Supporting Information

Organogelating and Narcissistic Self-Sorting Behaviour of Non-Preorganized Oligoamides

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Contents

1. General information

^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded on a Bruker Avance DPX 400 spectrometer. Other ^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) spectra were recorded on a Bruker Avance DPX 500 spectrometer. All measurements were performed at 25 °C in $CDCl_3$ unless otherwise stated. Chemical shifts were reported in ppm (δ) and coupling constants (J) were reported in hertz. Mass spectra were obtained from a ThermoFinnigan MAT 95 XL double focusing sector mass spectrometer with electron spray ionization (ESI) technique or a Bruker Daltonics Autoflex MALDI-TOF mass spectrometer. Melting points were measured on an Electrothermal® 9100 digital melting point apparatus and were uncorrected. All reactions were performed under N_2 unless specified. Reactions were monitored by thin layer chromatography (TLC) performed on Merck pre-coated silica gel 60F254 plates. Compounds were visualized by ultra–violet radiation, followed by heating after immersing in 5% (w/v) dodecamolybdophosphoric acid in ethanol. Flash chromatography was carried out on columns of Merck Keiselgel 60 (230–400 mesh). Unless otherwise specified, all reagents were purchased from commercial suppliers and used without further purification. Prior to be used, THF was distilled from sodium/benzophenone ketyl under N_2. Toluene was freshly distilled from sodium under N_2 and CH_2Cl_2 was freshly distilled from CaH_2. DIPEA was distilled from NaOH under N_2 and stored in molecular sieve.

Infra-red spectroscopic (FTIR) experiments were performed using a Bruker Vertex 70 Fourier-transform spectrometer fitted with a globar source, a CaF_2 beam splitter, and a liquid nitrogen cooled HgCdTe detector. All FTIR spectra were recorded at 0.2 cm^{-1} resolution. The sample cell was made of a 0.5 mm Teflon spacer sandwiched by two 4-mm thick CaF_2 disks of 25 mm in diameter, with 0.1 mm optical path. Built-in ‘atmospheric compensation” routine from the spectrometer was used to minimize the background noise due to atmosphere and solvent (*p*-xylene). All FTIR experiments were performed using spectrophotometric grade *p*-xylene at 20 °C.

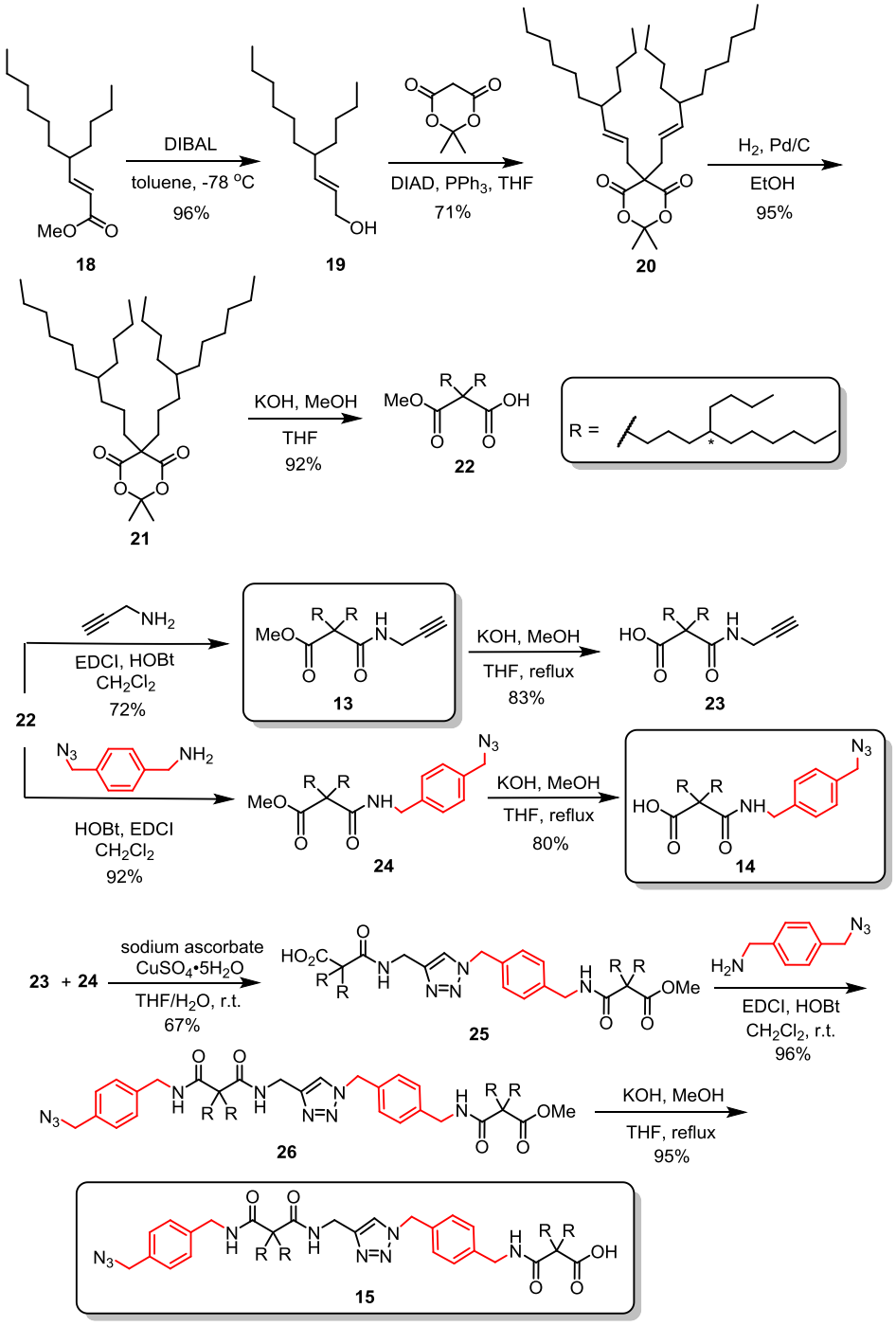
Scanning electron microscopy (SEM) was carried out by a FEI Quanta 400F field emission scanning electron microscope. All samples were xerogels prepared by freeze-

drying of the corresponding wet gel in *p*-xylene. The sample analyzed was stuck on the sample holder using a piece of double-sided adhesive tape and was sputter-deposited with minute amount of gold to prevent charging during analysis.

Differential scanning calorimetry (DSC) experiments were performed using a Mettler Toledo DSC StarIII calorimeter. Xerogels were prepared by freezing wet gels in *p*-xylene in liquid nitrogen and then removing the solvent using freeze-dry machine. Unless otherwise specified, all measurements on xerogels were performed by introducing 2~3 mg samples into the cell, followed by heating from 40 °C to 200 °C at 10 °C/min, annealing at 200 °C for 5 min and cooling down to 40 °C at 5 °C/min. After annealed once the sample was heated to 200 °C at 10 °C/min again and then cooled down to 40°C at 10 °C/min. For the wet gels in *p*-xylene, after transferred into the cell, the sample was annealed at 100 °C for 5 min, cooled down at 5 °C/min to 25 °C. After stabilized at 25 °C for 20 min, it was heated to 100 °C at 5 °C/min to furnish the heating curve. The heat capacitance data given were the results after baseline corrections.

2. Synthesis

(a) Synthesis of key intermediates **13**–**17**



Compound (*E*)-**19**: DIBAL (52.6 mL, 1 M in hexane, 52.6 mmol) was added in small portions to a stirred solution of compound **18**[1] (6.32 g, 26.3 mmol) in toluene (100 mL) at –78 °C. After stirring 8 h at room temperature, the reaction was quenched by pouring into ice-water and kept stirring for 30 min. HCl (1.2 M, 50 mL) was then added to the

[1] Leung, C.-F.; Chow, H. F. *Chem. Eur. J.* **2017**, *23*, 4827.

mixture, and extracted with Et_2O (3 × 150 mL). The combined extracts were washed with brine, dried ($NaSO_4$), filtered and concentrated *in vacuo* to give a colorless oil which was purified by chromatography (eluent: hexane/EtOAc = 9/1) to afford the target compound (*E*)-**19** (5.34 g, 96%) as a colorless oil. R_f = 0.35; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.57 (dt, CH=*CH*CH_2OH, *J* = 15.3 and 5.9 Hz, 1 H), 5.41 (dd, *CH*=$CHCH_2OH$, *J* = 15.4 and 8.7 Hz, 1 H), 4.10 (t, *CH*$_2$OH, *J* = 5.7 Hz, 2 H), 2.00–1.90 (m, *CH*CH=CH, 1 H), 1.34–1.20 (m, 17 H), 0.89–0.86 ppm (m, CH_3, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 137.7, 128.7, 63.6, 42.5, 35.3, 35.0, 31.9, 29.6, 29.5, 27.2, 22.9, 22.8, 14.1 ppm; accurate mass (ESI) calcd for $[C_{14}H_{28}O - H]^-$: 221.2067; found: 221.2067.

Compound (*E,E*)-**20**: DIAD (5.71 mL, 27.6 mmol) was added dropwise to a stirred solution of the allylic alcohol **19** (5.34 g, 25.1 mmol), PPh_3 (7.24 g, 27.6 mmol) and recrystallized Meldrum's acid (1.65 g, 11.4 mmol) in toluene (50 mL) at –10 °C. The solution was stirred at room temperature for 12 h and concentrated under reduced pressure. Hexane (50 mL) was then added to precipitate the Ph_3PO out. The mixture was filtered through a pad of Celite and the filtrate was concentrated *in vacuo* to give a pale oil which was purified by chromatography (eluent: hexane/Et_2O = 30/1) to afford the target compound (*E,E*)-**20** (4.35 g, 71%) as a colorless oil. R_f = 0.38; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.30 (dd, CH_2CH=*CH*, *J* = 15.2 and 8.7 Hz, 2 H), 5.17 (dt, CH_2*CH*=CH, *J* = 15.2 and 7.2 Hz, 2 H), 2.63 (d, *CH*$_2$C=C, *J* = 7.2 Hz, 4 H), 1.81 (m, *CH*CH=CH, 2 H), 1.63 (s, CCH_3, 6 H), 1.23–1.08 (m, 32 H), 0.83–0.79 ppm (m, CH_3, 12 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 168.9, 142.3, 121.7, 105.3, 55.8, 42.9, 42.5, 35.2, 34.8, 31.9, 30.0, 29.55, 29.54, 27.3, 22.9, 22.7, 14.12, 14.09 ppm; accurate mass (ESI) calcd for $[C_{34}H_{60}O_4 + Na]^+$: 555.4384; found: 555.4379.

Compound **21**: 10% Pd/C (0.36 g) was added to a solution of compound **20** (3.60 g, 6.7 mmol) in ethanol (50 mL) and the mixture was stirred under H_2 (1 atm) at room temperature for 12 h. The reaction mixture was then filtered through a pad of Celite and the filtrate was evaporated *in vacuo* to afford the target compound **21** (3.41 g, 95%) as

a colorless oil. R_f = 0.35 (hexane/EtOAc = 20/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.96–1.92 (m, CCH_2, 4 H), 1.71 (s, CCH_3, 6 H), 1.20–1.16 (m, 42 H), 0.87–0.84 ppm (m, CH_3, 12 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 169.7, 105.5, 55.1, 40.0, 37.2, 33.7, 33.6, 33.3, 32.0, 29.9, 29.8, 28.9, 26.7, 23.3, 23.2, 22.8, 14.2 ppm; accurate mass (ESI) calcd for $[C_{34}H_{64}O_4 + Na]^+$: 559.4697; found: 559.4691.

Compound **22**: KOH (1.50 mL, 2.5 M) was added to the Meldrum's acid **21** (0.51 g, 0.93 mmol) in THF/MeOH (*v*/*v* = 1/5, 24 mL). The reaction mixture was stirred at 25 °C for 24 h. Aqueous HCl (50 mL, 1.2 M) was added to the solution and the mixture was extracted with Et_2O (3 × 20 mL). The combined extracts were dried with Na_2SO_4, filtered and concentrated *in vacuo* to give a pale yellow oil which was purified by chromatography (eluent: hexane/EtOAc/HOAc = 240/30/1) to afford the target compound **22** (0.46 g, 92%) as a colorless oil. R_f = 0.29; ^{1}H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 3.78 (s, OCH_3, 3 H), 1.96–1.80 (m, CCH_2, 4 H), 1.25–1.18 (m, 42 H), 0.89–0.86 ppm (m, CH_3,12 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 175.7, 175.2, 58.0, 53.0, 37.1, 35.5, 33.8, 33.7, 33.6, 33.3, 33.2, 32.1, 29.9, 28.9, 26.7, 23.3, 22.8, 21.9, 14.29, 14.26 ppm; accurate mass (ESI) calcd for $[C_{32}H_{62}O_4 + Na]^+$: 533.4540; found: 533.4551.

Compound **13**: HOBt (0.14 g, 1.02 mmol) and EDCI (0.31 g, 1.02 mmol) were added in succession to a stirred solution of compound **22** (0.40 g, 0.78 mmol) in dichloromethane (DCM) (20 mL) at 0 °C. After 10 min, propargylamine (0.10 mL, 1.57 mmol) was added to the mixture. The reaction was allowed to stir at 25 °C for 12 h. H_2O (5 mL) was then poured into the solution and the reaction mixture was extracted with DCM (3 × 20 mL). The combined organic layers were washed with saturated $NaHCO_3$ (2 × 10 mL), saturated $NaHSO_4$ (2 × 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: hexane/Et_2O = 8/1) afforded the compound **13** as a pale yellow oil (0.31 g, 72%). R_f = 0.28; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.33 (t, NH, *J* = 4.8 Hz, 1 H), 4.07 (dd, CH≡CCH_2, *J* = 4.8 and 2.4 Hz, 2 H), 3.73 (s, OCH_3, 3 H), 2.18 (t, C≡CH, *J* = 2.4 Hz,

1 H), 2.01–1.95 (m, CCH_2, 2 H), 1.75–1.69 (m, CCH_2, 2 H), 1.21–1.16 (m, 42 H), 0.88–0.85 (m, CH_3, 12 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 176.3, 171.2, 79.8, 71.2, 58.2, 52.4, 38.3, 37.1, 33.72, 33.69, 33.57, 33.31, 33.21, 32.1, 29.9, 29.2, 28.94, 28.91, 26.69, 26.65, 23.3, 22.8, 22.6, 14.29, 14.25 ppm; accurate mass (ESI) calcd for $[C_{35}H_{65}NO_3 + Na]^+$: 570.4857; found: 570.4867.

Compound **23**: KOH (2.50 mL, 2.5 M) was added to compound **13** (0.41 g, 0.73 mmol) in THF/MeOH (*v*/*v* = 1/1, 20 mL). The reaction mixture was heated to reflux and stirred for 12 h. After cooling down to room temperature, the solution was quenched with aqueous HCl (10 mL, 1.2 M) and extracted with Et_2O (3 × 20 mL). The combined extracts were dried with Na_2SO_4, filtered and concentrated *in vacuo* to give a pale yellow oil which was purified by chromatography (eluent: hexane/EtOAc/HOAc = 400/40/1) to afford the target compound **23** (0.32 g, 83%) as a colorless oil. R_f = 0.21; ^1H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 6.83 (br s, NH, 1 H), 4.12 (dd, CH≡C*CH*$_2$, *J* = 5.3 and 2.5 Hz, 2 H), 2.25 (t, C≡CH, *J* = 2.5 Hz, 1 H), 2.10–2.04 (m, CCH_2, 2 H), 1.66–1.61 (m, CCH_2, 2 H), 1.29–1.17 (m, 42 H), 0.89–0.85 (m, CH_3, 12 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 175.5, 78.6, 72.2, 56.8, 39.8, 37.3, 33.8, 33.63, 33.61, 33.24, 33.21, 32.1, 29.9, 29.7, 29.0, 28.9, 26.73, 26.72, 23.2, 22.8, 22.6, 14.30, 14.26 ppm; accurate mass (ESI) calcd for $[C_{34}H_{63}NO_3 + Na]^+$: 556.4700; found: 556.4719.

Compound **24**: HOBt (0.95 g, 7.05 mmol) and EDCI (2.10 g, 7.05 mmol) were added in succession to a stirred solution of compound **22** (2.77 g, 5.42 mmol) in DCM (50 mL) at 0 °C. After 10 min, 4-(azidomethyl)benzylamine[2] (1.38 g, 8.51 mmol) was added to the mixture. The reaction solution was allowed to stir for 12 h at 25 °C. The reaction was quenched with water (20 mL) and extracted with DCM (3 × 40 mL). The organic layer was washed with saturated $NaHCO_3$ (2 × 30 mL), saturated $NaHSO_4$ (2 × 30 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of

[2] Lau, K.-N.; Chow, H.-F; Chan, M.-C.; Wong, K.-W. *Angew. Chem. Int. Ed.* **2008**, *47*, 6912.

the residue over silica gel (eluent: hexane/Et_2O = 8/1) afforded compound **24** as a colorless oil (3.25 g, 92%). R_f = 0.31; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.43 (t, NH, *J* = 5.6 Hz, 1 H), 7.32–7.25 (m, ArH, 4 H), 4.51 (d, NH*CH*$_2$Ar, *J* = 5.7 Hz, 2 H), 4.32 (s, Ar*CH*$_2$N$_3$, 2 H), 3.73 (s, OCH_3, 3 H), 2.06–1.99 (m, CCH_2, 2 H), 1.78–1.71 (m, CCH_2, 2 H), 1.31–1.23 (m, 40 H), 1.05–1.02 (m, *CH*CH_2CH_2, 2 H), 0.90–0.86 (m, CH_3, 12 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 176.5, 171.3, 139.0, 134.5, 128.6, 128.2, 58.3, 54.6, 52.4, 43.3, 38.1, 37.2, 33.8, 33.7, 33.6, 33.3, 33.2, 32.1, 30.0, 29.9, 29.0, 28.9, 26.72, 26.68, 23.3, 22.8, 22.7, 14.31, 14.26 ppm; accurate mass (ESI) calcd for [$C_{40}H_{70}N_4O_3$ + Na]$^+$: 677.5340; found: 677.5357.

Compound **14**: KOH (2.50 mL, 2.5 M) was added to compound **24** (0.37 g, 0.57 mmol) in THF/MeOH (*v*/*v* = 1/1, 20 mL). The reaction mixture was heated to reflux for 12 h. After cooling down to room temperature, the solution was quenched with aqueous HCl (10 mL, 1.2 M) and extracted with Et_2O (3 × 20 mL). The combined extracts were dried with Na_2SO_4, filtered and concentrated *in vacuo* to give a pale yellow oil which was purified by chromatography (eluent: hexane/EtOAc/HOAc = 240/30/1) to afford the target compound **14** (0.29 g, 80%) as a colorless oil. R_f = 0.25; ^{1}H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 7.32–7.27 (m, ArH, 4 H), 6.85 (br s, NH, 1 H), 4.53 (d, NH*CH*$_2$Ar, *J* = 5.6 Hz, 2 H), 4.34 (s, Ar*CH*$_2$N$_3$, 2 H), 2.12–2.01 (m, CCH_2, 2 H), 1.63–1.57 (m, CCH_2, 2 H), 1.30–1.17 (m, 42 H), 0.90–0.87 (m, CH_3, 12 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 176.1, 175.3, 137.4, 135.3, 128.8, 128.3, 56.6, 54.5, 43.7, 40.1, 37.3, 33.9, 33.6, 33.2, 32.1, 29.9, 29.0, 28.9, 26.8, 26.7, 23.2, 22.8, 22.7, 14.30, 14.26 ppm; accurate mass (ESI) calcd for [$C_{39}H_{68}N_4O_3$ – H]$^-$: 639.5219; found: 639.5219.

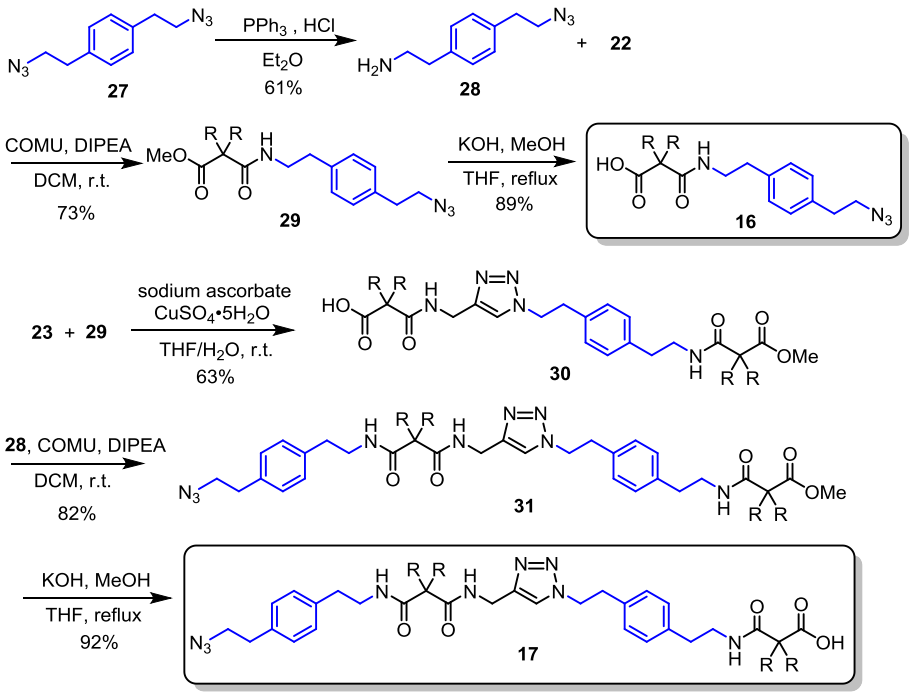
Compound **25**: Sodium ascorbate (0.23 g, 1.11 mmol) and $CuSO_4 \cdot 5H_2O$ (0.15 g, 0.46 mmol) were added to a solution of compound **24** (1.50 g, 2.29 mmol) and **23** (1.23 g, 2.29 mmol) in THF/H_2O (*v*/*v* = 9/1, 30 mL). The mixture was stirred at 25 °C for 12 h. Aqueous HCl (20 mL, 0.2 M) was poured into the solution and the reaction mixture was extracted with DCM (3 × 20 mL). The combined organic layers were washed with

H_2O (2 × 20 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: hexane/EtOAc/HOAc = 150/100/1) afforded compound **25** as a white solid (1.82 g, 67%). R_f = 0.35; M.p. 59–64 °C; ^{1}H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 8.49 (t, NH, *J* = 5.3 Hz, 1 H), 8.04 (br s, NH, 1 H), 7.46 (s, Triaz*H*, 1 H), 7.31–7.20 (m, ArH, 4 H), 5.45 (s, Ar*CH*$_2$Triaz, 2 H), 4.53–4.49 (m, NH*CH*$_2$Triaz and NH*CH*$_2$Ar, 4 H), 3.73 (s, OCH_3, 3 H), 2.05–1.95 (m, CCH_2, 4 H), 1.78–1.72 (m, CCH_2, 4 H), 1.24–1.00 (m, 84 H), 0.89–0.84 (m, CH_3, 24 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 176.5, 171.5, 144.5, 139.8, 133.0, 128.6, 128.5, 122.5, 58.3, 57.2, 54.3, 52.4, 43.2, 38.5, 38.2, 37.19, 37.18, 37.09, 37.07, 34.3, 33.9, 33.7, 33.59, 33.57, 33.5, 33.3, 33.2, 33.1, 32.10, 32.08, 29.96, 29.9, 28.92, 28.89, 26.72, 26.69, 26.6, 23.29, 23.27, 23.25, 22.8, 22.7, 22.6, 22.45, 22.42, 14.34, 14.29 ppm; accurate mass (ESI) calcd for $[C_{74}H_{133}N_5O_6 - H]^-$: 1187.0183; found: 1187.0183.

Compound **26**: COMU (0.26 g, 0.60 mmol) and DIPEA (0.20 mL, 1.10 mmol) were added in succession to a stirred solution of compound **25** (0.65 g, 0.55 mmol) in DCM (20 mL) under nitrogen at 0 °C. After 10 min, 4-(azidomethyl)benzylamine (0.27 g, 1.64 mmol) was added to the mixture. The reaction solution was allowed to stir at 25 °C for 12 h. H_2O (5 mL) was poured into the solution and the reaction mixture was extracted with DCM (3 × 20 mL). The combined organic layers were washed with saturated $NaHCO_3$ (2 × 10 mL), saturated $NaHSO_4$ (2 × 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: hexane/EtOAc = 2.5/1) afforded compound **26** as a colorless liquid (0.70 g, 96%). R_f = 0.32; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.44 (t, NH, *J* = 5.4 Hz, 1 H), 7.77 (t, NH, *J* = 5.5 Hz, 1 H), 7.46 (t, NH, *J* = 5.3 Hz, 1 H), 7.40 (s, Triaz*H*, 1 H), 7.29–7.19 (m, ArH, 8 H), 5.44 (s, Ar*CH*$_2$Triaz, 2 H), 4.50–4.45 (m, NH*CH*$_2$Triaz and NH*CH*$_2$Ar, 6 H), 4.31 (s, N_3*CH*$_2$Ar, 2 H), 3.72 (s, OCH_3, 3 H), 2.04–1.98 (m, CCH_2, 2 H), 1.90–1.84 (m, CCH_2, 2 H), 1.77–1.67 (m, CCH_2, 4 H), 1.29–0.99 (m, 84 H), 0.89–0.86 (m, CH_3, 24 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 176.5, 174.0, 172.9, 171.4, 144.8, 139.6, 138.7, 134.6, 133.6, 128.6, 128.5, 128.4, 128.2, 121.8, 58.3, 57.2, 54.6, 54.0, 52.4, 43.3,

43.2, 38.1, 37.2, 37.12, 37.09, 35.4, 34.1, 33.73, 33.71, 33.65, 33.59, 33.3, 33.2, 32.10, 32.07, 29.95, 29.93, 28.94, 28.92, 26.73, 26.70, 26.66, 23.3, 22.8, 22.7, 22.6, 22.3, 22.2, 14.33, 14.32, 14.27 ppm; accurate mass (ESI) calcd for $[C_{82}H_{141}N_9O_5 - H]^-$: 1355.0948; found: 1355.0951.

Compound **15**: KOH (0.50 mL, 2.5 M) was added to compound **26** (0.29 g, 0.22 mmol) in THF/MeOH (*v*/*v* = 1/1, 30 mL). The reaction mixture was heated to reflux and stirred for 12 h. After cooling down to room temperature, the solution was quenched with aqueous HCl (10 mL, 1.2 M) and extracted with DCM (3 × 15 mL). The combined extracts were dried with Na_2SO_4, filtered and concentrated *in vacuo* to give a pale yellow oil which was purified by chromatography (eluent: hexane/EtOAc/HOAc = 250/100/1) to afford the target compound **15** (0.27 g, 95%) as a colorless liquid. R_f = 0.24; ^{1}H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 7.67–7.64 (m, NH, 2 H), 7.41 (s, Triaz*H*, 1 H), 7.27–7.20 (m, ArH, 8 H), 7.03 (br s, NH, 1 H), 5.44 (s, Ar*CH*$_2$Triaz, 2 H), 4.50–4.47 (m, NHCH2Ar, 4 H), 4.44 (d, NH*CH*$_2$Triaz, *J* = 5.6 Hz, 2 H), 4.31 (s, N_3*CH*$_2$Ar, 2 H), 2.08–2.02 (m, CCH_2, 2 H), 1.88–1.64 (m, CCH_2, 6 H), 1.25–1.17 (m, 84 H), 0.89–0.85 (m, CH_3, 24 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 177.1, 174.2, 173.9, 172.9, 144.7, 139.2, 138.5, 134.5, 133.4, 128.5, 128.4, 128.3, 128.0, 122.1, 57.3, 57.0, 54.4, 54.0, 43.2, 38.6, 37.7, 37.2, 37.1, 34.6, 34.0, 33.9, 33.6, 33.5, 33.14, 33.08, 32.0, 31.98, 29.9, 28.8, 26.6, 23.2, 22.7, 22.6, 22.1, 14.23, 14.17 ppm; accurate mass (ESI) calcd for $[C_{81}H_{139}N_9O_5 - H]^-$: 1317.0826; found: 1317.0827.



Compound **28**: PPh_3 (2.89 g, 11.02 mmol) was added to a mixture of 1,4-bis-(2-azidoethyl)benzene **27**[3] (1.59 g, 8.45 mmol) in Et_2O (40 mL) and HCl (30 mL, 1 M) and the heterogeneous mixture was stirred vigorously at 25 °C for 24 h. The organic layer was then separated and the aqueous layer was extracted with Et_2O (3 × 50 mL) to remove the Ph_3PO and remaining starting materials. The pH of the aqueous layer was adjusted to 10 by adding KOH solution (2.5 M) and then extracted with DCM (4 × 50 mL). The combined extracts were washed with brine, dried Mg_2SO_4, filtered and evaporated in vacuo to give the product **28** as a pale yellow oil (0.84 g, 61%). It was used in the next reaction without further purification.

Compound **29**: COMU (2.62 g, 6.10 mmol) and DIPEA (1.32 g, 0.01 mol) were added in succession to a stirred solution of **22** (2.60 g, 5 mmol) in DCM (40 mL) at 0 °C. After 10 min, compound **28** (1.45 g, 7.64 mmol) was added to the mixture. The reaction solution was allowed to stir at 25 °C for 24 h. H_2O (10 mL) was poured to the solution and the reaction mixture was extracted with DCM (3 × 30 mL). The combined organic

[3] Schulz, M.; Christoffers, J. *Tetrahedron* **2013**, *69*, 802.

layers were washed with saturated $NaHCO_3$ (2 × 20 mL), saturated $NaHSO_4$ (2 × 20 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: hexane/EtOAc = 15/1) afforded **29** as a colorless liquid (2.50 g, 73%). R_f = 0.25; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.98 (t, NH, *J* = 5.6 Hz, 1 H), 7.18–7.13 (m, ArH, 4 H), 3.70 (s, OCH_3, 3 H), 3.55–3.47 (m, NH*CH*$_2$$CH_2$ and CH_2*CH*$_2$$N_3$, 4 H), 2.88–2.79 (m, CH_2*CH*$_2$Ar, 4 H), 2.00–1.93 (m, CCH_2, 2 H), 1.73–1.66 (m, CCH_2, 2 H), 1.25–1.17 (m, 42 H), 0.88–0.85 (m, CH_2*CH*$_3$, 12 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 176.3, 171.2, 137.8, 136.1, 129.1, 129.0, 58.1, 52.6, 52.3, 41.1, 37.7, 37.1, 35.6, 35.1, 33.8, 33.7, 33.6, 33.34, 33.27, 32.1, 30.0, 29.8, 28.94, 28.91, 26.70, 26.67, 23.3, 22.8, 22.5, 14.30, 14.25 ppm; accurate mass (ESI) calcd for $[C_{42}H_{74}N_4O_3 + Na]^+$: 705.5653; found: 705.5647.

Compound **16**: KOH (17 mL, 2.5 M) was added to compound **29** (5.94 g, 8.70 mmol) in THF/MeOH (*v*/*v* = 1/1, 100 mL). The reaction mixture was heated to reflux and stirred for 12 h. After cooling down to room temperature, the solution was quenched with aqueous HCl (40 mL, 1.2 M) and extracted with Et_2O (3 × 30 mL). The combined extracts were dried with Na_2SO_4, filtered and concentrated *in vacuo* to give a pale yellow oil which was purified by chromatography (eluent: hexane/EtOAc/HOAc = 240/30/1) to afford the target compound **16** (5.20 g, 89%) as a colorless oil. R_f = 0.22; ^{1}H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 7.20–7.13 (m, ArH, 4 H), 6.40 (br s, NH, 1 H), 3.55–3.47 (q, NH*CH*$_2$$CH_2$, *J* ~ 6.4 Hz, 2 H), 3.49 (t, CH_2*CH*$_2$$N_3$, *J* = 7.0 Hz, 2 H), 2.88 (t, Ar*CH*$_2$$CH_2$, *J* = 7.0 Hz, 2 H), 2.83 (t, CH_2*CH*$_2$Ar, *J* = 6.9 Hz, 2 H), 2.07–2.01 (m, CCH_2, 2 H), 1.46–1.40 (m, CCH_2, 2 H), 1.23–1.07 (m, 42 H), 0.88–0.85 (m, CH_2*CH*$_3$, 12 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 176.4, 175.7, 136.8, 136.7, 129.3, 129.0, 56.3, 52.5, 41.3, 39.9, 37.23, 37.22, 35.2, 35.1, 33.8, 33.6, 33.18, 33.16, 32.0, 29.9, 28.89, 28.85, 26.7, 26.6, 23.2, 22.8, 22.7, 22.6, 14.3, 14.2 ppm; accurate mass (ESI) calcd for $[C_{41}H_{72}N_4O_3 - H]^-$: 667.5532; found: 667.5534.

Compound **30**: Sodium ascorbate (0.074 g, 0.37 mmol) and $CuSO_4 \cdot 5H_2O$ (0.02 g, 0.08 mmol) were added to a solution of compounds **29** (0.28 g, 0.41 mmol) and **23** (0.20 g,

0.38 mmol) in THF/H_2O (*v*/*v* = 9/1, 15 mL). The mixture was stirred at 25 °C for 12 h. Aqueous HCl (10 mL, 0.2 M) was poured into the solution and the reaction mixture was extracted with DCM (3 × 10 mL). The combined organic layers were washed with H_2O (2 × 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: hexane/EtOAc/HOAc = 150/100/1) afforded compound **30** as a colorless liquid (0.29 g, 63%). R_f = 0.32; ^{1}H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 8.70 (br s, NH, 1 H), 8.22 (t, NH, *J* = 5.4 Hz, 1 H), 7.28 (s, Triaz*H*, 1 H), 7.14 (d, ArH, *J* = 7.9 Hz, 2 H), 6.98 (d, *J* = 7.9 Hz, ArH, 2 H), 4.51–4.48 (m, Triaz$CH_2$$CH_2$ and NHCH_2Triaz, 4 H), 3.70 (s, OCH_3, 3 H), 3.52 (q, CH_2CH_2NH, *J* ~ 6.9 Hz, 2 H), 3.13 (t, $ArCH_2$, *J* = 7.1 Hz, 2 H), 2.80 (t, $ArCH_2$, *J* = 7.4 Hz, 2 H), 1.99–1.68 (m, 8 H), 1.21–1.12 (m, 84 H), 0.86–0.84 (m, CH_2CH_3, 24 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 177.3, 176.3, 173.2, 171.5, 144.3, 138.1, 134.7, 129.3, 128.8, 123.0, 58.1, 57.4, 52.3, 52.1, 41.1, 38.1, 37.8, 37.2, 37.1, 36.4, 35.6, 34.1, 34.0, 33.70, 33.66, 33.62, 33.58, 33.3, 33.19, 33.16, 33.1, 32.1, 32.0, 29.92, 29.91, 28.89, 28.86, 26.68, 26.65, 26.6, 23.2, 22.8, 22.5, 22.4, 14.29, 14.26, 14.23, 14.22 ppm; accurate mass (ESI) calcd for $[C_{76}H_{137}N_5O_6 + Na]^+$: 1239.0461; found: 1239.0463.

Compound **31**: COMU (0.13 g, 0.30 mmol) and DIPEA (0.10 mL, 0.57 mmol) were added in succession to a stirred solution of compound **30** (0.29 g, 0.24 mmol) in DCM (10 mL) under nitrogen at 0 °C. After 10 min, 4-(azidoethyl)phenethylamine **28** (0.18 g, 0.96 mmol) was added to the mixture. The reaction solution was allowed to stir at 25 °C for 12 h. H_2O (5 mL) was poured into the solution and the reaction mixture was extracted with DCM (3 × 10 mL). The combined organic layers were washed with saturated $NaHCO_3$ (2 × 10 mL), saturated $NaHSO_4$ (2 × 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: hexane/EtOAc = 2/1) afforded compound **31** as a colorless liquid (0.27 g, 82%). R_f = 0.35; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.05 (t, NH, *J* = 5.6 Hz, 1 H), 7.76 (t, NH, *J* = 5.4 Hz, 1 H), 7.35 (s, Triaz*H*, 1 H), 7.17–7.05 (m, ArH and NH, 9 H), 4.52–4.48 (m, Triaz$CH_2$$CH_2$ and NHCH_2Triaz, 4 H), 3.70 (s, OCH_3, 3 H), 3.52–3.46 (m, CH_2CH_2NH

and $N_3CH_2CH_2$, 6 H), 3.15 (t, $ArCH_2$, J = 7.8 Hz, 2 H), 2.90–2.73 (m, 6 H), 2.00–1.94 (m, CCH_2, 2 H), 1.74–1.71 (m, CCH_2, 6 H), 1.22–1.17 (m, 84 H), 0.88–0.84 (m, CH_2CH_3, 24 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 176.4, 173.6, 173.2, 171.2, 144.6, 138.2, 137.5, 136.4, 135.0, 129.4, 129.2, 129.1, 128.9, 122.0, 58.2, 57.2, 52.6, 52.3, 51.8, 41.12, 41.04, 37.9, 37.8, 37.3, 37.1, 36.6, 35.7, 35.6, 35.3, 35.1, 34.1, 33.8, 33.73, 33.68, 33.35, 33.27, 32.1, 30.0, 29.8, 28.95, 28.92, 26.74, 26.71, 26.67, 23.3, 22.8, 22.5, 22.2, 14.31, 14.26 ppm; accurate mass (ESI) calcd for $[C_{86}H_{149}N_9O_5 + Na]^+$: 1411.1574; found: 1411.1564.

Compound **17**: KOH (0.50 mL, 2.5 M) was added to compound **31** (0.27 g, 0.19 mmol) in THF/MeOH (*v*/*v* = 1/1, 20 mL). The reaction mixture was heated to reflux and stirred for 12 h. After cooling down to room temperature, the solution was quenched with aqueous HCl (10 mL, 1.2 M) and extracted with DCM (3 × 10 mL). The combined extracts were dried with Na_2SO_4, filtered and concentrated *in vacuo* to give a pale yellow oil which was purified by chromatography (eluent: hexane/EtOAc/HOAc = 150/100/1) to afford the target compound **17** (0.24 g, 92%) as a colorless oil. R_f = 0.24; ^1H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 8.63 (br s, NH, 1 H), 7.64 (br s, NH, 1 H), 7.17–7.01 (m, Triaz*H* and ArH, 7 H), 6.89 (d, ArH, J = 7.8 Hz, 2 H), 6.53 (br s, NH, 1 H), 4.55 (t, TriazCH_2CH_2, J = 6.1 Hz, 2 H), 4.46 (d, NHCH_2Triaz, J = 5.6 Hz, 2 H), 3.63 (q, CH_2CH_2NH, J ~ 5.9 Hz, 2 H), 3.50–3.45 (m, CH_2CH_2NH, 4 H), 3.10 (t, $ArCH_2$, J = 6.1 Hz, 2 H), 2.86 (t, $ArCH_2$, J = 7.1 Hz, 2 H), 2.80–2.73 (m, $ArCH_2$, 4 H), 1.83–1.81 (m, CCH_2, 6 H), 1.58–1.51 (m, CCH_2, 2 H), 1.22–1.18 (m, 84 H), 0.88–0.85 (m, CH_2CH_3, 24 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 177.0, 173.8, 173.4, 143.9, 137.9, 137.3, 136.5, 135.5, 129.3, 129.24, 129.15, 129.06, 123.1, 57.3, 57.0, 52.6, 52.0, 41.0, 40.3, 39.0, 38.0, 37.4, 37.34, 37.28, 37.25, 36.7, 35.5, 35.3, 35.1, 35.0, 34.1, 33.8, 33.7, 33.6, 33.32, 33.28, 33.2, 32.1, 30.0, 28.95, 28.88, 26.8, 26.72, 26.68, 23.29, 23.27, 22.8, 22.2, 22.1, 14.32, 14.26 ppm; accurate mass (ESI) calcd for $[C_{85}H_{147}N_9O_5 + Na]^+$: 1397.1417; found: 1397.1413.

(b) Synthesis of **OAT-CO_2H-2n**, **OAT-COPrg(2n+1)** and **OAT-H-2n**

OAT-CO_2H-2: Sodium ascorbate (44 mg, 0.22 mmol) and $CuSO_4 \cdot 5H_2O$ (12 mg, 0.05 mmol) were added to a solution of compound **14** (0.29 g, 0.44 mmol) and **13** (0.31 g, 0.57 mmol) in THF/H_2O (*v*/*v* = 9/1, 20 mL). The mixture was stirred at 25 °C for 12 h. Aqueous HCl (10 mL, 0.2 M) was poured into the solution and the reaction mixture was extracted with DCM (3 × 20 mL). The combined organic layers were washed with H_2O (2 × 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: hexane/EtOAc/HOAc = 150/100/1) afforded **OAT-CO_2H-2** as a colorless oil (0.45 g, 86%). R_f = 0.28; ^{1}H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 8.47 (t, NH, *J* = 5.4 Hz, 1 H), 7.45 (s, Triaz*H*, 1 H), 7.34 (br s, NH, 1 H), 7.28–7.20 (m, ArH, 4 H), 5.44 (s, Ar*CH_2*Triaz, 2 H), 4.51–4.49 (d, NH*CH_2*Triaz and NH*CH_2*Ar, 4 H), 3.69 (s, OCH_3, 3 H), 2.03–1.88 (m, CCH_2, 4 H), 1.76–1.69 (m, CCH_2, 4 H), 1.25–1.17 (m, 84 H), 0.89–0.86 (m, CH_3, 24 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 175.7, 175.5, 175.4, 171.8, 145.3, 138.4, 134.2, 128.63, 128.58, 122.4, 58.1, 56.9, 53.9, 52.4, 43.6, 39.5, 37.4, 37.28, 37.25, 37.03, 37.00, 35.2, 33.91, 33.89, 33.80. 33.77, 33.7, 33.62, 33.56, 33.24, 33.21, 33.16, 32.1, 29.9, 29.8, 28.95, 28.91, 28.87, 26.74, 26.72, 26.68, 26.6, 23.2, 22.8, 22.72, 22.66, 22.4, 22.3, 14.31, 14.26 ppm; accurate mass (ESI) calcd for $[C_{74}H_{133}N_5O_6 - H]^-$: 1187.0183; found: 1187.0179.

OAT-COPrg-3: EDCI (0.95 g, 3.20 mmol) and HOBt (0.44 g, 3.20 mmol) were added in succession to a stirred solution of **OAT-CO_2H-2** (2.54 g, 2.14 mmol) in DCM (20 mL) at 0 °C. After 10 min, propargylamine (0.30 mL, 4.27 mmol) was added to the mixture. The reaction solution was allowed to stir at 25 °C for 12 h. H_2O (10 mL) was poured to the solution and the reaction mixture was extracted with DCM (3 × 30 mL). The combined organic layers were washed with saturated $NaHCO_3$ (2 × 20 mL), saturated $NaHSO_4$ (2 × 20 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: hexane/EtOAc = 3/1) afforded **OAT-COPrg-3** as a pale yellow oil (1.70 g, 65%). R_f = 0.35; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.36 (t, NH, *J* = 5.3 Hz, 1 H), 7.62 (t, NH, *J* = 5.5 Hz, 1 H), 7.44 (s, Triaz*H*,

1 H), 7.31 (br s, NH, 1 H), 7.31–7.19 (m, ArH, 4 H), 5.45 (s, Ar*CH*$_2$Triaz, 2 H), 4.52 (d, NH*CH*$_2$Ar, J = 5.4 Hz, 2 H), 4.46 (d, NH*CH*$_2$Triaz, J = 5.5 Hz, 2 H), 4.04 (dd, CH≡C*CH*$_2$, J = 5.1 and 2.4 Hz, 2 H), 3.70 (s, OCH_3, 3 H), 2.19 (t, J = 2.4 Hz, C≡CH, 1 H), 1.97–1.69 (m, CCH_2, 8 H), 1.29–1.00 (m, 84 H), 0.89–0.86 (m, CH_3, 24 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 175.7, 173.5, 173.1, 171.5, 145.4, 139.1, 133.9, 128.5, 128.4, 122.1, 79.4, 71.6, 58.1, 57.2, 53.9, 52.4, 43.4, 38.5, 37.4, 37.28, 37.27, 37.03, 37.01, 35.4, 34.0, 33.8, 33.7, 33.6, 33.26, 33.18, 32.1, 29.9, 29.4, 29.0, 28.91, 28.88, 26.74, 26.73, 26.7, 26.6, 23.3, 22.8, 22.39, 22.36, 22.23, 22.20, 14.30, 14.25 ppm; accurate mass (ESI) calcd for $[C_{77}H_{136}N_6O_5 + Na]^+$: 1248.0464; found: 1248.0461.

OAT-CO$_2$H-4: Sodium ascorbate (0.13 g, 0.66 mmol) and $CuSO_4 \cdot 5H_2O$ (0.04 g, 0.13 mmol) were added to a solution of **OAT-COPrg-3** (1.6 g, 1.31 mmol) and compound **14** (0.86 g, 1.31 mmol) in THF/H_2O (v/v = 9/1, 30 mL). The mixture was stirred at 25 °C for 12h. Aqueous HCl (20 mL, 0.2 M) was poured into the solution and the reaction mixture was extracted with DCM (3 × 30 mL). The combined organic layers were washed with H_2O (2 × 20 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: hexane/EtOAc/HOAc = 300/200/1) afforded **OAT-CO$_2$H-4** as a white solid (1.90 g, 79%). R_f = 0.25; M.p. 69–73 °C; ^{1}H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 8.48 (t, NH, J = 5.3 Hz, 1 H), 8.13 (br s, NH, 1 H), 8.07 (br s, NH, 1 H), 7.56 (br s, NH, 1 H), 7.50 (s, Triaz*H*, 1 H), 7.41 (s, Triaz*H*, 1 H), 7.29–7.16 (m, ArH, 8 H), 5.43 (s, Triaz*CH*$_2$Ar, 2 H), 5.42 (s, Triaz*CH*$_2$Ar, 2 H), 4.50–4.41 (m, NH*CH*$_2$Triaz and Ar*CH*$_2$NH, 8 H), 3.68 (s, OCH_3, 3 H), 1.96–1.89 (m, CCH_2, 8 H), 1.77–1.72 (m, CCH_2, 4 H), 1.24–1.00 (m, 126 H), 0.89–0.85 (m, CH_3, 36 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 177.2, 175.4, 174.2, 173.5, 172.8, 171.7, 145.1, 144.6, 139.4, 139.3, 133.5, 133.3, 128.32, 128.26, 128.2, 122.3, 121.9, 58.0, 57.4, 57.1, 53.9, 52.2, 43.1, 38.5, 37.7, 37.1, 37.0, 36.9, 35.0, 34.8, 33.9, 33.7, 33.6, 33.5, 33.4, 33.12, 33.08, 33.0, 31.9, 29.8, 28.8, 26.6, 26.5, 23.1, 22.7, 22.6, 22.13, 22.06, 14.2, 14.1 ppm; accurate mass (ESI) calcd for $[C_{116}H_{204}N_{10}O_8 + 2Na]^{2+}$: 956.2840; found: 956.2832.

OAT-COPrg-5: EDCI (0.51 g, 1.70 mmol) and HOBt (0.23 g, 1.70 mmol) were added in succession to a stirred solution of **OAT-CO_2H-4** (2.01 g, 1.10 mmol) in DCM (30 mL) at 0 °C. After 10 min, propargylamine (0.15 mL, 2.20 mmol) was added to the mixture. The reaction solution was allowed to stir for 12 h at 25 °C. H_2O (10 mL) was poured into the solution and the reaction mixture was extracted with DCM (3 × 30 mL). The combined organic layers were washed with saturated $NaHCO_3$ (2 × 20 mL), saturated $NaHSO_4$ (2 × 20 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: hexane/EtOAc = 2/1) afforded **OAT-COPrg-5** as a white solid (1.60 g, 78%). R_f = 0.25; M.p. 107–110 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.36 (t, NH, *J* = 5.4 Hz, 1 H), 7.80 (t, NH, *J* = 5.3 Hz, 1 H), 7.71, (t, NH, *J* = 5.3 Hz, 1 H), 7.44 (s, Triaz*H*, 1 H), 7.39 (s, Triaz*H*, 1 H), 7.39 (br s, NH, 1 H), 7.27–7.17 (m, ArH and NH, 9 H), 5.443 (s, Ar*CH*$_2$Triaz, 2 H), 5.442 (s, Ar*CH*$_2$Triaz, 2 H), 4.52–4.42 (m, NH*CH*$_2$Ar and NH*CH*$_2$Triaz, 8 H), 4.04 (dd, CH≡C*CH*$_2$, *J* = 5.2 and 2.5 Hz, 2 H), 3.69 (s, OCH_3, 3 H), 2.19 (t, *J* = 2.5 Hz, C≡CH, 1 H), 1.97–1.64 (m, CCH_2, 12 H), 1.29–1.15 (m, 126 H), 0.89–0.85 (m, CH_3, 36 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 175.7, 174.0, 173.5, 173.1, 172.9, 171.6, 145.4, 144.7, 139.3, 133.8, 133.7, 128.5, 128.40, 128.38, 122.2, 121.8, 79.4, 71.6, 58.1, 57.3, 57.2, 54.0, 53.9, 52.4, 43.32, 43.30, 38.4, 37.9, 37.34, 37.29, 37.27, 37.21, 37.02, 37.00, 35.36, 35.33, 34.0, 33.8, 33.65, 33.56, 33.3, 33.21, 33.20, 33.17, 32.1, 29.9, 29.4, 29.0, 28.91, 28.87, 26.74, 26.70, 26.6, 23.3, 22.8, 22.4, 22.3, 22.2, 14.31, 14.26 ppm; accurate mass (ESI) calcd for $[C_{119}H_{207}N_{11}O_7 + Na]^+$: 1926.6104; found: 1926.6097.

OAT-CO_2H-6: Sodium ascorbate (81 mg, 0.41 mmol) and $CuSO_4 \cdot 5H_2O$ (30 mg, 0.12 mmol) were added to a solution of **OAT-COPrg-3** (1.01 g, 0.82 mmol) and **15** (1.30 g, 0.98 mmol) in THF/H_2O (*v*/*v* = 9/1, 30 mL). The solution was stirred at 25 °C for 24 h. Aqueous HCl (20 mL, 0.2 M) was poured into the solution and the reaction mixture was extracted with DCM (3 × 30 mL). The combined organic layers were washed with H_2O (2 × 20 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: $CHCl_3$/MeOH = 20/1) afforded **OAT-CO_2H-6** as a white solid (1.85 g, 89%). R_f = 0.35; M.p. 104–108 °C; ^{1}H NMR

(400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 8.40 (t, NH, *J* = 5.2 Hz, 1 H), 8.04 (br s, NH, 1 H), 7.93 (br s, NH, 1 H), 7.89 (br s, NH, 1 H), 7.68 (br s, NH, 1 H), 7.46–7.45 (m, NH and Triaz*H*, 3 H), 7.40 (s, Triaz*H*, 1 H), 7.28–7.16 (m, ArH, 12 H), 5.43–5.42 (m, TriazCH_2Ar, 6 H), 4.51–4.41 (m, NHCH_2Triaz and ArCH_2NH, 12 H), 3.67 (s, OCH_3, 3 H), 1.93–1.87 (m, CCH_2, 12 H), 1.76–1.74 (m, CCH_2, 4 H), 1.22–1.15 (m, 168 H), 0.89–0.85 (m, CH_3, 48 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 176.7, 175.6, 174.13, 174.06, 172.9, 172.8, 171.6, 145.3, 144.71, 144.68, 139.4, 139.22, 139.19, 133.7, 133.6, 133.5, 128.5, 128.42, 128.38, 128.37, 128.34, 128.31, 122.2, 122.0, 121.9, 58.1, 57.4, 57.22, 57.18, 54.0, 53.94, 53.89, 52.3, 43.26, 43.21, 38.6, 37.8, 37.6, 37.21, 37.18, 36.98, 36.96, 35.23, 35.15, 35.0, 34.0, 33.8, 33.7, 33.6, 33.5, 33.21, 33.17, 32.0, 29.9, 28.9, 26.7, 26.6, 23.2, 22.8, 22.6, 22.30, 22.26, 22.2, 14.3, 14.2 ppm; accurate mass (ESI) calcd for $[C_{158}H_{275}N_{15}O_{10} - H + 2Na]^+$: 2589.1215; found: 2589.1205.

OAT-COPrg-7: COMU (0.21 g, 0.48 mmol) and DIPEA (0.14 mL,0.80 mmol) were added in succession to a stirred solution of **OAT-CO_2H-6** (1.01 g, 0.40 mmol) in DCM (30 mL) at 0 °C. After 10 min, propargylamine (0.25 mL, 4.10 mmol) was added to the mixture. The reaction solution was allowed to stir for 24 h at 25 °C. H_2O (10 mL) was poured into the solution and the reaction mixture was extracted with DCM (3 × 30 mL). The combined organic layers were washed with saturated $NaHCO_3$ (2 × 20 mL), saturated $NaHSO_4$ (2 × 20 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: $CHCl_3$/MeOH = 25/1) afforded **OAT-COPrg-7** as a white solid (0.82 g, 80%). R_f = 0.31; M.p. 123–128 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.36 (t, NH, *J* = 5.3 Hz, 1 H), 7.89 (t, NH, *J* = 5.6 Hz, 1 H), 7.84 (t, NH, *J* = 5.4 Hz, 1 H), 7.77 (t, NH, *J* = 5.6 Hz, 1 H), 7.48 (br s, NH, 1 H), 7.44 (s, Triaz*H*, 1 H), 7.40 (br s, NH and 2 Triaz*H*, 3 H), 7.27–7.17 (m, NH and ArH, 13 H), 5.44 (s, ArCH_2Triaz, 6 H), 4.52–4.42 (m, NHCH_2Ar and NHCH_2Triaz, 12 H), 4.04 (dd, CH≡CCH_2, *J* = 5.2 and 2.5 Hz, 2 H), 3.69 (s, OCH_3, 3 H), 2.19 (t, *J* = 2.5 Hz, C≡CH, 1 H), 1.97–1.74 (m, CCH_2, 16 H), 1.22–1.15 (m, 168 H), 0.89–0.85 (m, CH_3, 48 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 175.7, 174.00, 173.97, 173.6, 173.1, 172.9, 171.5,

145.4, 144.70, 144.65, 139.5, 139.3, 133.8, 133.7, 133.6, 128.5, 128.40, 128.38, 122.1, 121.74, 121.71, 79.4, 71.6, 58.1, 57.25, 57.18, 53.95, 53.88, 52.4, 43.3, 38.3, 37.9, 37.34, 37.28, 37.2, 37.02, 36.99, 35.4, 34.0, 33.8, 33.65, 33.56, 33.3, 33.2, 32.1, 29.9, 29.4, 29.0, 28.90, 28.87, 26.7, 26.6, 23.3, 22.8, 22.4, 22.3, 22.2, 14.32, 14.26 ppm; accurate mass (ESI) calcd for $[C_{161}H_{278}N_{16}O_9 + Na]^+$: 2604.1712; found: 2604.1719.

OAT-CO$_2$H-8: Sodium ascorbate (23 mg, 0.12 mmol) and $CuSO_4 \cdot 5H_2O$ (14 mg, 0.05 mmol) were added to a solution of **OAT-COPrg-5** (0.44 g, 0.24 mmol) and compound **15** (0.50 g, 0.38 mmol) in THF/H_2O (*v*/*v* = 9/1, 20 mL). The mixture was stirred at 25 °C for 24 h. Aqueous HCl (10 mL, 0.2 M) was poured into the solution and the reaction mixture was extracted with DCM (3 × 20 mL). The combined organic layers were washed with H_2O (2 × 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: $CHCl_3$/MeOH = 20/1) afforded **OAT-CO$_2$H-8** as a white solid (0.74 g, 96%). R_f = 0.31; M.p. 128–130 °C; ^{1}H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 8.39 (t, NH, *J* = 5.4 Hz, 1 H), 7.98 (br s, NH, 1 H), 7.89 (br s, NH, 1 H), 7.81 (br s, NH, 1 H), 7.74 (br s, NH, 1 H), 7.63 (br s, NH, 1 H), 7.58 (br s, NH, 1 H), 7.45 (s, Triaz*H*, 1 H), 7.44 (s, Triaz*H*, 1 H), 7.42 (s, Triaz*H*, 1 H), 7.39 (br s, NH and Triaz*H*, 2 H), 7.27–7.16 (m, ArH, 16 H), 5.43–5.42 (m, Triaz*CH*$_2$Ar, 8 H), 4.51–4.41 (m, NH*CH*$_2$Triaz and Ar*CH*$_2$NH, 16 H), 3.68 (s, OCH_3, 3 H), 1.97–1.87 (m, CCH_2, 14 H), 1.76–1.74 (m, CCH_2, 6 H), 1.21–1.15 (m, 210 H), 0.89–0.85 (m, CH_3, 60 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 176.5, 175.6, 174.10, 174.07, 174.0, 172.94, 172.90, 172.8, 171.6, 145.3, 144.7, 139.5, 139.4, 139.3, 139.2, 133.74, 133.67, 133.6, 133.5, 128.5, 128.41, 128.39, 128.37, 128.3, 122.2, 122.0, 121.9, 58.1, 57.4, 57.3, 54.03, 53.97, 53.9, 52.3, 43.3, 43.2, 38.5, 37.8, 37.7, 37.4, 37.2, 37.01, 36.99, 35.3, 35.23, 35.15, 35.1, 34.1, 33.8, 33.63, 33.55, 33.24, 33.19, 32.1, 29.92, 28.89, 28.86, 26.7, 26.6, 23.2, 22.8, 22.6, 22.33, 22.29, 22.2, 22.1, 14.3, 14.2 ppm; accurate mass (ESI) calcd for $[C_{200}H_{346}N_{20}O_{12} - H + 3Na]^{2+}$: 1645.3373; found: 1645.3368.

OAT-COPrg-9: COMU (97 mg, 0.23 mmol) and DIPEA (0.07 mL,0.38 mmol) were

added in succession to a stirred solution of **OAT-CO_2H-8** (0.60 g, 0.19 mmol) in DCM (20 mL) at 0 °C. After 10 min, propargylamine (0.24 mL, 3.60 mmol) was added to the mixture. The reaction solution was allowed to stir at 25 °C for 48 h. H_2O (5 mL) was poured into the solution and the reaction mixture was extracted with DCM (3 × 20 mL). The combined organic layers were washed with saturated $NaHCO_3$ (2 × 10 mL), saturated $NaHSO_4$ (2 × 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: $CHCl_3$/MeOH = 25/1) afforded **OAT-COPrg-9** as a white solid (0.49 g, 79%). R_f = 0.28; M.p. 139–143 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.35 (t, NH, *J* = 5.3 Hz, 1 H), 7.88 (br s, NH, 2 H), 7.82 (br s, NH, 2 H), 7.74 (t, NH, *J* = 5.6 Hz, 1 H), 7.49–7.42 (m, NH and Triaz*H*, 3 H), 7.39–7.36 (m, NH and Triaz*H*, 5 H), 7.25–7.17 (m, ArH, 16 H), 5.43 (br s, Ar*CH_2*Triaz, 8 H), 4.51–4.42 (m, NH*CH_2*Ar and NH*CH_2*Triaz, 16 H), 4.03 (dd, CH≡C*CH_2*, *J* = 5.0 and 2.3 Hz, 2 H), 3.68 (s, OCH_3, 3 H), 2.19 (t, C≡CH, *J* = 2.4 Hz, 1 H), 1.97–1.60 (m, CCH_2, 20 H), 1.28–1.15 (m, 210 H), 0.88–0.84 (m, CH_3, 60 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 175.6, 174.0, 173.6, 173.1, 172.9, 171.5, 145.3, 144.72, 144.67, 139.5, 139.34, 139.31, 133.8, 133.64, 133.58, 132.2, 132.11, 132.07, 132.04, 128.7, 128.5, 128.44, 128.37, 128.3, 122.1, 121.7, 79.4, 71.5, 58.1, 57.3, 57.2, 53.92, 53.86, 52.3, 43.3, 43.2, 38.2, 37.8, 37.3, 37.2, 37.01, 36.99, 35.33, 35.29, 34.0, 33.8, 33.64, 33.56, 33.25, 33.2, 32.0, 29.9, 29.3, 28.93, 28.89, 26.7, 26.6, 23.2, 22.8, 22.32, 22.28, 22.2, 14.3, 14.2 ppm; accurate mass (MALDI) calcd for $[C_{203}H_{349}N_{21}O_{11} + Na]^+$: 3282.7351; found: 3282.7380.

OAT-CO_2H-10: Sodium ascorbate (30 mg, 0.15 mmol) and $CuSO_4 \cdot 5H_2O$ (20 mg, 0.08 mmol) were added to a solution of **OAT-COPrg-7** (0.70 g, 0.27 mmol) and **15** (0.24 g, 0.31 mmol) in THF/H_2O (*v*/*v* = 9/1, 20 mL). The mixture was stirred at 25 °C for 48 h. Aqueous HCl (10 mL, 0.2 M) was poured into the solution and the reaction mixture was extracted with DCM (3 × 20 mL). The combined organic layers were washed with H_2O (2 × 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: $CHCl_3$/MeOH = 20/1) afforded **OAT-CO_2H-10** as a white solid (0.84 g, 80%). R_f = 0.21; M.p. 144–152 °C; ^{1}H NMR

(400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 8.37 (br s, NH, 1 H), 7.94–7.88 (m, NH, 4 H), 7.74 (br s, NH, 1 H), 7.59–7.54 (m, NH, 3 H), 7.45–7.39 (m, NH and Triaz*H*, 6 H), 7.27–7.17 (m, Ar*H*, 20 H), 5.43 (br s, Triaz*CH*$_2$Ar, 10 H), 4.51–4.41 (m, NH*CH*$_2$Triaz and Ar*CH*$_2$NH, 20 H), 3.68 (s, OCH_3, 3 H), 1.97–1.74 (m, CCH_2, 24 H), 1.21–1.15 (m, 252 H), 0.89–0.85 (m, CH_3, 72 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 174.12, 174.10, 174.07, 174.04, 172.94, 172.91, 171.6, 145.3, 144.7, 139.5, 139.4, 139.3, 133.8, 133.6, 133.5, 128.5, 128.41, 128.37, 128.32, 122.2, 122.0, 121.9, 58.1, 57.4, 57.3, 57.2, 54.02, 53.97, 53.9, 52.4, 43.32, 43.27, 43.24, 37.9, 37.8, 37.6, 37.4, 37.2, 37.01, 36.99, 35.3, 35.2, 34.0, 33.78, 33.76, 33.7, 33.63, 33.55, 33.24, 33.19, 33.18, 32.1, 29.9, 28.90, 28.86, 26.70, 26.69, 26.6, 23.2, 22.8, 22.6, 22.34, 22.30, 22.2, 22.1, 14.31, 14.26 ppm; accurate mass (ESI) calcd for $[C_{242}H_{417}N_{25}O_{14} + 2Na]^{2+}$: 1973.1268; found: 1973.1260.

OAT-COPrg-11: COMU (14 mg, 0.033 mmol) and DIPEA (0.01 mL,0.06 mmol) were added in succession to a stirred solution of **OAT-CO$_2$H-10** (0.10 g, 0.026 mmol) in DMF (10 mL) at 0 °C. After 10 min, propargylamine (0.02 mL, 0.30 mmol) was added to the mixture. The reaction solution was allowed to stir at 50 °C for 48 h. H_2O (3 mL) was poured into the solution and the reaction mixture was extracted with DCM (3 × 10 mL). The combined organic layers were washed with saturated $NaHCO_3$ (2 × 10 mL), saturated $NaHSO_4$ (2 × 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: $CHCl_3$/MeOH = 20/1) afforded **OAT-COPrg-11** as a white solid (0.08 g, 78%). R_f = 0.25; M.p. 151–155 °C; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.34 (br s, NH, 1 H), 7.90 (br s, NH, 3 H), 7.84 (br s, NH, 1 H), 7.76 (br s, NH, 1 H), 7.43–7.39 (m, NH and Triaz*H*, 10 H), 7.24–7.16 (m, Ar*H*, 20 H), 5.42 (br s, Ar*CH*$_2$Triaz, 10 H), 4.50–4.41 (m, NH*CH*$_2$Ar and NH*CH*$_2$Triaz, 20 H), 4.03 (br s, CH≡C*CH*$_2$, 2 H), 3.67 (s, OCH_3, 3 H), 2.18 (br s, C≡CH, 1 H), 1.87–1.73 (m, CCH_2, 24 H), 1.20–1.14 (m, 252 H), 0.88–0.85 (m, CH_3, 72 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 175.7, 174.0, 173.6, 173.1, 172.9, 171.6, 145.4, 144.7, 139.5, 139.3, 133.8, 133.65, 133.59, 128.5, 128.40, 128.38, 122.1, 121.8, 79.4, 71.6, 58.1, 57.25, 57.19, 53.95, 53.90, 52.4, 43.3, 38.3, 37.9, 37.8, 37.3, 37.2, 37.0, 35.3, 34.0, 33.8, 33.6, 33.2,

32.1, 29.9, 29.4, 28.9, 26.7, 23.2, 22.8, 22.3, 22.2, 14.30, 14.26 ppm; accurate mass (ESI) calcd for $[C_{245}H_{420}N_{26}O_{13} + 2Na]^{2+}$: 1991.6426; found: 1991.6429.

OAT-CO$_2$H-12: Sodium ascorbate (8 mg, 0.04 mmol) and $CuSO_4 \cdot 5H_2O$ (4 mg, 0.02 mmol) were added to a solution of **OAT-COPrg-9** (0.21 g, 0.06 mmol) and compound **15** (86 mg, 0.065 mmol) in THF/H_2O (*v*/*v* = 9/1, 5 mL). The mixture was stirred at 25 °C for 3 d. Aqueous HCl (5 mL, 0.2 M) was poured into the solution and the reaction mixture was extracted with $CHCl_3$ (3 × 10 mL). The combined organic layers were washed with H_2O (2 × 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: $CHCl_3$/MeOH = 20/1) afforded **OAT-CO$_2$H-12** as a white solid (0.18 g, 66%). R_f = 0.21; M.p. 156–161 °C; ^{1}H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 8.36 (br s, NH, 1 H), 7.92 (2 br s, NH, 5 H), 7.59 (br s, NH, 2 H), 7.51 (3 br s, NH, 3 H), 7.44–7.38 (m, NH and Triaz*H*, 7 H), 7.27–7.17 (m, Ar*H*, 24 H), 5.42 (br s, Triaz*CH$_2$*Ar, 12 H), 4.51–4.41 (m, NH*CH$_2$*Triaz and Ar*CH$_2$*NH, 24 H), 3.68 (s, OCH_3, 3 H), 1.87–1.74 (m, CCH_2, 28 H), 1.21–1.15 (m, 294 H), 0.87–0.85 (m, CH_3, 84 H); ^{13}C NMR (400 MHz, $CDCl_3$): δ = 175.7, 174.08, 174.06, 174.0, 172.9, 172.7, 171.6, 145.3, 144.72, 144.70, 139.44, 139.38, 139.3, 139.0, 133.8, 133.7, 133.6, 133.5, 128.5, 128.42, 128.38, 128.33, 128.30, 122.2, 122.1, 121.9, 121.8, 58.1, 57.4, 57.3, 57.2, 54.02, 53.95, 53.9, 52.4, 43.33, 43.27, 43.2, 38.5, 37.9, 37.8, 37.71, 37.6, 37.3, 37.2, 36.99, 36.96, 35.3, 35.23, 35.18, 35.0, 34.0, 33.74, 33.72, 33.66, 33.6, 33.5, 33.22, 33.17, 33.15, 32.1, 30.0, 29.8, 28.89, 28.85, 26.69, 26.68, 26.66, 26.6, 23.3, 22.9, 22.6, 22.34, 22.30, 22.2, 22.1, 14.4, 14.3 ppm; accurate mass (ESI) calcd for $[C_{284}H_{488}N_{30}O_{16} + 2Na]^{2+}$: 2312.4087; found: 2312.4092.

OAT-COProp-7: *N*-(3-Dimethylaminopropyl)-*N*'-ethylcarbodiimide hydrochloride (9.4 mg, 0.05 mmol) and HOBt (5.10 mg, 0.038 mmol) were added in succession to a stirred solution of **OAT-CO$_2$H-6** (95 mg, 0.038 mmol) in DCM (10 mL) at 0 °C,. After 10 min, propylamine (0.062 mL, 0.75 mmol) was added to the mixture. The reaction solution was allowed to stir at 40 °C for 12 h. H_2O (5 mL) was poured into the solution and the reaction mixture was extracted with DCM (3 × 10 mL). The combined organic

layers were washed with saturated $NaHCO_3$ (2 × 10 mL), saturated $NaHSO_4$ (2 × 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: $CHCl_3$/EtOAc = 4/1) afforded **OAT-COProp-7** as a white solid (83 mg, 85%). R_f = 0.34; M.p. 124–128 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.34 (br s, NH, 1 H), 8.12 (br s, NH, 1 H), 7.91 (br s, NH, 1 H), 7.84 (br s, NH, 1 H), 7.43 (br s, NH and Triaz*H*, 2 H), 7.39–7.38 (m, NH and Triaz*H*, 3 H), 7.24–7.16 (2 m, ArH, 12 H), 6.66 (br s, NH, 1 H), 5.43 (m, Triaz*CH*$_2$Ar, 6 H), 4.51–4.41 (m, NH*CH*$_2$Triaz and Ar*CH*$_2$NH, 12 H), 3.68 (s, OCH_3, 3 H), 3.21 (q, NH*CH*$_2$$CH_2CH_3$, J ~ 6.5 Hz, 2 H), 1.92–1.60 (m, 16 H), 1.54–1.49 (m, $NHCH_2$*CH*$_2$$CH_3$, 2 H), 1.25–1.15 (m, 168 H), 0.89–0.85 (m, CH_2*CH*$_3$, 51 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 176.1, 175.7, 174.0, 173.8, 173.2, 172.9, 171.5, 145.4, 144.7, 144.6, 139.52, 139.47, 139.3, 133.8, 133.6, 133.5, 128.4, 122.1, 121.75, 121.68, 58.1, 57.2, 57.1, 53.94, 53.88, 52.3, 43.2, 41.4, 38.3, 37.9, 37.3, 37.2, 37.0, 35.3, 34.0, 33.8, 33.6, 33.5, 33.3, 33.23, 33.19, 32.1, 29.9, 29.0, 28.9, 26.74, 26.68, 26.6, 23.2, 22.8, 22.3, 22.2, 14.3, 14.2, 11.5 ppm; accurate mass (MALDI) calcd for $[C_{161}H_{282}N_{16}O_9 + Na]^+$: 2586.2206; found: 2586.2221.

OAT-H-6: The compound was recovered from a xerogel sample of **OAT-CO$_2$H-6** after it was subjected to annealing at 200 ºC for 5 min in the DSC instrument. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.35 (t, NH, J = 5.3 Hz, 1 H), 7.80 and 7.79 (2 br s, NH, 2 H), 7.43–7.39 (m, NH and Triaz*H*, 5 H), 7.28–7.17 (m, ArH, 12 H), 5.74 (t, NH, J = 6.0 Hz, 1 H), 5.44–5.43 (m, Triaz*CH*$_2$Ar, 6 H), 4.51–4.42 (m, NH*CH*$_2$Triaz and Ar*CH*$_2$NH, 12 H), 3.68 (s, OCH_3, 3 H), 2.04 (br s, CH, 1 H), 1.92–1.63 (m, CCH_2, 16 H), 1.20–1.14 (m, 168 H), 0.88–0.86 (m, CH_3, 48 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 176.2, 175.8, 174.0, 173.0, 171.6, 145.5, 144.7, 139.7, 139.5, 133.9, 128.6, 128.5, 128.43, 128.40, 122.2, 121.8, 58.2, 57.3, 54.0, 52.4, 48.3, 43.1, 37.9, 37.5, 37.2, 37.1, 35.4, 34.1, 33.9, 33.8, 33.7, 33.42, 33.39, 33.2, 32.1, 30.0, 29.0, 28.9, 26.8, 26.7, 25.1, 23.3, 22.9, 22.4, 22.2, 14.33, 14.28 ppm; accurate mass (MALDI) calcd for $[C_{157}H_{275}N_{15}O_8 + Na]^+$: 2523.1497; found: 2523.1495.

OAT-H-8: The compound was recovered from a xerogel sample of **OAT-CO$_2$H-8** after it was subjected to annealing at 200 ºC for 5 min in the DSC instrument. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.35 (t, NH, *J* = 5.3 Hz, 1 H), 7.86 (t, NH, *J* = 5.3 Hz, 1 H), 7.82 (t, NH, *J* = 5.8 Hz, 2 H), 7.44–7.39 (m, NH and Triaz*H*, 7 H), 7.28–7.17 (m, ArH, 16 H), 5.74 (t, NH, *J* = 5.7 Hz, 1 H), 5.44–5.43 (m, Triaz*CH*$_2$Ar, 8 H), 4.51–4.42 (m, NH*CH*$_2$Triaz and Ar*CH*$_2$NH, 16 H), 3.68 (s, OCH_3, 3 H), 2.04 (br s, CH, 1 H), 2.02–1.74 (m, CCH_2, 20 H), 1.20–1.14 (m, 210 H), 0.88–0.86 (m, CH_3, 60 H); accurate mass (MALDI) calcd for $[C_{199}H_{346}N_{20}O_{10} + Na]^+$: 3201.7137; found: 3201.7151.

OAT-H-10: The compound was recovered from a xerogel sample of **OAT-CO$_2$H-10** after it was subjected to annealing at 200 ºC for 5 min in the DSC instrument. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.35 (t, NH, *J* = 5.5 Hz, 1 H), 7.89–7.81 (m, NH, 4 H), 7.46–7.39 (m, NH and Triaz*H*, 9 H), 7.46–7.16 (m, ArH, 20 H), 5.74 (t, NH, *J* = 5.7 Hz, 1 H), 5.44–5.43 (m, Triaz*CH*$_2$Ar, 10 H), 4.51–4.41 (m, NH*CH*$_2$Triaz and Ar*CH*$_2$NH, 20 H), 3.68 (s, OCH_3, 3 H), 2.04–2.03 (br s, CH, 1 H), 1.92–1.73 (m, CCH_2, 24 H), 1.24–1.14 (m, 252 H), 0.88–0.84 (m, CH_3, 72 H); accurate mass (MALDI) calcd for $[C_{241}H_{417}N_{25}O_{12} + Na]^+$: 3879.2745; found: 3879.2670.

OAT-H-12: The compound was recovered from a xerogel sample of **OAT-CO$_2$H-10** after it was subjected to annealing at 200 ºC for 5 min in the DSC instrument. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.35 (br s, NH, 1 H), 7.90–7.82 (m, NH, 5 H), 7.44–7.39 (m, NH and Triaz*H*, 11 H), 7.28–7.16 (m, ArH, 24 H), 5.74 (t, NH, *J* = 6.0 Hz, 1 H), 5.44–5.43 (m, Triaz*CH*$_2$Ar, 12 H), 4.51–4.41 (m, NH*CH*$_2$Triaz and Ar*CH*$_2$NH, 24 H), 3.68 (s, OCH_3, 3 H), 2.04 (br s, CH, 1 H), 1.87–1.73 (m, CCH_2, 28 H), 1.24–1.14 (m, 294 H), 0.88–0.84 (m, CH_3, 84 H).

(c) Synthesis of **OATe-CO$_2$H-2n** and **OATe-COPrg-(2n+1)**

OATe-CO$_2$H-2: Sodium ascorbate (0.42 g, 2.00 mmol) and $CuSO_4 \cdot 5H_2O$ (0.2 g, 0.84 mmol) were added to a solution of compounds **16** (2.76 g, 5.00 mmol) and **13** (2.80 g, 4.20 mmol) in THF/H_2O (*v*/*v* = 9/1, 30 mL). The mixture was stirred at 25 °C for 12 h.

Aqueous HCl (20 mL, 0.2 M) was poured into the solution and the reaction mixture was extracted with DCM (3 × 20 mL). The combined organic layers were washed with H_2O (2 × 20 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: hexane/EtOAc/HOAc = 200/40/1) afforded **OATe-CO$_2$H-2** as a colorless oil (4.19 g, 82%). R_f = 0.21; ^{1}H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 8.76 (br s, NH, 1 H), 8.00 (br s, NH, 1 H), 7.15 (s, Triaz*H*, 1 H), 7.09 (d, *J* = 7.9 Hz, ArH, 2 H), 6.89 (d, ArH, *J* = 7.9 Hz, 2 H), 4.55 (t, Triaz*CH*$_2$CH$_2$, *J* = 6.0 Hz, 2 H), 4.46 (d, NH*CH*$_2$Triaz, *J* = 5.6 Hz, 2 H), 3.68 (s, OCH_3, 3 H), 3.65–3.60 (m, CH_2*CH*$_2$NH, 2 H), 3.09 (t, $ArCH_2$, *J* = 6.0 Hz, 2 H), 2.78 (t, $ArCH_2$, *J* = 6.0 Hz, 2 H), 1.84–1.68 (m, 8 H), 1.21–1.17 (m, 84 H), 0.87–0.83 (m, CH_2*CH*$_3$, 24 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 177.2, 175.4, 173.0, 172.5, 143.7, 137.9, 135.4, 129.3, 129.1, 123.6, 58.0, 57.2, 52.4, 52.0, 40.1, 39.0, 37.5, 37.33, 37.29, 37.0, 36.9, 36.7, 35.3, 35.0, 34.0, 33.9, 33.70, 33.67, 33.6, 33.5, 33.3, 33.2, 33.1, 33.0, 32.0, 29.94, 29.89, 29.88, 28.88, 28.85, 28.8, 26.71, 26.67, 26.65, 26.56, 23.25, 23.2, 22.9, 22.8, 22.31, 22.26, 14.3, 14.2 ppm; accurate mass (ESI) calcd for $[C_{76}H_{137}N_5O_6 + Na]^+$: 1239.0461; found: 1239.0446.

OATe-COPrg-3: COMU (0.45 g, 1.05 mmol) and DIPEA (0.31 mL, 1.74 mmol) were added in succession to a stirred solution of **OATe-CO$_2$H-2** (1.06 g, 0.87 mmol) in DCM (15 mL) at 0 °C. After 10 min, propargylamine (0.17 mL, 2.61 mmol) was added to the mixture. The reaction solution was allowed to stir at 25 °C for 12 h. H_2O (10 mL) was poured into the solution and the reaction mixture was extracted with DCM (3 × 20 mL). The combined organic layers were washed with saturated $NaHCO_3$ (2 × 10 mL), saturated $NaHSO_4$ (2 × 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: hexane/EtOAc = 3/1) afforded **OATe-COPrg-3** as a pale yellow oil (0.77 g, 73%). R_f = 0.33; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.35 (t, NH, *J* = 5.5 Hz, 1 H), 7.75 (t, NH, *J* = 5.3 Hz, 1 H), 7.33 (s, Triaz*H*, 1 H), 7.19 (t, NH, *J* = 5.8 Hz, 1 H), 7.09 (d, ArH, *J* = 6.4 Hz, 2 H), 7.01 (d, ArH, *J* = 6.4 Hz, 2 H), 4.48–4.45 (m, Triaz*CH*$_2$CH$_2$ and NH*CH*$_2$Triaz, 4 H), 3.99 (dd, CH≡C*CH*$_2$, *J* = 5.5 and 2.5 Hz, 2 H), 3.67 (s, OCH_3, 3 H), 3.47 (q, CH_2*CH*$_2$NH, *J* ~ 6.5 Hz, 2 H),

3.11 (t, $ArCH_2$, J = 7.5 Hz, 2 H), 2.76 (t, $ArCH_2$, J = 7.3 Hz, 2 H), 2.14 (t, C≡CH, J = 2.5 Hz, 1 H), 1.93–1.87 (m, CCH_2, 2 H), 1.79–1.67 (m, CCH_2, 6 H), 1.19–1.13 (m, 84 H), 0.84–0.83 (m, CH_2CH_3, 24 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 175.6, 173.33, 173.25, 171.5, 144.7, 137.8, 135.1, 129.2, 128.9, 122.4, 79.6, 71.2, 58.0, 56.9, 52.3, 51.6, 41.0, 38.2, 37.3, 37.2, 37.0, 36.9, 36.5, 35.4, 35.2, 34.0, 33.71, 33.70, 33.55, 33.46, 33.2, 33.15, 33.08, 31.96, 29.84, 29.81, 29.2, 28.84, 28.81, 28.76, 26.62, 26.61, 26.57, 26.5, 23.1, 22.7, 22.29, 22.26, 22.1, 14.2, 14.1 ppm; accurate mass (ESI) calcd for $[C_{79}H_{140}N_6O_5 + Na]^+$: 1276.0777; found: 1276.0753.

OATe-CO_2H-4: Sodium ascorbate (0.06 g, 0.30 mmol) and $CuSO_4 \cdot 5H_2O$ (0.03 g, 0.12 mmol) were added to a solution of **OATe-COPrg-3** (0.72 g, 0.58 mmol) and compound **16** (0.39 g, 0.57 mmol) in THF/H_2O (v/v = 9/1, 20 mL). The mixture was stirred at 25 °C for 12 h. Aqueous HCl (10 mL, 0.2 M) was poured into the solution and the reaction mixture was extracted with DCM (3 × 20 mL). The combined organic layers were washed with H_2O (2 × 15 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: hexane/EtOAc/HOAc = 300/200/1) afforded **OATe-CO_2H-4** as a colorless liquid (1.30 g, 85%). R_f = 0.24; ^1H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 8.58 (br s, NH, 1 H), 8.40 (br s, NH, 1 H), 7.60 (br s, NH, 1 H), 7.39 (s, Triaz*H*, 1 H), 7.18 (s, Triaz*H*, 1 H), 7.12–7.04 (m, ArH, 6 H), 6.89 (d, ArH, J = 7.8 Hz, 2 H), 6.70 (br s, NH, 1 H), 4.57–4.46 (m, Triaz$CH_2$$CH_2$ and NHCH_2Triaz, 8 H), 3.70 (s, OCH_3, 3 H), 3.63 (q, CH_2CH_2NH, J ~ 6.0 Hz, 2 H), 3.47 (q, CH_2CH_2NH, J ~ 6.8 Hz, 2 H), 3.17–3.10 (m, $ArCH_2$, 4 H), 2.80–2.73 (m, $ArCH_2$, 4 H), 1.98–1.56 (m, 12 H), 1.22–1.18 (m, 126 H), 0.88–0.85 (m, CH_2CH_3, 36 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.9, 175.7, 173.9, 173.4, 173.2, 171.6, 144.9, 143.9, 137.9, 137.7, 135.4, 135.2, 129.3, 129.2, 129.1, 129.0, 123.0, 122.4, 58.1, 57.3, 57.0, 52.4, 52.0, 51.7, 41.1, 40.3, 38.9, 38.0, 37.4, 37.34, 37.30, 37.25, 37.2, 37.10, 37.07, 37.05, 36.69, 36.6, 35.5, 35.3, 35.0, 34.1, 34.0, 33.81, 33.80, 33.73, 33.69, 33.65, 33.62, 33.56, 33.3, 33.25, 33.17, 32.07, 32.06, 30.0, 29.9, 28.92, 28.86, 26.74, 26.70, 26.68, 26.66, 26.6, 23.3, 23.2, 22.8, 22.7, 22.41, 22.38, 22.2, 22.1, 14.30, 14.29, 14.2 ppm; accurate mass (ESI) calcd for $[C_{120}H_{212}N_{10}O_8 + Na]^+$:

1945.6414; found: 1945.6435.

OATe-COPrg-5: COMU (0.35 g, 0.82 mmol) and DIPEA (0.24 mL, 1.36 mmol) were added in succession to a stirred solution of **OATe-CO$_2$H-4** (1.30 g, 0.68 mmol) in DCM (30 mL) at 0 °C. After 10 min, propargylamine (0.22 mL, 3.40 mmol) was added to the mixture. The reaction solution was allowed to stir at 25 °C for 24 h. H_2O (10 mL) was poured into the solution and the reaction mixture was extracted with DCM (3 × 20 mL). The combined organic layers were washed with saturated $NaHCO_3$ (2 × 10 mL), saturated $NaHSO_4$ (2 × 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: hexane/EtOAc = 2/1) afforded **OATe-COPrg-5** as a colorless liquid (0.88 g, 66%). R_f = 0.25; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.38 (br s, NH, 1 H), 7.75 (br s, NH, 1 H), 7.56 (br s, NH, 1 H), 7.39 (s, Triaz*H*, 1 H), 7.35 (s, Triaz*H*, 1 H), 7.19 (br s, NH, 1 H), 7.13–7.04 (m, NH and ArH, 9 H), 4.52–4.48 (m, Triaz*CH$_2$*CH$_2$ and NH*CH$_2$*Triaz, 8 H), 4.03 (dd, CH≡C*CH$_2$*, *J* = 5.0 and 2.4 Hz, 2 H), 3.70 (s, OCH_3, 3 H), 3.50–3.47 (m, CH_2*CH$_2$*NH, 4 H), 3.17–3.12 (m, $ArCH_2$, 4 H), 2.80–2.75 (m, $ArCH_2$, 4 H), 2.19 (t, C≡CH, *J* = 2.3 Hz, 1 H), 1.96–1.73 (m, CCH_2, 12 H), 1.21–1.15 (m, 126 H), 0.87–0.83 (m, CH_2*CH$_3$*, 36 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 175.7, 173.7, 173.3, 173.2, 173.1, 171.6, 144.9, 144.5, 137.91, 137.87, 135.2, 129.3, 129.00, 128.98, 122.4, 122.0, 79.6, 71.5, 58.1, 57.14, 57.09, 52.4, 51.69, 51.67, 41.1, 38.3, 37.9, 37.5, 37.3, 37.09, 37.07, 36.59, 36.56, 35.6, 35.4, 34.10, 34.06, 33.83, 33.82, 33.7, 33.6, 33.30, 33.27, 33.2, 32.1, 30.0, 29.9, 29.3, 29.0, 28.94, 28.91, 28.88, 26.74, 26.69, 26.6, 23.3, 22.8, 22.44, 22.40, 22.2, 22.1, 14.30, 14.25 ppm; accurate mass (ESI) calcd for $[C_{123}H_{215}N_{11}O_7 + Na]^+$: 1982.6730; found: 1982.6731.

OATe-CO$_2$H-6: Sodium ascorbate (0.05 g, 0.25 mmol) and $CuSO_4 \cdot 5H_2O$ (0.03 g, 0.12 mmol) were added to a solution of **OATe-COPrg-5** (0.88 g, 0.45 mmol) and **16** (0.30 g, 0.45 mmol) in THF/H_2O (*v*/*v* = 9/1, 30 mL). The mixture was stirred at 25 °C for 24 h. Aqueous HCl (20 mL, 0.2 M) was poured into the solution and the reaction mixture was extracted with DCM (3 × 30 mL). The combined organic layers were washed with H_2O (2 × 20 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*.

Chromatography of the residue over silica gel (eluent: $CHCl_3$/MeOH = 20/1) afforded **OATe-CO_2H-6** as a white solid (0.91 g, 77%). R_f = 0.35; M.p. 72–77 °C; ^{1}H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 8.47 (br s, NH, 1 H), 8.39 (br s, NH, 1 H), 7.84 (br s, NH, 1 H), 7.67 (br s, NH, 1 H), 7.39 (s, Triaz*H*, 1 H), 7.38 (s, Triaz*H*, 1 H), 7.21 (br s, NH, 1 H), 7.19 (s, Triaz*H*, 1 H), 7.12–7.04 (m, ArH, 10 H), 6.90 (d, ArH, *J* = 7.8 Hz, 2 H), 6.80 (br s, NH, 1 H), 4.56–4.44 (m, Triaz$CH_2$$CH_2$ and NHCH_2Triaz, 12 H), 3.70 (s, OCH_3, 3 H), 3.61–3.60 (m, CH_2CH_2NH, 2 H), 3.48–3.45 (m, CH_2CH_2NH, 4 H), 3.16–3.08 (m, $ArCH_2$, 6 H), 2.78–2.72 (m, $ArCH_2$, 6 H), 1.96–1.61 (m, 16 H), 1.21–1.14 (m, 168 H), 0.86–0.84 (m, CH_2CH_3, 48 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 175.7, 173.9, 173.7, 173.3, 173.2, 171.6, 137.9, 137.8, 135.4, 135.1, 129.33, 129.26, 129.1, 129.0, 58.1, 57.14, 57.06, 51.7, 41.1, 38.8, 37.9, 37.4, 37.34, 37.26, 37.1, 36.7, 36.6, 35.6, 35.3, 34.1, 33.8, 33.7, 33.6, 33.3, 33.2, 32.1, 29.9, 28.93, 28.88, 26.74, 26.68, 26.6, 23.2, 22.8, 22.4, 22.2, 14.3, 14.2 ppm; accurate mass (ESI) calcd for $[C_{164}H_{287}N_{15}O_{10} + 2Na]^{2+}$: 1326.1204; found: 1236.1201.

OATe-COPrg-7: COMU (0.18 g, 0.42 mmol) and DIPEA (0.12 mL, 0.84 mmol) were added in succession to a stirred solution of **OATe-CO_2H-6** (0.88 g, 0.34 mmol) in DCM (30 mL) at 0 °C. After 10 min, propargylamine (0.20 mL, 3.28 mmol) was added to the mixture. The reaction solution was allowed to stir at 25 °C for 24 h. H_2O (5 mL) was poured into the solution and the reaction mixture was extracted with DCM (3 × 30 mL). The combined organic layers were washed with saturated $NaHCO_3$ (2 × 20 mL), saturated $NaHSO_4$ (2 × 20 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: $CHCl_3$/MeOH = 25/1) afforded **OATe-COPrg-7** as a white solid (0.83 g, 92%). R_f = 0.34; M.p. 95–100 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.38 (br s, NH, 1 H), 7.78 (br s, NH, 1 H), 7.70 (br s, NH, 1 H), 7.53 (br s, NH, 1 H), 7.39 (s, Triaz*H*, 1 H), 7.38 (s, Triaz*H*, 1 H), 7.36 (s, Triaz*H*, 1 H), 7.20 (br s, NH, 2 H), 7.14–7.05 (2 m, NH and ArH, 13 H), 4.52–4.48 (m, Triaz$CH_2$$CH_2$ and NHCH_2Triaz, 12 H), 4.03 (br s, CH≡CCH_2, 2 H), 3.70 (s, OCH_3, 3 H), 3.51–3.47 (m, CH_2CH_2NH, 6 H), 3.20–3.10 (m, $ArCH_2$, 6 H), 2.79–2.76 (m, $ArCH_2$, 6 H), 2.19 (br s, C≡CH, 1 H), 1.96–1.74 (m, CCH_2, 16 H), 1.25–1.15 (m, 168 H), 0.88–0.83 (m,

CH_2CH_3, 48 H); ^{13}C NMR (176 MHz, $CDCl_3$): δ = 175.8, 173.7, 173.31, 173.26, 171.6, 144.9, 137.9, 135.13, 135.09, 129.3, 129.0, 122.5, 122.1, 79.5, 71.5, 58.1, 57.11, 57.06, 52.4, 51.7, 41.1, 38.3, 38.0, 37.5, 37.3, 37.1, 37.0, 36.6, 35.6, 35.3, 34.1, 34.0, 33.8, 33.6, 33.5, 33.3, 33.24, 33.21, 33.16, 32.1, 30.3, 30.0, 29.95, 29.86, 29.5, 29.3, 28.93, 28.89, 28.87, 26.73, 26.68, 26.6, 23.3, 22.8, 22.43, 22.40, 22.2, 22.13, 14.35, 14.3 ppm; accurate mass (MALDI) calcd for $[C_{167}H_{290}N_{16}O_9 + Na]^+$: 2688.2651; found: 2688.2665.

OATe-CO_2H-8: Sodium ascorbate (12 mg, 0.061 mmol) and $CuSO_4 \cdot 5H_2O$ (4.00 mg, 0.016 mmol) were added to a solution of **OATe-COPrg-5** (0.14 g, 0.072 mmol) and **17** (0.10 g, 0.073 mmol) in THF/H_2O (*v*/*v* = 9/1, 10 mL). The solution was stirred at 50 °C for 12 h. After cooling down to room temperature, aqueous HCl (5 mL, 0.2 M) was poured into the solution and the reaction mixture was extracted with DCM (3 × 10 mL). The combined organic layers were washed with H_2O (2 × 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: $CHCl_3$/MeOH = 20/1) afforded **OATe-CO_2H-8** as a white solid (0.21 g, 88%). R_f = 0.34; M.p. 105–111 °C; ^1H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 8.49 (br s, NH, 1 H), 8.39 (br s, NH, 1 H), 7.85–7.75 (m, NH, 2 H), 7.59 (br s, NH, 1 H), 7.39 (s, Triaz*H*, 1 H), 7.38 (s, Triaz*H*, 2 H), 7.19 (br s, NH and Triaz*H*, 2 H), 7.14–7.05 (m, NH and ArH, 15 H), 6.89 (d, ArH, *J* = 7.8 Hz, 2 H), 6.75 (br s, NH, 1 H), 4.55–4.45 (m, Triaz$CH_2$$CH_2$ and NHCH_2Triaz, 16 H), 3.70 (s, OCH_3, 3 H), 3.63–3.61 (m, CH_2CH_2NH, 2 H), 3.49–3.45 (m, CH_2CH_2NH, 6 H), 3.17–3.10 (m, $ArCH_2$, 8 H), 2.79–2.77 (m, $ArCH_2$, 8 H), 1.96–1.65 (m, 20 H), 1.25–1.15 (m, 210 H), 0.86–0.83 (m, CH_2CH_3, 60 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 177.0, 175.6, 173.9, 173.8, 173.2, 173.11, 173.07, 171.5, 144.8, 144.5, 144.0, 137.89, 137.87, 137.8, 135.2, 135.1, 135.0, 129.3, 129.2, 129.0, 128.9, 122.8, 122.4, 122.0, 58.1, 57.3, 57.1, 57.0, 52.3, 51.9, 51.6, 41.0, 40.4, 38.6, 37.8, 37.7, 37.3, 37.2, 37.00, 36.99, 36.6, 36.50, 36.47, 35.53, 35.50, 35.3, 35.22, 35.17, 35.0, 34.03, 33.96, 33.76, 33.75, 33.7, 33.64, 33.58, 33.5, 33.24, 33.19, 33.17, 33.1, 32.0, 29.9, 29.8, 28.9, 28.83, 28.81, 26.7, 26.63, 26.61, 26.6, 25.7, 23.2, 22.8, 22.7, 22.32, 22.29, 22.1, 14.23, 14.17 ppm; accurate mass (ESI) calcd for

$[C_{208}H_{362}N_{20}O_{12} + 2Na]^{2+}$: 1690.4090; found: 1690.4091.

OATe-COPrg-9: COMU (0.04 g, 0.23 mmol) and DIPEA (0.03 mL, 0.38 mmol) were added in succession to a stirred solution of **OATe-CO$_2$H-8** (0.20 g, 0.06 mmol) in DCM (10 mL) at 0 °C. After 10 min, propargylamine (0.08 mL, 1.21 mmol) was added to the mixture. The reaction solution was allowed to stir at 25 °C for 48 h. H_2O (3 mL) was poured into the solution and the reaction mixture was extracted with DCM (3 × 10 mL). The combined organic layers were washed with saturated $NaHCO_3$ (2 × 10 mL), saturated $NaHSO_4$ (2 × 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: $CHCl_3$/MeOH = 25/1) afforded **OATe-COPrg-9** as a white solid (0.15 g, 74%). R_f = 0.28; M.p. 119–126 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.38 (br s, NH, 1 H), 7.80 (br s, NH, 1 H), 7.76 (br s, NH, 1 H), 7.72 (br s, NH, 1 H), 7.55 (br s, NH, 1 H), 7.39 (s, Triaz*H*, 1 H), 7.37 (s, Triaz*H*, 2 H), 7.35 (s, Triaz*H*, 1 H), 7.23–7.22 (m, NH, 3 H), 7.13–7.04 (m, NH and ArH, 17 H), 4.52–4.48 (m, Triaz*CH$_2$*CH$_2$ and NH*CH$_2$*Triaz, 16 H), 4.02 (dd, CH≡C*CH$_2$*, *J* = 5.0 and 2.4 Hz, 2 H), 3.70 (s, OCH_3, 3 H), 3.50–3.47 (m, CH_2*CH$_2$*NH, 8 H), 3.16–3.13 (m, $ArCH_2$, 8 H), 2.78–2.75 (m, $ArCH_2$, 8 H), 2.18 (t, C≡CH, *J* = 2.5 Hz, 1 H), 1.96–1.73 (m, CCH_2, 20 H), 1.21–1.14 (m, 210 H), 0.87–0.82 (m, CH_2*CH$_3$*, 60 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 175.7, 173.74, 173.72, 173.3, 173.2, 173.16, 173.10, 173.07, 171.6, 144.9, 144.54, 144.51, 144.46, 137.94, 137.90, 137.88, 135.14, 135.09, 129.4, 129.3, 129.0, 122.4, 122.0, 79.6, 71.4, 58.1, 57.13, 57.08, 52.4, 51.7, 41.1, 38.2, 37.95, 37.88, 37.8, 37.5, 37.4, 37.3, 37.07, 37.05, 36.6, 35.60, 35.58, 35.3, 34.1, 34.0, 33.81, 33.80, 33.7, 33.6, 33.28, 33.26, 33.23, 33.18, 32.1, 29.95, 29.92, 29.8, 29.3, 28.93, 28.90, 28.87, 26.72, 26.68, 26.6, 23.3, 22.8, 22.42, 22.39, 22.2, 22.1, 14.31, 14.25 ppm; accurate mass (MALDI) calcd for $[C_{211}H_{365}N_{21}O_{11} + Na]^+$: 3394.8604; found: 3394.8627.

OATe-CO$_2$H-10: Sodium ascorbate (0.02 g, 0.10 mmol) and $CuSO_4 \cdot 5H_2O$ (0.01 g, 0.04 mmol) were added to a solution of **OATe-COPrg-7** (0.30 g, 0.12 mmol) and **17** (0.17 g, 0.12 mmol) in THF/H_2O (*v*/*v* = 9/1, 15 mL). The solution was stirred at 50 °C for 48 h. Aqueous HCl (10 mL, 0.2 M) was poured into the solution and the reaction

mixture was extracted with DCM (3 × 15 mL). The combined organic layers were washed with H_2O (2 × 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chromatography of the residue over silica gel (eluent: $CHCl_3$/MeOH = 20/1) afforded **OATe-CO_2H-10** as a white solid (0.42 g, 87%). R_f = 0.22; M.p. 127–136 °C; ^{1}H NMR (400 MHz, $CDCl_3$, CO_2H signal too broad to be observed): δ = 8.49 (br s, NH, 1 H), 8.39 (br s, NH, 1 H), 7.80–7.77 (m, NH, 3 H), 7.61 (br s, NH, 1 H), 7.39 (s, Triaz*H*, 1 H), 7.38 (s, Triaz*H*, 3 H), 7.22–7.18 (m, NH and Triaz*H*, 4 H), 7.14–7.05 (m, ArH, 18 H), 6.89 (d, ArH, *J* = 7.8 Hz, 2 H), 6.75 (br s, NH, 1 H), 4.56–4.45 (m, Triaz$CH_2$$CH_2$ and NHCH_2Triaz, 20 H), 3.70 (s, OCH_3, 3 H), 3.63–3.61 (m, CH_2CH_2NH, 2 H), 3.50–3.45 (m, CH_2CH_2NH, 8 H), 3.17–3.08 (m, $ArCH_2$, 10 H), 2.79–2.73 (m, $ArCH_2$, 10 H), 1.96–1.67 (m, 24 H), 1.25–1.15 (m, 252 H), 0.86–0.83 (m, CH_2CH_3, 72 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 175.8, 173.9, 173.7, 173.3, 173.2, 173.1, 171.6, 144.9, 144.6, 144.5, 143.9, 137.94, 137.92, 137.8, 135.4, 135.2, 135.1, 129.4, 129.29, 129.26, 129.1, 129.00, 128.97, 123.0, 122.4, 122.0, 58.1, 57.3, 57.1, 57.0, 52.4, 52.0, 51.7, 41.1, 40.3, 38.8, 38.0, 37.9, 37.5, 37.4, 37.32, 37.26, 37.09, 37.06, 36.7, 36.6, 35.6, 35.5, 35.3, 35.0, 34.1, 33.67, 33.66, 33.3, 33.2, 32.1, 30.0, 29.8, 28.94, 28.91, 28.89, 26.8, 26.74, 26.69, 26.6, 23.3, 22.8, 22.44, 22.40, 22.2, 14.32, 14.27 ppm; accurate mass (ESI) calcd for $[C_{252}H_{437}N_{25}O_{14} + 2Na]^{2+}$: 2043.2050; found: 2043.2098.

3. Gel photos

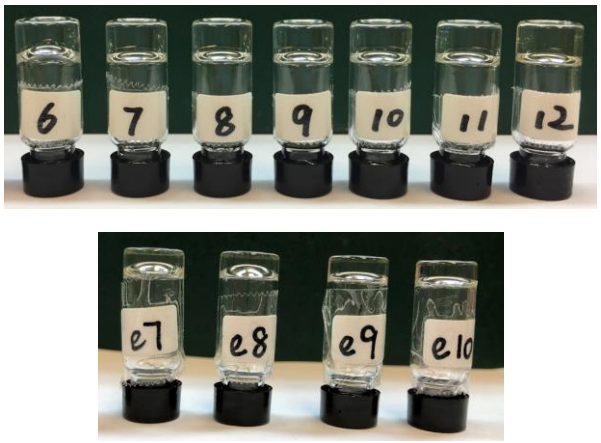


Figure S1. Photos of transparent *p*-xylene gels (2.5% *w*/*v*). (top) From left to right: **OAT-CO_2H-6, OAT-COPrg-7, OAT-CO_2H-8, OAT-COPrg-9, OAT-CO_2H-10, OAT-COPrg-11** and **OAT-CO_2H-12**. (bottom) From left to right: **OATe-COPrg-7, OATe-CO_2H-8, OATe-COPrg-9** and **OATe-CO_2H-10**.

4. Fourier transformed infrared spectroscopy

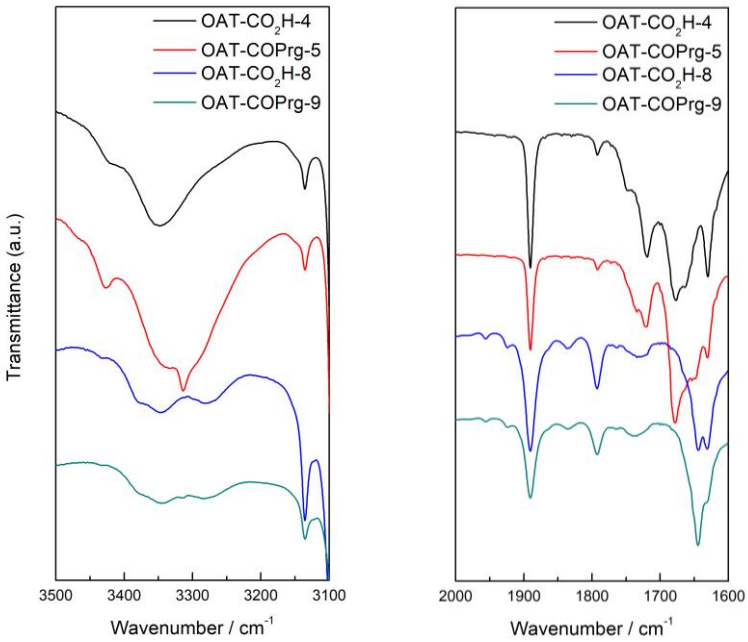


Figure S2. Stacked partial FTIR (2.5% *w*/*v* in *p*-xylene) spectra of oligomers (from top to bottom: **OAT-CO_2H-4, OAT-COPrg-5, OAT-CO_2H-8** and **OAT-COPrg-9**).

5. Small angle powdered X-ray diffraction

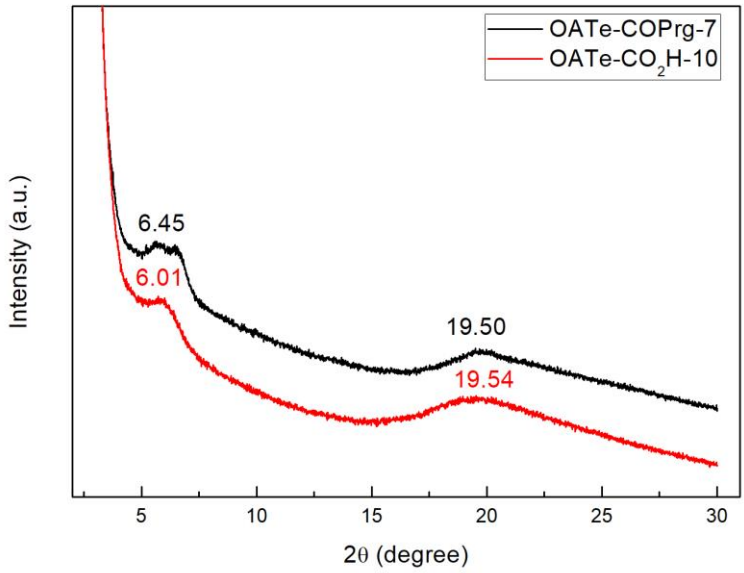


Figure S3. SAXRD patterns of 2.5% *w*/*v* xerogels from *p*-xylene of **OATe-COPrg-7** (black) and **OATe-CO₂H-10** (red). The positions of two peaks were very similar to those of the **OAT** series.

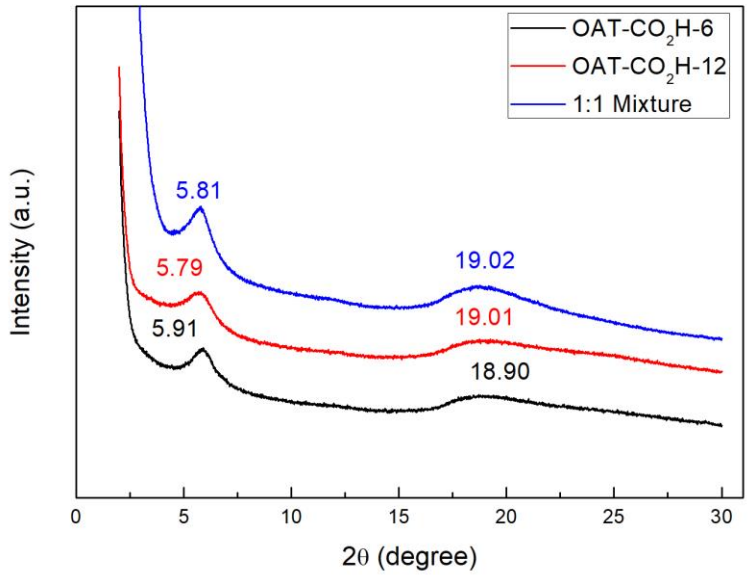


Figure S4. SAXRD patterns of 2.5% *w*/*v* xerogels from *p*-xylene of **OAT-CO₂H-6** (black), **OAT-CO₂H-12** (red) and their 1:1 mixture (blue).

6. Determination of dimerization constant - ^{1}H NMR dilution experiment

^{1}H NMR dilution experiment was carried out using **OAT-CO$_2$H-6** in $CDCl_3$. Two CONH signals exhibited significant shifting of chemical shift (Figure S4). The data were fitted into the dimerization model described by Hunter.[4] The fitting results were listed in Table S1.

$$\sigma_{obs} = \sigma_d - (\sigma_d - \sigma_m)\frac{-1 + \sqrt{1 + 8K_{dim}c_t}}{4K_{dim}c_t}$$

where

σ_{obs} is the observed chemical shift of a NMR signal,

σ_d is the chemical shift of dimer A_2,

σ_m is the chemical shift of monomer A,

c_t is the total concentration of A

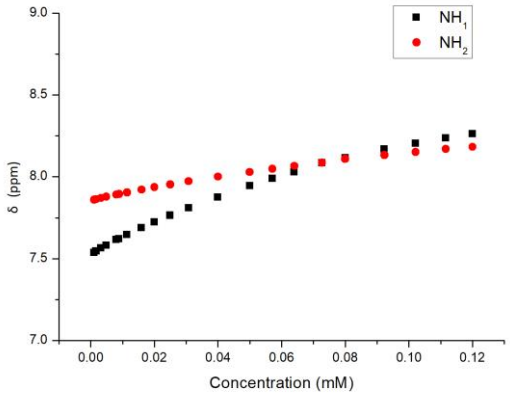


Figure S5. Concentration-dependent variation of chemical shifts of two CONHs of **OAT-CO$_2$H-6** (700 MHz, $CDCl_3$, 25 ºC).

Table S1. Summary of the parameters obtained from the best fit of concentration-dependent ^{1}H NMR chemical shift variation of two NH signals of **OAT-CO$_2$H-6**.

	NH_1	NH_2
r^2	0.9996	0.9998
K_{dim} (M^{-1})	2.1 ± 0.1	1.5 ± 0.05
δ_m (ppm)	7.53 ± 0.003	7.86 ± 0.001
δ_d (ppm)	10.28 ± 0.08	9.38 ± 0.04

[4] Hunter, C. A. et al. *J. Am. Chem. Soc.* **2000**, *122*, 8856–8868.

7. DSC thermograms

(a) Pure wet gels

DSC profile of the wet gels all showed only one endothermic peak upon heating indicated that the gel state was homogeneous involving mainly one single phase transition (Figures S5–S6).

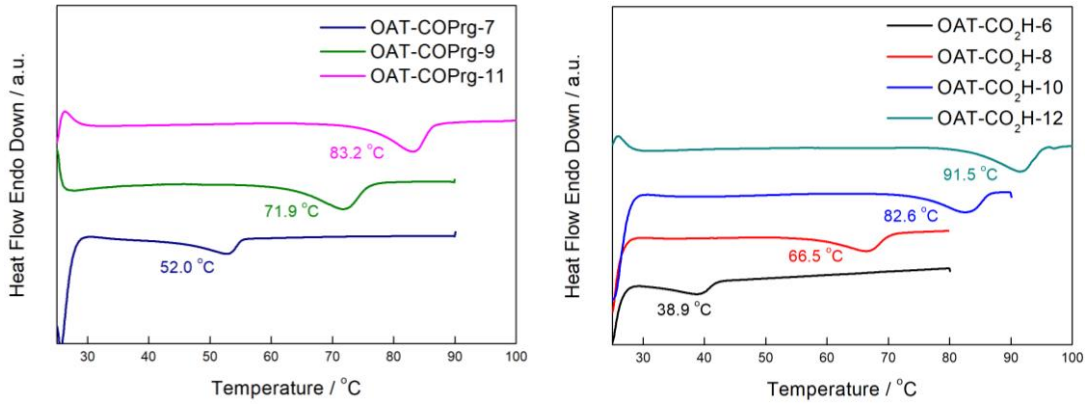


Figure S6. DSC thermograms of wet gels in *p*-xylene (2.5% *w*/*v*), left: **OAT-COPrg-(2n+1)** series; right: **OAT-CO₂H-2n** series. The value of endothermic peak represents the gel-to-sol temperature (T_{gs}).

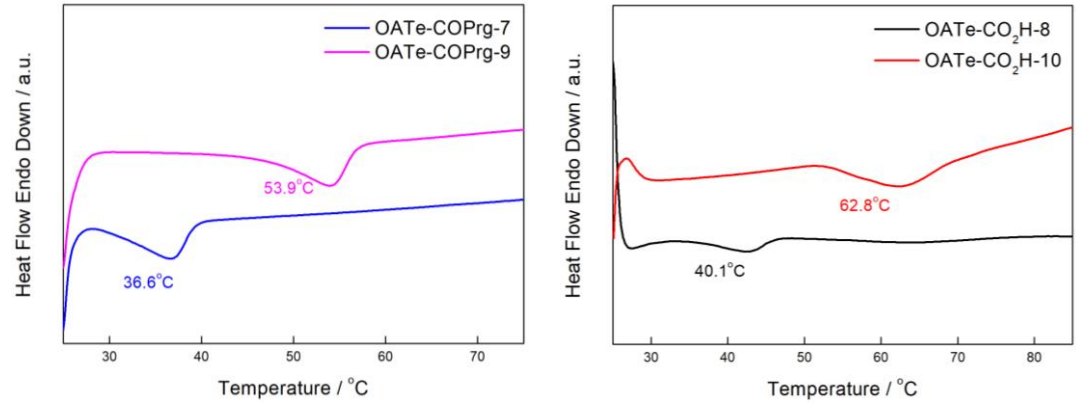


Figure S7. DSC thermograms of wet gels in *p*-xylene (2.5% *w*/*v*), left: **OATe-CO₂Prg-(2n+1)** series; right: **OATe-CO₂H-2n** series. The value of endothermic peak represents the gel-to-sol temperature (T_{gs}).

(b) Xerogels

(i) *N*-Propargyl amide terminated oligomers: A xerogel sample of **OAT-COPrg-(2n+1)** or **OATe-COPrg-(2n+1)** (2.5% in *p*-xylene) was subjected to first heating (rate = 10 °C/min) to give one solid-to-liquid melting peak (T_m) (Figures S7 and S8). After annealing at 200 °C for 5 min, cooling down to 40 °C (rate = –5 °C/min) and reheating (rate = 10 °C/min) again generated the second heating curve. For the **OAT-**

COPrg(2n+1) series, one melting peak was identified, but the ΔH_m and ΔS_m values decreased sharply as compared to those of the first cycle (Table S2). On the other hand, no peak could be found in the second heating profile for the **OATe-COPrg(2n+1)** series.

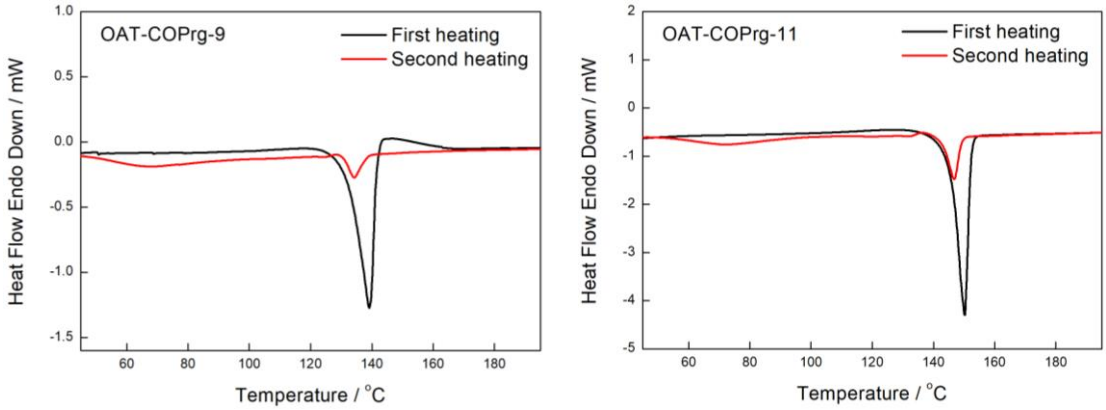


Figure S8. DSC thermograms of xerogels of **OAT-COPrg-(2n+1)**.

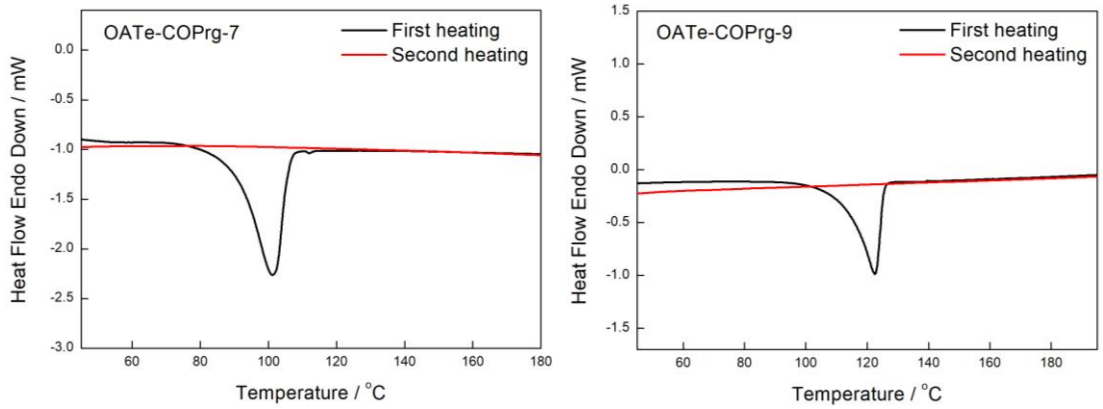
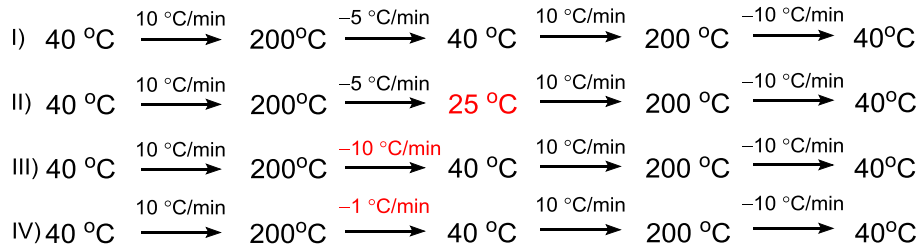


Figure S9. DSC thermograms of xerogels of **OATe-COPrg-(2n+1)**.

Table S2. T_m, ΔH_m and ΔS_s values of xerogels of **OAT-COPrg-(2n+1)** and **OATe-COPrg-(2n+1)** before and after annealing.

	First heating cycle			Second heating cycle		
Compound	T_m (ºC)	ΔH_m ($J \cdot g^{-1}$)	ΔS_m ($J \cdot g^{-1} \cdot K^{-1}$) × 10^3	T_m (ºC)	ΔH_m ($J \cdot g^{-1}$)	ΔS_m ($J \cdot g^{-1} \cdot K^{-1}$) × 10^3
OAT-COPrg-7	123	34.2	87.9	118	5.2	11.3
OAT-COPrg-9	139	31.6	74.1	134	3.0	7.3
OAT-COPrg-11	150	36.1	86.8	147	8.2	19.5
OATe-COPrg-7	101	33.9	89.5	–	–	–
OATe-COPrg-9	122	34.9	88.8	–	–	–

(ii) Effect of cooling profile on the DSC thermograms of **OAT-COPrg-(2n+1)**: DSC experiments on **OAT-COPrg-7** and **OAT-COPrg-9** following different cooling protocols were conducted (Scheme S1) and the corresponding ΔH_m values were listed (Table S3).



Scheme S1. DSC cooling protocols.

Table S3. ΔH_m value changes of **OAT-COPrg-7** and **OAT-COPrg-9** under different cooling protocols.[a]

	OAT-COPrg-7			**OAT-COPrg-9**		
Cooling protocol	ΔH_{m1} ($J \cdot g^{-1}$)	ΔH_{m2} ($J \cdot g^{-1}$)	$\Delta H_{m2}/\Delta H_{m1}$	ΔH_{m1} ($J \cdot g^{-1}$)	ΔH_{m2} ($J \cdot g^{-1}$)	$\Delta H_{m2}/\Delta H_{m1}$
I	34.3	5.0	14.6%	30.7	2.9	9.6%
II	28.2	3.2	11.2%	32.0	3.4	10.7%
III	33.0	2.9	8.8%	29.87	1.9	6.3%
IV	29.3	10.4	35.6%	39.64	12.9	32.5%

[a] ΔH_{m1} and ΔH_{m2} are the enthalpy of solid-to-liquid melting values of the first and second heating, respectively.

(iii) Carboxylic acid- and hydride-terminated oligomers: A xerogel sample of **OAT-CO_2H-2n** or **OATe-CO_2H-2n** (2.5% in *p*-xylene) was subjected to first heating (rate = 10 °C/min) to give one solid-to-liquid melting peak (T_m) (Figures S9 and S10). After annealing at 200 °C for 5 min, cooling down (rate = –5 °C/min) to 40 °C and reheating (rate = 10 °C/min) again generated the second heating curve. In the second scanning profile, a decarboxylation reaction occurred and the DSC profile was, accordingly, that of **OAT-H-2n** or **OATe-H-2n**, respectively. The T_m, ΔH_m and ΔS_m values were tabulated (Table S4).

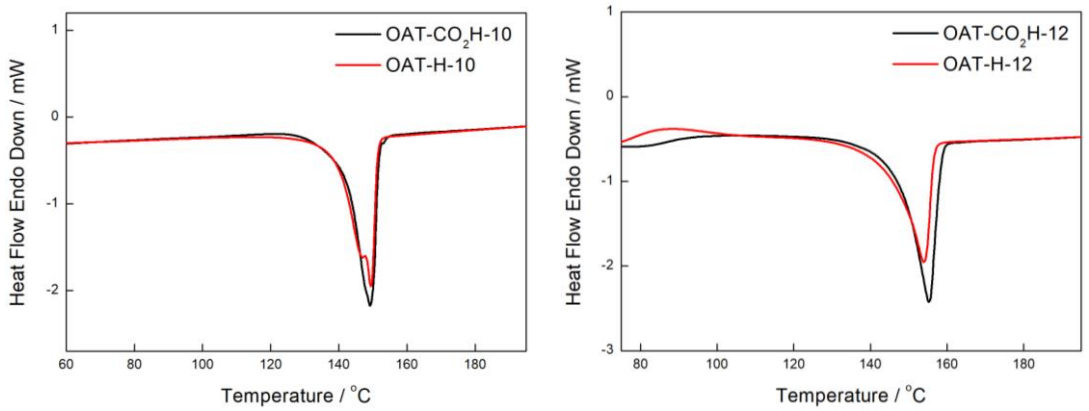


Figure S10. DSC thermograms of xerogels of **OAT-CO₂H-2n** and **OAT-H-2n** series.

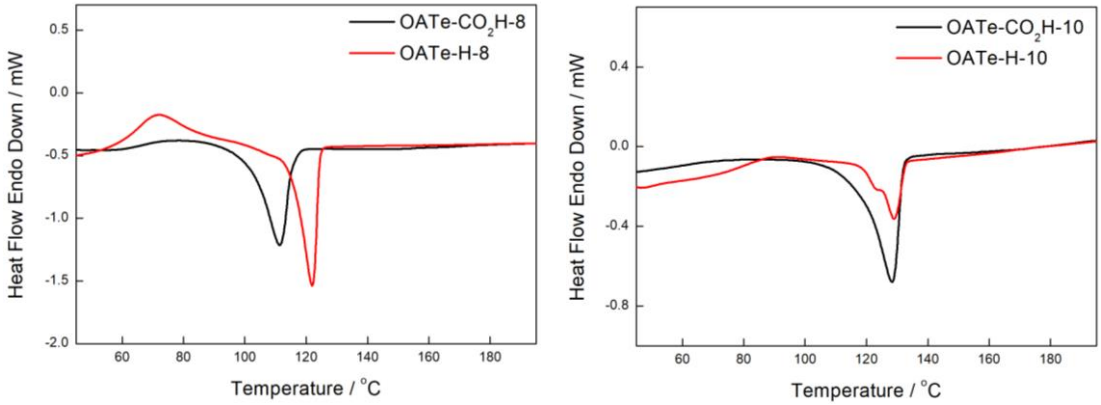


Figure S11. DSC thermograms of xerogels of **OATe-CO₂H-2n** and **OATe-H-2n** series.

Table S4. T_m, ΔH_m and ΔS_m values of xerogels of **OAT-CO₂H-2n**, **OATe-CO₂H-2n**, **OAT-H-2n** and **OATe-H-2n**.

Compound	T_m (ºC)	ΔH_m ($J \cdot g^{-1}$)	ΔS_m ($J \cdot g^{-1} \cdot K^{-1}$) $\times 10^3$	Compound	T_m (ºC)	ΔH_m ($J \cdot g^{-1}$)	ΔS_m ($J \cdot g^{-1} \cdot K^{-1}$) $\times 10^3$
OAT-CO₂H-6	100	21.0	55.5	**OAT-H-6**	117	20.3	48.5
OAT-CO₂H-8	132	25.0	65.6	**OAT-H-8**	137	24.8	57.8
OAT-CO₂H-10	149	29.9	73.6	**OAT-H-10**	149	27.2	63.0
OAT-CO₂H-12	155	30.7	74.6	**OAT-H-12**	154	24.8	60.4
OATe-CO₂H-8	111	21.3	57.5	**OATe-H-8**	122	20.9	57.8
OATe-CO₂H-10	128	18.9	48.0	**OATe-H-10**	129	7.5	20.5

(c) Self-sorting of mixed xerogels

(i) **OAT-CO$_2$H-6/OAT-CO$_2$H-10** and **OAT-H-6/OAT-H-10**

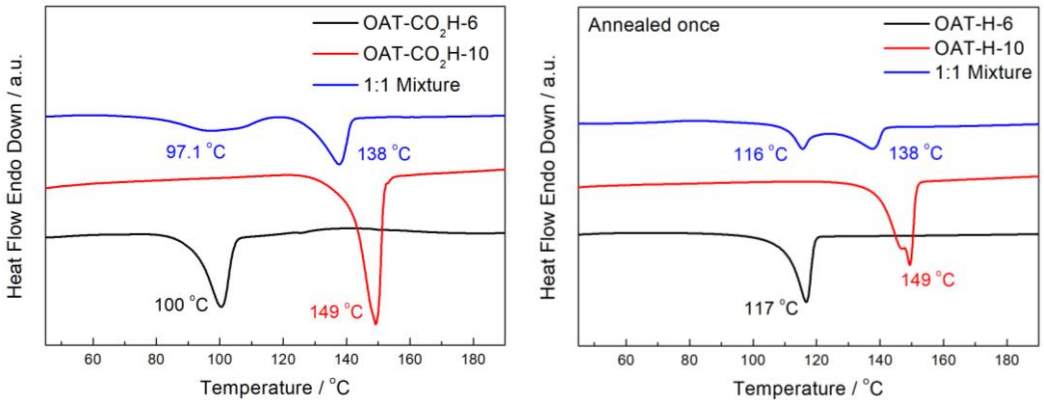


Figure S12. Left: DSC thermograms of xerogels (2.5 % *w*/*v* in *p*-xylene) of a 1:1 mixture of **OAT-CO$_2$H-6/OAT-CO$_2$H-10** (blue), pure **OAT-CO$_2$H-10** (red), and pure **OAT-CO$_2$H-6** (black); Right: DSC thermograms of xerogels of a 1:1 mixture of **OAT-H-6/OAT-H-10** (blue), pure **OAT-H-10** (red), and pure **OAT-H-6** (black).

(ii) **OAT-CO$_2$H-2m/OAT-COPrg-(2n+1)** and **OAT-H-2m/OAT-COPrg-(2n+1)**

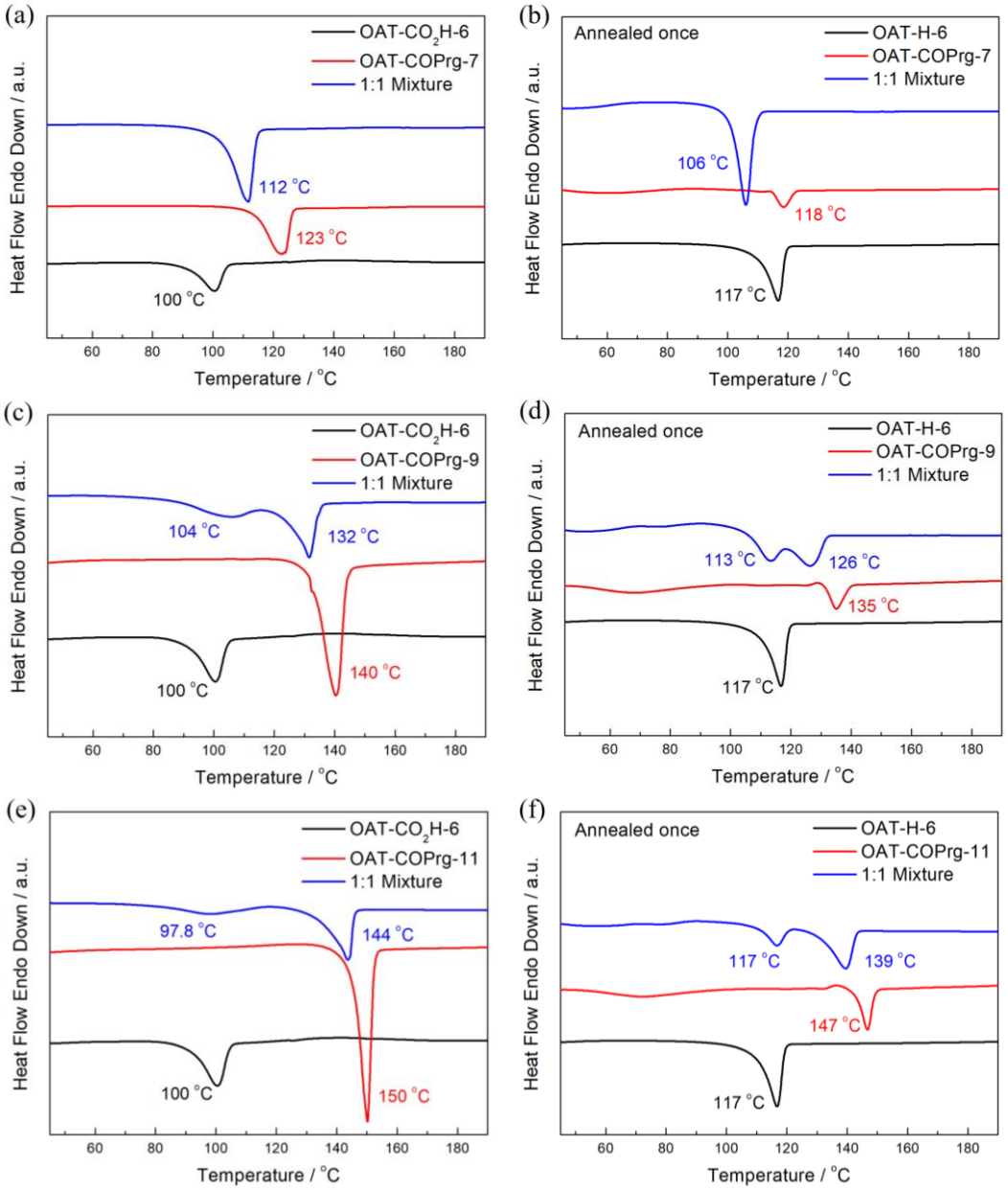


Figure S13. (a) DSC thermograms of xerogels (2.5 % *w*/*v* in *p*-xylene) of a 1:1 mixture of **OAT-CO$_2$H-6**/**OAT-COPrg-7** (blue), pure **OAT-COPrg-7** (red), and pure **OAT-CO$_2$H-6** (black); (b) DSC thermograms of xerogels of a 1:1 mixture of **OAT-H-6**/**OAT-COPrg-7** (blue), pure **OAT-COPrg-7** (red), and pure **OAT-H-6** (black); (c) DSC thermograms of xerogels (2.5 % *w*/*v* in *p*-xylene) of a 1:1 mixture of **OAT-CO$_2$H-6**/**OAT-COPrg-9** (blue), pure **OAT-COPrg-9** (red), and pure **OAT-CO$_2$H-6** (black); (d) DSC thermograms of xerogels of a 1:1 mixture of **OAT-H-6**/**OAT-COPrg-9** (blue), pure **OAT-COPrg-9** (red), and pure **OAT-H-6** (black); (e) DSC thermograms of xerogels (2.5 % w/v in *p*-xylene) of a 1:1 mixture of **OAT-CO$_2$H-6**/**OAT-COPrg-11** (blue), pure **OAT-COPrg-11** (red), and pure **OAT-CO$_2$H-6** (black); (f) DSC thermograms of xerogels of a 1:1 mixture of **OAT-H-6**/**OAT-COPrg-11** (blue), pure **OAT-COPrg-11** (red), and pure **OAT-H-6** (black).

(d) Semi-quantitative assessment of self-sorting: In the xerogel phase diagrams of these oligomers, it was noticed that the eutectic temperature was very close to the T_m of the lower oligomer, such that the terminal solid solution zone was too narrow to be represented on the phase diagram. In such a simplified case where the terminal solid solution zone is not observable, we could make the following rationalizations.

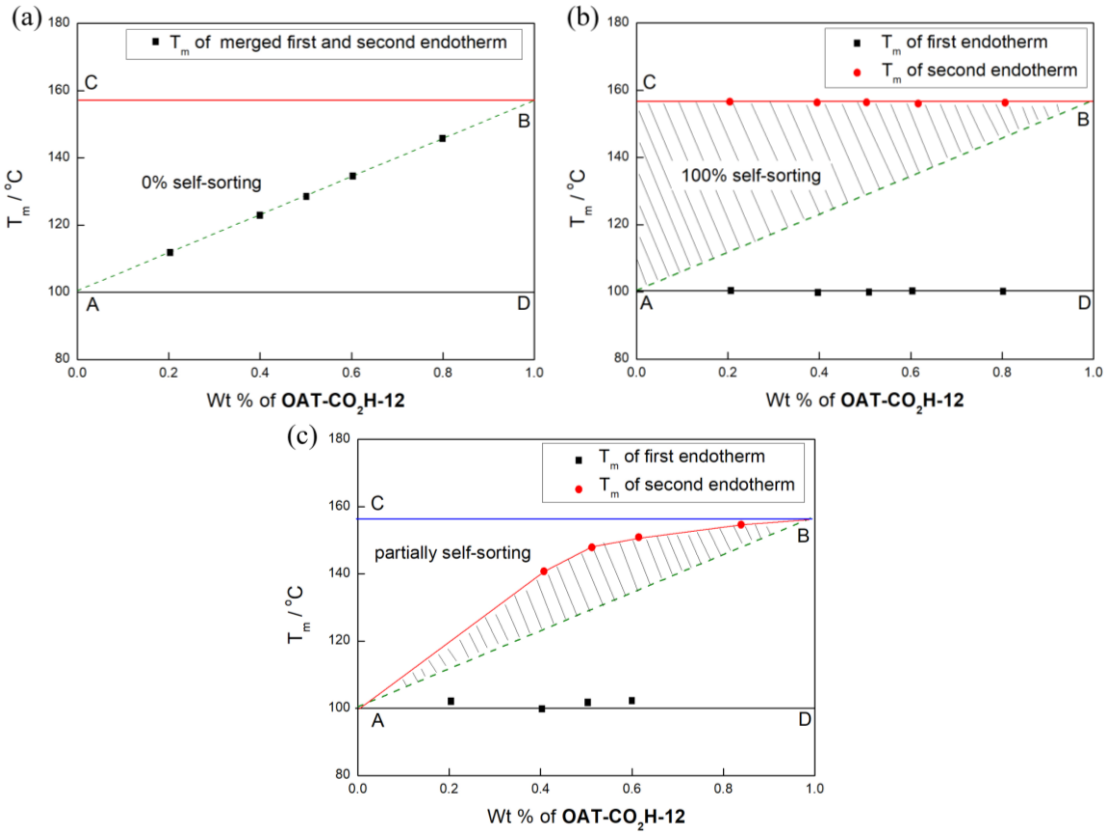


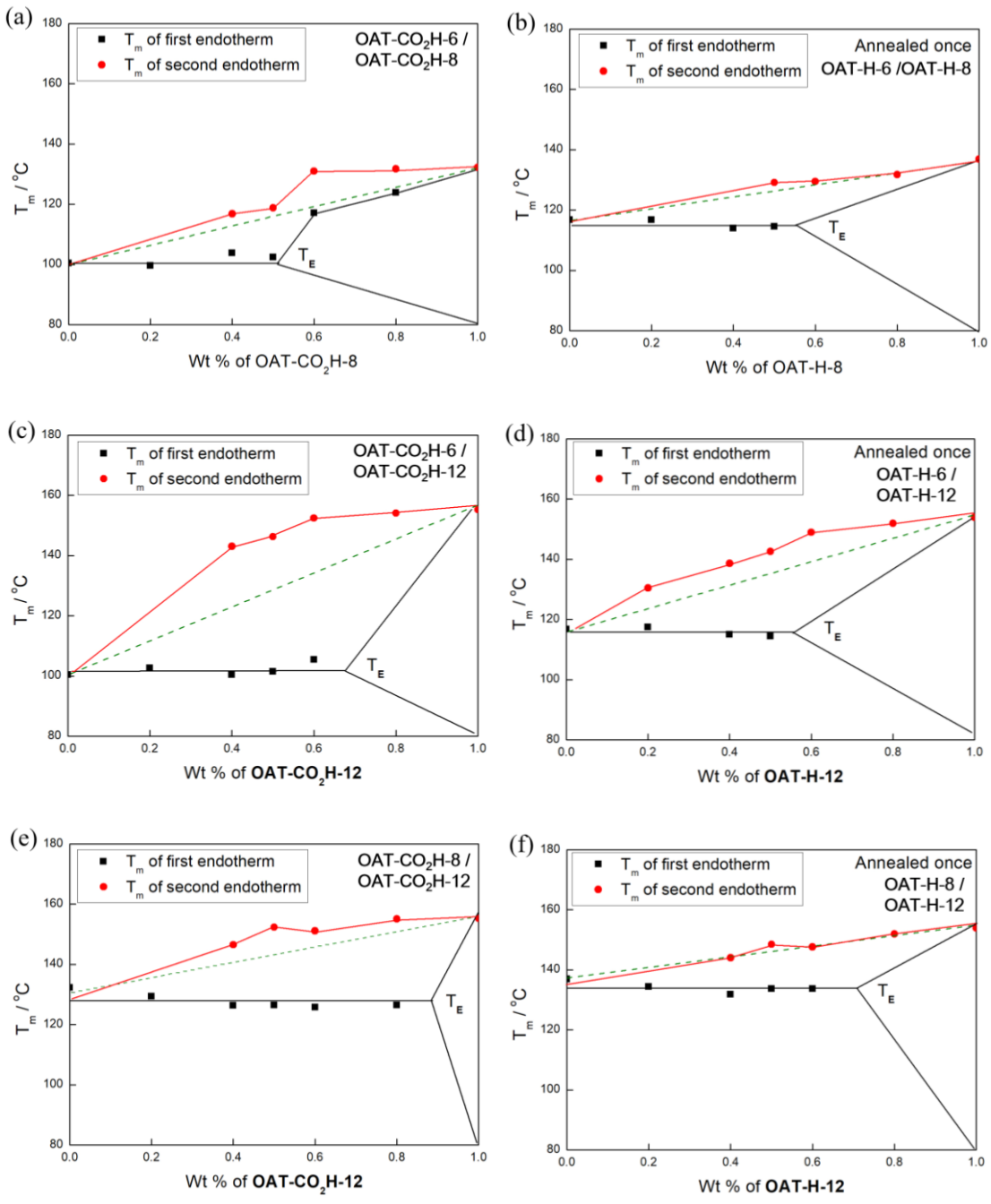
Figure S14. Hypothetical phase diagrams of (a) 0% self-sorted, (b) 100% self-sorted and (c) partially self-sorted systems.

(i) In a completely non-self-sorted system, the first and second endotherm peaks will merge together, and only one T_m, whose value will depend upon the molecular composition, will show up on the green dash line *AB* (Figure S13a).

(ii) In a hypothetical 100% self-sorted mixed gel system, two T_m points, each corresponds to the T_m of the two pure samples, should always be observed and they appear as the horizontal lines *AD* (black) and *CB* (red) (Figure S13b).

(iii) While for a partially self-sorted mixture, in some composition range only one T_m can be identified (*i.e.* non-self-sorted composition), and in another composition range, two T_m values can be found (*i.e.* fully or partially self-sorted composition), and

the higher T_m value will locate within any position inside the triangle ΔABC (Figure S13c). As a result, the polygonal red line *AFB* is always on the top of the dashed line *AB*. According to above elaborations, we proposed the ratio (β) of the area of the shadowed polygon to the area of the triangle (ABC) can be used to represent the degree of self-sorting in the binary mixture. The extents of self-sorting of the various mixed xerogels were then calculated from the corresponding phase diagrams (Figure S14).



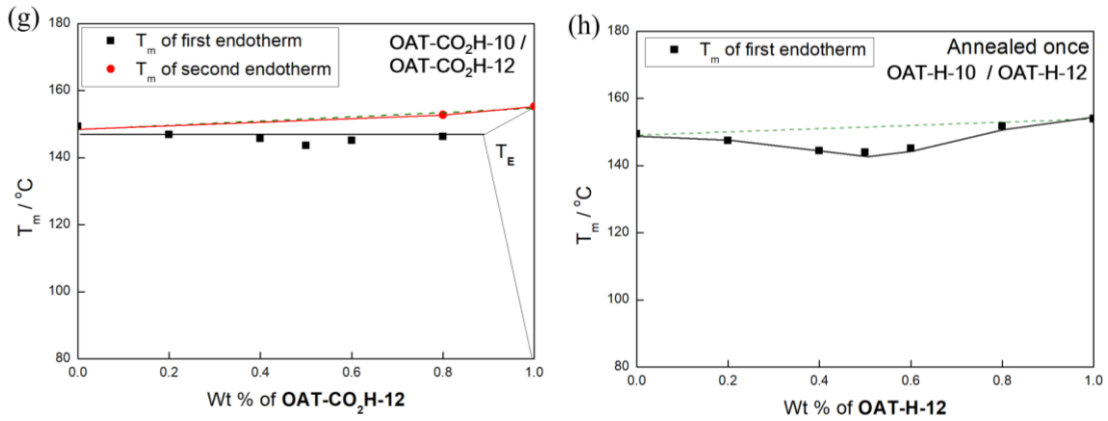


Figure S15. Melting temperatures (T_m) at various compositions and the miscibility curve (---) for (a) xerogels of **OAT-CO₂H-6**/**OAT-CO₂H-8,** (b) annealed blends of **OAT-H-6**/**OAT-H-8**, (c) xerogels of **OAT-CO₂H-8**/**OAT-CO₂H-12**, (d) annealed blends of **OAT-H-6**/**OAT-H-12**, (e) xerogels of **OAT-CO₂H-8**/**OAT-CO₂H-12**, (f) annealed blends of **OAT-H-8**/**OAT-H-12**, (g) xerogels of **OAT-CO₂H-10**/**OAT-CO₂H-12**, (h) annealed blends of **OAT-H-10**/**OAT-H-12**. The black and red symbols represent the T_m of first and second endothermic peak, respectively, and T_E denotes the eutectic temperature.

8. SEM images

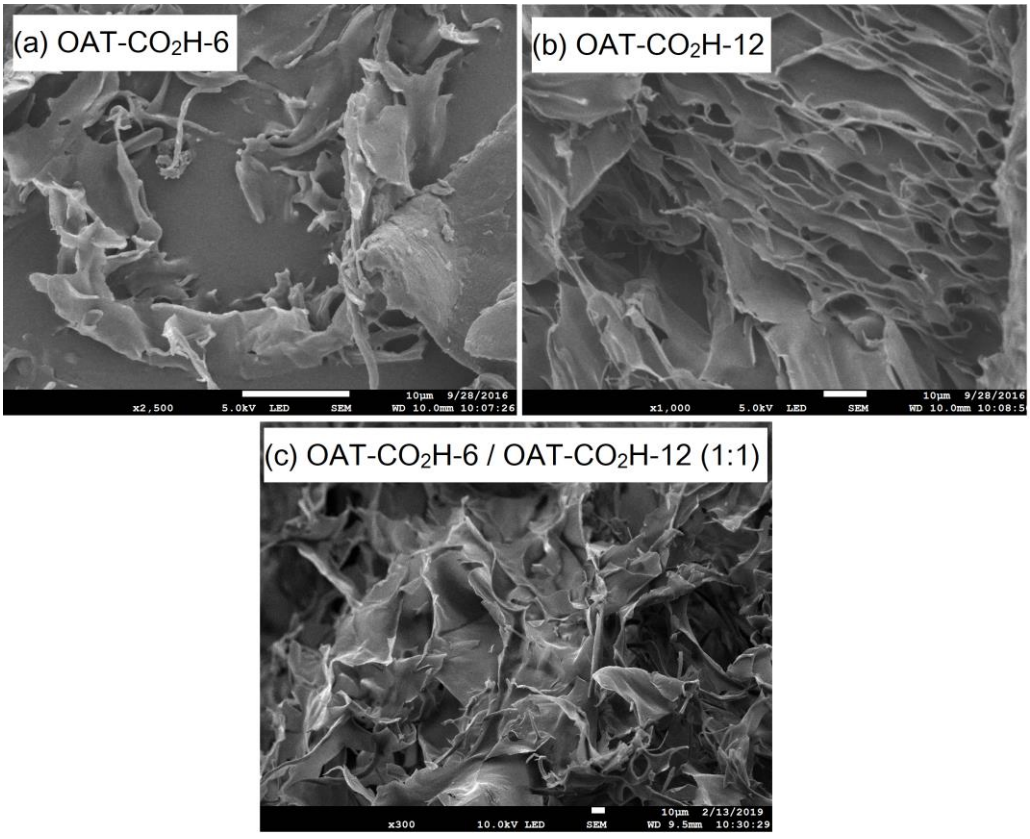


Figure S16. SEM images of freeze dried gels of (a) **OAT-CO₂H-6**, (b) **OAT-CO₂H-12** and (c) 1:1 mixture of **OAT-CO₂H-6**/**OAT-CO₂H-12**.

9. ^{1}H NMR spectra of annealed samples

For annealed **OAT-CO$_2$H-8**, **OAT-CO$_2$H-10** and **OAT-CO$_2$H-12**, similar changes were observed in the ^{1}H NMR spectra in which one signal of NH was upfield-shifted to about 5.8 ppm and a new small peak showed up at around 2.08 ppm (Figure S15). Such results proved the decarboxylation reaction happened during annealing process. For annealed **OAT-COPrg-7**, no obvious change was found in the spectrum after annealing (Figure S16).

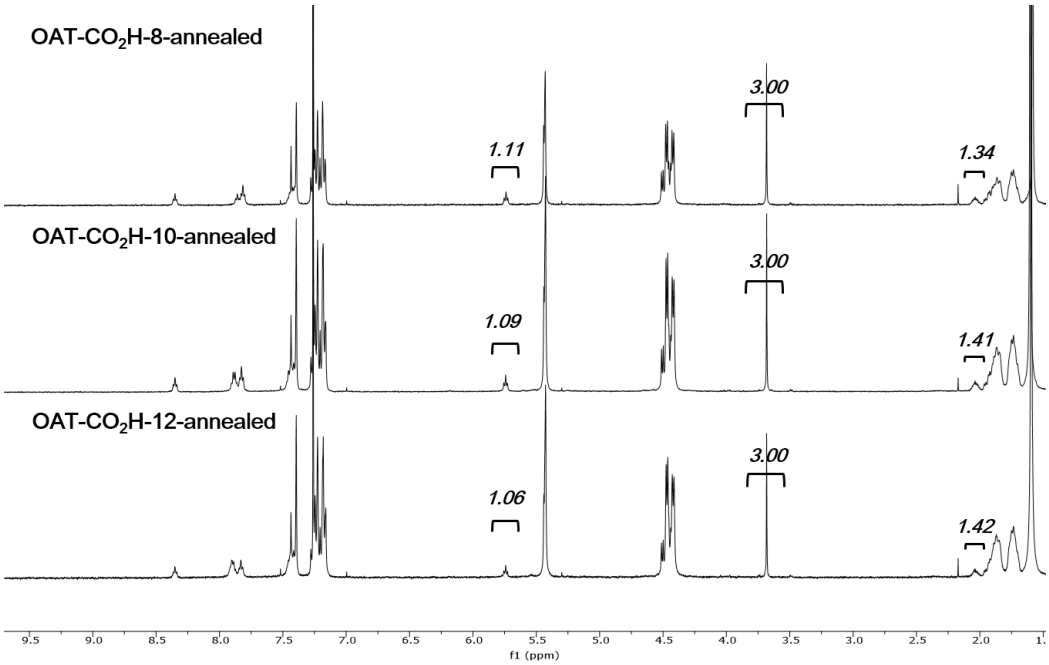


Figure S17. Stacked ^{1}H NMR (400 MHz, $CDCl_3$, 25 ºC) spectra of annealed **OAT-CO$_2$H-8**, **OAT-CO$_2$H-10** and **OAT-CO$_2$H-12**.

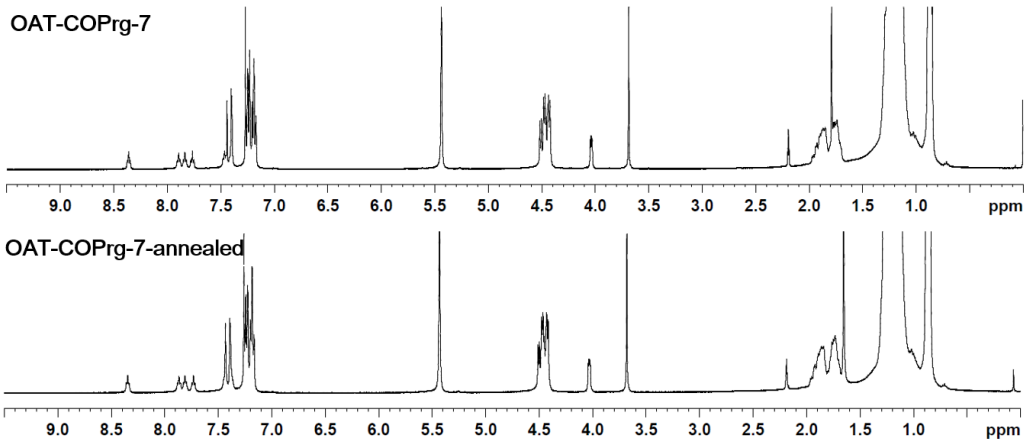


Figure S18. Stacked ^{1}H NMR (400 MHz, $CDCl_3$, 25 ºC) spectra of original **OAT-COPrg-7** (top) and annealed **OAT-COPrg-7** (bottom).

10. ^{1}H NMR spectral end group analysis

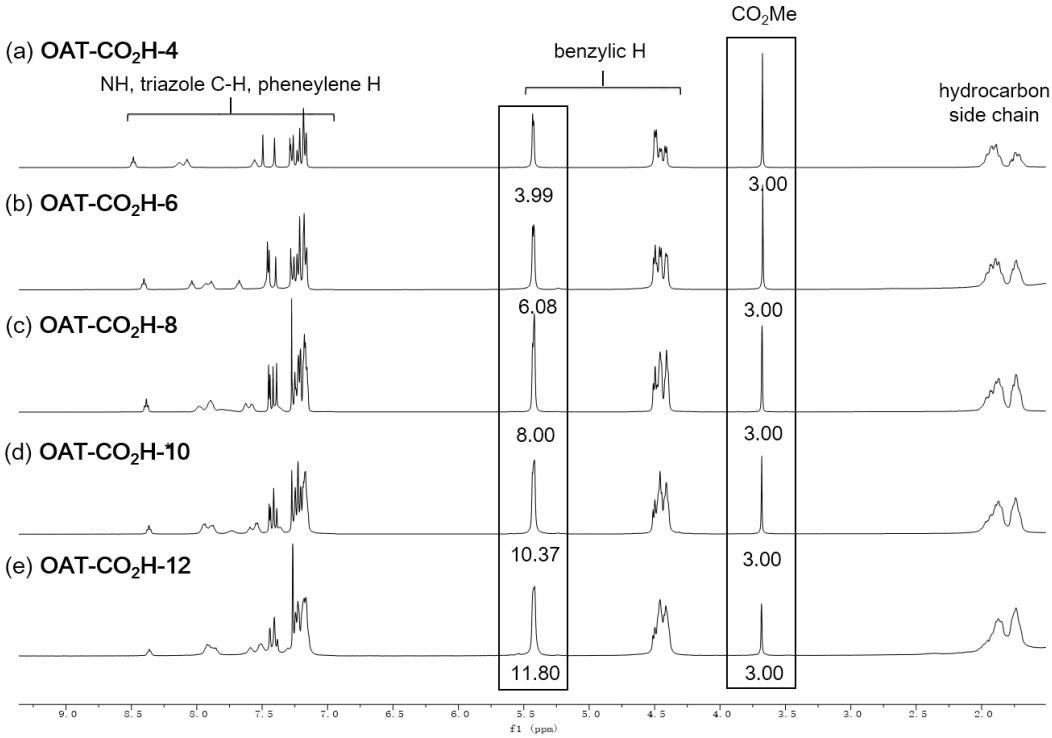


Figure S19. Stacked partial ^{1}H NMR spectra (400 MHz, $CDCl_3$) of **OAT-CO₂H-2n** (n = 2–6) marked with relative integrals.

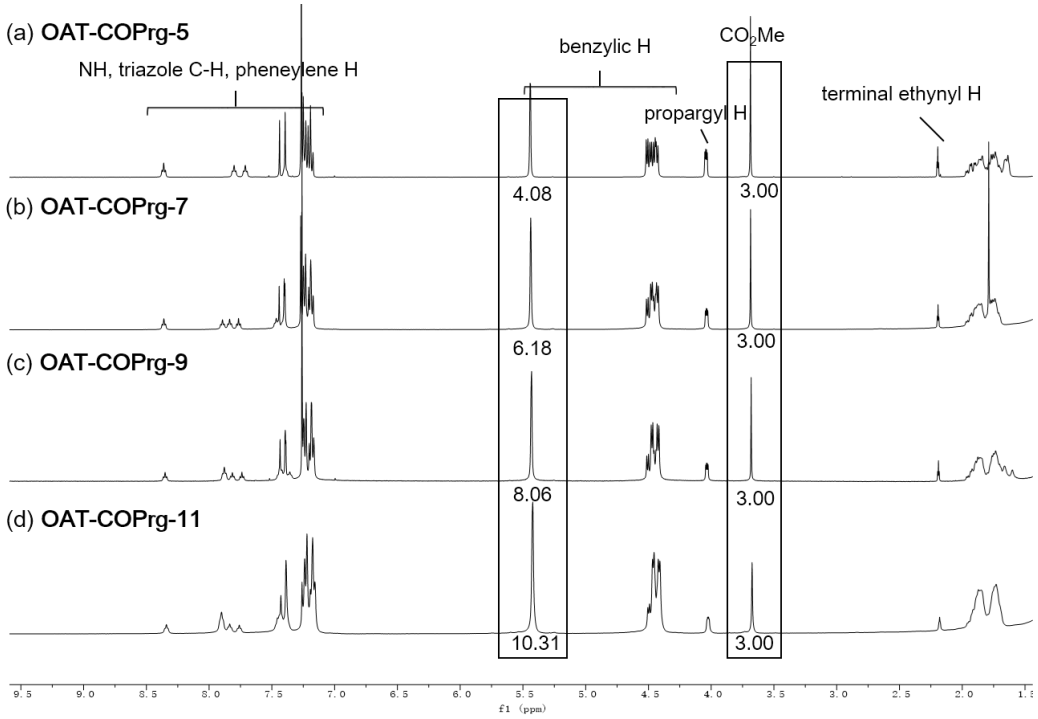


Figure S20. Stacked partial ^{1}H NMR spectra (400 MHz, $CDCl_3$) of **OAT-COPrg-(2n+1)** (n = 2–5) marked with relative integrals.

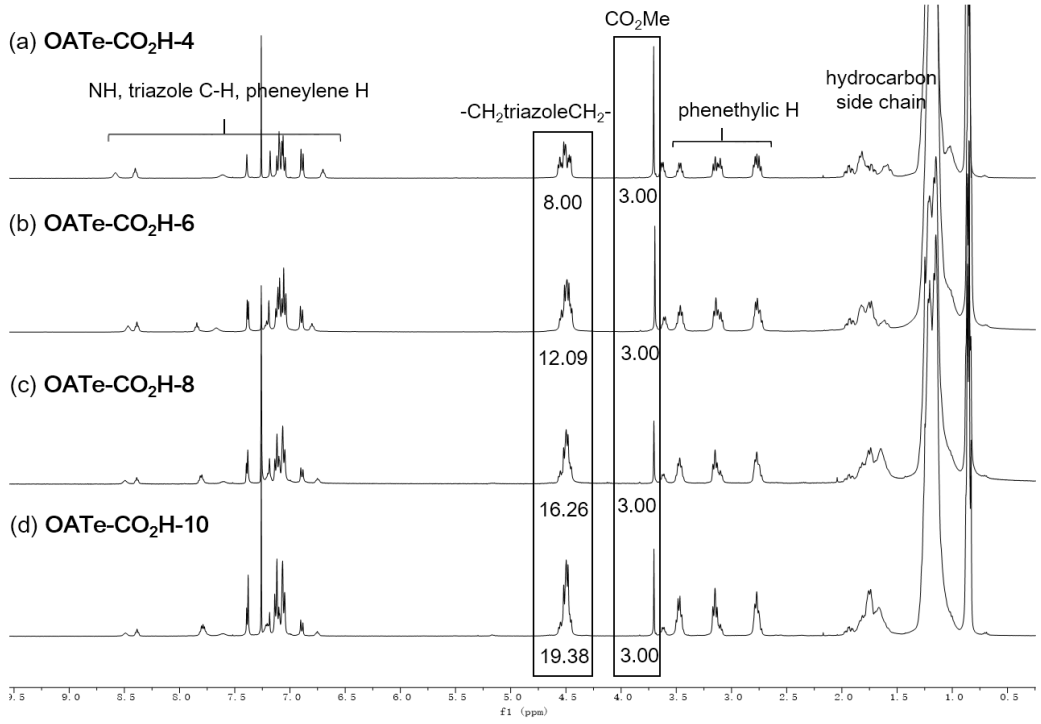


Figure S21. Stacked partial ^{1}H NMR spectra (400 MHz, $CDCl_3$) of **OATe-CO₂H-2n** (m = 2–5) marked with relative integrals.

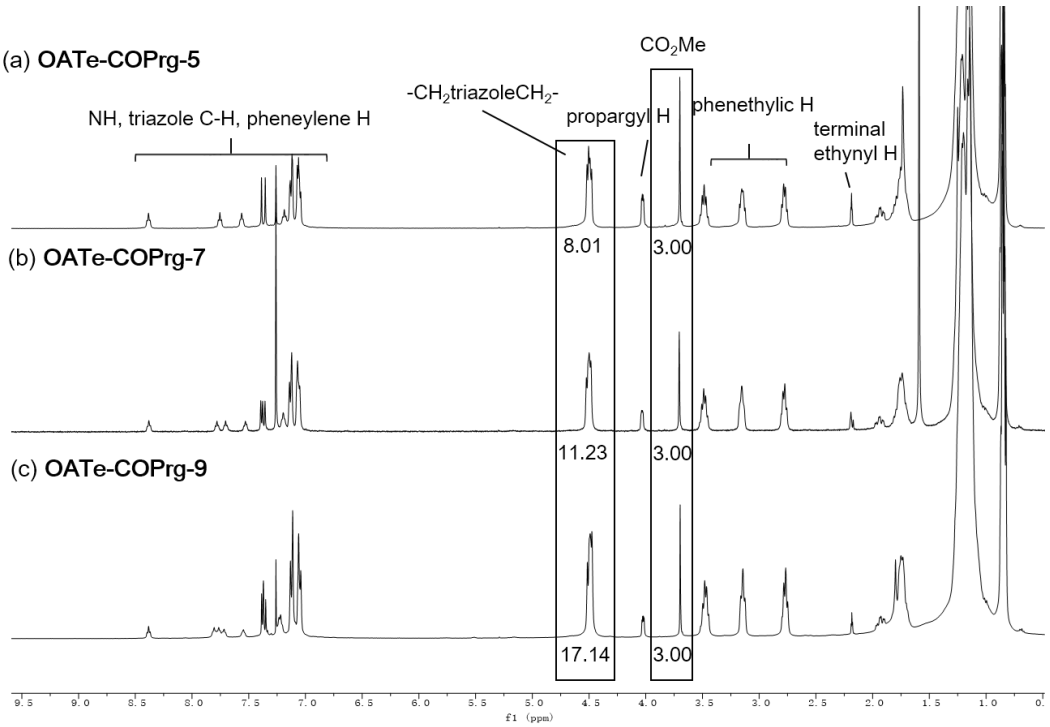
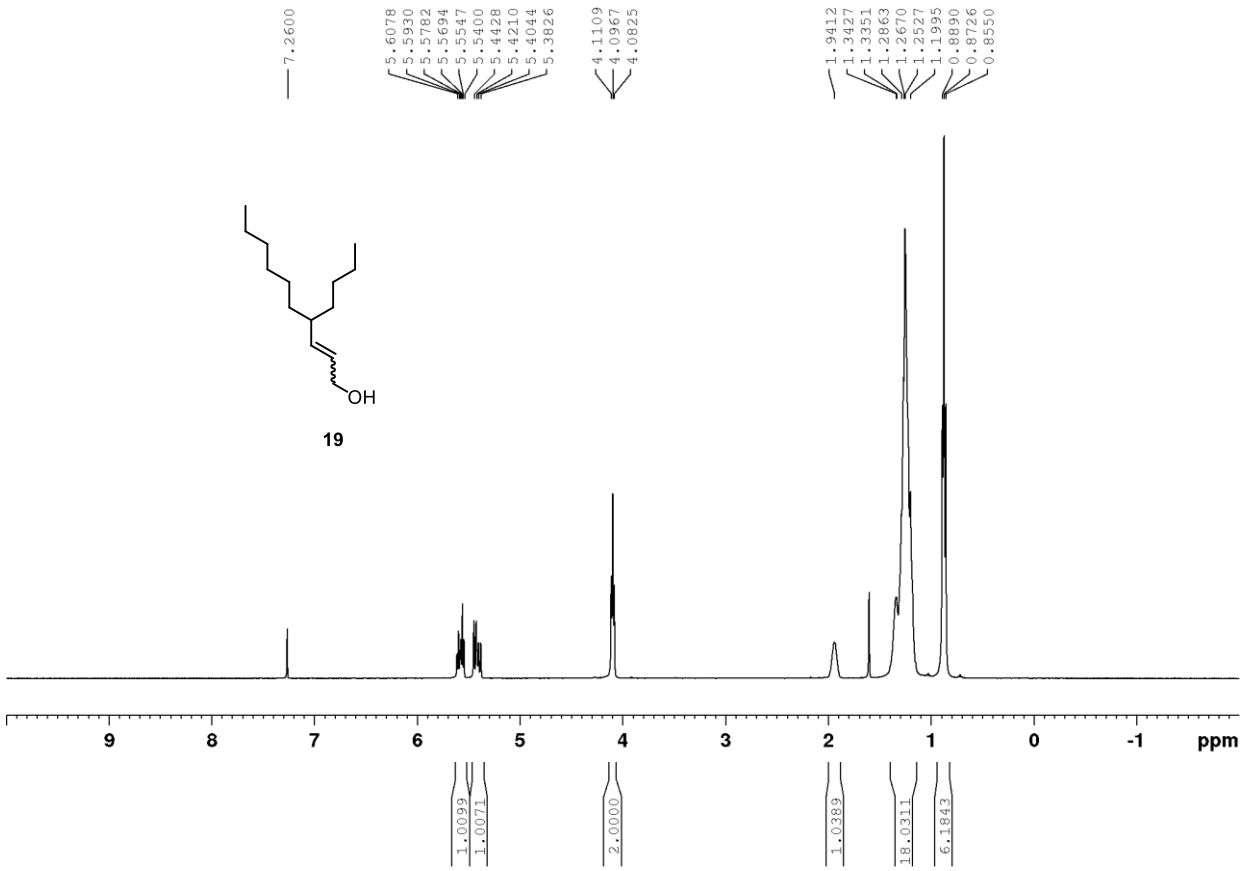


Figure S22. Stacked partial ^{1}H NMR spectra (400 MHz, $CDCl_3$) of **OATe-COPrg-(2n+1)** (n = 2–4) marked with relative integrals.

10. List of spectra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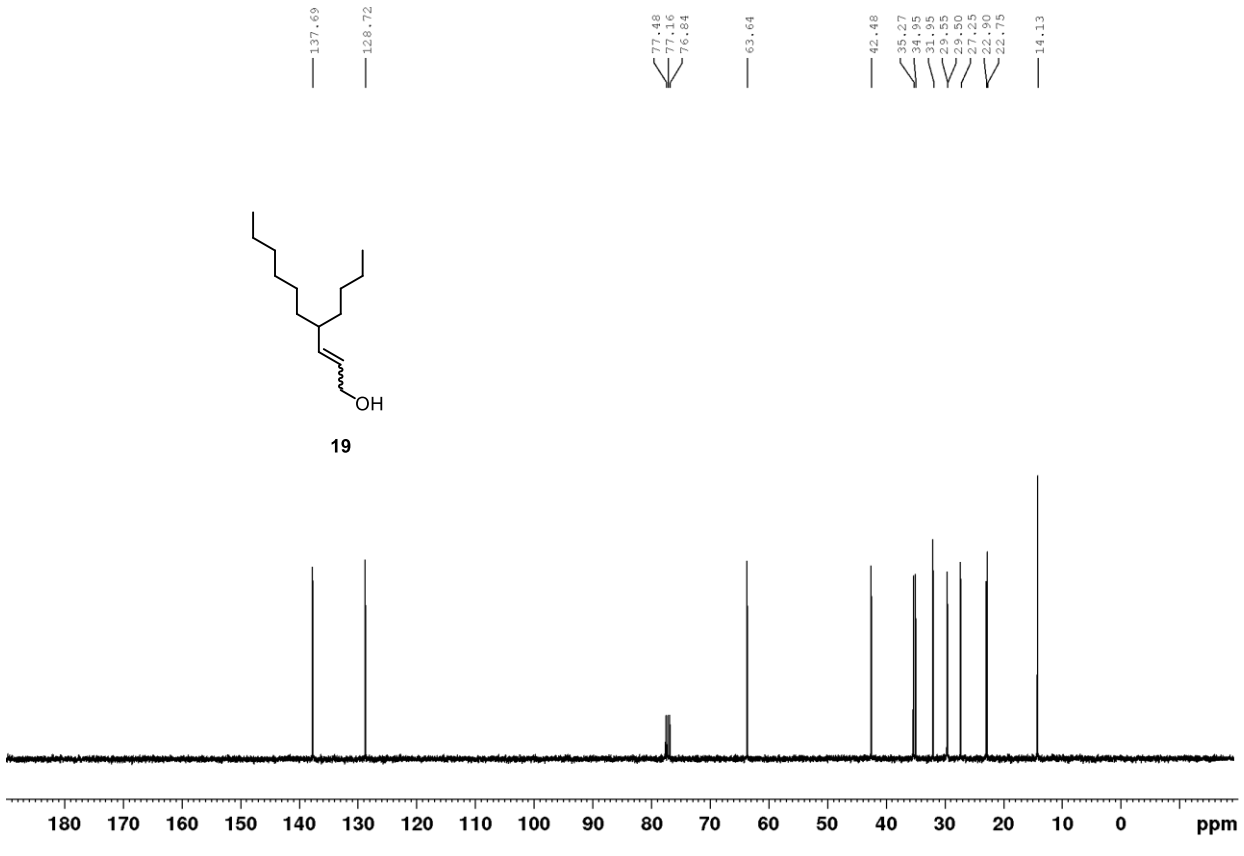
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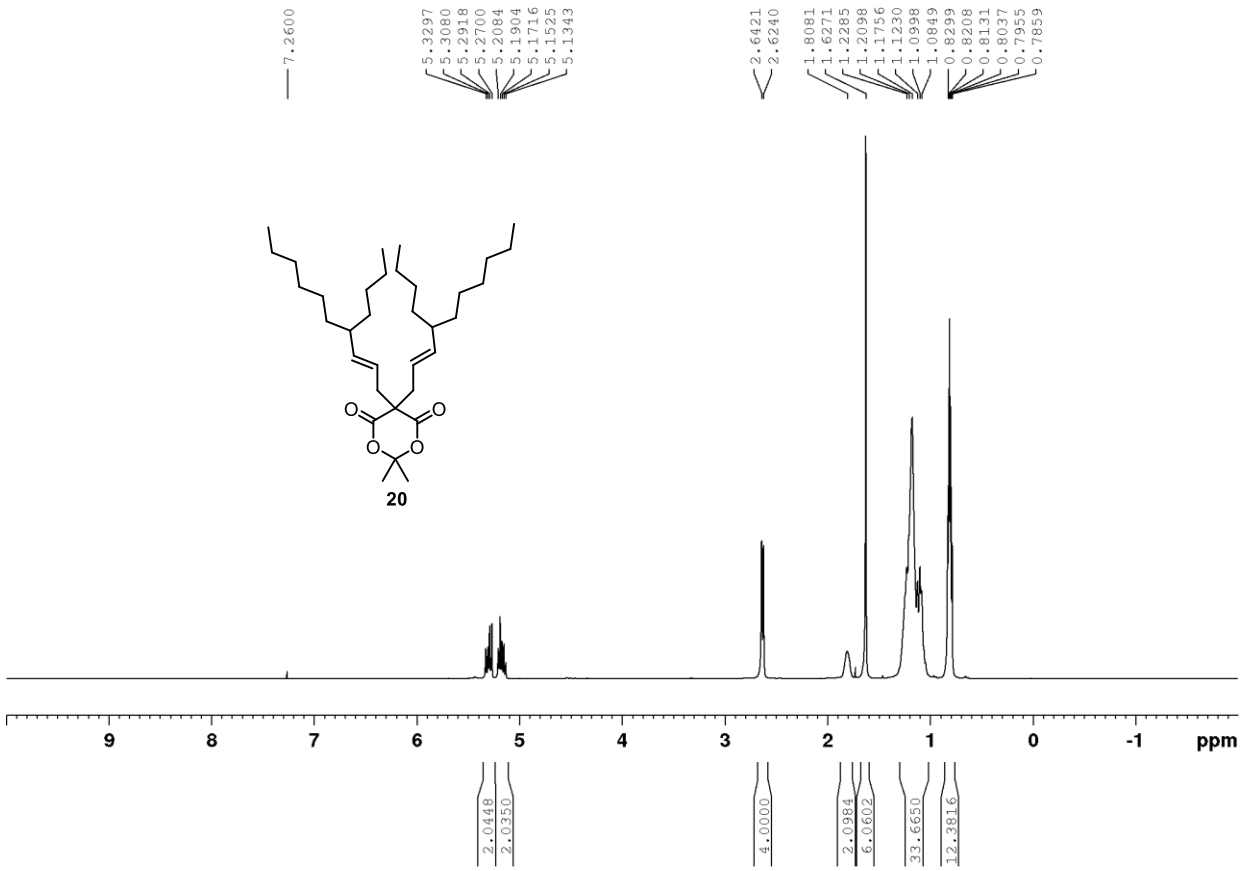
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0.8208
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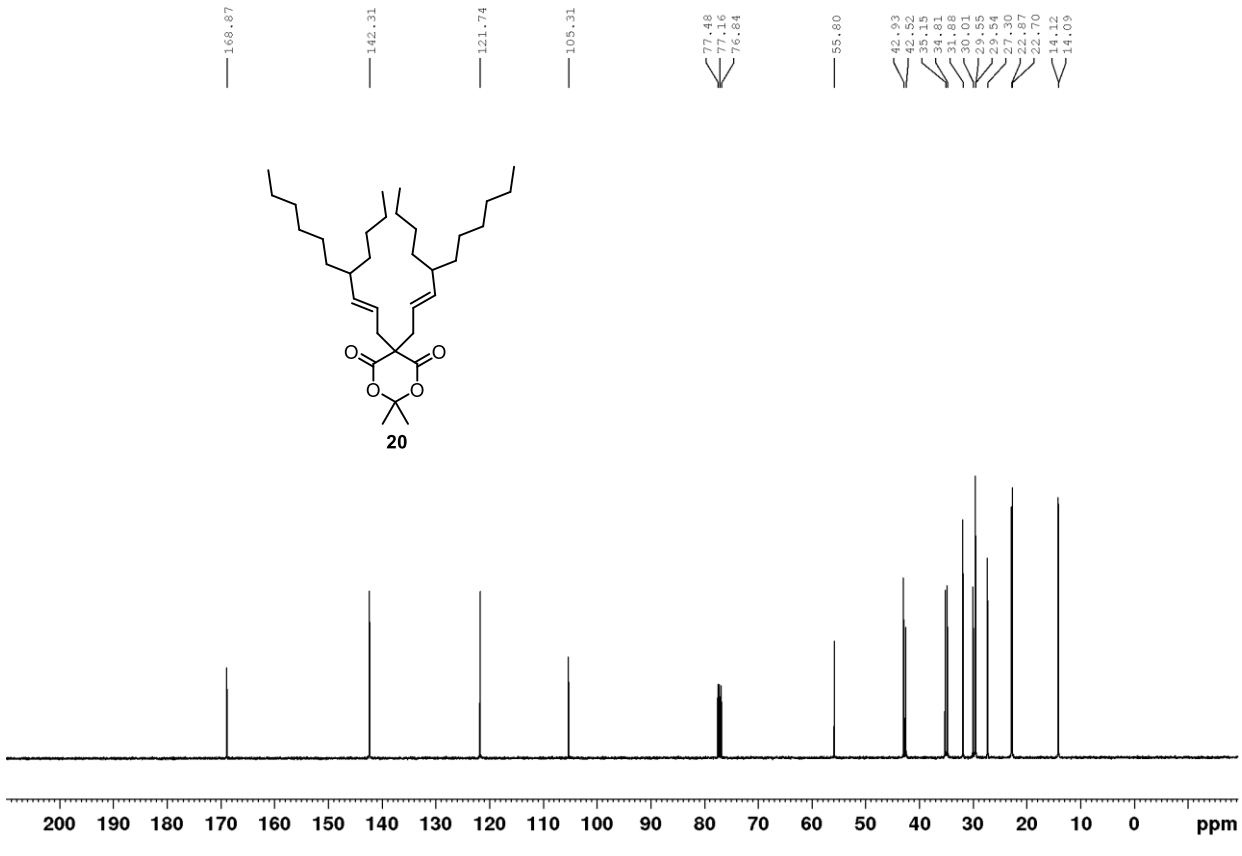
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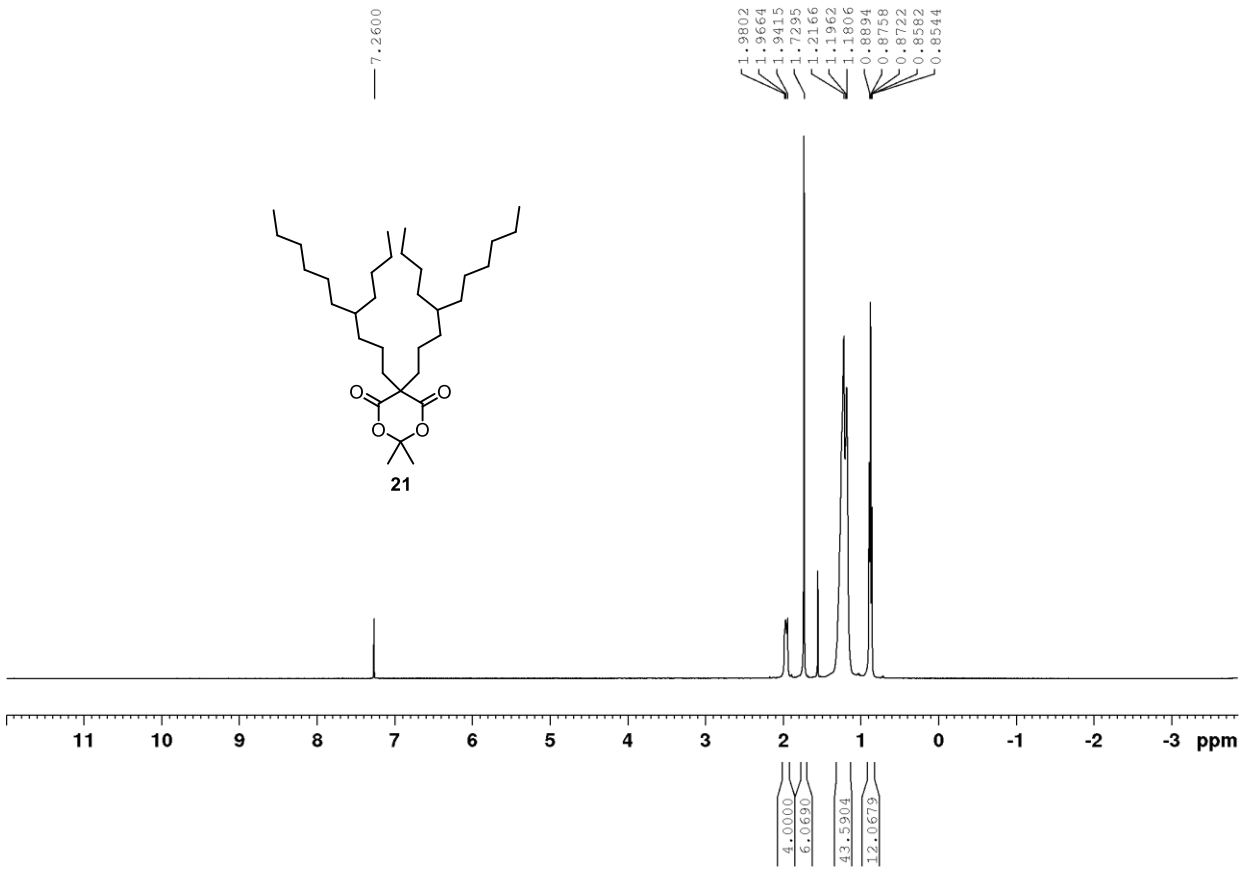
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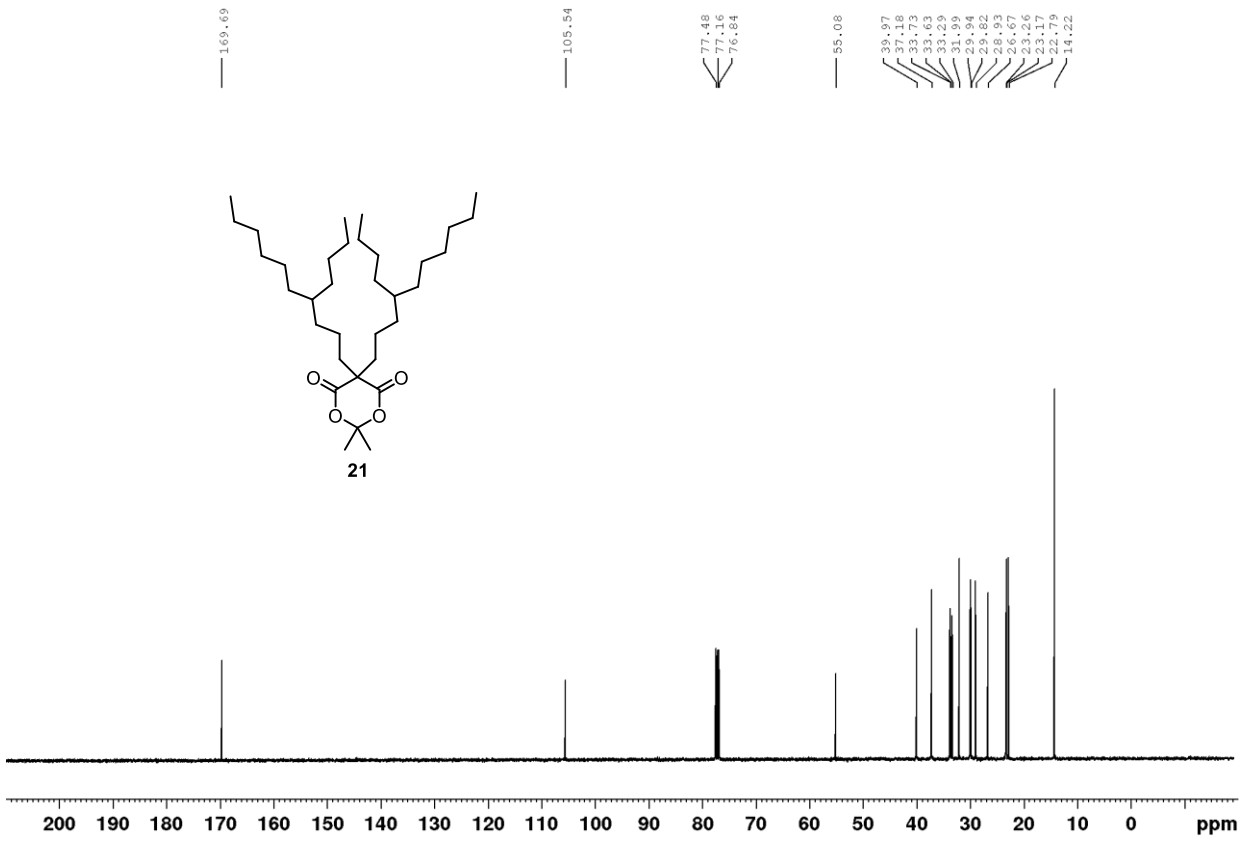
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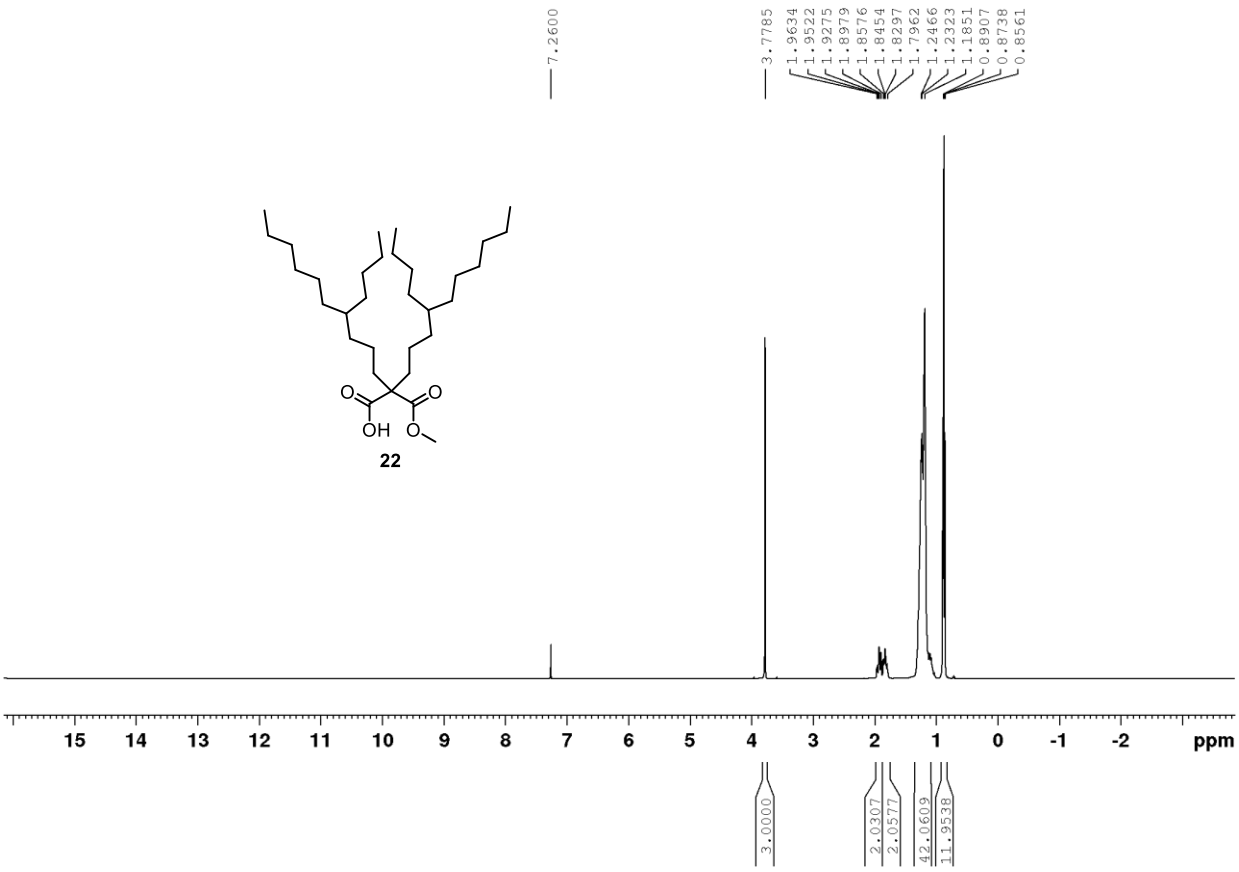
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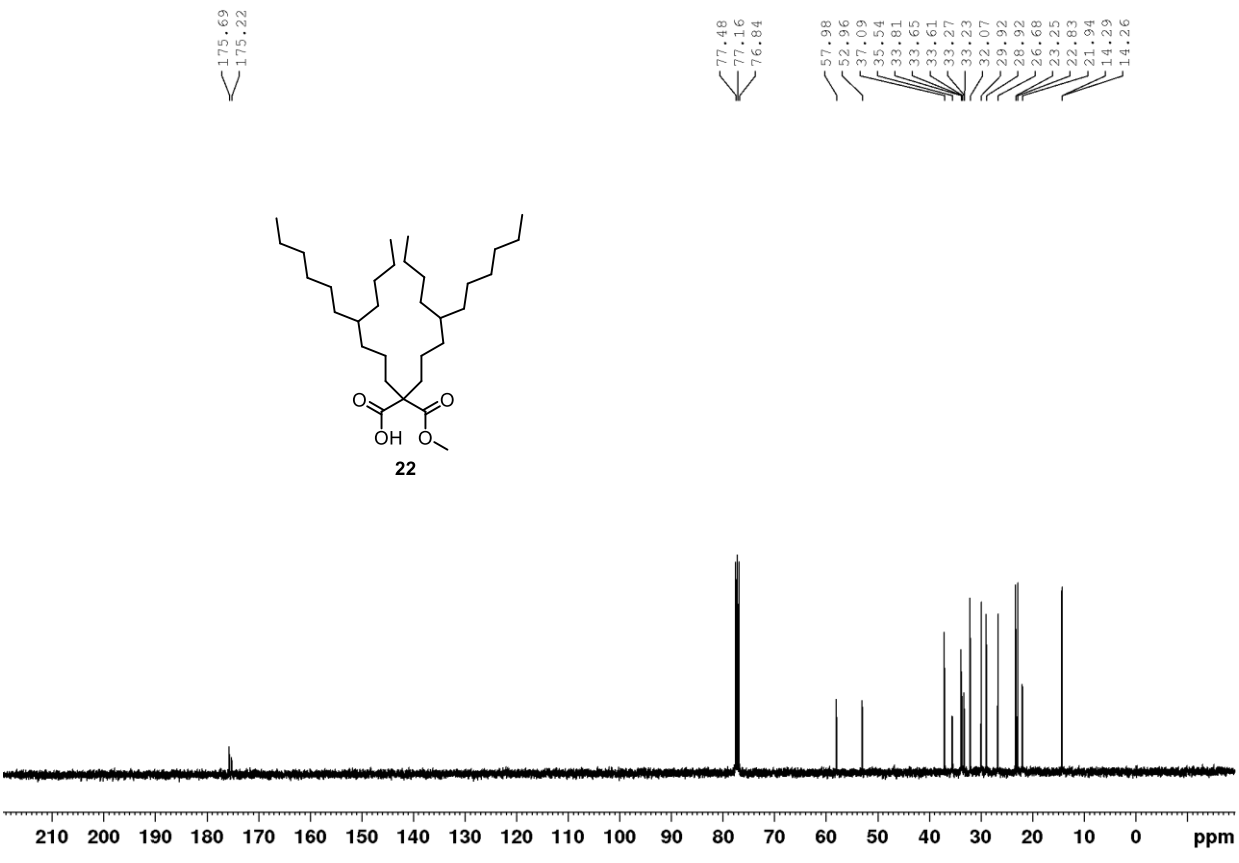
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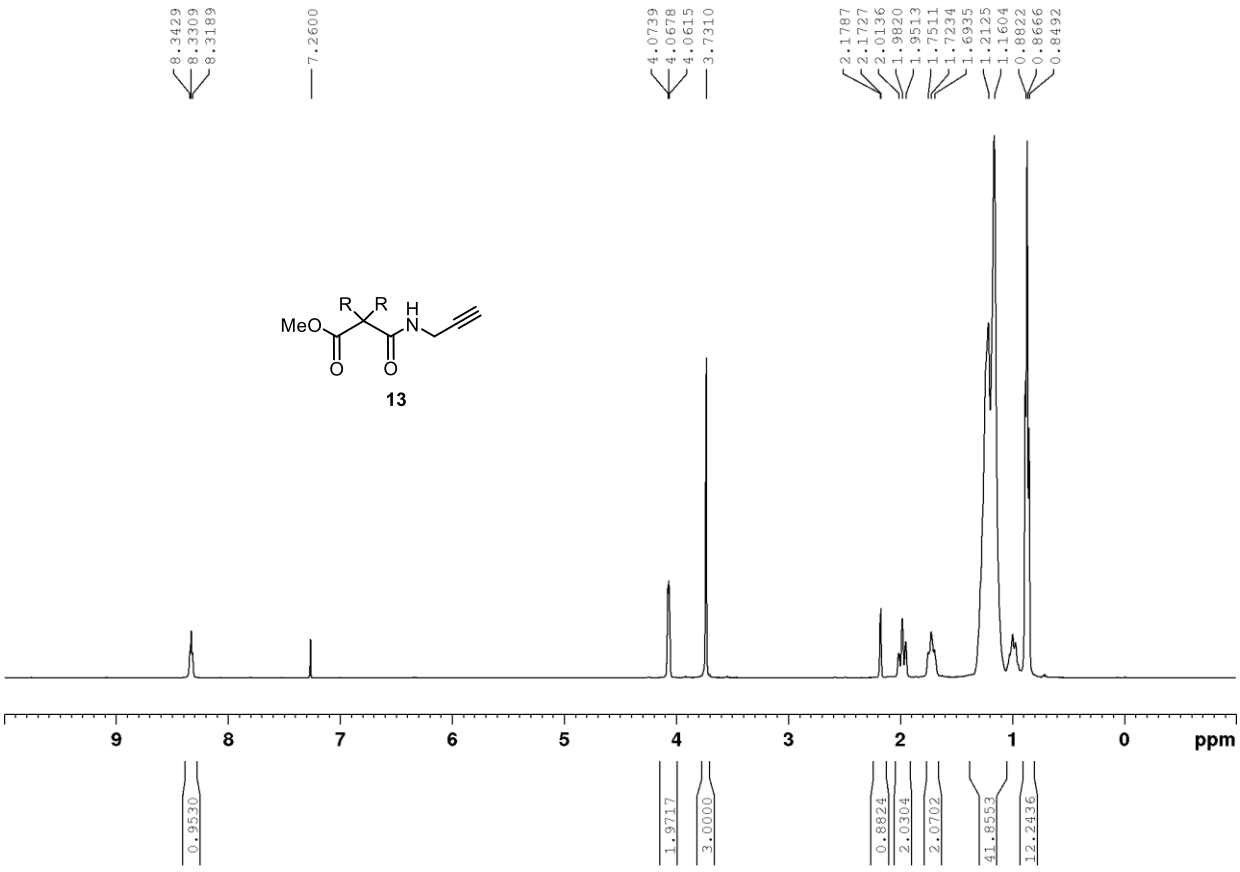
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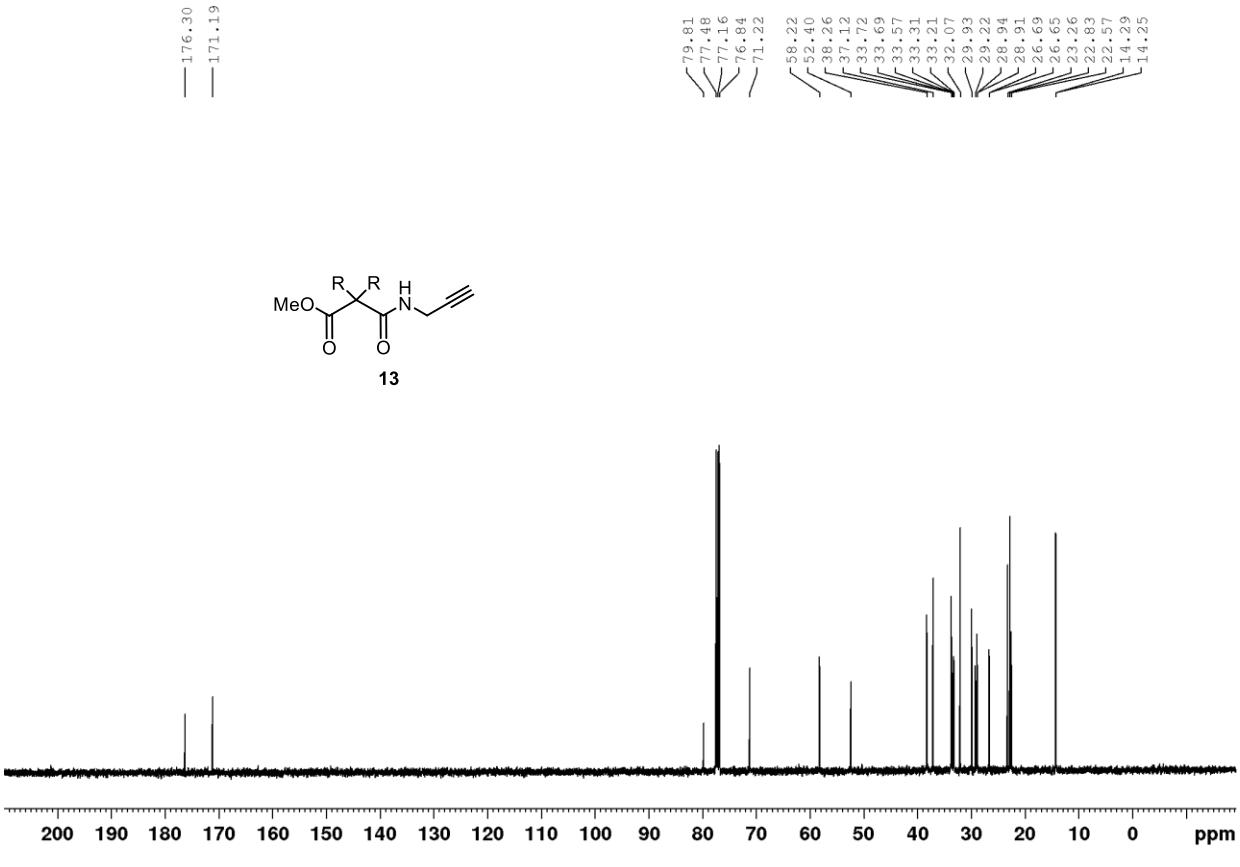
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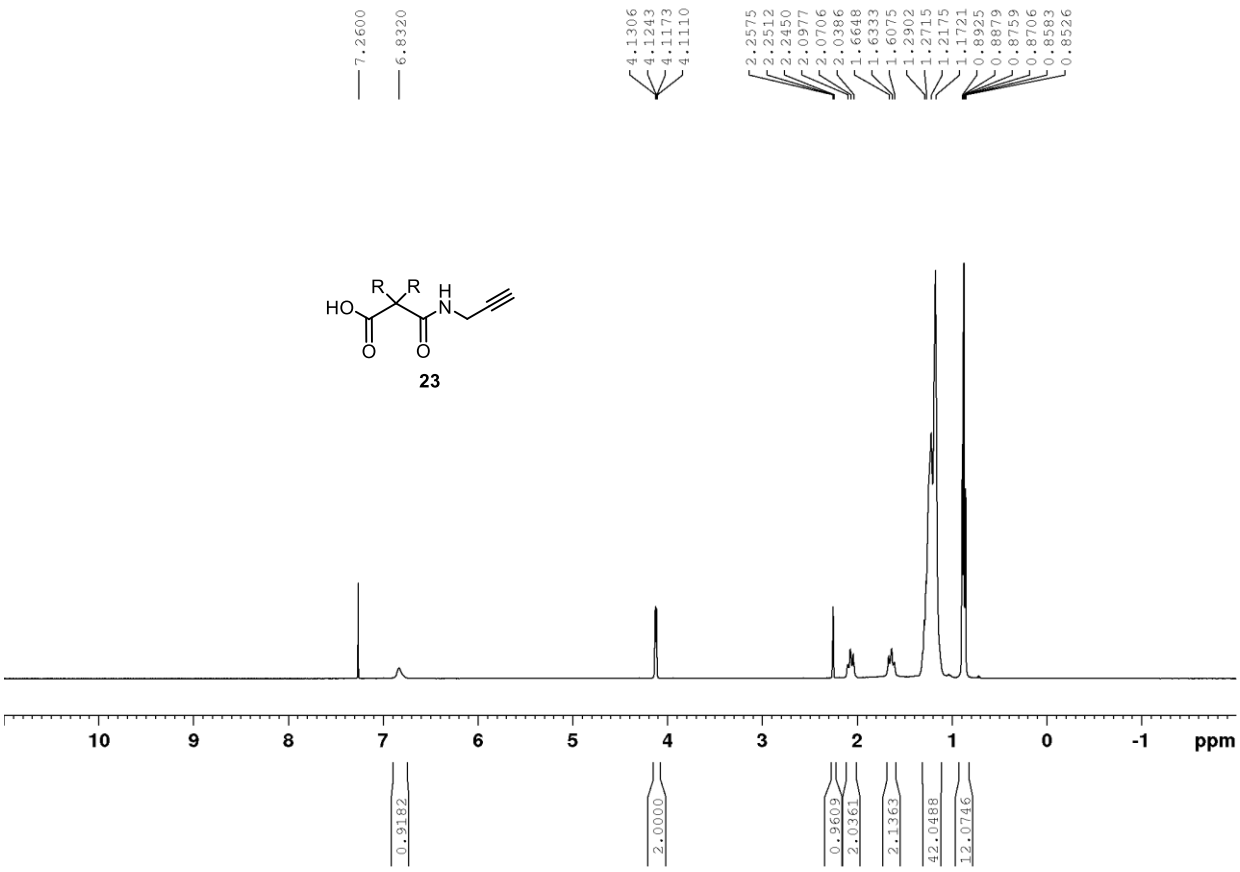
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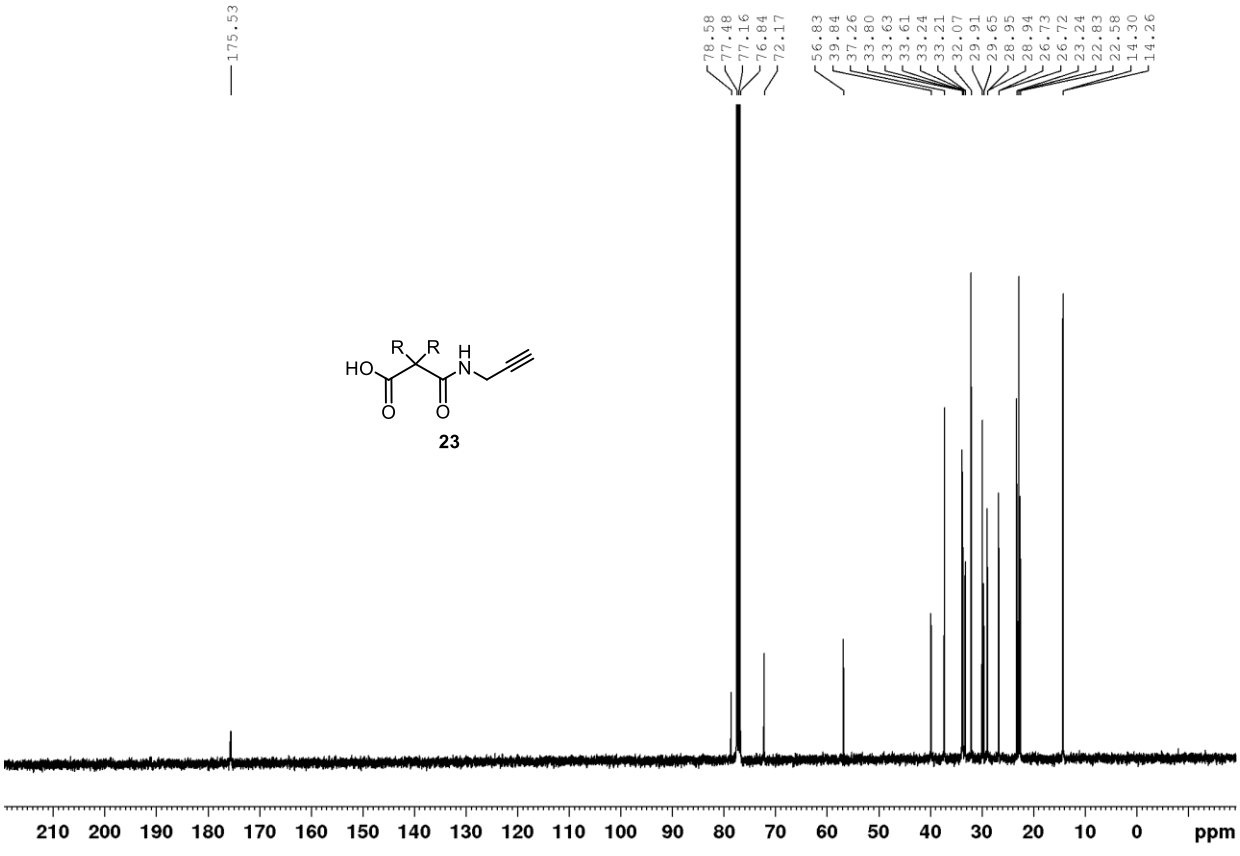
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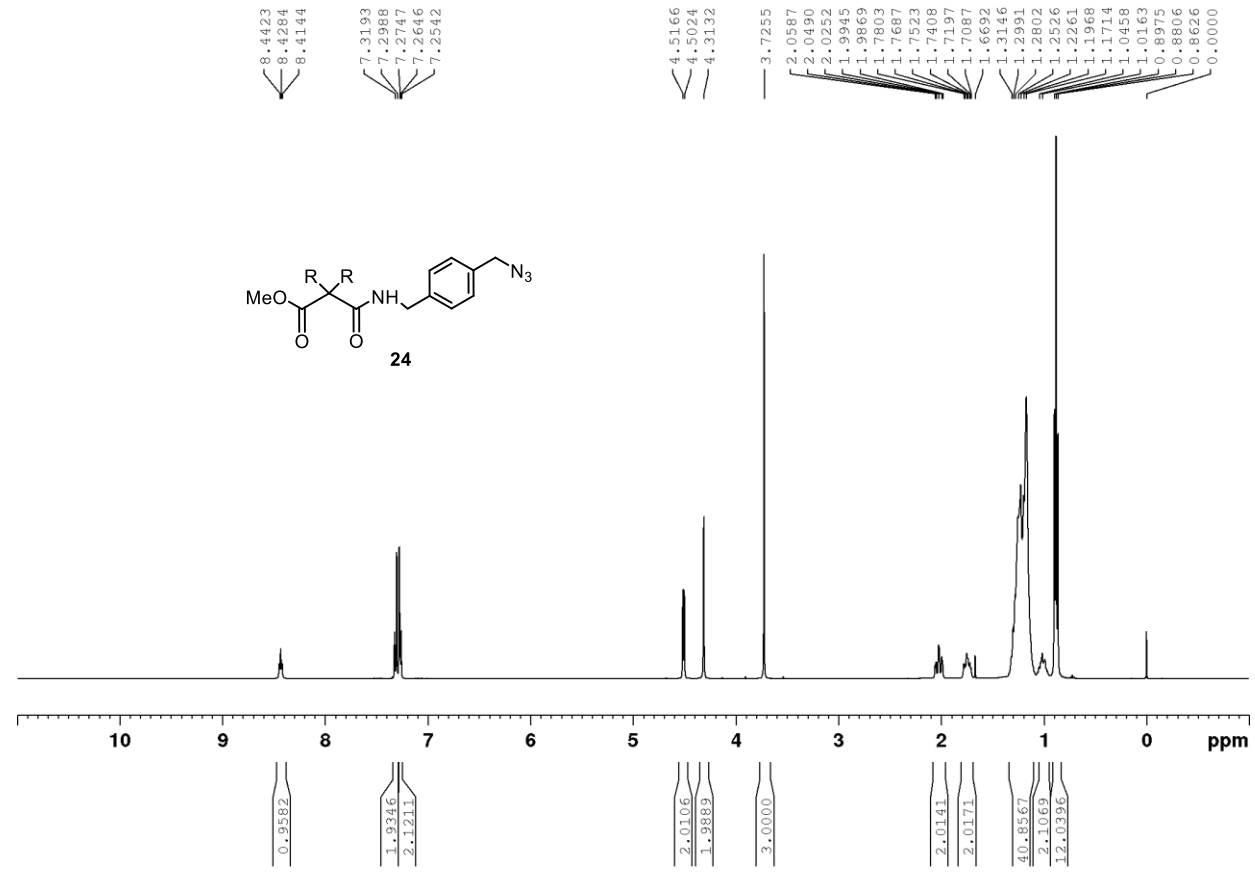
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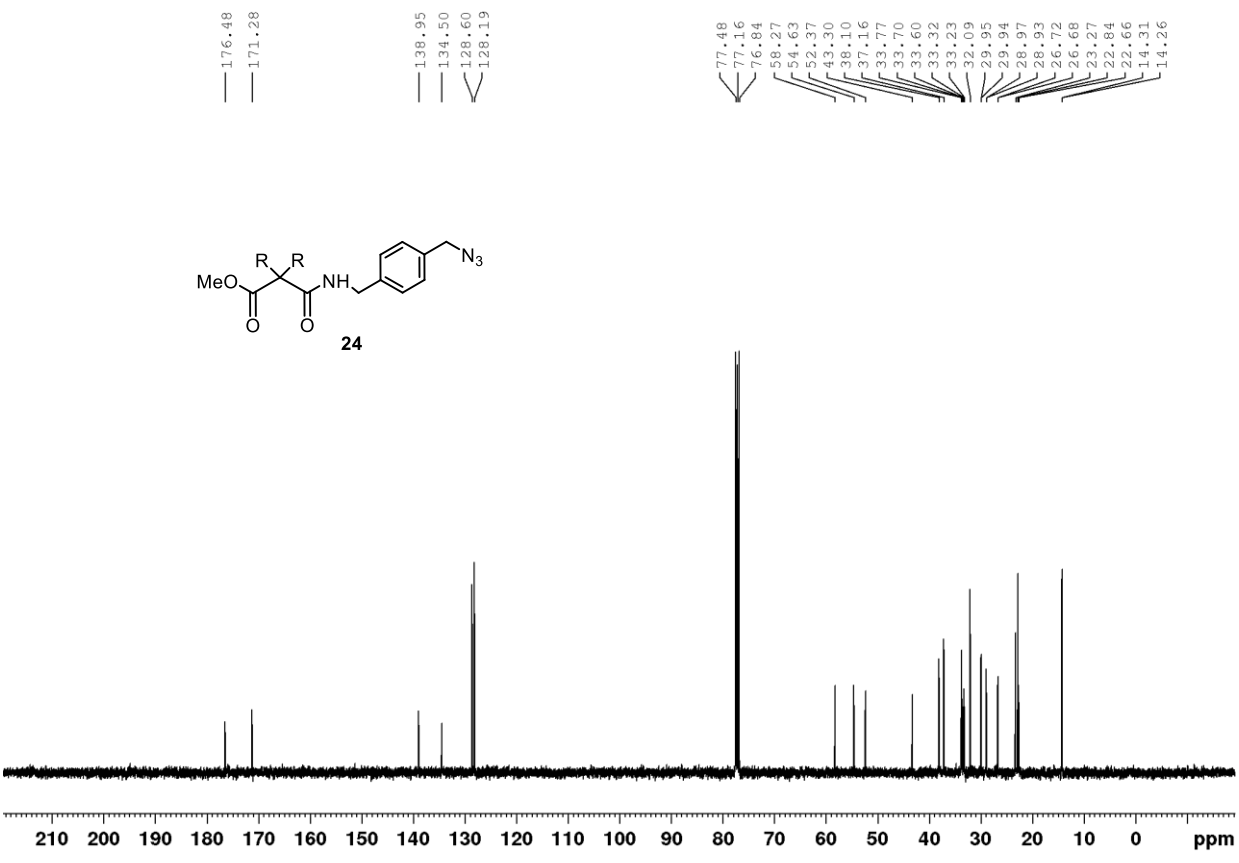
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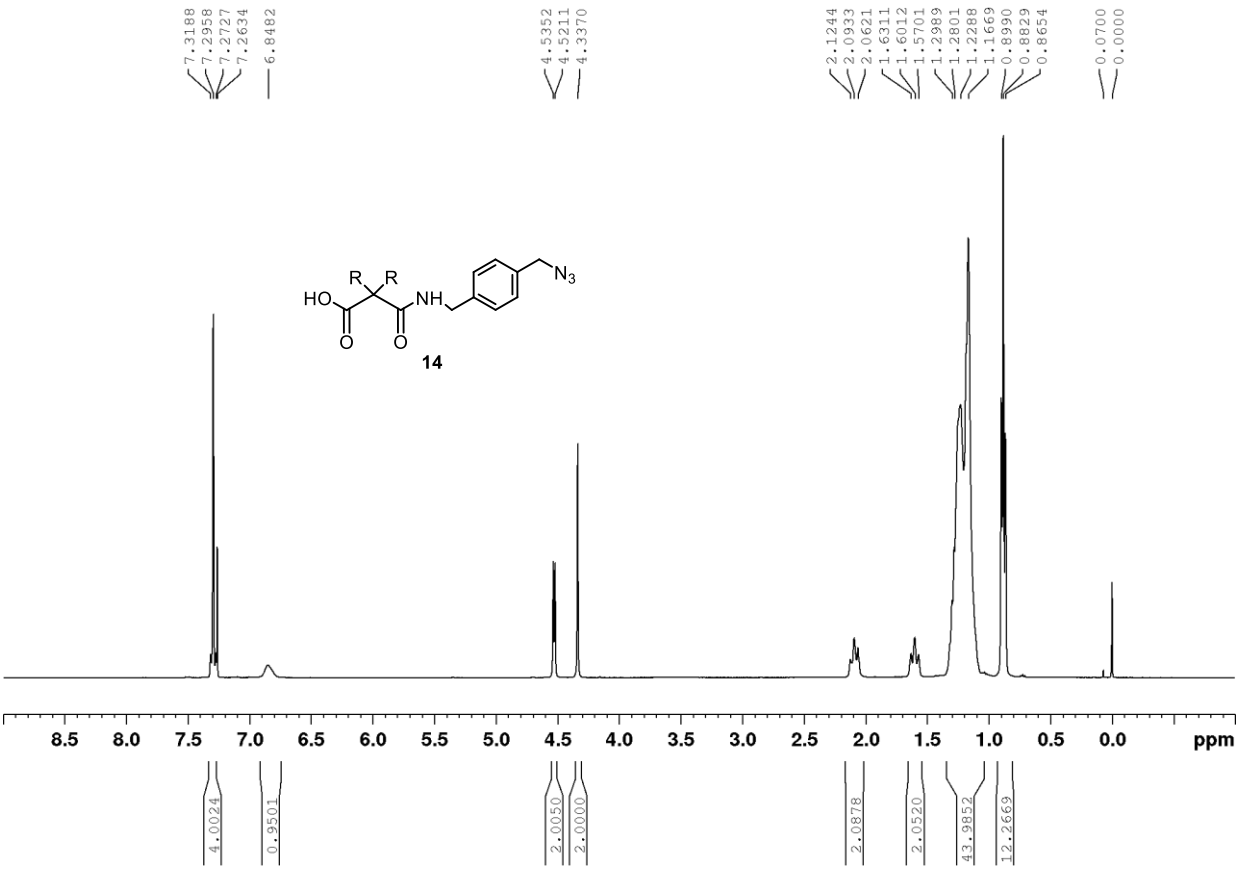
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4.5211
4.3370
2.1244
2.0933
2.0621
1.6311
1.6012
1.5701
1.2989
1.2801
1.2288
1.1669
0.8990
0.8829
0.8654
0.0700
0.0000
R
R
HO
NH
N3
O
O
14
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
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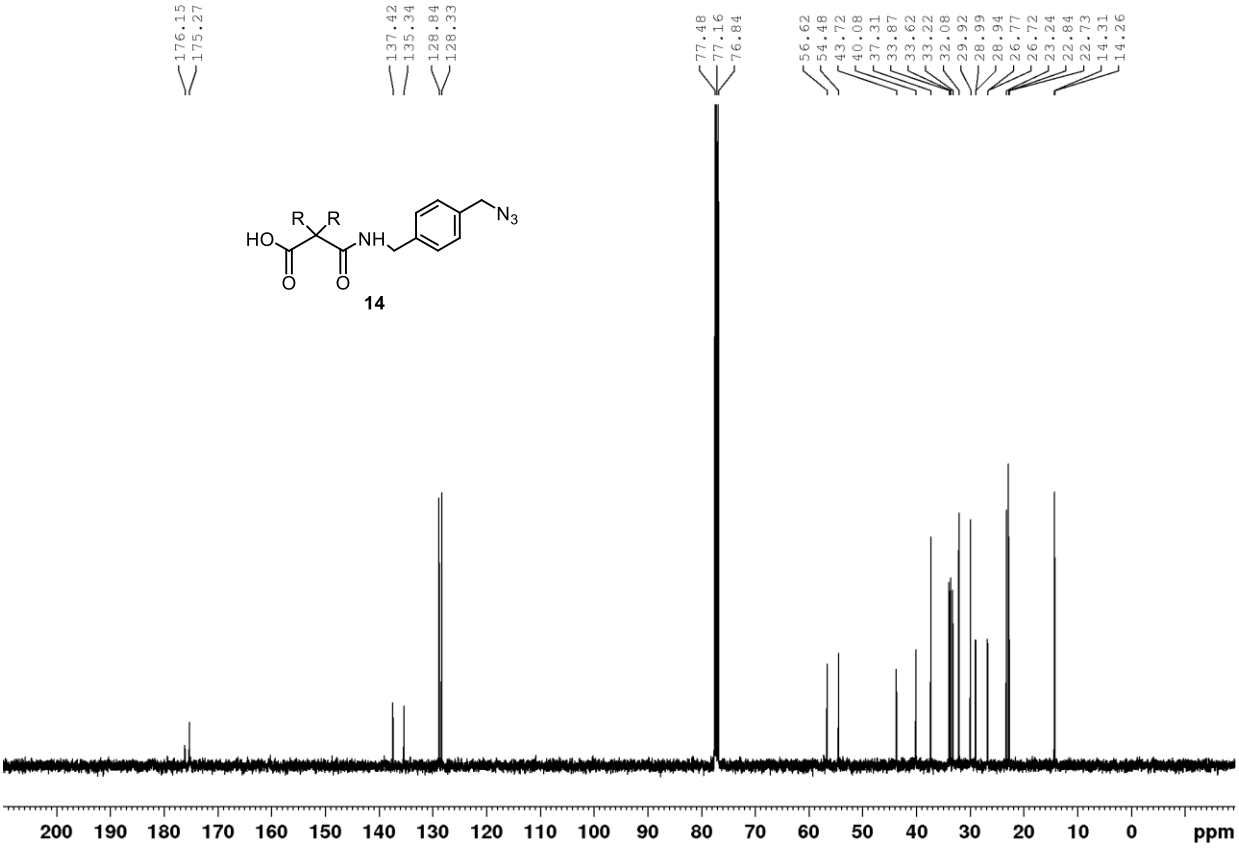
43.9852
12.2669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77.16
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40.08
37.31
33.87
33.62
33.22
32.08
29.92
28.99
28.94
26.77
26.72
23.24
22.84
22.73
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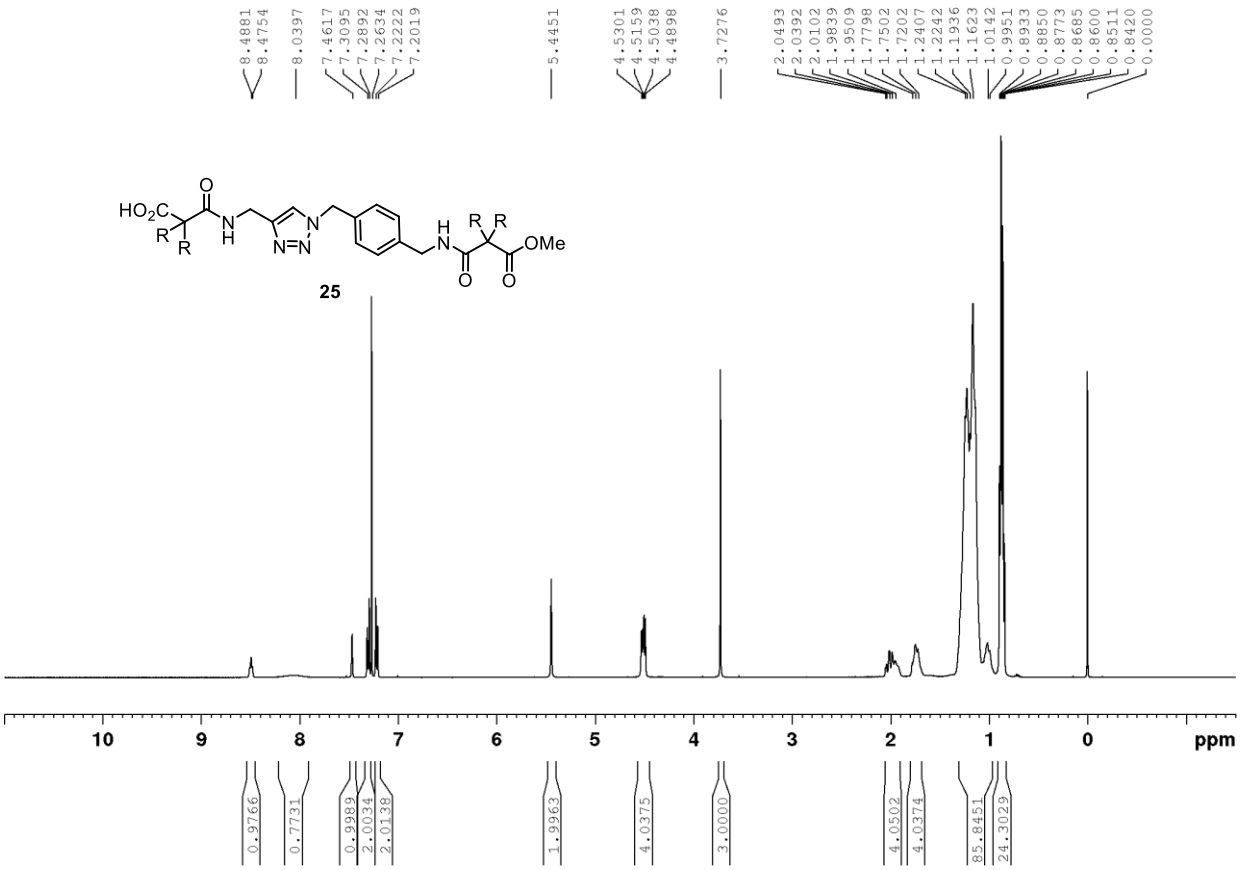
R
R
HO
NH
N3
O
O
14
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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4.5159
4.5038
4.4898
3.7276
2.0493
2.0392
2.0102
1.9839
1.9509
1.7798
1.7502
1.7202
1.2407
1.2242
1.1936
1.1623
1.0142
0.9951
0.8933
0.8850
0.8773
0.8685
0.8600
0.8511
0.8420
0.0000
HO2C
R
R
O
N
H
N
N=N
H
N
R
R
O
O
OMe
25
10
9
8
7
6
5
4
3
2
1
0
ppm
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0.7731
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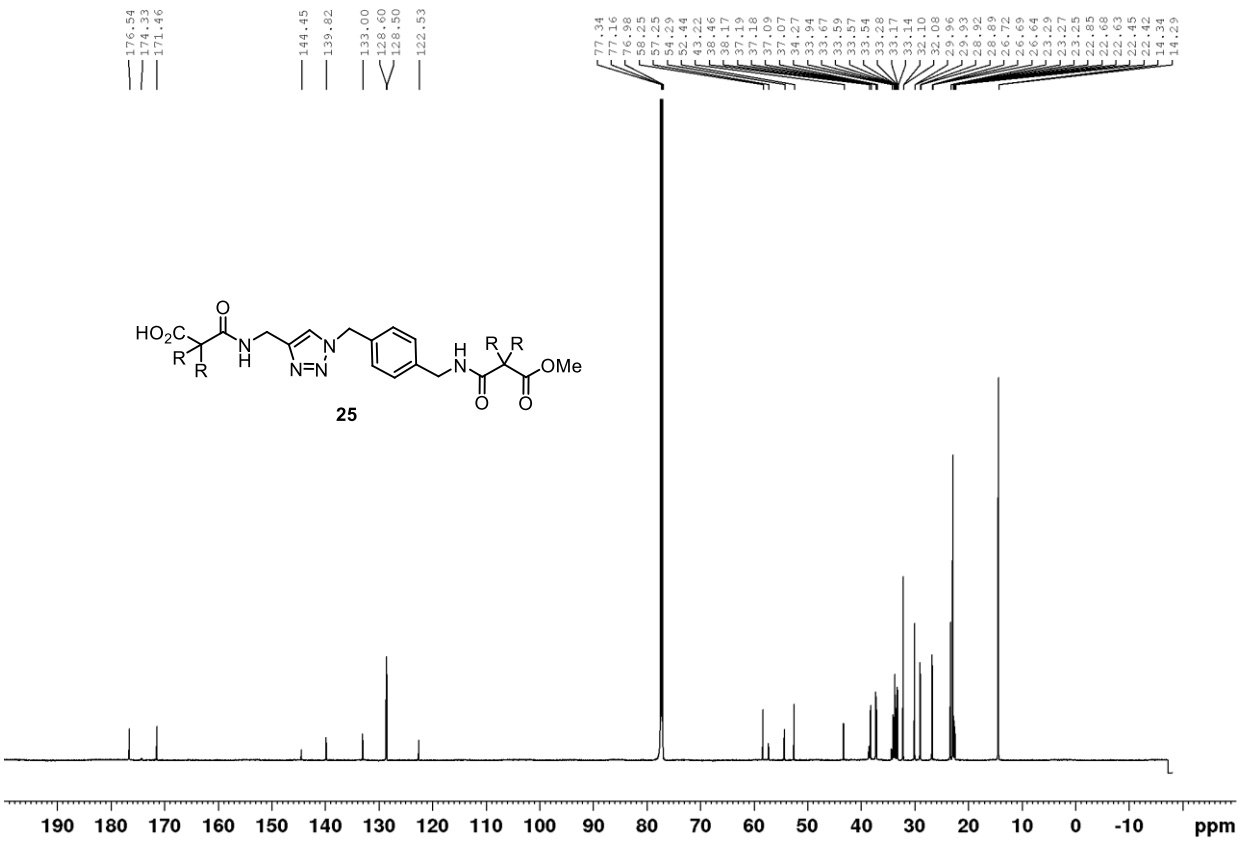
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57.25
54.29
52.44
43.22
38.46
38.17
37.19
37.18
37.09
37.07
34.27
33.94
33.67
33.59
33.57
33.54
33.28
33.17
33.14
32.10
32.08
29.96
29.93
28.92
28.89
26.72
26.69
26.64
23.29
23.27
23.25
22.85
22.68
22.63
22.45
22.42
14.34
14.29
HO2C
R
R
O
N
H
N
N=N
H
N
R
R
O
O
OMe
25
190
180
170
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150
140
130
120
110
100
90
80
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50
40
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10
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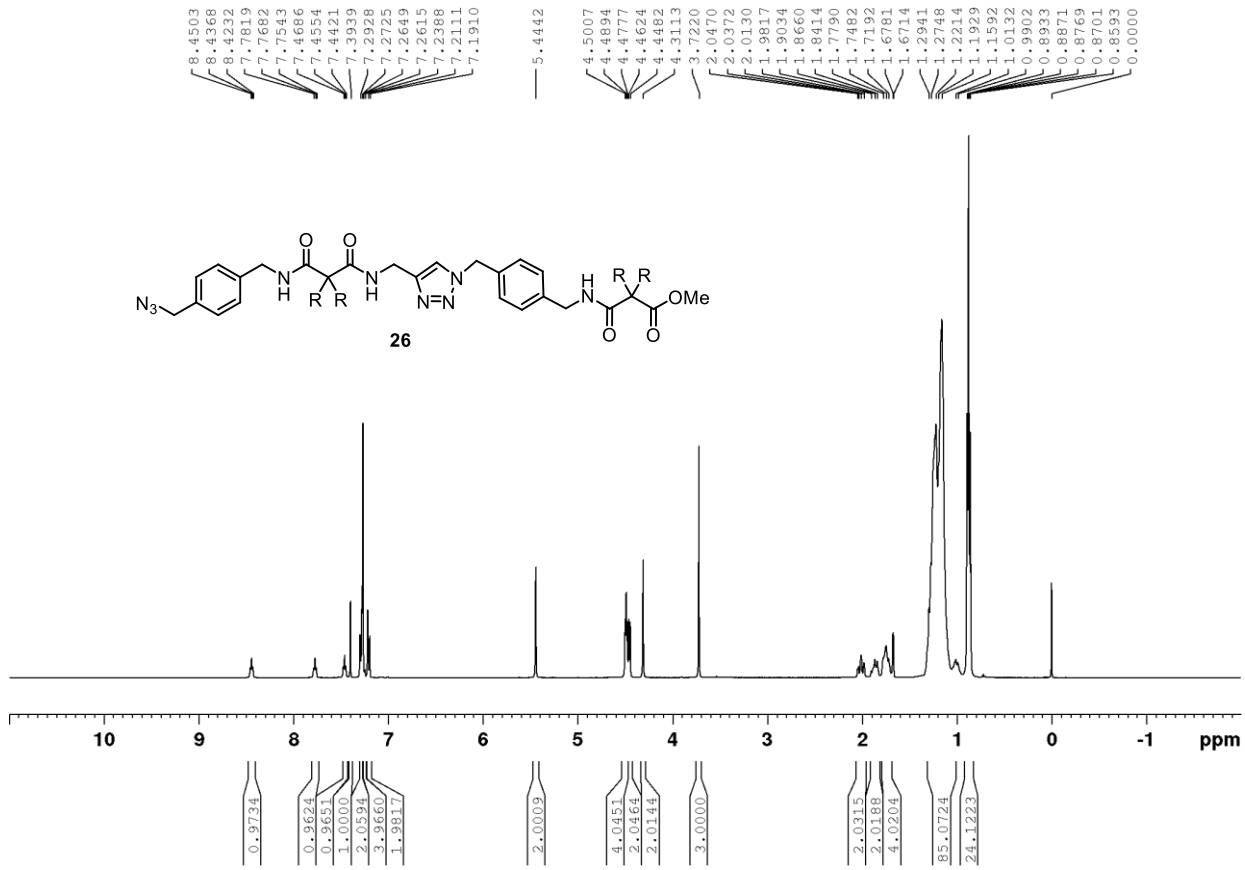
ppm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7.2725
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7.2615
7.2388
7.2111
7.1910
5.4442
4.5007
4.4894
4.4777
4.4624
4.4482
4.3113
3.7220
2.0470
2.0372
2.0130
1.9817
1.9034
1.8660
1.8414
1.7790
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1.7192
1.6781
1.6714
1.2941
1.2748
1.2214
1.1929
1.1592
1.0132
0.9902
0.8933
0.8871
0.8769
0.8701
0.8593
0.0000
26
10
9
8
7
6
5
4
3
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-1
ppm
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1.9817
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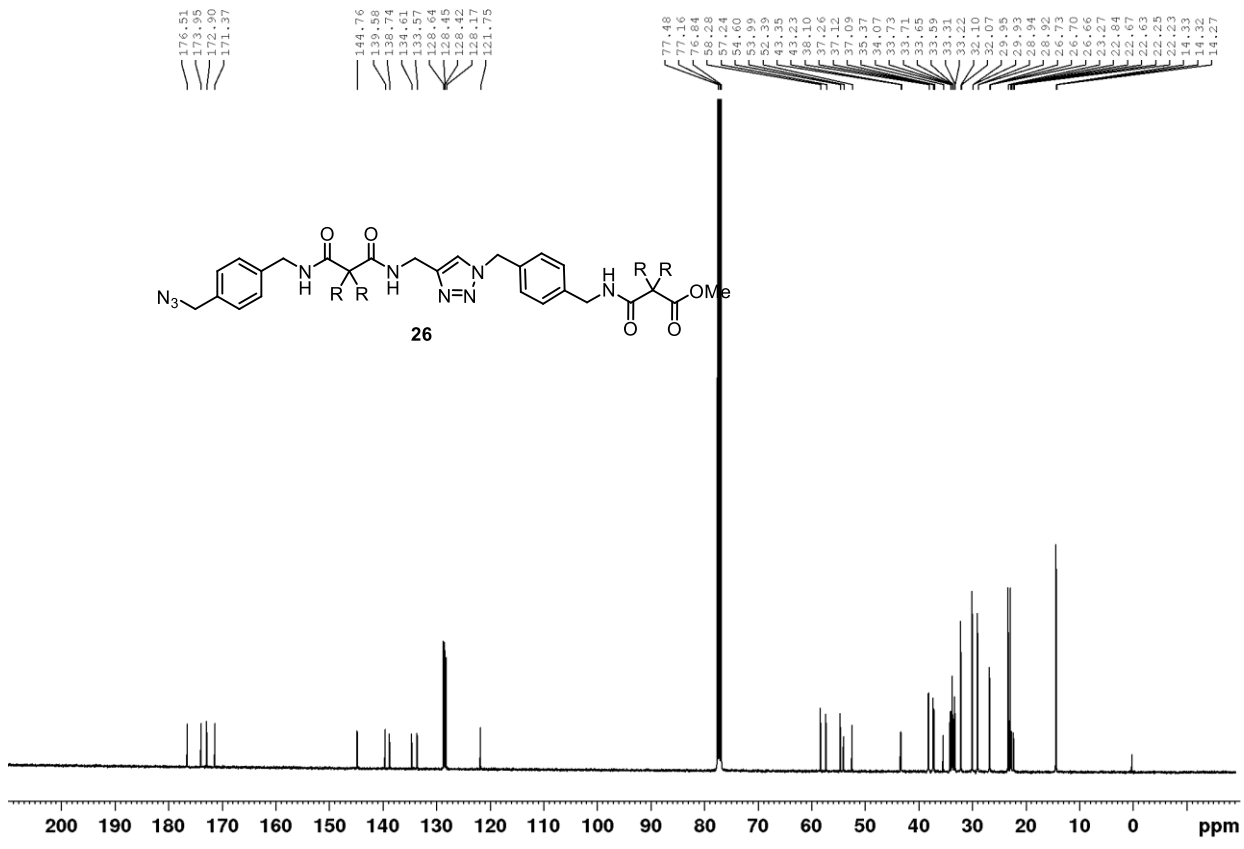
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128.42
128.17
121.75
77.48
77.16
76.84
58.28
57.44
54.60
53.99
52.39
43.35
43.23
38.10
37.26
37.12
37.09
35.37
34.07
33.73
33.71
33.65
33.59
33.31
33.22
32.10
32.07
29.95
29.93
28.94
28.92
26.73
26.70
26.66
23.27
22.84
22.67
22.63
22.25
22.23
14.33
14.32
14.27
26
200
190
180
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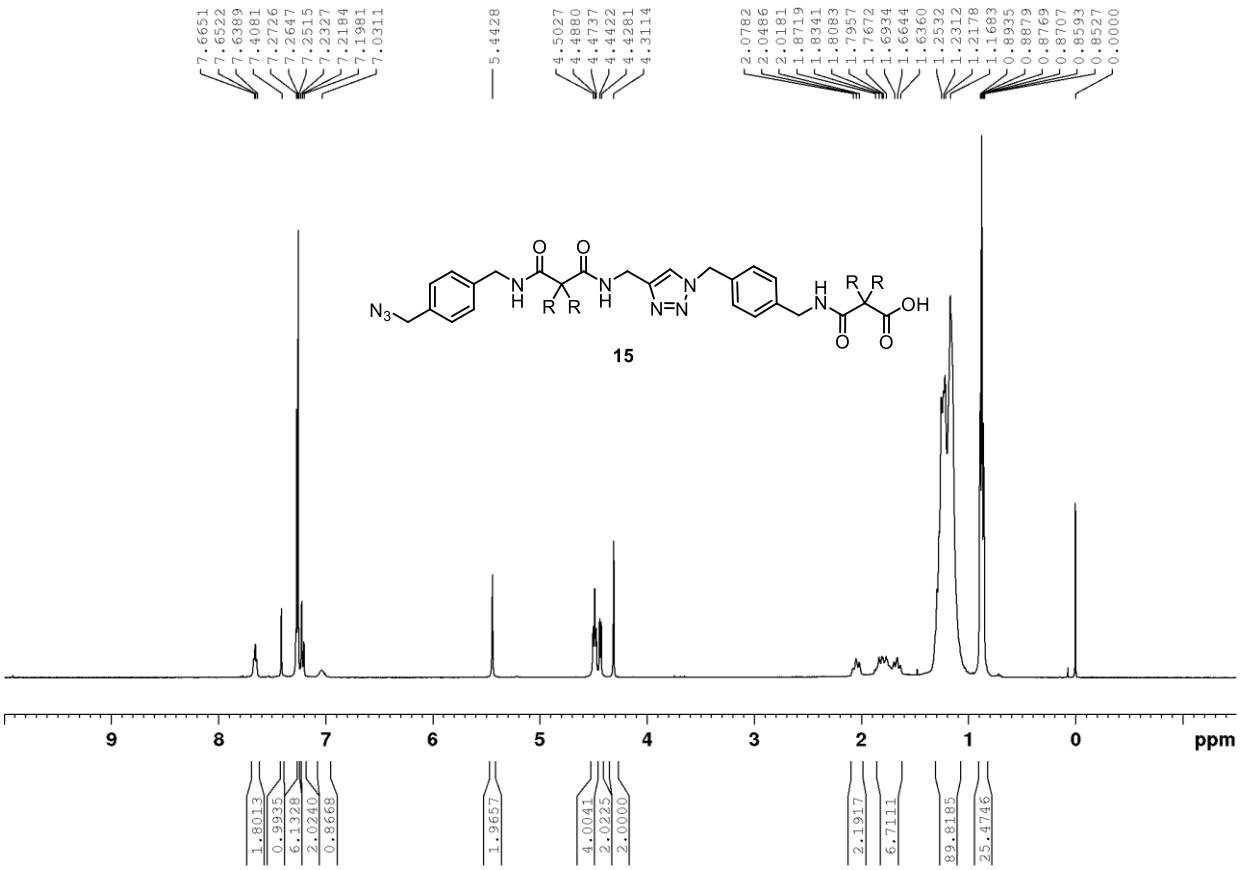
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.2647
7.2515
7.2327
7.2184
7.1981
7.0311
5.4428
4.5027
4.4880
4.4737
4.4422
4.4281
4.3114
2.0782
2.0486
2.0181
1.8719
1.8341
1.8083
1.7957
1.7672
1.6934
1.6644
1.6360
1.2532
1.2312
1.2178
1.1683
0.8935
0.8879
0.8769
0.8707
0.8593
0.8527
0.0000
15
9
8
7
6
5
4
3
2
1
0
ppm
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0.9935
6.1328
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2.1917
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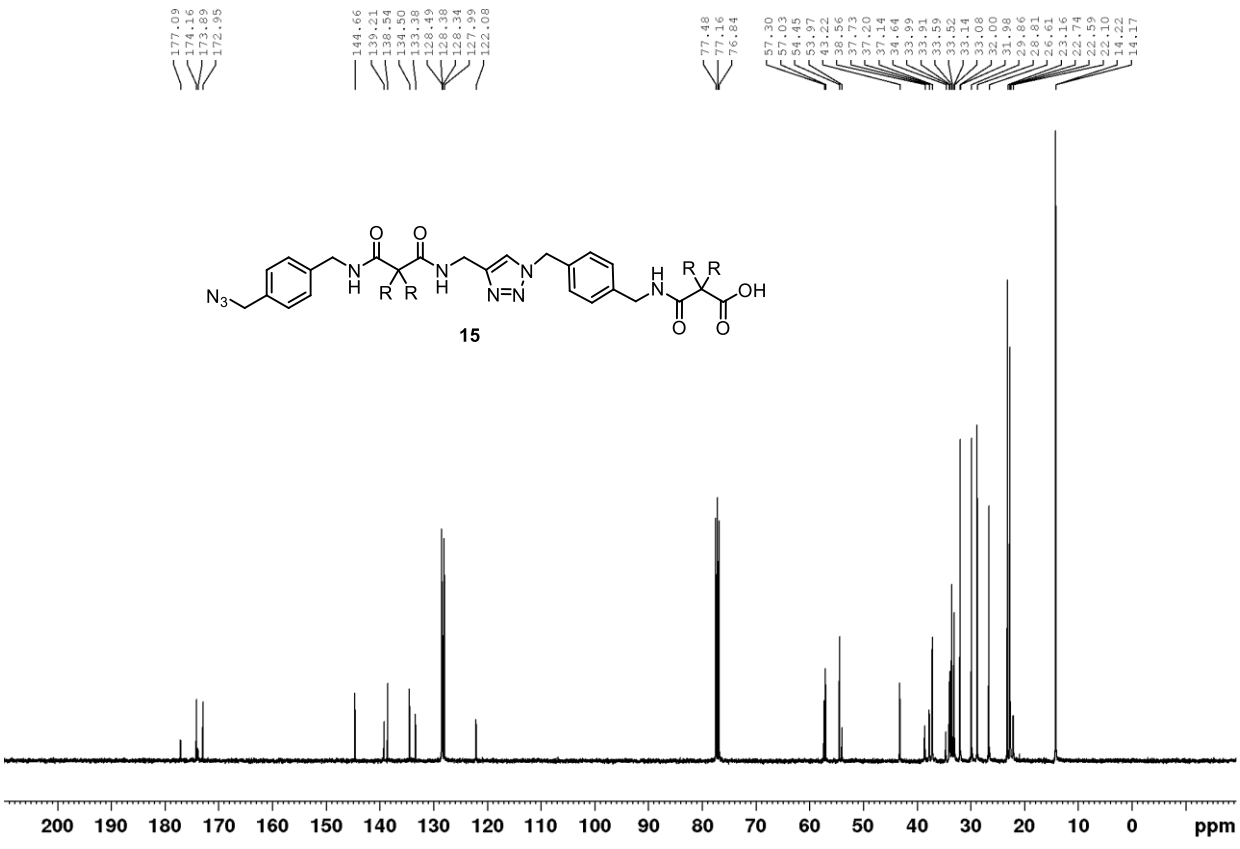
25.4746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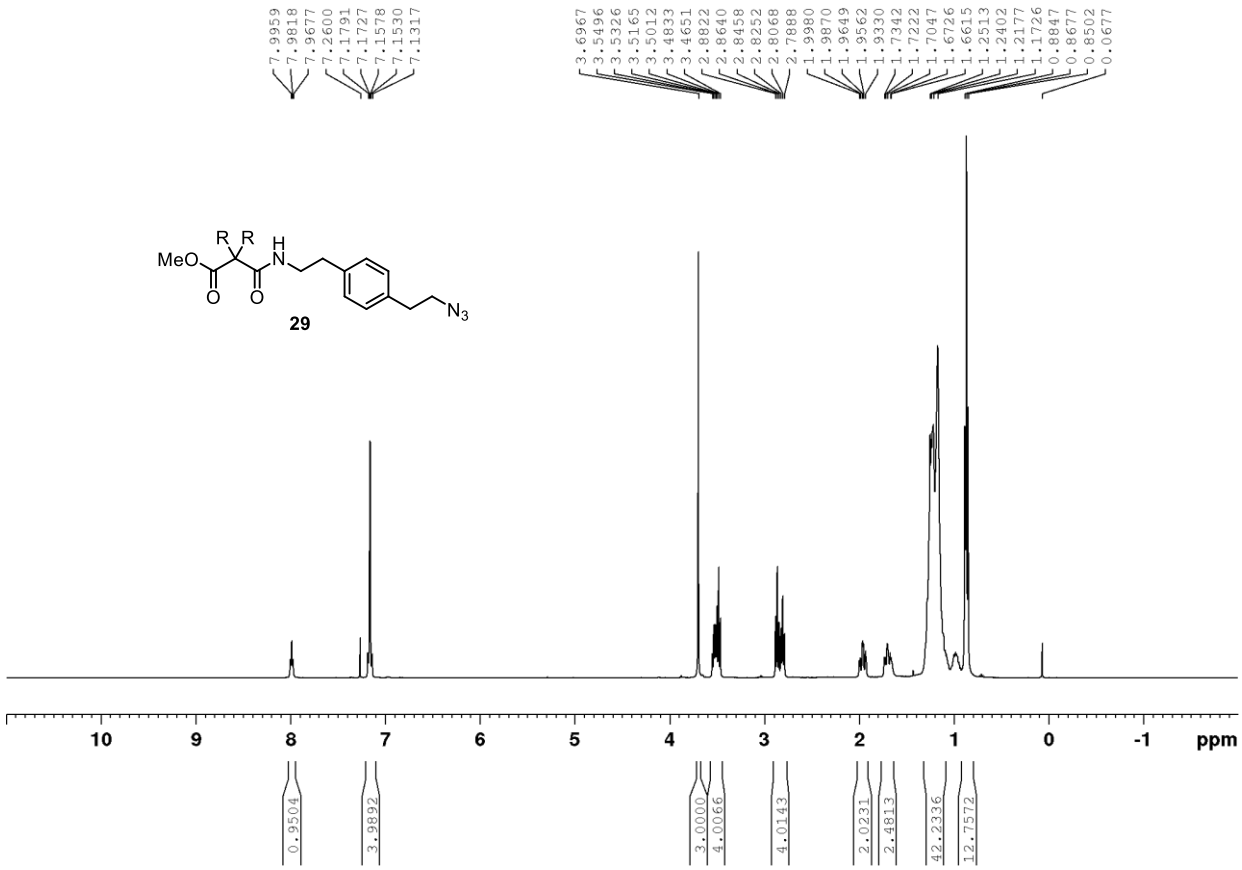
128.38
128.34
127.99
122.08
77.48
77.16
76.84
57.30
57.03
54.45
53.97
43.22
38.56
37.73
37.20
37.14
34.64
33.99
33.91
33.59
33.52
33.14
33.08
32.00
31.98
29.86
28.81
26.61
23.16
22.74
22.59
22.10
14.22
14.17
15
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.1317
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3.5326
3.5165
3.5012
3.4833
3.4651
2.8822
2.8640
2.8458
2.8252
2.8068
2.7888
1.9980
1.9870
1.9649
1.9562
1.9330
1.7342
1.7222
1.7047
1.6726
1.6615
1.2513
1.2402
1.2177
1.1726
0.8847
0.8677
0.8502
0.0677
MeO
R R
H
N
O
O
N3
29
10 9 8 7 6 5 4 3 2 1 0 -1 ppm
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3.9892
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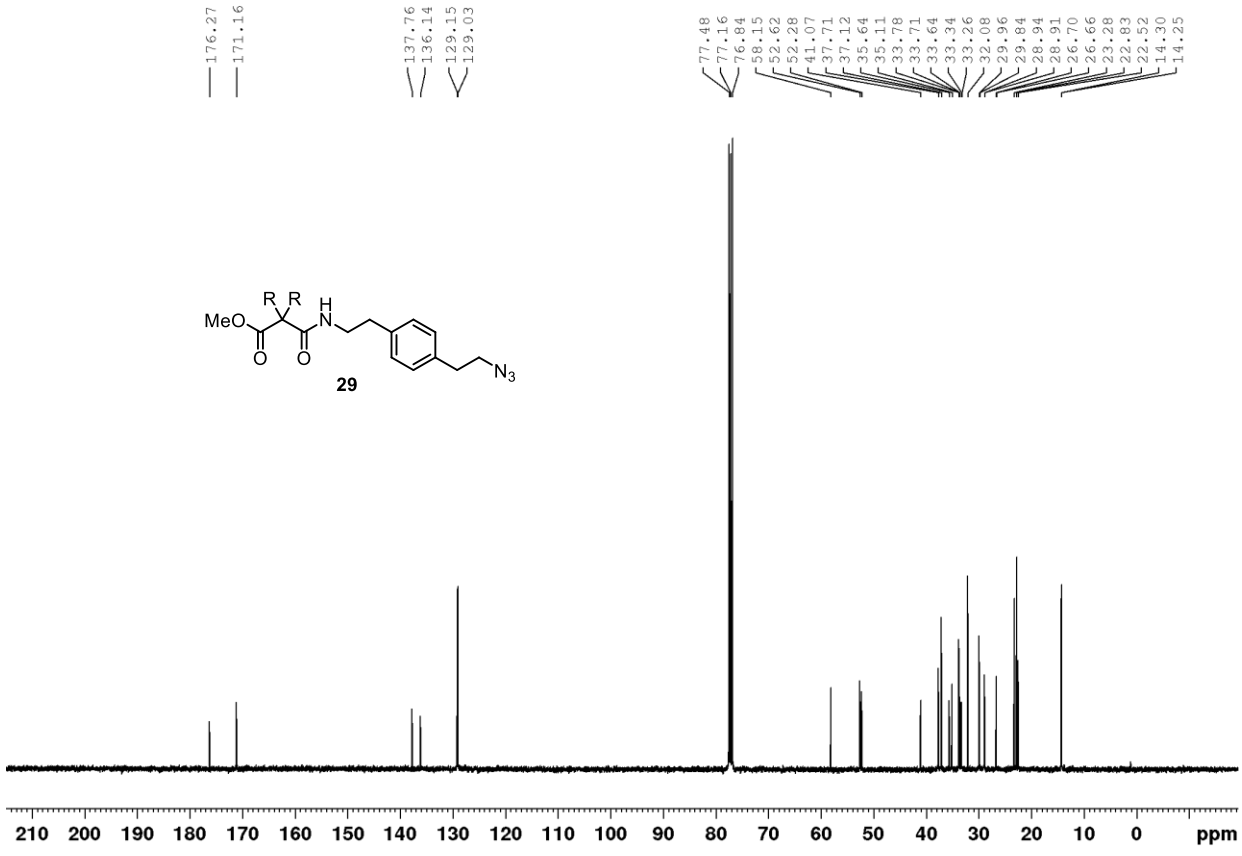
12.7572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76.84
58.15
52.62
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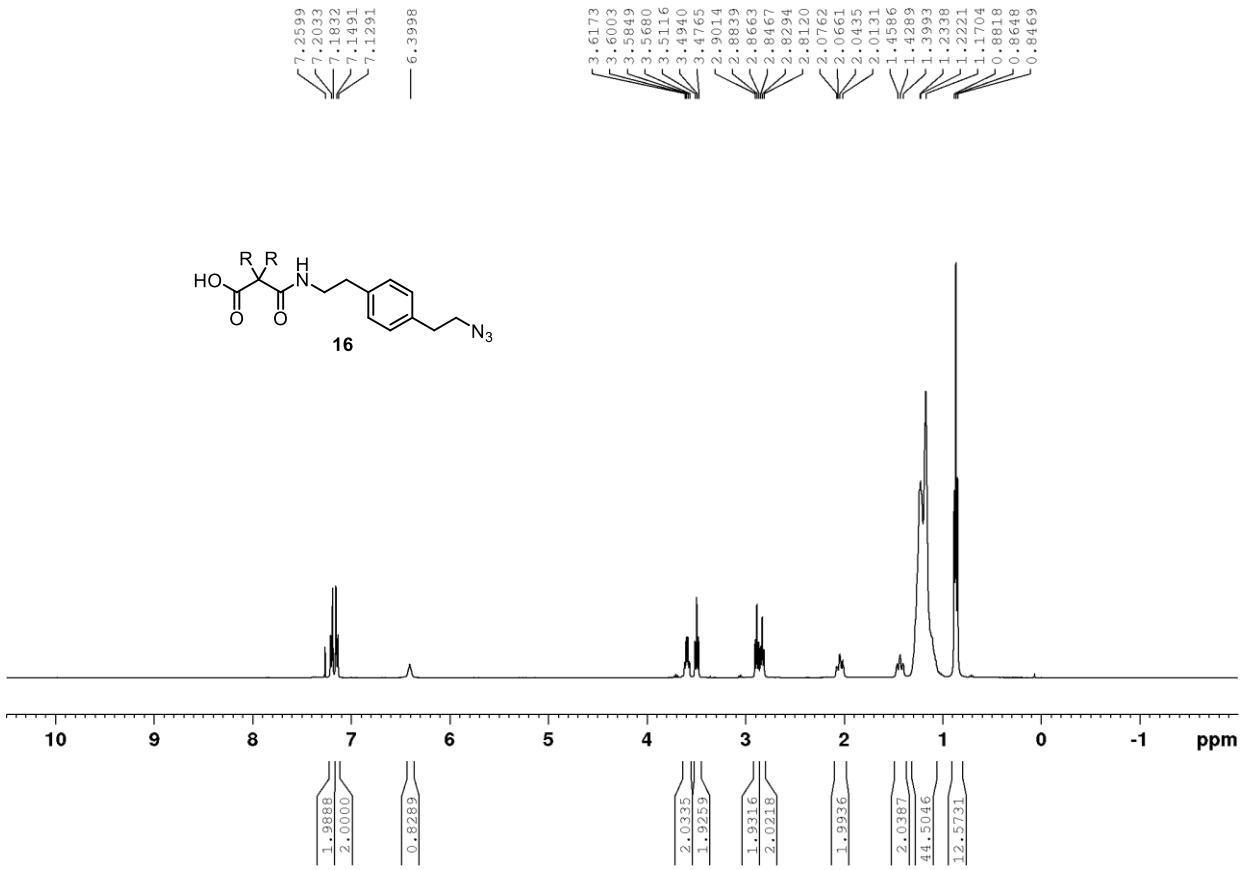
41.07
37.71
37.12
35.64
35.11
33.78
33.71
33.64
33.34
33.26
32.08
29.96
29.84
28.94
28.91
26.70
26.66
23.28
22.83
22.52
14.30
14.25
MeO
R R
H
N
O
O
N3
29
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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3.4940
3.4765
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2.8839
2.8663
2.8467
2.8294
2.8120
2.0762
2.0661
2.0435
2.0131
1.4586
1.4289
1.3993
1.2338
1.2221
1.1704
0.8818
0.8648
0.8469
HO
R
R
H
N
O
O
N3
16
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
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1.9936
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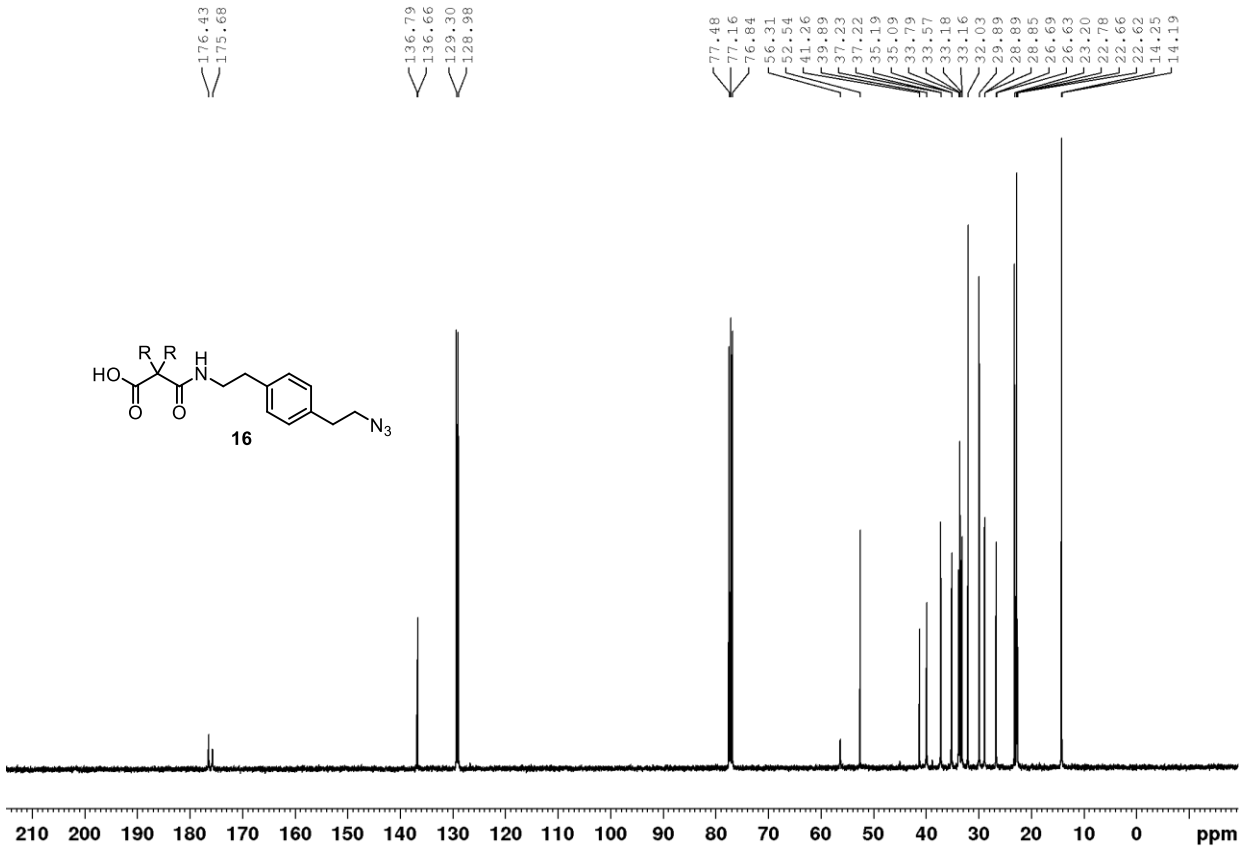
12.5731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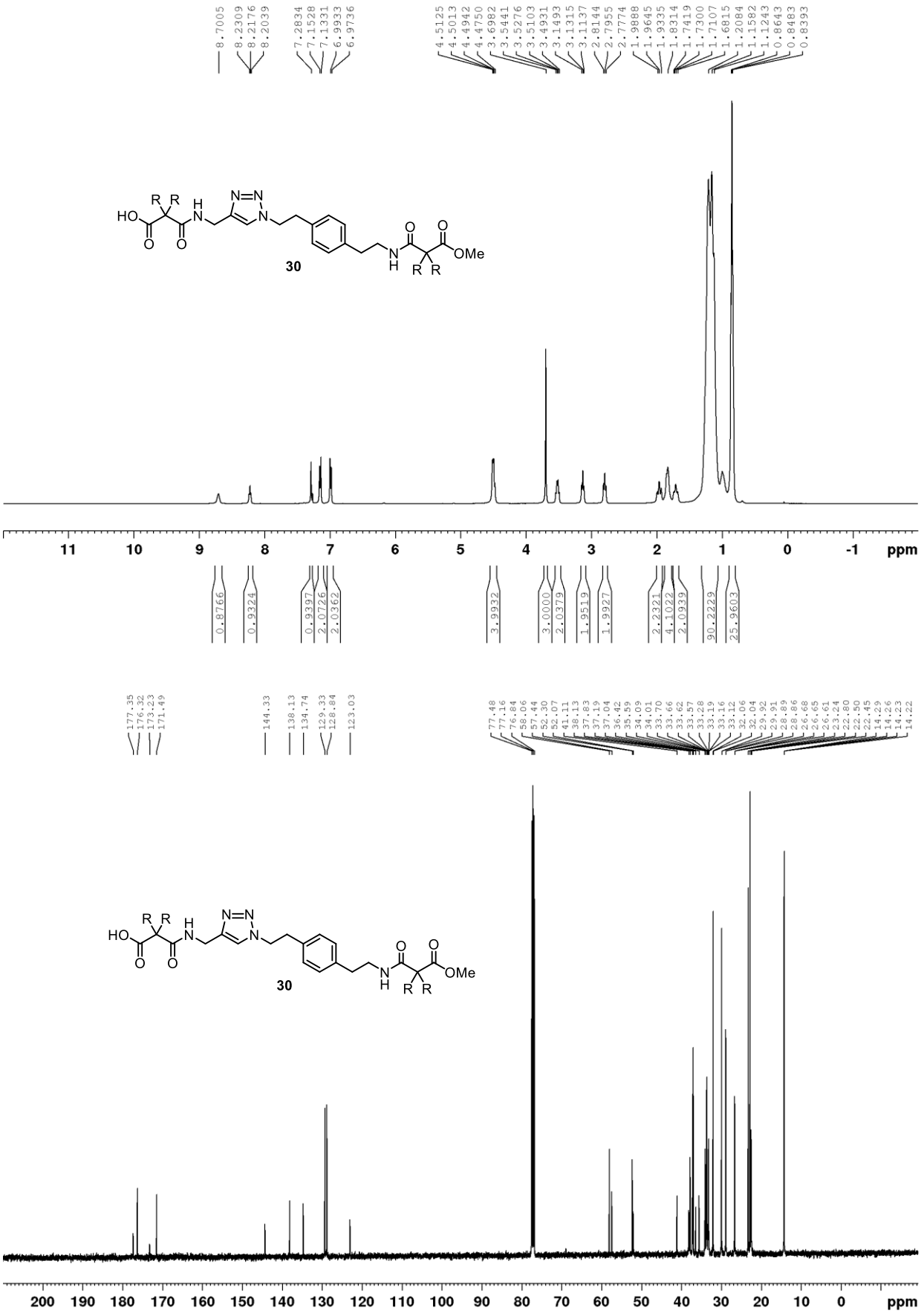
37.23
37.22
35.19
35.09
33.79
33.57
33.18
33.16
32.03
29.89
28.89
28.85
26.69
26.63
23.20
22.78
22.66
22.62
14.25
14.19
HO
R
R
H
N
O
O
N3
16
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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4.4750
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3.5276
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3.4931
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3.1315
3.1137
2.8144
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1.8314
1.7419
1.7300
1.7107
1.6815
1.2084
1.1582
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0.8643
0.8483
0.8393
30
11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm
0.8766
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0.9397
2.0726
2.0362
3.9932
3.0000
2.0379
1.9519
1.9927
2.2321
4.1022
2.0939
30.2229
25.9603
177.35
176.32
173.23
171.49
144.33
138.13
134.74
129.33
128.84
123.03
77.48
77.16
76.84
58.06
57.44
52.30
52.07
41.11
38.13
37.83
37.19
37.04
36.42
35.59
34.09
34.01
33.70
33.66
33.62
33.57
33.28
33.19
33.16
33.12
32.06
32.04
29.92
29.91
28.89
28.86
26.68
26.65
26.61
23.24
22.80
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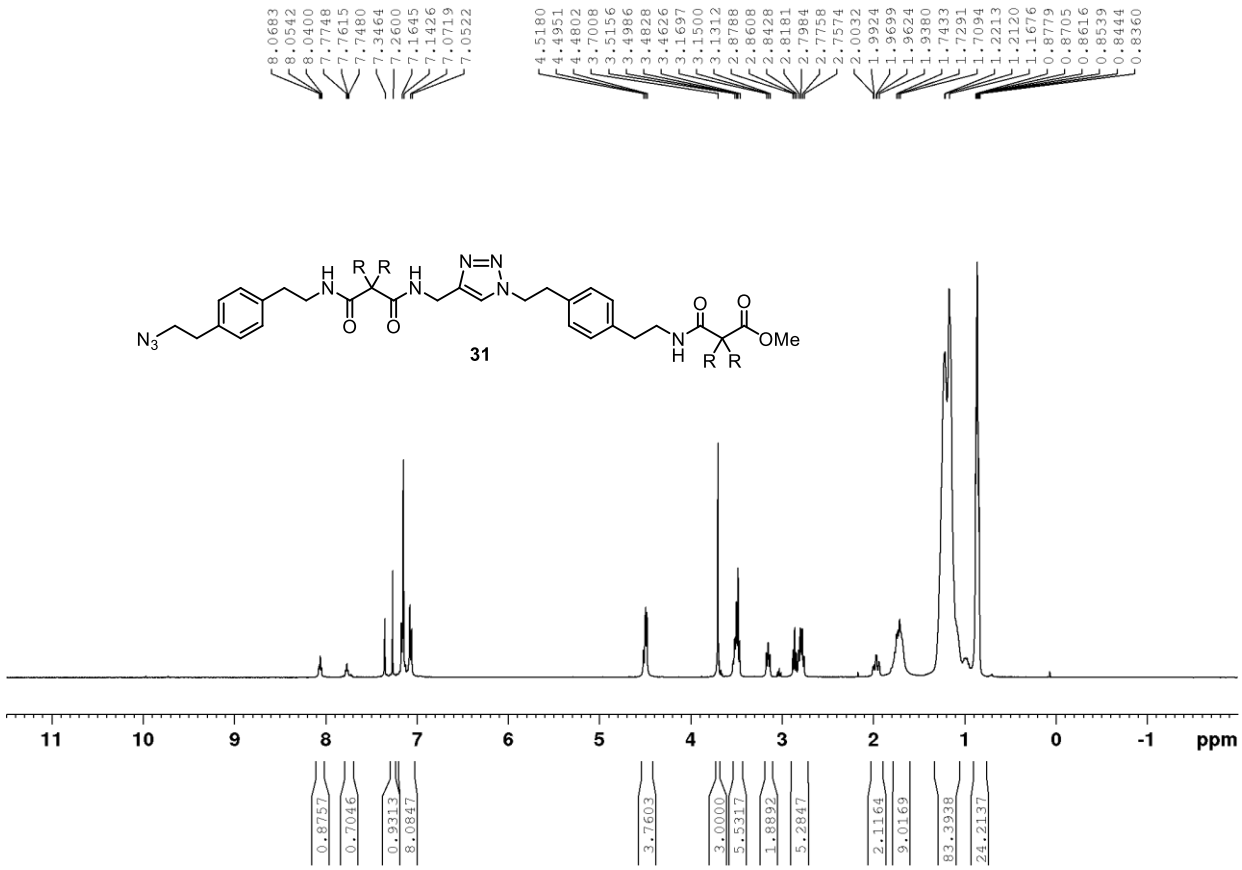
14.29
14.26
14.23
14.22
30
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.1426
7.0719
7.0522
4.5180
4.4951
4.4802
3.7008
3.5156
3.4986
3.4828
3.4626
3.1697
3.1500
3.1312
2.8788
2.8608
2.8428
2.8181
2.7984
2.7758
2.7574
2.0032
1.9924
1.9699
1.9624
1.9380
1.7433
1.7291
1.7094
1.4213
1.2120
1.1676
0.8779
0.8705
0.8616
0.8539
0.8444
0.8360
N3
R R
H N
O
O
N=N
N
H N
O
O
OMe
R R
31
11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm
0.8757
0.7046
0.9313
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3.0000
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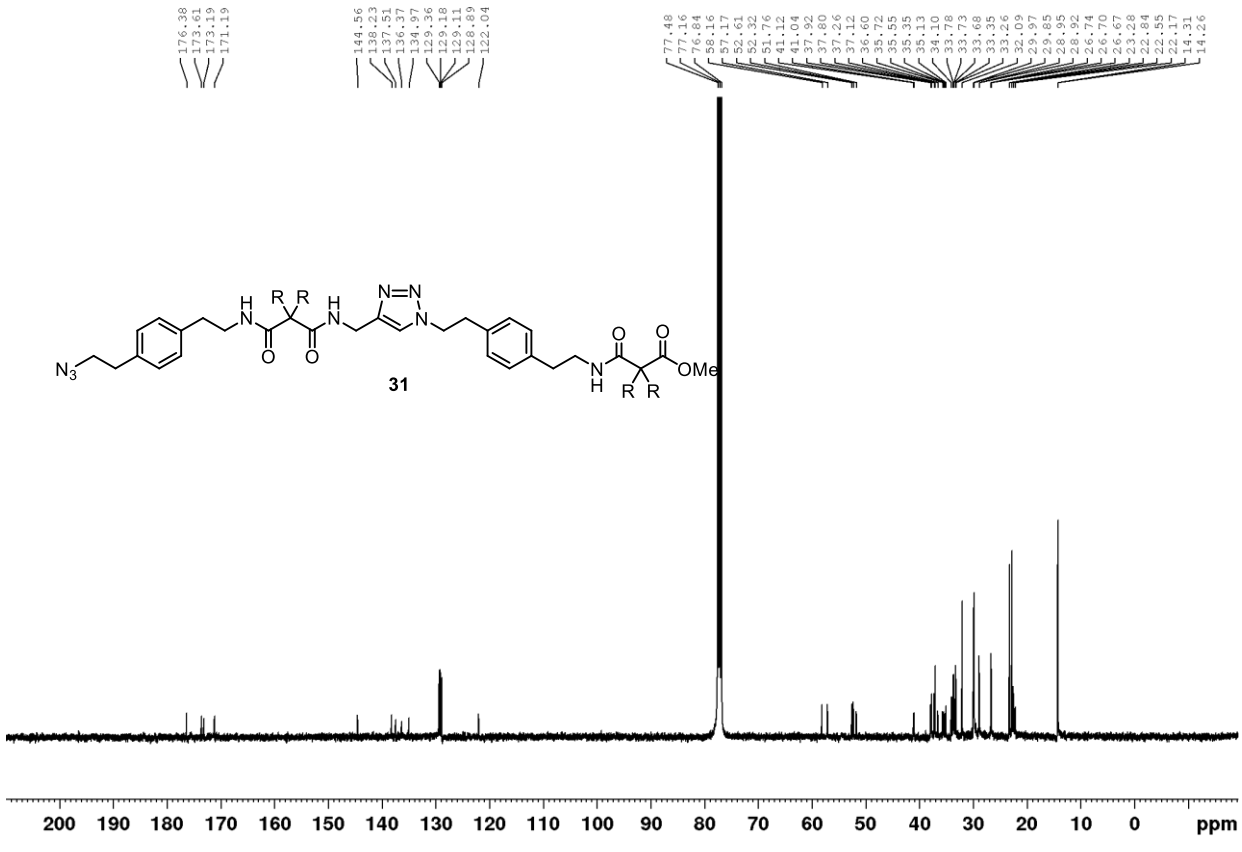
2.1164
9.0169
83.3938
24.2137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129.36
129.18
129.11
128.89
122.04
77.48
77.16
76.84
58.16
57.17
52.61
52.32
51.76
41.12
41.04
37.92
37.80
37.26
37.12
36.60
35.72
35.55
35.35
35.13
34.10
33.78
33.73
33.68
33.35
33.26
32.09
29.97
29.85
28.95
28.92
26.74
26.70
26.67
23.28
22.84
22.55
22.17
14.31
14.26
N3
R R
H N
O
O
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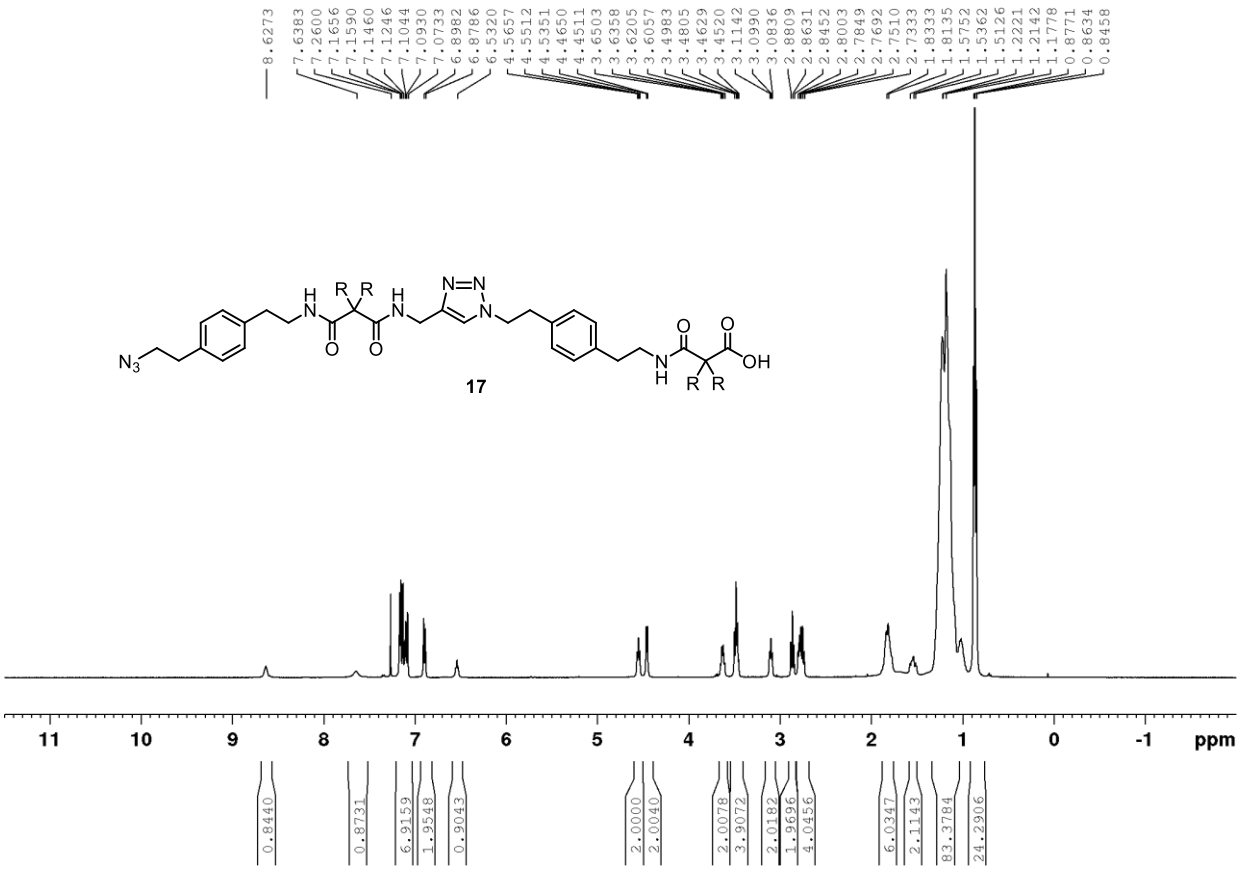
N
H N
O
O
OMe
R R
31
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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6.8786
6.5320
4.5657
4.5512
4.5351
4.4650
4.4511
3.6503
3.6358
3.6205
3.6057
3.4983
3.4805
3.4629
3.4520
3.1142
3.0990
3.0836
2.8809
2.8631
2.8452
2.8003
2.7849
2.7692
2.7510
2.7333
1.8333
1.8135
1.5752
1.5362
1.5126
1.2221
1.2142
1.1778
0.8771
0.8634
0.8458
17
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
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0.8731
6.9159
1.9548
0.9043
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6.0347
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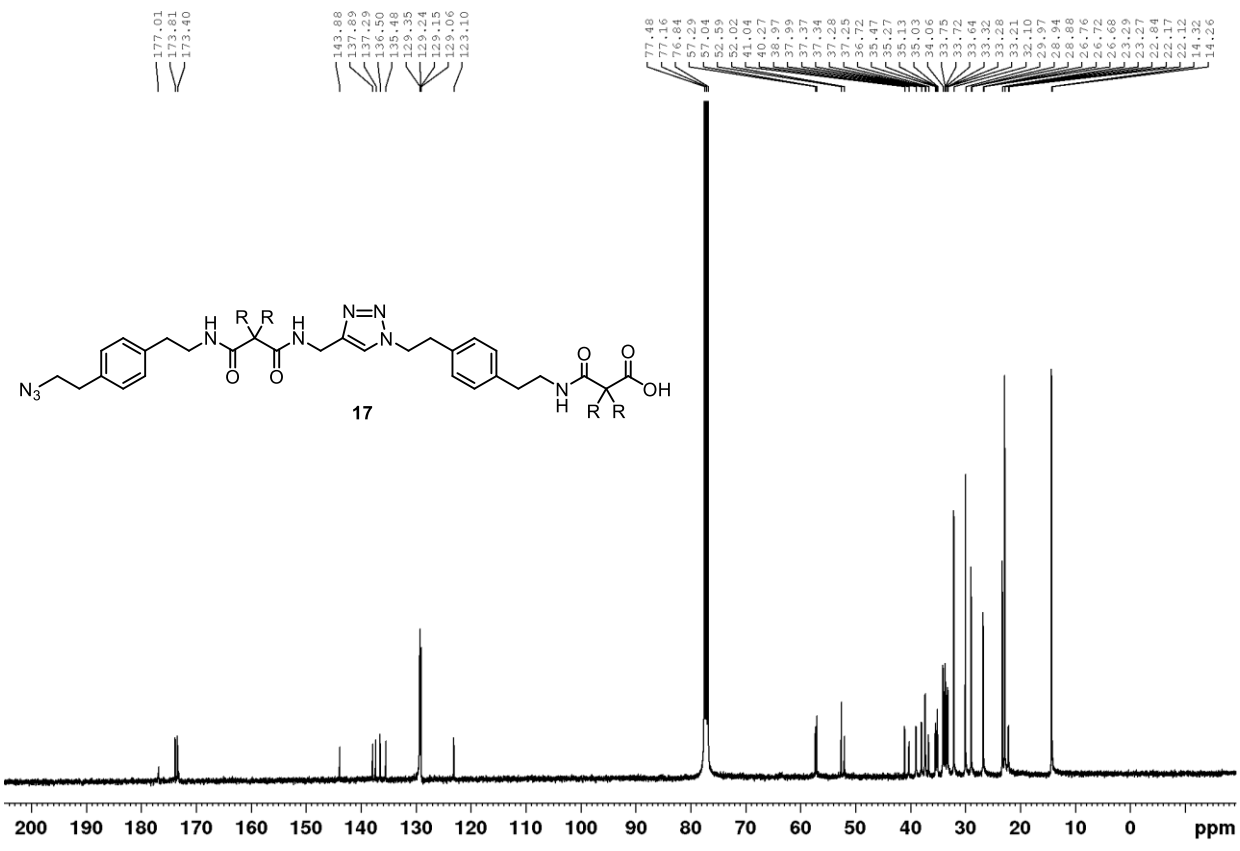
24.2906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129.15
129.06
123.10
77.48
77.16
76.84
57.29
57.04
52.59
52.02
41.04
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37.37
37.34
37.28
37.25
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35.27
35.13
35.03
34.06
33.75
33.72
33.64
33.32
33.28
33.21
32.10
29.97
28.94
28.88
26.76
26.72
26.68
23.29
23.27
22.84
22.17
22.12
14.32
14.26
17
200
190
180
170
160
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140
130
120
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100
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60
50
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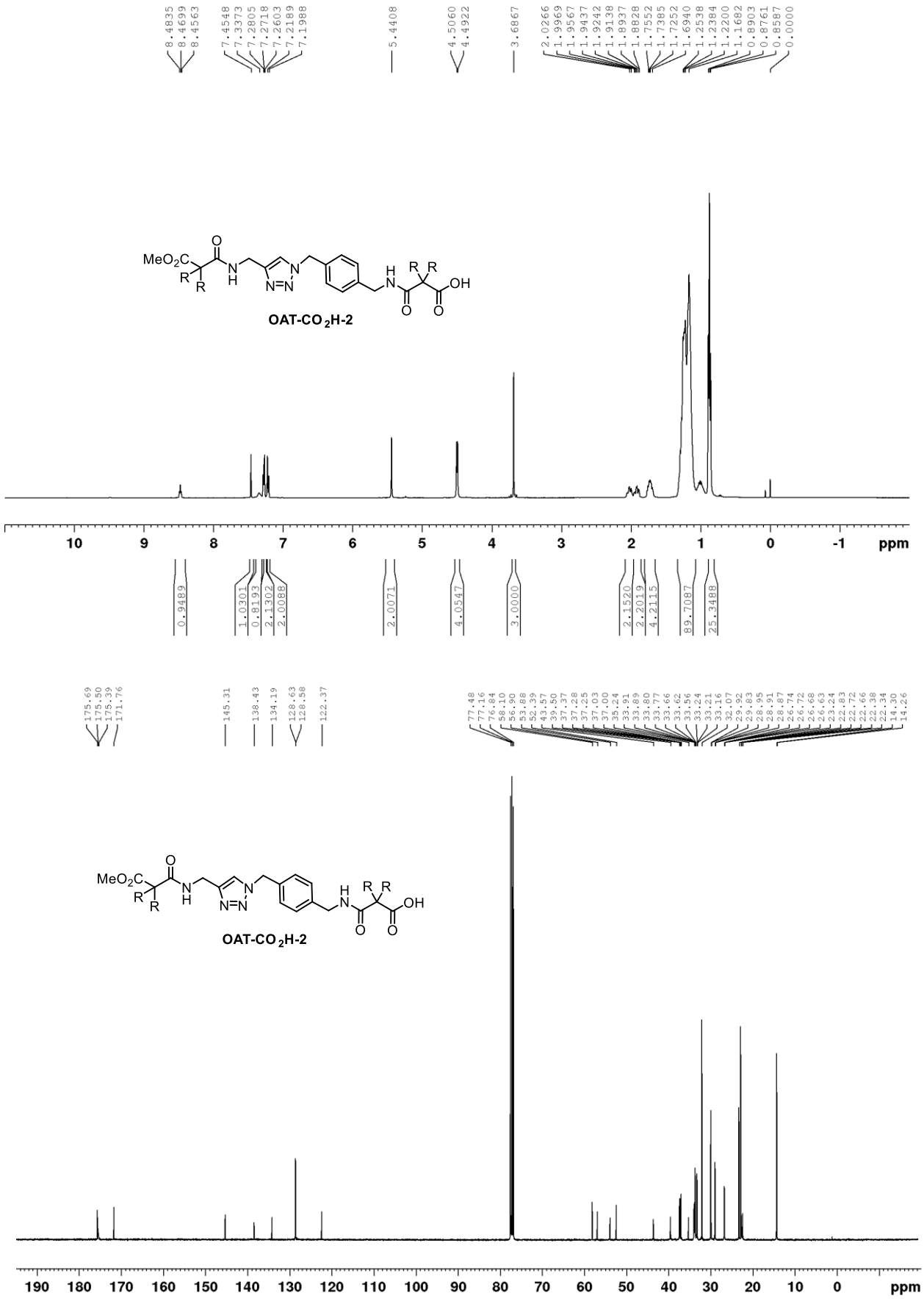
20
10
0
ppm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5.4408
4.5060
4.4922
3.6867
2.0266
1.9969
1.9567
1.9437
1.9242
1.9138
1.8937
1.8828
1.7552
1.7385
1.7252
1.6940
1.2538
1.2384
1.2200
1.1682
0.8903
0.8761
0.8587
0.0000
OAT-CO2H-2
10 9 8 7 6 5 4 3 2 1 0 -1 ppm
0.9489
1.0301
0.8193
2.1302
2.0088
2.0071
4.0547
3.0000
2.1520
2.2019
4.2115
89.7087
25.3488
175.69
175.50
175.39
171.76
145.31
138.43
134.19
128.63
128.58
122.37
77.48
77.16
76.84
58.10
56.90
53.88
52.39
43.57
39.50
37.37
37.28
37.25
37.03
37.00
35.24
33.91
33.89
33.80
33.77
33.66
33.62
33.56
33.24
33.21
33.16
32.07
29.92
29.83
28.95
28.91
28.87
26.74
26.72
26.68
26.63
23.24
22.83
22.72
22.66
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22.34
14.30
14.26
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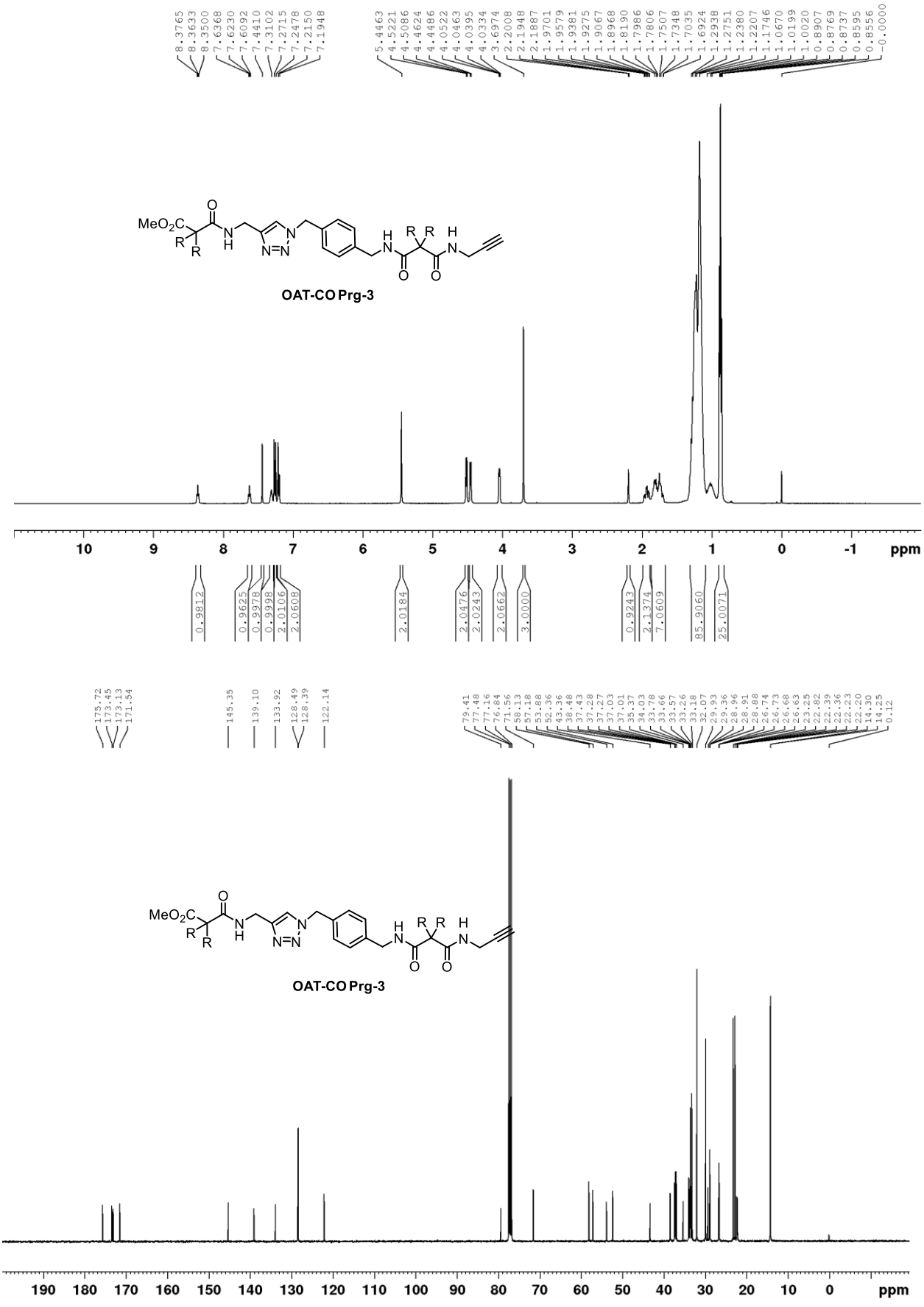
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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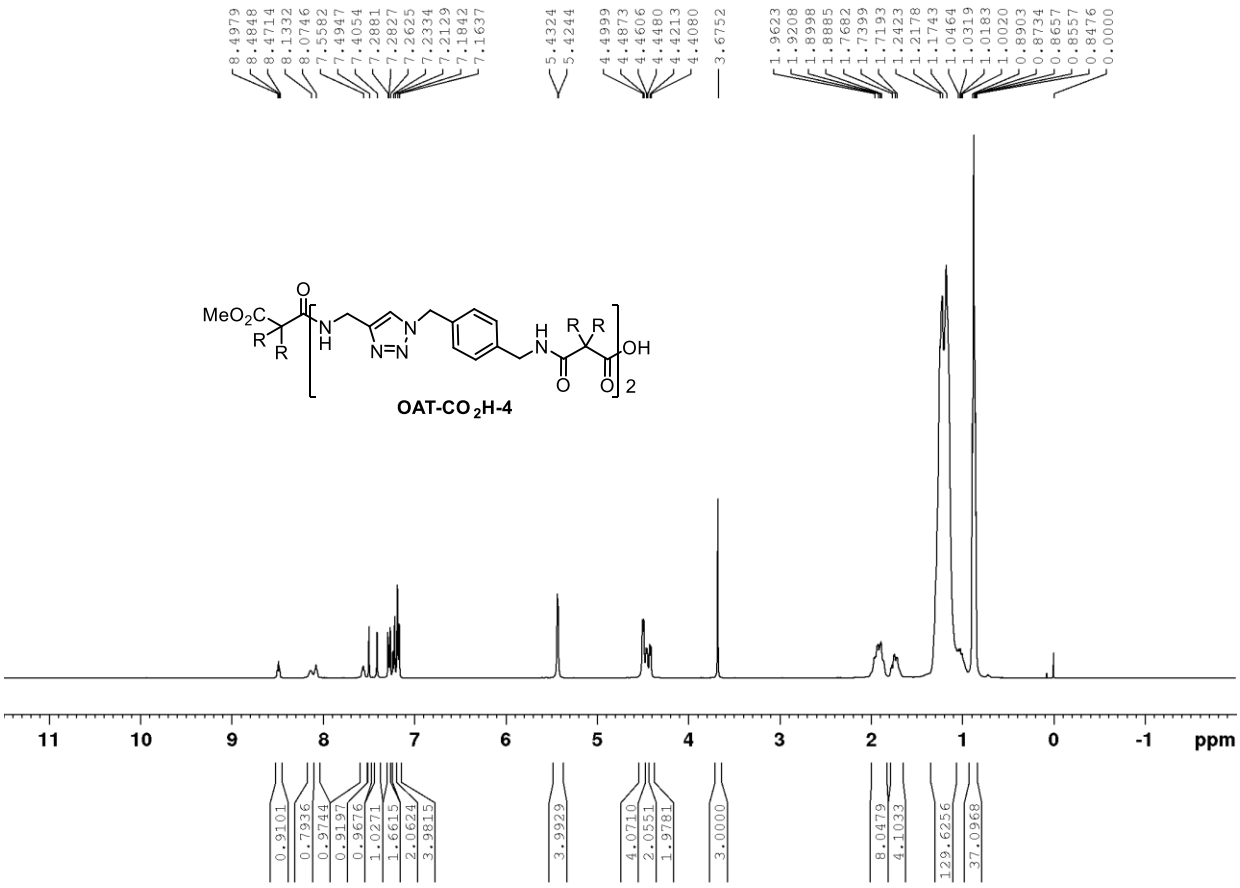
7.2150
7.1948
5.4463
4.5221
4.5086
4.4624
4.4486
4.0522
4.0463
4.0395
4.0334
3.6974
2.2008
2.1948
2.1887
1.9701
1.9579
1.9381
1.9275
1.9067
1.8968
1.8190
1.7986
1.7806
1.7507
1.7348
1.7035
1.6924
1.2938
1.2751
1.2380
1.2207
1.1746
1.0670
1.0199
1.0020
0.8907
0.8769
0.8737
0.8595
0.8556
0.0000
OAT-CO Prg-3
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
0.9812
0.9625
0.9978
0.9998
2.0106
2.0608
2.0184
2.0476
2.0243
2.0662
3.0000
0.9243
2.1374
7.0609
85.9060
25.0071
175.72
173.45
173.13
171.54
145.35
139.10
133.92
128.49
128.39
122.14
79.41
77.48
77.16
76.84
76.56
58.13
57.88
53.88
52.36
43.36
38.48
37.43
37.28
37.27
37.03
37.01
35.37
34.03
33.78
33.66
33.57
33.26
33.18
32.07
29.93
29.36
28.96
28.91
28.88
26.74
26.73
26.68
26.63
23.25
22.92
22.39
22.36
22.23
22.20
14.30
14.25
0.12
OAT-CO Prg-3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.4947
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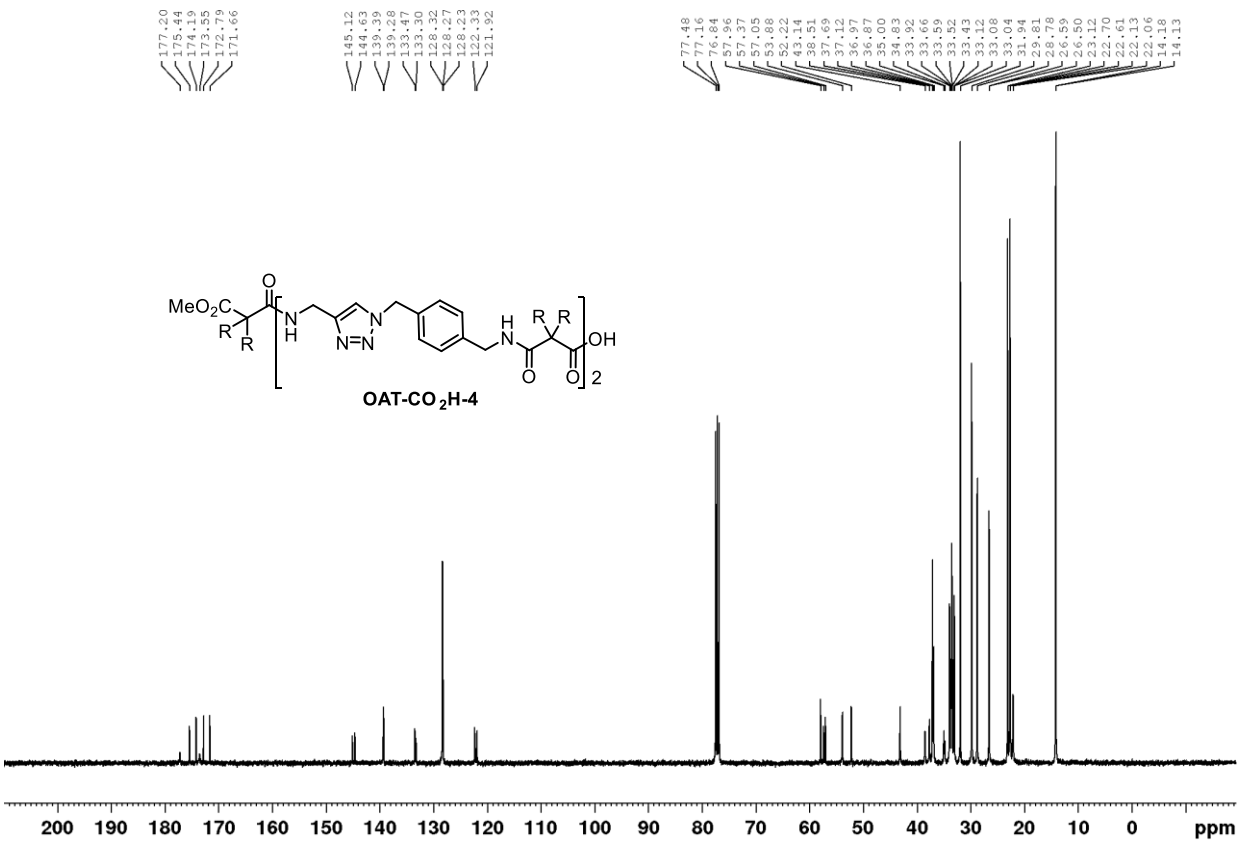
7.2625
7.2334
7.2129
7.1842
7.1637
5.4324
5.4244
4.4999
4.4873
4.4606
4.4480
4.4213
4.4080
3.6752
1.9623
1.9208
1.8998
1.8885
1.7682
1.7399
1.7193
1.2423
1.2178
1.1743
1.0464
1.0319
1.0183
1.0020
0.8903
0.8734
0.8657
0.8557
0.8476
0.0000
OAT-CO2H-4
0.9101
0.7936
0.9744
0.9197
0.9676
1.0271
1.6615
2.0624
3.9815
3.9929
4.0710
2.0551
1.9781
3.0000
8.0479
4.1033
129.6256
37.0968
ppm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174.19
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171.66
145.12
144.63
139.39
139.28
133.47
133.30
128.32
128.27
128.23
122.33
121.92
77.48
77.16
76.84
57.96
57.37
57.05
53.88
52.22
43.14
38.51
37.69
37.12
36.97
36.87
35.00
34.83
33.92
33.66
33.59
33.52
33.43
33.12
33.08
33.04
31.94
29.81
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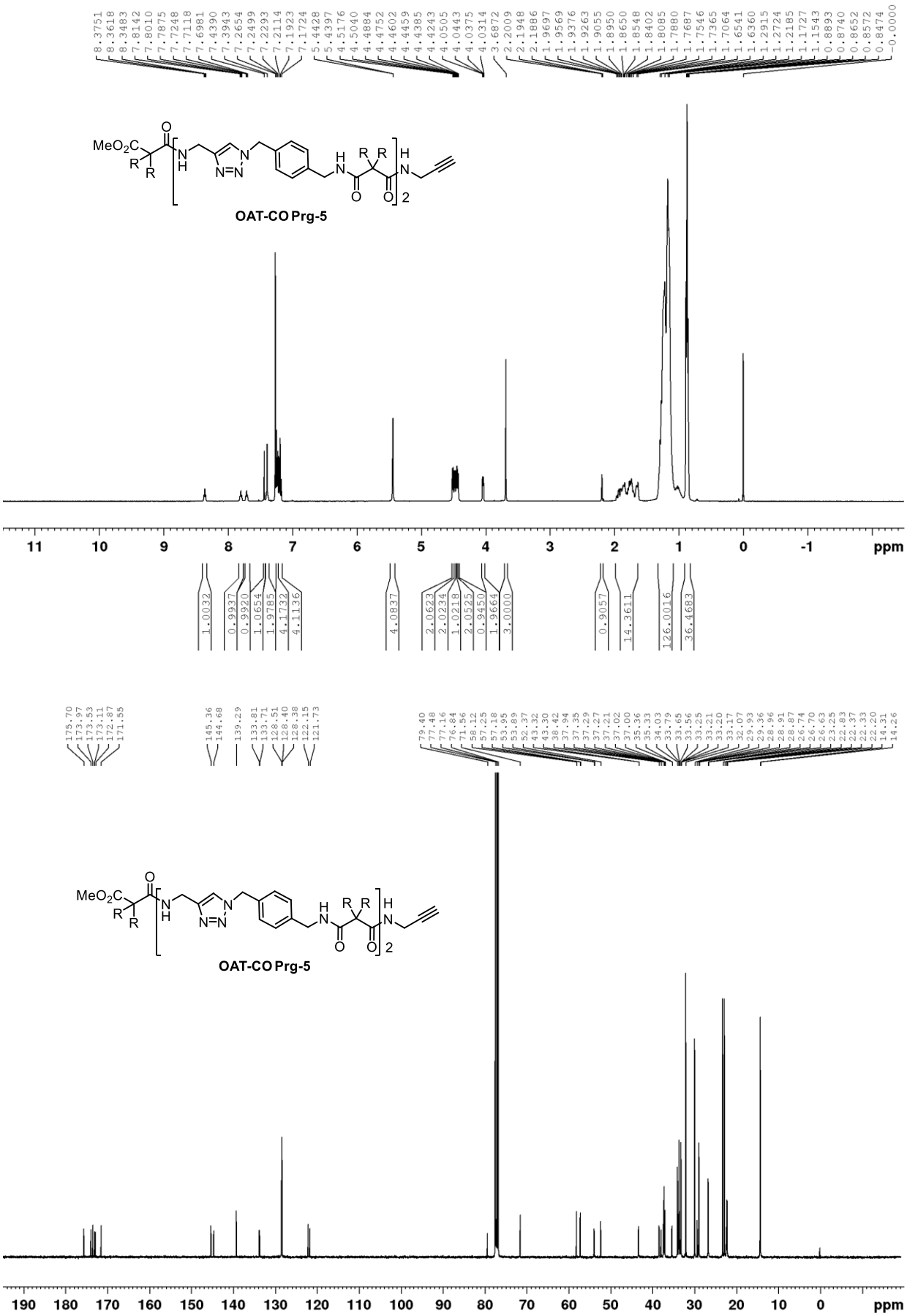
26.50
23.12
22.70
22.61
22.13
22.06
14.18
14.13
OAT-CO2H-4
ppm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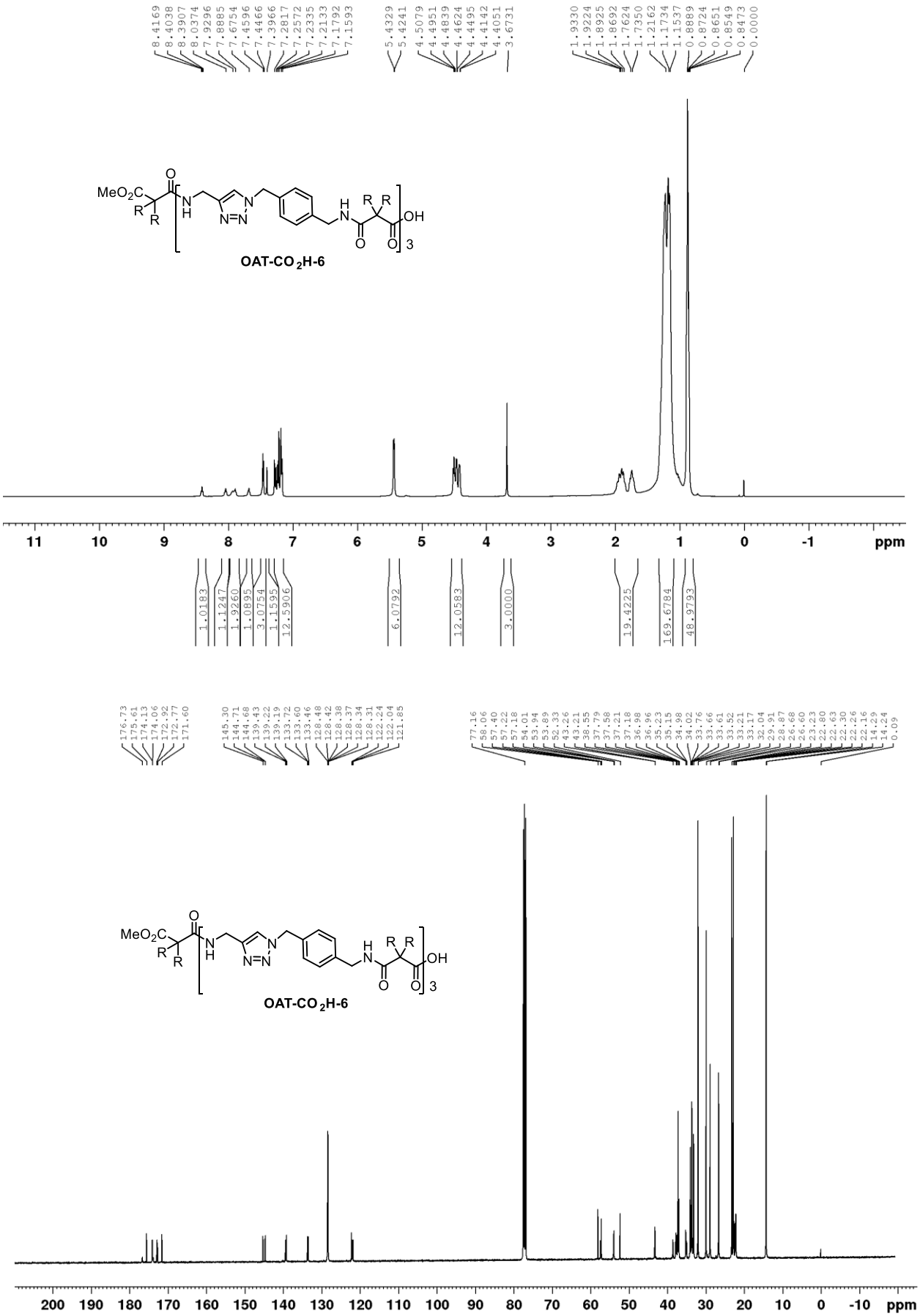
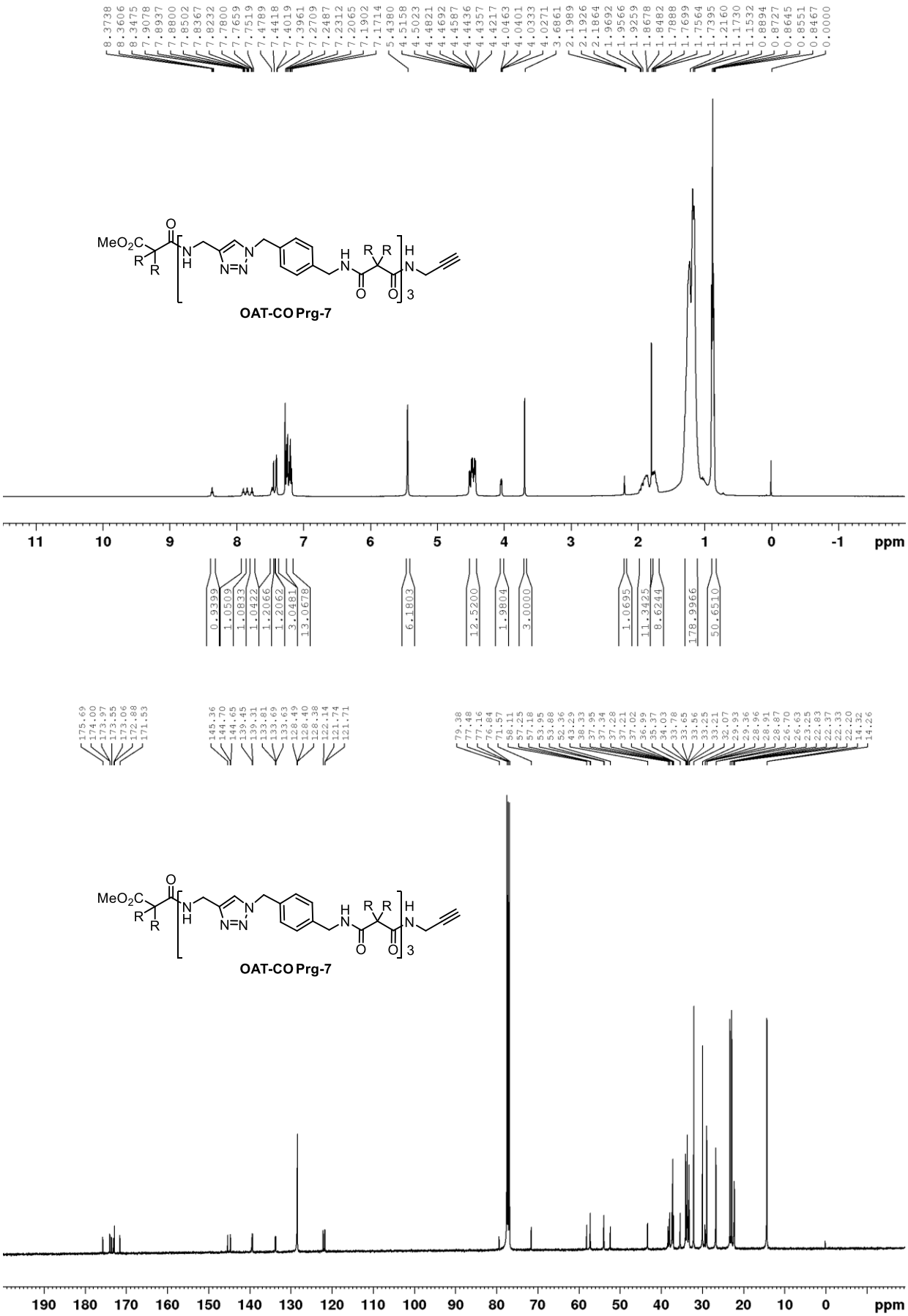
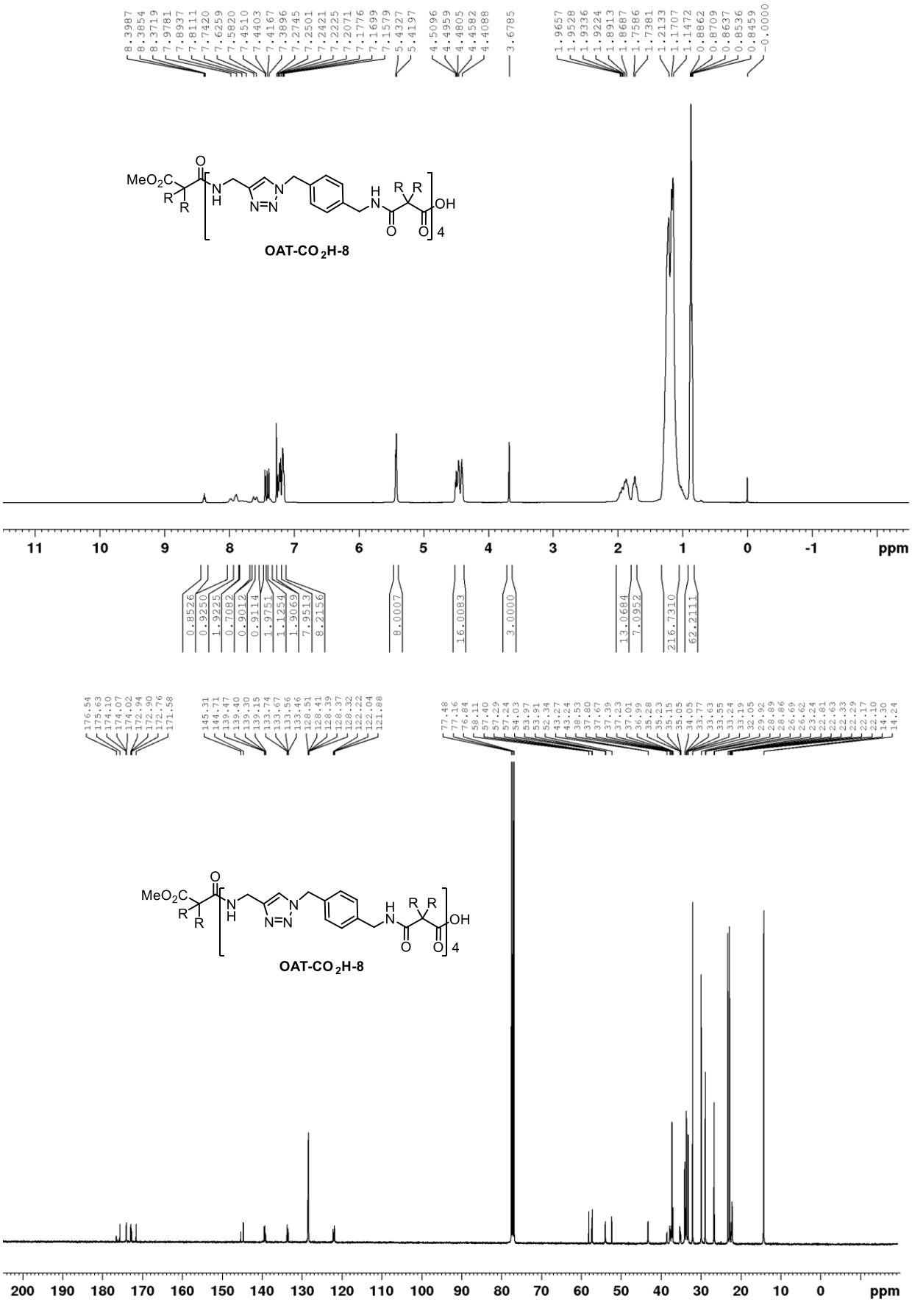
ppm
OAT-CO Prg-5
ppm


OAT-CO2H-6
OAT-CO2H-6
ppm
ppm


OAT-CO Prg-7
ppm
OAT-CO Prg-7
ppm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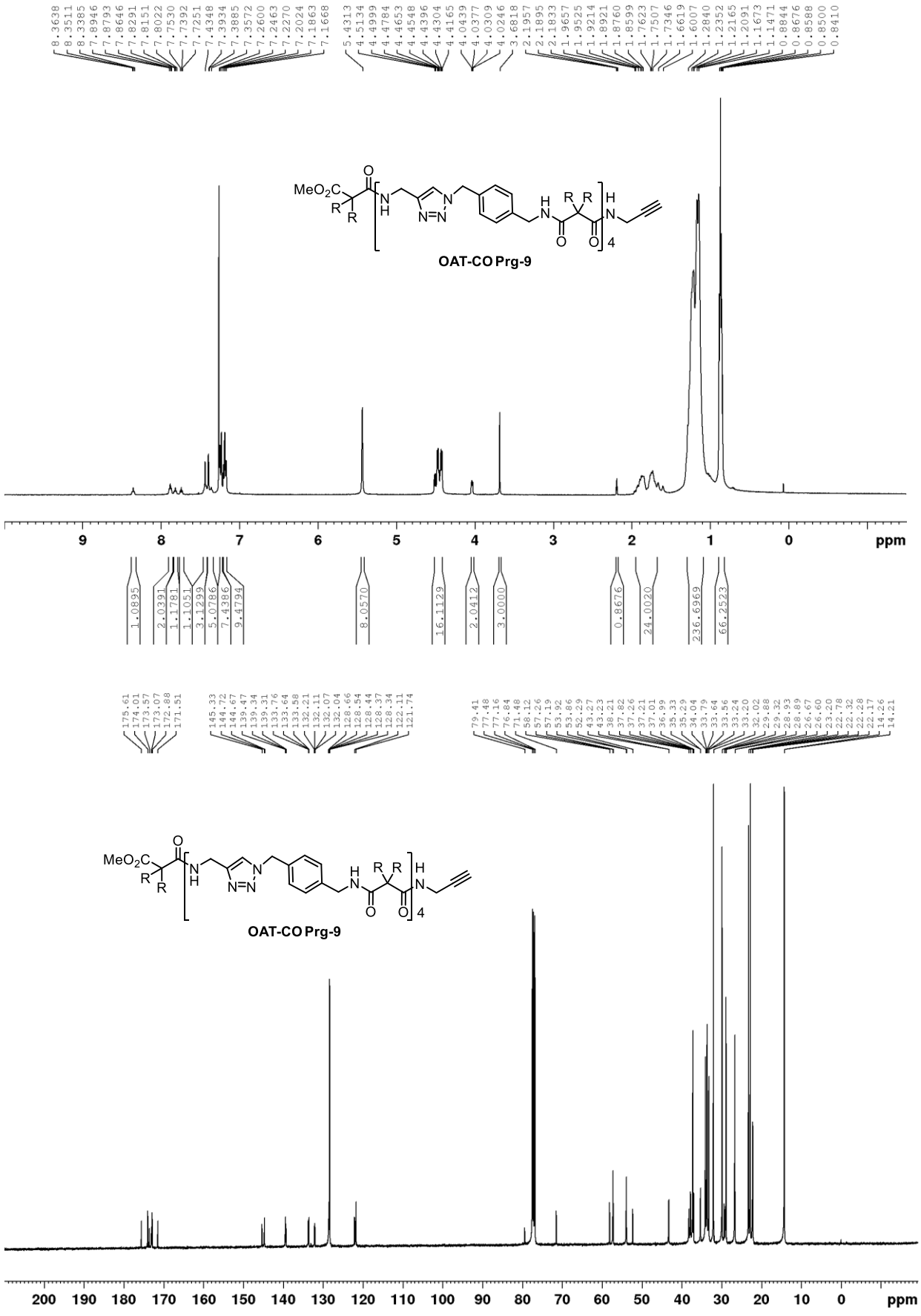
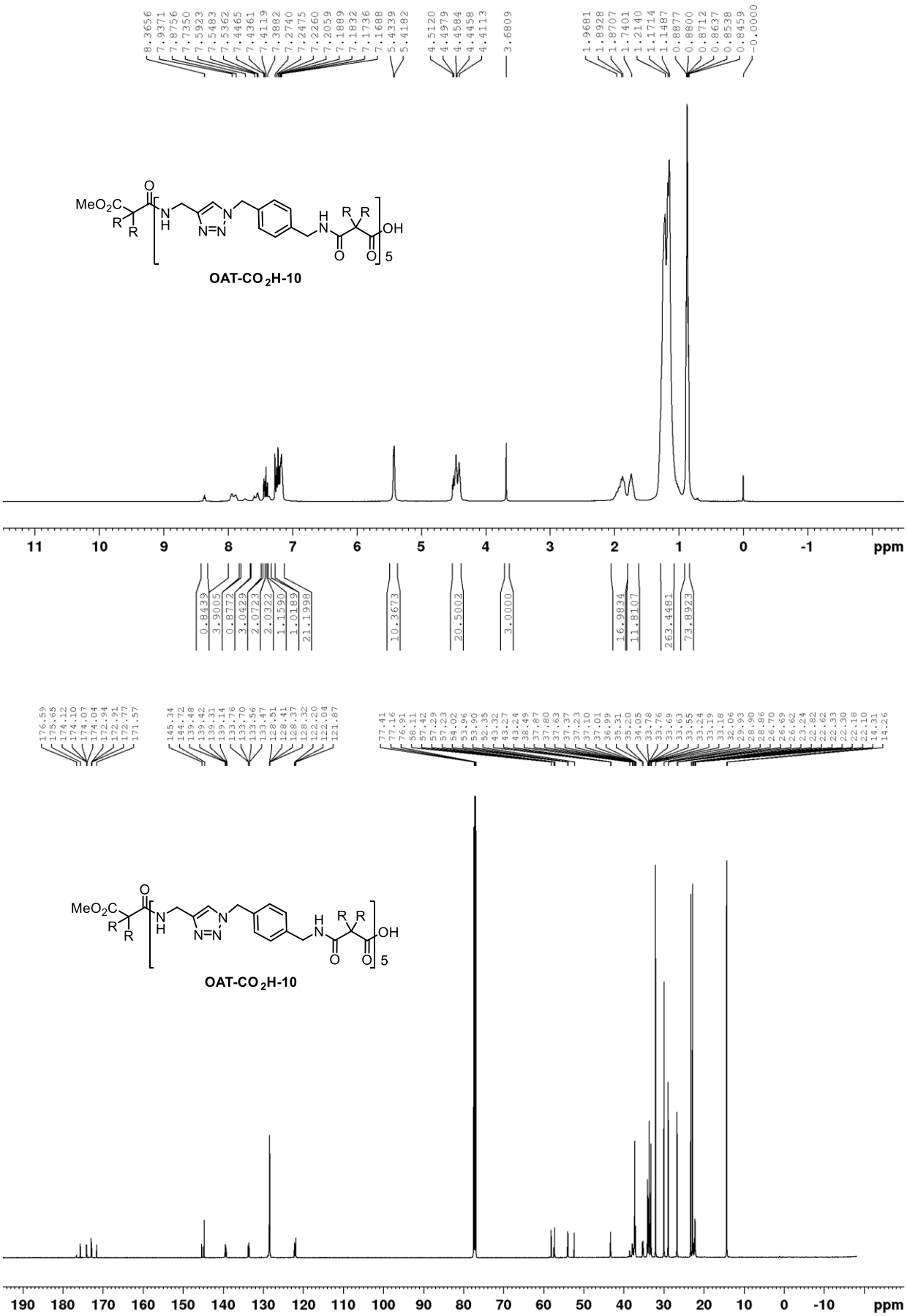
7.4403
7.4167
7.3896
7.2745
7.2501
7.2421
7.2225
7.2071
7.1776
7.1699
7.1579
5.4327
5.4197
4.5096
4.4959
4.4805
4.4582
4.4088
3.6785
1.9657
1.9528
1.9336
1.9224
1.8913
1.8687
1.7586
1.7381
1.2133
1.1707
1.1472
0.8862
0.8709
0.8637
0.8536
0.8459
-0.0000
OAT-CO2H-8
11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm
0.8526
0.9250
1.9225
0.7082
0.9012
0.9114
1.9751
1.1254
1.9069
7.9513
8.2156
8.0007
16.0083
3.0000
13.0684
7.0952
216.7310
62.2111
176.54
175.63
174.10
174.07
174.02
172.94
172.90
172.76
171.58
145.31
144.71
139.47
139.40
139.30
139.15
133.74
133.67
133.56
133.46
128.51
128.41
128.39
128.37
128.32
122.22
122.04
121.88
77.48
77.16
76.84
58.11
57.40
57.29
57.24
54.03
53.97
53.91
52.34
43.27
43.24
38.53
37.80
37.67
37.39
37.23
37.01
36.99
35.28
35.23
35.15
35.05
34.05
33.77
33.63
33.55
33.24
33.19
32.05
29.92
28.89
28.86
26.69
26.62
23.24
22.81
22.63
22.33
22.29
22.17
22.10
14.30
14.24
OAT-CO2H-8
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


OAT-CO Prg-9
ppm
OAT-CO Prg-9
ppm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7.4361
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7.2740
7.2475
7.2260
7.2059
7.1889
7.1832
7.1736
7.1688
5.4339
5.4182
4.5120
4.4979
4.4584
4.4458
4.4113
3.6809
1.9681
1.8928
1.8707
1.7401
1.2140
1.1714
1.1487
0.8877
0.8800
0.8712
0.8637
0.8538
0.8459
-0.0000
MeO2C
R
R
N
H
O
N
N=N
H
N
R
R
OH
O
O
5
OAT-CO2H-10
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
0.8439
3.9005
0.8772
3.0429
2.0723
2.0322
1.1590
1.0189
21.1998
10.3673
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3.0000
16.9834
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263.4481
73.8923
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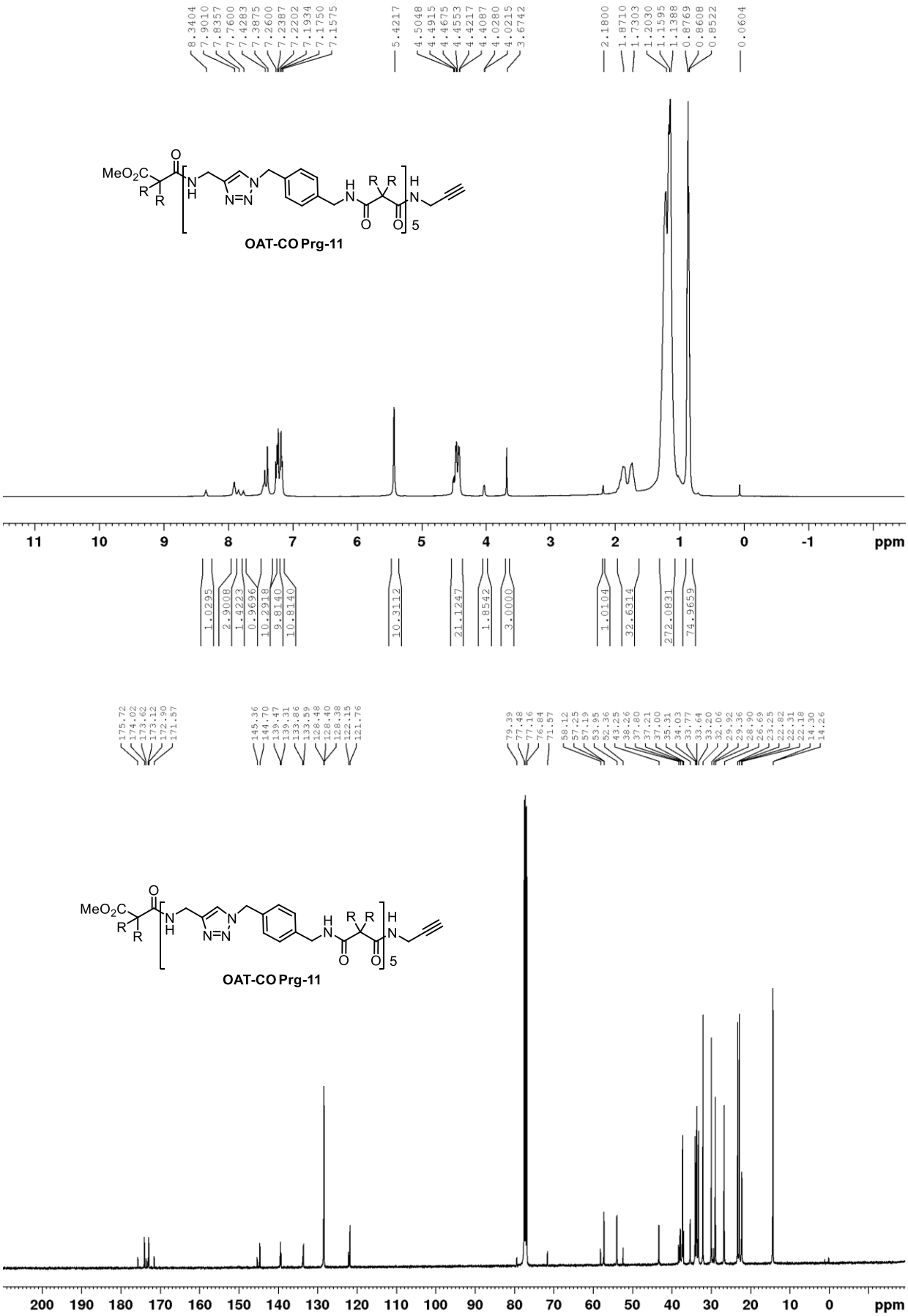
175.65
174.12
174.10
174.07
174.04
172.94
172.91
172.77
171.57
145.34
144.72
139.48
139.42
139.31
139.14
133.76
133.70
133.56
133.47
128.51
128.41
128.37
128.32
122.20
122.04
121.87
77.41
77.16
76.91
58.11
57.42
57.29
57.23
54.02
53.96
53.90
52.35
43.32
43.27
43.24
38.49
37.87
37.80
37.63
37.37
37.23
37.10
37.01
36.99
35.31
35.20
34.05
33.78
33.76
33.69
33.63
33.55
33.24
33.19
33.18
32.06
29.33
28.90
28.86
26.70
26.69
26.62
23.24
22.82
22.62
22.33
22.30
22.18
22.10
14.31
14.26
MeO2C
R
R
N
H
O
N
N=N
H
N
R
R
OH
O
O
5
OAT-CO2H-10
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm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7.1934
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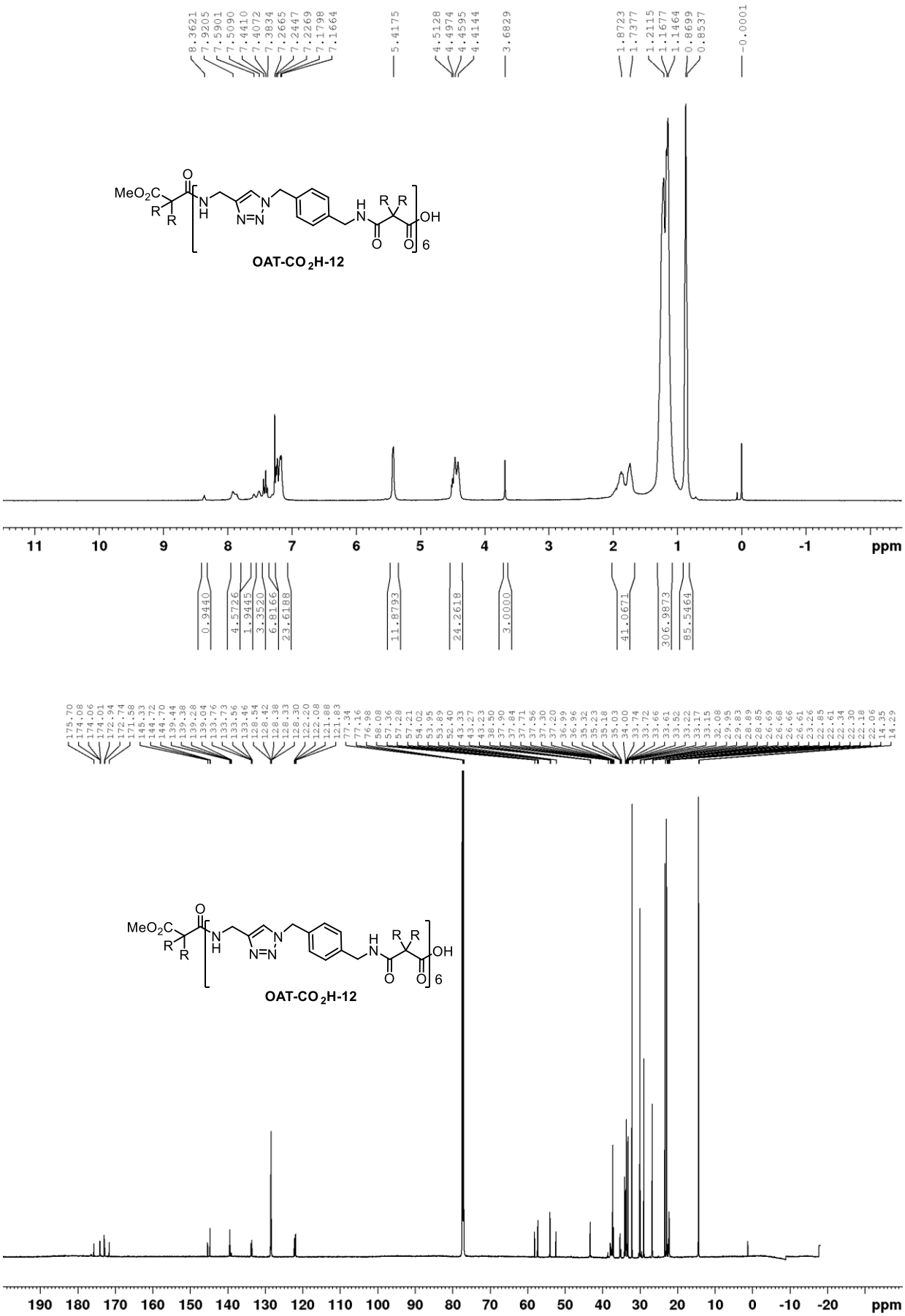
7.1575
5.4217
4.5048
4.4915
4.4675
4.4553
4.4217
4.4087
4.0280
4.0215
3.6742
2.1800
1.8710
1.7303
1.2030
1.1595
1.1388
0.8769
0.8608
0.8522
0.0604
MeO2C
R
R
N
H
N=N
R R
H
N
O
O
5
OAT-CO Prg-11
11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm
1.0295
2.9008
1.4223
0.9696
10.2918
9.8140
10.8140
10.3112
21.1247
1.8542
3.0000
1.0104
32.6314
272.0831
74.9659
175.72
174.02
173.62
173.12
172.90
171.57
145.36
144.70
139.47
139.31
133.86
133.59
128.48
128.40
128.38
122.15
121.76
79.39
77.48
77.16
76.84
71.57
58.12
57.25
57.19
53.95
52.36
43.25
38.26
37.80
37.21
37.00
35.31
34.03
33.77
33.64
33.20
32.06
29.92
29.36
28.90
26.69
23.25
22.82
22.31
22.18
14.30
14.26
MeO2C
R
R
N
H
N=N
R R
H
N
O
O
5
OAT-CO Prg-11
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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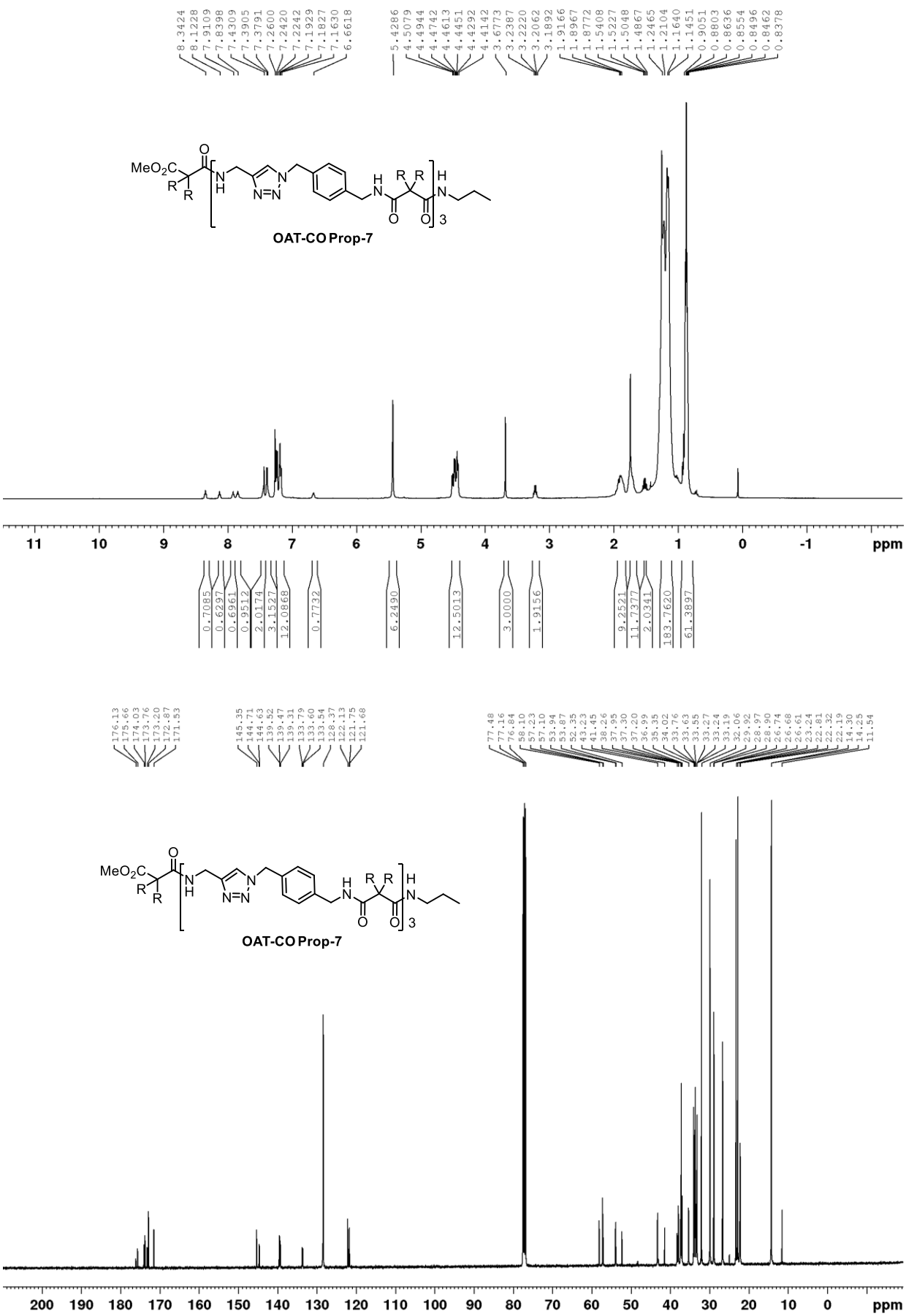
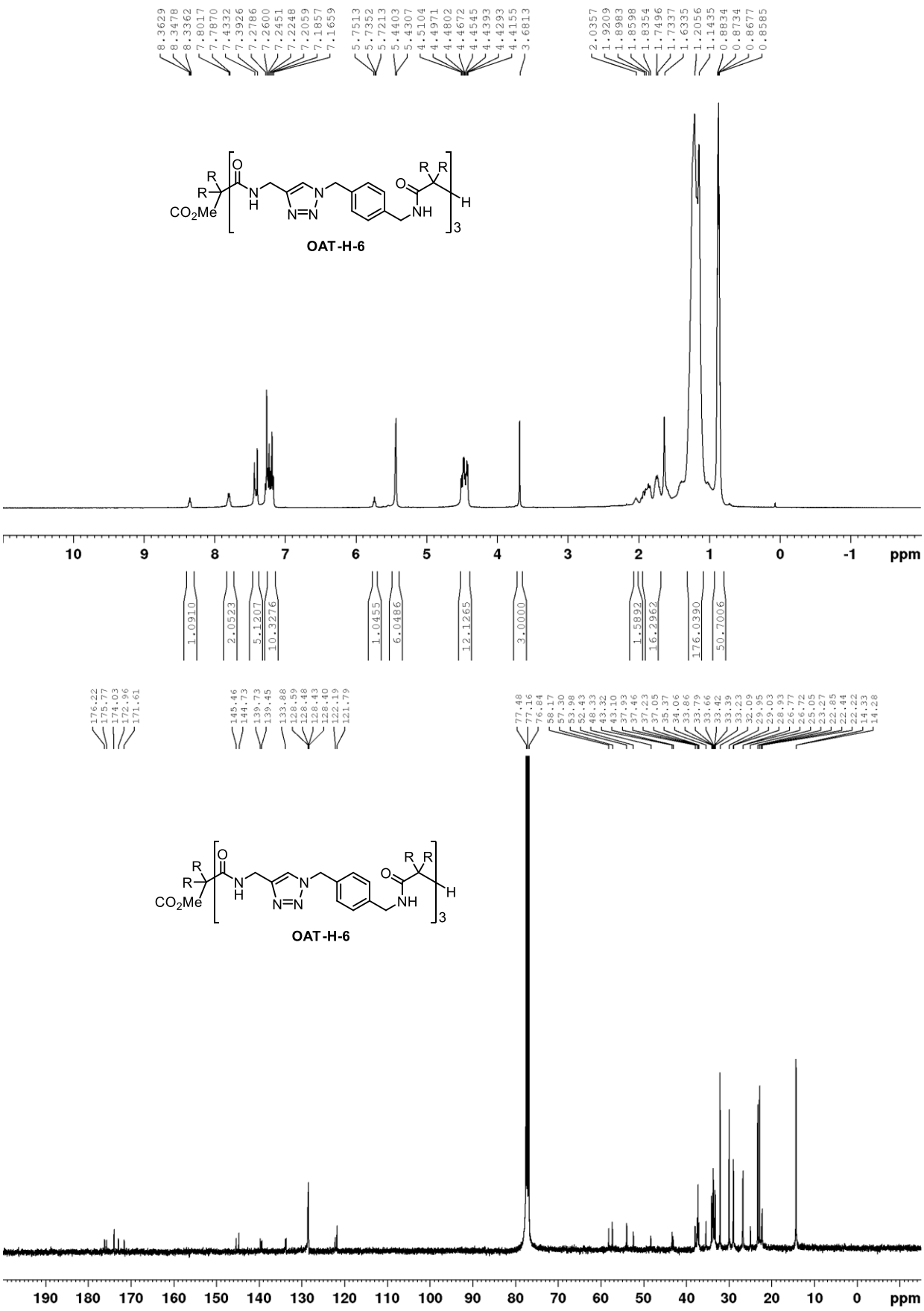
7.2447
7.2269
7.1798
7.1664
5.4175
4.5128
4.4974
4.4595
4.4144
3.6829
1.8723
1.7377
1.2115
1.1677
1.1464
0.8699
0.8537
-0.0001
OAT-CO2H-12
11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm
0.9440
4.5726
1.9445
3.3520
6.8166
23.6188
11.8793
24.2618
3.0000
41.0671
306.9873
85.5464
OAT-CO2H-12
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 ppm


OAT-CO Prop-7
OAT-CO Prop-7
ppm
ppm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7.2451
7.2248
7.2059
7.1857
7.1659
5.7513
5.7352
5.7213
5.4403
5.4307
4.5104
4.4971
4.4802
4.4672
4.4545
4.4393
4.4293
4.4155
3.6813
2.0357
1.9209
1.8983
1.8598
1.8354
1.7496
1.7337
1.6335
1.2056
1.1435
0.8834
0.8734
0.8677
0.8585
OAT-H-6
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.0910
2.0523
5.1207
10.3276
1.0455
6.0486
12.1265
3.0000
1.5892
16.2962
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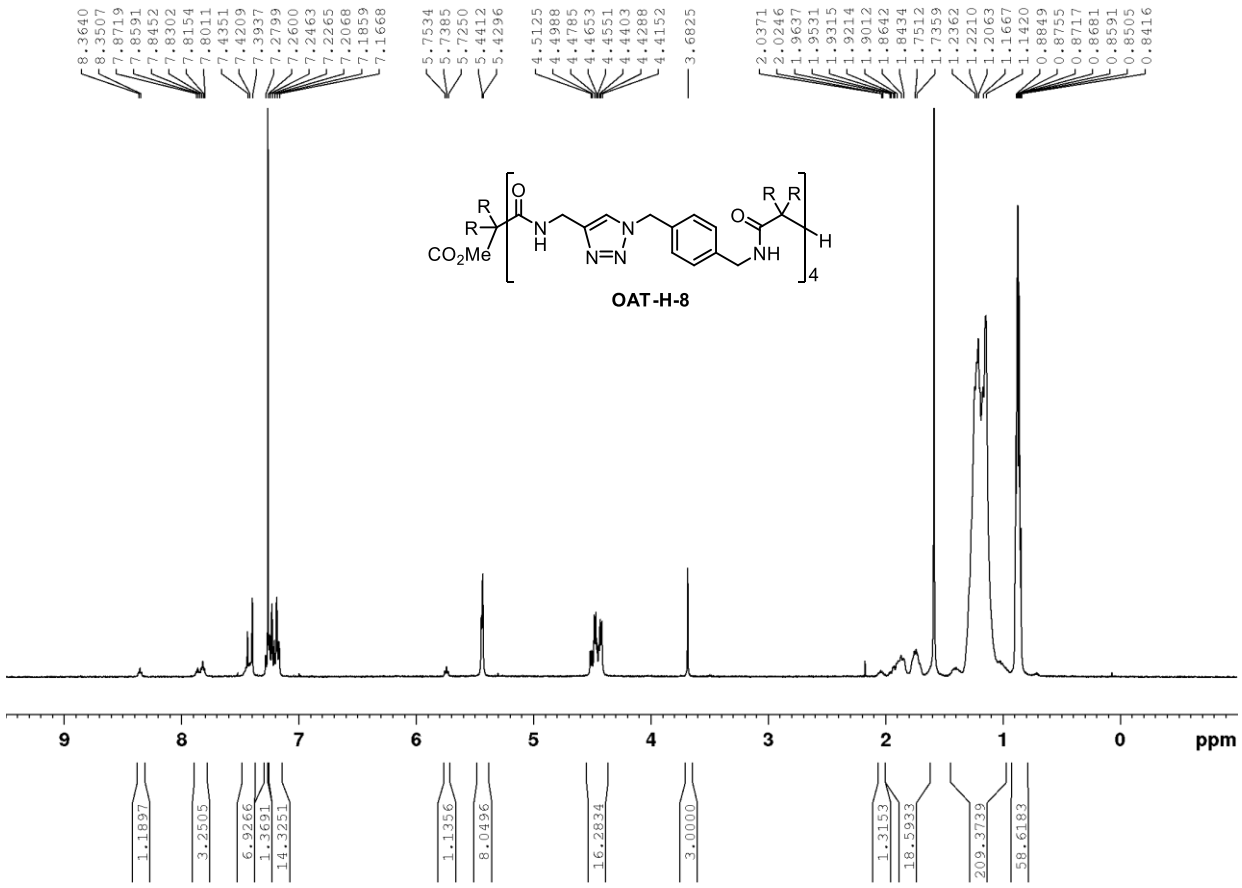
50.7006
176.22
175.77
174.03
172.96
171.61
145.46
144.73
139.73
139.45
133.88
128.59
128.48
128.43
128.40
122.19
121.79
77.48
77.16
76.84
58.17
57.30
53.98
52.43
48.33
43.32
43.10
37.93
37.46
37.23
37.05
35.37
34.06
33.86
33.79
33.66
33.42
33.39
33.23
32.09
29.95
29.03
28.93
26.77
26.72
25.05
23.27
22.85
22.44
22.22
14.33
14.28
OAT-H-6
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.3937
7.2799
7.2600
7.2463
7.2265
7.2068
7.1859
7.1668
5.7534
5.7385
5.7250
5.4412
5.4296
4.5125
4.4988
4.4785
4.4653
4.4551
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4.4288
4.4152
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2.0246
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1.9531
1.9315
1.9214
1.9012
1.8642
1.8434
1.7512
1.7359
1.2362
1.2210
1.2063
1.1667
1.1420
0.8849
0.8755
0.8717
0.8681
0.8591
0.8505
0.8416
R
R
CO2Me
O
N
H
N
N=N
O
R R
NH
H
4
OAT-H-8
9
8
7
6
5
4
3
2
1
0
ppm
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3.2505
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1.1356
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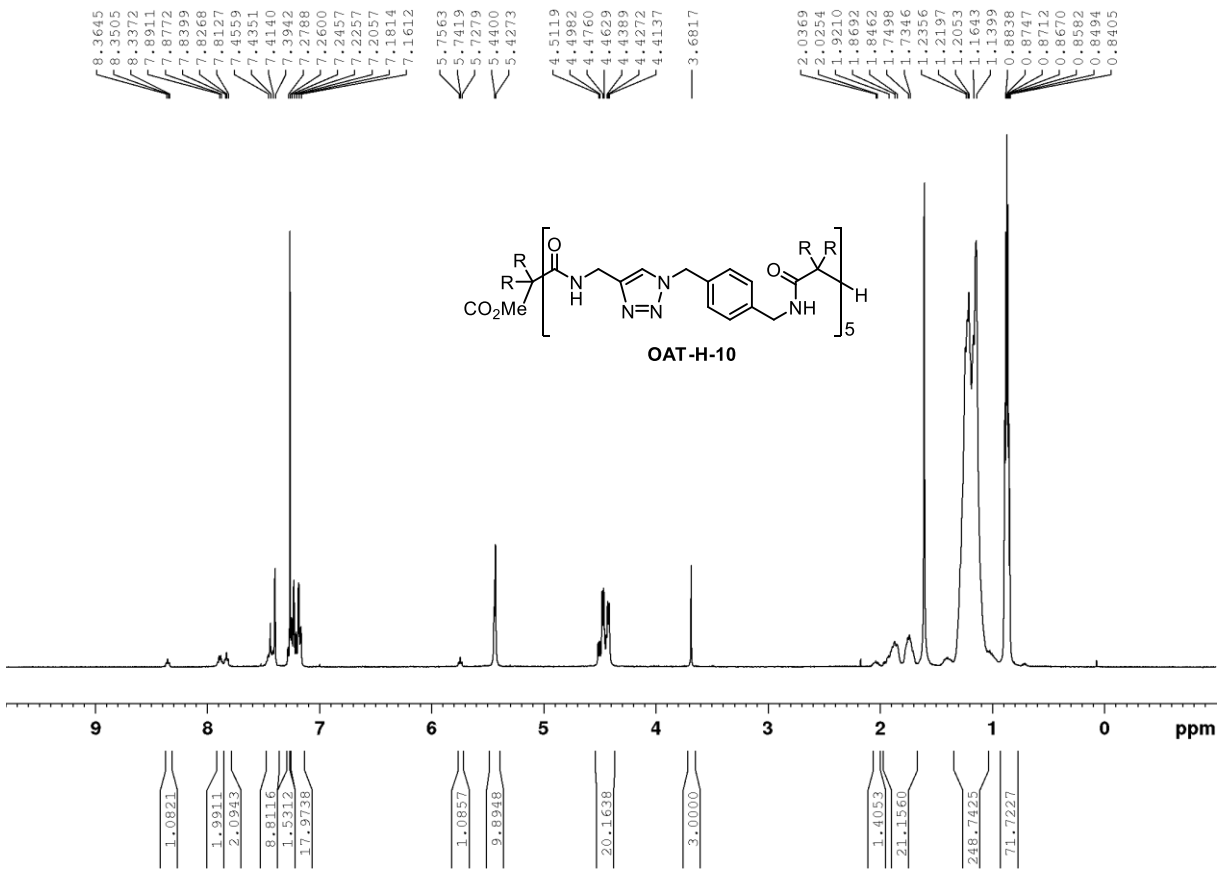
58.6183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7.2057
7.1814
7.1612
5.7563
5.7419
5.7279
5.4400
5.4273
4.5119
4.4982
4.4760
4.4629
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4.4272
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2.0254
1.9210
1.8692
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1.2356
1.2197
1.2053
1.1643
1.1399
0.8838
0.8747
0.8712
0.8670
0.8582
0.8494
0.8405
R
R
CO2Me
O
N
H
N
N=N
O
R R
NH
H
5
OAT-H-10
9
8
7
6
5
4
3
2
1
0
ppm
1.0821
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8.8116
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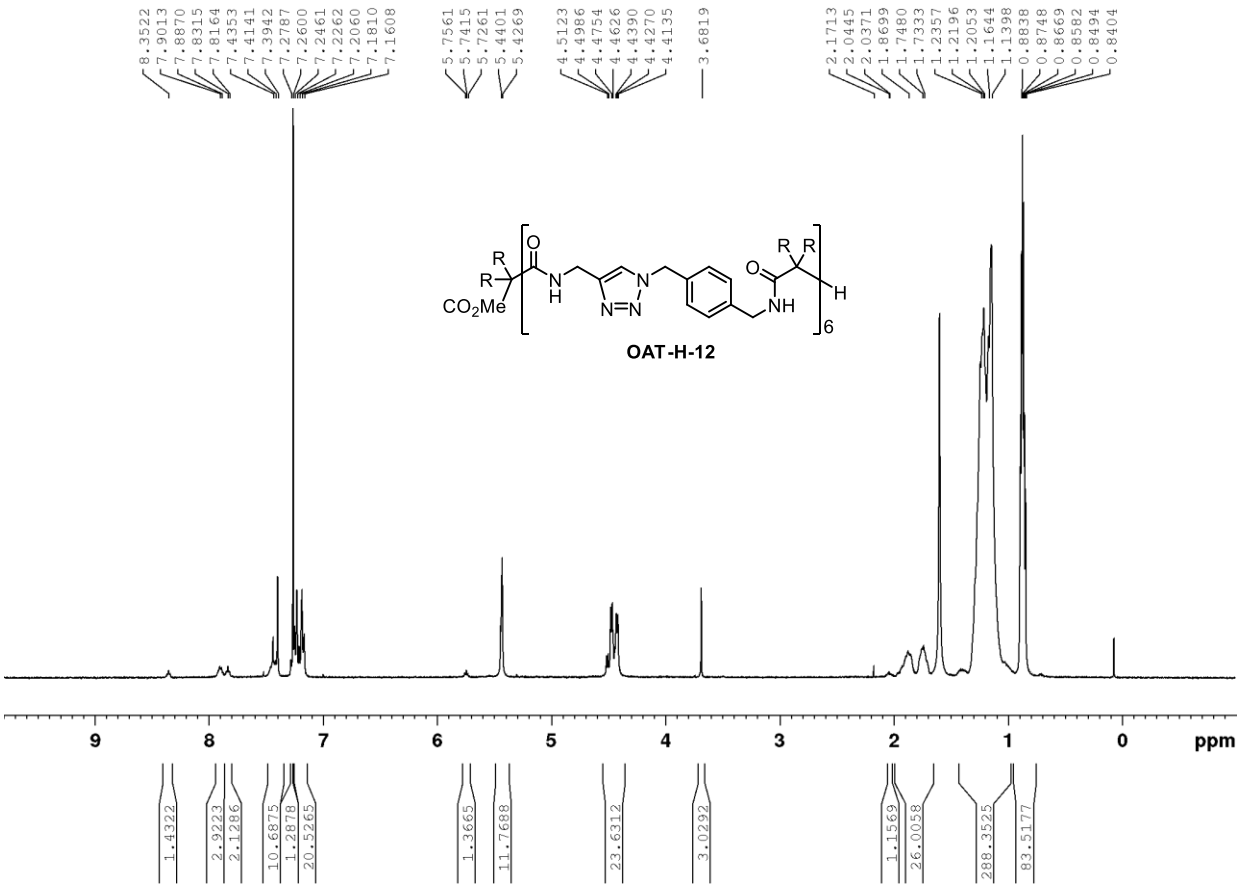
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OAT-H-12
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8
7
6
5
4
3
2
1
0
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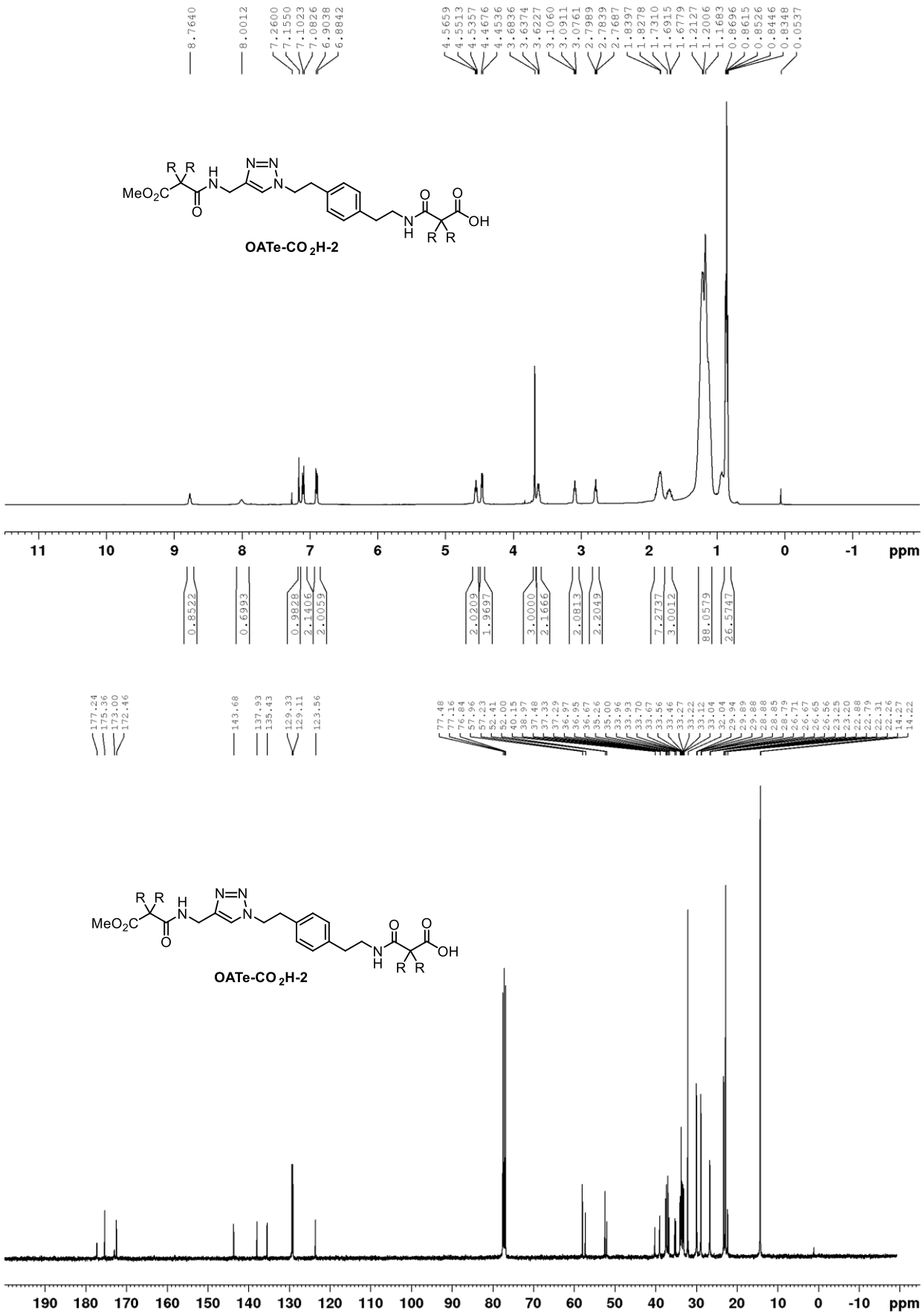
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1.8278
1.7310
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1.2006
1.1683
0.8696
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0.0537
OATe-CO2H-2
11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm
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0.9828
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173.00
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129.11
123.56
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57.96
57.23
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40.15
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37.29
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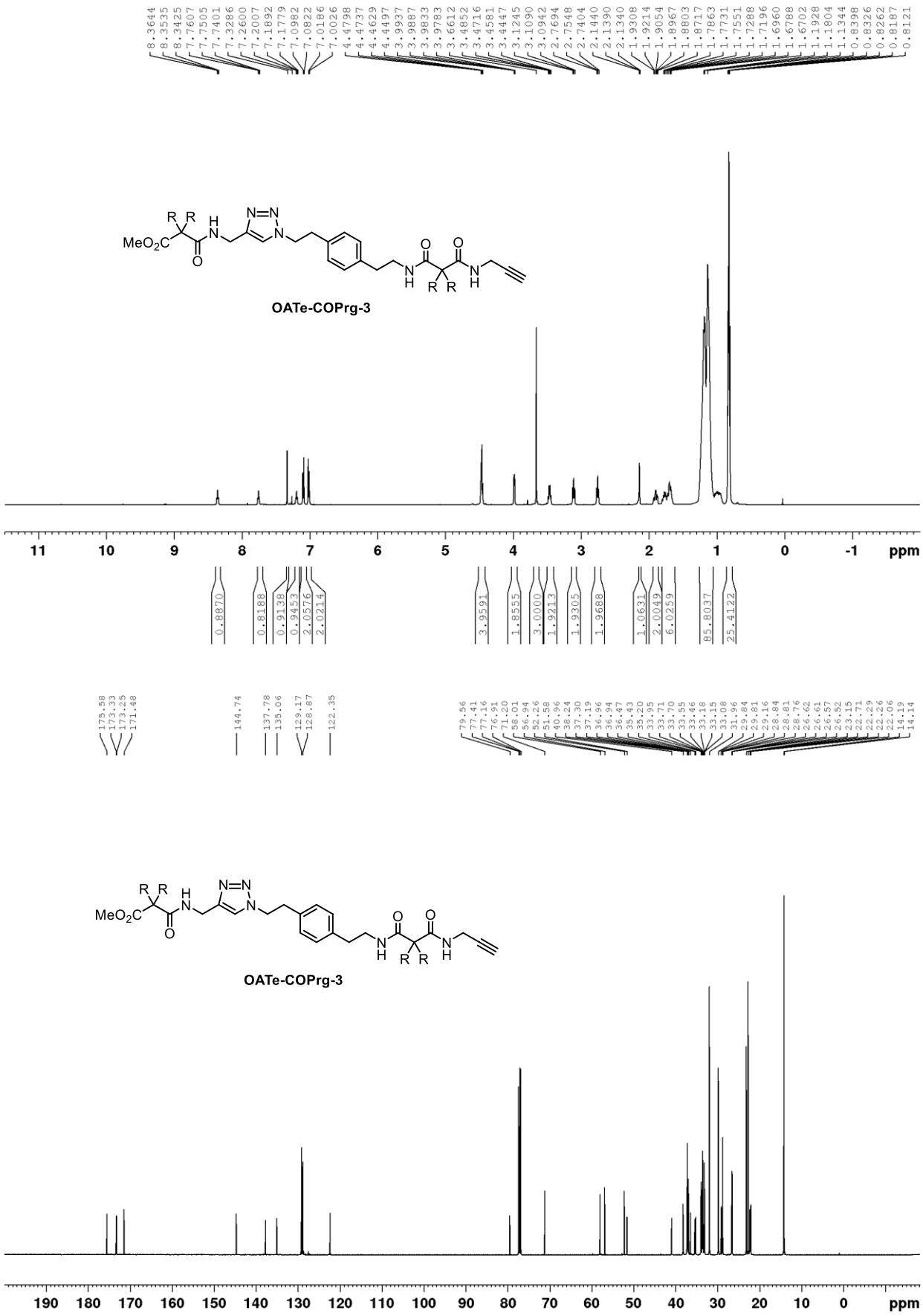
33.93
33.70
33.67
33.56
33.46
33.27
33.22
33.12
33.04
32.94
29.84
29.89
29.88
28.85
28.79
26.71
26.67
26.65
26.56
23.25
23.20
22.88
22.79
22.31
22.26
14.27
14.22
OATe-CO2H-2
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm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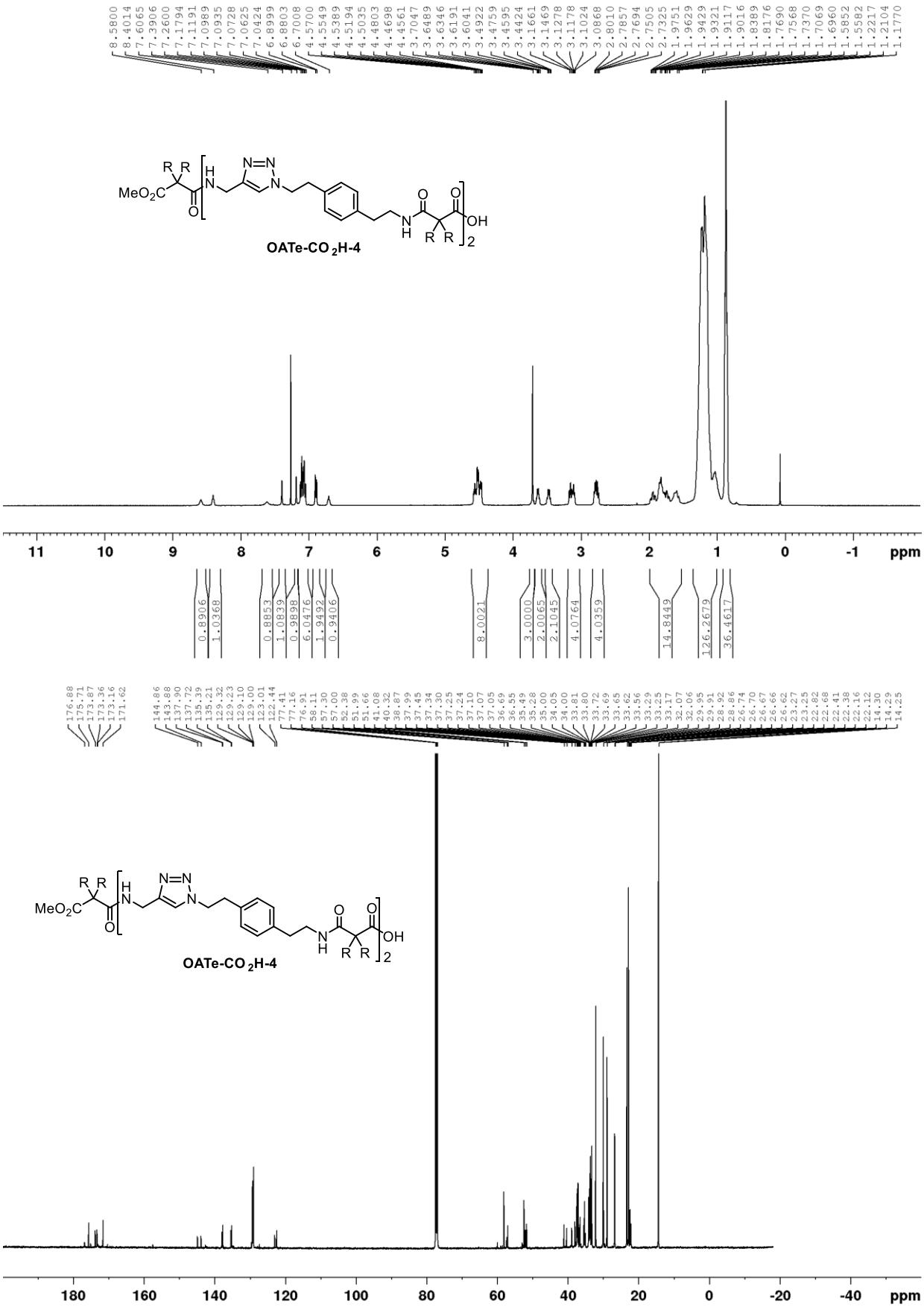
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ppm
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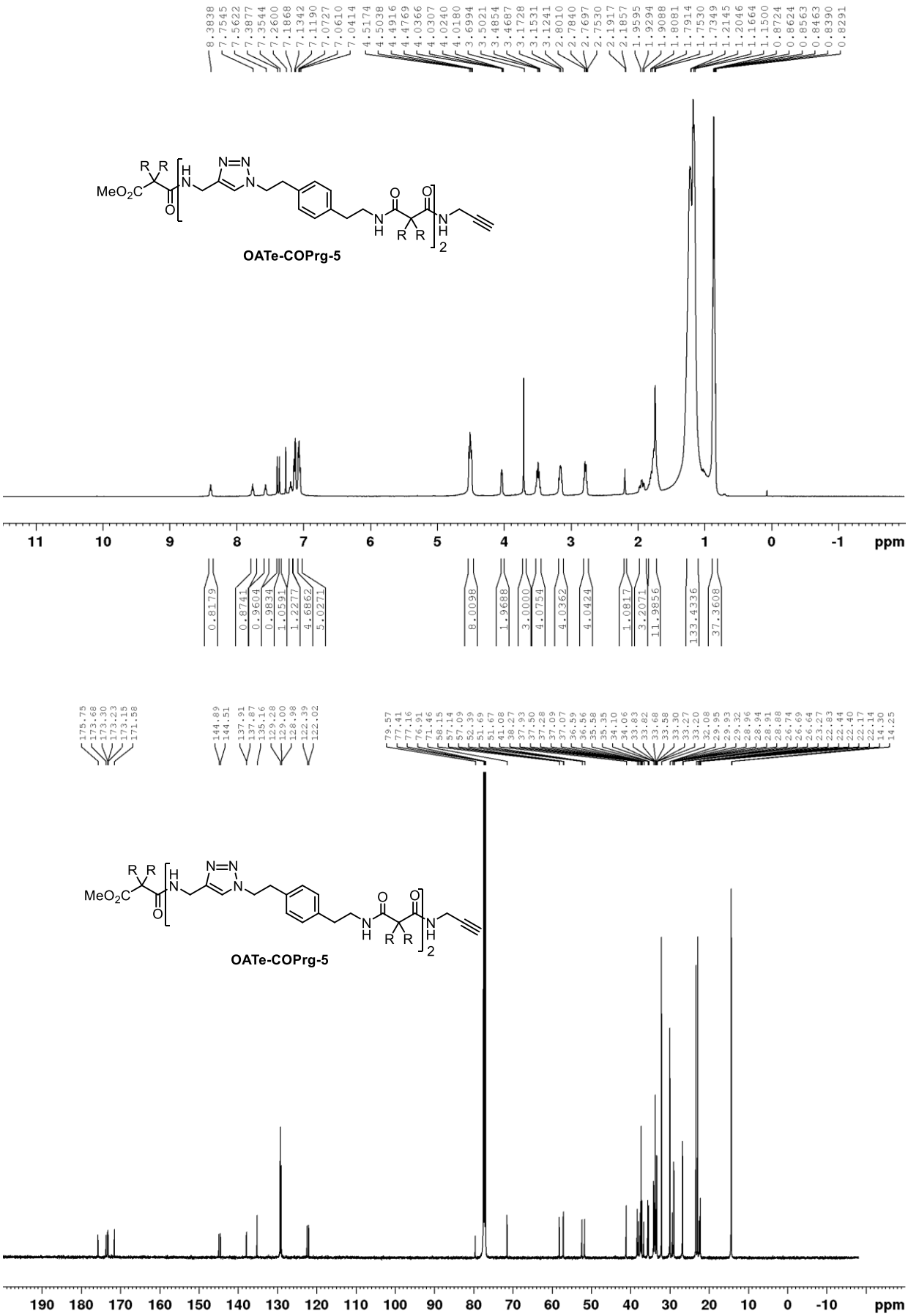
ppm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ppm
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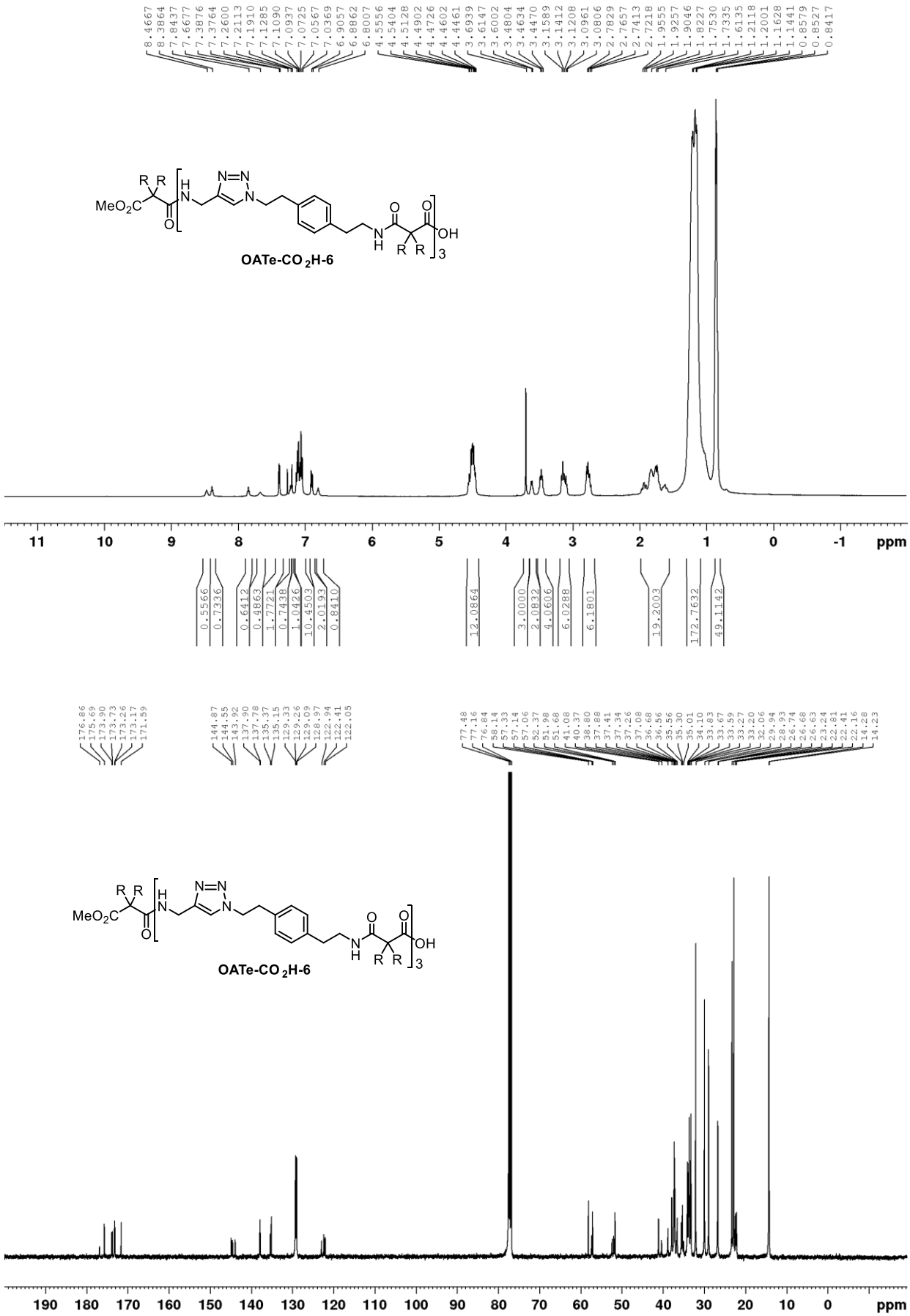
ppm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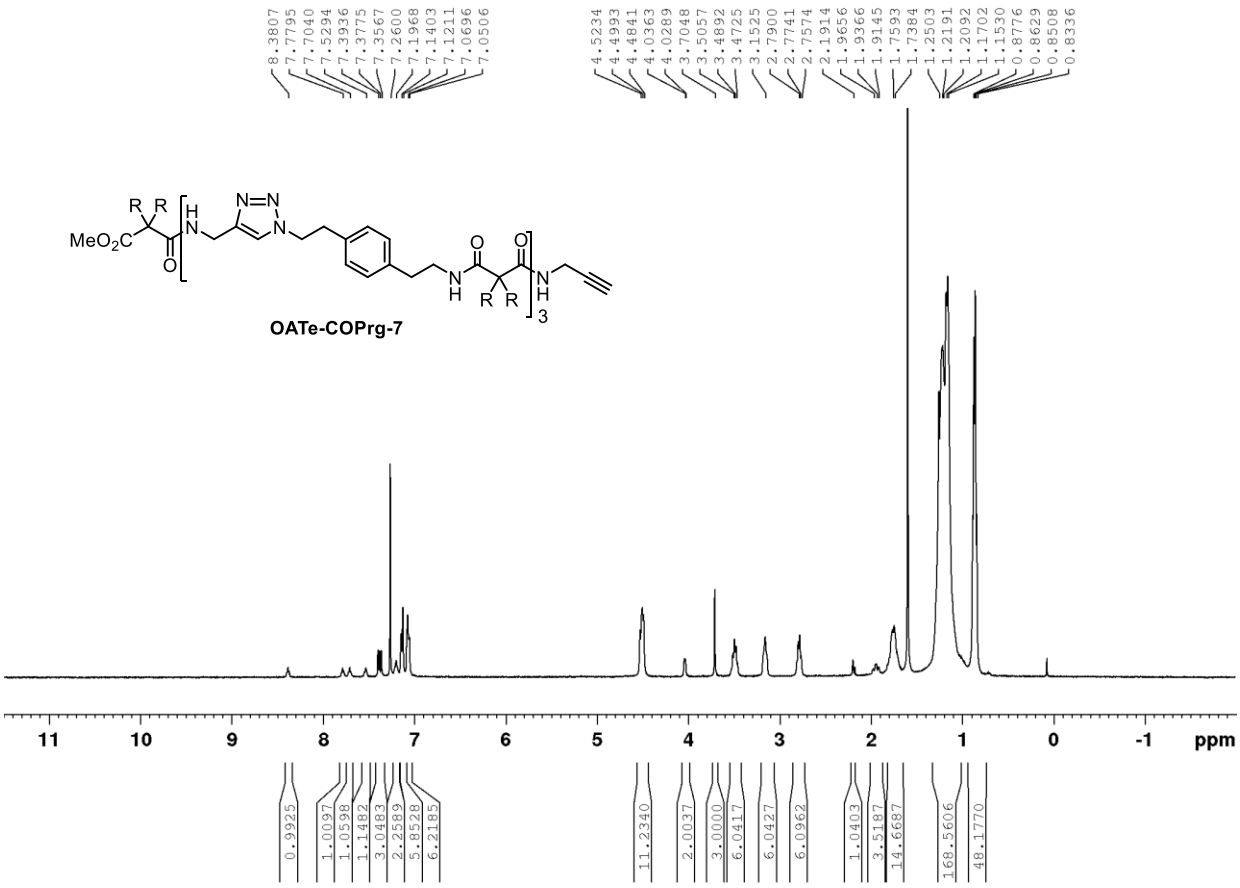
ppm
OATe-CO2H-6
ppm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1.1530
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OATe-COPrg-7
11
10
9
8
7
6
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2
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-1
ppm
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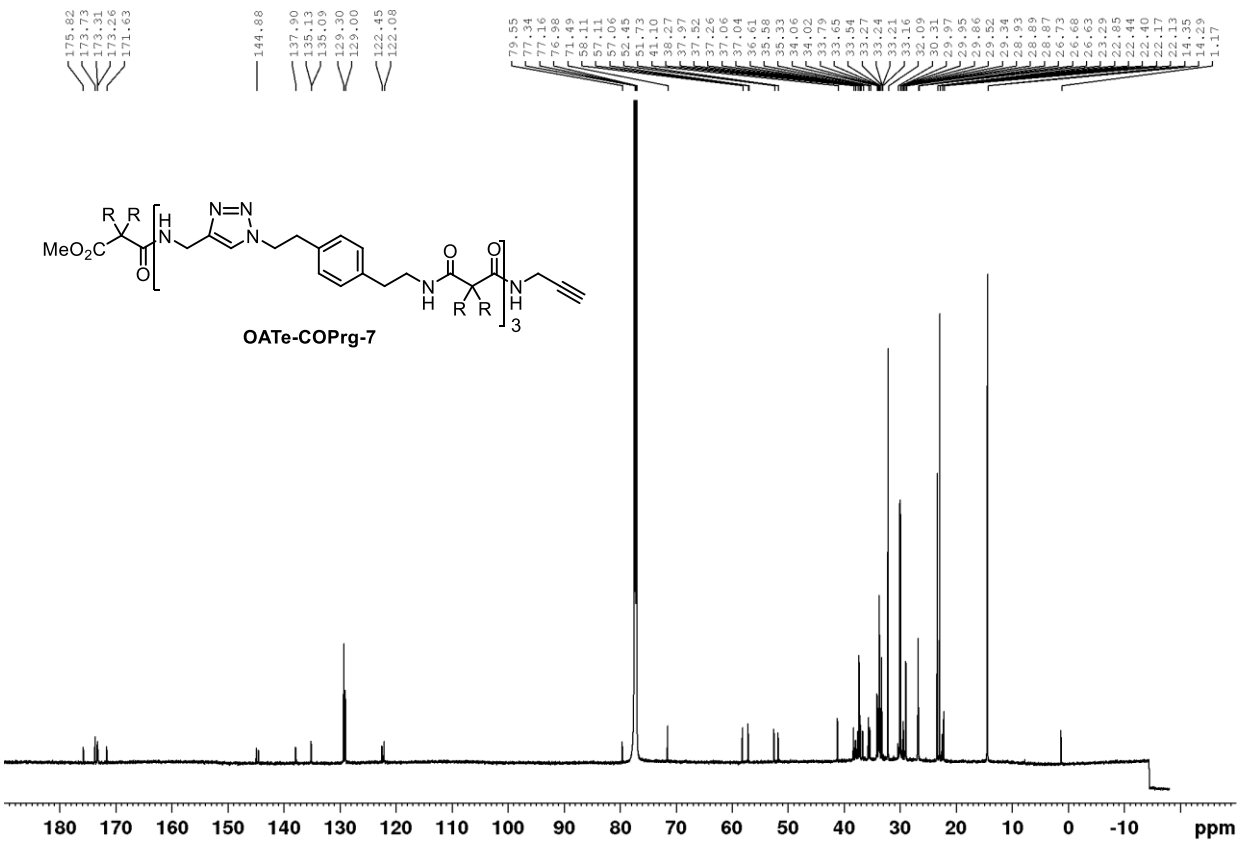
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77.34
77.16
76.98
71.49
58.11
57.11
57.06
52.45
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37.97
37.52
37.26
37.06
37.04
36.61
35.58
35.33
34.06
34.02
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33.65
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28.93
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22.85
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14.35
14.29
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OATe-COPrg-7
180
170
160
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110
100
90
80
70
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50
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30
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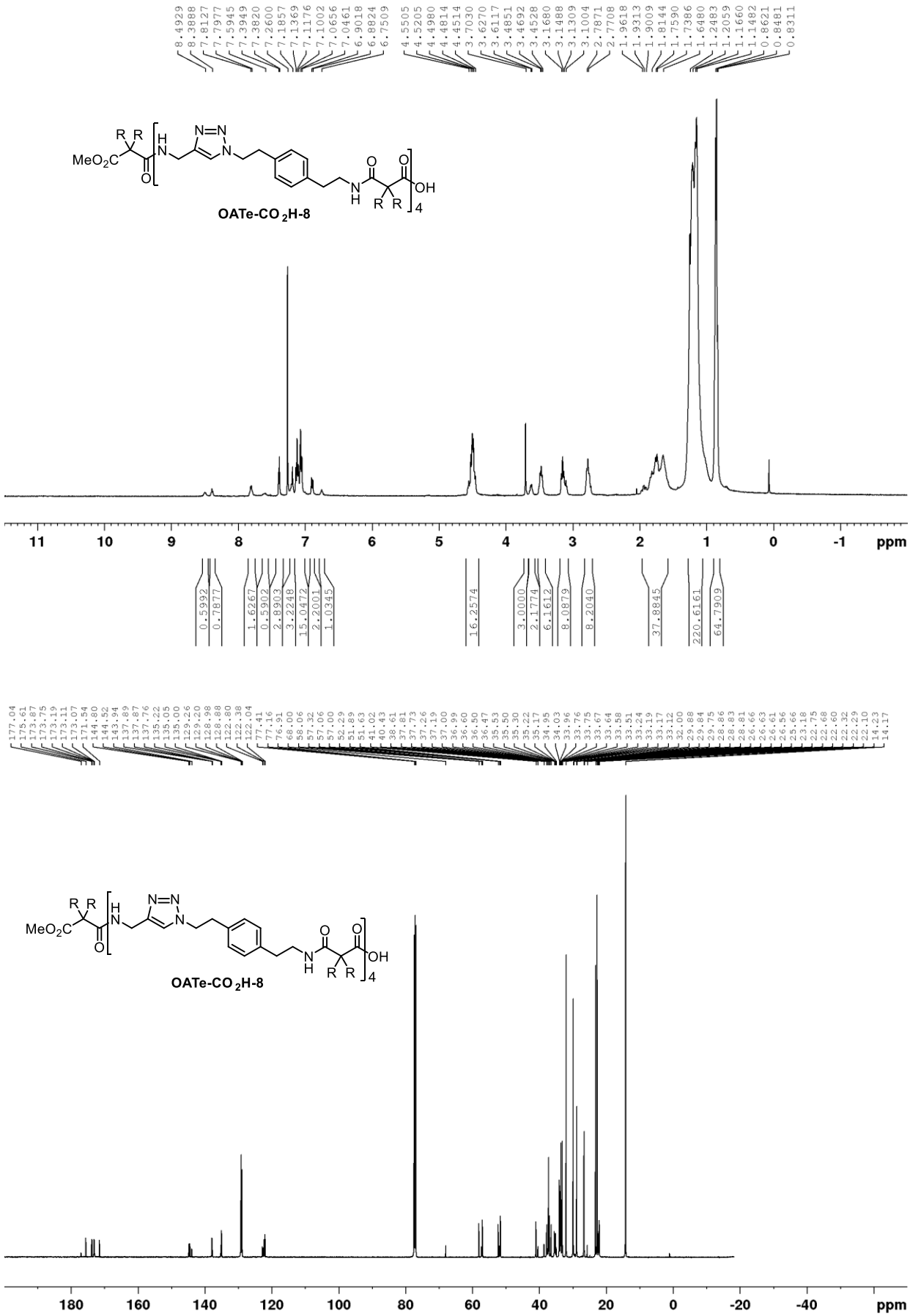
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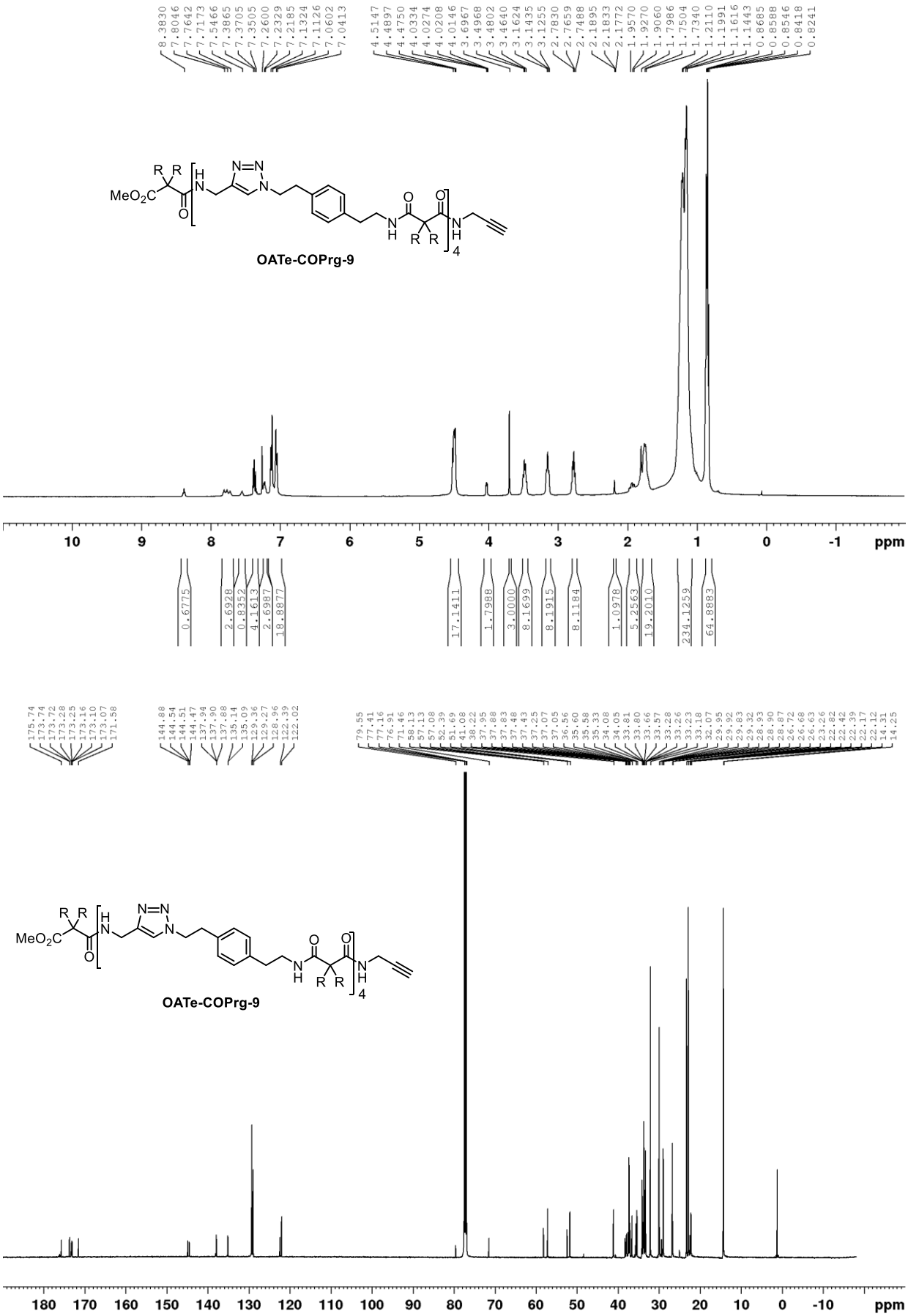
OATe-CO2H-8
ppm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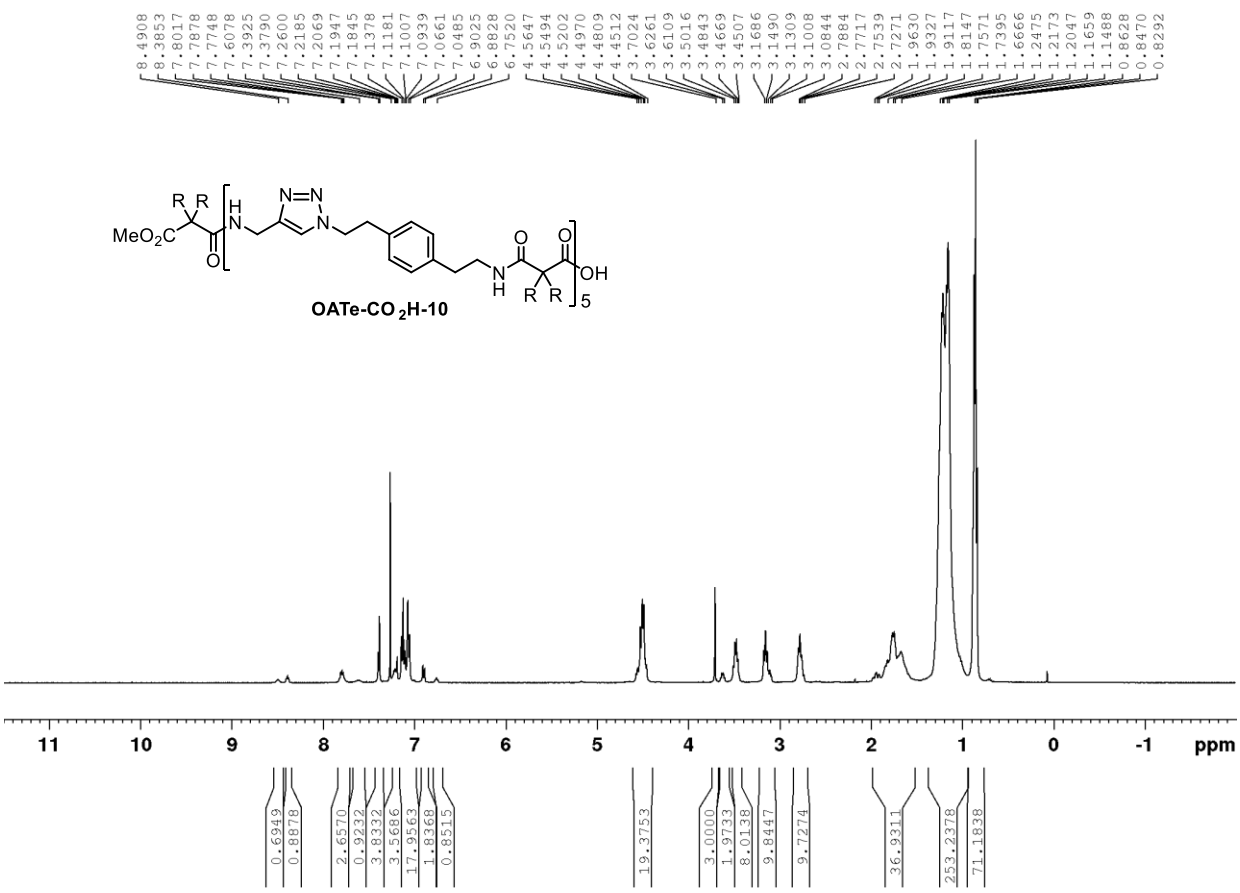
ppm
ppm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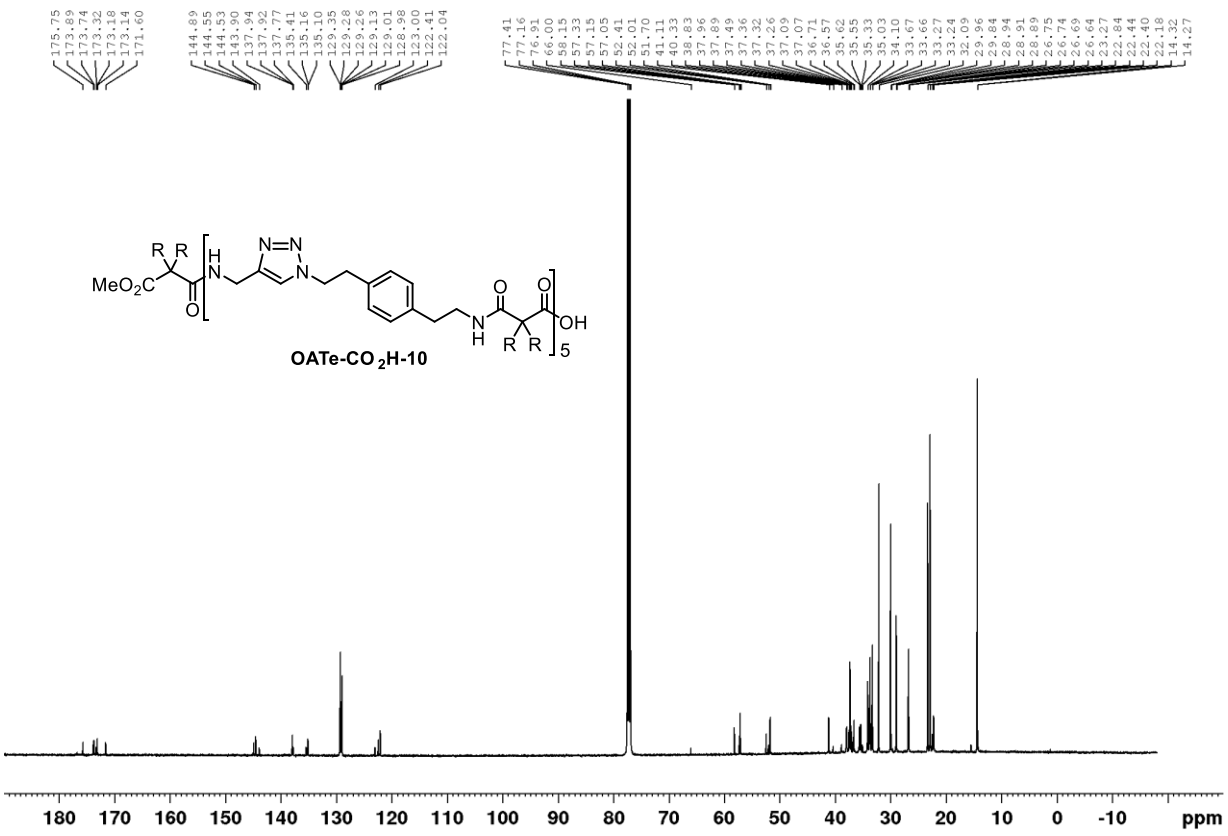

OATe-CO2H-10

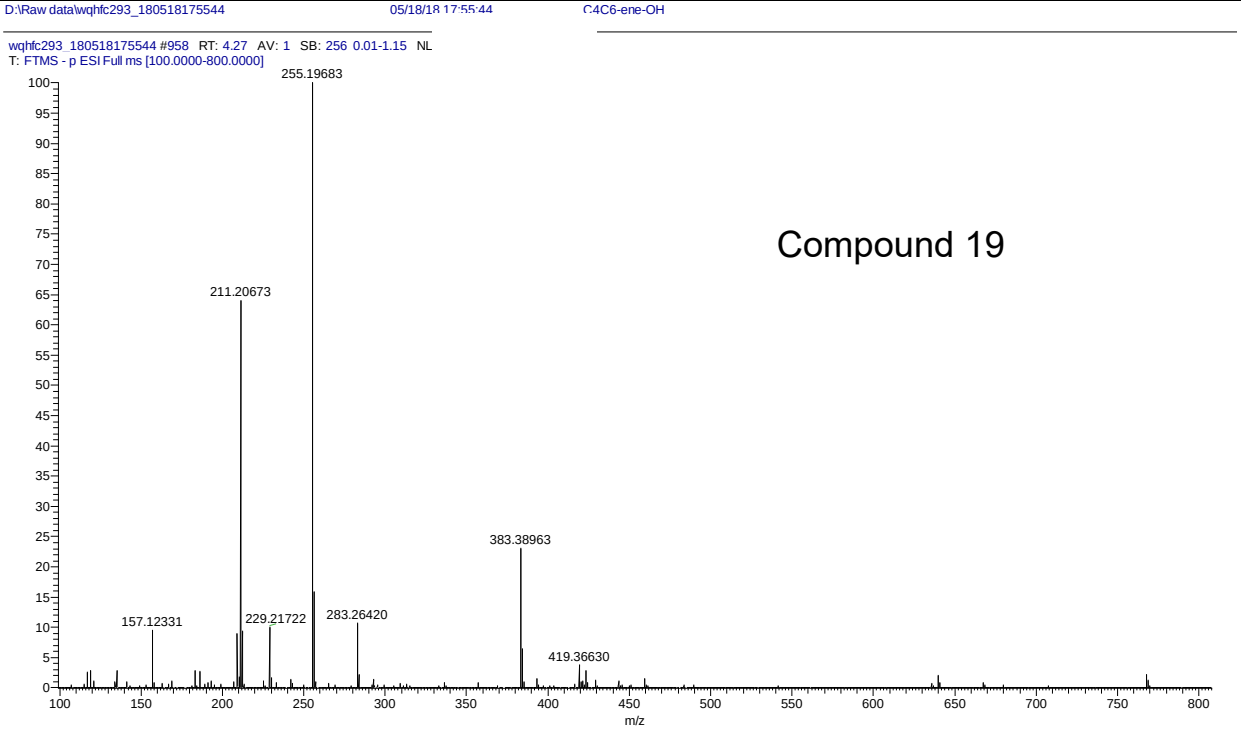
Thermo QEFMS Analysis Report

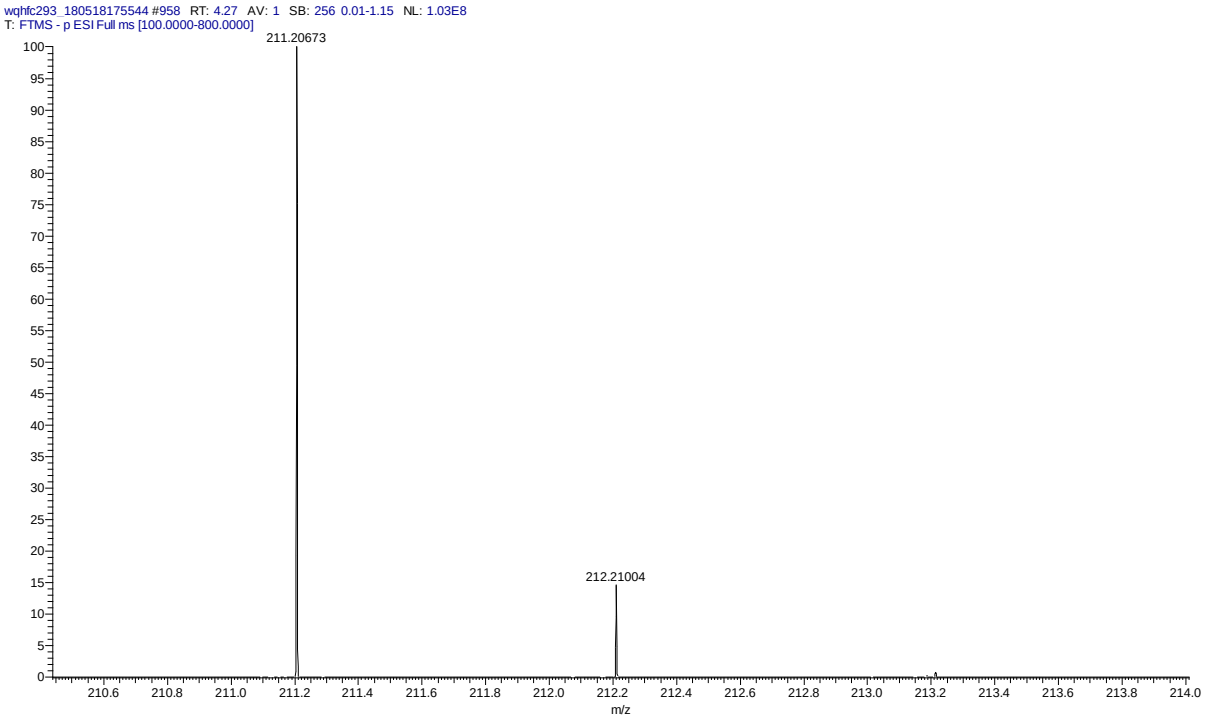
Analysis Info

Sample Name :	C4C6-ene-OH	Reference No.:	Wqhfc293
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Negative
Comment :	ESI neg, 3.0kV, by infusion, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{14}H_{28}O$
Experimental Mass $[M-H]^-$:	211.20673
Theoretical Mass $[M-H]^-$:	211.20674
Error (ppm) :	0.0





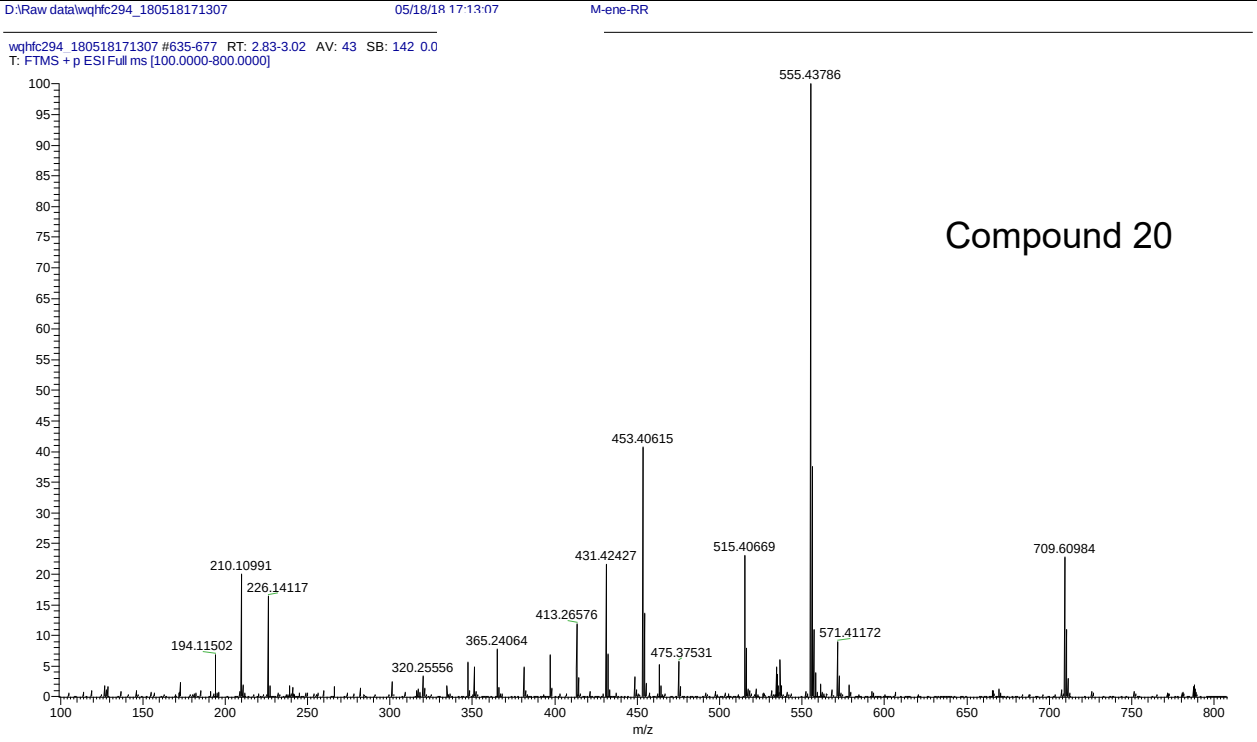
Thermo QEFMS Analysis Report

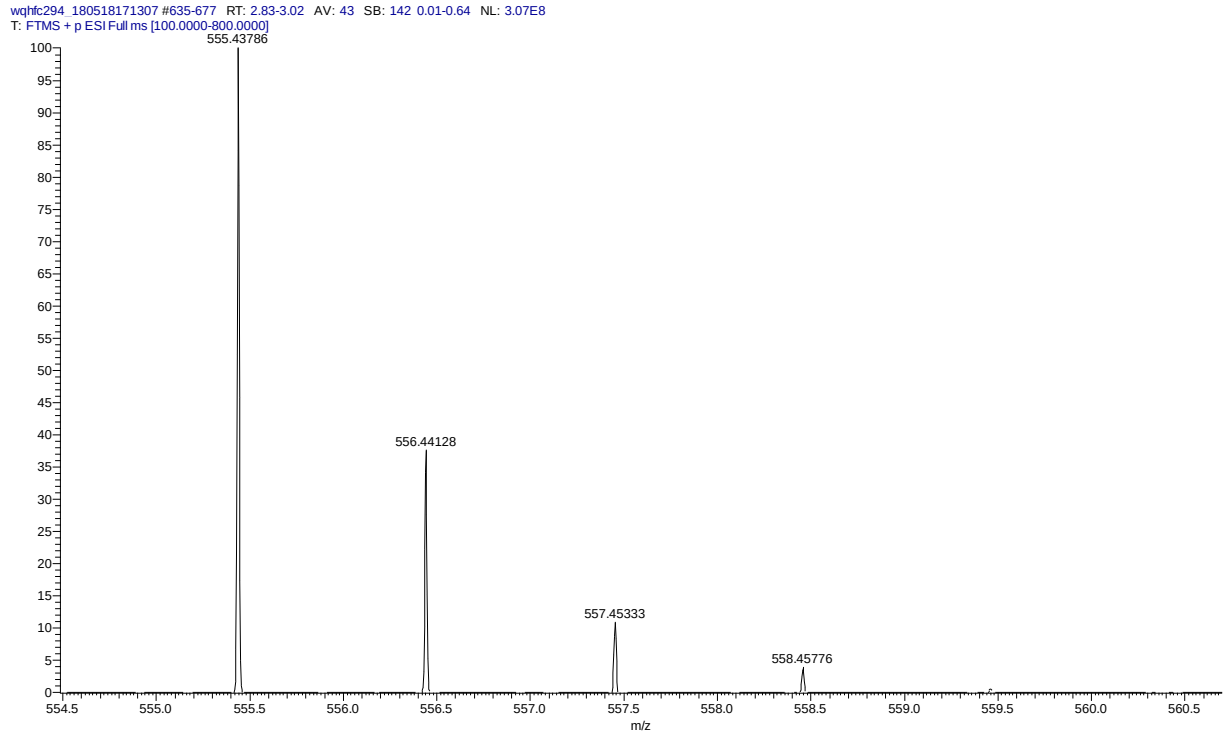
Analysis Info

Sample Name :	M-ene-RR	Reference No.:	Wqhfc294
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	positive
Comment :	ESI pos, 3.5kV, by infusion, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{34}H_{60}O_4$
Experimental Mass $[M+Na]^+$:	555.43786
Theoretical Mass $[M+Na]^+$:	555.43838
Error (ppm) :	0.9





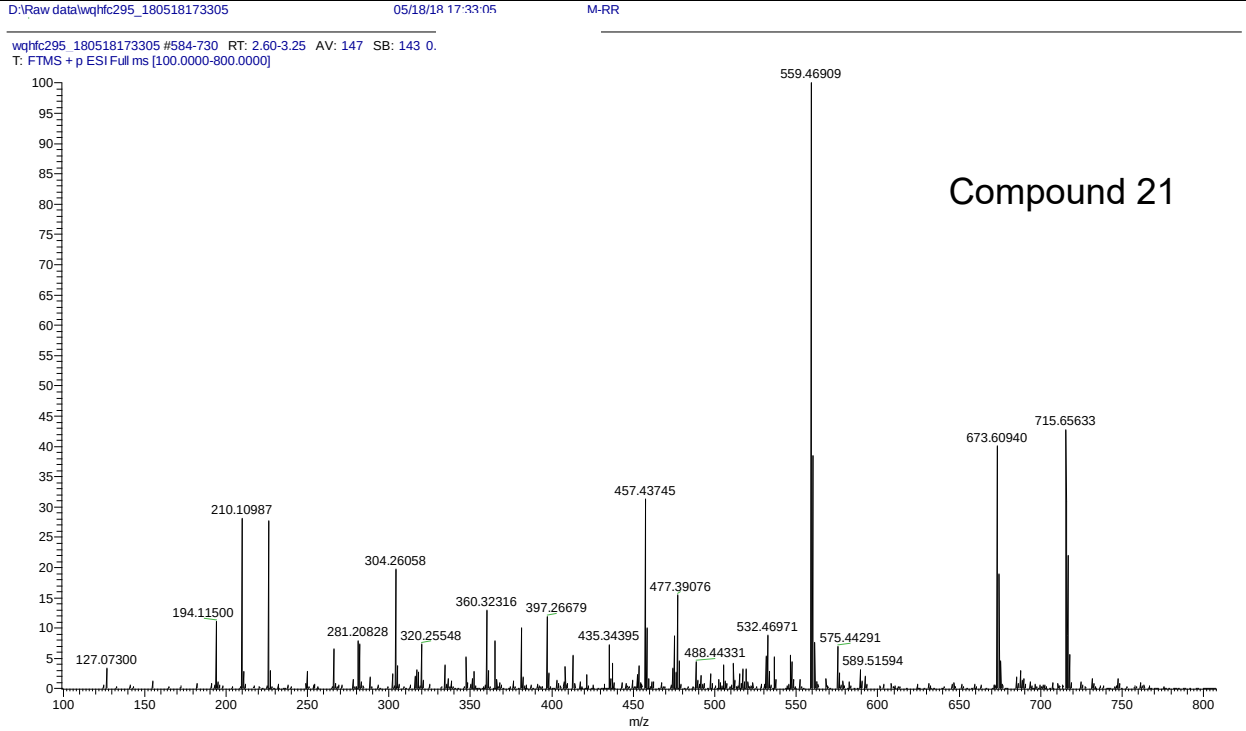
Thermo QEFMS Analysis Report

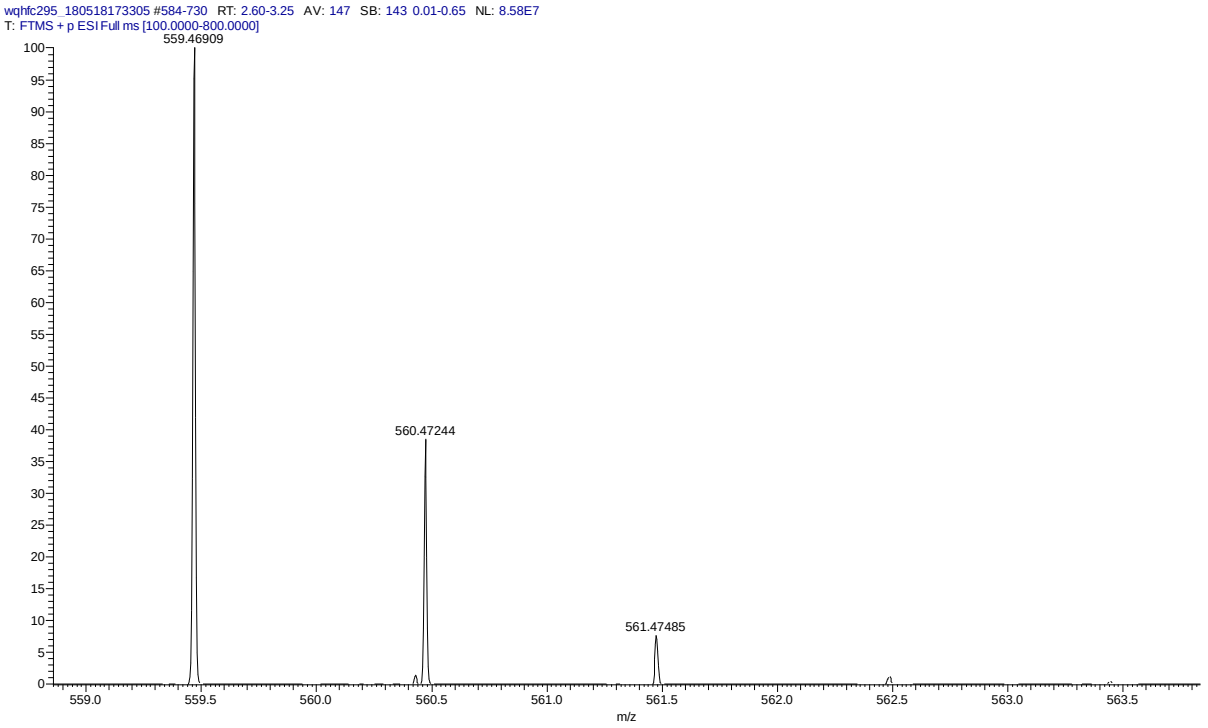
Analysis Info

Sample Name :	M-RR	Reference No.:	Wqhfc295
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	positive
Comment :	ESI pos, 3.5kV, by infusion, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{34}H_{64}O_4$
Experimental Mass $[M+Na]^+$:	559.46909
Theoretical Mass $[M+Na]^+$:	559.46968
Error (ppm) :	1.1





Bruker 9.4T FTICR MS Analysis Report

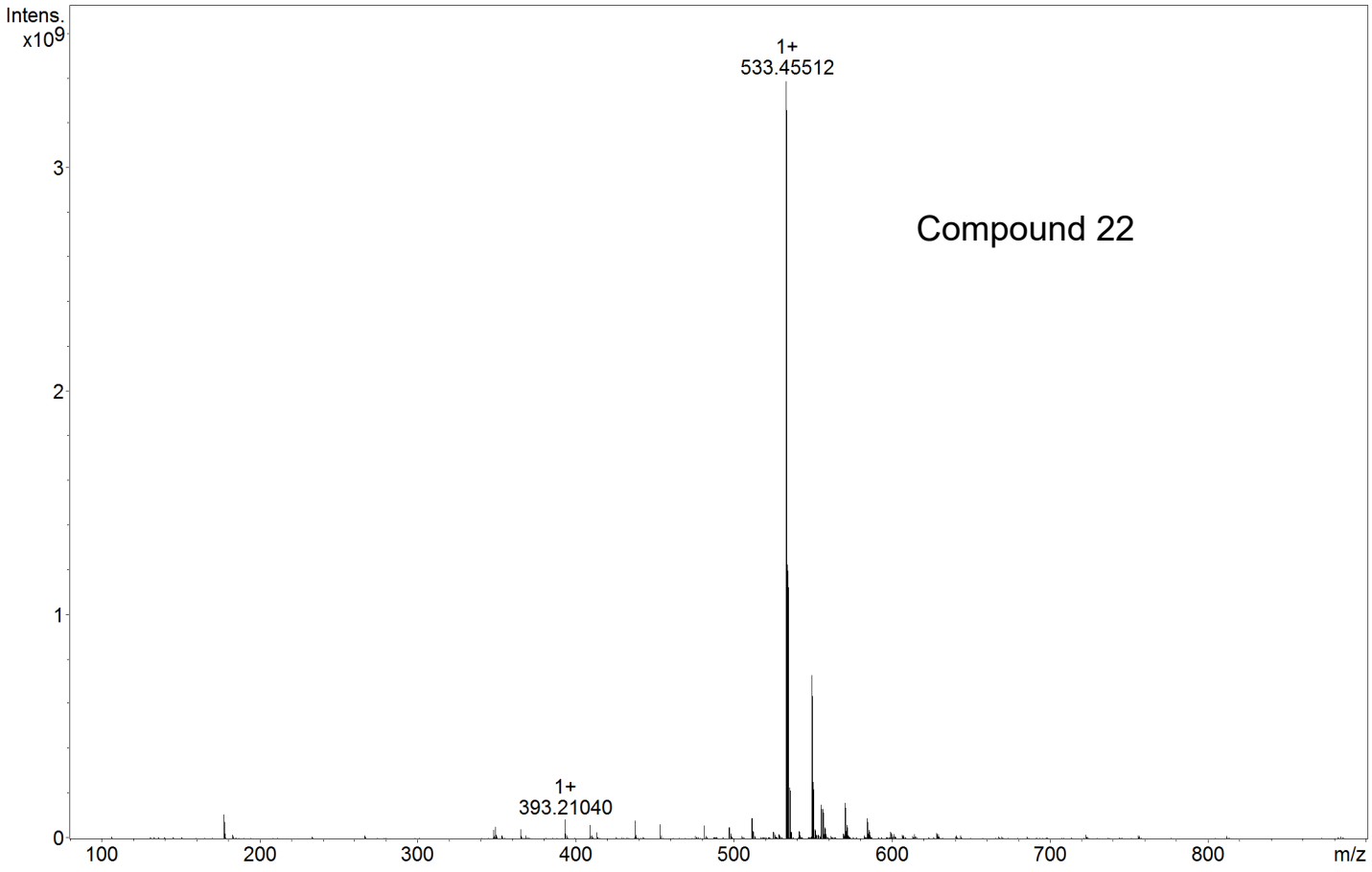
Faculty of Science, The Chinese University of Hong Kong

Analysis Info

Sample Name :	RRCOOHCOOMe	Reference No. :	xhfc039
Applicant Name :	Qi Qiuli	Analysis Date :	25/1/2016 13:00:09
Analysis Path :	xhfc039_000001.d		
Instrument :	solariX	Polarity	Positive
Method	4_17_mass_range_pos_7T	Acquired Scans	16
Comment :	4.4kV, 4ul/min, 0.8 bar nebulizer gas		

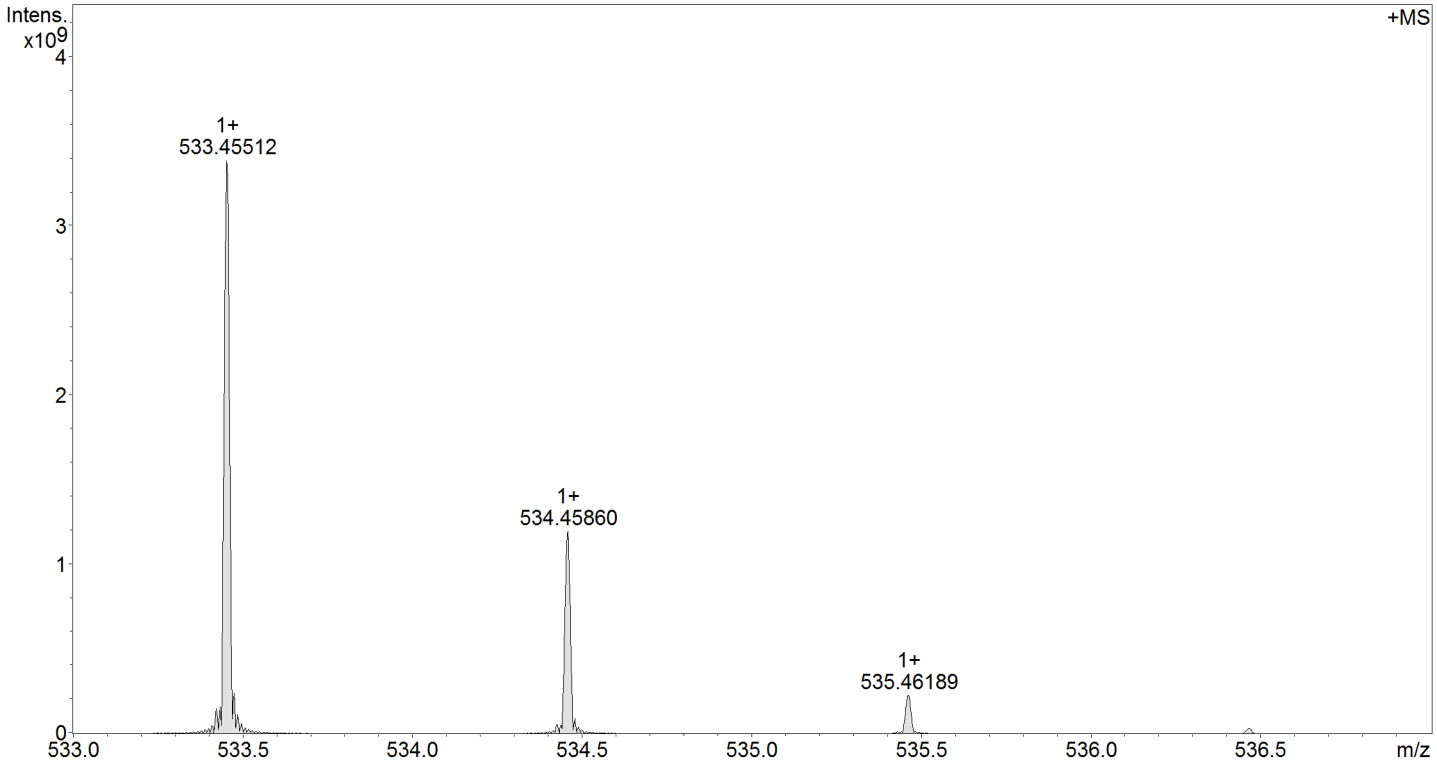
Accurate Mass Measurement

Molecular formula :	C32H62O4
Abundant Isotopic (theoretical) [M+Na]+ :	533.454032
Monoisotopic (theoretical) [M+Na]+ :	533.454032
(experimental) [M+Na]+ :	533.45512
error (ppm) :	2.0



Bruker 9.4T FTICR MS Analysis Report

Faculty of Science, The Chinese University of Hong Kong



Bruker 9.4T FTICR MS Analysis Report

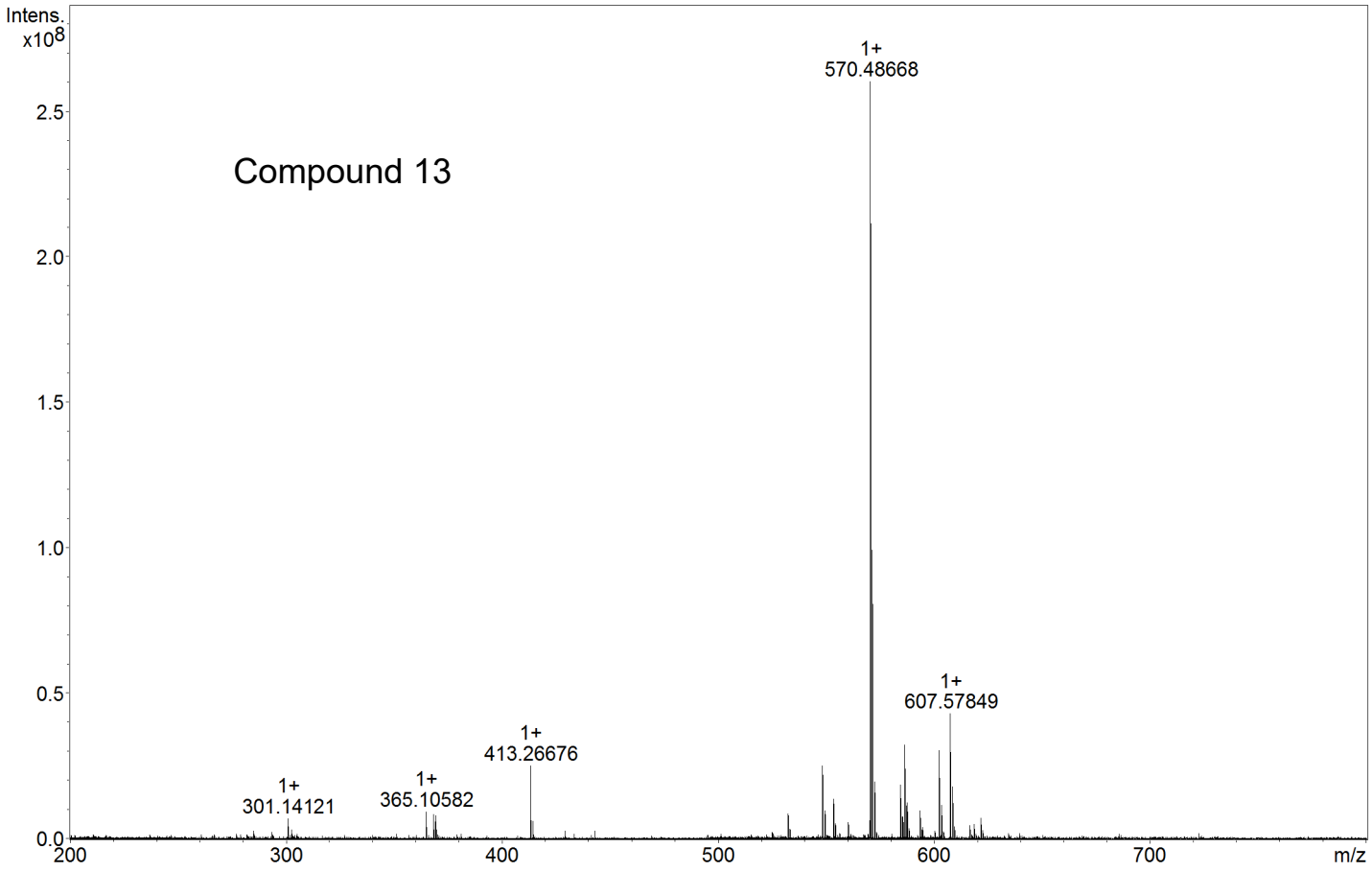
Faculty of Science, The Chinese University of Hong Kong

Analysis Info

Sample Name :	RRC3	Reference No. :	xhfc061
Applicant Name :	Zheng Kun	Analysis Date :	11/3/2016 12:43:18
Analysis Path :	xhfc061_000002.d		
Instrument :	solariX	Polarity	Positive
Method	4_17_mass_range_pos_7T	Acquired Scans	11
Comment :	4.5kV, 140ul/hr, 1.0 bar nebulizer gas, TOF = 0.9		

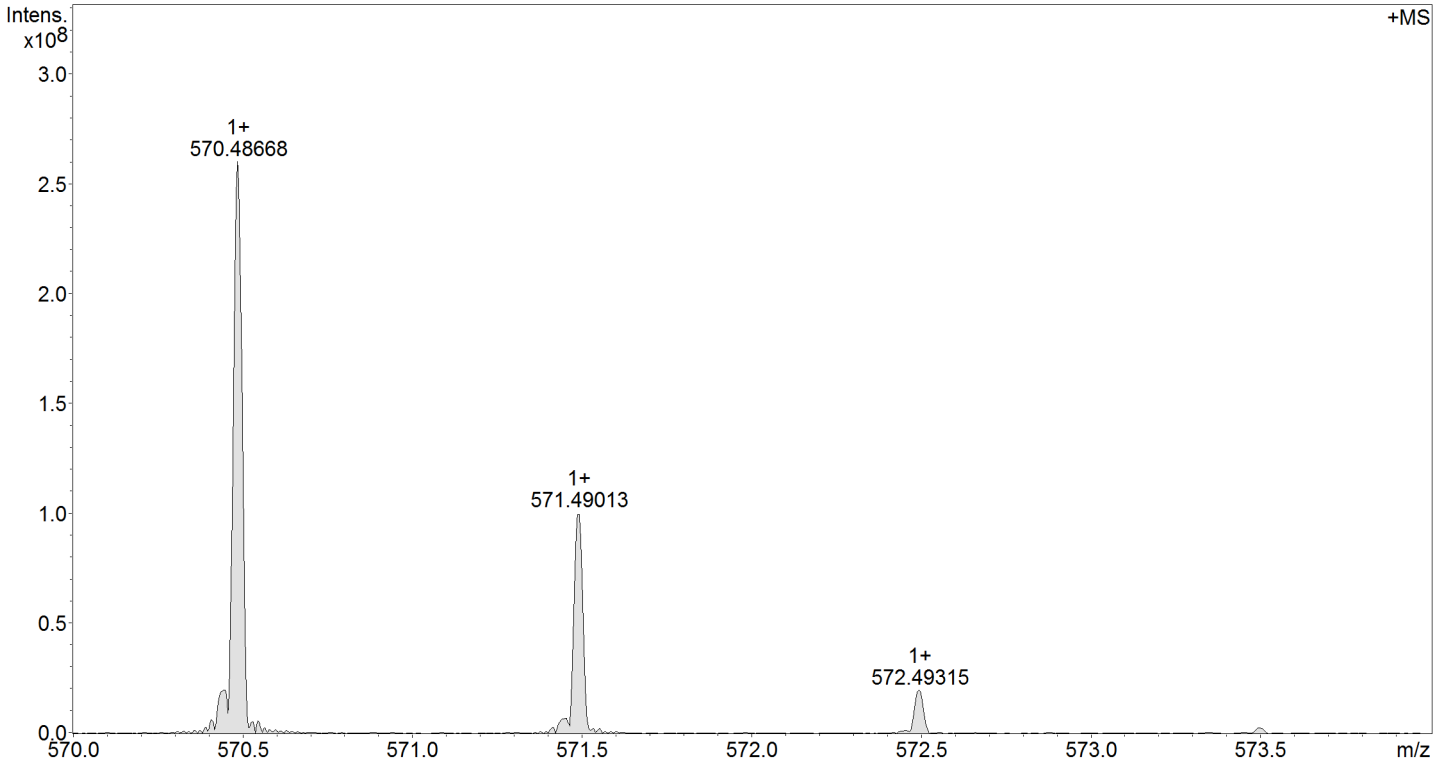
Accurate Mass Measurement

Molecular formula :	C35H65NO3
Abundant Isotopic (theoretical) [M+Na]+ :	570.485666
Monoisotopic (theoretical) [M+Na]+ :	570.485666
(experimental) [M+Na]+ :	570.48668
error (ppm) :	1.6



Bruker 9.4T FTICR MS Analysis Report

Faculty of Science, The Chinese University of Hong Kong



Bruker 9.4T FTICR MS Analysis Report

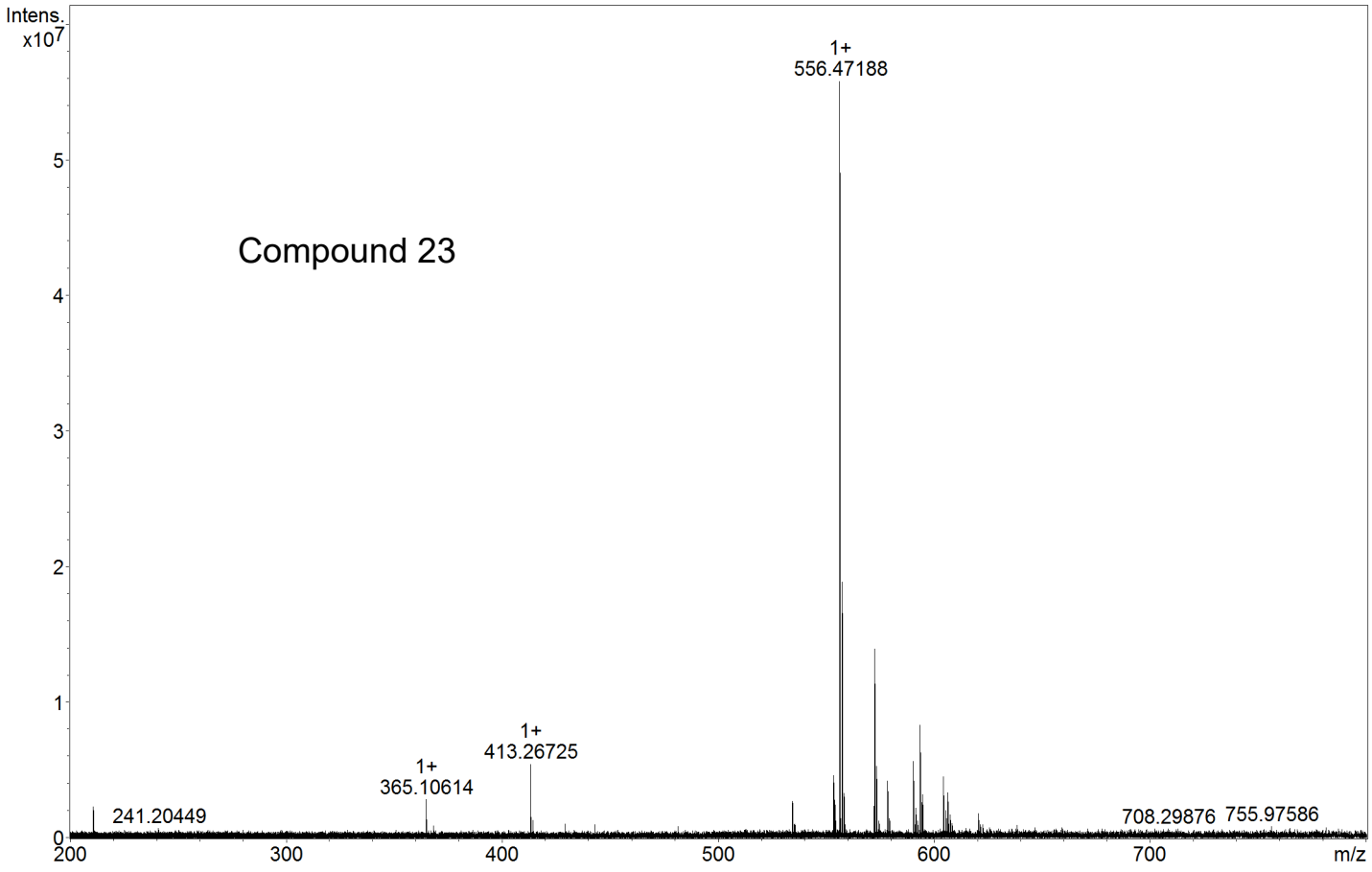
Faculty of Science, The Chinese University of Hong Kong

Analysis Info

Sample Name :	RRC3COOH	Reference No. :	xhfc059
Applicant Name :	Zheng Kun	Analysis Date :	11/3/2016 12:12:17
Analysis Path :	xhfc059_000002.d		
Instrument :	solariX	Polarity	Positive
Method	4_17_mass_range_pos_7T	Acquired Scans	9
Comment :	4.5kV, 140ul/hr, 1.0 bar nebulizer gas, TOF = 0.9		

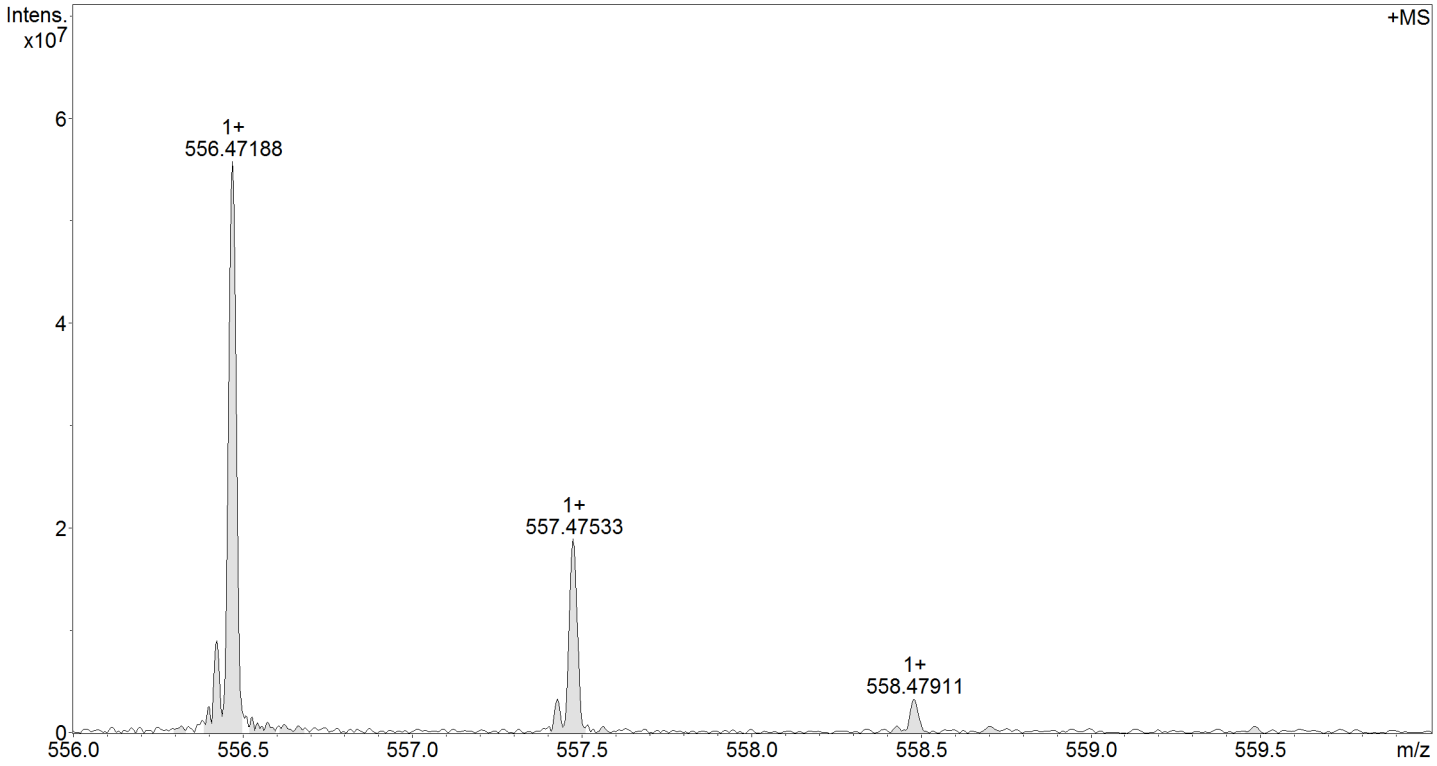
Accurate Mass Measurement

Molecular formula :	C34H63NO3
Abundant Isotopic (theoretical) [M+Na]+ :	556.470016
Monoisotopic (theoretical) [M+Na]+ :	556.470016
(experimental) [M+Na]+ :	556.47188
error (ppm) :	3.3



Bruker 9.4T FTICR MS Analysis Report

Faculty of Science, The Chinese University of Hong Kong



Bruker 9.4T FTICR MS Analysis Report

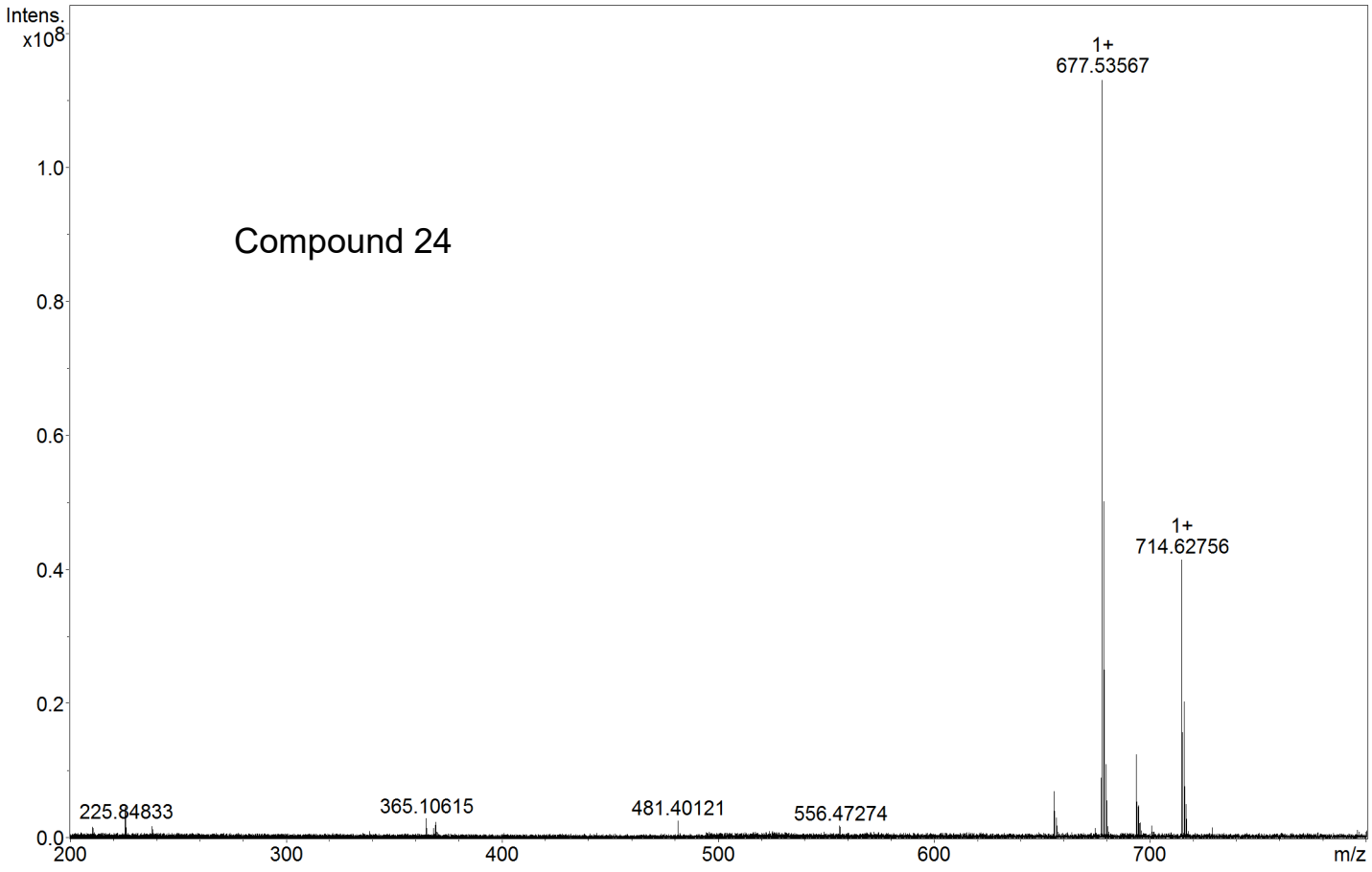
Faculty of Science, The Chinese University of Hong Kong

Analysis Info

Sample Name :	RRBenN3	Reference No. :	xhfc060
Applicant Name :	Zheng Kun	Analysis Date :	11/3/2016 12:26:29
Analysis Path :	xhfc060_000004.d		
Instrument :	solariX	Polarity	Positive
Method	4_17_mass_range_pos_7T	Acquired Scans	11
Comment :	4.5kV, 140ul/hr, 1.0 bar nebulizer gas, TOF = 0.9		

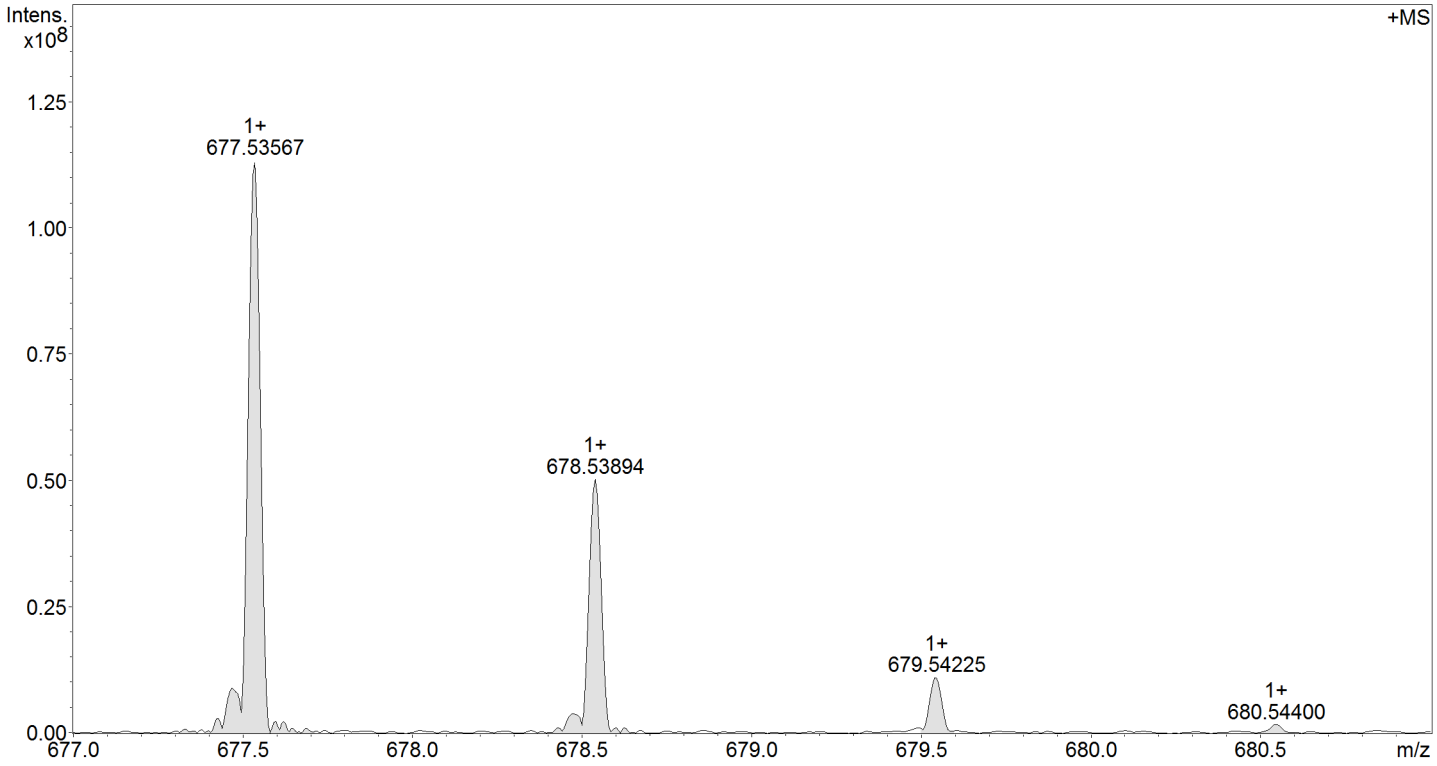
Accurate Mass Measurement

Molecular formula :	C40H70N4O3
Abundant Isotopic (theoretical) [M+Na]+ :	677.534013
Monoisotopic (theoretical) [M+Na]+ :	677.534013
(experimental) [M+Na]+ :	677.53567
error (ppm) :	2.4



Bruker 9.4T FTICR MS Analysis Report

Faculty of Science, The Chinese University of Hong Kong



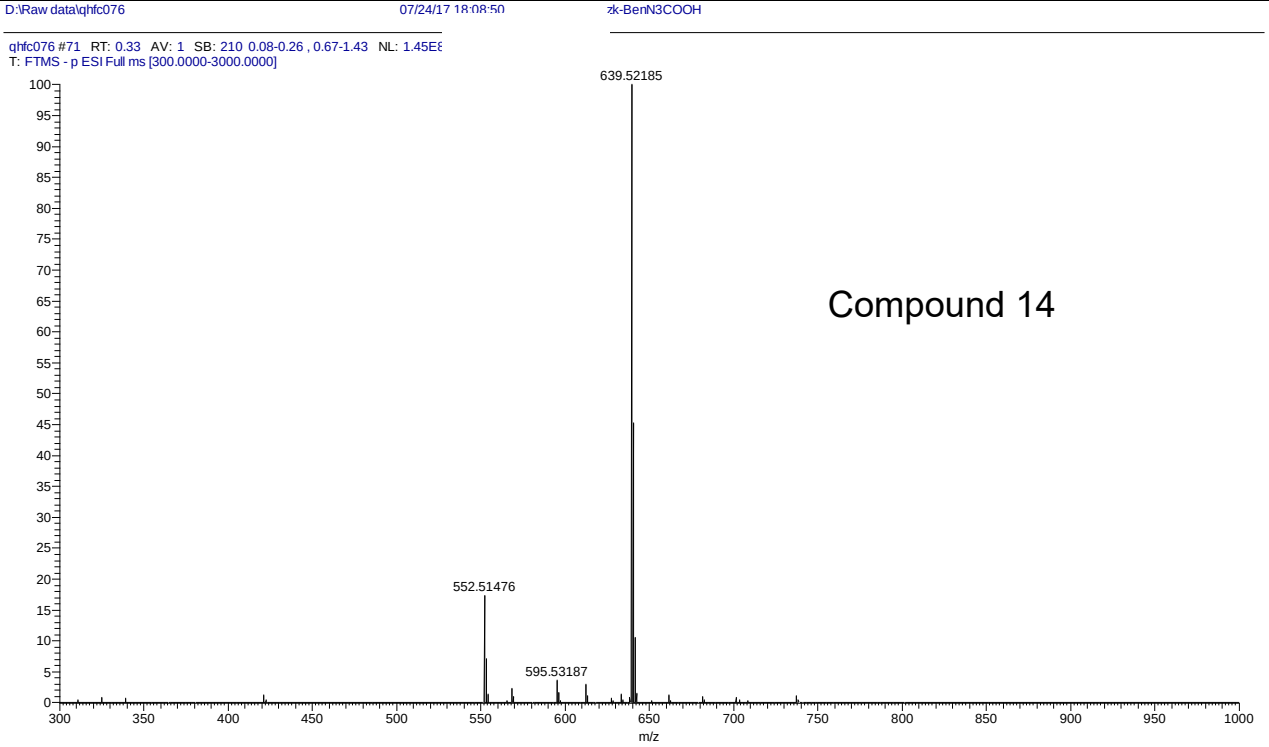
Thermo QEFMS Analysis Report

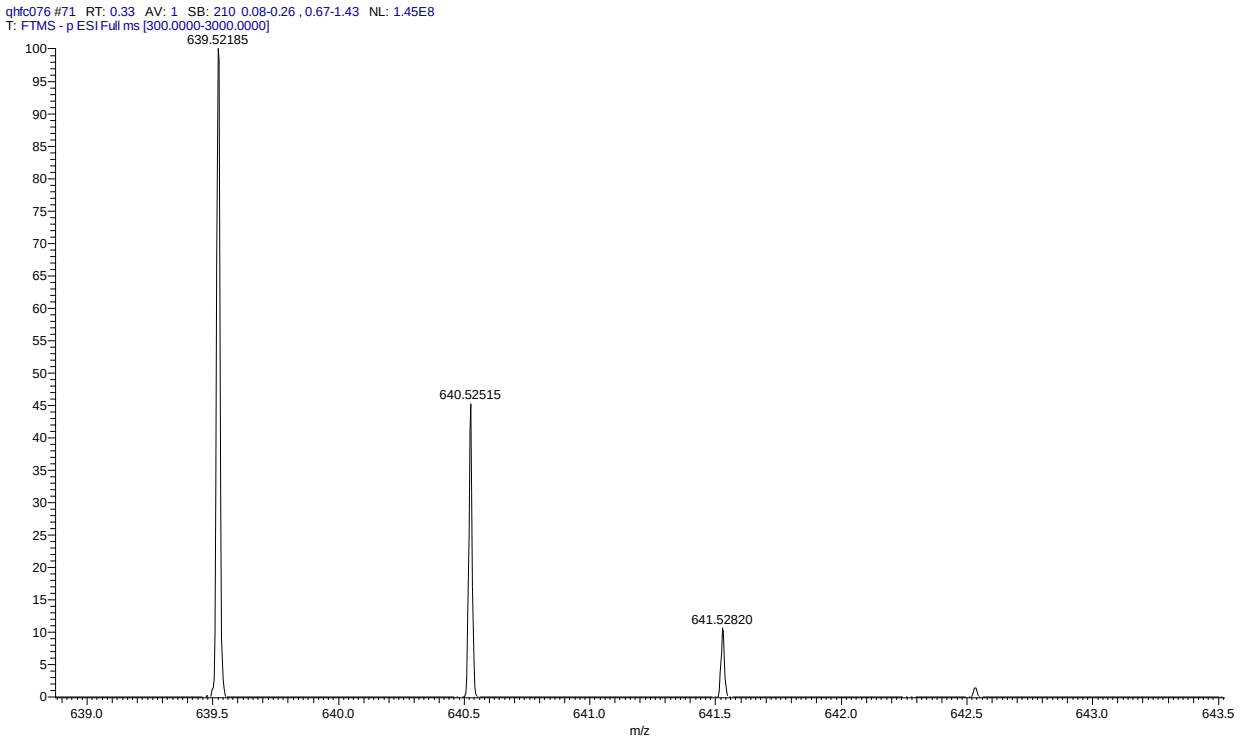
Analysis Info

Sample Name :	Zk-BenN3COOH	Reference No.:	Qhfc076
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Negative
Comment :	ESI neg, 3.0kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{39}H_{68}N_4O_3$
Experimental Mass $[M-H]^-$:	639.52185
Theoretical Mass $[M-H]^-$:	639.52187
Error (ppm) :	0.0





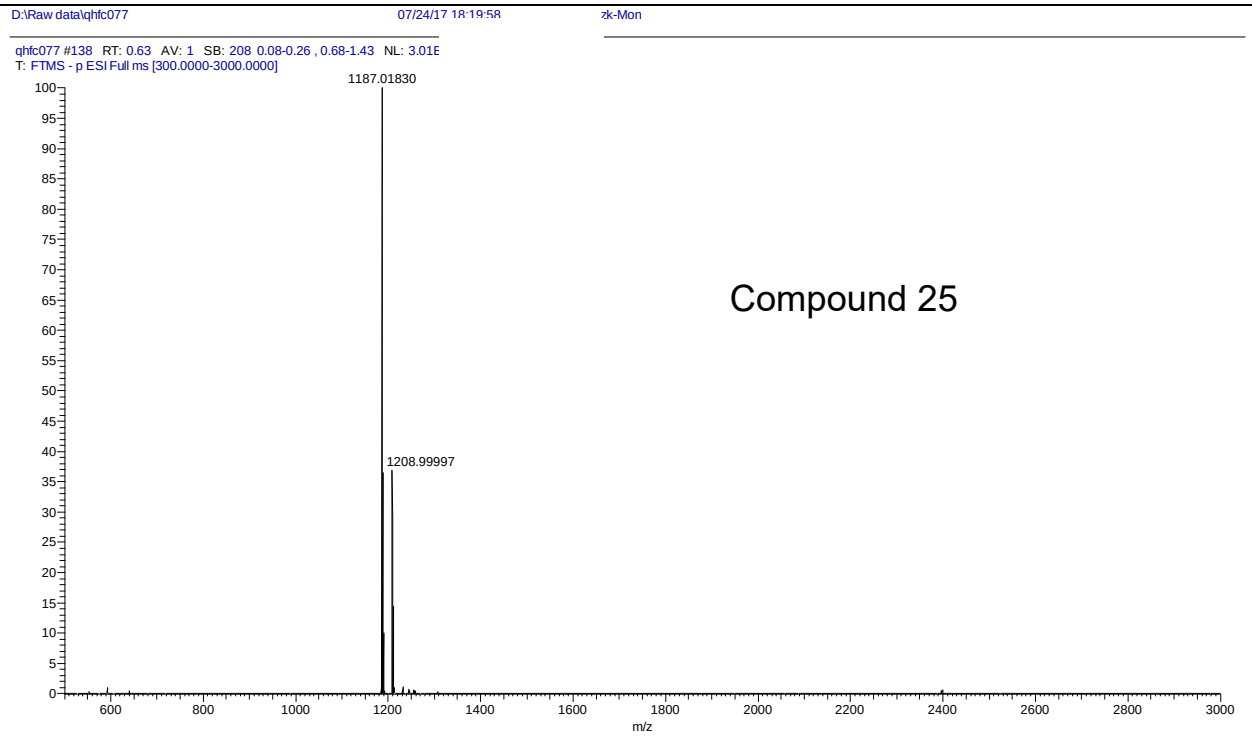
Thermo QEFMS Analysis Report

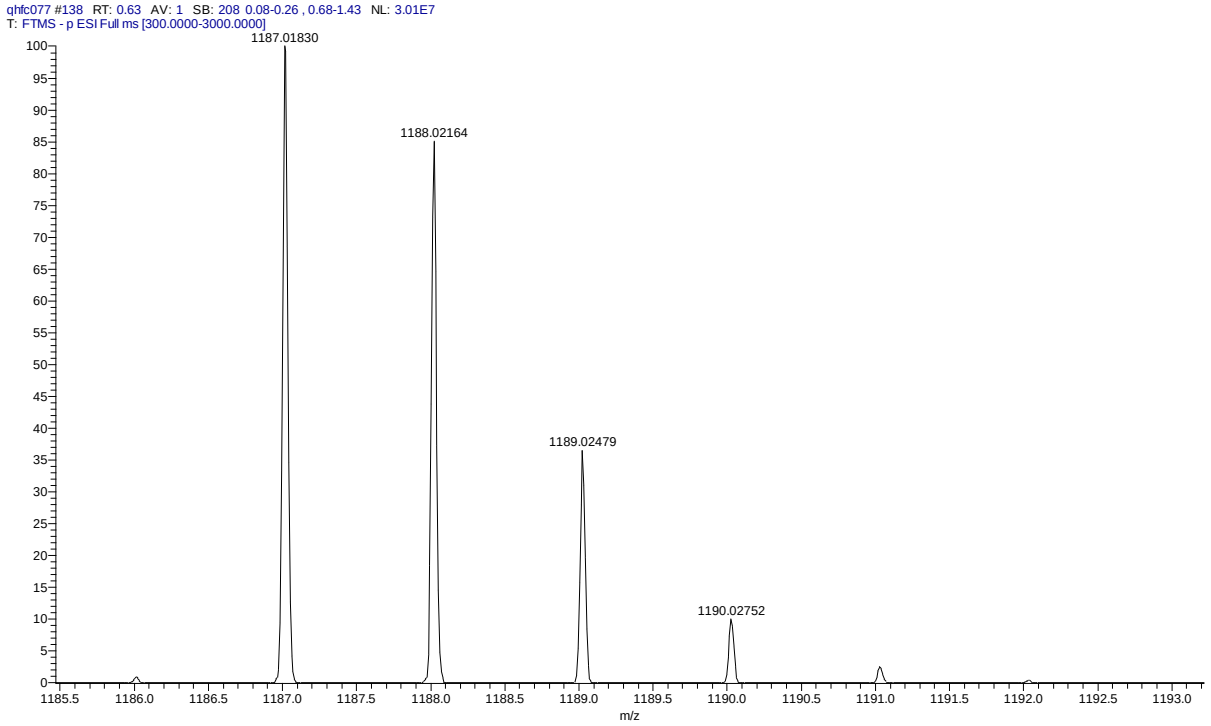
Analysis Info

Sample Name :	Zk-Mon	Reference No.:	Qhfc077
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Negative
Comment :	ESI neg, 3.0kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{74}H_{133}N_5O_6$
Experimental Mass $[M-H]^-$:	1187.01830
Theoretical Mass $[M-H]^-$:	1187.01831
Error (ppm) :	0.0





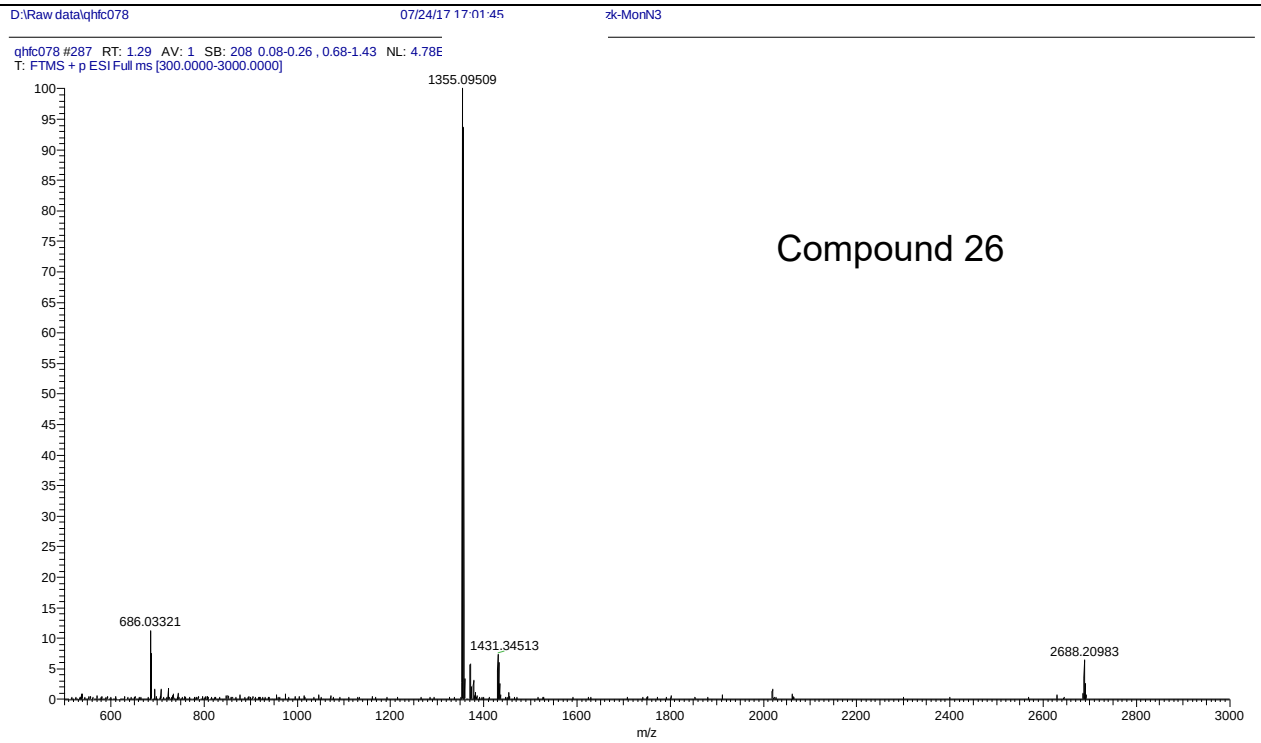
Thermo QEFMS Analysis Report

Analysis Info

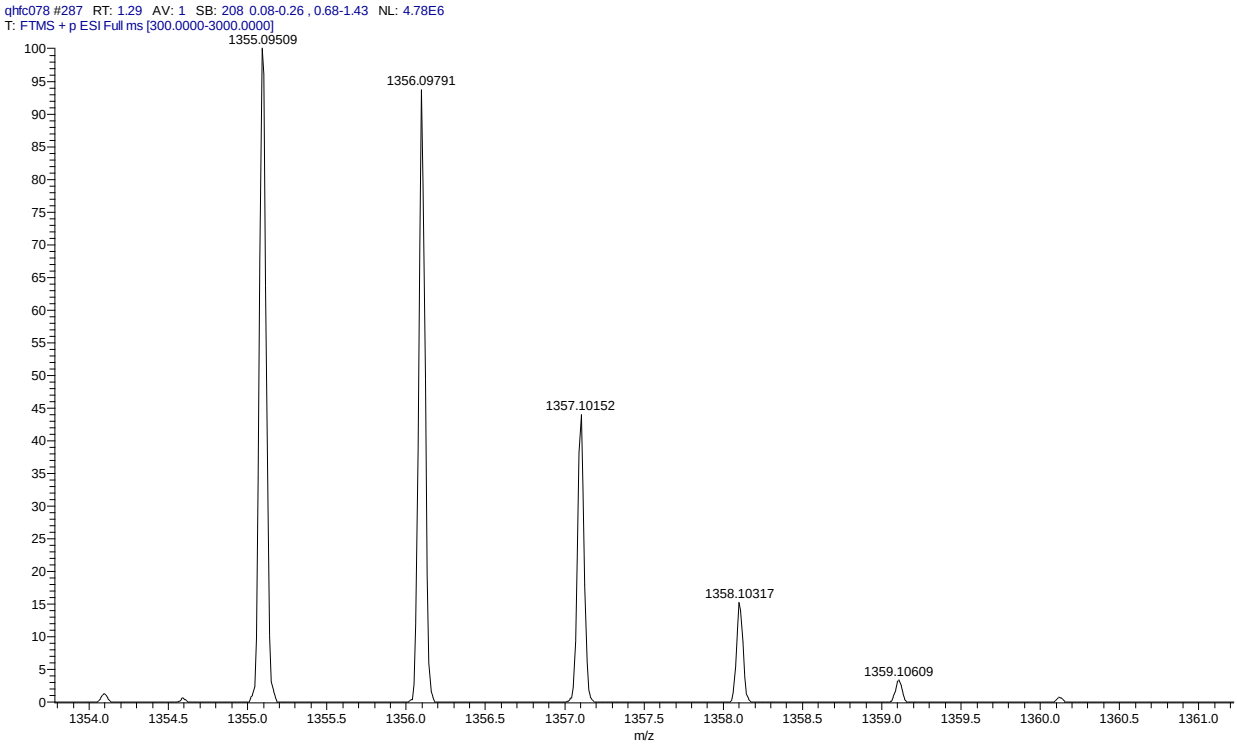
Sample Name :	Zk-MonN3	Reference No.:	Qhfc078
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{82}H_{141}N_9O_5$
Experimental Mass $[M-H]^-$:	1355.09509
Theoretical Mass $[M-H]^-$:	1355.09479
Error (ppm) :	0.2



Compound 26



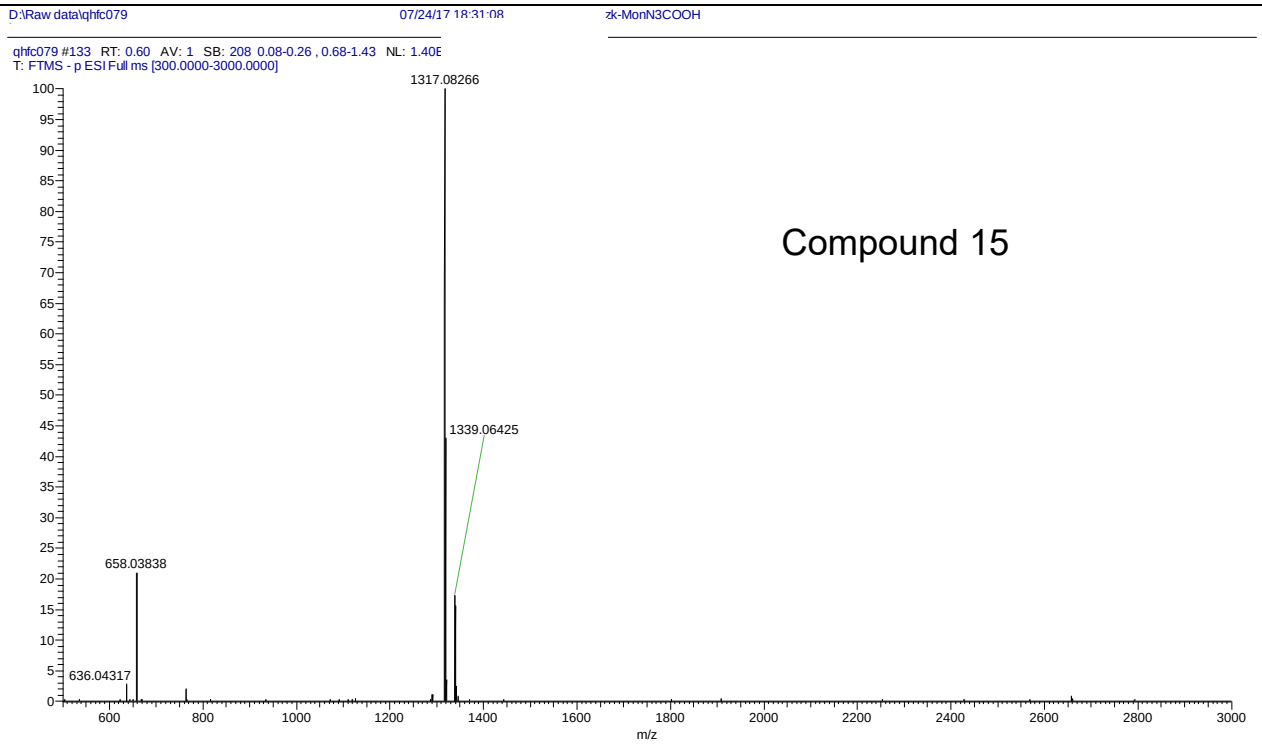
Thermo QEFMS Analysis Report

Analysis Info

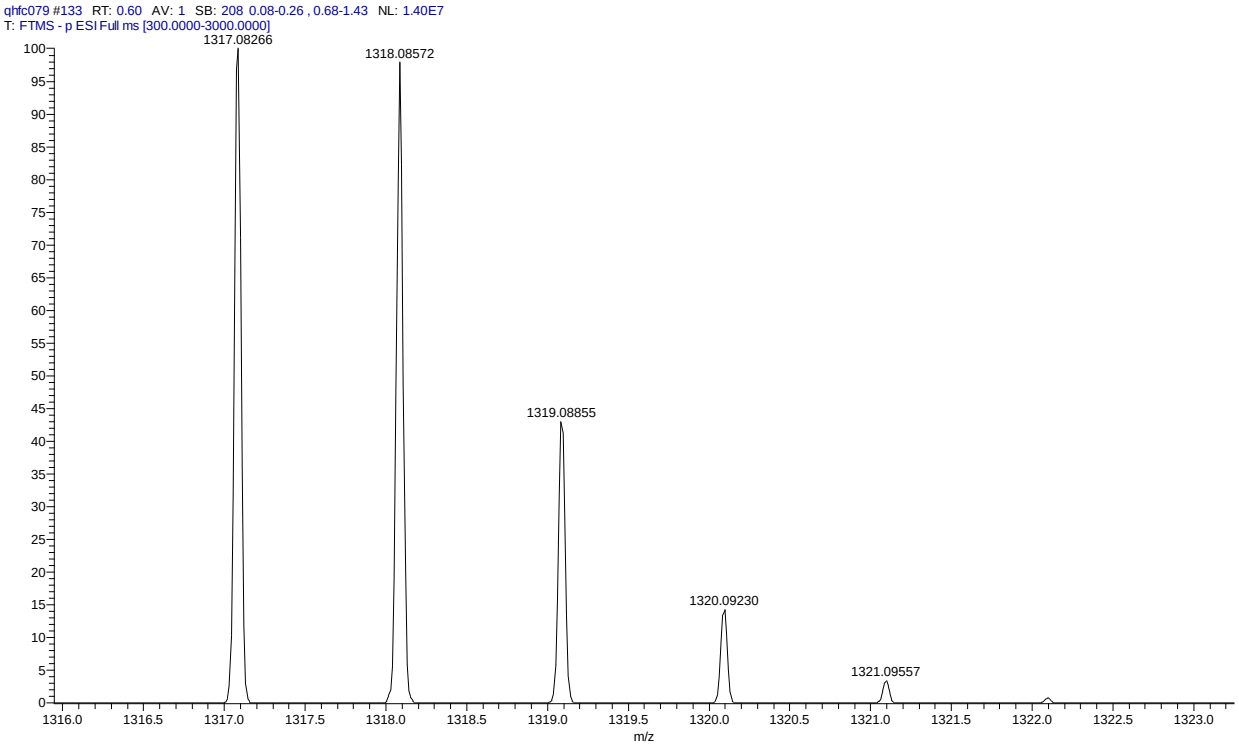
Sample Name :	Zk-MonN3COOH	Reference No.:	Qhfc079
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Negative
Comment :	ESI neg, 3.0kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{81}H_{139}N_9O_5$
Experimental Mass $[M-H]^-$:	1317.08266
Theoretical Mass $[M-H]^-$:	1317.08264
Error (ppm) :	0.0



Compound 15



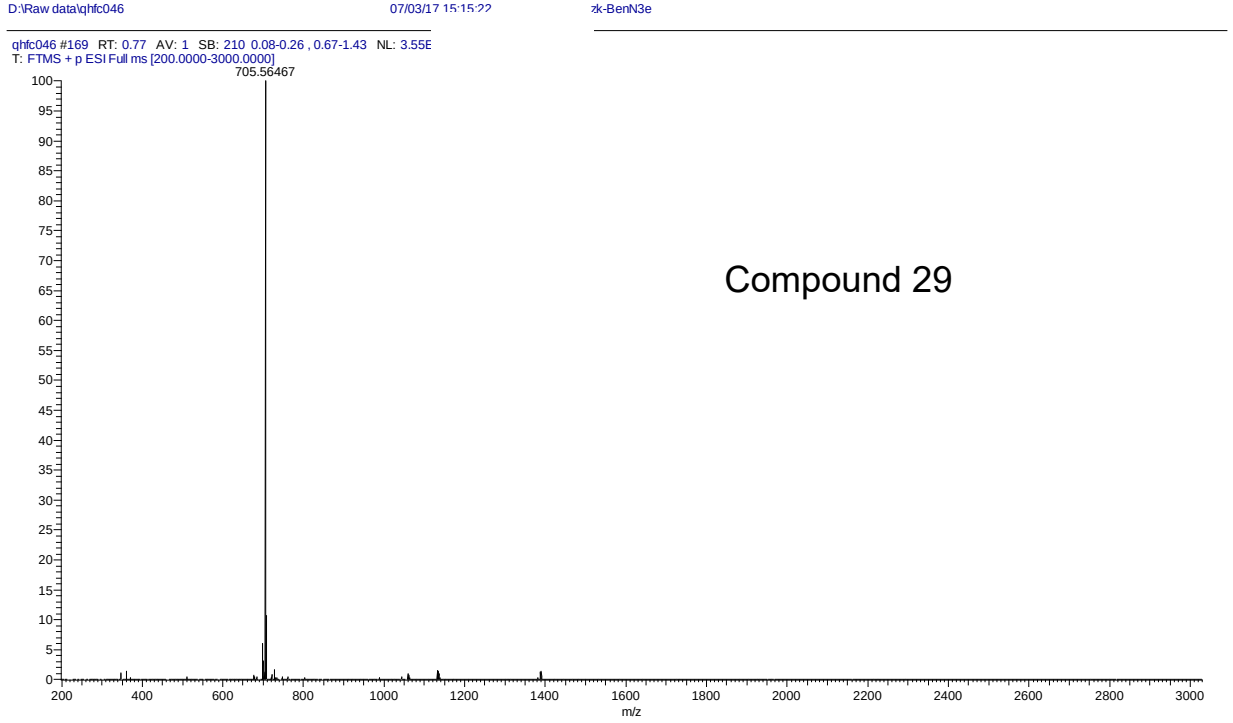
Thermo QEFMS Analysis Report

Analysis Info

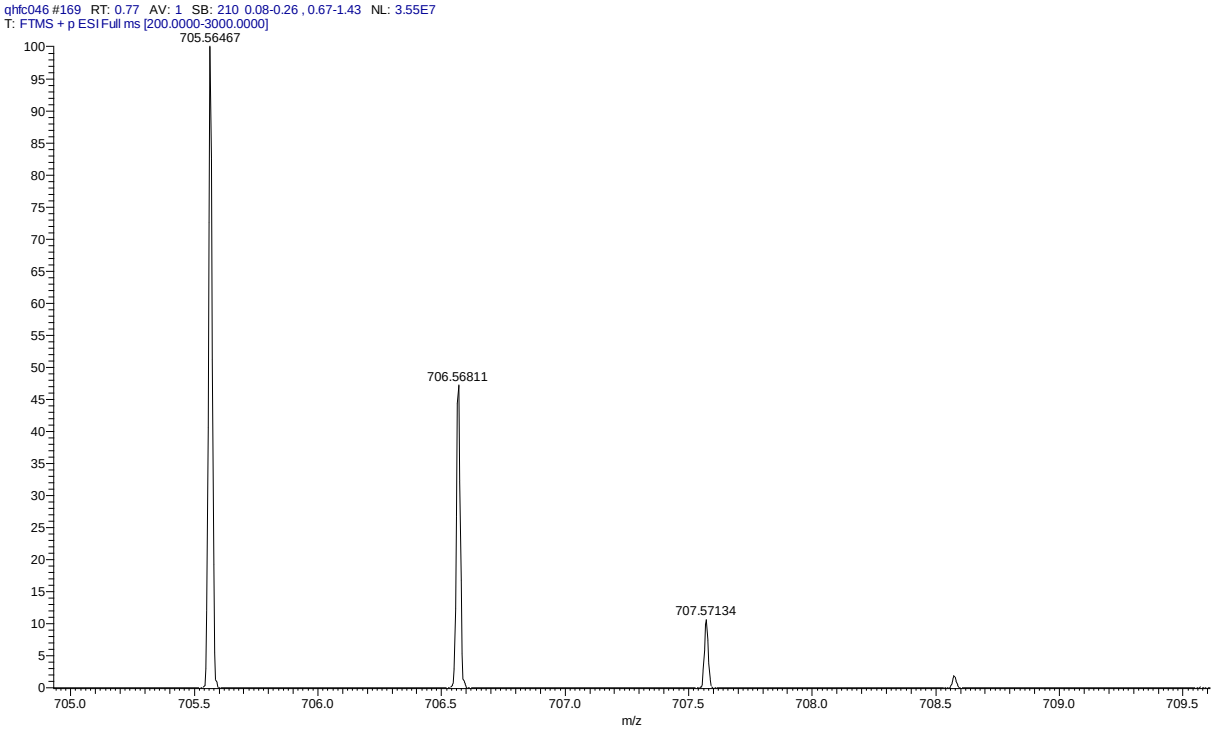
Sample Name :	Zk-BenN3e	Reference No.:	Qhfc046
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{42}H_{74}N_4O_3$
Experimental Mass $[M+Na]^+$:	705.56467
Theoretical Mass $[M+Na]^+$:	705.56531
Error (ppm) :	0.9



Compound 29



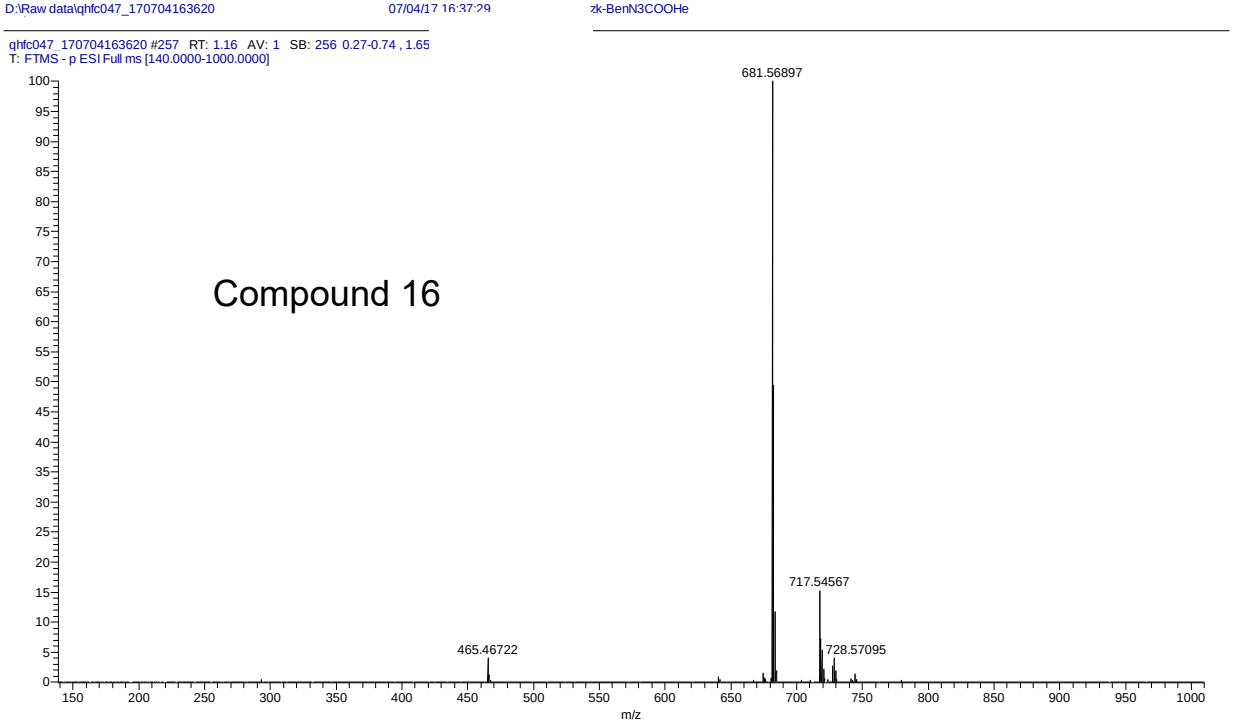
Thermo QEFMS Analysis Report

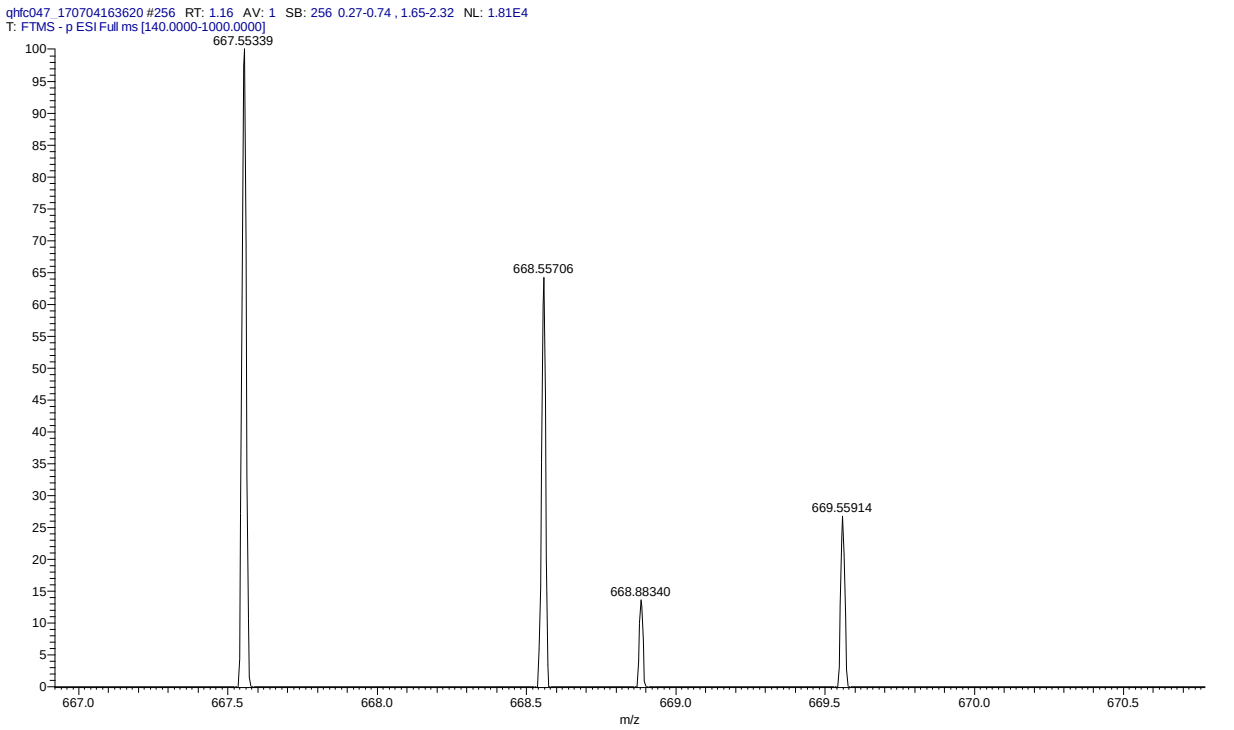
Analysis Info

Sample Name :	Zk-BenN3COOHe	Reference No.:	Qhfc047
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Negative
Comment :	ESI neg, 2.8kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{41}H_{72}N_4O_3$
Experimental Mass $[M-H]^-$:	667.55339
Theoretical Mass $[M-H]^-$:	667.55317
Error (ppm) :	0.3





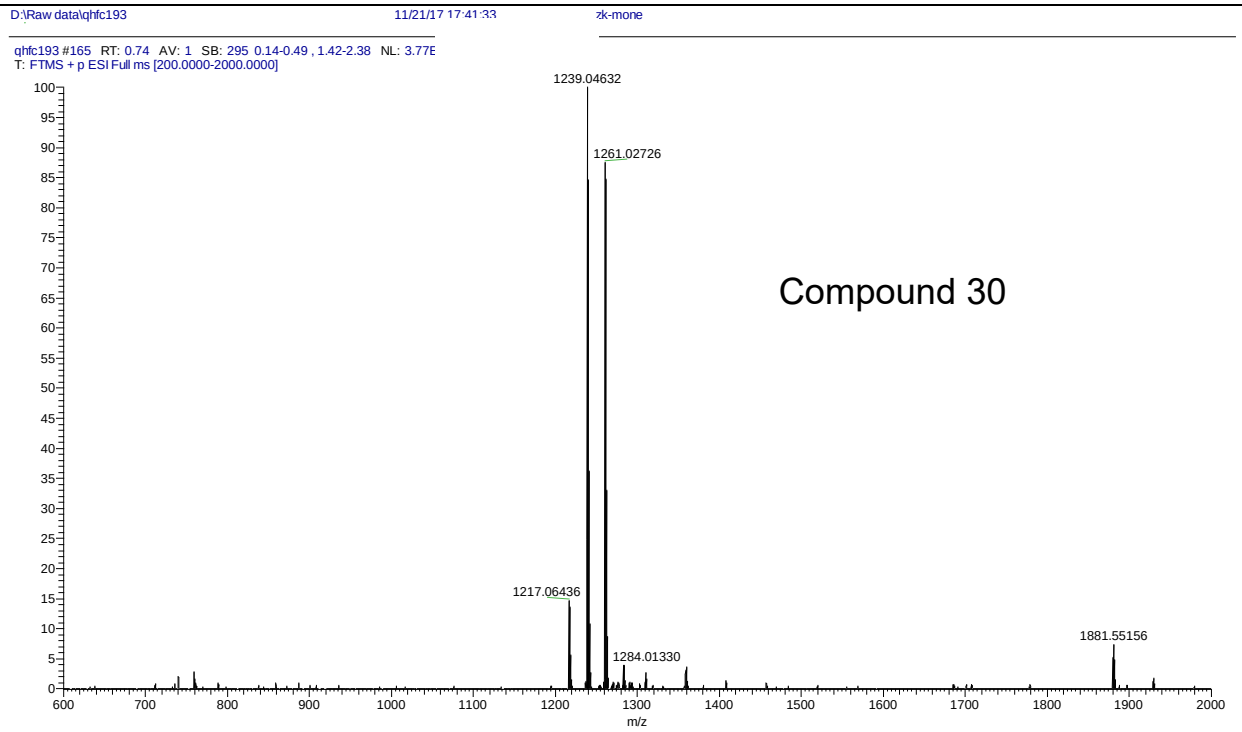
Thermo QEFMS Analysis Report

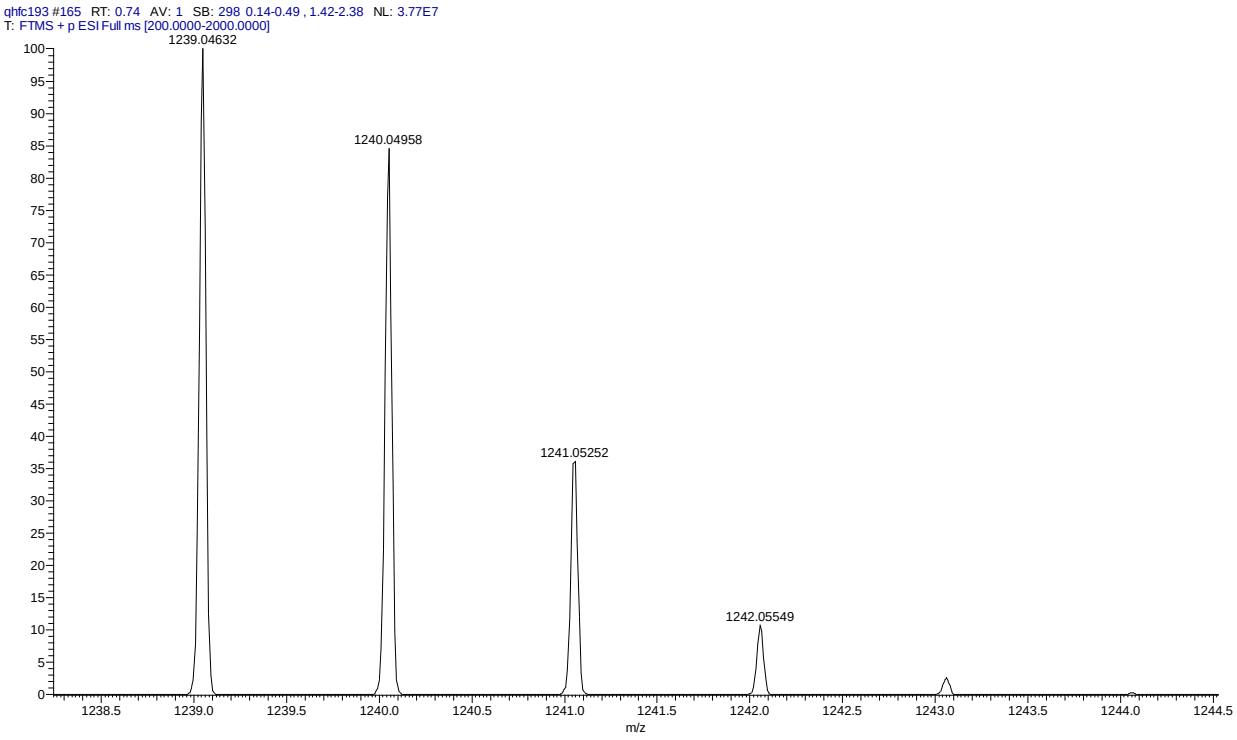
Analysis Info

Sample Name :	Zk-mone	Reference No.:	Qhfc193
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{76}H_{137}N_5O_6$
Experimental Mass $[M+Na]^+$:	1239.04632
Theoretical Mass $[M+Na]^+$:	1239.04611
Error (ppm) :	0.1





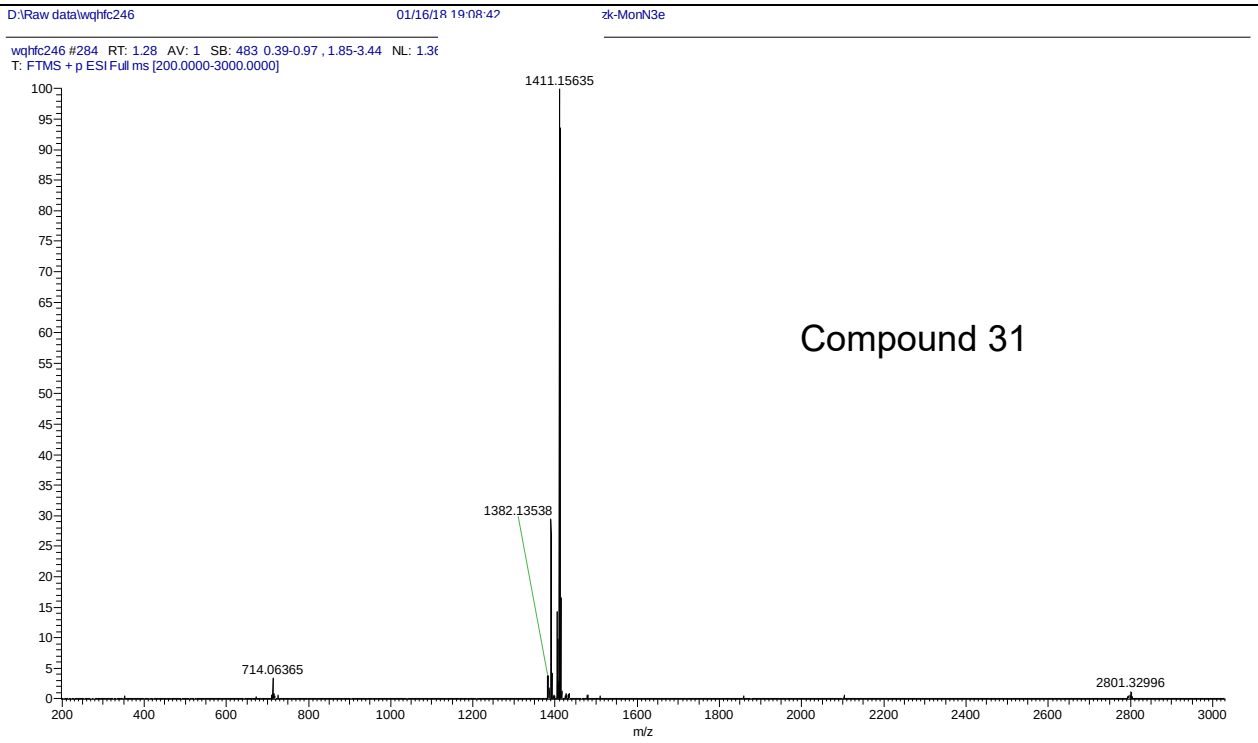
Thermo QEFMS Analysis Report

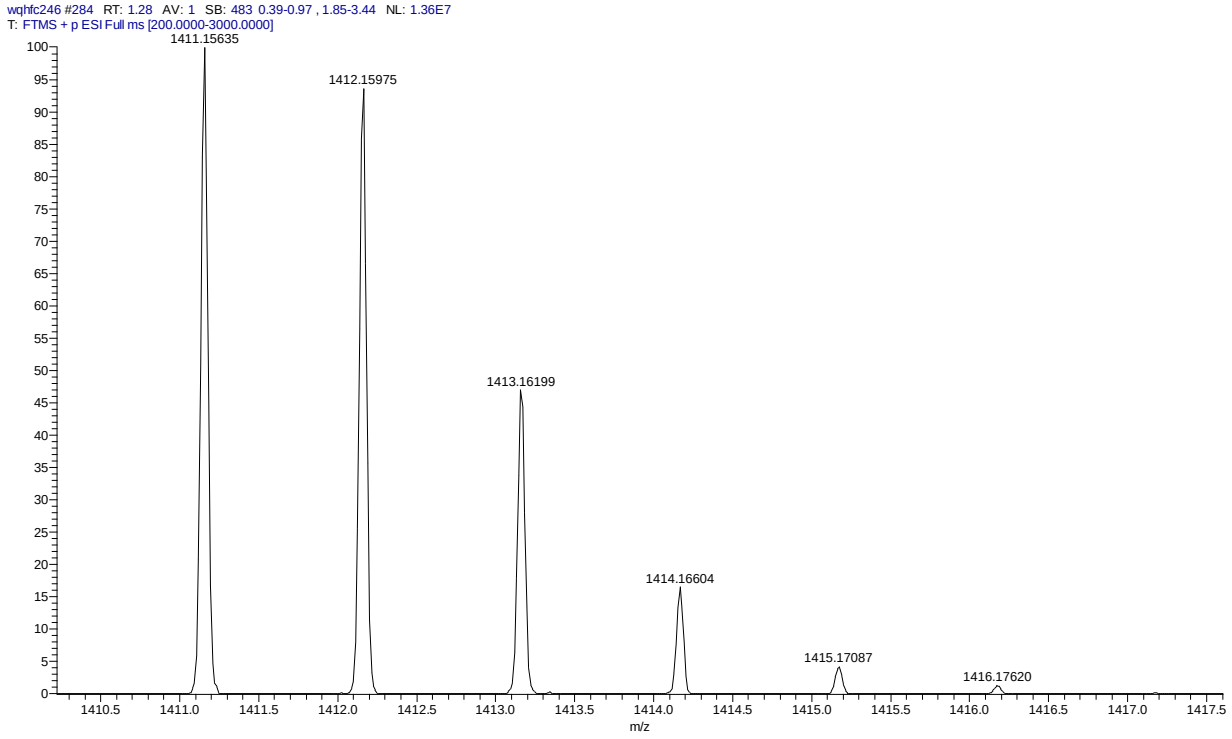
Analysis Info

Sample Name :	Zk-MonN3-e	Reference No.:	Wqhfc246
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{86}H_{149}N_9O_5$
Experimental Mass $[M+Na]^+$:	1411.15635
Theoretical Mass $[M+Na]^+$:	1411.15739
Error (ppm) :	0.7





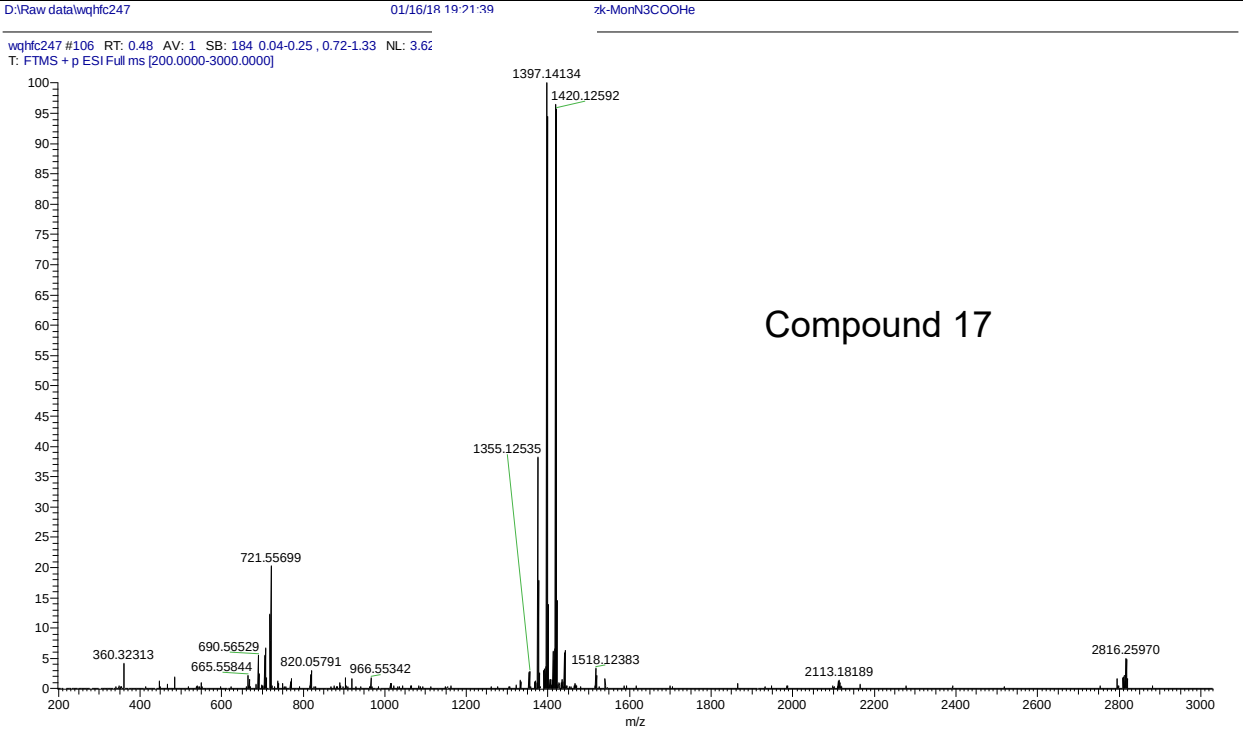
Thermo QEFMS Analysis Report

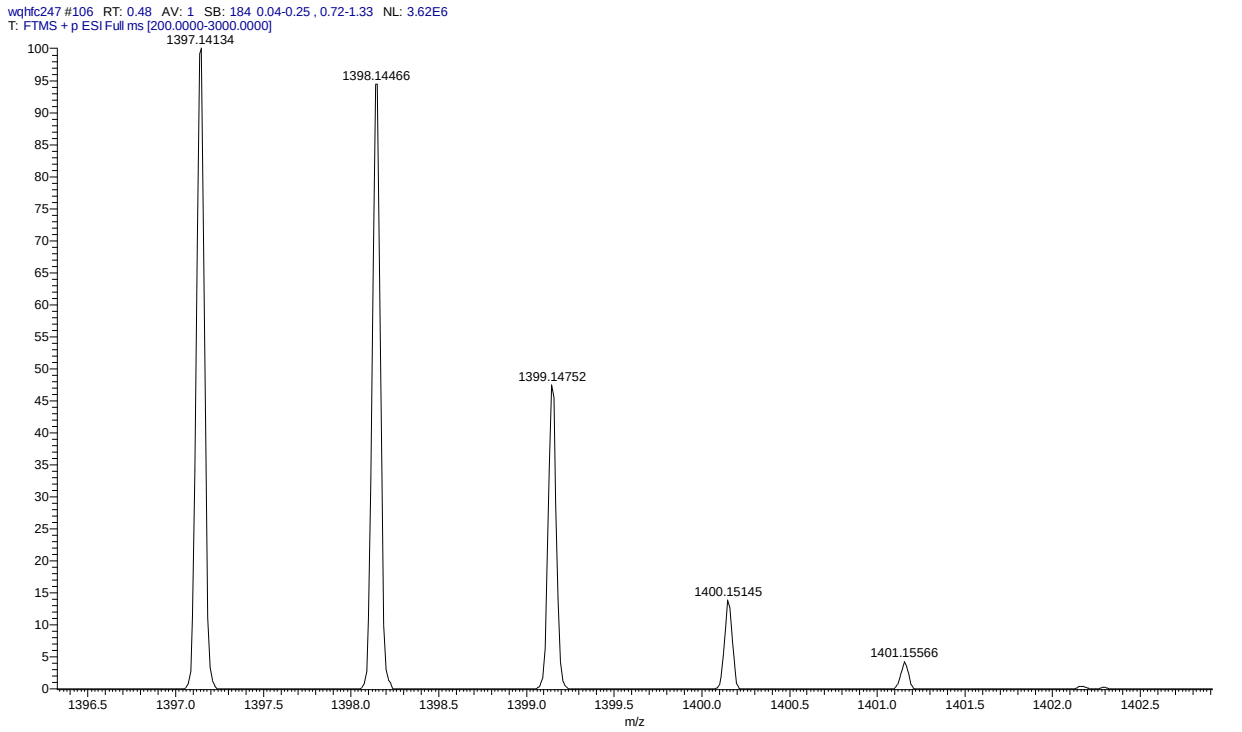
Analysis Info

Sample Name :	Zk-MonN3COOH-e	Reference No.:	Wqhfc247
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{85}H_{147}N_9O_5$
Experimental Mass $[M+Na]^+$:	1397.14134
Theoretical Mass $[M+Na]^+$:	1397.14174
Error (ppm) :	0.2





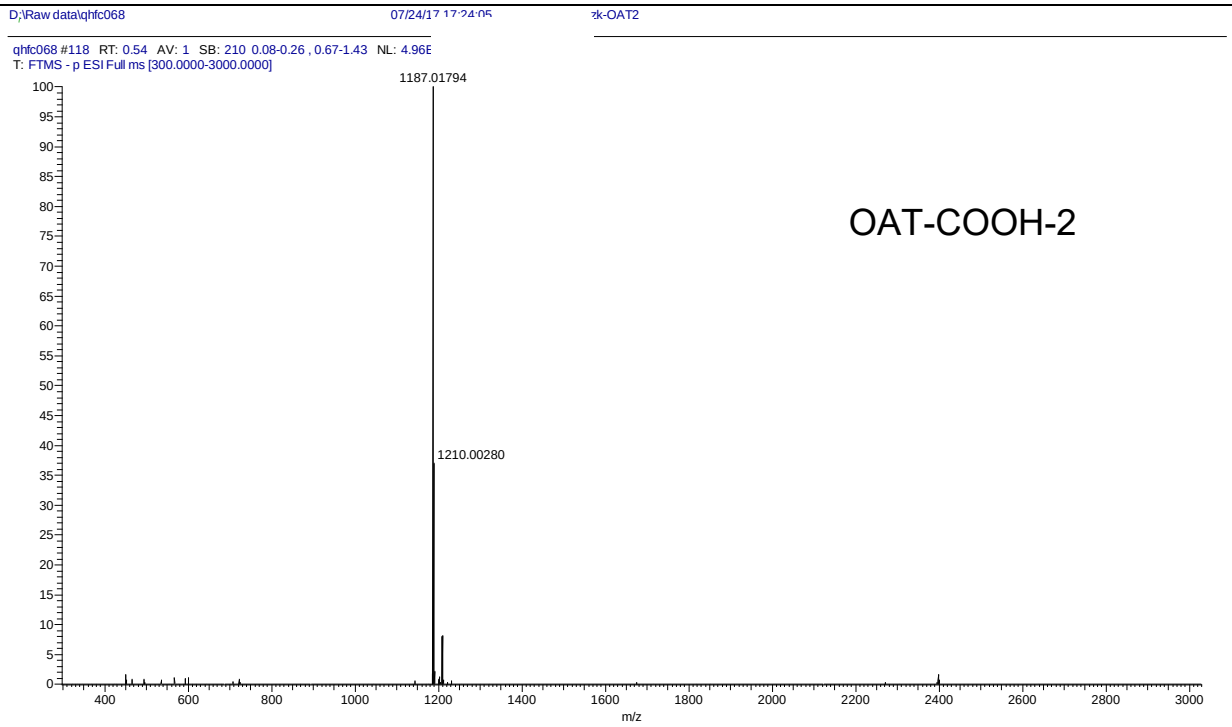
Thermo QEFMS Analysis Report

Analysis Info

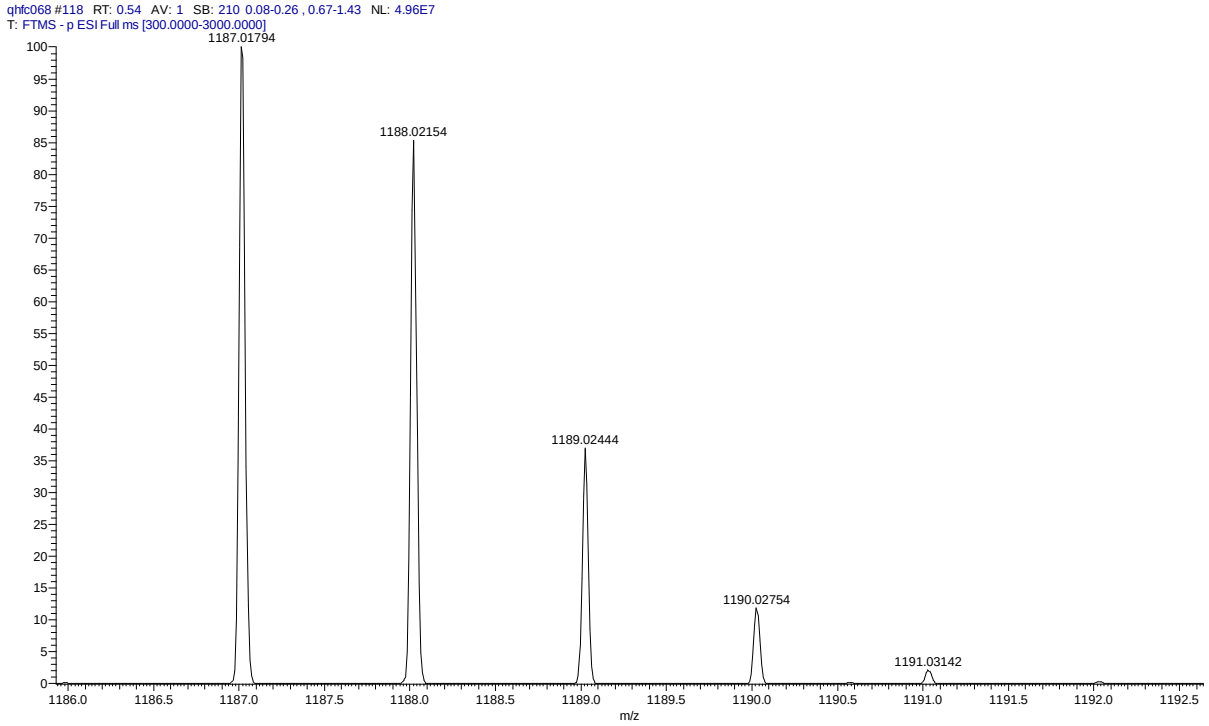
Sample Name :	Zk-OAT2	Reference No.:	Qhfc068
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Negative
Comment :	ESI neg, 3.0kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{74}H_{133}N_5O_6$
Experimental Mass $[M-H]^-$:	1187.01794
Theoretical Mass $[M-H]^-$:	1187.01831
Error (ppm) :	0.3



OAT-COOH-2



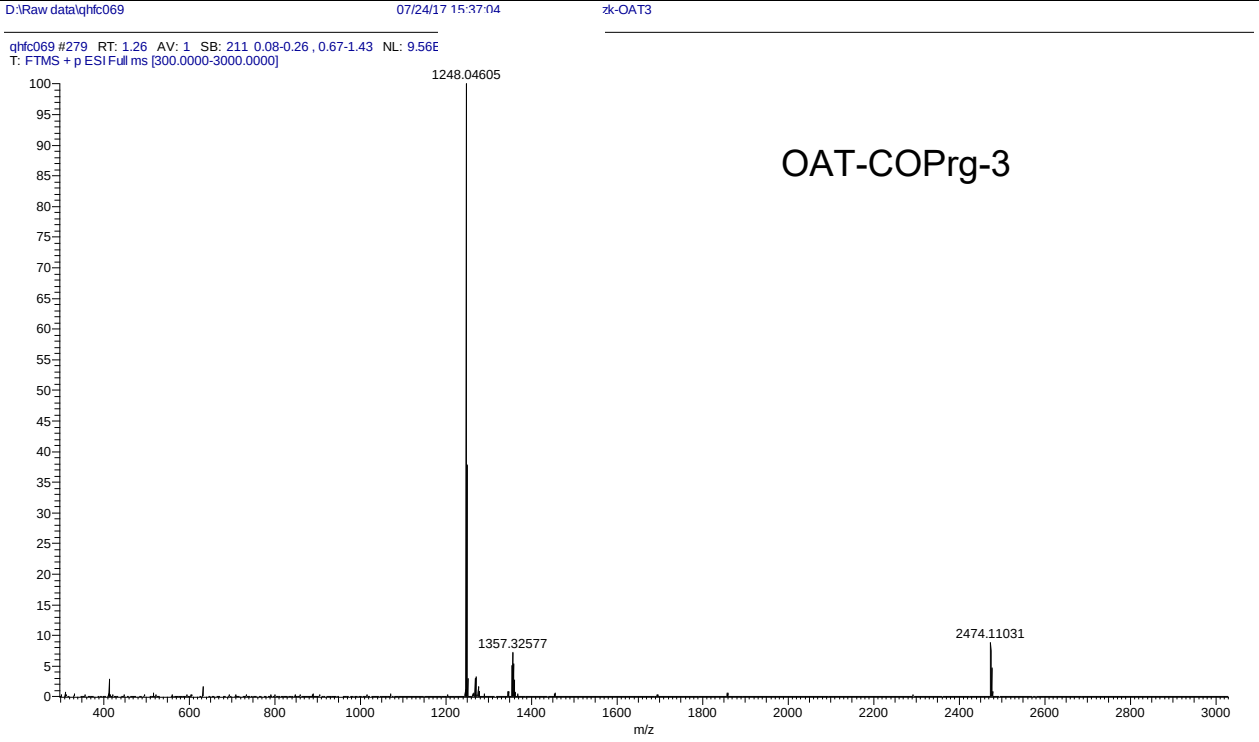
Thermo QEFMS Analysis Report

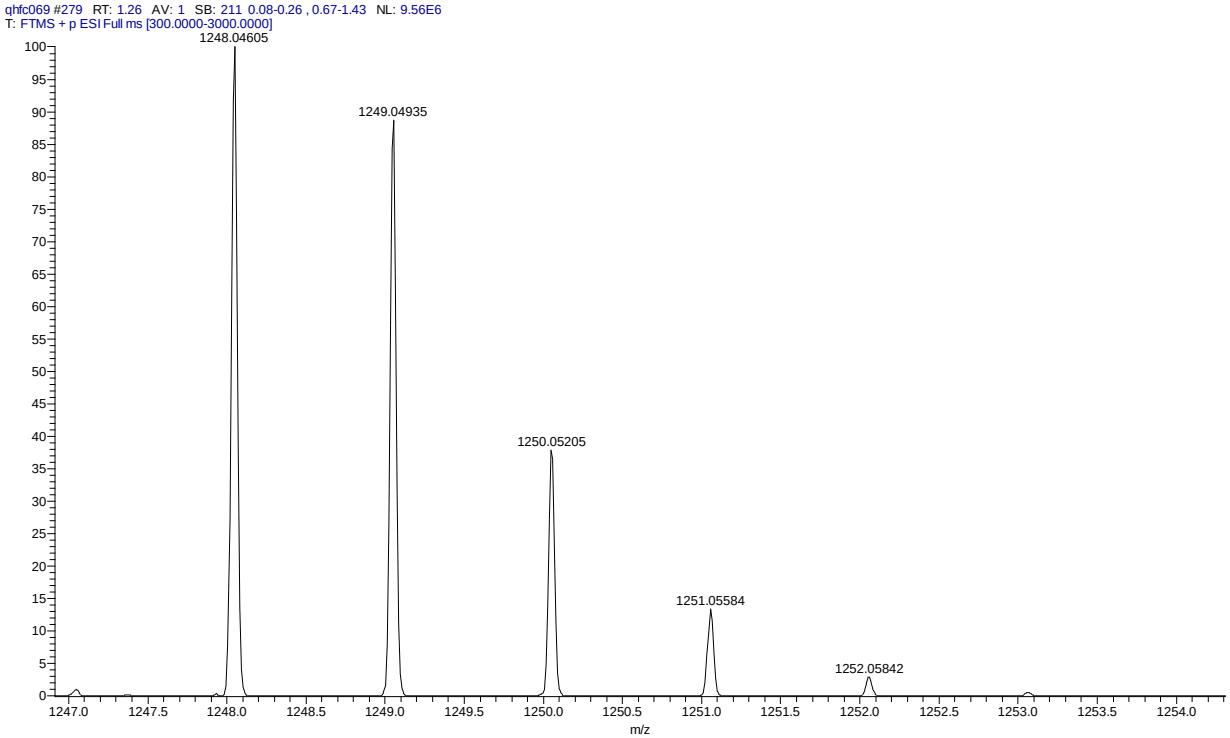
Analysis Info

Sample Name :	Zk-OAT3	Reference No.:	Qhfc069
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{77}H_{136}N_6O_5$
Experimental Mass $[M+Na]^+$:	1248.04605
Theoretical Mass $[M+Na]^+$:	1248.04644
Error (ppm) :	0.3





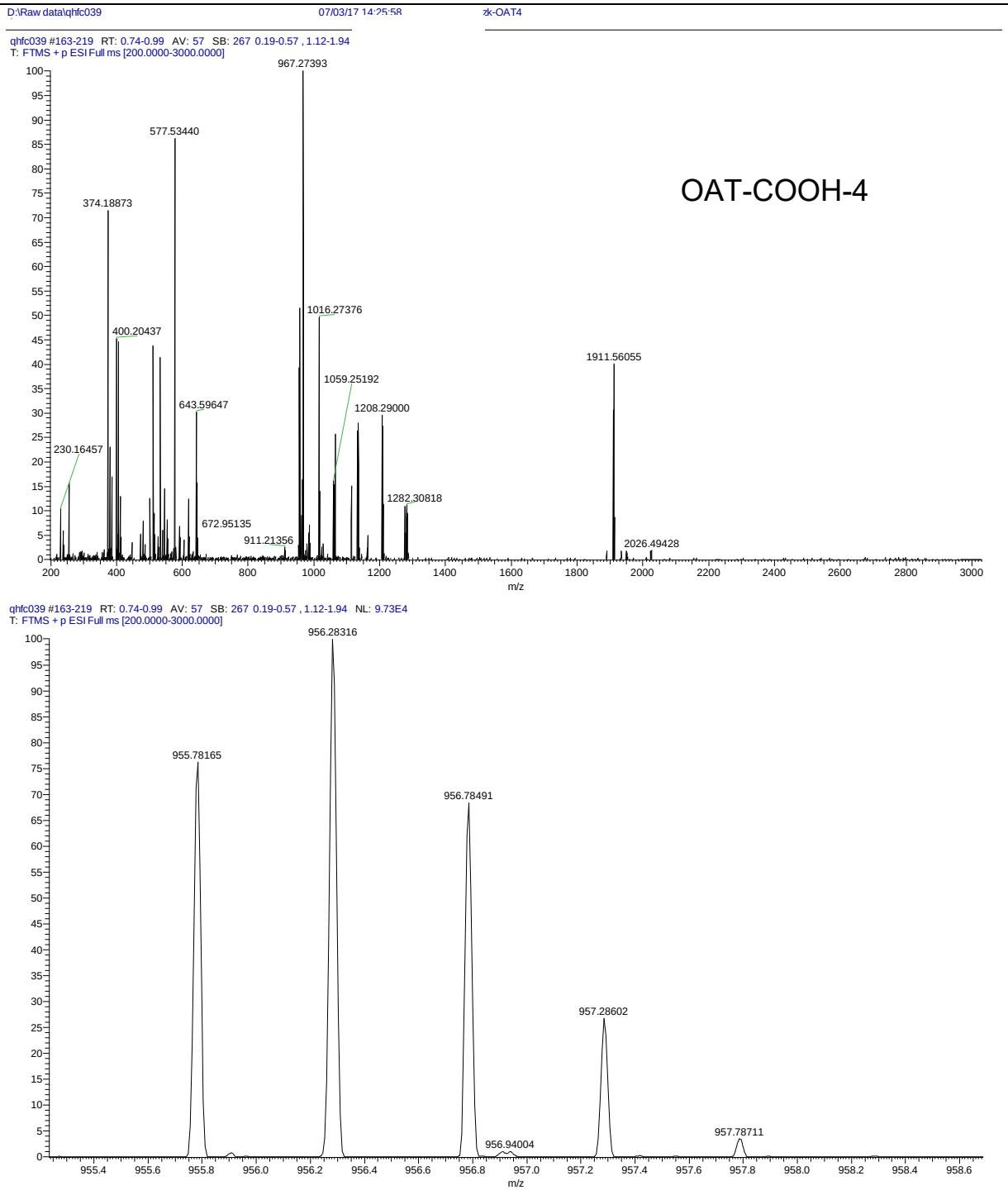
Thermo QEFMS Analysis Report

Analysis Info

Sample Name :	Zk-OAT4	Reference No.:	Qhfc039
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{116}H_{204}N_{10}O_8$
Experimental Mass $[M-H+2Na]^+$, $[M+2Na]^{2+}$, $[M+Na]^+$:	1911.56056, 956.28316, 1889.57585
Theoretical Mass $[M-H+2Na]^+$, $[M+2Na]^{2+}$, $[M+Na]^+$:	1911.56076, 956.28402, 1889.57882
Error (ppm) :	0.1, 0.8, 1.5



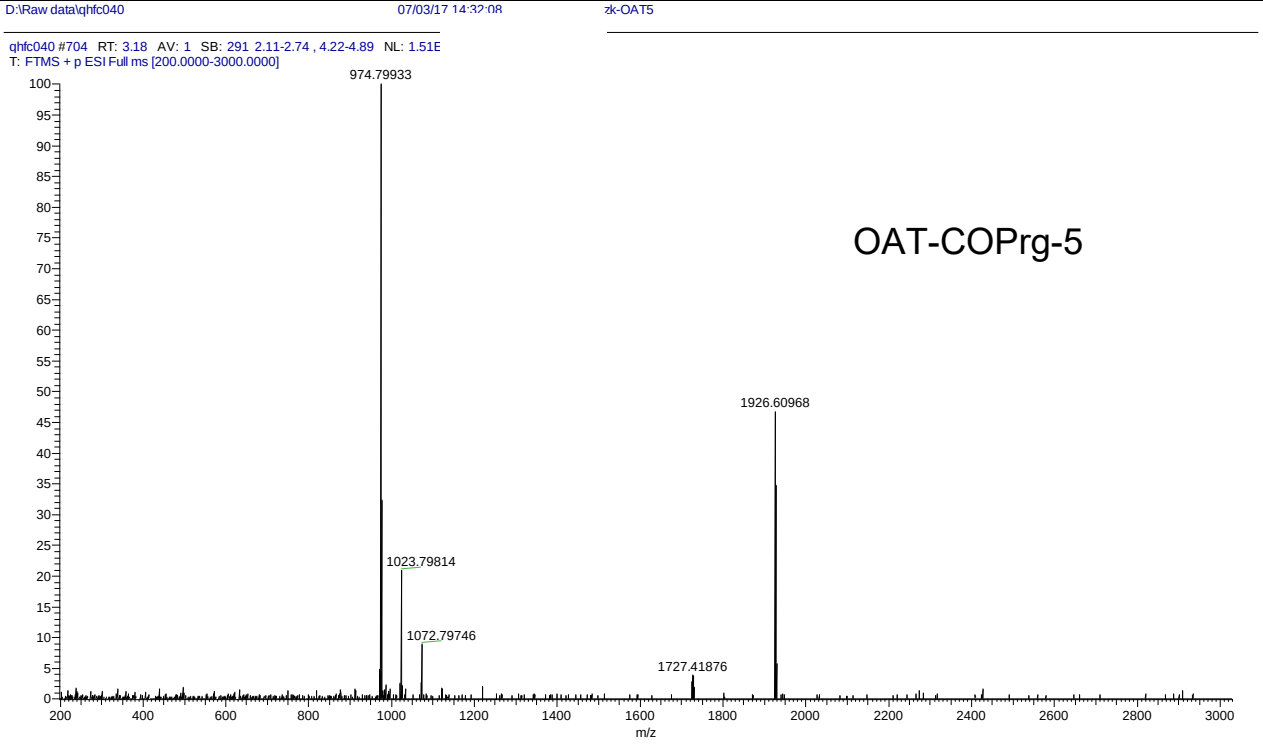
Thermo QEFMS Analysis Report

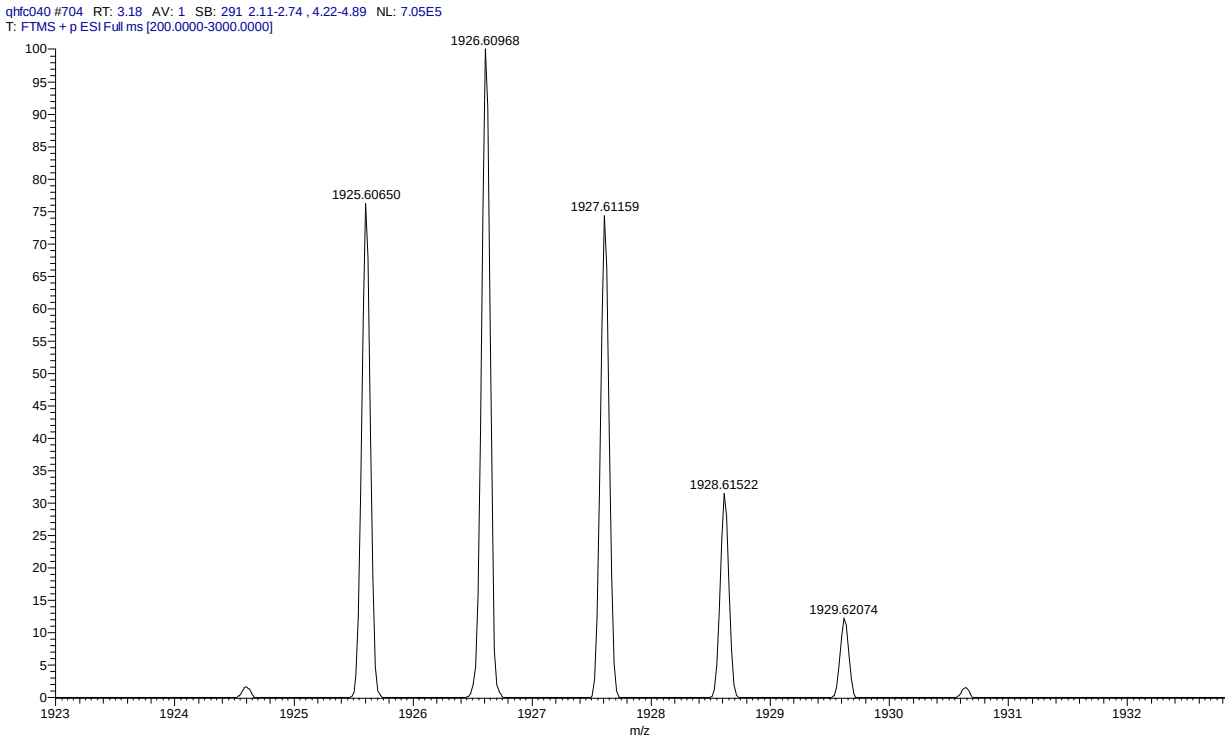
Analysis Info

Sample Name :	Zk-OAT5	Reference No.:	Qhfc040
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{119}H_{207}N_{11}O_7$
Experimental Mass $[M+Na]^+$:	1926.60968
Theoretical Mass $[M+Na]^+$:	1926.61044
Error (ppm) :	0.3





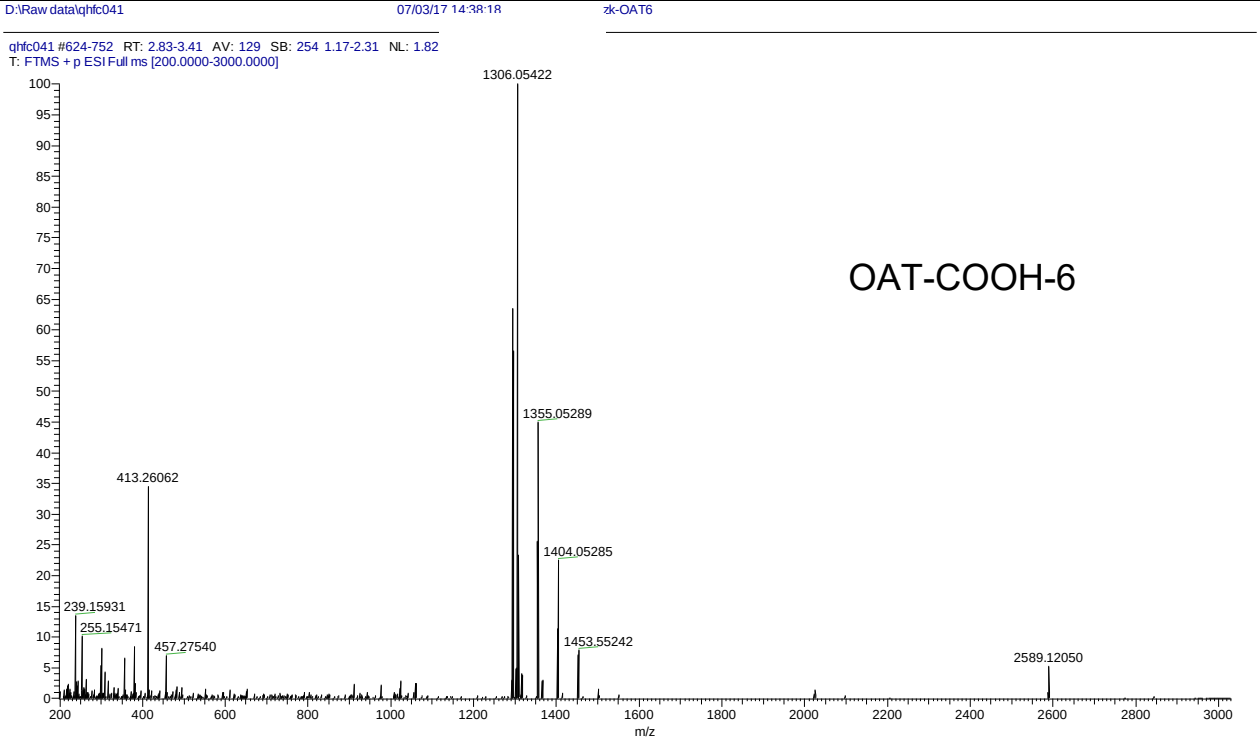
Thermo QEFMS Analysis Report

Analysis Info

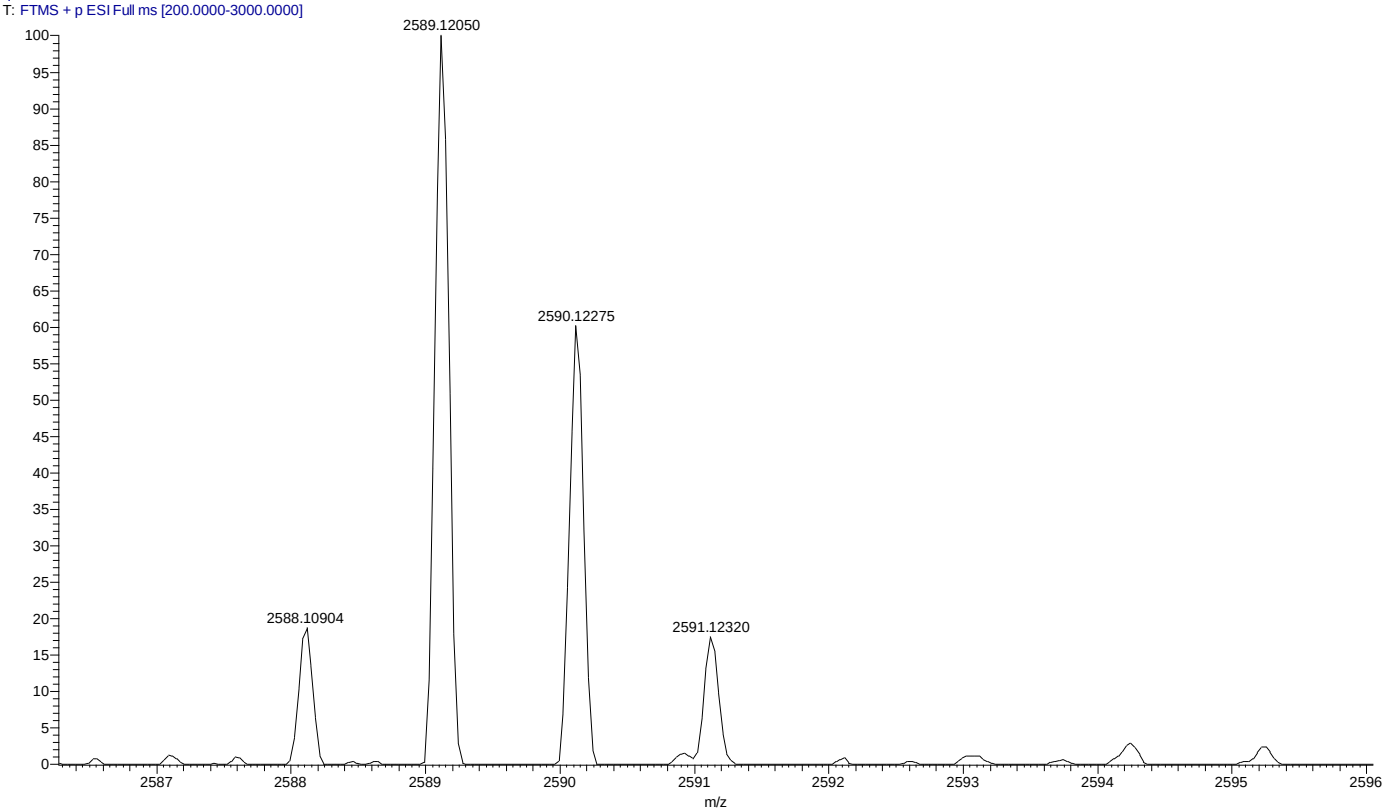
Sample Name :	Zk-OAT6	Reference No.:	Qhfc041
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

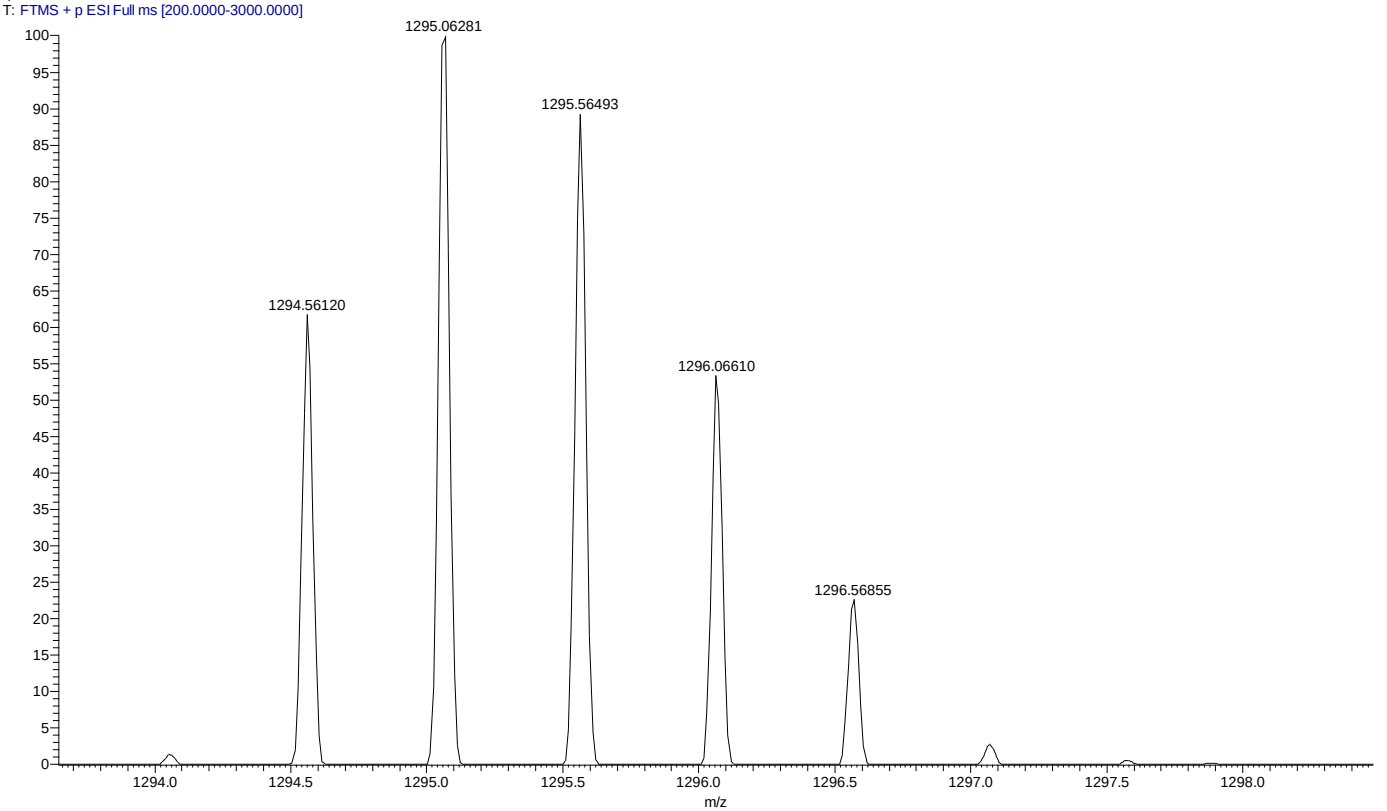
Molecular formula :	$C_{158}H_{275}N_{15}O_{10}$
Experimental Mass $[M-H+2Na]^+$, $[M+2Na]^{2+}$:	2589.12050, 1295.06281
Theoretical Mass $[M-H+2Na]^+$, $[M+2Na]^{2+}$:	2589.12152, 1295.06440
Error (ppm) :	0.3, 1.2



qhfc041 #625-753 RT: 2.83-3.41 AV: 129 SB: 253 1.17-2.31 NL: 9.65E3
T: FTMS + p ESI Full ms [200.0000-3000.0000]



qhfc041 #624-752 RT: 2.83-3.41 AV: 129 SB: 254 1.17-2.31 NL: 1.15E5
T: FTMS + p ESI Full ms [200.0000-3000.0000]



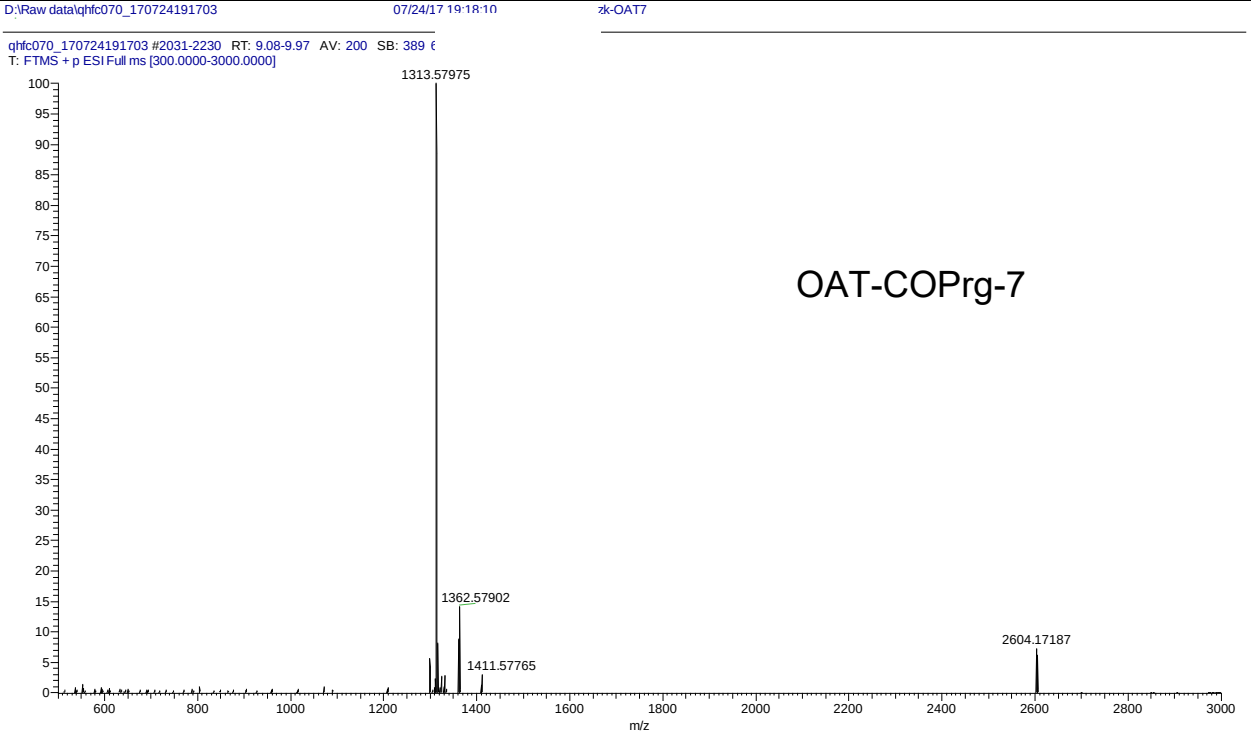
Thermo QEFMS Analysis Report

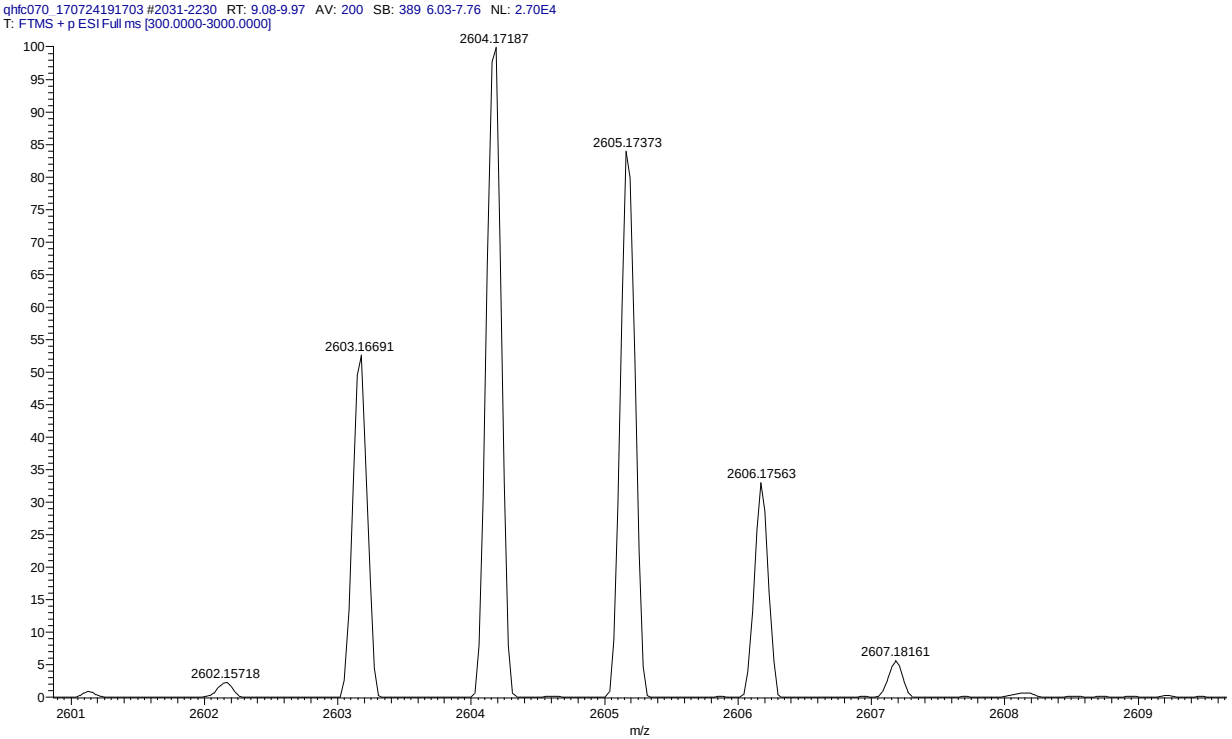
Analysis Info

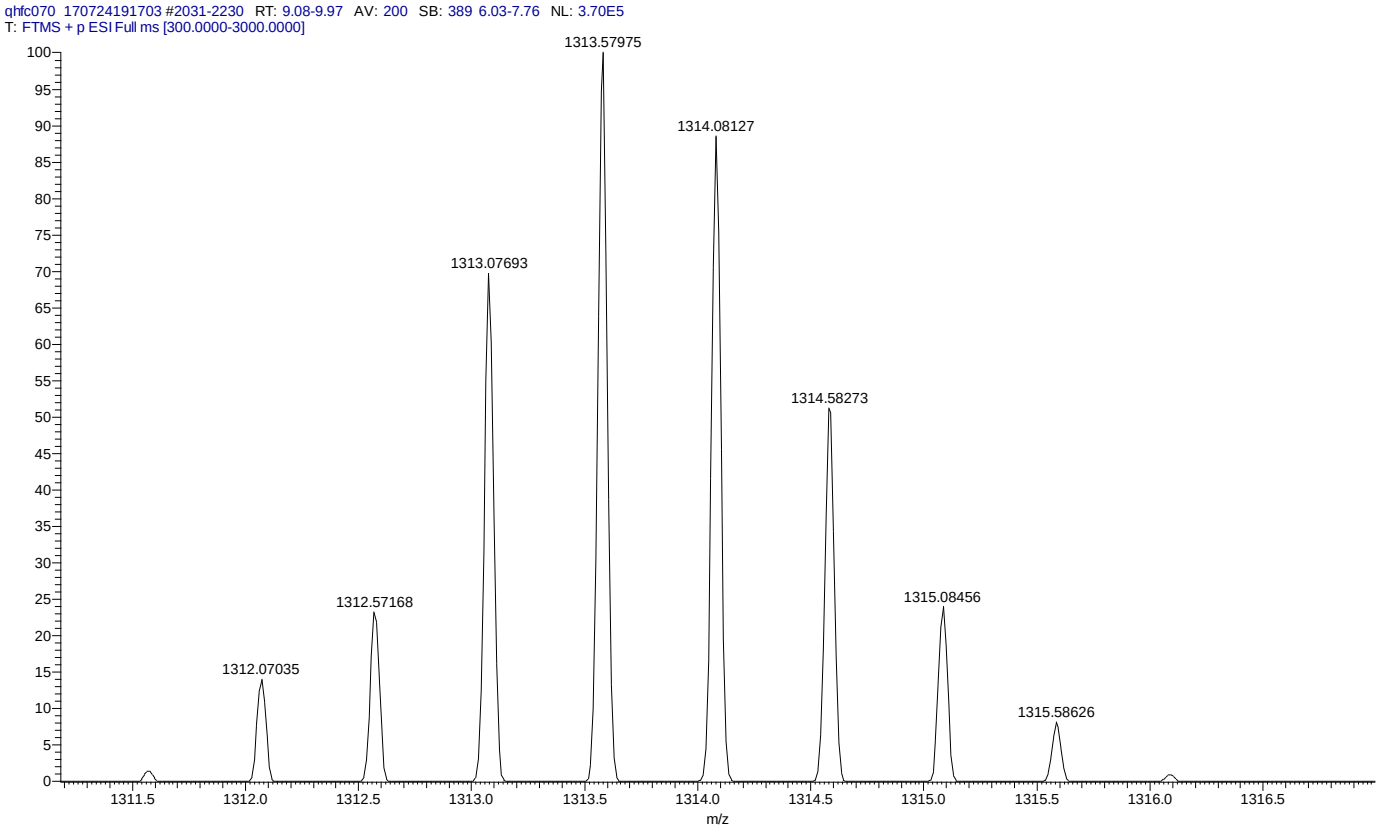
Sample Name :	Zk-OAT7	Reference No.:	Qhfc070
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{161}H_{278}N_{16}O_9$
Experimental Mass $[M+Na]^+$, $[M+2Na]^{2+}$:	2604.17187, 1313.57975
Theoretical Mass $[M+Na]^+$, $[M+2Na]^{2+}$:	2604.17120, 1313.58021
Error (ppm) :	0.2, 0.3






qhfc070 170724191703 #2031-2230 RT: 9.08-9.97 AV: 200 SB: 389 6.03-7.76 NL: 3.70E5
T: FTMS + p ESI Full ms [300.0000-3000.0000]
1313.57975
1314.08127
1313.07693
1314.58273
1315.08456
1312.57168
1312.07035
1315.58626
m/z

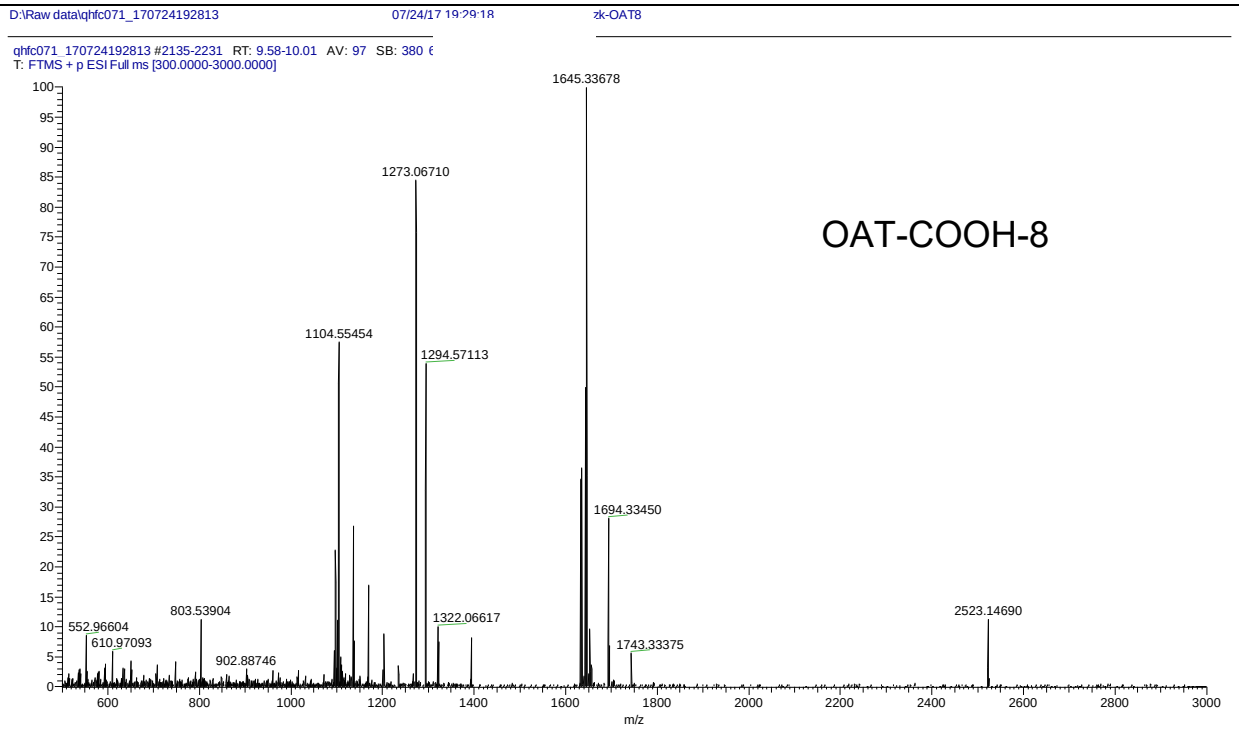
Thermo QEFMS Analysis Report

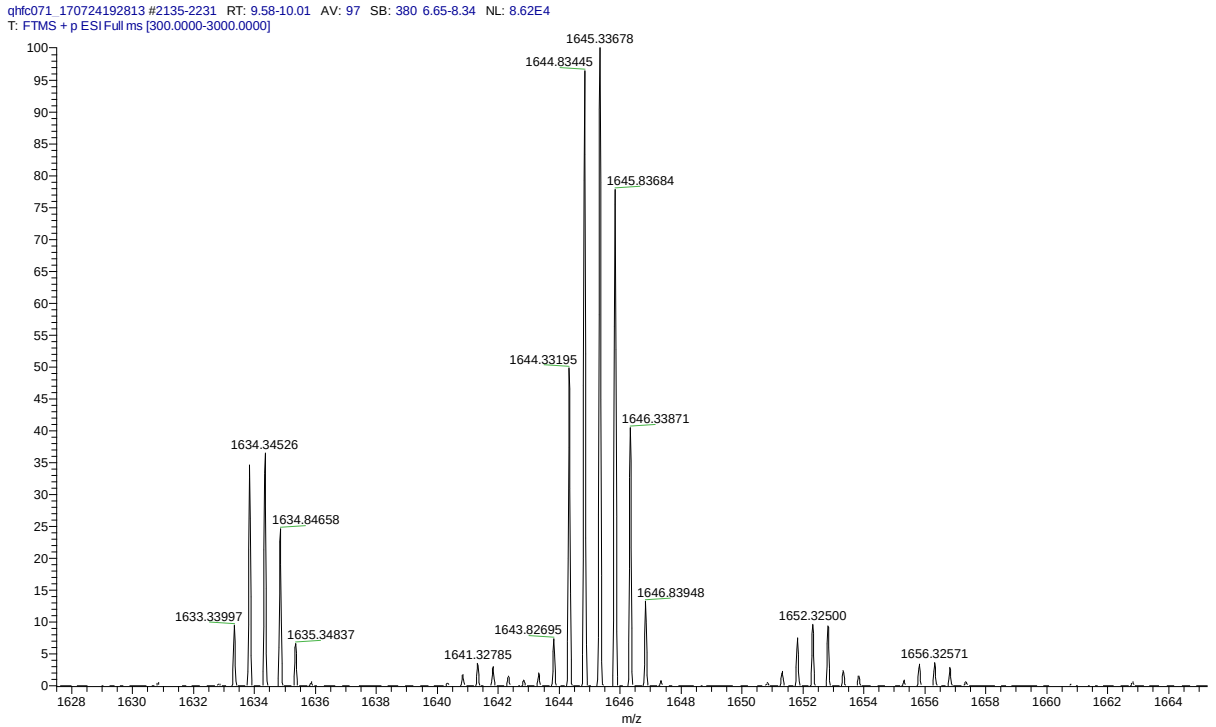
Analysis Info

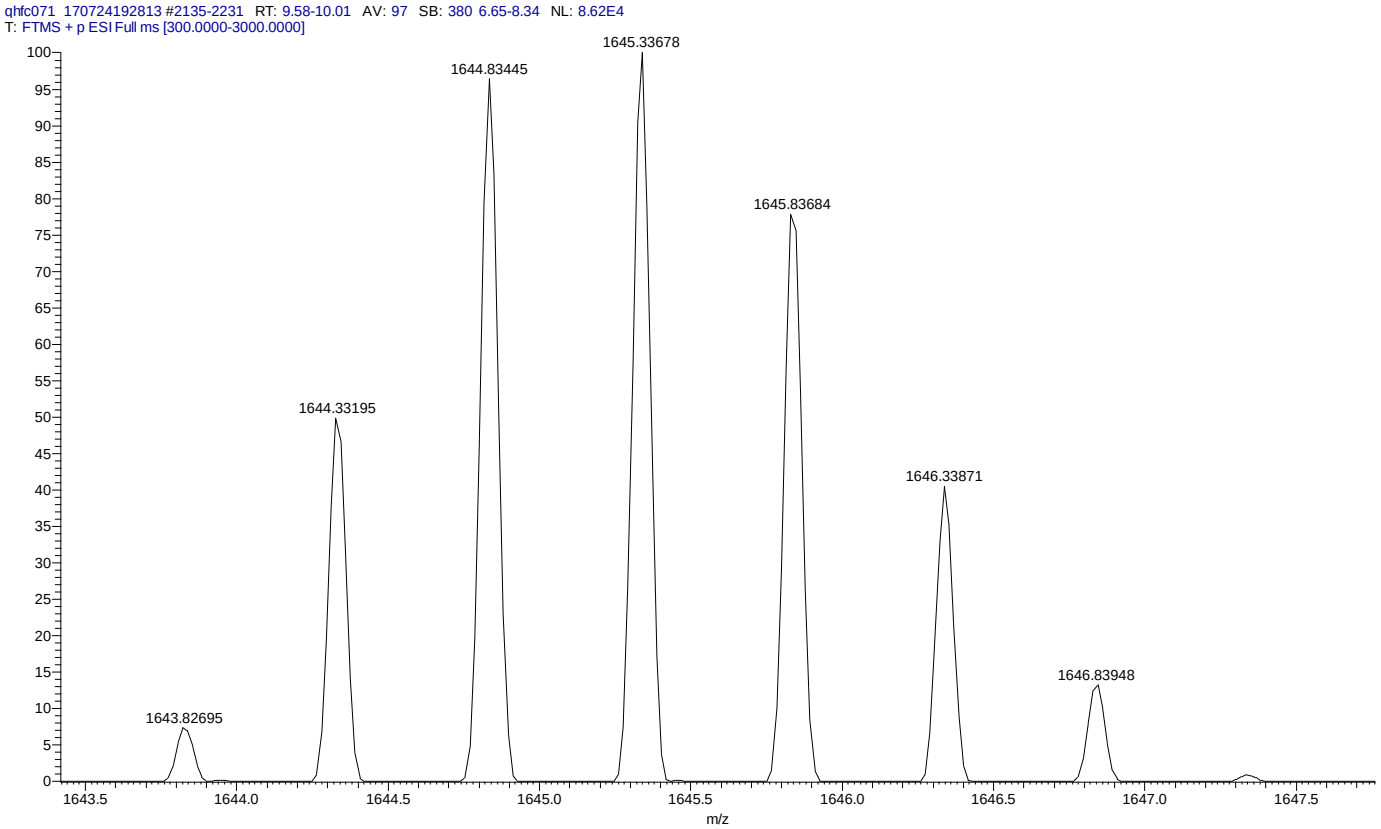
Sample Name :	Zk-OAT8	Reference No.:	Qhfc071
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{200}H_{346}N_{20}O_{12}$
Experimental Mass $[M+2Na]^{2+}$, $[M-H+3Na]^{2+}$:	1634.34526, 1645.33678
Theoretical Mass $[M+2Na]^{2+}$, $[M-H+3Na]^{2+}$:	1634.34637, 1645.33734
Error (ppm) :	0.6, 0.3






qhfc071 170724192813 #2135-2231 RT: 9.58-10.01 AV: 97 SB: 380 6.65-8.34 NL: 8.62E4
T: FTMS + p ESI Full ms [300.0000-3000.0000]
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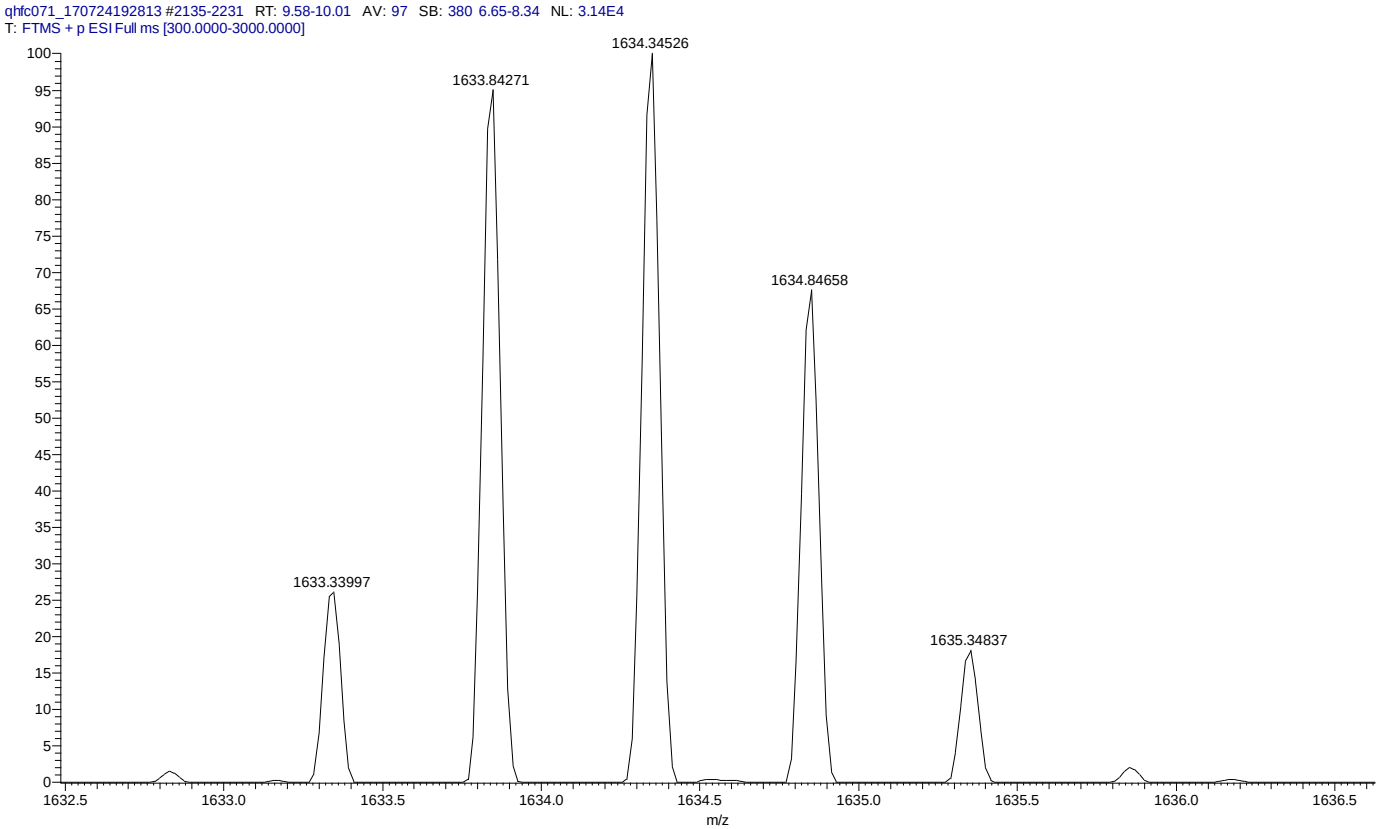
1645.83684
1644.33195
1646.33871
1646.83948
1643.82695
m/z


qhfc071_170724192813 #2135-2231 RT: 9.58-10.01 AV: 97 SB: 380 6.65-8.34 NL: 3.14E4
T: FTMS + p ESI Full ms [300.0000-3000.0000]
1634.34526
1633.84271
1634.84658
1633.33997
1635.34837
m/z

Bruker Autoflex speed MALDI-TOF MS Analysis Report

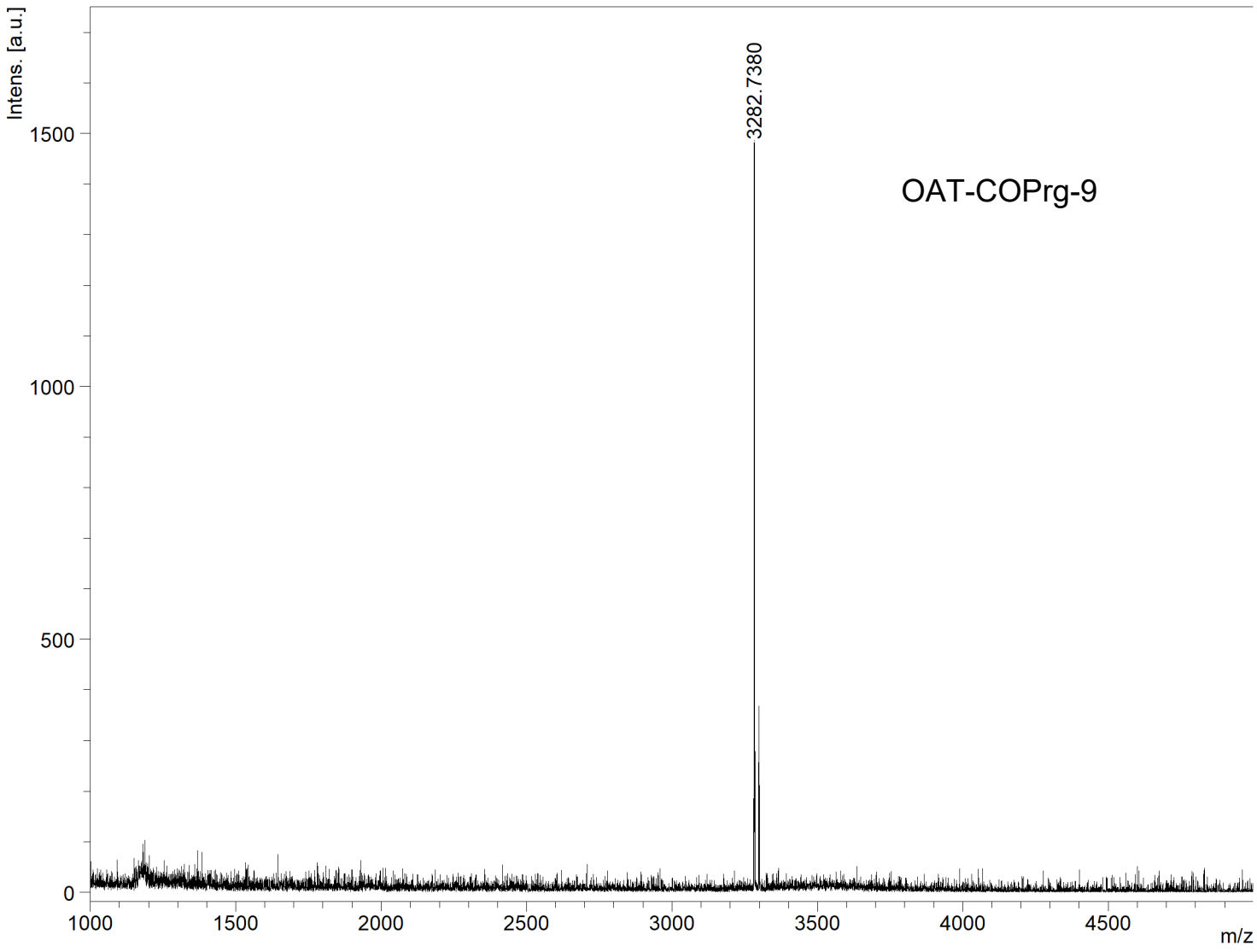
Department of Chemistry, The Chinese University of Hong Kong

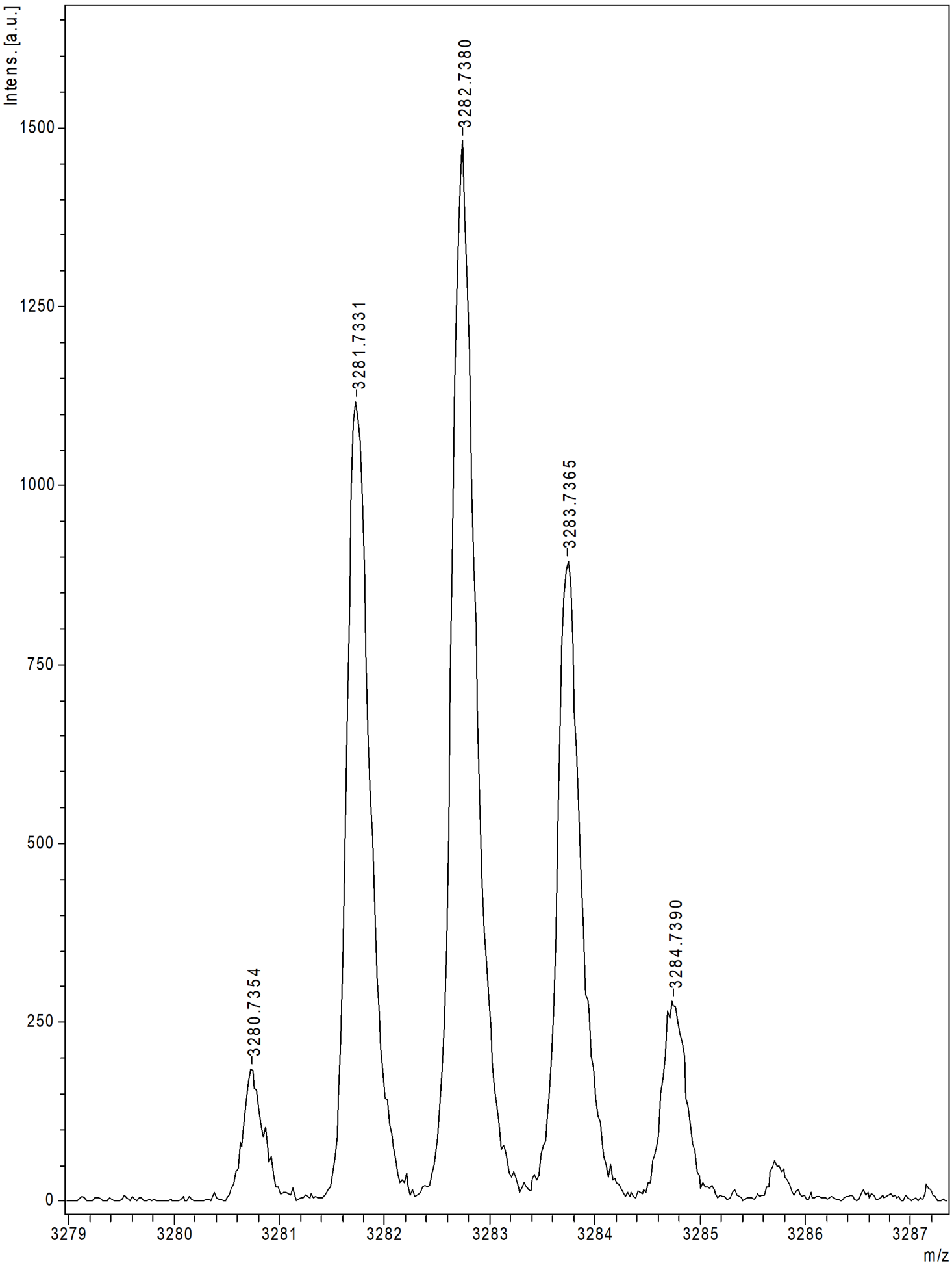
Analysis Info

Sample Name:	zk-OAT9	Reference No.:	wfhfc068\0_C1\3
Applicant Name:	Zheng Kun	Date of Analysis :	2018-02-13T15:16:33.000
Method:	D:\Methods\flexControlMethods\Test_RP_1000-4500_Da.par		
Polarity:	POS	PIE delay: 150 ns	No. of shots: 800
Comment:	Reflector mode, DCTB as matrix, 384 polished stainless steel target plate		

Accurate Mass Measurement

Molecular formula:	C203H349N21O11
Abundant Isotopic (theoretical) [M+Na]+:	3282.7351
Experimental [M+Na]+:	3282.7380
Error (ppm):	0.88




Intens. [a.u.]
1500
1250
1000
750
500
250
0
3279
3280
3281
3282
3283
3284
3285
3286
3287
m/z
3280.7354
3281.7331
3282.7380
3283.7365
3284.7390

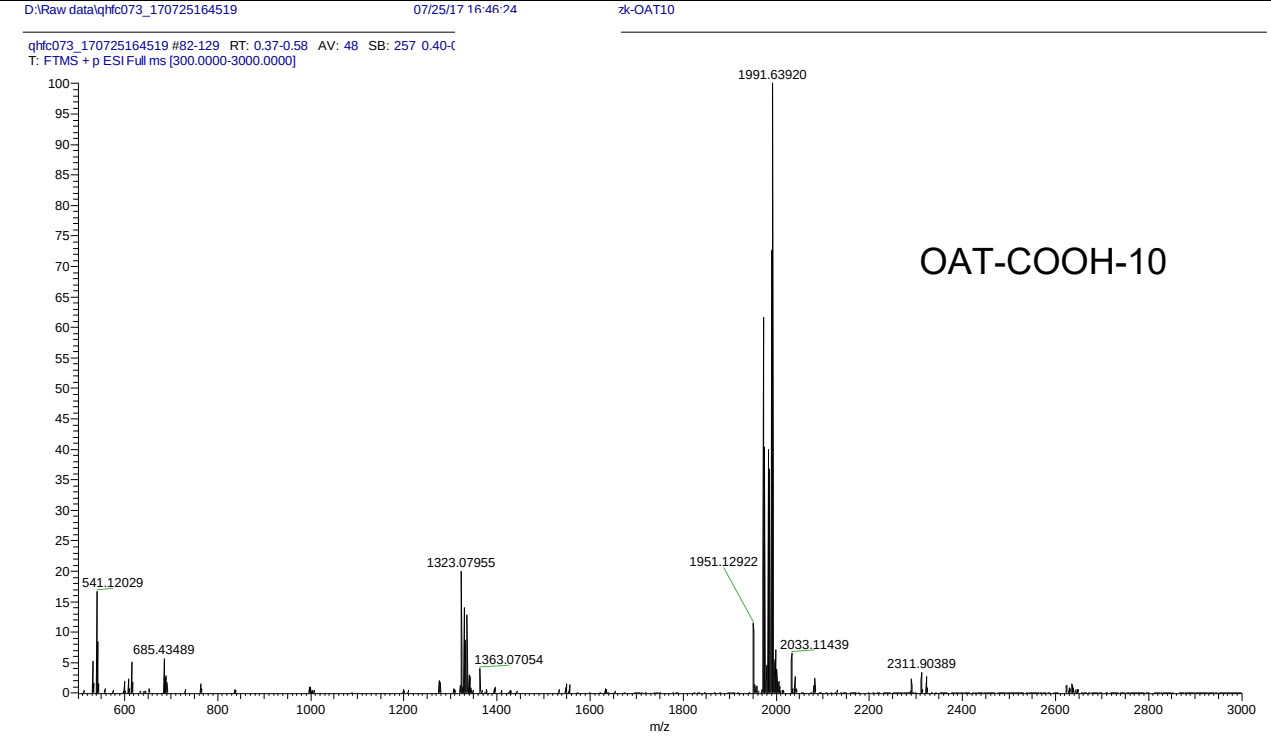
Thermo QEFMS Analysis Report

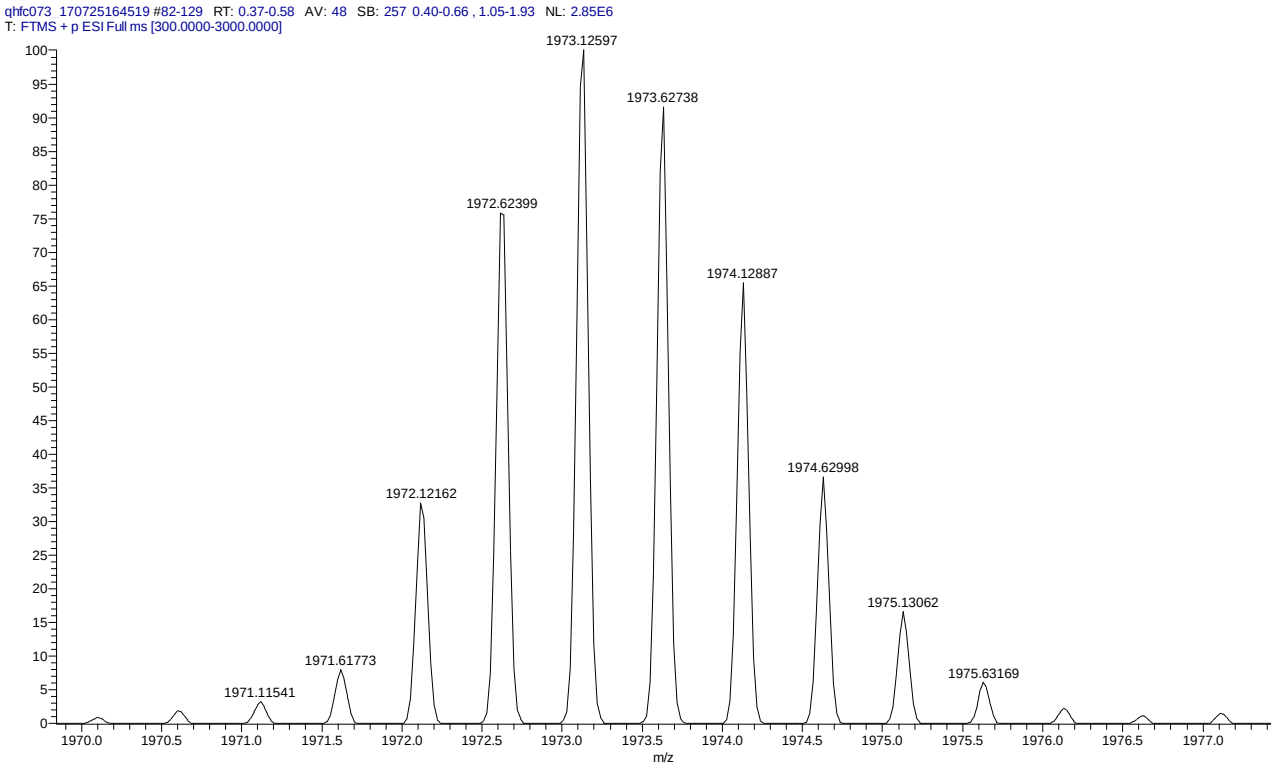
Analysis Info

Sample Name :	Zk-OAT10	Reference No.:	Qhfc073
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{242}H_{417}N_{25}O_{14}$
Experimental Mass $[M+2Na]^{2+}$:	1973.12597
Theoretical Mass $[M+2Na]^{2+}$:	1973.12675
Error (ppm) :	0.3





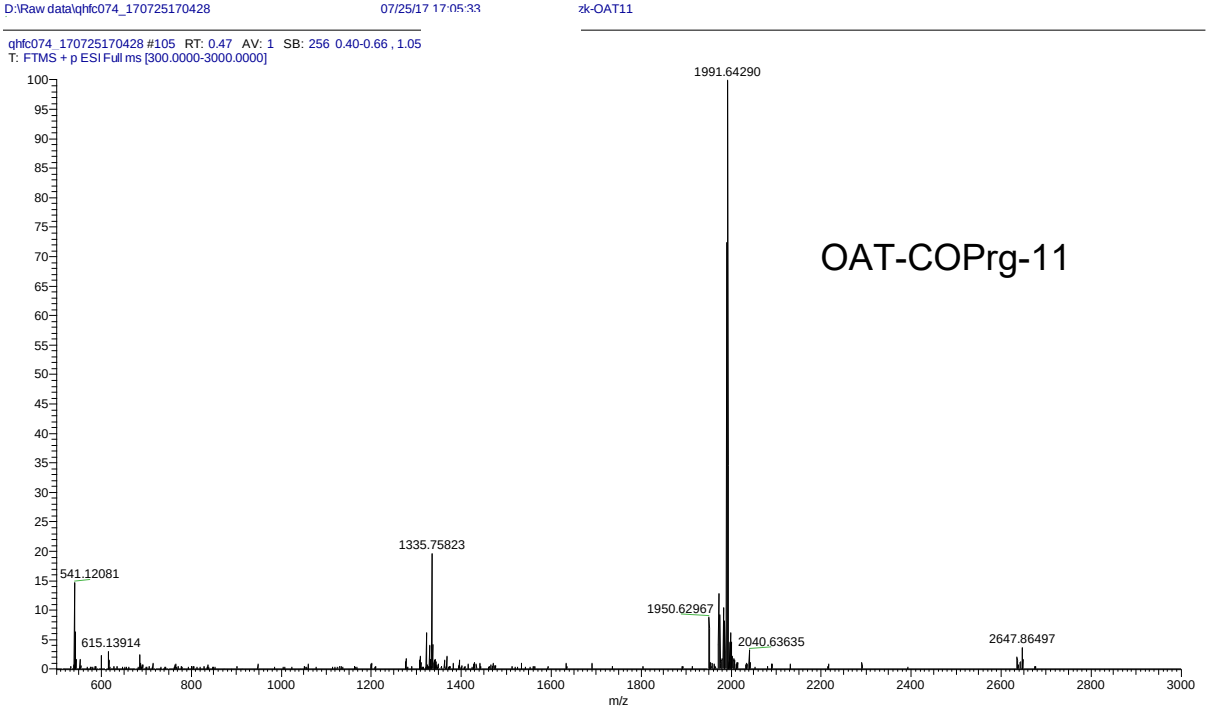
Thermo QEFMS Analysis Report

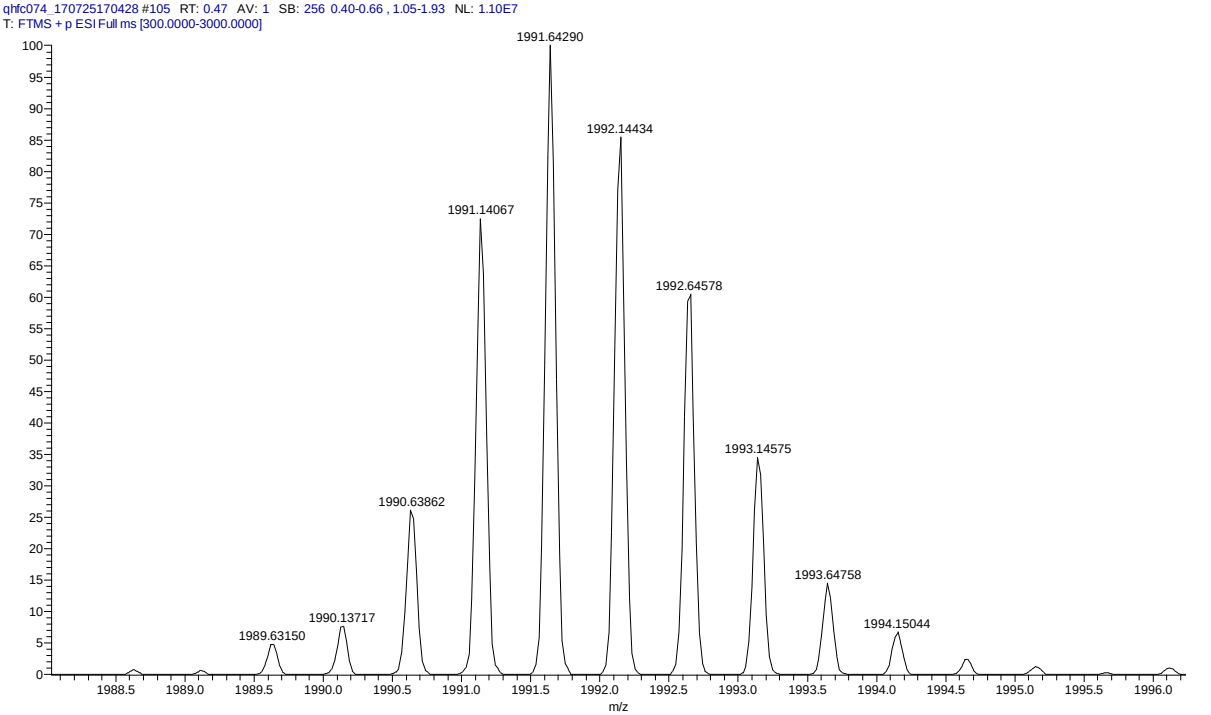
Analysis Info

Sample Name :	Zk-OAT11	Reference No.:	Qhfc074
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{245}H_{420}N_{26}O_{13}$
Experimental Mass $[M+2Na]^{2+}$:	1991.64290
Theoretical Mass $[M+2Na]^{2+}$:	1991.64257
Error (ppm) :	0.1





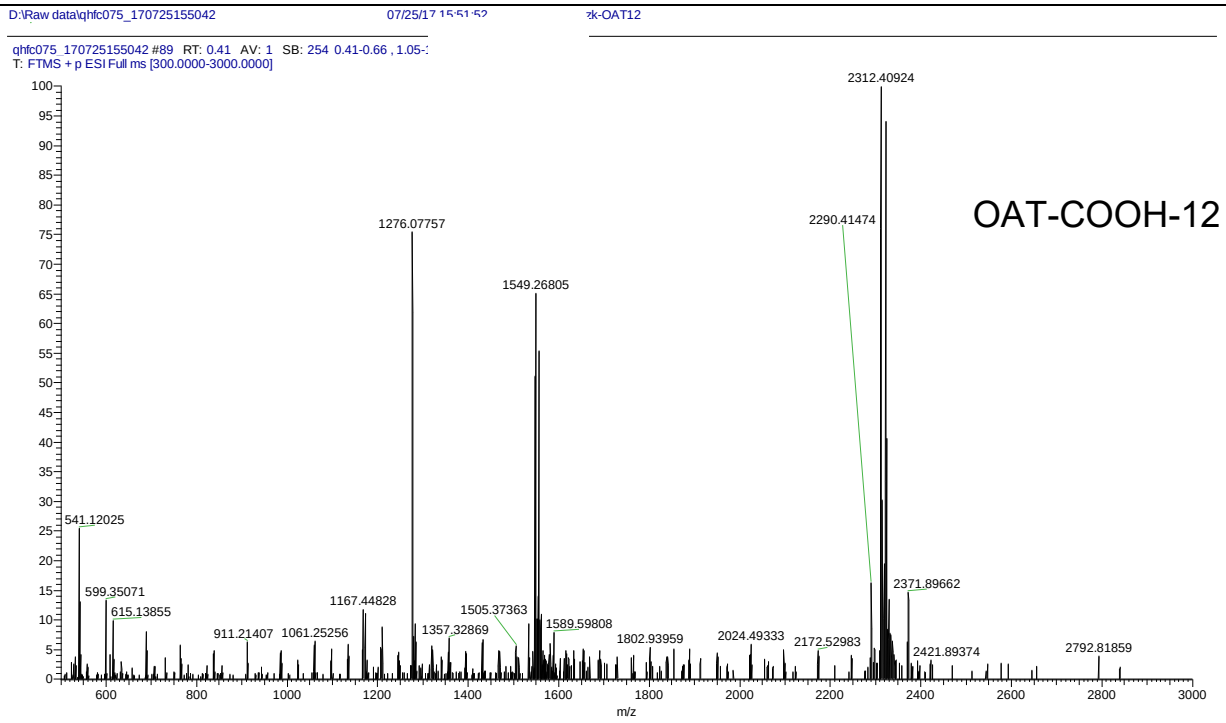
Thermo QEFMS Analysis Report

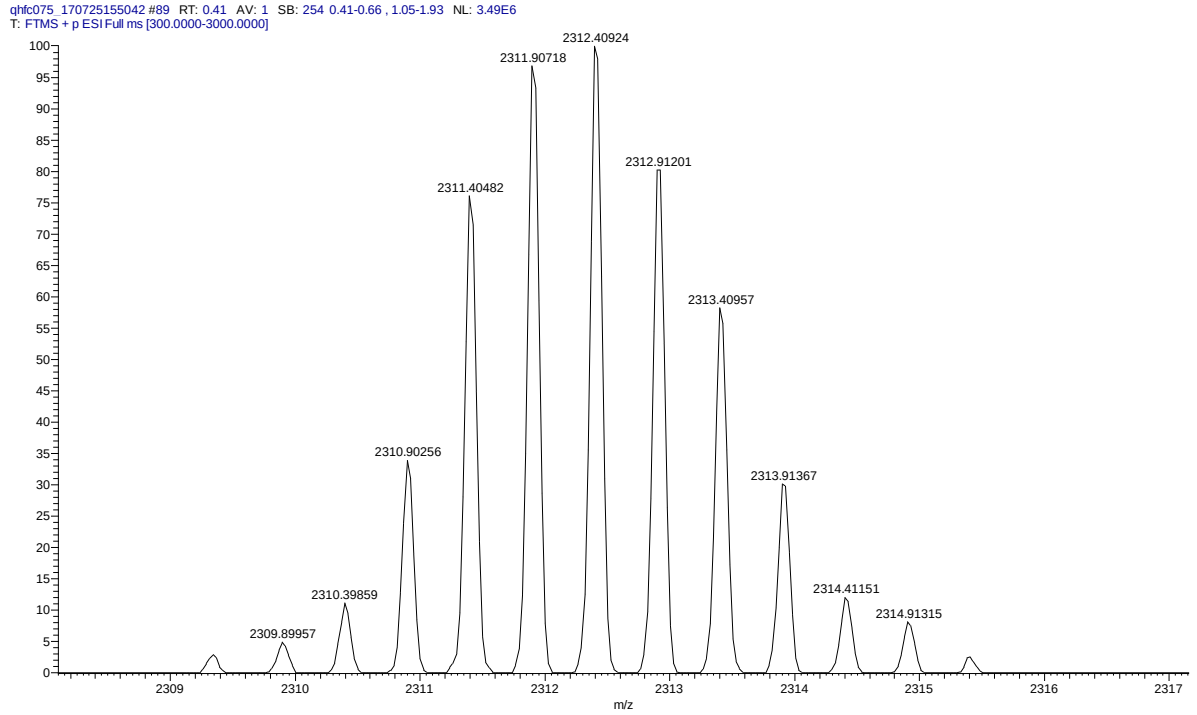
Analysis Info

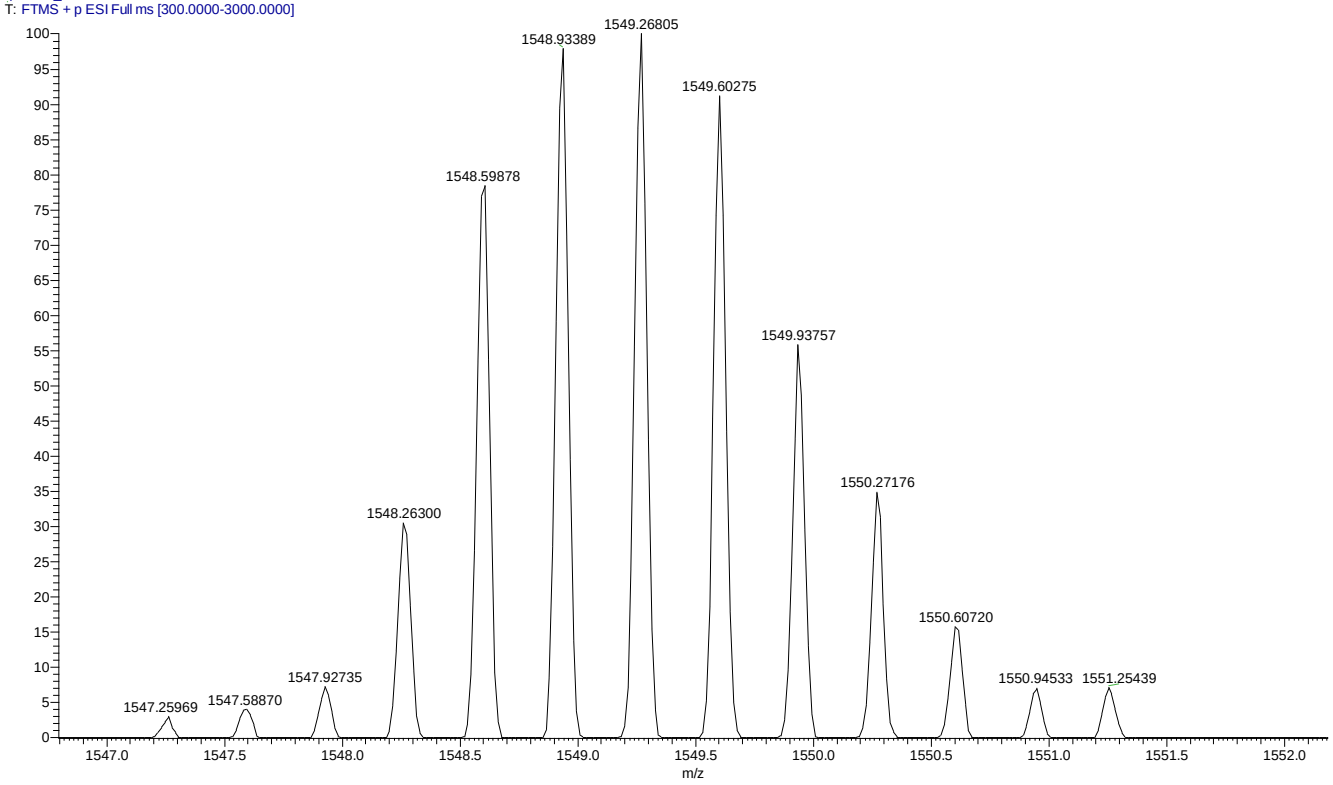
Sample Name :	Zk-OAT12	Reference No.:	Qhfc075
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{284}H_{488}N_{30}O_{16}$
Experimental Mass $[M+2Na]^{2+}$, $[M+3Na]^{3+}$:	2312.40924, 1549.26805
Theoretical Mass $[M+2Na]^{2+}$, $[M+3Na]^{3+}$:	2312.40872, 1549.26889
Error (ppm) :	0.2, 0.5






qhfc075_170725155042 #89 RT: 0.41 AV: 1 SB: 254 0.41-0.66 , 1.05-1.93 NL: 2.27E6
T: FTMS + p ESI Full ms [300.0000-3000.0000]
1549.26805
1548.93389
1549.60275
1548.59878
1549.93757
1550.27176
1548.26300
1550.60720
1547.92735
1550.94533
1551.25439
1547.25969
1547.58870
m/z

Bruker Autoflex speed MALDI-TOF MS Analysis Report

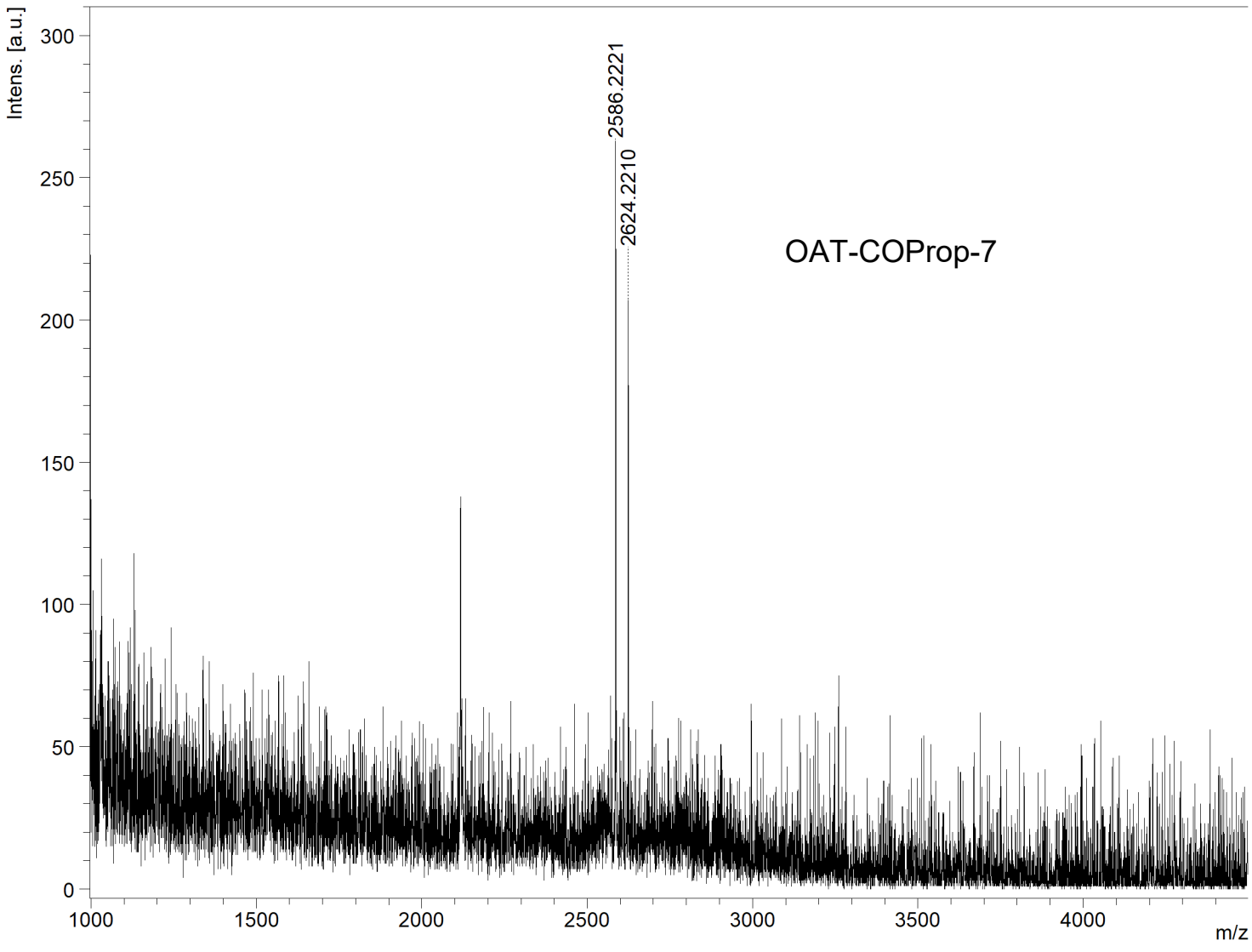
Department of Chemistry, The Chinese University of Hong Kong

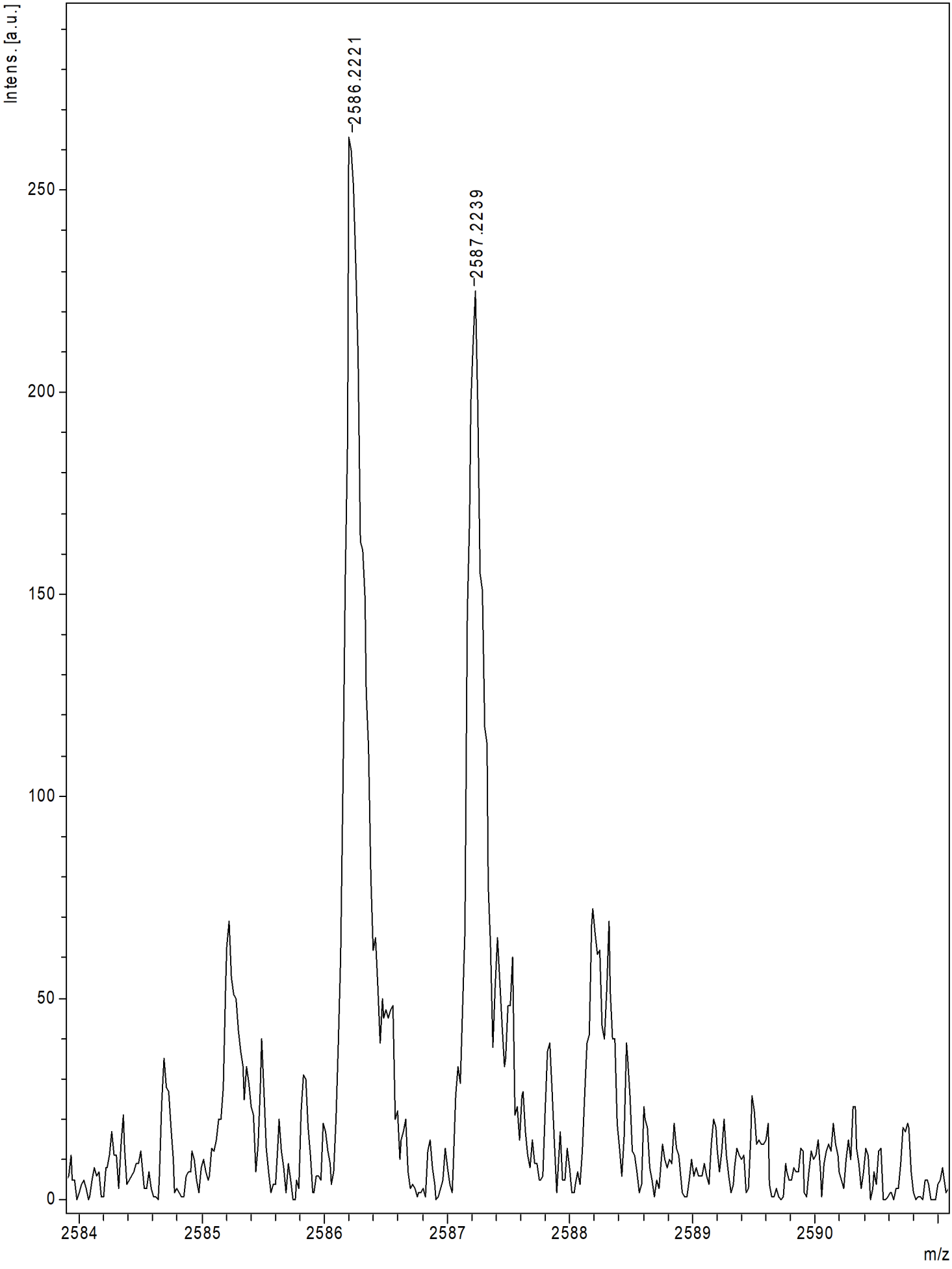
Analysis Info

Sample Name:	ZK-OAT7-prop	Reference No.:	wfhfc075\0_C9\1
Applicant Name:	Zheng Kun	Date of Analysis :	2018-04-25T15:17:52.000
Method:	D:\Methods\flexControlMethods\Test_RP_1000-4500_Da.par		
Polarity:	POS	PIE delay: 150 ns	No. of shots: 600
Comment:	Reflector mode, DCTB as matrix, 384 polished stainless steel target plate		

Accurate Mass Measurement

Molecular formula:	C161H282N16O9
Abundant Isotopic (theoretical) [M+H]+:	2586.2206
Experimental [M+H]+:	2586.2221
Error (ppm):	0.58




Intens. [a.u.]
2586.2221
2587.2239
250
200
150
100
50
0
2584
2585
2586
2587
2588
2589
2590
m/z

Bruker Autoflex speed MALDI-TOF MS Analysis Report

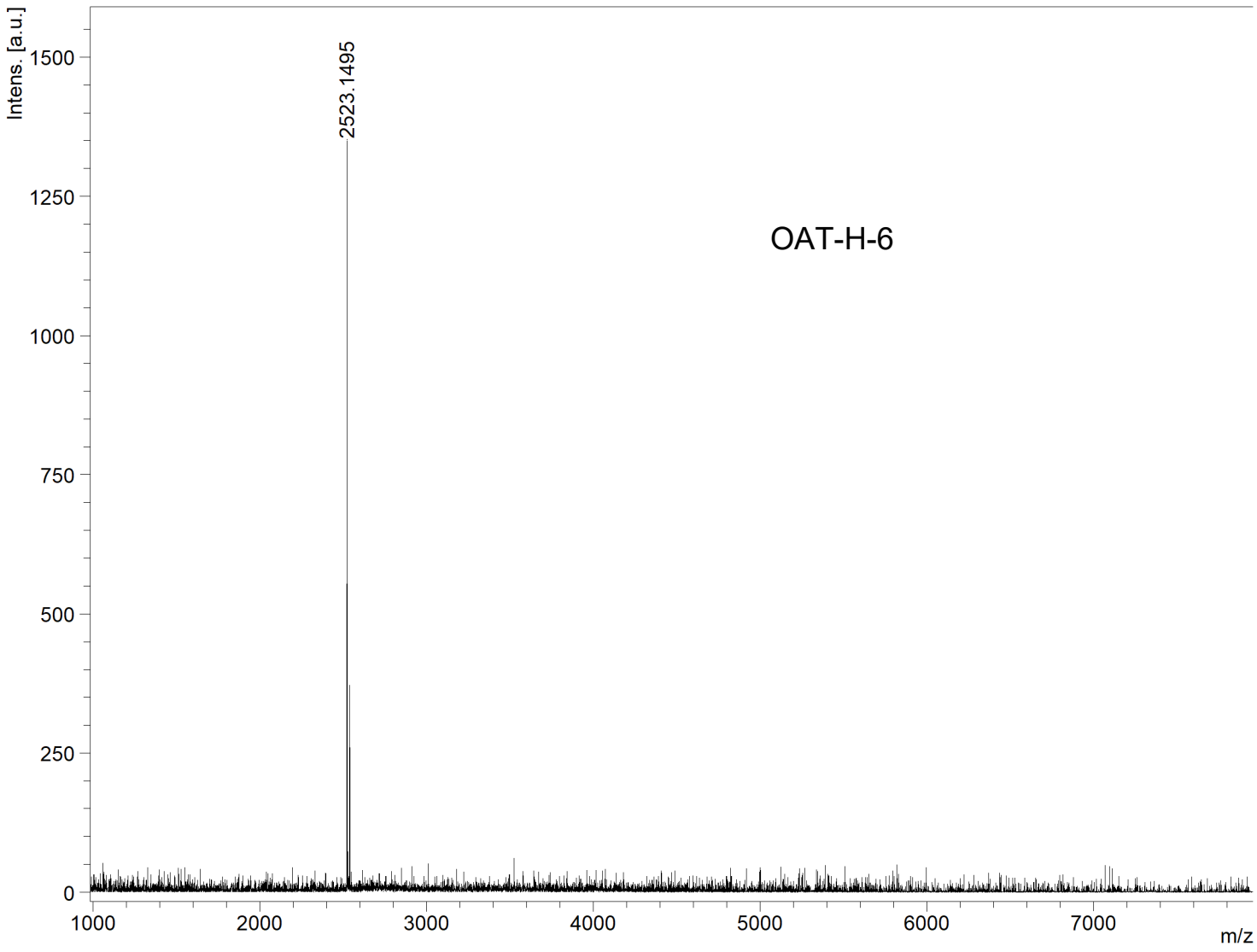
Department of Chemistry, The Chinese University of Hong Kong

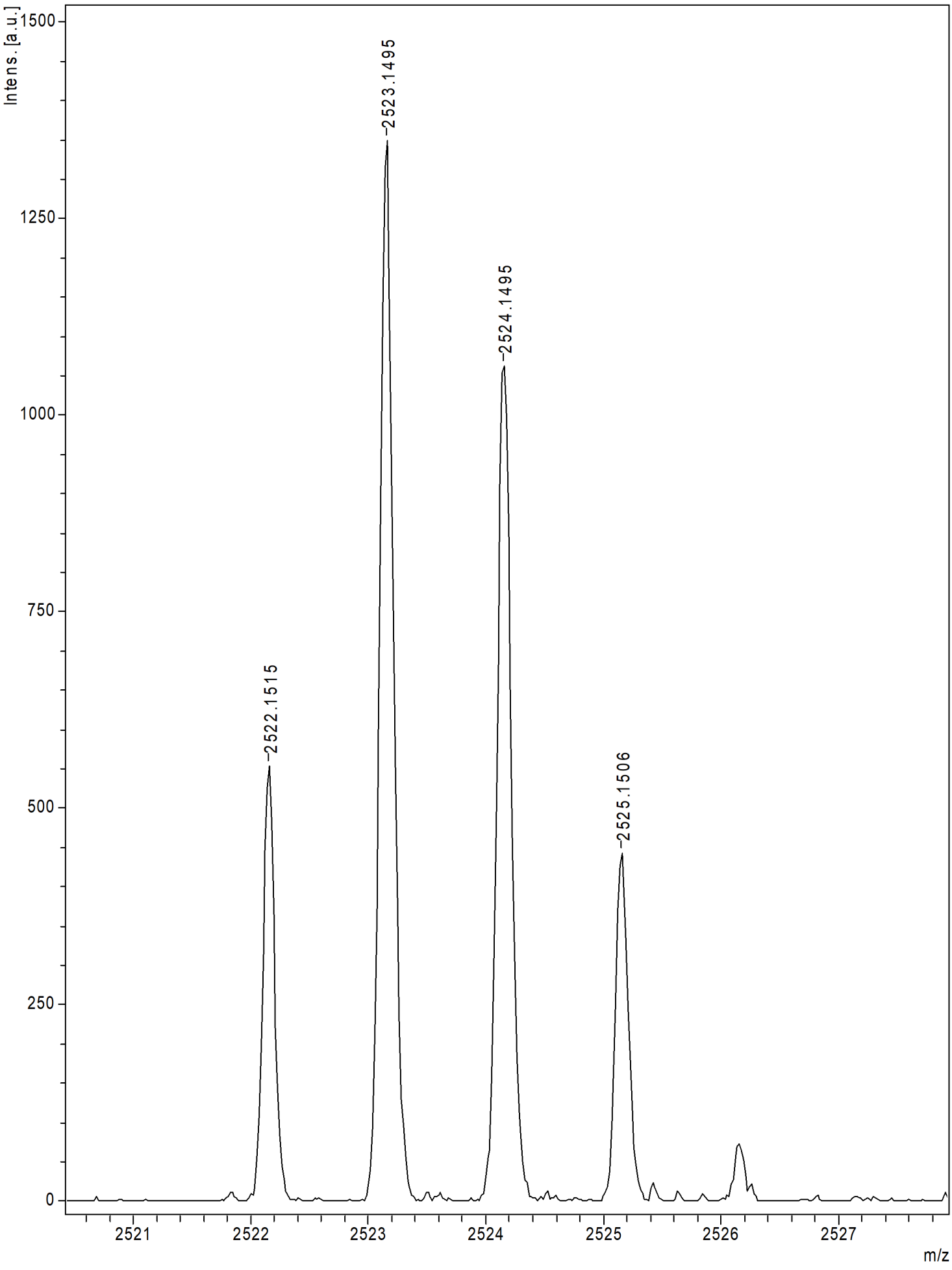
Analysis Info

Sample Name:	OAT6-a	Reference No.:	wfhfc065\0_B2\1
Applicant Name:	Zheng Kun	Date of Analysis :	2018-02-13T16:10:04.000
Method:	D:\Methods\flexControlMethods\Test_RP_1000-4500_Da.par		
Polarity:	POS	PIE delay: 150 ns	No. of shots: 400
Comment:	Reflector mode, DCTB as matrix, 384 polished stainless steel target plate		

Accurate Mass Measurement

Molecular formula:	C157H275N15O8
Abundant Isotopic (theoretical) [M+Na]+:	2523.1497
Experimental [M+Na]+:	2523.1495
Error (ppm):	0.08




Intens. [a.u.]
1500
1250
1000
750
500
250
0
2522.1515
2523.1495
2524.1495
2525.1506
2521
2522
2523
2524
2525
2526
2527
m/z

Bruker Autoflex speed MALDI-TOF MS Analysis Report

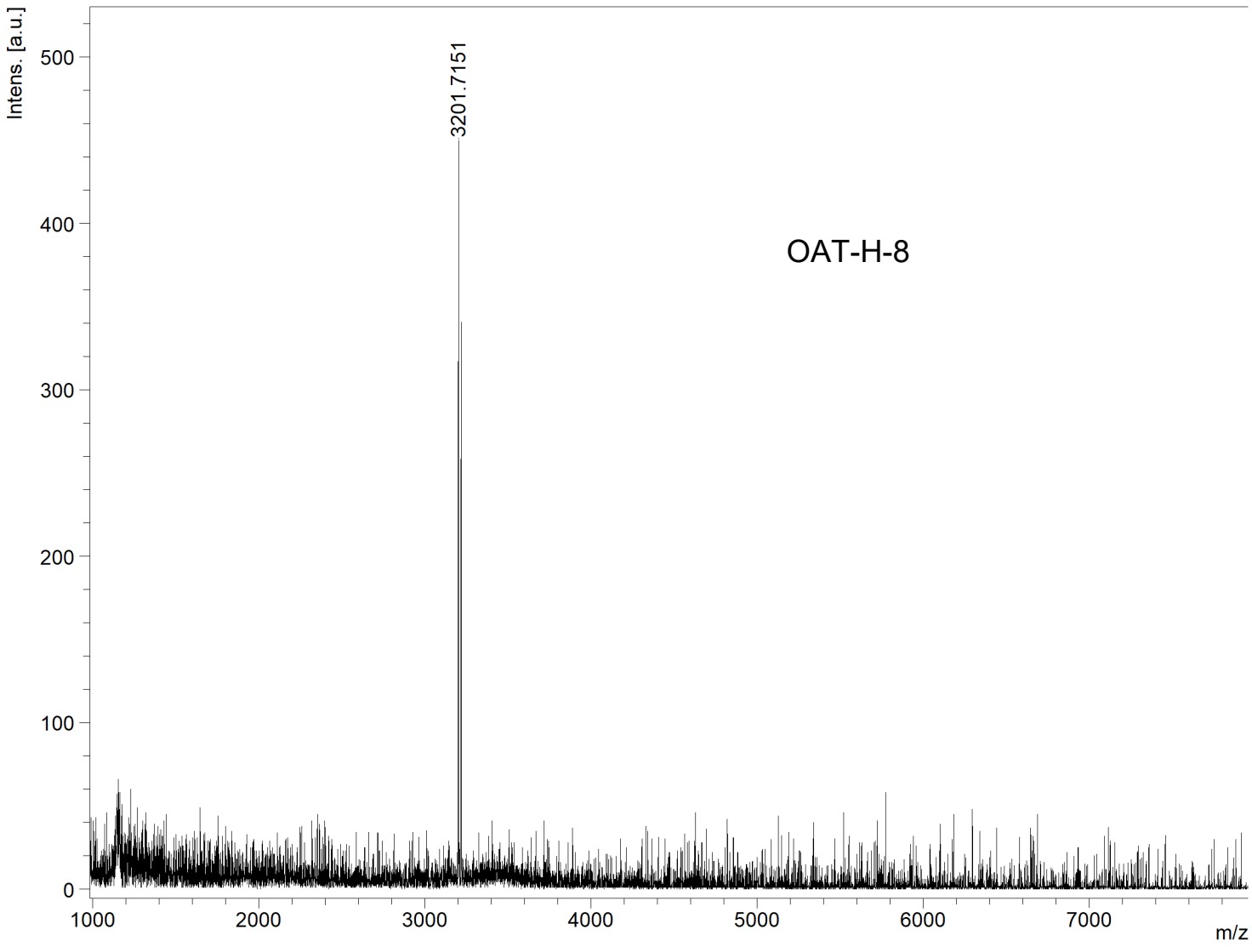
Department of Chemistry, The Chinese University of Hong Kong

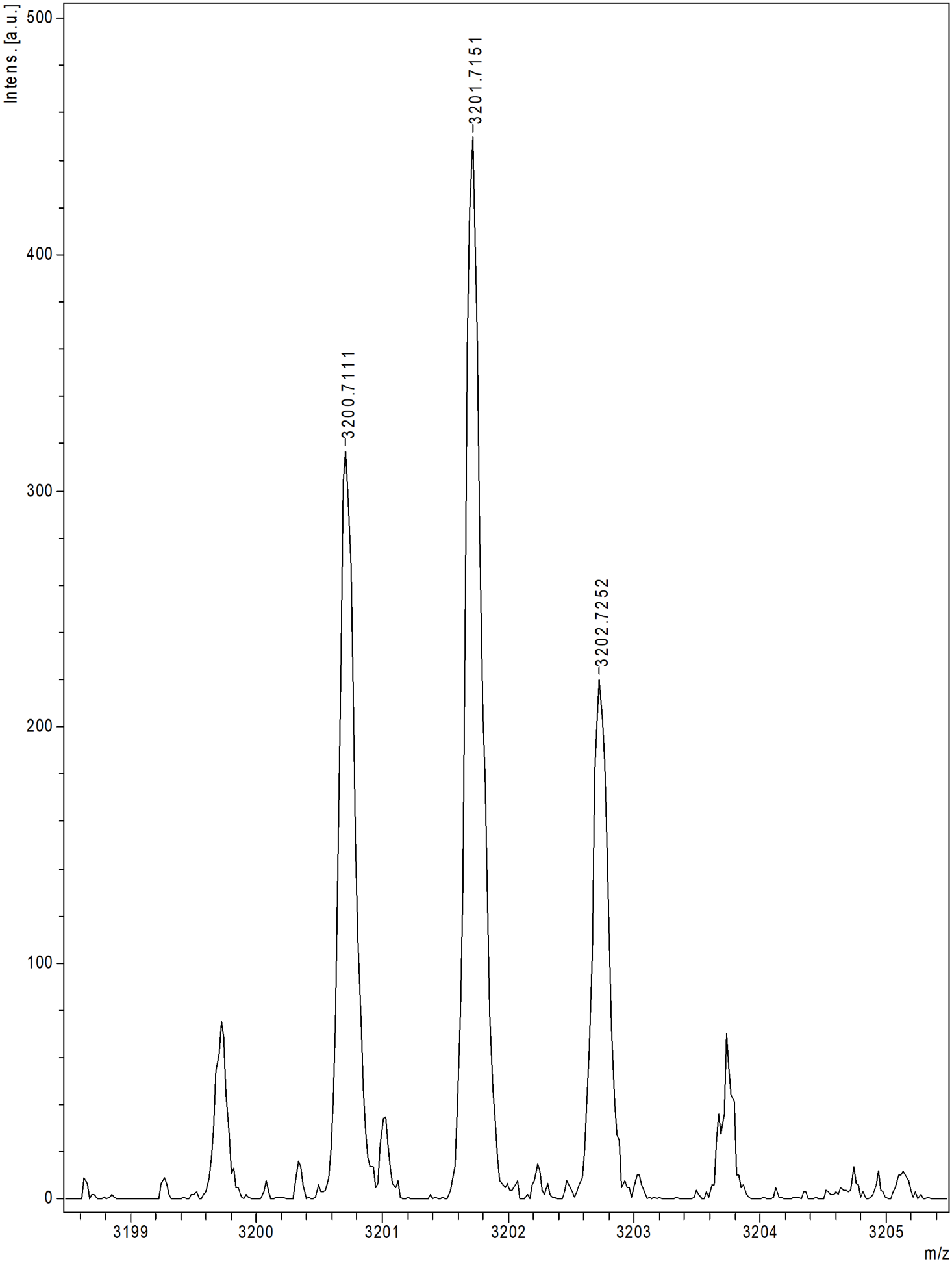
Analysis Info

Sample Name:	OAT8-a	Reference No.:	wfhfc066\0_B1\2
Applicant Name:	Zheng Kun	Date of Analysis :	2018-02-13T15:30:47.000
Method:	D:\Methods\flexControlMethods\Test_RP_1000-4500_Da.par		
Polarity:	POS	PIE delay: 150 ns	No. of shots: 400
Comment:	Reflector mode, DCTB as matrix, 384 polished stainless steel target plate		

Accurate Mass Measurement

Molecular formula:	C199H346N20O10
Abundant Isotopic (theoretical) [M+Na]+:	3201.7137
Experimental [M+Na]+:	3201.7151
Error (ppm):	0.44




Intens. [a.u.]
500
400
300
200
100
0
3200.7111
3201.7151
3202.7252
3199
3200
3201
3202
3203
3204
3205
m/z

Bruker Autoflex speed MALDI-TOF MS Analysis Report

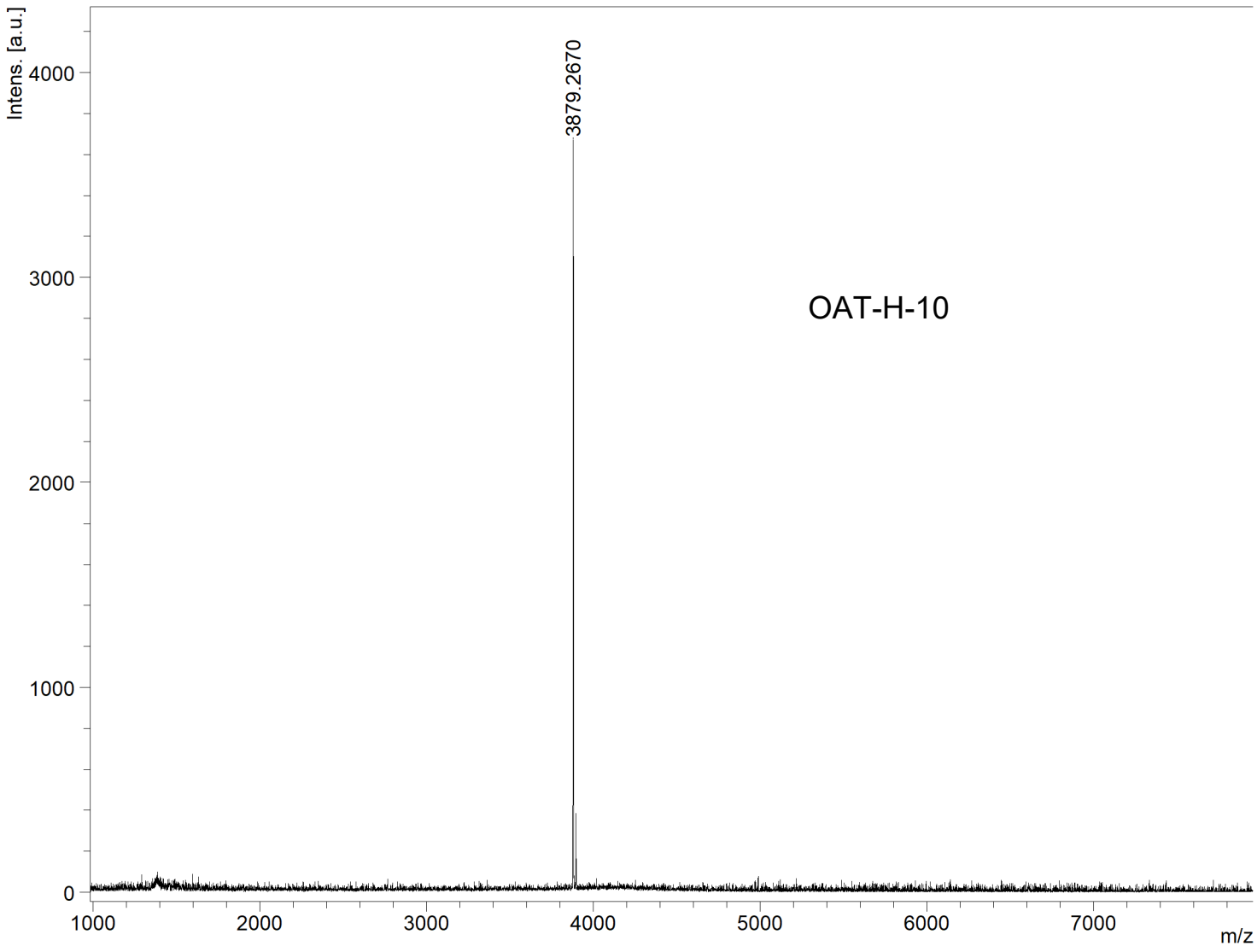
Department of Chemistry, The Chinese University of Hong Kong

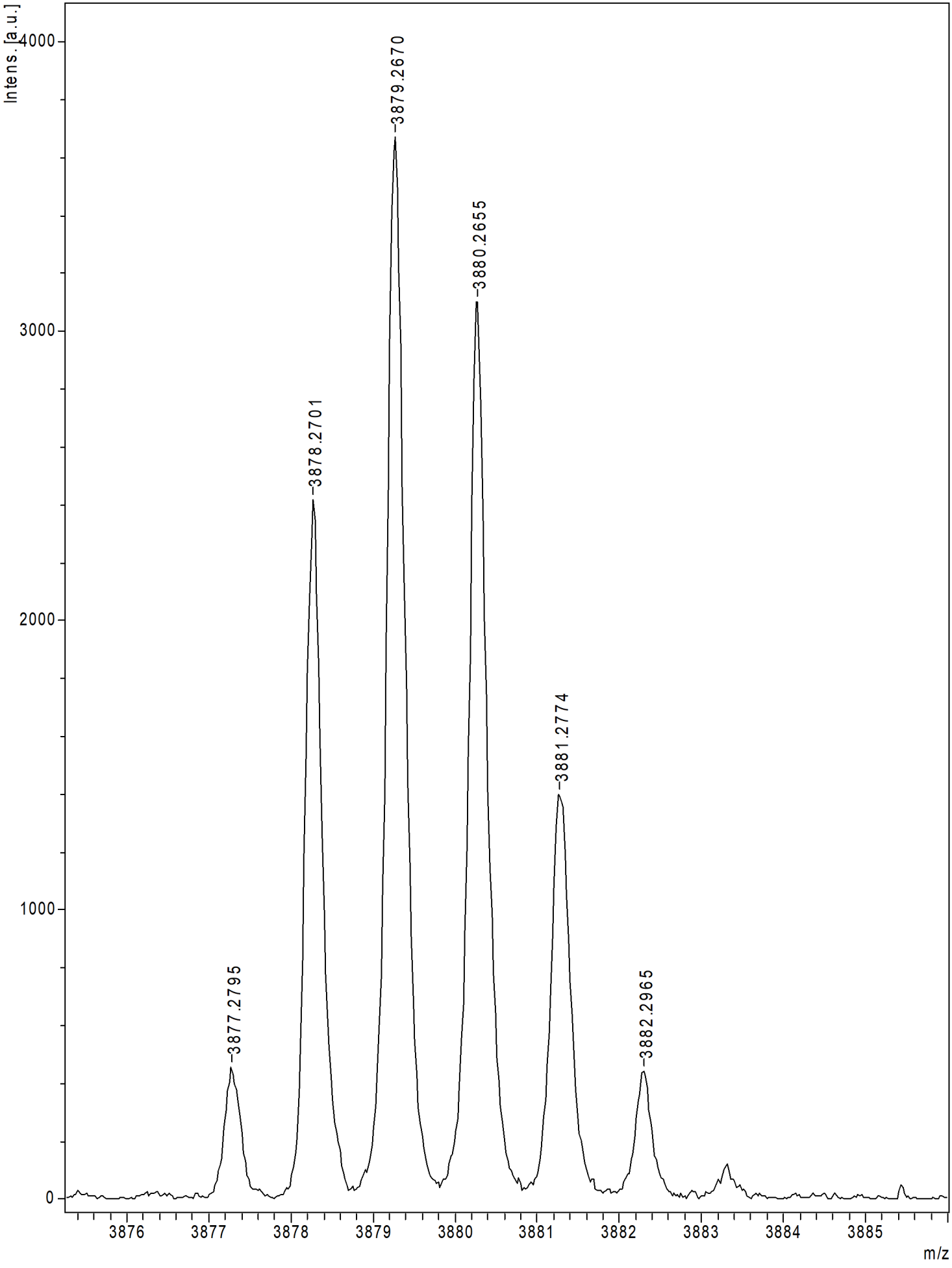
Analysis Info

Sample Name:	OAT10-a	Reference No.:	wfhfc067\0_B3\4
Applicant Name:	Zheng Kun	Date of Analysis :	2018-02-13T15:41:18.000
Method:	D:\Methods\flexControlMethods\Test_RP_1000-4500_Da.par		
Polarity:	POS	PIE delay: 150 ns	No. of shots: 2000
Comment:	Reflector mode, DCTB as matrix, 384 polished stainless steel target plate		

Accurate Mass Measurement

Molecular formula:	C241H417N25O12
Abundant Isotopic (theoretical) [M+Na]+:	3879.2745
Experimental [M+Na]+:	3879.2670
Error (ppm):	1.93




Intens. [a.u.]
4000
3000
2000
1000
0
3877.2795
3878.2701
3879.2670
3880.2655
3881.2774
3882.2965
3876
3877
3878
3879
3880
3881
3882
3883
3884
3885
m/z

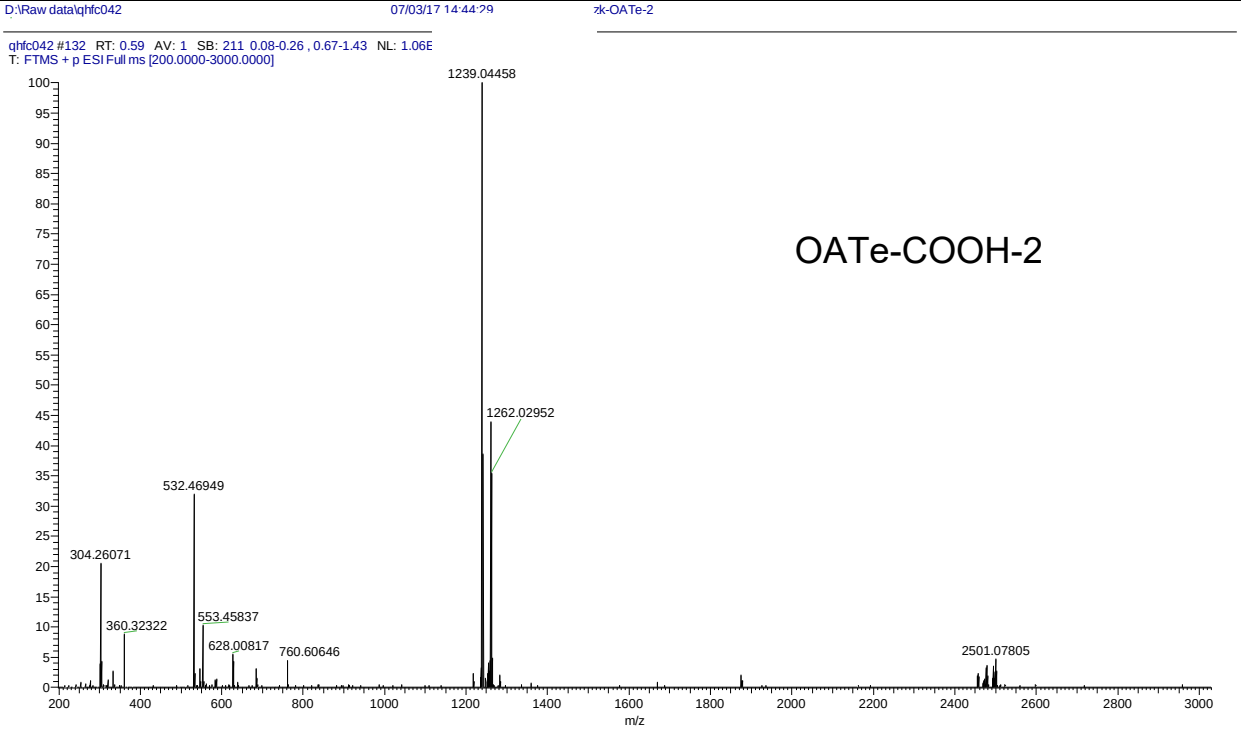
Thermo QEFMS Analysis Report

Analysis Info

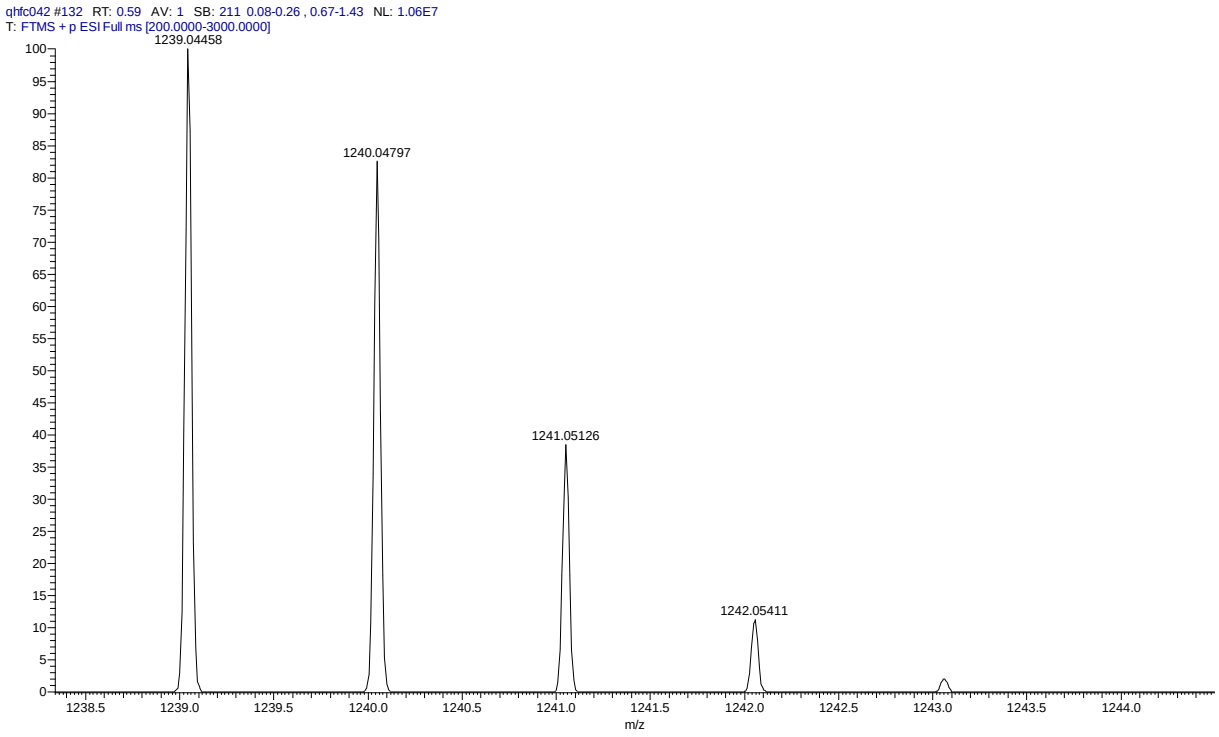
Sample Name :	Zk-OATe-2	Reference No.:	Qhfc042
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{76}H_{137}N_5O_6$
Experimental Mass $[M+Na]^+$:	1239.04458
Theoretical Mass $[M+Na]^+$:	1239.04611
Error (ppm) :	1.2



OATe-COOH-2



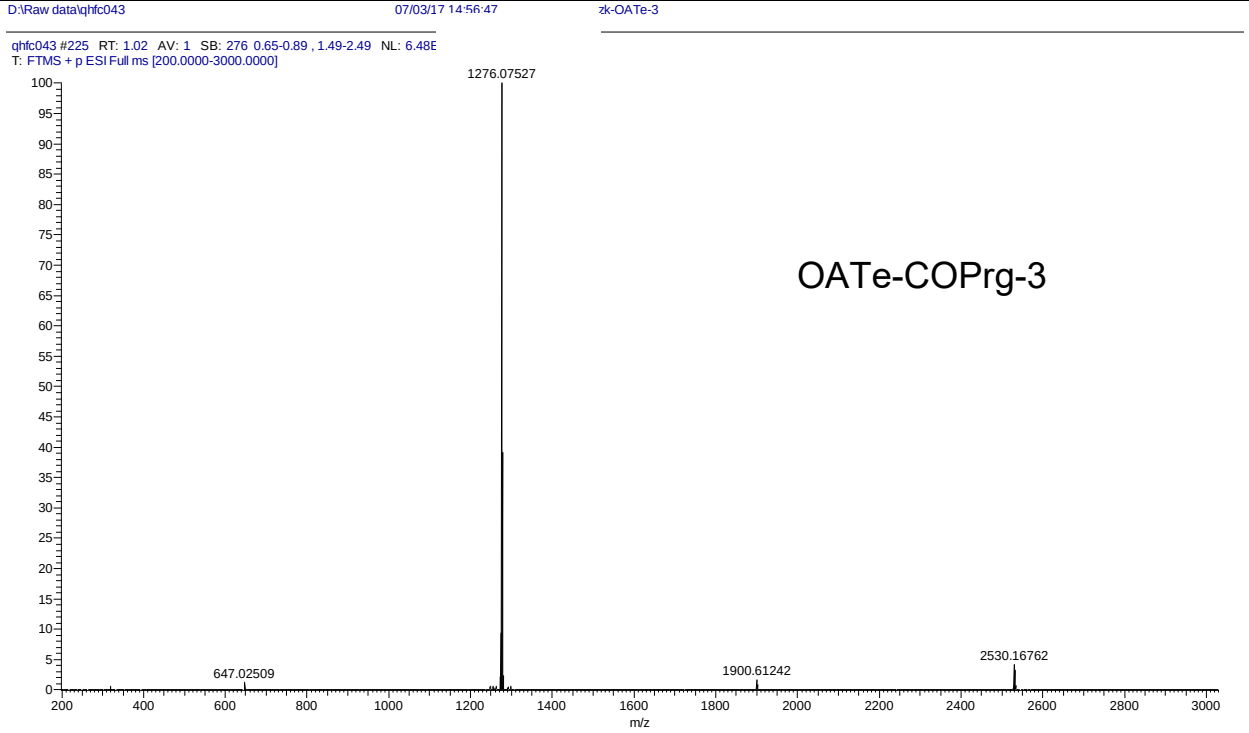
Thermo QEFMS Analysis Report

Analysis Info

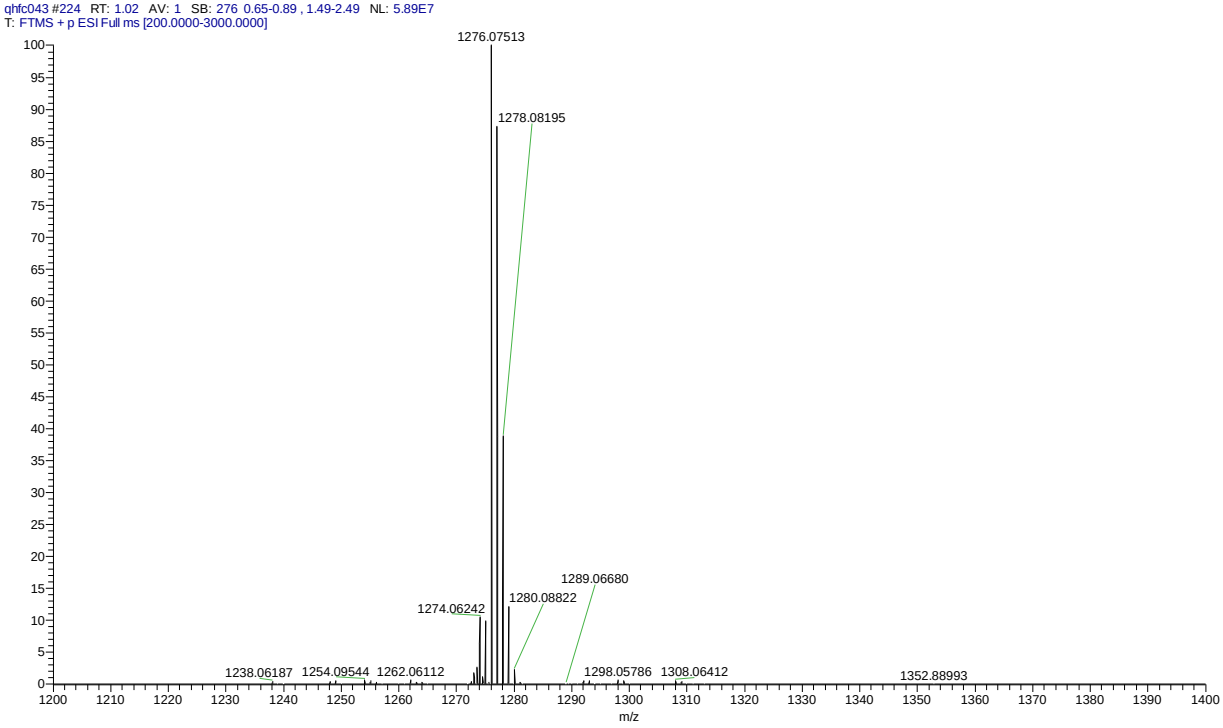
Sample Name :	Zk-OATe-3	Reference No.:	Qhfc043
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{79}H_{140}N_6O_5$
Experimental Mass $[M+Na]^+$:	1276.07527
Theoretical Mass $[M+Na]^+$:	1276.07774
Error (ppm) :	1.9



OATe-COPrg-3



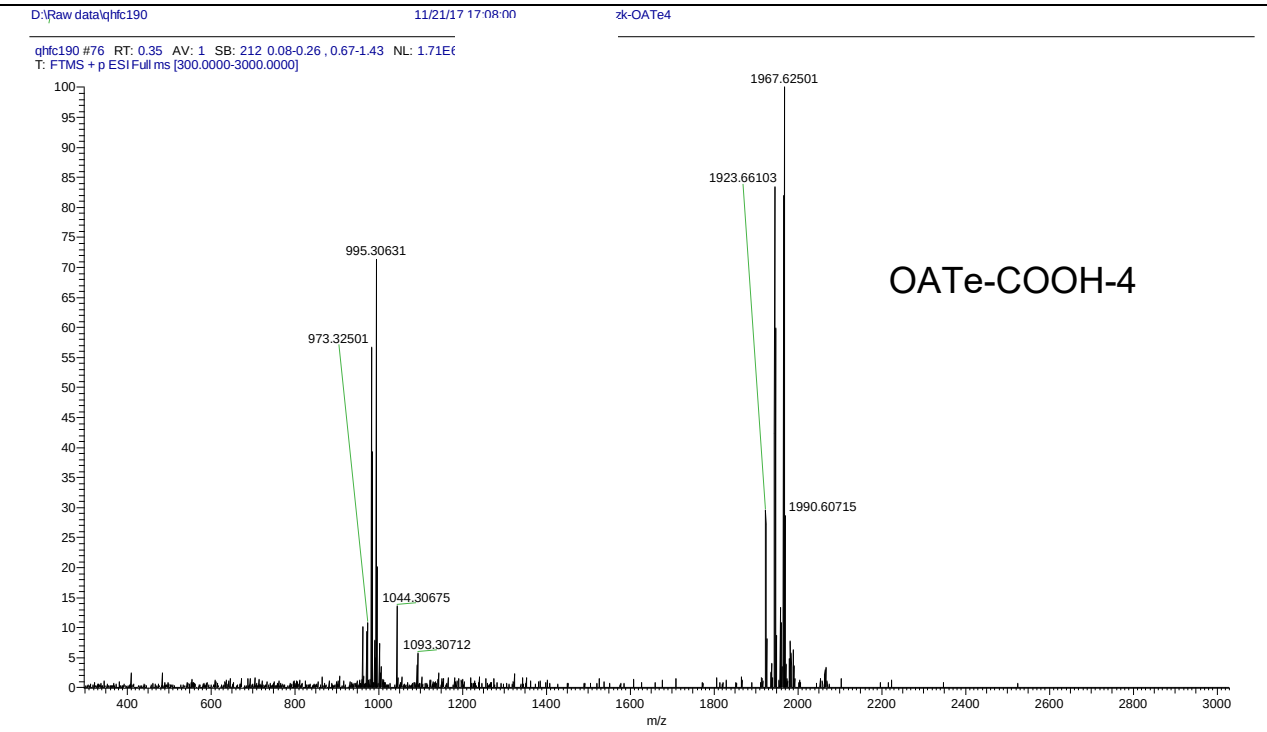
Thermo QEFMS Analysis Report

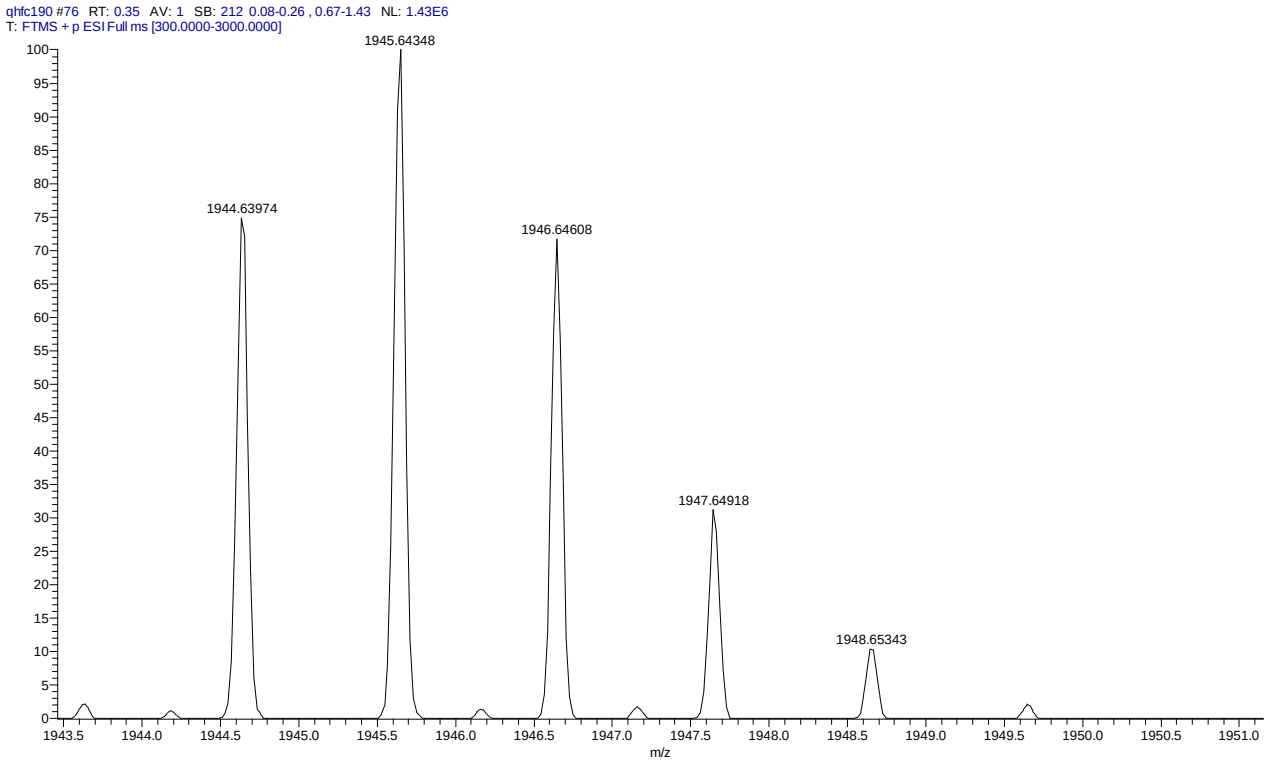
Analysis Info

Sample Name :	Zk-OATe4	Reference No.:	Qhfc190
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{120}H_{212}N_{10}O_8$
Experimental Mass $[M+Na]^+$:	1945.64348
Theoretical Mass $[M+Na]^+$:	1945.64142
Error (ppm) :	1.0





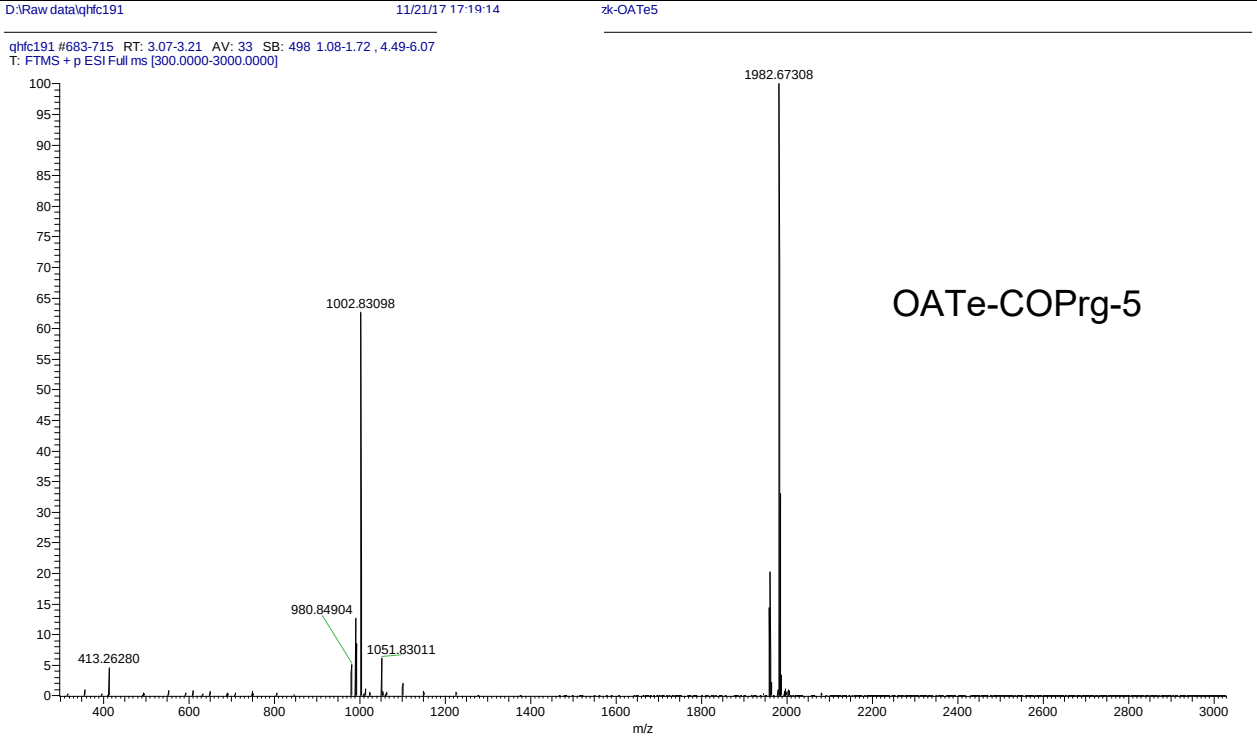
Thermo QEFMS Analysis Report

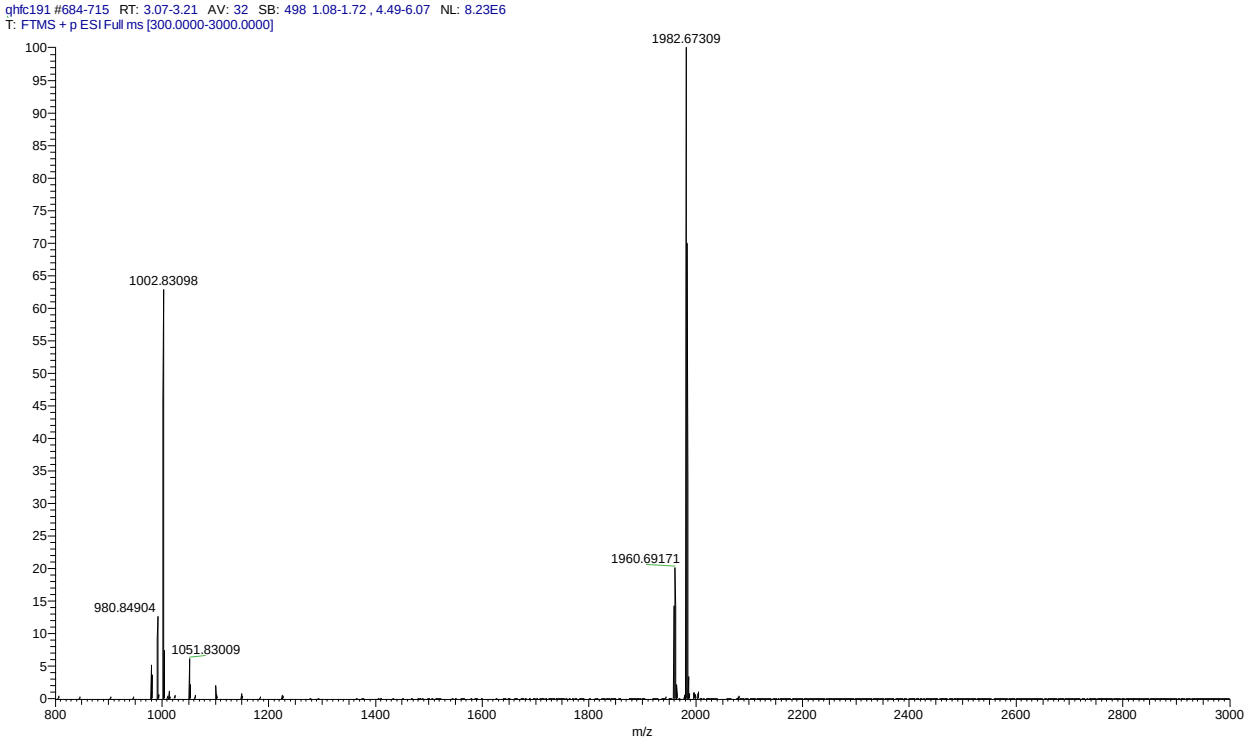
Analysis Info

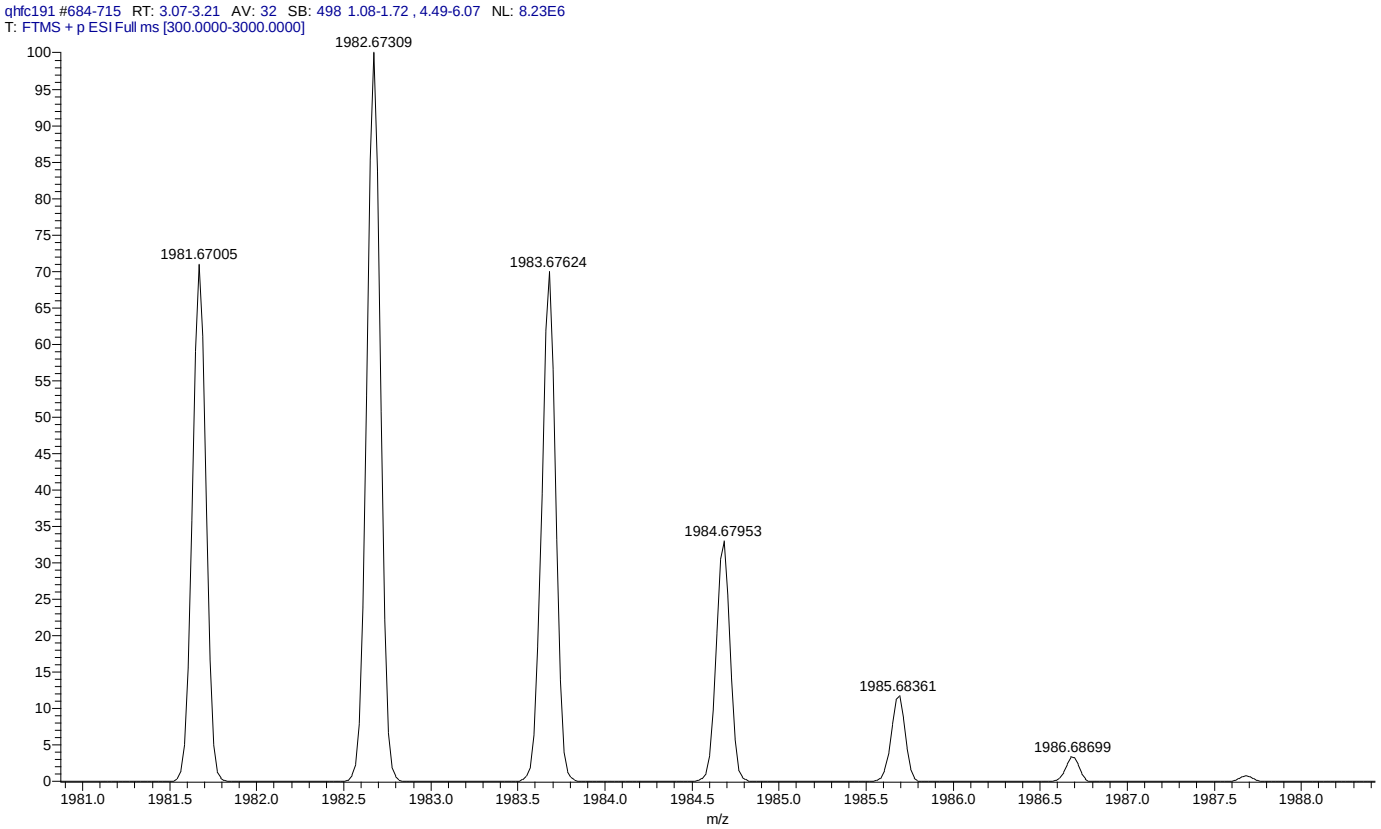
Sample Name :	Zk-OATe5	Reference No.:	Qhfc191
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

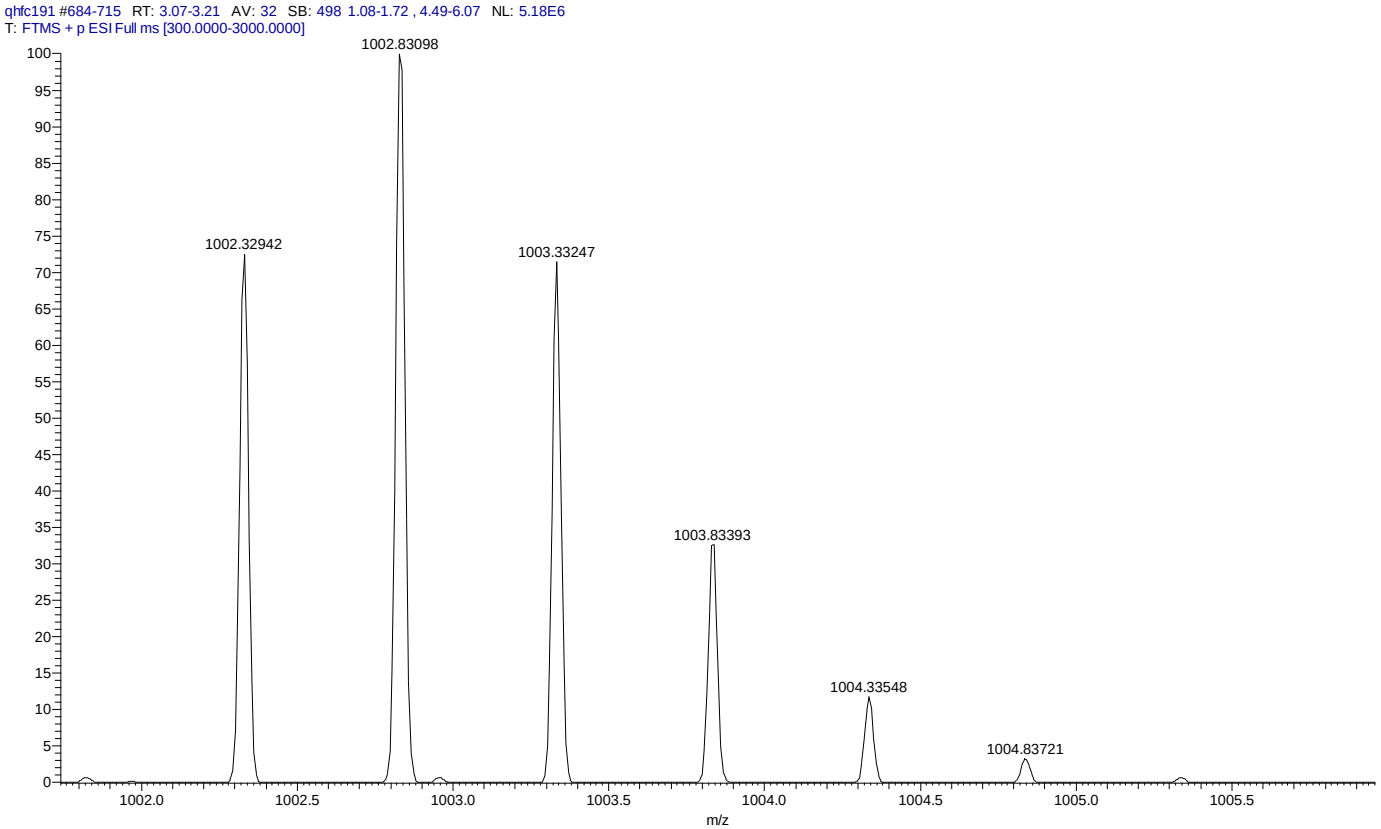
Accurate Mass Measurement

Molecular formula :	$C_{123}H_{215}N_{11}O_7$
Experimental Mass $[M+Na]^+$, $[M+2Na]^{2+}$:	1982.67308, 1002.83098
Theoretical Mass $[M+Na]^+$, $[M+2Na]^{2+}$:	1982.67304, 1002.83113
Error (ppm) :	0.0, 0.1






qhfc191 #684-715 RT: 3.07-3.21 AV: 32 SB: 498 1.08-1.72 , 4.49-6.07 NL: 8.23E6
T: FTMS + p ESI Full ms [300.0000-3000.0000]
1982.67309
1981.67005
1983.67624
1984.67953
1985.68361
1986.68699
m/z


qhfc191 #684-715 RT: 3.07-3.21 AV: 32 SB: 498 1.08-1.72 , 4.49-6.07 NL: 5.18E6
T: FTMS + p ESI Full ms [300.0000-3000.0000]
1002.83098
1002.32942
1003.33247
1003.83393
1004.33548
1004.83721
m/z

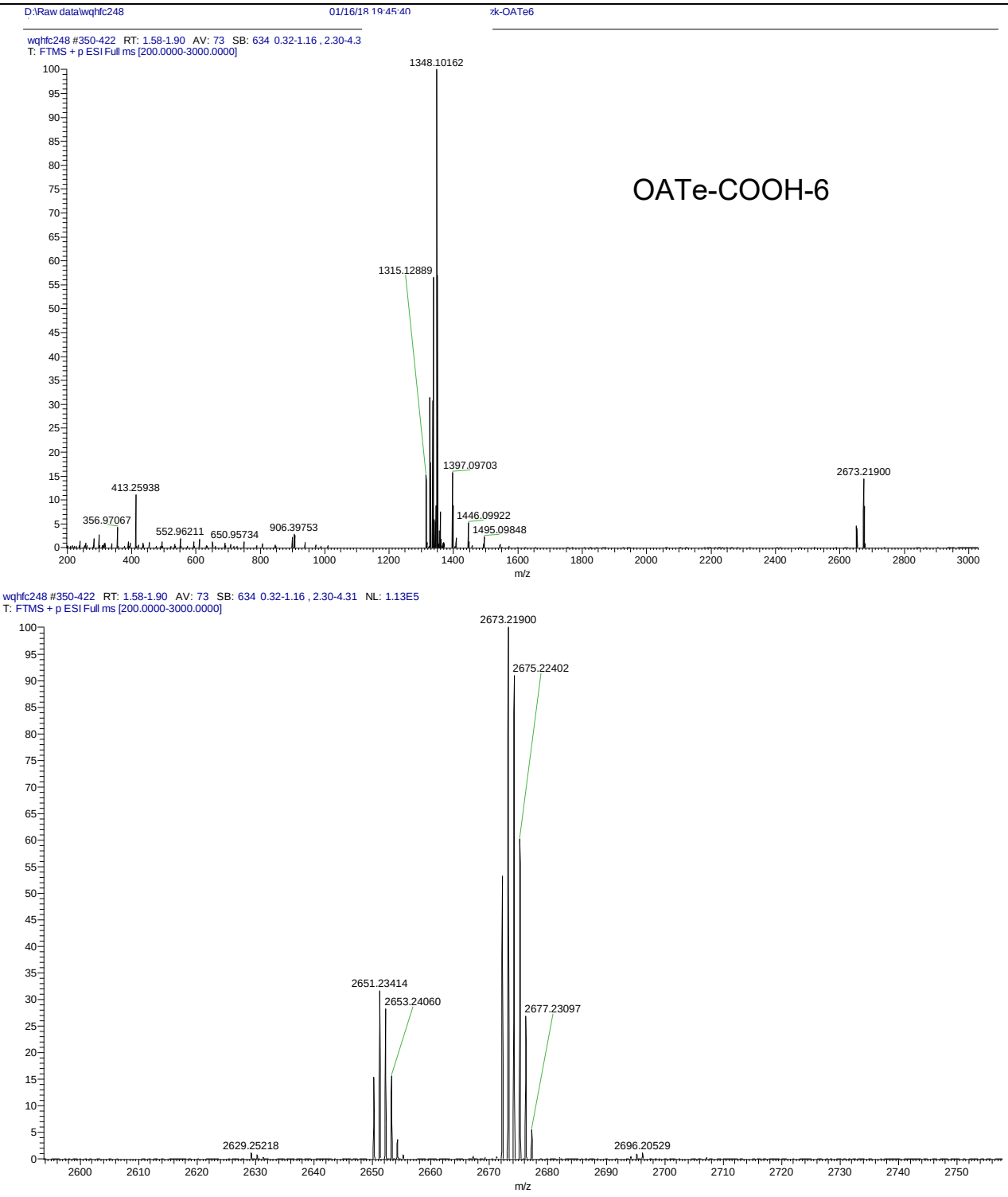
Thermo QEFMS Analysis Report

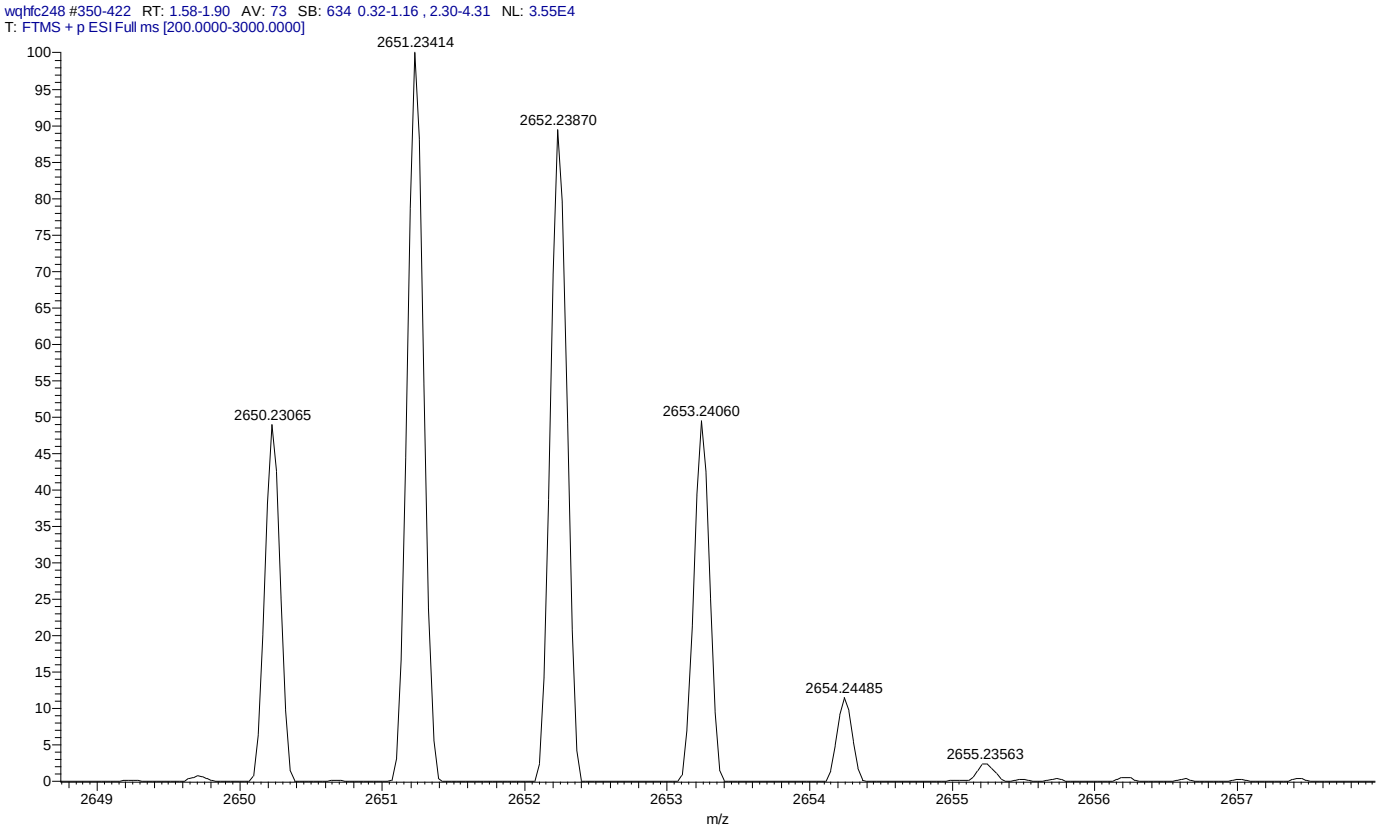
Analysis Info

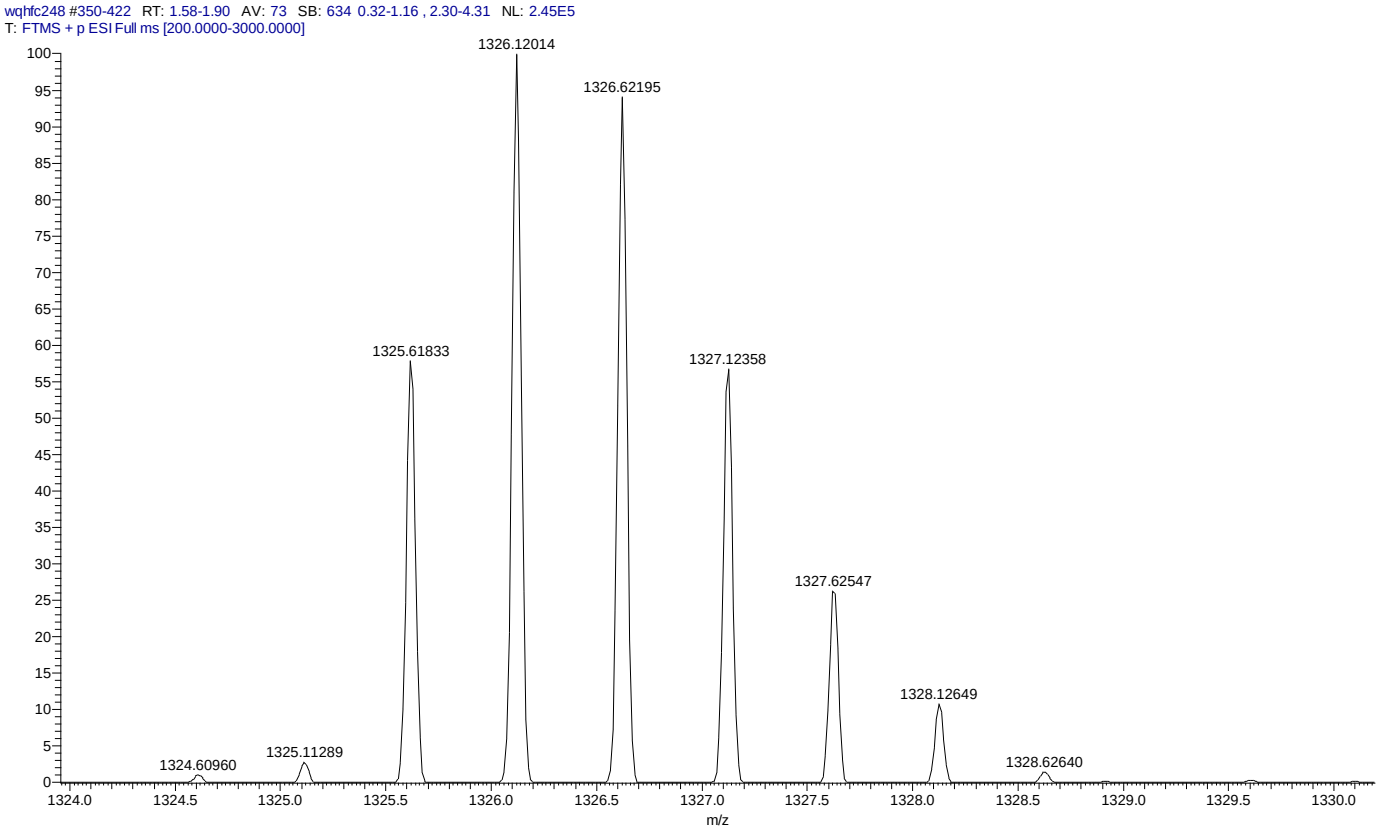
Sample Name :	Zk-OATe6	Reference No.:	Wqhfc248
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{164}H_{287}N_{15}O_{10}$
Experimental Mass $[M+Na]^+$, $[M+2Na]^{2+}$:	2651.23414, 1326.12014
Theoretical Mass $[M+Na]^+$, $[M+2Na]^{2+}$:	2651.23348, 1326.12038
Error (ppm) :	0.2, 0.1




wqhfc248 #350-422 RT: 1.58-1.90 AV: 73 SB: 634 0.32-1.16 , 2.30-4.31 NL: 3.55E4
T: FTMS + p ESI Full ms [200.0000-3000.0000]
2651.23414
2652.23870
2650.23065
2653.24060
2654.24485
2655.23563
m/z


wqhfc248 #350-422 RT: 1.58-1.90 AV: 73 SB: 634 0.32-1.16 , 2.30-4.31 NL: 2.45E5
T: FTMS + p ESI Full ms [200.0000-3000.0000]
1326.12014
1326.62195
1325.61833
1327.12358
1327.62547
1328.12649
1324.60960
1325.11289
1328.62640
m/z

Bruker Autoflex speed MALDI-TOF MS Analysis Report

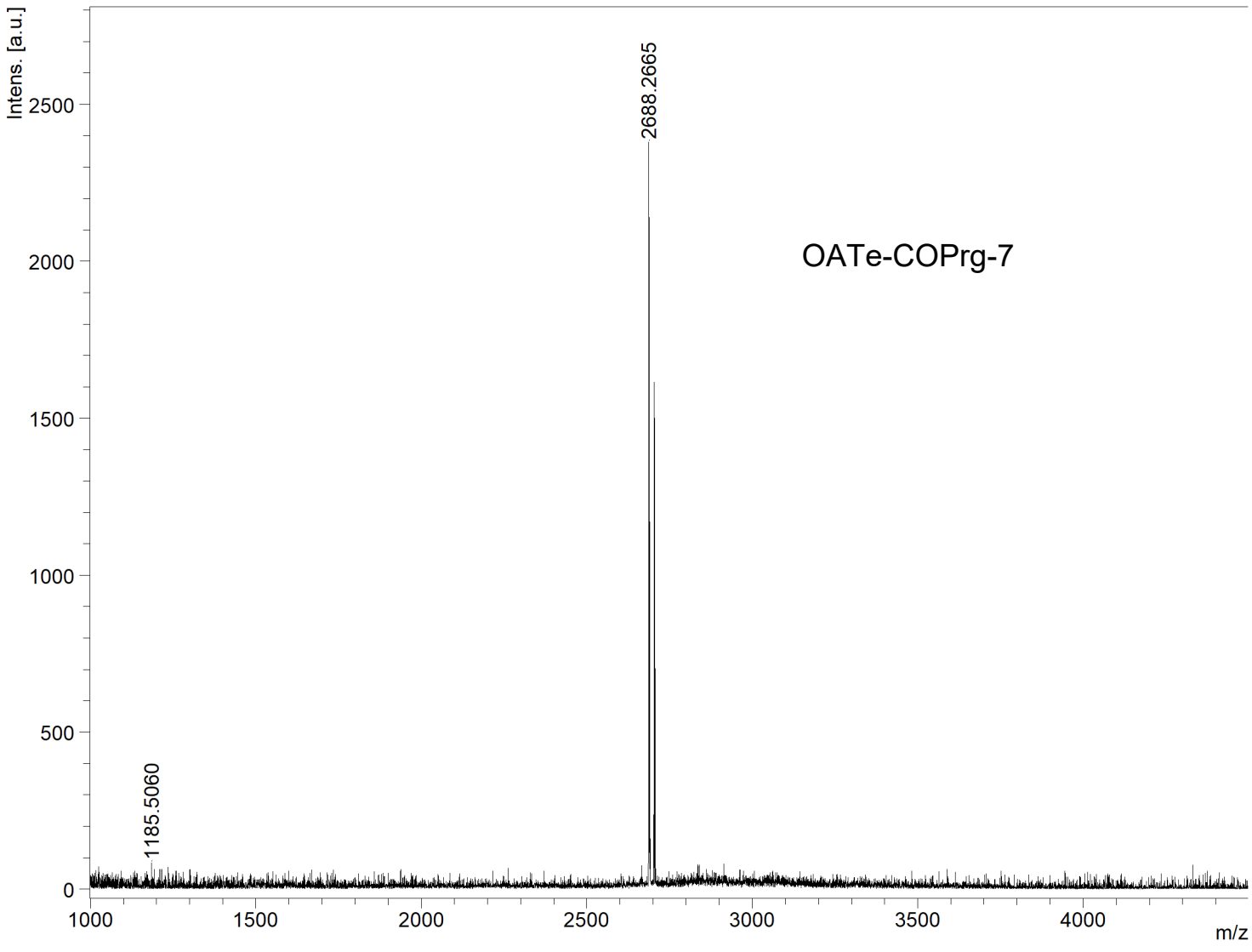
Department of Chemistry, The Chinese University of Hong Kong

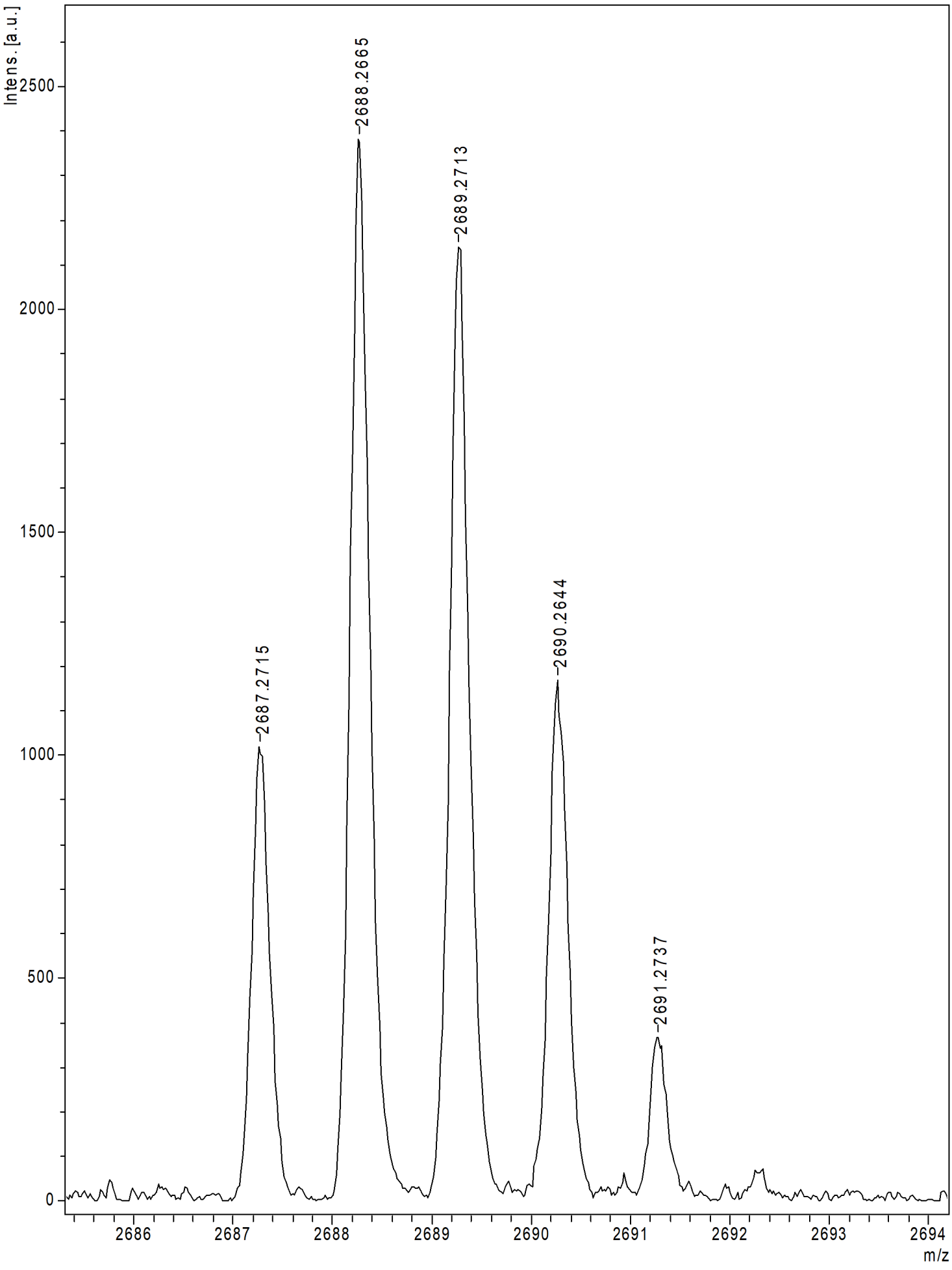
Analysis Info

Sample Name:	ZK-OATe7	Reference No.:	wfhfc073\0_C7\2
Applicant Name:	Zheng Kun	Date of Analysis :	2018-04-25T15:01:56.000
Method:	D:\Methods\flexControlMethods\Test_RP_1000-4500_Da.par		
Polarity:	POS	PIE delay: 150 ns	No. of shots: 2400
Comment:	Reflector mode, DCTB as matrix, 384 polished stainless steel target plate		

Accurate Mass Measurement

Molecular formula:	C167H290N16O9
Abundant Isotopic (theoretical) [M+Na]+:	2688.2651
Experimental [M+Na]+:	2688.2665
Error (ppm):	0.52




Intens. [a.u.]
2500
2000
1500
1000
500
0
2687.2715
2688.2665
2689.2713
2690.2644
2691.2737
2686
2687
2688
2689
2690
2691
2692
2693
2694
m/z

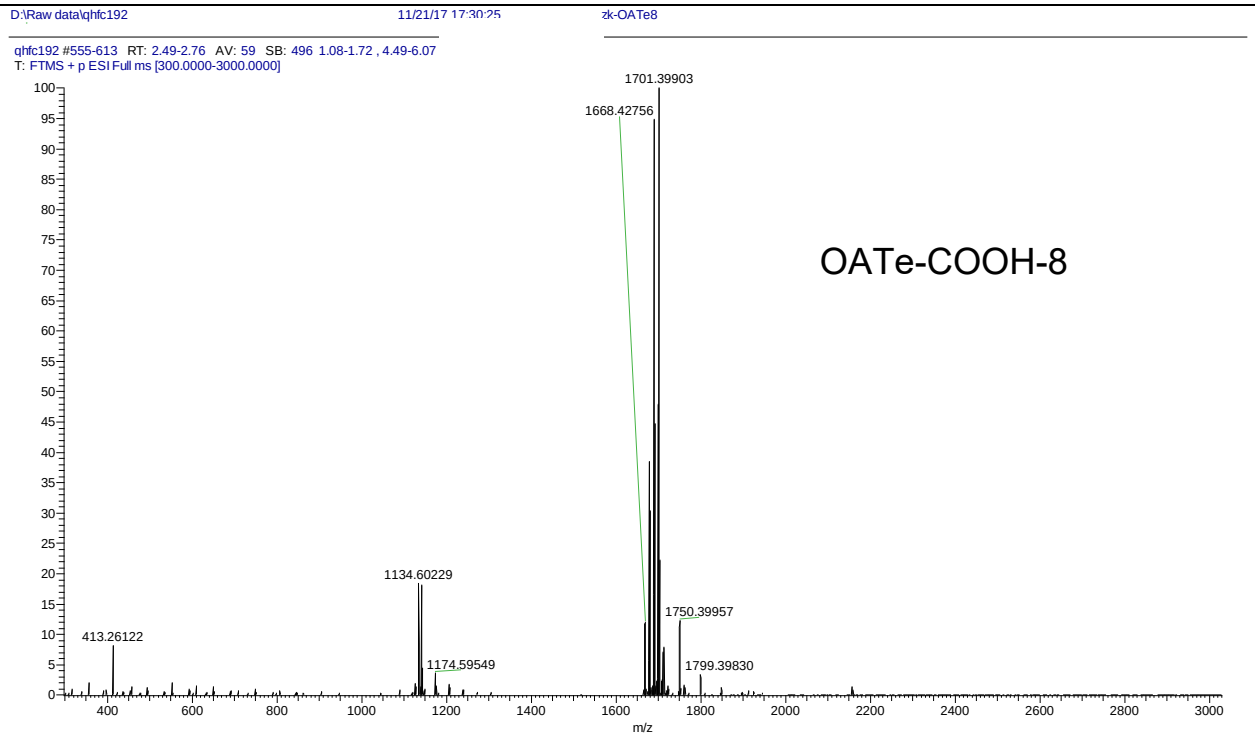
Thermo QEFMS Analysis Report

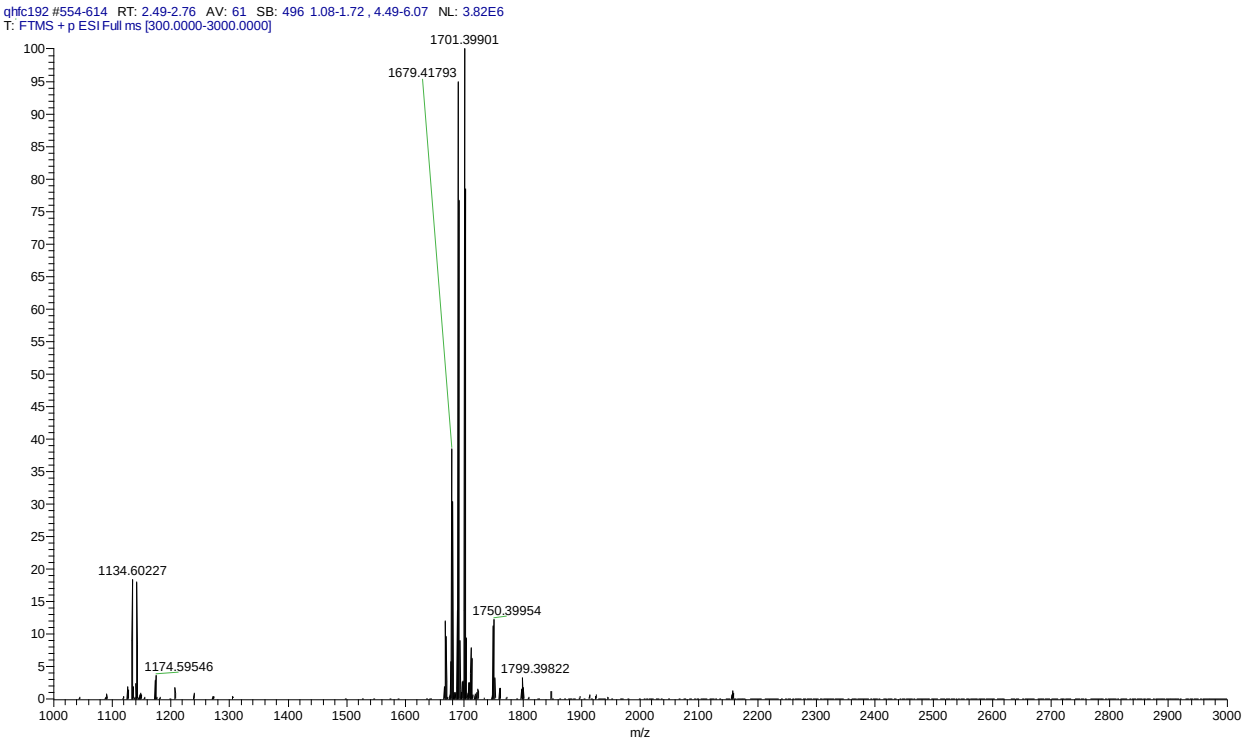
Analysis Info

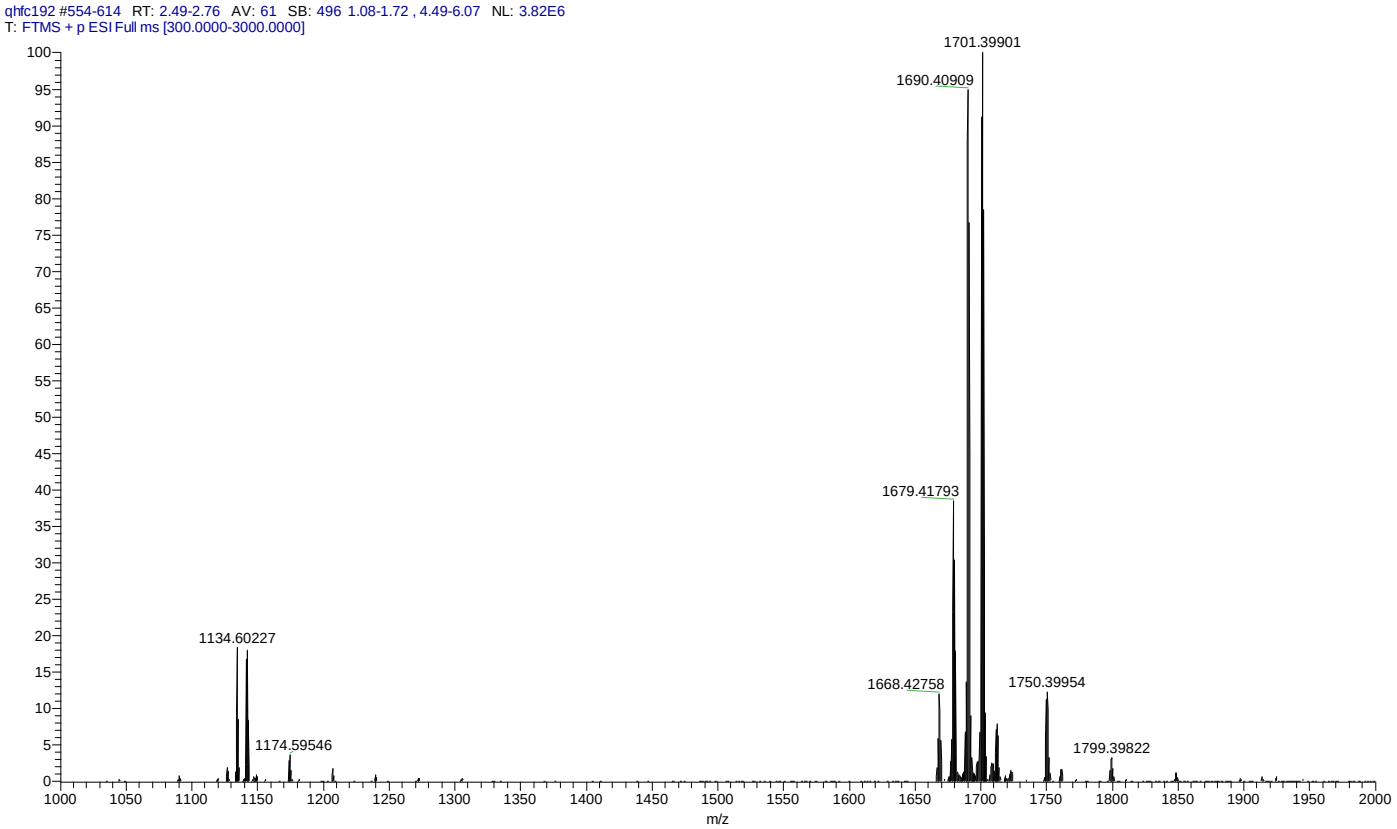
Sample Name :	Zk-OATe8	Reference No.:	Qhfc192
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{208}H_{362}N_{20}O_{12}$
Experimental Mass $[M+2Na]^{2+}$, $[M+3Na]^{3+}$:	1690.40909, 1134.60229
Theoretical Mass $[M+2Na]^{2+}$, $[M+3Na]^{3+}$:	1690.40898, 1134.60239
Error (ppm) :	0.0, 0.0






qhfc192 #554-614 RT: 2.49-2.76 AV: 61 SB: 496 1.08-1.72 , 4.49-6.07 NL: 3.82E6
T: FTMS + p ESI Full ms [300.0000-3000.0000]
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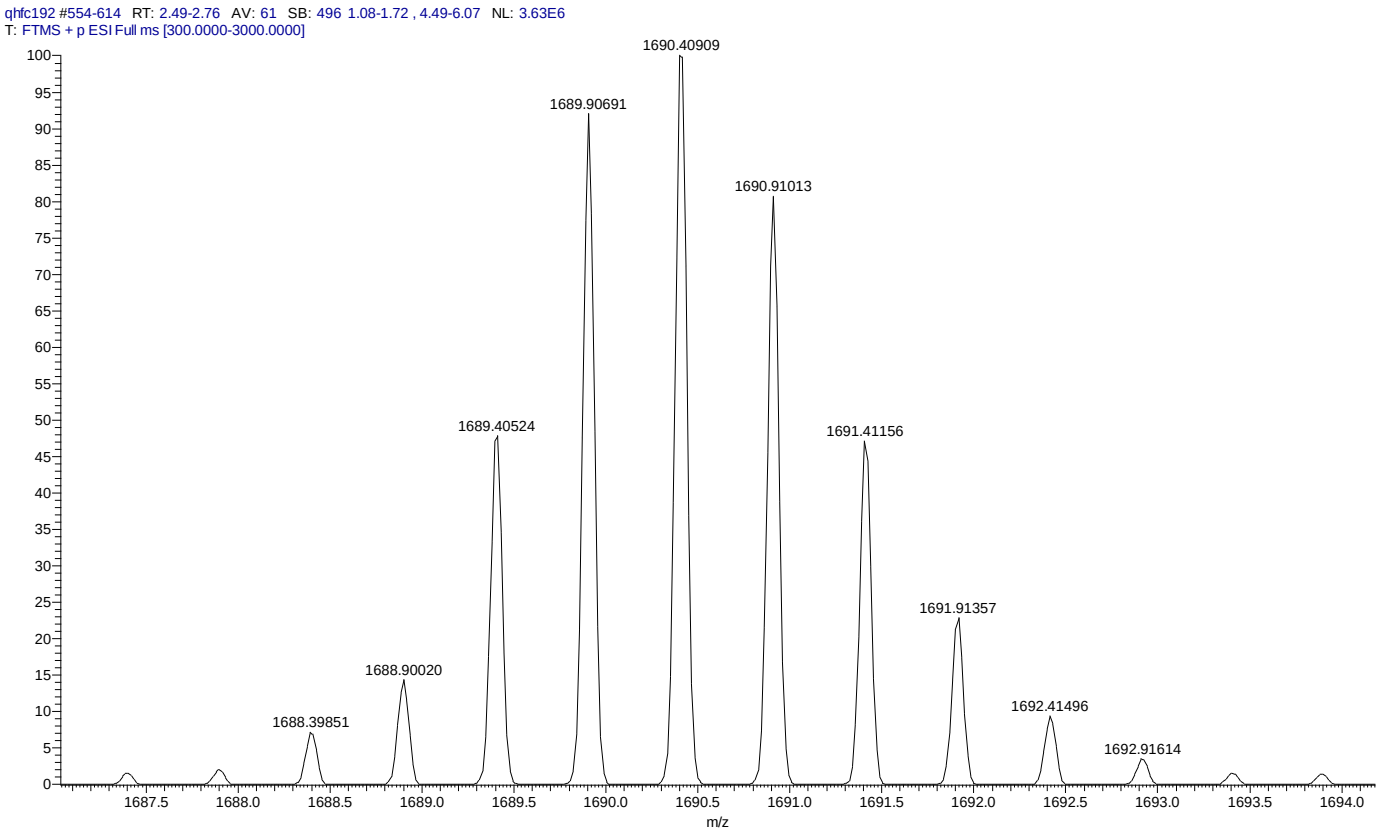
1690.40909
1679.41793
1134.60227
1668.42758
1750.39954
1174.59546
1799.39822
m/z


qhfc192 #554-614 RT: 2.49-2.76 AV: 61 SB: 496 1.08-1.72 , 4.49-6.07 NL: 3.63E6
T: FTMS + p ESI Full ms [300.0000-3000.0000]
1690.40909
1689.90691
1690.91013
1689.40524
1691.41156
1691.91357
1688.90020
1688.39851
1692.41496
1692.91614
m/z

Bruker Autoflex speed MALDI-TOF MS Analysis Report

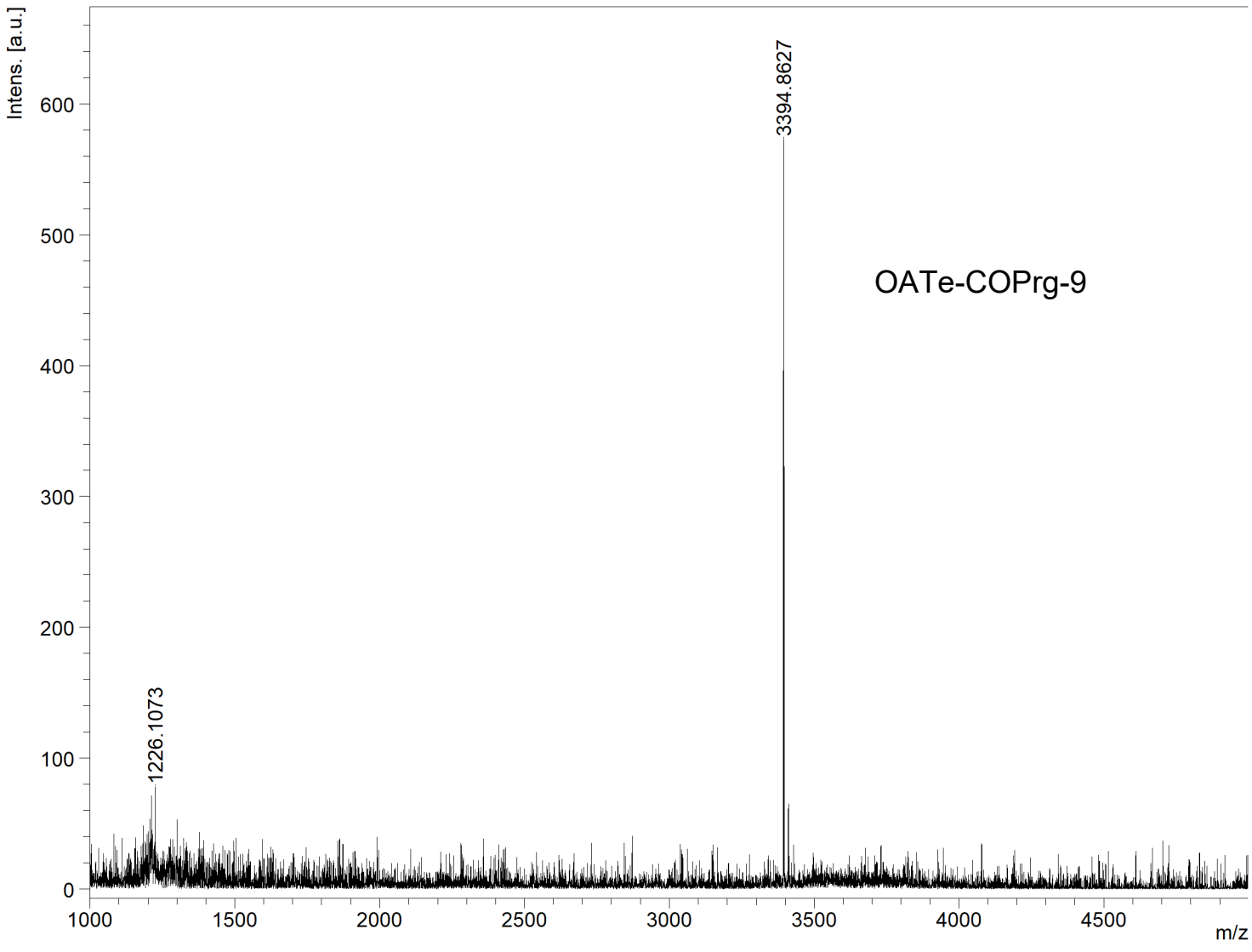
Department of Chemistry, The Chinese University of Hong Kong

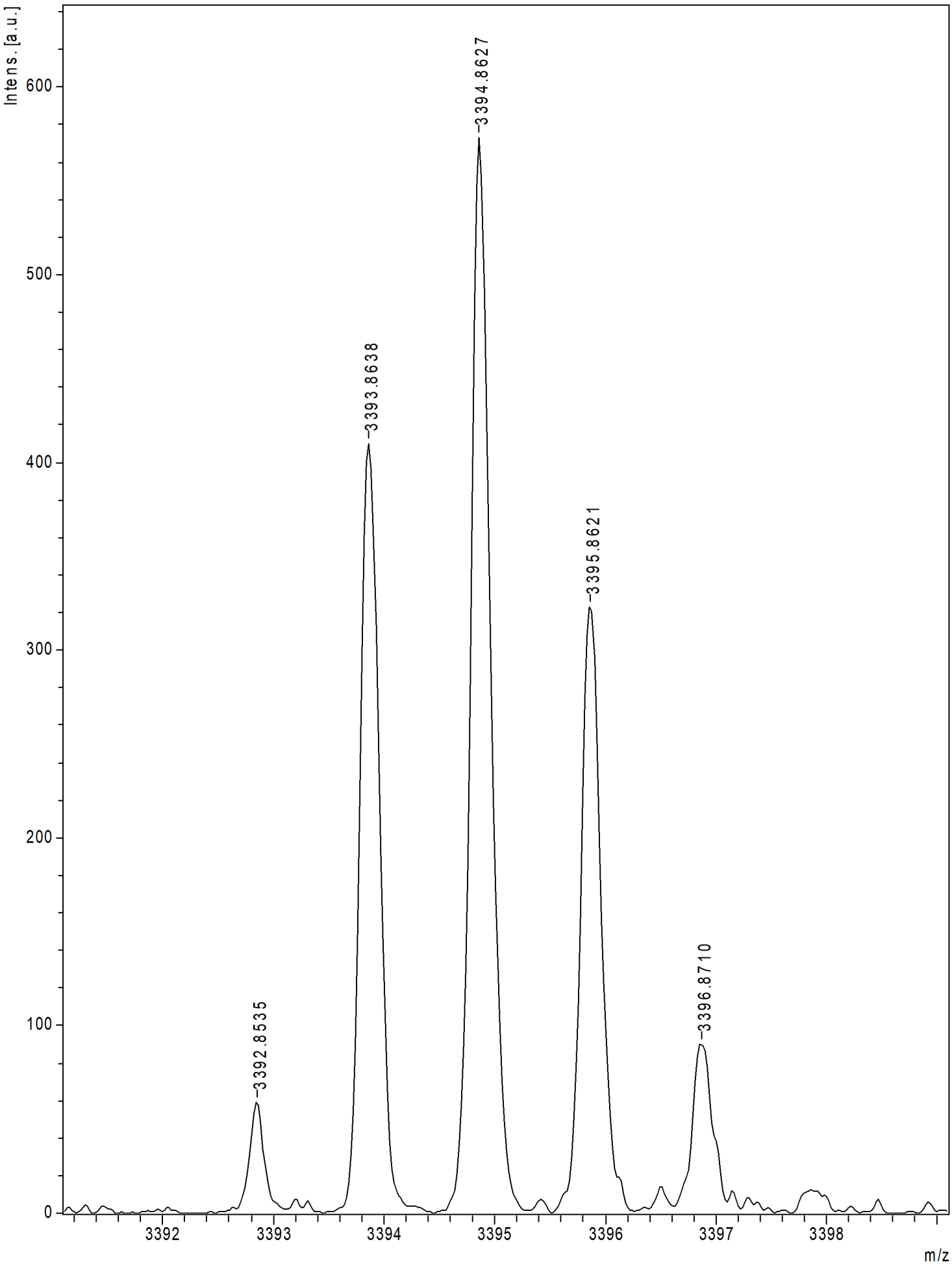
Analysis Info

Sample Name:	zk-OATe-9	Reference No.:	wfhfc069\0_C2\2
Applicant Name:	Zheng Kun	Date of Analysis :	2018-02-13T15:26:23.000
Method:	D:\Methods\flexControlMethods\Test_RP_1000-4500_Da.par		
Polarity:	POS	PIE delay: 150 ns	No. of shots: 600
Comment:	Reflector mode, DCTB as matrix, 384 polished stainless steel target plate		

Accurate Mass Measurement

Molecular formula:	C211H365N21O11
Abundant Isotopic (theoretical) [M+Na]+:	3394.8604
Experimental [M+Na]+:	3394.8627
Error (ppm):	0.68




Intens. [a.u.]
600
500
400
300
200
100
0
3392.8535
3393.8638
3394.8627
3395.8621
3396.8710
3392
3393
3394
3395
3396
3397
3398
m/z

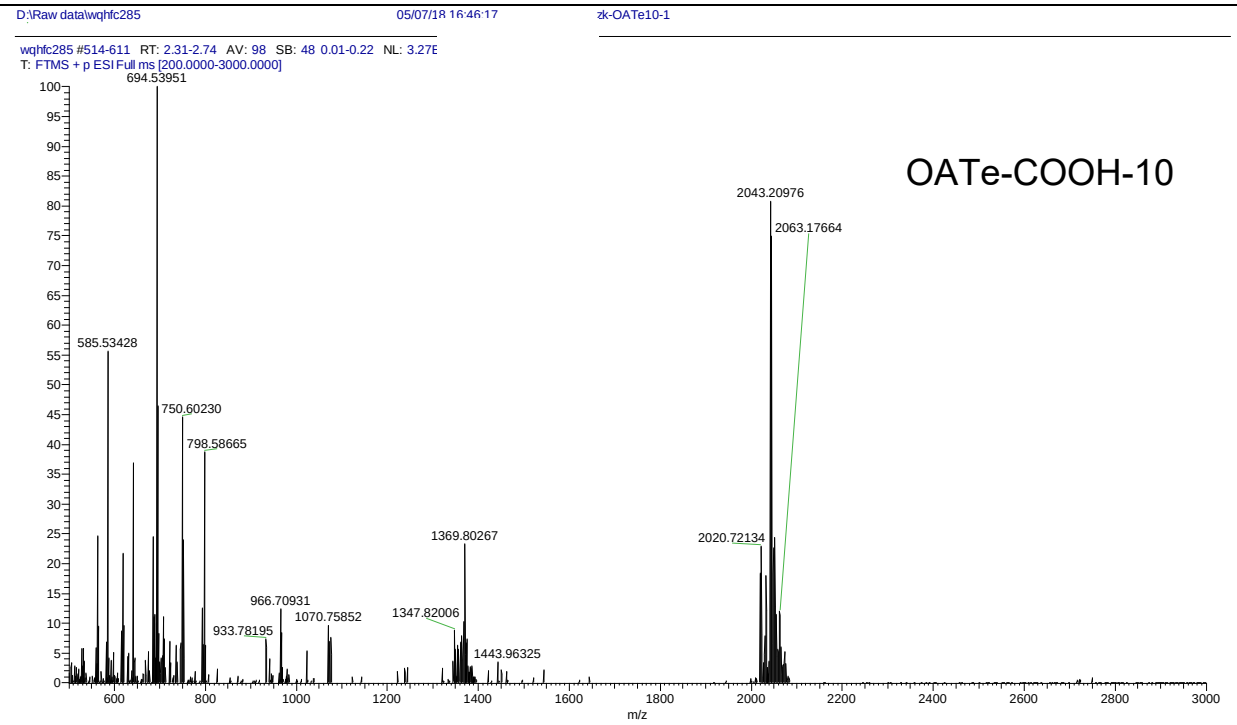
Thermo QEFMS Analysis Report

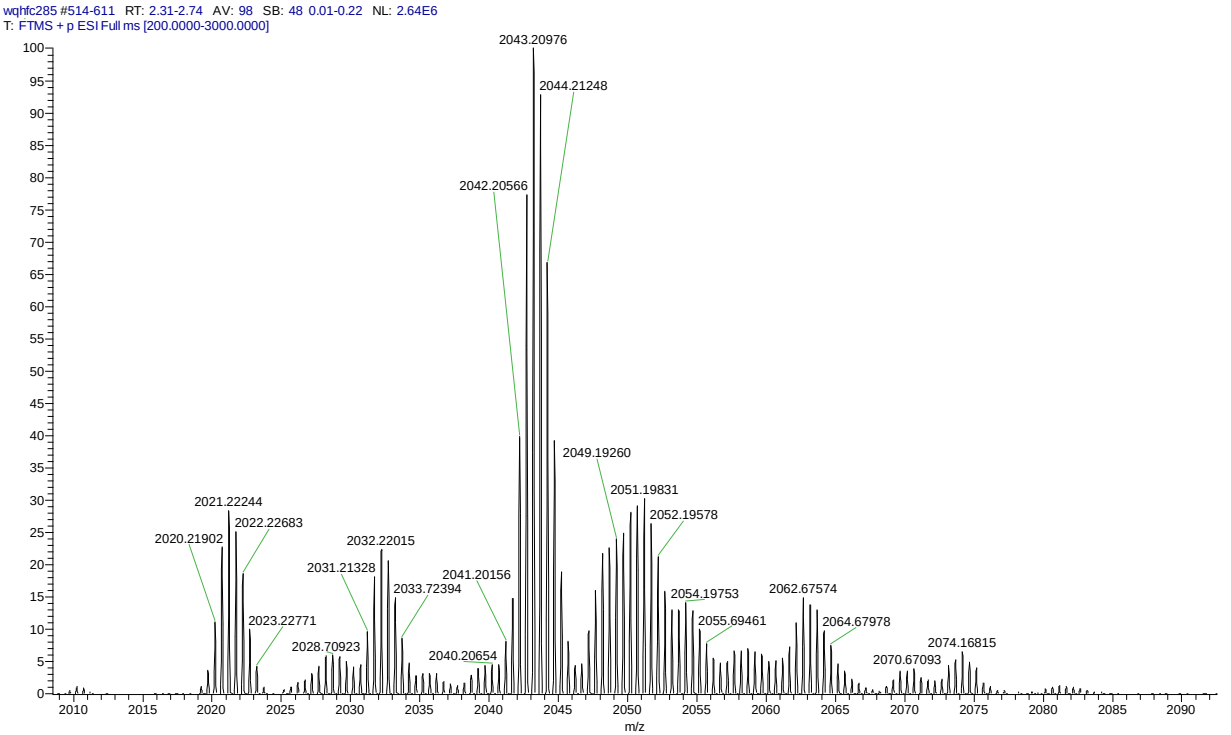
Analysis Info

Sample Name :	Zk-OATe10-1	Reference No.:	Wqhfc285
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	positive
Comment :	ESI pos, 3.5kV, by infusion, with sheath gas		

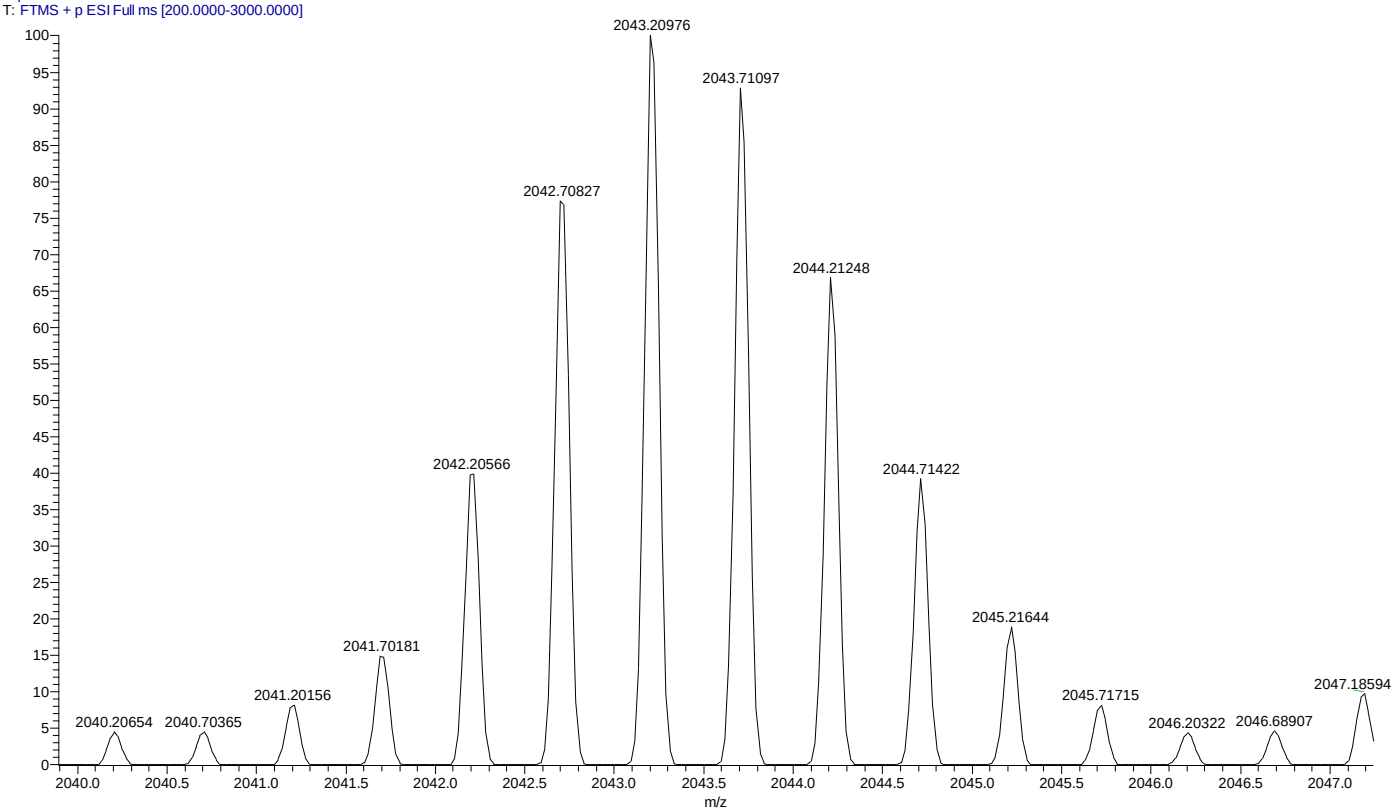
Accurate Mass Measurement

Molecular formula :	$C_{252}H_{437}N_{25}O_{14}$
Experimental Mass $[M+2Na]^{2+}$, $[M+2H]^{2+}$:	2043.20976, 2021.22244
Theoretical Mass $[M+2Na]^{2+}$, $[M+2H]^{2+}$:	2043.20501, 2021.22307
Error (ppm) :	2.3, 0.3





wqhfc285 #514-611 RT: 2.31-2.74 AV: 98 SB: 48 0.01-0.22 NL: 2.64E6
T: FTMS + p ESI Full ms [200.0000-3000.0000]



wqhfc285 #514-611 RT: 2.31-2.74 AV: 98 SB: 48 0.01-0.22 NL: 7.52E5
T: FTMS + p ESI Full ms [200.0000-3000.0000]