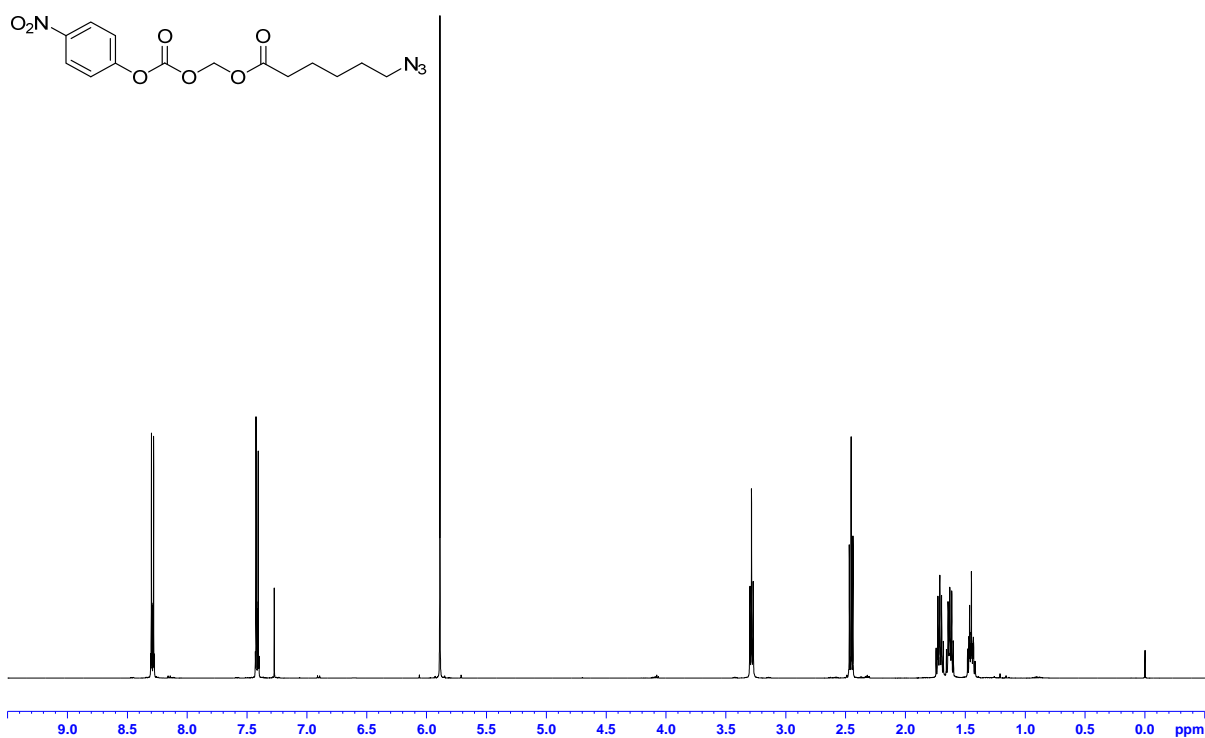
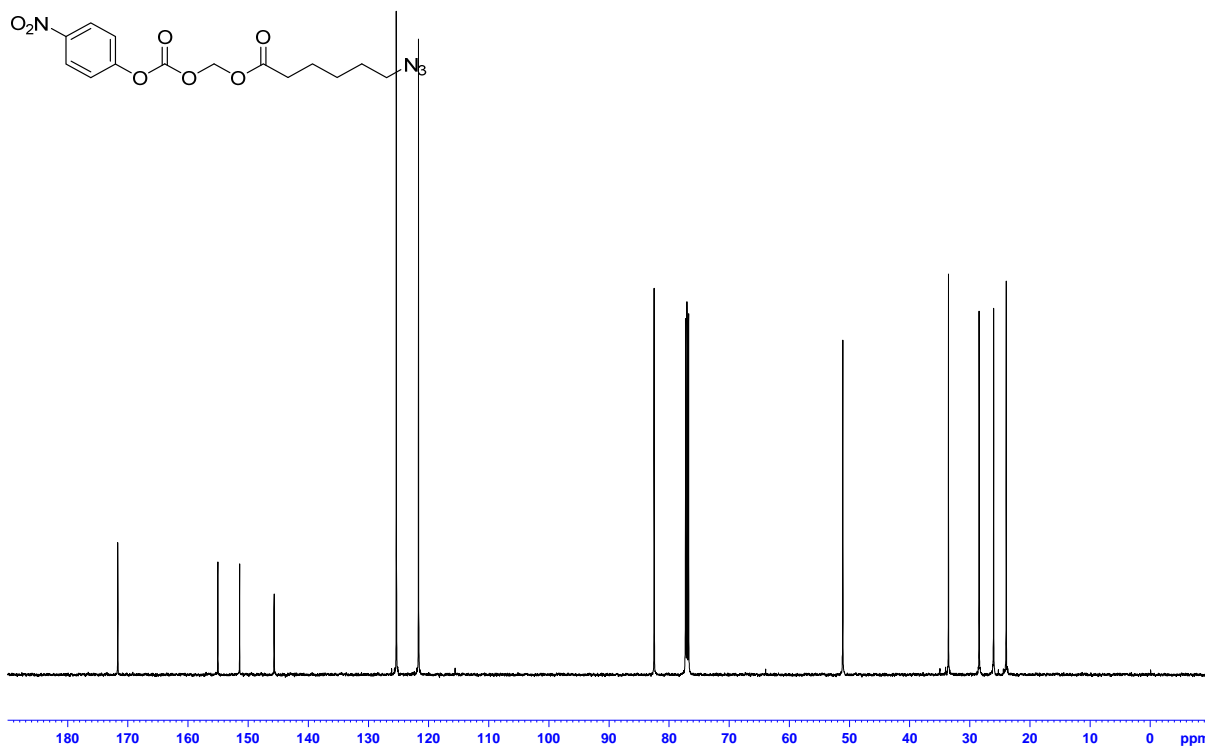


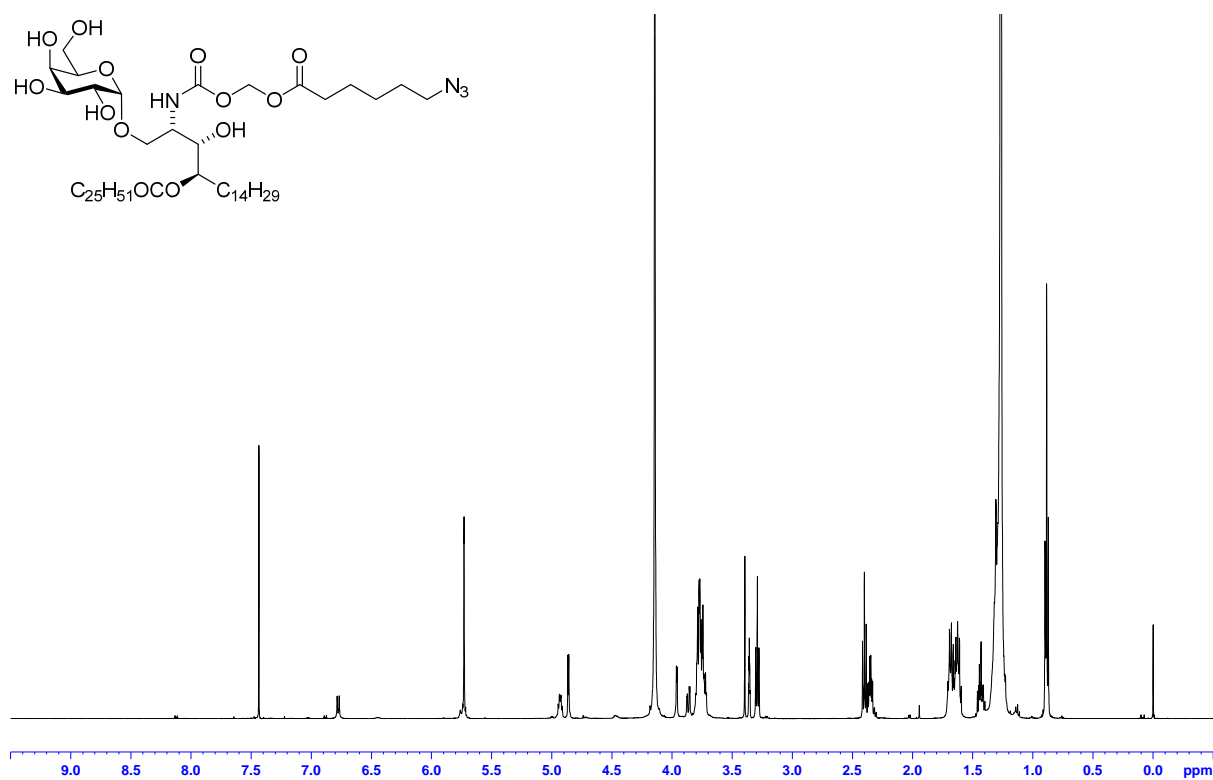
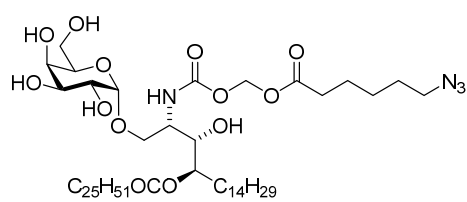
9 - ^1H NMR



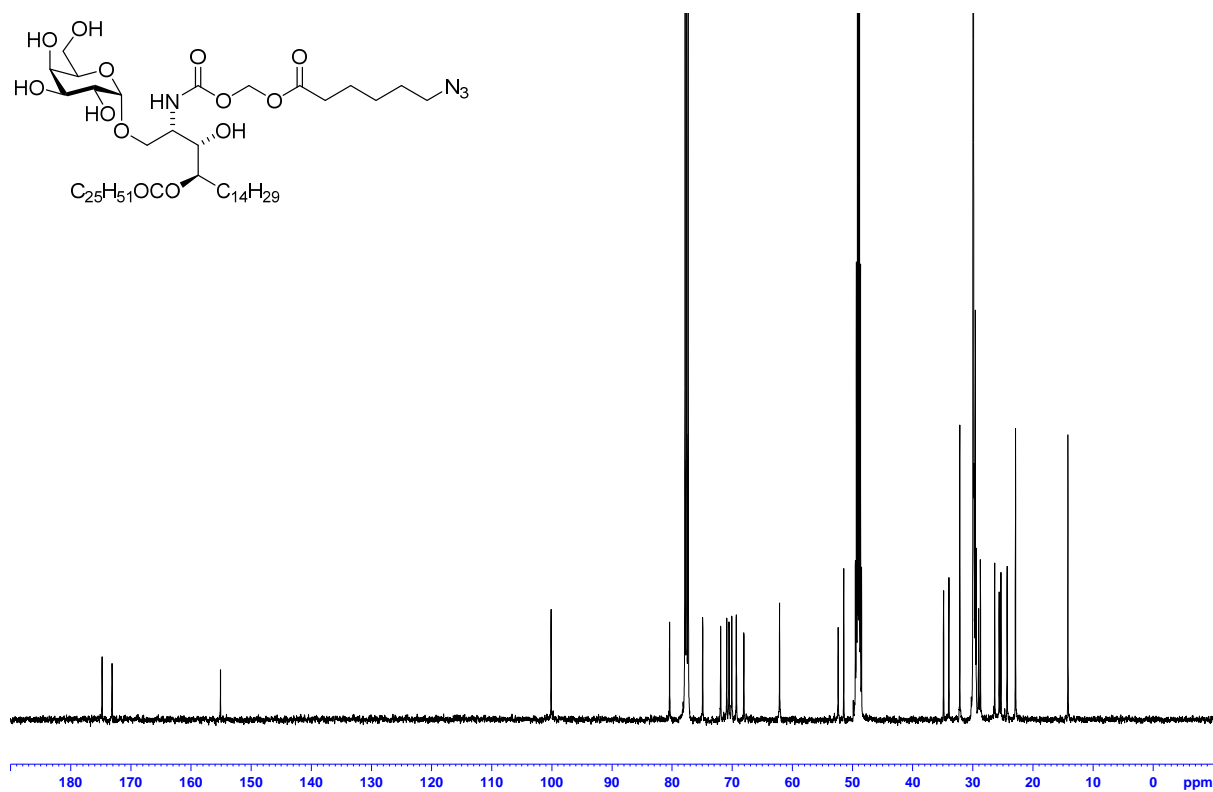
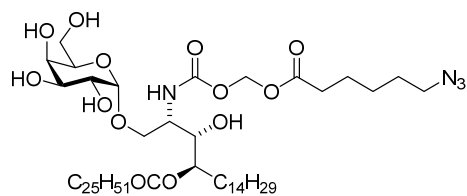
9 - ^{13}C NMR proton decoupled



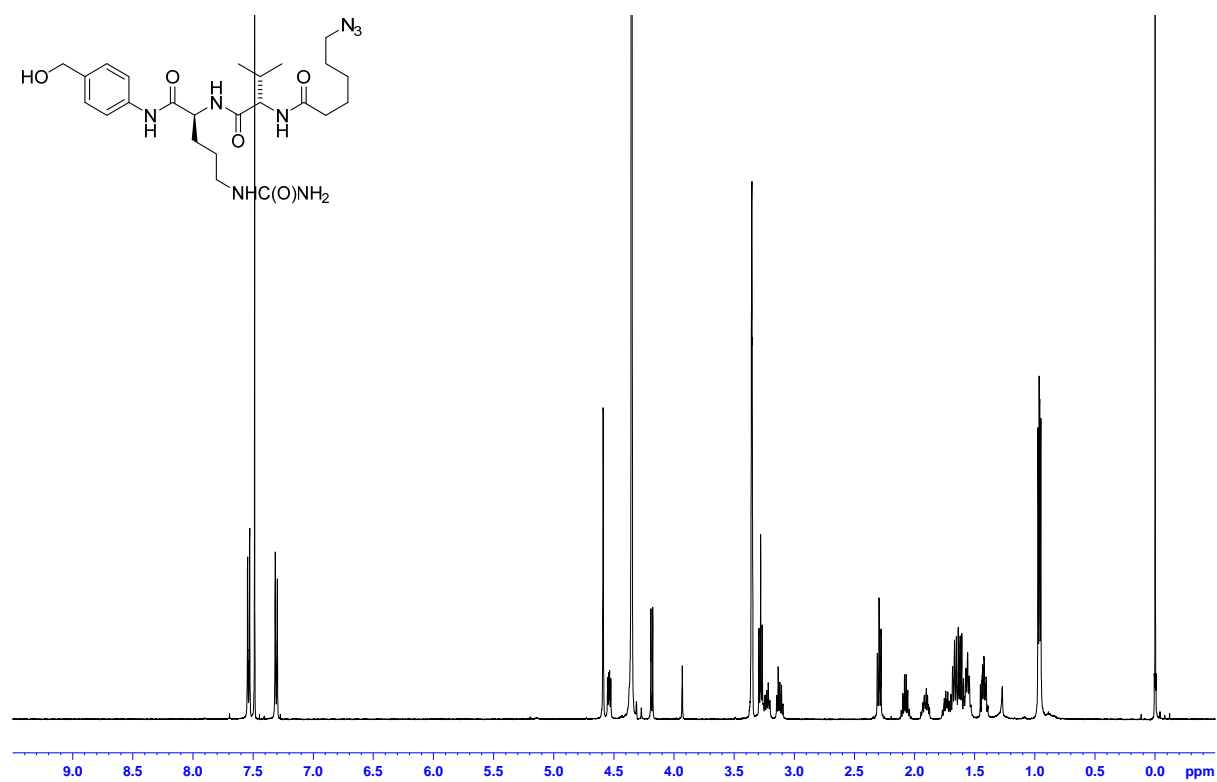
11 - ^1H NMR



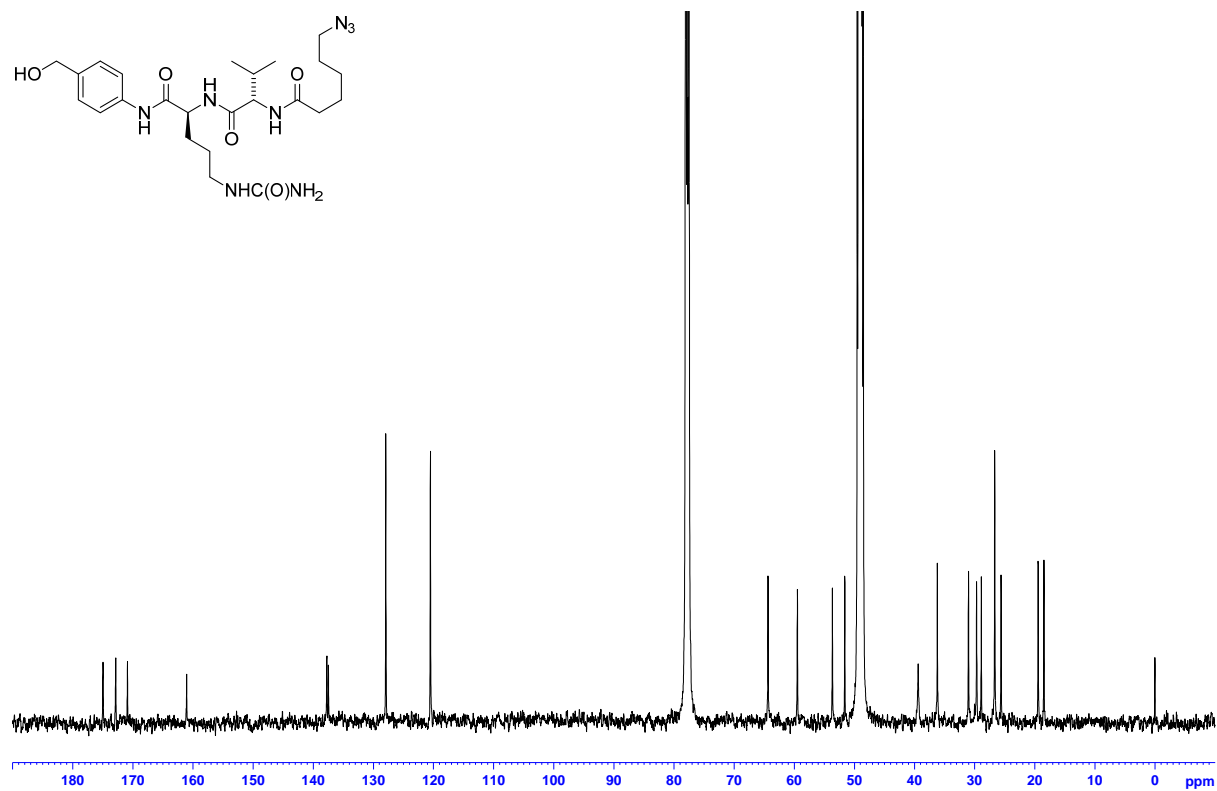
11 - ^{13}C NMR proton decoupled



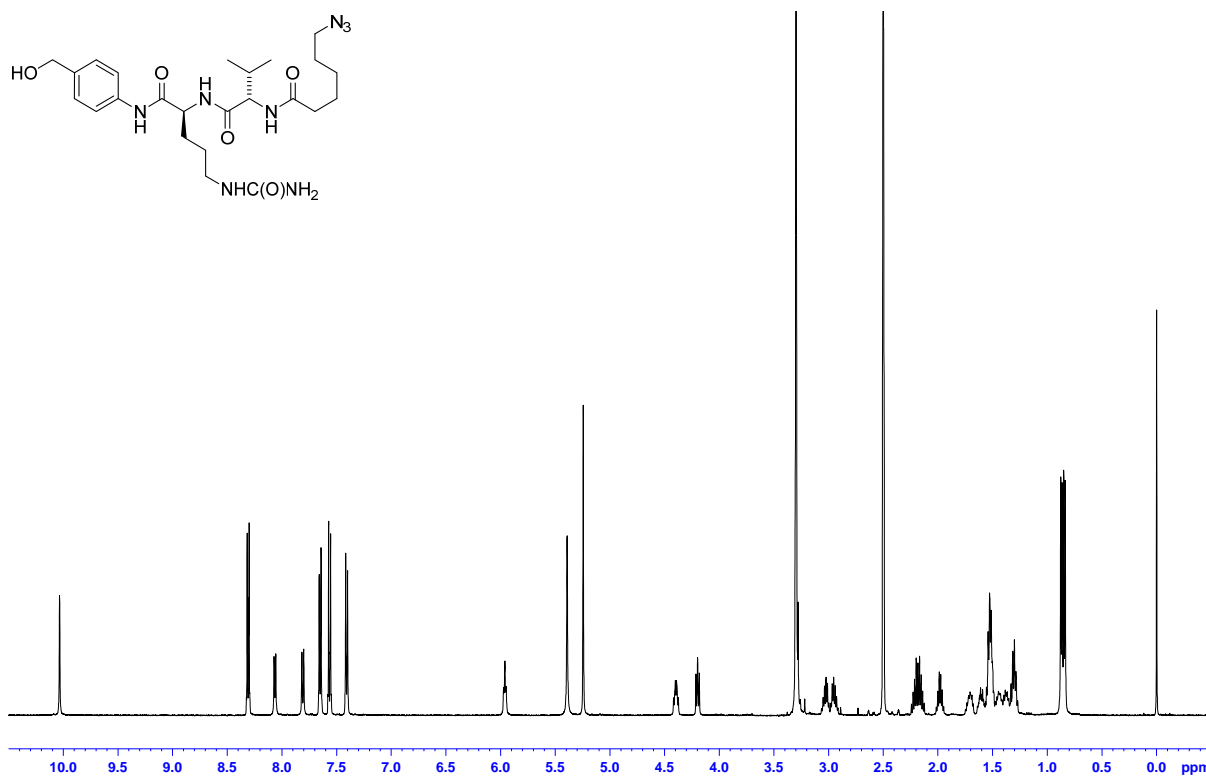
14 - ^1H NMR



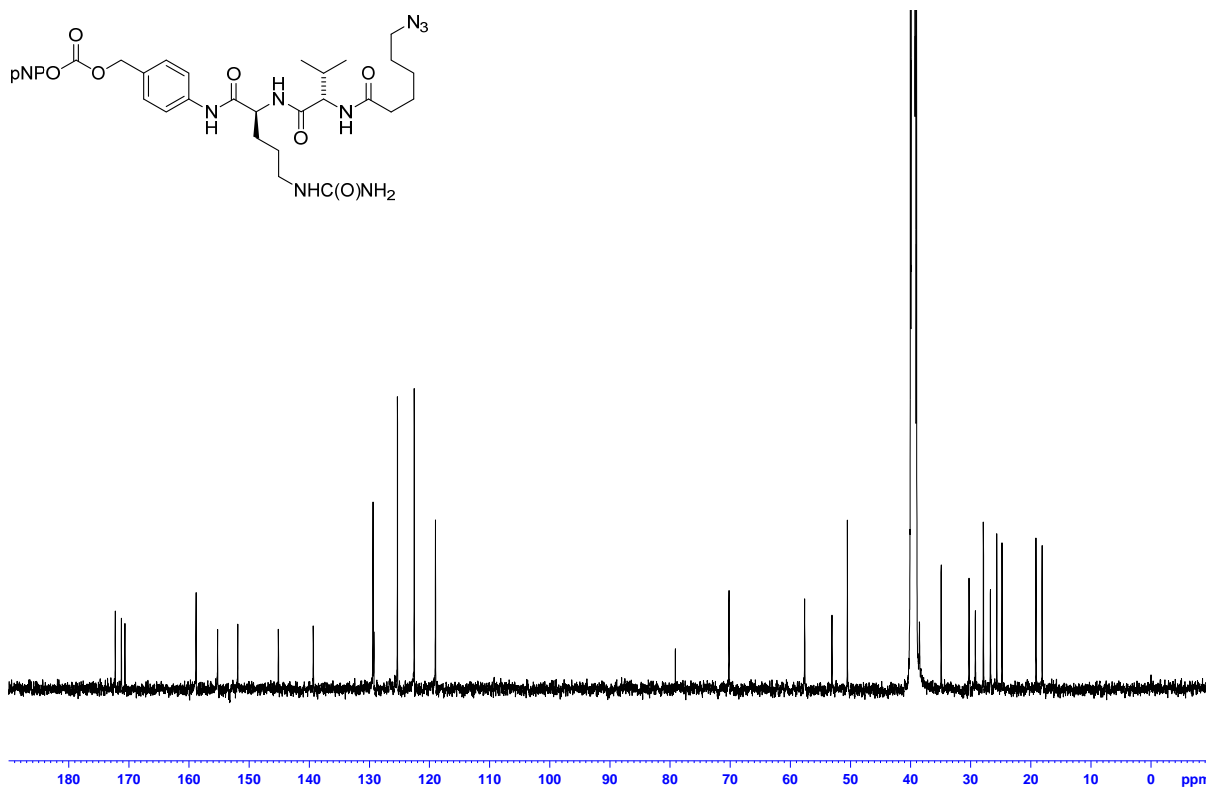
14 - ^{13}C NMR proton decoupled



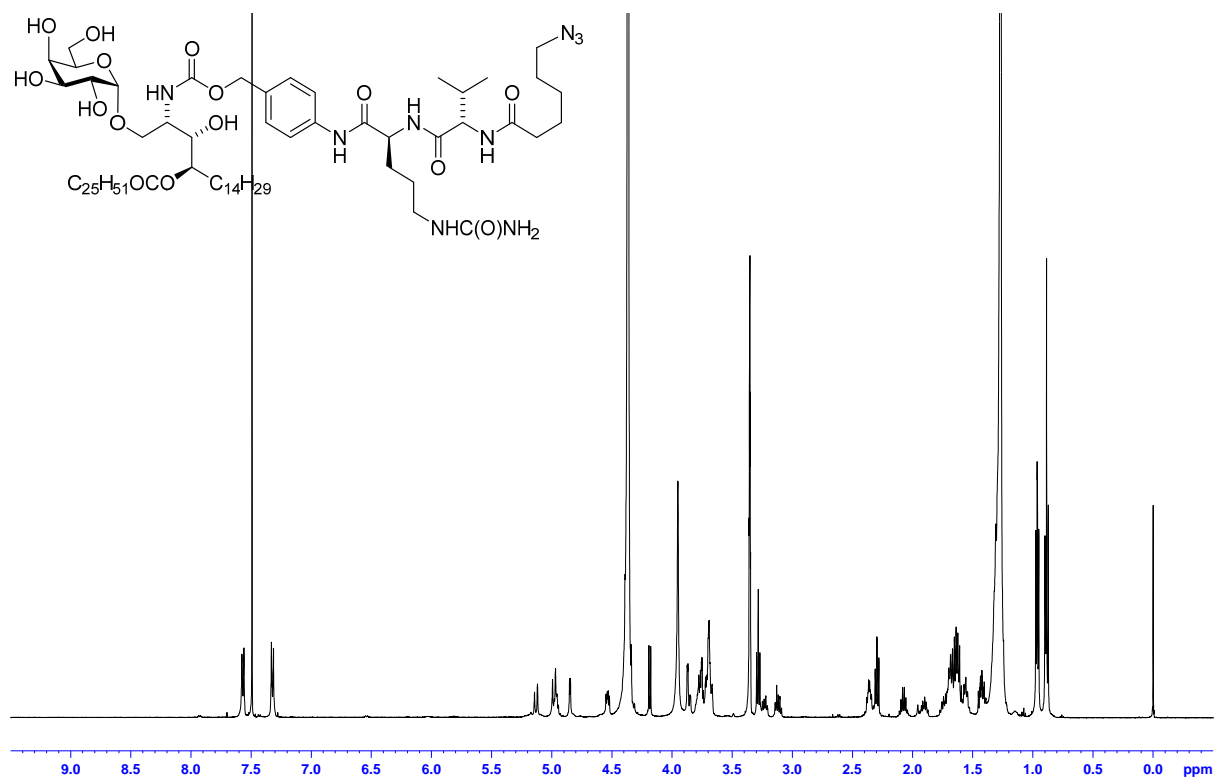
16 - ^1H NMR



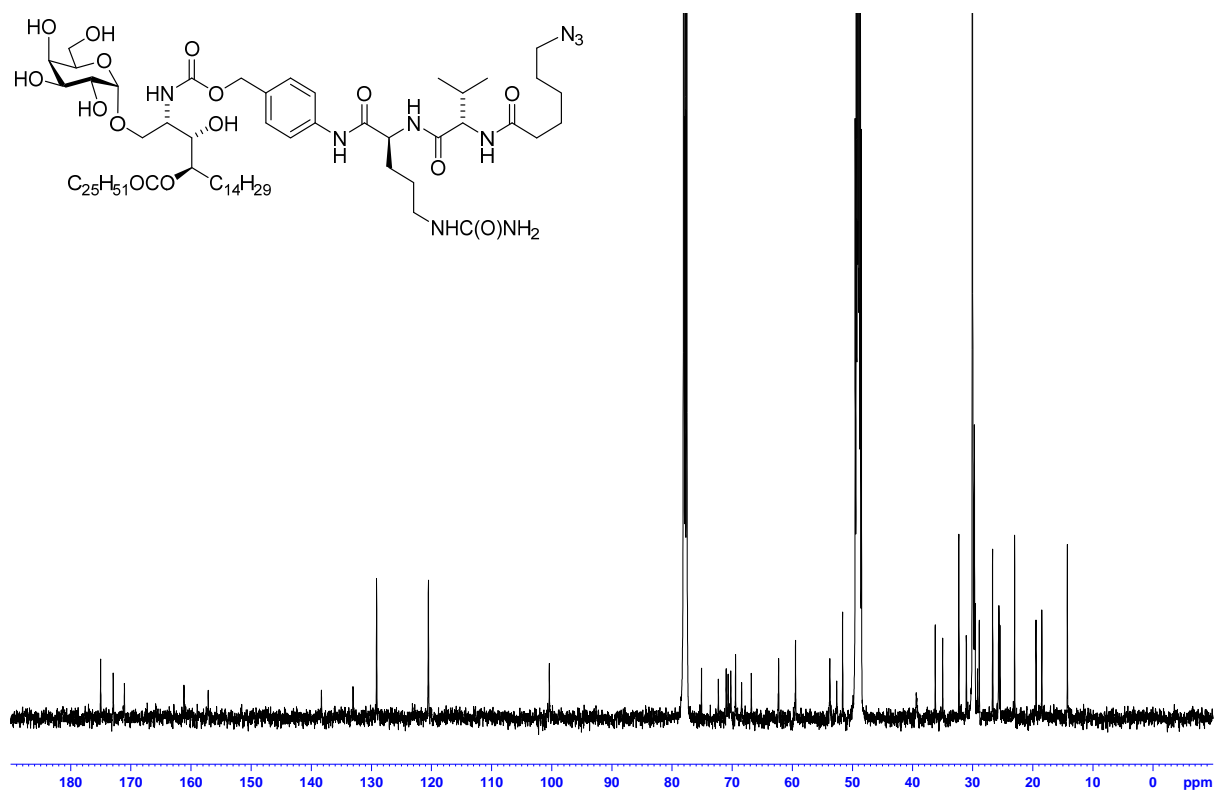
16 - ^{13}C NMR proton decoupled



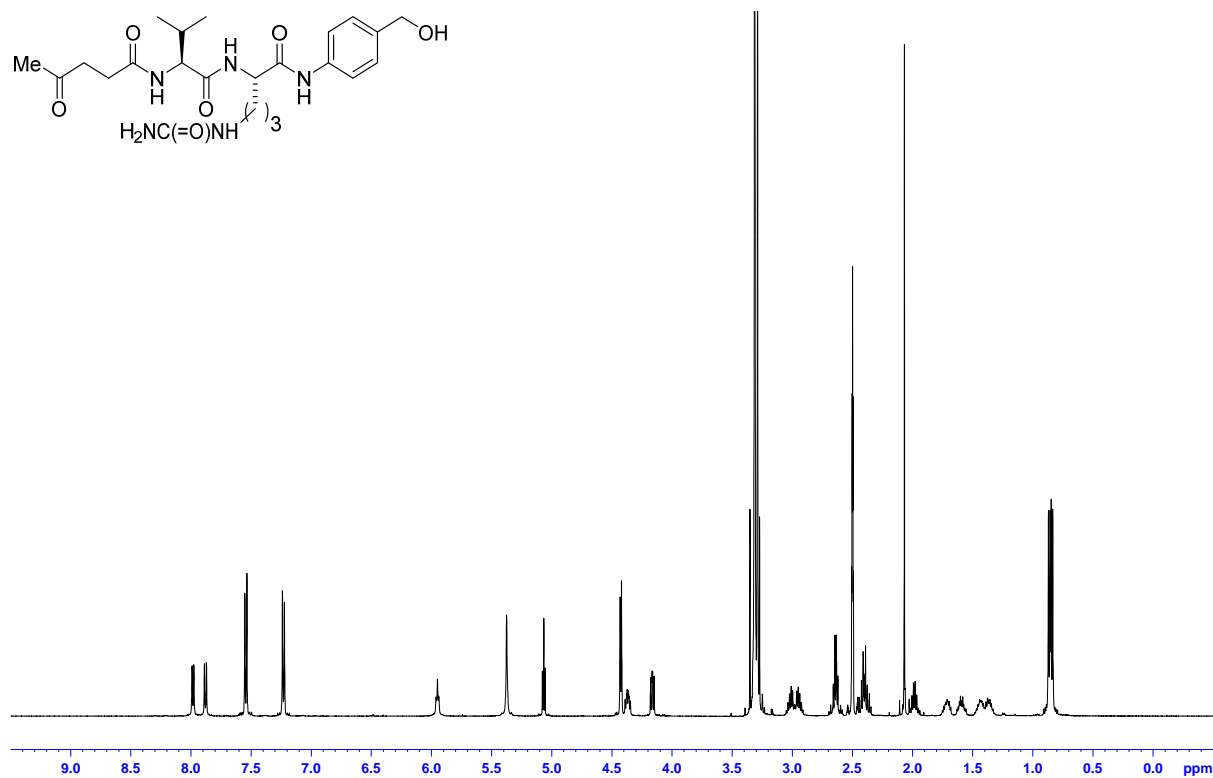
18 - ^1H NMR



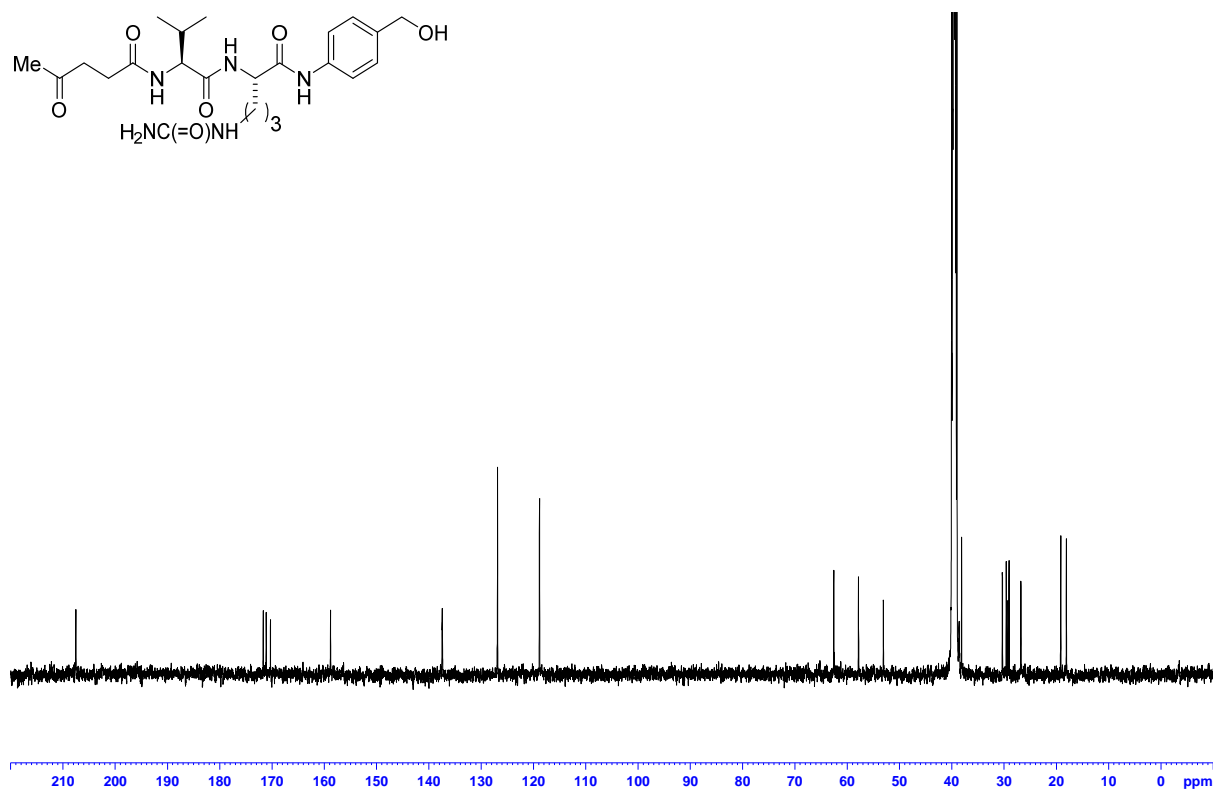
18 - ^{13}C NMR proton decoupled



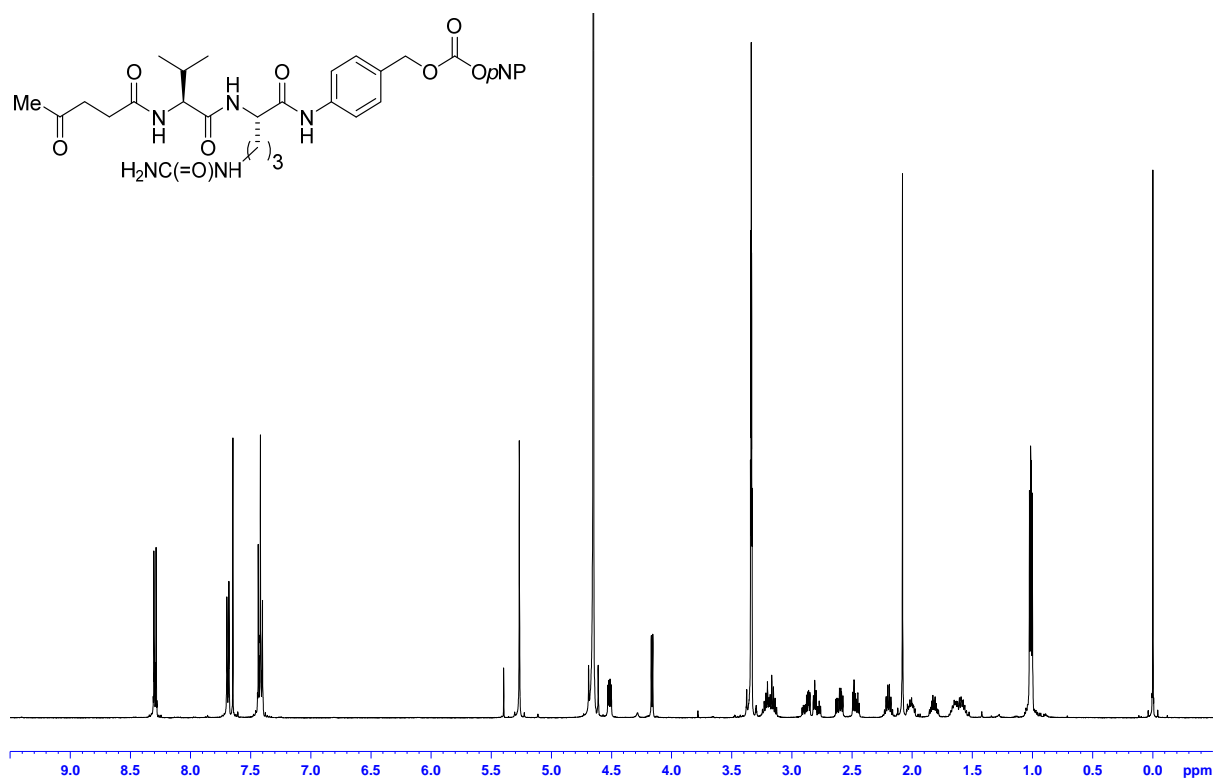
13 - ¹H NMR



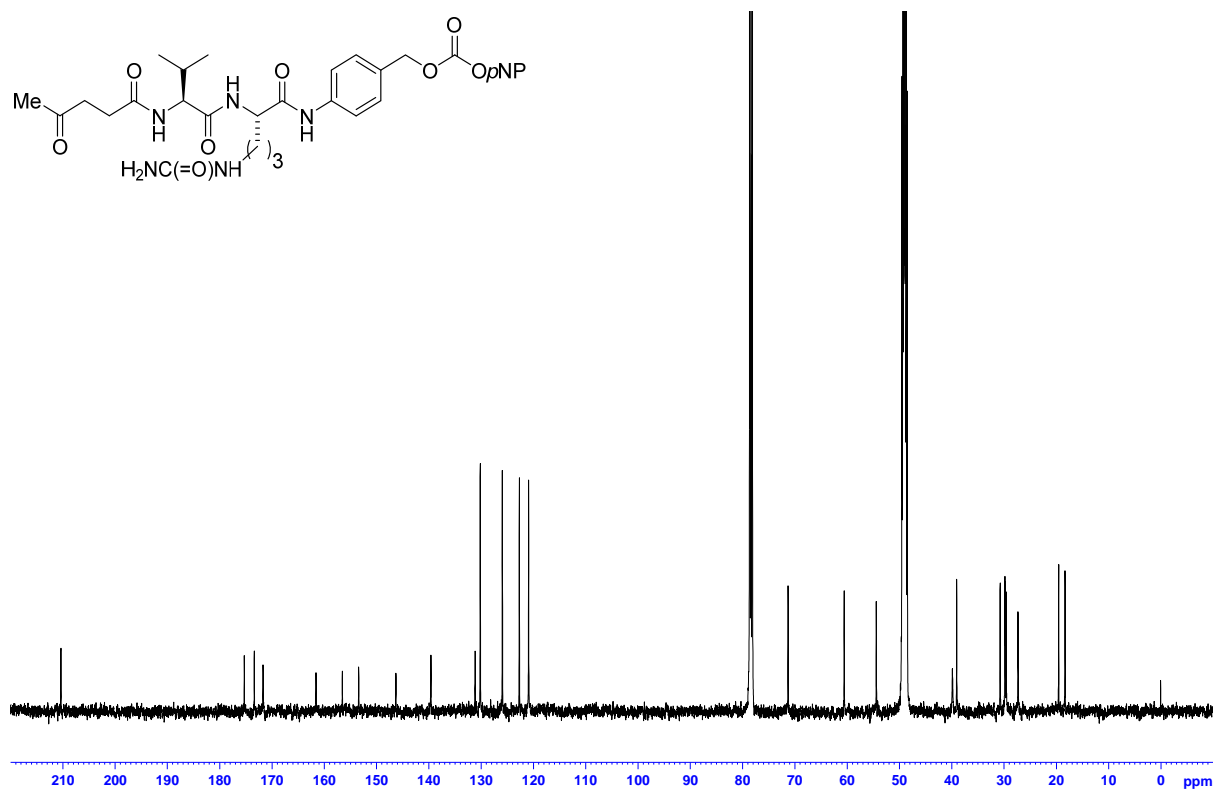
13 - ¹³C NMR proton decoupled



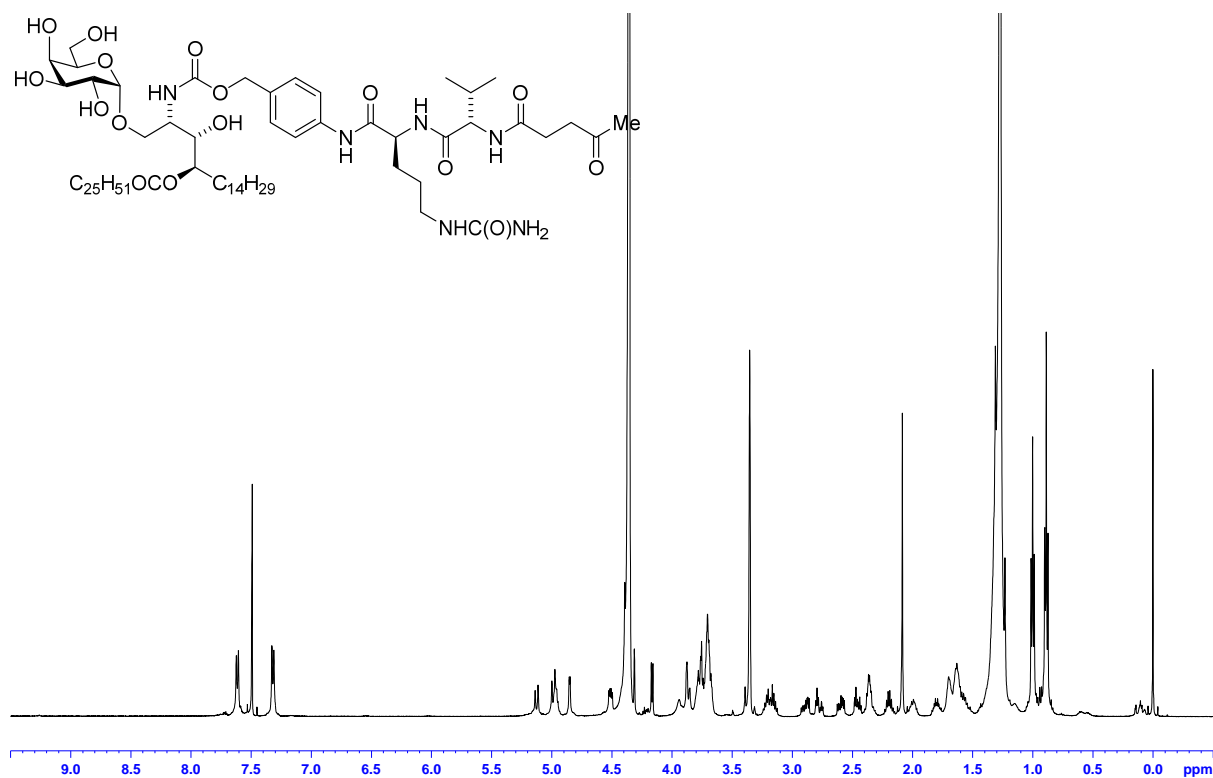
15 - ^1H NMR



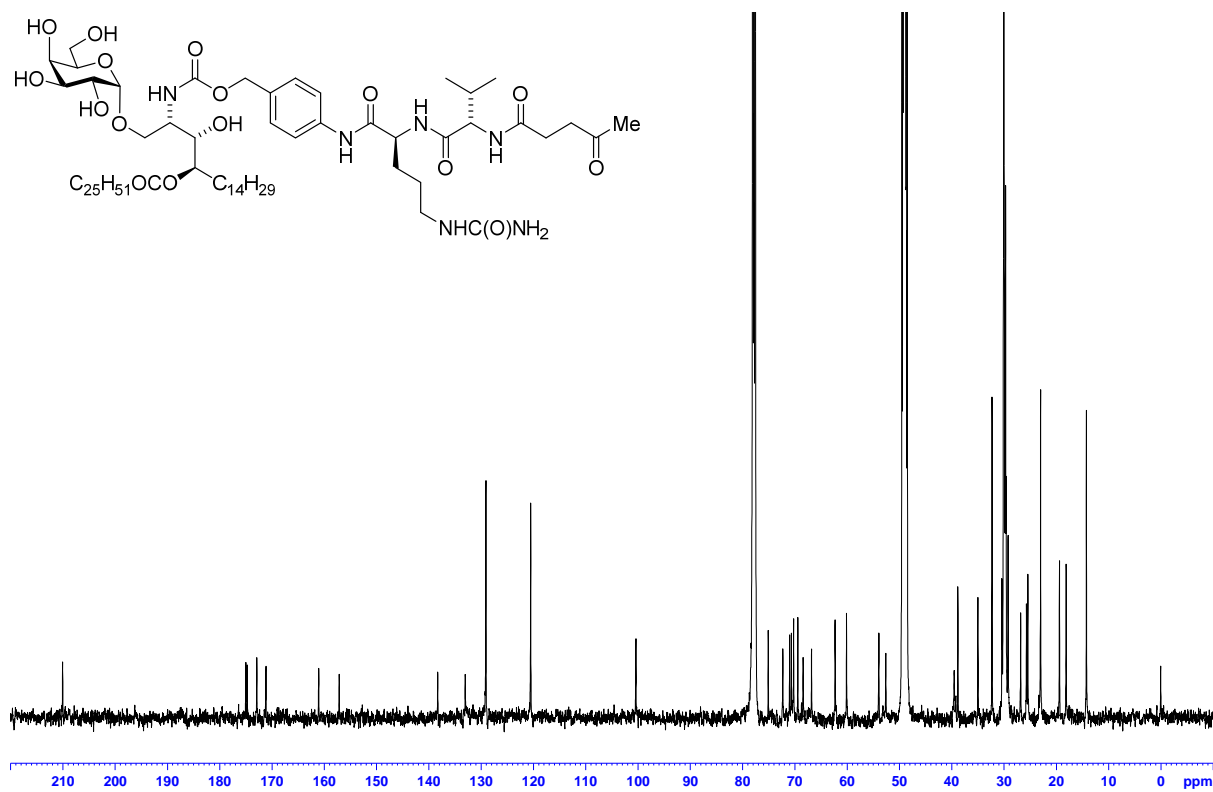
15 - ^{13}C NMR proton decoupled



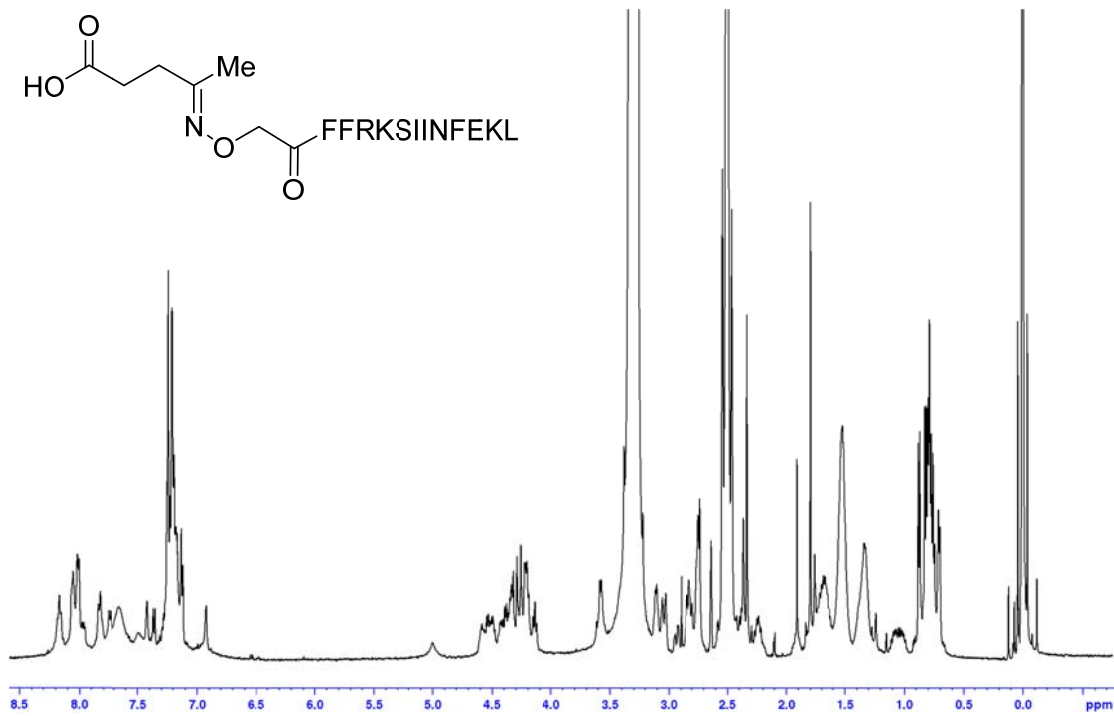
17 - ^1H NMR



17 - ^{13}C NMR proton decoupled



21 - ¹H NMR



21 – HPLC/LCMS

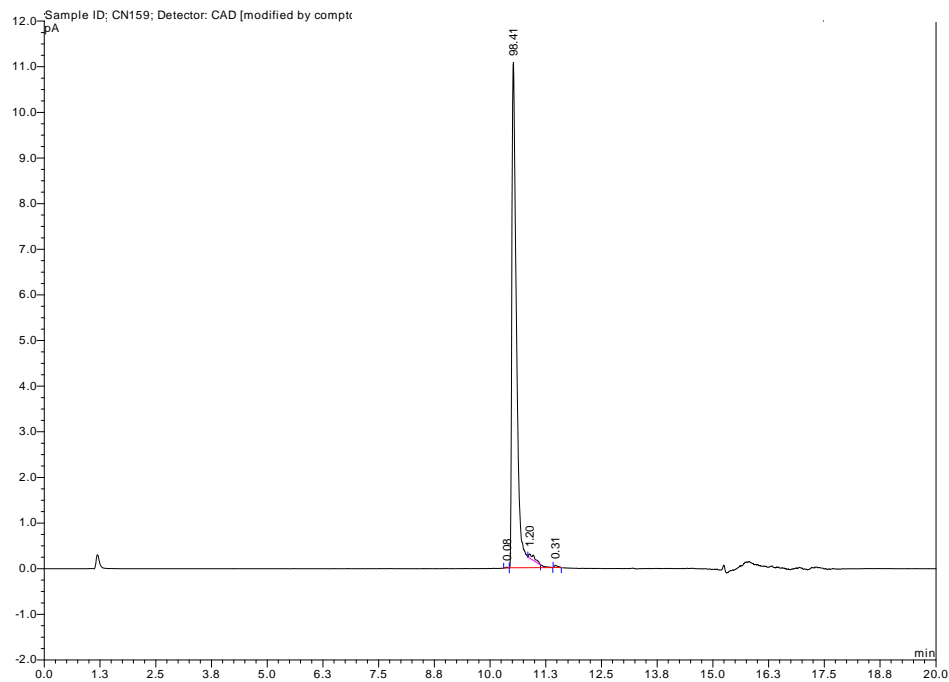
HPLC (sample purity)

Column: Phenomenex Kinetex C18, 2.6 μm, 3 x 100 mm.

Mobile Phase A: Water + 0.05% Trifluoroacetic acid

Mobile Phase B: Methanol + 0.05% Trifluoroacetic acid

Gradient (A:B): T0 = 20:80, T12 = 0:100, T14 = 0:100, T16 = 20:80; 0.4 mL/min, T = 40 °C



21 – HPLC/LCMS

HPLC-MS-CAD (peak ID)

Column: Agilent Poroshell 120 EC-C18 2.7 μm , 3 x 50 mm.

Mobile Phase A: Water + 0.05% Formic acid

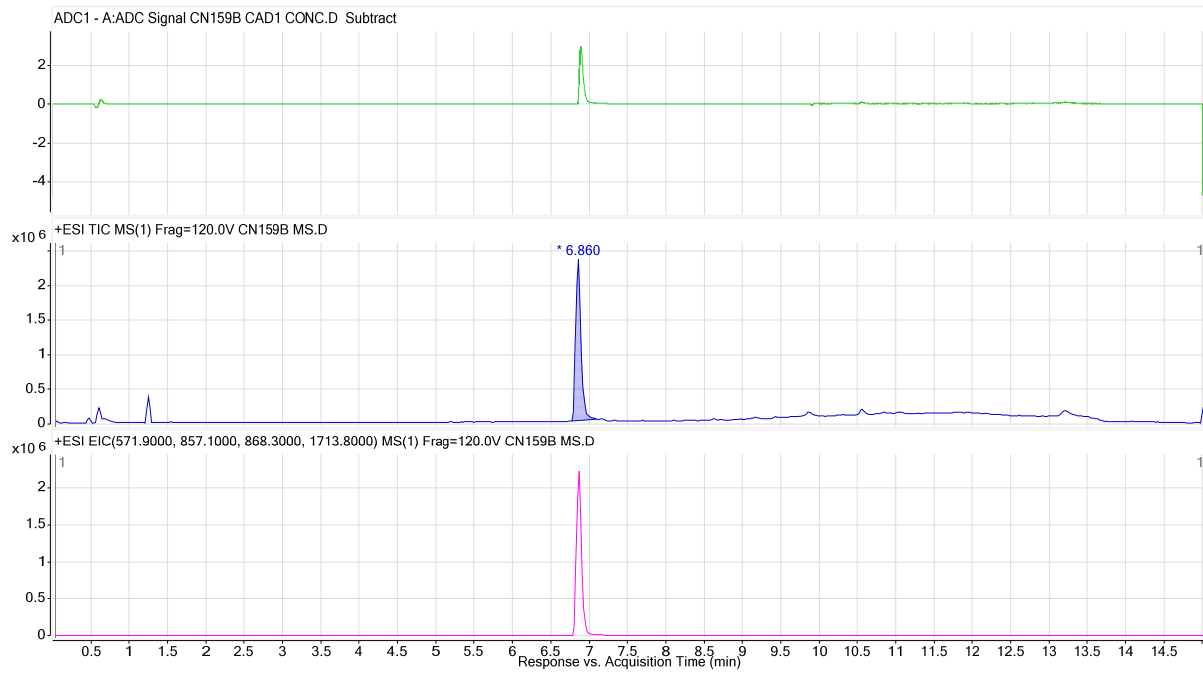
Mobile Phase B: Methanol

Gradient (A:B): T0 = 5:95, T9 = 0:100, T12 = 0:100, T13 = 5:95; 0.5 mL/min, T = 40 $^{\circ}\text{C}$

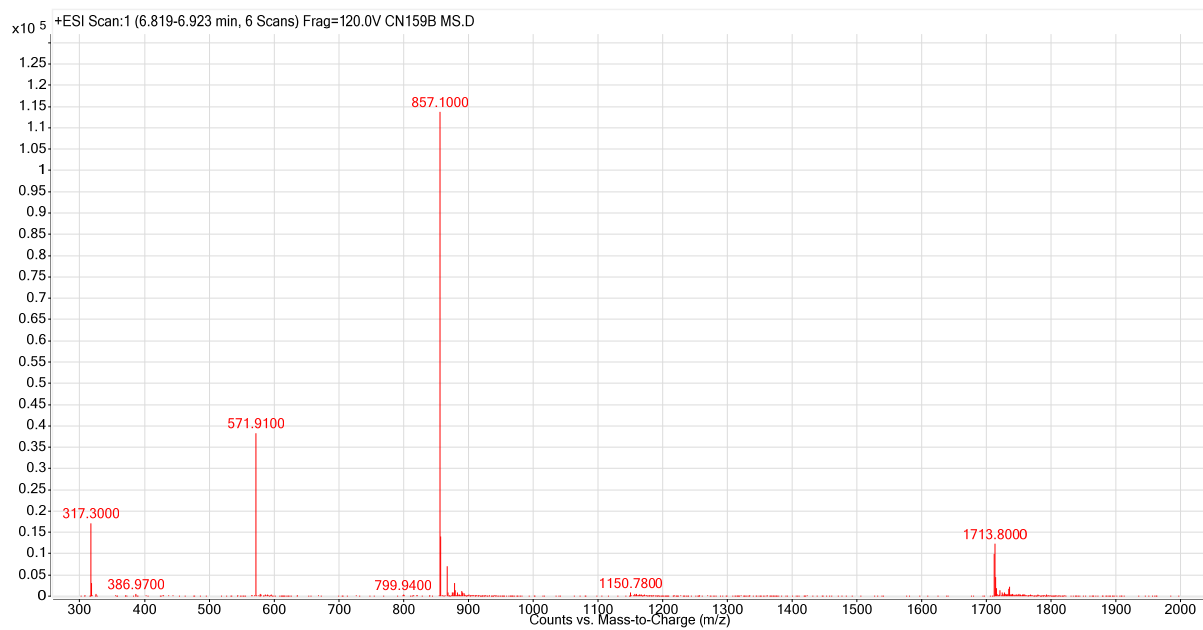
CAD (top)

TIC (middle)

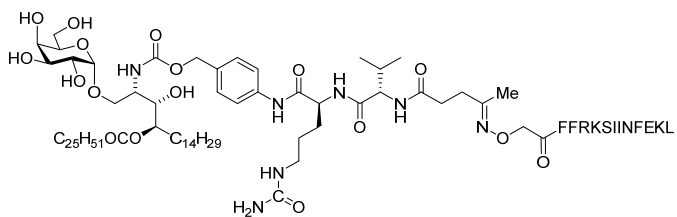
EIC (bottom)



Extracted MS from shaded peak in the TIC



3 – HPLC/LCMS



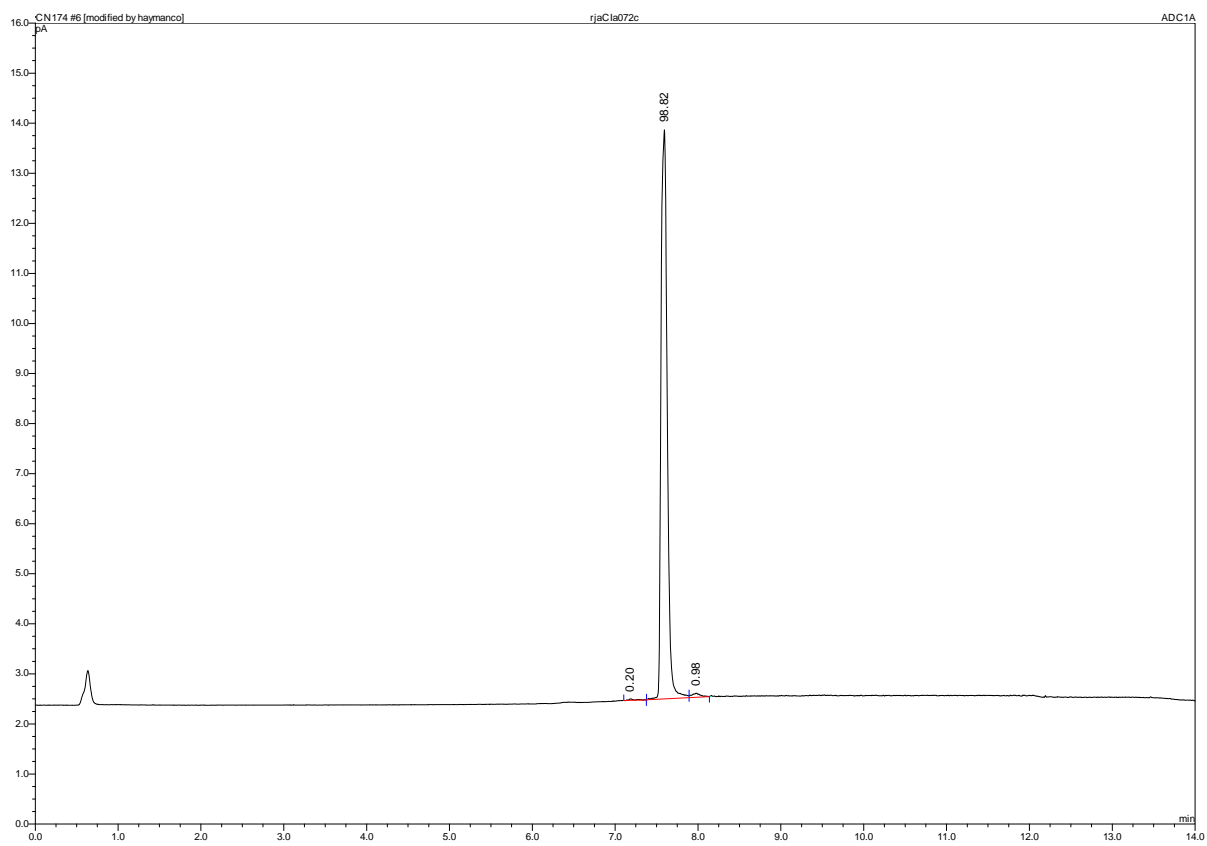
HPLC-CAD (sample purity)

Column: Agilent Poroshell 120 EC-C18 2.7 μ m, 3 x 50 mm.

Mobile Phase A: Water + 0.01% Trifluoroacetic acid

Mobile Phase B: Methanol + 0.01% Trifluoroacetic acid

Gradient (A:B): T0 = 30:70, T7 = 0:100, T12 = 0:100, T13 = 30:70; 0.5 mL/min, T = 40 °C



3 – HPLC/LCMS

HPLC-MS-CAD (peak ID)

Column: Agilent Poroshell 120 EC-C18 2.7 μm , 3 x 50 mm.

Mobile Phase A: Water + 0.01% Trifluoroacetic acid

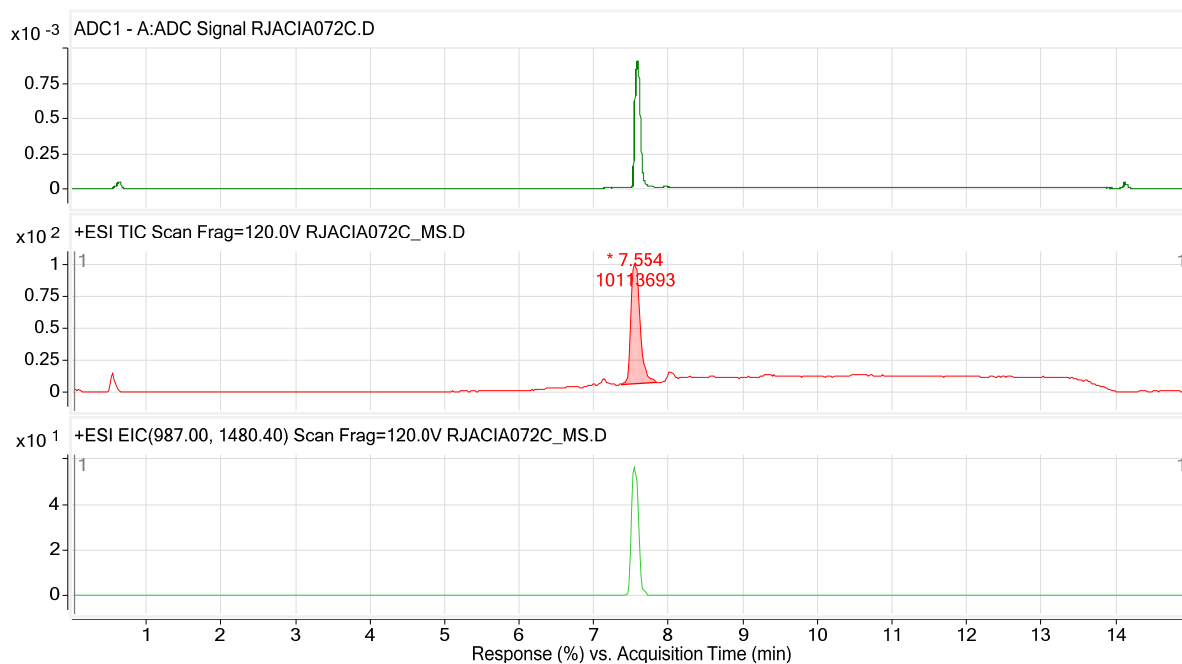
Mobile Phase B: Methanol + 0.01% Trifluoroacetic acid

Gradient (A:B): T0 = 30:70, T7 = 0:100, T12 = 0:100, T13 = 30:70; 0.5 mL/min, T = 40 °C

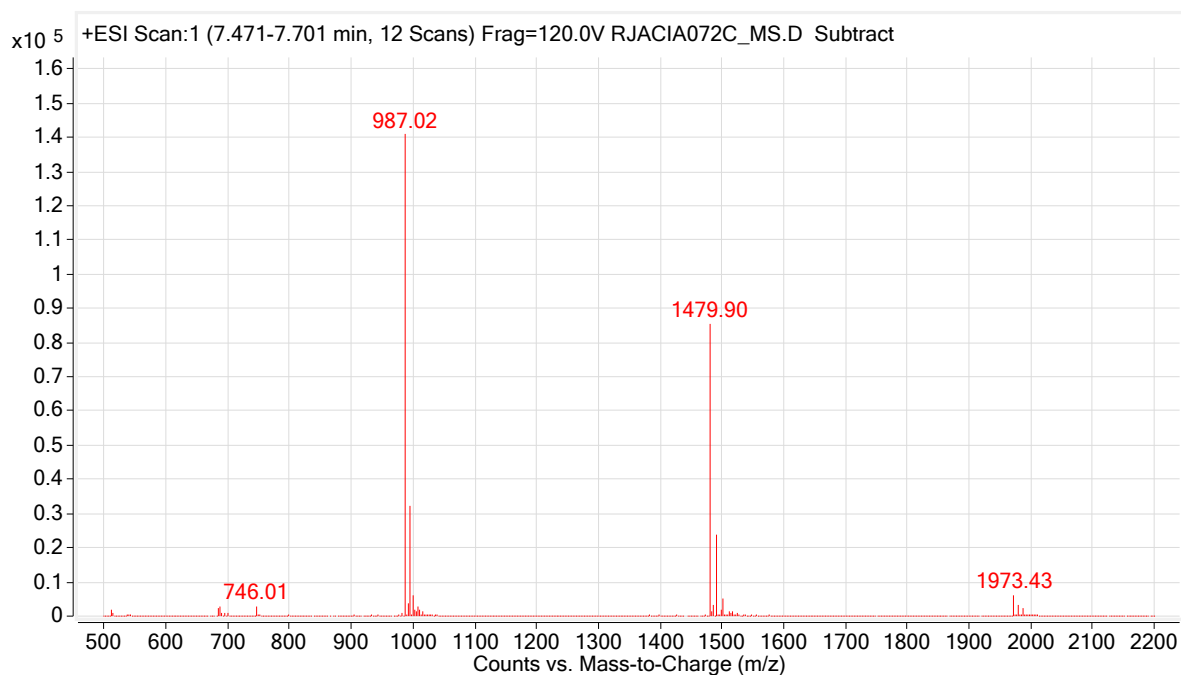
CAD (top)

TIC (middle)

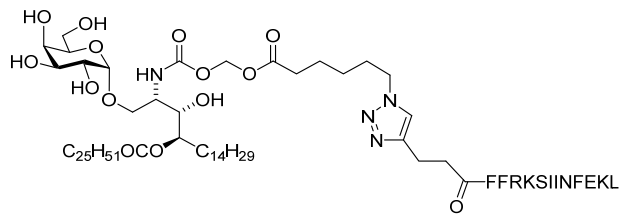
EIC (bottom)



Extracted MS from shaded peak in the TIC



4 – HPLC/LCMS



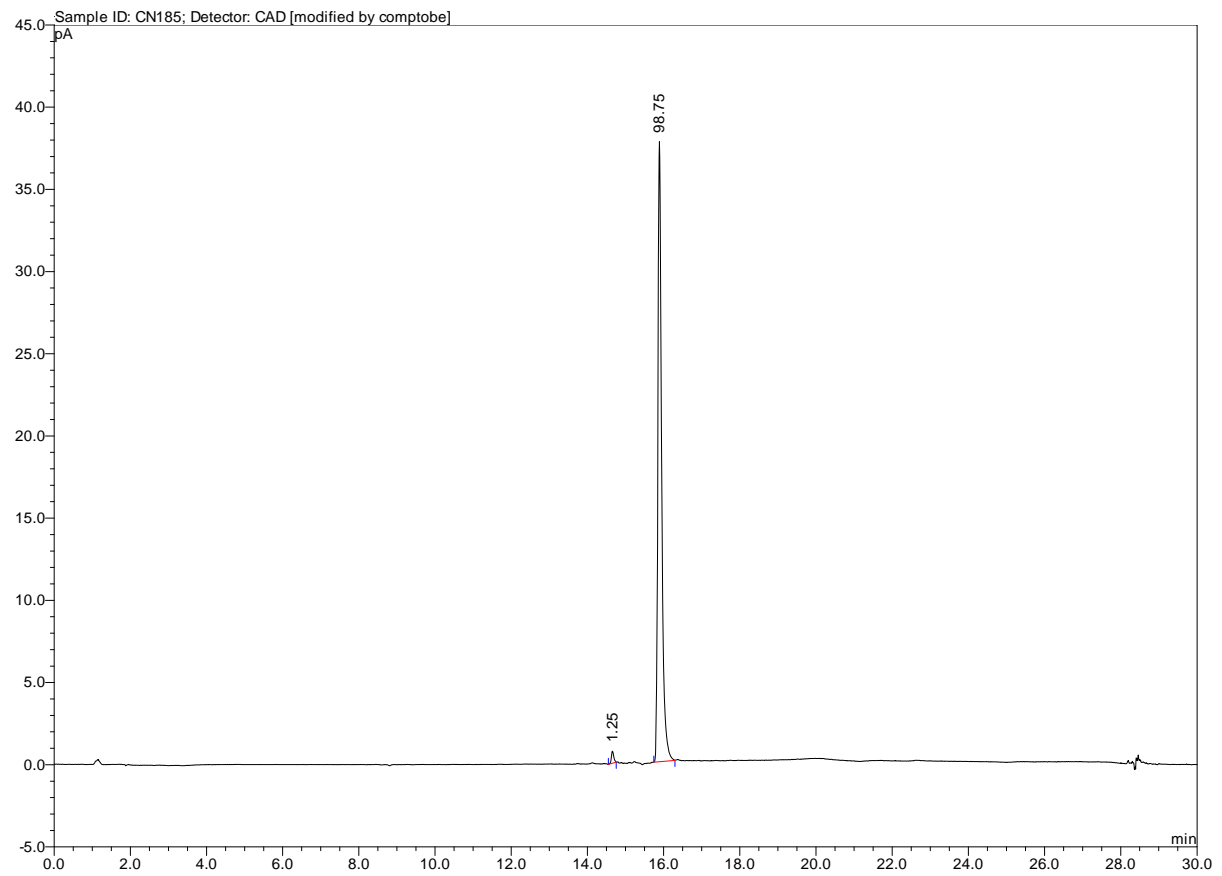
HPLC (sample purity)

Column: Phenomenex Kinetex C18, 2.6 μ m, 3 x 100 mm.

Mobile Phase A: Water + 0.05% Trifluoroacetic acid

Mobile Phase B: Methanol + 0.05% Trifluoroacetic acid

Gradient (A:B): T0 = 40:60, T12 = 0:100, T24 = 0:100, T25 = 40:60; 0.4 mL/min, T = 40 °C



4 – HPLC/LCMS

HPLC-MS-CAD (peak ID)

Column: Agilent Zorbax SB-C18 1.8 μm , 2.1 x 50 mm.

Mobile Phase A: Water + 0.05% Formic acid

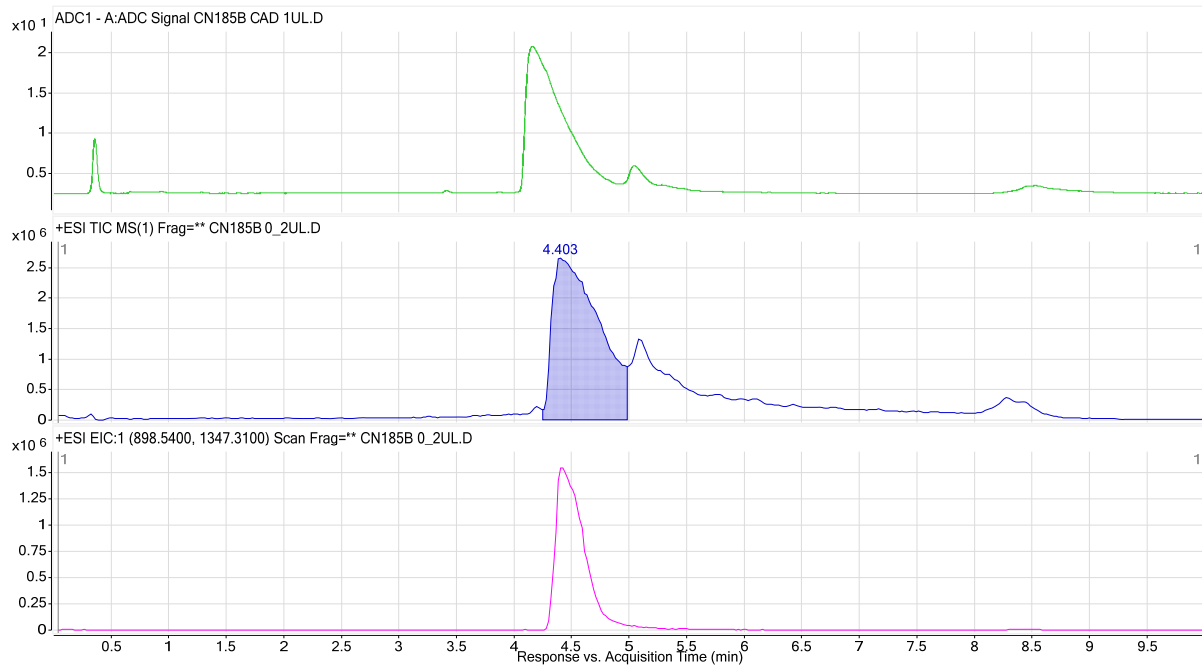
Mobile Phase B: Methanol

Gradient (A:B): T0 = 20:80, T5 = 0:100, T7 = 0:100, T10 = 20:80; 0.5 mL/min, T = 40 $^{\circ}\text{C}$

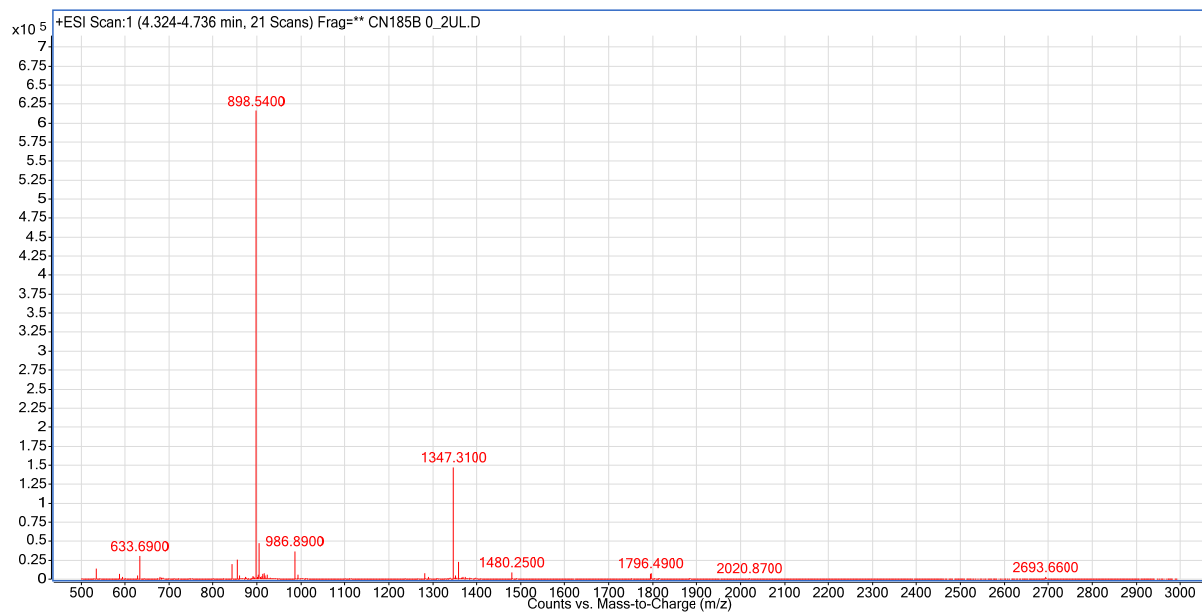
CAD (top)

TIC (middle)

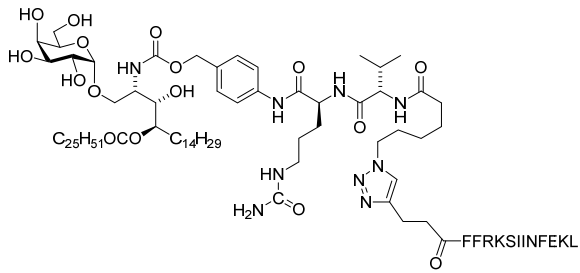
EIC (bottom)



Extracted MS from shaded peak in the TIC



5 – HPLC/LCMS



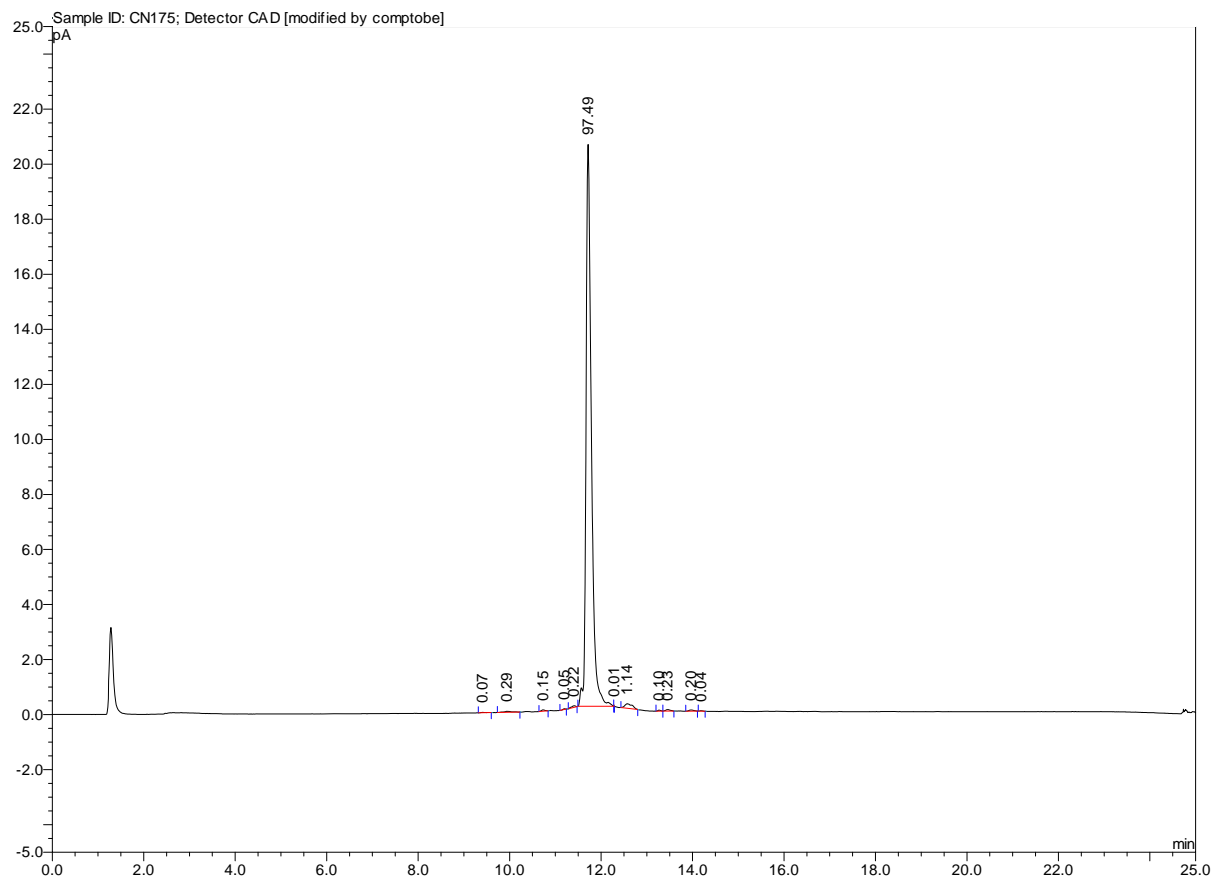
HPLC (sample purity)

Column: Phenomenex Kinetex C18, 2.6 μ m, 3 x 100 mm.

Mobile Phase A: Water + 0.05% Trifluoroacetic acid

Mobile Phase B: Methanol + 0.05% Trifluoroacetic acid

Gradient (A:B): T₀ = 40:60, T₈ = 0:100, T₂₀ = 0:100, T₂₁ = 40:60; 0.4 mL/min, T = 40 °C



5 – HPLC/LCMS

HPLC-MS-CAD (peak ID)

Column: Agilent Zorbax SB-C18 1.8 μm , 2.1 x 50 mm.

Mobile Phase A: Water + 0.05% Trifluoroacetic acid

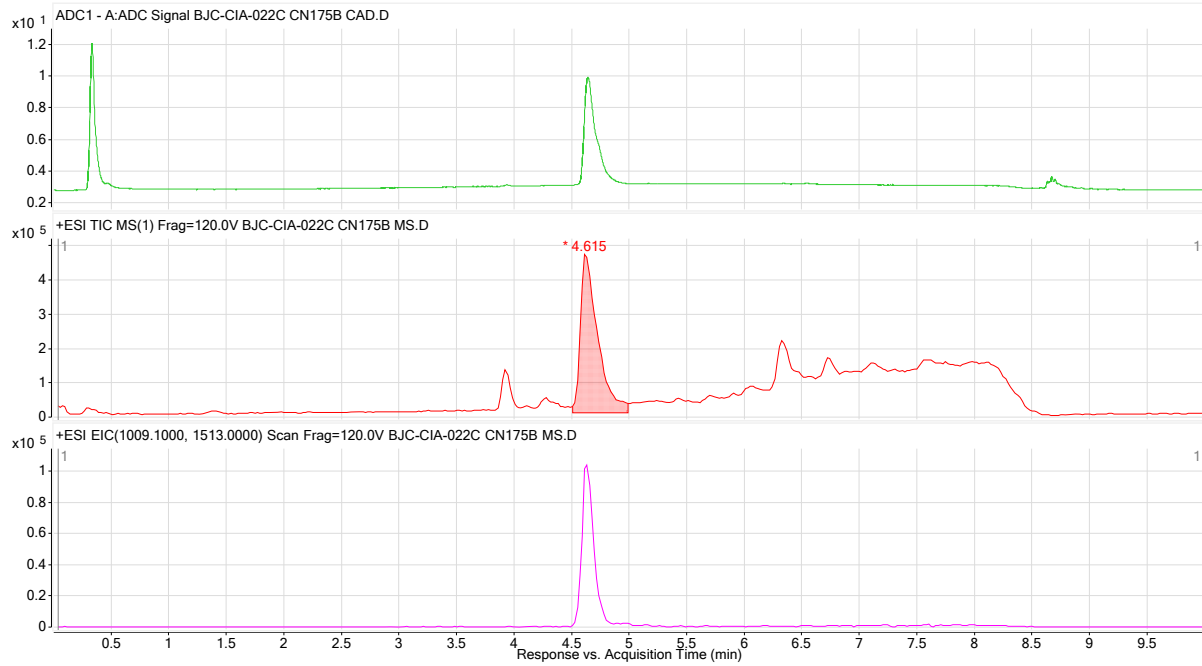
Mobile Phase B: Methanol

Gradient (A:B): T0 = 20:80, T5 = 0:100, T7 = 0:100, T10 = 20:80; 0.5 mL/min, T = 40 $^{\circ}\text{C}$

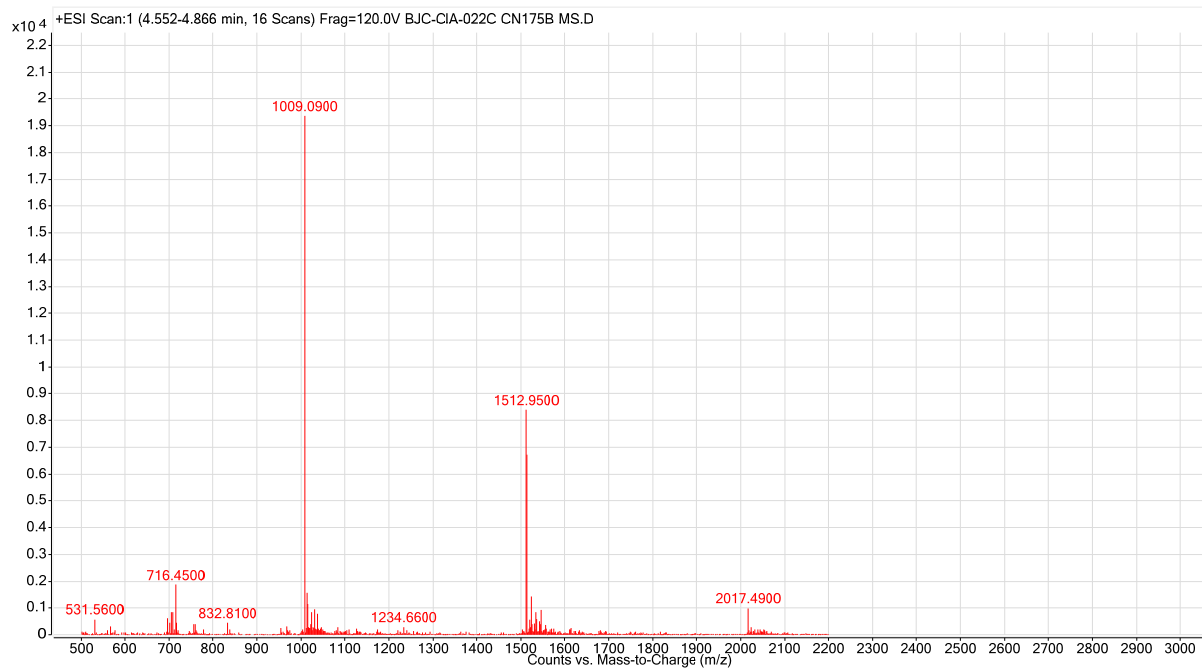
CAD (top)

TIC (middle)

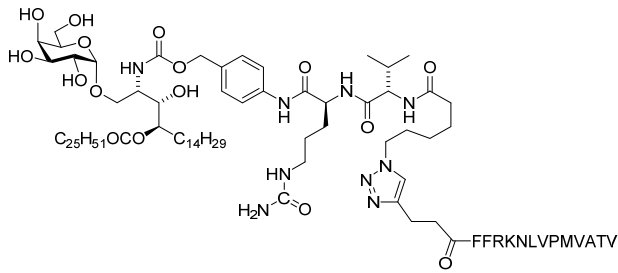
EIC (bottom)



Extracted MS from shaded peak in the TIC



6 – HPLC/LCMS



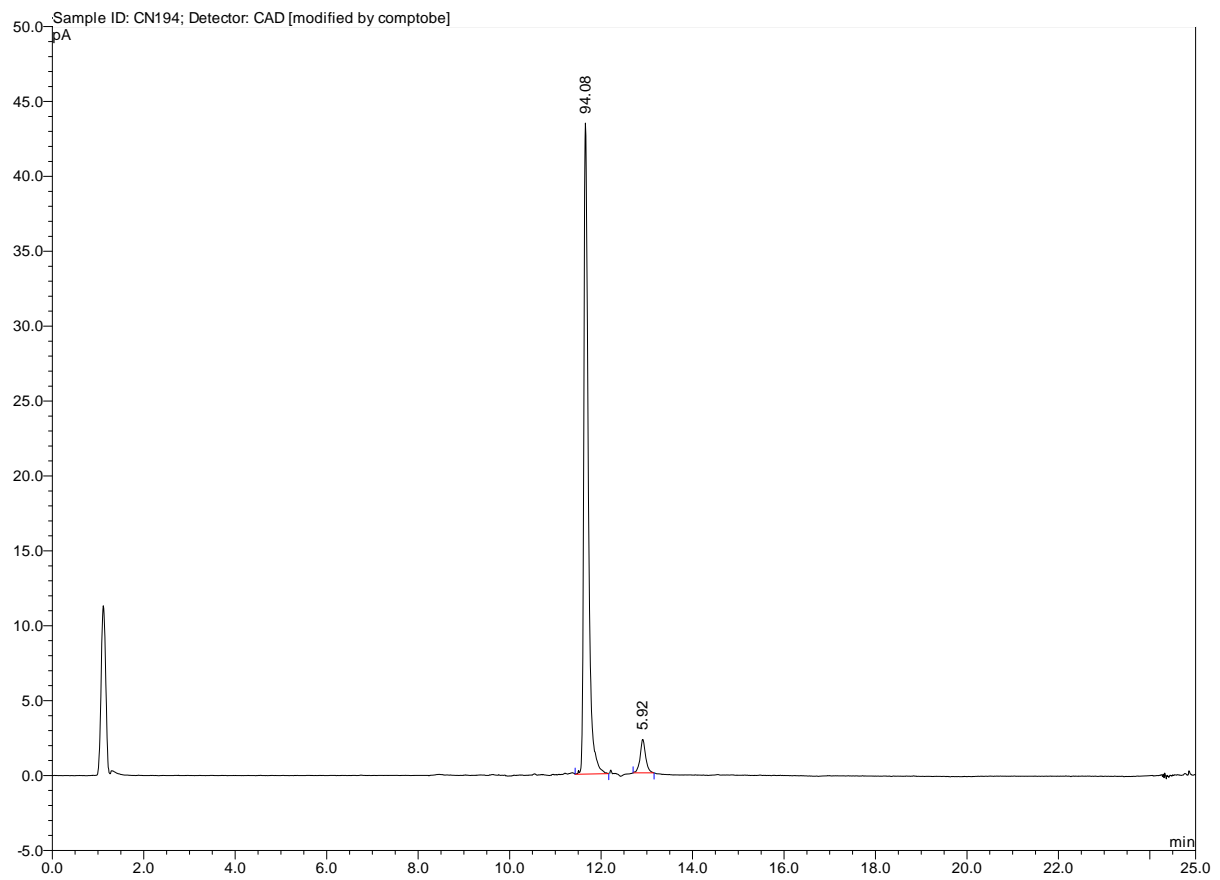
HPLC (sample purity)

Column: Phenomenex Kinetex C18, 2.6 μ m, 3 x 100 mm.

Mobile Phase A: Water + 0.05% Trifluoroacetic acid

Mobile Phase B: Methanol + 0.05% Trifluoroacetic acid

Gradient (A:B): T₀ = 40:60, T₈ = 0:100, T₂₀ = 0:100, T₂₁ = 40:60; 0.4 mL/min, T = 40 °C



6 – HPLC/LCMS

HPLC-MS-CAD (peak ID)

Column: Agilent Zorbax SB-C18 1.8 μm , 2.1 x 50 mm.

Mobile Phase A: Water + 0.05% Formic acid

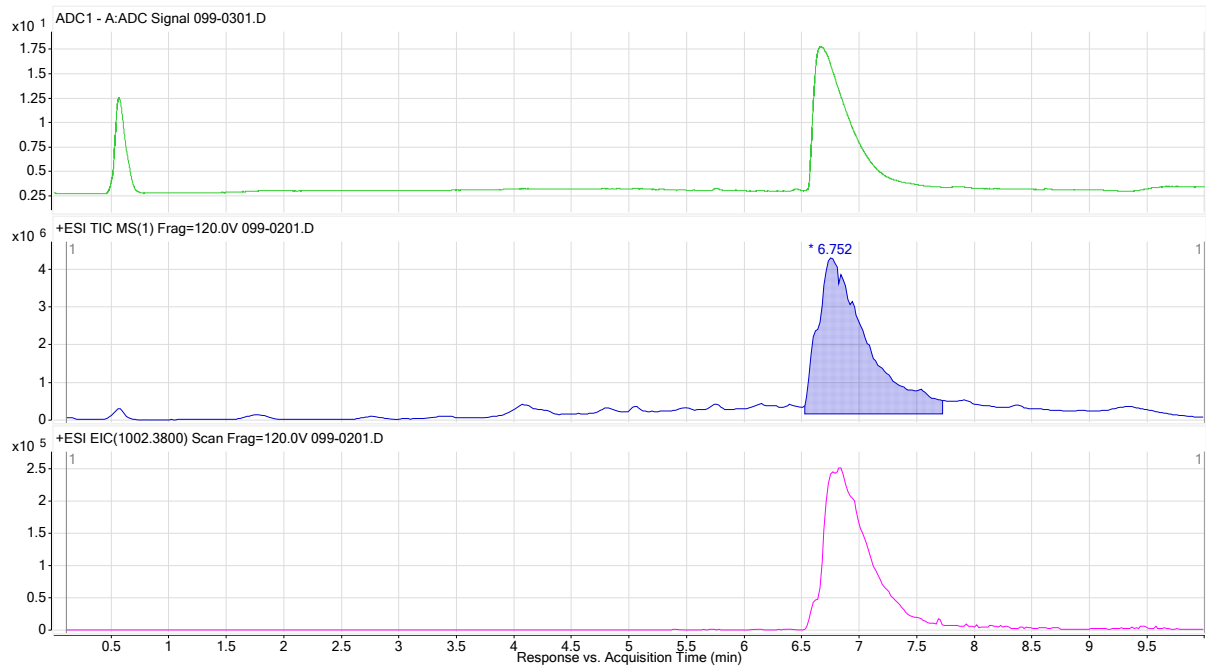
Mobile Phase B: Methanol

Gradient (A:B): T0 = 20:80, T5 = 0:100, T7 = 0:100, T10 = 20:80; 0.5 mL/min, T = 40 $^{\circ}\text{C}$

CAD (top)

TIC (middle)

EIC (bottom)



Extracted MS from shaded peak in the TIC

