Supplementary Information

Asymmetric [3+2] Cycloaddition of Morita-Baylis-Hillman Carbonates with Maleimides Catalyzed by Chiral Ferrocenylphosphines

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1. General procedure for synthesis of chiral ferrocenylphosphines LB1-LB9

(*S*)-1-(2-methyl-phenyl)-3-[(*R*)-2-(diphenylphosphino) ferrocenyl] ethylthiourea (LB1)

A suspension of (*S*)-1-(*R*) diphenylphosphino ferrocenyl ethylamine (0.75 g, 2 mmol) in CH₂Cl₂ (10 mL) was cooled to 0 °C. Then 2-Methylphenyl isothiocyanate (0.41 g, 3 mmol) was added in 10 min and The reaction mixture was stirred at room temperature for 12 h. After the reaction was completed, the solvent was removed under reduced pressure and the residue was purified by a flash column chromatography to afford **LB1** (0.94g, 94%) as a yellow solid. m.p.: 190.6-192.0 °C; $[\alpha]_D^{20} = +265.8^\circ$ (c 0.5, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 8.01-7.96 (m, 1H), 7.52-7.17 (m, 11H), 7.09-7.05 (m, 4H), 5.42-5.38 (m, 1H), 4.42 (s, 1H), 4.23 (t, *J* = 2.4 Hz, 1H), 3.82 (s, 5H), 3.65 (s, 1H), 2.36 (s, 3H), 1.21 (d, *J* = 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 178.2, 138.3, 136.4, 135.8, 134.8, 134.6, 134.4, 132.1, 132.0, 131.9, 129.2, 128.5, 128.3, 128.03 (2C), 127.97, 127.8, 127.7, 127.6, 95.9, 95.6, 72.7, 71.9, 69.6 (5C), 69.4, 52.2, 24.7, 18.2; ³¹P NMR (162 MHz, CDCl₃, 85 % H₃PO₄) δ -24.54; MS (ESI) *m/z* (%): 585.0 (100) [M⁺ + Na]; HRMS (ESI) calcd. for C₃₂H₃₂FeN₂PS 563.1373 [M+H]; found 563.1383 .

(*S*)-1-[(*R*)-2-diphenylphosphino]ferrocenylethyl acetamide (LB 2)

A suspension of (*S*)-1-(*R*) diphenylphosphinoferrocenyl ethylamine (0.20g, 0.50 mmol) in CH₂Cl₂ (10 mL) was cooled to 0 °C. Then acetyl chloride (0.06 g, 0.73 mmol) was added in 10 min and The reaction mixture was stirred at room temperature

for 12 h. After the reaction was completed, the solvent was removed under reduced pressure and the residue was purified by a flash column chromatography to afford **LB2** (0.20g, 91%) as a yellow solid; m.p.: 165.8-166.9 °C; $[\alpha]_D^{20} = +299.1^\circ$ (c 1.0, CHCl₃), {Lit.^[1] 157-158 °C; $[\alpha]_D^{23} = +312.0^\circ$ (c 1.0, methanol)}; ¹H NMR (400 MHz, CDCl₃): δ 7.51-7.47 (m, 2H), 7.36-7.35 (m, 3H), 7.22-7.20 (m, 5H), 5.65 (s, 1H), 5.18 (s, 1H), 4.46 (s, 1H), 4.29 (s, 1H), 4.03 (s, 5H), 3.77 (s, 1H), 1.40 (d, *J* = 4.0 Hz, 3H), 1.36 (s, 3H); ³¹P NMR (162 MHz, CDCl₃, 85 % H₃PO₄): δ -24.89; MS (ESI) *m/z* (%): 494.2 (100) [*M*⁺+K].

2-(diphenylphosphino)-*N*-[(*R*)-1'-[(*R*)-2'-diphenylphosphino]ferrocenyl]ethyl benzamide (LB 3)

A suspension of *N,N*-dimethyl-(*R*)-1-ferrocenylethylamine (2.57g, 0.01 mol) in acetic anhydride (125 mL), the reaction mixture was stirred at room temperature for 24h. After the reaction was completed, the redundant acetic anhydride was removed under reduced pressure. A mixture of the concentrated solution in 100 mL of ammonia-methanol was heated at 90°C for 7h in a microautoclave. The solvent was removed under reduced pressure, yielding 3.19g black residue.

A suspension of the black residue in CH₂Cl₂ (20 mL) was cooled to 0°C. Then a mixture of *N,N'*-methanediylidenedicyclohexanamine (4.53g, 22 mmol) and *N,N*-dimethyl pyridin-4-amine (0.12g, 1 mmol) in CH₂Cl₂ was added in 30 min and the reaction mixture was stirred at room temperature for overnight. Water (60 mL) was added to the reactant, followed by diluted with dichloromethane (2 × 30 mL) and

dried over anhydrous sodium sulfate. After evaporation of the solvents, the residue was chromatographed on silica gel to afford **LB3** (0.79 g, 15.3%) as a yellow solid; m.p.: 96.4-98.1 °C; $[\alpha]_{\text{D}}^{20} = -36.2^\circ$ (c 1.0, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.59 (ddd, *J* = 7.6, 4.0, 1.2 Hz, 1H), 7.40 – 7.19 (m, 12H), 6.92 (ddd, *J* = 7.6, 4.0, 1.2 Hz, 1H), 6.15 (d, *J* = 8.0 Hz, 1H), 4.98 – 4.91(m, 1H), 4.10 – 4.05 (m, 9H), 1.32 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 167.4, 141.9, 141.6, 137.1, 137.0, 136.8, 135.6, 135.4, 134.0, 133.8, 133.6, 129.9, 128.73, 128.67, 128.6, 128.51, 128.46, 128.4, 127.7, 91.2, 68.5(5C), 68.0, 67.6, 67.3, 66.0, 44.4, 20.7; ³¹P NMR (162 MHz, CDCl₃, 85 % H₃PO₄) δ -9.48; MS (ESI) *m/z* (%) : 540.1 (100) [M⁺ +Na]; HRMS (ESI) Calcd. for C₃₁H₂₈FeNNaOP 540.1156 [M+Na], found: 540.1144.

(*S*)-*N,N*-Dimethyl-1-[(*R*)-2-(diphenylphosphino)ferrocenyl]ethylamine (LB4)

The procedure was accorded to Lit.^[2] and **LB4** (7.97 g, 60%) was obtained as a yellow solid; m.p.: 138.0-138.9 °C; $[\alpha]_{\text{D}}^{20} = +325.6^\circ$ (c 1.0, CHCl₃), {Lit.^[3] 140-142 °C; Lit.^[4] $[\alpha]_{\text{D}}^{20} = +352.0^\circ$ (c 0.6, CHCl₃)}; ¹H NMR (400 MHz, CDCl₃): δ 7.59-7.55 (m, 2H), 7.34-7.32 (m, 3H), 7.18-7.15 (m, 5H), 4.36 (s, 1H), 4.24 (s, 1H), 4.18-4.14 (m, 1H), 3.92 (s, 5H), 3.85 (s, 1H), 1.77 (s, 6H), 1.27-1.22 (m, 3H); MS (ESI) *m/z* (%): 441.9 (100) [M⁺+1].

(*R*)-1-[(*S*)-1,2-Bis(diphenylphosphino)ferrocenyl]ethyl-3-phenylthiourea (LB5)

Following a similar procedure as **LB1**, **LB5** (0.35g, 95%) was obtained as a yellow solid; m.p.: 173.5-1734.7 °C; $[\alpha]_{\text{D}}^{20} = -287.5^\circ$ (c 0.50, CHCl₃); ¹H NMR (400

MHz, CDCl₃): δ 7.87 (s, 1H), 7.61 (s, 1H), 7.52 (t, J =8.0 Hz, 2H), 7.38 (t, J =7.6 Hz, 1H), 7.29-7.25 (m, 3H), 7.23-7.12 (m, 13H), 7.03-6.97 (m, 4H), 5.47 (t, J =7.2 Hz, 1H), 4.39 (d, J =5.6 Hz, 2H), 4.26 (s, 1H), 4.15 (t, J =2.4 Hz, 1H), 3.75 (s, 1H), 3.41 (s, 1H), 3.17 (s, 1H), 1.25 (d, J =6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 178.0, 139.0, 138.3, 137.7, 135.9, 135.5, 134.5(2C), 133.6(2C), 132.5(2C), 132.0(2C), 130.1(4C), 129.1, 128.5, 127.9(6C), 127.2, 126.1(4C), 96.3, 75.4, 75.2, 73.6(2C), 73.0, 72.9(2C), 72.60, 72.0, 51.7, 23.9; ³¹P NMR (162 MHz, CDCl₃, 85 % H₃PO₄): δ -21.23, -29.76; MS (ESI) m/z (%): 755.2 (100) [M⁺+Na]; HRMS (ESI) calcd. for C₄₃H₃₈FeN₂NaP₂S 755.1478 [M+Na]; found 755.1497 .

(*R*)-1-(4-fluoro-phenyl)-3-[(*S*)-1,2-Bis(diphenylphosphino) ferrocenyl] ethylthiourea (LB 6)

Following a similar procedure as **LB1**, **LB6** (0.31g, 87%) was obtained as a yellow solid; m.p.: 139.0-141.0 °C; [α]_D²⁰ = -283.7° (c 0.50, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 7.66 (s, 1H), 7.58 (s, 1H), 7.31-7.27 (m, 5H), 7.25-7.15 (m, 15H), 7.09-7.04 (m, 4H), 5.51-5.44 (m, 1H), 4.41 (d, J =8.0 Hz, 2H), 4.28 (s, 1H), 4.18 (t, J =2.8 Hz, 1H), 3.81 (s, 1H), 3.47 (s, 1H), 3.25 (s, 1H), 1.25 (d, J =6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 178.4, 162.6, 160.1, 138.9, 138.3, 137.7, 135.4, 134.5, 134.3, 133.6, 133.4, 132.7, 132.6, 132.0(2C), 131.9, 129.3(2C), 128.52(2C), 128.46, 128.1(2C), 128.0(5C), 127.9(2C), 117.0(2C), 96.1, 75.4, 73.6, 73.0, 72.8, 72.7, 72.0, 58.3, 51.6, 23.7, 18.5; ³¹P NMR (162 MHz, CDCl₃, 85 % H₃PO₄): δ -17.18, -25.26;

MS (ESI) m/z (%): 773.1 (100) $[M^+ + Na]$; HRMS (ESI) Calcd. for $C_{43}H_{37}FFeN_2NaP_2S$ 773.1384 $[M + Na]$; found 773.1396.

***N*-[(*S*)-1'-(*R*)-2'-diphenylphosphinoferrocenyl]ethyl-(*R*)-methyl pyrrolidine-2-carboxylate (LB7)**

A solution of (*S*)-1-(*R*) diphenylphosphinoferrocenyl ethyl acetate (0.23g, 0.5 mmol) and (*S*)-methyl pyrrolidine-2-carboxylate (0.26g, 2.0 mmol) in methanol (5 mL) was refluxed for 5h. The solvent was removed under induced pressure and the residue was purified by column chromatography to afford **LB7** (0.69g, 66%) as a yellow solid; m.p.: 105.2–106.4 °C; $[\alpha]_D^{20} = +277.0^\circ$ (c 1.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.60–7.55 (m, 2H), 7.38–7.35 (m, 3H), 7.21–7.18 (m, 5H), 4.42 (s, 2H), 4.30 (s, 1H), 3.92 (s, 1H), 3.91 (s, 5H), 3.62 (s, 3H), 2.76 – 2.70 (m, 1H), 2.47 – 2.37 (m, 1H), 2.27 – 2.21 (m, 1H), 1.65 – 1.58 (m, 2H), 1.49 (d, $J = 6.8$ Hz, 3H), 1.45 – 1.40 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.2, 140.5, 138.2, 135.3, 135.1, 132.3, 132.1, 128.8, 127.8, 127.7, 127.5, 127.4, 127.2, 97.5, 75.0, 71.3, 69.6 (5C), 69.3, 68.7, 61.9, 51.5, 51.3, 45.1, 29.0, 22.7, 15.7; ^{31}P NMR (162 MHz, $CDCl_3$, 85 % H_3PO_4): δ -22.77; MS (ESI) m/z (%) : 525.0 (100) $[M^+ + 1]$; HRMS (ESI) Calcd. for $C_{30}H_{32}FeNNaO_2P$ 548.1418 $[M + Na]$, found: 548.1420.

***N*-[(*S*)-1-(*R*)-2'-diphenylphosphinoferrocenyl]ethylpyrrolidine (LB8)**

Following a similar procedure as **LB7**, **LB8** (0.44g, 94%) was obtained as a yellow solid; m.p.: 98.6–100.4 °C; $[\alpha]_D^{20} = +340.8^\circ$ (c 1.0, $CHCl_3$), (Lit.^[2] no data); ^1H

NMR (400 MHz, CDCl₃) δ 7.58–7.54 (m, 2H), 7.34–7.32 (m, 3H), 7.23–7.16 (m, 5H), 4.46 (s, 1H), 4.27 (s, 1H), 4.12 (m, 2H), 3.92 (s, 5H), 2.30–2.17 (m, 4H), 1.44 (d, J = 4.8 Hz, 3H), 1.21–1.08 (m, 4H); HRMS (ESI) Calcd. for C₂₈H₃₀FeNP 467.1465 [M⁺], found 467.1459.

***N*-[(*S*)-1-(*R*)-2-diphenylphosphinoferrocenyl]ethylmorpholine (LB9)**

Following a similar procedure as **LB7**, **LB9** (0.31g, 64%) was obtained as a yellow solid; m.p.: 180.9–182.0 °C; $[\alpha]_{\text{D}}^{20}$ = +341.6° (c 1.0, CHCl₃), (Lit.^[2] no data); ¹H NMR (400 MHz, CDCl₃) δ 7.58–7.54 (m, 2H), 7.36–7.35 (m, 3H), 7.22 (s, 5H), 4.39 (s, 1H), 4.28–4.16 (m, 3H), 3.95 (s, 5H), 3.00–2.81 (m, 4H), 2.34–2.28 (m, 4H), 1.33 (d, J = 6.4 Hz, 3H); HRMS (ESI) Calcd. for C₂₈H₃₀FeNOP 483.1414 [M⁺], found 484.1415.

2. Representative procedure for the asymmetric [3+2] cycloaddition

A solution of methyl 2-(((*tert*-butoxycarbonyl)oxy)-(4-nitrophenyl)methyl)-acrylate **1a** (0.17g, 0.5 mmol) and *N*-Phenyl maleimide **2a** (0.17g, 1 mmol) and chiral ferrocenylphosphine **LB7** (26.25mg, 0.05 mmol) in toluene (1.0 mL) was stirred at room temperature for 48h. The solvent was removed under reduced pressure and the residue was purified by column chromatography to give **3a** (0.07 g, 37%) as a white solid.

(3a*R*,4*S*,6a*S*)-methyl 4-(4-nitrophenyl)-1,3-dioxo-2-phenyl-1,2,3,3a,4,6a-hexahyd-rocyclopenta[*c*]pyrrole-5-carboxylate(3a); white solid; m.p.: 62.2–64.2 °C; $[\alpha]_{\text{D}}^{20}$ =

+97.4° (c 1.0, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.21 (d, *J* = 8.8 Hz, 2H), 7.52 – 7.43 (m, 2H), 7.43 – 7.39 (m, 3H), 7.28 – 7.25 (m, 2H), 7.05 (dd, *J* = 2.4, 1.6 Hz, 1H), 4.77 – 4.75 (m, 1H), 4.37 – 4.33 (m, 1H), 3.68 (s, 3H), 3.51 (dd, *J* = 7.6, 2.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 175.9, 173.3, 162.7, 148.6, 147.2, 140.3, 138.7, 131.2, 129.1 (2C), 128.8, 127.9 (2C), 126.4 (2C), 124.2 (2C), 53.2, 53.1, 52.3, 52.2; MS (ESI) *m/z* (%): 391.1 (100) [M⁻1]; HRMS (ESI) Calcd. for C₂₁H₁₅N₂O₆ 391.0930 [M-H], found: 391.0917; Enantiomeric excess was determined by HPLC with a Chiral OD-RH column [λ = 214 nm; eluent: acetonitrile/water = 60/40; flow rate: 0.5 mL/min; *t*_{major} = 16.365 min, *t*_{minor} = 13.167 min; *ee* = 48%].

(3a*R*,4*S*,6a*S*)-methyl 1,3-dioxo-2,4-diphenyl-1,2,3,3a,4,6a-hexahydrocyclopenta[c]pyrrole-5-carboxylate (3b); Following a similar procedure as **3a**, **3b** was obtained with as a colorless oil; [α]_D²⁰ = +23.7° (c 0.6, CHCl₃), {Lit.^[5] a colorless oil; [α]_D²⁵ = +239.4° (c 1.0, CHCl₃)}; ¹H NMR (400 MHz, CDCl₃) δ 7.46 – 7.42 (m, 2H), 7.40 – 7.36 (m, 1H), 7.32 (t, *J* = 7.6 Hz, 3H), 7.27 – 7.25 (m, 2H), 7.22 – 7.19 (m, 2H), 6.95 (dd, *J* = 2.4, 1.2 Hz, 1H), 4.67 – 4.65 (m, 1H), 4.28 – 4.25 (m, 1H), 3.64 (s, 3H), 3.50 (dd, *J* = 7.6, 2.0 Hz, 1H). MS (ESI) *m/z* (%): 348.0 (100) [M⁺+1]; [Lit.^[5] HRMS (ESI) Calcd. for C₂₁H₁₇NO₄ 347.1153 [M⁺], found: 347.1154]. Enantiomeric excess was determined by HPLC with a Chiral OD-RH column [λ = 214 nm; eluent: acetonitrile/water=65/35; flow rate: 0.5 mL/min; *t*_{major} = 13.041 min, *t*_{minor} = 9.374 min; *ee* = 43%].

(3aR,4S,6aS)-methyl 1,3-dioxo-2-phenyl-4-(p-tolyl)-1,2,3,3a,4,6a-hexahydrocyclopenta[c]pyrrole-5-carboxylate (3c); Following a similar procedure as **3a**, **3c** was obtained with as a colorless oil; $[\alpha]_{\text{D}}^{20} = +19.0^{\circ}$ (c 0.6, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.47 – 7.43 (m, 2H), 7.40 – 7.36 (m, 1H), 7.27 – 7.24 (m, 2H), 7.14 (d, $J = 8.0$ Hz, 2H), 7.10 (d, $J = 8.0$ Hz, 2H), 6.95 – 6.93 (m, 1H), 4.64 (s, 1H), 4.29 – 4.26 (m, 1H), 3.66 (s, 3H), 3.50 (dd, $J = 7.6, 2.0$ Hz, 1H), 2.33 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 176.7, 174.1, 163.2, 141.5, 138.4, 137.2, 136.9, 131.4, 129.5 (2C), 129.0 (2C), 128.6, 126.6 (2C), 126.2 (2C), 53.15, 53.09, 53.0, 52.0, 21.2; MS (ESI) m/z (%): 360.2 (100) [M⁻1]; HRMS (ESI) Calcd. for C₂₂H₁₈NO₄ 360.1236 [M-H], found: 360.1242; Enantiomeric excess was determined by HPLC with a Chiral OD-RH column [$\lambda = 214$ nm; eluent: acetonitrile/water = 60/40; flow rate: 0.5 mL/min; $t_{\text{major}} = 13.707$ min, $t_{\text{minor}} = 11.293$ min; $ee = 40\%$].

(3aR,4S,6aS)-methyl 4-(4-methoxyphenyl)-1,3-dioxo-2-phenyl-1,2,3,3a,4,6a-hexahydrocyclopenta[c]pyrrole-5-carboxylate (3d); Following a similar procedure as **3a**, **3d** was obtained with as a colorless oil; $[\alpha]_{\text{D}}^{20} = +42.3^{\circ}$ (c 0.6, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.48 (t, $J = 7.6$ Hz, 2H), 7.42 – 7.39 (m, 1H), 7.29 – 7.27 (m, 2H), 7.15 (d, $J = 8.8$ Hz, 2H), 6.96 – 6.94 (m, 1H), 6.88 (d, $J = 8.8$ Hz, 2H), 4.66 – 4.63 (m, 1H), 4.32 – 4.29 (m, 1H), 3.81 (s, 3H), 3.68 (s, 3H), 3.51 (dd, $J = 7.6, 2.0$ Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 176.7, 174.1, 173.0, 163.2, 158.6, 141.5, 137.0, 136.9, 133.4, 131.3, 129.0, 128.6, 127.9, 127.8, 126.2, 114.2, 114.1, 55.3, 52.9, 52.8, 52.7, 52.0; MS (ESI) m/z (%): 378.0 (100) [M⁺+1]; HRMS (ESI) Calcd. for C₂₂H₁₉NNaO₅

400.1161 [M+Na], found: 400.1164; Enantiomeric excess was determined by HPLC with a Chiral OD-RH column [λ = 214 nm; eluent: acetonitrile/water = 60/40; flow rate: 0.5 mL/min; t_{major} = 12.030 min, t_{minor} = 10.246 min; ee = 37%].

(3aR,4S,6aS)-methyl 4-(4-fluorophenyl)-1,3-dioxo-2-phenyl-1,2,3,3a,4,6a-hexahydrocyclopenta[c]pyrrole-5-carboxylate (3e); Following a similar procedure as **3a**, **3e** was obtained with as a white solid; m.p.: 113.3-114.8 °C; $[\alpha]_{\text{D}}^{20}$ = +103.8° (c 1.0, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.46 – 7.44 (m, 2H), 7.39 – 7.35 (m, 1H), 7.26 – 7.23 (m, 2H), 7.18 (dd, J = 8.0, 5.2 Hz, 2H), 7.01 (t, J = 8.8 Hz, 2H), 6.95 (s, 1H), 4.64 (s, 1H), 4.29 – 4.26 (m, 1H), 3.65 (s, 3H), 3.47 (dd, J = 7.2, 2.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 176.50, 173.9, 163.0, 160.6, 141.1, 137.6, 137.5, 137.1, 131.2, 129.0, 128.7, 128.44, 128.38, 126.2, 126.1, 115.8, 115.7, 52.9, 52.8, 52.7, 52.1; MS (ESI) m/z (%): 366.0 (100) [M⁺+1]; HRMS (ESI) Calcd. for C₂₁H₁₅FO₄ 364.0985 [M-H], found: 364.0985; Enantiomeric excess was determined by HPLC with a Chiral OD-RH column [λ = 214 nm; eluent: acetonitrile/water = 65/35; flow rate: 0.4 mL/min; t_{major} = 11.817 min, t_{minor} = 9.987 min; ee = 41%].

(3aR,4S,6aS)-methyl 4-(2-chlorophenyl)-1,3-dioxo-2-phenyl-1,2,3,3a,4,6a-hexahydrocyclopenta[c]pyrrole-5-carboxylate (3f); Following a similar procedure as **3a**, **3f** was obtained with as a white solid; m.p.: 44.6-47.4 °C; $[\alpha]_{\text{D}}^{20}$ = +104.0° (c 0.6, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.50 – 7.37 (m, 5H), 7.30 – 7.27 (m, 2H), 7.25 – 7.21 (m, 2H), 7.08 – 7.06 (m, 1H), 5.15 (s, 1H), 4.31 – 4.28 (m, 1H), 3.68 (s, 3H),

3.56 – 3.53 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 176.0, 173.8, 163.0, 140.3, 139.0, 138.8, 138.3, 133.5, 131.3, 130.3, 130.2, 129.1, 129.0, 128.6, 127.2, 126.3, 126.1, 53.1, 52.1, 52.0, 50.3; MS (ESI) m/z (%): 382.0 (100) $[\text{M}^+ + 1]$; HRMS (ESI) Calcd. for $\text{C}_{21}\text{H}_{16}\text{ClNNaO}_4$ 404.0666 $[\text{M} + \text{Na}]$, found: 404.0658; Enantiomeric excess was determined by HPLC with a Chiral OD-RH column [λ = 214 nm; eluent: acetonitrile/water = 60/40; flow rate: 0.5 mL/min; t_{major} = 14.345 min, t_{minor} = 11.414 min; ee = 42%].

(3a*R*,4*S*,6a*S*)-methyl 4-(3-chlorophenyl)-1,3-dioxo-2-phenyl-1,2,3,3a,4,6a-hexahydrocyclopenta[*c*]pyrrole-5-carboxylate (3g); Following a similar procedure as **3a**, **3g** was obtained with as a white solid; m.p.: 33.7-35.8 °C; $[\alpha]_{\text{D}}^{20}$ = +98.0° (c 1.0, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 7.47 – 7.44 (m, 2H), 7.40 – 7.37 (m, 1H), 7.30 – 7.23 (m, 4H), 7.18 (s, 1H), 7.13 (dd, J = 6.8, 1.6 Hz, 1H), 6.98 (dd, J = 2.4, 1.6 Hz, 1H), 4.63 (s, 1H), 4.30 – 4.27 (m, 1H), 3.67 (s, 3H), 3.48 (dd, J = 7.6, 2.0 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 176.3, 173.7, 162.9, 143.3, 140.7, 138.2, 138.0, 134.7, 131.2, 130.2, 129.1, 128.8, 127.7, 126.9, 126.8, 126.2, 125.2, 53.1, 53.0, 52.6, 52.2; MS (ESI) m/z (%): 380.0 (100) $[\text{M}^- - 1]$; HRMS (ESI) Calcd. for $\text{C}_{21}\text{H}_{15}\text{ClNO}_4$ 380.0690 $[\text{M} - \text{H}]$, found: 380.0695; Enantiomeric excess was determined by HPLC with a Chiral OD-RH column [λ = 214 nm; eluent: acetonitrile/water = 60/40; flow rate: 0.5 mL/min; t_{major} = 16.402 min, t_{minor} = 11.096 min; ee = 48%].

(3a*R*,4*S*,6a*S*)-methyl 4-(4-chlorophenyl)-1,3-dioxo-2-phenyl-1,2,3,3a,4,6a-hexahydro-

drocyclopenta[c]pyrrole-5 carboxylate (3h); Following a similar procedure as **3a**, **3h** was obtained with as a white solid; m.p.: 72.9-74.5 °C; $[\alpha]_D^{20} = +114.8^\circ$ (c 1.0, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.47 – 7.43 (m, 2H), 7.40 – 7.36 (m, 1H), 7.30 (d, *J* = 8.4 Hz, 2H), 7.25 (d, *J* = 6.8 Hz, 2H), 7.15 (d, *J* = 8.4 Hz, 2H), 6.96 (dd, *J* = 2.4, 1.6 Hz 1H), 4.63 (s, 1H), 4.29 – 4.26 (m, 1H), 3.66 (s, 3H), 3.47 (dd, *J* = 7.2, 2.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 176.4, 173.7, 163.0, 141.0, 140.1 (2C), 139.9, 137.8 (2C), 133.2 (2C), 131.3, 129.1, 128.7, 128.2, 126.2 (2C), 53.0, 52.8, 52.4, 52.1; MS (ESI) *m/z* (%): 380.1 (100) [M⁻1]; HRMS (ESI) Calcd. for C₂₁H₁₅ClNO₄ 380.0690 [M-H], found: 380.0679; Enantiomeric excess was determined by HPLC with a Chiral OD-RH column [λ = 214 nm; eluent: acetonitrile/water = 60/40; flow rate: 0.5 mL/min; *t*_{major} = 16.705 min, *t*_{minor} = 13.345 min; *ee* = 41%].

(3a*R*,4*S*,6a*S*)-methyl 4-(3-bromophenyl)-1,3-dioxo-2-phenyl-1,2,3,3a,4,6a-hexahydrocyclopenta[c]pyrrole-5-carboxylate (3i); Following a similar procedure as **3a**, **3i** was obtained with as a white solid; m.p.: 47.6-49.4 °C; $[\alpha]_D^{20} = +107.4^\circ$ (c 1.0, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.46 (t, *J* = 8.0 Hz, 2H), 7.42 – 7.37 (m, 2H), 7.33 (t, *J* = 1.6 Hz, 1H), 7.28 – 7.26 (m, 2H), 7.23 – 7.15 (m, 2H), 6.99 (dd, *J* = 2.4, 1.6 Hz, 1H), 4.65 – 4.63 (m, 1H), 4.34 – 4.31 (m, 1H), 3.68 (s, 3H), 3.49 (dd, *J* = 7.6, 2.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 176.2, 173.6, 162.9, 143.7, 140.7, 138.1, 131.3, 130.5, 130.4, 129.8, 129.0(2C), 128.6, 126.2, 125.7(2C), 122.9, 53.1, 53.0, 52.7, 52.1; MS (ESI) *m/z* (%): 424.0 (100) [M⁻1]; HRMS (ESI) Calcd. for C₂₁H₁₅BrNO₄ 424.0184 [M-H], found: 424.0190; Enantiomeric excess was

determined by HPLC with a Chiral OD-RH column [λ = 214 nm; eluent: acetonitrile/water = 60/40; flow rate: 0.5 mL/min; t_{major} = 20.965 min, t_{minor} = 12.739 min; ee = 47%].

(3a*R*,4*S*,6a*S*)-methyl 4-(4-bromophenyl)-1,3-dioxo-2-phenyl-1,2,3,3a,4,6a-hexahydrocyclopenta[c]pyrrole-5-carboxylate (3j); Following a similar procedure as **3a**, **3j** was obtained with as a white solid; m.p.: 70.2-72.2 °C; $[\alpha]_{\text{D}}^{20}$ = +50.5° (c 0.4, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.49 – 7.43 (m, 4H), 7.41 – 7.36 (m, 1H), 7.27 – 7.24 (m, 2H), 7.10 (d, J = 8.4 Hz, 2H), 6.97 (dd, J = 2.4, 1.6 Hz, 1H), 4.63 – 4.62 (m, 1H), 4.30 – 4.27 (m, 1H), 3.67 (s, 3H), 3.48 (dd, J = 7.6, 2.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 176.3, 173.7, 162.9, 140.9, 140.4, 137.8, 131.9 (2C), 131.2, 129.0 (2C), 128.7, 128.5 (2C), 126.2 (2C), 121.3, 52.9, 52.6, 52.4, 52.1; MS (ESI) m/z (%): 424.1(100) [M^-]; HRMS (ESI) Calcd. for C₂₁H₁₅BrNO₄ 424.0184 [$M-H$], found: 424.0178; Enantiomeric excess was determined by HPLC with a Chiral OD-RH column [λ = 214 nm; eluent: acetonitrile/water = 60/40; flow rate: 0.5 mL/min; t_{major} = 19.298 min, t_{minor} = 14.879 min; ee = 34%].

(3a*R*,4*S*,6a*S*)-methyl 1,3-dioxo-2-phenyl-4-((*E*)-styryl)-1,2,3,3a,4,6a-hexahydrocyclopenta[c]pyrrole-5-carboxylate (3k); Following a similar procedure as **3a**, **3k** was obtained with as a yellow solid; m.p.: 157.6-158.8 °C; $[\alpha]_{\text{D}}^{20}$ = +65.0° (c 0.6, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.47 – 7.44 (m, 2H), 7.40 – 7.35 (m, 3H), 7.30 (t, J = 7.6 Hz, 2H), 7.27 – 7.18 (m, 3H), 6.85 (dd, J = 2.4, 1.6 Hz, 1H), 6.57 (d, J =

16.0 Hz, 1H), 6.22 (dd, $J = 16.0, 8.0$ Hz, 1H), 4.32 – 4.28 (m, 1H), 4.24 – 4.21 (m, 1H), 3.75 (s, 3H), 3.50 (dd, $J = 7.6, 2.0$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 176.6, 174.2, 163.3, 140.7, 137.3, 137.2, 136.2, 131.7, 131.6, 131.3, 129.1, 129.0, 128.5 (2C), 127.8, 126.3 (2C), 126.1 (2C), 52.7, 52.2, 50.5, 50.2; MS (ESI) m/z (%): 374.3(100) [$\text{M}^+ + 1$]; HRMS (ESI) Calcd. for $\text{C}_{23}\text{H}_{19}\text{NNaO}_4$ 396.1212 [$\text{M} + \text{Na}$], found: 396.1199; Enantiomeric excess was determined by HPLC with a Chiral OD-RH column [$\lambda = 214$ nm; eluent: acetonitrile/water = 60/40; flow rate: 0.5 mL/min; $t_{\text{major}} = 17.728$ min, $t_{\text{minor}} = 14.160$ min; $ee = 24\%$].

(3a*R*,4*S*,6a*S*)-methyl 4-(furan-2-yl)-1,3-dioxo-2-phenyl-1,2,3,3a,4,6a-hexahydro-cyclopenta[*c*]pyrrole-5-carboxylate (3l); Following a similar procedure as **3a**, **3l** was obtained with as a white solid; m.p.: 87.4-88.6 °C; $[\alpha]_{\text{D}}^{20} = +66.8^\circ$ (c 1.0, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 7.47 – 7.43 (m, 2H), 7.40 – 7.36 (m, 1H), 7.32 (d, $J = 2.0$ Hz, 1H), 7.26 – 7.23 (m, 2H), 6.93 – 6.90 (m, 1H), 6.31 (dd, $J = 3.2, 2.0$ Hz, 1H), 6.20 (d, $J = 3.2$ Hz, 1H), 4.78 – 4.77 (m, 1H), 4.33 – 4.30 (m, 1H), 3.72 (s, 3H), 3.71 – 3.69 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 176.3, 174.0, 162.9, 152.7, 141.9, 138.7, 138.2, 131.3, 129.0(2C), 128.7, 126.2(2C), 110.6, 106.6, 53.0, 52.1, 49.9, 46.6; MS (ESI) m/z (%): 336.2 (100) [$\text{M}^- - 1$]; HRMS (ESI) Calcd. for $\text{C}_{19}\text{H}_{14}\text{NO}_5$ 336.0872 [$\text{M} - \text{H}$], found 336.0866; Enantiomeric excess was determined by HPLC with a Chiral AD-RH column [$\lambda = 274$ nm; eluent: acetonitrile / 0.05mmol.L $^{-1}$ $\text{KH}_2\text{PO}_4 = 70/30$; flow rate: 0.6 mL/min; $t_{\text{major}} = 5.997$ min, $t_{\text{minor}} = 4.148$ min; $ee = 53\%$].

(3aR,4S,6aS)-methyl 2-methyl-4-(4-nitrophenyl)-1,3-dioxo-1,2,3,3a,4,6a-hexahydrocyclopenta[c]pyrrole-5-carboxylate (3m); Following a similar procedure as **3a**, **3m** was obtained with as a colorless oil; $[\alpha]_D^{20} = +40.6^\circ$ (c 1.0, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.19 (d, *J* = 8.4 Hz, 2H), 7.38 (d, *J* = 8.4 Hz, 2H), 6.97 – 6.96 (m, 1H), 4.64 (s, 1H), 4.22 – 4.20 (m, 1H), 3.65 (s, 3H), 3.34 (dd, *J* = 7.6, 2.4 Hz, 1H), 3.02 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 176.9, 174.4, 162.7, 148.7, 147.1, 139.9, 138.8, 138.7, 127.9, 127.8, 124.2, 53.0, 52.8, 52.3, 52.2, 25.5; MS (ESI) *m/z* (%): 329.0 (100) [M⁻-1]; HRMS (ESI) Calcd. for C₁₆H₁₃N₂O₆ 329.0774 [M-H], found: 329.0779].

(3aR,4S,6aS)-methyl 2-benzyl-4-(4-nitrophenyl)-1,3-dioxo-1,2,3,3a,4,6a-hexahydrocyclopenta[c]pyrrole-5-carboxylate (3n); Following a similar procedure as **3a**, **3n** was obtained with as a white solid; m.p.: 47.8-49.8 °C; $[\alpha]_D^{20} = +25.3^\circ$ (c 0.6, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.18 (d, *J* = 8.8 Hz, 2H), 7.38 – 7.33 (m, 4H), 7.31 – 7.28 (m, 3H), 6.98 – 6.97 (m, 1H), 4.66 – 4.65 (m, 3H), 4.22 – 4.19 (m, 1H), 3.65 (s, 3H), 3.34 (dd, *J* = 7.6, 2.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 176.6, 174.0, 162.7, 148.7, 147.1, 139.9, 138.8, 134.9, 128.6 (2C), 128.0 (2C), 127.80 (2C), 127.75, 124.1 (2C), 58.4, 53.0, 52.8, 52.2, 42.9; MS (ESI) *m/z* (%): 405.1 (100) [M⁻-1]; HRMS (ESI) Calcd. for C₂₂H₁₇N₂O₆ 405.1087 [M-H], found: 405.1105; Enantiomeric excess was determined by HPLC with a Chiral OD-RH column [λ = 214 nm; eluent: acetonitrile/water = 60/40; flow rate: 0.5 mL/min; *t*_{major} = 18.878 min, *t*_{minor} = 16.276 min; *ee* = 26%].

(3aR,4S,6aS)-methyl 2-(4-chlorophenyl)-4-(4-nitrophenyl)-1,3-dioxo-1,2,3,3a,4,6a-hexahydrocyclopenta[c]pyrrole-5-carboxylate (3o); Following a similar procedure as **3a**, **3o** was obtained with as a yellow solid; m.p.: 85.8-87.8 °C; $[\alpha]_{\text{D}}^{20} = +68.4^\circ$ (c 1.0, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.20 (d, *J* = 8.8 Hz, 2H), 7.45 – 7.39(m, 5H), 7.23 (s, 1H), 7.03 (s, 1H), 4.75 (s, 1H), 4.37 – 4.34 (m, 1H), 3.68 (s, 3H), 3.51 (dd, *J* = 7.6, 2.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 175.7, 173.1, 162.6, 148.4, 147.2, 140.4, 138.6, 138.4, 134.7, 129.6, 129.4, 129.3, 127.9, 127.8, 127.4, 127.3, 124.3, 53.3, 53.1, 53.0, 52.3; MS (ESI) *m/z* (%): 425.0 (100) [M⁻1]; HRMS (ESI) Calcd. for C₂₁H₁₄ClN₂O₆ 425.0540 [M-H], found: 425.0546; Enantiomeric excess was determined by HPLC with a Chiral OD-RH column [λ = 214 nm; eluent: acetonitrile/water = 60/40; flow rate: 0.5 mL/min; *t*_{major} = 16.489 min, *t*_{minor} = 14.861 min; *ee* = 52%].

(3aR,4S,6aS)-methyl 2-(4-methoxyphenyl)-4-(4-nitrophenyl)-1,3-dioxo- 1,2,3,3a,4,6a-hexahydrocyclopenta[c]pyrrole-5-carboxylate (3p); Following a similar procedure as **3a**, **3p** was obtained with as a white solid; m.p.: 65.1-67.5 °C; $[\alpha]_{\text{D}}^{20} = +130.2^\circ$ (c 1.0, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.21 (d, *J* = 8.8 Hz, 2H), 7.41 (d, *J* = 8.8 Hz, 2H), 7.18 (d, *J* = 8.8 Hz, 2H), 7.07-7.04 (m, 1H), 6.98 (d, *J* = 8.8 Hz, 2H), 4.76 (s, 1H), 4.36-4.33 (m, 1H), 3.84 (s, 3H), 3.69 (s, 3H), 3.50 (dd, *J* = 7.6, 2.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 176.2, 173.6, 162.7, 159.5, 148.6, 147.2, 140.2, 138.8, 127.9 (2C), 127.4 (2C), 124.2 (2C), 123.7, 114.4 (2C), 55.5, 53.2, 53.0, 52.2 (2C); MS (ESI) *m/z* (%): 421.1 (100) [M⁻1]; HRMS (ESI) Calcd. for

C₂₂H₁₇N₂O₇ 421.1036 [M-H], found: 421.1031; Enantiomeric excess was determined by HPLC with a Chiral AD-RH column [λ = 274 nm; eluent: acetonitrile/0.05mmol.L⁻¹ KH₂PO₄ = 70/30-90/10; flow rate: 0.6 mL/min; t_{major} = 13.901 min, t_{minor} = 5.760 min; ee = 45%].

(3aR,4S,6aS)-ethyl 4-(4-nitrophenyl)-1,3-dioxo-2-phenyl-1,2,3,3a,4,6a-hexahydro-cyclopenta[c]pyrrole-5-carboxylate (3q); Following a similar procedure as **3a**, **3q** was obtained with as a yellow oil; $[\alpha]_{\text{D}}^{20} = +96.6^{\circ}$ (c 0.6, CHCl₃), {Lit.^[6] yellow oil; $[\alpha]_{\text{D}}^{20} = +383.9^{\circ}$ (c 1.0, CHCl₃)}; ¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, J = 8.4 Hz, 2H), 7.50 – 7.47 (m, 2H), 7.44 – 7.41 (m, 3H), 7.30 – 7.27 (m, 2H), 7.06 (dd, J = 2.0, 1.6 Hz, 1H), 4.77 (s, 1H), 4.38 – 4.35 (m, 1H), 4.16 – 4.10 (m, 2H), 3.53 (dd, J = 7.6, 2.0 Hz, 1H), 1.21 (t, J = 7.2 Hz, 3H); MS (ESI) m/z (%): 405.1 (100) [M⁻1]; [Lit.^[6] HRMS (ESI) Calcd. for C₂₂H₁₈N₂NaO₆ 429.1057 [M+Na], found: 429.1062]. Enantiomeric excess was determined by HPLC with a Chiral OD-RH column [λ = 214 nm; eluent: acetonitrile/water = 60/40; flow rate: 0.5 mL/min; t_{major} = 18.103 min, t_{minor} = 14.778 min; ee = 44%].

(3aR,4S,6aS)-butyl 4-(4-nitrophenyl)-1,3-dioxo-2-phenyl-1,2,3,3a,4,6a-hexahydro-cyclopenta[c]pyrrole-5-carboxylate (3r); Following a similar procedure as **3a**, **3r** was obtained with as a colorless oil; $[\alpha]_{\text{D}}^{20} = +65.4^{\circ}$ (c 1.0, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.20 (d, J = 8.4 Hz, 2H), 7.49 - 7.46 (m, 2H), 7.42 - 7.40 (m, 3H), 7.27 (d, J = 7.2 Hz, 2H), 7.05 (s, 1H), 4.75 (s, 1H), 4.36 – 4.33 (m, 1H), 4.12 – 3.99

(m, 2H), 3.51 (dd, $J = 7.6, 2.0$ Hz, 1H), 1.55 -1.48 (m, 2H), 1.22 – 1.17 (m, 2H), 0.85 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 176.0, 173.4, 162.3, 148.8, 147.1, 140.6, 138.5, 131.2, 129.0 (2C), 128.7, 127.8 (2C), 126.1(2C), 124.0 (2C), 65.1, 53.2, 52.9, 52.3, 30.4, 19.0, 13.6; MS (ESI) m/z (%): 433.1 (100) $[\text{M}^- - 1]$; HRMS (ESI) Calcd. for $\text{C}_{24}\text{H}_{21}\text{N}_2\text{O}_6$ 433.1400 $[\text{M} - \text{H}]$, found: 433.1395; Enantiomeric excess was determined by HPLC with a Chiral OD-RH column [$\lambda = 214$ nm; eluent: acetonitrile/water = 60/40; flow rate: 0.5 mL/min; $t_{\text{major}} = 22.622\text{min}$, $t_{\text{minor}} = 18.937\text{min}$; $ee = 42\%$].

3. Analytical spectra ($^1\text{H}/^{13}\text{C}$ -NMR & MS / HRMS & HPLC) for compounds

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2013\3.21\TQljjbj.d
 Method tune_POS_low.m
 Sample Name TQljjbj
 Comment

Acquisition Date 3/21/2013 4:41:04 PM

Operator bruker
 Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Meas. m/z	Formula	m/z	err [ppm]	mSigma
563.1383	C 32 H 32 Fe N 2 P S	563.1368	-2.5	28.6

20130518t ap-P31
20130518t ap QIC13 P31

E:\Data\2013\20130518-1TAP

05/30/2013 09:04:20 AM

20130518-1TAP

20130518-1TAP #19 RT: 0.54 AV: 1 NL: 3.50E7
T: + c ESI ms [50.00-1000.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2013\12.25\20131221-3TAP.d
Method tune_POS_low.m
Sample Name 20131221-3TAP
Comment

Acquisition Date 12/25/2013 9:26:00 AM

Operator bruker
Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

TQ20120518-2
TQ20120518-2 CDCl₃ H1

TQ20120518-2
TQ20120518-2 CDCl₃ C13

³¹P NMR spectrum of compound 10 in CDCl₃. The chemical structure of 10 is shown in the inset. The spectrum displays two main signals: a doublet at δ 21.23 ppm and a doublet at δ 29.76 ppm. The x-axis represents the chemical shift in ppm, ranging from 300 to -300.

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2013\1.31\TQslbi.d
Method tune_POS_low.m
Sample Name TQslbi
Comment

Acquisition Date 1/31/2013 2:24:48 PM

Operator bruker
Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Meas. m/z	Formula	m/z	err [ppm]	mSigma
755.1497	C 43 H 38 Fe N 2 Na P 2 S	755.1473	-3.2	11.7

TQSLDF-P31
TQSLDF CDCl₃ F31

TQ20120518-4 #6 RT: 0.22 AV: 1 NL: 1.65E8

T: - C ESI Full ms [150.00-1500.00]

TQ20120518-4 #4-5 RT: 0.14-0.17 AV: 2 NL: 3.14E7

T: - C ESI Full ms [150.00-1500.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2013\1.31\TQsldFbi.d
Method tune_POS_low.m
Sample Name TQsldFbi
Comment

Acquisition Date 1/31/2013 2:17:42 PM

Operator bruker
Instrument / Ser# microTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Meas. m/z	Formula	m/z	err [ppm]	mSigma
773.1396	C 43 H 37 F Fe N 2 Na P 2 S	773.1379	-2.2	10.4

20131118tap-P31
20131118tap QC13 P31

E:\Data\2013\201312\20131202TAP

12/09/2013 02:51:19 PM

20131202TAP

20131202TAP #17 RT: 0.46 AV: 1 SB: 5 0.17-0.25, 0.17 NL: 2.22E6
T: + c ESI Full ms [50.00-1000.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2013\12.25\20131221-4TAP.d
Method tune_POS_low.m
Sample Name 20131221-4TAP
Comment

Acquisition Date 12/25/2013 9:16:54 AM

Operator bruker
Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2013\12.27\20131221-1TAP.d
Method tune_POS_low.m
Sample Name 20131221-1TAP
Comment

Acquisition Date 12/27/2013 2:31:37 PM

Operator bruker
Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2013\12.27\20131221-2TAP.d
Method tune_POS_low.m
Sample Name 20131221-2TAP
Comment

Acquisition Date 12/27/2013 2:39:01 PM

Operator bruker
Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

20130227tap #1 RT: 0.01 AV: 1 NL: 5.48E6
T: - c ESI ms [50.00-1200.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2014\1.3\20131112-2TAP-2.d
 Method tune_NEG_low.m
 Sample Name 20131112-2TAP
 Comment

Acquisition Date 1/3/2014 11:57:00 AM

Operator bruker
 Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Meas. m/z	Formula	m/z	err [ppm]	mSigma
391.0917	C ₂₁ H ₁₅ N ₂ O ₆	391.0936	4.8	5.8

Data File E:\DATA\TAP\TAP 2013-06-05 18-55-22\001-0101.D
Sample Name: 20130530-1TAP

```
=====
Acq. Operator   : ZHONG                      Seq. Line :    1
Acq. Instrument : Instrument 1                Location  : Vial 1
Injection Date  : 6/5/2013 6:56:12 PM        Inj       :    1
                                           Inj Volume: 5.0 µl
Different Inj Volume from Sequence !      Actual Inj Volume: 3.0 µl
Acq. Method     : E:\DATA\TAP\TAP 2013-06-05 18-55-22\TAP.M
Last changed    : 6/5/2013 6:55:20 PM by ZHONG
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/17/2014 2:22:39 PM by wky
                  (modified after loading)
=====
```

Area Percent Report

```
=====
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.101	BV	0.3506	2.36655e4	1037.29126	50.8613
2	16.185	BB	0.4110	2.28640e4	863.08838	49.1387

Totals : 4.65295e4 1900.37964

*** End of Report ***

Data File E:\DATA\TAP\TAP 2013-03-18 15-03-20\052-0101.D
Sample Name: 20130311TAP

=====

Acq. Operator : PAN	Seq. Line : 1
Acq. Instrument : Instrument 1	Location : Vial 52
Injection Date : 3/18/2013 3:04:04 PM	Inj : 1
	Inj Volume : 5.0 µl
Different Inj Volume from Sequence !	Actual Inj Volume : 2.0 µl

Acq. Method : E:\DATA\TAP\TAP 2013-03-18 15-03-20\TAP.M
Last changed : 3/14/2013 2:55:34 PM by PAN
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed : 3/14/2014 9:49:32 AM by wky
(modified after loading)

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.167	BB	0.3543	1720.61230	75.60212	74.4694
2	16.365	BB	0.4310	589.88318	21.48078	25.5306

Totals : 2310.49548 97.08290

=====
*** End of Report ***

20130822-1TAP #37 RT: 0.59 AV: 1 NL: 1.09E7
T: + c ESI Full ms [200.00-400.00]

20130822-1TAP #13 RT: 0.26 AV: 1 NL: 1.23E5
T: - c ESI Full ms [200.00-400.00]

Data File E:\DATA\MFR\TAP 2013-08-27 09-48-00\2010121403.D
Sample Name: 20130822-1tap

=====

Acq. Operator : L	Seq. Line : 3
Acq. Instrument : Instrument 1	Location : Vial 19
Injection Date : 8/27/2013 10:56:18 AM	Inj : 1
	Inj Volume : 5.0 µl

Different Inj Volume from Sequence ! Actual Inj Volume : 30.0 µl

Acq. Method : E:\DATA\MFR\TAP 2013-08-27 09-48-00\TAP.M
Last changed : 8/27/2013 10:54:51 AM by L
(modified after loading)

Analysis Method : C:\CHEM32\1\METHODS\TAP.M

Last changed : 3/14/2014 3:51:15 PM by L
(modified after loading)

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.182	BB	0.2135	3149.83032	227.56776	52.6031
2	10.865	BB	0.3305	2838.09131	132.19363	47.3969

Totals : 5987.92163 359.76140

=====
*** End of Report ***
=====

Data File E:\DATA\TAP\TAP 2013-12-02 09-39-44\035-0101.D
Sample Name: 20131121-1tap

```
=====
Acq. Operator   : L                               Seq. Line :    1
Acq. Instrument : Instrument 1                     Location  : Vial 35
Injection Date  : 12/2/2013 9:40:36 AM             Inj       :    1
                                                    Inj Volume: 5.0 µl
Acq. Method     : E:\DATA\TAP\TAP 2013-12-02 09-39-44\TAP.M
Last changed    : 11/6/2009 11:03:31 AM by L
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 10:11:11 AM by wky
                  (modified after loading)
=====
```

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.374	BB	0.2944	1947.90881	103.93105	71.5222
2	13.041	BB	0.4999	775.59222	24.15096	28.4778

Totals : 2723.50104 128.08202

=====
*** End of Report ***

20130824-2TAP #103 RT: 2.05 AV: 1 NL: 5.42E6
T: - c ESI Full ms [50.00-1000.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2014\1.18\20130824-2TAP-1.d
Method tune_NEG_low.m
Sample Name 20130824-2TAP
Comment

Acquisition Date 1/18/2014 10:18:05 AM

Operator bruker
Instrument / Ser# microTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Meas. m/z	Formula	m/z	err [ppm]	mSigma
360.1242	C ₂₂ H ₁₈ N ₂ O ₄	360.1241	-0.2	8.7

Data File E:\DATA\MFR\TAP 2013-08-29 15-14-36\2010121407.D
Sample Name: 20130824-2tap

=====

Acq. Operator : L	Seq. Line : 7
Acq. Instrument : Instrument 1	Location : Vial 16
Injection Date : 8/29/2013 6:29:45 PM	Inj : 1
	Inj Volume : 5.0 µl
Different Inj Volume from Sequence !	Actual Inj Volume : 20.0 µl
Acq. Method : E:\DATA\MFR\TAP 2013-08-29 15-14-36\TAP.M	
Last changed : 8/29/2013 7:05:43 PM by L	
(modified after loading)	
Analysis Method : C:\CHEM32\1\METHODS\TAP.M	
Last changed : 3/14/2014 3:52:37 PM by L	
(modified after loading)	

=====

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=214,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.575	BB	0.3321	2585.47217	119.65311	50.1126
2	14.141	BB	0.4030	2573.84839	98.46755	49.8874

Totals : 5159.32056 218.12067

=====
*** End of Report ***
=====

Data File E:\DATA\TAP\TAP 2013-12-02 09-39-44\037-0401.D
Sample Name: 20131123-1tap

```
=====
Acq. Operator   : L                               Seq. Line :    4
Acq. Instrument : Instrument 1                     Location  : Vial 37
Injection Date  : 12/2/2013 11:29:14 AM            Inj       :    1
                                                Inj Volume: 5.0 µl
Different Inj Volume from Sequence !      Actual Inj Volume: 2.0 µl
Acq. Method     : E:\DATA\TAP\TAP 2013-12-02 09-39-44\TAP.M
Last changed    : 11/6/2009 11:03:31 AM by L
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 10:12:18 AM by wky
                  (modified after loading)
=====
```

Area Percent Report

```
=====
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.293	BB	0.3455	1.36972e4	622.66211	70.4297
2	13.707	BB	0.4299	5750.83789	210.14459	29.5703

Totals : 1.94481e4 832.80670

*** End of Report ***

20140312-1tap #68 RT: 1.38 AV: 1 NL: 7.52E5
T: + c ESI Full ms [100.00-600.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2014\3.31\20140312-1TAP.d
 Method tune_POS_low.m
 Sample Name 20140312-1TAP
 Comment

Acquisition Date 4/14/2014 2:35:40 PM

Operator bruker
 Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Data File E:\DATA\TAP\TAP 2014-03-29 16-39-14\074-0401.D
Sample Name: 20140312-Itap

=====

Acq. Operator : wky	Seq. Line : 4
Acq. Instrument : Instrument 1	Location : Vial 74
Injection Date : 3/29/2014 6:28:09 PM	Inj : 1
	Inj Volume : 5.0 µl
Different Inj Volume from Sequence !	Actual Inj Volume : 1.0 µl
Acq. Method : E:\DATA\TAP\TAP 2014-03-29 16-39-14\TAP1.M	
Last changed : 3/29/2014 4:58:00 PM by wky	
	(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\TAP.M	
Last changed : 3/31/2014 9:56:46 AM by wky	
	(modified after loading)

=====

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.220	BB	0.2764	3.57180e4	2003.82874	49.8587
2	12.002	BV	0.3221	3.59204e4	1731.21252	50.1413

Totals : 7.16384e4 3735.04126

=====
*** End of Report ***
=====

Data File E:\DATA\TAP\TAP 2014-03-29 19-45-30\075-0101.D
Sample Name: 20140312-2tap

```
=====
Acq. Operator   : wky                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 75
Injection Date  : 3/29/2014 7:46:15 PM      Inj       :    1
                                           Inj Volume: 5.0 µl
                                           Actual Inj Volume: 2.0 µl
Different Inj Volume from Sequence !
Acq. Method     : E:\DATA\TAP\TAP 2014-03-29 19-45-30\TAP1.M
Last changed    : 3/29/2014 8:08:19 PM by wky
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/31/2014 9:57:31 AM by wky
                  (modified after loading)
=====
```

Area Percent Report

```
=====
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	10.246	BB	0.2740	3840.81812	216.40285	68.9608
2	12.030	VB	0.3210	1728.75159	83.21284	31.0392

Totals : 5569.56970 299.61568

*** End of Report ***

20130822-2TAP #24 RT: 0.54 AV: 1 NL: 1.34E7
T: -c ESI Full ms [50.00-600.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2014\3.11\20130822-2TAP.d
 Method tune_NEG_low.m
 Sample Name 20130822-2TAP
 Comment

Acquisition Date 3/11/2014 7:05:25 PM

Operator bruker
 Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Data File E:\DATA\MFR\TAP 2013-08-27 14-15-58\2010121402.D
Sample Name: 20130822-2tap

```
=====
Acq. Operator   : L                               Seq. Line :    2
Acq. Instrument : Instrument 1                     Location  : Vial 20
Injection Date  : 8/27/2013 2:50:19 PM             Inj       :    1
                                                    Inj Volume: 5.0 µl
                                                    Actual Inj Volume: 10.0 µl

Different Inj Volume from Sequence !

Acq. Method     : E:\DATA\MFR\TAP 2013-08-27 14-15-58\TAP.M
Last changed    : 8/27/2013 2:47:38 PM by L
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 3:54:24 PM by L
                  (modified after loading)
=====
```

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.078	BB	0.2466	1.02153e4	638.70624	50.0120
2	11.670	VB	0.2869	1.02104e4	549.32574	49.9880

Totals : 2.04257e4 1188.03198

=====
*** End of Report ***
=====

Data File E:\DATA\TAP\TAP 2013-12-23 10-58-39\084-0301.D
Sample Name: 20131206-2tap

=====

Acq. Operator	: L	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 84
Injection Date	: 12/23/2013 12:11:34 PM	Inj	: 1
		Inj Volume	: 5.0 µl
Different Inj Volume from Sequence !		Actual Inj Volume	: 8.0 µl
Acq. Method	: E:\DATA\TAP\TAP 2013-12-23 10-58-39\TAP.M		
Last changed	: 12/23/2013 10:33:00 AM by L		
Analysis Method	: C:\CHEM32\1\METHODS\TAP.M		
Last changed	: 3/14/2014 10:13:18 AM by wky		
	(modified after loading)		

=====

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.987	BB	0.3340	1394.28333	65.96073	70.5763
2	11.817	BB	0.4002	581.28656	22.84443	29.4237

Totals : 1975.56989 88.80516

=====
*** End of Report ***
=====

Instrument 1 3/14/2014 10:13:21 AM wky

Page 1 of 1

20140224-2TAP #156 RT: 2.76 AV: 1 NL: 1.47E6
T: + c ESI Full ms [50.00-500.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2014\3.22\20140224-2TAP..d
 Method tune_POS_low.m
 Sample Name 20140224-2TAP.
 Comment

Acquisition Date 4/5/2014 4:17:18 PM

Operator bruker
 Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Meas. m/z	Formula	m/z	err [ppm]	mSigma
404.0658	C 21 H 16 Cl N Na O 4	404.0660	0.5	17.5

Data File E:\DATA\TAP\TAP 2013-12-02 13-59-07\048-0301.D
Sample Name: 20130918-1tap

=====

Acq. Operator : L	Seq. Line : 3
Acq. Instrument : Instrument 1	Location : Vial 48
Injection Date : 12/2/2013 3:12:13 PM	Inj : 1
	Inj Volume : 5.0 µl
Different Inj Volume from Sequence !	Actual Inj Volume : 7.0 µl
Acq. Method : E:\DATA\TAP\TAP 2013-12-02 13-59-07\TAP.M	
Last changed : 11/6/2009 11:03:31 AM by L	
Analysis Method : C:\CHEM32\1\METHODS\TAP.M	
Last changed : 3/14/2014 10:14:22 AM by wky	
(modified after loading)	

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.603	BB	0.3678	4714.86914	199.27615	50.1943
2	14.624	BB	0.4762	4678.37207	152.96103	49.8057

Totals : 9393.24121 352.23718

=====
*** End of Report ***

Data File E:\DATA\TAP\TAP 2013-12-23 10-58-39\083-0201.D
Sample Name: 20131206-1tap

=====

Acq. Operator	: L	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 83
Injection Date	: 12/23/2013 11:35:30 AM	Inj	: 1
		Inj Volume	: 5.0 µl
Different Inj Volume from Sequence !		Actual Inj Volume	: 8.0 µl
Acq. Method	: E:\DATA\TAP\TAP 2013-12-23 10-58-39\TAP.M		
Last changed	: 12/23/2013 10:33:00 AM by L		
Analysis Method	: C:\CHEM32\1\METHODS\TAP.M		
Last changed	: 3/14/2014 10:15:08 AM by wky		
	(modified after loading)		

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.414	BB	0.4052	1375.13159	53.92714	71.1608
2	14.345	BB	0.5378	557.29816	16.51479	28.8392

Totals : 1932.42975 70.44193

=====
*** End of Report ***
=====

Instrument 1 3/14/2014 10:15:11 AM wky

Page 1 of 1

20130912-1TAP #70 RT: 1.31 AV: 1 NL: 1.56E7
T: - c F3I Full ms [50.00-500.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2014\1.9\20130912-1TAP.d
Method tune_NEG_low.m
Sample Name 20130912-1TAP
Comment

Acquisition Date 1/9/2014 7:56:58 PM

Operator bruker
Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Meas. m/z	Formula	m/z	err [ppm]	mSigma
380.0687	C ₂₁ H ₁₅ ClNO ₄	380.0695	2.0	8.9

Data File E:\DATA\TAP\TAP 2013-12-02 13-59-07\046-0101.D
Sample Name: 20130912-1tap

=====

Acq. Operator	: L	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 46
Injection Date	: 12/2/2013 1:59:58 PM	Inj	: 1
		Inj Volume	: 5.0 µl

Acq. Method : E:\DATA\TAP\TAP 2013-12-02 13-59-07\TAP.M
Last changed : 11/6/2009 11:03:31 AM by L
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed : 3/14/2014 10:15:53 AM by wky
(modified after loading)

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.955	BB	0.3605	2192.09692	95.66399	49.9527
2	17.852	BB	0.6084	2196.24585	56.42459	50.0473

Totals : 4388.34277 152.08858

=====
*** End of Report ***
=====

Data File E:\DATA\TAP\TAP 2013-12-13 09-32-15\002-0201.D
Sample Name: 20131130-1tap

```
=====
Acq. Operator   : L                               Seq. Line :    2
Acq. Instrument : Instrument 1                     Location  : Vial 2
Injection Date  : 12/13/2013 10:09:07 AM           Inj       :    1
                                                    Inj Volume: 5.0 µl
Acq. Method     : E:\DATA\TAP\TAP 2013-12-13 09-32-15\TAP.M
Last changed    : 11/6/2009 11:03:31 AM by L
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 10:16:53 AM by wky
                  (modified after loading)
=====
```

Area Percent Report

```
=====
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.096	BB	0.3296	1.65740e4	784.08484	74.3012
2	16.402	BB	0.5559	5732.51367	160.14815	25.6988

Totals :   2.23065e4   944.23299

*** End of Report ***

20130829-1TAP #43 RT: 1.20 AV: 1 SB: 77 1.01-2.05, 0.98-2.05 NL: 7.70E5
T: - c ESI ms [50.00-1000.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2014\1.3\20130918-2TAP.d
 Method tune_NEG_low.m
 Sample Name 20130918-2TAP
 Comment

Acquisition Date 1/3/2014 12:30:11 PM

Operator bruker
 Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Data File E:\DATA\TAP\TAP 2013-12-02 13-59-07\049-0501.D
Sample Name: 20130918-2tap

```
=====
Acq. Operator   : L                               Seq. Line :    5
Acq. Instrument : Instrument 1                     Location  : Vial 49
Injection Date  : 12/2/2013 4:24:20 PM             Inj       :    1
                                                    Inj Volume: 5.0 µl
Different Inj Volume from Sequence !      Actual Inj Volume: 2.0 µl
Acq. Method     : E:\DATA\TAP\TAP 2013-12-02 13-59-07\TAP.M
Last changed    : 11/6/2009 11:03:31 AM by L
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 10:17:52 AM by wky
                  (modified after loading)
=====
```

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.634	BB	0.4233	1.71187e4	632.81403	50.0954
2	17.083	BB	0.5328	1.70535e4	502.47000	49.9046

Totals : 3.41722e4 1135.28403

=====
*** End of Report ***
=====

Data File E:\DATA\TAP\TAP 2013-12-13 15-38-47\004-0101.D
Sample Name: 20131128-2tap

```
=====
Acq. Operator   : L                               Seq. Line :    1
Acq. Instrument : Instrument 1                     Location  : Vial 4
Injection Date  : 12/13/2013 3:39:38 PM            Inj       :    1
                                                    Inj Volume: 5.0 µl
Acq. Method     : E:\DATA\TAP\TAP 2013-12-13 15-38-47\TAP.M
Last changed    : 11/6/2009 11:03:31 AM by L
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 10:19:07 AM by wky
                  (modified after loading)
=====
```

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	13.345	BB	0.4218	1.19486e4	441.84705	70.8210
2	16.705	BB	0.5269	4922.97803	146.71283	29.1790

Totals : 1.68716e4 588.55988

=====
*** End of Report ***

20130912-2TAP #50-51 RT: 0.84-0.85 AV: 2 NL: 5.95E6
T₁ - c ESI Full ms [50.00-500.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2014\1.9\20130912-2TAP.d
 Method tune_NEG_low.m
 Sample Name 20130912-2TAP
 Comment

Acquisition Date 1/9/2014 7:48:52 PM

Operator bruker
 Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Meas. m/z	Formula	m/z	err [ppm]	mSigma
424.0183	C 21 H 15 Br N O 4	424.0190	1.6	12.0

Data File E:\DATA\TAP\TAP 2013-12-02 13-59-07\047-0201.D
Sample Name: 20130912-2tap

```
=====
Acq. Operator   : L                               Seq. Line :    2
Acq. Instrument : Instrument 1                     Location  : Vial 47
Injection Date  : 12/2/2013 2:36:05 PM             Inj       :    1
                                                    Inj Volume: 5.0 µl
Different Inj Volume from Sequence !      Actual Inj Volume: 7.0 µl
Acq. Method     : E:\DATA\TAP\TAP 2013-12-02 13-59-07\TAP.M
Last changed    : 11/6/2009 11:03:31 AM by L
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 10:20:19 AM by wky
                  (modified after loading)
=====
```

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	13.067	VV	0.4062	1.88106e4	724.58966	49.9746
2	21.501	BB	0.7588	1.88297e4	385.73520	50.0254

Totals : 3.76403e4 1110.32486

=====
*** End of Report ***
=====

Data File E:\DATA\TAP\TAP 2013-12-07 10-14-50\008-0101.D
Sample Name: 20131130-2tap

```
=====
Acq. Operator   : L                               Seq. Line :    1
Acq. Instrument : Instrument 1                     Location  : Vial 8
Injection Date  : 12/7/2013 10:15:39 AM           Inj       :    1
                                                Inj Volume: 5.0 µl
Acq. Method     : E:\DATA\TAP\TAP 2013-12-07 10-14-50\TAP.M
Last changed    : 12/7/2013 10:48:17 AM by L
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 10:21:35 AM by wky
                  (modified after loading)
=====
```

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	12.739	BB	0.3676	4532.85059	192.69672	73.9637
2	20.965	BB	0.7107	1595.62915	34.73177	26.0363

Totals : 6128.47974 227.42848

=====
*** End of Report ***
=====

E:\Data\2013\20130824-1TAP

09/02/2013 08:55:35 AM

20130824-1TAP

20130824-1TAP #25 RT: 0.71 AV: 1 NL: 1.48E5
T: - c ESI ms [50.00-1000.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2014\1.3\20130824-1TAP-1.d
 Method tune_NEG_low.m
 Sample Name 20130824-1TAP
 Comment

Acquisition Date 1/3/2014 12:15:27 PM

Operator bruker
 Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Data File E:\DATA\MFR\TAP 2013-08-29 15-14-36\2010121405.D
Sample Name: 20130824-1tap

```
=====
Acq. Operator   : L                               Seq. Line :    5
Acq. Instrument : Instrument 1                     Location  : Vial 15
Injection Date  : 8/29/2013 5:16:47 PM             Inj       :    1
                                                    Inj Volume: 5.0 µl

Acq. Method     : E:\DATA\MFR\TAP 2013-08-29 15-14-36\TAP.M
Last changed    : 8/29/2013 4:57:03 PM by L
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 3:55:37 PM by L
                  (modified after loading)
=====
```

Area Percent Report

```
=====
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: DAD1 A, Sig=214,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.404	BB	0.4395	4529.70801	160.37756	49.8197
2	19.940	BB	0.5675	4562.49561	125.09035	50.1803

Totals : 9092.20361 285.46792

*** End of Report ***

Data File E:\DATA\TAP\TAP 2013-12-02 09-39-44\036-0201.D
Sample Name: 20131121-2tap

```
=====
Acq. Operator   : L                               Seq. Line :    2
Acq. Instrument : Instrument 1                     Location  : Vial 36
Injection Date  : 12/2/2013 10:16:48 AM           Inj       :    1
                                                Inj Volume: 5.0 µl
Different Inj Volume from Sequence !      Actual Inj Volume: 15.0 µl
Acq. Method     : E:\DATA\TAP\TAP 2013-12-02 09-39-44\TAP.M
Last changed    : 11/6/2009 11:03:31 AM by L
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 10:22:37 AM by wky
                  (modified after loading)
=====
```

=====
Area Percent Report
=====

```
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.879	VB	0.6324	1.19386e5	2967.88501	67.3240
2	19.298	BB	0.6460	5.79443e4	1408.33191	32.6760

Totals :   1.77330e5 4376.21692

=====
*** End of Report ***

E:\Data\2013\20130824-3TAP

09/02/2013 08:36:53 AM

20130824-3TAP

20130824-3TAP #9-11 RT: 0.28-0.33 AV: 3 SB: 6 1.97-2.04, 1.69-1.75 NL: 1.96E5

T: + c ESI ms [50.00-1000.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2014\3.12\20140227-1TAP.d
 Method tune_POS_low.m
 Sample Name 20140227-1TAP
 Comment

Acquisition Date 3/12/2014 1:57:23 PM

Operator bruker
 Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Meas. m/z	Formula	m/z	err [ppm]	mSigma
396.1199	C 23 H 19 N Na O 4	396.1206	1.7	9.7

Data File E:\DATA\MFR\TAP 2013-08-29 15-14-36\2010121404.D
Sample Name: 20130824-3tap

```
=====
Acq. Operator   : L                               Seq. Line :    4
Acq. Instrument : Instrument 1                     Location  : Vial 17
Injection Date  : 8/29/2013 4:40:18 PM             Inj       :    1
                                                    Inj Volume: 5.0 µl
Different Inj Volume from Sequence !      Actual Inj Volume: 8.0 µl
Acq. Method     : E:\DATA\MFR\TAP 2013-08-29 15-14-36\TAP.M
Last changed    : 8/29/2013 4:57:03 PM by L
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 3:57:33 PM by L
                  (modified after loading)
=====
```

Area Percent Report

```
=====
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: DAD1 A, Sig=214,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.585	BB	0.4152	7215.00000	270.45349	52.0828
2	18.177	BB	0.5245	6637.95215	196.28455	47.9172

Totals : 1.38530e4 466.73804

*** End of Report ***

Data File E:\DATA\TAP\TAP 2014-01-06 09-37-56\075-0301.D
Sample Name: 20131214tap

```
=====
Acq. Operator   : L                               Seq. Line :    3
Acq. Instrument : Instrument 1                     Location  : Vial 75
Injection Date  : 1/6/2014 10:35:27 AM             Inj       :    1
                                                    Inj Volume: 5.0 µl
Different Inj Volume from Sequence !      Actual Inj Volume: 10.0 µl
Acq. Method     : E:\DATA\TAP\TAP 2014-01-06 09-37-56\TAP.M
Last changed    : 1/6/2014 10:41:57 AM by L
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 10:23:32 AM by wky
                  (modified after loading)
=====
```

Area Percent Report

```
=====
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.160	BB	0.4743	926.31561	31.08714	62.0422
2	17.728	BB	0.5828	566.72559	14.97345	37.9578

Totals : 1493.04120 46.06059

*** End of Report ***

E:\Data\...20140307-1TAP_140317092201

03/17/2014 09:22:01 AM

20140307-1TAP

20140307-1TAP_140317092201 #108 RT: 1.82 AV: 1 NL: 5.65E6
T: - c ESI Full ms [50.00-500.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2014\3.27\20140307-1TAP..d
 Method tune_NEG_low.m
 Sample Name 20140307-1TAP.
 Comment

Acquisition Date 4/10/2014 9:44:41 AM

Operator bruker
 Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Data File E:\DATA\TAP\TAP 2014-03-15 13-39-06\024-0201.D
Sample Name: 20140307-1tap

```
=====
Acq. Operator   : wky                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 24
Injection Date  : 3/15/2014 2:05:49 PM      Inj       :    1
                                           Inj Volume: 5.0 µl
Different Inj Volume from Sequence !      Actual Inj Volume: 2.0 µl
Acq. Method     : E:\DATA\TAP\TAP 2014-03-15 13-39-06\TAP.M
Last changed    : 3/15/2014 2:25:51 PM by wky
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/15/2014 3:12:45 PM by wky
=====
```

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	4.150	BV	0.1548	8462.98535	834.18024	49.9862
2	4.798	VB	0.1419	58.83668	6.41843	0.3475
3	6.020	BB	0.4284	8408.82617	303.14957	49.6663

Totals : 1.69306e4 1143.74824

=====
*** End of Report ***

Data File E:\DATA\TAP\TAP 2014-03-15 13-39-06\025-0301.D
Sample Name: 20140307-2tap

```
=====
Acq. Operator   : wky                      Seq. Line :    3
Acq. Instrument : Instrument 1              Location  : Vial 25
Injection Date  : 3/15/2014 2:28:48 PM      Inj       :    1
                                           Inj Volume: 5.0 µl
                                           Actual Inj Volume: 1.0 µl
Different Inj Volume from Sequence !
Acq. Method     : E:\DATA\TAP\TAP 2014-03-15 13-39-06\TAP.M
Last changed    : 3/15/2014 2:49:20 PM by wky
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/15/2014 3:31:42 PM by wky
                  (modified after loading)
=====
```

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	4.148	BB	0.1519	8160.64014	824.13599	76.3456
2	4.797	BB	0.1446	55.70586	6.08575	0.5211
3	5.997	BB	0.4139	2472.72656	92.89463	23.1332

Totals : 1.06891e4 923.11637

=====
*** End of Report ***

Instrument 1 3/15/2014 3:31:54 PM wky

Page 1 of 1

20131224-1TAP #112 RT: 2.19 AV: 1 NL: 1.56E7
T: -c ESI Full ms [50.00-400.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2014\1.9\20131224-1TAP.d
 Method tune_NEG_low.m
 Sample Name 20131224-1TAP
 Comment

Acquisition Date 1/9/2014 8:10:38 PM

Operator bruker
 Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Meas. m/z	Formula	m/z	err [ppm]	mSigma
329.0779	C ₁₆ H ₁₃ N ₂ O ₆	329.0779	0.2	8.6

20131213-1TAP #58 RT: 1.54 AV: 1 NL: 2.84E7
T: -c ESI Full ms [50.00-1000.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2013\12.28\20131213-1TAP-2.d
 Method tune_NEG_low.m
 Sample Name 20131213-1TAP
 Comment

Acquisition Date 12/28/2013 10:09:45 AM

Operator bruker
 Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Data File E:\DATA\TAP\TAP 2013-12-26 10-28-45\051-0101.D
Sample Name: 20131213-1tap

=====

Acq. Operator	: L	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 51
Injection Date	: 12/26/2013 10:29:34 AM	Inj	: 1
		Inj Volume	: 5.0 µl

Acq. Method : E:\DATA\TAP\TAP 2013-12-26 10-28-45\TAP.M
Last changed : 12/26/2013 10:28:43 AM by L
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed : 3/14/2014 10:45:37 AM by wky
(modified after loading)

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	16.284	BB	0.5493	6142.51367		167.83238	50.0797
2	18.878	BB	0.6563	6122.96191		139.08620	49.9203

Totals : 1.22655e4 306.91858

=====
*** End of Report ***

Data File E:\DATA\TAP\TAP 2013-12-26 10-28-45\052-0201.D
Sample Name: 20131213-1tap

```
=====
Acq. Operator   : L                               Seq. Line :    2
Acq. Instrument : Instrument 1                     Location  : Vial 52
Injection Date  : 12/26/2013 11:05:40 AM           Inj       :    1
                                                    Inj Volume: 5.0 µl
Acq. Method     : E:\DATA\TAP\TAP 2013-12-26 10-28-45\TAP.M
Last changed    : 12/26/2013 10:28:43 AM by L
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 10:46:31 AM by wky
                  (modified after loading)
=====
```

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	16.276	BV	0.5743	1.43581e4	385.48404	63.3464
2	18.878	VB	0.6759	8307.95020	187.40392	36.6536

Totals : 2.26661e4 572.88795

=====
*** End of Report ***
=====

20131226-1TAP #54-55 RT: 1.36-1.39 AV: 2 NL: 1.14E7
T: - c ESI Full ms [50.00-1000.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2014\1.9\20131226-1TAP.d
 Method tune_NEG_low.m
 Sample Name 20131226-1TAP
 Comment

Acquisition Date 1/9/2014 8:02:38 PM

Operator bruker
 Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Meas. m/z	Formula	m/z	err [ppm]	mSigma
425.0545	C 21 H 14 Cl N 2 O 6	425.0546	0.3	15.4

Data File E:\DATA\TAP\TAP 2014-01-10 12-17-35\017-0301.D
Sample Name: 20131226-1tap

=====

Acq. Operator : L	Seq. Line : 3
Acq. Instrument : Instrument 1	Location : Vial 17
Injection Date : 1/10/2014 1:30:37 PM	Inj : 1
	Inj Volume : 5.0 µl
Different Inj Volume from Sequence !	Actual Inj Volume : 10.0 µl
Acq. Method : E:\DATA\TAP\TAP 2014-01-10 12-17-35\TAP.M	
Last changed : 1/10/2014 12:17:47 PM by L	
	(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\TAP.M	
Last changed : 3/14/2014 10:47:24 AM by wky	
	(modified after loading)

=====

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.877	BB	0.4856	3489.09985	112.96248	49.8434
2	16.500	BB	0.5368	3511.01978	102.40843	50.1566

Totals : 7000.11963 215.37091

=====
*** End of Report ***

Data File E:\DATA\TAP\TAP 2014-01-10 12-17-35\018-0201.D
Sample Name: 20131226-2tap

```
=====
Acq. Operator   : L                               Seq. Line :    2
Acq. Instrument : Instrument 1                     Location  : Vial 18
Injection Date  : 1/10/2014 12:54:29 PM            Inj       :    1
                                                Inj Volume: 5.0 µl
Acq. Method     : E:\DATA\TAP\TAP 2014-01-10 12-17-35\TAP.M
Last changed    : 1/10/2014 12:17:47 PM by L
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 10:47:24 AM by wky
                  (modified after loading)
=====
```

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.861	BB	0.4970	2111.03125	67.29232	76.3629
2	16.489	BB	0.5411	653.44141	18.92850	23.6371

Totals : 2764.47266 86.22082

=====
*** End of Report ***

20140116-2TAP #84 RT: 1.62 AV: 1 NL: 8.72E7
T: -c ESI Full.ms [50.00-1000.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2014\3.11\20140116-2TAP.d
 Method tune_NEG_low.m
 Sample Name 20140116-2TAP
 Comment

Acquisition Date 3/11/2014 7:11:29 PM

Operator bruker
 Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Data File E:\DATA\TAP\TAP 2014-02-22 15-37-36\026-0201.D
Sample Name: 20140116-1tap

=====

Acq. Operator	: L	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 26
Injection Date	: 2/22/2014 4:14:11 PM	Inj	: 1
		Inj Volume	: 5.0 µl
Acq. Method	: E:\DATA\TAP\TAP 2014-02-22 15-37-36\TAP.M		
Last changed	: 2/22/2014 3:54:53 PM by L (modified after loading)		
Analysis Method	: C:\CHEM32\1\METHODS\TAP.M		
Last changed	: 3/14/2014 10:49:02 AM by wky (modified after loading)		

=====

=====

Area Percent Report

=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=274 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	5.760	BB	0.3427	4872.44336	217.66606	50.8958
2	13.947	BB	0.9198	4700.91943	78.67982	49.1042

Totals : 9573.36279 296.34588

=====

*** End of Report ***

Data File E:\DATA\TAP\TAP 2014-02-22 15-37-36\027-0301.D
Sample Name: 20140116-3tap

```
=====
Acq. Operator   : L                               Seq. Line :    3
Acq. Instrument : Instrument 1                     Location  : Vial 27
Injection Date  : 2/22/2014 4:49:59 PM             Inj       :    1
                                                    Inj Volume: 5.0 µl

Acq. Method     : E:\DATA\TAP\TAP 2014-02-22 15-37-36\TAP.M
Last changed    : 2/22/2014 3:54:53 PM by L
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 10:49:29 AM by wky
                  (modified after loading)
=====
```

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=274 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	5.760	BB	0.3375	1465.46204	66.79996	72.8118
2	13.901	BB	0.8635	547.21027	9.44844	27.1882

Totals : 2012.67230 76.24840

=====
*** End of Report ***

Data File E:\DATA\TAP\TAP 2013-09-14 09-38-40\2010121403.D
Sample Name: 20130906-1tap

=====

Acq. Operator : L	Seq. Line : 3
Acq. Instrument : Instrument 1	Location : Vial 56
Injection Date : 9/14/2013 10:35:51 AM	Inj : 1
	Inj Volume : 5.0 µl
Different Inj Volume from Sequence !	Actual Inj Volume : 15.0 µl
Acq. Method : E:\DATA\TAP\TAP 2013-09-14 09-38-40\TAP.M	
Last changed : 9/14/2013 9:38:39 AM by L	
Analysis Method : C:\CHEM32\1\METHODS\TAP.M	
Last changed : 3/17/2014 3:49:37 PM by L	
(modified after loading)	

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.141	BB	0.1301	14.90601	1.60843	0.4914
2	4.716	BV	0.1769	14.26647	1.25253	0.4703
3	4.999	VB	0.1474	11.03178	1.09664	0.3637
4	6.565	BB	0.2076	47.74760	3.32418	1.5741
5	7.396	BB	0.2308	199.64729	13.17628	6.5817
6	14.699	BB	0.4454	1379.59326	48.28056	45.4805
7	17.702	BB	0.5222	1366.18384	40.84678	45.0384

Totals : 3033.37626 109.58540

=====
*** End of Report ***
=====

Data File E:\DATA\TAP\TAP 2013-12-28 09-57-59\008-0201.D
Sample Name: 20131218-1tap

=====

Acq. Operator : L	Seq. Line : 2
Acq. Instrument : Instrument 1	Location : Vial 8
Injection Date : 12/28/2013 10:34:53 AM	Inj : 1
	Inj Volume : 5.0 µl
Different Inj Volume from Sequence !	Actual Inj Volume : 10.0 µl
Acq. Method : E:\DATA\TAP\TAP 2013-12-28 09-57-59\TAP.M	
Last changed : 12/28/2013 9:58:10 AM by L	
	(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\TAP.M	
Last changed : 3/14/2014 10:49:29 AM by wky	
	(modified after loading)

=====

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.778	BB	0.5151	2300.16064	69.86562	72.1393
2	18.103	BB	0.6030	888.33649	22.87610	27.8607

Totals : 3188.49713 92.74173

=====
*** End of Report ***

20140107-TAP #49 RT: 1.26 AV: 1 NL: 7.32E7
T: -c ESI ms [50.00-1000.00]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\2014\1.21\20140107-1TAP.d
 Method tune_NEG_low.m
 Sample Name 20140107-1TAP
 Comment

Acquisition Date 1/21/2014 3:56:43 PM

Operator bruker
 Instrument / Ser# micrOTOF-Q II 10366

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Meas. m/z	Formula	m/z	err [ppm]	mSigma
433.1395	C ₂₄ H ₂₁ N ₂ O ₆	433.1405	2.3	7.3

Data File E:\DATA\TAP\TAP 2014-01-18 09-42-24\003-0301.D
Sample Name: 20140107-1tap

```
=====
Acq. Operator   : L                               Seq. Line :    3
Acq. Instrument : Instrument 1                     Location  : Vial 3
Injection Date  : 1/18/2014 10:45:01 AM           Inj       :    1
                                                Inj Volume: 5.0 µl
Acq. Method     : E:\DATA\TAP\TAP 2014-01-18 09-42-24\TAP.M
Last changed    : 1/18/2014 10:10:54 AM by L
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 10:53:14 AM by wky
                  (modified after loading)
=====
```

Area Percent Report

```
=====
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.679	BB	0.8233	3.71979e4	666.08594	49.9234
2	22.415	BB	0.9033	3.73121e4	607.16791	50.0766

Totals : 7.45100e4 1273.25385

*** End of Report ***

Data File E:\DATA\TAP\TAP 2014-01-18 09-42-24\004-0401.D
Sample Name: 20140107-2tap

```
=====
Acq. Operator   : L                               Seq. Line :    4
Acq. Instrument : Instrument 1                     Location  : Vial 4
Injection Date  : 1/18/2014 11:21:01 AM           Inj       :    1
                                                Inj Volume: 5.0 µl

Acq. Method     : E:\DATA\TAP\TAP 2014-01-18 09-42-24\TAP.M
Last changed    : 1/18/2014 10:10:54 AM by L
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\TAP.M
Last changed    : 3/14/2014 10:55:26 AM by wky
                  (modified after loading)
=====
```

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.937	BB	0.7883	1.17133e4	220.09407	71.1285
2	22.622	BB	0.8865	4754.49170	79.22266	28.8715

Totals : 1.64678e4 299.31673

=====
*** End of Report ***

4. References

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