Supporting Information

Synthesis of functionalized 2-pyridones via Michael addition and cyclization reaction of amines, alkynes and dialkyl acetylene dicarboxylates

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EXPERIMENTAL SECTION

General Considerations. All reactions were carried out under an atmosphere of argon using standard Schlenk techniques unless otherwise noted. Column chromatography was carried out on silica gel (300–400 mesh) using a forced flow of eluent at 0.3–0.5 bar pressure. For TLC, silica gel GF254 was used and visualized by fluorescence quenching under UV light. Solvents were dried according to the standard procedure and were distilled prior to use.

$^1$H NMR, $^{13}$C NMR, $^{19}$F NMR spectra were recorded at 400 MHz spectrometers. The chemical shifts for $^1$H NMR were recorded in ppm downfield from tetramethylsilane (TMS) with the solvent resonance as the internal standard (2.05 ppm for CD$_3$COCD$_3$ or 7.26 ppm for CDCl$_3$). The chemical shifts for $^{13}$C NMR were recorded in ppm downfield using the central peak of CDCl$_3$ (77.16 ppm) or CD$_3$COCD$_3$ (29.84 ppm) as the internal standard. Coupling constants ($J$) are reported in Hz and refer to apparent peak multiplications. The abbreviations $s$, $d$, $t$, $q$, and $m$ stand for singlet, doublet, triplet, quartet, and multiplet in that order. All $^{13}$C NMR spectra were proton decoupled.

Experimental Section.

General Procedure for the Cyclization of Alkynes with Aliphatic Amines.

Terminal alkyne (0.5 mmol for monamine, 1.0 mmol for diamine) and aliphatic
amines (0.5 mmol) were mixed in wet EtOH (2 ml) under argon atmosphere. The solution was stirred at room temperature for 24 h. Subsequently, dialkyl acetylene dicarboxylate (0.5 mmol for monamine, 1.0 mmol for diamine) was added and the solution was stirred at 78 °C under argon atmosphere. After completion of the reaction (as monitored by TLC), the solvent was removed under reduced pressure and the resulting residue was subjected to silica gel chromatography or recrystallized to give the desired product.

**General Procedure for the Cyclization of Alkyne with Aromatic Amines.**

A solution of aromatic amine (0.5 mmol), dialkyl acetylene dicarboxylate (1 mmol) and wet ethanol (2 mL) were stirred vigorously at room temperature for 10 min. Then the mixture was heated for 24 h. The progress of reaction was monitored by TLC (ether/ethyl acetate = 2:1). The solvent was removed under reduced pressure; the resulting residue was purified by silica gel chromatography. Further purification was done by recrystallization in mixture of ether/ethyl acetate (4:1).

**Analytical Data**

3-Ethyl 4-methyl 1-methyl-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4aa): white solid; 80.3 mg; 67% yield; mp 146 – 149 °C; 

\[
\begin{align*}
\text{H NMR (400 MHz, CDCl}_3\text{)} & \delta 8.10 (s, 1H), 6.47 (s, 1H), 4.22 (s, 2H), 3.84 (s, 3H), 3.53 (s, 3H), 1.26 (s, 3H); \\
\text{13C NMR (100 MHz, CDCl}_3\text{)} & \delta 166.7, 163.1, 161.9, 144.3, 144.1, 118.2, 107.3, 63.5, 52.9, 38.2, 14.2; \\
\text{HRMS (Q-TOF, } & \text{ESI)} \\
\end{align*}
\]

S3
m/z) calcd for C_{11}H_{13}NO_{5}Na [M+Na]^+ 262.0691, found 262.0701.

3-Ethyl 4-methyl

1-ethyl-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ab): white solid; 104 mg; 83% yield; mp 67 – 68 °C; ^1H NMR (400 MHz, CD_{3}COCD_{3}) δ 8.43 (s, 1H), 6.42 (s, 1H), 4.23 (s, 2H), 4.08 (s, 2H), 3.84 (s, 3H), 1.34 (s, 3H), 1.29 (s, 3H); ^13C NMR (100 MHz, CD_{3}COCD_{3}) δ 166.9, 163.5, 161.5, 145.3, 118.4, 107.4, 61.7, 52.9, 46.0, 14.6, 14.4; HRMS (Q-TOF, m/z) calcd for C_{12}H_{15}NO_{5}Na [M+Na]^+ 276.0848, found 276.0853.

3-Ethyl 4-methyl

6-oxo-1-propyl-1,6-dihydropyridine-3,4-dicarboxylate (4ac): oil; 119 mg; 89% yield; ^1H NMR (400 MHz, CD_{3}COCD_{3}) δ 8.41 (s, 1H), 6.43 (s, 1H), 4.24 – 4.21 (m, 2H), 4.03 – 4.01 (m, 2H), 3.99 – 3.84 (m, 3H), 1.76 (d, J = 7.2 Hz, 2H), 1.28 (t, J = 6.9 Hz, 3H), 0.94 (t, J = 7.3 Hz, 3H); ^13C NMR (100 MHz, CD_{3}COCD_{3}) δ 167.1, 163.9, 161.6, 145.5, 144.8, 118.5, 107.1, 61.5, 52.9, 52.1, 22.9, 14.4, 11.1; HRMS (Q-TOF, m/z) calcd for C_{13}H_{17}NO_{5}Na [M+Na]^+ 290.0997, found 290.1004.

3-Ethyl 4-methyl

1-cyclopropyl-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ad): white solid; 104.6 mg; 79% yield; mp 115 – 116 °C; ^1H NMR (400 MHz, CD_{3}COCD_{3}) δ 8.20 (s,
1H), 6.39 (s, 1H), 4.24 (q, $J = 6.5$ Hz, 2H), 3.84 (s, 3H), 3.43 – 3.34 (m, 1H), 1.29 (t, $J = 6.4$ Hz, 3H), 1.11 (d, $J = 6.0$ Hz, 2H), 0.98 (s, 2H); $^{13}$C NMR (100 MHz, CD$_3$COCD$_3$) δ 167.4, 164.1, 162.1, 144.8, 144.7, 118.3, 106.7, 61.7, 52.9, 33.6, 14.4, 6.9; HRMS (Q-TOF, m/z) calcd for C$_{13}$H$_{15}$NO$_5$Na [M+Na]$^+$ 288.0855, found 288.0848.

3-Ethyl 4-methyl 1-butyl-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ae): oil; 109.6 mg; 78% yield; $^1$H NMR (400 MHz, CD$_3$COCD$_3$) δ 8.39 (s, 1H), 6.41 (s, 1H), 4.22 (m, 2H), 4.03 (s, 2H), 3.82 (s, 3H), 1.71 (s, 2H), 1.34 (s, 2H), 1.26 (s, 3H), 0.92 (s, 3H); $^{13}$C NMR (100 MHz, CD$_3$COCD$_3$) δ 167.29, 163.9, 160.6, 145.5, 144.8, 118.4, 107.2, 61.7, 52.9, 50.4, 31.8, 20.3, 14.4, 13.9; HRMS (Q-TOF, m/z) calcd for C$_{14}$H$_{19}$NO$_5$Na [M+Na]$^+$ 304.1161, found 304.1153.

3-Ethyl 4-methyl 1-cyclohexyl-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4af): white solid; 106.3 mg; 69% yield; mp 77 – 78 °C; $^1$H NMR (400 MHz, CD$_3$COCD$_3$) δ 8.31 (s, 1H), 6.43 (s, 1H), 4.67-4.70 (t, $J = 11.7$ Hz, 1H), 4.24 (d, $J = 3.3$ Hz, 2H), 3.84 (s, 3H), 1.92 (d, $J = 10.1$ Hz, 4H), 1.70 (t, $J = 11.3$ Hz, 3H), 1.49 (dd, $J = 24.3$, 12.4 Hz, 2H), 1.28 (s, 4H); $^{13}$C NMR (100 MHz, CD$_3$COCD$_3$) δ 167.4, 164.4, 161.1, 143.9), 141.5, 118.4, 107.3, 61.7, 56.2, 52.9, 32.4, 26.4, 25.7, 14.4; HRMS (Q-TOF, m/z) calcd for
C$_{16}$H$_{21}$NO$_5$Na [M+Na]$^+$ 330.1317, found 330.1310.

![Chemical Structure of 3-Ethyl 4-methyl 1-(2-fluorobenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ag)]

3-Ethyl 4-methyl 1-(2-fluorobenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ag): white solid; 114.7 mg; 69% yield; mp 106 – 117 °C; $^1$H NMR (400 MHz, CD$_3$COCD$_3$) δ 8.52 (s, 1H), 7.35 (t, $J$ = 6.4 Hz, 2H), 7.16 (dd, $J$ = 15.4, 8.1 Hz, 2H), 6.48 (s, 1H), 5.31 (s, 2H), 4.26 (q, $J$ = 7.1 Hz, 2H), 3.84 (s, 3H), 1.28 – 1.25 (m, 3H); $^{13}$C NMR (100 MHz, CD$_3$COCD$_3$) δ 167.1, 163.8, 163.0, 161.5, 160.2, 145.7, 145.1, 131.1, 131.0, 125.3, 118.7, 116.3, 107.7, 61.8, 52.9, 47.94, 47.90, 14.4; 19F NMR (377 MHz, CD$_3$COCD$_3$) δ -119.4; HRMS (Q-TOF, m/z) calcd for C$_{17}$H$_{16}$FNO$_5$Na [M+Na]$^+$ 356.0910, found 356.0897.

![Chemical Structure of 3-Ethyl 4-methyl 1-(2-chlorobenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ah)]

3-Ethyl 4-methyl 1-(2-chlorobenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ah): white solid; 141.4 mg; 81% yield; mp 114 – 115 °C; $^1$H NMR (400 MHz, CD$_3$COCD$_3$) δ 8.47 (s, 1H), 7.45 (s, 1H), 7.31 (s, 2H), 7.20 (s, 1H), 6.50 (s, 1H), 5.32 (s, 2H), 4.19-4.21 (d, $J$ = 6.8 Hz, 2H), 3.84 (s, 3H), 1.23 (s, 3H); $^{13}$C NMR (100 MHz, CD$_3$COCD$_3$) δ 166.9, 163.6, 161.4, 145.7, 145.6, 145.2, 134.2, 133.8, 130.49, 130.44, 0, 118.8, 107.6, 61.8, 53.0, 51.2, 14.4; HRMS (Q-TOF, m/z) calcd for C$_{17}$H$_{16}$ClNO$_5$Na [M+Na]$^+$ 372.0615, found 372.0631.
3-Ethyl 4-methyl

1-(4-chlorobenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ai): white solid; 128.9 mg; 74% yield; mp 132 – 133 ºC; \(^1\)H NMR (400 MHz, CD\(_3\)COCD\(_3\)) \(\delta\) 8.55 (s, 1H), 7.41 (dd, \(J = 19.1, 7.3\) Hz, 4H), 6.50 (s, 1H), 5.27 (s, 2H), 4.23 (d, \(J = 6.0\) Hz, 2H), 3.85 (s, 3H), 1.26 (t, \(J = 6.4\) Hz, 3H); \(^13\)C NMR (100 MHz, CD\(_3\)COCD\(_3\)) \(\delta\) 166.6, 163.6, 161.5, 145.4, 145.1, 136.2, 134.2, 130.7, 129.5, 118.9, 107.9, 61.8, 53.0, 52.5, 14.4; HRMS (Q-TOF, m/z) calcd for C\(_{17}\)H\(_{16}\)ClNO\(_5\)Na [M+Na]\(^+\) 372.0615, found 372.0631.

1-(2-methoxybenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4aj): white solid; 135.5 mg; 80% yield; mp 119 – 120 ºC; \(^1\)H NMR (400 MHz, CD\(_3\)COCD\(_3\)) \(\delta\) 8.51 (s, 1H), 7.31 (s, 2H), 7.03 (d, \(J = 6.3\) Hz, 1H), 6.93 (s, 1H), 6.43 (s, 1H), 5.16 (s, 2H), 4.23 (d, \(J = 2.4\) Hz, 2H), 3.89 (s, 3H), 3.83 (s, 3H), 1.28 (s, 3H); \(^13\)C NMR (100 MHz, CD\(_3\)COCD\(_3\)) \(\delta\) 167.2, 164.0, 161.6, 158.2, 146.2, 144.5, 131.7, 130.7, 121.3, 118.4, 111.6, 107.2, 61.7, 49.3, 14.4; HRMS (Q-TOF, m/z) calcd for C\(_{18}\)H\(_{19}\)NO\(_6\)Na [M+Na]\(^+\) 368.1110, found 368.1100.
1-(3-methoxybenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ak): white solid; 124.2 mg; 72% yield; mp 66 – 67 °C; \(^1\)H NMR (400 MHz, CD\(_3\)COCD\(_3\)) \(\delta\) 8.44 (s, 1H), 7.21 (d, J = 6.2 Hz, 1H), 6.93 (d, J = 9.9 Hz, 1H), 6.89 (s, 1H), 6.82 (s, 1H), 6.47 (s, 1H), 5.19 (s, 2H), 4.15 (s, 2H), 3.80 (s, 3H), 3.72 (s, 3H), 1.19 (s, 3H); \(^{13}\)C NMR (100 MHz, CD\(_3\)COCD\(_3\)) \(\delta\) 167.2, 163.8, 161.6, 160.9, 145.3, 144.9, 138.7, 130.7, 120.8, 118.8, 111.4, 107.8, 61.8, 55.5, 52.9, 52.8, 14.4; HRMS (Q-TOF, m/z) calcd for C\(_{18}\)H\(_{19}\)NO\(_6\)Na [M+Na]\(^+\) 368.1110, found 368.1100.

1-(4-methoxybenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4al): white solid; 104.3 mg; 61% yield; mp 97 – 98 °C; \(^1\)H NMR (400 MHz, CD\(_3\)COCD\(_3\)) \(\delta\) 8.43 (s, 1H), 7.31-7.34 (d, J = 8.7 Hz, 2H), 6.84-6.91 (d, J = 8.7 Hz, 2H), 6.44 (s, 1H), 5.13 (s, 2H), 4.14-4.19 (q, J = 7.1 Hz, 2H), 3.79 (s, 3H), 3.72 (s, 3H), 1.18-1.25 (t, J = 7.1 Hz, 3H); \(^{13}\)C NMR (100 MHz, CD\(_3\)COCD\(_3\)) \(\delta\) 167.2, 163.9, 161.7, 160.5, 145.1, 144.9, 130.6, 129.1, 118.8, 114.9, 107.7, 61.8, 55.5, 53.0, 52.5, 14.4; HRMS (Q-TOF, m/z) calcd for C\(_{18}\)H\(_{19}\)NO\(_6\)Na [M+Na]\(^+\) 368.1110, found 368.1098.
3-Ethyl 4-methyl 1-(2-methylbenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4am): white solid; 120.7 mg; 74% yield; mp 122 – 123 °C; $^1$H NMR (400 MHz, CD$_3$COCD$_3$) δ 8.31 (d, $J$ = 5.2 Hz, 1H), 7.20-7.24 (d, $J$ = 13.0 Hz, 3H), 7.04 (d, $J$ = 5.3 Hz, 1H), 6.50-6.52 (d, $J$ = 5.3 Hz, 1H), 5.26 (s, 2H), 4.18 (d, $J$ = 12.4 Hz, 2H), 3.84-3.87 (d, $J$ = 5.3 Hz, 3H), 2.35 (d, $J$ = 5.3 Hz, 3H), 1.22 – 1.25 (m, 3H); $^{13}$C NMR (100 MHz, CD$_3$COCD$_3$) δ 166.1, 163.9, 161.8, 145.2, 131.4, 128.8, 128.3, 127.1, 118.6, 107.3, 61.8, 53.0, 50.6, 19.1, 14.3; HRMS (Q-TOF, m/z) calcd for C$_{18}$H$_{19}$NO$_5$Na [M+Na]$^+$ 352.1161, found 352.1176.

3-Ethyl 4-methyl 1-(3-methylbenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4an): white solid; 129.2 mg; 79% yield; mp 118 – 119 °C; $^1$H NMR (400 MHz, CD$_3$COCD$_3$) δ 8.48 (s, 1H), 7.28 – 7.04 (m, 4H), 6.43 (s, 1H), 5.22 (s, 2H), 4.20-4.23 (s, 2H), 3.84 (s, 3H), 2.29 (s, 3H), 1.26 (s, 3H); $^{13}$C NMR (100 MHz, CD$_3$COCD$_3$) δ 167.1, 164.6, 162.2, 145.4, 137.2, 129.5, 125.9, 118.8, 107.8, 61.8, 52.98, 52.92, 21.3, 14.4; HRMS (Q-TOF, m/z) calcd for C$_{18}$H$_{19}$NO$_5$Na [M+Na]$^+$ 352.1161, found 352.1176.
3-Ethyl 4-methyl 1-(4-methylbenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ao): white solid; 127.6 mg; 78% yield; mp 128 – 129 °C; $^1$H NMR (400 MHz, CD$_3$COCD$_3$) $\delta$ 8.47 (s, 1H), 7.28-7.30 (d, $J = 8.0$ Hz, 2H), 7.16-7.18 (d, $J = 7.9$ Hz, 2H), 6.49 (s, 1H), 5.22 (s, 2H), 4.19-4.21 (q, $J = 7.1$ Hz, 2H), 3.84 (s, 3H), 2.29 (s, 3H), 1.24-1.27 (t, $J =$
7.1 Hz, 3H); $^{13}$C NMR (100 MHz, CD$_3$COCD$_3$) $\delta$ 166.7, 163.7, 161.3, 145.3, 144.9, 138.5, 134.3, 130.2, 129.0, 118.8, 107.5, 61.7, 52.9, 52.7, 21.0, 14.4; HRMS (Q-TOF, m/z) calcd for C$_{19}$H$_{19}$NO$_5$Na [M+Na]$^+$ 352.1161, found 352.1167.

Diethyl

1-(4-methylbenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ap): white solid; 116.5 mg; 68% yield; mp 121 – 122 °C; $^1$H NMR (400 MHz, CD$_3$COCD$_3$) $\delta$ 8.46 (s, 1H), 7.28-7.30 (d, $J$ = 8.0 Hz, 2H), 7.17-7.19 (d, $J$ = 7.9 Hz, 2H), 6.48 (s, 1H), 5.22 (s, 2H), 4.31 (q, $J$ = 7.1 Hz, 2H), 4.22 (q, $J$ = 7.1 Hz, 2H), 2.30 (s, 3H), 1.31 (t, $J$ = 7.1 Hz, 3H), 1.26 (t, $J$ = 7.1 Hz, 3H); $^{13}$C NMR (100 MHz, CD$_3$COCD$_3$) $\delta$ 166.1, 163.9, 161.7, 145.27, 145.23, 138.5, 134.3, 130.2, 128.9, 118.8, 107.8, 62.4, 61.7, 52.6, 21.0, 14.4, 14.2; HRMS (Q-TOF, m/z) calcd for C$_{19}$H$_{21}$NO$_5$Na [M+Na]$^+$ 366.1317, found 366.1311.

3-Ethyl

1-benzyl-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4aq): white solid; 146.7 mg; 93% yield; mp 130 – 131 °C; $^1$H NMR (400 MHz, DMSO) $\delta$ 8.67 (s, 1H), 7.28-7.35 (dd, $J$ = 17.2, 6.8 Hz, 5H), 6.58 (s, 1H), 5.23 (s, 2H), 4.18-4.24 (q, $J$ = 7.1 Hz, 2H), 3.80 (s, 3H), 1.18-1.26 (t, $J$ = 7.1 Hz, 3H); $^{13}$C NMR (100 MHz, DMSO) $\delta$ 167.5, 164.2, 161.9, 146.5, 145.0, 137.6, 130.0, 129.1, 129.0,
118.9, 107.5, 62.4, 54.0, 53.2, 15.3; HRMS (Q-TOF, m/z) calcd for C₁₇H₁₇NO₅Na+ [M+Na]+ 338.1004, found 338.1020.

**Dimethyl**

1-benzyl-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ar): white solid; 115.9 mg; 77% yield; mp 125 – 127 °C; ¹H NMR (400 MHz, CD₃COCD₃) δ 8.49 (s, 1H), 7.42 – 7.29 (m, 5H), 6.50 (s, 1H), 5.27 (s, 2H), 3.84 (s, 3H), 3.75 (s, 3H); ¹³C NMR (100 MHz, CD₃COCD₃) δ 167.1, 163.8, 161.7, 145.4, 145.0, 137.3, 129.6, 128.9, 128.8, 118.9, 107.4, 53.03, 53.00, 52.4.; HRMS (Q-TOF, m/z) calcd for C₁₆H₁₅NO₅Na+ [M+Na]+ 324.0848, found 324.0843.

3-Ethyl 4-methyl

1-(naphthalen-1-ylmethyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4as): white solid; 174 mg; 95% yield; mp 117 – 119 °C; ¹H NMR (400 MHz, CD₃COCD₃) δ 8.34 (s, 1H), 8.11 (d, J = 7.9 Hz, 1H), 7.94-7.98 (dd, J = 15.7, 8.0 Hz, 2H), 7.61 – 7.51 (m, 3H), 7.39-7.40 (d, J = 6.9 Hz, 1H), 6.58 (s, 1H), 5.76 (s, 2H), 4.12-4.18 (q, J = 7.0 Hz, 2H), 3.85 (s, 3H), 1.17-1.20 (t, J = 7.0 Hz, 3H); ¹³C NMR (100 MHz, CD₃COCD₃) δ 167.1, 163.7, 161.7, 144.9, 144.6, 129.81, 129.80, 129.73, 129.70, 127.13, 127.1, 123.9, 118.7, 107.7, 61.7, 53.0, 49.8, 14.3; HRMS (Q-TOF, m/z) calcd for C₂₁H₁₉NO₅Na+ [M+Na]+ 388.1161, found 388.1158.
3-Diethyl 4-dimethyl
1,1′-(butane-1,4-diyl)bis(6-oxo-1,6-dihydropyridine-3,4-dicarboxylate) (4at):
white solid; 0.305 mg; 61% yield; mp 216 – 217 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\)
8.11 (s, 2H), 6.51 (s, 2H), 4.26-4.28 (d, \(J = 6.6\) Hz, 4H), 4.01 (s, 4H), 3.88 (s, 6H),
1.79 (s, 4H), 1.32 (d, \(J = 26.7\) Hz, 6H); \(^1^3\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 166.5, 163.0,
161.5, 144.1, 143.5, 118.7, 107.8, 61.6, 53.0, 49.8, 26.1, 14.2; HRMS (Q-TOF, m/z) calcd for C\(_{24}\)H\(_{28}\)N\(_2\)O\(_{10}\)Na [M+Na]\(^+\) 527.1642, found 527.1649.

3-Diethyl 4-dimethyl
1,1′-(ethane-1,2-diyl)bis(6-oxo-1,6-dihydropyridine-3,4-dicarboxylate) (4au):
white solid; 141 mg; 30% yield; mp 214 – 215 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\)
7.91 (s, 2H), 6.56 (s, 2H), 4.32 (s, 4H), 4.22-4.27 (q, \(J = 7.1\) Hz, 4H), 3.88 (s, 6H),
1.27-1.31 (t, \(J = 7.1\) Hz, 6H); \(^1^3\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 166.4, 162.4, 161.5,
144.9, 143.5, 118.7, 108.4, 61.8, 53.1, 48.8, 14.2; HRMS (Q-TOF, m/z) calcd for C\(_{23}\)H\(_{24}\)N\(_2\)O\(_{10}\)Na [M+Na]\(^+\) 499.1329, found 499.1335.

3-Ethyl 4-methyl
6-oxo-1-phenyl-1,6-dihydropyridine-3,4-dicarboxylate (4av): white solid; 176 mg;
28% yield (2 mmol); mp 123 – 124 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.17 (s, 1H),
7.50 – 7.52 (m, 3H), 7.37 (s, 2H), 6.63 (s, 1H), 4.28 (d, \(J = 4.1\) Hz, 2H), 3.93 (s, 3H),
1.30 (t, J = 12.9 Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 166.6, 162.6, 161.3, 144.0, 139.4, 129.7, 126.3, 119.6, 107.6, 61.6, 53.1, 14.2; HRMS (Q-TOF, m/z) calcd for C$_{16}$H$_{15}$NO$_5$Na [M+Na]$^+$ 324.0848, found 324.0846.

**Trimethyl**

6-oxo-1-phenyl-1,6-dihydropyridine-2,3,4-tricarboxylate (5aa): white solid; 138mg; 83% yield; mp 112 – 113 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.48 (d, J = 8.0 Hz, 3H), 7.25 (d, J = 8.1 Hz, 2H), 6.84 (s, 1H), 3.91 (s, 3H), 3.80 (s, 3H), 3.48 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 165.6, 163.5, 161.2, 160.5, 145.6, 143.1, 136.2, 129.9, 129.3, 128.1, 121.3, 106.6, 53.1, 53.0, 52.9; HRMS (Q-TOF, m/z) calcd for C$_{17}$H$_{15}$NO$_7$Na [M+Na]$^+$ 368.0746, found 368.0740.

**Trimethyl**

6-oxo-1-o-tolyl-1,6-dihydropyridine-2,3,4-tricarboxylate (5ab): white solid; 131.1 mg; 73% yield; mp 113 – 114 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.35-7.37 (dt, J = 8.0 Hz, 3H), 7.27 (d, J = 7.2 Hz, 1H), 6.83 (s, 1H), 3.92 (s, 3H), 3.79 (s, 3H), 3.46 (s, 3H), 2.16 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 165.7, 163.4, 161.1, 159.8, 145.8, 143.4, 136.5, 135.6, 135.4, 131.0, 130.2, 127.9, 126.7, 121.0, 106.3, 53.1, 52.9, 52.8, 17.4; HRMS (Q-TOF, m/z) calcd for C$_{18}$H$_{17}$NO$_7$Na [M+Na]$^+$ 382.0903, found 382.0908.
Trimethyl 6-oxo-1-m-tolyl-1,6-dihydropyridine-2,3,4-tricarboxylate (5ac): white solid; 137.7 mg; 74% yield; mp 134 – 135 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.36 (t, J = 7.7 Hz, 1H), 7.32-7.28 (d, J = 7.7 Hz, 1H), 7.04 – 7.05 (m, 2H), 6.83 (s, 1H), 3.91 (s, 3H), 3.80 (s, 3H), 3.50 (s, 3H), 2.39 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 165.7, 163.6, 161.3, 160.6, 145.6, 143.1, 139.5, 136.1, 129.1, 128.5, 124.9, 121.3, 106.5, 53.1, 53.0, 52.9, 21.2; HRMS (Q-TOF, m/z) calcd for C₁₈H₁₇NO₇Na [M+Na]⁺ 382.0903, found 382.0912.

Trimethyl 6-oxo-1-p-tolyl-1,6-dihydropyridine-2,3,4-tricarboxylate (5ad): white solid; 168.8 mg; 90% yield; mp 124 – 125 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.27-7.29 (d, J = 8.0 Hz, 2H), 7.11-7.13 (d, J = 8.1 Hz, 2H), 6.83 (s, 1H), 3.92 (s, 3H), 3.80 (s, 3H), 3.52 (s, 3H), 2.40 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 165.7, 163.6, 161.4, 160.9, 145.6, 143.2, 140.2, 133.6, 130.1, 127.8, 121.3, 106.6, 53.2 53.1, 53.0, 21.3; HRMS (Q-TOF, m/z) calcd for C₁₈H₁₇NO₇Na [M+Na]⁺ 382.0903, found 382.0906.

Trimethyl 1-(4-fluorophenyl)-6-oxo-1,6-dihydropyridine-2,3,4-tricarboxylate (5ae): white solid; 215.2 mg; 67% yield; mp 148 – 150 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.35 (t, J = 7.7 Hz, 1H), 7.13 (t, J = 7.7 Hz, 1H), 6.86 (s, 1H), 3.90 (s, 3H), 3.81 (s, 3H), 3.52 (s, 3H), 2.37 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 165.8, 163.6, 161.3, 160.8, 145.6, 143.1, 139.5, 136.1, 129.1, 128.5, 124.9, 121.3, 106.5, 53.1, 53.0, 52.9, 21.2; HRMS (Q-TOF, m/z) calcd for C₁₈H₁₇NO₇Na [M+Na]⁺ 382.0903, found 382.0906.
solid; 134.7 mg; 71% yield; mp 160 – 162 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\)
7.23-7.27 (d, \(J = 8.0\) Hz, 2H), 7.17-7.19 (d, \(J = 8.1\) Hz, 2H), 6.81 (s, 1H), 3.92 (s, 3H),
3.81 (s, 3H), 3.55 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 165.6, 164.3, 163.5, 161.8,
161.3, 160.6, 145.6, 143.3, 132.1, 130.3, 130.2, 121.4, 116.7, 116.4, 106.8, 53.28,
53.026, 53.0; \(^{19}\)F NMR (377 MHz, CDCl\(_3\)) \(\delta\) -110.6; HRMS (Q-TOF, m/z) calcd for
C\(_{17}\)H\(_{14}\)FNO\(_7\)Na [M+Na]\(^+\) 386.0652, found 386.0644.

Trimethyl

1-(4-chlorophenyl)-6-oxo-1,6-dihydropyridine-2,3,4-tricarboxylate (5af): white solid; 139.8 mg; 71% yield; mp 150 – 151 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\)
7.45-7.47 (d, \(J = 8.0\) Hz, 2H), 7.21 (d, \(J = 8.1\) Hz, 2H), 6.83 (s, 1H), 3.92 (s, 3H), 3.81
(s, 3H), 3.56 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 165.5, 163.5, 161.2, 160.4, 145.3,
143.3, 136.1, 134.7, 129.7, 129.6, 121.5, 106.8, 53.3, 53.2, 53.0; HRMS (Q-TOF, m/z)
calcd for C\(_{17}\)H\(_{14}\)ClNO\(_7\)Na [M+Na]\(^+\) 402.0356, found 402.0347.

Trimethyl

1-(3-bromophenyl)-6-oxo-1,6-dihydropyridine-2,3,4-tricarboxylate (5ag): white solid; 145.5 mg; 69% yield; mp 151 – 152 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\)
7.61-7.63 (d, \(J = 8.1\) Hz, 1H), 7.39 (s, 1H), 7.45 (t, \(J = 8.0\) Hz, 1H), 7.37 (d, \(J = 8.0\)
Hz, 1H), 6.84 (s, 1H), 3.92 (s, 3H), 3.81 (s, 3H), 3.56 (s, 3H); \(^{13}\)C NMR (100 MHz,
CDCl\(_3\)) \(\delta\) 165.5, 163.4, 162.3, 161.1, 160.3, 145.0, 143.2, 137.3, 131.3, 131.1, 130.4,
127.1, 122.5, 121.6, 107.1, 53.3, 53.2, 53.0; HRMS (Q-TOF, m/z) calcd for 
C_{17}H_{14}BrNO_{7}Na [M+Na]^+ 445.9851, found 445.9858.

Trimethyl 
1-(4-ethoxyphenyl)-6-oxo-1,6-dihydropyridine-2,3,4-tricarboxylate (5ah): white 
solid; 180.7 mg; 89% yield; mp 103 – 104 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 
7.13-7.16 (d, \(J = 8.7\) Hz, 2H), 6.94-6.96 (d, \(J = 8.7\) Hz, 2H), 6.81 (d, \(J = 8.4\) Hz, 1H), 
4.06 – 4.08 (m, 2H), 3.91 (d, \(J = 8.7\) Hz, 3H), 3.80 (d, \(J = 8.1\) Hz, 3H), 3.54 (d, \(J = 
7.5\) Hz, 3H), 1.41 – 1.44 (m, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 165.8, 163.6, 161.4, 
160.9, 159.8, 146.2, 143.2, 129.2, 128.4, 121.1, 115.0, 105.9, 63.8, 53.2, 53.1, 52.9, 
14.7; HRMS (Q-TOF, m/z) calcd for C\(_{19}\)H\(_{19}\)NO\(_8\)Na [M+Na]^+ 412.1008, found 412.1015.

Triethyl 
6-oxo-1-p-tolyl-1,6-dihydropyridine-2,3,4-tricarboxylate (5ai): oil; 126.7 mg; 63% 
yield; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.18-7.20 (d, \(J = 8.0\) Hz, 2H), 7.04-7.06 (d, \(J = 
8.0\) Hz, 2H), 6.75 (s, 1H), 4.26-4.32 (q, \(J = 7.1\) Hz, 2H), 4.15-4.21 (q, \(J = 7.1\) Hz, 2H), 
3.86-3.91 (d, \(J = 21.3\) Hz, 2H), 2.31 (s, 3H), 1.27-1.29 (t, \(J = 7.1\) Hz, 3H), 1.19-1.21 
(d, \(J = 7.2\) Hz, 3H), 0.88-0.92 (t, \(J = 7.1\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 
165.2, 163.2, 160.8, 145.7, 143.6, 140.0, 133.7, 129.9, 128.0, 121.1, 106.8, 62.6, 62.3, 
62.0, 29.6, 21.2, 13.9, 13.8, 13.3; HRMS (Q-TOF, m/z) calcd for C\(_{21}\)H\(_{25}\)NO\(_7\)Na
[M+Na]$^+$ 424.1372, found 424.1378.

Trimethyl

1-(4-nitrophenyl)-6-oxo-1,6-dihydropyridine-2,3,4-tricarboxylate (5aj): white solid; 96.2 mg; 49% yield; mp 145–149 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 8.34-8.36 (d, J = 8.8 Hz, 2H), 7.46-7.48 (d, J = 8.8 Hz, 2H), 6.88 (s, 1H), 3.92 (s, 3H), 3.82 (s, 3H), 3.56 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 165.3, 163.5, 161.1, 160.1, 148.5, 144.2, 143.4, 141.8, 129.7, 124.8, 122.1, 108.4, 53.6, 53.4, 53.3; HRMS (Q-TOF, m/z) calcd for C$_{17}$H$_{14}$N$_2$O$_9$Na [M+Na]$^+$, 413.0597, found. 413.0591

Trimethyl

6-oxo-1-(3-(trifluoromethyl)phenyl)-1,6-dihydropyridine-2,3,4-tricarboxylate (5ak): white solid; 80.5 mg; 39% yield; mp 92–94 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.74 (d, J = 7.9 Hz, 1H), 7.64 (t, J = 7.9 Hz, 1H), 7.54 (s, 1H), 7.50 (d, J = 8.1 Hz, 1H), 6.86 (s, 1H), 3.93 (s, 3H), 3.81 (s, 3H), 3.52 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 165.5, 163.5, 161.2, 160.4, 145.1, 143.5, 137.0, 132.1, 129.9, 125.4, 125.1, 121.8, 107.6, 53.4, 53.3, 53.2; $^{19}$F NMR (377 MHz, CDCl$_3$) δ -63.2; HRMS (Q-TOF, m/z) calcd for C$_{18}$H$_{14}$F$_3$NO$_7$Na [M+Na]$^+$ 436.0620, found 436.0607.

6-1 and 6-2: white solid; $^1$H NMR (400 MHz, CDCl$_3$) δ 9.67 (s, 1H), 7.27 (s, 2H), 7.08 (s, 1H), 6.90 (d, J = 6.6 Hz, 2H), 5.39 (s, 1H), 3.73 (s, 3H), 3.68 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 169.9, 164.9, 148.0, 140.3, 129.2, 124.3, 120.7, 93.6, 52.8,
6-oxo-1-phenyl-1,6-dihydropyridine-2,3,4-tricarboxylate (D-5aa): white solid; 137.1 mg; 79% yield; mp 115–117 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.47 (s, 3.00H), 7.25 (d, $J$ = 4.4 Hz, 2.00H), 6.83 (s, 0.48H), 3.91 (s, 3.00H), 3.80 (s, 3.00H), 3.48 (s, 3.00H).

6-oxo-1-phenyl-1,6-dihydropyridine-2,3,4-tricarboxylate (D’-5aa): white solid; 180 mg (1 mmol); 51% yield; mp 121 – 122 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 6.83 (s, 0.17H), 3.92 (s, 2.99H), 3.80 (s, 3.02H), 3.48 (s, 3H); HRMS (Q-TOF, m/z) calcd for C$_{17}$H$_9$D$_6$NO$_7$Na [M+Na]$^+$ 374.1123, found 374.1112.
$^1$H NMR (400 MHz, in CDCl$_3$)
$^1$H NMR (400 MHz, in $d_6$-Acetone)
$^{13}$C NMR (100 MHz, in d$_6$-Acetone)

Electronic Supplementary Material (ESI) for RSC Advances
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\[ ^{13}\text{C} \text{ NMR (100 MHz, in } \text{d}_6 \text{-Acetone)} \]

4 ac
Electronic Supplementary Material (ESI) for RSC Advances
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$^{13}$C NMR (100 MHz, in d$_5$-Acetone)
$^1$H NMR (400 MHz, in $d_6$-Acetone)
$^{13}$C NMR (100 MHz, in d$_6$-Acetone)
$^{19}$F NMR (377 MHz, in d$_7$Acetone)
$^1H$ NMR (400 MHz, in $d_6$-Acetone)
$^{13}$C NMR (100 MHz, in $d_6$-Acetone)
1H NMR (400 MHz, in d$_6$-Acetone)
\(^{13}\)C NMR (100 MHz, in d\textsubscript{6}-Acetone)
$^1$H NMR (400 MHz, in $d_6$-Acetone)
\[ ^{13}C \text{NMR (100 MHz, in d}_6\text{-Acetone)} \]
Electronic Supplementary Material (ESI) for RSC Advances
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$^1$H NMR (400 MHz, in d$_6$-Acetone)

S43
$^1$H NMR (400 MHz, in $d_6$-acetone)
$^{13}$C NMR (100 MHz, in $d_6$-Acetone)
Electronic Supplementary Material (ESI) for RSC Advances
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^1H NMR (400 MHz, in d_8-Acetone)
$^{13}$C NMR (100 MHz, in $d_6$-Acetone)
$^1$H NMR (400 MHz, in d$_7$-Acetone)
$^1$H NMR (400 MHz, in $d_8$-Acetone)
$^{13}$C NMR (100 MHz, in $d_6$-Acetone)
**Electronic Supplementary Material (ESI) for RSC Advances**

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**13C NMR (100 MHz, d6-DMSO)**

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**S54**
$^1$H NMR (400 MHz, in d$_6$-Acetone)

**4ar**
$^{13}$C NMR (100 MHz, in $d_6$-Acetone)
\[^{13}\text{C} \text{NMR (100 MHz, in d}_6\text{-Acetone)}\]
$^1$H NMR (400 MHz, in CDCl$_3$)
$^{13}$C NMR (100 MHz, in CDCl$_3$)
$^1$H NMR (400 MHz, in CDCl$_3$)
$^{13}$C NMR (100 MHz, in CDCl$_3$)
$^1$H NMR (400 MHz, in CDCl$_3$)
$\text{H} \quad \text{O} \quad \text{O} \quad \text{H}$

13C NMR (100 MHz, in CDCl3)
$^1$H NMR (400 MHz, in CDCl$_3$)
$^{13}$C NMR (100 MHz, in CDCl$_3$)
$^1$H NMR (400 MHz, in CDCl$_3$)
Electronic Supplementary Material (ESI) for RSC Advances
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$^{13}$C NMR (100 MHz, in CDCl$_3$)
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$^13$C NMR (100 MHz, in CDCl$_3$)
$^1$H NMR (400 MHz, in CDCl$_3$)
$^{13}$C NMR (100 MHz, in CDCl$_3$)
13C NMR (100 MHz, in CDCl₃)
$^{19}$F NMR (377 MHz, in CDCl$_3$)
$^1$H NMR (400 MHz, in CDCl$_3$)
$^{13}$C NMR (100 MHz, in CDCl$_3$)
$^{1}$H NMR (400 MHz, in CDCl$_3$)
$^{13}$C NMR (100 MHz, in CDCl$_3$)
1H NMR (400 MHz, in CDCl3)
$^{13}$C NMR (100 MHz, in CDCl$_3$)
$^1$H NMR (400 MHz, in CDCl$_3$)
$^{13}$C NMR (100 MHz, in CDCl$_3$)
$\text{H NMR (400 MHz, in CDCl}_3\text{)}$
Electronic Supplementary Material (ESI) for RSC Advances
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$^{19}$F NMR (377 MHz, in CDCl$_3$)


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$^{13}$C NMR (100 MHz, in CDCl$_3$)
\( ^1H \text{ NMR (400 MHz, in } \text{CDCl}_3) \)
**Elemental Composition Report**

**Single Mass Analysis (displaying only valid results)**
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:
C: 0-100  H: 0-150  N: 1-1  O: 5-5  Na: 1-1

12092637 43 (1.113) AM (Top, 4, Ar, 5000.0, 475.27, 1.00, LS 10); Sm (Mn, 2x1.00); Sb (1.40.00); Cm (43:44)

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Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100  H: 0-150  N: 1-1  O: 5-5  Na: 1-1

12120303 66 (1.666) AM (Top, 4, Ar, 5000.0, 475.27, 1.00, LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (66)

Minimum:  Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
276.0853  276.0848  0.5  1.8  5.5  4.0  C12 H15 N O5 Na
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 50.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1

Minimum: -200.0
Maximum: 5.0 50.0 200.0

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Page 1
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100  H: 0-150  N: 1-1  O: 5-5  Na: 1-1

4ad
12092639 31 (0.810) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00 ); Cm (31:32)

Minimum:                  Maximum:                -200.0
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288.0855  288.0848  0.7  2.4  6.5  34.7  Cl3 H15 N O5 Na
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 50.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100  H: 0-150  N: 1-1  O: 5-5  Na: 1-1

[^1] 12120305 58 (1.476) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00 ); Cm (57:64)
304.1153

Minimum:
Maximum:
Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
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Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
12 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100    H: 0-150    N: 1-1    O: 5-5    Na: 1-1

Minimum: 5.0  5.0  -200.0
Maximun: 5.0  5.0  200.0

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Elemental Composition Report

Single Mass Analysis (displaying only valid results)
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Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100  H: 0-150  N: 1-1  O: 5-5  F: 1-1  Na: 1-1

4 a q
1212.00306 53 (1.338) AM (Top, Ar, 5000.0, 475.27, 1.00, LS 10); Sm (Mn, 2x1.00); Sb (1,40.00 ); Cm (52:78)

Minimum:
Maximum:
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356.0897  356.0910  -1.3  -3.7  9.5  14.3  C17 H16 N O5 F Na
 Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100  H: 0-150  N: 1-1  O: 5-5  Na: 1-1  Cl: 1-1

4ar
12092641 35 (0.881) AM (Top, 4, Ar, 5000.0, 475.27, 1.00, LS 10); Sm (Mn, 2x1.00); Sb (1.40.00); Cm (35:38)

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Maximum:
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Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100  H: 0-150  N: 1-1  O: 5-5  Na: 1-1  Cl: 1-1

12092642 12 (0.289) AM (Top, Ar, 5000.0, 475.27, 1.00, LS 10): Sm (Mn, 2x1.00); Sb (1,40.00); Cm (12:13)
372.0631

<table>
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Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:
C: 0-100  H: 0-150  N: 1-1  O: 6-6  Na: 1-1

12092643 56 (1.450) AM (Top,4, Ar,5000.0,175.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (55:56)

Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
368.1100  368.1110  -1.0  -2.7  9.5  1.3  C18 H19 N O6 Na
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100   H: 0-150   N: 1-1   O: 6-6   Na: 1-1

4aK
12092644 48 (1.205) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00 ); Cm (48:49)

Minimum:  
Maximum:  5.0  5.0  200.0

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<td>368.1110</td>
<td>-1.0</td>
<td>-2.7</td>
<td>9.5</td>
<td>2.4</td>
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**Elemental Composition Report**

**Single Mass Analysis (displaying only valid results)**
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100  H: 0-150  N: 1-1  O: 6-6  Na: 1-1

12092645 67 (1.741) AM (Top,4, Ar,5000,0,475,27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1.40.00 ); Cm (66:67)

Minimum:
Maximum:  5.0  5.0  -200.0  200.0

Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
368.1098  368.1110  -1.2  -3.3  9.5  36.2  C18 H19 N O6 Na
### Elemental Composition Report

**Single Mass Analysis (displaying only valid results)**

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<th>DBE: min = -200.0, max = 200.0</th>
</tr>
</thead>
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Selected filters: None

Monoisotopic Mass, Even Electron Ions

13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

- C: 0-100
- H: 0-150
- N: 1-1
- O: 5-5
- Na: 1-1

**Calc. Mass**

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**Elemental Composition Report**

**Single Mass Analysis (displaying only valid results)**
Tolerance = 5.0 PPM  
DBE: min = -200.0, max = 200.0  
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100  
H: 0-150  
N: 1-1  
O: 5-5  
Na: 1-1

Minimum:  
Maximum: 5.0  
5.0  
-200.0  
200.0

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<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>i-FIT</th>
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<td>352.1161</td>
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<td>4.3</td>
<td>9.5</td>
<td>16.6</td>
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**Elemental Composition Report**

**Single Mass Analysis (displaying only valid results)**
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:
C: 0-100  H: 0-150  N: 1-1  O: 5-5  Na: 1-1

4ar
12092702 72 (1.829) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00 ); Cm (65:77)

| Minimum: | 5.0 | 5.0 | -200.0 |
| Maximum: | 200.0 |
| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
| 352.1167 | 352.1161 | 0.6 | 1.7 | 9.5 | 35.7 | C18 H19 N O5 Na |
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
12 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100  H: 0-150  N: 1-1  O: 5-5  Na: 1-1

18:01:47
1: TOF MS ES+
1.10e3

Minimum:
Maximum:
Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
366.1311  366.1317  -0.6  -1.6  9.5  6.7  C19 H21 N O5 Na
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 50.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1

12092704 47 (0.967) AM (Top, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00 ); Cm (35.38)

Minimum:
Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
338.1020  338.1004  1.6  4.7  9.5  4.6  C17 H17 N O5 Na
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 50.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100  H: 0-150  N: 1-1  O: 5-5  Na: 1-1

12092705 42 (1.058) AM (Top,4,Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00 ); Cm (41:42)

Minimum: -200.0
Maximum: 

Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
324.0843  324.0840  -0.5  -1.5  9.5  6.6  C16 H15 N O5 Na
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 50.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100  H: 0-150  N: 1-1  O: 5-5  Na: 1-1

Minimum:  Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
Maximum:  5.0  50.0  -200.0

388.1158  388.1161  -0.3  -0.8  12.5  8.4  C21 H19 N O5 Na
**Elemental Composition Report**

**Single Mass Analysis (displaying only valid results)**
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:
C: 0-120  H: 0-150  N: 2-2  O: 10-10  Na: 1-1

13032812 21 (0.390) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (19.25)
11:22:07
T: TOF MS ES+
2.45e3

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<td>0.6</td>
<td>C24 H28 N2 O10 Na</td>
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</table>
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 50.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 2-2 O: 10-10 Na: 1-1

Minimum:
Maximum:
Mass Calc. Mass mDa PPM DBE i-FIT Formula
499.1335 499.1329 0.6 1.2 11.5 14.7 C22 H24 N2 O10 Na
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120  H: 0-150  N: 1-1  O: 5-5  Na: 1-1

13032814 29 (0.543) AM (Cen, 2, 80.00, Ht, 5000.0, 0.00, 1.00); Sm (Mn, 2x1.00); Cm (24.34)

Minimum:
5.0  5.0  -200.0
Maximum:
5.0  200.0

Mass Calc. Mass mDa PPM DBE i-FIT Formula
324.0846 324.0848 -0.2 -0.6 9.5 0.1  C16 H15 N O5 Na
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:
C: 0-120  H: 0-150  N: 1-1  O: 7-7  Na: 1-1

COX-1
13032802 51 (0.952) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Crm (51:70)
368.0740

Minimum:  S.0  5.0  -200.0
Maximum:  200.0

Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
368.0740  368.0746  -0.6  -1.6  10.5  28.2  Cl7 H15 N O7 Na

m/z  811.0461  838.1450
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(s) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120  H: 0-150  N: 1-1  O: 7-7  Na: 1-1

Minimum: 5.0  Maximum: 5.0

Mass    Calc. Mass    mDa    PPM    DBE    i-FIT    Formula
382.0908 382.0903   0.5   1.3    10.5   1.9    C18 H17 N O7 Na
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:
C: 0-120  H: 0-150  N: 1-1  O: 7-7  Na: 1-1

COX-3
13032804 57 (1.059) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (50:58)
382.0912

100%

% 189.0738 268.1175 340.2903 383.0963 384.0970 496.1207 526.1218 631.1759 663.1956 741.1734 743.1767 855.1949 m/z

Minimum:  5.0  5.0  -200.0
Maximum:  200.0

Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
382.0912  382.0903  0.9  2.4  10.5  0.2  C18 H17 N O7 Na
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120  H: 0-150  N: 1-1  O: 7-7  Na: 1-1

C 13032805 36 (0.669) AM (Cen,2, 80.00, Ht,5000,0.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (28:36)

Minimum: 382.0906
Maximum: 382.0903

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<th>Mass</th>
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Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:

COX-5
13032806 67 (1.249) AM (Cen.2, 80.00, Ht,5000.0,0,00,1.00); Sm (Mn, 2x1.00); Cm (53.70)

Minimum:
Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
386.0644  386.0652  -0.8  -2.1  10.5  0.2  C17 H14 N O7 F Na
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120  H: 0-150  N: 1-1  O: 7-7  Na: 1-1  Cl: 1-1

COX-6
13032807 48 (0.891) AM (Cen, 2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (41:52)
402.0347

Minimum:
5.0
5.0
-200.0

Maximum:

Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
402.0347  402.0356  -0.9  -2.2  10.5  0.4  C17 H14 N O7 Na Cl

Page 1
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotropic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120  H: 0-150  N: 1-1  O: 7-7  Na: 1-1  Br: 1-1

Minimum:  5.0  5.0  -200.0
Maximum:  200.0

Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
445.9858  445.9851  0.7  1.6  10.5  0.1  C17 H14 N O7 Na Br
Elemental Composition Report

**Single Mass Analysis (displaying only valid results)**

Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-120  H: 0-150  N: 1-1  O: 8-8  Na: 1-1

COX-8
13032809 32 (0.594) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (22:33)

11:02:25
1: TOF MS ES+-
2.41e3

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<td>C19 H19 N O8 Na</td>
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**Elemental Composition Report**

- **Single Mass Analysis (displaying only valid results)**
  - Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
  - Selected filters: None

**Monoisotopic Mass, Even Electron Ions**
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

**Elements Used**:
- C: 0-120
- H: 0-150
- N: 1-1
- O: 7-7
- Na: 1-1

CQX-9
13032810 59 (1.098) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (57.69)

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<td>10.5</td>
<td>0.1</td>
<td>C21 H23 N O7 Na</td>
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Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 150.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
4 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-150  H: 0-150  N: 2-2  O: 9-9  Na: 1-1

COX-19
13042008 37 (0.614) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x3.00); Cm (27.39)

Minimum:
Mass     Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
413.0591 413.0597  -0.6  -1.5 11.5  1.5  C17 H14 N2 09 Na
Elemental Composition Report

Single Mass Analysis
Tolerance = 50.0 PPM / DEB: min = -1.5, max = 150.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
4 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-150  H: 0-150  N: 1-1  O: 7-7  F: 3-3  Na: 1-1

COX-1
13042007 2 (0.033) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x3.00); Cm (1:4)

Minimum:
Maximum:
Mass Calc. Mass mDa PPM DBE i-FIT Formula
436.0607 436.0620  -1.3  -3.0  10.5  5.3  C18 H14 N 07 P3 Na
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
20 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120  H: 0-120  2H: 6-6  N: 1-1  O: 7-7  Na: 1-1

13032811 45 (0.837) AM (Cen.2.  80.00.  Ht.5000.0.0.00.1.00); Sm (Mn. 2x1.00); Cm (41:47)

Minimum: 5.0  5.0  -200.0
Maximum: 200.0

Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
374.1112  374.1123  -1.1  -2.9  10.5  188757.8  C17  1H9  2H6  N  07  Na
We used the slow evaporation method to grow the crystals of 4aq. We first prepared a saturated solution of compound 4aq, employing petroleum ether/ethyl acetate (4:1, v/v) as the solvent. Then the solution was transferred to a clean bottle and covered, with a few small holes in the cover. After that the bottle was placed in a quiet out of the way place. A few days later, the crystals of 4aq were obtained.

CCDC (4aq) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.