

Supplementary Information

for

Aerobic flow oxidation of alcohols in water catalyzed by an amphiphilic polymer-dispersion of platinum nanoparticles

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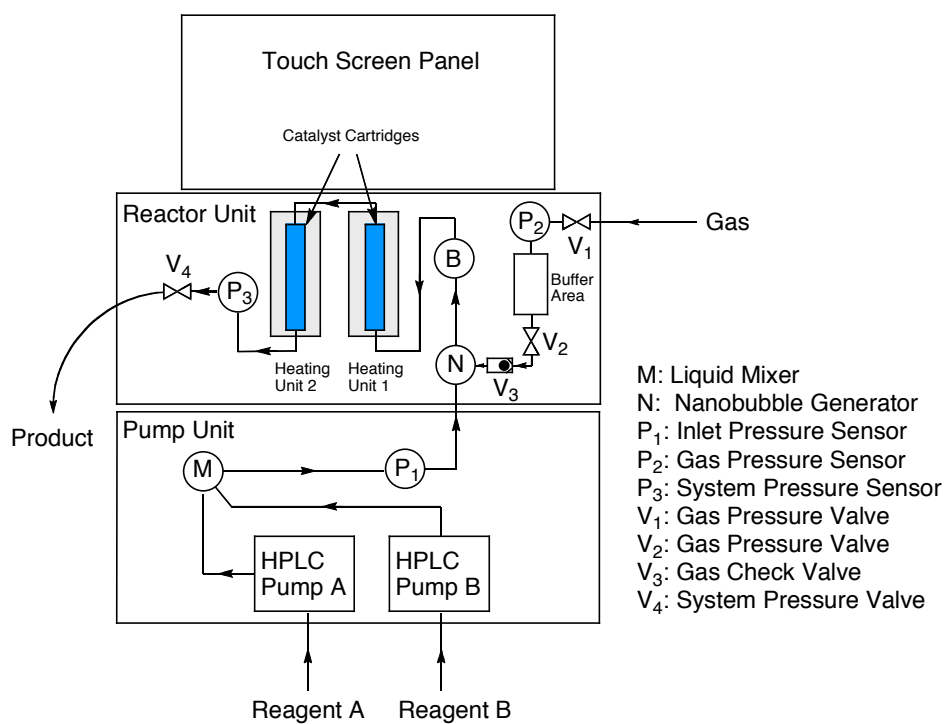
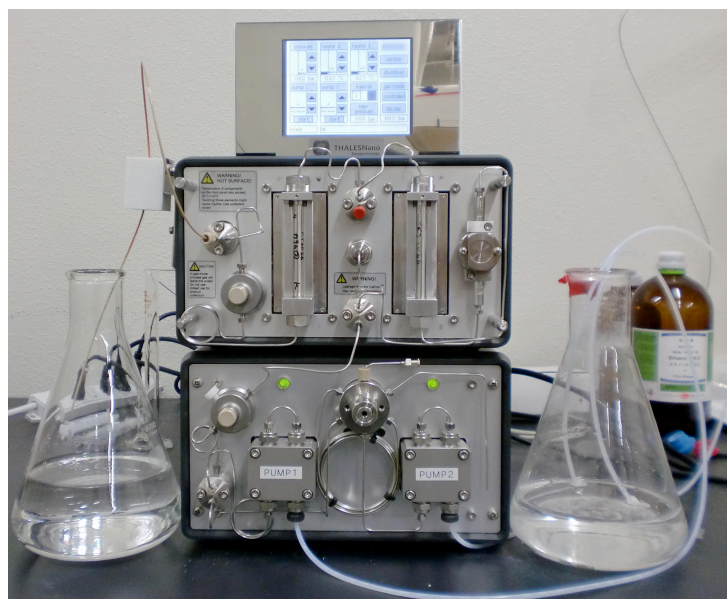


Fig. S1 The picture (top) and the schematic diagram (bottom) of the X-Cube reactor.

Characterization of products.

Acetophenone (**2a**) [CAS: 98-86-2]: ^1H NMR (500 MHz, CDCl_3): $\delta = 7.96$ (dd, $J = 8.5$ and 1.0 Hz, 2H, $\text{Ph}_{\text{H-2}}$ and $\text{Ph}_{\text{H-6}}$), 7.57 (t, $J = 8.0$ Hz, 1H, $\text{Ph}_{\text{H-4}}$), 7.47 (t, $J = 8.0$ Hz, 2H, $\text{Ph}_{\text{H-3}}$ and $\text{Ph}_{\text{H-5}}$), 2.61 (s, 3H, $-\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3): $\delta = 198.10$ (C=O), 137.06 (Ph), 133.04 (Ph), 128.50 (Ph), 128.24 (Ph), 26.54 (CH_3). EI-TOF-MS $m/z = 120$ (M).

4'-Methoxyacetophenone (**2b**) [CAS: 100-06-1]: ^1H NMR (500 MHz, CDCl_3): $\delta = 7.93$ (d, $J = 9.0$ Hz, 2H, $\text{Ar}_{\text{H-2}}$ and $\text{Ar}_{\text{H-6}}$), 6.92 (d, $J = 9.5$ Hz, 2H, $\text{Ar}_{\text{H-3}}$ and $\text{Ar}_{\text{H-5}}$), 3.86 (s, 3H, $-\text{OCH}_3$), 2.55 (s, 3H, $-\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3): $\delta = 196.72$ (C=O), 163.44 (Ar), 130.54 (Ar), 130.30 (Ar), 113.63 (Ar), 55.42 (OCH_3), 26.29 (CH_3). EI-TOF-MS $m/z = 150$ (M).

4'-Methylacetophenone (**2c**) [CAS: 122-00-9]: ^1H NMR (500 MHz, CDCl_3): $\delta = 7.85$ (d, $J = 10.0$ Hz, 2H, $\text{Ar}_{\text{H-2}}$ and $\text{Ar}_{\text{H-6}}$), 7.26 (d, $J = 10.0$ Hz, 2H, $\text{Ar}_{\text{H-3}}$ and $\text{Ar}_{\text{H-5}}$), 2.56 (s, 3H, $-\text{CH}_3$), 2.40 (s, 3H, $-\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3): $\delta = 197.74$ (C=O), 143.75 (Ar), 134.59 (Ar), 129.12 (Ar), 128.32 (Ar), 26.38 (CH_3), 21.49 (CH_3). EI-TOF-MS $m/z = 134$ (M).

4'-Chloroacetophenone (**2d**) [CAS: 99-91-2]: ^1H NMR (396 MHz, CDCl_3): $\delta = 7.90$ (dt, $J = 8.7$ and 2.0 Hz, 2H, $\text{Ar}_{\text{H-2}}$ and $\text{Ar}_{\text{H-6}}$), 7.44 (dt, $J = 8.7$ and 2.0 Hz, 2H, $\text{Ar}_{\text{H-3}}$ and $\text{Ar}_{\text{H-5}}$), 2.59 (s, 3H, $-\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): $\delta = 196.81$ (C=O), 139.50 (Ar), 135.34 (Ar), 129.67 (Ar), 128.83 (Ar), 26.53 (CH_3). EI-TOF-MS $m/z = 154$ (M).

4'-(Trifluoromethyl)acetophenone (**2e**) [CAS: 709-63-7]: ^1H NMR (396 MHz, CDCl_3): $\delta = 8.07$ (d, $J = 7.9$ Hz, 2H, $\text{Ar}_{\text{H-2}}$ and $\text{Ar}_{\text{H-6}}$), 7.74 (d, $J = 7.9$ Hz, 2H, $\text{Ar}_{\text{H-3}}$ and $\text{Ar}_{\text{H-5}}$), 2.66 (s, 3H, $-\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): $\delta = 197.00$ (C=O), 139.61 (Ar), 134.38 (q, $J = 32.5$ Hz, Ar), 128.60 (Ar), 125.65 (q, $J = 3.8$ Hz Ar), 123.56 (q, $J = 272.8$ Hz, Ar), 26.78 (CH_3). EI-TOF-MS $m/z = 188$ (M).

Cyclopropyl phenyl ketone (**2f**) [CAS: 3481-02-5]: ^1H NMR (396 MHz, CDCl_3): $\delta = 8.02$ (d, $J = 7.5$ Hz, 2H, $\text{Ar}_{\text{H-2}}$ and $\text{Ar}_{\text{H-6}}$), 7.57 (tt, $J = 7.5$ and 1.2 Hz, 1H, $\text{Ar}_{\text{H-4}}$), 7.48 (t, $J = 7.5$ Hz, 2H, $\text{Ar}_{\text{H-3}}$ and $\text{Ar}_{\text{H-5}}$), 2.69 (sept, $J = 4.5$ Hz, 1H, CH), 1.27 - 1.24 (m, 2H, CH_2), 1.07 - 1.03 (m, 2H, CH_2).

$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): $\delta = 200.58$ (C=O), 137.85 (Ar), 132.63 (Ar), 128.39 (Ar), 127.89 (Ar), 17.02 (CH), 11.60 (CH_2). EI-TOF-MS $m/z = 146$ (M).

2-Pentanone (**2g**) [CAS: 107-87-9]: ^1H NMR (500 MHz, CDCl_3): $\delta = 2.41$ (t, $J = 7.5$ Hz, 2H, $-\text{CH}_2\text{COCH}_3$), 2.14 (s, 3H, $\text{CH}_3\text{CO}-$), 1.61 (sext, $J = 7.5$ Hz, 2H, $\text{CH}_3\text{CH}_2\text{CH}_2-$), 0.92 (t, $J = 7.5$ Hz, 3H, CH_3CH_2-). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3): $\delta = 209.18$ (C=O), 45.63 ($-\text{CH}_2\text{COCH}_3$), 29.79 ($-\text{COCH}_3$), 17.24 (CH_3CH_2-), 13.62 (CH_3CH_2-). EI-TOF-MS $m/z = 86$ (M).

2-Hexanone (**2h**) [CAS: 591-78-6]: ^1H NMR (500 MHz, CDCl_3): $\delta = 2.43$ (t, $J = 7.5$ Hz, 2H, $-\text{CH}_2\text{COCH}_3$), 2.14 (s, 3H, $\text{CH}_3\text{CO}-$), 1.56 (quint, $J = 7.5$ Hz, 2H, $-\text{CH}_2\text{CH}_2\text{COCH}_3$), 1.32 (sext, $J = 7.5$ Hz, 2H, $\text{CH}_3\text{CH}_2\text{CH}_2-$), 0.91 (t, $J = 7.5$ Hz, 3H, CH_3CH_2-). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3): $\delta = 209.35$ (C=O), 43.48 ($-\text{CH}_2\text{COCH}_3$), 29.80 ($-\text{COCH}_3$), 25.93 ($\text{CH}_3\text{CH}_2\text{CH}_2-$), 22.25 ($\text{CH}_3\text{CH}_2\text{CH}_2-$), 13.80 (CH_3CH_2-). EI-TOF-MS $m/z = 100$ (M).

2-Octanone (**2i**) [CAS: 111-13-7]: ^1H NMR (396 MHz, CDCl_3): $\delta = 2.43$ (t, $J = 7.1$ Hz, 2H, $-\text{CH}_2\text{COCH}_3$), 2.14 (s, 3H, $\text{CH}_3\text{CO}-$), 1.57 (quint, $J = 7.1$ Hz, 2H, $\text{CH}_3\text{CH}_2\text{CH}_2-$), 1.33-1.25 (m, 6H, $-\text{CH}_2-$), 0.88 (t, $J = 7.1$ Hz, 3H, CH_3CH_2-). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): $\delta = 209.40$ (C=O), 43.73 ($-\text{CH}_2\text{COCH}_3$), 31.52 ($-\text{CH}_2-$), 29.77 ($-\text{CH}_2-$), 28.77 ($-\text{COCH}_3$), 23.74 ($-\text{CH}_2-$), 22.42 (CH_3CH_2-), 13.95 (CH_3CH_2-). EI-TOF-MS $m/z = 128$ (M).

2-Decanone (**2j**) [CAS: 693-54-9]: ^1H NMR (396 MHz, CDCl_3): $\delta = 2.42$ (t, $J = 7.5$ Hz, 2H, $-\text{CH}_2\text{COCH}_3$), 2.14 (s, 3H, $\text{CH}_3\text{CO}-$), 1.57 (quint, $J = 7.5$ Hz, 2H, $\text{CH}_3\text{CH}_2\text{CH}_2-$), 1.28 (br-s, 10H, $-\text{CH}_2-$), 0.88 (t, $J = 6.7$ Hz, 3H, CH_3CH_2-). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): $\delta = 209.49$ (C=O), 43.80 ($-\text{CH}_2\text{COCH}_3$), 31.78 ($-\text{CH}_2-$), 29.84, 29.33, 29.15, 29.11, 23.83, 22.61 (CH_3CH_2-), 14.07 (CH_3CH_2-). EI-TOF-MS $m/z = 156$ (M).

Cyclopentanone (**2k**) [CAS: 120-92-3]: ^1H NMR (500 MHz, CDCl_3): $\delta = 2.18$ -2.15 (m, 4H, $\text{CH}_2\text{COCH}_2-$), 1.98-1.95 (m, 4H, $-\text{CH}_2\text{CH}_2-$). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3): $\delta = 220.60$ (C=O), 38.25 ($-\text{CH}_2\text{COCH}_2-$), 23.13 ($-\text{CH}_2\text{CH}_2-$). EI-TOF-MS $m/z = 84$ (M).

Cyclohexanone (**2l**) [CAS: 108-94-1]: ^1H NMR (500 MHz, CDCl_3): $\delta = 2.34$ (t, $J = 7.0$ Hz, 4H, $-\text{CH}_2\text{COCH}_2-$), 1.87 (quint, $J = 7.0$ Hz, 4H, $-\text{CH}_2\text{CH}_2\text{COCH}_2\text{CH}_2-$), 1.75-1.70 (m, 2H, $-\text{CH}_2-$).

CH₂CH₂CH₂CH₂CH₂-). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ = 212.05 (C=O), 41.88 (-CH₂COCH₂-), 26.92 (-CH₂CH₂COCH₂CH₂-), 24.89 (-CH₂CH₂CH₂CH₂CH₂-). EI-TOF-MS *m/z* = 98 (M).

Cycloheptanone (**2m**) [CAS: 502-42-1]: ¹H NMR (500 MHz, CDCl₃): δ = 2.50 (t, *J* = 6.0 Hz, 4H, -CH₂COCH₂-), 1.72-1.68 (m, 8H, -CH₂(CH₂)₄CH₂-). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ = 215.31 (C=O), 43.79 (-CH₂COCH₂-), 30.34 (-CH₂CH₂COCH₂CH₂-), 24.26 (-CH₂CH₂(CH₂)₂CH₂CH₂-). EI-TOF-MS *m/z* = 112 (M).

2-Adamantanone (**2n**) [CAS: 700-58-3]: ¹H NMR (500 MHz, CDCl₃): δ = 2.55 (s, 2H, -CHC(O)-), 2.10-1.94 (m, 12H, alkyl). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ = 218.47 (C=O), 46.91 (-CHC(O)-), 39.20 (-CH₂-), 36.23 (-CH₂-), 27.39 (-CH-). EI-TOF-MS *m/z* = 150 (M).

(+)-Camphor (**2o**) [CAS: 464-49-3]: The yield of the ketone (82%) was determined by ¹H-NMR with an internal standard (CHCl₂CHCl₂: δ = 6.01). The starting alcohol was also observed (13%). ¹H NMR (396 MHz, CDCl₃): δ = 2.36 (dt, *J* = 18.2 and 3.6 Hz, 1H, COCH_H), 2.10 (t, *J* = 4.4 Hz, 1H, CH), 2.00-1.90 (m, 1H, CH₂), 1.85 (d, *J* = 18.2 Hz, 1H, COCH_H), 1.69 (td, *J* = 13.1 and 3.6 Hz, 1H, CH₃), 1.44-1.31 (m, 2H, CH₂), 0.96 (s, 3H, CH₃), 0.91 (s, 3H, CH₃), 0.84 (s, 2H, CH₃). ¹³C{¹H} NMR (100 MHz, CDCl₃): δ = 219.71 (C=O), 57.62, 46.71, 43.21, 42.93, 29.81, 26.95, 19.70, 19.06, 9.18. EI-TOF-MS *m/z* = 152 (M).

Benzoic acid (**4a**) [CAS: 65-85-0]. ¹H NMR (500 MHz, CDCl₃): δ = 8.14 (dd, *J* = 8.0 and 1.0 Hz, 2H, Ph_{H-2} and Ph_{H-6}), 7.62 (td, *J* = 7.5 and 1.0 Hz, 1H, Ph_{H-4}), 7.49 (t, *J* = 8.0 Hz, 2H, Ph_{H-3} and Ph_{H-5}). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ = 172.34 (C=O), 133.82 (Ph), 130.21 (Ph), 129.30 (Ph), 128.48 (Ph). EI-TOF-MS *m/z* = 122 (M).

p-Anisic acid (**4b**) [CAS: 100-09-4]. ¹H NMR (500 MHz, DMSO-*d*₆): δ = 12.63 (s, 1H, COOH), 7.89 (dt, *J* = 9.5 and 2.5 Hz, 2H, Ar_{H-2} and Ar_{H-6}), 7.02 (dt, *J* = 8.5 and 2.0 Hz, 2H, Ar_{H-3} and Ar_{H-5}), 3.82 (s, 3H, CH₃). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆): δ = 167.06 (C=O), 162.87 (Ar), 131.39 (Ar), 122.99 (Ar), 113.84 (Ar), 55.47 (CH₃). EI-TOF-MS *m/z* = 152 (M).

p-Toluic acid (**4c**) [CAS: 99-94-5]. ¹H NMR (500 MHz, DMSO-*d*₆): δ = 12.84 (br-s, 1H, COOH), 7.88 (d, *J* = 8.5 Hz, 2H, Ar_{H-2} and Ar_{H-6}), 7.34 (d, *J* = 8.5 Hz, 2H, Ar_{H-3} and Ar_{H-5}), 2.41 (s, 3H, -CH₃).

$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6): δ = 167.33 (C=O), 143.04 (Ar), 129.35 (Ar), 129.13 (Ar), 128.04 (Ar), 21.13 (CH₃). EI-TOF-MS m/z = 136 (M).

4-Chlorobenzoic acid (**4d**) [CAS: 74-11-3]. ^1H NMR (500 MHz, DMSO- d_6): δ = 13.19 (br-s, 1H, COOH), 7.95 (dt, J = 9.0 and 2.0 Hz, 2H, Ar_{H-2} and Ar_{H-6}), 7.58 (dt, J = 8.5 and 2.0 Hz, 2H, Ar_{H-3} and Ar_{H-5}). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6): δ = 166.46 (C=O), 137.80 (Ar), 131.14 (Ar), 129.64 (Ar), 128.73 (Ar). EI-TOF-MS m/z = 156 (M).

4-Nitrobenzoic acid (**4e**) [CAS: 62-23-7]. ^1H NMR (500 MHz, DMSO- d_6): δ = 13.69 (br-s, 1H, COOH), 8.33 (dt, J = 9.0 and 2.5 Hz, 2H, Ar_{H-2} and Ar_{H-6}), 8.17 (dt, J = 9.0 and 2.5 Hz, 2H, Ar_{H-3} and Ar_{H-5}). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6): δ = 165.77 (C=O), 150.02 (Ar), 136.37 (Ar), 130.66 (Ar), 123.71 (Ar). EI-MS m/z = 167 (M).

4-(Trifluoromethyl)benzoic acid (**4f**) [CAS: 455-24-3]. ^1H NMR (500 MHz, DMSO- d_6): δ = 13.48 (br-s, 1H, COOH), 8.17 (d, J = 8.0 Hz, 2H, Ar_{H-2} and Ar_{H-6}), 7.88 (d, J = 8.5 Hz, 2H, Ar_{H-3} and Ar_{H-5}). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6): δ = 166.26 (C=O), 134.64 (Ar), 132.56 (q, J = 32.1 Hz, Ar), 130.13 (Ar), 123.841 (q, J = 272.7 Hz, CF₃). EI-TOF-MS m/z = 190 (M).

Terephthalic acid (**4g**) [CAS: 100-21-0]. ^1H NMR (500 MHz, DMSO- d_6): δ = 13.28 (br-s, 2H, COOH), 8.04 (s, 4H, Ar). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6): δ = 166.79 (C=O), 134.54 (Ar), 129.57 (Ar). EI-MS m/z = 166 (M).

o-Toluic acid (**4h**) [CAS: 118-90-1]. ^1H NMR (500 MHz, CDCl₃): δ = 12.52 (br-s, 1H, COOH), 8.08 (d, J = 7.5 Hz, 1H, Ar_{H-6}), 7.45 (td, J = 7.5 and 1.5 Hz, 1H, Ar_{H-4}), 7.28-7.25 (m, 2H, Ar_{H-3} and Ar_{H-5}). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl₃): δ = 173.47 (C=O), (Ar), 141.37 (Ar), 132.96 (Ar), 131.92 (Ar), 131.59 (Ar), 128.32 (Ar), 125.85 (Ar), 22.11 (CH₃). EI-TOF-MS m/z = 136 (M).

m-Toluic acid (**4i**) [CAS: 99-04-7]. ^1H NMR (396 MHz, CDCl₃): δ = 11.31 (br-s, 1H, COOH), 7.93 (s, 1H, Ar_{H-2}), 7.92 (d, J = 7.9 Hz, 1H, Ar_{H-6}), 7.42-7.33 (m, 2H, Ar_{H-4} and Ar_{H-5}), 2.41 (s, 3H, CH₃). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl₃): δ = 172.60 (C=O), 138.30 (Ar), 134.59 (Ar), 130.71 (Ar), 129.24 (Ar), 128.37 (Ar), 127.37 (Ar), 21.24 (CH₃). EI-TOF-MS m/z = 136 (M).

2-Chlorobenzoic acid (**4j**) [CAS: 118-91-2]. ^1H NMR (500 MHz, DMSO- d_6): δ = 13.38 (br-s, 1H, COOH), 7.78 (d, J = 8.0 Hz, 1H, Ar_{H6}), 7.56-7.52 (m, 2H, Ar_{H3} and Ar_{H4}), 7.43 (ddd, J = 7.8, 5.5 and 2.5 Hz, 1H, Ar_{H5}). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6): δ = 166.72 (C=O), 132.56 (Ar), 131.54 (Ar), 131.47 (Ar), 130.77 (Ar), 130.59 (Ar), 127.22 (Ar). EI-TOF-MS m/z = 156 (M).

3-Chlorobenzoic acid (**4k**) [CAS: 535-80-8]. ^1H NMR (500 MHz, DMSO- d_6): δ = 13.34 (br-s, 1H, COOH), 7.91-7.90 (m, 2H, Ar_{H2} and Ar_{H6}), 7.71 (d, J = 8.5 Hz, 1H, Ar_{H4}), 7.55 (t, J = 8.5 Hz, 1H, Ar_{H5}). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6): δ = 166.11 (C=O), 133.37 (Ar), 132.91 (Ar), 132.76 (Ar), 130.71 (Ar), 128.86 (Ar), 127.96 (Ar). EI-TOF-MS m/z = 156 (M).

Methyl terephthalate (**4l**) [CAS: 39379-10-7]. ^1H NMR (500 MHz, DMSO- d_6): δ = 13.36 (br-s, 1H, COOH), 8.09-8.06 (m, 4H, Ar), 3.90 (s, 3H, CH₃). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6): δ = 166.56 (COOH), 165.62 (COOCH₃), 134.82 (Ar), 133.17 (Ar), 129.59 (Ar), 129.34 (Ar), 52.45 (CH₃). EI-TOF-MS m/z = 180 (M).

Piperonylic acid (**4m**) [CAS: 94-53-1]. ^1H NMR (500 MHz, DMSO- d_6): δ = 12.81 (br-s, 1H, COOH), 7.59 (dd, J = 8.0 and 2.0 Hz, 1H, Ar_{H6}), 7.41 (d, J = 2.0 Hz, 1H, Ar_{H2}), 7.04 (d, J = 8.0 Hz, 1H, Ar_{H5}), 6.17 (s, 2H, CH₂). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6): δ = 166.64 (COOH), 151.14 (Ar), 147.48 (Ar), 124.97 (Ar), 124.66 (Ar), 108.80 (Ar), 108.06 (Ar), 101.95 (CH₂). EI-TOF-MS m/z = 166 (M).

4-(Methylthio)benzoic acid (**4n**) [CAS: 13205-48-6]. ^1H NMR (500 MHz, DMSO- d_6): δ = 12.86 (br-s, 1H, COOH), 7.87 (d, J = 8.5 Hz, 2H, Ar_{H2} and Ar_{H6}), 7.35 (d, J = 8.5 Hz, 2H, Ar_{H3} and Ar_{H5}), 2.53 (s, 3H, CH₃). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6): δ = 167.04 (COOH), 144.77 (Ar), 129.70 (Ar), 126.72 (Ar), 124.87 (Ar), 13.95 (CH₃). EI-TOF-MS m/z = 168 (M).

4-Aminobenzaldehyde (**5o**) [CAS: 556-23-8]. The yield (94%) was determined by ^1H -NMR with an internal standard (CHCl₂CHCl₂: δ = 6.14) due to instability of product **5o**. ^1H NMR (500 MHz, CDCl₃): δ = 9.71 (s, 1H, CHO), 7.65 (dt, J = 8.5 and 2.5 Hz, 2H, Ar_{H2} and Ar_{H6}), 6.70 (d, J = 9.0 Hz, 2H, Ar_{H3} and Ar_{H5}). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl₃): δ = 190.18 (C=O), 153.29 (Ar), 132.19 (Ar), 126.40 (Ar), 113.60 (Ar). EI-MS m/z = 135 (M).

4-Hydroxybenzaldehyde (**5p**) [CAS: 123-08-0]. ^1H NMR (396 MHz, DMSO- d_6): δ = 10.60 (br-s, 1H, phenolic OH), 9.79 (s, 1H, CHO), 7.76 (dt, J = 8.7 and 2.0 Hz, 2H, Ar_{H-2} and Ar_{H-6}), 6.93 (dt, J = 8.7 and 2.4 Hz, 2H, Ar_{H-3} and Ar_{H-5}). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6): δ = 191.01 (C=O), 163.32 (Ar), 132.13 (Ar), 128.42 (Ar), 115.85 (Ar). EI-TOF-MS m/z = 121(M-1), 122 (M).

4-(*tert*-Butoxycarbonylamino)benzoic acid (**4q**) [CAS: 66493-39-8]. ^1H NMR (500 MHz, DMSO- d_6): δ = 12.61 (br-s, 1H, COOH), 9.74 (s, 1H, NH), 7.83 (dt, J = 9.0 and 2.0 Hz, 2H, Ar_{H-2} and Ar_{H-6}), 7.55 (d, J = 8.5 Hz, 2H, Ar_{H-3} and Ar_{H-5}), 1.49 (s, 9H, -C(CH₃)₃). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl₃): δ = 166.99 (C=O), 152.52 (NHCO), 143.76 (Ar), 130.34 (Ar), 123.93 (Ar), 117.19 (Ar), 79.64 (C(CH₃)₃), 28.04 (C(CH₃)₃). EI-MS m/z = 237 (M).

4-Acetoxybenzoic acid (**4r**) [CAS: 2345-34-8]. ^1H NMR (500 MHz, CDCl₃): δ = 8.15 (dt, J = 9.0 and 2.0 Hz, 2H, Ar_{H-2} and Ar_{H-6}), 7.22 (dt, J = 8.5 and 2.0 Hz, 2H, Ar_{H-3} and Ar_{H-5}), 2.34 (s, 3H, CH₃). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl₃): δ = 171.13 (COOH), 168.89 (OC(O)CH₃), 154.91 (Ar), 131.84 (Ar), 126.84 (Ar), 121.74 (Ar), 21.16 (CH₃). EI-TOF-MS m/z = 180 (M).

2-Picolinic acid (**4s**) [CAS: 98-98-6]. ^1H NMR (396 MHz, DMSO- d_6): δ = 13.16 (br-s, 1H, COOH), 8.72 (d, J = 4.0 Hz, 1H, Ar_{H-6}), 8.06 (d, J = 8.0 Hz, 1H, Ar_{H-3}), 7.99 (td, J = 8.0 and 1.2 Hz, 1H, Ar_{H-5}), 7.64 (ddd, J = 8.0, 4.6 and 1.2 Hz, 1H, Ar_{H-4}). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6): δ = 166.21 (C=O), 149.46 (Ar), 148.34 (Ar), 137.55 (Ar), 127.12 (Ar), 124.69 (Ar). EI-TOF-MS m/z = 123 (M).

2-Thiophenecarboxylic acid (**4t**) [CAS: 527-72-0]. ^1H NMR (396 MHz, DMSO- d_6): δ = 13.09 (br-s, 1H, COOH), 7.89 (dd, J = 4.8 and 1.2 Hz, 1H, Ar_{H-5}), 7.74 (d, J = 3.6 Hz, 1H, Ar_{H-3}), 7.20 (dd, J = 5.0 and 3.6 Hz, 1H, Ar_{H-4}). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6): δ = 162.96 (C=O), 134.66 (Ar), 133.32 (Ar), 133.25 (Ar), 128.27 (Ar). EI-TOF-MS m/z = 128 (M).

2-Furancarboxylic acid (**4u**) [CAS: 88-14-2]. ^1H NMR (396 MHz, CDCl₃): δ = 7.68 (d, J = 2.0 Hz, 1H, Ar_{H-5}), 7.35 (d, J = 3.6 Hz, 1H, Ar_{H-3}), 6.57 (dd, J = 3.6 and 2.0 Hz, 1H, Ar_{H-4}). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl₃): δ = 163.40 (C=O), 147.44 (Ar), 143.76 (Ar), 120.15 (Ar), 112.29 (Ar). EI-TOF-MS m/z = 112 (M).

Cinnamic acid (**4v**) [CAS: 140-10-3]. ^1H NMR (500 MHz, CDCl_3): δ = 11.84 (br-s, 1H, COOH), 7.80 (d, J = 16.0 Hz, 1H, $-\underline{\text{C}}\text{H}=\text{CHCOOH}$), 7.57-7.55 (m, 2H, $\text{Ar}_{\text{H-2}}$ and $\text{Ar}_{\text{H-6}}$), 7.42-7.39 (m, 3H, $\text{Ar}_{\text{H-3}}$, $\text{Ar}_{\text{H-4}}$, and $\text{Ar}_{\text{H-5}}$), 6.46 (d, J = 15.5, 1H, $-\text{CH}=\underline{\text{C}}\text{HCOOH}$). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3): δ = 172.61 (C=O), 147.12 ($-\underline{\text{C}}\text{H}=\text{CHCOOH}$), 134.01 (Ar), 130.75 (Ar), 128.95 (Ar), 128.37 (Ar), 117.31 ($-\text{CH}=\underline{\text{C}}\text{HCOOH}$). EI-TOF-MS m/z = 147 (M-1), 148 (M).

Pentanoic acid (**4w**) [CAS: 109-52-4]. ^1H NMR (500 MHz, CDCl_3): δ = 2.36 (t, J = 7.5 Hz, 2H, $\text{CH}_3(\text{CH}_2)_2\underline{\text{C}}\text{H}_2\text{COOH}$), 1.62 (quint, J = 7.5 Hz, 2H, $\text{CH}_3\text{CH}_2\underline{\text{C}}\text{H}_2\text{CH}_2\text{COOH}$), 1.37 (sext, J = 7.5 Hz, 2H, $\text{CH}_3\underline{\text{C}}\text{H}_2(\text{CH}_2)_2\text{COOH}$), 0.92 (t, J = 6.5, 3H, CH_3 -). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ = 180.22 (C=O), 33.78 ($\text{CH}_3(\text{CH}_2)_2\underline{\text{C}}\text{H}_2\text{COOH}$), 26.71 ($\text{CH}_3\text{CH}_2\underline{\text{C}}\text{H}_2\text{CH}_2\text{COOH}$), 22.15 ($\text{CH}_3\underline{\text{C}}\text{H}_2(\text{CH}_2)_2\text{COOH}$), 13.65 (CH_3 -). EI-TOF-MS m/z = 87 (M-15).

Hexanoic acid (**4x**) [CAS: 142-62-1]. ^1H NMR (500 MHz, CDCl_3): δ = 2.35 (t, J = 7.5 Hz, 2H, $-\underline{\text{C}}\text{H}_2\text{COOH}$), 1.64 (quint, J = 7.5 Hz, 2H, $-\underline{\text{C}}\text{H}_2\text{CH}_2\text{COOH}$), 1.36-1.31 (m, 4H, $-(\text{CH}_2)_2$ -), 0.90 (t, J = 6.5, 3H, CH_3 -). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ = 180.08 (C=O), 34.01 ($\text{CH}_3(\text{CH}_2)_3\underline{\text{C}}\text{H}_2\text{COOH}$), 31.19 ($\text{CH}_3\text{CH}_2\underline{\text{C}}\text{H}_2(\text{CH}_2)_2\text{COOH}$), 24.35 ($\text{CH}_3(\text{CH}_2)_2\underline{\text{C}}\text{H}_2\text{CH}_2\text{COOH}$), 22.28 ($\text{CH}_3\underline{\text{C}}\text{H}_2(\text{CH}_2)_3\text{COOH}$), 13.85 (CH_3 -). EI-TOF-MS m/z = 87 (M-29).

Octanoic acid (**4y**) [CAS: 124-07-2]. ^1H NMR (500 MHz, CDCl_3): δ = 2.35 (t, J = 7.5 Hz, 2H, $-\underline{\text{C}}\text{H}_2\text{COOH}$), 1.64 (quint, J = 7.5 Hz, 2H, $-\underline{\text{C}}\text{H}_2\text{CH}_2\text{COOH}$), 1.33-1.28 (m, 8H, $-(\text{CH}_2)_4$ -), 0.88 (t, J = 7.0 Hz, 3H, CH_3 -). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ = 180.35 (C=O), 34.09 ($\text{CH}_3(\text{CH}_2)_5\underline{\text{C}}\text{H}_2\text{COOH}$), 31.61 ($\text{CH}_3\text{CH}_2\underline{\text{C}}\text{H}_2(\text{CH}_2)_4\text{COOH}$), 29.00 ($\text{CH}_3(\text{CH}_2)_2\underline{\text{C}}\text{H}_2(\text{CH}_2)_3\text{COOH}$), 28.87 ($\text{CH}_3(\text{CH}_2)_3\underline{\text{C}}\text{H}_2(\text{CH}_2)_2\text{COOH}$), 24.66 ($\text{CH}_3(\text{CH}_2)_4\underline{\text{C}}\text{H}_2\text{CH}_2\text{COOH}$), 22.56 ($\text{CH}_3\underline{\text{C}}\text{H}_2(\text{CH}_2)_5\text{COOH}$), 14.01 (CH_3 -). EI-TOF-MS m/z = 144 (M).

Decanoic acid (**4z**) [CAS: 334-48-5]. ^1H NMR (500 MHz, CDCl_3): δ = 2.36 (t, J = 7.5 Hz, 2H, $-\underline{\text{C}}\text{H}_2\text{COOH}$), 1.63 (quint, J = 7.5 Hz, 2H, $-\underline{\text{C}}\text{H}_2\text{CH}_2\text{COOH}$), 1.33-1.27 (m, 12H, $-(\text{CH}_2)_6$ -), 0.88 (t, J = 7.0 Hz, 3H, CH_3 -). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ = 179.62 (C=O), 33.99 ($\text{CH}_3(\text{CH}_2)_7\underline{\text{C}}\text{H}_2\text{COOH}$), 31.87 ($\text{CH}_3\text{CH}_2\underline{\text{C}}\text{H}_2(\text{CH}_2)_6\text{COOH}$), 29.40 ($\text{CH}_3(\text{CH}_2)_3\underline{\text{C}}\text{H}_2(\text{CH}_2)_4\text{COOH}$), 29.25 ($\text{CH}_3(\text{CH}_2)_2\underline{\text{C}}\text{H}_2\text{CH}_2\underline{\text{C}}\text{H}_2(\text{CH}_2)_3\text{COOH}$), 29.07 ($\text{CH}_3(\text{CH}_2)_5\underline{\text{C}}\text{H}_2(\text{CH}_2)_2\text{COOH}$),

24.70 ($\text{CH}_3(\text{CH}_2)_6\text{CH}_2\text{CH}_2\text{COOH}$), 22.67 ($\text{CH}_3\text{CH}_2(\text{CH}_2)_7\text{COOH}$), 14.10 (CH_3 -). EI-TOF-MS m/z = 172 (M).

Adipic acid (**4aa**) [CAS : 124-04-9]. ^1H NMR(500 MHz, $\text{DMSO}-d_6$) : δ = 12.00 (br-s, 2H, COOH), 2.23-2.18 (m, 4H, $-\text{CH}_2\text{COOH}$), 1.50 (quint, J = 3.5 Hz, 4H, $-\text{CH}_2\text{CH}_2\text{COOH}$). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, $\text{DMSO}-d_6$): δ = 174.28 (C=O), 33.35 ($-\text{CH}_2\text{COOH}$), 24.00 ($-\text{CH}_2\text{CH}_2\text{COOH}$). EI-MS m/z = 128 (M-18).

Benzaldehyde (**5a**) [CAS: 100-52-7]. ^1H NMR (396 MHz, CDCl_3): δ = 10.02 (s, 1H, CHO), 7.89 (d, J = 7.2 Hz, 2H, $\text{Ar}_{\text{H}2}$ and $\text{Ar}_{\text{H}6}$), 7.64 (tt, J = 7.6 and 1.2 Hz, 1H, $\text{Ar}_{\text{H}4}$), 7.53 (t, J = 7.6 Hz, 2H, $\text{Ar}_{\text{H}3}$ and $\text{Ar}_{\text{H}5}$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ = 192.35 (C=O), 136.35 (Ar), 134.42 (Ar), 129.68 (Ar), 128.94 (Ar). EI-TOF-MS m/z = 105 (M-1), 106 (M).

p-Anisaldehyde (**5b**) [CAS: 123-11-5]. ^1H NMR (396 MHz, CDCl_3): δ = 9.89 (s, 1H, CHO), 7.84 (dt, J = 8.8 and 2.0 Hz, 2H, $\text{Ar}_{\text{H}2}$ and $\text{Ar}_{\text{H}6}$), 7.01 (dt, J = 8.8 and 2.0 Hz, 2H, $\text{Ar}_{\text{H}3}$ and $\text{Ar}_{\text{H}5}$), 3.89 (s, 3H, CH_3). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ = 190.76 (C=O), 164.54 (Ar), 131.91 (Ar), 129.87 (Ar), 114.24 (Ar), 55.51 (CH_3). EI-TOF-MS m/z = 135 (M-1), 136 (M).

p-Tolualdehyde (**5c**) [CAS: 104-87-0]. ^1H NMR (396 MHz, CDCl_3): δ = 9.89 (s, 1H, CHO), 7.70 (d, J = 7.6 Hz, 2H, $\text{Ar}_{\text{H}2}$ and $\text{Ar}_{\text{H}6}$), 7.26 (d, J = 7.6 Hz, 2H, $\text{Ar}_{\text{H}3}$ and $\text{Ar}_{\text{H}5}$), 2.37 (s, 3H, CH_3). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ = 192.02 (C=O), 145.54 (Ar), 134.13 (Ar), 129.83 (Ar), 129.67 (Ar), 21.87 (CH_3). EI-TOF-MS m/z = 119 (M-1), 120 (M).

p-Chlorobenzaldehyde (**5d**) [CAS: 104-88-1]. The yield of the aldehyde (62%) was determined by ^1H -NMR with an internal standard ($\text{CHCl}_2\text{CHCl}_2$: δ = 6.88). The starting alcohol and the carboxylic acid were also obtained in 22% and 14% yield, respectively. ^1H NMR (396 MHz, $\text{DMSO}-d_6$): δ = 10.01 (s, 1H, CHO), 7.92 (dt, J = 8.4 and 2.0 Hz, 2H, $\text{Ar}_{\text{H}2}$ and $\text{Ar}_{\text{H}6}$), 7.64 (dt, J = 8.8 and 2.0 Hz, 2H, $\text{Ar}_{\text{H}3}$ and $\text{Ar}_{\text{H}5}$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ = 191.90 (C=O), 139.49 (Ar), 134.91 (Ar), 131.18 (Ar), 129.33 (Ar). EI-TOF-MS (EI) m/z = 139 (M-1), 140 (M).

Cinnamaldehyde (**5v**) [CAS: 140-55-2]. ^1H NMR (396 MHz, CDCl_3): δ = 9.71 (d, J = 8.0 Hz, 1H, CHO), 7.59-7.57 (m, 2H, $\text{Ar}_{\text{H}2}$ and $\text{Ar}_{\text{H}6}$), 7.49 (d, J = 16.0 Hz, 2H, $-\text{CH}=\text{CHCOOH}$), 7.46-7.43 (m,

3H, Ar_{H-3}, Ar_{H-4}, and Ar_{H-5}), 6.73 (dd, $J = 16.0$ and 7.6 Hz, 1H, -CH=CHCOOH). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): $\delta = 193.77$ (C=O), 152.85 (-CH=CHCOOH), 133.95 (Ar), 131.27 (-CH=CHCOOH), 129.09 (Ar), 128.55 (Ar), 128.48 (Ar). EI-TOF-MS $m/z = 131$ (M-1), 132 (M).

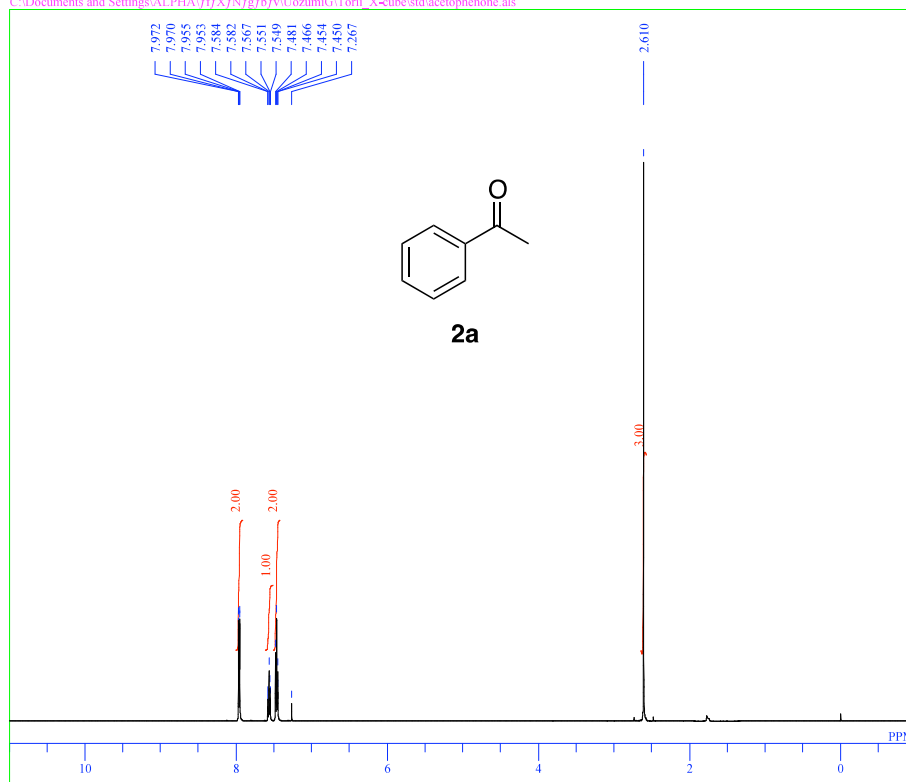
3,6,9,12-Tetraoxatetracosanoic acid (**8a**) [CAS: 20858-25-7]: ^1H NMR (500 MHz, CDCl_3): $\delta = 4.15$ (s, 2H, -OCH₂COOH), 3.78-3.77 (m, 2H, -OCH₂CH₂OCH₂COOH), 3.70-3.60 (m, 8H, -OCH₂CH₂O-), 3.47 (t, $J = 7.0$ Hz, 2H, C₁₂H₂₅OCH₂CH₂O-), 1.62-1.58 (m, 2H, C₁₁H₂₃CH₂O-), 1.33-1.21 (m, 20H, -C₁₀H₂₀-), 0.88 (t, $J = 7.0$ Hz, 3H, CH₃-). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3): $\delta = 172.20$ (C=O), 71.61 (-CH₂COOH), 71.42 (-OCH₂CH₂O-), 70.62 (-OCH₂CH₂O-), 70.39 (-OCH₂CH₂O-), 70.37 (-OCH₂CH₂O-), 70.29 (-OCH₂CH₂O-), 70.06 (-OCH₂CH₂O-), 69.10 (-OCH₂CH₂), 31.88 (-OCH₂CH₂CH₂-), 29.63 (-CH₂CH₂CH₂-), 29.60 (-CH₂CH₂CH₂-), 29.57 (-CH₂CH₂CH₂-), 29.42 (-CH₂CH₂CH₂-), 29.40 (-CH₂CH₂CH₂-), 29.32 (-CH₂CH₂CH₂-), 25.99 (-CH₂CH₂CH₂-), 22.65 (-CH₂CH₂CH₂-), 22.65 (CH₃CH₂-), 14.08 (CH₃-). ESI-TOF-MS (neg) $m/z = 375$ (M⁻).

Detergent **8b** ^1H NMR (500 MHz, CDCl_3): $\delta = 4.17$ -4.15 (s x 4), 3.77-3.73 (m), 3.71-3.63 (m), 3.61-3.58 (m), 3.48-3.44 (m), 1.57 (quint, $J = 7.0$ Hz, CH₃CH₂CH₂-), 1.32-1.26 (m), 0.88 (t, $J = 7.0$ Hz, CH₃). $^{13}\text{C}\{^1\text{H}\}$ NMR (100MHz, CDCl_3): $\delta = 172.02$ (C=O), 170.49 (C=O), 71.71, 71.64, 71.60, 71.58, 71.55, 71.50, 71.34, 70.98, 70.92, 70.88, 70.71, 70.58, 70.55, 70.53, 70.50, 70.46, 70.44, 70.33, 70.30, 70.20, 70.18, 70.14, 70.04, 70.00, 69.95, 69.81, 69.32, 69.24, 68.95, 68.79, 68.49, 63.79, 33.83, 91.92, 29.68, 29.65, 29.61, 29.59, 29.50, 29.43, 29.36, 29.32, 29.27, 29.10, 26.08, 26.04, 25.99, 25.84, 24.76, 22.70, 14 (pos) $m/z = 333$ ([M_(n=3) + 1]⁺), 377 ([M_(n=4) + 1]⁺), 421 ([M_(n=5) + 1]⁺), 465 ([M_(n=5) + 1] .14. FAB-MS ⁺).

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of acetophenone (**2a**).

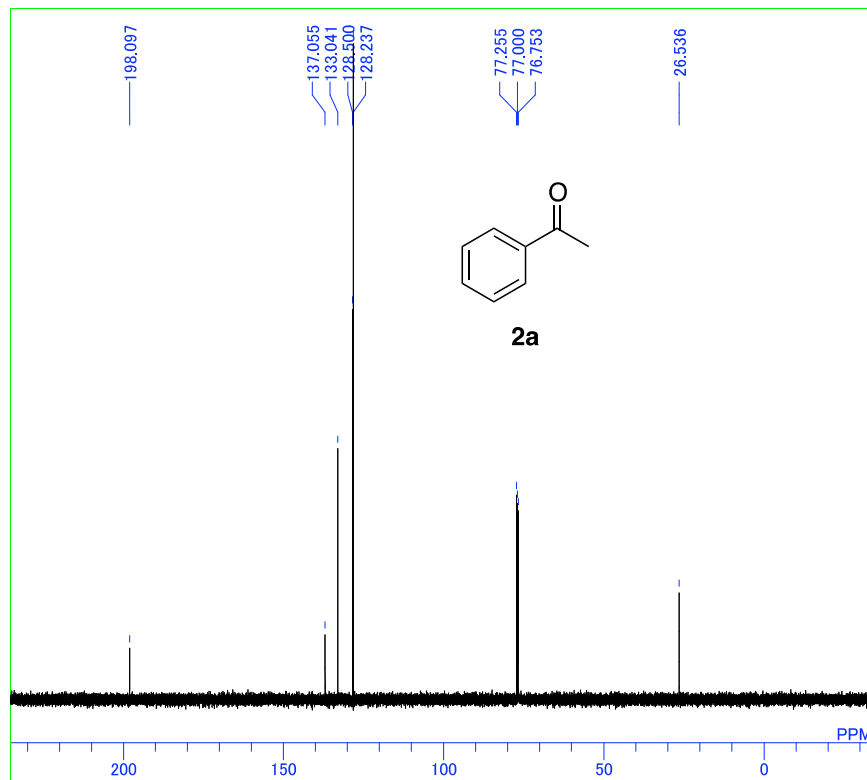
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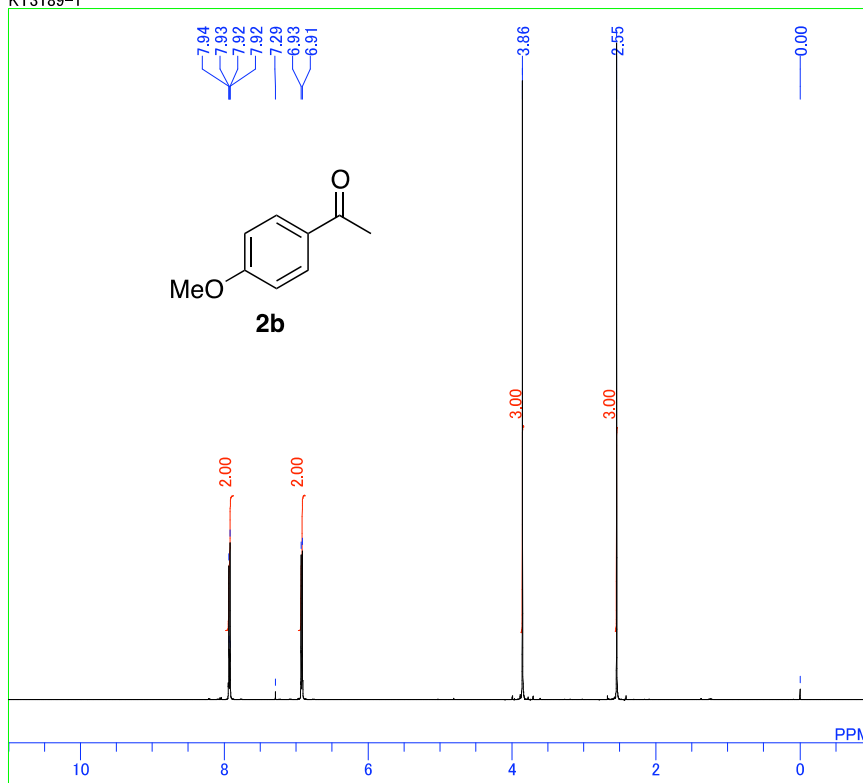
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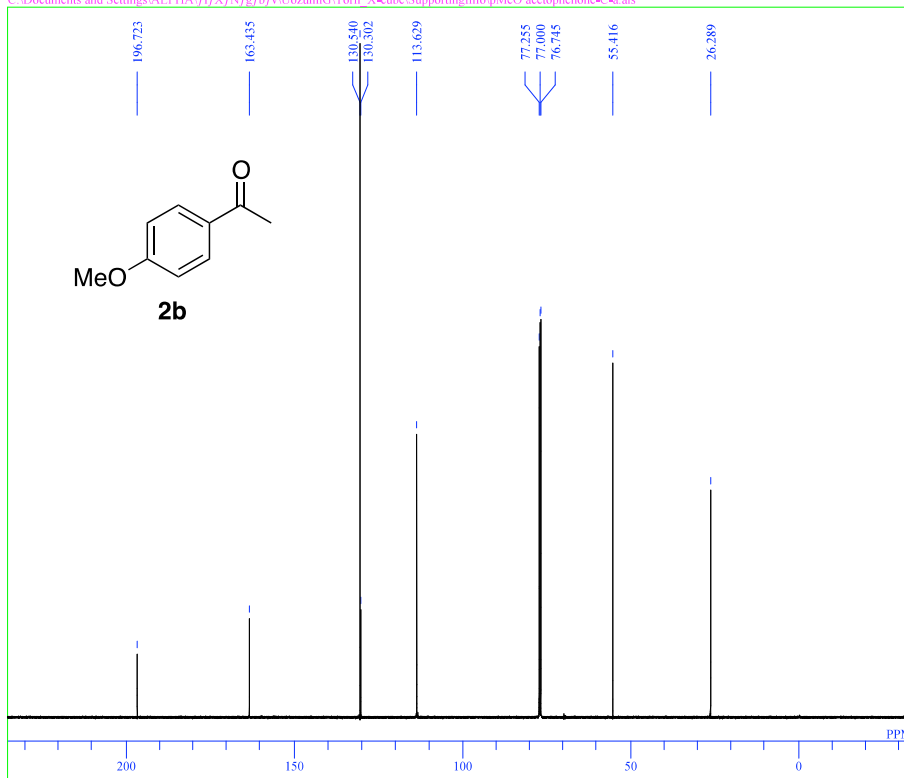
^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 4'-methoxyacetophenone (**2b**).

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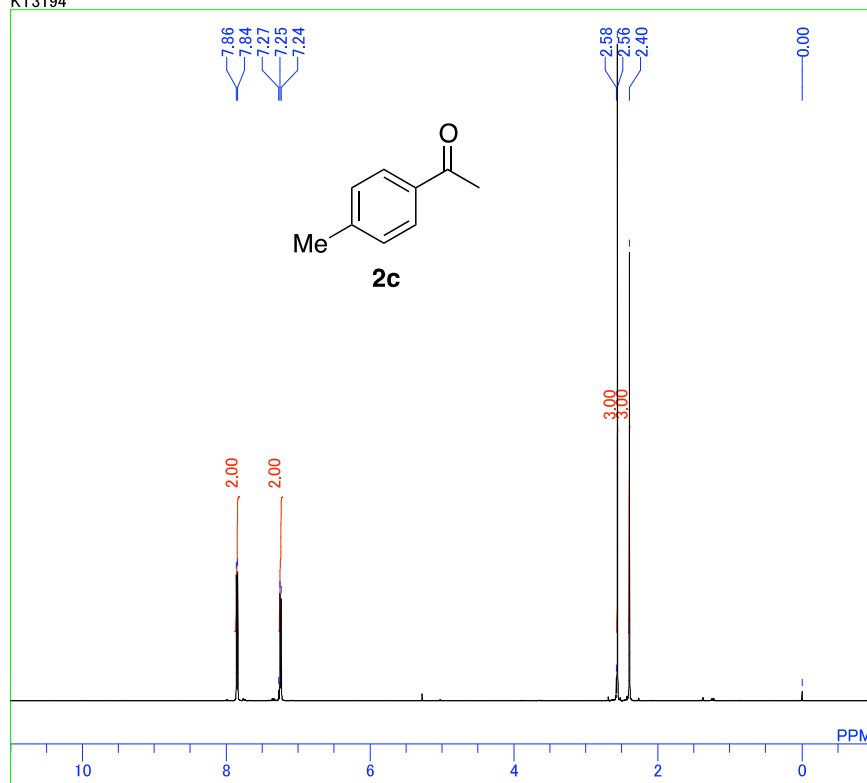
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^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 4'-methylacetophenone (**2c**).

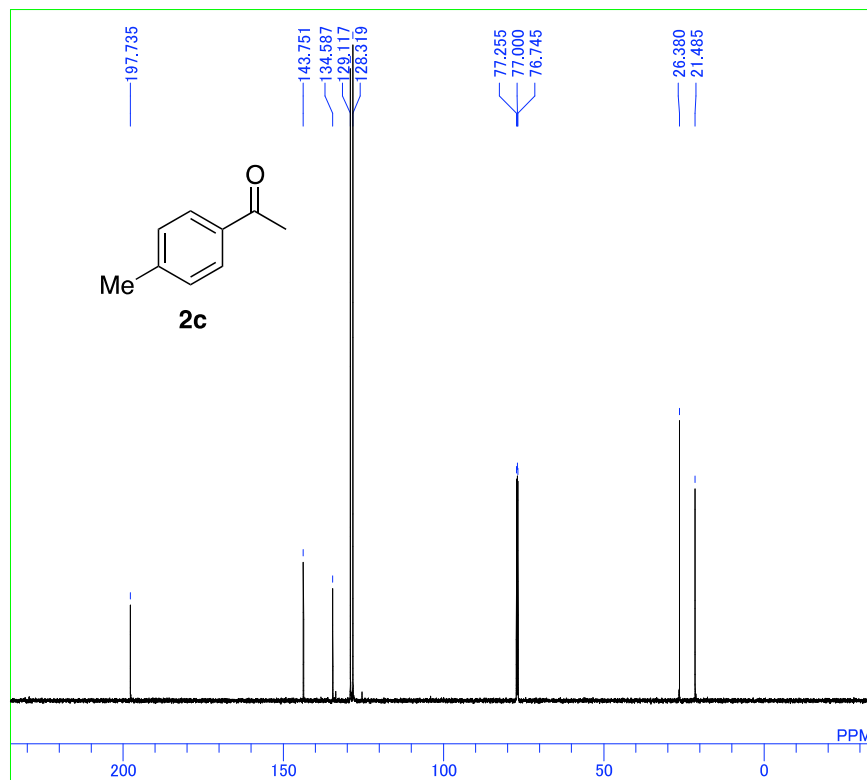
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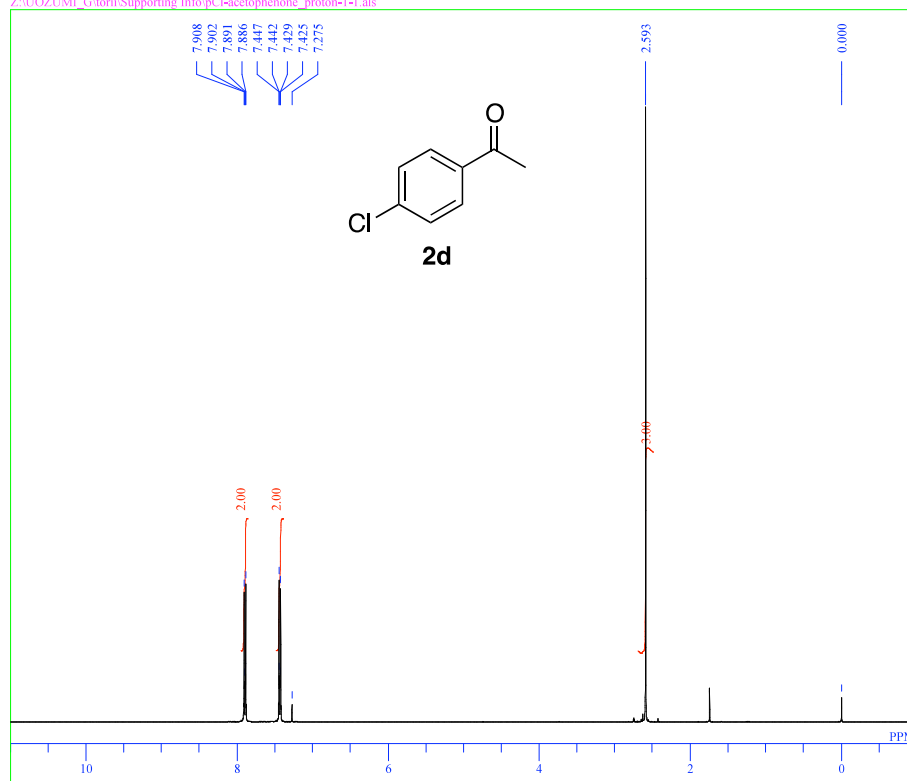
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^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 4'-chloroacetophenone (**2d**).

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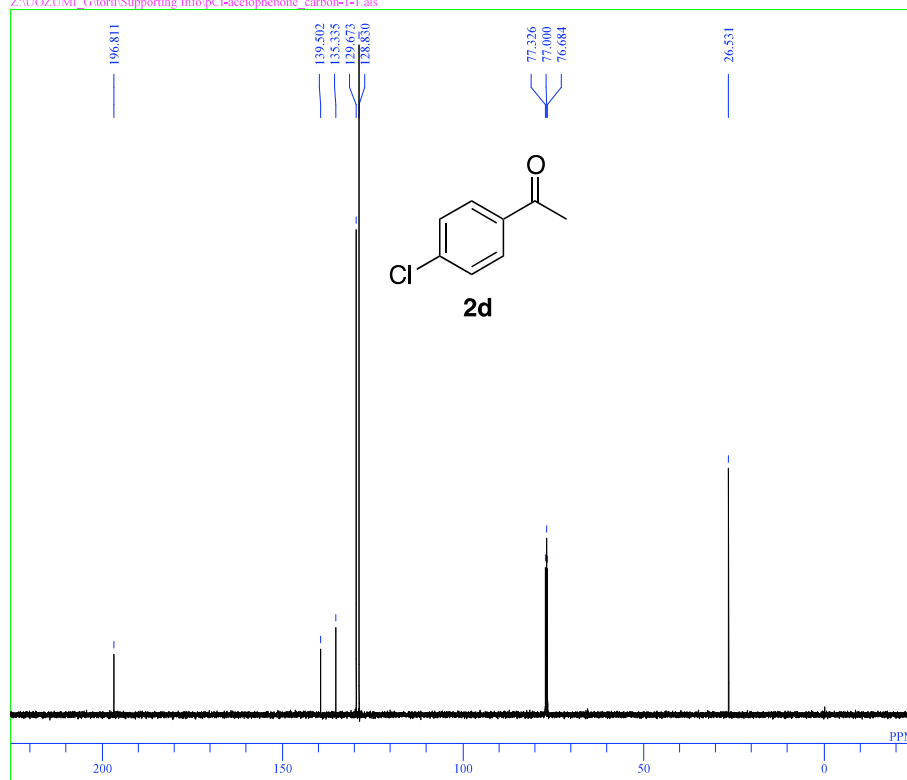
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 EXMOD proton.jsp
 OBFREQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.12 usec
 IRNUC ^1H
 CTEMP 19.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.40 Hz
 RGAIN 30

single pulse decoupled gated NOE

Z:\UOZUMI_G\oria\Supporting Info\pCl-acetophenone_carbon-1-1.als

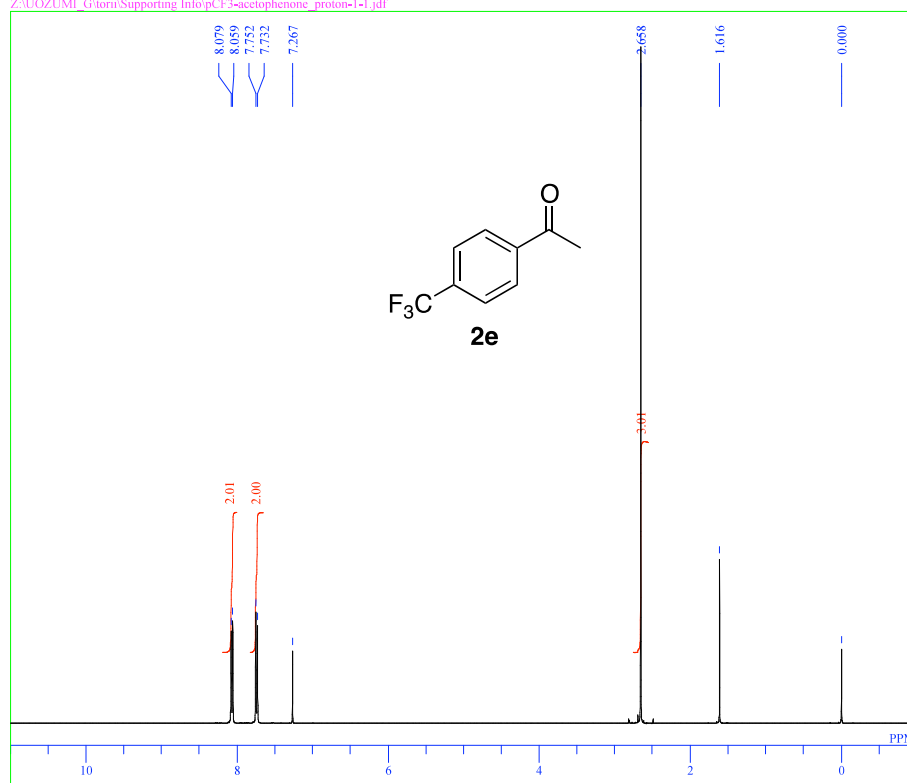


DFILE pCl-acetophenone_carbon-1-1.als
 COMNT single pulse decoupled gated NOE
 DATIM 2013-06-10 10:19:46
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 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 156
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 IRNUC ^1H
 CTEMP 19.2 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.40 Hz
 RGAIN 58

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 4'-(trifluoromethyl)acetophenone (**2e**).

single_pulse

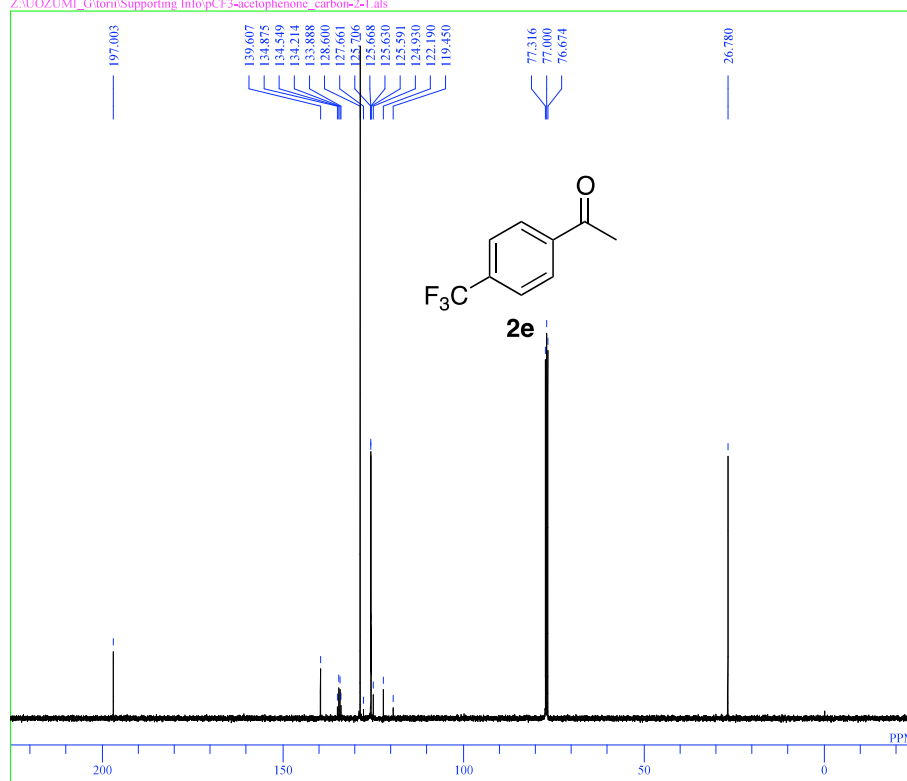
Z:\UOZUMI_G\torii\Supporting Info\pCF3-acetophenone_proton-1-1.jdf



DFILE pCF3-acetophenone_proton-1-1.jdf
 COMNT single_pulse
 DATIM 2013-06-10 10:33:35
 OBNUC ^1H
 EXMOD proton.jxp
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 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.12 usec
 IRNUC ^1H
 CTEMP 19.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.50 Hz
 RGAIN 40

single pulse decoupled gated NOE

Z:\UOZUMI_G\torii\Supporting Info\pCF3-acetophenone_carbon-2-1.als

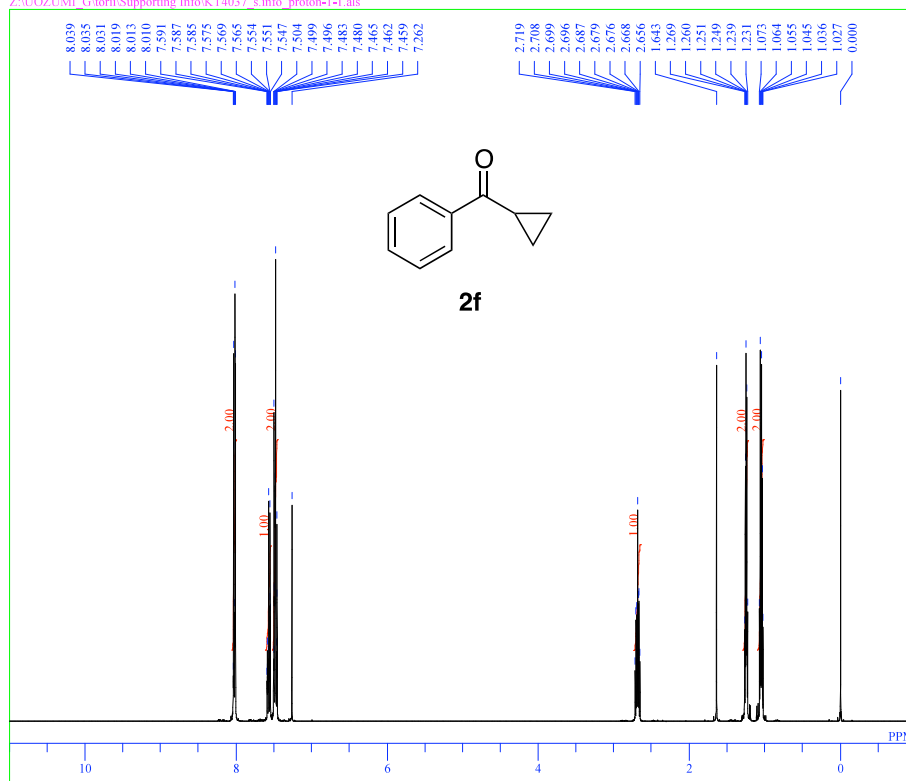


DFILE pCF3-acetophenone_carbon-2-1.als
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 OBNUC ^{13}C
 EXMOD carbon.jxp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 722
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 IRNUC ^1H
 CTEMP 19.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 60

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of cyclopropyl phenyl ketone (**2f**).

single_pulse

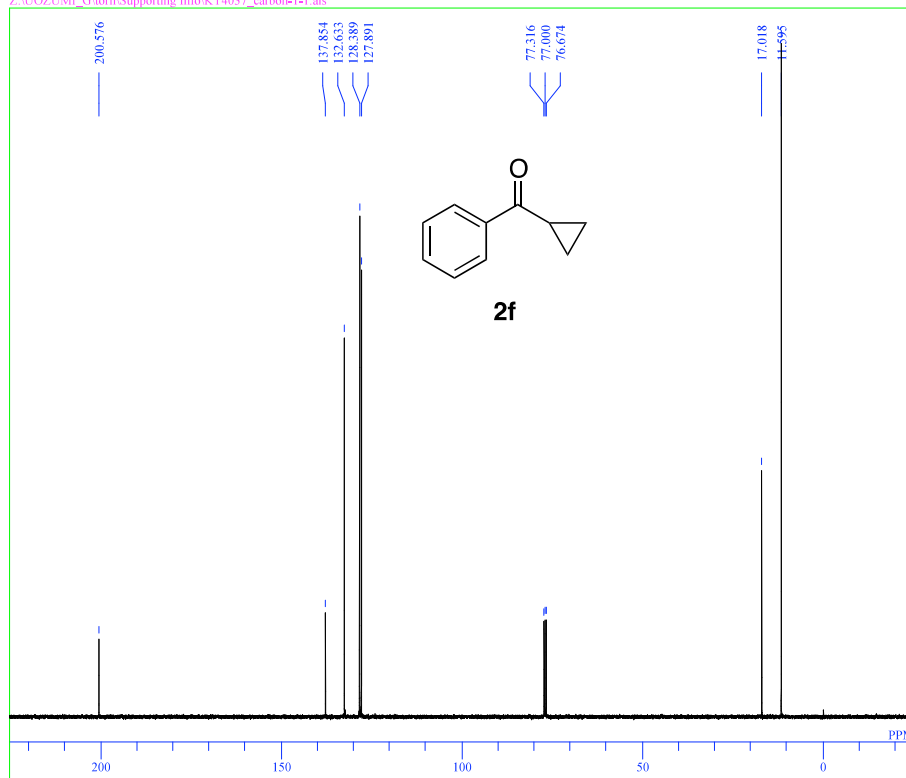
Z:\UOZUMI_G\Torii\Supporting Info\KT4037_s_info_proton-1-1.als



DFILE KT4037_s_info_proton-1-1.als
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 DATIM 2013-06-24 09:46:04
 OBNUC ^1H
 EXMOD proton.jsp
 OBFREQ 395.88 MHz
 OBSET 6.28 kHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 16
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.12 usec
 IRNUC ^1H
 CTEMP 19.3 c
 SLVNT CDCL₃
 EXREF 0.00 ppm
 BF 0.25 Hz
 RGAIN 34

single pulse decoupled gated NOE

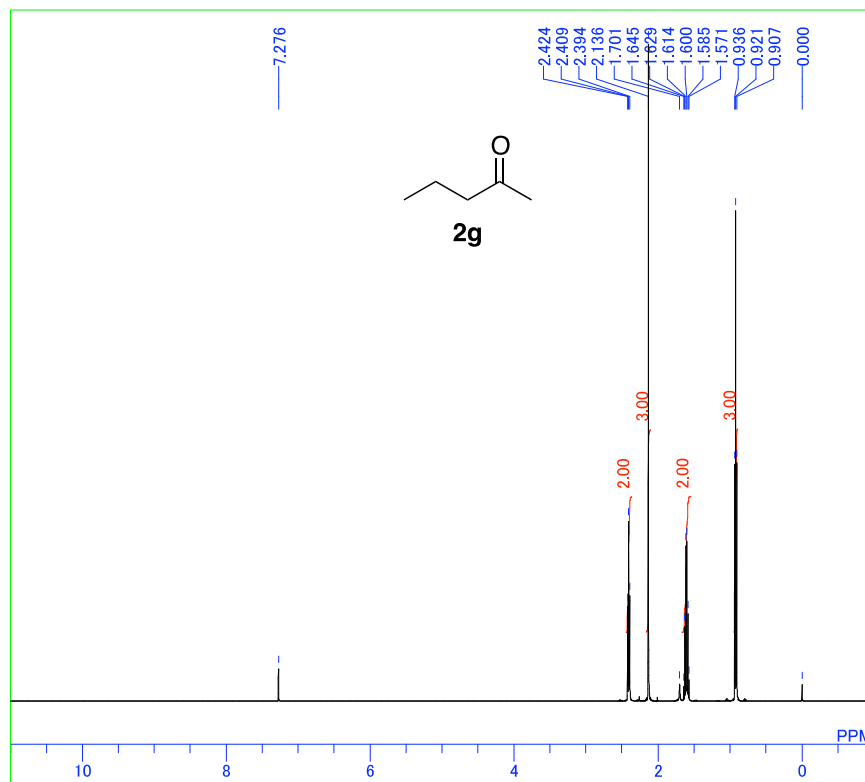
Z:\UOZUMI_G\Torii\Supporting Info\KT4037_carbon-1-1.als



DFILE KT4037_carbon-1-1.als
 COMNT single pulse decoupled gated NOE
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 EXMOD carbon.jsp
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 OBSET 5.13 kHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 195
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 IRNUC ^1H
 CTEMP 19.2 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 0.60 Hz
 RGAIN 60

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 2-pentanone (**2g**).

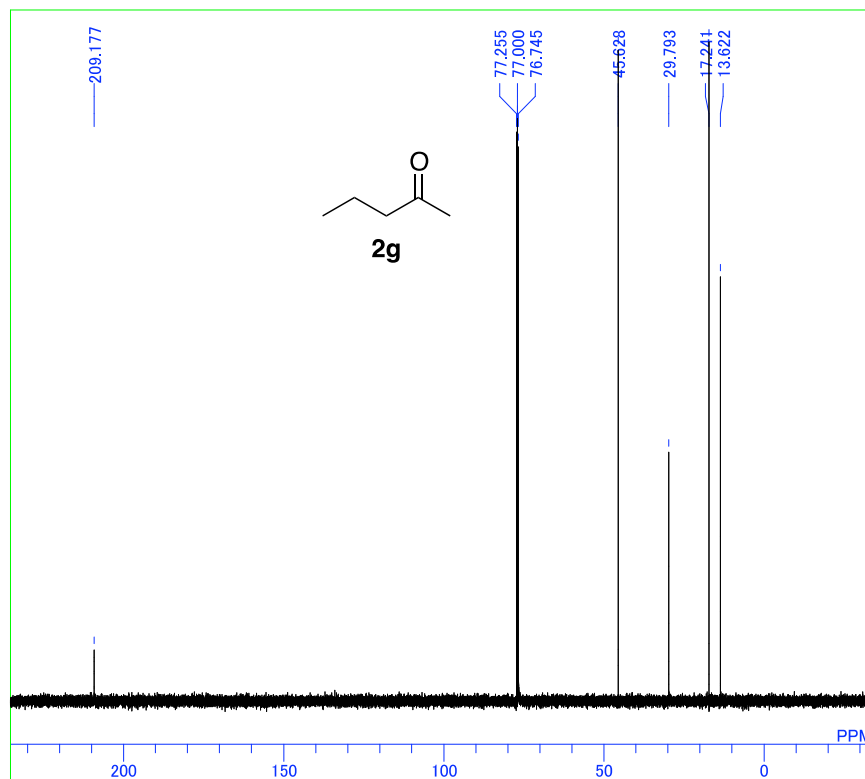
C:\Documents and Settings\ALPHA\1-K-4-2\UozumiG\Torii_X-cube\std\2-pentanone.als



```

MENUMF non_th5atfTH5ATFG2_22-pentanone
OBNUC 1H
OBFRQ 500.00 MHz
OBFIN 162410.00 Hz
PW1 6.40 usec
DEADT 56.80 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 16384
SAMPO 16384
TIMES 8
DUMMY 1
FREQU 10000.00 Hz
FILTR 5000 Hz
DELAY 40.00 usec
ACQTM 1.6384 sec
PD 2.0000 sec
ADBIT 16
RGAIN 16
BF 0.23 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\Documents and Settings\ALPHA\
SHMFL TH5ATFG2_22-pentanone
LKFIN 70334.0 Hz
LKLEV 180
LGAIN 20
LKPHS 210
LKSIG 577
CSPED 17 Hz
FILDC
FILDF
    
```

C:\Documents and Settings\ALPHA\1-K-4-2\UozumiG\Torii_X-cube\std\2-pentanone_13C.als

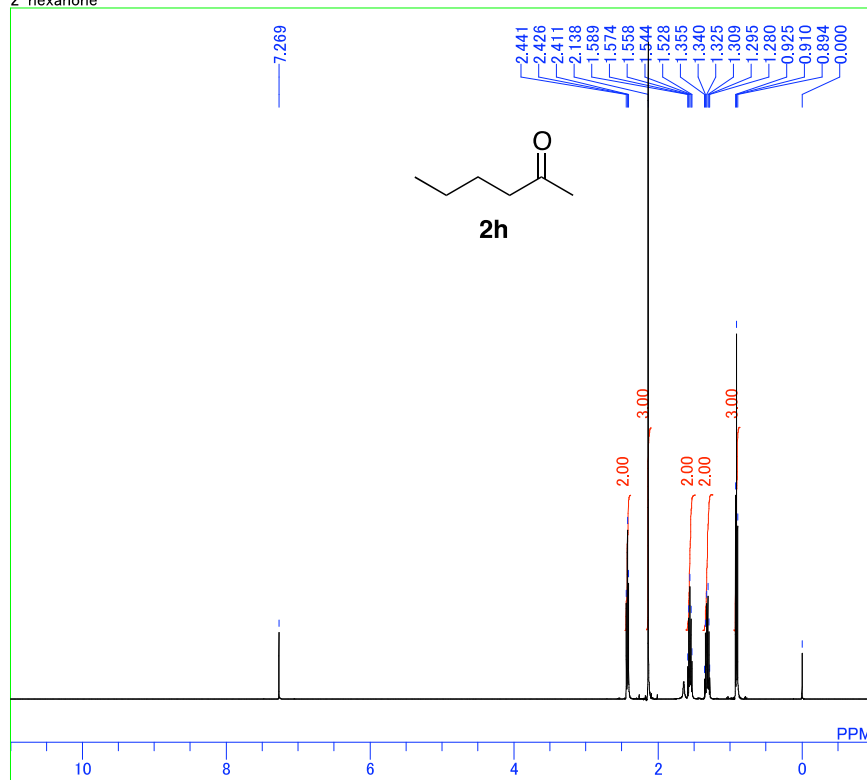


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MENUMF bcm_th5atfTH5ATFG2_2acetophenone
OBNUC 13C
OBFRQ 125.65 MHz
OBFIN 127958.00 Hz
PW1 5.75 usec
DEADT 10.00 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 32768
SAMPO 32768
TIMES 2000
DUMMY 1
FREQU 33898.30 Hz
FILTR 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 1.0000 sec
ADBIT 16
RGAIN 25
BF 0.25 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\Documents and Settings\ALPHA\
SHMFL TH5ATFG2_2acetophenone
LKFIN 70334.0 Hz
LKLEV 180
LGAIN 20
LKPHS 210
LKSIG 572
CSPED 17 Hz
FILDC
FILDF
    
```

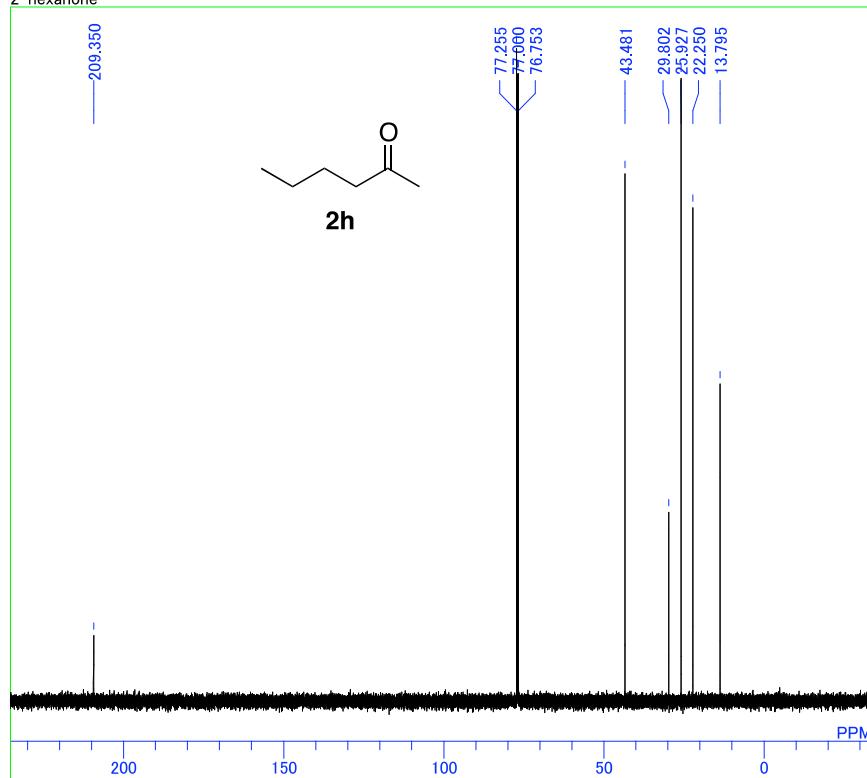
^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 2-hexanone (**2h**).

C:\Documents and Settings\ALPHA\1-K-4-2\UozumiG\Torii_X-cube\std\2-hexanone.als
2-hexanone



MENUF non_th5atfTH5ATFG2_22-hexanone
 1H
 OBNUC 500.00 MHz
 OBFRQ 162410.00 Hz
 OBFIN 6.40 usec
 DEADT 56.80 usec
 PREDL 0.20000 msec
 INIWT 10.0000 msec
 POINT 16384
 SAMPO 16384
 TIMES 16
 DUMMY 1
 FREQU 10000.00 Hz
 FILTR 5000 Hz
 DELAY 40.00 usec
 ACQTM 1.6384 sec
 PD 2.0000 sec
 ADBIT 16
 RGAIN 17
 BF 0.00 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD SINGL
 EXPCM Single pulse
 IRNUC 1H
 IRFRQ 500.00 MHz
 IRFIN 162410.00 Hz
 IRRPW 50 usec
 IRATN 511
 DFILE C:\Documents and Settings\ALPHA\1-K-4-2\UozumiG\Torii_X-cube\std\2-hexanone.als
 SHMFL TH5ATFG2_22-hexanone
 LKFIN 70334.0 Hz
 LKLEV 180
 LGAIN 26
 LKPHS 210
 LKSIG 2772
 CSPED 16 Hz
 FILDC
 FILDF

C:\Documents and Settings\ALPHA\1-K-4-2\UozumiG\Torii_X-cube\std\2-hexanone_13Cfid.als
2-hexanone

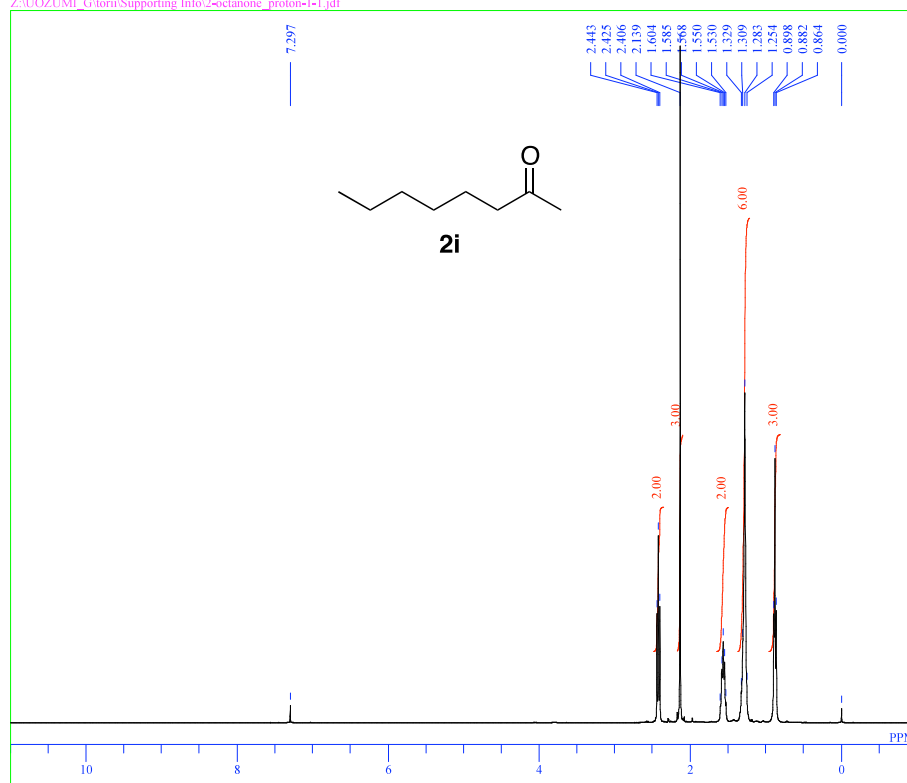


MENUF bcm_th5atfTH5ATFG2_22-hexanone
 13C
 OBNUC 125.65 MHz
 OBFRQ 127958.00 Hz
 OBFIN 5.75 usec
 DEADT 10.00 usec
 PREDL 0.20000 msec
 INIWT 10.0000 msec
 POINT 32768
 SAMPO 32768
 TIMES 2000
 DUMMY 1
 FREQU 33898.30 Hz
 FILTR 16950 Hz
 DELAY 11.80 usec
 ACQTM 0.9667 sec
 PD 1.0000 sec
 ADBIT 16
 RGAIN 27
 BF 0.23 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD SINGL
 EXPCM Single pulse
 IRNUC 1H
 IRFRQ 500.00 MHz
 IRFIN 162410.00 Hz
 IRRPW 50 usec
 IRATN 511
 DFILE C:\Documents and Settings\ALPHA\1-K-4-2\UozumiG\Torii_X-cube\std\2-hexanone.als
 SHMFL TH5ATFG2_22-hexanone
 LKFIN 70334.0 Hz
 LKLEV 180
 LGAIN 20
 LKPHS 210
 LKSIG 561
 CSPED 17 Hz
 FILDC
 FILDF

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 2-octanone (**2i**).

single_pulse

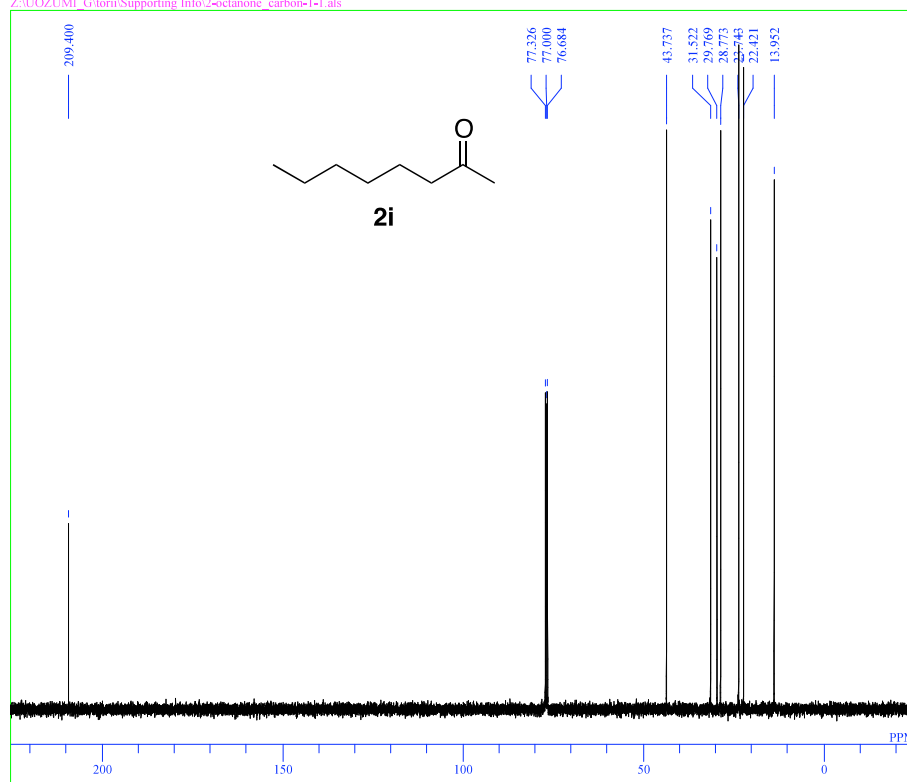
Z:\UOZUMI_G\torii\Supporting Info\2-octanone_proton-1-1.jdf



DFILE 2-octanone_proton-1-1.jdf
 COMNT single_pulse
 DATIM 2013-06-10 11:10:47
 OBNUC 1H
 EXMOD proton.jsp
 OBFREQ 395.88 MHz
 OBSSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.12 usec
 IRNUC 1H
 CTEMP 18.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 1.00 Hz
 RGAIN 22

single pulse decoupled gated NOE

Z:\UOZUMI_G\torii\Supporting Info\2-octanone_carbon-1-1.als

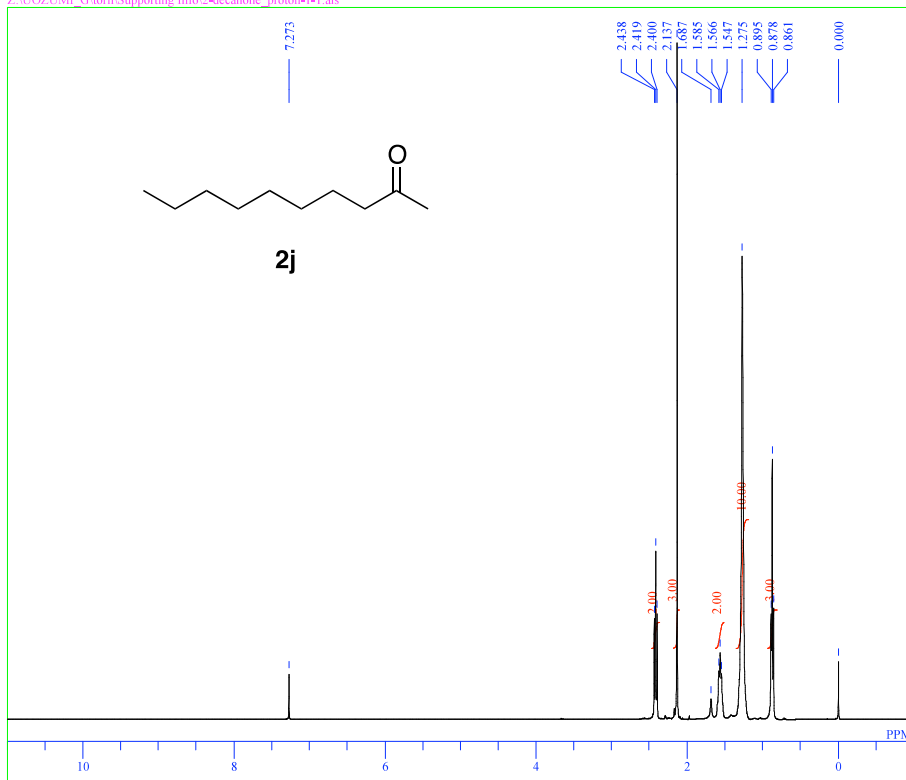


DFILE 2-octanone_carbon-1-1.als
 COMNT single pulse decoupled gated NOE
 DATIM 2013-06-10 14:06:12
 OBNUC ^{13}C
 EXMOD carbon.jsp
 OBFREQ 99.55 MHz
 OBSSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 74
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 IRNUC 1H
 CTEMP 19.7 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 60

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 2-decanone (**2j**).

single_pulse

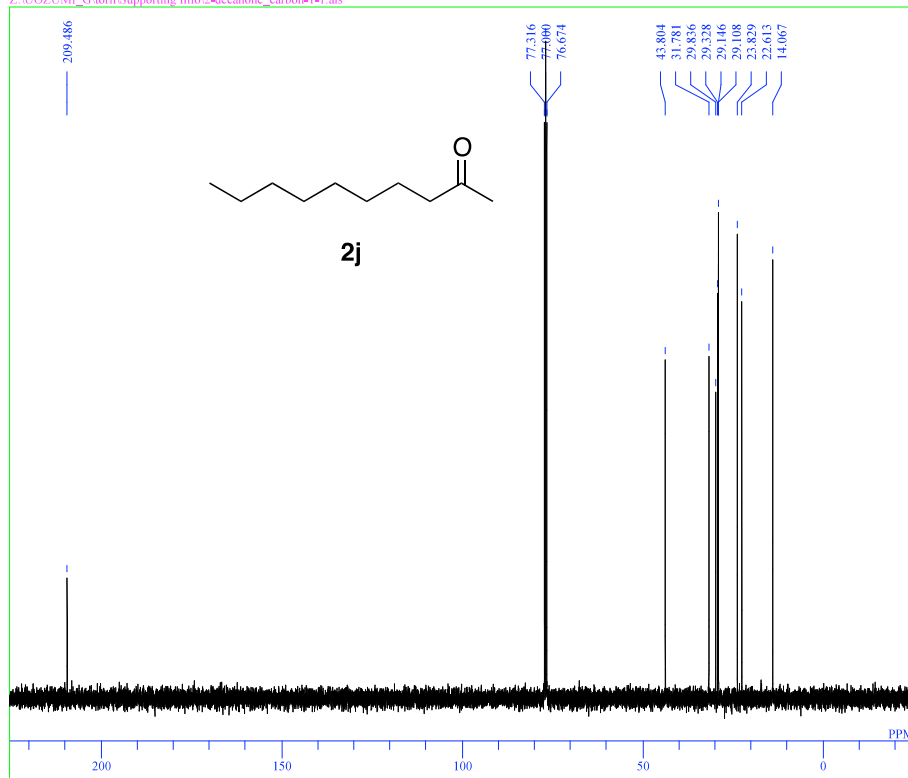
Z:\UOZUMI_G\torii\Supporting Info\2-decanone_proton-1-1.als



DFILE 2-decanone_proton-1-1.als
 COMNT single_pulse
 DATIM 2015-06-10 14:25:48
 OBNUC ^1H
 EXMOD proton.jsp
 OBFREQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.12 usec
 IRNUC ^1H
 CTEMP 20.0 c
 SLVNT CDCL₃
 EXREF 0.00 ppm
 BF 1.00 Hz
 RGAIN 30

single pulse decoupled gated NOE

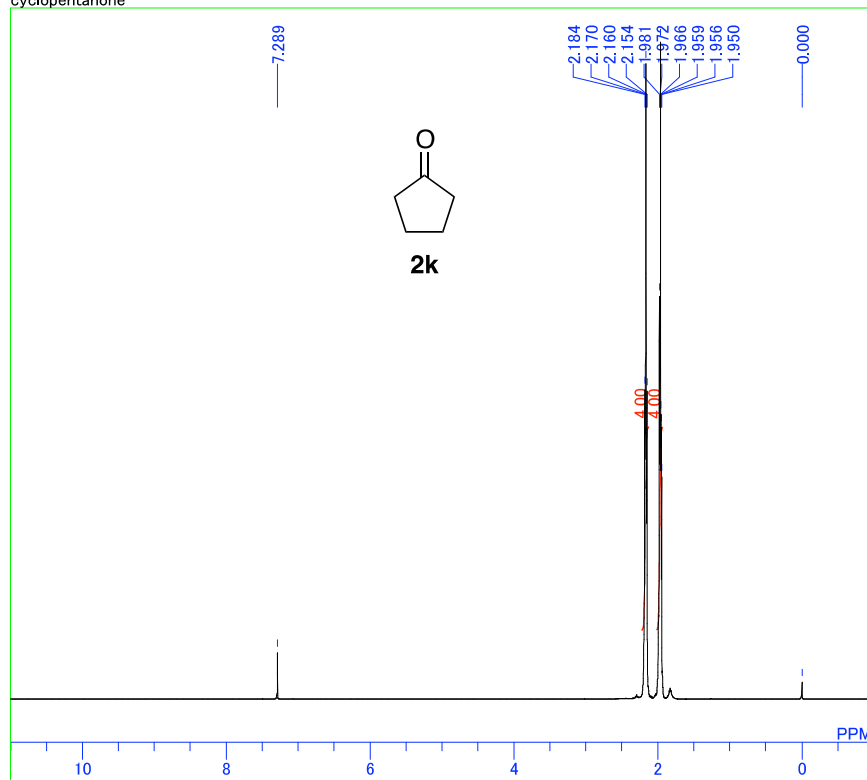
Z:\UOZUMI_G\torii\Supporting Info\2-decanone_carbon-1-1.als



DFILE 2-decanone_carbon-1-1.als
 COMNT single pulse decoupled gated NOE
 DATIM 2013-06-10 14:27:14
 OBNUC ^{13}C
 EXMOD carbon.jsp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 121
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 IRNUC ^1H
 CTEMP 19.7 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 60

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of cyclopentanone (**2k**).

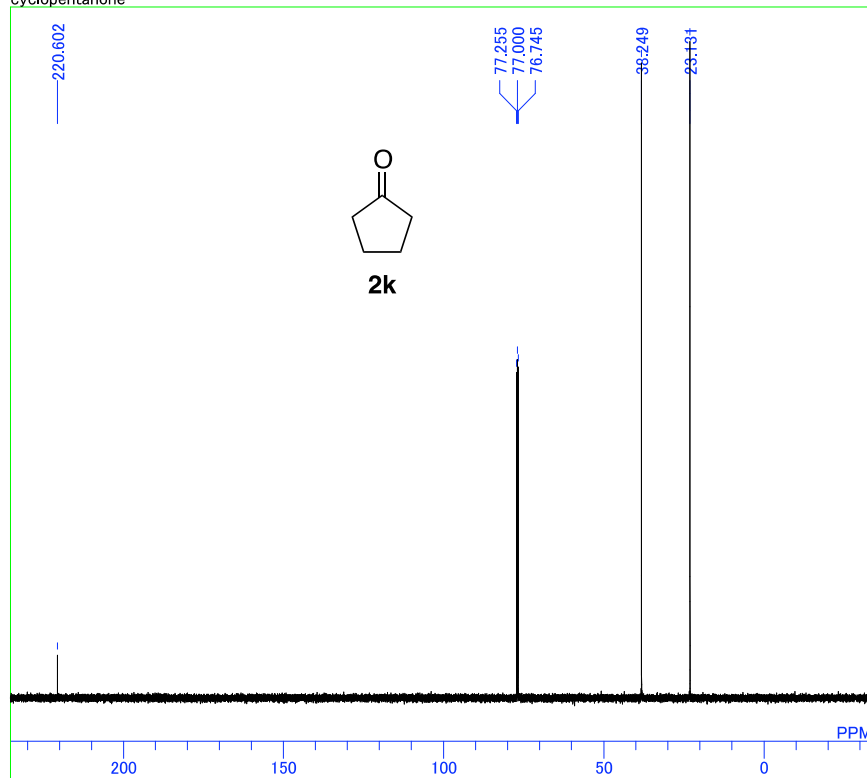
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cyclopentanone



```

MENUF non_th5atfTH5ATFG2_cyclopentanor
OBNUC 1H
OBFRQ 500.00 MHz
OBFIN 162410.00 Hz
PW1 6.40 usec
DEADT 56.80 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 16384
SAMPO 16384
TIMES 16
DUMMY 1
FREQU 10000.00 Hz
FILTR 5000 Hz
DELAY 40.00 usec
ACQTM 1.6384 sec
PD 2.0000 sec
ADBIT 16
RGAIN 14
BF 0.23 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\Documents and Settings\ALPHA\
SHMFL TH5ATFG2_cyclopentanone
LKFIN 70334.0 Hz
LKLEV 180
LGAIN 23
LKPHS 210
LKSIG 936
CSPED 13 Hz
FILDC
FILDF
    
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cyclopentanone

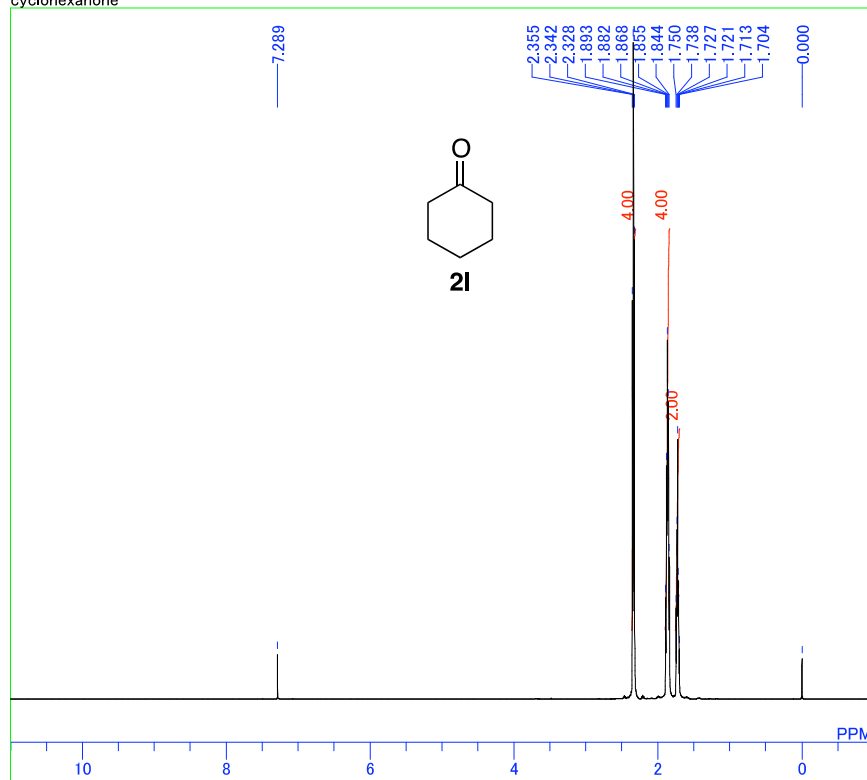


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MENUF bcm_th5atfTH5ATFG2_cyclopentanor
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OBFIN 127958.00 Hz
PW1 5.75 usec
DEADT 10.00 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 32768
SAMPO 32768
TIMES 2000
DUMMY 1
FREQU 33898.30 Hz
FILTR 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 1.0000 sec
ADBIT 16
RGAIN 25
BF 0.25 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\Documents and Settings\ALPHA\
SHMFL TH5ATFG2_cyclopentanone
LKFIN 70334.0 Hz
LKLEV 180
LGAIN 23
LKPHS 210
LKSIG 1176
CSPED 14 Hz
FILDC
FILDF
    
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^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of cyclohexanone (**21**).

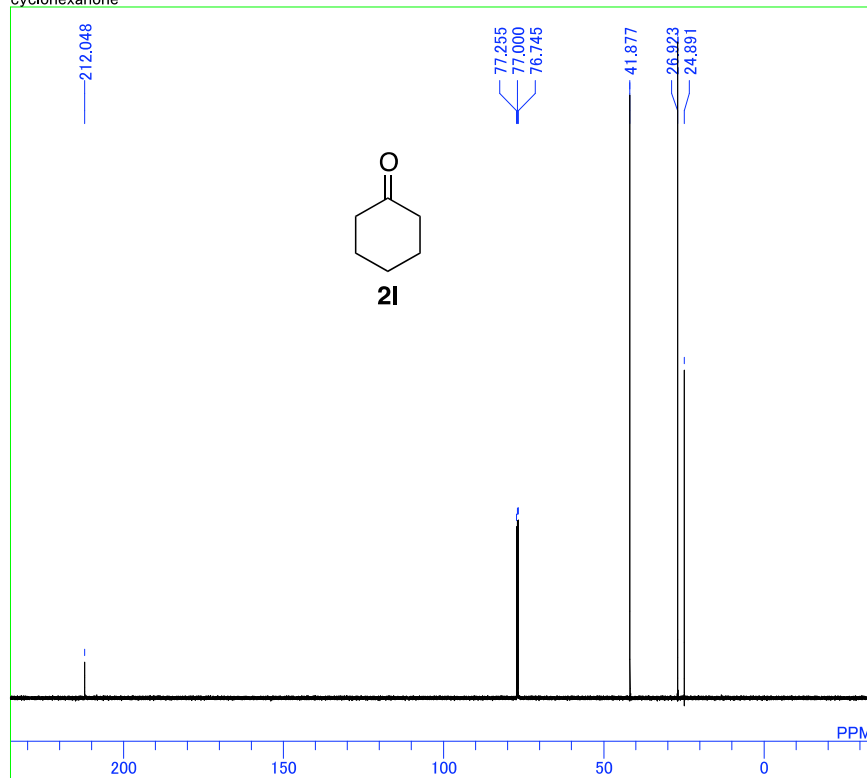
C:\Documents and Settings\ALPHA\1-K-4-2\UozumiG\Torii_X-cube\std\cyclohexanone.als
cyclohexanone



```

MENUM non_th5atfTH5ATFG2_cyclohexanone
OBNUC 1H
OBFRQ 500.00 MHz
OBFIN 162410.00 Hz
PW1 6.40 usec
DEADT 56.80 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 16384
SAMPO 16384
TIMES 16
DUMMY 1
FREQU 10000.00 Hz
FILTR 5000 Hz
DELAY 40.00 usec
ACQTM 1.6384 sec
PD 2.0000 sec
ADBIT 16
RGAIN 13
BF 0.23 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\Documents and Settings\ALPHA\
SHMFL TH5ATFG2_cyclohexanone
LKFIN 70334.0 Hz
LKLEV 180
LGAIN 21
LKPHS 210
LKSIG 737
CSPED 16 Hz
FILDC
FILDF
    
```

C:\Documents and Settings\ALPHA\1-K-4-2\UozumiG\Torii_X-cube\std\cyclohexanone-C.als
cyclohexanone

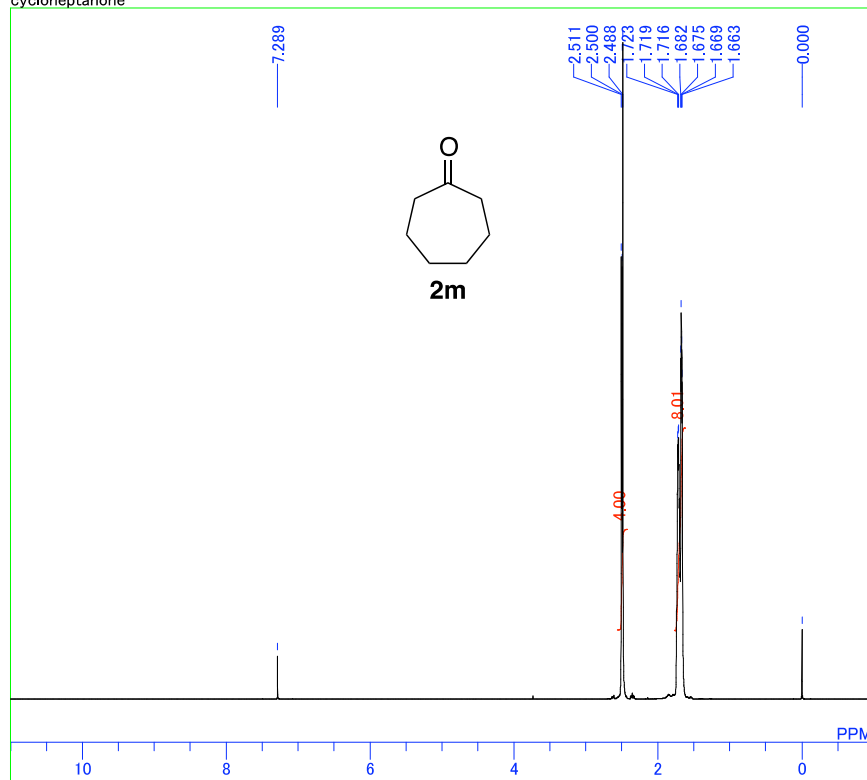


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MENUM bcm_th5atfTH5ATFG2_cyclohexanone
OBNUC 13C
OBFRQ 125.65 MHz
OBFIN 127958.00 Hz
PW1 5.75 usec
DEADT 10.00 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 32768
SAMPO 32768
TIMES 2000
DUMMY 1
FREQU 33898.30 Hz
FILTR 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 1.0000 sec
ADBIT 16
RGAIN 27
BF 0.25 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\Documents and Settings\ALPHA\
SHMFL TH5ATFG2_cyclohexanone
LKFIN 70334.0 Hz
LKLEV 180
LGAIN 21
LKPHS 210
LKSIG 697
CSPED 15 Hz
FILDC
FILDF
    
```

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of cycloheptanone (**2m**).

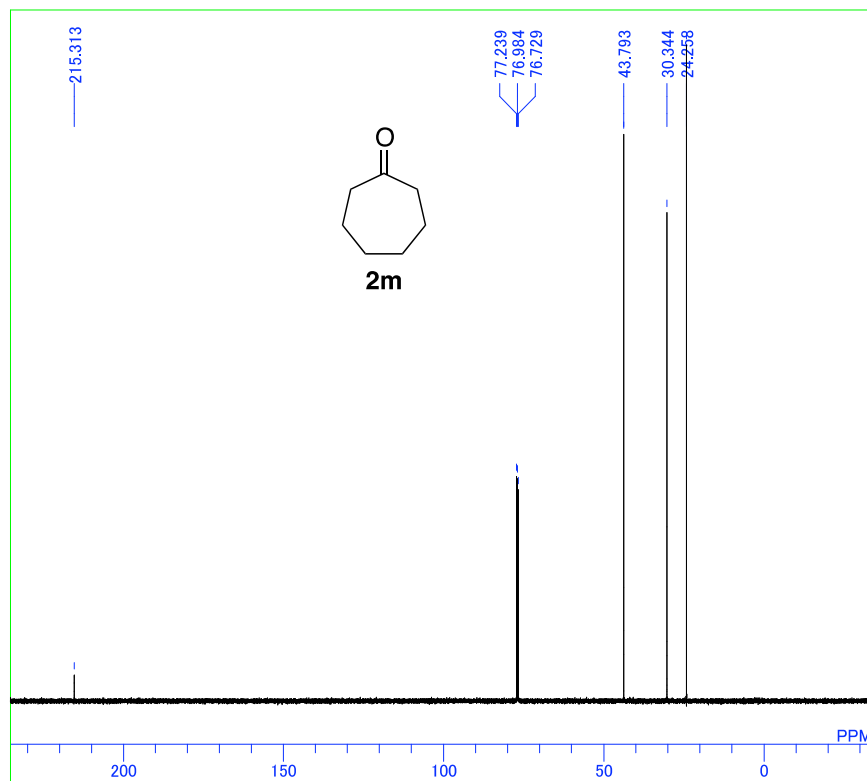
C:\Documents and Settings\ALPHA\1-K-4-2\UozumiG\Torii_X-cube\std\cycloheptanone.als
cycloheptanone



```

MENUF non_th5atfTH5ATFG2_cycloheptanon
OBNUC 1H
OBFRQ 500.00 MHz
OBFIN 162410.00 Hz
PW1 6.40 usec
DEADT 56.80 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 16384
SAMPO 16384
TIMES 16
DUMMY 1
FREQU 10000.00 Hz
FILTR 5000 Hz
DELAY 40.00 usec
ACQTM 1.6384 sec
PD 2.0000 sec
ADBIT 16
RGAIN 13
BF 0.23 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\Documents and Settings\ALPHA\
SHMFL TH5ATFG2_cycloheptanone
LKFIN 70334.0 Hz
LKLEV 180
LGAIN 20
LKPHS 210
LKSIG 557
CSPED 16 Hz
FILDC
FILDF
    
```

C:\Documents and Settings\ALPHA\1-K-4-2\UozumiG\Torii_X-cube\std\cycloheptanone-C.als

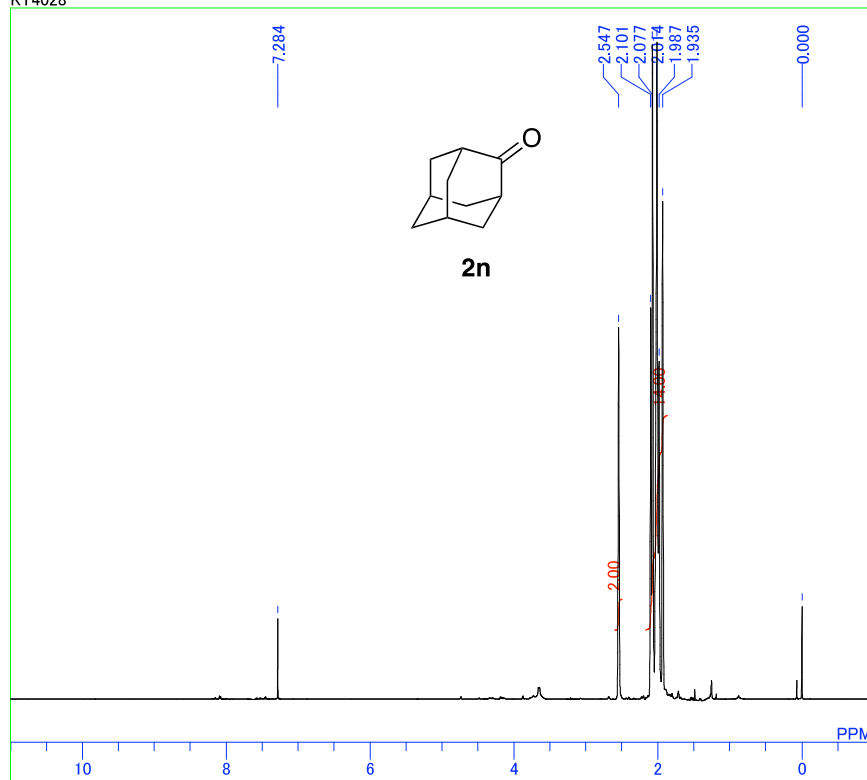


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MENUF bcm_th5atfTH5ATFG2_2
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PW1 5.75 usec
DEADT 10.00 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 32768
SAMPO 32768
TIMES 6000
DUMMY 1
FREQU 33898.30 Hz
FILTR 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 1.0000 sec
ADBIT 16
RGAIN 27
BF 0.25 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\Documents and Settings\ALPHA\
SHMFL TH5ATFG2_2
LKFIN 70334.0 Hz
LKLEV 180
LGAIN 21
LKPHS 210
LKSIG 689
CSPED 17 Hz
FILDC
FILDF
    
```


^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 2-adamantanone (**2n**).

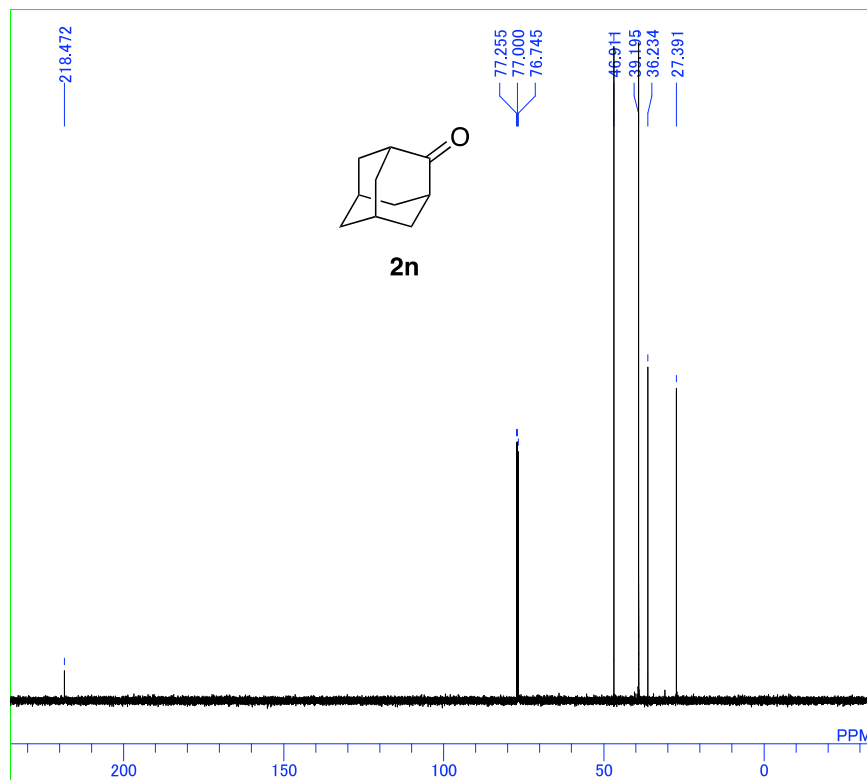
C:\WINALPHA\COMMON\DEFAULT.ALS
KT4028



```

MENUF non_th5atfTH5ATFG2_1KT4028
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OBFRQ 500.00 MHz
OBFIN 162410.00 Hz
PW1 6.40 usec
DEADT 56.80 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 16384
SAMPO 16384
TIMES 32
DUMMY 1
FREQU 10000.00 Hz
FILTR 5000 Hz
DELAY 40.00 usec
ACQTM 1.6384 sec
PD 2.0000 sec
ADBIT 16
RGAIN 13
BF 0.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\WINALPHA\COMMON\DEFAULT
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LKPHS 214
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FILDC
FILDF
    
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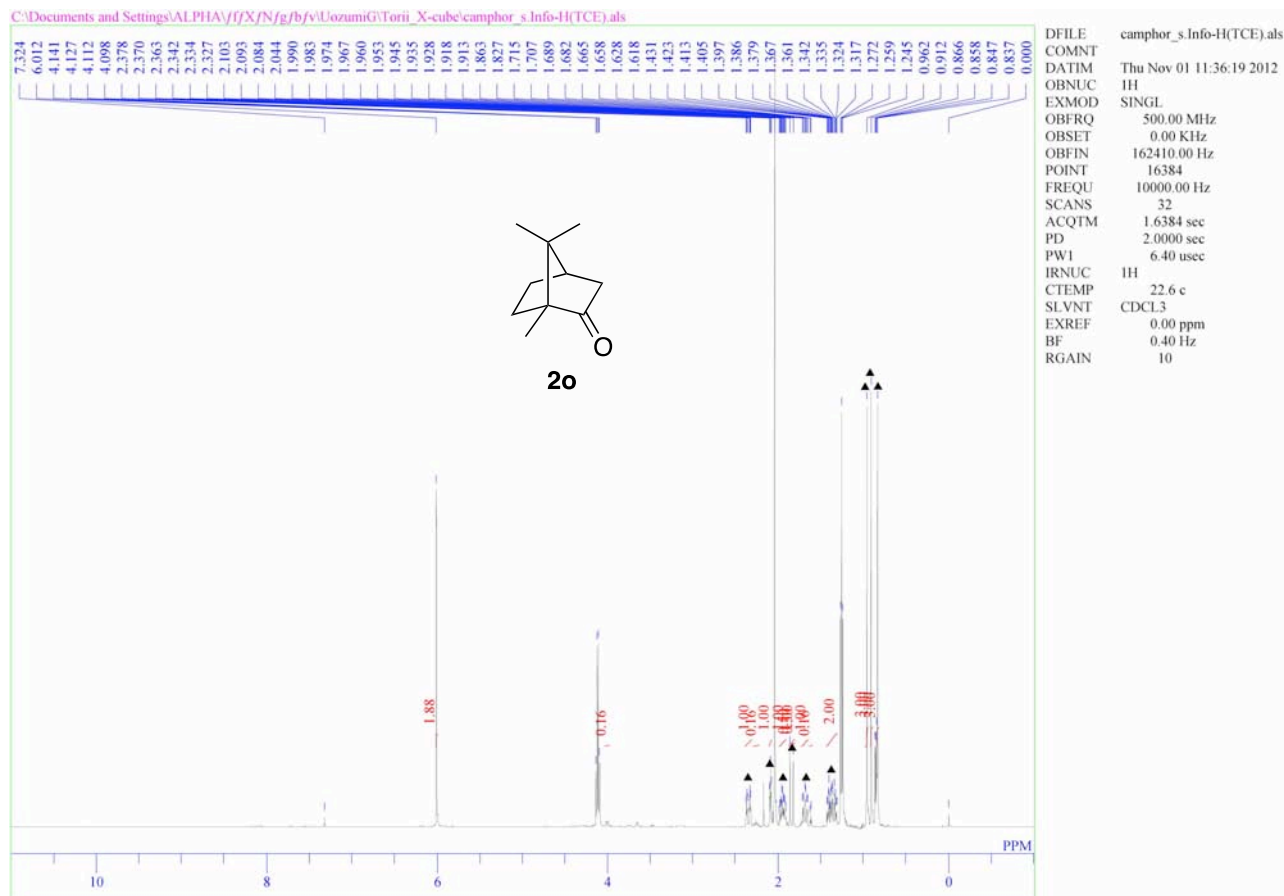
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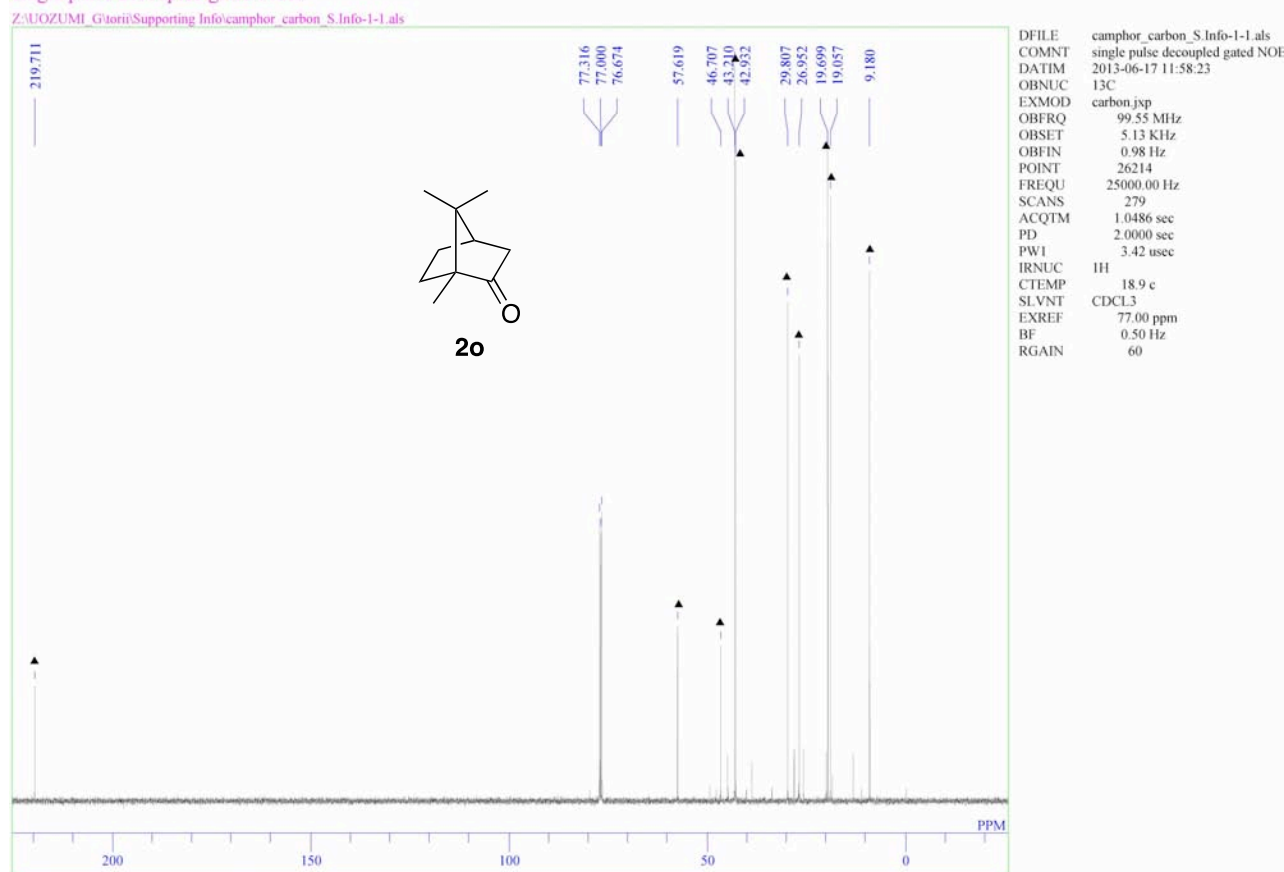
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OBFIN 127958.00 Hz
PW1 5.75 usec
DEADT 10.00 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 32768
SAMPO 32768
TIMES 5000
DUMMY 1
FREQU 33898.30 Hz
FILTR 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 1.0000 sec
ADBIT 16
RGAIN 26
BF 0.30 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\Documents and Settings\ALPHA\
SHMFL TH5ATFG2_1
LKFIN 70334.0 Hz
LKLEV 180
LGAIN 21
LKPHS 214
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FILDC
FILDF
    
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^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of camphor (**2o**).

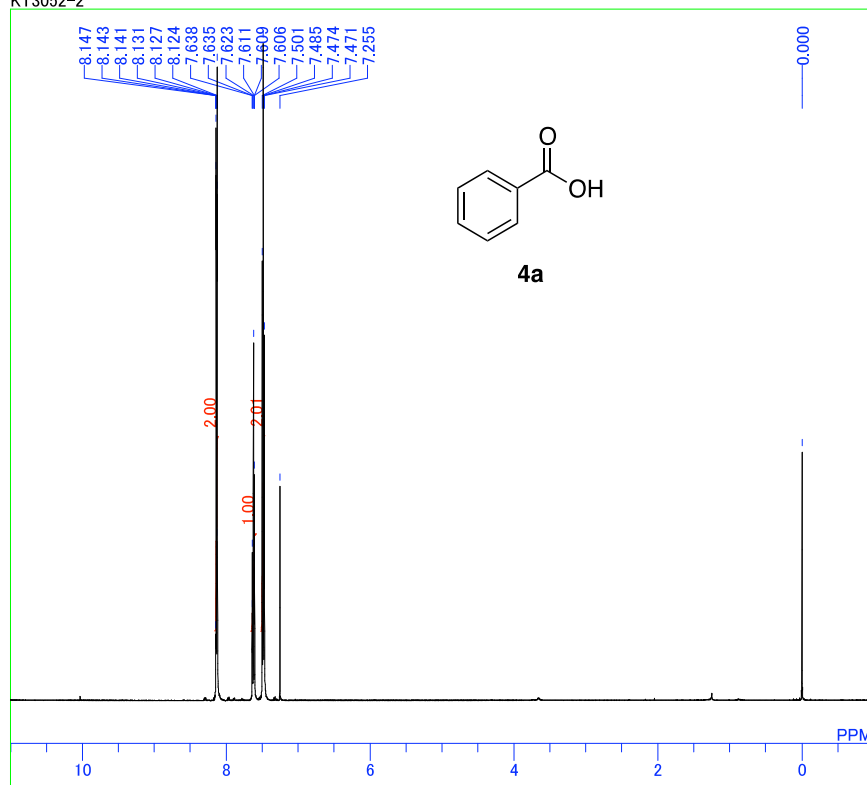


single pulse decoupled gated NOE



^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of benzoic acid (**4a**).

C:\Documents and Settings\ALPHA\1-K\4-2\UozumiG\Torii_X-cube\KT3052-2a.als
KT3052-2

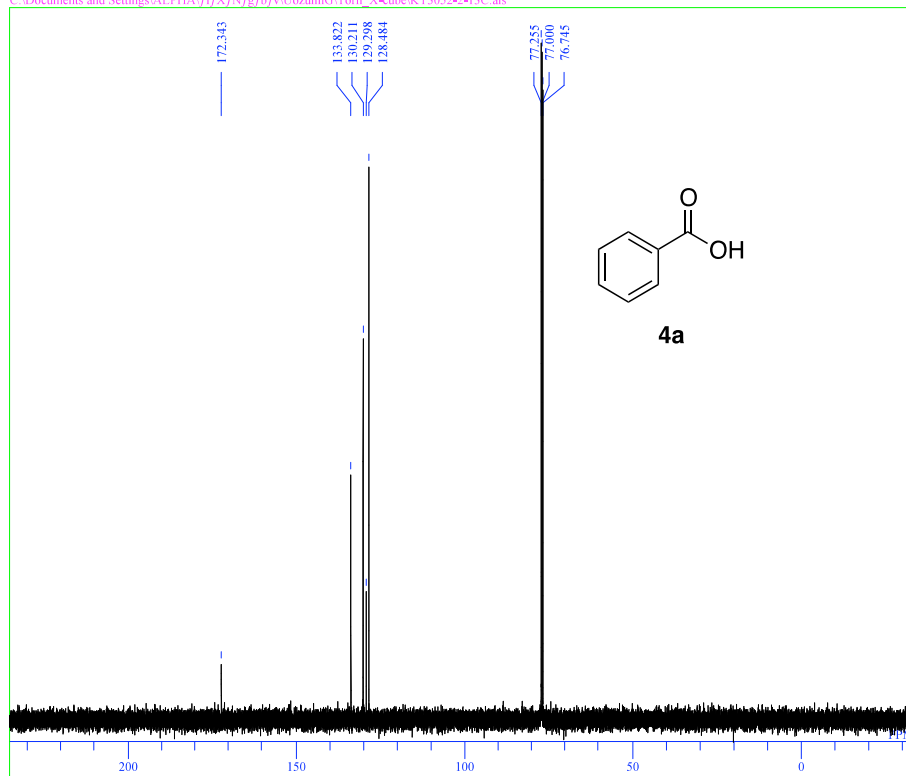


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MENUF non_th5atTH5ATFG2
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PW1 6.40 usec
DEADT 56.80 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 16384
SAMPO 16384
TIMES 16
DUMMY 1
FREQU 10000.00 Hz
FILTR 5000 Hz
DELAY 40.00 usec
ACQTM 1.6384 sec
PD 2.0000 sec
ADBIT 16
RGAIN 17
BF 0.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\Documents and Settings\ALPHA\
SHMFL TH5ATFG2
LKFIN 70334.0 Hz
LKLEV 180
LGAIN 20
LKPHS 214
LKSIG 609
CSPED 16 Hz
FILDC
FILDF
    
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KT3052-2

C:\Documents and Settings\ALPHA\1-K\4-2\UozumiG\Torii_X-cube\KT3052-2-13C.als



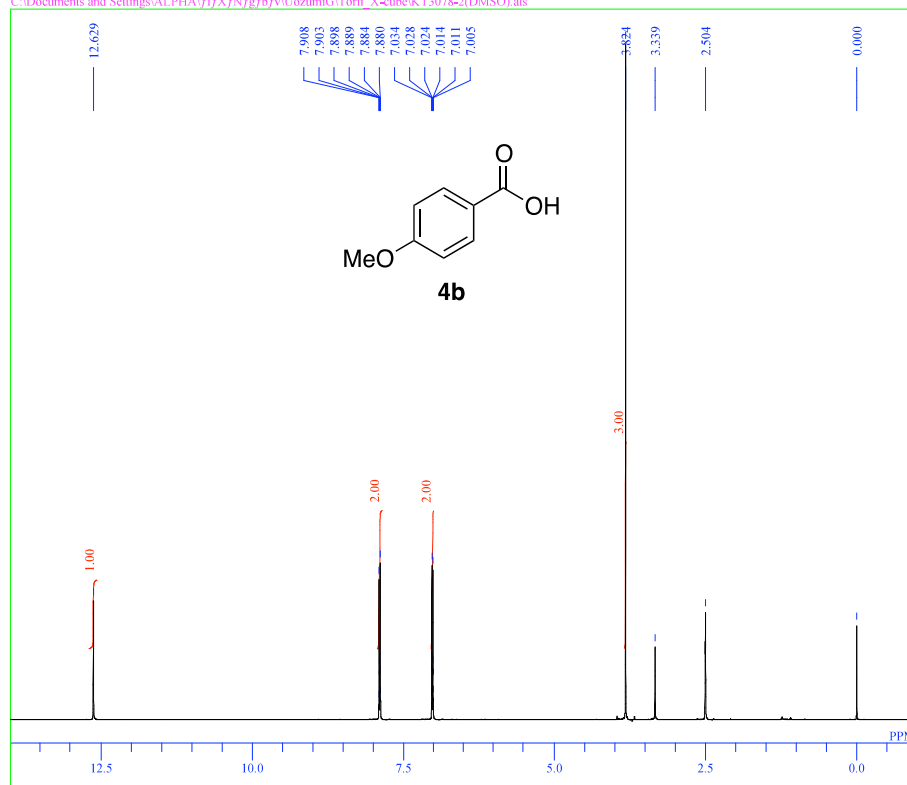
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OBFIN 127958.00 Hz
POINT 32768
FREQU 33898.30 Hz
SCANS 365
ACQTM 0.9667 sec
PD 1.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.25 Hz
RGAIN 27
    
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^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of *p*-anisic acid (**4b**).

KT3073.1

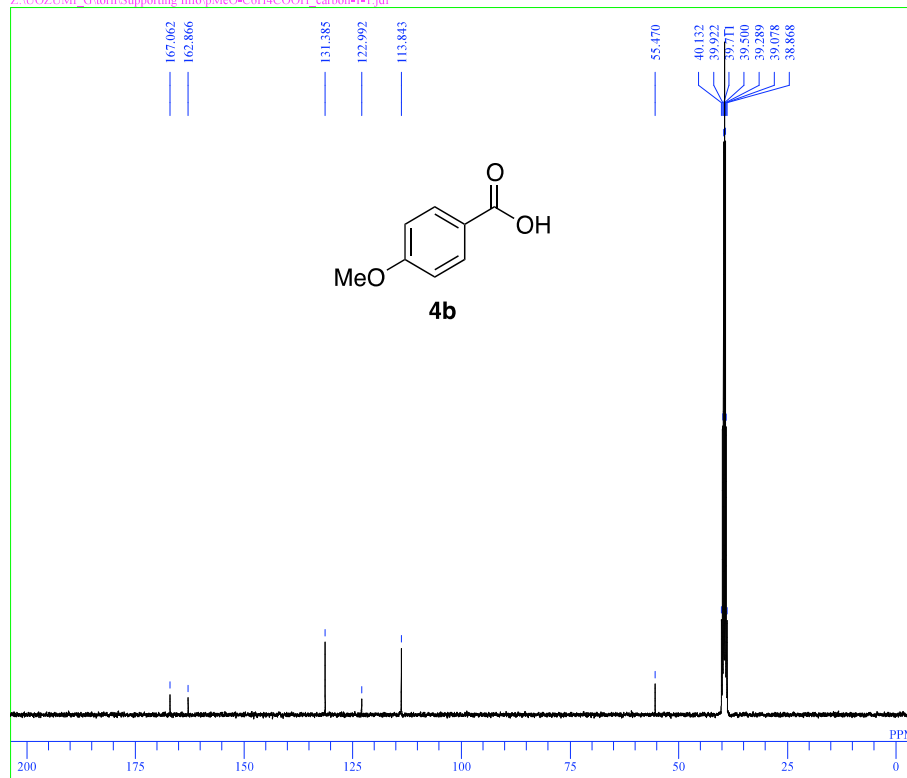
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DFILE KT3078-2(DMSO).als
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 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
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 IRNUC ^1H
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 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 21

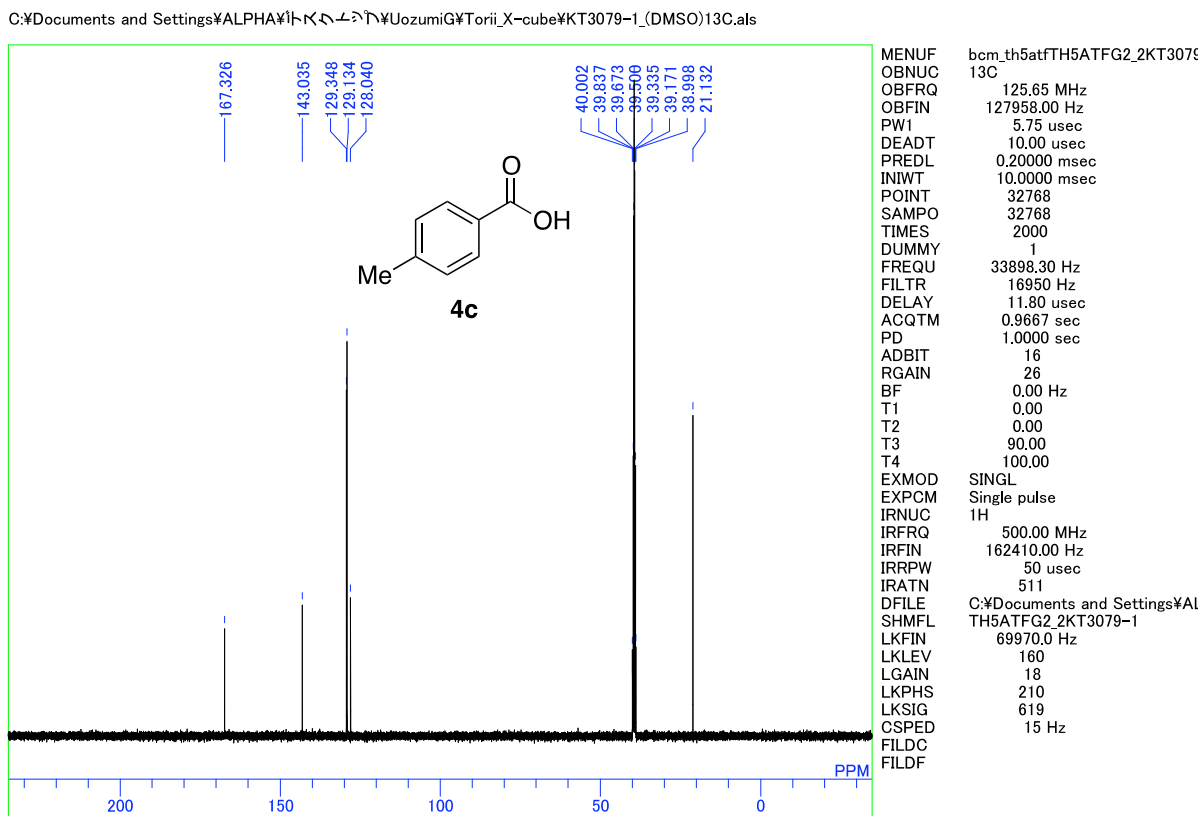
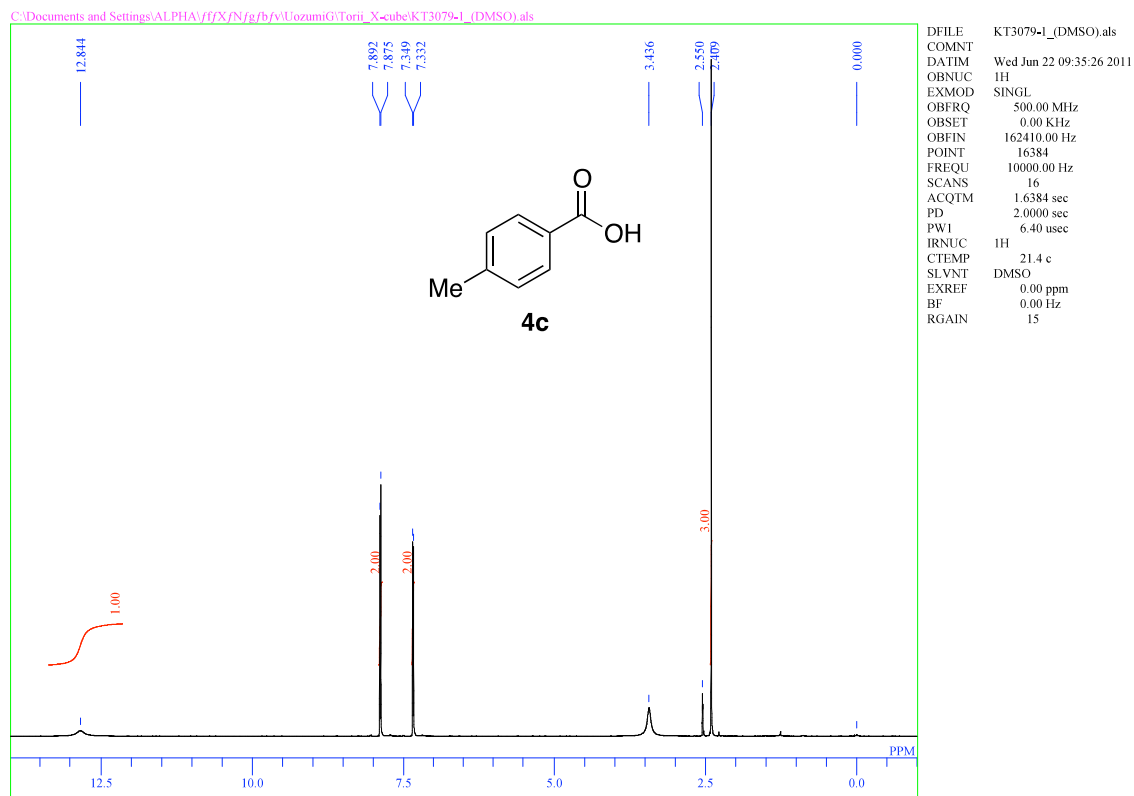
single pulse decoupled gated NOE

Z:\UOZUMI_G\Torii\Supporting_Info\pMeO-C6H4COOH_carbon-1-1.jdf



DFILE pMeO-C6H4COOH_carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2013-07-17 15:20:12
 OBNUC ^{13}C
 EXMOD carbon.jxp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 456
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 IRNUC ^1H
 CTEMP 20.0 c
 SLVNT DMSO
 EXREF 39.50 ppm
 BF 2.80 Hz
 RGAIN 60

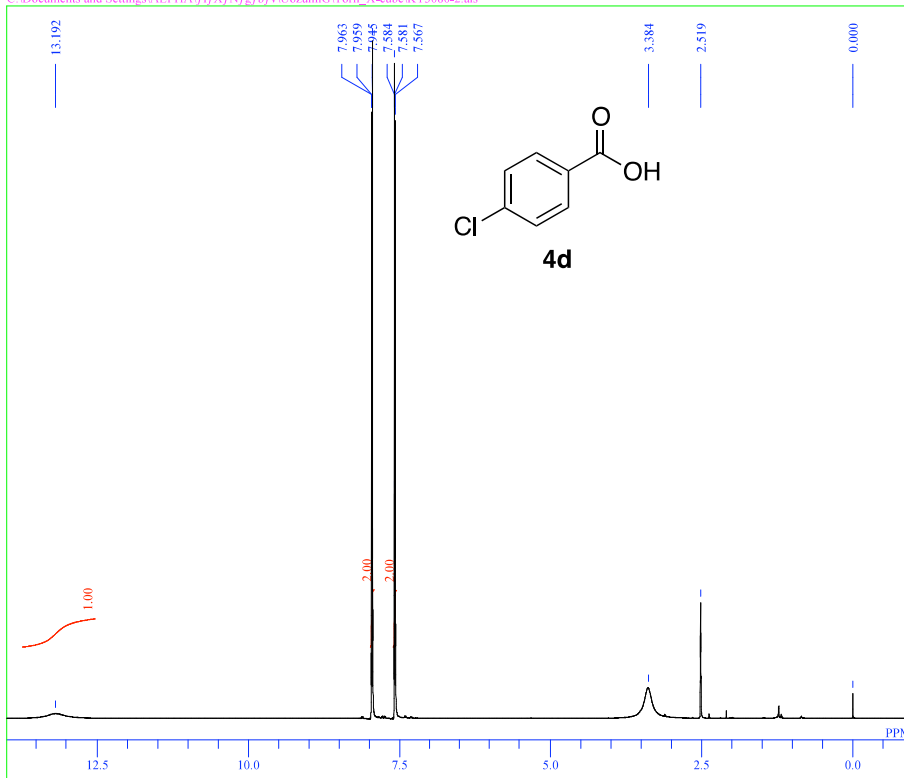
^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of *p*-toluic acid (**4c**).



^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 4-chlorobenzoic acid (**4d**).

KT3080-2

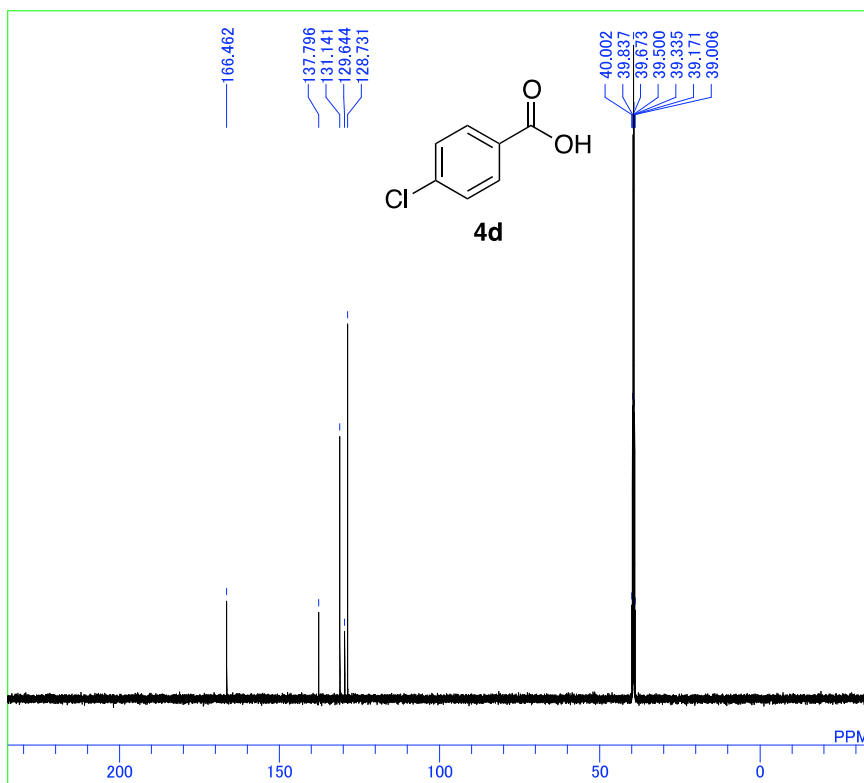
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DFILE      KT3080-2.als
COMINT     KT3080-2
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OBNUC      1H
EXMOD      SINGL
OBFREQ     500.00 MHz
OBSET      0.00 KHz
OBFIN      162410.00 Hz
POINT      16384
FREQU      10000.00 Hz
SCANS      16
ACQTM      1.6384 sec
PD          2.0000 sec
PW1        6.40 usec
IRNUC      1H
CTEMP      22.7 c
SLVNT      DMSO
EXREF      0.00 ppm
BF          0.00 Hz
RGAIN      18
    
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C:\WINALPHA\COMMON\DEFAULT.ALS



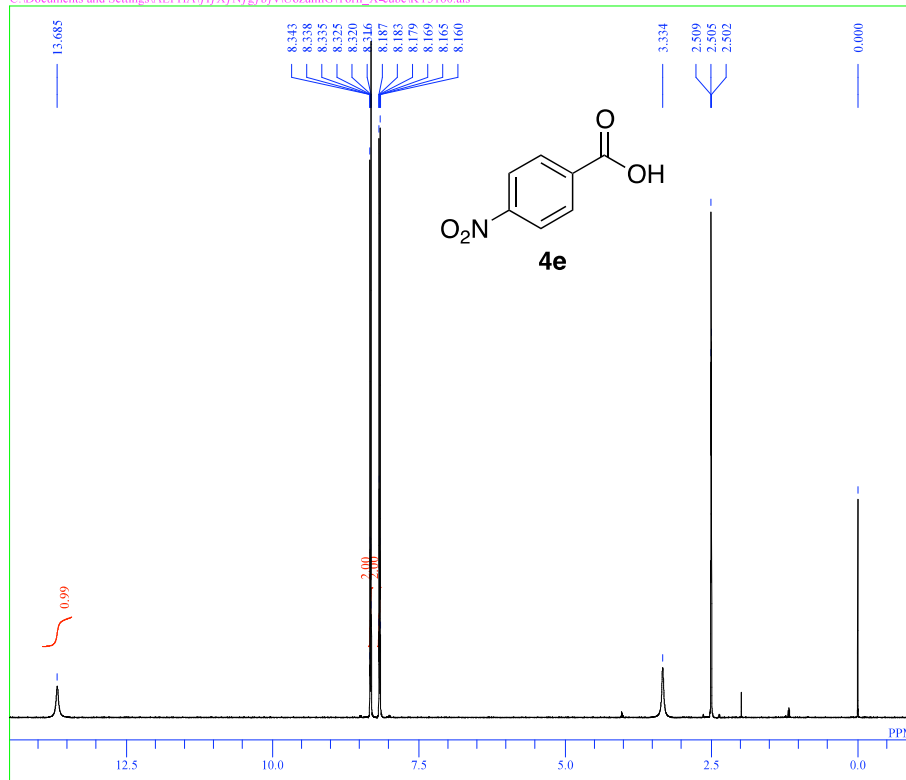
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DEADT      10.00 usec
PREDL      0.20000 msec
INIWT      10.0000 msec
POINT      32768
SAMPO      32768
TIMES      2000
DUMMY      1
FREQU      33898.30 Hz
FILTR      16950 Hz
DELAY      11.80 usec
ACQTM      0.9667 sec
PD          1.0000 sec
ADBIT      16
RGAIN      25
BF          0.25 Hz
T1          0.00
T2          0.00
T3          90.00
T4          100.00
EXMOD      SINGL
EXPCM      Single pulse
IRNUC      1H
IRFRQ      500.00 MHz
IRFIN      162410.00 Hz
IRRPW      50 usec
IRATN      511
DFILE      C:\WINALPHA\COMMON\DEFAULT
SHMFL      TH5ATFG2_2KT3080-2
LKFIN      69970.0 Hz
LKLEV      160
LGAIN      18
LKPHS      210
LKSIG      648
CSPED      17 Hz
FILDC
FILDF
    
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^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of *p*-nitrobenzoic acid (**4e**).

KT3160

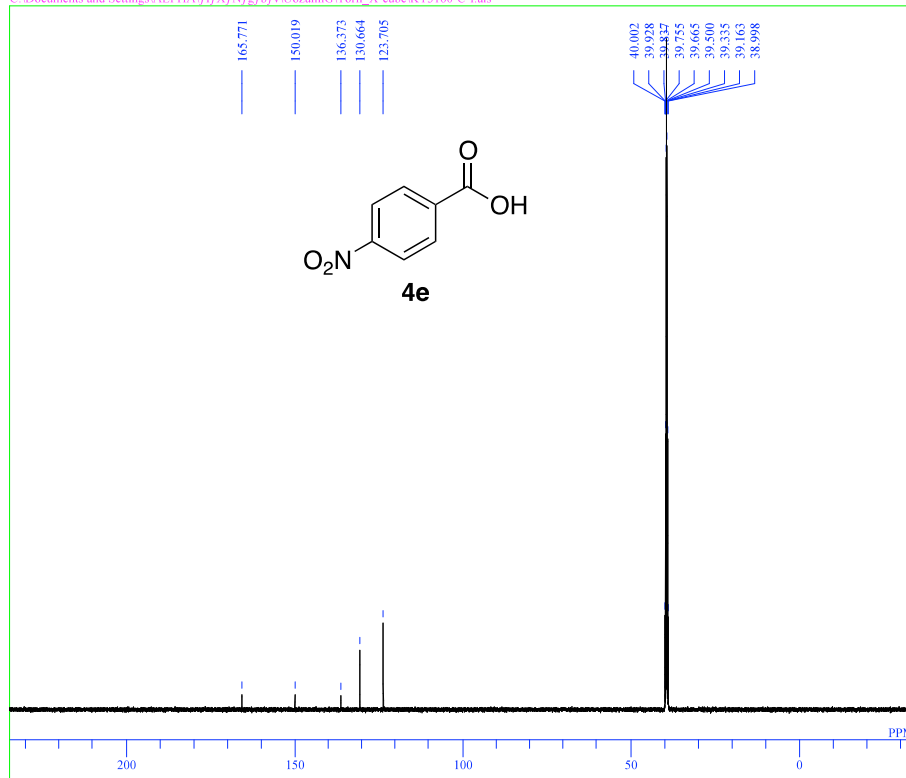
C:\Documents and Settings\ALPHA\ff\X\N\g\b\y\UozumiG\Torii_X-cube\KT3160.als



DFILE KT3160.als
 COMNT KT3160
 DATIM Fri Feb 03 13:40:56 2012
 OBNUC ^1H
 EXMOD SINGL
 OBFREQ 500.00 MHz
 OBSETE 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 32
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PW1 6.40 usec
 IRNUC ^1H
 CTEMP 22.9 c
 SLVNT DMSO
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 23

KT3160_13C

C:\Documents and Settings\ALPHA\ff\X\N\g\b\y\UozumiG\Torii_X-cube\KT3160-C-f.als

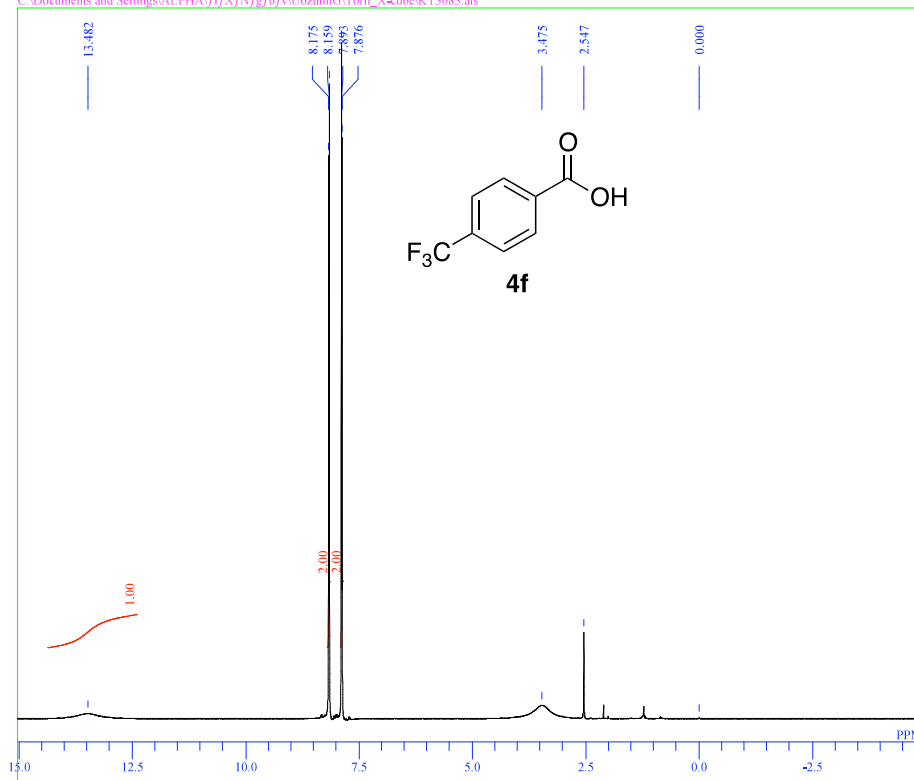


DFILE KT3160-C-f.als
 COMNT KT3160_13C
 DATIM Fri Feb 03 15:11:29 2012
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 EXMOD SINGL
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 OBSETE 0.00 KHz
 OBFIN 127958.00 Hz
 POINT 32768
 FREQU 33898.30 Hz
 SCANS 2197
 ACQTM 0.9667 sec
 PD 1.0000 sec
 PW1 5.75 usec
 IRNUC ^{13}C
 CTEMP 24.7 c
 SLVNT DMSO
 EXREF 39.50 ppm
 BF 0.70 Hz
 RGAIN 25

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 4-(trifluoromethyl)benzoic acid (**4f**).

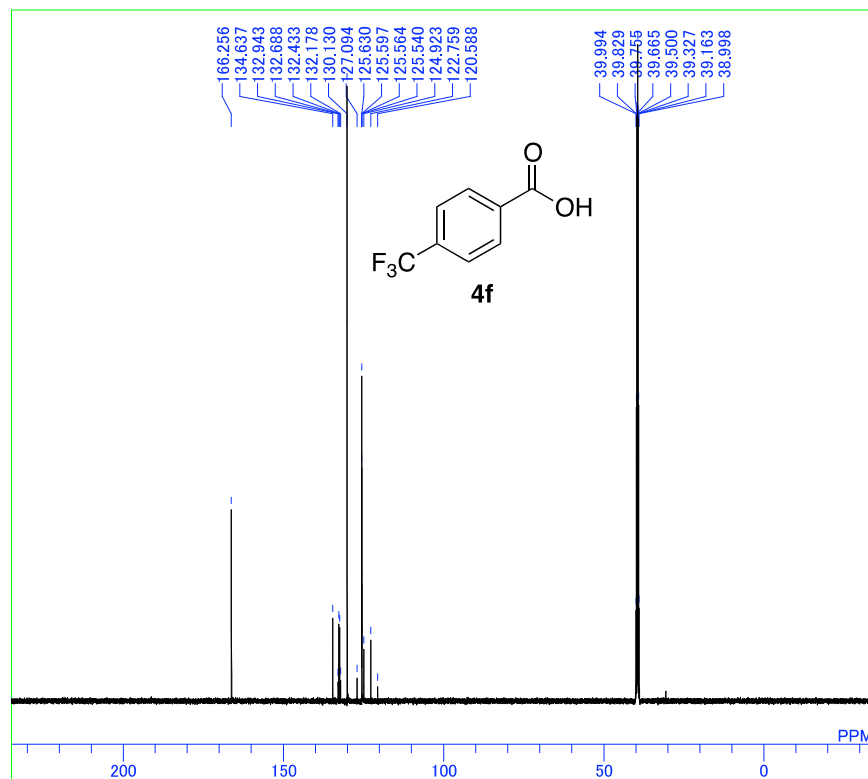
KT3083

C:\Documents and Settings\ALPHA\ff\X\N\g\fb\UozumiG\Torii_X-cube\KT3083.als



DFILE KT3083.als
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 DATIM Fri Jun 24 14:37:09 2011
 OBNUC 1H
 EXMOD SINGL
 OBFREQ 500.00 MHz
 OBSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PW1 6.40 usec
 IRNUC 1H
 CTEMP 23.7 c
 SLVNT DMSO
 EXREF 0.00 ppm
 BF 0.25 Hz
 RGAIN 16

C:\Documents and Settings\ALPHA\ff\X\N\g\fb\UozumiG\Torii_X-cube\KT3083.1fid.als

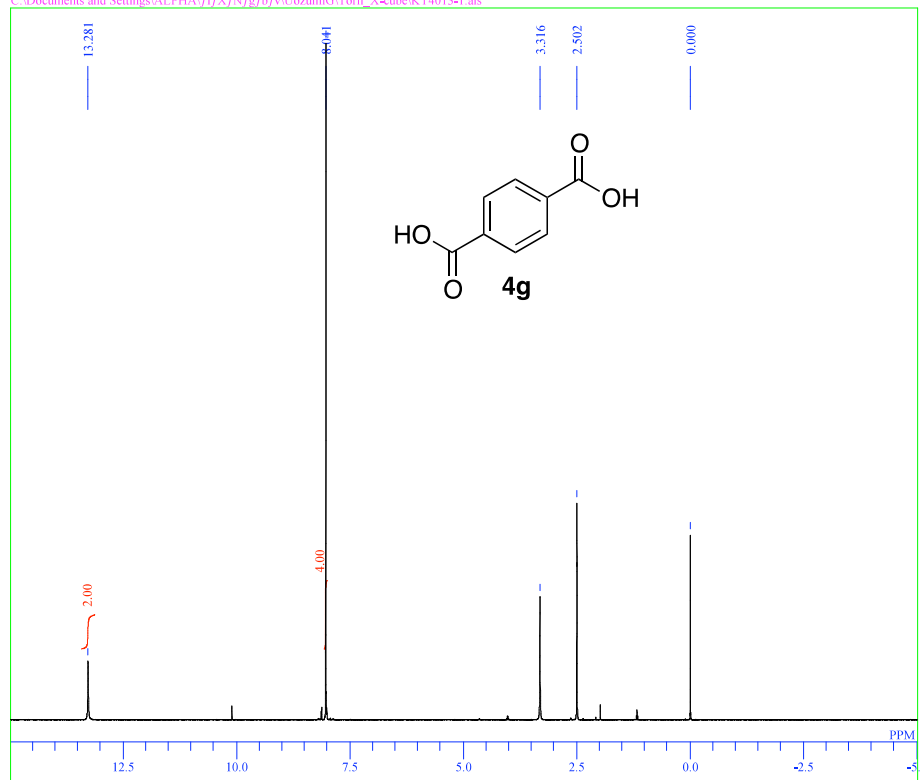


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 OBFREQ 127958.00 Hz
 OBFIN 127958.00 Hz
 PW1 5.75 usec
 DEADT 10.00 usec
 PREDL 0.20000 msec
 INIWT 10.0000 msec
 POINT 32768
 SAMPO 32768
 TIMES 4000
 DUMMY 1
 FREQU 33898.30 Hz
 FILTR 16950 Hz
 DELAY 11.80 usec
 ACQTM 0.9667 sec
 PD 1.0000 sec
 ADBIT 16
 RGAIN 25
 BF 0.25 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD SINGL
 EXPCM Single pulse
 IRNUC 1H
 IRFRQ 500.00 MHz
 IRFIN 162410.00 Hz
 IRRPW 50 usec
 IRATN 511
 DFILE C:\Documents and Settings\ALPHA\ff\X\N\g\fb\UozumiG\Torii_X-cube\KT3083.1
 SHMFL TH5ATFG2_2KT3083.1
 LKFIN 69970.0 Hz
 LKLEV 160
 LGAIN 21
 LKPHS 214
 LKSIG 1373
 CSPED 16 Hz
 FILDC
 FILDF

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of terephthalic acid (**4g**).

KT4013isolate

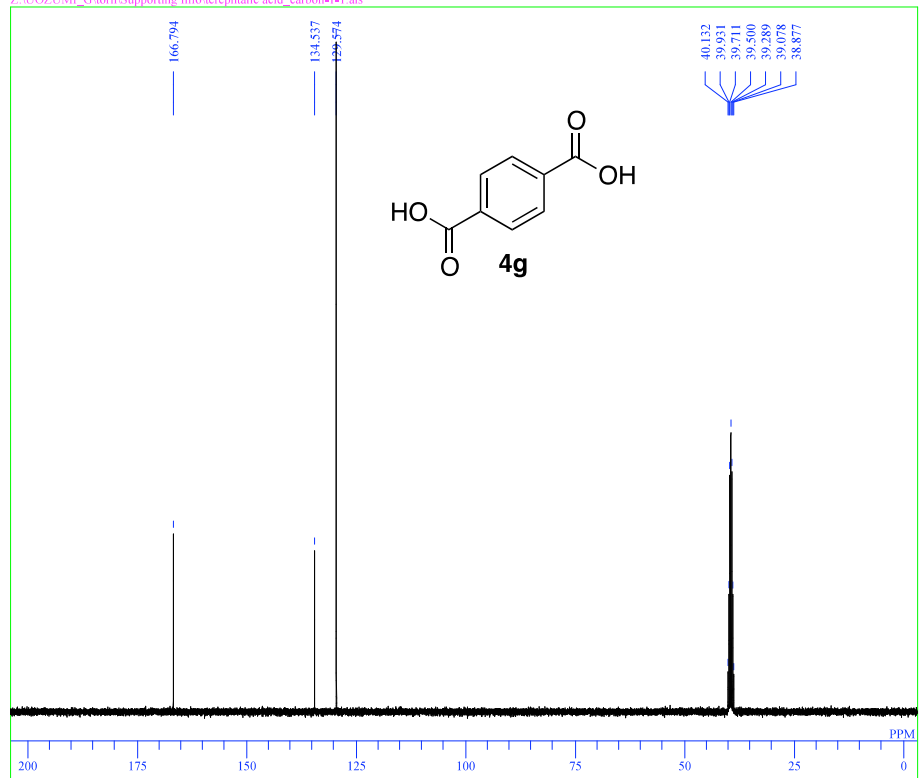
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DFILE KT4013-1.als
 COMNT KT4013isolate
 DATIM Tue Jul 31 15:52:28 2012
 OBNUC ^1H
 EXMOD SINGL
 OBFREQ 500.00 MHz
 OBSSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 32
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PW1 6.40 usec
 IRNUC ^1H
 CTEMP 26.1 c
 SLVNT DMSO
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 23

single pulse decoupled gated NOE

Z:\UOZUMI_G\torii\Supporting_Info\terephthalic_acid_carbon-1-1.als

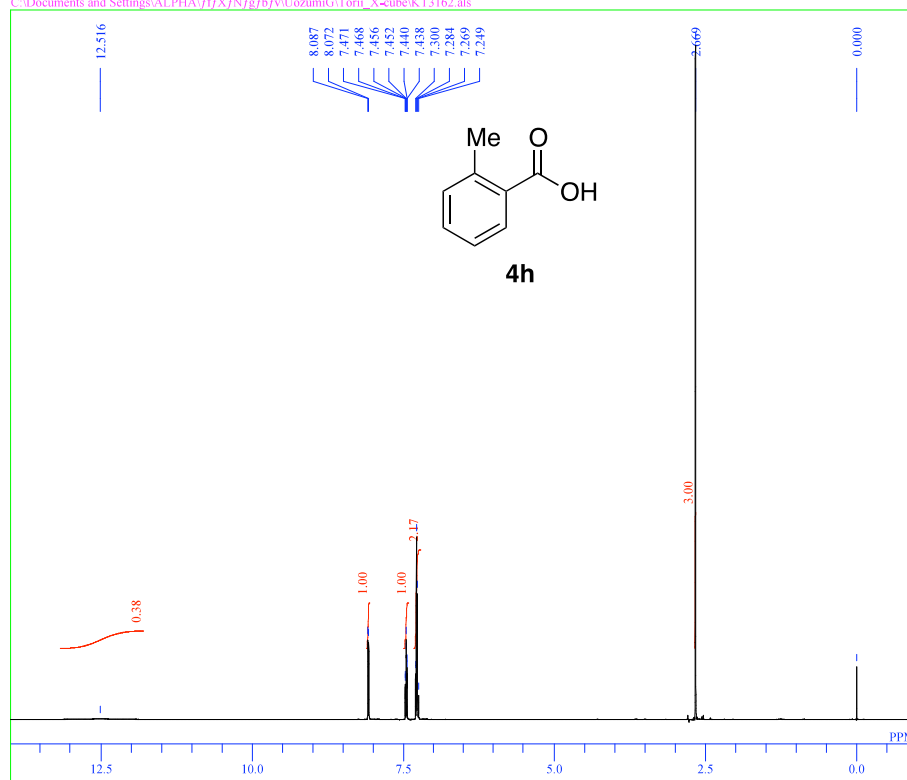


DFILE terephthalic_acid_carbon-1-1.als
 COMNT single pulse decoupled gated NOE
 DATIM 2013-06-14 14:23:60
 OBNUC ^{13}C
 EXMOD carbon.jsp
 OBFREQ 99.55 MHz
 OBSSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 185
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 IRNUC ^1H
 CTEMP 20.3 c
 SLVNT DMSO
 EXREF 39.50 ppm
 BF 0.00 Hz
 RGAIN 60

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of *o*-toluic acid (**4h**).

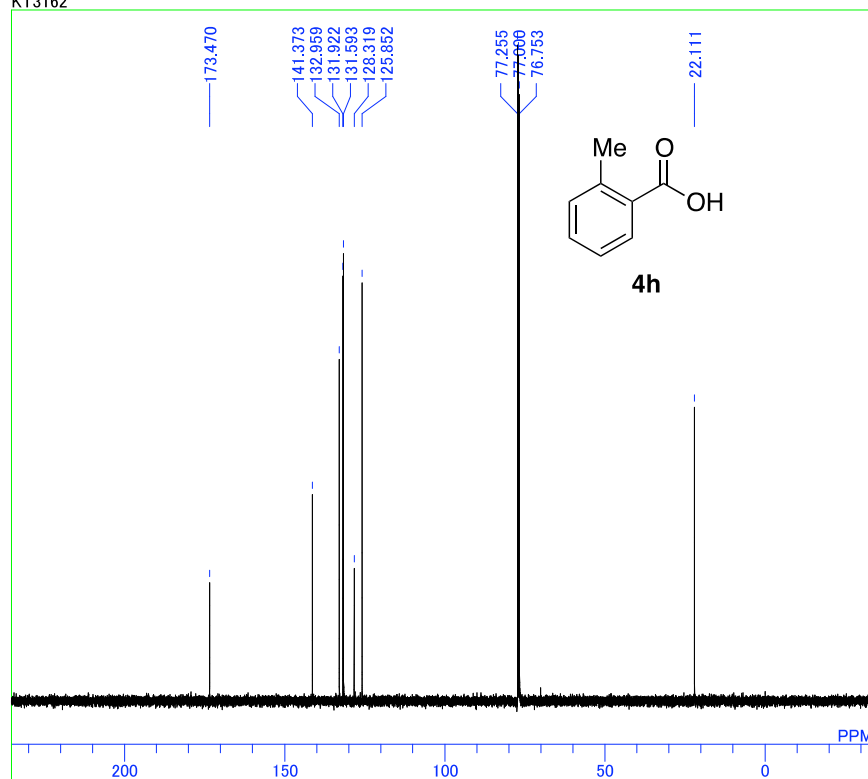
KT3162

C:\Documents and Settings\ALPHA\ff\X\N\g\fb\UozumiG\Torii_X-cube\KT3162.als



DFILE KT3162.als
 COMNT KT3162
 DATIM Tue Jan 31 10:29:27 2012
 OBNUC 1H
 EXMOD SINGL
 OBFREQ 500.00 MHz
 OBSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PW1 6.40 usec
 IRNUC 1H
 CTEMP 23.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 16

C:\Documents and Settings\ALPHA\ff\X\N\g\fb\UozumiG\Torii_X-cube\KT3162_13C-f.als
KT3162

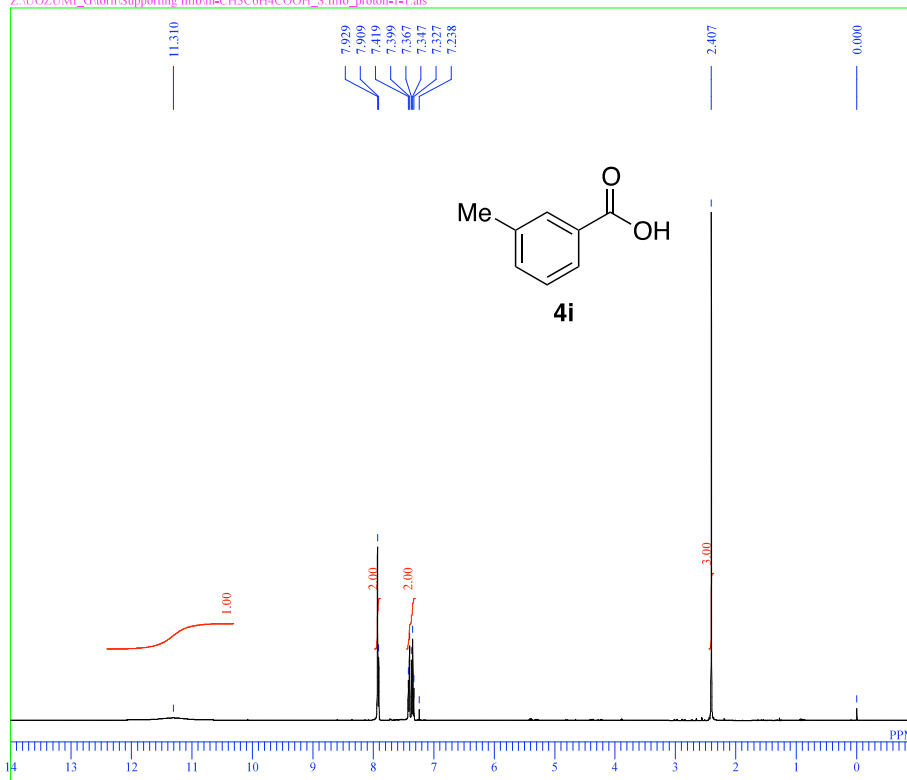


MENUF bcm_th5atfTH5ATFG2_1KT3162
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 PW1 5.75 usec
 DEADT 10.00 usec
 PREDL 0.20000 msec
 INIWT 10.0000 msec
 POINT 32768
 SAMPO 32768
 TIMES 5000
 DUMMY 1
 FREQU 33898.30 Hz
 FILTR 16950 Hz
 DELAY 11.80 usec
 ACQTM 0.9667 sec
 PD 1.0000 sec
 ADBIT 16
 RGAIN 25
 BF 0.80 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD SINGL
 EXPCM Single pulse
 IRNUC 1H
 IRFRQ 500.00 MHz
 IRFIN 162410.00 Hz
 IRRPW 50 usec
 IRATN 511
 DFILE C:\Documents and Settings\ALPHA\ff\X\N\g\fb\UozumiG\Torii_X-cube\KT3162_13C-f.als
 SHMFL TH5ATFG2_1KT3162
 LKFIN 70334.0 Hz
 LKLEV 180
 LGAIN 21
 LKPHS 208
 LKSIG 850
 CSPED 11 Hz
 FILDC
 FILDF

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of *m*-toluic acid (**4i**).

single_pulse

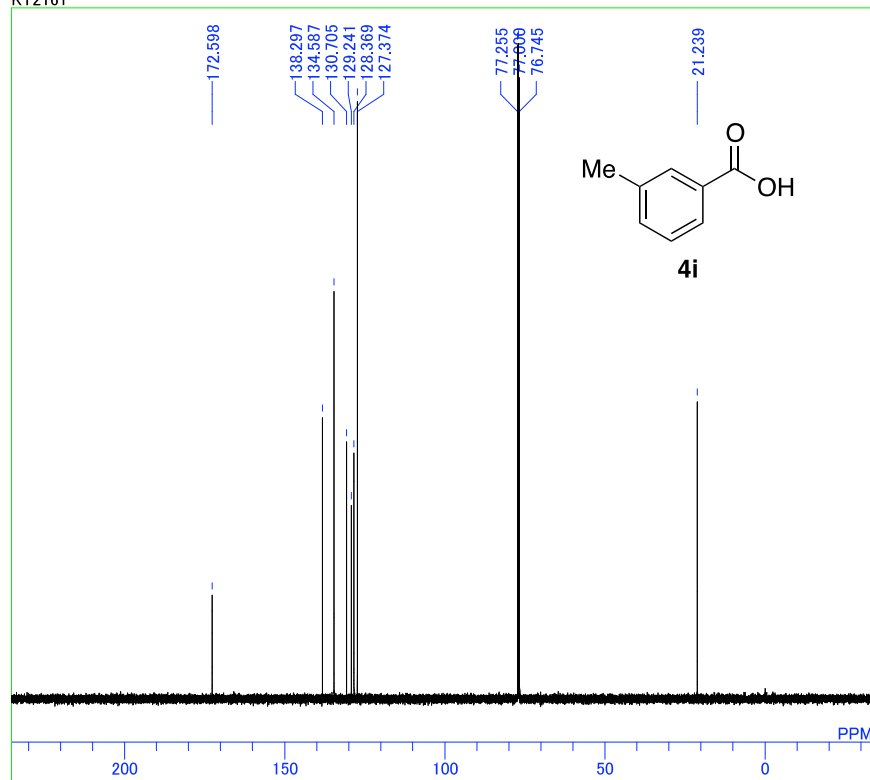
Z:\UOZUMI_G\Torii\Supporting_Inf\m-CH3C6H4COOH_S_Info_proton-1-1.als



```

DFILE m-CH3C6H4COOH_S_Info_proton-1-1.als
COMNT single_pulse
DATIM 2013-07-23 16:36:42
OBNUC 1H
EXMOD proton.jsp
OBFREQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 16384
FREQU 9904.91 Hz
SCANS 18
ACQTM 1.6541 sec
PD 5.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 23.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.25 Hz
RGAIN 22
    
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C:\Documents and Settings\ALPHA\1-K-4-7\UozumiG\Torii_X-cube\KT3161_13C-f.als
KT2161

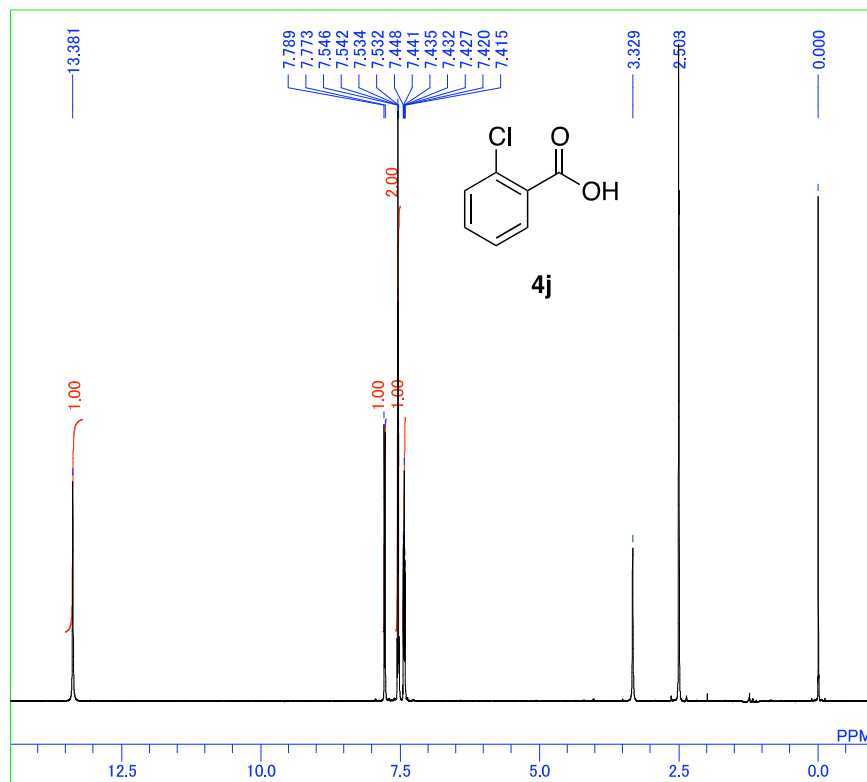


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MENUF bcm_th5atfTH5ATFG2_1KT2161
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OBFIN 127958.00 Hz
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DEADT 10.00 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 32768
SAMPO 32768
TIMES 5000
DUMMY 1
FREQU 33898.30 Hz
FILTR 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 1.0000 sec
ADBIT 16
RGAIN 27
BF 0.30 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\Documents and Settings\ALPHA\
SHMFL TH5ATFG2_1KT2161
LKFIN 70334.0 Hz
LKLEV 180
LGAIN 24
LKPHS 208
LKSIG 1952
CSPED 14 Hz
FILDC
FILDF
    
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^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 2-chlorobenzoic acid (**4j**).

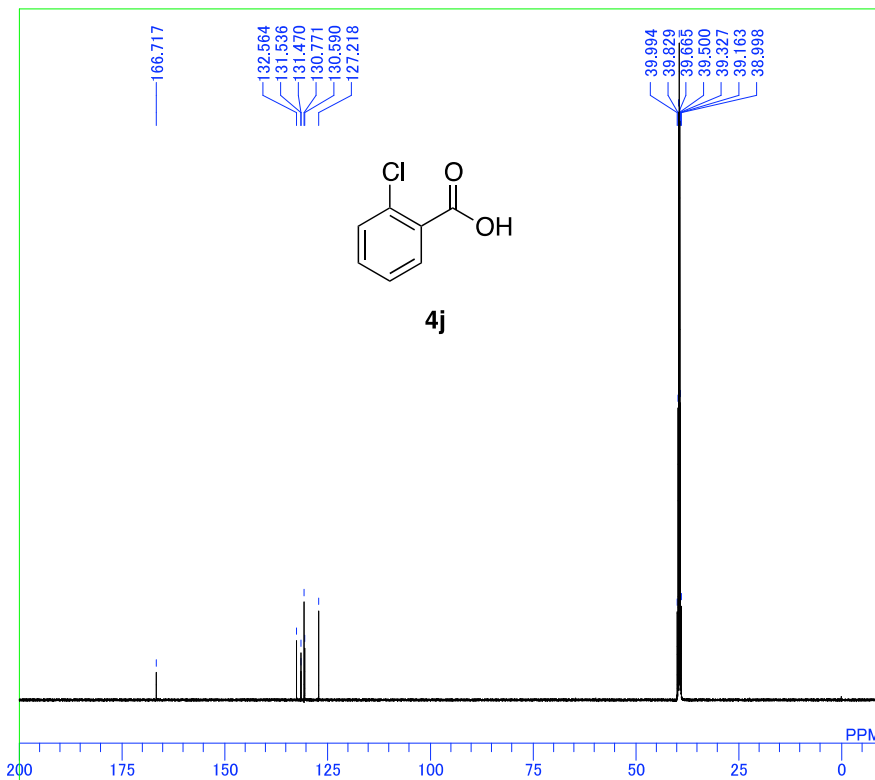
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```

MENUF non_th5atfTH5ATFG2_1
OBNUC 1H
OBFRQ 500.00 MHz
OBFIN 162410.00 Hz
PW1 6.40 usec
DEADT 56.80 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 16384
SAMPO 16384
TIMES 32
DUMMY 1
FREQU 10000.00 Hz
FILTR 5000 Hz
DELAY 40.00 usec
ACQTM 1.6384 sec
PD 2.0000 sec
ADBIT 16
RGAIN 23
BF 0.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\Documents and Settings\ALPHA\
SHMFL TH5ATFG2_1
LKFIN 69970.0 Hz
LKLEV 160
LGAIN 18
LKPHS 214
LKSIG 647
CSPED 16 Hz
FILDC
FILDF
    
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C:\Documents and Settings\ALPHA\1-k-4-2\UozumiG\Torii_X-cube\KT4041-2.1-isolate-13C.als

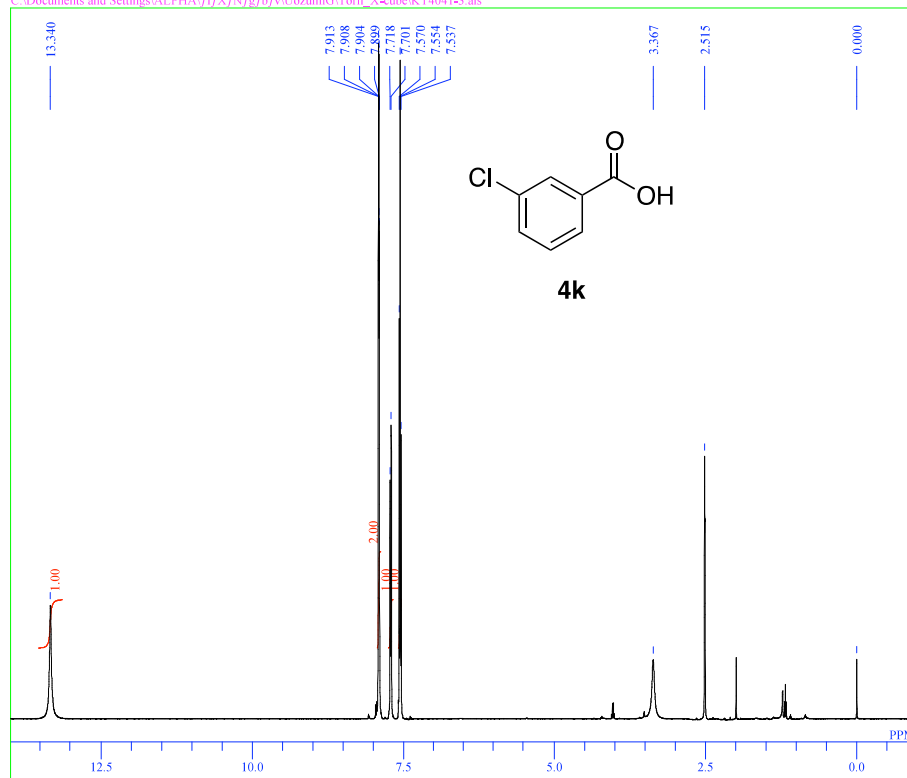


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MENUF bcm_th5atfTH5ATFG2_1
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OBFIN 127958.00 Hz
PW1 5.75 usec
DEADT 10.00 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 32768
SAMPO 32768
TIMES 16000
DUMMY 1
FREQU 33898.30 Hz
FILTR 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 1.0000 sec
ADBIT 16
RGAIN 27
BF 0.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\Documents and Settings\AL
SHMFL TH5ATFG2_1
LKFIN 69970.0 Hz
LKLEV 160
LGAIN 19
LKPHS 214
LKSIG 880
CSPED 16 Hz
FILDC
FILDF
    
```

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 3-chlorobenzoic acid (**4k**).

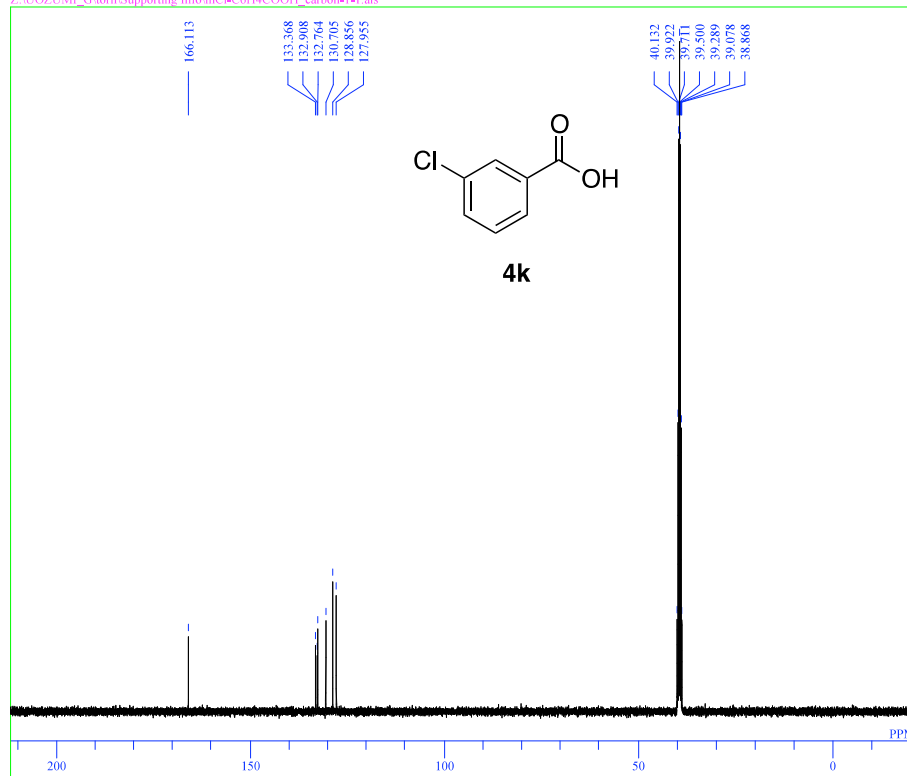
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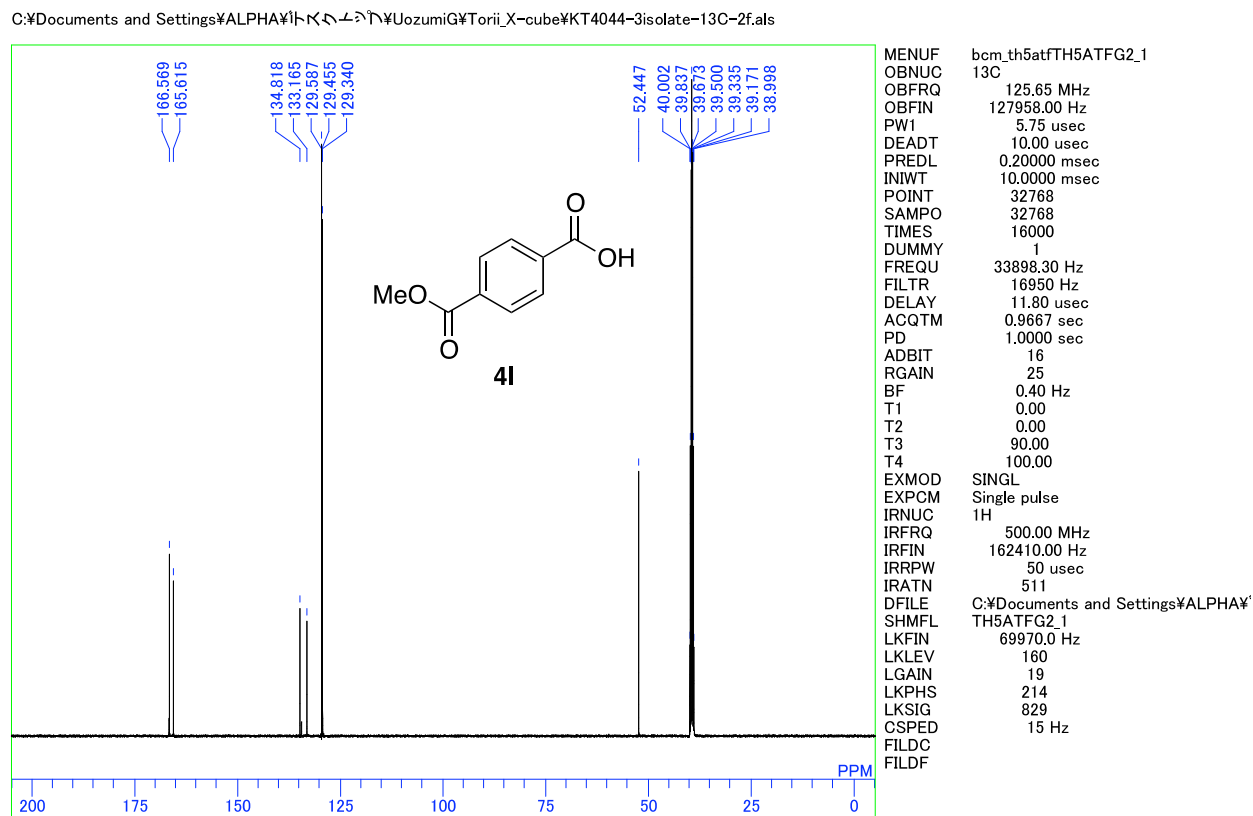
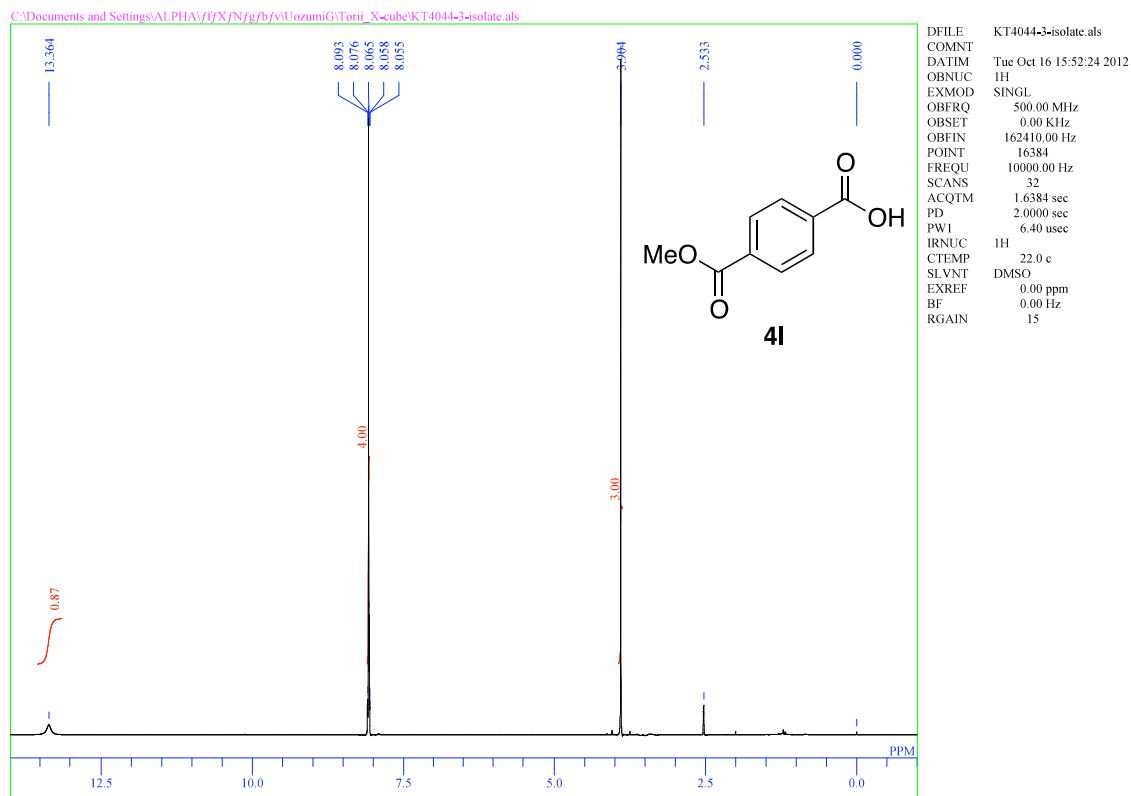
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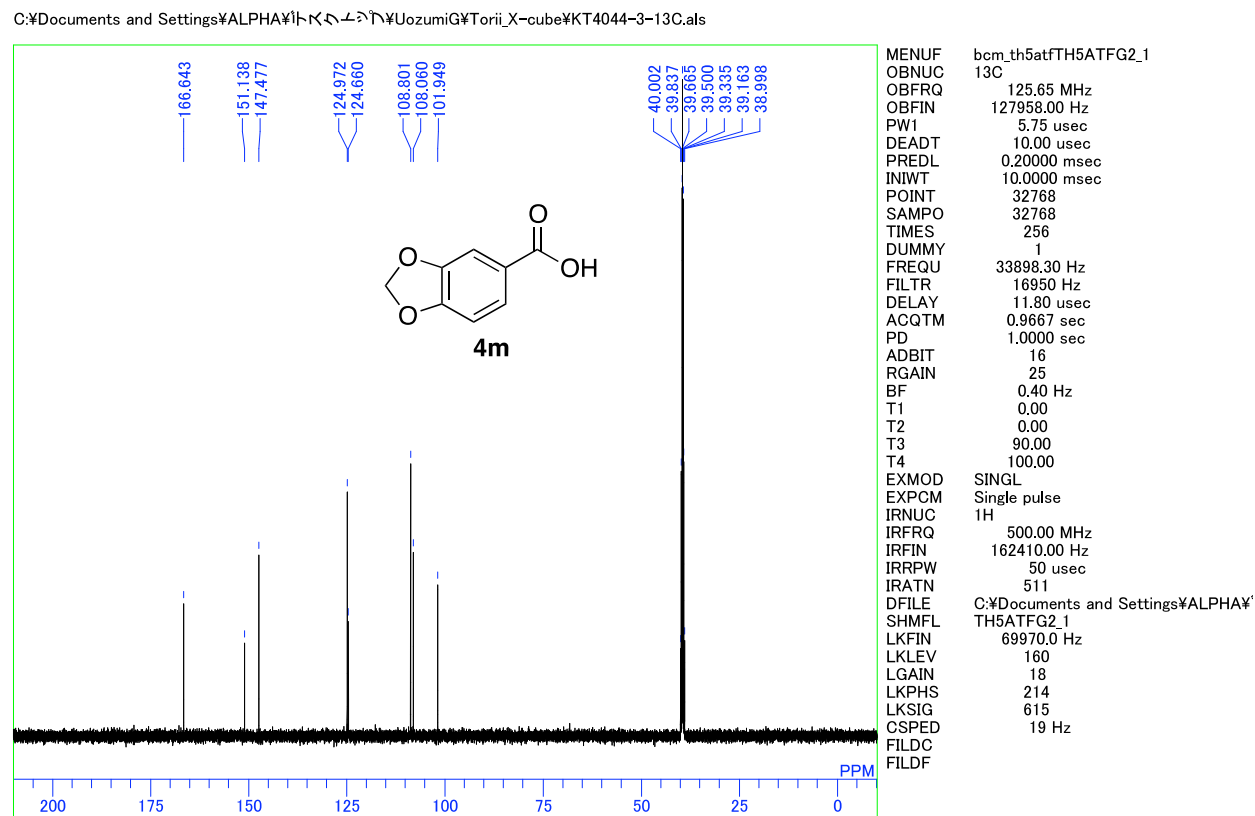
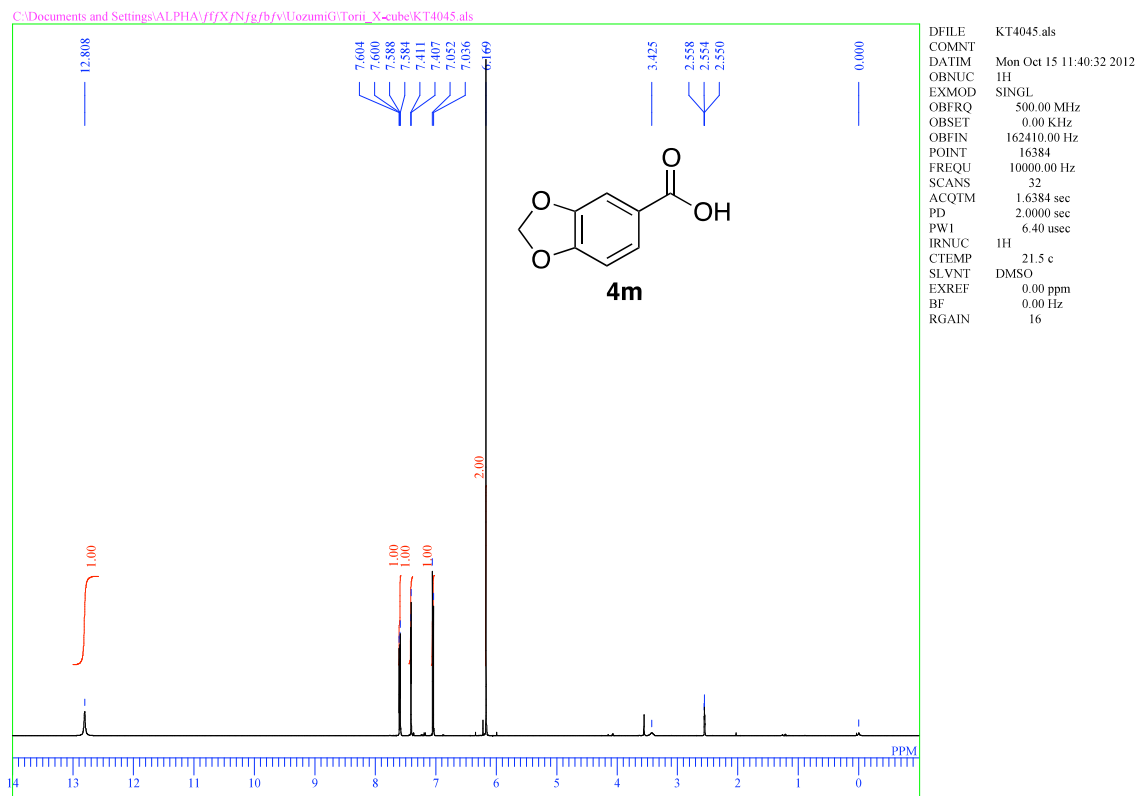


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^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of methyl terephthalate (**41**).



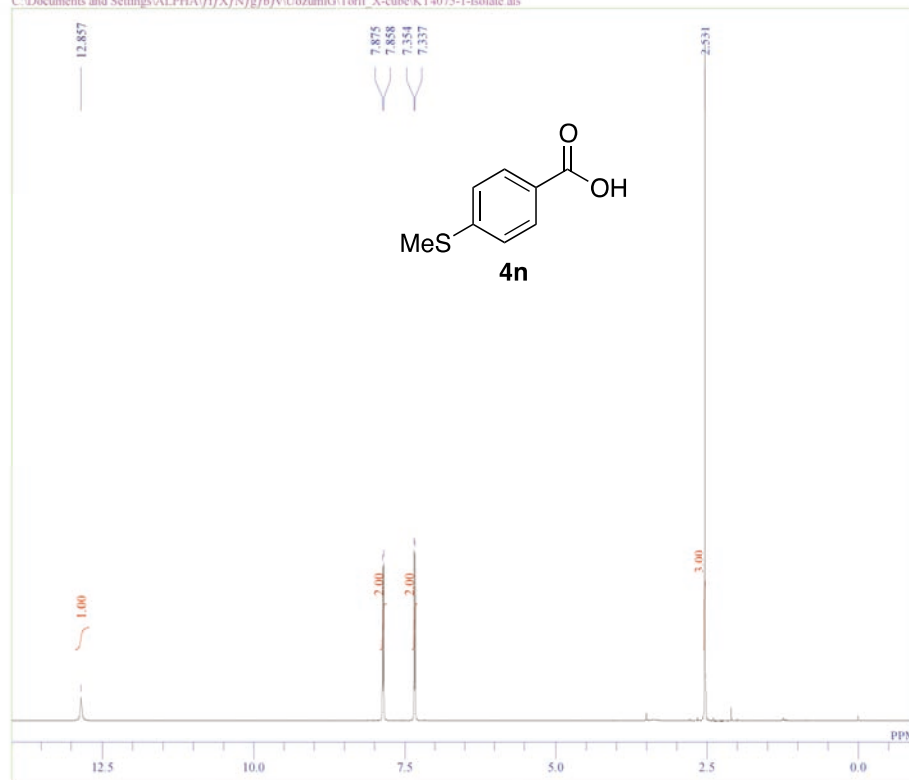
^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of piperonylic acid (**4m**).



^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 4-(methylthio)benzoic acid (**4n**).

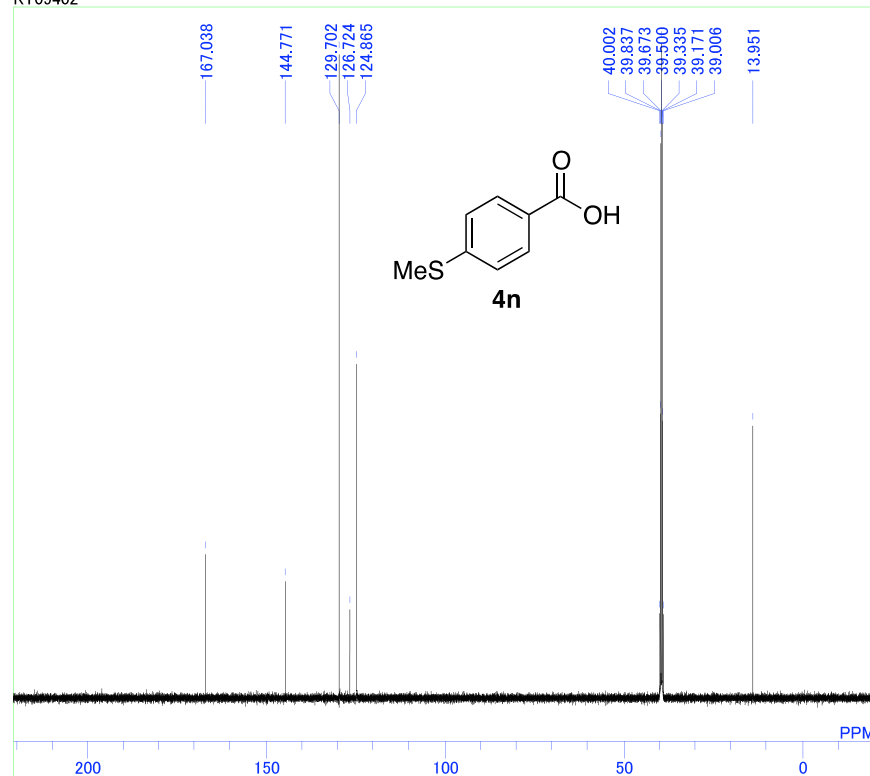
KT4075-1

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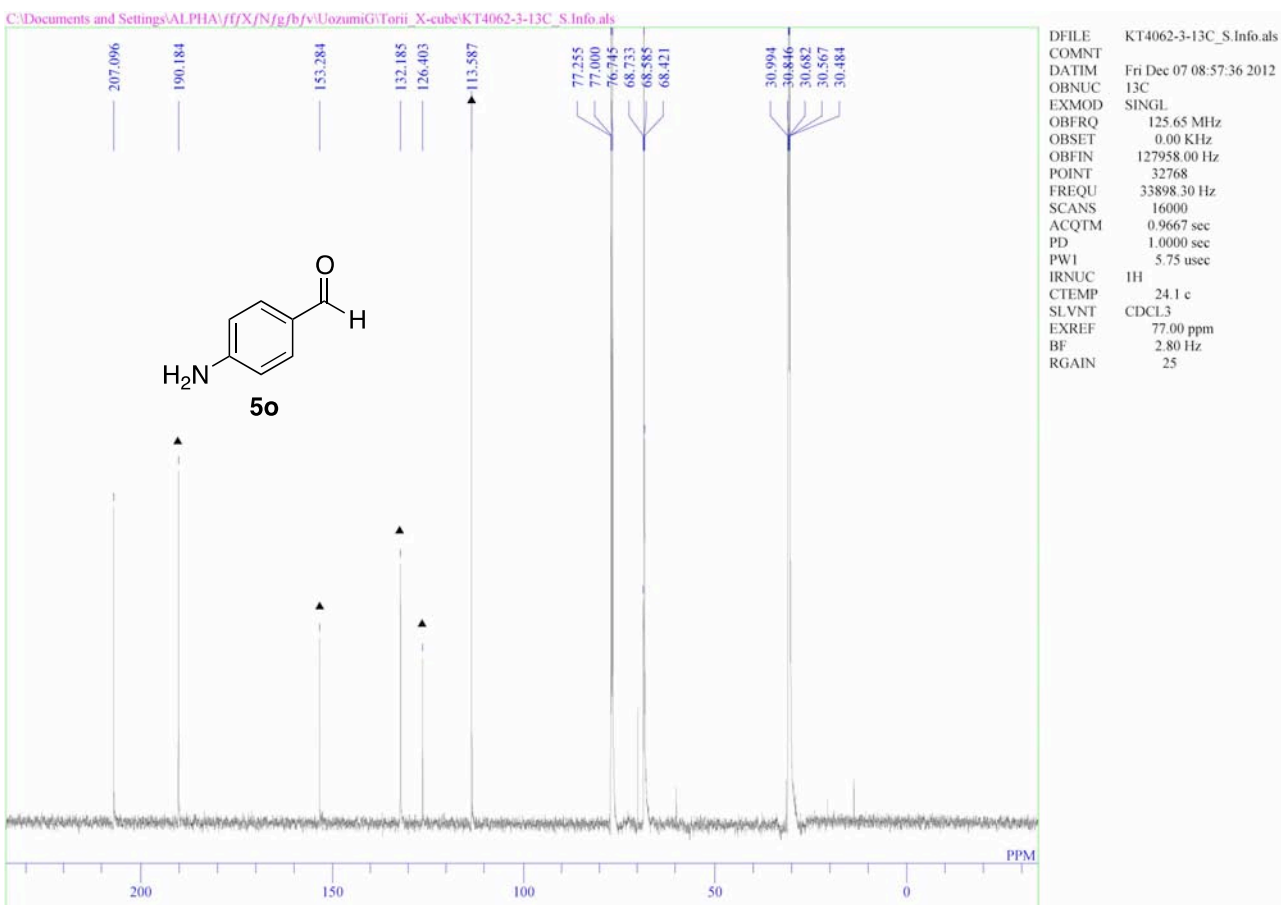
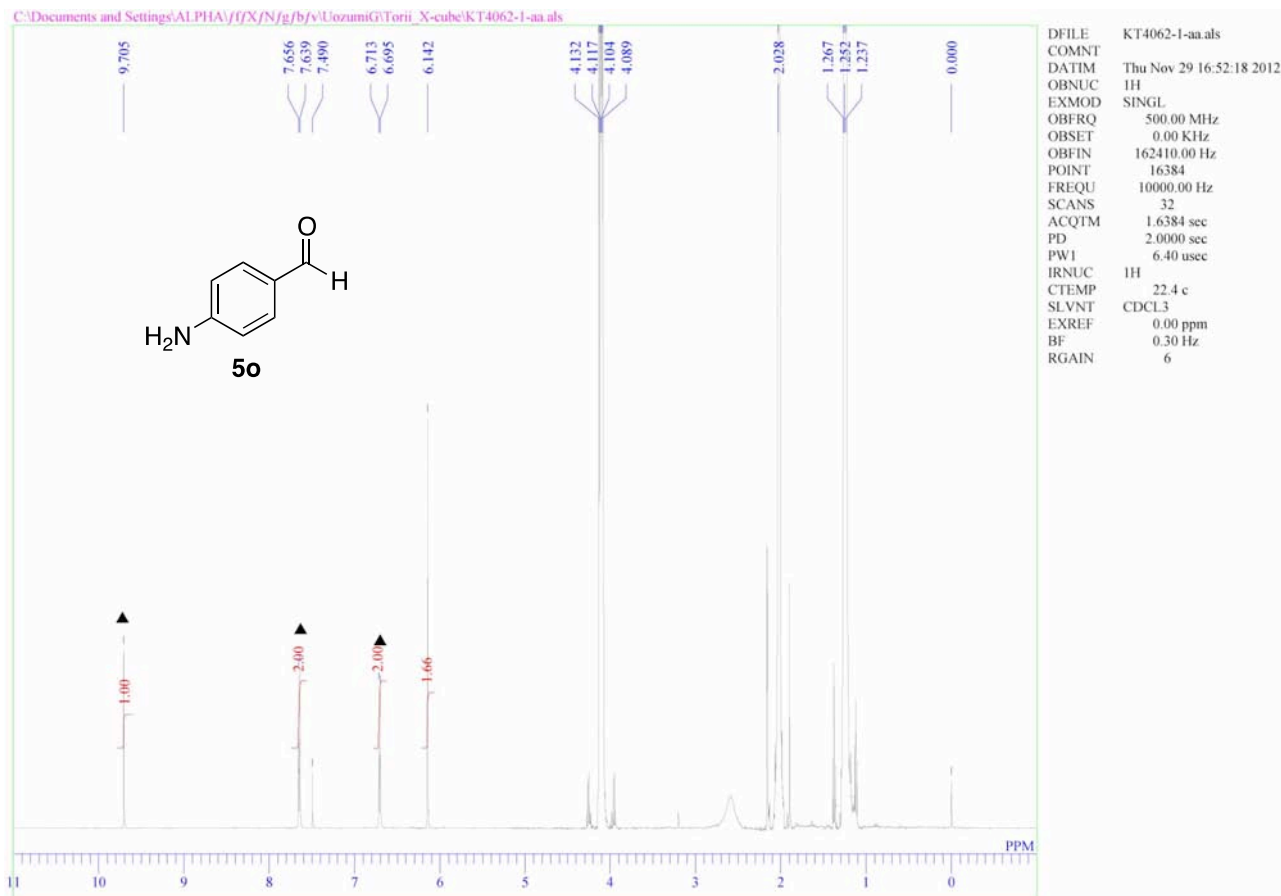
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 BF 0.00 Hz
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 TIMES 18000
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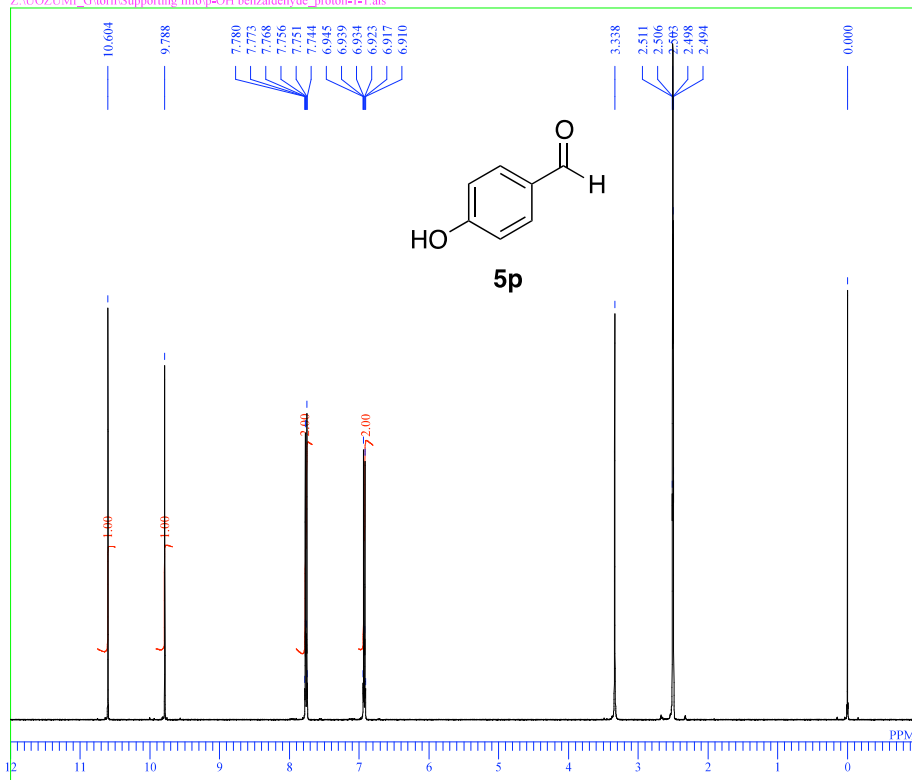
^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 4-aminobenzaldehyde (**5o**).



^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 4-hydroxybenzaldehyde (**5p**).

single_pulse

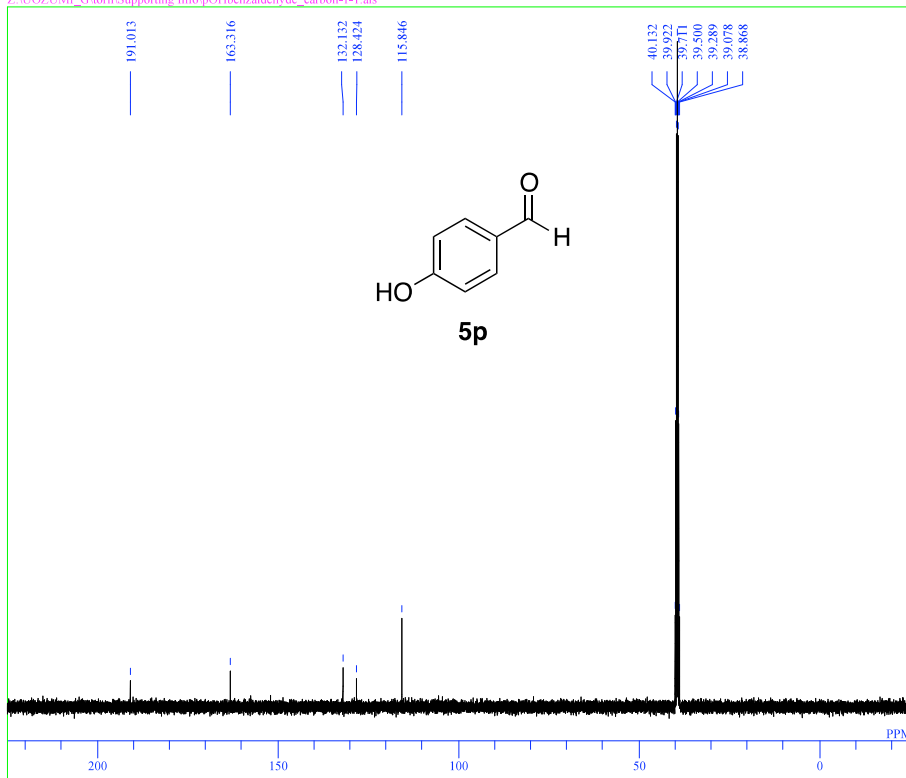
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single pulse decoupled gated NOE

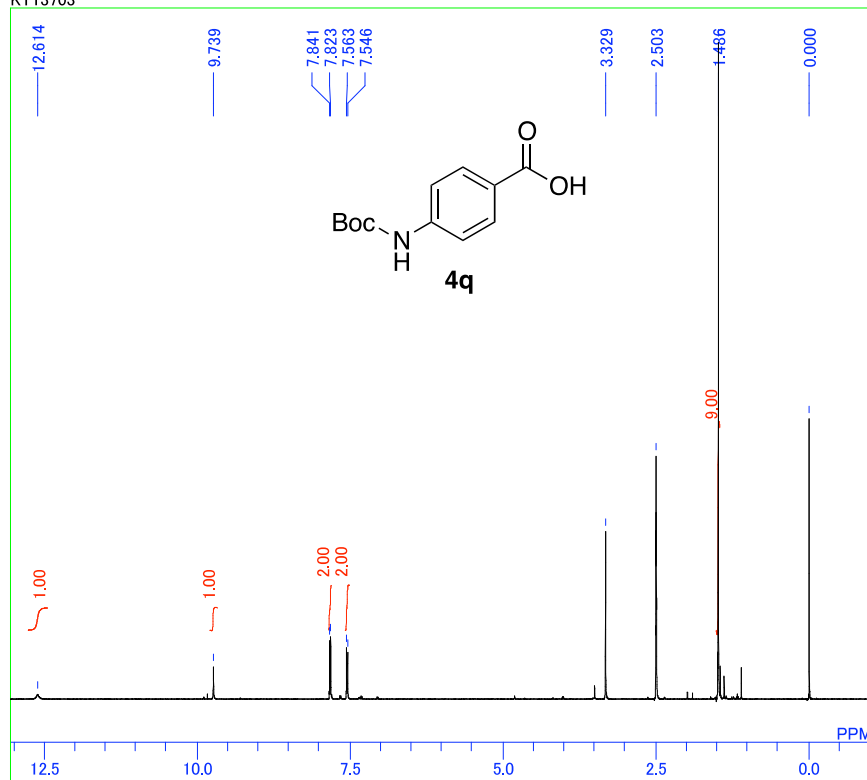
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 CTEMP 20.1 c
 SLVNT DMSO
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 RGAIN 60

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 4-(*tert*-butoxycarbonylamino)benzoic acid (**4q**).

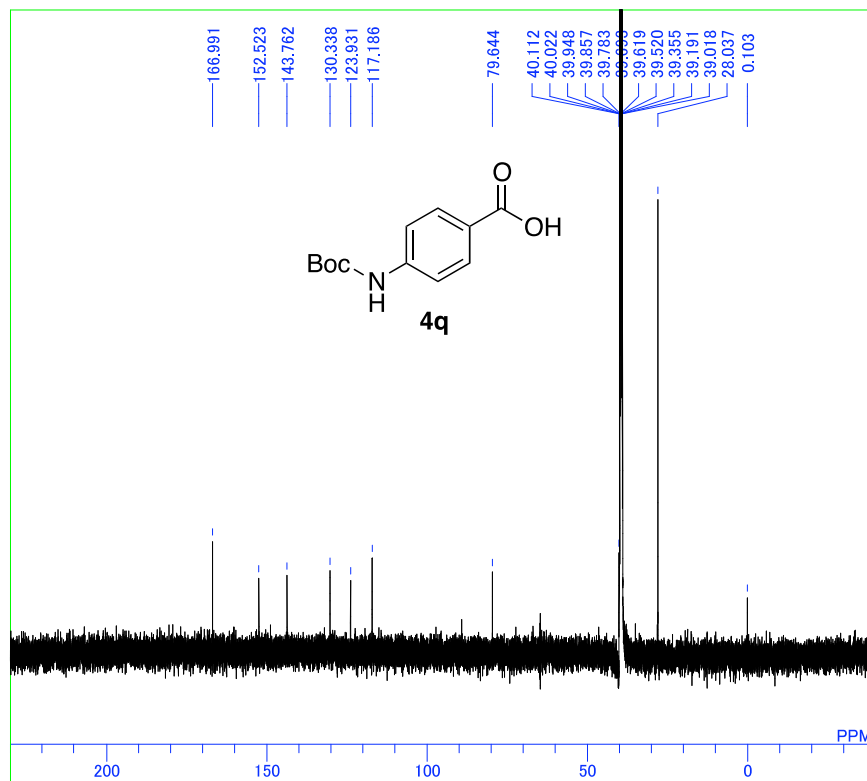
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DUMMY 1
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FILTR 5000 Hz
DELAY 40.00 usec
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PD 2.0000 sec
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T2 0.00
T3 90.00
T4 100.00
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EXPCM Single pulse
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IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
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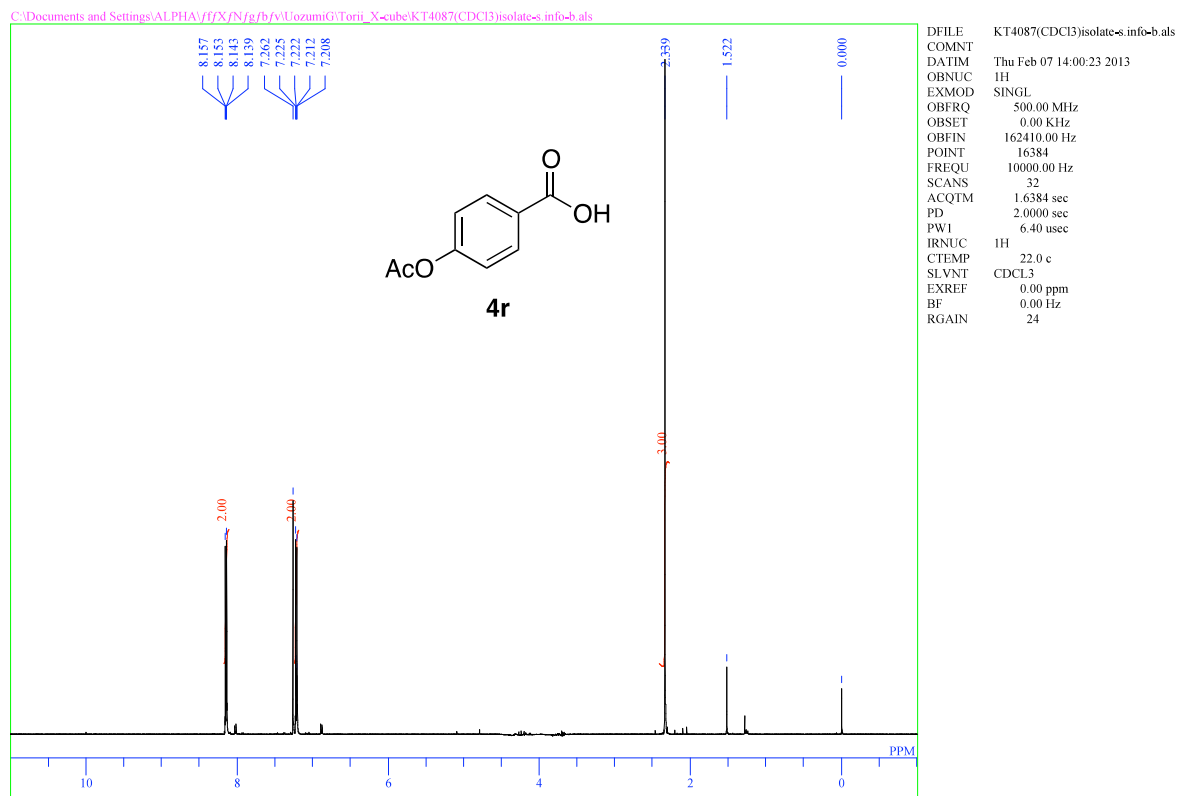
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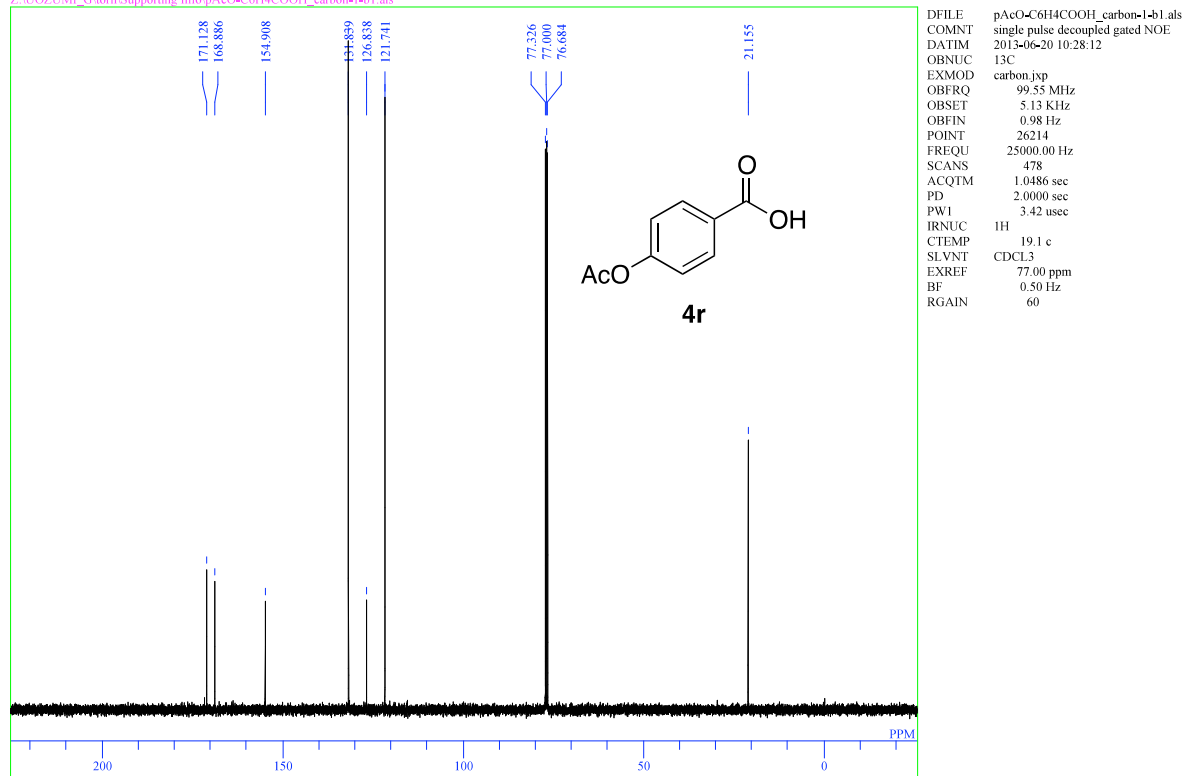
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T4 100.00
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EXPCM Single pulse
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IRFRQ 500.00 MHz
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LKPHS 214
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CSPED 18 Hz
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^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 4-acetoxybenzoic acid (**4r**).



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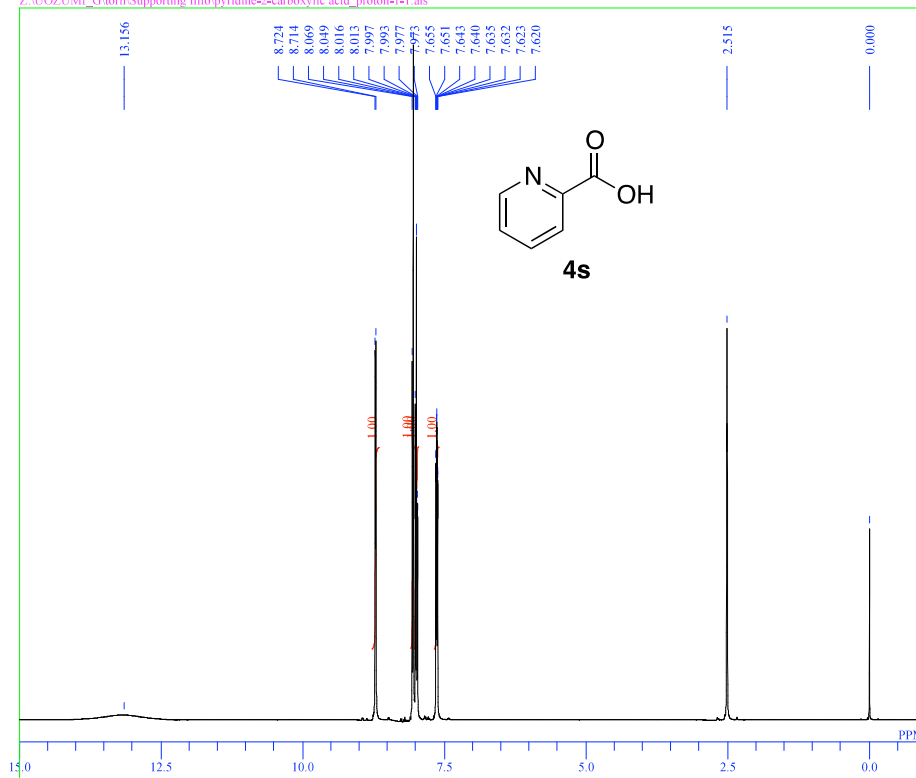
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^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 2-picolinic acid (**4s**).

single_pulse

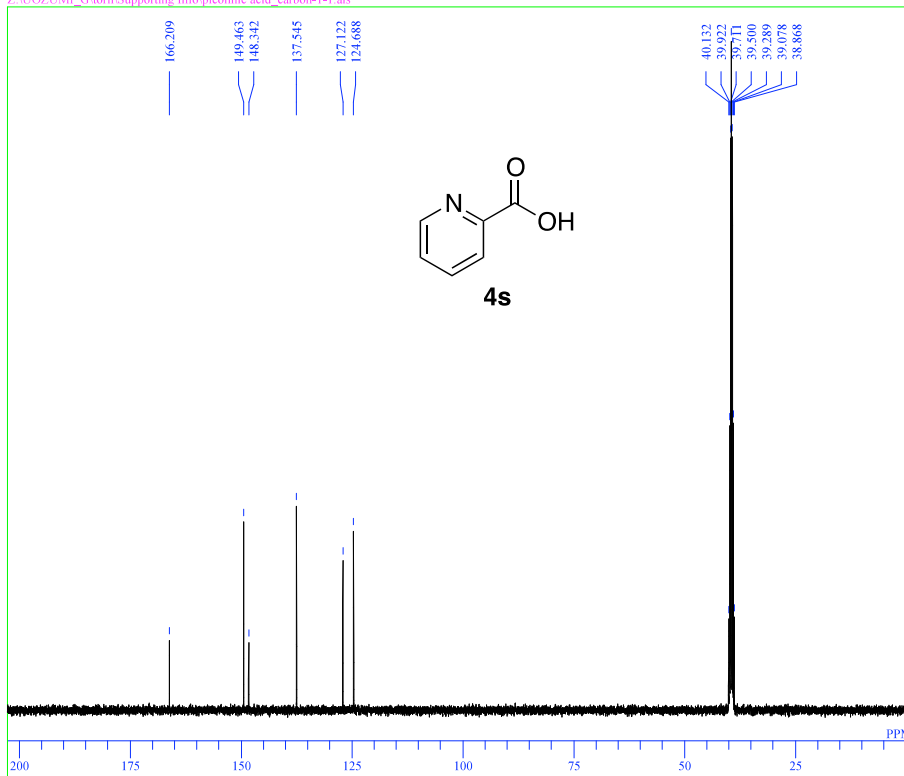
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pyridine-2-carboxylic acid_proton-1-1.als
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 PD 5.0000 sec
 PW1 3.12 usec
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 RGAIN 36

single pulse decoupled gated NOE

Z:\UOZUMI_G\Torii\Supporting Info\picolinic acid_carbon-1-1.als

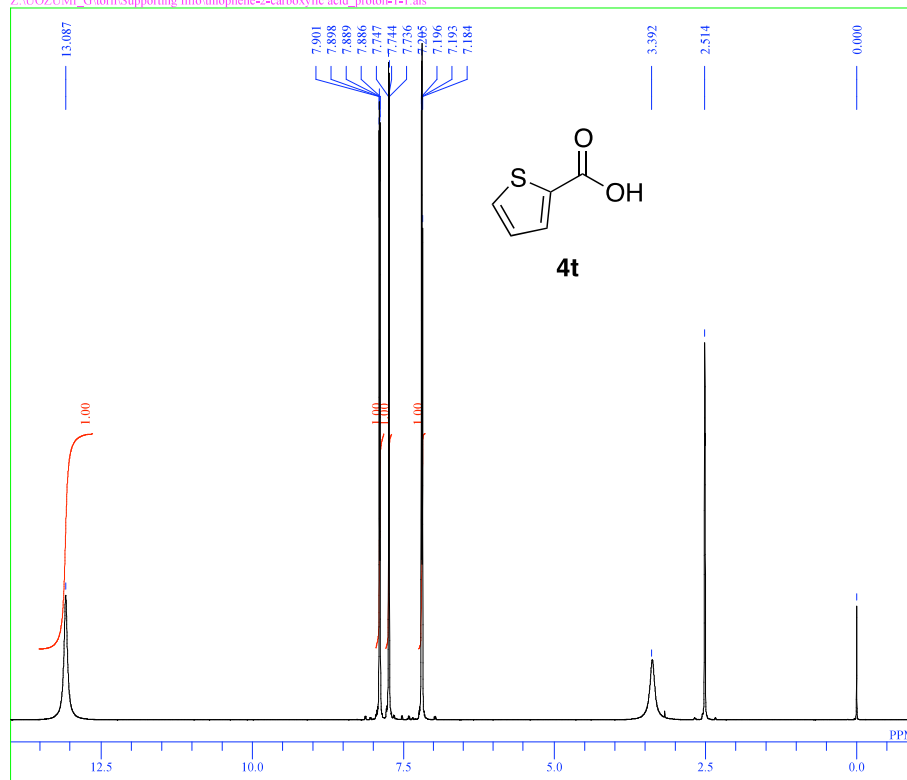


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 CTEMP 20.0 c
 SLVNT DMSO
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 RGAIN 60

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 2-thiophenecarboxylic acid (**4t**).

single_pulse

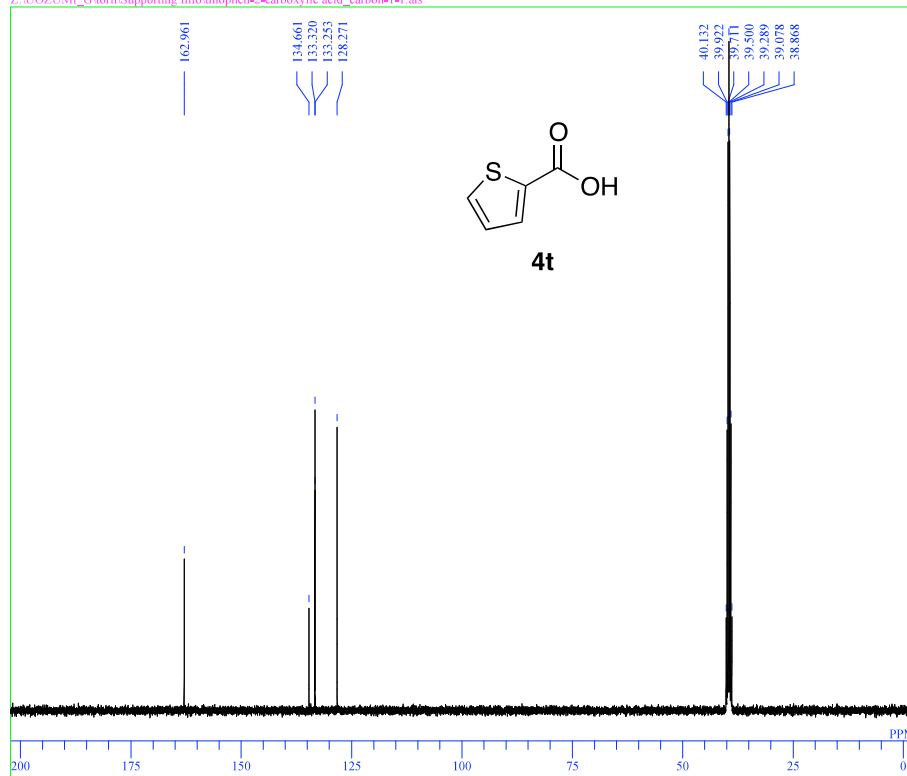
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thiophene-2-carboxylic acid_proton-1-1.als
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 PD 5.0000 sec
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single pulse decoupled gated NOE

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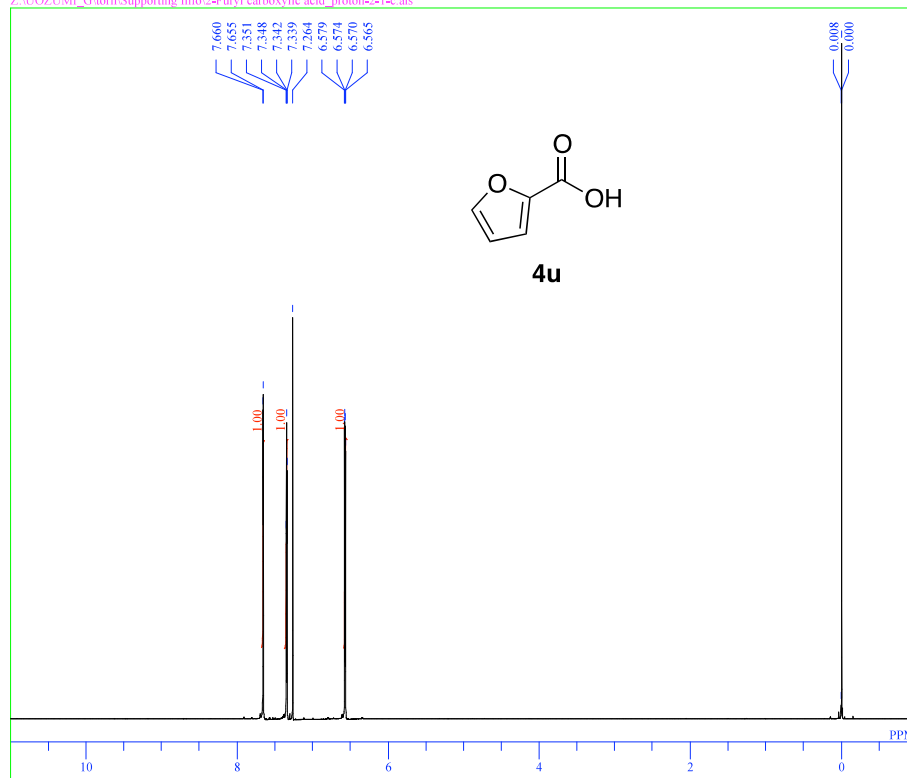


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 CTEMP 20.0 c
 SLVNT DMSO
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^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of 2-furancarboxylic acid (**4u**).

single_pulse

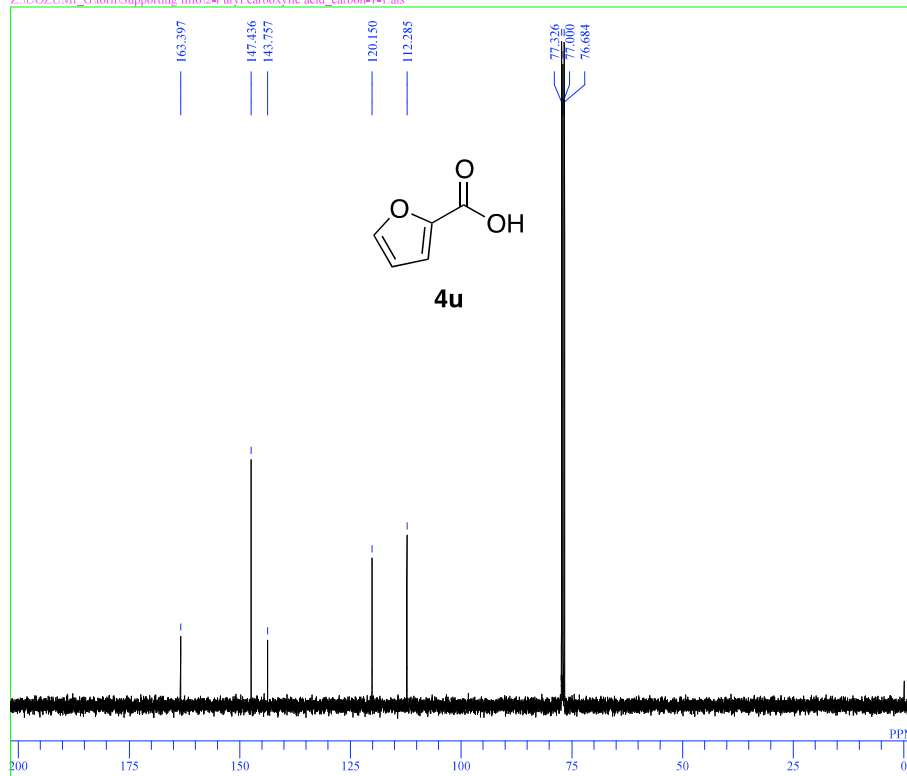
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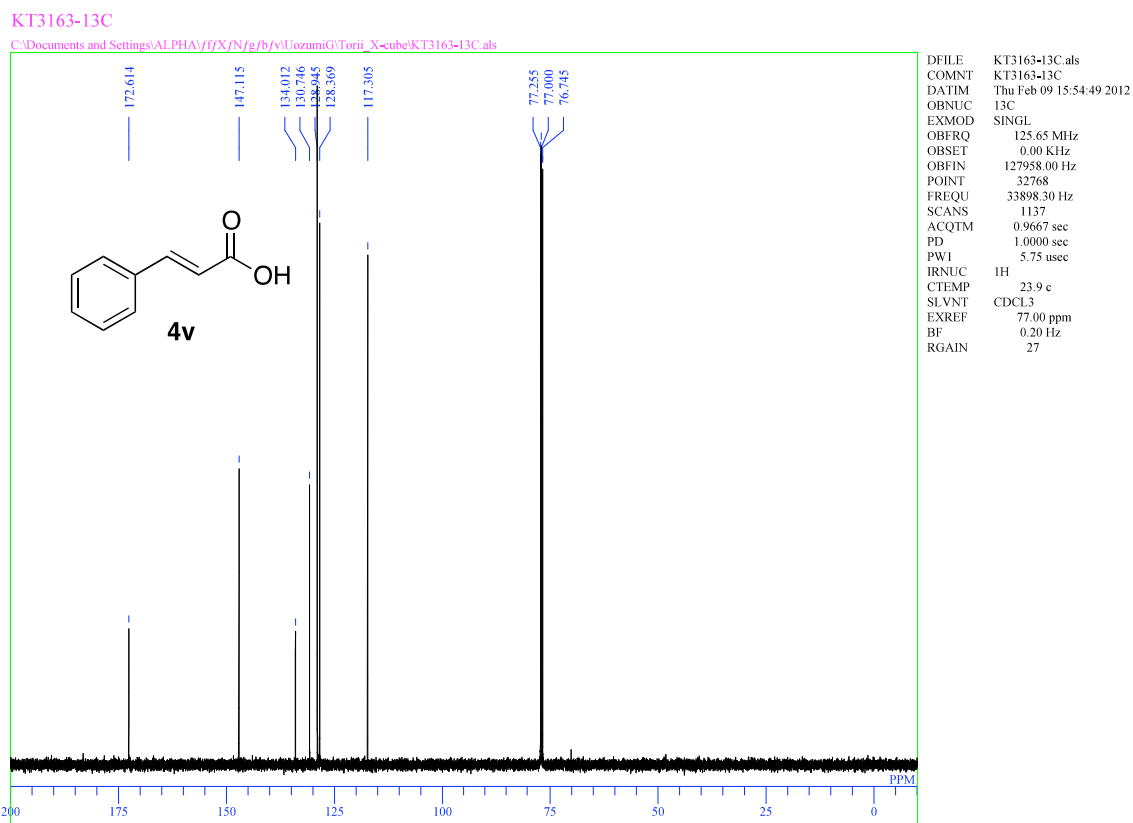
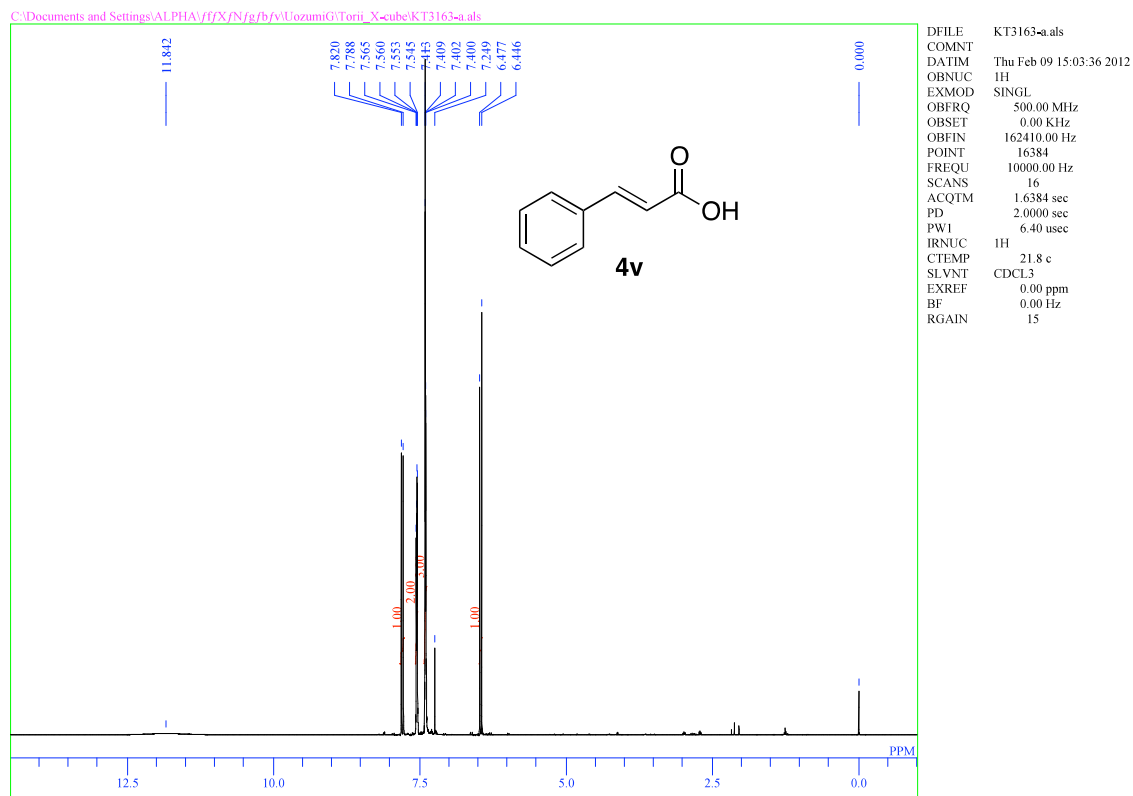
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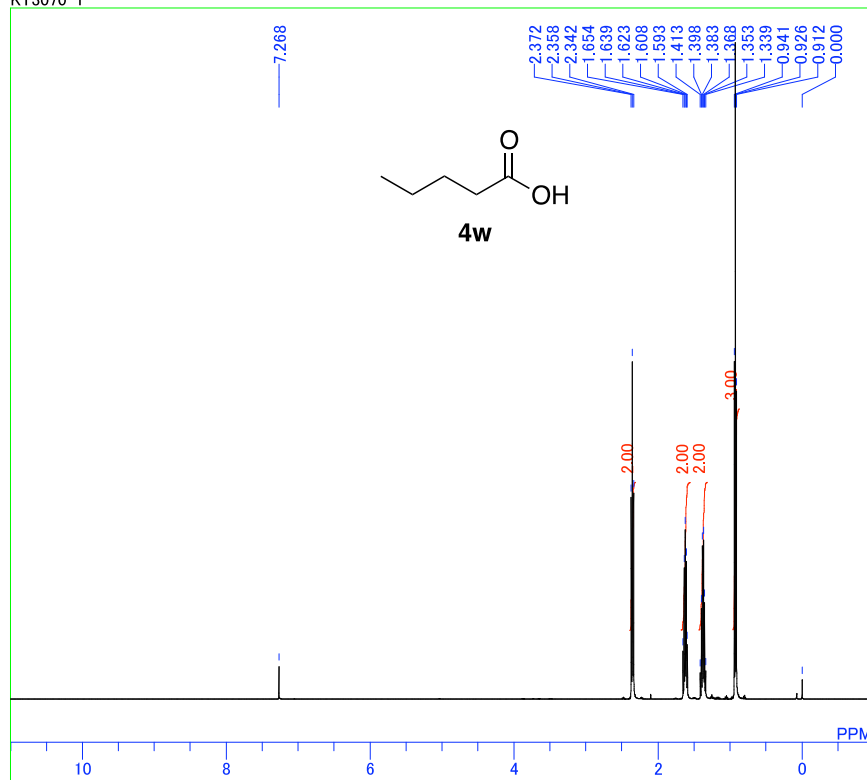
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EXREF 77.00 ppm
BF 0.80 Hz
RGAIN 60

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of cinnamic acid (**4v**).



^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of pentanoic acid (**4w**).

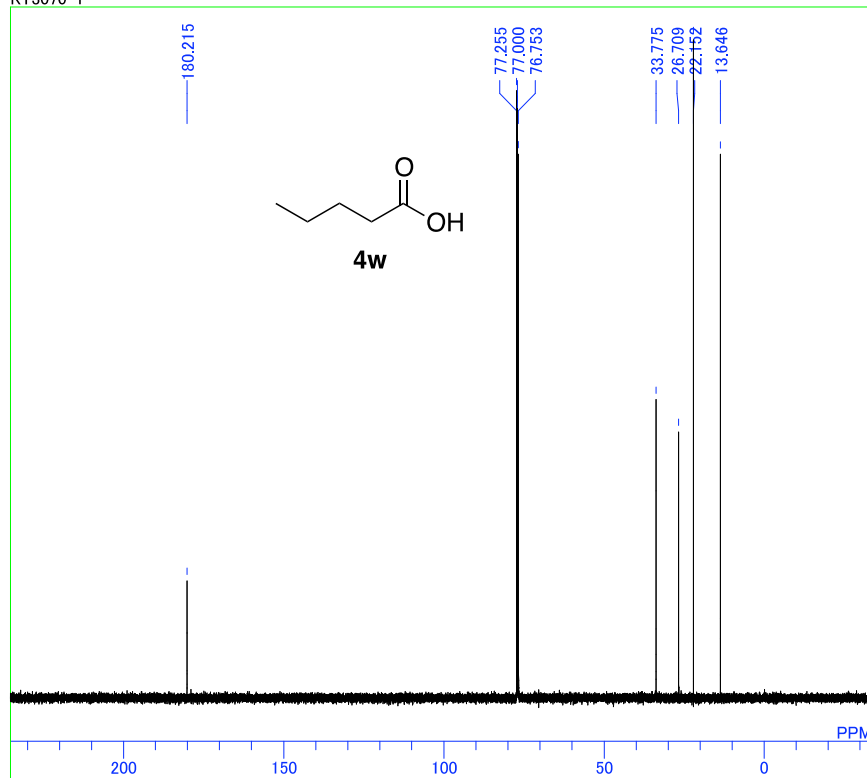
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KT3070-1

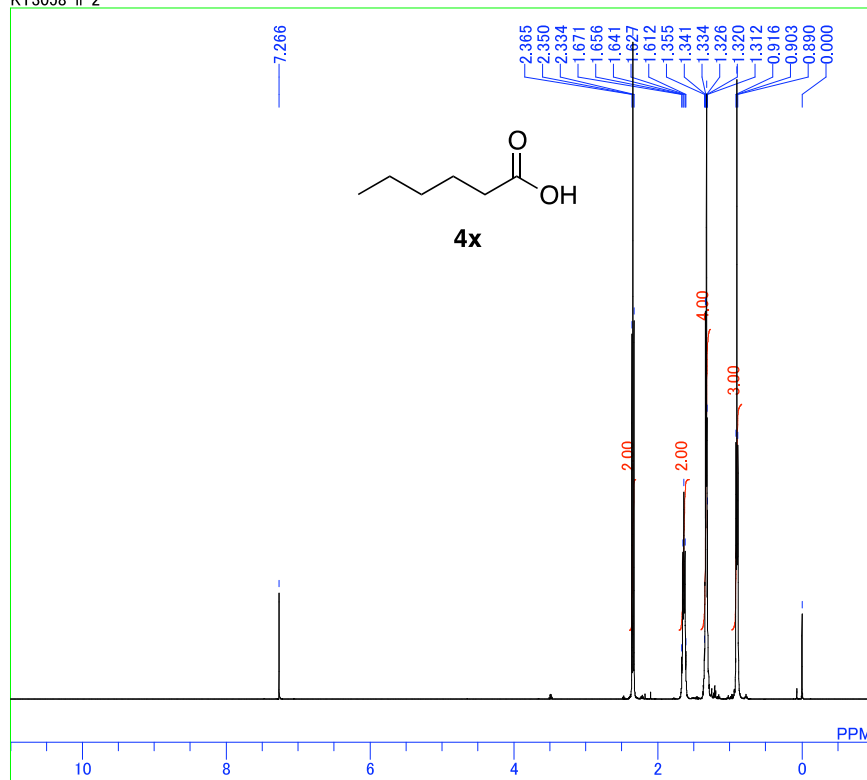


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INIWT 10.0000 msec
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^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of hexanoic acid (**4x**).

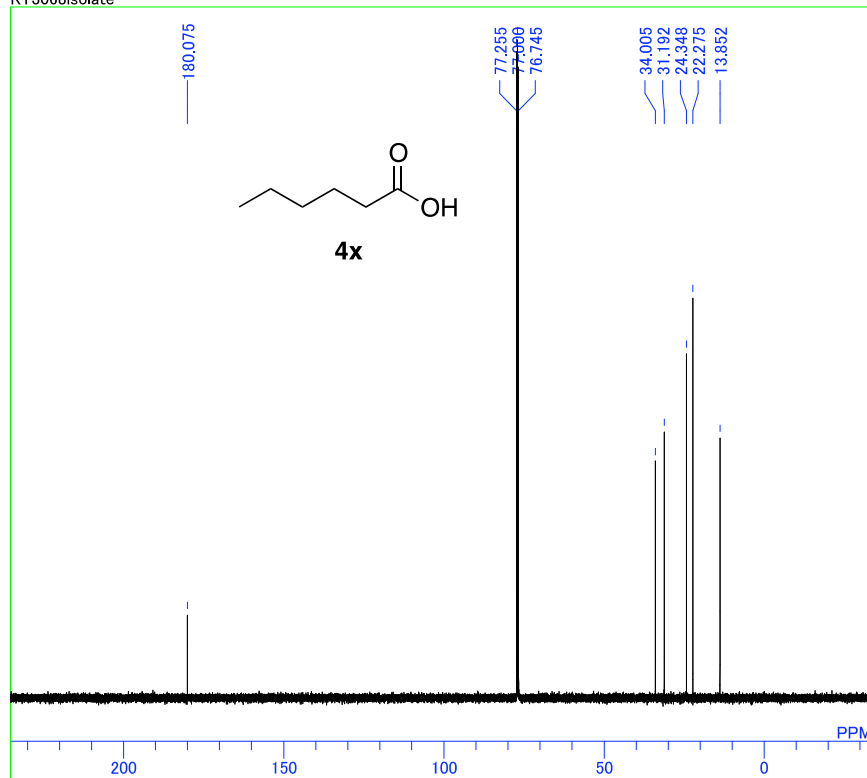
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KT3058-ii-2



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FREQU 10000.00 Hz
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BF 0.00 Hz
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T2 0.00
T3 90.00
T4 100.00
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EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
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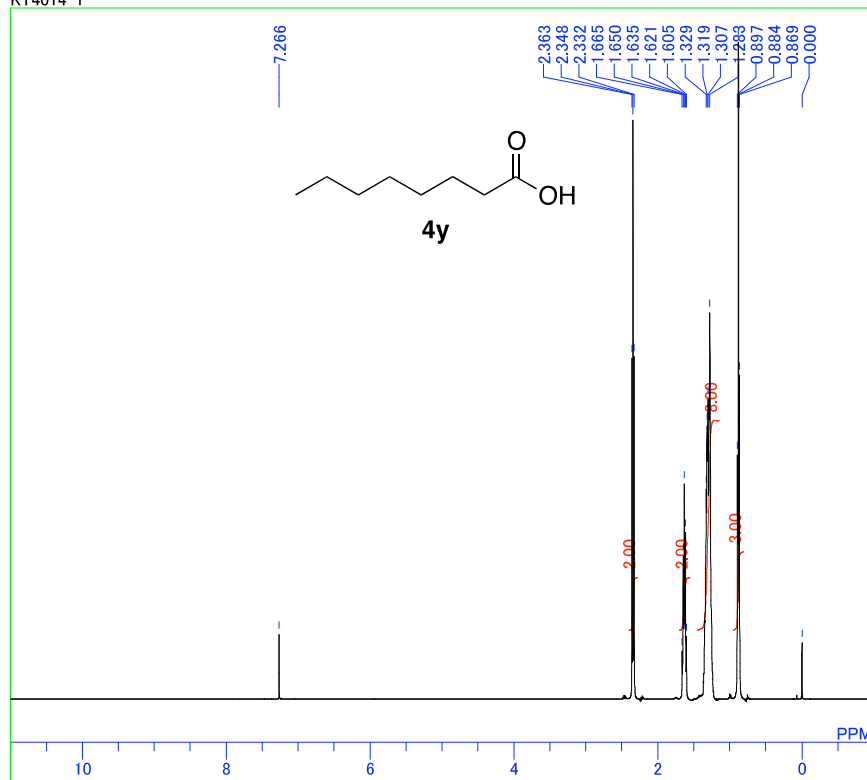


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EXPCM Single pulse
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^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of octanoic acid (**4y**).

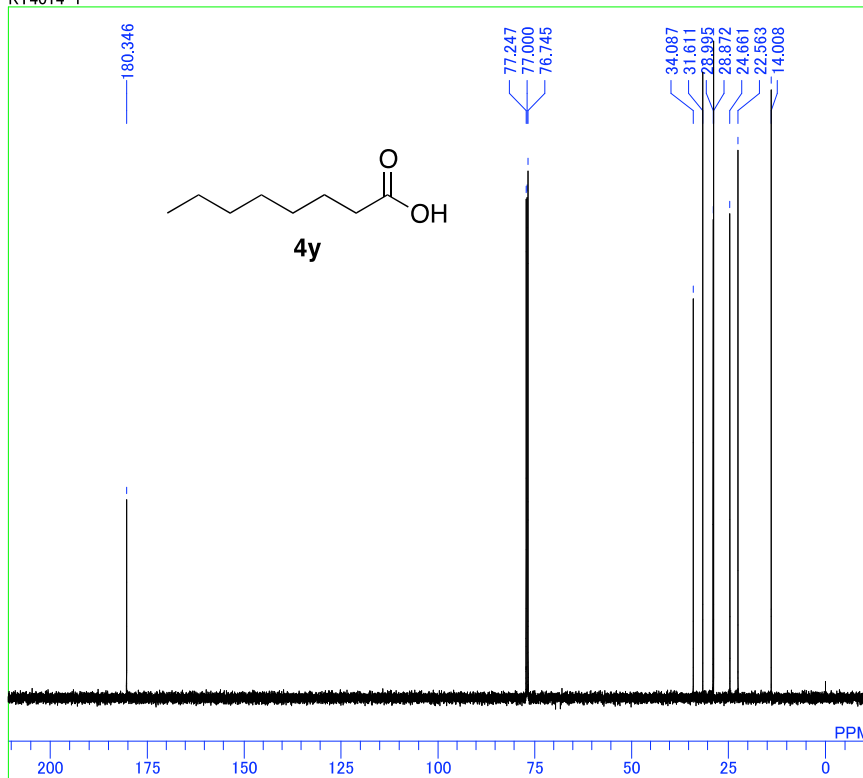
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KT4014-1



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PW1 6.40 usec
DEADT 56.80 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 16384
SAMPO 16384
TIMES 32
DUMMY 1
FREQU 10000.00 Hz
FILTR 5000 Hz
DELAY 40.00 usec
ACQTM 1.6384 sec
PD 2.0000 sec
ADBIT 16
RGAIN 13
BF 0.00 Hz
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T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
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IRRPW 50 usec
IRATN 511
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SHMFL TH5ATFG2
LKFIN 70334.0 Hz
LKLEV 180
LGAIN 21
LKPHS 214
LKSIG 639
CSPED 12 Hz
FILDC
FILDF
    
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KT4014-1

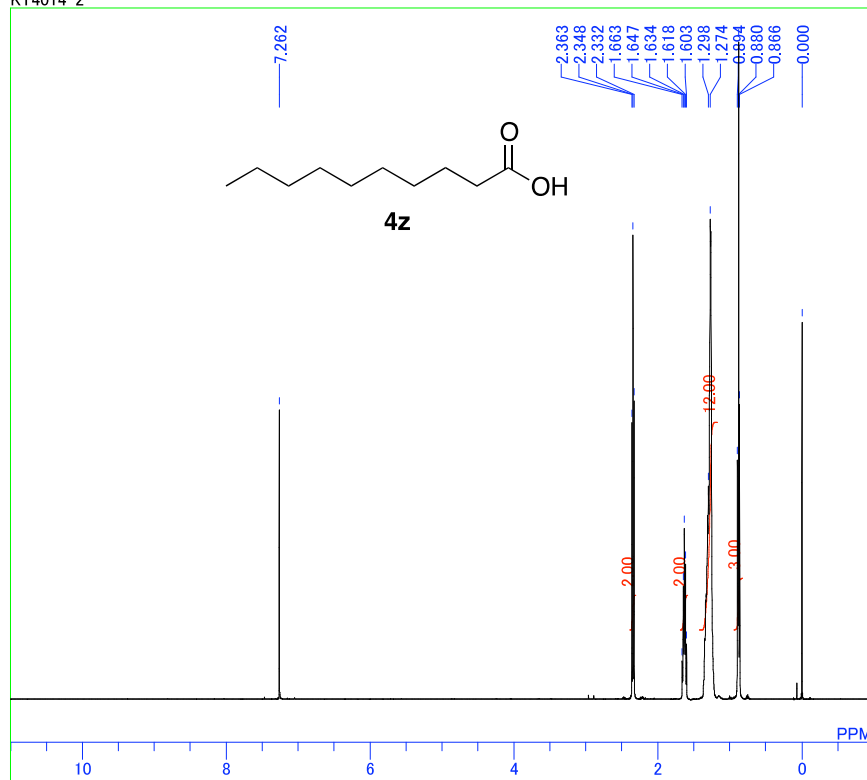


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TIMES 5000
DUMMY 1
FREQU 33898.30 Hz
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DELAY 11.80 usec
ACQTM 0.9667 sec
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RGAIN 27
BF 0.40 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\Documents and Settings\AL
SHMFL TH5ATFG2
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FILDC
FILDF
    
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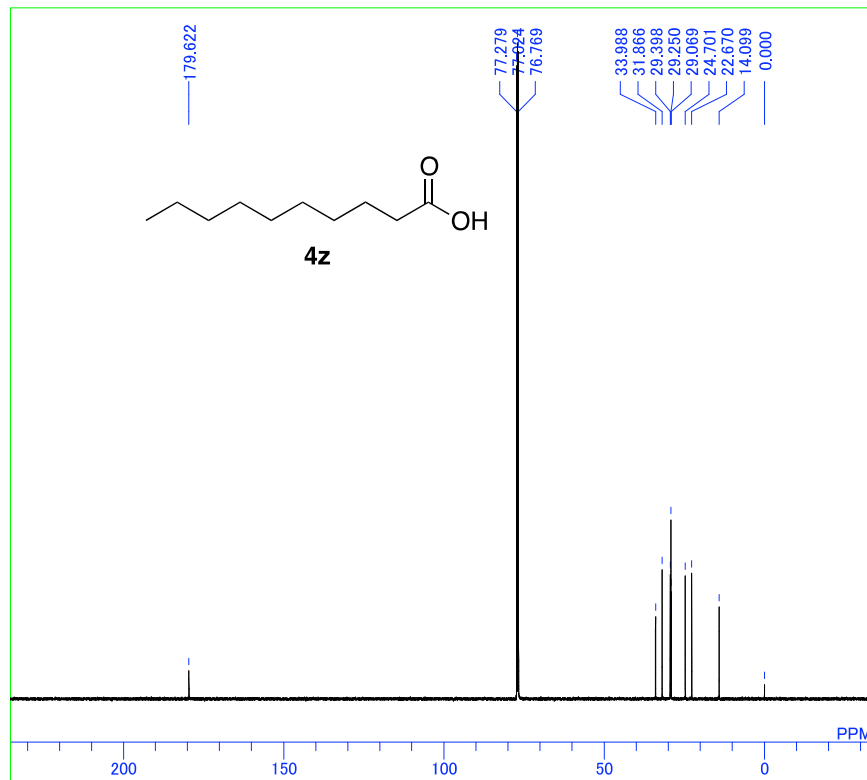
^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of decanoic acid (**4z**).

C:\Documents and Settings\ALPHA\1-1-1-1\UozumiG\Torii_X-cube\KT4014-Dec.als
KT4014-2"



MENUF non_th5atfTH5ATFG2
OBNUC 1H
OBFRQ 500.00 MHz
OBFIN 162410.00 Hz
PW1 6.40 usec
DEADT 56.80 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 16384
SAMPO 16384
TIMES 32
DUMMY 1
FREQU 10000.00 Hz
FILTR 5000 Hz
DELAY 40.00 usec
ACQTM 1.6384 sec
PD 2.0000 sec
ADBIT 16
RGAIN 18
BF 0.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\Documents and Settings\ALPHA\1-1-1-1\UozumiG\Torii_X-cube\KT4014-2-
SHMFL TH5ATFG2
LKFIN 70334.0 Hz
LKLEV 180
LGAIN 22
LKPHS 214
LKSIG 947
CSPED 14 Hz
FILDC
FILDF

C:\Documents and Settings\ALPHA\1-1-1-1\UozumiG\Torii_X-cube\KT4014-2-dec.als

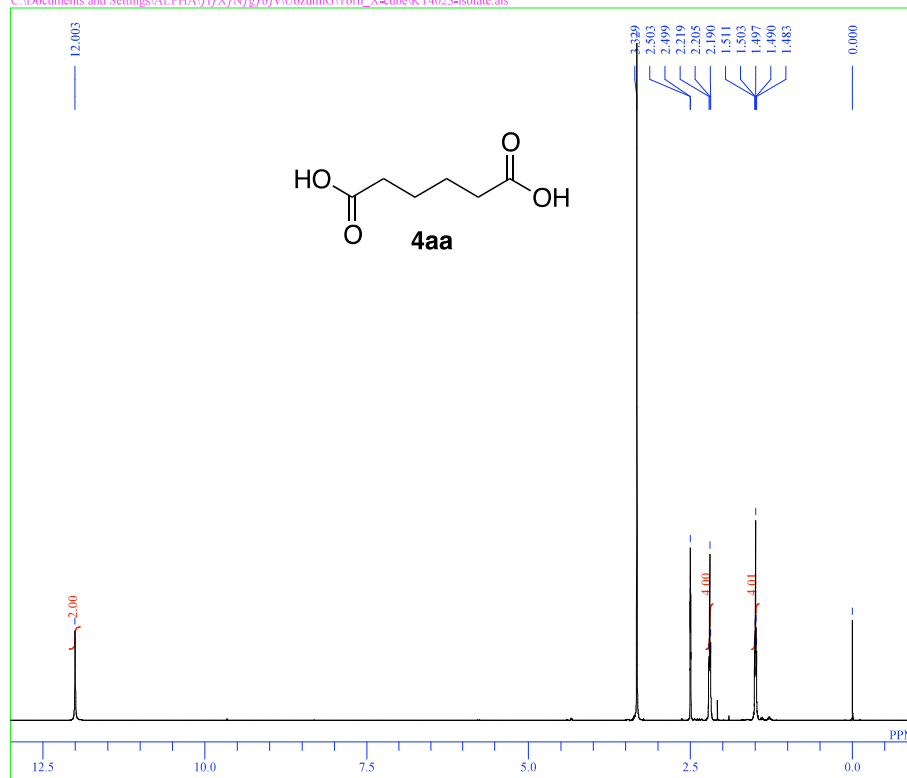


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PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 32768
SAMPO 32768
TIMES 16000
DUMMY 1
FREQU 33898.30 Hz
FILTR 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 1.0000 sec
ADBIT 16
RGAIN 26
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
IRATN 511
DFILE C:\Documents and Settings\ALPHA\1-1-1-1\UozumiG\Torii_X-cube\KT4014-2-
SHMFL TH5ATFG2
LKFIN 70334.0 Hz
LKLEV 180
LGAIN 25
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LKSIG 2370
CSPED 14 Hz
FILDC
FILDF

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of adipic acid (**4aa**).

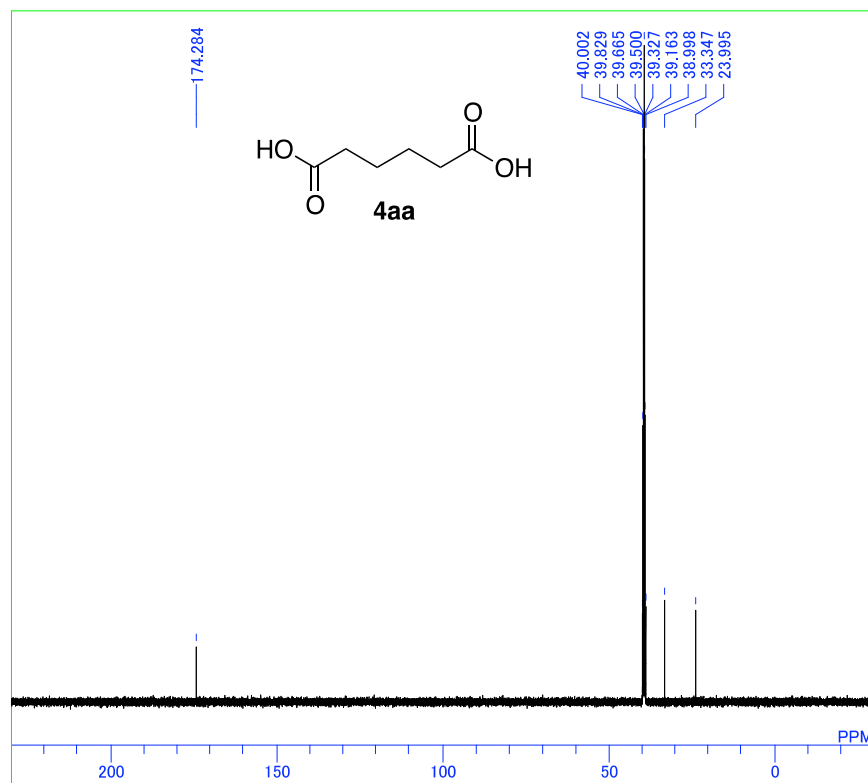
KT4023

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 EXMOD SINGL
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 OBSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 32
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PW1 6.40 usec
 IRNUC 1H
 CTEMP 24.1 c
 SLVNT DMSO
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 20

C:\Documents and Settings\ALPHA\ff\X\N\g\fb\UozumiG\Torii_X-cube\KT4023-isolate-13C-f.als

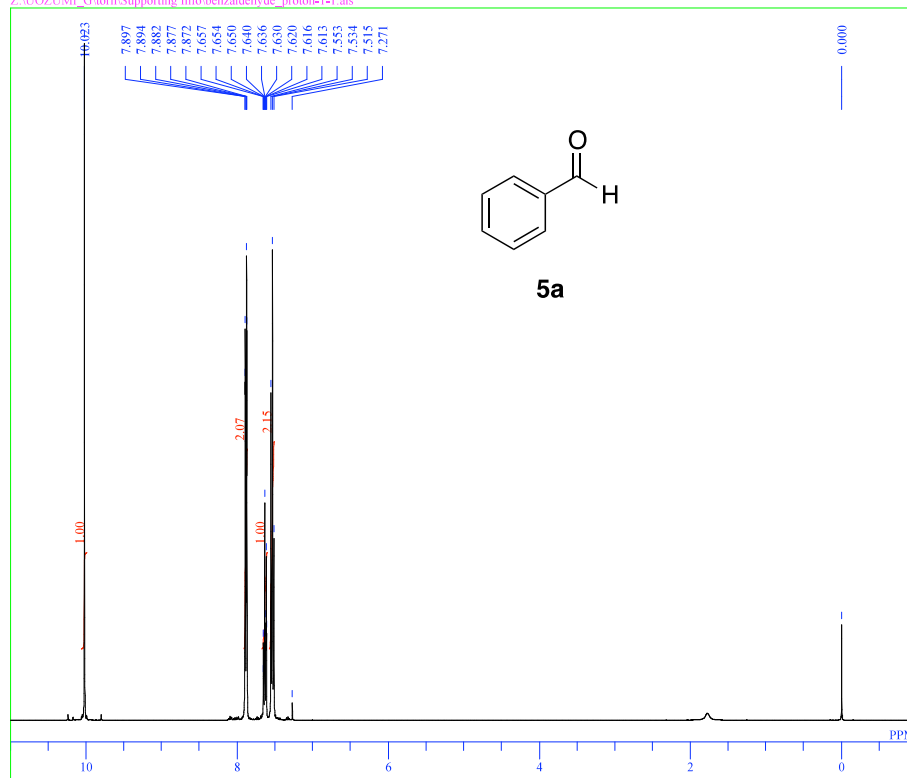


MENUB bcm_th5atfTH5ATFG2
 13C
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 OBFREQ 127958.00 Hz
 OBFIN 5.75 usec
 PW1 10.00 usec
 DEADT 0.20000 msec
 PREDL 10.0000 msec
 INIWT 32768
 POINT 32768
 SAMPO 5000
 TIMES 1
 DUMMY 33898.30 Hz
 FREQU 16950 Hz
 FILTR 11.80 usec
 DELAY 0.9667 sec
 ACQTM 1.0000 sec
 PD 16
 ADBIT 25
 RGAIN 0.00 Hz
 BF 0.00 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD SINGL
 EXPCM Single pulse
 IRNUC 1H
 IRFREQ 500.00 MHz
 IRFIN 162410.00 Hz
 IRRPW 50 usec
 IRATN 511
 DFILE C:\Documents and Settings\ALPHA\ff\X\N\g\fb\UozumiG\Torii_X-cube\KT4023-isolate-13C-f.als
 SHMFL TH5ATFG2
 LKFIN 69970.0 Hz
 LKLEV 160
 LGAIN 18
 LKPHS 214
 LKSIG 687
 CSPED 16 Hz
 FILDC
 FILDF

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of benzaldehyde (**5a**).

single_pulse

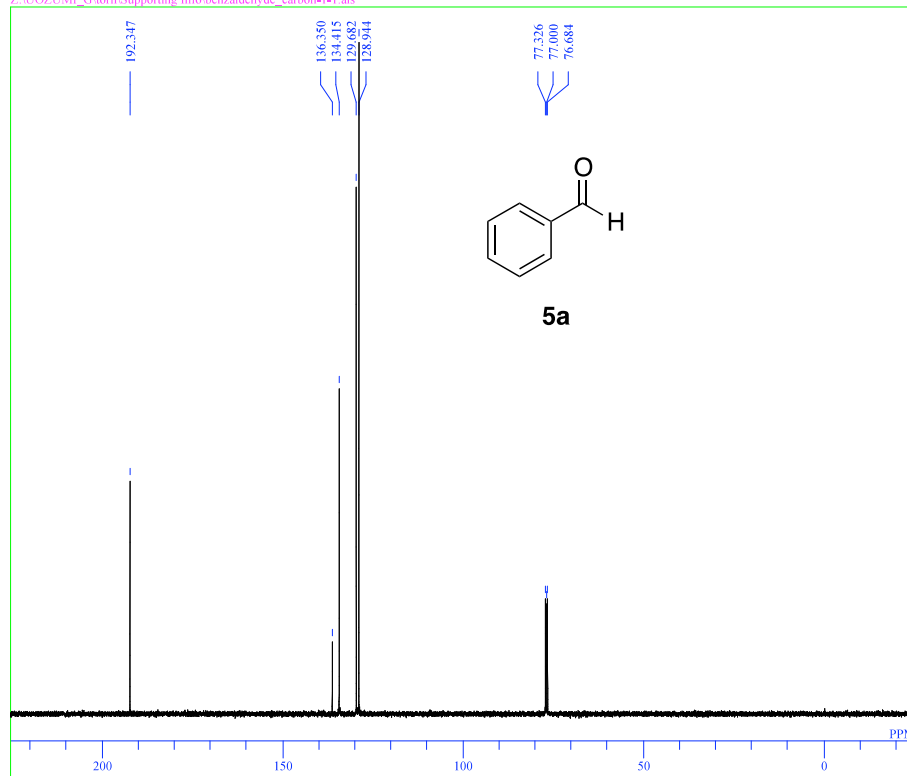
Z:\UOZUMI_G\Torii\Supporting Info\benzaldehyde_proton-1-1.als



DFILE benzaldehyde_proton-1-1.als
 COMNT single_pulse
 DATIM 2013-07-11 11:38:22
 OBNUC ^1H
 EXMOD proton.jsp
 OBFREQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.12 usec
 IRNUC ^1H
 CTEMP 22.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.30 Hz
 RGAIN 32

single pulse decoupled gated NOE

Z:\UOZUMI_G\Torii\Supporting Info\benzaldehyde_carbon-1-1.als

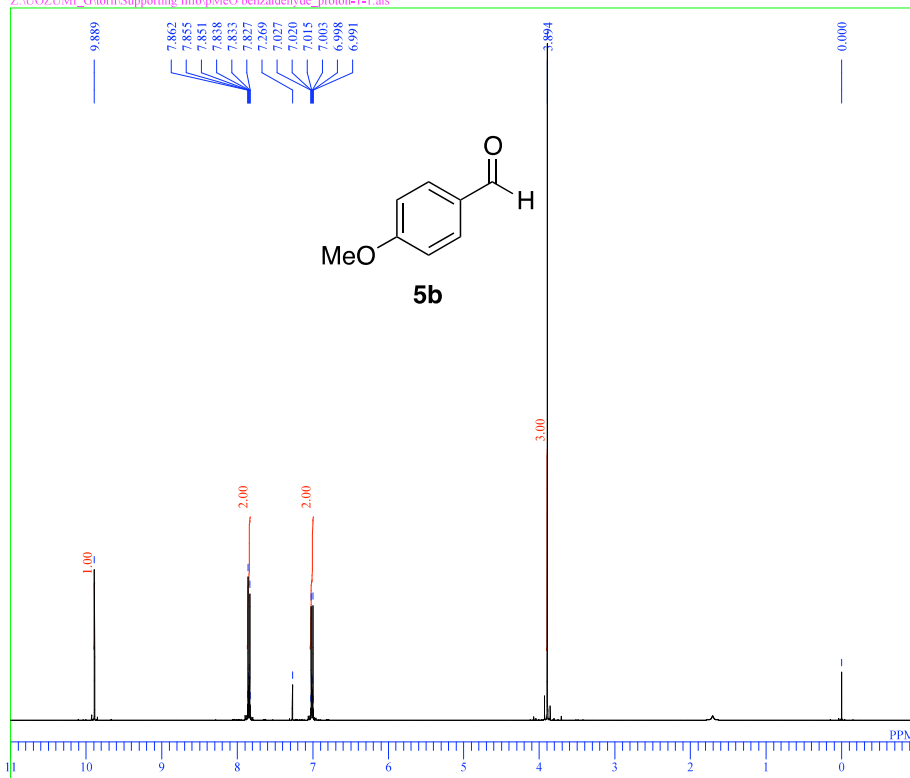


DFILE benzaldehyde_carbon-1-1.als
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 EXMOD carbon.jsp
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 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 237
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 IRNUC ^{13}C
 CTEMP 22.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.30 Hz
 RGAIN 60

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of *p*-anisaldehyde (**5b**).

single_pulse

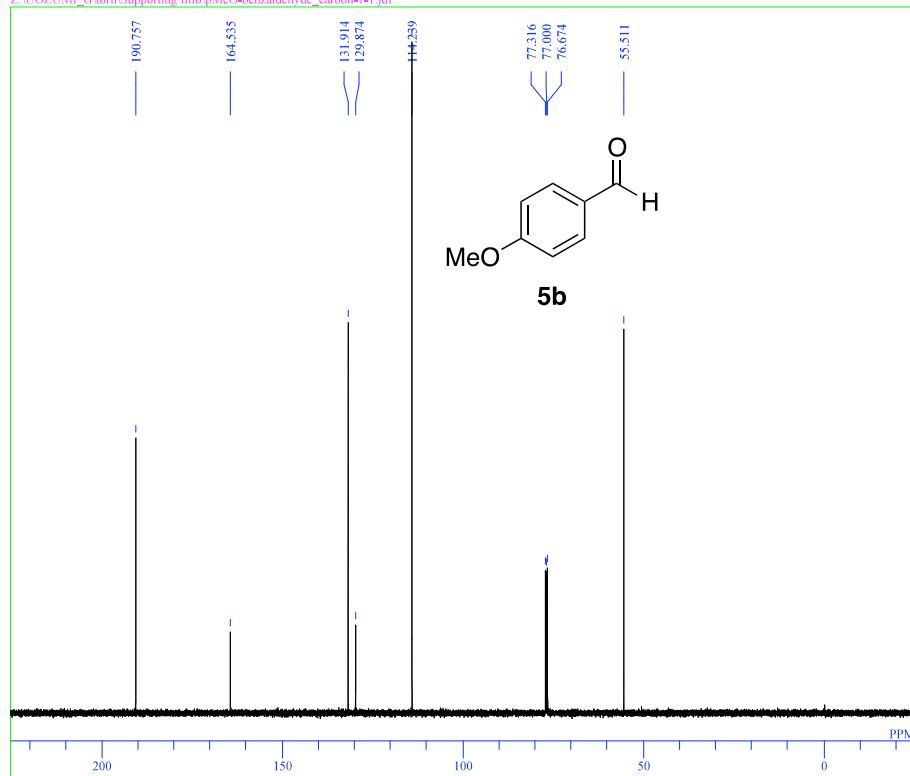
Z:\UOZUMI_G\Torii\Supporting_Info\pMeO benzaldehyde_proton-1-1.als



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 EXMOD proton.jsp
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 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 17
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.12 usec
 IRNUC ^1H
 CTEMP 21.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 34

single pulse decoupled gated NOE

Z:\UOZUMI_G\Torii\Supporting_Info\pMeO-benzaldehyde_carbon-1-1.jdf

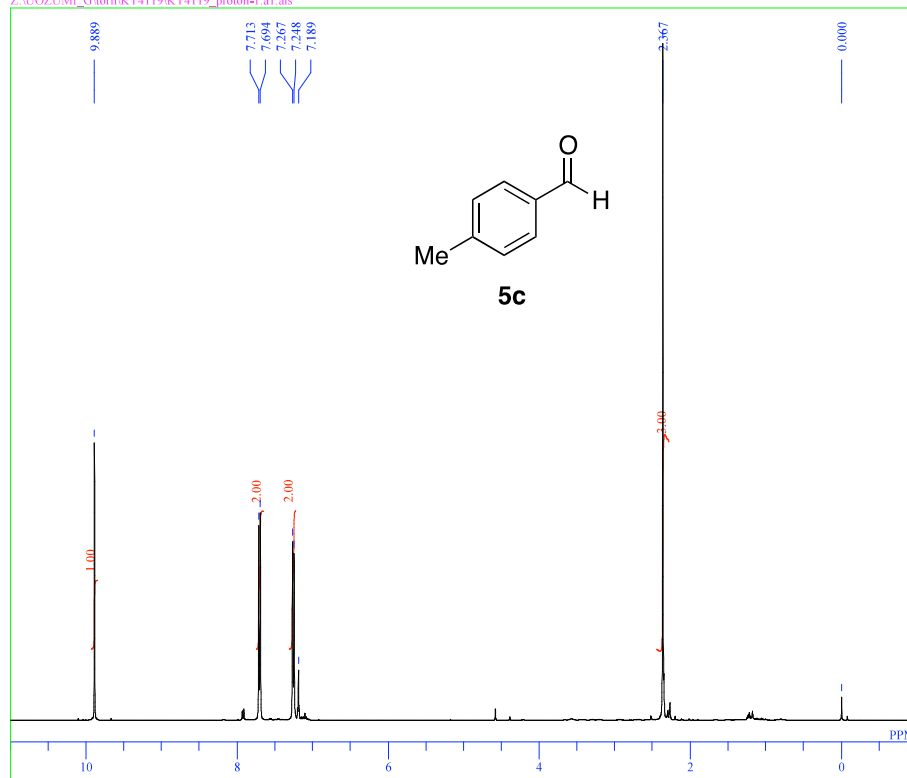


DFILE pMeO-benzaldehyde_carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2013-07-11 11:23:32
 OBNUC ^{13}C
 EXMOD carbon.jsp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 176
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 IRNUC ^1H
 CTEMP 22.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.30 Hz
 RGAIN 58

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of *p*-tolualdehyde (**5c**).

single_pulse

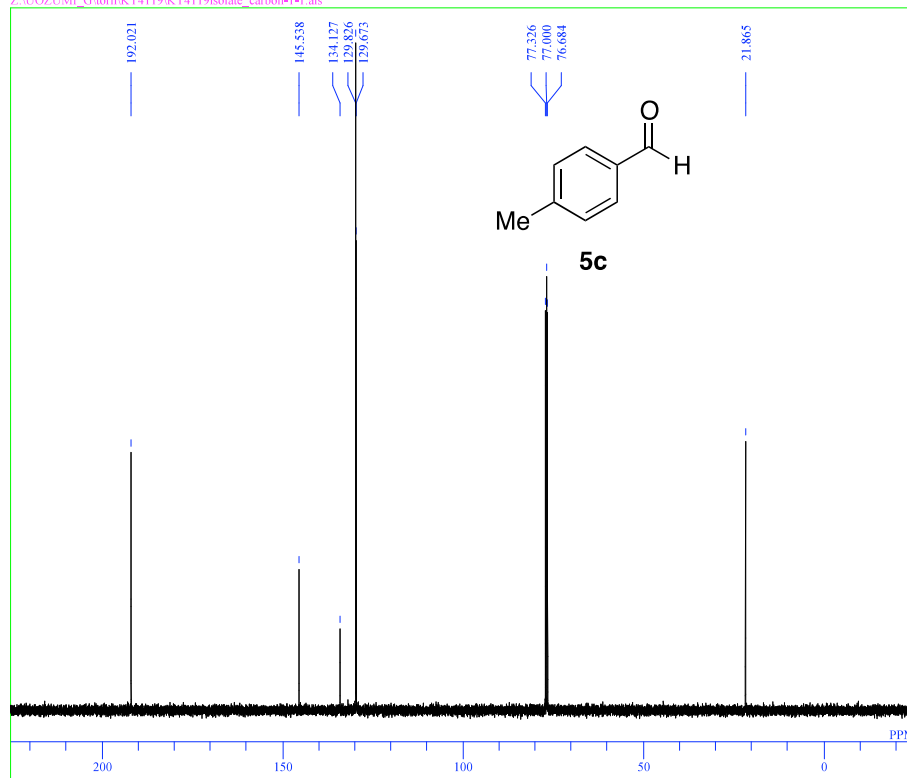
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COMNT single_pulse
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OBNUC ^1H
EXMOD proton.jsp
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.24 Hz
SCANS 16
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 3.12 usec
IRNUC ^1H
CTEMP 19.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 30

single pulse decoupled gated NOE

Z:\UOZUMI_G\Tori\KT4119\KT4119isolate_carbon-1-1.als

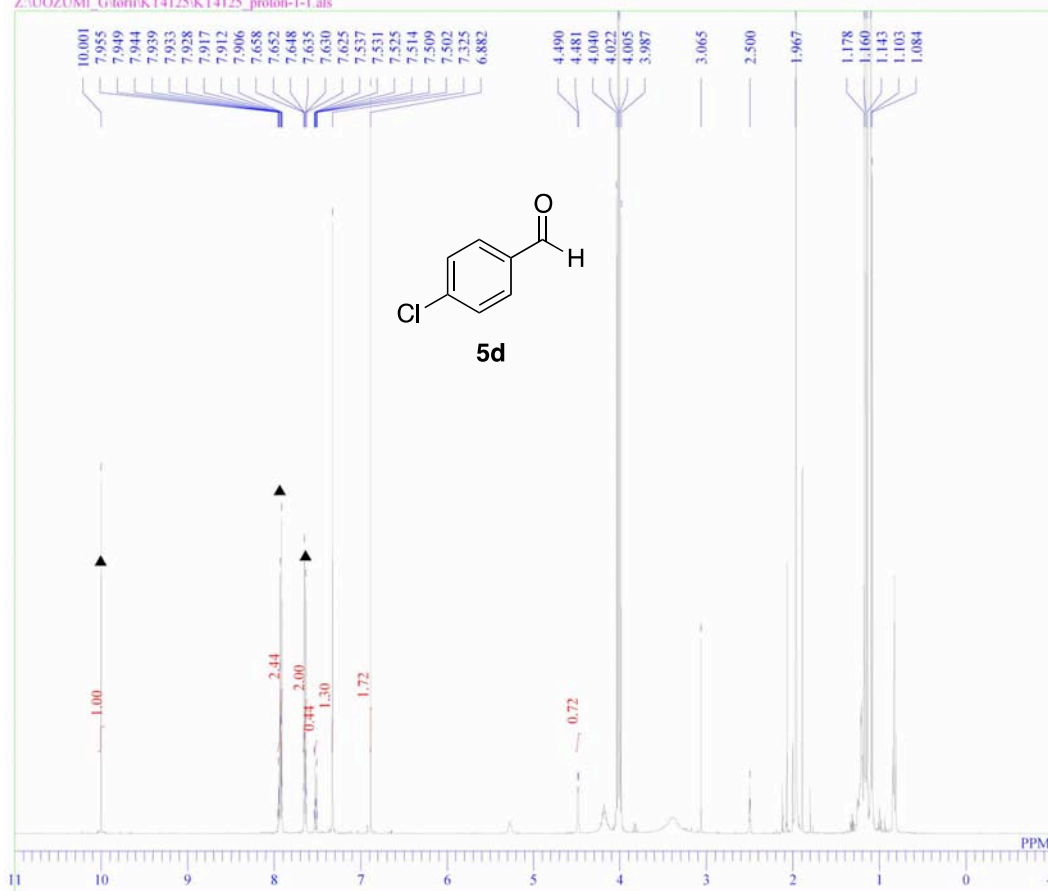


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EXMOD carbon.jsp
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 32767
FREQU 31250.00 Hz
SCANS 224
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.42 usec
IRNUC ^1H
CTEMP 18.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.60 Hz
RGAIN 60

¹H NMR spectra of *p*-chlorobenzaldehyde (**5d**).

single_pulse

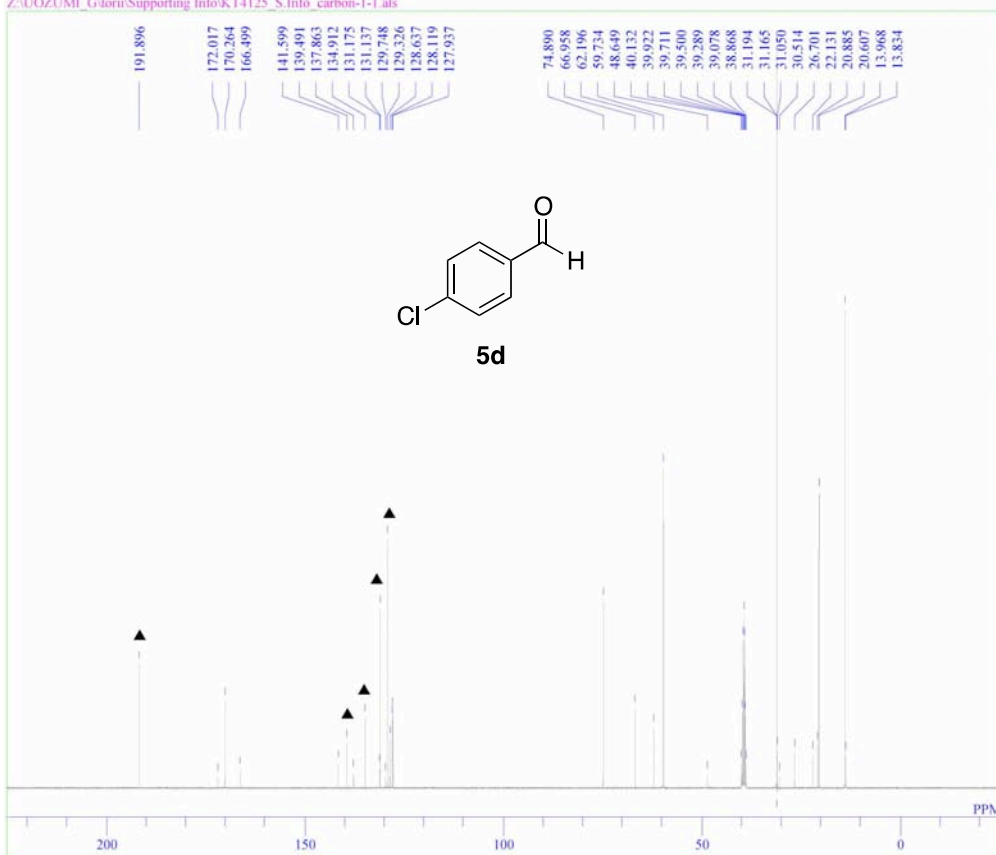
Z:\UOZUMI_G\toni\KT4125\KT4125_proton-1-1.als



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EXMOD	proton.jsp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	16
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	19.1 c
SLVNT	DMSO
EXREF	2.50 ppm
BF	0.00 Hz
RGAIN	16

single pulse decoupled gated NOE

Z:\UOZUMI_G\toni\Supporting Info\KT4125_S.Info_carbon-1-1.als

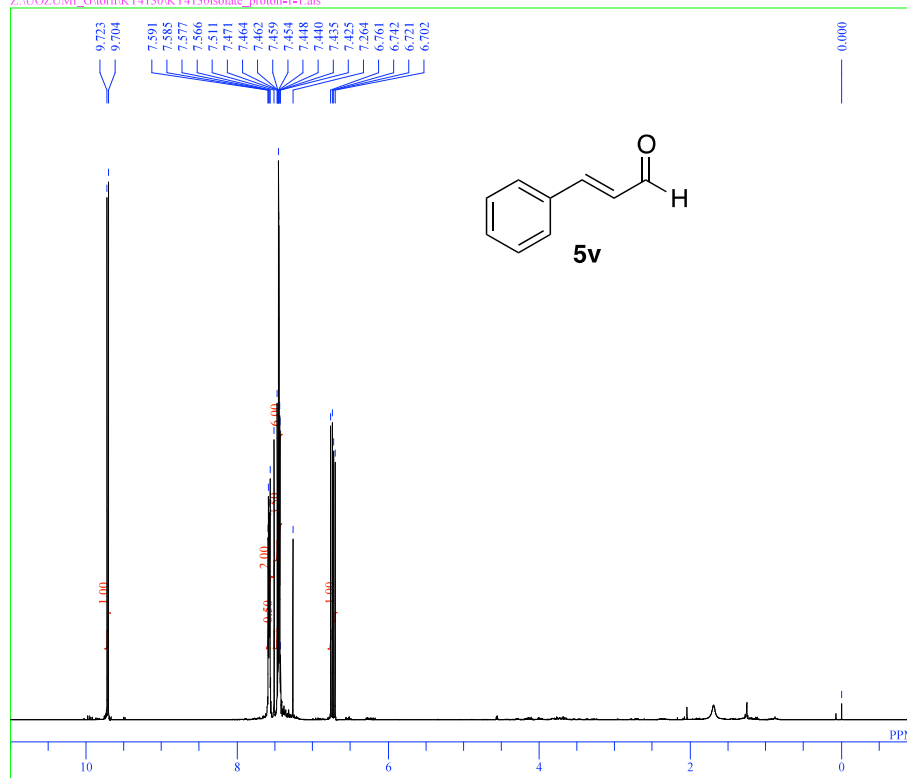


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OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	886
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.42 usec
IRNUC	1H
CTEMP	22.5 c
SLVNT	DMSO
EXREF	39.50 ppm
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RGAIN	60

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of cinnamaldehyde (**5v**).

single_pulse

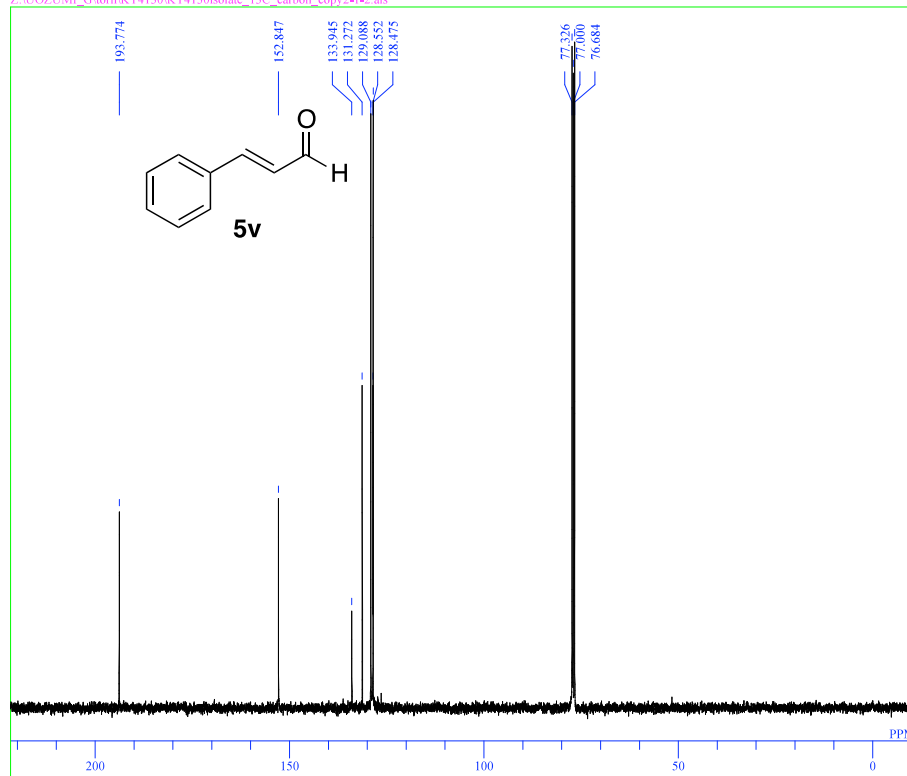
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 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.12 usec
 IRNUC ^1H
 CTEMP 19.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 34

single pulse decoupled gated NOE

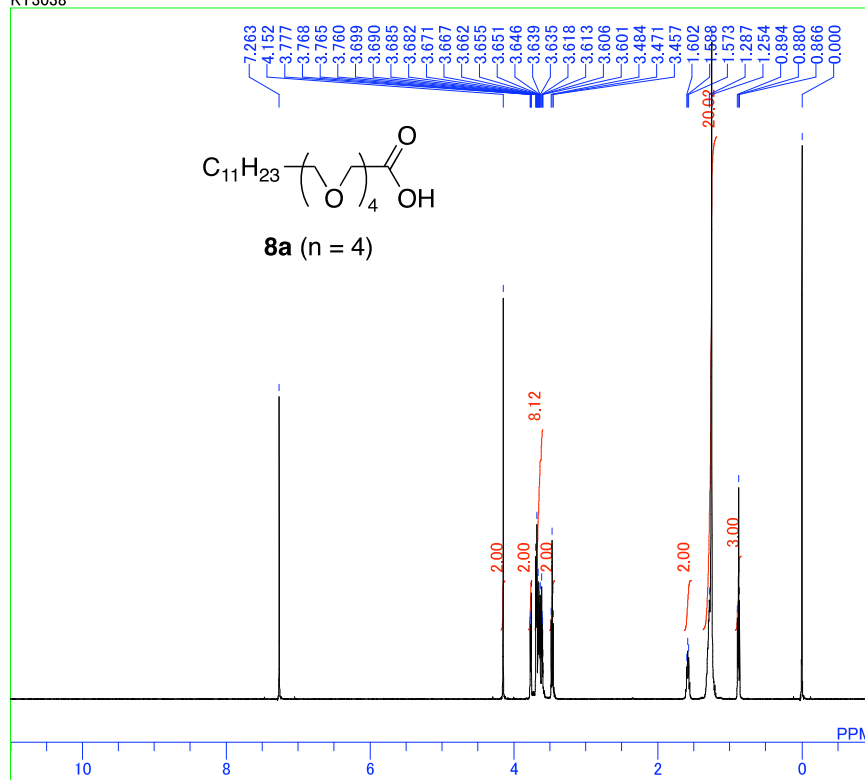
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 OBFIN 0.98 Hz
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 FREQU 39062.50 Hz
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 ACQTM 0.0000 sec
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 CTEMP 20.6 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.00 Hz
 RGAIN 60

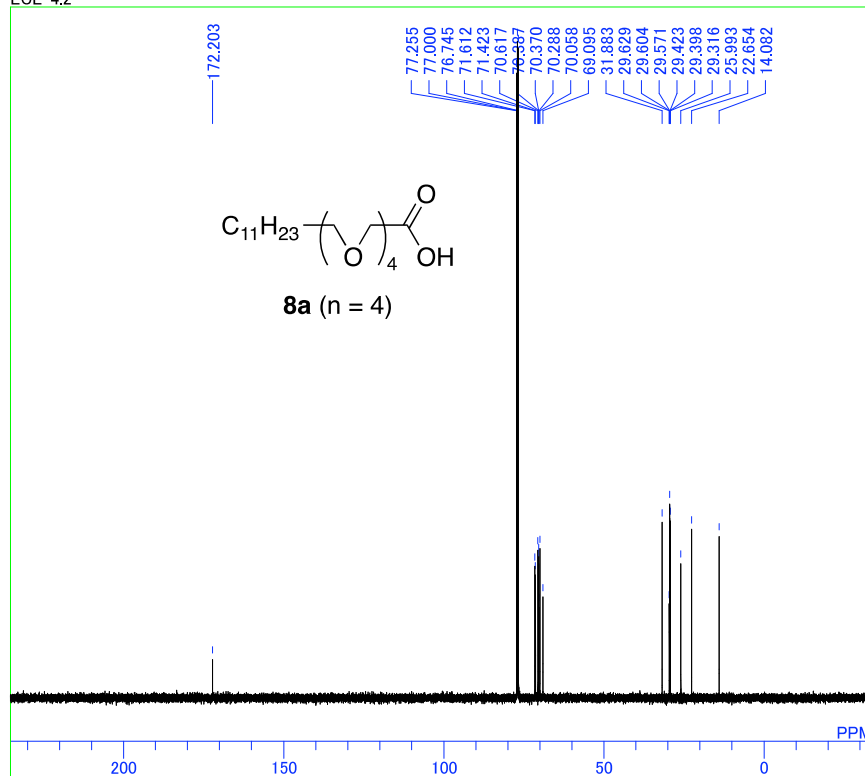
^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of ECL-4.2(3,6,9,12-tetraoxatetracosanoic acid) (**8a**).

C:\Documents and Settings\ALPHA\1-K-4-2\UozumiG\Torii_X-cube\std\ECL-4.2_H.als
KT3038



MENUF non_th5atfTH5ATFG2_2KT3038
 OBNUC 1H
 OBFREQ 500.00 MHz
 OBFIN 162410.00 Hz
 PW1 6.40 usec
 DEADT 56.80 usec
 PREDL 0.20000 msec
 INIWT 10.0000 msec
 POINT 16384
 SAMPO 16384
 TIMES 16
 DUMMY 1
 FREQU 10000.00 Hz
 FILTR 5000 Hz
 DELAY 40.00 usec
 ACQTM 1.6384 sec
 PD 2.0000 sec
 ADBIT 16
 RGAIN 20
 BF 0.00 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD SINGL
 EXPCM Single pulse
 IRNUC 1H
 IRFRQ 500.00 MHz
 IRFIN 162410.00 Hz
 IRRPW 50 usec
 IRATN 511
 DFILE C:\Documents and Settings\ALPHA\1-
 SHMFL TH5ATFG2_2KT3038
 LKFIN 70334.0 Hz
 LKLEV 180
 LGAIN 21
 LKPHS 212
 LKSIG 729
 CSPED 12 Hz
 FILDC
 FILDF

C:\Documents and Settings\ALPHA\1-K-4-2\UozumiG\Torii_X-cube\std\ECL-4.2_C-f.als
ECL-4.2

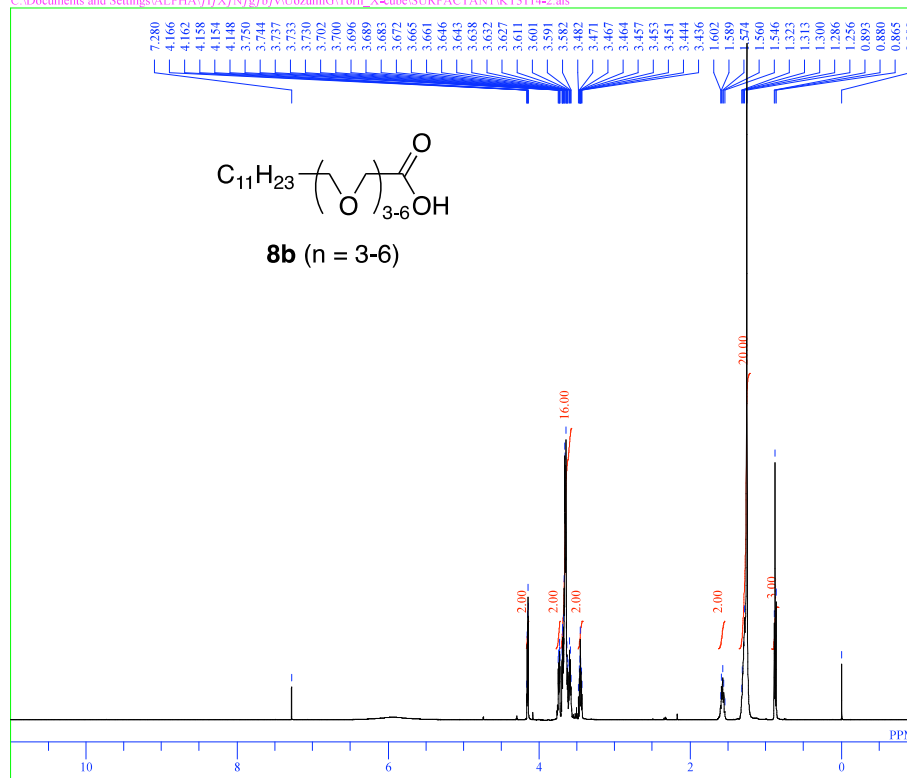


MENUF bcm_th5atfTH5ATFG2_2ECL-4.2
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 OBFREQ 125.65 MHz
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 PW1 5.75 usec
 DEADT 10.00 usec
 PREDL 0.20000 msec
 INIWT 10.0000 msec
 POINT 32768
 SAMPO 32768
 TIMES 2000
 DUMMY 1
 FREQU 33898.30 Hz
 FILTR 16950 Hz
 DELAY 11.80 usec
 ACQTM 0.9667 sec
 PD 1.0000 sec
 ADBIT 16
 RGAIN 27
 BF 0.25 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD SINGL
 EXPCM Single pulse
 IRNUC ^{13}C
 IRFRQ 500.00 MHz
 IRFIN 162410.00 Hz
 IRRPW 50 usec
 IRATN 511
 DFILE C:\Documents and Settings\ALPHA\1-
 SHMFL TH5ATFG2_2ECL-4.2
 LKFIN 70334.0 Hz
 LKLEV 180
 LGAIN 22
 LKPHS 210
 LKSIG 849
 CSPED 15 Hz
 FILDC
 FILDF

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of detergent **8b**.

KT3114-2

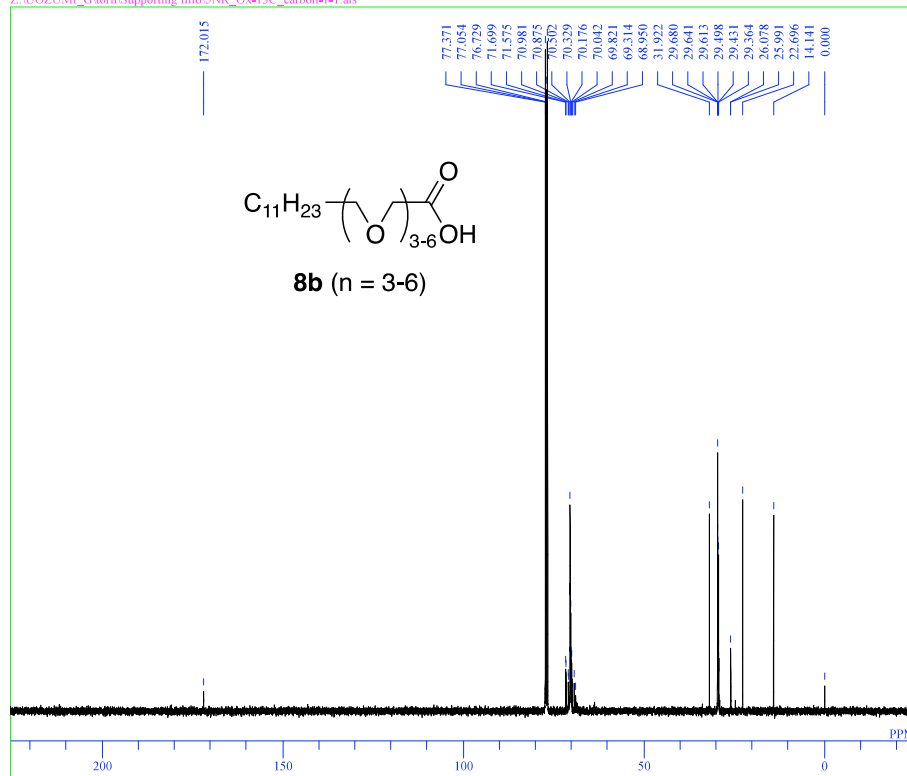
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 EXMOD SINGL
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 OBSSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 32
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PW1 6.40 usec
 IRNUC 1H
 CTEMP 22.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 12

single pulse decoupled gated NOE

Z:\UOZUMI_G\Torii\Supporting_Info\5NR_Ox=13C_carbon-1-1.als



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 COMNT single pulse decoupled gated NOE
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 EXMOD carbon.jsp
 OBFREQ 99.55 MHz
 OBSSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 1062
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 IRNUC 1H
 CTEMP 19.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.80 Hz
 RGAIN 60