57 mmol) in dry DMF (12.8 mL) was added imidazole (1.17 g, 17.1 mmol) and tert-butylchlorodiphenylsilane (2.42 mL, 9.43 mmol, 1.1 eq.) at 0 °C. After being stirred at room temperature for 2.5 h, the reaction mixture was poured into NH_4Cl aq. and EtOAc at 0 ˚C. The aqueous layer was extracted with ethyl acetate twice. The combined extract was washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel with hexane/ethyl acetate (1/1, 0/1) to give **S1** (3.26 g, 6.94 mmol, 81%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61-7.64 (m, 4H), 7.46-7.36 (m, 6H), 6.44 (br, 1H), 5.28 (br, 1H), 3.93 (s, 2), 3.76 (m, 4H), 3.63 (d, 2H, *J* = 4.4 Hz), 1.40 (s, 6H), 1.06 (s, 9H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 172.5, 135.5, 132.7, 129.9, 127.8, 98.4, 70.5, 63.1, 62.6, 39.8, 26.8, 25.3, 22.1, 19.3; FT-IR (neat) 3476, 3312, 3223, 3071, 3048, 2992, 2957, 2931, 2886, 2857, 1692, 1428, 1200, 1112, 1082, 832, 824, 756, 702, 614, 505 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{26}H_{37}NO_5Si$ 494.23387 [M+Na] +, found 494.23393.

2-((5-(((tert-butyldiphenylsilyl)oxy)methyl)-2,2-dimethyl-1,3-dioxan-5-yl)methoxy)-N-(hydroxymethyl)acetamide (S2)



To a stirred solution of **S1** (2.62 g, 5.55 mmol) in THF (15.0 mL) and water (15 mL) was added potassium carbonate (2.30 g, 16.7 mmol) and paraformaldehyde (1.67 g, 55.5 mmol) at 0 °C. After being stirred at room temperature for 2.5 h, the reaction mixture was poured into NH_4Cl aq. and EtOAc at 0 ˚C. The aqueous layer was extracted with ethyl acetate twice. The combined extract was washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was recrystallized from hexane/EtOAc to give **S2** (2.09 g, 4.17 mmol, 75%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64-7.61 (m, 4H), 7.46-7.35 (m, 6H), 4.71-4.74 (m, 2H), 3.95 (s, 2), 3.85-3.69 (m, 4H), 3.65 (s, 2H, H-d), 3.59 (s, 2), 3.16 (t, 1H, J = 7.6 Hz), 1.41 (d, 6H, J = 3.6 Hz), 1.06 (s, 9H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 171.7, 135.5, 133.0, 129.9, 127.8, 98.5, 70.4, 64.3, 63.2, 62.6, 39.9, 26.8, 26.0, 21.4, 19.3; FT-IR (neat) 3591, 3380, 2083, 1644, 1080, 821, 771, 742, 702 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{27}H_{39}NO_6Si$ 524.24443 $[M+Na]^+$, found 524.24403.

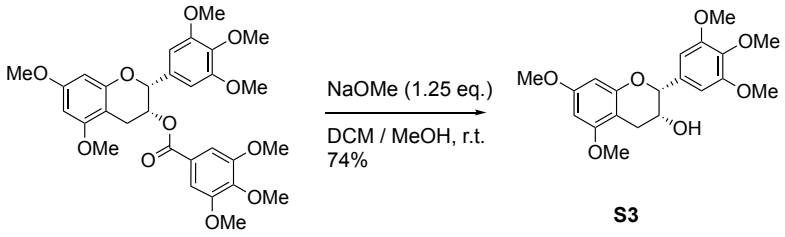
(2-((5-(((tert-butyldiphenylsilyl)oxy)methyl)-2,2-dimethyl-1,3-dioxan-5-yl)methoxy)acetamido)methyl 2-(cyclopropylethynyl)benzoate (22b)



To a stirred solution of **S2** (20.8 mg, 41.5 μmol) and **30** (8.49 mg, 45.6 μmol) in DCM (0.4 mL) were added EDCI•HCl (11.9 mg, 62.2 μmol) and DMAP (5.07 mg, 41.5 μmol) at 0 ˚C. After being stirred at room temperature for 13 h, the reaction mixture was poured into NH_4Cl aq. and EtOAc at 0 ˚C. The aqueous layer was extracted with ethyl acetate twice. The combined extract was washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel with EtOAc:toluene = 2% and 10% to give **22b** (18.5 mg, 27.6 μmol, 66%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (dd, 1H, J = 7.8 Hz, 1.0 Hz), 7.63-7.61 (m, 4H), 7.53(t,

1H, *J* = 7.6Hz), 7.43-7.41 (m, 2H), 7.34-7.40 (m, 6H), 7.25-7.28(m, 1), 5.48 (d, 2H, *J* = 7.2 Hz), 3.98 (s, 2), 3.72-3.83 (m, 4H), 3.63 (s, 4H), 1.46-1.55 (m, 1H), 1.39 (s, 6), 1.04 (s, 9), 0.846-0.887 (m, 4); ^{13}C NMR (400 MHz, $CDCl_3$) δ 169.7, 165.6, 134.9, 133.6, 132.4, 131.3, 130.1, 129.2, 127.1, 126.3, 124.3, 99.3, 97.8, 73.7, 70.1, 63.3, 62.5, 61.9, 39.3, 26.2, 24.5, 21.7, 18.7, 8.33; FT-IR (neat) 3654, 3435, 3361, 2931, 2857, 2230, 1703, 1516, 1238, 1112, 1081, 833, 757, 702, 614, 505, 491 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{39}H_{47}NO_7Si$ 692.30195 [M+Na] +, found 692.30273.

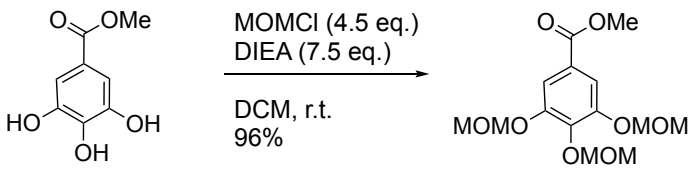
(2*R*,3*R*)-5,7-dimethoxy-2-(3,4,5-trimethoxyphenyl)chroman-3-ol (S3)



S3

To a stirred solution of (2*R*,3*R*)-5,7-dimethoxy-2-(3,4,5-trimethoxyphenyl)chroman-3-yl 3,4,5-trimethoxybenzoate (**13**) (1.50 g, 2.63 mmol) in MeOH (8.75 mL) and DCM (8.75 mL) was added sodium methanolate (178 mg, 3.29 mmol) at 0 ℃. After being stirred at room temperature for 12h, the reaction mixture was poured into NH_4Cl aq. and EtOAc at 0 ℃. The aqueous layer was extracted with ethyl acetate twice. The combined extract was washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was recrystallized from hexane/ EtOAc to give **S3** (728 mg, 1.93 mmol, 74%). [α]$_D^{21}$ +10.76 (*c* 1.00, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.75 (s, 2H), 6.21 (d, 1H, *J* =2.4Hz), 6.12 (d, 1H, *J* =2.4Hz), 4.94 (s, 1H), 4.29 (br, 1H), 3.90 (s, 6H), 3.86 (s, 3H), 3.80 (s, 3H), 3.78 (s, 3H), 3.00-2.86 (m, 2H, H-4); ^{13}C NMR (400 MHz, $CDCl_3$) δ 159.7, 159.3, 155.0, 153.5, 137.7, 133.9, 103.3, 100.2, 93.3, 92.3, 78.6, 66.5, 60.8, 56.2, 55.5, 55.4, 28.0; FT-IR (neat) 3482, 2998, 2937, 2839, 1619, 1592, 1507, 1458, 1419, 1356, 1236, 1199, 1147, 1124, 1004, 816, 758 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{20}H_{24}O_7$ 399.14197 [M+Na] +, found 399.14147.

methyl 3,4,5-tris(methoxymethoxy)benzoate (S4)

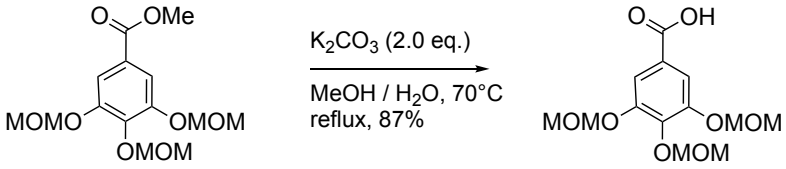


S4

To a stirred solution of methyl 3,4,5-trihydroxybenzoate (1.00 g, 5.43 mmol) in DCM (54 mL) was added DIEA (7.10 mL, 40.7 mmol) and chloro(methoxy)methane (1.86 mL, 24.4 mmol) at 0 ℃. After

being stirred at room temperature for 2 h, the reaction mixture was poured into NH_4Cl aq. and EtOAc at 0 ˚C. The aqueous layer was extracted with ethyl acetate twice. The combined extract was washed with and brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel with EtOAc/toluene = 7% and was recrystallized from hexane/ EtOAc to give **S4** (1.64 g, 5.21 mmol, 96%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (s, 2H), 5.24 (s, 4H), 5.21 (s, 2H), 3.88 (s, 3H), 3.61 (s, 3H), 3.51 (s, 6H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 166.3, 150.7, 140.6, 125.9, 111.6, 98.5, 95.2, 57.2, 56.4, 52.2; FT-IR (neat) 2956, 2830, 1721, 1594, 1499, 1435, 1330, 1156, 1079, 1049, 956, 923, 767 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{14}H_{20}O_8$ 399.10559 [M+Na] +, found 399.10558.

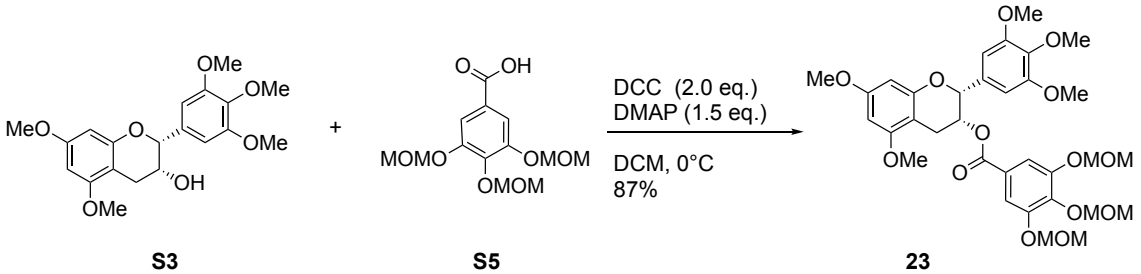
3,4,5-tris(methoxymethoxy)benzoic acid (S5)



S4 **S5**

To a stirred solution of **S4** (623 mg, 1.97 mmol) in MeOH (3.20 mL) was added potassium carbonate (544 mg, 3.94 mmol) and water (3.20 mL) at room temperature. After being stirred at 70 ˚C for 2 h, the reaction mixture was poured into NH_4Cl aq. and EtOAc and 1 M aq. HCl was added until the reaction mixture was acidic at 0 ˚C. The aqueous layer was extracted with ethyl acetate twice. The combined extract was washed with $NaHCO_3$ and brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was recrystallized from hexane/$CHCl_3$ to give **S5** (518 mg, 1.71 mmol, 87%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (s, 2H), 5.26 (s, 4H), 5.24 (s, 2H), 3.62 (s, 3H), 3.52 (s, 6H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 170.5, 150.7, 141.3, 124.8, 112.2, 98.4, 95.3, 57.2, 56.4; FT-IR (neat) 3444, 2965, 2920, 2843, 1686, 1433, 1325, 1151, 1046, 1033, 953, 919, 762 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{13}H_{18}O_8$ 325.08994 [M+Na] +, found 325.09015.

(2*R*,3*R*)-5,7-dimethoxy-2-(3,4,5-trimethoxyphenyl)chroman-3-yl 3,4,5-tris(methoxymethoxy)benzoate (23)

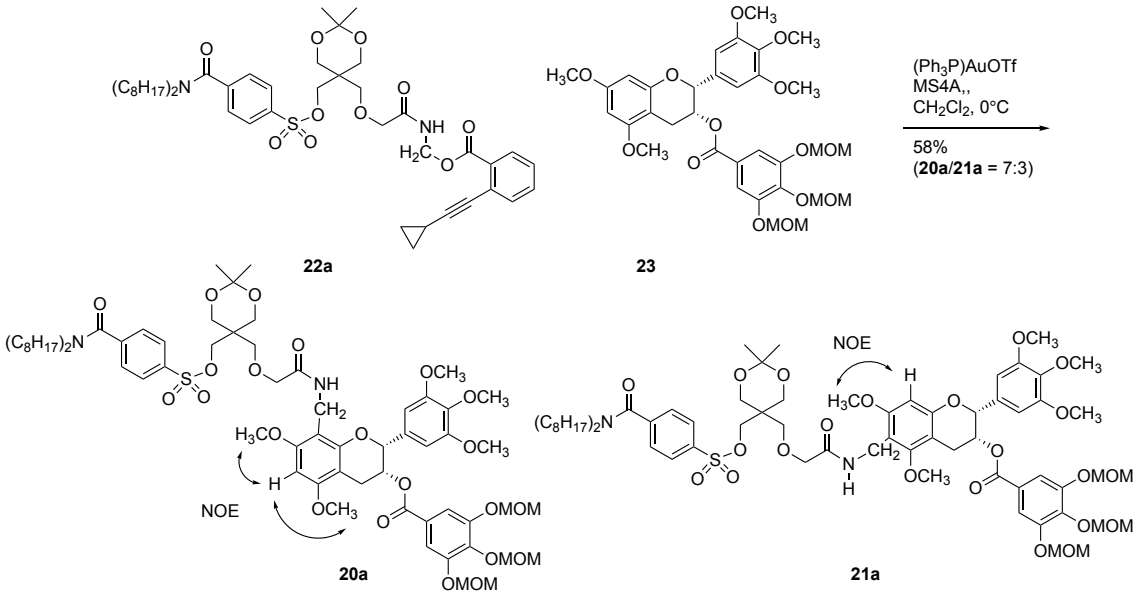


S3 **S5** **23**

To a stirred solution of **S3** (50.0 mg, 0.133 mol) and **S5** (88.4 mg, 0.292 mmol) in DCM (2.00 mL)

were added DCC (54.8 mg, 0.266 mmol) and DMAP (24.3 mg, 0.199 mmol) at 0 ˚C. After being stirred at room temperature for 3 h, the reaction mixture was filtered through celite and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel with EtOAc:toluene = 15% to give **23** (98.8mg, 0.150 mmol, 87%). $[\alpha]_D^{21}$ -67.12 (*c* 1.06, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.41 (s, 2H), 6.70 (s, 2H), 6.24 (d, 1H, *J* = 2.8 Hz), 6.11 (d, 1H, *J* = 2.0 Hz), 5.63 (br, 1H), 5.16-5.07 (m, 7H,), 3.84 (s, 3H), 3.79 (s, 3H), 3.77 (s, 3H), 3.73 (s, 6H), 3.56 (s, 3H), 3.44 (s, 6), 3.03 (m, 2); ^{13}C NMR (400 MHz, $CDCl_3$) δ 164.7, 159.7, 158.8, 155.4, 153.1, 150.6, 141.2, 137.8, 133.4, 125.7, 112.3, 103.8, 100.0, 98.5, 95.4, 93.2, 91.8, 77.8, 68.7, 60.8, 57.2, 56.3, 56.0, 55.4, 26.0; FT-IR (neat) 2938, 2839, 1716, 1621, 1593, 1506, 1498, 1433, 1361, 1322, 1221, 1188, 1153, 1125, 1048, 949, 924, 765 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{33}H_{40}O_{14}$ 683.23157 [M+Na] +, found 683.23273.

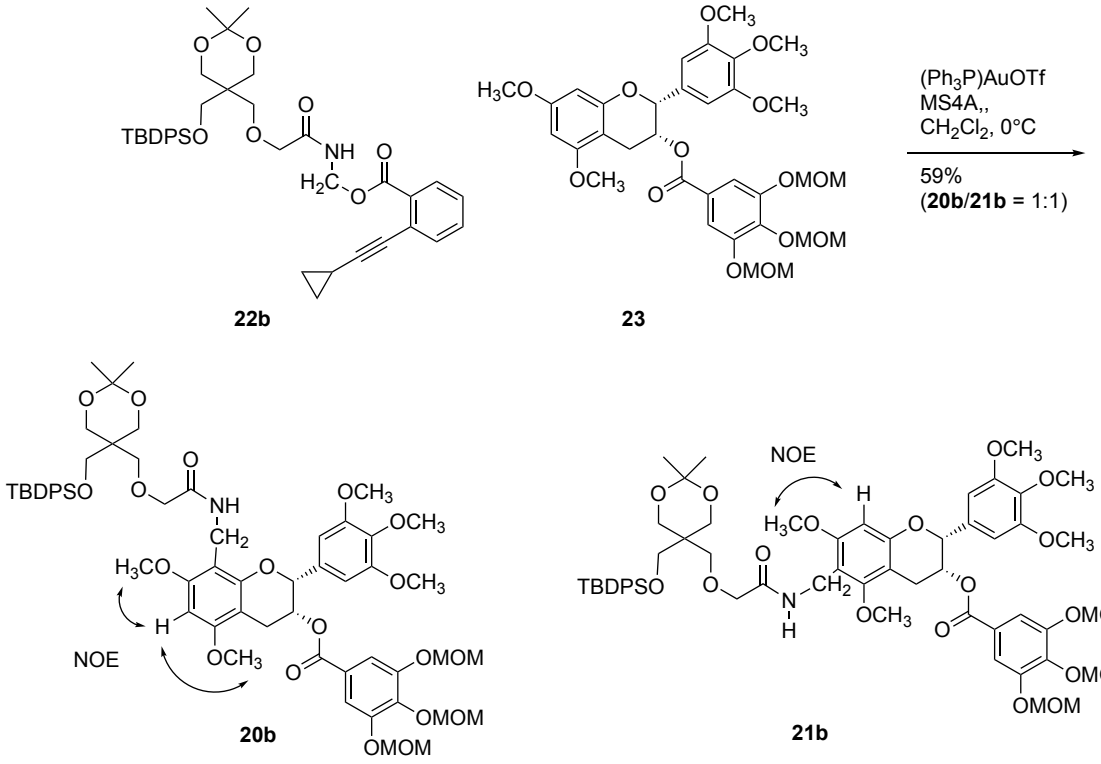
A gold-catalyzed electrophilic substitution of EGCG with 22a



To MS4A (27.8 mg, 2.00 g/mmol) heated by heat gun for 15 min was added **22a** (10.0 mg, 11.9 μmol) and **23** (8.45 mg, 13.1 μmol) in DCM (0.30 mL) at 0 ˚C. After being stirred at room temperature for 1 h, (PPh3)AuOTf (11.9 μL, 11.9 μmol, 1.00 mmol/mL in DCM) was added to the reaction mixture at 0 ºC. After being stirred at 0 ˚C for 10 min, the reaction mixture was filtered through celite, washed by DCM and concentrated *in vacuo*. The residue was purified by a column chromatography on silica gel with EtOAc/toluene (1:1) and further purified by gel permeation chromatography (GPC) to give mixture to give the mixture of (2*R*,3*R*)-8-((2-((5-((((4-(diundecylcarbamoyl)phenyl)sulfonyl)oxy)methyl)-2,2-dimethyl-1,3-dioxan-5-

yl)methoxy)acetamido)methyl)-5,7-dimethoxy-2-(3,4,5-trimethoxyphenyl)chroman-3-yl 3,4,5-tris(methoxymethoxy)benzoate (**20a**) and (2*R*,3*R*)-6-((2-((5-((((4-(diundecylcarbamoyl)phenyl)sulfonyl)oxy)methyl)-2,2-dimethyl-1,3-dioxan-5-yl)methoxy)acetamido)methyl)-5,7-dimethoxy-2-(3,4,5-trimethoxyphenyl)chroman-3-yl 3,4,5-tris(methoxymethoxy)benzoate (**21a**) (9.60 mg, 6.87 μmol, 58%). Then ratio of the mixture was decided by 1H NMR which showed **20a**/**21a** = 7/3. Then the mixture was separated by HPLC with ethanol/hexane (1/4) to give **20a** (4.80 mg, 3.43μmol, 29%) and **21a** (1.80 mg, 1.28μmol, 12%). **20a**:$[\alpha]_D^{21}$ -18.33 (*c* 0.12, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, 2H, *J* = 8.4 Hz), 7.49 (d, 2H, *J* = 8.4 Hz), 7.35 (s, 2H), 6.79 (m, 3H), 6.14 (s, 1H), 5.64 (br, 1H), 5.20 (br, 1H), 5.14 (s, 2H), 5.12 (d, 4H, *J* = 2.4 Hz), 4.89 (dd, 1H, *J* = 6.6 Hz, 13.8 Hz), 4.46 (dd, 1H, *J* = 4.4 Hz, 14.0 Hz), 4.06 (s, 2H), 3.86 (s, 3H), 3.84 (s, 1H), 3.81 (s, 6H), 3.80 (s, 6H), 3.55 (s, 3H), 3.55-3.52 (m, 2H), 3.48-3.45 (m, 4H), 3.42 (s, 6H), 3.36-3.30 (m, 2H), 3.13-3.04 (m, 4H), 1.34 (br, 4H), 1.26-1.25 (m, H-42), 0.88 (t, 6H, *J* = 6.0 Hz); ^{13}C NMR (400 MHz, $CDCl_3$) δ 169.3, 167.9, 158.1, 153.2, 153.1, 150.5, 142.9, 136.0, 133.3, 128.1, 127.3, 125.6, 112.0, 105.7, 103.1, 100.3, 98.6, 98.4, 95.4, 88.1, 71.2, 70.0, 69.1, 68.5, 61.8, 61.6, 60.8, 57.2, 56.3, 56.1, 55.9, 55.4, 38.4, 31.9, 29.6 29.4, 29.3, 29.2, 27.0, 26.6, 25.9, 24.9, 22.7, 21.9, 14.1; FT-IR (neat)2923, 2852, 1717, 1683, 1636, 1593, 1508, 1457, 1362, 1188, 1154, 1127, 1090, 1047, 954, 800 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{75}H_{112}N_2O_{22}S$ 1447.73251 [M+Na] +, found 1447.73205; **21a**: $[\alpha]_D^{21}$ -5.09 (*c* 0.22, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, 2H, *J* = 8.4 Hz), 7.51 (d, 2H, *J* = 8.4 Hz), 7.37 (s, 2H), 6.95 (t, 1H, *J* = 5.6 Hz), 6.68 (s, 2H), 6.44 (s, 1H), 5.64 (br, 1H), 5.16 (s, 2H), 5.13 (d, 4H,*J* = 3.2 Hz), 5.10 (br, 1H),4.72 (dd, 1H, *J* = 6.2 Hz, 14.2 Hz), 4.36 (dd, 1H, *J* = 5.2 Hz, 14.0 Hz), 4.16 (s, 2H), 3.88 (m, 2H), 3.86 (s, 3H), 3.80 (s, 3H), 3.77 (s, 3H), 3.73 (s, 6H), 3.63-3.59 (m, 4H), 3.56 (s, 3H), 3.43 (s, 6H), 3.42-3.39 (m, 2), 3.15 (br, 2H), 3.11 (t, 2H, *J* = 4.8 Hz), 1.33-1.25 (m, 46H), 0.88 (t, 6H, *J* = 6.2 Hz); ^{13}C NMR (400 MHz, $CDCl_3$) δ 169.3, 167.9, 157.8, 155.2, 153.2, 150.6, 143.0, 135.9, 133.0, 128.2, 127.4, 112.2, 104.8, 103.8, 98.6, 98.5, 95.9, 95.4, 71.2, 70.1, 69.2, 68.3, 61.8, 61.4, 60.8, 57.2, 56.3, 56.0, 55.8, 38.5, 31.9, 29.6, 29.4, 29.3, 29.2, 27.4, 26.6, 25.0, 22.7, 21.9, 14.1; FT-IR (neat) 2920, 2850, 1731, 1716, 1682, 1635, 1617, 1592, 1507, 1456, 1362, 1187, 1155, 1128, 1107, 1047, 961, 799, 773(cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{75}H_{112}N_2O_{22}S$ 1447.73251 [M+Na] +, found 1447.73357.

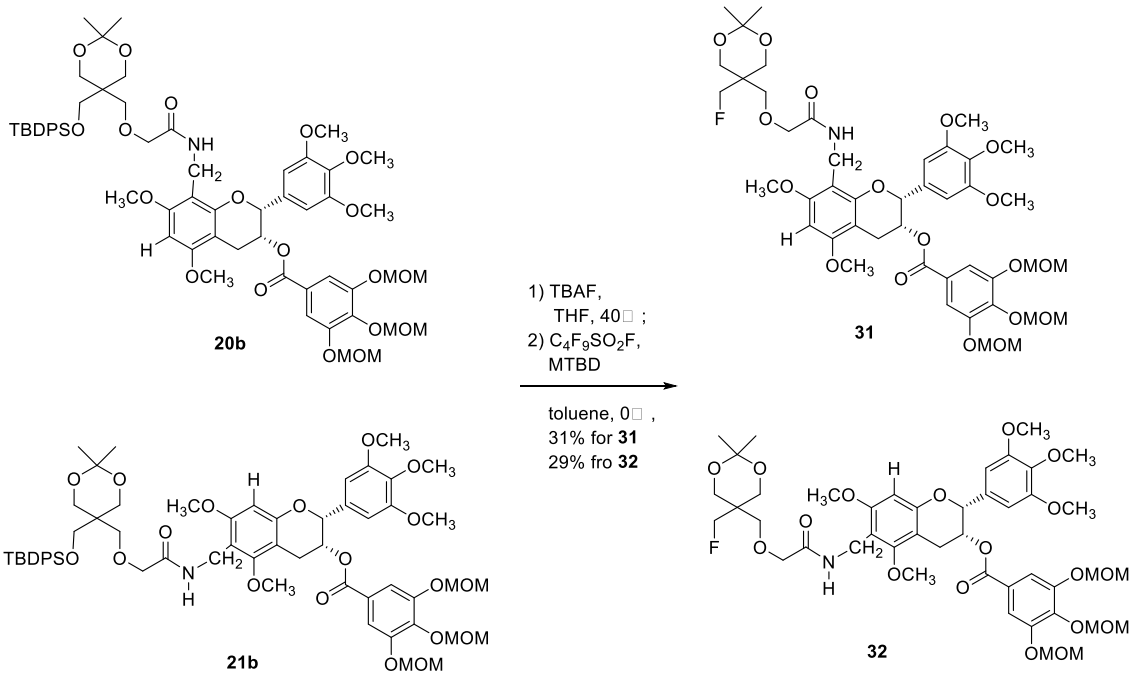
A gold-catalyzed electrophilic substitution of EGCG with 22b



To MS4A (212 mg, 2.00 g/mmol) heated by heat gun for 10min was added **22b** (60.4 mg, 90.0 μmol) and **23** (64.0 mg, 99.0 μmol) in DCM (1.80 mL) at 0 ˚C. After being stirred at room temperature for 1 h, $(PPh_3)AuOTf$ (90.0 μL , 90.0 μmol , 1.00 mmol/mL in DCM) was added to the reaction mixture at 0 ºC. After being stirred at 0 ºC for 15min, the reaction mixture was filtered through celite, washed by DCM, and concentrated *in vacuo*. The residue was short passed by column chromatography on silica gel with EtOAc : toluene = 50% and further purified by Gel Permeation Chromatography (GPC) to give mixture to give the mixture of (2*R*,3*R*)-8-((2-((5-(((tert-butyldiphenylsilyl)oxy)methyl)-2,2-dimethyl-1,3-dioxan-5-yl)methoxy)acetamido)methyl)-5,7-dimethoxy-2-(3,4,5-trimethoxyphenyl)chroman-3-yl 3,4,5-tris(methoxymethoxy)benzoate (**20b**) and (2*R*,3*R*)-6-((2-((5-(((tert-butyldiphenylsilyl)oxy)methyl)-2,2-dimethyl-1,3-dioxan-5-yl)methoxy)acetamido)methyl)-5,7-dimethoxy-2-(3,4,5-trimethoxyphenyl)chroman-3-yl 3,4,5-tris(methoxymethoxy)benzoate (**21b**) (60.8 mg, 53.1 μmol, 59%). Then ratio of the mixture was decided by 1H NMR which showed **20b**/**21b** = 1:1. Then the 30.0 mg mixture was separated by HPLC with ethanol/hexane(1/4) to give **20b** (10.4 mg) and **21b** (10.9 mg). **20b**: $[\alpha]_D^{21}$ -14.81 (*c* 0.52, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.60-7.51 (m, 4H), 7.38-7.27 (m, 6H), 7.26 (s, 2H), 6.88 (t, 1H, *J* = 4.8 Hz), 6.61 (s, 2H), 6.00 (s, 1H), 5.49 (br, 1H), 5.07 (s, 2H), 5.04 (s, 2H), 5.03 (s, 2H), 4.90 (m, 2H), 4.31 (dd, 1H, *J* = 4.4Hz, 7.0 Hz), 3.83 (s, 2H), 3.73 (s, 3H), 3.70 (s, 3H), 3.68

(s, 3H), 3.66 (s, 6H), 3.47 (s, 3H), 3.34 (s, 6H), 3.60 (s, 2H), 3.58 (s, 2H), 3.43-3.40 (m, 4H), 3.00 (dd, 1H, *J* = 6.8 Hz, 17.2 Hz), 2.83 (dd, 1H, *J* = 4.2 Hz, 17.2 Hz), 1.25 (d, 6H, *J* = 10.8 Hz), 0.92 (s, 9H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 163.6, 157.0, 155.6, 153.9, 152.2, 149.8, 149.7, 149.6, 140.2, 136.6, 134.5, 134.4, 134.3, 131.9, 129.1, 129.0, 126.8, 124.6, 111.4, 110.9, 102.3, 100.3, 97.4, 94.4, 94.3, 88.0, 69.3, 67.4, 63.3, 59.8, 56.2, 55.4, 55.3, 55.2, 55.1, 55.0, 54.8, 54.5, 54.4, 30.9, 30.0, 28.7, 25.8, 25.7, 25.6, 21.6, 18.2, 13.1, 0.00; FT-IR (neat) 2998, 2955, 2928, 2854, 1719, 1659, 1594, 1508, 1463, 1431, 1417, 1358, 1327, 1236, 1222, 1189, 1154, 1128, 1111, 1047, 925, 769, 760, 702 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{60}H_{77}NO_{19}Si$ 1166.47567 [M+Na] +, found 1166.47537; **21b**: $[\alpha]_D^{21}$ -11.70 (*c* 0.54, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64-7.62 (m, 4H), 7.44-7.32 (m, 6H), 7.37 (s, 2H), 7.02 (t, 1H, *J* = 5.8 Hz), 6.67 (s, 2H), 6.36 (s, 1H), 5.63 (br, 1H), 5.15 (s, 2H), 5.11 (d, 4H, *J* = 2.8 Hz), 5.08 (d, 2H, *J* = 3.2 Hz), 4.67 (dd, 1H, *J* = 6.8 Hz, 14.3 Hz), 4.35 (dd, 1H, *J* = 4.6 Hz, 14.3 Hz), 3.90 (s, 2H), 3.80 (s, 3H), 3.76 (s, 2H), 3.74 (s, 3H), 3.72 (s, 6H), 3.66 (s, 2H), 3.64 (s, 3H), 3.56-3.55 (m, 5H), 3.42 (s, 6H), 3.13 (d, 2H, *J* = 2.8 Hz), 1.36 (s, 6H), 1.02 (s, 9H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 164.6, 158.1, 157.7, 155.1, 153.2, 150.6, 141.3, 135.5, 133.0, 132.7, 130.9, 130.0, 129.8, 128.8, 127.8, 127.7, 125.5, 112.2, 103.8, 98.5, 98.2, 95.9, 95.4, 77.9, 77.2, 70.9, 70.6, 68.3, 68.1, 64.4, 64.3, 62.7, 62.4, 61.5, 60.8, 57.2, 56.3, 56.0, 55.5, 45.8, 39.8, 32.4, 31.9, 30.9, 29.7, 26.8, 26.7, 26.3, 23.7, 23.0, 22.7, 19.3, 19.2, 14.1, 1.03; FT-IR (neat) 2994, 2955, 2928, 2855, 1719, 1666, 1592, 1462, 1428, 1361, 1321, 1300, 1220, 1188, 1155, 1129, 1107, 1047, 949, 924, 771, 702 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{60}H_{77}NO_{19}Si$ 1166.47567 [M+Na] +, found 1166.47553.

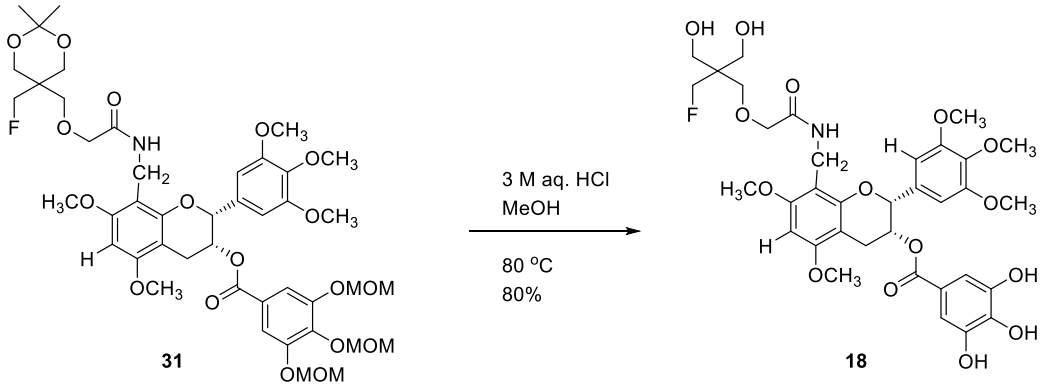
Synthesis of fluorides 30 and 31



To a stirred solution of the mixture of **20b** and (2*R*,3*R*)-6-((2-((5-(((tert-butyldiphenylsilyl)oxy)methyl)-2,2-dimethyl-1,3-dioxan-5-yl)methoxy)acetamido)methyl)-5,7-dimethoxy-2-(3,4,5-trimethoxyphenyl)chroman-3-yl 3,4,5-tris(methoxymethoxy)benzoate (**21b**) (420 mg, 367 μmol, 1.0 eq.) in THF (2.00 mL) was added tetra-n-butylammonium fluoride (4.40 mL, 4.40 mmol, 12 eq.) at 0 ˚C. After being stirred at 40 ˚C for 3 h, the reaction mixture was poured into NH_4Cl aq. and DCM at 0 ˚C. The aqueous layer was extracted with DCM twice. The combined extract was washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. To a stirred solution of the residue in dry toluene (0.210 mL) was added 1-methyl-1,3,4,6,7,8-hexahydro-2H-pyrimido[1,2-a]pyrimidine (MTBD) (223 μL, 1.54 mmol) and 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonyl fluoride (81.3 μL, 463 μmol) at 0 ˚C. After being stirred at 0 ˚C for 40 min, the reaction mixture was poured into NH_4Cl aq. and chloroform at 0 ˚C. The aqueous layer was extracted with chloroform twice. The combined extract was washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel with EtOAc : chloroform = 2% to give the mixture of (2*R*,3*R*)-8-((2-((5-(fluoromethyl)-2,2-dimethyl-1,3-dioxan-5-yl)methoxy)acetamido)methyl)-5,7-dimethoxy-2-(3,4,5-trimethoxyphenyl)chroman-3-yl 3,4,5-tris(methoxymethoxy)benzoate (**31**) and (2*R*,3*R*)-6-((2-((5-(fluoromethyl)-2,2-dimethyl-1,3-dioxan-5-yl)methoxy)acetamido)methyl)-5,7-dimethoxy-2-(3,4,5-trimethoxyphenyl)chroman-3-yl 3,4,5-tris(methoxymethoxy)benzoate (**32**) (227.3 mg,

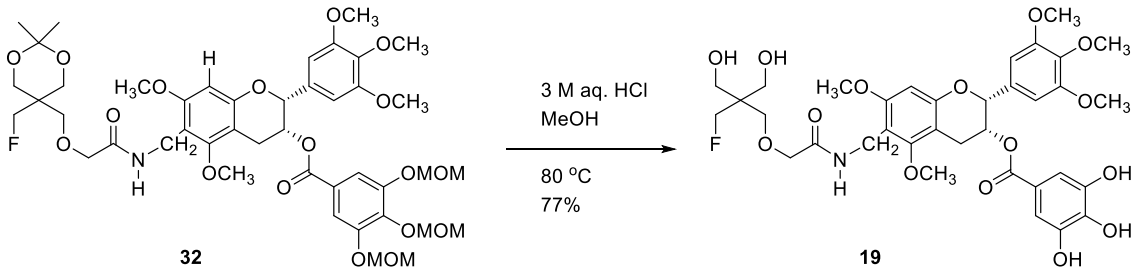
231.2 μmol, 63% in 2 steps). Then the mixture was separated by column chromatography on silica gel with hexane/ethyl acetate (3/7) five times to give **31** (112 mg, 112 μmol, 31%) and **32** (104 mg, 106 μmol, 29%). **31**: $[\alpha]_D^{21}$ -76.18 (*c* 0.38, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.35 (s, 2H), 6.84 (t, 1H, *J* = 4.4 Hz), 6.78 (s, 2H), 6.13 (s, 1H), 5.64 (br, 1), 5.14 (m, 3H), 5.12 (d, 4H, *J* = 2.4 Hz), 4.93 (dd, 1H, *J* = 7.2 Hz, 14.0 Hz), 4.42 (dd, 1H, *J* = 4.4 Hz, 14.0 Hz), 4.38, 4.26 (m, 2H), 3.94 (s, 2H), 3.87 (s, 3H), 3.82 (s, 3H), 3.81 (s, 3), 3.80 (s, 6H), 3.77-3.75 (m, 2H), 3.64-3.60 (m, 4H), 3.55 (s, 3H), 3.42 (s, 6H), 3.15-2.99 (m, 2H), 1.35 (d, 6H, *J* = 14.8 Hz); ^{13}C NMR (400 MHz, $CDCl_3$) δ 168.1, 164.8, 158.1, 157.6, 153.2, 153.0, 150.6, 141.0, 137.5, 133.3, 125.6, 112.3, 112.0, 105.7, 103.1, 100.2, 98.5, 95.3, 88.1, 83.3, 81.6, 77.2, 71.1, 70.3, 68.4, 61.4, 61.3, 60.8, 57.2, 56.3, 56.1, 56.0, 55.9, 55.4, 39.1, 38.9, 31.6, 31.5, 29.7, 25.8, 24.3, 22.7, 14.1; ^{19}F NMR (376 MHz, $CDCl_3$) δ -235.5 (t, 1F, *J* = 47 Hz); FT-IR (neat) 2937, 1716, 1682, 1617, 1594, 1507, 1456, 1360, 1328, 1223, 1154, 1128, 1089, 1048, 947, 925, 767(cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{44}H_{58}NO_{18}$ 930.35356 [M+Na] +, found 930.35397; **32**: $[\alpha]_D^{21}$ -59.04 (*c* 0.25, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.38 (s, 2H), 7.00 (t, 1H, *J* = 5.8 Hz), 6.68 (s, 2H), 6.44 (s, 1H), 5.64 (br, 1H), 5.16 (s, 2H), 5.13 (d, 4H, *J* = 2.8 Hz), 5.11 (s, 1H), 4.68 (dd, 1H, *J* = 6.8 Hz, 14.0 Hz), 4.48 (d, 2H, *J* = 47.2 Hz), 4.38 (dd, 1H, *J* = 4.8 Hz, 14.0 Hz), 3.94 (s, 2H), 3.87 (s, 3H), 3.81 (s, 3H), 3.77 (s, 3H), 3.73 (m, 10H), 3.56 (s, 3H), 3.48 (br, 2H), 3.43 (s, 6H), 3.15 (d, 2H, *J* = 3.2 Hz), 1.40 (d, 6H, *J* = 4.0 Hz); ^{13}C NMR (400 MHz, $CDCl_3$) δ 168.2, 164.6, 158.1, 157.7, 155.2, 153.2, 150.6, 141.3, 137.9, 133.0, 125.5, 112.3, 112.2, 104.8, 103.8, 98.5, 95.8, 95.4, 83.5, 81.8, 77.9, 71.1, 70.4, 68.3, 61.5, 61.4, 60.8, 57.2, 56.3, 56.0, 55.7, 39.3, 39.1, 32.4, 29.7, 26.3, 24.4, 22.7, 14.1; ^{19}F NMR (376 MHz, $CDCl_3$) δ -235.6 (t, 1F, *J* = 47 Hz); FT-IR (neat) 2939, 1717, 1682, 1617, 1521, 1592, 1507, 1456, 1223, 1189, 1155, 1129, 1108, 1089, 1047, 948, 923, 828, 768 (cm^{-1}); HRMS (ESI-TOF) calcd. for $C_{44}H_{58}NO_{18}$ 930.35356 [M+Na] +, found 930.35419.

(2*R*,3*R*)-8-((2-(3-fluoro-2,2-bis(hydroxymethyl)propoxy)acetamido)methyl)-5,7-dimethoxy-2-(3,4,5-trimethoxyphenyl)chroman-3-yl 3,4,5-trihydroxybenzoate (18)



To a stirred solution of the mixture of **31** (18.0 mg, 19.8 μmol) MeOH (0.30 mL) was added 3 M aq. HCl (0.30 mL) at 0 ℃. After being stirred at 80 ºC for 5 min, the reaction mixture was purified through solid phase extraction with water : MeOH = 1:1 to give **18** (11.9 mg, 15.8 μmol, 80%). $[\alpha]_D^{21}$ -8.811 (*c* 0.18, MeOH); ^{1}H NMR (400 MHz, MeOD) δ 6.96 (s, 2H), 6.87 (s, 2H), 6.34 (s, 1H), 5.62 (br, 1H), 5.20 (s, 1H), 4.44-4.32 (m, 4H), 3.95 (s, 2H), 3.92 (s, 3H), 3.87 (s, 3H), 3.73 (s, 6H), 3.71 (s, 3H), 3.52-3.49 (m, 4H), 3.45 (br, 2H), 3.16-2.93 (m, 2H) ; ^{13}C NMR (400 MHz, MeOD) δ 170.4, 165.7, 158.4, 157.8, 152.9, 145.1, 138.4, 137.1, 134.1, 129.1, 119.8, 108.8, 104.7, 103.6, 99.9, 87.9, 83.1, 81.4, 77.6, 69.9, 69.3, 67.8, 59.8, 59.7, 55.1, 54.9, 54.6, 46.0, 31.5, 25.6; ^{19}F NMR (376 MHz, MeOD) δ -239.8 (t, 1F, *J* = 47 Hz); FT-IR (neat) (cm^{-1}) 3419, 2523, 2064, 1645, 1635, 1617, 1456, 1338, 1232, 1127, 1033; HRMS (ESI-TOF) calcd. for $C_{35}H_{42}FNO_{15}$ 758.24362 [M+Na] +, found 758.24517.

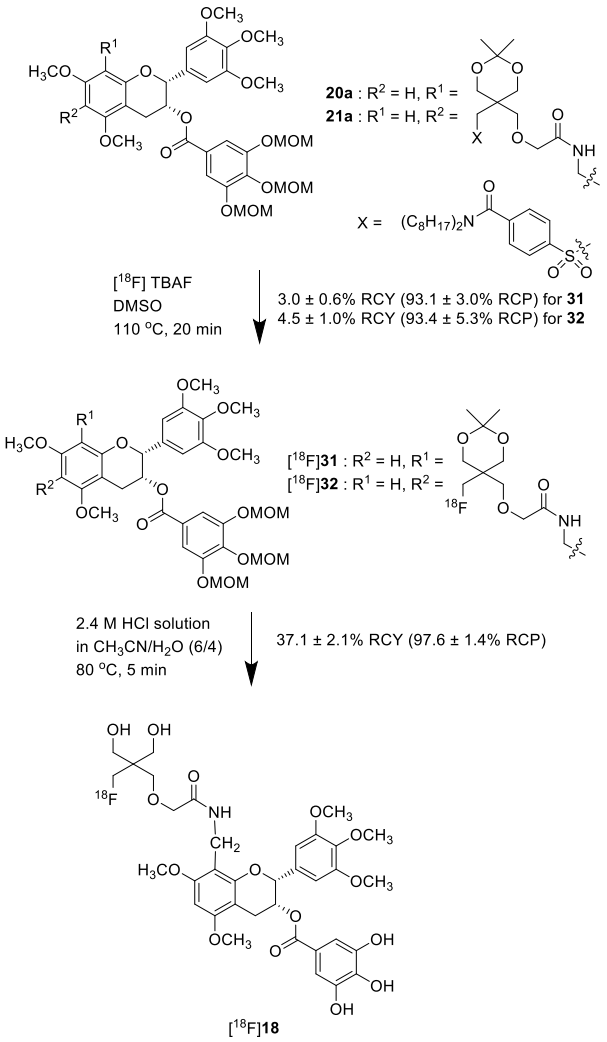
(2*R*,3*R*)-6-((2-(3-fluoro-2,2-bis(hydroxymethyl)propoxy)acetamido)methyl)-5,7-dimethoxy-2-(3,4,5-trimethoxyphenyl)chroman-3-yl 3,4,5-trihydroxybenzoate (19)



32 **19**

To a stirred solution of the mixture of **32** (12.0 mg, 13.2 μmol) in MeOH (0.30 mL) was added 3 M aq. HCl (0.30 mL) at 0 ℃. After being stirred at 40 ℃ for 3 h, the reaction mixture was purified through solid phase extraction with water : MeOH = 1:1 to give **19** (7.50 mg, 10.0 μmol, 77%). $[\alpha]_D^{21}$ +10.76 (*c* 0.31, MeOH); ^{1}H NMR (400 MHz, MeOD) δ 6.95 (s, 2H), 6.82 (s, 2H), 6.51 (s, 1H), 5.63 (br, 1H), 5.22 (s, 1H), 4.55-4.38 (m, 4H), 3.96 (s, 2H), 3.89 (s, 3H), 3.78 (s, 3H), 3.73 (s, 3H), 3.70 (s, 6H), 3.57 (s, 4H), 3.50 (s, 2H), 3.32-3.02 (m, 2H) ; ^{13}C NMR (400 MHz, MeOD) δ 170.3, 165.6, 158.1, 157.9, 155.6, 152.9, 145.1, 138.6, 137.3, 134.1, 119.7, 111.3, 108.7, 104.7, 103.9, 95.6, 83.2, 81.5, 77.8, 69.9, 69.4, 67.7, 60.5, 59.9, 59.7, 55.1, 55.0, 46.1, 45.9, 32.1, 25.8; ^{19}F NMR (376 MHz, MeOD) δ-235.5 (t, 1F, *J* = 47 Hz); FT-IR (neat) (cm^{-1}) 3420, 2064, 1652, 1636, 1617, 1540, 1456, 1420, 1339, 1231, 1128, 1034; HRMS (ESI-TOF) calcd. for $C_{35}H_{42}FNO_{15}$ 758.24362 [M+Na] +, found 758.24413.

Radiochemistry



General

[^{18}F]Fluoride ion was produced by proton irradiation of ^{18}O-enriched water (Taiyo Nippon Sanso, Tokyo, Japan) using an HM-20 cyclotron (Sumitomo Heavy Industries, Tokyo, Japan). Kryptofix® 222 was purchased from Merck (Darmstadt, Germany). Other reagents were purchased from Tokyo Chemical Industry (Tokyo, Japan), Sigma-Aldrich (St. Louis, MO) or FUJIFILM Wako Pure Chemical (Osaka, Japan), unless otherwise stated.

Radiosynthesis of [^{18}F]31 and [^{18}F]32

[^{18}F]**31** and [^{18}F]**32** were radiosynthesized using an F100 synthesizer (Sumitomo Heavy Industries, Tokyo, Japan) with conventional semi-preparative HPLC equipment. Irradiated ^{18}O-enriched water containing [^{18}F]fluoride (20–25 GBq) was passed through a Sep-Pak Accell Plus QMA Carbonate Plus Light cartridge (washed before use with 1 M $KHCO_3$ 5.00 mL and then water 8.00 mL; Waters). The trapped [^{18}F]fluoride was eluted into a reaction vessel using 50% acetonitrile aqueous solution containing $TBAHCO_3$ (37.5 mM, 1.00 mL). After the solvent was dried by heating, the residue was azeotropically dried with acetonitrile (1.00 mL). A mixture of **20a** and **21a** in DMSO (2.00 mg/500 μL) was added into the reaction vessel and heated at 110 °C for 20 min. The reaction mixture was mixed with eluent for semi-preparative HPLC (1.00 mL), and then purified by HPLC on a system equipped with a radioactivity detector (Column: Ecripse XDB-C18, 5 μm, 9.4 × 250 mm [Agilent Technologies, Santa Clara, CA]; Eluent: acetonitrile/ethanol/50.0 mM acetic acid/50.0 mM ammonium acetate = 55/5/20/20; Flow rate: 5.00 mL/min; UV: 254 nm). The collected fractions containing [^{18}F]**31** (retention time = 12 min) or [^{18}F]**32** (retention time = 14 min) were analysed by analytical HPLC with a radioactivity detector (Column: YMC-Pack ODS-A, 3 μm, 4.6 × 150 mm [YMC, Kyoto, Japan]; Eluent: acetonitrile/50.0 mM acetic acid/50.0 mM ammonium acetate = 60/20/20; Flow rate: 1.00 mL/min; UV: 254 nm).

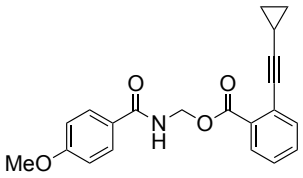
LC-MS analysis

Non-radioactive side-products of the ^{18}F-fluorination reaction for [^{18}F]**31** and [^{18}F]**32** were analysed by HRMS using a reaction mixture prepared with a COSMiC-Mini radiosynthesizer (NMP Business Support, Hyogo, Japan). [^{18}F]Fluoride (approximately 50.0 MBq) trapped on a Sep-Pak Accell Plus QMA Carbonate Plus Light cartridge (washed before use with 1 M $KHCO_3$ 5.00 mL and then water 8.00 mL) was eluted into a reaction vessel using 50% acetonitrile aqueous solution containing $TBAHCO_3$ (37.5 mM, 0.50 mL). After the solvent was dried by heating, the residue was azeotropically dried with acetonitrile (0.50 mL). A mixture of **20a** and **21a** in DMSO (2.00 mg/200 μL) was added into the reaction vessel and heated at 110 °C for 20 min. The reaction mixture was diluted with a 30% acetonitrile aqueous solution (4.00 mL) and then passed through a Sep-Pak C18 Plus Short cartridge (washed before use with ethanol 5.00 mL and water 10.0 mL). After the cartridge was washed with a 30% acetonitrile aqueous solution (4.0 mL), the product was eluted with an 80% acetonitrile aqueous

solution (2.00 mL). LC-MS (electrospray ionization, positive ion mode) analysis of the 80% acetonitrile eluate was conducted using a Q Exactive equipped with an UltiMate 3000 HPLC system (Thermo Fisher Scientific, Waltham, MA). The HPLC was performed under the following conditions: column, Hypersil GOLD C18 (2.1 × 20 mm, 5 μm; Thermo Fisher Scientific); mobile phase, acetonitrile/water/formic acid (50/50/0.1: 0-3 min; 50/50/0.1 to 100/0/0.1: 3-10 min; 100/0/0.1: 10–15 min); flow rate, 0.15 ml/min; and UV, 254 nm.

Deprotection of [^{18}F]31

A deprotection study for [^{18}F]**3** was conducted using a COSMiC-Mini radiosynthesizer with manual interventions. An HPLC fraction containing [^{18}F]**31** was diluted with water (5.00 mL) and then passed through a Sep-Pak C18 Plus Short cartridge (washed before use with ethanol 5.00 mL and water 10.0 mL). After the cartridge was washed with water (10.0 mL), [^{18}F]**31** was eluted into a reaction vessel with an 80% acetonitrile aqueous solution (2.00 mL). The eluate was mixed with 5 M aq. HCl (1.80 mL) and heated at 80 °C for 5 min. The vessel was cooled, and the reaction mixture was diluted with water (10.0 mL) and passed through an Oasis HLB Short cartridge (washed before use with ethanol 5 mL and water 10 mL). After the cartridge was washed with water (10.0 mL), [^{18}F]**18** was eluted with ethanol (2.00 mL) into a product vial containing ascorbic acid injection (250 mg/mL, 2–50 μL). The product solution was analysed by analytical HPLC with a radioactivity detector (Column: PEGASIL C8 SP100, 5 μm, 4.6 × 150 mm [Senshu Scientific, Tokyo, Japan]; Eluent: acetonitrile/water/formic acid = 50/50/0.1 to 100/0/0.1 from 0 min to 5 min, 100/0/0.1 after 5 min; Flow rate: 1.00 mL/min; UV: 254 nm).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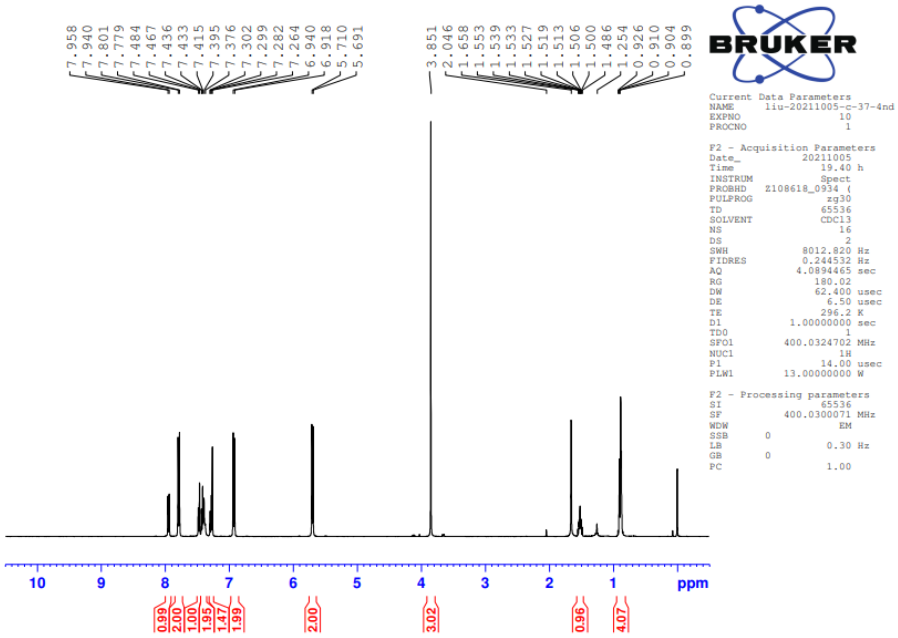
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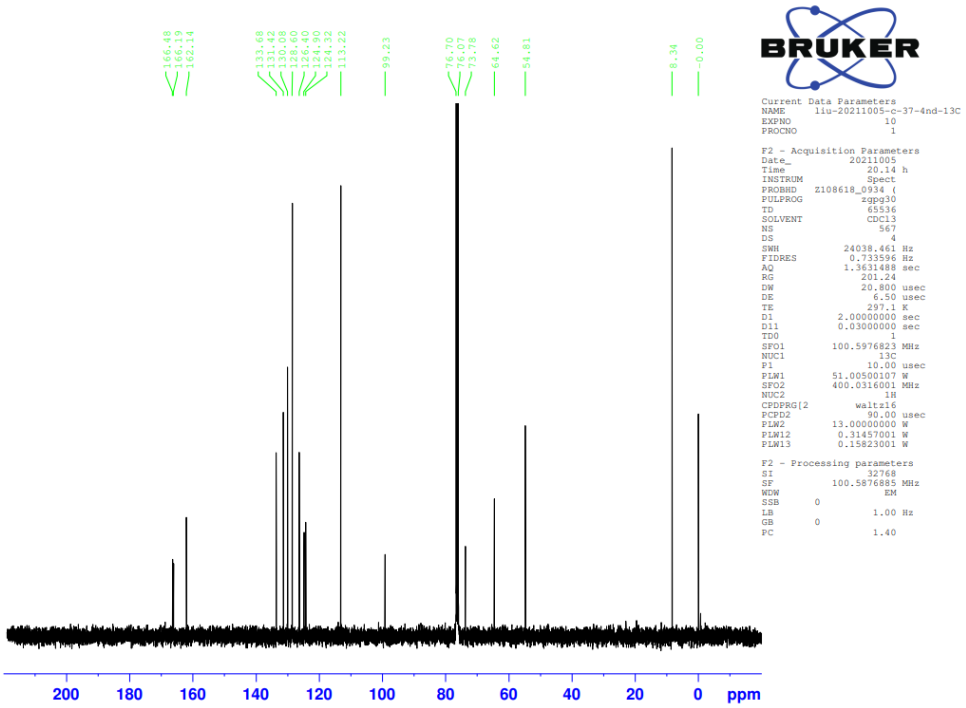
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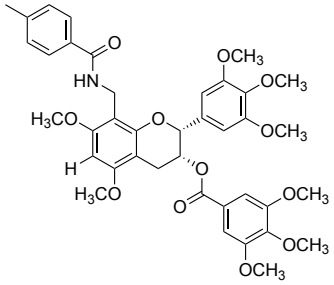
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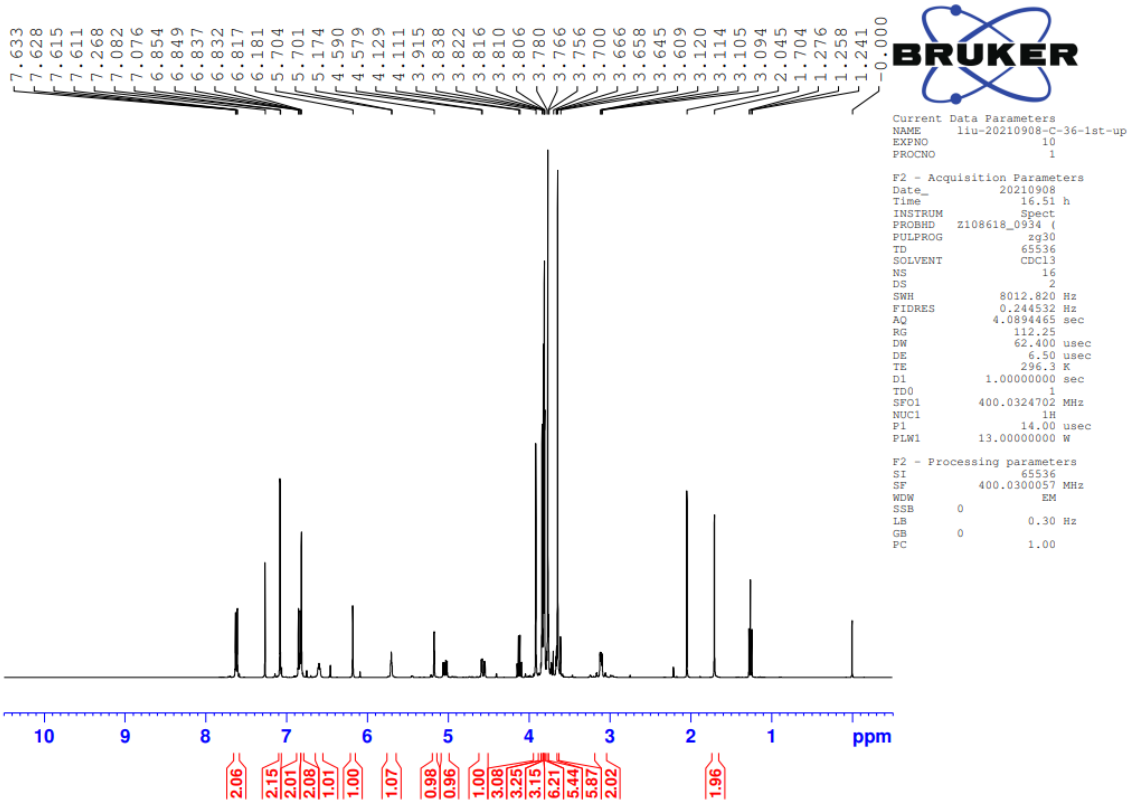
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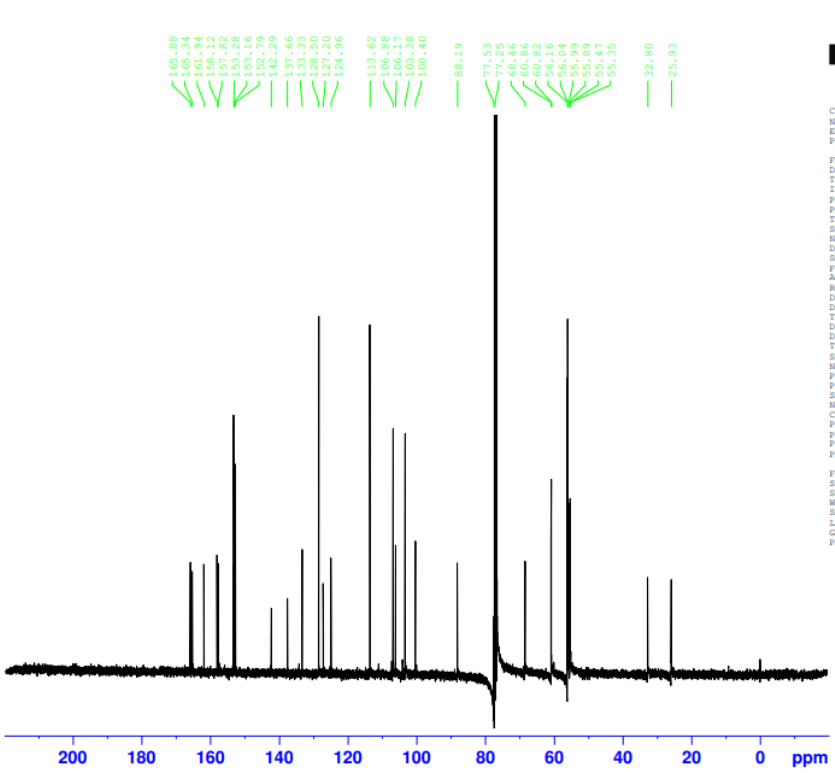
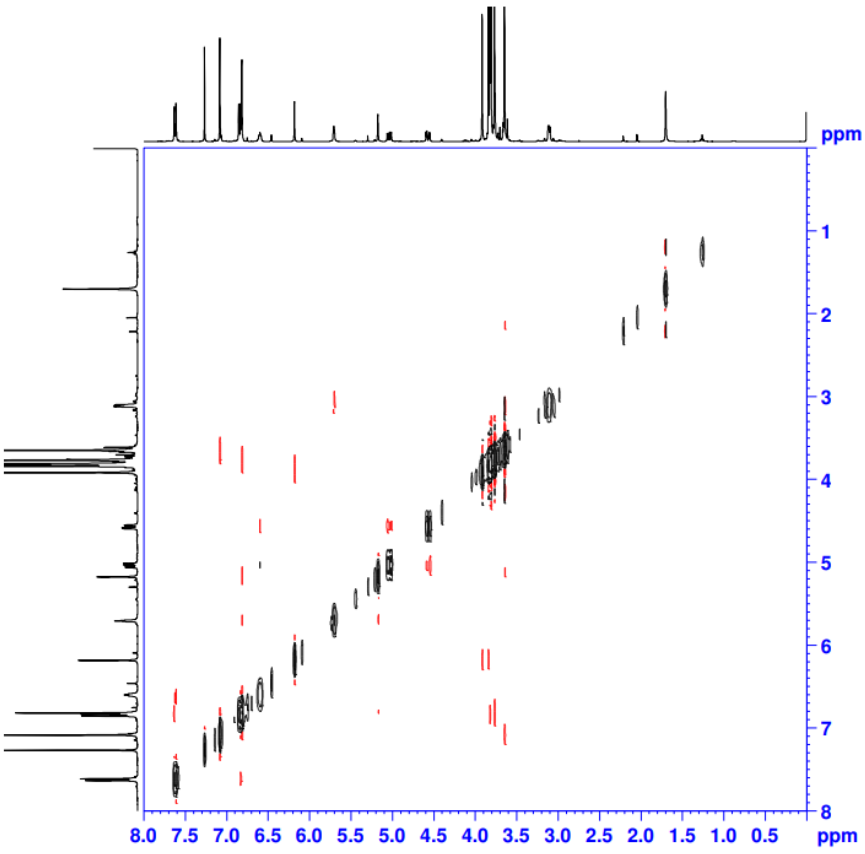
6.181
5.704
5.701
5.174
4.590
4.579
4.129
4.111
3.915
3.838
3.822
3.816
3.810
3.806
3.780
3.766
3.756
3.700
3.666
3.658
3.645
3.609
3.120
3.114
3.105
3.094
2.045
1.704
1.276
1.258
1.241
0.000
BRUKER
Current Data Parameters
NAME liu-20210908-C-36-1st-up
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210908
Time 16.51 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 112.25
DW 62.400 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.0324702 MHz
NUC1 1H
P1 14.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.0300057 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
2.06
2.15
2.01
2.08
1.01
1.00
1.07
0.98
0.96
1.00
3.08
3.25
3.15
6.21
5.44
5.87
2.02
1.96



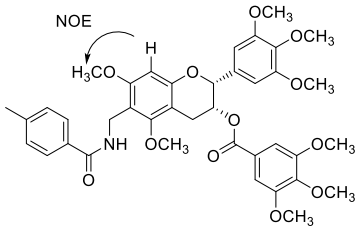
BRUKER

```
Current Data Parameters
NAME      liu-20210908-C-36-1st-up-13C
EXPNO                20
PROCNO                1

F2 - Acquisition Parameters
Date_              20210908
Time                 21.12 h
INSTRUM              Spect
PROBHD   Z108618_0934 (
PULPROG            zgpg30
TD                 655536
SOLVENT             CDCl3
NS                   2048
DS                      4
SWH             24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.36314488 sec
RG                 201.24
DW                20.800 usec
DE                 6.50 usec
TE                297.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1
SFO1           100.5976823 MHz
NUC1                  13C
P1                  10.00 usec
PLW1           51.00500107 W
SFO2           400.0316001 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2           13.00000000 W
PLW12           0.31457001 W
PLW13           0.15823001 W

F2 - Processing parameters
SI                   32768
SF             100.5876235 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```

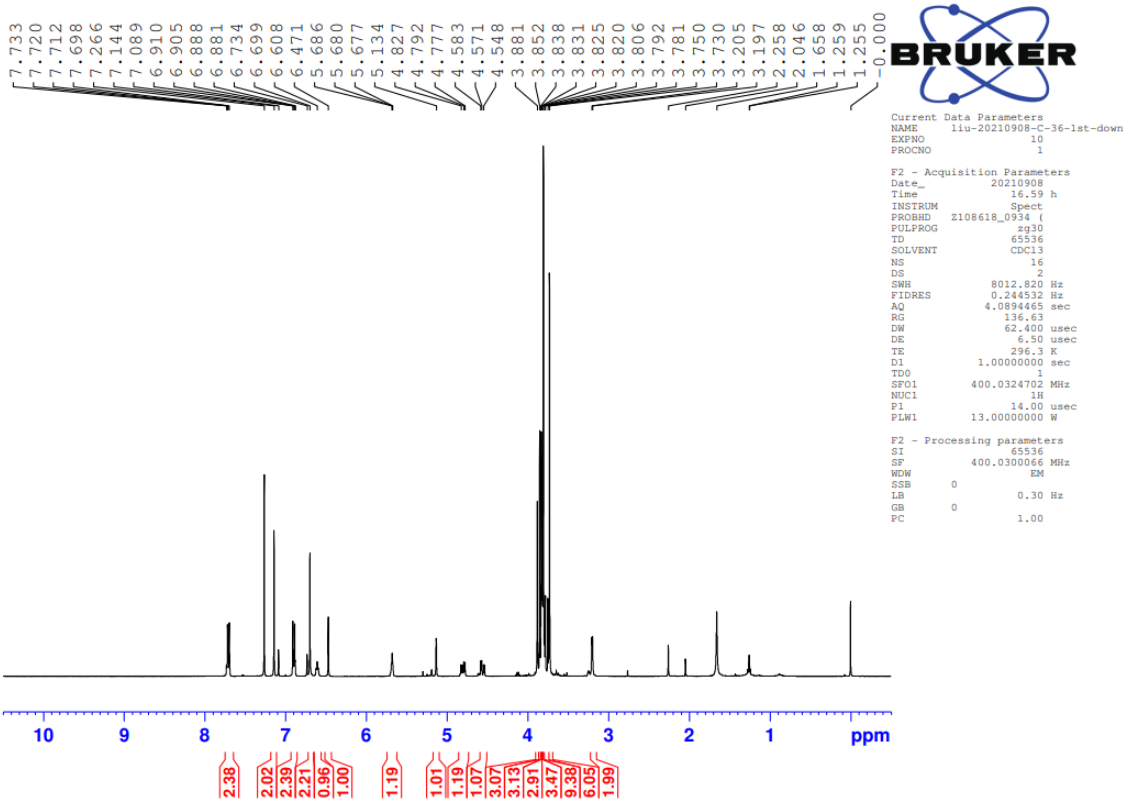


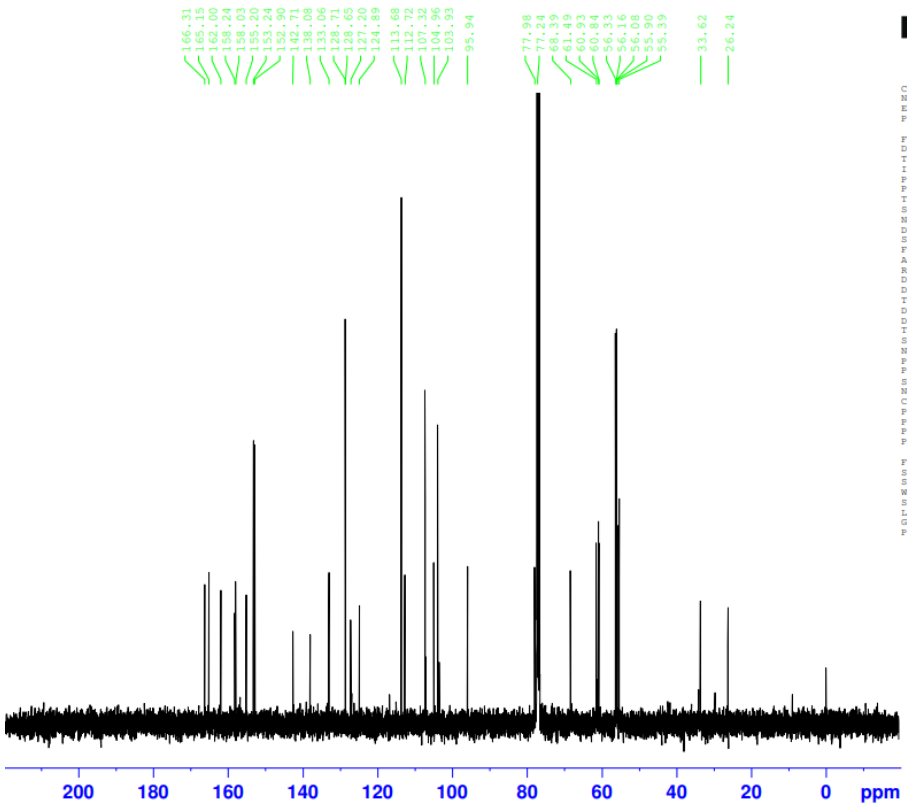
BRUKER

```
Current Data Parameters
NAME      liu-20210908-c-36-1st-up-noesy
EXPNO                20
PROCNO                1

F2 - Acquisition Parameters
Date_              20210908
Time                 21.26 h
INSTRUM              Spect
PROBHD   Z108618_0934 (
PULPROG       noesygpphpp
TD                   2048
SOLVENT             CDCl3
NS                    256
DS                     32
SWH              3424.657 Hz
FIDRES           3.344392 Hz
AQ               0.2990080 sec
RG                  60.03
DW                146.000 usec
DE                   6.50 usec
TE                296.5 K
D0              0.00012817 sec
D1              1.95699203 sec
D8              0.30000001 sec
D11             0.03000000 sec
D12             0.00002000 sec
D16             0.00020000 sec
IN0             0.00029200 sec
TDav                    1
SFO1           400.0318065 MHz
NUC1                  1H
P1                  14.00 usec
P2                  28.00 usec
P17               2500.00 usec
PLW1           13.00000000 W
PLW10           2.83109999 W
GPNAM[1]       SMSQ10.100
GPZ1                40.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                     256
SFO1           400.0318 MHz
FIDRES         91.324203 Hz
SW                 8.561 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                    1024
SF             400.0300064 MHz
WDW                 QSINE
SSB                     2
LB        0 Hz
GB        0
PC                   1.00

F1 - Processing parameters
SI                    1024
MC2          States-TPPI
SF             400.0300064 MHz
WDW                 QSINE
SSB                     2
LB        0 Hz
GB        0
```


NOE
H
H_3CO
OCH_3
OCH_3
OCH_3
O
O
NH
OCH_3
O
OCH_3
OCH_3
OCH_3

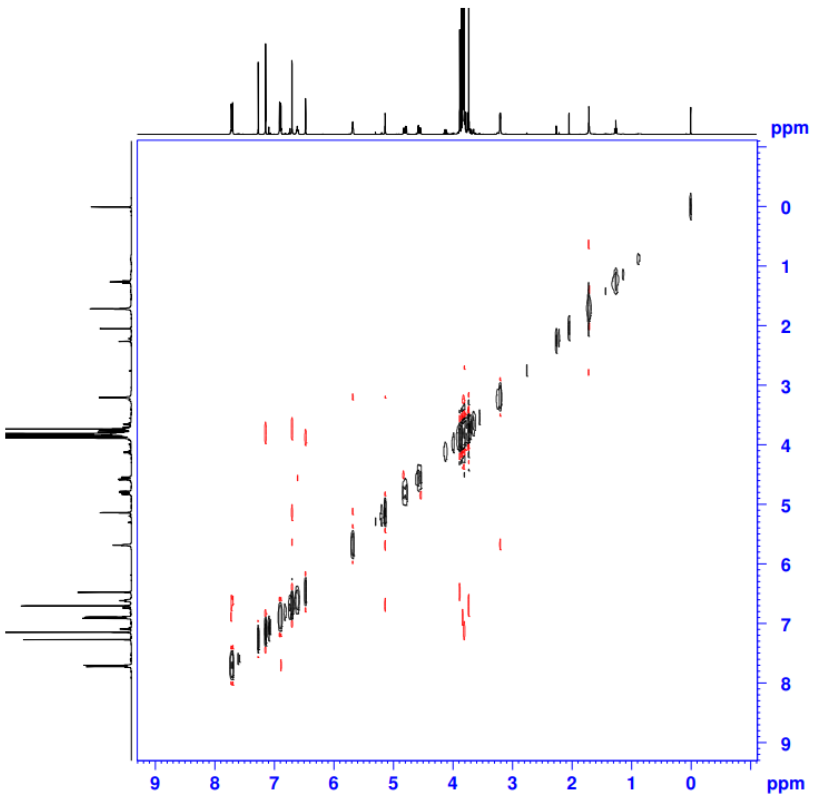
17


7.733
7.720
7.712
7.698
7.266
7.144
7.089
6.910
6.905
6.888
6.881
6.734
6.699
6.608
6.471
5.686
5.680
5.677
5.134
4.827
4.792
4.777
4.583
4.571
4.548
3.881
3.852
3.838
3.831
3.825
3.820
3.806
3.792
3.781
3.750
3.730
3.205
3.197
2.258
2.046
1.658
1.259
1.255
0.000
BRUKER
Current Data Parameters
NAME liu-20210908-C-36-1st-down
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210908
Time 16.59 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 136.63
DW 62.400 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.0324702 MHz
NUC1 1H
P1 14.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.0300066 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
2.38
2.02
2.39
2.21
0.96
1.00
1.19
1.01
1.19
1.07
3.07
3.13
2.91
3.47
9.38
6.05
1.99



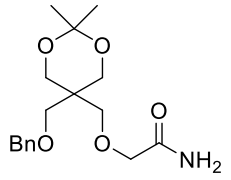
BRUKER

```
Current Data Parameters
NAME          liu-20210908-C-36-1st-down-13C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210908
Time              18.37 h
INSTRUM             Spect
PROBHD   Z108618_0934 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG               201.24
DW               20.800 usec
DE                6.50 usec
TE              297.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1
SFO1          100.5976823 MHz
NUC1                13C
P1                10.00 usec
PLW1          51.00500107 W
SFO2          400.0316001 MHz
NUC2                1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2          13.00000000 W
PLW12          0.31457001 W
PLW13          0.15823001 W

F2 - Processing parameters
SI                 32768
SF            100.5876235 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

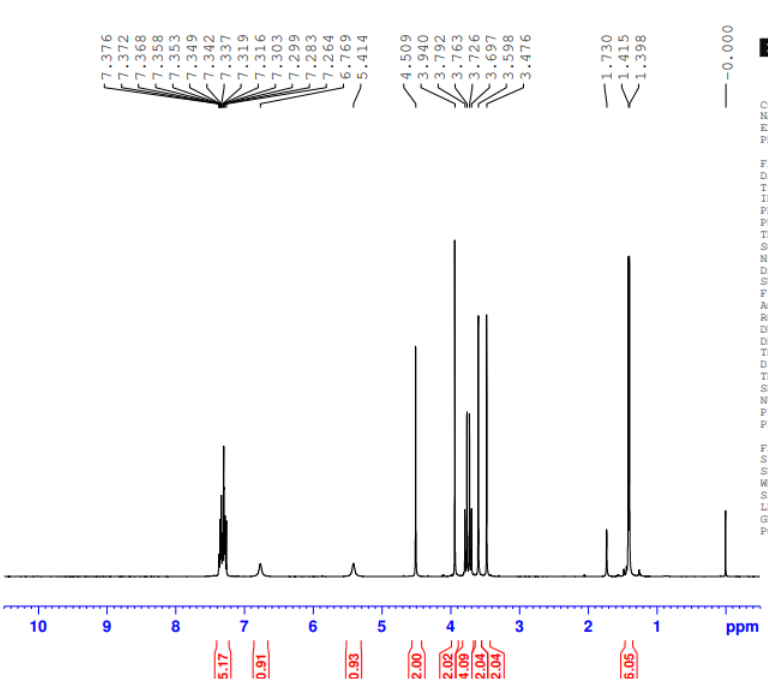


BRUKER

```
Current Data Parameters
NAME    liu-20210909-c-36-1st-down-noesy-overnight
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210909
Time              20.02 h
INSTRUM             Spect
PROBHD   Z108618_0934 (
PULPROG       noesygpphpp
TD                 2048
SOLVENT           CDCl3
NS                  256
DS                   32
SWH           4166.667 Hz
FIDRES        4.069010 Hz
AQ            0.2457600 sec
RG                60.63
DW              120.000 usec
DE                6.50 usec
TE              296.3 K
D0            0.00010217 sec
D1            2.01024008 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00024001 sec
TDav                  1
SFO1          400.0316441 MHz
NUC1                 1H
P1                14.00 usec
P2                28.00 usec
P17             2500.00 usec
PLW1          13.00000000 W
PLW10         2.83109999 W
GPNAM[1]         SMSQ10.100
GPZ1              40.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                   81
SFO1          400.0316 MHz
FIDRES        102.880661 Hz
SW               10.416 ppm
FnMODE        States-TPPI

F2 - Processing parameters
SI                 1024
SF            400.0300056 MHz
WDW                QSINE
SSB                   2
LB        0 Hz
GB        0
PC                 1.00

F1 - Processing parameters
SI                 1024
MC2           States-TPPI
SF            400.0300056 MHz
WDW                QSINE
SSB                   2
LB        0 Hz
GB        0
```

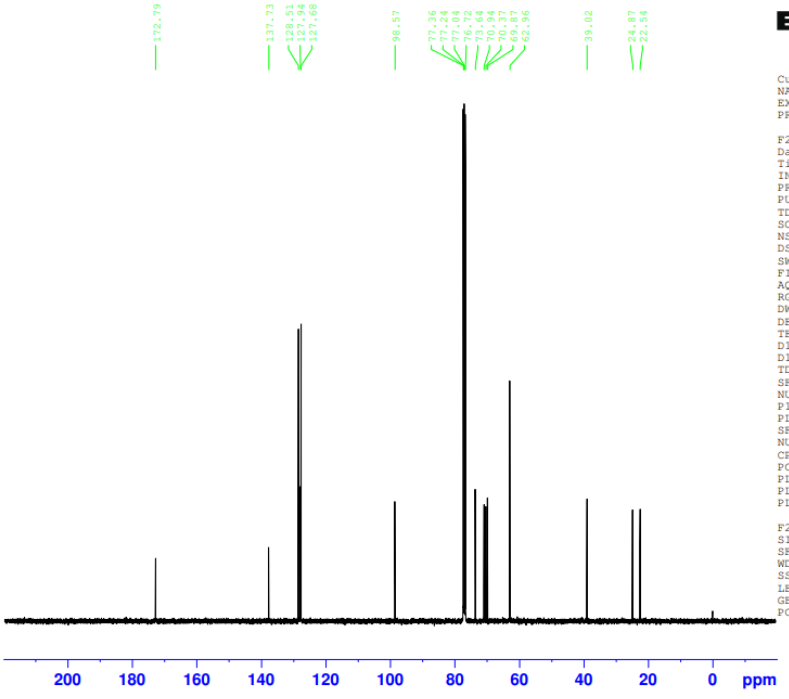


25



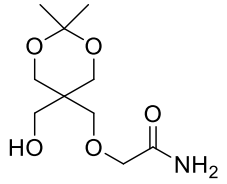
BRUKER

```
Current Data Parameters
NAME      liu-20210401-d-1H
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_              20210401
Time                 16.14 h
INSTRUM              Spect
PROBHD    Z108618_0934 (
PULPROG              zg30
TD                  65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8012.820 Hz
FIDRES               0.244532 Hz
AQ                4.0894465 sec
RG                   121.35
DW                   62.400 usec
DE                    6.50 usec
TE                   296.3 K
D1                1.00000000 sec
TD0                      1
SFO1            400.0324702 MHz
NUC1                    1H
P1                   14.00 usec
PLW1           13.00000000 W

F2 - Processing parameters
SI                    65536
SF              400.0300073 MHz
WDW                     EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.00
```

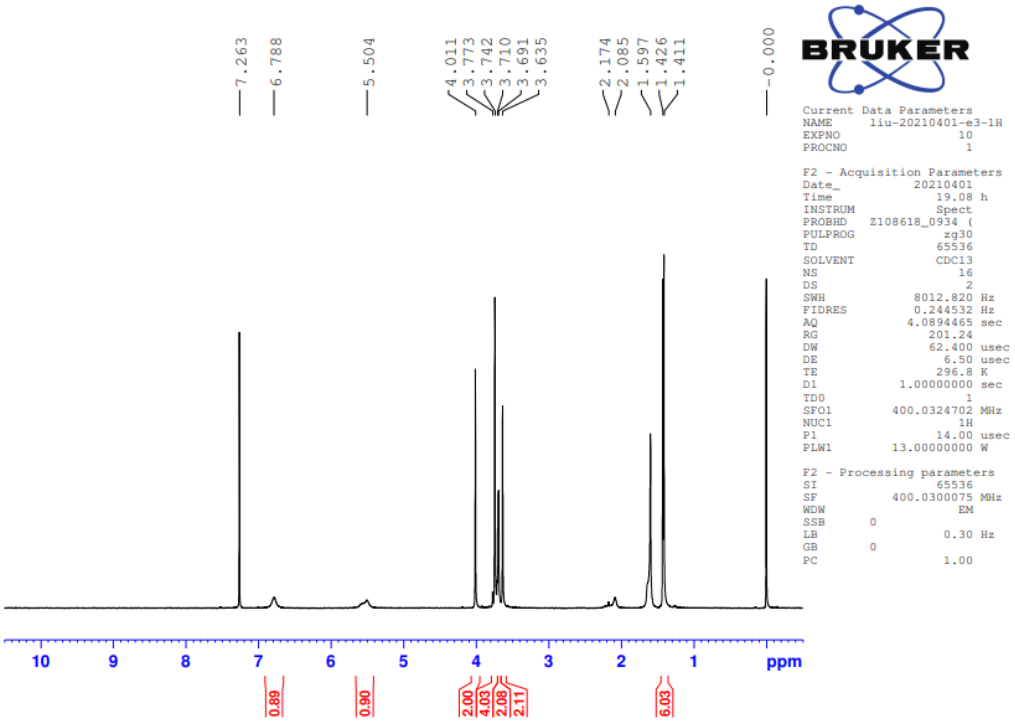


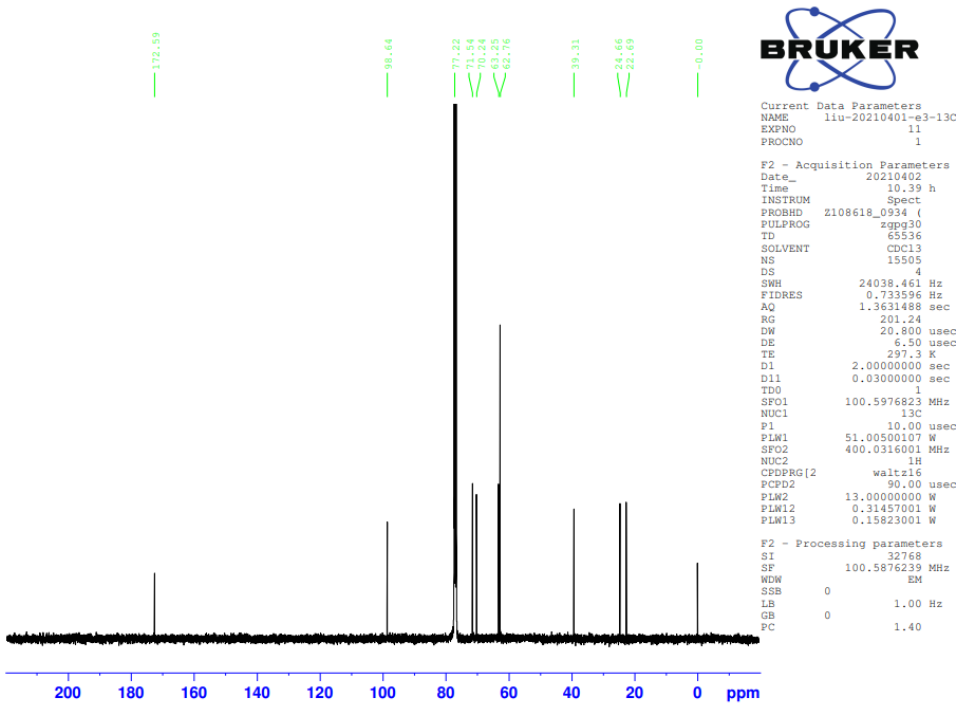
BRUKER

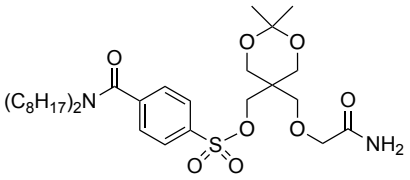
```
Current Data Parameters
NAME      liu-20210401-d-13C
EXPNO                   20
PROCNO                   1

F2 - Acquisition Parameters
Date_              20210401
Time                 19.02 h
INSTRUM              Spect
PROBHD    Z108618_0934 (
PULPROG            zgpg30
TD                  65536
SOLVENT              CDCl3
NS                     866
DS                       4
SWH              24038.461 Hz
FIDRES               0.733596 Hz
AQ                1.3631488 sec
RG                   201.24
DW                   20.800 usec
DE                    6.50 usec
TE                   297.4 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1
SFO1            100.5976823 MHz
NUC1                    13C
P1                   10.00 usec
PLW1           51.00500107 W
SFO2            400.0316001 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                90.00 usec
PLW2           13.00000000 W
PLW12           0.31457001 W
PLW13           0.15823001 W

F2 - Processing parameters
SI                    32768
SF              100.5876235 MHz
WDW                     EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
```


O
O
HO
O
O
NH2

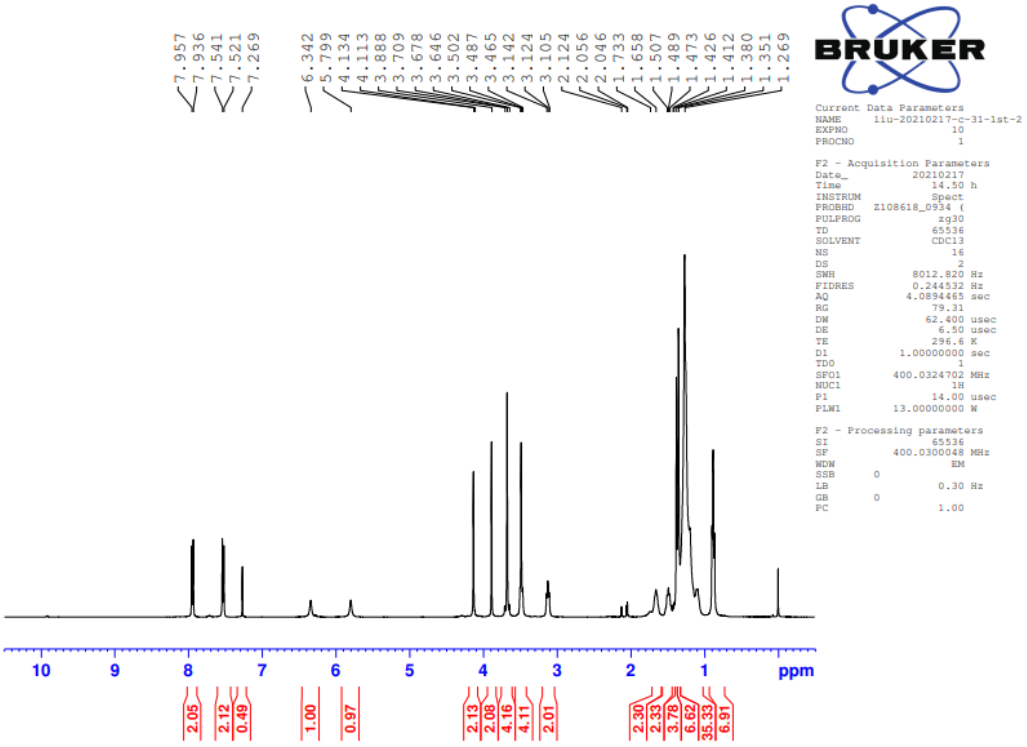
26

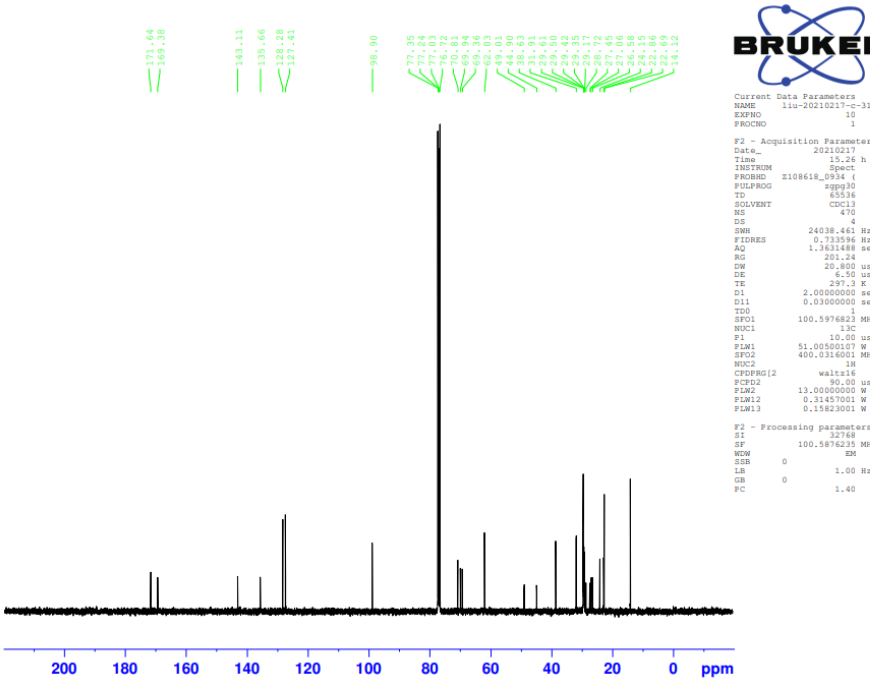

7.263
6.788
5.504
4.011
3.773
3.742
3.710
3.691
3.635
2.174
2.085
1.597
1.426
1.411
0.000
BRUKER
Current Data Parameters
NAME liu-20210401-e3-1H
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210401
Time 19.08 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 201.24
DW 62.400 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1
SFO1 400.0324702 MHz
NUC1 1H
P1 14.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.0300075 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
0.89
0.90
2.00
4.03
2.08
2.11
6.03

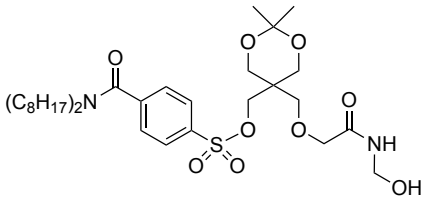

172.59
98.64
77.22
71.54
70.34
63.26
62.76
39.31
24.66
22.69
0.00
BRUKER
Current Data Parameters
NAME liu-20210401-e3-13C
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210402
Time 10.39 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 15505
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 201.24
DW 20.800 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5976823 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.00500107 W
SFO2 400.0316001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.31457001 W
PLW13 0.15823001 W
F2 - Processing parameters
SI 32768
SF 100.5876239 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


(C8H17)2N
O
O
O
O
O
O
S
O
O
O
NH2

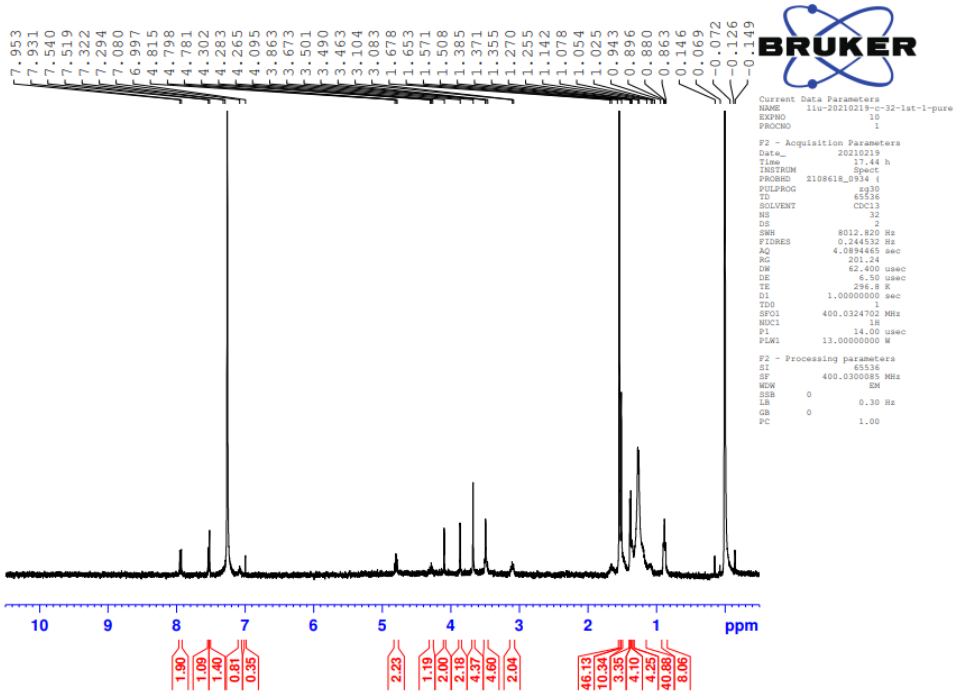
28

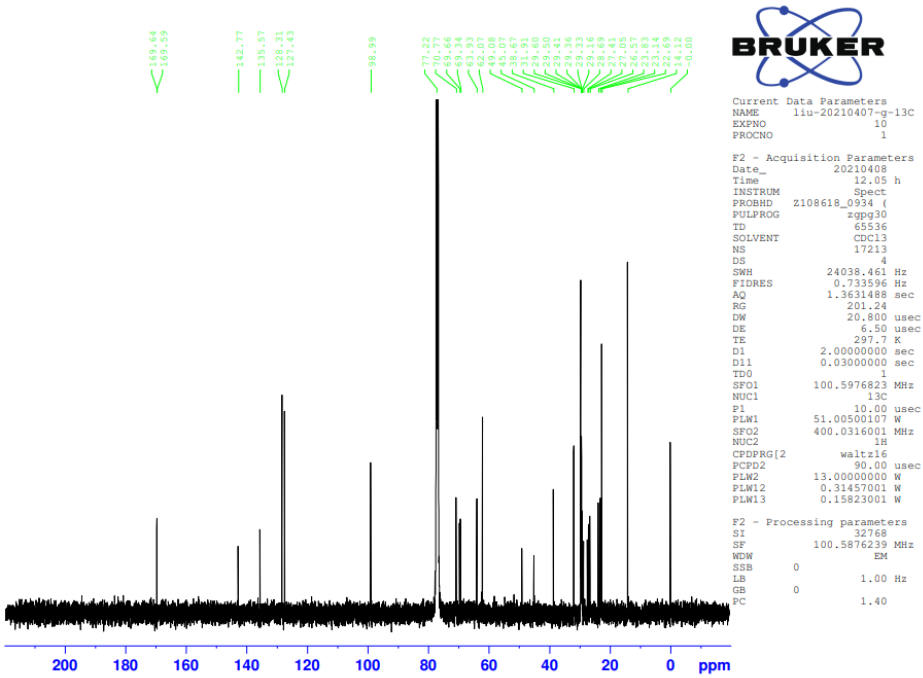

7.957
7.936
7.541
7.521
7.269
6.342
5.799
4.134
4.113
3.888
3.709
3.678
3.646
3.502
3.487
3.465
3.142
3.124
3.105
2.124
2.056
2.046
1.733
1.658
1.507
1.489
1.473
1.426
1.412
1.380
1.351
1.269
BRUKER
Current Data Parameters
NAME liu-20210217-c-31-1st-2
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210217
Time 14.50 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 79.31
DW 62.400 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
SFO1 400.0324702 MHz
NUC1 1H
P1 14.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.0300048 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
2.05
2.12
0.49
1.00
0.97
2.13
2.08
4.16
4.11
2.01
2.30
2.33
3.78
6.62
35.33
6.91

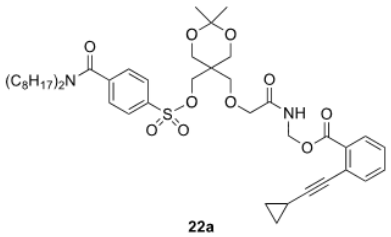

171.64
169.38
143.11
135.66
128.28
127.41
98.90
77.35
77.04
76.72
70.81
69.94
69.46
62.03
49.01
44.90
38.63
31.21
29.61
29.30
29.42
29.35
29.17
28.72
27.45
27.06
25.68
24.15
22.86
22.69
14.12
BRUKER
Current Data Parameters
NAME liu-20210217-c-31-1st-2-13C
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210217
Time 15.26 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 470
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 201.24
DW 20.800 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5976823 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.00500107 W
SFO2 400.0316001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.31457001 W
PLW13 0.15823001 W
F2 - Processing parameters
SI 32768
SF 100.5876235 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


(C8H17)2N
O
O
O
O
O
S
O
O
O
NH
OH

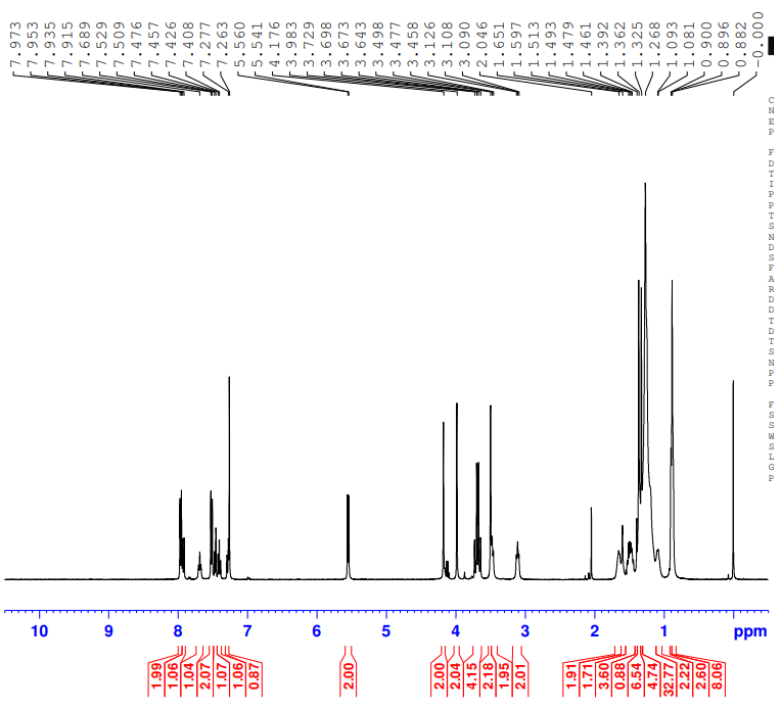
29


7.953
7.931
7.540
7.519
7.322
7.294
7.080
6.997
4.815
4.798
4.781
4.302
4.283
4.265
4.095
3.863
3.673
3.501
3.490
3.463
3.104
3.083
1.678
1.653
1.571
1.508
1.385
1.371
1.355
1.270
1.255
1.142
1.078
1.054
1.025
0.943
0.896
0.880
0.863
0.146
0.069
-0.072
-0.126
-0.149
BRUKER
Current Data Parameters
NAME liu-20210219-c-32-1st-1-pure
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210219
Time 17.44 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 201.24
DW 62.400 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1
SFO1 400.0324702 MHz
NUC1 1H
P1 14.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.0300085 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.90
1.09
1.40
0.61
0.35
2.23
1.19
2.00
2.18
4.37
4.60
2.04
46.13
10.24
3.35
4.10
4.25
40.88
8.06

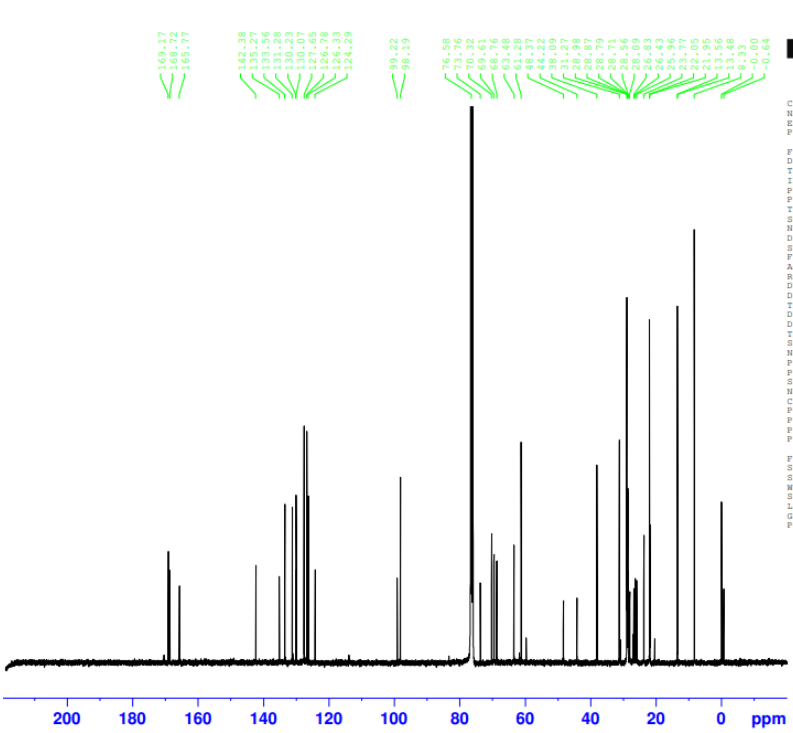

169.64
169.59
142.77
135.57
128.31
127.43
98.99
77.22
70.77
69.66
69.34
69.03
62.07
49.08
45.07
38.67
33.21
29.60
29.50
29.41
29.36
29.33
29.16
28.69
27.41
27.35
26.57
23.83
23.14
14.19
14.02
0.00
BRUKER
Current Data Parameters
NAME liu-20210407-g-13C
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210408
Time 12.05 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 17213
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 201.24
DW 20.800 usec
DE 6.50 usec
TE 297.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5976823 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.00500107 W
SFO2 400.0316001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.31457001 W
PLW13 0.15823001 W
F2 - Processing parameters
SI 32768
SF 100.5876239 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm



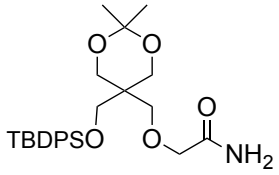
22a



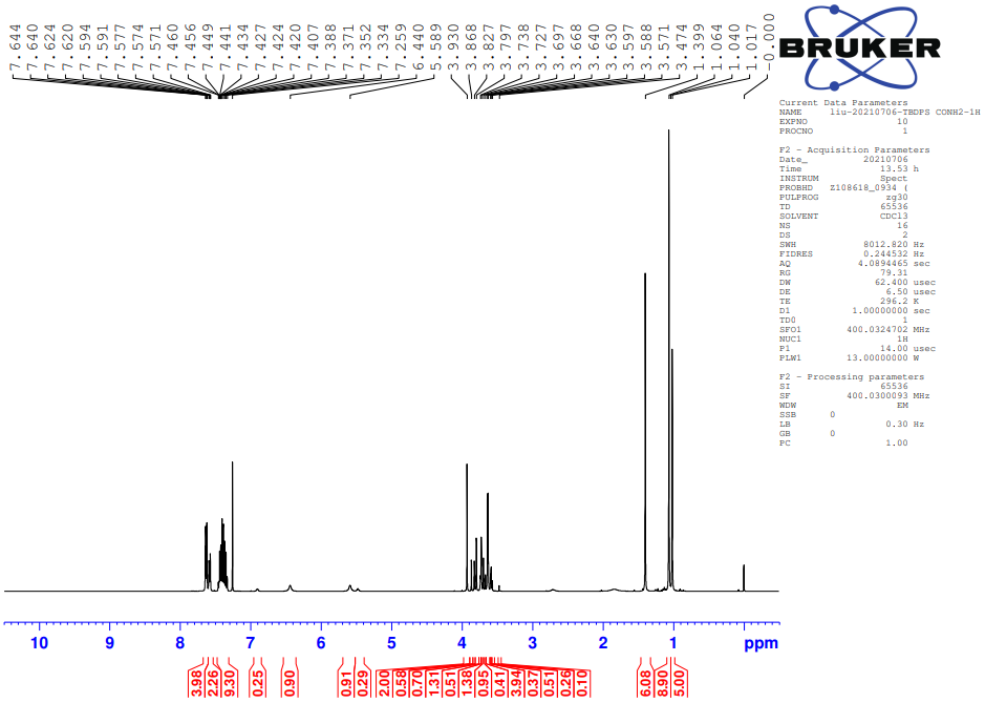

```
Current Data Parameters
NAME      liu-20210222-c-33-1st-3
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20210222
Time               17.41 h
INSTRUM              Spect
PROBHD    Z108618_0934 (
PULPROG              zg30
TD                  65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ               4.0894465 sec
RG                  157.94
DW                 62.400 usec
DE                  6.50 usec
TE                 296.9 K
D1              1.00000000 sec
TD0                     1
SFO1           400.0324702 MHz
NUC1                   1H
P1                  14.00 usec
PLW1           13.00000000 W

F2 - Processing parameters
SI                  65536
SF             400.0300073 MHz
WDW                    EM
SSB          0
LB                   0.30 Hz
GB           0
PC                   1.00
```

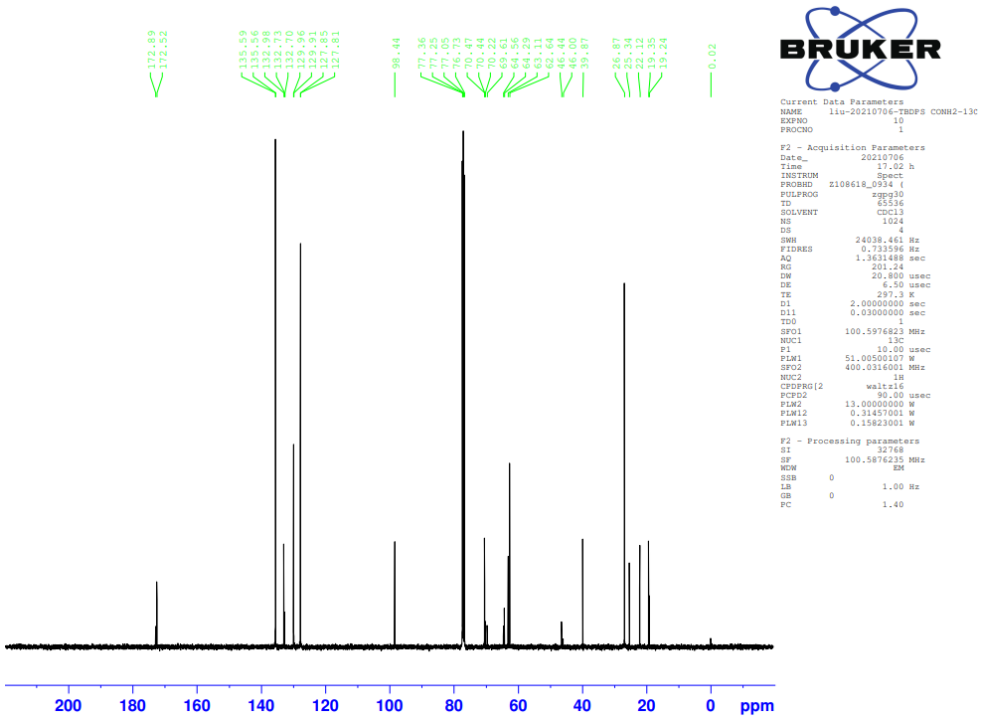


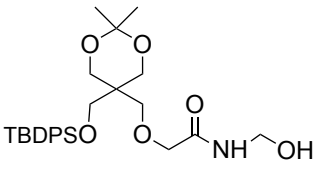

```
Current Data Parameters
NAME      liu-20210222-c-33-1st-3-13C
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20210224
Time               10.39 h
INSTRUM              Spect
PROBHD    Z108618_0934 (
PULPROG           zgpg30
TD                  65536
SOLVENT              CDCl3
NS                   43042
DS                       4
SWH             24038.461 Hz
FIDRES           0.733596 Hz
AQ               1.3631488 sec
RG                  201.24
DW                 20.800 usec
DE                  6.50 usec
TE                 297.8 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1
SFO1           100.5976823 MHz
NUC1                   13C
P1                  10.00 usec
PLW1           51.00500107 W
SFO2           400.0316001 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2           13.00000000 W
PLW12           0.31457001 W
PLW13           0.15823001 W

F2 - Processing parameters
SI                  32768
SF             100.5876881 MHz
WDW                    EM
SSB          0
LB                   1.00 Hz
GB           0
PC                   1.40
```


TBDPSO
O
O
O
O
NH2

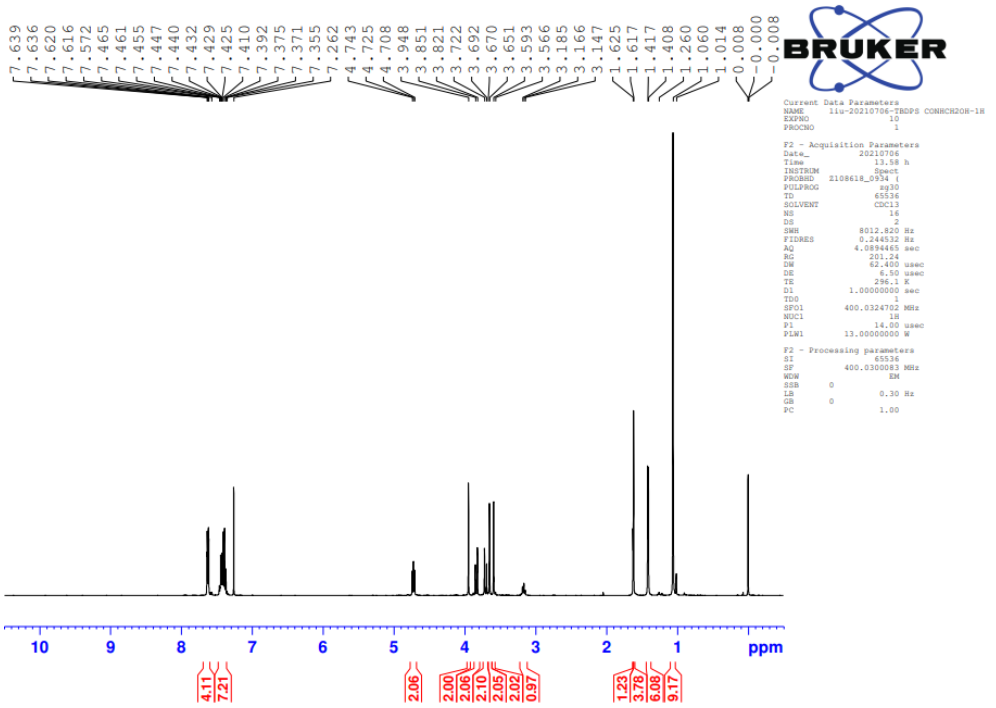
S1

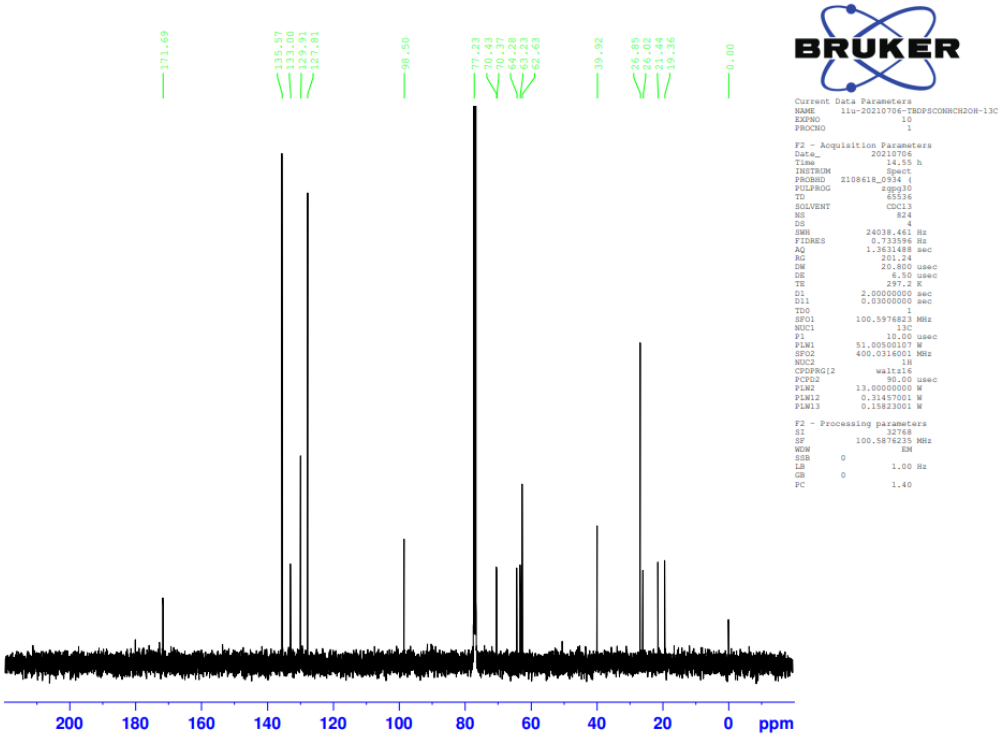

7.644
7.640
7.624
7.620
7.594
7.591
7.577
7.574
7.571
7.460
7.456
7.449
7.441
7.434
7.427
7.424
7.420
7.407
7.388
7.371
7.352
7.334
7.259
6.440
5.589
3.930
3.868
3.827
3.797
3.738
3.727
3.697
3.668
3.640
3.630
3.597
3.588
3.571
3.474
1.399
1.064
1.040
1.017
0.000
BRUKER
Current Data Parameters
NAME liu-20210706-TBDPS CONH2-1H
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210706
Time 13.53 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 79.31
DW 62.400 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.0324702 MHz
NUC1 1H
P1 14.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.0300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
3.98
2.26
9.30
0.25
0.90
0.91
0.29
2.00
0.58
0.70
1.31
0.51
1.38
0.95
0.47
3.94
0.37
0.51
0.26
0.10
6.08
8.90
5.00

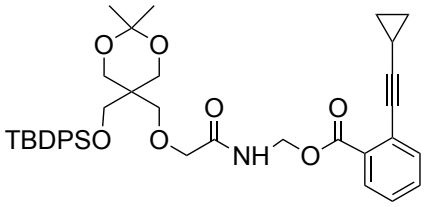

172.89
172.52
135.59
135.56
132.98
132.73
132.70
129.96
129.90
127.85
127.81
98.44
77.36
77.25
77.05
76.73
70.47
70.44
70.22
69.61
64.56
64.29
63.11
63.04
46.44
46.00
39.87
26.87
25.34
22.12
19.35
19.24
0.02
BRUKER
Current Data Parameters
NAME liu-20210706-TBDPS CONH2-13C
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210706
Time 17.02 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 201.24
DW 20.800 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5976823 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.00500107 W
SFO2 400.0316001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.31457001 W
PLW13 0.15823001 W
F2 - Processing parameters
SI 32768
SF 100.5876235 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


TBDPSO
O
O
O
NH
OH

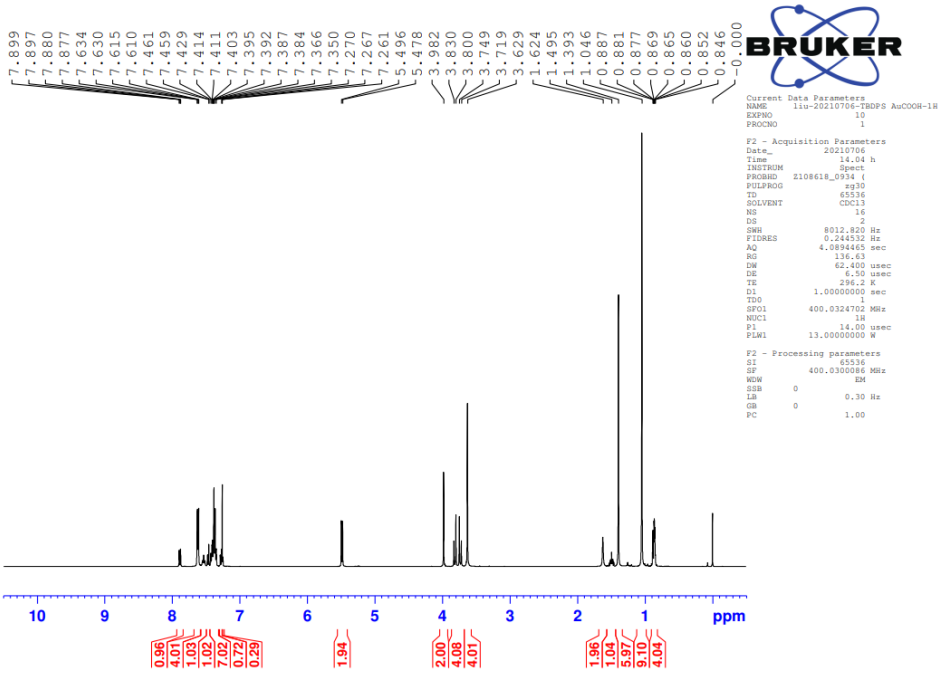
S2

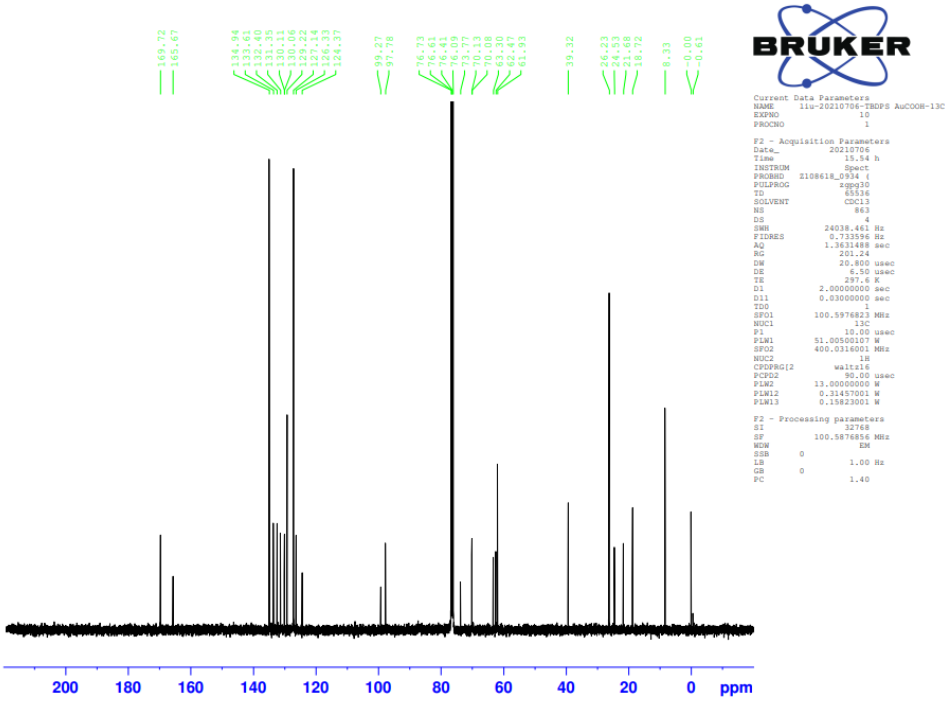

7.639
7.636
7.620
7.616
7.572
7.465
7.461
7.455
7.447
7.440
7.432
7.429
7.425
7.410
7.392
7.375
7.371
7.355
7.262
4.743
4.725
4.708
3.948
3.851
3.821
3.722
3.692
3.670
3.651
3.593
3.566
3.185
3.166
3.147
1.625
1.617
1.417
1.408
1.260
1.060
1.014
0.008
0.000
-0.008
BRUKER
Current Data Parameters
NAME 1iu-20210706-TBDPS CONHCH2OH-1H
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210706
Time 13.58 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 201.24
DW 62.400 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.0324702 MHz
NUC1 1H
P1 14.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.0300083 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
4.11
7.21
2.06
2.00
2.06
2.10
2.05
2.02
0.97
1.23
3.78
6.08
9.17

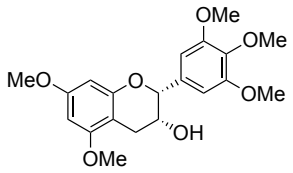

171.69
135.57
133.00
129.91
127.81
98.50
77.23
70.43
70.37
64.28
63.23
62.63
39.92
26.85
26.02
21.44
19.36
0.00
BRUKER
Current Data Parameters
NAME 1iu-20210706-TBDPSCONHCH2OH-13C
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210706
Time 14.55 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 824
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 201.24
DW 20.800 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5976823 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.00500107 W
SFO2 400.0316001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.31457001 W
PLW13 0.15823001 W
F2 - Processing parameters
SI 32768
SF 100.5876235 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


TBDPSO
O
O
O
O
NH
O
O

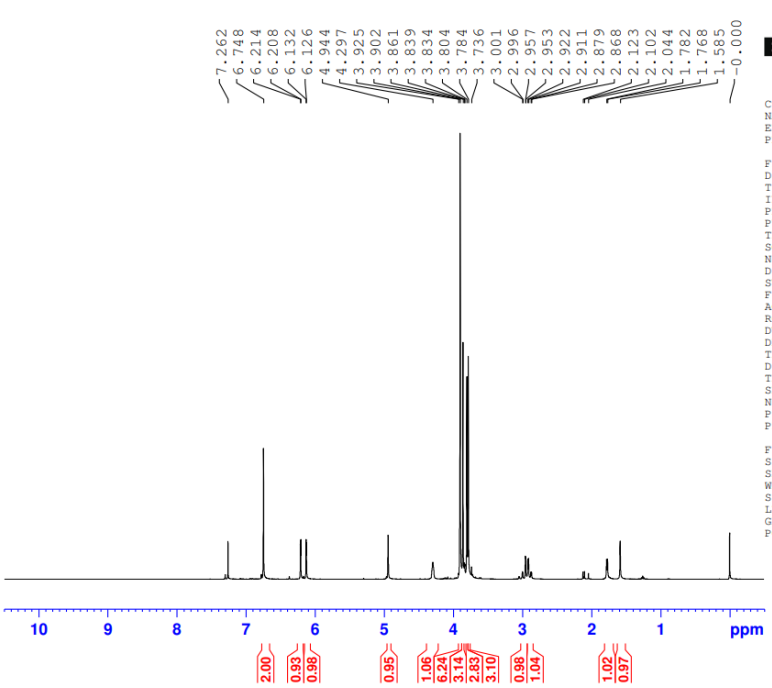
22b


7.899
7.897
7.880
7.877
7.634
7.630
7.615
7.610
7.461
7.459
7.429
7.414
7.411
7.403
7.395
7.392
7.387
7.384
7.366
7.350
7.270
7.267
7.261
5.496
5.478
3.982
3.830
3.800
3.749
3.719
3.629
1.624
1.495
1.393
1.046
0.887
0.881
0.877
0.869
0.865
0.860
0.852
0.846
0.000
BRUKER
Current Data Parameters
NAME liu-20210706-TBDPS AuCOOH-1H
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210706
Time 14.04 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 136.63
DW 62.400 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.0324702 MHz
NUC1 1H
P1 14.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.0300086 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
0.96
4.01
1.03
1.02
7.02
0.72
0.29
1.94
2.00
4.08
4.01
1.96
1.04
5.97
9.10
4.04


169.72
165.67
134.94
133.61
132.40
131.35
130.01
130.00
129.22
127.14
126.33
124.37
99.27
97.78
76.73
76.41
76.09
73.77
70.13
70.06
63.30
62.47
61.93
39.32
26.23
24.53
21.68
18.72
0.33
-0.00
-0.61
BRUKER
Current Data Parameters
NAME liu-20210706-TBDPS AuCOOH-13C
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210706
Time 15.54 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 863
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 201.24
DW 20.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5976823 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.00500107 W
SFO2 400.0316001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.31457001 W
PLW13 0.15823001 W
F2 - Processing parameters
SI 32768
SF 100.5876856 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

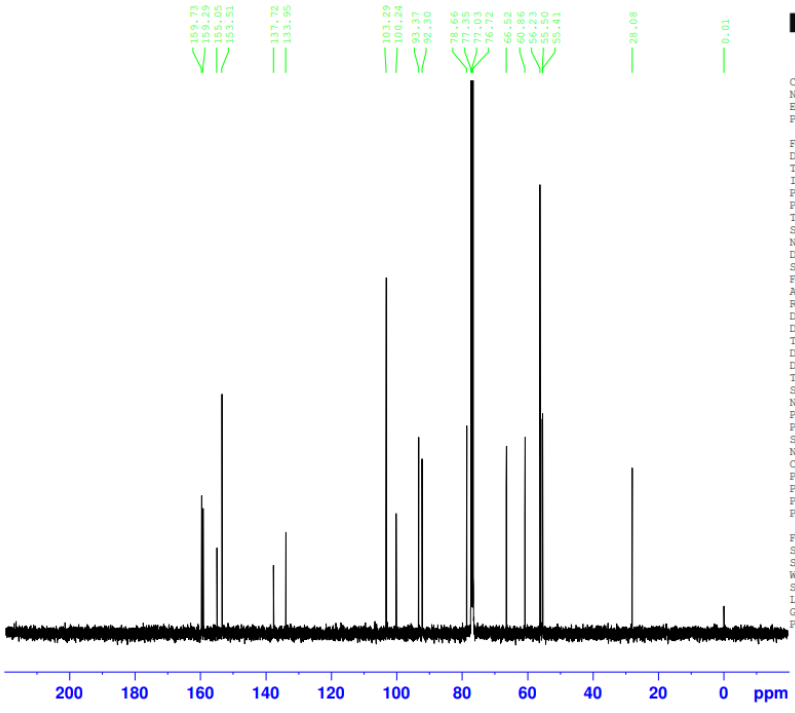


S3



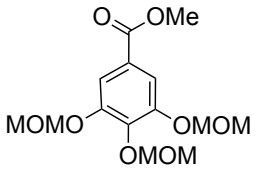
BRUKER

```
Current Data Parameters
NAME      liu-20210406-k5-1H
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_           20210406
Time              12.01 h
INSTRUM             Spect
PROBHD   Z108618_0934 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ               4.0894465 sec
RG                  157.94
DW                62.400 usec
DE                 6.50 usec
TE                296.2 K
D1             1.00000000 sec
TD0                     1
SFO1          400.0324702 MHz
NUC1                   1H
P1                 14.00 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                  65536
SF            400.0300087 MHz
WDW                    EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

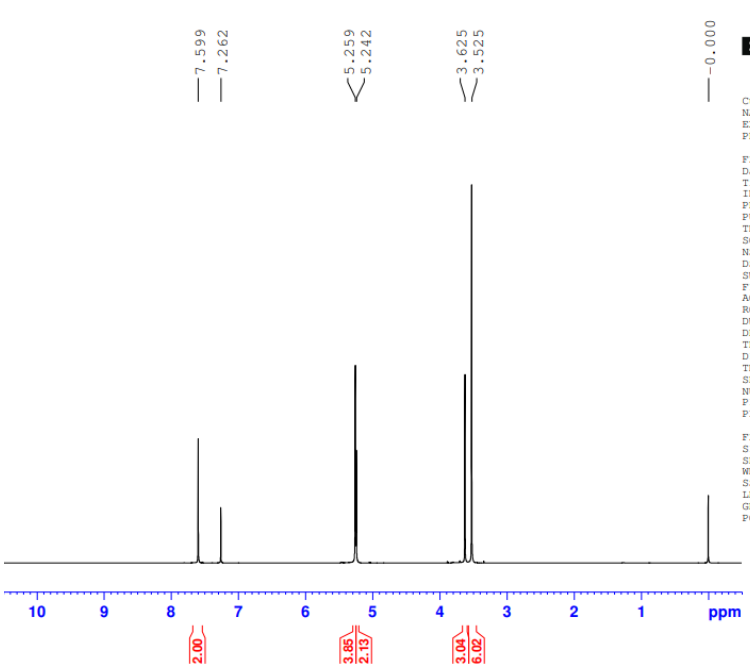


BRUKER

```
Current Data Parameters
NAME      liu-20210406-k5-13C
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_           20210406
Time              13.01 h
INSTRUM             Spect
PROBHD   Z108618_0934 (
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH             24038.461 Hz
FIDRES           0.733596 Hz
AQ               1.3631488 sec
RG                  201.24
DW                20.800 usec
DE                 6.50 usec
TE                297.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1
SFO1          100.5976823 MHz
NUC1                   13C
P1                 10.00 usec
PLW1          51.00500107 W
SFO2          400.0316001 MHz
NUC2                   1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          13.00000000 W
PLW12          0.31457001 W
PLW13          0.15823001 W

F2 - Processing parameters
SI                  32768
SF            100.5876252 MHz
WDW                    EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

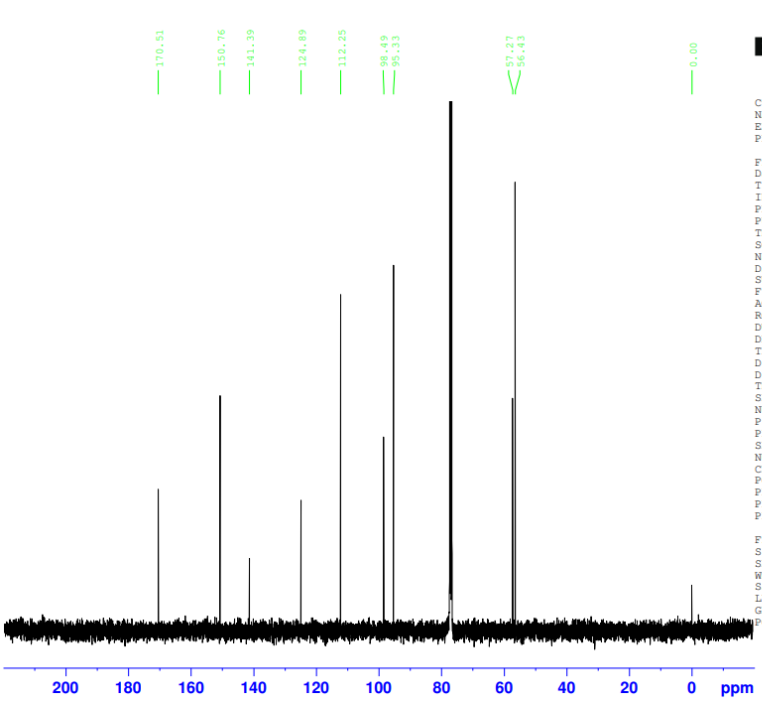


S4



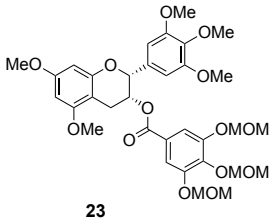
BRUKER

```
Current Data Parameters
NAME      liu-20210401-m-1H
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_              20210402
Time                12.07 h
INSTRUM              Spect
PROBHD    Z108618_0934 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ               4.0894465 sec
RG                  201.24
DW                62.400 usec
DE                 6.50 usec
TE                296.7 K
D1             1.00000000 sec
TD0                     1
SFO1          400.0324702 MHz
NUC1                   1H
P1                 14.00 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                  65536
SF            400.0300081 MHz
WDW                    EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

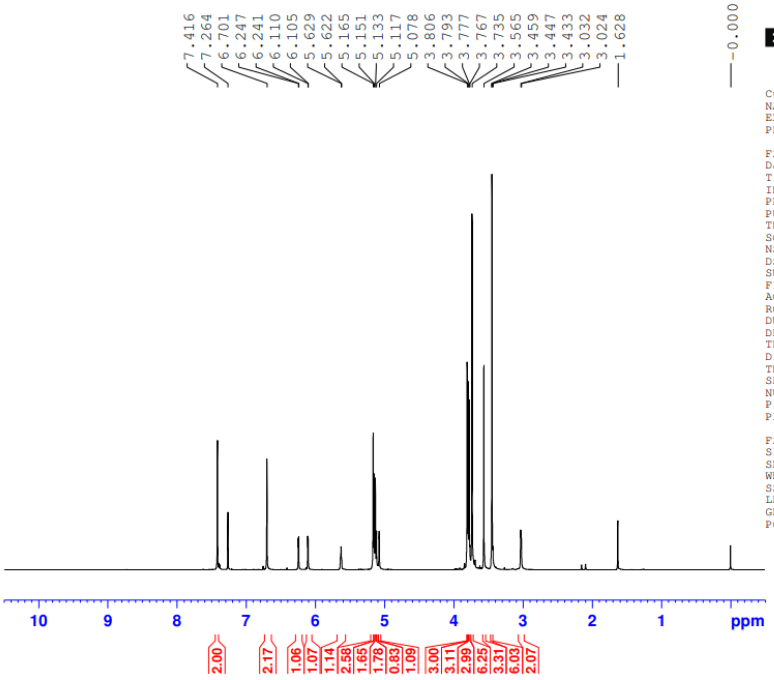


BRUKER

```
Current Data Parameters
NAME      liu-20210406-m4-13C
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_              20210406
Time                14.11 h
INSTRUM              Spect
PROBHD    Z108618_0934 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH             24038.461 Hz
FIDRES           0.733596 Hz
AQ               1.3631488 sec
RG                  201.24
DW                20.800 usec
DE                 6.50 usec
TE                297.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1
SFO1          100.5976823 MHz
NUC1                  13C
P1                10.00 usec
PLW1          51.00500107 W
SFO2          400.0316001 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2             90.00 usec
PLW2          13.00000000 W
PLW12          0.31457001 W
PLW13          0.15823001 W

F2 - Processing parameters
SI                  32768
SF            100.5876235 MHz
WDW                    EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

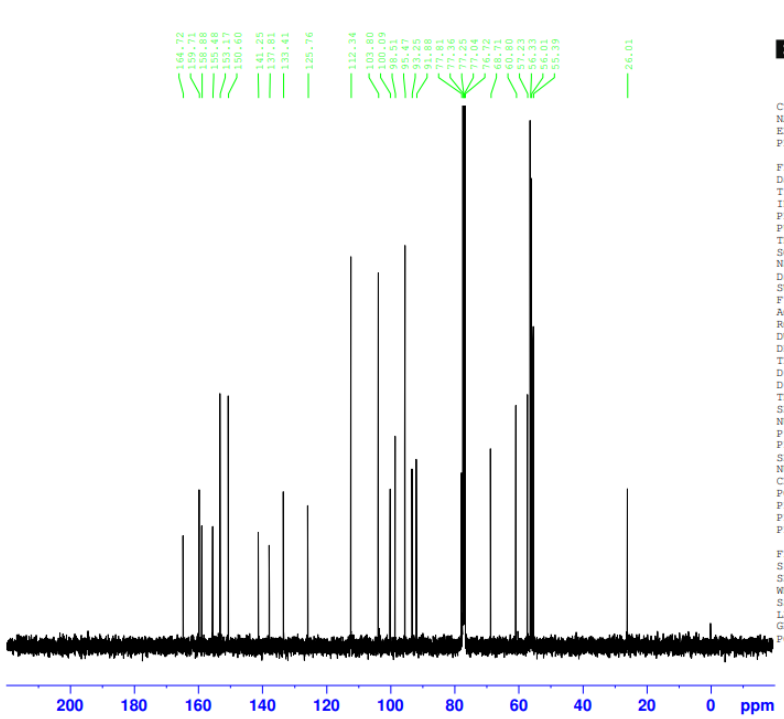


23



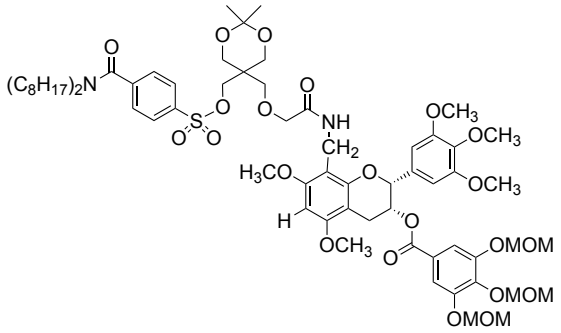
BRUKER

```
Current Data Parameters
NAME      liu-0406-n2-1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210406
Time              19.16 h
INSTRUM             Spect
PROBHD   Z108618_0934 (
PULPROG             zg30
TD                 65536
SOLVENT             CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                112.25
DW                62.400 usec
DE                 6.50 usec
TE                296.7 K
D1             1.00000000 sec
TD0                    1
SFO1         400.0324702 MHz
NUC1                  1H
P1                14.00 usec
PLW1         13.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.0300079 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

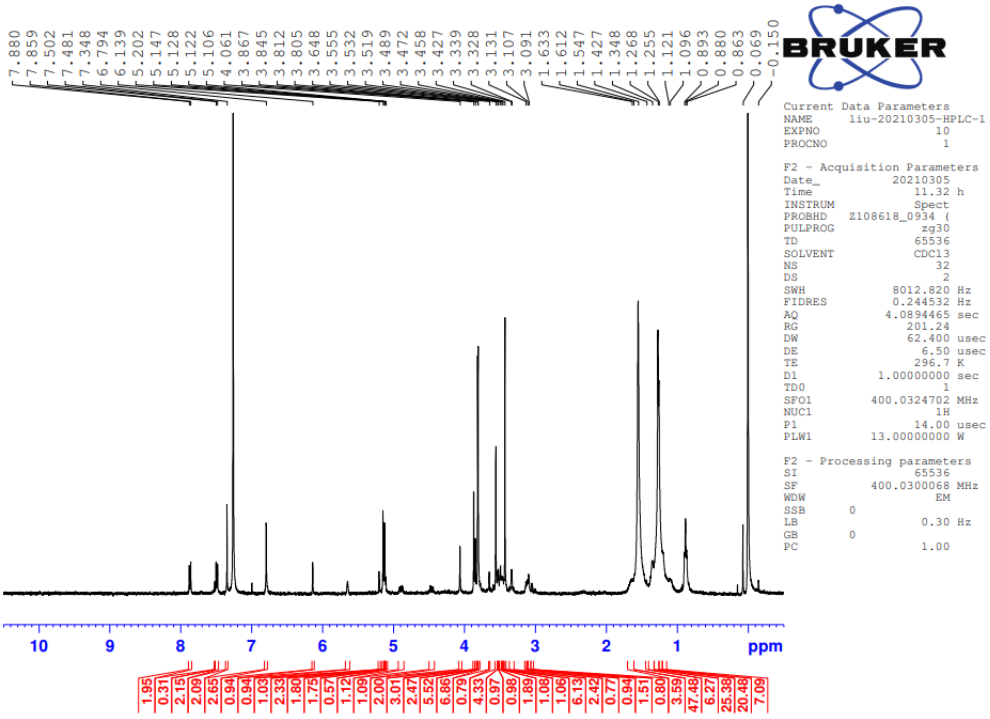


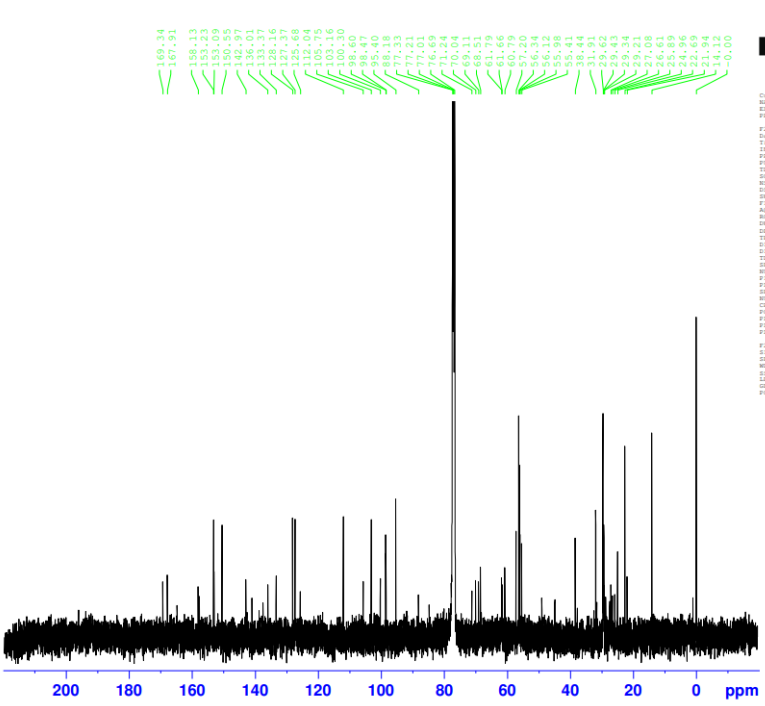
BRUKER

```
Current Data Parameters
NAME      liu-0406-n2-13C
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20210406
Time               19.50 h
INSTRUM              Spect
PROBHD   Z108618_0934 (
PULPROG            zgpg30
TD                 65536
SOLVENT             CDCl3
NS                    577
DS                      4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                201.24
DW                20.800 usec
DE                 6.50 usec
TE                297.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1         100.5976823 MHz
NUC1                  13C
P1                10.00 usec
PLW1         51.00500107 W
SFO2         400.0316001 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2         13.00000000 W
PLW12         0.31457001 W
PLW13         0.15823001 W

F2 - Processing parameters
SI                 32768
SF           100.5876235 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



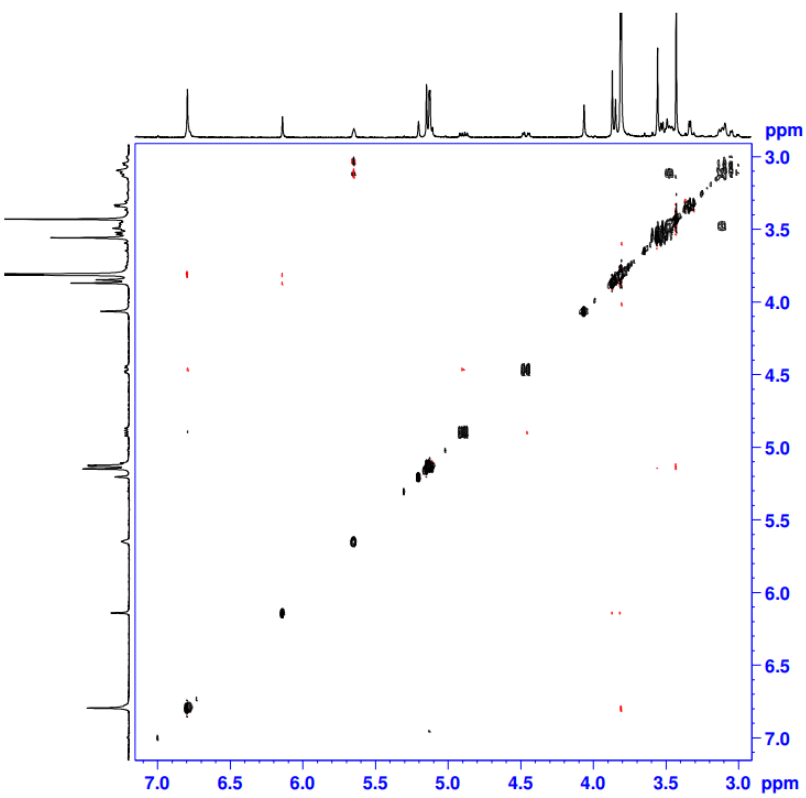
20a


7.880
7.859
7.502
7.481
7.348
6.794
6.139
5.202
5.147
5.128
5.122
5.106
4.061
3.867
3.845
3.812
3.805
3.648
3.555
3.532
3.519
3.489
3.472
3.458
3.427
3.339
3.328
3.131
3.107
3.091
1.633
1.612
1.547
1.427
1.348
1.268
1.255
1.121
1.096
0.893
0.880
0.863
0.069
-0.150
BRUKER
Current Data Parameters
NAME liu-20210305-HPLC-1
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210305
Time 11.32 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 201.24
DW 62.400 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.0324702 MHz
NUC1 1H
P1 14.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.0300068 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.95
0.31
2.15
2.09
2.65
0.94
0.94
1.03
2.33
1.80
1.75
0.57
1.12
1.09
2.00
3.01
2.47
5.52
6.86
0.79
4.33
0.97
0.98
1.89
1.06
1.06
6.13
2.42
0.77
0.94
1.51
0.80
3.59
47.48
6.27
25.38
20.48
7.09



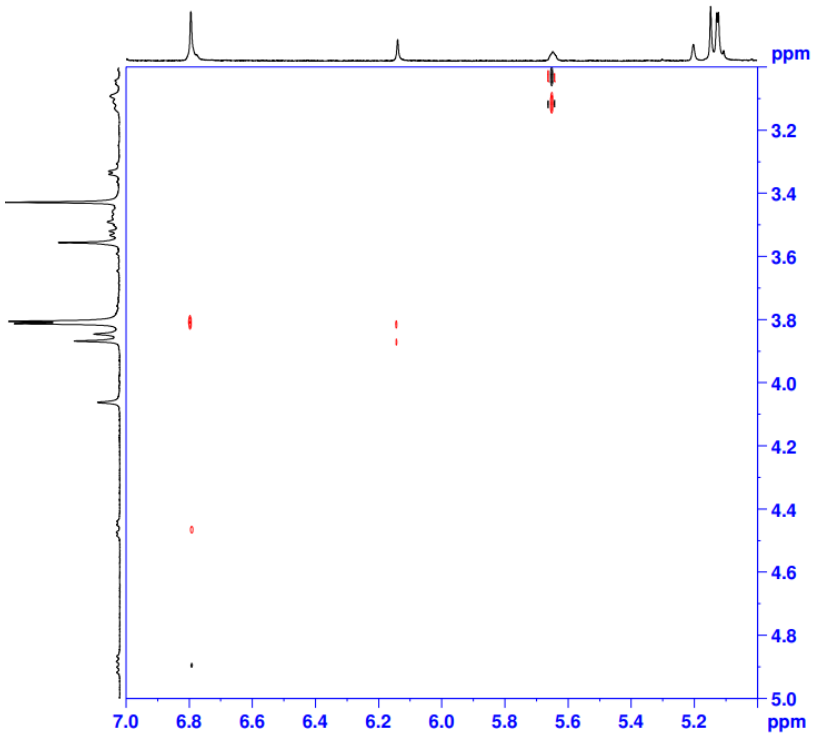
BRUKER

```
Current Data Parameters
NAME      liu-20210309-HPLC-down-13C-2nd overnight
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210310
Time              11.42 h
INSTRUM             Spect
PROBHD   Z108618_0934 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                17947
DS                    4
SWH           24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG                201.24
DW               20.800 usec
DE                6.50 usec
TE                297.9 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
SFO1        100.5976823 MHz
NUC1               13C
P1                10.00 usec
PLW1       51.00500107 W
SFO2       400.0316001 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2       13.00000000 W
PLW12       0.31457001 W
PLW13       0.15823001 W

F2 - Processing parameters
SI                32768
SF          100.5876239 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



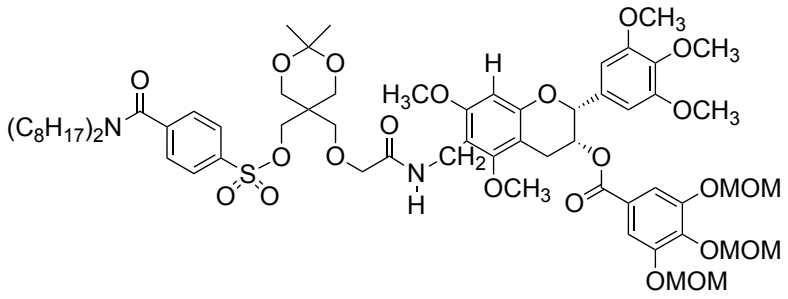
BRUKER

```
Current Data Parameters
NAME      liu-20210309-HPLC-up-NOESY
EXPNO                15
PROCNO                1

F2 - Acquisition Parameters
Date_          20210309
Time              12.30 h
INSTRUM             Spect
PROBHD   Z108618_0934 (
PULPROG         noesygpph
TD                 2048
SOLVENT           CDCl3
NS                    4
DS                   32
SWH            1698.370 Hz
FIDRES         1.658564 Hz
AQ             0.6029312 sec
RG                201.24
DW              294.400 usec
DE                6.50 usec
TE                296.8 K
D0             0.00027657 sec
D1             2.00000000 sec
D8             0.30000001 sec
D16            0.00020000 sec
IN0            0.00058880 sec
TDav                  1
SFO1         400.0320199 MHz
NUC1                  1H
P1                14.00 usec
P2                28.00 usec
PLW1        13.00000000 W
GPNAM[1]      SMSQ10.100
GPZ1              40.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  256
SFO1         400.032 MHz
FIDRES      13.268512 Hz
SW              4.246 ppm
FnMODE             TPPI

F2 - Processing parameters
SI                 1024
SF          400.0300068 MHz
WDW               QSINE
SSB                   2
LB        0 Hz
GB        0
PC                 1.00

F1 - Processing parameters
SI                 1024
MC2                TPPI
SF          400.0300068 MHz
WDW               QSINE
SSB                   2
LB        0 Hz
GB        0
```

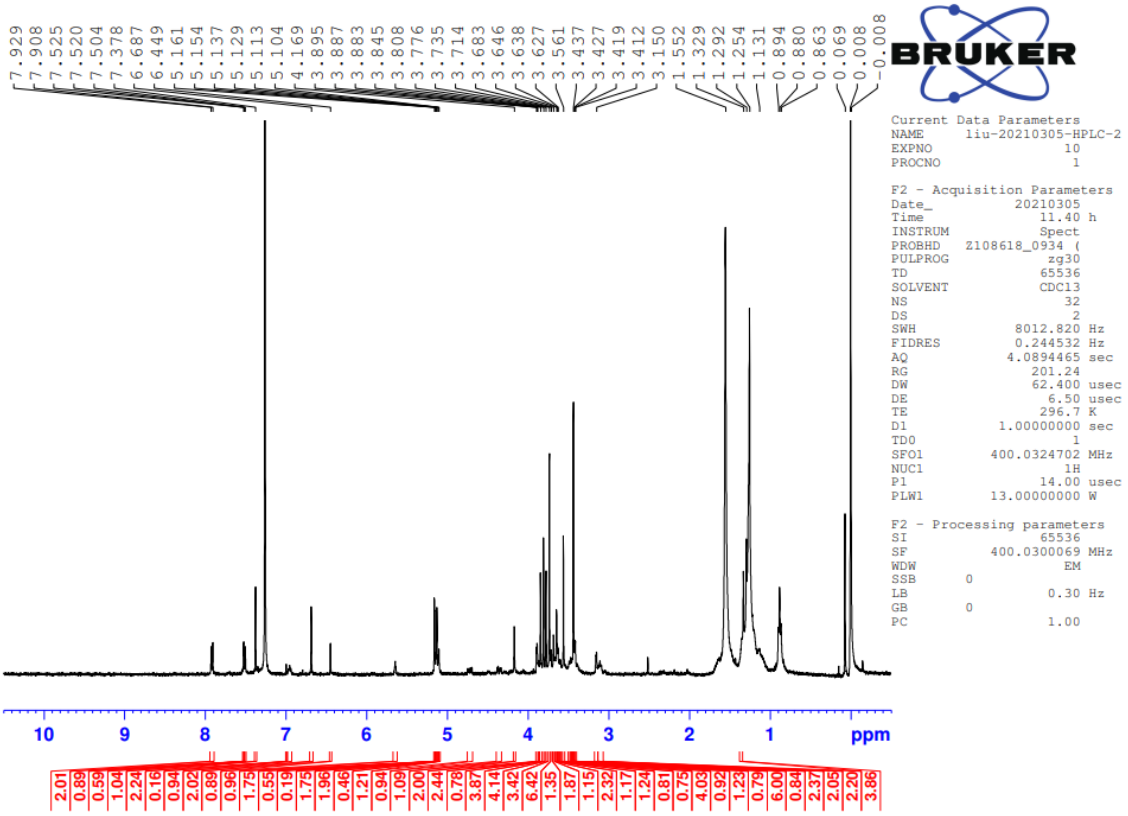


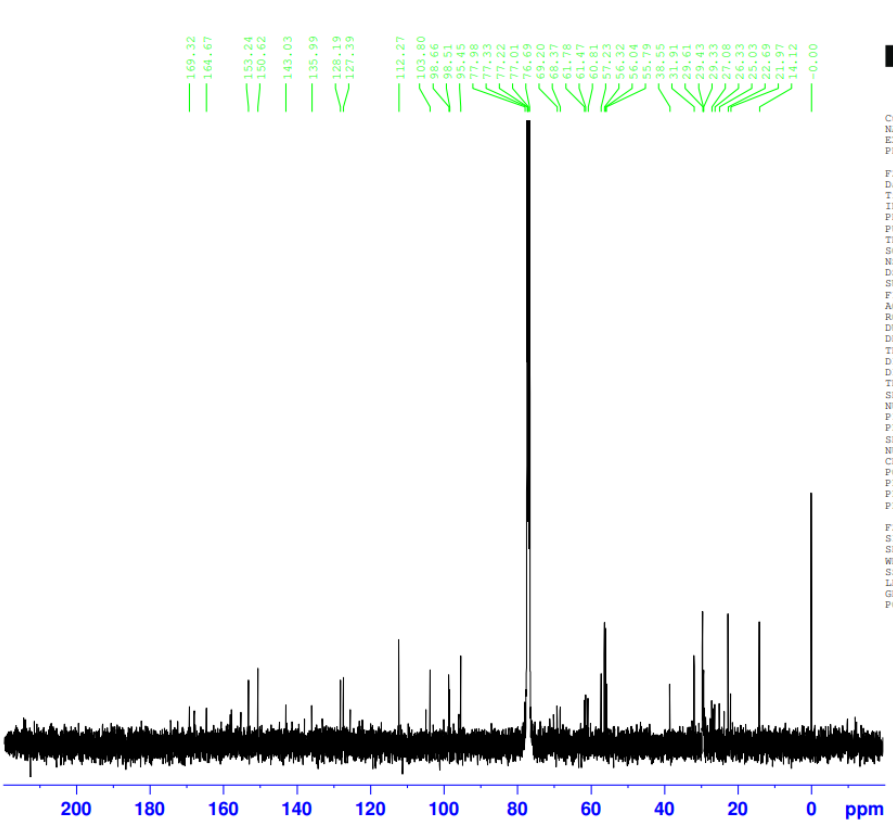
BRUKER

```
Current Data Parameters
NAME      liu-20210309-HPLC-up-NOESY
EXPNO                 15
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210309
Time              12.30 h
INSTRUM             Spect
PROBHD    Z108618_0934 (
PULPROG        noesygpph
TD                  2048
SOLVENT             CDC13
NS                     4
DS                    32
SWH            1698.370 Hz
FIDRES         1.658564 Hz
AQ             0.6029312 sec
RG               201.24
DW               294.400 usec
DE                 6.50 usec
TE               296.8 K
D0          0.00027657 sec
D1          2.00000000 sec
D8          0.30000001 sec
D16         0.00020000 sec
IN0         0.00058880 sec
TDav                   1
SFO1       400.0320199 MHz
NUC1                  1H
P1                14.00 usec
P2                28.00 usec
PLW1       13.00000000 W
GPNAM[1]     SMSQ10.100
GPZ1              40.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                   256
SFO1          400.032 MHz
FIDRES       13.268512 Hz
SW               4.246 ppm
FnMODE              TPPI

F2 - Processing parameters
SI                  1024
SF         400.0300068 MHz
WDW                 QSINE
SSB                     2
LB        0 Hz
GB        0
PC                 1.00

F1 - Processing parameters
SI                  1024
MC2                 TPPI
SF         400.0300068 MHz
WDW                 QSINE
SSB                     2
LB        0 Hz
GB        0
```



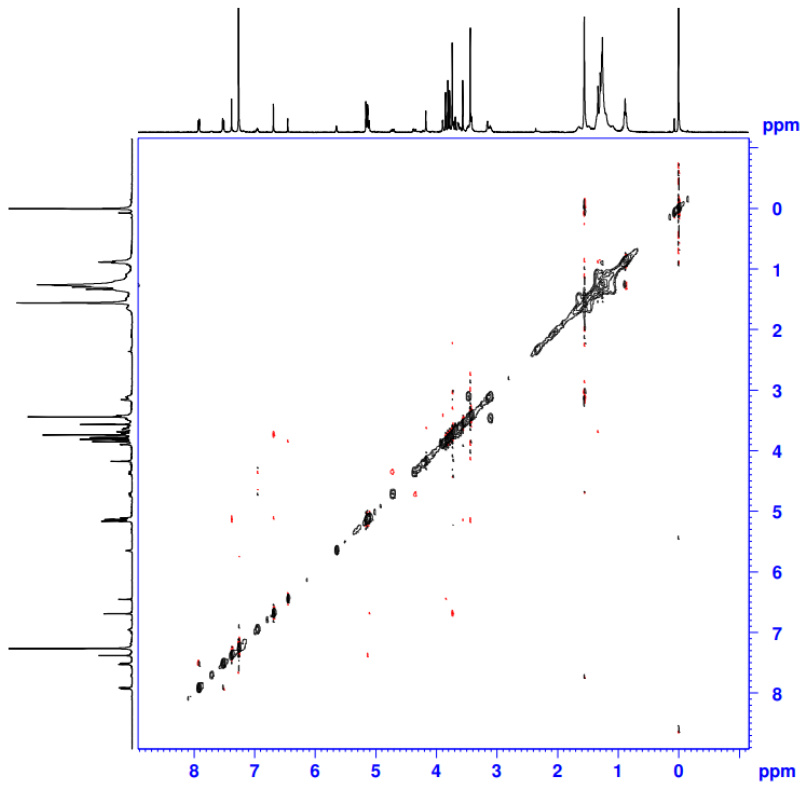
21a


7.929
7.908
7.525
7.520
7.504
7.378
6.687
6.449
5.161
5.154
5.137
5.129
5.113
5.104
4.169
3.895
3.887
3.883
3.845
3.808
3.776
3.735
3.714
3.683
3.646
3.638
3.627
3.561
3.437
3.427
3.419
3.412
3.150
1.552
1.329
1.292
1.254
1.131
0.894
0.880
0.863
0.069
0.008
-0.008
BRUKER
Current Data Parameters
NAME liu-20210305-HPLC-2
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210305
Time 11.40 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 201.24
DW 62.400 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.0324702 MHz
NUC1 1H
P1 14.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.0300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
2.01
0.89
0.59
1.04
2.24
0.16
0.94
2.02
0.89
0.96
1.75
0.55
0.19
1.75
1.96
0.46
1.21
0.94
1.09
2.00
2.44
0.78
3.87
4.14
3.42
6.42
1.35
1.87
1.15
2.32
1.17
1.24
0.81
0.75
4.03
0.92
1.23
0.79
6.00
0.84
2.37
2.05
2.20
3.86



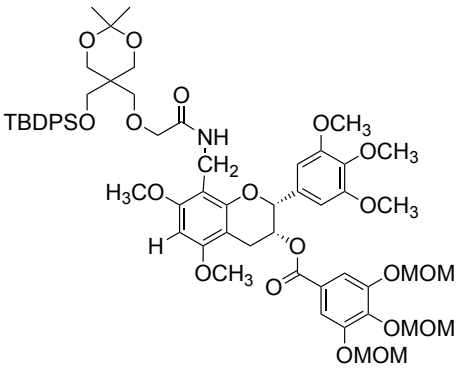
BRUKER

```
Current Data Parameters
NAME      liu-20210308-HPLC-dc
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_              20210309
Time                 10.34 h
INSTRUM              Spect
PROBHD    Z108618_0934 (
PULPROG              zgpg30
TD                    65536
SOLVENT              CDCl3
NS                    12700
DS                        4
SWH              24038.461 Hz
FIDRES            0.733596 Hz
AQ               1.3631488 sec
RG                   201.24
DW                   20.800 usec
DE                    6.50 usec
TE                  298.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1
SFO1            100.5976823 MHz
NUC1                    13C
P1                   10.00 usec
PLW1            51.00500107 W
SFO2            400.0316001 MHz
NUC2                    1H
CPDPRG[2          waltz16
PCPD2                90.00 usec
PLW2            13.00000000 W
PLW12            0.31457001 W
PLW13            0.15823001 W

F2 - Processing parameters
SI                    32768
SF              100.5876238 MHz
WDW                      EM
SSB         0
LB                    1.00 Hz
GB          0
PC                    1.40
```

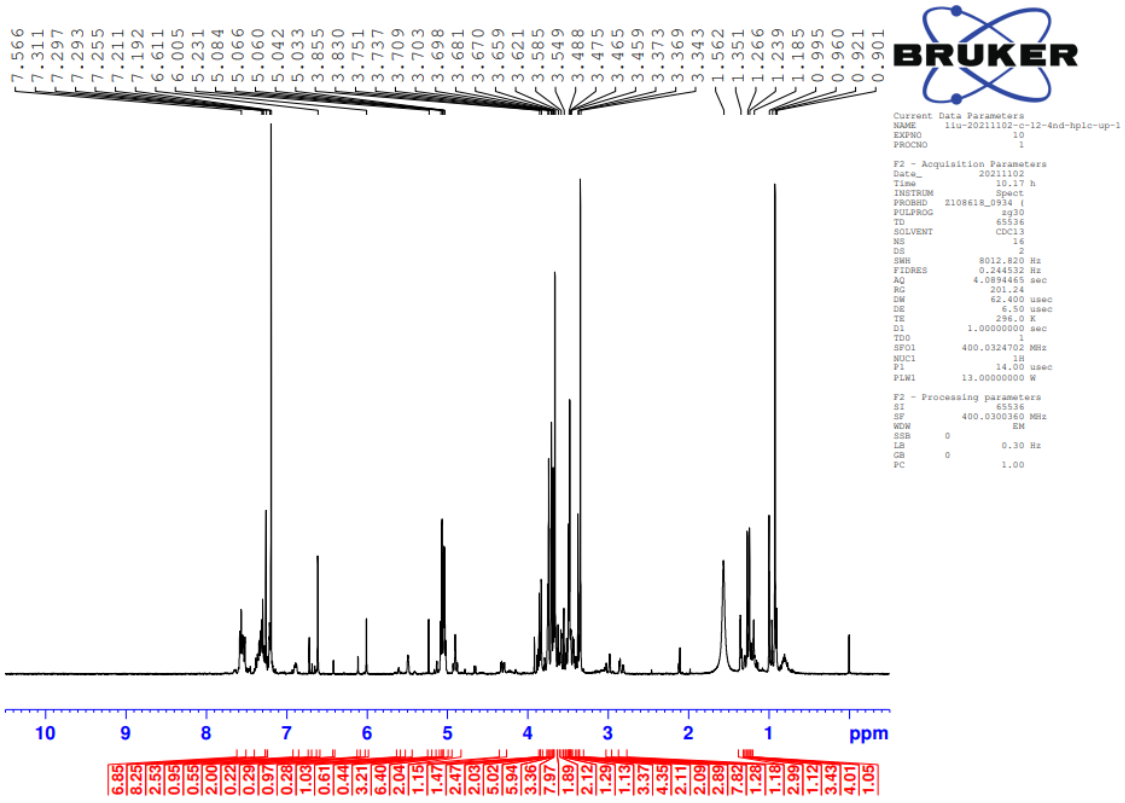


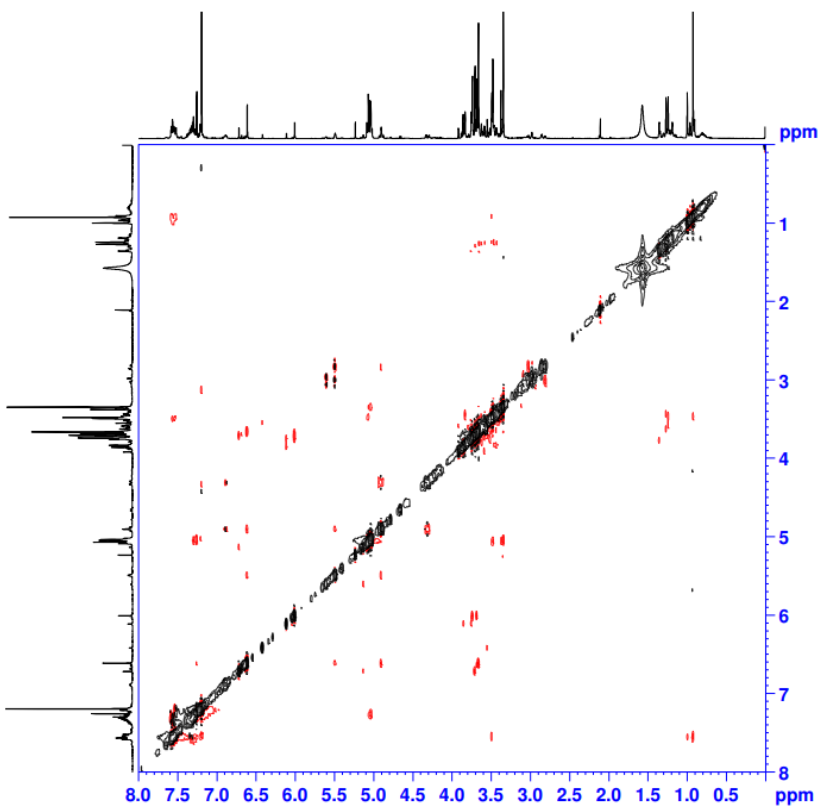
BRUKER

```
Current Data Parameters
NAME      liu-20210308-HPLC-down-NOESY
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_              20210308
Time                 19.30 h
INSTRUM              Spect
PROBHD    Z108618_0934 (
PULPROG        noesygpphpp
TD                    2048
SOLVENT              CDCl3
NS                      16
DS                      32
SWH              4032.258 Hz
FIDRES           3.937752 Hz
AQ               0.2539520 sec
RG                  180.02
DW                  124.000 usec
DE                    6.50 usec
TE                  297.0 K
D0               0.00010617 sec
D1               2.00204802 sec
D8               0.30000001 sec
D11              0.03000000 sec
D12              0.00002000 sec
D16              0.00020000 sec
IN0              0.00024800 sec
TDav                      1
SFO1            400.0315598 MHz
NUC1                    1H
P1                   14.00 usec
P2                   28.00 usec
P17                2500.00 usec
PLW1            13.00000000 W
PLW10            2.83109999 W
GPNAM[1]       SMSQ10.100
GPZ1                 40.00 %
P16                1000.00 usec

F1 - Acquisition parameters
TD                       256
SFO1            400.0316 MHz
FIDRES          31.502016 Hz
SW                  10.080 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                     1024
SF              400.0300064 MHz
WDW                    QSINE
SSB                        2
LB          0 Hz
GB          0
PC                     1.00

F1 - Processing parameters
SI                     1024
MC2          States-TPPI
SF              400.0300064 MHz
WDW                    QSINE
SSB                        2
LB          0 Hz
GB          0
```


TBDPSO
O
O
O
NH
CH2
H3CO
O
H
OCH3
OCH3
OCH3
OCH3
O
O
OMOM
OMOM
OMOM

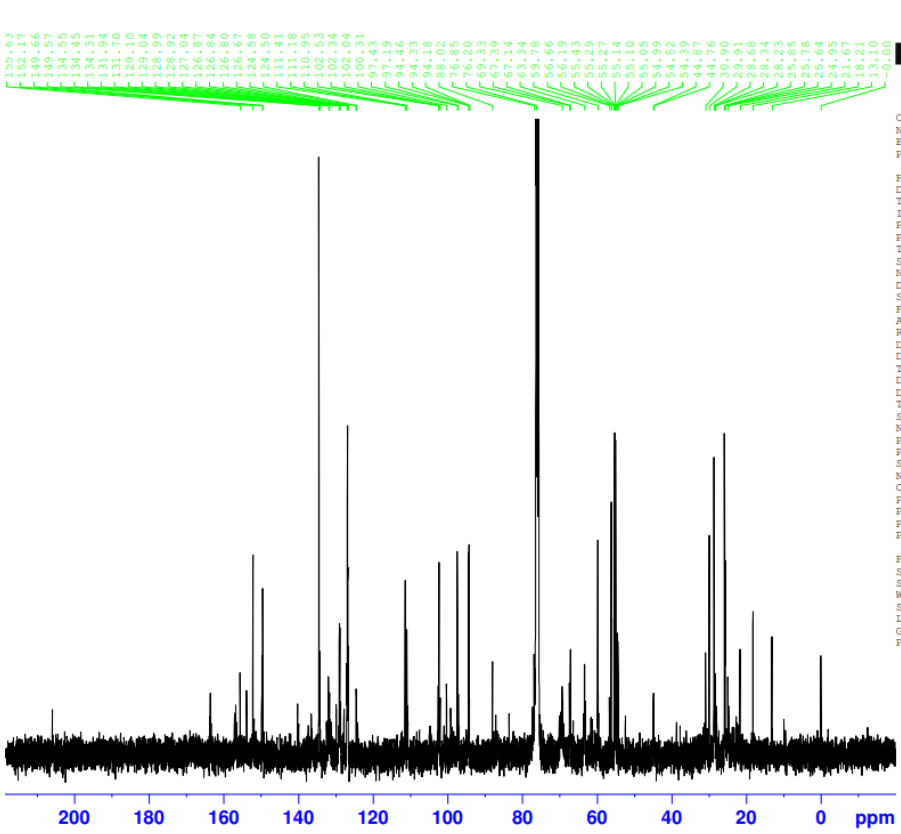
20b


7.566
7.311
7.297
7.293
7.255
7.211
7.192
6.611
6.005
5.231
5.084
5.066
5.060
5.042
5.033
3.855
3.830
3.751
3.737
3.709
3.703
3.698
3.681
3.670
3.659
3.621
3.585
3.549
3.488
3.475
3.465
3.459
3.373
3.369
3.343
1.562
1.351
1.266
1.239
1.185
0.995
0.960
0.921
0.901
BRUKER
Current Data Parameters
NAME liu-20211102-c-12-4nd-hplc-up-1
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20211102
Time 10.17 h
INSTRUM Spect
PROBHD 2108618_0934 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 201.24
DW 62.400 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.0324702 MHz
NUC1 1H
P1 14.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.0300360 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
6.85
8.25
2.53
0.95
0.55
2.00
0.22
0.29
0.97
0.28
1.03
0.61
0.44
3.21
6.40
2.04
1.15
1.47
2.47
2.03
5.02
5.94
3.36
7.97
1.89
2.12
1.29
1.13
3.37
4.35
2.11
2.09
2.88
7.82
1.28
1.18
2.99
1.12
3.43
4.01
1.05



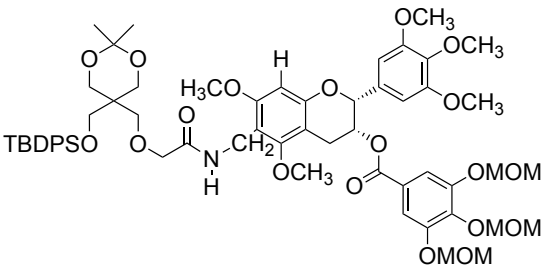
BRUKER

```
Current Data Parameters
NAME      liu-20211102-c-12-4nd-hplc-up-noesy
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211102
Time              10.48 h
INSTRUM             Spect
PROBHD   Z108618_0934 (
PULPROG     noesygpphpp
TD                  2048
SOLVENT             CDCl3
NS                     4
DS                    32
SWH            3875.969 Hz
FIDRES         3.785126 Hz
AQ             0.2641920 sec
RG                136.63
DW                129.000 usec
DE                  6.50 usec
TE                296.1 K
D0             0.00011117 sec
D1             1.99180806 sec
D8             0.30000001 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00025800 sec
TDav                   1
SFO1         400.0315392 MHz
NUC1                  1H
P1                 14.00 usec
P2                 28.00 usec
P17              2500.00 usec
PLW1          13.00000000 W
PLW10          2.83109999 W
GPNAM[1]       SMSQ10.100
GPZ1                40.00 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                    256
SFO1         400.0315 MHz
FIDRES        30.281008 Hz
SW                9.689 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                   1024
SF           400.0300361 MHz
WDW                 QSINE
SSB                     2
LB          0 Hz
GB          0
PC                   1.00

F1 - Processing parameters
SI                   1024
MC2          States-TPPI
SF           400.0300361 MHz
WDW                 QSINE
SSB                     2
LB          0 Hz
GB          0
```

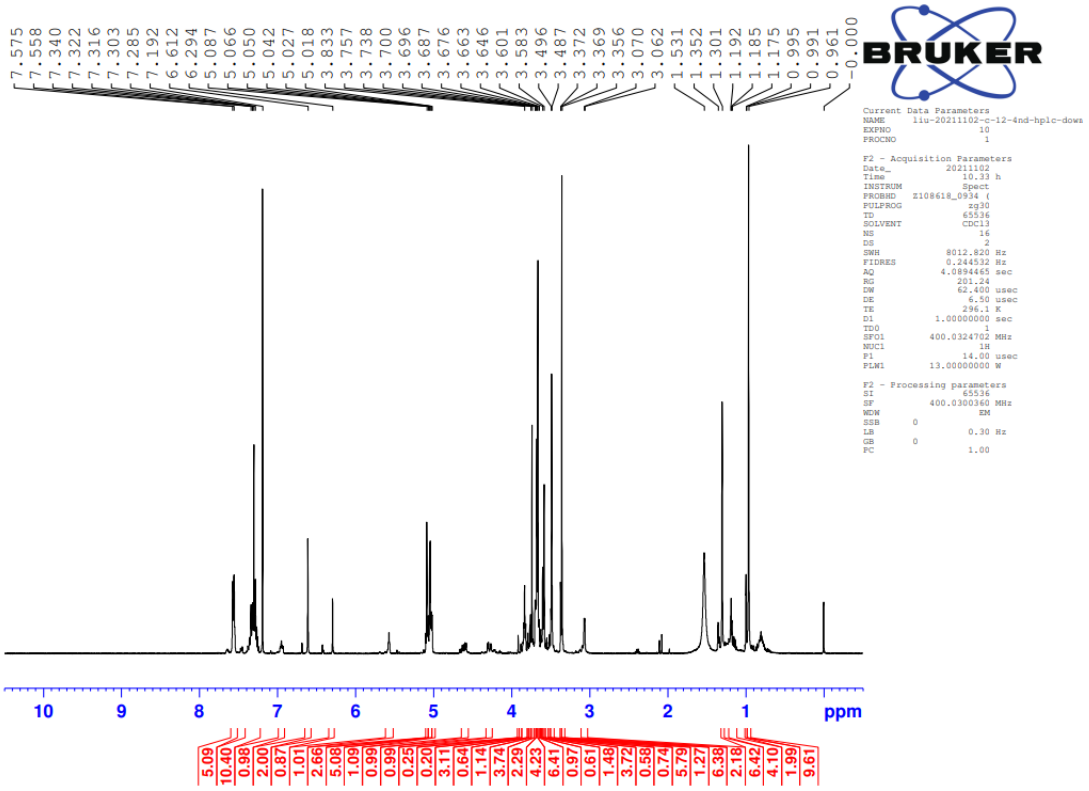


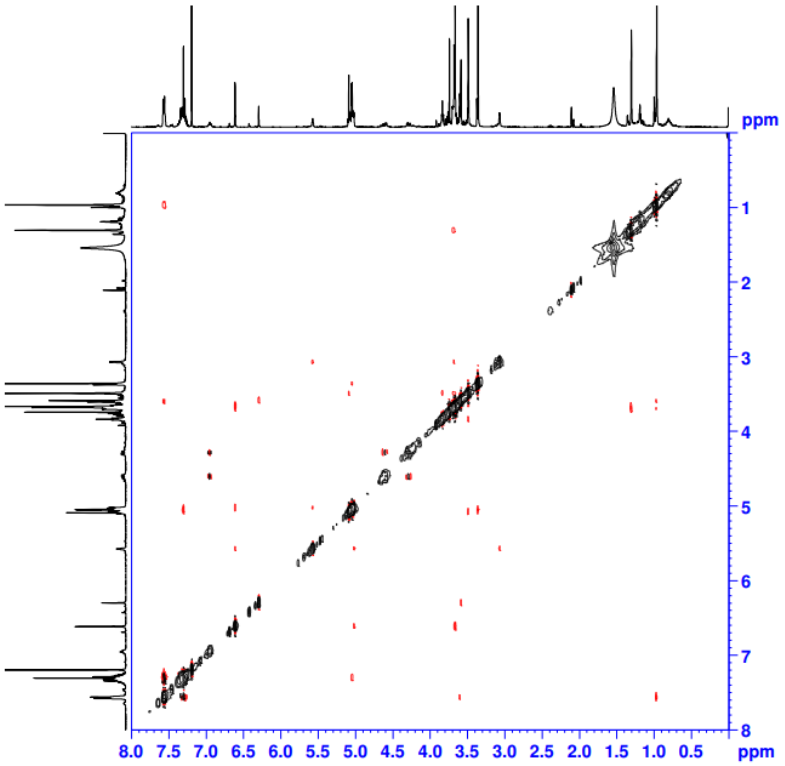
BRUKER

```
Current Data Parameters
NAME      liu-2021103-c-12-4nd-13C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211104
Time              10.04 h
INSTRUM             Spect
PROBHD   Z108618_0934 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                  22315
DS                      4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             1.3631488 sec
RG                201.24
DW                 20.800 usec
DE                  6.50 usec
TE                297.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1         100.5976823 MHz
NUC1                  13C
P1                 10.00 usec
PLW1          51.00500107 W
SFO2         400.0316001 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          13.00000000 W
PLW12          0.31457001 W
PLW13          0.15823001 W

F2 - Processing parameters
SI                   32768
SF           100.5877272 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

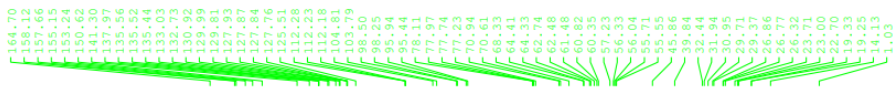

OCH3
OCH3
OCH3
H
H3CO
O
O
O
O
TBDPSO
O
N
H
CH2
OCH3
O
O
OMOM
OMOM
OMOM

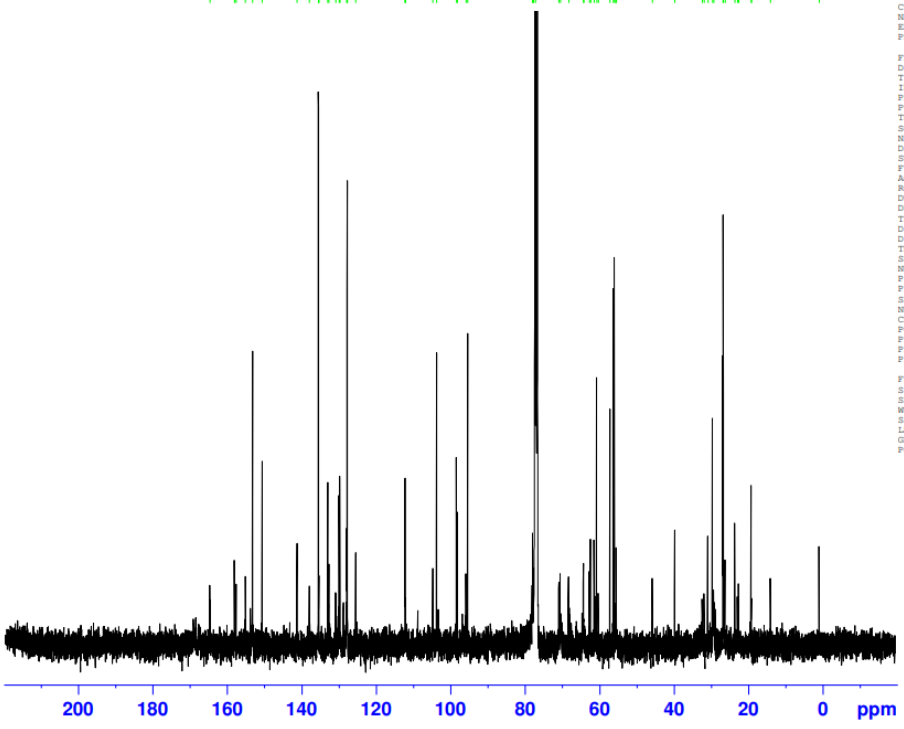
21b


7.575
7.558
7.340
7.322
7.316
7.303
7.285
7.192
6.612
6.294
5.087
5.066
5.050
5.042
5.027
5.018
3.833
3.757
3.738
3.700
3.696
3.687
3.676
3.663
3.646
3.601
3.583
3.496
3.487
3.372
3.369
3.356
3.070
3.062
1.531
1.352
1.301
1.192
1.185
1.175
0.995
0.991
0.961
0.000
BRUKER
Current Data Parameters
NAME 11u-20211102-c-12-4nd-hplc-dows
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20211102
Time 10.33 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 201.24
DW 62.400 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.0324702 MHz
NUC1 1H
P1 14.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.0300360 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
5.09
10.40
0.98
2.00
0.87
1.01
2.66
5.08
1.09
0.99
0.99
0.25
0.20
3.11
0.64
1.14
3.74
2.29
4.23
6.41
0.97
0.61
1.48
3.72
0.58
0.74
5.79
1.27
6.38
2.18
6.42
4.10
1.99
9.61

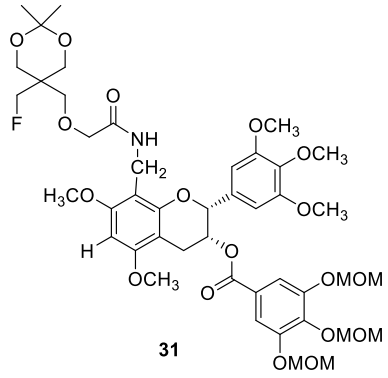



```
Current Data Parameters
NAME      liu-20211102-c-12-4nd-hplc-down-noesy
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20211102
Time              12.31 h
INSTRUM             spect
PROBHD   Z108618_0934 (
PULPROG    noesygpphpp
TD                 2048
SOLVENT           CDCl3
NS                    4
DS                   32
SWH           3875.969 Hz
FIDRES        3.785126 Hz
AQ            0.2641920 sec
RG                136.63
DW                129.000 usec
DE                  6.50 usec
TE                296.4 K
D0           0.00011117 sec
D1            1.99180806 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00025800 sec
TDav                  1
SFO1         400.0315453 MHz
NUC1                1H
P1                14.00 usec
P2                28.00 usec
P17             2500.00 usec
PLW1         13.00000000 W
PLW10         2.83109999 W
GPNAM[1]       SMSQ10.100
GPZ1              40.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  256
SFO1         400.0315 MHz
FIDRES       30.281008 Hz
SW             9.683 ppm
FnMODE      States-TPPI

F2 - Processing parameters
SI                 1024
SF           400.0300360 MHz
WDW                QSINE
SSB                   2
LB          0 Hz
GB          0
PC                 1.00

F1 - Processing parameters
SI                 1024
MC2         States-TPPI
SF           400.0300360 MHz
WDW                QSINE
SSB                   2
LB          0 Hz
GB          0
```

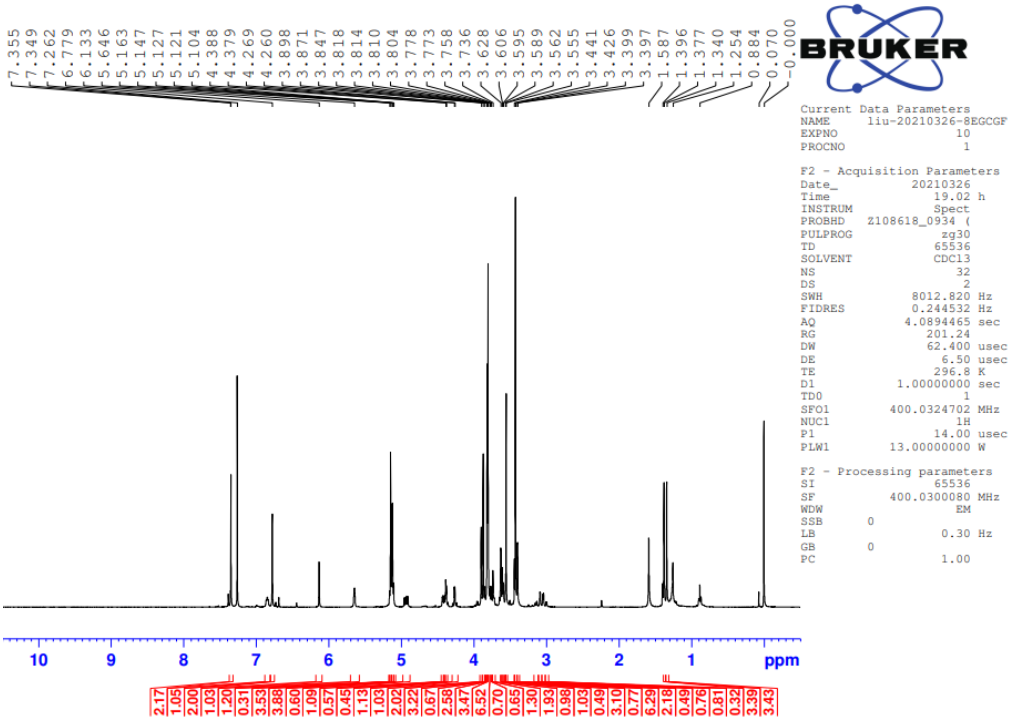


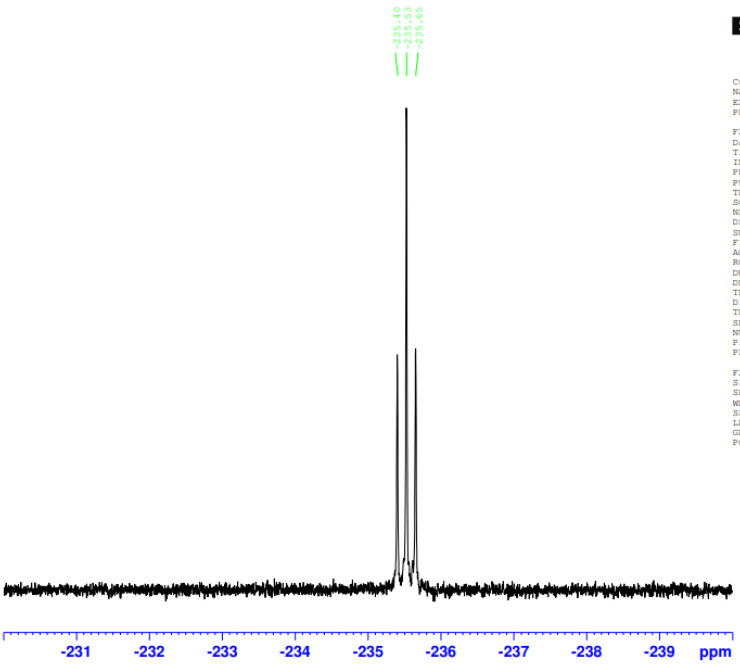



```
Current Data Parameters
NAME      liu-20211105-c-12-4nd-down-13C
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20211106
Time              12.06 h
INSTRUM             spect
PROBHD   Z108618_0934 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                15448
DS                    4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG                201.24
DW                20.800 usec
DE                 6.50 usec
TE                297.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.5976823 MHz
NUC1                13C
P1                10.00 usec
PLW1         51.00500107 W
SFO2        400.0316001 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         13.00000000 W
PLW12         0.31457001 W
PLW13         0.15823001 W

F2 - Processing parameters
SI                32768
SF          100.5876235 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



31

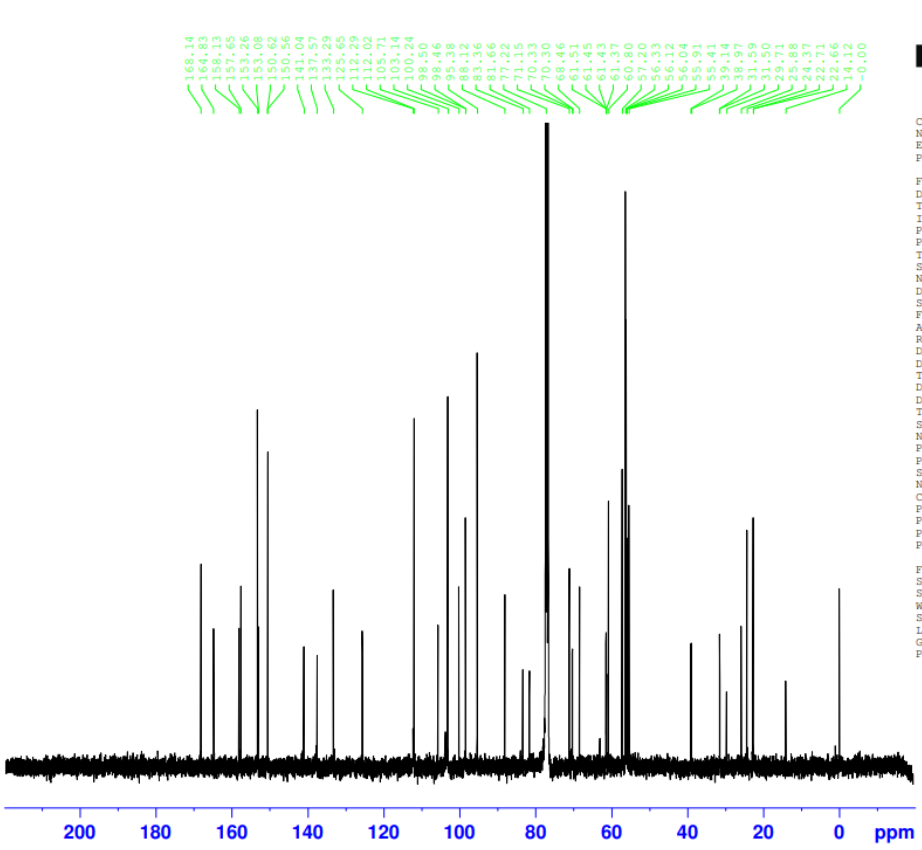

7.355
7.349
7.262
6.779
6.133
5.646
5.163
5.147
5.127
5.121
5.104
4.388
4.379
4.269
4.260
3.898
3.871
3.847
3.818
3.814
3.810
3.804
3.778
3.773
3.758
3.736
3.628
3.606
3.595
3.589
3.562
3.555
3.441
3.426
3.399
3.397
1.587
1.396
1.377
1.340
1.254
0.884
0.070
-0.000
BRUKER
Current Data Parameters
NAME liu-20210326-8EGCGF
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210326
Time 19.02 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 201.24
DW 62.400 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1
SFO1 400.0324702 MHz
NUC1 1H
P1 14.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.0300080 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
2.17
1.05
2.00
1.03
1.20
0.31
3.53
3.88
0.60
1.09
0.57
0.45
1.13
1.03
2.02
3.22
0.67
2.58
3.47
6.52
0.70
0.65
1.30
1.93
0.98
1.03
0.49
3.10
0.77
6.29
2.18
0.49
0.76
0.81
0.32
3.39
3.43



Current Data Parameters
NAME liu-20210326-8EGCGF-19F
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210326
Time 19.03 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zgflqn
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 75000.000 Hz
FIDRES 1.144409 Hz
AQ 0.8738133 sec
RG 201.24
DW 6.667 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1
SFO1 376.3289914 MHz
NUC1 19F
P1 15.00 usec
PLW1 20.81999969 W

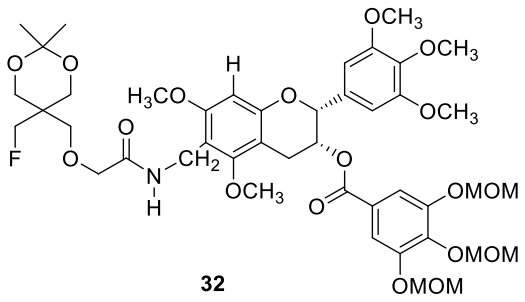
F2 - Processing parameters
SI 65536
SF 376.4042722 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME liu-20210329-8EGCG F-
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210330
Time 10.35 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16272
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 201.24
DW 20.800 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5976823 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.00500107 W
SFO2 400.0316001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.31457001 W
PLW13 0.15823001 W

F2 - Processing parameters
SI 32768
SF 100.5876239 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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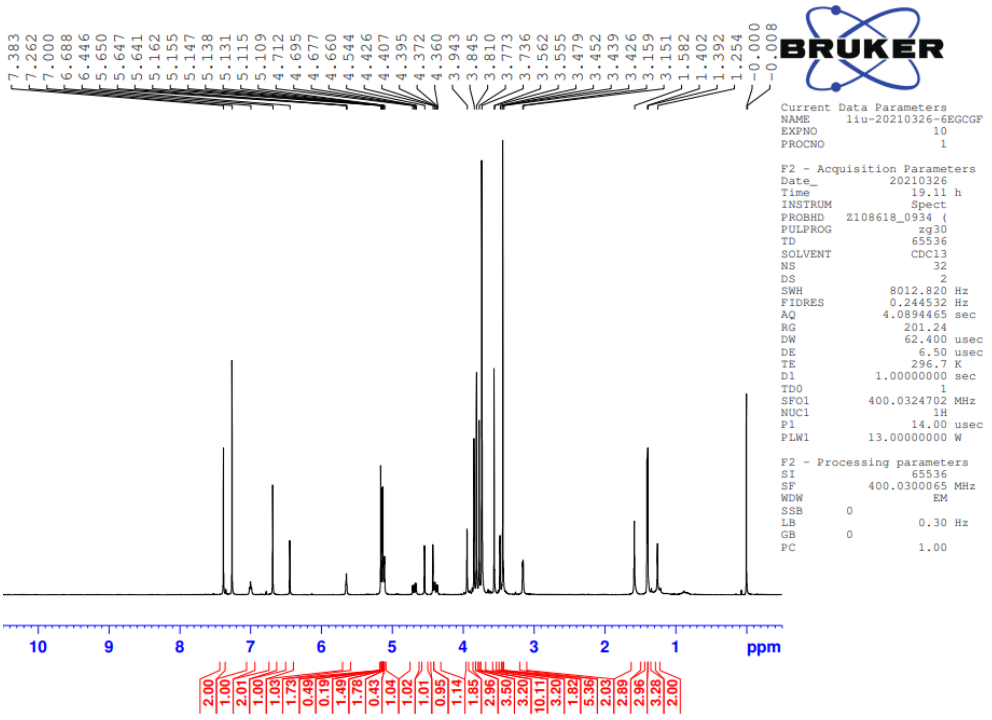
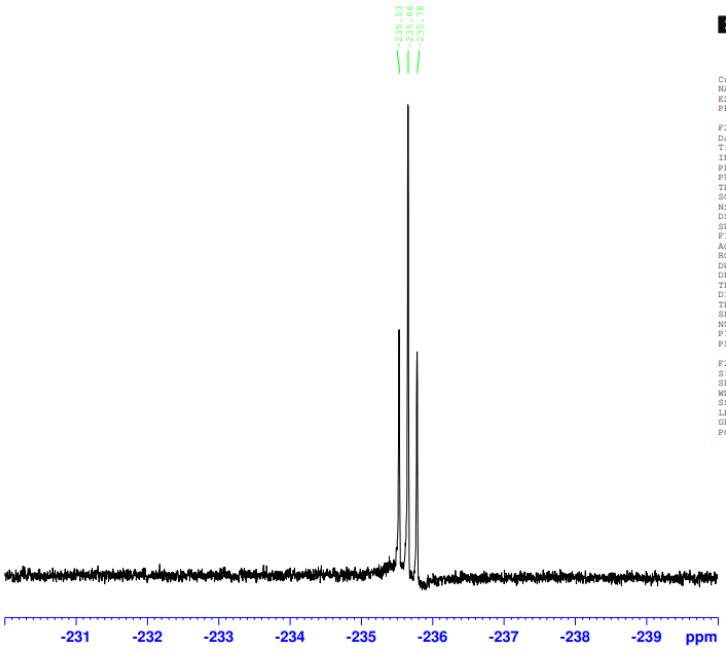
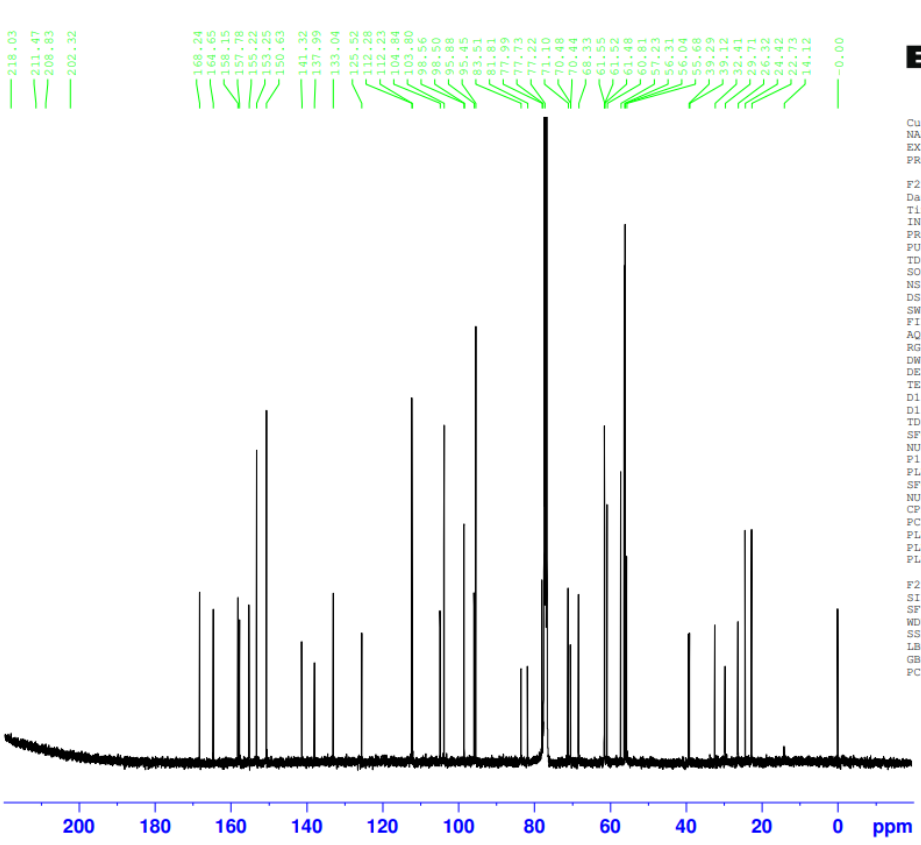

7.383
7.262
7.000
6.688
6.446
5.650
5.647
5.641
5.162
5.155
5.147
5.138
5.131
5.115
5.109
4.712
4.695
4.677
4.660
4.544
4.426
4.407
4.395
4.372
4.360
3.943
3.845
3.810
3.773
3.736
3.562
3.555
3.479
3.452
3.439
3.426
3.159
3.151
1.582
1.402
1.392
1.254
0.000
-0.008
BRUKER
Current Data Parameters
NAME liu-20210326-6EGCGF
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210326
Time 19.11 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 201.24
DW 62.400 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.0324702 MHz
NUC1 1H
P1 14.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.0300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
2.00
1.00
2.01
1.00
1.03
1.73
0.49
0.19
1.49
1.76
0.43
1.04
1.02
1.01
0.95
1.14
1.85
2.96
3.50
10.11
3.20
1.82
5.36
2.03
2.89
2.96
3.28
2.00



Current Data Parameters
NAME liu-20210326-6EGCGF-19F
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210326
Time 19.12 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zgflqn
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 75000.000 Hz
FIDRES 1.144409 Hz
AQ 0.8738133 sec
RG 201.24
DW 6.667 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
SFO1 376.3289914 MHz
NUC1 19F
P1 15.00 usec
PLW1 20.81999969 W

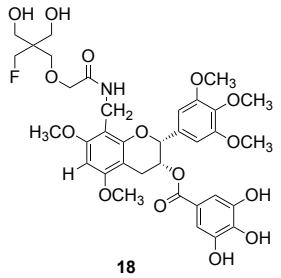
F2 - Processing parameters
SI 65536
SF 376.4042722 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME liu-20210326-6EGCGF-1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210329
Time 11.15 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 67285
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 201.24
DW 20.800 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5976823 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.00500107 W
SFO2 400.0316001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.31457001 W
PLW13 0.15823001 W

F2 - Processing parameters
SI 32768
SF 100.5876239 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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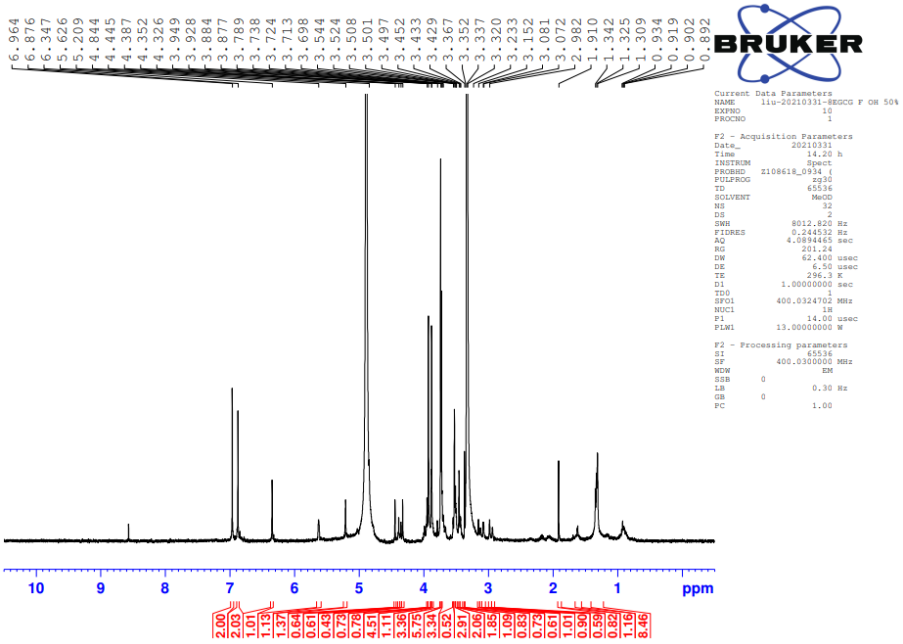
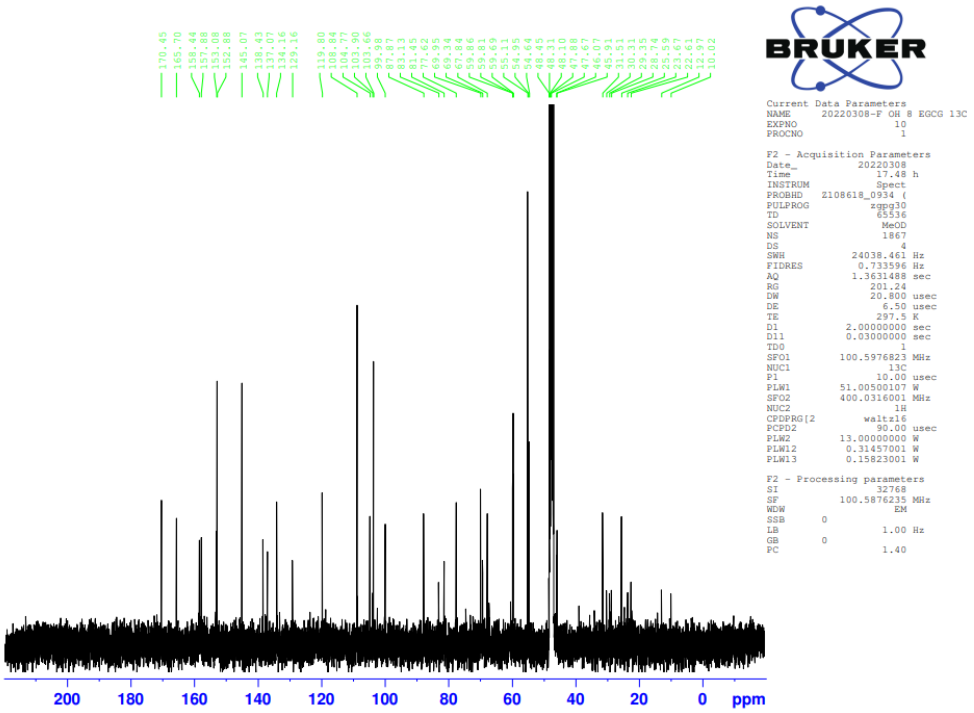
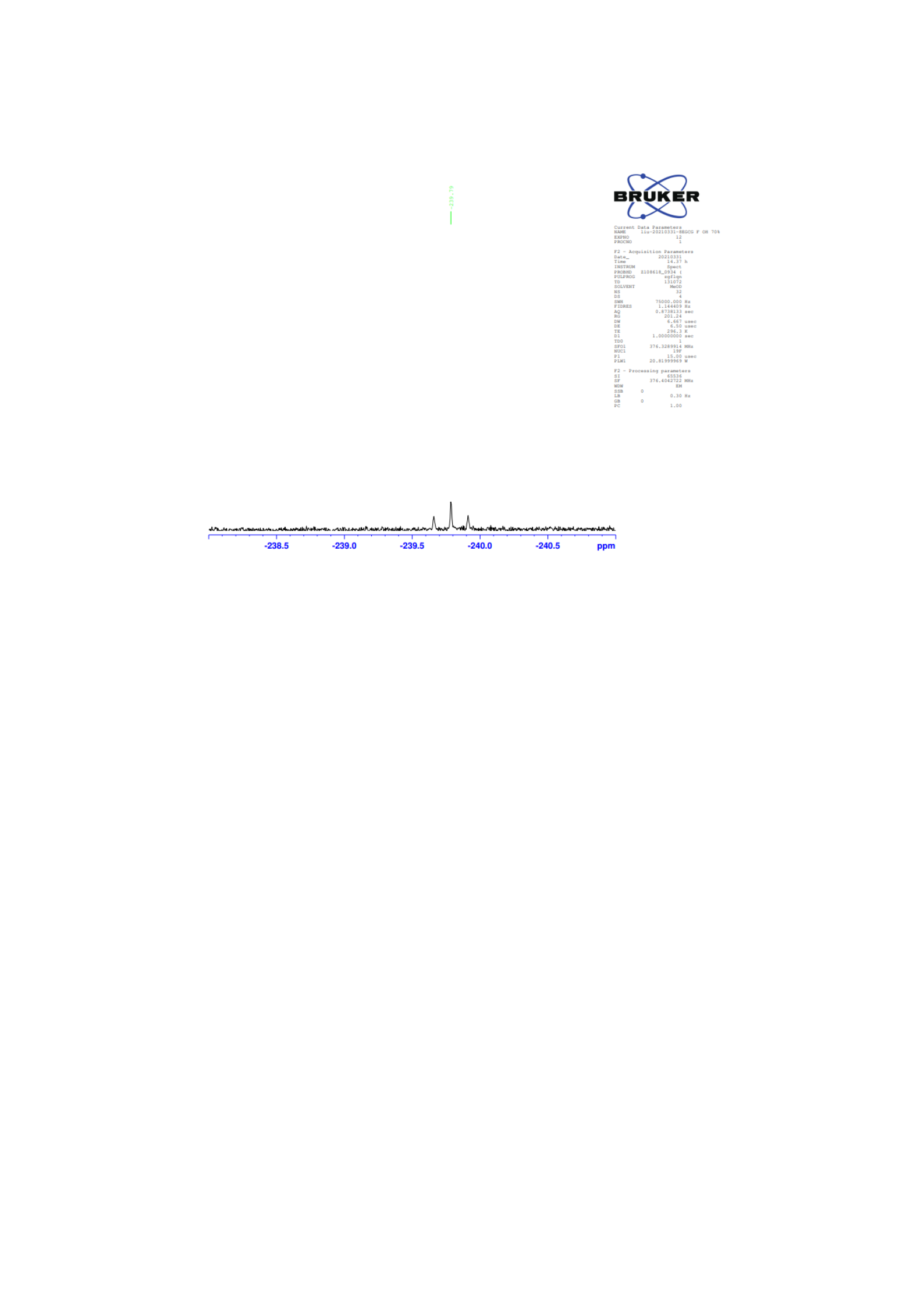
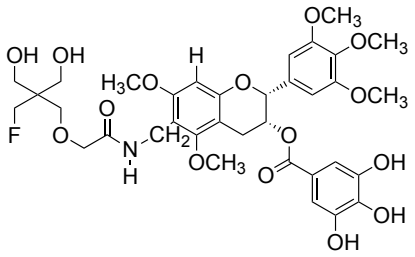
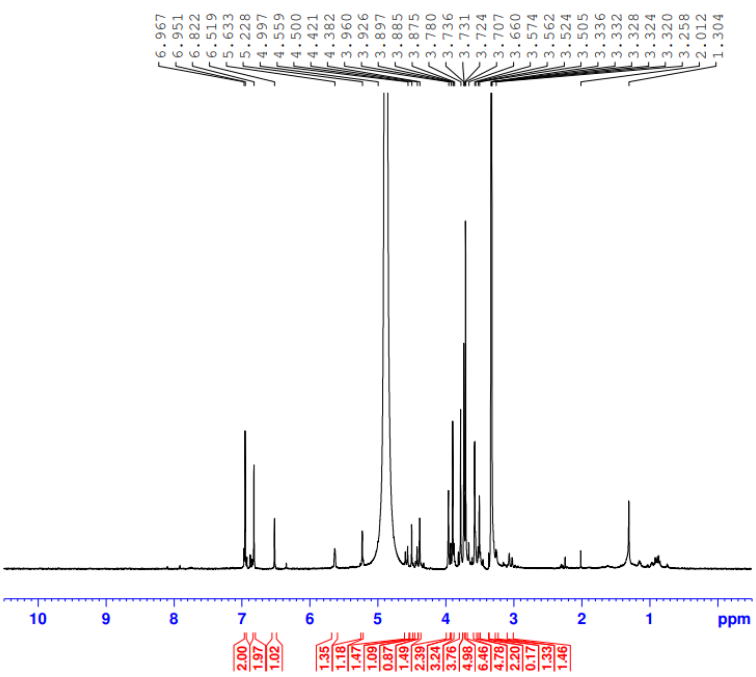

6.964
6.876
6.347
5.626
5.209
4.844
4.445
4.387
4.352
4.326
3.949
3.928
3.884
3.877
3.789
3.738
3.724
3.713
3.698
3.544
3.524
3.508
3.501
3.497
3.452
3.433
3.429
3.367
3.352
3.337
3.320
3.233
3.152
3.081
3.072
2.982
1.910
1.342
1.325
1.309
0.934
0.919
0.902
0.892
BRUKER
Current Data Parameters
NAME 11u-20210331-EGCG F OH 50%
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210331
Time 14.20 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 32
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 201.24
DW 62.400 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.0324702 MHz
NUC1 1H
P1 14.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.0300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
2.00
2.03
1.01
1.13
1.37
0.64
0.61
0.43
0.78
0.78
4.51
1.11
3.36
5.75
3.34
0.52
2.91
2.06
1.85
1.09
0.83
0.72
0.61
1.01
0.90
0.58
0.82
1.16
8.46


170.45
165.70
158.44
157.88
153.08
152.88
145.07
138.43
137.07
134.16
129.16
119.80
118.84
104.77
103.90
103.66
99.88
83.13
81.45
77.62
69.95
69.34
69.04
59.86
59.81
59.69
55.11
54.35
54.04
48.45
48.31
48.10
47.88
47.67
46.07
45.91
31.51
30.31
29.35
28.98
25.59
23.67
22.61
12.97
10.02
BRUKER
Current Data Parameters
NAME 20220308-F OH 8 EGCG 13C
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220308
Time 17.48 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1867
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 201.24
DW 20.800 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5976823 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.00500107 W
SFO2 400.0316001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.31457001 W
PLW13 0.15823001 W
F2 - Processing parameters
SI 32768
SF 100.5876235 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


-239.79
BRUKER
Current Data Parameters
NAME liu-20210331-8EGCG F OH 70%
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210331
Time 14.37 h
INSTRUM Spect
PROBHD Z108618_0934 (
PULPROG zgflqn
TD 131072
SOLVENT MeOD
NS 32
DS 4
SWH 75000.000 Hz
FIDRES 1.144409 Hz
AQ 0.8738133 sec
RG 203.24
DW 6.667 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
SFO1 376.3289914 MHz
NUC1 19F
P1 15.00 usec
PLW1 20.81999969 W
F2 - Processing parameters
SI 65536
SF 376.4042722 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-238.5
-239.0
-239.5
-240.0
-240.5
ppm



19




```
Current Data Parameters
NAME      liu-20210311-down-pink
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210311
Time              18.08 h
INSTRUM             Spect
PROBHD    Z108618_0934 (
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                    16
DS                     2
SWH            8012.820 Hz
FIDRES            0.244532 Hz
AQ              4.0894465 sec
RG                 98.29
DW                62.400 usec
DE                 6.50 usec
TE                296.8 K
D1              1.00000000 sec
TD0                    1
SFO1          400.0324702 MHz
NUC1                  1H
P1                14.00 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.0300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
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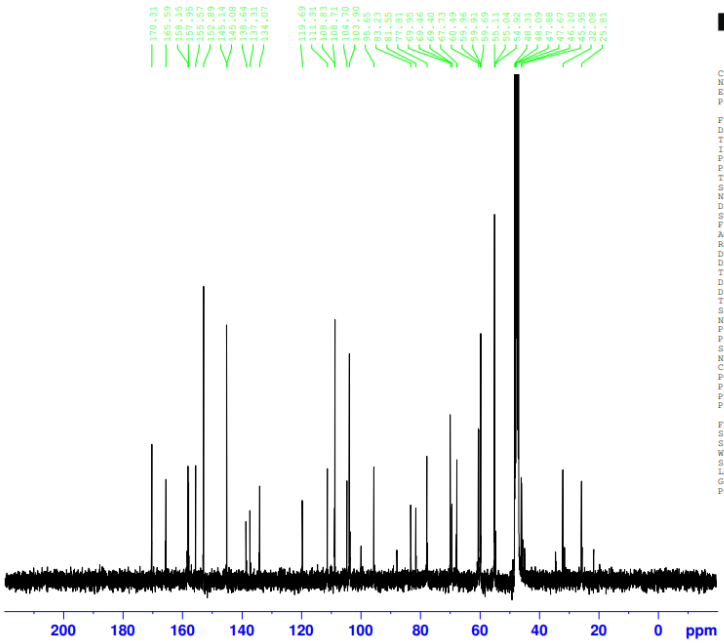


```
Current Data Parameters
NAME     20220308-F OH 6 EGCG 13C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20220309
Time              10.22 h
INSTRUM             Spect
PROBHD    Z108618_0934 (
PULPROG          zgpg30
TD                 65536
SOLVENT             MeOD
NS                 17301
DS                     4
SWH           24038.461 Hz
FIDRES            0.733596 Hz
AQ              1.3631488 sec
RG                201.24
DW                20.800 usec
DE                 6.50 usec
TE                297.2 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1
SFO1         100.5976823 MHz
NUC1                  13C
P1                10.00 usec
PLW1         51.00500107 W
SFO2         400.0316001 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2         13.00000000 W
PLW12         0.31457001 W
PLW13         0.15823001 W

F2 - Processing parameters
SI                 32768
SF           100.5876235 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
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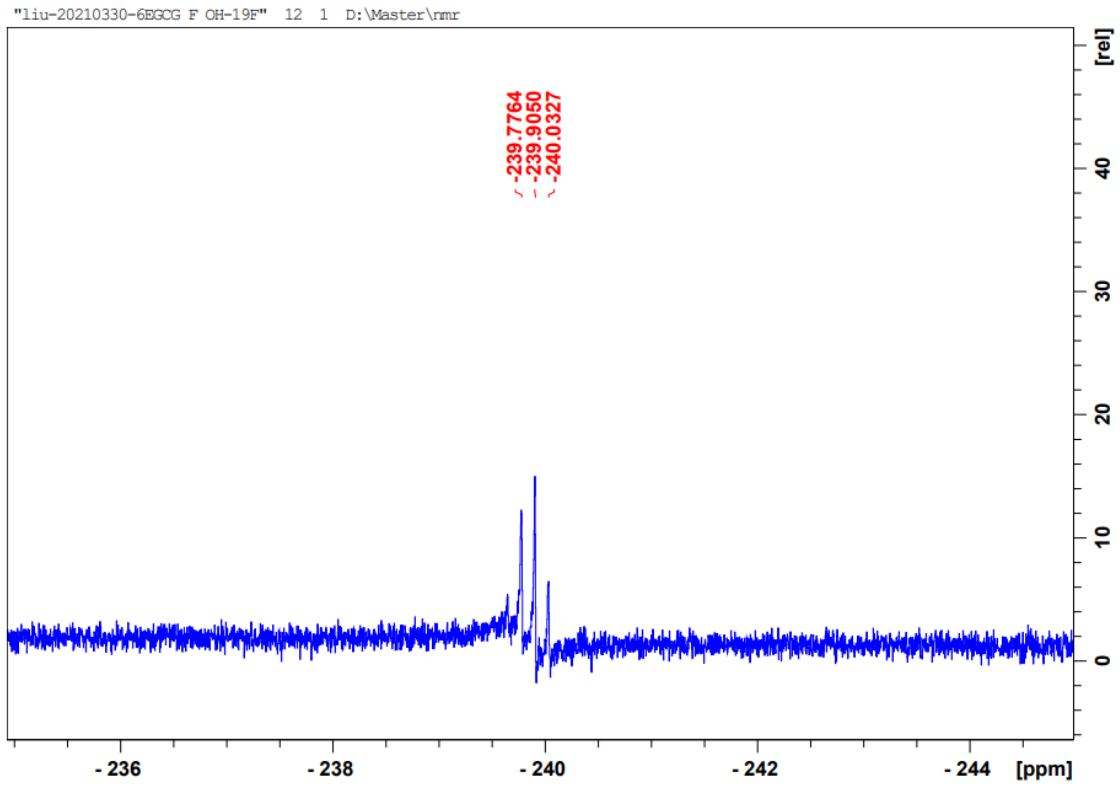
"liu-20210330-6EGCG F OH-19F" 12 1 D:\Master\nmr
-239.7764
-239.9050
-240.0327
[rel]
40
30
20
10
0
- 236
- 238
- 240
- 242
- 244
[ppm]