Supporting Information

Oxidative cleavage of C-2/C-3 bond in isatin using (diacetoxyiodo)benzene: A facile synthesis of carbamates of alkyl anthranilates

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5. Copies of $^1$H and $^{13}$C NMR spectra
1. General Remarks

All reagents and solvents were purchased and used without further purification. $^1$H NMR and $^{13}$C NMR spectra were measured on Bruker spectrometer ($^1$H 400 MHz and $^{13}$C 100 MHz), using CDCl$_3$ as the solvent with tetramethylsilane (TMS) as the internal standard at room temperature. All chemical shifts (δ) are given in parts per million (ppm) and $J$ values are given in hertz (Hz). IR spectra were recorded on a Bruker FTIR-αE. LCMS spectra were recorded using Shimadzu 2010 (single quadrupole). Melting points were determined in a capillary tube and are uncorrected. Thin-layer chromatography (TLC) was carried out on SiO$_2$ (silica gel 60F254, Merck), and the spots were located with ultraviolet (UV) light. Flash chromatography was carried out on SiO$_2$ (silica gel 60, Qualigens, 100 to 200-mesh ASTM). Drying of organic extracts after workup of reactions was performed over anhydrous Na$_2$SO$_4$. Evaporation of solvents was accomplished with a Buchi rotatory evaporator.

3,5-Dibromoisatin, 5-nitroisatin and $N$-acetyl isatin were obtained by bromination, nitration and $N$-acetylation of isatin respectively using the literature procedure.$^1$

2. General experimental procedure for the synthesis of carbamates of alkyl anthranilates: To the stirred solution of isatin (1 mmol) in an appropriate alcohol (5 mL), (diacetoxyiodo)benzene (1.7 mmol) was added. The reaction mixture was stirred for 9-24 h at room temperature. The progress of the reaction was monitored by TLC. After completion of reaction, the solvent was evaporated on rotatory vacuum evaporator and the crude product was purified by column chromatography using petroleum ether and ethyl acetate to afford 2a-q in moderate yields.

General experimental procedure for the synthesis of alkyl 2-acetamidobenzoate from $N$-acetyl isatin: To the stirred solution of $N$-acetyl isatin (1 mmol) in an appropriate alcohol (5 mL), (diacetoxyiodo)benzene (2 mmol) was added. The reaction mixture was stirred for 48 h at room temperature. The progress of the reaction was monitored by TLC. After completion of reaction, the solvent was evaporated on rotatory vacuum evaporator and the crude product was purified by column chromatography using petroleum ether and ethyl acetate to afford 4a-g in moderate yields.
3. Spectral data:
Methyl 2-(methoxycarbonylamino)benzoate (2a): 

Yield: 0.1651 g (79%), mp: 121 °C.

$^1$H NMR 400 MHz (CDCl$_3$): $\delta$ 3.78 (3H, s, CH$_3$), 3.90 (3H, s, CH$_3$), 7.01 (1H, td, J = 1.1 Hz, 8.2 Hz, ArH), 7.52 (1H, td, J = 1.6 Hz, 8.4 Hz, ArH), 7.99 (1H, dd, J = 1.6 Hz, 8.0 Hz, ArH), 8.43 (1H, d, J = 8.5 Hz, ArH), 10.5 (1H, brs, NH).

$^{13}$C NMR 100 MHz (CDCl$_3$): $\delta$ 52.2, 114.4, 118.7, 121.5, 130.8, 134.5, 141.7, 154.1, 168.5.

IR (cm$^{-1}$): 3261, 1733, 1691, 1591, 1535, 1452, 1261, 1217, 1061, 752.

MS (ESI): m/z (M)$^+$ calculated for C$_{10}$H$_{11}$NO$_4$: 209.0688, Found [M + 23]: 232.

Ethyl 2-(ethoxycarbonylamino)benzoate (2b):

Yield: 0.1453 g (64%), mp: 139 °C.

$^1$H NMR 400 MHz (CDCl$_3$): $\delta$ 1.32 (3H, t, J = 7.1 Hz, CH$_3$), 1.41 (3H, t, J = 7.1 Hz, CH$_3$), 4.23 (2H, q, J = 7.1 Hz, CH$_2$), 4.30 (2H, q, J = 7.1 Hz, CH$_2$), 7.01 (1H, td, J = 1 Hz, 8.1 Hz, ArH), 7.52 (1H, td, J = 1.6 Hz, 8.6 Hz, ArH), 8.02 (1H, dd, J = 1.6 Hz, 8 Hz, ArH), 8.44 (1H, dd, J = 0.5 Hz, 8.4 Hz, ArH), 10.51 (1H, brs, NH).

$^{13}$C NMR 100 MHz (CDCl$_3$): $\delta$ 14.2, 14.5, 61.1, 61.2, 114.4, 118.7, 121.3, 130.8, 134.4, 141.9, 153.7, 168.1.

IR (cm$^{-1}$): 3251, 1722, 1690, 1591, 1535, 1452, 1261, 1217, 1061, 753.

MS (ESI): m/z (M)$^+$ calculated for C$_{12}$H$_{15}$NO$_4$: 237.1001, Found [M + 23]: 260.

Propyl 2-(propoxycarbonylamino)benzoate (2c):

Yield: 0.1462 g (55%), Pale yellow oil.

$^1$H NMR 300 MHz (CDCl$_3$): $\delta$ 0.983 (3H, t, J = 7.8 Hz, CH$_3$), 1.03 (3H, t, J = 7.5 Hz, CH$_3$), 1.67-1.84 (4H, m, 2CH$_2$), 4.12 (2H, t, J = 9.2 Hz, OCH$_2$), 4.27 (2H, t, J = 8.8 Hz, OCH$_2$),
7.02 (1H, t, J = 10.4 Hz, ArH), 7.54 (1H, t, J = 9.6 Hz, ArH), 8.02 (1H, dd, J = 2 Hz, 10.8 Hz, ArH), 8.45 (1H, d, J = 11.2 Hz, ArH), 10.56 (1H, brs, NH).

$^1$C NMR 75 MHz (CDCl$_3$): 10.4, 10.6, 22.1, 22.3, 66.9 (2C), 114.8, 118.8, 121.4, 130.9, 134.5, 142, 153.9, 168.2.

2,2,2-Trifluoroethyl 2-((2,2,2-trifluoroethoxy)carbonylamino)benzoate (2d):

![Structure of 2d](image)

Yield: 0.2130 g (61%), mp 82 °C.

$^1$H NMR 400 MHz (CDCl$_3$): δ 4.58 (2H, q, J = 8.4 Hz, CH$_2$), 4.71 (2H, q, J = 8.2 Hz, CH$_2$), 7.14 (1H, td, J = 1 Hz, 8.1 Hz, ArH), 7.62 (1H, td, J = 1.6 Hz, 8.7 Hz, ArH), 8.08 (1H, dd, J = 1.6 Hz, 8.0 Hz, ArH), 8.42 (1H, d, J = 8.0 Hz, ArH), 10.40 (1H, brs, NH).

$^1$C NMR 100 MHz (CDCl$_3$): δ 60.6 (q, $^2$J$_{CF}$ = 44 Hz), 61.3 (q, $^2$J$_{CF}$ = 44 Hz), 113.4, 119.1, 122.8 (q, $^1$J$_{CF}$ = 276 Hz), 122.9 (q, $^1$J$_{CF}$ = 276 Hz), 131.1, 135.7, 141.3, 151.4, 166.2.

IR (cm$^{-1}$): 3287, 1741, 1700, 1592, 1529, 1450, 1277, 1157, 1077, 751.

MS (ESI): m/z (M)$^+$ calculated for C$_{12}$H$_9$F$_6$NO$_4$: 345.0436, Found [M + NH$_4^+$]: 363.

2-Ethoxyethyl 2-((2-ethoxyethoxy)carbonylamino)benzoate (2e):

![Structure of 2e](image)

Yield: 0.2123 g (65%), Yellow oil.

$^1$H NMR 300 MHz (CDCl$_3$): δ 1.25 (6H, t, J = 9.2 Hz, 2CH$_3$), 3.54-3.59 (4H, m, 2OCH$_2$), 3.68 (2H, t, J = 6.4 Hz, OCH$_2$), 4.33 (2H, t, J = 6.4 Hz, OCH$_2$), 4.45 (2H, t, J = 6.4 Hz, OCH$_2$), 7.05 (1H, t, J = 9.6 Hz, ArH), 7.52 (1H, td, J = 2 Hz, 9.6 Hz , ArH), 8.05 (1H, dd, J = 2 Hz, 10.8 Hz, ArH), 8.44 (1H, d, J = 11.2 Hz, ArH), 10.46 (1H, brs, NH).

$^1$C NMR 75 MHz (CDCl$_3$): δ 15.2, 19.5, 64, 64.5, 66.7, 66.8, 68.6, 69.9, 114.7, 118.9, 121.6, 131.1, 134.6, 141.7, 153.6, 168.

IR (cm$^{-1}$): 3304, 1736, 1689, 1525, 1450, 1385, 1238, 1211, 1260, 752.

Methyl 5-fluoro-2-(methoxycarbonylamino)benzoate (2f):

![Structure of 2f](image)

Yield: 0.1756 g (77%), mp: 98 °C.
$^1$H NMR 400 MHz (CDCl$_3$): δ 3.78 (3H, s, CH$_3$), 3.92 (3H, s, CH$_3$), 7.22-7.27 (1H, m, ArH), 7.66 (1H, dd, $J = 3.1$ Hz, 9.2 Hz, ArH), 8.42 (1H, dd, $J = 5.0$ Hz, 9.3 Hz ArH), 10.41 (1H, brs, NH).

$^{13}$C NMR 100 MHz (CDCl$_3$): δ 52.3, 52.5, 115.5 (d, $^3J_{C-F} = 6$ Hz), 116.6 (d, $^2J_{C-F} = 24$ Hz), 120.5 (d, $^3J_{C-F} = 7$ Hz), 121.6 (d, $^2J_{C-F} = 23$ Hz), 138.1 (d, $^4J_{C-F} = 2$ Hz), 154.1, 158.3, (d, $^1J_{C-F} = 241$ Hz), 167.4 (d, $^4J_{C-F} = 2$ Hz).

IR (cm$^{-1}$): 3260, 1733, 1689, 1601, 1427, 1255, 1214, 1056.

MS (ESI): m/z (M$^+$) calculated for C$_{10}$H$_{10}$FNO$_4$: 227.0594, Found [M + 23]: 250.

**Methyl 5-chloro-2-(methoxycarbonylamino)benzoate (2g):**

![Methyl 5-chloro-2-(methoxycarbonylamino)benzoate](image)

Yield: 0.1905 g (78%), mp: 122 °C.

$^1$H NMR 400 MHz (CDCl$_3$): δ 3.78 (3H, s, CH$_3$), 3.92 (3H, s, CH$_3$), 7.46 (1H, dd, $J = 2.6$ Hz 9 Hz , ArH), 7.95 (1H, d, $J = 2.6$ Hz, ArH), 8.40 (1H, d, $J = 9.0$ Hz, ArH), 10.41 (1H, s, NH).

$^{13}$C NMR 100 MHz (CDCl$_3$): δ 52.4, 52.5, 115.6, 120.2, 126.6, 130.3, 134.4, 140.3, 153.9, 167.4.

IR (cm$^{-1}$): 3251, 1730, 1686, 1587, 1513, 1447, 1281, 1213.

MS (ESI): m/z (M$^+$) calculated for C$_{10}$H$_{10}$FNO$_4$: 243.0298, Found [M + 23]: 266.

**Ethyl 5-chloro-2-(ethoxycarbonylamino)benzoate (2h):**

![Ethyl 5-chloro-2-(ethoxycarbonylamino)benzoate](image)

Yield: 0.1676 g (61%), mp: 84 °C.

$^1$H NMR 400 MHz (CDCl$_3$): δ 1.32 (3H, t, $J = 7.1$ Hz, CH$_3$), 1.41 (3H, t, $J = 7.1$ Hz, CH$_3$), 4.22 (2H, q, $J = 7.1$ Hz, CH$_2$), 4.38 (2H, q, $J = 7.1$ Hz, CH$_2$), 7.46 (1H, dd, $J = 2.6$ Hz, 9Hz, ArH), 7.97 (1H, d, $J = 2.6$ Hz, ArH), 8.42 (1H, d, $J = 9.1$ Hz, ArH), 10.43 (1H, brs, NH).

$^{13}$C NMR 100 MHz (CDCl$_3$): δ 14.1, 14.4, 61.3, 61.7, 115.9, 120.2, 126.4, 130.3, 134.3, 140.5, 153.5, 167.0.

IR (cm$^{-1}$): 3233, 1727, 1694, 1588, 1516, 1458, 1237, 1211, 1053.

MS (ESI): m/z (M$^+$) for C$_{12}$H$_{14}$ClNO$_4$: 271.0611, Found [M + 1] and [M + 23]: 272 and 294.
Methyl 5-bromo-2-(methoxycarbonylamino)benzoate (2i)\(^2\):

![Structure of 2i]

Yield: 0.2144 g (74\%), mp: 120 °C.

\(^1\)H NMR 400 MHz (CDCl\(_3\)): \(\delta\) 3.78 (3H, s, CH\(_3\)), 3.92 (3H, s, CH\(_3\)), 7.27 (1H, dd, \(J = 2.4\) Hz, 9 Hz, ArH), 8.10 (1H, d, \(J = 2.4\) Hz, ArH), 8.36 (1H, d, \(J = 9\) Hz, ArH), 10.42 (1H, brs, NH).

\(^13\)C NMR 100 MHz (CDCl\(_3\)): \(\delta\) 52.4, 52.5, 113.8, 116.0, 120.5, 133.3, 137.2, 140.8, 153.8, 167.3.

IR (cm\(^{-1}\)): 3255, 1737, 1687, 1586, 1525, 1428, 1240, 1214, 1062, 785.

MS (ESI): m/z (M)\(^+\) calculated for C\(_{10}\)H\(_{10}\)BrNO\(_4\): 288.9793, Found [M + 23]: 311.

Ethyl 5-bromo-2-(ethoxycarbonylamino) benzoate (2j):

![Structure of 2j]

Yield: 0.5709 g (57\%), mp: 88 °C.

\(^1\)H NMR 400 MHz (CDCl\(_3\)): \(\delta\) 1.32 (3H, t, \(J = 7.1\) Hz, CH\(_3\)), 1.41 (3H, t, \(J = 7.1\) Hz, CH\(_3\)), 4.23 (2H, q, \(J = 7.1\) Hz, CH\(_2\)), 4.39 (2H, q, \(J = 7.1\) Hz, CH\(_2\)), 7.60 (1H, dd, \(J = 2.4\) Hz, 9Hz, ArH), 8.12 (1H, d, \(J = 2.4\) Hz, ArH), 8.36 (1H, d, \(J = 9.0\) Hz, ArH), 10.44 (1H, brs, NH).

\(^13\)C NMR 100 MHz (CDCl\(_3\)): \(\delta\) 14.1, 14.4, 61.3, 61.7, 113.6, 116.2, 120.5, 133.2, 137.1, 141, 153.5, 166.9.

IR (cm\(^{-1}\)): 3239, 1730, 1693, 1586, 1511, 1369, 1246, 1212, 1053, 840.

MS (ESI): m/z (M\(^+\)) Calculated for C\(_{12}\)H\(_{14}\)BrNO\(_4\): 315.0106, Found [M + 23]: 338.

Methyl 3,5-dibromo-2-(methoxycarbonylamino)benzoate (2k):

![Structure of 2k]

Yield: 0.2818 g (77\%), mp: 104 °C.

\(^1\)H NMR 400 MHz (CDCl\(_3\)): \(\delta\) 3.77 (3H, s, CH\(_3\)), 3.90 (3H, s, CH\(_3\)), 7.55 (1H, brs, NH), 7.89 (1H, d, \(J = 2.2\) Hz, ArH), 7.94 (1H, d, \(J = 2.2\) Hz, ArH).

\(^13\)C NMR 100 MHz (CDCl\(_3\)): \(\delta\) 52.8, 53.1, 118.5, 121.5, 128, 132.6, 135.1, 138.8, 153.9, 165.4.
IR (cm\(^{-1}\)): 3255, 1729, 1706, 1553, 1512, 1434, 1260, 1228, 1062.

(ESI): m/z (M\(^+\)) Calculated for C\(_{10}\)H\(_9\)Br\(_2\)NO\(_4\): 366.8898, Found [M+ 23]: 390.

**Ethyl 3,5-dibromo-2-(ethoxycarbonylamino)benzoate (2l):**

![Chemical Structure](image)

Yield: 0.2233 g (56%), mp: 59 °C.

\(^1\)H NMR 400 MHz (CDCl\(_3\)): \(\delta\) 1.28 (3H, t, \(J = 7.1\) Hz, CH\(_3\)), 1.36 (3H, t, \(J = 7.1\) Hz, CH\(_3\)), 4.21 (2H, q, \(J = 7.1\) Hz, CH\(_2\)), 4.35 (2H, q, \(J = 7.1\) Hz, CH\(_2\)), 7.49 (1H, brs, NH), 7.88 (1H, d, \(J = 2.3\) Hz, ArH), 7.94 (1H, d, \(J = 2.3\) Hz, ArH).

\(^{13}\)C NMR 100 MHz (CDCl\(_3\)): \(\delta\) 14.1, 14.4, 62, 62.1, 118.3, 121.4, 128.3, 129.4, 132.6, 135.1, 138.7, 153.5, 165.

IR (cm\(^{-1}\)): 3240, 1698, 1512, 1450, 1382, 1255, 1190, 1063.

MS (ESI): m/z (M\(^+\)) calculated for C\(_{12}\)H\(_{13}\)Br\(_2\)NO\(_4\): 394.9211 Found [M + 23]: 418.

**Methyl 2-(methoxycarbonylamino)-5-nitrobenzoate (2m):**

![Chemical Structure](image)

Yield: 0.1956 g (77%), mp: 158 °C.

\(^1\)H NMR 400 MHz (CDCl\(_3\)): \(\delta\) 3.84 (3H, s, CH\(_3\)), 4.00 (3H, s, CH\(_3\)), 8.34 (1H, dd, \(J = 2.7\)Hz, 9.4 Hz, ArH), 8.65 (1H, d, \(J = 9.4\) Hz, ArH), 8.89 (1H, d, \(J = 2.7\) Hz, ArH), 10.8 (1H, brs, NH).

\(^{13}\)C NMR 100 MHz (CDCl\(_3\)): \(\delta\) 52.9, 53.0, 114.1, 118.8, 127, 129.3, 141.2, 146.9, 153.5, 167.

IR (cm\(^{-1}\)): 3260, 1752, 1690, 1590, 1512, 1438, 1254, 1209, 1054.

**Ethyl 2-(ethoxycarbonylamino)-5-nitrobenzoate (2n):**

![Chemical Structure](image)

Yield: 0.1546 g (55%), mp: 70 °C.

\(^1\)H NMR 400 MHz (CDCl\(_3\)): \(\delta\) 1.35 (3H, t, \(J = 7.1\) Hz, CH\(_3\)), \(\delta\) 1.46 (3H, t, \(J = 7.1\) Hz, CH\(_3\)), 4.28 (2H, q, \(J = 7.1\) Hz, CH\(_2\)), 4.45 (2H, q, \(J = 7.1\) Hz, CH\(_2\)), 8.35 (1H, dd, \(J = 2.8\) Hz, 9.4 Hz, ArH), 8.66 (1H, d, \(J = 9.4\) Hz, ArH), 8.91 (1H, d, \(J = 2.7\) Hz, ArH), 10.89 (1H, brs, NH).
$^{13}$C NMR 100 MHz (CDCl$_3$): $\delta$ 14.4(2C), 62.0, 62.4, 114.3, 118.8, 127.0, 129.2, 140, 147.2, 153.1, 166.6.
IR (cm$^{-1}$): 3253, 1734, 1682, 1584, 1505, 1462, 1243, 1202, 1003.
MS (ESI): m/z (M$^+$) calculated for C$_{12}$H$_{14}$N$_2$O$_6$: 282.0852 Found [M+1] and [M+18]: 283 and 305.

**Methyl 2-(methoxycarbonylamino)-5-methylbenzoate (2o):**

Yield: 0.0961 g (43%), mp 102 °C.

$^1$H NMR 400 MHz (CDCl$_3$): $\delta$ 2.30 (3H, s, CH$_3$), 3.77 (3H, s, CH$_3$), 3.89 (3H, s, CH$_3$), 7.33 (1H, dd, $J = 2.1$ Hz, 8.5 Hz, ArH), 7.78 (1H, d, $J = 2.0$ Hz, ArH), 8.29 (1H, d, $J = 8.6$ Hz, ArH), 10.35 (1H, brs, NH).

$^{13}$C NMR 100 MHz (CDCl$_3$): $\delta$ 20.5, 52.1 (2C), 114.4, 118.8, 130.7, 130.8, 135.2, 139.5, 153.7, 168.1.
IR (cm$^{-1}$): 3303, 1727, 1687, 1591, 1523, 1470, 1241, 1206, 747.

**Ethyl 2-(ethoxycarbonylamino)-5-methylbenzoate (2p):**

Yield: 0.0942 g (37%), Pale yellow oil.

$^1$H NMR 400 MHz (CDCl$_3$): $\delta$ 1.31 (3H, t, $J = 7.1$ Hz, CH$_3$), 1.41 (3H, t, $J = 7.1$ Hz, CH$_3$), 2.31 (3H, s, CH$_3$), 4.22 (2H, q, $J = 7.0$ Hz, CH$_2$), 4.36 (2H, q, $J = 7.1$ Hz, CH$_2$), 7.32 (1H, dd, $J = 2.0$ Hz, 8.6 Hz, ArH), 7.80 (1H, d, $J = 1.8$ Hz, ArH), 8.31 (1H, d, $J = 8.6$ Hz, ArH), 10.36 (1H, brs, NH).

$^{13}$C NMR 100 MHz (CDCl$_3$): $\delta$ 14.2, 14.5, 20.5, 22.0, 61.0, 61.2, 114.6, 118.8, 130.7, 130.8, 135.2, 139.5, 153.7, 168.1.
IR (cm$^{-1}$): 3303, 1727, 1687, 1591, 1523, 1470, 1241, 1206, 747.

**2,2,2-Trifluoroethyl 5-methyl-2-((2,2,2-trifluoroethoxy)carbonylamino)benzoate (2q):**

Yield: 0.112 g (31%), Pale yellow oil.
\[ ^1 \text{H NMR} \ 400 \text{ MHz (CDCl}_3 \text{):} \delta \ 2.36 \ (3 \text{H, s, CH}_3), 4.56 \ (2 \text{H, q, } J=8.4 \text{ Hz, CH}_2), 4.70 \ (2 \text{H, q, } J=8.2 \text{ Hz, CH}_2), 7.43 \ (1 \text{H, dd, } J=1.9 \text{ Hz, 8.6 Hz, ArH}) 7.84 \ (1 \text{H, d, } J=1.7 \text{ Hz, ArH}), 8.29 \ (1 \text{H, d, } J=8.6 \text{ Hz, ArH}), 10.27 \ (1 \text{H, brs, NH}). \]

IR (cm\(^{-1}\)): 3290, 1751, 1707, 1596, 1539, 1457, 1286, 1167, 785.

**Methyl 2-acetamidobenzoate (4a):**

![Methyl 2-acetamidobenzoate (4a)](image)

Yield: 0.1325 g (68\%), mp: 94 °C.

\[ ^1 \text{H NMR} \ 400 \text{ MHz (CDCl}_3 \text{):} \delta \ 2.23 \ (3 \text{H, s, CH}_3), 3.92 \ (3 \text{H, s, OCH}_3), 7.07 \ (1 \text{H, td, } J=1.2 \text{ Hz, 8.2 Hz, ArH}), 7.53 \ (1 \text{H, td, } J=1.6 \text{ Hz, 8.6 Hz, ArH}), 8.01 \ (1 \text{H, dd, } J=1.6 \text{ Hz, 8 Hz, ArH}), 8.69 \ (1 \text{H, dd, } J=0.76 \text{ Hz, 8.6 Hz, ArH}), 11.04 \ (1 \text{H, brs, NH}). \]

\[ ^{13} \text{C NMR} \ 100 \text{ MHz (CDCl}_3 \text{):} \delta \ 25.4, 52.3, 114.7, 120.3, 122.4, 130.7, 134.6, 141.6, 168.7, 169. \]

IR (cm\(^{-1}\)): 3270, 2921, 2853, 1687, 1584, 1450, 1367, 1260, 1080, 756.

**Ethyl 2-acetamidobenzoate (4b):**

![Ethyl 2-acetamidobenzoate (4b)](image)

Yield: 0.1296 g (62\%), mp: 62 °C.

\[ ^1 \text{H NMR} \ 400 \text{ MHz (CDCl}_3 \text{):} \delta \ 1.41 \ (3 \text{H, t, } J=7.1 \text{ Hz, CH}_3), 2.23 \ (3 \text{H, s, CH}_3), 4.37 \ (3 \text{H, s, } J=7.1 \text{ Hz, CH}_3), 7.06 \ (1 \text{H, td, } J=1.1 \text{ Hz, 8.2 Hz, ArH}), 7.52 \ (1 \text{H, td, } J=1.6 \text{ Hz, 8.7 Hz, ArH}), 8.03 \ (1 \text{H, dd, } J=1.6 \text{ Hz, 8 Hz, ArH}), 8.69 \ (1 \text{H, dd, } J=1.6 \text{ Hz, 8 Hz, ArH}), 11.09 \ (1 \text{H, brs, NH}). \]

\[ ^{13} \text{C NMR} \ 100 \text{ MHz (CDCl}_3 \text{):} \delta \ 14.1, 25.4, 61.3, 115, 120.2, 122.3, 130.7, 134.5, 141.6, 168.3, 169. \]

IR (cm\(^{-1}\)): 3252, 2923, 2852, 1705, 1681, 1590, 1442, 1360, 1261, 1085, 752.

**Methyl 2-acetamido-5-chlorobenzoate (4c):**

![Methyl 2-acetamido-5-chlorobenzoate (4c)](image)

Yield: 0.1571 g (69\%).
$^1{\text{H}}$ NMR 400 MHz (CDCl$_3$): δ 2.23 (3H, s, CH$_3$), 3.94 (3H, s, OCH$_3$), 7.48 (1H, dd, $J = 2.6$ Hz, 9 Hz, ArH), 7.99 (1H, d, $J = 2.6$ Hz, ArH), 8.69 (1H, d, $J = 9$ Hz, ArH), 10.96 (1H, brs, NH).

$^{13}{\text{C}}$ NMR 100 MHz (CDCl$_3$): δ 25.4, 52.6, 116, 121.7, 127.4, 130.3, 134.5, 140.1, 169.

IR (cm$^{-1}$): 3273, 2923, 2853, 1688, 1600, 1511, 1469, 1359, 1250, 1106, 835, 788, 733.

**Ethyl 2-acetamido-5-chlorobenzoate (4d):**

Yield: 0.1466 g (60%).

$^1{\text{H}}$ NMR 400 MHz (CDCl$_3$): δ 1.42 (3H, t, $J = 7$ Hz, CH$_3$), 2.23 (3H, s, CH$_3$), 4.39 (2H, q, $J = 7$ Hz, CH$_2$), 7.48 (1H, dd, $J = 2.6$ Hz, 9 Hz, ArH), 8.00 (1H, d, $J = 2.6$ Hz, ArH), 8.69 (1H, d, $J = 9$ Hz, ArH), 11.02 (1H, brs, NH).


IR (cm$^{-1}$): 3266, 2922, 2852, 1699, 1681, 1523, 1400, 1366, 1234, 838, 789, 734.

**Methyl 2-acetamido-5-bromobenzoate (4e):**

Yield: 0.1958 g (72%), mp: 122 °C.

$^1{\text{H}}$ NMR 400 MHz (CDCl$_3$): δ 2.24 (3H, s, CH$_3$), 3.93 (3H, s, OCH$_3$), 7.62 (1H, dd, $J = 2.3$ Hz, 9 Hz, ArH), 8.14 (1H, d, $J = 2.4$ Hz, ArH), 8.63 (1H, d, $J = 9$ Hz, ArH), 10.97 (1H, brs, NH).

$^{13}{\text{C}}$ NMR 100 MHz (CDCl$_3$): δ 25.4, 52.6, 114.7, 116.3, 122, 133.3, 137.3, 140.6, 167.6, 169.

IR (cm$^{-1}$): 3282, 2922, 2852, 1696, 1680, 1597, 1465, 1361, 1257, 1086, 834, 787, 754.

**Ethyl 2-acetamido-5-bromobenzoate (4f):**

Yield: 0.1746 g (61 %), mp: 120 °C.
$^1$H NMR 400 MHz (CDCl$_3$): δ 1.42 (3H, t, J = 7.1 Hz, CH$_3$), 2.23 (3H, s, CH$_3$), 4.37 (2H, q, J = 7.1 Hz, CH$_2$), 7.60 (1H, dd, J = 2.4 Hz, 9 Hz, ArH), 8.14 (1H, d, J = 2.4 Hz, ArH), 8.64 (1H, d, J = 9 Hz, ArH), 11.02 (1H, brs, NH).

$^{13}$C NMR 100 MHz (CDCl$_3$): δ 14.1, 25.4, 61.8, 114.7, 116.6, 122, 133.2, 137.2, 140.6, 167.1, 169.

IR (cm$^{-1}$): 3260, 2922, 2852, 1698, 1599, 1469, 1365, 1255, 11089, 836, 788, 719.

Methyl 2-acetamido-5-methylbenzoate (4g):

Yield: 0.0789 g (48%), mp: 68 °C.

$^1$H NMR 400 MHz (CDCl$_3$): δ 2.22 (3H, s, CH$_3$), δ 2.32 (3H, s, CH$_3$), 3.92 (3H, s, OCH$_3$), 7.35 (1H, dd, J = 2 Hz, 8.5 Hz, ArH), 7.82 (1H, d, J = 2 Hz, ArH), 8.57 (1H, d, J = 8.5 Hz, ArH), 10.92 (1H, brs, NH).

$^{13}$C NMR 100 MHz (CDCl$_3$): δ 20.6, 25.4, 52.2, 114.7, 120.3, 130.8, 131.9, 135.4, 139.2, 168.8, 168.9.

IR (cm$^{-1}$): 3270, 2922, 2853, 1683, 1594, 1455, 1367, 1266, 1087, 838, 790, 722.

4. References:


Current Data Parameters
NAME: Dec09-2014
EXPNO: 170
PROCNO: 1

F2 - Acquisition Parameters
Date: 20141209
Time: 20.38
INSTRUM: spect
PROBHD: 5 mm PABBO BB-
PULPROG: zg30
TD: 65536
SOLVENT: CDCl3
NS: 8
DS: 2
SWH: 12019.230 Hz
FIDRES: 0.183399 Hz
AQ: 2.7263477 sec
RG: 228
DW: 41.600 usec
DE: 6.00 usec
D1: 1.00000000 sec
TD0: 1

F2 - Processing parameters
SI: 32768
SF: 400.1300043 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00

avtar_saifpu@yahoo.co.in
**Current Data Parameters**

- **NAME**: Dec09-2014
- **EXPNO**: 171
- **PROCNO**: 1

**F2 - Acquisition Parameters**

- **Date**: 20141209
- **Time**: 21.07
- **INSTRUM**: spect
- **PROBHD**: 5 mm PABBO BB-
- **PULPROG**: zgpg30
- **TD**: 65536
- **SOLVENT**: CDCl3
- **NS**: 512
- **DS**: 4
- **SWH**: 29761.904 Hz
- **FIDRES**: 0.454131 Hz
- **AQ**: 1.1010548 sec
- **RG**: 1030
- **DW**: 0.05 usec
- **DE**: 6.00 usec
- **TE**: 295.8 K
- **D1**: 2.00000000 sec
- **d11**: 0.03000000 sec
- **DELTA**: 1.89999998 sec
- **TD0**: 1

**F1 - Processing parameters**

- **SI**: 32768
- **SF**: 100.6127669 MHz
- **WDW**: EM
- **SSB**: 0
- **LB**: 0.00 Hz
- **GB**: 0
- **PC**: 1.40

**Bruker Spectrometer**

AVANCE II 400 NMR Spectrometer
SAIF
Panjab University Chandigarh

avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME       Jan23-2015
EXPNO                  180
PROCNO                1

F2 - Acquisition Parameters
Date_          20150123
Time              17.40
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH           12019.230 Hz
FIDRES         0.183399 Hz
AQ            2.7263477 sec
RG                  144
DW               41.600 usec
DE                 6.00 usec
TE                293.7 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.90 usec
PL1               -3.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300073 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

data analysis and interpretation

COOCH₂CH₃
NHCOOCH₂CH₃

2b
Current Data Parameters
NAME: Jun23-2015
EXPNO: 270
PROCNO: 1
F2 - Acquisition Parameters
Date: 20150623
Time: 16.51
INSTRUM: spect
PROBHD: 5 mm PABBO BB-
PULPROG: zg30
TD: 65536
SOLVENT: CDCl3
NS: 8
DS: 2
SWH: 12019.230 Hz
FIDRES: 0.183399 Hz
AQ: 2.7263477 sec
RG: 41.600 usec
TE: 298.6 K
D1: 1.00000000 sec
TD0: 1

======== CHANNEL f1 ========
NUC1: 1H
P1: 10.90 usec
PL1: -3.00 dB
SFO1: 400.1324710 MHz

F2 - Processing parameters
SI: 32768
SF: 400.1300100 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00
\[
\text{COOCH}_2\text{CH}_2\text{OCHCH}_3
\]

\[
\text{NHCOOCH}_2\text{CH}_2\text{OCHCH}_3
\]

\[
\text{2e}
\]
Current Data Parameters
NAME         Mar03-2015
EXPNO               141
PROCNO                1

F2 - Acquisition Parameters
Date_          20150303
Time              14.58
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  422
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010548 sec
RG                  645
DW               16.800 usec
DE                 6.00 usec
TE                296.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.60 usec
PL1               -2.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -3.00 dB
PL12              14.31 dB
PL13              18.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME: Jan23-2015
EXPNO: 231
PROCNO: 1

F2 - Acquisition Parameters
Data: 20150123
Time: 20.58
INSTRUM: spect
BANDW: 5.00 mm PABBO BB-
PULPROG: zgpg30
TD: 8192
SOLVENT: CDCl3
NS: 512
SWH: 29761.904 Hz
FIDRES: 0.454131 Hz
AQ: 1.1010548 sec
RG: 812
DW: 16.800 usec
DE: 6.00 usec
TE: 294.4 K
D1: 2.0000000 sec
d11: 0.3300000 sec

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.60 usec
PL1: -2.00 dB
SFO1: 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 80.00 usec
PL2: -3.00 dB
PL12: 14.31 dB
PL13: 18.00 dB
SFO2: 400.1316005 MHz

F2 - Processing parameters
SI: 32768
SF: 100.6127692 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

ACK-083
ACK-069

BRUKER
AVANCE II 400 NMR
Spectrometer
SAIF
Panjab University
Chandigarh

Current Data Parameters
NAME: Dec09-2014
EXPNO: 191
PROCNO: 1

F2 - Acquisition Parameters
Date: 20141209
Time: 22:14
INSTNAME: nami
PRBSAD: 5 mm PABBO BB-
FREQBG: 4:00 ppm
TD: 45536
SOLOVENT: CDCl3
NS: 512
DE: 16.80 usec
DD: 6.00 usec
TE: 295.7 K
DG: 2.00000000 sec
d11: 0.03000000 sec
DELTA: 1.00000000 sec

TD0: 1

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.60 usec
PL1: -2.00 dB
SFO1: 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2: watz16
NUC2: 1H
PCPD2: 80.00 usec
PL2: -3.00 dB
PL12: 14.31 dB
PL13: 18.00 dB
SFO2: 400.1316005 MHz

F2 - Processing parameters
SI: 32768
SF: 100.6127706 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME         Mar03-2015
EXPNO               151
PROCNO                1
F2 - Acquisition Parameters
Date_          20150303
Time              15.33
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010548 sec
RG                  724
DW               16.800 usec
DE                 6.00 usec
TE                296.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
PC                 1.40

======== CHANNEL f1 ========
NUC1                13C
P1                 9.60 usec
PL1               -2.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -3.00 dB
PL12              14.31 dB
PL13              18.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
PC                 1.40

avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME         Dec09-2014
EXPNO               181
PROCNO                1
F2 - Acquisition Parameters
Date_          20141209
Time              21.40
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010548 sec
RG                 1030
DW               16.800 usec
DE                 6.00 usec
TE                296.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TOO                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.60 usec
PL1               -2.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -3.00 dB
PL12              14.31 dB
PL13              18.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127653 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

SAIF
Panjab University
Chandigarh
avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME: Jan23-2015
EXPNO: 220
PROCNO: 1

F2 - Acquisition Parameters
Date: 20150123
Time: 19.55
INSTRUM: spect
PROBHD: 5 mm PABBO BB-
PULPROG: zg30
TD: 65536
SOLVENT: CDCl3
NS: 8
DS: 2
SW: 12019.230 Hz
Aq: 2.7263477 sec
B: 41.00 usec
DE: 4.00 usec
TE: 294.0 K
D1: 1.00000000 sec
TD0: 1

======== CHANNEL f1 ========
NUC1: 1H
P1: 10.90 usec
PL1: -3.00 dB
SFO1: 400.1324710 MHz

F2 - Processing parameters
SI: 32768
SF: 400.1300006 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00

avtar_saifpu@yahoo.co.in
### Current Data Parameters

**NAME**

Jun23-2015

**EXPNO**

280

**PROCNO**

1

---

**F2 - Acquisition Parameters**

- **Date**: 20150623
- **Time**: 20.29
- **INSTRUM**: spect
- **FROBRD**: 5 mm PABBO BB-
- **TD**: 65536
- **SOLVENT**: CDCl3
- **NS**: 8
- **DS**: 2
- **SWH**: 12019.230 Hz
- **AQ**: 2.7263477 sec
- **RG**: 114
- **DW**: 41.600 usec
- **DE**: 6.00 usec
- **TE**: 298.6 K
- **TD0**: 1.00000000 sec

R1

******** CHANNEL f1 ********

**NUC1**: 1H

**P1**: 10.90 usec

**PL1**: -3.00 dB

**SFO**: 400.1324710 MHz

---

**F2 - Processing parameters**

- **SI**: 32768
- **SF**: 400.1300056 MHz
- **WDW**: EM
- **SSB**: 0
- **LB**: 0.30 Hz
- **GB**: 0
- **PC**: 1.00

---

**avtar_saifpu@yahoo.co.in**
Current Data Parameters
NAME         Jun23-2015
EXPNO               281
PROCNO                1
F2 - Acquisition Parameters
Date_          20150623
Time              20.58
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010548 sec
RG                  645
DW               16.800 usec
DE                 6.00 usec
TE                299.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1
======== CHANNEL f1 ========
NUC1                13C
P1                 9.60 usec
PL1               -2.00 dB
SFO1        100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -3.00 dB
PL12              14.31 dB
PL13              18.00 dB
SFO2        400.1316005 MHz
F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME         Jun23-2015
EXPNO               300
PROCNO                1

F2 - Acquisition Parameters
Date_          20150624
Time               9.30
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH           12019.230 Hz
FIDRES         0.183399 Hz
AQ            2.7263477 sec
RG                  128
DW               41.600 usec
DE                 6.00 usec
TE                298.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.90 usec
PL1               -3.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300076 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME: Sep16-2015
EXPNO: 471
PROCNO: 1

F2 - Acquisition Parameters
Date: 20150917
Time: 6.20
INSTRUM: spect
PULPROG: zgpg30
SOLVENT: CDCl3
PROBHD: 5 mm PABBO BB-
TD: 65536
SOLVENT: CDCl3
PROBHD: 5 mm PABBO BB-
TD: 65536

F2 - Processing parameters
SI: 32768
SF: 100.6127690 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

AHK-497
BRUKER
AVANCE II 400 NMR Spectrometer
SAIF
Panjab University Chandigarh

manishkumarmanu1986@gmail.com
Current Data Parameters

NAME: Sep16-2015
EXPNO: 401
PROCNO: 1

F2 - Acquisition Parameters
Date: 20150917
Time: 6.54

INSTRUM: spect
PROBHD: 5 mm PABBO BB-
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 512

SWH: 29761.904 Hz
FIDRES: 0.454131 Hz
AQ: 1.1010548 sec
RG: 36
DW: 16.800 usec
DE: 6.00 usec
TE: 298.8 K

D1: 2.00000000 sec
d11: 0.03000000 sec
DELTA: 1.89999998 sec
TD0: 1

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.60 usec
PL1: -2.00 dB
SFO1: 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 80.00 usec
PL2: -3.00 dB
PL12: 14.31 dB
PL13: 18.00 dB
SFO2: 400.1316005 MHz

F2 - Processing parameters
SI: 32768
SF: 100.6127690 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz

PC: 1.40

manishkumarmanu1986@gmail.com
Current Data Parameters
NAME         Sep16-2015
EXPNO               490
PROCNO                1
F2 - Acquisition Parameters
Date_          20150917
Time               6.59
INSTRUM           spect
PROBHD   5 mm PABBO BB-
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH           12019.230 Hz
FIDRES         0.183399 Hz
AQ            2.7263477 sec
RG                  406
DW               41.600 usec
DE                 6.00 usec
D1           1.00000000 sec
TD0                   1
======== CHANNEL f1 ========
NUC1                 1H
P1                10.90 usec
PL1               -3.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300090 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

manishkumarmanul986@gmail.com
AHK-537

Br

COOCH₃

NHCOCH₃

2v

AHK-537

Br

COOCH₃

NHCOCH₃

2v
### Current Data Parameters

**NAME** | Oct08-2015  
--- | ---  
**EXPNO** | 330  
**PROCNO** | 1  

---

**F2 - Acquisition Parameters**

- **Date**: 20151008  
- **Time**: 23.38  
- **INSTRUM**: spect  
- **PROBHD**: 5 mm PABBO BB-  
- **PULPROG**: zg30  
- **TD**: 65536  
- **SOLVENT**: CDCl3  
- **NS**: 8  
- **DS**: 2  
- **SWH**: 12019.230 Hz  
- **FIDRES**: 0.183399 Hz  
- **AQ**: 2.7263477 sec  
- **RG**: 724  
- **DW**: 41.600 usec  
- **DE**: 6.00 usec  
- **TE**: 297.9 K  
- **D1**: 1.00000000 sec  
- **TD0**: 1  

---

**F2 - Processing parameters**

- **SI**: 32768  
- **SF**: 400.1300088 MHz  
- **WDW**: EM  
- **SSB**: 0  
- **LB**: 0.30 Hz  
- **GB**: 0  
- **PC**: 1.00

---

**AHK-539**

![NMR Spectrum of AHK-539](image)

**BRUKER AVANCE II 400 NMR Spectrometer**  
**SAIF**  
**Panjab University Chandigarh**

---

**manishkumarmanu1986@gmail.com**
**Current Data Parameters**

**NAME** | Oct08-2015
---|---
**EXPNO** | 341
**PROCNO** | 1

**F2 - Acquisition Parameters**

**Date:** 20151009  
**Time:** 1.35

**INSTRUM** | spec
**FREQM** | 45534  
**STRING** | 5 mm MAS Rb-
**SOFT** | 1024
**DS** | 4

**FID**

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<th>Value</th>
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<td>TD</td>
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<td>AQ</td>
<td>1.1010548 sec</td>
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<td>RW</td>
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<tr>
<td>DW</td>
<td>16.800 usec</td>
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<tr>
<td>TE</td>
<td>298.0 K</td>
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<td>DELTA</td>
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**TD0** | 1

**======== CHANNEL f1 ========**

**NUC1** | 13C
**PCF1** | 9.60 usec  
**PL1** | -2.00 dB

**SFO1** | 100.6228298 MHz

**======== CHANNEL f2 ========**

**NUC2** | 1H
**PCPD2** | 80.00 usec  
**PL2** | -3.00 dB

**SFO2** | 400.1316005 MHz

**F2 - Processing parameters**

**SI** | 32768  
**SF** | 100.6127690 MHz
**WDW** | EM
**SSB** | 0

**LB** | 1.00 Hz
**PC** | 1.40

**TD0** | 1

**AHK-541**

---

**Current Data Parameters**

**NAME** | Oct08-2015
---|---
**EXPNO** | 341
**PROCNO** | 1

**F2 - Acquisition Parameters**

**Date:** 20151009  
**Time:** 1.35

**INSTRUM** | spec
**FREQM** | 45534  
**STRING** | 5 mm MAS Rb-
**SOFT** | 1024
**DS** | 4

**FID**

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<td>298.0 K</td>
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<tr>
<td>D1</td>
<td>0.03000000 sec</td>
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<tr>
<td>DELTA</td>
<td>1.89999998 sec</td>
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</table>

**TD0** | 1

**======== CHANNEL f1 ========**

**NUC1** | 13C
**PCF1** | 9.60 usec  
**PL1** | -2.00 dB

**SFO1** | 100.6228298 MHz

**======== CHANNEL f2 ========**

**NUC2** | 1H
**PCPD2** | 80.00 usec  
**PL2** | -3.00 dB

**SFO2** | 400.1316005 MHz

**F2 - Processing parameters**

**SI** | 32768  
**SF** | 100.6127690 MHz
**WDW** | EM
**SSB** | 0

**LB** | 1.00 Hz
**PC** | 1.40

**TD0** | 1

**AHK-541**

---

**Current Data Parameters**

**NAME** | Oct08-2015
---|---
**EXPNO** | 341
**PROCNO** | 1

**F2 - Acquisition Parameters**

**Date:** 20151009  
**Time:** 1.35

**INSTRUM** | spec
**FREQM** | 45534  
**STRING** | 5 mm MAS Rb-
**SOFT** | 1024
**DS** | 4

**FID**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>TD</td>
<td>65536</td>
</tr>
<tr>
<td>SOLVENT</td>
<td>CDCl3</td>
</tr>
<tr>
<td>NS</td>
<td>1024</td>
</tr>
<tr>
<td>SWH</td>
<td>29761.904 Hz</td>
</tr>
<tr>
<td>FIDRES</td>
<td>0.454131 Hz</td>
</tr>
<tr>
<td>AQ</td>
<td>1.1010548 sec</td>
</tr>
<tr>
<td>RW</td>
<td>64</td>
</tr>
<tr>
<td>DW</td>
<td>16.800 usec</td>
</tr>
<tr>
<td>TE</td>
<td>298.0 K</td>
</tr>
<tr>
<td>D1</td>
<td>0.03000000 sec</td>
</tr>
<tr>
<td>DELTA</td>
<td>1.89999998 sec</td>
</tr>
</tbody>
</table>

**TD0** | 1

**======== CHANNEL f1 ========**

**NUC1** | 13C
**PCF1** | 9.60 usec  
**PL1** | -2.00 dB

**SFO1** | 100.6228298 MHz

**======== CHANNEL f2 ========**

**NUC2** | 1H
**PCPD2** | 80.00 usec  
**PL2** | -3.00 dB

**SFO2** | 400.1316005 MHz

**F2 - Processing parameters**

**SI** | 32768  
**SF** | 100.6127690 MHz
**WDW** | EM
**SSB** | 0

**LB** | 1.00 Hz
**PC** | 1.40

**TD0** | 1

**AHK-541**
**2a**

Exact mass: 209.06
Mass Found (M+23): 232

**2b**

Exact mass: 237.10
Mass Found (M+23): 260
**2d**

Exact mass: 345.06  
Mass Found (M+NH₄): 363

---

**2f**

Exact mass: 227.05  
Mass Found (M+23): 250


**Exact mass:** 243.03  
**Mass Found (M+23):** 266

**Exact mass:** 271.06  
**Mass Found (M+23):** 294
Exact mass: 286.97
Mass Found [(M+2)+23]: 311

Exact mass: 315.01
Mass Found (M+23): 338
**2k**

Exact mass: 366.88  
Mass Found [M+23]: 390

**2l**

Exact mass: 394.92  
Mass Found [M+23]: 418
Exact mass: 282.08
Mass Found (M+23): 305