9 - $^1$H NMR

9 - $^{13}$C NMR proton decoupled
11 - $^1$H NMR

11 - $^{13}$C NMR proton decoupled
14 - $^1$H NMR

14 - $^{13}$C NMR proton decoupled
16: $^1$H NMR

16: $^{13}$C NMR proton decoupled
18 - \(^1\)H NMR

18 - \(^{13}\)C NMR proton decoupled
13 - $^1$H NMR

13 - $^{13}$C NMR proton decoupled
17 - $^1$H NMR

17 - $^{13}$C NMR proton decoupled
21 - $^1$H NMR

![Chemical structure](image1)

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

![LC/MS chromatogram](image2)
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 °C

CAD (top)
TIC (middle)
EIC (bottom)
3 – HPLC/LCMS

HPLC-CAD (sample purity)
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
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)
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 = 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 °C

CAD (top)
TIC (middle)
EIC (bottom)
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 = 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 °C

CAD (top)
TIC (middle)
EIC (bottom)

Extracted MS from shaded peak in the TIC
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 = 40:60, T8 = 0:100, T20 = 0:100, T21 = 40:60; 0.4 mL/min, T = 40 °C

Sample ID: CN194; Detector: CAD [modified by comptobe]
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 °C

CAD (top)
TIC (middle)
EIC (bottom)

Extracted MS from shaded peak in the TIC